# Supporting Information

# Facile access to 3,5-disubstituted 1,2,4-thiadiazoles via T3P® mediated oxidative dimerization of thioamides

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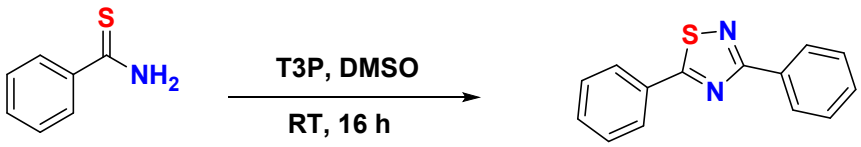
## Contents

## General Information:

Dry solvents were purchased from chemical suppliers and used without further purification. Analytical thin-layer chromatography (TLC) was performed on commercially available Merck TLC Silica gel 60 $F_{254}$. Silica gel column chromatography was performed on silica gel 60 (spherical 100-200 μm). IR spectra were recorded on Perkin-Elmer FT/IR-4000 using ATR.$^{1}$H NMR spectra were recorded on Varian-400 (400 MHz) spectrometer. Chemical shifts of $^{1}$H NMR spectra were reported relative to tetra methyl silane ($^{13}$C NMR spectra were recorded on Varian-400 (100 MHz) spectrometer. Chemical shifts of $^{13}$C NMR spectra were reported to relative to $CDCl_3$ (77.16) and DMSO-$d_6$ (39.5). Splitting patterns were reported as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad.

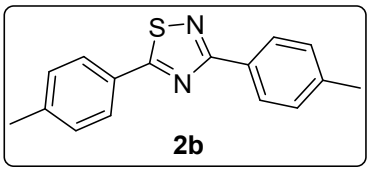
**General Procedure:** ***Preparation of 3,5-diphenyl-1,2,4-thiadiazole (2a)[1]***




**1a** **2a**

To a stirred solution of compound **1a** (10 g, 72.992 mmol) in DMSO (250 ml) was added 50% $T_3P$ in ethyl acetate solution (46.4 ml, 72.992 mmol) at 25°C and the reaction mixture was stirred at 25°C for 16 h. The progress of the reaction was monitored by TLC, the reaction mixture was poured in to ice cold water (250 ml) and ethyl acetate (500 ml). The organic layer was separated and washed with water (200 ml) and brine solution (200 ml), dried over anhydrous sodium sulphate, filtered and concentrated under reduced pressure to afford the crude compound. The crude compound was purified by flash column chromatography (silica gel; 20% ethyl acetate/pet ether) to give the pure compound **2a** (8.45 g, 97%) as an off white solid.; m.p. 89-90°C (Reported 87-89 $^0$C)1 ; $^1$H NMR (400 MHz, DMSO-$d_6$) δ = 6.31-8.34 (m, 2H), 6.14 (dd, *J* = 1.6 Hz, 2H), 7.56 - 7.67 (m, 6H) ($^1$H-NMR data matching with the literature value)1 ; $^{13}$C NMR (100 MHz, DMSO-$d_6$) = 188.0, 172.8, 132.5, 132.1, 130.7, 129.7, 129.6, 129.0, 127.8, 127.4; MS (EI): *m/z* = 239.42 (M+1,100).

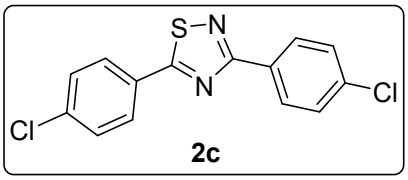
***3,5-di-p-tolyl-1,2,4-thiadiazole (2b)[1]***




**2b**

The title compound was prepared from 4-methylbenzothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (165 mg, 93%),; m.p. 128-130°C ; $^1$H NMR (400 MHz, DMSO-$d_6$) δ = 8.20 (d, *J* = 6.8 Hz, 2H), 8.01 (d, *J* = 6.8 Hz, 2H), 7.43 (d, *J* = 6.4 Hz, 2H), 7.38 (d, *J* = 6.8 Hz, 2H), 2.41 (s, 3H), 2.39 (s, 3H),; $^{13}$C NMR (100 MHz, DMSO-$d_6$) = 187.7, 172.7, 142.7, 140.4, 130.0, 129.5, 129.52, 127.8 127.3, 127.1, 21.1, 21.0; MS (EI): *m/z* = 267.02 (M+1,100).

***3,5-bis(4-chlorophenyl)-1,2,4-thiadiazole (2c)[2,3,5]***

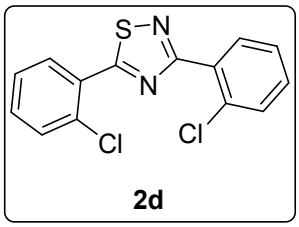



**2c**

The title compound was prepared from 4-chlorobenzothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (161 mg, 90%),; m.p. 150-152°C; $^1$H NMR (400 MHz, $CDCl_3$) δ = 8.31 (d, *J*

= 8.4 Hz, 2H), 7.97 (d, *J* = 8.8 Hz, 2H), 7.50 (d, *J* = 8.8 Hz, 2H), 7.7.47 (d, *J* = 8.8 Hz, 2H) ; $^{13}$C NMR (100 MHz, $CDCl_3$) = 187.0, 172.7, 138.1, 136.5, 131.1, 130.2, 129.63, 129.60, 128.9, 128.6,; MS (EI): *m/z* = 307.34 (M+1,100).

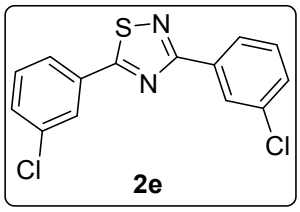
***3,5-bis(2-chlorophenyl)-1,2,4-thiadiazole (2d)[1,3]***




**2d**

The title compound was prepared from 2-chlorobenzothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (158 mg, 88%),; m.p. 84-86°C; $^1$H NMR (500 MHz, DMSO-$d_6$) δ = 8.55 (dd, *J* = 1.0 Hz, 1.5 Hz, 1H), 8.05 (dd, *J* = 1.0 Hz, 1.5 Hz, 1H), 7.82 (d, *J* = 7.5 Hz, 1H), 7.70-7.53 (m, 5H),; $^{13}$C NMR (125 MHz, DMSO-$d_6$) = 182.6, 169.1, 133.3, 132.7, 132.2, 131.9, 131.6, 131.2, 130.6, 130.68, 130.2, 128.4, 128.2, 127.4,; MS (EI): *m/z* = 306.91 (M+1,100).

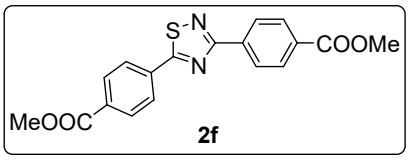
***3,5-bis(3-chlorophenyl)-1,2,4-thiadiazole (2e)[2]***




**2e**

The title compound was prepared from 3-chlorobenzothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (161 mg, 90%),; m.p. 121-123°C; $^1$H NMR (400 MHz, DMSO-$d_6$) δ = 8.27 (s, 1H), 8.25 (d, *J* = 7.5 Hz, 1H), 8.19 (s, 1H), 8.09 (d, *J* = 8.0 Hz, 1H), 8.72-7.57 (m, 4H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) = 186.8, 171.2, 134.2, 133.781, 133.768, 132.2, 131.5, 131.3, 131.1, 130.6, 127.4, 126.8,126.5,126.4; MS (EI): *m/z* = 307.39 (M+1,100).

***Dimethyl 4,4'-(1,2,4-thiadiazole-3,5-diyl)dibenzoate (2f)[7]***

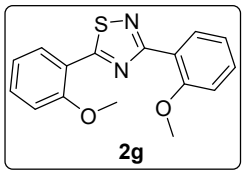



**2f**

The title compound was prepared from methyl 4-carbamothioylbenzoate (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (108 mg, 59%),; m.p. 253-255°C,; $^1$H NMR (400 MHz, $CDCl_3$) δ = 8.83 (d, *J* = 8.0 Hz, 2H), 8.47 (d, *J* = 8.0 Hz, 1H), 8.25 - 8.12 (m, 4H), 3.99 (Br s, 6H), 1.25 (s, 1H, Extra peak), 0.84 (m, 0.24H, Extra

peak) ; $^{13}$C NMR (100 MHz, DMSO-$d_6$) = 187.2, 171.2, 166.6, 166.5, 166.0, 139.6, 133.8, 130.5, 130.0, 129.9, 128.9, 128.3, 127.4, 52.4, 29 (Extra peak),; MS (EI): *m/z* = 355.43 (M+1,100).

***3,5-bis(2-methoxyphenyl)-1,2,4-thiadiazole (2g)[3]***

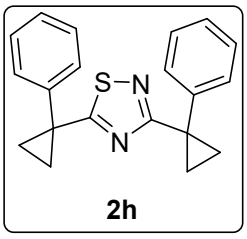



**2g**

The title compound was prepared from 2-methoxybenzothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (161 mg. 90%),; m.p. 123-125°C; $^1$H NMR (400 MHz, DMSO-$d_6$) δ = 3.85 (s, 3H), 4.14 (s, 3H), 7.12 (t, *J* = 7.6 Hz, 1H), 7.23 (m, 2H), 7.38 (d, *J* = 8.4 Hz, 1H), 7.53 (td, *J* = 8.0 Hz, *J* = 1.6 Hz), 7.66 (td, *J* = 8.0 Hz, *J* = 1.6 Hz), 7.90 (dd, *J* = 7.6 Hz, 1.6 Hz, 1H), 8.38 (dd, *J* = 7.6 Hz, *J* = 1.6 Hz, 1H),; $^{13}$C NMR (100 MHz, DMSO-$d_6$) = 179.9, 169.1, 157.5, 157.3, 133.3, 131.4, 131.3, 127.5, 122.4, 121.3, 120.2, 118.8, 112.5, 112.25, 56.3, 55.8; MS (EI): *m/z* = 299.01 (M+1,100).

Note: At 7.23 ppm two peaks were merged

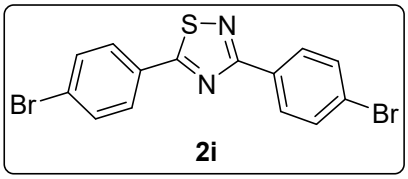
***3,5-bis(1-phenylcyclopropyl)-1,2,4-thiadiazole (2h):***




**2h**

The title compound was prepared from 1-phenylcyclopropane-1-carbothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (125 mg, 69%),; m.p. 115-117°C; $^1$H NMR (500 MHz, CDCl3) δ = 1.36 (q, *J* = 4.0 Hz, 2H), 1.50 (q, *J* = 4.0 Hz, 2H), 1.68 (q, *J* = 4.0 Hz, 2H), 1.80 (q, *J* = 4.0 Hz, 2H), 7.47 - 7.24 (m, 10H), ; $^{13}$C NMR (100 MHz, DMSO-$d_6$) = 198.1, 178.9, 141.4, 140.3, 130.2, 129.8, 128.9, 128.4, 128.0, 126.6, 28.8, 28.6, 20.2, 16.8,; MS (EI): *m/z* = 319.09 (M+1,100).

***3,5-bis(4-bromophenyl)-1,2,4-thiadiazole (2i)[2,5]***

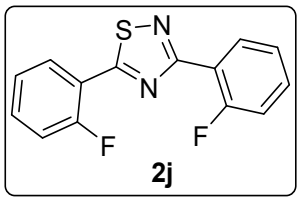



**2i**

The title compound was prepared from 4-bromobenzothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (165 mg, 90%),; m.p. 158-160°C; $^1$H NMR (400 MHz, $CDCl_3$) δ = 8.24 (d,

*J* = 8.8 Hz, 2H), 7.90 (d, *J* = 8.8 Hz, 2H), 7.67 (d, *J* = 8.8 Hz, 2H), 7.63 (d, *J* = 8.8 Hz, 2H),; $^{13}$C NMR (100 MHz, $CDCl_3$) = 187.1, 172.8, 132.5, 131.9, 131.5, 129.8, 129.3, 128.8, 126.5, 125.0,; MS (EI): *m/z* = 395.25 (M+1,100).

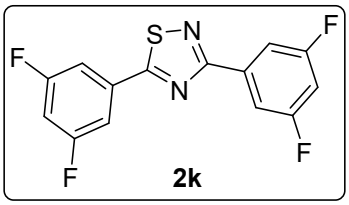
***3,5-bis(2-fluorophenyl)-1,2,4-thiadiazole (2j)[4]***




**2j**

The title compound was prepared from 2-Fluorobenzothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (155 mg, 88%),; m.p. 113-115°C; $^1$H NMR (400 MHz, DMSO-$d_6$) δ = 7.46-7.39 (m, 2H), 7.53 (td, *J* = 1.2 Hz, *J* = 1.2 Hz, *J* = 1.2 Hz, 1H), 7.67 – 7.56 (m, 2H), 7.75 – 7.71 (m, 1H), 8.30 (td, *J* = 2.0 Hz, *J* = 2.0 Hz, *J* = 1.6, 1H), 8.42 (td, *J* = 1.6 Hz, *J* = 1.6 Hz, *J* = 1.6 Hz, 1H) ; $^{13}$C NMR (100 MHz, DMSO-$d_6$) = 179.59 ($^3$J-CF : d, 4.4Hz), 167.63 ($^3$J-CF : d, 5.1Hz), 158.70 ($^1$J-CF : d, 253.60Hz), 159.39 ($^1$J-CF : d, 250.10Hz), 134.37 ($^3$J-CF : d, 9.0Hz), 132.65 ($^3$J-CF : d, 8.5Hz), 131.64, 128.36, 125.74 ($^4$J-CF : d, 2.8Hz), 124.75 ($^4$J-CF : d, 3.5Hz), 120.03 ($^2$J-CF : d, 102Hz), 117.60 ($^2$J-CF : d, 12.1Hz), 116.81 ($^2$J-CF : d, 21.2Hz), 116.31 ($^2$J-CF : d, 20.6Hz),; MS (EI): *m/z* = 275.07 (M+1,100).

