Electronic Supplementary Material (ESI) for RSC Advances.

Efficient synthesis of polyfunctionalized thiophene-2,3-diones and thiophen-3(2*H*)-ones using β-oxodithioesters

Sridhar Madabhushi,*[,a] Srinivas Kurva,[a] Vinodkumar Sriramoju,[a] Jagadeesh Babu Nanubolu[b] and Suresh Reddy Cirandur[c]

Fluoroorganics Division, CSIR-Indian Institute of Chemical Technology, Hyderabad 500607, India
Tel.:(+91)-40-27191772; fax: (+91)-40-27160387

E- mail: smiict@gmail.com

Supporting Information

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Crystal data for 3b: $C_{12}H_9ClO_2S_2$, M = 284.76, block, 0.45 x 0.32 x 0.30 mm^3, monoclinic, space group $P2_1/c$ (No. 14), a = 12.2624(8), b = 13.4433(9), c = 7.9968(5) Å, β = 106.1680(10)º, V = 1266.11(14) Å^3, Z = 4, D_c = 1.494 g/cm^3, F_{000} = 584, CCD area detector, MoKα radiation, λ = 0.71073 Å, T = 293(2)K, $2\theta_{max}$ = 50.0º, 11867 reflections collected, 2225 unique (R_{int} = 0.0203), Final *GooF* = 1.054, $R1$ = 0.0354, $wR2$ = 0.0940, R indices based on 2099 reflections with I >2σ(I) (refinement on F^2), 155 parameters, μ = 0.616 mm^{-1}.

Crystal data for 2f: $C_{14}H_{12}O_3S_2$, M = 292.36, 0.48 x 0.21 x 0.08 mm^3, triclinic, space group $P\bar{1}$ (No. 2), a = 5.7321(15), b = 8.788(2), c = 13.769(4) Å, α = 99.381(5), β = 101.216(4), γ = 90.696(4)º, V = 670.6(3) Å^3, Z = 2, D_c = 1.448 g/cm^3, F_{000} = 304, CCD area detector, MoKα radiation, λ = 0.71073 Å, T = 293(2)K, $2\theta_{max}$ = 50.0º, 6310 reflections collected, 2349 unique (*R*int = 0.0395), Final *GooF* = 1.177, $R1$ = 0.0726, $wR2$ = 0.2068, R indices based on 2070 reflections with I >2σ(I) (refinement on F^2), 194 parameters, μ = 0.397 mm^{-1}.

CCDC 1402849 (3b) and CCDC 1402850 (2f) contain the supplementary crystallographic data for this paper which can be obtained free of charge at https://summary.ccdc.cam.ac.uk/structure-summary-form or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0) 1223 336 033; email: deposit@ccdc.cam.ac.uk.

Crystal structure determination: X-ray data for the compounds were collected at room temperature using a Bruker Smart Apex CCD diffractometer with graphite monochromated Mo-Kα radiation (λ=0.71073Å) with ω-scan method.[1] Preliminary lattice parameters and orientation matrices were obtained from four sets of frames. Unit cell dimensions were determined using 9189 reflections for **3b** and 2233 reflections for **2f** data sets. Integration and scaling of intensity data were accomplished using SAINT program.[1] The structures were solved by Direct Methods using SHELXS97[2] and refinement was carried out by full-matrix

least-squares technique using SHELXL97.[2] Anisotropic displacement parameters were included for all non-hydrogen atoms. All H atoms were positioned geometrically and treated as riding on their parent C atoms, with C-H distances of 0.93--0.97 Å, and with $U_{iso}(H)$ = $1.2U_{eq}$ (C) or $1.5U_{eq}$ for methyl atoms. Two atoms of ethyl group of **2f** were disordered over two sites with site occupancy factor of 0.77 for C1/C2 atoms (major component) and 0.23 for C1D/C2D atoms (minor component). The anisotropic displacement parameters of the disordered carbon atoms were restrained to be similar (SIMU instruction) and the direction of motion along the axis between the atoms was also restrained (DELU instruction).[3] The C-C bond distances were restrained to a set target value of 1.50-1.55 Å.

1. SMART & SAINT. Software Reference manuals. Versions 6.28a & 5.625, Bruker Analytical X-ray Systems Inc., Madison, Wisconsin, U.S.A., 2001.
2. Sheldrick, G. M. SHELXS97 and SHELXL97, Programs for crystal structure solution and refinement; University of Gottingen: Germany, 1997.
3. Muller, P, Herbst-Imer, R, Spek, A. L, Schneider, T. R, and Sawaya, M. R. Crystal Structure Refinement: A Crystallographer's Guide to SHELXL. Muller, P. Ed. 2006 Oxford University Press: Oxford, New York, pp. 57–91.

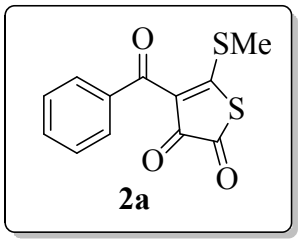
General Information: Melting points of all the compounds were recorded on Veego programmable melting point apparatus and are uncorrected. IR spectra were recorded on a PerkineElmer FT-IR 240-C spectrophotometer using KBr optics. ^{1}H NMR spectra were recorded on Bruker AV 300 MHz in $CDCl_3$ using TMS as internal standard. Electron Spray Ionization (ESI) and high-resolution spectra were recorded on QSTARXL hybrid MS/MS system (Applied Biosystems, USA) under electrospray ionization. All the reactions were monitored by thin layer chromatography (TLC) on precoated silica gel 60 F254 (mesh)

plates; spots were visualized with UV light. Merck silica gel (60-120 mesh) was used for column chromatography.

Typical procedure for synthesis of 4-benzoyl-5-(methylthio)thiophene-2,3-dione 2a: methyl 3-oxo-3-phenylpropanedithioate **1a** (500 mg, 2.37 mmol) was added to oxalylchloride (362 mg, 2.85 mmol) and reaction mixture was stirred at room temperature for 2 minutes. After completion of the reaction (monitored by TLC), the precipitate was washed with 3×5 ml of hexane to afford the pure 4-benzoyl-5-(methylthio)thiophene-2,3-dione **2a.**

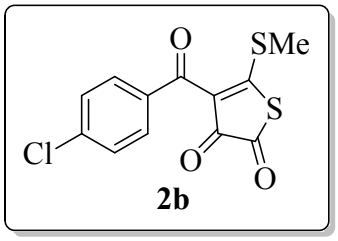
Typical procedure for synthesis of 4-benzoyl-5-(methylthio)thiophen-3(2*H*)-one 3a: Chloroacetic anhydride (485 mg, 2.85 mmol), methyl 3-oxo-3-phenylpropanedithioate **1a** (500 mg, 2.38 mmol), DMAP (145 mg, 1.18 mmol) and dichloromethane (10 mL) were taken in a 50 mL round bottom flask and the mixture was stirred at room temperature for 4 h. After completion of the reaction (monitored by TLC), the reaction mixture was extracted with dichloromethane (2×20 mL) and dried over anhyd. Na_2SO_4. Solvent was removed using rotavapor , and the crude residue obtained was purified by normal column chromatography (silica gel 60-120 mesh, ethylacetate/hexane gradient mixture as eluent to afford 4-benzoyl-5-(methylthio)thiophen-3(2*H*)-one **3a.**

Characterization data obtained for polyfunctionalized 4-benzoyl-5-(methylthio) thiophene-2,3-diones 2a-2h:



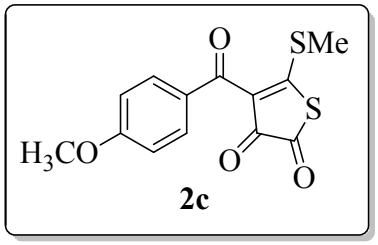
2a

4-Benzoyl-5-(methylthio)thiophene-2,3-dione (2a): Yield 96% (0.60 g), brown solid, m.p. 107-109 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.65 (d, *J* = 7.1 Hz, 2H), 7.56 (t, *J* = 7.4, 7.4 Hz, 1H), 7.43 (t, *J* = 7.7, 7.7 Hz, 2H), 2.68 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 190.7, 188.7, 180.8, 174.9, 136.7, 132.9, 129.1, 128.0, 123.2, 17.7; IR (KBr): υ 3439, 3058, 1742, 1685, 1620, 1404, 1294, 1229, 949 cm^{-1}; MS (ESI) 287 (M+Na). ESI-HRMS obtained for $C_{12}H_9O_3S_2$ (M+H) = 264.9983 (calculated: 264.9987).

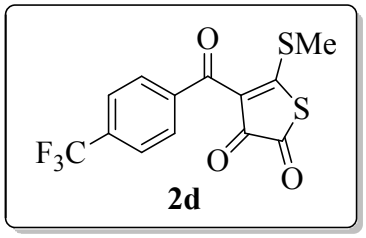


2b

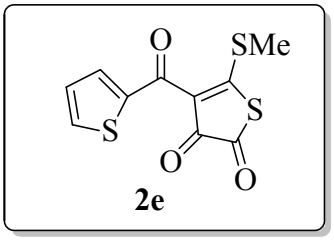
4-(4-Chlorobenzoyl)-5-(methylthio)thiophene-2,3-dione (2b): Yield 98 % (0.60 g), orange solid, m.p. 182 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.60 (d, *J* = 8.5 Hz, 2H), 7.4 (d, *J* = 8.5 Hz, 2H), 2.71 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 191.7, 187.4, 180.6, 174.9, 139.3, 135.1, 130.6, 128.4, 122.9, 17.9; IR (KBr): υ 3424, 3082, 2932, 1720, 1685, 1617, 1396, 1299, 1230, 970 cm^{-1}; MS (ESI) 321 (M+Na). ESI-HRMS obtained for $C_{12}H_7ClO_3NaS_2$ (M+Na) = 321.0154 (calculated: 321.0151).



2c **4-(4-Methoxybenzoyl)-5-(methylthio)thiophene-2,3-dione (2c):** Yield 96% (0.59 g), yellow solid, m.p. 148 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.70 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 3.87 (s, 3H), 2.68 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ =188.9, 187.0, 181.0, 175.1, 163.8, 131.9, 129.2, 124.1, 113.5, 55.4, 17.5; IR (KBr): υ 3458, 2983, 2838, 1748, 1680, 1594, 1424, 1304, 1245, 1177 cm^{-1}; MS (ESI) 317 (M+Na). ESI-HRMS obtained for $C_{13}H_{10}O_4NaS_2$ (M+Na) = 317.0357 (calculated: 317.0353).

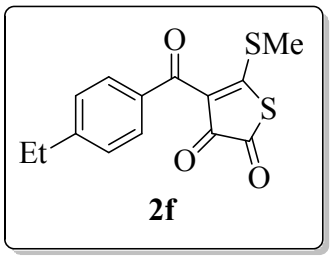


2d **5-(Methylthio)-4-(4-(trifluoromethyl)benzoyl)thiophene-2,3-dione (2d):** Yield 98% (0.58 g), yellow solid, m.p. 172-174 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.74-7.68 (q, *J* = 9.0, 9.8 Hz, 4H), 2.74 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 193.0, 187.7, 180.4, 174.8, 139.9, 133.6, 129.2, 126.6, 125.5, 125.07, 125.04, 124.9, 122.3, 122.2, 18.0; IR (KBr): υ 3458, 3354, 1739, 1688, 1620, 1414, 1322, 1134, 1065 cm^{-1}; MS (ESI) 355 (M+Na). ESI-HRMS obtained for $C_{13}H_7F_3O_3NaS_2$ (M+Na) = 355.0078 (calculated: 355.0082)



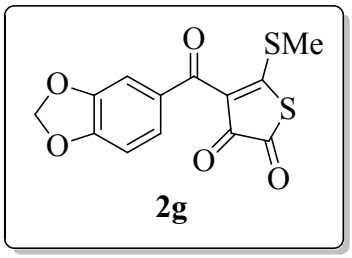
2e **5-(Methylthio)-4-(thiophene-2-carbonyl)thiophene-2,3-dione (2e):** Yield 97% (0.61 g), yellow solid, m.p. 135-137 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ =

7.87-7.86 (dd, *J* = 1.0, 3.9 Hz, 1H), 7.72-7.71 (dd, *J* = 1.0, 3.9 Hz, 1H), 7.15-7.13 (dd, *J* = 3.9, 3.9 Hz, 1H), 2.68 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 190.5, 180.6, 179.2, 174.6, 142.6, 135.0, 134.6, 128.1, 123.5, 18.0; IR (KBr): υ 3449, 2922, 1745, 1675, 1615, 1458, 1414, 1305, 1242, 723 cm^{-1}; MS (ESI) 293 (M+Na). ESI-HRMS obtained for $C_{10}H_6O_3NaS_3$ (M+Na) = 293.0177 (calculated: 293.0181).



2f

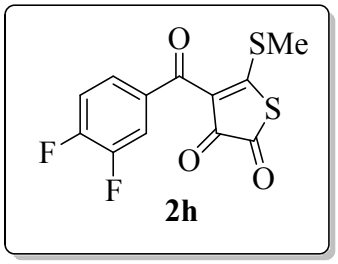
4-(4-Ethylbenzoyl)-5-(methylthio)thiophene-2,3-dione (2f): Yield 98 % (0.60 g), yellow solid, m.p. 143 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.61 (d, *J* = 8.0 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 2.73-2.69 (q, *J* = 7.6, 7.4 Hz, 2H), 2.68 (s, 3H), 1.26 (t, *J* = 7.6, 7.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 189.7, 188.3, 181.0, 175.0, 150.2, 134.2, 129.5, 127.6, 123.8, 28.9, 17.6, 15.0; IR (KBr): υ 3476, 2960, 2361, 1749, 1694, 1606, 1425, 1293, 1238, 950 cm^{-1}; MS (ESI) 315 (M+Na). ESI-HRMS obtained for $C_{14}H_{12}O_3NaS_2$ (M+Na) = 315.0229 (calculated: 315.0232).



