Supporting Information

Visible-light-induced Iron-catalyzed S-N Cross-coupling of Thiols with Dioxazolones

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1. General information

All reactions were carried out under a nitrogen atmosphere. Solvents were purified by standard techniques without special instructions. All commercially available compounds were purchased from Energy Chemical, J&K, Across, Alfa, TCI or Adamas. TLC was carried out on SiO_2 (silica gel 60 F254, Merck), and the spots were located with UV light (254 nm). Flash chromatography was carried out on SiO_2 (silica gel 60, 200-300 mesh). ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance II-400 spectrometer (400 MHz for ^1H, 100 MHz for ^{13}C) and a Bruker AvanceNEO 600M NMR spectrometer (600 MHz for ^1H, 150 MHz for ^{13}C); DMSO-d_6, Acetone-d_6, CD_3OD, $CDCl_3$ and TMS were used as a solvent and an internal standard, respectively. The chemical shifts were reported in ppm downfield (δ) from TMS, the coupling constants *J* are given in Hz. The peak patterns were indicated as follows: s, singlet; d, doublet; t, triplet; m, multiplet; q, quartet. IR spectra were recorded on a NEXUS FT-IR spectrometer. High resolution mass spectra (HRMS) were recorded on quadrupole analyzer using an ESI source (Agilent Technologies G6224A). UV/Vis absorption spectra were obtained with Agilent 8453 UV-Vis spectrometer.

The photocatalytic reactions were performed on Xi'an WATTCAS Parallel Light Reactor (WP-TEC-1020HSL) with 450 nm 10W blue COB LED. And the temperature of the heated reactor was set to indicate temperature.

***Figure S1*. Setup for photocatalytic reactions**

WATTCAS
华太科思
西安华太科思化学技术有限公司
http://www.wattcas.com
Tel: +4006515117
Fax: +86()

CLED Test Report

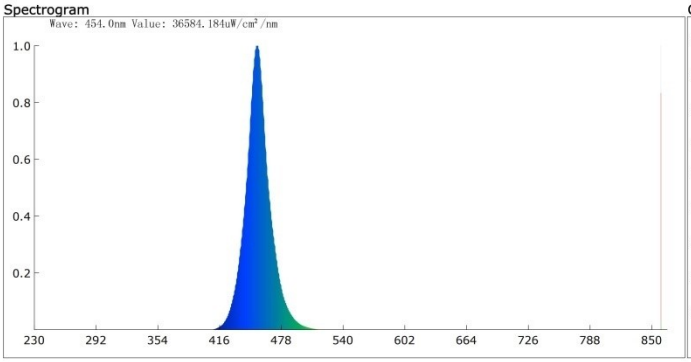
Product Mark

Model: 450-455nm 10W
Manufacture:
Temperature: 20°C
Humidity: 35%
Tester: lilu
Test Date: 2020-07-09,11:27:03

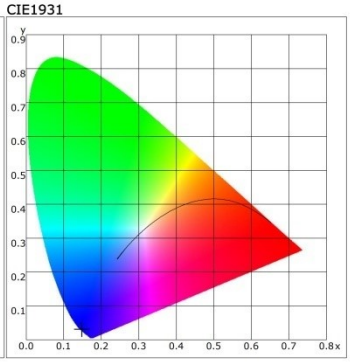
Parameter

Name	Value	Name	Value	Name	Value	Name	Value
FlickerFrequency Hz	0.00	CIE u',v'	0.1930,0.0892	CIE1931 Y	569034.188		
FluctuateDepth(%)	36.2	Duv y0,dy	0.0000,0.0000	CIE1931 Z	15369010.000		
BinkPercent(%)	22.1	SDCM	100.00	TLCI-2012	0		
BinkExponent	0.00	Ra	-67.8	Integral Time(ms)	0		
EffectiveLux(lx)	0.0	Ee(mW/cm²)	975.913	Peak Signal	54611		
PeakLux(lx)	0.0	S/P	22.028	Dark Signal	2072		
LuxIntgarl(lx.s)	0.0	Dominant(nm)	458.50	Compensate level	2854		
FlashTime(us)	0	Purity(%)	99.0				
E(lx)	388650.38	HalfWidth(nm)	22.2				
Candle E(fc)	36106.50	Peak(nm)	454.6				
CCT(K)	100000	Center(nm)	454.6				
Duv	-0.06310	Centroid(nm)	455.7				
CIE x,y	0.1480,0.0304	Color Ratio(RGB)	0.0,7.5,92.5				
CIE u,v	0.1930,0.0595	CIE1931 X	2769507.500				

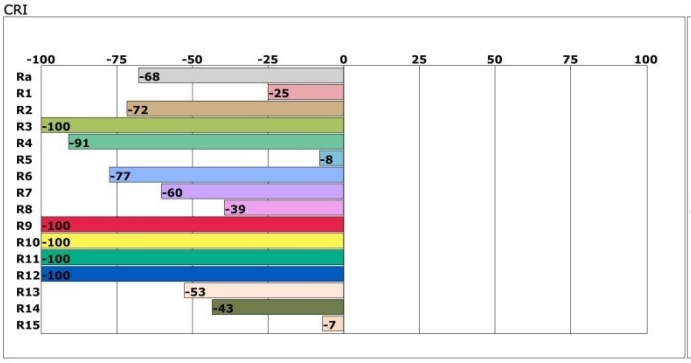
Spectrogram

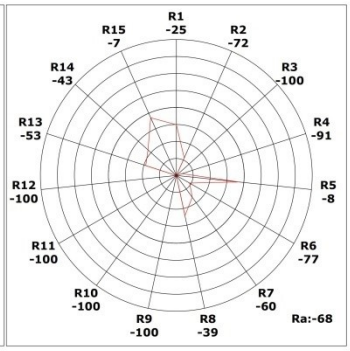


CIE1931



CRI





Instrument Status

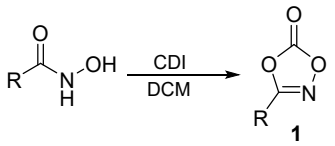
Type: PCS230850
SN: 0
Scan Range: 230-850nm
Integral Time: 0.117ms
VPeak: 54611
VDark: 2072

Remark:

***Figure S2*. Spectrophotocolormeter analysis report**

2. Procedure for the preparation of starting materials

2.1 General procedure for the preparation of dioxazolone (1)[1]

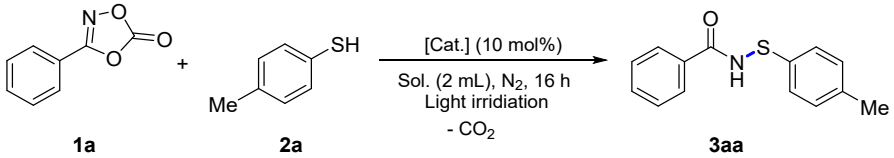


1

To a stirred solution of hydroxamic acid (10.0 mmol) in dichloromethane (DCM, was added 1,1'-carbonyldiimidazole (CDI 1.62 g, 10.0 mmol) at room temperature. The reaction mixture was stirred for 30 min until a high conversion of hydroxamic acid was reached (detected by TLC). Then, the reaction mixture was quenched with 1 $molL^{-1}$ HCl (50 mL), and the mixture was extracted with DCM, dried over $MgSO_4$ and concentrated under reduced pressure to give the dioxazolone **1**. Product was recrystallized with acetone/hexane, if necessary.

3. Procedure for optimization studies

***Table S1*. Optimization of the catalyst, solvent and others.**



1a **2a** **3aa**

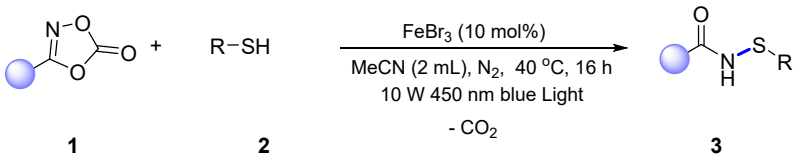
Entry	Catalyst	Solvent	Wavelength/nm	Yield[%][a]
1	$FeCl_2$	DCM	450	49
2	$Fe(OAc)_2$	DCM	450	13
3	$FeSO_4·7H_2O$	DCM	450	22
4	$Fe(acac)_2$	DCM	450	trace
5	FeF_3	DCM	450	trace
6	$Fe(OTf)_3$	DCM	450	NR
7	$FeCl_3$	DCM	450	58
8	$FeBr_3$	DCM	450	70
9	$FeBr_2$	DCM	450	67
10	$FeBr_3$	Et_2O	450	33

11	$FeBr_3$	DCE	450	59
12	$FeBr_3$	1,4-Dioxane	450	65
13	$FeBr_3$	Toluene	450	trace
14	$FeBr_3$	DMF	450	NR
15	$FeBr_3$	MeCN	450	83
16	$FeBr_3$	MeCN	365	trace
17	$FeBr_3$	MeCN	400	trace
18	$FeBr_3$	MeCN	500	11
19[b]	$FeBr_3$	MeCN	450	40
20[c]	$FeBr_3$	MeCN	---	trace
21	---	MeCN	450	17

[a] Reaction conditions: **1a** (0.4 mmol), **2a** (0.2 mmol), Cat. (10 mol%) and Sol. (2.0 mL) at 40 °C under light irradiation for 16 h, yield of isolated product. [b] Cat. (5 mol%). [c] Without light irradiation.

4. Experimental procedures

4.1 General procedure for the S-N cross coupling of thiols with dioxazolones

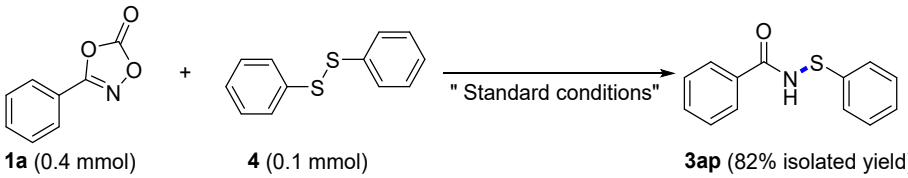


1 **2** **3**

To an oven-dried 20 mL quartz tube equipped with a magnetic stir bar was added dioxazolone (0.40 mmol, 2.0 equiv.) and $FeBr_3$ (5.9 mg, 0.02 mmol, 0.10 equiv.) in the glove box under inert gas atmosphere. Then, MeCN or DCM (2.0 mL) was added followed by thiol substrate (0.2 mmol, 1.0 equiv. for liquid substrates) via syringe under a nitrogen atmosphere. The mixture was stirred and irradiated with a 10 W blue LEDs (λ_{max} = 450 nm) and the temperature of the heated reactor was set to 40 °C for 16 h. After the reaction completed, the mixture was quenched with brine and washed with DCM. The combined organic layer was dried over Na_2SO_4, concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography to give the pure desired product **3**.

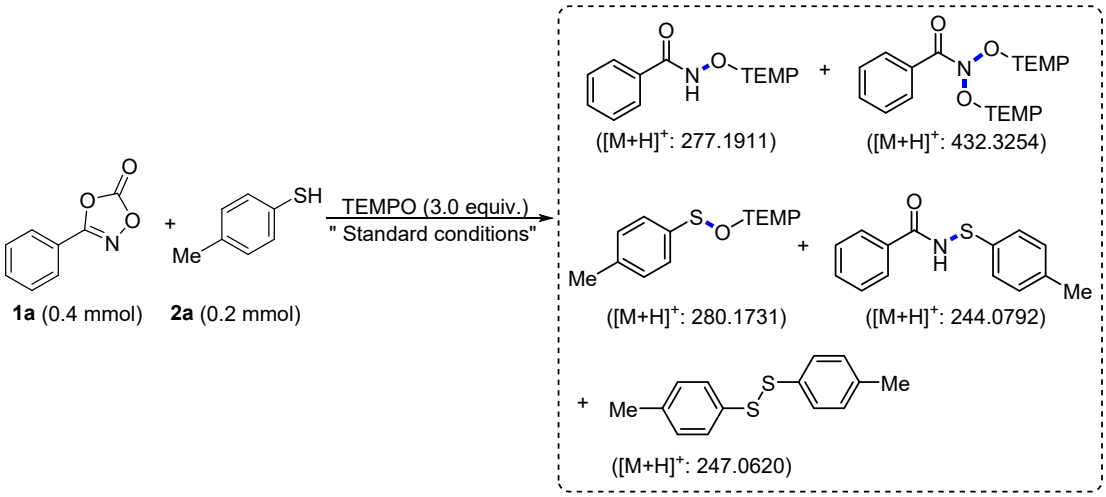
5. Mechanistic studies

5.1 S-N cross-coupling of 1,2-diphenyldisulfane 4 with 1a



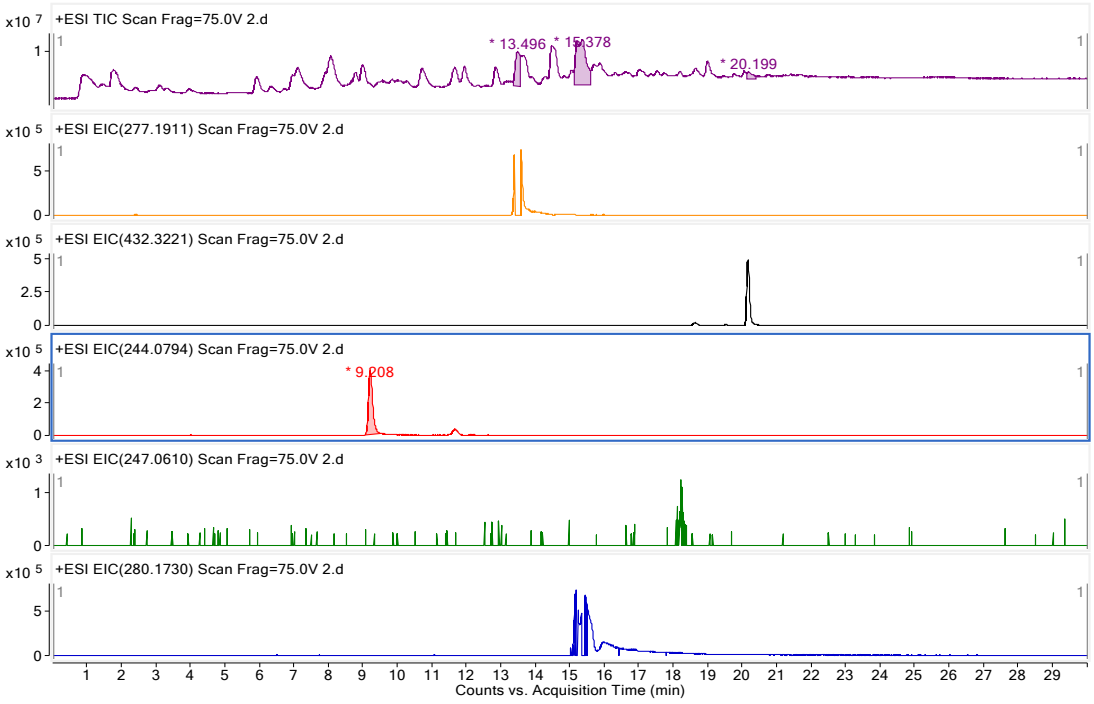
In a glovebox, the oven-dried quartz tube (20 mL) containing a stirring bar was charged with 3-phenyl-1,4,2-dioxazol-5-one (**1a**) (65.2 mg, 0.40 mmol), 1,2-diphenyldisulfane **4** (21.8 mg, 0.10 mmol), and $FeBr_3$ (5.9 mg, 0.02 mmol, 0.10 equiv.). Then, dry MeCN (2.0 mL) was injected into the tube via syringe under a nitrogen atmosphere. Subsequently, the reaction mixture was stirred under the irradiation of a 10 W blue LEDs (λ_{max} = 450 nm) at 40 °C for 16 h. After the reaction completed, the mixture was purified by column chromatography on silica gel to give the **3ap** with 82% isolated yield. This result indicates that sulfur radical is maybe a potential intermediate for S-N cross-coupling.

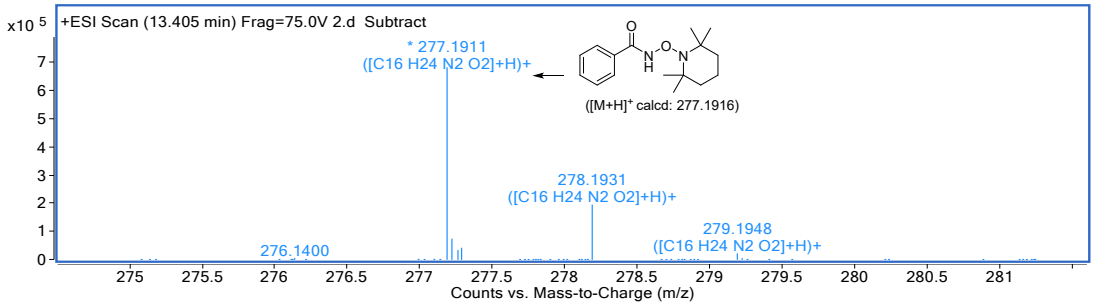
5.3 Radical capture experiment with TEMPO



To an oven-dried 20 mL quartz tube equipped with a magnetic stir bar was added **1a** (65.2 mg, 0.40 mmol), 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO, 93.7 mg, 0.6 mmol, 3.0 equiv.), and $FeBr_3$ (5.9 mg, 0.02 mmol, 0.10 equiv.) in the glove box under N_2 atmosphere. Then, dry MeCN (2.0 mL) was added followed by 4-

methylbenzenethiol (**2a**, 24.8 mg, 0.20 mmol) via syringe under a nitrogen atmosphere. Subsequently, the reaction mixture was stirred under the irradiation of a 10 W blue LEDs (λ_{max} = 450 nm) at 40 °C for 16 h. And LC-HRMS analyses revealed the presence of TEMPO-trapped acyl nitrene adducts with cluster peaks at 277.1911 and 432.3254 m/z (Figure S3). Notably, we also detected a TEMPO-trapped thiophenol adduct with a cluster peak at 280.1731 m/z through LC-HRMS analysis (Figure S3). These results suggest that the visible-light induced iron-catalyzed S-N cross coupling may occur via a radical pathway.





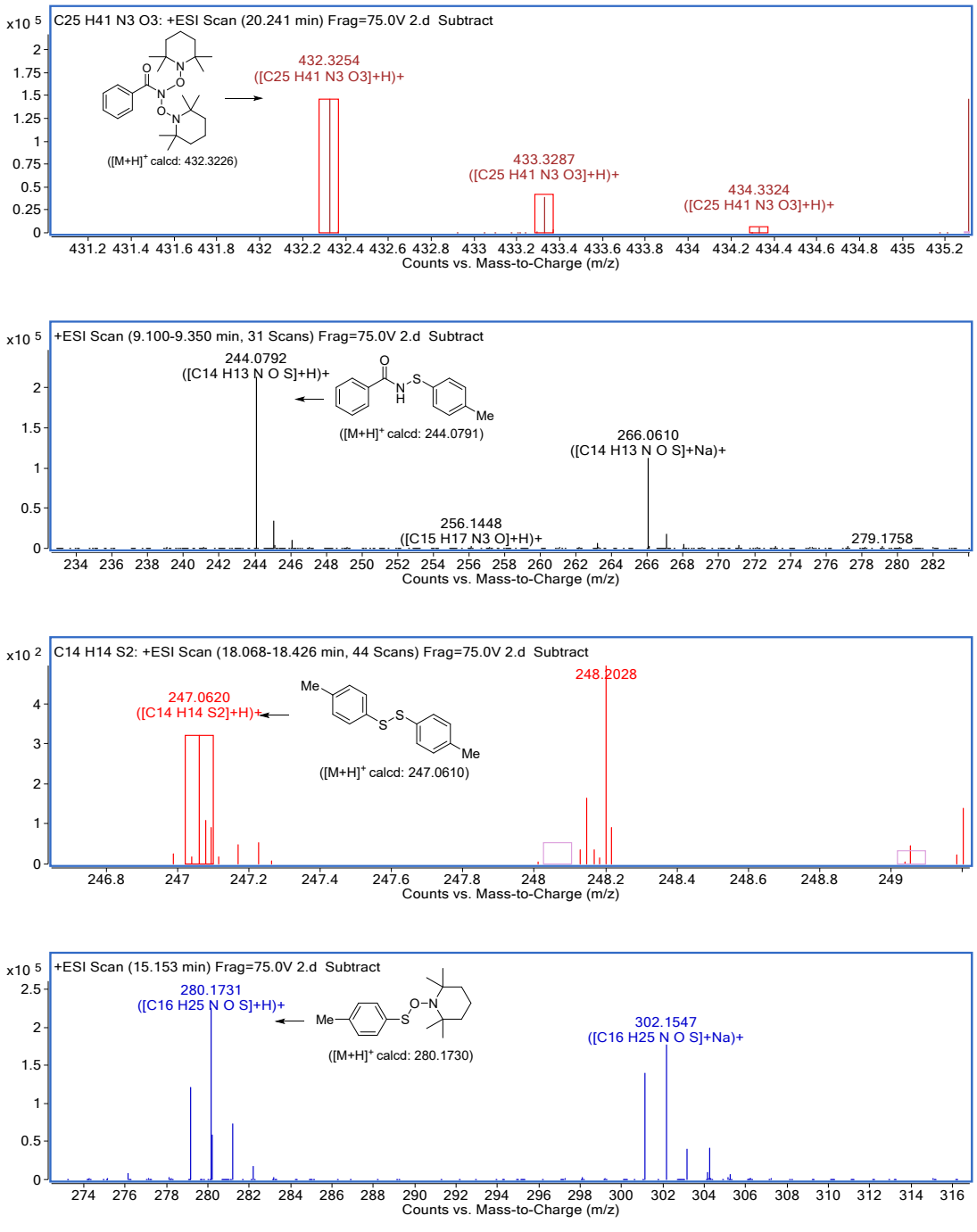
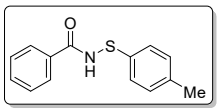


Figure S3 LC/MS analysis of radical capture experiment for S-N cross-coupling

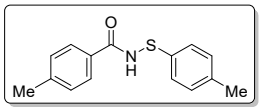
6. Characterization for all compounds

N-(p-tolylthio)benzamide (3aa)[2]



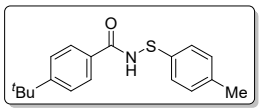
White solid (40.4 mg, 83% yield), mp 129−131 °C, (lit.[2] mp130–131 °C). ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.17 (s, 1H), 7.95 (d, J = 7.4 Hz, 2H), 7.61–7.57 (m, 1H), 7.52–7.49 (m, 2H), 7.15 (s, 4H), 2.24 (s, 3H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 168.3, 136.0, 135.5, 133.2, 132.2, 129.7, 128.6, 127.9, 124.2, 20.5.

4-methyl-N-(p-tolylthio)benzamide (3ba)



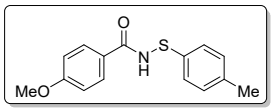
White solid (36.0 mg, 70% yield), mp 132−134 °C. ^{1}H NMR (Acetone-d_6, 400 MHz) δ 9.19 (s, 1H), 7.91 (d, J = 8.2 Hz, 2H), 7.30 (d, J = 8.2 Hz, 2H), 7.25–7.23 (m, 2H), 7.14 (d, J = 8.3 Hz, 2H), 2.39 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (Acetone-d_6, 100 MHz) δ 169.0, 143.5, 137.5, 137.2, 132.0, 130.5, 130.1, 128.8, 126.4, 21.5, 21.0; IR (KBr): 3252, 2920, 1659, 1491, 1434, 1397, 1263, 1189, 1100, 1017, 801, 751 (cm^{-1}); HRMS (ESI): ([M+H]$^+$) calcd for $C_{15}H_{16}NOS^+$: 258.0947; found: 258.0951.

4-(tert-butyl)-N-(p-tolylthio)benzamide (3ca)



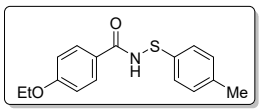
White solid (46.7 mg, 78% yield), mp 160−162 °C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.78 (d, J = 8.4 Hz, 2H), 7.45–7.43 (m, 3H), 7.30 (d, J = 8.2 Hz, 2H), 7.10 (d, J = 7.9 Hz, 2H), 2.31 (s, 3H),1.33 (s, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 169.0, 156.1, 137.6, 135.3, 130.6, 129.9, 127.6, 127.3, 125.8, 35.2, 31.2, 21.2; IR (KBr): 3278, 2962, 1655, 1492, 1432, 1400, 1364, 1261, 1118, 1095, 1030, 801 (cm^{-1}); HRMS (ESI): ([M-H]$^-$) calcd for $C_{18}H_{20}NOS^-$: 298.1271; found: 298.1274.

4-methoxy-N-(p-tolylthio)benzamide (3da)



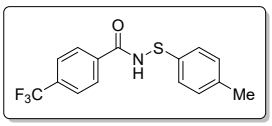
White solid (44.8 mg, 82% yield), mp 144−146 °C. ^{1}H NMR (Acetone-d_6, 400 MHz) δ 9.12 (s, 1H), 8.01–7.98 (m, 2H), 7.23 (d, J = 8.2 Hz, 2H), 7.14 (d, J = 8.0 Hz, 2H), 7.04–7.00 (m, 2H), 3.87 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (Acetone-d_6, 100 MHz) δ 168.5, 163.8, 137.6, 137.1, 130.8, 130.5, 126.9, 126.3, 114.7, 56.0, 21.0; IR (KBr): (cm^{-1}) 3287, 2919, 1655, 1605, 1515, 1433, 1405, 1176, 1097, 1029, 802, 606; HRMS (ESI): ([M-H]$^{-}$) calcd for $C_{15}H_{14}NO_2S^{-}$: 272.0751; found: 272.0753.

4-methoxy-N-(p-tolylthio)benzamide (3ea)



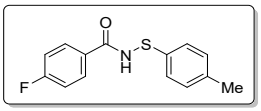
White solid (46.0 mg, 80% yield), mp 138−139 °C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.82–7.78 (m, 2H), 7.50 (s, 1H), 7.27 (d, J = 8.3 Hz, 2H), 7.08 (d, J = 8.0 Hz, 2H), 6.89–6.85 (m, 2H), 4.05 (q, J = 7.0 Hz, 2H), 2.29 (s, 3H), 1.42 (t, J = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 168.6, 162.4, 137.4, 135.4, 129.9, 129.7, 127.1, 125.4, 114.5, 63.8, 21.2, 14.8; IR (KBr): 3252, 2923, 1655, 1605, 1436, 1396, 1248, 1177, 1100, 1043, 802, 627 (cm^{-1}); HRMS (ESI): ([M-H]$^{-}$) calcd for $C_{16}H_{16}NO_2S^{-}$: 286.0907; found: 286.0913.

N-(p-tolylthio)-4-(trifluoromethyl)benzamide (3fa)



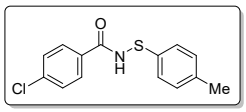
White solid (26.1 mg, 42% yield), mp 169−170 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.4 (s, 1H), 8.14 (d, J = 8.1 Hz, 2H), 7.91 (d, J = 8.1 Hz, 2H), 7.21–7.16 (m, 4H), 2.27 (s, 3H); ^{13}C NMR(DMSO-d_6, 100 MHz) δ 167.4, 137.0, 135.8, 135.5, 131.9 (q, $J_{C\text{-}F}$ = 32.1 Hz), 129.7, 128.8, 125.6 (q, $J_{C\text{-}F}$ = 3.8 Hz), 124.6, 123.8 (q, $J_{C\text{-}F}$ = 272.6 Hz), 20.5; IR (KBr): 3424, 3273, 1665, 1428, 1330, 1163, 1128, 1051, 1027, 1006, 805, 622 (cm^{-1}); HRMS (ESI): ([M-H]$^{-}$) calcd for $C_{15}H_{11}F_3NOS^{-}$: 310.0519; found: 310.0521.

4-fluoro-N-(p-tolylthio)benzamide (3ga)



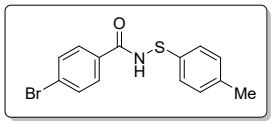
Yellow solid (44.4 mg, 85% yield), mp 162−163 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.19 (s, 1H), 8.05–8.01 (m, 2H), 7.37–7.32 (m, 2H), 7.16 (s, 4H), 2.25 (s, 3H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 167.3, 164.4 (d, $J_{C\text{-}F}$ = 250.0 Hz), 135.9, 135.6, 130.7 (d, $J_{C\text{-}F}$ = 9.2 Hz), 129.7, 129.6, 124.3, 115.6 (d, $J_{C\text{-}F}$ = 21.9 Hz), 20.5; IR (KBr): 3435, 2973, 1676, 1443, 1258, 1232, 1054, 1027, 1008, 822, 762, 597 (cm^{-1}); HRMS (ESI): ([M-H]$^{-}$) calcd for $C_{14}H_{11}FNOS^{-}$: 260.0051; found: 260.0054.