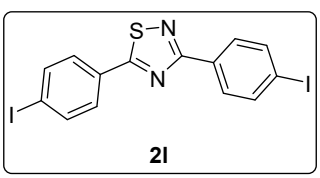
***3,5-bis(3,5-difluorophenyl)-1,2,4-thiadiazole (2k):***




**2k**

The title compound was prepared from 3,5-difluorobenzothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid, Yield (152 mg, 84%),; m.p. 168-172°C; $^1$H NMR (400 MHz, $CDCl_3$) δ = 7.90 (dd, *J* = 2.0 Hz, *J* = 2.4 Hz, 2H), 7.58 (dd, *J* = 2.0 Hz, *J* = 2.4 Hz, 2H), 7.04 (tt, *J* = 8.4 Hz, *J* = 2.4 Hz, 1H), 6.96 (tt, *J* = 8.80 Hz, *J* = 2.4 Hz, 1H),; $^{13}$C NMR (100 MHz, $CDCl_3$) = 186.1, 171.67, 164.37, 161.91 (dd, $^1$J-CF: 246.80Hz, 12.40Hz), 162.064, 164.56 (dd, $^1$J-CF: 246.80Hz, 12.40Hz), 135.27 (t, $^3$J-CF: 10.0 Hz), 132.86 (t, $^3$J-CF: 10.0 Hz), 111.28 (dd, $^2$J-CF: 26.0 Hz, 7.40 Hz), 110.59 (dd, $^2$J-CF: 27.0 Hz, 8.0 Hz), 107.38 (t, $^2$J-CF: 25.2 Hz), 105.96 (t, $^2$J-CF: 25.2 Hz);. MS (EI): *m/z* = 311.38 (M+1,100).

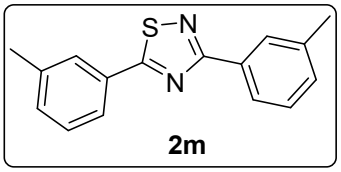
***3,5-bis(4-iodophenyl)-1,2,4-thiadiazole (2l)[4]***




**2l**

The title compound was prepared from 4-iodobenzothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (160 mg, 86%),; m.p. 232-235°C; $^1$H NMR (500 MHz, CDCl3) δ = 8.11 (d, *J* = 8.4 Hz, 2H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.85 (d, *J* = 8.8 Hz, 2H), 7.77 (d, *J* = 8.4 Hz, 2H),; $^{13}$C NMR (100 MHz, $CDCl_3$) = 187.3, 173.0, 138.5, 138.0, 137.9, 132.1, 129.9, 128.7, 98.77, 97.23; MS (EI): *m/z* = 491.22 (M+1,100).

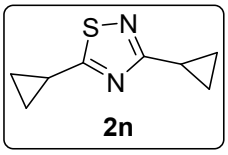
***3,5-di-m-tolyl-1,2,4-thiadiazole (2m)[3,5]***



**2m**

The title compound was prepared from 3-methylbenzothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (153 mg, 86%),; m.p. 53-55°C; $^1$H NMR (500 MHz, DMSO-$d_6$) δ = 8.13 (br s, 1H), 8.11 (d, *J* = 7.5 Hz, 1H), 7.93 (br s, 1H), 7.90 (dd, *J* = 7.5 Hz, 1.98 Hz, 1H), 7.51-7.43 (m, 3H), 7.37 (d, *J* = 7.5 Hz, 1H), 2.435 (s, 3H), 2.431 (s, 3H),; $^{13}$C NMR (125 MHz, DMSO-$d_6$) = 188.0, 172.9, 139.1, 138.2, 133.0, 132.0, 131.3, 129.6, 129.4, 128.8, 128.3, 127.6, 125.0, 124.5, 20.9, 20.7,; MS (EI): *m/z* = 267.24 (M+1,100).

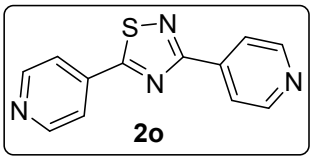
***3,5-dicyclopropyl-1,2,4-thiadiazole (2n)[6]***



**2n**

The title compound was prepared from cyclopropanecarbothioamide (200 mg) according to the general procedure and purified by column chromatography to give pale yellow liquid, Yield (106 mg, 64%); $^1$H NMR (500 MHz, DMSO-$d_6$) δ = 2.60-2.57 (m, 1H), 2.23-2.19 (m, 1H), 1.25-1.21 (m, 2H), 1.04-0.92 (m, 6H); $^{13}$C NMR (125 MHz, DMSO-$d_6$) = 193.8, 177.2, 13.2, 12.3, 12.1, 10.86 (Extra peak), 9.0; MS (EI): *m/z* = 167.0932 (M+1,100).

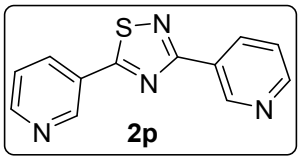
***3,5-di(pyridin-4-yl)-1,2,4-thiadiazole (2o)[5]***



**2o**

The title compound was prepared from pyridine-4-carbothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (104 mg, 60%),; m.p. 88-90°C; $^1$H NMR (400 MHz, CDCl3) δ = 8.83 (dd, *J* = 1.6 Hz, *J* = 1.6 Hz, 4H), 7.55 (dd, *J* = 1.6 Hz, *J* = 1.6 Hz, 4H), 2.62 (DMSO solvent present),; $^{13}$C NMR (100 MHz, DMSO-$d_6$) = 186.8, 171.0, 151.1, 150.8, 138.2, 135.9, 121.6, 121.1,; MS (EI): *m/z* = 241.07 (M+1,100).

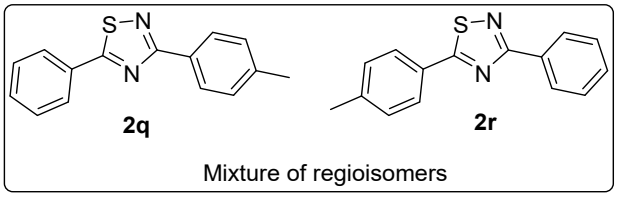
***3,5-di(pyridin-4-yl)-1,2,4-thiadiazole (2p)[1,3]***



**2p**

The title compound was prepared from pyridine-3-carbothioamide (200 mg) according to the general procedure and purified by column chromatography to give off white solid,; Yield (130 mg, 75%),; m.p. 129-131°C; $^{1}$H NMR (500 MHz, DMSO-$d_6$) δ = 9.48 (d, *J* = 1.5 Hz, 1H), 9.33 (d, *J* = 2.0 Hz, 1H), 8.83 (dd, *J* = 1.5 Hz, *J* = 2.0 Hz, 1H), 8.77 (dd, *J* = 1.5 Hz, *J* = 1.5 Hz, 1H), 8.64 (dt, *J* = 2.0 Hz, *J* = 2.0 Hz, 1H), 8.54 (dt, *J* = 2.0 Hz, *J* = 2.0 Hz, 1H), 7.68-7.67 (m, 1H), 7.66-7.63 (m, 1H), $^{13}$C NMR (125 MHz, DMSO-$d_6$) = 182.8, 170.6, 152.9, 151.4, 148.7, 148.0, 135.2, 135.0, 127.7, 125.8, 124.4, 124.1,; MS (EI): *m/z* = 240.90 (M+1,100).

***5-phenyl-3-(p-tolyl)-1,2,4-thiadiazole (2q+2r):***



**2q** **2r**

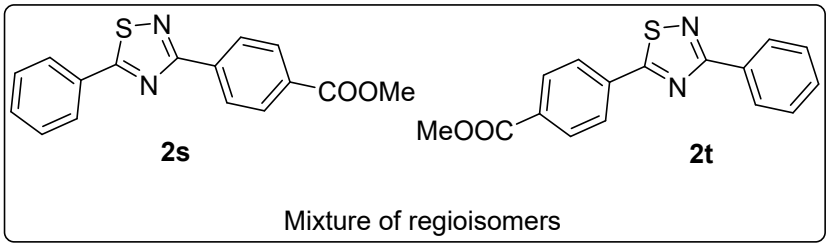
Mixture of regioisomers

The title compound was prepared from Benzothioamide (**1a**, 100 mg) and 4-methylbenzothioamide (**1b**) (110 mg) according to the general procedure and purified by column chromatography to give the mixture of (**2q+2r**) as an off white solid,; Yield (49 mg mixture of 2q & 2r, 27%), 28% of compound **2a** and 30% of compound **2b** ; $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ = 2.41 (d, *J* = 5.6 Hz, 3H), 7.43 (dd, *J* = 8.0 Hz, *J* = 8.0 Hz, 2H), 7.66-7.56 (m, 3H), 8.02 (d, *J* = 8.0 Hz, 1H), 8.12 (dd, *J* = 1.6 Hz, *J* = 1.6 Hz, 1H), 8.22 (d, *J* = 8.4 Hz, 1H), 8.32-8.30 (m, 1H),; $^{13}$C NMR (100 MHz, DMSO-$d_6$) = 187.9, 187.8, 172.9, 172.7, 142.7, 140.5, 132.4, 132.1, 130.6, 130.1, 129.7, 129.57, 129.55, 128.9, 127.8, 127.37, 127.33, 127.1, 21.1, 21.0,; MS (EI): *m/z* = 252.99 (M+1,100).

**Note: The separation of 2q and 2r by prep HPLC was not successful. So, it was not possible to assign the peaks in the mixtures as the difference between 2q and 2r is the phenyl substitution at the 3 and 5 position of thiadiazole ring. The $^{1}$H-NMR and $^{13}$C NMR clearly shows mixture of regioisomers.**

**HPLC ratio is 56.8:43.92 (~3:2)**

***methyl 4-(5-phenyl-1,2,4-thiadiazol-3-yl) benzoate (2s+2t):***



**2s** **2t**

Mixture of regioisomers

The title compound was prepared from Benzothioamide (**1a**, 100 mg) and compound **1f** according to the general procedure and purified by column chromatography to give the mixture of (**2s+2t**) as an off white solid,; Yield (64 mg mixture of 2s & 2t, 30%),

27% of compound **2a** and 28% of compound **2f** off white solid,; $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ = 3.90 (s,3H), 7.69-7.56 (m, 3H), 8.15-8.11 (m, 3H), 8.25 (d, *J* = 8.4 Hz, 1H), 8.32-8.30 (m, 1H), 8.42 (d, *J* = 8.4 Hz, 1H),; $^{13}$C NMR (100 MHz, DMSO-$d_6$) = 188.4, 186.7, 173.0, 171.7, 165.7,165.3, 135.8, 133.4, 132.6, 131.9, 131.1, 130.8, 130.2, 129.8, 129.6, 129.5, 129.0, 128.7, 128.1, 127.9, 52.4, 52.3,; MS (EI): *m/z* = 297.47 (M+1,100).

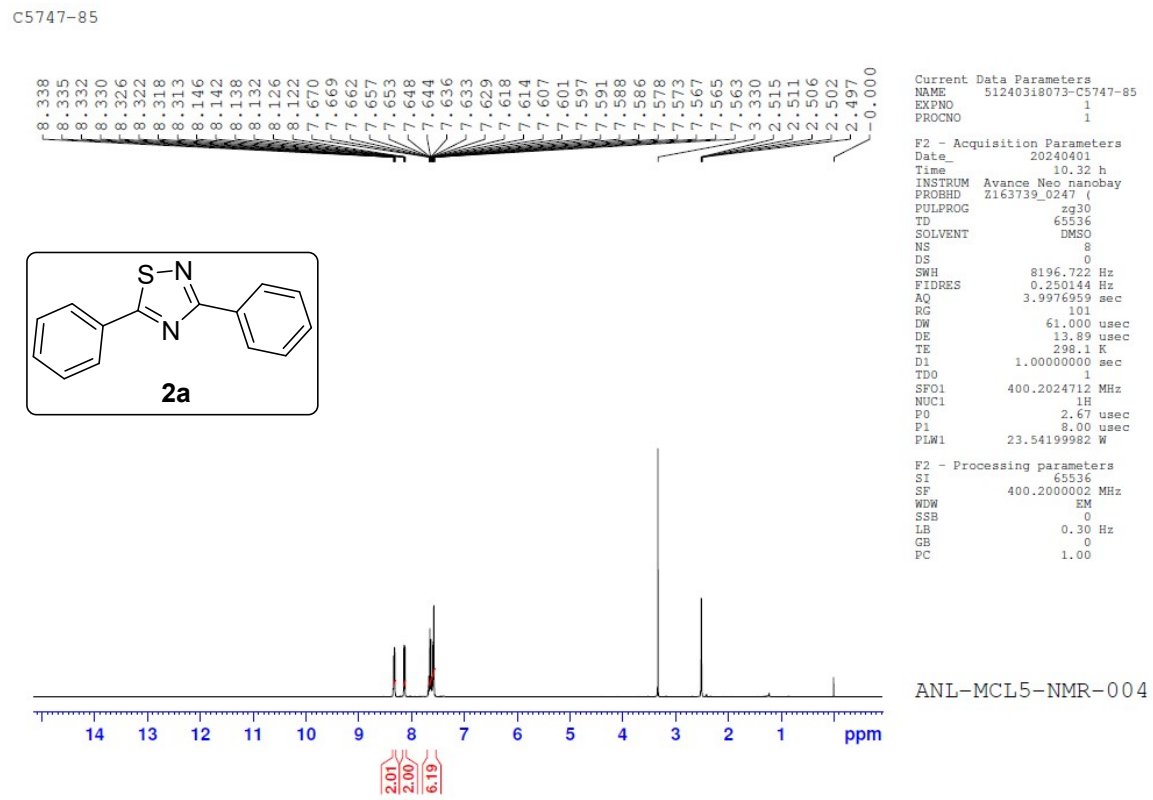
**Note: The separation of 2s and 2t by prep HPLC was not successful. So, it was not possible to assign the peaks in the mixtures as the difference between 2s and 2t is the phenyl substitution at the 3 and 5 position of thiadiazole ring. The $^{1}$H-NMR and $^{13}$C NMR clearly shows mixture of regioisomers.**