2g

4-(Benzo[d][1,3]dioxole-5-carbonyl)-5-(methylthio)thiophene-2,3-dione (2g): Yield 98 % (0.59 g), brown solid, m.p. 166-168 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ =7.29-7.27 (dd, *J* = 1.8, 1.6 Hz, 1H), 7.21 (d, *J* = 1.5 Hz, 1H), 6.84 (d, *J* = 8.2 Hz, 1H), 6.05 (s, 2H), 2.69 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 188.9, 186.7, 180.8, 175.0, 152.2, 147.8, 130.9, 126.3, 124.1, 109.1, 107.7, 101.9, 17.6; IR (KBr): υ 3462, 3005, 2921,

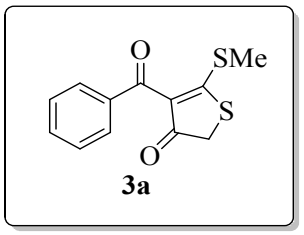
2621, 1741, 1665, 1425, 1259, 1030 cm^{-1}; MS (ESI) 331 (M+Na). ESI-HRMS obtained for $C_{13}H_8O_5NaS_2$ (M+Na) = 330.9915 (calculated: 330.9920).



2h

4-(3,4-Difluorobenzoyl)-5-(methylthio)thiophene-2,3-dione (2h): Yield 97 % (0.58 g), orange solid, m.p. 152 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.45-7.41 (dd, *J* = 1.5, 1.7 Hz, 1H), 7.38-7.34 (dd, *J* = 1.5, 1.5 Hz, 1H), 7.20 (t, *J* = 7.5, 7.9 Hz, 1H), 2.71 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 189.5, 185.5, 180.2, 174.3, 159.7, 159.0, 133.7, 133.7, 132.3, 132.1, 125.9, 124.8, 124.4, 124.3, 122.2, 114.5, 114.2, 112.7, 112.4, 16.8; IR (KBr): υ 3447, 2925, 2361, 1732, 1676, 1618, 1422, 1304, 1228, 960 cm^{-1}; MS (ESI) 323 (M+Na). ESI-HRMS obtained for $C_{12}H_6F_2O_3NaS_2$ (M+Na) = 323.0015 (calculated: 323.0011).

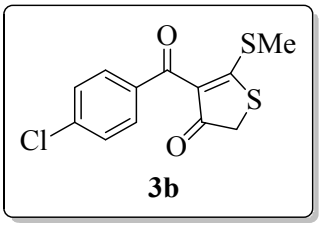
Characterization data obtained for polyfunctionalized 4-benzoyl-5-(methylthio) thiophen-3(2*H*)-ones 3a-3g:



3a

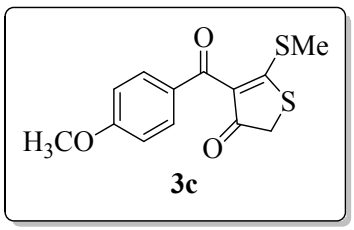
4-Benzoyl-5-(methylthio)thiophen-3(2*H*)-one (3a): Yield 71% (0.42 g), brown solid, m.p 138-140 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.67 (d, *J* = 7.3 Hz, 2H), 7.52 (t, *J* = 7.3, 7.2 Hz, 1H), 7.41 (t, *J* = 7.5, 7.3 Hz, 2H), 3.87 (s, 2H), 2.64 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 196.3, 193.6, 189.9, 137.6, 132.3, 129.0, 127.8, 125.9, 41.7,

16.2; IR (KBr): υ 3424, 3052, 2974, 2924, 1671, 1624, 1458, 1286, 1231 cm^{-1}; MS (ESI) 251 (M+H). ESI-HRMS obtained for $C_{12}H_{11}O_2S_2$ (M+H) = 251.0194 (calculated: 251.0195).



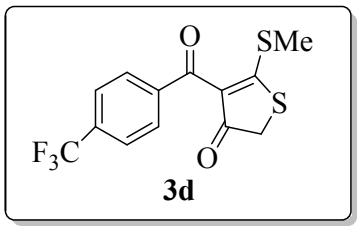
3b

4-(4-Chlorobenzoyl)-5-(methylthio)thiophen-3(2*H*)-one (3b): Yield 77% (0.45 g), brown solid, m.p. 116 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.61 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 8.5 Hz, 2H), 3.87 (s, 2H), 2.64 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 197.4, 193.5, 188.5, 138.4, 135.9, 130.4, 128.1, 125.4, 41.8, 16.2; IR (KBr): υ 3059, 2976, 2928, 1664, 1607, 1429, 1298, 1232, 1087 cm^{-1}; MS (ESI) 307 (M+Na). ESI-HRMS obtained for $C_{12}H_{10}O_2ClS_2$ (M+H) = 284.9807 (calculated: 284.9805).



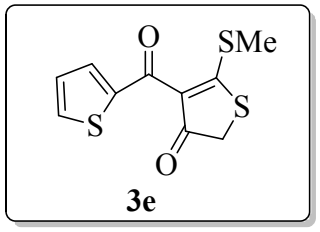
3c

4-(4-Methoxybenzoyl)-5-(methylthio)thiophen-3(2*H*)-one (**3c**): Yield 68% (0.4 g), brown solid, m.p. 178-180 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.72 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 3.87 (s, 2H), 3.85 (s, 3H), 2.62 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 194.4, 193.8, 188.4, 163.3, 131.7, 129.9, 126.6, 113.2, 55.3, 41.6, 16.0; IR (KBr): υ 3327, 2928, 2850, 1625, 1600, 1431, 1241, 1175, 1024 cm^{-1}; MS (ESI) 303 (M+Na). ESI-HRMS obtained for $C_{13}H_{12}O_3NaS_2$ (M+Na) = 303.0115 (calculated: 303.0120).



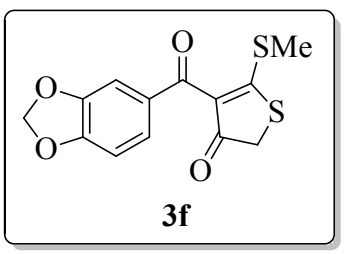
3d

5-(Methylthio)-4-(4-(trifluoromethyl)benzoyl)thiophen-3(2*H*)-one (3d): Yield 66% (0.38 g), pale yellow solid, m.p. 118-120 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.73-7.64 (q, *J* = 8.3, 8.3 Hz, 4H), 3.88 (s, 2H), 2.67 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 198.7, 193.4, 188.7, 140.9, 133.2, 133.0, 129.0, 128.1, 127.9, 125.9, 125.4, 124.9, 124.8, 124.7, 122.6, 41.9, 16.4; IR (KBr): υ 3314, 3063, 2979, 2931, 1668, 1614, 1435, 1329, 1112 cm^{-1}; MS (ESI) 341 (M+Na). ESI-HRMS obtained for $C_{13}H_9F_3O_2NaS_2$ (M+Na) = 341.0030 (calculated: 341.0031).



3e

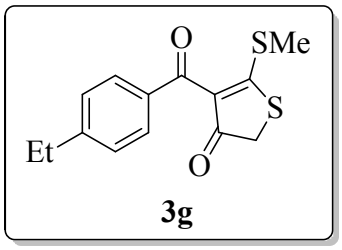
5-(Methylthio)-4-(thiophene-2-carbonyl)thiophen-3(2*H*)-one (3e): Yield 62% (0.37 g), brown solid, m.p. 181 ^{0}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.87 (d, *J* = 2.4 Hz, 1H), 7.65 (d, *J* = 4.8 Hz, 1H), 7.10 (t, *J* = 4.8, 3.9 Hz, 1H) 3.90 (s, 2H), 2.62 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 195.9, 193.4, 180.5, 153.0, 143.3, 134.1, 127.7, 126.3, 41.7, 16.3; IR (KBr): υ 3295, 2937, 2857, 1710, 1658, 1419, 1231, 1054 cm^{-1}; MS (ESI) 279 (M+Na). ESI-HRMS obtained for $C_{10}H_8O_2NaS_3$ (M+Na) = 278.9985 (calculated: 278.9987).



3f

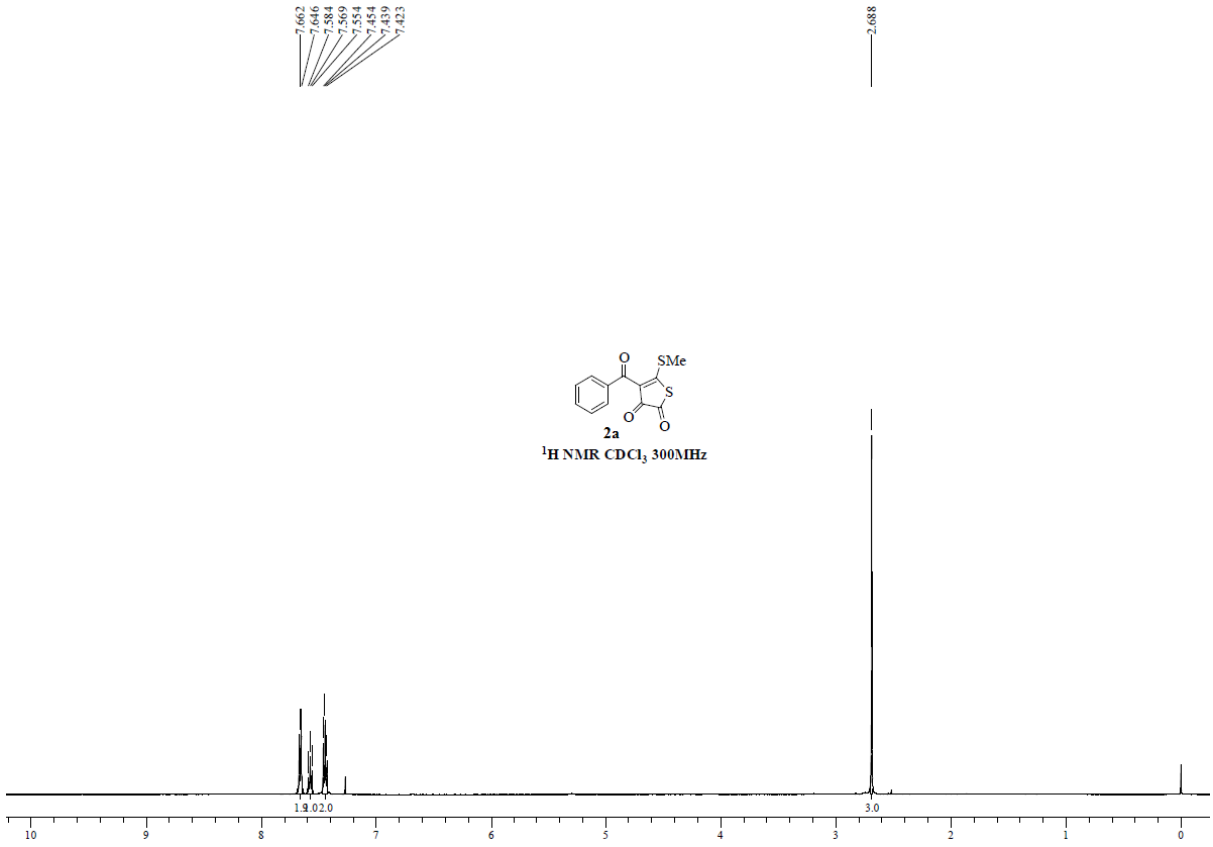
4-(Benzo[d][1,3]dioxole-5-carbonyl)-5-(methylthio)thiophen-3(2*H*)-one (3f): Yield 61% (0.35 g), pale yellow solid, m.p. 198 ^{0}C; ^{1}H NMR (300 MHz,

$CDCl_3$+DMSO): δ =7.31 (d, *J* = 8.1 Hz, 1H), 7.19 (s, 1H), 6.82 (d, *J* = 8.1 Hz, 1H), 6.05 (s, 2H), 3.92 (s, 2H), 2.65 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$+DMSO): δ = 197.6, 193.2, 187.3, 156.7, 150.8, 146.8, 130.9, 125.0, 108.2, 106.8, 101.0, 41.0, 15.2; IR (KBr): υ 3327, 2929, 2851, 1625, 1626, 1575, 1447, 1250, 1035 cm^{-1}; MS (ESI) 307 (M+Na). ESI-HRMS obtained for $C_{13}H_{10}O_4NaS_2$ (M+Na) = 307.0174(calculated: 307.0177).



3g

4-(4-Ethylbenzoyl)-5-(methylthio)thiophen-3(2*H*)-one (3g): Yield 72% (0.42 g), pale pink solid; m.p. 124-126 ^0C ^1H NMR (300 MHz, $CDCl_3$): δ = 7.63 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 3.87 (s, 2H), 2.71-2.67 (q, *J* = 7.6, 7.6 Hz, 2H), 2.63 (s, 3H), 1.25 (t, *J* = 7.6, 7.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 195.2, 193.7, 189.5, 149.3, 134.9, 129.4, 127.4, 126.3, 41.7, 28.9, 16.0, 15.0; IR (KBr): υ 3312, 2929, 2363, 1656, 1605, 1427, 1235, 846 cm^{-1}; MS (ESI) 301 (M+Na). ESI-HRMS obtained for $C_{14}H_{15}O_2S_2$ (M+H) = 279.0505 (calculated: 279.0508).

