Supporting Information

Regioselective synthesis of novel 4,5-diaryl functionalized 3,4-dihydro-pyrimidine-2(1*H*)-thiones *via* non-Biginelli-type approach and evaluation of their *in vitro* anticancer activity

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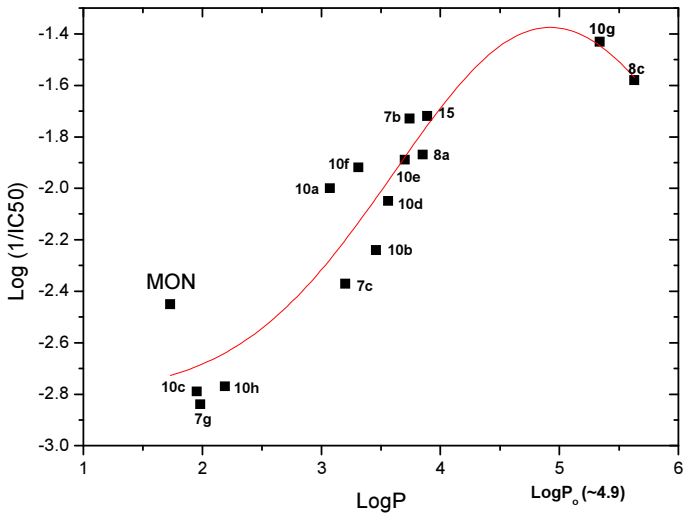


Figure 1. Dependence of Log(1/IC50) vs LogP

WST-1 test. Cancer cell lines and WST-1 test were purchased from Aldrich and Roche, respectively. For the present study, the cancer cell lines were seeded into a 96-well plate at density of 3 x 10^3/well, and then cultured in a humidified incubator with 5% CO_2 at 37°C in 100 µl of medium. The following cell lines were cultured in respective media: MCF7 - human breast adenocarcinoma (ECACC No. 86012803) in Dulbecco's modified eagle's medium (D-MEM), A375 – human malignant melanoma (ECACC No. 88113005) in Eagle's Minimum Essential Medium (EMEM), PC-3 - human Caucasian prostate adenocarcinoma (ECACC No. 90112714) in Coons Modified Ham's F12 medium, and SK-OV-3 - human Caucasian ovary adenocarcinoma (ECACC No. 91091004) in McCoy's 5A medium. The cell culture medium was supplemented with 10% of fetal bovine serum (FBS), 0.4% streptomycin/penicillin and glutamine (2 mM). After incubation period of 24 hours, cells were exposed to the compounds tested in increasing concentrations, i.e. 1.6; 8; 40; 100; 200; 500 and 1000 µM for 48 hours. In parallel, the cells were treated with vehicle (DMSO 0.2%, v/v). After incubation, WST-1 reagent was added for 1 hour, and the absorbance was measured spectrophotometrically at 450 nm (with 620 nm background correction) with the use of Sunrise Reader (Tecan,

Switzerland). The results were acquired from three independent experiments (conducted in triplicate) using cells from different passages (2-10). The data were presented as % of proliferation inhibition for each concentration, as well as mean IC50 ± SD (μM) values together with their standard deviations, calculated from viability curves. IC50 values were calculated by linear regression analysis of dose-response curves, plotting tested concentrations vs. respective % of cell growth inhibition, using ED50 plus 1.0 software.

LDH test. Direct toxicity evaluation of the compounds was based on measurement of activity of lactate dehydrogenase (LDH) in culture medium, with the use of a commercially available kit CytoTox96 Non-Radioactive Cytotoxicity Assay (Promega, WI, USA), applied according to the manufacturer's instructions. The LDH leakage assay is based on the measurement of lactate dehydrogenase activity in the extracellular medium. The loss of intracellular LDH and its release into the culture medium is an indicator of irreversible cell death due to cell membrane damage. MCF7 cells were seeded at a density of 1 x 10^4 per well and cultured at the same conditions as for WST-1 test. After incubation period of 24 hours, the cells were exposed to the compounds studied in increasing concentrations, i.e. 1.6; 8; 40; 100; 200; and 500 μM for 24 hours. In parallel, the cells were treated with vehicle (DMSO 0.2%, v/v). After 24 h of incubation plate was centrifuged at 250×g for 4 minutes in order to obtain a cell-free supernatant and aliquots of supernatant were then transferred to fresh 96-well flat-bottom plates. Unexposed to the tested compounds cells were considered as control (untreated, as well as positive – after complete lysis). The reconstituted substrate mix (50 μl) was added to each well, and incubated at room temperature for 30 minutes, protected from light. Finally, 50 μl of stop solution was added to each well, and the absorbance was measured at 490 nm (with 620 nm background correction) using Sunrise microplate reader (Tecan, Switzerland). The results were acquired from three independent experiments (each conducted in triplicate) using cells from different passages (2-10). Results were normalized to the control cells, and the percentage of necrotic cells was calculated using the following formula: % cytotoxicity= [(experimental LDH release – control / maximum LDH release – control)] × 100%.

General procedure for the synthesis of 5-aryl substituted pyrimidine-2(1*H*)-thiones 6a-6g: In a 100-mL flask, equipped with a magnetic stirrer bar, condenser (protected with $CaCl_2$ drying tube), 3.0 g (0.01 mol) of 2-aryl-3-(dimethylamino)allylidene(dimethyl)-ammonium perchlorate (**5**) and of thiourea or N-Me or N-Bn derivative **3** (0.015 mol) was dissolved in 30 mL of abs. ethanol. Subsequently, of MeONa solution (20 mL, 1 M in MeOH) was added and the reaction mixture was stirred at rt. for 0.5 h and next refluxed for 18 h. After cooling to rt. acetic acid (5 mL) was added. For N-substituted thiourea derivatives additionally water (50 mL) was added. Precipitated product separated by suction, washed with water, dried and crystallized from methanol yielded **6** as yellow solid.

5-phenylpyrimidine-2(1*H*)-thione (**F1**) or **5-phenylpyrimidine-2-thiol** (**F2**) (**6a**): Yield 90%. Yellow solid. M.p. 228 – 230 °C (MeOH); ref[1] m.p. 212–214 °C (MeOH). ^{1}H NMR (400 MHz, DMSO-d_6, 23 °C, recorded shortly after dissolution, tautomer **F1**): δ = 7.35 - 7.43 (m, 1 H, C_6H_5), 7.43 - 7.51 (m, 2 H, C_6H_5), 7.71 (d, J = 7.8 Hz, 2 H, C_6H_5), 8.64 (br. s., 2 H, =CH-4, =CH-6) 14.15 (br. s., 1 H, NH) ppm. ^{1}H NMR (400 MHz, CF_3COOD, 23 °C, tautomer **F2**): δ = 7.50 - 7.68 (m, 5 H, C_6H_5), 8.85 (s, 2 H, =CH-4, =CH-6) ppm. ^{13}C NMR (100 MHz, CF_3COOD, 23 °C; tautomer **F2**): δ = 126.30, 127.54, 129.74, 132.05, 132.99, 156.47, 170.39 ppm. IR (KBr): 3040, 2588 br, 2056 br, 1952 br, 1624, 1598, 1584, 1488, 1444, 1374, 1350, 1306, 1276, 1222, 1206, 1178, 1156, 1032, 1004, 992, 926, 792, 760, 696 cm^{-1}.

5-(4-methoxyphenyl)pyrimidine-2-thiol (**6b**): Yield 87%. Yellow solid. M.p. 246 – 249 °C (EtOH); ref[2] m.p. 218 °C (*n*-PrOH). ^{1}H NMR (400 MHz, CF_3COOH, DMSO-d_6 – external lock, 23°C): δ = 4.04 (s, 3 H, OCH_3), 7.24 (d, J = 8.8 Hz, 2 H, ArH), 7.56 (d, J = 8.8 Hz, 2 H, ArH), 8.86 (s, 2 H, =CH-4, =CH-6) ppm. ^{13}C NMR (100 MHz, CF_3COOH, DMSO-d_6 – external lock): δ = 57.42, 117.98, 123.68, 126.24, 129.56, 156.29, 162.90, 170.24) ppm. IR (KBr): 3052, 2960, 2836, 2652 br, 2048 br, 1938 br, 1610, 1572, 1518, 1484, 1372, 1348, 1290, 1256, 1218, 1184, 1046, 1028, 994, 920, 836, 812, 768, 740 cm^{-1}.

5-(4-fluorophenyl)pyrimidine-2-thiol (**6c**): Yield 81%. Yellow solid. M.p. 253 – 256 °C (*n*-BuOH); ref[2] m.p. 226 °C (*n*-BuOH). ^{1}H NMR (400 MHz, CF_3COOH, DMSO-d_6 – external lock, 23°C): δ = 7.30 (t, J = 8.6 Hz, 2 H, ArH), 7.54 - 7.60 (m, 2 H, ArH), 8.86 (s, 2 H, =CH-4, =CH-6) ppm. ^{13}C NMR (100 MHz, CF_3COOH, DMSO-d_6 – external lock) δ = 119.28 (d, J = 23.4 Hz), 125.59, 126.33 (d, J = 4.4 Hz), 130.06 (d, J = 8.8 Hz), 156.66, 166.94 (d, J = 251.8 Hz), 170.34 ppm. IR (KBr): 3048, 2528 br, 2060 br, 1956 br, 1632, 1610, 1572, 1520, 1376, 1358, 1308, 1272, 1242, 1226, 1196, 1164, 1036, 998, 924, 832, 778, 736 cm^{-1}.

1-Methyl-5-phenylpyrimidine-2(1*H*)-thione (**6d**): Yield 59%. Yellow solid. M.p. 247–250 °C (MeOH). ^{1}H NMR (400 MHz, DMSO-d_6, 23°C): δ = 3.89 (s, 3 H, NCH_3), 7.39 - 7.45 (m, 1 H, Ph), 7.51 (t, J = 7.6 Hz, 2 H, Ph), 7.73 (d, J = 7.3 Hz, 2 H, Ph), 8.93 (s, 2 H, CH-4, CH-6) ppm. ^{13}C NMR (100 MHz; DMSO-d_6): 46.17, 121.53, 125.65, 128.21, 129.10, 132.22, 147.99, 157.35, 180.11 ppm. IR (KBr): 3036, 2980, 1656, 1624, 1576, 1512, 1460, 1410, 1300, 1192, 1174, 1158, 1128, 1100, 1082, 916, 904, 766, 736, 716, 696 cm^{-1}. GC-MS (70eV): 202 (M^+, 100), 201 (49), 169 (10), 157 (8), 144 (31), 115 (11), 102 (17), 42 (13). HRMS (ESI-TOF): *m/z* [M^++H] calcd. for $C_{11}H_{11}N_2S$ 203.0643; found 203.0642.

5-(4-Methoxyphenyl)-1-methylpyrimidine-2(1*H*)-thione (**6e**): Yield 50%. Yellow solid. M.p. 237 – 240 °C (MeOH). ^{1}H NMR (400 MHz, DMSO-d_6, 23°C): δ = 3.80 (s, 3 H, NCH_3), 3.88 (s, 3 H, OCH_3), 7.04 - 7.09 (m, 2 H, ArH), 7.64 - 7.69 (m, 2 H, ArH), 8.85 (d, J = 2.9 Hz, 1 H, CH), 8.89 (d, J = 2.9 Hz, 1 H, CH) ppm. ^{13}C NMR (100 MHz; DMSO-d_6, 23°C): 46.13, 55.19, 114.55, 121.50, 124.40, 126.96, 147.18, 157.14, 159.37, 179.55 ppm. GC-MS (70eV): 232 (M^+, 100), 231 (23), 201 (34), 174 (47), 159 (9), 132 (10), 89 (9), 42 (11). IR (KBr): 3044, 2988, 2920, 2836, 1626, 1610, 1580, 1522, 1470, 1412, 1324, 1304, 1284, 1258, 1200, 1110, 1086, 1036, 1022, 928, 908, 832, 804, 796, 732, 712 cm^{-1}. HRMS (ESI-TOF): *m/z* [M^++H] calcd for $C_{12}H_{13}N_2OS$: 233.0749; found 233.0759.

5-(4-fluorophenyl)-1-methylpyrimidine-2(1*H*)-thione (**6f**): Yield 41%. Yellow solid. M.p. 250-256 °C (MeOH). ^{1}H NMR (400 MHz, DMSO-d_6, 23°C): δ = 3.88 (s, 3 H, CH_3), 7.36 (t, J = 8.9 Hz, 2 H, ArH), 7.74 - 7.81 (m, 2 H, ArH), 8.91 (s, 2 H, CH-4, CH-6) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 23°C): δ = 46.13, 116.00 (d, J = 26.4 Hz), 120.67 (C, 127.90 (d, J = 7.3 Hz), 128.79 (d, J = 2.9 Hz), 147.98, 157.29, 162.47 (d, J = 245.9 Hz),180.04 ppm. GC-MS (70eV): 220 (M^+, 100), 219 (39), 162 (42), 133 (10), 120 (18), 42 (14). IR (KBr): 3024, 1628, 1598, 1516, 1464, 1410, 1310, 1232, 1196, 1168, 1100, 1026, 840, 812, 736, 712 cm^{-1}. HRMS (ESI-TOF): *m/z* [M^++H] calcd for $C_{11}H_{10}FN_2S$: 221.0549; found 221.0549.

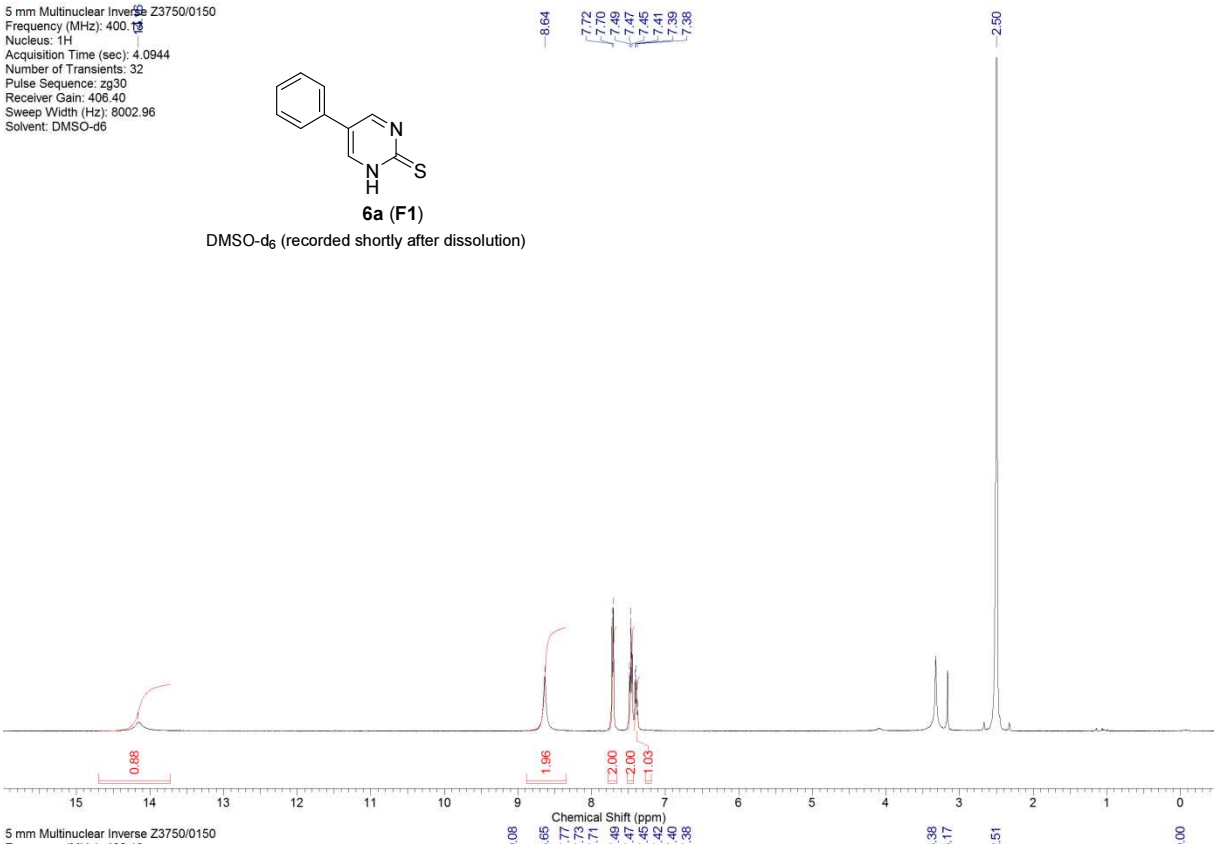
1-Benzyl-5-phenylpyrimidine-2(1*H*)-thione (**6g**): Yield 50%. Yellow solid. M.p. 201 – 204 °C (MeOH). ^{1}H NMR (400 MHz, DMSO-d_6, 23°C): δ = 5.77 (s, 2 H, NCH_2), 7.25 - 7.56 (m, 8 H, Ph), 7.75 (d, J = 7.4 Hz, 2 H, Ph), 8.98 (d, J = 2.8 Hz, 1 H, CH-4), 9.03 (d, J = 2.8 Hz, 1H, CH-6) ppm. ^{13}C NMR (100 MHz; DMSO-d_6, 23°C): δ = 59.25, 122.05, 125.73, 127.68, 127.81, 128.35, 128.41, 129.10, 131.95, 135.10, 147.18, 157.80, 180.19 ppm. GC-MS (70eV): 278 (M^+, 75), 245 (100), 91 (11). IR (KBr): 3032, 1622, 1516, 1456, 1430, 1412, 1332, 1228, 1206, 1168, 1080, 764, 736, 714, 692 cm^{-1}. HRMS (ESI-TOF): *m/z* [M^++H] calcd for $C_{17}H_{15}NO$: 279.0956; found 279.0960.

Synthesis of 12 (Suzuki coupling): To a 50-mL Schlenk flask, equipped with a condenser (connected with gas washing bottle filled with silicone oil), argon balloon and magnetic stir bar, charged with 8 : 2 : 1 mixture of toluene, ethanol and water (9 mL), degassed for 2 h using a stream of argon slowly bubbled through the solution at rt. during vigorous stirring, 1-benzyl-5-bromopyrimidin-2(1*H*)-one (0.32 g, 1.2 mmol), phenylboronic acid (0.22 g, 1.8 mmol), Na_2CO_3 (0.176 g, 1.66 mmol) and $Pd[(PPh_3)]_4$ (97 mg, 0.084 mmol) as catalyst was added. The mixture was stirred at 80 ºC for 20 h. After this time aqueous saturated NH_4Cl (5 mL) was added, the mixture was extracted with ethyl acetate (3 x 50 mL) and the combined organic layers were dried over $MgSO_4$. Filtration, concentration in vacuo and purification by flash column chromatography (silica gel, ethyl acetate) yielded **12** contaminated by PPh_3, which being less soluble was removed by crystallization from a mixture of (*n*-hexane and ethyl acetate). Pure product was obtained as white solid (0.22 g) in 70% yield .

1-Benzyl-5-phenylpyrimidin-2(1*H*)-one (**12**): White solid. M.p. 151 – 153 °C (*n*-hexane and ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$, 23ºC): δ = 5.19 (s, 2 H, NCH_2), 7.31 - 7.46 (m, 10 H, 2Ph), 7.77 (d, J = 3.3 Hz, 1 H, CH-6), 8.86 (d, J=3.3 Hz, 1 H, CH-4) ppm. ^{13}C NMR (100 MHz; $CDCl_3$, 23ºC): δ = 54.20, 118.68, 125.78, 128.12, 128.67, 128.79, 129.25, 129.36, 133.09, 134.77, 144.00, 155.90, 165.21 ppm. GC-MS (70eV): 262 (M^+, 72), 261 (20), 220 (14), 156 (10), 116 (16), 91 (100), 65 (17). IR (KBr: 3064, 1648, 1600, 1580, 1526, 1496, 1454, 1432, 1416, 1398, 1332, 1308, 1232, 1074, 1030, 964, 938, 798, 760, 712, 692, 644 cm^{-1}. HRMS (ESI-TOF): *m/z* [M^++H] calcd for $C_{17}H_{15}N_2O$: 263.1184; found 263.1188.

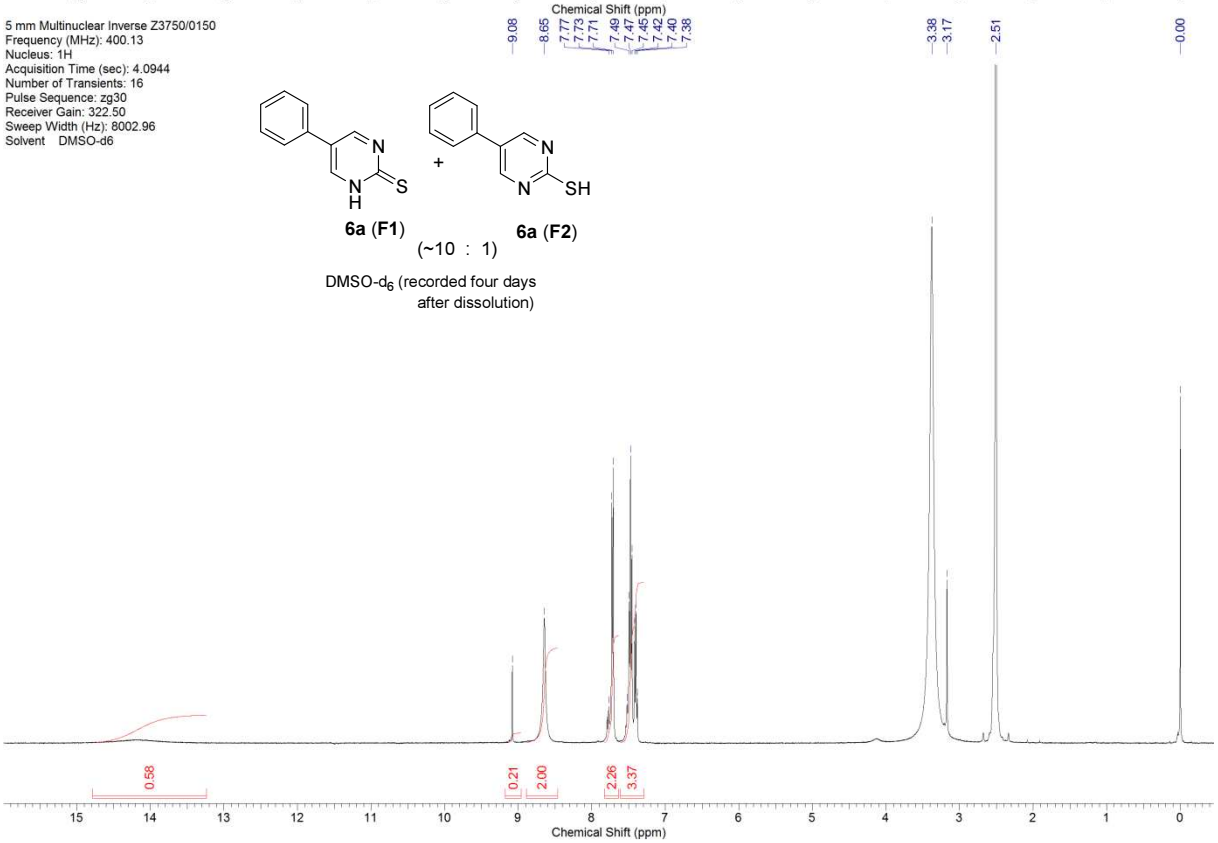
[1] R. M. Wagner, C. Jutz, *Chem. Ber.* **1971**, *104*, 2975-2983;

[2] D. J. Brown, B. T. England, *J. Chem. Soc. C* **1971**, 425-431;



6a (**F1**)

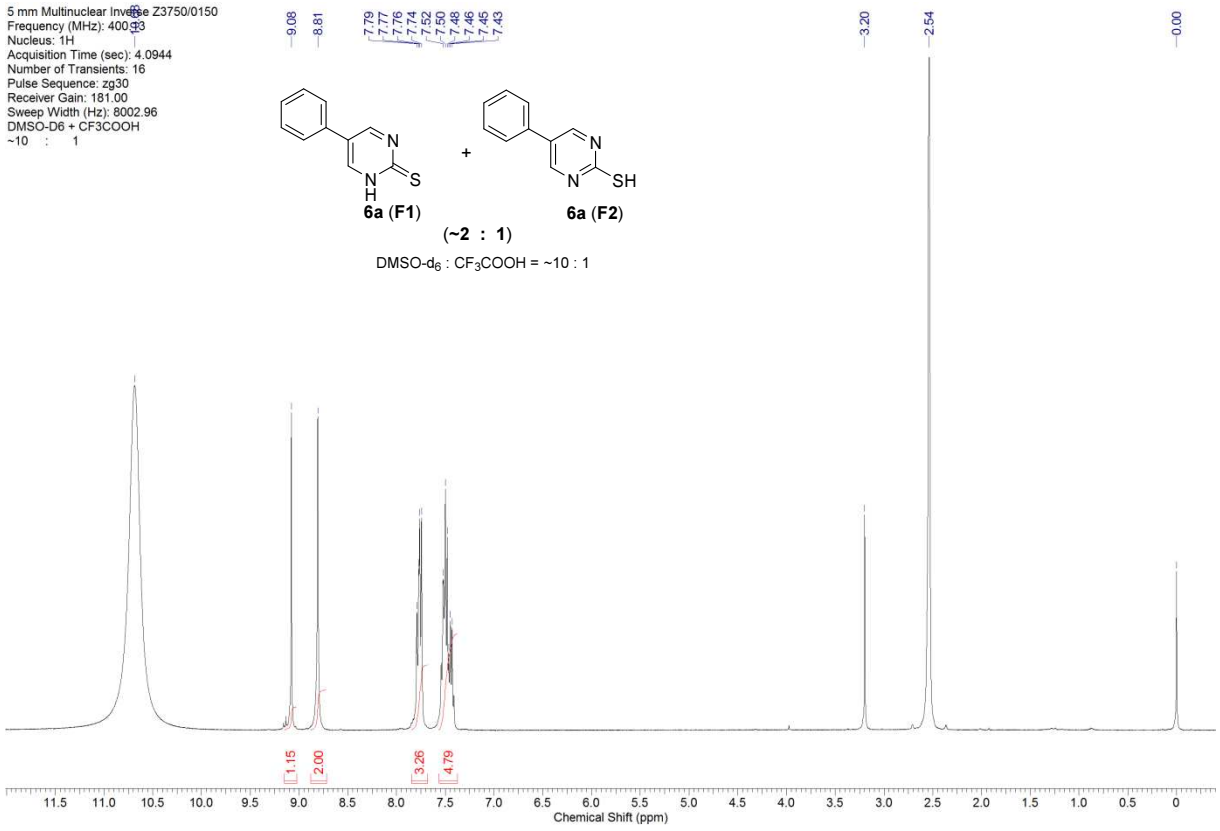
DMSO-d_6 (recorded shortly after dissolution)



6a (**F1**) + **6a** (**F2**)

(~10 : 1)

DMSO-d_6 (recorded four days after dissolution)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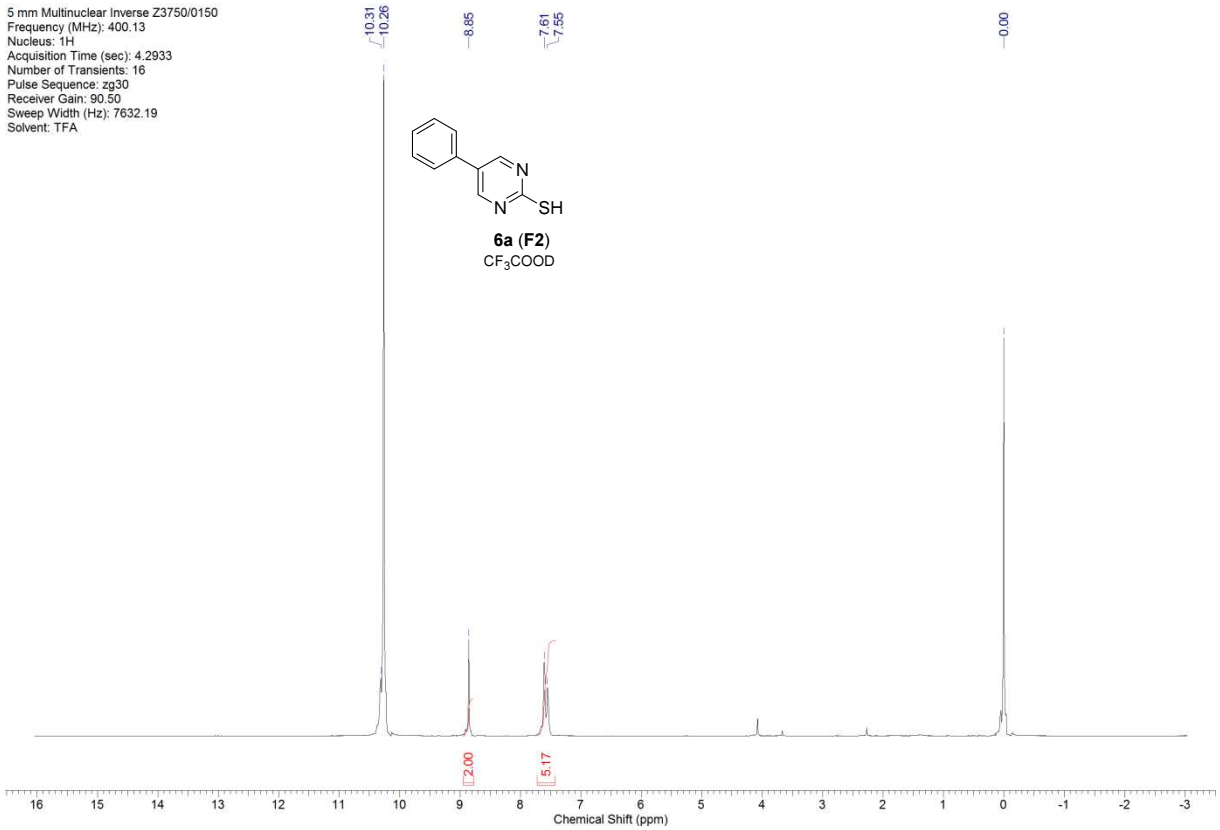
Nucleus: 1H
Acquisition Time (sec): 4.0944
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 181.00
Sweep Width (Hz): 8002.96
DMSO-D6 + CF3COOH
~10 : 1
9.09
8.81
7.79
7.77
7.76
7.74
7.52
7.50
7.48
7.46
7.45
7.43
3.20
2.54
0.00
6a (F1)
+
6a (F2)
(~2 : 1)
DMSO-d6 : CF3COOH = ~10 : 1
1.15
2.00
3.26
4.79
Chemical Shift (ppm)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Nucleus: 1H
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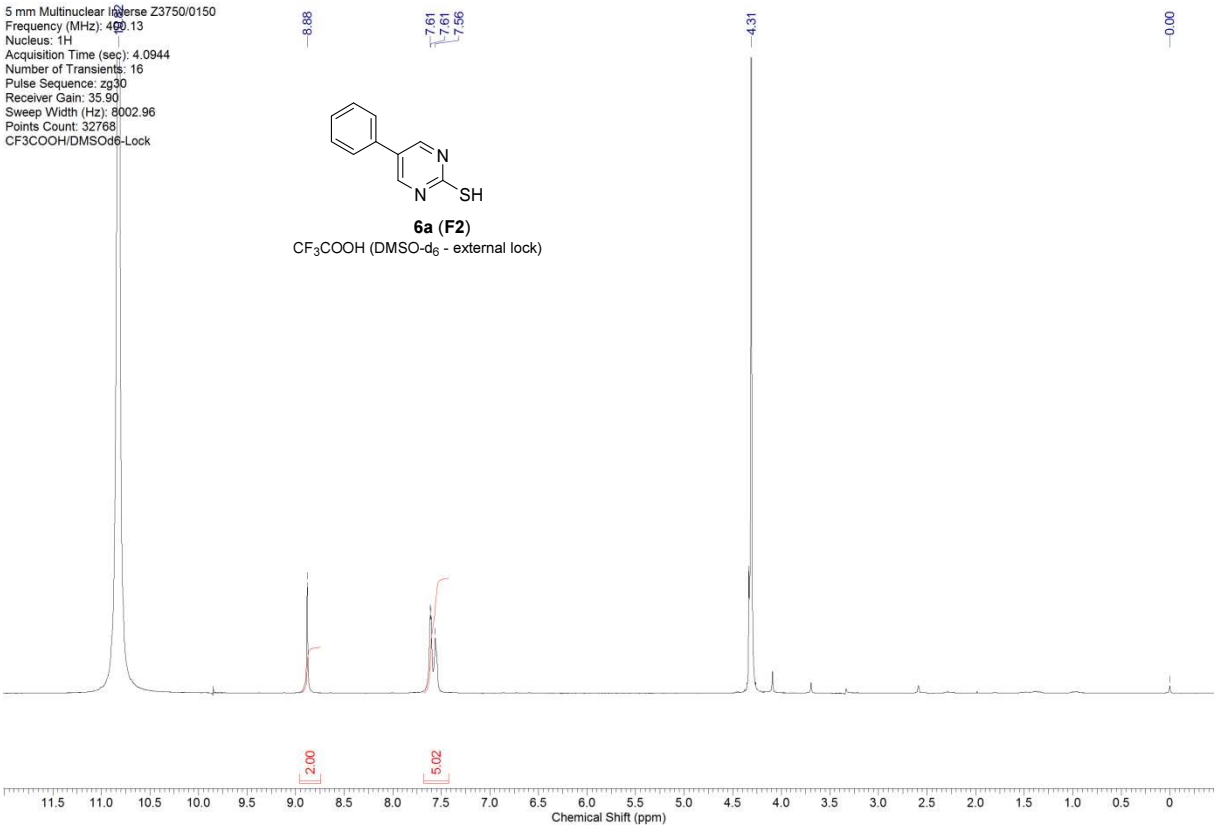
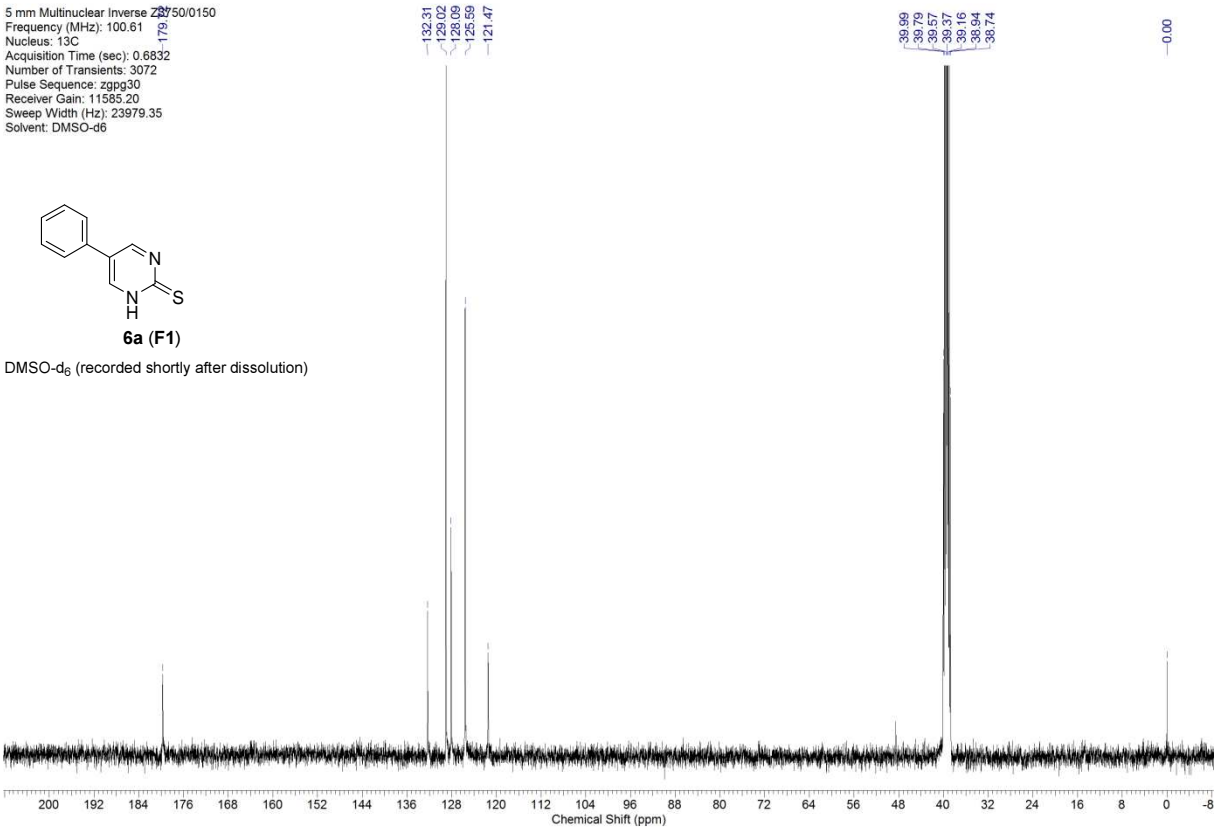
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 90.50
Sweep Width (Hz): 7632.19
Solvent: TFA
10.31
10.26
8.85
7.61
7.55
0.00
6a (F2)
CF3COOD
2.00
5.17
Chemical Shift (ppm)



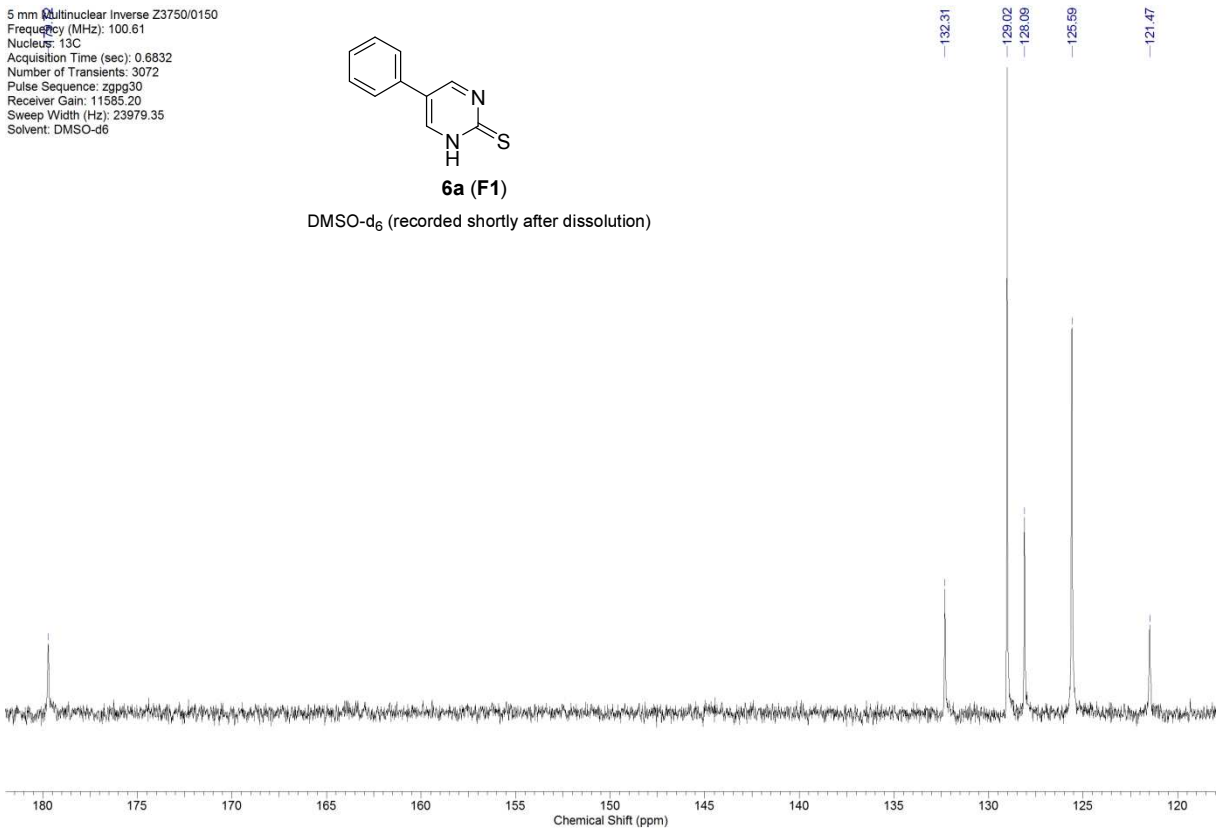
6a (**F2**)

CF_3COOH ($DMSO\text{-}d_6$ - external lock)



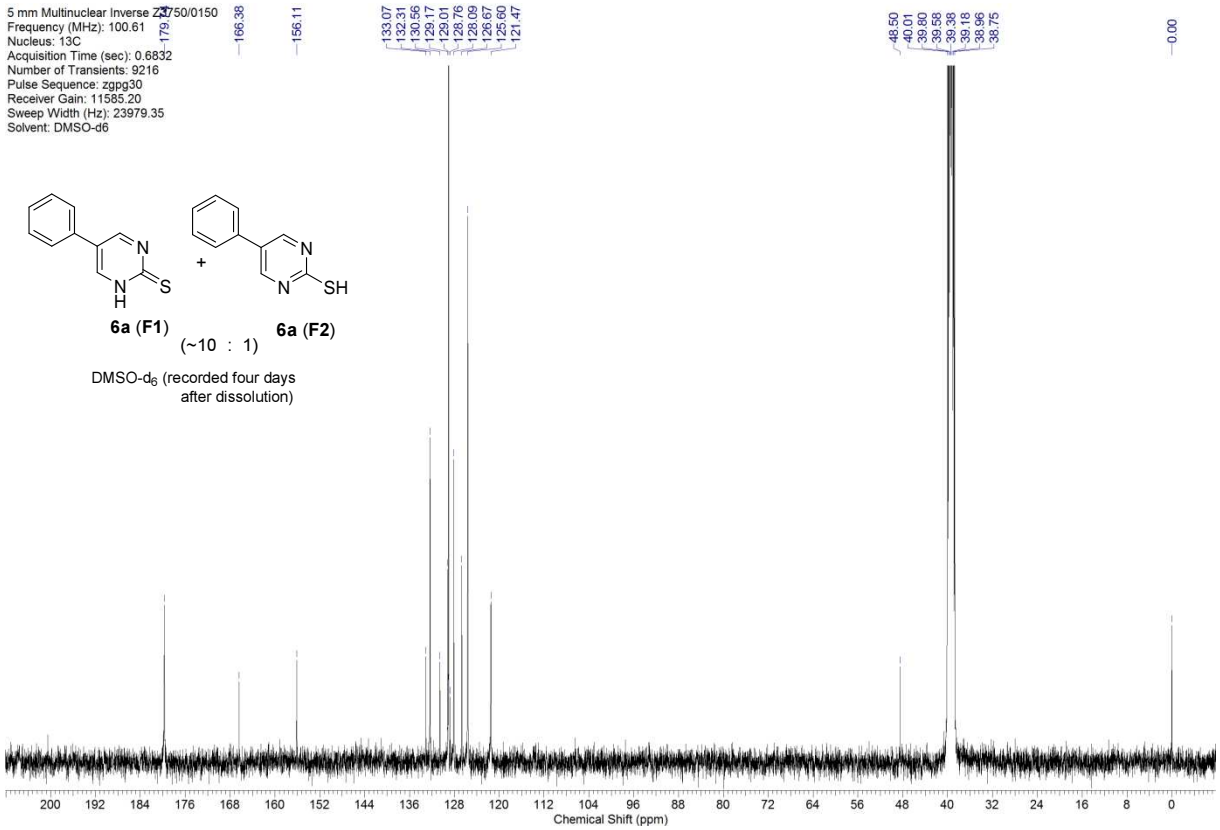
6a (**F1**)

$DMSO\text{-}d_6$ (recorded shortly after dissolution)



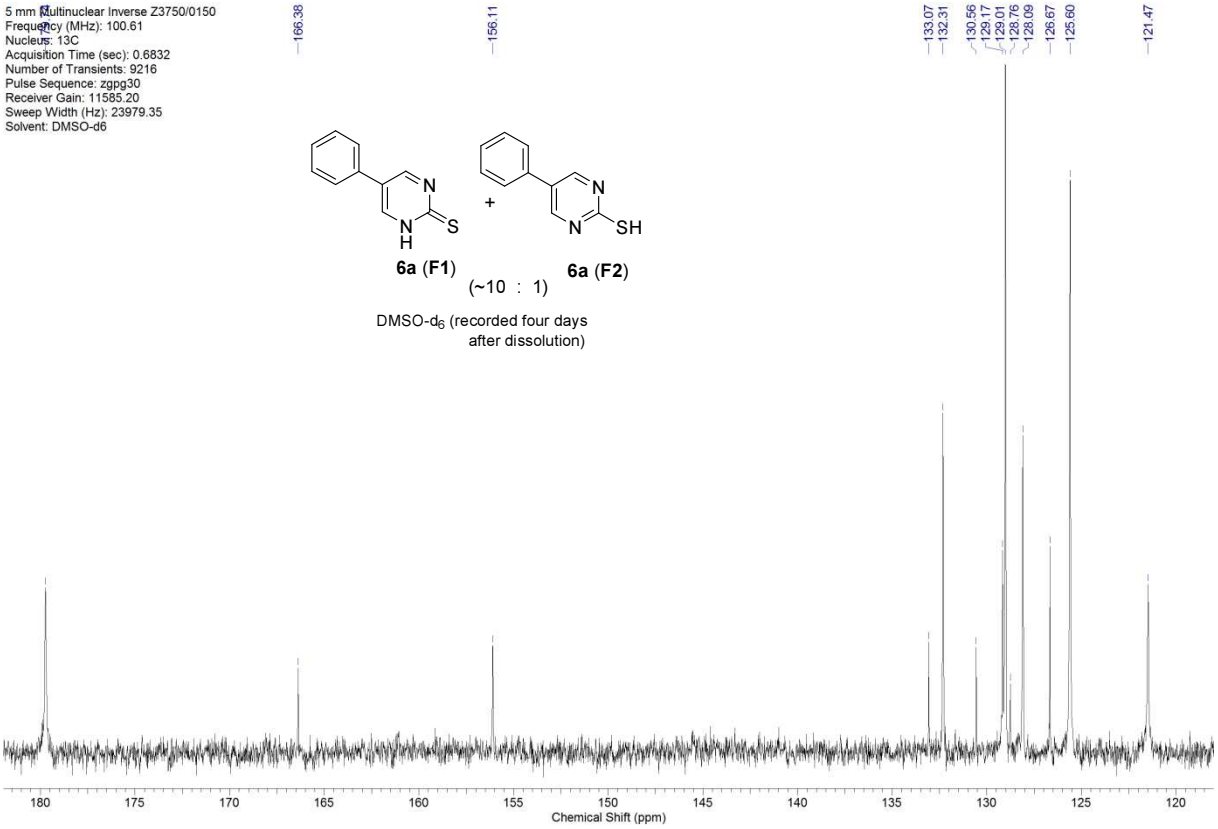
6a (**F1**)

