

DBU mediated One Pot Synthesis of Triazolotriazine isomers via Dimroth Type Rearrangement.

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SUPPORTING INFORMATION

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1. Experiment:

The reaction was performed in 10 ml glass reaction tube at room temperature. 4,4'-(6-hydrazinyl-1,3,5-triazine-2,4-diyl)dimorpholine and simple benzaldehyde resulted in schiff base formation in 10mins and was monitored by TLC. Addition of NBS led to spontaneous reaction forming isomer **1** and it took 20 mins for its completion. The spot was isolated and characterised by NMR. Next step was addition of DBU which resulted in formation of isomer **2** in 2-4h. The progress of the reaction was monitored by TLC. The isomer **2** on TLC appeared as bluish rather than its isomer **1**.

The equivalents of DBU influenced the reaction rate drastically, the 2 eq. of DBU gave the isomer **2** in about 45 mins. The progress of reaction using different equivalents of DBU was studied for isomer **2** as shown in **Figure S1**.

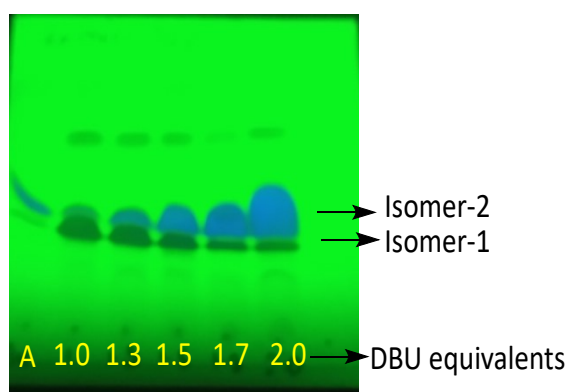


Figure S1: Visualisation of isomer-1 and isomer-2 (**2a**) under UV lamp with 254nm. (A= Authentic spot for **2a**)

Addition of water caused precipitation of the product (isomer **2**) and followed by the filtration. 1.5 eq. of DBU resulted in high yield % as compared to 2.0 eq. of DBU and it saved the super-equivalents of reagent.

2. General consideration

All the required chemicals and solvents used in this research work were purchased from commercial suppliers Sigma Aldrich, Alfa Aesar and Merck. For Thin-layered chromatography (TLC) precoated with silica gel plates were used for monitoring the progress of the reaction which was procured from E. Merck and Co. (Darmstadt, Germany) and visualized under UV lamp (254 or 365 nm). Melting point for the synthesized compounds was obtained from digital Stuart SMP10 melting point apparatus. The compounds were characterized by Bruker Alpha FT-IR spectrometer using the ATR technique in the 400-4000 cm^{-1} spectral range. The NMR (^1H & ^{13}C) spectra were obtained Bruker AVANCE III 400

MHz spectrometer using deuterated solvents- CDCl_3 and DMSO. Tetramethylsilane (TMS) was used as an internal standard at δ 0.0 parts per million (ppm) and the coupling constants (J) were stated in Hertz. The NMR multiplicities were abbreviated as s for singlet, d for doublet, dd for doublet of doublet, t for triplet, q for quartet and m for multiplet.

3. Synthesis

3.1. Synthesis and spectral characterization of 1a, 3a and 4a.

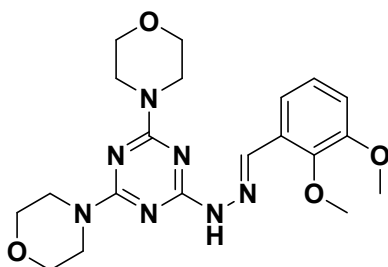
The starting materials **1a**, **3a** and **4a** were synthesised by the previously reported method.¹

3.2. Synthesis of Schiff base:

In a 10 mL glass reaction tubes equipped with a magnetic stirrer **1a** (200mg, 1 eq) was placed in 2ml of methanol. To this solution was added 2,3-dimethoxybenzaldehyde (95.3mg, 1 eq) and allowed to stir at room temperature for 20-30 mins resulting in a Schiff base and the same was monitored by the TLC. Next, addition of water (2-3ml) in reaction vessel and filtration resulted in white solid with 96% yield.

3.2.1 (E)-4,4'-(6-(2-(2,3-dimethoxybenzylidene)hydrazinyl)-1,3,5-triazine-2,4-diyl)di

morpholine (Schiff base);



White solid, Yield: 96%; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 9.39 (s, 1H), 8.13 7.59 (s, 1H), 7.60 (d, $J = 7.83$ Hz, 1H), 7.00 (t, $J = 7.30$ Hz, 1H), 6.84 (d, $J = 8.08$ Hz, 1H), 3.81-3.79 (m, 10H), 3.74-3.73 (m, 4H), 3.69-3.67 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 165.35, 164.91, 164.15, 152.63, 147.94, 138.73, 128.15, 124.12, 118.05, 113.15, 66.78, 61.51, 55.73, 43.69; MS m/z (ESI) calcd for $\text{C}_{20}\text{H}_{27}\text{N}_7\text{O}_4$ 429.21, found 430.0 $[\text{M} + \text{H}]^+$.

3.3. A General procedure for synthesis and spectral characterization of 2 (a-o), 5(a-e), 6(a-c) and 7 (a-b);

In a 10 mL glass reaction tubes equipped with a magnetic stirrer **1a**, **3a**, or **4a** (200mg, 1 eq.) was placed in 2ml of methanol. To this solution was added aldehydes (1 eq.) and allowed to stir at room temperature (20-30 mins), resulting in respective Schiff base and the same was monitored by the TLC. To the Schiff base NBS (1eq.) was slowly added to the reaction mixture and allowed to stir at room temperature for 5-30 mins, yielding isomer-**1**, which when treated with 1.5 eq. of DBU resulted in isomerisation to give isomer-**2**. After the completion of reaction, 2-3 ml of ice-cold water was added to the crude reaction mixture leading to the Precipitation, which was filtered, washed with pentane and vacuum dried to afford the final products in good to excellent yields 65-95%.

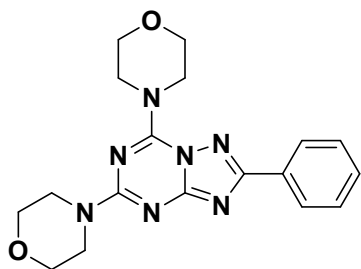
3.3.B General procedure for synthesis and spectral characterization of 1b, 3(b, c) and 4(b-d);

In a 10 mL glass reaction tubes equipped with a magnetic stirrer **1a**, **3a**, or **4a** (200mg, 1 eq.) was placed in 2ml of methanol. To this solution was added aldehydes (1 eq.) and allowed to stir at room temperature for 10-30 mins, resulting in the formation of respective Schiff bases. Further slow addition of NBS (1eq.) to this reaction mixture and stirred at room temperature (5-30 mins) yielded isomer-**1**. The crude reaction mixture was poured onto an ice-cold water (2-3ml) resulting in a precipitate. The precipitate was then filtered and washed with pentane and vacuum dried to afford the final products with yields ranging 83-89%.

3.3.C General procedure for Gram scale synthesis of 2a.

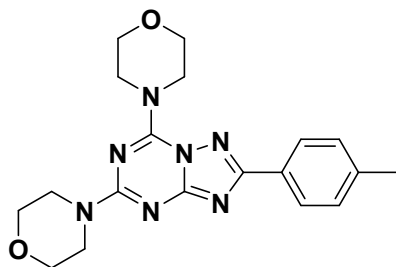
The gram scale reaction was performed to further validate this synthetic procedure. The reaction was carried out using **1a** (1g) with Benzaldehyde (0.38g) and stirred for 30 min. The NBS (1.26g, 1eq) was added slowly and stirred till we get new spot for isomer-1 followed by slow addition of DBU (1.5eq) which results in isomerisation to give **2a**. After the completion of reaction, 30-40 ml of ice-cold water was added to the crude reaction mixture leading to the Precipitation, which was filtered, washed with pentane (20ml) and vacuum dried. The product **2a** was obtained in good yields (1.1g, 84%).

3.3.1 4,4'-(2-phenyl-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (2a);



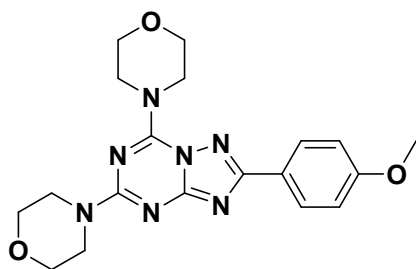
White solid, Yield: 85% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.22-8.20 (m, 2H), 7.44-7.43 (m, 3H), 4.33 (s, 4H), 3.87 (t, $J = 4.40\text{Hz}$, 8H), 3.73 (t, $J = 4.43\text{Hz}$, 4H), ; ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 163.23, 161.52, 152.44, 149.05, 130.52, 130.23, 128.61, 127.42, 66.81, 66.72, 47.20, 44.70. MS m/z calcd for $\text{C}_{18}\text{H}_{21}\text{N}_7\text{O}_2$ 367.18 found 368.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{18}\text{H}_{21}\text{N}_7\text{O}_2$: C, 58.84; H, 5.76; N, 26.69; Found: C, 57.98; H, 6.03; N, 26.25.

3.3.2 4,4'-(2-(*p*-tolyl)-[1,2,4]triazolo[1,5-*a*][1,3,5]triazine-5,7-diyl)dimorpholine (2b);



White solid, Yield: 96% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.08 (d, $J = 7.56\text{ Hz}$, 2H), 7.23 (d, $J = 7.31\text{ Hz}$, 2H), 4.32 (s, 4H), 3.85 (s, 8H), 3.72 (s, 4H), 2.38 (s, 3H) ; ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 163.73, 161.70, 159.57, 149.21, 140.51, 129.25, 127.81, 127.37, 66.79, 66.70, 47.25, 44.73, 21.51 ; MS m/z calcd for $\text{C}_{19}\text{H}_{23}\text{N}_7\text{O}_2$ 381.19, found 382.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{19}\text{H}_{23}\text{N}_7\text{O}_2$: C, 59.83; H, 6.08; N, 25.70; Found: C, 58.31; H, 6.41; N, 25.01.

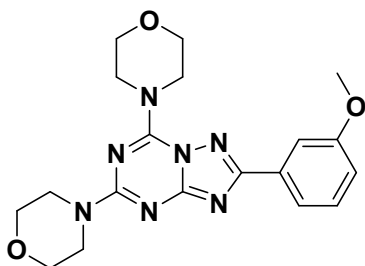
3.3.3 4,4'-(2-(4-methoxyphenyl)-[1,2,4]triazolo[1,5-*a*][1,3,5]triazine-5,7-diyl)dimorpholine (2c);



White solid, Yield: 85%; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.20 (s, 2H), 6.90 (s, 2H), 4.34 (s, 4H), 3.95-3.85 (m, 11H), 3.75 (s, 4H) ; ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 161.59,

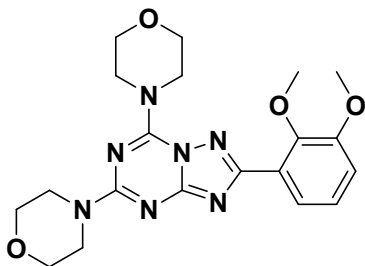
159.42, 149.10, 128.06, 122.86, 114.03, 66.84, 66.74, 55.45, 47.22, 44.73 ; MS m/z calcd for $C_{19}H_{23}N_7O_3$ 397.19, found 398.0 $[M + H]^+$. Anal.Calcd for $C_{19}H_{23}N_7O_3$: C, 57.42; H, 5.83; N, 24.67; Found: C, 56.85; H, 6.08; N, 24.25.

3.3.4 4,4'-(2-(3-methoxyphenyl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (2d);



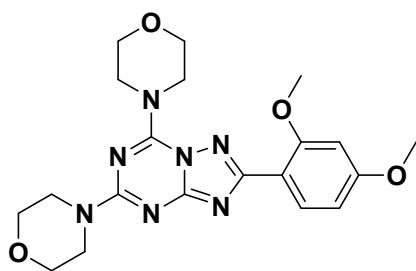
White solid, Yield: 81% ; 1H NMR (400 MHz, $CDCl_3$, δ ppm): 7.77-7.75 (m, 2H), 7.32 (t, J = 8.15 Hz, 1H), 6.98-6.95 (m, 1H), 4.31(s, 4H), 3.86-3.83(m, 11H), 3.72 (t, J = 4.75 Hz, 4H) ; ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 163.52, 161.73, 159.84, 159.43, 149.07, 131.84, 129.60, 119.62, 116.76, 112.32, 66.80, 66.71, 55.53, 47.15, 43.87 ; MS m/z calcd for $C_{19}H_{23}N_7O_3$ 397.19, found 398.0 $[M + H]^+$. Anal.Calcd for $C_{19}H_{23}N_7O_3$: C, 57.42; H, 5.83; N, 24.67; Found: C, 56.57; H, 6.15; N, 24.28.

3.3.5 4,4'-(2-(2,3-dimethoxyphenyl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (2e);



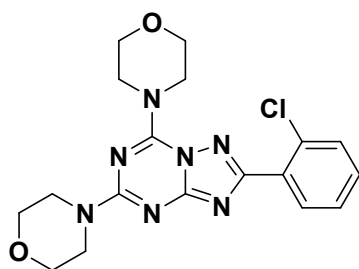
White solid, Yield: 85% ; 1H NMR (400 MHz, $DMSO-d_6$, δ ppm): 7.65 (d, J = 8.43 Hz, 1H), 7.59 (s, 1H), 7.06 (d, J = 8.39 Hz, 1H), 4.24 (s, 4H), 3.83 (s, 3H), 3.81 (s, 3H), 3.78-3.76 (m, 4H), 3.75-3.72 (m, 4H), 3.66-3.64 (m, 4H) ; ^{13}C NMR (100 MHz, $DMSO$, δ ppm): 162.24, 161.02, 158.87, 150.65, 148.66, 148.52, 122.83, 119.70, 111.64, 109.96, 65.88, 65.76, 55.54, 55.49, 46.73, 44.05; MS m/z calcd for $C_{20}H_{25}N_7O_4$ 427.20, found 428.0 $[M + H]^+$. Anal.Calcd for $C_{20}H_{25}N_7O_4$: C, 56.20; H, 5.90; N, 22.94; Found: C, 55.82 ; H, 6.13; N, 22.68.

3.3.6 4,4'-(2-(2,4-dimethoxyphenyl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine dimorpholine (2f);



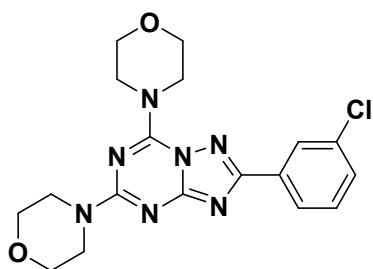
White solid, Yield: 79% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.14 (d, $J = 8.64$ Hz, 1H), 6.59-6.53 (m, 2H), 4.34 (s, 4H), 3.94-3.93 (m, 1H), 3.89 (s, 3H), 3.86-3.84 (m, 10H), 3.73-3.71 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 162.58, 159.52, 159.48, 149.06, 132.81, 112.27, 105.00, 99.30, 66.73, 56.11, 55.53, 47.14, 44.61; MS m/z calcd for $\text{C}_{20}\text{H}_{25}\text{N}_7\text{O}_4$ 427.20, found 428.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{20}\text{H}_{25}\text{N}_7\text{O}_4$: C, 56.20; H, 5.90; N, 22.94; Found: C, 53.88; H, 6.03; N, 21.50.

3.3.7 4,4'-(2-(2-chlorophenyl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (2g);



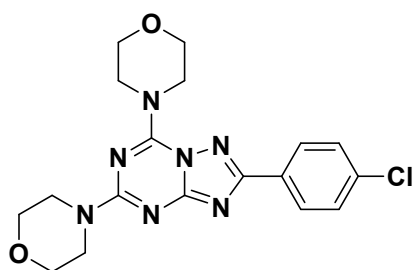
White solid, Yield: 96% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.20-8.17 (m, 1H), 7.48-7.46 (m, 1H), 7.36-7.32 (m, 2H), 4.35 (s, 4H), 3.84 (t, $J = 4.61$ Hz, 8H), 3.72 (t, $J = 4.61$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 161.95, 160.95, 159.55, 149.02, 132.93, 132.24, 131.06, 130.74, 129.21, 126.84, 66.82, 66.74, 47.18, 44.57; MS m/z calcd for $\text{C}_{18}\text{H}_{20}\text{ClN}_7\text{O}_2$ 401.14, found 402.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{18}\text{H}_{20}\text{ClN}_7\text{O}_2$: C, 53.80; H, 5.02; N, 24.40; Found: C, 53.49; H, 5.34; N, 24.29.

3.3.8 4,4'-(2-(2-chlorophenyl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (2h);



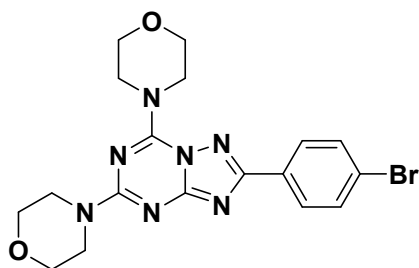
White solid, Yield: 93% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.16 (s, 1H), 8.08 (d, $J = 7.11$ Hz, 1H), 7.40-7.33 (m, 2H), 4.32 (s, 4H), 3.86 (t, $J = 4.38$ Hz, 8H), 3.72 (t, $J = 4.63$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 162.40, 161.77, 159.42, 149.02, 134.58, 132.32, 130.35, 129.92, 127.27, 125.57, 66.79, 66.70, 47.18, 44.43 ; MS m/z calcd for $\text{C}_{18}\text{H}_{20}\text{ClN}_7\text{O}_2$ 401.14 found 402.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{18}\text{H}_{20}\text{ClN}_7\text{O}_2$: C, 53.80; H, 5.02; N, 24.40; Found: C, 51.50; H, 5.59; N, 23.28.

3.3.9 4,4'-(2-(4-chlorophenyl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (2i);



White solid, Yield: 95% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.12 (d, $J = 8.70$ Hz, 2H), 7.39 (d, $J = 8.62$ Hz, 2H), 4.32 (s, 4H), 3.86 (t, $J = 4.71$ Hz, 8H), 3.73 (t, $J = 4.75$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 162.62, 161.74, 159.45, 149.04, 136.46, 128.98, 128.87, 128.69, 66.82, 66.72, 47.22, 44.61 ; MS m/z calcd for $\text{C}_{18}\text{H}_{20}\text{ClN}_7\text{O}_2$ 401.14, found 402.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{18}\text{H}_{20}\text{ClN}_7\text{O}_2$: C, 53.80; H, 5.02; N, 24.40; Found: C, 53.44; H, 5.30; N, 24.06.

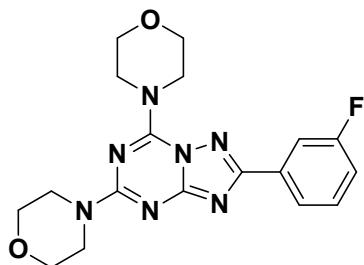
3.3.10 4,4'-(2-(4-bromophenyl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (2j);



White solid, Yield: 89% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.04 (d, $J = 8.20$ Hz, 2H), 7.54 (d, $J = 8.24$ Hz, 2H), 4.31 (s, 4H), 3.86 (t, $J = 4.39$ Hz, 8H), 3.73 (t, $J = 4.39$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 162.30, 161.51, 159.41, 148.95, 131.83, 129.18, 128.93, 124.96, 66.80, 66.70, 47.21, 44.65 ; MS m/z calcd for $\text{C}_{18}\text{H}_{20}\text{BrN}_7\text{O}_2$ 445.09, found

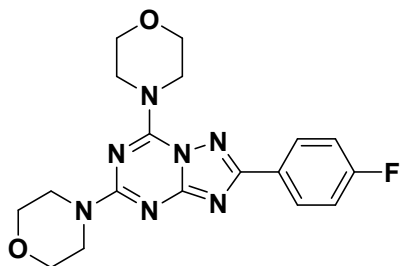
446 [M + H]⁺. Anal.Calcd for C₁₈H₂₀BrN₇O₂: C, 48.44; H, 4.52; N, 21.97; Found: C, 47.83; H, 4.75; N, 24.49.

3.3.11 4,4'-(2-(3-fluorophenyl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (2k);



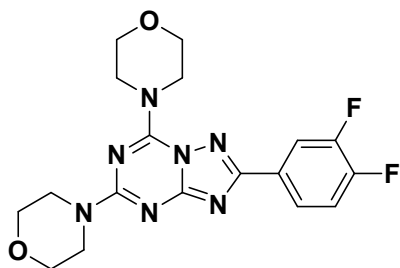
White solid, Yield: 84% ; ¹H NMR (400 MHz, CDCl₃, δ ppm): 8.00 (d, *J* = 7.68 Hz, 1H), 7.87 (d, *J* = 9.69 Hz, 1H), 7.42-7.37 (m, 1H), 7.14-7.10 (m, 1H), 4.32 (s, 4H), 3.87 (s, 8H), 3.73 (s, 4H) ; ¹³C NMR (100 MHz, CDCl₃, δ ppm): 164.19, 162.05, 161.75, 161.45, 159.42, 148.98, 132.38-132.30 (d, *J*_{C-F} = 7.97 Hz, 1C), 130.32-130.24 (d, *J*_{C-F} = 8.12 Hz, 1C), 123.24-123.21 (d, *J*_{C-F} = 2.81 Hz, 1C), 117.55-117.34 (d, *J*_{C-F} = 21.31 Hz, 1C), 114.34-114.11(d, *J*_{C-F} = 23.58 Hz, 1C), 66.81, 66.71, 47.24, 44.84; MS *m/z* calcd for C₁₈H₂₀FN₇O₂ 385.17 found 386.0 [M + H]⁺. Anal.Calcd for C₁₈H₂₀FN₇O₂: C, 56.10; H, 5.23; N, 25.44; Found: C, 55.58; H, 5.39; N, 25.27.

3.3.12 4,4'-(2-(4-fluorophenyl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (2l);



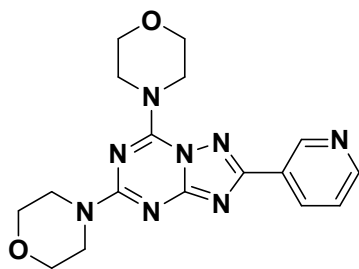
White solid Yield: 90% ; ¹H NMR (400 MHz, CDCl₃, δ ppm): 8.18 (s, 2H), 7.10 (t, *J* = 8.05 Hz, 2H), 4.31 (s, 4H), 3.85 (t, *J* = 4.53 Hz, 8H), 3.72 (t, *J* = 4.26 Hz, 4H) ; ¹³C NMR (100 MHz, CDCl₃, δ ppm): 165.62, 163.13, 159.37, 129.47-129.38 (d, *J*_{C-F} = 8.52 Hz, 1C), 126.47-126.43 (d, *J*_{C-F} = 3.31 Hz, 1C), 115.83-115.62 (d, *J*_{C-F} = 21.90 Hz, 1C), 66.82, 66.72, 47.24, 44.79 ; MS *m/z* calcd for C₁₈H₂₀FN₇O₂ 385.17 found 386.0 [M + H]⁺. Anal.Calcd for C₁₈H₂₀FN₇O₂: C, 56.10; H, 5.23; N, 25.44; Found: C, 55.29; H, 5.47; N, 24.77.

3.3.13 4,4'-(2-(3,4-difluorophenyl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (**2m**);



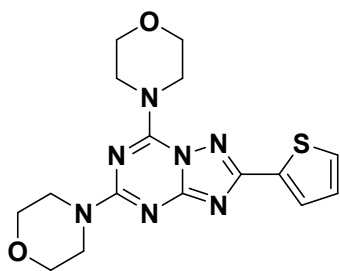
White solid, Yield: 86% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.02-7.97 (m, 2H), 7.24-7.17 (m, 1H), 4.32 (s, 4H), 3.87 (t, $J = 4.60$ Hz, 8H), 3.73 (t, $J = 4.60$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 161.55-161.42 (d, $J_{\text{C-F}} = 12.67$ Hz, 1C), 159.42, 153.28-153.16 (d, $J_{\text{C-F}} = 12.92$ Hz, 1C), 151.79-151.66 (d, $J_{\text{C-F}} = 12.92$ Hz, 1C), 150.78-150.65 (d, $J_{\text{C-F}} = 12.92$ Hz, 1C), 149.32-149.20 (d, $J_{\text{C-F}} = 12.06$ Hz, 1C), 148.96, 127.36-127.32 (d, $J_{\text{C-F}} = 4.33$ Hz, 1C), 124.00-123.90 (q, $J_{\text{C-F}} = 3.23$ Hz, 1C), 117.69-117.62 (d, $J_{\text{C-F}} = 17.95$ Hz, 1C), 116.55-116.36 (d, $J_{\text{C-F}} = 19.51$ Hz, 1C), 66.79, 66.69, 47.23, 44.81; MS m/z calcd for $\text{C}_{18}\text{H}_{19}\text{F}_2\text{N}_7\text{O}_2$ 403.16 found 426.0 $[\text{M} + \text{Na}]^+$. Anal.Calcd for $\text{C}_{18}\text{H}_{19}\text{F}_2\text{N}_7\text{O}_2$: C, 53.59; H, 4.75; N, 24.31; Found: C, 53.65; H, 4.93; N, 24.33.

3.3.14 4,4'-(2-(pyridin-3-yl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (**2n**);



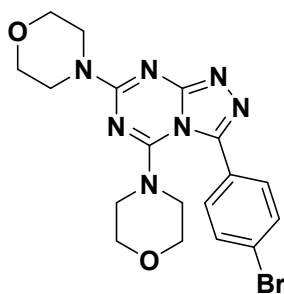
Pale yellow solid , Yield: 70% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 9.38 (s, 1H), 8.65 (d, $J = 3.59$ Hz, 1H), 8.46 (d, $J = 7.90$ Hz, 1H), 7.40-7.37 (m, 1H), 4.33 (s, 4H), 3.87 (t, $J = 5.01$ Hz, 8H), 3.73 (t, $J = 5.01$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 161.91, 161.46, 159.46, 150.77, 149.04, 148.35, 134.98, 126.81, 123.66, 66.78, 66.68, 47.22, 43.76 ; MS m/z calcd for $\text{C}_{17}\text{H}_{20}\text{N}_8\text{O}_2$ 368.17 found 369.0 $[\text{M} + \text{H}]^+$. Anal.Calcd for $\text{C}_{17}\text{H}_{20}\text{N}_8\text{O}_2$: C, 55.43; H, 5.47; N, 30.42; Found: C, 54.30; H, 5.72; N, 29.70.

3.3.15 4,4'-(2-(thiophen-2-yl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (**2o**);



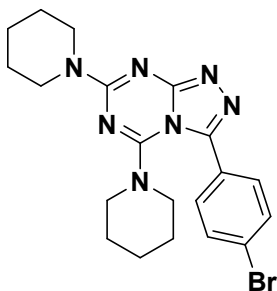
Light brown solid, Yield: 77% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 7.83 (d, $J = 3.46$ Hz, 1H), 7.40 (d, $J = 4.96$ Hz, 1H), 7.10 (t, $J = 4.10$ Hz, 1H), 4.30 (s, 4H), 3.86 (t, $J = 4.50$ Hz, 8H), 3.72 (t, $J = 4.64$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 161.61, 159.89, 159.50, 148.99, 133.52, 128.60, 128.18, 127.91, 66.82, 66.73, 47.17, 44.06 ; MS m/z calcd for $\text{C}_{16}\text{H}_{19}\text{N}_7\text{O}_2\text{S}$ 373.13 found 374.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{16}\text{H}_{19}\text{N}_7\text{O}_2\text{S}$: C, 51.46; H, 5.13; N, 26.26; Found: C, 48.80; H, 5.19; N, 24.71.

3.3.16 4,4'-(3-(4-bromophenyl)-[1,2,4]triazolo[4,3-a][1,3,5]triazine-5,7-diyl)dimorpholine (**1b**);



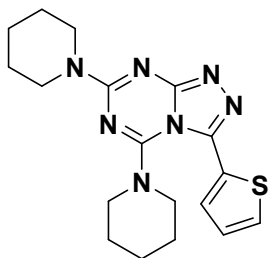
White solid, Yield: 89% ; ^1H NMR (400 MHz, $\text{DMSO}-d_6$, δ ppm): 7.86 (d, $J = 8.46$ Hz, 2H), 7.71 (d, $J = 8.46$ Hz, 2H), 3.93 (s, 2H), 3.87 (s, 2H), 3.72 (s, 4H), 3.36-3.34 (m, 4H), 3.20-3.18 (m, 4H) ; ^{13}C NMR (100 MHz, DMSO , δ ppm): 158.68, 155.07, 150.85, 142.71, 132.17, 130.69, 125.15, 124.78, 65.91, 65.70, 64.66, 49.37, 44.49 ; MS m/z (ESI) calcd for $\text{C}_{18}\text{H}_{20}\text{BrN}_7\text{O}_2$ 445.09, found 446.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{18}\text{H}_{20}\text{BrN}_7\text{O}_2$: C, 48.44; H, 4.52; N, 21.97; Found: C, 48.23; H, 4.43; N, 21.63.

3.3.17 3-(4-bromophenyl)-5,7-di(piperidin-1-yl)-[1,2,4]triazolo[4,3-a][1,3,5]triazine (**3b**);



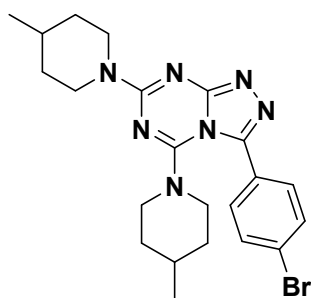
White solid, Yield: 84% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 7.59-7.52 (m, 4H), 3.83 (s, 4H), 3.11 (t, $J = 5.27$ Hz, 4H), 1.66-1.60 (m, 6H), 1.49-1.43 (m, 2H), 1.31-1.26 (m, 4H) ; ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 159.23, 158.03, 152.34, 142.80, 131.73, 129.94, 127.18, 124.01, 50.21, 24.65, 24.34, 23.76 ; MS m/z (ESI) calcd for $\text{C}_{20}\text{H}_{24}\text{BrN}_7$ 441.13, found 442.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{20}\text{H}_{24}\text{BrN}_7$: C, 54.30; H, 5.47; N, 22.16; Found: C, 52.85; H, 5.43; N, 21.25.

3.3.18 5,7-di(piperidin-1-yl)-3-(thiophen-2-yl)-[1,2,4]triazolo[4,3-a][1,3,5]triazine (3c);



White solid, Yield: 83% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 7.83 (d, $J = 3.13$ Hz, 1H), 7.36 (d, $J = 5.07$ Hz, 1H), 7.09-7.06 (m, 1H), 4.17 (s, 4H), 3.80 (s, 4H), 1.71 (s, 6H), 1.64-1.63 (m, 2H), 1.58-1.57 (m, 4H) ; ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 161.82, 159.31, 159.09, 148.79, 133.97, 128.17, 127.74, 127.69, 48.08, 25.98, 24.81, 24.56 ; MS m/z (ESI) calcd for $\text{C}_{18}\text{H}_{23}\text{N}_7\text{S}$ 369.17 found 370.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{18}\text{H}_{23}\text{N}_7\text{S}$: C, 58.51; H, 6.27; N, 26.54; Found: C, 58.09; H, 6.87; N, 26.34.

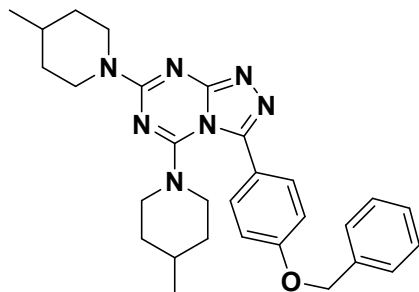
3.3.19 3-(4-bromophenyl)-5,7-bis(4-methylpiperidin-1-yl)-[1,2,4]triazolo[4,3-a][1,3,5]triazine (4b);



White solid, Yield: 87% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 7.59-7.52 (m, 4H), 4.82-4.70 (m, 3H), 3.62-3.59 (m, 2H), 2.96-2.88 (m, 2H), 2.63 (t, $J = 12.47$ Hz, 2H), 1.72-1.65 (m, 3H), 1.43 (d, $J = 10.94$ Hz, 3H), 1.16-1.14 (m, 2H), 0.94 (d, $J = 6.12$ Hz, 3H), 0.81 (d, $J = 5.76$ Hz, 4H) ; ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 159.03, 158.06, 152.17, 142.78, 131.74, 130.01, 127.09, 124.10, 49.59, 33.49, 31.15, 30.35, 21.80, 21.56 ; MS m/z (ESI) calcd for

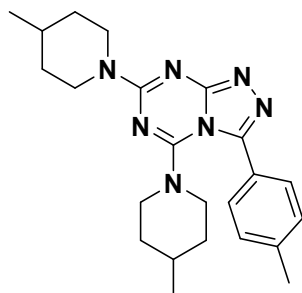
$C_{22}H_{28}BrN_7$ 469.16, found 470.0 $[M + H]^+$. Anal.Calcd for $C_{22}H_{28}BrN_7$: C, 56.17; H, 6.00; N, 20.84; Found: C, 54.23; H, 5.90; N, 20.11.

3.3.20 3-(4-(benzyloxy)phenyl)-5,7-bis(4-methylpiperidin-1-yl)-[1,2,4]triazolo[4,3-a][1,3,5]triazine (**4c**);



White solid, Yield: 88% ; 1H NMR (400 MHz, $CDCl_3$, δ ppm): 7.57 (d, $J = 8.60$ Hz, 2H), 7.43-7.35 (m, 4H), 7.32 (d, $J = 7.05$ Hz, 1H), 7.04 (d, $J = 8.68$ Hz, 2H), 5.11 (s, 2H), 4.82-4.60 (m, 2H), 4.60-4.33 (m, 1H), 3.64 (d, $J = 12.59$ Hz, 2H), 2.92 (s, 2H), 2.62 (t, $J = 12.22$ Hz, 2H), 1.73-1.63 (m, 3H), 1.42 (d, $J = 10.87$ Hz, 3H), 1.21-1.15 (m, 2H), 0.95 (d, $J = 6.28$ Hz, 3H), 0.81 (d, $J = 6.05$ Hz, 4H) ; ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 160.10, 158.12, 158.07, 152.21, 143.49, 136.45, 130.11, 128.73, 128.23, 127.51, 120.60, 115.03, 70.26, 49.67, 32.54, 31.14, 30.33, 21.80, 21.61 ; MS m/z (ESI) calcd for $C_{29}H_{35}N_7O$ 497.29, found 498.0 $[M + H]^+$. Anal.Calcd for $C_{29}H_{35}N_7O$: C, 69.99; H, 7.09; N, 19.70; Found: C, 67.22; H, 7.20; N, 18.89.

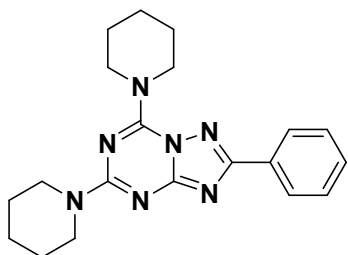
3.3.21 5,7-bis(4-methylpiperidin-1-yl)-3-(p-tolyl)-[1,2,4]triazolo[4,3-a][1,3,5]triazine (**4d**);



White solid, Yield: 89% ; 1H NMR (400 MHz, $CDCl_3$, δ ppm): 7.53 (d, $J = 7.90$ Hz, 2H), 7.27 (d, $J = 7.64$ Hz, 2H), 4.86-4.73 (m, 2H), 4.52-4.29 (m, 1H), 3.67-3.64 (m, 2H), 2.99-2.90 (m, 2H), 2.64 (t, $J = 12.48$ Hz, 2H), 1.42 (s, 3H), 1.76-1.64 (m, 3H), 1.43 (d, $J = 11.76$ Hz, 3H), 1.23-1.14 (m, 2H), 0.98 (d, $J = 6.41$ Hz, 3H), 0.81 (d, $J = 6.00$ Hz, 4H) ; ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 158.31, 128.12, 152.18, 149.77, 140.19, 129.28, 128.49, 125.08, 49.64, 32.46, 31.15, 30.30, 21.80, 21.57, 21.51 ; MS m/z (ESI) calcd for $C_{23}H_{31}N_7$ 405.26,

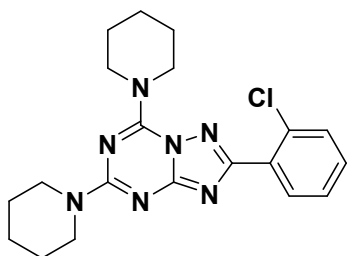
found 406.0 $[M + H]^+$. Anal.Calcd for $C_{23}H_{31}N_7$: C, 68.12; H, 7.71; N, 24.28; Found: C, 64.64; H, 7.75; N, 22.64.