4-chloro-N-(p-tolylthio)benzamide (3ha)



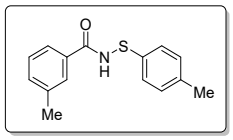
White solid (40.6 mg, 73% yield), mp 166−168 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.24 (s, 1H), 7.96 (d, J = 8.6 Hz, 2H), 7.59 (d, J = 8.6 Hz, 2H), 7.18–7.14 (m, 4H), 2.26 (s, 3H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 167.4, 137.1, 135.8, 135.7, 131.9, 129.8, 129.7, 128.7, 124.4, 20.5; IR (KBr): 3258, 2919, 1661, 1591, 1491, 1426, 1261, 1091, 1012, 846, 803, 756 (cm^{-1}); HRMS (ESI): ([M+H]$^{+}$) calcd for $C_{14}H_{13}ClNOS^{+}$: 278.0401; found: 278.0388.

4-bromo-N-(p-tolylthio)benzamide (3ia)



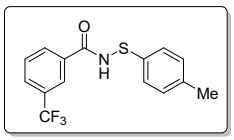
White solid (47.0 mg, 73% yield), mp 154−156 °C. ^{1}H NMR (DMSO-d_6, 600 MHz) δ 10.26 (s, 1H), 7.87 (d, J = 8.2 Hz, 2H), 7.73 (d, J = 8.4 Hz, 2H), 7.16–7.13 (m, 4H), 2.24 (s, 3H); ^{13}C NMR (DMSO-d_6, 150 MHz) δ 167.5, 135.8, 135.7, 132.3, 131.7, 130.0, 129.7, 126.2, 124.4, 20.6; IR (KBr): 3268, 1667, 1588, 1491, 1438, 1262, 1117, 1068, 1009, 802, 754, 643 (cm^{-1}); HRMS (ESI): ([M+H]$^{+}$) calcd for $C_{14}H_{13}BrNOS^{+}$: 321.9896; found: 321.9896.

3-methyl-N-(p-tolylthio)benzamide (3ja)



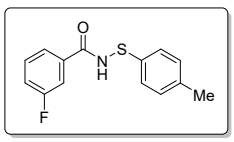
Colorless viscous oil (23.2 mg, 45% yield). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.65 (s, 1H), 7.60 (d, J = 7.2 Hz, 1H), 7.49 (s, 1H), 7.35–7.32 (m, 2H), 7.30 (d, J = 7.8 Hz, 2H), 7.10 (d, J = 8.0 Hz, 2H), 2.37 (s, 3H), 2.31 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 169.4, 138.8, 137.6, 135.1, 133.4, 133.2, 129.9, 128.7, 128.4, 127.4, 124.6, 21.4, 21.2.; IR (KBr): 3252, 2921, 1660, 1491, 1432, 1270, 1195, 1082, 921, 801, 774, 689 (cm^{-1}); HRMS (ESI): ($[M+H]^+$) calcd for $C_{15}H_{16}NOS^+$: 258.0947; found: 258.0957.

N-(p-tolylthio)-3-(trifluoromethyl)benzamide (3ka)



Yellow solid (39.8 mg, 64% yield), mp 100−102 °C. ^{1}H NMR (Acetone-d_6, 400 MHz) δ 9.53 (s, 1H), 8.31–8.27 (m, 2H), 7.94 (d, J = 7.8 Hz, 1H), 7.79–7.75 (m, 1H), 7.29 (d, J = 8.2 Hz, 2H), 7.16 (d, J = 8.2 Hz, 2H), 2.28 (s, 3H); ^{13}C NMR(Acetone-d_6, 100 MHz) δ 168.0, 137.7, 136.7, 135.7, 132.7, 131.3 (q, $J_{C\text{-}F}$ = 32.2 Hz), 130.7, 130.6, 129.5 (q, $J_{C\text{-}F}$ = 3.8 Hz), 127.0, 125.5 (q, $J_{C\text{-}F}$ = 3.9 Hz), 125.0 (q, $J_{C\text{-}F}$ = 271.8 Hz), 21.1; IR (KBr): 3243, 2924, 1665, 1492, 1420, 1331, 1251, 1169, 1130, 1073, 802, 696 (cm^{-1}); HRMS (ESI): ($[M+H]^+$) calcd for $C_{15}H_{13}F_3NOS^+$: 312.0664; found: 312.0663.

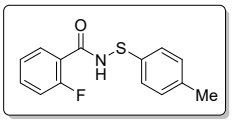
3-fluoro-N-(p-tolylthio)benzamide (3la)



Yellow solid (24.0 mg, 46% yield), mp 106−108 °C. ^{1}H NMR (DMSO-d_6, 600 MHz) δ 10.25 (s, 1H), 7.79 (d, J = 7.6 Hz, 1H), 7.74 (d, J = 9.7 Hz, 1H), 7.58–7.55 (m, 1H), 7.47–7.44 (m, 1H), 7.16 (s, 4H), 2.25 (s, 3H); ^{13}C NMR (DMSO-d_6, 150 MHz) δ 167.1, 161.9 (d, $J_{C\text{-}F}$ = 244.8 Hz), 135.7 (d, $J_{C\text{-}F}$ = 14.1 Hz), 135.4 (d, $J_{C\text{-}F}$ = 6.6 Hz),

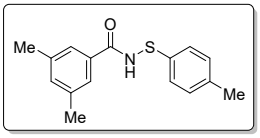
130.8 (d, $J_{C\text{-}F}$ = 7.7 Hz), 129.7, 124.5, 124.1 (d, $J_{C\text{-}F}$ = 2.9 Hz), 119.2 (d, $J_{C\text{-}F}$ = 21.0 Hz), 114.7 (d, $J_{C\text{-}F}$ = 23.1 Hz), 20.5; IR (KBr): 3251, 2922, 1663, 1588, 1492, 1456, 1268, 1204, 1091, 931, 802, 680 (cm^{-1}); HRMS (ESI): ($[M+H]^+$) calcd for $C_{14}H_{13}FNOS^+$: 262.0696; found: 262.0701.

2-fluoro-N-(p-tolylthio)benzamide (3ma)



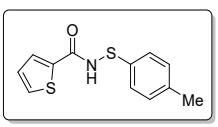
White solid (43.4 mg, 83% yield), mp 85−87 °C. ^1H NMR (DMSO-d_6, 400 MHz) δ 10.13 (s, 1H), 7.66–7.56 (m, 2H), 7.36–7.29 (m, 2H), 7.22–7.17 (m, 4H), 2.27 (s, 3H); ^{13}C NMR(DMSO-d_6, 100 MHz) δ 166.3, 159.1 (d, $J_{C\text{-}F}$ = 250.4 Hz), 135.7 (d, $J_{C\text{-}F}$ = 9.6 Hz), 133.1 (d, $J_{C\text{-}F}$ = 8.7 Hz), 130.0 (d, $J_{C\text{-}F}$ = 2.8 Hz), 129.7, 124.7 (d, $J_{C\text{-}F}$ = 3.3 Hz), 124.3, 123.4 (d, $J_{C\text{-}F}$ = 14.7 Hz), 116.3 (d, $J_{C\text{-}F}$ = 21.7 Hz), 20.6; IR (KBr): 3208, 2973, 1672, 1612, 1455, 1279, 1220, 1116, 1085, 1024, 801, 753 (cm^{-1}); HRMS (ESI): ($[M+H]^+$) calcd for $C_{14}H_{13}FNOS^+$: 262.0696; found: 262.0706.

3,5-dimethyl-N-(p-tolylthio)benzamide (3na)



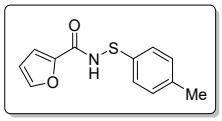
Colorless viscous oil (46.1 mg, 85% yield). ^1H NMR ($CDCl_3$, 400 MHz) δ 7.52 (s, 1H), 7.43 (s, 2H), 7.28 (d, J = 8.2 Hz, 2H), 7.15 (s, 1H), 7.08 (d, J = 8.0 Hz, 2H), 2.33 (s, 6H), 2.30 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 169.5, 138.6, 137.5, 135.2, 134.0, 133.4, 129.8, 127.2, 125.4, 21.3, 21.2; IR (KBr): 3242, 2920, 1659, 1605, 1452, 1405, 1302, 1220, 1117, 867, 802, 685 (cm^{-1}); HRMS (ESI): ($[M-H]^-$) calcd for $C_{16}H_{16}NOS^-$: 270.0958; found: 270.0960.

N-(p-tolylthio)thiophene-2-carboxamide (3oa)



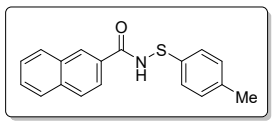
Brown solid (18.0 mg, 36% yield), mp 127−129 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.18 (s, 1H), 8.00 (dd, J = 3.8, 1.0 Hz, 1H), 7.89 (dd, J = 5.0, 1.0 Hz, 1H), 7.21 (dd, J = 5.0, 3.8 Hz, 1H), 7.16 (s, 4H), 2.25 (s, 3H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 162.9, 137.9, 135.9, 135.8, 132.7, 130.2, 129.6, 128.2, 124.5, 20.5. IR (KBr): 3250, 2921, 1648, 1521, 1492, 1436, 1267, 1093, 1040, 863, 720, 656 (cm^{-1}); HRMS (ESI): ([M-H]$^-$) calcd for $C_{12}H_{10}NOS_2^-$: 248.0209; found: 248.0210.

N-(p-tolylthio)thiophene-2-carboxamide (3pa)



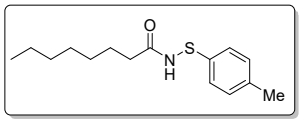
Colorless viscous oil (27.1 mg, 58% yield). ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.09 (s, 1H), 7.94 (d, J = 1.0 Hz, 1H), 7.38 (d, J = 3.5 Hz, 1H), 7.18–7.13 (m, 4H), 6.69 (dd, J = 3.5, 1.7 Hz, 1H), 2.26 (s, 3H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 159.3, 146.5, 146.4, 135.9, 135.7, 129.6, 124.5, 115.7, 112.1, 20.5. IR (KBr): 3225, 2920, 1664, 1471, 1416, 1275, 1165, 1113, 1013, 933, 801, 758 (cm^{-1}); HRMS (ESI): ([M-H]$^-$) calcd for $C_{12}H_{10}NO_2S^-$: 232.0438; found: 232.0428.

N-(p-tolylthio)-2-naphthamide (3qa)



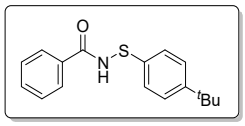
Yellow solid (45.2 mg, 77% yield), mp 125−127 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.32 (s, 1H), 8.60 (s, 1H), 8.07–8.05 (m, 2H), 8.01–7.98 (m, 2H), 7.67–7.60 (m, 2H), 7.21–7.16 (m, 4H), 2.26 (s, 3H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 168.5, 136.0, 135.6, 134.5, 132.1, 130.5, 129.7, 129.1, 128.7, 128.21, 128.16, 127.7, 127.0, 124.38, 124.35, 20.6. IR (KBr): (cm^{-1}) 3223, 2922, 1664, 1412, 1384, 1248, 1193, 1135, 1087, 1027, 803, 784; HRMS (ESI): ([M-H]$^-$) calcd for $C_{18}H_{14}NOS^-$: 292.0802; found: 292.0804.

N-(p-tolylthio)octanamide (3ra)



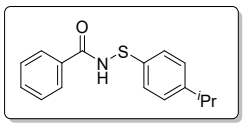
White solid (40.3 mg, 76% yield), mp 79−80 °C. ^{1}H NMR (CD_3OD, 400 MHz) δ 7.19–7.12 (m, 4H), 2.35 (t, J = 7.4 Hz, 2H), 2.30 (s, 3H), 1.67–1.62 (m, 2H), 1.30 (s, 8H), 0.90 (t, J = 6.6 Hz, 3H); ^{13}C NMR (CD_3OD, 100 MHz) δ 177.1, 136.7, 135.5, 129.3, 125.9, 35.8, 31.5, 28.8, 28.7, 25.5, 22.3, 19.7, 13.1. IR (KBr): (cm^{-1}) 3242, 2924, 2852, 1672, 1468, 1451, 1410, 1225, 1173, 1118, 804, 708; HRMS (ESI): ($[M+H]^+$) calcd for $C_{15}H_{24}NOS^+$: 266.1573; found: 266.1569.

N-[(4-(tert-butyl)-phenyl)thio]benzamide (3ab)



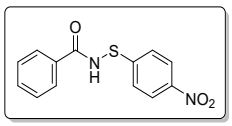
White solid (40.5 mg, 71% yield), mp 137−139 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.17 (s, 1H), 7.95 (d, J = 7.3 Hz, 2H), 7.62–7.58 (m, 1H), 7.53–7.49 (m, 2H), 7.36 (d, J = 8.5 Hz, 2H), 7.18 (d, J = 8.5 Hz, 2H), 1.23 (s, 9H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 168.4, 148.8, 136.1, 133.2, 132.2, 128.6, 127.9, 125.9, 124.0, 34.2, 31.0; IR (KBr): 3192, 2962, 1679, 1498, 1452, 1426, 1262, 1050, 1026, 1010, 820, 702 (cm^{-1}); HRMS (ESI): ($[M-H]^-$) calcd for $C_{17}H_{18}NOS^-$: 284.1115; found: 284.1113.

N-[(4-(tert-butyl)-phenyl)thio]benzamide (3ac)



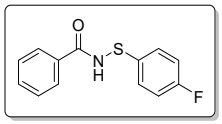
White solid (39.6 mg, 73% yield), mp 108−110 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.17 (s, 1H), 7.96 (d, J = 7.5 Hz, 2H), 7.63–7.59 (m, 1H), 7.54–7.50 (m, 2H), 7.24–7.17 (m, 4H), 2.89–2.79 (m, 1H), 1.16 (d, J = 6.9 Hz, 6H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 168.4, 146.6, 136.4, 133.2, 132.2, 128.6, 127.9, 127.1, 124.3, 33.0, 23.8; IR (KBr): 3260, 2960, 1660, 1494, 1451, 1420, 1261, 1098, 1027, 819, 709, 692 (cm^{-1}); HRMS (ESI): ($[M-H]^-$) calcd for $C_{16}H_{16}NOS^-$: 270.0958; found: 270.0957.

N-[(4-nitrophenyl)thio]benzamide (3ad)



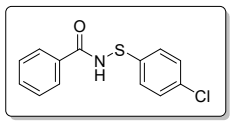
Yellow solid (38.4 mg, 70% yield), mp 154−156 °C, (lit.[2] mp157–158 °C). ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.41 (s, 1H), 8.18 (d, J = 7.2 Hz, 2H), 8.00 (d, J = 5.6 Hz, 2H), 7.64–7.55 (m, 3H), 7.41 (d, J = 7.3 Hz, 2H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 168.2, 149.7, 145.0, 132.6, 132.6, 128.7, 128.1, 124.2, 122.4; IR (KBr): 3250, 2923, 1663, 1580, 1514, 1451, 1417, 1338, 1257, 1087, 853, 711 (cm^{-1}); HRMS (ESI): ([M+H]$^+$) calcd for $C_{13}H_{11}N_2O_3S^+$: 275.0485; found: 275.0487.

N-[(4-fluorophenyl)thio]benzamide (3ae)[4]



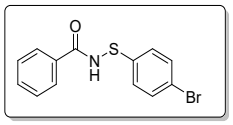
White solid (39.1 mg, 79% yield), mp 120−122 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.23 (s, 1H), 7.95 (d, J = 7.3 Hz, 2H), 7.63–7.59 (m, 1H), 7.54–7.50 (m, 2H), 7.33–7.30 (m, 2H), 7.23–7.19 (m, 2H); ^{13}C NMR(DMSO-d_6, 100 MHz) δ 168.3, 160.8 (d, $J_{C\text{-}F}$ = 243.3 Hz), 135.0 (d, $J_{C\text{-}F}$ = 3.0 Hz), 133.0, 132.3, 128.6, 127.9, 126.5 (d, $J_{C\text{-}F}$ = 8.3 Hz), 116.1 (d, $J_{C\text{-}F}$ = 22.2 Hz); IR (KBr): 3235, 2925, 1660, 1589, 1490, 1452, 1418, 1260, 1227, 1096, 822, 692 (cm^{-1}); HRMS (ESI): ([M-H]$^-$) calcd for $C_{13}H_9FNOS^-$: 246.0394; found: 246.0395.

N-[(4-chlorophenyl)thio]benzamide (3af)[2]



White solid (27.4 mg, 52% yield), mp 142−143 °C, (lit.[2] mp 143 °C).^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.84 (d, J = 7.6 Hz, 2H), 7.72 (s, 1H), 7.57–7.53 (m, 1H), 7.44–7.41 (m, 2H), 7.23 (s, 4H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 169.3, 137.2, 133.1, 133.1, 132.7, 129.3, 128.9, 127.8, 127.3.

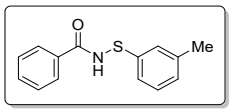
N-[(4-bromophenyl)thio]benzamide (3ag)[4]



White solid (50.5 mg, 82% yield), mp 146−148 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.26 (s, 1H), 7.98–7.96 (m, 2H), 7.64–7.60 (m, 1H), 7.55–7.51 (m, 4H), 7.19–7.16

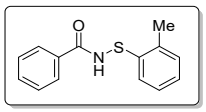
(m, 2H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 168.3, 139.3, 132.9, 132.4, 131.8, 128.6, 128.0, 125.2, 118.5; IR (KBr): 3227, 2921, 1660, 1471, 1451, 1418, 1259, 1085, 1026, 1006, 808, 693 (cm^{-1}); HRMS (ESI): ([M-H]$^-$) calcd for $C_{13}H_9BrNOS^-$: 305.9594; found: 305.9584.

N-(m-tolylthio)benzamide (3ah)[5]



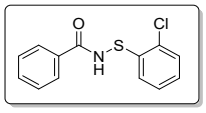
White solid (37.0 mg, 76% yield), mp 82−84 °C. ^{1}H NMR (Acetone-d_6, 400 MHz) δ 9.25 (s, 1H), 8.03 (d, J = 7.5 Hz, 2H), 7.63–7.59 (m, 1H), 7.54–7.50 (m, 2H), 7.23–7.19 (m, 1H), 7.13–7.09 (m, 2H), 7.00 (d, J = 7.5 Hz, 1H), 2.28 (s, 3H); ^{13}C NMR (Acetone-d_6, 100 MHz) δ 169.2, 140.8, 139.6, 134.8, 133.1, 129.8, 129.5, 128.8, 127.9, 125.6, 122.3, 21.4; IR (KBr): 3253, 2923, 1661, 1580, 1452, 1421, 1261, 1098, 1073, 1027, 771, 690 (cm^{-1}); HRMS (ESI): ([M-H]$^-$) calcd for $C_{14}H_{12}NOS^-$: 242.0645; found: 242.0641.

N-(o-tolylthio)benzamide (3ai)[4]



Yellow solid (39.9 mg, 82% yield), mp 68−70 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.09 (s, 1H), 7.99 (d, J = 7.3 Hz, 2H), 7.64–7.60 (m, 1H), 7.55–7.52 (m, 2H), 7.20–7.16 (m, 2H), 7.10–7.03 (m, 2H), 2.29 (s, 3H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 168.4, 137.9, 133.1, 132.3, 131.9, 130.1, 128.6, 128.0, 126.6, 125.3, 122.3, 18.4; IR (KBr): 3251, 2974, 1659, 1579, 1452, 1420, 1260, 1097, 1073, 1027, 746, 692 (cm^{-1}); HRMS (ESI): ([M-H]$^-$) calcd for $C_{14}H_{12}NOS^-$: 242.0645; found: 242.0647.

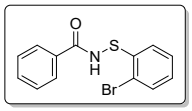
N-[(2-chlorophenyl)thio]benzamide (3aj)[3]



Yellow solid (46.4 mg, 88% yield), mp 129−131 °C, (lit.[3] mp 129–131 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.88–7.86 (m, 2H), 7.82 (s, 1H), 7.54–7.50 (m, 1H), 7.40–7.36

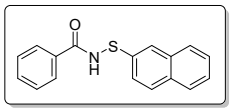
(m, 2H), 7.26–7.24 (m, 1H), 7.16–7.04 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 169.4, 137.3, 132.8, 132.7, 129.6, 128.9, 128.8, 127.9, 127.4, 127.0, 124.4.

N-[(2-bromophenyl)thio]benzamide (3ak)



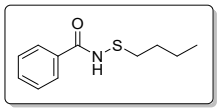
Yellow solid (49.9 mg, 81% yield), mp 147−149 °C. ^{1}H NMR (Acetone-d_6, 400 MHz) δ 9.34 (s, 1H), 8.08–8.06 (m, 2H), 7.66–7.62 (m, 1H), 7.56–7.53 (m, 3H), 7.37–7.33 (m, 1H), 7.16 (dd, J = 8.0, 1.4 Hz, 1H), 7.12–7.08 (m, 1H); ^{13}C NMR (Acetone-d_6, 100 MHz) δ 183.3, 141.2, 134.3, 133.5, 133.3, 129.6, 129.0, 128.9, 127.6, 124.4, 116.6; IR (KBr): 3250, 2925, 1660, 1580, 1451, 1427, 1259, 1097, 1019, 865, 745, 691 (cm^{-1}); HRMS (ESI): ([M+H]$^+$) calcd for $C_{13}H_{11}BrNOS^+$: 307.9739; found: 307.9743.

N-(naphthalen-2-ylthio)benzamide (3al)[5]



Yellow solid (35.8 mg, 64% yield), mp 152−154 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.33 (s, 1H), 8.02–8.00 (m, 2H), 7.91 (d, J = 8.7 Hz, 1H), 7.85 (dd, J = 18.3, 7.9 Hz, 2H), 7.70 (d, J = 1.4 Hz, 1H), 7.65–7.62 (m, 1H), 7.57–7.53 (m, 2H), 7.52–7.44 (m, 2H), 7.40 (dd, J = 8.7, 1.9 Hz, 1H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 168.4, 137.2, 133.11, 133.08, 132.3, 131.3, 128.7, 128.6, 128.0, 127.7, 127.0, 126.9, 125.7, 121.9, 120.9; IR (KBr): 3439, 2923, 1675, 1454, 1430, 1260, 1053, 1027, 1007, 822, 761, 706 (cm^{-1}); HRMS (ESI): ([M+H]$^+$) calcd for $C_{17}H_{14}NOS^+$: 280.0791; found: 280.0799.

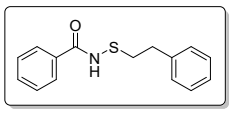
N-(butylthio)benzamide (3am)[4]



Light yellow viscous oil (26.8 mg, 64% yield). ^{1}H NMR (DMSO-d_6, 400 MHz) δ 9.42 (s, 1H), 7.85 (d, J = 7.3 Hz, 2H), 7.56–7.53 (m, 1H), 7.48–7.44 (m, 2H), 2.74 (t, J = 7.1 Hz, 2H), 1.55–1.48 (m, 2H), 1.43–1.34 (m, 2H), 0.85 (t, J = 7.3 Hz, 3H); ^{13}C

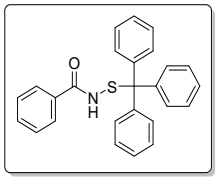
NMR (DMSO-d_6, 100 MHz) δ 169.2, 134.1, 132.2, 128.9, 128.1, 37.7, 29.9, 21.5, 14.0; IR (KBr): 3388, 3182, 2962, 1645, 1577, 1405, 1260, 1092, 1024, 796, 685, 634 (cm^{-1}); HRMS (ESI): ($[M+H]^+$) calcd for $C_{11}H_{16}NOS^+$: 210.0947; found: 210.0950.

N-(phenethylthio)benzamide (3an)



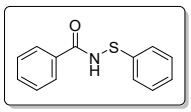
White solid (18.5 mg, 36% yield), mp 70−72 °C. ^{1}H NMR (Acetone-d_6, 400 MHz) δ 8.67 (s, 1H), 7.95–7.93 (m, 2H), 7.59–7.55 (m, 1H), 7.50–7.46 (m, 2H), 7.27 (d, J = 4.3 Hz, 4H), 7.21–7.16 (m, 1H), 3.10–3.06 (m, 2H), 2.98–2.94 (m, 2H); ^{13}C NMR (Acetone-d_6, 100 MHz) δ 169.7, 141.5, 135.3, 132.7, 129.6, 129.4, 129.3, 128.6, 127.1, 40.7, 35.5; IR (KBr): 3396, 2926, 2251, 1673, 1454, 1431, 1262, 1055, 1028, 1009, 821, 703 (cm^{-1}); HRMS (ESI): ($[M-H]^-$) calcd for $C_{15}H_{14}NOS^-$: 256.0802; found: 256.0800.

N-(tritylthio)benzamide (3ao)



White solid (20.6 mg, 26% yield), mp 137−139 °C. ^{1}H NMR (Acetone-d_6, 400 MHz) δ 8.15 (s, 1H), 7.92–7.90 (m, 2H), 7.53–7.49 (m, 1H), 7.45–7.40 (m, 8H), 7.32–7.28 (m, 6H), 7.25–7.21 (m, 3H); ^{13}C NMR(Acetone-d_6, 100 MHz) δ 167.1, 146.2, 136.8, 132.1, 129.8, 129.2, 128.5, 128.3, 127.5, 71.3; IR (KBr): 3443, 3057, 1735, 1676, 1507, 1477, 1275, 1029, 766, 751, 699, 611 (cm^{-1}); HRMS (ESI): ($[M-H]^-$) calcd for $C_{26}H_{20}NOS^-$:394.1271; found: 394.1276.

N-(phenylthio)benzamide (3ap)[2]



White solid (37.6 mg, 82% yield), mp 125−127 °C, (lit.[2] mp123–124 °C). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.88 (d, J = 7.4 Hz, 2H), 7.66 (s, 1H), 7.59–7.55 (m, 1H), 7.47–

7.44 (m, 2H), 7.36–7.29 (m, 4H), 7.24–7.20 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 169.3, 138.7, 133.3, 132.6, 129.2, 128.9, 127.8, 127.1, 125.7.

Reference

[1] Bizet, V., Buglioni, L., Bolm, C. Light-Induced Ruthenium-Catalyzed Nitrene Transfer Reactions: A Photochemical Approach towards *N*-Acyl Sulfimides and Sulfoximines. *Angew. Chem. Int. Ed.* **2014**, *53*, 5639–5642.

[2] Bao, M., Shimizu, M., Shimada, S., Tanaka M. Efficient Synthesis of *N*-acylarenesulfenamides by Acylation of Arenesulfenamides. *Tetrahedron* **2003**, *59*, 303–309.

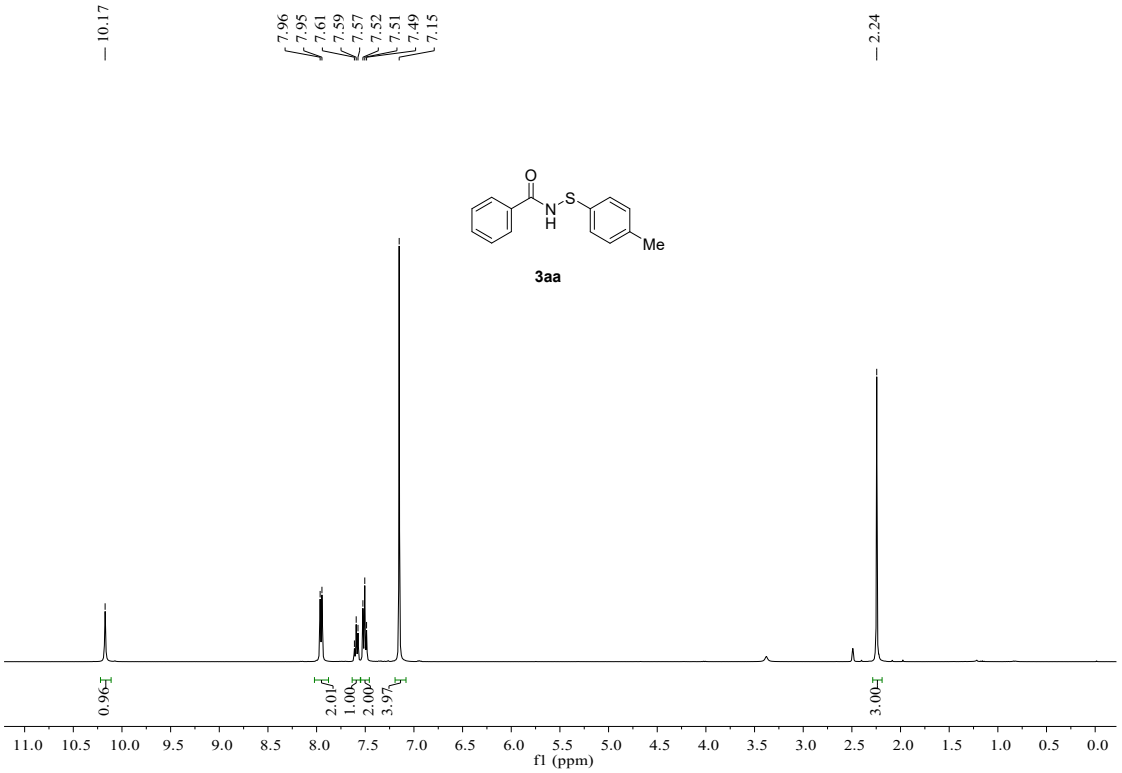
[3] Zhang, X. S., Zhang, X. H. Mild Synthesis of *N*-acylsulfenamides from Arylamides and Disulfides. *Phosphorus, Sulfur, and Silicon and the Related Elements*. **2016**, *191*, 89-94.