**HPLC ratio is 62.68:37.32 (~2:1)**

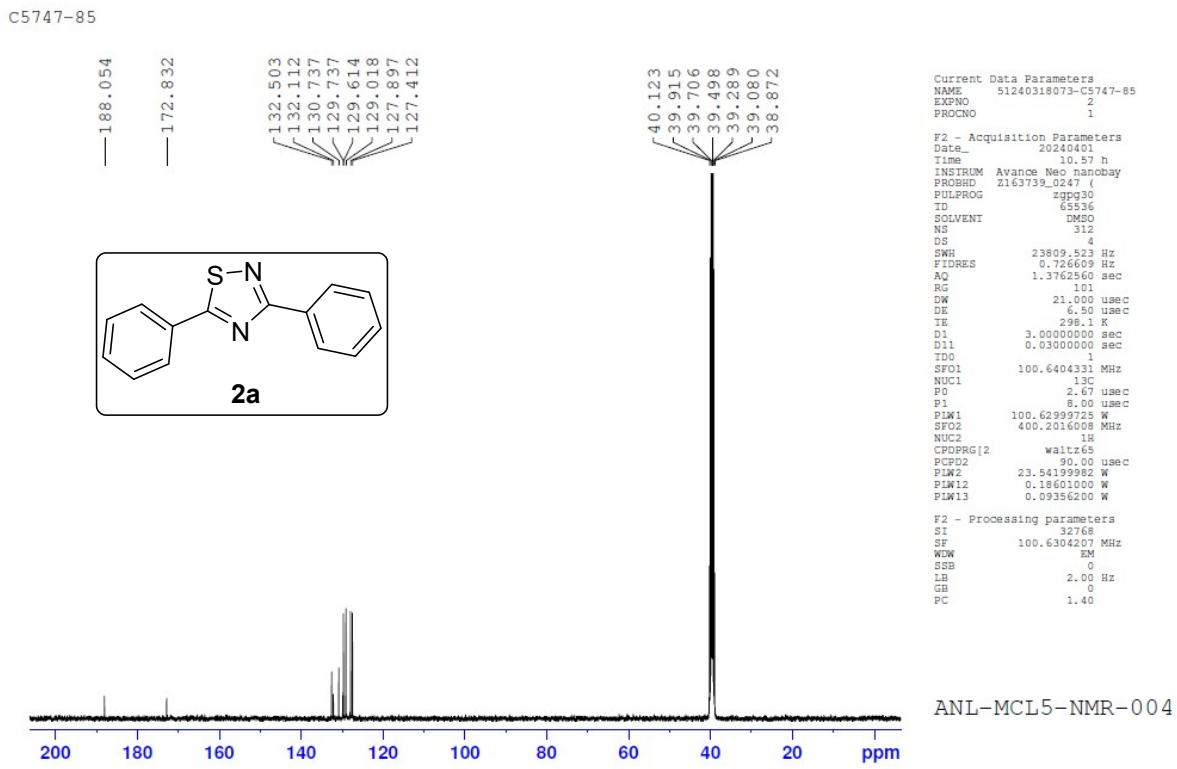
**References:**


1. C. P. Pravin, S. B. Dinesh, S. D. Prasad, G. A. Krishnacharya, *Tetrahedron Lett*. 2009, 50, 5820–5822
2. C. Dong-Ping, C. Zhen-Chu, *Synth. Commun.*2002, 32, 2155-2159
3. X. Yali, C. Jiuxi, G. Wenxia, J. Huile, D. Jinchang, W. Huayue, *J. Chem. Res.* 2010, 34, 151-153
4. a) S. M. Abdelrahman, M. Laura, P. K. Tamara, P. Eun-Jung, M. P. John, C. Mark, *Bioorg. Med. Chem.* 2012, 20, 510–520 b) L. Zhuo, S. H. Xie, H. Wang and Prof. H. J. Zhu, *Eur. J. Org. Chem.* 2021, 23, 3398-3402
5. S. Manik, A. P. Cameron, K. J. Stacey, G. B. Sophia, M. D. Katherine, F. B. Kyle, *J. Am. Chem. Soc.* 2025, 147, 10698−10705
6. Q. Huang, J. Liu, J. Wan, *Org. Lett.* 2024, 26, 5263−5268
7. Anais da Academia Brasileira de Ciencias (1963), 35(2), 197-201

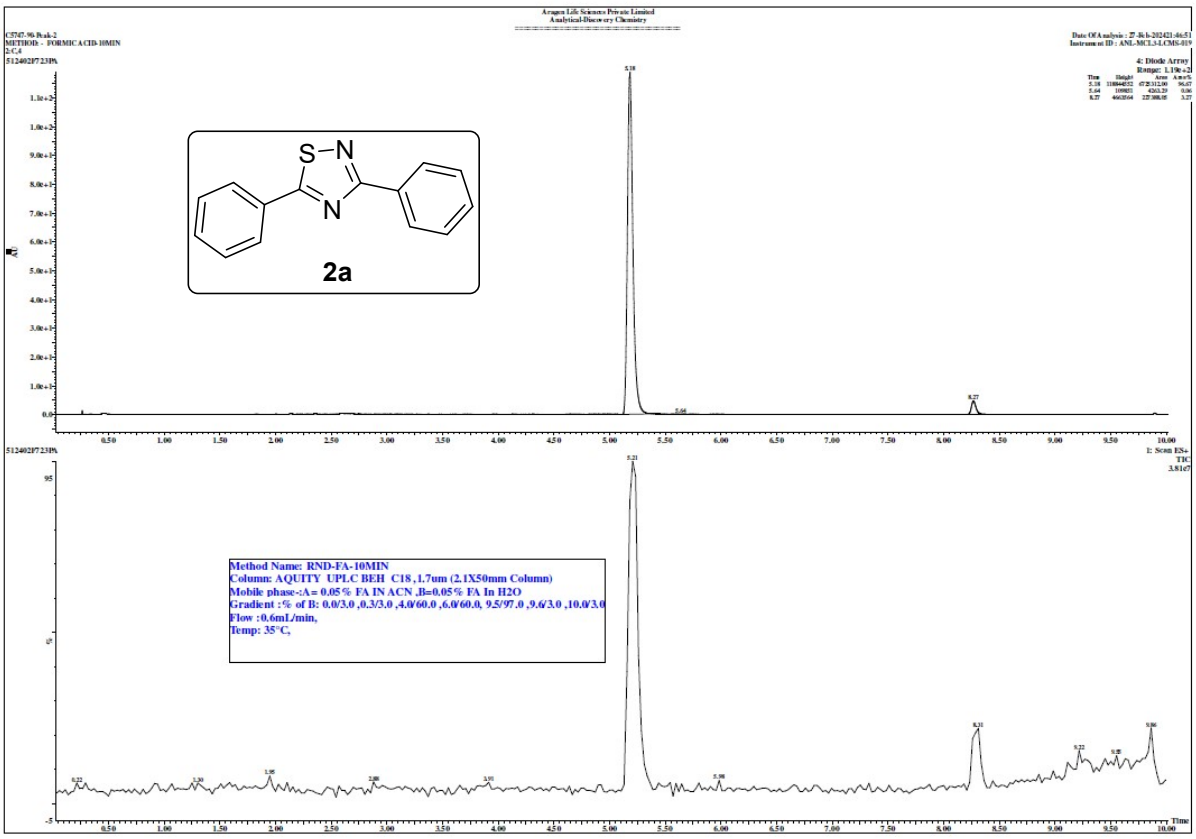
**$^{1}$H NMR spectrum (400 MHz) of Compound (2a) in DMSO-$d_6$**