3.3.22 2-phenyl-5,7-di(piperidin-1-yl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine (**5a**);



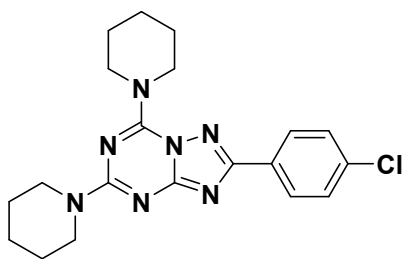
White solid, Yield: 89% ; 1H NMR (400 MHz, $CDCl_3$, δ ppm): 8.26-8.24 (m, 2H), 7.44-7.43 (m, 3H), 4.23 (s, 4H), 3.83(s, 4H), 1.75 (s, 6H), 1.67-1.66 (m, 2H), 1.61-1.60 (m, 4H) ; ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 161.85, 159.31, 148.91, 130.71, 130.15, 128.50, 127.36, 48.15, 45.26, 26.04, 25.93, 24.85, 24.60; MS m/z (ESI) calcd for $C_{20}H_{25}N_7$ 363.22, found 364.0 $[M + H]^+$. Anal.Calcd for $C_{20}H_{25}N_7$: C, 66.09; H, 6.93; N, 26.98; Found: C, 65.16; H, 7.23; N, 26.76.

3.3.23 2-(2-chlorophenyl)-5,7-di(piperidin-1-yl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine (**5b**);



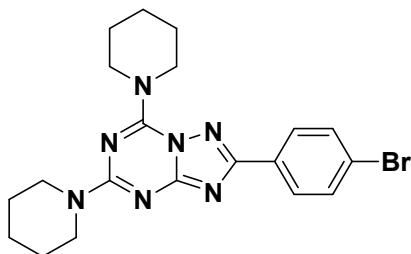
White solid, Yield: 92% ; 1H NMR (400 MHz, $CDCl_3$, δ ppm): 8.23-8.21 (m, 1H), 7.47-7.46 (m, 1H), 7.33-7.31 (m, 2H), 4.24 (s, 4H), 3.82 (s, 4H), 1.73 (s, 6H), 1.67-1.64 (m, 2H), 1.60-1.59 (m, 4H) ; ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 161.35, 161.30, 159.35, 148.79, 132.85, 132.13, 130.90, 130.29, 126.66, 126.62, 48.07, 45.25, 26.03, 25.87, 24.79, 24.53; MS m/z (ESI) calcd for $C_{20}H_{24}ClN_7$ 397.18, found 398.0 $[M + H]^+$. Anal.Calcd for $C_{20}H_{24}ClN_7$: C, 60.37; H, 6.08; N, 24.64; Found: C, 65.16; H, 6.72; N, 26.58.

3.3.24 2-(4-chlorophenyl)-5,7-di(piperidin-1-yl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine (**5c**);



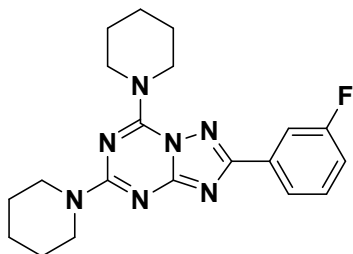
White solid, Yield: 91% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.17 (d, $J = 8.41$ Hz, 2H), 7.40 (d, $J = 8.33$ Hz, 2H), 4.21 (s, 4H), 3.83 (s, 4H), 1.75 (s, 6H), 1.67-1.66 (m, 2H), 1.60-1.59 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 162.12, 161.99, 159.26, 148.84, 135.90, 129.55, 128.66, 128.55, 48.07, 45.21, 25.99, 25.89, 24.80, 24.54; MS m/z (ESI) calcd for $\text{C}_{20}\text{H}_{24}\text{ClN}_7$ 397.18, found 398.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{20}\text{H}_{24}\text{ClN}_7$: C, 60.37; H, 6.08; N, 24.64; Found: C, 59.89; H, 6.27; N, 24.47.

3.3.25 2-(4-bromophenyl)-5,7-di(piperidin-1-yl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine (**5d**);



White solid, Yield: 87% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.11 (d, $J = 8.38$ Hz, 2H), 7.56 (d, $J = 8.43$ Hz, 2H), 4.21 (s, 4H), 3.83 (s, 4H), 1.75 (s, 6H), 1.67-1.66 (m, 2H), 1.60-1.59 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 161.93, 161.77, 159.27, 148.83, 131.70, 129.78, 128.87, 124.48, 48.15, 45.35, 26.02, 25.92, 24.83, 24.57 ; MS m/z (ESI) calcd for $\text{C}_{20}\text{H}_{24}\text{BrN}_7$ 441.13, found 442.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{20}\text{H}_{24}\text{BrN}_7$: C, 54.30; H, 5.47; N, 22.16; Found: C, 54.40; H, 5.77; N, 21.95.

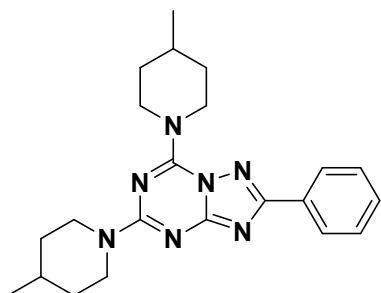
3.3.26 2-(3-fluorophenyl)-5,7-di(piperidin-1-yl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine (**5e**);



White solid, Yield: 86% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.04 (s, 1H), 7.72 (d, $J = 9.34$ Hz, 1H), 7.42-7.37 (m, 1H), 7.11 (t, $J = 8.33$ Hz, 1H), 4.21 (s, 4H), 3.83 (s, 4H), 1.75 (s, 6H), 1.67-1.66 (m, 2H), 1.60-1.59 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 164.17, 161.74, 159.26, 148.89, 133.16-133.07 (d, $J_{\text{C-F}} = 8.87$ Hz, 1C), 130.12-130.04 (d, $J_{\text{C-F}} = 8.20$

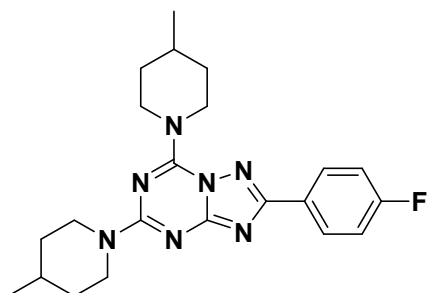
Hz, 1C), 123.06-123.04 (d, $J_{\text{C-F}} = 2.82$ Hz, 1C), 117.02-116.80 (d, $J_{\text{C-F}} = 21.59$ Hz, 1C), 114.20-113.97 (d, $J_{\text{C-F}} = 23.27$ Hz, 1C), 48.15, 45.23, 26.02, 25.91, 24.82, 24.56 ; MS m/z (ESI) calcd for $\text{C}_{20}\text{H}_{24}\text{FN}_7$ 381.21, found 382.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{20}\text{H}_{24}\text{FN}_7$: C, 62.97; H, 6.34; N, 25.70; Found: C, 62.68; H, 6.47; N, 25.56.

3.3.27 5,7-bis(4-methylpiperidin-1-yl)-2-phenyl-[1,2,4]triazolo[1,5-a][1,3,5]triazine (6a);



White solid, Yield: 88% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.23-8.21 (m, 2H), 7.43-7.41 (m, 3H), 5.34 (s, 2H), 4.80-4.61 (m, 2H), 3.10 (t, $J = 12.44$ Hz, 2H), 2.86 (t, $J = 12.44$ Hz, 2H), 1.82-1.59 (m, 6H), 1.38-1.28 (m, 2H), 1.20-1.10 (m, 2H), 0.99 (d, $J = 6.24$ Hz, 3H), 0.95 (d, $J = 6.44$ Hz, 3H) ; ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 162.97, 162.10, 159.30, 148.94, 130.99, 130.02, 128.45, 127.28, 47.39, 44.62, 34.21, 31.31, 31.13, 21.94, 21.78; MS m/z (ESI) calcd for $\text{C}_{24}\text{H}_{29}\text{N}_7$ 391.25, found 392.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{24}\text{H}_{29}\text{N}_7$: C, 67.49; H, 7.47; N, 25.04; Found: C, 66.13; H, 7.88; N, 24.51.

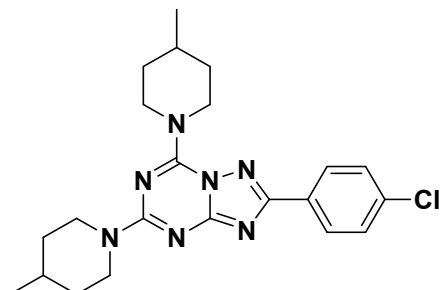
3.3.28 2-(4-fluorophenyl)-5,7-bis(4-methylpiperidin-1-yl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine (6b);



White solid, Yield: 86% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 8.23-8.20 (m, 2H), 7.13-7.08 (m, 2H), 5.32 (s, 2H), 4.83-4.59 (m, 2H), 3.11 (t, $J = 12.63$ Hz, 2H), 2.87 (t, $J = 12.63$ Hz, 2H), 1.82-1.60 (m, 6H), 1.38-1.28 (m, 2H), 1.20-1.10 (m, 2H), 0.99 (d, $J = 6.34$ Hz, 3H), 0.95 (d, $J = 6.34$ Hz, 3H) ; ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 165.33, 162.85, 162.08, 159.27, 148.87, 129.29-129.21 (d, $J_{\text{C-F}} = 8.63$ Hz, 1C), 127.20-127.17 (d, $J_{\text{C-F}} = 3.05$ Hz, 1C), 115.56-115.34 (d, $J_{\text{C-F}} = 21.93$ Hz, 1C), 47.38, 44.67, 34.18, 31.28, 31.10, 21.92, 21.75 ; MS

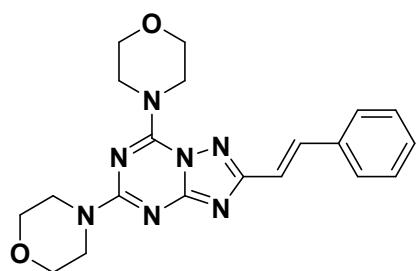
m/z (ESI) calcd for $C_{22}H_{28}FN_7$ 409.24, found 410.0 $[M + H]^+$. Anal. Calcd for $C_{22}H_{28}FN_7$: C, 64.53; H, 6.89; N, 23.94; Found: C, 63.64; H, 6.93; N, 23.50.

3.3.29 2-(4-chlorophenyl)-5,7-bis(4-methylpiperidin-1-yl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine (**6c**);



White solid, Yield: 90%; FTIR (ATR, V_{max} , cm^{-1}): 3470.65 (Amide N-H Str.), 2919.78 (Alkyl C-H Str.), 1628.03 (Amide C=O Str.); 1H NMR (400 MHz, $CDCl_3$, δ ppm): 8.16 (d, $J = 8.54$ Hz, 2H), 7.39 (d, $J = 8.42$ Hz, 2H), 5.31 (s, 2H), 4.84-4.73 (m, 2H), 3.11 (t, $J = 12.57$ Hz, 2H), 2.87 (t, $J = 12.26$ Hz, 2H), 1.83-1.60 (m, 6H), 1.38-1.28 (m, 2H), 1.20-1.10 (m, 2H), 0.99 (d, $J = 6.38$ Hz, 3H), 0.95 (d, $J = 6.31$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 161.73, 159.27, 138.82, 136.23, 129.11, 128.82, 128.71, 47.50, 44.75, 34.23, 31.31, 31.14, 21.95, 21.79; MS m/z (ESI) calcd for $C_{22}H_{28}ClN_7$ 425.21, found 426.0 $[M + H]^+$. Anal. Calcd for $C_{22}H_{28}ClN_7$: C, 62.03; H, 6.63; N, 23.02; Found: C, 61.78; H, 6.67; N, 22.87.

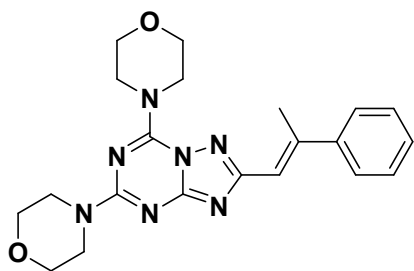
3.3.30 (E)-4,4'-(2-styryl-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)dimorpholine (**7a**);



White solid, Yield: 84%; 1H NMR (400 MHz, $CDCl_3$, δ ppm): 7.83 (d, $J = 16.15$ Hz, 1H), 7.54 (d, $J = 7.18$ Hz, 2H), 7.37-7.34 (m, 2H), 7.30 (d, $J = 7.15$ Hz, 1H), 6.98 (d, $J = 16.12$ Hz, 1H), 4.28 (s, 4H), 3.84 (t, $J = 4.74$ Hz, 8H), 3.72 (t, $J = 4.99$ Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 163.28, 161.19, 159.53, 149.03, 138.04, 136.21, 128.96, 128.84, 127.40, 117.21, 66.81, 66.72, 47.01, 44.60; MS m/z (ESI) calcd for $C_{20}H_{23}N_7O_2$ 393.19, found 394.0 $[M + H]^+$. Anal. Calcd for $C_{20}H_{23}N_7O_2$: C, 61.05; H, 5.89; N, 24.92; Found: C, 59.83; H, 6.18; N, 23.93.

3.3.31 (E)-4,4'-(2-(2-phenylprop-1-en-1-yl)-[1,2,4]triazolo[1,5-a][1,3,5]triazine-5,7-diyl)

dimorpholine (**7b**);



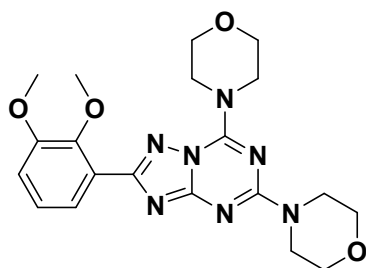
White solid, Yield: 82% ; ^1H NMR (400 MHz, CDCl_3 , δ ppm): 7.90 (s, 1H), 7.44-7.43 (m, 2H), 7.36 (t, $J = 7.56$ Hz, 2H), 7.27-7.24 (m, 1H), 4.30 (s, 4H), 3.84 (t, $J = 4.61$ Hz, 8H), 3.72 (t, $J = 4.80$ Hz, 4H), 2.32 (s, 3H) ; ^{13}C NMR (100 MHz, CDCl_3 , δ ppm): 166.25, 161.24, 159.50, 149.05, 137.19, 133.52, 129.65, 128.31, 127.40, 127.20, 66.79, 66.69, 47.10, 44.60, 14.51 ; MS m/z (ESI) calcd for $\text{C}_{21}\text{H}_{25}\text{N}_7\text{O}_2$ 407.21, found 408.0 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{21}\text{H}_{25}\text{N}_7\text{O}_2$: C, 61.90; H, 6.18; N, 24.06; Found: C, 59.25; H, 6.63; N, 22.84.

4. References:

1. El-faham A, Soliman SM, Ghabbour HA, Albericio F. Ultrasonic promoted synthesis of novel s -triazine-Schiff base derivatives ; molecular structure , spectroscopic studies and their preliminary anti-proliferative activities. *J Mol Struct.* 2016;1125:121-135. doi:10.1016/j.molstruc.2016.06.061

5. X-ray crystallographic structures of **2e**.

Method of crystallization: The compound **2e** was dissolved in isopropanol in a conical flask and heated till the solution was reduced to half. The hot solution was then closed using aluminium foil and kept in dark for slow evaporation. The slow evaporation of the solutions leads to the formation of crystals which were further taken for the X-ray crystallographic studies.



Datablock 20zs_rk_a2_aldoi_ipa_0m - ellipsoid plot

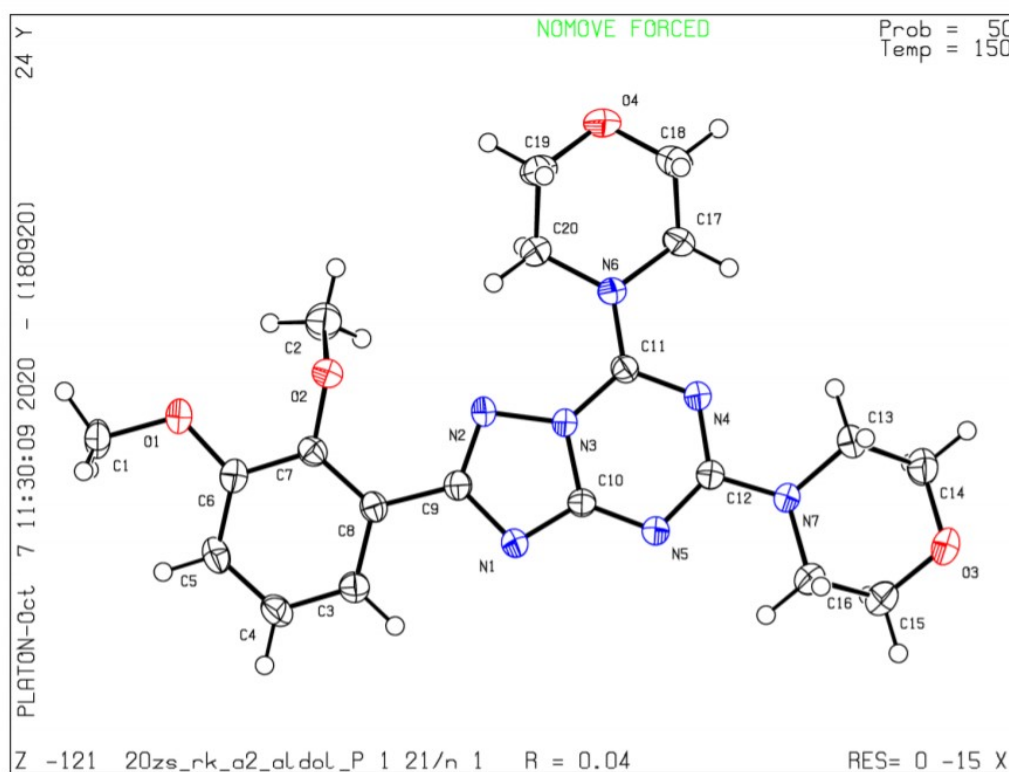
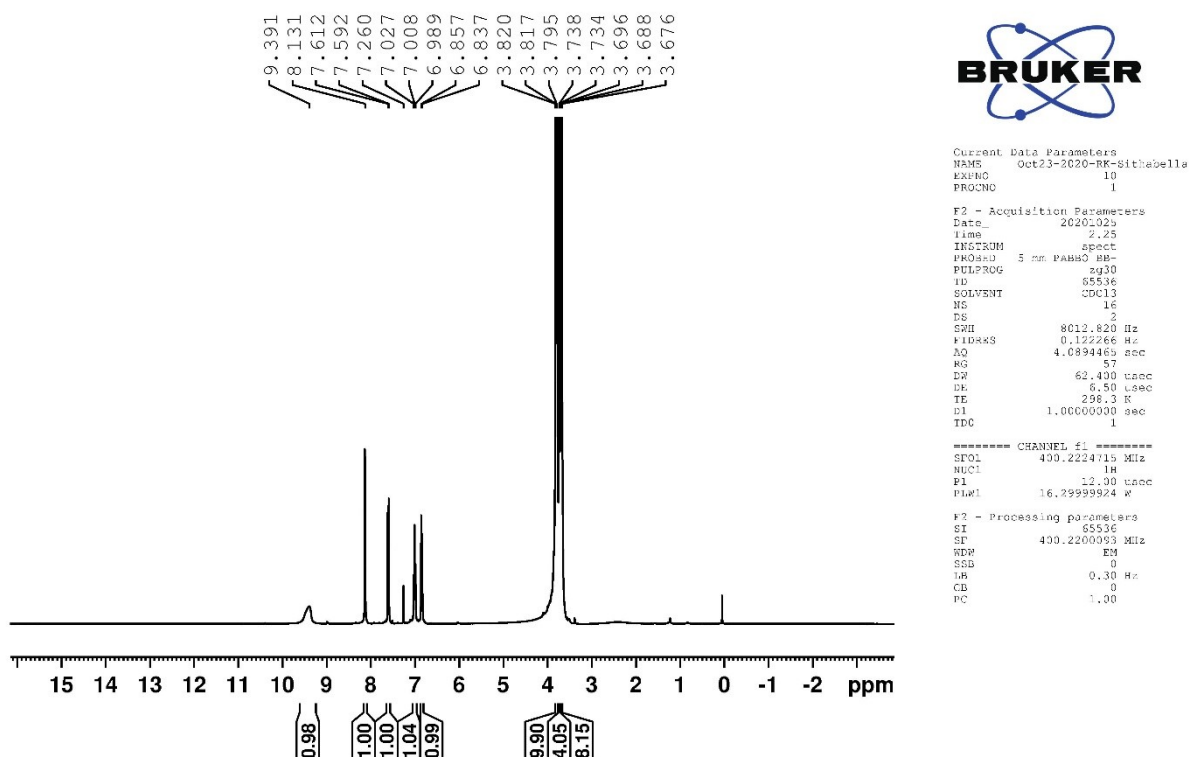


Figure S2. Crystal structure of **2e** (CCDC No. 2053179)

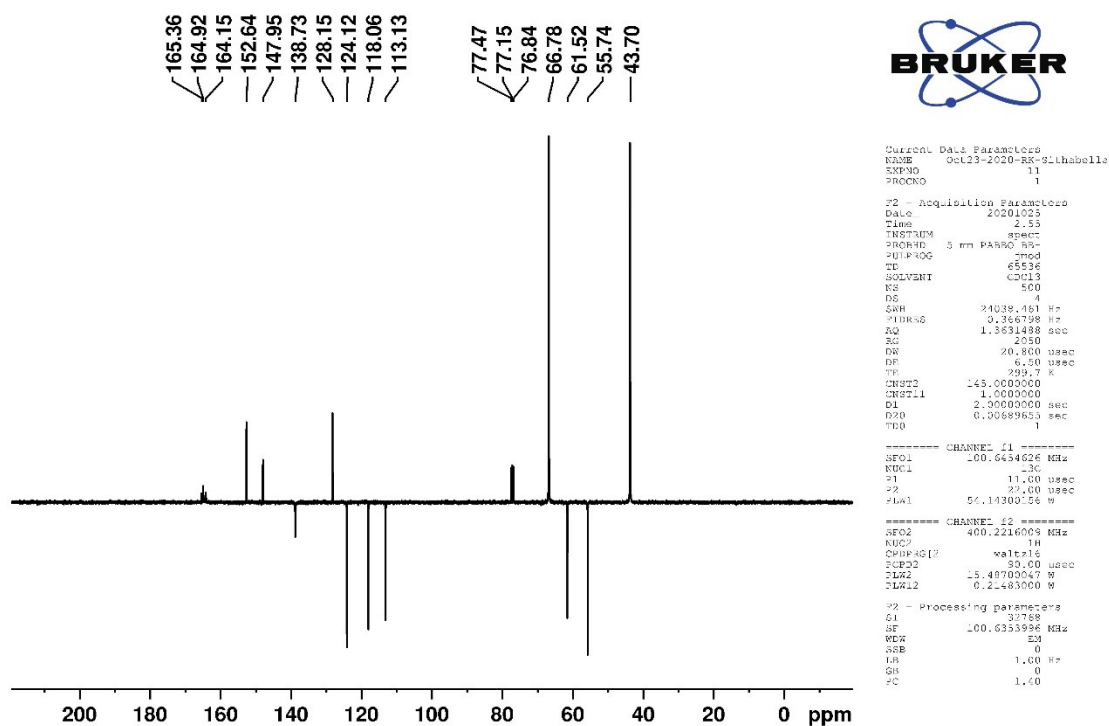
Table S1: X-ray crystallographic data and structure refinement.

Identification code	2e
Empirical formula	C ₂₀ H ₂₅ N ₇ O ₄
Formula weight	427.47
Temperature/K	150.0
Crystal system	monoclinic
Space group	P2 ₁ /n
a/Å	11.320(3)
b/Å	12.339(3)
c/Å	15.403(4)
$\alpha/^\circ$	90
$\beta/^\circ$	109.964(14)
$\gamma/^\circ$	90
Volume/Å ³	2022.3(9)
Z	4
$\rho_{\text{calc}}/\text{g}/\text{cm}^3$	1.404
μ/mm^{-1}	0.101
F(000)	904.0
Crystal size/mm ³	0.23 × 0.17 × 0.08
Radiation	MoK α (λ = 0.71073)
2 Θ range for data collection/ $^\circ$	3.9 to 52.346
Index ranges	-8 ≤ h ≤ 14, -15 ≤ k ≤ 12, -19 ≤ l ≤ 19
Reflections collected	13314
Independent reflections	3995 [R_{int} = 0.0206, R_{sigma} = 0.0228]
Data/restraints/parameters	3995/0/282
Goodness-of-fit on F ²	1.031
Final R indexes [$I \geq 2\sigma(I)$]	R_1 = 0.0384, wR_2 = 0.0979
Final R indexes [all data]	R_1 = 0.0507, wR_2 = 0.1064
Largest diff. peak/hole / e Å ⁻³	0.33/-0.21

8. NMR Spectra: ^1H NMR, ^{13}C NMR and Mass spectra

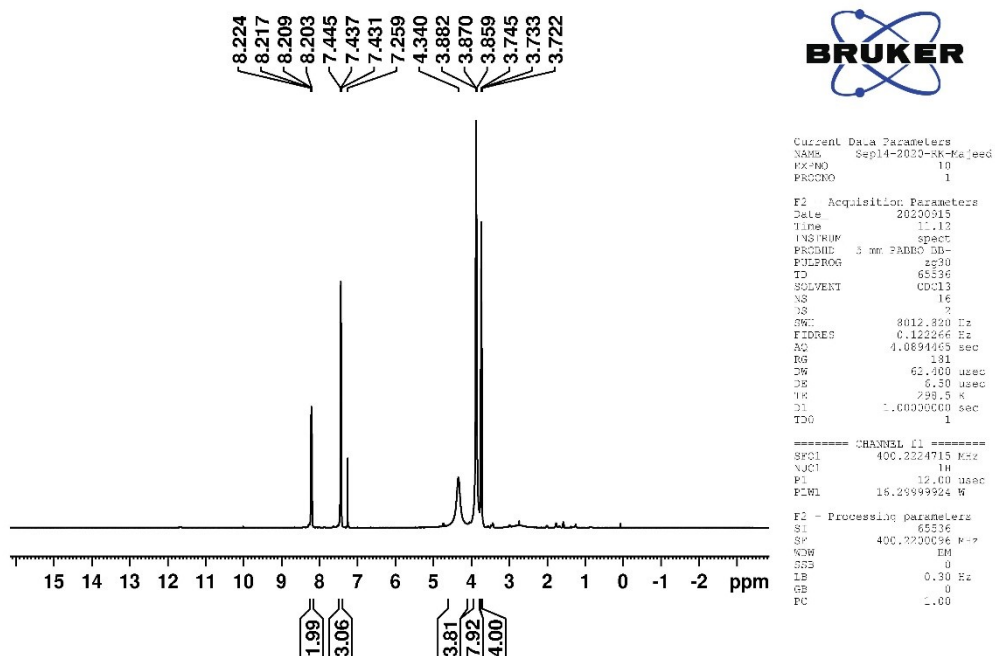
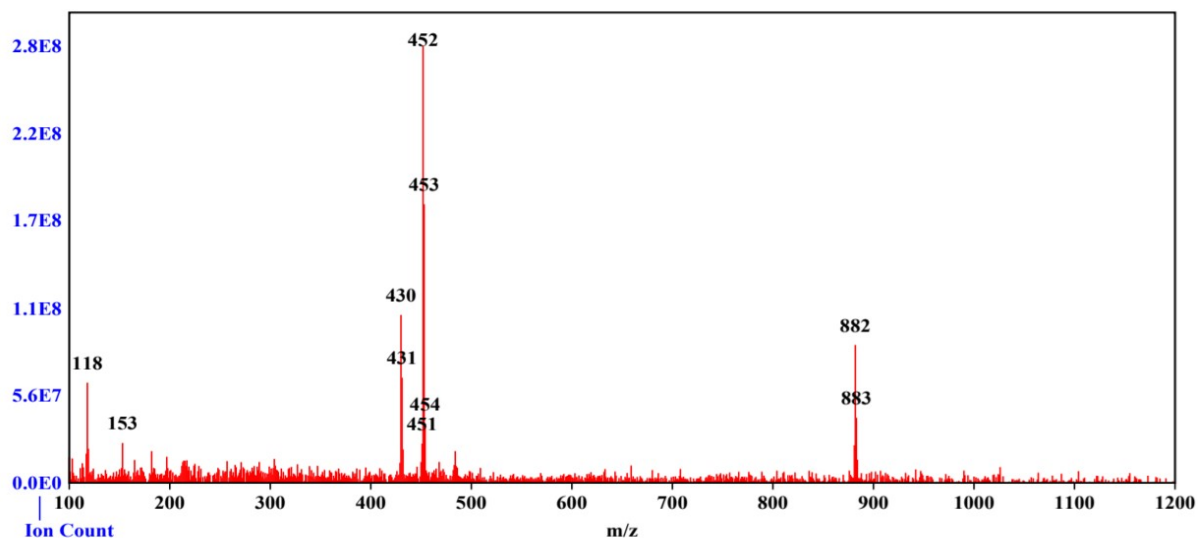


^1H NMR spectrum for **schiff base** (CDCl_3 , 400 MHz)

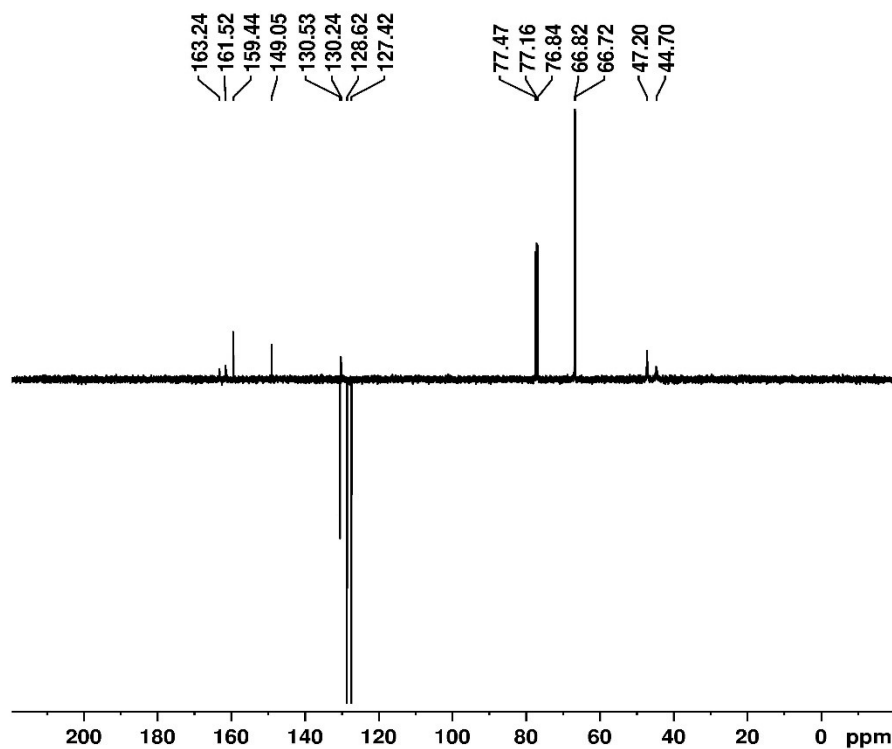


^{13}C NMR spectrum for **schiff base** (CDCl_3 , 100 MHz)

Spectrum Name: SB-15
 Start Ion: 100
 End Ion: 1200
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



