Copper-catalyzed high selectively synthesis of 2-benzylbenzo[b]thiazinones and 2-benzylidenebenzo[b]thiazinones from 2-iodophenyl cinnamamides and potassium sulfide

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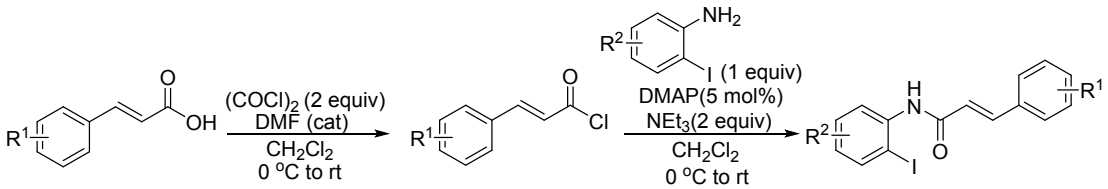
Supporting Information

1) General Information

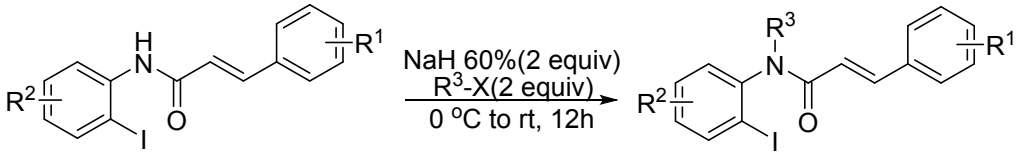
NMR spectra of the products 2a-2s, 2u, 2v, 3a were obtained using Bruker Avance-500 instruments, calibrated to TMS (^{1}H NMR spectra) and CD(H)Cl_3 (^{13}C NMR spectra) as the internal reference (0.00 ppm for ^{1}H NMR spectra and 77.00 ppm for ^{13}C NMR spectra). NMR spectra of the products 2t, 3t-3tg were recorded using Bruker Avance-500 instruments, calibrated to residual DMSO-d_6 as the internal reference (2.50 ppm for ^{1}H NMR spectra and 40.00 ppm for ^{13}C NMR spectra). High-resolution massspectra. (HRMS) were recorded on a Bruker Apex IV FTMS mass spectrometer using ESI (electrospray ionization). Reactions were monitored by thin-layer chromatography. Column chromatography (petroleum ether/ethyl acetate) was performed on silica gel(200-300 mesh). Unless stated otherwise, all reagents were used as received and the following reaction solvents were distilled under anhydrous conditions over the appropriate drying agent and transferred under argon via a syringe. Dichloromethane was distilled over CaH_2, tetrahydrofuran was distilled over Na and benzophenone.

2) Synthesis of Starting Materials[1]



A solution of substituted cinnamic acid (1.0 equiv) and DMF (2~3 drops) in CH_2Cl_2 was prepared and cooled to 0 °C. A bubbler was attached to the vessel and $(COCl)_2$ (2 equiv) was added dropwise. After 5 minutes, the reaction was allowed to warm to room temperature and was stirred for 1 hour. The acyl chloride was concentrated *in* vacuum and redissolved in CH_2Cl_2. A solution of the substituted 2-iodoaniline (1.0 equiv), DMAP (0.05 equiv) and NEt_3 (2.0 equiv) was prepared in CH_2Cl_2 and cooled to 0 °C. The acyl chloride solution was added dropwise into the vessel containing the substituted 2-iodoaniline. After 5 minutes, the reaction was allowed to warm to room temperature and was stirred 5 hours. The reaction was quenched with a saturated $NaHCO_3$ solution and extracted with CH_2Cl_2 (3 x 15 mL). The combined extracts

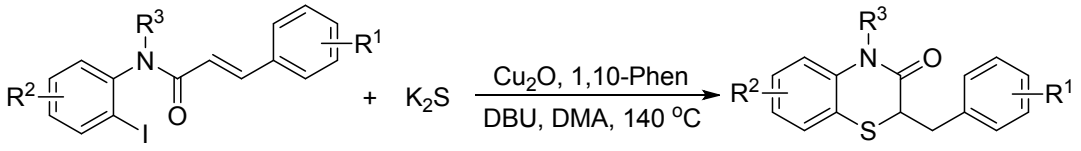
were washed with brine, dried over Na_2SO_4, concentrated under reduced pressure and purified by column chromatography to afford the pure product.



A solution of the unsubstituted acrylamide (1.0 equiv) in THF was prepared and cooled to 0 °C. NaH (60%, 2.0 equiv) was added to the solution and the mixture was stirred for 15 minutes before adding R_3-X (2 equiv) dropwise. The reaction was allowed to warm at room temperature after 10 minutes and was stirred for 2 hours to overnight. The reaction was quenched with cold water and extracted with EtOAc (3 x 15 mL). The combined extracts were washed with brine, dried over Na_2SO_4, concentrated under reduced pressure and purified by column chromatography to afford the pure product.

3) Typical Procedures

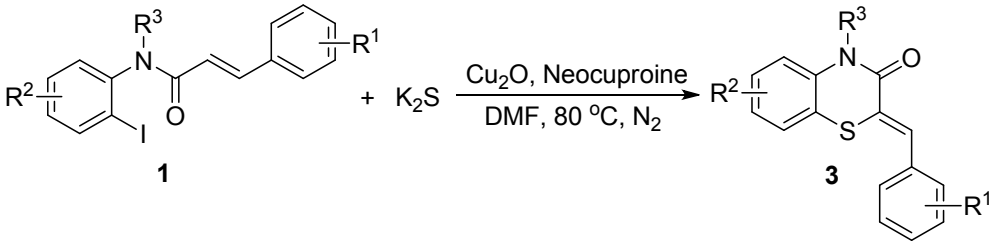
General Procedure for the Synthesis of 2-benzyl-4-methyl-2H-benzo[b][1,4]thia -zin-3(4H)-ones



1 **2**

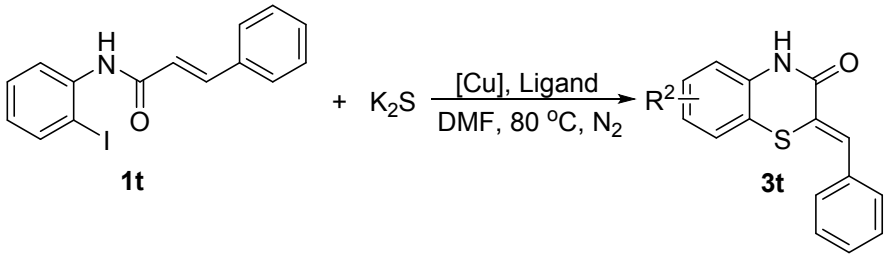
The sealed Schlenk tube was charged with 2-iodophenyl cinnamamides (0.2 mmol), K_2S (3 equiv), Cu_2O (10 mol %), 1,10-Phen (20 mol %), DBU(3 equiv) and DMA (2 mL). Then the mixture was stirred at 140 °C (oil bath temperature). After the reaction was finished, the reaction mixture was cooled to room temperature, quenched by water and extracted with ethyl acetate. The combined organic layer was washed with brine, and dried over Na_2SO_4, and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the pure product.

General Procedure for the Synthesis of (Z)-2-benzylidene-2H-benzo[b][1,4]thiaz- in-3(4H)-ones



The sealed Schlenk tube was charged with 2-iodophenyl cinnamamides (0.2 mmol), K_2S (3 equiv), Cu_2O (10 mol %), Neocuproine (20 mol %), and DMF (2 mL). Then under the protection of nitrogen atmosphere, the mixture was stirred at 80 °C (oil bath temperature). After the reaction was finished, the reaction mixture was cooled to room temperature, quenched by water and extracted with ethyl acetate. The combined organic layer was washed with brine, and dried over Na_2SO_4, and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate/ dichloromethane) to afford the pure product.

Table S1 Optimization of reaction condition for the Synthesis of (Z)-2-benzylidene-2H-benzo[b][1,4]thiazin-3(4H)-ones[a]

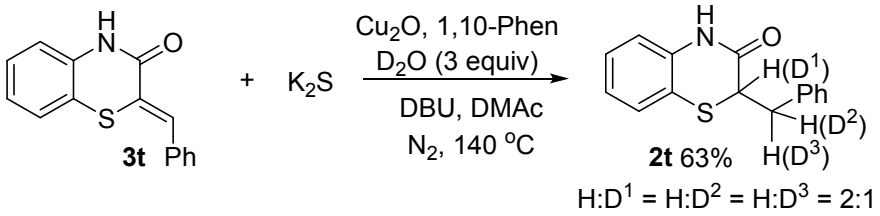


Entry	Catalyst	Ligand	Solvent	S	Yield 3t[b](%)
1	Cu_2O	2,2-py	DMF	K_2S	72%
2	CuBr	2,2-py	DMF	K_2S	50%
3	CuCl	2,2-py	DMF	K_2S	38%
4	CuI	2,2-py	DMF	K_2S	30%
5	CuSCN	2,2-py	DMF	K_2S	34%
6	CuCN	2,2-py	DMF	K_2S	40%
7	$CuBr_2$	2,2-py	DMF	K_2S	46%
8	$Cu(OAc)_2$	2,2-py	DMF	K_2S	47%
9	Cu_2O	—	DMF	K_2S	80%
10	Cu_2O	TMEDA	DMF	K_2S	76%
11	Cu_2O	1,10-phen	DMF	K_2S	79%
12	Cu_2O	Neocuproine	DMF	K_2S	91%

13	Cu_2O	2,2'-Biquinoline	DMF	K_2S	84%
14	Cu_2O	Bathocuproine	DMF	K_2S	81%
15	Cu_2O	Neocuproine	DMSO	K_2S	55%
16	Cu_2O	Neocuproine	NMP	K_2S	50%
17	Cu_2O	Neocuproine	DMA	K_2S	62%
18	Cu_2O	Neocuproine	MeCN	K_2S	48%
19[c]	Cu_2O	Neocuproine	DMF	K_2S	Trace
20[d]	Cu_2O	Neocuproine	DMF	K_2S	53%
21	Cu_2O	Neocuproine	DMF	Na_2S	—
22	Cu_2O	Neocuproine	DMF	Li_2S	Trace
23	Cu_2O	Neocuproine	DMF	S	—
24[e]	Cu_2O	Neocuproine	DMF	K_2S	25%
25[f]	Cu_2O	Neocuproine	DMF	K_2S	Trace

[a] Conditions: **1t** (0.20 mmol), K_2S (0.60 mmol), Cu catalyst (10 mol %), ligand (20 mol %), solvent (2 mL), N_2, at 80 °C for 12 h.[b] Isolated yield. [c] 60 °C. [d] 100 °C. [e] air. [f] O_2.

4) Deuterated Control Experiment



The sealed Schlenk tube was charged with (Z)-2-benzylidene-2H-benzo[b][1,4]thiazin-3(4H)-one **3t** (0.2 mmol), K_2S (3 equiv), DBU (3 equiv), D_2O (3 equiv), Cu_2O (10 mol %), 1,10-Phen (20 mol %), and DMAc (2 mL). Then under the protection of nitrogen atmosphere, the mixture was stirred at 140 °C (oil bath temperature). After the reaction was finished, the reaction mixture was cooled to room temperature, quenched by water and extracted with ethyl acetate. The combined organic layer was washed with brine, and dried over Na_2SO_4, and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate/ dichloromethane) to afford **2t** in 63% yield.

Scanned ^{1}H NMR Spectra of deuterated 2t

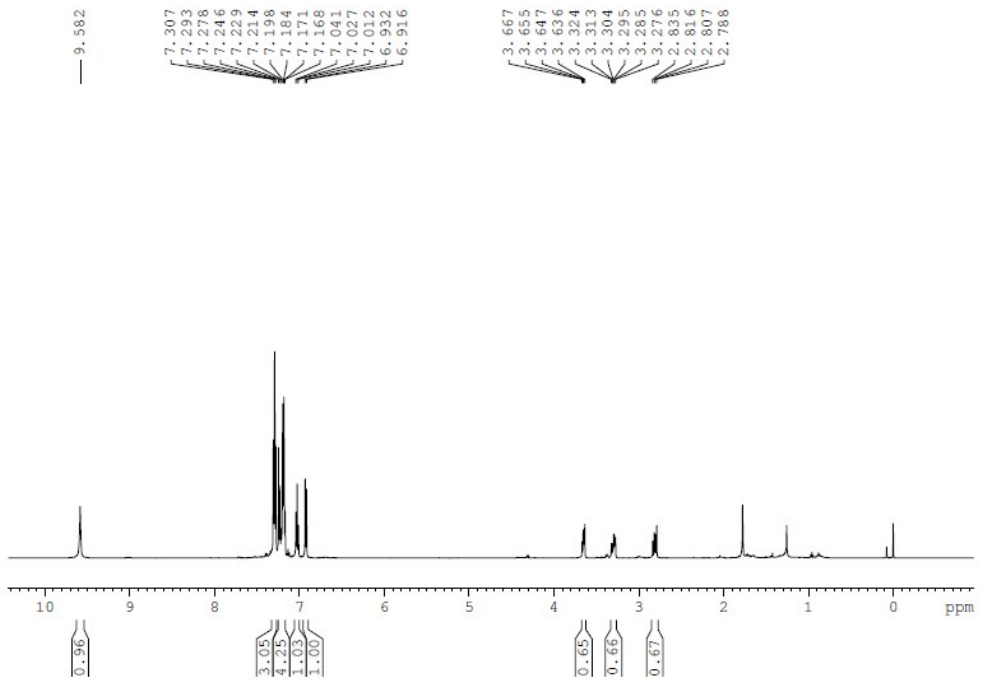
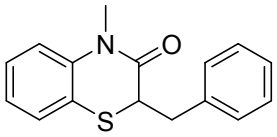
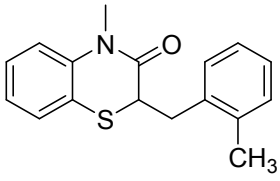


Figure S1 Scanned ^{1}H NMR Spectra of deuterated **2t**

5) Characterization Data

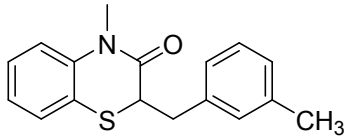


2-benzyl-4-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2a**)[2]: yellow oil (50 mg, 93%); **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.33 (d, *J* = 7.5 Hz, 1H), 7.30-7.22 (m, 4H), 7.14 (d, *J* = 7.0 Hz, 2H), 7.08 (d, *J* = 8.5 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 3.62 (dd, *J* =10.0, 5.0 Hz, 1H), 3.46 (s, 3H), 3.26 (dd, *J* = 14.0, 5.0 Hz, 1H), 2.72 (dd, *J* = 14.5, 10.0 Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 167.0, 139.7, 137.3, 129.2, 128.9, 128.3, 127.2, 126.8, 123.5, 121.4, 117.2, 45.0, 35.0, 32.4.

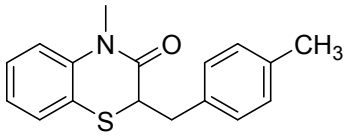


4-methyl-2-(2-methylbenzyl)-2H-benzo[b][1,4]thiazin-3(4H)-one (**2b**): yellow oil (48 mg, 86%); **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.32 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.26 (td, *J* = 8.5, 1.5 Hz, 1H), 7.13-7.12 (m, 3H), 7.08 (d, *J* = 8.0 Hz, 1H), 7.04-7.01 (m, 2H), 3.62

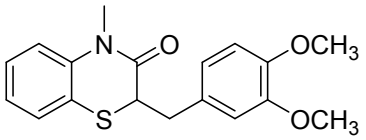
(dd, *J* =10.0, 5.5 Hz, 1H), 3.45 (s, 3H), 3.26 (dd, *J* = 14.5, 5.5 Hz, 1H), 2.74 (dd, *J* = 14.0, 10.0Hz, 1H), 2.26 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.9, 139.7, 136.3, 135.5, 130.3, 130.0, 128.9, 127.1, 126.9, 125.7, 123.4, 121.5, 117.1, 44.0, 32.4, 32.3, 19.2. HRMS (ESI) m/z calcd for $C_{17}H_{18}NOS^+$ $(M+H)^+$ 284.11036, found 284.11014.



4-methyl-2-(3-methylbenzyl)-2H-benzo[b][1,4]thiazin-3(4H)-one (**2c**): yellow oil (49 mg, 87%); **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.32 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.25 (td, *J* = 8.5, 1.5 Hz, 1H), 7.17 (t, *J* = 7.5 Hz, 1H), 7.08-7.01 (m, 3H), 6.69 (s, 1H), 6.93 (d, *J* = 7.5 Hz, 1H), 3.61 (dd, *J* =10.0, 5.0 Hz, 1H), 3.45 (s, 3H), 3.23 (dd, *J* = 14.0, 5.0 Hz, 1H), 2.69 (dd, *J* = 14.0, 10.0Hz, 1H), 2.32 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 167.0, 139.8, 137.9, 137.2, 129.9, 128.8, 128.2, 127.6, 127.1, 126.2, 123.4, 121.6, 117.1, 45.0, 35.0, 32.3, 21.3. HRMS (ESI) m/z calcd for $C_{17}H_{18}NOS^+$ $(M+H)^+$ 284.11036, found 284.11063.

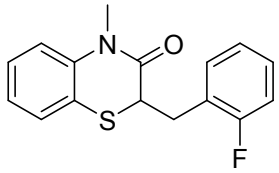


4-methyl-2-(4-methylbenzyl)-2H-benzo[b][1,4]thiazin-3(4H)-one (**2d**): yellow oil (46 mg, 81%); **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.32 (d, *J* = 8.0 Hz, 1H), 7.25 (t, *J* = 7.0 Hz, 1H), 7.10-7.06 (m, 3H), 7.03-7.00 (m, 3H), 3.59 (dd, *J* =10.0, 5.0 Hz, 1H), 3.45 (s, 3H), 3.21 (dd, *J* = 14.0, 5.5 Hz, 1H), 2.67 (dd, *J* = 14.5, 10.0Hz, 1H), 2.31 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 167.0, 139.7, 136.3, 134.2, 129.0(2C), 128.8, 127.1, 123.4, 121.5, 117.1, 45.1, 34.6, 32.3, 21.0. HRMS (ESI) m/z calcd for $C_{17}H_{18}NOS^+$ $(M+H)^+$ 284.11036, found 284.11038.

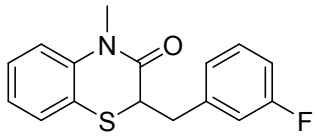


2-(3,4-dimethoxybenzyl)-4-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2e**): yellow solid (45 mg,69%); mp: 97.1-98.5 ^{o}C **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.33 (d, *J* = 7.5 Hz, 1H), 7.26 (t, *J* = 7.5 Hz, 1H), 7.08 (d, *J* = 8.0 Hz, 1H), 7.04 (t, *J* = 8.0 Hz, 1H),

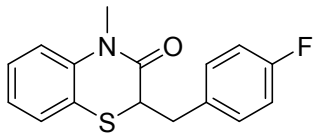
6.78 (d, *J* = 8.5 Hz, 1H), 6.68 (s, 2H), 3.85 (s, 3H), 3.84 (s, 3H), 3.61 (dd, *J* =10.0, 5.0 Hz, 1H), 3.46 (s, 3H), 3.20 (dd, *J* = 14.0, 5.0 Hz, 1H), 2.69 (dd, *J* = 14.5, 10.0Hz, 1H), 2.29 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 167.0, 148.6, 147.8, 139.7, 129.7, 128.7, 127.1, 123.4, 121.4, 121.3, 117.1, 112.3, 110.9, 55.7(2C), 45.1, 34.6, 32.3. HRMS (ESI) m/z calcd for $C_{18}H_{20}NO_3S^+$ $(M+H)^+$ 330.11584, found 330.11594.



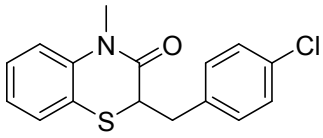
2-(2-fluorobenzyl)-4-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2f**): yellow oil (53 mg, 93%); **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.31 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.26 (td, *J* = 8.5, 1.5 Hz, 1H), 7.23-7.19 (m, 1H), 7.11 (td, *J* = 7.5, 1.5 Hz, 1H), 7.09-6.98 (m, 4H), 3.70 (dd, *J* =9.5, 6.0 Hz, 1H), 3.45 (s, 3H), 3.30 (dd, *J* = 14.0, 6.0 Hz, 1H), 2.80 (dd, *J* = 14.0, 9.5Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.6, 161.2 (d, *J* = 244.6 Hz, 1C), 139.6, 131.3 (d, *J* = 4.4 Hz, 1C), 128.9, 128.7 (d, *J* = 8.1 Hz, 1C), 127.2, 124.3 (d, *J* = 15.5 Hz, 1C), 123.9 (d, *J* = 3.5 Hz, 1C), 123.4, 121.2, 117.1, 115.2 (d, *J* = 21.8 Hz, 1C), 43.4, 32.3, 29.0 (d, *J* = 1.6 Hz, 1C). HRMS (ESI) m/z calcd for $C16H15FNOS^+$ $(M+H)^+$ 288.08529, found 288.08527.



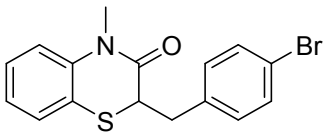
2-(3-fluorobenzyl)-4-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2g**): yellow soild (42 mg, 73%); **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.33 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.29-7.22 (m, 2H), 7.08 (d, *J* = 8.0 Hz, 1H),7.04 (td, *J* = 8.0, 1.0 Hz, 1H), 6.94-6.90 (m, 2H),6.86 (d, *J* = 9.5 Hz, 1H), 3.60 (dd, *J* =10.0, 5.5 Hz, 1H), 3.46 (s, 3H), 3.26 (dd, *J* = 14.0, 5.5 Hz, 1H), 2.74 (dd, *J* = 14.5, 10.0Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.7, 162.6 (d, *J* = 244.5 Hz, 1C), 139.8 (d, *J* = 7.4 Hz, 1C), 139.7, 129.7 (d, *J* = 8.3 Hz, 1C), 128.8, 127.3, 124.8 (d, *J* = 2.6 Hz, 1C), 123.6, 121.3, 117.2, 116.1 (d, *J* = 21.4 Hz, 1C), 113.8 (d, *J* = 20.9 Hz, 1C), 44.6, 34.8, 32.4. HRMS (ESI) m/z calcd for $C_{16}H_{15}FNOS^+$ $(M+H)^+$ 288.08529, found 288.08536.



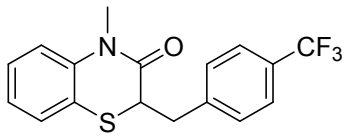
2-(4-fluorobenzyl)-4-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2h**): yellow soild (47 mg, 82%); mp: 127.4-128.8 °C **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.32 (d, *J* = 7.5 Hz, 1H), 7.26 (t, *J* = 8.0 Hz, 1H), 7.11-7.02 (m, 3H), 7.03 (t, *J* = 7.5 Hz, 1H), 6.96 (t, *J* = 8.5 Hz, 2H), 3.57 (dd, *J* =9.5, 5.0 Hz, 1H), 3.45 (s, 3H), 3.21 (dd, *J* = 14.5, 5.5 Hz, 1H), 2.73 (dd, *J* = 14.5, 10.0Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.8, 161.7 (d, *J* = 243.8 Hz, 1C), 139.7, 133.0 (d, *J* = 3.1 Hz, 1C), 130.7 (d, *J* = 8.0 Hz, 1C), 128.8, 127.2, 123.5, 121.3, 117.2, 115.1 (d, *J* = 21.0 Hz, 1C), 45.0, 34.3, 32.2. HRMS (ESI) m/z calcd for $C_{16}H_{15}FNOS^+$ $(M+H)^+$ 288.08529, found 288.08533.



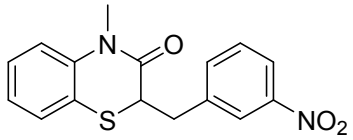
2-(4-chlorobenzyl)-4-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2i**): yellow solid (50 mg, 83%); mp: 101.0-105.4 °C **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.32 (d, *J* = 7.5 Hz, 1H), 7.28-7.23 (m, 3H), 7.08-7.06 (m, 3H), 7.03 (t, *J* = 7.5 Hz, 1H), 3.59 (dd, *J* =9.5, 5.0 Hz, 1H), 3.45 (s, 3H), 3.22 (dd, *J* = 14.0, 5.5 Hz, 1H), 2.74 (dd, *J* = 14.5, 10.0Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.7, 139.6, 135.7, 132.7, 130.5, 128.8, 128.4, 127.2, 123.5, 121.2, 117.2, 44.7, 34.4, 32.4. HRMS (ESI) m/z calcd for $C_{16}H_{15}ClNOS^+$ $(M+H)^+$ 304.05574, found 304.05594.