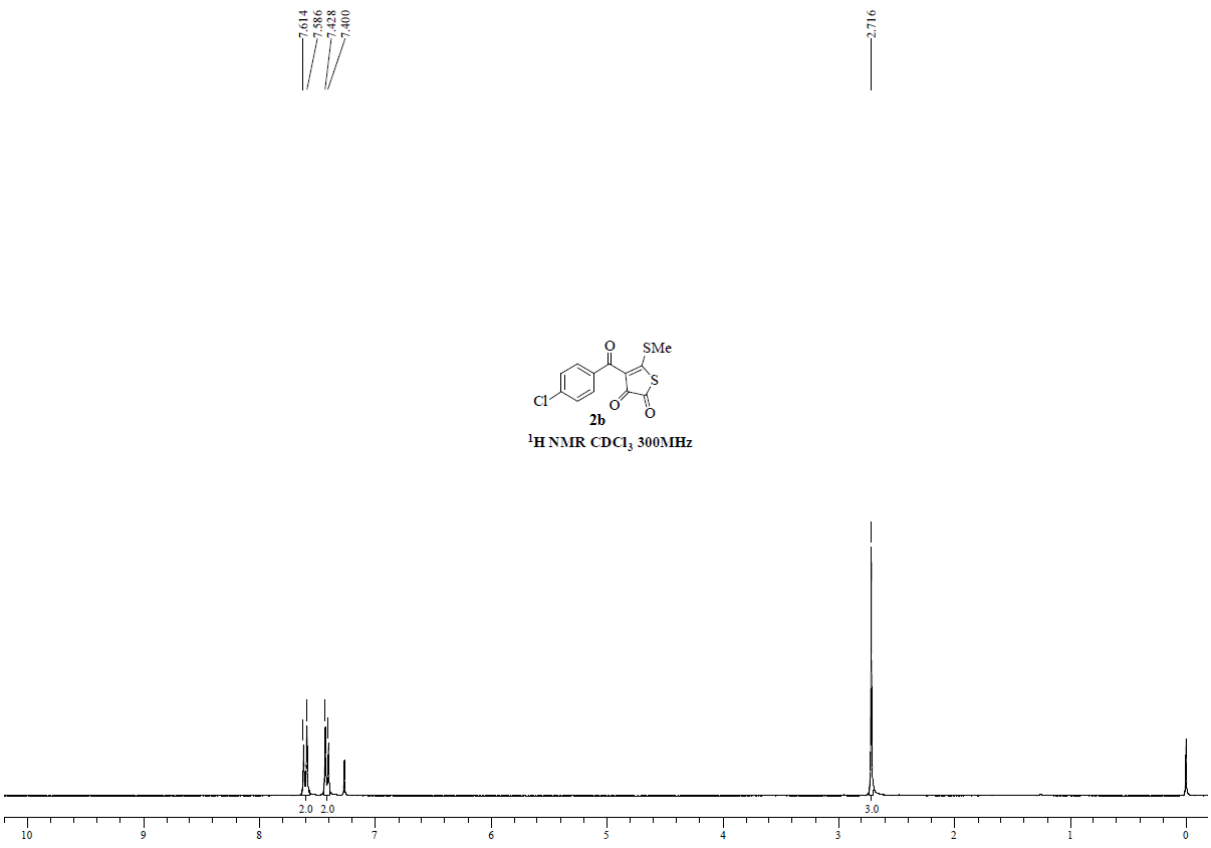


2a

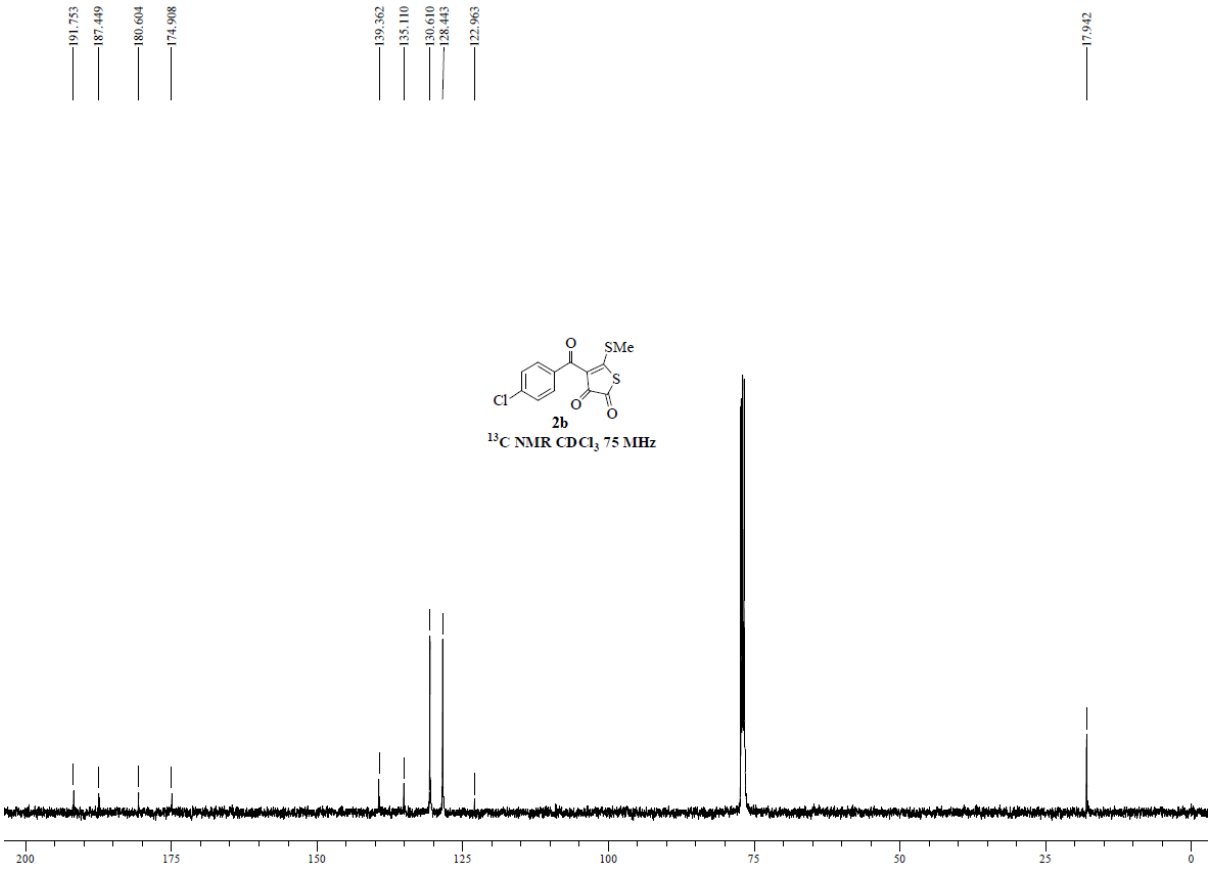
^{1}H NMR CDCl$_3$ 300MHz

2a

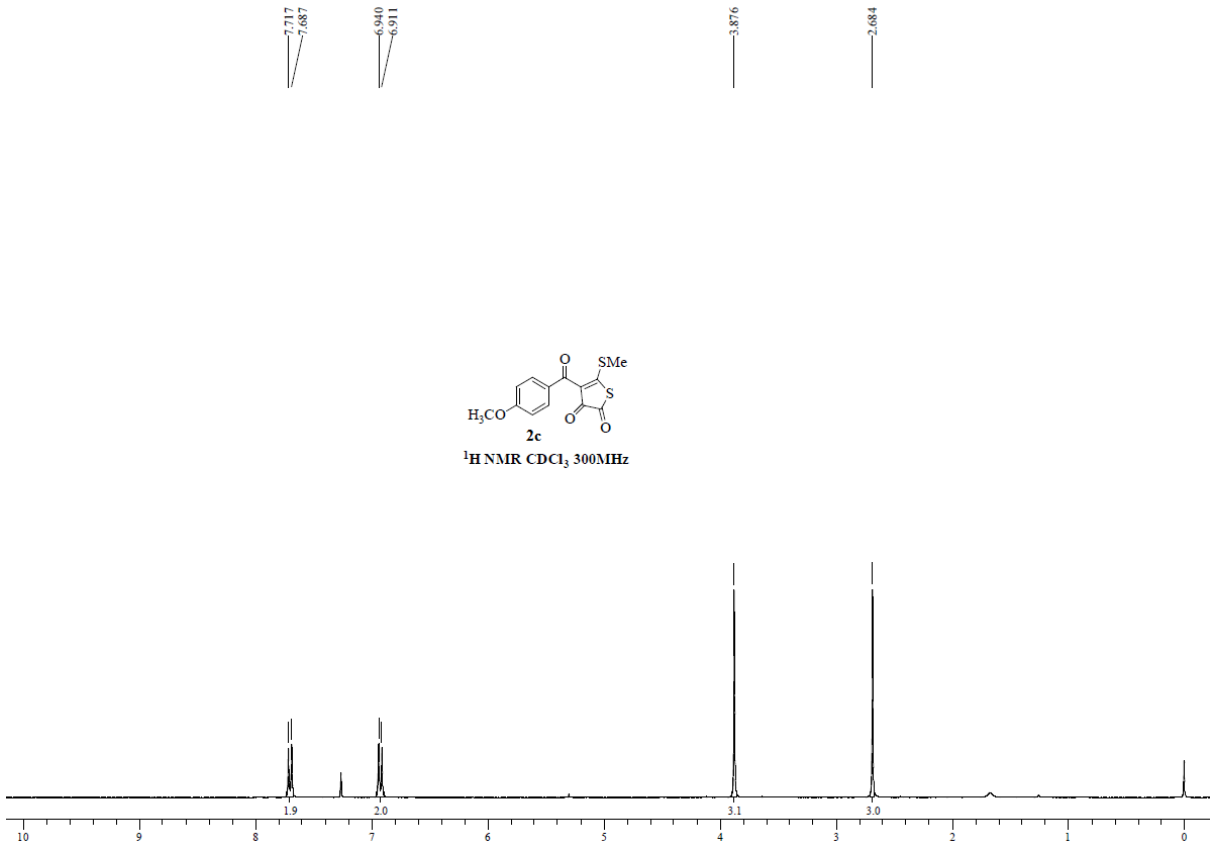
^{13}C NMR CDCl$_3$ 75 MHz



^{1}H NMR $CDCl_3$ 300MHz

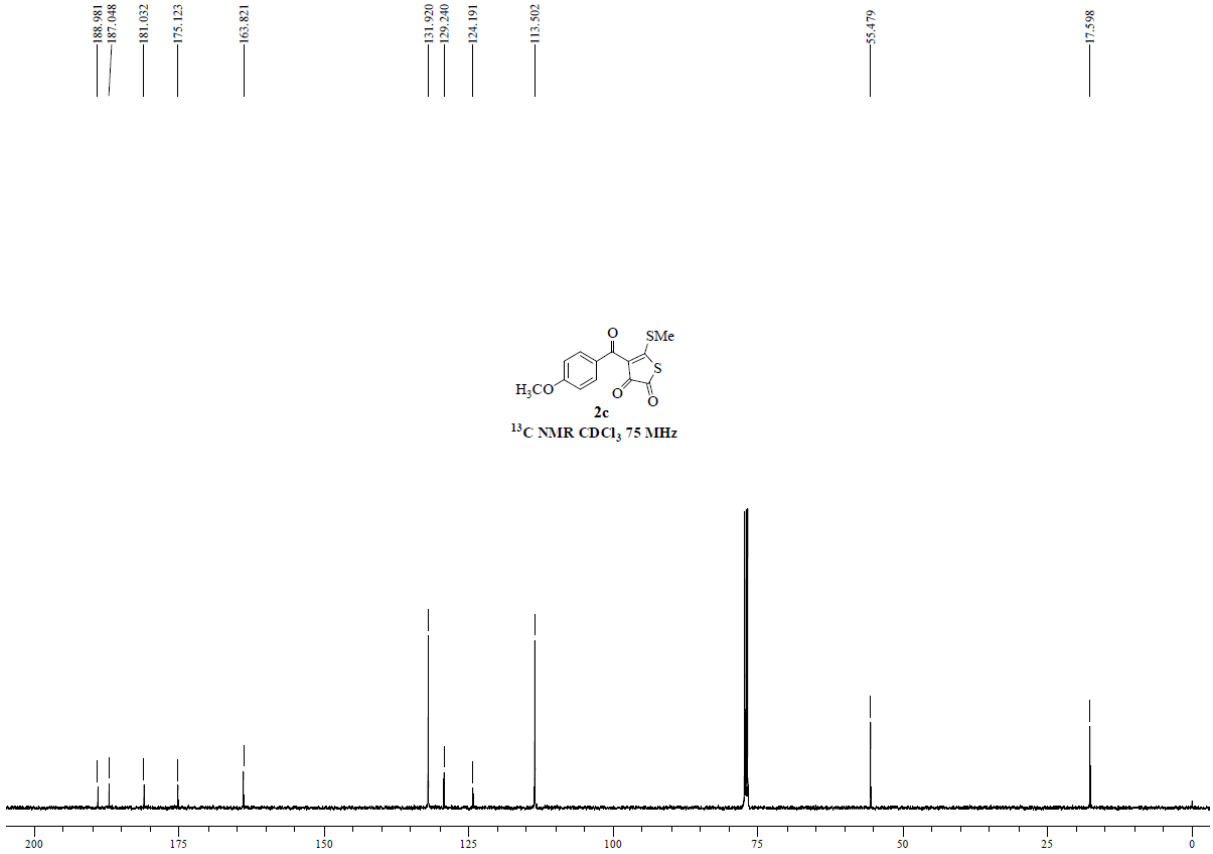


^{13}C NMR $CDCl_3$ 75 MHz



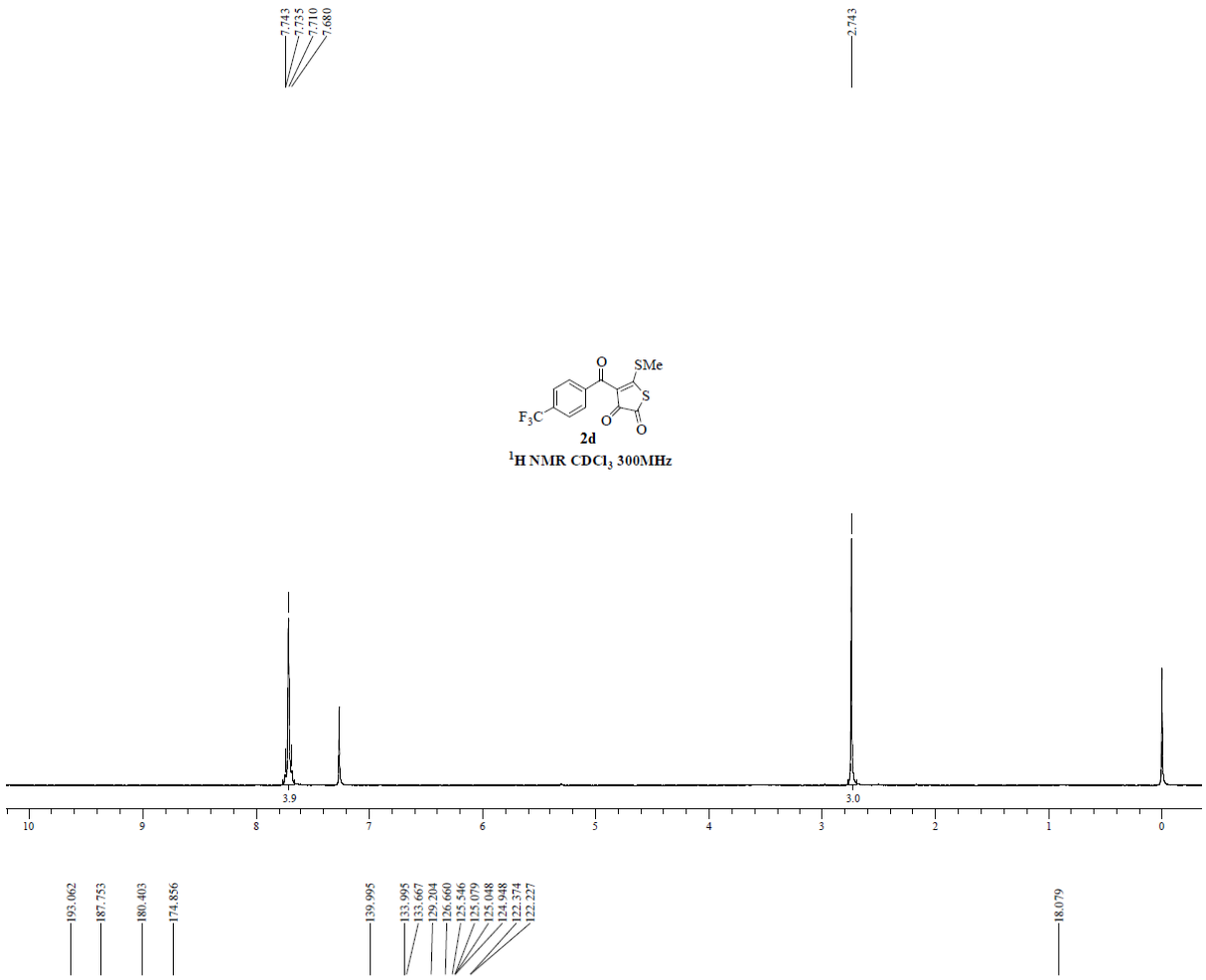
2c

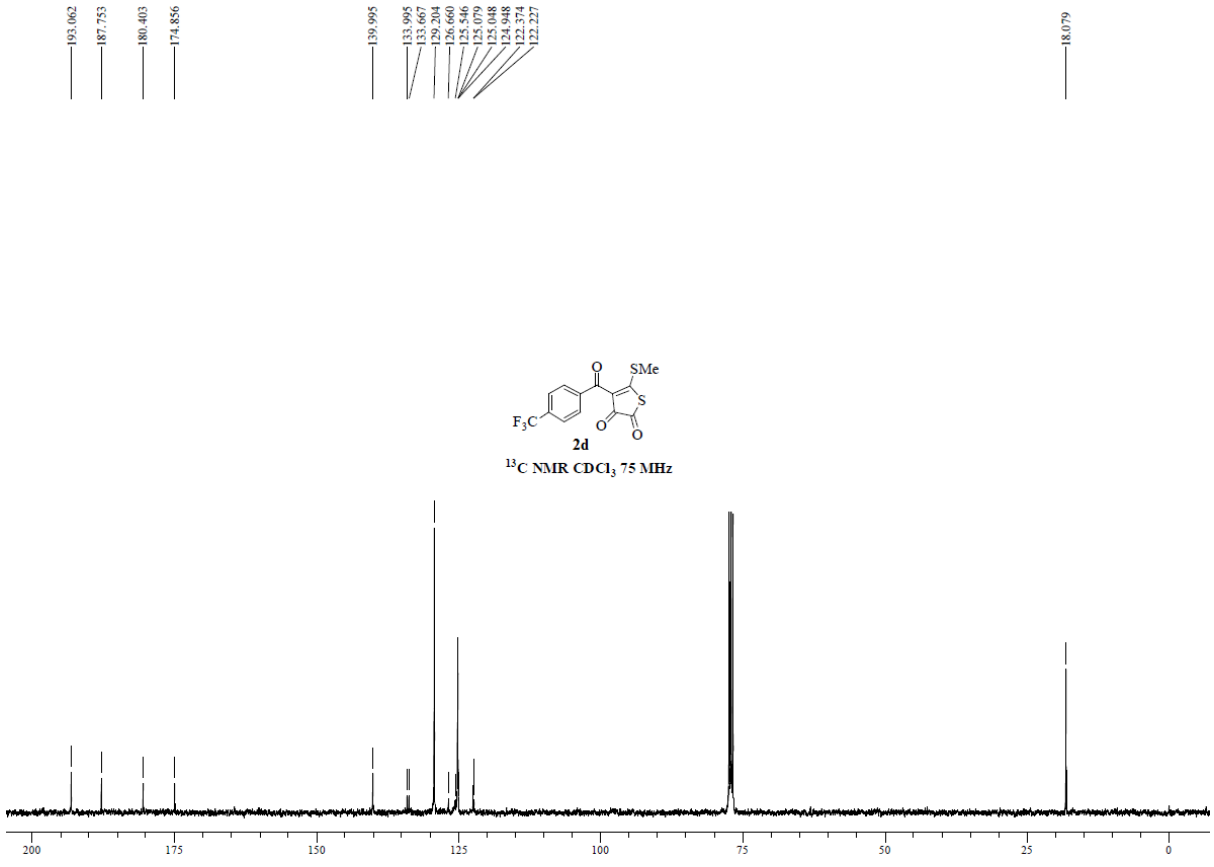
^{1}H NMR $CDCl_3$ 300MHz

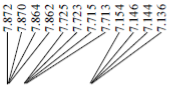