^1H NMR spectrum for compound **2a** (CDCl_3 , 400 MHz)



```

Current Data Parameters
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EXPNO     11
PROCNO    1

F2 - Acquisition Parameters
Date_     20200915
Time      11:48
INSTRUM   spect
PROBHD    5 mm FAREO BB-
PULPROG   prod
NUC1       13C
SOLVENT    CDCl3
DE         600
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3531488 sec
RG         2055
DA         20.800 usec
DE         6.50 usec
TE         300.2 K
CST2       143.0000000
CST11      1.0000000
D1         2.00000000 sec
D2C        0.00689655 sec
SFO        100.626156 M

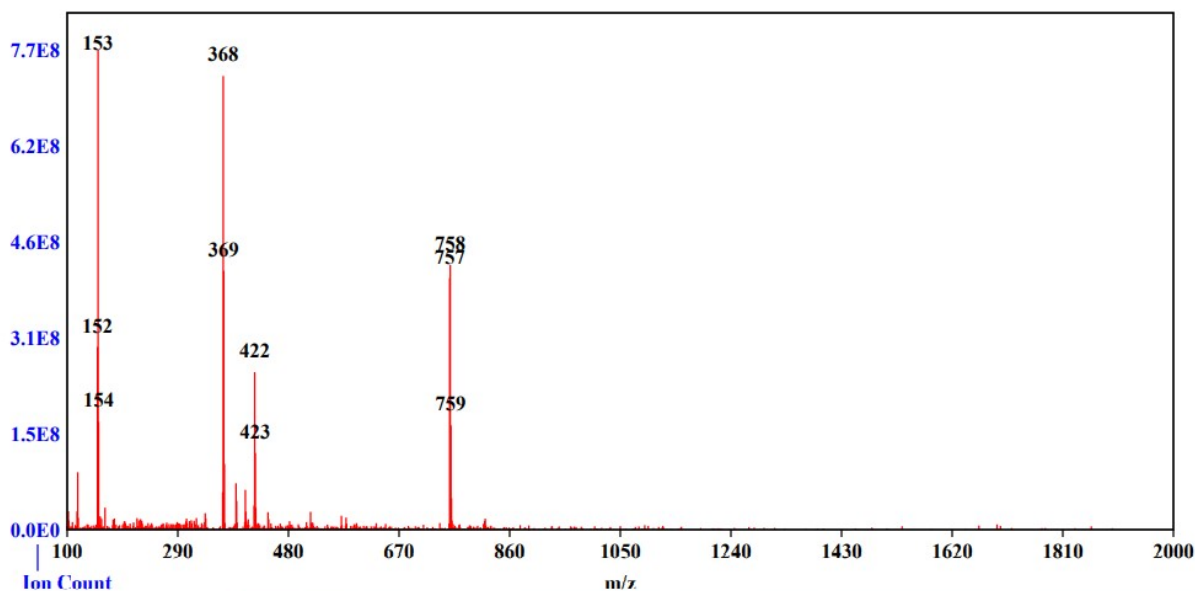
===== CHANNEL f1 =====
SFO1       100.626156 MHz
NUC1        13C
P1         11.00 usec
P2         22.00 usec
PLW1       36.14300156 W

===== CHANNEL f2 =====
SFO2       100.626156 MHz
NUC2        1H
CDEPSC12   waltz16
PCPD2      90.00 usec
PLW2       15.48700047 W
PLW12      0.21783000 W

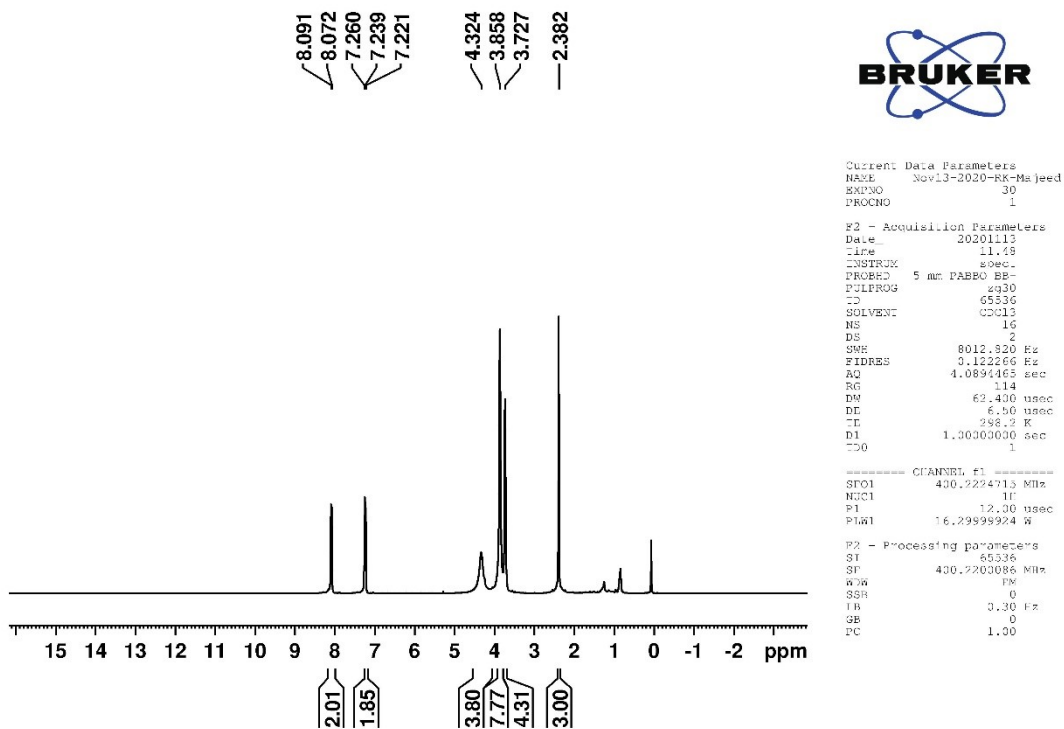
F2 - Processing parameters
SI         32768
SF         100.626156 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

¹³C NMR spectrum for compound **2a** (CDCl₃, 100 MHz)

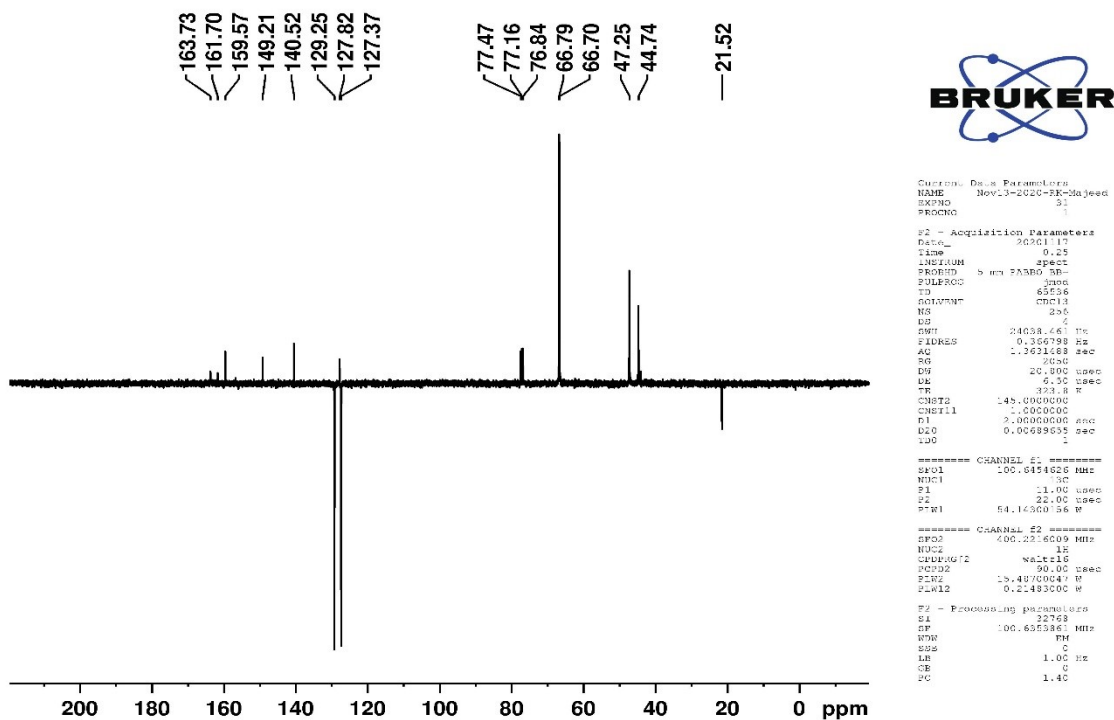
Spectrum Name: DIM-01
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



Mass spectrum for **2a**

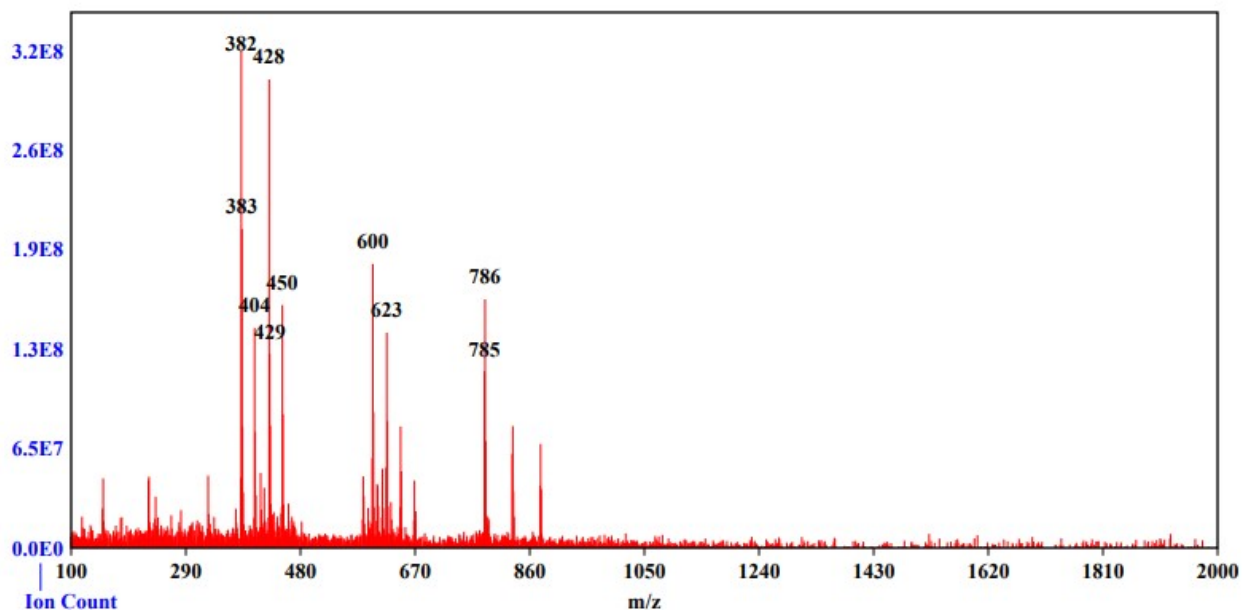


¹H NMR spectrum for compound **2b** (CDCl₃, 400 MHz)

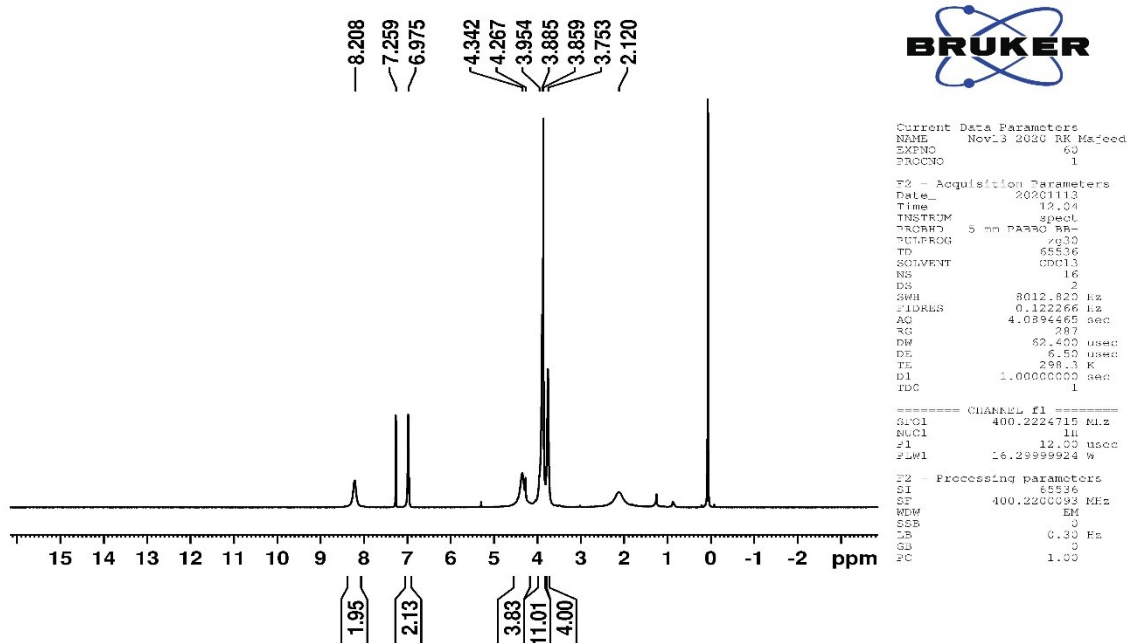


¹³C NMR spectrum for compound **2b** (CDCl₃, 100 MHz)

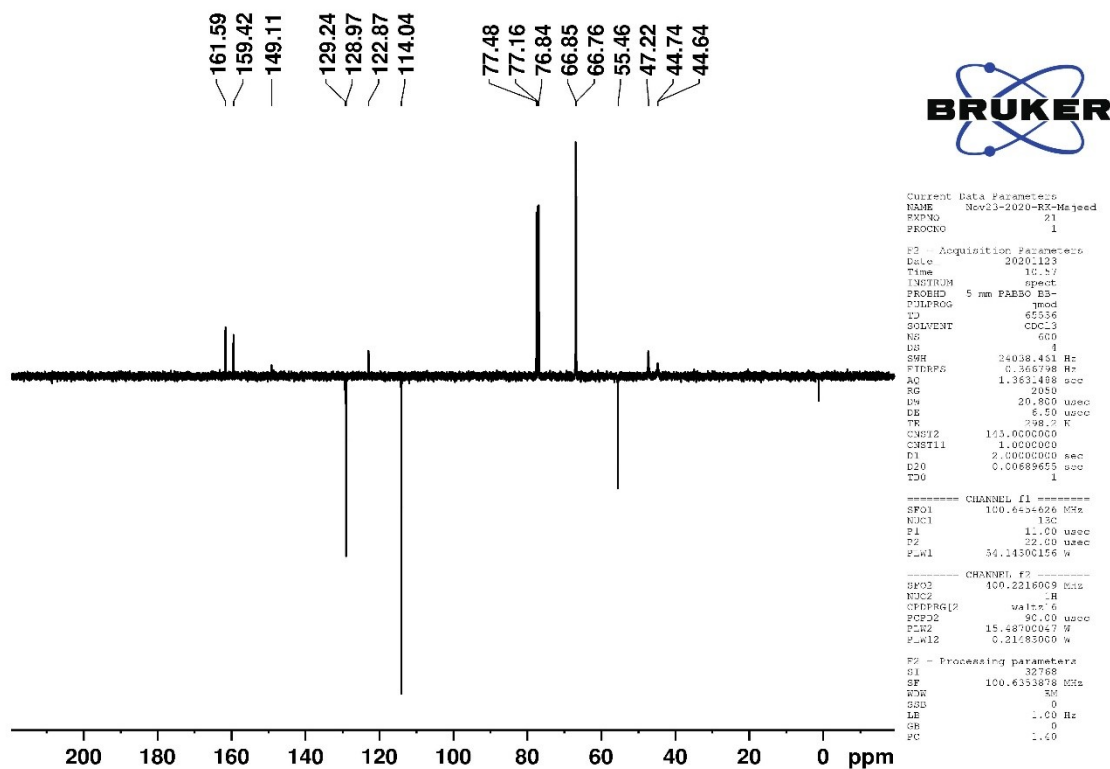
Spectrum Name: dim16
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



Mass spectrum for **2b**

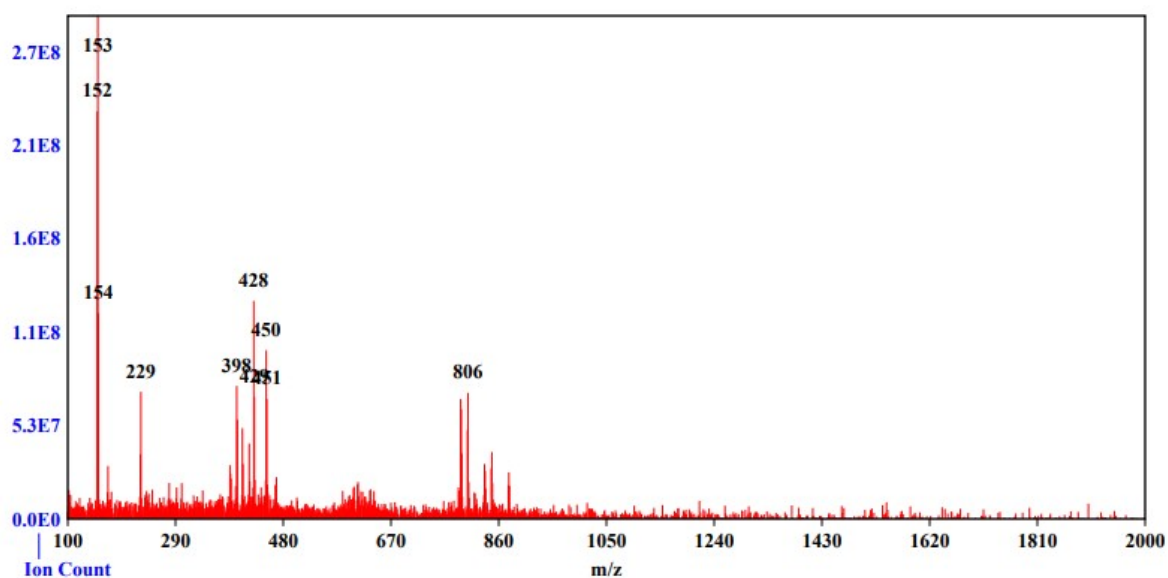


¹H NMR spectrum for compound **2c** (CDCl₃, 400 MHz)

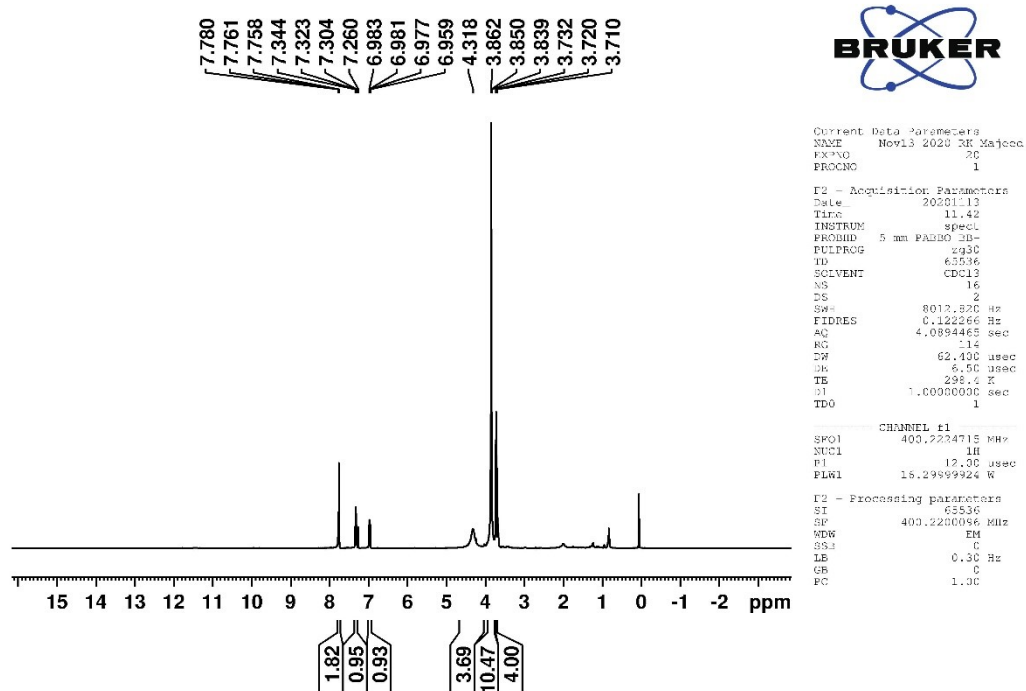


¹³C NMR spectrum for compound **2c** (CDCl₃, 100 MHz)

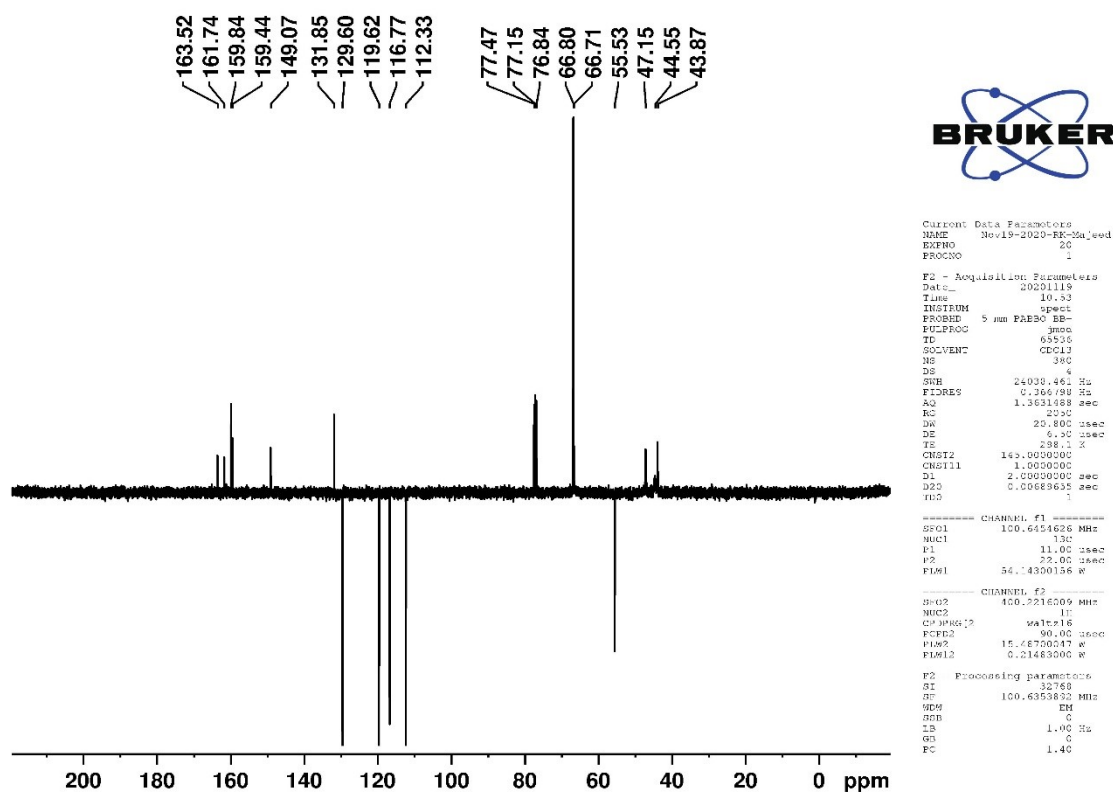
Spectrum Name: dim19
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



Mass spectrum for **2c**

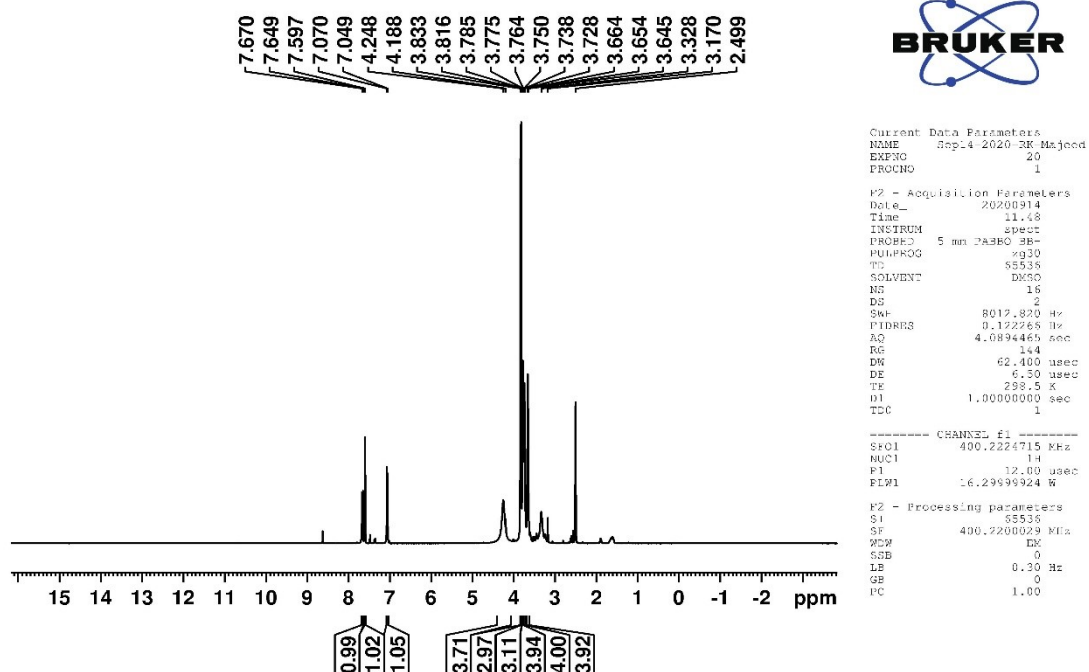
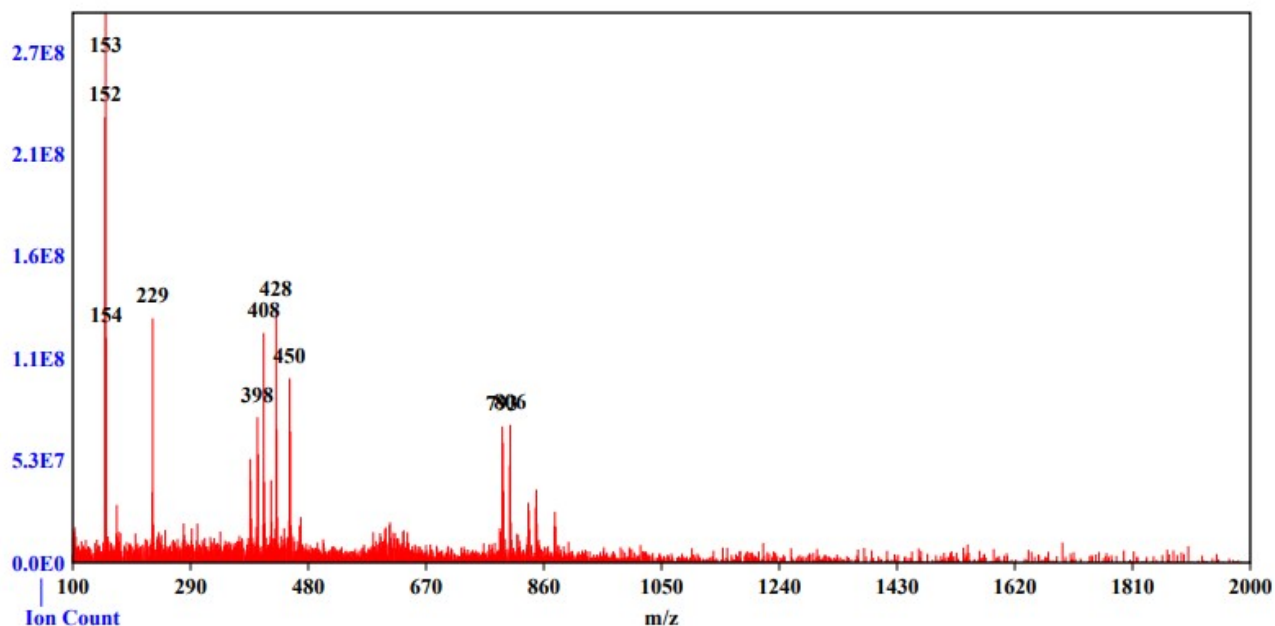


¹H NMR spectrum for compound **2d** (CDCl₃, 400 MHz)



¹³C NMR spectrum for compound **2d** (CDCl₃, 100 MHz)

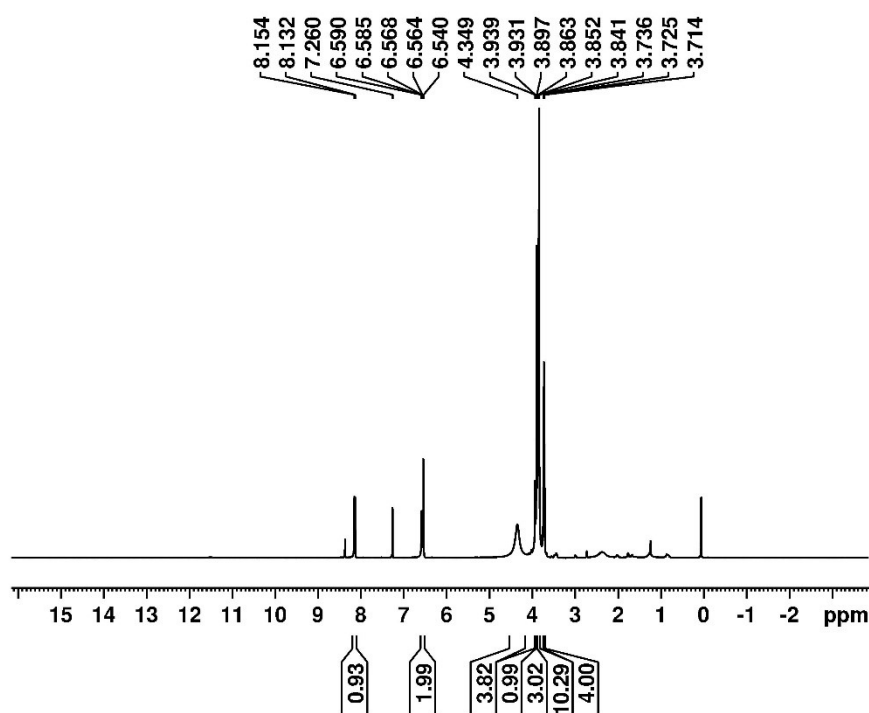
Spectrum Name: dim20
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



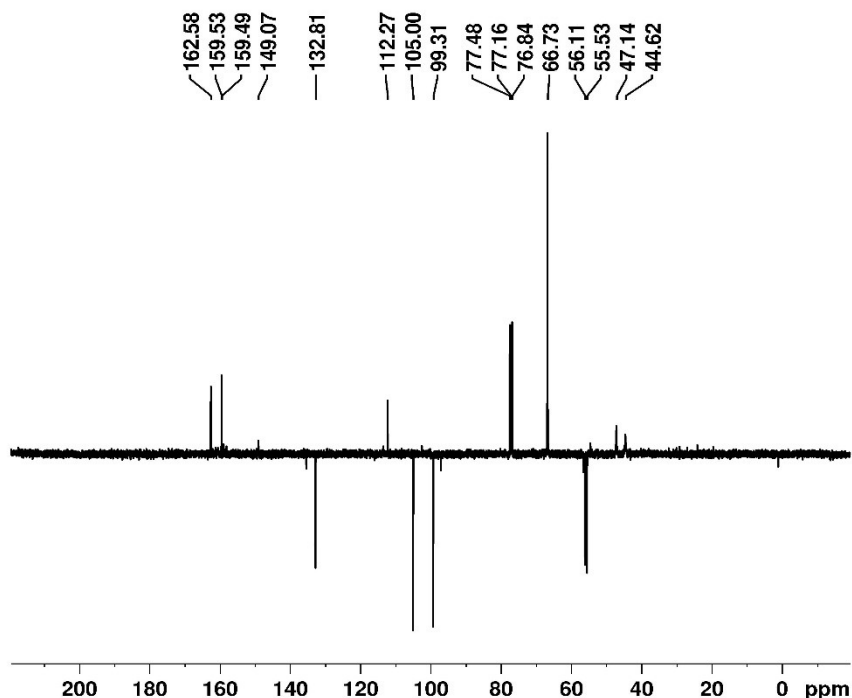


Mass spectrum showing relative intensity (Y-axis, 0.0E0 to 1.0E9) versus m/z (X-axis, 100 to 2000). The base peak is at m/z 428. Other labeled peaks include m/z 429, 450, 461, 477, 482, 669, and 878.

S29

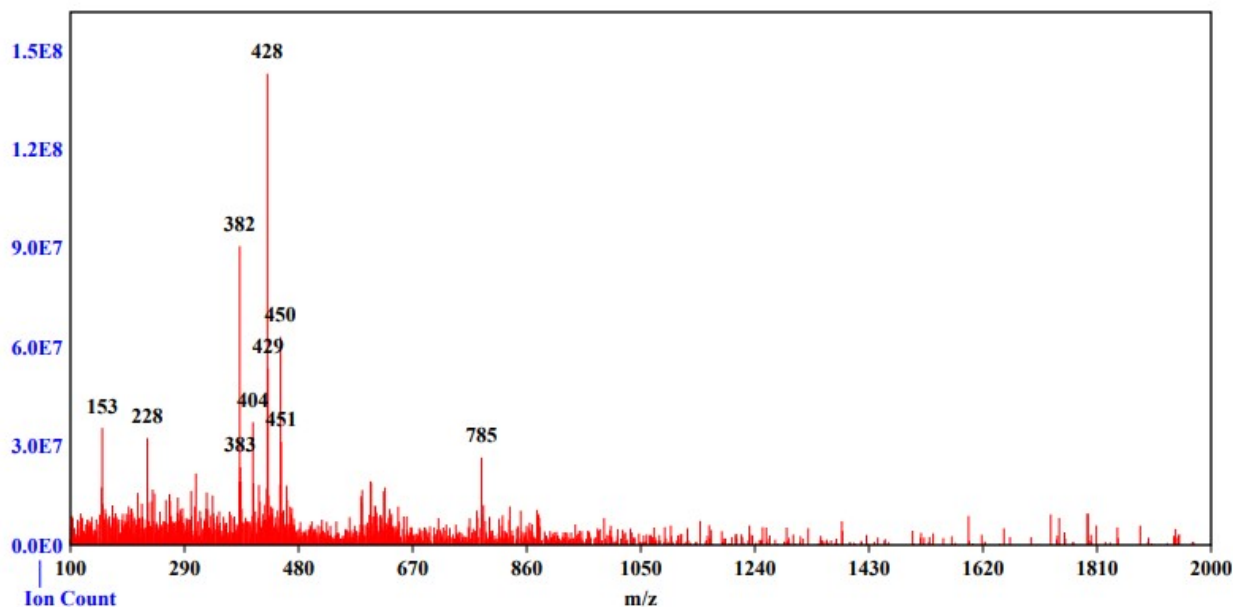


¹H NMR spectrum for compound **2f** (CDCl₃, 400 MHz)

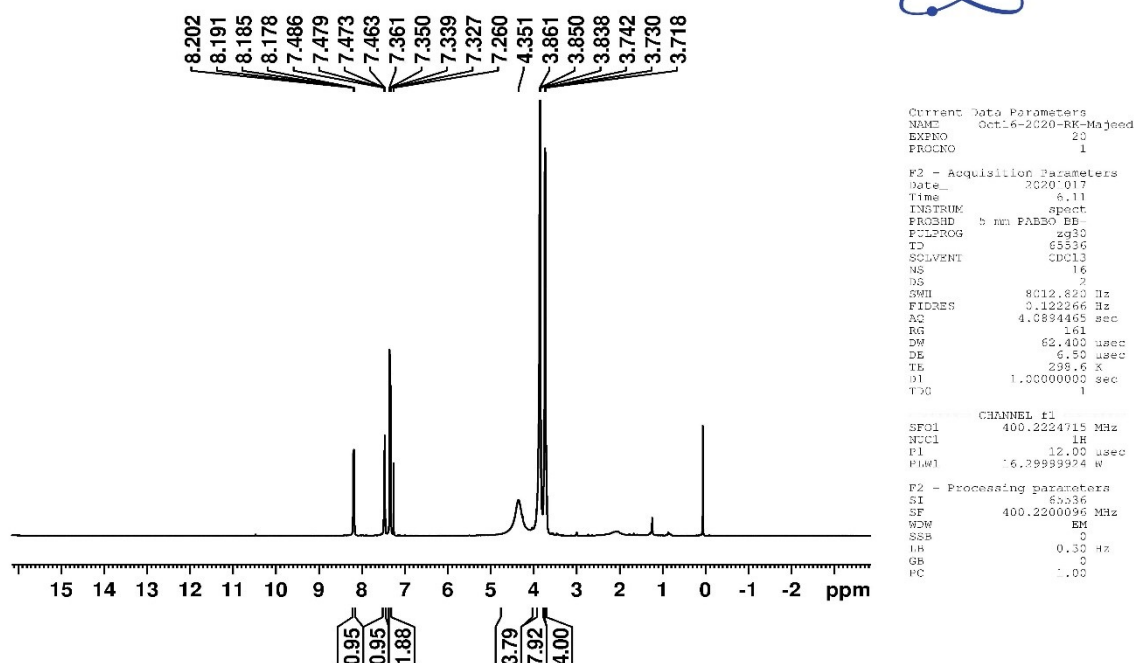


¹³C NMR spectrum for compound **2f** (CDCl₃, 100 MHz)

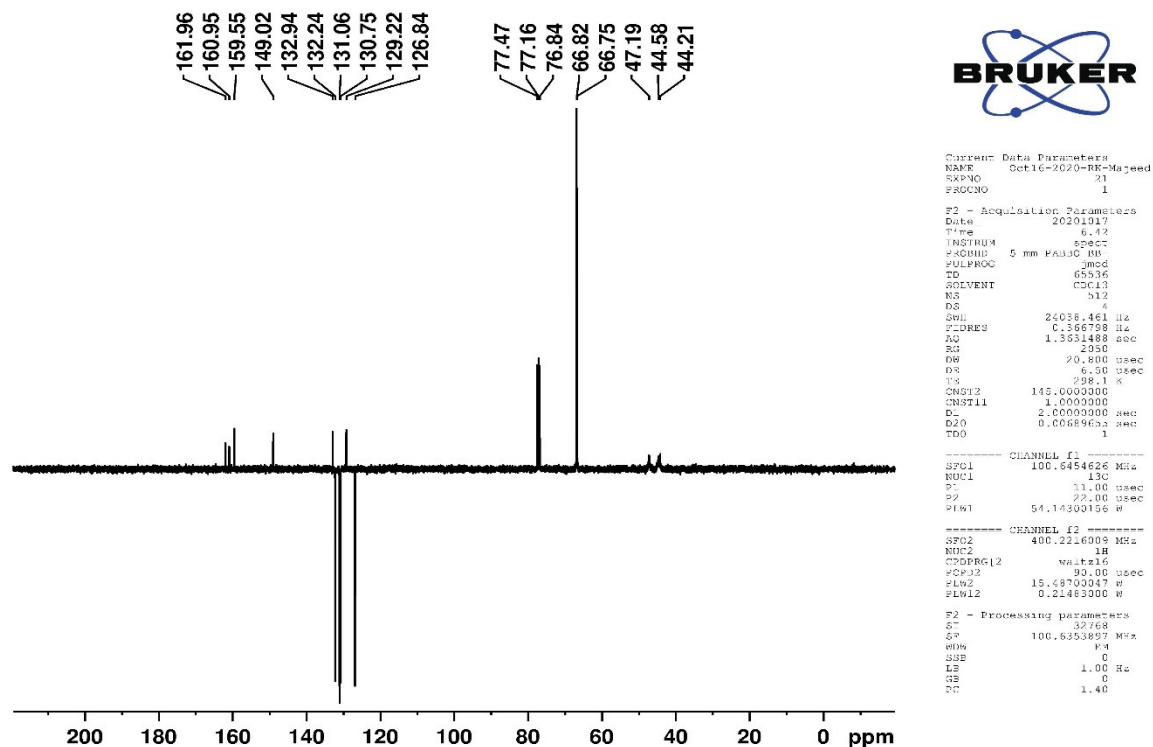
Spectrum Name: dim17
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



Mass spectrum for **2f**

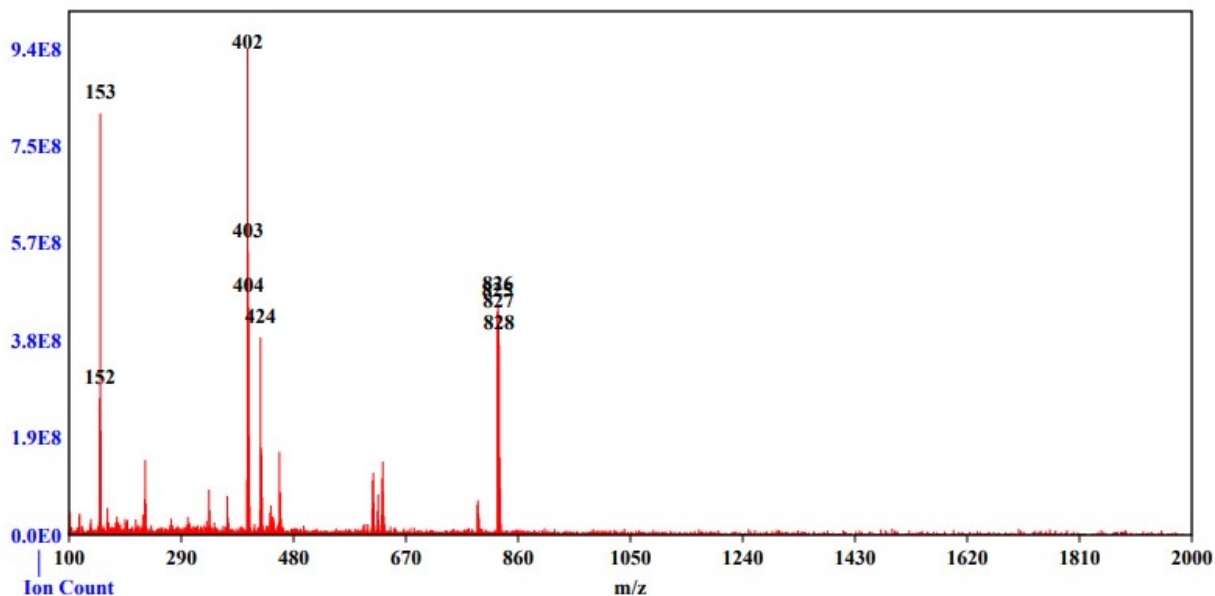


¹H NMR spectrum for compound **2g** (CDCl₃, 400 MHz)

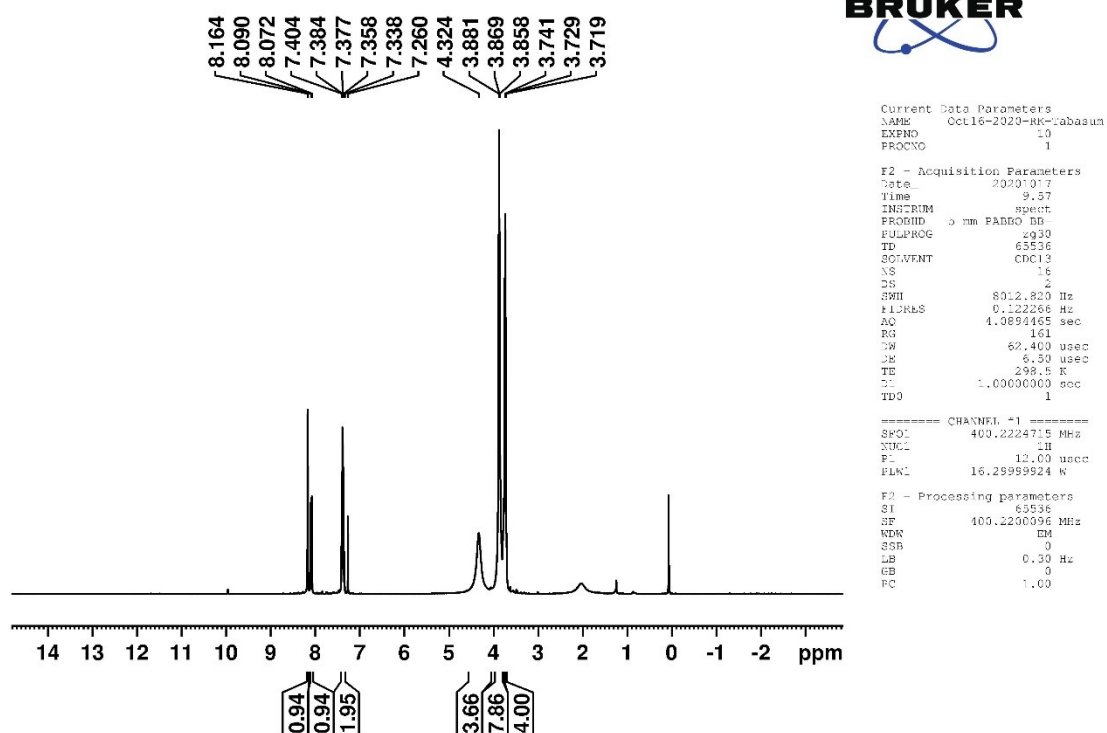