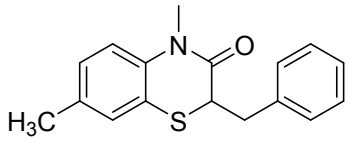
2-(4-bromobenzyl)-4-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2j**): yellow solid (39 mg, 56%); mp: 76.5-78.0 °C **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.40 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 7.5 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.07 (d, *J* = 8.5 Hz, 1H), 7.06-7.01 (m, 3H), 3.58 (dd, *J* =9.5, 5.0 Hz, 1H), 3.45 (s, 3H), 3.20 (dd, *J* = 14.5, 5.5 Hz, 1H), 2.71 (dd, *J* = 14.0, 9.5Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.7, 139.7, 136.3, 131.4, 130.9, 128.8, 127.3, 123.6, 121.2, 120.8, 117.2, 44.7, 34.5, 32.4. HRMS (ESI) m/z calcd for $C_{16}H_{15}BrNOS^+$ $(M+H)^+$ 348.00522, found 348.00534.



4-methyl-2-(4-(trifluoromethyl)benzyl)-2H-benzo[b][1,4]thiazin-3(4H)-one (**2k**): yellow solid (53 mg, 78%); mp: 79.0-82.0 °C **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.54 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 1H), 7.29-7.26 (m, 3H), 7.08 (d, *J* = 8.5 Hz, 1H) 7.05 (t, *J* = 8.0 Hz, 1H), 3.63 (dd, *J* =10.0, 5.5 Hz, 1H), 3.46 (s, 3H), 3.32 (dd, *J* = 14.0, 5.0 Hz, 1H), 2.83 (dd, *J* = 14.5, 10.0 Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.6, 141.4, 139.7, 129.6, 129.1(q, *J* =32.1 Hz), 128.8, 127.4, 125.2(q, *J* =3.6 Hz), 123.6, 123.0, 121.1, 117.3, 44.5, 34.9, 32.4. **^{19}F NMR** (470 MHz, $CDCl_3$) δ: -62.4. HRMS (ESI) m/z calcd for $C_{17}H_{15}F_3NOS^+$ $(M+H)^+$ 338.08210, found 338.08224.

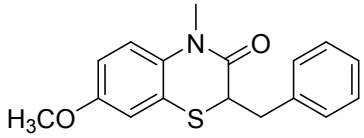


4-methyl-2-(3-nitrobenzyl)-2H-benzo[b][1,4]thiazin-3(4H)-one (**2l**): yellow solid (17 mg, 27%); mp: 89.2-90.1 °C **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.40 (dd, *J* = 8.0, 1.0 Hz, 1H), 8.01 (s, 1H), 7.52 (d, *J* = 7.5 Hz, 1H), 7.47 (t, *J* = 8.0 Hz, 1H), 7.35 (d, *J* = 7.5 Hz, 1H), 7.29 (td, *J* = 8.5, 1.5 Hz, 1H), 7.08 (q, *J* = 8.0 Hz, 2H), 3.65 (dd, *J* =9.5, 5.5 Hz, 1H), 3.48 (s, 3H), 3.36 (dd, *J* = 14.0, 5.5 Hz, 1H), 2.91 (dd, *J* = 14.0, 9.0Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.4, 148.2, 139.7, 139.4, 135.5, 128.3, 128.9, 127.6, 124.3, 123.8, 122.1, 120.9, 117.4, 44.4, 34.9, 32.5. HRMS (ESI) m/z calcd for $C_{16}H_{15}N_2O_3S^+$ $(M+H)^+$ 315.07979, found 315.07977.

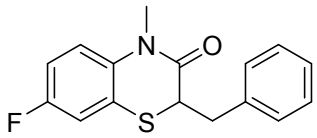


2-benzyl-4,7-dimethyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2n**): yellow oil (48 mg,88%); **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.28 (t, *J* = 7.0 Hz, 2H), 7.24-7.21 (m, 1H), 7.15-7.13 (m, 3H), 7.05 (d, *J* = 8.5 Hz, 1H), 6.96 (d, *J* = 8.5 Hz, 1H), 3.59 (dd, *J* =10.0, 5.0 Hz, 1H), 3.42 (s, 3H), 3.25 (dd, *J* = 14.0, 5.0 Hz, 1H), 2.72 (dd, *J* = 14.0, 10.0Hz, 1H), 2.29 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.8, 137.4, 133.2, 129.2, 129.1, 128.3, 127.8, 126.7, 121.1, 117.0, 45.1, 35.0, 32.3, 20.4. HRMS (ESI) m/z

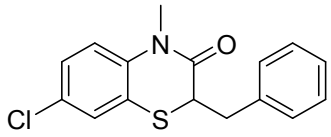
calcd for $C_{17}H_{18}NOS^+$ $(M+H)^+$ 284.11036, found 284.11026.



2-benzyl-7-methoxy-4-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2o**): yellow oil (33 mg,55%); **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.29 (t, *J* = 7.0 Hz, 2H), 7.25 (d, *J* = 8.5 Hz, 1H), 7.16 (d, *J* = 7.0 Hz, 2H), 7.00 (d, *J* = 9.0 Hz, 1H), 6.88 (d, *J* =3.0 Hz, 1H), 6.81 (dd, *J* = 8.5, 2.5 Hz, 1H), 3.79 (s, 3H), 3.62 (dd, *J* = 10.0, 5.5 Hz, 1H), 3.43 (s, 3H), 3.28 (dd, *J* = 14.0, 5.0 Hz, 1H), 2.74 (dd, *J* = 14.5, 10.5Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.6, 155.5, 137.5, 133.6, 129.2, 128.4, 126.9, 122.8, 118.8, 113.7, 113.2, 55.6, 453.4, 35.1, 32.5. HRMS (ESI) m/z calcd for $C_{17}H_{18}NO_2S^+$ $(M+H)^+$ 300.10528, found 300.10492.

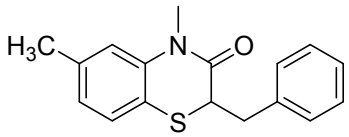


2-benzyl-7-fluoro-4-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2p**): yellow solid (46 mg, 81%); mp: 77.4-78.3 ^{o}C **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.31-7.28 (m, 2H), 7.25-7.22 (m, 1H), 7.14 (d, *J* = 6.5 Hz, 1H), 7.05 (dd, *J* =8.0, 2.5 Hz, 1H), 7.02 (dd, *J* = 9.0, 5.0 Hz, 1H), 6.96 (td, *J* = 7.5, 2.5 Hz, 1H), 3.63 (dd, *J* =10.0, 5.0 Hz, 1H), 3.44 (s, 3H), 3.27 (dd, *J* = 14.0, 5.0 Hz, 1H), 2.73 (dd, *J* = 14.0, 10.0Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.5, 158.1 (d, *J* = 244.5 Hz, 1C), 137.0, 136.2 (d, *J* = 2.6 Hz, 1C), 129.1, 128.4, 126.9, 123.4 (d, *J* = 8.5 Hz, 1C), 118.3 (d, *J* = 8.3 Hz, 1C), 115.5 (d, *J* = 24.4 Hz, 1C), 114.0 (d, *J* = 22.5 Hz, 1C), 45.0, 35.1, 32.6. HRMS (ESI) m/z calcd for $C_{16}H_{15}FNOS^+$ $(M+H)^+$ 288.08529, found 288.08536.

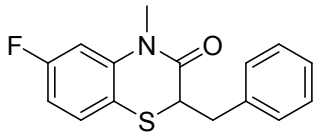


2-benzyl-7-chloro-4-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2q**): yellow solid (49 mg, 81%); mp: 69.3-70.6 ^{o}C **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.31-7.28 (m, 3H), 7.24 (d, *J* = 7.0 Hz, 1H), 7.21 (dd, *J* =8.5, 2.0 Hz, 1H), 7.14 (d, *J* = 7.5 Hz, 2H), 6.98 (d, *J* = 9.0 Hz, 1H), 3.61 (dd, *J* =10.0, 5.5 Hz, 1H), 3.43 (s, 3H), 3.26 (dd, *J* = 14.5,

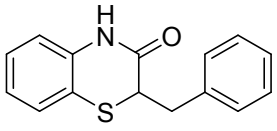
5.5 Hz, 1H), 2.73 (dd, *J* = 14.5, 10.0Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.5, 138.3, 136.9, 129.1, 128.5, 128.4, 128.3, 127.1, 127.0, 123.2, 118.1, 44.8, 35.1, 32.4. HRMS (ESI) m/z calcd for $C_{16}H_{15}ClNOS^+$ $(M+H)^+$ 304.05574, found 304.05582.



2-benzyl-4,6-dimethyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2r**): yellow oil (42 mg,74%); **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.28 (t, *J* = 7.0 Hz, 2H), 7.23 (d, *J* = 7.0 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 1H), 7.14 (d, *J* = 7.5 Hz, 2H), 6.90 (s, 1H), 6.86 (d, *J* =7.5 Hz, 1H), 3.59 (dd, *J* = 10.0, 5.0 Hz, 1H), 3.45 (s, 3H), 3.23 (dd, *J* = 14.0, 5.0 Hz, 1H), 2.71 (dd, *J* = 14.0, 10.0Hz, 1H), 2.37 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 167.1, 139.6, 137.4, 137.2, 129.2, 128.7, 128.3, 126.8, 124.3, 117.9, 117.8, 45.2, 34.9, 32.3, 21.4. HRMS (ESI) m/z calcd for $C_{17}H_{18}NOS^+$ $(M+H)^+$ 284.11036, found 284.11026.

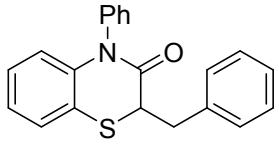


2-benzyl-6-fluoro-4-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2s**): yellow solid (46 mg, 80%); mp: 62.0-62.8 ^{o}C **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.28 (q, *J* = 8.0 Hz, 3H), 7.23 (t, *J* = 7.0 Hz, 1H), 7.13 (d, *J* =7.0 Hz, 2H), 6.81 (dd, *J* = 10.5, 2.5 Hz, 1H), 6.76 (td, *J* = 8.5, 2.5 Hz, 1H), 3.61 (dd, *J* =10.0, 5.5 Hz, 1H), 3.43 (s, 3H), 3.25 (dd, *J* = 14.5, 5.5 Hz, 1H), 2.73 (dd, *J* = 14.0, 10.0Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.8, 161.9 (d, *J* = 243.6 Hz, 1C), 140.9 (d, *J* = 9.9 Hz, 1C), 137.1, 129.8 (d, *J* = 9.1 Hz, 1C), 129.1, 128.3, 126.9, 116.3 (d, *J* = 2.9 Hz, 1C), 110.3 (d, *J* = 22.0 Hz, 1C), 105.0 (d, *J* = 26.5 Hz, 1C), 44.8, 35.0, 32.3. HRMS (ESI) m/z calcd for $C_{16}H_{15}FNOS^+$ $(M+H)^+$ 288.08529, found 288.08530.

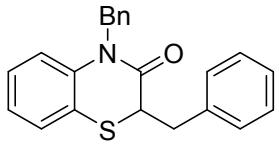


2-benzyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2t**)[3]: yellow soild (43 mg, 85%); **^{1}H NMR** (500 MHz, DMSO-d_6) δ: 10.65 (s, 1H),7.28 (t, *J* = 6.5 Hz, 3H), 7.23-7.18 (m, 4H), 7.02 (d, *J* = 8.0 Hz, 1H), 6.98 (d, *J* = 7.5 Hz, 1H), 3.80 (dd, *J* =10.0, 6.0Hz, 1H),

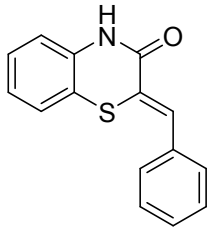
3.19 (dd, *J* = 14.5, 6.0 Hz, 1H), 2.70 (dd, *J* = 14.0, 9.5 Hz, 1H). **^{13}C NMR** (125 MHz, DMSO-d_6) δ: 166.7, 137.9, 137.4, 129.6, 128.7, 128.3, 127.5, 127.1, 123.6, 118.2, 117.3, 43.2, 35.3.



2-benzyl-4-phenyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2u**): yellow solid (21 mg, 32%); mp: 141.9-143.2 °C **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.48 (t, *J* = 7.5 Hz, 2H), 7.41 (t, *J* = 7.0 Hz, 1H), 7.37 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.23 (t, *J* = 7.0 Hz, 2H), 7.26 (t, *J* = 7.5 Hz, 1H), 7.21 (dd, *J* =11.5, 7.0 Hz, 1H), 7.05 (td, *J* = 7.5, 1.5 Hz, 1H), 7.01 (td, *J* = 7.5, 1.5 Hz, 1H), 6.50 (dd, *J* = 8.0, 1.0 Hz, 1H), 3.80 (dd, *J* =10.0, 5.5 Hz, 1H), 3.37 (dd, *J* = 14.5, 5.5 Hz, 1H), 2.89 (dd, *J* = 14.0, 9.5Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.5, 140.7, 139.7, 137.2, 129.8, 129.4, 128.8, 128.7, 128.4, 128.2, 127.0, 126.8, 123.7, 121.2, 119.8, 45.3, 35.0. HRMS (ESI) m/z calcd for $C_{21}H_{18}NOS^+$ $(M+H)^+$ 332.11036, found 332.11041.

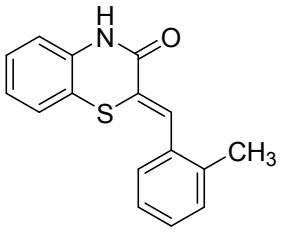


2,4-dibenzyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**2v**)[4]: yellow solid (58 mg, 82%); **^{1}H NMR** (500 MHz, $CDCl_3$) δ: 7.32-7.27 (m, 5H), 7.23 (q, *J* = 7.0 Hz, 2H), 7.18 (t, *J* = 8.0 Hz, 4H), 7.10 (td, *J* = 8.0, 1.5 Hz, 1H), 7.01 (d, *J* = 8.5 Hz, 1H), 6.97 (td, *J* = 7.5, 1.0 Hz, 1H), 5.33 (d, *J* =16.0 Hz, 1H), 5.12 (d, *J* = 16.5 Hz, 1H), 3.73 (dd, *J* =10.0, 5.0 Hz, 1H), 3.36 (dd, *J* = 14.5, 5.5 Hz, 1H), 2.82 (dd, *J* = 14.0, 10.0 Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ: 166.8, 139.2, 137.2, 136.8, 129.3, 128.9, 128.7, 128.3, 127.2, 127.1, 126.9, 126.2, 123.7, 121.6, 117.7, 48.7, 45.1, 34.8.

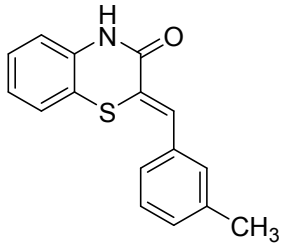


(Z)-2-benzylidene-2H-benzo[b][1,4]thiazin-3(4H)-one (**3t**)[5]: yellow soild (46 mg,

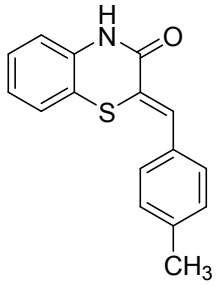
91%); **^{1}H NMR** (500 MHz, DMSO-d_6) δ: 11.04 (s, 1H), 7.80 (s, 1H), 7.66 (d, *J* = 7.5 Hz, 2H), 7.50 (t, *J* = 7.5 Hz, 2H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.18 (td, *J* = 8.0, 1.0 Hz, 1H), 7.06 (d, *J* = 8.0 Hz, 1H), 7.00 (td, *J* = 8.5, 1.0 Hz, 1H). **^{13}C NMR** (125 MHz, DMSO-d_6) δ: 159.0, 134.8, 134.7, 131.1, 130.4, 129.3, 127.5, 125.7, 123.7, 120.9, 117.2, 115.5.



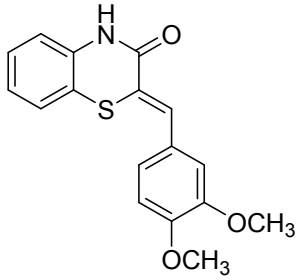
(Z)-2-(2-methylbenzylidene)-2H-benzo[b][1,4]thiazin-3(4H)-one (**3ta**): yellow soild (34 mg, 63%); mp: 171.8-173.1 °C **^{1}H NMR** (500 MHz, DMSO-d_6) δ: 11.02 (s, 1H), 7.85 (s, 1H), 7.44 (d, *J* = 5.5 Hz, 1H), 7.29 (s, 3H), 7.22 (d, *J* = 8.0 Hz, 1H), 7.16 (t, *J* = 7.5 Hz, 1H), 7.06 (d, *J* = 8.0 Hz, 1H), 6.97 (t, *J* = 7.5 Hz, 1H), 2.27 (s, 3H). **^{13}C NMR** (125 MHz, DMSO-d_6) δ: 159.2, 137.4, 134.8, 133.9, 130.8, 130.4, 129.3, 128.7, 127.3, 126.2, 125.7, 123.6, 122.4, 117.2, 115.8, 20.0. HRMS (ESI) m/z calcd for $C_{16}H_{14}NOS^+$ $(M+H)^+$ 268.07906, found 268.07907.



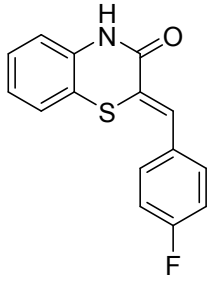
(Z)-2-(3-methylbenzylidene)-2H-benzo[b][1,4]thiazin-3(4H)-one (**3tb**): yellow soild (36 mg, 68%); mp: 138.7-139.8 °C **^{1}H NMR** (500 MHz, DMSO-d_6) δ: 11.00 (s, 1H), 7.76 (s, 1H), 7.48-7.45 (m, 2H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.31 (d, *J* = 7.5 Hz, 1H), 7.22 (d, *J* = 7.5 Hz, 1H), 7.18 (td, *J* = 8.5, 1.0 Hz, 1H), 7.06 (d, *J* = 8.0 Hz, 1H), 7.01 (td, *J* = 7.5, 1.0 Hz, 1H), 2.37 (s, 3H). **^{13}C NMR** (125 MHz, DMSO-d_6) δ: 159.1, 138.4, 134.8, 134.7, 131.3, 131.0, 130.0, 129.0, 127.4, 127.4, 125.7, 123.7, 120.7, 117.2, 115.6, 21.5. HRMS (ESI) m/z calcd for $C_{16}H_{14}NOS^+$ $(M+H)^+$ 268.07906, found 268.07898.



(Z)-2-(4-methylbenzylidene)-2H-benzo[b][1,4]thiazin-3(4H)-one (**3tc**)[6]: yellow soild (36 mg, 67%); **^{1}H NMR** (500 MHz, DMSO-d_6) δ: 10.97 (s, 1H), 7.76 (s, 1H), 7.57 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 3H), 7.18 (td, *J* = 8.5, 1.5 Hz, 1H), 7.05 (d, *J* = 8.0 Hz, 1H), 7.00 (td, *J* = 8.5, 1.0 Hz, 1H), 2.35 (s, 3H). **^{13}C NMR** (125 MHz, DMSO-d_6) δ: 159.2, 139.2, 134.7, 132.0, 131.2, 130.4, 129.7, 127.4, 125.7, 123.6, 119.7, 117.1, 115.5, 21.5.

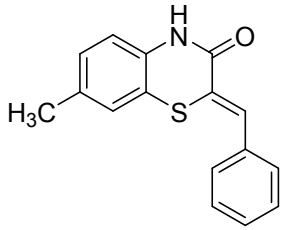


(Z)-2-(3,4-dimethoxybenzylidene)-2H-benzo[b][1,4]thiazin-3(4H)-one (**3td**)[6]: yellow soild (44 mg, 71%); **^{1}H NMR** (500 MHz, DMSO-d_6) δ: 10.91 (s, 1H), 7.75 (s, 1H), 7.31-7.29 (m, 3H), 7.17 (t, *J* = 7.5 Hz, 1H), 7.08 (d, *J* = 9.0 Hz, 1H), 7.05 (d, *J* = 8.0 Hz, 1H), 7.00 (t, *J* = 7.5 Hz, 1H), 3.82 (s, 3H) ,3.81(s, 3H). **^{13}C NMR** (125 MHz, DMSO-d_6) δ: 159.5, 149.9, 148.9, 134.8, 131.4, 127.6, 127.4, 125.7, 123.7, 123.6, 118.0, 117.1, 115.6, 114.2, 112.1, 56.1.

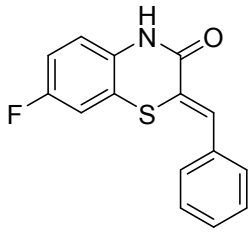


(Z)-2-(4-fluorobenzylidene)-2H-benzo[b][1,4]thiazin-3(4H)-one (**3te**): yellow soild (31 mg, 57%); mp: 135.2-137.6 ^{o}C **^{1}H NMR** (500 MHz, DMSO-d_6) δ: 11.03 (s, 1H), 7.79 (s, 1H), 7.74 (dd, *J* = 8.5, 6.0 Hz, 2H), 7.34 (t, *J* = 8.5 Hz, 2H), 7.31 (d, *J* = 7.5 Hz, 1H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.06 (d, *J* = 7.5 Hz, 1H), 7.01 (t, *J* = 7.5 Hz, 1H).

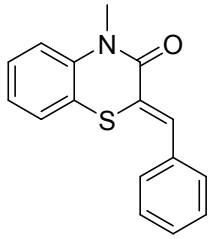
^{13}C NMR (125 MHz, DMSO-d6) δ: 162.3 (d, *J* = 246.4 Hz, 1C), 159.0, 134.7, 132.6 (d, *J* = 8.4 Hz, 1C), 131.4 (d, *J* = 3.1 Hz, 1C), 130.1, 127.5, 125.7, 123.7, 120.6, 117.3, 116.2 (d, *J* = 21.5 Hz, 1C), 115.2. HRMS (ESI) m/z calcd for $C_{15}H_{11}FNOS^+$ $(M+H)^+$ 272.05399, found 272.05402.



(Z)-2-benzylidene-7-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**3tf**): yellow soild (44 mg, 82%); mp: 178.6-180.0 °C **^{1}H NMR** (500 MHz, DMSO-d_6) δ: 10.94 (s, 1H), 7.78 (s, 1H), 7.66 (d, *J* = 7.5 Hz, 2H), 7.49 (t, *J* = 7.5 Hz, 2H), 7.40 (t, *J* = 7.0 Hz, 1H), 7.10 (s, 1H), 6.99-6.94 (m, 2H), 2.20 (s, 3H). **^{13}C NMR** (125 MHz, DMSO-d_6) δ: 158.9, 134.9, 133.0, 132.3, 130.9, 130.3, 129.2, 129.1, 128.1, 125.7, 121.1, 117.2, 115.2, 20.0. HRMS (ESI) m/z calcd for $C_{16}H_{14}NOS^+$ $(M+H)^+$ 268.07906, found 268.07901.



(Z)-2-benzylidene-7-fluoro-2H-benzo[b][1,4]thiazin-3(4H)-one (**3tg**): yellow soild (27 mg, 50%); mp: 174.7-177.1 °C **^{1}H NMR** (500 MHz, DMSO-d_6) δ: 11.07 (s, 1H), 7.81 (s, 1H), 7.65 (d, *J* = 7.5 Hz, 2H), 7.50 (t, *J* = 7.5 Hz, 2H), 7.41 (t, *J* = 7.5 Hz, 1H), 7.29 (d, *J* = 9.0 Hz, 1H), 7.09-7.03 (m, 2H). **^{13}C NMR** (125 MHz, DMSO-d6) δ: 158.7, 158.0 (d, *J* = 239.6 Hz, 1C), 134.6, 131.7, 131.4, 130.3, 129.4, 129.1, 120.1, 118.6 (d, *J* = 8.6 Hz, 1C), 117.1 (d, *J* = 8.9 Hz, 1C), 114.5 (d, *J* = 22.9 Hz, 1C), 112.4 (d, *J* = 26.1 Hz, 1C). HRMS (ESI) m/z calcd for $C_{15}H_{11}FNOS^+$ $(M+H)^+$ 272.05399, found 272.05389.