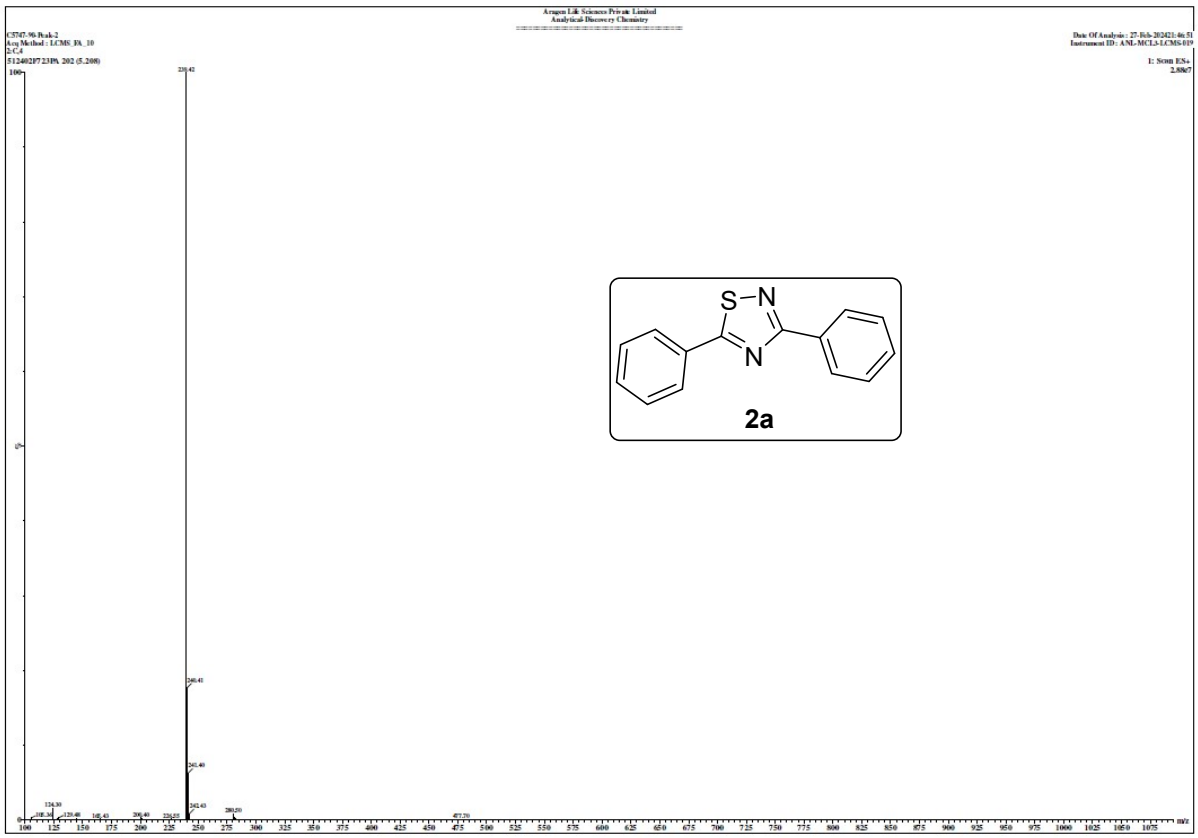



**$^{13}$C NMR spectrum (100 MHz) of Compound (2a) in DMSO-$d_6$**

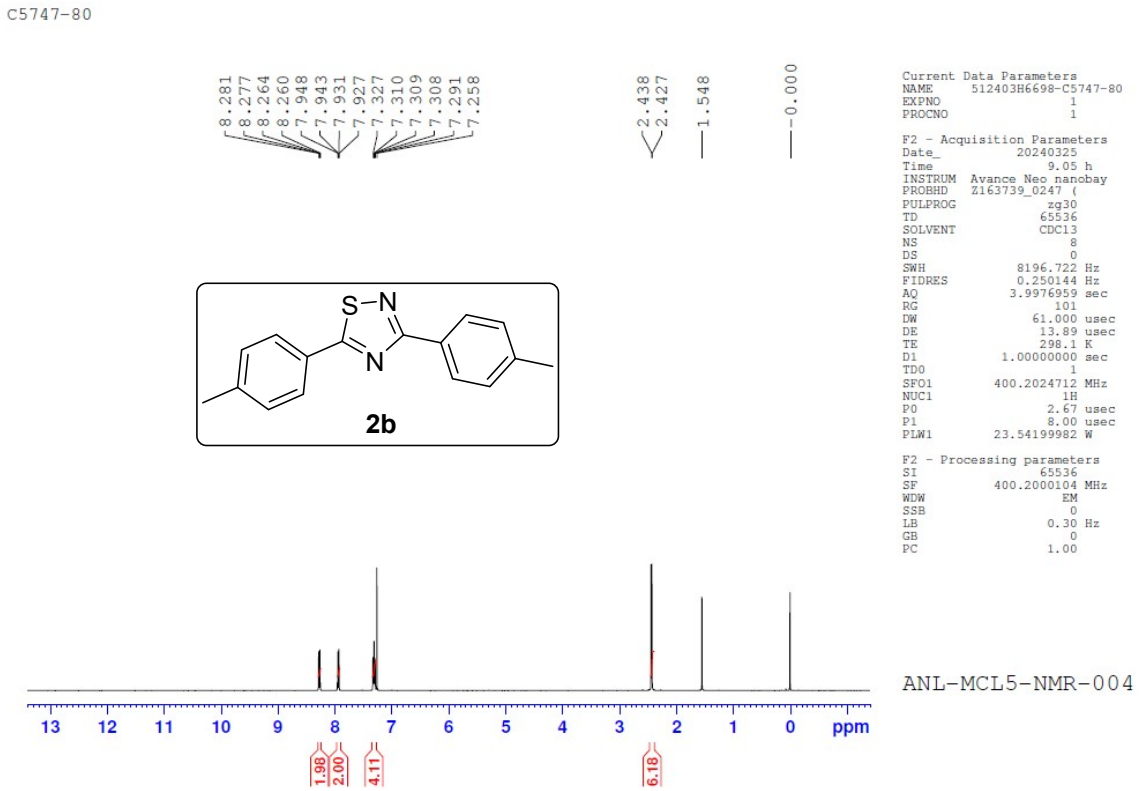



**LCMS spectrum of Compound (2a)**

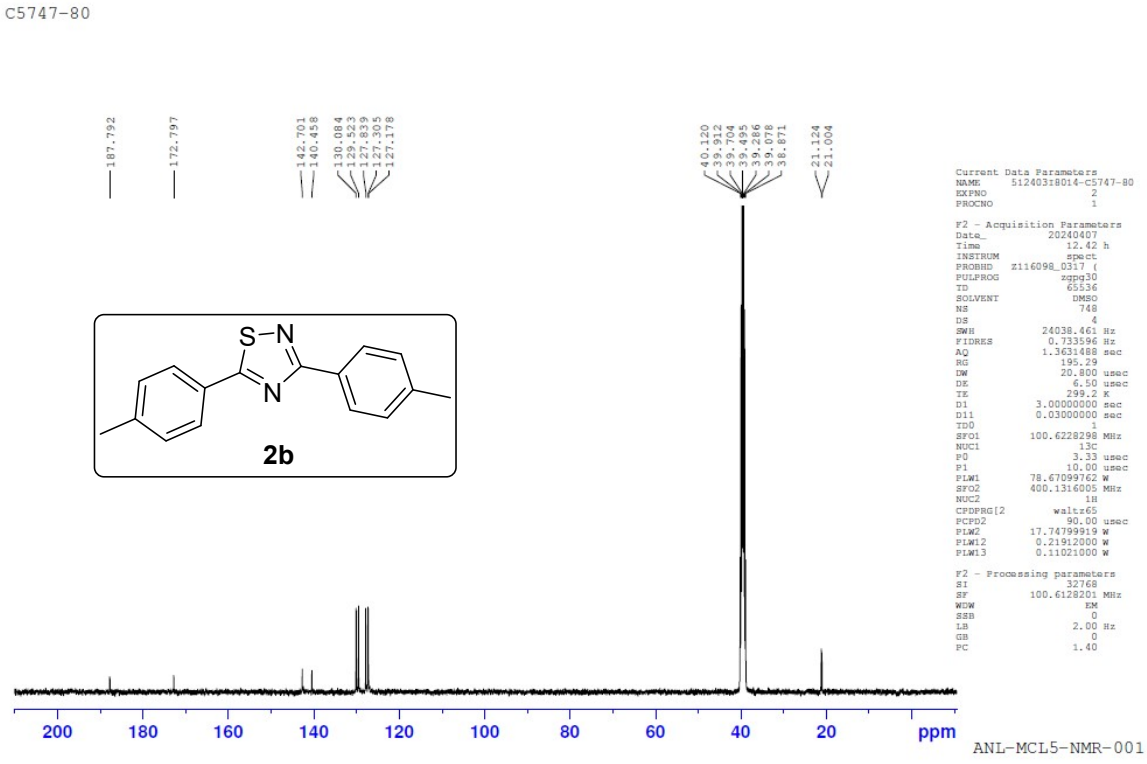


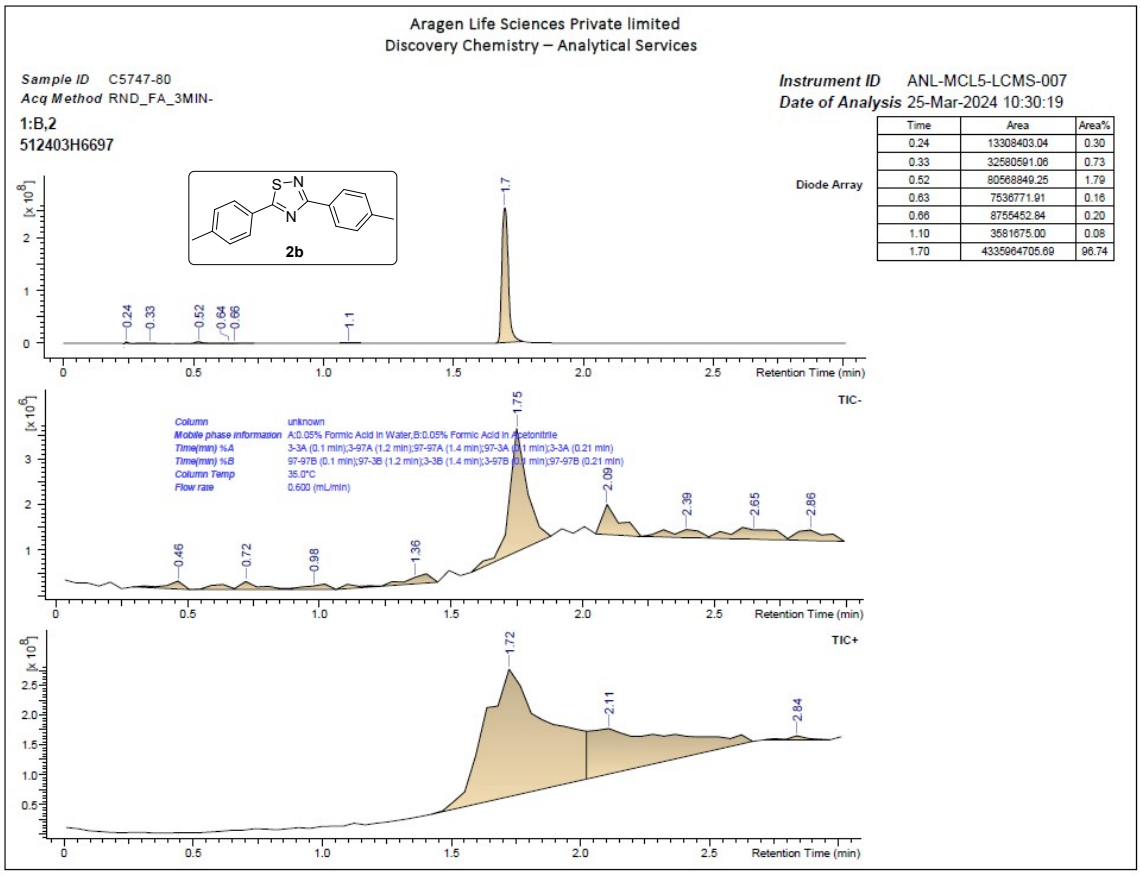

**$^{1}$H NMR spectrum (400 MHz) of Compound (2b) in DMSO-$d_6$**




**$^{13}$C NMR spectrum (100 MHz) of Compound (2b) in DMSO-$d_6$**




**LCMS spectrum of Compound (2b)**

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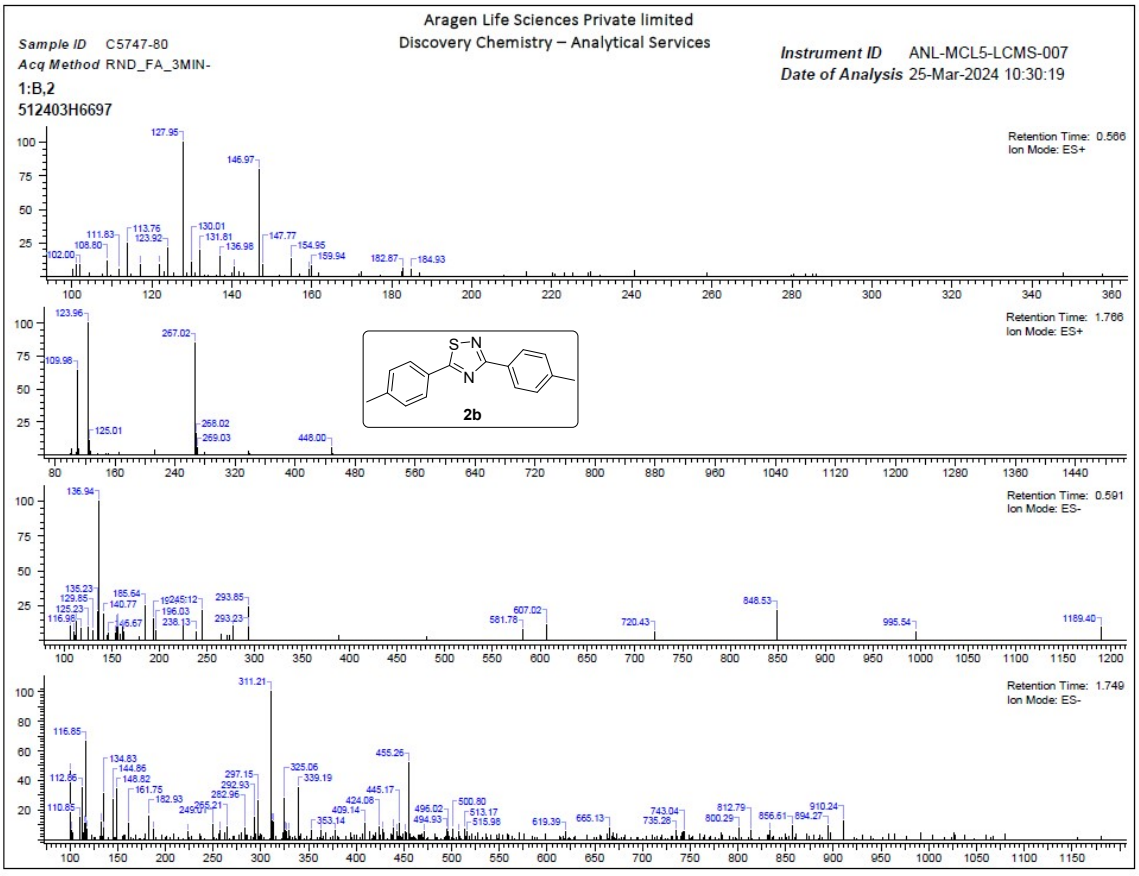
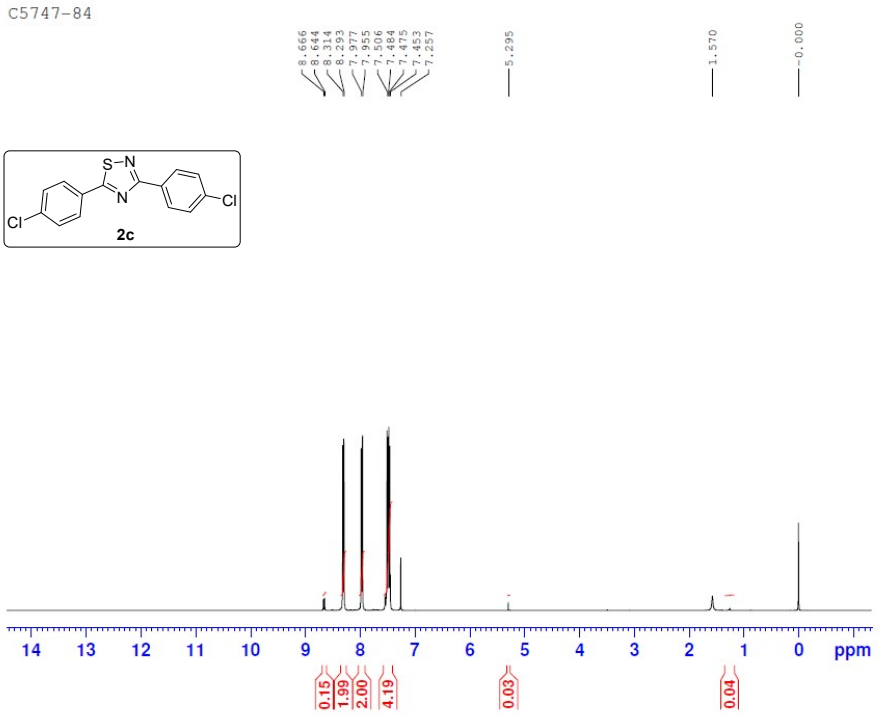
| 0.52 | 80568849.25 | 1.79 |
| 0.63 | 7536771.91 | 0.16 |
| 0.66 | 8755452.84 | 0.20 |
| 1.10 | 3581675.00 | 0.08 |
| 1.70 | 4335964705.69 | 96.74 |



## $^{1}$H NMR spectrum (400 MHz) of Compound (2c) in $CDCl_3$-$d_6$

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PROCNO                         1

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DS                           0
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FIDRES                0.250144 Hz
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RG                         101
DW                      61.000 usec
DE                      13.89 usec
TE                      298.2 K
D1                    1.00000000 sec
TD0                          1
SFO1                400.3324714 MHz
NUC1                        1H
P0                       2.67 usec
P1                       8.00 usec
PLW1                23.46000000 W

F2 - Processing parameters
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WDW                         EM
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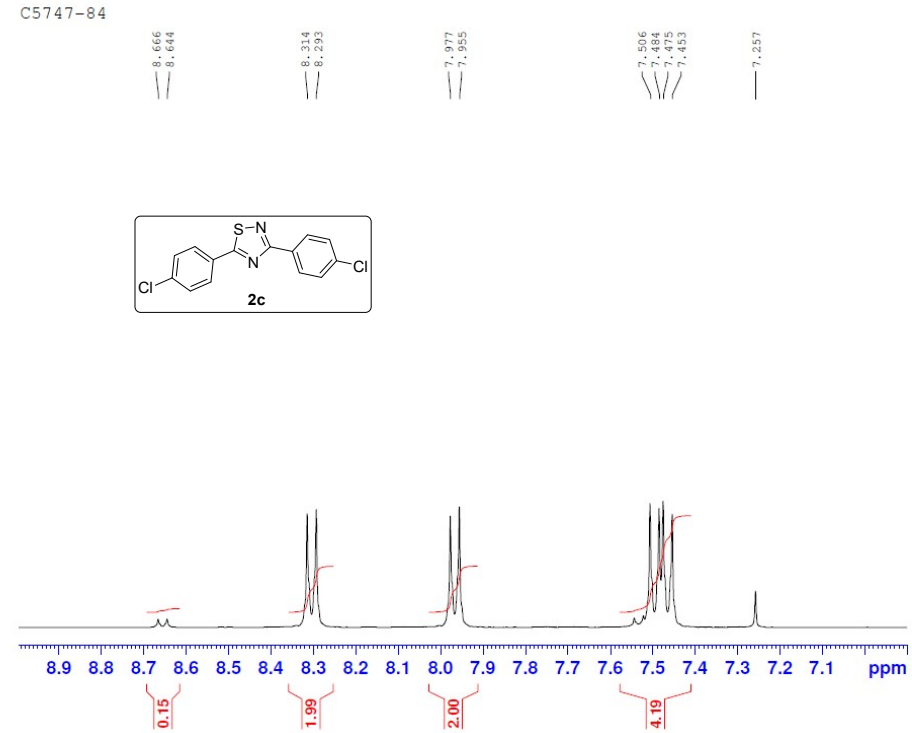


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TD                       65536
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FIDRES                0.250144 Hz
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RG                         101
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TE                      298.2 K
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NUC1                        1H
P0                       2.67 usec
P1                       8.00 usec
PLW1                23.46000000 W

