Electronic Supplementary Material (ESI) for RSC Advances

Copper-Mediated Synthesis of N-fused Heterocycles via Csp-S Coupling Reaction and 5-endo-dig Cyclization Sequence

(Supporting Information)

Dongmei Xiao[†], Liqiang Han[†], Qi Sun[†,], Qianxi Chen[§], Ningbo Gong[§], Yang Lv[§], Franck Suzenet[‡], Gerald Guillaumet[‡], Tieming Cheng[†], Runtao Li[†,*]*

[†] State key Laboratory of Natural and Biomimetic Drugs, School of Pharmaceutical Sciences, Peking University, 38 Xueyuan Road, Beijing, 100191, China

[§] Institute of Materia Medica (IMM), Chinese Academy of Medical Sciences & Peking Union Medical College

[‡] Institut de Chimie Organique et Analytique, Université d'Orléans, UMR-CNRS 6005, rue de Chartres, BP 6759, 45067 Orléans cedex 2, France

E-mail: sunqi@bjmu.edu.cn.

Telphone: (+86)10-82801504

CONTENTS:

1. General Information

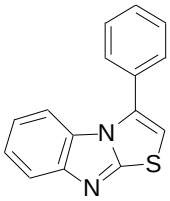
All manipulations were conducted with a standard Schlenk technique under air atmosphere. ^{1}H-NMR spectra were recorded with a Bruker AVIII-400 spectrometer. Chemical shifts (in ppm) were referenced to tetramethylsilane (δ = 0 ppm) in $CDCl_3$ (δ = 7.26 ppm) or d_6-DMSO (δ = 2.50 ppm) as an internal standard. ^{13}C-NMR spectra were obtained by the same NMR spectrometer and were calibrated with $CDCl_3$ (δ = 77.00 ppm) or d_6-DMSO (δ = 39.50 ppm). High-resolution mass spectra were recorded by APEX IV Fourier Transform Ion Cyclotron Resonance Mass Spectrometer spectrometer in ESI. Silica gel (200-300 mesh) was used for column chromatography. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. All solvents were used as received. All reactions were carried out without taking precautions to exclude air and moisture. Dihydropyrimidine-2-thiones **4a**-**4n** were synthesized by the well-known Bignelli's one-pot condensation reaction[1]. ICy•HCl was synthesized according to the literature[2].

2. Experimental procedure and characterization of products

A General procedure for Csp-S coupling reaction and 5-endo-dig cyclization.

To a 25 mL flask was added toluene (10 mL), Et_3N (0.71 mL, 5mmol), CuCl (198mg, 2mmol), ICy•HCl (537mg, 2mmol) sequentially. The formed mixture was stirred at room temperature for 2 hours. After 2 hours, compound **3** (1mmol), compound **8a** (2mmol) were added. Then the reaction mixture was stirred at 110 ℃ for 24 hours as monitored by TLC. For work-up, after cooling down to the room temperature and concentrating in *vacuo*, the residue was purified by flash chromatograph on silica gel (eluent: petroleum ether / ethyl acetate = 4:1) to afford the product.

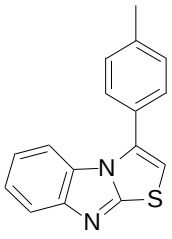
3-phenylbenzo[d]thiazolo[3,2-a]imidazole (1a)



1a

Obtained as a white solid in 74% yield, mp 138~140 ℃(Lit. 138-140℃[3]). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.2 Hz, 1H), 7.64 (dd, *J* = 7.1, 2.5 Hz, 2H), 7.59 – 7.50 (m, 3H), 7.35 – 7.28 (m, 1H), 7.22 (d, *J* = 8.2 Hz, 1H), 7.08 – 7.02 (m, 1H), 6.57 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.23, 148.77, 134.25, 130.11, 129.46, 128.90, 123.34, 120.39, 119.25, 111.67, 107.11; HRMS *m/z* (ESI) calcd for $C_{15}H_{11}N_2S$ $(M + H)^+$ 251.06375, found 251.06365.

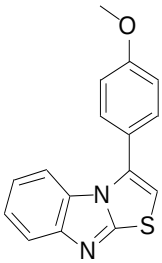
3-p-tolylbenzo[d]thiazolo[3,2-a]imidazole (1b)



1b

Obtained as a white solid in 61% yield, mp 117~118 ℃(Lit. 138-140℃[3]). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.2 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.36 (d, *J* = 7.9 Hz, 2H), 7.34 – 7.28 (m, 1H), 7.27 – 7.22 (m, 1H), 7.08 – 7.02 (m, 1H), 6.53 (s, 1H), 2.48 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.41, 147.93, 139.43, 133.50, 129.30, 128.78, 127.87, 125.66, 122.42, 119.45, 118.33, 110.89, 105.76, 20.63; HRMS *m/z* (ESI) calcd for $C_{16}H_{13}N_2S$ $(M + H)^+$ 265.07940, found 265.07901.

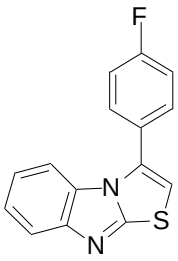
3-(4-methoxyphenyl)benzo[d]thiazolo[3,2-a]imidazole (1c)



1c

Obtained as a white solid in 42% yield, mp 148~150 ℃(Lit. 148-150℃[3]). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.2 Hz, 1H), 7.57 (d, *J* = 8.7 Hz, 2H), 7.31 (t, *J* = 7.7 Hz, 1H), 7.27 – 7.23 (m, 1H), 7.06 (t, *J* = 8.8 Hz, 3H), 6.51 (s, 1H), 3.92 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.99, 157.22, 148.80, 134.09, 130.31, 123.27, 121.63, 120.32, 119.20, 114.37, 111.64, 106.29, 55.48, 1.02; HRMS *m/z* (ESI) calcd for $C_{16}H_{13}N_2OS$ $(M + H)^+$ 281.07431, found 281.07361.

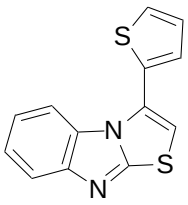
3-(4-fluorophenyl)benzo[d]thiazolo[3,2-a]imidazole (1d)



1d

Obtained as a white solid in 44% yield, mp 145~147 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.2 Hz, 1H), 7.68 – 7.61 (m, 2H), 7.33 (t, *J* = 7.7 Hz, 1H), 7.30 – 7.24 (m, 2H), 7.17 (d, *J* = 8.2 Hz, 1H), 7.07 (t, *J* = 7.7 Hz, 1H), 6.58 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.00, 157.08, 148.78, 133.10, 130.89, 130.01, 125.53, 123.45, 120.52, 119.37, 116.35, 116.13, 111.38, 107.38, 1.02; HRMS *m/z* (ESI) calcd for $C_{15}H_{10}FN_2S$ $(M + H)^+$ 269.05432, found 269.05427. Anal. calcd for $C_{15}H_9FN_2S$: C 67.15, H 3.38, N 10.44; found C 67.12, H 3.38 N 10.46.

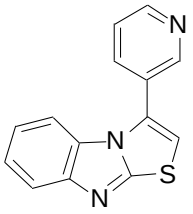
3-(thiophen-2-yl)benzo[d]thiazolo[3,2-a]imidazole (1e)



1e

Obtained as a white solid in 63% yield, mp 93~95 ℃(Lit. 94-95℃[3]). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.2 Hz, 1H), 7.56 (dd, *J* = 5.2, 1.1 Hz, 1H), 7.45 (dd, *J* = 3.6, 1.1 Hz, 1H), 7.34 (dt, *J* = 8.3, 4.7 Hz, 2H), 7.25 (dd, *J* = 3.1, 2.0 Hz, 1H), 7.14 – 7.08 (m, 1H), 6.72 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.52, 148.65, 130.07, 129.56, 129.06, 128.11, 127.72, 127.02, 123.49, 120.62, 119.30, 111.50, 109.42; HRMS *m/z* (ESI) calcd for $C_{13}H_9N_2S_2$ $(M + H)^+$ 257.02017, found 257.01989.

3-(pyridin-3-yl)benzo[d]thiazolo[3,2-a]imidazole (1f)

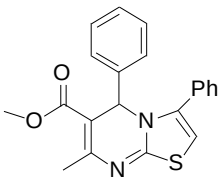


1f

Obtained as a yellow solid in 41% yield, mp 137~139 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.92 (d, *J* = 37.1 Hz, 2H), 7.99 (d, *J* = 7.7 Hz, 1H), 7.79 (s, 1H), 7.54 (s, 1H), 7.31 (d, *J* = 7.2 Hz, 1H), 7.18 (d, *J* = 7.7 Hz, 1H), 7.07 (t, *J* = 7.6 Hz, 1H), 6.71 (s, 1H); 13C NMR (101 MHz, CDCl3) δ 156.95, 151.15, 149.27, 148.64, 136.00, 130.77, 129.84, 125.69, 123.54(d), 120.73, 119.41, 111.18, 109.00; HRMS *m/z* (ESI) calcd for $C_{14}H_{10}N_3S$ $(M + H)^+$ 252.05899, found 252.05851;

Anal. calcd for $C_{14}H_9N_3S$: C 66.91, H 3.61, N 16.72; found C 66.92, H 3.66, N 16.77.

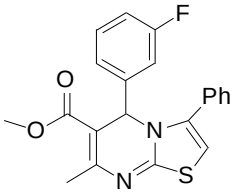
Methyl 7-methyl-3,5-diphenyl-5H-thiazolo[3,2-a]pyrimidine-6-carboxylate (2a)



2a

Obtained as a yellow solid in 75% yield, mp 166~167 ℃[4]. ^{1}H NMR (400 MHz, DMSO) δ 7.58 (d, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.5 Hz, 2H), 7.43 (s, 1H), 7.29 (d, *J* = 7.2 Hz, 2H), 7.22 (t, *J* = 7.3 Hz, 1H), 7.14 (t, *J* = 7.5 Hz, 2H), 6.65 (d, *J* = 7.3 Hz, 2H), 6.36 (s, 1H), 3.59 (s, 3H), 2.48 (s, 3H); ^{13}C NMR (101 MHz, DMSO) δ 165.03, 142.89, 140.32, 139.63, 131.01, 130.04, 129.73–128.93 (m), 127.68, 126.87, 111.93, 103.16, 59.30, 52.15, 18.17; HRMS *m/z* (ESI) calcd for $C_{21}H_{19}N_2O_2S$ $(M + H)^+$ 363.11617, found 363.11528.

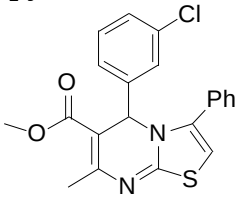
Methyl 5-(3-fluorophenyl)-7-methyl-3-phenyl-5H-thiazolo[3,2-a] pyrimidine-6-carboxylate (2b)



2b

Obtained as a yellow solid in 98% yield, mp 142~144 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (dt, *J* = 14.7, 7.2 Hz, 3H), 7.13 (d, *J* = 7.4 Hz, 2H), 7.03 (d, *J* = 6.5 Hz, 1H), 6.82 (d, *J* = 8.2 Hz, 1H), 6.58 (d, *J* = 7.6 Hz, 1H), 6.45 (d, *J* = 9.6 Hz, 1H), 6.18 (d, *J* = 12.1 Hz, 2H), 3.62 (s, 3H), 2.45 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.06, 166.56, 163.69, 161.24, 156.43, 144.48, 139.63, 129.77, 129.39, 128.85, 122.04, 115.07, 113.57, 102.93, 100.45, 57.43, 50.97, 23.64; HRMS *m/z* (ESI) calcd for $C_{21}H_{18}FN_2O_2S$ $(M + H)^+$ 381.10675, found 381.10623. Anal. calcd for $C_{21}H_{17}FN_2O_2S$: C 66.30, H 4.50, N 7.36; found C 66.18, H 4.55, N 7.40.

Methyl 5-(3-chlorophenyl)-7-methyl-3-phenyl-5H-thiazolo[3,2-a] pyrimidine-6-carboxylate (2c)

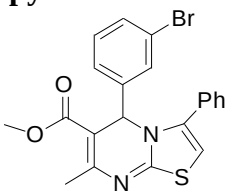


2c

Obtained as a yellow solid in 93% yield, mp 147~148℃[4]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (dt, *J* = 14.7, 7.2 Hz, 15H),

7.27 (s, 1H), 7.19 – 7.05 (m, 15H), 7.00 (t, *J* = 7.8 Hz, 5H), 6.75 – 6.59 (m, 10H), 6.19 (s, 5H), 6.12 (s, 5H), 3.62 (s, 14H), 2.45 (s, 15H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.03, 165.49, 155.49, 142.93, 138.60, 132.96, 128.80, 128.46, 127.86, 127.25, 125.74, 123.72, 101.96, 99.33, 56.55, 50.00, 22.69; HRMS *m/z* (ESI) calcd for $C_{21}H_{18}ClN_2O_2S$ $(M + H)^+$ 397.07720, found 397.07642.

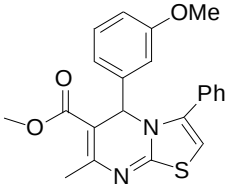
Methyl 5-(3-bromophenyl)-7-methyl-3-phenyl-5H-thiazolo[3,2-a] pyrimidine-6-carboxylate (2d)



2d

Obtained as a yellow solid in 88% yield, mp 158~160 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 17.2 Hz, 2H), 7.23 (s, 1H), 7.11 (s, 1H), 6.94 (s, 1H), 6.74 (s, 1H), 6.15 (d, *J* = 31.7 Hz, 2H), 3.61 (s, 2H), 2.45 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.99, 166.44, 156.49, 144.16, 139.57, 131.18, 130.03 – 129.43 (m), 129.36, 128.87, 125.23, 122.13, 102.98, 100.27, 57.55, 51.01, 23.68; HRMS *m/z* (ESI) calcd for $C_{21}H_{18}BrN_2O_2S$ $(M + H)^+$ 441.02669, found 441.02566. Anal. calcd for $C_{21}H_{17}BrN_2O_2S$: C 57.15, H 3.88, N 6.35; found C 57.01, H 3.90, N 6.30.

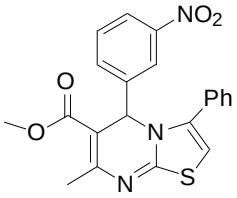
Methyl 5-(3-methoxyphenyl)-7-methyl-3-phenyl-5H-thiazolo[3,2-a] pyrimidine-6-carboxylate (2e)



2e

Obtained as a yellow solid in 89% yield, mp 142~144 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.36 (m, 3H), 7.14 (d, *J* = 6.4 Hz, 2H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.65 (d, *J* = 7.2 Hz, 1H), 6.44 (d, *J* = 6.7 Hz, 1H), 6.27 (s, 1H), 6.15 (s, 2H), 3.62 (d, *J* = 7.5 Hz, 3H), 3.58 (d, *J* = 8.0 Hz, 3H), 2.45 (d, *J* = 4.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.17, 166.60, 159.28, 156.17, 143.56, 139.74, 129.60, 129.28 (s, 3H), 128.78, 118.76, 113.91, 111.61, 102.76, 100.76, 57.80, 54.95, 50.90, 23.60; HRMS *m/z* (ESI) calcd for $C_{22}H_{21}N_2O_3S$ $(M + H)^+$ 393.12674, found 393.12625. Anal. calcd for $C_{22}H_{20}N_2O_3S$: C 67.33, H 5.14, N 7.14; found C 67.13, H 5.21, N 7.08.

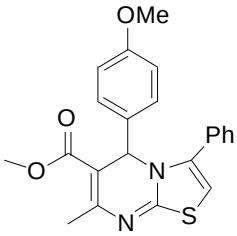
Methyl 7-methyl-5-(3-nitrophenyl)-3-phenyl-5H-thiazolo[3,2-a] pyrimidine-6-carboxylate (2f)



2f

Obtained as a yellow solid in 72% yield, mp 147~149 ℃[4]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 7.4 Hz, 1H), 7.50 (d, *J* = 7.0 Hz, 1H), 7.42 (dd, *J* = 14.5, 6.6 Hz, 3H), 7.32 – 7.22 (m, 2H), 7.11 (d, *J* = 7.1 Hz, 2H), 6.28 (s, 1H), 6.25 (s, 1H), 3.62 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.89, 166.49, 156.98, 147.80, 143.99, 139.40, 132.82, 130.13, 129.48, 128.96, 123.04, 121.53, 103.36, 99.99, 57.42, 51.15, 23.74; HRMS *m/z* (ESI) calcd for $C_{21}H_{18}N_3O_4S$ $(M + H)^+$ 408.09885, found 408.09986.

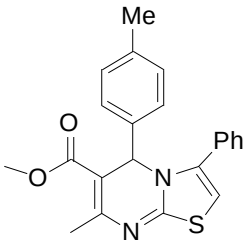
Methyl 5-(4-methoxyphenyl)-7-methyl-3-phenyl-5H-thiazolo[3,2-a] pyrimidine-6-carboxylate (2g)



2g

Obtained as a yellow solid in 68% yield, mp 134~136 ℃[4]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (dt, *J* = 14.7, 7.2 Hz, 3H), 7.14 (d, *J* = 7.5 Hz, 1H), 6.68 (d, *J* = 8.5 Hz, 1H), 6.57 (d, *J* = 8.4 Hz, 1H), 6.12 (d, *J* = 14.8 Hz, 1H), 3.69 (s, 1H), 3.60 (s, 1H), 2.45 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.23, 166.45, 159.30, 155.81, 139.82, 134.71, 129.75, 129.52, 128.88, 128.65, 127.78, 113.50, 102.61, 101.18, 57.38, 55.08, 50.84, 23.56; HRMS *m/z* (ESI) calcd for $C_{22}H_{21}N_2O_3S$ $(M + H)^+$ 393.12674, found 393.12655.

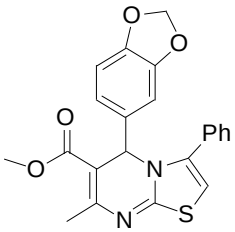
Methyl 7-methyl-3-phenyl-5-p-tolyl-5H-thiazolo[3,2-a]pyrimidine-6-carboxylate (2h)



2h

Obtained as a yellow solid in 82% yield, mp 180~182 ℃[4]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (dt, *J* = 22.4, 7.2 Hz, 2H), 7.12 (d, *J* = 7.0 Hz, 1H), 6.85 (d, *J* = 7.9 Hz, 1H), 6.66 (d, *J* = 8.0 Hz, 2H), 6.12 (d, *J* = 4.3 Hz, 2H), 3.59 (s, 3H), 2.44 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.22, 166.62, 156.02, 139.80, 139.34, 137.77, 129.62, 128.90, 128.67, 126.37, 102.70, 101.08, 57.62, 50.89, 23.63, 21.09; HRMS *m/z* (ESI) calcd for $C_{22}H_{21}N_2O_2S$ $(M + H)^+$ 377.13183, found 377.13124.

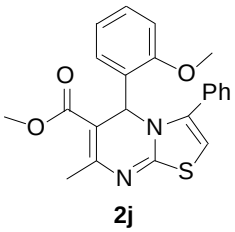
Methyl 5-(benzo[d][1,3]dioxol-5-yl)-7-methyl-3-phenyl-5H-thiazolo[3,2-a] pyrimidine-6-carboxylate (2i)



2i

Obtained as a yellow solid in 75% yield, mp 148~151 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (d, *J* = 7.0 Hz, 1H), 7.07 (d, *J* = 5.6 Hz, 1H), 6.33 (d, *J* = 7.4 Hz, 0H), 6.25 (s, 0H), 6.07 (s, 0H), 6.02 (d, *J* = 13.5 Hz, 1H), 5.70 (s, 2H), 3.50 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.10, 166.36, 155.90, 147.50, 147.29, 139.66, 136.17, 129.60, 128.72, 119.98, 107.59, 107.00, 102.81, 100.93, 57.57, 50.86, 23.55; HRMS *m/z* (ESI) calcd for $C_{22}H_{19}N_2O_4S$ $(M + H)^+$ 407.10600, found 407.10498. Anal. calcd for $C_{22}H_{18}N_2O_4S$: C 65.01, H 4.46, N 6.89; found C 64.80, H 4.62, N 6.83.

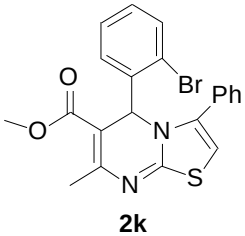
Methyl 5-(2-methoxyphenyl)-7-methyl-3-phenyl-5H-thiazolo[3,2-a] pyrimidine-6-carboxylate (2j)



2j

Obtained as a yellow solid in 67% yield, mp 143~146 ℃[4]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (dt, *J* = 14.0, 6.9 Hz, 1H), 7.06 (d, *J* = 6.1 Hz, 1H), 6.82 (d, *J* = 7.2 Hz, 0H), 6.59 (d, *J* = 5.6 Hz, 1H), 6.47 (s, 0H), 6.01 (s, 0H), 3.54 (s, 3H), 3.38 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.47, 167.11, 156.48, 155.85, 140.20, 130.27, 129.67, 129.22, 128.82, 128.38, 120.06, 110.32, 102.00, 100.20, 55.06, 54.38, 50.72, 23.65; HRMS *m/z* (ESI) calcd for $C_{22}H_{21}N_2O_3S$ $(M + H)^+$ 393.12674, found 393.12587.

Methyl 5-(2-bromophenyl)-7-methyl-3-phenyl-5H-thiazolo[3,2-a] pyrimidine-6-carboxylate (2k)

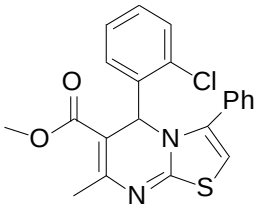


2k

Obtained as a yellow solid in 66% yield, mp 145~148 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (dd, *J* = 9.9, 4.2 Hz, 2H), 7.36 (t, *J* = 7.3 Hz, 2H), 7.24 – 7.18 (m, 1H), 7.08 (t, *J* = 7.5 Hz, 3H), 6.98 – 6.89 (m, 1H), 6.50 (s, 1H), 6.06 (s, 1H), 3.56 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.85, 156.02, 141.96, 139.77, 132.71, 129.59, 131.37 – 128.25, 129.93 – 128.25 (m) , 129.33 – 127.74

(m), 121.38, 102.72, 101.03, 57.44, 50.61, 23.43; HRMS *m/z* (ESI) calcd for $C_{21}H_{18}BrN_2O_2S$ $(M + H)^+$ 441.02669, found 441.02654. Anal. calcd for $C_{21}H_{17}BrN_2O_2S$: C 57.15, H 3.88, N 6.35; found C 56.97, H 3.93, N 6.33.