[4] Yang, G., Yuan, Y., Tian, Y., Zhang, S., Cui, X., Xia, B., Li, G., Tang Z. Synthesis of Chiral Sulfonimidoyl Chloride via Desymmetrizing Enantioselective Hydrolysis. *J. Am. Chem. Soc.* **2023**, *145*, 5439–5446.

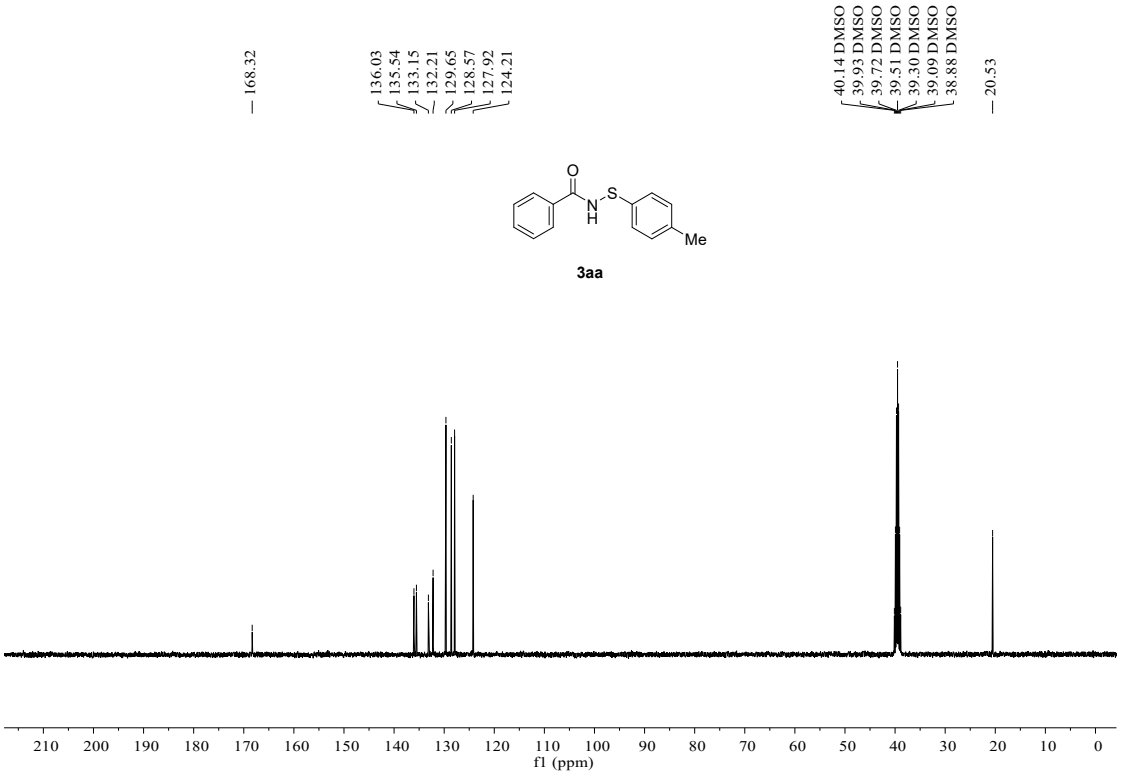
[5] Chen, Y., Fang, D. Huang, H., Nie, X., Zhang, S., Cui, X., Tang, Z. Li, G. Synthesis of Sulfilimines via Selective S–C Bond Formation in Water. *Org. Lett.* **2023**, *25*, 2134–2138.

