Electronic Supplementary Information

Metal-free, *tert*-butyl nitrite promoted C(sp^2)-S coupling reaction: Synthesis of aryl dithiocarbamates and analysis of antimicrobial activity by '*in silico'* and '*in vitro'* methods for drug modification

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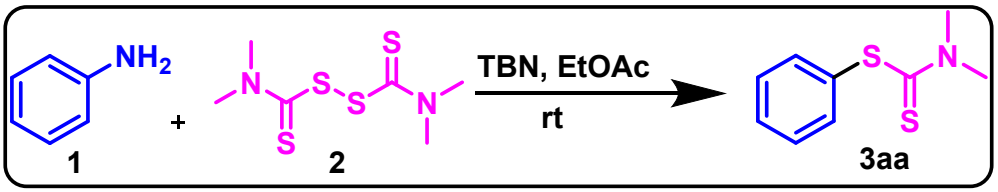
Contents

1. General information:

^{1}H NMR spectra were determined on a Bruker 400 (400 MHz) spectrometer as solutions in $CDCl_3$. Chemical shifts are expressed in parts per million (δ), and the signals were reported as s (singlet), d (doublet), t (triplet), m (multiplet), and coupling constants *J* were given in Hz. $^{13}C\{^{1}H\}$ NMR and ^{19}F NMR spectra were recorded at 100 MHz and 376 MHz in $CDCl_3$ solution, respectively. Chemical shifts are expressed in parts per million (δ) and are referenced to $CDCl_3$ (δ = 77.16) as an internal standard. TLC was done on a silica gel-coated glass slide (Merck, Silica gel G for TLC). Silica gel (60-120 mesh, SRL, India) was used for column chromatography. Unless otherwise mentioned, petroleum ether refers to the fraction boiling in the 60-80 °C range. Commercially available substrates were freshly distilled before the reaction. Solvents, reagents, and chemicals were purchased from Aldrich, Merck, and Spectrochem Chemicals.

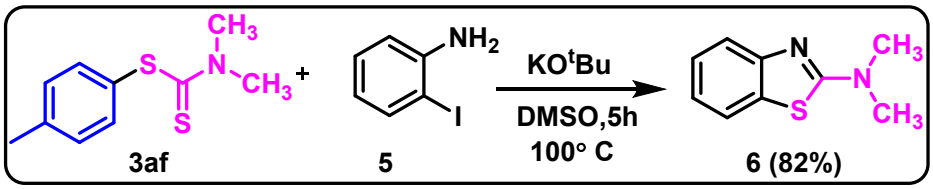
2. General experimental procedure for the synthesis of 3aa: A mixture of aniline (**1**) (0.5 mmol), ^{t}BuONO (0.6 mmol), thiuram disulfide (**2**) (0.5 mmol) and 2 ml EtOAc was stirred under room temperature and open to the air for 6 hours. After the completion of the reaction, confirmed by TLC, the mixture was diluted with saturated saline water (3 × 15 mL), and extracted with ethyl acetate. The combined organic layer was collected and dried over anhydrous Na_2SO_4. The residue was purified by column chromatography on silica gel to afford the desired products **3aa** (eluent: ethyl acetate/petroleum ether).



3. Experimental procedure for the synthesis of N,N-dimethylbenzo[d]thiazol-2-amine (6):

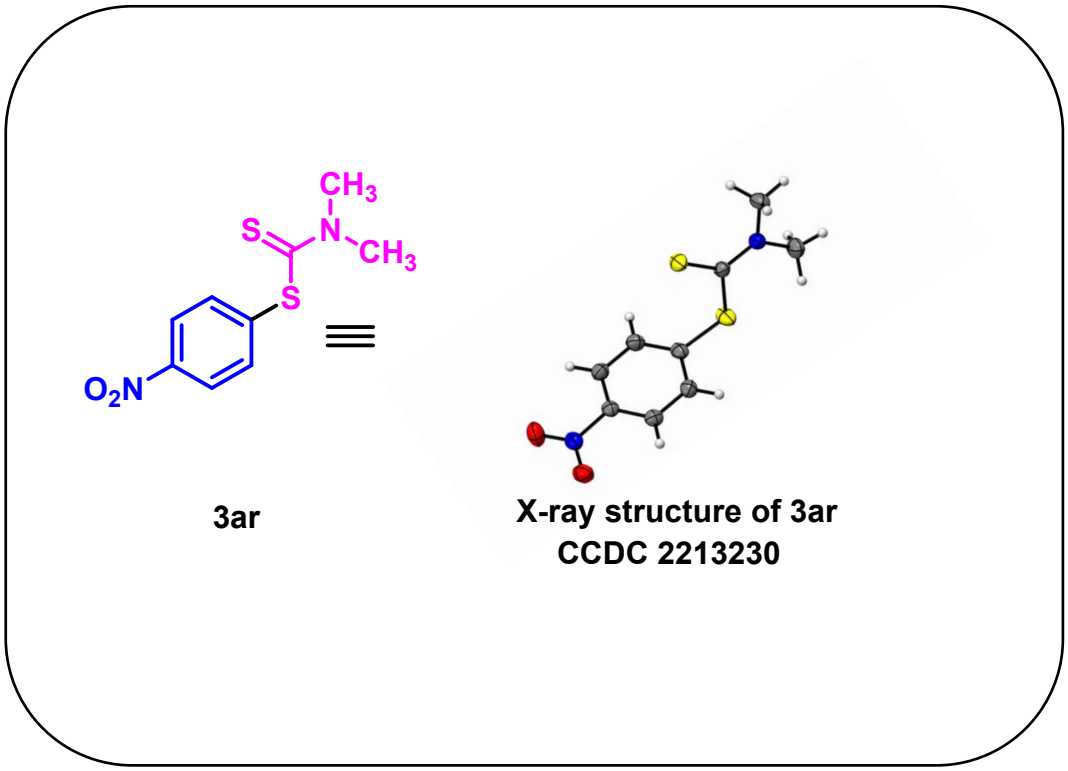
Dithiocarbamate (0.75 mmol) was added in a mixture of 2-iodoaniline (**5**) (0.5 mmol), DMSO (3 mL), and KOt-Bu (1.5 mmol). The reaction mixture was heated at 100°C and checked by TLC until the starting material was finished. After that, the reaction mixture was cooled at room temperature, and the mixture was diluted with saturated saline water (3 × 15

mL), and extracted with ethyl acetate. The combined organic layer was collected and dried over anhydrous Na_2SO_4. The residue was purified by column chromatography on silica gel to afford the desired products **6** (eluent: ethyl acetate/petroleum ether).



4. Structure determination (X-ray crystallographic data for 3ar):

The yellow block crystal of 3ar was obtained by crystallization from a solution in dichloromethane/petroleum ether after purification by column chromatography. The chemical formula of compound 3ba: $C_9H_{10}N_2O_2S_2$.



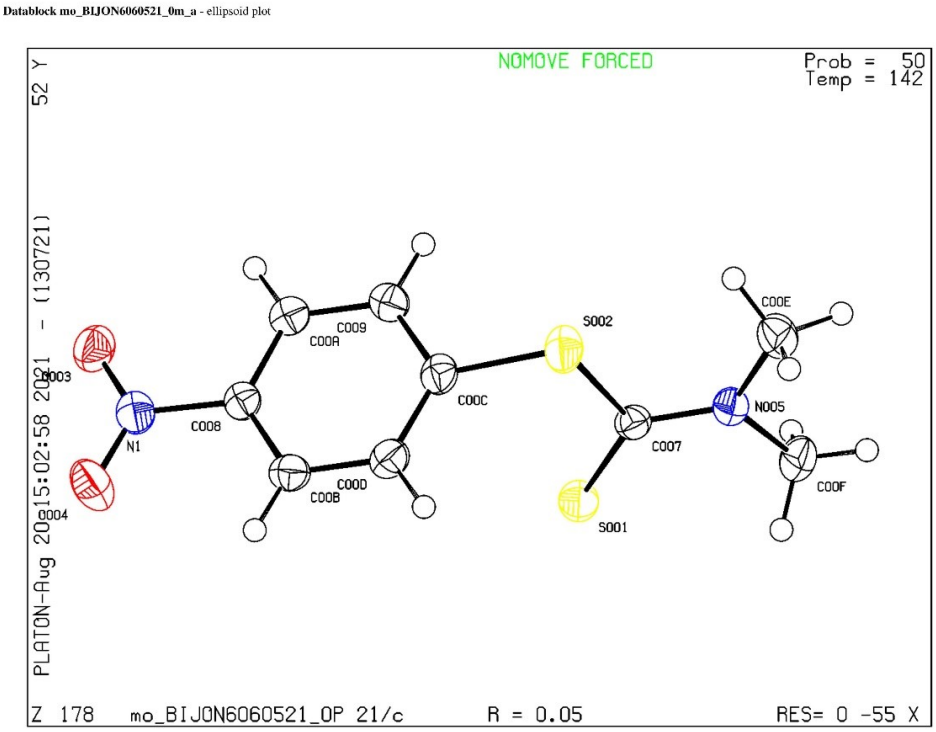


Table 1: Crystal data

Wavelength	**0.71073Å**	
Formula	$C_9H_{10}N_2O_2S_2$	
Crystal system	Monoclinic	
Space group	P $2_1/c$	
Unit cell dimensions	a = 7.5418(3) Å	α = 90
	b = 11.3152(4) Å	β = 96.939(1)
	c = 12.9845(5) Å	γ = 90
Volume	1099.94(7) $Å^3$	
Z	4	
R-factor (%)	4.75	

The crystallographic data have been deposited with the Cambridge Crystallographic Data Centre as a supplementary publication with a CCDC reference number CCDC 2213230.

5. Biology:

1. Material method

1.1. Antibacterial assay

Resazurin microtiter plate-based assay is used to test the antibacterial activity of sulfacetamide (X) and its synthetic derivatives X1, X2, and X3 using a modified technique of Sarker[1] et al. 2007. Resazurin solution (0.01%) was prepared by dissolving the resazurin powder in sterile distilled water in a sterile vial. The solution was mixed for 1 h in a vortex mixer to ensure homogeneity. In this study, six pathogenic bacteria, including *Bacillus cereus* ATCC 13061, *Bacillus subtilis* MTCC 121, *Listeria monocytogenes* MTCC 657, *Staphylococcus aureus* MTCC 96, *Salmonella typhimurium* MTCC 98, and *Escherichia coli* MTCC 1667 were used which were procured from the Microbial Type Culture Collection (MTCC) IMTech, Chandigarh. Since the test materials are serially diluted while the bacterial concentration is lowered serially, this technique cannot provide a "true" indication of the minimum inhibitory concentration (MIC) intended to be assessed in this experiment. We modified the resazurin-based experiment, namely the dilution procedures, and used a standard concentration of the bacterial solution to obtain a "true" MIC result. At first, the bacterial test strains were taken out of the agar slants, inoculated in freshly produced nutrient broth, incubated at 37° C, and kept as instructed by MTCC. Pathogenic bacterial cultures were cultivated overnight, diluted with sterile nutrient broth, and quantified in a spectrophotometer to a specific OD of 0.00075. Each test metabolite solution was introduced to the relevant wells at a volume of 2.5 µl. The total test volume was increased to 100.0 µL by adding 97.5 µL of the diluted bacterial broth to each well. Negative control was used to ascertain whether the bacteria were growing; only bacterial suspensions were added to those wells. The resazurin solution was added to each well in a volume of 4.0 µL and well mixed. Plates were incubated at 37° C for 6–8 hours, and any color changes were visually observed. Samples with various concentrations were used to determine the Minimum Inhibitory Concentration (MIC) that produced positive findings in the antibacterial test, namely, 100.0 µg/mL, 50.0 µg/mL, 25.0 µg/mL, 12.5 µg/mL, 6.25 µg/mL, 3.12 µg/mL, 1.56µg/ml, 0.78µg/ml were prepared by dissolved in DMSO. These various amounts were added to a predetermined volume of nutrient broth-based bacterial cultures. The creation of a precise MIC value, which can be compared to antibiotics currently on the market, gives the scientist the capacity to determine if the extracts and compounds are worthwhile to continue investigating regarding their antibacterial potential.

1.2. In-silico pharmacokinetic property analysis:

The in-silico prediction studies were performed using SWISS ADME (http://www.swissadme.ch/) and pkCSM (https://biosig.lab.uq.edu.au/pkcsm/) online prediction platforms (Pires, Blundell & Ascher 2015), to assess the theoretical pharmacokinetic parameters of the ligands to predict the drug-likeness of ligands. The software calculated pharmaceutically relevant properties such as H-bond donor, H-bond acceptor, octanol-water partition coefficient (LogP), surface area, and rotatable bonds count. In addition, the effect of ligands on ADMET parameters like water solubility, Caco2 permeability, human intestinal absorption, skin permeability, P-glycoprotein I, and II inhibition, the volume of distribution, fraction of unbound drug, Blood Brain Barrier (BBB), and Central Nervous System (CNS) permeability, cytochrome P450 inhibition, total clearance, OCT2 (organic cation transporter 2) substrate, Skin Sensitization, Hepatotoxicity, Carcinogenicity, etc. were also evaluated.

1.3. Molecular Docking:

Docking studies of sulfacetamide (X) and its three synthetic derivatives X1, X2, X3 against dihydropteroate synthase (DHPS) and dihydrofolate reductase (DHFR) were done using molecular docking program AutoDock tools[2] in order to find the preferred binding conformations between them. The 3D structure of DHPS (PDB ID: 5U13) and DHFR (PDB ID: 4DFR) retrieve from the protein data bank (https://www.rcsb.org/). The protein structures were then minimized, and adding missing amino acids side chains were using Swiss PDB viewer[3]. Further protein preparation was done using AutoDock tools. The ligands were drawn using ChemDraw and optimized by PM6 of Semi-empirical Method using GaussView 5.0. For Docking, A grid box was generated that was large enough to cover the active site and accommodate ligands to move freely. The number of grid points in the x, y, and z-axes for DHPS and DHFR was 70×70×70 Å and 64×64×64 Å respectively. The distance between two connecting grid points for the two proteins was 0.375 Å. The center of the ligand in the X-ray crystal structure was used as the center of the grid box. AutoDock4 and a Lamarckian Genetic Algorithm (LGA), which has enhanced performance relative to simulated annealing and genetic algorithm alone, were used for receptor-fixed ligand-flexible docking calculations. Ten search attempts were performed for ligand. The maximum number of energy evaluations before the termination of the LGA run was 2500000, and the maximum number of generations of the LGA run before termination was 27000. Other parameters of

docking were set to the default values. During the docking process, a maximum of 10 different conformations were considered for the ligand. The lowest binding free energy conformer was used for further analysis. The docked complex of protein and ligand was visualized by PyMOL and Discovery Studio Visualizer of Accelrys Discovery Studio.

2. Result and Discussion:

2.1 Antibacterial assay:

Table 2: MIC values of the mother drug X and their derivatives X1, X2, X3 against six different pathogenic bacteria.

Test bacteria	Gram-positive/ negative	MIC value of X (µg/ml)	MIC value of X1 (µg/ml)	MIC value of X2 (µg/ml)	MIC value of X3 (µg/ml)
Escherichia coli	Gram (-) ve	50µg/ml	3µg/ml	6µg/ml	3µg/ml
Bacillus subtilis	Gram (+) ve	100µg/ml	6µg/ml	3µg/ml	3µg/ml
Listeria monocytogenes	Gram (+) ve	100µg/ml	6µg/ml	6µg/ml	3µg/ml
Salmonella typhimurium	Gram (-) ve	25µg/ml	3µg/ml	3µg/ml	3µg/ml
Staphylococcus aureus	Gram (+) ve	50µg/ml	6µg/ml	3µg/ml	3µg/ml
Bacillus cereus	Gram (+) ve	100µg/ml	3µg/ml	3µg/ml	3µg/ml

2.2 Pharmacokinetic properties of the three derivatives X1, X2, X3:

Table 3: Pharmacokinetic properties of the three derivatives X1, X2, X3

Sl. No.	Properties	X1	X2	X3
1.	LogP	1.45	2.2302	3.7906
2.	Rotatable Bond	3	5	9
3.	Acceptor	5	5	5
4.	Donars	1	1	1
5.	Surface area	121.970	134.700	160.160
6.	Water Solubility (Log mol/L)	-3.581	-3.549	-5.082
7.	Caco2 Permeability (log Papp in 10-6cm/s)	0.896	1.043	0.968
8.	Intestinal absorption (human) (% Absorbed)	92.27	88.815	90.868
9.	Skin Permeability (log Kp)	-3.023	-2.98	-2.852
10.	P-glycoprotein substrate (Yes/No)	NO	NO	YES
11.	P-glycoprotein I inhibitor (Yes/No)	NO	NO	NO
12.	P-glycoprotein II inhibitor (Yes/No)	NO	NO	NO
13.	VDss (human) (log L/kg)	-0.267	-0.219	0.072
14.	Fraction unbound (human) (Fu)	0.524	0.393	0.28
15.	BBB permeability (log BB)	-0.256	-0.183	-0.18
16.	CNS permeability (log PS)	-2.976	-2.559	-2.597
17.	CYP2D6 substrate (Yes/No)	NO	NO	YES
18.	CYP3A4 substrate (Yes/No)	NO	YES	YES
19.	CYP1A2 inhibitors (Yes/No)	NO	NO	NO
20.	CYP2C19 inhibition (Yes/No)	NO	NO	NO
21.	CYP2C9 inhibitor (Yes/No)	NO	NO	YES
22.	CYP2D6 inhibitor (Yes/No)	NO	NO	NO
23.	CYP3A4 inhibitors (Yes/No)	NO	NO	NO
24.	Total Clearance (log ml/min/kg)	0.083	0.013	0.139

25.	Renal OCT2 substrate (Yes/No)	NO	NO	NO
26.	AMES toxicity (Yes/No)	NO	NO	NO
27.	Max. tolerated dose (human) (log mg/kg/day)	0.198	0.4	0.227
28.	hERG I inhibitor (Yes/No)	NO	NO	NO
29.	hERG II inhibitor (Yes/No)	NO	NO	YES
30.	Oral Rat Acute Toxicity (LD50) (mol/kg)	2.49	2.812	2.427
31.	Oral Rat Chronic Toxicity (LOAEL) (log mg/kg_bw/day)	1.379	0.599	1.061
32.	Hepatotoxicity (Yes/No)	NO	YES	YES
33.	Skin Sensitisation (Yes/No)	NO	NO	NO
34.	T.Pyriformis toxicity (log ug/L)	0.864	1.148	1.252
35.	Minnow toxicity (log mM)	1.365	1.269	-0.704

1-5 = molecular properties 6-12= absorption (A) 13-16= distribution (D) 17-23= metabolism (M) 24-25= excretion (E)

26-35= toxicity (T)

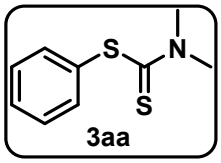
2.3 Molecular Docking:

Table 4: Interaction details of the sulfacetamide (X) and its three synthetic derivatives X1, X2, and X3 with the DHFR and DHPS enzymes.

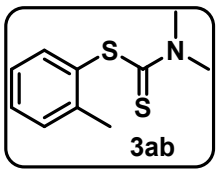
SL. NO.	Docked Complex	Binding Affinity (kcal/mol)	Interacting resides	Types of bond	Bond Distance (Å)
1.	**X_DHFR**	-5.26	ILE5 ALA7 TRP22 TRP22 TRP22 TRP22 ASP27 ILE94	H-Bond Pi-sigma Pi-Sulfur Pi-Sulfur H-Bond H-Bond H-Bond H-Bond	2.03 3.90 5.64 5.07 2.22 2.24 1.91 2.17
2.	**X1_DHFR**	-6.53	ALA7 ASP27 PHE31	H-Bond H-Bond Pi-Pi Stacked	2.03 2.49 3.93
3.	**X2_DHFR**	-6.61	ILE5 ILE50 ILE50 LEU54 ILE94 ILE94	H-Bond Alkyl Alkyl Alkyl Pi- Alkyl H-Bond	2.13 4.28 4.70 5.11 5.18 3.02
4.	**X3_DHFR**	-7.04	ALA7 ASP27 PHE31 PHE31 LYS32 ARG52	H-Bond H-Bond Pi-Alkyl Pi-Pi Stacked Alkyl Alkyl	2.64 2.12 4.52 3.86 4.09 5.01
5.	**X_DHPS**	-5.58	THR62	H-Bond	2.15

			THR62	H-Bond	2.44
			PRO64	Pi-Alkyl	5.36
			ASN197	H-Bond	3.09
			LYS221	Pi-Cation	4.82
			LYS221	Pi-Alkyl	4.97
			SER222	H-Bond	2.81
6.	**X1_DHPS**	-6.06	THR62	H-Bond	1.99
			ARG63	Pi-Alkyl	5.27
			PHE190	Pi-Sulfur	4.26
			LYS221	H-Bond	5.09
			ARG255	H-Bond	2.12
7.	**X2_DHPS**	-6.30	ILE20	Alkyl	5.23
			GLY58	Carbon	3.50
			THR62	H-Bond	2.37
			THR62	H-Bond	2.77
			ARG63	Carbon	3.04
			ARG63	Pi-Alkyl	4.94
			PRO64	Carbon	3.37
			SER222	H-Bond	2.13
			ARG255	H-Bond	2.67
8.	**X3_DHPS**	-6.40	THR62	H-Bond	1.90
			THR62	H-Bond	2.92
			PRO64	Alkyl	4.74
			PHE190	Pi-Sigma	3.79
			PHE190	Pi-Sulfur	5.50
			ARG220	Alkyl	4.77
			ARG255	Pi-Cation	3.26
			HIS257	Pi-Alkyl	5.37

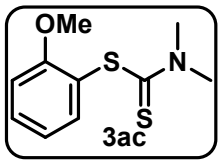
6. Characterization data of the synthesized compounds:



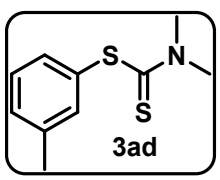
phenyl dimethylcarbamodithioate (3aa)[4]: Yield: 79%, 78 mg; white solid; Mp: 95-95.7 °C; R_f = 0.5 (EA: PE=6: 94); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.49-7.45 (m, 5H), 3.56 (s, 3H), 3.51 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 197.8, 137.1, 131.9, 130.2, 129.3, 45.8, 42.1.



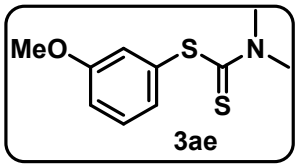
o-tolyl dimethylcarbamodithioate (3ab)[4]: Yield: 71%, 75 mg; white solid; Mp: 81.2-82 °C; R_f = 0.5 (EA: PE = 6: 94); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.44-7.38 (m, 2H), 7.36-7.34 (m, 1H), 7.28-7.24 (m, 2H), 3.56 (s, 3H), 3.52 (s, 3H), 2.41 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.7, 144.0, 137.9, 131.3, 130.9, 130.8, 126.9, 45.7, 42.1, 21.0.



2-methoxyphenyl dimethylcarbamodithioate (3ac)[4]: Yield: 76%, 86 mg; white solid; Mp: 88-89.7 °C; R_f = 0.5 (EA: PE = 10: 90); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.51-7.47 (m, 1H), δ 7.42-7.40 (m, 1H), δ 7.05-7.00 (m, 2H), 3.86 (s, 3H), 3.54 (s, 3H), 3.52 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.9, 160.6, 138.9, 132.6, 121.3, 119.9, 111.9, 56.3, 45.8, 42.1.

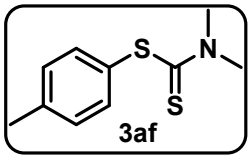


m-tolyl dimethylcarbamodithioate (3ad)[5]: Yield: 75%, 79 mg; white solid; deep yellow liquid; R_f = 0.5 (EA: PE = 6: 94); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.36-7.27 (m, 4H), 3.56 (s, 3H), 3.50 (s, 3H), 2.39 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.0, 139.1, 137.6, 134.1, 131.5, 131.1, 129.1, 45.8, 42.1, 21.4.



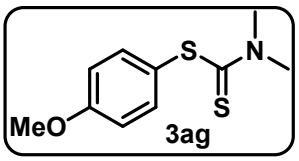
3ae

3-methoxyphenyl dimethylcarbamodithioate (3ae)[5,6]: Yield: 78%, 86 mg; deep yellow liquid; R_f = 0.5 (EA: PE = 10: 90); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.37-7.33(m, 1H), δ 7.09-7.06 (m, 1H), δ 7.04-7.00 (m, 2H), 3.82 (s, 3H), 3.56 (s, 3H), 3.49 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 197.5, 159.9, 132.6, 129.9, 129.2, 122.0, 116.4, 55.5, 45.8, 42.1.



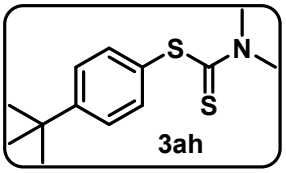
3af

p-tolyl dimethylcarbamodithioate (3af)[4]: Yield: 84%, 88.6 mg; white solid; Mp: 112.8-113.5 °C; R_f = 0.5 (EA: PE = 6: 94); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.30 (d, J = 8 Hz, 2H), δ 7.20 (d, J = 8 Hz, 2H), 3.50 (s, 3H), 3.43 (s, 3H), 2.35 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.2, 140.5, 136.9, 130.1, 128.3, 45.8, 42.1, 21.6.



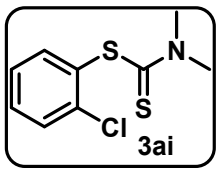
3ag

4-methoxyphenyl dimethylcarbamodithioate (3ag)[4]: Yield: 81%, 92 mg; white solid; Mp: 97-99 °C; R_f = 0.5 (EA: PE = 10: 90); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.38-7.35 (m, 2H), 6.97-6.94 (m, 2H), 3.85 (s, 3H), 3.56 (s, 3H), 3.49 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.9, 161.3, 138.6, 122.7, 114.9, 55.4, 45.9, 42.0.



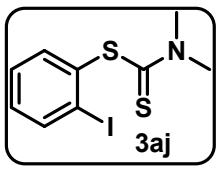
3ah

4-(tert-butyl)phenyl dimethylcarbamodithioate (3ah)[4]: Yield: 80 %, 101 mg; white solid; Mp: 61-62 °C; R_f = 0.5 (EA: PE = 8: 92); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7. 40-7.38 (m, 2H), 7.34-7.31 (m, 2H), 3.48 (s, 3H), 3.42 (s, 3H), 1.28 (s, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.0, 153.3, 136.5, 130.0, 128.3, 126.3, 126.1, 45.8, 42.1, 34.9, 31.3.



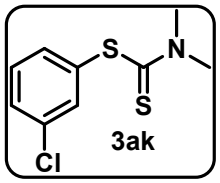
3ai

2-chlorophenyl dimethylcarbamodithioate (3ai)[4]: Yield: 78 %, 90 mg; grey solid; Mp: 105-106.8 °C; R_f = 0.5 (EA: PE = 15: 85); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.57-7.54 (m, 2H), δ 7.45-7.41 (m, 1H), δ 7.36-7.32 (m, 2H), 3.56 (s, 3H), 3.53 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 195.1, 140.7, 139.3, 131.9, 131.1, 130.4, 127.6, 45.8, 42.3.



3aj

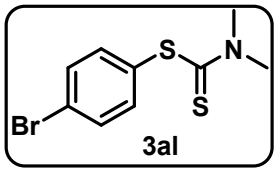
2-iodophenyl dimethylcarbamodithioate (3aj): Yield: 80%, 129 mg; red solid; white solid; Mp 64-66 °C; R_f = 0.45 (EA: PE = 10: 90); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.02-7.99 (m, 1H), 7.64-7.62 (m, 1H), 7.45-7.41 (m, 1H), 7.16-7.11 (m, 1H), 3.57 (s, 3H), 3.53 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 195.2, 140.3, 138.3, 137.4, 131.5, 129.2, 110.5, 45.7, 42.3. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calculated for $[C_9H_{11}INS_2]^+$: 323.9372; Found : 323.9337.



3ak

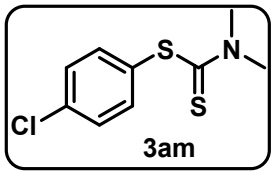
3-chlorophenyl dimethylcarbamodithioate (3ak)[5]: Yield: 77 %, 89 mg; deep yellow liquid ; R_f = 0.5 (EA: PE = 15: 85); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.48-7.47 (m, 1H), δ 7.45-7.41 (m, 1H), δ 7.39-7.36 (m, 2H), 3.55 (s, 3H), 3.48 (s, 3H); ^{13}C

NMR ($CDCl_3$, 100 MHz): δ 196.4, 136.8, 135.2, 134.5, 133.4, 130.3, 130.1, 45.8, 42.1.



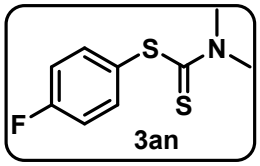
3al

4-bromophenyl dimethylcarbamodithioate (3al)[4]: Yield: 82%, 113 mg; white solid; Mp: 120-121.4 °C; R_f = 0.5 (EA: PE = 15: 85); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.58-7.55 (m, 2H), 7.34-7.30 (m, 2H), 3.55 (s, 3H), 3.48 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.7, 138.5, 132.5, 130.9, 125.1, 45.9, 42.2.



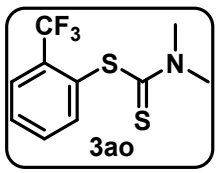
3am

4-chlorophenyl dimethylcarbamodithioate (3am)[4]: Yield: 83 %, 96 mg; white solid; Mp: 100.9-101.4 °C; R_f = 0.5 (EA: PE = 15: 85); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.42-7.37 (m, 4H), 3.55 (s, 3H), 3.48 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.8, 138.3, 136.6, 130.2, 129.5, 45.9, 42.1.



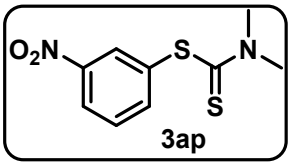
3an

4-fluorophenyl dimethylcarbamodithioate (3an)[4]: Yield: 85%, 92 mg; white solid; Mp: 91.7-92.7 °C; R_f = 0.45 (EA: PE = 10: 90); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.45-7.42 (m, 2H), 7.15- 7.10 (m, 2H), 3.54 (s, 3H), 3.48 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 197.4, 163.9 (d, J = 250 Hz), 139.1 (d, J = 9 Hz), 132.8 (d, J = 9 Hz), 127.2 (d, J = 3 Hz), 116.4 (d, J = 220 Hz), 45.8, 42.



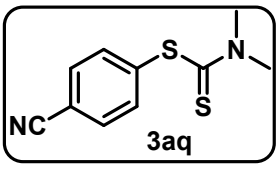
3ao

2-(trifluoromethyl)phenyl dimethylcarbamodithioate (3ao): Yield: 83%, 110 mg; white solid; Mp: 96-98 °C; R_f = 0.5 (EA: PE = 12: 88); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.79 (d, J = 7.2 Hz, 1H), 7.65-7.62 (m, 2H), 7.61-7.57 (m, 1H), 3.54 (s, 3H), 3.53 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 195.9, 141.6, 133.9 (q, J= 310 Hz), 132.3, 130.4 (2C), 126.9 (q, J= 6 Hz), 123.3 (q, J= 273 Hz), 45.7, 42.3. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calculated for$[C_{10}H_{11}F_3NS_2]^+$: 266.0280; Found : 266.0291.



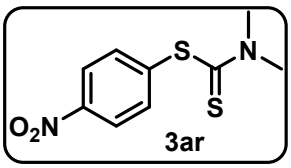
3ap

3-nitrophenyl dimethylcarbamodithioate (3ap): Yield: 82%, 99 mg; yellowish-white; Mp 153-155 °C; R_f = 0.5 (EA: PE = 15: 85); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.32-8.30 (m, 2H), 7.79-7.77 (m, 1H), 7.63-7.59 (m, 1H), 3.55 (s, 3H), 3.52 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 195.2, 148.4, 143.2, 134.0, 131.9, 129.8, 124.9, 45.9, 42.2. HRMS (ESI-TOF) m/z: $[M+Na]^+$ Calculated for$[C_9H_{11}N_2O_2S_2]^+$: 243.0256; Found : 243.0257.



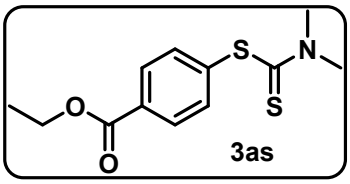
3aq

4-cyanophenyl dimethylcarbamodithioate (3aq)[4]: Yield: 81%, 90 mg; white solid; Mp: 125-126 °C; R_f = 0.5 (EA: PE = 16: 84); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.69 (d, J = 8.4 Hz, 2H), 7.57 (d, J = 8.4 Hz, 2H), 3.54 (s, 3H), 3.50 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 195.0, 137.6, 137.5, 132.5, 118.4, 113.7, 45.8, 42.3.

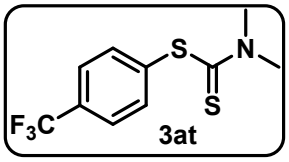


3ar

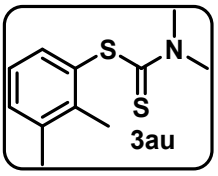
4-nitrophenyl dimethylcarbamodithioate (3ar)[4]: Yield: 87%, 105 mg; yellow solid Mp: 153-154.5 °C; R_f = 0.5 (EA: PE = 20: 80); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.25 (d, J = 8.8 Hz, 2H), 7.64 (d, J = 8.8 Hz, 2H), 3.55 (s, 3H), 3.51 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 194.6, 148.6, 139.6, 137.8, 124.0, 45.8, 42.3.



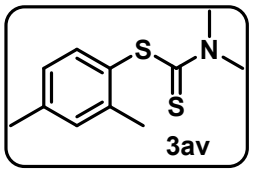
ethyl 4-((dimethylcarbamothioyl)thio)benzoate (3as): Yield: 88%, 119 mg; white solid; Mp: 78-80 °C; R_f = 0.5 (EA: PE = 16: 84); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.09 (d, J = 8.4 Hz, 2H), 7.54 (d, J = 8.4 Hz, 2H), 4.41-4.35 (m, 2H), 3.54 (s, 3H), 3.49 (s, 3H), 1.38 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.1, 166.0, 136.9, 136.8, 131.7, 130.1, 61.3, 45.7, 42.2, 14.4. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calculated for$[C_{12}H_{16}NO_2S_2]^+$: 270.0617; Found : 270.0592.