¹³C NMR spectrum for compound **2g** (CDCl₃, 100 MHz)

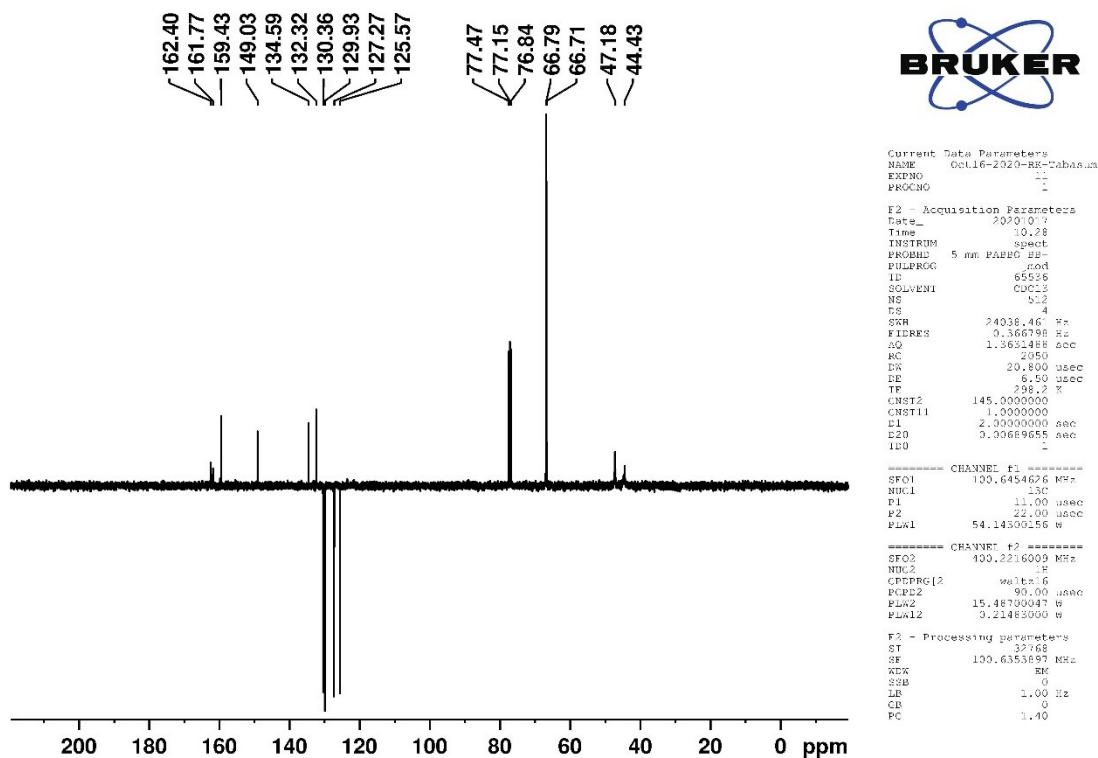
Spectrum Name: DIM-03
Start Ion: 100
End Ion: 2000
Source: ESI + 3.5kV 350C
Capillary: 150V 300C Offset: 25V Span: 0V



Mass spectrum for **2g**

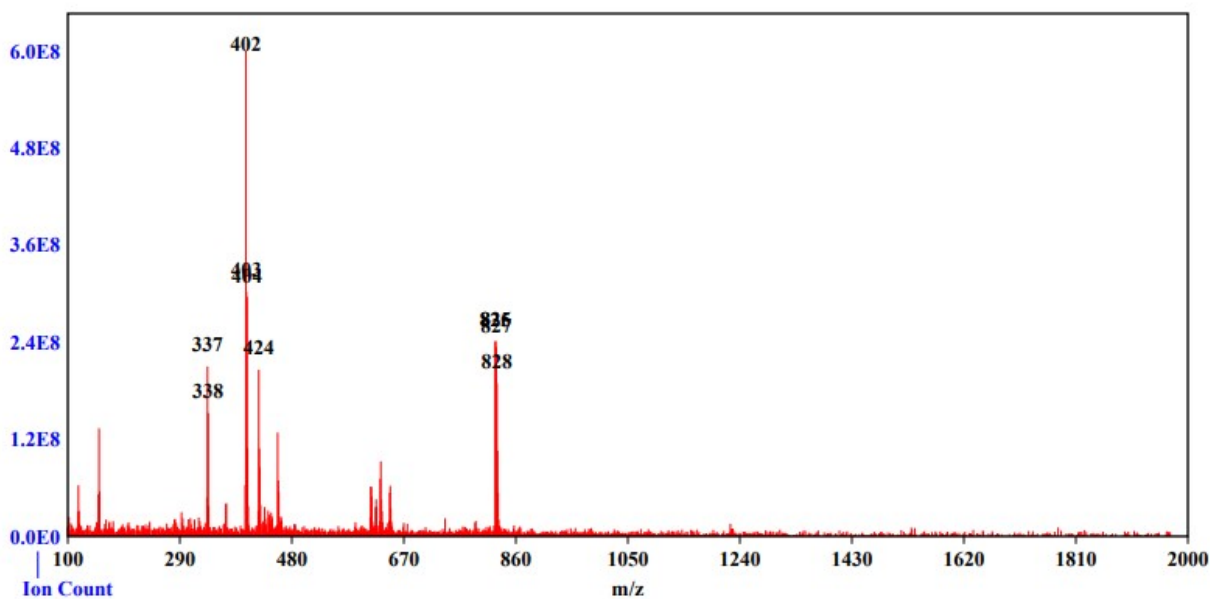


^1H NMR spectrum for compound **2h** (CDCl_3 , 400 MHz)

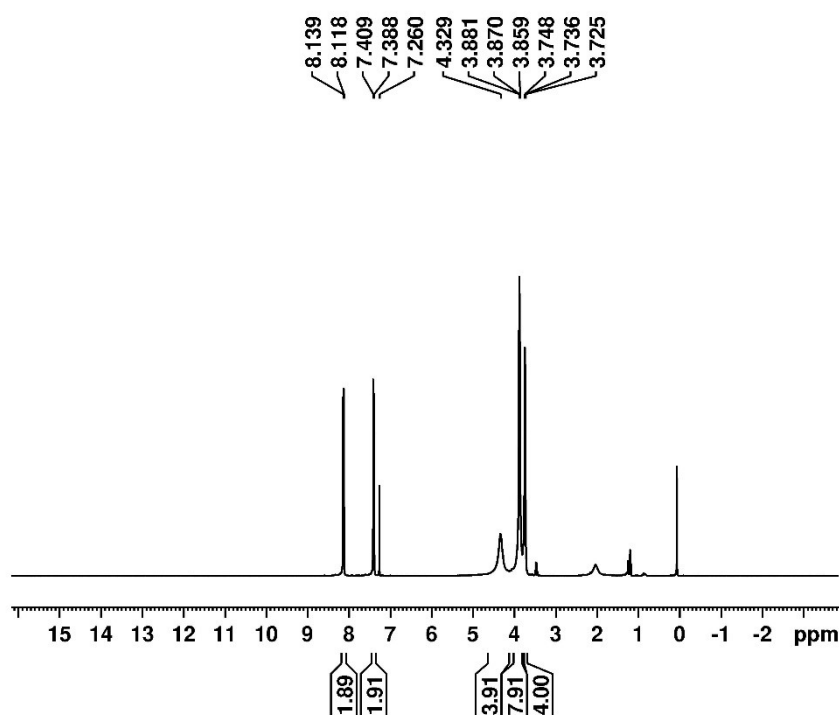


^{13}C NMR spectrum for compound **2h** (CDCl_3 , 100 MHz)

Spectrum Name: DIM-02
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



Mass spectrum for 2h

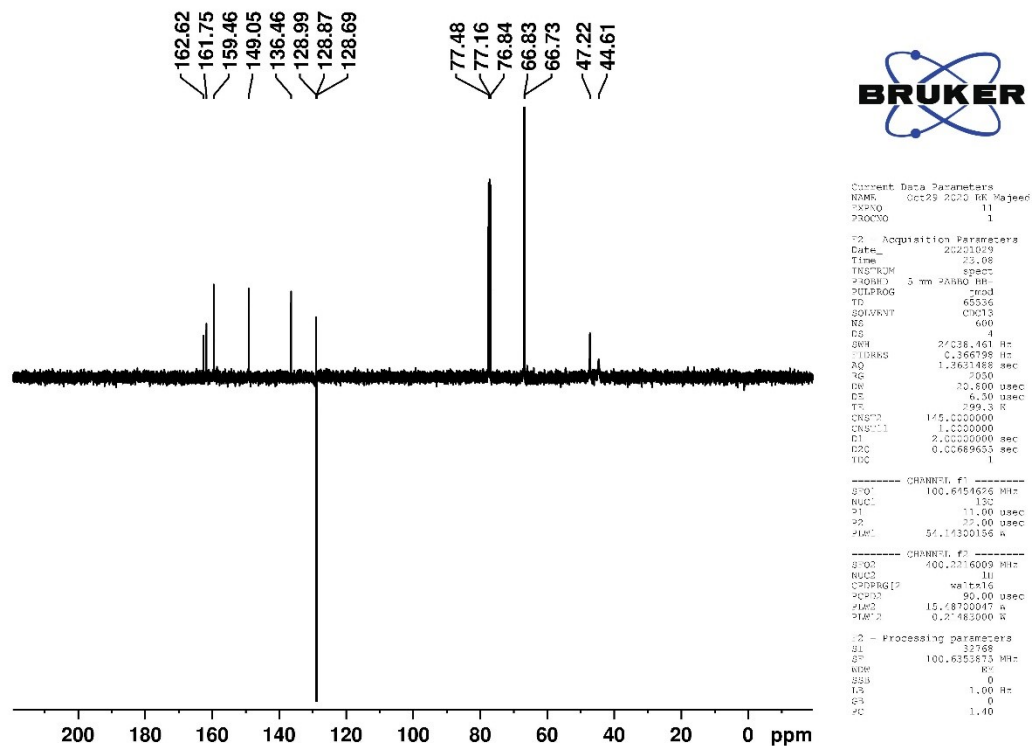


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 SOLVENT CDCl3
 NS 16
 DS 2
 SWE 8012.620 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 161
 DR 62.400 usec
 DE 6.50 usec
 TE 298.4 K
 D1 1.0000000 sec
 TDO 1

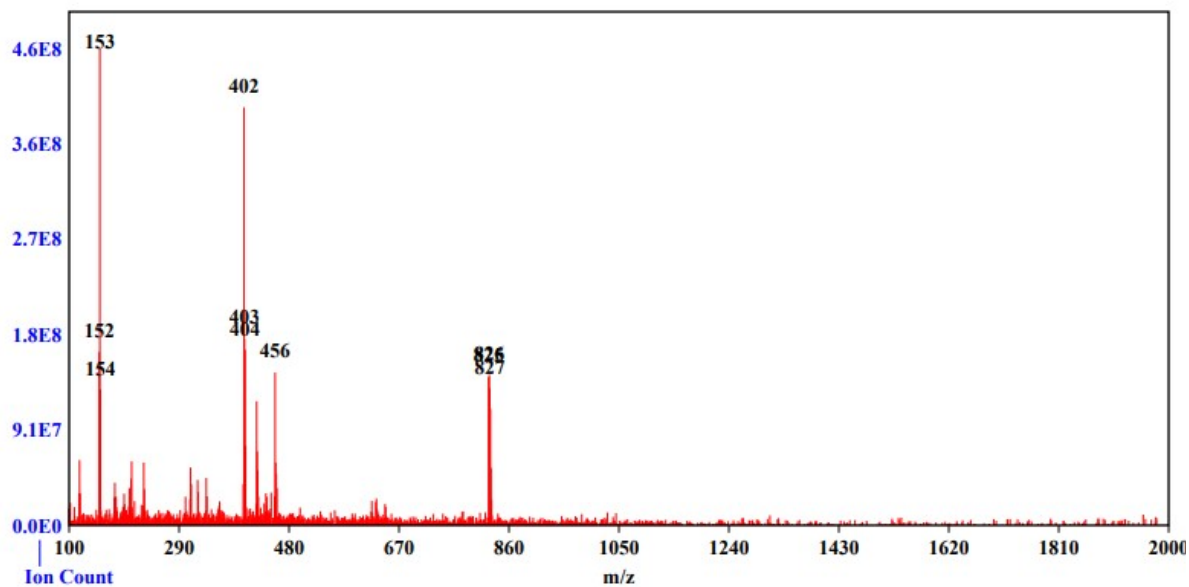
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F2 - Processing parameters
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 GB 0
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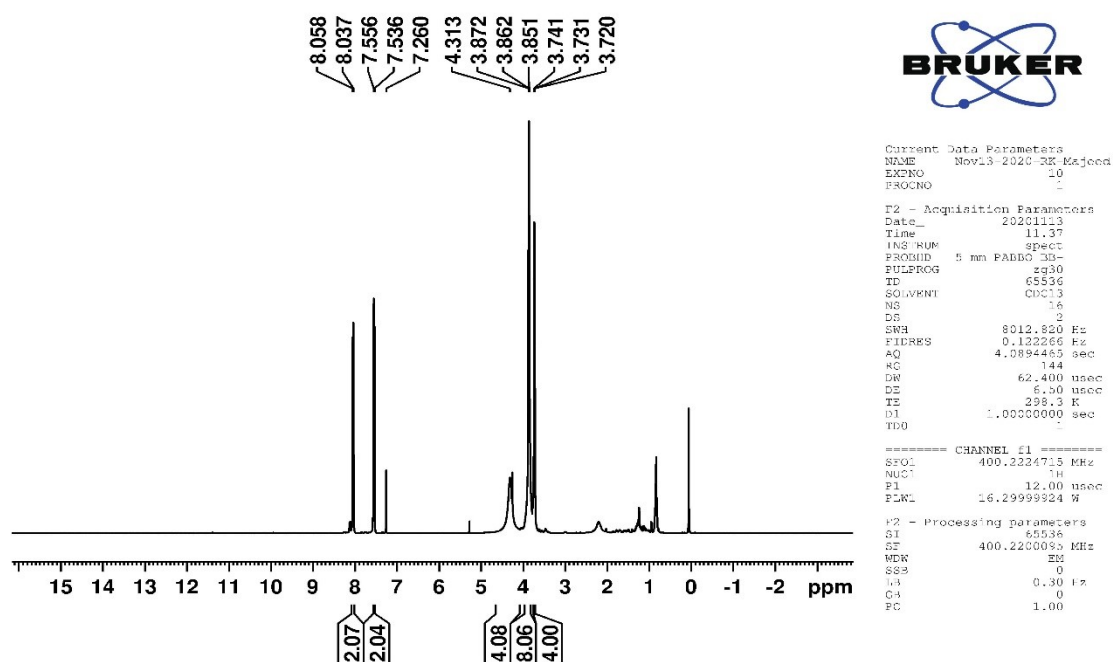


¹³C NMR spectrum for compound **2i** (CDCl₃, 100 MHz)

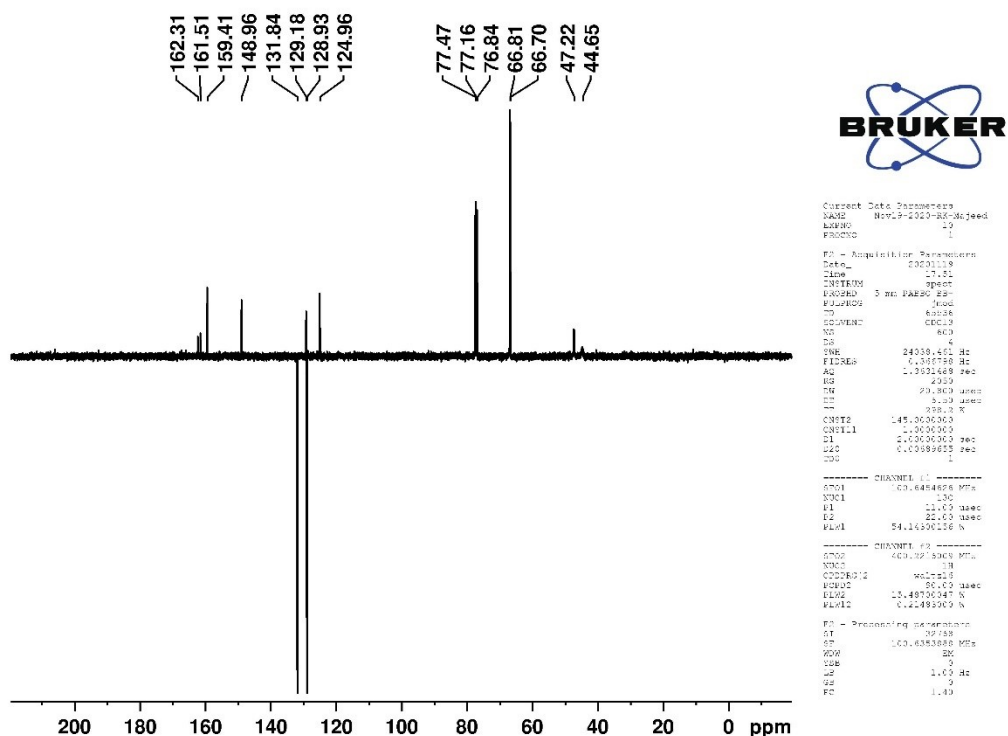
Spectrum Name: DIM-04
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



Mass spectrum for **2i**

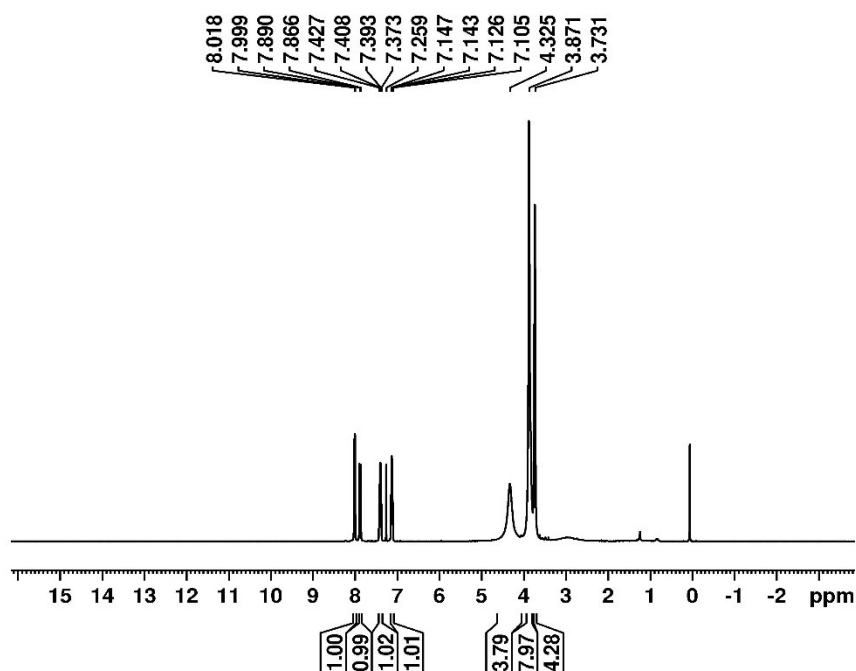
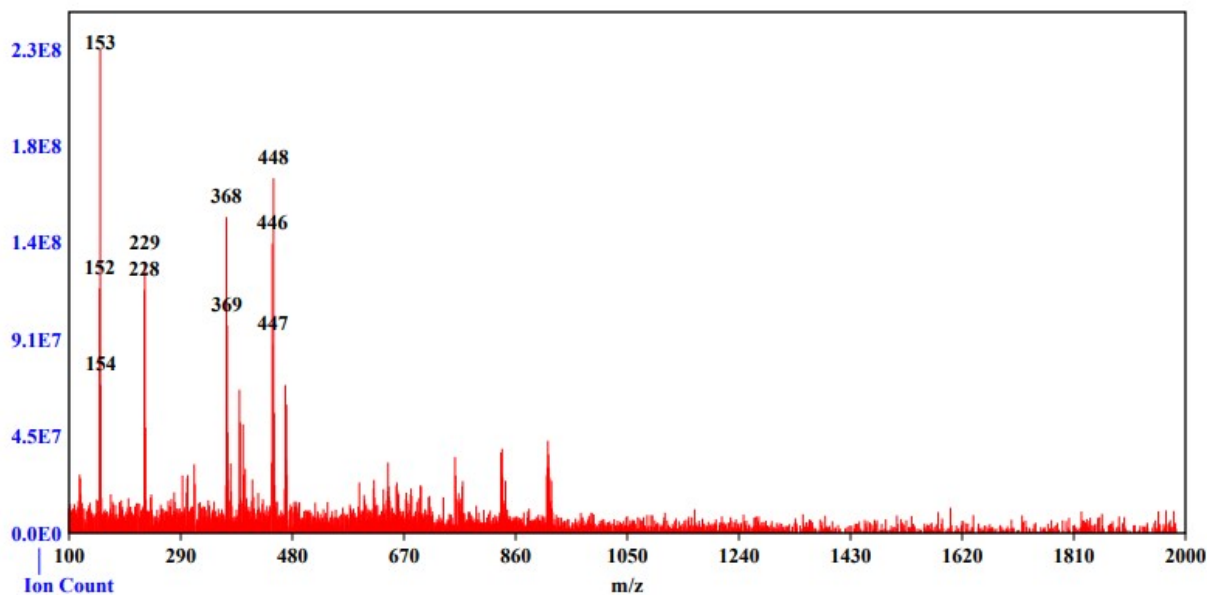


¹H NMR spectrum for compound **2j** (CDCl₃, 400 MHz)



¹³C NMR spectrum for compound **2j** (CDCl₃, 100 MHz)

Spectrum Name: dim11
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V

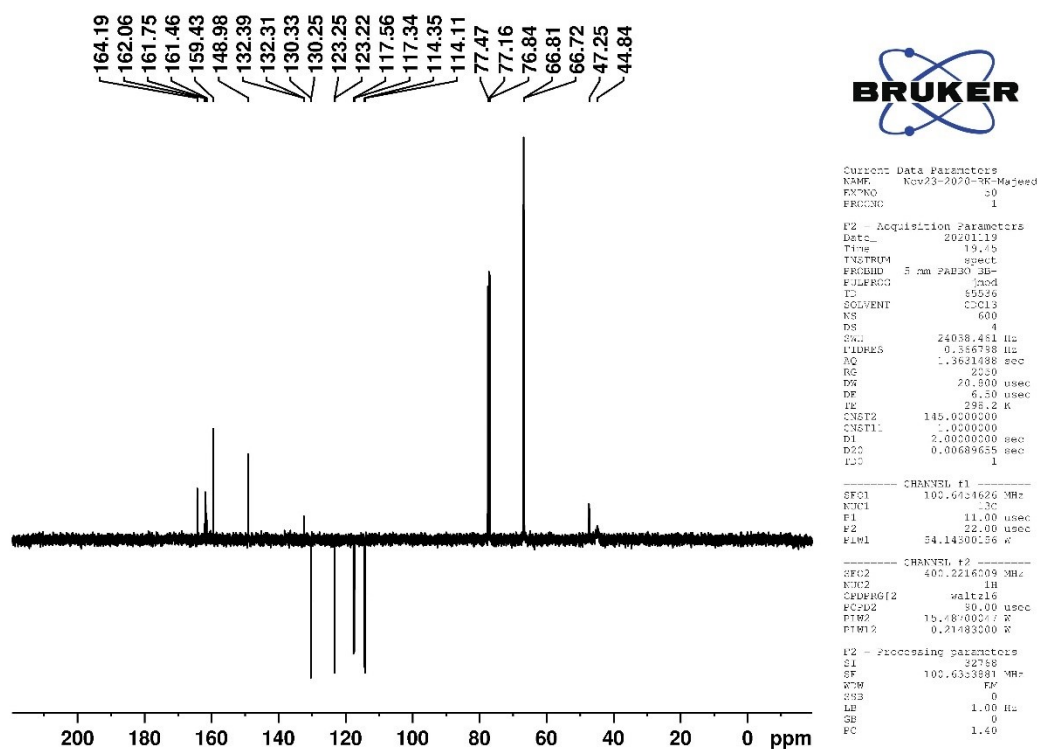


Current Data Parameters
 NAME Nov23-2020-RK-Majeed
 KKXNO 10
 PROCNO 1

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 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 181
 DW 62.400 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.00000000 sec
 TDC 1

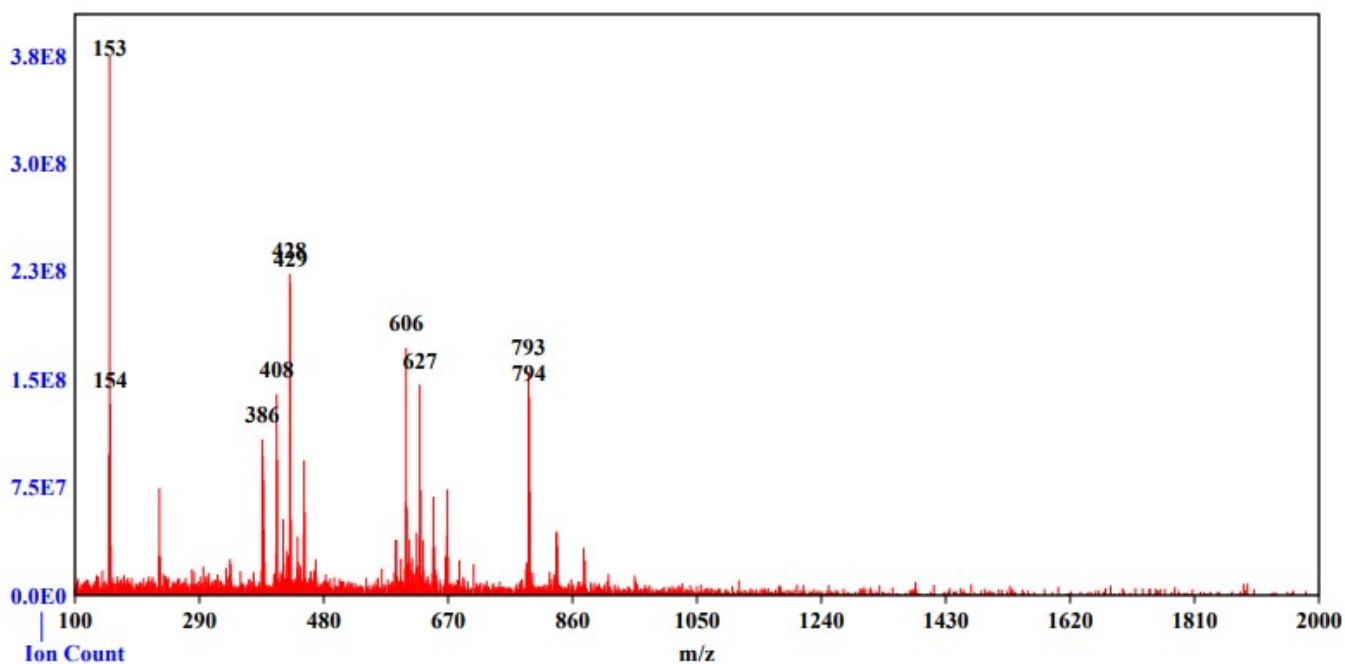
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 NUC1 1H
 P1 12.00 usec
 PLW1 16.23999924 W

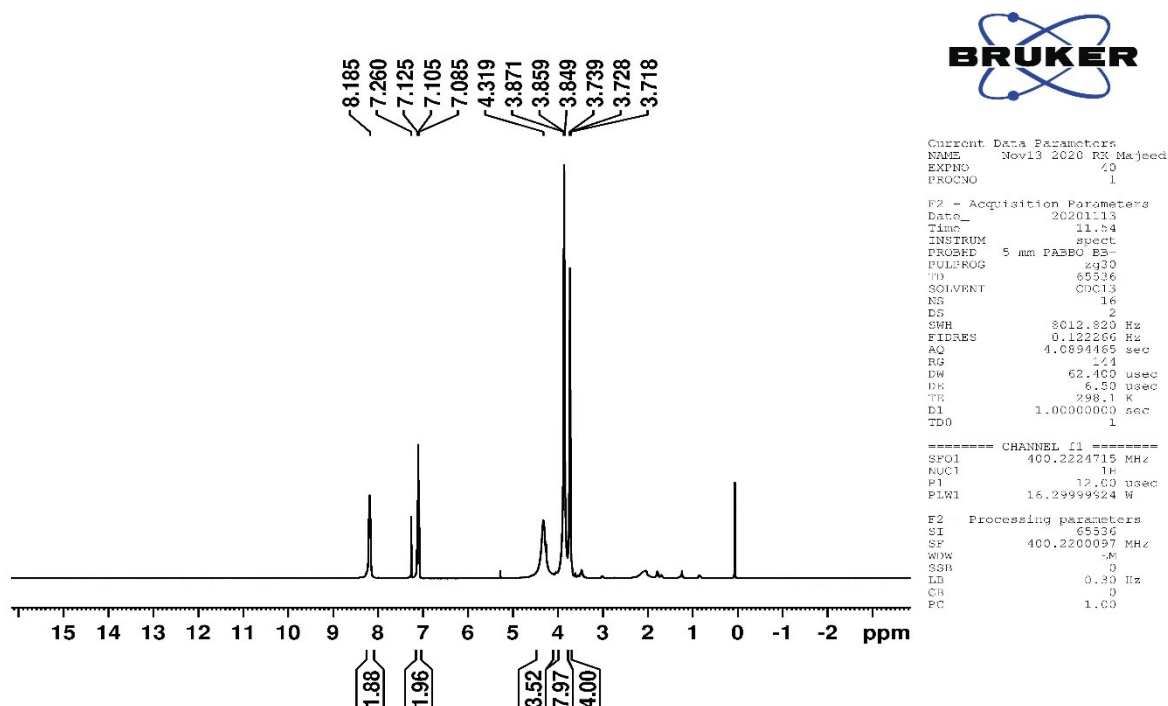
F2 - Processing parameters
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 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



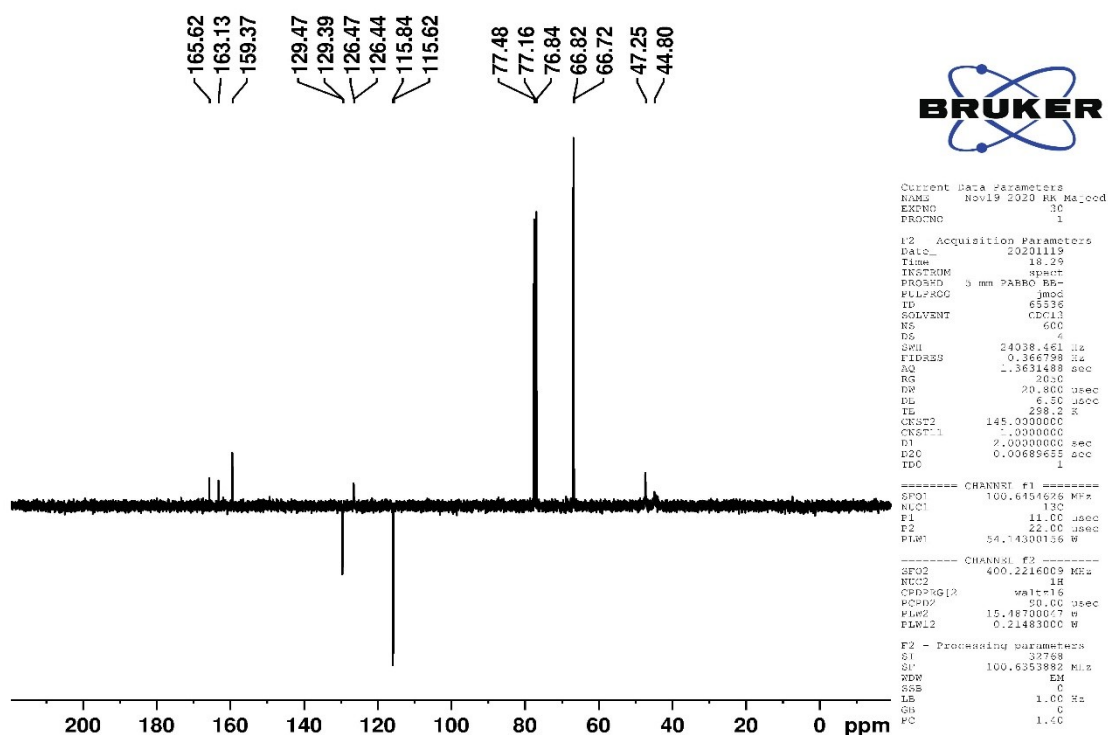
¹³C NMR spectrum for compound **2k** (CDCl₃, 100 MHz)

Spectrum Name: dim18
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



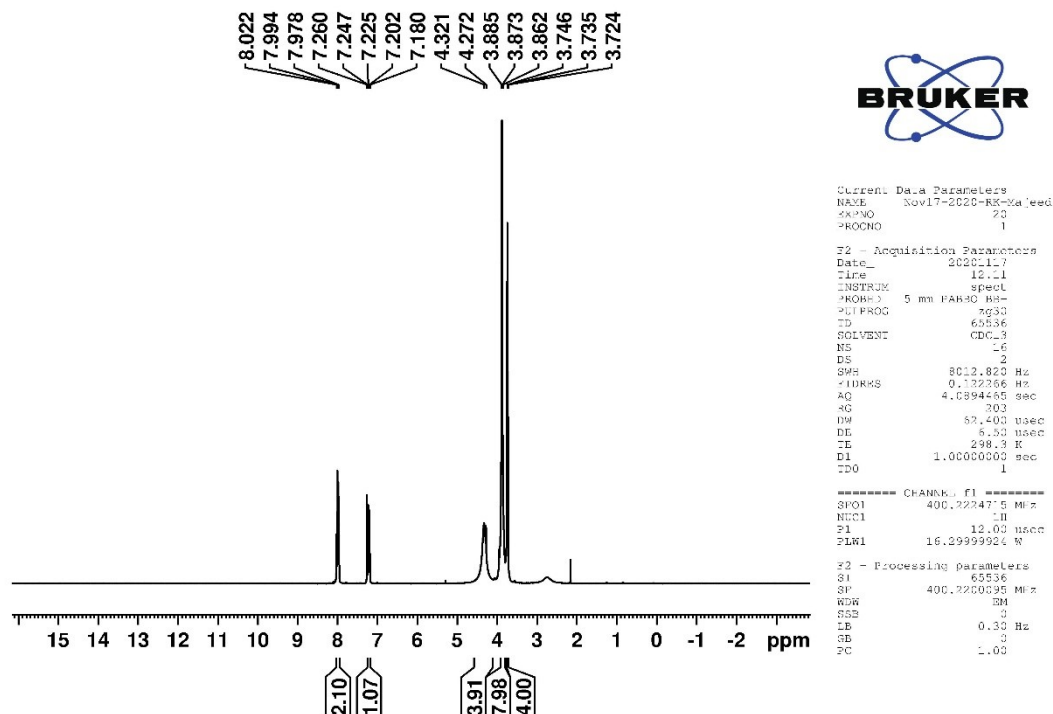
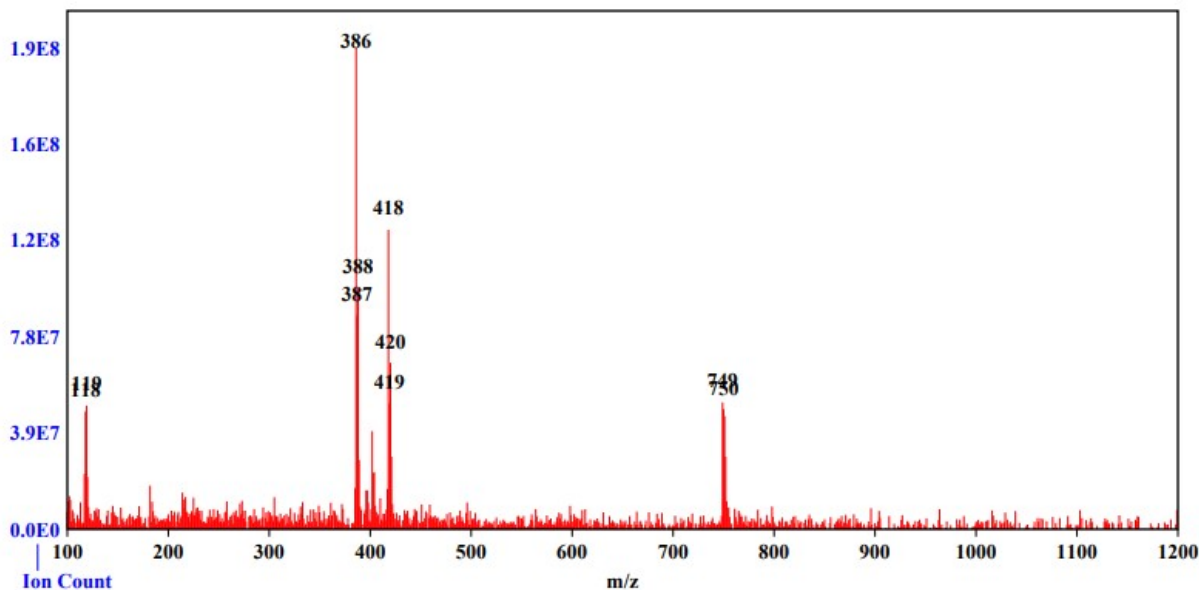


^1H NMR spectrum for compound **2l** (CDCl_3 , 400 MHz)



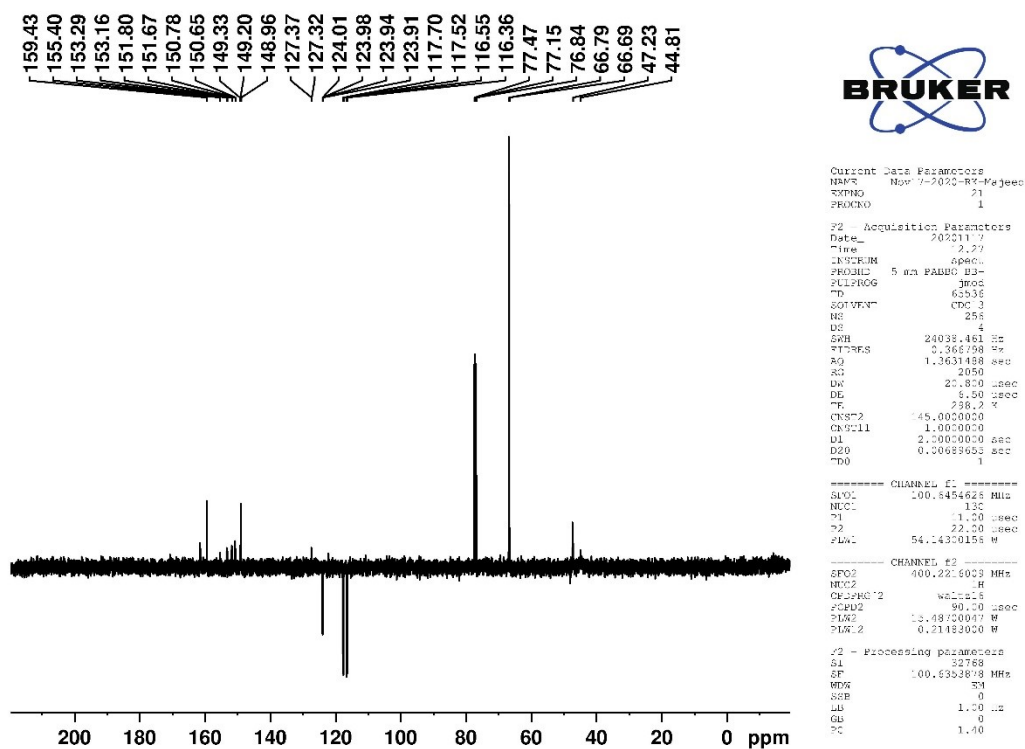
^{13}C NMR spectrum for compound **2l** (CDCl_3 , 100 MHz)

Start Ion: 100
End Ion: 1200
Source: ESI + 3.5kV 350C
Capillary: 150V 300C Offset: 25V Span: 0V



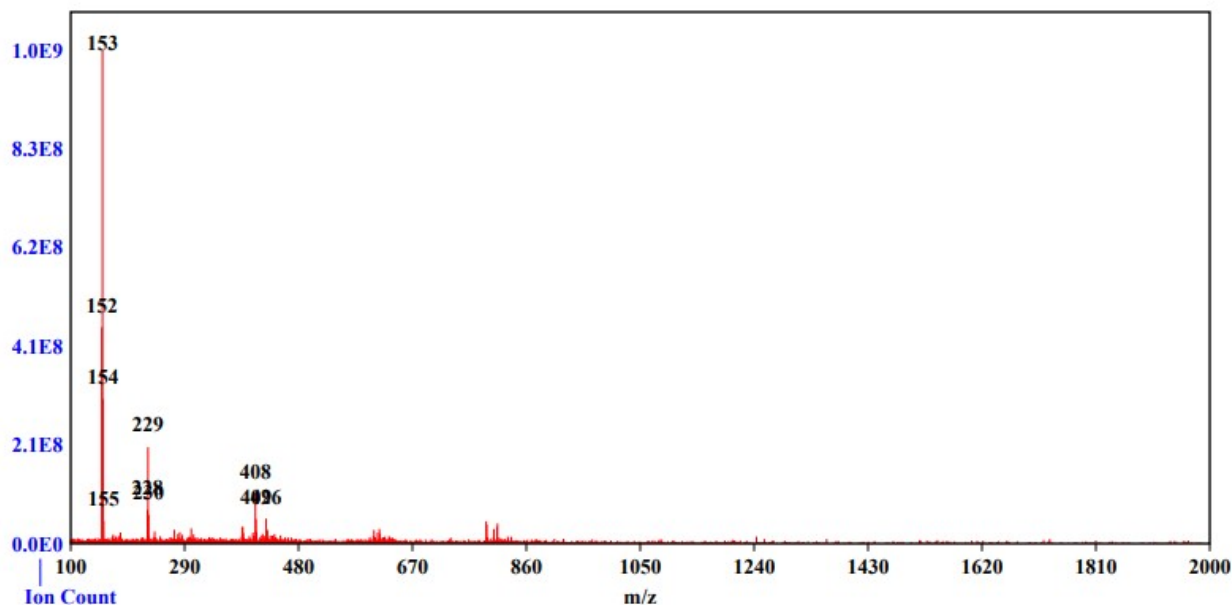
¹H

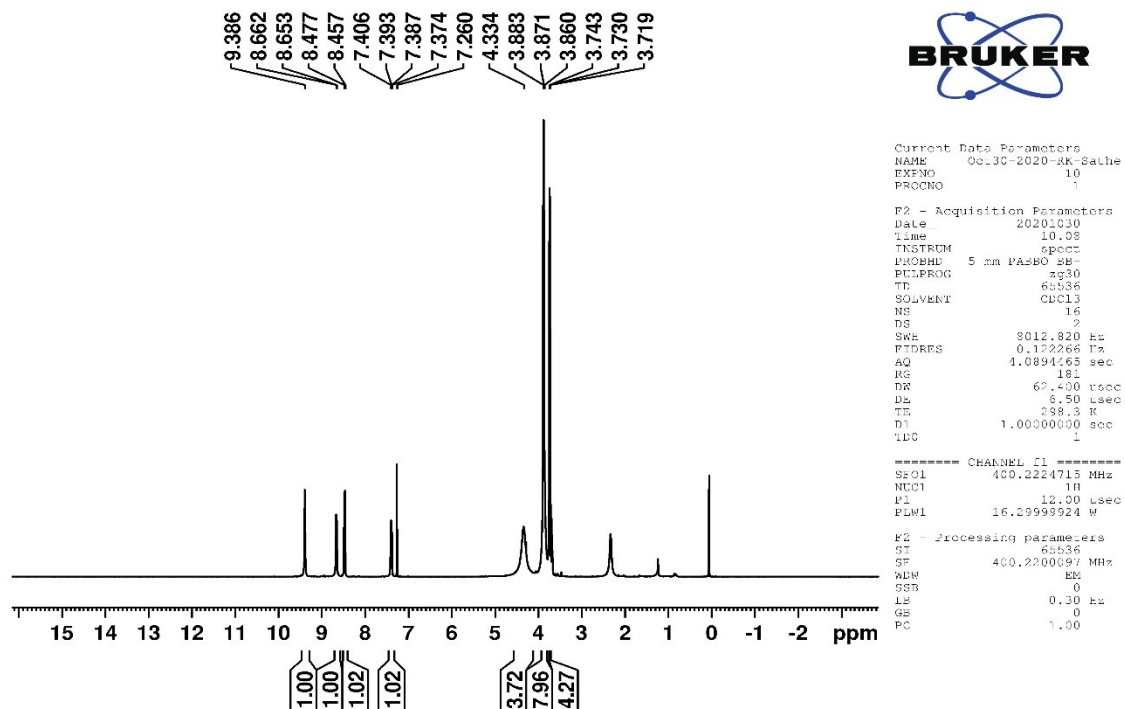
NMR spectrum for compound 2m (CDCl₃, 400 MHz)



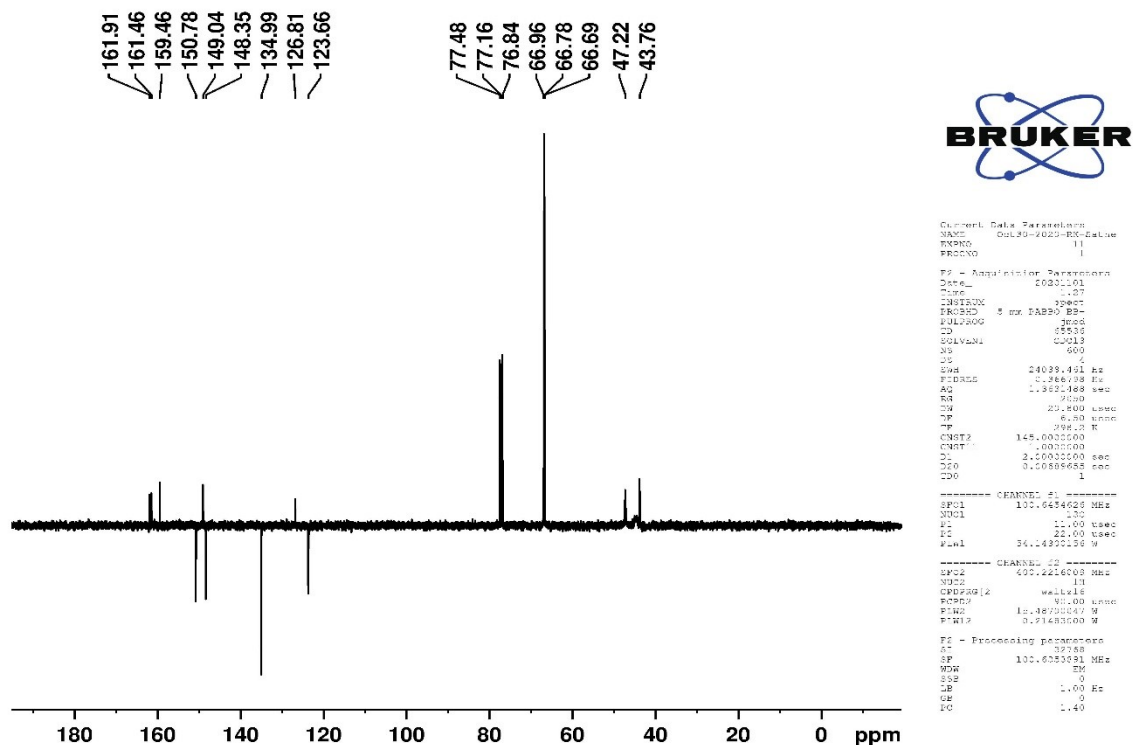
¹³C NMR spectrum for compound **2m** (CDCl₃, 100 MHz)

Spectrum Name: dim27
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