Copies of ^{1}H and ^{13}C NMR spectra of products

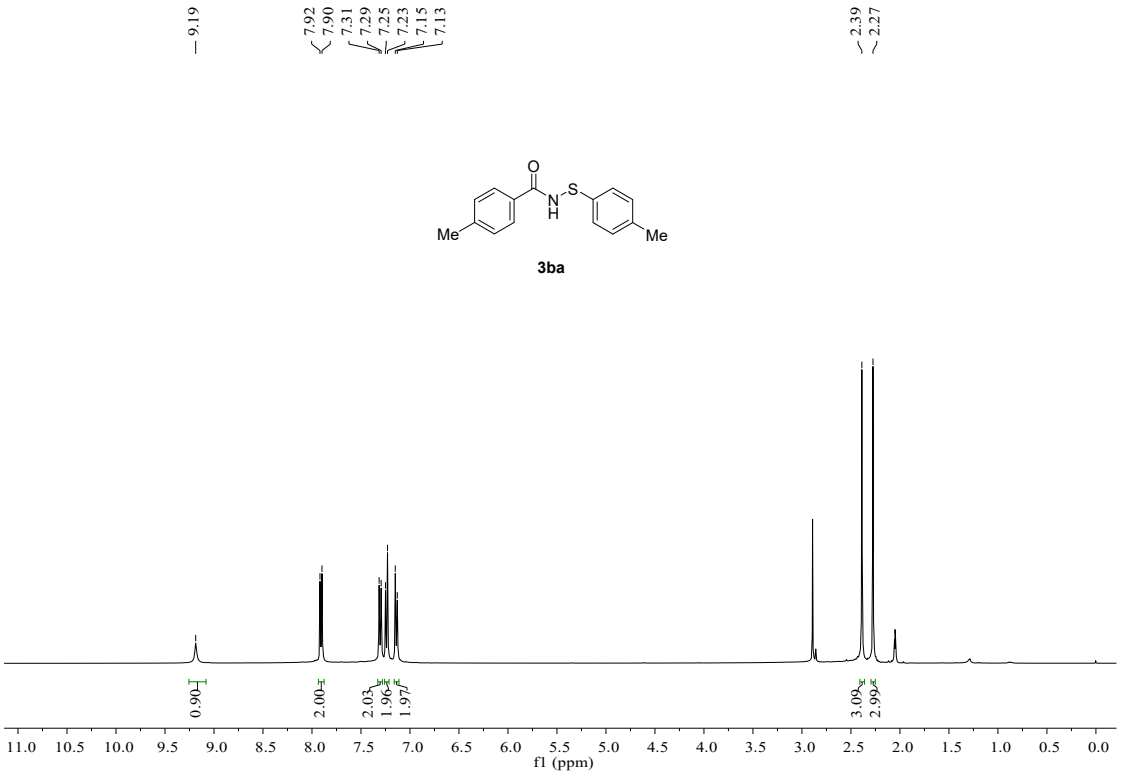
^{1}H NMR, 400 MHz, DMSO-d_6



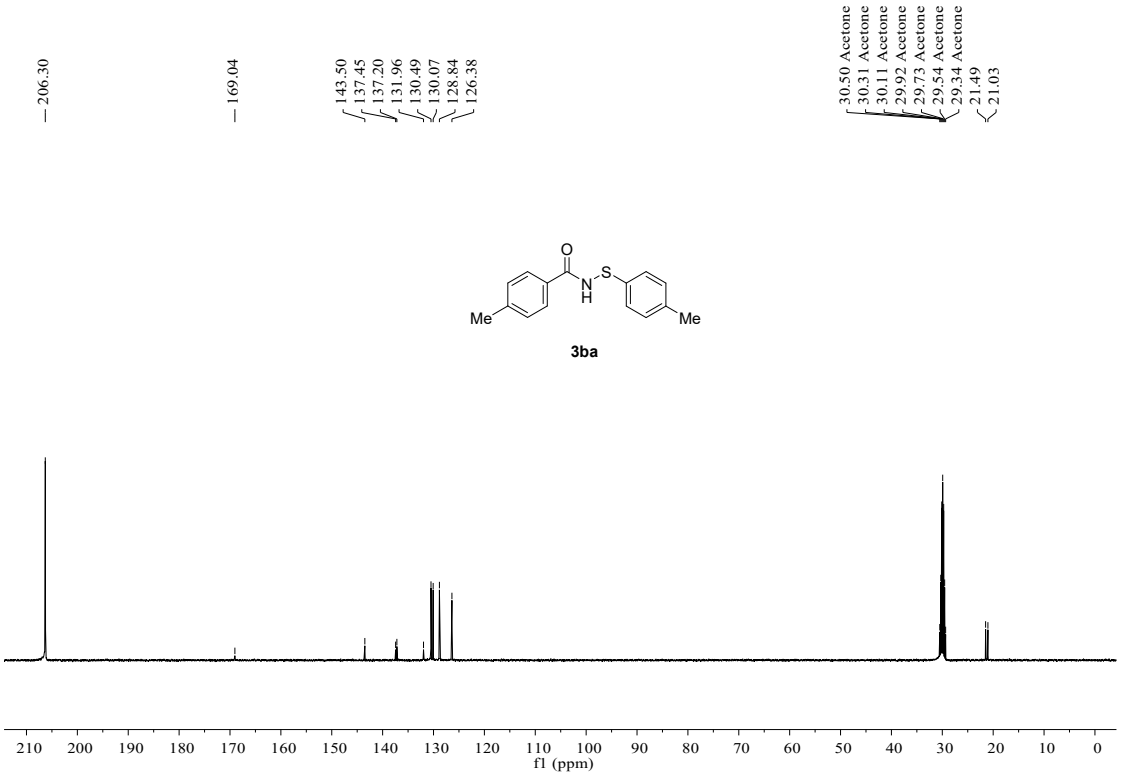
^{13}C NMR, 100 MHz, DMSO-d_6



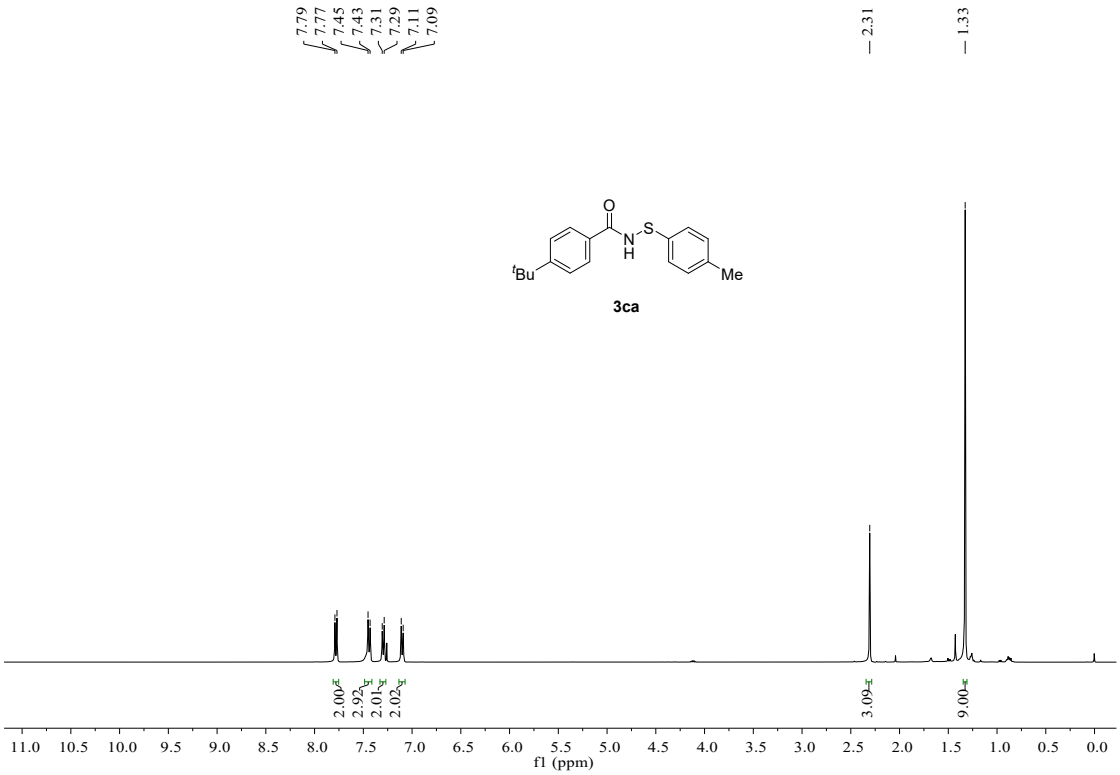
^{1}H NMR, 400 MHz, Acetone-d_6



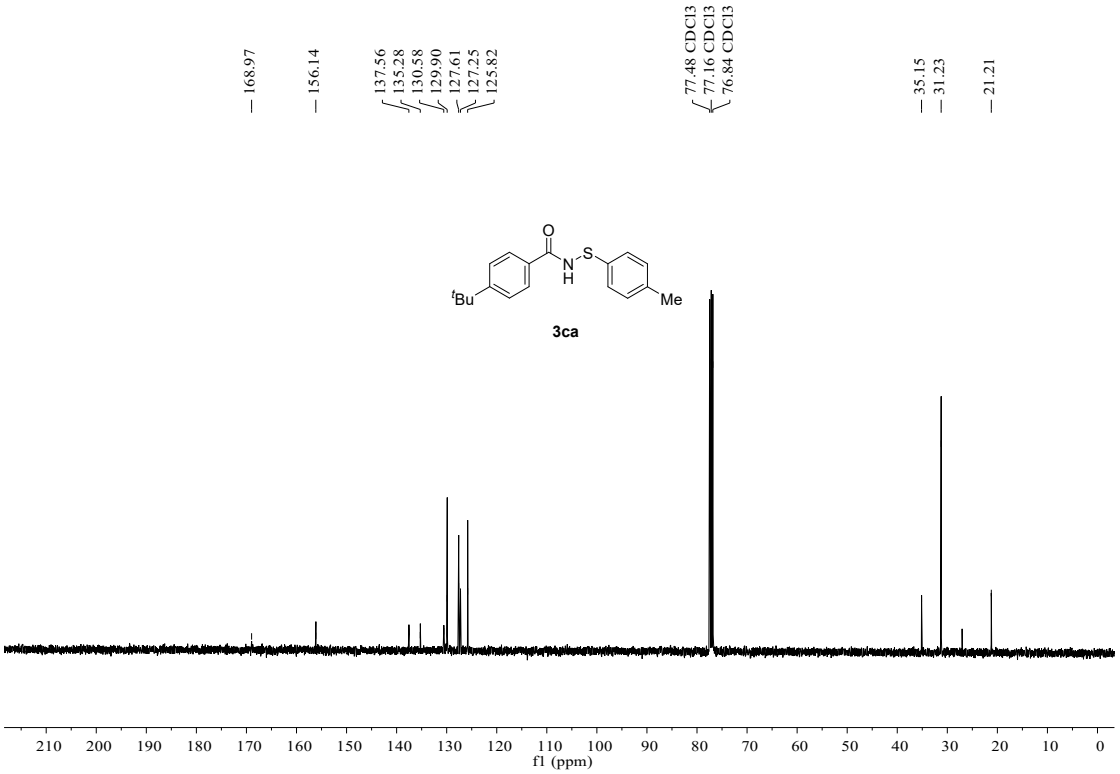
^{13}C NMR, 100 MHz, Acetone-d_6



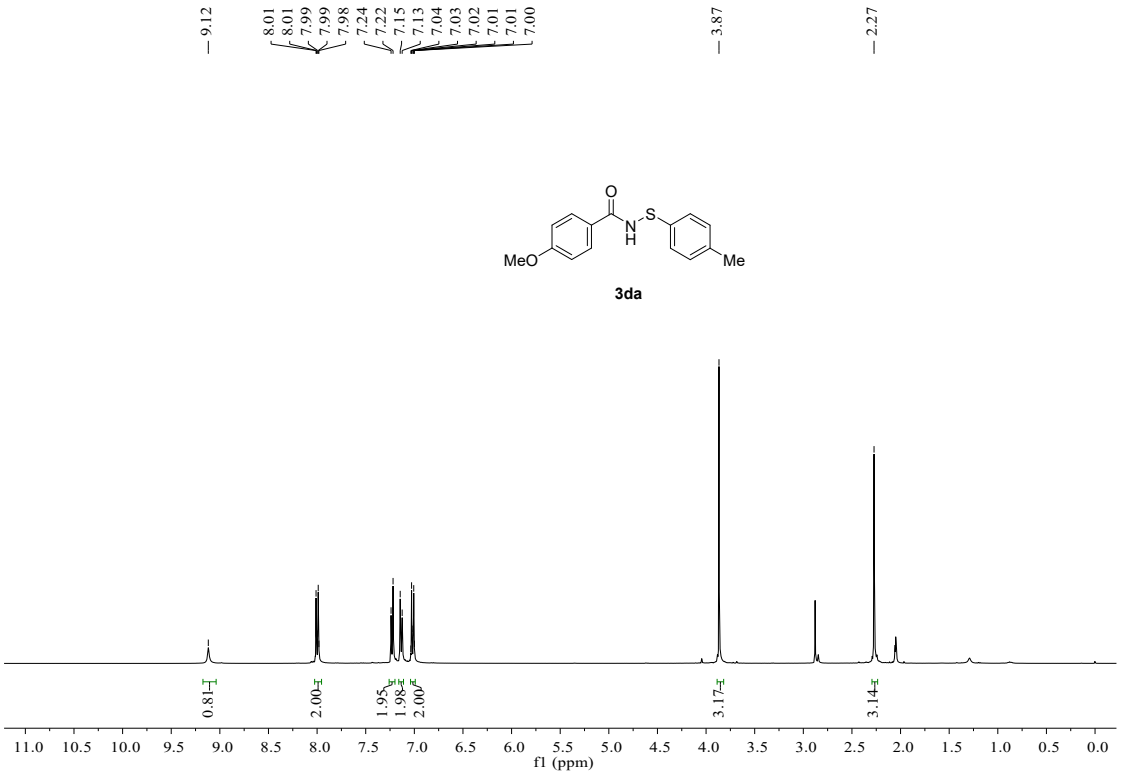
^{1}H NMR, 400 MHz, $CDCl_3$



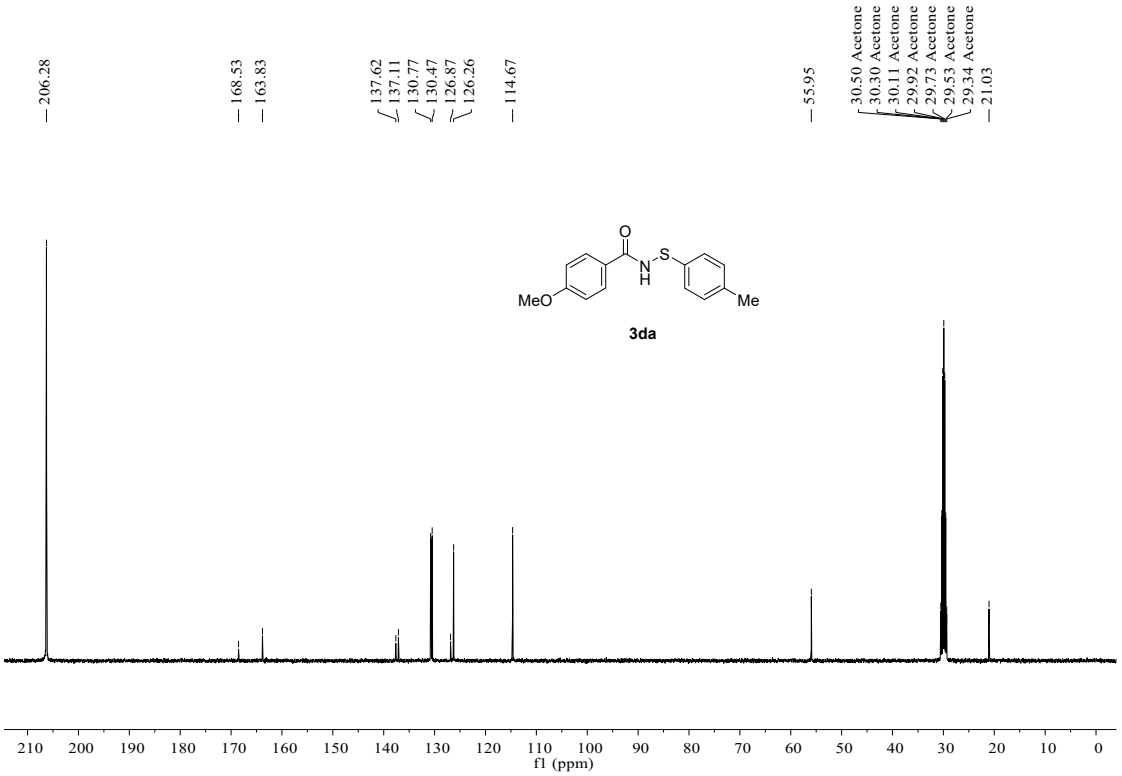
^{13}C NMR, 100 MHz, $CDCl_3$



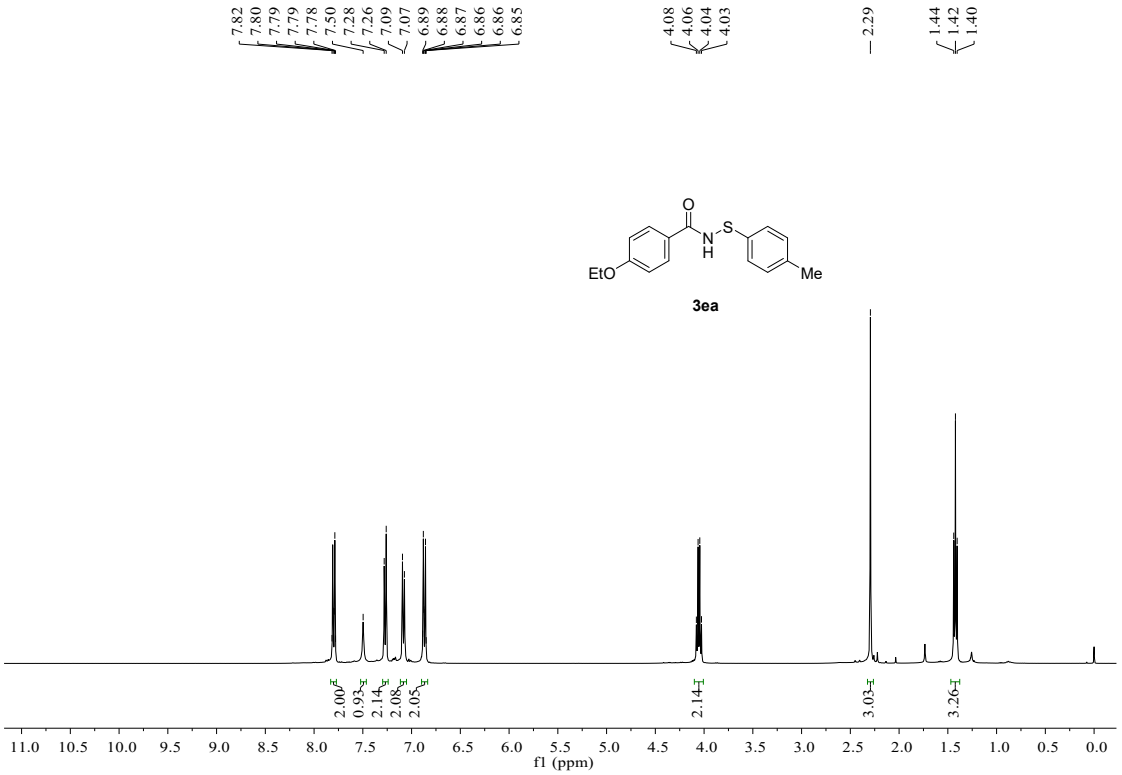
^{1}H NMR, 400 MHz, Acetone-d_6



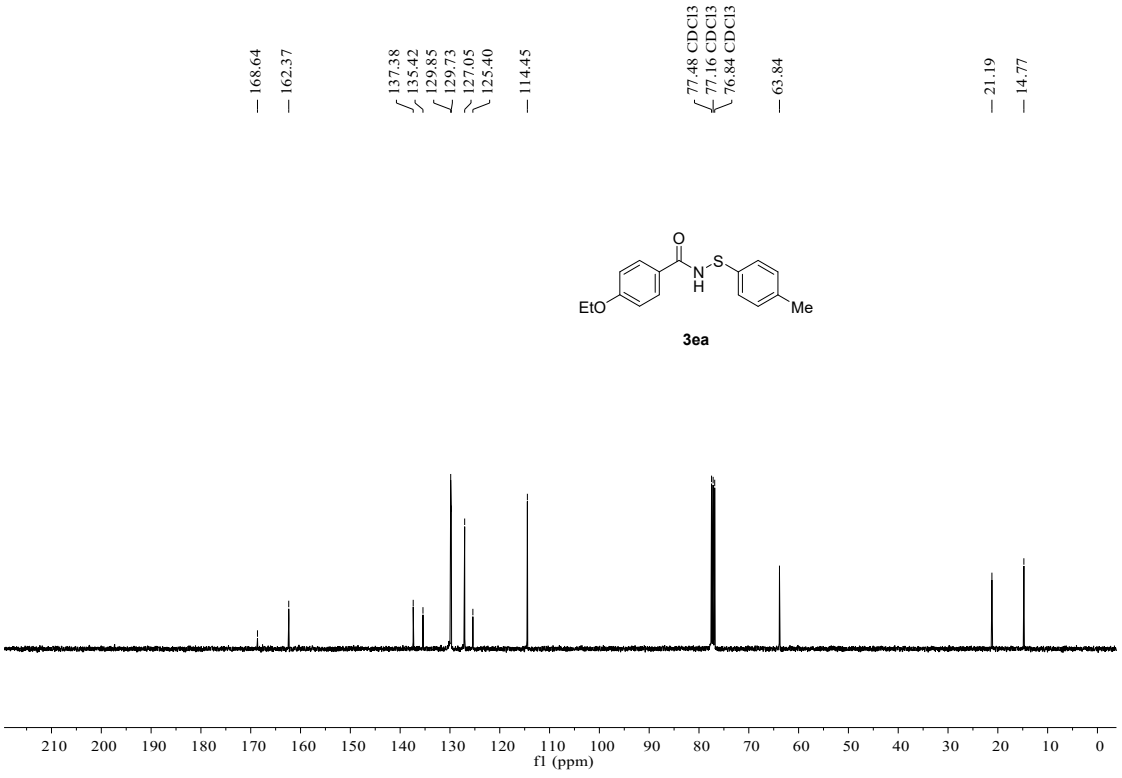
^{13}C NMR, 100 MHz, Acetone-d_6



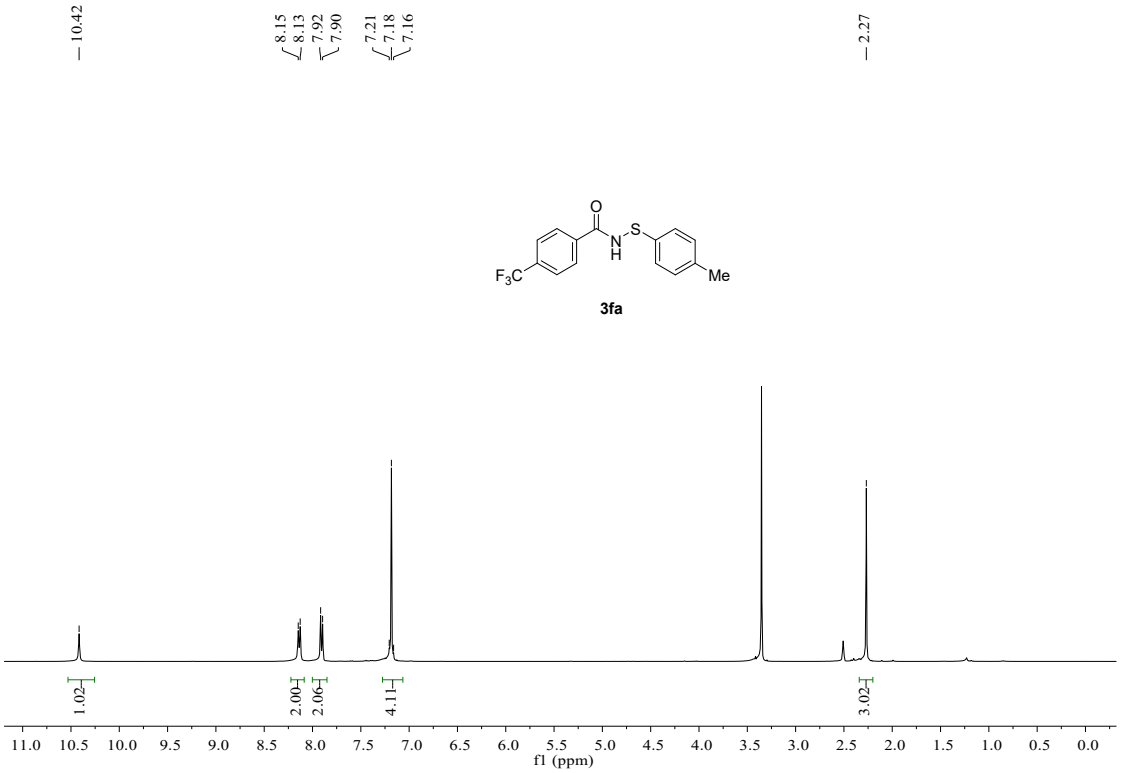
^{1}H NMR, 400 MHz, $CDCl_3$



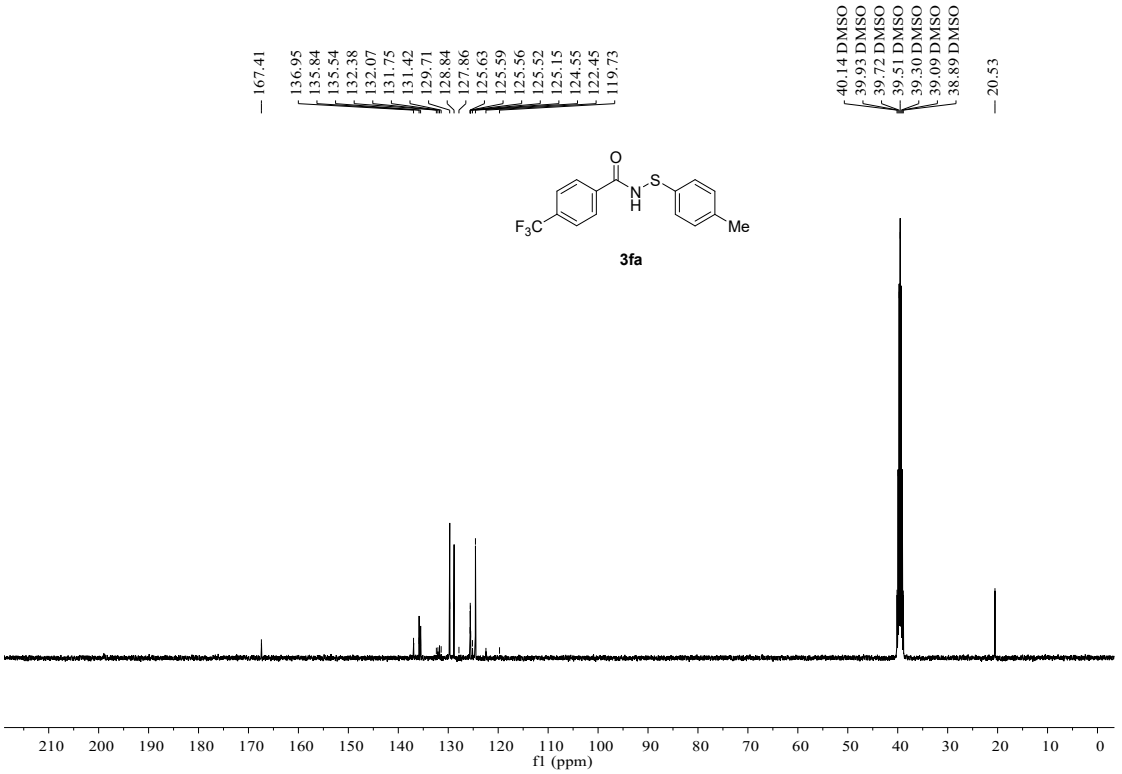
^{13}C NMR, 100 MHz, $CDCl_3$



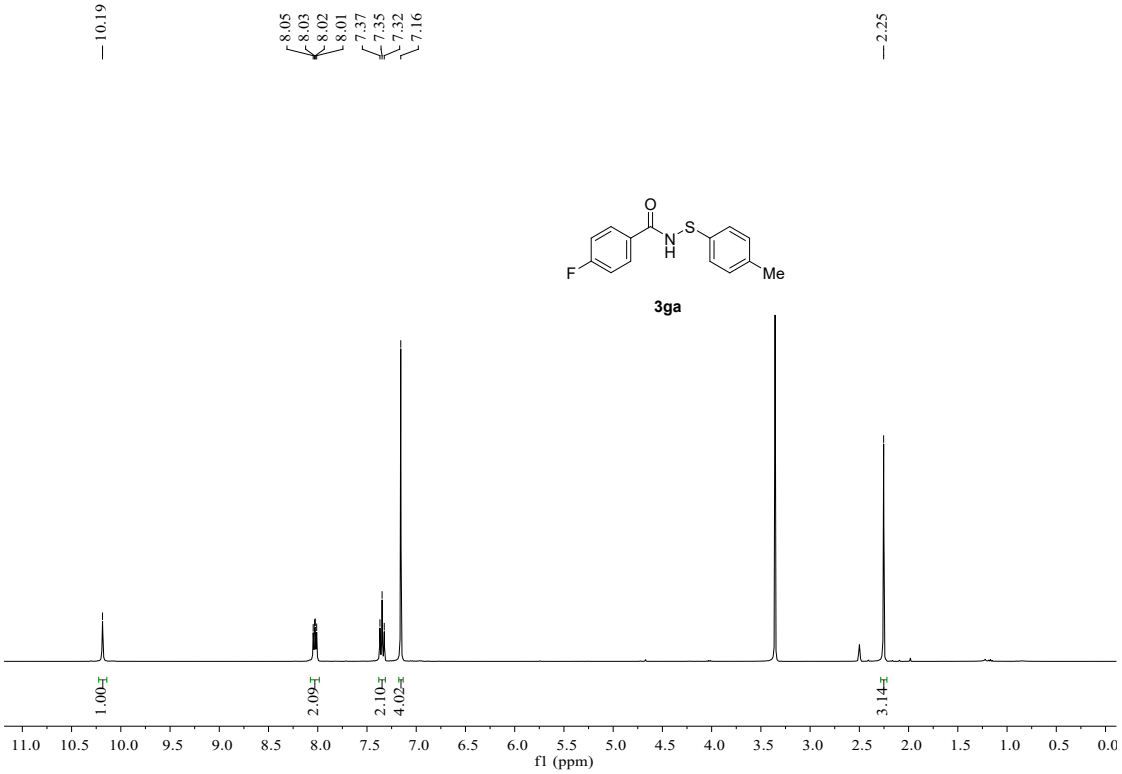
^{1}H NMR, 400 MHz, DMSO-d_6



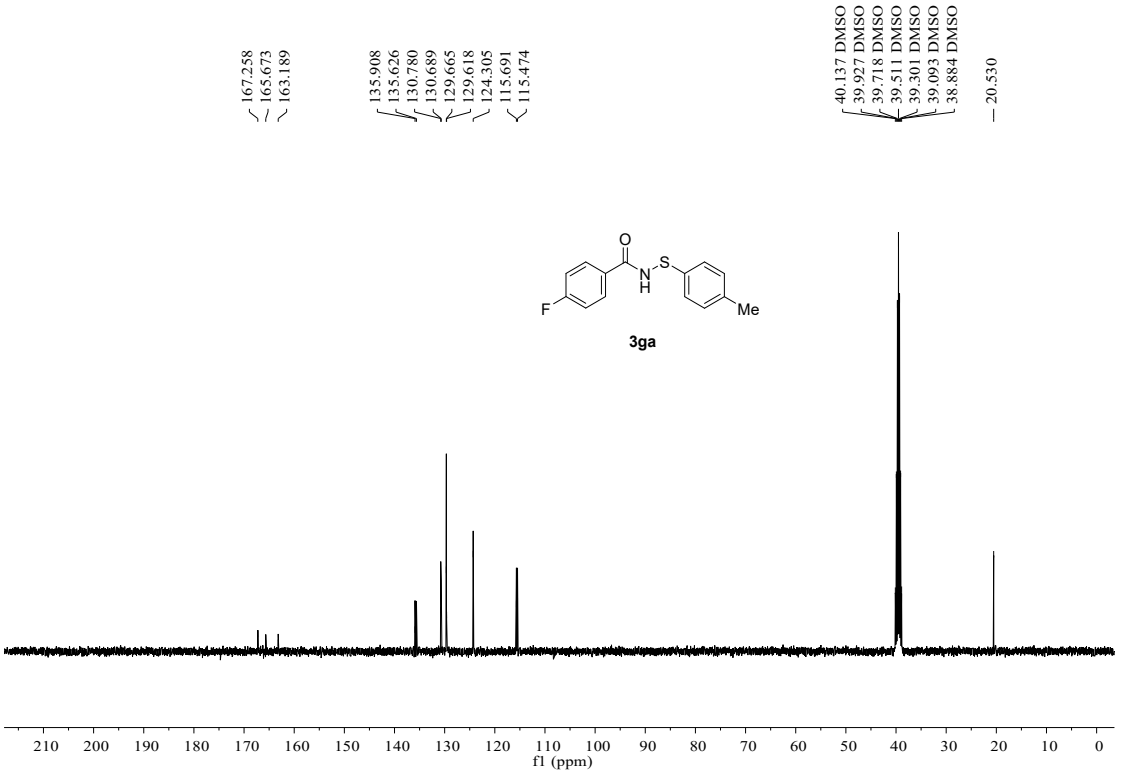
^{13}C NMR, 100 MHz, DMSO-d_6



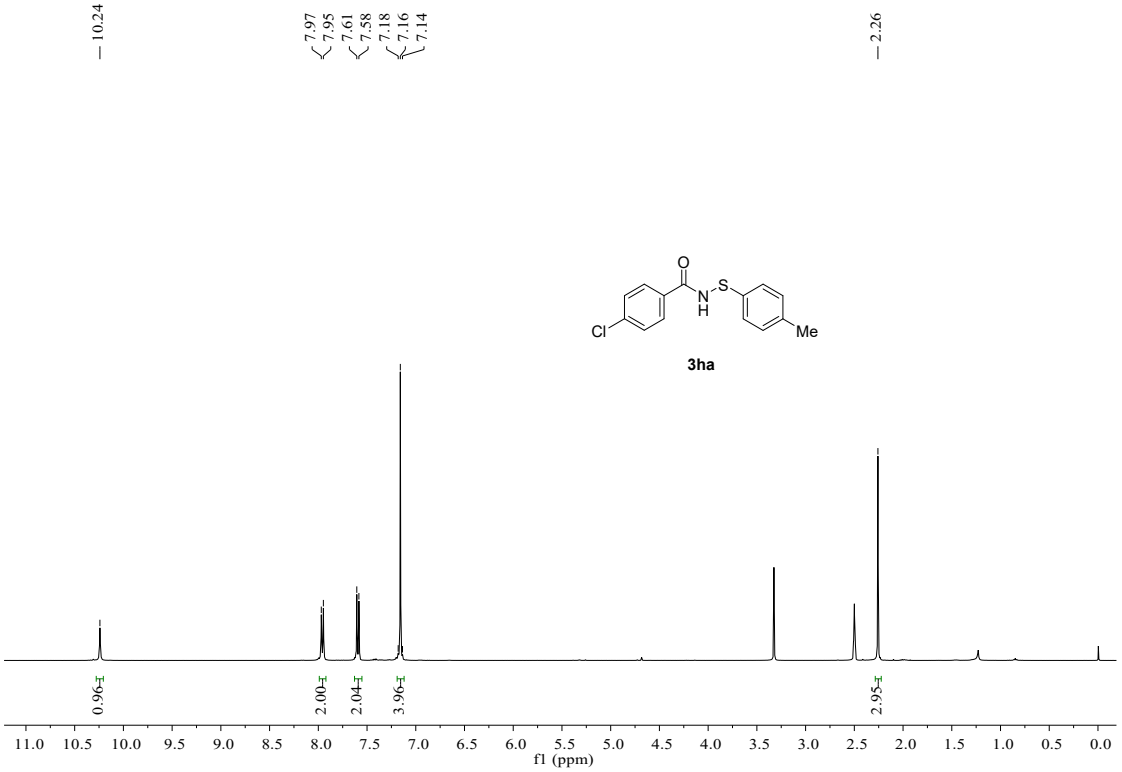
^{1}H NMR, 400 MHz, DMSO-d_6



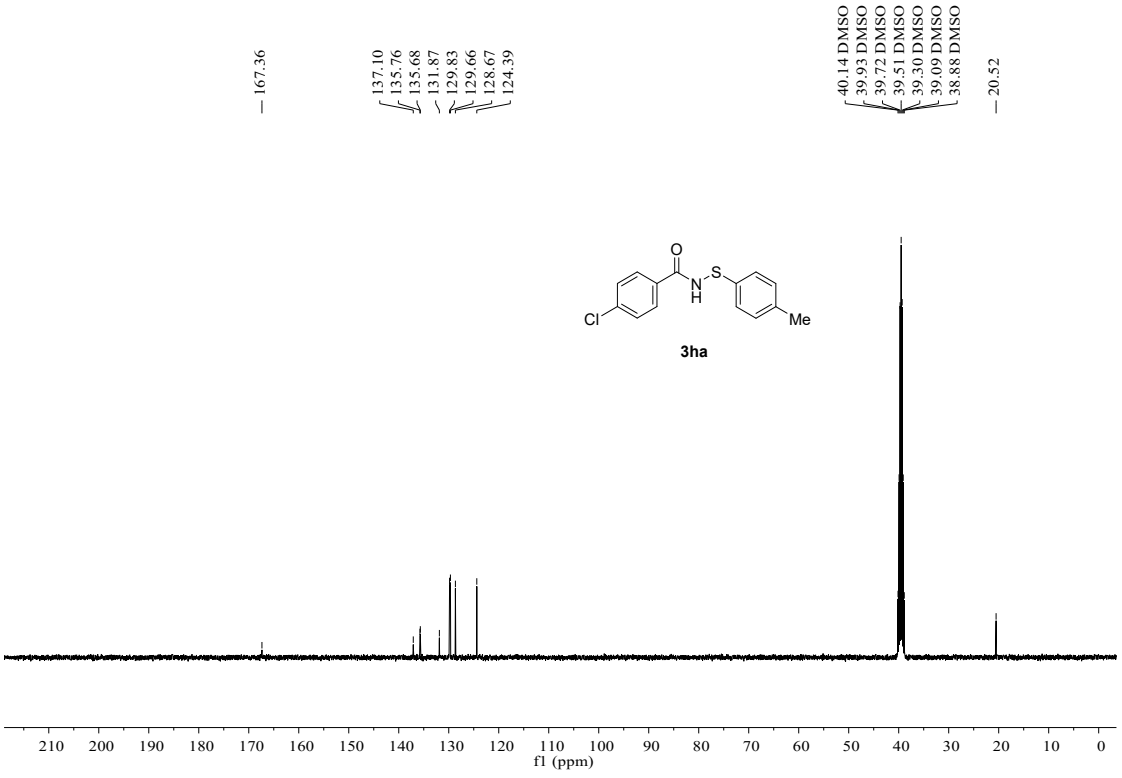
^{13}C NMR, 100 MHz, DMSO-d_6