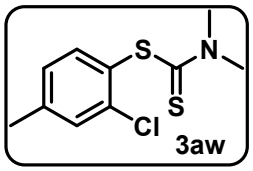
4-(trifluoromethyl)phenyl dimethylcarbamodithioate (3at)[4]: Yield: 84%, 112 mg; white solid; Mp: 85.2-86.2 °C; R_f = 0.5 (EA: PE = 12: 88); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.67 (d, J = 8.4 Hz, 2H), δ 7.59 (d, J = 8.4 Hz, 2H), 3.54 (s, 3H), 3.48 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 195.6, 137.2, 136.1, 132.6 (q, J= 320 Hz), 125.8 (q, J= 4 Hz), 123.9 (q, J= 271 Hz), 45.6, 42.1.



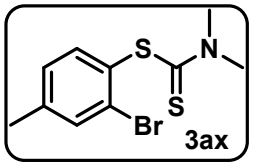
2,3-dimethylphenyl dimethylcarbamodithioate (3au): Yield: 74 %, 83.5 mg; yellow solid; Mp 71-73 °C; R_f = 0.5 (EA: PE = 6: 94); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.32-7.27 (m, 2H), 7.15 (t, J = 8.4 Hz, 1H), 3.56 (s, 3H), 3.52 (s, 3H), 2.35 (s, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 197.2, 142.5, 138.1, 135.6, 132.5, 131.4, 126.2, 45.7, 42.1, 21.2, 17.6. Anal. Calcd. For $C_{11}H_{15}NS_2$: C, 58.62; H, 6.71; N, 6.22%; Found: C, 58.54; H, 6.65; N, 6.15%.



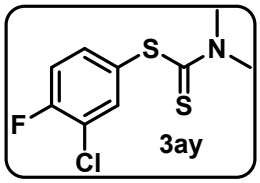
2,4-dimethylphenyl dimethylcarbamodithioate (3av): Yield: 76%, 85.5 mg; red solid; Mp 76 -78 °C; R_f = 0.5 (EA: PE = 6: 94); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.31-7.27 (m, 1H), 7.20-7.18 (m, 2H), 3.56 (s, 6H), 2.40 (s, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 195.7, 144.2, 131.1, 130.4, 128.4, 45.6, 42.1, 29.8, 21.9. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calculated for$[C_{11}H_{16}NS_2]^+$: 226.0719; Found : 226.0712.



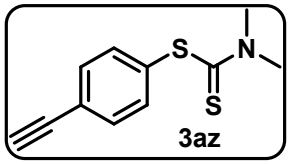
2-chloro-4-methylphenyl dimethylcarbamodithioate (3aw): Yield: 74 %, 91 mg; white solid; Mp: 96-98 °C; R_f = 0.5 (EA: PE = 15: 85); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.42 (d, J = 8 Hz, 1H), 7.38 (s, 1H), 7.15 (d, J = 7.6 Hz, 1H), 3.56 (s, 3H), 3.52 (s, 3H), 2.39 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 195.7, 142.8, 140.3, 138.9, 131.1, 128.6, 127.6, 45.8, 42.2, 21.4. HRMS (ESI-TOF) m/z: $[M+Na]^+$ Calculated for$[C_{10}H_{12}ClNNaS_2]^+$: 267.9992; Found : 267.9979.



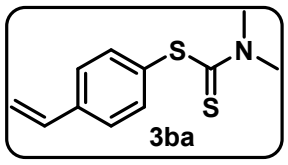
2-bromo-4-methylphenyl dimethylcarbamodithioate (3ax): Yield: 80 %, 116 mg; white solid; Mp: 85-87 °C; R_f = 0.5 (EA: PE = 15: 85); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.57 (s, 1H), 7.45 (d, J = 8 Hz, 1H), 7.20- 7.18 (m, 1H), 3.56 (s, 3H), 3.51 (s, 3H), 2.38 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 195.6, 142.7, 138.9, 134.3, 131.7, 129.8, 126.2, 45.8, 42.2, 21.3. HRMS (ESI-TOF) m/z: $[M+Na]^+$ Calculated for$[C_{10}H_{12}BrNNaS_2]^+$: 311.9487; Found : 311.9492.



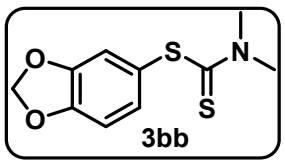
3-chloro-4-fluorophenyl dimethylcarbamodithioate (3ay): Yield: 78%, 97.5 mg; gummy mass; R_f = 0.45 (EA: PE = 15: 85); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.52-7.50 (m, 1H), 7.35-7.31 (m, 1H), 7.19 (t, J = 8.8 Hz, 1H), 3.54 (s, 3H), 3.47 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.3, 159.3 (d, J = 252 Hz), 139.1, 137.2 (d, J= 8 Hz), 128.3 (d, J = 4 Hz), 121.5 (d, J = 18 Hz), 117.2 (d, J = 22 Hz), 45.9, 42.0. Anal. Calcd. For: $C_9H_9ClFNS_2$: C, 43.28; H, 3.63; N, 5.61%; Found: C, 43.36; H, 3.54; N, 5.55%.



4-ethynylphenyl dimethylcarbamodithioate (3az): Yield: 78 %, 86.5 mg; gummy mass; R_f = 0.5 (EA: PE = 10: 90); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7. 55-7.53 (m, 2H), 7.43-7.41 (m, 2H), 3.53 (s, 3H), 3.47 (s, 3H), 3.18 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.5, 136.8, 132.6, 128.5, 123.9, 83.0, 79.2, 45.7, 42.1. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calculated for$[C_{11}H_{12}NS_2]^+$: 222.0406; Found : 222.0398.

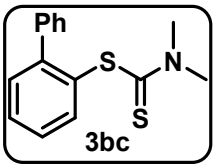


4-vinylphenyl dimethylcarbamodithioate (3ba)[5]: Yield: 70 %, 78 mg; light yellow solid; Mp. 66-67 °C; R_f = 0.5 (EA: PE = 10: 90); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.49-7.47 (m, 2H), 7.43-7.41 (m, 2H), 6.78 -6.70 (m, 1H), δ 5.82 (d, J = 17.6 Hz, 1H), 5.34 (d, J = 11.2 Hz, 2H), 3.56 (s, 3H), 3.50 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 197.7, 139.4, 137.2, 136.3, 130.9, 127.1, 115.8, 45.8, 42.1.

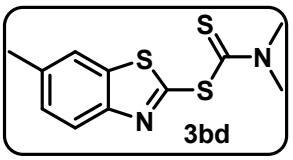


benzo[d][1,3]dioxol-5-yl dimethylcarbamodithioate (3bb): Yield: 79%, 95.5 mg; white solid; Mp: 111-113 °C; R_f = 0.45 (EA: PE = 15: 85); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.97-6.95 (m, 1H), 6.90 (d, J = 1.6 Hz, 1H), 6.86 (d, J = 8.4 Hz, 1H), 6.03 (s, 2H), 3.55 (s, 3H), 3.47 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.4, 149.6, 148.1,

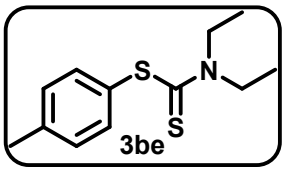
131.6, 123.8, 117.0, 109.0, 101.8, 45.9, 42. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calculated for $[C_{10}H_{12}NO_2S_2]^+$: 242.0304; Found : 242.2999.



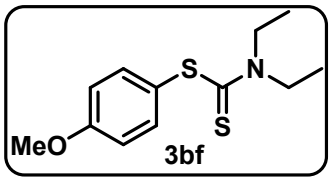
[1,1'-biphenyl]-2-yl dimethylcarbamodithioate (3bc): Yield: 85 %, 116 mg; white solid; Mp: 122-124 °C; R_f = 0.5 (EA: PE = 10: 90); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.60 -7.58 (m, 1H), 7.56 -7.52 (m, 1H), 7.46-7.42 (m, 4H), 7.39-7.34 (m, 3H), 3.50 (s, 3H), 3.33 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 197.8, 147.4, 141.0, 139.2, 131.1, 130.5, 130.4, 129.5, 128.2, 127.7, 127.4, 45.7, 42.2. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calculated for$[C_{15}H_{16}NS_2]^+$: 274.0719; Found : 274.0731.



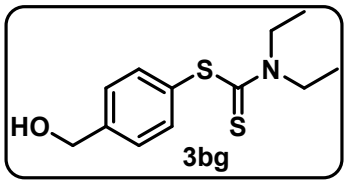
6-methylbenzo[d]thiazol-2-yl dimethylcarbamodithioate (3bd): Yield: 81 %, 108.5 mg; white solid; Mp: 144-146 °C R_f = 0.5 (EA: PE = 20: 80); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.73 (d, J = 8.0 Hz 2H), 7.34 (d, J = 8 Hz 2H), 7.29 (d, J = 6.8 Hz 2H), 3.53 (s, 3H), 3.50 (s, 3H), 2.78 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 192.0, 157.5, 152.5, 138.6, 134.2, 127.0, 126.3, 119.1, 45.5, 42.6, 18.7. Anal. Calcd. For: $C_{11}H_{12}N_2S_3$: C, 49.22; H, 4.51; N, 10.44%; Found: C, 49.31; H, 4.60; N, 10.36%



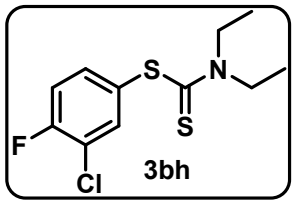
p-tolyl diethylcarbamodithioate (3be)[4]: Yield: 82 %, 98 mg; white solid; Mp: 75-76 °C; R_f = 0.5 (EA: PE = 6: 94); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.38 (d, J = 8.0 Hz 2H), 7.27 (d, J = 7.6 Hz 2H), 4.08-4.03 (m, 2H), 3.91-3.85 (m, 2H), 2.42 (s, 3H), 1.42 (t, J = 7.2 Hz, 3H), 1.31 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.6, 140.4, 137.1, 130.1, 128.3, 50.0, 47.3, 21.6, 12.8, 11.7.



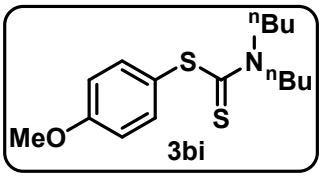
4-methoxyphenyl diethylcarbamodithioate (3bf)[4]: Yield: 80 %, 102 mg; white solid; Mp: 73-75 °C; R_f = 0.5 (EA: PE = 15: 85); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.38 (d, J = 8.8 Hz 2H), 6.95 (d, J = 8.8 Hz 2H), 4.05-4.00 (m, 2H), 3.88-3.86 (m, 2H), 3.84 (s, 3H), 1.39 (t, J = 7.2 Hz, 3H), 1.28 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 197.2, 161.1, 138.7, 122.5, 114.7, 55.4, 50.1, 47.2, 12.8, 11.7.



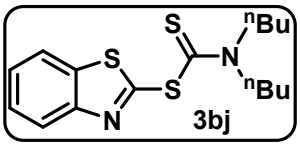
4-(hydroxymethyl)phenyl diethylcarbamodithioate (3bg): Yield: 85%, 108.5 mg; yellow gummy mass; R_f = 0.5 (EA: PE = 20: 80); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.49-7.43 (m, 1H), 4.74 (d, J = 7.2 Hz, 2H), 4.03(s, 3H), 3.87 (s, 3H), 1.88 (s, 1H), 1.4 (s, 1H), 1.30 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.2, 143.1, 137.4, 130.7, 127.5, 65.0, 50.0, 47.4, 12.9, 11.7. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calculated for $[C_{12}H_{18}NOS_2]^+$: 256.0824; Found : 256.0818.



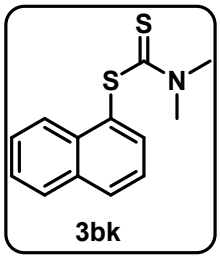
3-chloro-4-fluorophenyl diethylcarbamodithioate (3bh): Yield: 83%, 115.5 mg; yellow liquid; R_f = 0.5 (EA: PE = 15: 85); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.53-7.51 (m, 1H), 7.36-7.32 (m, 1H), 7.18 (t, J = 8.4 Hz, 3H), 4.03-3.98 (m, 2H), 3.85-3.79 (m, 2H), 1.38 (t, J = 7.2 Hz, 3H), 1.27 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 194.6, 159.3 (d, J = 252 Hz), 139.3, 137.4 (d, J = 9 Hz), 128.2 (d, J = 4 Hz), 121.4 (d, J = 17 Hz), 117.1 (d, J = 22 Hz), 50.1, 47.4, 12.8, 11.6. Anal. Calcd. For: $C_{11}H_{13}ClFNS_2$: C, 47.56; H, 4.72; N, 5.04%; Found: C, 47.65; H, 4.79; N, 5.12%.



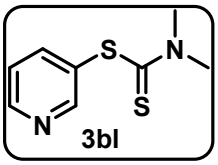
4-methoxyphenyl dibutylcarbamodithioate (3bi)[4]: Yield: 79 %, 123 mg; yellow oil; R_f = 0.5 (EA: PE = 8: 92); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.39-7.36 (m, 2H), 6.95-6.93 (m, 2H), 3.94 (t, J = 8 Hz, 2H), 3.81 (s, 3H), 3.75 (t, J = 8 Hz, 2H), 1.84- 1.75 (m, 2H), 1.73 -1.68 (m, 2H), 1.45 -1.40 (m, 2H) 1.37- 1.32 (m, 2H), 1.01 (t, J = 7.2 Hz, 3H), 0.95 (t, J = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 197.1, 160.8, 138.4, 122.4, 114.4, 55.3, 55.1, 52.7, 29.4, 28.3, 20.0, 13.7, 13.6.



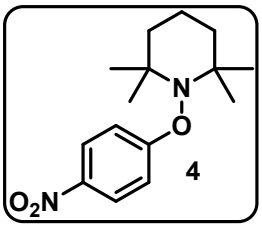
benzo[d]thiazol-2-yl dibutylcarbamodithioate (3bj)[7]: Yield: 76%, 151.5 mg; yellow liquid; R_f = 0.5 (EA: PE = 20: 80); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.11 (d, J = 8 Hz, 2H), 7.9 (d, J = 8 Hz, 2H), 7.52-7.42 (m, 2H),), 3.92 (t, J = 8 Hz, 2H), 3.75 (t, J = 8Hz, 2H), 1.86 -1.80 (m, 2H), 1.78 -1.70 (m, 2H), 1.48-1.40 (m, 2H), 1.38-1.31 (m, 2H), 1.00 (t, J = 7.6 Hz, 3H), 0.94 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 190.1, 159.6, 152.8, 138.4, 126.4, 126.1, 124.0, 121.5, 55.2, 53.9, 51.3, 30.0, 28.3, 20.2, 13.9, 13.8.



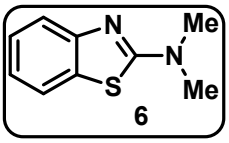
naphthalen-1-yl dimethylcarbamodithioate (3bk)[4]: Yield: 74%, 91.5 mg; yellow solid; Mp: 150.8-152.8 °C ; R_f = 0.5 (EA: PE = 10: 90); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.25 (d, J = 8.4 Hz, 1H), 8.00 (d, J = 8.4 Hz, 1H), 7.89 (d, J = 7.6 Hz, 1H), 7.75 (d, J = 7.2 Hz, 1H), 7.58-7.50 (m, 3H),), 3.63 (s, 3H), 3.57 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.7, 137.2, 135.2, 134.3, 131.7, 129.1, 128.8, 127.4, 126.5, 125.9, 45.7, 42.3.



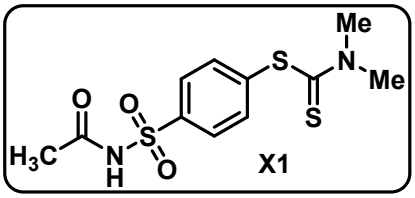
pyridin-3-yl dimethylcarbamodithioate (3bl)[4]**:** Yield: 78%, 77 mg; brown solid; Mp: 58-58.6 °C; R_f = 0.5 (EA: PE = 20: 80); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.64 (d, J = 4.8 Hz, 1H), 8.58 (s, 1H), 7.76 (d, J = 8 Hz, 1H), 7.38-7.35 (m, 1H),), 3.52 (s, 3H), 3.49 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 195.8, 156.4, 150.4, 144.6, 129.4, 124.0, 45.9, 42.1.



2,2,6,6-tetramethyl-1-(4-nitrophenoxy)piperidine (**4**)[8–10]: Yield: 43%, 60 mg; yellowish white; M.p: 73−75 °C; R_f = 0.5 (EA: PE = 4: 96); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.15-8.12 (m, 2H), 7.28-7.25 (m, 2H), 1.66- 1.58 (m, 5H), 1.46- 1.42 (m, 1H), 1.23 (s, 6H), 0.98 (s, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.8, 141.2, 125.7, 114.3, 61.0, 39.8, 32.4, 20.6, 17.0.

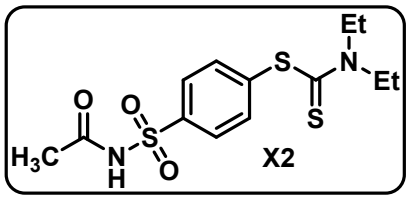


N,N-dimethylbenzo[d]thiazol-2-amine (6)[11]**:** Yield: 82%, 73 mg; Brown solid, Mp: 83–85°C.; R_f = 0.5 (EA: PE = 15: 85); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.59-7.56 (m , 2H), 7.31-7.26 (m, 1H), 7.07-7.03 (m , 2H), 3.16 (s , 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.8, 153.3, 131.2, 126.0, 121.0, 120.7, 118.8, 40.2.

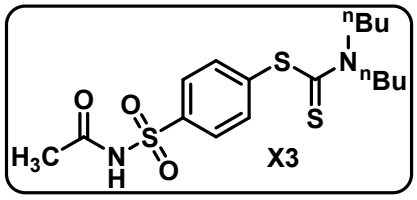


4-(*N*-acetylsulfamoyl)phenyl dimethylcarbamodithioate (X1): Yield: 71 %, 113 mg; white solid; Mp: 135-137 °C; R_f = 0.5 (EA: PE = 30: 70); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.09-8.06 (m, 2H), 7.66 -7.63 (m, 2H), 3.55 (m, 3H), 3.51 (m, 3H), 2.07 (m,

3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 194.9, 168.5, 139.7, 138.9, 137.3, 128.7, 45.8, 42.4, 23.8. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calculated for$[C_{11}H_{15}N_2O_3S_3]^+$: 319.0239; Found : 319.0212.



4-(*N*-acetylsulfamoyl)phenyl diethylcarbamodithioate (X2): Yield: 74 %, 128 mg; white solid; Mp: 144-146 °C ; R_f = 0.5 (EA: PE = 25: 75); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 9.17 (s, 1H), 8.07 (d, J = 8.4 Hz, 2H), 7.66 (d, J = 8.4 Hz, 2H), 4.03- 3.98 (m, 2H), 3.88 - 3.82 (m, 2H), 2.07 (m, 3H), 1.40 (t , J = 7.2 Hz, 2H), 1.28 (t , J = 7.6 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 193.2, 168.6, 139.5, 138.8, 137.4, 128.6, 50.0, 47.8, 23.7, 12.9, 11.6. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calculated for$[C_{13}H_{19}N_2O_3S_3]^+$: 347.0552; Found : 347.0572.



4-(*N*-acetylsulfamoyl)phenyl dibutylcarbamodithioate (X3): Yield: 78 %, 157 mg; white solid; Mp: 120-122 °C; R_f = 0.5 (EA: PE = 20: 80); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.05 (d , J = 8.4 Hz, 2H), 7.63 (d , J = 8.4 Hz, 2H), 3.91 (t , J = 7.6 Hz, 2H), 3.75 (t , J = 7.6 Hz, 2H), 2.04 (m, 3H), 1.83 -1.76 (m, 2H), 1.74 -1.67 (m, 2H), 1.45-1.39 (m, 2H), 1.36-1.30 (m, 2H), 0.99 (t, J = 7.6 Hz, 3H), 0.92 (t, J = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 193.5, 168.9, 139.5, 138.8, 137.2, 128.5, 55.4, 53.5, 29.7, 28.4, 23.7, 20.1, 13.9, 13.8. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calculated for$[C_{17}H_{27}N_2O_3S_3]^+$: 403.1178; Found : 403.1195.

7. References:

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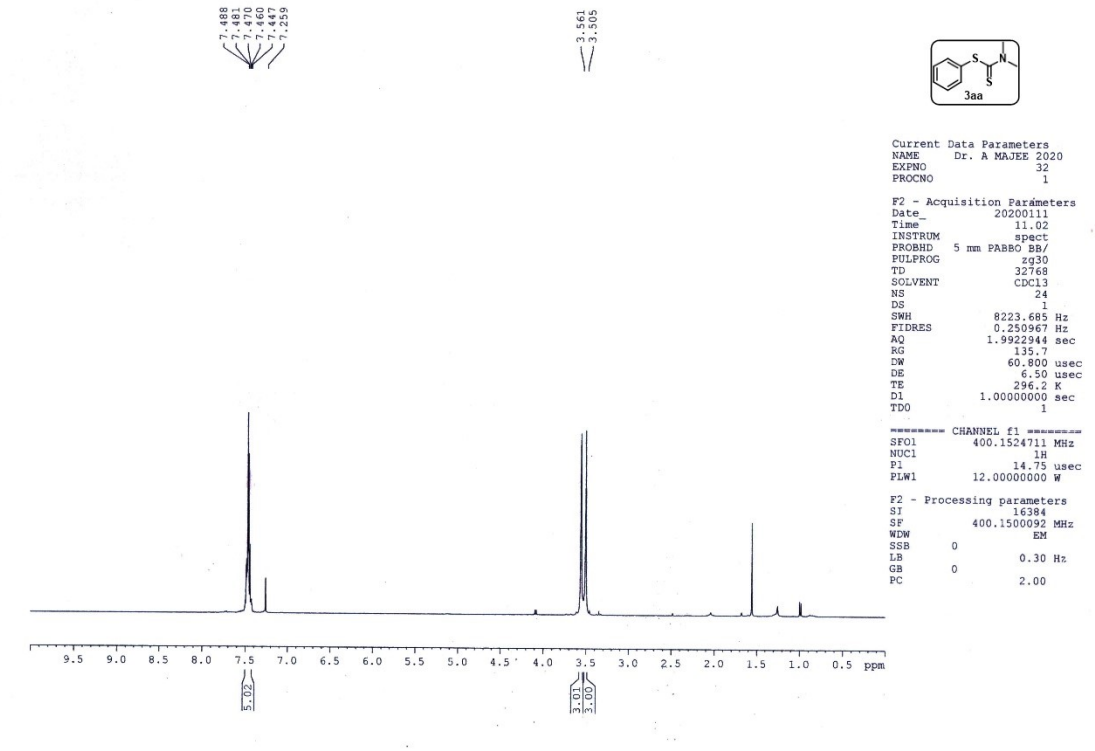
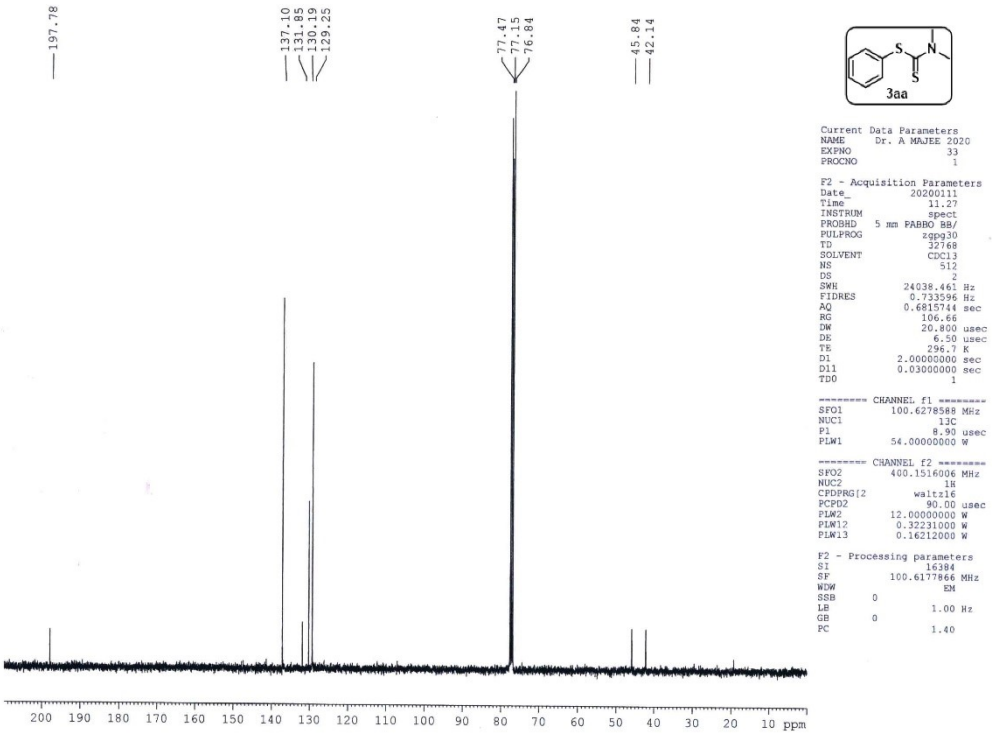
8. NMR spectra [^{1}H, and ^{13}C{^{1}H}] of synthesized products:

^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



$^{13}C\{^1H\}$ NMR: 100 MHz; Solvent: $CDCl_3$



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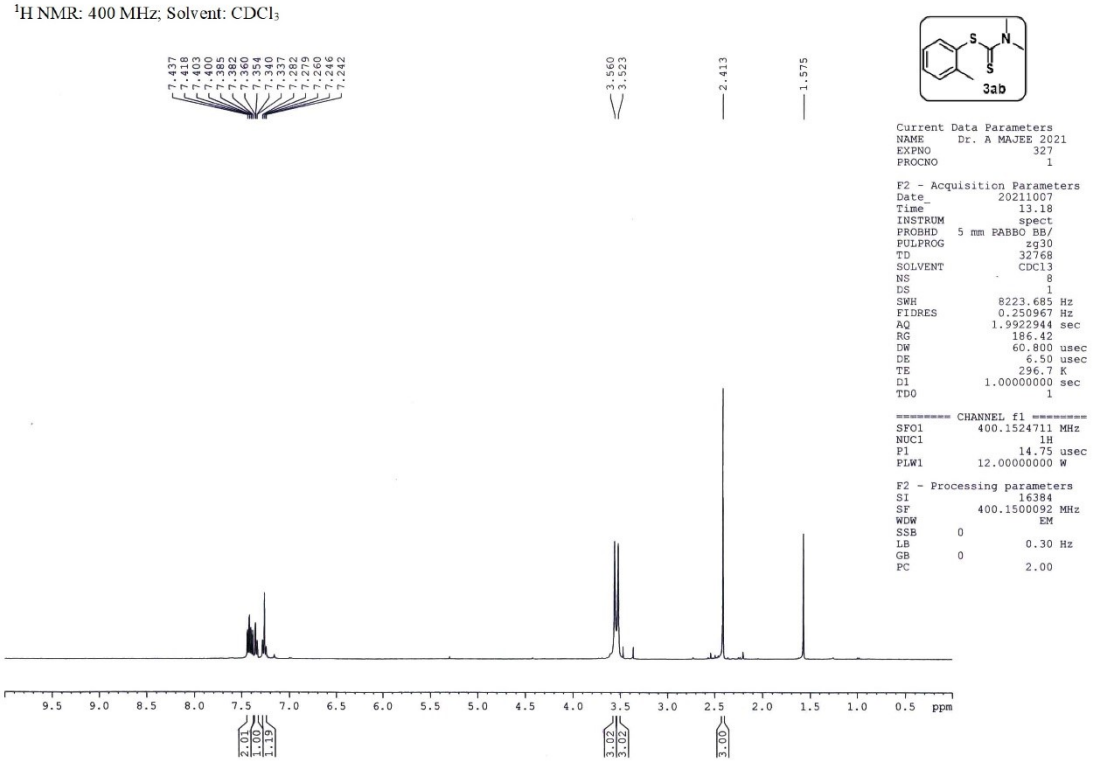
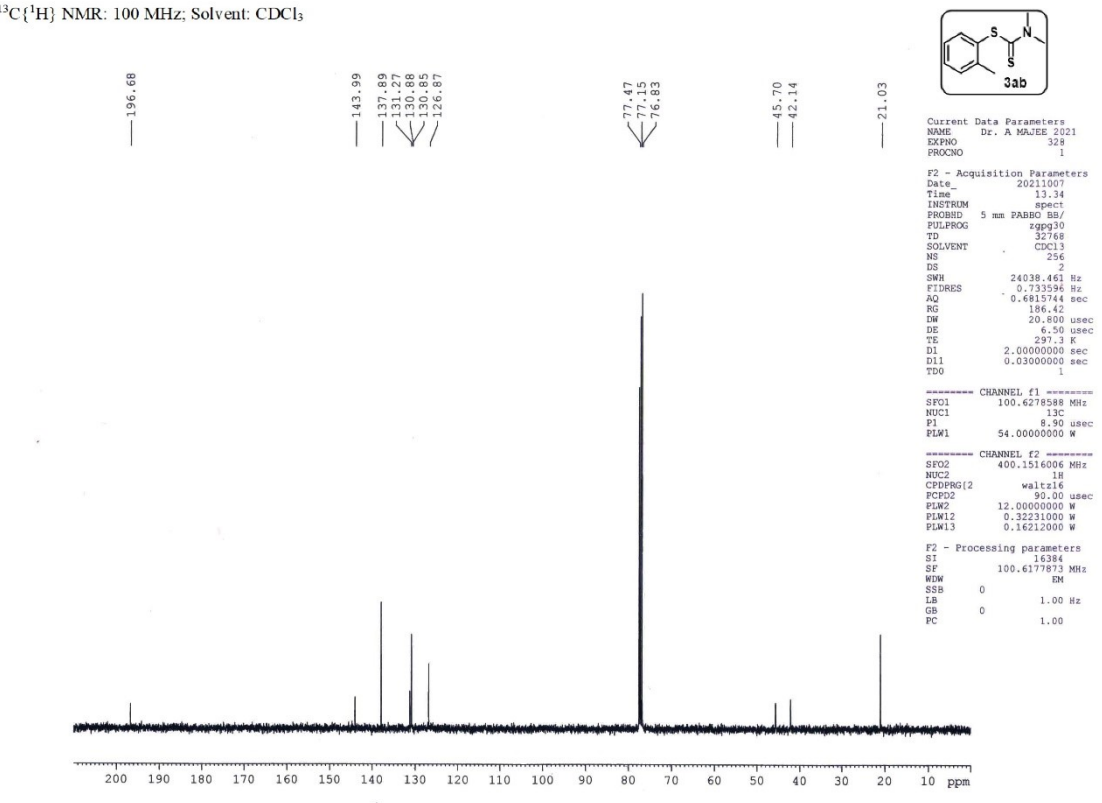
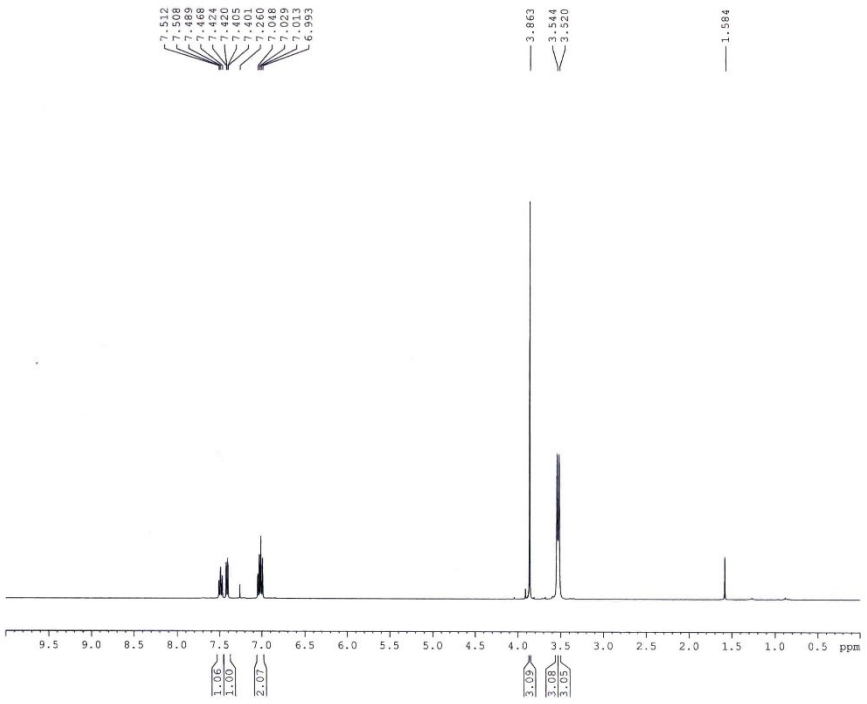
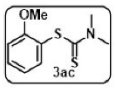


^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



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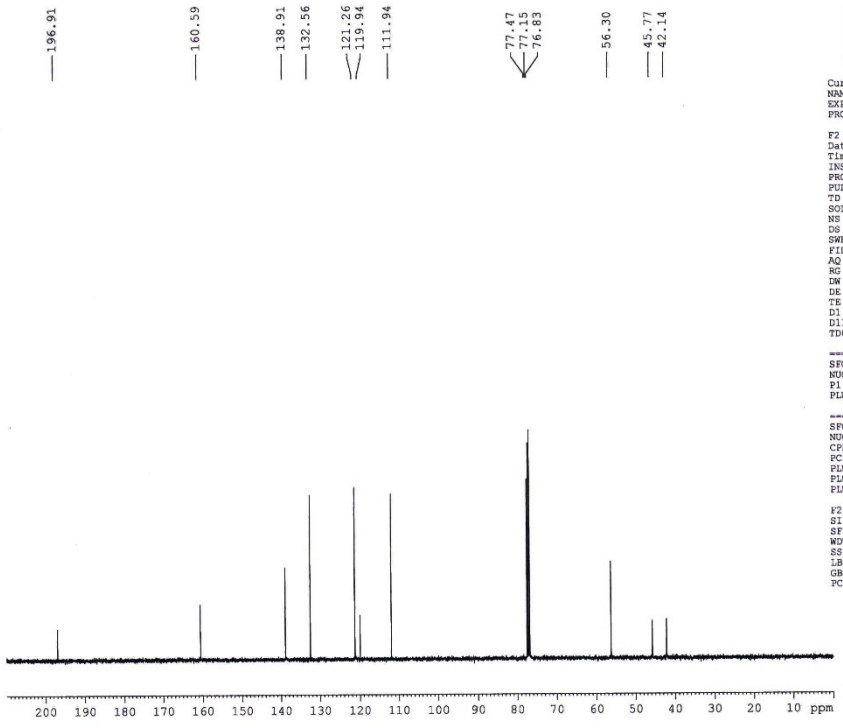