DMSO-d_6 (recorded shortly after dissolution)



6a (**F1**) (~10 : 1) **6a** (**F2**)

DMSO-d_6 (recorded four days after dissolution)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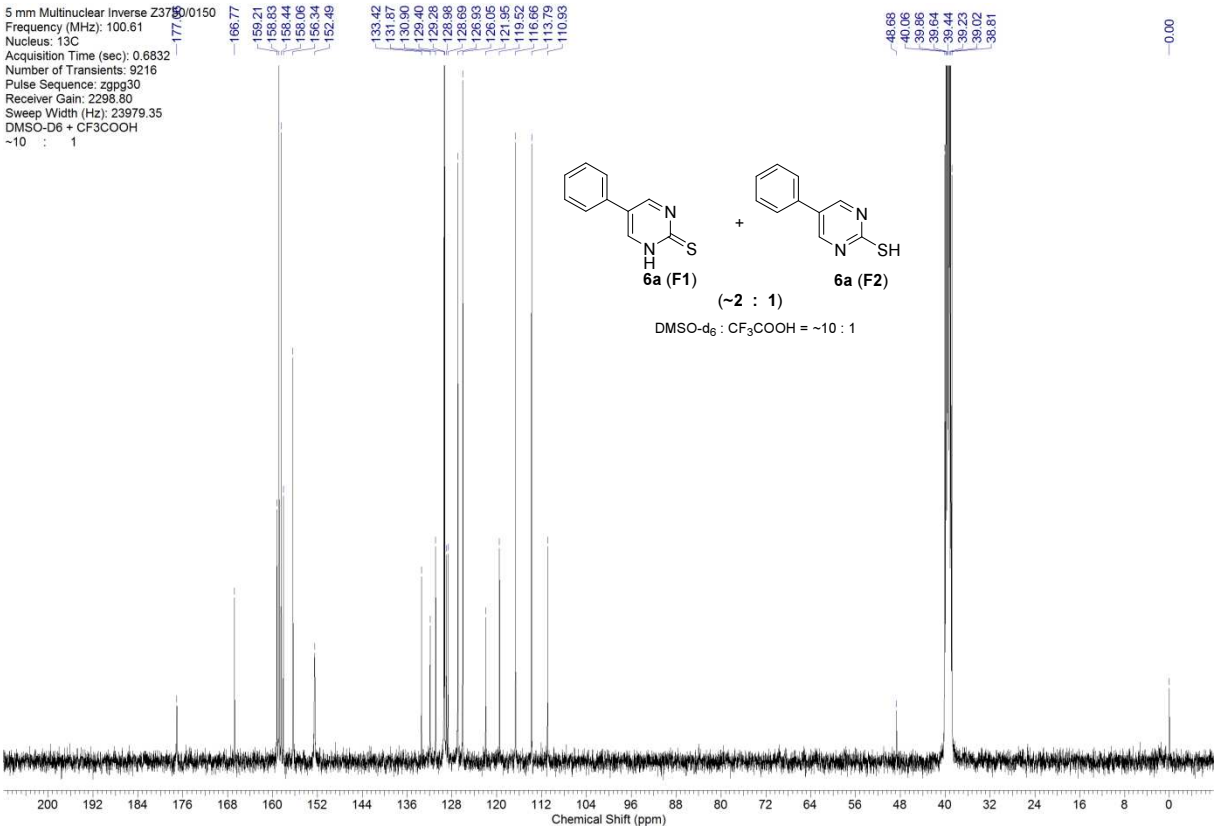
Number of Transients: 9216
Pulse Sequence: zgpg30
Receiver Gain: 11585.20
Sweep Width (Hz): 23979.35
Solvent: DMSO-d6
166.38
156.11
133.07
132.31
130.56
129.17
129.01
128.76
128.09
126.67
125.60
121.47
6a (F1)
(~10 : 1)
6a (F2)
DMSO-d6 (recorded four days after dissolution)
Chemical Shift (ppm)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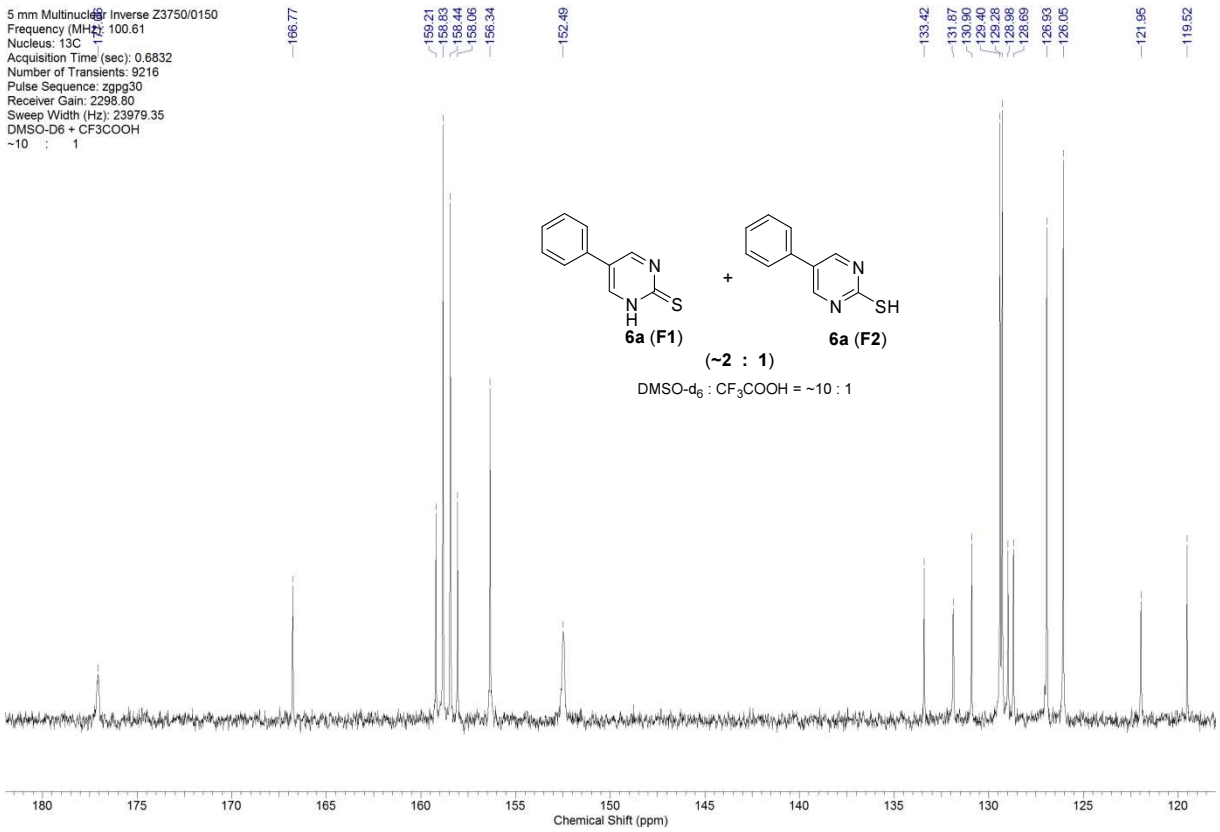
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 9216
Pulse Sequence: zgpg30
Receiver Gain: 2298.80
Sweep Width (Hz): 23979.35
DMSO-D6 + CF3COOH
~10 : 1
177.50
166.77
159.21
158.83
158.44
158.06
156.34
152.49
133.42
131.87
130.90
129.40
129.28
128.98
128.69
126.93
126.05
121.95
119.52
116.66
113.79
110.93
48.68
40.06
39.86
39.64
39.44
39.23
39.02
38.81
0.00
6a (F1)
(~2 : 1)
6a (F2)
DMSO-d6 : CF3COOH = ~10 : 1
Chemical Shift (ppm)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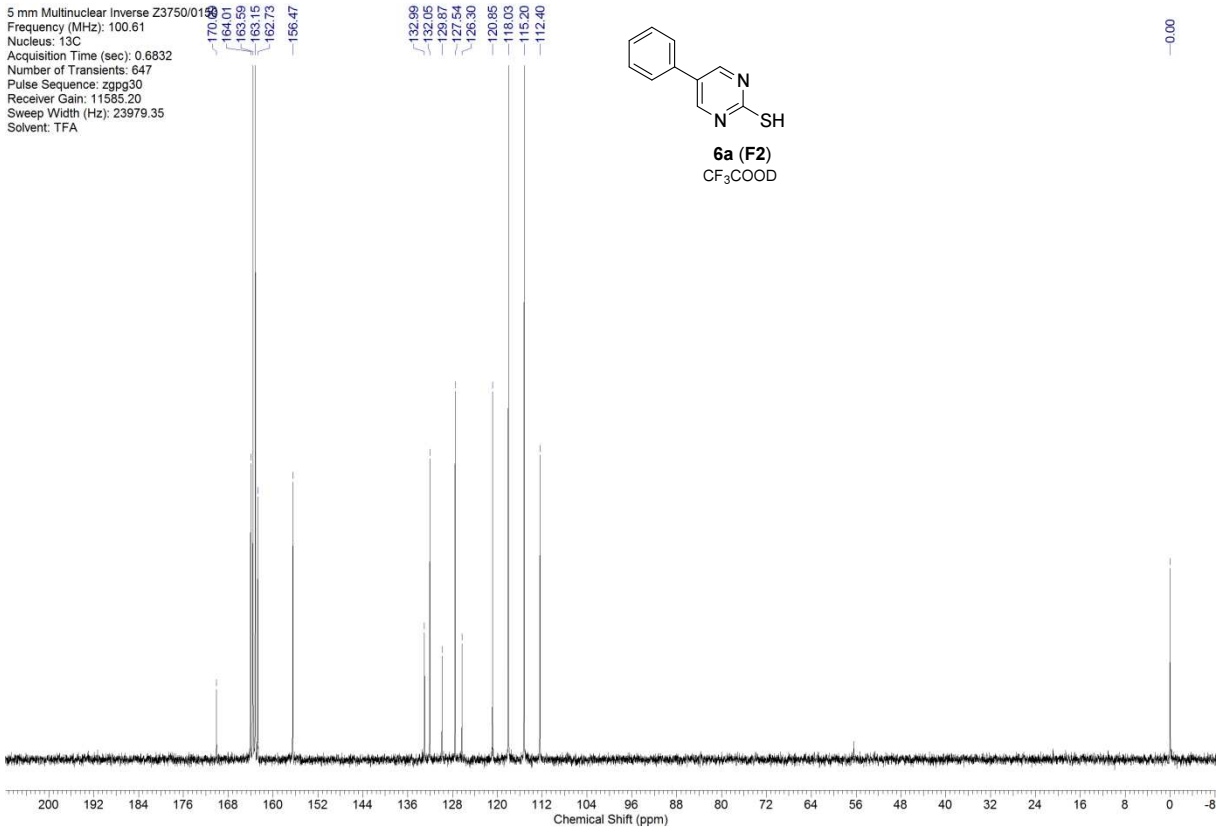
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 9216
Pulse Sequence: zgpg30
Receiver Gain: 2298.80
Sweep Width (Hz): 23979.35
DMSO-D6 + CF3COOH
~10 : 1
166.77
159.21
158.83
158.44
158.06
156.34
152.49
133.42
131.87
130.90
129.40
129.28
128.98
128.69
126.93
126.05
121.95
119.52
6a (F1)
6a (F2)
(~2 : 1)
DMSO-d6 : CF3COOH = ~10 : 1
Chemical Shift (ppm)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Nucleus: 13C
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Number of Transients: 647
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Receiver Gain: 11585.20
Sweep Width (Hz): 23979.35
Solvent: TFA
170.01
164.01
163.59
163.15
162.73
156.47
132.99
132.05
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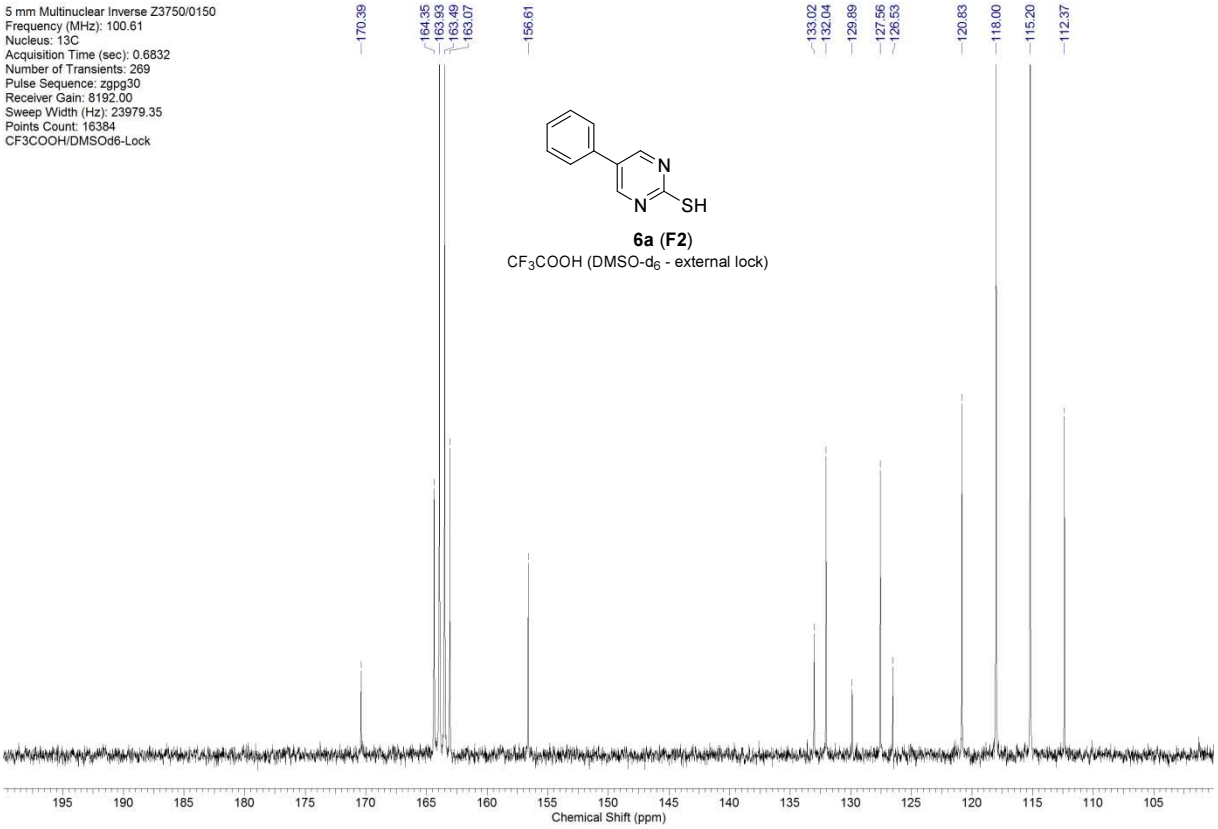
0.00
6a (F2)
CF3COOD
Chemical Shift (ppm)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Nucleus: 13C
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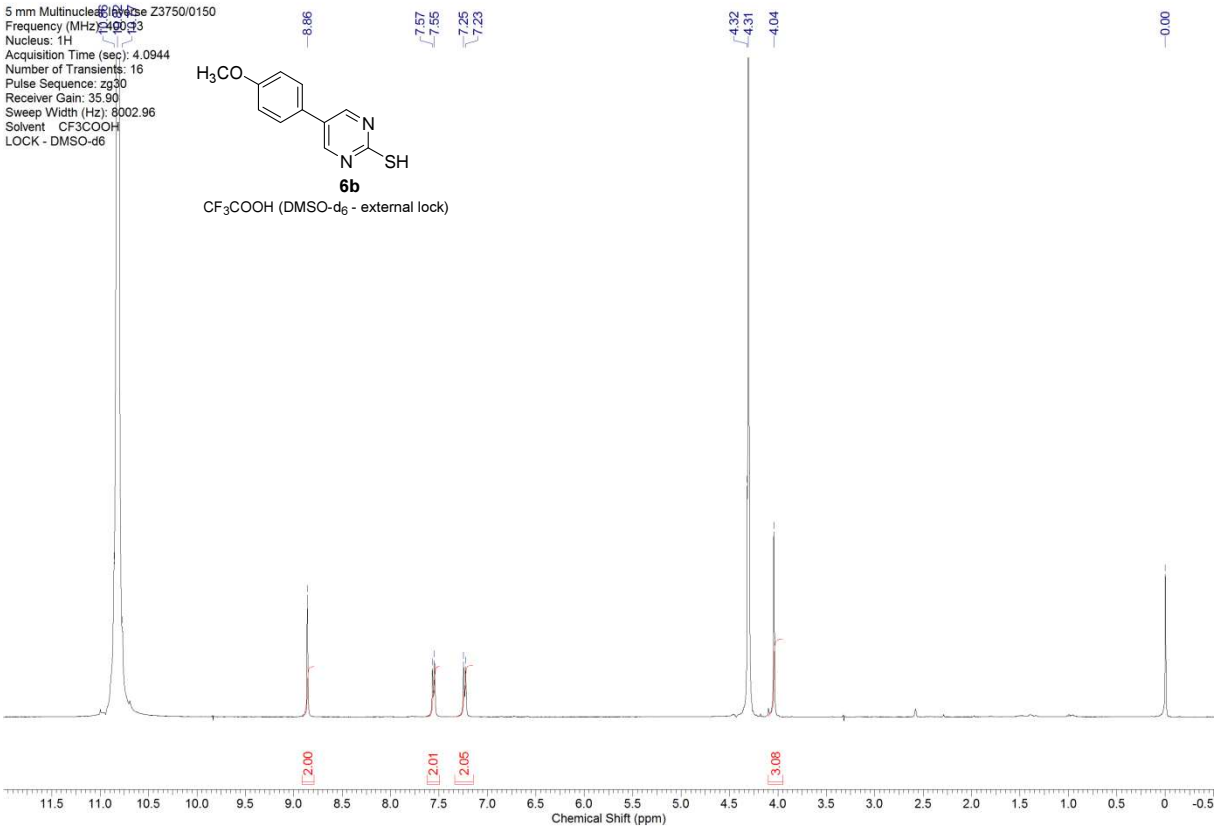
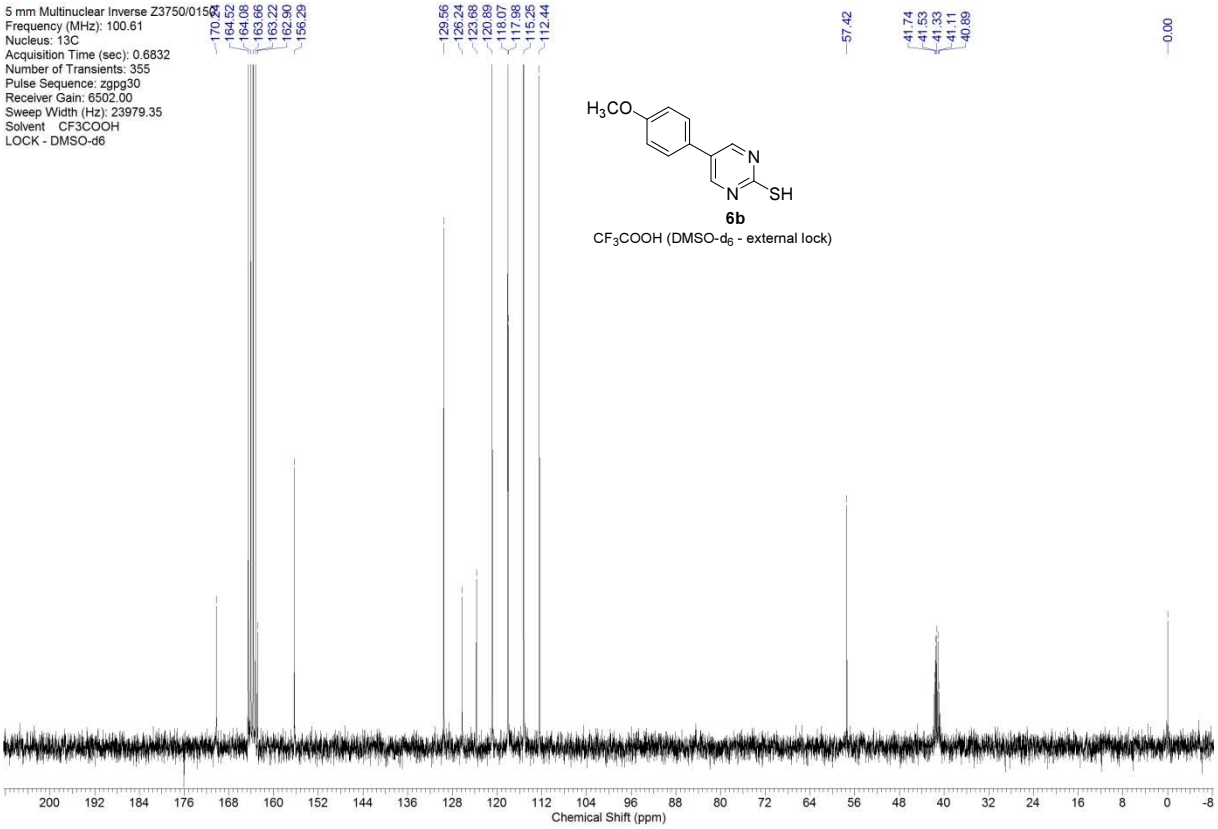
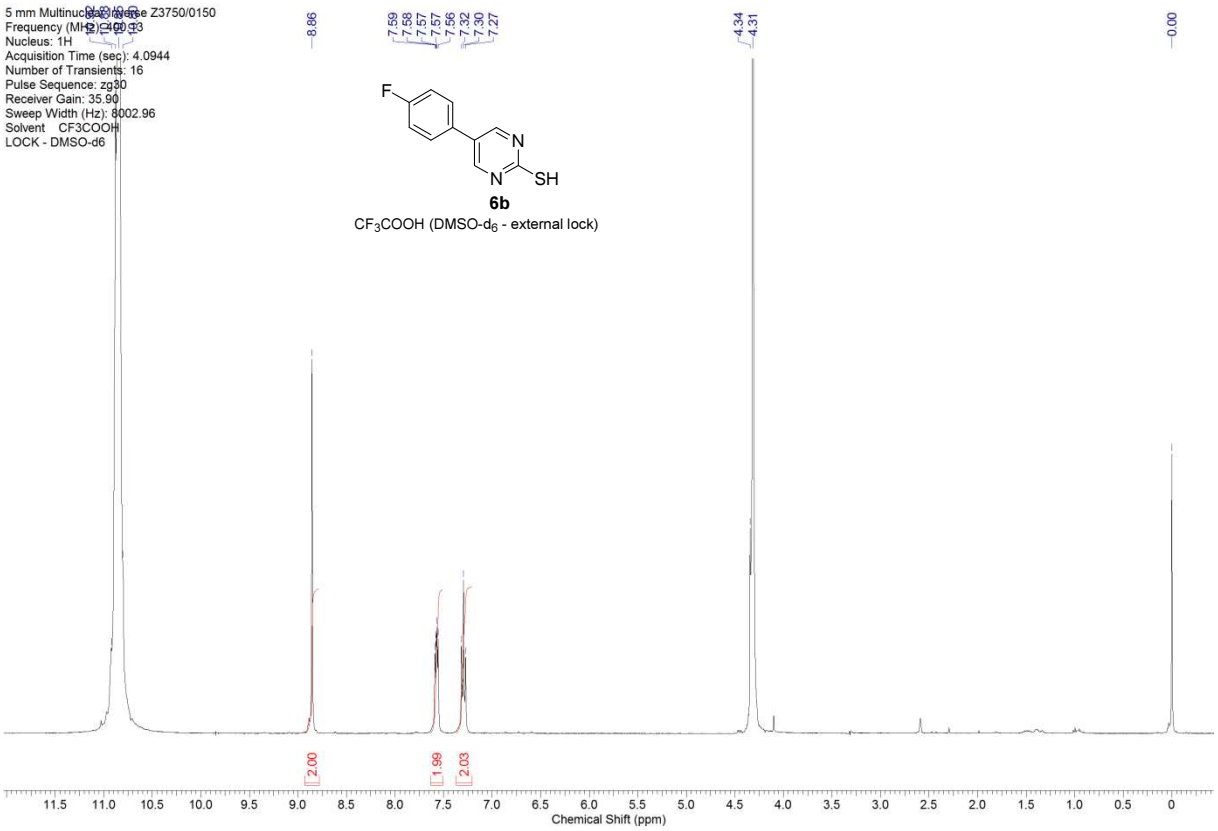
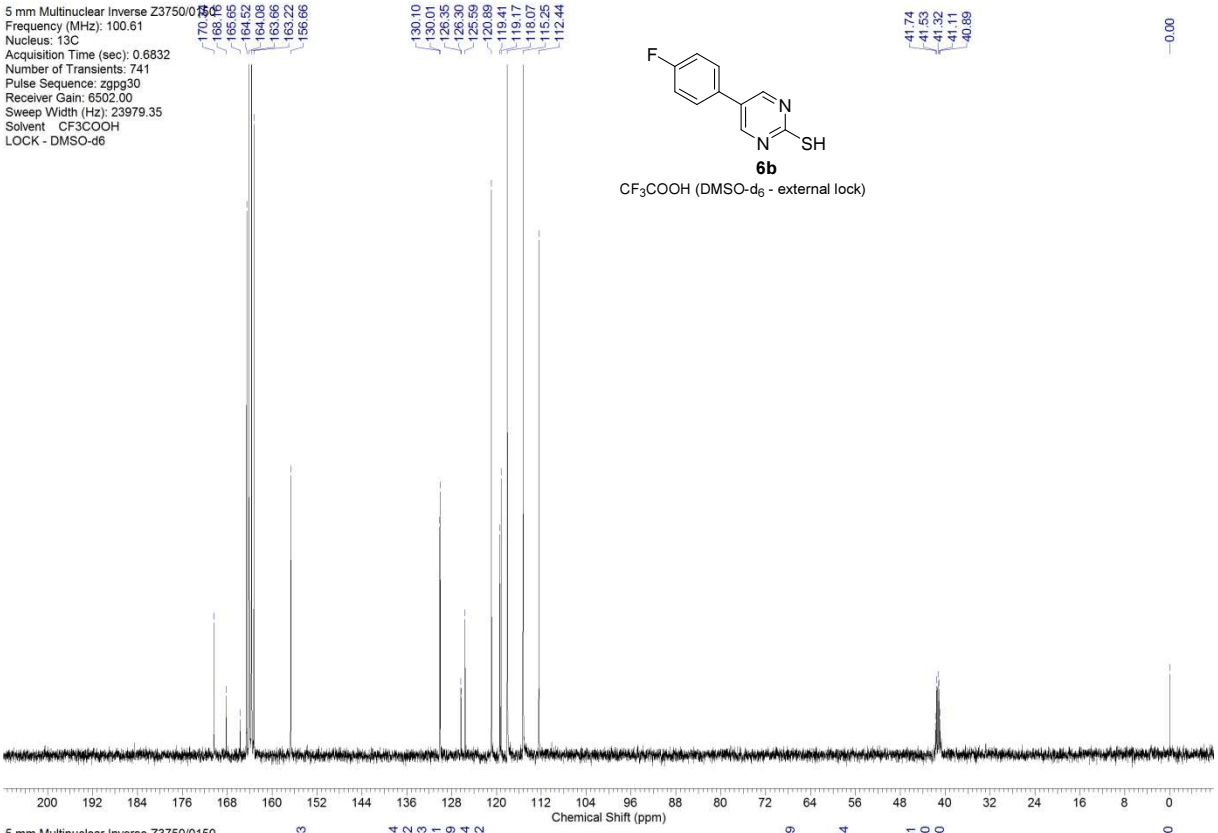
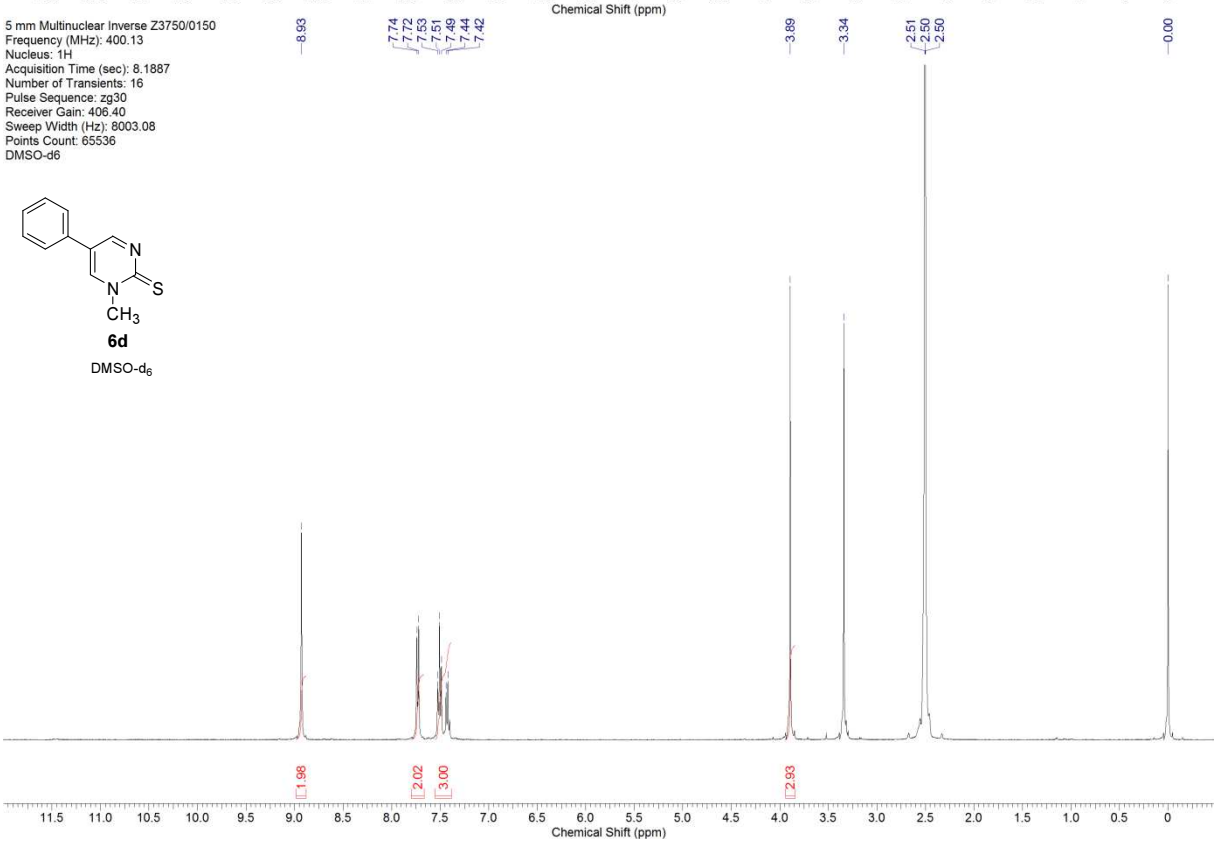
Number of Transients: 269
Pulse Sequence: zgpg30
Receiver Gain: 8192.00
Sweep Width (Hz): 23979.35
Points Count: 16384
CF3COOH/DMSOd6-Lock
170.39
164.35
163.93
163.49
163.07
156.61
133.02
132.04
129.89
127.56
126.53
120.83
118.00
115.20
112.37
N
N
SH
6a (F2)
CF3COOH (DMSO-d6 - external lock)
Chemical Shift (ppm)

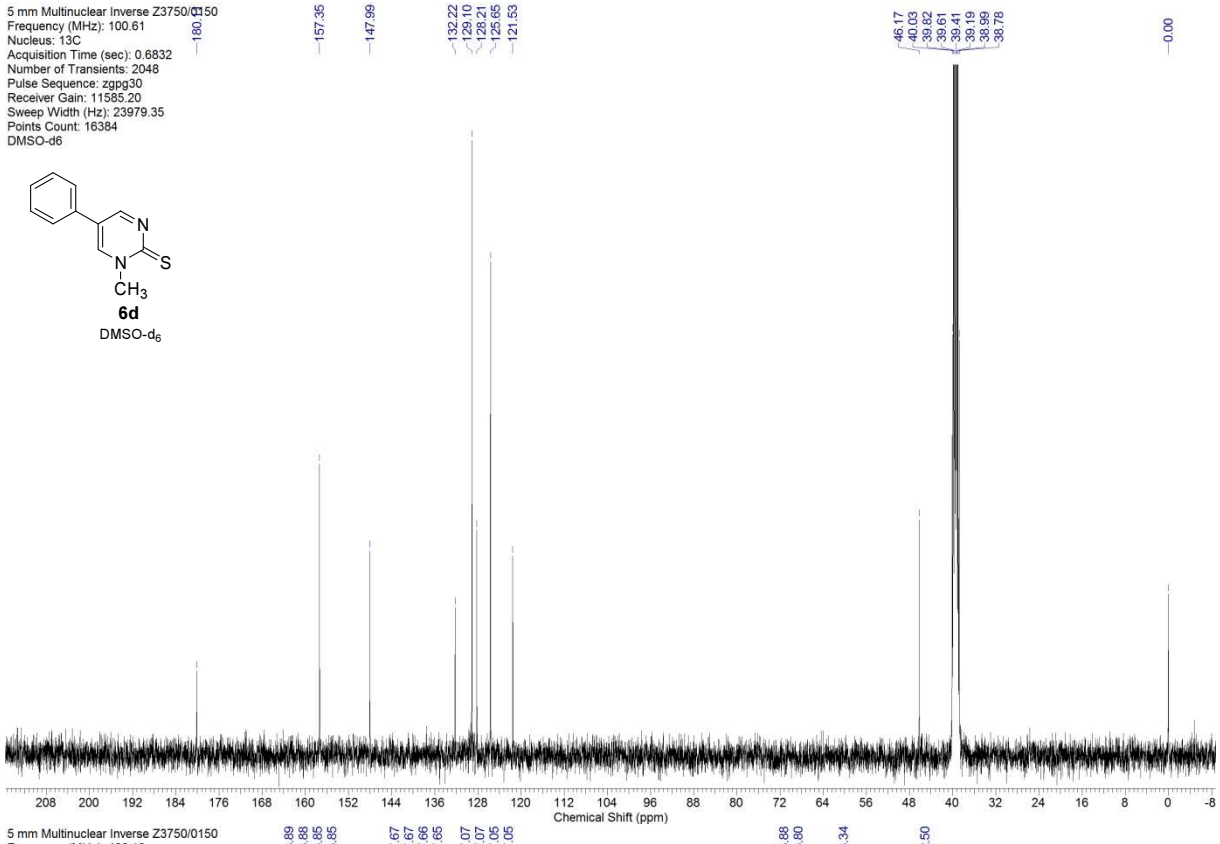

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 4.0944
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 35.90
Sweep Width (Hz): 8002.96
Solvent CF3COOH
LOCK - DMSO-d6
8.86
7.57
7.55
7.25
7.23
4.32
4.31
4.04
0.00
H3CO
N
N
SH
6b
CF3COOH (DMSO-d6 - external lock)
2.00
2.01
2.05
3.08
Chemical Shift (ppm)

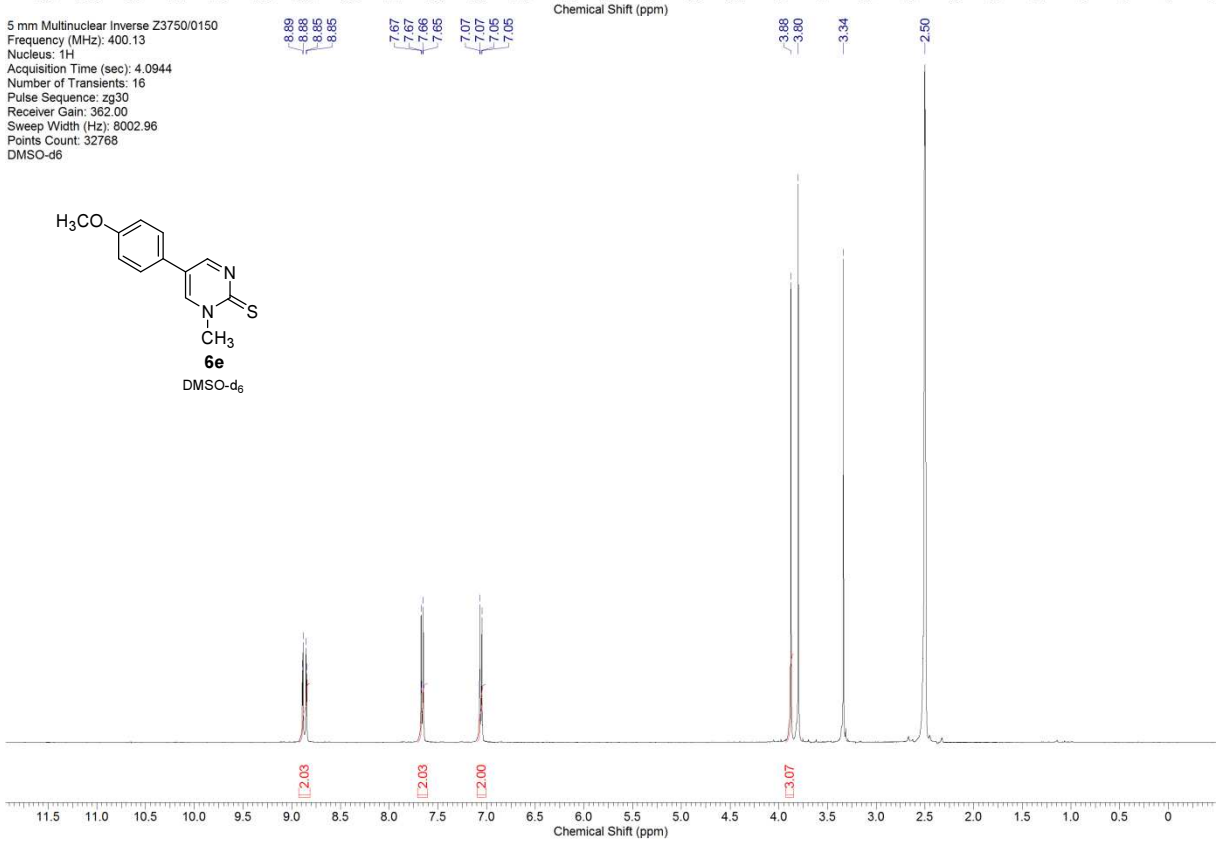

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 355
Pulse Sequence: zgpg30
Receiver Gain: 6502.00
Sweep Width (Hz): 23979.35
Solvent CF3COOH
LOCK - DMSO-d6
170.24
164.52
164.08
163.66
163.22
162.90
156.29
129.56
126.24
123.68
120.89
118.07
117.98
115.25
112.44
57.42
41.74
41.53
41.33
41.11
40.89
0.00
H3CO
N
N
SH
6b
CF3COOH (DMSO-d6 - external lock)
Chemical Shift (ppm)

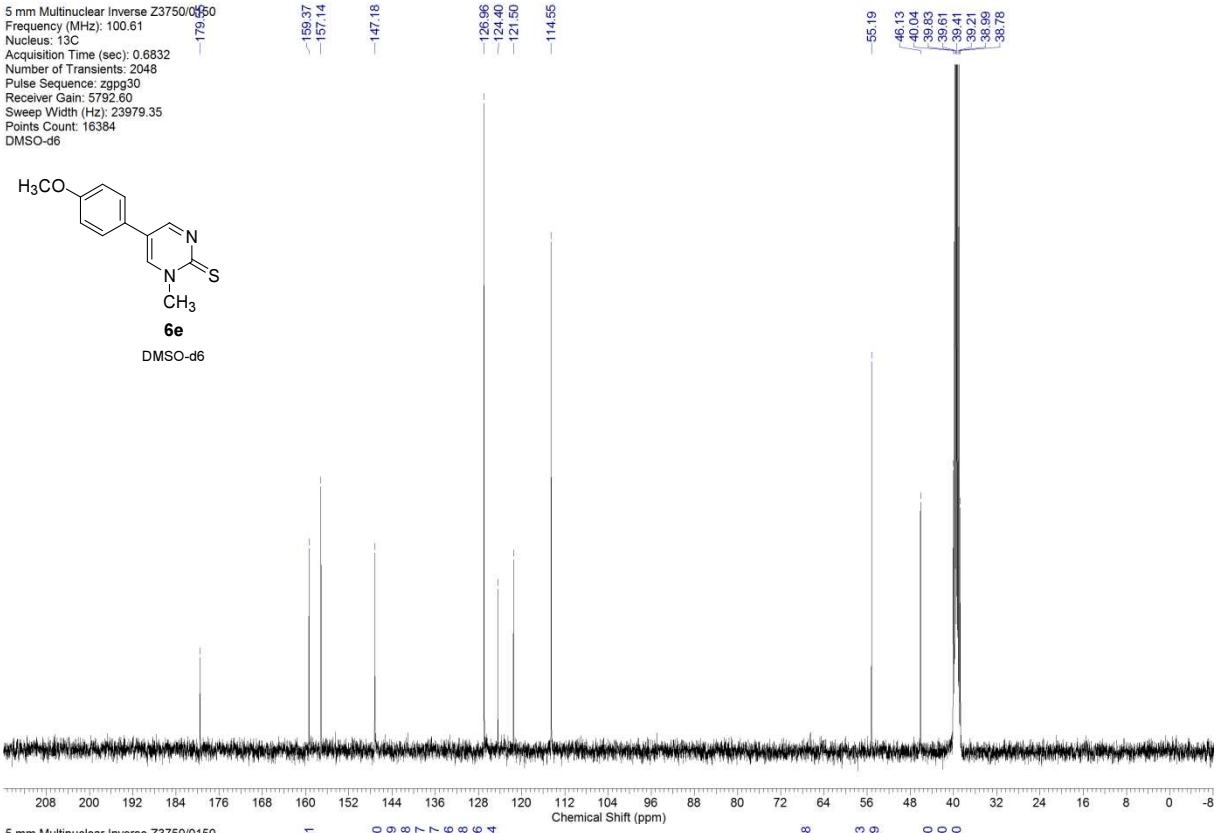

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 4.0944
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 35.90
Sweep Width (Hz): 8002.96
Solvent CF3COOH
LOCK - DMSO-d6
8.86
7.59
7.58
7.57
7.57
7.56
7.32
7.30
7.27
4.34
4.31
0.00
F
N
N
SH
6b
CF3COOH (DMSO-d6 - external lock)
2.00
1.99
2.03
Chemical Shift (ppm)

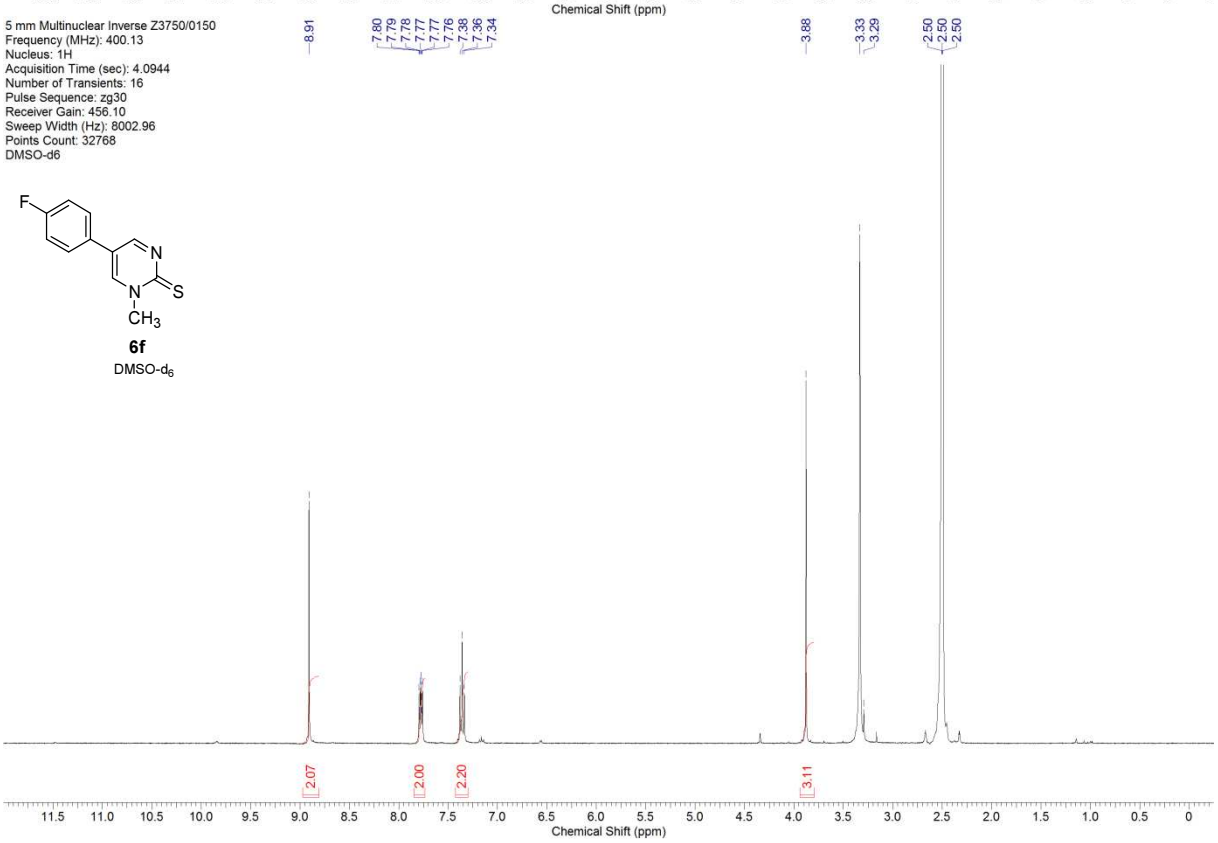

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 741
Pulse Sequence: zgpg30
Receiver Gain: 6502.00
Sweep Width (Hz): 23979.35
Solvent CF3COOH
LOCK - DMSO-d6
170.46
168.15
165.65
164.52
164.08
163.66
163.22
156.66
130.10
130.01
126.35
126.30
125.59
120.89
119.41
119.17
118.07
115.25
112.44
41.74
41.53
41.32
41.11
40.89
0.00
F
N
N
SH
6b
CF3COOH (DMSO-d6 - external lock)
Chemical Shift (ppm)