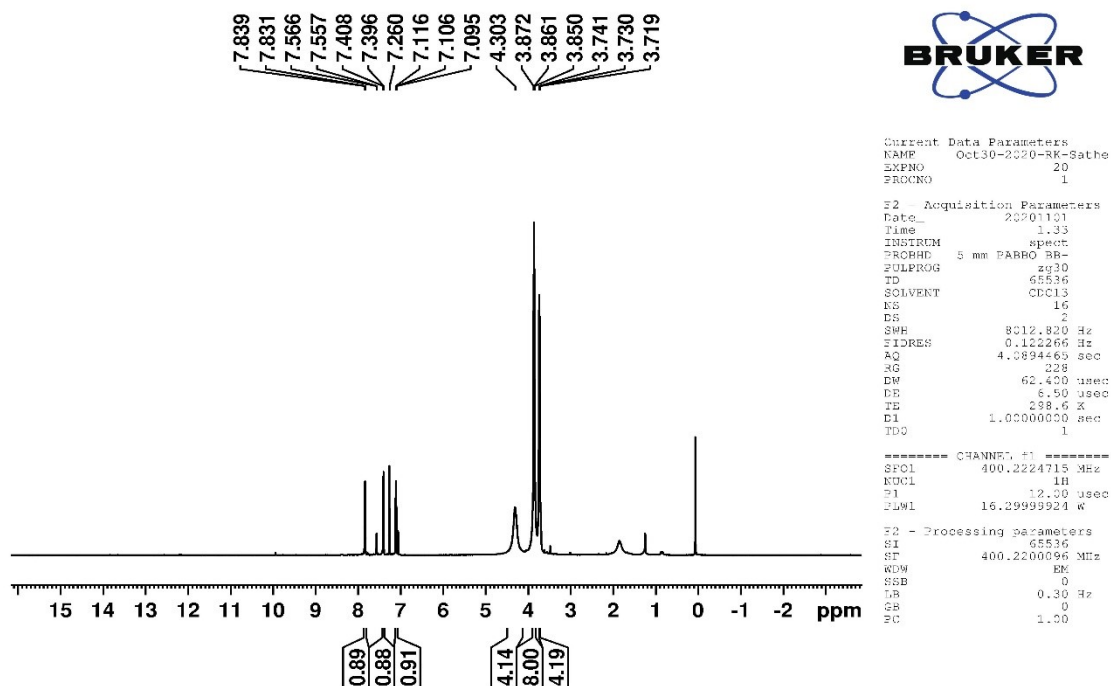
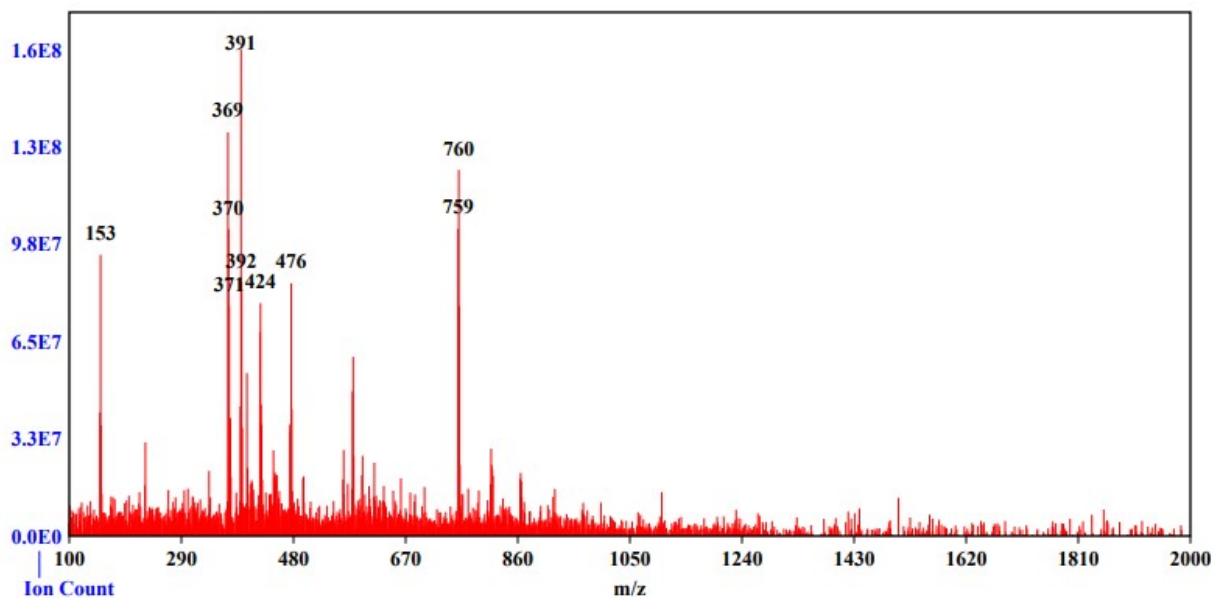


^1H NMR spectrum for compound **2n** (CDCl_3 , 400 MHz)

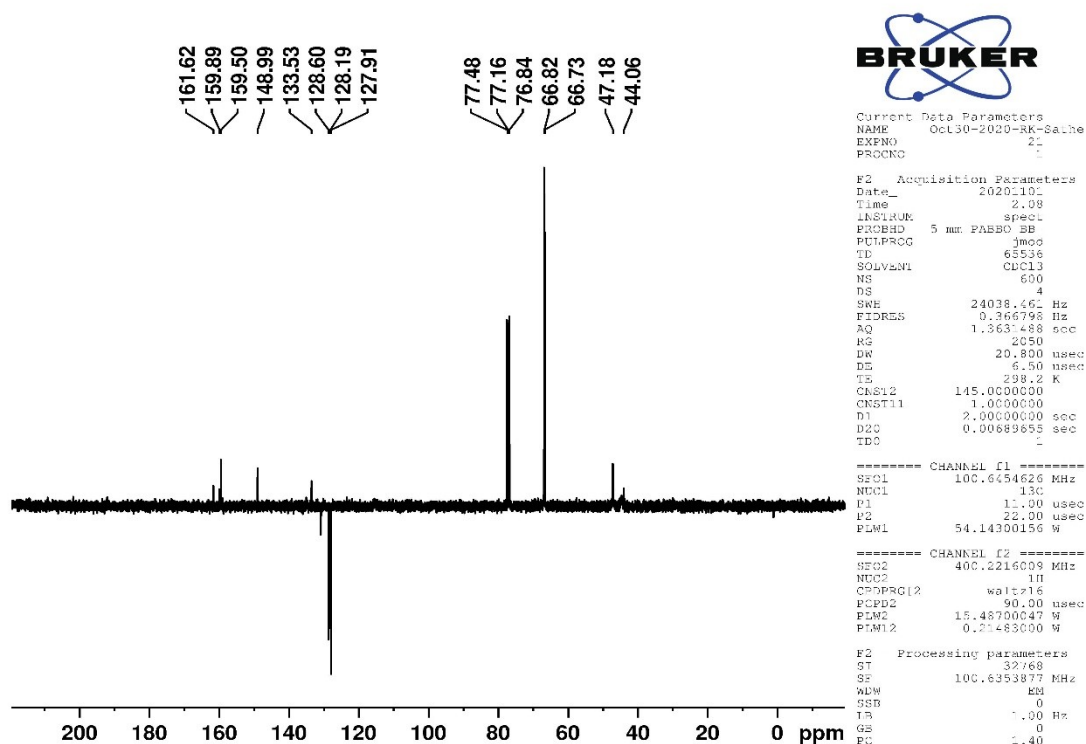


^{13}C NMR spectrum for compound **2n** (CDCl_3 , 100 MHz)

Spectrum Name: DIM-06
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V

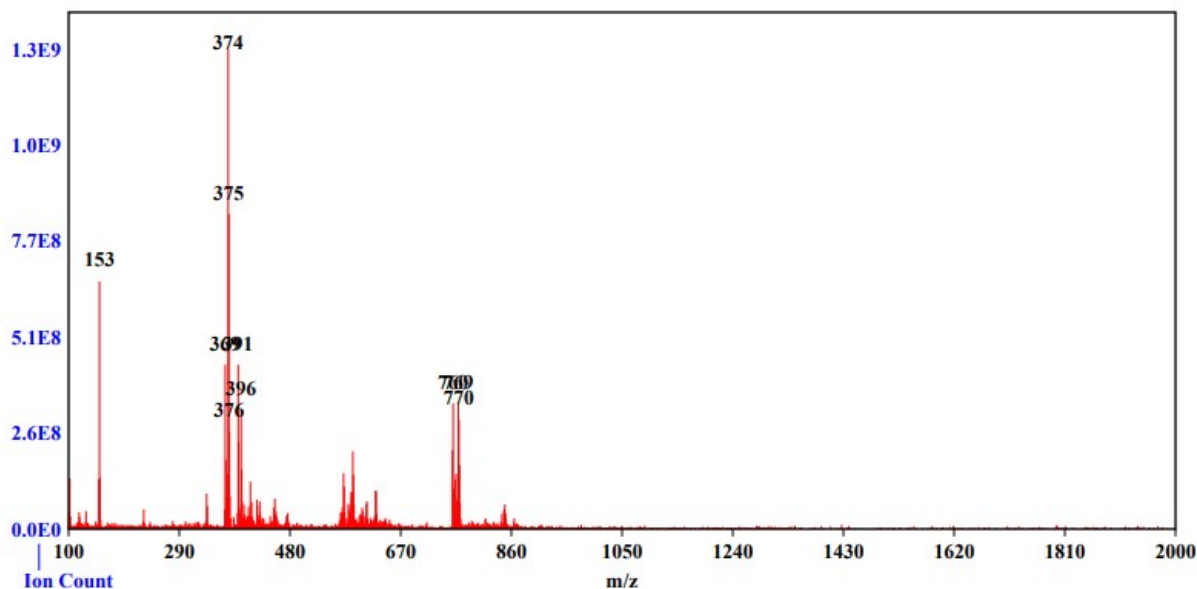


¹H NMR spectrum for compound **2o** (CDCl₃, 400 MHz)

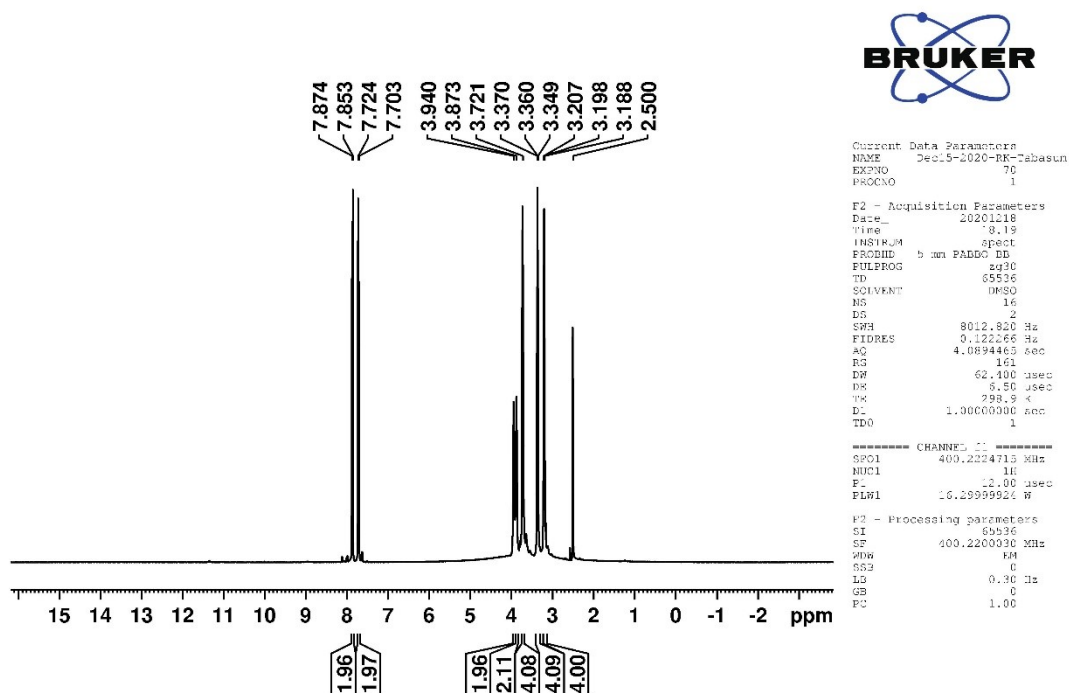


^{13}C NMR spectrum for compound **2o** (CDCl_3 , 100 MHz)

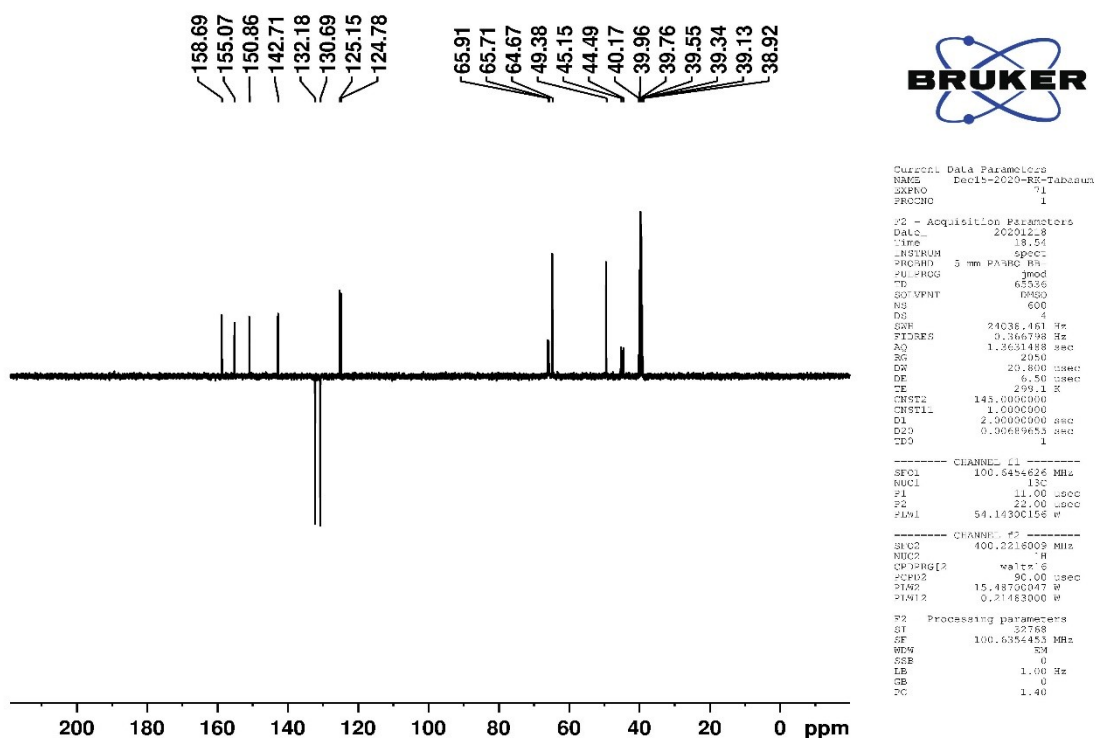
Spectrum Name: DIM-11
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



Mass spectrum for **2o**

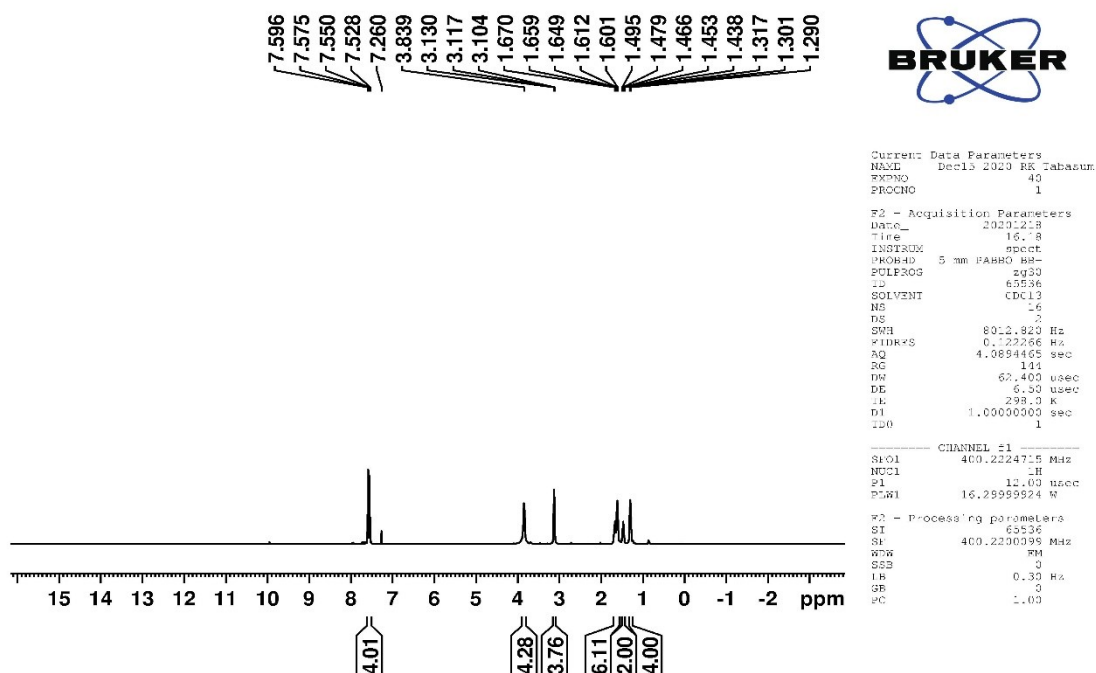
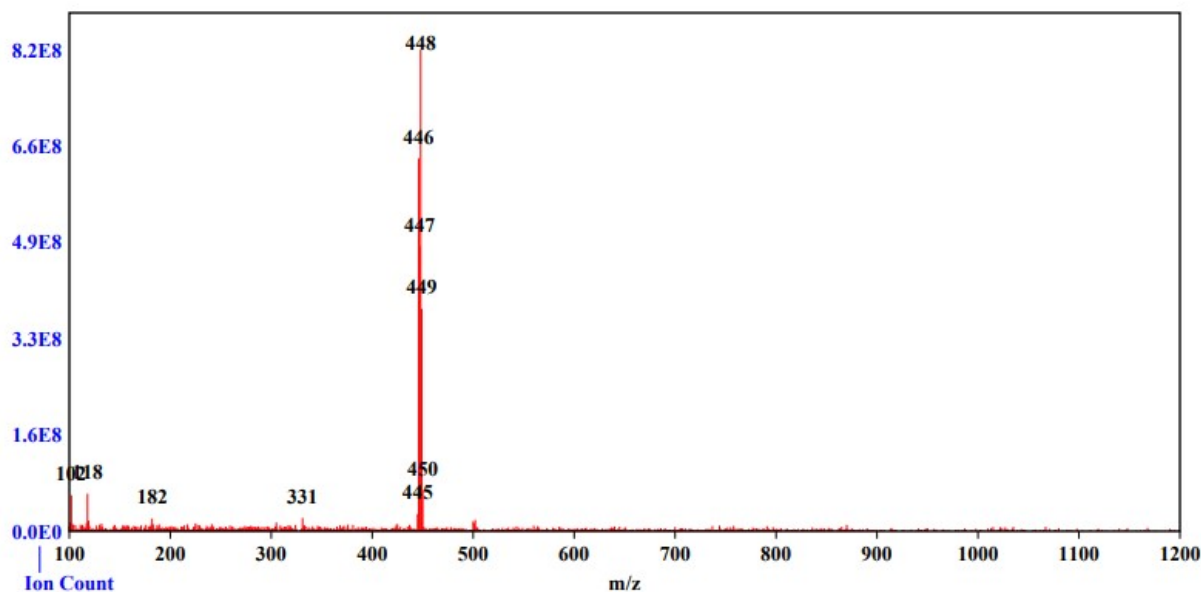


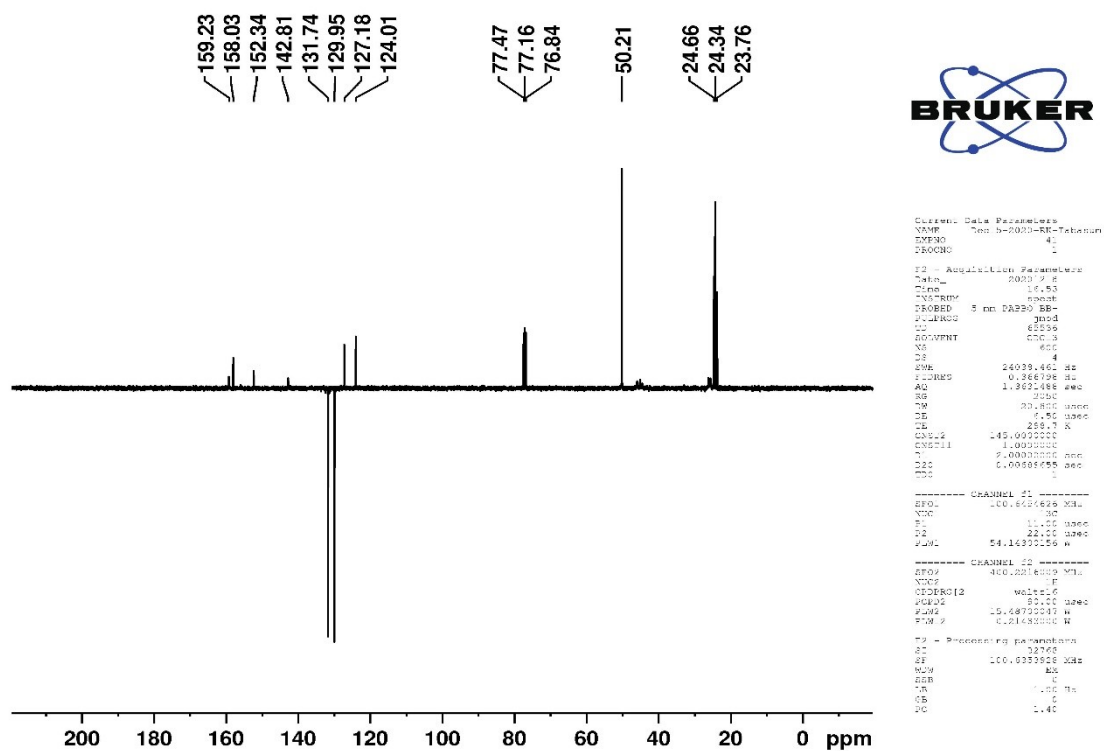
^1H NMR spectrum for compound **1b** (DMSO- d_6 , 400 MHz)



^{13}C NMR spectrum for compound **1b** (DMSO- d_6 , 100 MHz)

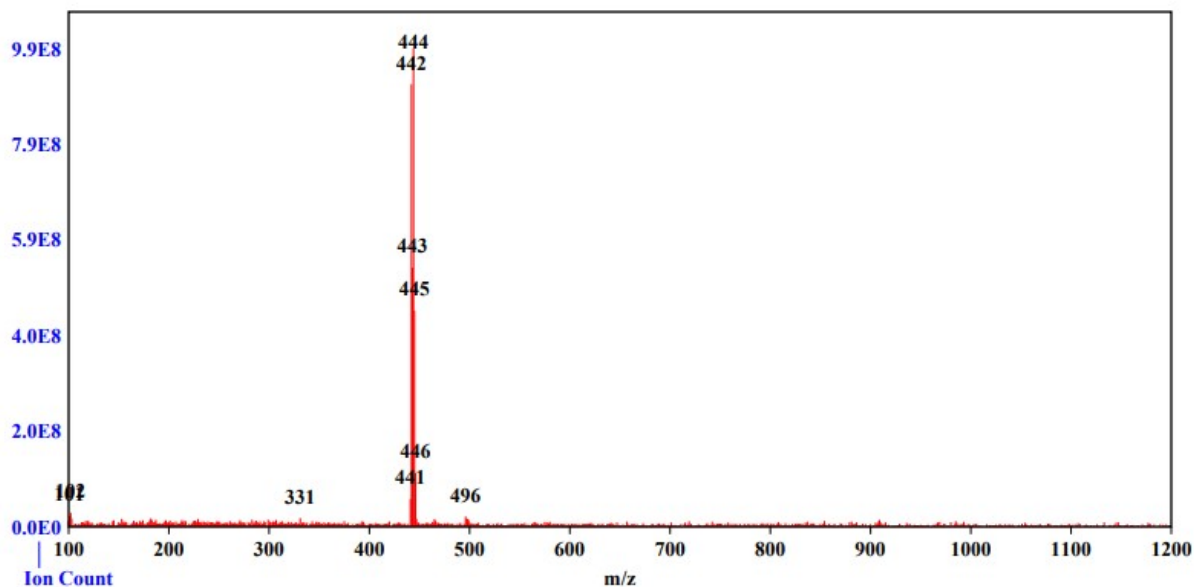
Spectrum Name: DS-06
 Start Ion: 100
 End Ion: 1200
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V

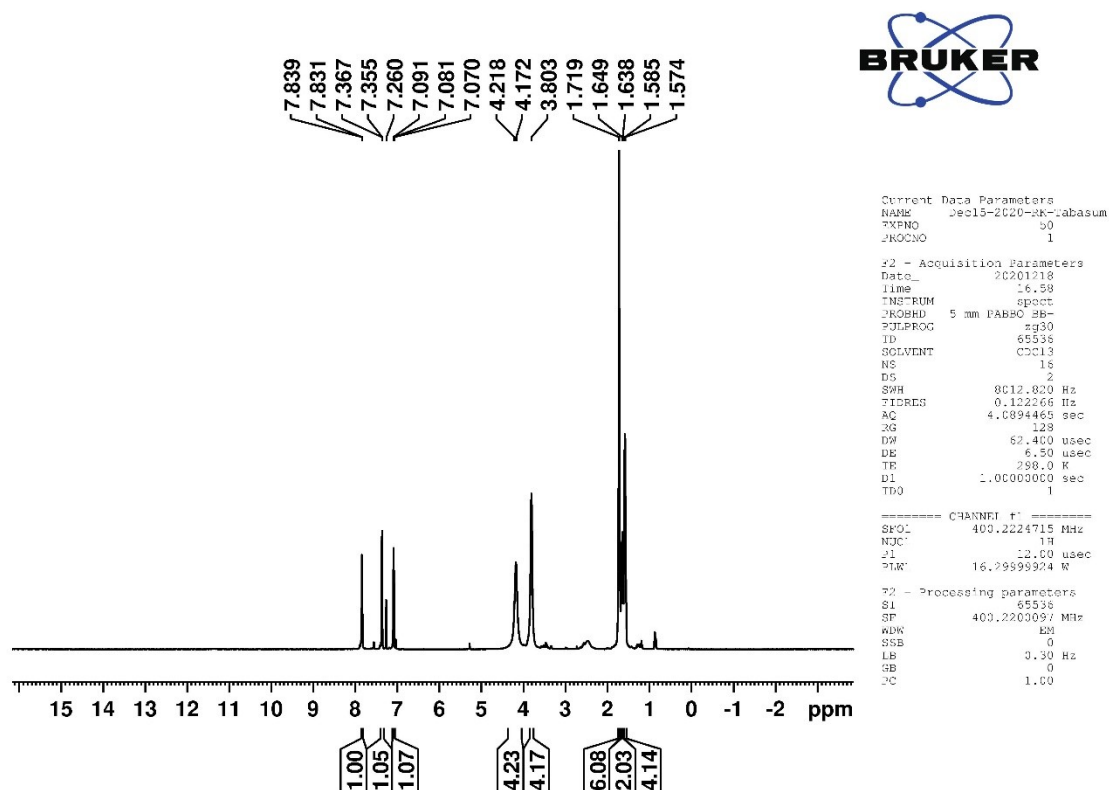




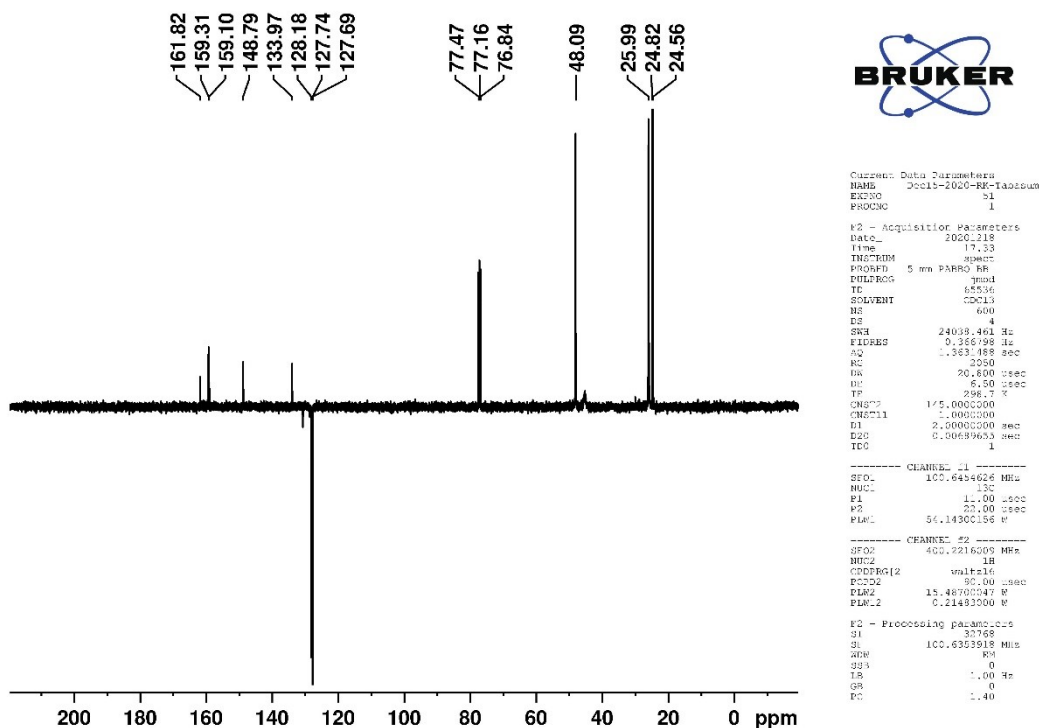
^{13}C NMR spectrum for compound **3b** (CDCl_3 , 100 MHz)

Spectrum Name: DS-101
 Start Ion: 100
 End Ion: 1200
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



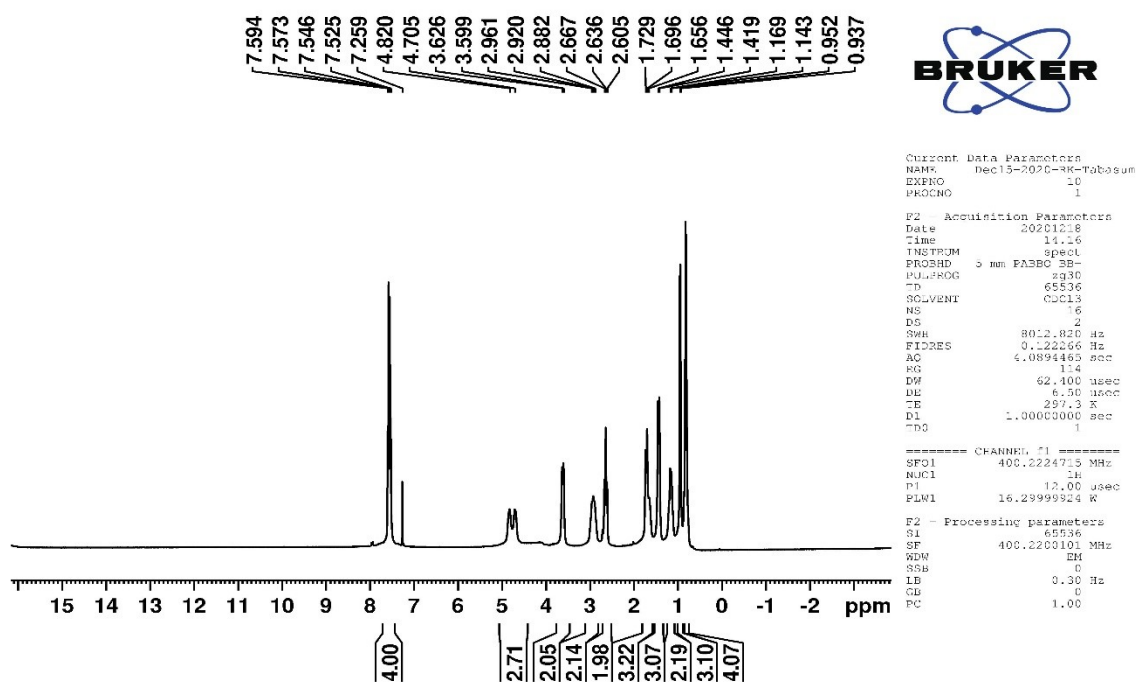
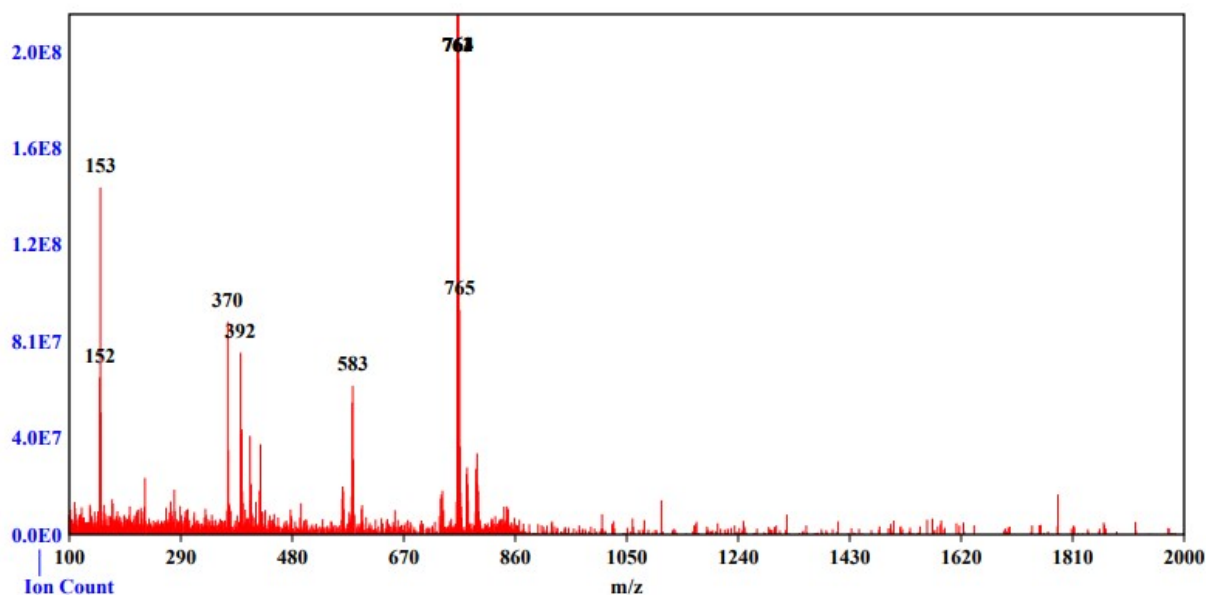


¹H NMR spectrum for compound **3c** (CDCl₃, 400 MHz)

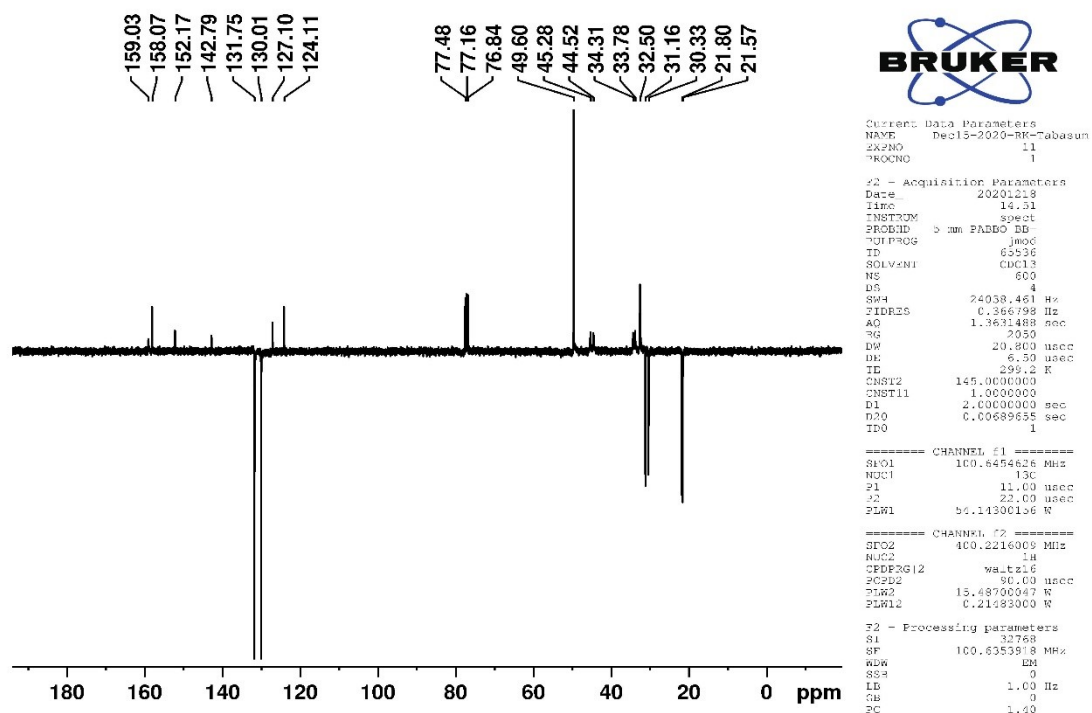


¹³C NMR spectrum for compound **3c** (CDCl₃, 100 MHz)

Spectrum Name: dim105-1
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V

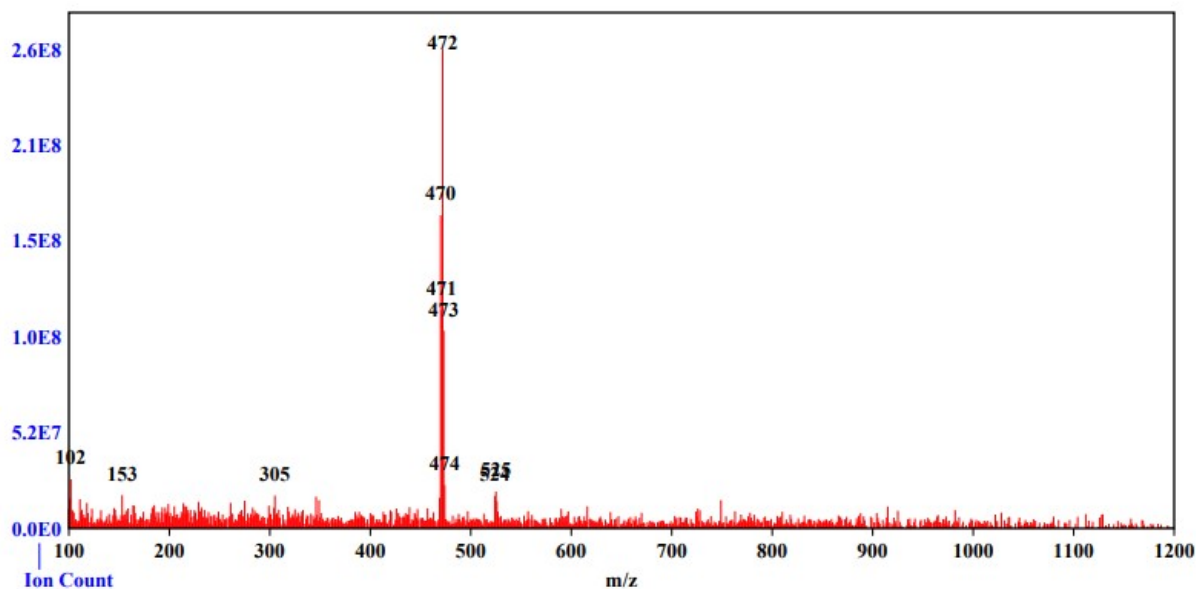


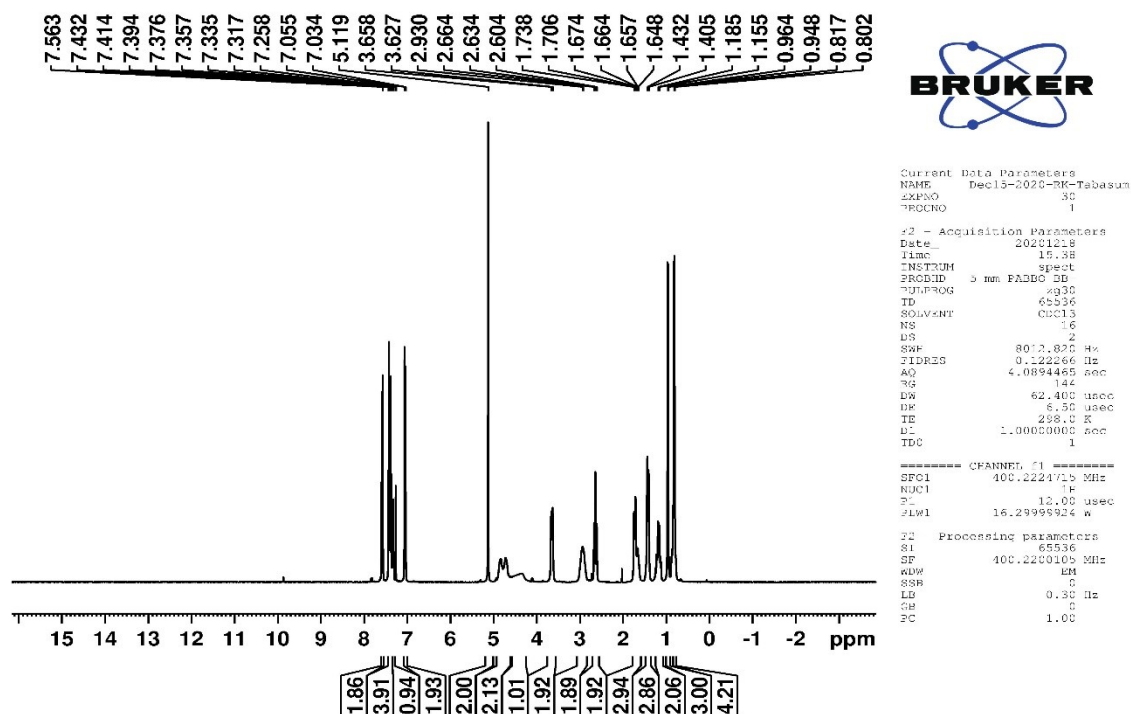
¹H NMR spectrum for compound 4b (CDCl₃, 400 MHz)



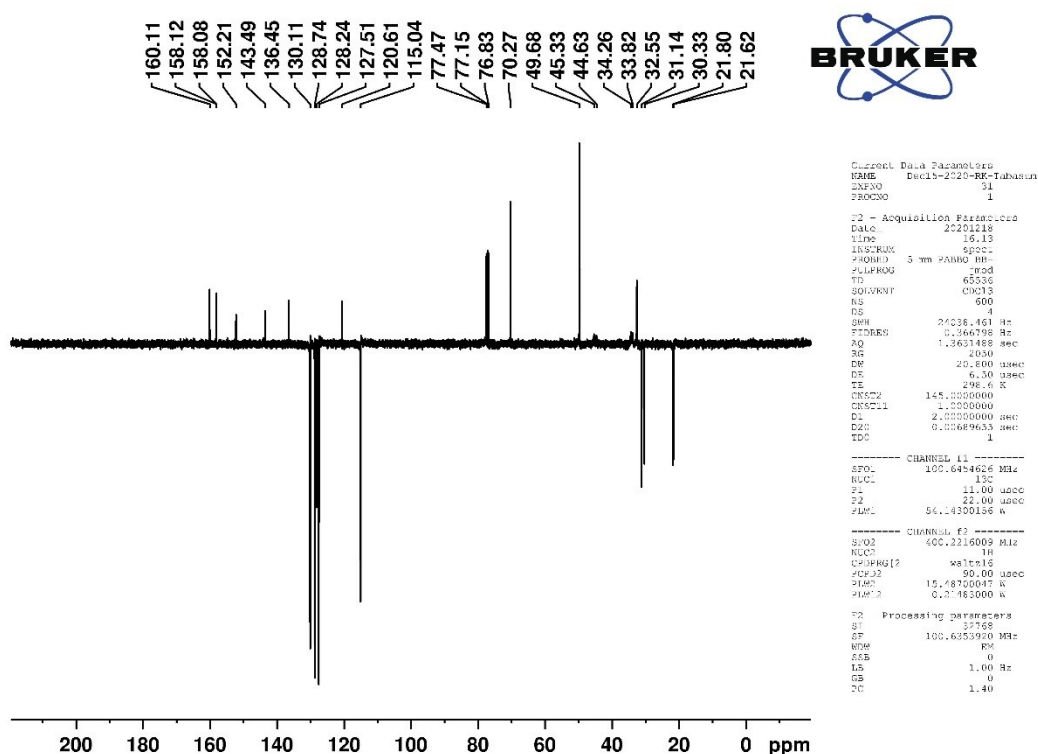
^{13}C NMR spectrum for compound **4b** (CDCl_3 , 100 MHz)

Spectrum Name: DS-201
 Start Ion: 100
 End Ion: 1200
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



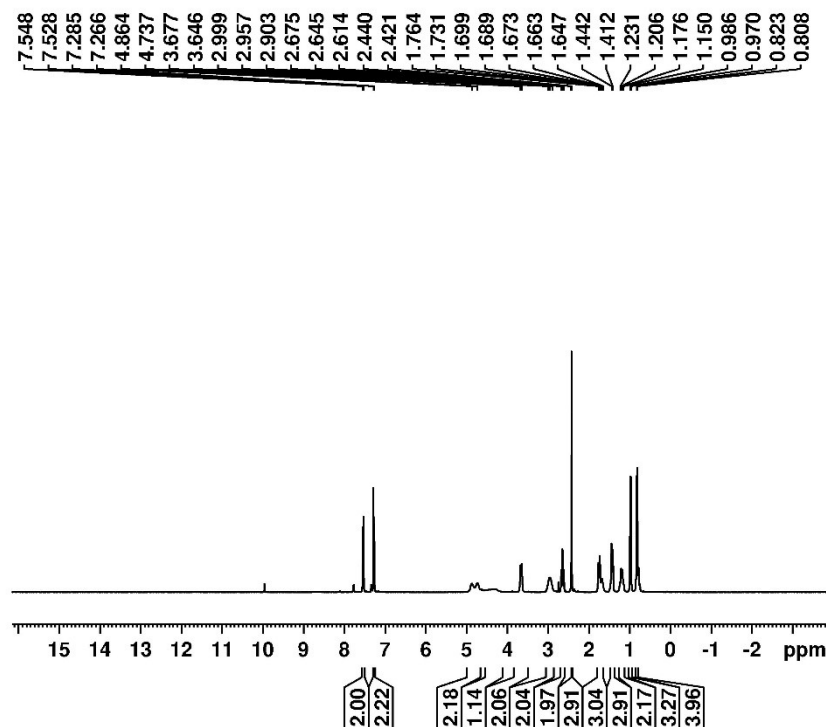
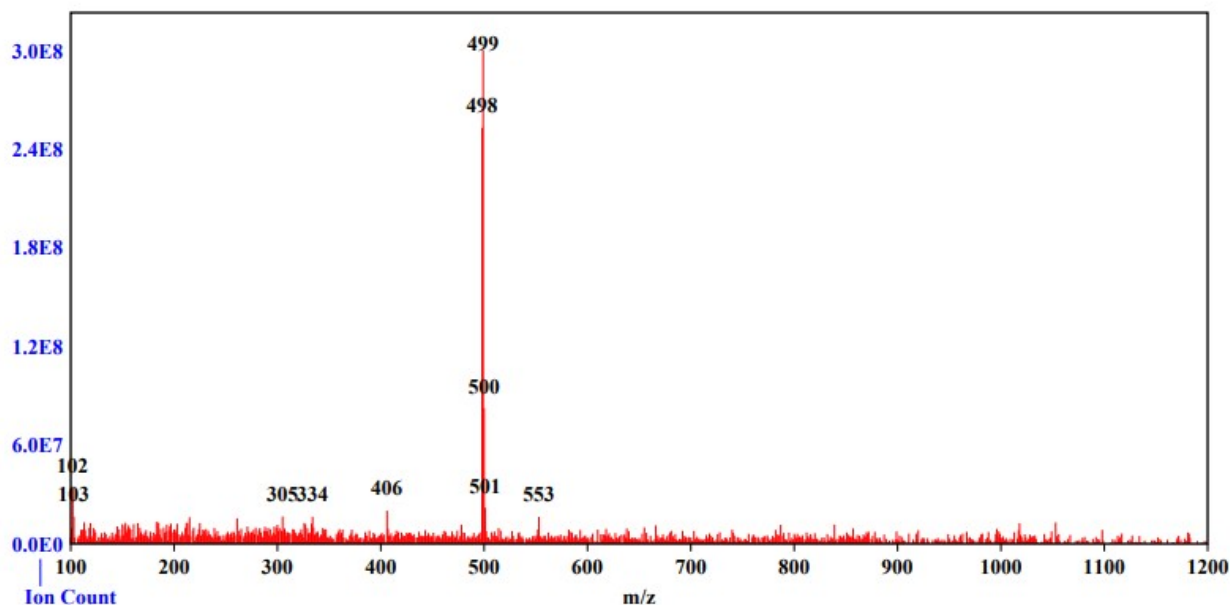


^1H NMR spectrum for compound **4c** (CDCl_3 , 400 MHz)



^{13}C NMR spectrum for compound **4c** (CDCl_3 , 100 MHz)

Spectrum Name: DS-203
 Start Ion: 100
 End Ion: 1200
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



Current Data Parameters
 NAME Dec15-2020-2K-Tabasum
 EXPNO 20
 PROCNO 1

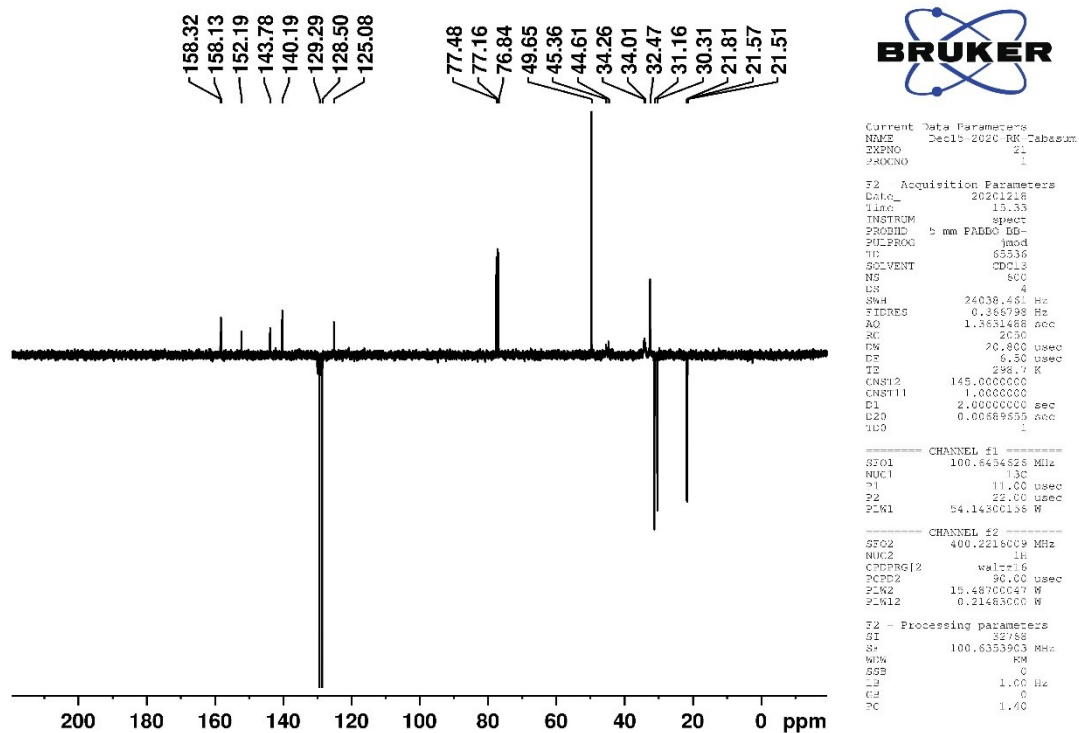
F2 - Acquisition Parameters
 Date_ 2020-12-18
 Time 14:58
 INSTRUM spect
 PROBEID 5 mm F400 QNP-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CHCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122286 Hz
 AQ 4.0894455 sec
 RG 144
 DW 62.400 usec
 DE 6.50 usec
 TE 298.5 K
 D1 1.00000000 sec
 TD0 1

----- CHANNEL f1 -----
 SFO1 400.2224715 MHz
 NUCL1 1H
 P1 12.00 usec
 PL1 16.29999924 dB

F2 - Processing parameters
 SI 65536
 SF 400.2224715 MHz
 WPR 3M
 SSB 0
 LB 0.30 Hz
 GR 0
 PC 1.00

¹H

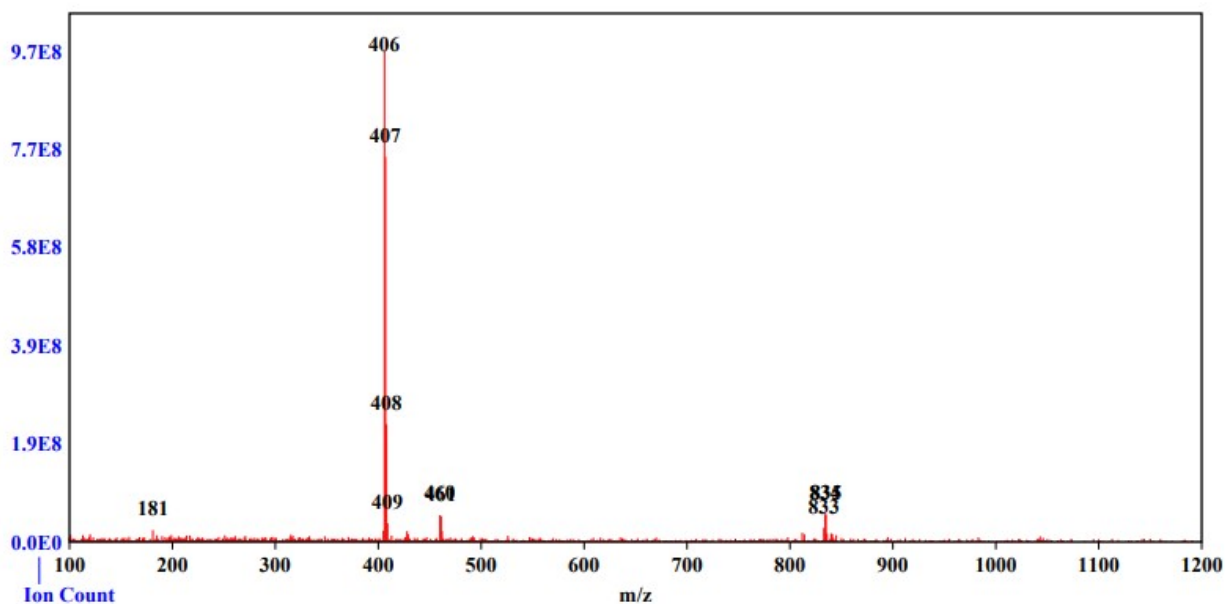
NMR spectrum for compound 4d (CDCl₃, 400 MHz)