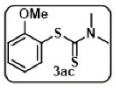


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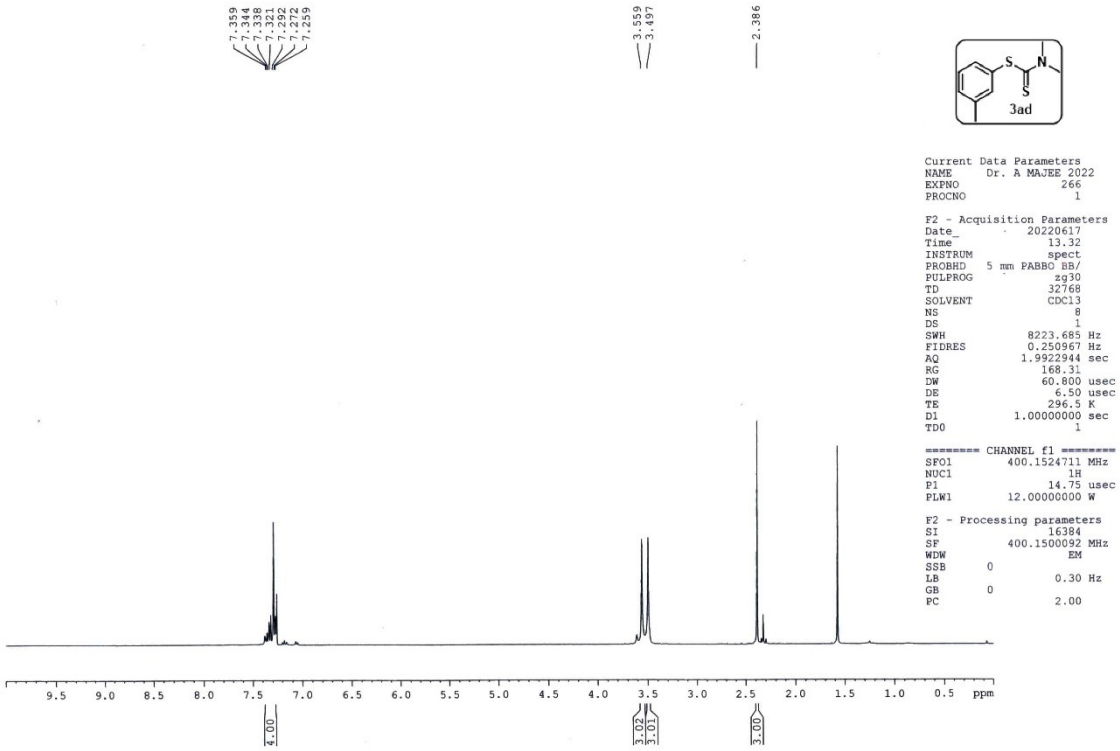

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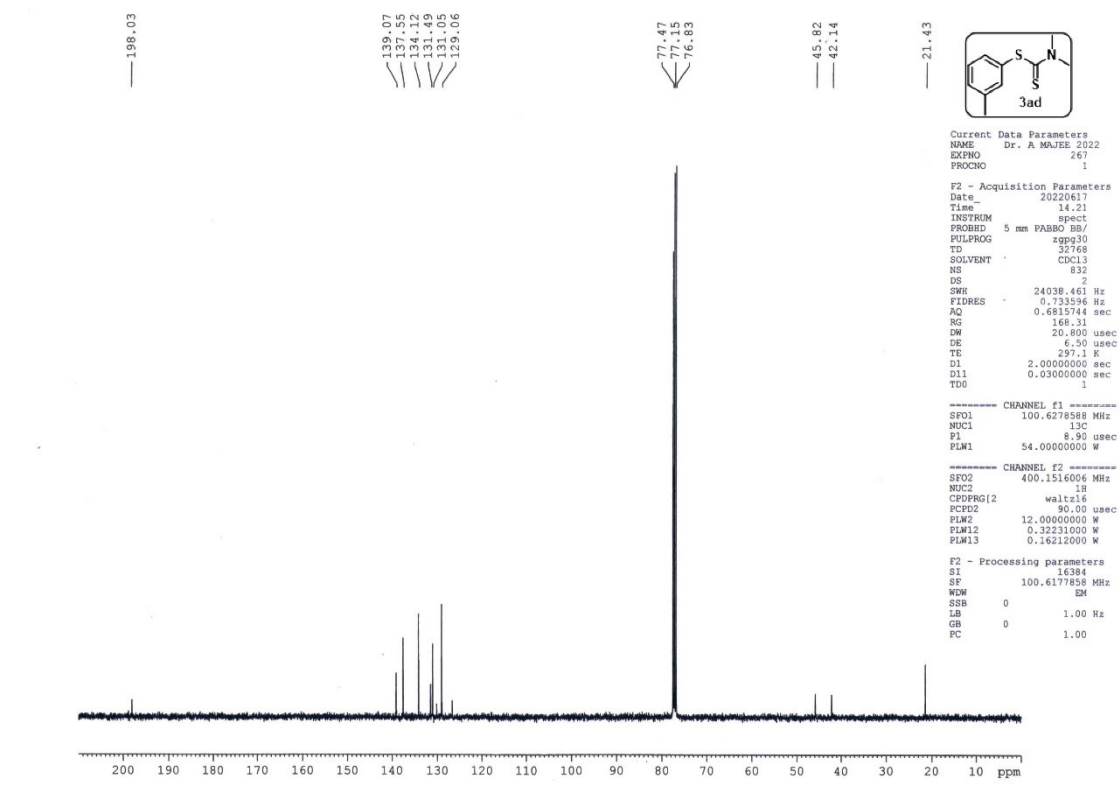

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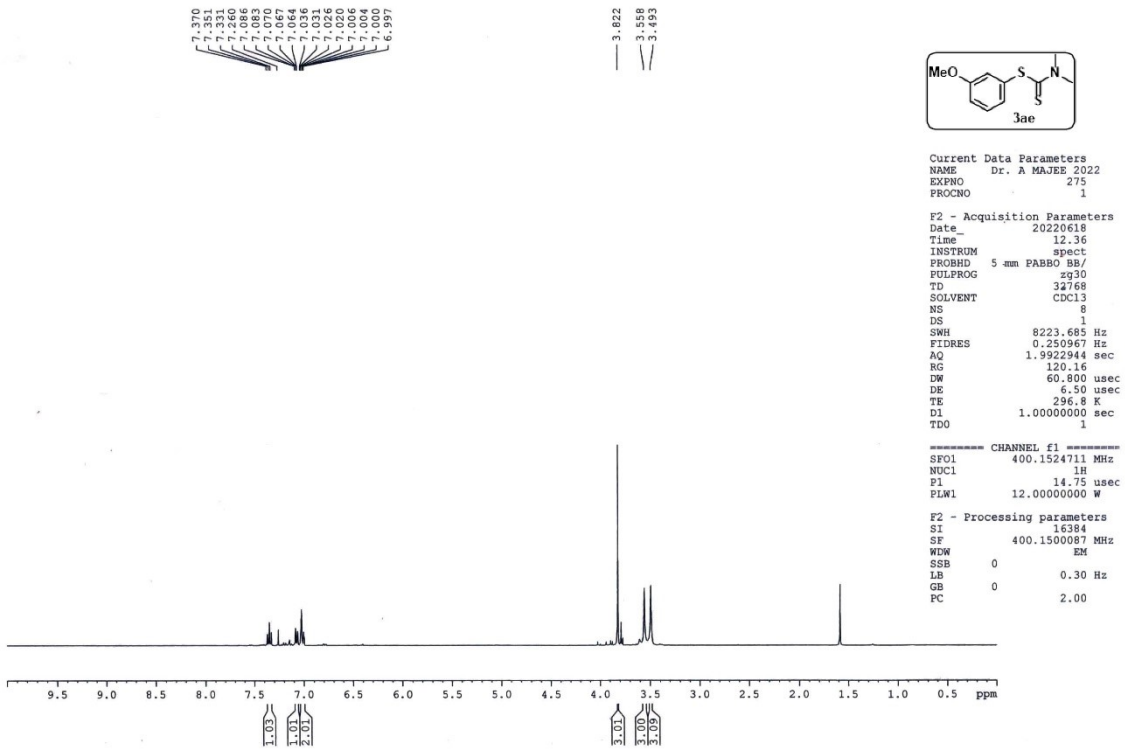

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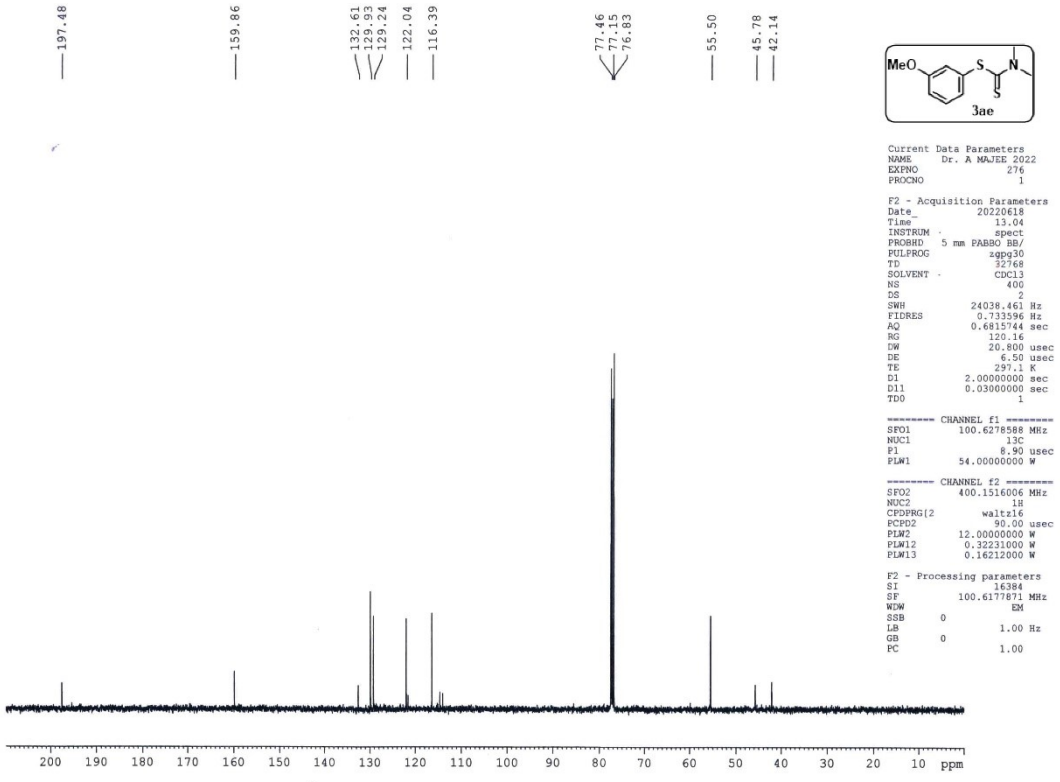

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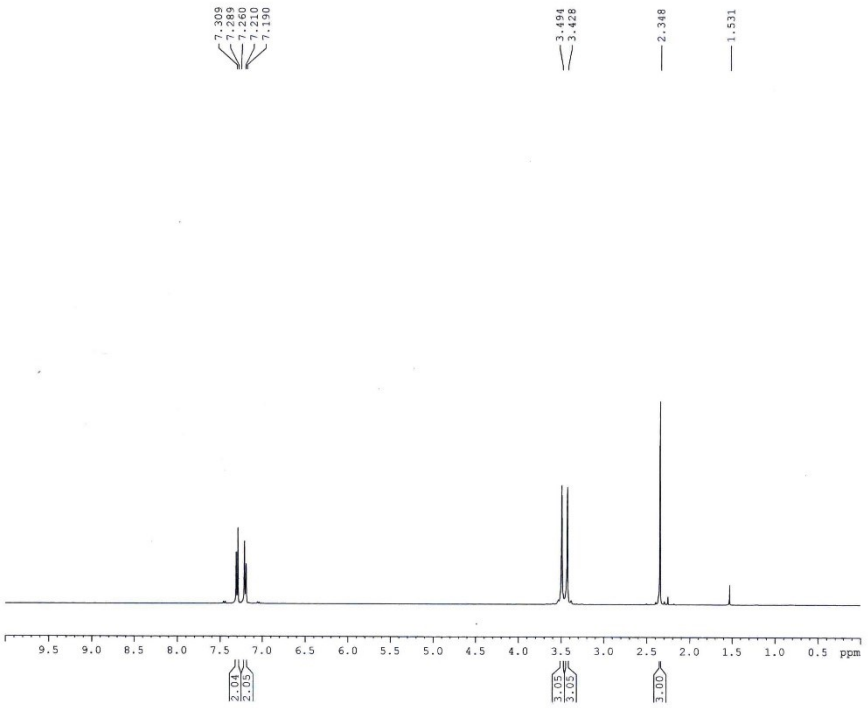


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TE                293.1 K
D1             1.00000000 sec
TD0                      1

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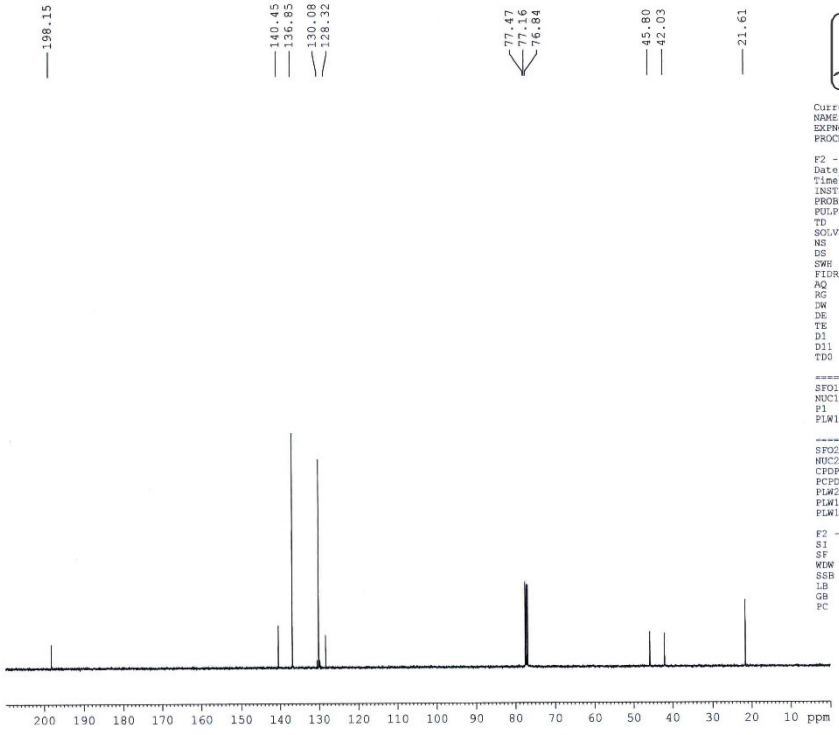
```

^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$




```
Current Data Parameters
NAME      Dr. A MAJEE 2021
EXPNO                  56
PROCNO                  1

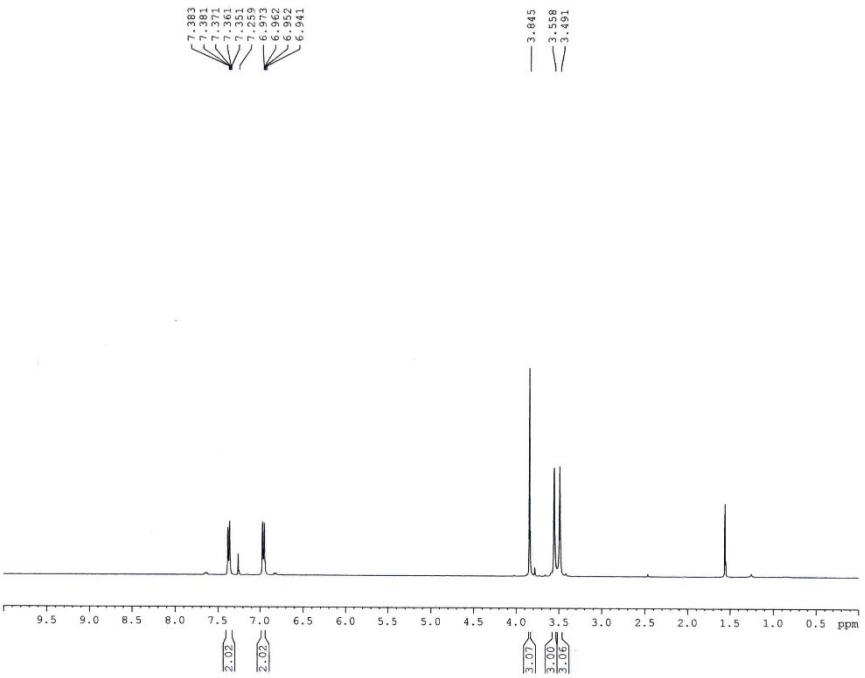
F2 - Acquisition Parameters
Date_              20210126
Time                 23.38
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG            zgpg30
TD                  32768
SOLVENT              CDCl3
NS                     160
DS                       2
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                   62.69
DW                20.800 usec
DE                 6.50 usec
TE                293.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          100.6278588 MHz
NUC1                   13C
P1                    8.90 usec
PLW1          54.00000000 W

======== CHANNEL f2 ========
SFO2          400.1516006 MHz
NUC2                    1H
CPDPRG[2          waltz16
PCPD2                90.00 usec
PLW2          12.00000000 W
PLW12          0.32231000 W
PLW13          0.16212000 W

F2 - Processing parameters
SI                   16384
SF             100.6177962 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



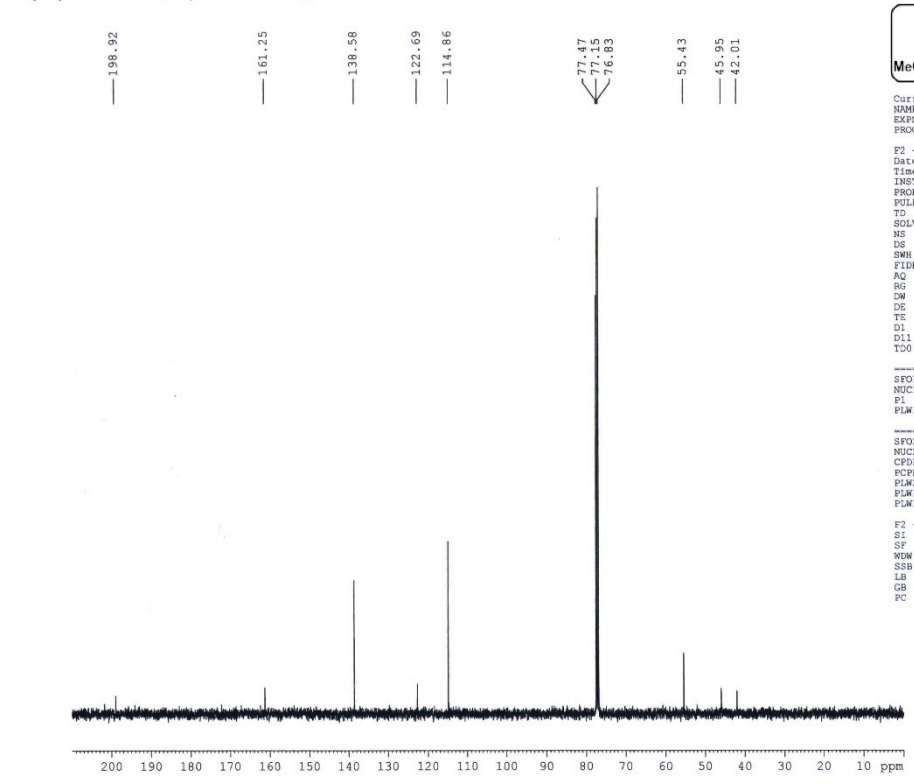

```
Current Data Parameters
NAME      Dr. A MAJEE 2020
EXPNO                  45
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200120
Time                12.07
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     1
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              1.9922944 sec
RG                 120.16
DW                 60.800 usec
DE                  6.50 usec
TE                 295.9 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1524711 MHz
NUC1                   1H
P1                 14.75 usec
PLW1          12.00000000 W

F2 - Processing parameters
SI                 16384
SF            400.1500093 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  2.00
```

^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$




```
Current Data Parameters
NAME      Dr. A MAJEE 2020
EXPNO                  46
PROCNO                  1

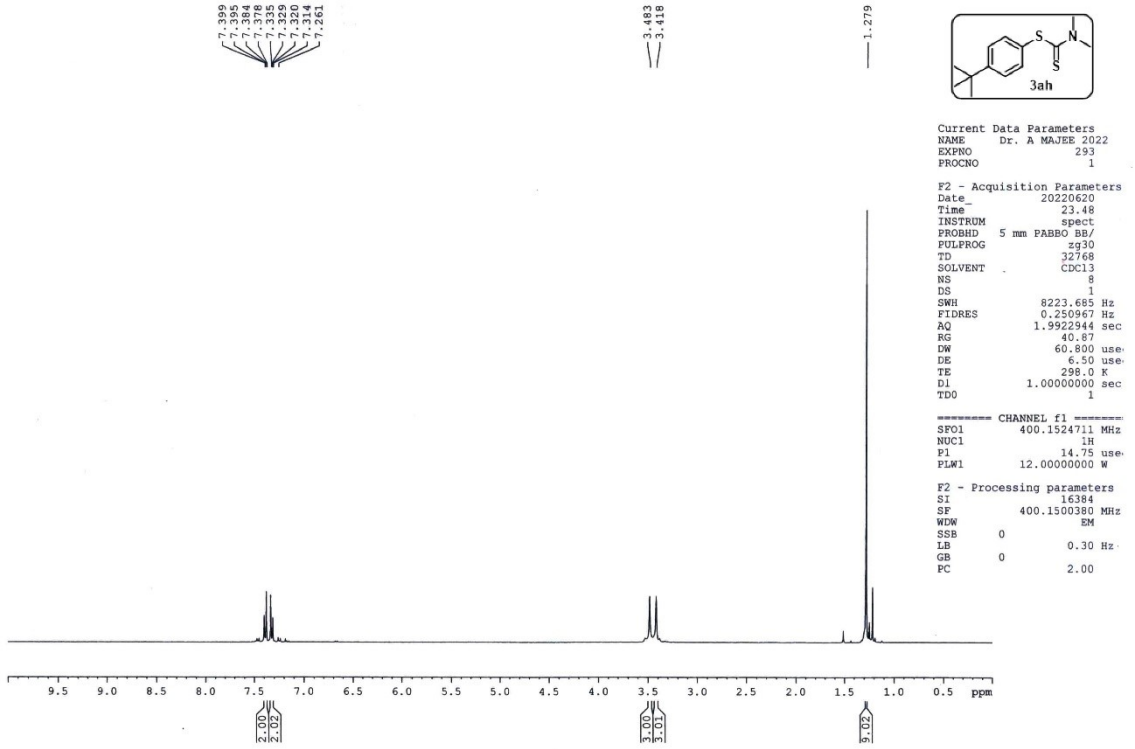
F2 - Acquisition Parameters
Date_            20200120
Time                12.20
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   256
DS                     2
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                  57.28
DW                 20.800 usec
DE                  6.50 usec
TE                 296.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6278588 MHz
NUC1                   13C
P1                  8.90 usec
PLW1          54.00000000 W

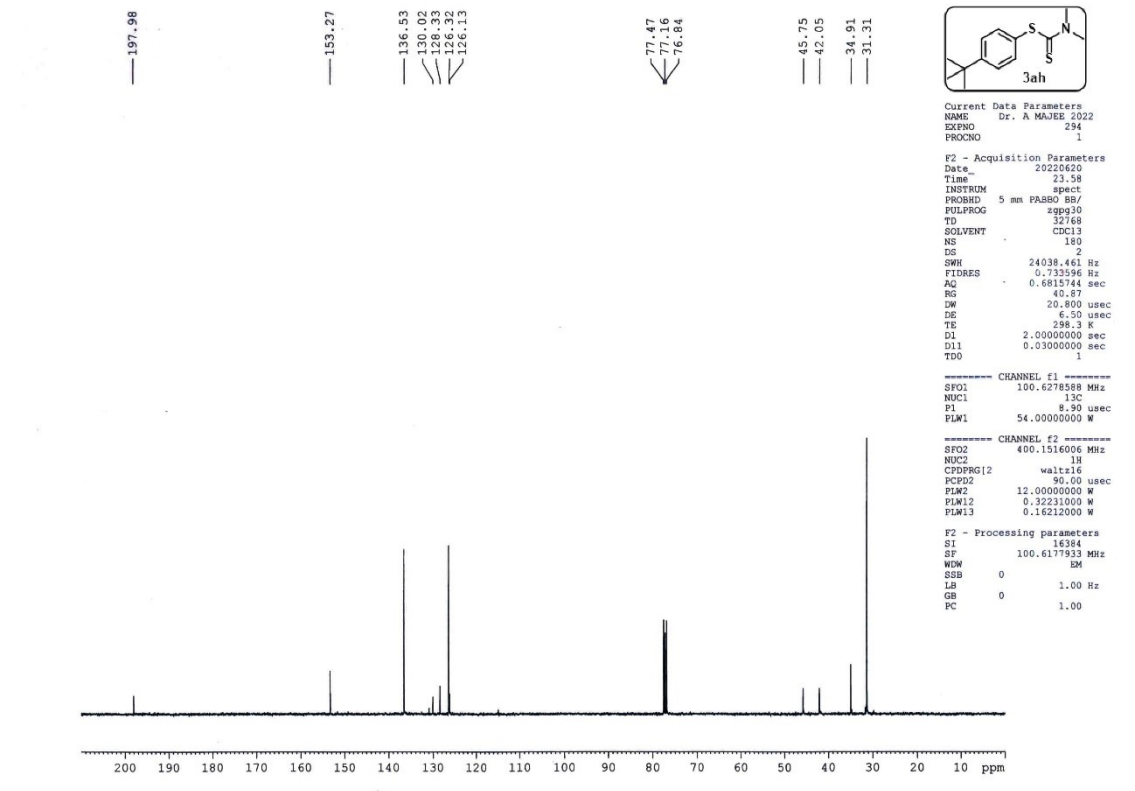
======== CHANNEL f2 ========
SFO2          400.1516006 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          12.00000000 W
PLW12          0.32231000 W
PLW13          0.16212000 W

F2 - Processing parameters
SI                 16384
SF            100.6177858 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

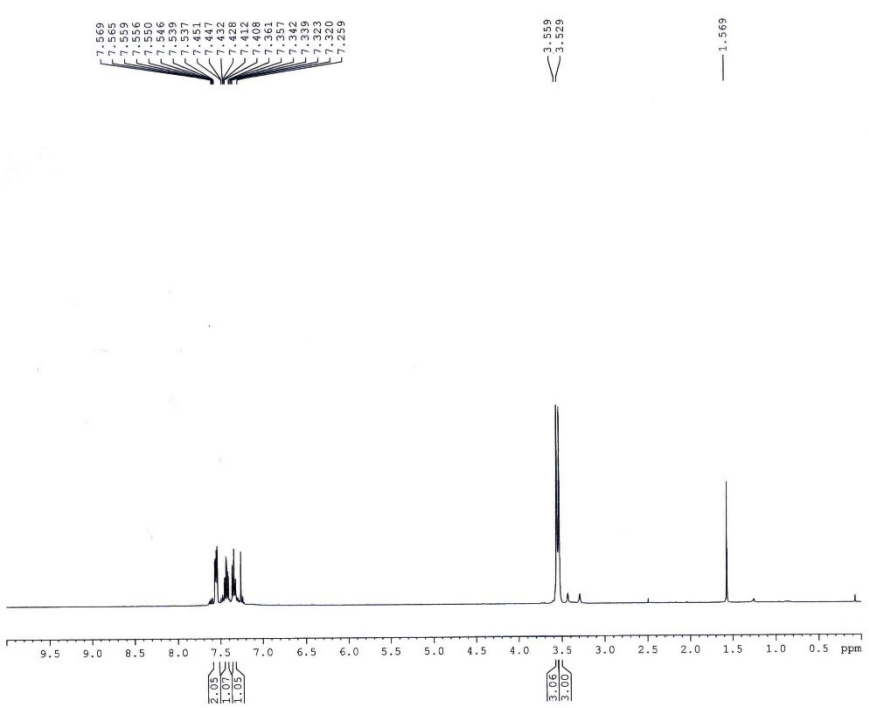
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$

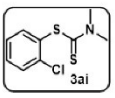


^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



^{1}H NMR: 400 MHz; Solvent: $CDCl_3$





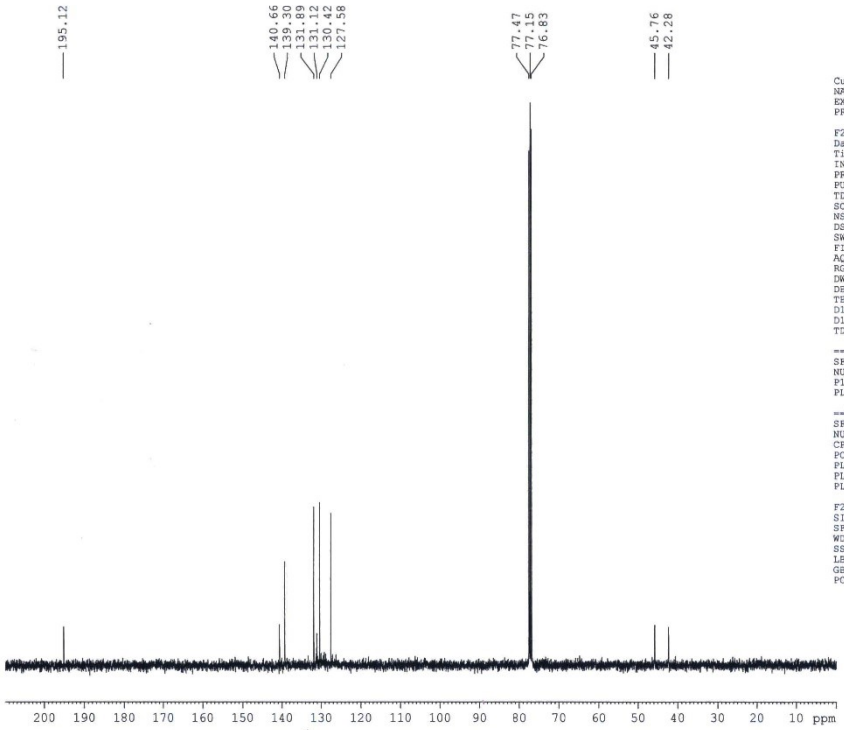

```
Current Data Parameters
NAME       Dr. A MAJEE 2021
EXPNO                  43
PROCNO                  1

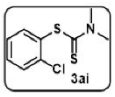
F2 - Acquisition Parameters
Date_           20210119
Time              19.59
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   8
DS                   1
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9922944 sec
RG                135.7
DW               60.800 usec
DE                6.50 usec
TE               295.6 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1524711 MHz
NUC1               1H
P1               14.75 usec
PLW1        12.00000000 W

F2 - Processing parameters
SI                16384
SF          400.1500092 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               2.00
```

^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$






```
Current Data Parameters
NAME       Dr. A MAJEE 2021
EXPNO                  44
PROCNO                  1

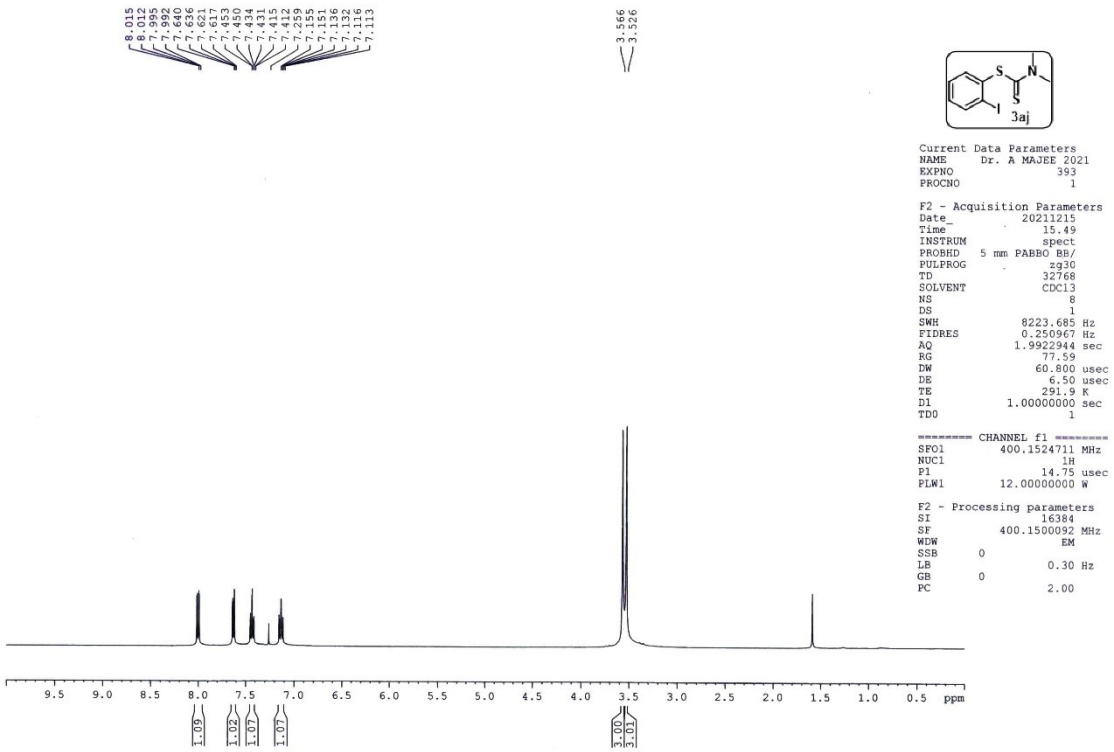
F2 - Acquisition Parameters
Date_           20210119
Time              20.13
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               32768
SOLVENT          CDCl3
NS                 256
DS                   2
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG                135.7
DW               20.800 usec
DE                6.50 usec
TE               296.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6278588 MHz
NUC1               13C
P1                8.90 usec
PLW1        54.00000000 W