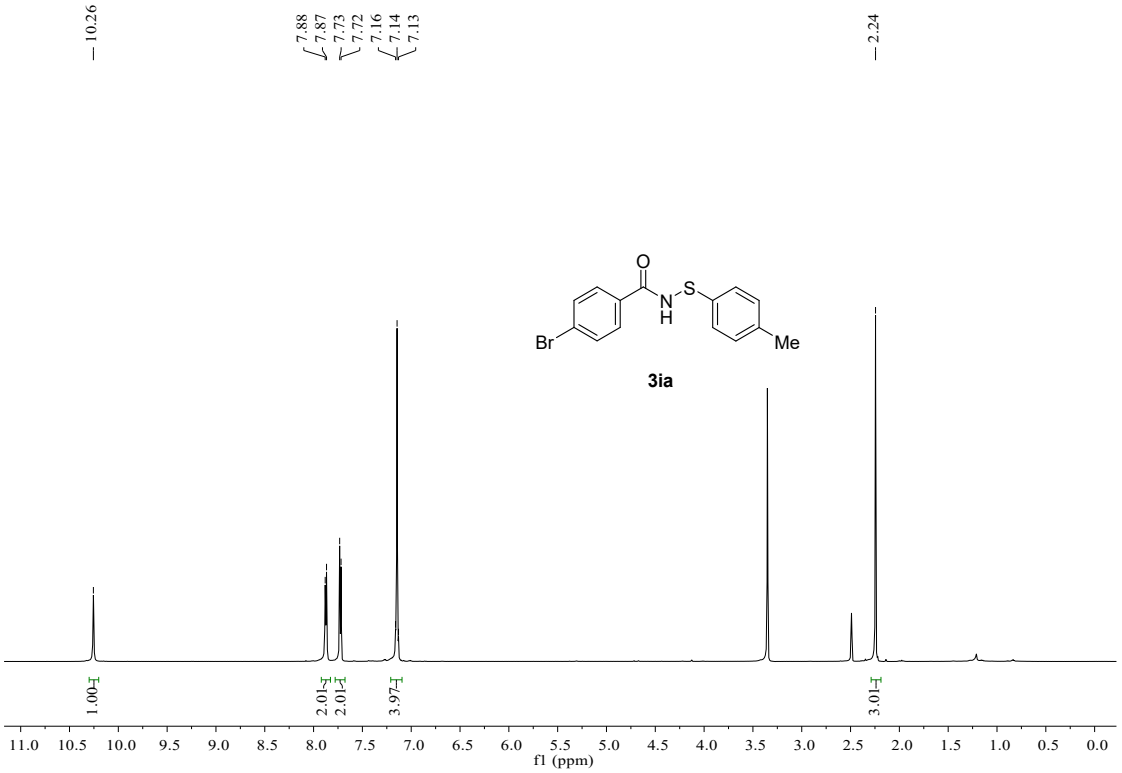
^{1}H NMR, 400 MHz, DMSO-d_6



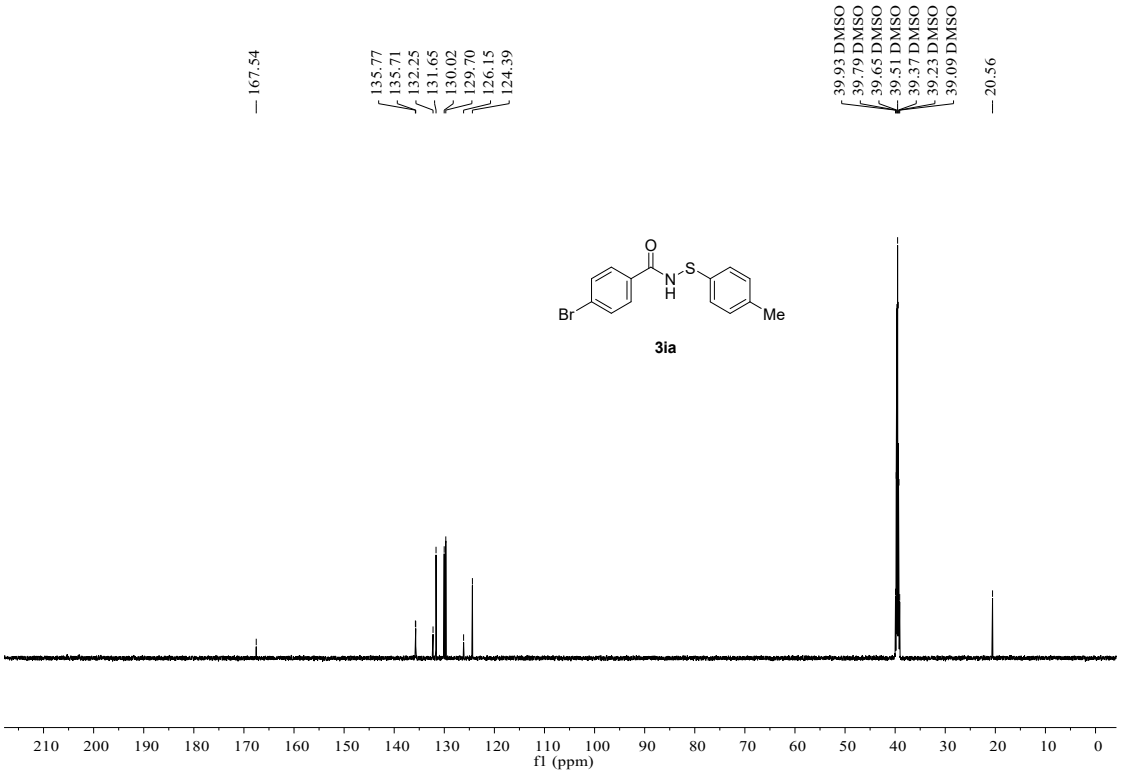
^{13}C NMR, 100 MHz, DMSO-d_6



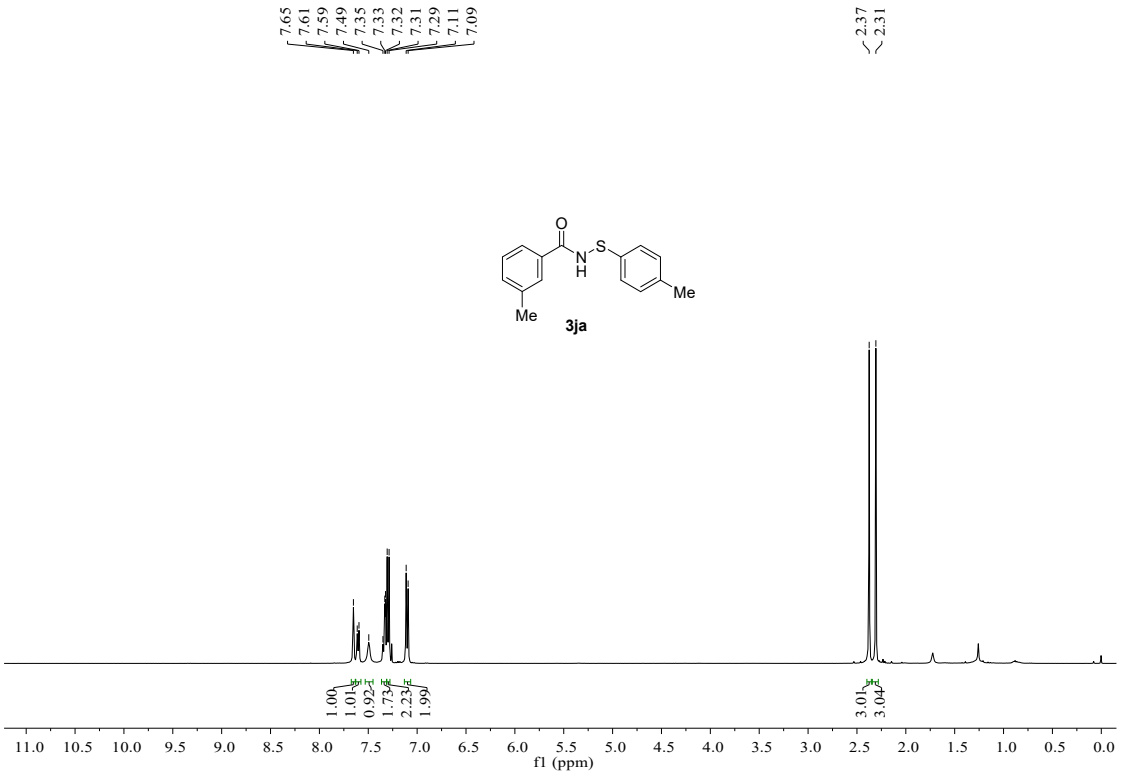
^{1}H NMR, 600 MHz, DMSO-d_6



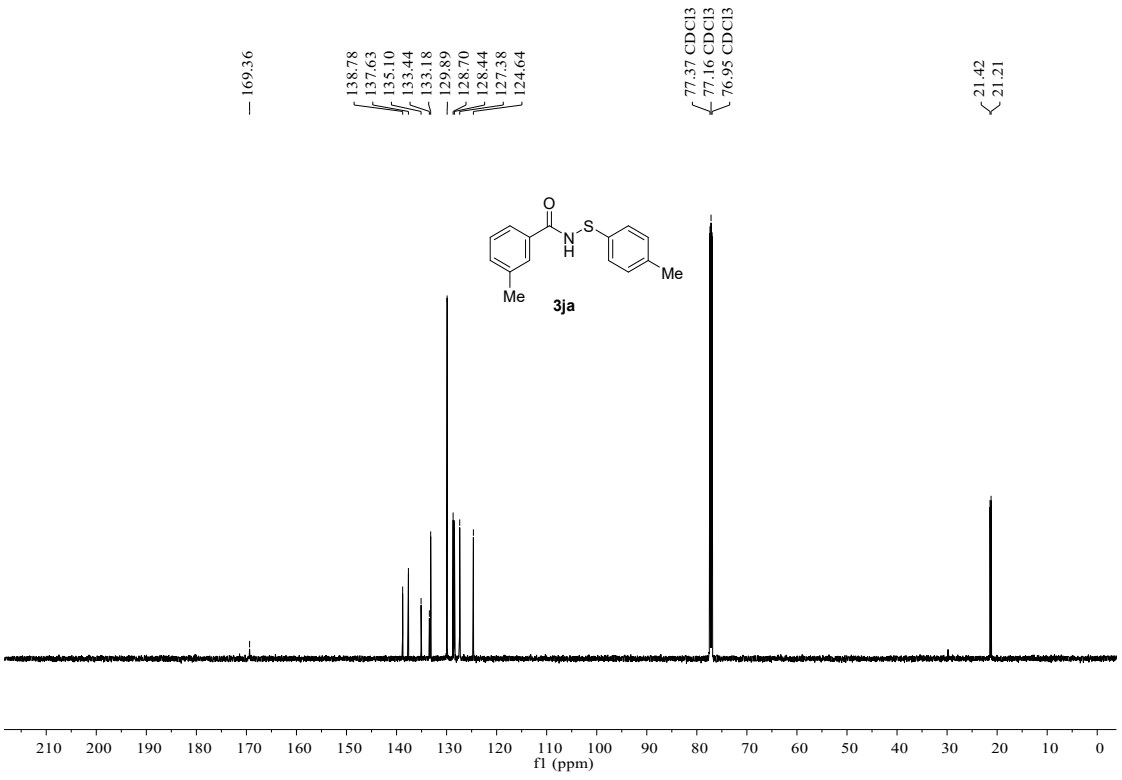
^{13}C NMR, 150 MHz, DMSO-d_6



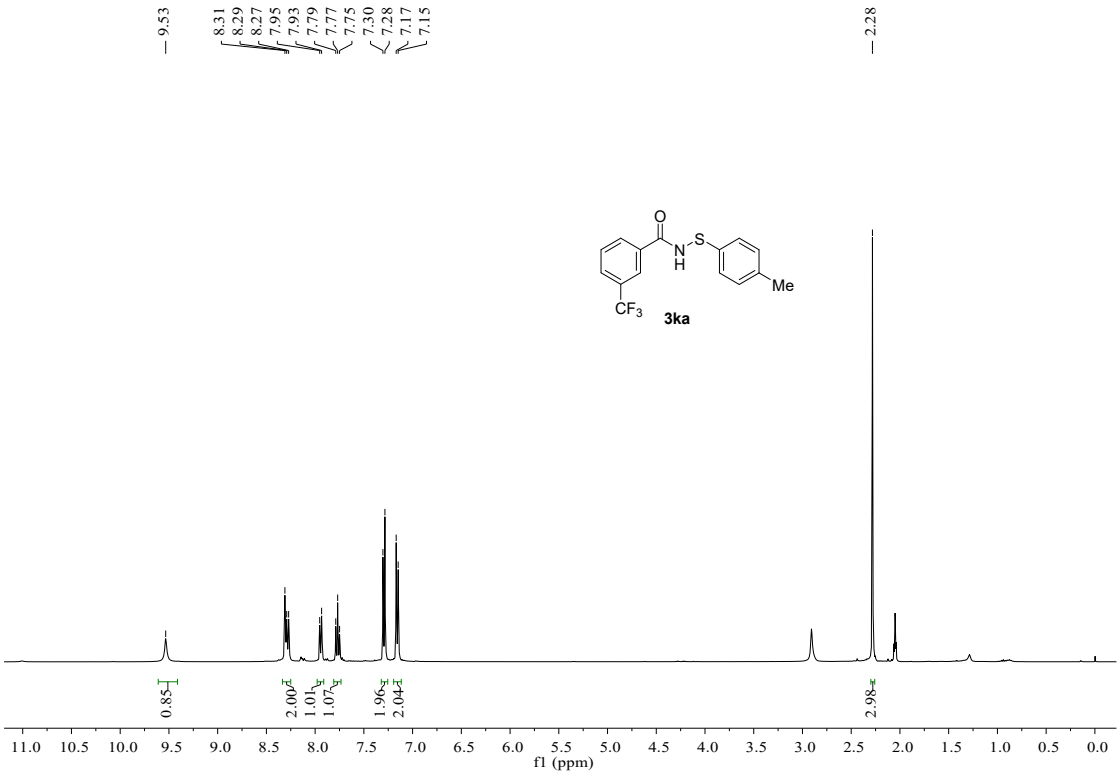
^{1}H NMR, 400 MHz, $CDCl_3$



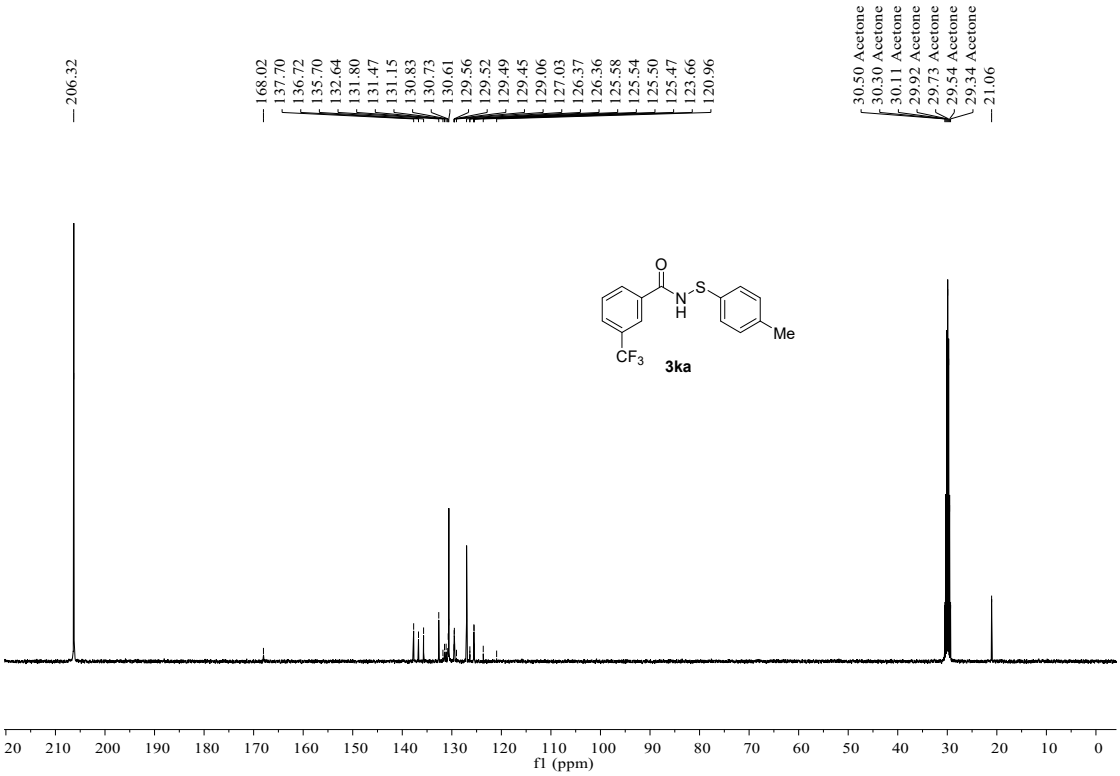
^{13}C NMR, 150 MHz, $CDCl_3$



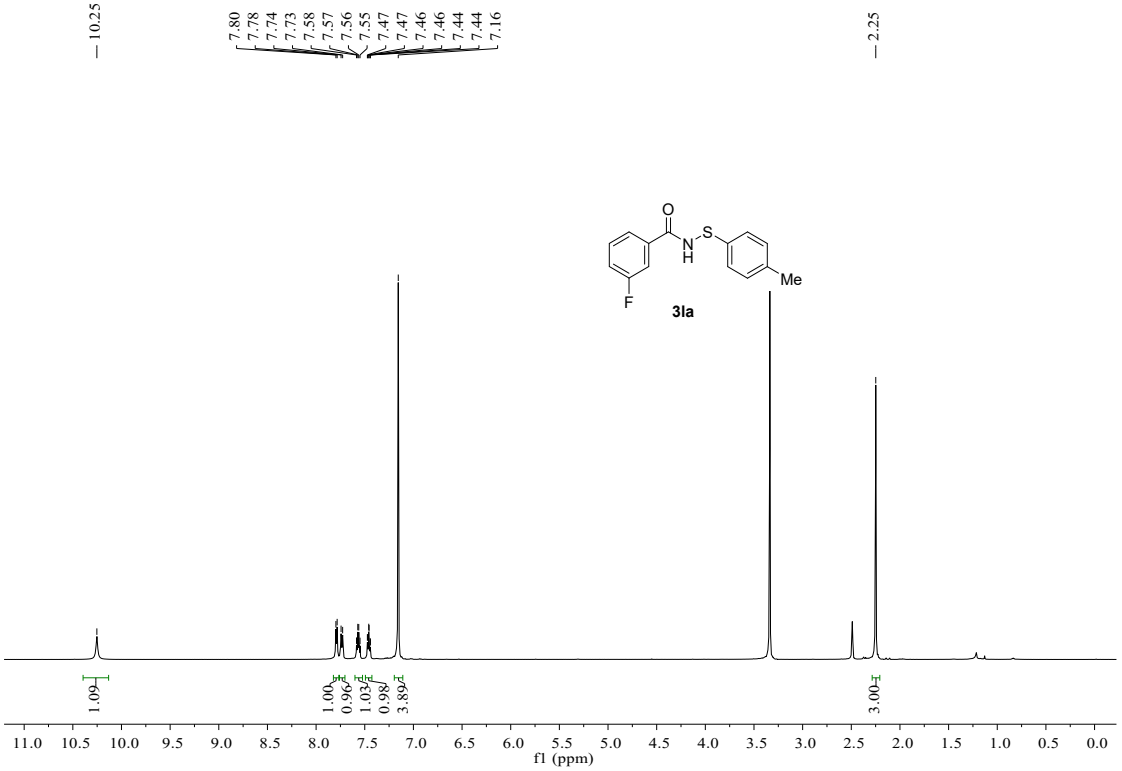
^{1}H NMR, 400 MHz, Acetone-d_6



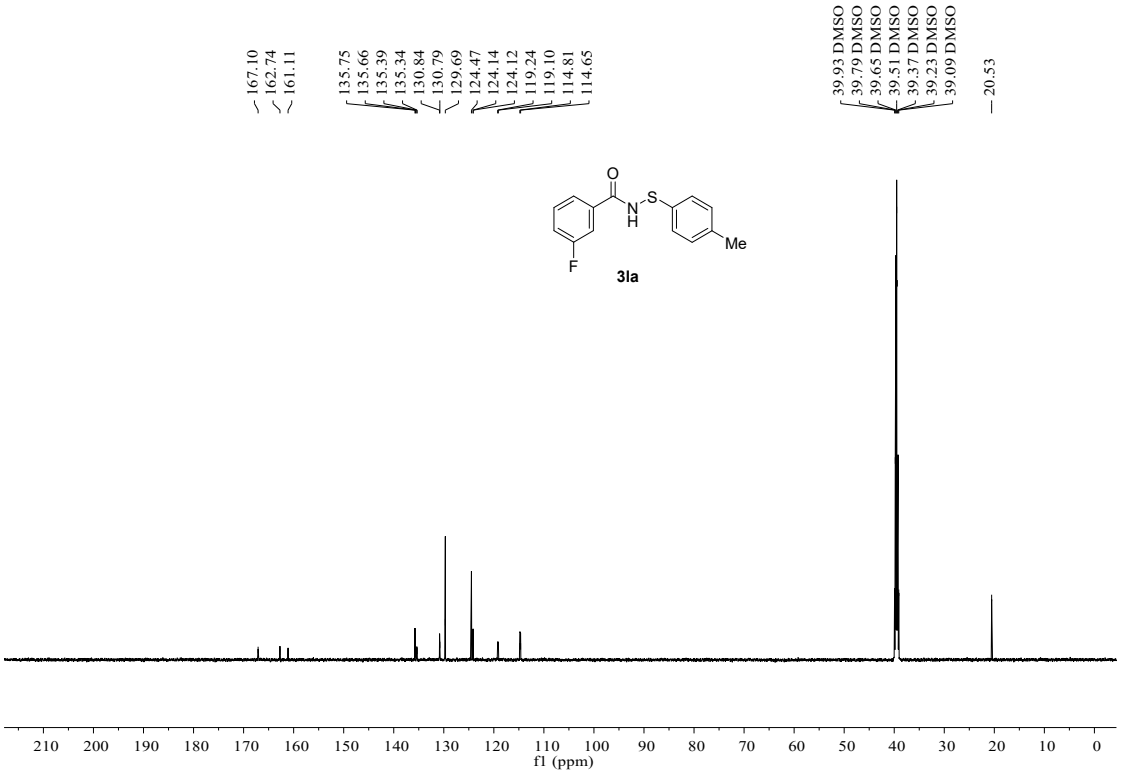
^{13}C NMR, 100 MHz, Acetone-d_6



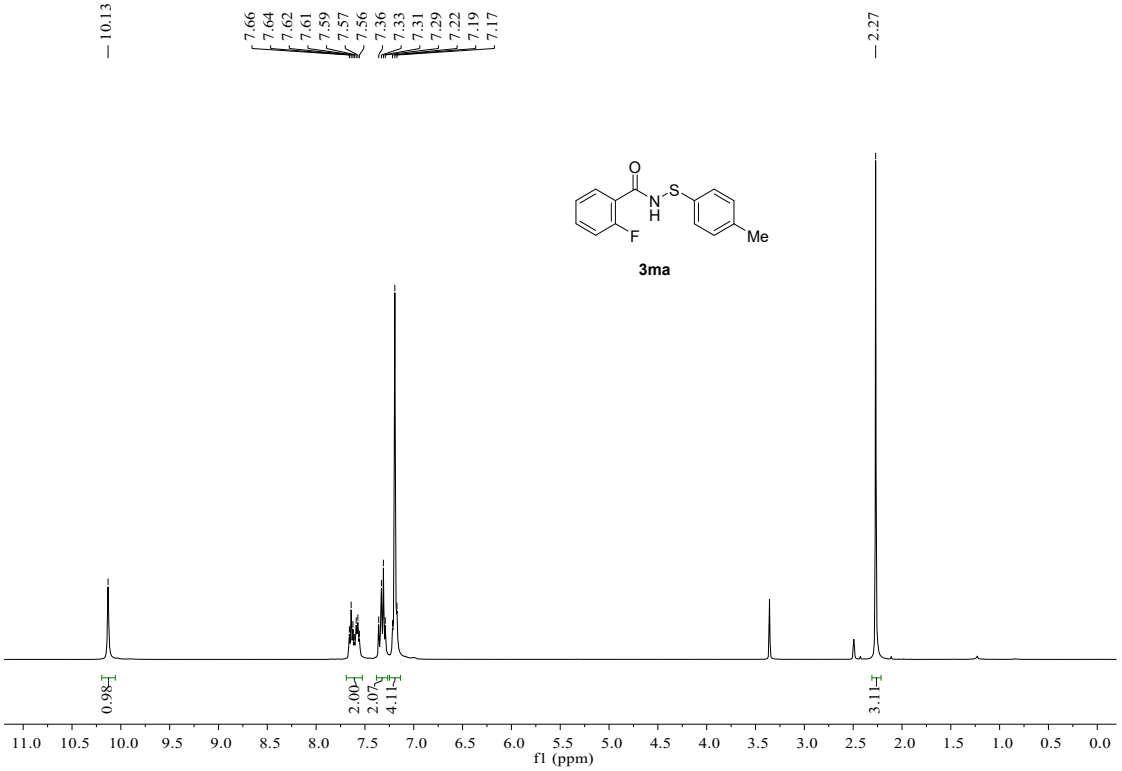
^{1}H NMR, 600 MHz, DMSO-d_6



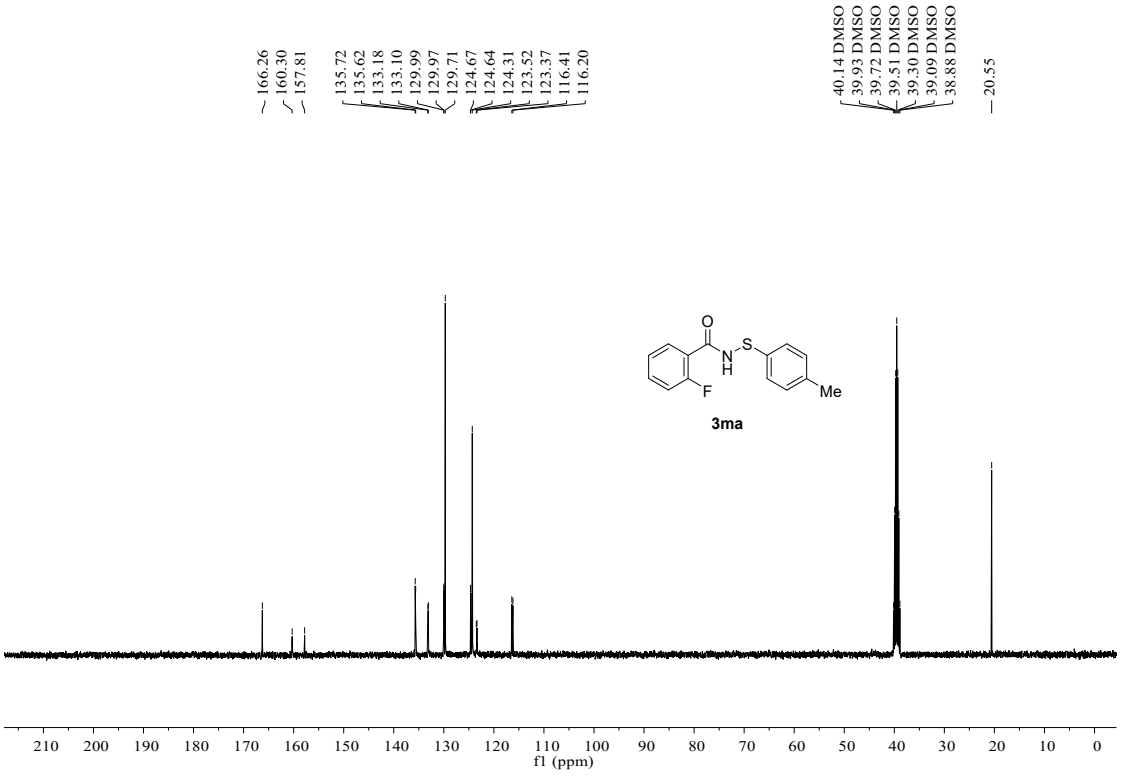
^{13}C NMR, 150 MHz, DMSO-d_6



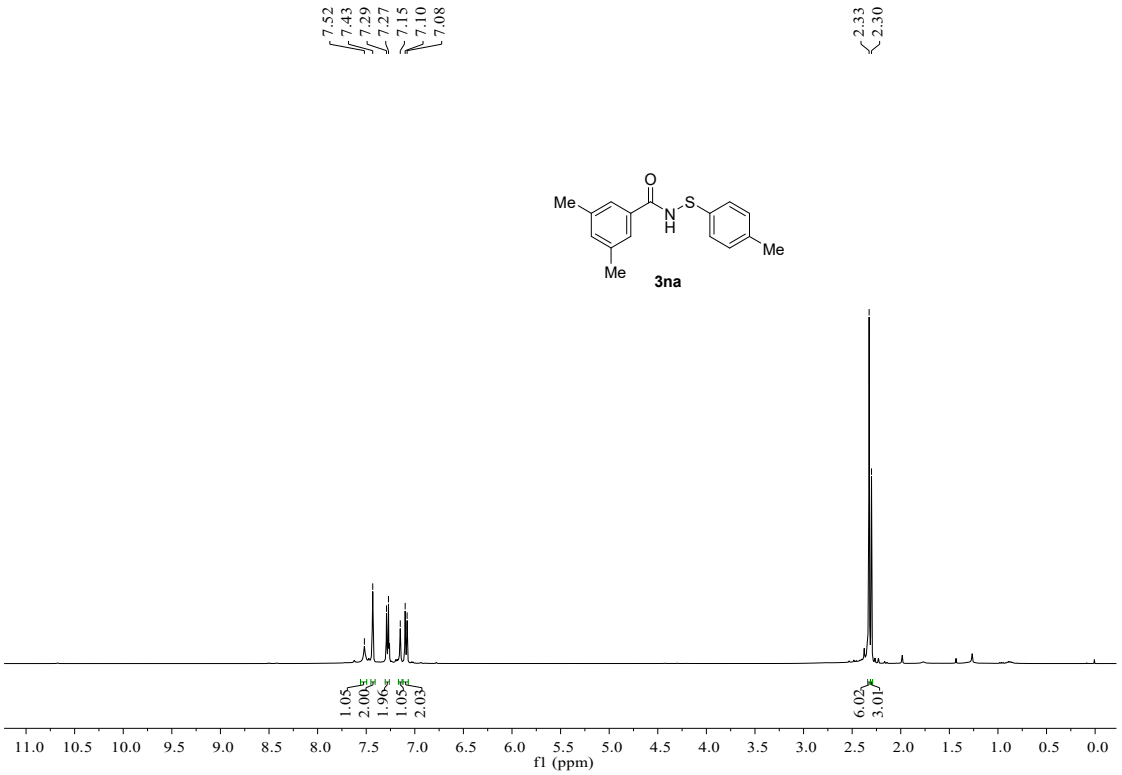
^{1}H NMR, 400 MHz, DMSO-d_6



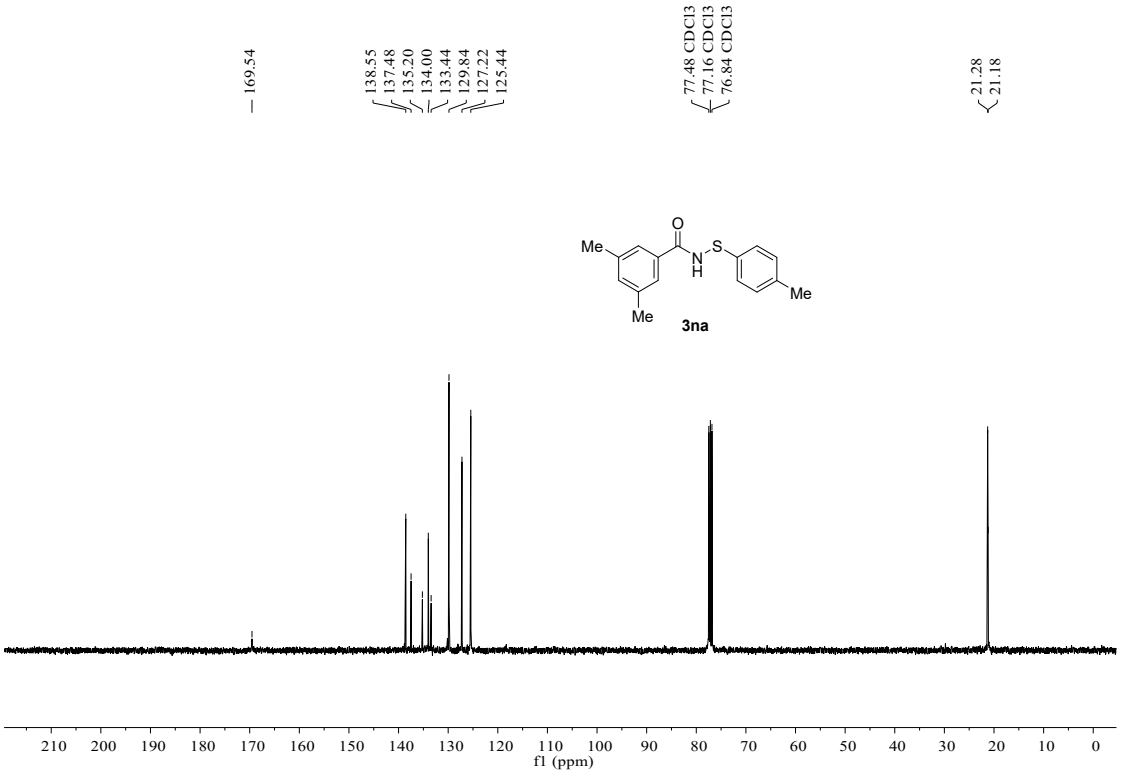
^{13}C NMR, 100 MHz, DMSO-d_6



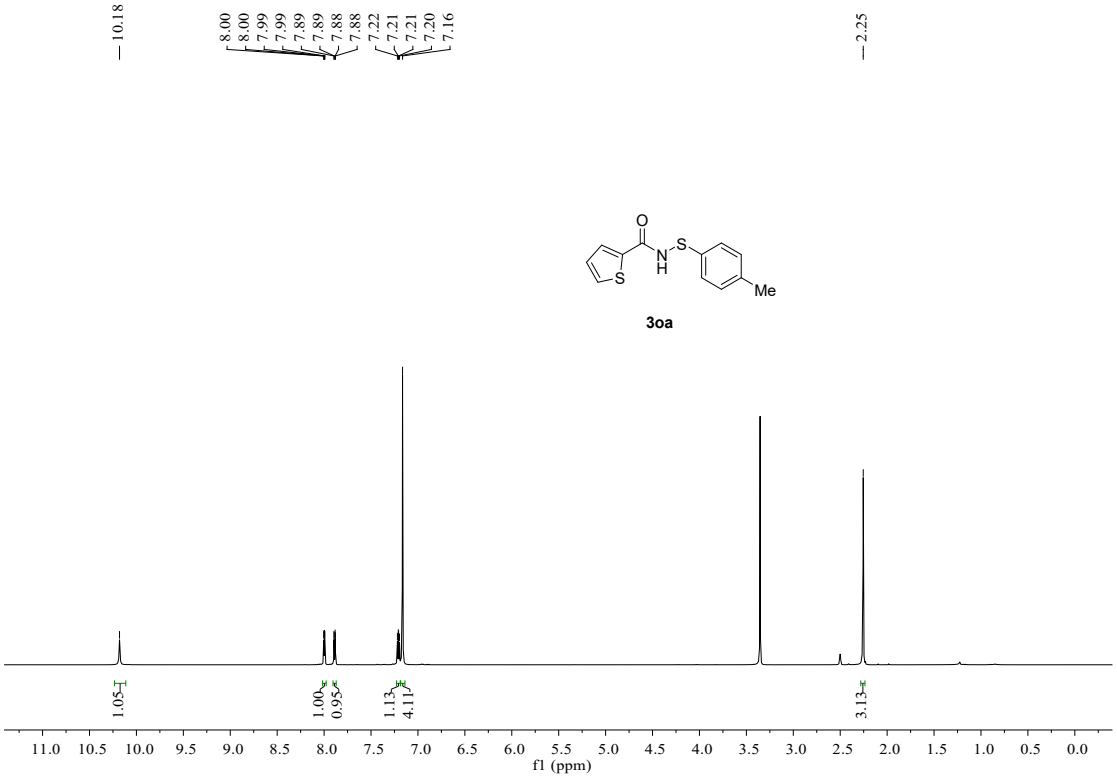
^{1}H NMR, 400 MHz, $CDCl_3$



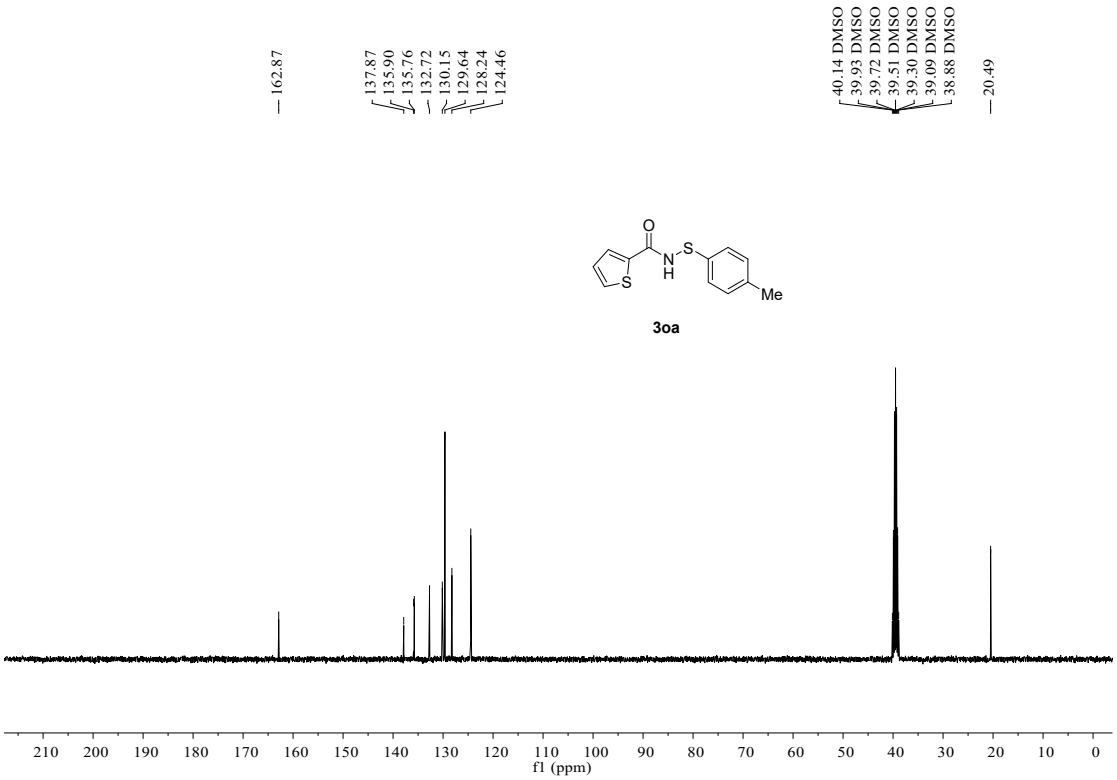
^{13}C NMR, 100 MHz, $CDCl_3$



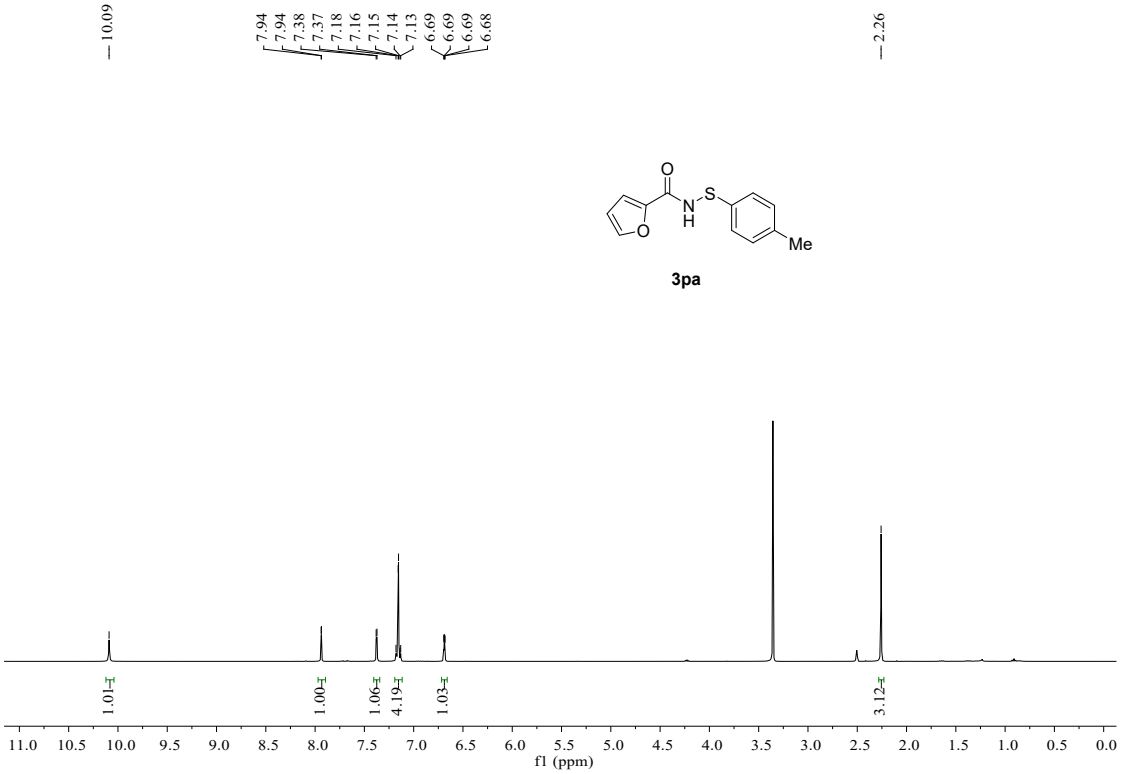