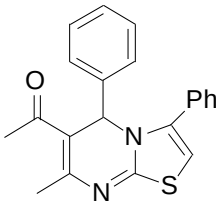
Methyl 5-(2-chlorophenyl)-7-methyl-3-phenyl-5H-thiazolo[3,2-a] pyrimidine-6-carboxylate (2l)



2l

Obtained as a yellow solid in 67% yield, mp 151~153 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 7.4 Hz, 1H), 7.38 – 7.31 (m, 3H), 7.07 (d, *J* = 7.1 Hz, 2H), 7.05 – 6.99 (m, 3H), 6.53 (s, 1H), 6.07 (s, 1H), 3.56 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.98, 156.16, 140.22, 139.88, 131.83, 130.30, 129.72, 129.50, 129.39 – 129.00 (m), 128.80, 127.21, 102.67, 100.69, 55.49, 50.79, 23.56; HRMS *m/z* (ESI) calcd for $C_{21}H_{18}ClN_2O_2S$ $(M + H)^+$ 397.07720, found 397.07633. Anal. calcd for $C_{21}H_{17}ClN_2O_2S$: C 63.55, H 4.32, N 7.06; found C 63.62, H 4.33, N 7.07.

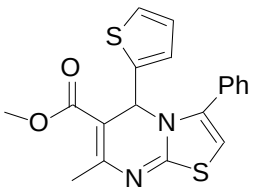
1-(7-methyl-3,5-diphenyl-5H-thiazolo[3,2-a]pyrimidin-6-yl)ethanone (2m)



2m

Obtained as a yellow solid in 83% yield, mp 140~142 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (t, *J* = 7.3 Hz, 0H), 7.39 (t, *J* = 7.3 Hz, 1H), 7.15 – 7.03 (m, 2H), 6.80 (d, *J* = 6.9 Hz, 2H), 6.34 (s, 1H), 6.22 (s, 1H), 2.45 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.58, 165.64, 154.61, 140.95, 139.33, 128.71, 128.38, 127.82, 127.22 (s, 6H), 126.90, 125.43, 111.38, 102.15, 56.36, 30.43, 24.08; HRMS *m/z* (ESI) calcd for $C_{21}H_{19}N_2OS$ $(M + H)^+$ 347.12126, found 347.12099.

Methyl 7-methyl-3-phenyl-5-(thiophen-2-yl)-5H-thiazolo[3,2-a]pyrimidine-6-carboxylate (2n)

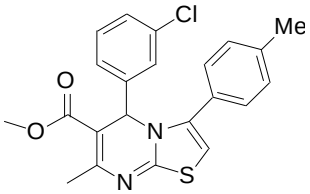


2n

Obtained as a yellow solid in 63% yield, mp 162~163 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.43 (m, 3H), 7.28 (dd, *J* = 7.3, 1.8 Hz, 2H), 7.04 (d, *J* = 5.0 Hz, 1H), 6.70 (dd, *J* =

4.9, 3.7 Hz, 1H), 6.50 (s, 1H), 6.35 (d, *J* = 3.2 Hz, 1H), 6.23 (d, *J* = 7.5 Hz, 1H), 3.67 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.93, 166.37, 156.67, 144.52, 139.37, 129.74, 129.31, 128.97, 128.66, 126.39, 125.45, 124.55, 103.25, 100.76, 52.61, 51.14, 23.43; HRMS *m/z* (ESI) calcd for $C_{19}H_{17}N_2O_2S_2$ $(M + H)^+$ 369.07260, found 369.07214. Anal. calcd for $C_{19}H_{16}N_2O_2S_2$: C 61.93, H 4.38, N 7.60; found C 61.76, H 4.37, N 6.34.

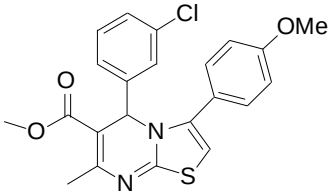
Methyl 5-(3-chlorophenyl)-7-methyl-3-p-tolyl-5H-thiazolo[3,2-a]pyrimidine -6-carboxylate (2o)



2o

Obtained as a yellow solid in 55% yield, mp 157~160 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 (d, *J* = 7.8 Hz, 2H), 7.10 (d, *J* = 8.0 Hz, 1H), 7.02 (t, *J* = 8.3 Hz, 3H), 6.73 (d, *J* = 7.6 Hz, 1H), 6.63 (s, 1H), 6.16 (s, 1H), 6.13 (s, 1H), 3.62 (s, 3H), 2.46 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.03, 166.50, 156.54, 143.98, 140.04, 139.70, 133.90, 129.51, 128.83, 128.17, 126.57, 124.68, 102.74, 102.46, 100.17, 57.58, 57.35, 50.89, 23.74, 21.30; HRMS *m/z* (ESI) calcd for $C_{22}H_{20}ClN_2O_2S$ $(M + H)^+$ 411.09285, found 411.09143. Anal. calcd for $C_{22}H_{19}ClN_2O_2S$: C 64.30, H 4.66, N 6.82; found C 64.21, H 4.71, N 6.77.

Methyl 5-(3-chlorophenyl)-3-(4-methoxyphenyl)-7-methyl-5H-thiazolo[3,2-a] pyrimidine-6-carboxylate (2p)

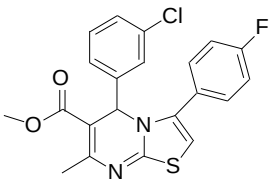


2p

Obtained as a yellow solid in 67% yield, mp 186~188 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 – 7.06 (m, 1H), 7.01 (t, *J* = 7.8 Hz, 3H), 6.92 (d, *J* = 8.7 Hz, 2H), 6.71 (d, *J* = 7.7 Hz, 1H), 6.65 (t, *J* = 1.7 Hz, 1H), 6.13 (s, 1H), 6.08 (s, 1H), 3.85 (s, 3H), 3.60 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.04 (s, 1H), 166.47 (s, 1H), 160.71 (s, 2H), 156.58 (s, 1H), 144.08 (s, 2H), 139.41 (s, 1H), 133.89 (s, 2H), 130.40 (s, 7H), 129.58 (s, 4H), 128.20 (s, 4H), 126.74 (s, 3H), 124.74 (s, 4H), 121.53 (s, 2H), 114.18 (s, 7H), 102.44 (s, 4H), 100.06 (s, 2H), 57.49 (s, 4H), 55.45 (s, 4H), 51.01 (s, 3H),

23.71 (s, 3H); HRMS *m/z* (ESI) calcd for $C_{22}H_{20}ClN_2O_3S$ $(M + H)^+$ 427.08777, found 427.08739. Anal. calcd for $C_{22}H_{19}ClN_2O_3S$: C 61.89, H 4.49, N 6.56; found C 62.10, H 4.58, N 6.47.

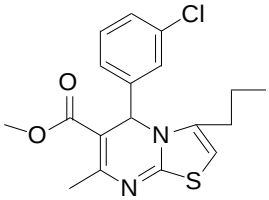
Methyl 5-(3-chlorophenyl)-3-(4-fluorophenyl)-7-methyl-5H-thiazolo[3,2-a] pyrimidine-6-carboxylate (2q)



2q

Obtained as a yellow solid in 76% yield, mp 187~189 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 – 7.02 (m, 5H), 6.98 (t, *J* = 8.0 Hz, 1H), 6.67 (s, 2H), 6.17 (s, 1H), 6.02 (s, 1H), 3.57 (s, 3H), 2.40 (s,3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.90, 166.30, 164.67, 162.17, 156.36, 143.89, 138.39, 134.04, 130.98, 129.63, 128.33 126.61, 125.41, 124.61, 116.07, 115.85, 103.40, 100.28, 57.60, 51.03, 23.66; HRMS *m/z* (ESI) calcd for $C_{21}H_{17}ClFN_2O_2S$ $(M + H)^+$ 415.06778, found 415.06765. Anal. calcd for $C_{21}H_{16}ClFN_2O_2S$: C 60.79, H 3.89, N 6.75; found C 60.89, H 3.89, N 6.79.

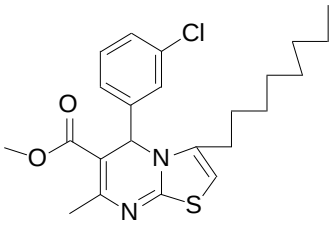
Methyl 3-butyl-5-(3-chlorophenyl)-7-methyl-5H-thiazolo[3,2-a]pyrimidine-6-carboxylate (2r)



2r

Obtained as a yellow solid in 67% yield, mp 110~112 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (d, *J* = 19.1 Hz, 3H), 7.18 (s, 1H), 6.19 (s, 1H), 5.99 (s, 1H), 3.74 (s, 3H), 2.38 (s, 3H), 1.89 (d, *J* = 56.1 Hz, 2H), 1.49 – 1.29 (m, 4H), 0.91 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.19, 156.79, 144.84 – 144.62 (m), 139.57, 134.89 – 134.50 (m), 130.09, 128.41, 126.30, 124.31, 106.90, 99.89, 57.02, 51.06, 32.79, 28.90, 27.06, 22.05, 13.69; HRMS *m/z* (ESI) calcd for $C_{19}H_{22}ClN_2O_2S$ $(M + H)^+$ 377.10850, found 377.10801. Anal. calcd for $C_{19}H_{21}ClN_2O_2S$: C 60.55, H 5.62, N7.43; found C 60.80, H 5.75, N 7.59.

Methyl 5-(3-chlorophenyl)-7-methyl-3-octyl-5H-thiazolo[3,2-a]pyrimidine-6 -carboxylate (2s)



2s

Obtained as a yellow oil in 68% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (s, 1H), 7.22 – 7.17 (m, 2H), 7.14 (dd, *J* = 4.1, 1.9 Hz, 1H), 6.17 (s, 1H), 5.96 (s, 1H), 3.70 (s, 3H), 2.35 (s, 3H), 2.27 – 2.15 (m, 1H), 1.61 – 1.49 (m, 1H), 1.47 – 1.34 (m, 1H), 1.30 – 1.16 (m, 10H), 0.86 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.05, 156.73, 144.74, 139.58, 134.58, 130.07, 128.37, 126.26, 124.28, 99.92, 99.18, 56.99, 51.00, 31.74, 29.28 – 28.76, 27.30, 26.79, 23.93, 22.60, 14.09; HRMS *m/z* (ESI) calcd for $C_{23}H_{30}ClN_2O_2S$ $(M + H)^+$ 433.17110, found 433.17015. Anal. calcd for $C_{23}H_{29}ClN_2O_2S$: C 63.80, H 6.75, N 6.47; found C 63.86, H 6.75, N 6.31.

3. References

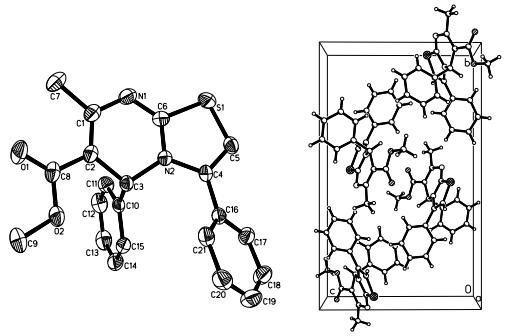
(1). Sun, Q.; Wang, Y. Q.; Ge, Z. M.; Cheng, T. M.; Li, R. T. *Synthesis* **2004**, *7*, 1047.

(2). Arduengo III, A. J.; Harlow, R. L.; Kline, M. *J. Am. Chem. Soc.* **1991**, *113*, 361.

(3) Xu, H.; Zhang, Y.; Huang, J.; Chen, W. *Org. Lett.* **2010**, *12*(16), 3704.

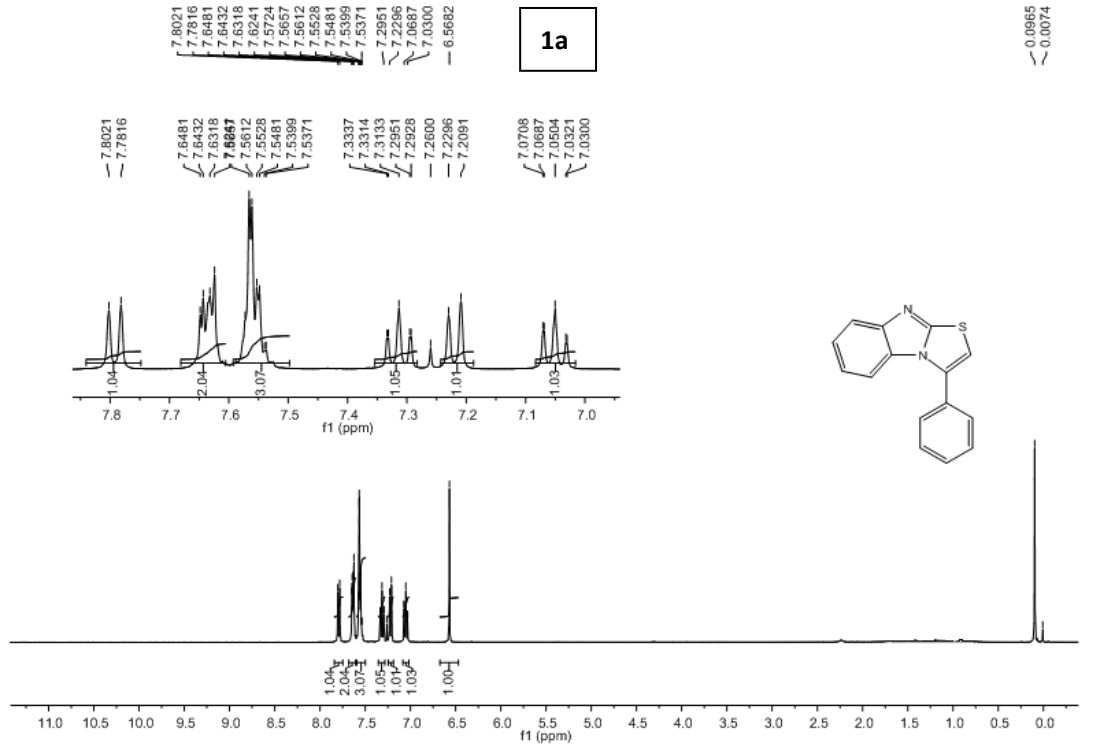
(4) Balkan, A.; Uma, S.; Ertan, M.; Wiegrebe, W. *Pharmazie*, **1992**, *47*(9), 687.

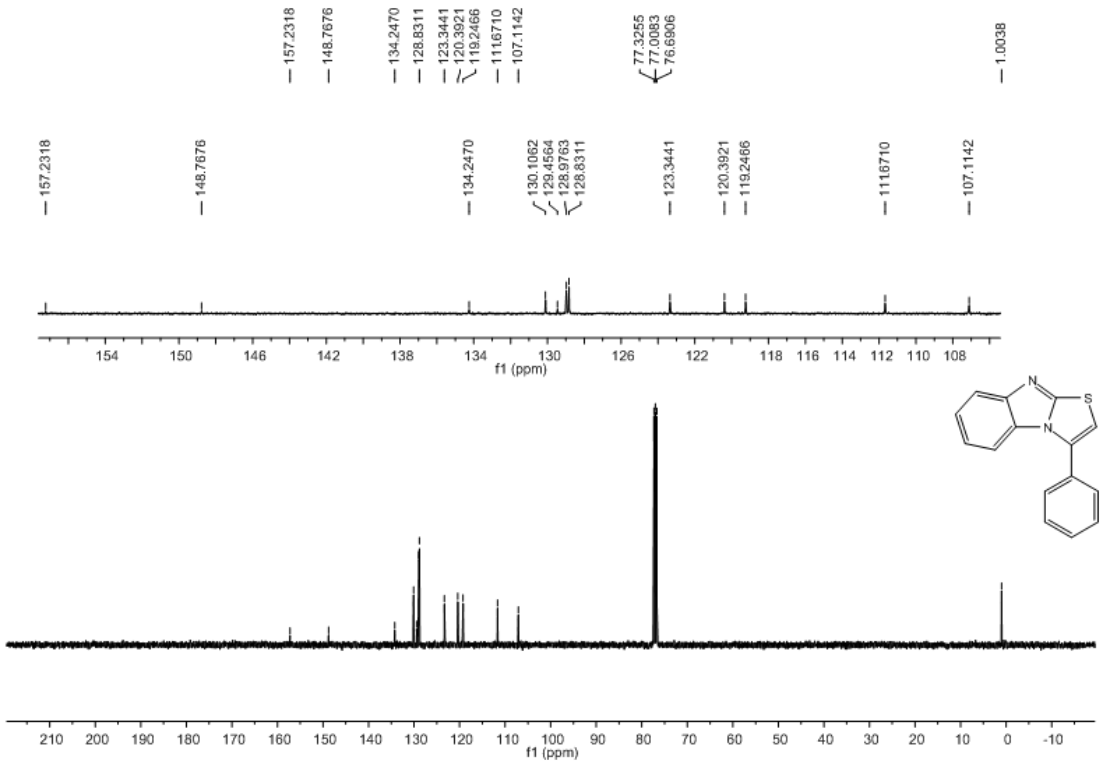
4. X-ray of compound 2a

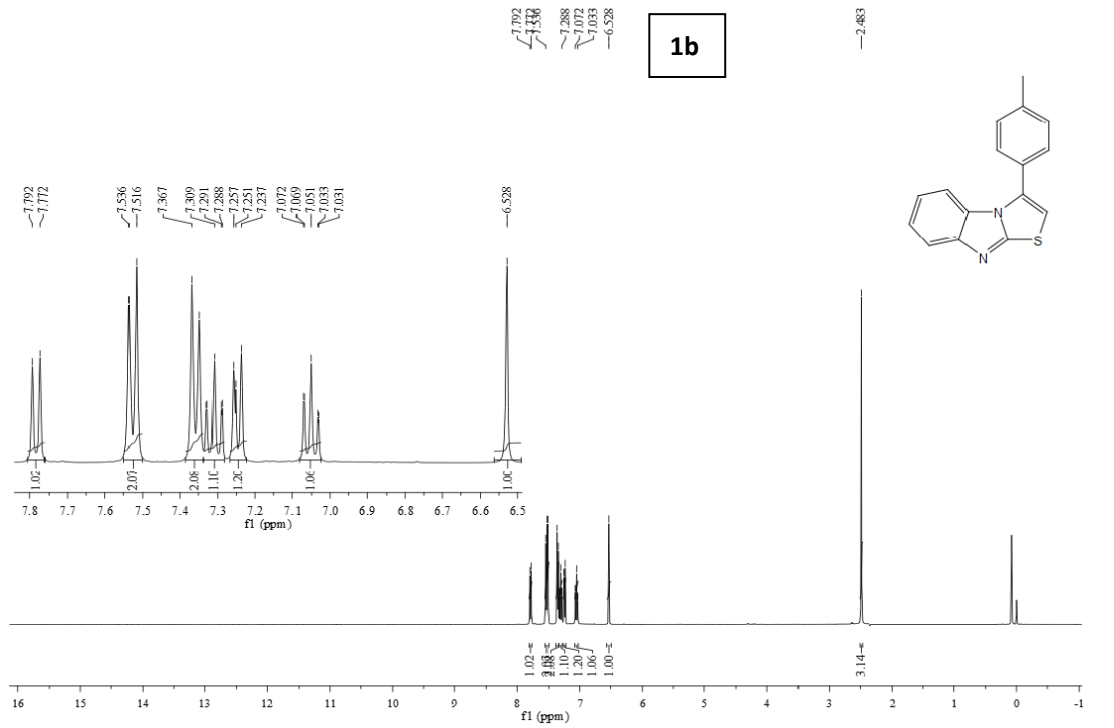


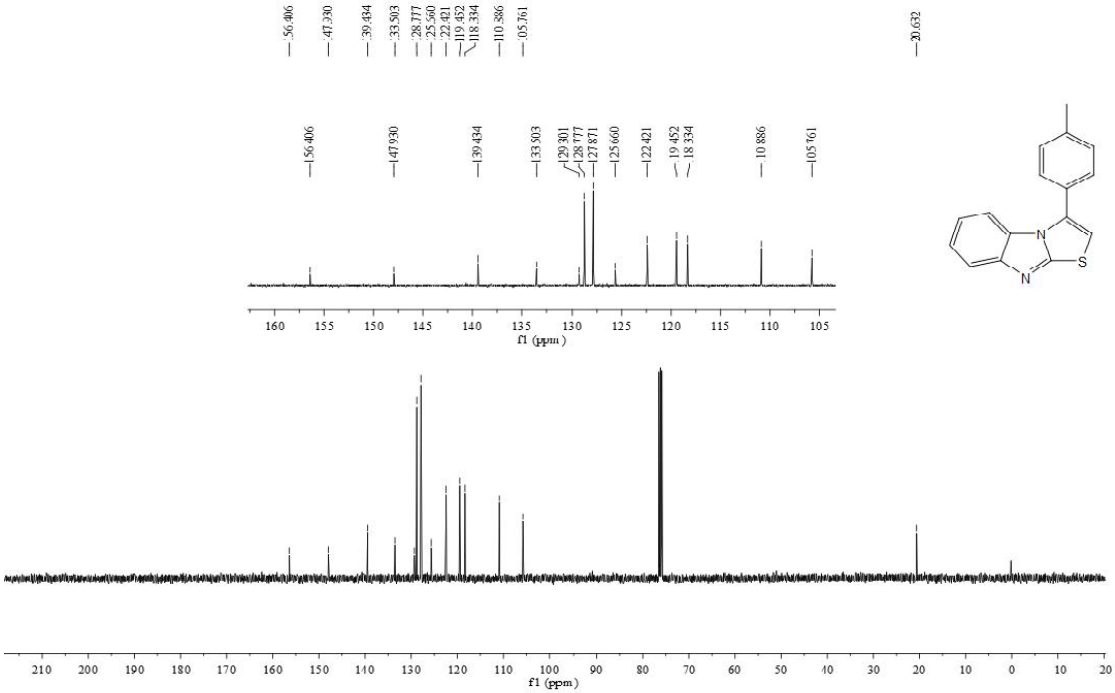
The analysis of single crystal X-ray diffraction of compound **2a,** $C_{21}H_{18}N_2O_2S$, M_r = 362.45, monoclinic, space group $P2_1/c$, a=7.917(5)，b=19.319(8)，c=11.750(5)Å，β=91.25(1)°，V = 1796.7(16) Å^3, Z = 4, D_c = 1.340g/cm^3. Intensity data were collected with *Rigaku MicroMax 002+* CCD diffractometer with a graphite monochromator (ω and κ scans, $2\theta_{max}$ =144.96°), *CuK* (λ= 1.54187Å) radiation. A total of 3521 unique reflections were collected, of which 2995 were observed ($|F|^2 \geq 2\sigma|F|^2$). The structure was solved by direct method and expanded by difference *Fourier* techniques with SHELX-97, refined on F^2 by successive full matrix least-squares techniques for non-H-atoms. H-Atoms were fixed at calculated positions. The final indices were R_1 = 0.0482, wR_2 = 0.1322, S = 1.104.