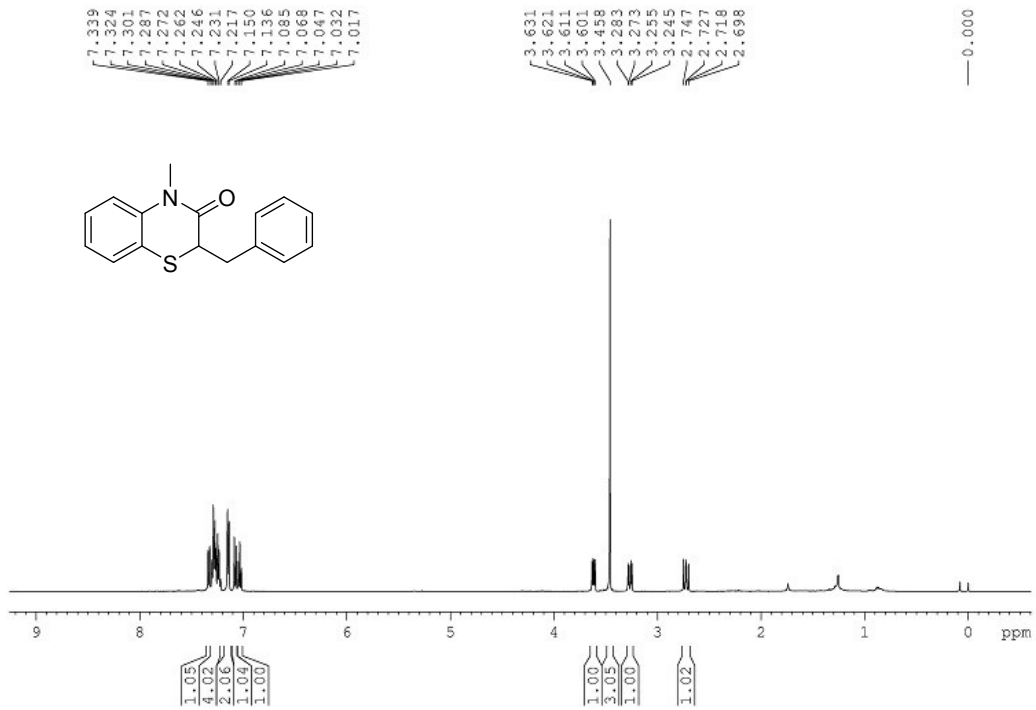


(Z)-2-benzylidene-4-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (**3a**)[5]: yellow soild (15 mg, 28%); **^{1}H NMR** (500 MHz, DMSO-d_6) δ: 7.90 (s, 1H), 7.62 (d, *J* = 8.0 Hz, 2H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.35 (t, *J* = 7.5 Hz, 1H), 7.25-7.21 (m, 2H), 7.05 (d, *J* = 8.0 Hz, 1H), 7.01 (td, *J* = 7.5, 1.0 Hz, 1H), 3.55 (s, 3H). **^{13}C NMR** (125 MHz, DMSO-d_6) δ: 162.5, 137.5, 134.9, 134.8, 130.3, 128.7, 128.4, 127.1, 126.3, 123.4, 119.7, 116.6, 32.8.

6) References

1. Vachhani, D. D.; Butani, H. H.; Sharma, N.; Bhoya, U. C.; Shah, A. K.; Van der Eycken, E. V. *Chem. Commun.* **2015**, *51*, 14862.
2. Babudri, F.; Nunno, Di; Floroi, S. *Synthesis.* **1982**, *1982*, 488.
3. Trapani, G.; Latrofa, A.; Reho, A.; Franco, M.; Liso, G. *J. Heterocyclic Chem.* **1992**, *29*,1155.
4. Takamizawa, A.; Sato, H.; Sato, Y. *Chem. Pharm. Bull.* **1972**, *20*, 892.
5. Sharifi, A.; Ansari, M.; Darabi, H. R.; Abaee, M. S. *J. Sulfur Chem.* **2016**, *37*,593.
6. Baliah, V.; Rangarajan, T. *J. Chem. Soc.* **1960** , *0*, 4703.