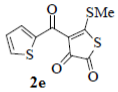
2c

^{13}C NMR $CDCl_3$ 75 MHz


7.743
7.735
7.710
7.680
2.743
SMe
O
S
O
O
F3C
2d
1H NMR CDCl3 300MHz
3.9
3.0
10
9
8
7
6
5
4
3
2
1
0

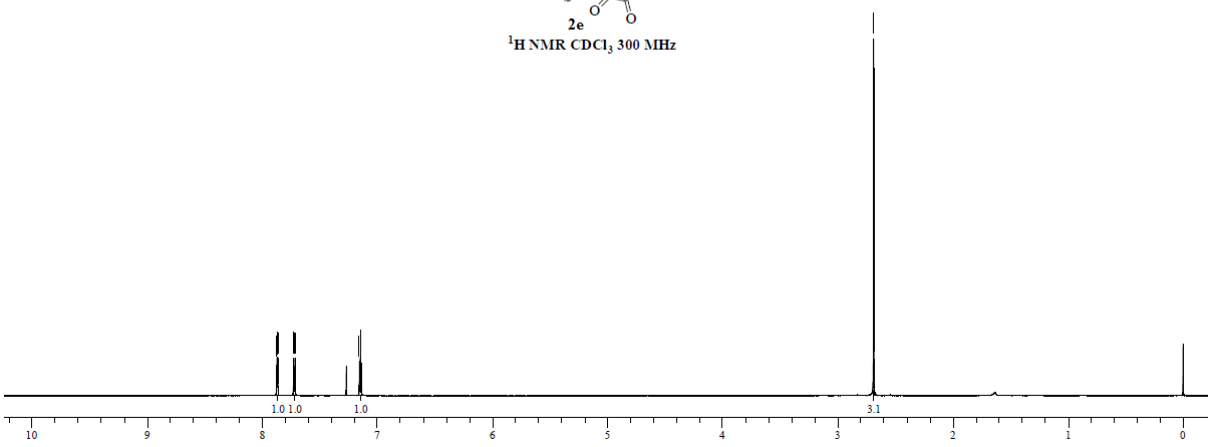

193.062
187.753
180.403
174.856
139.995
133.995
133.667
129.204
126.660
125.546
125.079
125.048
124.948
122.374
122.227
18.079
SMe
O
S
O
O
F3C
2d
13C NMR CDCl3 75 MHz
200
175
150
125
100
75
50
25
0