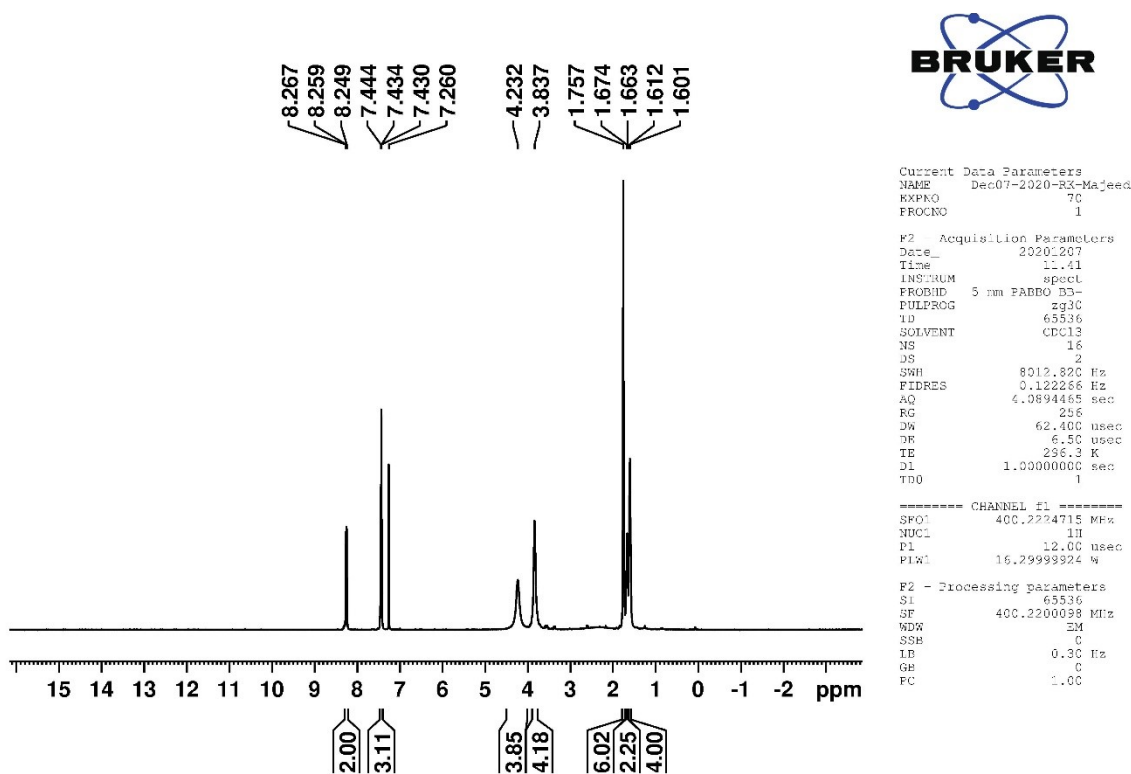
13

C NMR spectrum for compound **4d** (CDCl₃, 100 MHz)

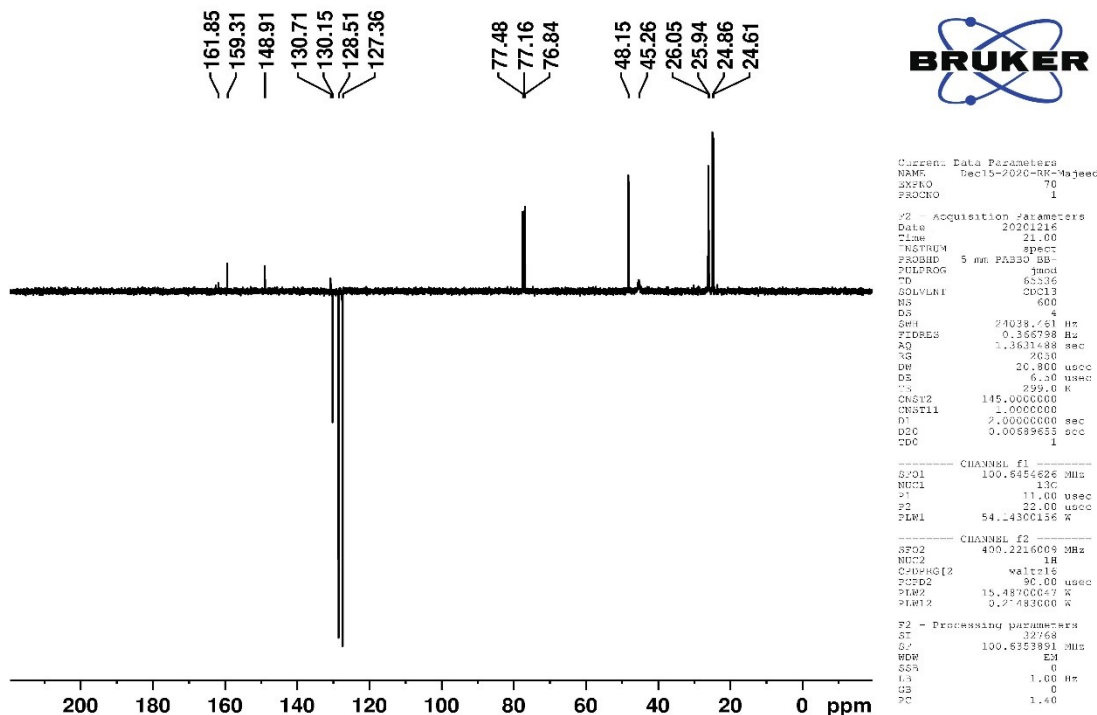
Spectrum Name: DS-202
Start Ion: 100
End Ion: 1200
Source: ESI + 3.5kV 350C
Capillary: 150V 300C Offset: 25V Span: 0V



Mass spectrum for **4d**

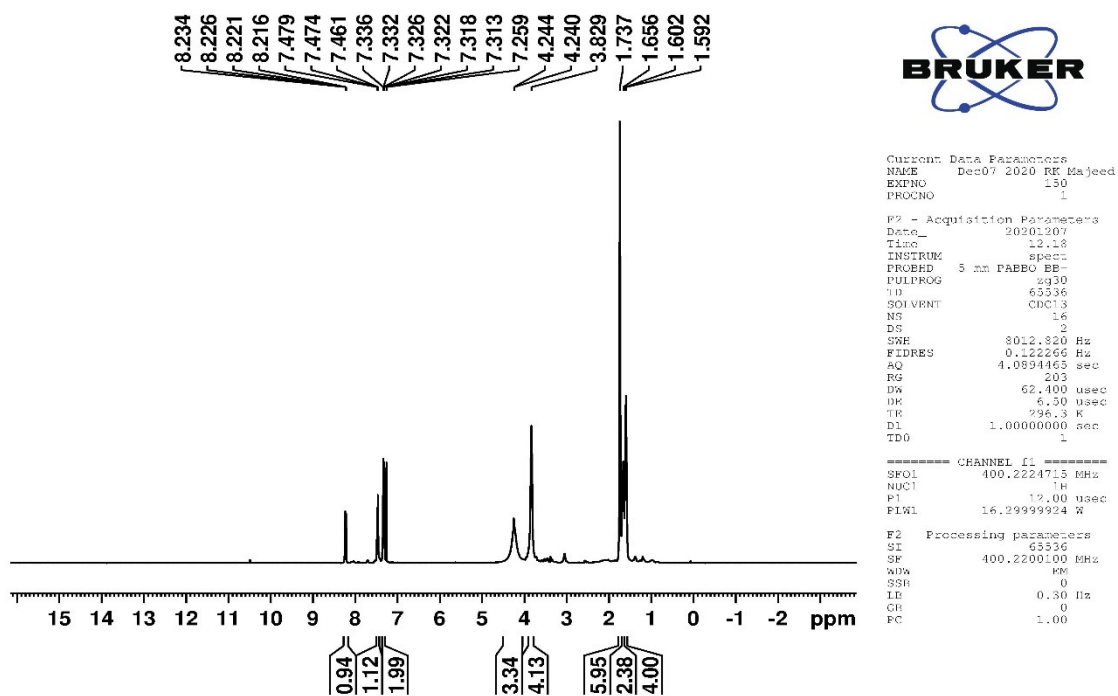
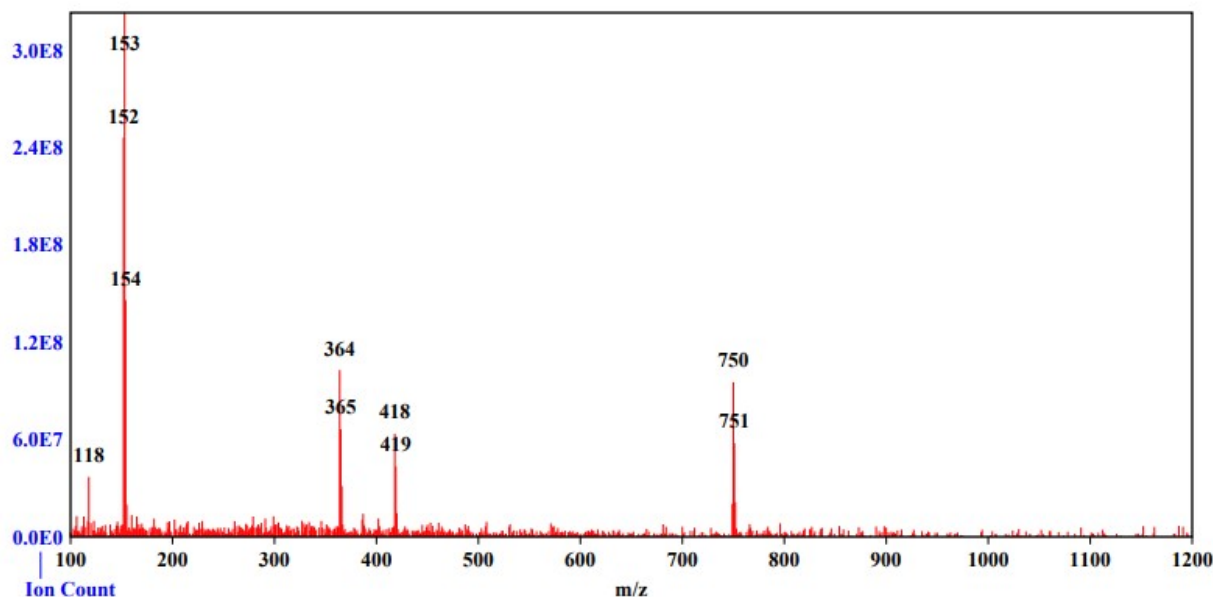


¹H NMR spectrum for compound **5a** (CDCl₃, 400 MHz)

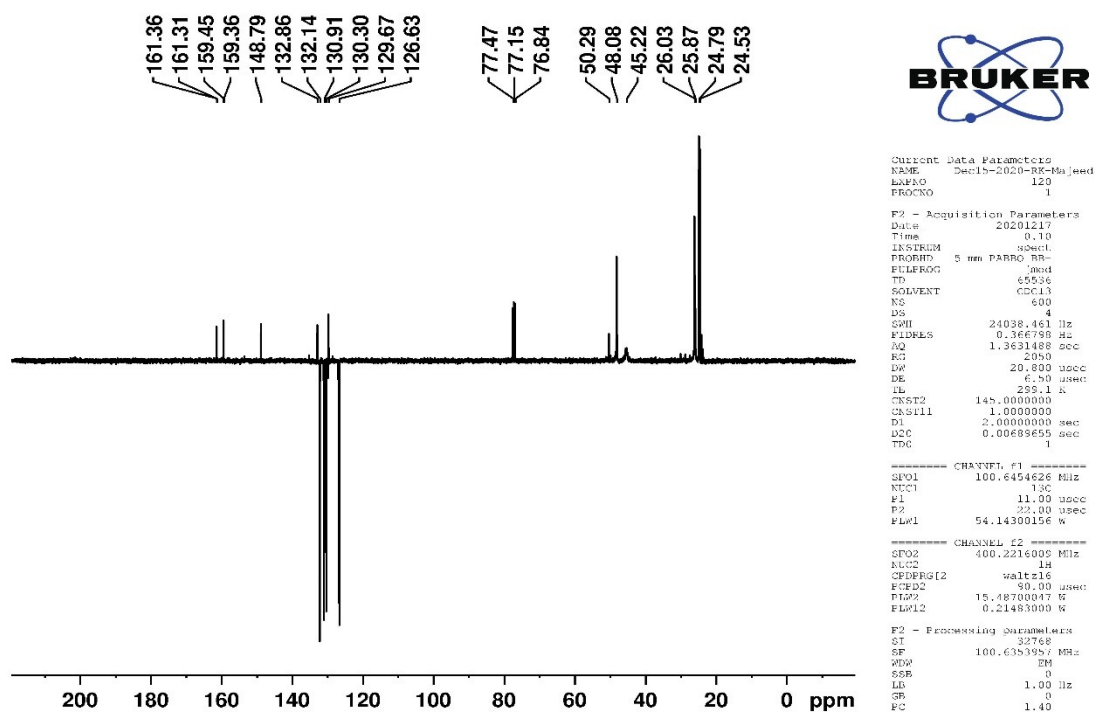


¹³C NMR spectrum for compound **5a** (CDCl₃, 100 MHz)

Spectrum Name: DIM-113
 Start Ion: 100
 End Ion: 1200
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V

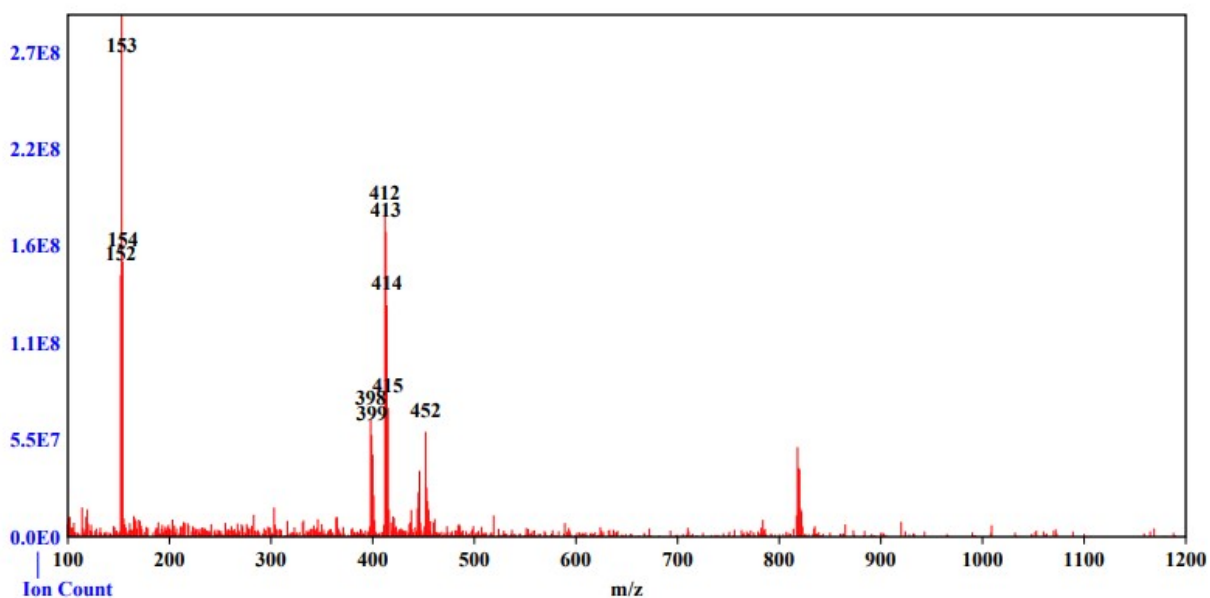


¹H NMR spectrum for compound **5b** (CDCl₃, 400 MHz)

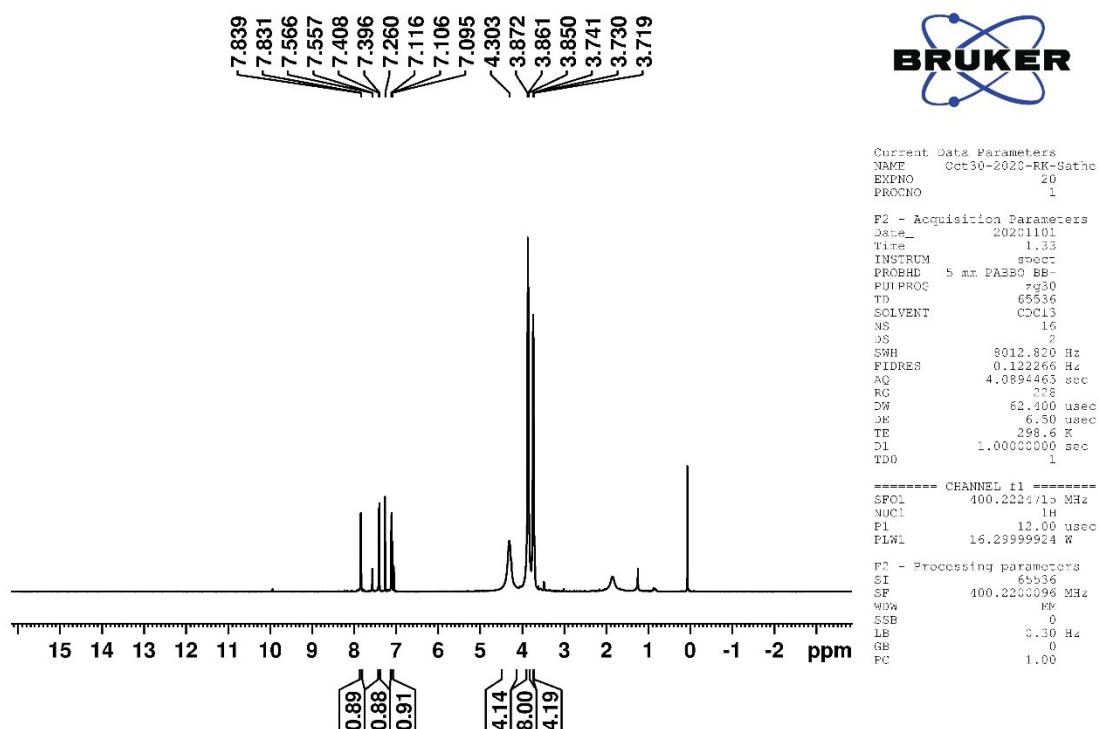


¹³C NMR spectrum for compound **5b** (CDCl₃, 100 MHz)

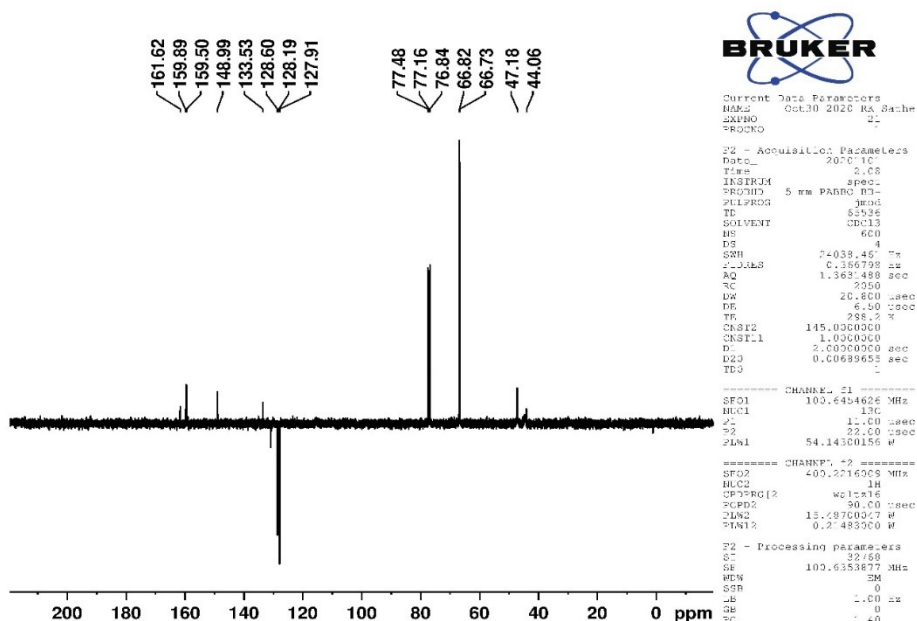
Spectrum Name: DIM-140
 Start Ion: 100
 End Ion: 1200
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



Mass spectrum for **5b**

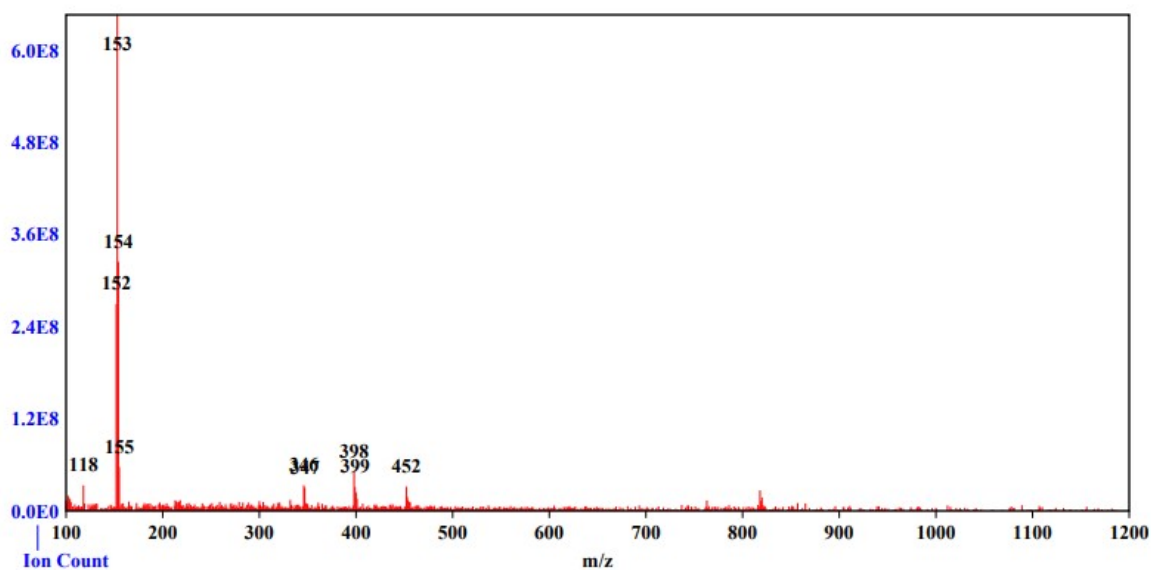


¹H NMR spectrum for compound **5c** (CDCl₃, 400 MHz)

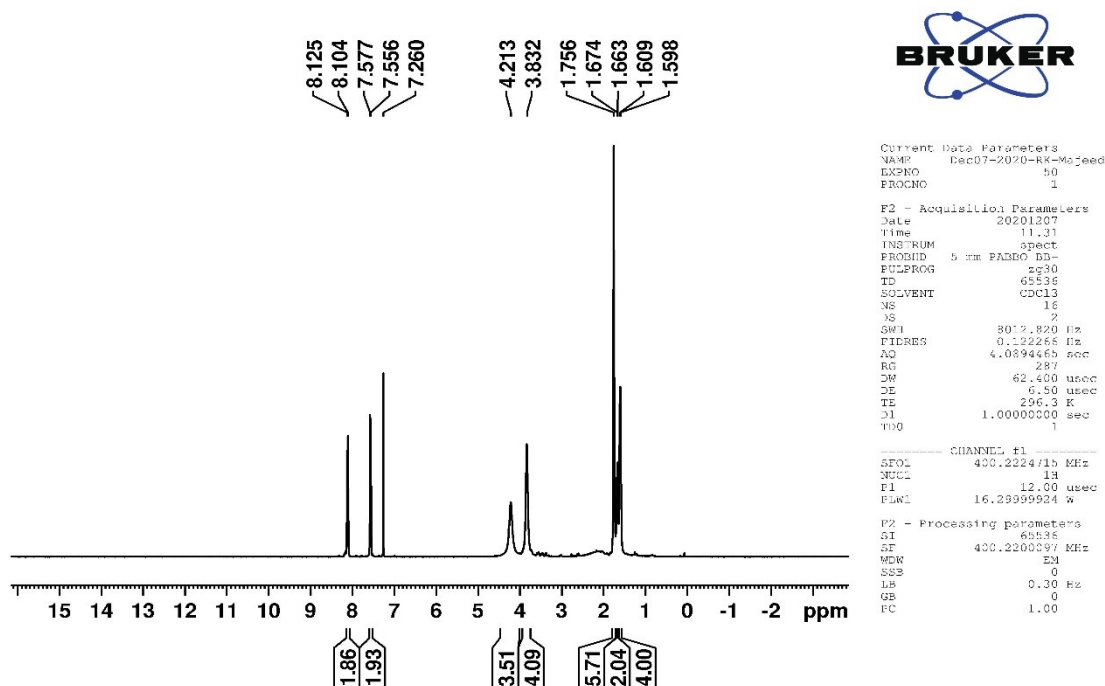


¹³C NMR spectrum for compound **5c** (CDCl₃, 100 MHz)

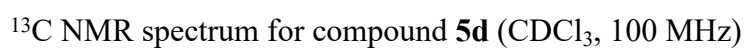
Spectrum Name: DIM-110
 Start Ion: 100
 End Ion: 1200
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



Mass spectrum for 5c

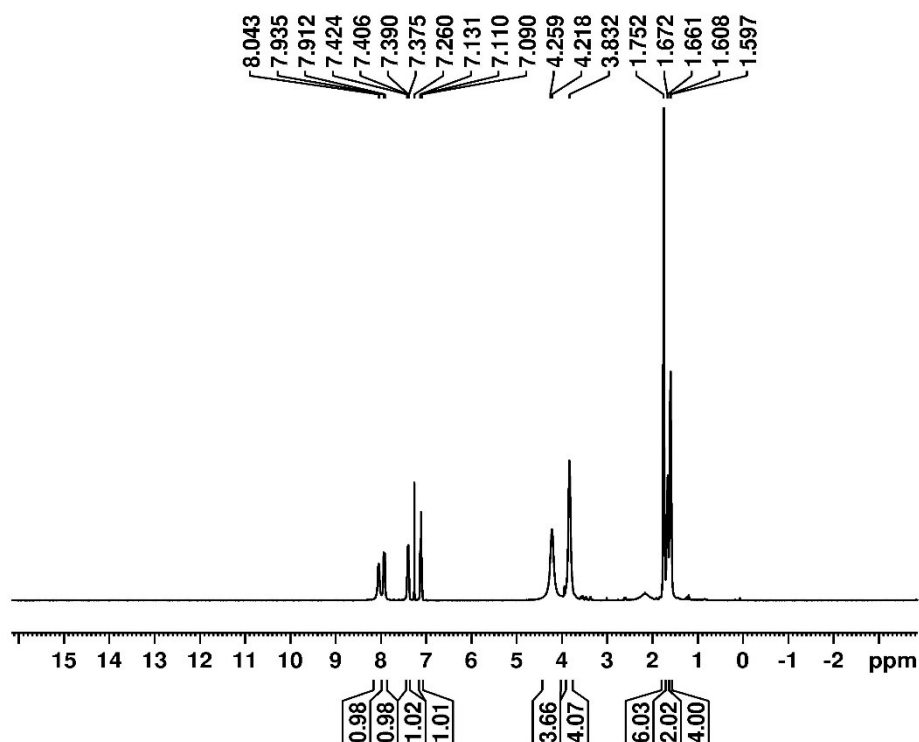


¹H NMR spectrum for compound 5d (CDCl₃, 400 MHz)



Mass spectrum showing relative intensity (Y-axis, 0.0E0 to 5.2E8) versus m/z (X-axis, 100 to 1200). The base peak is at m/z 153. Other labeled peaks include m/z 102, 118, 152, 154, 346, 383, and 445.

Mass spectrum for **5d**



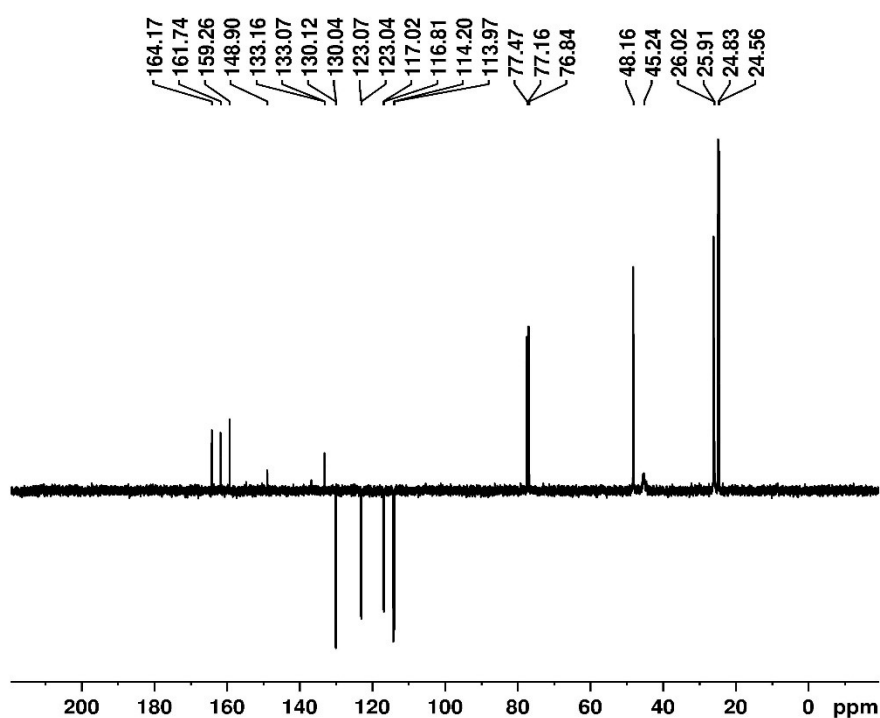
Current Data Parameters
NAME Dec07-2020-RK-Majeed
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20201207
Time 11.16
INSTRUM spect
PROBHD 5 mm PARBO B3
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 203
DW 62.400 usec
DE 6.50 usec
TE 296.3 K
D1 1.0000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 400.222413 MHz
P1 12.00 usec
PLW1 16.29999924 W

F2 - Processing parameters
ST 65536
SF 400.2200093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^1H NMR spectrum for compound **5e** (CDCl_3 , 400 MHz)



Current Data Parameters
NAME Dec10-2020-RK-Majeed
EXPNO 41
PROCNO 1

F2 - Acquisition Parameters
Date_ 20201210
Time 16.27
INSTRUM spect
PROBHD 5 mm PARBO B3
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 800
DS 4
SWH 24638.401 Hz
FIDRES 0.368788 Hz
AQ 1.3631488 sec
RG 2030
DW 20.800 usec
DE 6.50 usec
TE 299.1 K
D1 45.0000000 sec
D2 2.0000000 sec
D3 2.0000000 sec
TD0 0.0000000 sec
TD1 1

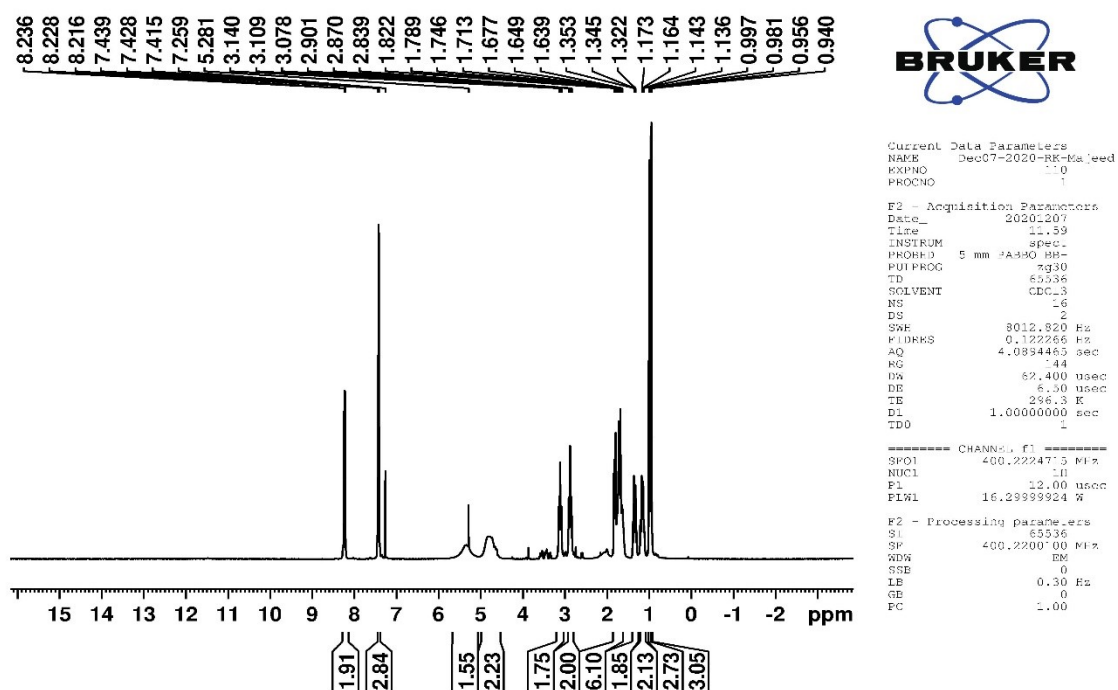
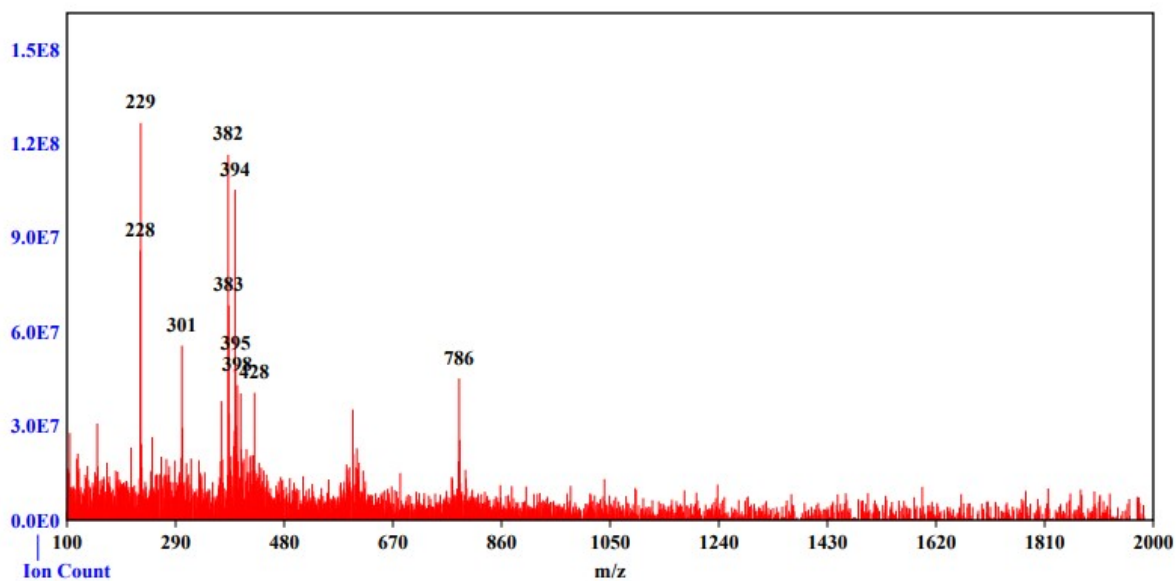
----- CHANNEL f1 -----
NUC1 100.626183 MHz
P1 11.00 usec
PLW1 22.00 usec
PLW2 54.14200124 W

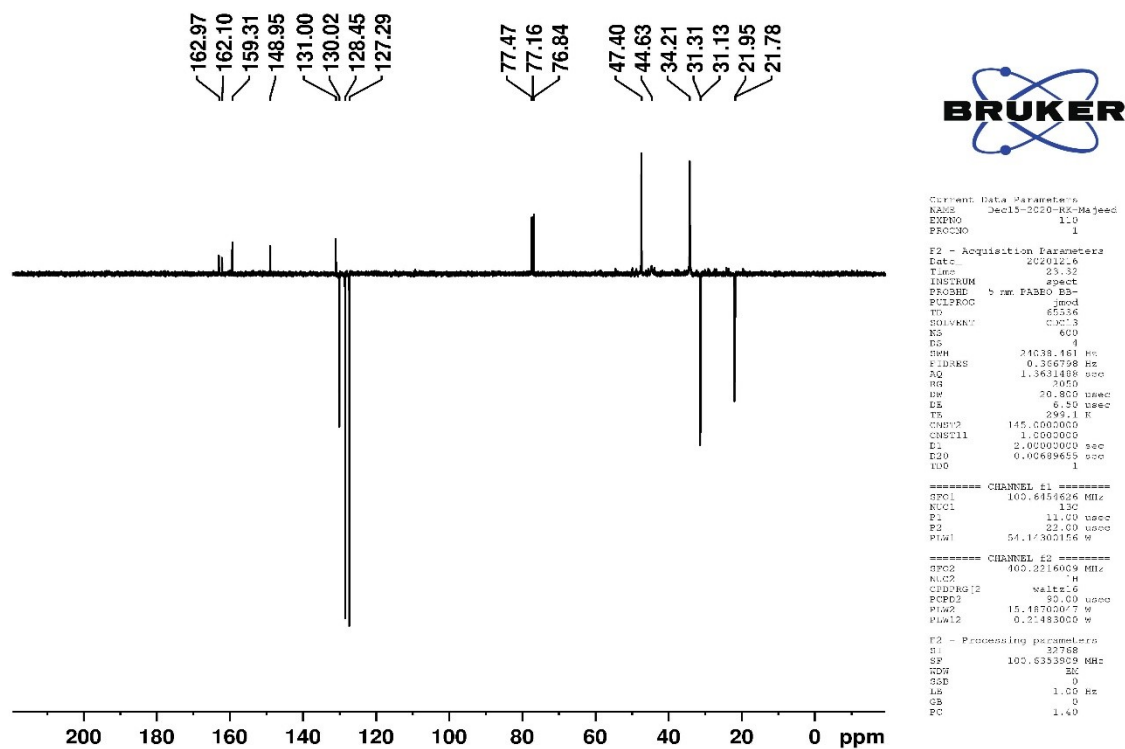
----- CHANNEL f2 -----
NUC2 400.2210009 MHz
P2 11.16 usec
PLW2 15.44700047 W
PLW3 0.214483000 W