7) Scanned ^{1}H NMR and ^{13}C NMR Spectra of Al Compounds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7.017
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3.621
3.611
3.601
3.458
3.283
3.273
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3.245
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O
S
9
8
7
6
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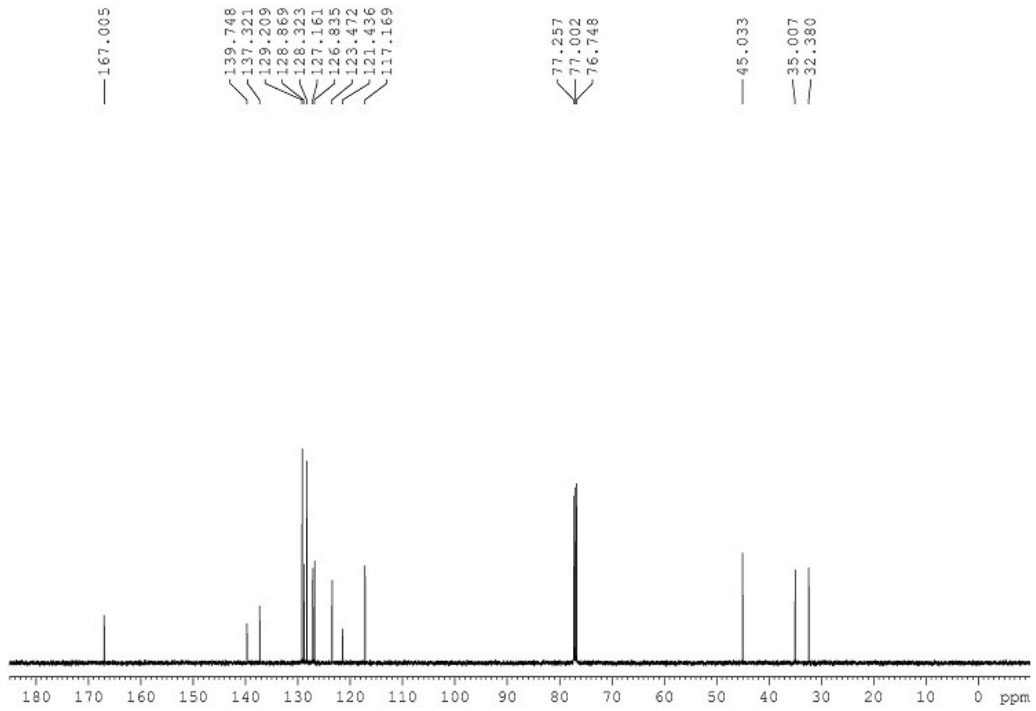
1.02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76.748
45.033
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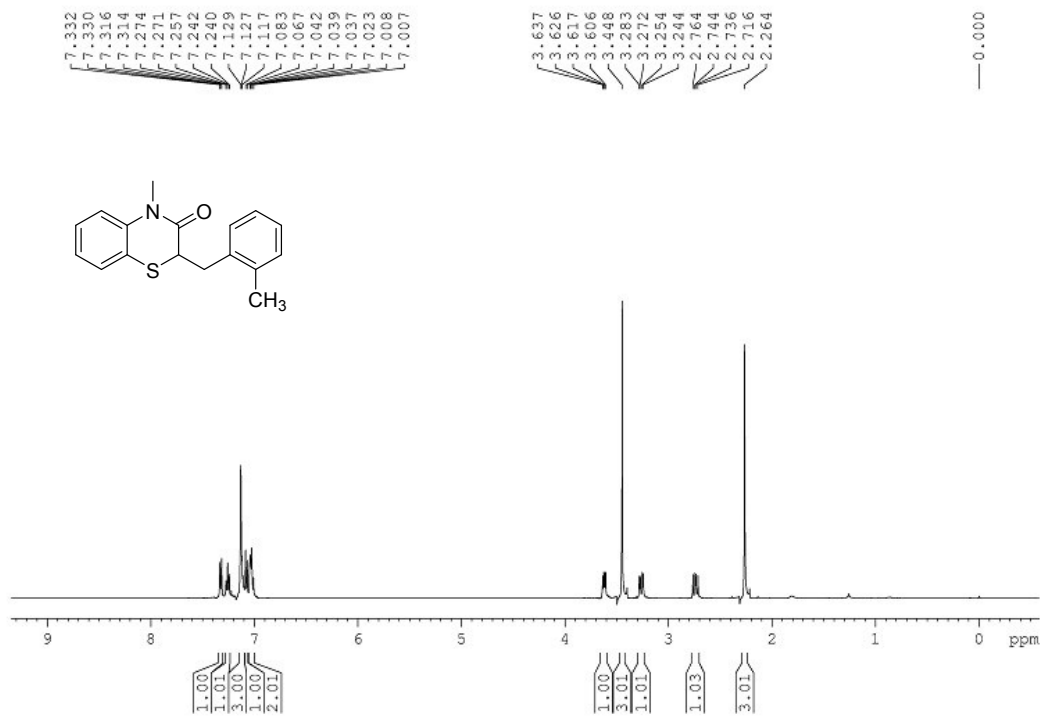
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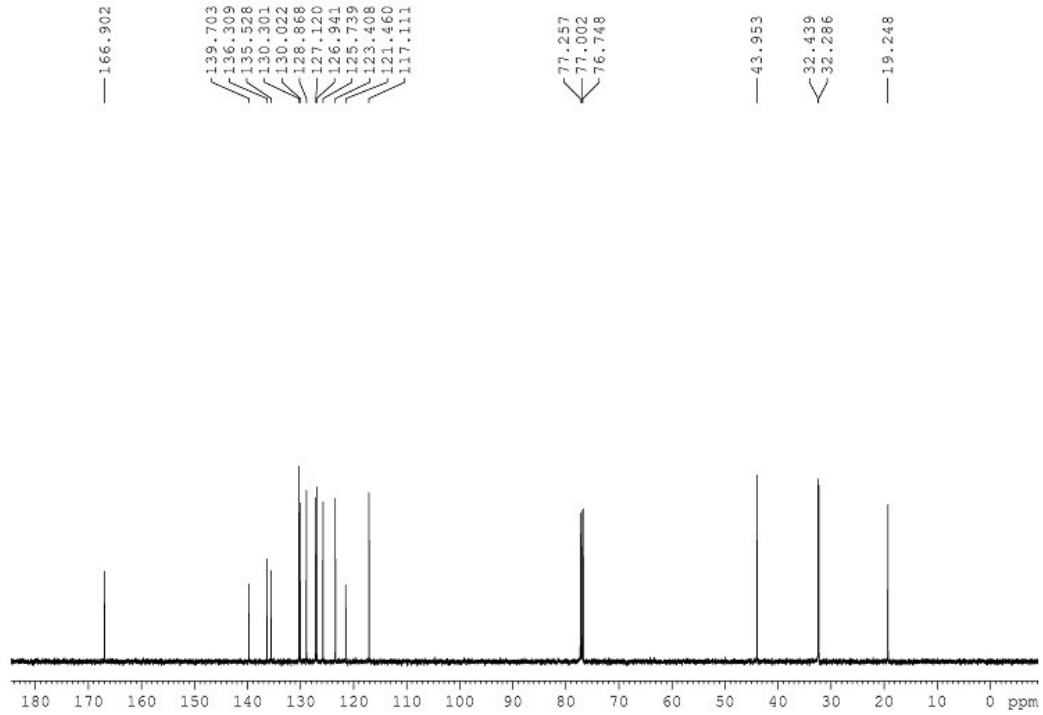
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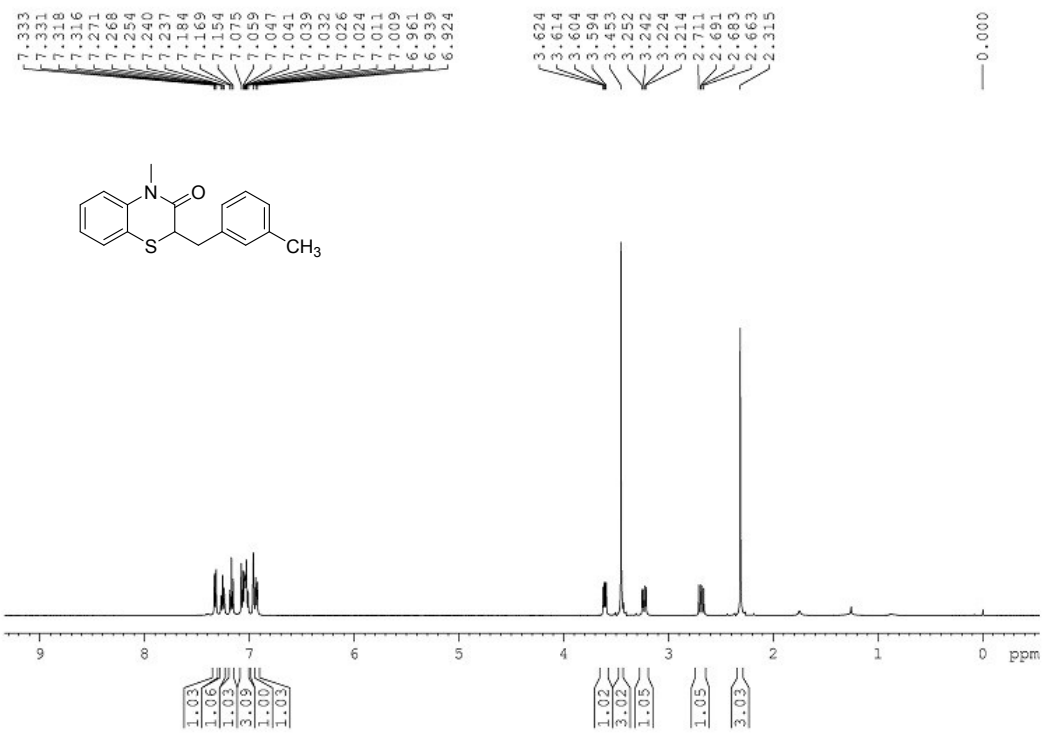
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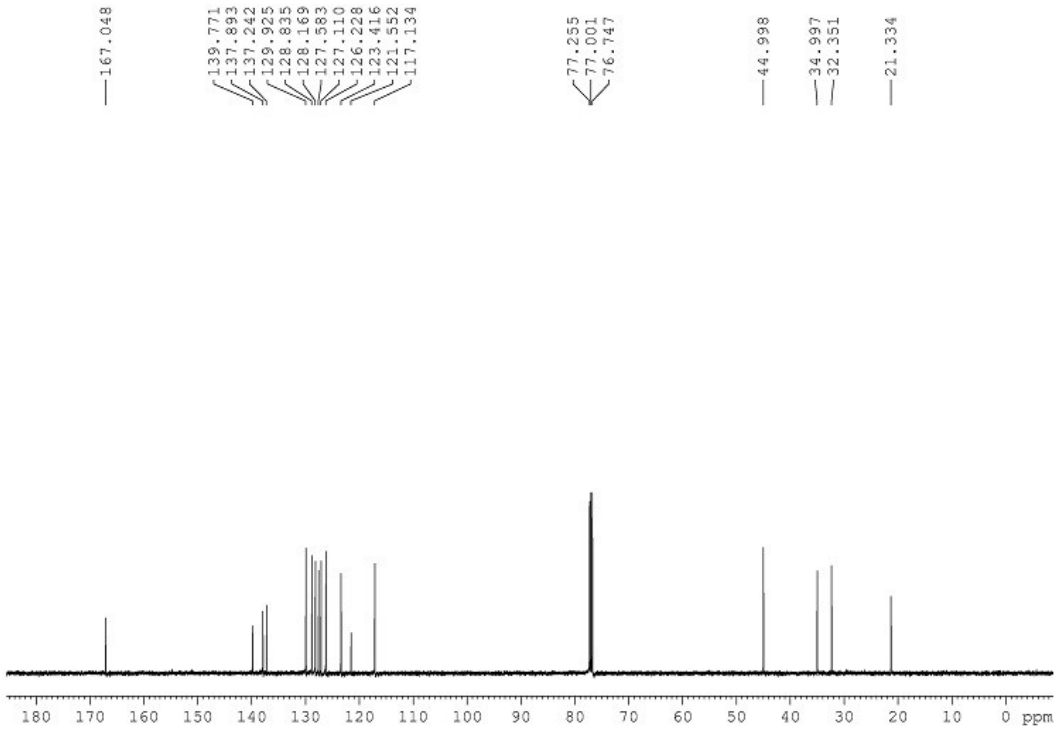
3.03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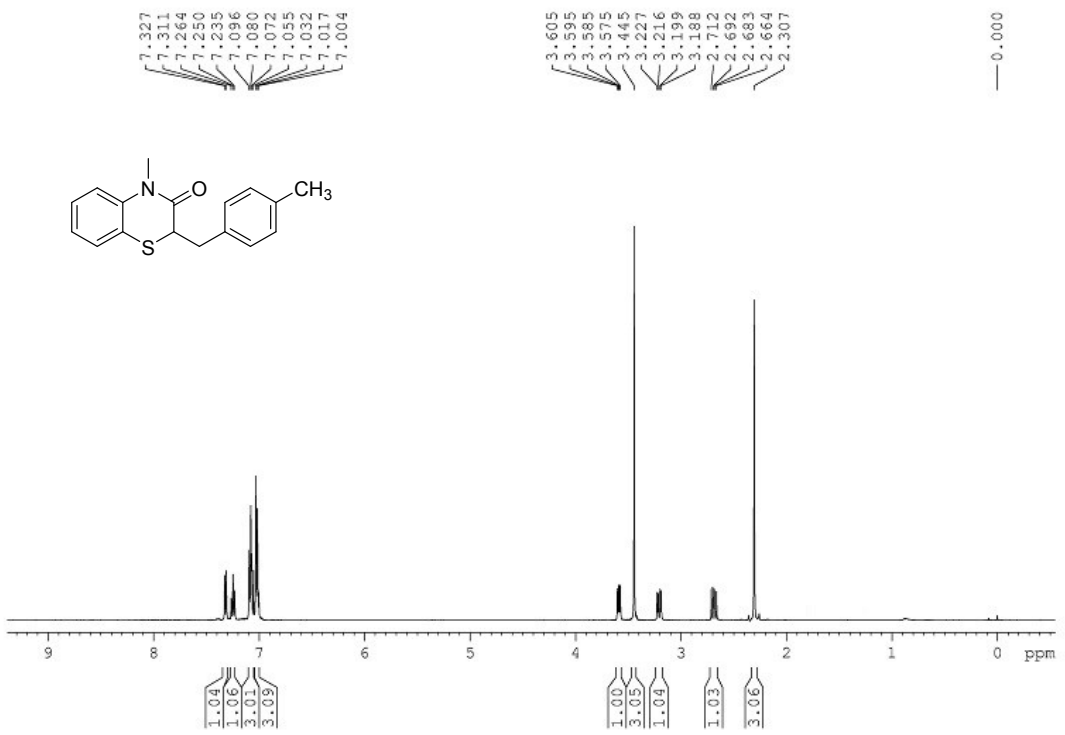
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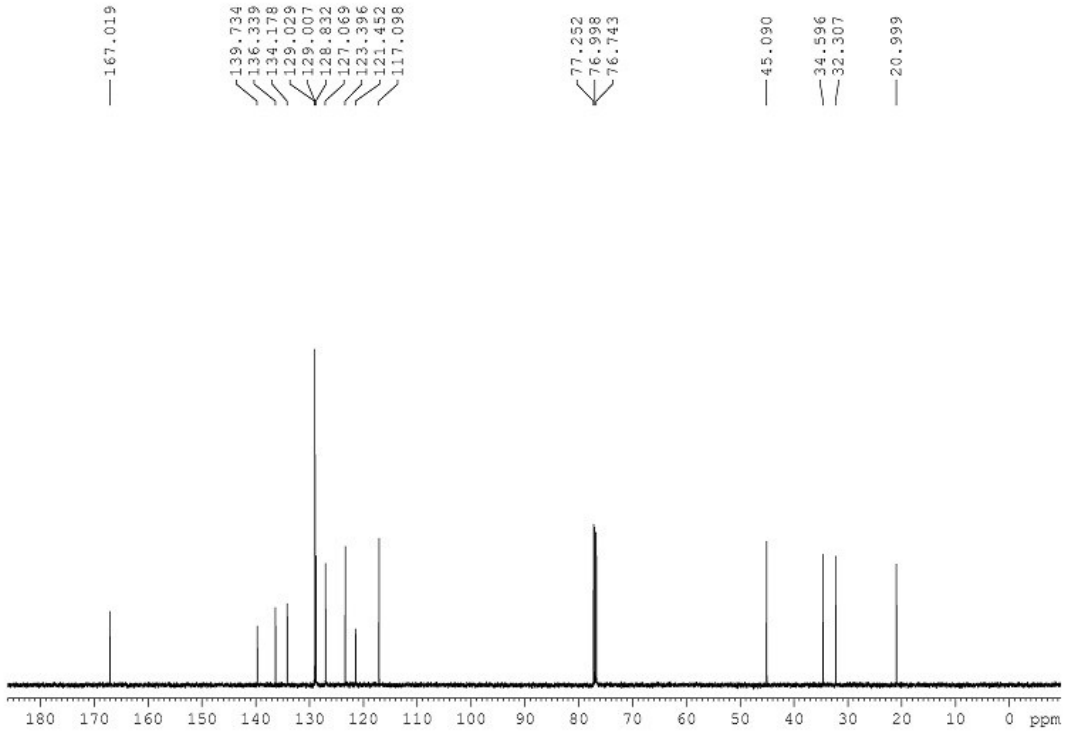
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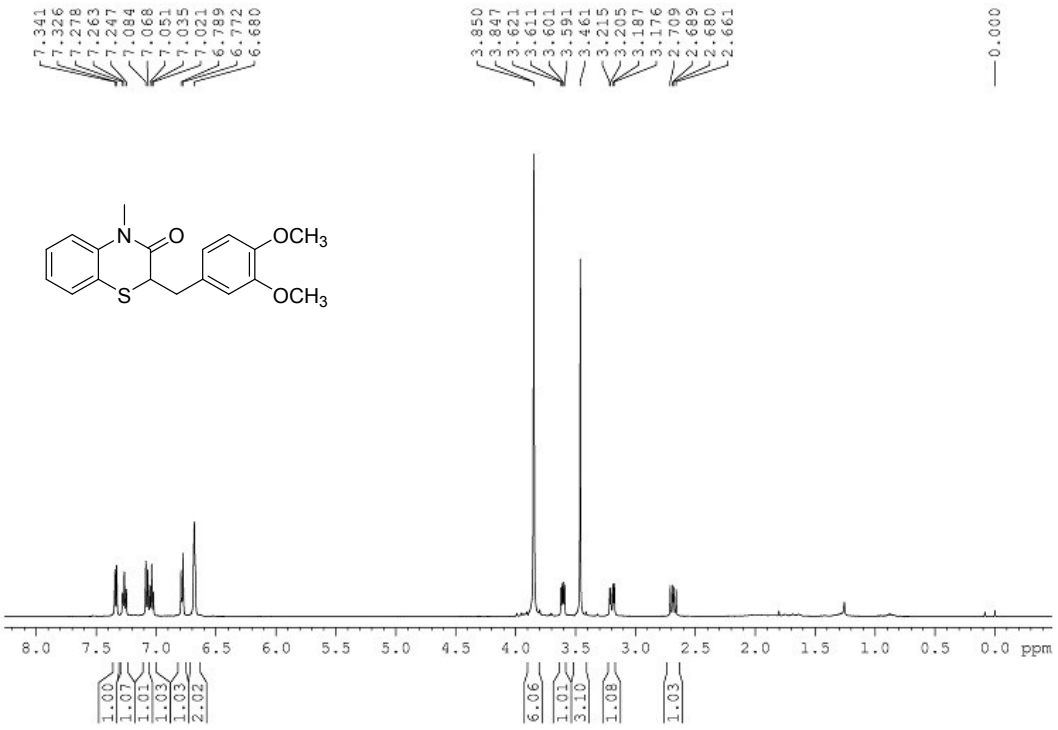
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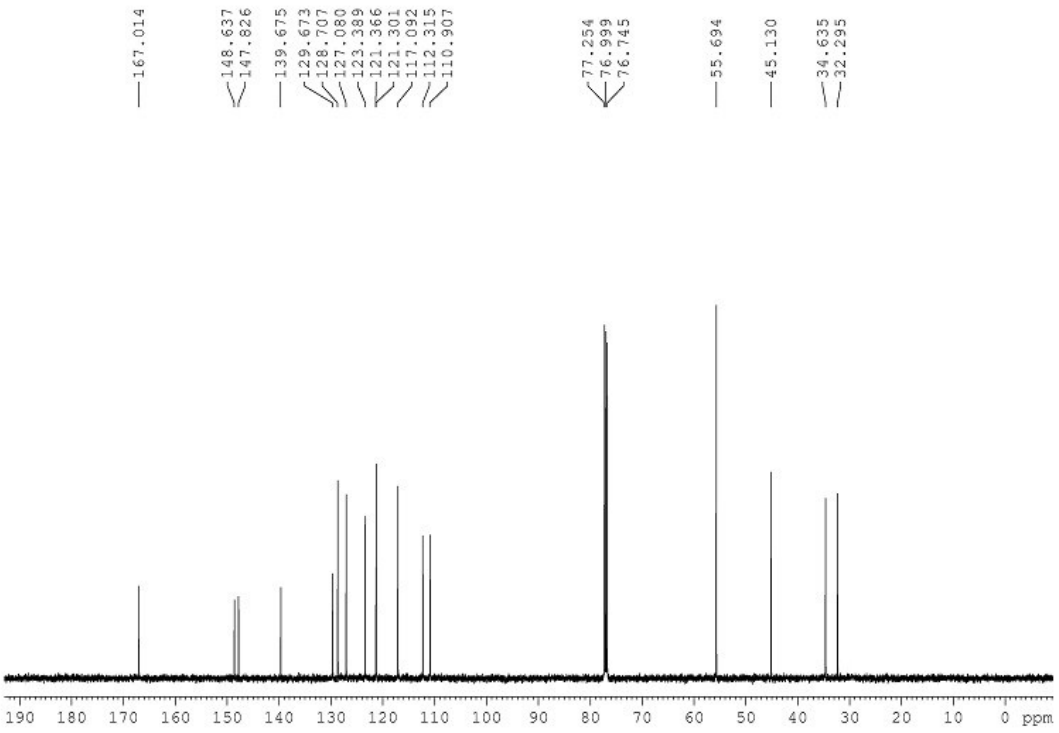
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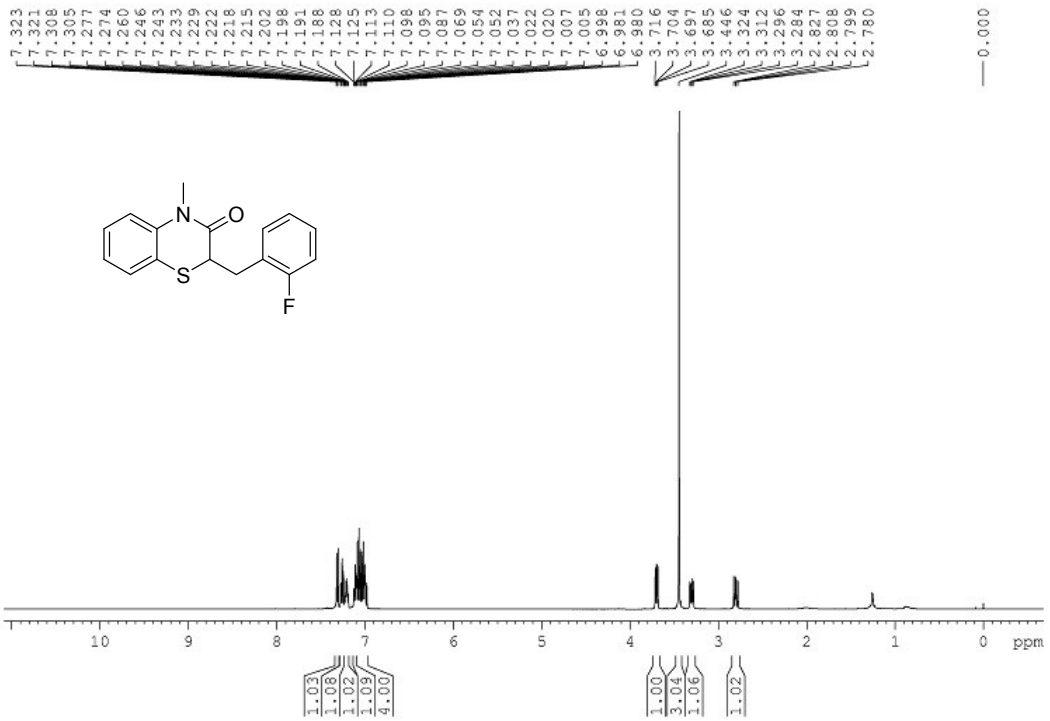
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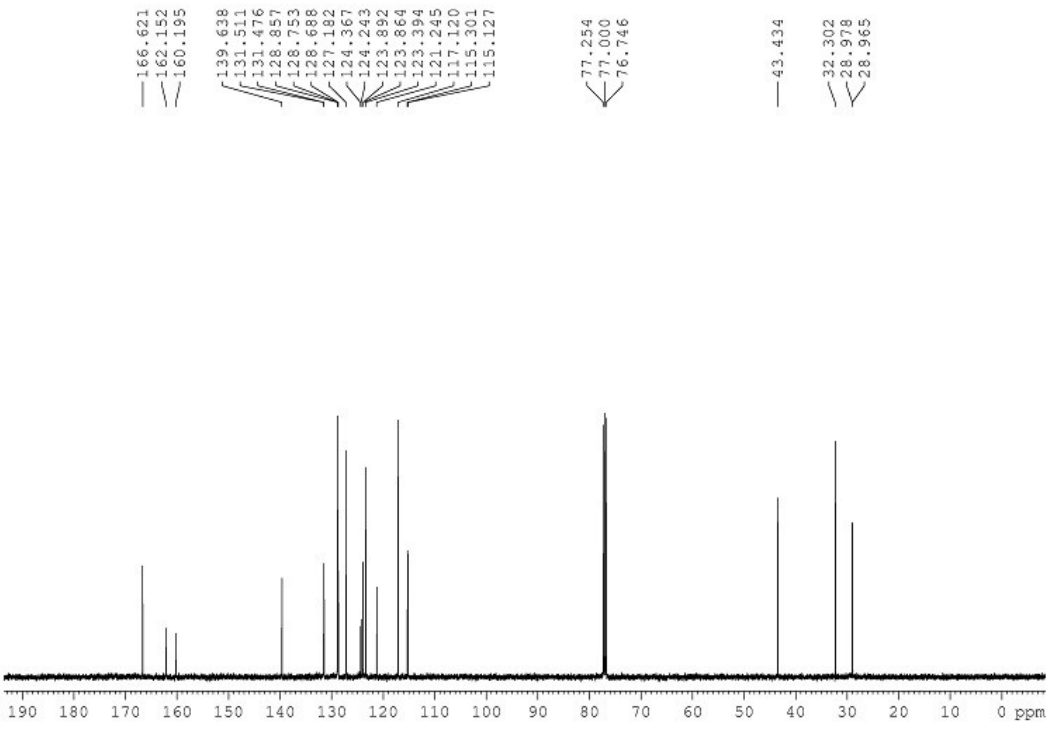
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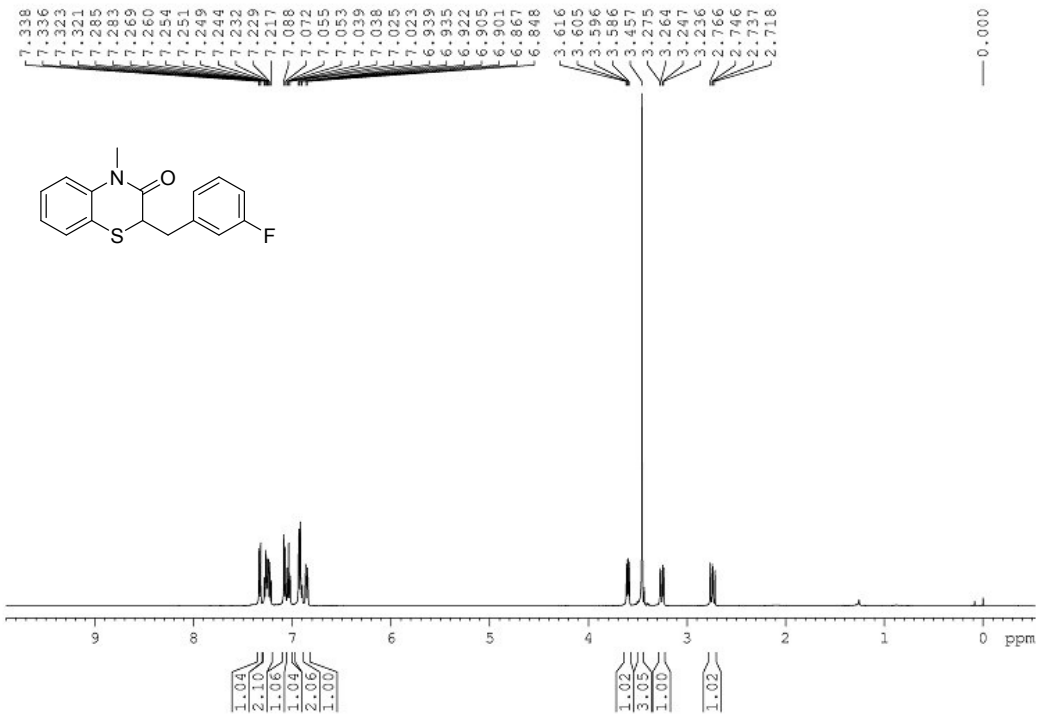
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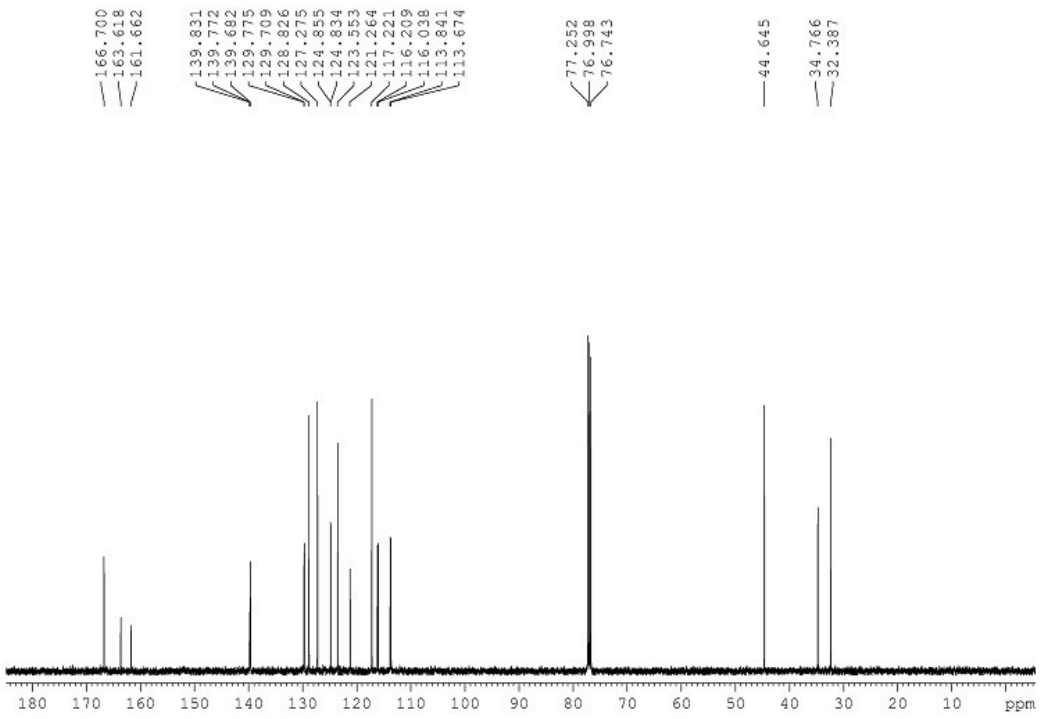
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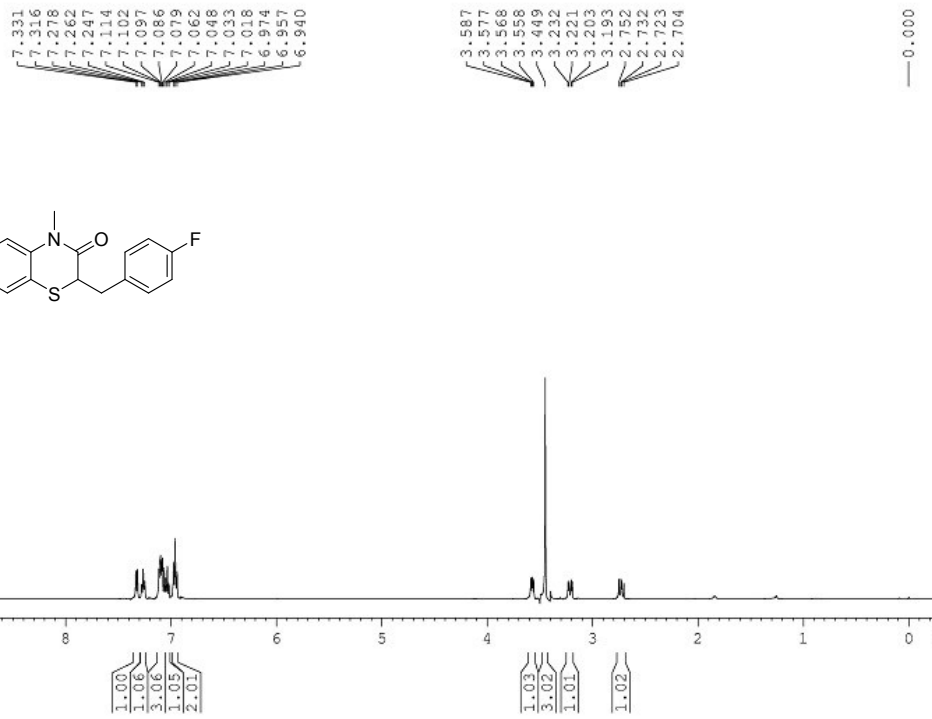
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7.018
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6.957
6.940
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3.577
3.568
3.558
3.449
3.232
3.221
3.203
3.193
2.752
2.732
2.723
2.704
0.000
N
O
F
S
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8
7
6
5
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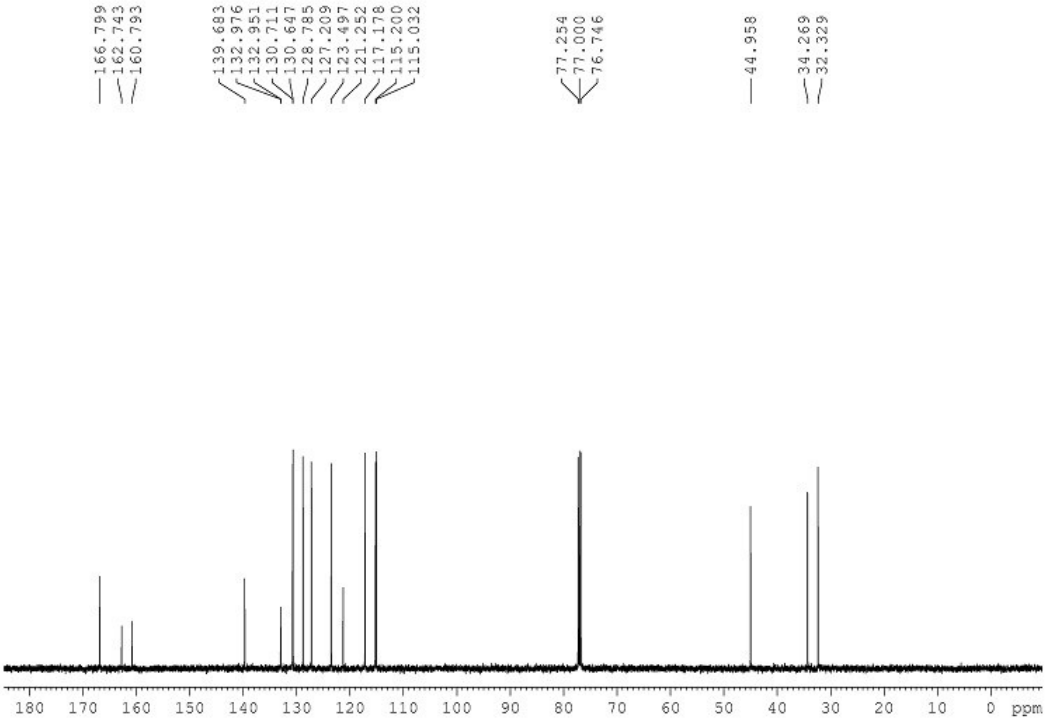
1.02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121.252
117.178
115.200
115.032
77.254
77.000
76.746
44.958
34.269
32.329
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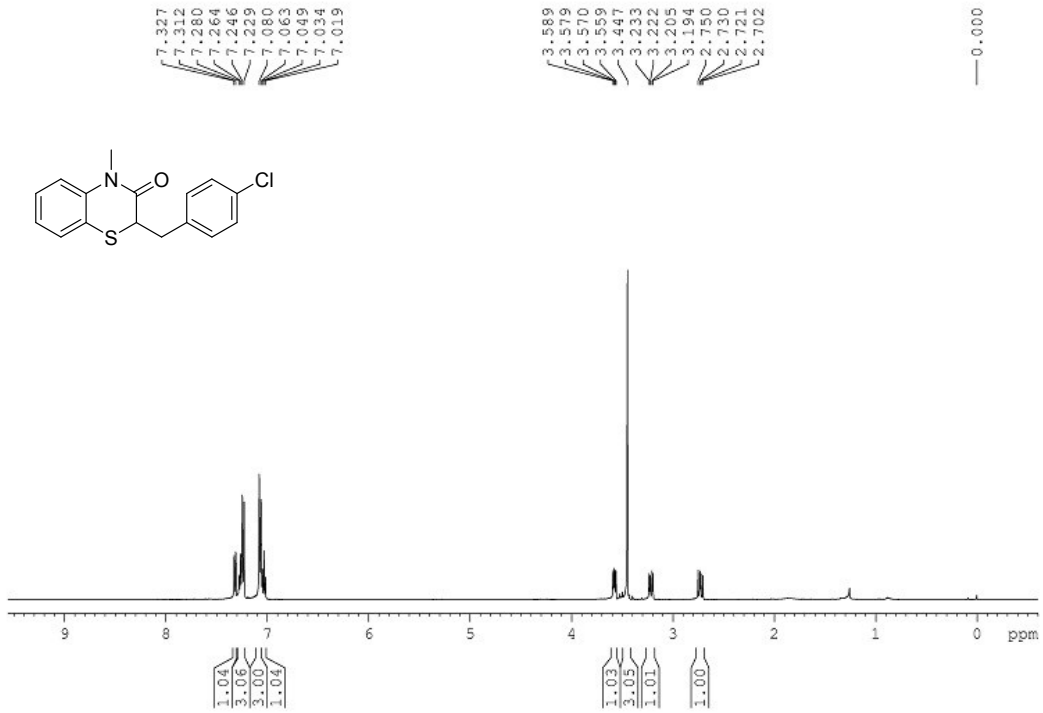
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3.570
3.559
3.447
3.233
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3.205
3.194
2.750
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2.721
2.702
0.000
N
O
Cl
S
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8
7
6
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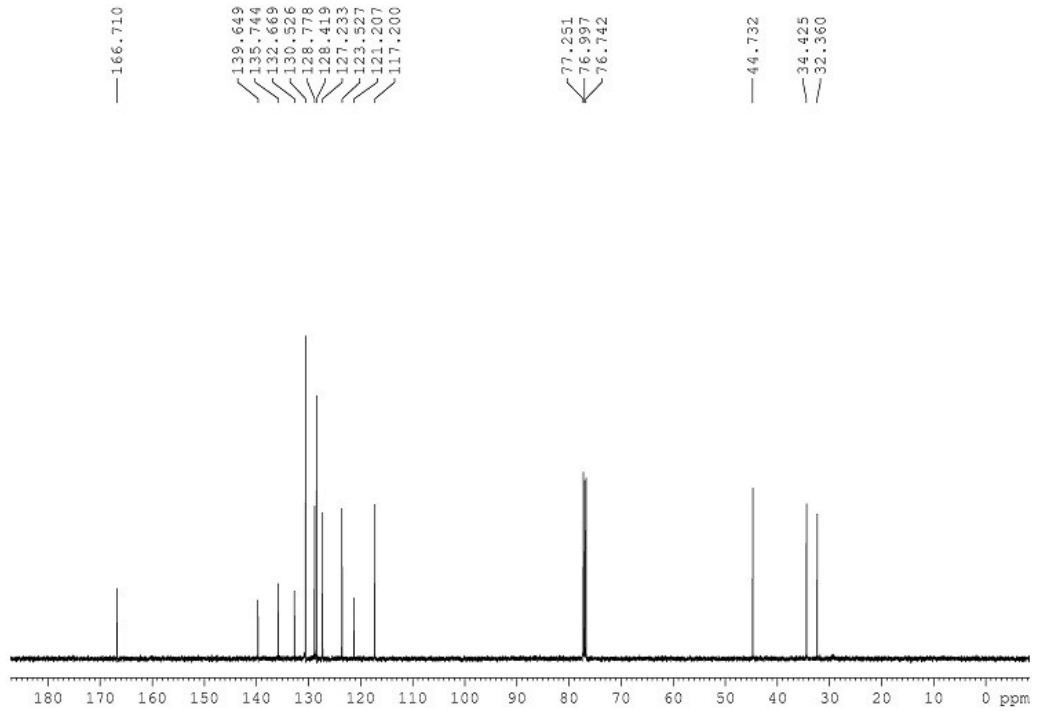
1.00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44.732
34.425
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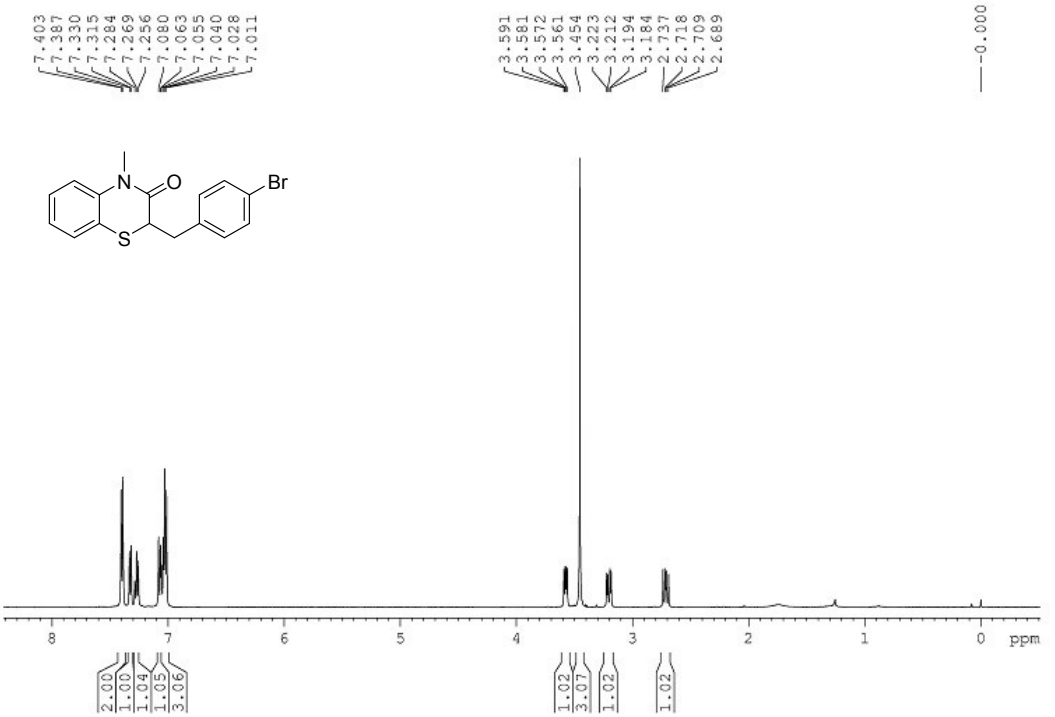
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3.581
3.572
3.561
3.454
3.223
3.212
3.194
3.184
2.737
2.718
2.709
2.689
0.000
N
O
Br
S
8
7
6
5
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3.06
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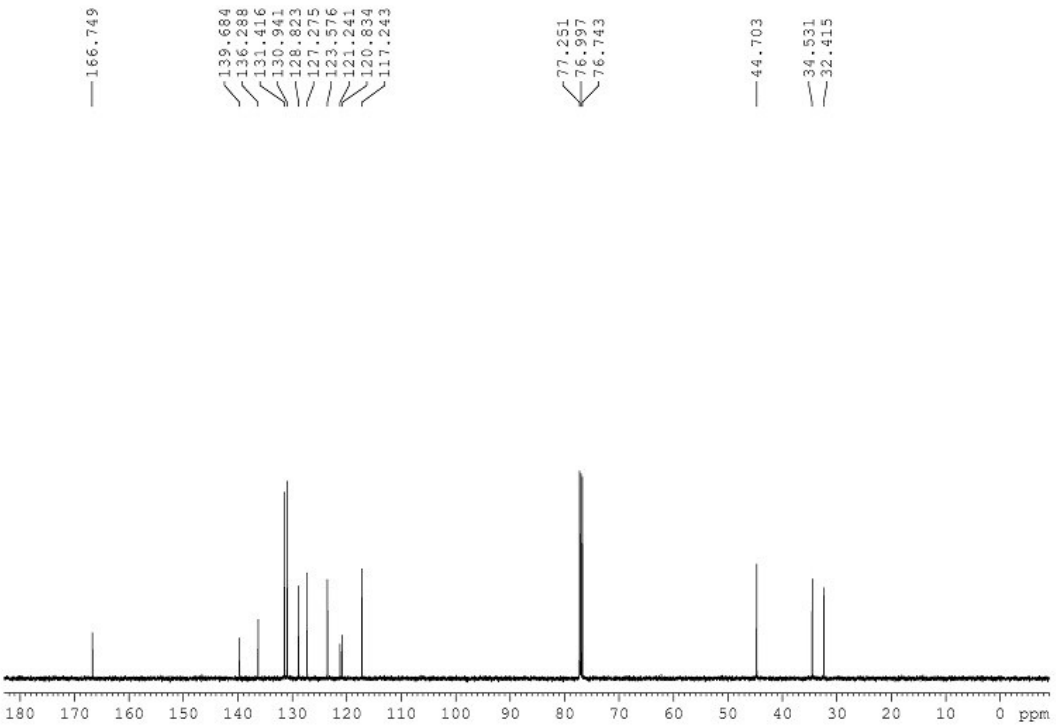
1.02
1.02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140
130
120
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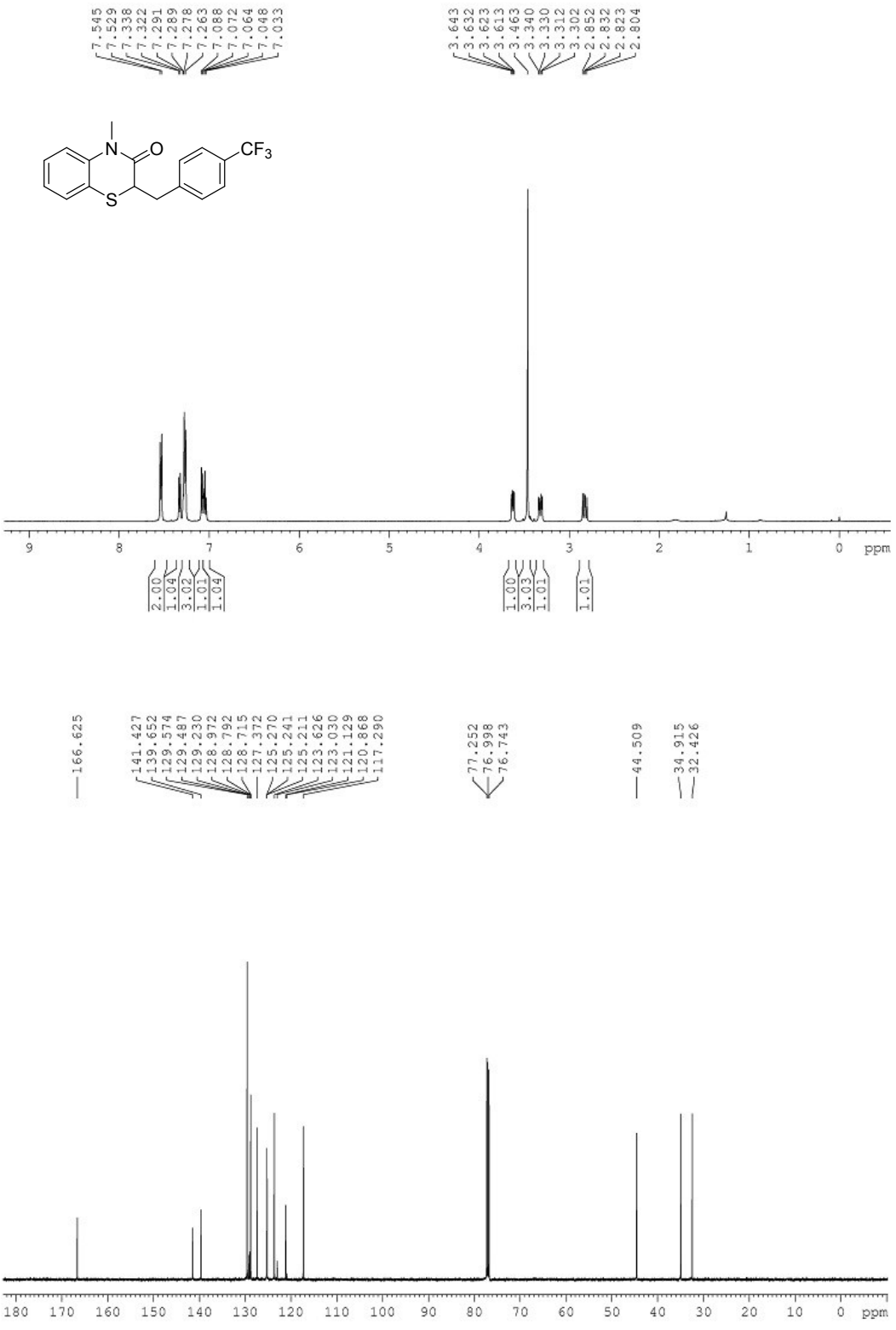
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7.048
7.033
3.643
3.632
3.623
3.613
3.463
3.340
3.330
3.312
3.302
2.852
2.832
2.823
2.804
N
O
S
CF3
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8
7
6
5
4
3
2
1
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ppm
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1.04
3.02
1.01
1.04
1.00
3.03
1.01
1.01
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141.427
139.652
129.574
129.487
129.230
128.972
128.792
128.715
127.372
125.270
125.241
125.211
123.626
123.030
121.129
120.868
117.290
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44.509
34.915
32.426
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130
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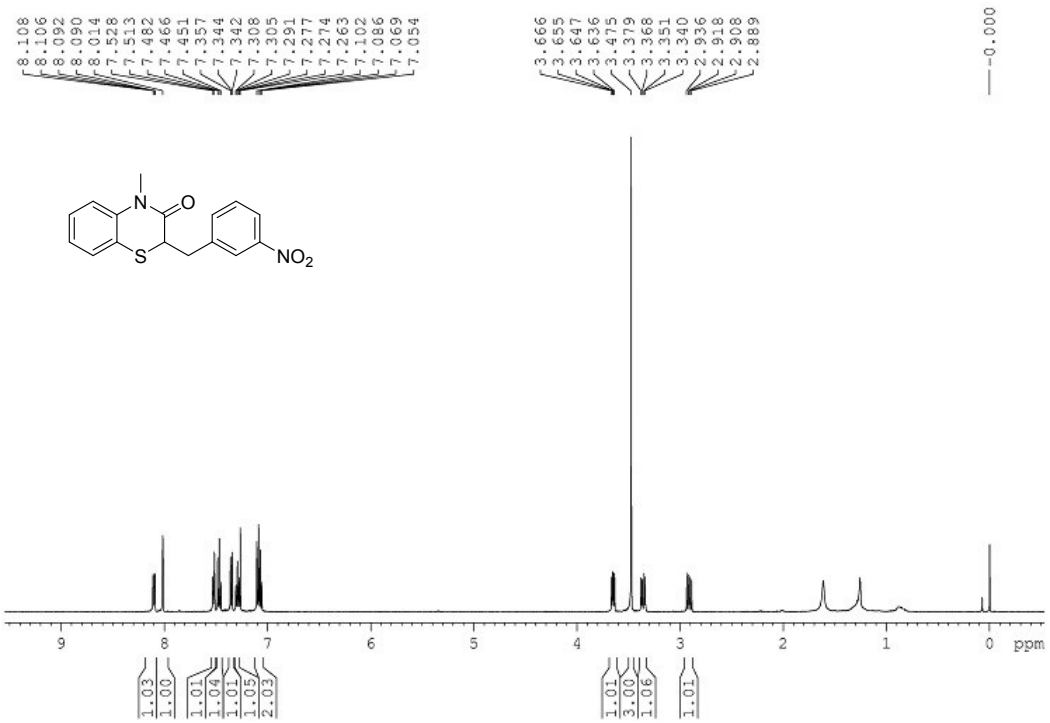
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7.357
7.344
7.342
7.308
7.305
7.291
7.277
7.274
7.263
7.102
7.086
7.069
7.054
3.666
3.655
3.647
3.636
3.475
3.379
3.368
3.351
3.340
2.936
2.918
2.908
2.889
0.000
N
O
S
NO2
9
8
7
6
5
4
3
2
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1.01
1.04
1.01
1.05
2.03
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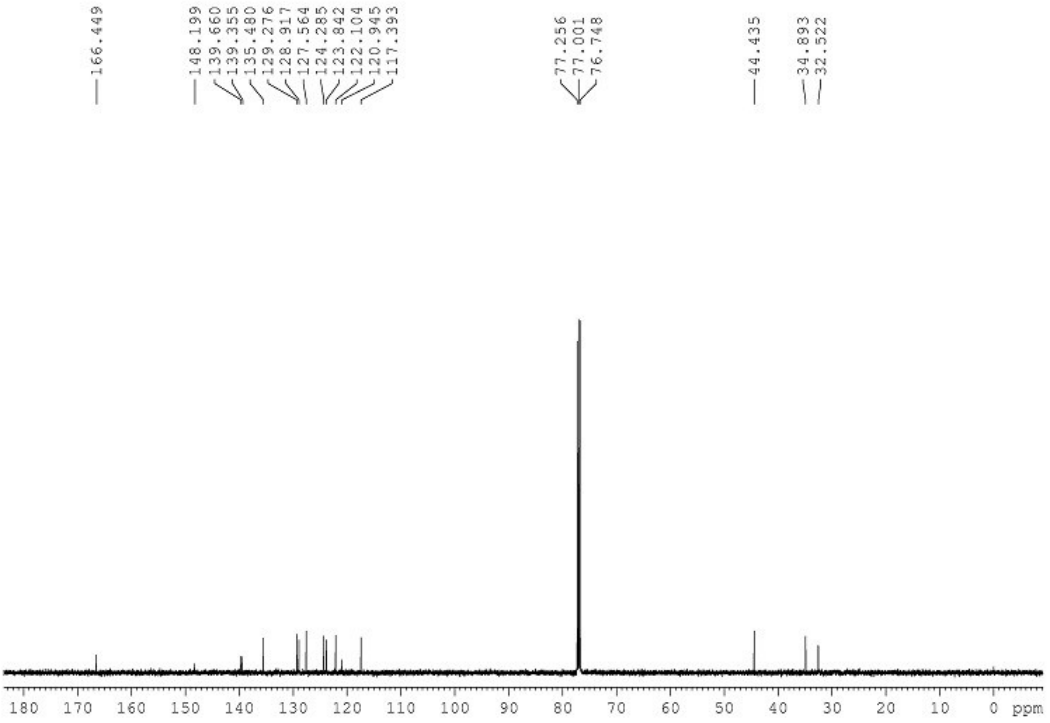
3.00
1.06
1.01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122.104
120.945
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77.001
76.748
44.435
34.893
32.522
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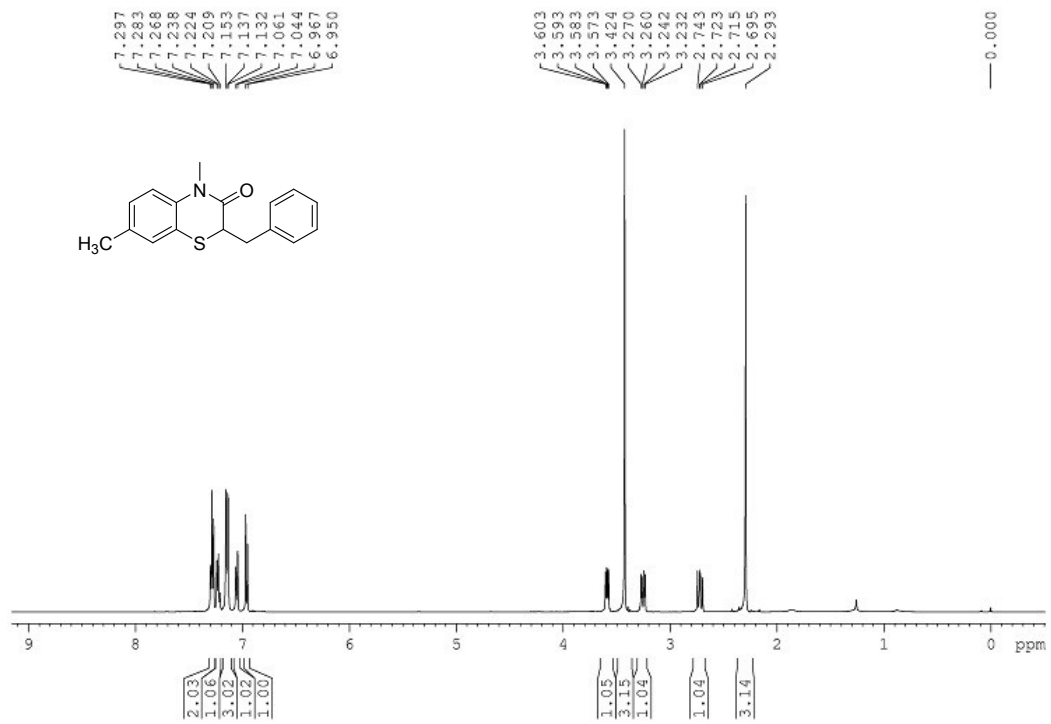
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7.137
7.132
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7.044
6.967
6.950
3.603
3.593
3.583
3.573
3.424
3.270
3.260
3.242
3.232
2.743
2.723
2.715
2.695
2.293
0.000
N
O
S
H3C
9
8
7
6
5
4
3
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1.06
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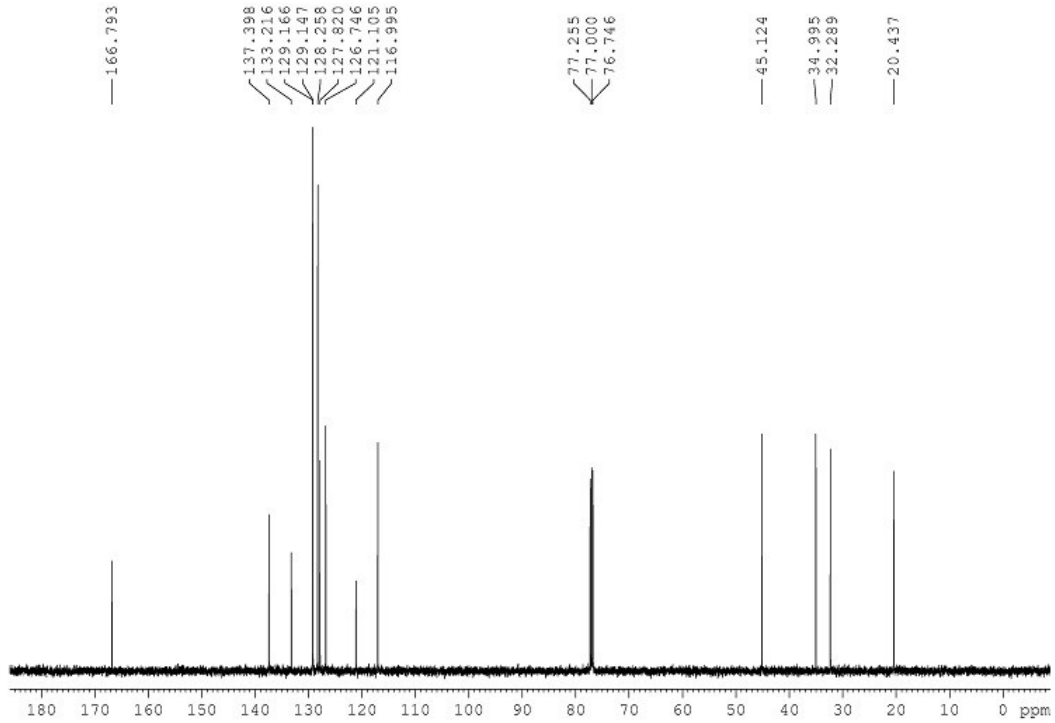
1.04
3.14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20.437
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140
130
120
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60
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20
10
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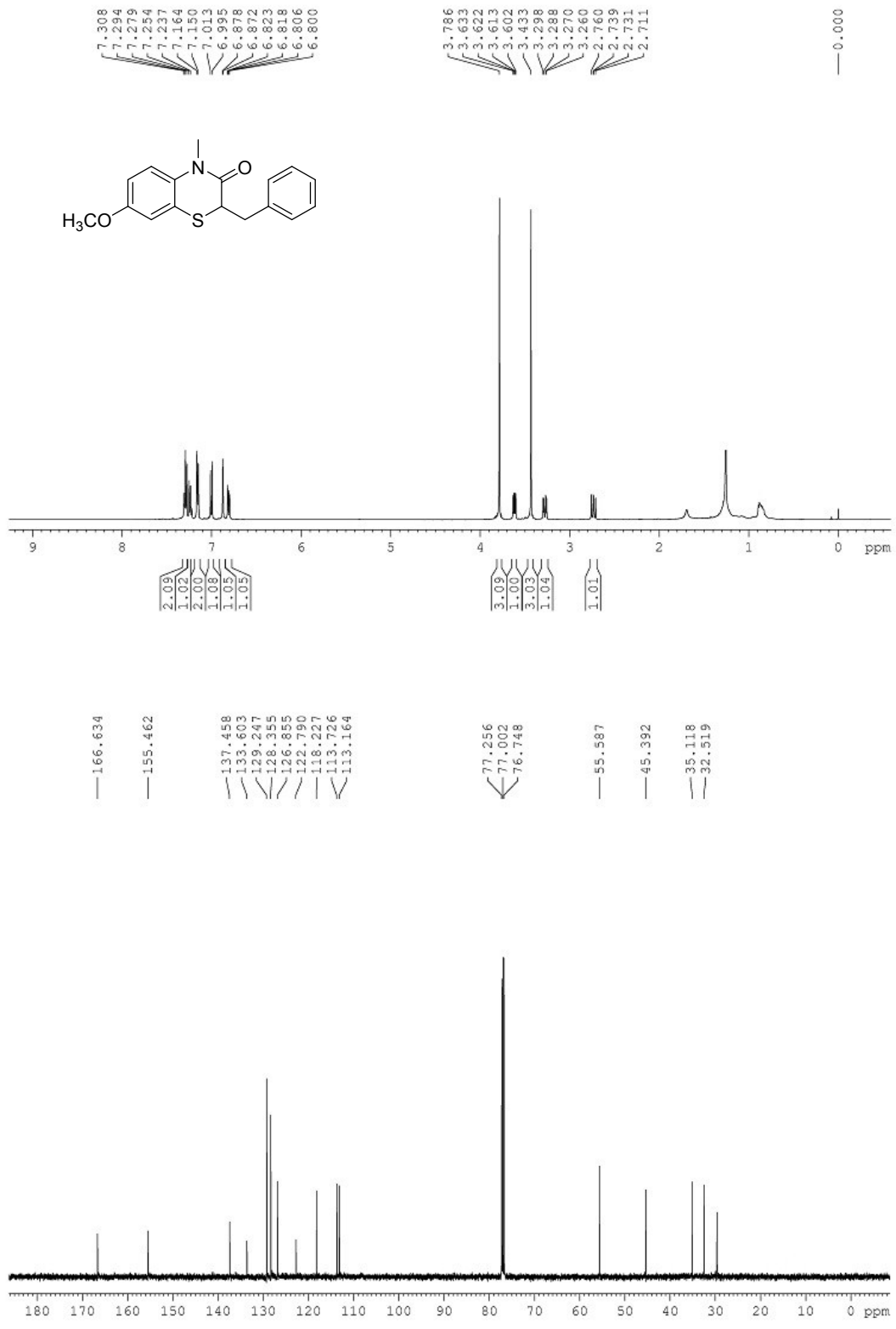
ppm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7.013
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6.872
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6.818
6.806
6.800
3.786
3.633
3.622
3.613
3.602
3.433
3.298
3.288
3.270
3.260
2.760
2.739
2.731
2.711
0.000
N
O
H3CO
S
ppm
2.09
1.02
2.00
1.08
1.05
1.05
3.09
1.00
3.03
1.04
1.01
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133.603
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128.355
126.855
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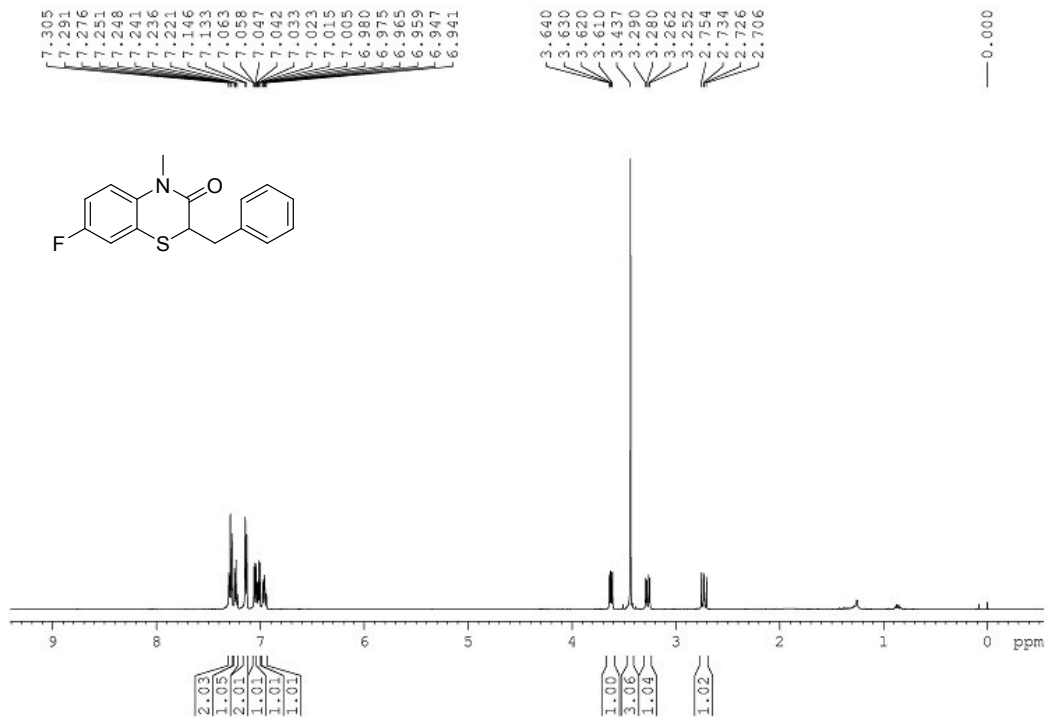
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7.251
7.248
7.241
7.236
7.221
7.146
7.133
7.063
7.058
7.047
7.042
7.033
7.023
7.015
7.005
6.980
6.975
6.965
6.959
6.947
6.941
3.640
3.630
3.620
3.610
3.437
3.290
3.280
3.262
3.252
2.754
2.734
2.726
2.706
0.000
N
O
F
S
9
8
7
6
5
4
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1.01
1.01
1.01
1.00
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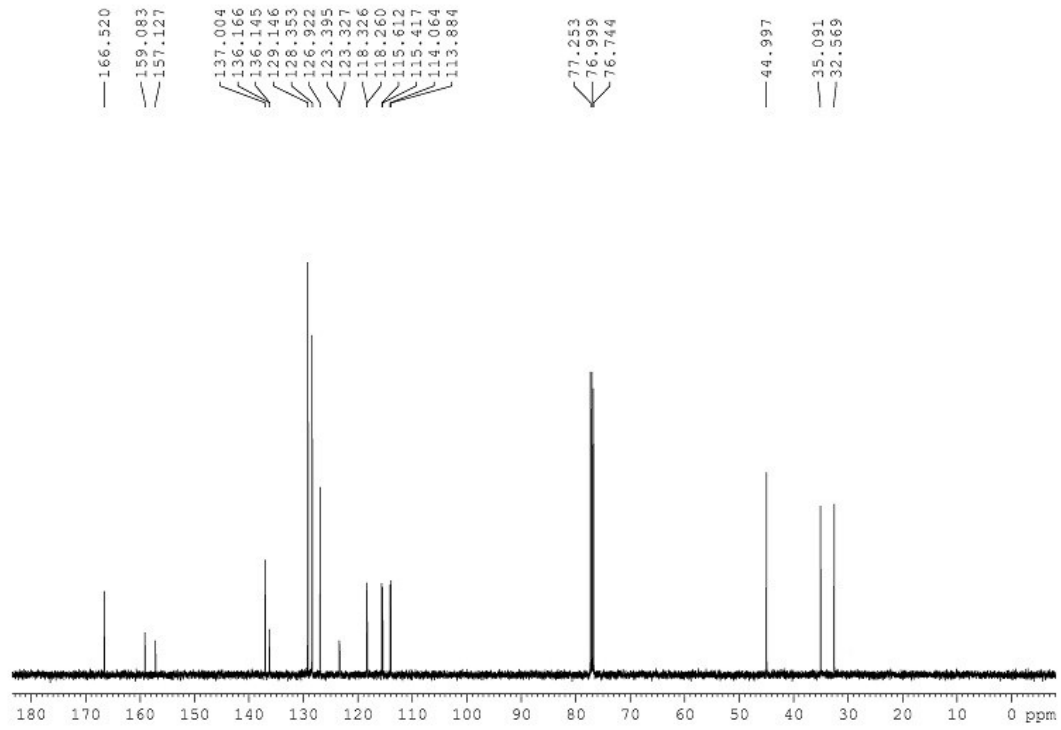
1.04
1.02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115.417
114.064
113.884
77.253
76.999
76.744
44.997
35.091
32.569
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170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm