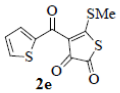




2e

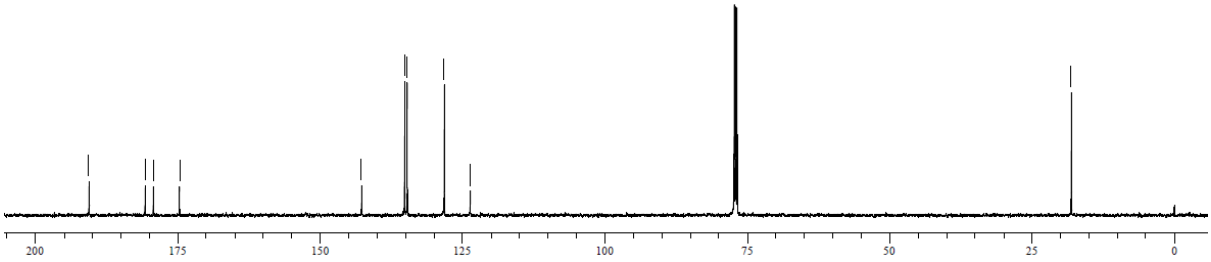
^{1}H NMR $CDCl_3$ 300 MHz

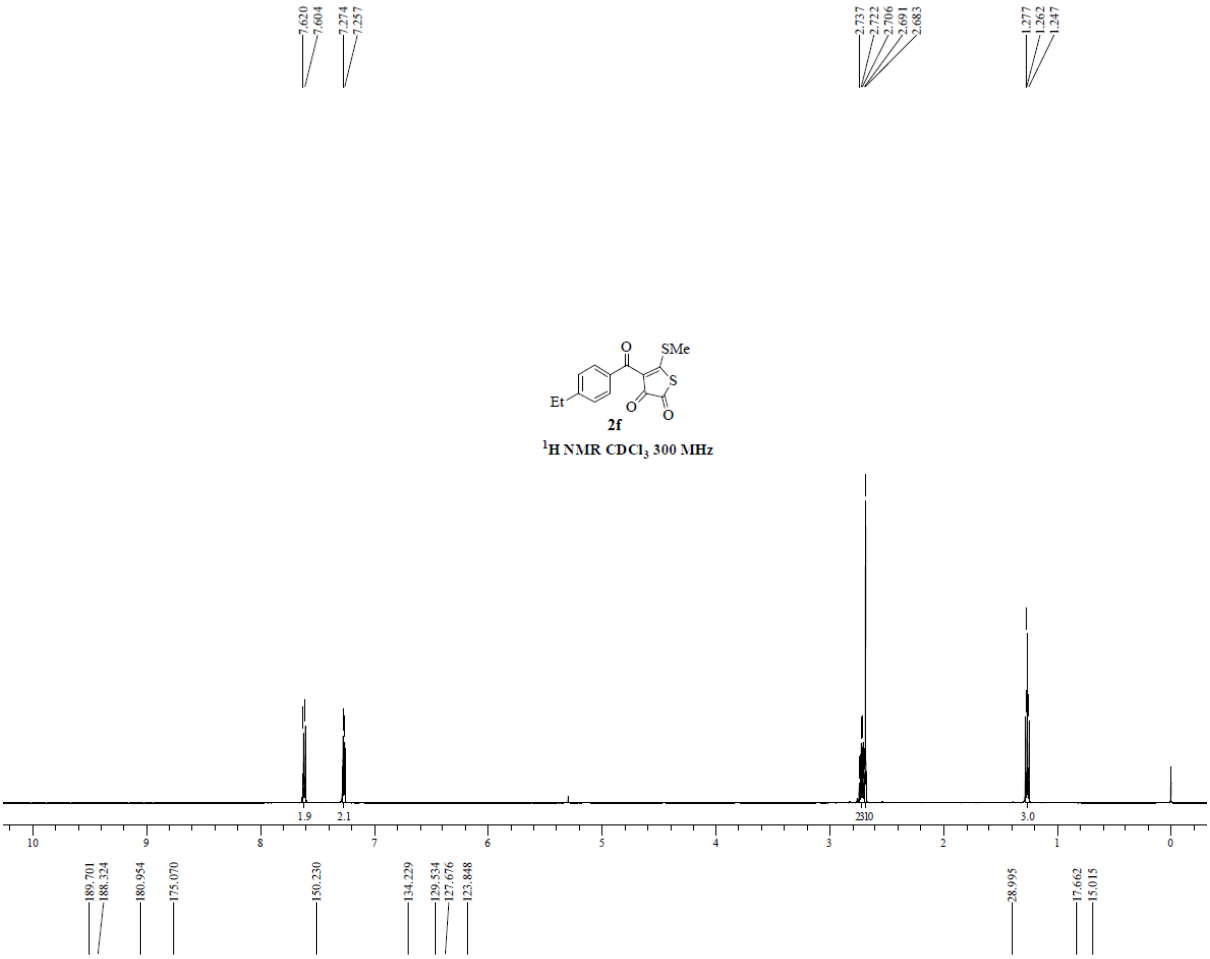


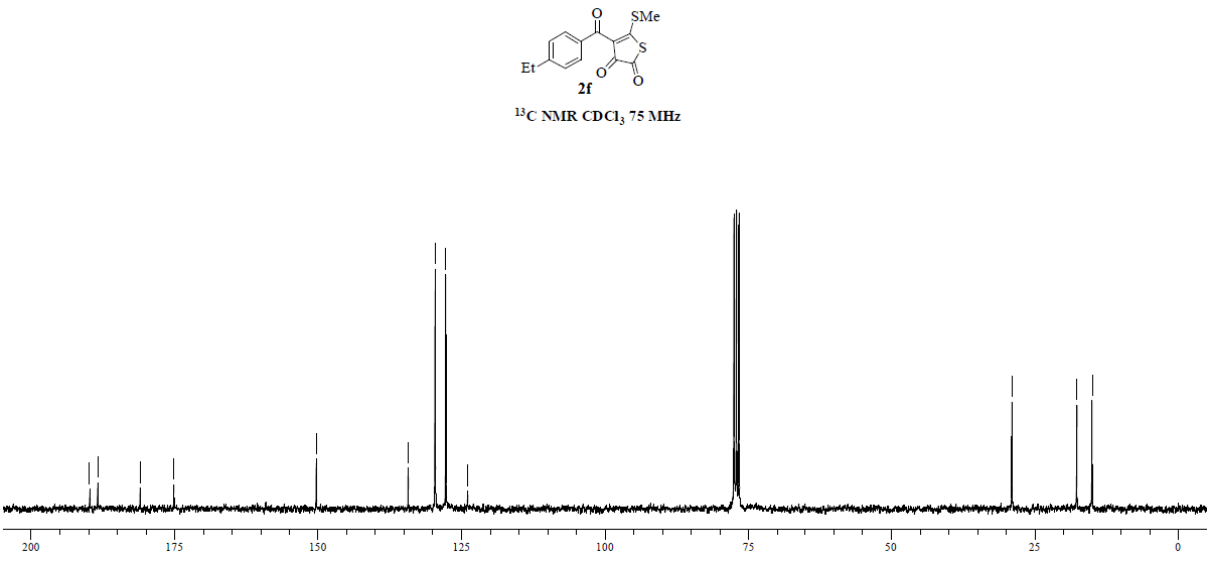


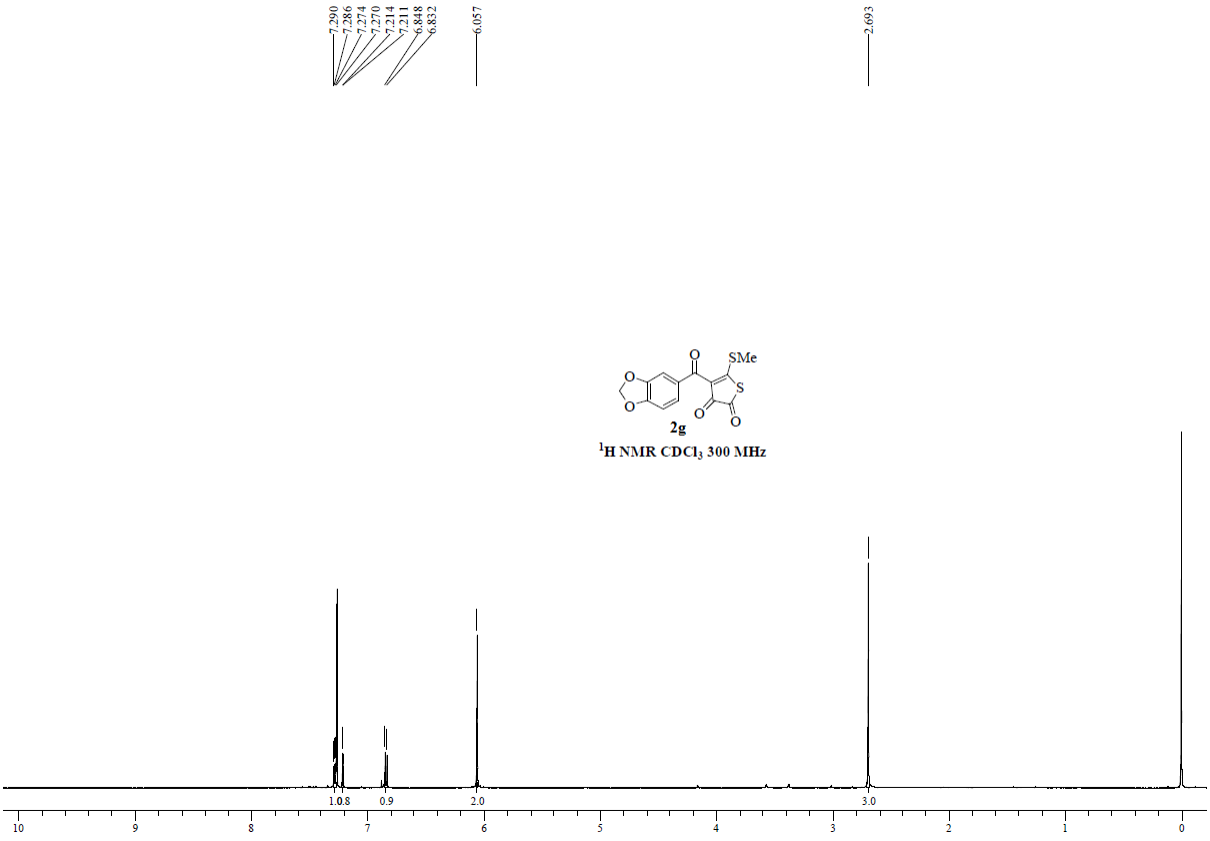
2e

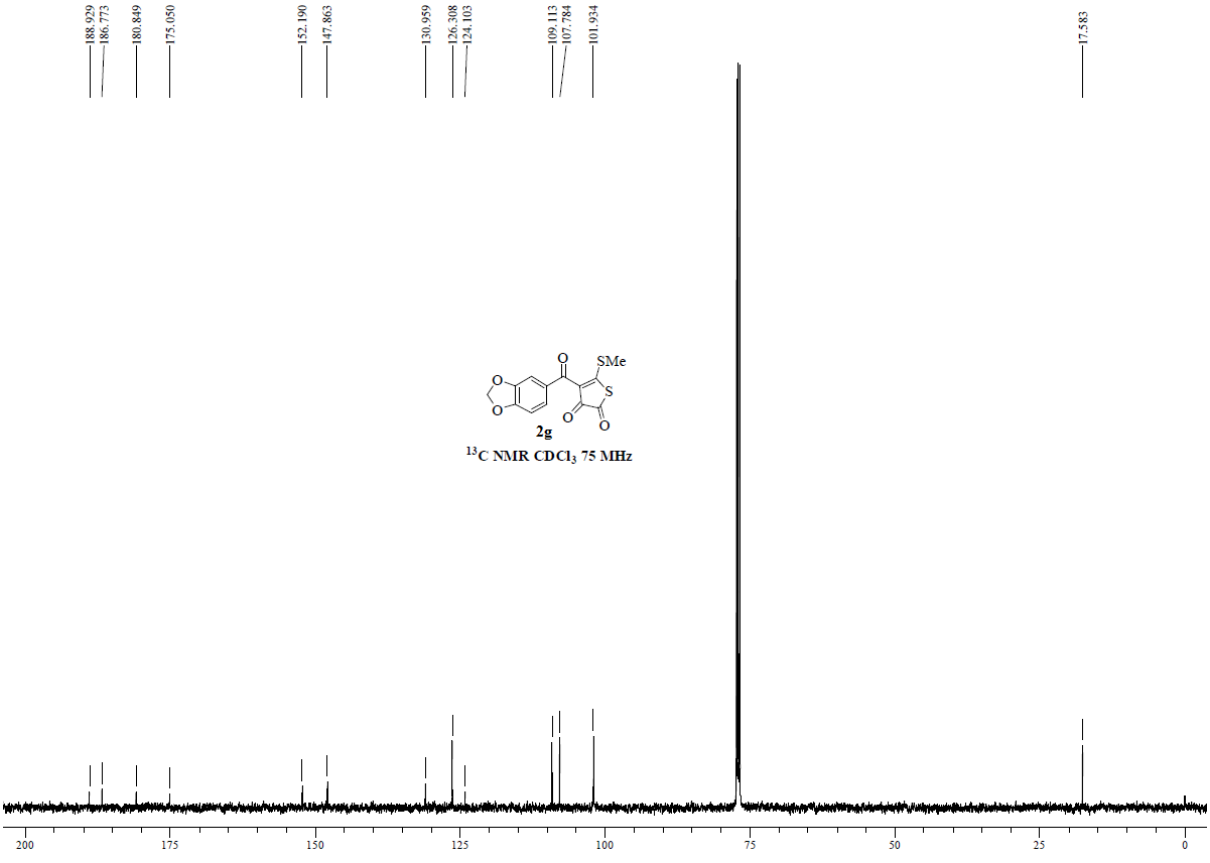
^{13}C NMR $CDCl_3$ 75 MHz

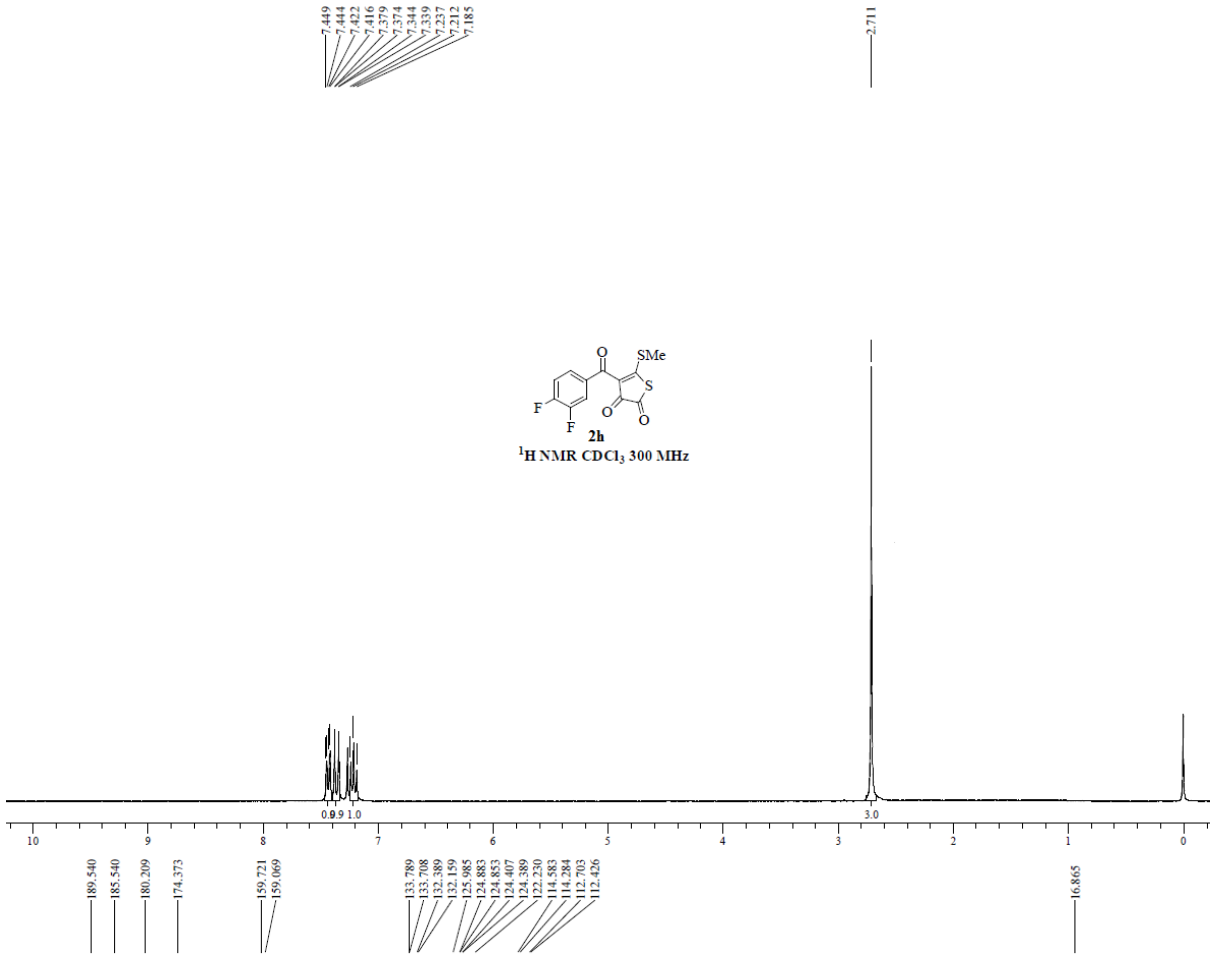



7.620
7.604
7.274
7.257
2.737
2.722
2.706
2.691
2.683
1.277
1.262
1.247
SMe
S
Et
O
O
O
2f
1H NMR CDCl3 300 MHz
1.9
2.1
2.0
3.0
10
9
8
7
6
5
4
3
2
1
0
189.701
188.324
180.954
175.070
150.230
134.229
129.534
127.676
123.848
28.995
17.662
15.015