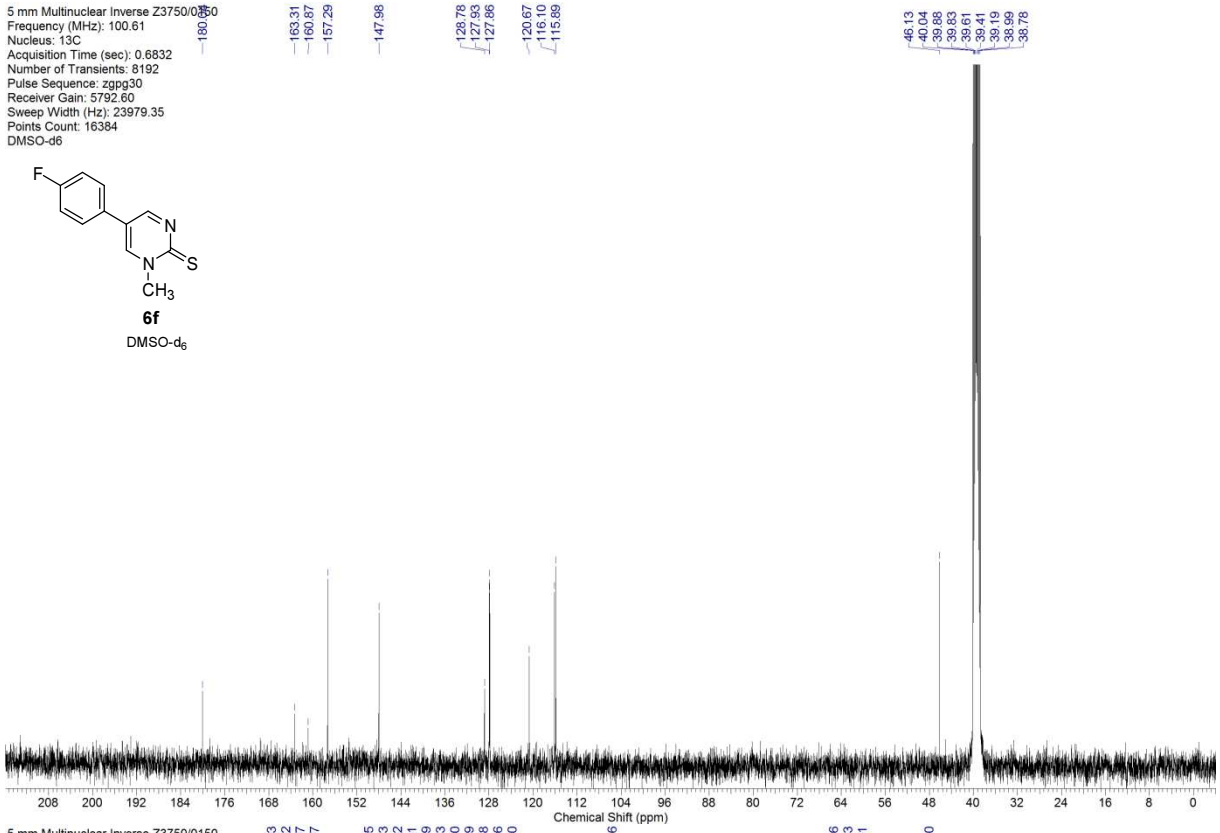

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 406.40
Sweep Width (Hz): 8003.08
Points Count: 65536
DMSO-d6
8.93
7.74
7.72
7.53
7.51
7.49
7.44
7.42
3.89
3.34
2.51
2.50
2.50
0.00
N
N
S
CH3
6d
DMSO-d6
1.98
2.02
3.00
2.93
Chemical Shift (ppm)

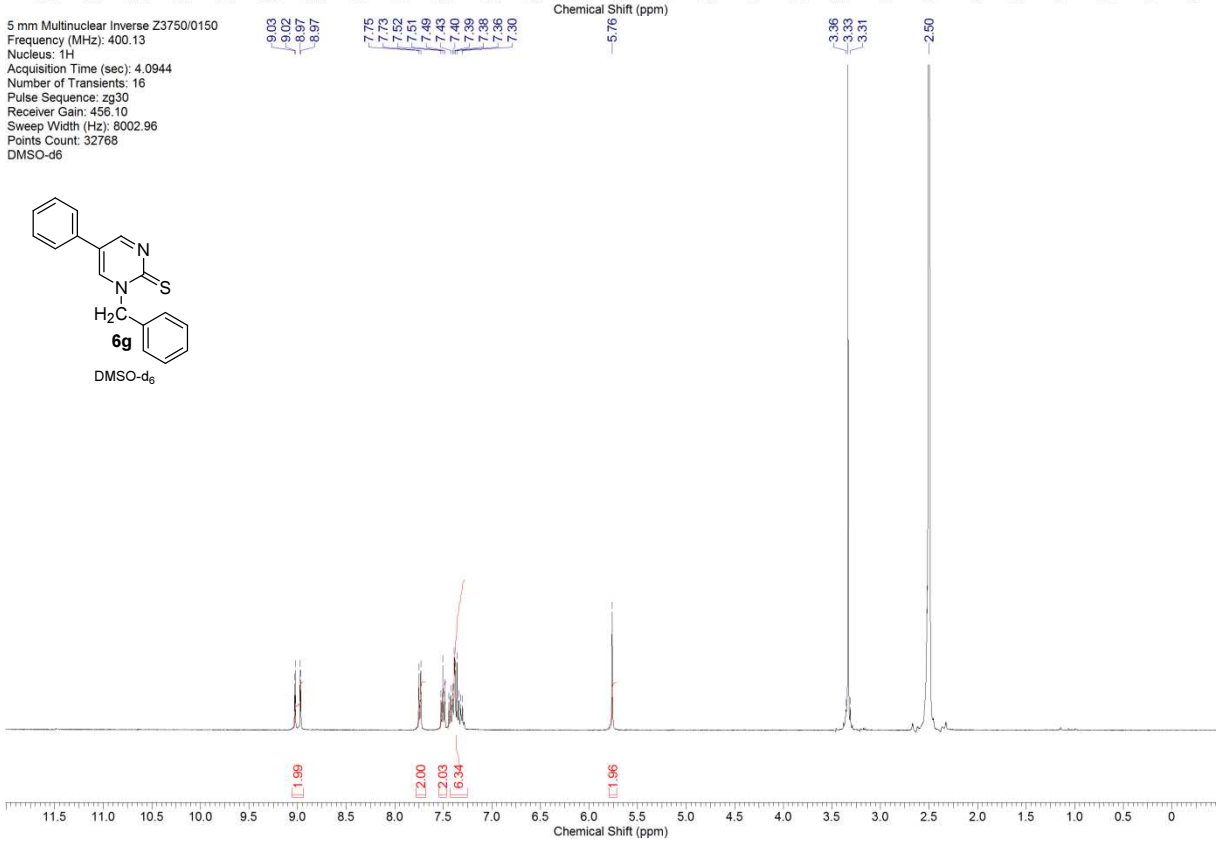

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 2048
Pulse Sequence: zgpg30
Receiver Gain: 11585.20
Sweep Width (Hz): 23979.35
Points Count: 16384
DMSO-d6
180.61
157.35
147.99
132.22
129.10
128.21
125.65
121.53
46.17
40.03
39.82
39.61
39.41
39.19
38.99
38.78
0.00
N
N
S
CH3
6d
DMSO-d6
Chemical Shift (ppm)

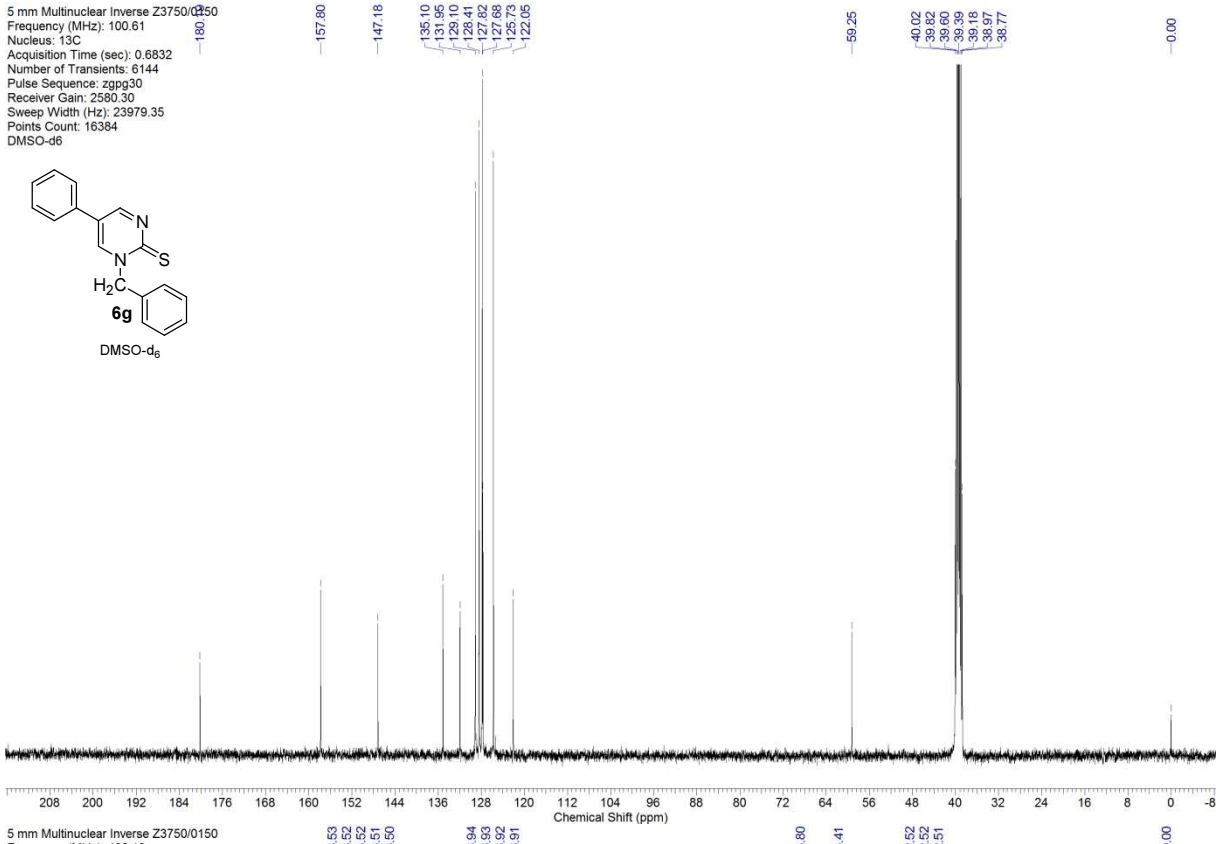

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 4.0944
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 362.00
Sweep Width (Hz): 8002.96
Points Count: 32768
DMSO-d6
8.89
8.88
8.85
8.85
7.67
7.67
7.66
7.65
7.07
7.07
7.05
7.05
3.88
3.80
3.34
2.50
H3CO
N
N
S
CH3
6e
DMSO-d6
2.03
2.03
2.00
3.07
Chemical Shift (ppm)

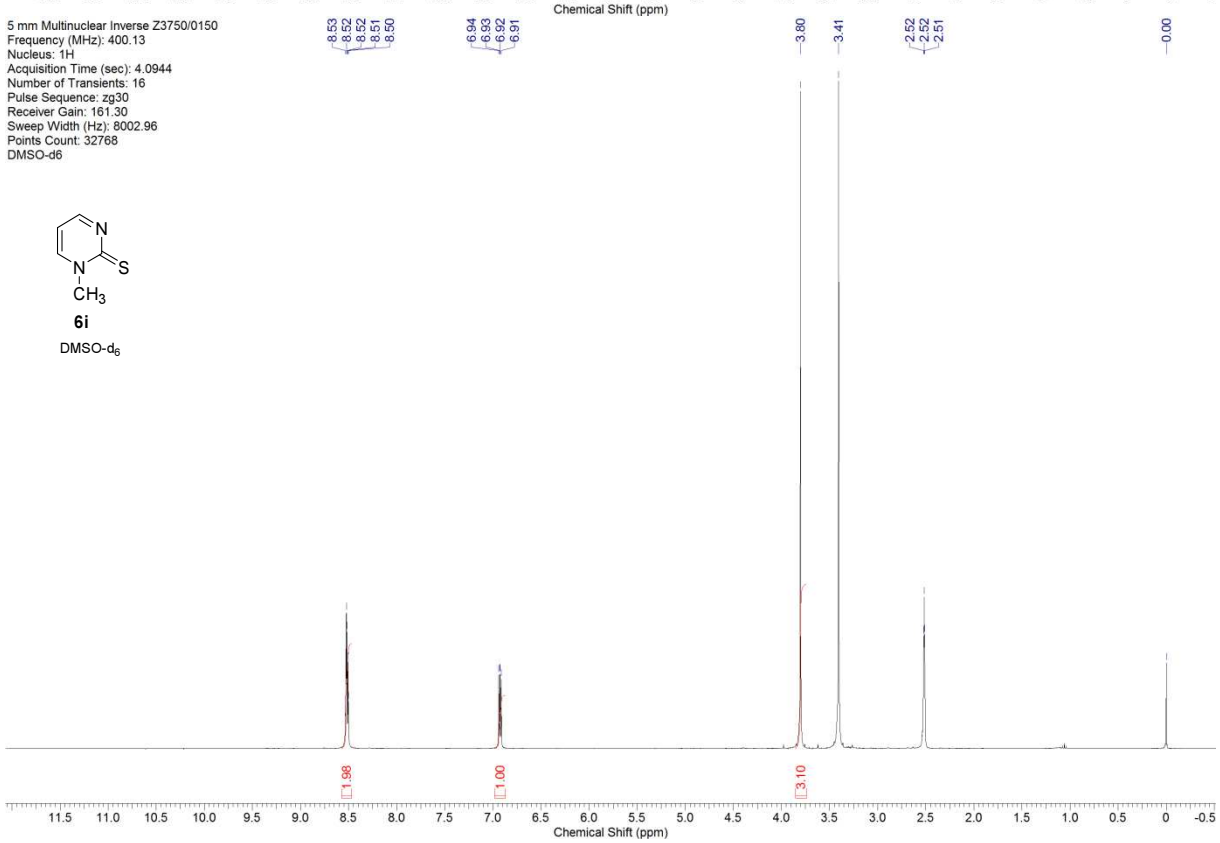

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 2048
Pulse Sequence: zgpg30
Receiver Gain: 5792.60
Sweep Width (Hz): 23979.35
Points Count: 16384
DMSO-d6
179.55
159.37
157.14
147.18
126.96
124.40
121.50
114.55
55.19
46.13
40.04
39.83
39.61
39.41
39.21
38.99
38.78
H3CO
N
N
S
CH3
6e
DMSO-d6
Chemical Shift (ppm)

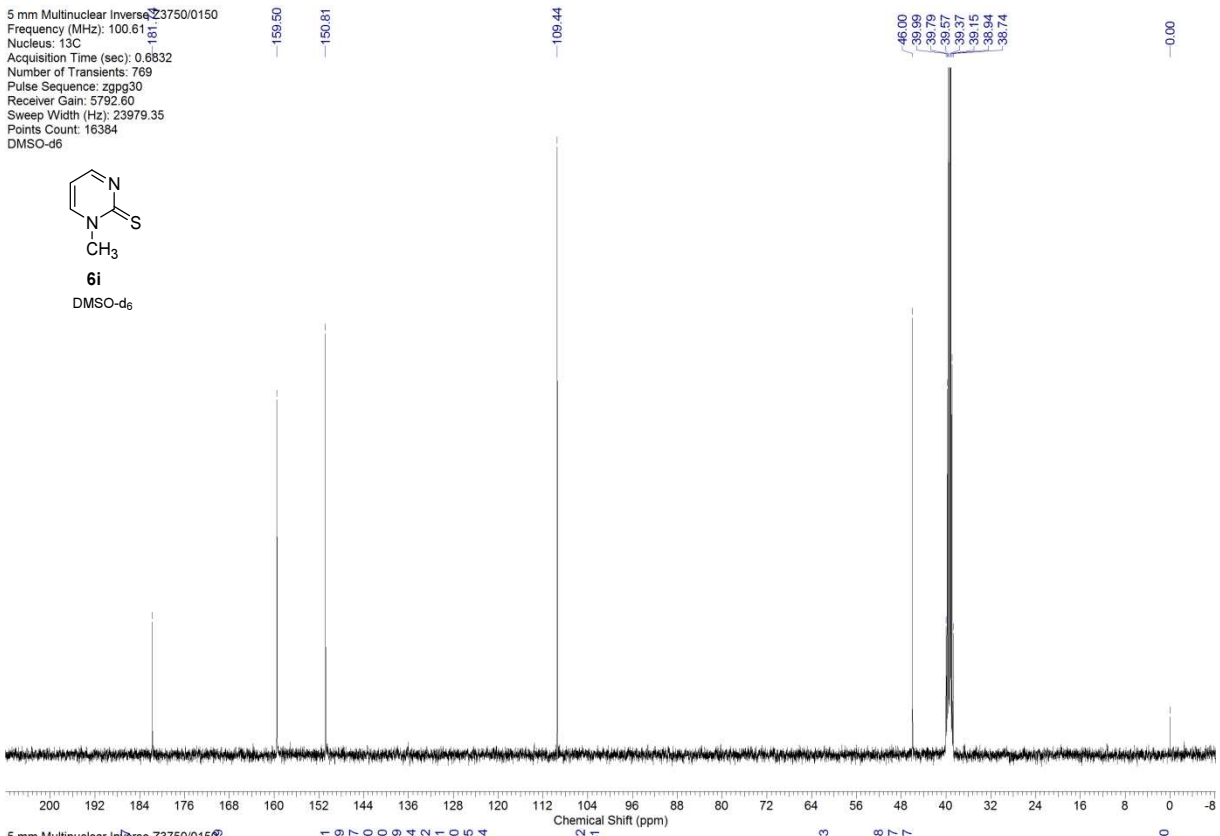

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 4.0944
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 456.10
Sweep Width (Hz): 8002.96
Points Count: 32768
DMSO-d6
8.91
7.80
7.79
7.78
7.77
7.77
7.76
7.38
7.36
7.34
3.88
3.33
3.29
2.50
2.50
2.50
F
N
N
S
CH3
6f
DMSO-d6
2.07
2.00
2.20
3.11
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 8192
Pulse Sequence: zgpg30
Receiver Gain: 5792.60
Sweep Width (Hz): 23979.35
Points Count: 16384
DMSO-d6
180.66
163.31
160.87
157.29
147.98
128.78
127.93
127.86
120.67
116.10
115.89
46.13
40.04
39.88
39.83
39.61
39.41
39.19
38.99
38.78
F
N
S
N
CH3
6f
DMSO-d6
208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 4.0944
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 456.10
Sweep Width (Hz): 8002.96
Points Count: 32768
DMSO-d6
9.03
9.02
8.97
8.97
7.75
7.73
7.52
7.51
7.49
7.43
7.40
7.39
7.38
7.36
7.30
5.76
3.36
3.33
3.31
2.50
N
S
N
H2C
6g
DMSO-d6
1.99
2.00
2.03
6.34
1.96
11.5 11.0 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
Chemical Shift (ppm)

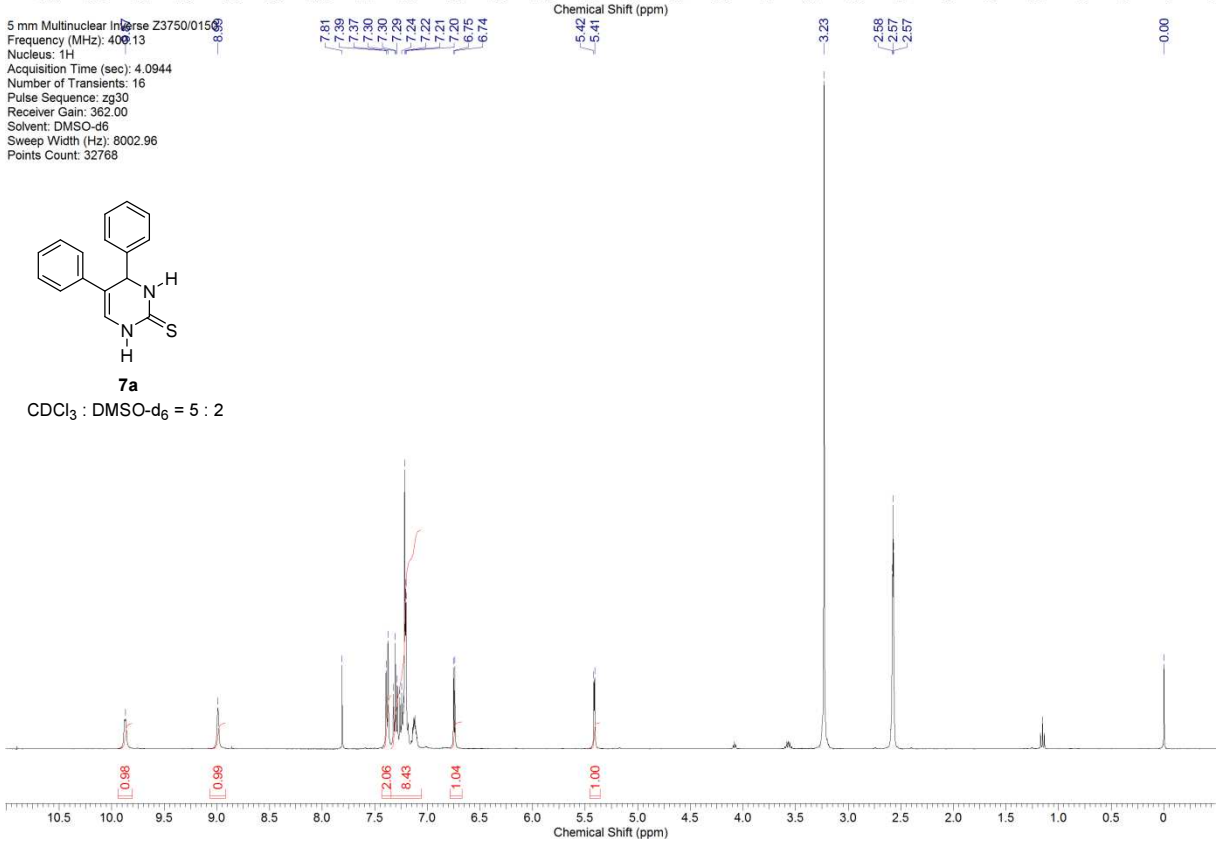

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 6144
Pulse Sequence: zgpg30
Receiver Gain: 2580.30
Sweep Width (Hz): 23979.35
Points Count: 16384
DMSO-d6
180.02
157.80
147.18
135.10
131.95
129.10
128.41
127.82
127.68
125.73
122.05
59.25
40.02
39.82
39.60
39.39
39.18
38.97
38.77
0.00
N
N
S
H2C
6g
DMSO-d6
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 4.0944
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 161.30
Sweep Width (Hz): 8002.96
Points Count: 32768
DMSO-d6
8.53
8.52
8.52
8.51
8.50
6.94
6.93
6.92
6.91
3.80
3.41
2.52
2.52
2.51
0.00
N
N
S
CH3
6i
DMSO-d6
1.98
1.00
3.10
Chemical Shift (ppm)



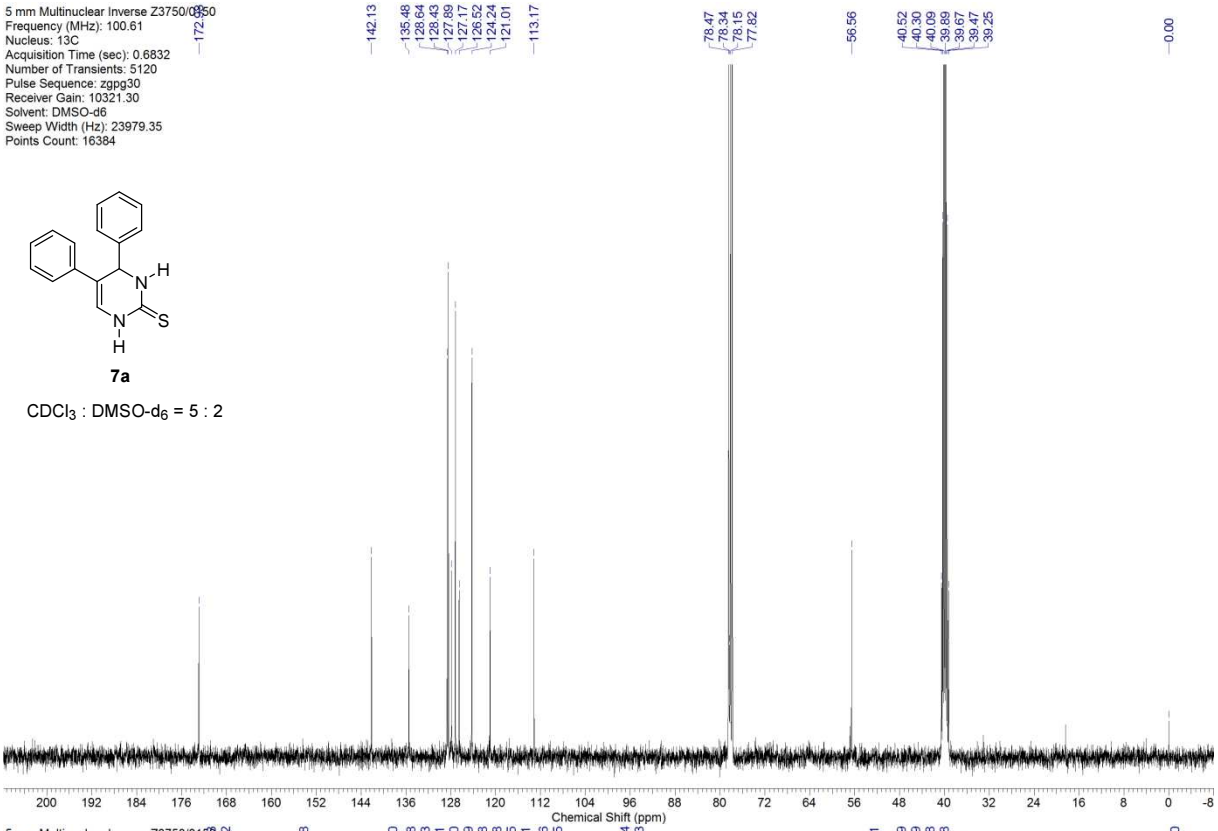
6i

DMSO-d_6



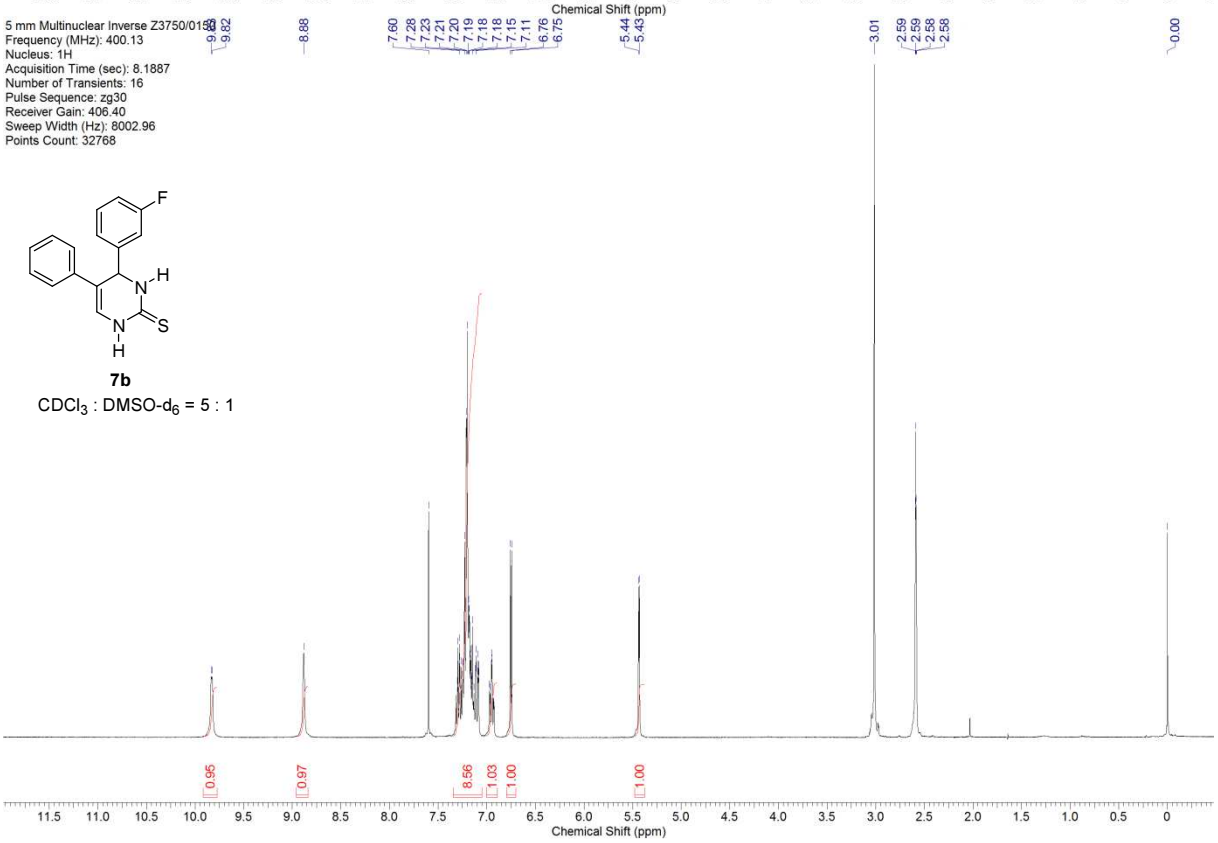
7a

$CDCl_3$: DMSO-d_6 = 5 : 2



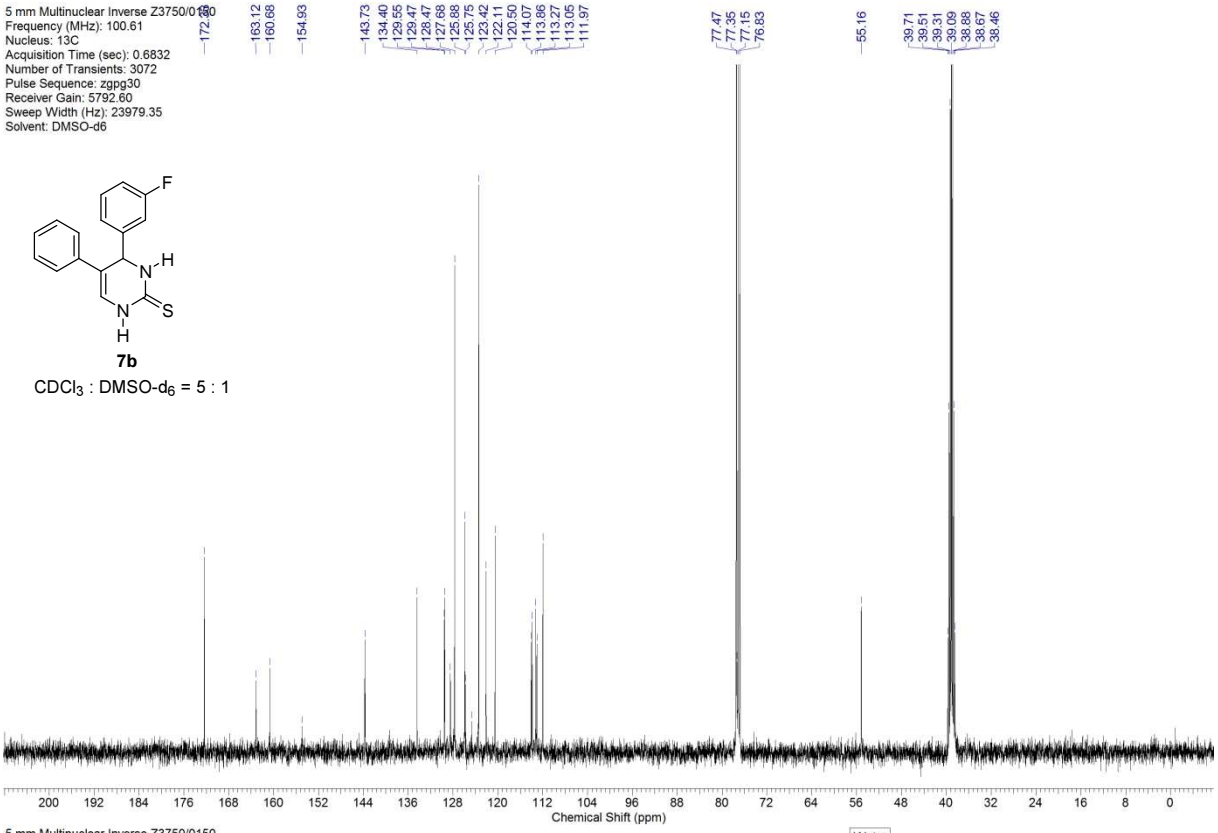
7a

$CDCl_3$: DMSO-d_6 = 5 : 2



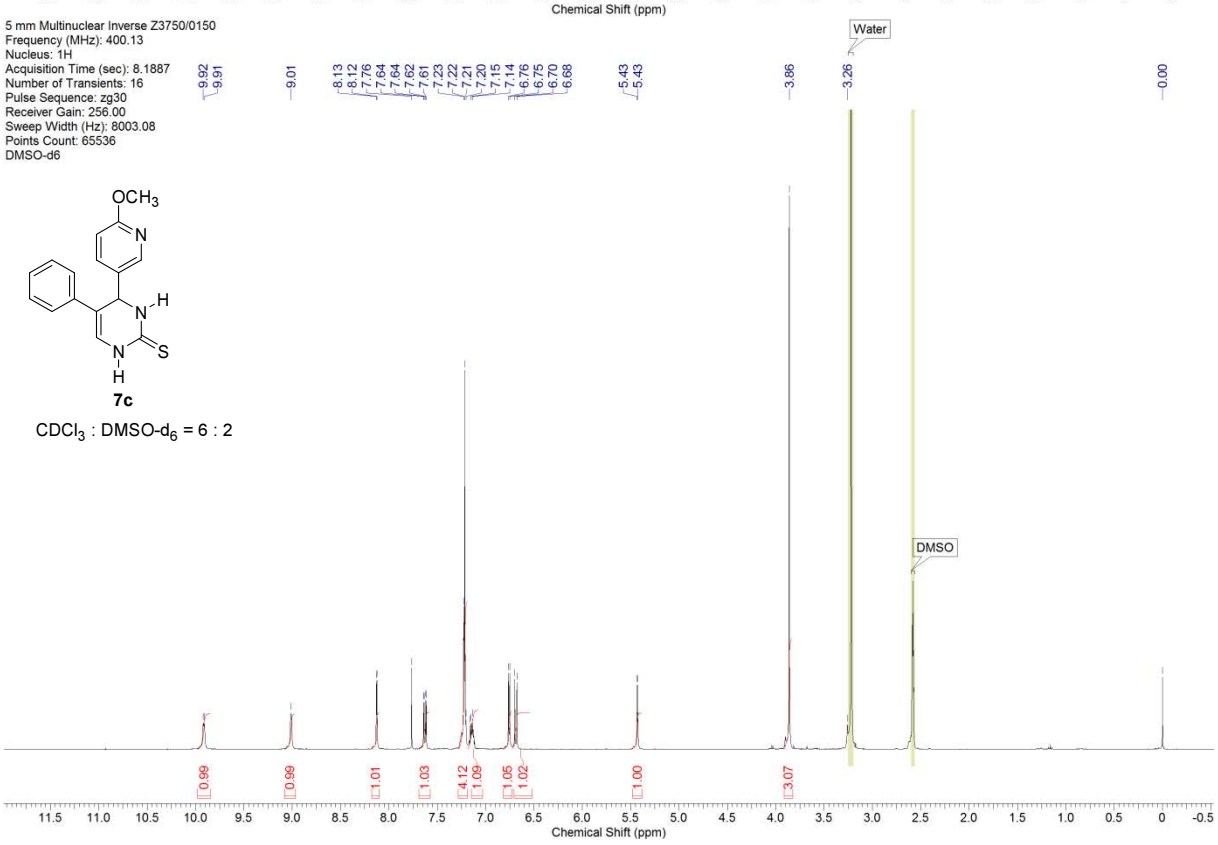
7b

$CDCl_3$: DMSO-d_6 = 5 : 1



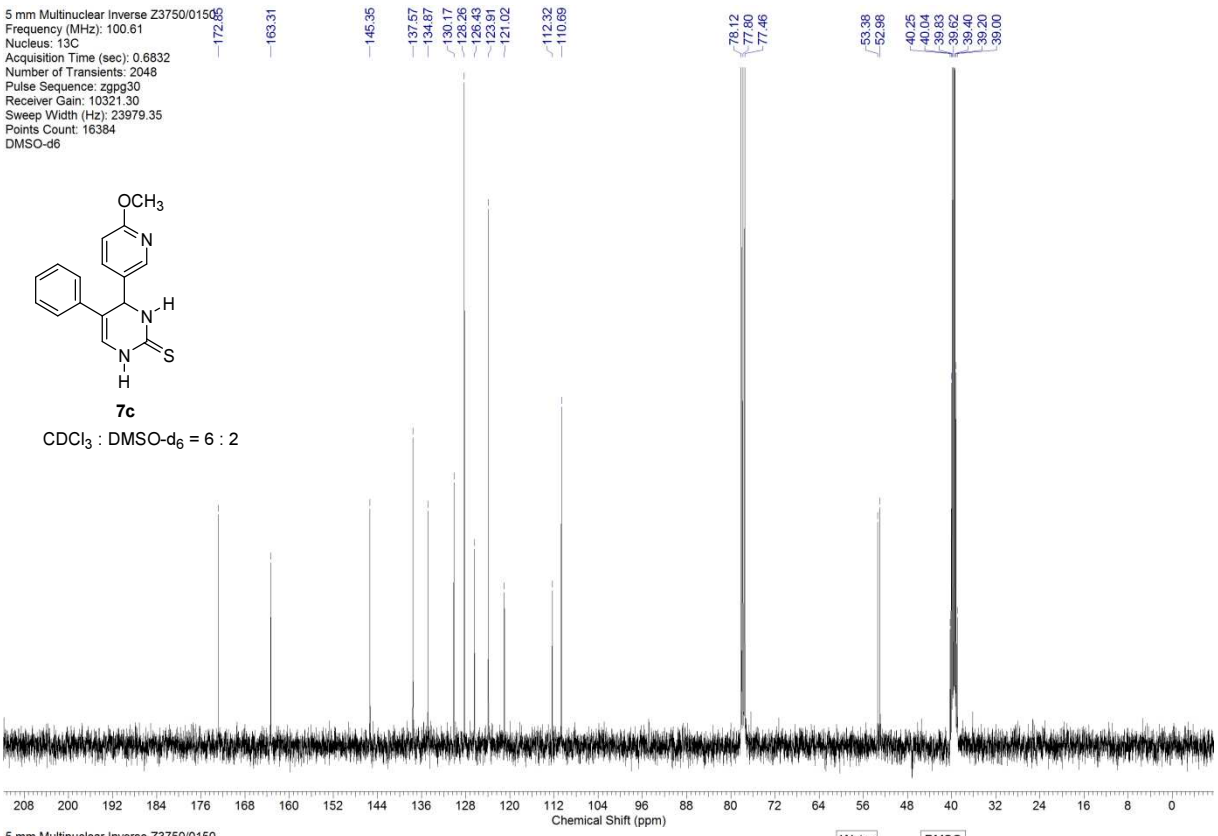
7b

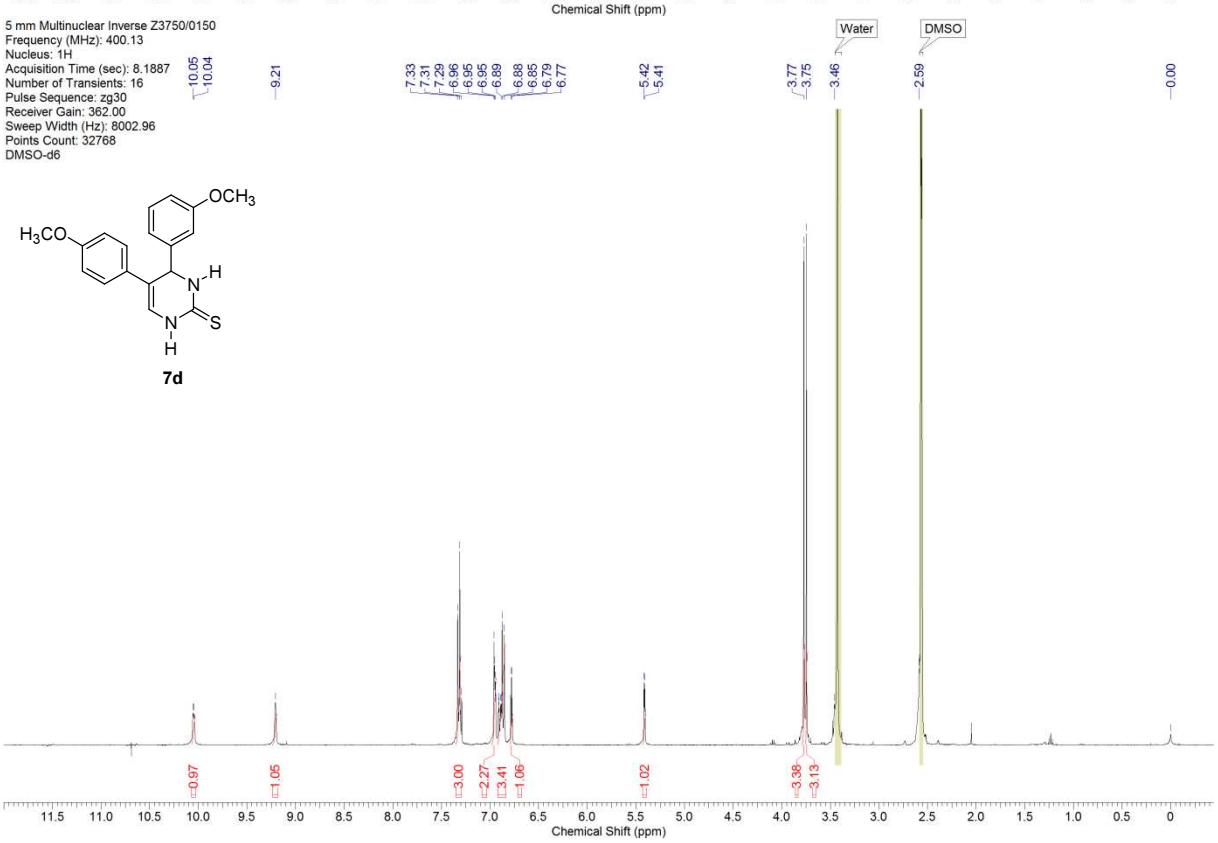
$CDCl_3$: DMSO-d_6 = 5 : 1

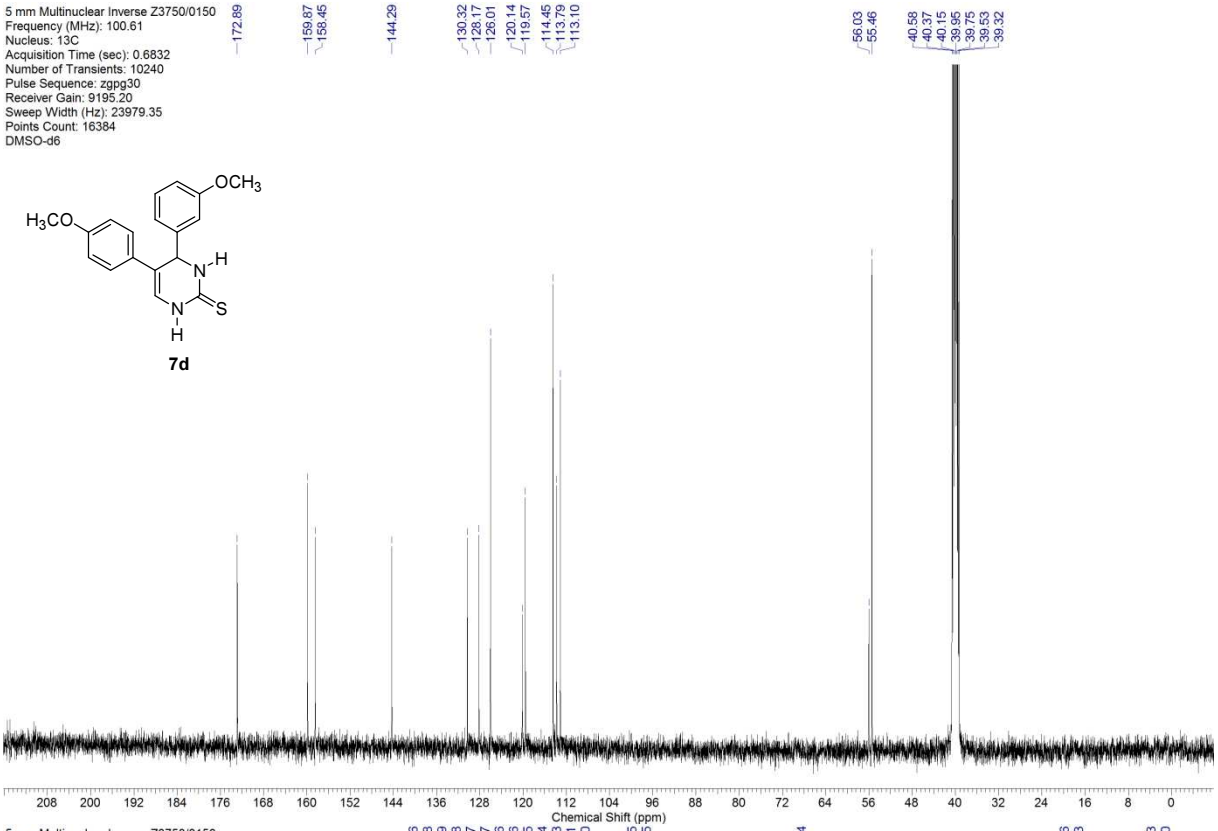


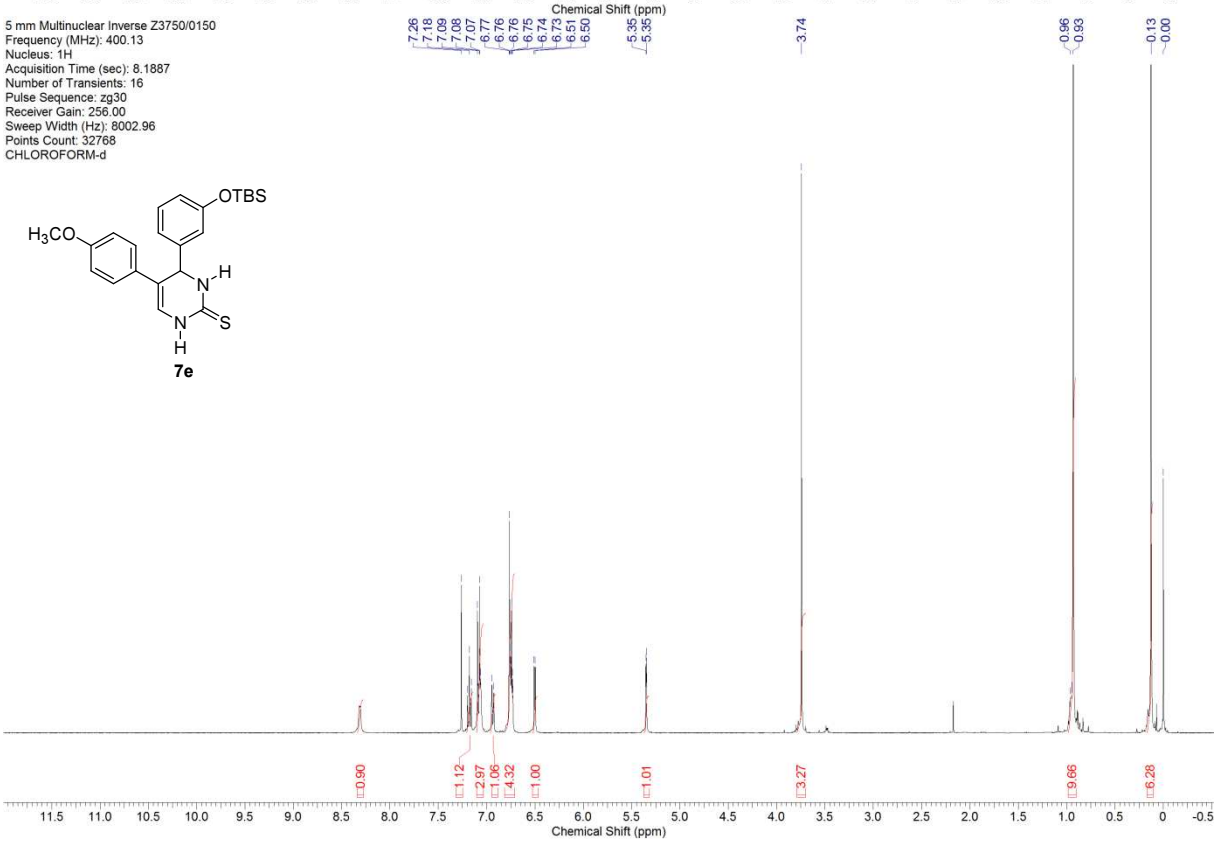
7c

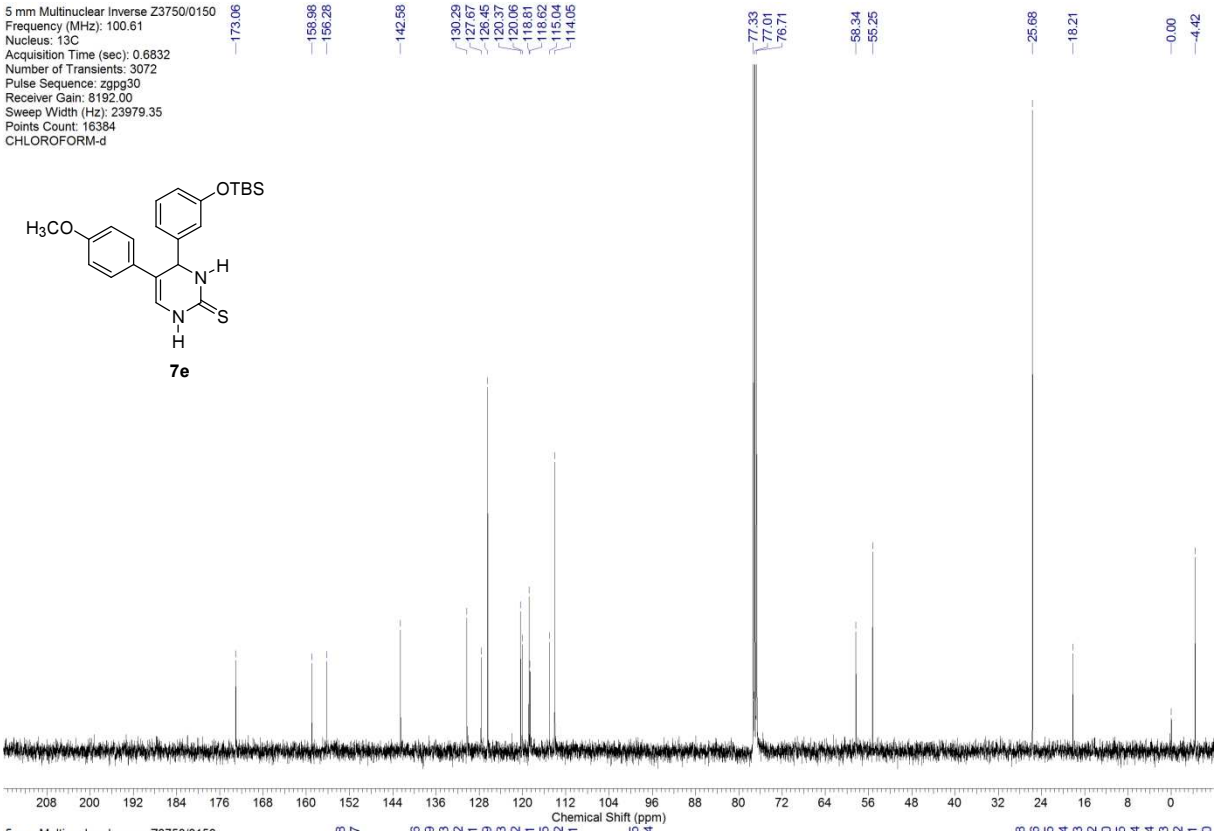
$CDCl_3$: DMSO-d_6 = 6 : 2


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 2048
Pulse Sequence: zgpg30
Receiver Gain: 10321.30
Sweep Width (Hz): 23979.35
Points Count: 16384
DMSO-d6
172.85
163.31
145.35
137.57
134.87
130.17
128.26
126.43
123.91
121.02
112.32
110.69
78.12
77.80
77.46
53.38
52.98
40.25
40.04
39.83
39.62
39.40
39.20
39.00
OCH3
N
N
H
N
H
S
7c
CDCl3 : DMSO-d6 = 6 : 2
Chemical Shift (ppm)