7.305
7.301
7.291
7.276
7.251
7.237
7.221
7.217
7.204
7.199
7.145
7.130
6.989
6.971
3.630
3.619
3.610
3.599
3.427
3.280
3.269
3.251
3.241
2.756
2.736
2.727
2.708
0.000
N
O
Cl
S
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
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1.10
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1.00
1.00
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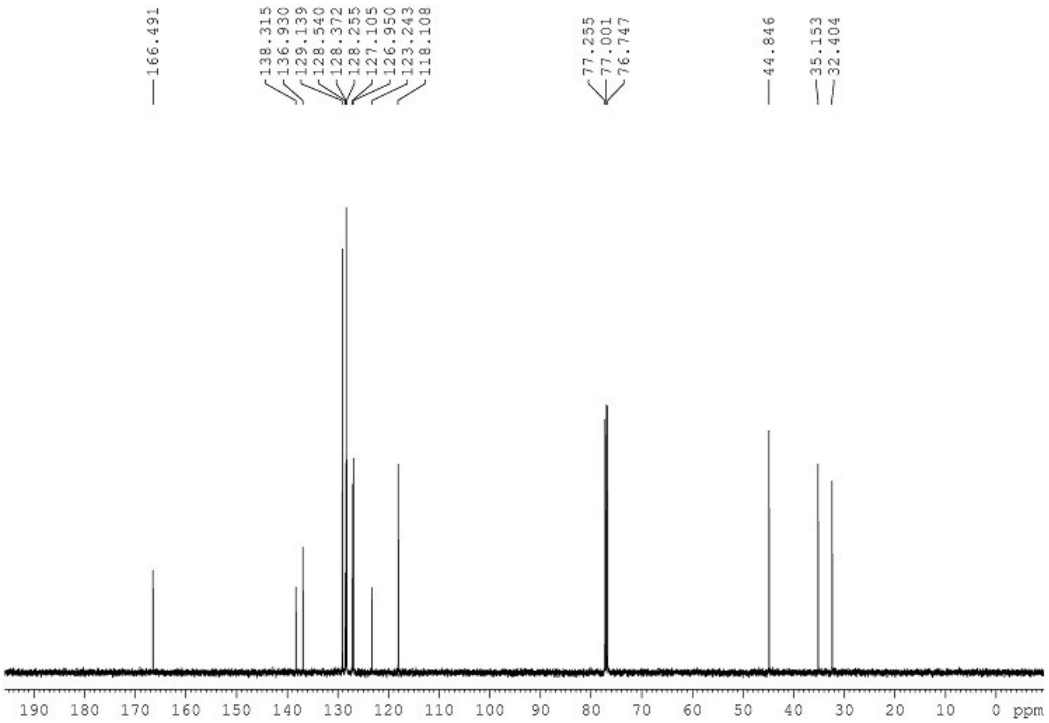
1.03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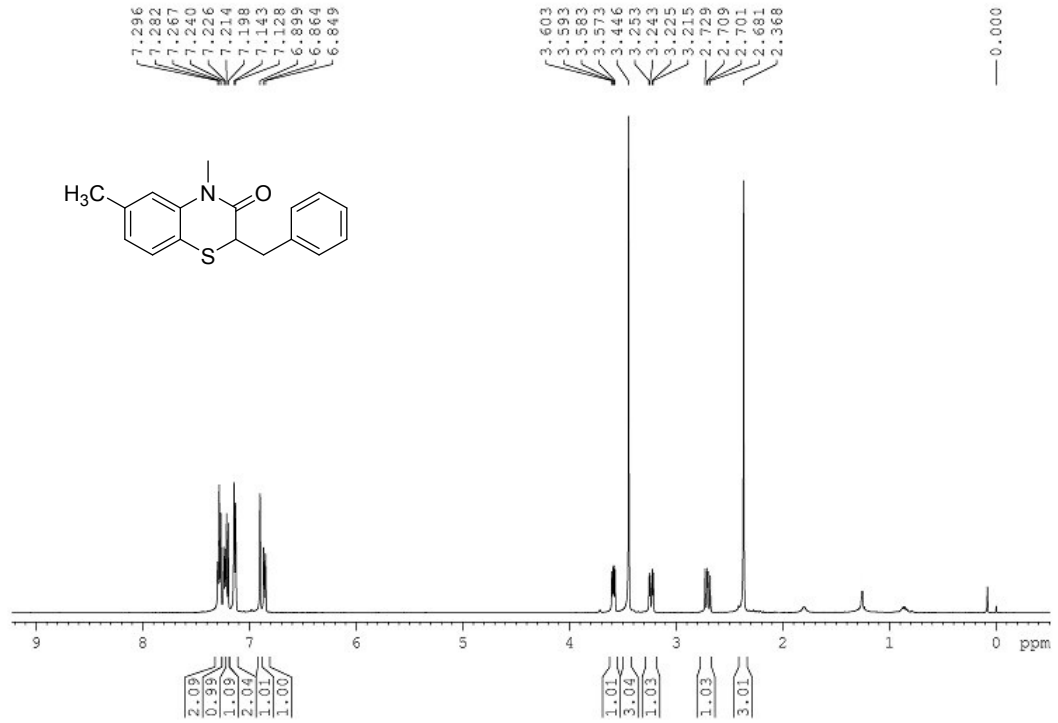
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6.864
6.849
3.603
3.593
3.583
3.573
3.446
3.253
3.243
3.225
3.215
2.729
2.709
2.701
2.681
2.368
0.000
H3C
N
O
S
9
8
7
6
5
4
3
2
1
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1.09
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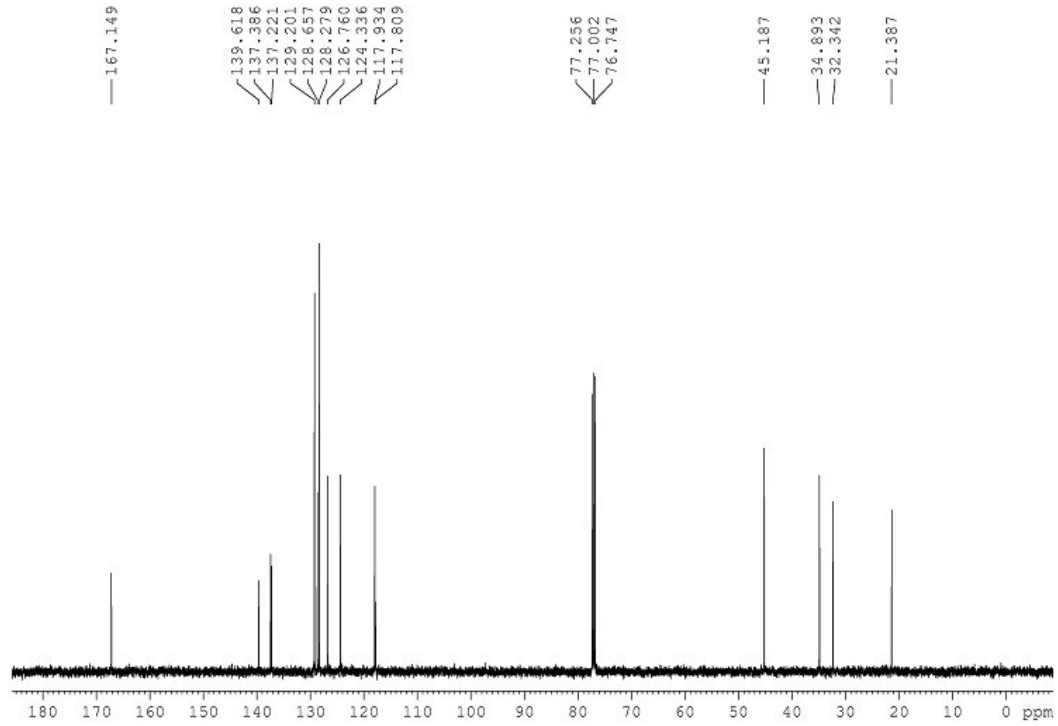
1.00
1.01
3.04
1.03
1.03
3.01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117.809
77.256
77.002
76.747
45.187
34.893
32.342
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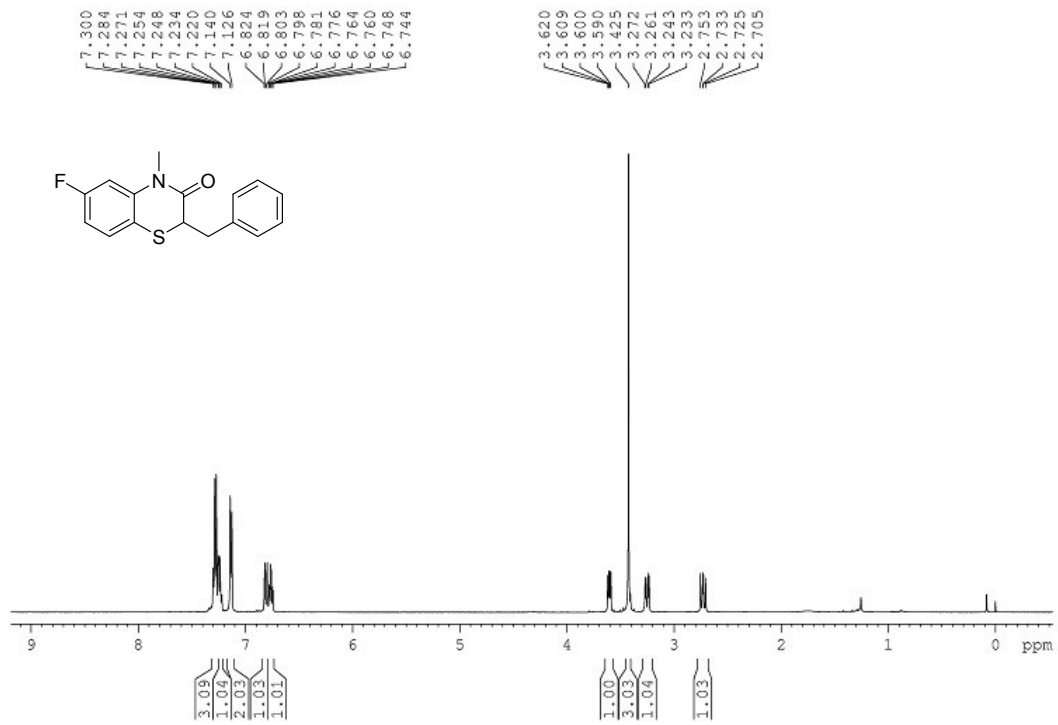
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm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7.126
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6.803
6.798
6.781
6.776
6.764
6.760
6.748
6.744
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3.609
3.600
3.590
3.425
3.272
3.261
3.243
3.233
2.753
2.733
2.725
2.705
F
N
O
S
9
8
7
6
5
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1.01
1.00
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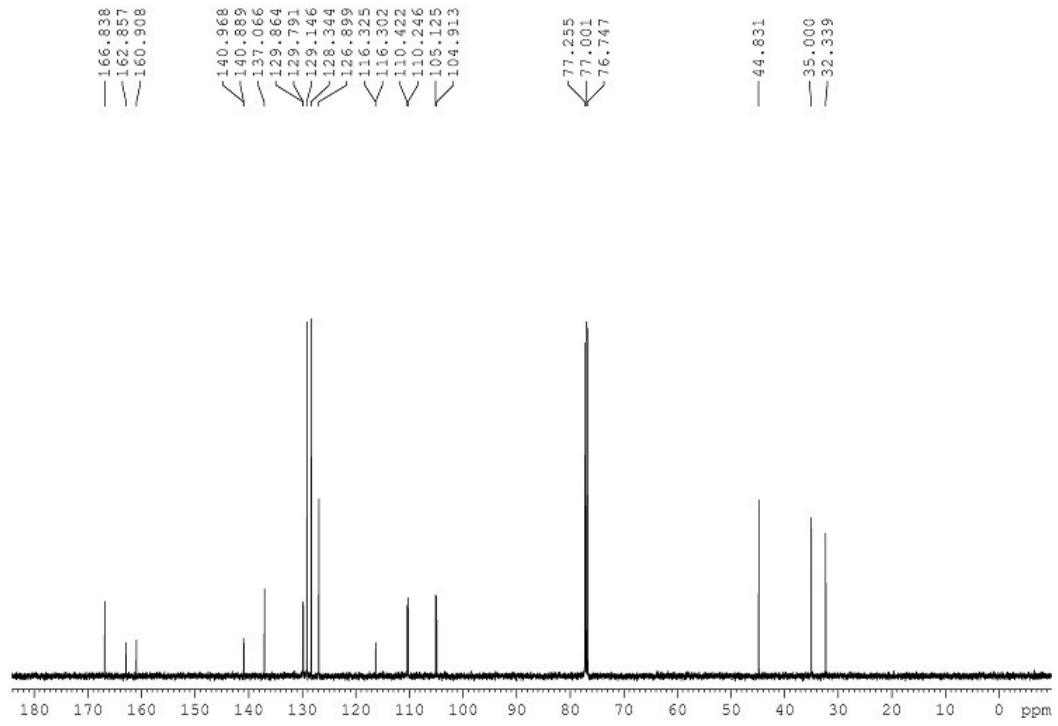
1.04
1.03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116.302
110.422
110.246
105.125
104.913
77.255
77.001
76.747
44.831
35.000
32.339
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160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