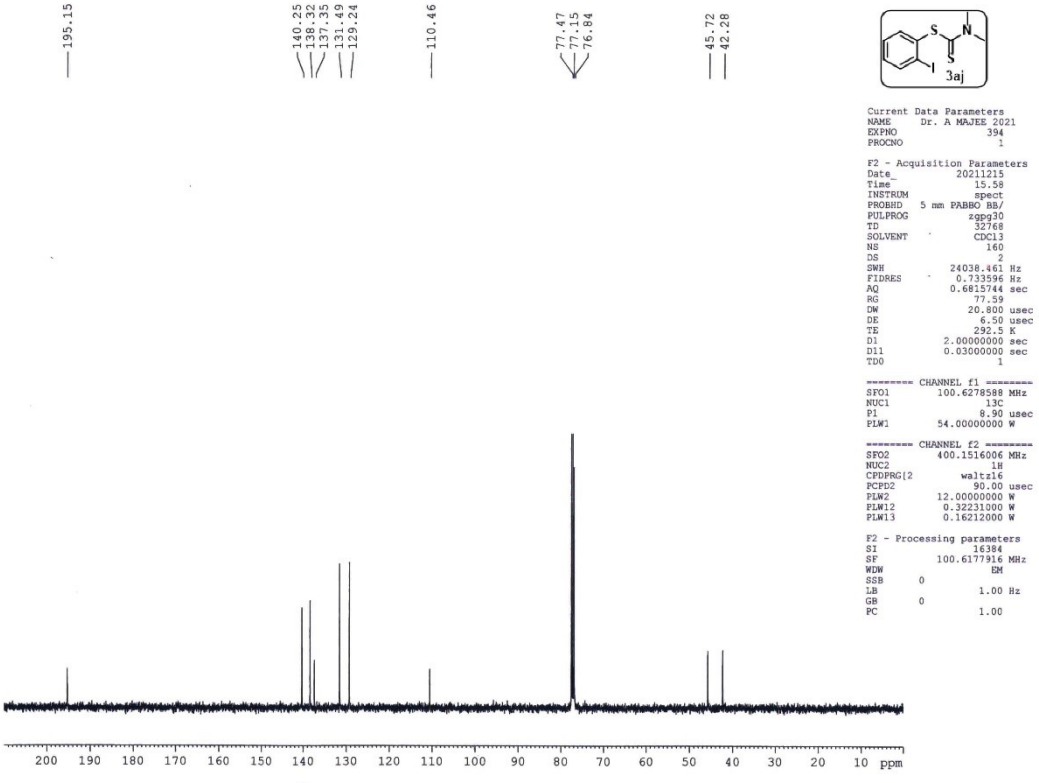
======== CHANNEL f2 ========
SFO2        400.1516006 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        12.00000000 W
PLW12        0.32231000 W
PLW13        0.16212000 W

F2 - Processing parameters
SI                16384
SF          100.6177873 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

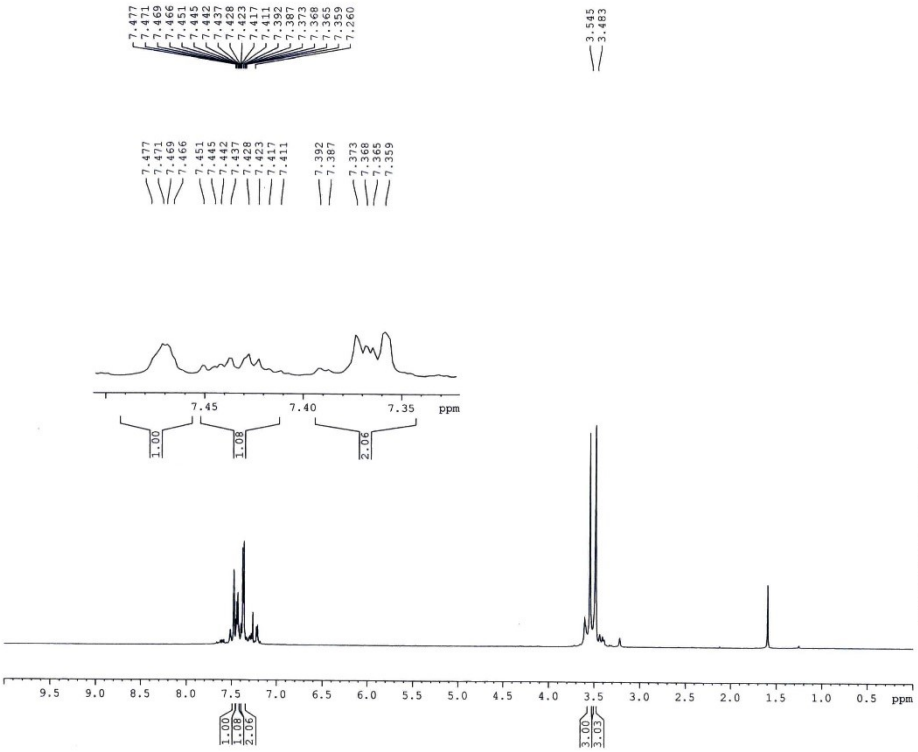
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



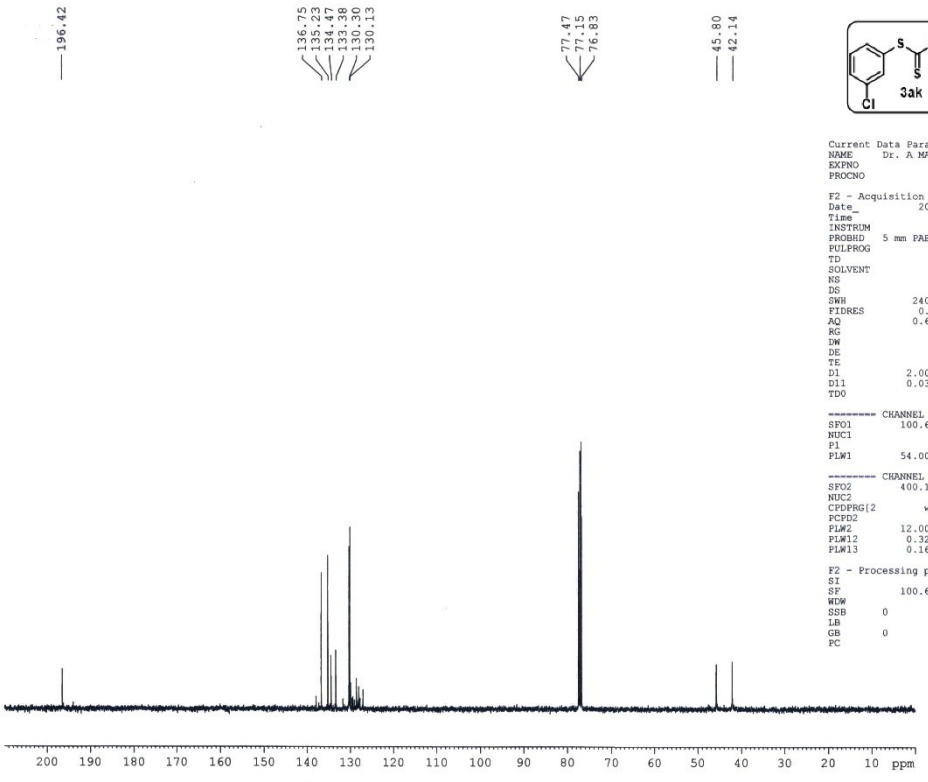

```
Current Data Parameters
NAME        Dr. A MAJEE 2022
EXPNO                  420
PROCNO                   1

F2 - Acquisition Parameters
Date_               20220927
Time                   15.32
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                        8
DS                        1
SWH                8223.685 Hz
FIDRES            0.250967 Hz
AQ                1.9922944 sec
RG                     62.69
DW                  60.800 usec
DE                   6.50 usec
TE                   297.1 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            400.1524711 MHz
NUC1                      1H
P1                     14.75 usec
PLW1           12.00000000 W

F2 - Processing parameters
SI                    16384
SF              400.1500087 MHz
WDW                      EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    2.00
```

^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$




```
Current Data Parameters
NAME        Dr. A MAJEE 2022
EXPNO                  421
PROCNO                   1

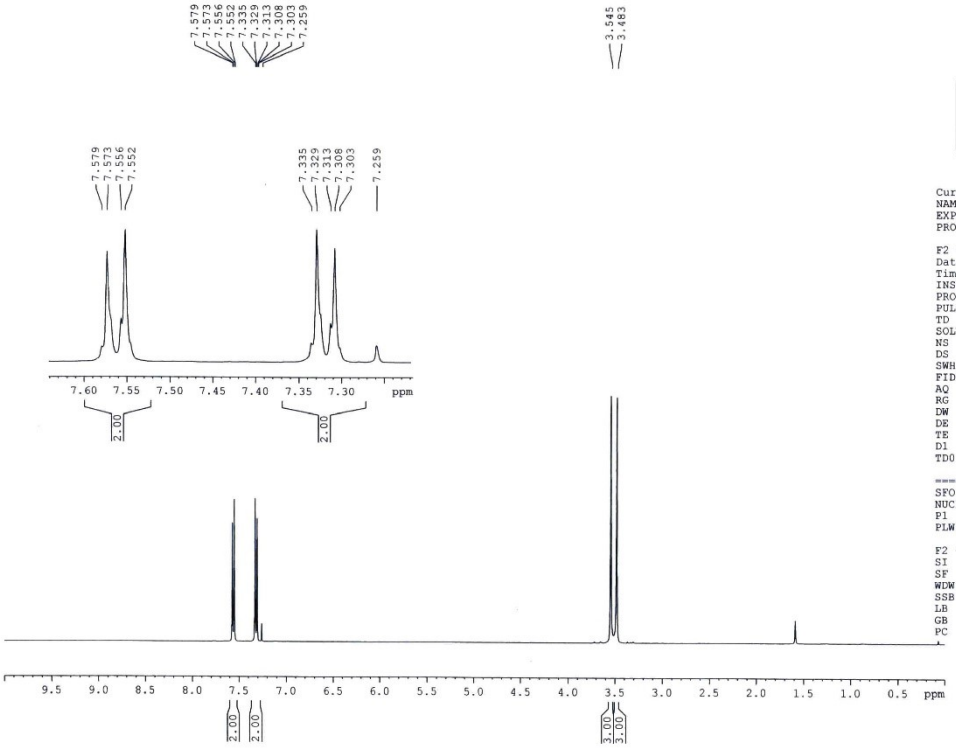
F2 - Acquisition Parameters
Date_               20220927
Time                   15.46
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      240
DS                        2
SWH               24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                     62.69
DW                  20.800 usec
DE                   6.50 usec
TE                   297.6 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            100.6278588 MHz
NUC1                      13C
P1                     8.90 usec
PLW1           54.00000000 W

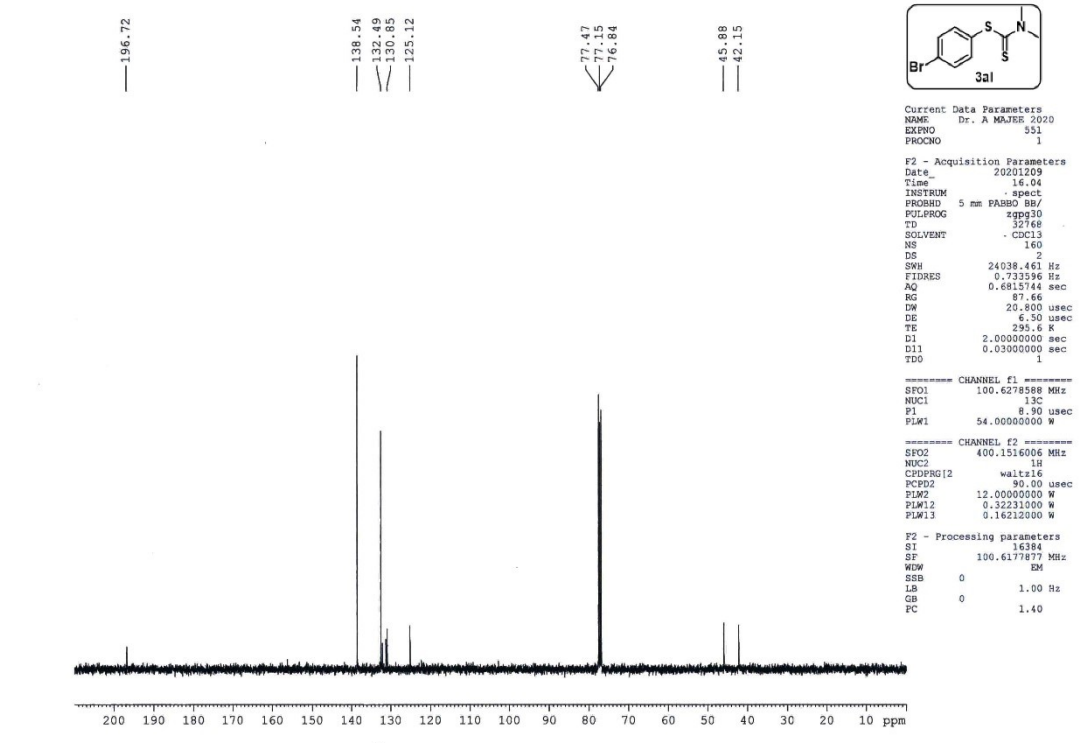
======== CHANNEL f2 ========
SFO2            400.1516006 MHz
NUC2                      1H
CPDPRG[2            waltz16
PCPD2                 90.00 usec
PLW2           12.00000000 W
PLW12           0.32231000 W
PLW13           0.16212000 W

F2 - Processing parameters
SI                    16384
SF              100.6177917 MHz
WDW                      EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.00
```

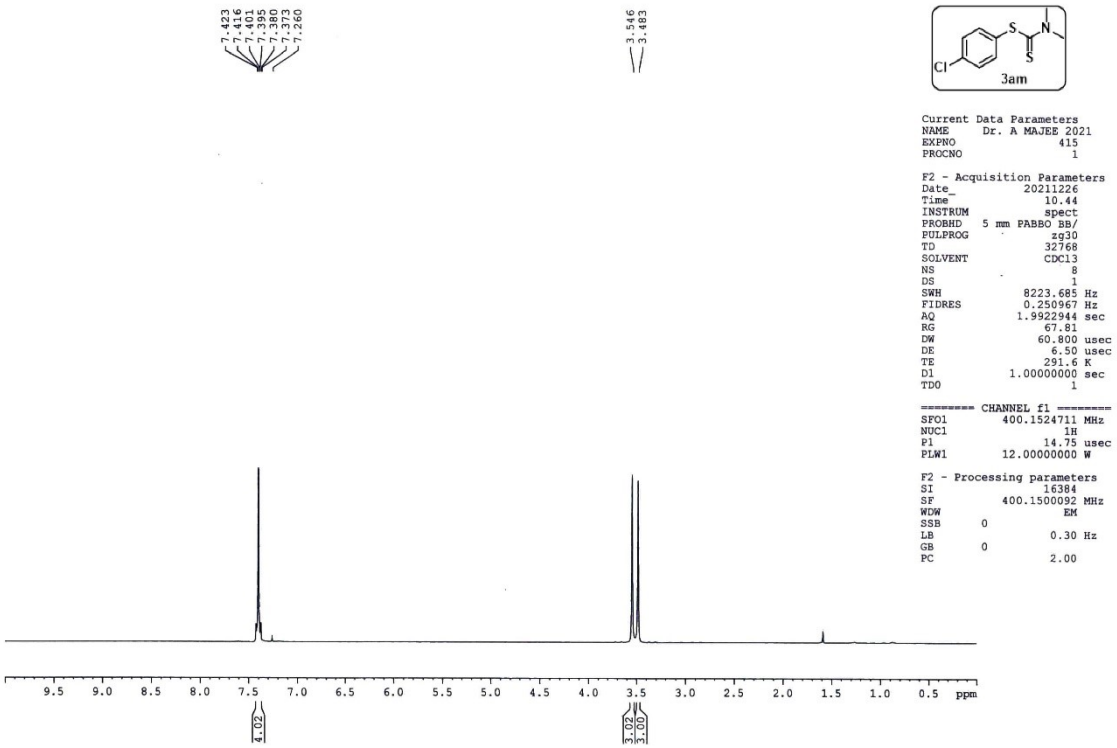
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



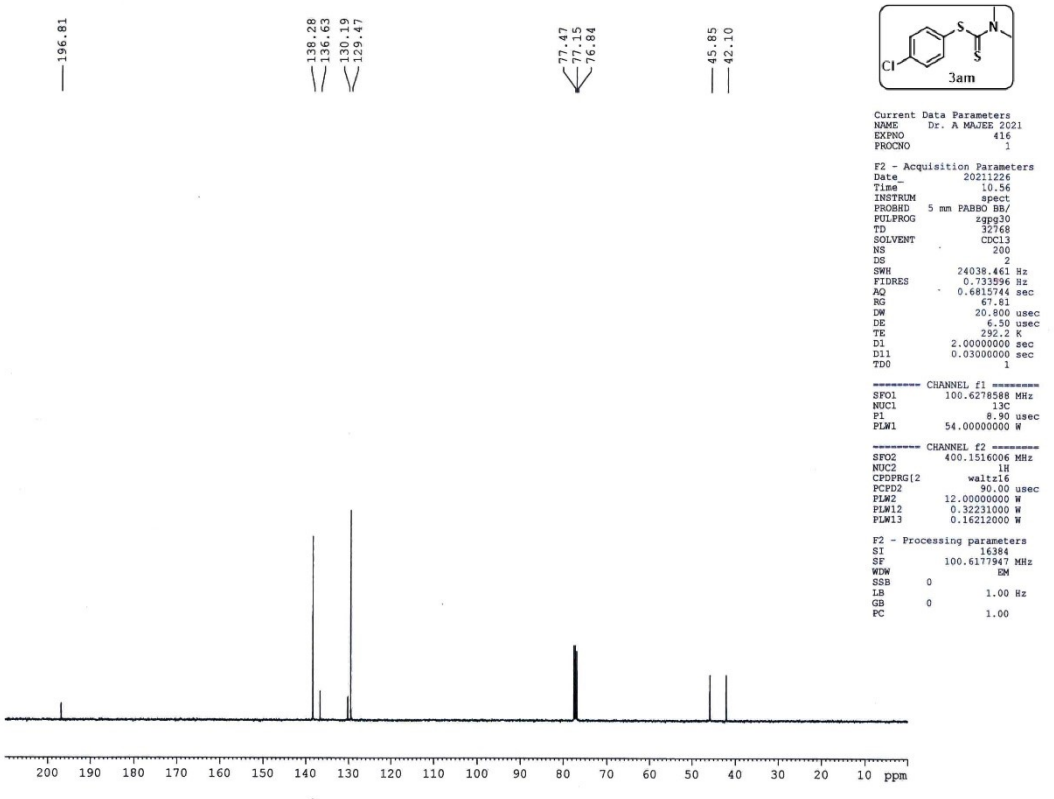
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



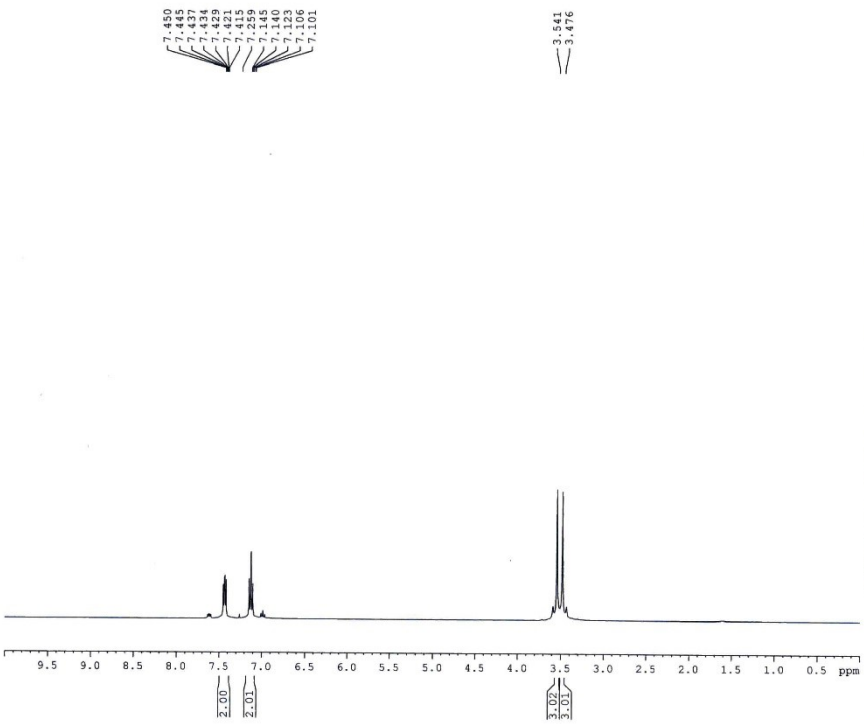
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



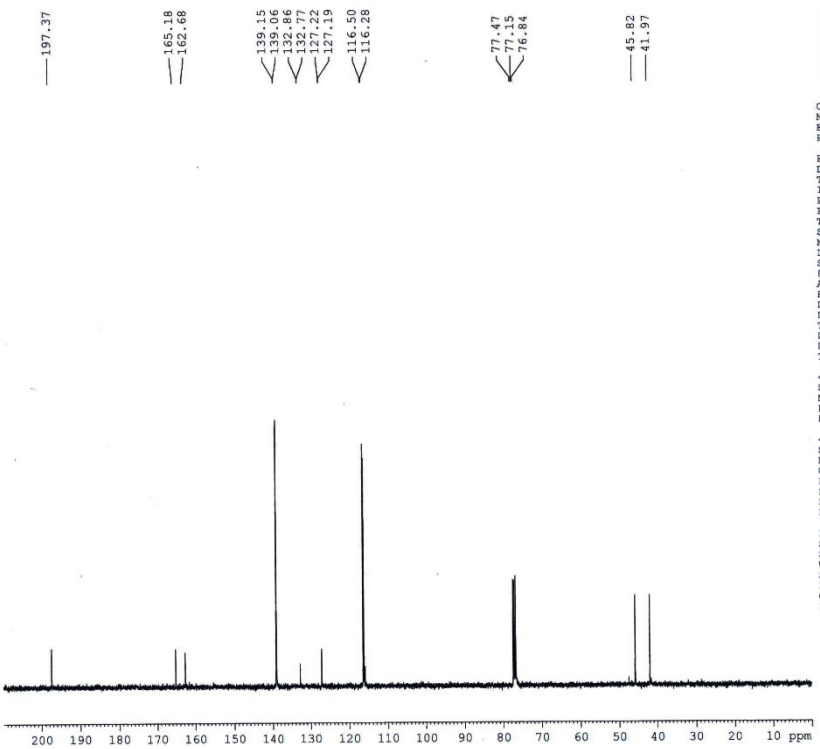
Current Data Parameters
NAME Dr. A MAJEE 2021
EXPNO 68
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210202
Time 16.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9922944 sec
RG 37.83
DW 60.800 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1524711 MHz
NUC1 1H
P1 14.75 usec
PLW1 12.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1500092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



Current Data Parameters
NAME Dr. A MAJEE 2021
EXPNO 69
PROCNO 1

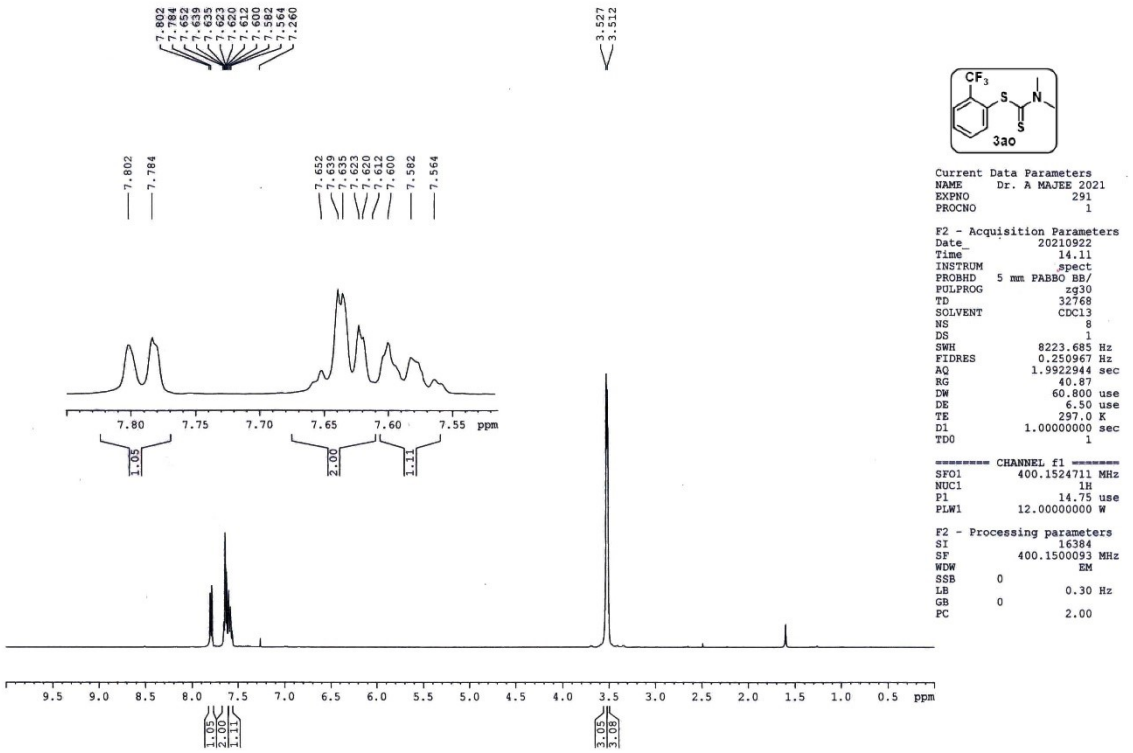
F2 - Acquisition Parameters
Date_ 20210202
Time 16.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 188
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 37.83
DW 20.800 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

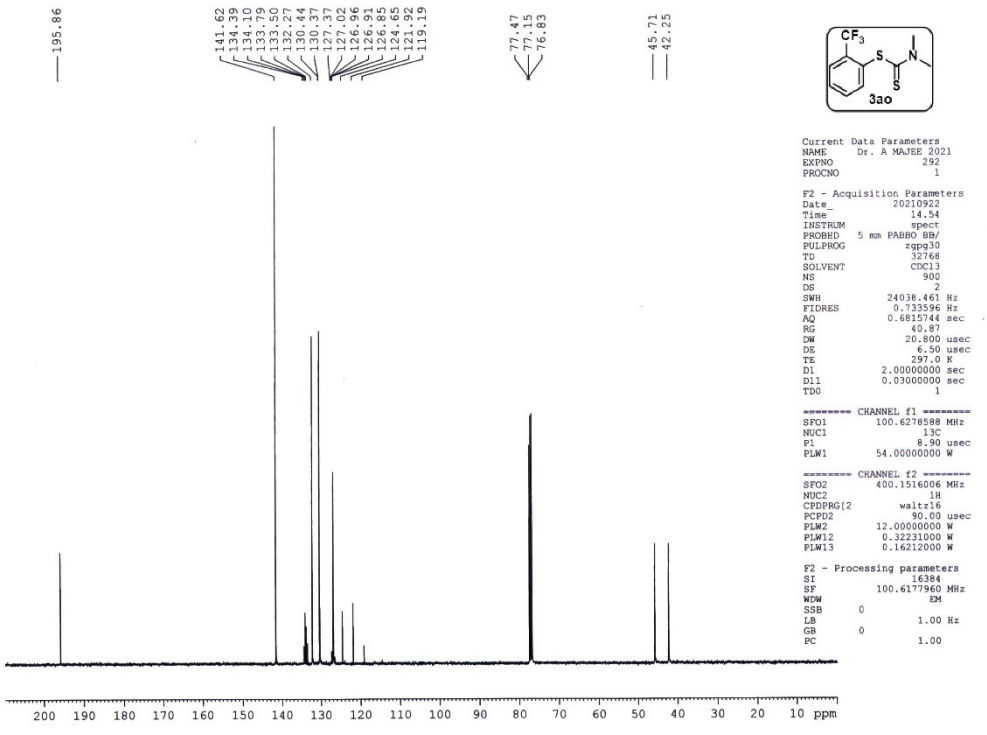
======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177995 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

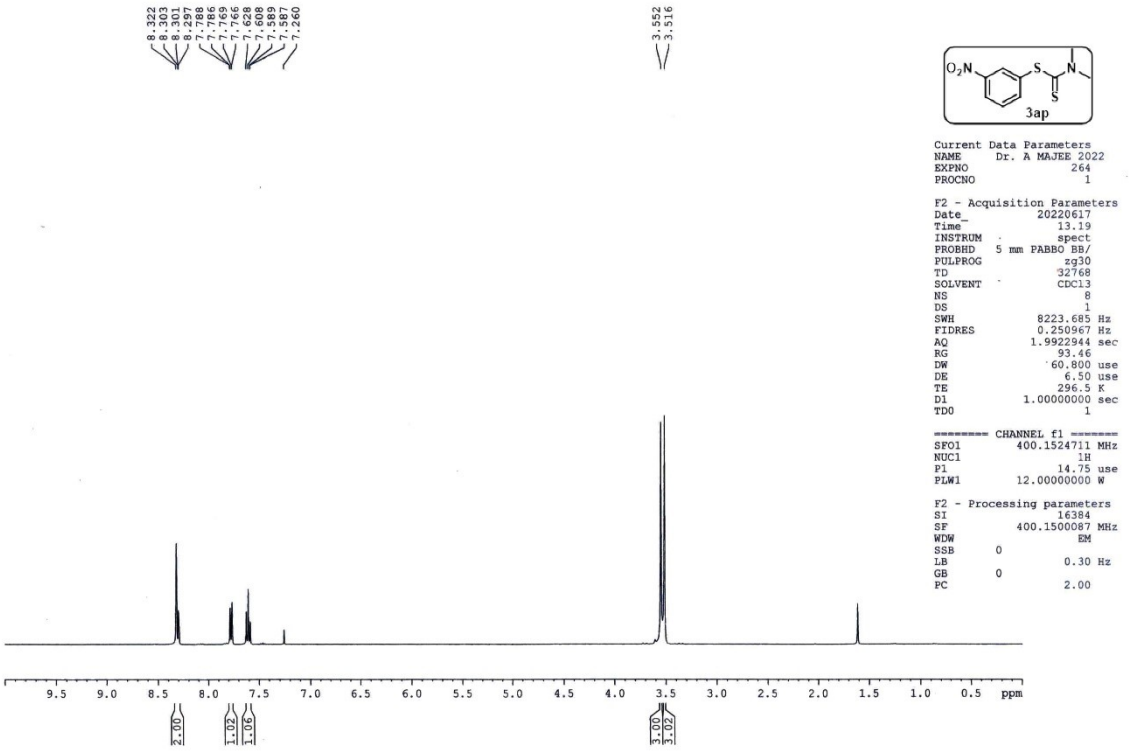
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



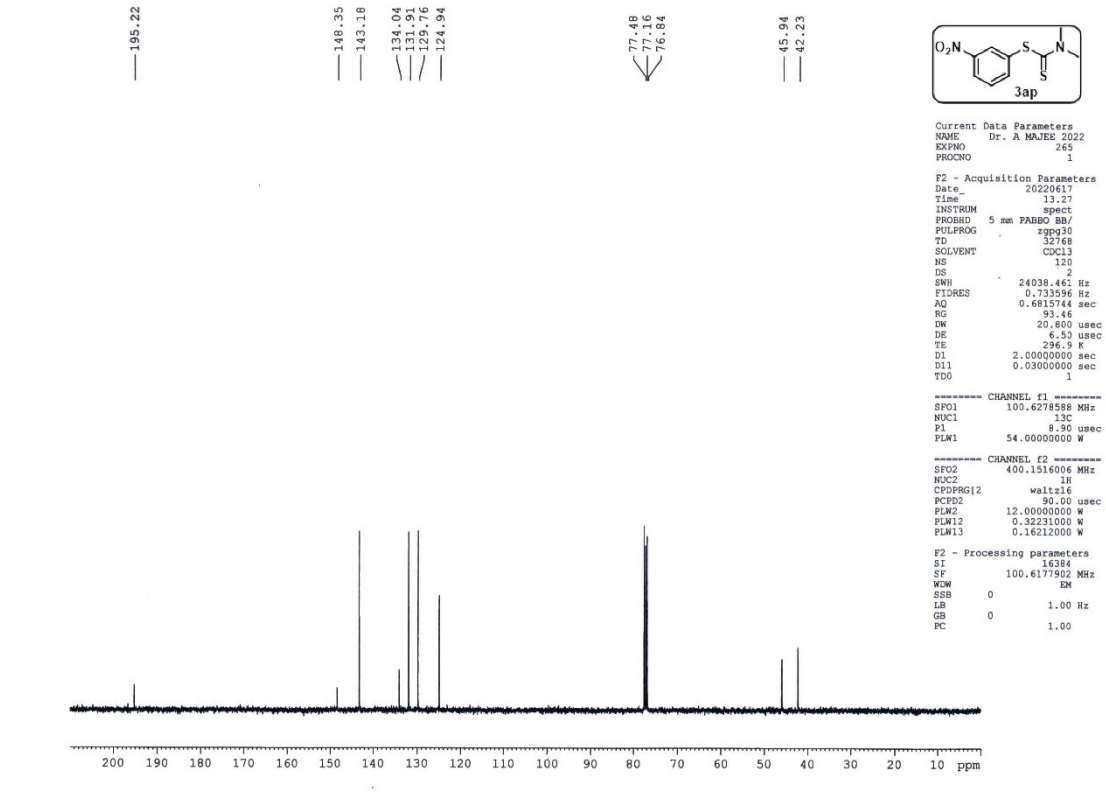
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



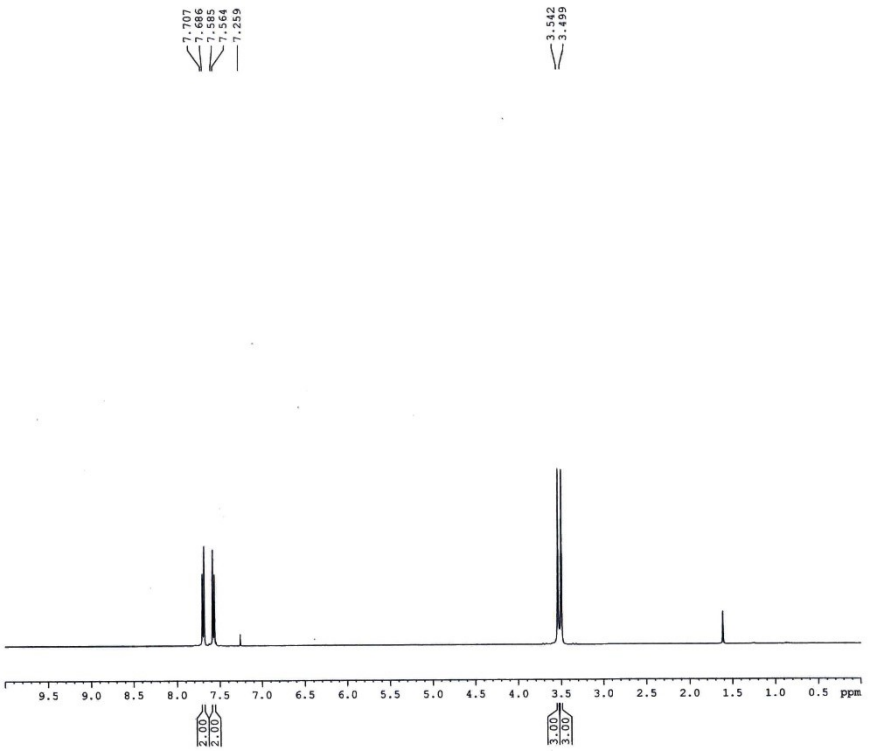
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$