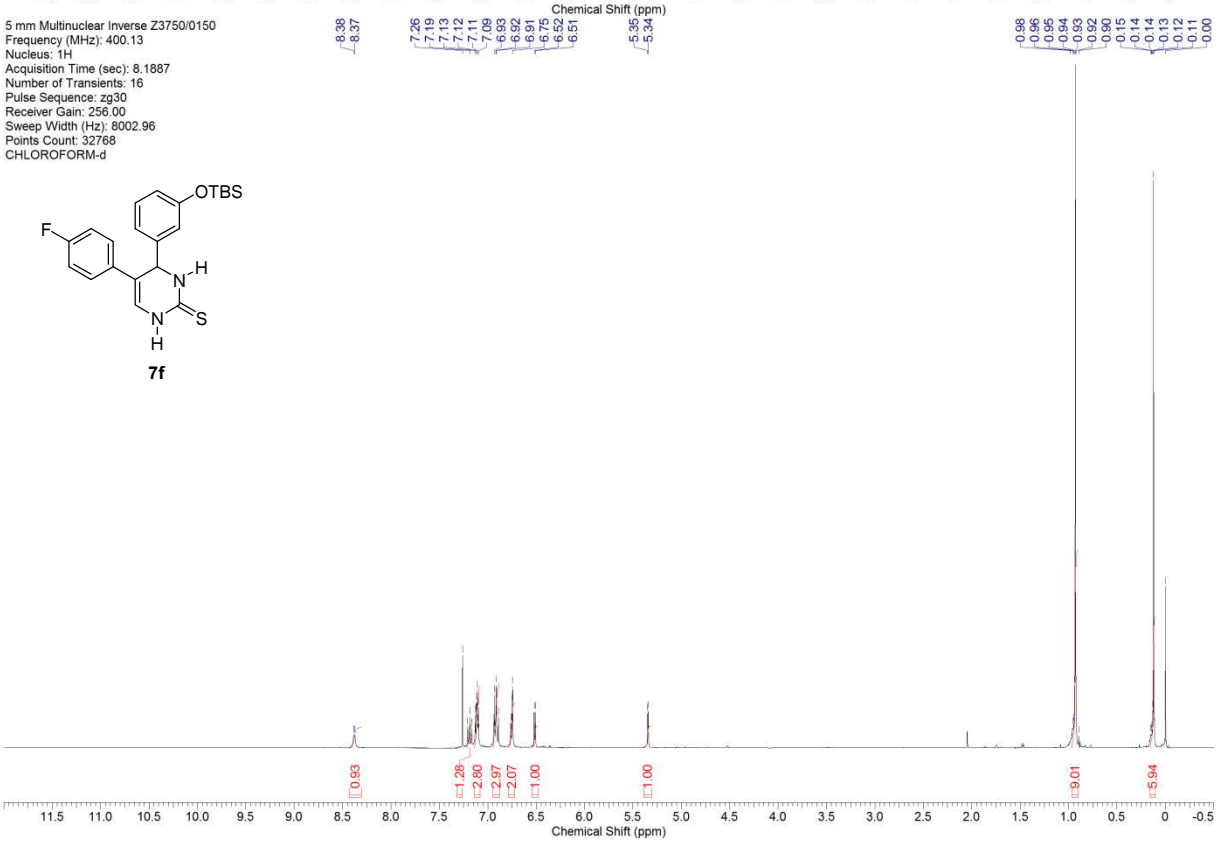

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 362.00
Sweep Width (Hz): 8002.96
Points Count: 32768
DMSO-d6
Water
DMSO
10.05
10.04
9.21
7.33
7.31
7.29
6.96
6.95
6.95
6.89
6.88
6.85
6.79
6.77
5.42
5.41
3.77
3.75
3.46
2.59
0.00
OCH3
H3CO
N
H
N
H
S
7d
0.97
1.05
3.00
2.27
3.41
1.06
1.02
3.38
3.13
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 10240
Pulse Sequence: zgpg30
Receiver Gain: 9195.20
Sweep Width (Hz): 23979.35
Points Count: 16384
DMSO-d6
172.89
159.87
158.45
144.29
130.32
128.17
126.01
120.14
119.57
114.45
113.79
113.10
56.03
55.46
40.58
40.37
40.15
39.95
39.75
39.53
39.32
OCH3
H3CO
N
H
S
7d
Chemical Shift (ppm)

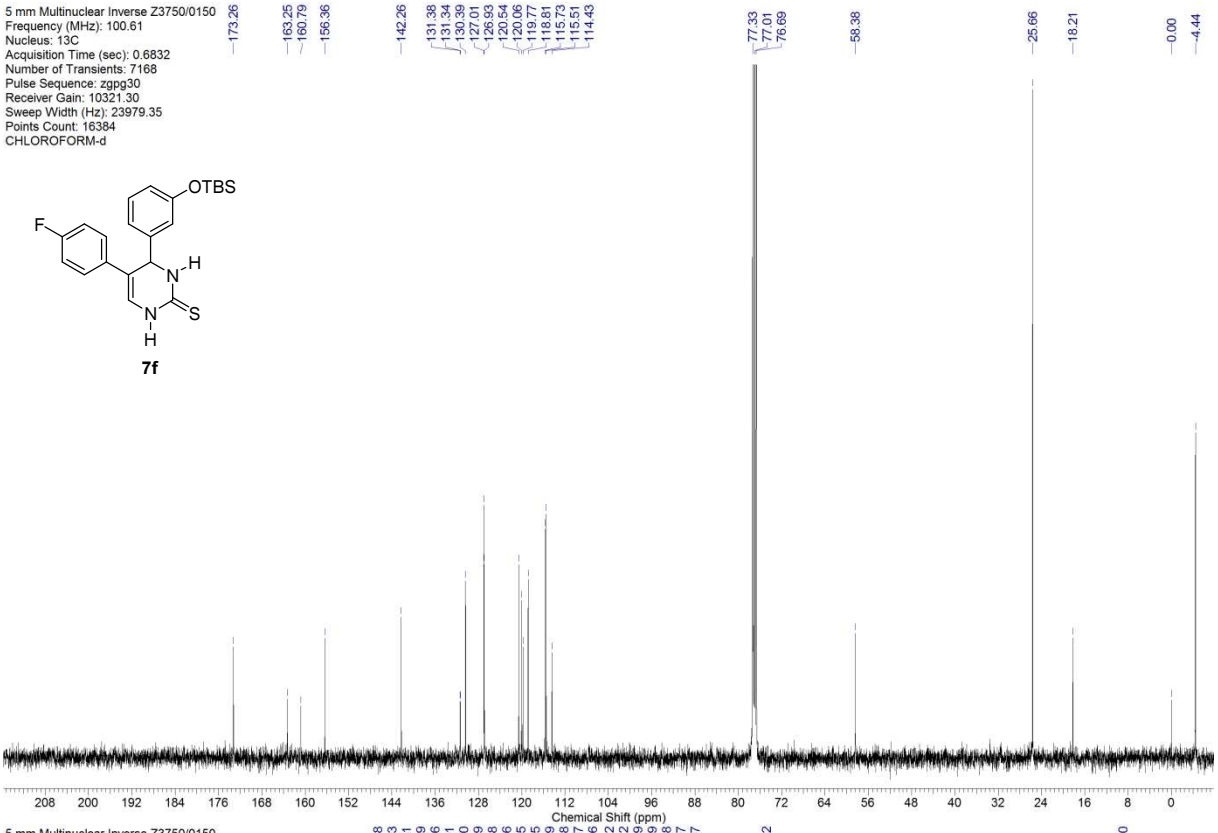

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 256.00
Sweep Width (Hz): 8002.96
Points Count: 32768
CHLOROFORM-d
7.26
7.18
7.09
7.08
7.07
6.77
6.76
6.76
6.75
6.74
6.73
6.51
6.50
5.35
5.35
3.74
0.96
0.93
0.13
0.00
OTBS
H3CO
N
H
S
7e
0.90
1.12
2.97
1.06
4.32
1.00
1.01
3.27
9.66
6.28
Chemical Shift (ppm)

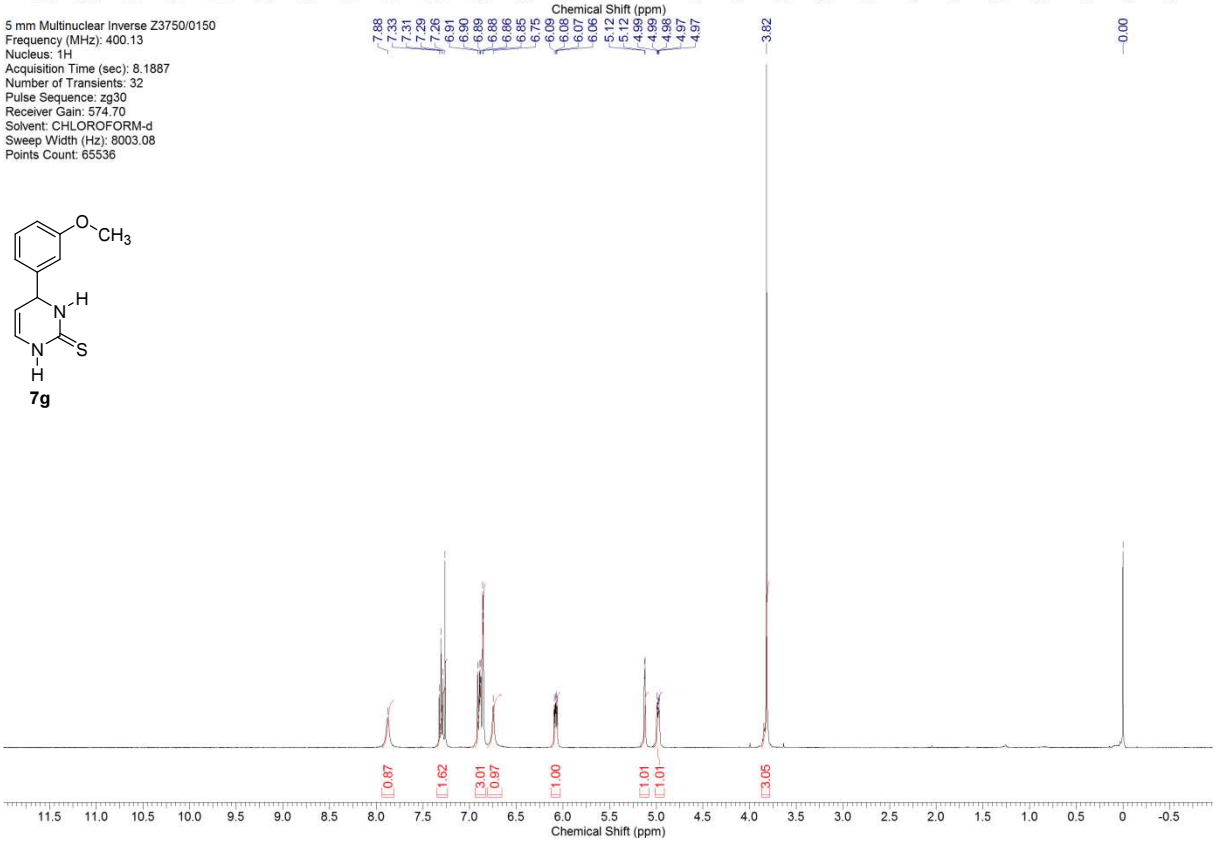

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 3072
Pulse Sequence: zgpg30
Receiver Gain: 8192.00
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
173.06
158.98
156.28
142.58
130.29
127.67
126.45
120.37
120.06
118.81
118.62
115.04
114.05
77.33
77.01
76.71
58.34
55.25
25.68
18.21
0.00
-4.42
OTBS
H3CO
N
H
N
H
S
Chemical Shift (ppm)

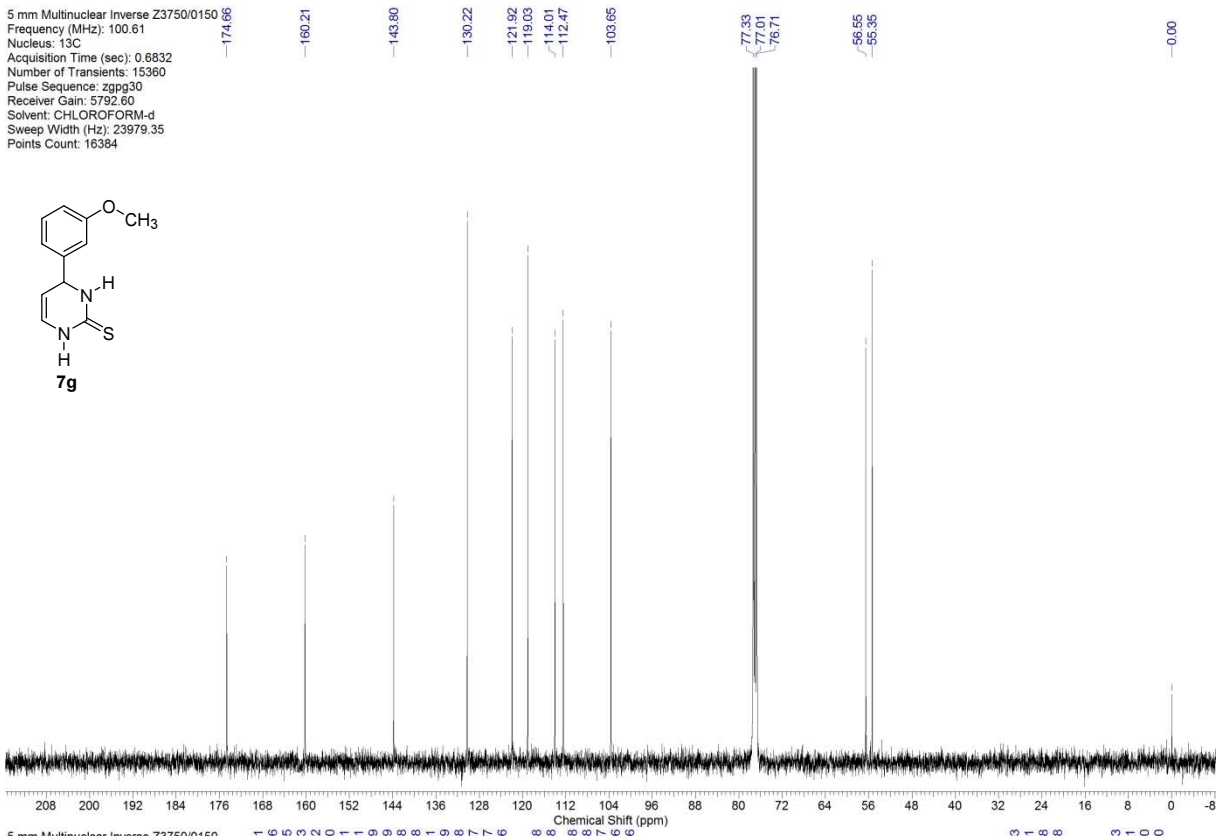
7e

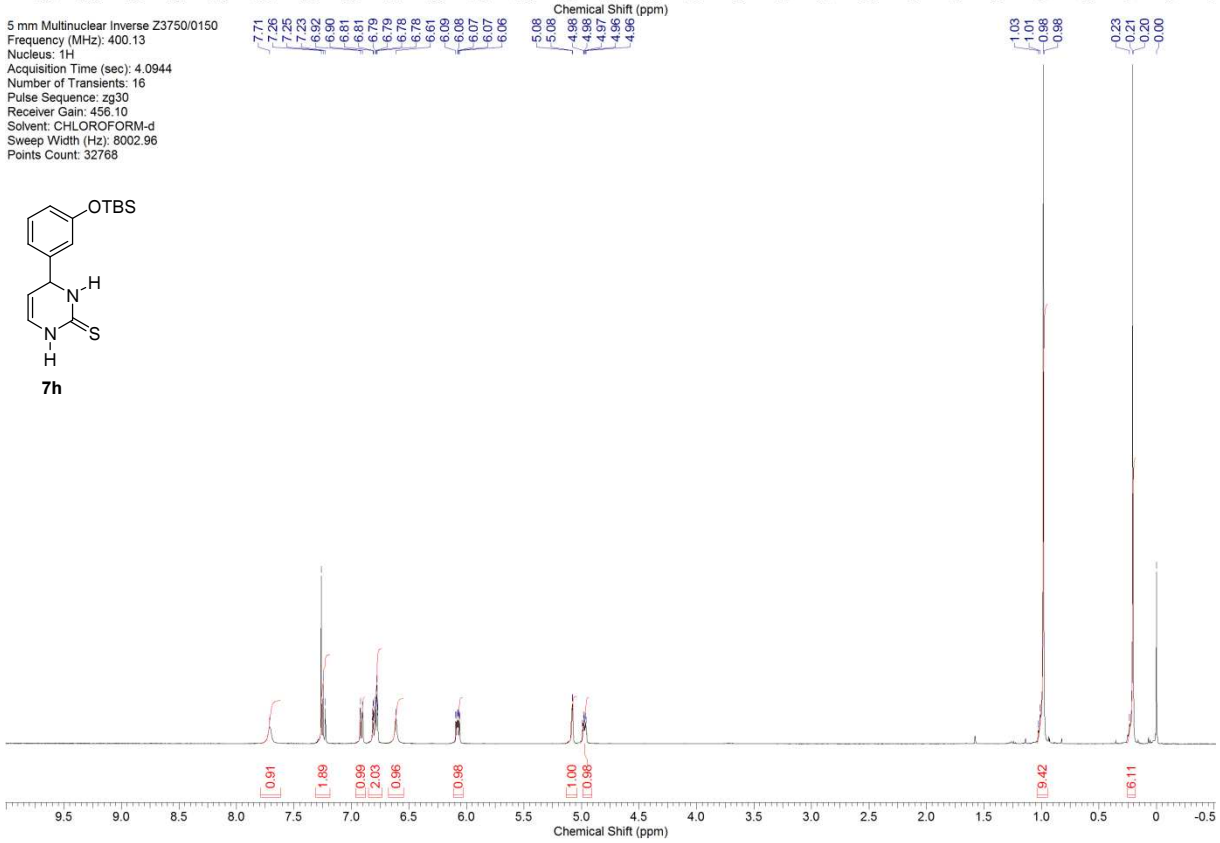

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 256.00
Sweep Width (Hz): 8002.96
Points Count: 32768
CHLOROFORM-d
8.38
8.37
7.26
7.19
7.13
7.12
7.11
7.09
6.93
6.92
6.91
6.75
6.52
6.51
5.35
5.34
0.98
0.96
0.95
0.94
0.93
0.92
0.90
0.15
0.14
0.14
0.13
0.12
0.11
0.00
OTBS
F
N
H
N
H
S
0.93
1.28
2.80
2.97
2.07
1.00
1.00
9.01
5.94
Chemical Shift (ppm)

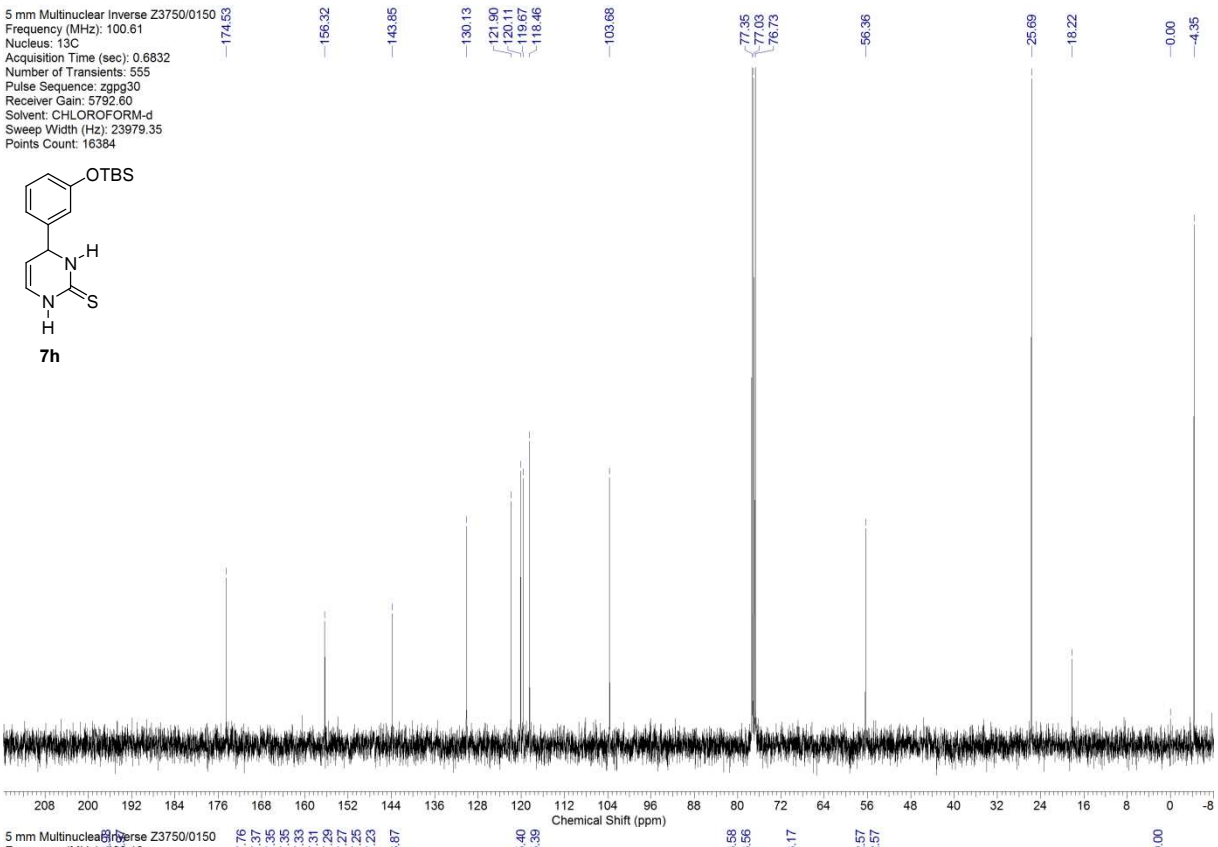
7f

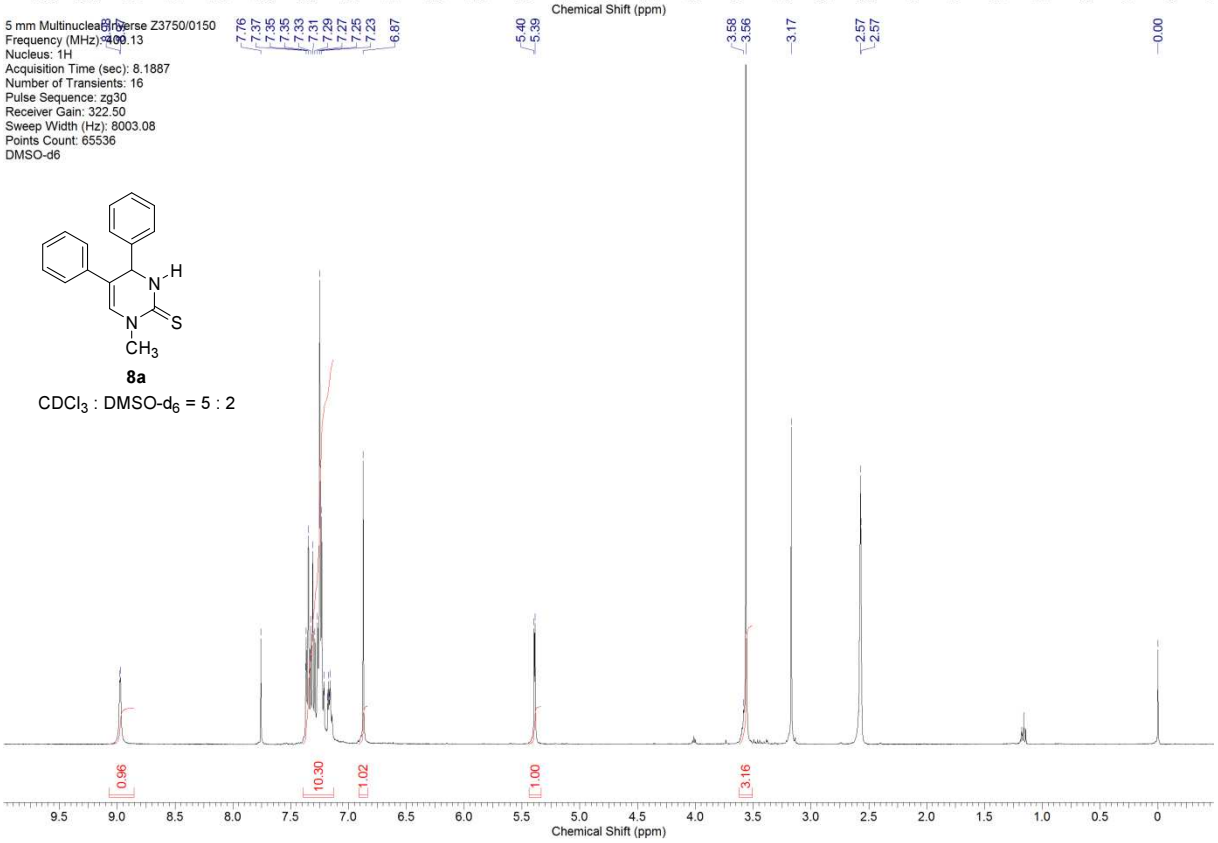

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 7168
Pulse Sequence: zgpg30
Receiver Gain: 10321.30
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
OTBS
F
N
H
S
7f
Chemical Shift (ppm)

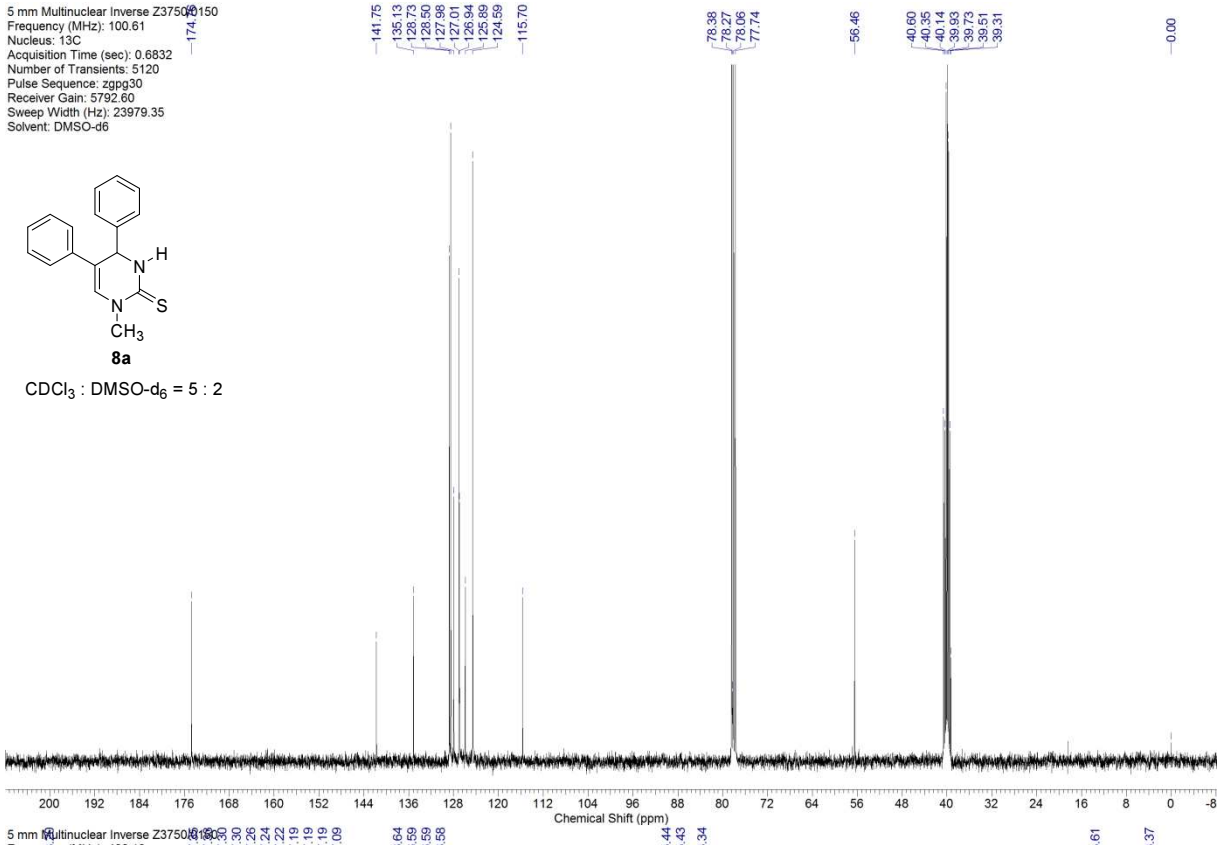

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 32
Pulse Sequence: zg30
Receiver Gain: 574.70
Solvent: CHLOROFORM-d
Sweep Width (Hz): 8003.08
Points Count: 65536
O
CH3
N
H
S
7g
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 15360
Pulse Sequence: zgpg30
Receiver Gain: 5792.60
Solvent: CHLOROFORM-d
Sweep Width (Hz): 23979.35
Points Count: 16384
174.66
160.21
143.80
130.22
121.92
119.03
114.01
112.47
103.65
77.33
77.01
76.71
56.55
55.35
0.00
O
CH3
N
H
N
H
S
7g
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 4.0944
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 456.10
Solvent: CHLOROFORM-d
Sweep Width (Hz): 8002.96
Points Count: 32768
7.71
7.26
7.25
7.23
6.92
6.90
6.81
6.81
6.79
6.79
6.78
6.78
6.61
6.09
6.08
6.07
6.07
6.06
5.08
5.08
4.98
4.98
4.97
4.96
4.96
1.03
1.01
0.98
0.98
0.23
0.21
0.20
0.00
OTBS
N
H
N
H
S
7h
0.91
1.89
0.99
2.03
0.96
0.98
1.00
0.98
9.42
6.11
Chemical Shift (ppm)

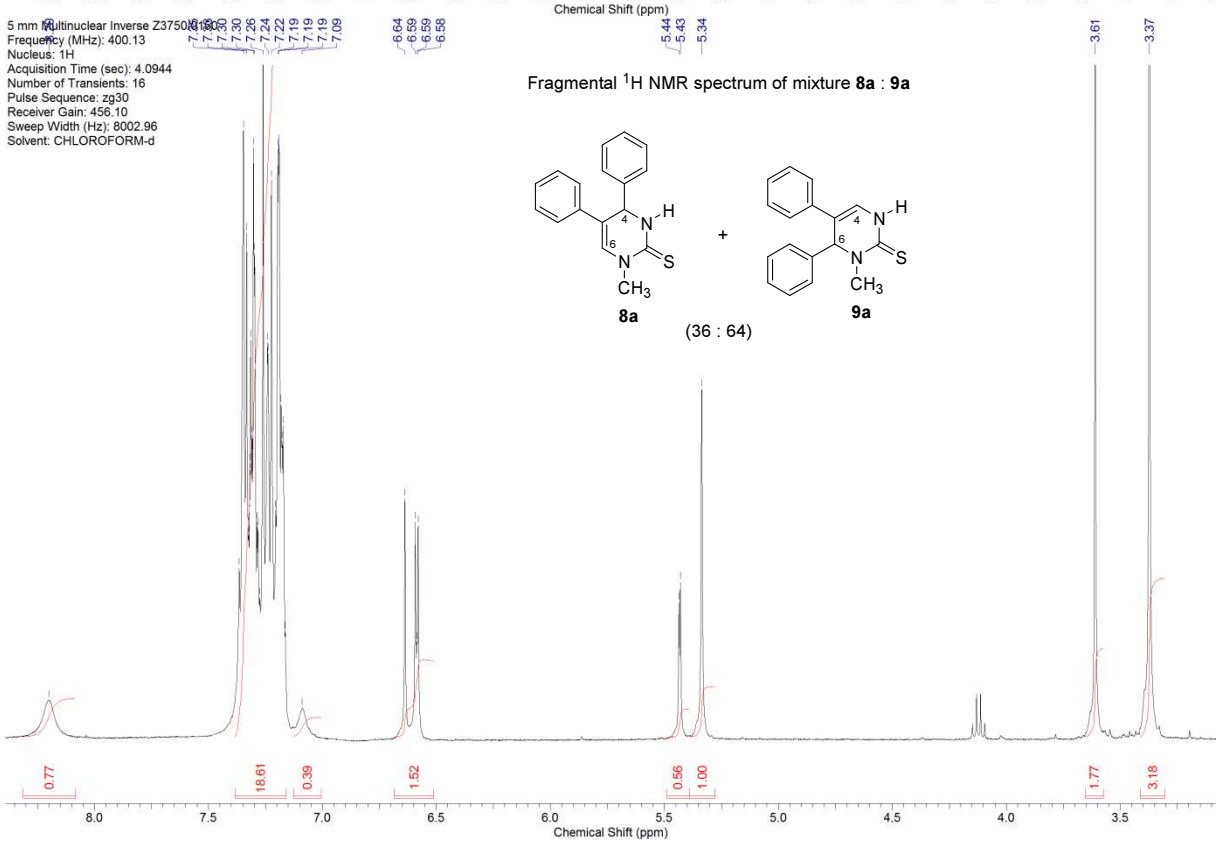

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 555
Pulse Sequence: zgpg30
Receiver Gain: 5792.60
Solvent: CHLOROFORM-d
Sweep Width (Hz): 23979.35
Points Count: 16384
174.53
156.32
143.85
130.13
121.90
120.11
119.67
118.46
103.68
77.35
77.03
76.73
56.36
25.69
18.22
0.00
-4.35
OTBS
H
N
N
H
S
7h
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 322.50
Sweep Width (Hz): 8003.08
Points Count: 65536
DMSO-d6
7.76
7.37
7.35
7.35
7.33
7.31
7.29
7.27
7.25
7.23
6.87
5.40
5.39
3.58
3.56
3.17
2.57
2.57
0.00
H
N
N
S
CH3
8a
CDCl3 : DMSO-d6 = 5 : 2
0.96
10.30
1.02
1.00
3.16
Chemical Shift (ppm)



8a

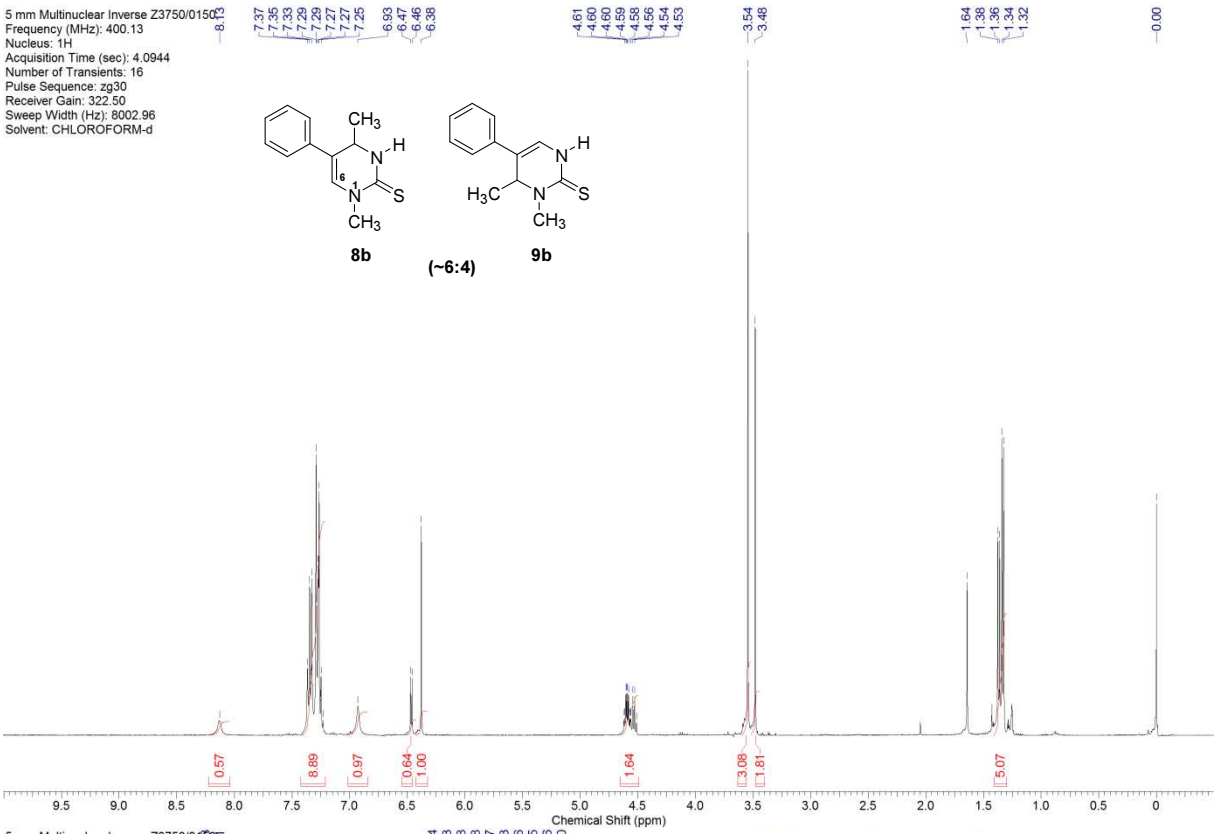
$CDCl_3$: DMSO-d_6 = 5 : 2



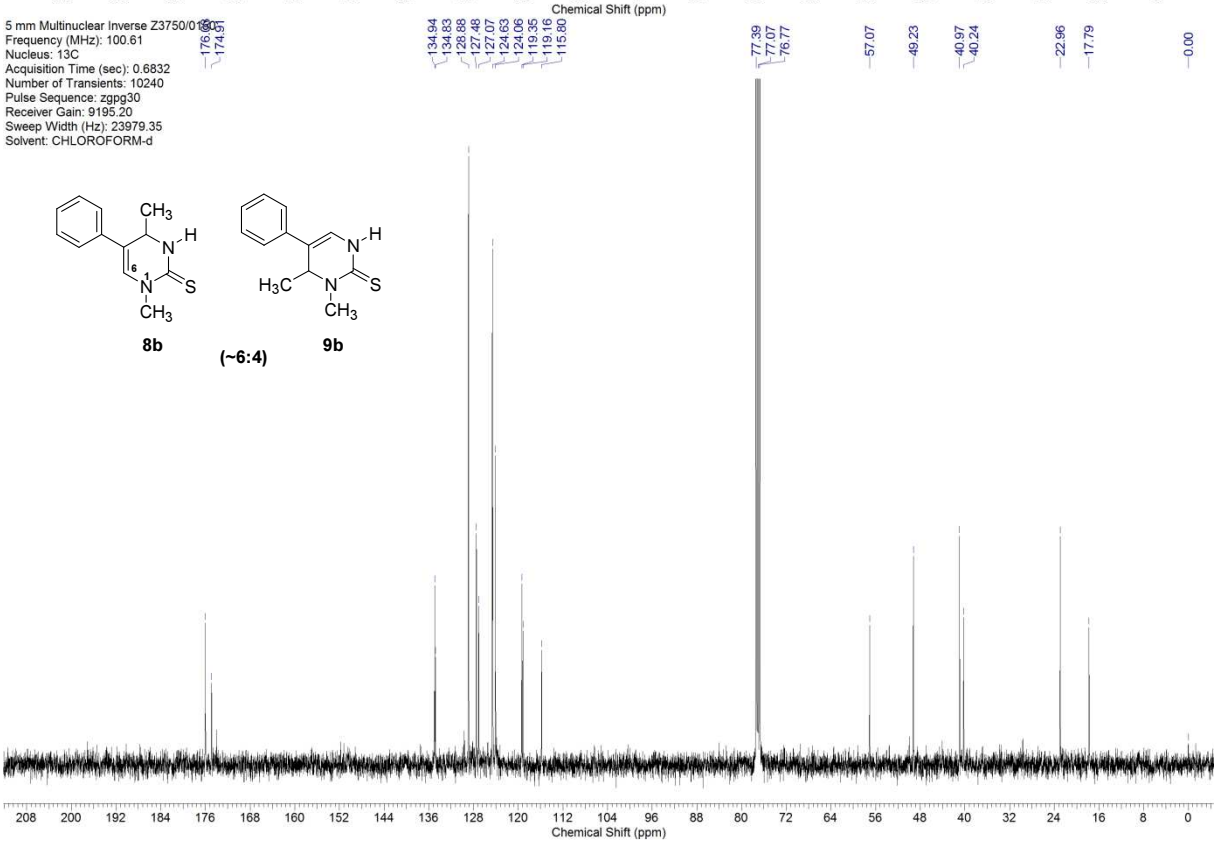
Fragmental ^{1}H NMR spectrum of mixture **8a** : **9a**