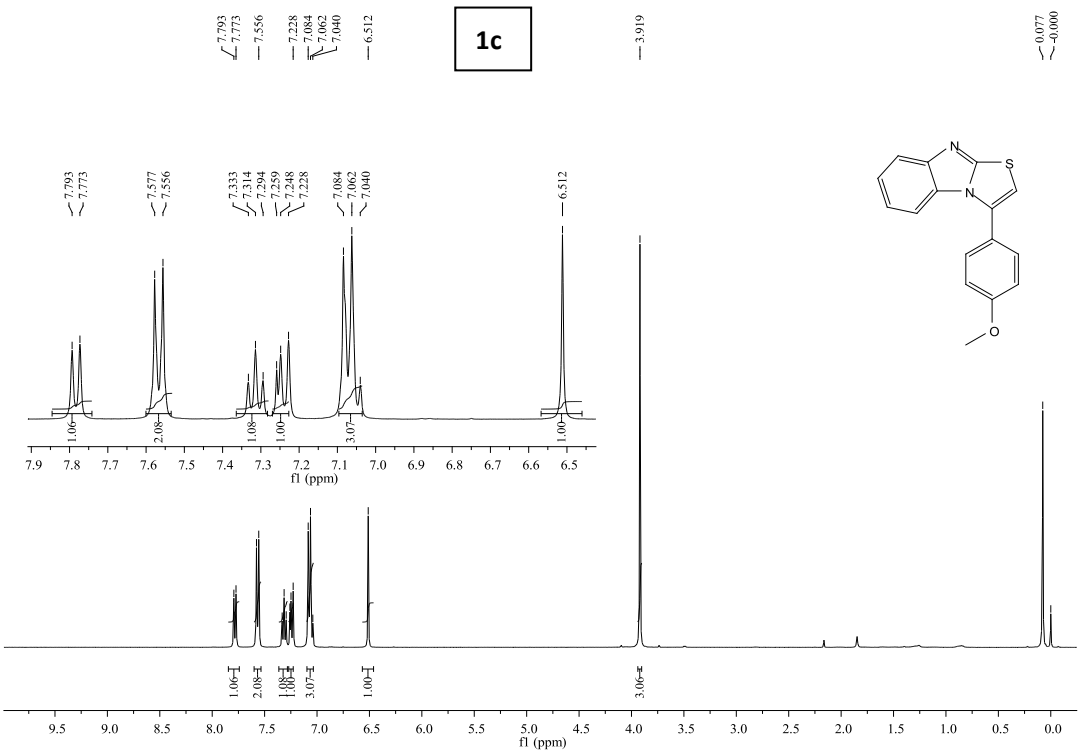
5. ^{1}H NMR and ^{13}C NMR spectra of those compounds

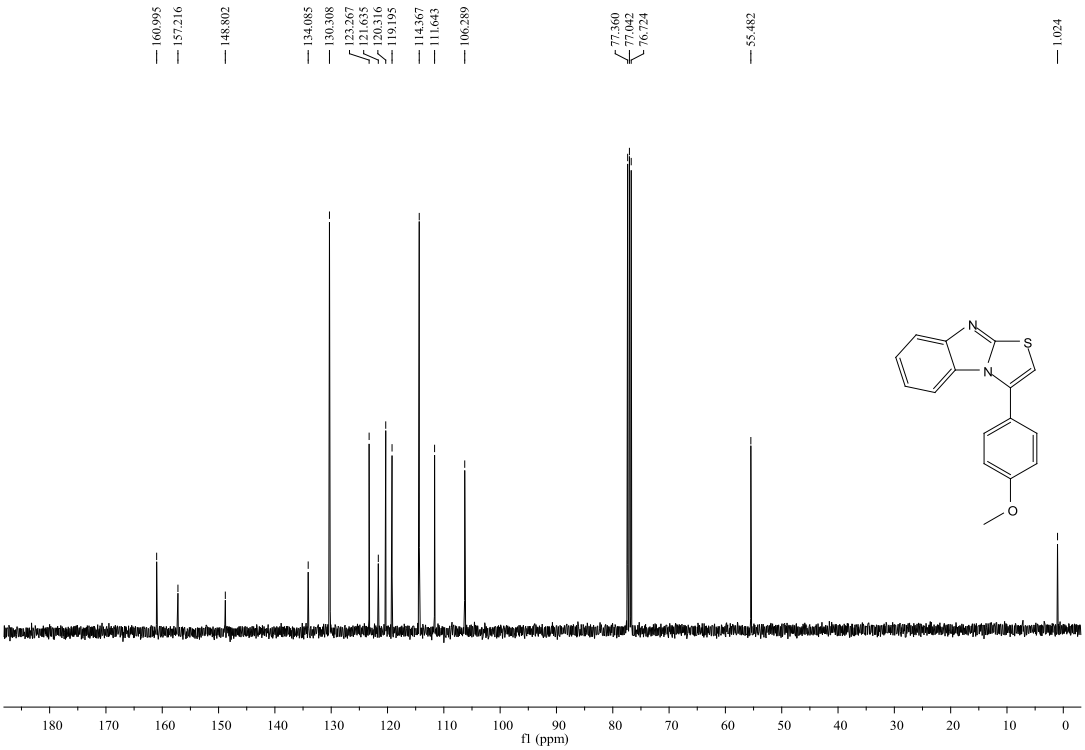


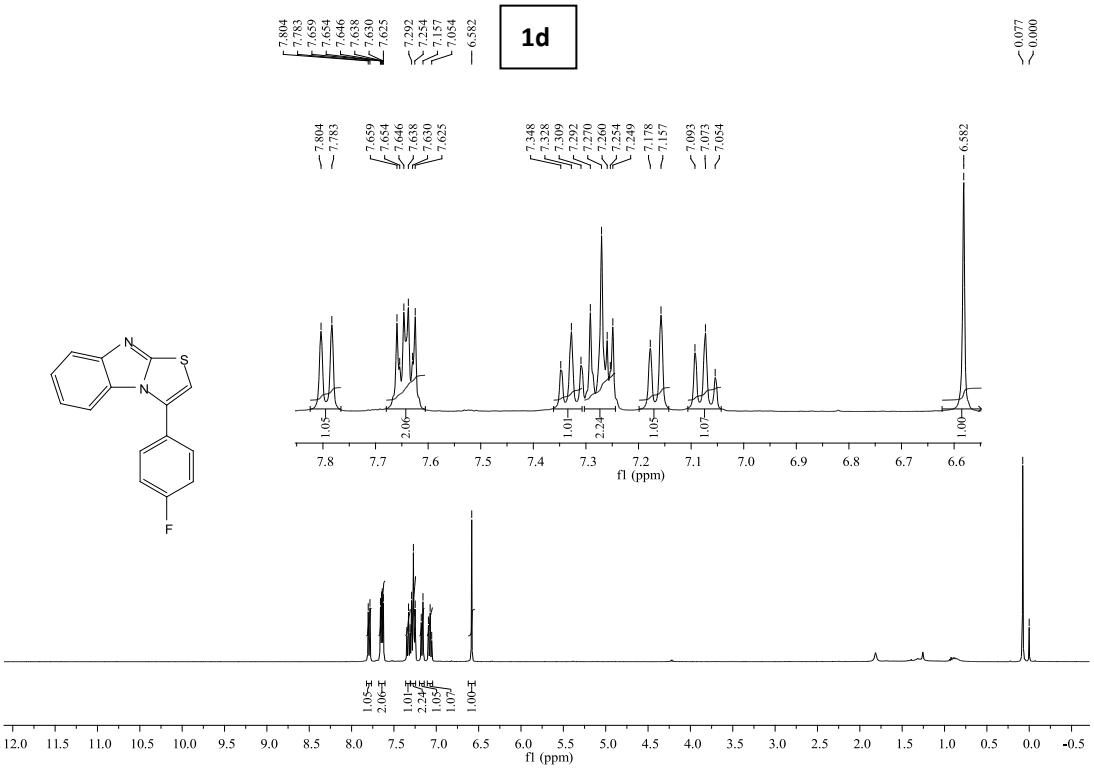


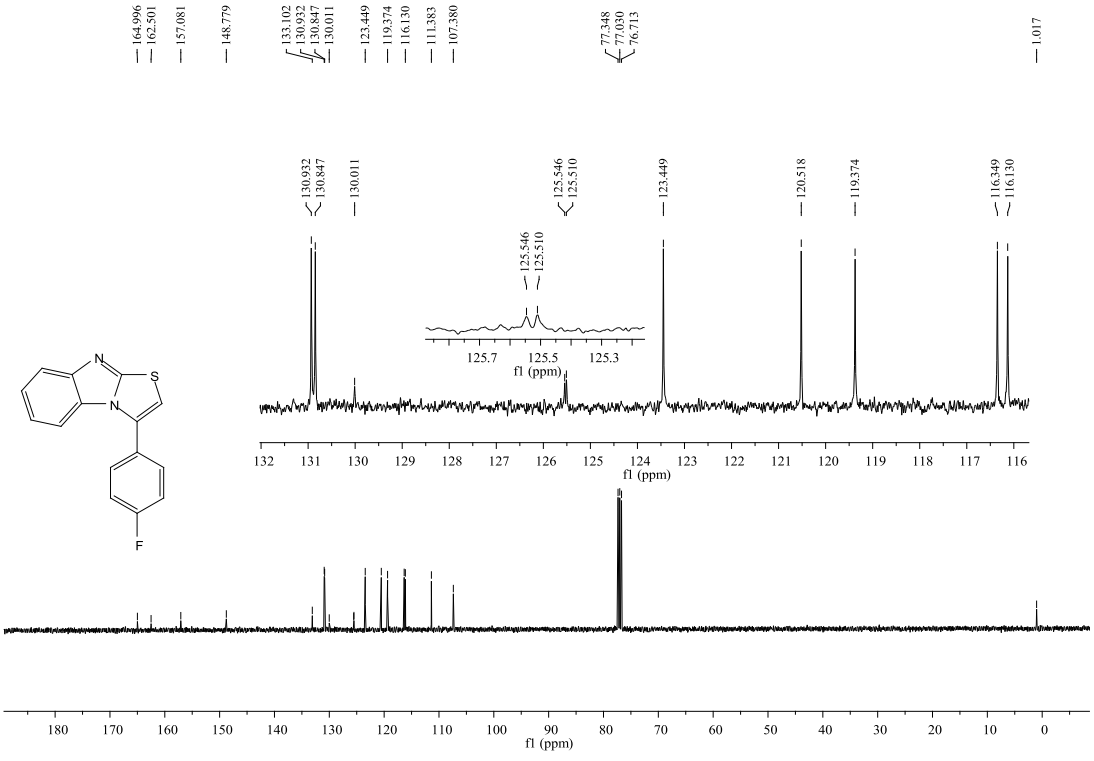

1b
f1 (ppm)

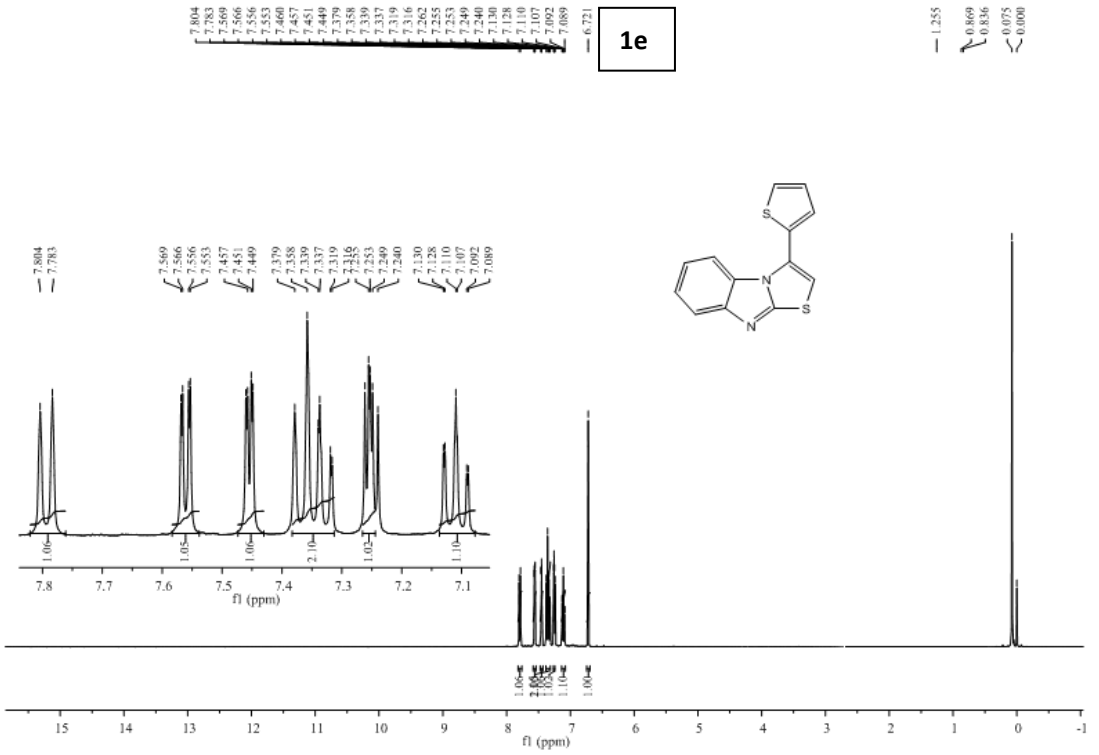

f1 (ppm)

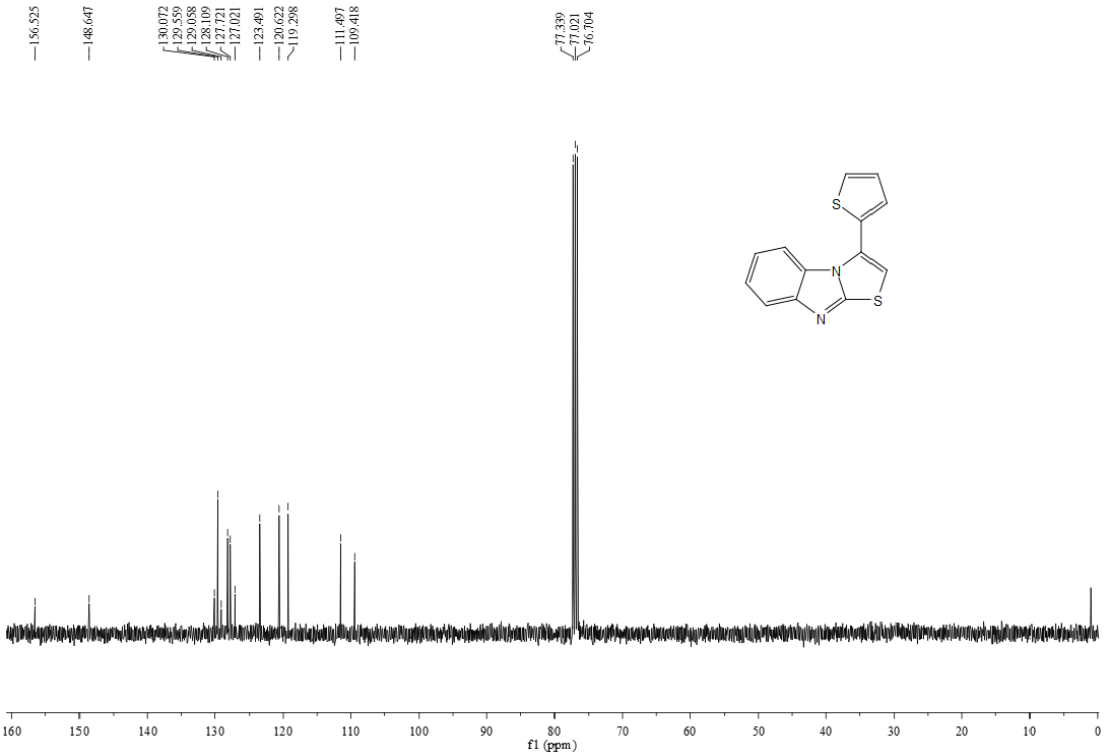

1c
7.793
7.773
7.556
7.228
7.084
7.062
7.040
6.512
3.919
0.077
-0.000
7.577
7.333
7.314
7.294
7.259
7.248
1.06
2.08
1.08
1.00
3.07
1.00
3.06
fl (ppm)


160.995
157.216
148.802
134.085
130.308
123.267
121.635
120.316
119.195
114.367
111.643
106.289
77.360
77.042
76.724
55.482
1.024
fl (ppm)

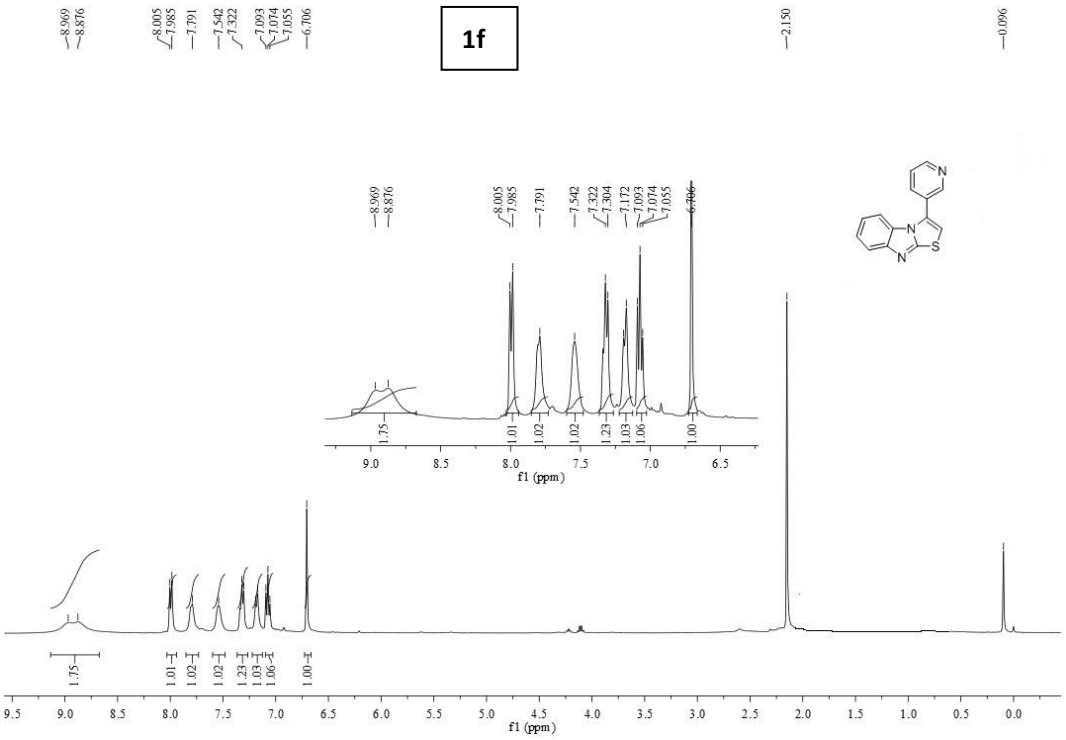

1d
7.804
7.783
7.659
7.654
7.646
7.638
7.630
7.625
7.292
7.254
7.157
7.054
6.582
0.077
0.000
7.348
7.328
7.309
7.292
7.270
7.260
7.254
7.249
7.178
7.157
7.093
7.073
7.054
1.05
2.06
1.01
2.24
1.05
1.07
1.00
fl (ppm)
N
S
F

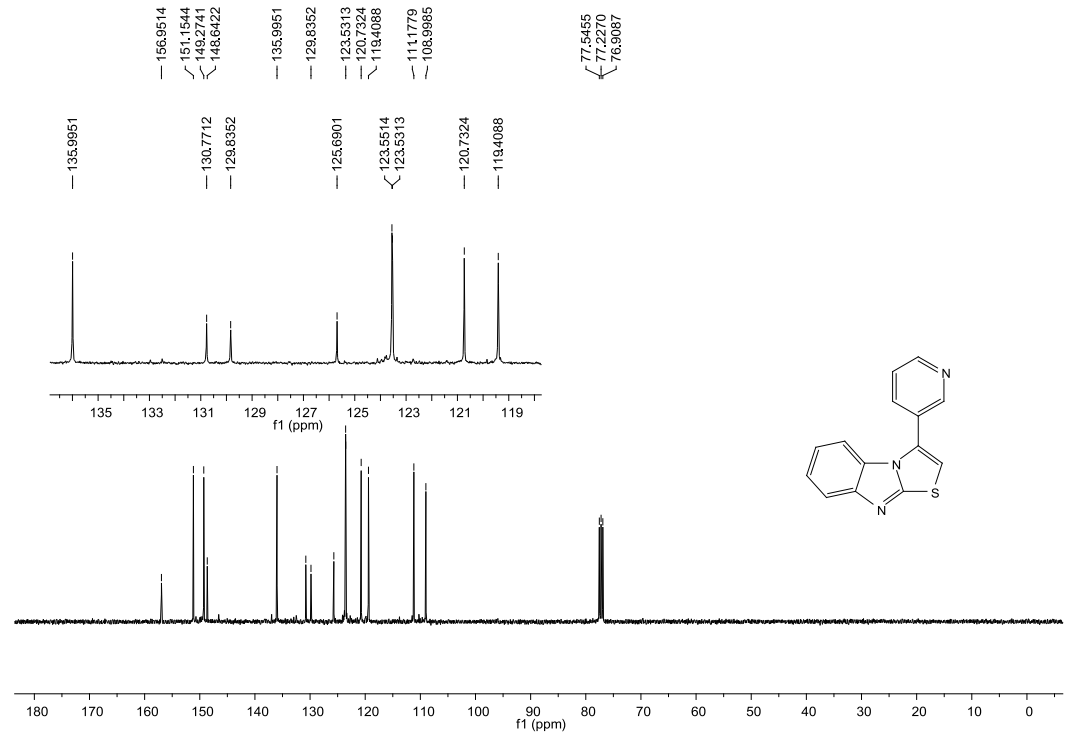

164.996
162.501
157.081
148.779
133.102
130.932
130.847
130.011
123.449
119.374
116.130
111.383
107.380
77.348
77.030
76.713
1.017
130.932
130.847
130.011
125.546
125.510
123.449
120.518
119.374
116.349
116.130
fl (ppm)
N
S
F