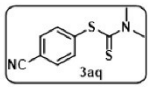


$^{13}C\{^{1}H\}$ NMR: 100 MHz; Solvent: $CDCl_3$



^{1}H NMR: 400 MHz; Solvent: $CDCl_3$





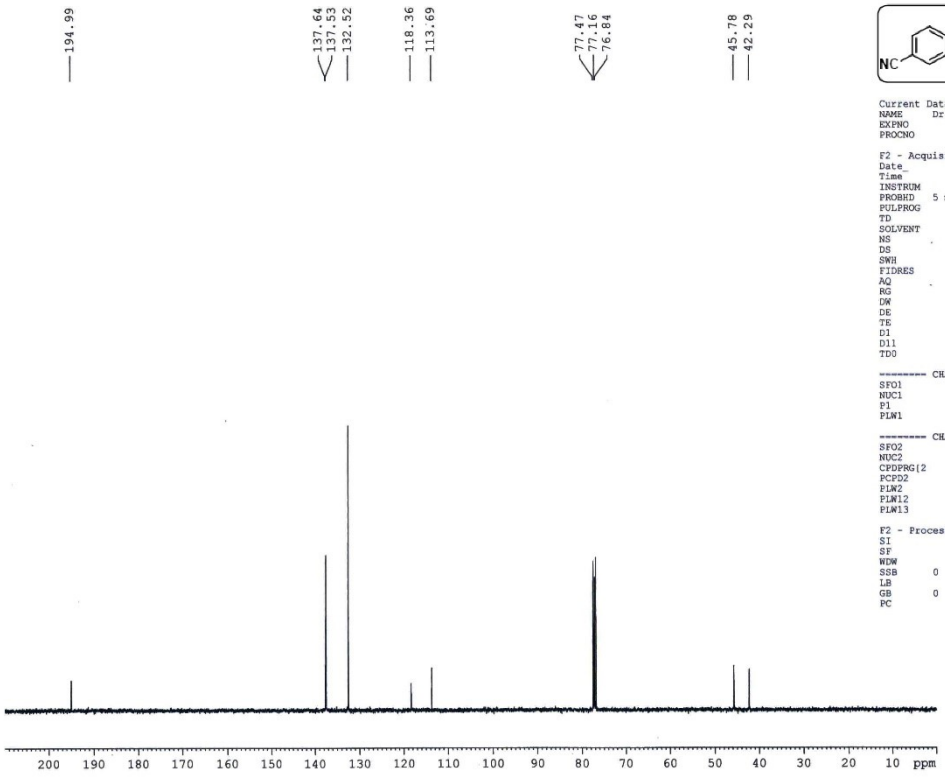

```
Current Data Parameters
NAME       Dr. A MAJEE 2022
EXPNO                 278
PROCNO                  1

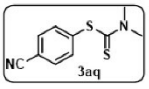
F2 - Acquisition Parameters
Date_           20220619
Time              14.40
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   8
DS                   1
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9922944 sec
RG               120.16
DW               60.800 usec
DE                6.50 usec
TE               296.5 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1524711 MHz
NUC1               1H
P1               14.75 usec
PLW1        12.00000000 W

F2 - Processing parameters
SI                16384
SF          400.1500092 MHz
WDW                EM
SSB       0
LB                0.30 Hz
GB        0
PC                2.00
```

^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$






```
Current Data Parameters
NAME       Dr. A MAJEE 2022
EXPNO                 279
PROCNO                  1

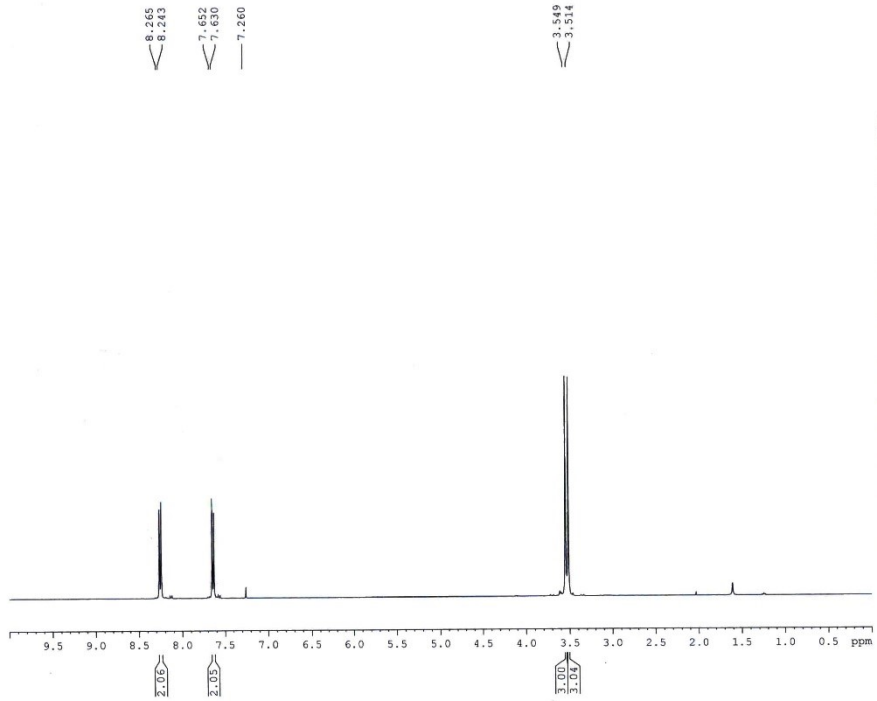
F2 - Acquisition Parameters
Date_           20220619
Time              14.50
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               32768
SOLVENT          CDCl3
NS                 180
DS                   2
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG               120.16
DW               20.800 usec
DE                6.50 usec
TE               296.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

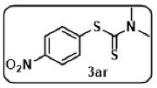
======== CHANNEL f1 ========
SFO1        100.6278588 MHz
NUC1               13C
P1                8.90 usec
PLW1        54.00000000 W

======== CHANNEL f2 ========
SFO2        400.1516006 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        12.00000000 W
PLW12        0.32231000 W
PLW13        0.16212000 W

F2 - Processing parameters
SI                16384
SF          100.6177905 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.00
```

^{1}H NMR: 400 MHz; Solvent: $CDCl_3$






```
Current Data Parameters
NAME      Dr. A MAJEE 2021
EXPNO                 48
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210120
Time              15.57
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                    8
DS                    1
SWH            8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9922944 sec
RG                67.81
DW               60.800 usec
DE                6.50 usec
TE               294.7 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1524711 MHz
NUC1                 1H
P1                14.75 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                16384
SF           400.1500092 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                2.00
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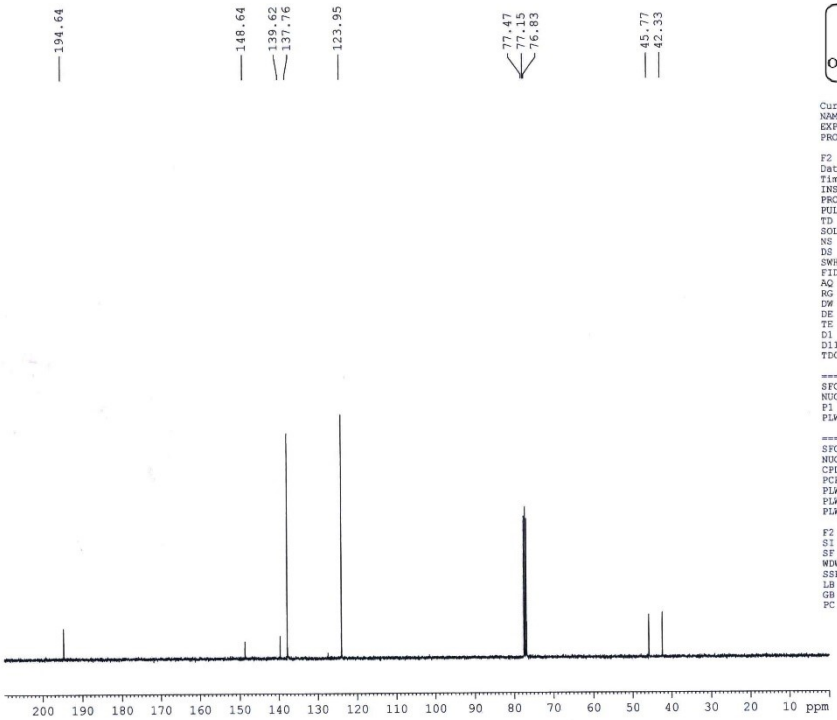
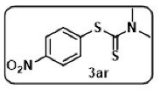
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



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Current Data Parameters
NAME      Dr. A MAJEE 2021
EXPNO                 49
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210120
Time              16.07
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               32768
SOLVENT          CDCl3
NS                  160
DS                    2
SWH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG                67.81
DW               20.800 usec
DE                6.50 usec
TE               295.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                 13C
P1                8.90 usec
PLW1         54.00000000 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.32231000 W
PLW13         0.16212000 W

F2 - Processing parameters
SI                16384
SF           100.6177917 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
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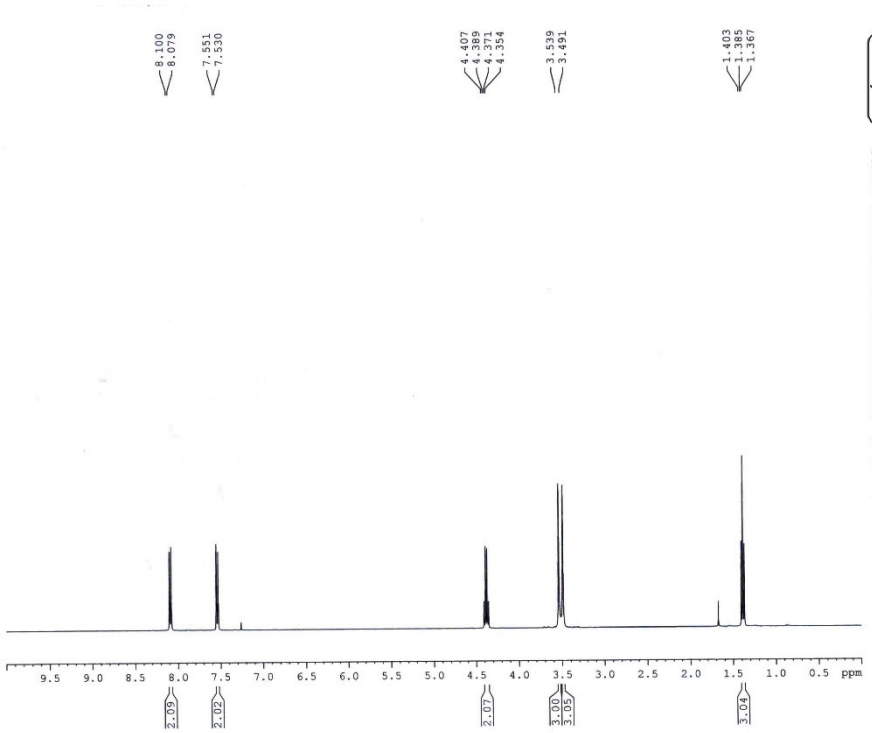
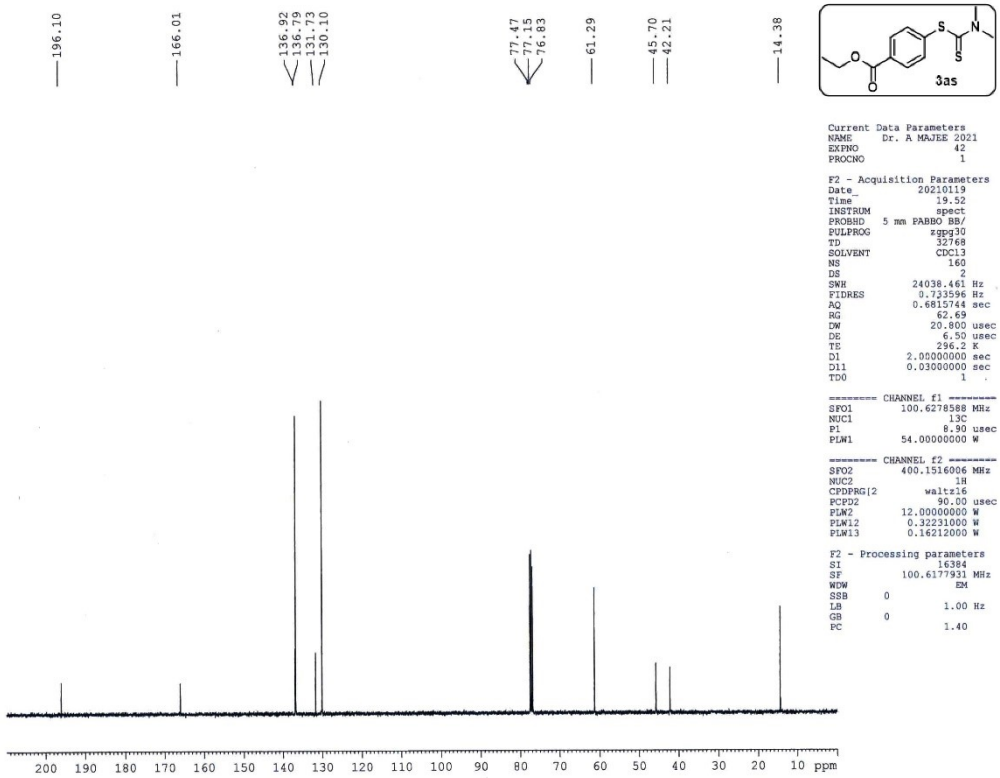
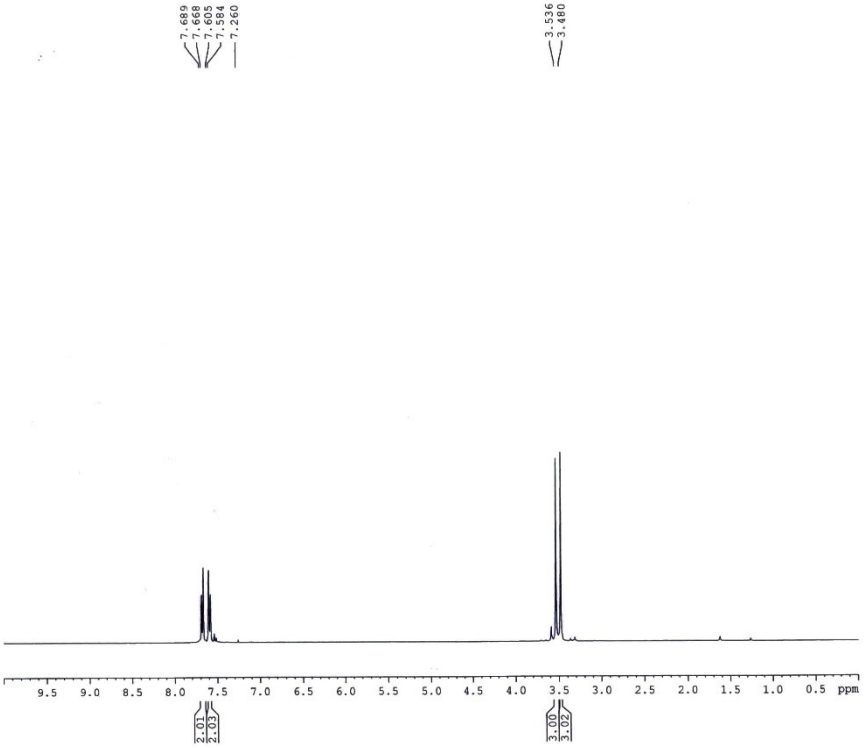
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$

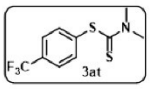


^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



^{1}H NMR: 400 MHz; Solvent: $CDCl_3$






```
Current Data Parameters
NAME      Dr. A MAJEE 2021
EXPNO              395
PROCNO               1

F2 - Acquisition Parameters
Date_          20211215
Time             16.05
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG          zg30
TD              32768
SOLVENT          CDCl3
NS                   8
DS                   1
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9922944 sec
RG               26.53
DW              60.800 usec
DE               6.50 usec
TE              292.1 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1524711 MHz
NUC1               1H
P1              14.75 usec
PLW1       12.00000000 W

F2 - Processing parameters
SI                16384
SF          400.1500092 MHz
WDW                EM
SSB       0
LB              0.30 Hz
GB        0
PC              2.00
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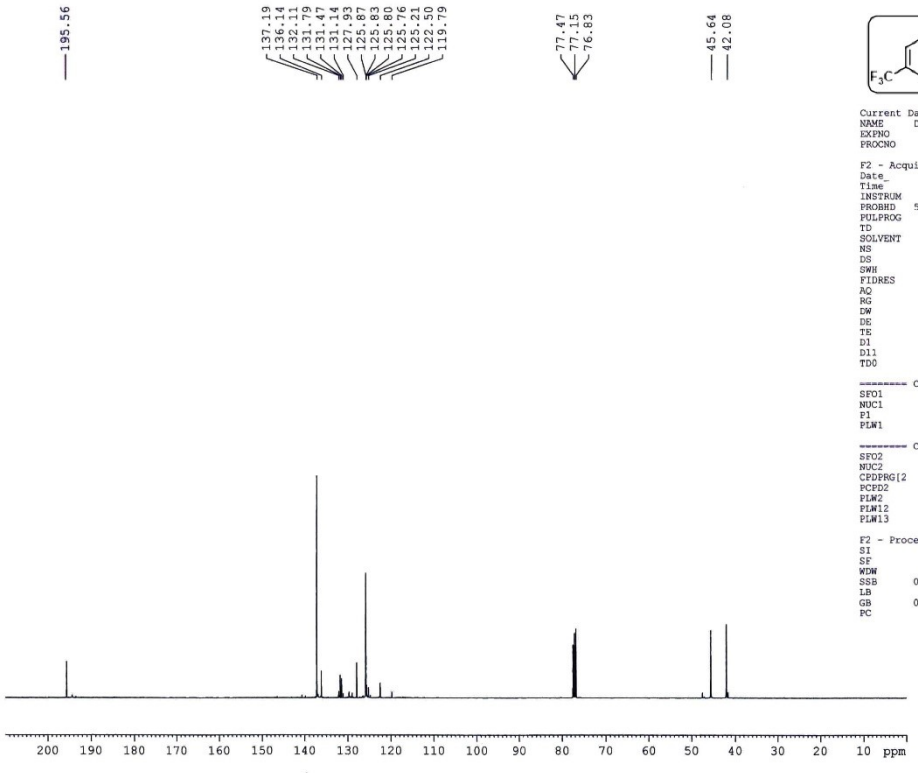
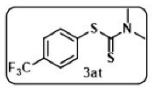
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



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Current Data Parameters
NAME      Dr. A MAJEE 2021
EXPNO              396
PROCNO               1

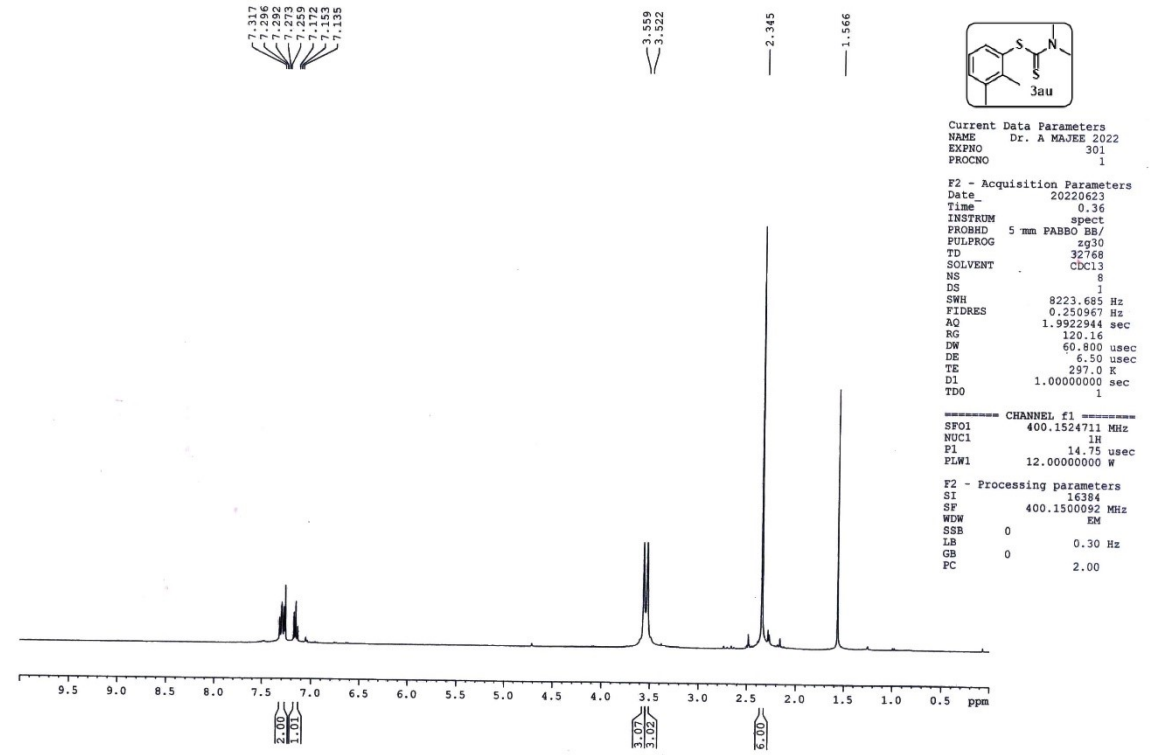
F2 - Acquisition Parameters
Date_          20211215
Time             16.53
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG        zgpg30
TD              32768
SOLVENT          CDCl3
NS                1024
DS                   2
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG               26.53
DW              20.800 usec
DE               6.50 usec
TE              292.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6278588 MHz
NUC1               13C
P1               8.90 usec
PLW1       54.00000000 W

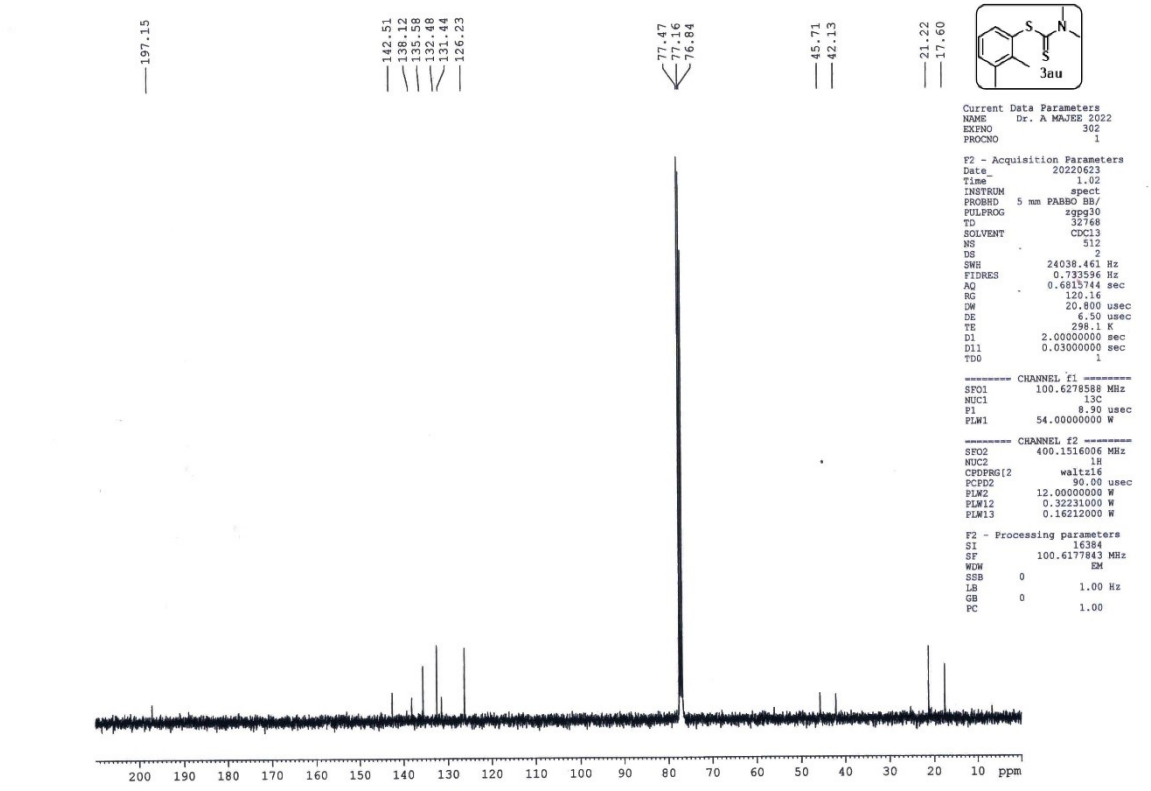
======== CHANNEL f2 ========
SFO2        400.1516006 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2       12.00000000 W
PLW12       0.32231000 W
PLW13       0.16212000 W