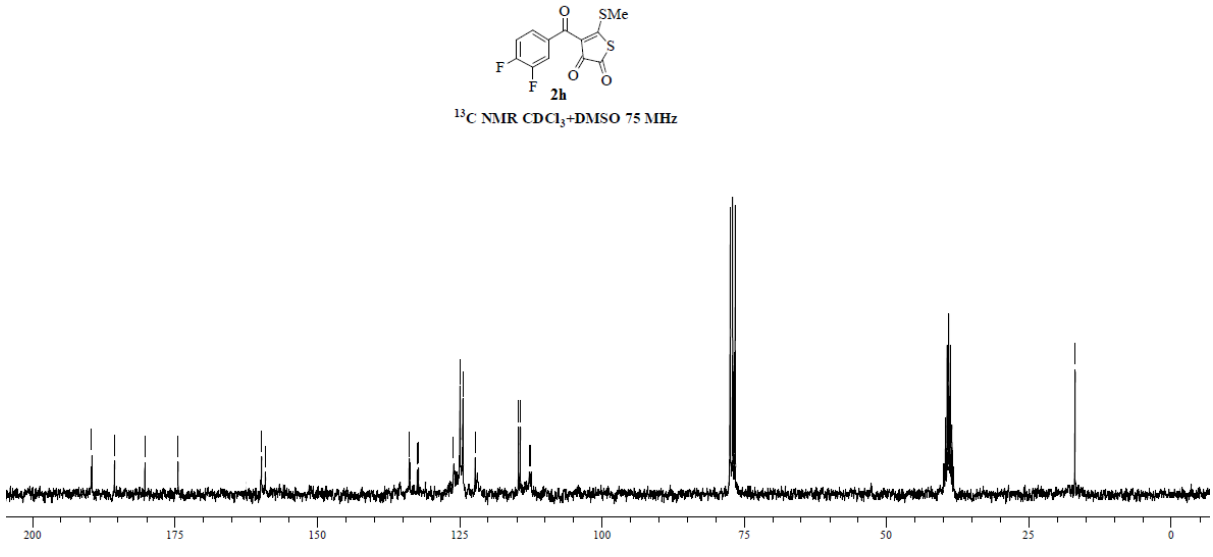

SMe
S
Et
O
O
O
2f
13C NMR CDCl3 75 MHz
200
175
150
125
100
75
50
25
0


7.290
7.286
7.274
7.270
7.214
7.211
6.848
6.832
6.057
2.693
SMe
O
S
O
O
O
O
2g
1H NMR CDCl3 300 MHz
1.0
0.8
0.9
2.0
3.0
10
9
8
7
6
5
4
3
2
1
0

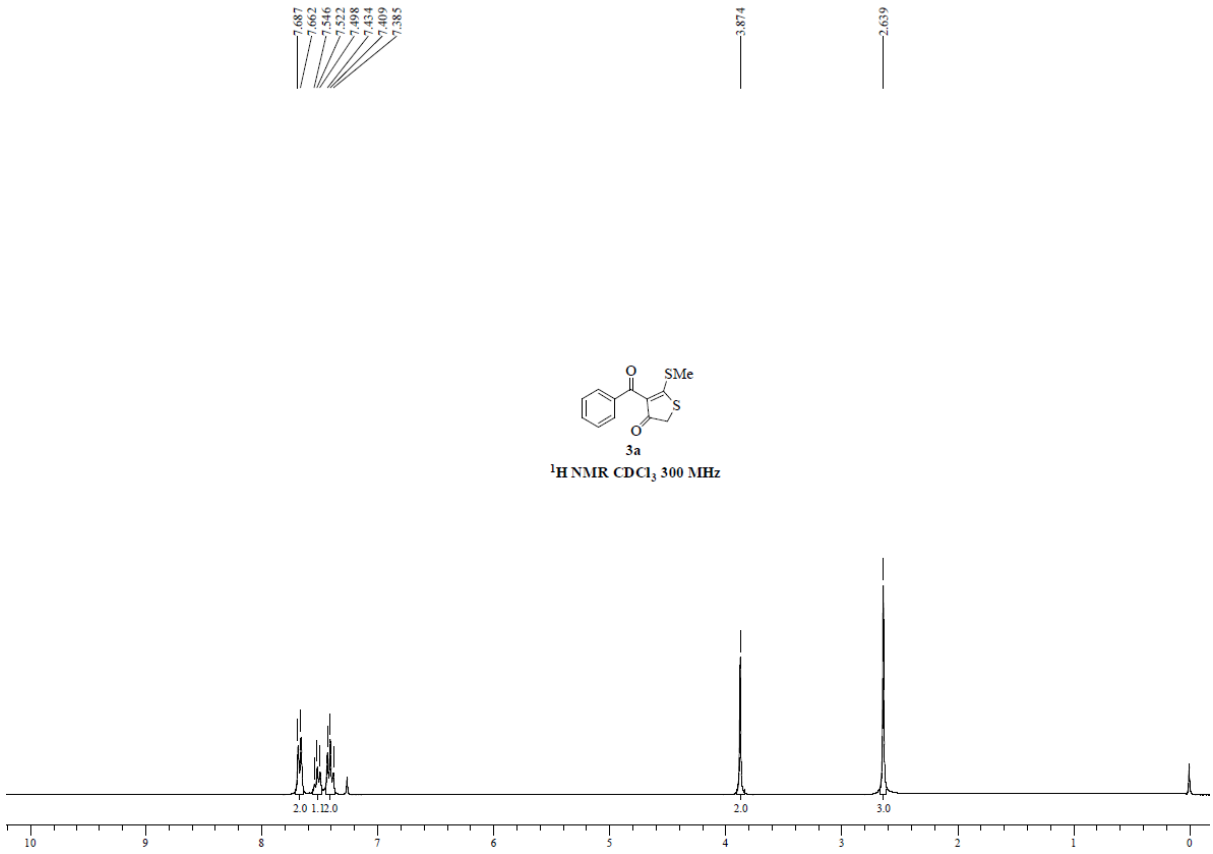

188.929
186.773
180.849
175.050
152.190
147.863
130.959
126.308
124.103
109.113
107.784
101.934
17.583
SMe
O
S
O
O
O
O
2g
13C NMR CDCl3 75 MHz
200
175
150
125
100
75
50
25
0