8a + **9a**

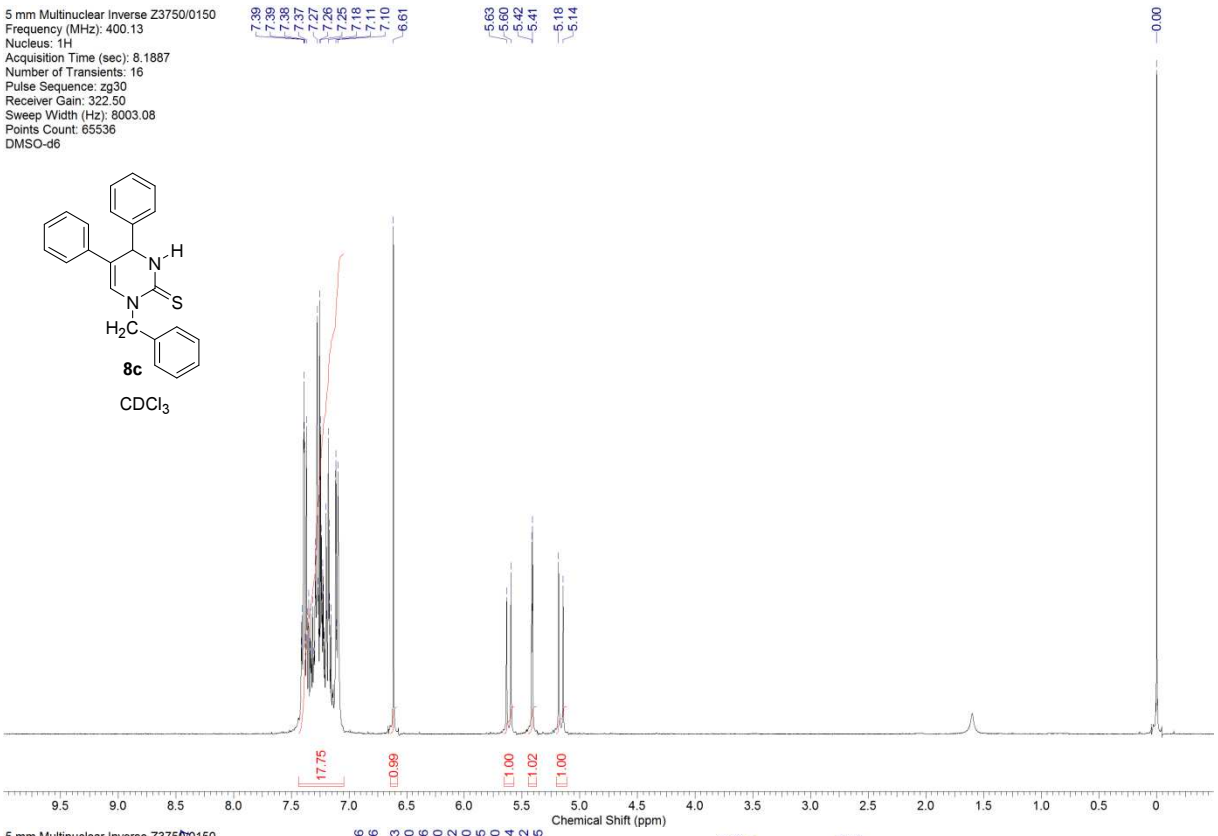
(36 : 64)

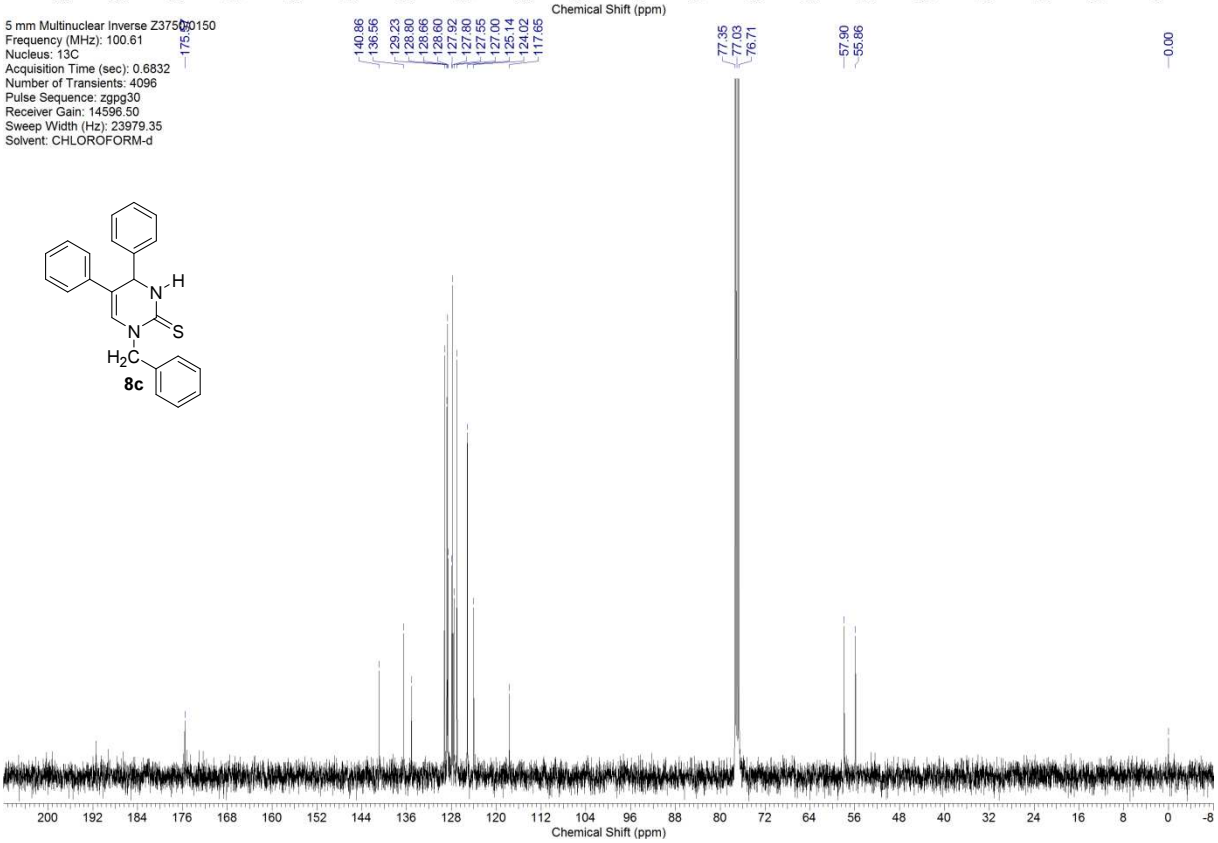


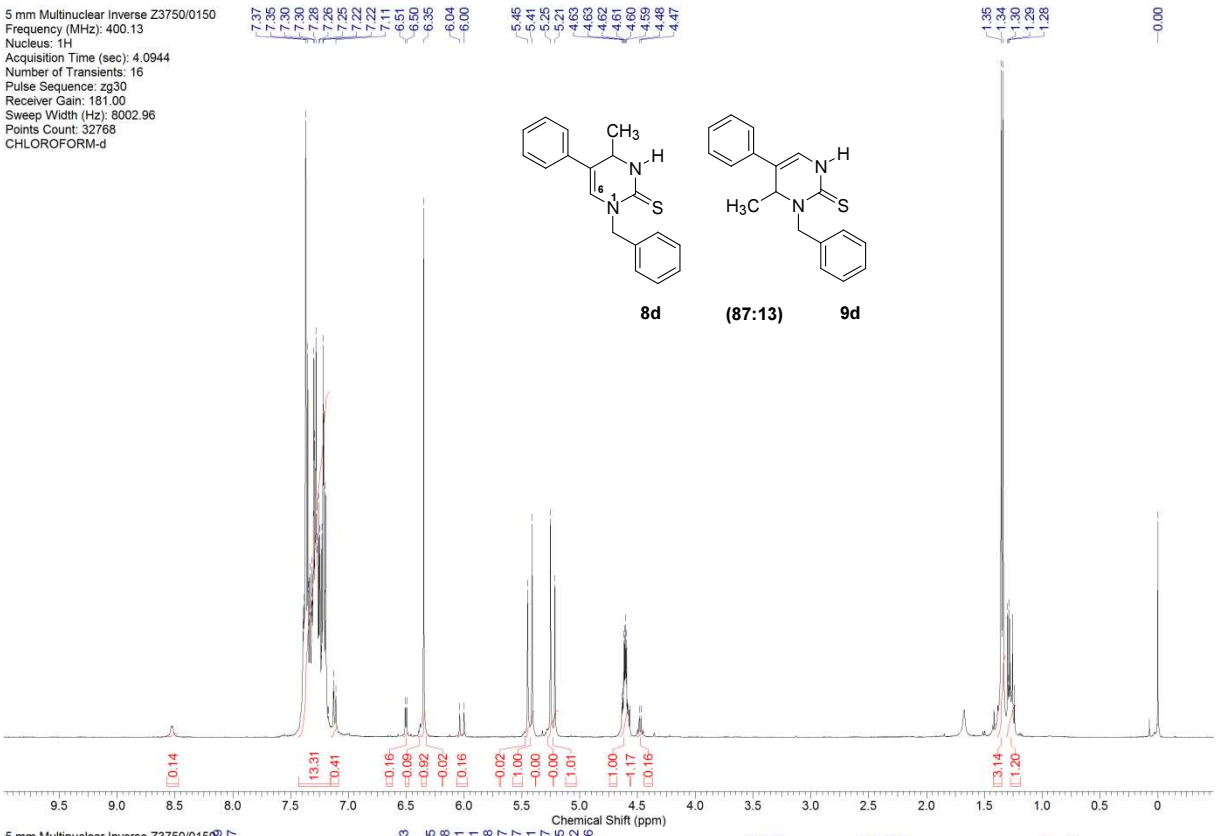
8b **(~6:4)** **9b**

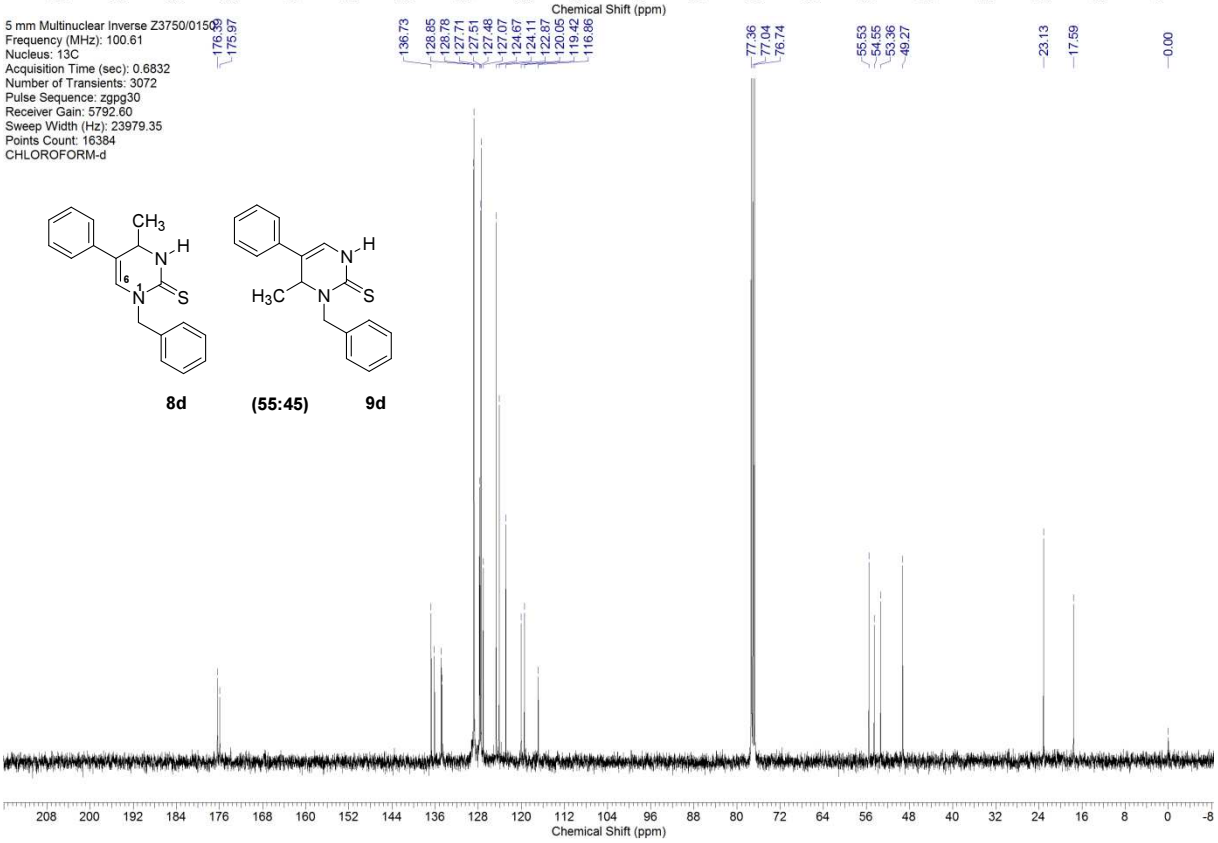


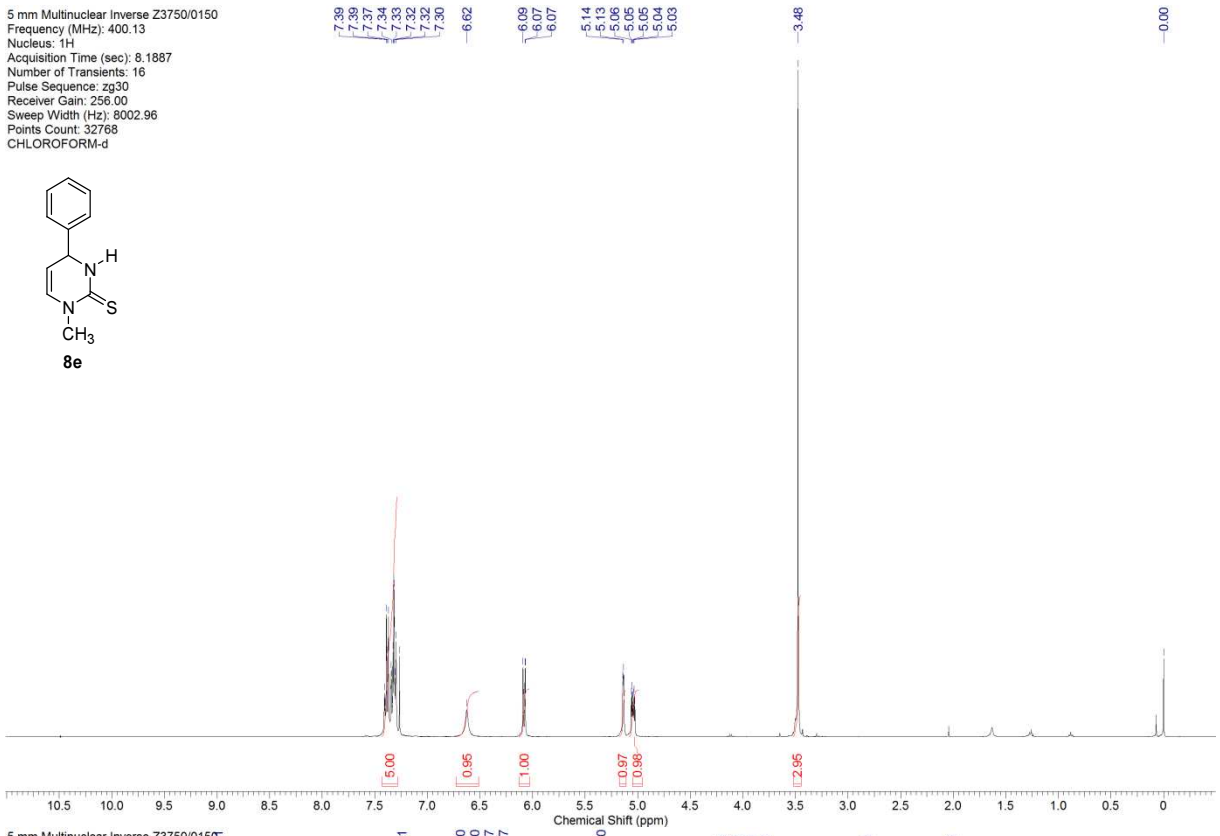
8b **(~6:4)** **9b**

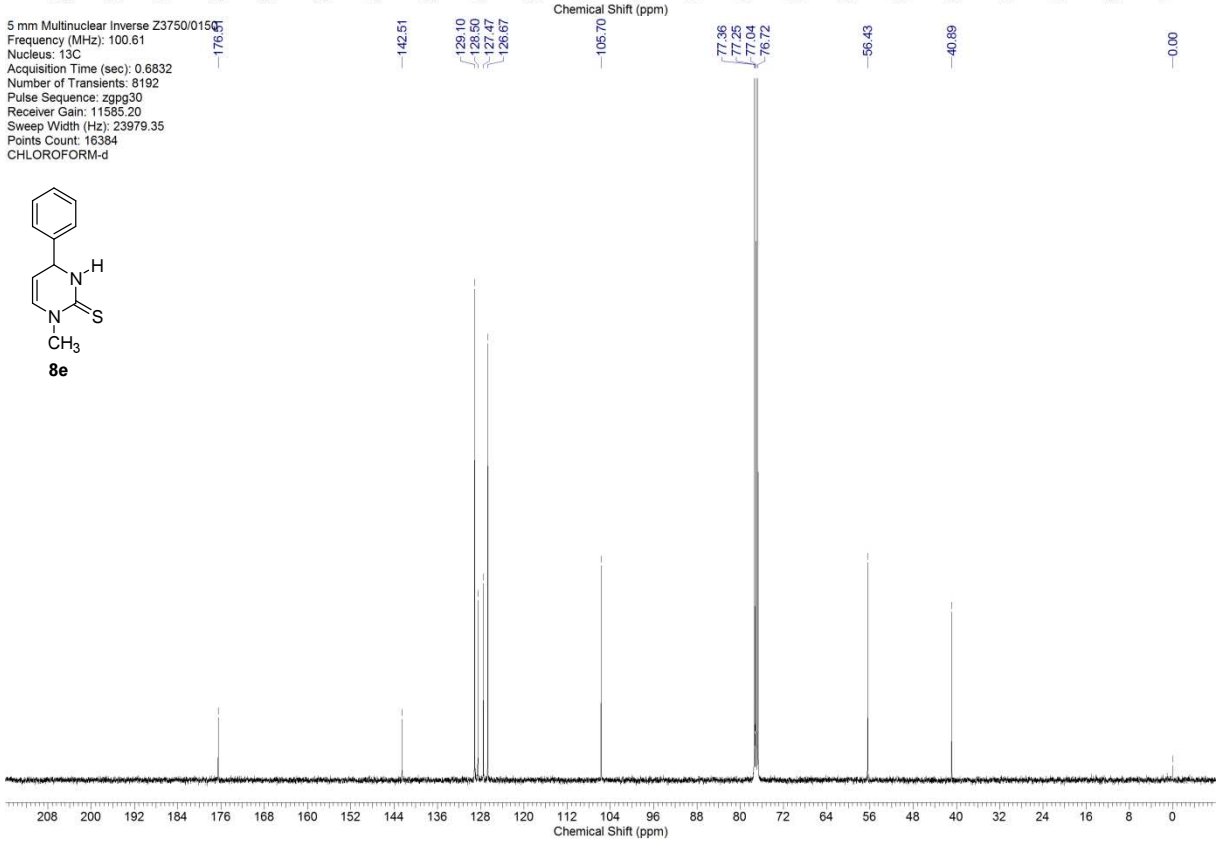

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 322.50
Sweep Width (Hz): 8003.08
Points Count: 65536
DMSO-d6
7.39
7.39
7.38
7.37
7.27
7.26
7.25
7.18
7.11
7.10
6.61
5.63
5.60
5.42
5.41
5.18
5.14
0.00
8c
CDCl3
17.75
0.99
1.00
1.02
1.00
Chemical Shift (ppm)

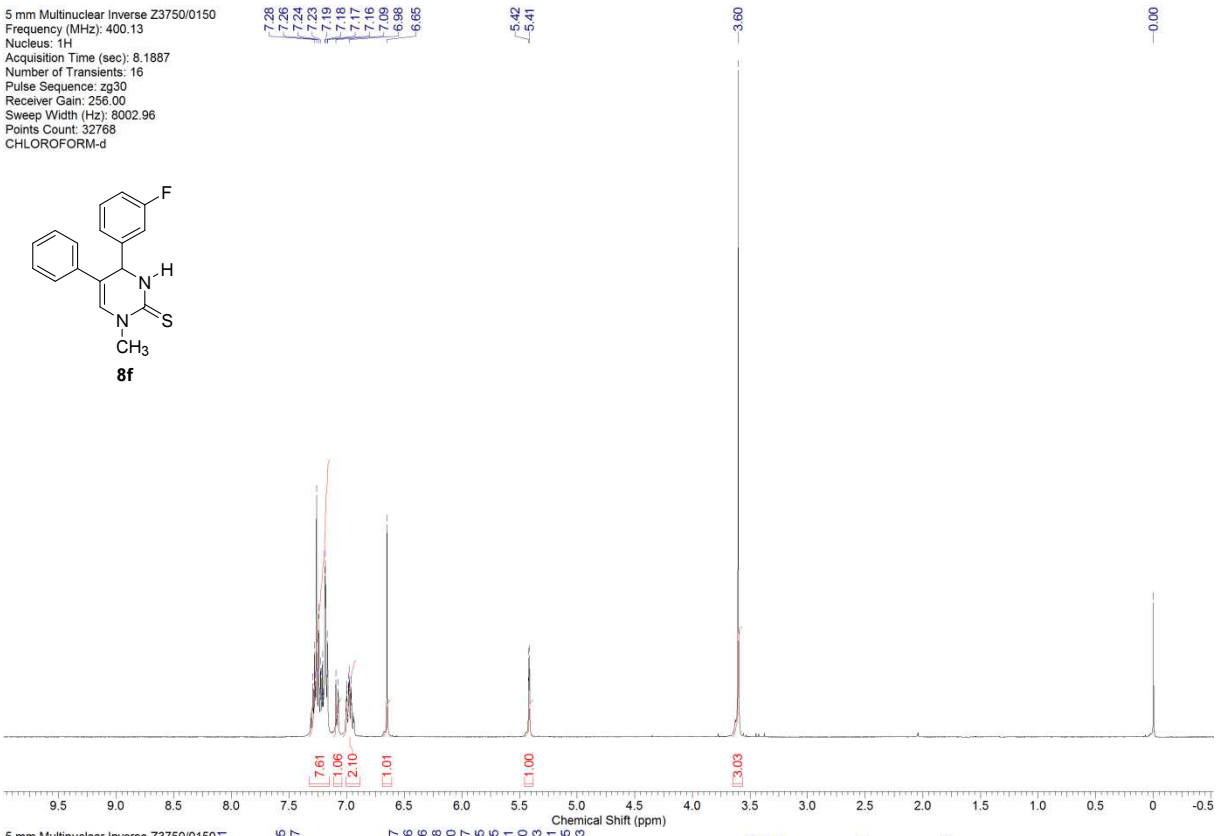

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 4096
Pulse Sequence: zgpg30
Receiver Gain: 14596.50
Sweep Width (Hz): 23979.35
Solvent: CHLOROFORM-d
175.50
140.86
136.56
129.23
128.80
128.66
128.60
127.92
127.80
127.55
127.00
125.14
124.02
117.65
77.35
77.03
76.71
57.90
55.86
0.00
8c
Chemical Shift (ppm)

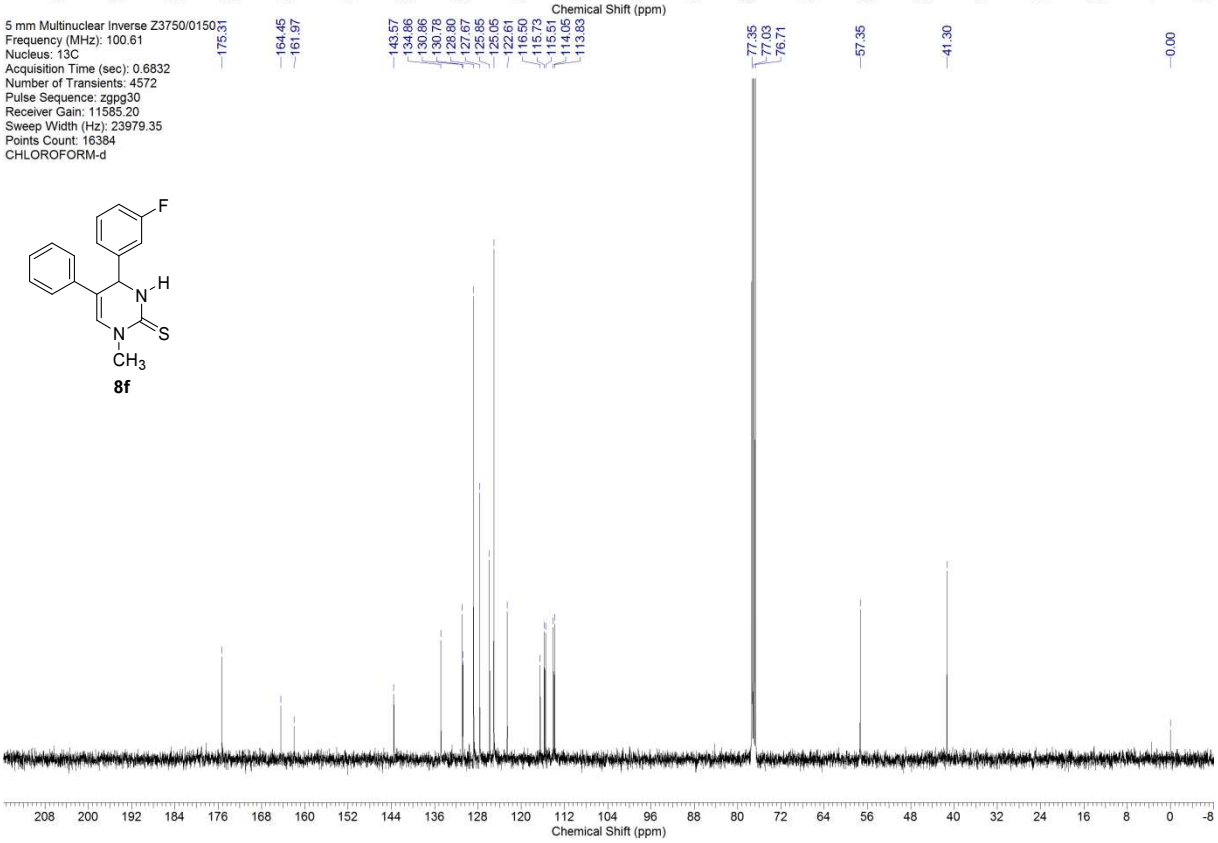

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 4.0944
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 181.00
Sweep Width (Hz): 8002.96
Points Count: 32768
CHLOROFORM-d
8d
(87:13)
9d
Chemical Shift (ppm)

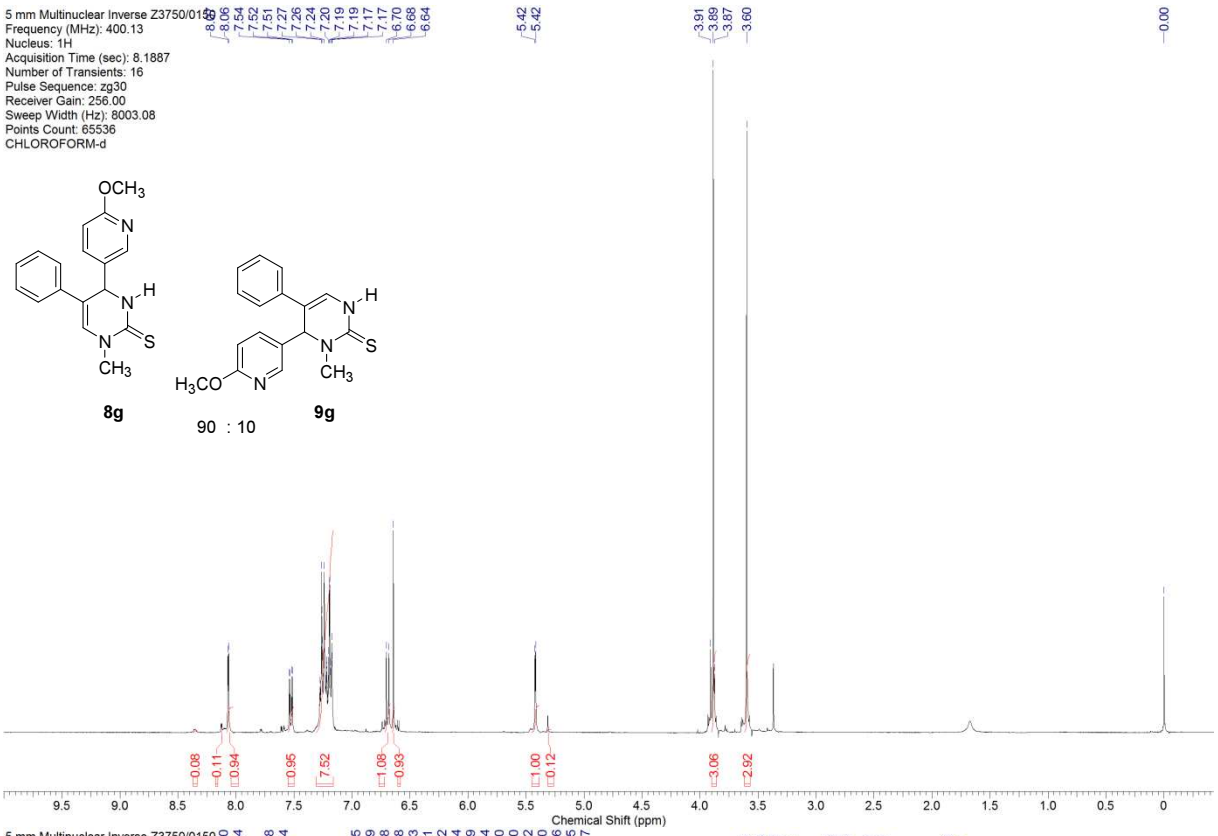

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 3072
Pulse Sequence: zgpg30
Receiver Gain: 5792.60
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
8d
(55:45)
9d
Chemical Shift (ppm)

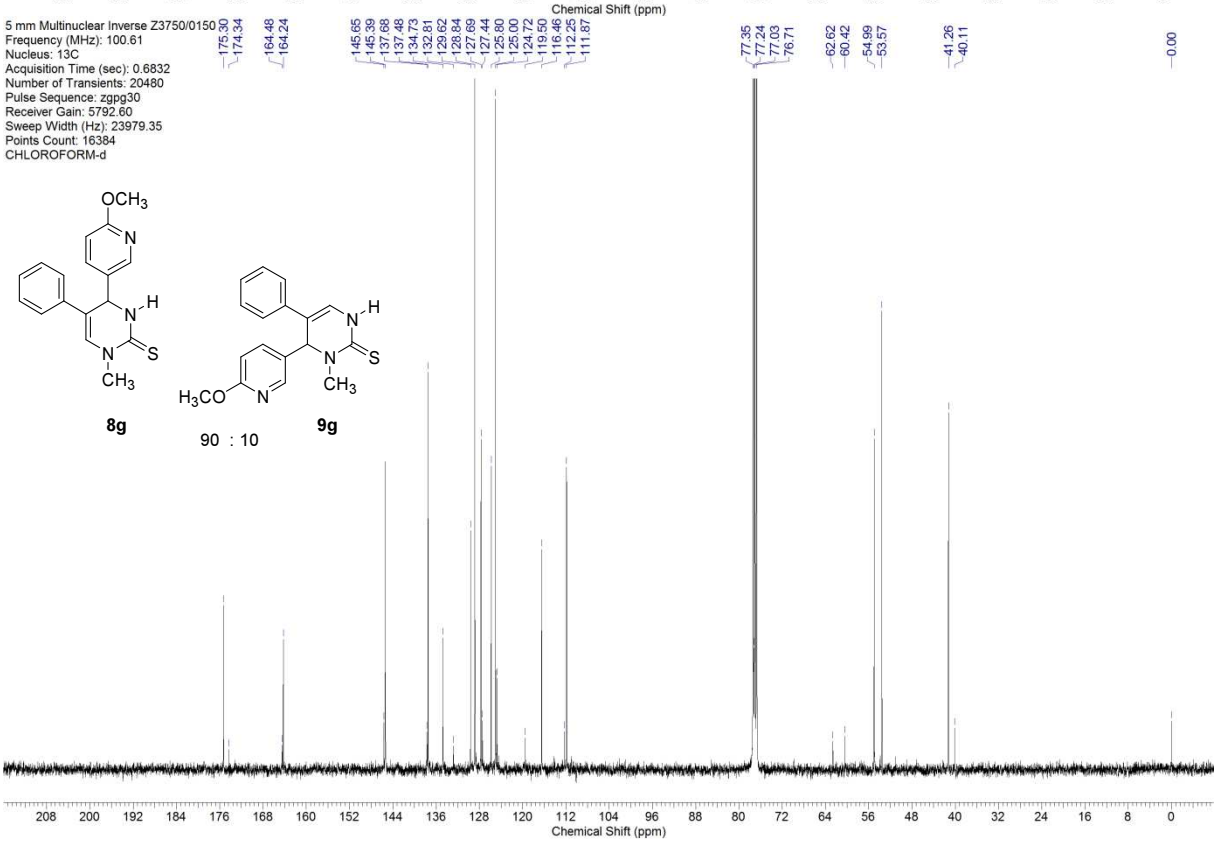

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 256.00
Sweep Width (Hz): 8002.96
Points Count: 32768
CHLOROFORM-d
7.39
7.39
7.37
7.34
7.33
7.32
7.32
7.30
6.62
6.09
6.07
6.07
5.14
5.13
5.06
5.05
5.05
5.04
5.03
3.48
0.00
5.00
0.95
1.00
0.97
0.98
2.95
Chemical Shift (ppm)
8e

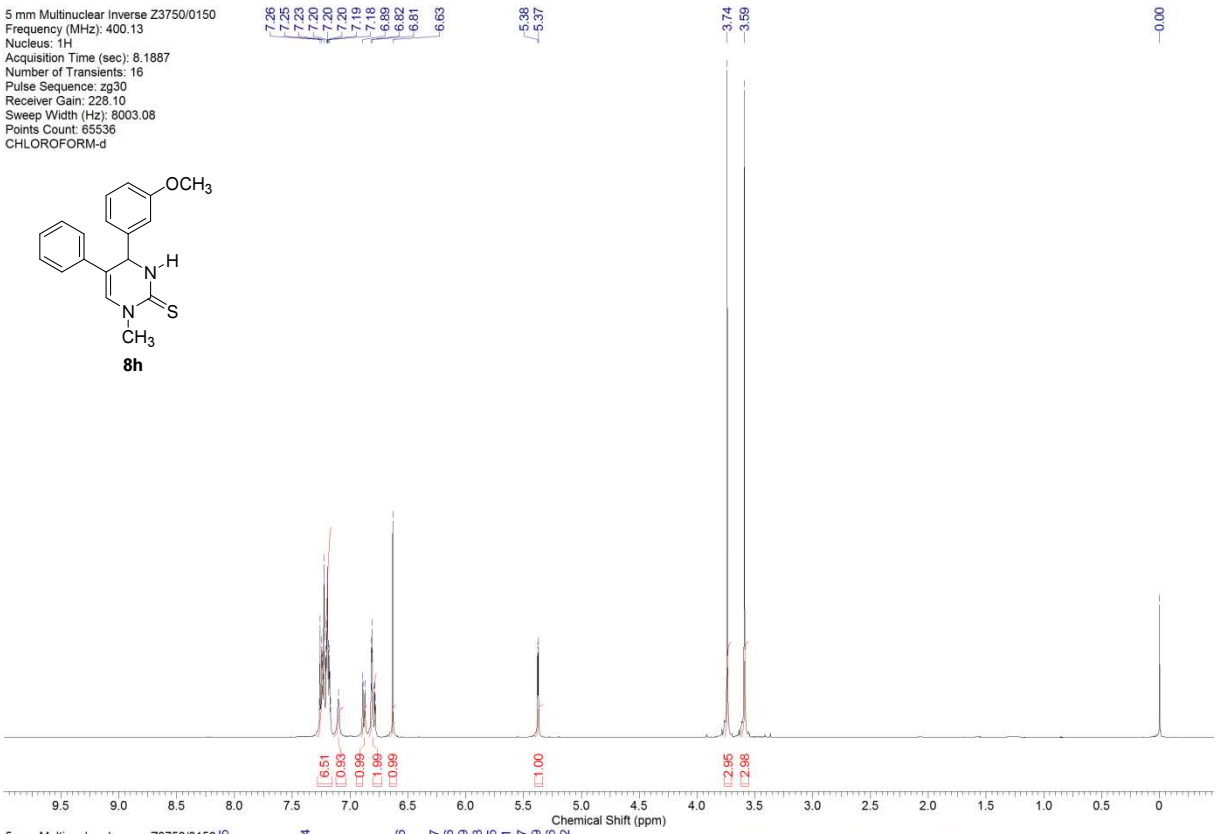

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 8192
Pulse Sequence: zgpg30
Receiver Gain: 11585.20
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
176.61
142.51
129.10
128.50
127.47
126.67
105.70
77.36
77.25
77.04
76.72
56.43
40.89
0.00
Chemical Shift (ppm)
8e

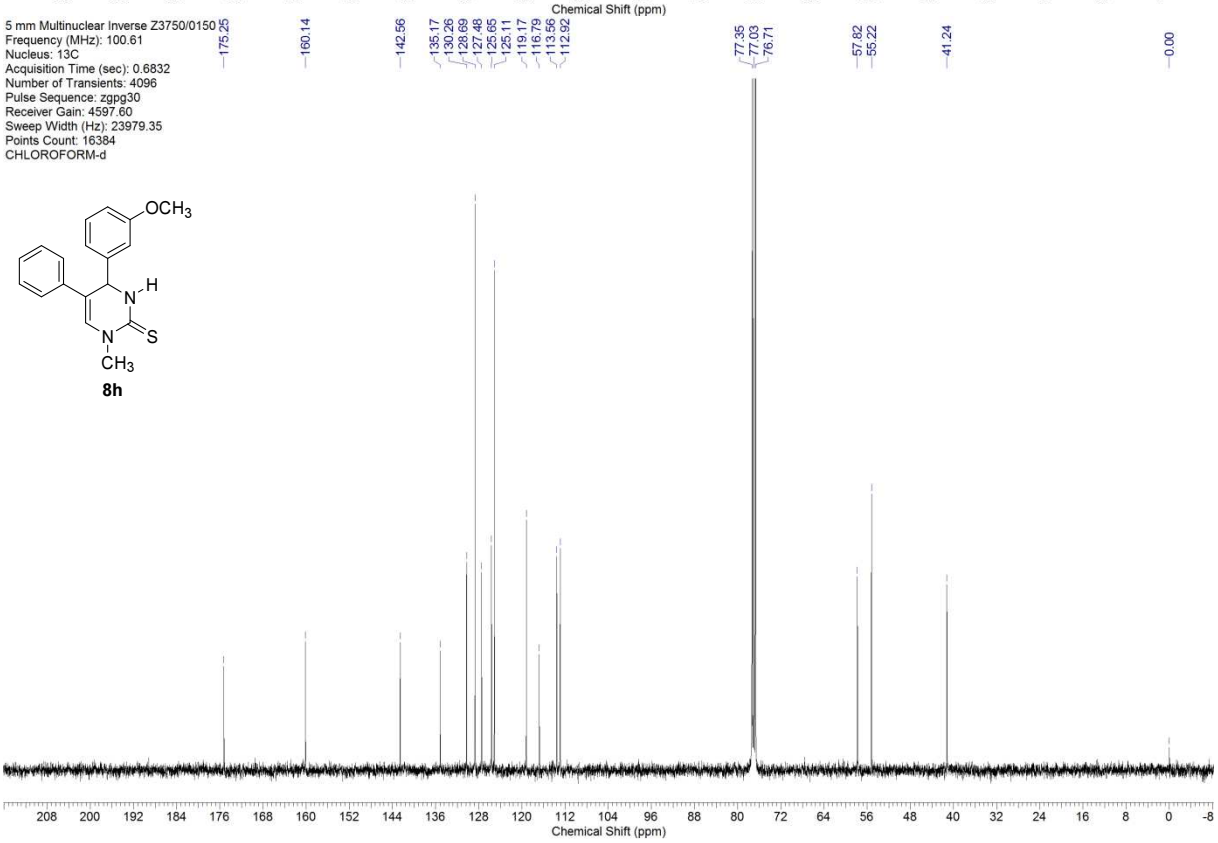

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 256.00
Sweep Width (Hz): 8002.96
Points Count: 32768
CHLOROFORM-d
7.28
7.26
7.24
7.23
7.19
7.18
7.17
7.16
7.09
6.98
6.65
5.42
5.41
3.60
0.00
F
H
N
N
S
CH3
8f
7.61
1.06
2.10
1.01
1.00
3.03
Chemical Shift (ppm)

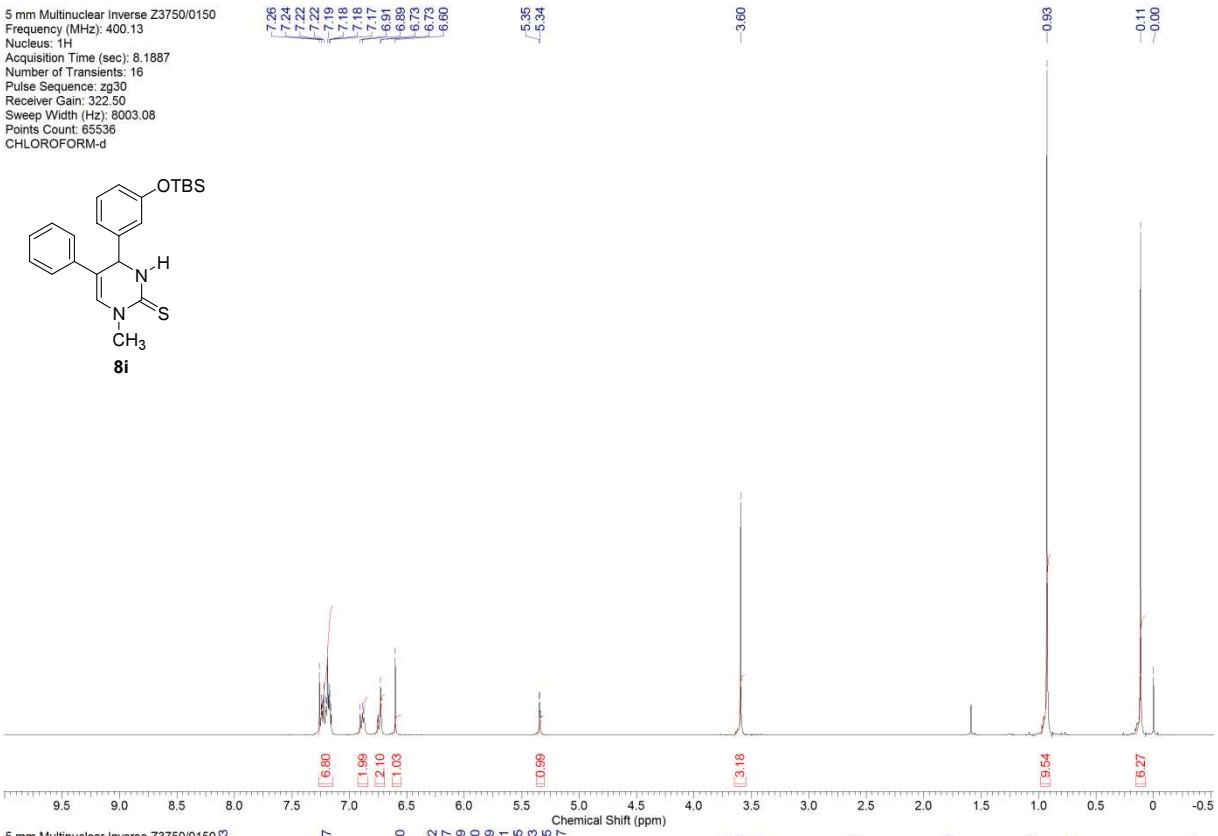

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 4572
Pulse Sequence: zgpg30
Receiver Gain: 11585.20
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
175.31
164.45
161.97
143.57
134.86
130.96
130.78
128.80
127.67
125.85
125.05
122.61
116.50
115.73
115.51
114.05
113.83
77.35
77.03
76.71
57.35
41.30
0.00
F
H
N
N
S
CH3
8f
Chemical Shift (ppm)