F2 - Processing parameters
SI                16384
SF          100.6178034 MHz
WDW                EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.00
```

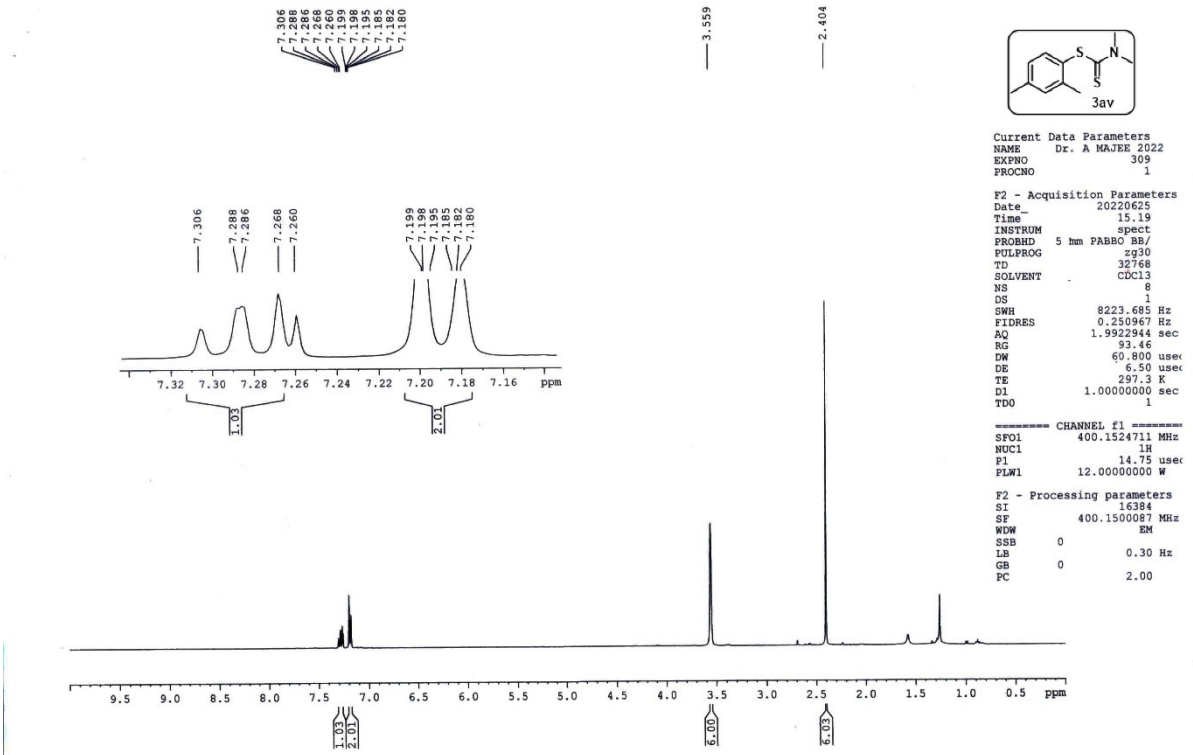
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



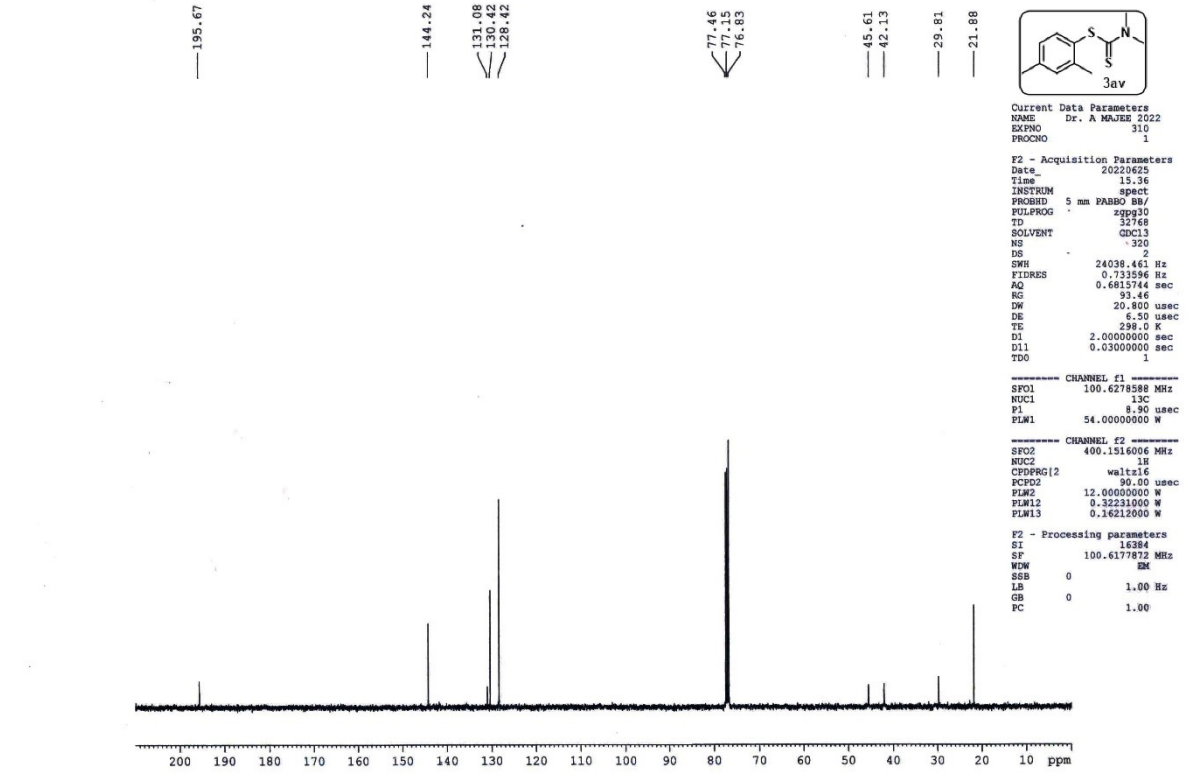
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



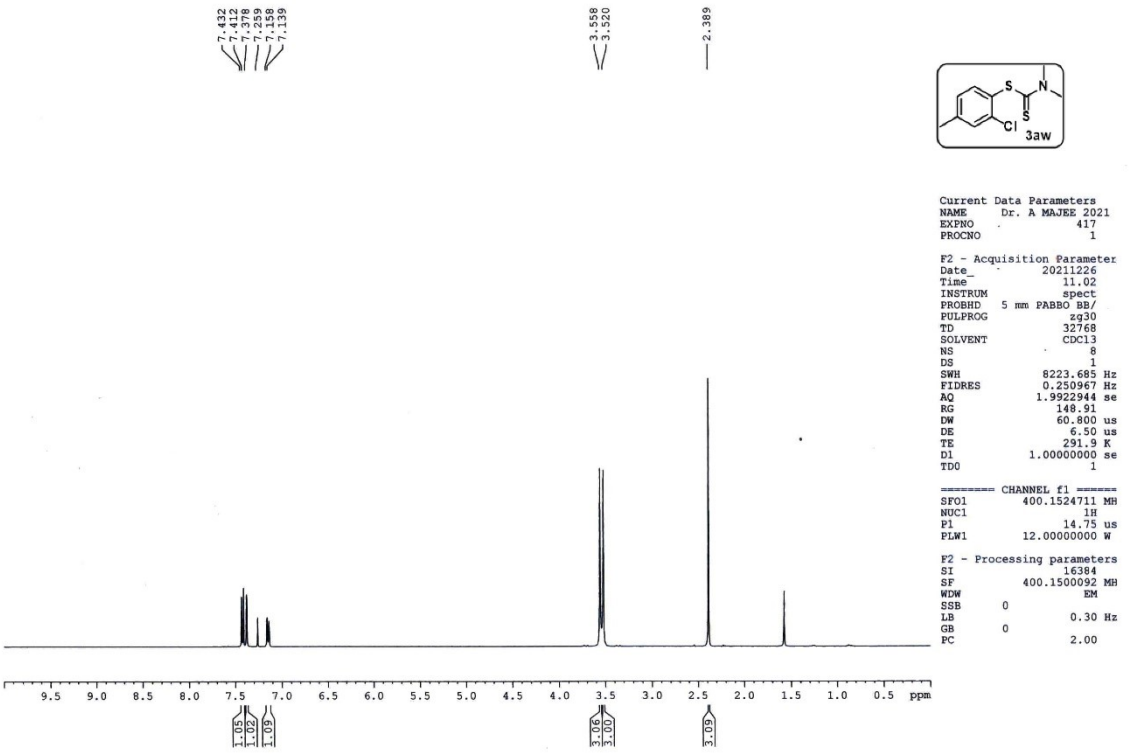
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



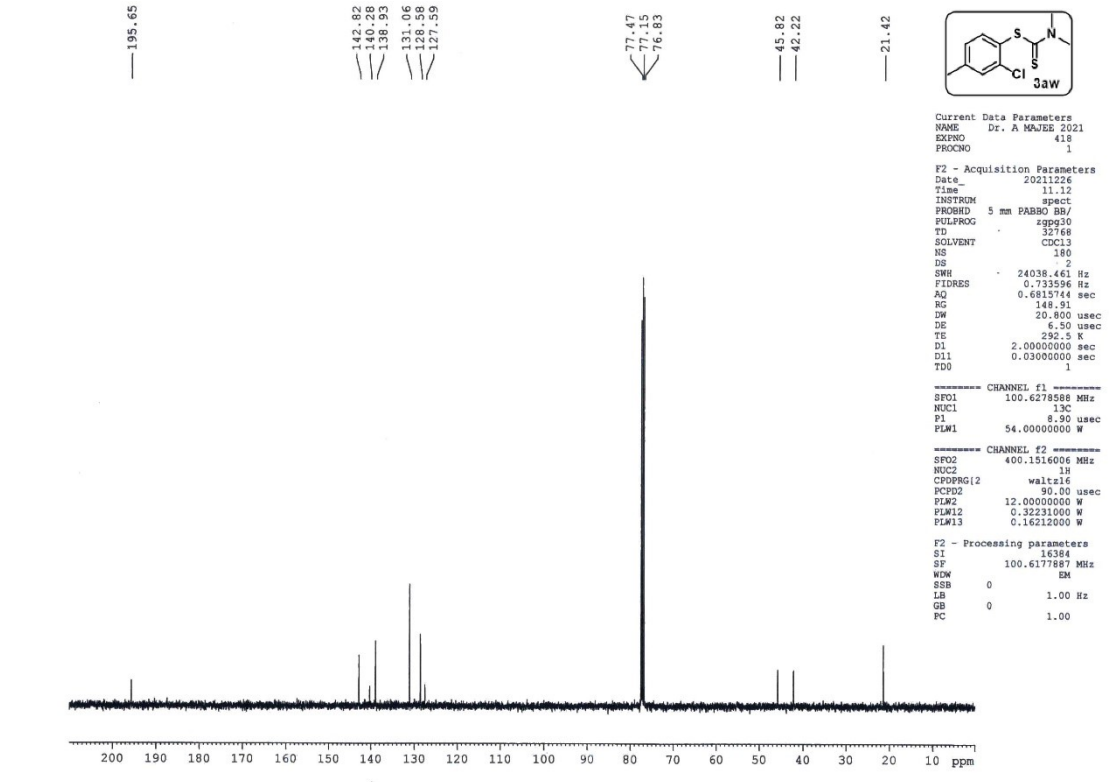
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



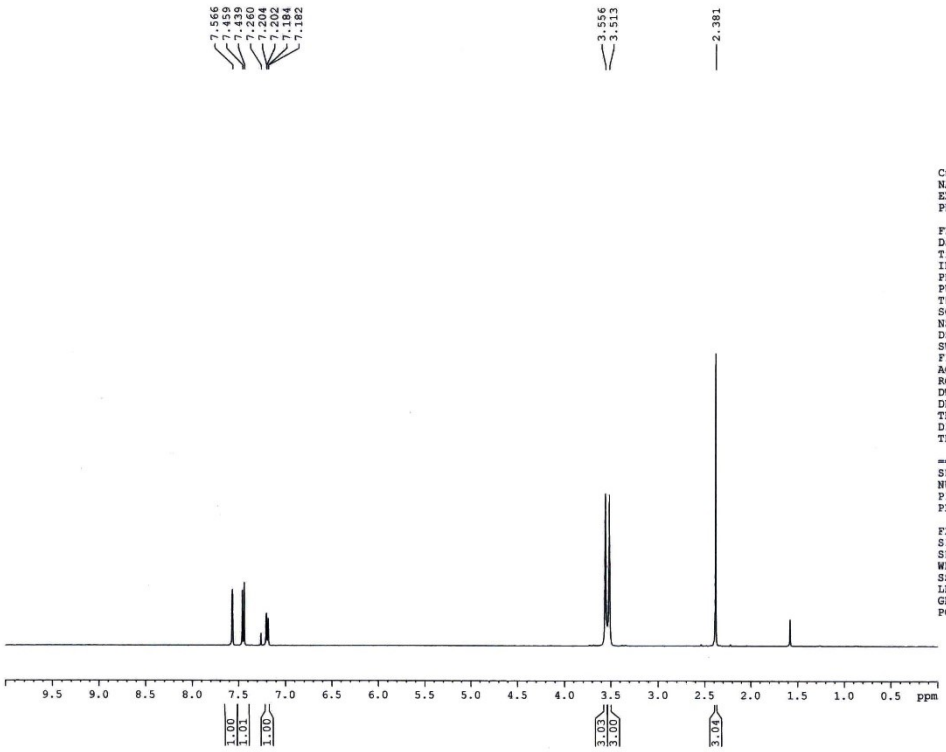
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



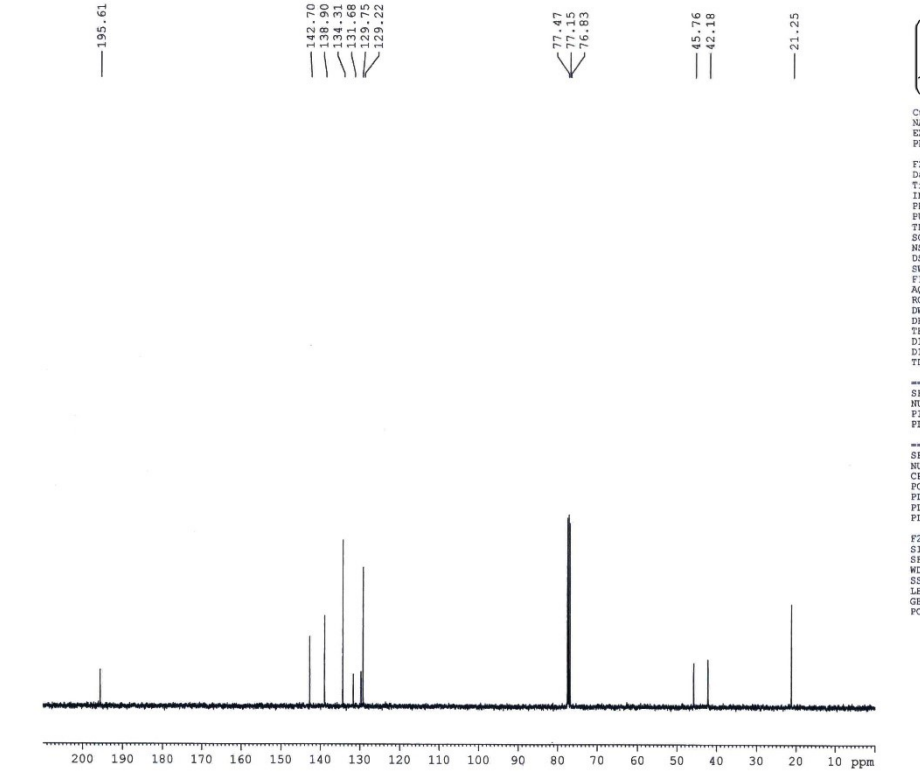
$^{13}C\{^{1}H\}$ NMR: 100 MHz; Solvent: $CDCl_3$



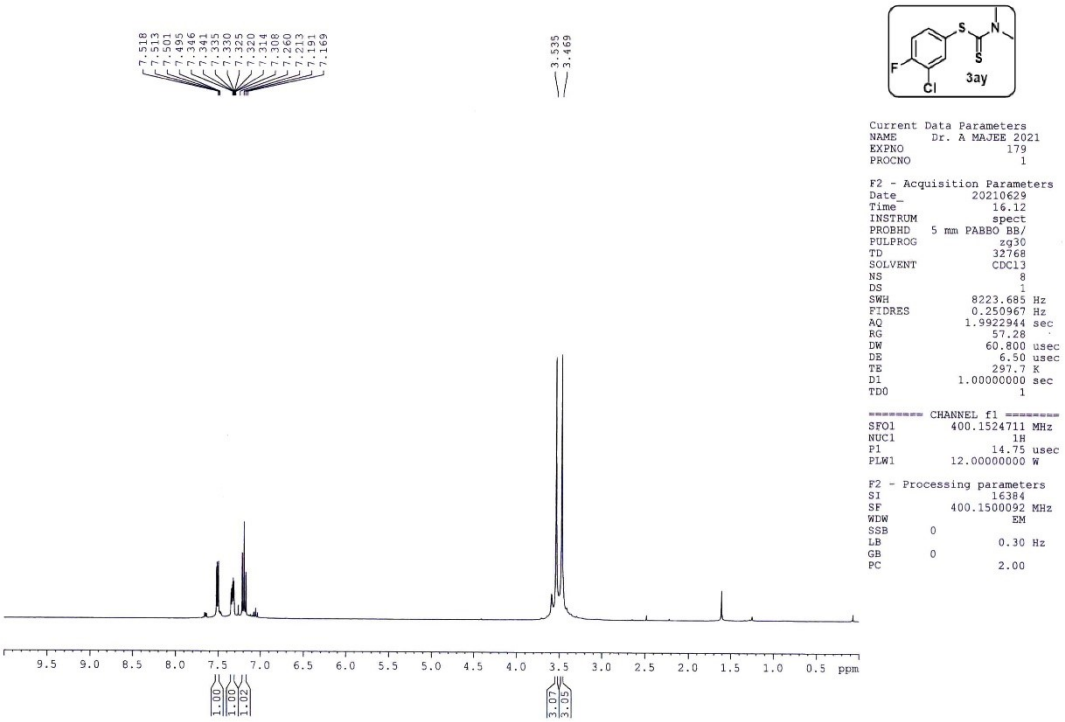
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



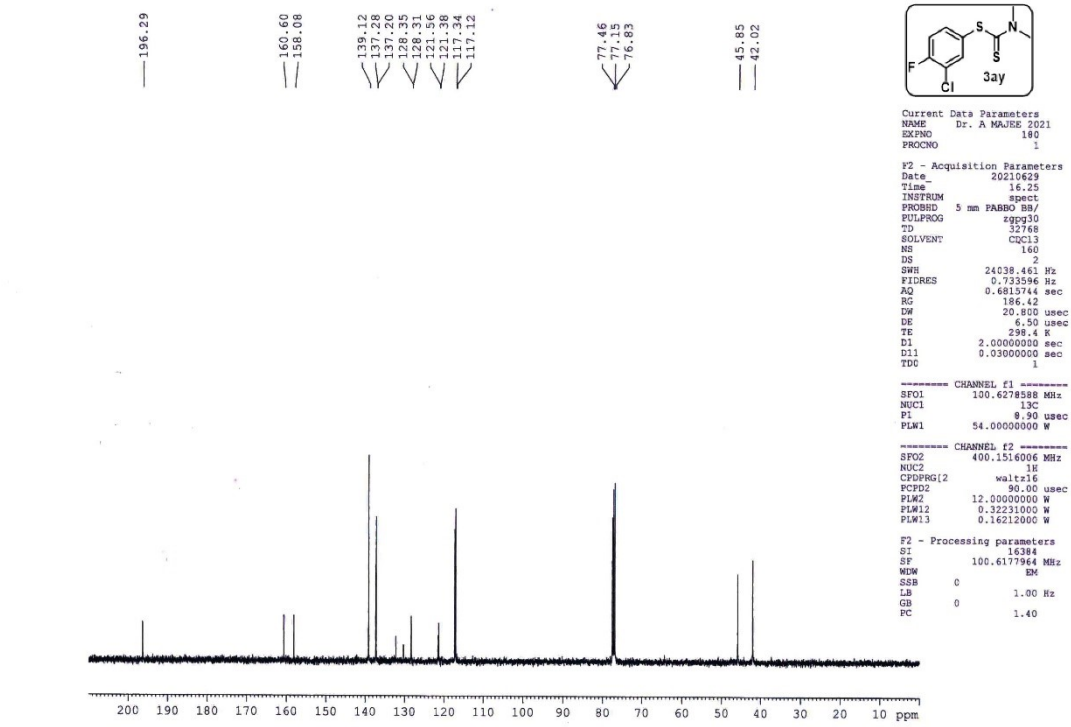
$^{13}C\{^{1}H\}$ NMR: 100 MHz; Solvent: $CDCl_3$



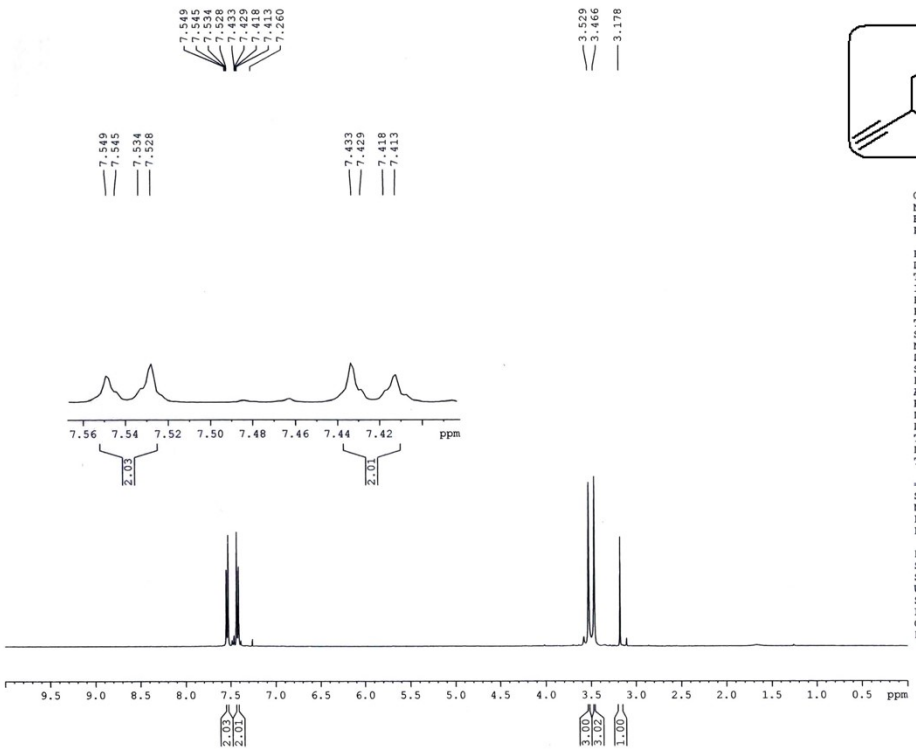
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$

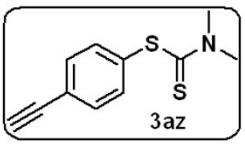


^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



^{1}H NMR: 400MHz; Solvent: $CDCl_3$





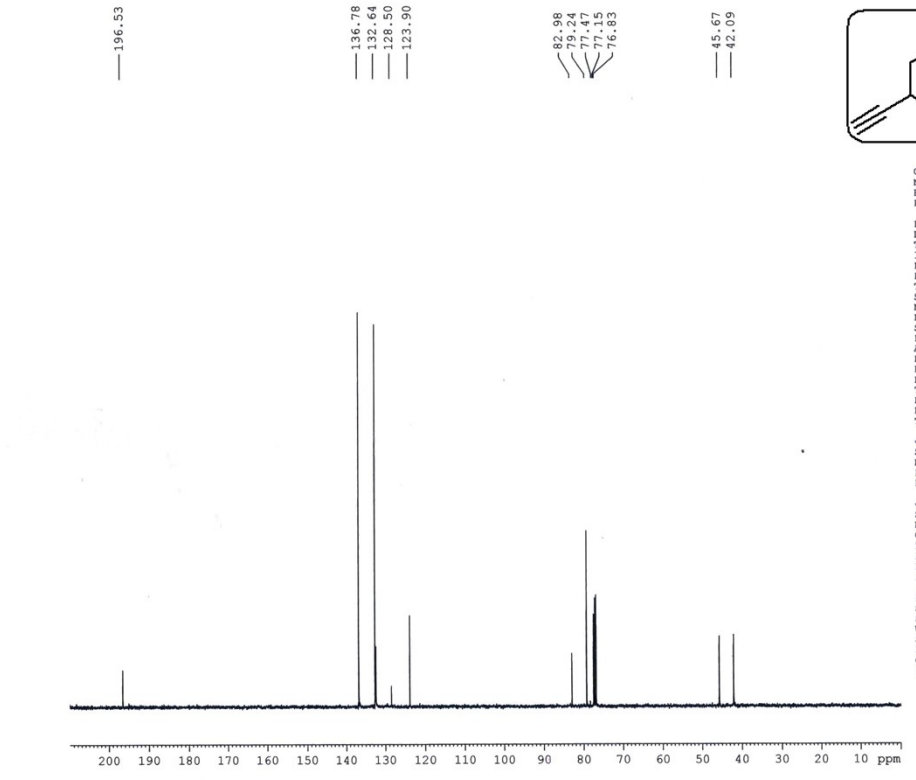

```
Current Data Parameters
NAME      Dr. A MAJEE 2023
EXPNO                672
PROCNO                 1

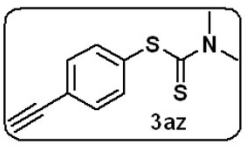
F2 - Acquisition Parameters
Date_           20230926
Time              16.23
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    1
SWH            8223.685 Hz
FIDRES         0.250967 Hz
AQ             1.9922944 sec
RG                47.25
DW               60.800 usec
DE                6.50 usec
TE               296.2 K
D1          1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1       400.1524711 MHz
NUC1               1H
P1               14.75 usec
PLW1       12.00000000 W

F2 - Processing parameters
SI                16384
SF         400.1500097 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                2.00
```

^{13}C{^{1}H} NMR: 100MHz; Solvent: $CDCl_3$



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Current Data Parameters
NAME      Dr. A MAJEE 2023
EXPNO                673
PROCNO                 1

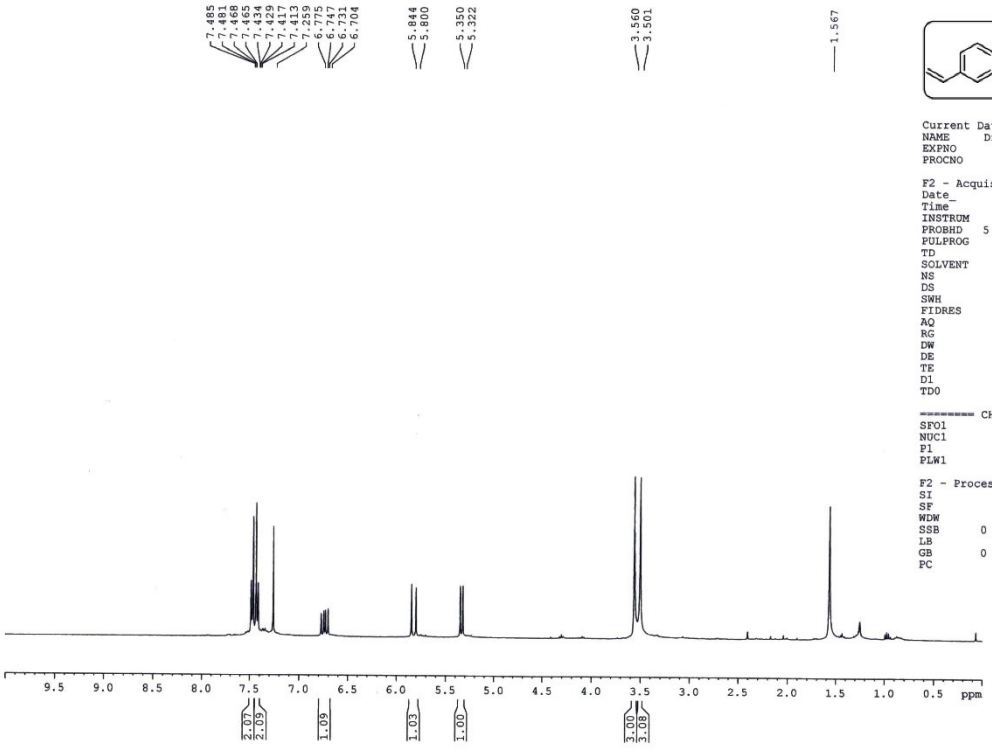
F2 - Acquisition Parameters
Date_           20230926
Time              16.36
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  160
DS                    2
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                47.25
DW               20.800 usec
DE                6.50 usec
TE               296.6 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1       100.6278588 MHz
NUC1               13C
P1                8.90 usec
PLW1       54.00000000 W

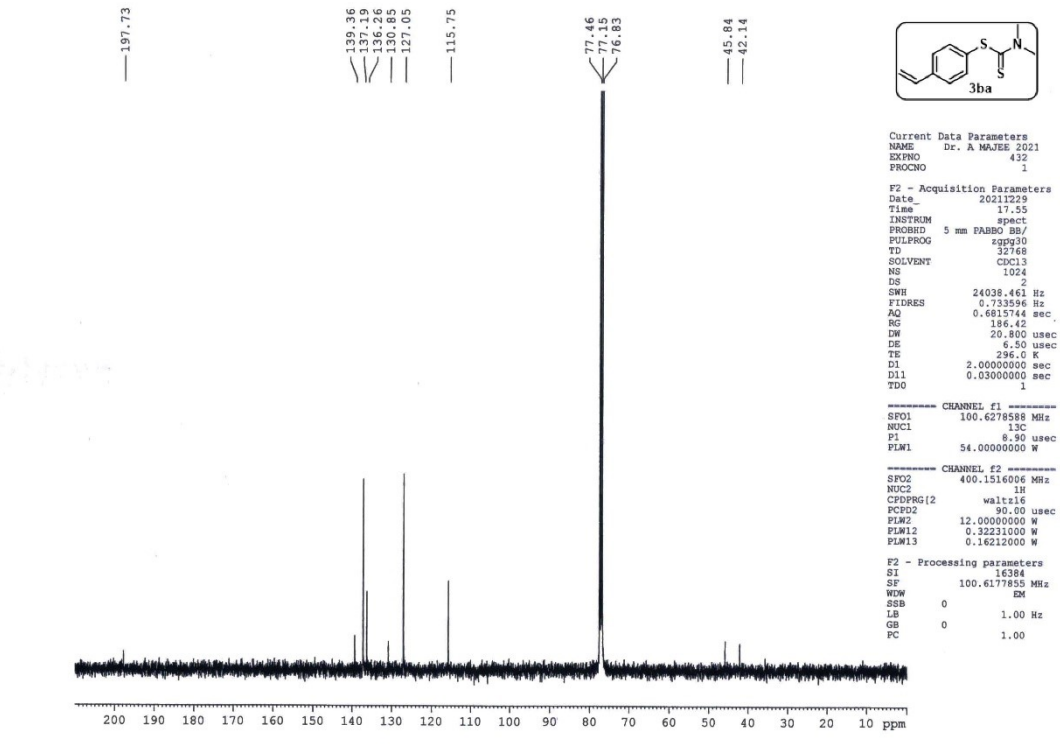
======== CHANNEL f2 ========
SFO2       400.1516006 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2       12.00000000 W
PLW12       0.32231000 W
PLW13       0.16212000 W

F2 - Processing parameters
SI                16384
SF         100.6178005 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.00
```

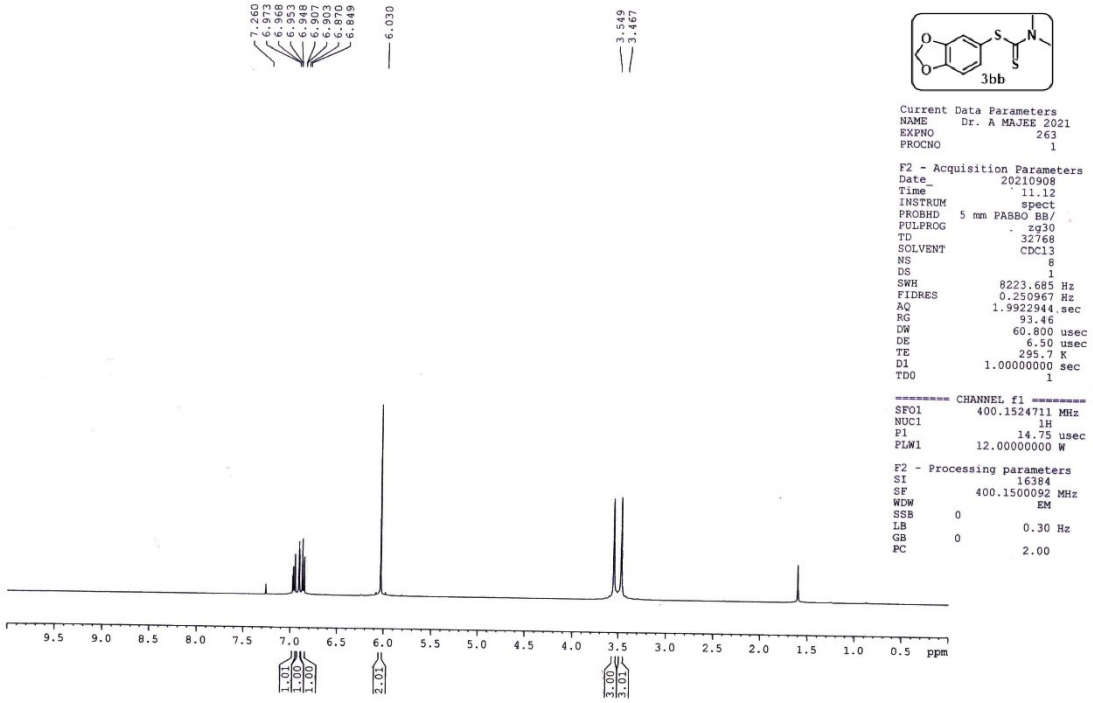
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



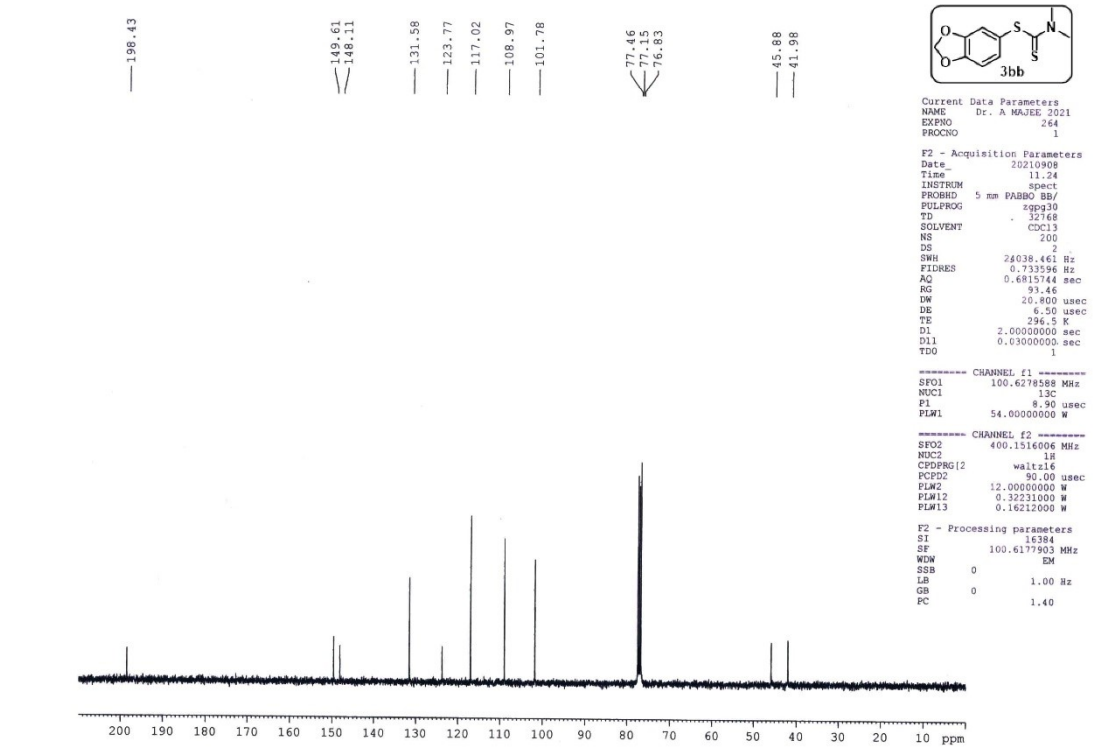
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



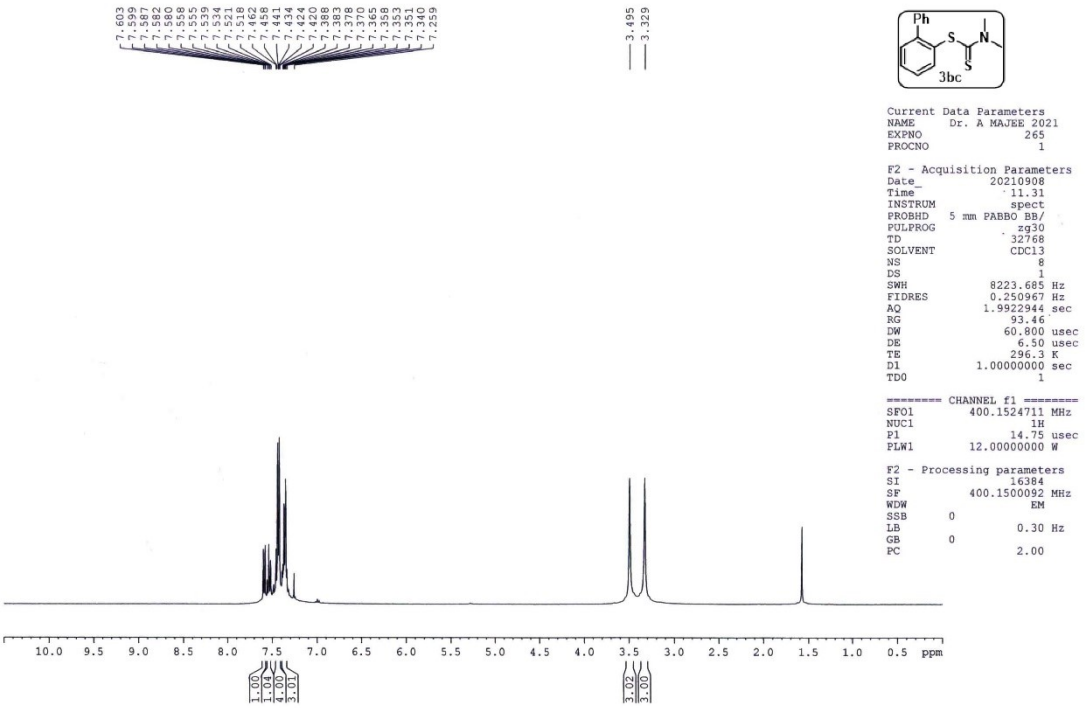
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



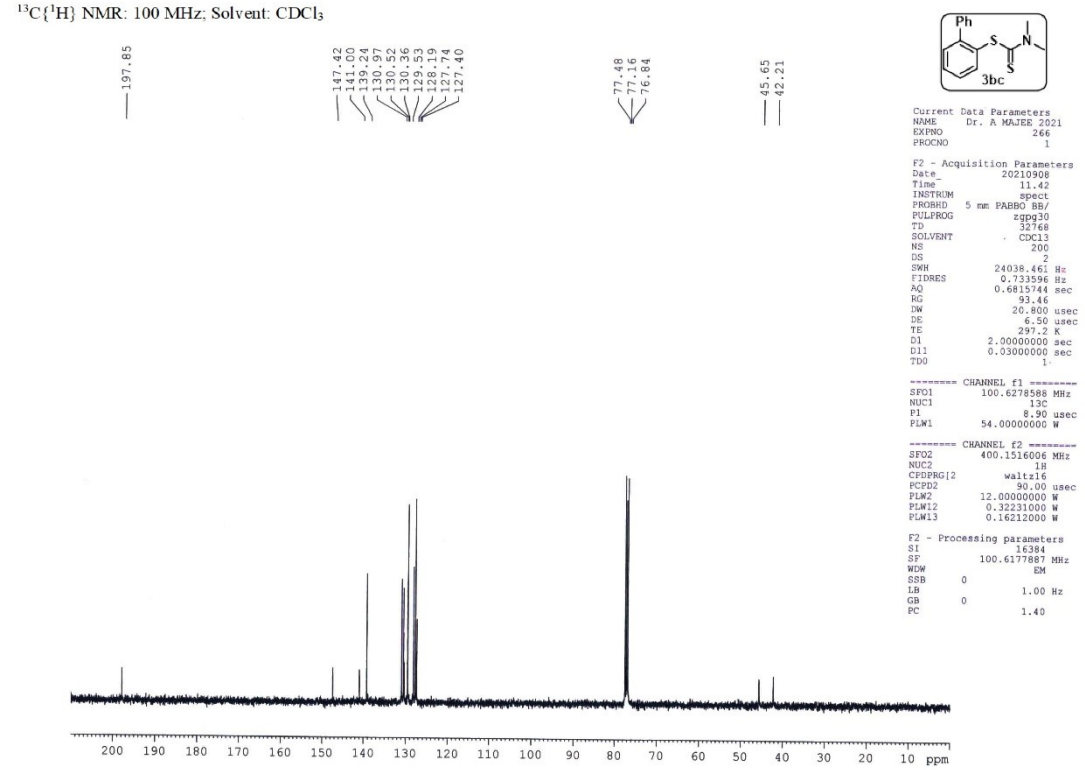
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



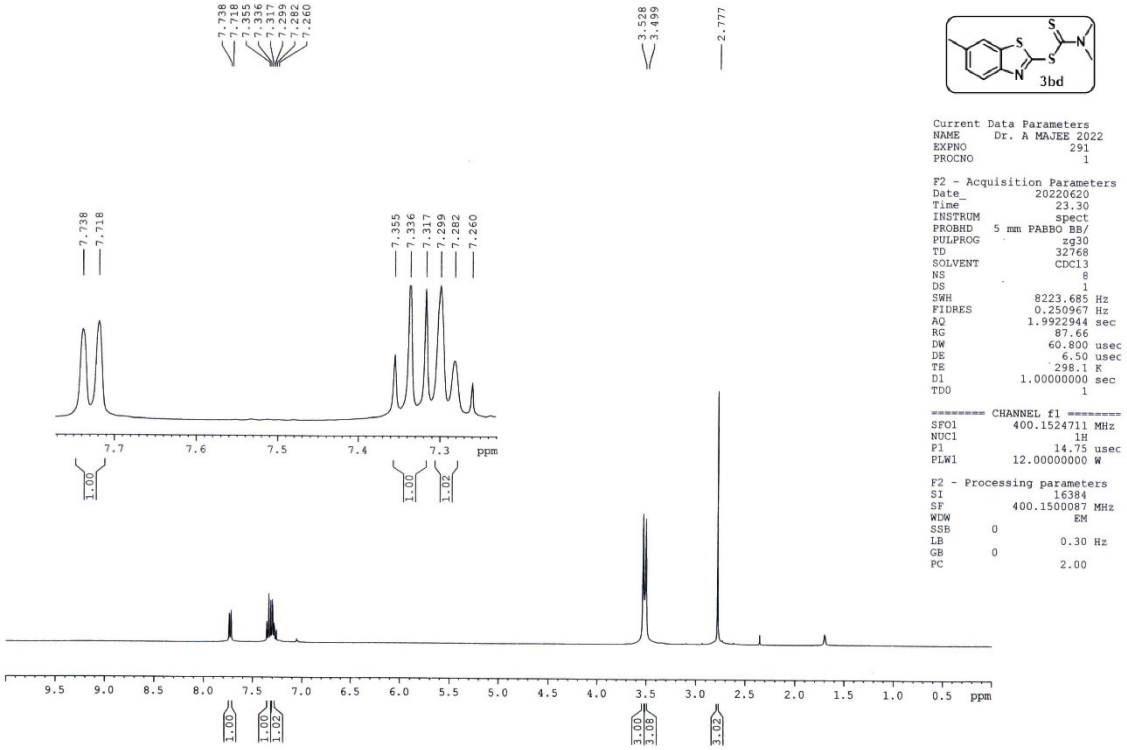
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