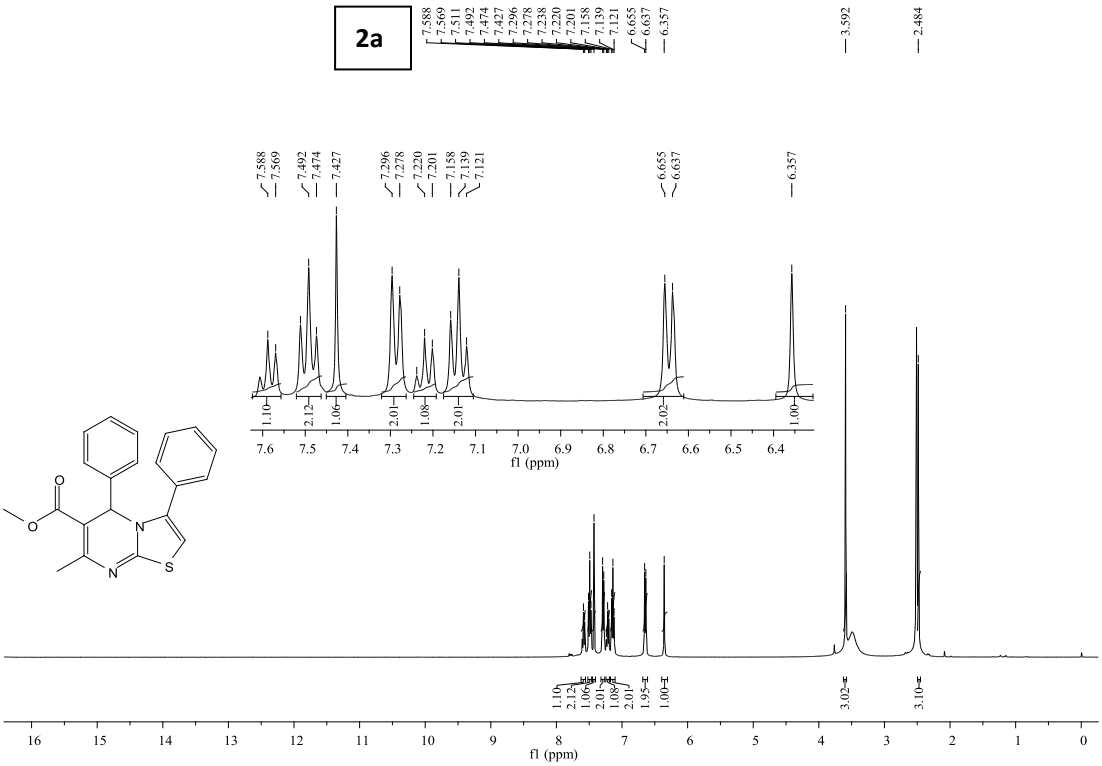

1e
f1 (ppm)

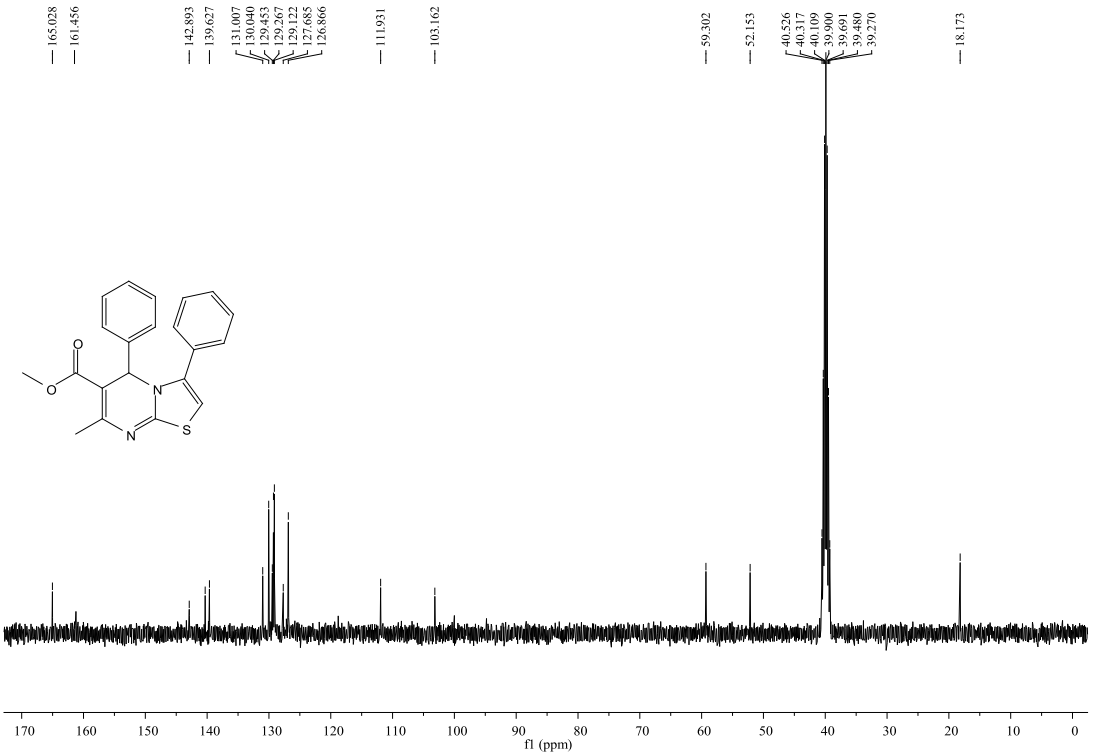

f1 (ppm)

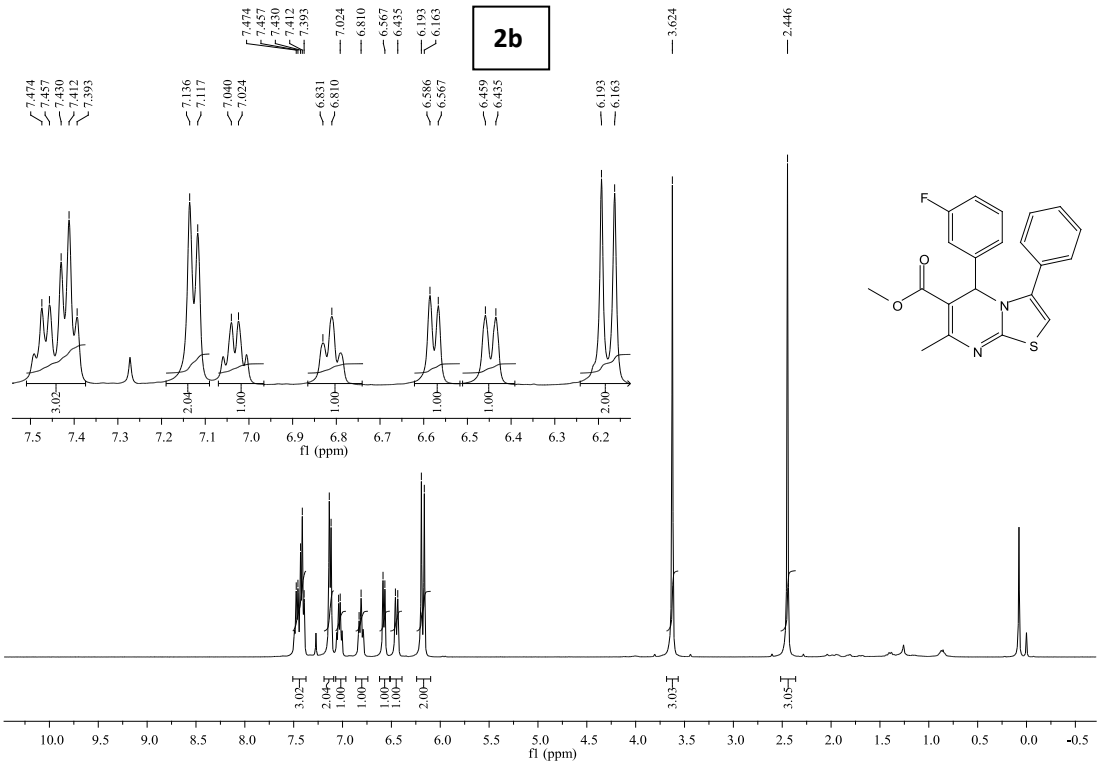
1f

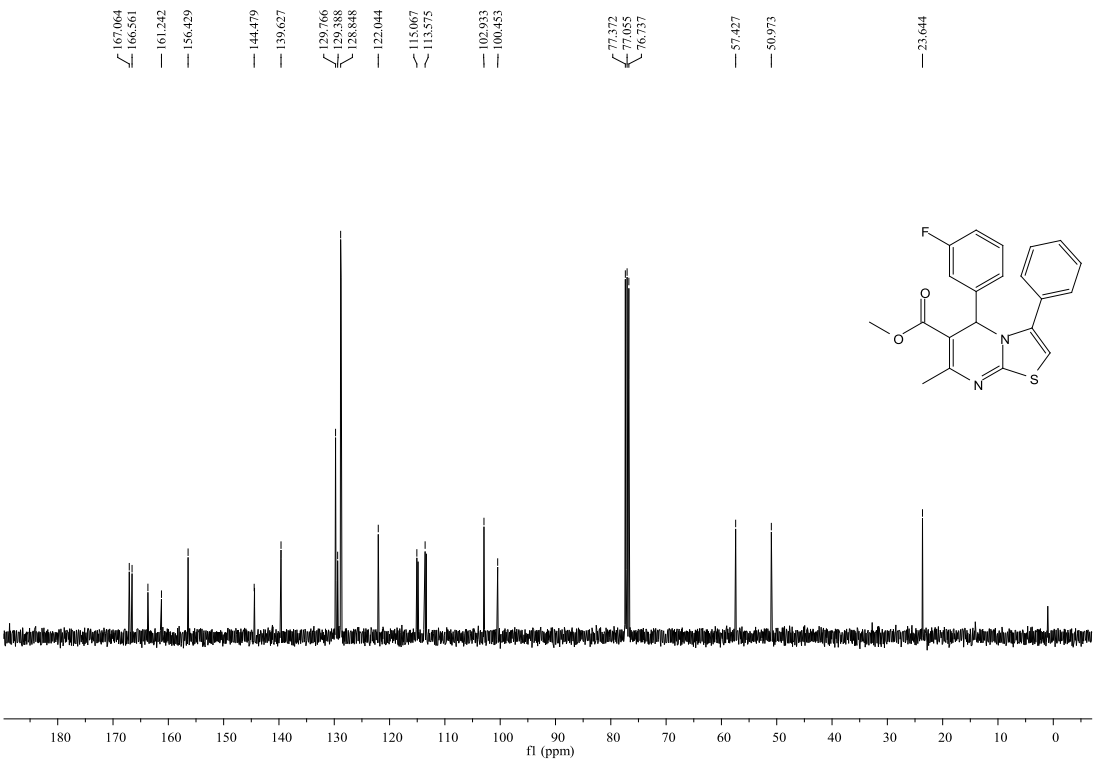

8.969
8.876
8.005
7.985
7.791
7.542
7.322
7.304
7.172
7.093
7.074
7.055
6.706
2.150
0.096
1.75
1.01
1.02
1.02
1.23
1.03
1.06
1.00
f1 (ppm)

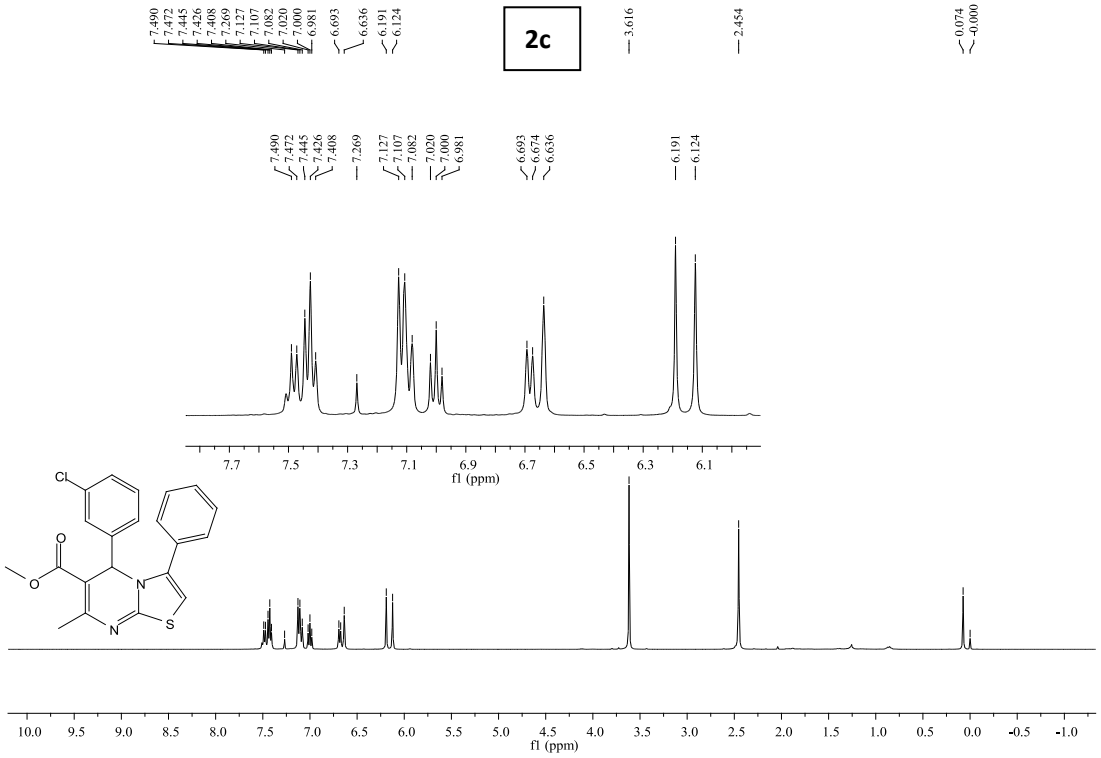

156.9514
151.1544
149.2741
148.6422
135.9951
129.8352
123.5313
120.7324
119.4088
111.1779
108.9985
77.5455
77.2270
76.9087
130.7712
125.6901
123.5514
f1 (ppm)

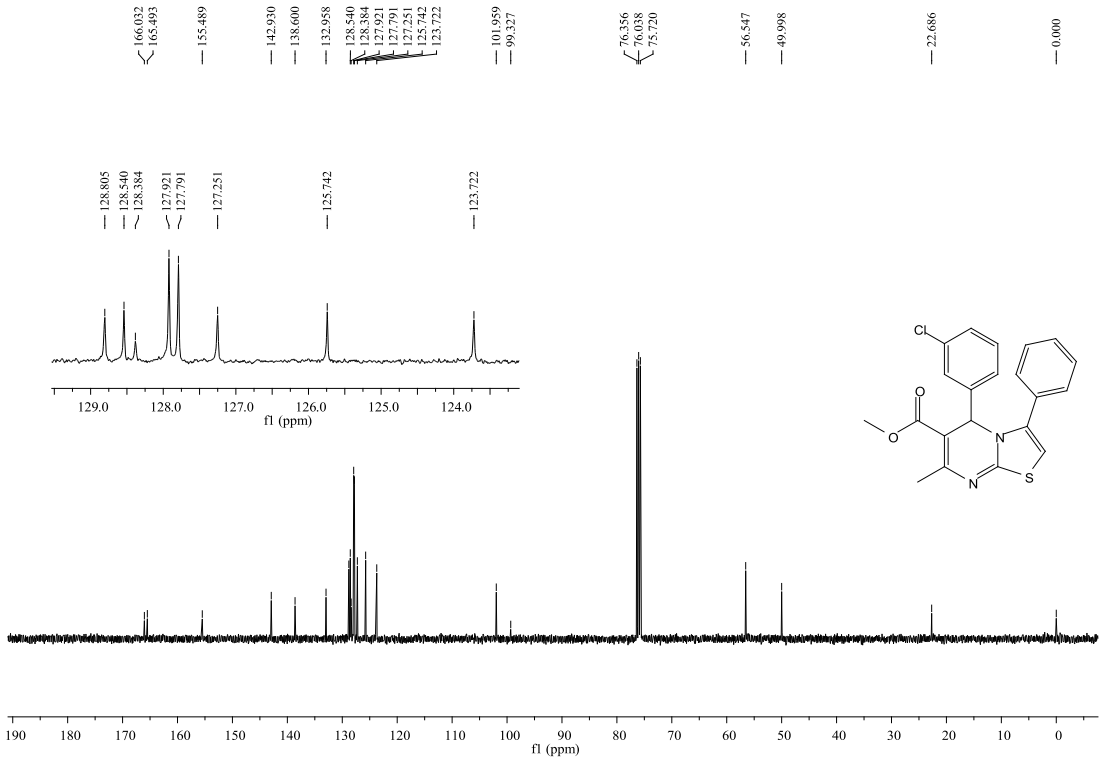

2a
7.588
7.569
7.511
7.492
7.474
7.427
7.296
7.278
7.238
7.220
7.201
7.158
7.139
7.121
6.655
6.637
6.357
3.592
2.484
fl (ppm)

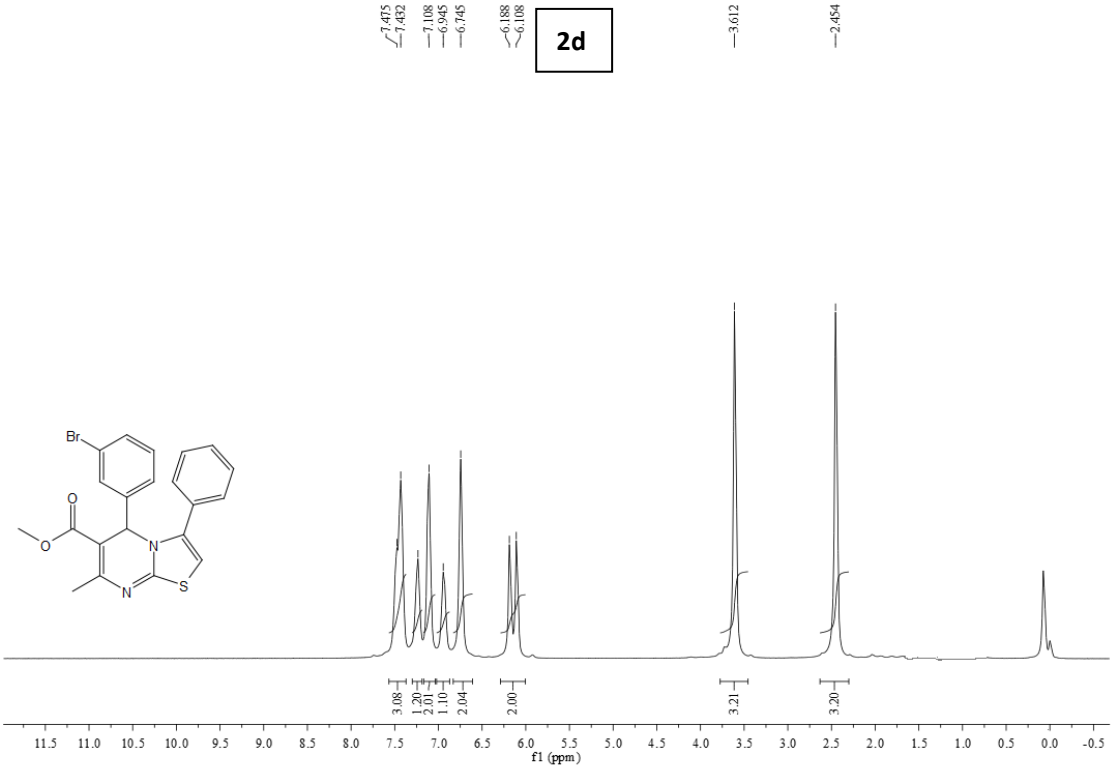

165.028
161.456
142.893
139.627
131.007
130.040
129.453
129.267
129.122
127.685
126.866
111.931
103.162
59.302
52.153
40.526
40.317
40.109
39.900
39.691
39.480
39.270
18.173
fl (ppm)

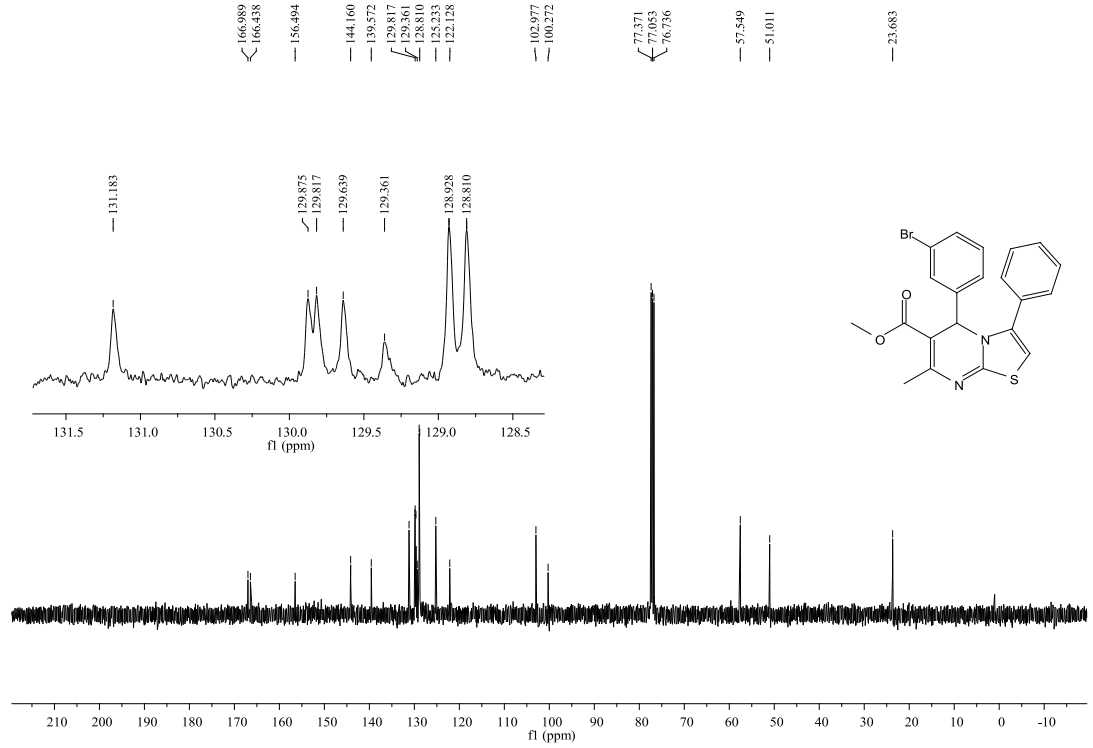

2b
7.474
7.457
7.430
7.412
7.393
7.024
6.810
6.567
6.435
6.193
6.163
3.624
2.446
7.136
7.117
7.040
6.831
6.586
6.459
f1 (ppm)
F
O
N
S