F2 - Processing parameters
ST 32768
SF 100.626183 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{13}C NMR spectrum for compound **5e** (CDCl_3 , 100 MHz)

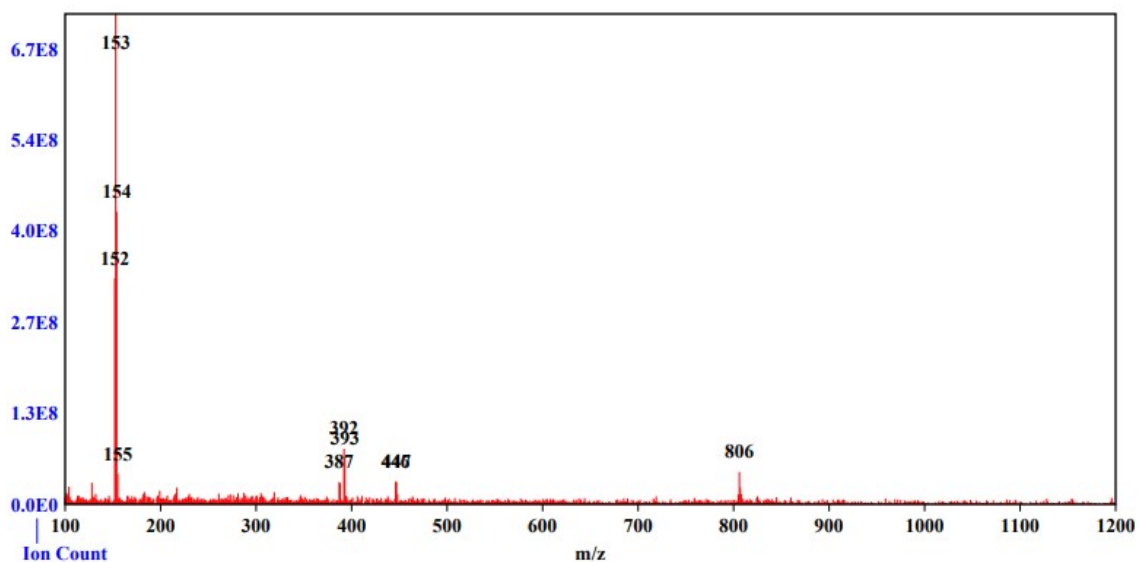
Spectrum Name: dim106
 Start Ion: 100
 End Ion: 2000
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V





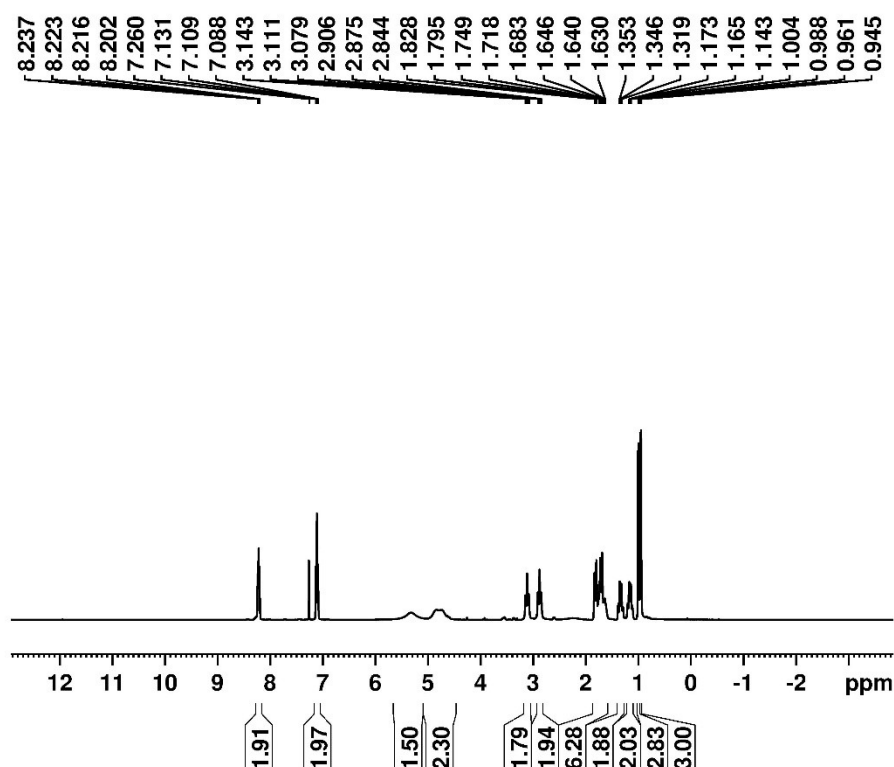
¹³C NMR spectrum for compound **6a** (CDCl₃, 100 MHz)

Spectrum Name: DIM-404
 Start Ion: 100
 End Ion: 1200
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



Mass spectrum for **6a**

Mas

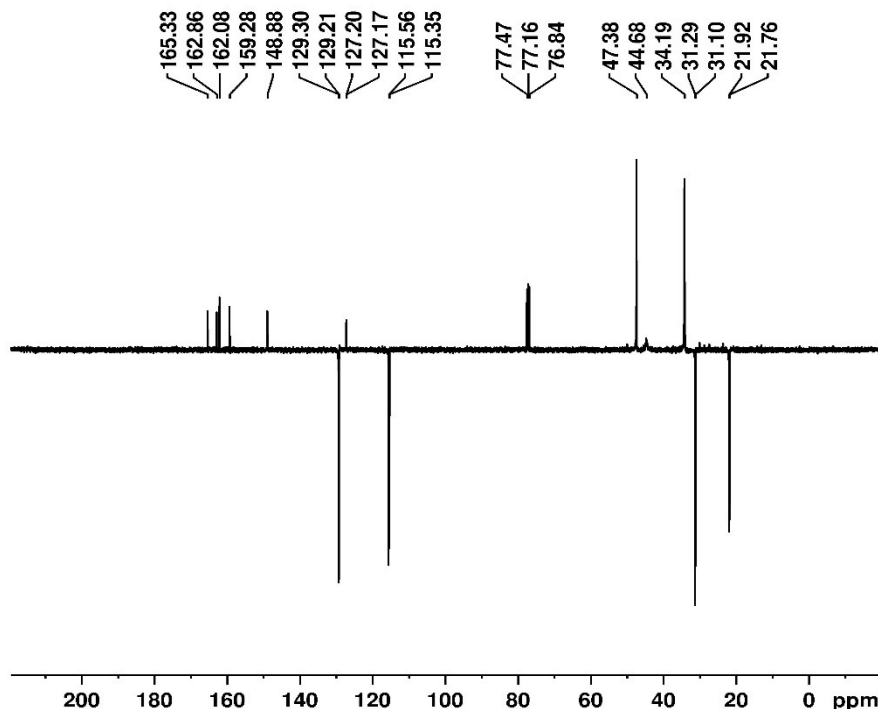


Current Data Parameters
NAME Dec07-2020-RK-Majeed
EXPNO 100
PROCNO 1

F2 - Acquisition Parameters
Date_ 20201207
Time 11.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 181
DW 62.400 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SF01 400.2224715 MHz
NUC1 1H
P1 12.00 usec
PLW1 16.29999924 W

F2 - Processing parameters
SI 65536
SF 400.2200098 MHz
WDW RM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME Dec-3-2020-RK-Majeed
EXPNO 100
PROCNO 1

F2 - Acquisition Parameters
Date_ 20201216
Time 22.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632488 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 299.1 K
CNS17 145.0000000
CNS11 1.0000000
D1 2.00000000 sec
D20 0.00689653 sec
TD0 1

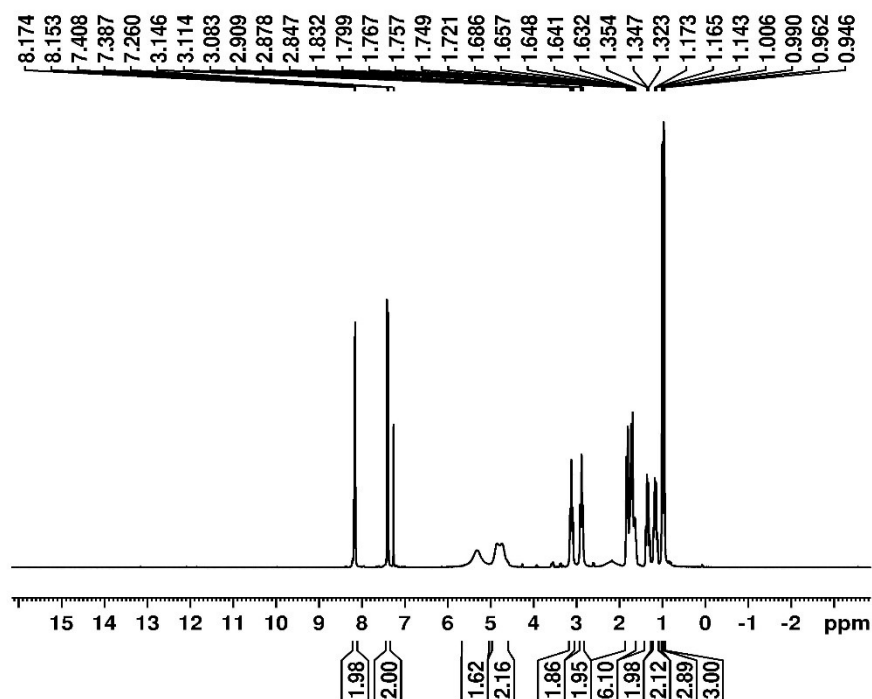
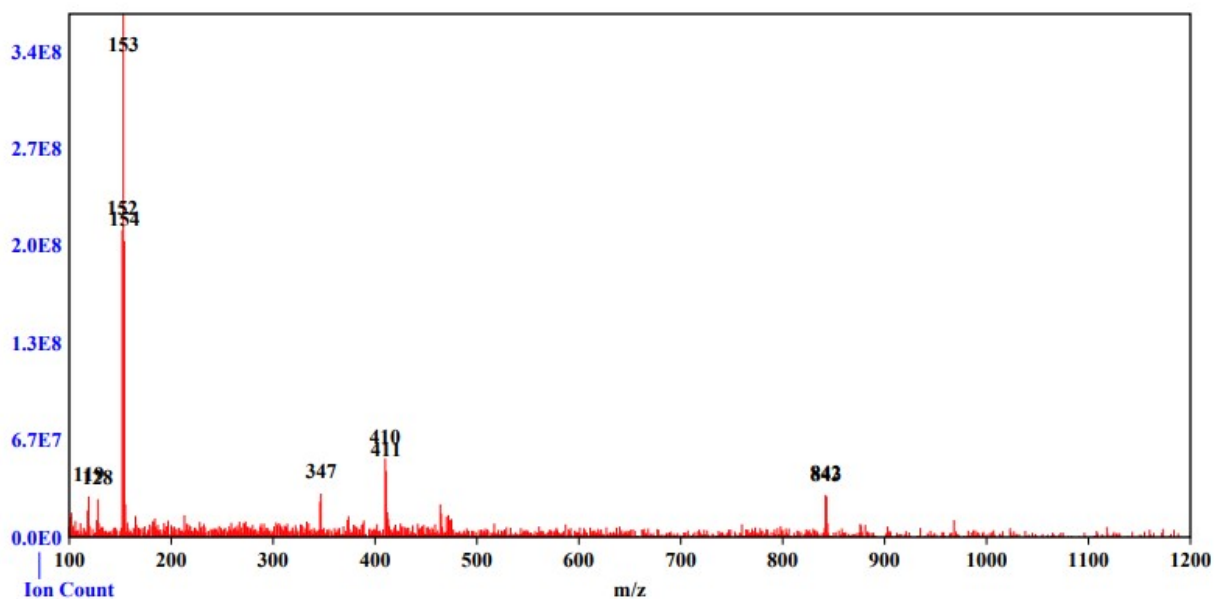
===== CHANNEL f1 =====
SF01 100.6264562 MHz
NUC1 13C
P1 12.00 usec
PLW1 54.14300156 W

===== CHANNEL f2 =====
SF02 400.2216009 MHz
NUC2 1H
CPCPRG2 waltz16
PCPD2 90.00 usec
P2A2 15.48700047 K
PLW2 0.21483000 W

F2 - Processing parameters
SI 32768
SF 100.6253914 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹³C NMR spectrum for compound **6b** (CDCl₃, 100 MHz)

Spectrum Name: DIM-403
 Start Ion: 100
 End Ion: 1200
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V

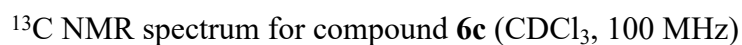


Current Data Parameters
 NAME Dec07-2020-HK-Majeed
 EXPNO 80
 PROCNO 1

F2 Acquisition Parameters
 Date_ 20201207
 Time 11.45
 INSTRUM spect
 PROBRD 5 mm PAHCO BH-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 8012.620 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 181
 DW 52.400 usec
 DE 6.00 usec
 TE 296.3 K
 DI 1.00000000 sec
 TDO 1

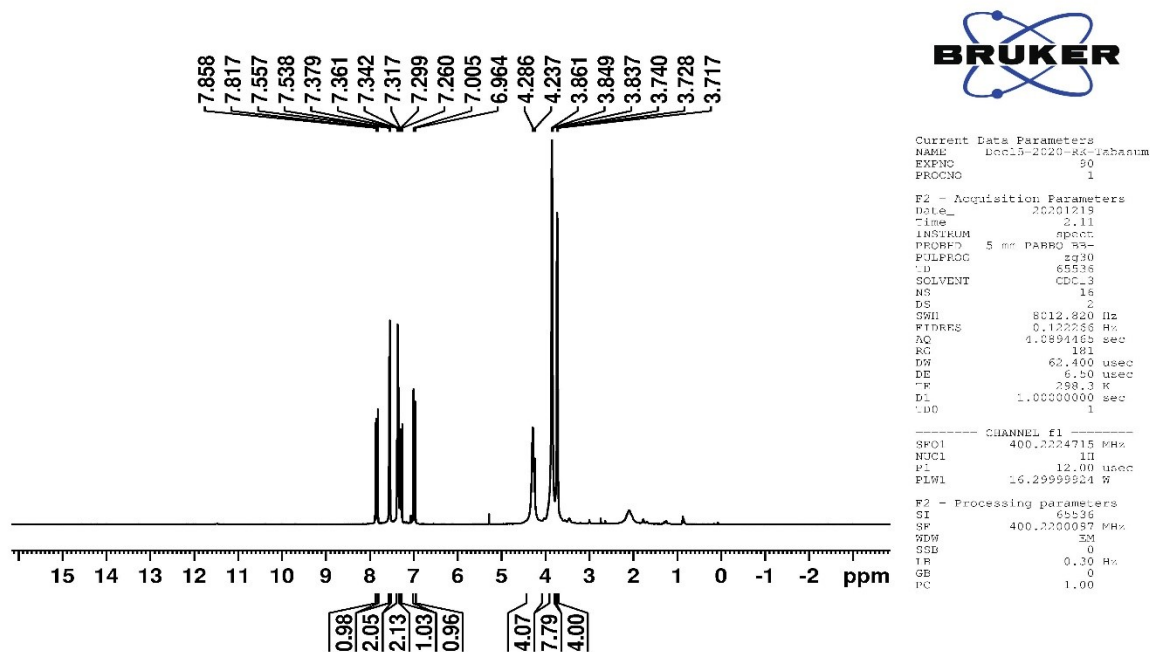
===== CHANNEL f1 =====
 SPOL 400.2224715 MHz
 NUCL 1H
 P1 12.00 usec
 PLW 16.29999924 W

F2 - Processing parameters
 SI 65536
 SF 400.2224715 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

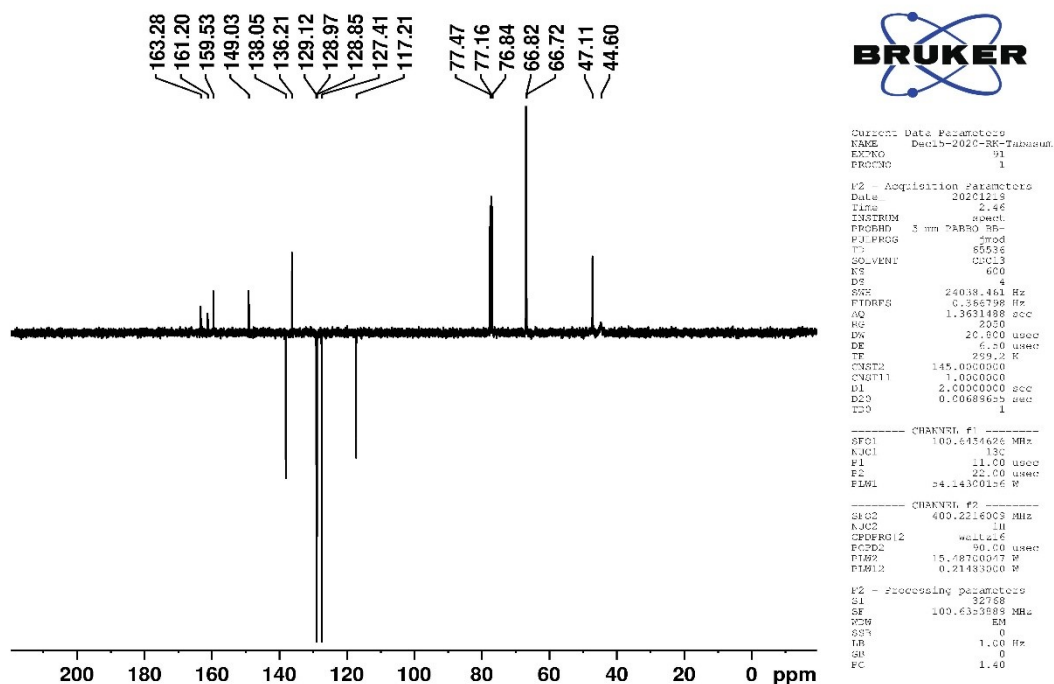


Mass spectrum showing relative intensity (Y-axis, 0.0E0 to 4.0E8) versus m/z (X-axis, 100 to 1200). The base peak is at m/z 153. Other labeled peaks include m/z 102, 152, 154, 155, 398, 426, and 480.

S65

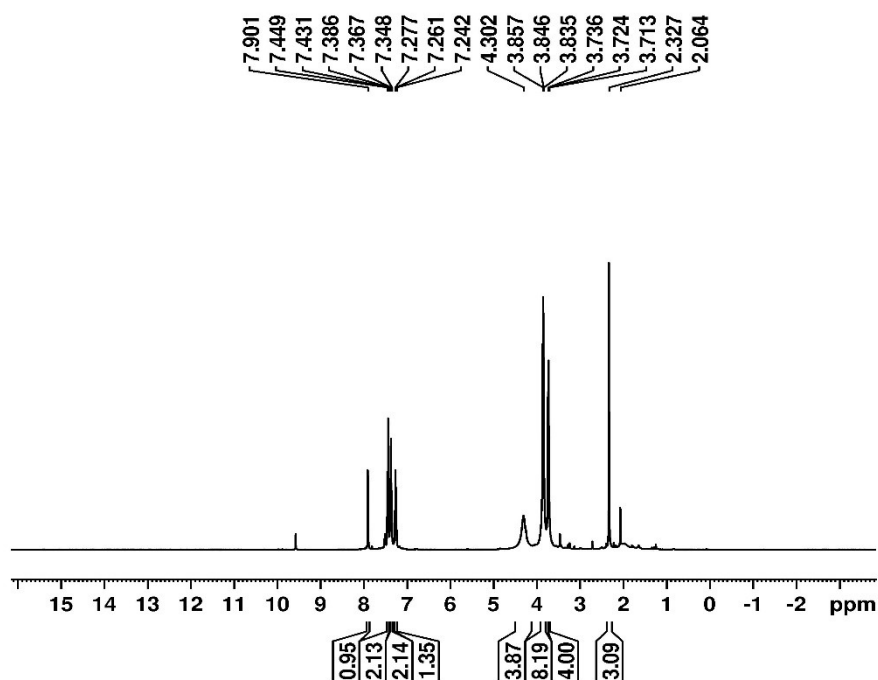
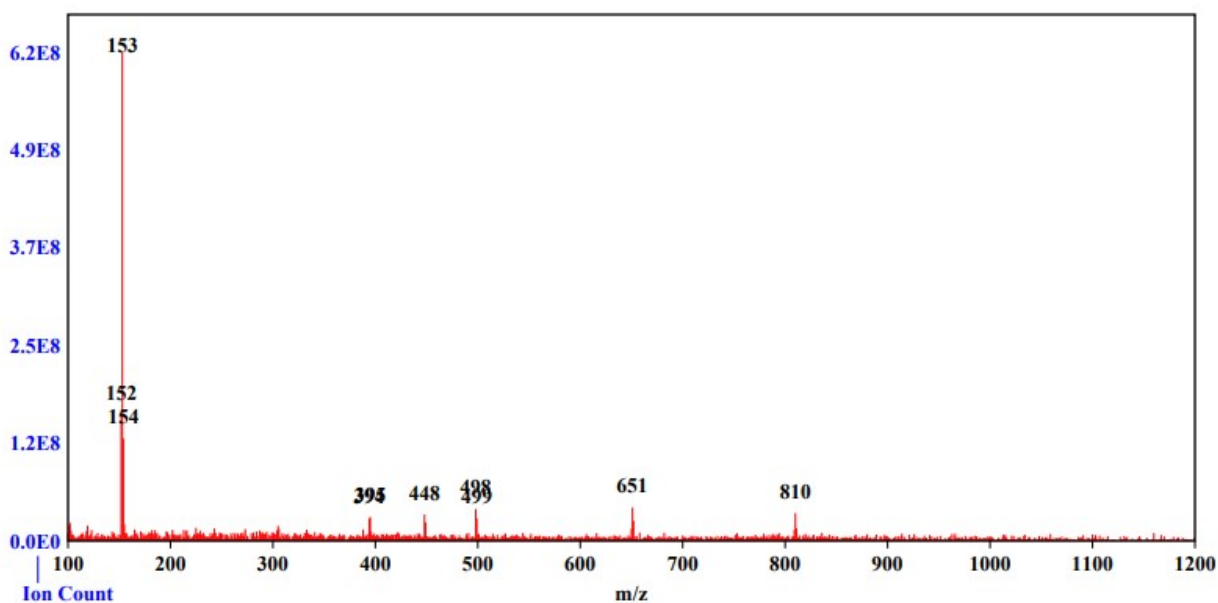


^1H NMR spectrum for compound **7a** (CDCl_3 , 400 MHz)



^{13}C NMR spectrum for compound **7a** (CDCl_3 , 100 MHz)

Spectrum Name: CN-01
 Start Ion: 100
 End Ion: 1200