^{1}H NMR, 400 MHz, DMSO-d_6



^{13}C NMR, 100 MHz, DMSO-d_6

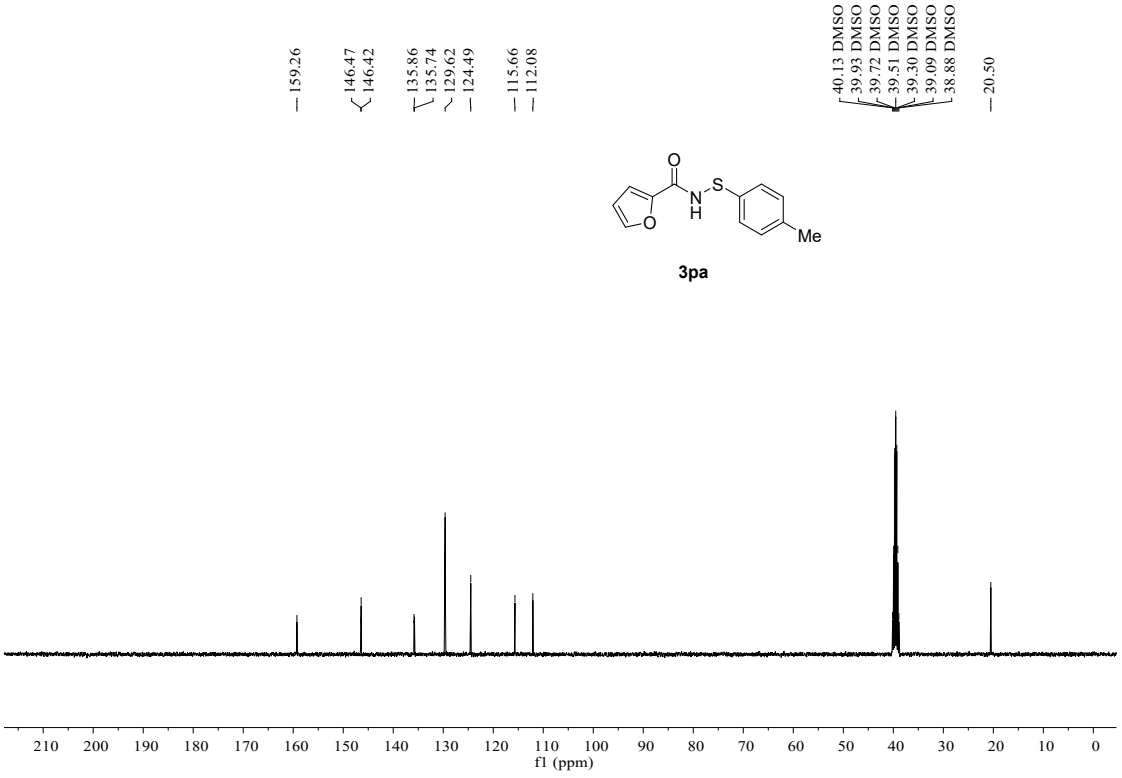


^{1}H NMR, 400 MHz, DMSO-d_6



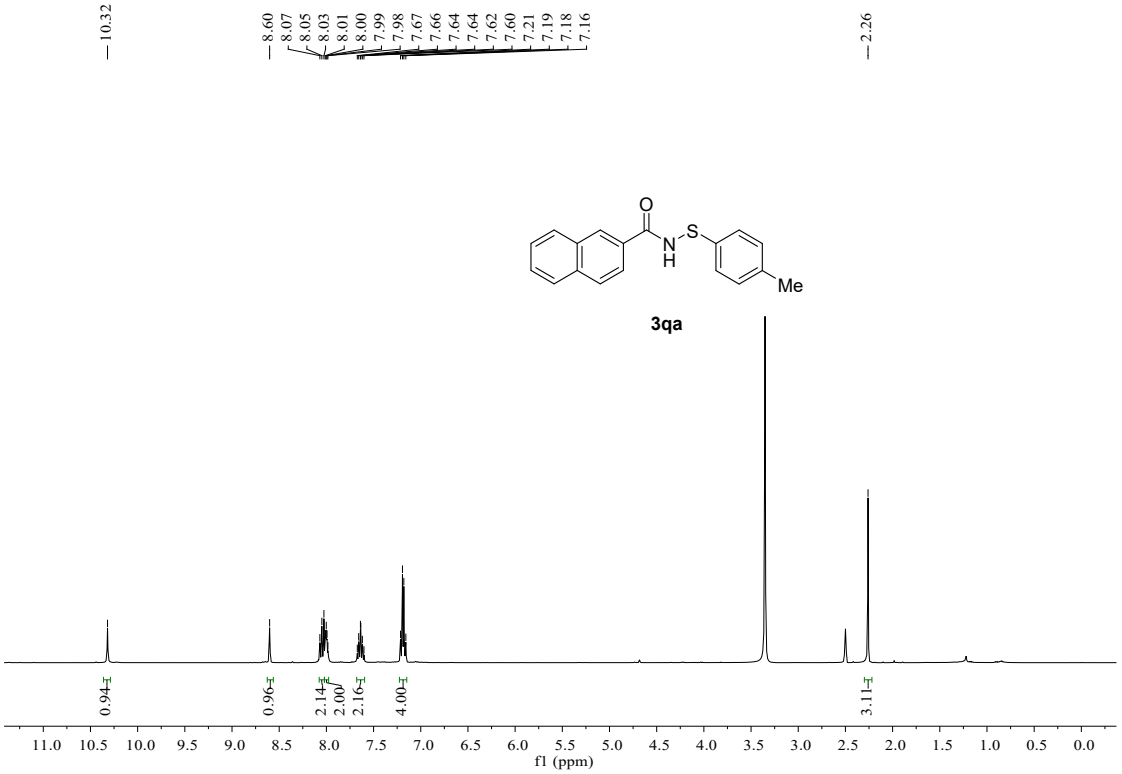
3pa

^{13}C NMR, 100 MHz, DMSO-d_6

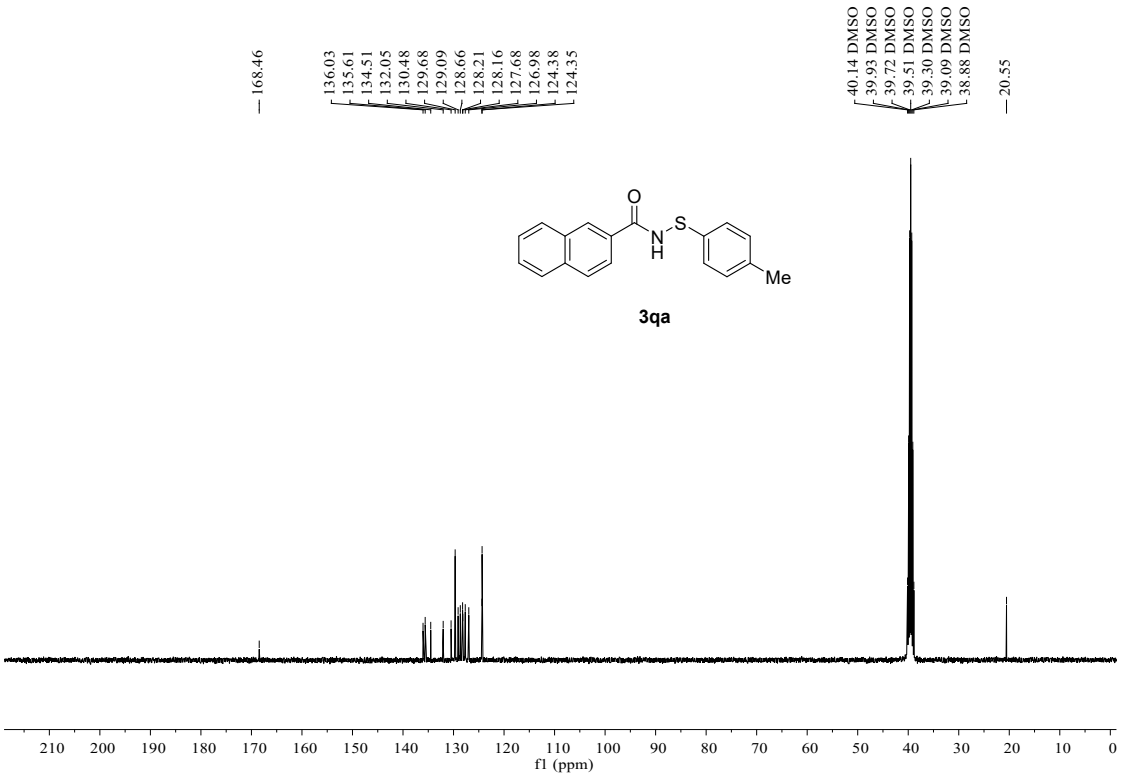


3pa

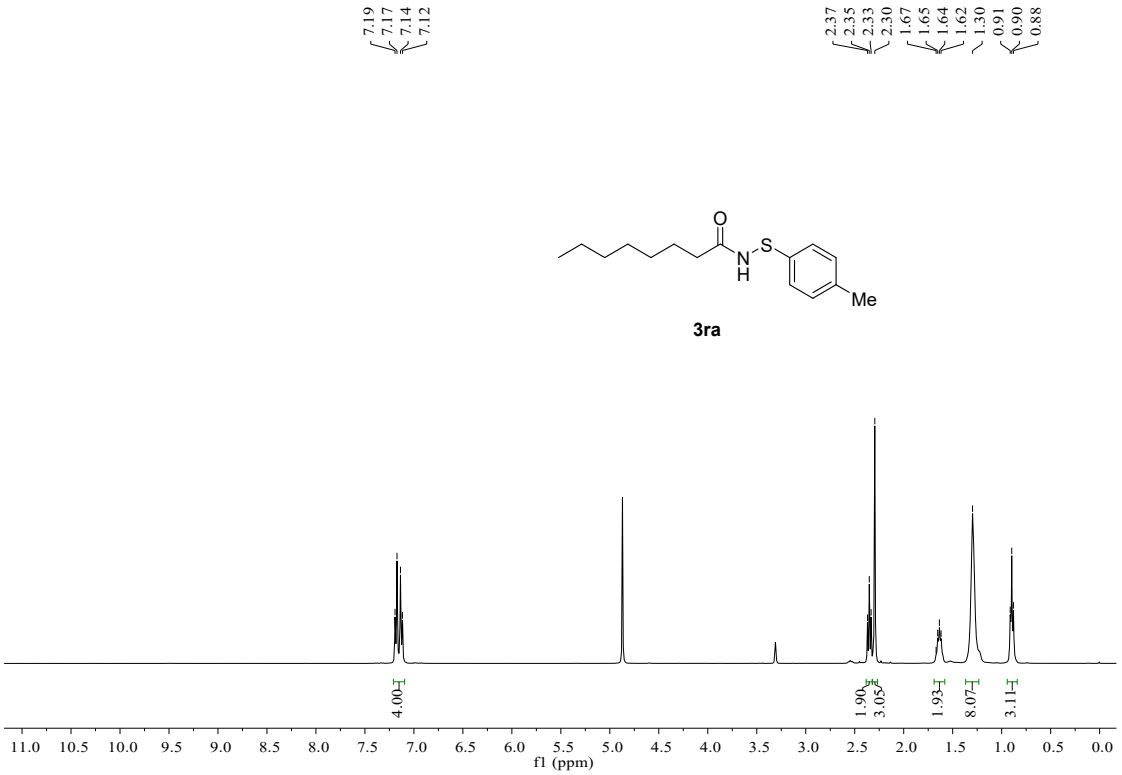
^{1}H NMR, 400 MHz, DMSO-d_6



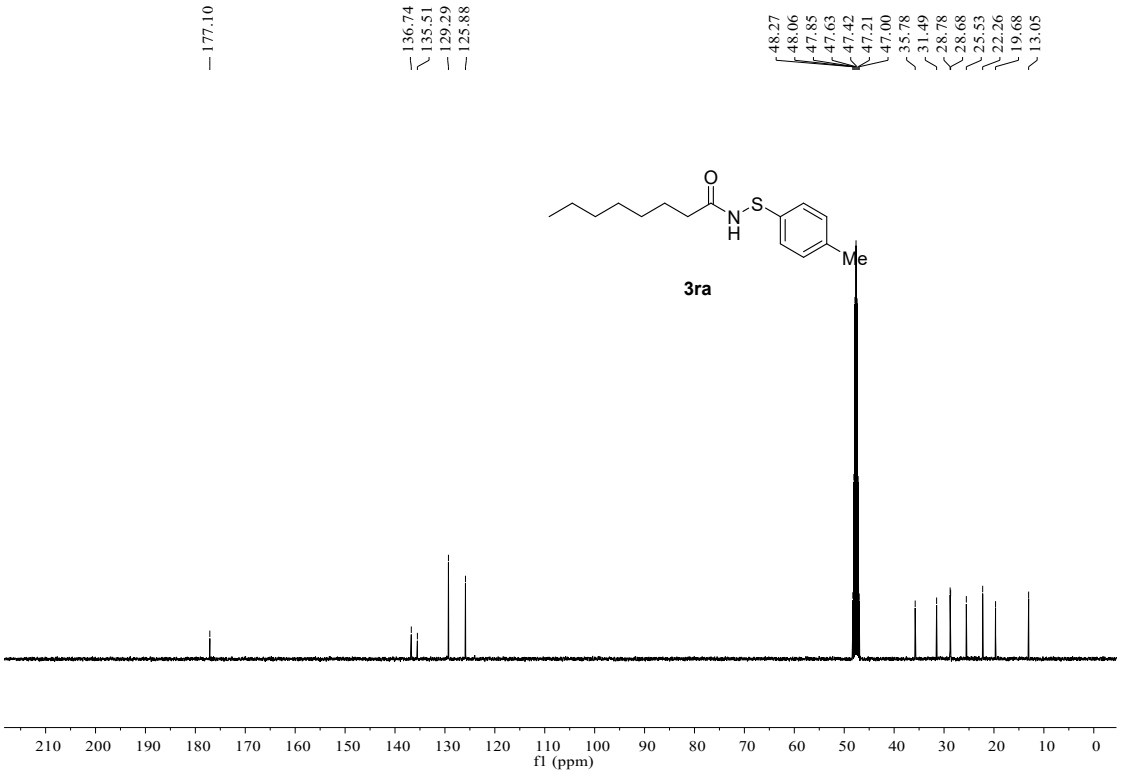
^{13}C NMR, 100 MHz, DMSO-d_6



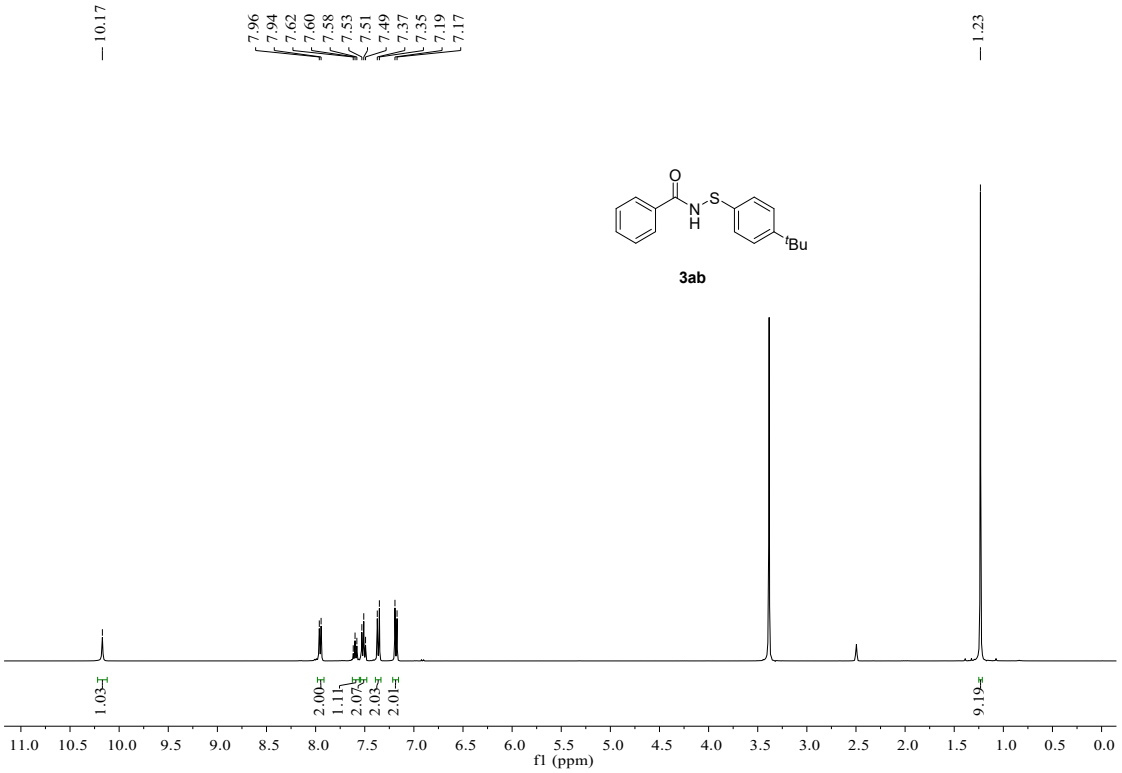
^{1}H NMR, 400 MHz, CD_3OD



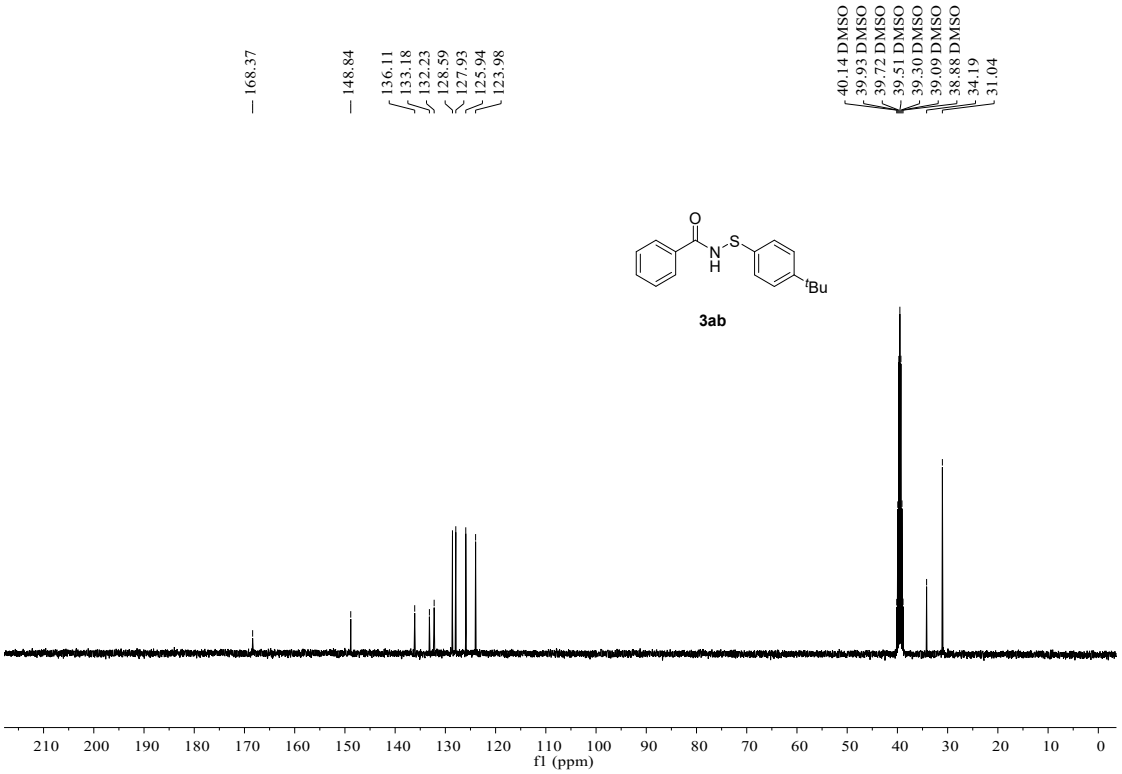
^{13}C NMR, 100 MHz, CD_3OD



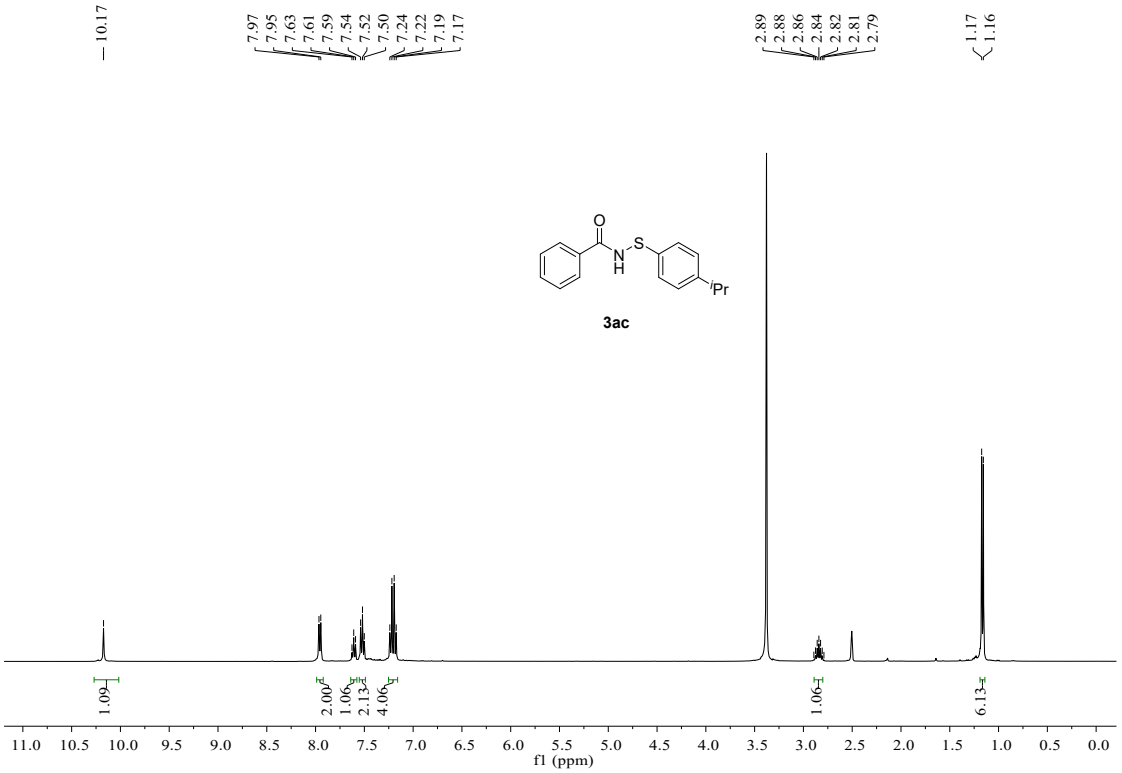
^{1}H NMR, 400 MHz, DMSO-d_6



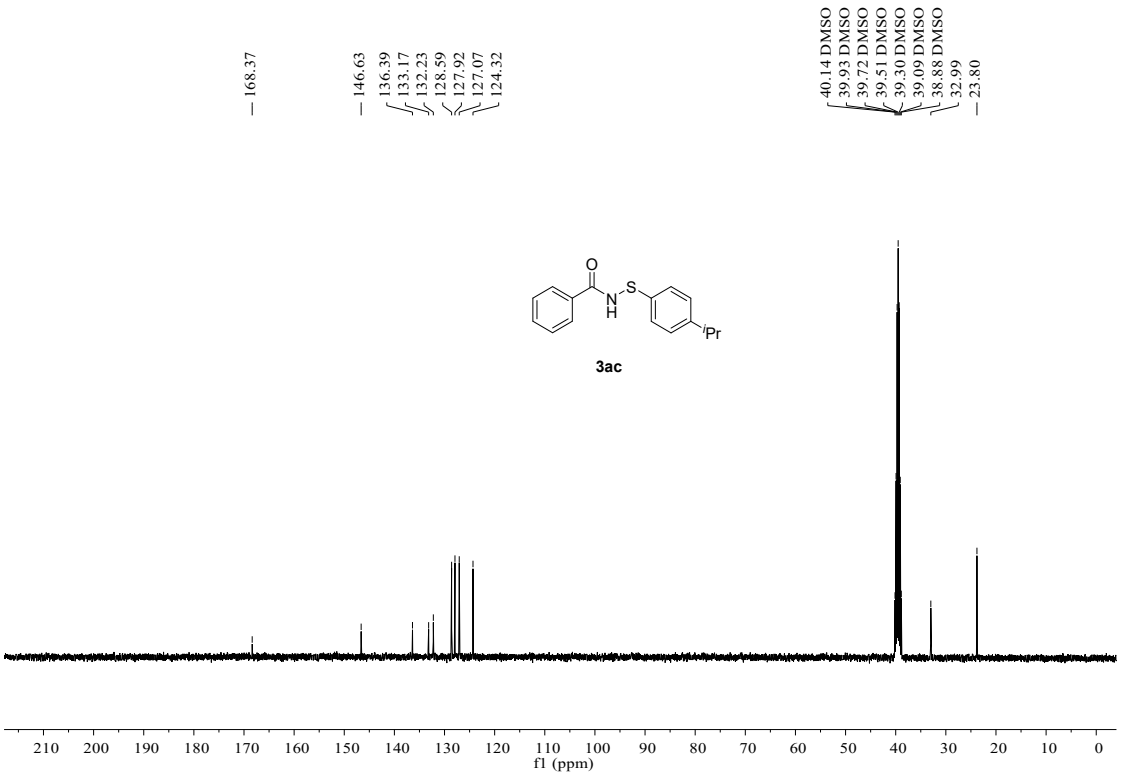
^{13}C NMR, 100 MHz, DMSO-d_6



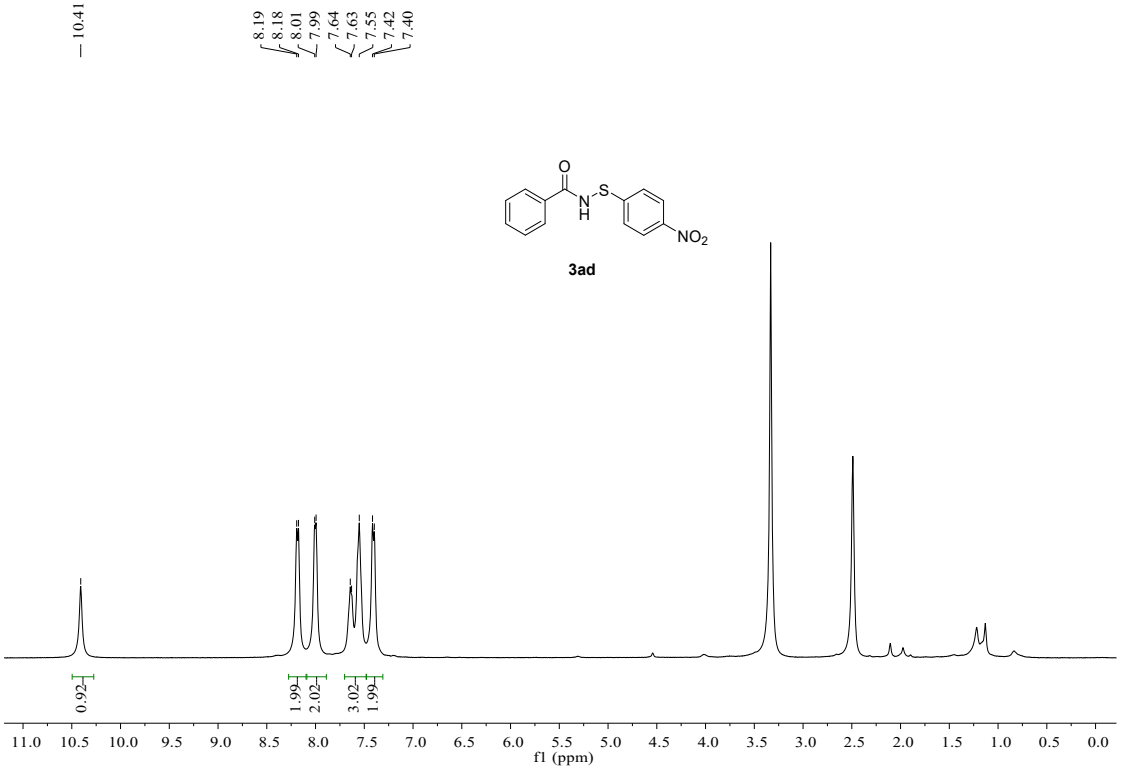
^{1}H NMR, 400 MHz, DMSO-d_6



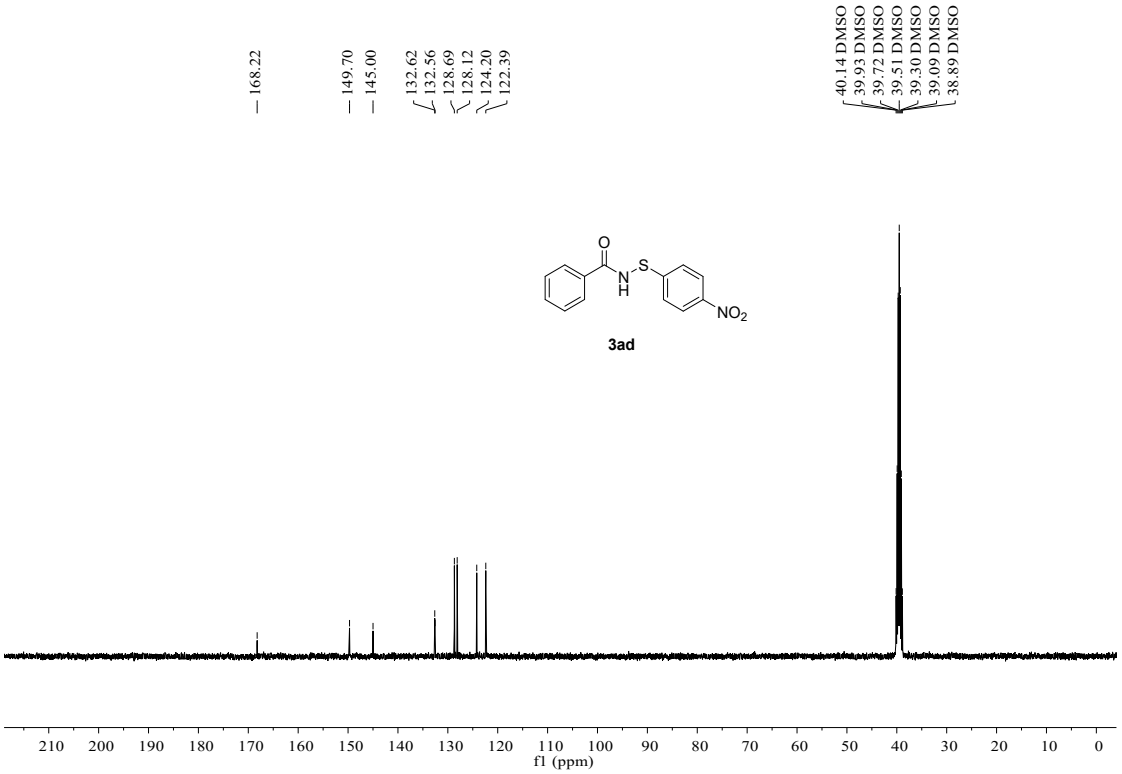
^{13}C NMR, 100 MHz, DMSO-d_6



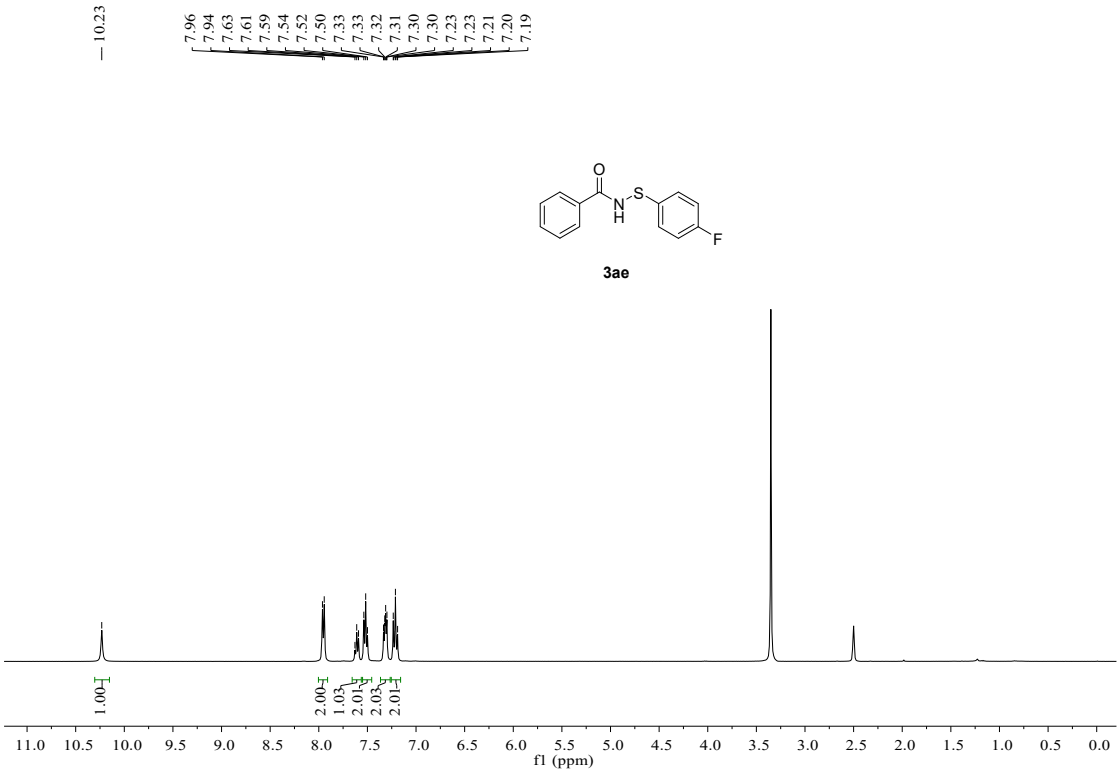
^{1}H NMR, 400 MHz, DMSO-d_6



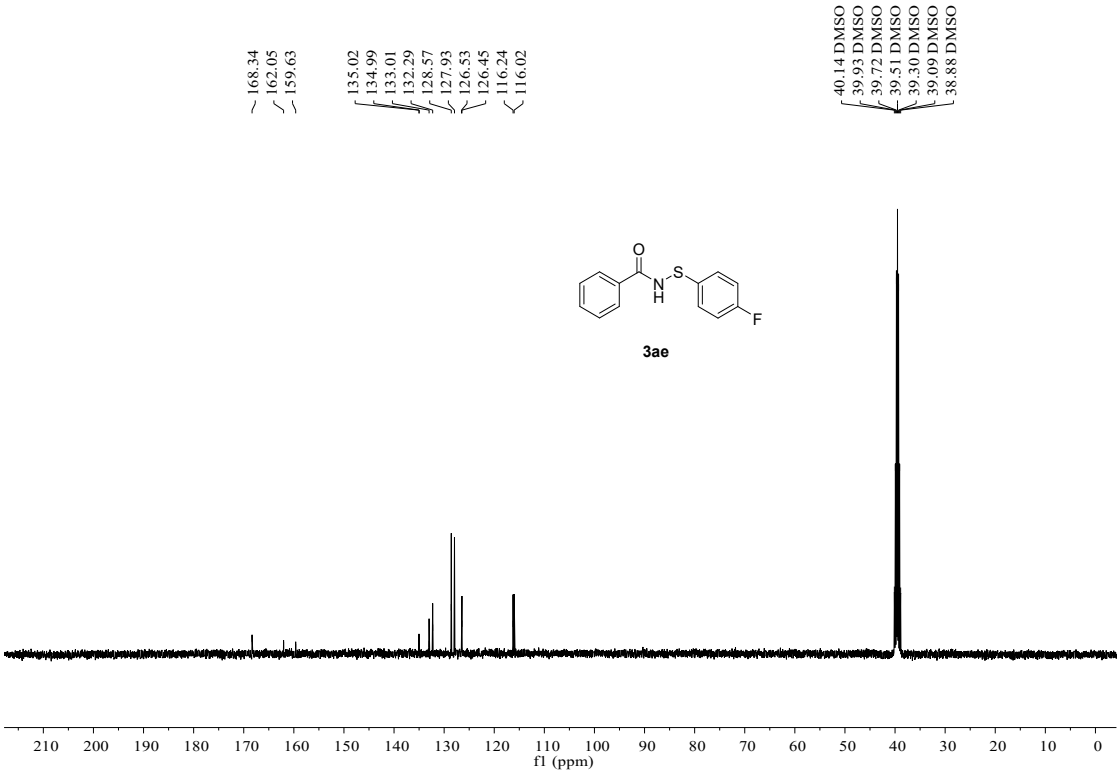
^{13}C NMR, 100 MHz, DMSO-d_6



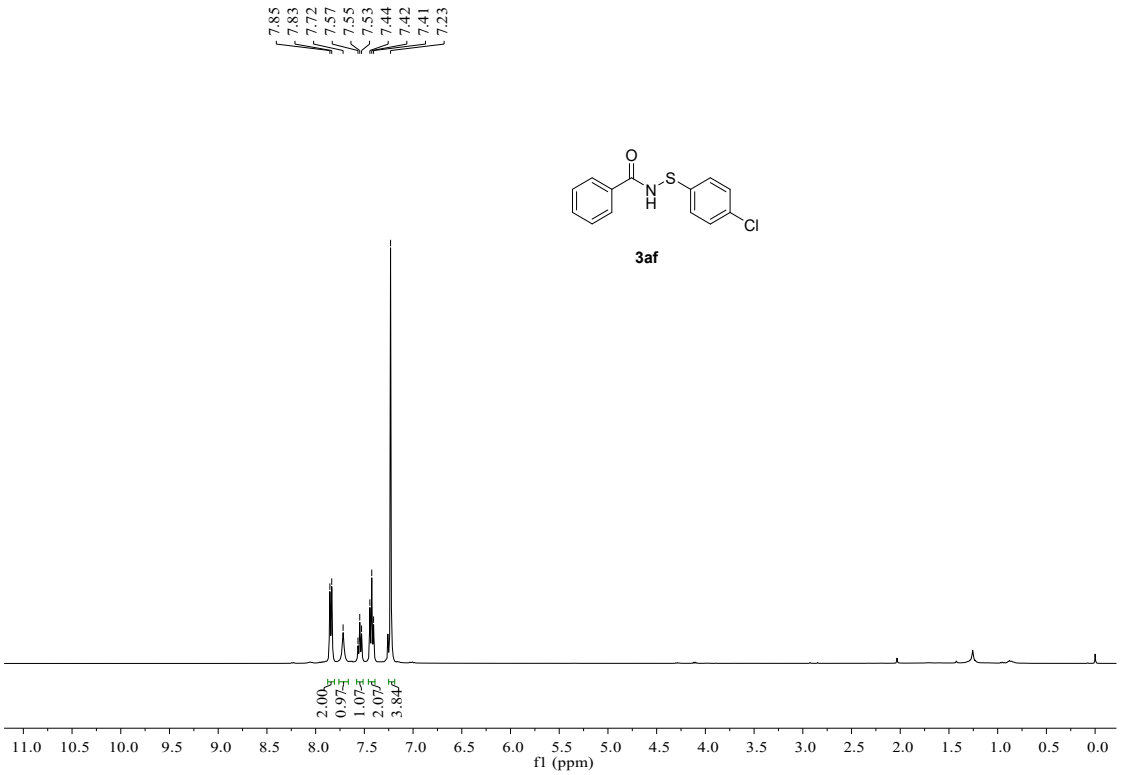
^{1}H NMR, 400 MHz, DMSO-d_6