167.064
166.561
161.242
156.429
144.479
139.627
129.766
129.388
128.848
122.044
115.067
113.575
102.933
100.453
77.372
77.055
76.737
57.427
50.973
23.644
f1 (ppm)
F
O
N
S

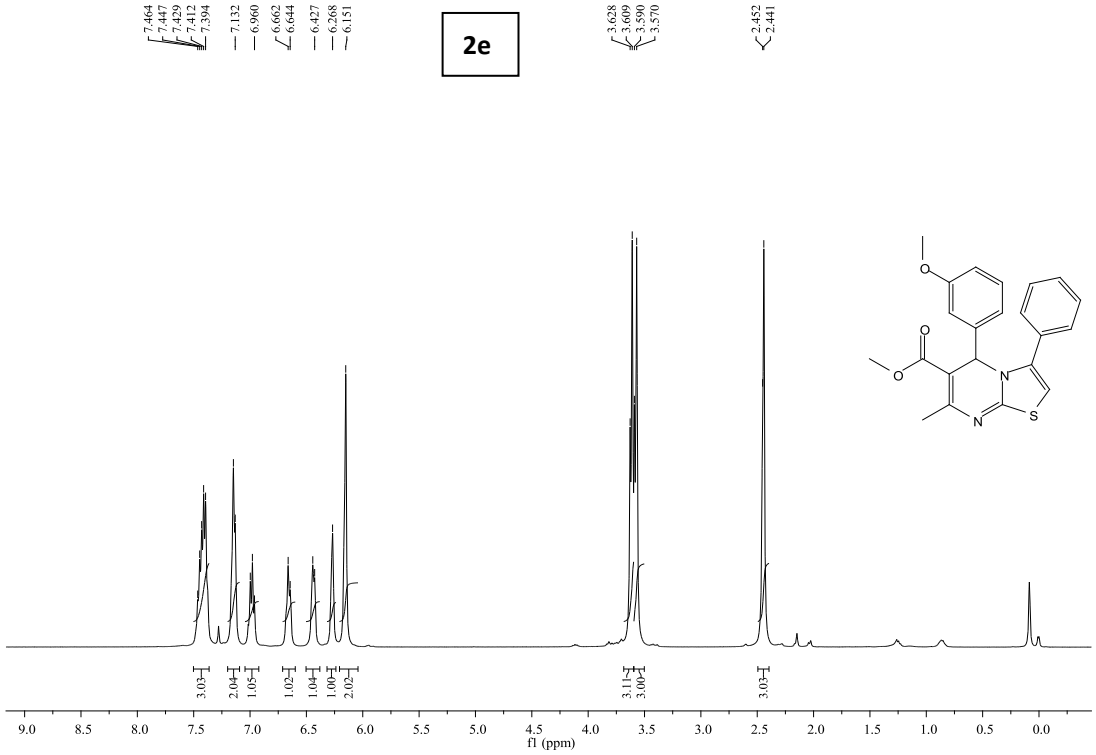

2c
7.490
7.472
7.445
7.426
7.408
7.269
7.127
7.107
7.082
7.020
7.000
6.981
6.693
6.636
6.191
6.124
3.616
2.454
0.074
-0.000
6.693
6.674
6.636
6.191
6.124
f1 (ppm)
Cl
O
O
N
N
S

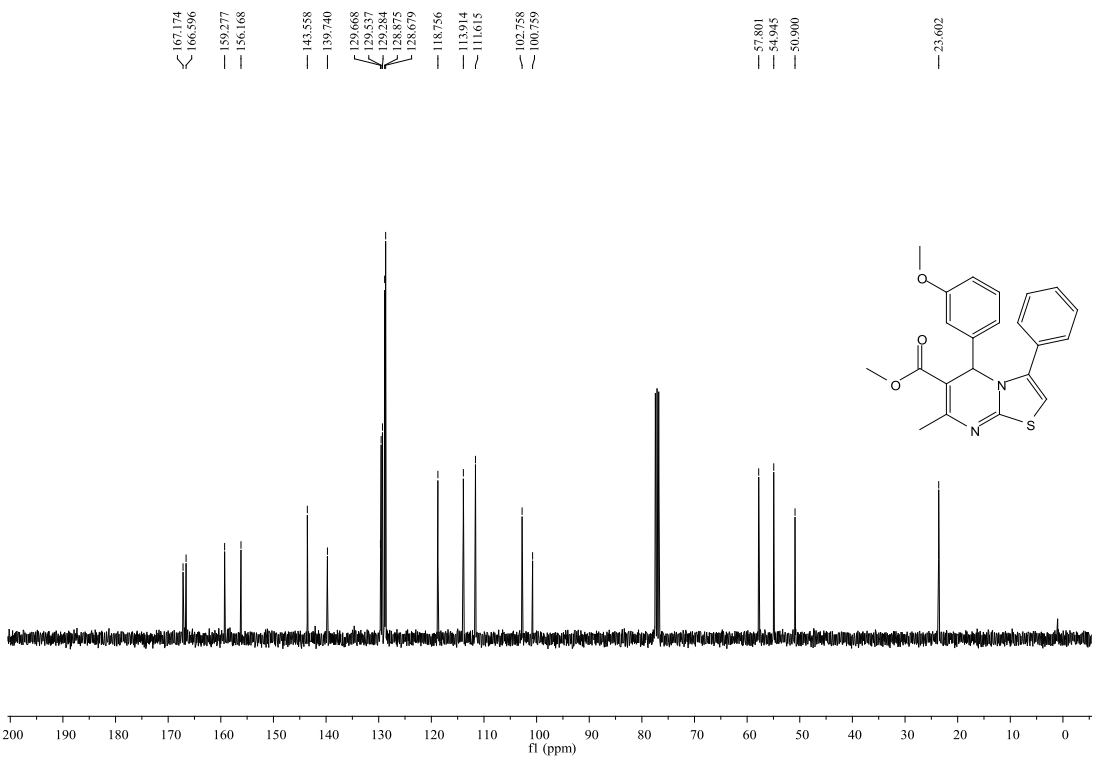

166.032
165.493
155.489
142.930
138.600
132.958
128.540
128.384
127.921
127.791
127.251
125.742
123.722
101.959
99.327
76.356
76.038
75.720
56.547
49.998
22.686
0.000
128.805
128.540
128.384
127.921
127.791
127.251
125.742
123.722
f1 (ppm)
Cl
O
O
N
N
S

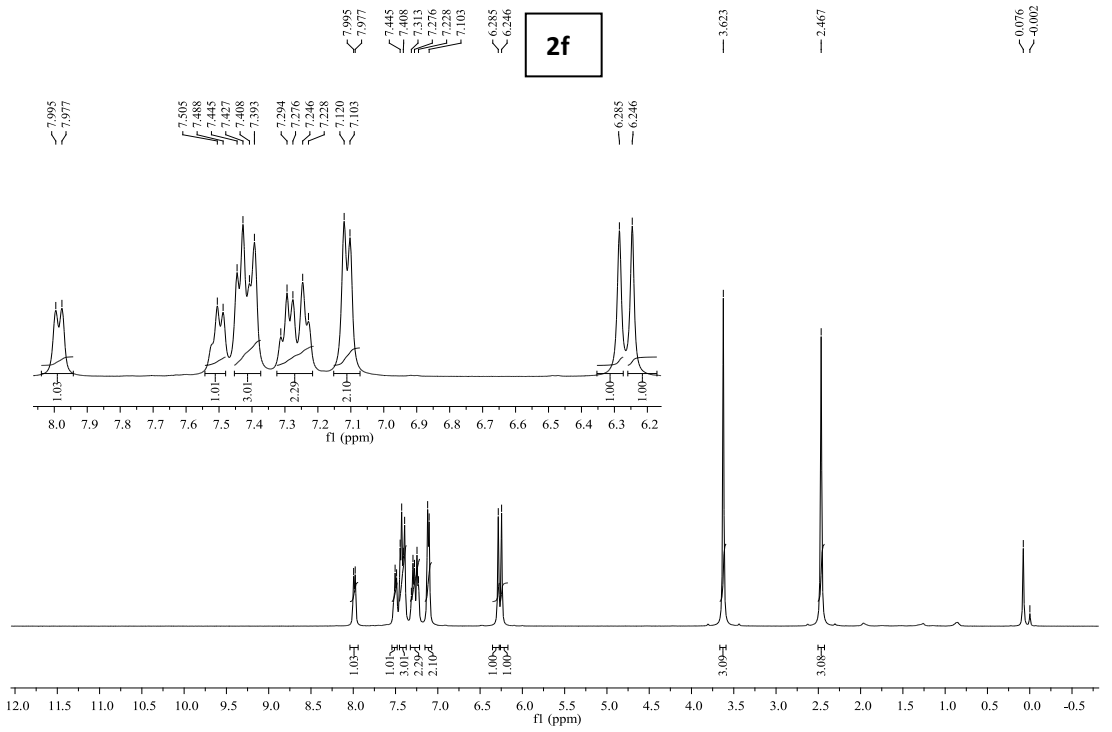

2d
7.475
7.432
7.108
6.945
6.745
6.188
6.108
3.612
2.454
Br
O
O
N
S
N
3.08
1.20
2.01
1.10
2.04
2.00
3.21
3.20
f1 (ppm)

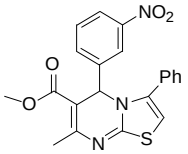

166.989
166.438
156.494
144.160
139.572
129.817
129.361
128.810
125.233
122.128
102.977
100.272
77.371
77.053
76.736
57.549
51.011
23.683
131.183
129.875
129.817
129.639
129.361
128.928
128.810
Br
O
O
N
S
N
f1 (ppm)
f1 (ppm)

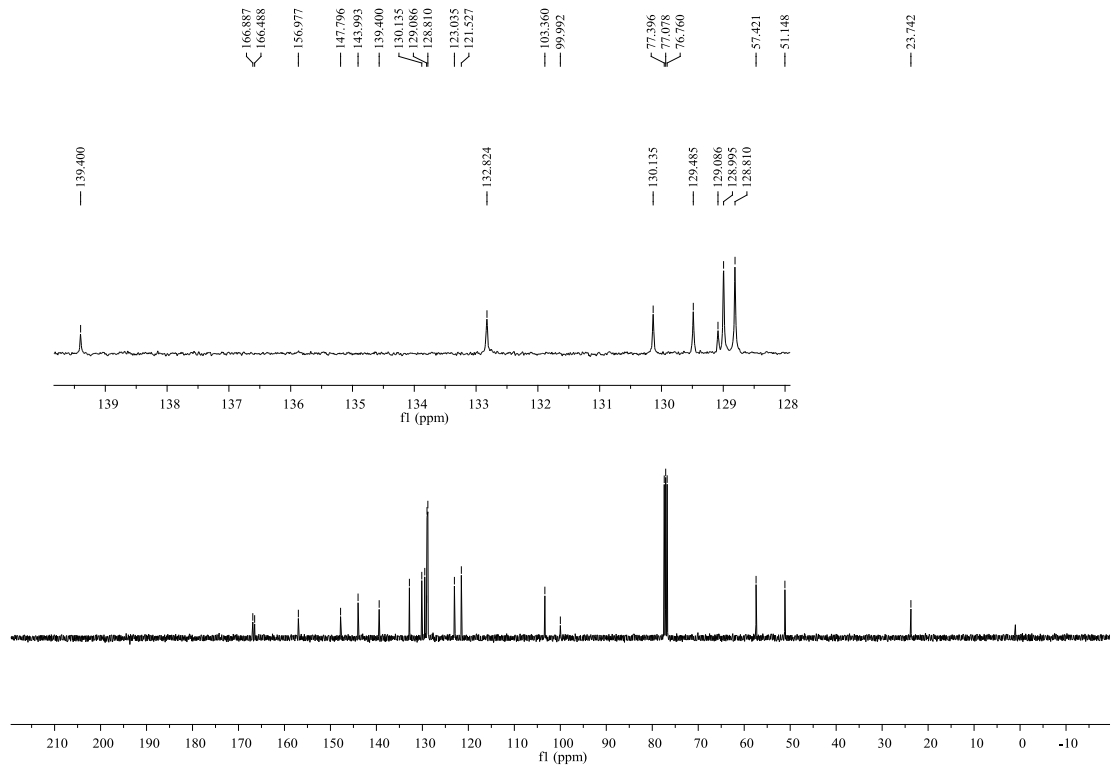
2e

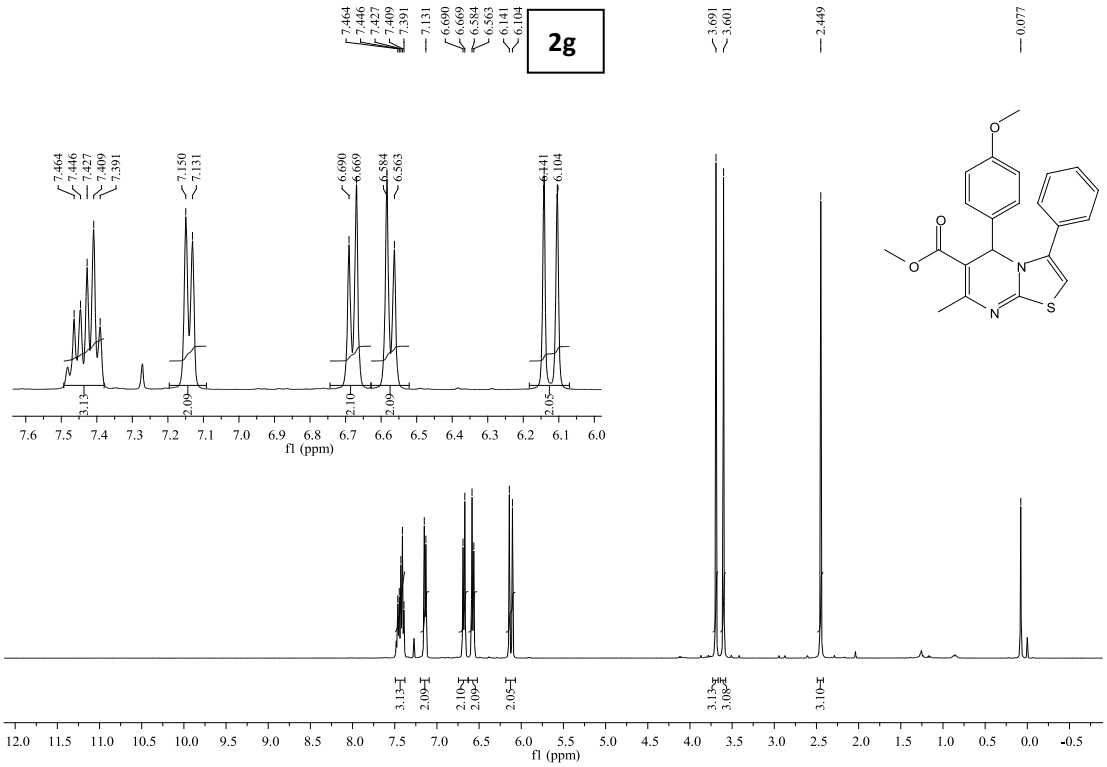

7.464
7.447
7.429
7.412
7.394
7.132
6.960
6.662
6.644
6.427
6.268
6.151
3.628
3.609
3.590
3.570
2.452
2.441
3.03
2.04
1.05
1.02
1.04
1.00
2.02
3.11
3.00
3.03
f1 (ppm)

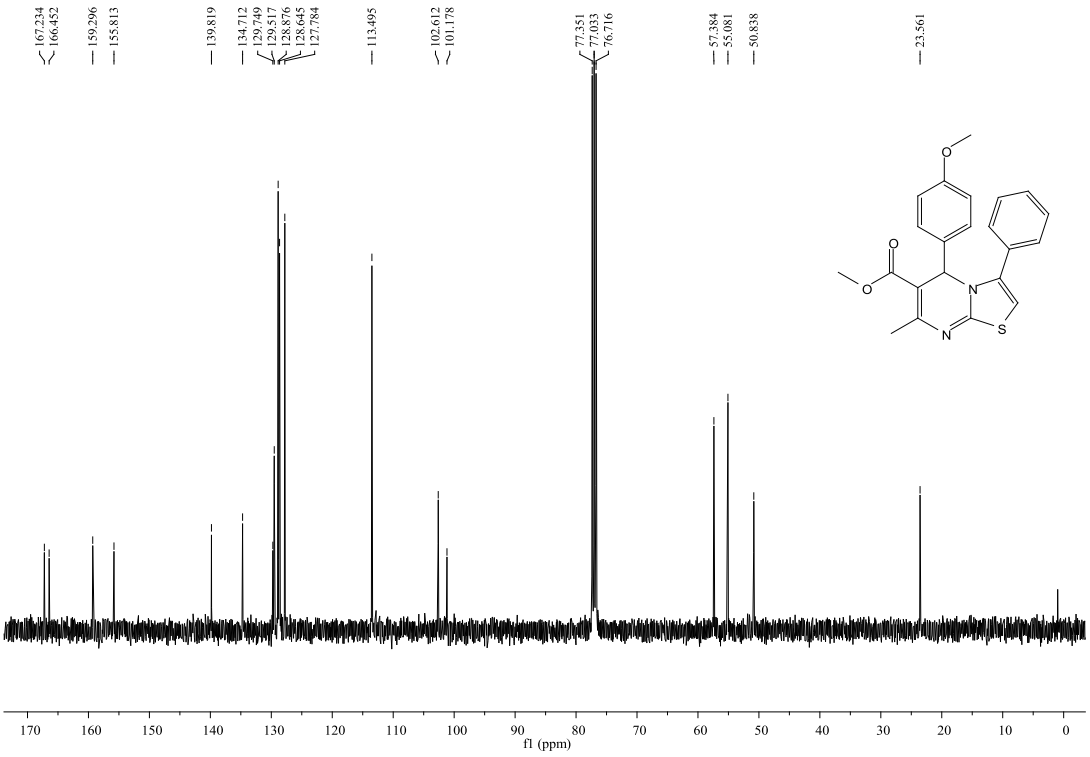

167.174
166.596
159.277
156.168
143.558
139.740
129.668
129.537
129.284
128.875
128.679
118.756
113.914
111.615
102.758
100.759
57.801
54.945
50.900
23.602
f1 (ppm)

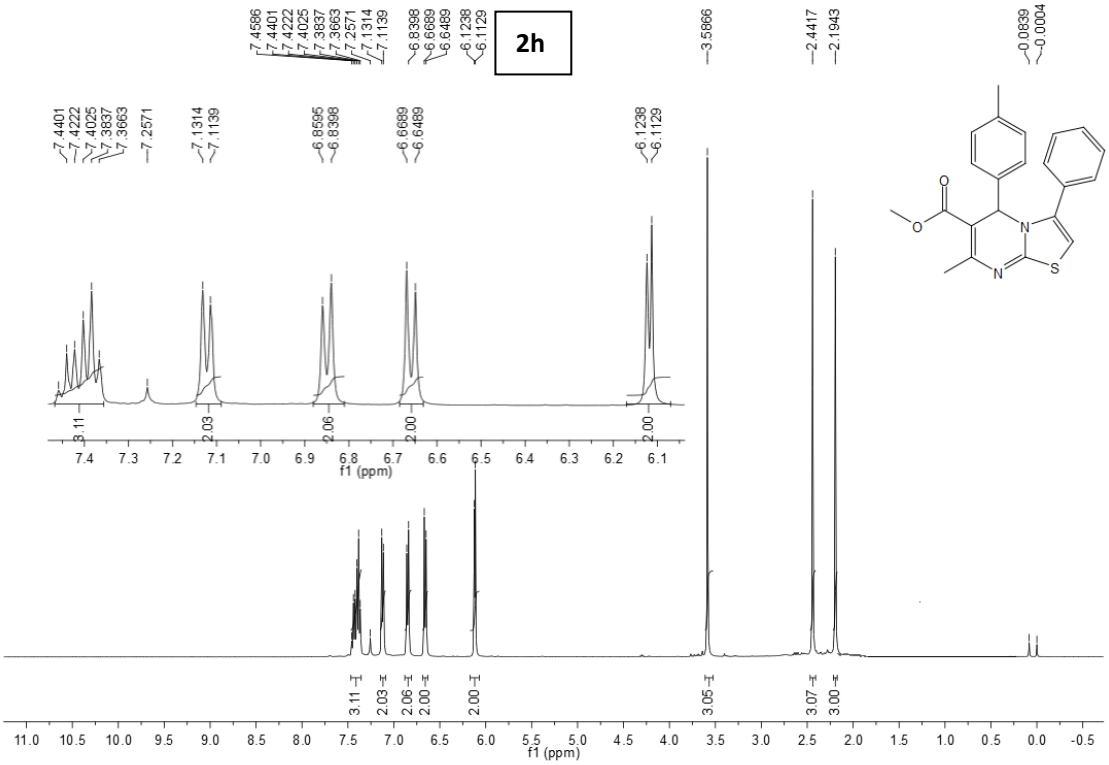

2f
f1 (ppm)