F2 - Processing parameters
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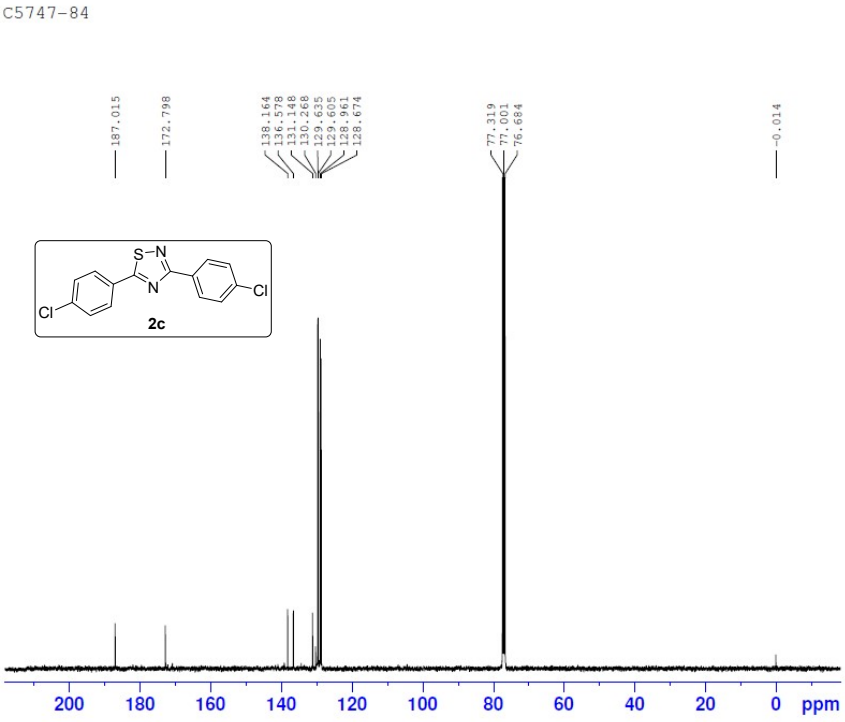
## $^{13}$C NMR spectrum (100 MHz) of Compound (2c) in DMSO-$d_6$

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PULPROG                    zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                           1024
DS                              4
SWH                     23809.524 Hz
FIDRES                   0.726609 Hz
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RG                            101
DW                         21.000 usec
DE                          6.50 usec
TE                         298.2 K
D1                       2.00000000 sec
D11                      0.03000000 sec
TD0                             1
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NUC1                         13C
P0                          2.67 usec
P1                          8.00 usec
PLW1                  98.76400000 W
SFO2                  400.3316013 MHz
NUC2                         1H
CPDPRG2                  waltz65
PCPD2                      90.00 usec
PLW2                  23.46000000 W
PLW12                  0.18537000 W
PLW13                  0.09323800 W

F2 - Processing parameters
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SF                    100.6630617 MHz
WDW                             EM
SSB                              0
LB                            2.00 Hz
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PC                            1.40
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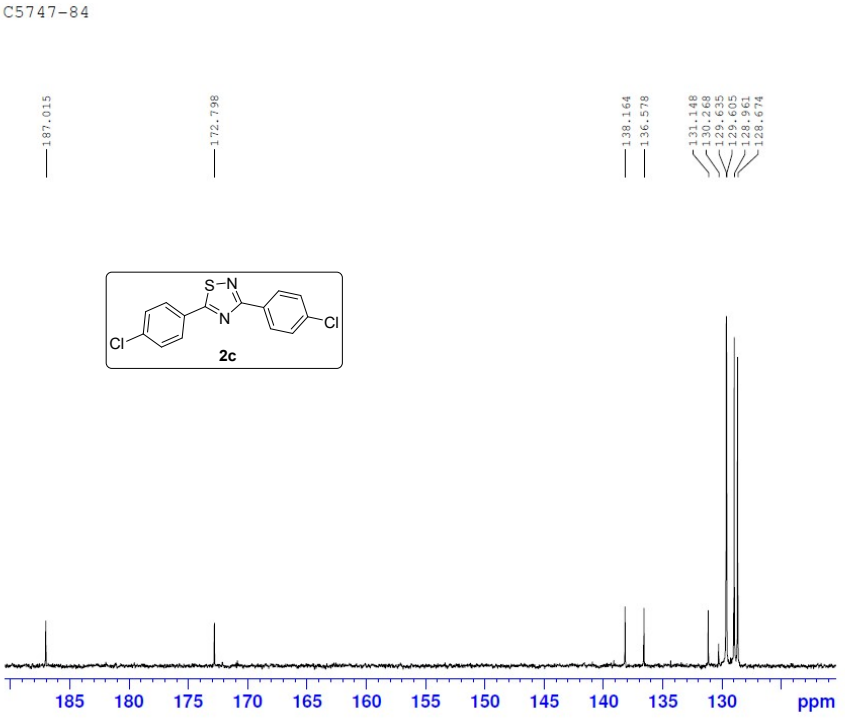


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EXPNO                            2
PROCNO                           1

F2 - Acquisition Parameters
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Time                         11.03 h
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PULPROG                    zgpg30
TD                          65536
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NUC1                         13C
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P1                          8.00 usec
PLW1                  98.76400000 W
SFO2                  400.3316013 MHz
NUC2                         1H
CPDPRG2                  waltz65
PCPD2                      90.00 usec
PLW2                  23.46000000 W
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PLW13                  0.09323800 W

F2 - Processing parameters
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LB                            2.00 Hz
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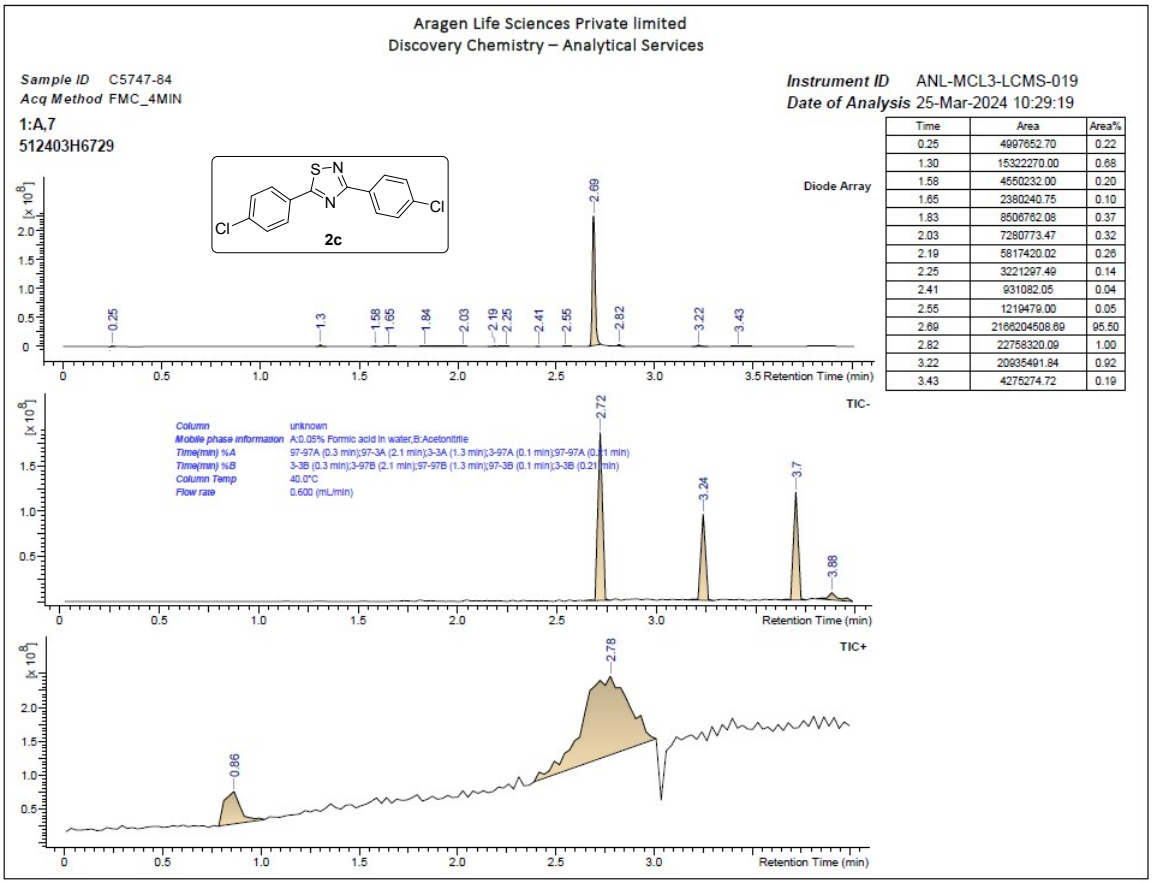
## LCMS spectrum of Compound (2c)




| Time | Area | Area% |
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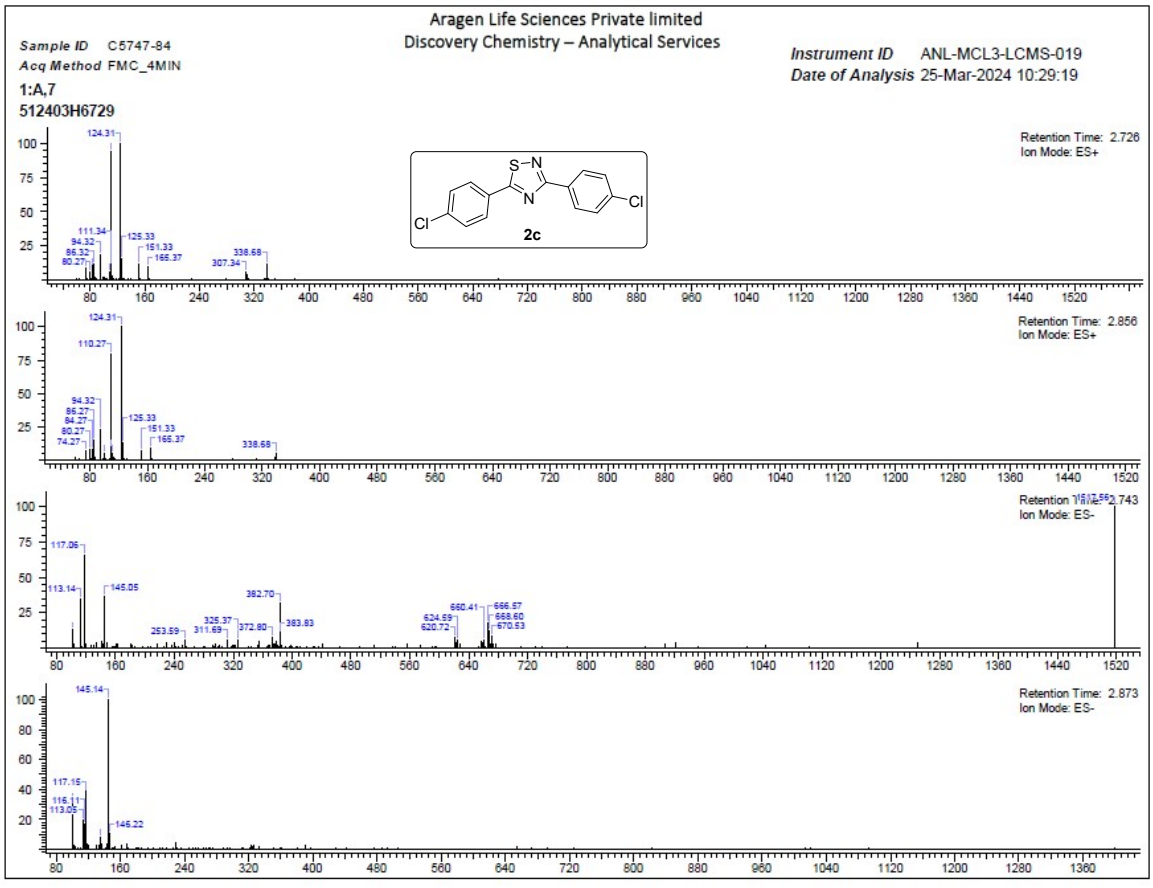
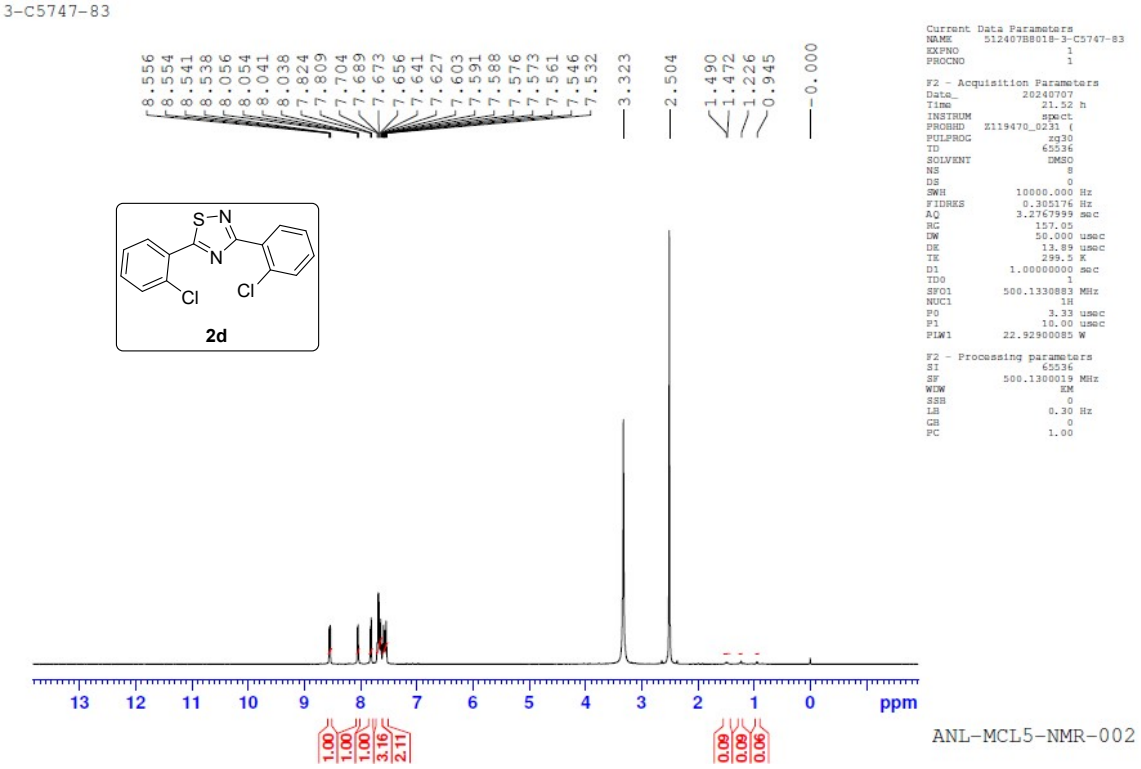
| 1.30 | 15322270.00 | 0.68 |
| 1.58 | 4550232.00 | 0.20 |
| 1.65 | 2380240.75 | 0.10 |
| 1.83 | 8506762.08 | 0.37 |
| 2.03 | 7280773.47 | 0.32 |
| 2.19 | 5817420.02 | 0.26 |
| 2.25 | 3221297.49 | 0.14 |
| 2.41 | 931082.05 | 0.04 |
| 2.55 | 1219479.00 | 0.05 |
| 2.69 | 2166204508.69 | 95.50 |
| 2.82 | 22758320.09 | 1.00 |
| 3.22 | 20935491.84 | 0.92 |
| 3.43 | 4275274.72 | 0.19 |