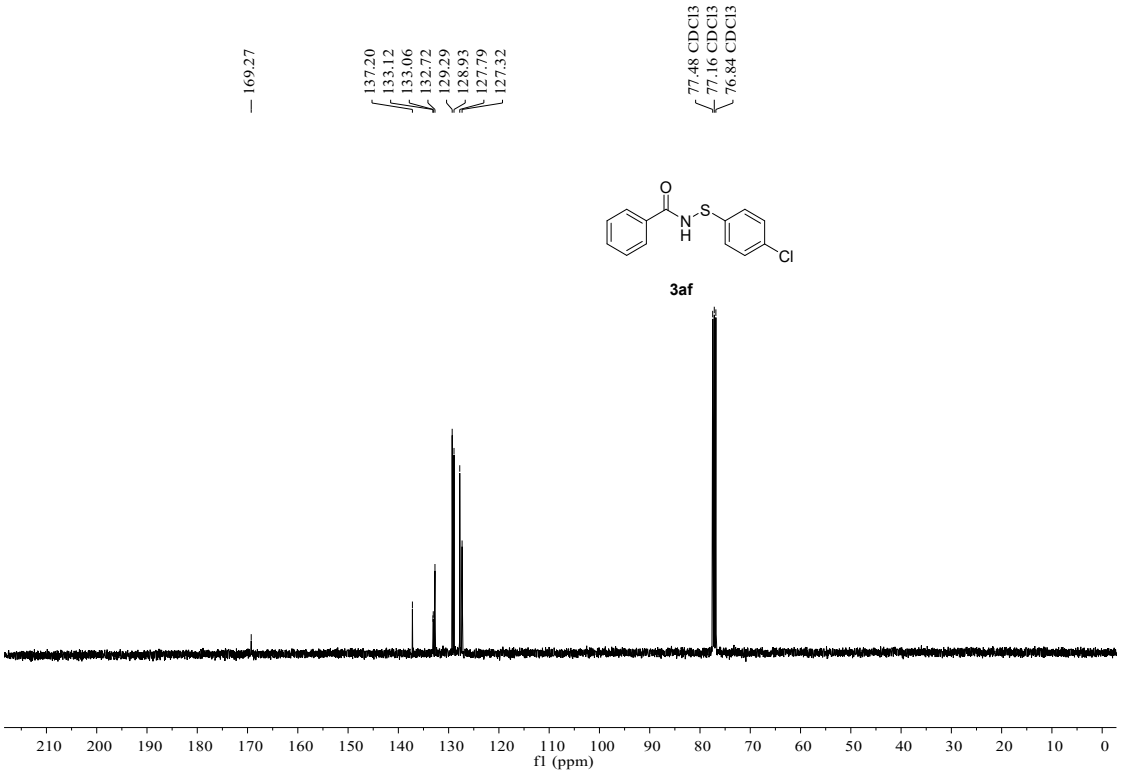
^{13}C NMR, 100 MHz, DMSO-d_6



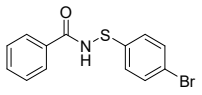
^{1}H NMR, 400 MHz, $CDCl_3$



^{13}C NMR, 100 MHz, $CDCl_3$



^{1}H NMR, 400 MHz, DMSO-d_6



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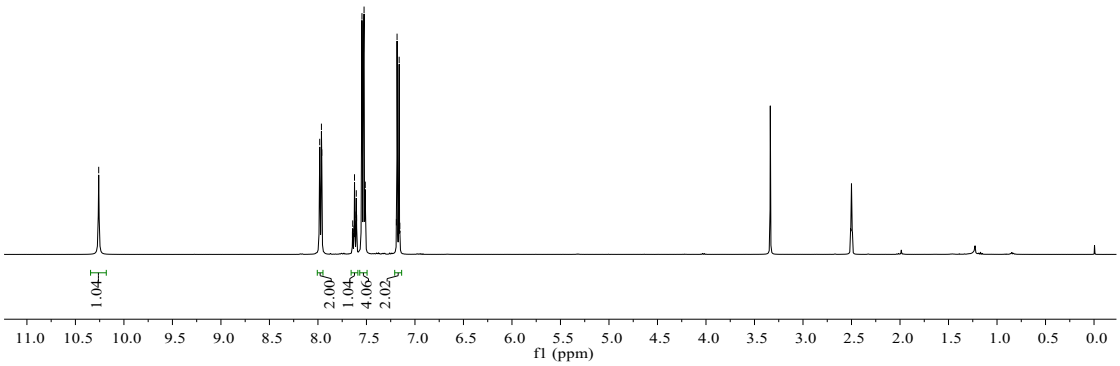
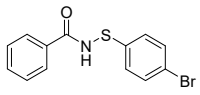


^{13}C NMR, 100 MHz, DMSO-d_6



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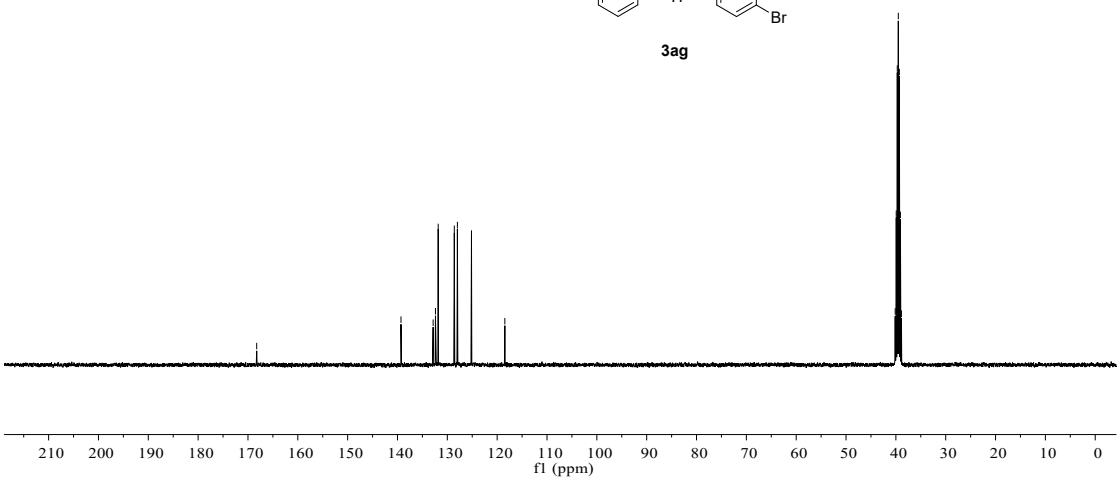
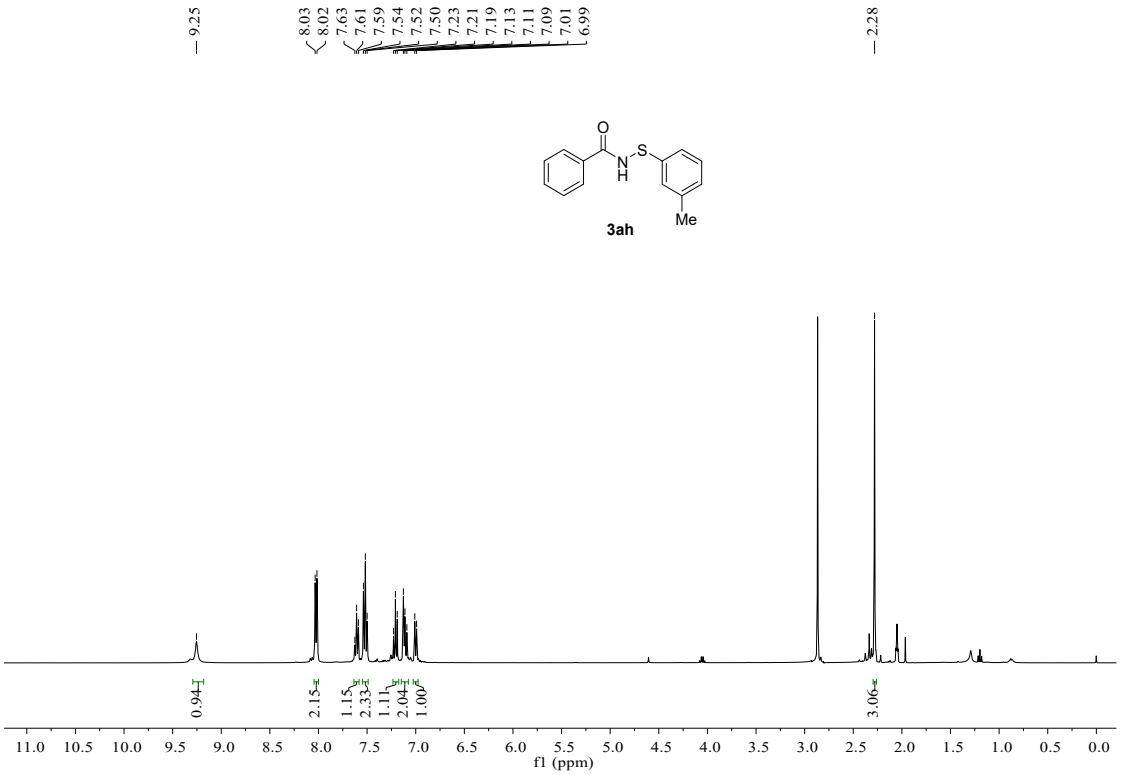
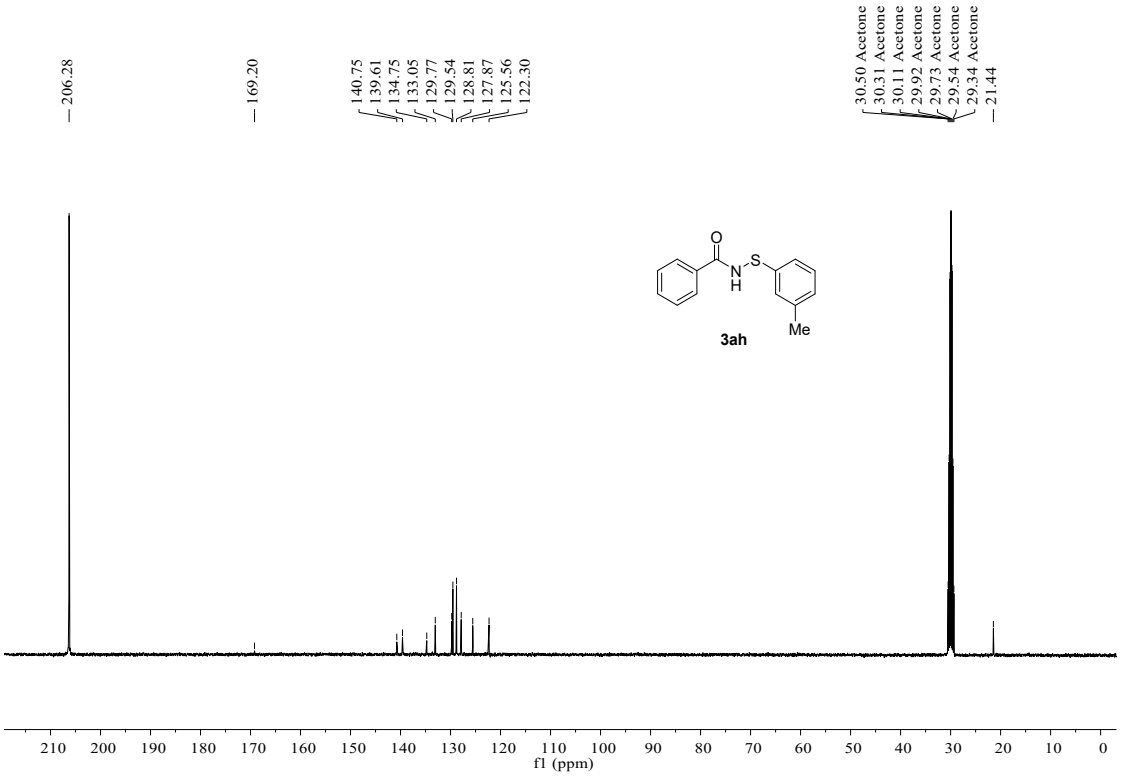


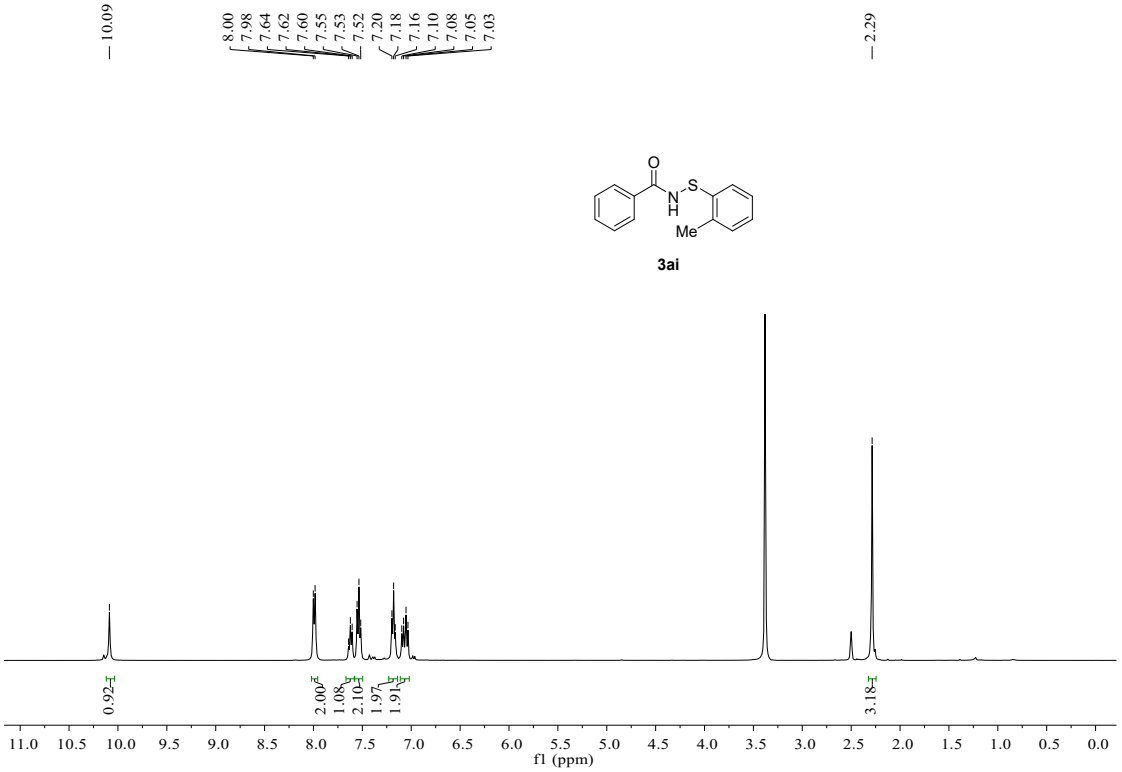
^{1}H NMR, 400 MHz, Acetone-d_6



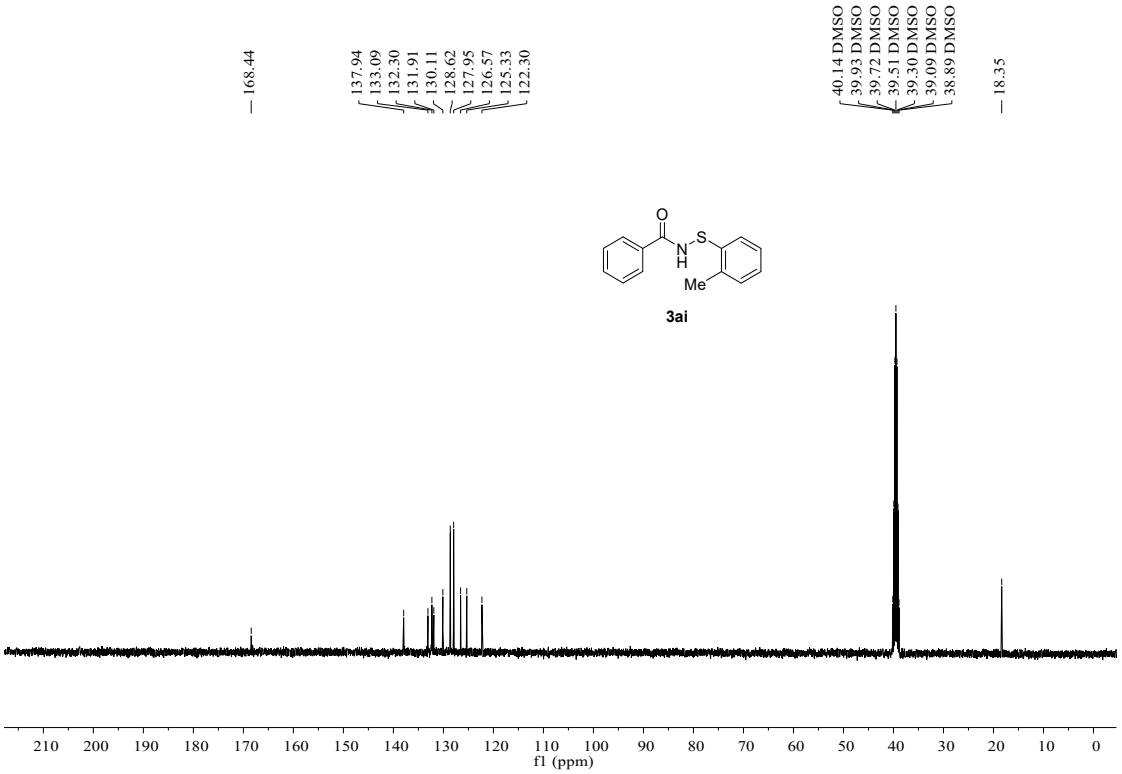
^{13}C NMR, 100 MHz, Acetone-d_6



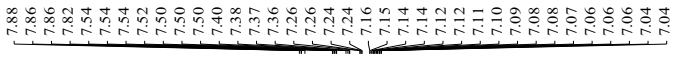
^{1}H NMR, 400 MHz, DMSO-d_6

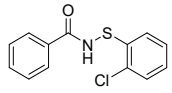


^{13}C NMR, 100 MHz, DMSO-d_6



^{1}H NMR, 400 MHz, $CDCl_3$





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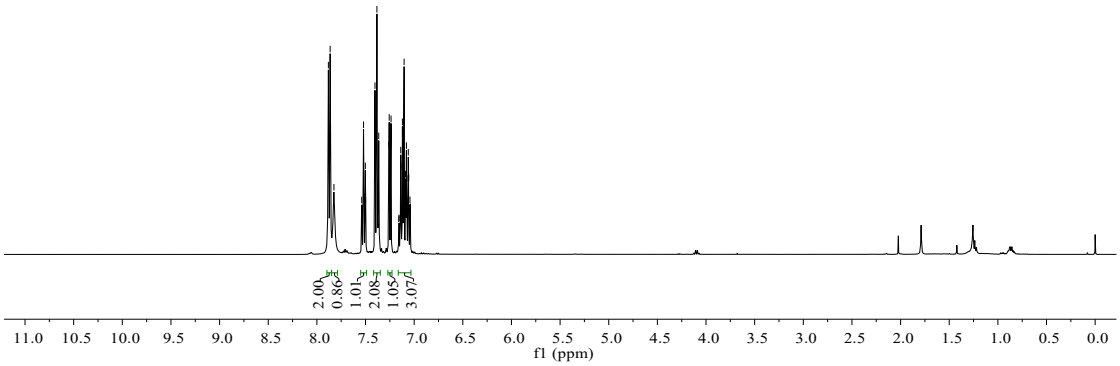
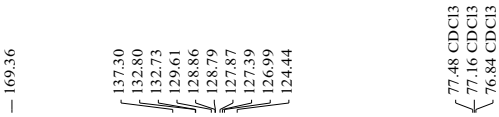
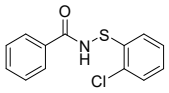