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7.267
7.231
7.216
7.211
7.208
7.197
7.183
7.023
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6.991
6.976
6.960
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3.805
3.797
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3.371
3.205
3.193
3.176
3.165
2.722
2.703
2.694
2.674
2.500
H
N
O
S
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10
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8
7
6
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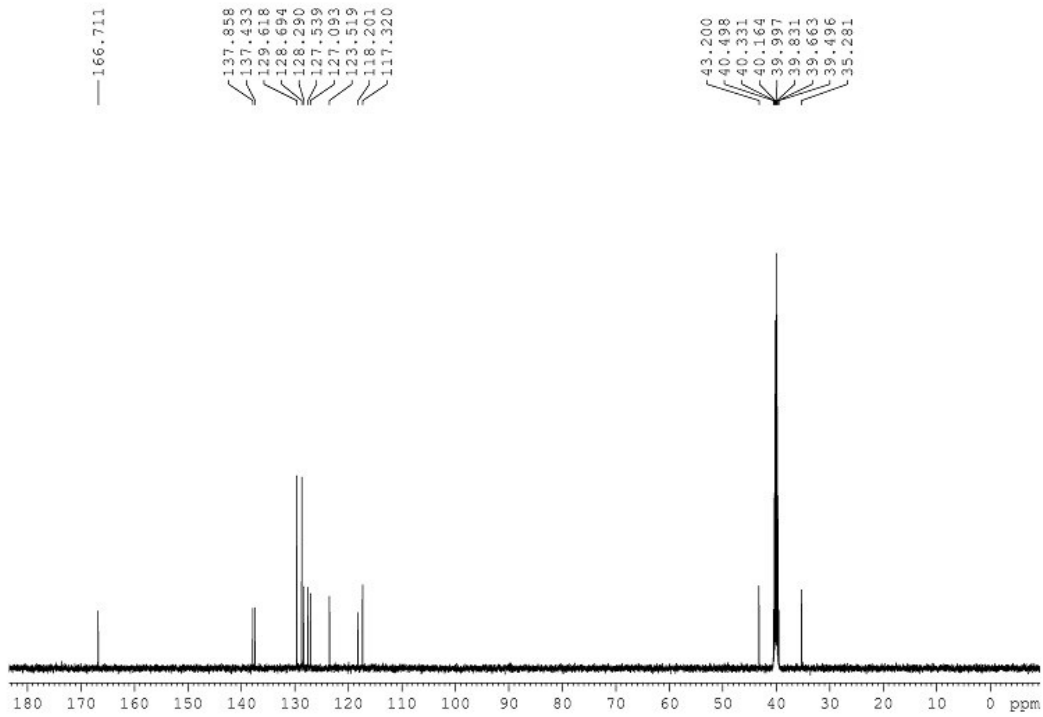
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117.320
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40.331
40.164
39.997
39.831
39.663
39.496
35.281
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130
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60
50
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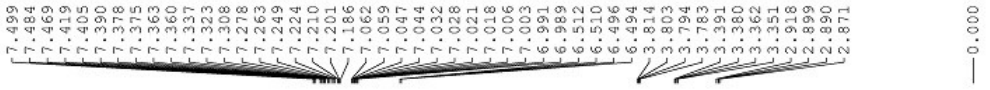
30
20
10
0
ppm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7.249
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7.210
7.201
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7.062
7.059
7.047
7.044
7.032
7.028
7.021
7.018
7.006
7.003
6.991
6.989
6.512
6.510
6.496
6.494
3.814
3.803
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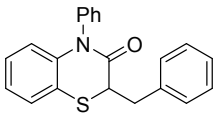
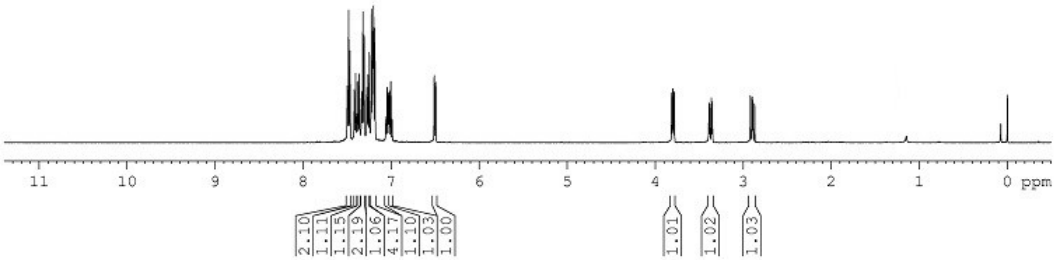

Ph
N
O
S


11
10
9
8
7
6
5
4
3
2
1
0 ppm
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1.15
2.19
1.06
4.17
1.10
1.03
1.00
1.01
1.02
1.03


166.478
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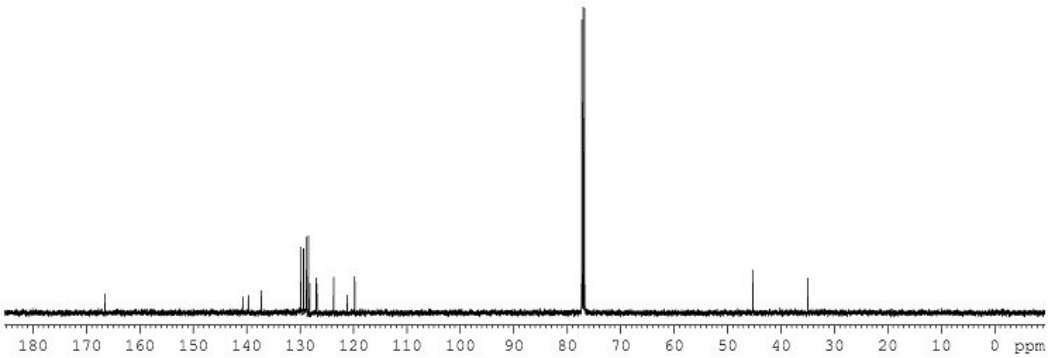
139.690
137.220
129.798
129.358
128.813
128.741
128.438
128.176
126.992
126.831
123.692
121.172
119.763
77.255
77.001
76.747
45.286
34.980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160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
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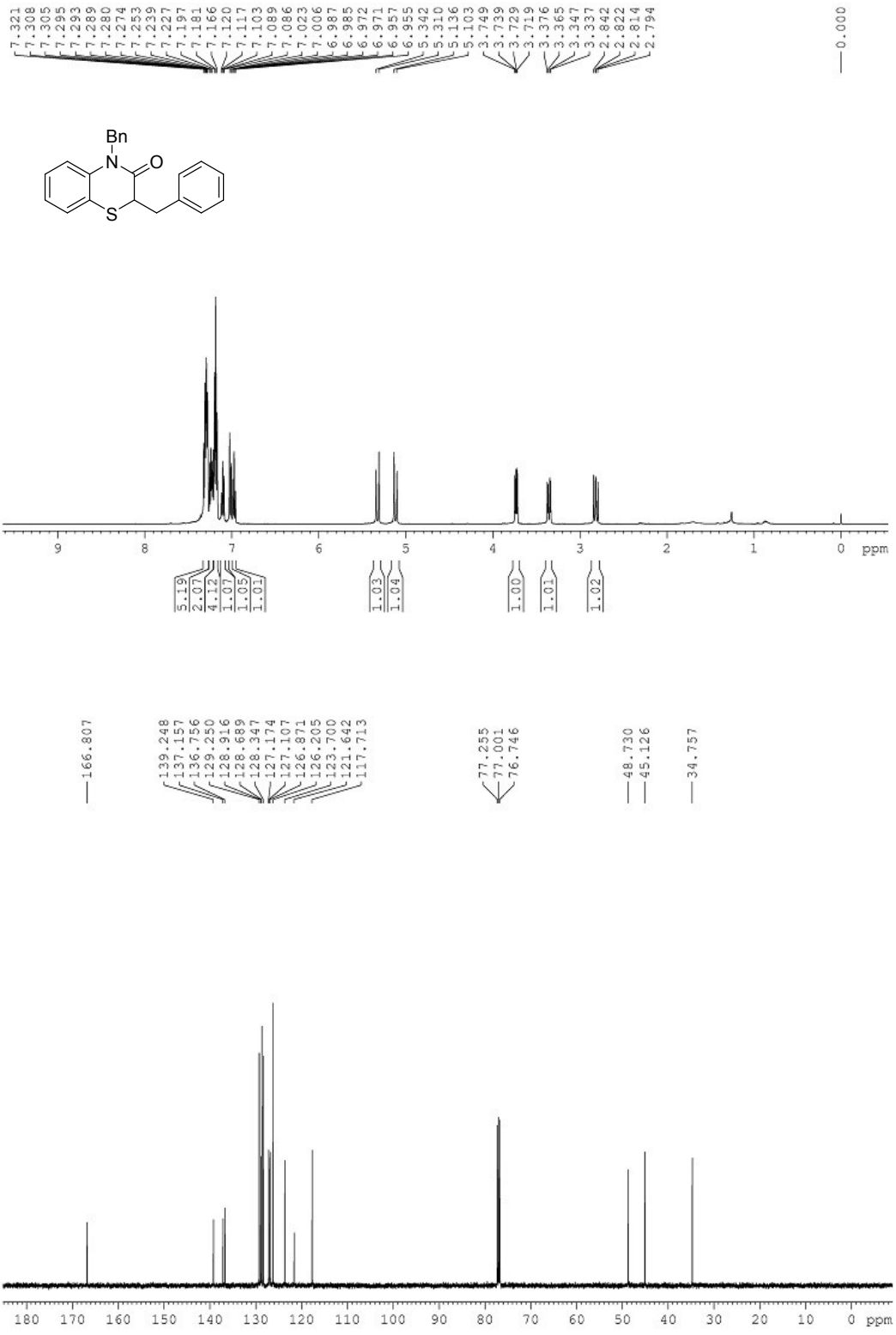
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7.305
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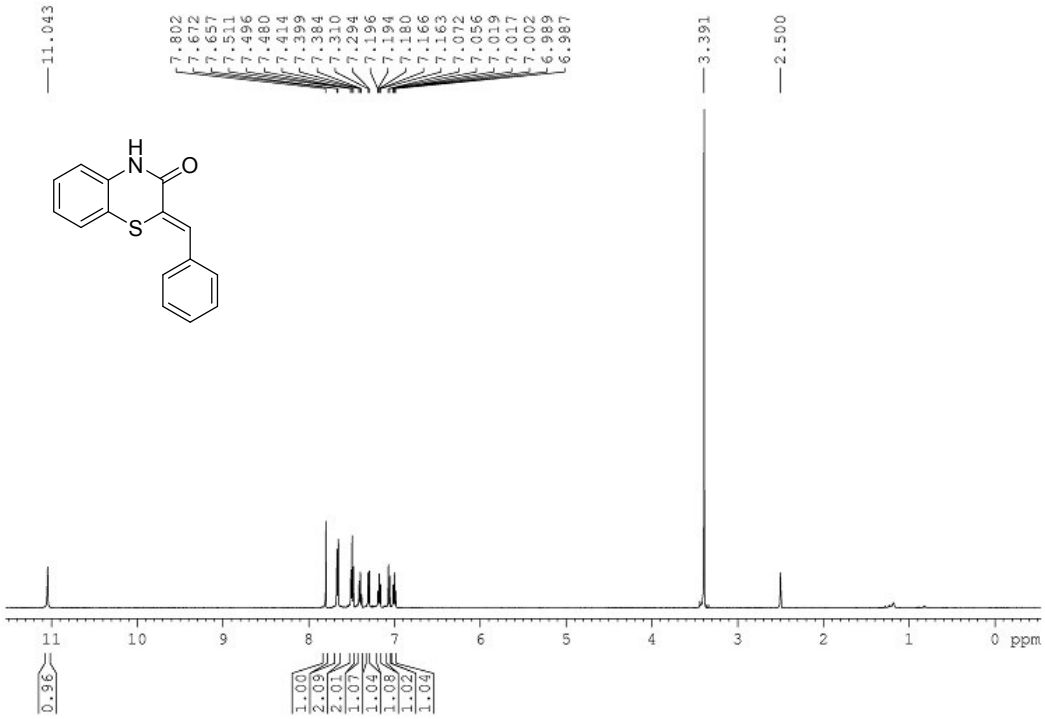
7.289
7.280
7.274
7.253
7.239
7.227
7.197
7.181
7.166
7.120
7.117
7.103
7.089
7.086
7.023
7.006
6.987
6.985
6.972
6.971
6.957
6.955
5.342
5.310
5.136
5.103
3.749
3.739
3.729
3.719
3.376
3.365
3.347
3.337
2.842
2.822
2.814
2.794
0.000
Bn
N
O
S
9
8
7
6
5
4
3
2
1
0
ppm
5.19
2.07
4.12
1.07
1.05
1.01
1.03
1.04
1.00
1.01
1.02
166.807
139.248
137.157
136.756
129.250
128.916
128.689
128.347
127.174
127.107
126.871
126.205
123.700
121.642
117.713
77.255
77.001
76.746
48.730
45.126
34.757
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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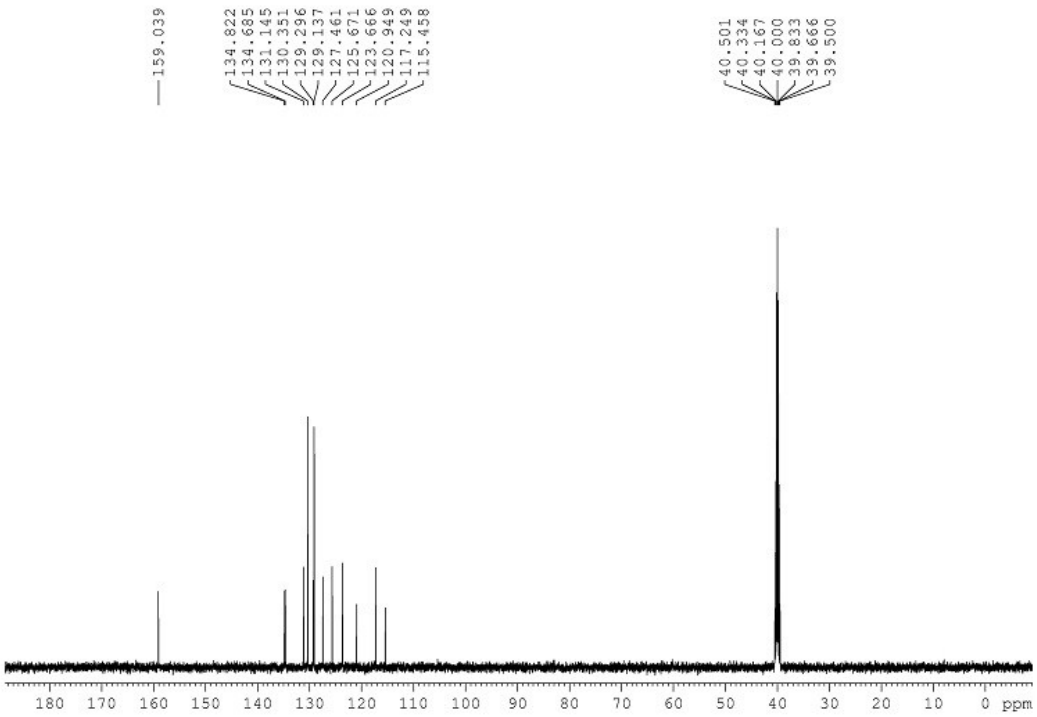
7.802
7.672
7.657
7.511
7.496
7.480
7.414
7.399
7.384
7.310
7.294
7.196
7.194
7.180
7.166
7.163
7.072
7.056
7.019
7.017
7.002
6.989
6.987
3.391
2.500
H
N
O
S
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.96
1.00
2.09
2.01
1.07
1.04
1.08
1.02
1.04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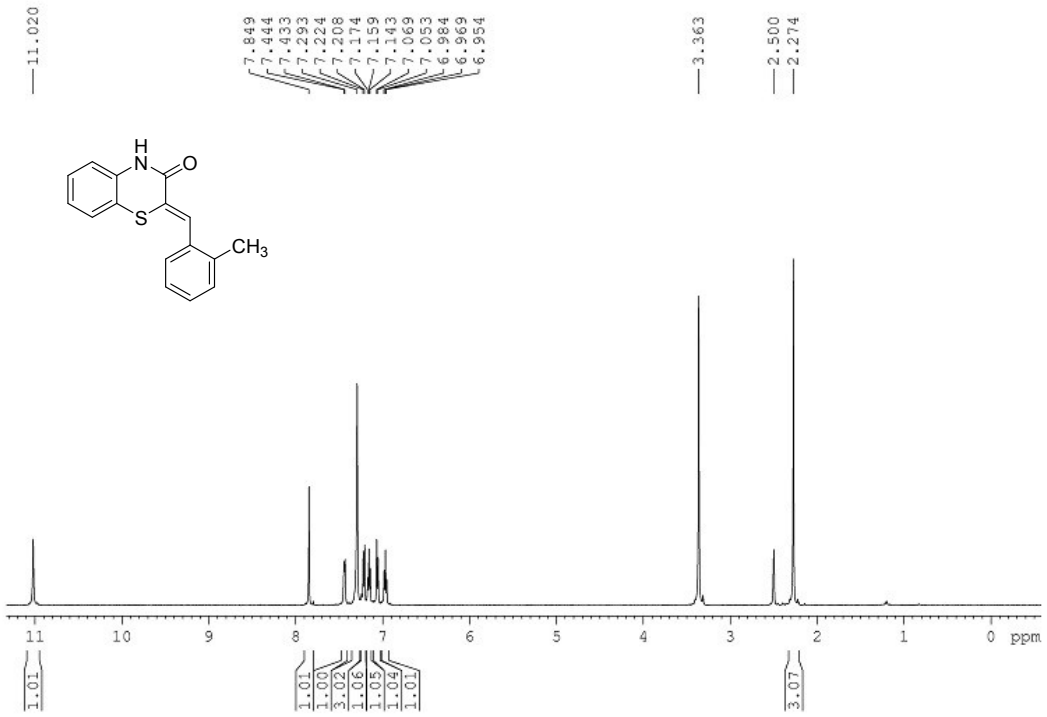
134.685
131.145
130.351
129.296
129.137
127.461
125.671
123.666
120.949
117.249
115.458
40.501
40.334
40.167
40.000
39.833
39.666
39.500
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm