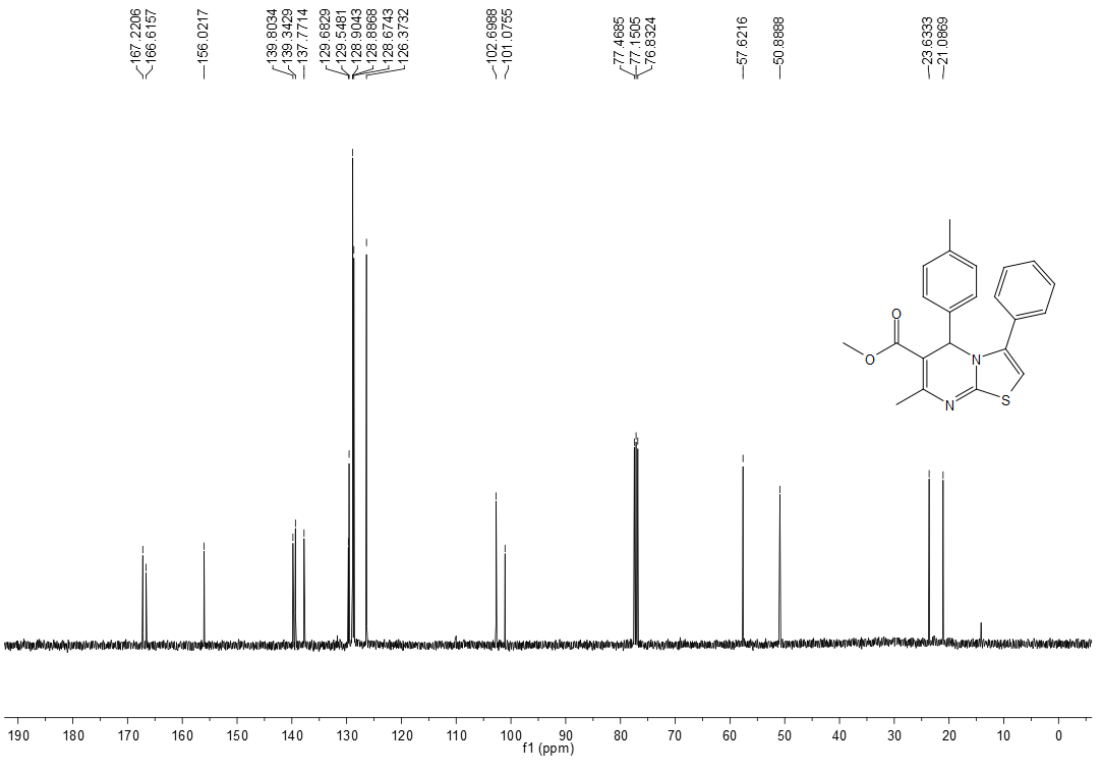

NO2
O
O
Ph
N
N
S

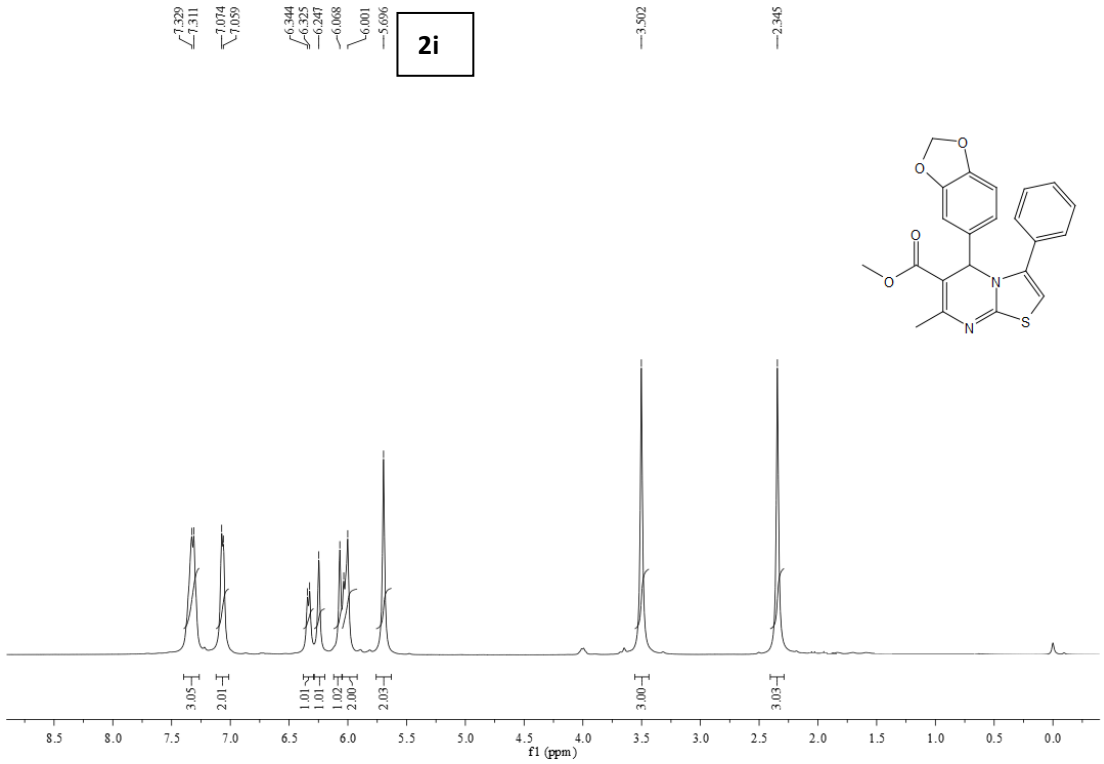

f1 (ppm)

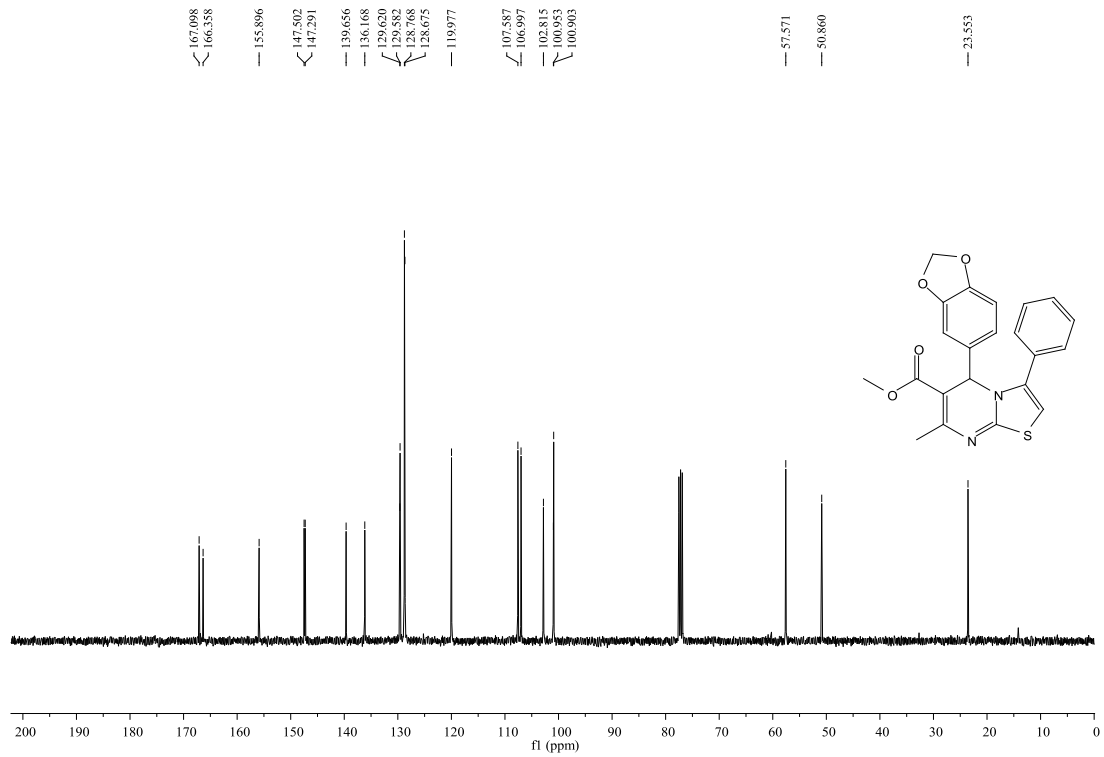

2g
7.464
7.446
7.427
7.409
7.391
7.131
6.690
6.669
6.584
6.563
6.141
6.104
3.691
3.601
2.449
0.077
fl (ppm)

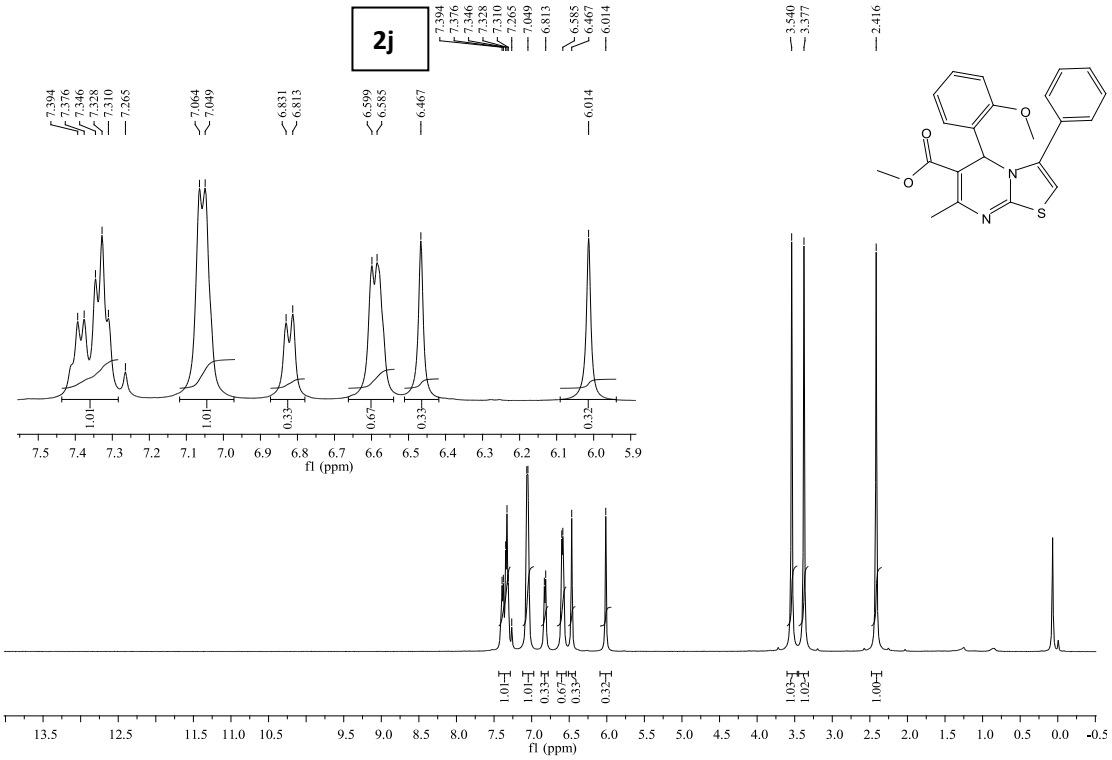

167.234
166.452
159.296
155.813
139.819
134.712
129.749
129.517
128.876
128.645
127.784
113.495
102.612
101.178
77.351
77.033
76.716
57.384
55.081
50.838
23.561
fl (ppm)

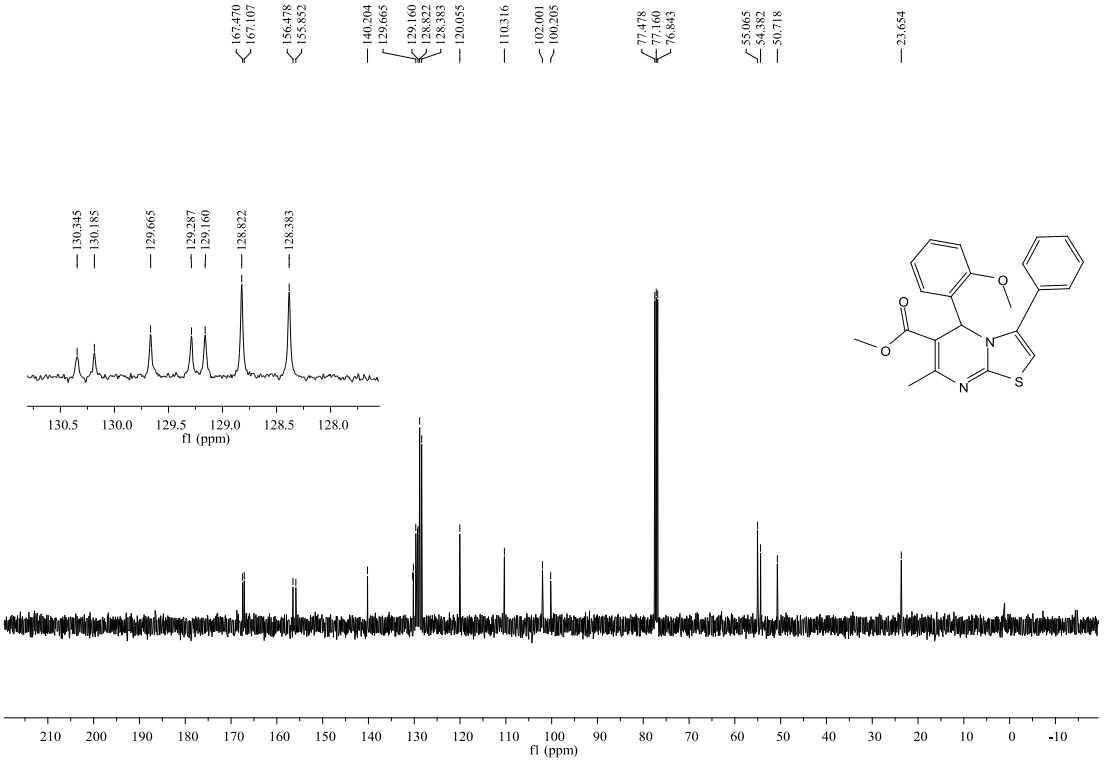

2h
7.4586
7.4401
7.4222
7.4025
7.3837
7.3663
7.2571
7.1314
7.1139
6.8398
6.6689
6.6489
6.1238
6.1129
3.5866
2.4417
2.1943
0.0839
0.0004
3.11
2.03
2.06
2.00
2.00
3.05
3.07
3.00
f1 (ppm)

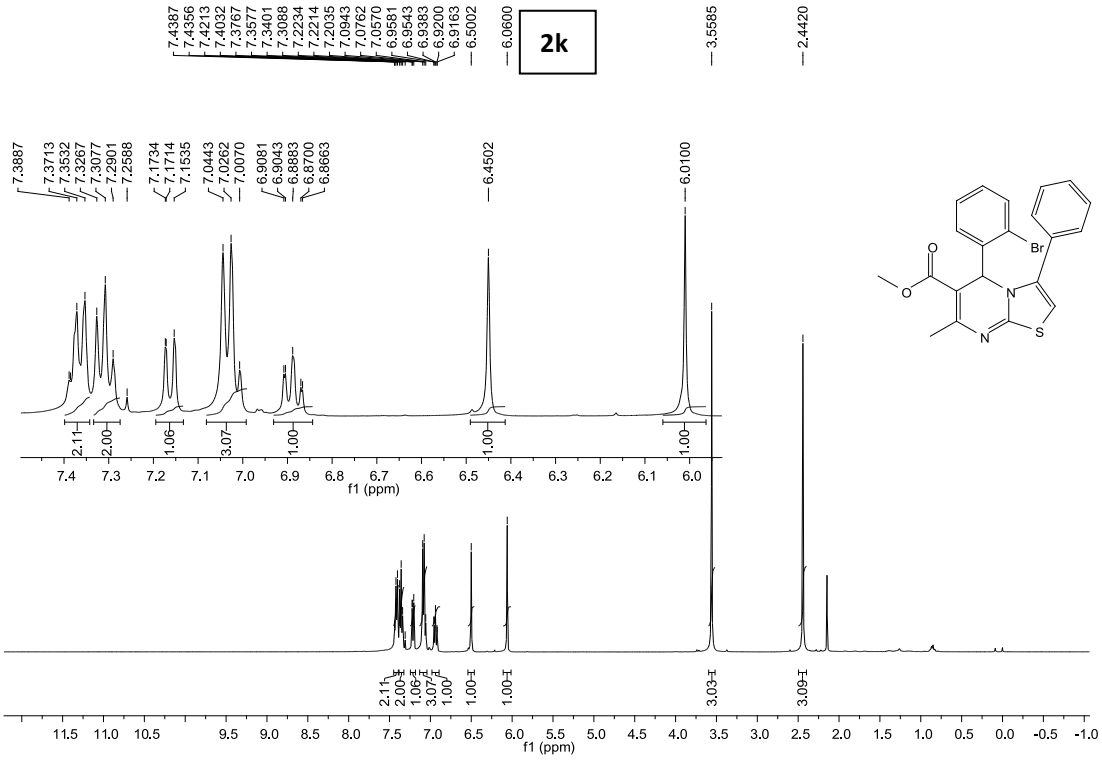

167.2206
166.6157
156.0217
139.8034
139.3429
137.7714
129.6829
129.5481
128.9043
128.8868
128.6743
126.3732
102.6988
101.0755
77.4685
77.1505
76.8324
57.6216
50.8888
23.6333
21.0869
f1 (ppm)

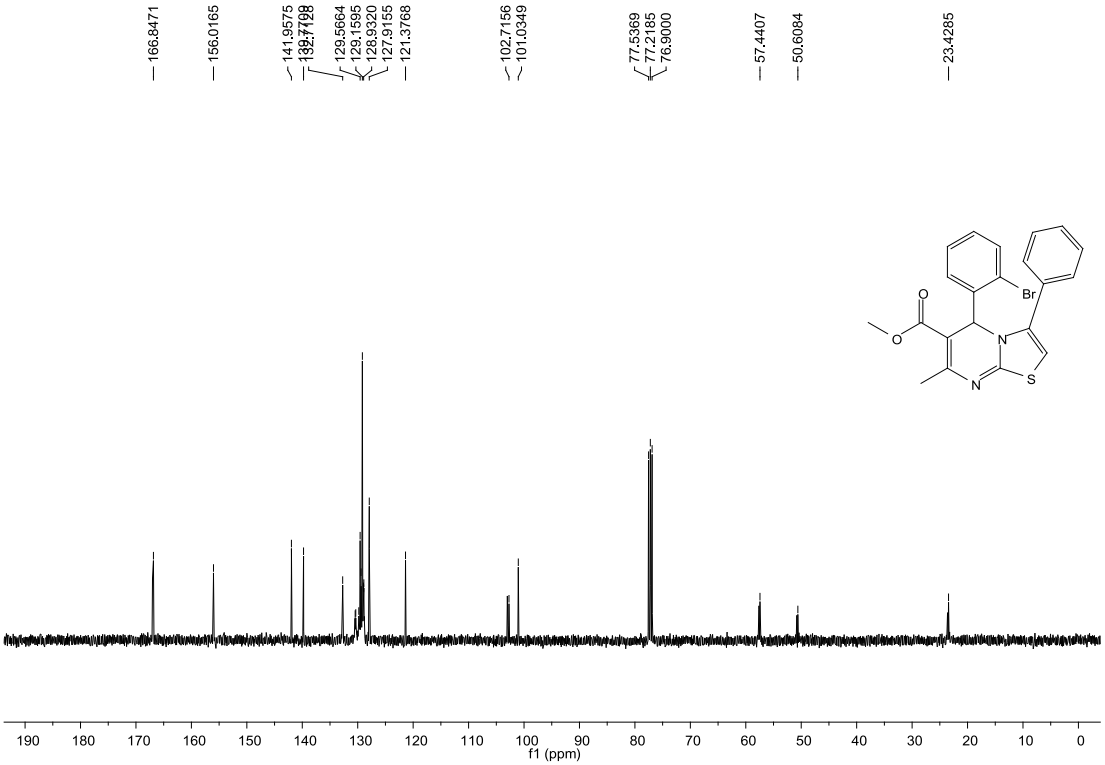

2i
7.329
7.311
7.074
7.059
6.344
6.325
6.247
6.068
6.001
5.696
3.502
2.345
3.05
2.01
1.01
1.01
1.02
2.00
2.03
3.00
3.03
f1 (ppm)

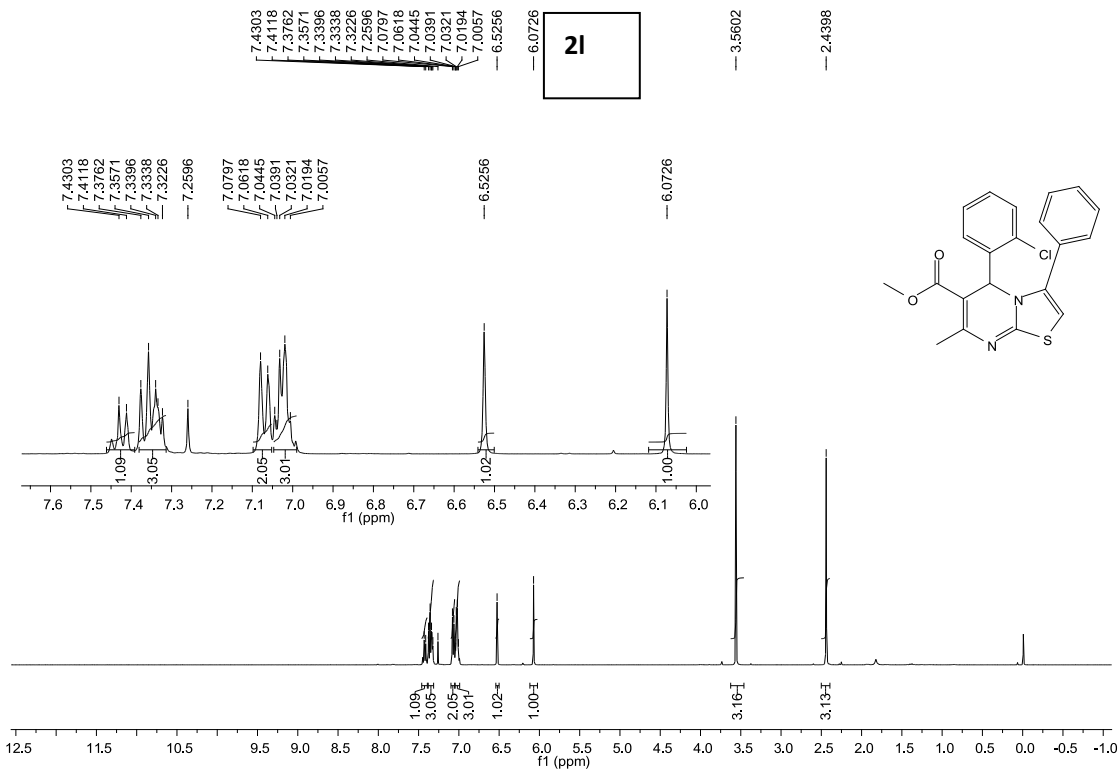

167.098
166.358
155.896
147.502
147.291
139.656
136.168
129.620
129.582
128.768
128.675
119.977
107.587
106.997
102.815
100.953
100.903
57.571
50.860
23.553
f1 (ppm)