## $^{1}$H NMR spectrum (500 MHz) of Compound (2d) in DMSO-$d_6$





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TD                        65536
SOLVENT                    DMSO
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DS                            0
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FIDRES                    0.305176 Hz
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RG                          157.05
DW                         50.000 usec
DE                         13.89 usec
TE                         299.5 K
D1                    1.00000000 sec
TD0                           1
SFO1                500.1330883 MHz
NUC1                         1H
P0                         3.33 usec
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F2 - Processing parameters
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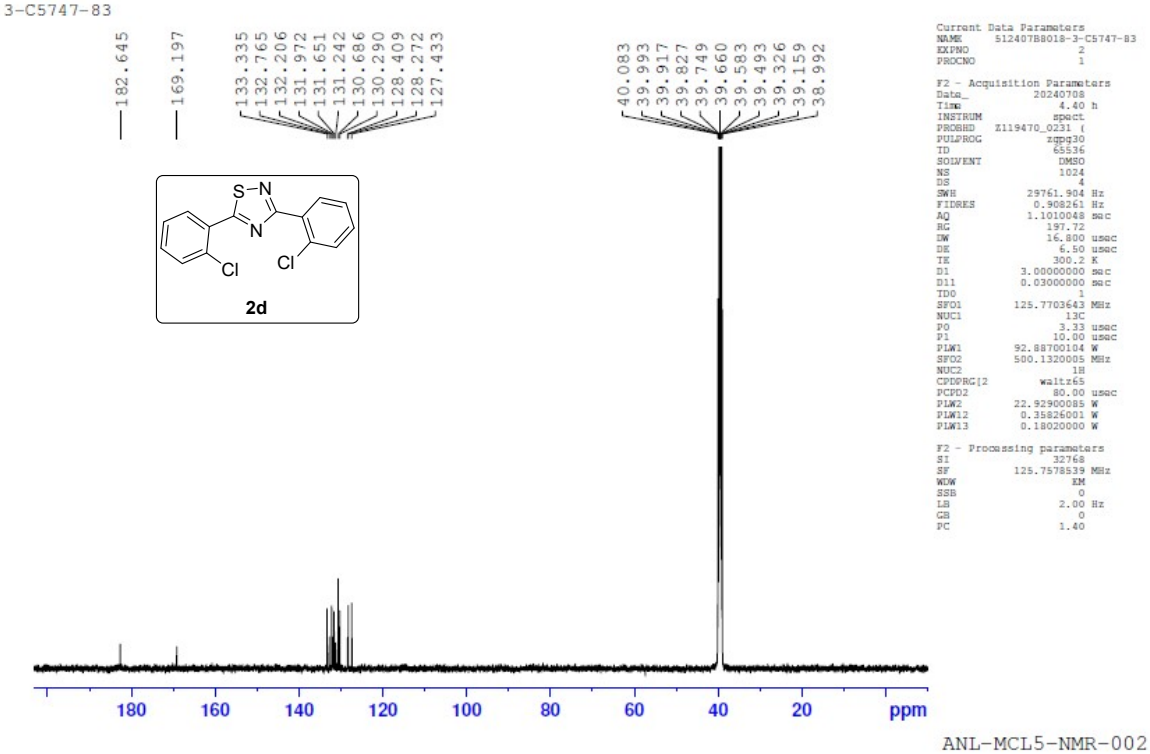
## $^{13}$C NMR spectrum (125 MHz) of Compound (2d) in DMSO-$d_6$





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TD                        65536
SOLVENT                    DMSO
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FIDRES                    0.908261 Hz
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DE                          6.50 usec
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TD0                           1
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NUC1                         13C
P0                         3.33 usec
P1                        10.00 usec
PLW1                92.88700104 W
SFO2                500.1320005 MHz
NUC2                         1H
CPDPRG[2                waltz65
PCPD2                     80.00 usec
PLW2                22.92900085 W
PLW12                0.35826001 W
PLW13                0.18020000 W

F2 - Processing parameters
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WDW                          EM
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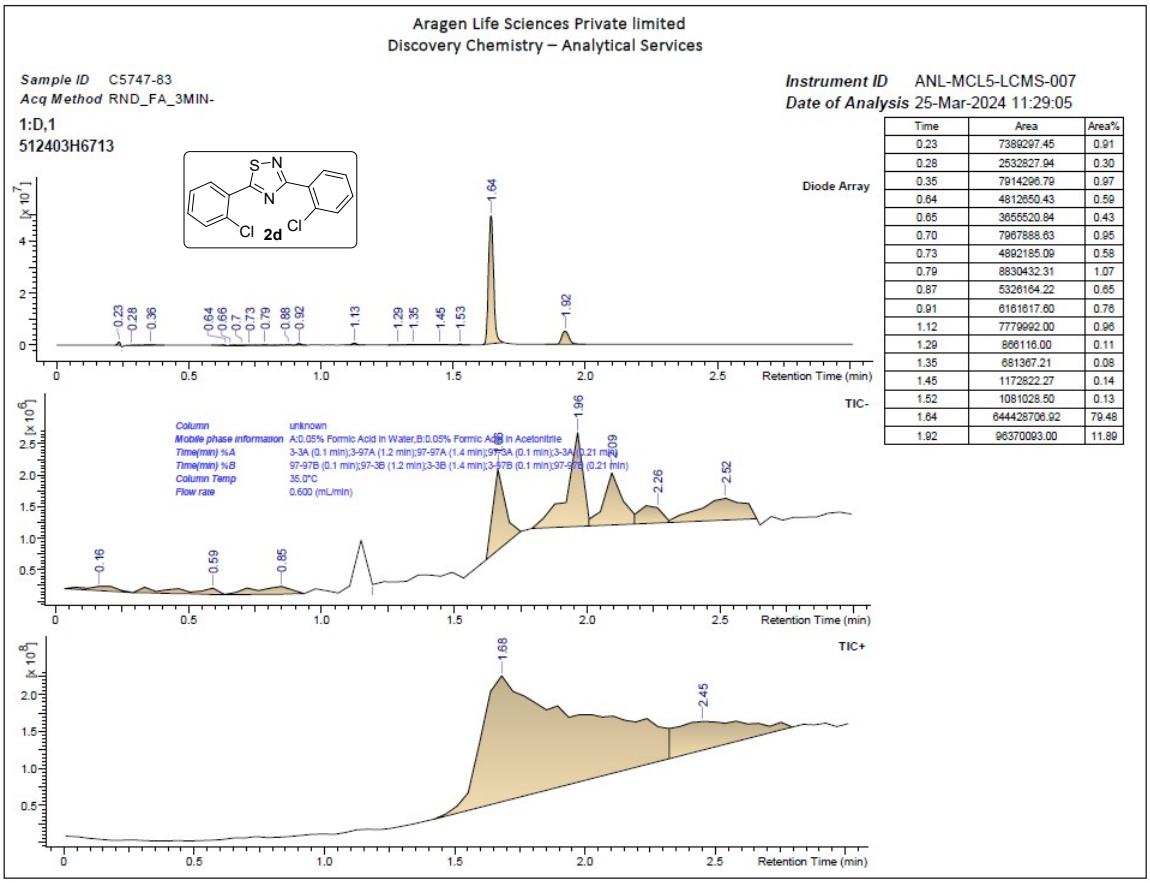
## LCMS spectrum of Compound (2d)

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| 0.35 | 7914296.79 | 0.97 |
| 0.64 | 4812650.43 | 0.59 |
| 0.65 | 3655520.84 | 0.43 |
| 0.70 | 7967888.63 | 0.95 |
| 0.73 | 4892185.09 | 0.58 |
| 0.79 | 8830432.31 | 1.07 |
| 0.87 | 5326164.22 | 0.65 |
| 0.91 | 6161617.60 | 0.76 |
| 1.12 | 7779992.00 | 0.96 |
| 1.29 | 866116.00 | 0.11 |
| 1.35 | 681367.21 | 0.08 |
| 1.45 | 1172822.27 | 0.14 |
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| 1.64 | 644426706.92 | 79.48 |
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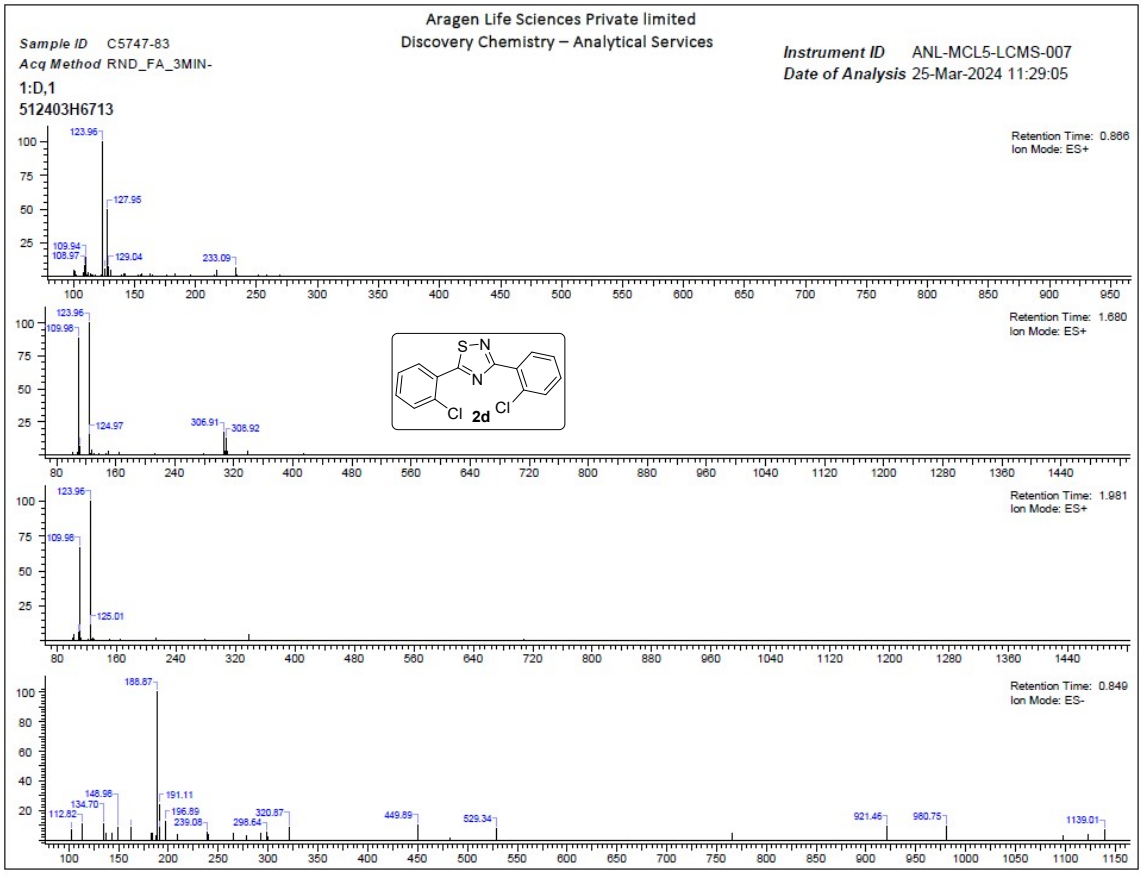
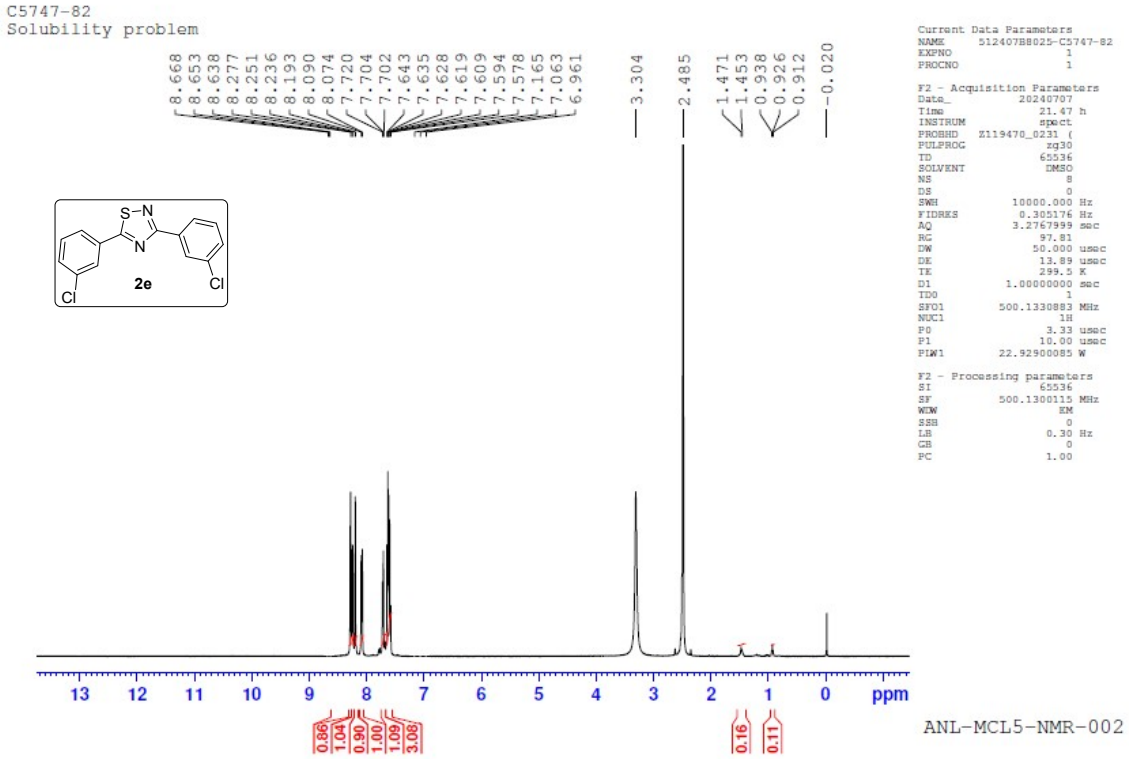
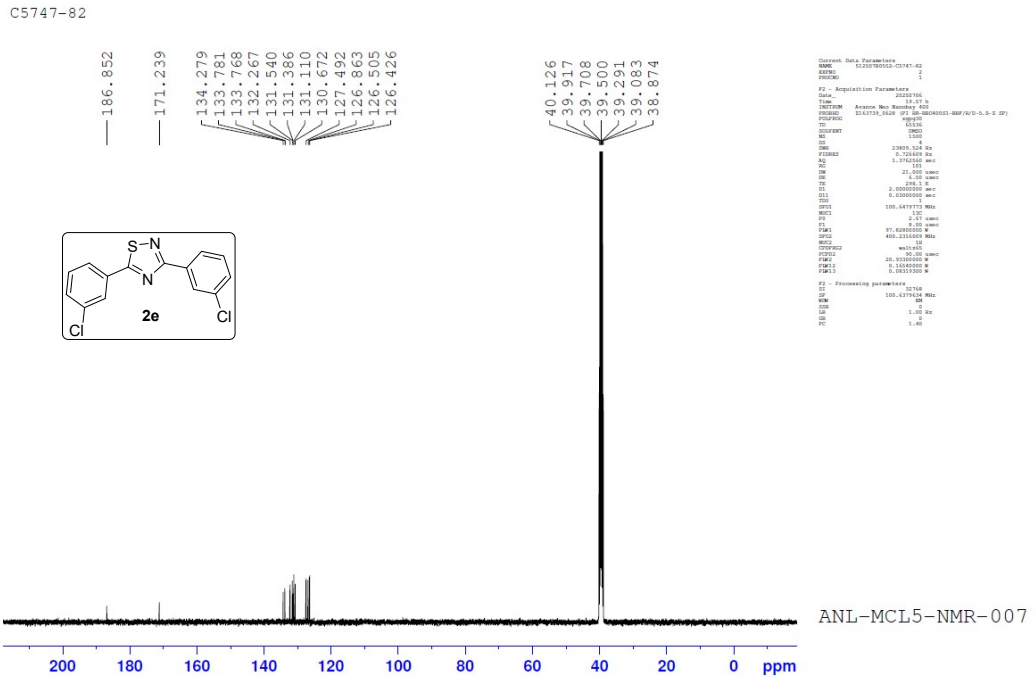