^{1}H NMR $CDCl_3$ 300 MHz

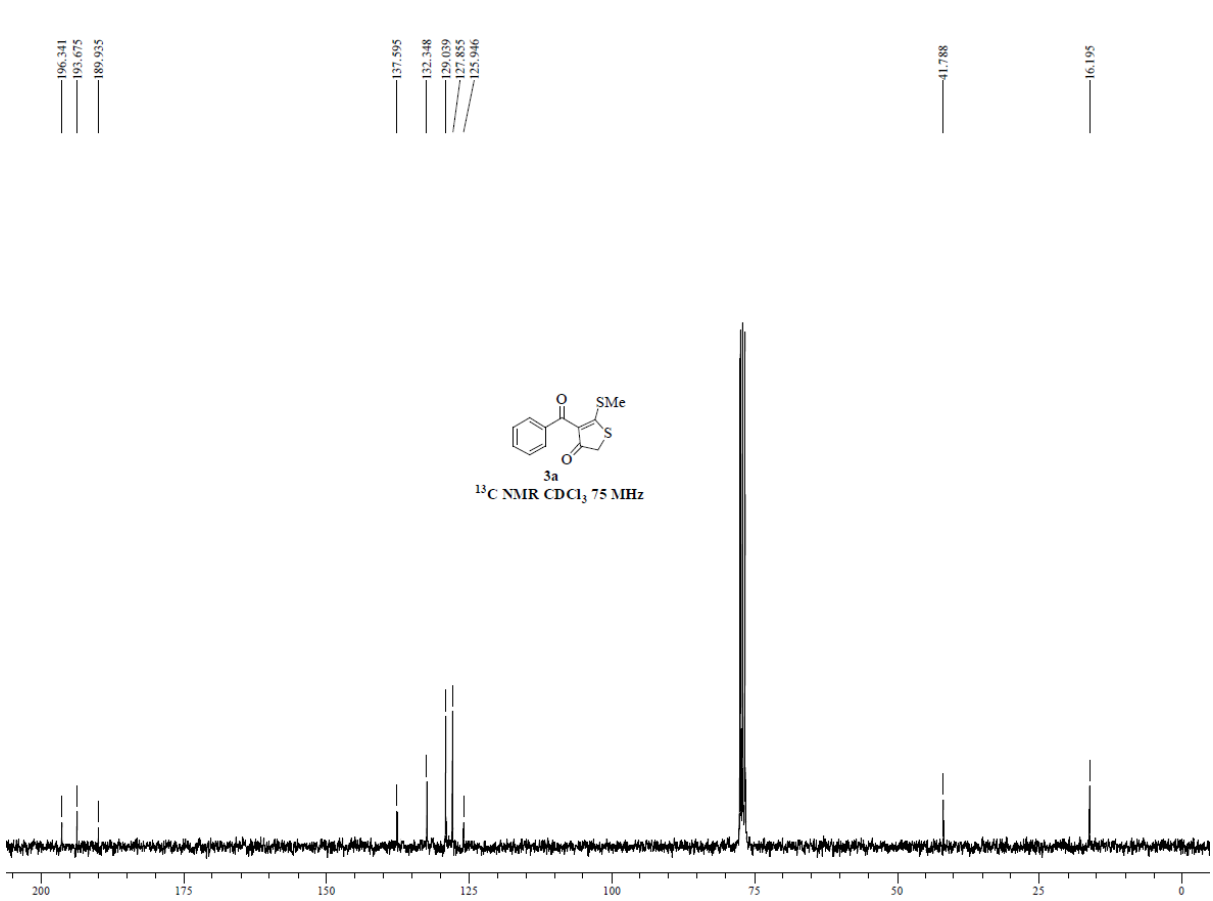


^{13}C NMR $CDCl_3$+DMSO 75 MHz



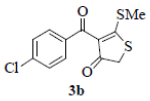
3a

^{1}H NMR $CDCl_3$ 300 MHz



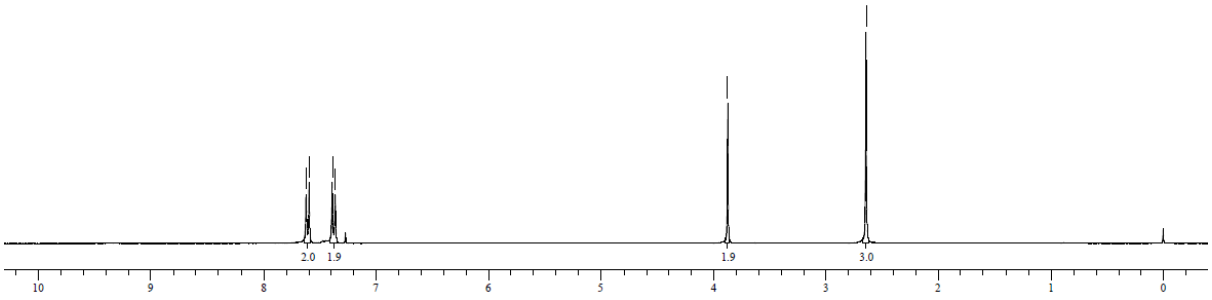
3a

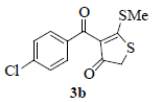
^{13}C NMR $CDCl_3$ 75 MHz



3b

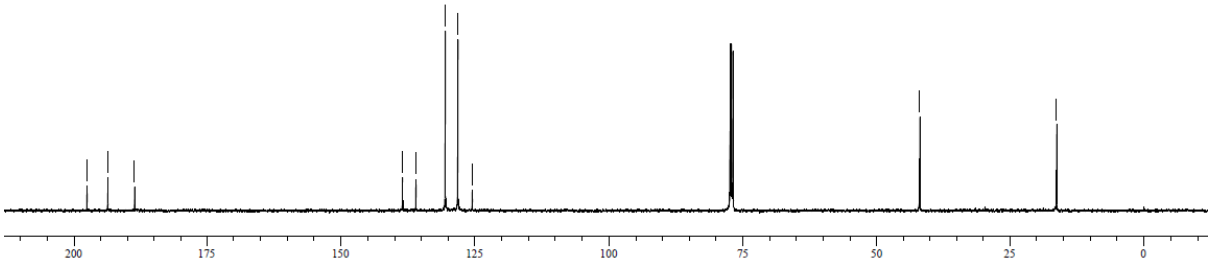
^{1}H NMR $CDCl_3$ 300 MHz

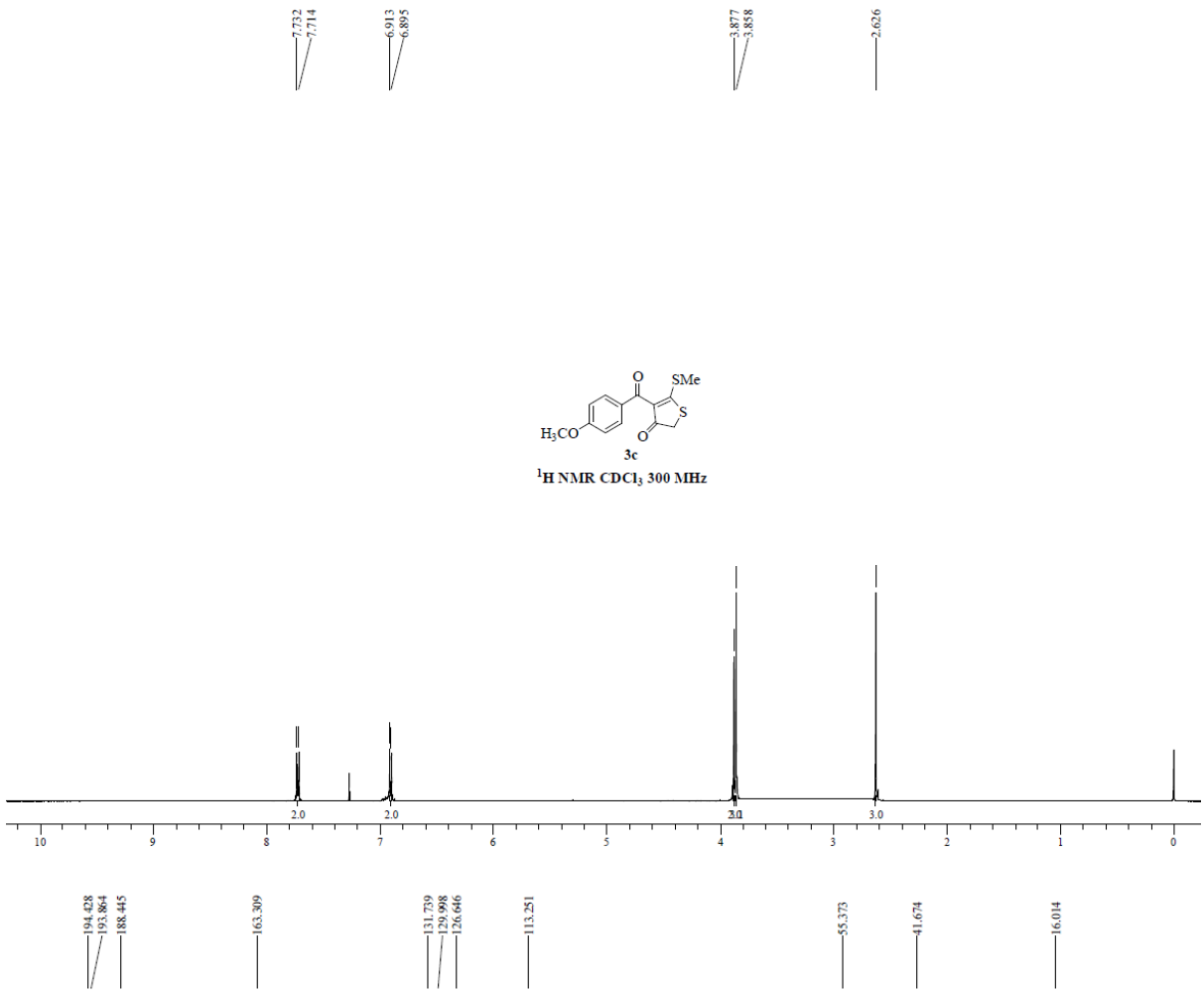




3b

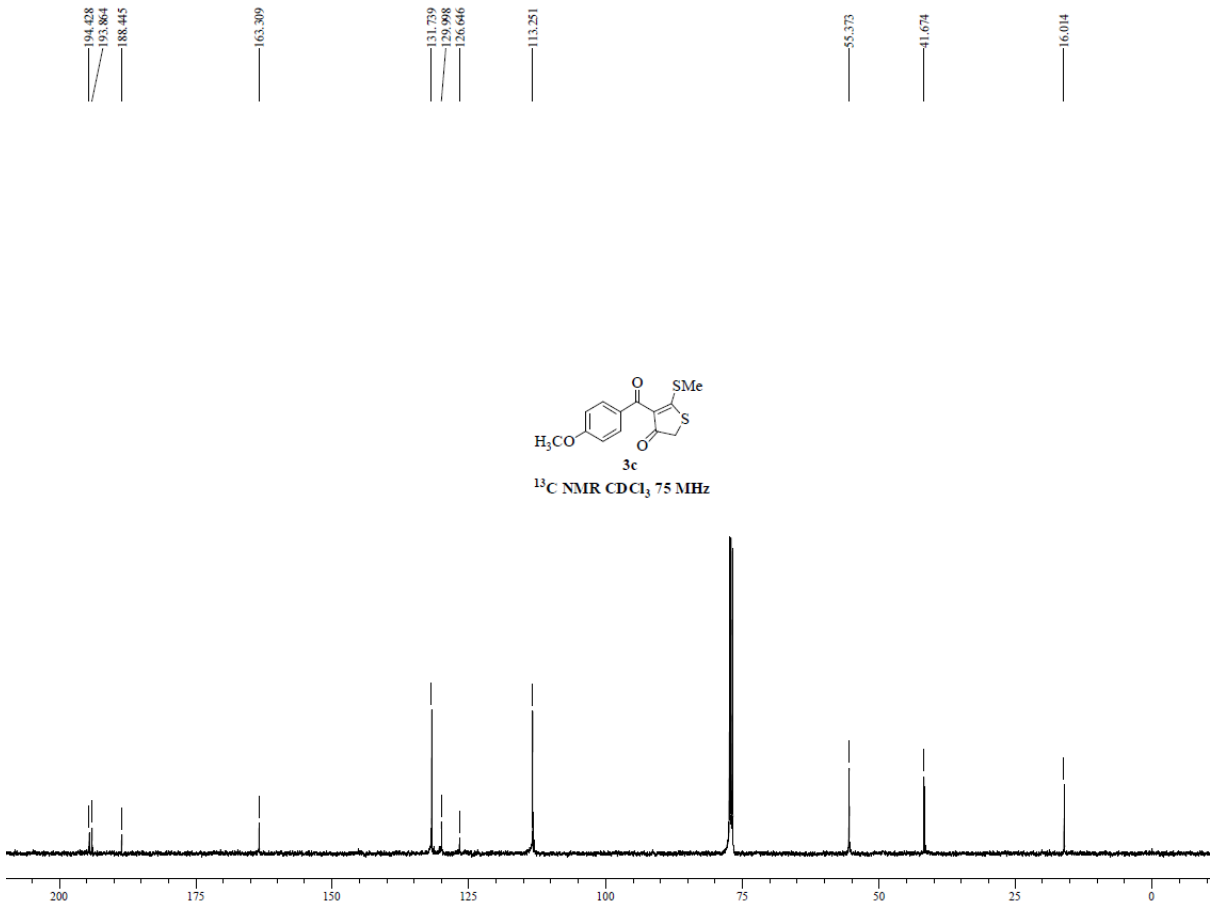
^{13}C NMR $CDCl_3$ 75 MHz





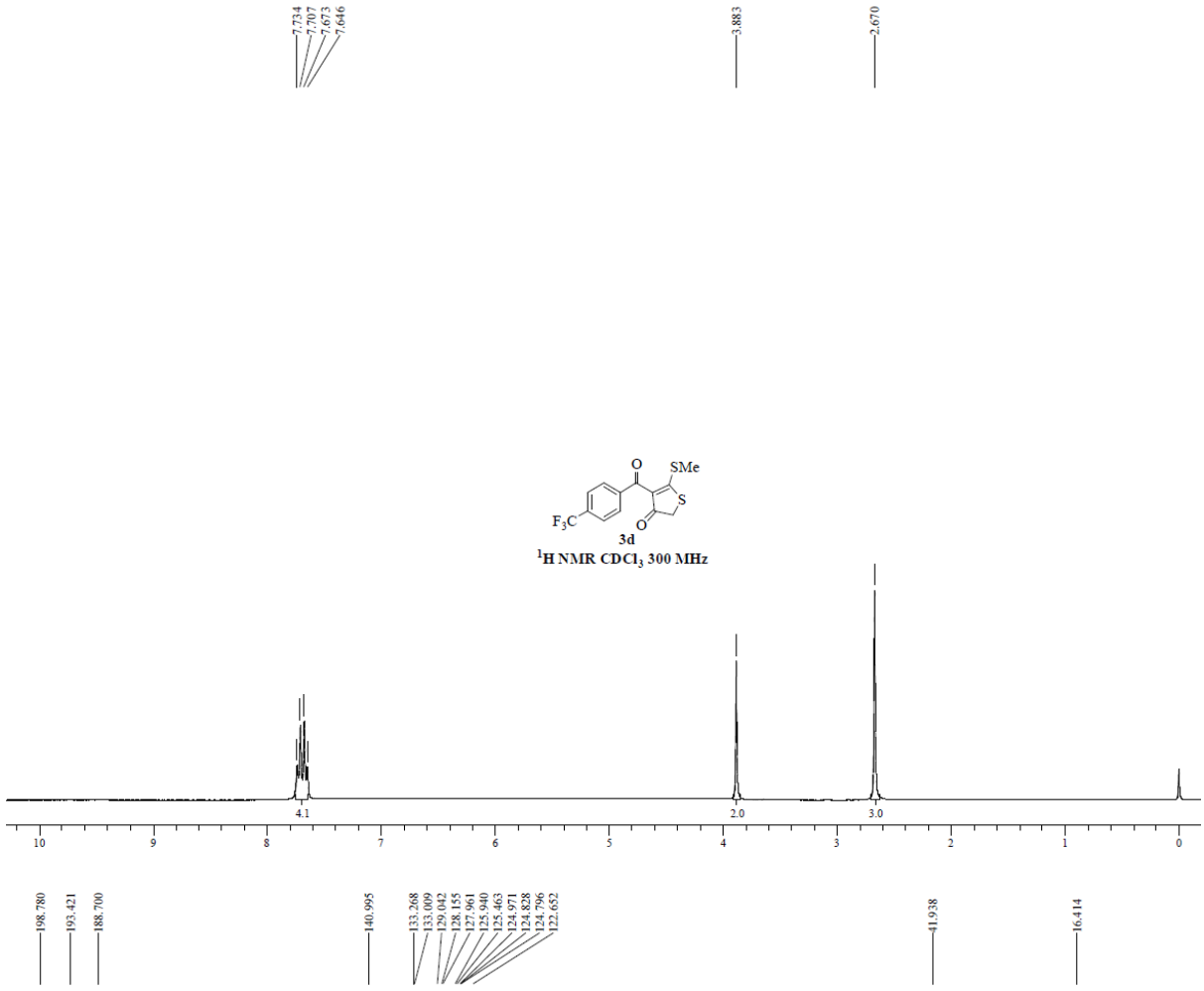
3c

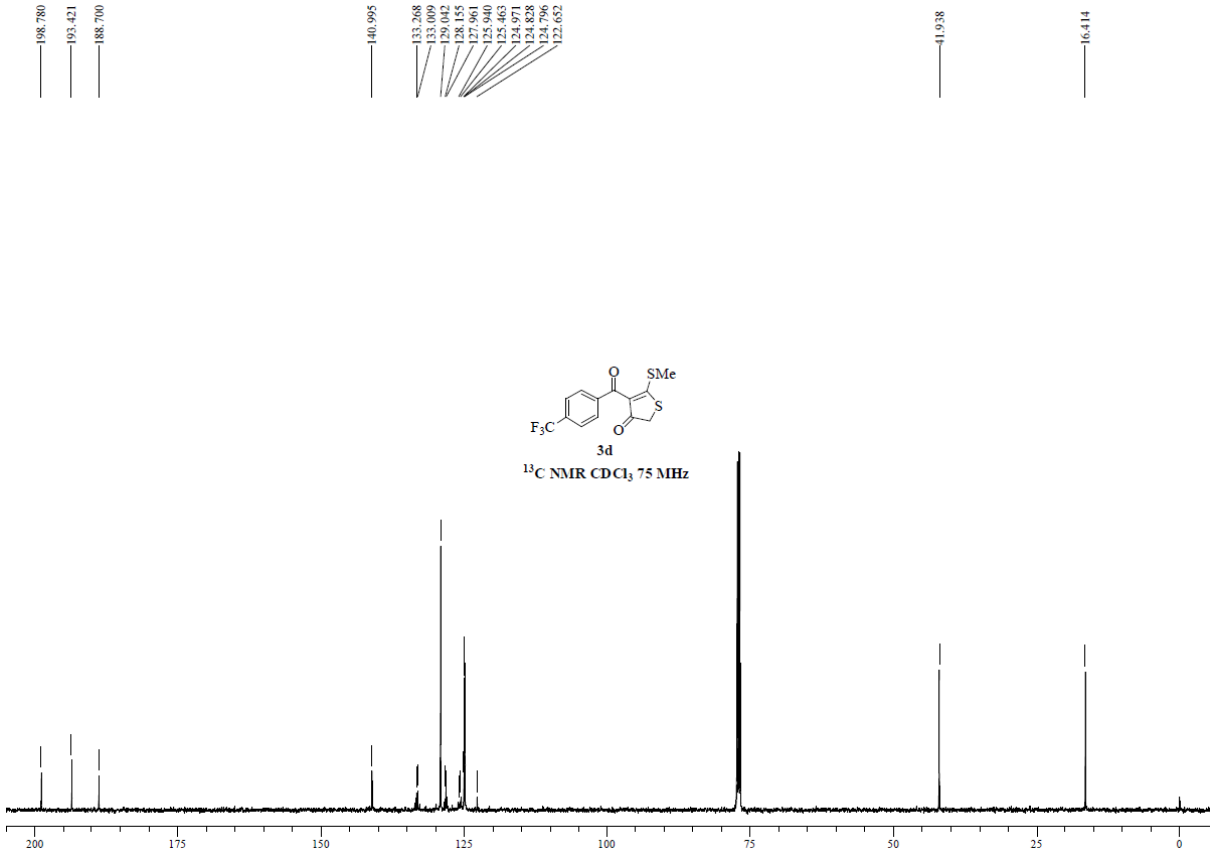
^{1}H NMR $CDCl_3$ 300 MHz

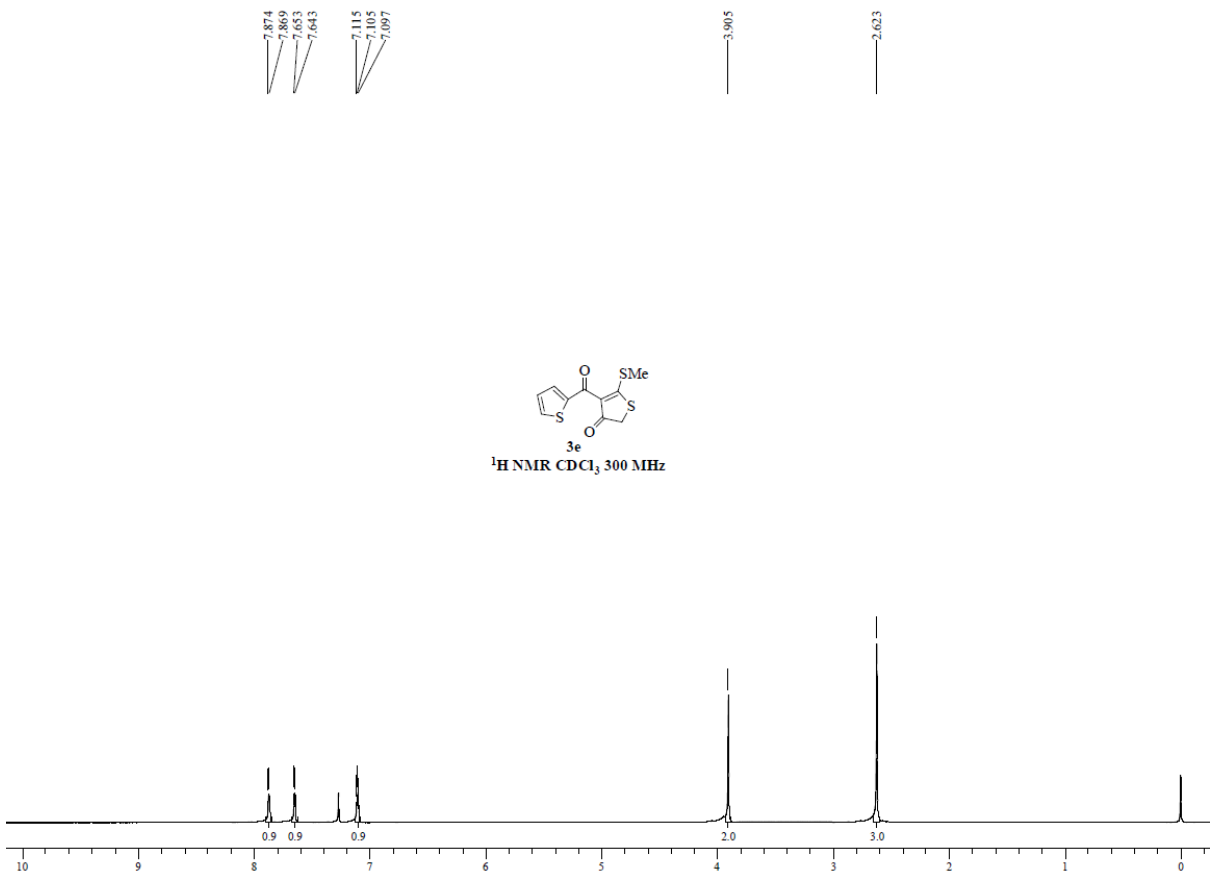


3c

^{13}C NMR $CDCl_3$ 75 MHz

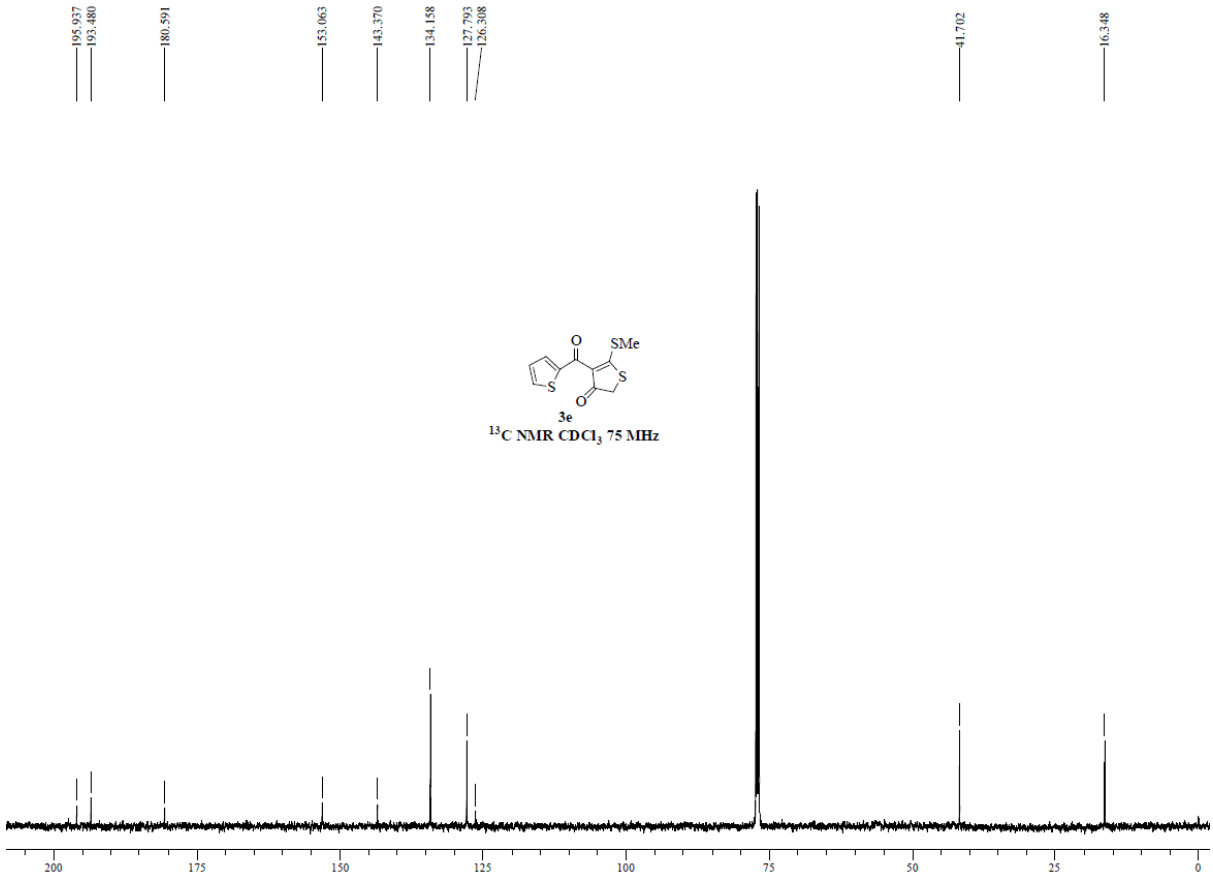

7.734
7.707
7.673
7.646
3.883
2.670
SMe
S
O
F3C