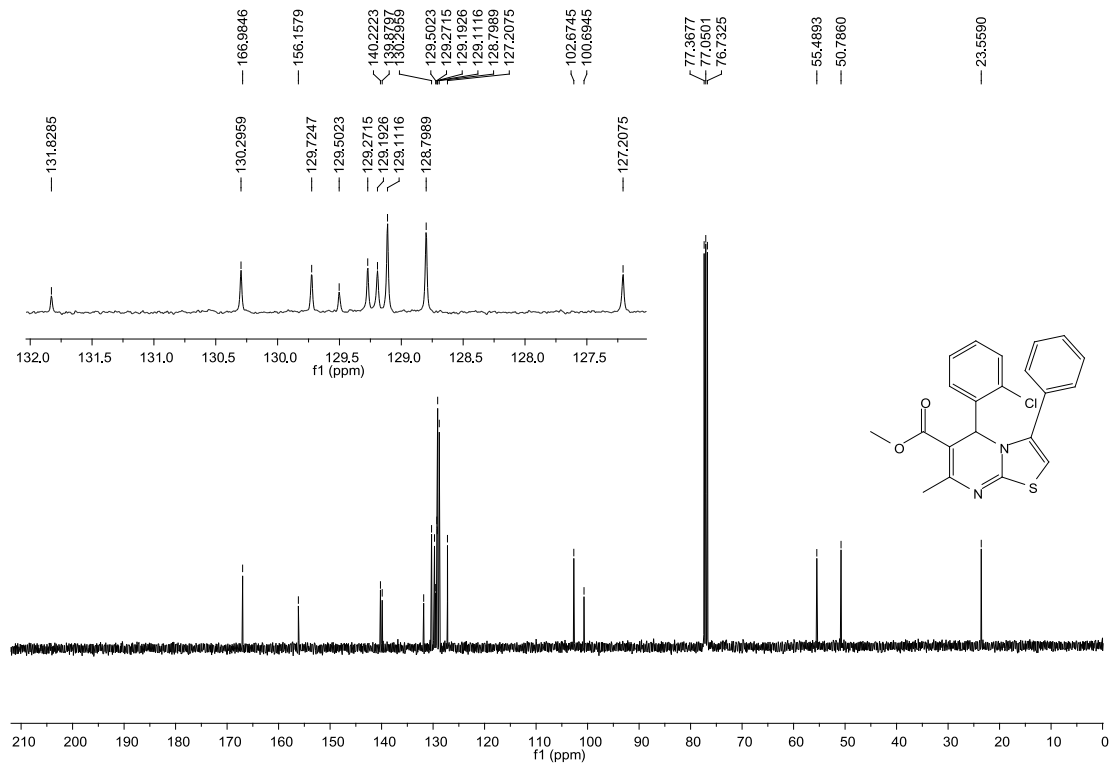

2j
7.394
7.376
7.346
7.328
7.310
7.265
7.064
7.049
6.831
6.813
6.599
6.585
6.467
6.014
3.540
3.377
2.416
fl (ppm)

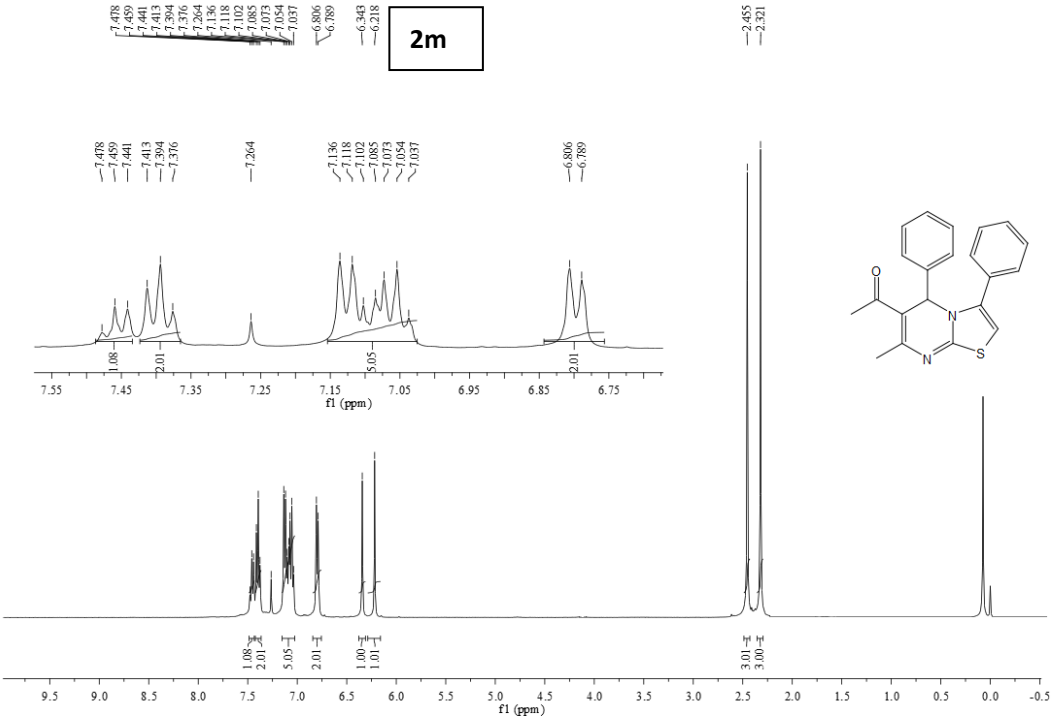

167.470
167.107
156.478
155.852
140.204
129.665
129.160
128.822
128.383
120.055
110.316
102.001
100.205
77.478
77.160
76.843
55.065
54.382
50.718
23.654
130.345
130.185
129.665
129.287
129.160
128.822
128.383
fl (ppm)

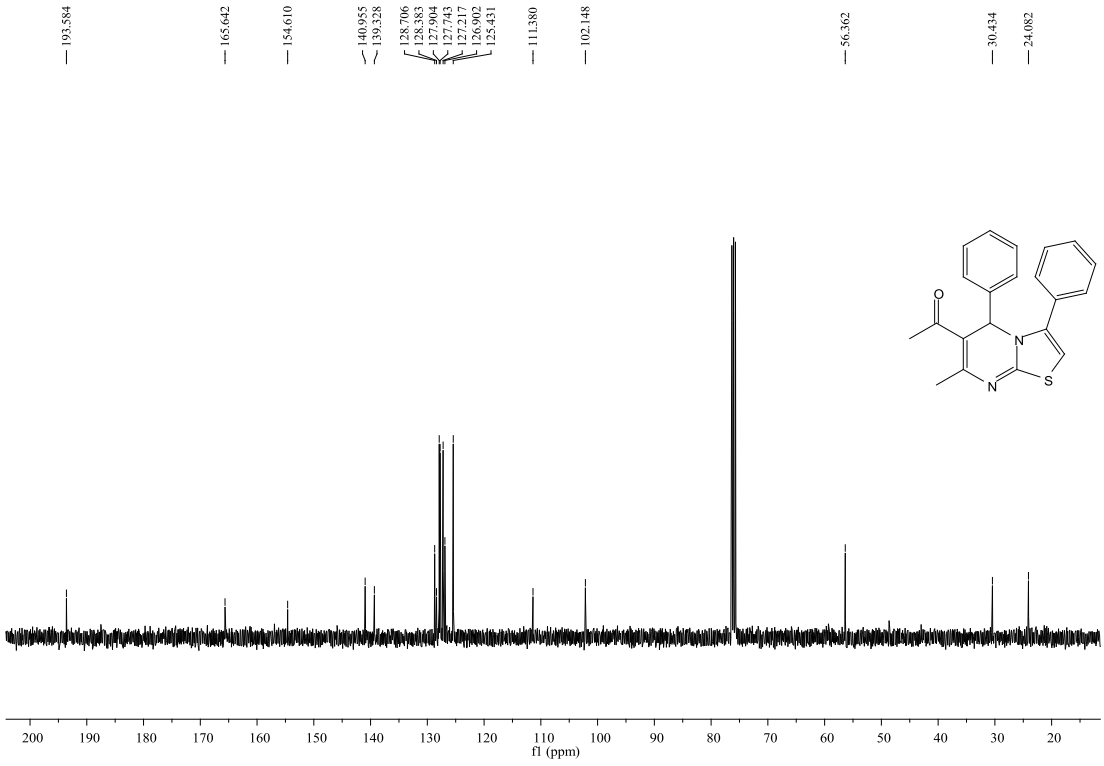

2k
7.4387
7.4356
7.4213
7.4032
7.3767
7.3577
7.3401
7.3088
7.2234
7.2214
7.2035
7.0943
7.0762
7.0570
6.9581
6.9543
6.9383
6.9200
6.9163
6.5002
6.0600
3.5585
2.4420
7.3887
7.3713
7.3532
7.3267
7.3077
7.2901
7.2588
7.1734
7.1714
7.1535
7.0443
7.0262
7.0070
6.9081
6.9043
6.8883
6.8700
6.8663
6.4502
6.0100
2.11
2.00
1.06
3.07
1.00
1.00
1.00
1.00
3.03
3.09
f1 (ppm)
Br
O
O
N
N
S

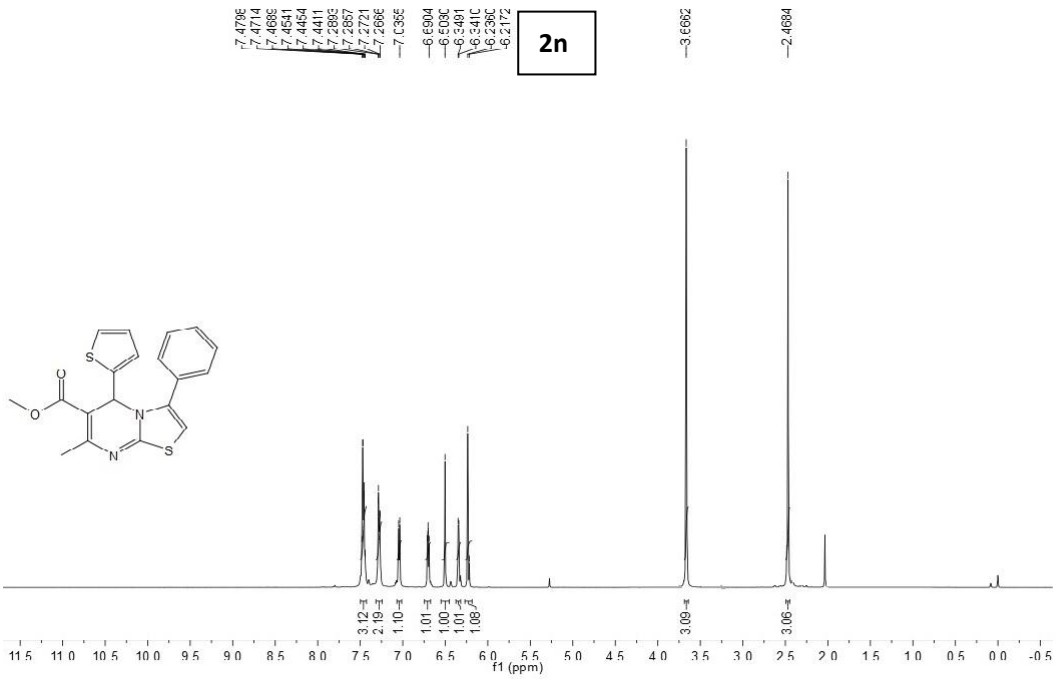

166.8471
156.0165
141.9575
139.7198
129.5664
129.1595
128.9320
127.9155
121.3768
102.7156
101.0349
77.5369
77.2185
76.9000
57.4407
50.6084
23.4285
f1 (ppm)
Br
O
O
N
N
S

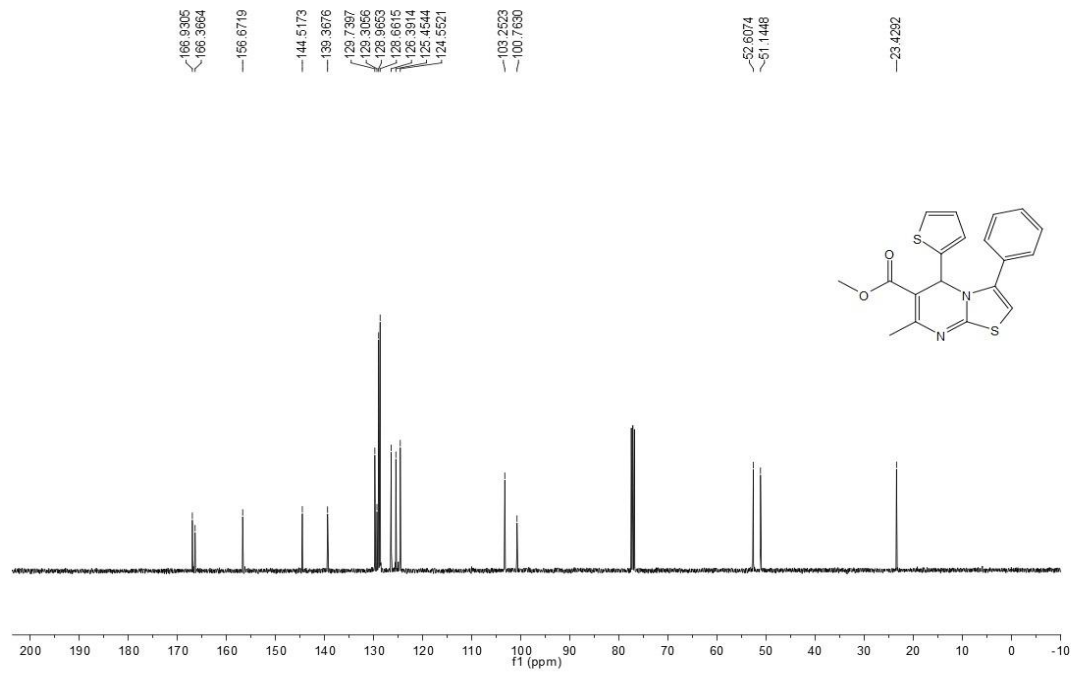

2l
7.4303
7.4118
7.3762
7.3571
7.3396
7.3338
7.3226
7.2596
7.0797
7.0618
7.0445
7.0391
7.0321
7.0194
7.0057
6.5256
6.0726
3.5602
2.4398
Cl
O
N
S
f1 (ppm)

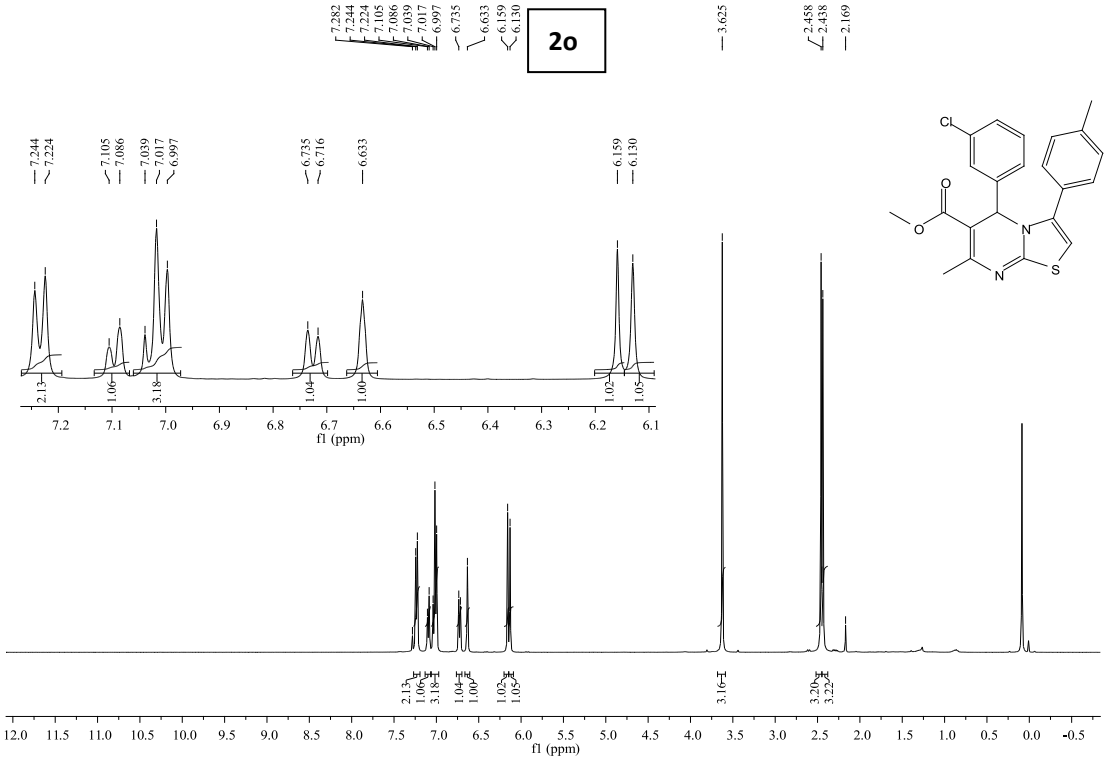

166.9846
156.1579
140.2223
139.8797
130.2959
129.5023
129.2715
129.1926
129.1116
128.7989
127.2075
102.6745
100.6945
77.3677
77.0501
76.7325
55.4893
50.7860
23.5590
131.8285
130.2959
129.7247
129.5023
129.2715
129.1926
129.1116
128.7989
127.2075
Cl
O
N
S
f1 (ppm)

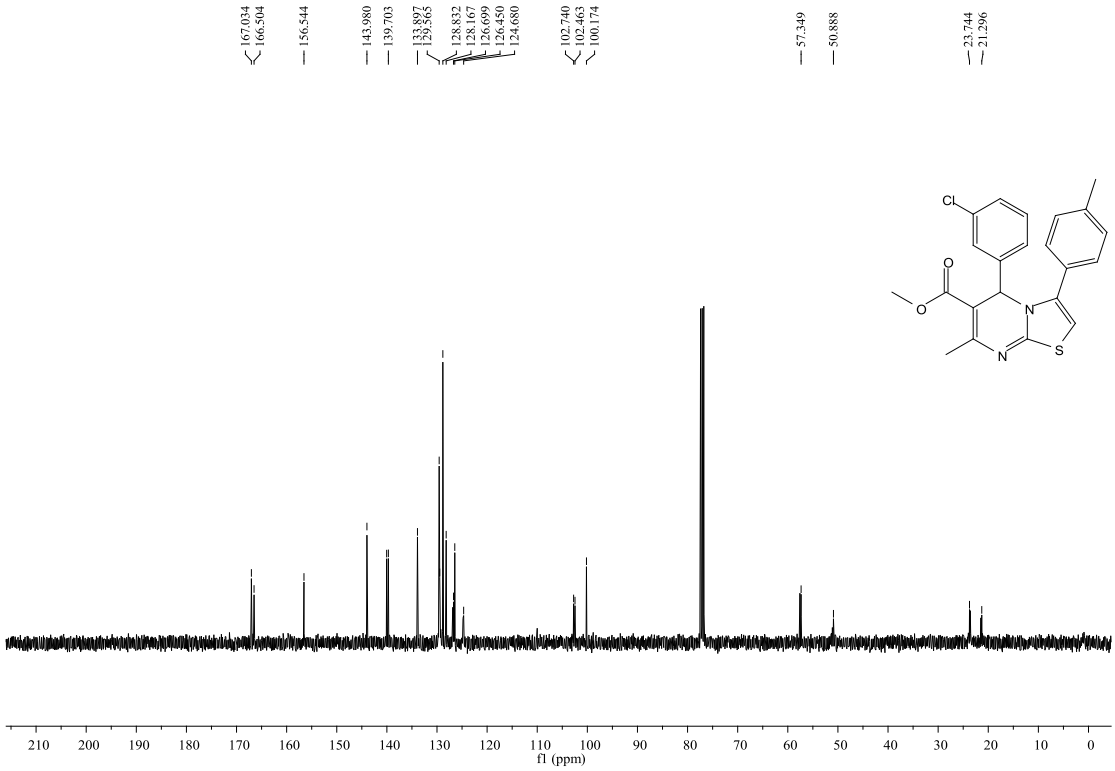

2m
7.478
7.459
7.441
7.413
7.394
7.376
7.264
7.136
7.118
7.102
7.085
7.073
7.054
7.037
6.806
6.789
6.343
6.218
2.455
2.321
1.08
2.01
5.05
2.01
1.00
1.01
3.01
3.00
f1 (ppm)