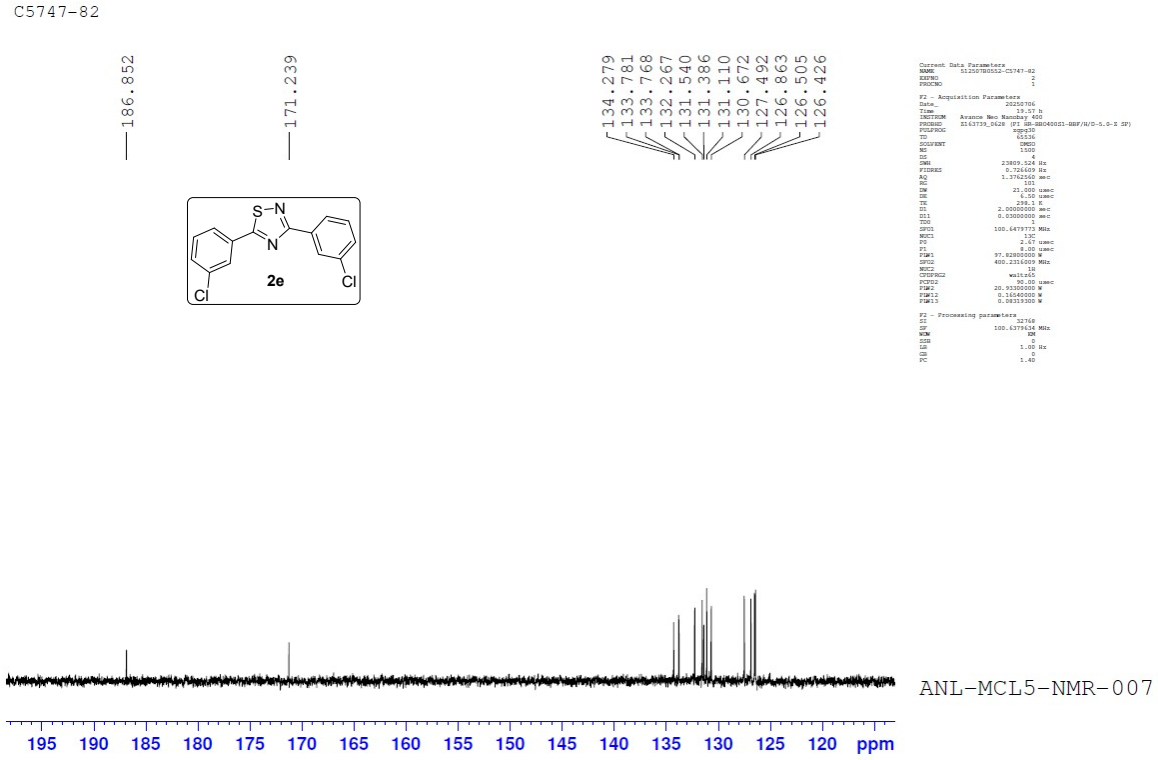
## $^{1}$H NMR spectrum (500 MHz) of Compound (2e) in DMSO-$d_6$



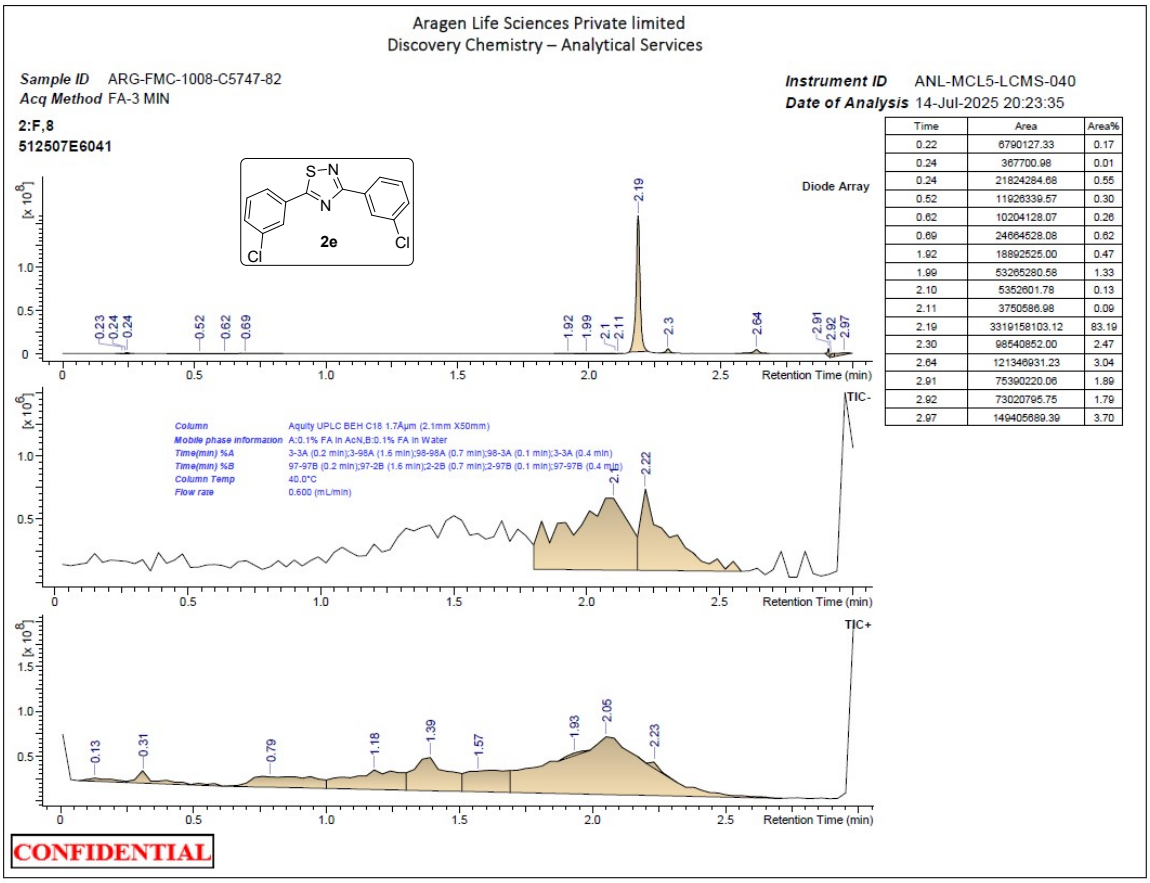

## $^{13}$C NMR spectrum (100 MHz) of Compound (2e) in DMSO-$d_6$



## $^{13}$C NMR spectrum (100 MHz) of Compound (2e) in DMSO-$d_6$-*Expansion*



## LCMS spectrum of Compound (2e)

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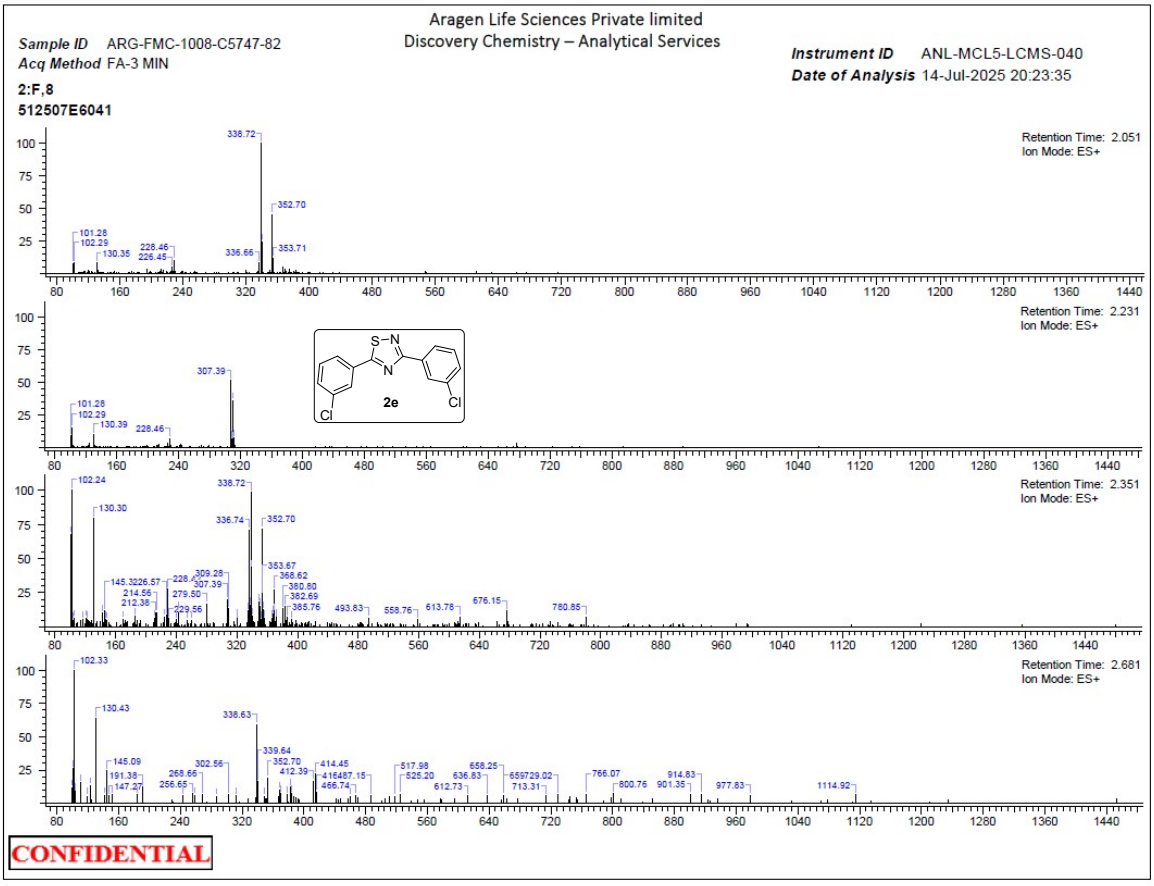
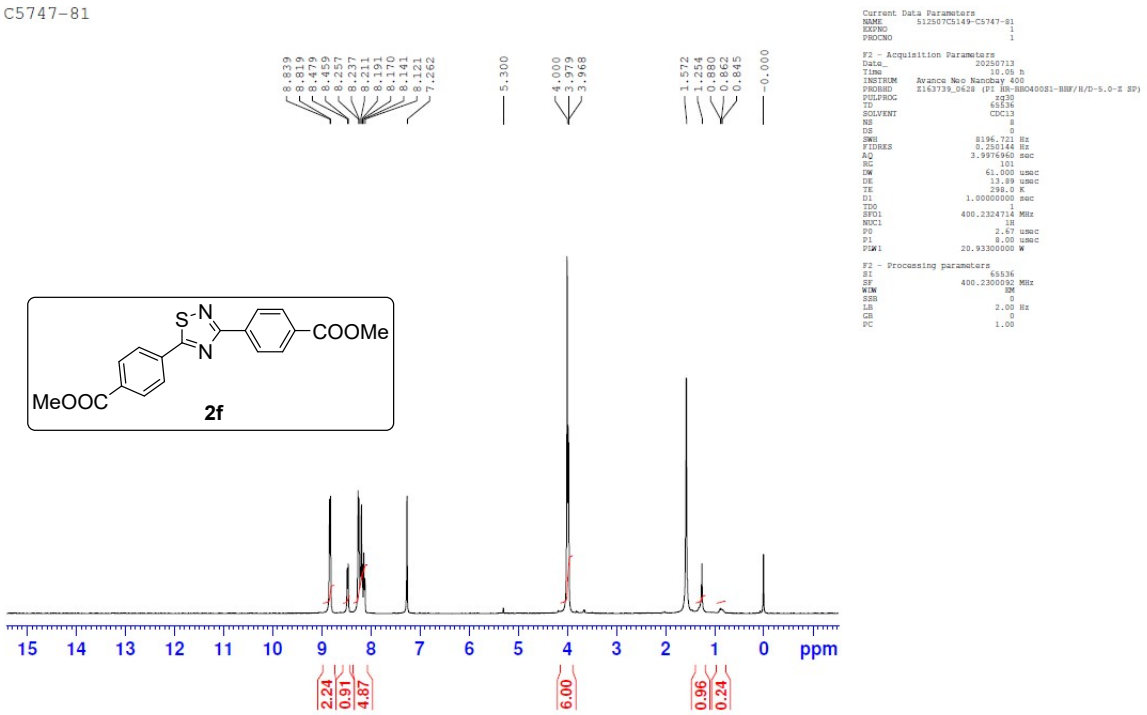
| 0.24 | 367700.98 | 0.01 |
| 0.24 | 21824284.68 | 0.55 |
| 0.52 | 11926339.57 | 0.30 |
| 0.62 | 10204128.07 | 0.26 |
| 0.69 | 24664528.08 | 0.62 |
| 1.92 | 18892525.00 | 0.47 |
| 1.99 | 53265280.58 | 1.33 |
| 2.10 | 5352601.78 | 0.13 |
| 2.11 | 3750586.98 | 0.09 |
| 2.19 | 3319158103.12 | 83.19 |
| 2.30 | 98540852.00 | 2.47 |
| 2.64 | 121346931.23 | 3.04 |
| 2.91 | 75390220.06 | 1.89 |
| 2.92 | 73020795.75 | 1.79 |
| 2.97 | 149405689.39 | 3.70 |