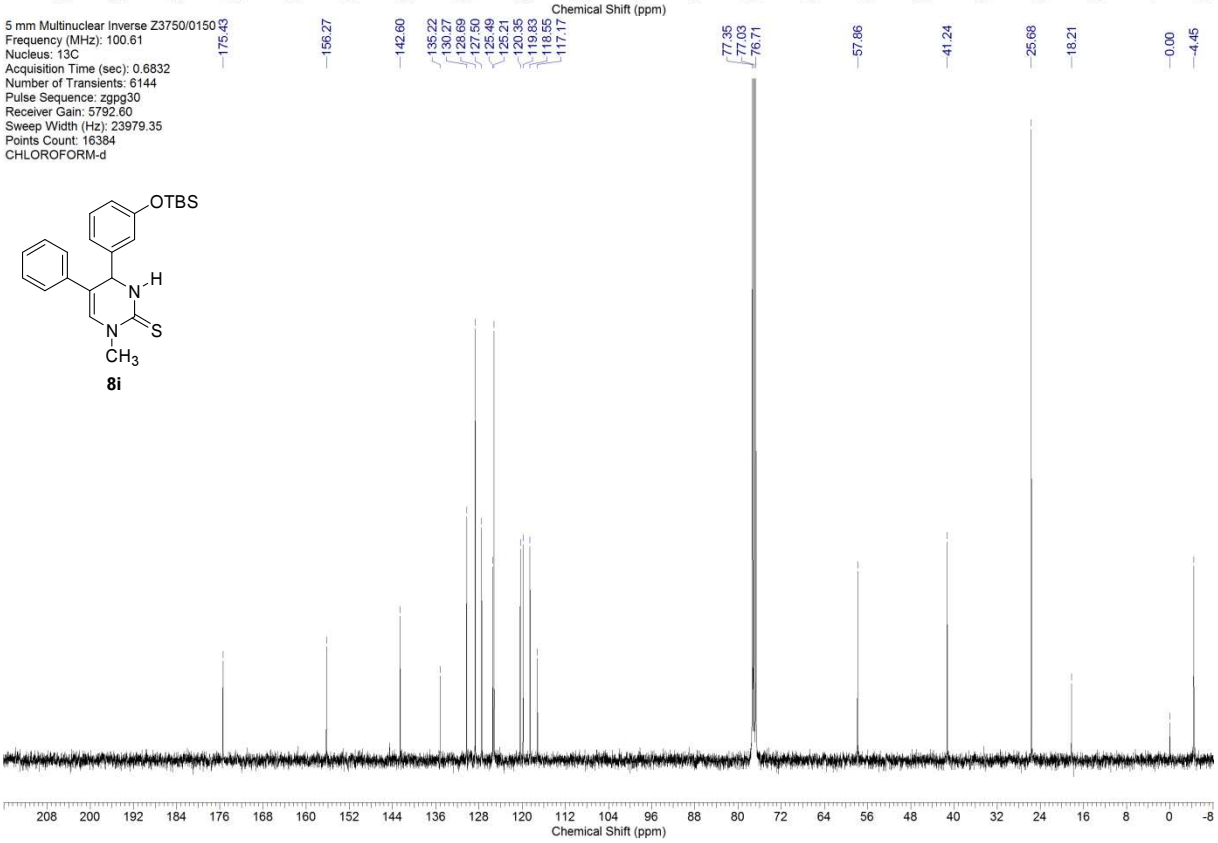

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 256.00
Sweep Width (Hz): 8003.08
Points Count: 65536
CHLOROFORM-d
8.08
8.06
7.54
7.52
7.51
7.27
7.26
7.24
7.20
7.19
7.19
7.17
7.17
6.70
6.68
6.64
5.42
5.42
3.91
3.89
3.87
3.60
0.00
OCH3
N
H
N
S
N
CH3
H3CO
N
8g
90 : 10
9g
0.08
0.11
0.94
0.95
7.52
1.06
0.93
1.00
0.12
3.06
2.92
Chemical Shift (ppm)

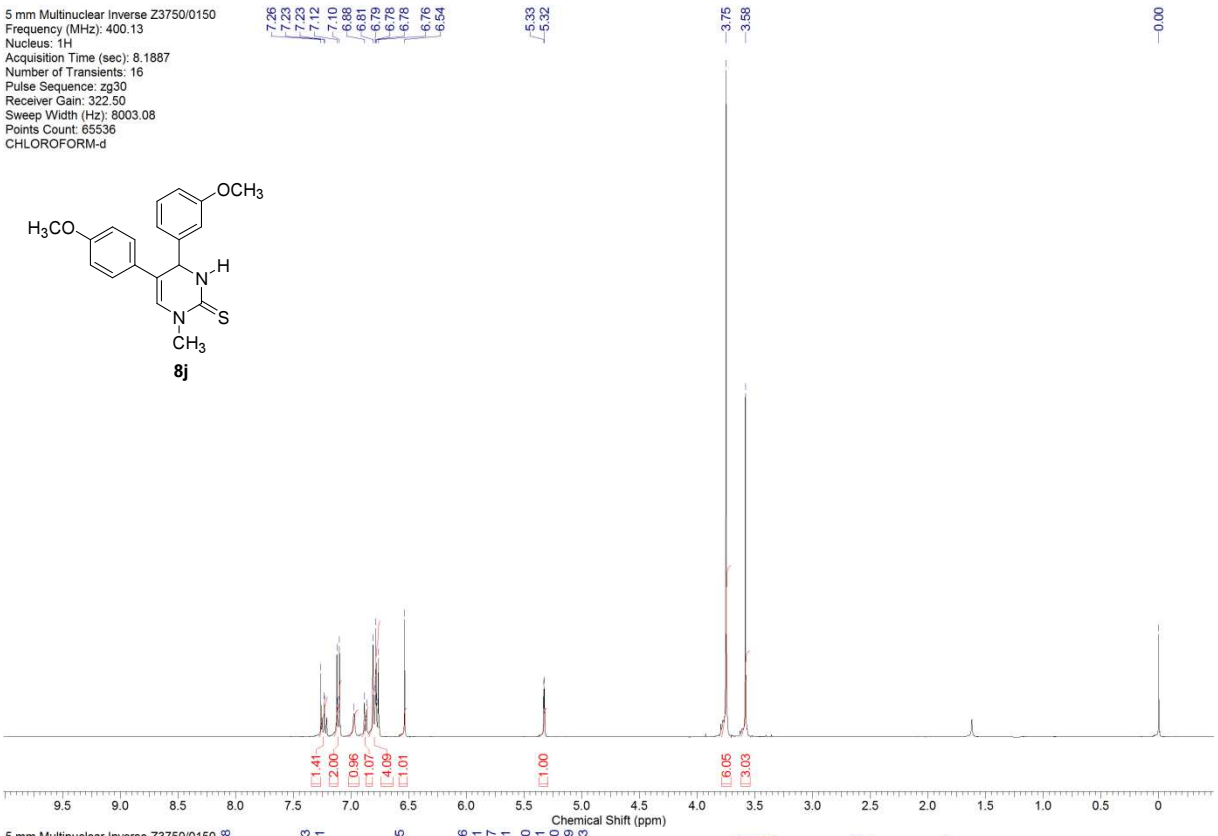

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 20480
Pulse Sequence: zgpg30
Receiver Gain: 5792.60
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
175.90
174.34
164.48
164.24
145.65
145.39
137.68
137.48
134.73
132.81
129.62
128.84
127.69
127.44
125.80
125.00
124.72
119.50
116.46
112.25
111.87
77.35
77.24
77.03
76.71
62.62
60.42
54.99
53.57
41.26
40.11
0.00
OCH3
N
H
N
S
N
CH3
H3CO
N
8g
90 : 10
9g
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 228.10
Sweep Width (Hz): 8003.08
Points Count: 65536
CHLOROFORM-d
7.26
7.25
7.23
7.20
7.20
7.20
7.19
7.18
6.89
6.82
6.81
6.63
5.38
5.37
3.74
3.59
0.00
OCH3
N
H
N
S
CH3
8h
6.51
0.93
0.99
1.99
0.99
1.00
2.95
2.98
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 4096
Pulse Sequence: zgpg30
Receiver Gain: 4597.60
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
175.25
160.14
142.56
135.17
130.26
128.69
127.48
125.65
125.11
119.17
116.79
113.56
112.92
77.35
77.03
76.71
57.82
55.22
41.24
0.00
OCH3
N
H
N
S
CH3
8h
Chemical Shift (ppm)

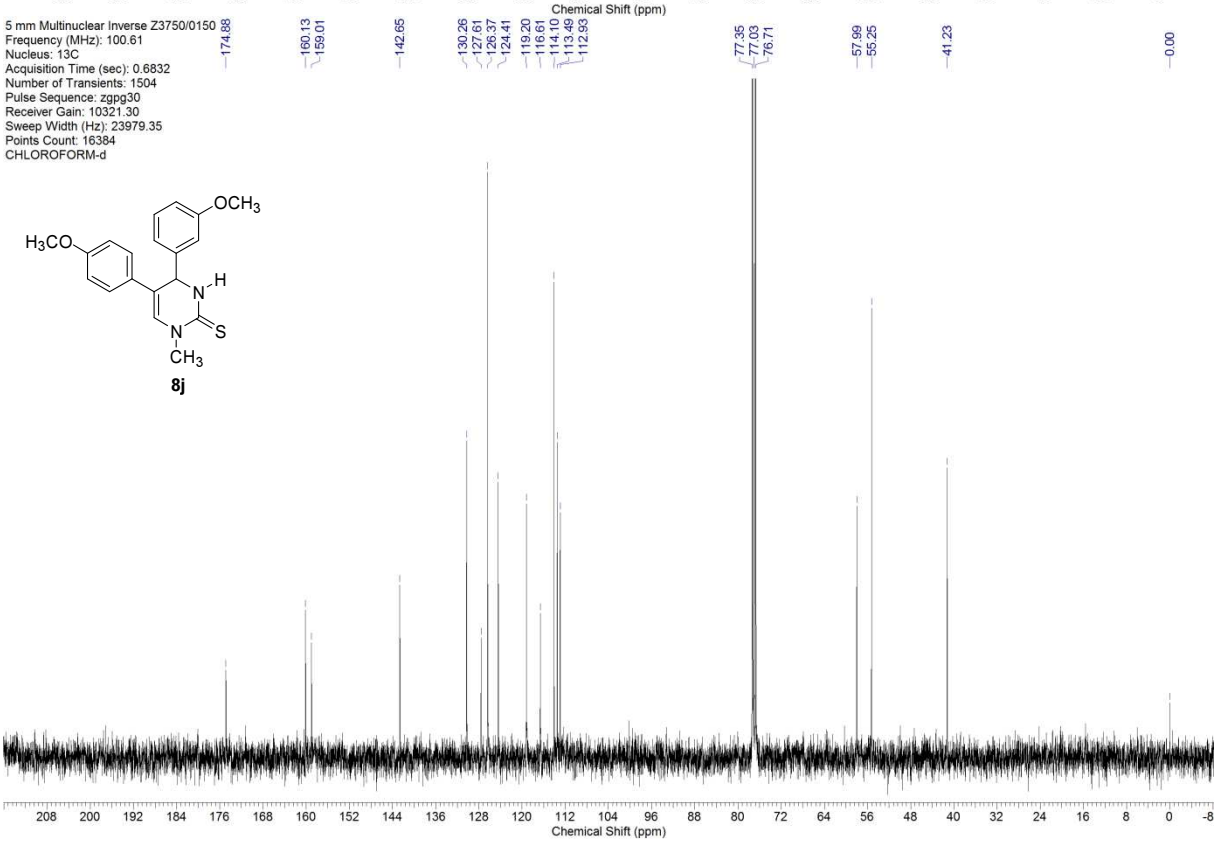

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 322.50
Sweep Width (Hz): 8003.08
Points Count: 65536
CHLOROFORM-d
OTBS
N
H
N
S
CH3
8i
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 6144
Pulse Sequence: zgpg30
Receiver Gain: 5792.60
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
OTBS
N
H
N
S
CH3
8i
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 322.50
Sweep Width (Hz): 8003.08
Points Count: 65536
CHLOROFORM-d
7.26
7.23
7.23
7.12
7.10
6.88
6.81
6.79
6.78
6.78
6.76
6.54
5.33
5.32
3.75
3.58
0.00
1.41
2.00
0.96
1.07
4.09
1.01
1.00
6.05
3.03
Chemical Shift (ppm)

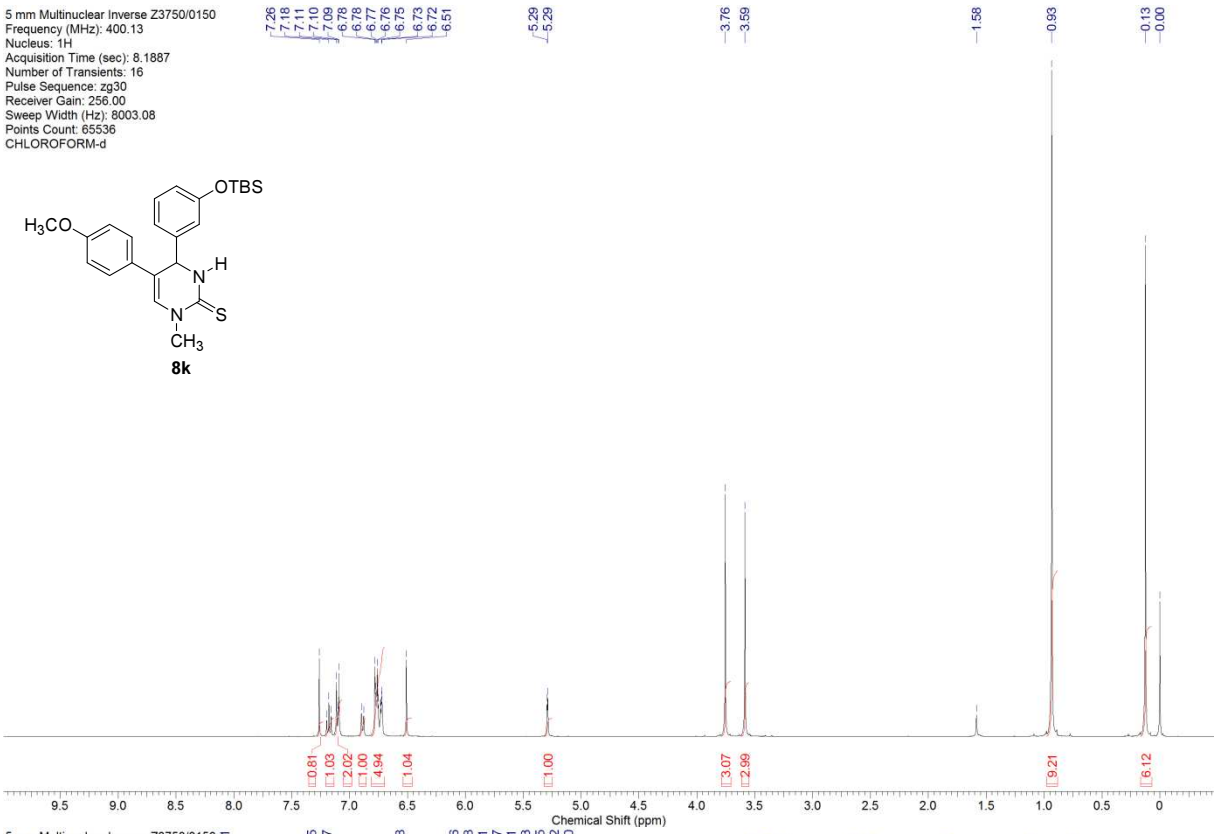
OCH3
H3CO
H
N
N
S
CH3

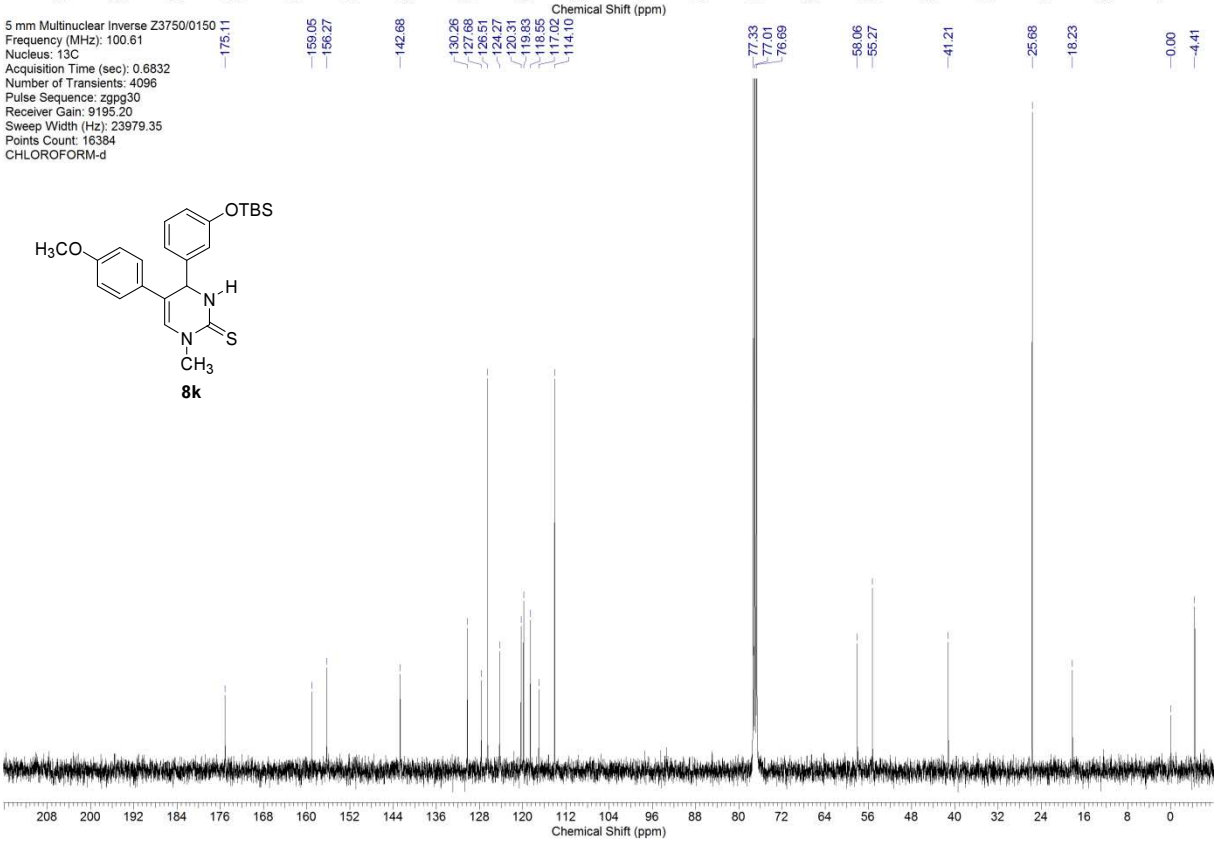
8j

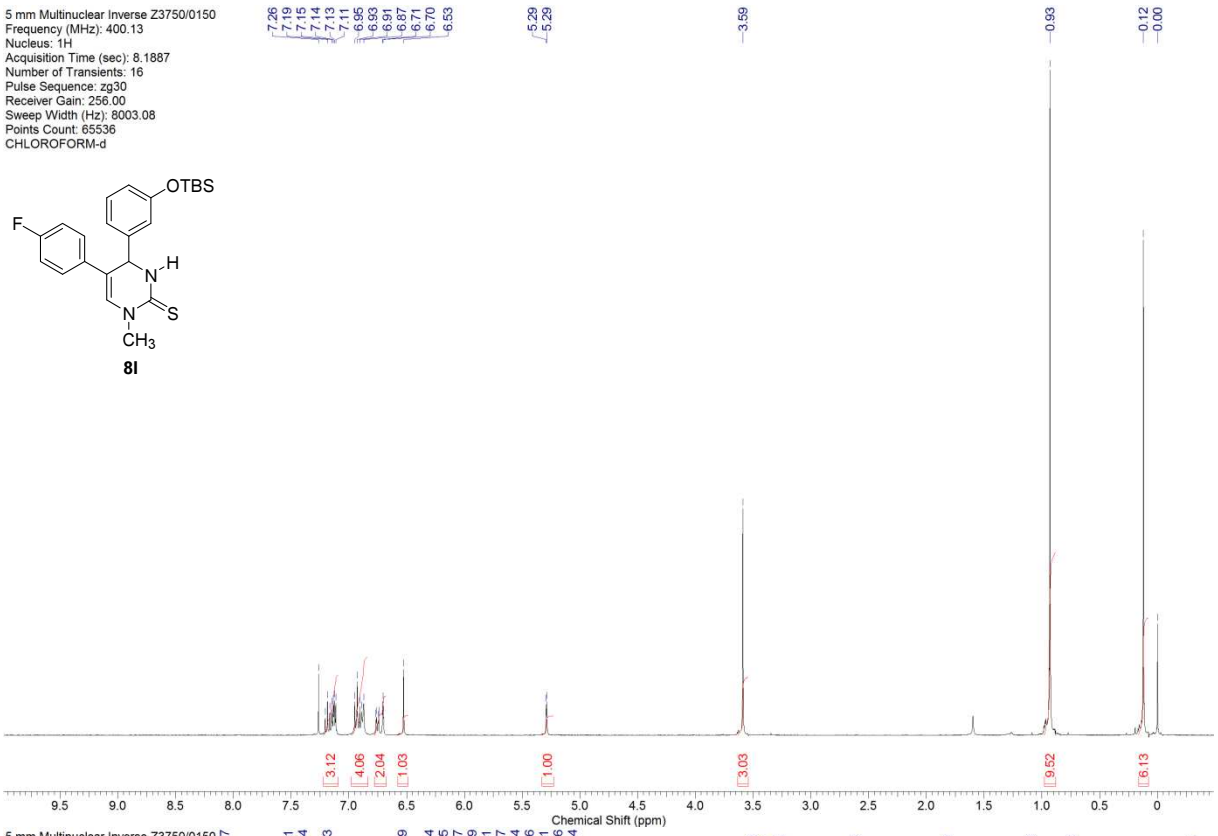

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 1504
Pulse Sequence: zgpg30
Receiver Gain: 10321.30
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
174.88
160.13
159.01
142.65
130.26
127.61
126.37
124.41
119.20
116.61
114.10
113.49
112.93
77.35
77.03
76.71
57.99
55.25
41.23
0.00
Chemical Shift (ppm)

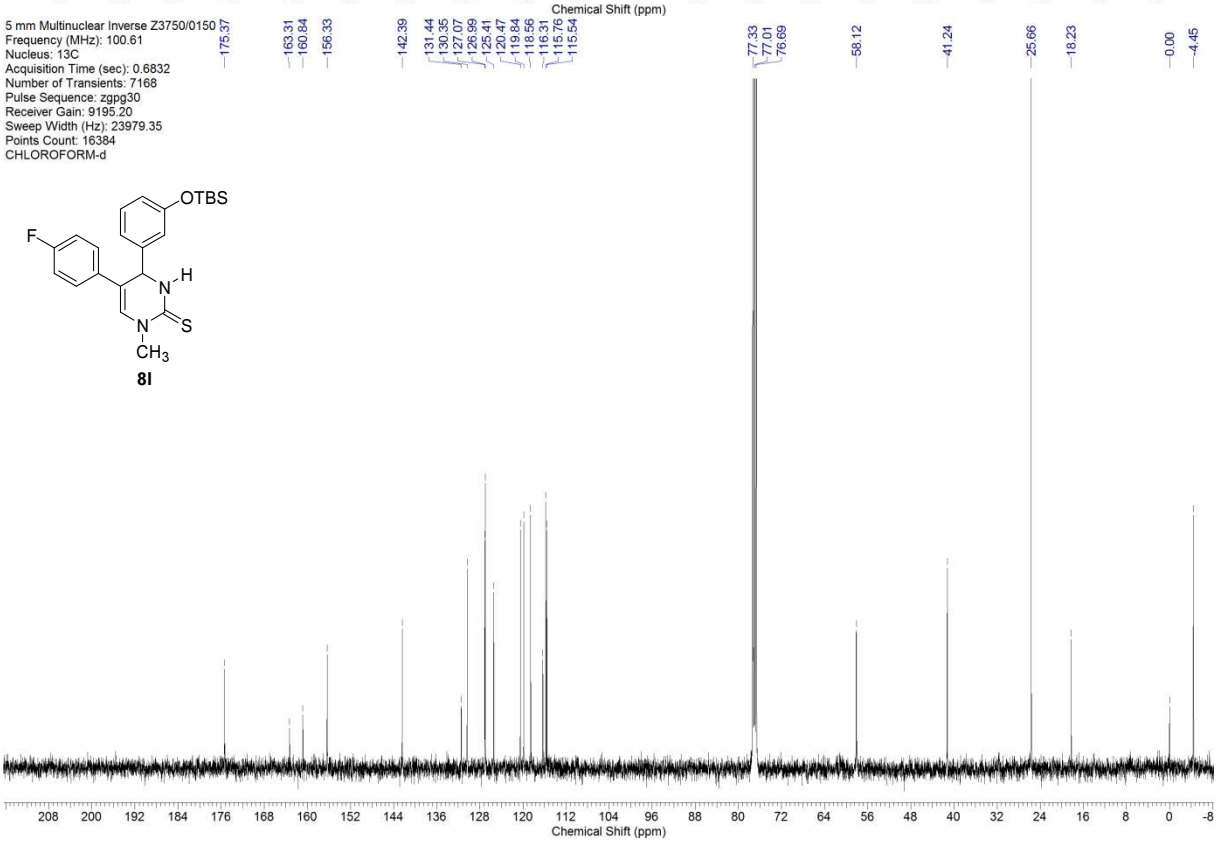
OCH3
H3CO
H
N
N
S
CH3

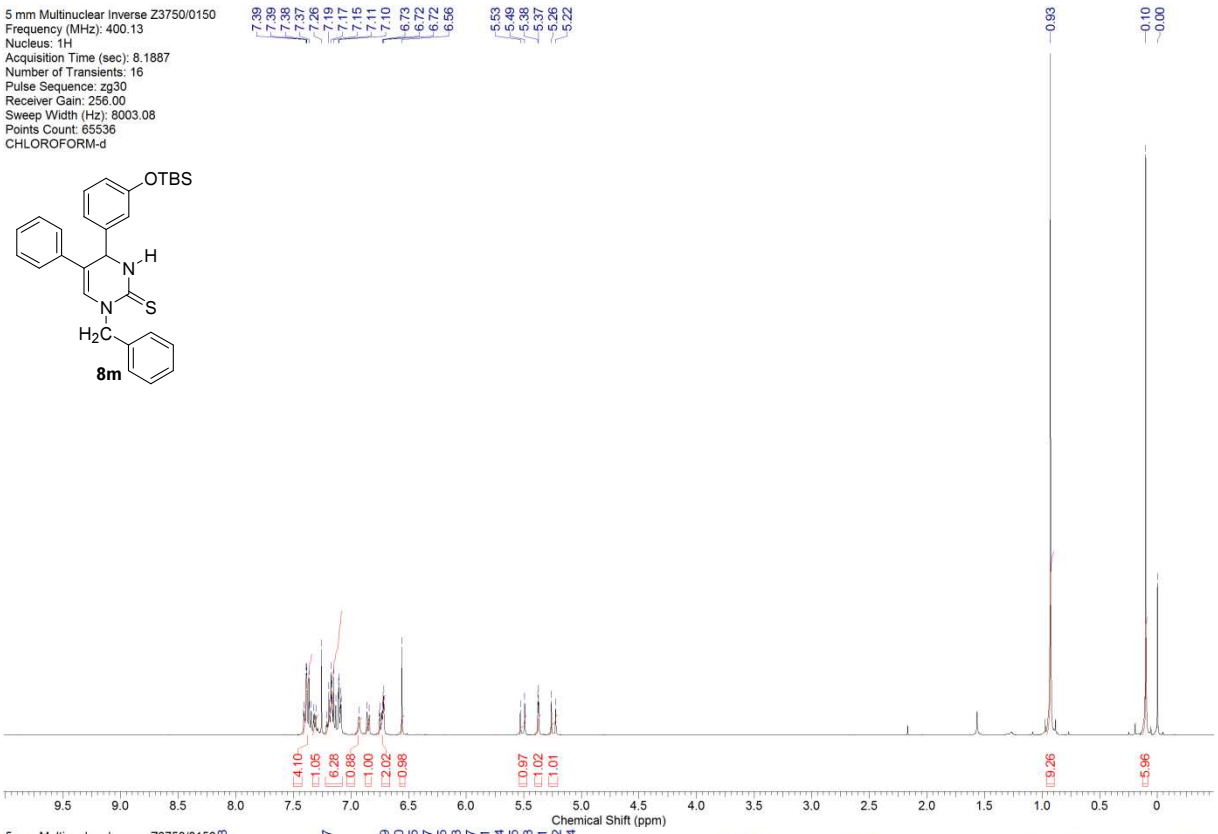
8j

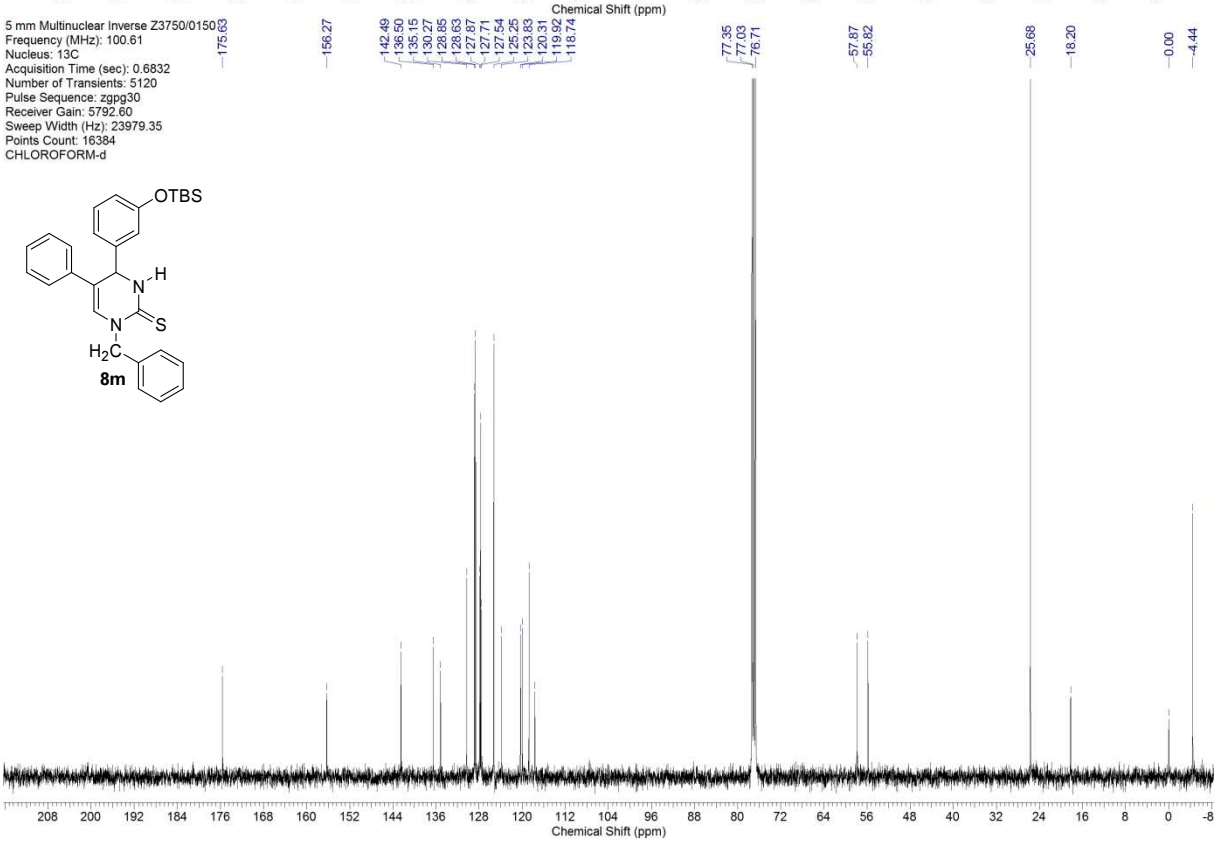

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 256.00
Sweep Width (Hz): 8003.08
Points Count: 65536
CHLOROFORM-d
OTBS
H3CO
N
H
N
S
CH3
8k
Chemical Shift (ppm)

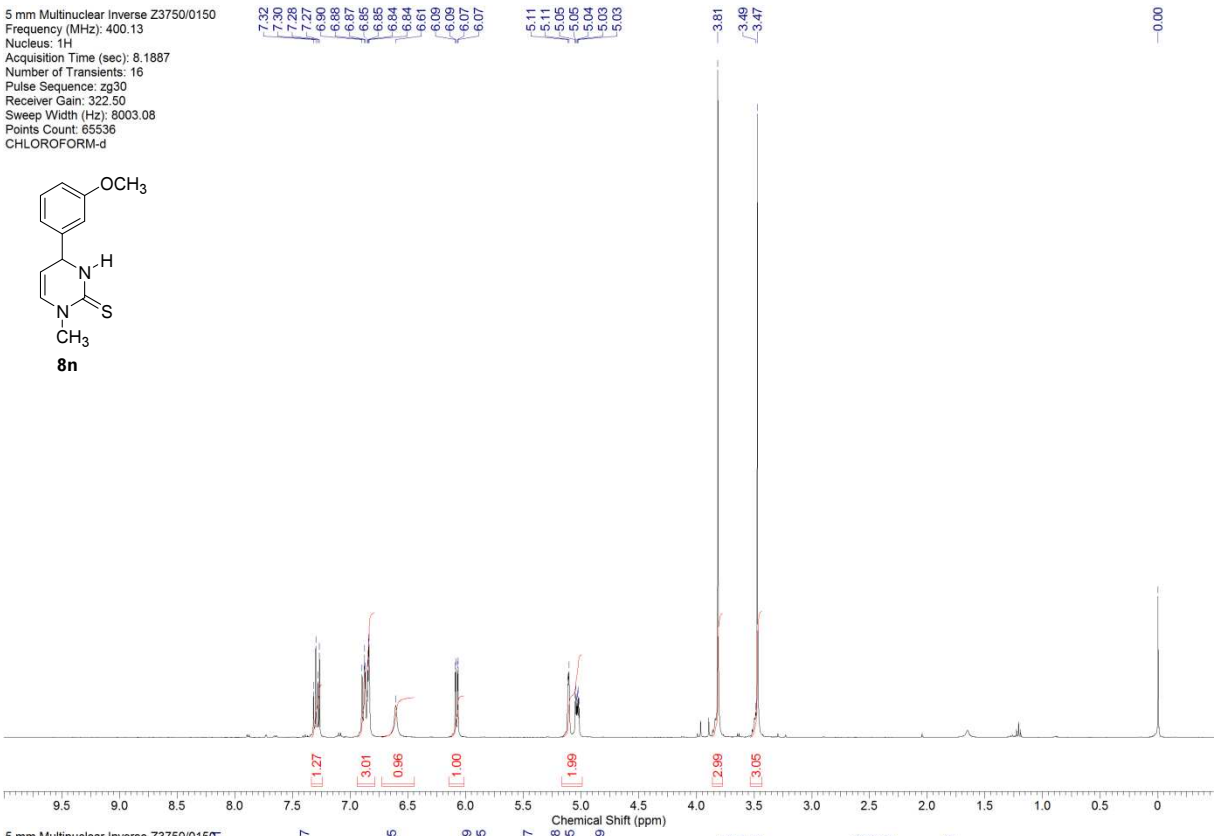

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 4096
Pulse Sequence: zgpg30
Receiver Gain: 9195.20
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
OTBS
H3CO
N
H
N
S
CH3
8k
Chemical Shift (ppm)

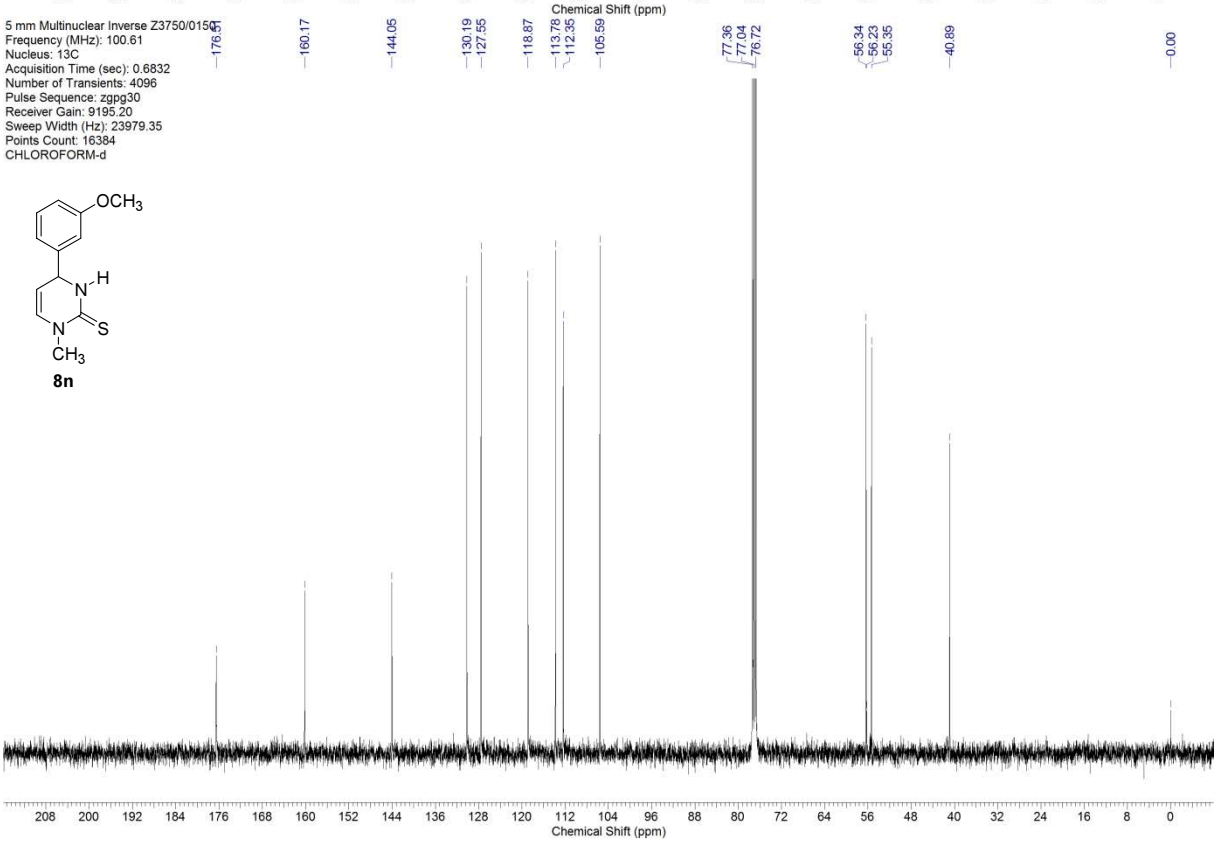

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 256.00
Sweep Width (Hz): 8003.08
Points Count: 65536
CHLOROFORM-d
7.26
7.19
7.15
7.14
7.13
7.11
6.95
6.93
6.91
6.87
6.71
6.70
6.53
5.29
5.29
3.59
0.93
0.12
0.00
OTBS
F
N
H
N
S
CH3
8l
3.12
4.06
2.04
1.03
1.00
3.03
9.52
6.13
Chemical Shift (ppm)

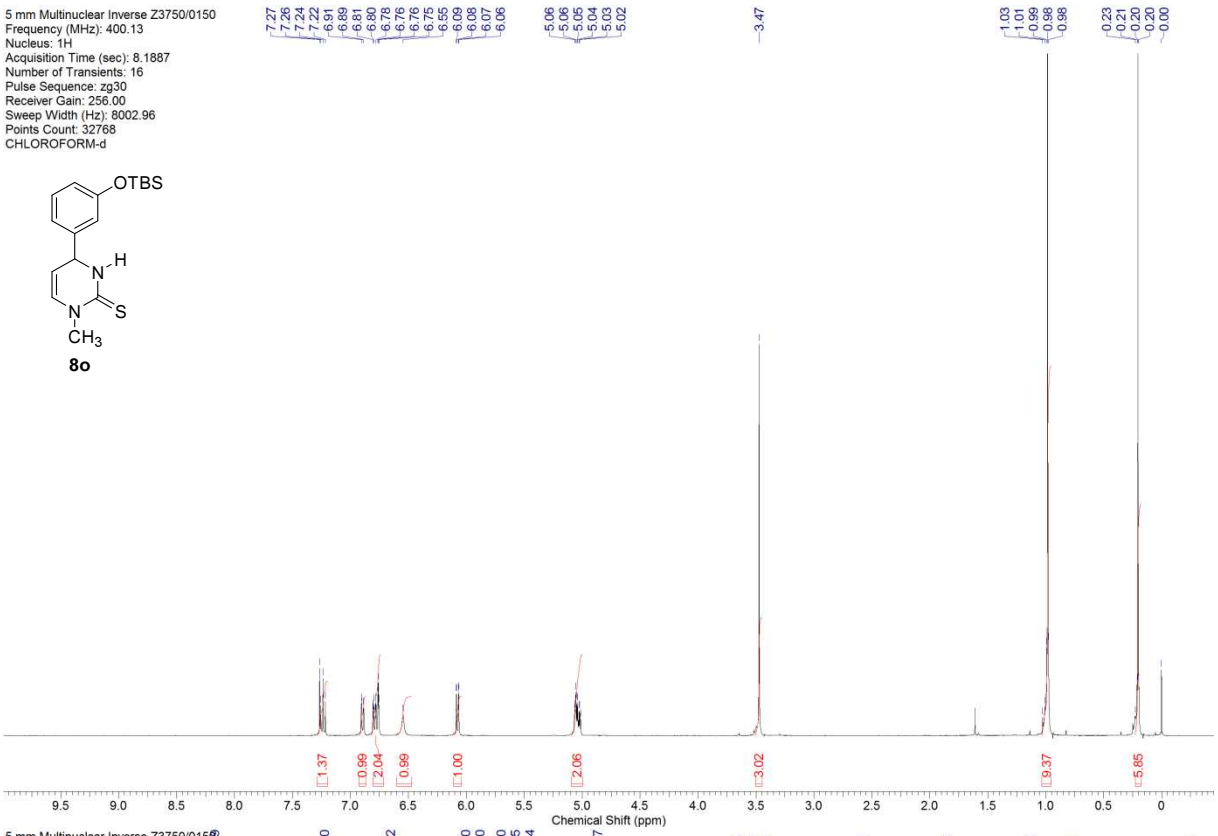

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 7168
Pulse Sequence: zgpg30
Receiver Gain: 9195.20
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
175.37
163.31
160.84
156.33
142.39
131.44
130.35
127.07
126.99
125.41
120.47
119.84
118.56
116.31
115.76
115.54
77.33
77.01
76.69
58.12
41.24
25.66
18.23
0.00
-4.45
OTBS
F
N
H
N
S
CH3
8l
Chemical Shift (ppm)

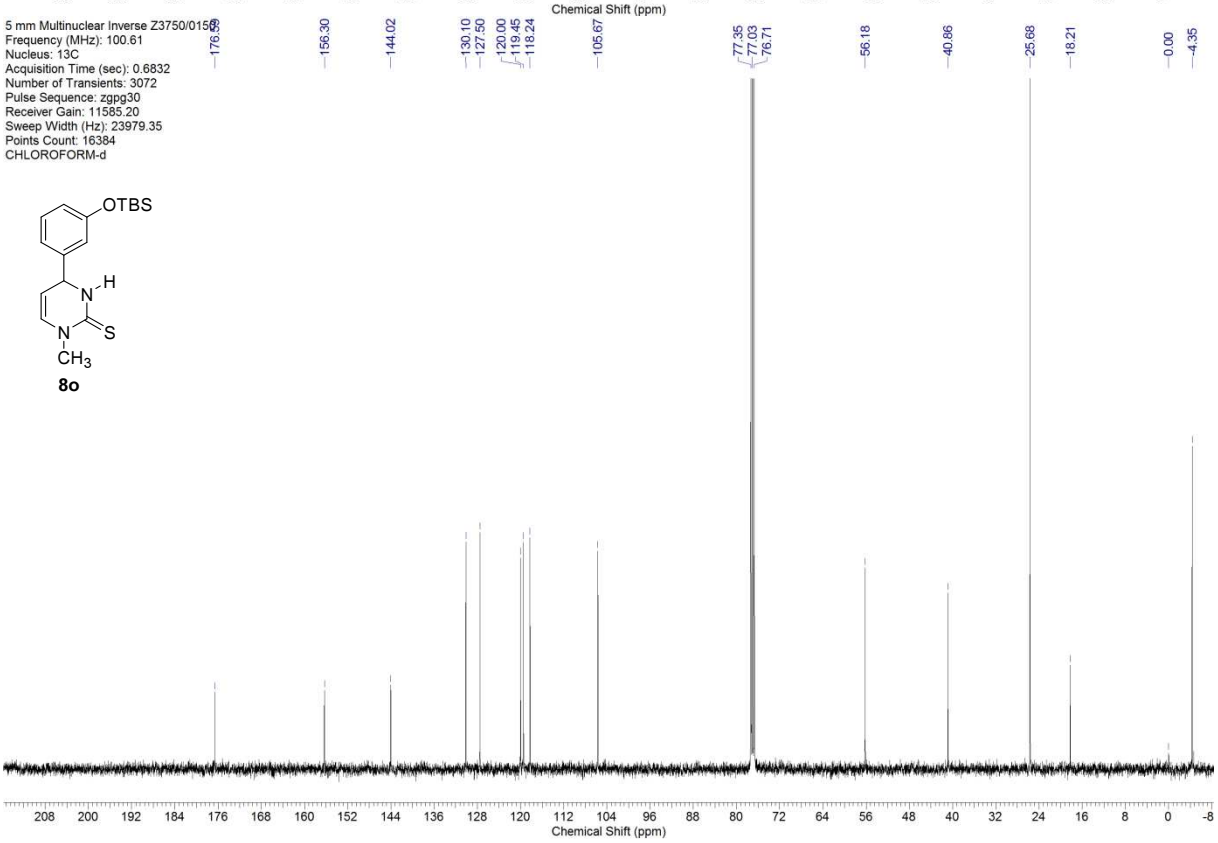

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 256.00
Sweep Width (Hz): 8003.08
Points Count: 65536
CHLOROFORM-d
OTBS
N
H
N
S
H2C
8m
Chemical Shift (ppm)

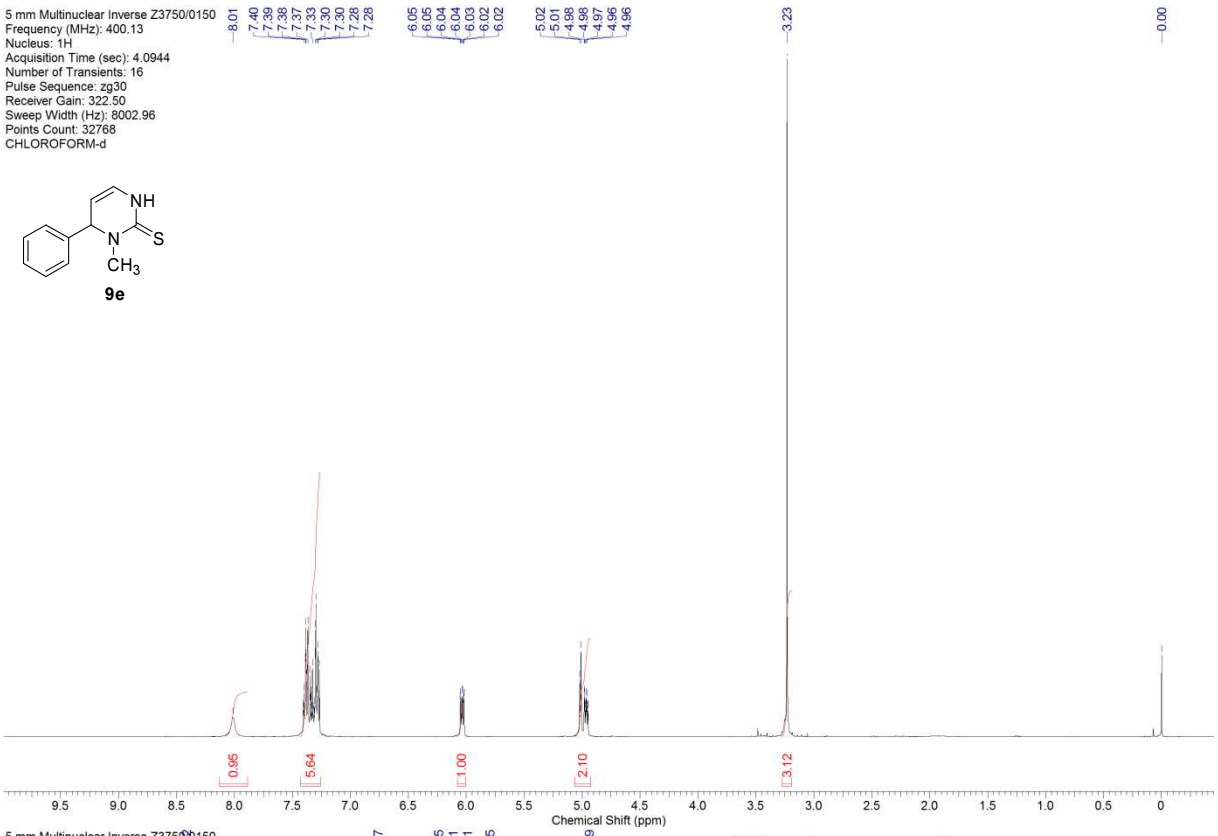

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 5120
Pulse Sequence: zgpg30
Receiver Gain: 5792.60
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
OTBS
N
H
N
S
H2C
8m
Chemical Shift (ppm)