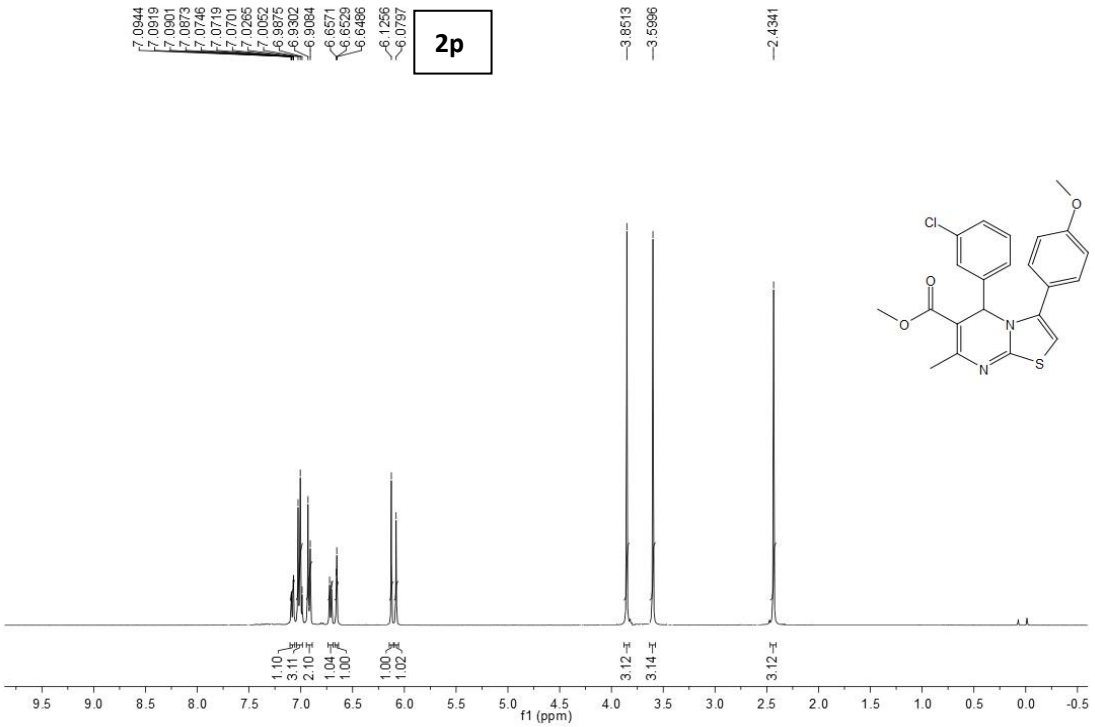

193.584
165.642
154.610
140.955
139.328
128.706
128.383
127.904
127.743
127.217
126.902
125.431
111.380
102.148
56.362
30.434
24.082
f1 (ppm)

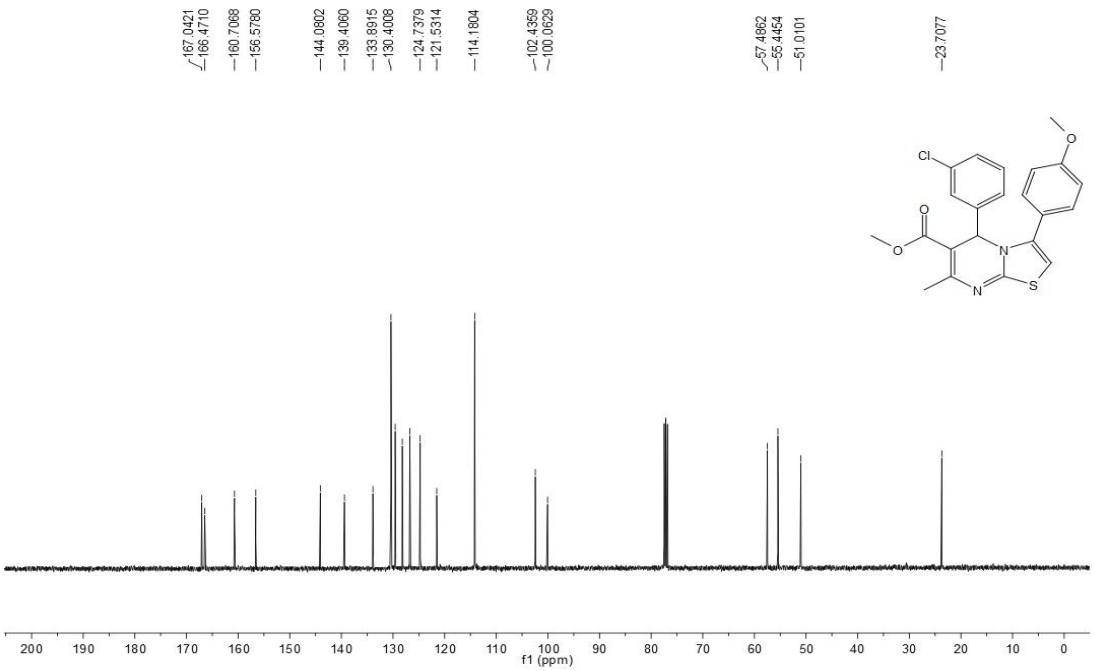

2n
7.4706
7.4714
7.4688
7.4541
7.4454
7.4411
7.2893
7.2857
7.2721
7.2666
7.0365
6.6904
6.5036
6.3491
6.3410
6.2365
6.2172
3.6662
2.4684
3.12
2.19
1.10
1.01
1.00
1.01
1.08
3.09
3.06
f1 (ppm)


166.9305
166.3664
156.6719
144.5173
139.3676
129.7397
129.3056
128.9653
128.6615
126.3914
125.4544
124.5521
103.2523
100.7630
52.6074
51.1448
23.4292
f1 (ppm)

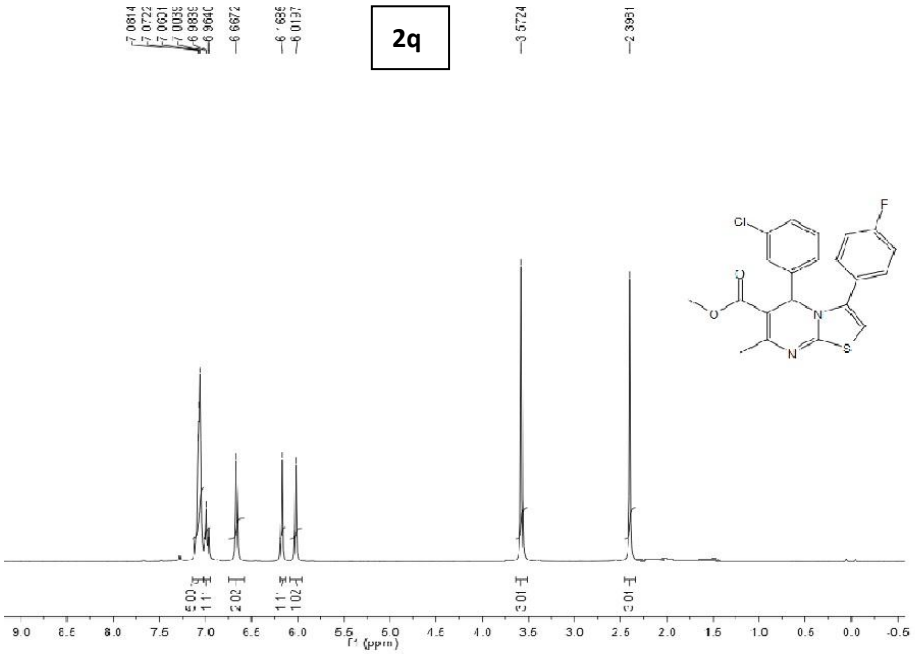

2o
7.282
7.244
7.224
7.105
7.086
7.039
7.017
6.997
6.735
6.633
6.159
6.130
3.625
2.458
2.438
2.169
Cl
O
O
N
N
S
fl (ppm)

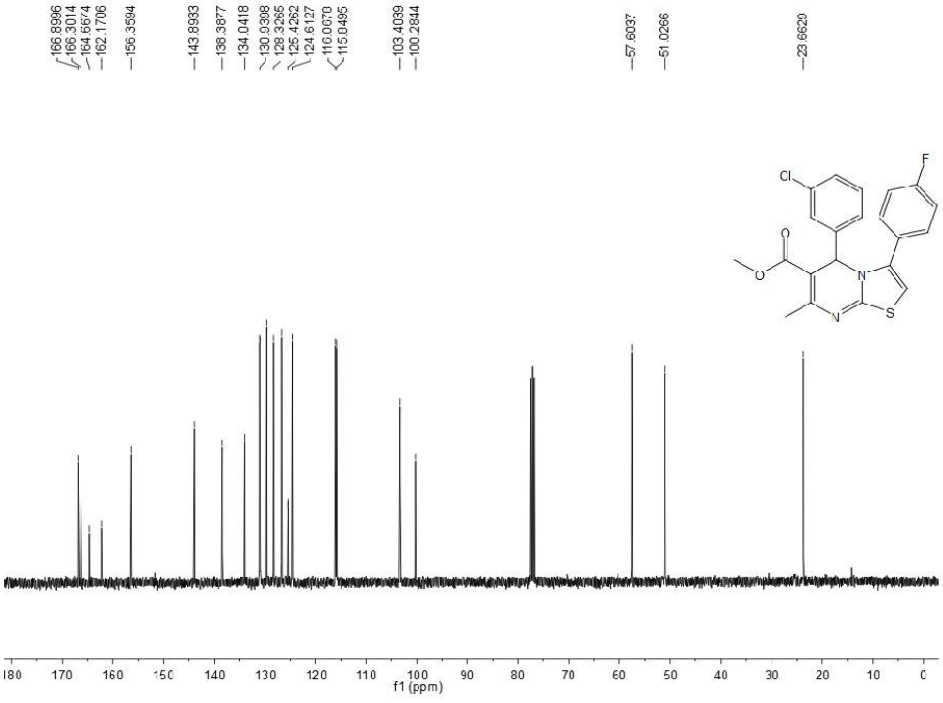

167.034
166.504
156.544
143.980
139.703
133.897
129.565
128.832
128.167
126.699
126.450
124.680
102.740
102.463
100.174
57.349
50.888
23.744
21.296
Cl
O
O
N
N
S
fl (ppm)

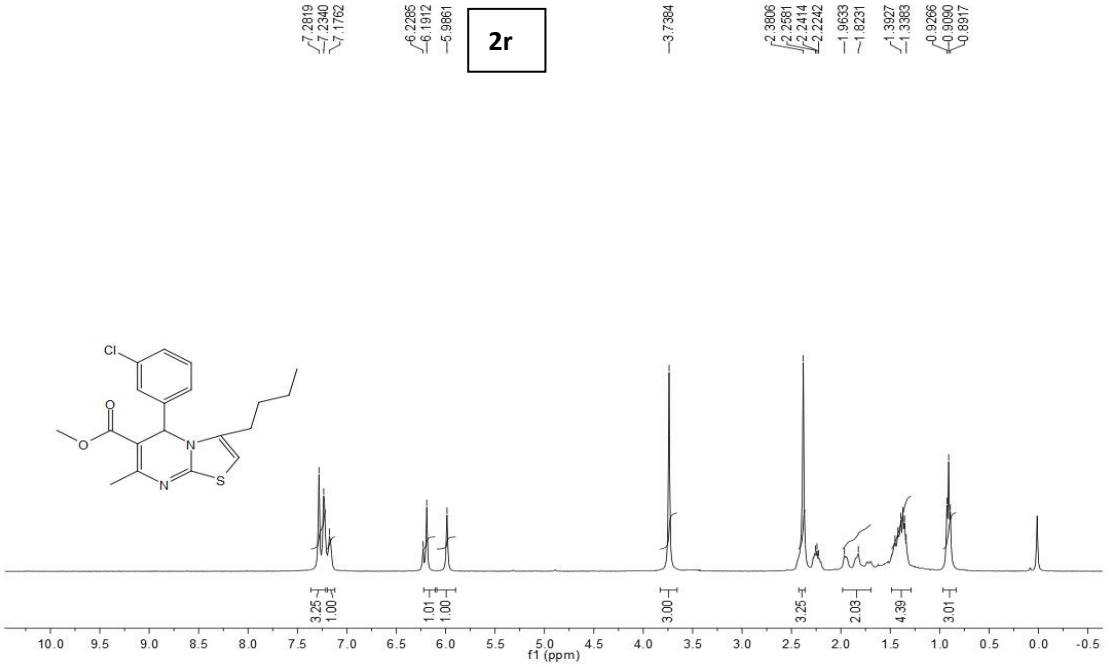

7.0944
7.0919
7.0901
7.0873
7.0746
7.0719
7.0701
7.0265
7.0052
6.9875
6.9302
6.9084
6.6571
6.6529
6.6486
6.1256
6.0797
2p
3.8513
3.5996
2.4341
Cl
O
O
O
N
N
S
1.10
3.11
2.10
1.04
1.00
1.00
1.02
3.12
3.14
3.12
f1 (ppm)

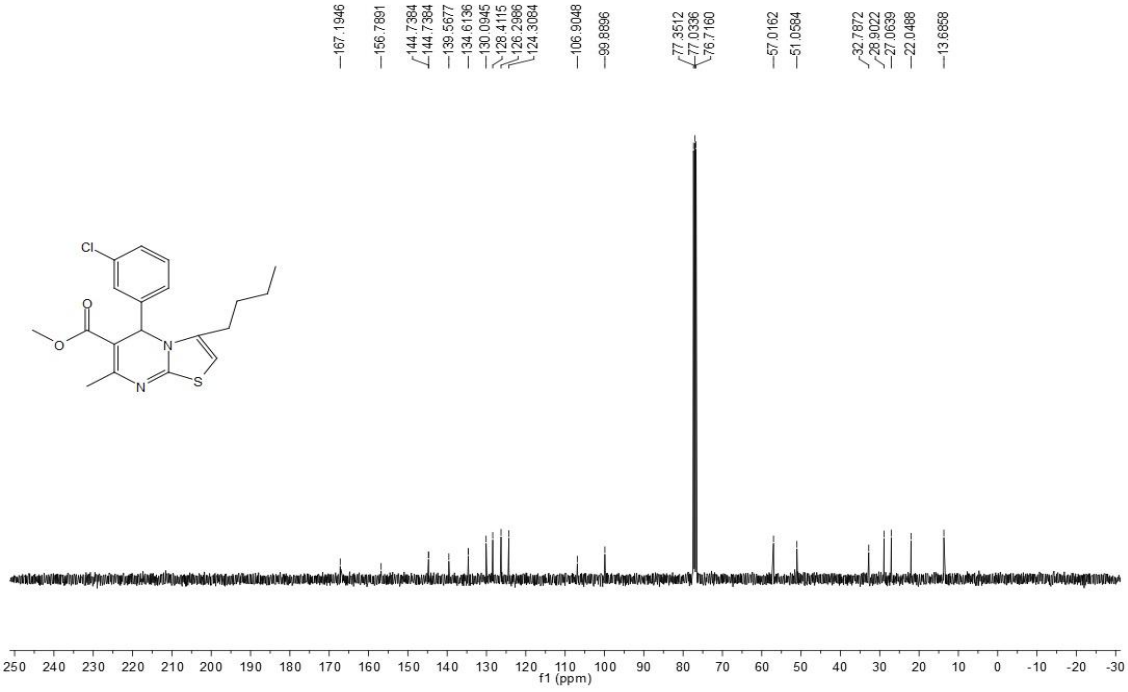

167.0421
166.4710
160.7068
156.5780
144.0802
139.4060
133.8915
130.4008
124.7379
121.5314
114.1804
102.4359
100.0629
57.4862
55.4454
51.0101
23.7077
Cl
O
O
O
N
N
S
f1 (ppm)

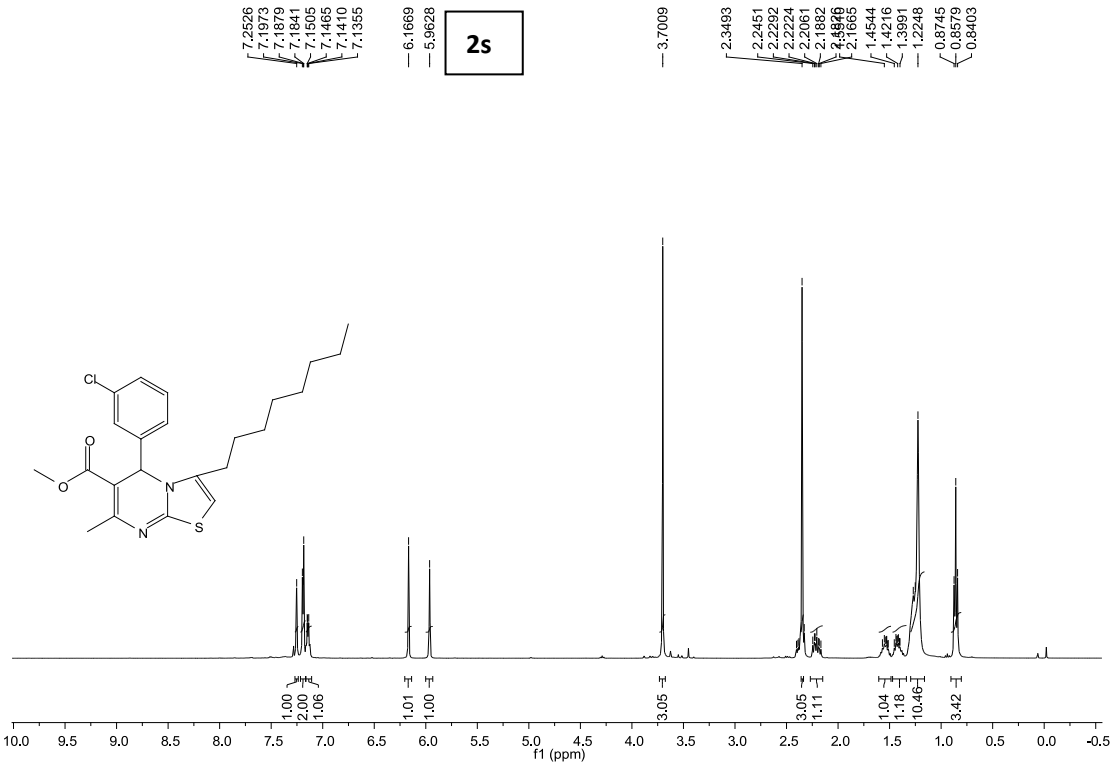
2q

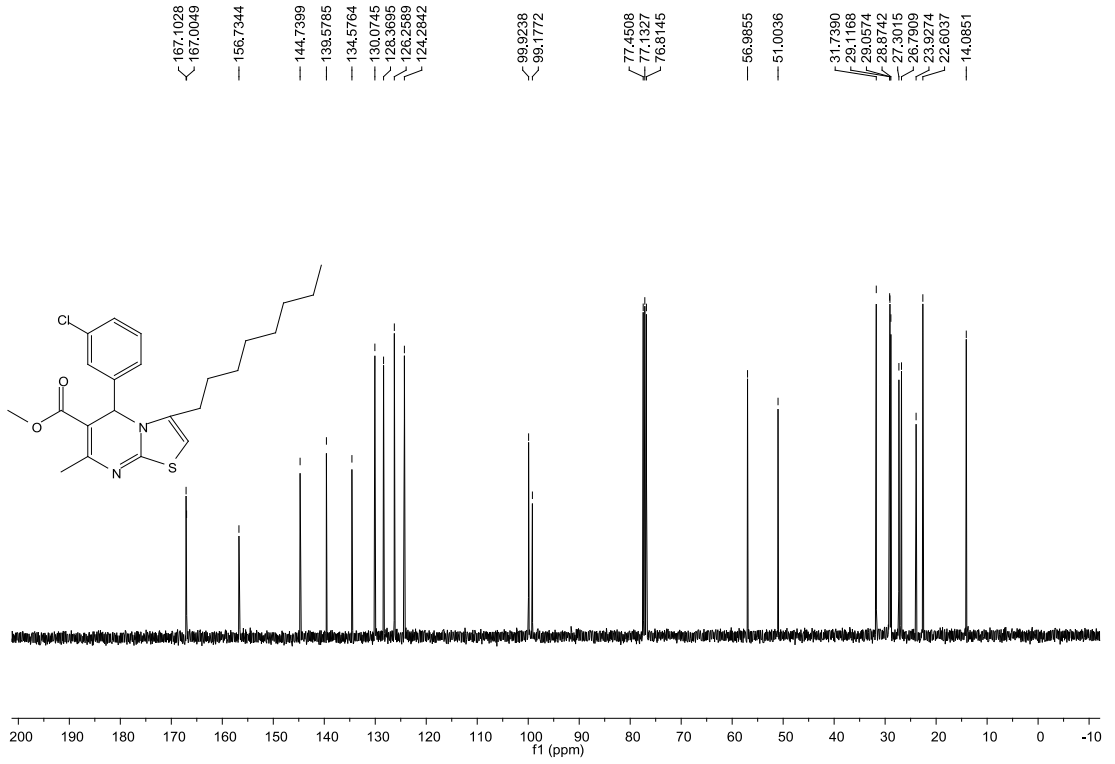

7.0814
7.0722
7.0621
7.0038
6.9838
6.9640
6.6672
6.6485
6.6137
3.5724
2.3931
Cl
F
O
O
N
N
S
5.00
1.11
2.02
1.11
1.02
3.01
3.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


166.8996
166.3014
164.6674
162.1706
156.3594
143.8933
138.3877
134.0418
130.0398
128.3285
125.4262
124.6127
116.0070
115.0495
103.4039
100.2844
57.6037
51.0266
29.6620
Cl
F
O
O
N
N
S
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.2819
7.2340
7.1762
6.2285
6.1912
5.9861
2r
3.7384
2.3806
2.2581
2.2414
2.2242
1.9633
1.8231
1.3927
1.3383
0.9266
0.9090
0.8917
Cl
O
O
N
N
S
3.25
1.00
1.01
1.00
3.00
3.25
2.03
4.39
3.01
f1 (ppm)


167.1946
156.7891
144.7384
144.7384
139.5677
134.6136
130.0945
128.4115
126.2986
124.3084
106.9048
99.8896
77.3512
77.0336
76.7160
57.0162
51.0584
32.7872
28.9022
27.0639
22.0488
13.6858
Cl
O
O
N
N
S
f1 (ppm)


2s
7.2526
7.1973
7.1879
7.1841
7.1505
7.1465
7.1410
7.1355
6.1669
5.9628
3.7009
2.3493
2.2451
2.2292
2.2224
2.2061
2.1882
2.1665
1.4544
1.4216
1.3991
1.2248
0.8745
0.8579
0.8403
Cl
O
O
N
N
S
1.00
2.00
1.06
1.01
1.00
3.05
3.05
1.11
1.04
1.18
10.46
3.42
f1 (ppm)


167.1028
167.0049
156.7344
144.7399
139.5785
134.5764
130.0745
128.3695
126.2589
124.2842
99.9238
99.1772
77.4508
77.1327
76.8145
56.9855
51.0036
31.7390
29.1168
29.0574
28.8742
27.3015
26.7909
23.9274
22.6037
14.0851
Cl
O
O
N
N
S
f1 (ppm)