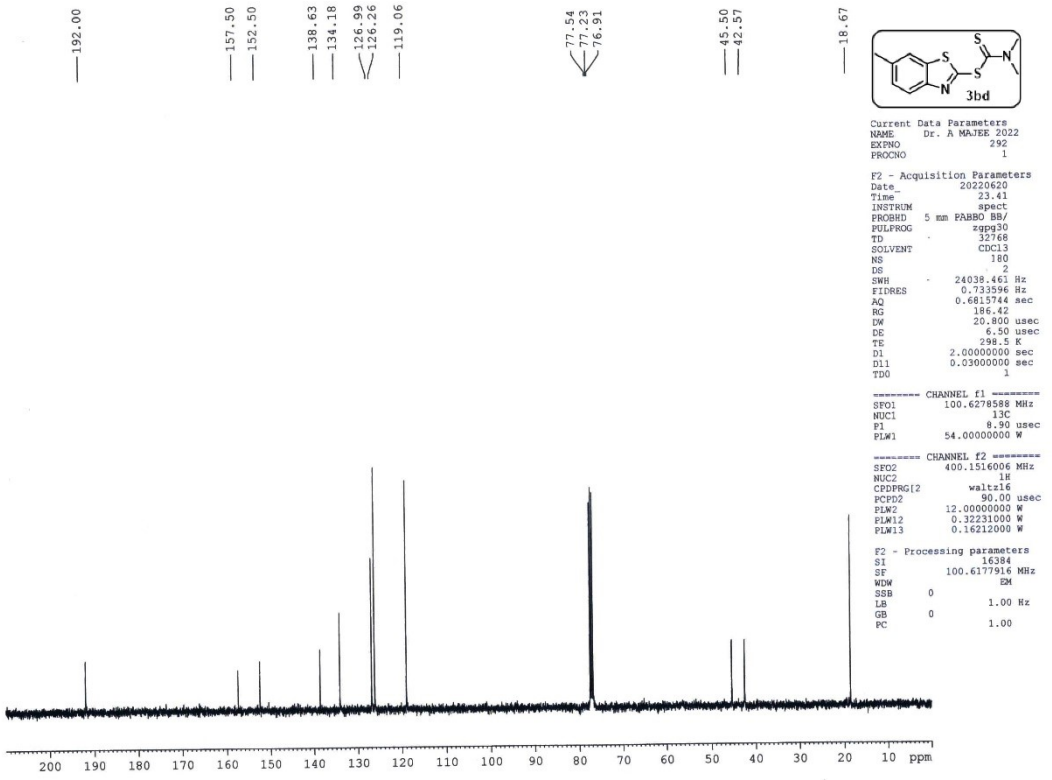
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



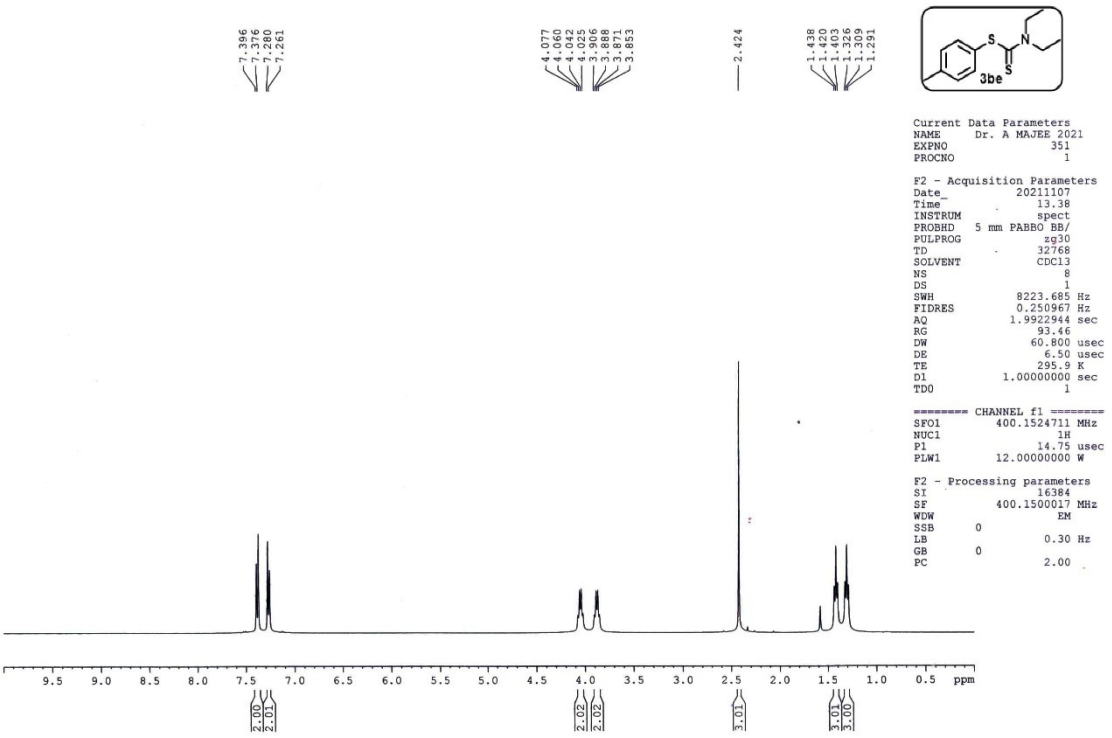
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



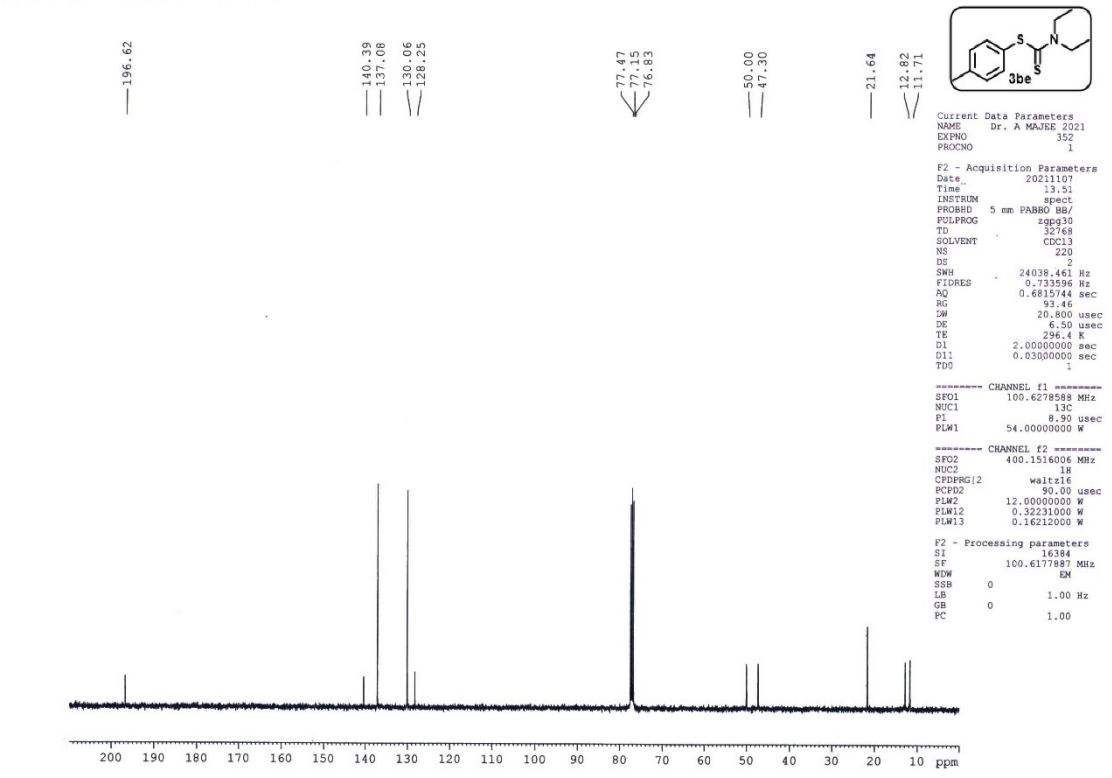
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



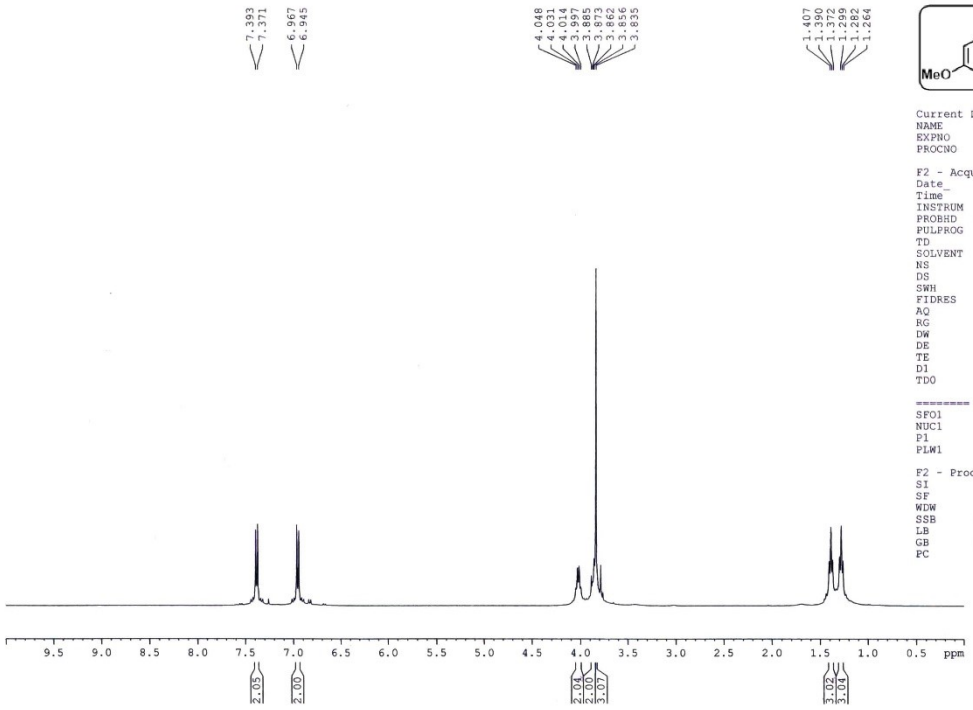
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



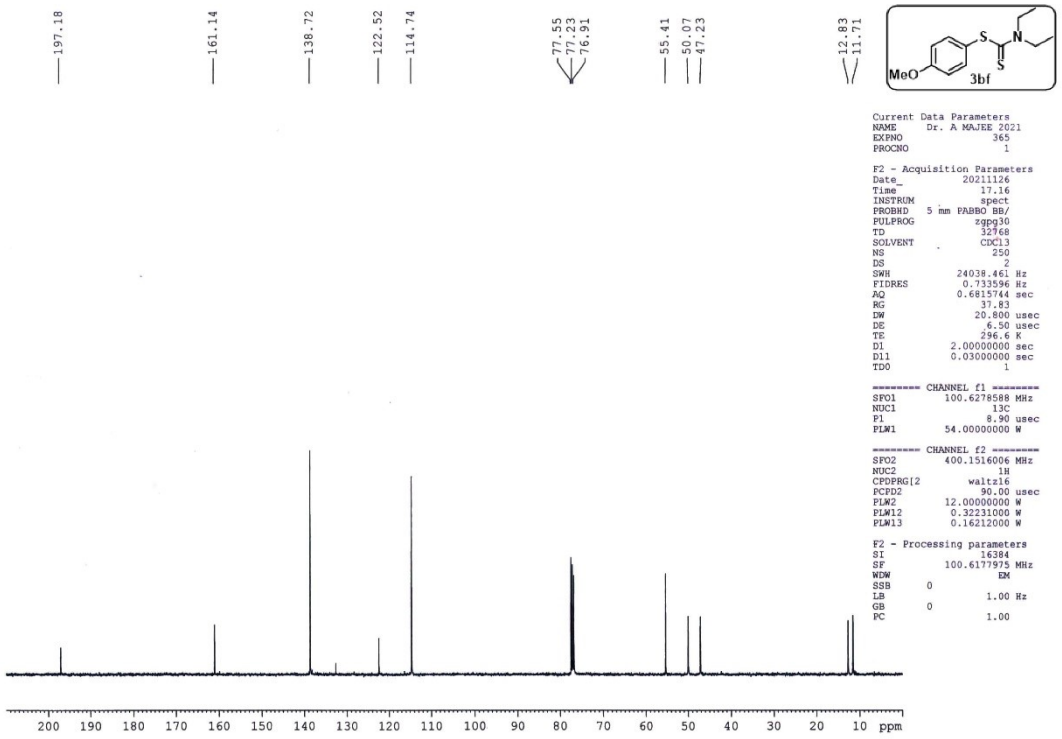
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



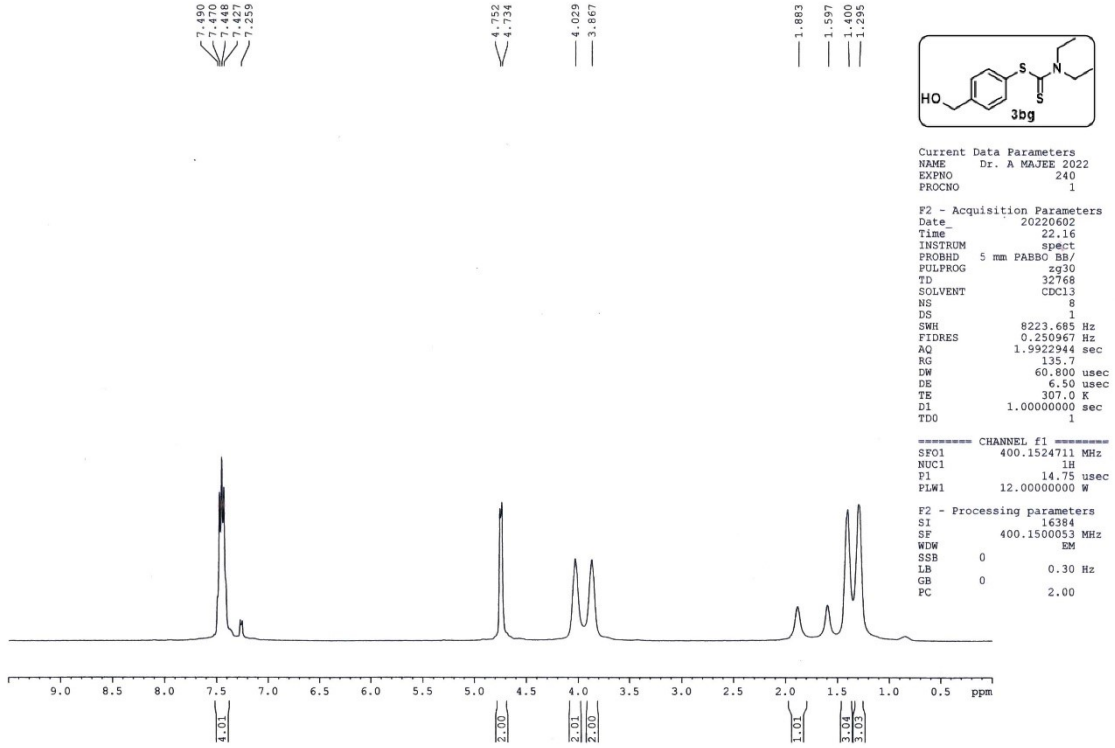
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



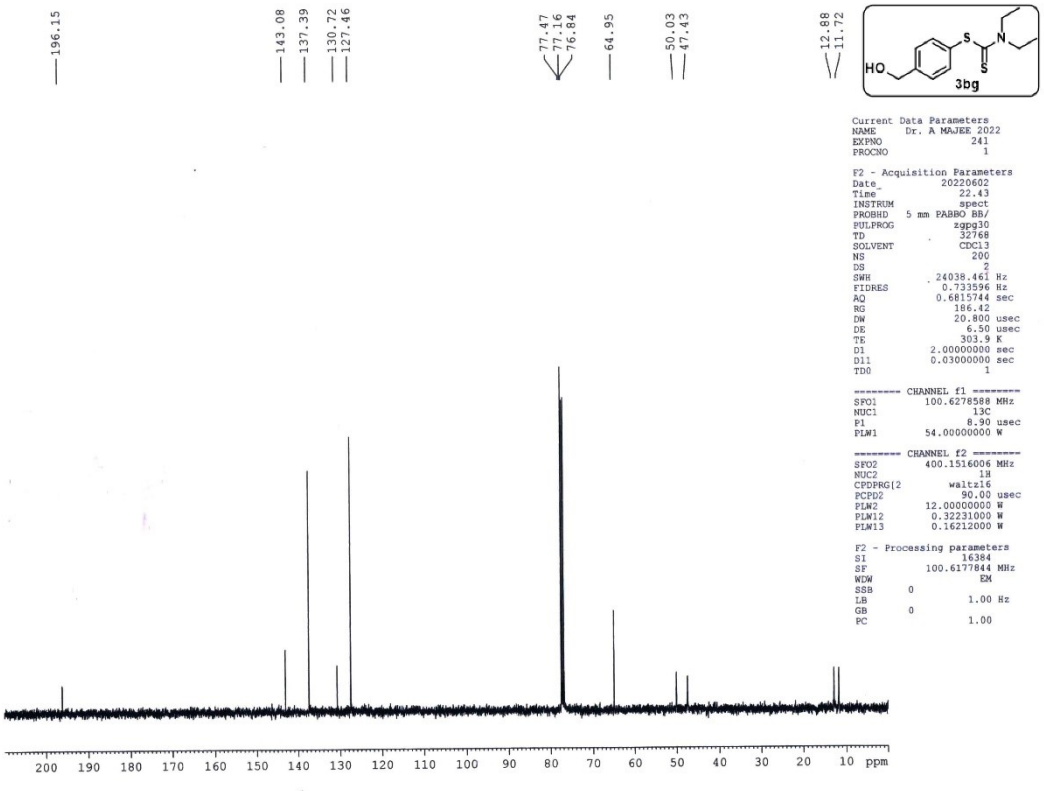
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



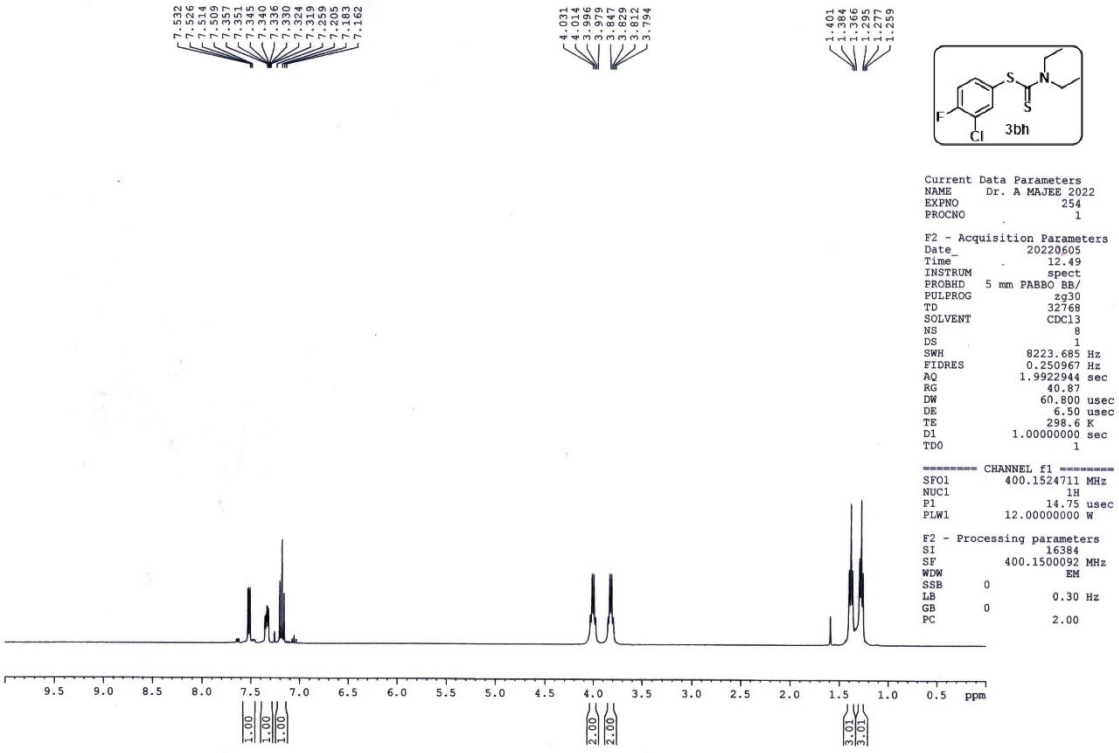
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



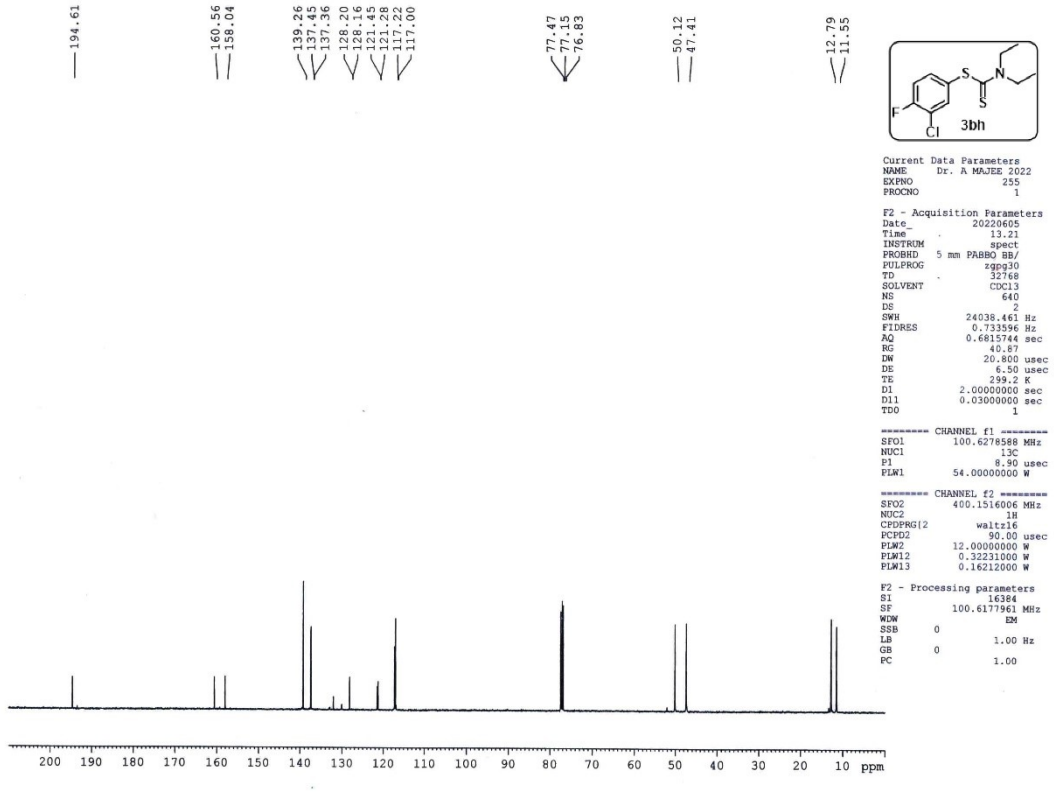
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



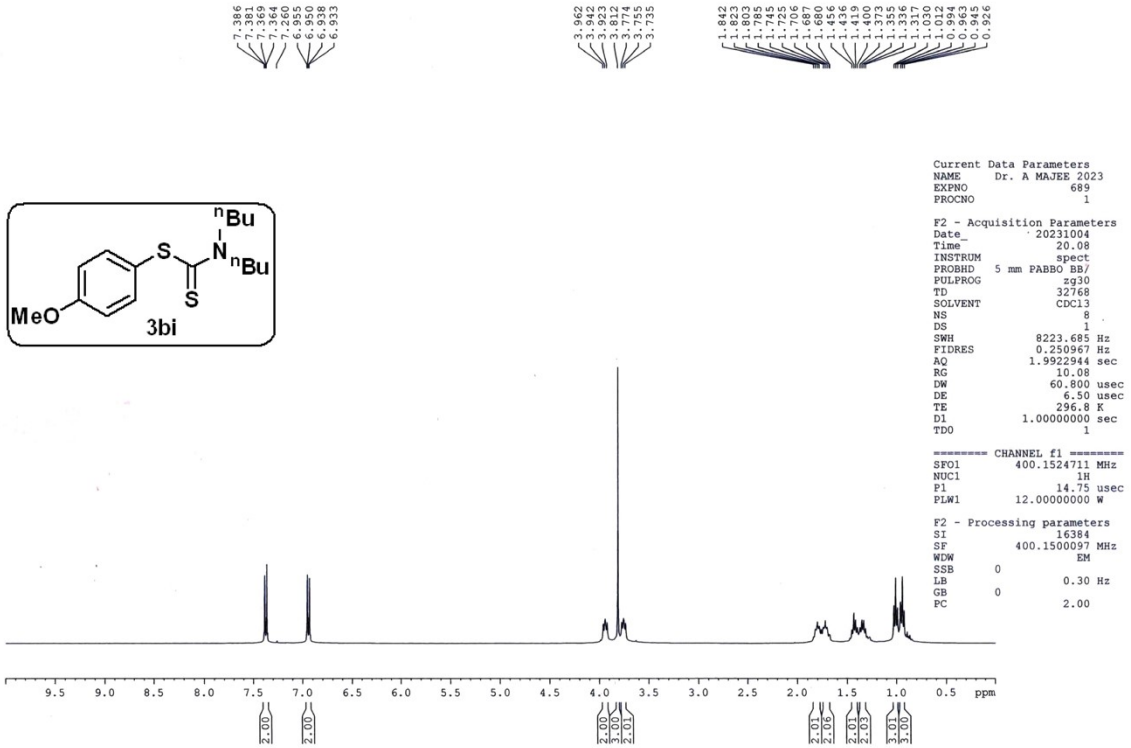
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



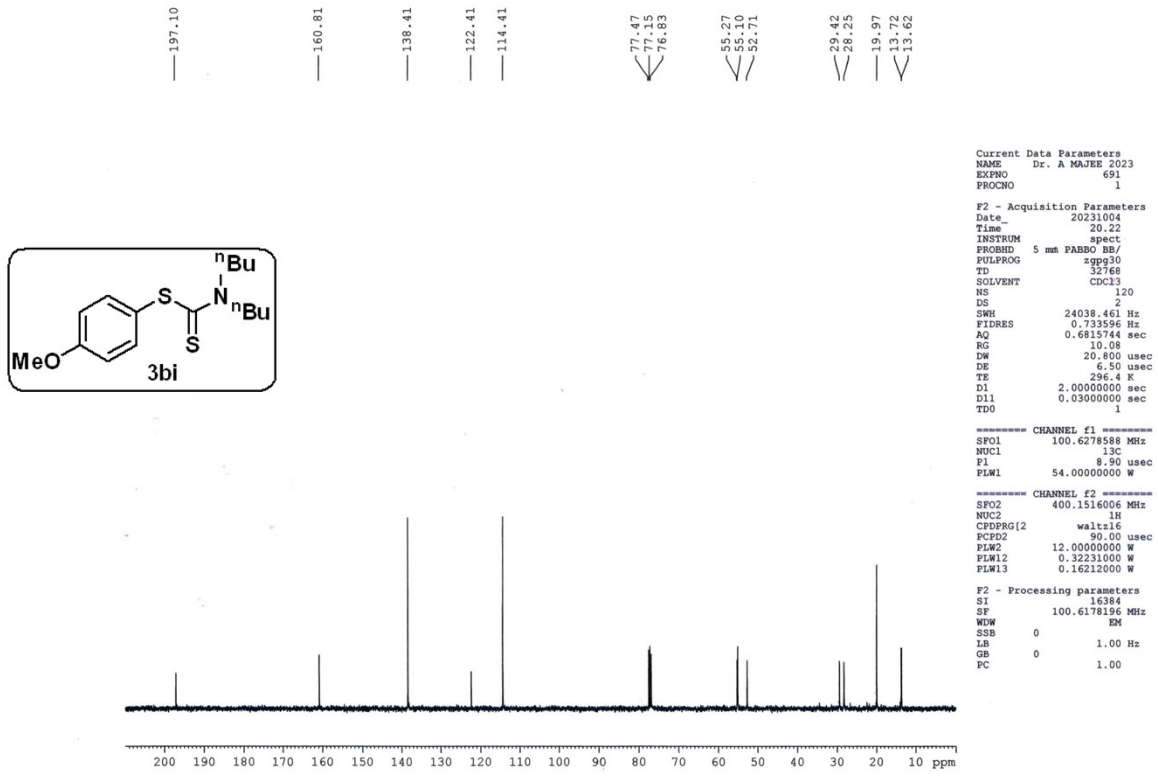
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



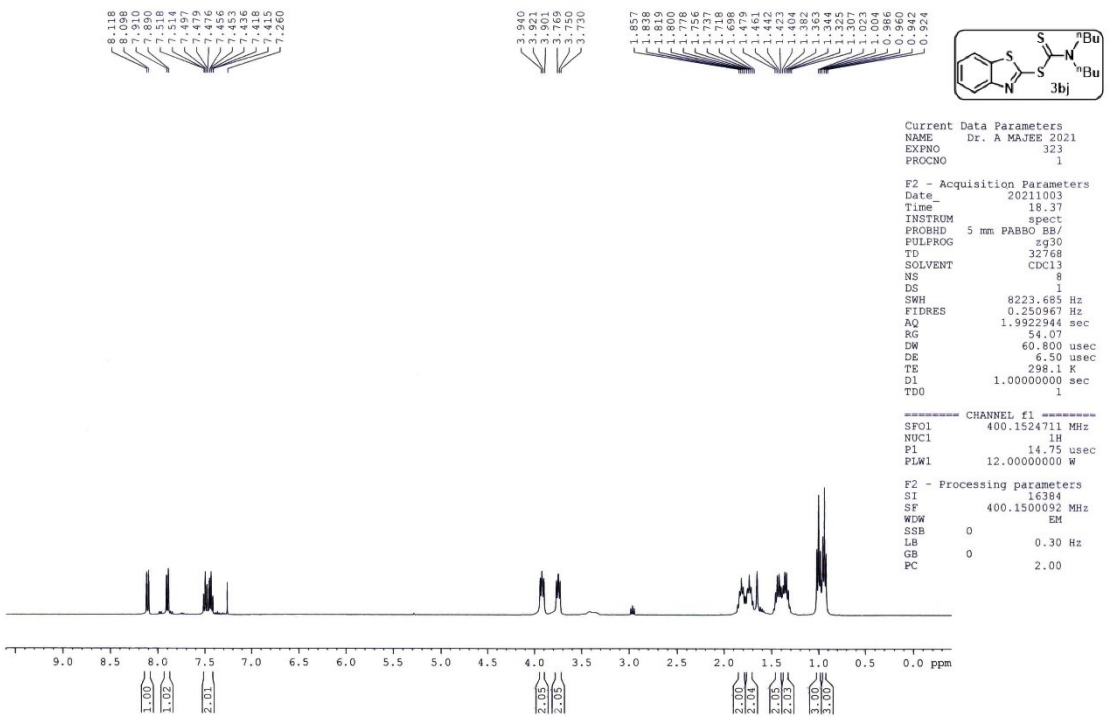
^{1}H NMR: 400MHz; Solvent: $CDCl_3$



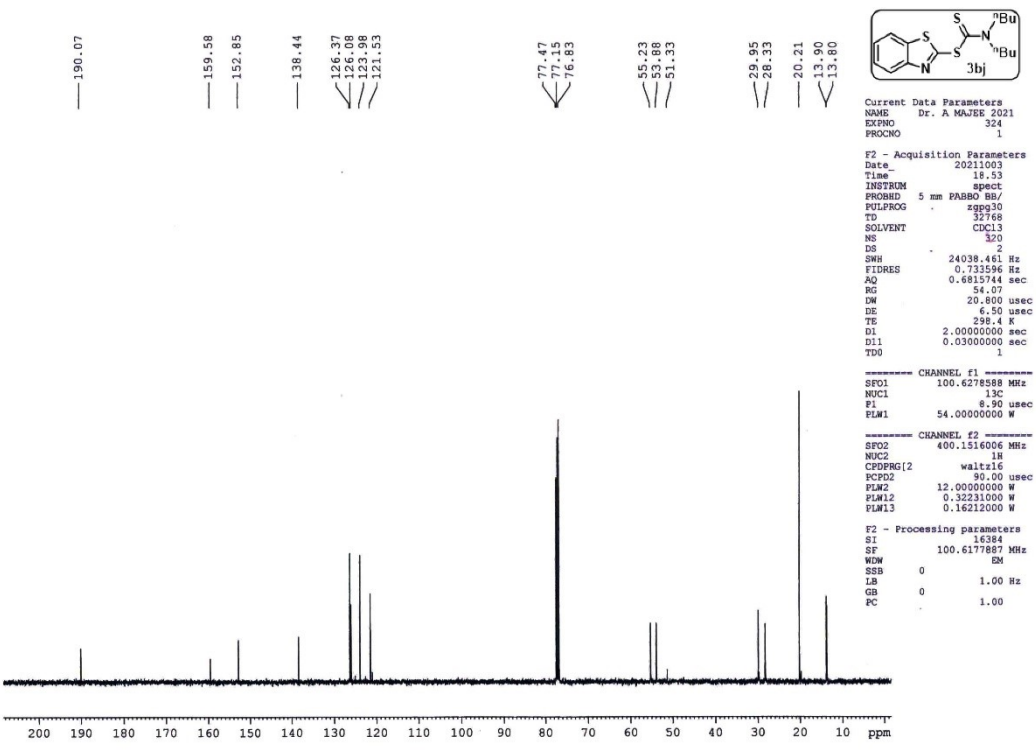
^{13}C{^{1}H} NMR: 100MHz; Solvent: $CDCl_3$