^{13}C NMR, 100 MHz, $CDCl_3$





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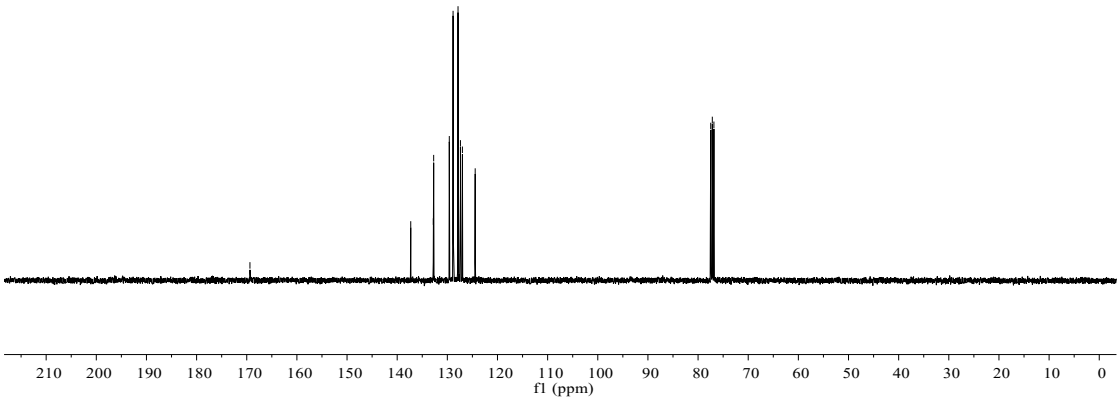
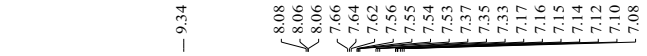
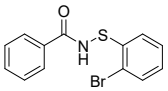


^{1}H NMR, 400 MHz, Acetone-d_6





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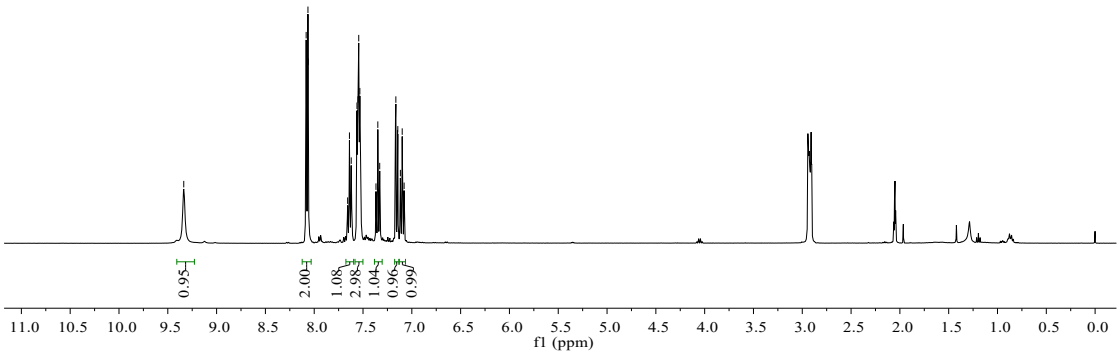
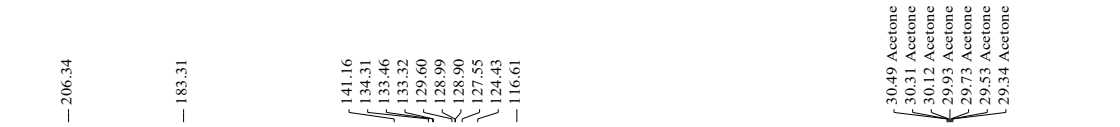
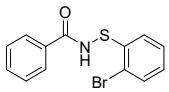


^{13}C NMR, 100 MHz, Acetone-d_6





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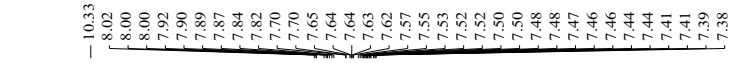
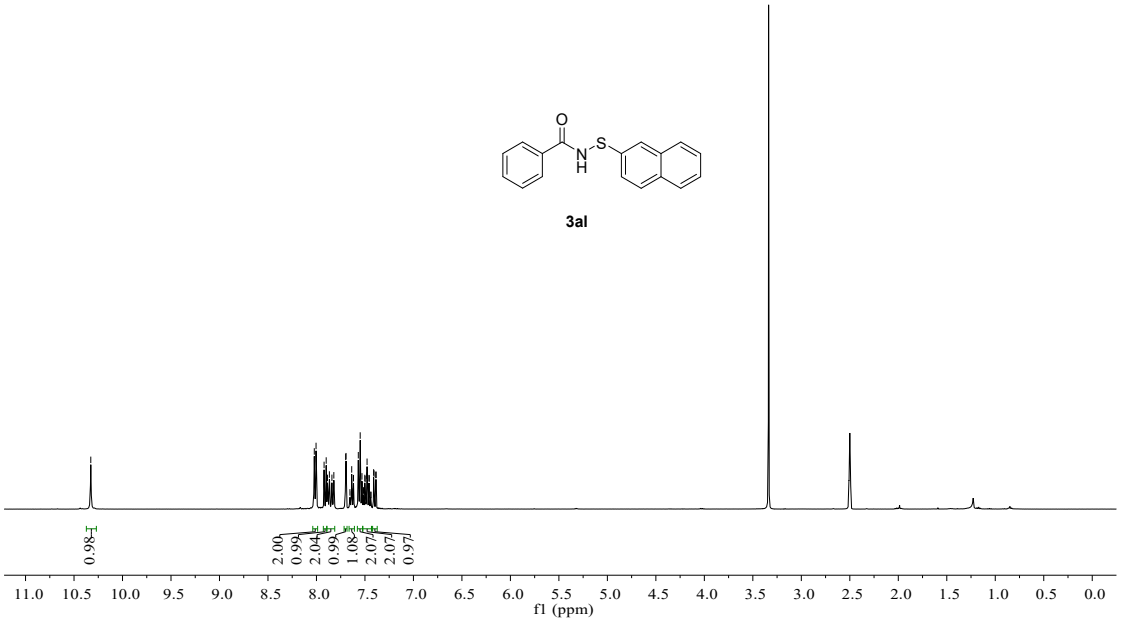
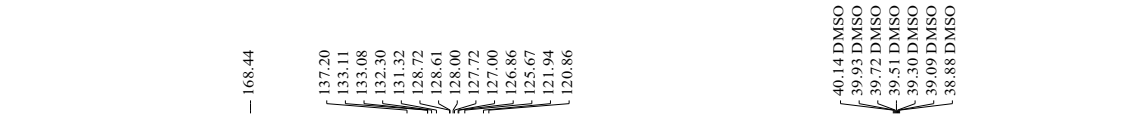
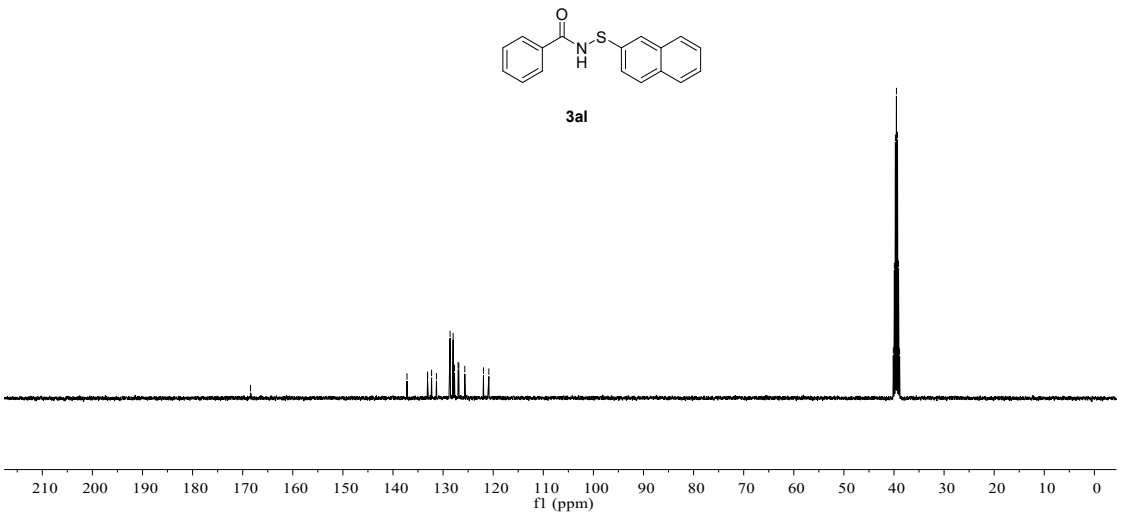
^{1}H NMR, 400 MHz, DMSO-d_6



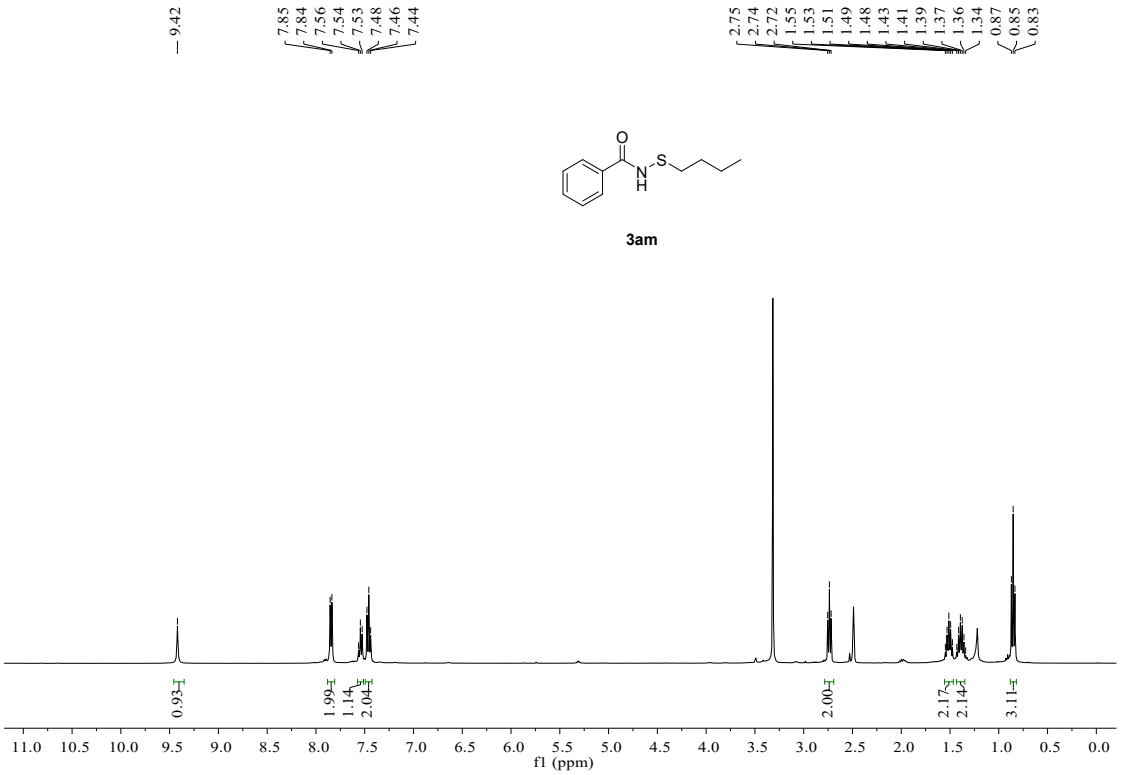


^{13}C NMR, 100 MHz, DMSO-d_6

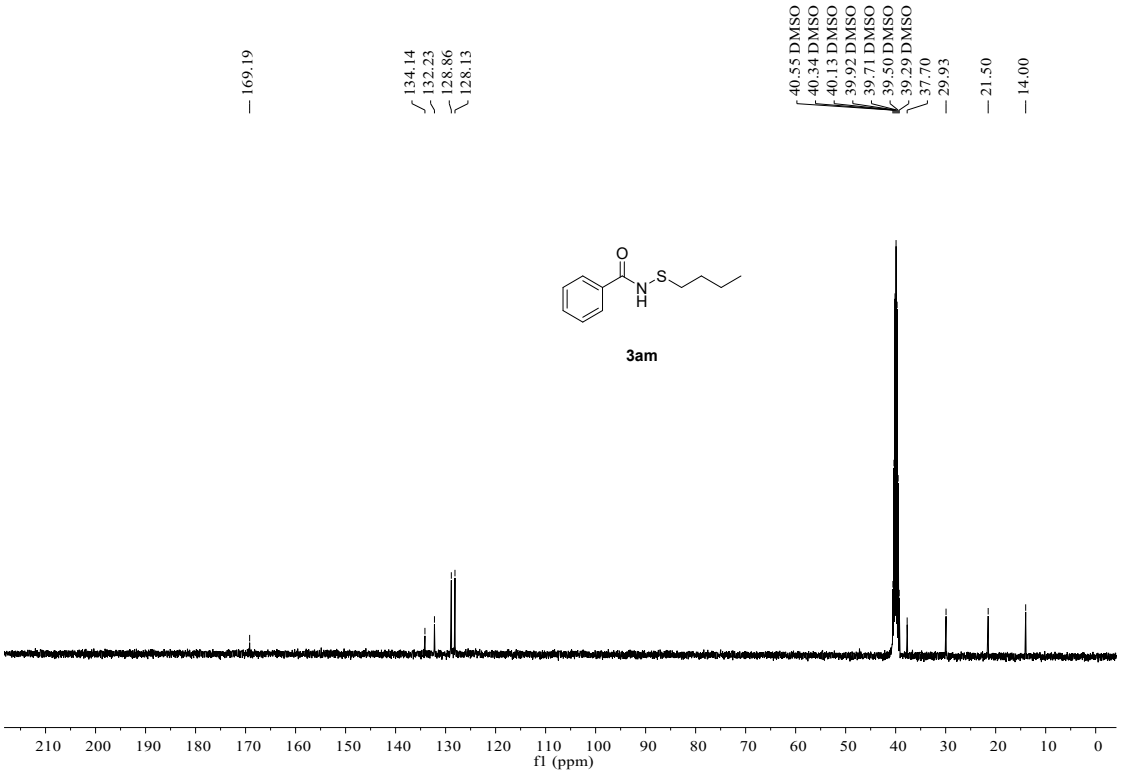




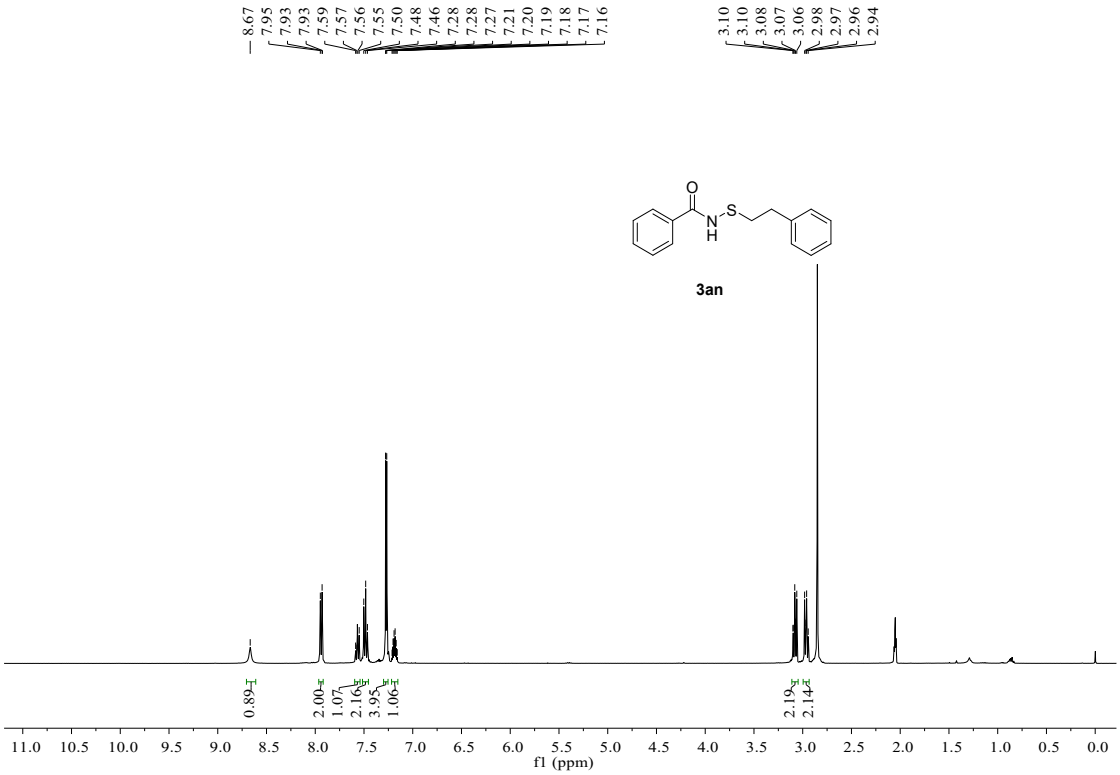
^{1}H NMR, 400 MHz, DMSO-d_6



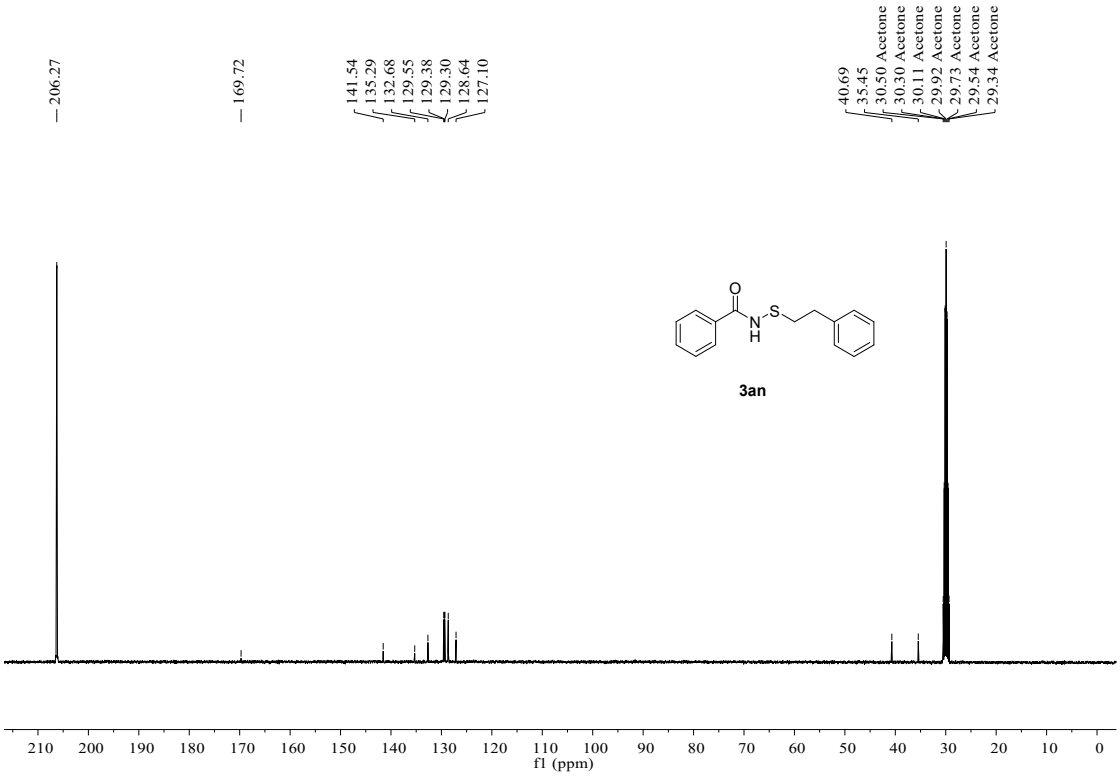
^{13}C NMR, 100 MHz, DMSO-d_6



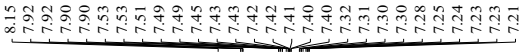
^{1}H NMR, 400 MHz, Acetone-d_6

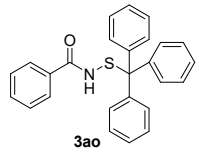


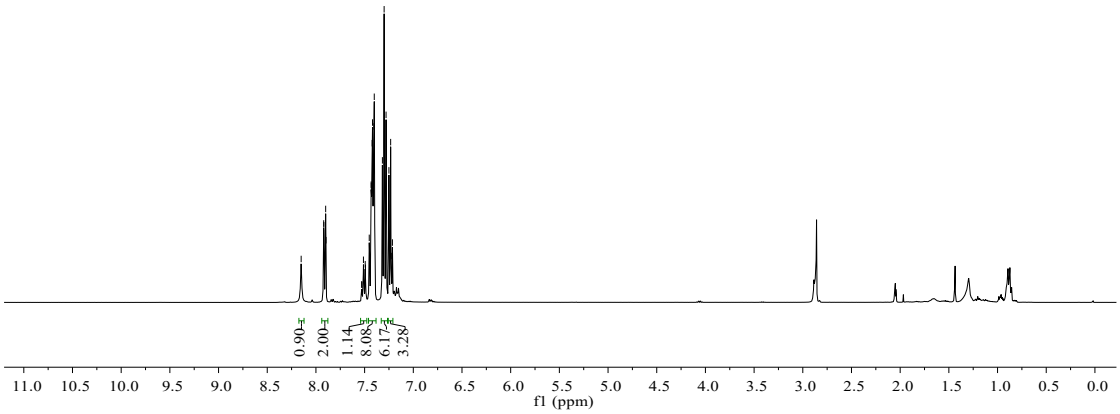
^{13}C NMR, 100 MHz, Acetone-d_6



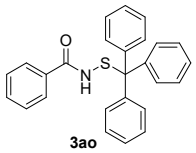
^{1}H NMR, 400 MHz, Acetone-d_6

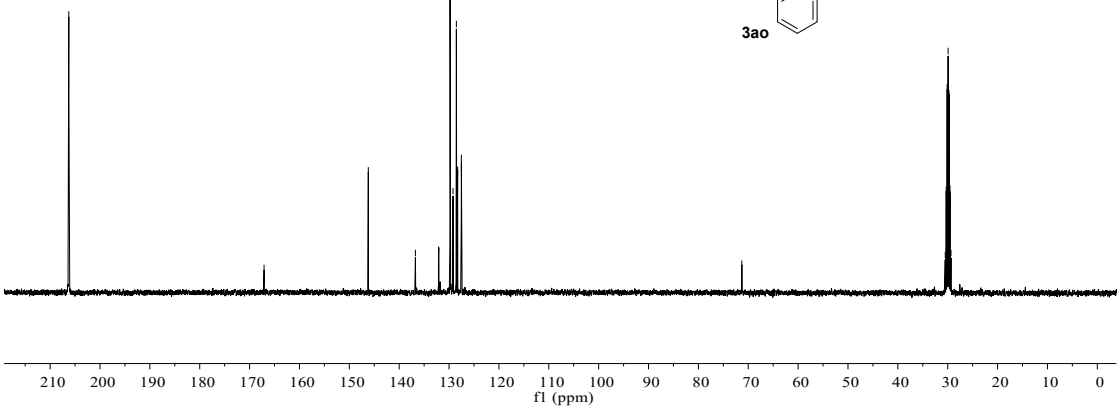




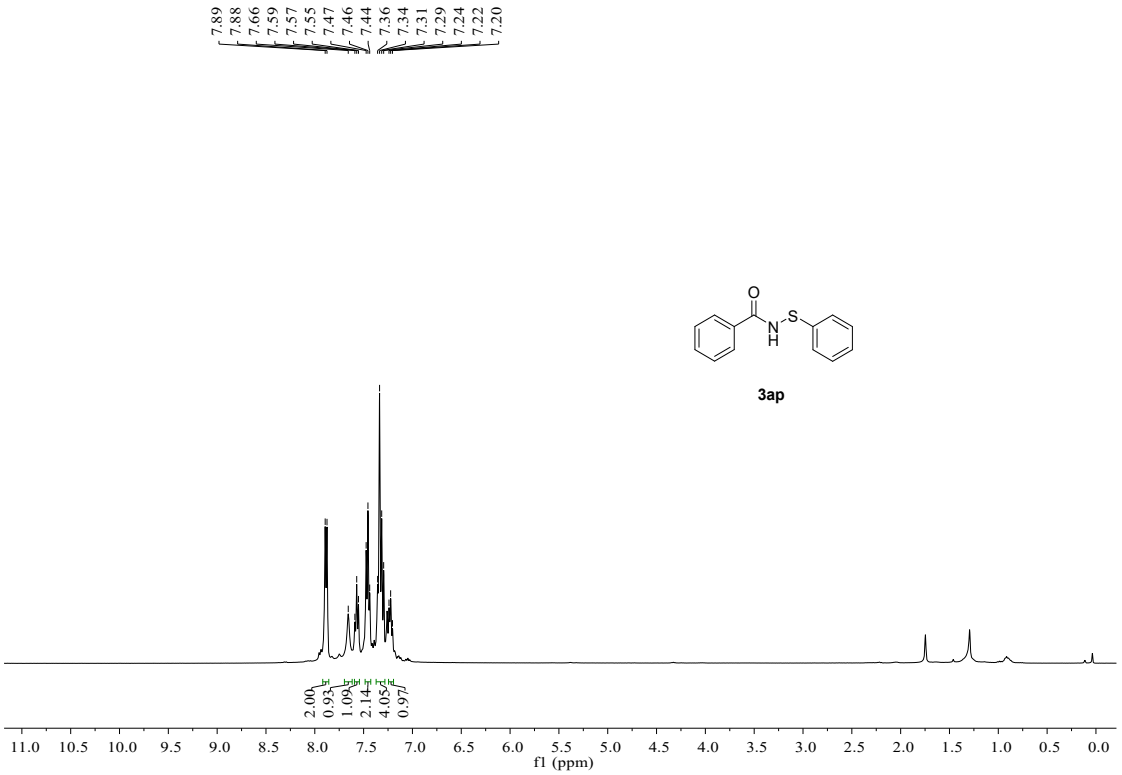


^{13}C NMR, 100 MHz, Acetone-d_6





^{1}H NMR, 400 MHz, $CDCl_3$



^{13}C NMR, 100 MHz, $CDCl_3$