11.020
7.849
7.444
7.433
7.293
7.224
7.208
7.174
7.159
7.143
7.069
7.053
6.984
6.969
6.954
3.363
2.500
2.274
H
N
O
S
CH3
11
10
9
8
7
6
5
4
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2
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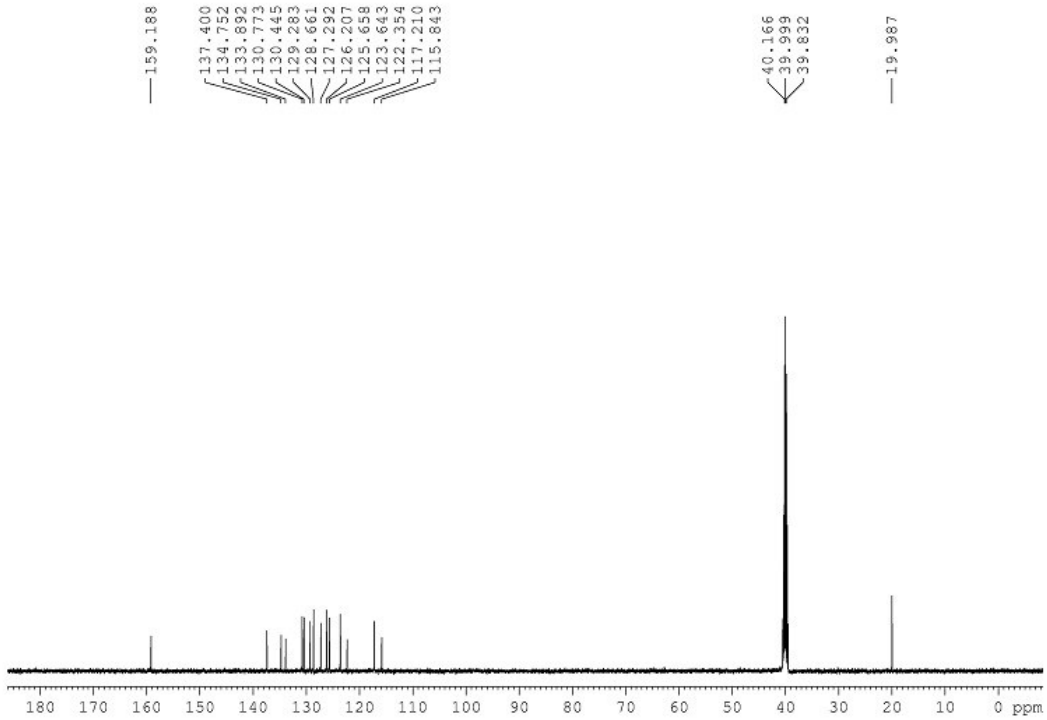
1.01
1.01
1.00
3.02
1.06
1.05
1.04
1.01
3.07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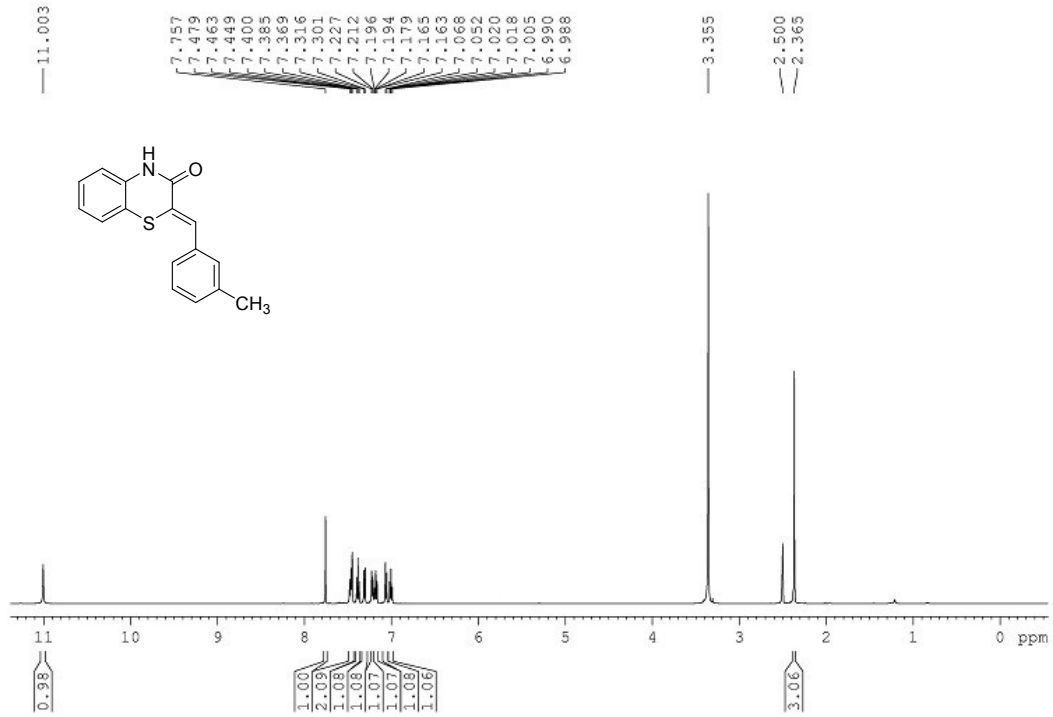
137.400
134.752
133.892
130.773
130.445
129.283
128.661
127.292
126.207
125.658
123.643
122.354
117.210
115.843
40.166
39.999
39.832
19.987
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm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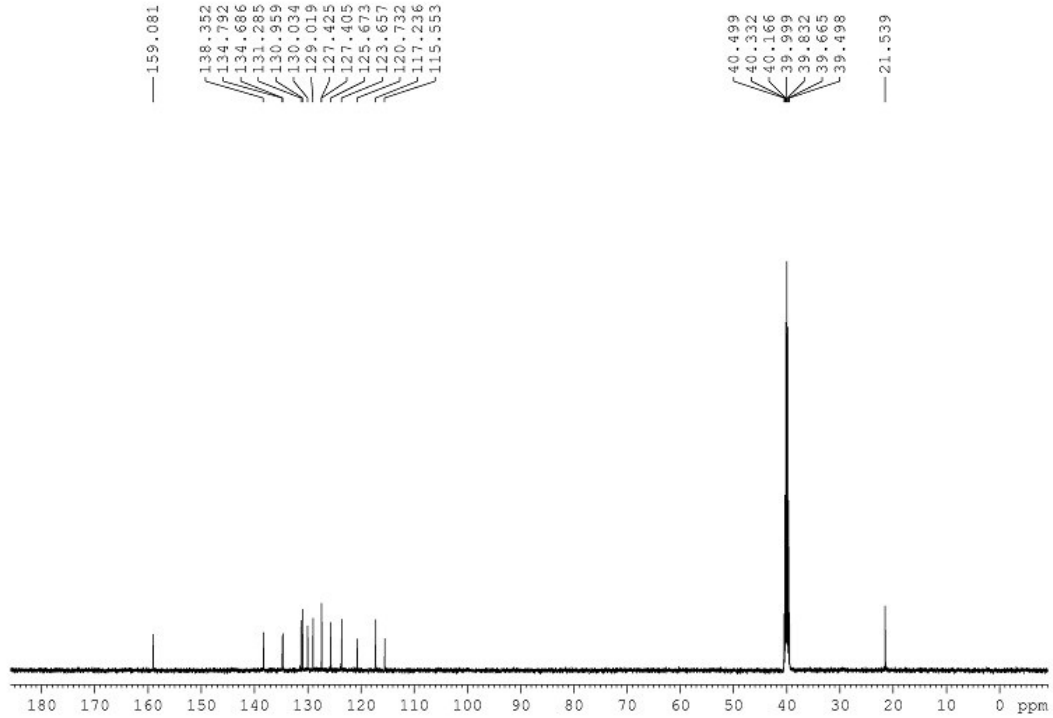
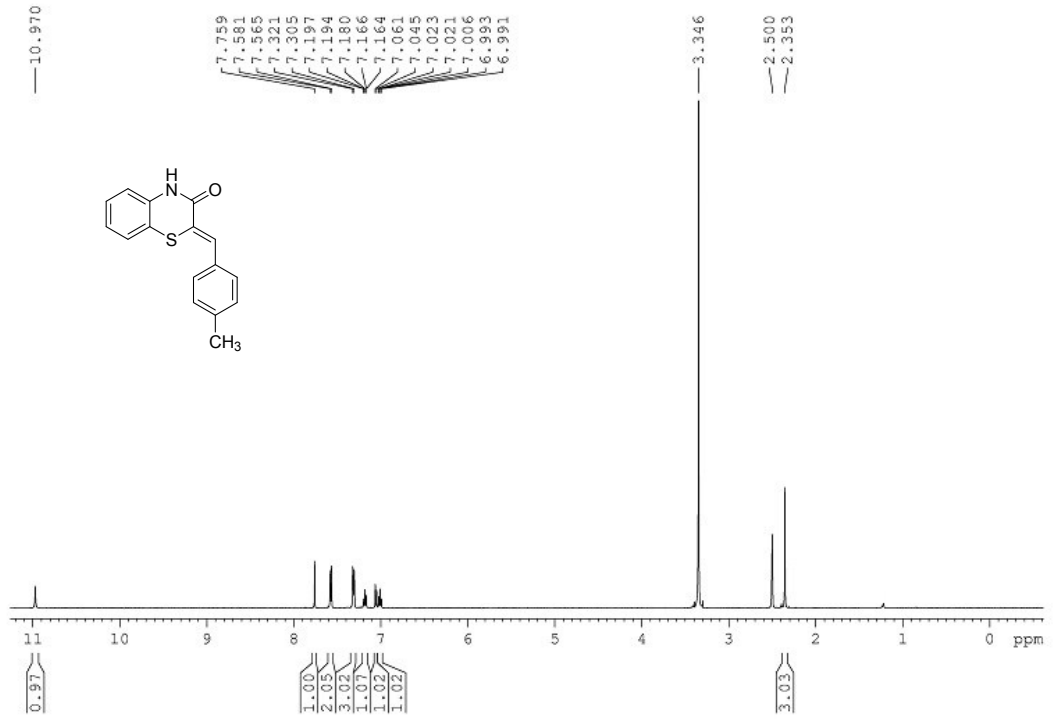
7.757
7.479
7.463
7.449
7.400
7.385
7.369
7.316
7.301
7.227
7.212
7.196
7.194
7.179
7.165
7.163
7.068
7.052
7.020
7.018
7.005
6.990
6.988
3.355
2.500
2.365
H
N
O
S
CH3
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.98
1.00
2.09
1.08
1.08
1.07
1.07
1.08
1.06
3.06


159.081
138.352
134.792
134.686
131.285
130.959
130.034
129.019
127.425
127.405
125.673
123.657
120.732
117.236
115.553
40.499
40.332
40.166
39.999
39.832
39.665
39.498
21.539
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm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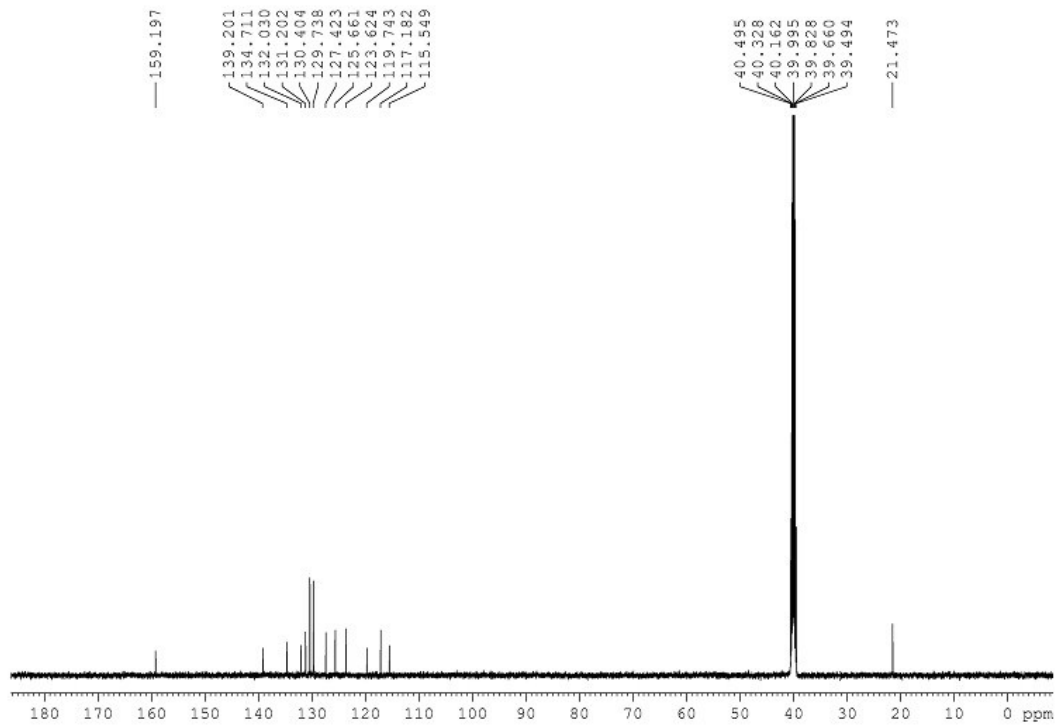
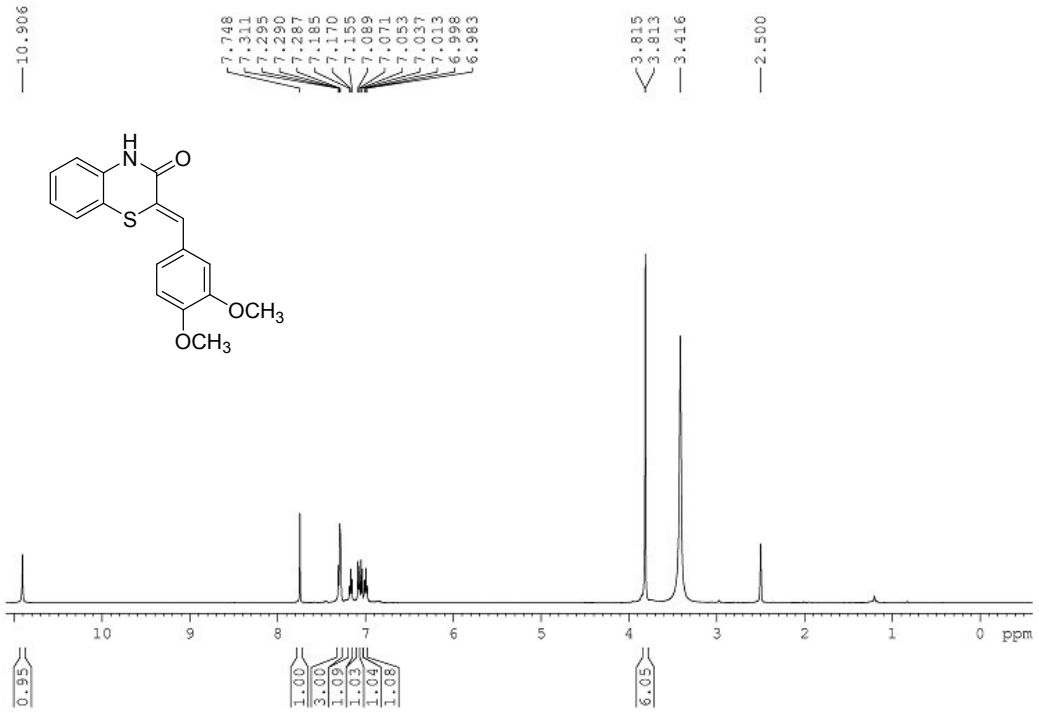
7.581
7.565
7.321
7.305
7.197
7.194
7.180
7.166
7.164
7.061
7.045
7.023
7.021
7.006
6.993
6.991
3.346
2.500
2.353
H
N
O
S
CH3
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.97
1.00
2.05
3.02
1.07
1.02
1.02
3.03