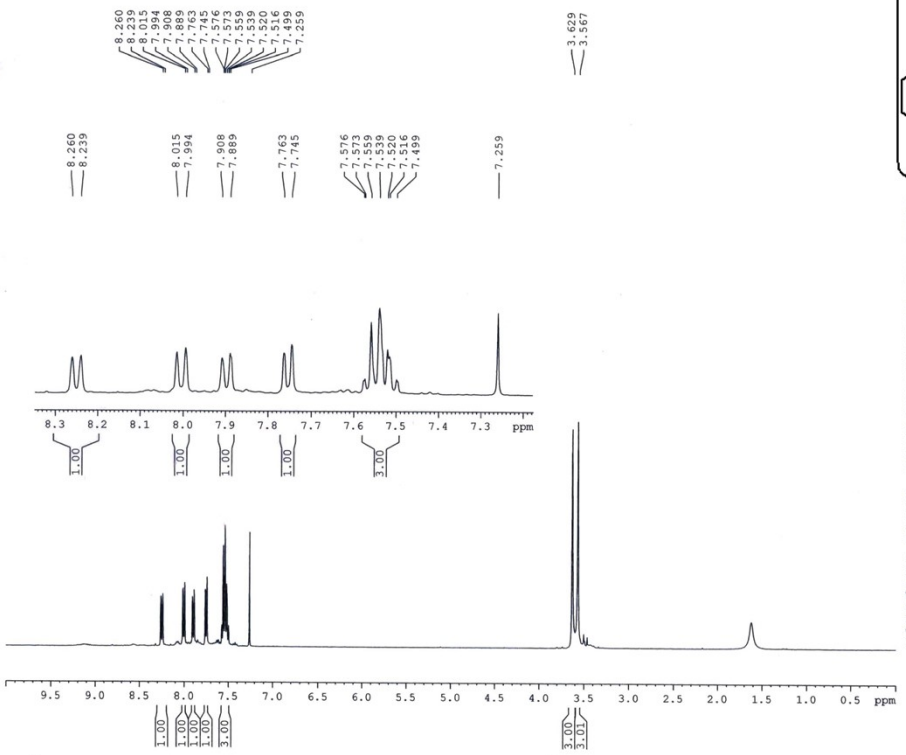
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$

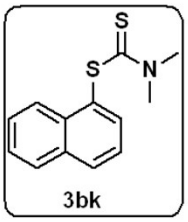


^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



^{1}H NMR: 400MHz; Solvent: $CDCl_3$



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Current Data Parameters
NAME      Dr. A MAJEE 2023
EXPNO              674
PROCNO               1

F2 - Acquisition Parameters
Date_          20230927
Time_             10.50
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   8
DS                   1
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9922944 sec
RG                135.7
DW               60.800 usec
DE                6.50 usec
TE                295.5 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         400.1524711 MHz
NUC1                1H
P1                14.75 usec
PLW1        12.00000000 W

F2 - Processing parameters
SI                16384
SF           400.1500097 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                2.00
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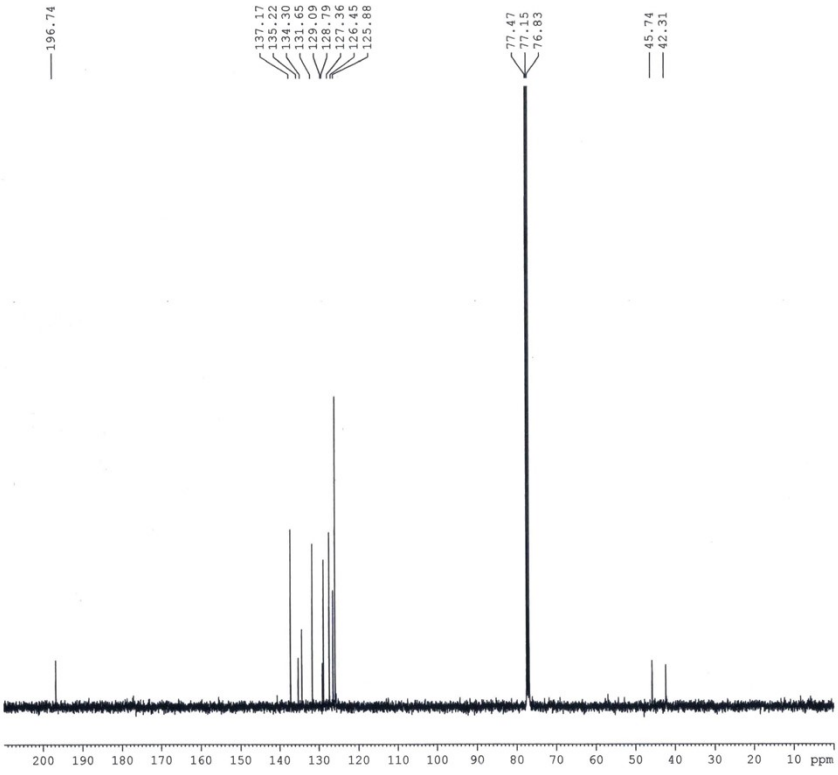
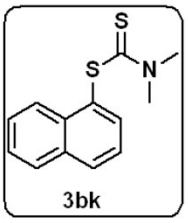
^{13}C{^{1}H} NMR: 100MHz; Solvent: $CDCl_3$



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Current Data Parameters
NAME      Dr. A MAJEE 2023
EXPNO              675
PROCNO               1

F2 - Acquisition Parameters
Date_          20230927
Time              11.32
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               32768
SOLVENT          CDCl3
NS                 800
DS                   2
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG                135.7
DW               20.800 usec
DE                6.50 usec
TE                295.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                13C
P1                8.90 usec
PLW1        54.00000000 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2            90.00 usec
PLW2        12.00000000 W
PLW12        0.32231000 W
PLW13        0.16212000 W

F2 - Processing parameters
SI                16384
SF           100.6177873 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.00
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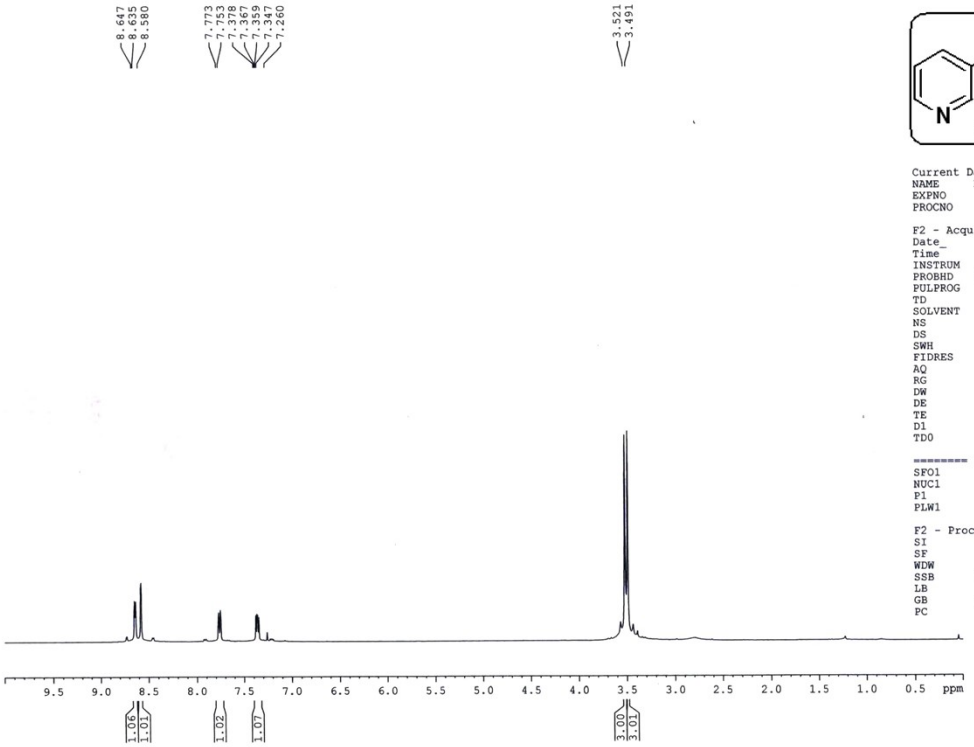
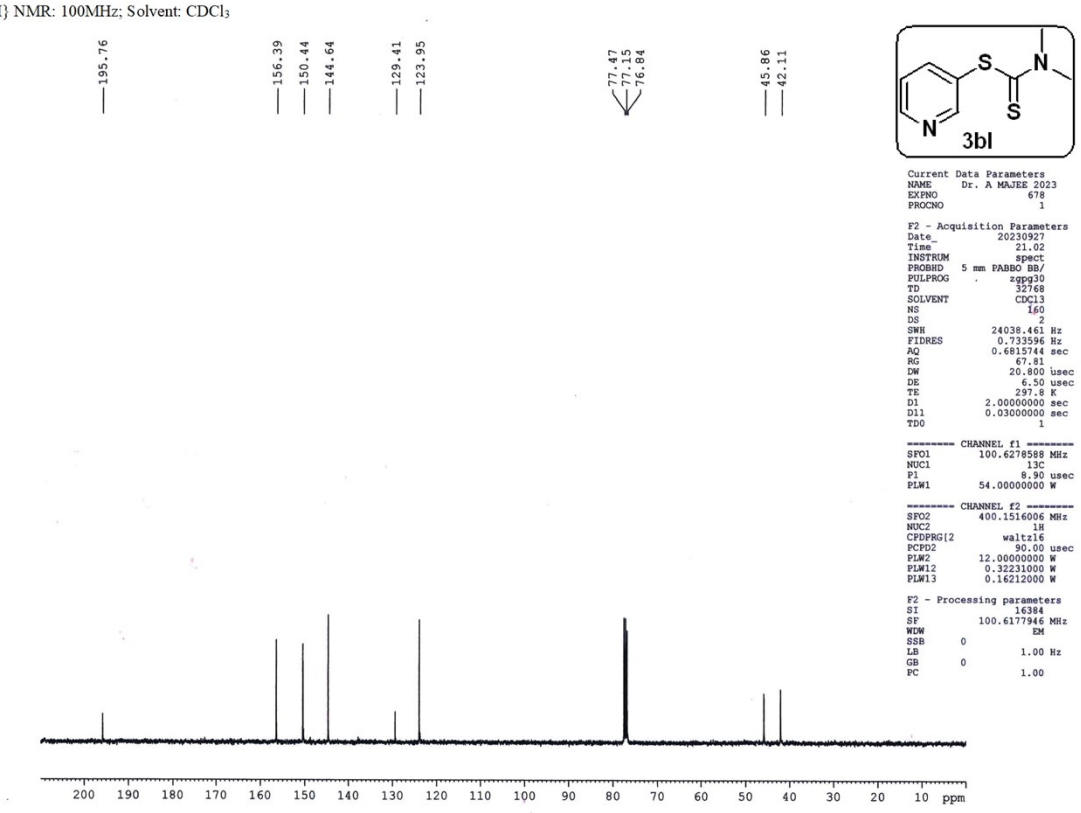
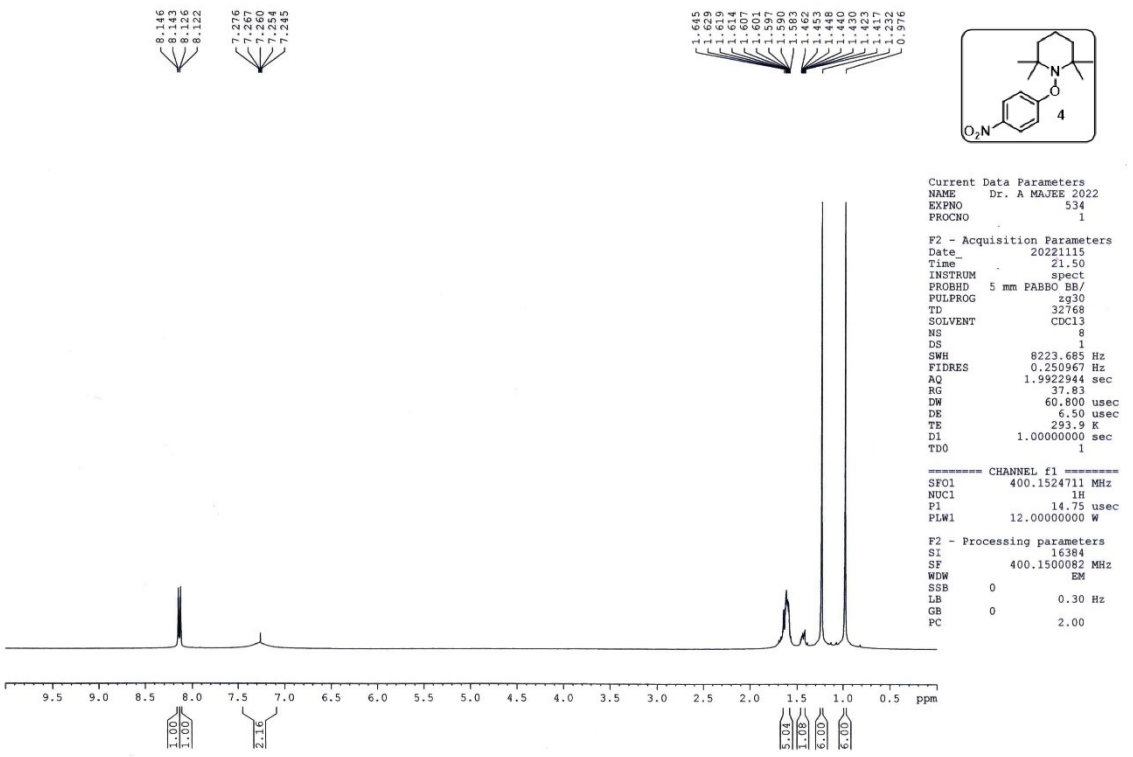
^{1}H NMR: 400MHz; Solvent: $CDCl_3$



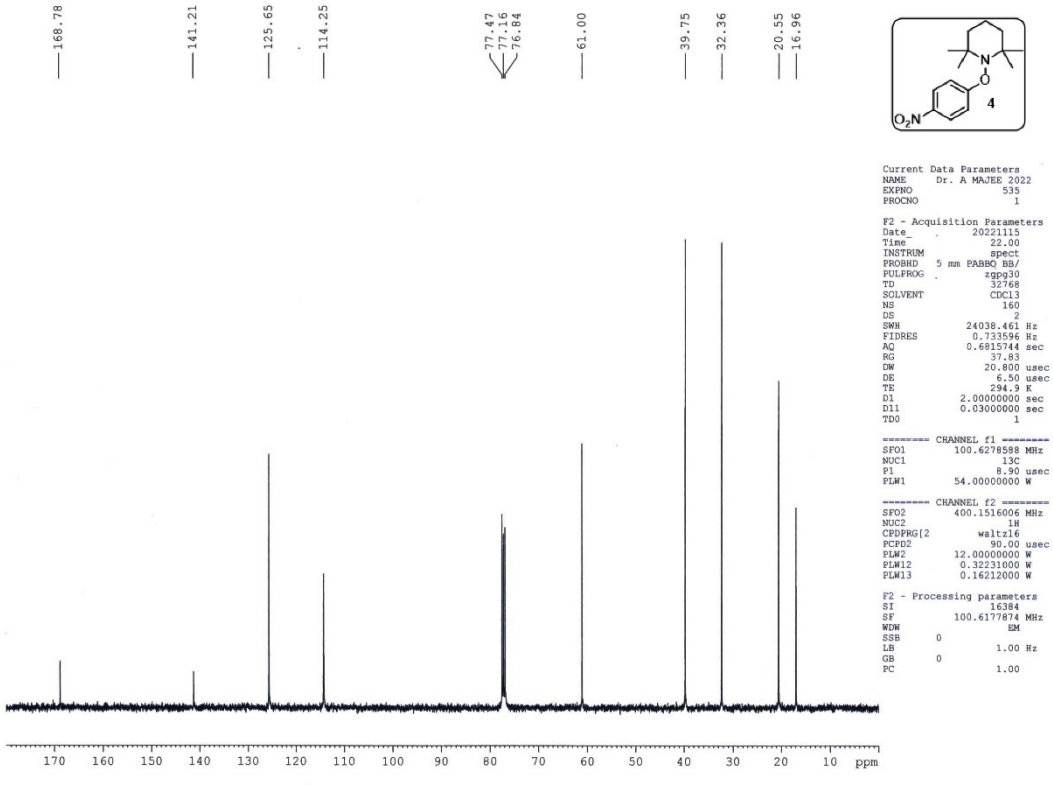
^{13}C{^{1}H} NMR: 100MHz; Solvent: $CDCl_3$



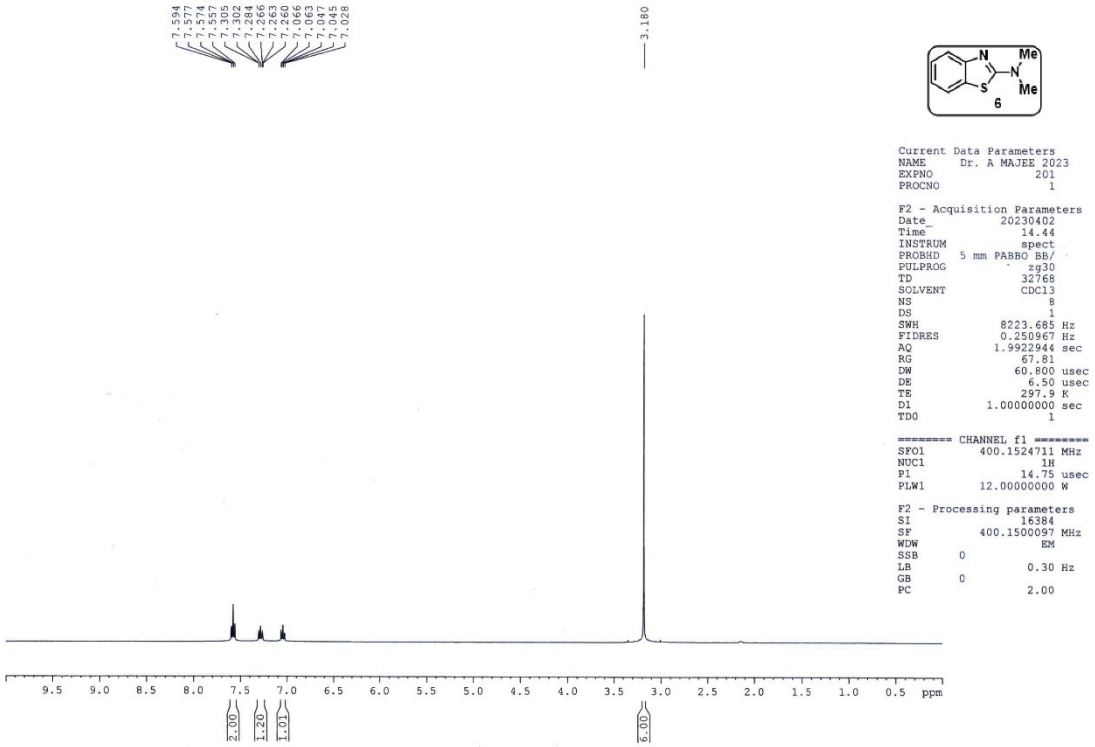
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



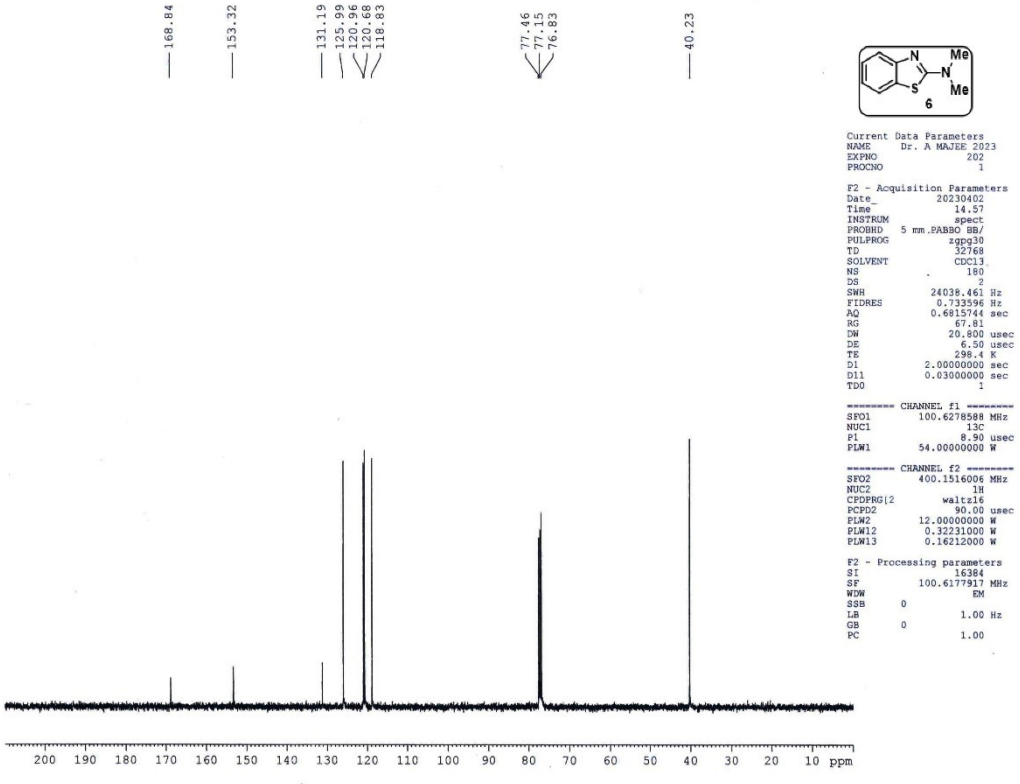
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



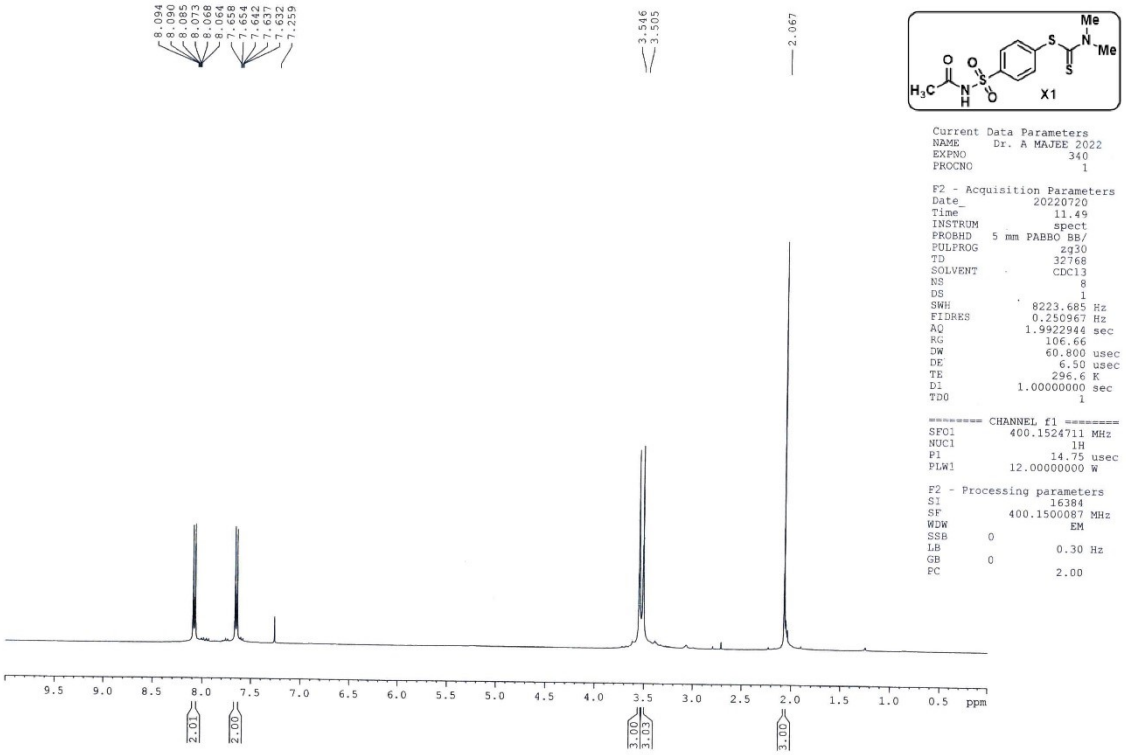
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



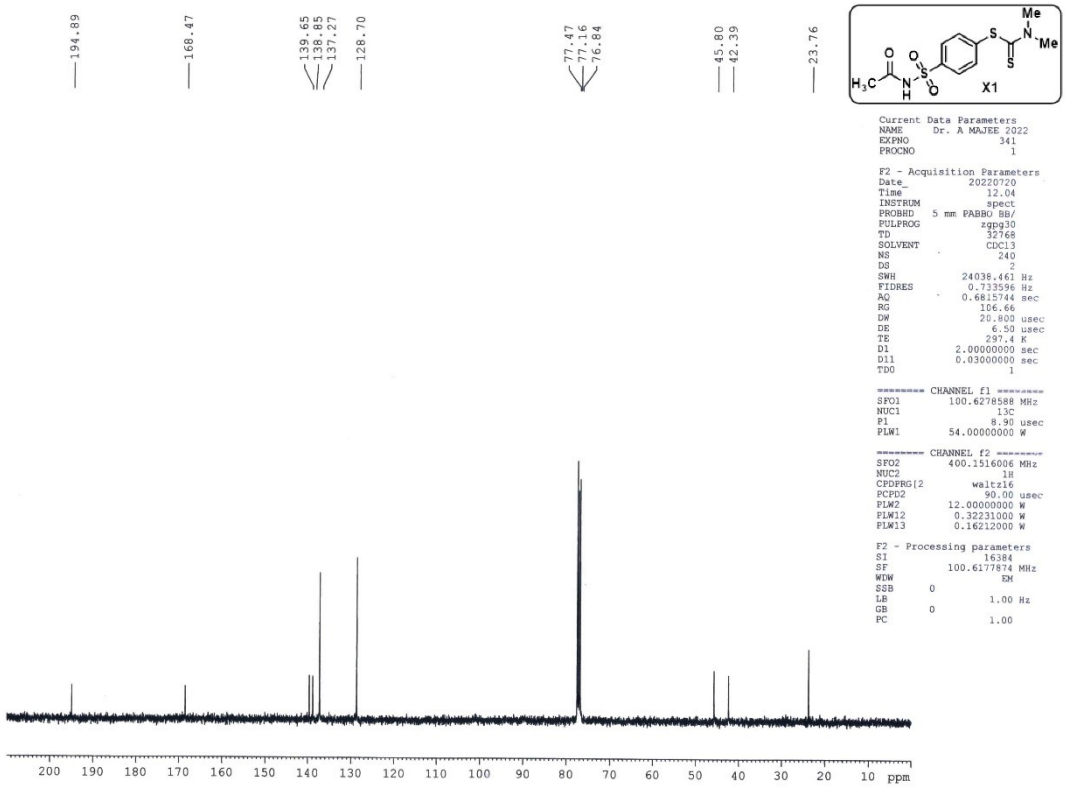
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



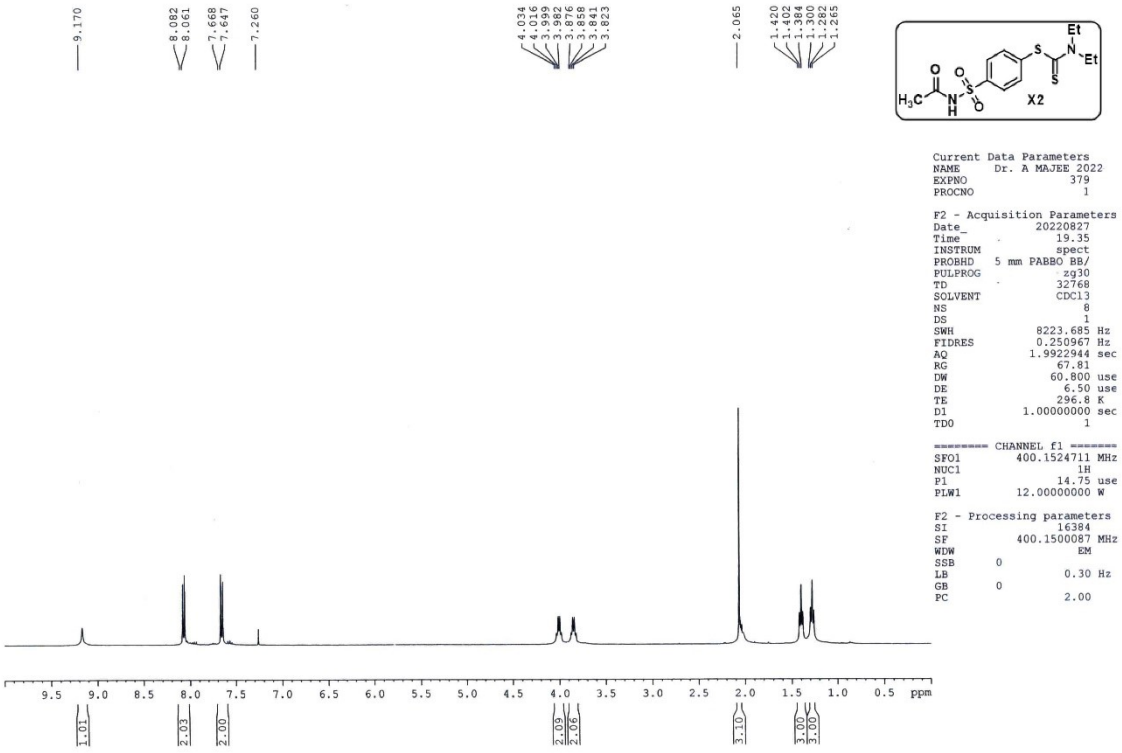
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



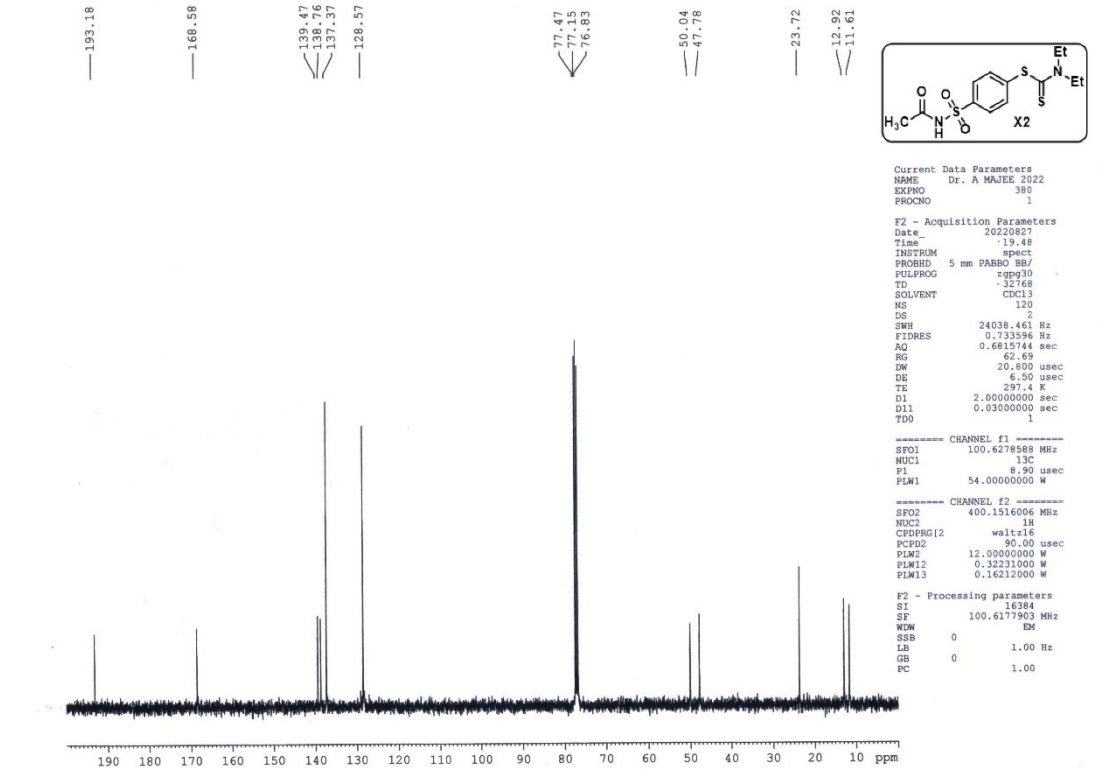
^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



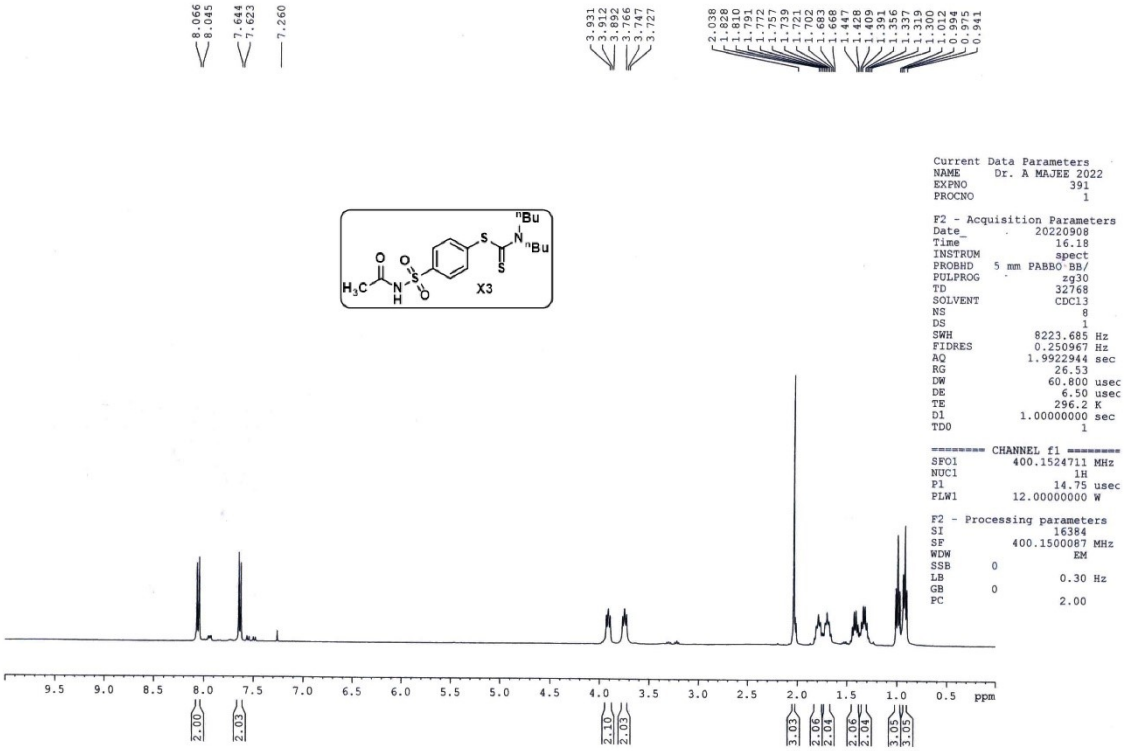
^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$



^{1}H NMR: 400 MHz; Solvent: $CDCl_3$



^{13}C{^{1}H} NMR: 100 MHz; Solvent: $CDCl_3$