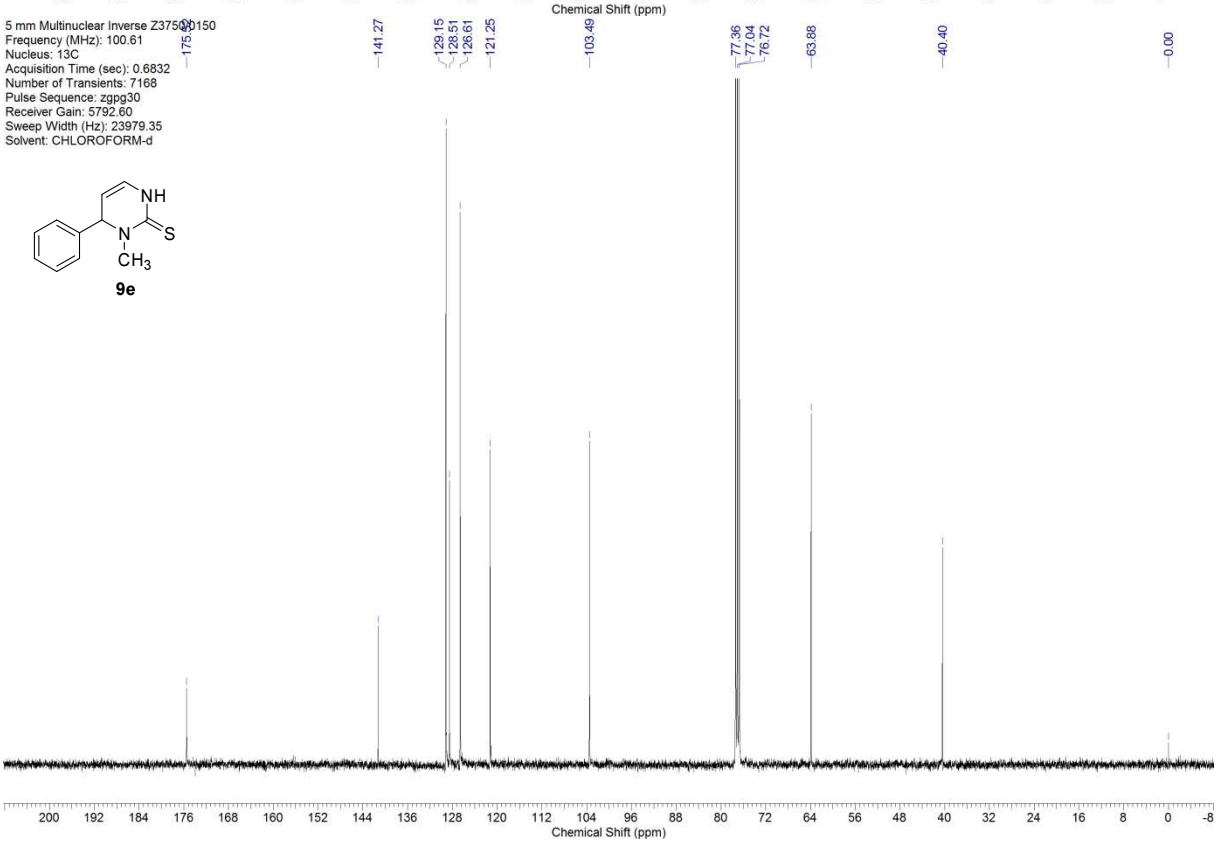

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 322.50
Sweep Width (Hz): 8003.08
Points Count: 65536
CHLOROFORM-d
7.32
7.30
7.28
7.27
6.90
6.88
6.87
6.85
6.85
6.84
6.84
6.61
6.09
6.09
6.07
6.07
5.11
5.11
5.05
5.05
5.04
5.03
5.03
3.81
3.49
3.47
0.00
1.27
3.01
0.96
1.00
1.99
2.99
3.05
Chemical Shift (ppm)
OCH3
N
H
N
S
CH3
8n

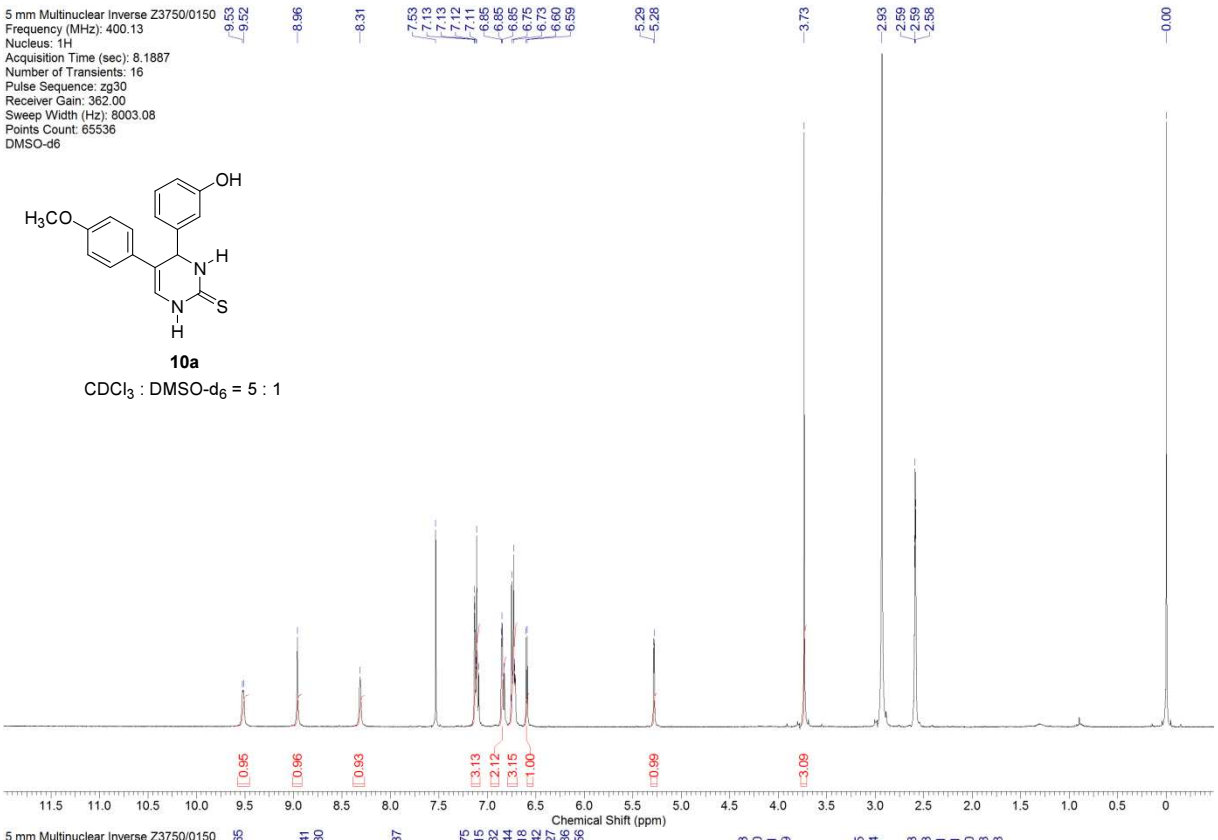

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 4096
Pulse Sequence: zgpg30
Receiver Gain: 9195.20
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
176.61
160.17
144.05
130.19
127.55
118.87
113.78
112.35
105.59
77.36
77.04
76.72
56.34
56.23
55.35
40.89
0.00
Chemical Shift (ppm)
OCH3
N
H
N
S
CH3
8n


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 256.00
Sweep Width (Hz): 8002.96
Points Count: 32768
CHLOROFORM-d
OTBS
N
H
N
S
CH3
8o
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 3072
Pulse Sequence: zgpg30
Receiver Gain: 11585.20
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
OTBS
N
H
N
S
CH3
8o
Chemical Shift (ppm)

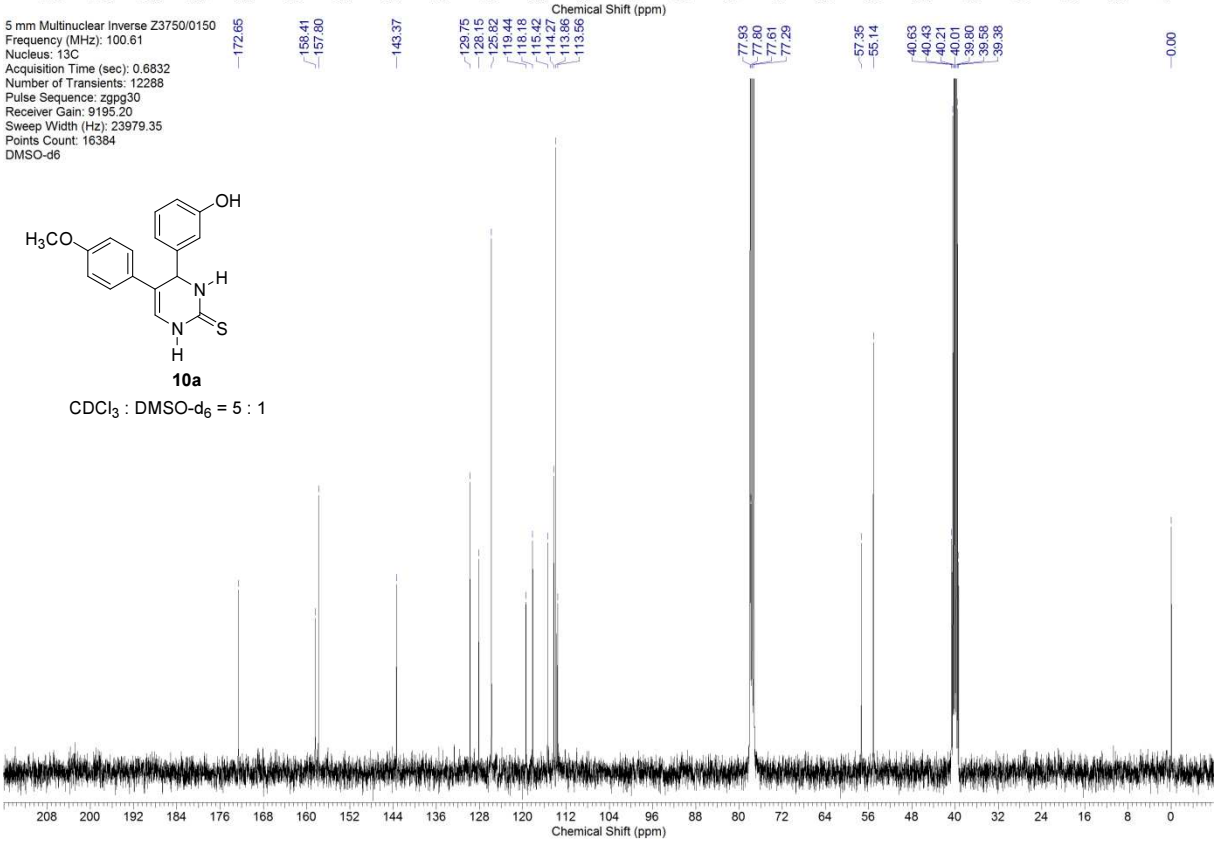

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 4.0944
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 322.50
Sweep Width (Hz): 8002.96
Points Count: 32768
CHLOROFORM-d
8.01
7.40
7.39
7.38
7.37
7.33
7.30
7.30
7.28
7.28
6.05
6.05
6.04
6.04
6.03
6.02
6.02
5.02
5.01
4.98
4.98
4.97
4.96
4.96
3.23
0.00
NH
N
S
CH3
9e
0.95
5.64
1.00
2.10
3.12
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 7168
Pulse Sequence: zgpg30
Receiver Gain: 5792.60
Sweep Width (Hz): 23979.35
Solvent: CHLOROFORM-d
175.50
141.27
129.15
128.51
126.61
121.25
103.49
77.36
77.04
76.72
63.88
40.40
0.00
NH
N
S
CH3
9e
Chemical Shift (ppm)



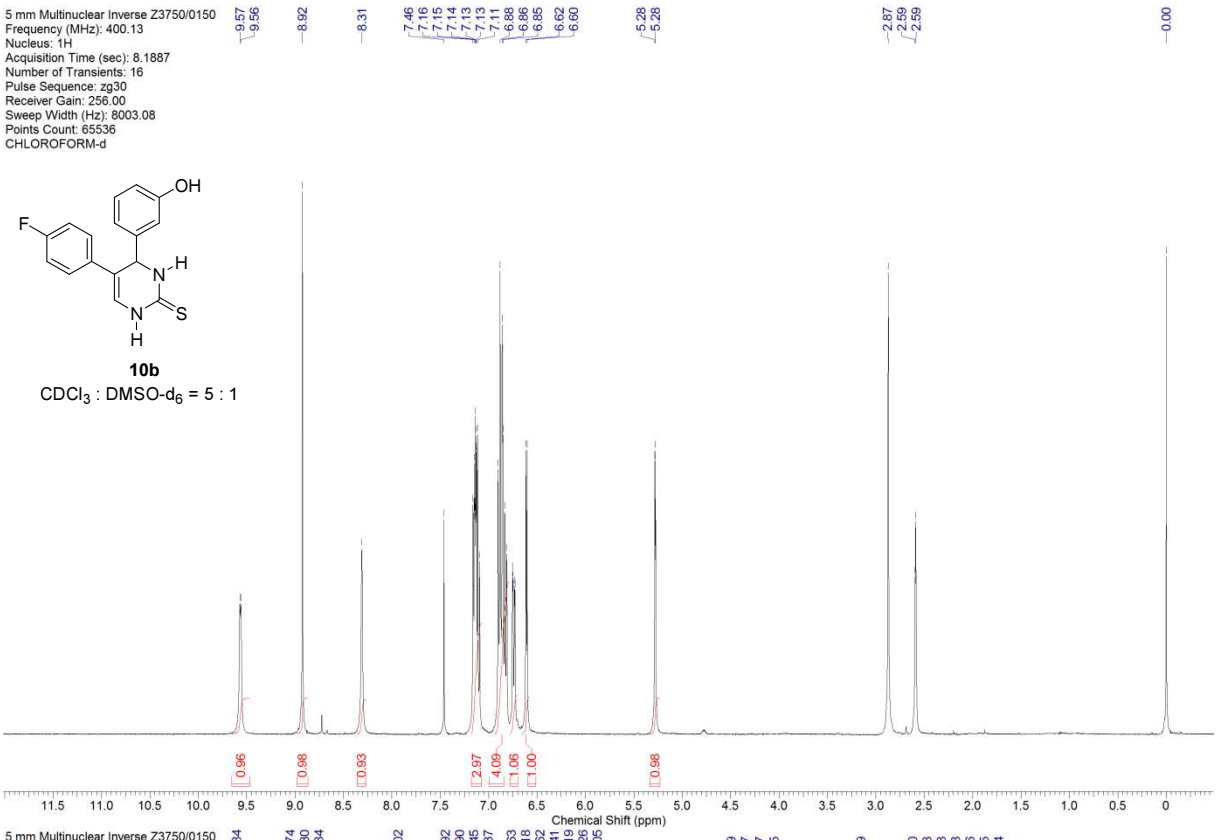
10a

$CDCl_3$: DMSO-d_6 = 5 : 1



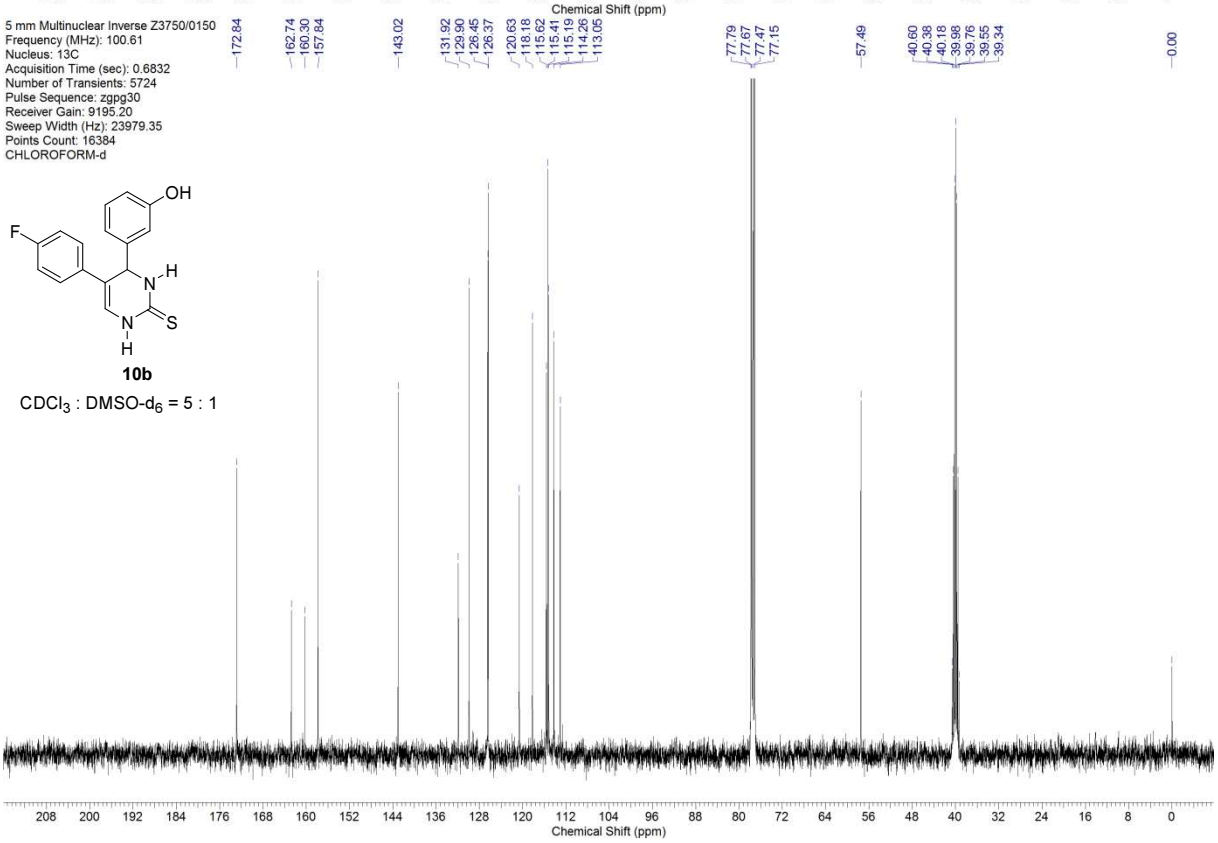
10a

$CDCl_3$: DMSO-d_6 = 5 : 1



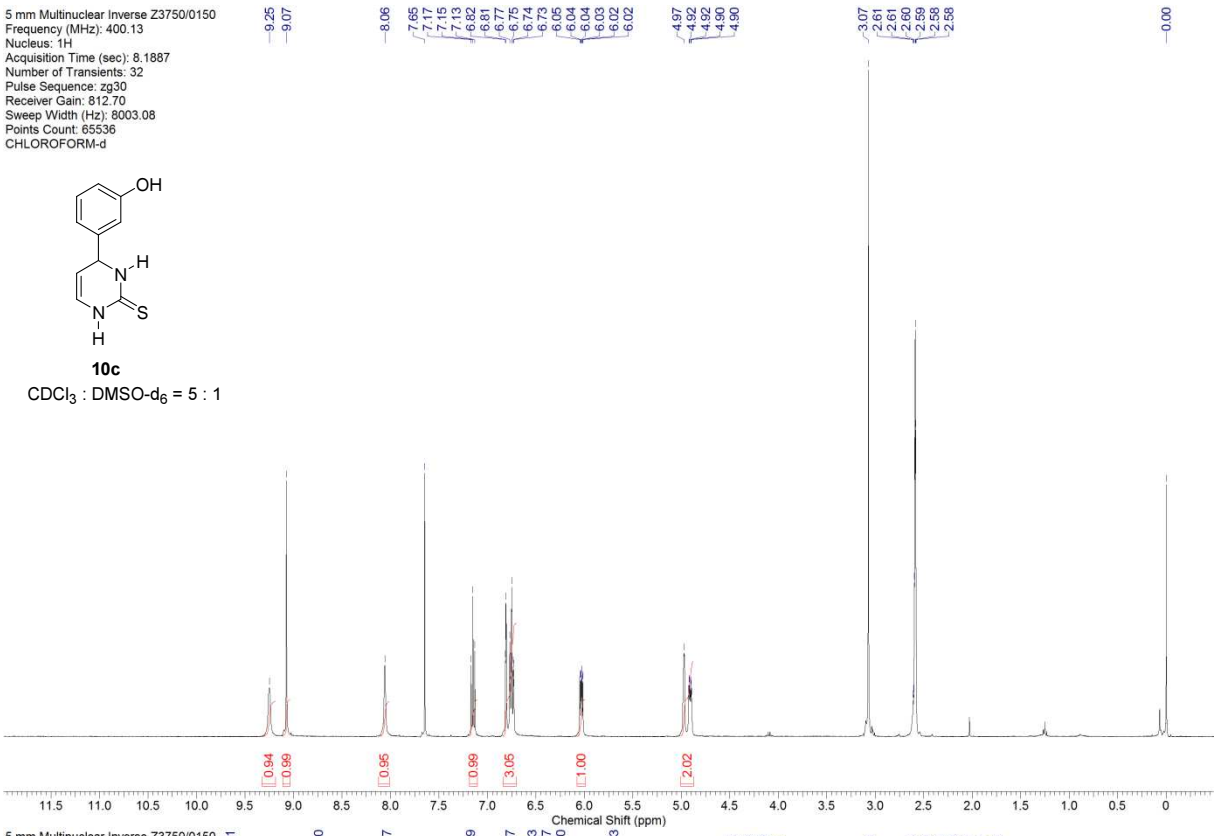
10b

$CDCl_3$: DMSO-d_6 = 5 : 1



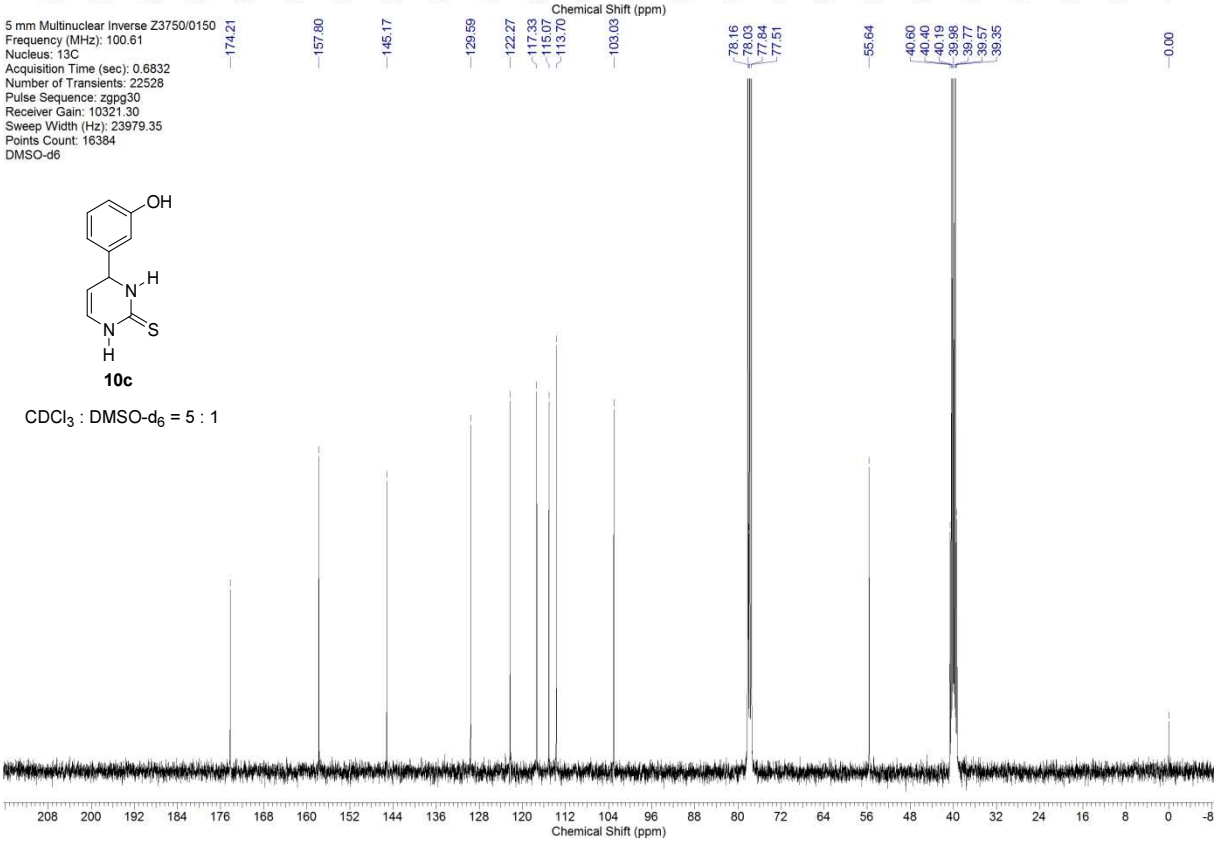
10b

$CDCl_3$: DMSO-d_6 = 5 : 1



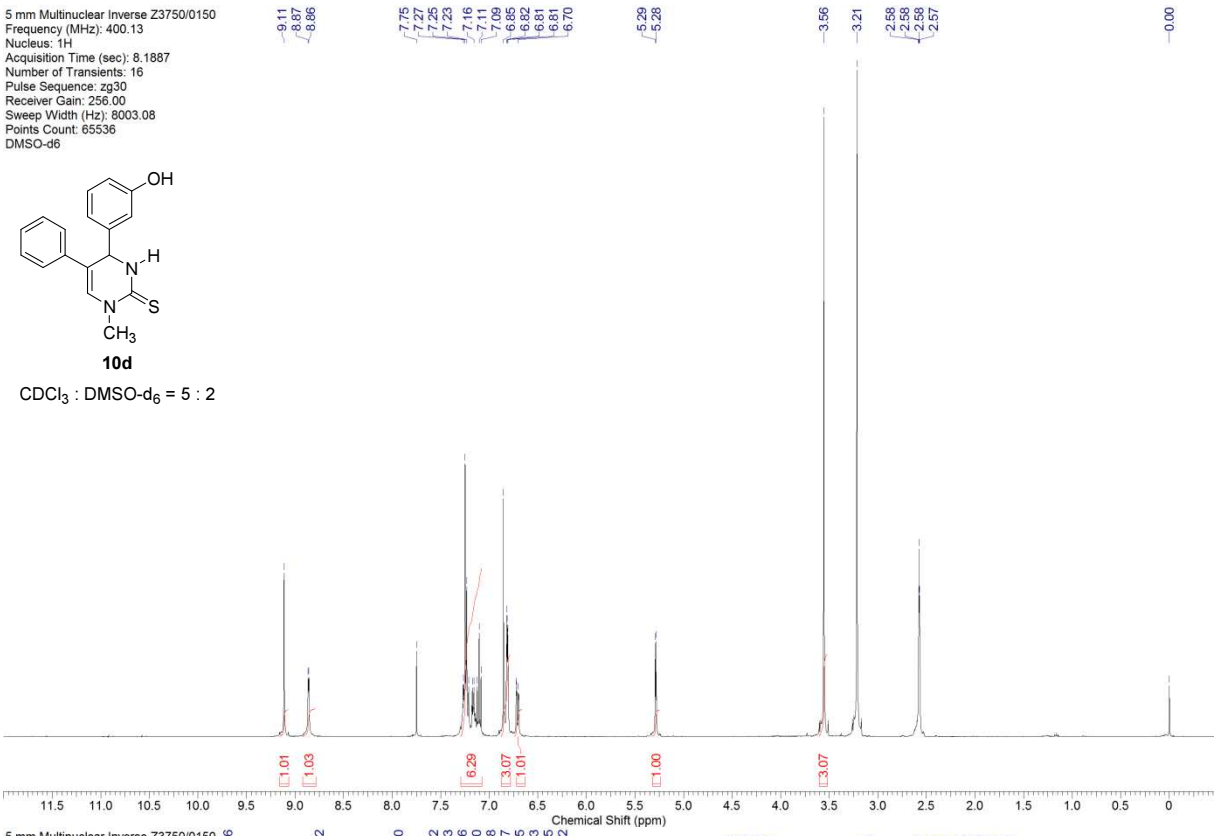
10c

$CDCl_3$: DMSO-d_6 = 5 : 1



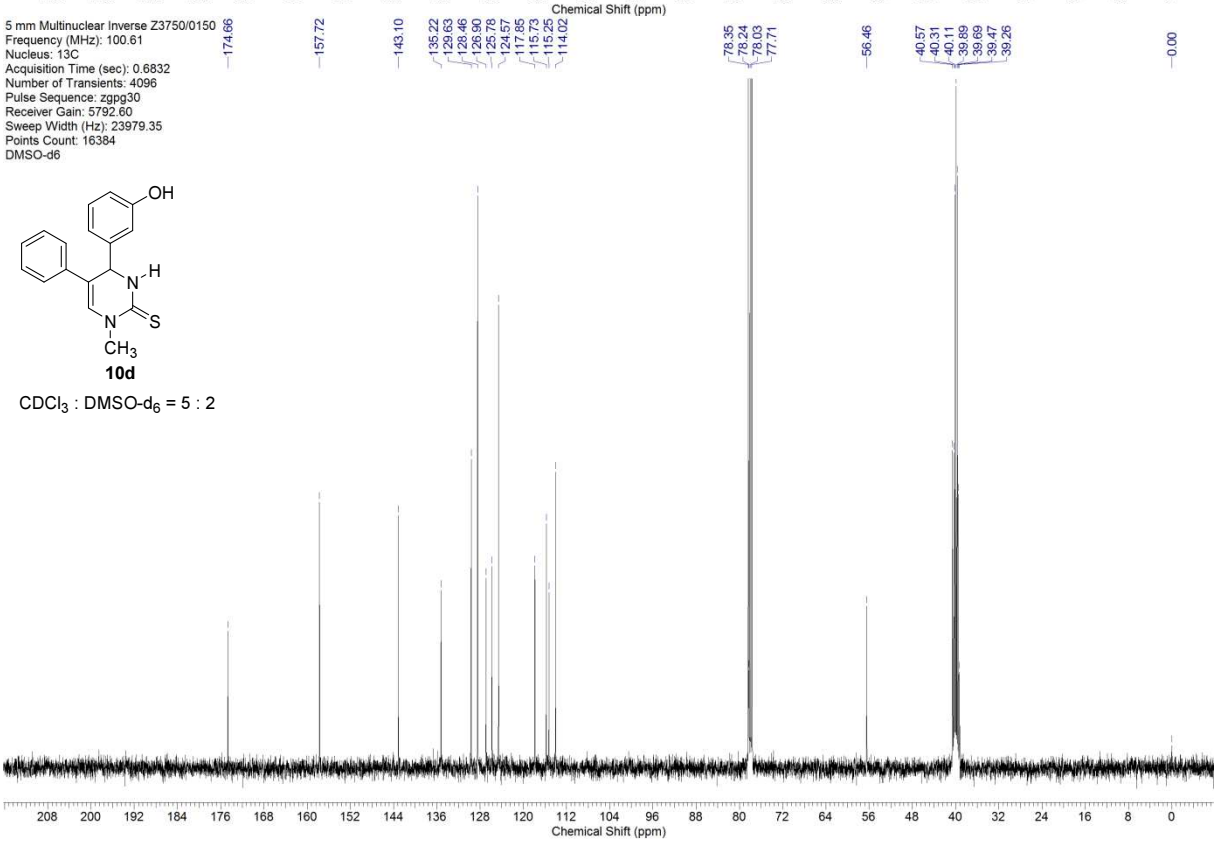
10c

$CDCl_3$: DMSO-d_6 = 5 : 1



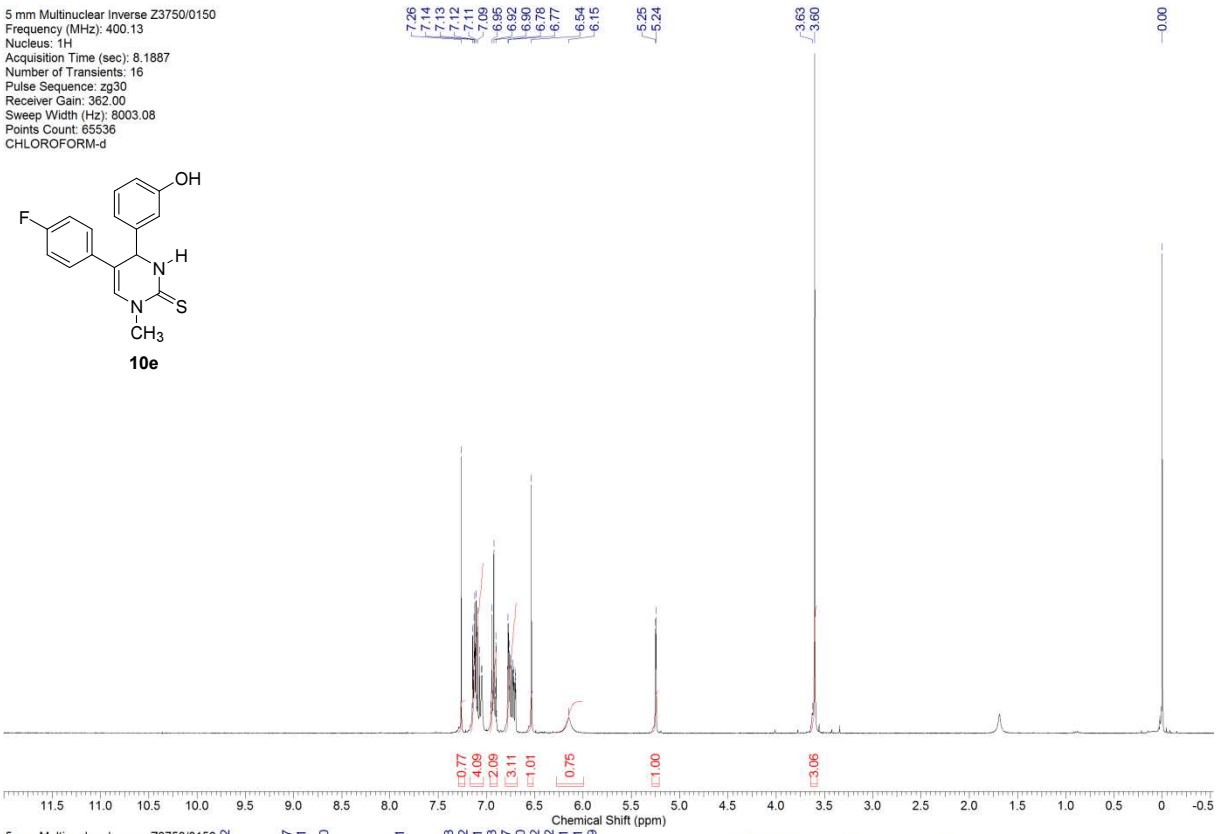
10d

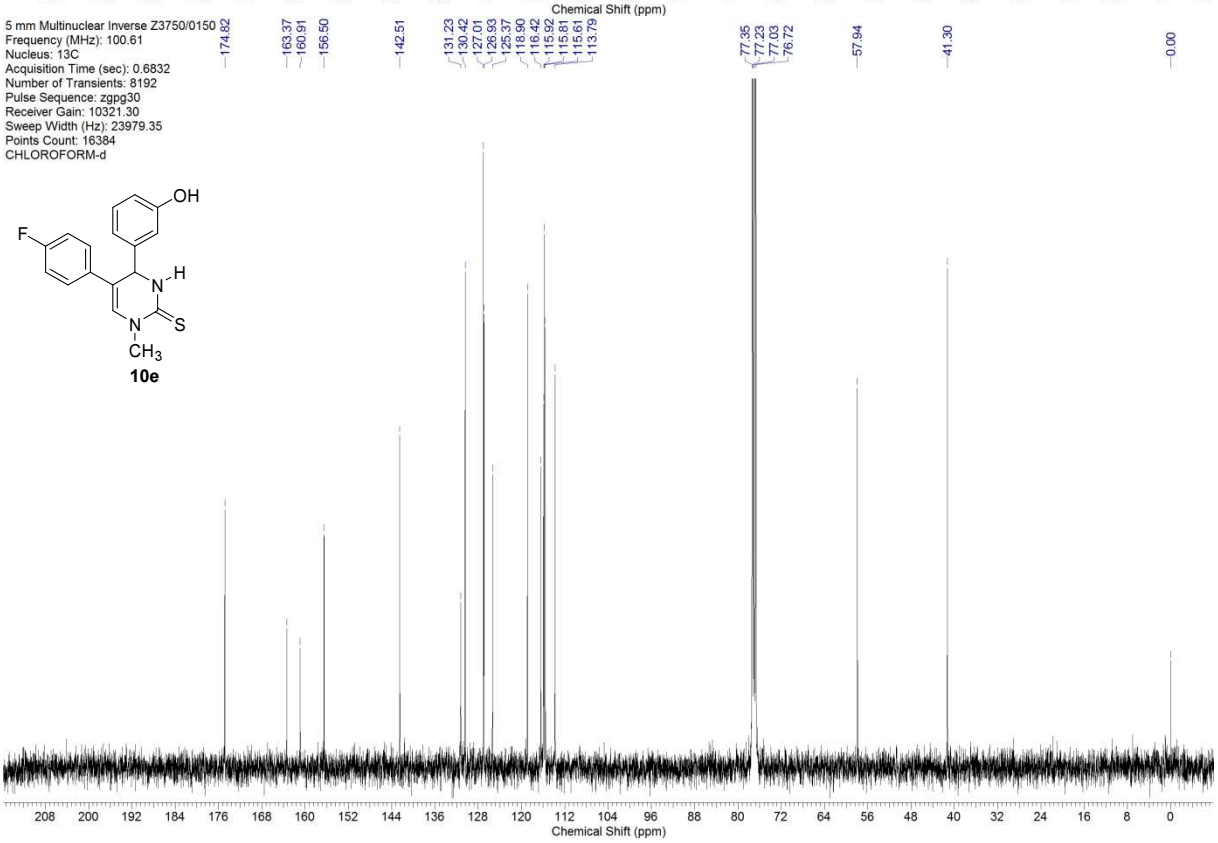
$CDCl_3$: DMSO-d_6 = 5 : 2

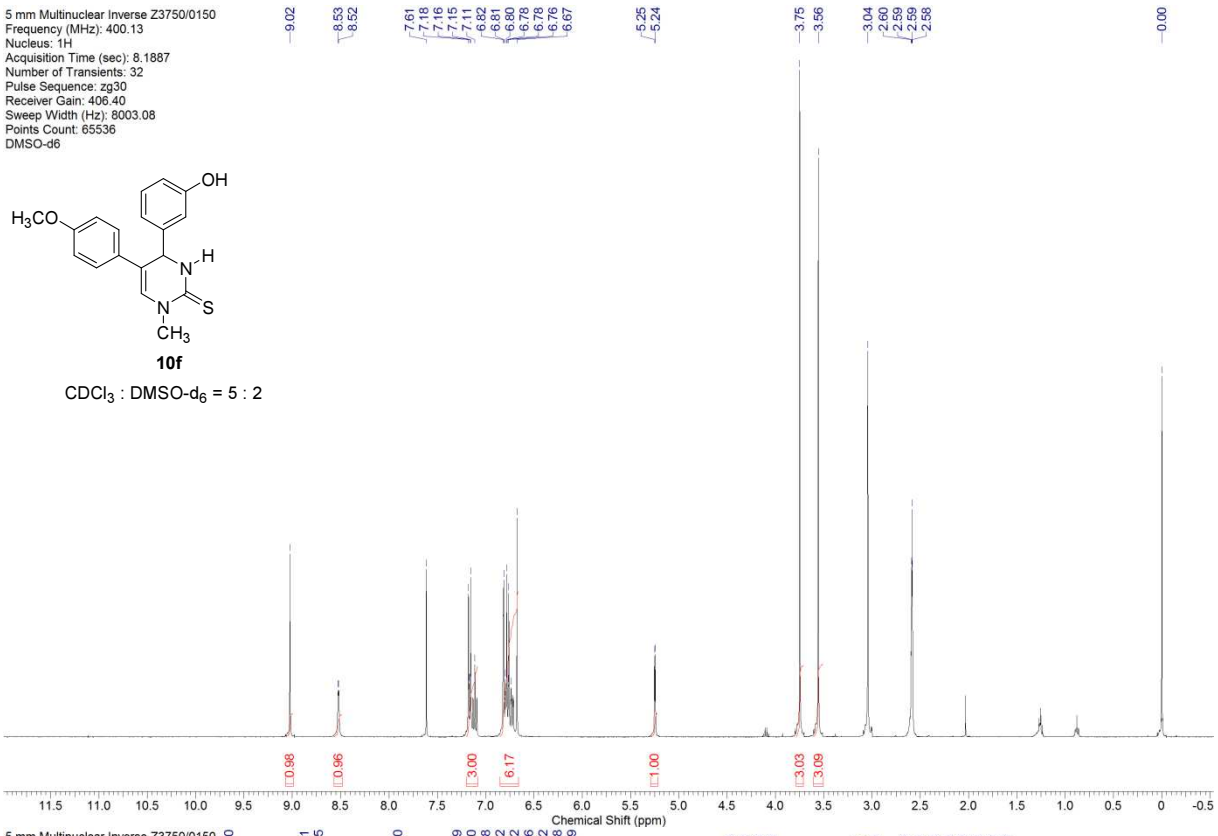


10d

$CDCl_3$: DMSO-d_6 = 5 : 2

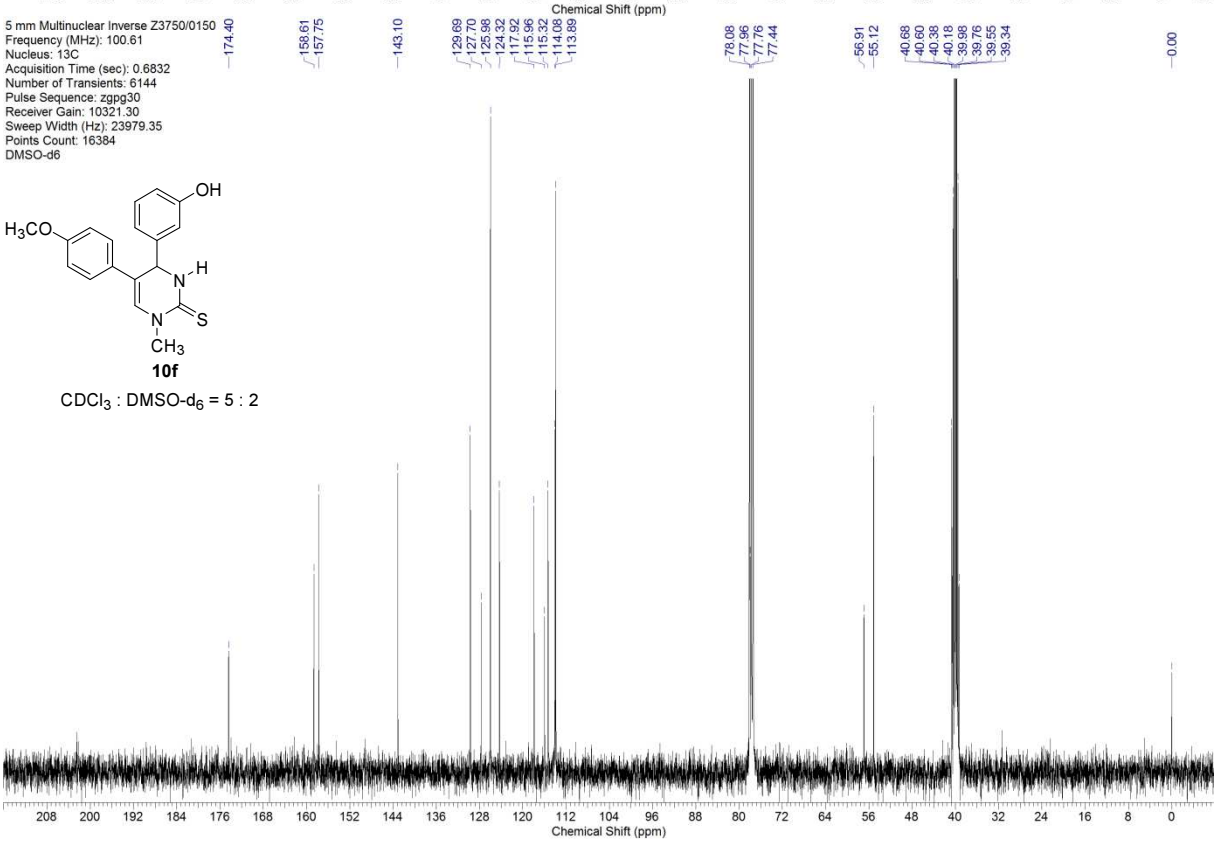

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 362.00
Sweep Width (Hz): 8003.08
Points Count: 65536
CHLOROFORM-d
7.26
7.14
7.13
7.12
7.11
7.09
6.95
6.92
6.90
6.78
6.77
6.54
6.15
5.25
5.24
3.63
3.60
0.00
0.77
4.09
2.09
3.11
1.01
0.75
1.00
3.06
Chemical Shift (ppm)
OH
F
H
N
S
N
CH3
10e


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 8192
Pulse Sequence: zgpg30
Receiver Gain: 10321.30
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
174.62
163.37
160.91
156.50
142.51
131.23
130.42
127.01
126.93
125.37
118.90
116.42
115.92
115.81
115.61
113.79
77.35
77.23
77.03
76.72
57.94
41.30
0.00
Chemical Shift (ppm)
OH
F
H
N
S
N
CH3
10e