159.197
139.201
134.711
132.030
131.202
130.404
129.738
127.423
125.661
123.624
119.743
117.182
115.549
40.495
40.328
40.162
39.995
39.828
39.660
39.494
21.473
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm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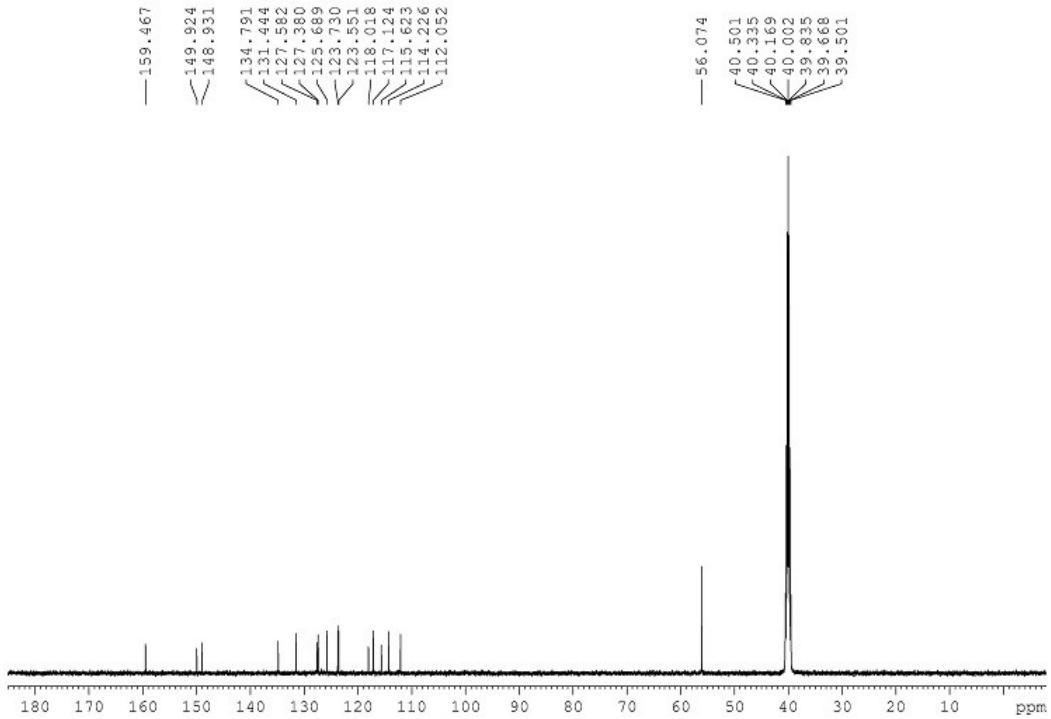
7.311
7.295
7.290
7.287
7.185
7.170
7.155
7.089
7.071
7.053
7.037
7.013
6.998
6.983
3.815
3.813
3.416
2.500
H
N
O
S
OCH3
OCH3
10
9
8
7
6
5
4
3
2
1
0
ppm
0.95
1.00
3.00
1.09
1.03
1.04
1.08
6.05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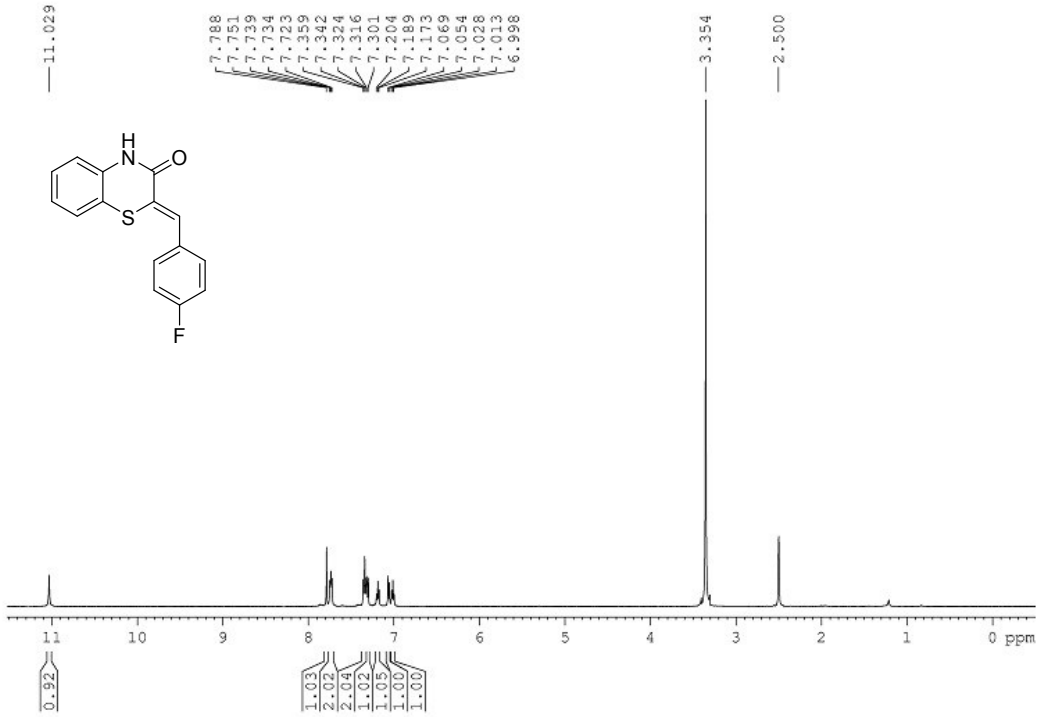
148.931
134.791
131.444
127.582
127.380
125.689
123.730
123.551
118.018
117.124
115.623
114.226
112.052
56.074
40.501
40.335
40.169
40.002
39.835
39.668
39.501
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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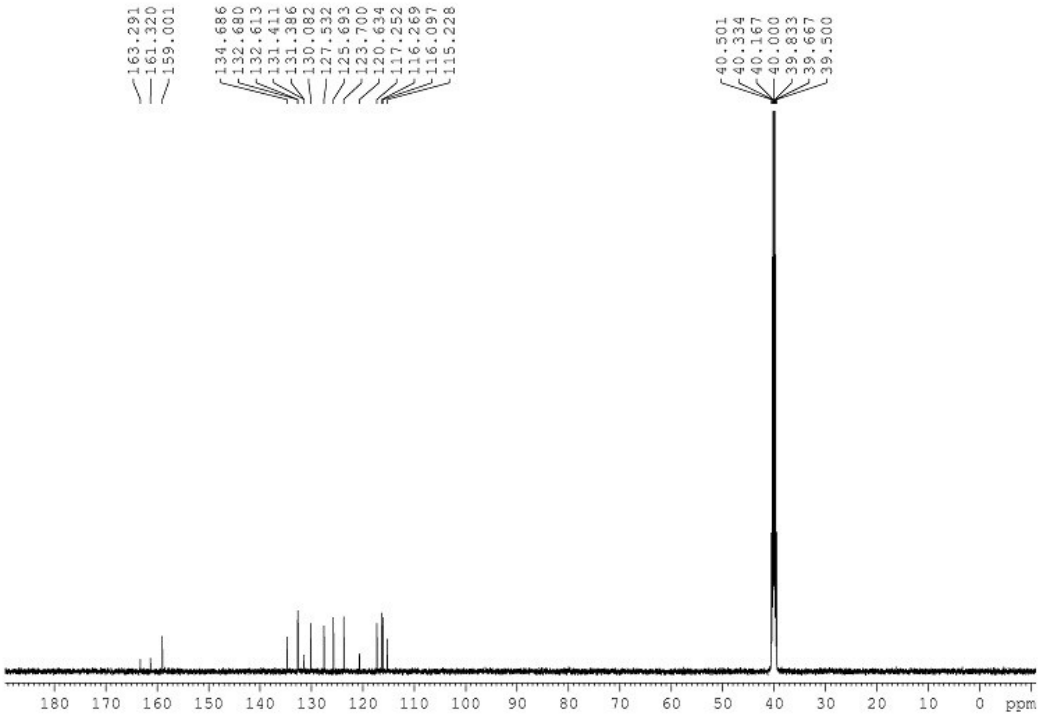
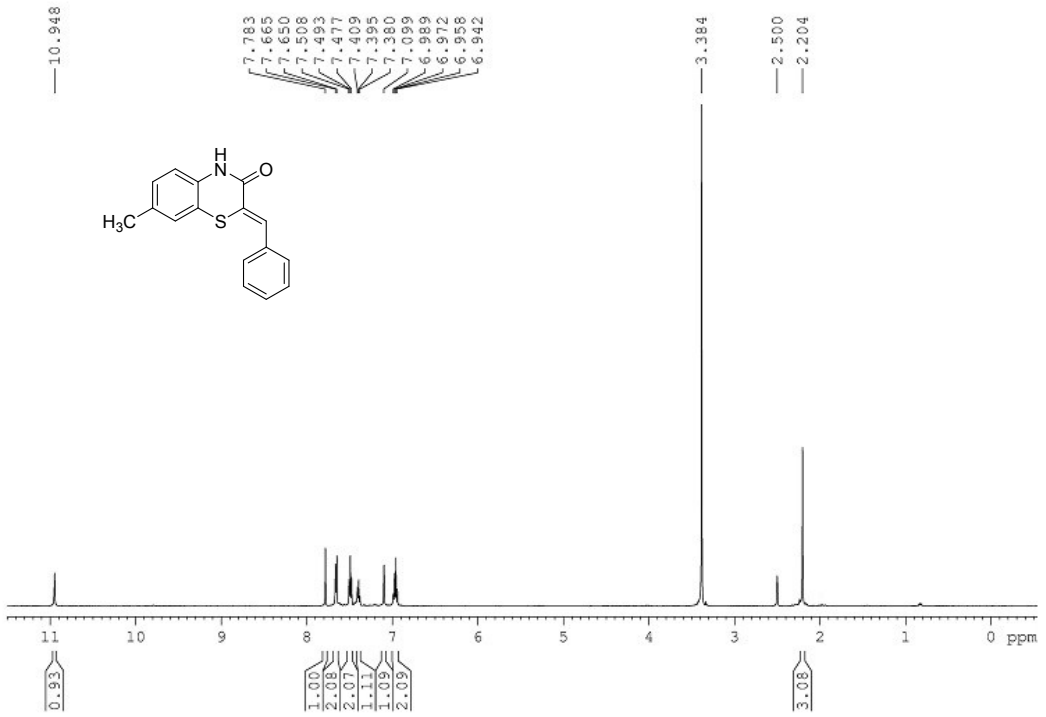
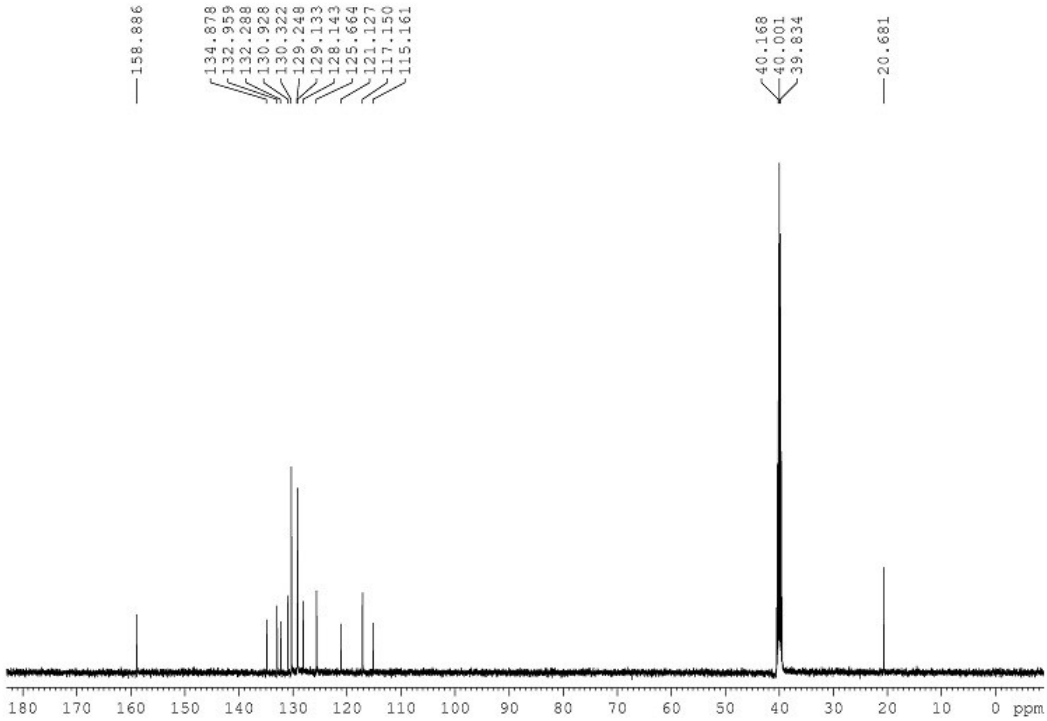
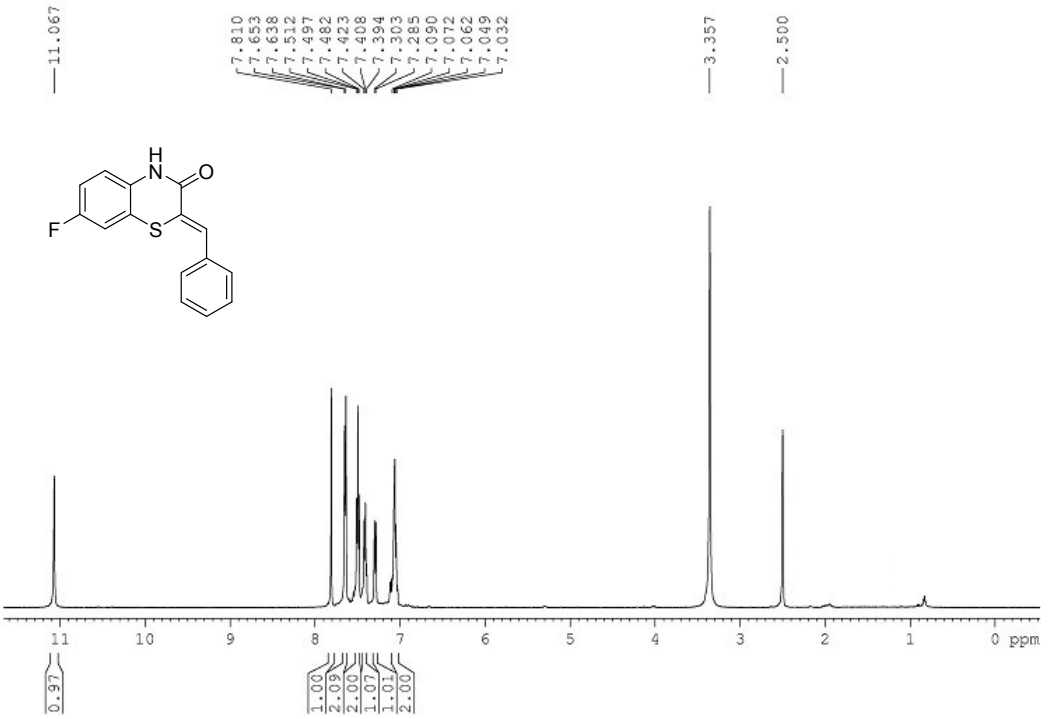
7.788
7.751
7.739
7.734
7.723
7.359
7.342
7.324
7.316
7.301
7.204
7.189
7.173
7.069
7.054
7.028
7.013
6.998
3.354
2.500
H
N
O
S
F
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.92
1.03
2.02
2.04
1.02
1.05
1.00
1.00


163.291
161.320
159.001
134.686
132.680
132.613
131.411
131.386
130.082
127.532
125.693
123.700
120.634
117.252
116.269
116.097
115.228
40.501
40.334
40.167
40.000
39.833
39.667
39.500
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm


10.948
7.783
7.665
7.650
7.508
7.493
7.477
7.409
7.395
7.380
7.099
6.989
6.972
6.958
6.942
3.384
2.500
2.204
H
N
O
H3C
S
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.93
1.00
2.08
2.07
1.11
1.09
2.09
3.08


158.886
134.878
132.959
132.288
130.928
130.322
129.248
129.133
128.143
125.664
121.127
117.150
115.161
40.168
40.001
39.834
20.681
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm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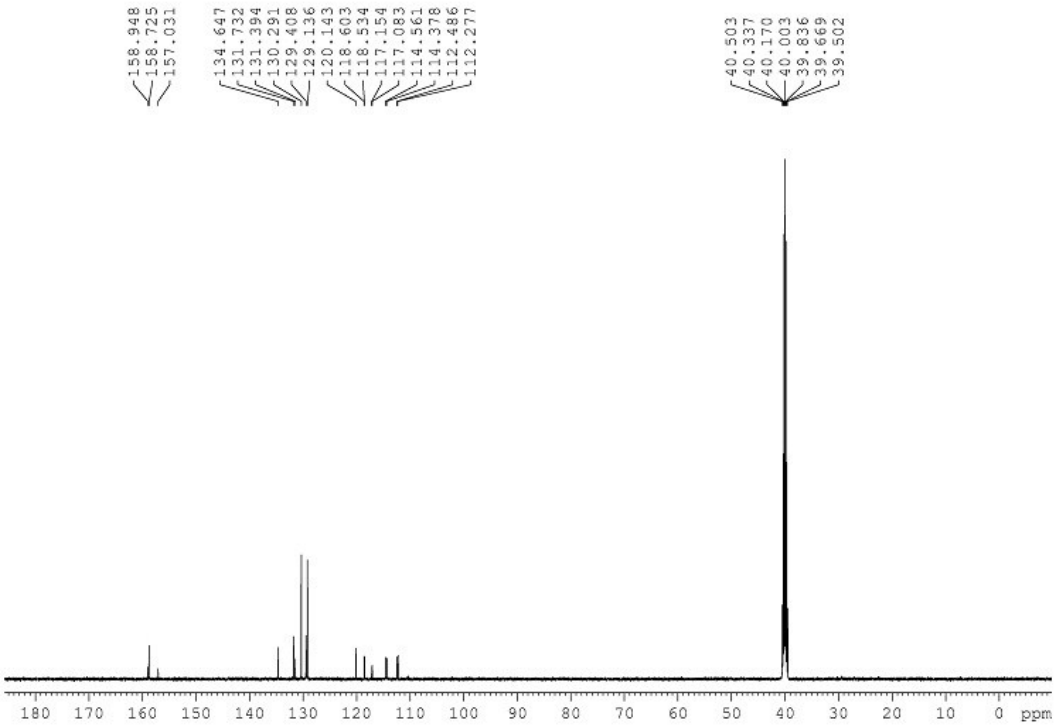
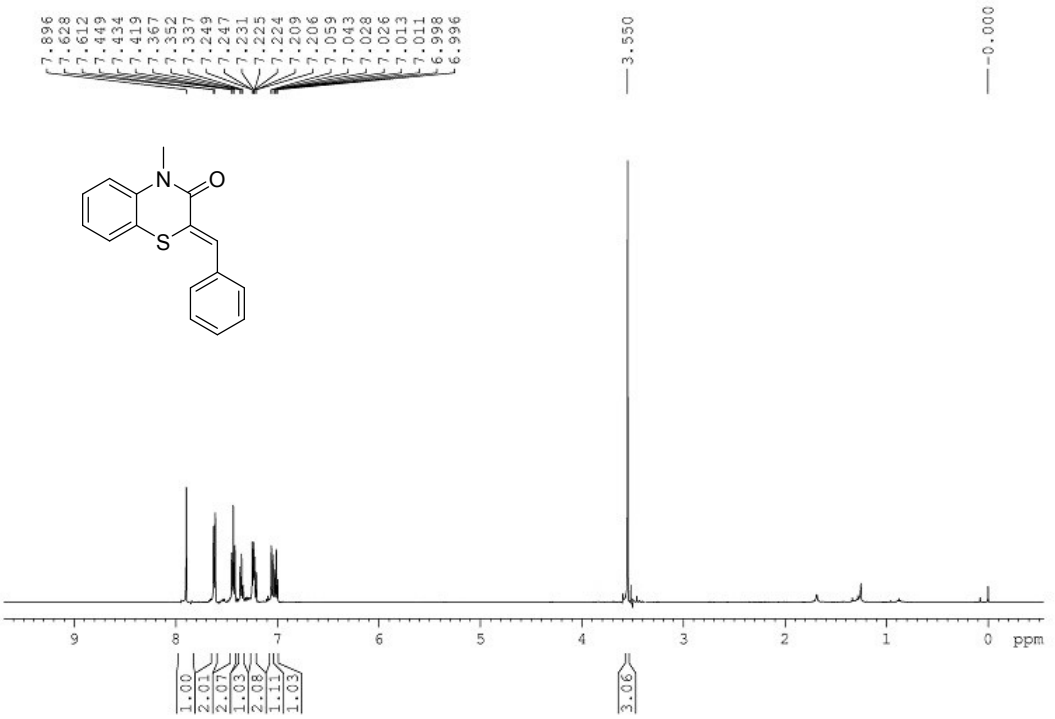
7.810
7.653
7.638
7.512
7.497
7.482
7.423
7.408
7.394
7.303
7.285
7.090
7.072
7.062
7.049
7.032
3.357
2.500
H
N
O
F
S
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.97
1.00
2.05
2.00
1.07
1.01
2.00


158.948
158.725
157.031
134.647
131.732
131.394
130.291
129.408
129.136
120.143
118.603
118.534
117.154
117.083
114.561
114.378
112.486
112.277
40.503
40.337
40.170
40.003
39.836
39.669
39.502
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm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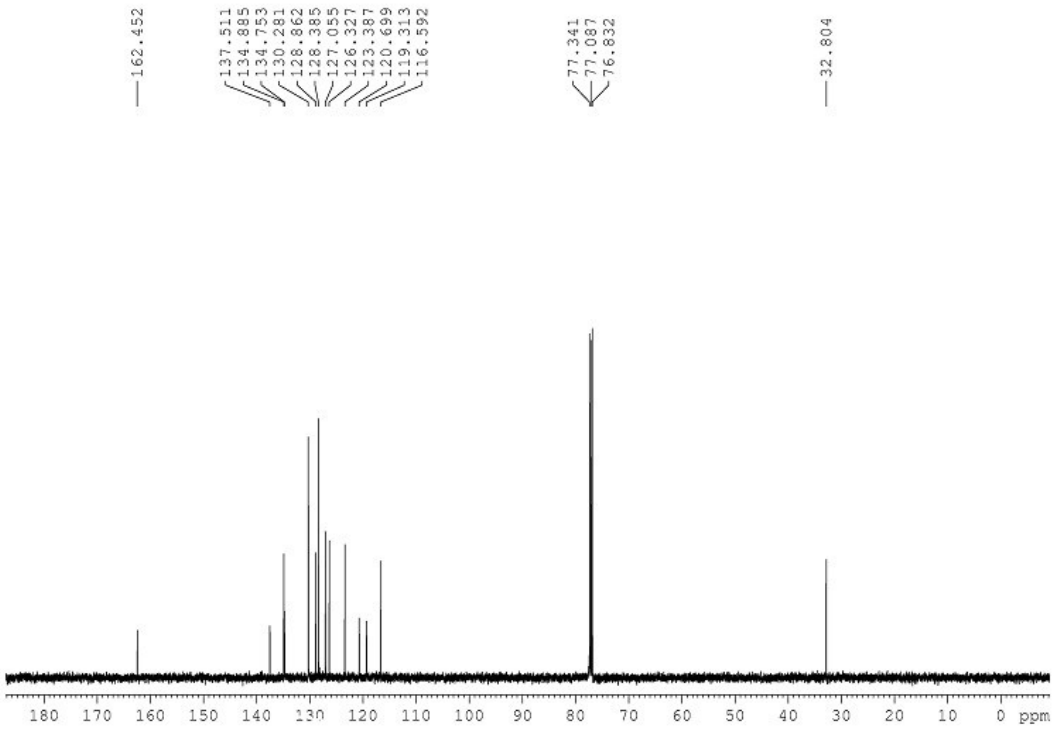
7.612
7.449
7.434
7.419
7.367
7.352
7.337
7.249
7.247
7.231
7.225
7.224
7.209
7.206
7.059
7.043
7.028
7.026
7.013
7.011
6.998
6.996
3.550
0.000
N
O
S
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.01
2.07
1.03
2.08
1.11
1.03
3.06


162.452
137.511
134.885
134.753
130.281
128.862
128.385
127.055
126.327
123.387
120.699
119.313
116.592
77.341
77.087
76.832
32.804
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

8) Crystal Data

Table 1 Crystal data and structure refinement for 2u.

Identification code	2u
Empirical formula	$C_{22}H_{19}NOS$
Formula weight	345.44
Temperature/K	293(2)
Crystal system	triclinic
Space group	P-1
a/Å	9.6100(5)
b/Å	9.7305(5)
c/Å	10.0325(5)
α/°	74.741(5)
β/°	85.698(4)
γ/°	81.494(4)
Volume/Å^3	894.47(8)
Z	2
ρ_{calc}g/cm^3	1.283
μ/mm^{-1}	1.663
F(000)	364.0
Crystal size/mm^3	0.3 × 0.3 × 0.2
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.312 to 133.186
Index ranges	-10 ≤ h ≤ 11, -10 ≤ k ≤ 11, -10 ≤ l ≤ 11
Reflections collected	5274
Independent reflections	3160 [R_{int} = 0.0164, R_{sigma} = 0.0239]
Data/restraints/parameters	3160/0/226
Goodness-of-fit on F^2	1.041
Final R indexes [I>=2σ (I)]	R_1 = 0.0407, wR_2 = 0.1092

Final R indexes [all data]	$R_1 = 0.0441$, $wR_2 = 0.1139$
Largest diff. peak/hole / e Å^{-3}	0.18/-0.22

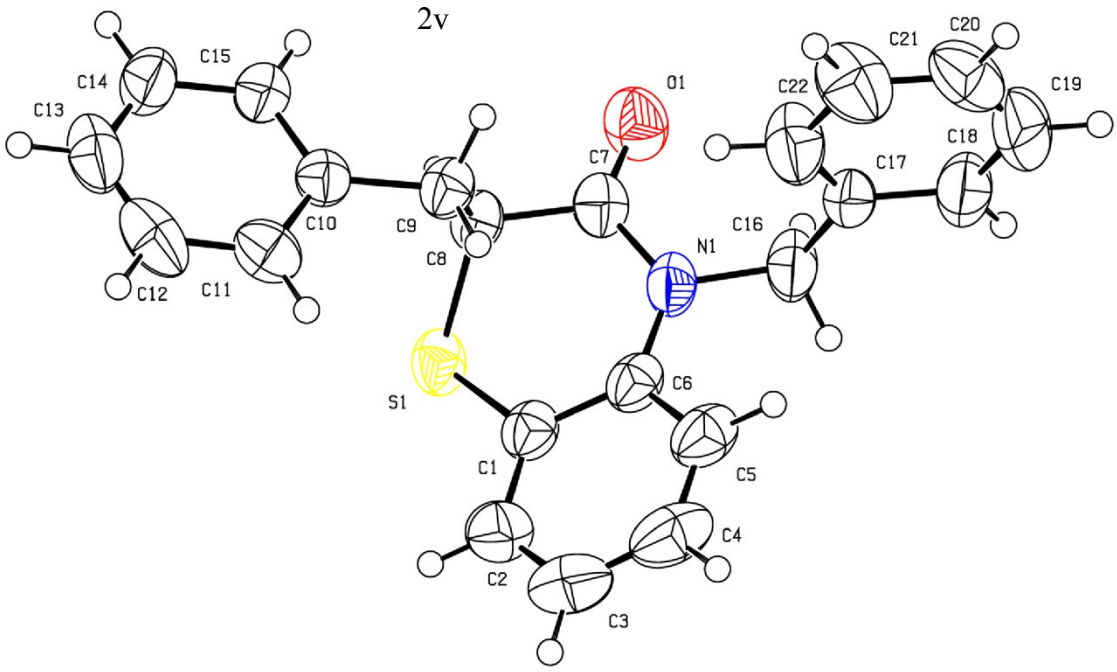


Table 2 Crystal data and structure refinement for 3t.

Identification code	3t
Empirical formula	$C_{15}H_{11}NOS$
Formula weight	253.31
Temperature/K	293
Crystal system	orthorhombic
Space group	Pbca
a/Å	14.55837(15)
b/Å	6.82995(9)
c/Å	25.1099(2)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	2496.75(5)
Z	8
ρ_{calc}g/cm^3	1.348
μ/mm^{-1}	2.180
F(000)	1056.0
Crystal size/mm^3	0.4 × 0.3 × 0.05
Radiation	CuKα (λ = 1.54184)

2Θ range for data collection/°	9.302 to 133.136
Index ranges	$-17 \leq h \leq 17$, $-7 \leq k \leq 8$, $-29 \leq l \leq 29$
Reflections collected	30991
Independent reflections	2202 [R_{int} = 0.0429, R_{sigma} = 0.0155]
Data/restraints/parameters	2202/0/163
Goodness-of-fit on F^2	1.061
Final R indexes [I>=2σ (I)]	R_1 = 0.0344, wR_2 = 0.0903
Final R indexes [all data]	R_1 = 0.0356, wR_2 = 0.0913
Largest diff. peak/hole / e $Å^{-3}$	0.15/-0.23

3t