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V

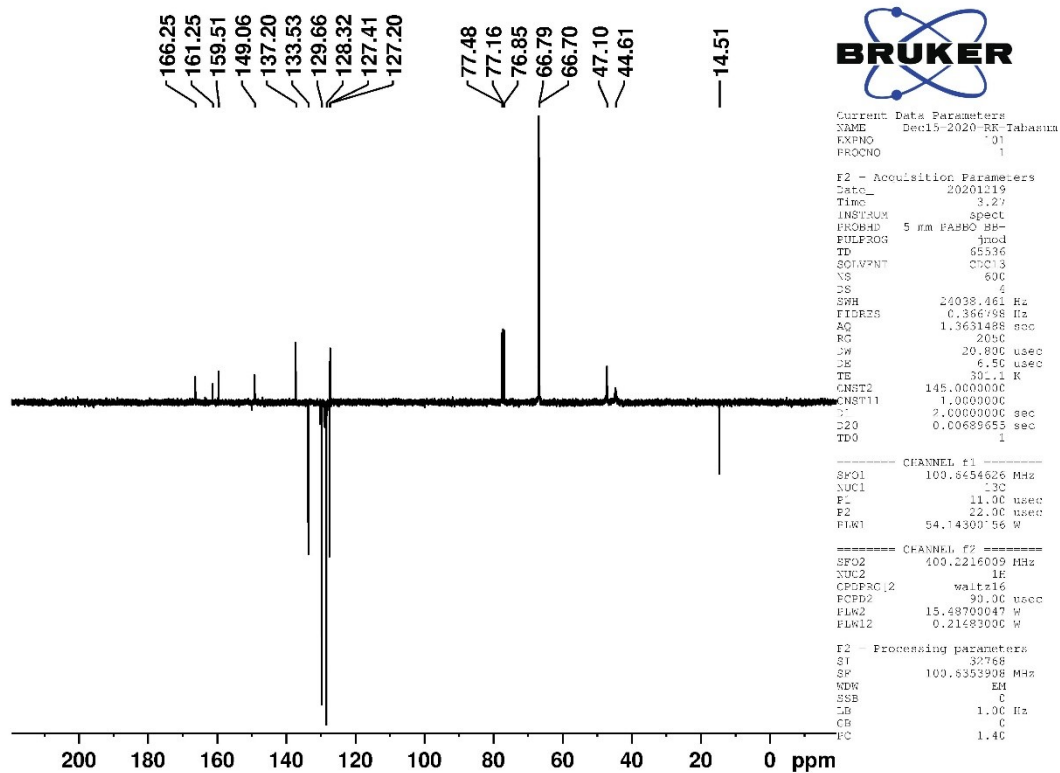


Current Data Parameters
 NAME Dec16-2020-RA-Tabasum
 EXPNO 100
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20201219
 Time 2.52
 INSTRUM spect
 PROBHD 5 mm PABBO B3-
 PULPROG zg30
 TD 65536
 SFO1 400.2226715 MHz
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 3002.820 Hz
 FIDRES 0.122265 Hz
 AQ 4.0894485 sec
 RG 128
 DW 62.400 usec
 DE 6.50 usec
 TE 298.7 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.2226715 MHz
 NUC1 1H
 P1 12.00 usec
 PLW1 16.29999924 W

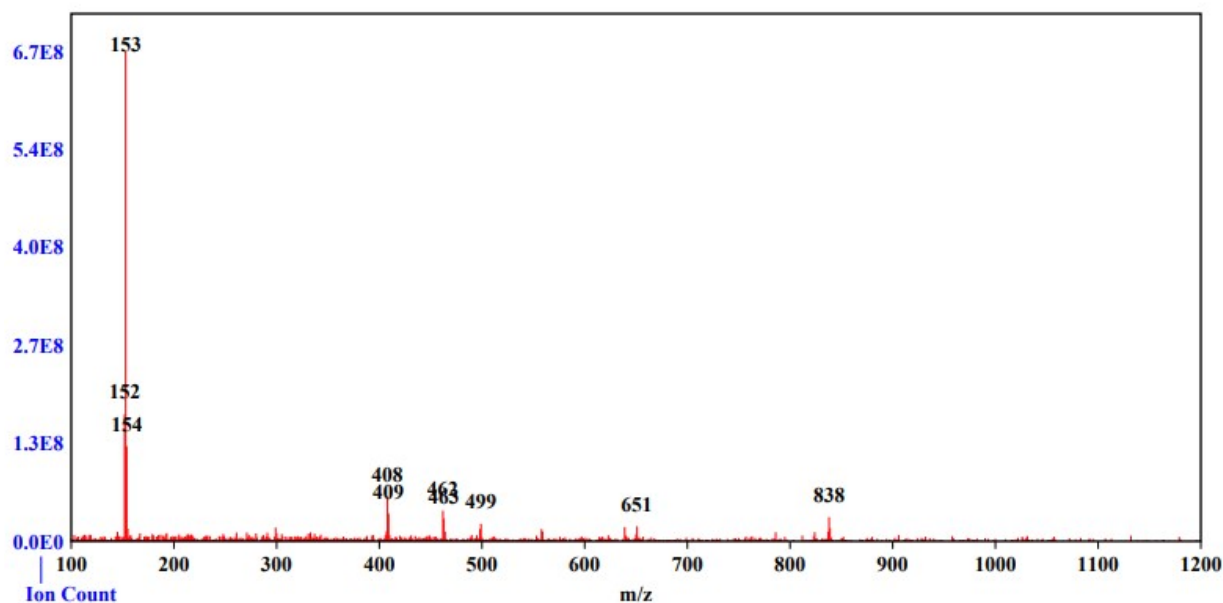
F2 - Processing parameters
 SI 65536
 SF 400.2200002 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



1

^{13}C NMR spectrum for compound **7b** (CDCl_3 , 100 MHz)

Spectrum Name: CN-02
 Start Ion: 100
 End Ion: 1200
 Source: ESI + 3.5kV 350C
 Capillary: 150V 300C Offset: 25V Span: 0V



Document: 10 May 2021 Batch 5a (VarioELcube) from: --.--- (modified)

INSTALLATION TEST - 25_03_2019

varioELcube serial number: 19181072

Text report

No	Name	Manuscript Compound ID	C [%]	H [%]	N [%]	S [%]
01	DIM_109	5d	54.40	5.771	21.95	0.719
02	DIM_110	5c	59.89	6.273	24.47	0.442
03	DIM_140	5b	65.02	6.722	26.58	0.332
04	DIM_401	6c	61.78	6.674	22.87	0.219
05	DS_02/06	1b	39.90	4.282	18.30	0.195
06	DS_101	3b	52.85	5.437	21.25	0.122
07	DS_201	4b	54.23	5.904	20.11	0.109
08	DIM_18	2k	55.58	5.398	25.27	0.131
09	DIM_25	2l	55.29	5.471	24.77	0.115
10	DIM_27	2m	53.65	4.930	24.33	0.146
11	DIM_106	5e	62.68	6.475	25.56	0.170
13	DIM_403	6b	63.64	6.931	23.50	0.214

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Mon May 10

18:34:43 2021 varioEL cube V4.0.16 (366251fb2)2018-07-25, CHNS Mode, Ser. No.:

19181072

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INSTALLATION TEST - 25_03_2019

varioELcube serial number: 19181072

Text report

No	Name	Manuscript Compound ID	C [%]	H [%]	N [%]	S [%]
14	DIM_01	2a	57.98	6.031	26.25	0.441
15	DIM_02	2h	51.50	5.592	23.28	0.126
16	DIM_03	2g	53.49	5.348	24.29	0.089
17-	DIM_04	2i	53.44	5.300	24.06	0.088
18	DIM_06	2n	54.30	5.720	29.70	0.062
19	DIM_10	2o	48.80	5.195	24.71	7.731

20	DIM_11	2j	47.83	4.752	21.49	0.125
21	DIM_15	2e	55.82	6.134	22.68	0.052
22	DIM_17	2f	53.88	6.034	21.50	0.041
23	DIM_19	2c	56.85	6.081	24.25	0.026
24	DIM_20	2d	56.57	6.152	24.28	0.020
25	DIM_16	2b	58.31	6.417	25.01	0.015
26	CN_01	7a	59.83	6.184	23.93	0.017
27	CN_02	7b	59.25	6.630	22.84	0.014
28	DIM_113	5a	65.16	7.236	26.76	0.009
29	DIM_404	6a	66.13	7.888	24.51	0.009
30	DS_103	3c	68.09	6.875	19.84	0.013
31	DS_203	4c	67.22	7.209	18.89	0.011
32	DS_202	4d	64.64	7.752	22.64	0.016
33	M_PIP		58.72	9.272	31.73	0.010

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Mon Apr 12

07:45:18 2021 varioEL cube V4.0.16 (366251fb2)2018-07-25, CHNS Mode, Ser. No.:

19181072

Elementar Analysensysteme GmbH

Parameter report

Temperatures		
	Pyrol.tube	1170
	CO col.standby	40
	Desorpt.Mid.	40
	Cool temp	60
Time values		

Figure S2. Elemental analysis report