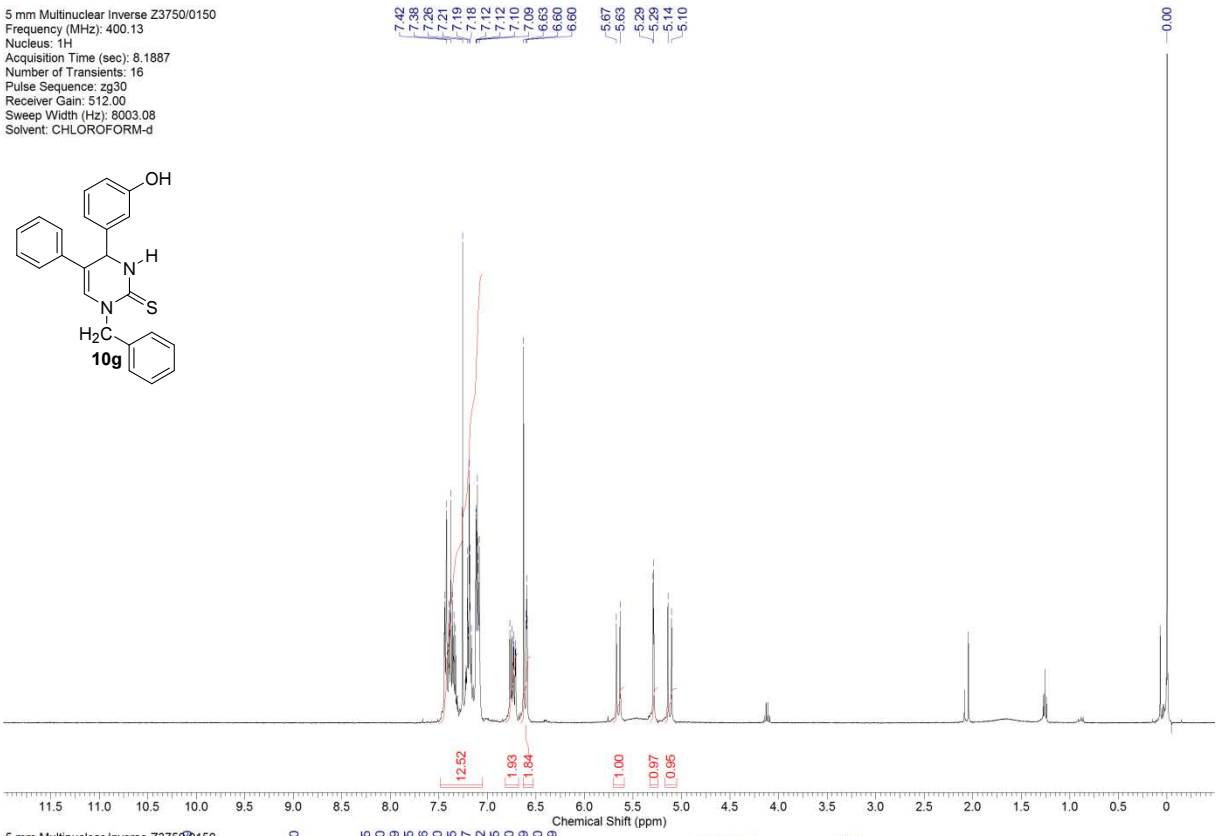
10f

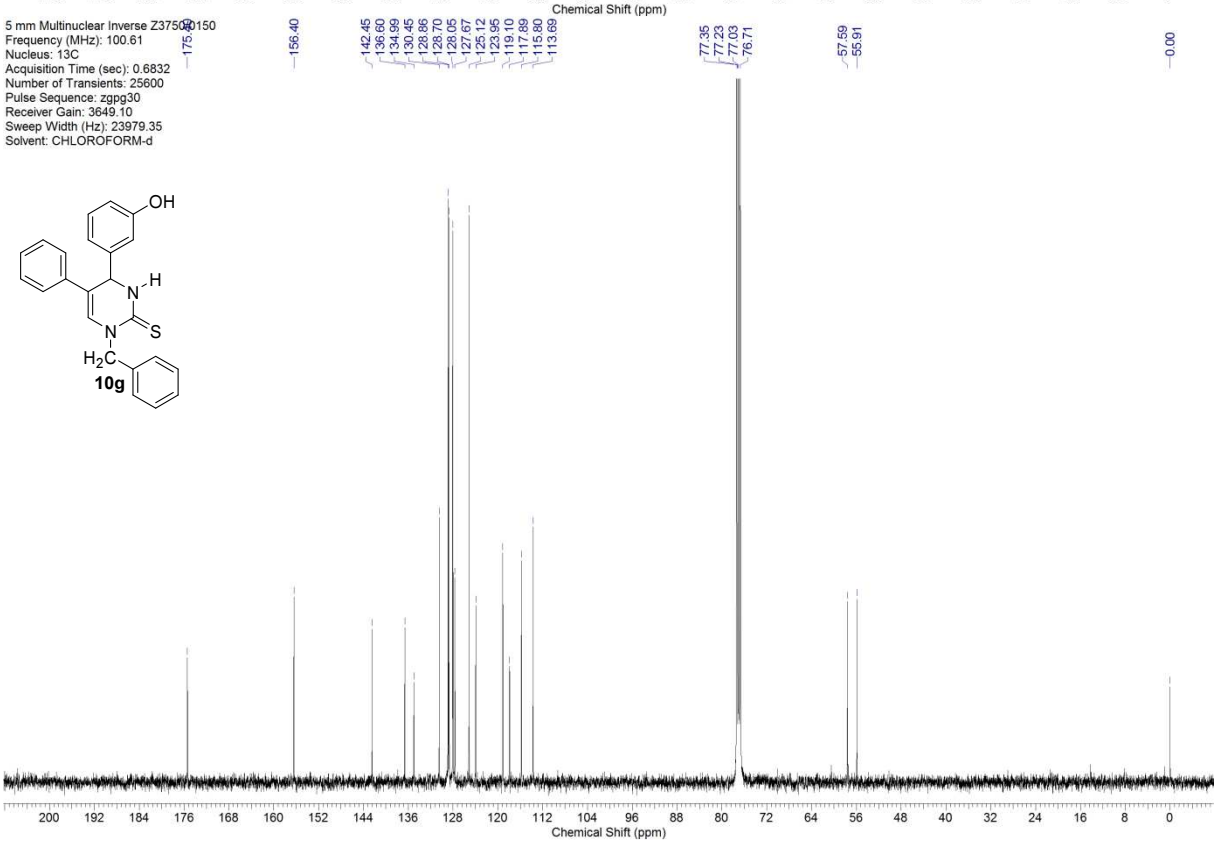
$CDCl_3$: DMSO-d_6 = 5 : 2

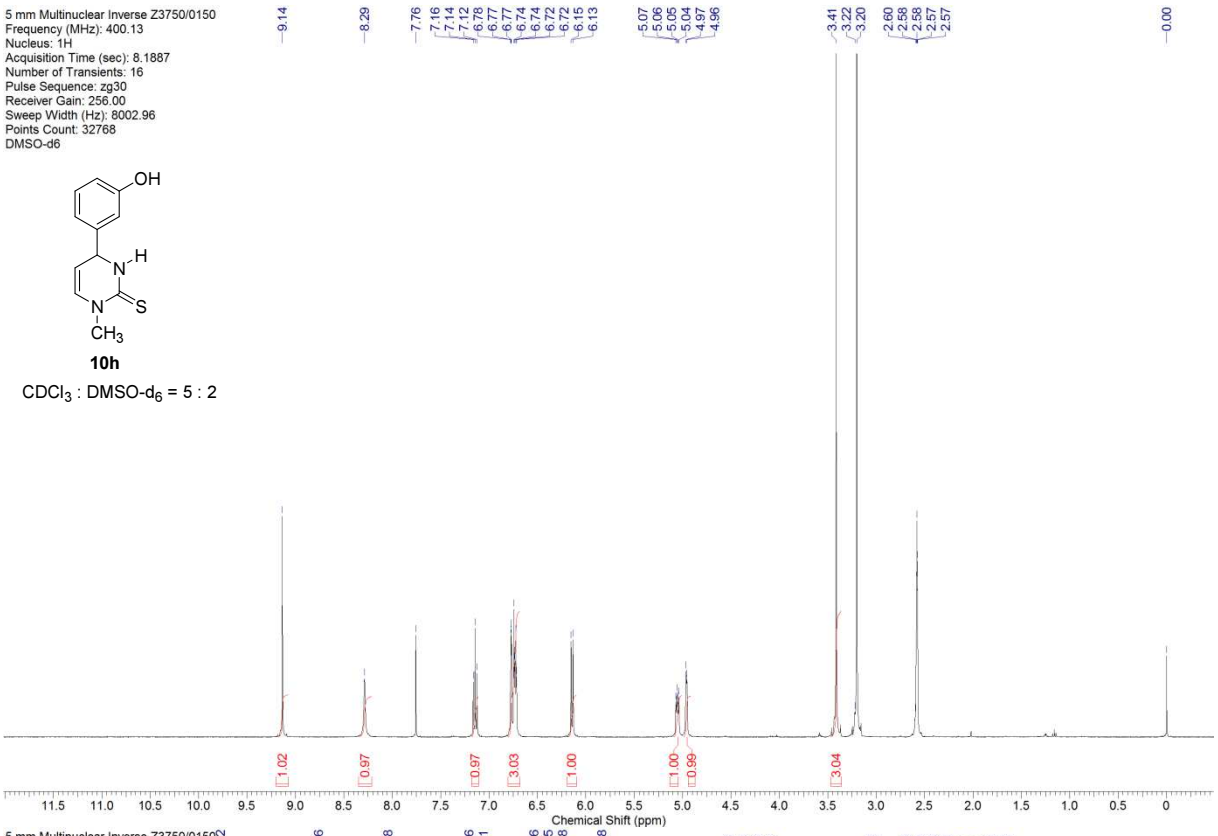


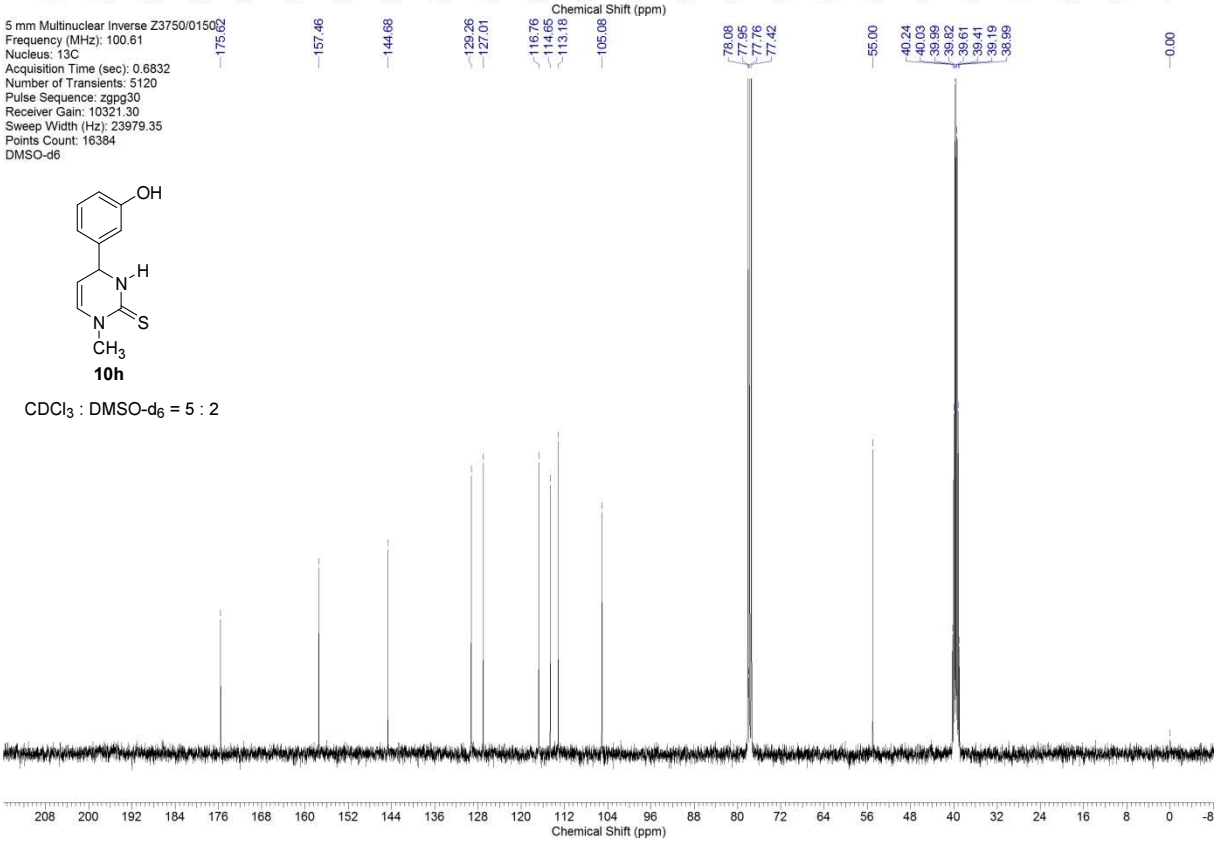
10f

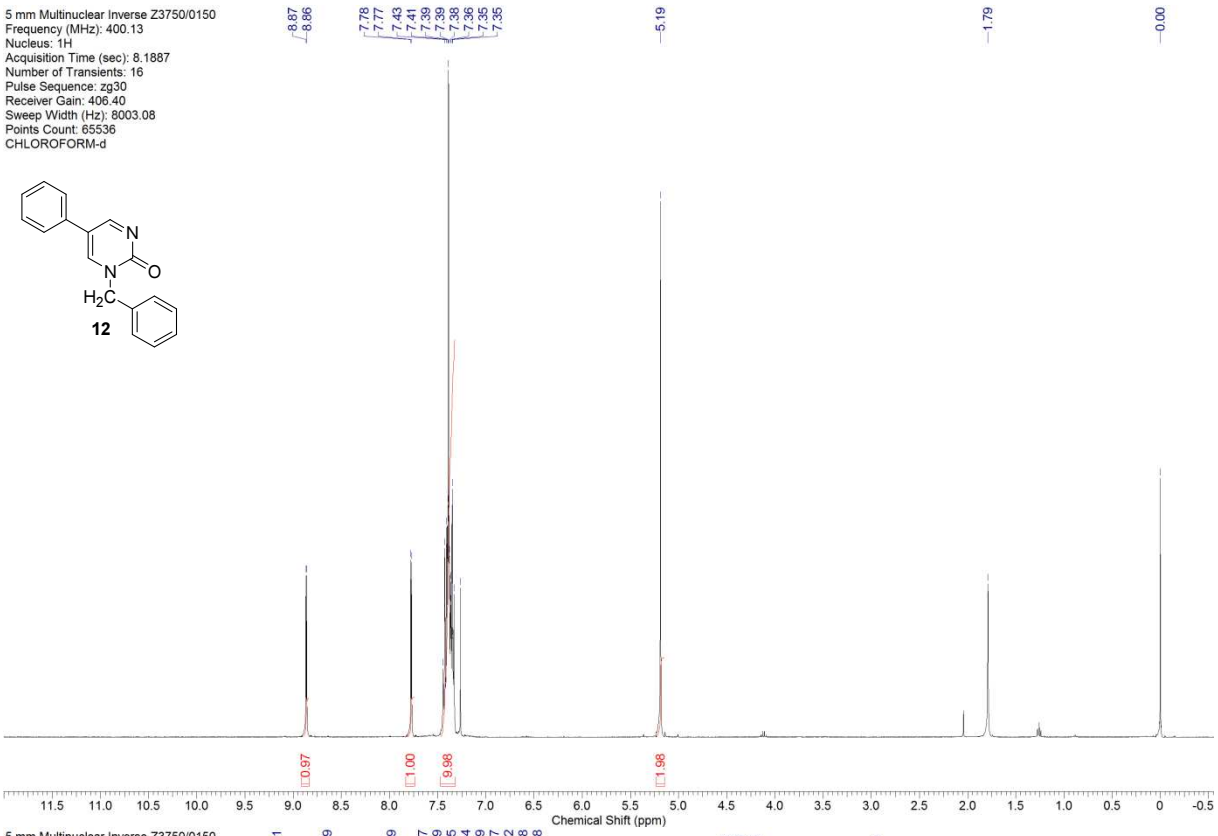
$CDCl_3$: DMSO-d_6 = 5 : 2

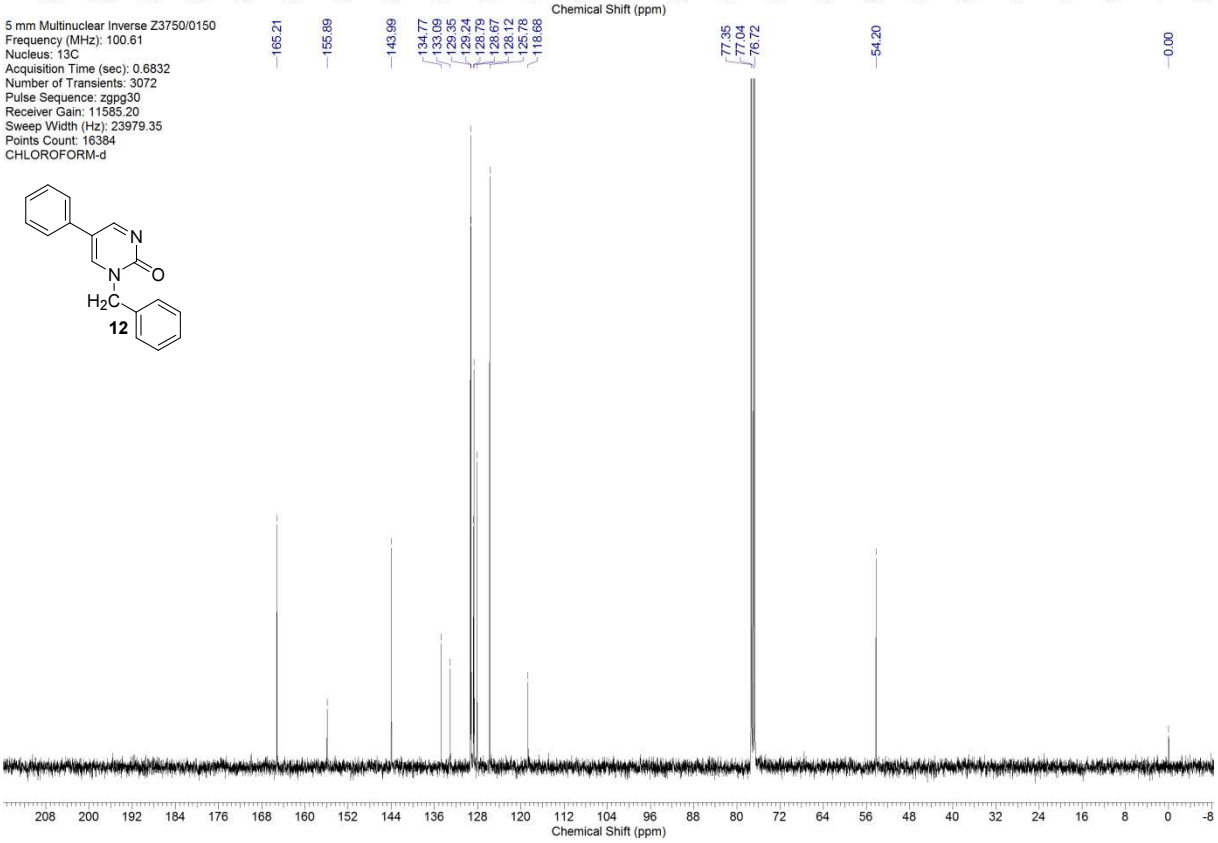

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 512.00
Sweep Width (Hz): 8003.08
Solvent: CHLOROFORM-d
OH
N
H
N
S
H2C
10g
7.42
7.38
7.26
7.21
7.19
7.18
7.12
7.12
7.10
7.09
6.63
6.60
6.60
5.67
5.63
5.29
5.29
5.14
5.10
0.00
12.52
1.93
1.84
1.00
0.97
0.95
Chemical Shift (ppm)

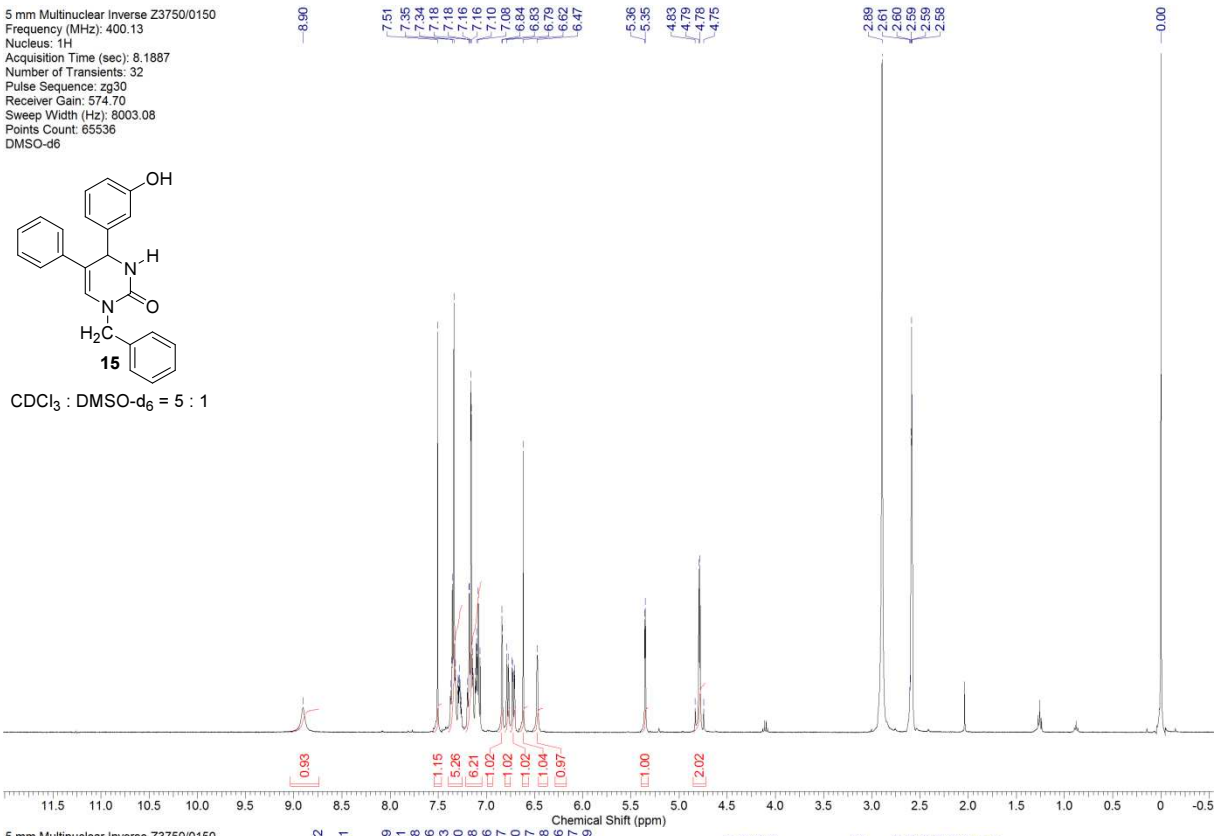

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 25600
Pulse Sequence: zgpg30
Receiver Gain: 3649.10
Sweep Width (Hz): 23979.35
Solvent: CHLOROFORM-d
OH
N
H
N
S
H2C
10g
175.30
156.40
142.45
138.60
134.99
130.45
128.86
128.70
128.05
127.67
125.12
123.95
119.10
117.89
115.80
113.69
77.35
77.23
77.03
76.71
57.59
55.91
0.00
Chemical Shift (ppm)

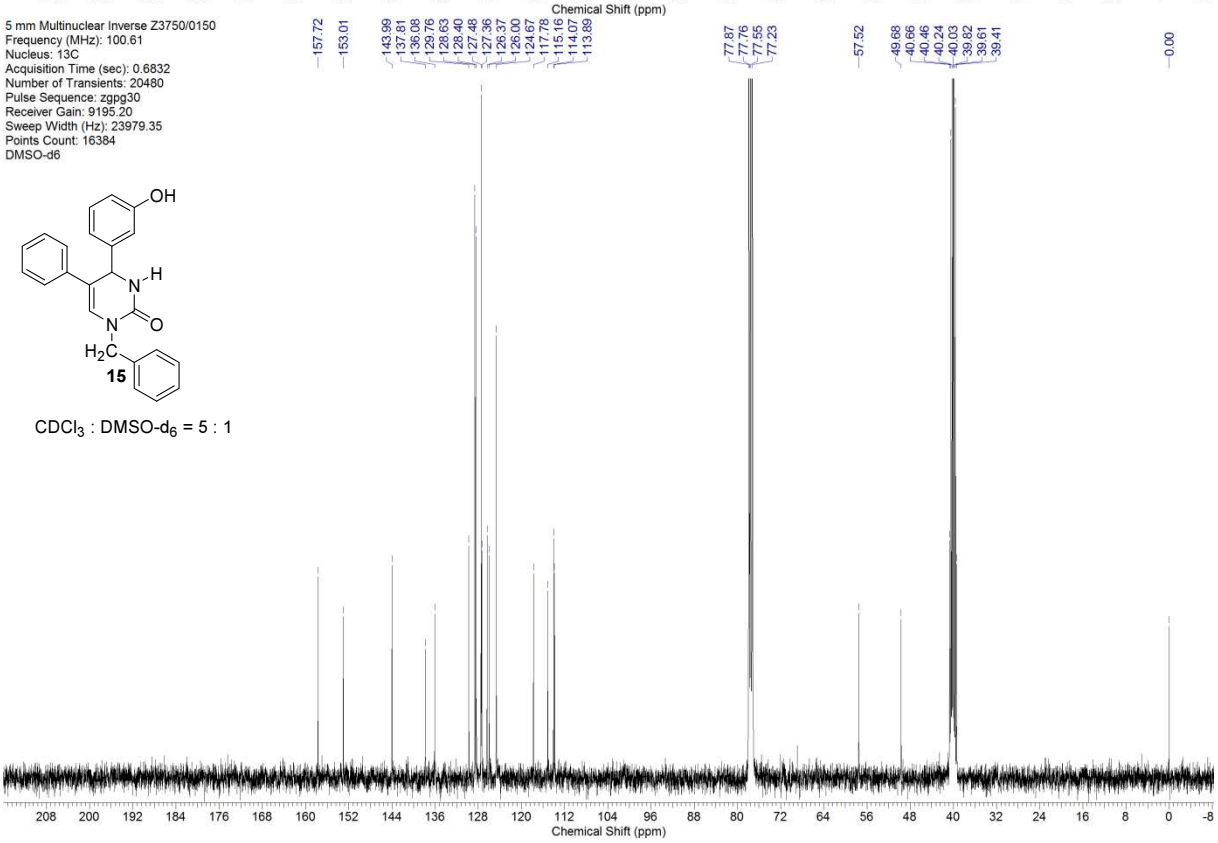

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 256.00
Sweep Width (Hz): 8002.96
Points Count: 32768
DMSO-d6
OH
H
N
N
S
CH3
10h
CDCl3 : DMSO-d6 = 5 : 2
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 5120
Pulse Sequence: zgpg30
Receiver Gain: 10321.30
Sweep Width (Hz): 23979.35
Points Count: 16384
DMSO-d6
OH
H
N
N
S
CH3
10h
CDCl3 : DMSO-d6 = 5 : 2
Chemical Shift (ppm)

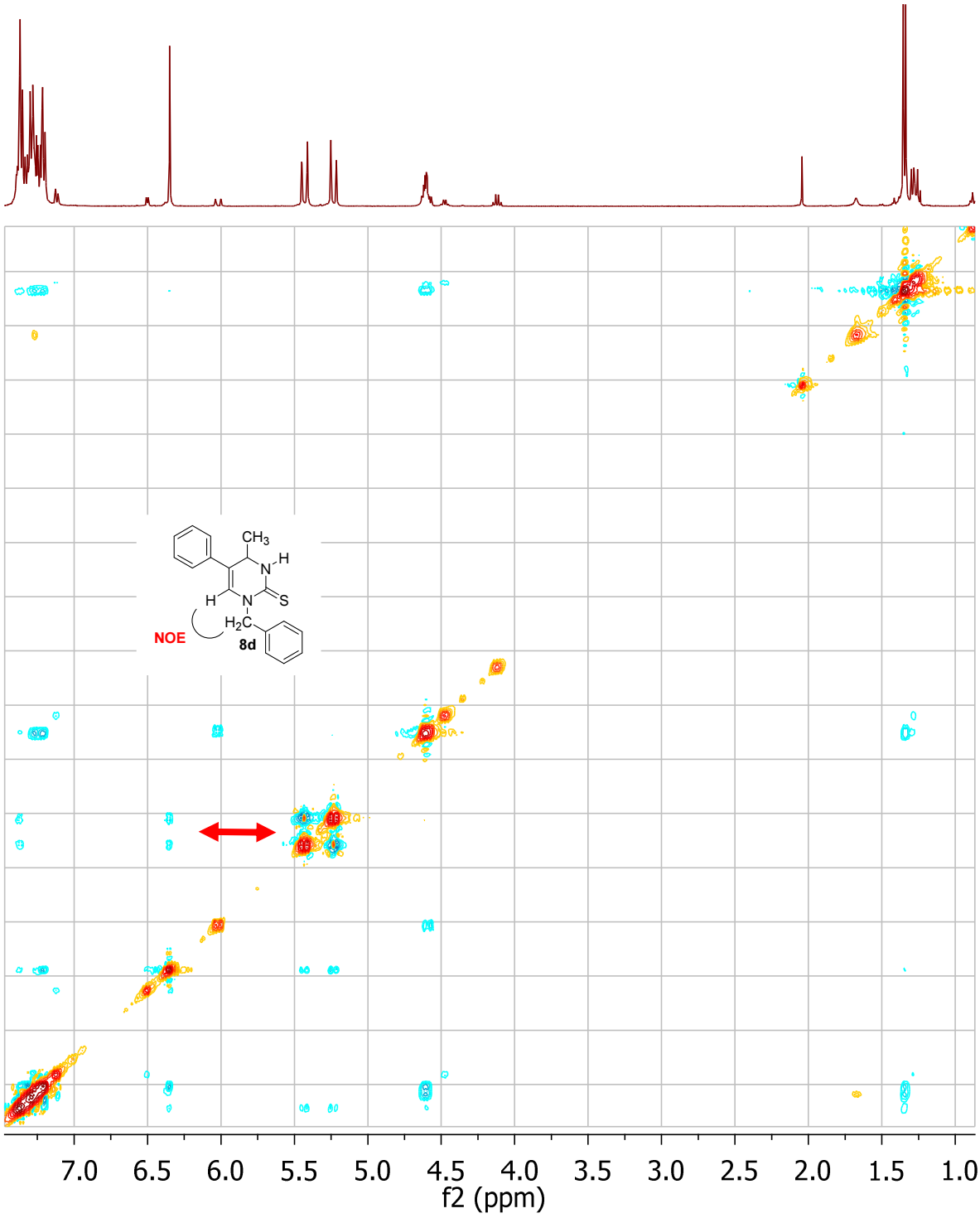

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 16
Pulse Sequence: zg30
Receiver Gain: 406.40
Sweep Width (Hz): 8003.08
Points Count: 65536
CHLOROFORM-d
8.87
8.86
7.78
7.77
7.43
7.41
7.39
7.39
7.38
7.36
7.35
7.35
5.19
1.79
0.00
0.97
1.00
9.98
1.98
Chemical Shift (ppm)
12


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 3072
Pulse Sequence: zgpg30
Receiver Gain: 11585.20
Sweep Width (Hz): 23979.35
Points Count: 16384
CHLOROFORM-d
165.21
155.89
143.99
134.77
133.09
129.35
129.24
128.79
128.67
128.12
125.78
118.68
77.35
77.04
76.72
54.20
0.00
Chemical Shift (ppm)
12

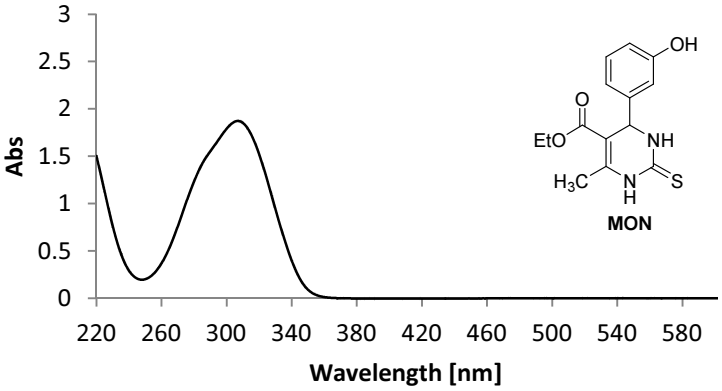

5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 400.13
Nucleus: 1H
Acquisition Time (sec): 8.1887
Number of Transients: 32
Pulse Sequence: zg30
Receiver Gain: 574.70
Sweep Width (Hz): 8003.08
Points Count: 65536
DMSO-d6
OH
H
N
O
N
H2C
15
CDCl3 : DMSO-d6 = 5 : 1
Chemical Shift (ppm)


5 mm Multinuclear Inverse Z3750/0150
Frequency (MHz): 100.61
Nucleus: 13C
Acquisition Time (sec): 0.6832
Number of Transients: 20480
Pulse Sequence: zgpg30
Receiver Gain: 9195.20
Sweep Width (Hz): 23979.35
Points Count: 16384
DMSO-d6
OH
H
N
O
N
H2C
15
CDCl3 : DMSO-d6 = 5 : 1
Chemical Shift (ppm)

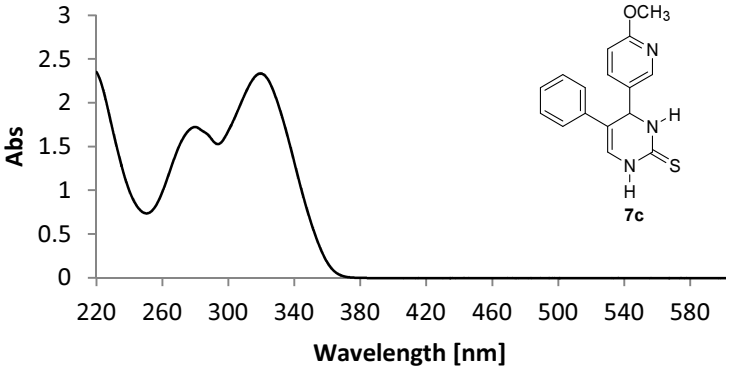
^{1}H, ^{1}H NOESY spectrum of **8d**



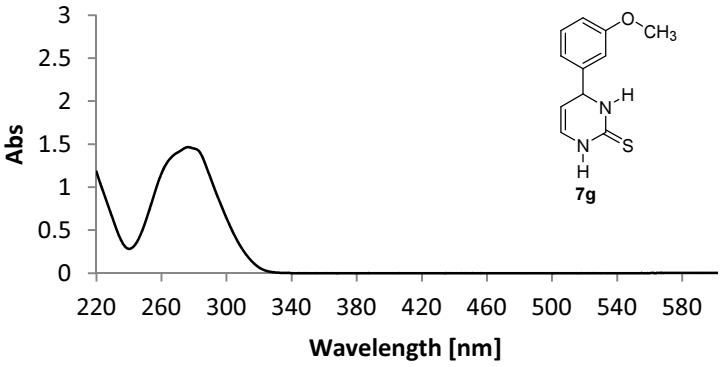
UV-VIS Spectra



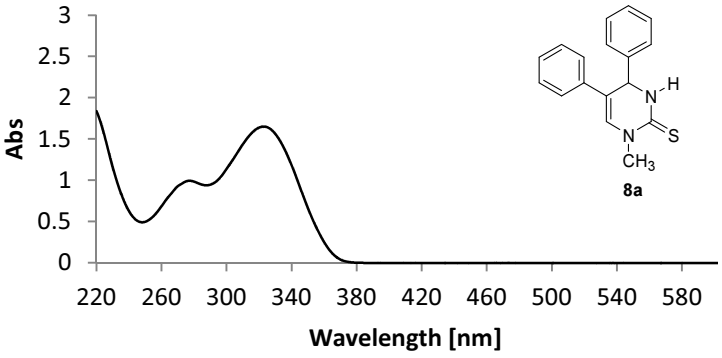
MON (λ_{max} = 306.8 nm)



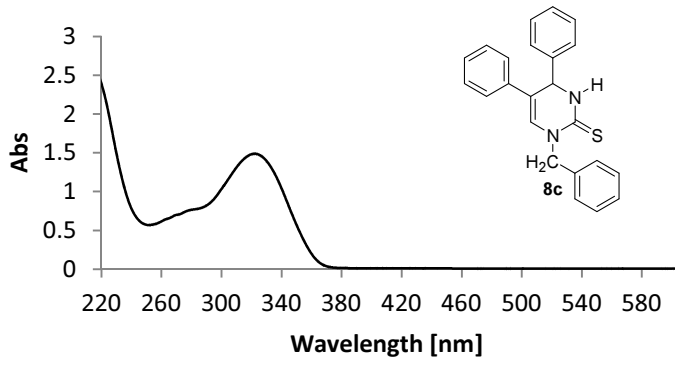
7c (λ_{max} = 320 nm)



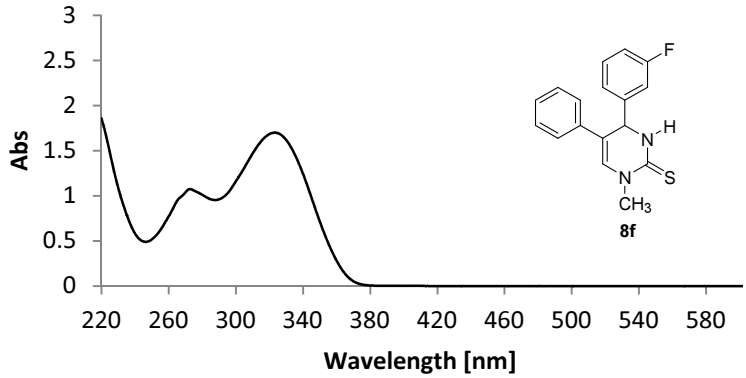
7g (λ_{max} = 276.3 nm)



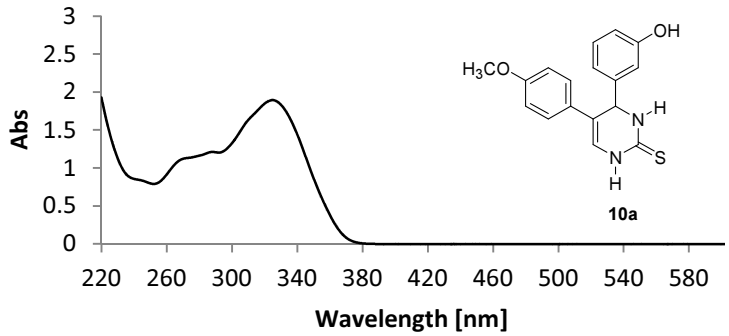
8a (λ_{max} = 322.8 nm)



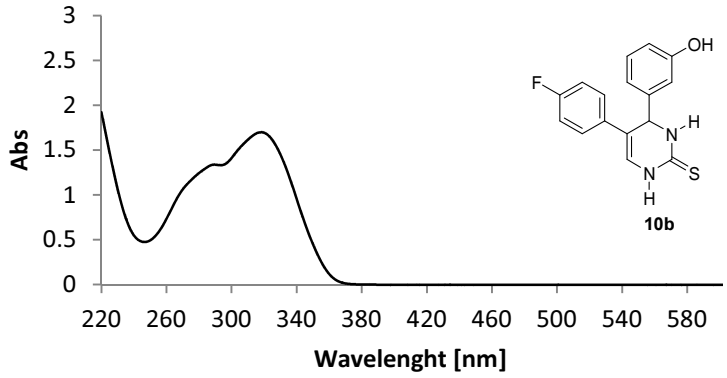
8c (λ_{max} = 322.1 nm)



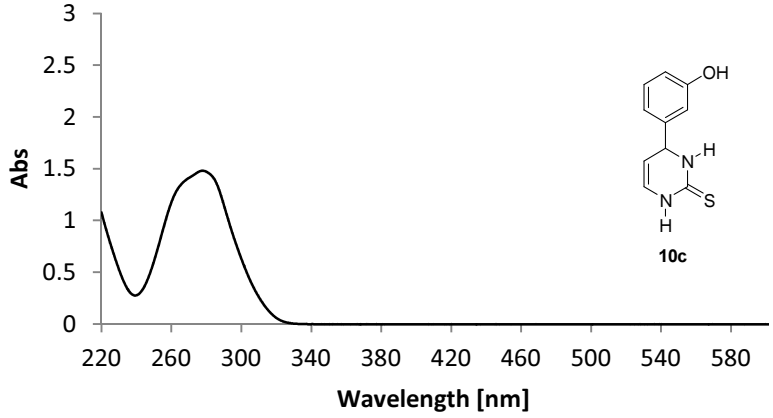
8f (λ_{max} = 323.2 nm)



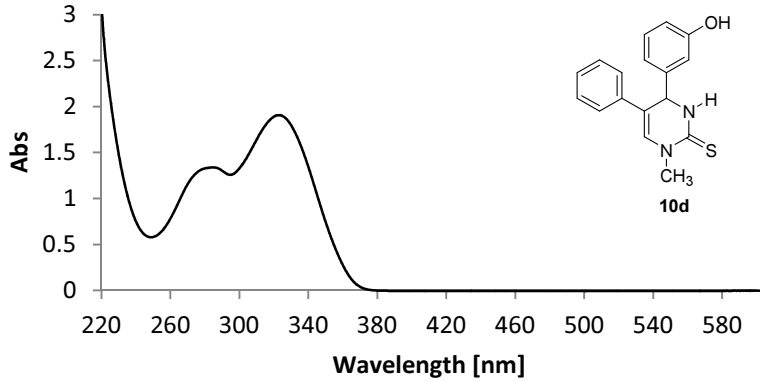
10a (λ_{max} = 324.3 nm)



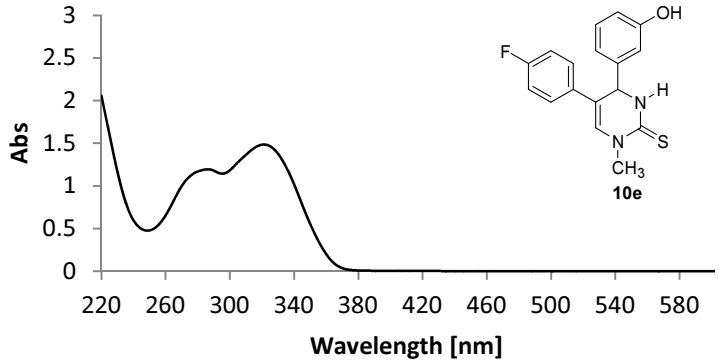
10b (λ_{max} = 318.5 nm)



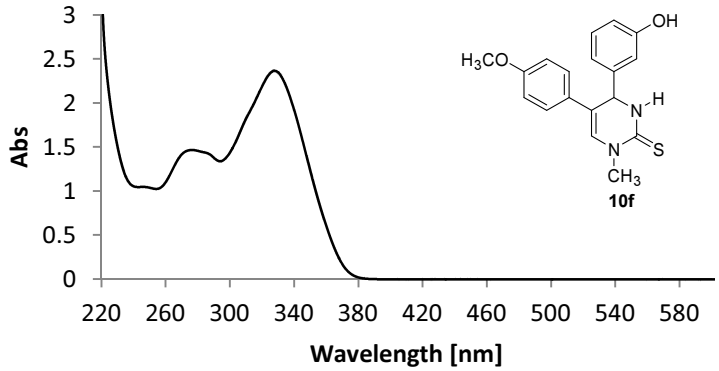
10c (λ_{max} = 277.7 nm)



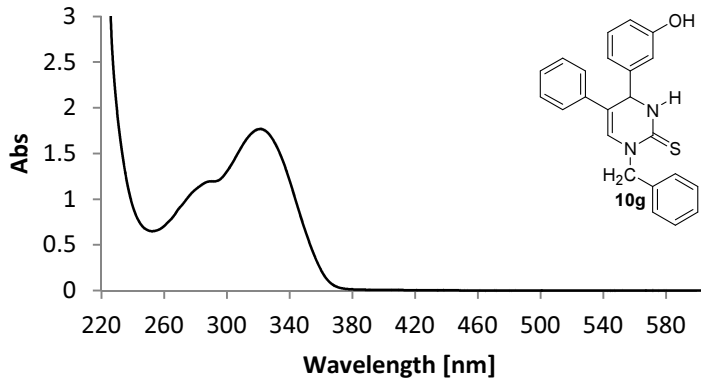
10d (λ_{max} = 322.7 nm)



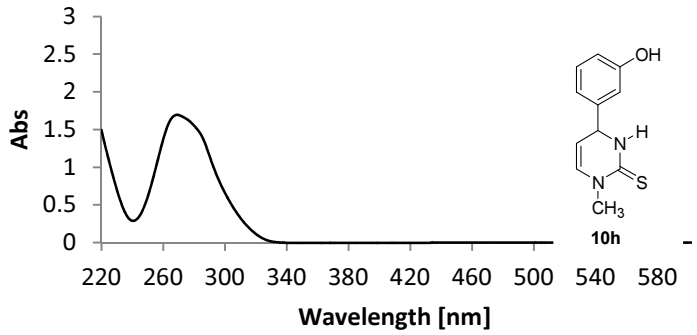
10e (λ_{max} = 321.1 nm)



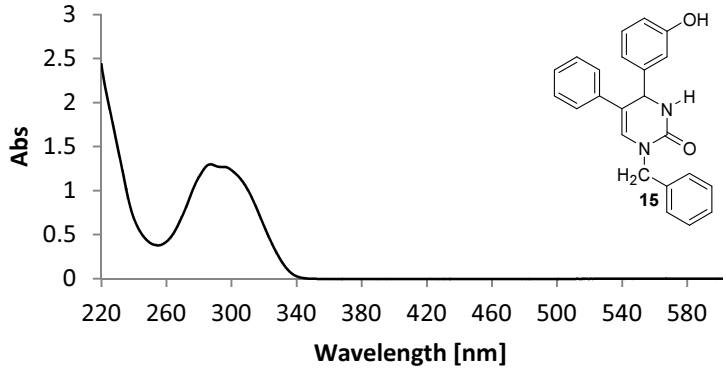
10f (λ_{max} = 327.6 nm)



10g (λ_{max} = 321.6 nm)



10h (λ_{max} = 269 nm)



15 (λ_{max} = 287.1 nm)