3d
1H NMR CDCl3 300 MHz
4.1
2.0
3.0
10
9
8
7
6
5
4
3
2
1
0


198.780
193.421
188.700
140.995
133.268
133.009
129.042
128.155
127.961
125.940
125.463
124.971
124.828
124.796
122.652
41.938
16.414
SMe
S
O
F3C
3d
13C NMR CDCl3 75 MHz
200
175
150
125
100
75
50
25
0



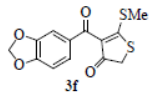
3e

^{1}H NMR $CDCl_3$ 300 MHz



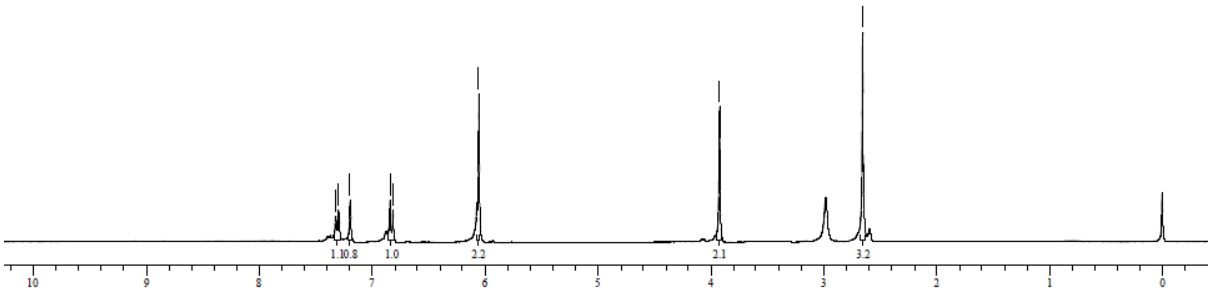
3e

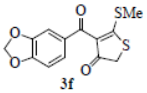
^{13}C NMR $CDCl_3$ 75 MHz



3f

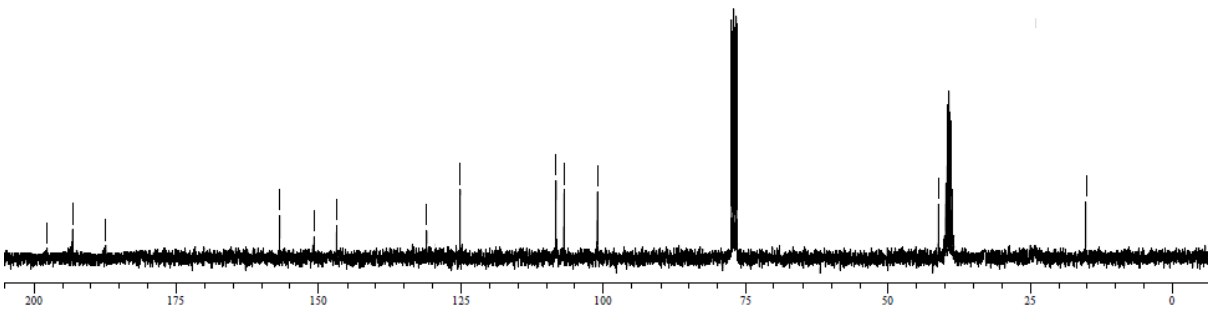
^{1}H NMR $CDCl_3$+DMSO 300 MHz

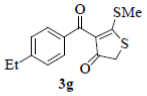




3f

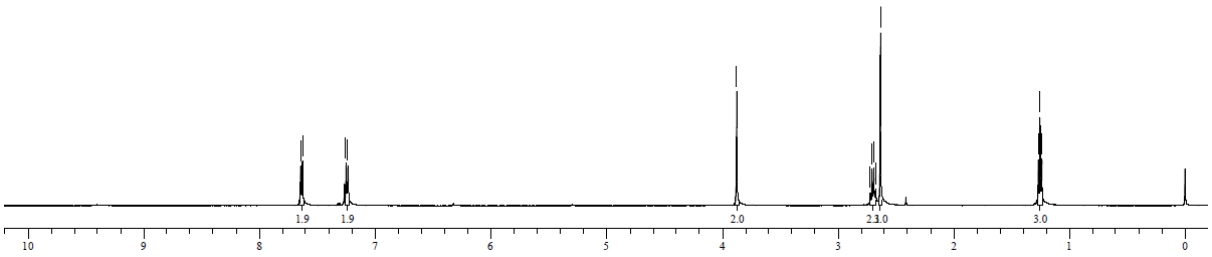
^{13}C NMR $CDCl_3$+DMSO 75 MHz

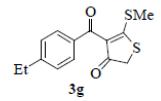




3g

^{1}H NMR $CDCl_3$ 300 MHz





3g

^{13}C NMR $CDCl_3$ 75 MHz