## $^{1}$H NMR spectrum (400 MHz) of Compound (2f) in $CDCl_3$





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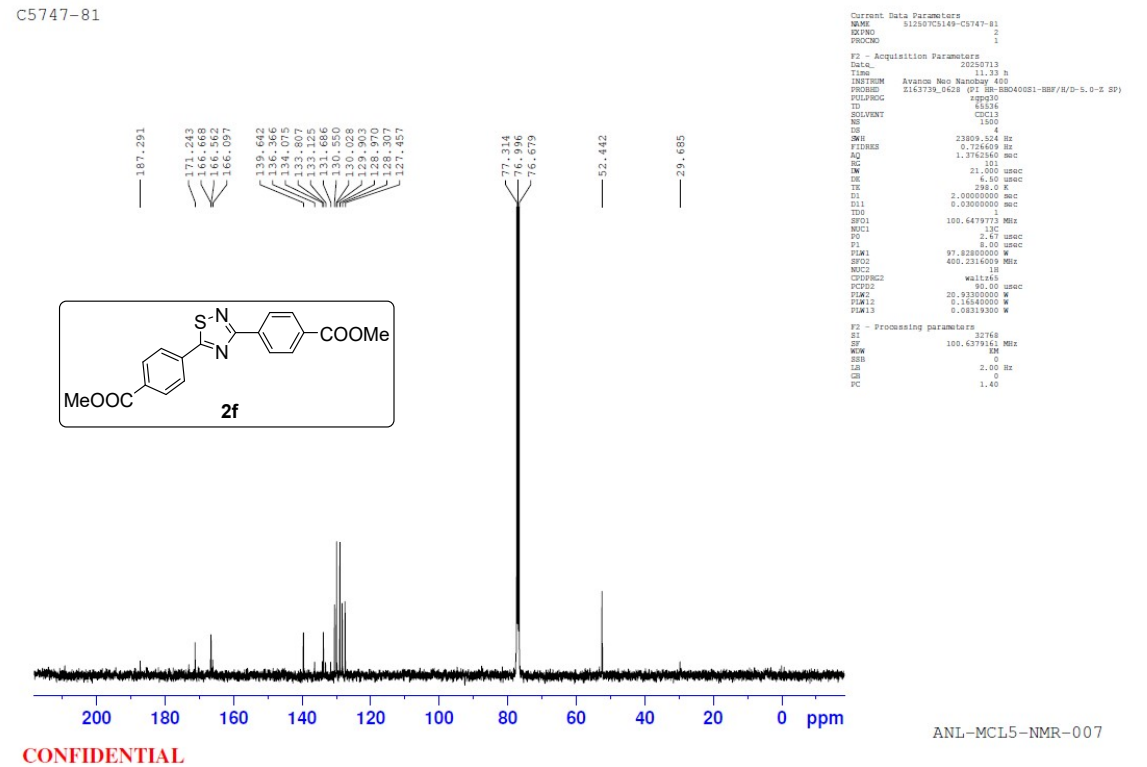
Time 10.05 h
INSTRUM Avance Neo Nanobay 400
PROBHD Z163739_0628 (P1 BB-BBO400S1-BBF/H/D-5.0-Z SP)
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976960 sec
RG 101
DW 61.000 usec
DE 13.89 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.2324714 MHz
NUC1 1H
P0 2.67 usec
P1 8.00 usec
PLW1 20.93300000 W

F2 - Processing parameters
SI 65536
SF 400.2300092 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00




ANL-MCL5-NMR-007

## $^{13}$C NMR spectrum (100 MHz) of Compound (2f) in $CDCl_3$





Current Data Parameters
NAME 512507C5149-C5747-81
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20250713
Time 11.33 h
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PROBHD Z163739_0628 (P1 BB-BBO400S1-BBF/H/D-5.0-Z SP)
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1500
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 101
DW 21.000 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6479773 MHz
NUC1 13C
P0 2.67 usec
P1 8.00 usec
PLW1 97.82800000 W
SFO2 400.2316009 MHz
NUC2 1H
CPDPRG2 waltz65
PCPD2 90.00 usec
PLW2 20.93300000 W
PLW12 0.16540000 W
PLW13 0.08319300 W

F2 - Processing parameters
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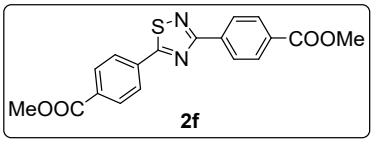
SF 100.6379161 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

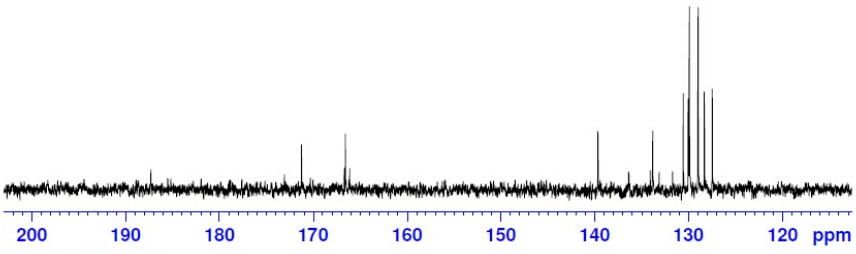



ANL-MCL5-NMR-007

C5747-81


**2f**





Current Data Parameters
NAME 512507C5149-C5747-81
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
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Time 11.33 h
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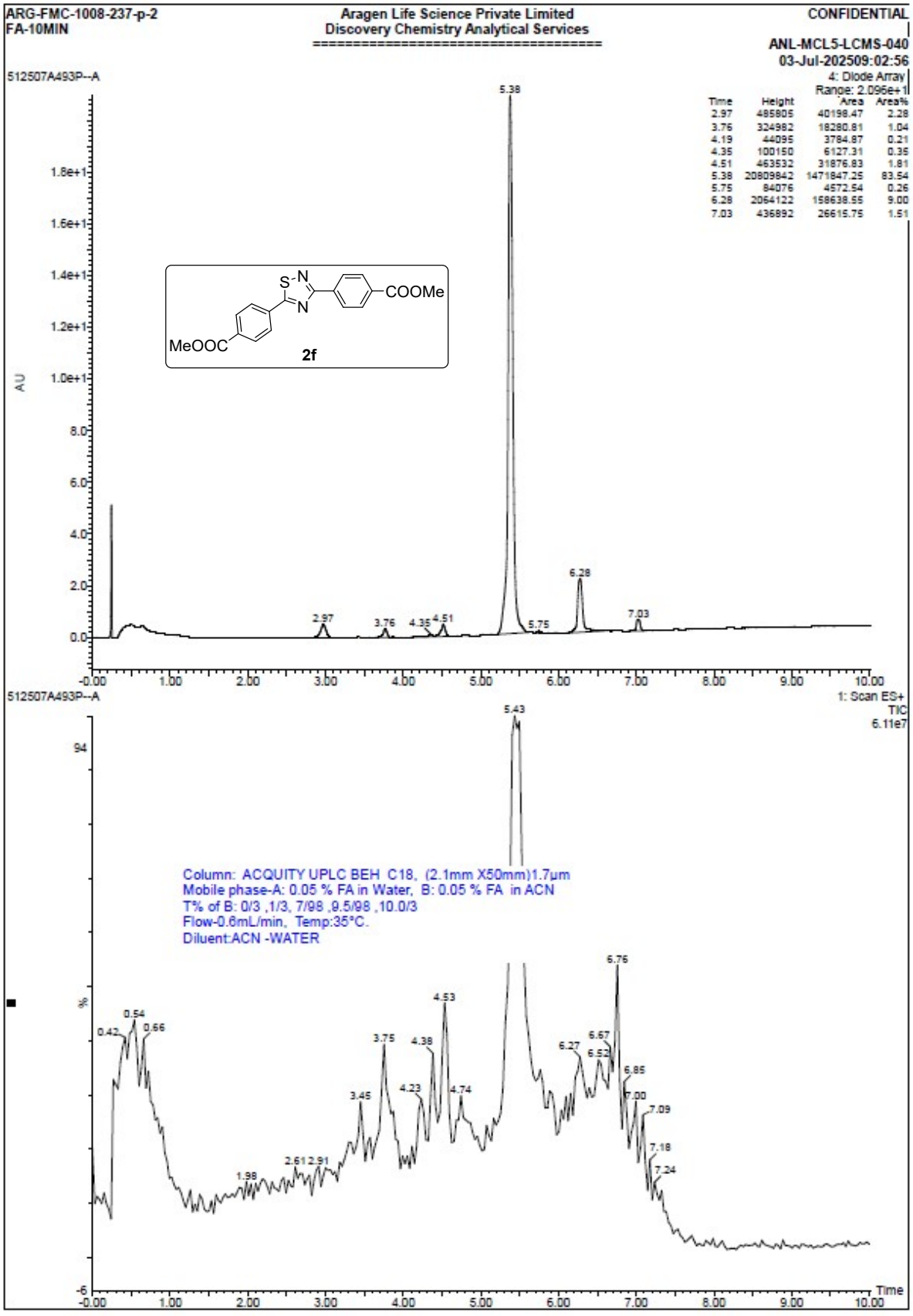
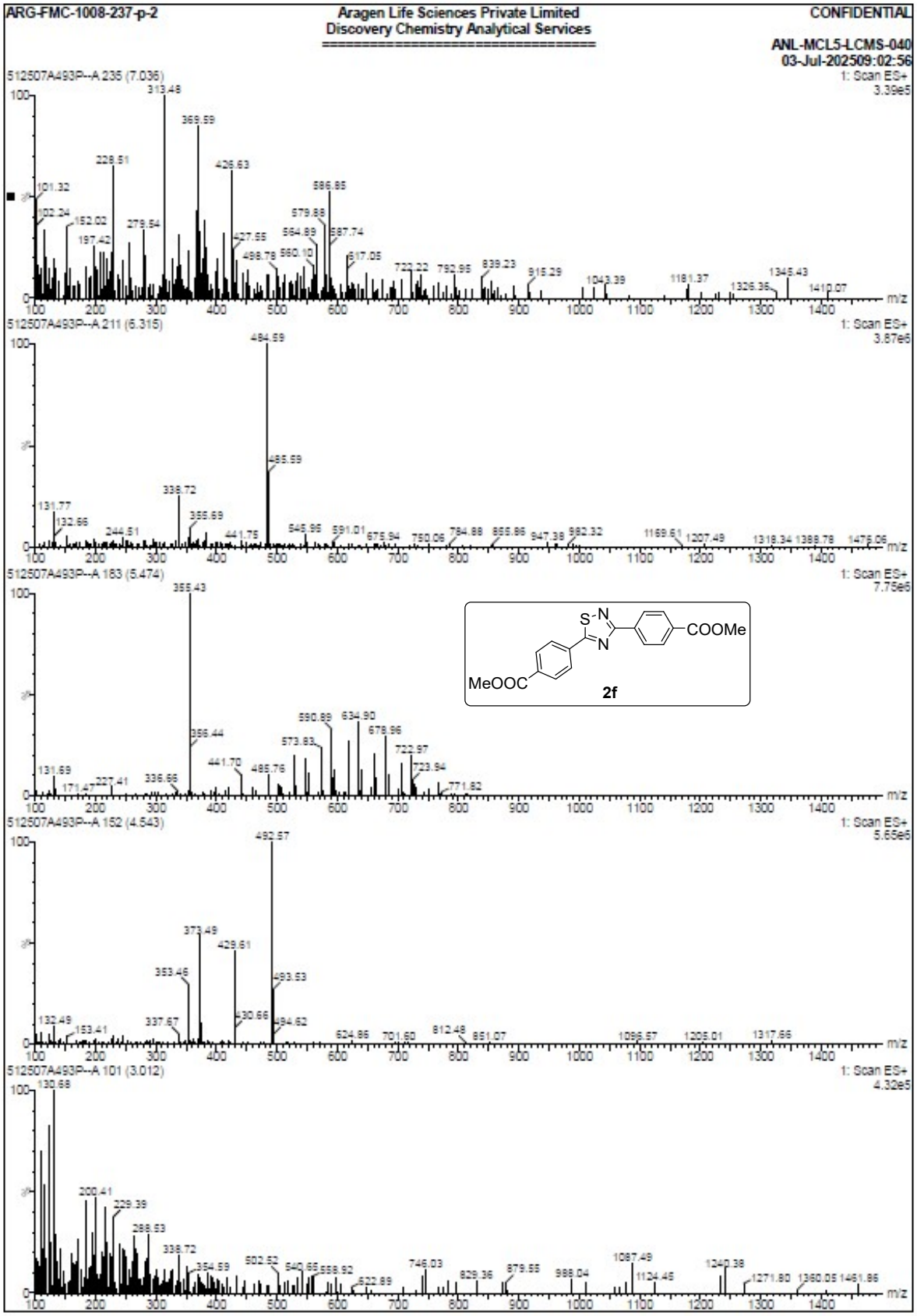
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1500
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 101
DW 21.000 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6479773 MHz
NUC1 13C
P0 2.67 usec
P1 8.00 usec
PLW1 97.82800000 W
SFO2 400.2316009 MHz
NUC2 1H
CPDPRG2 waltz65
PCPD2 90.00 usec
PLW2 20.93300000 W
PLW12 0.16540000 W
PLW13 0.08319300 W

F2 - Processing parameters
SI 32768
SF 100.6379161 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

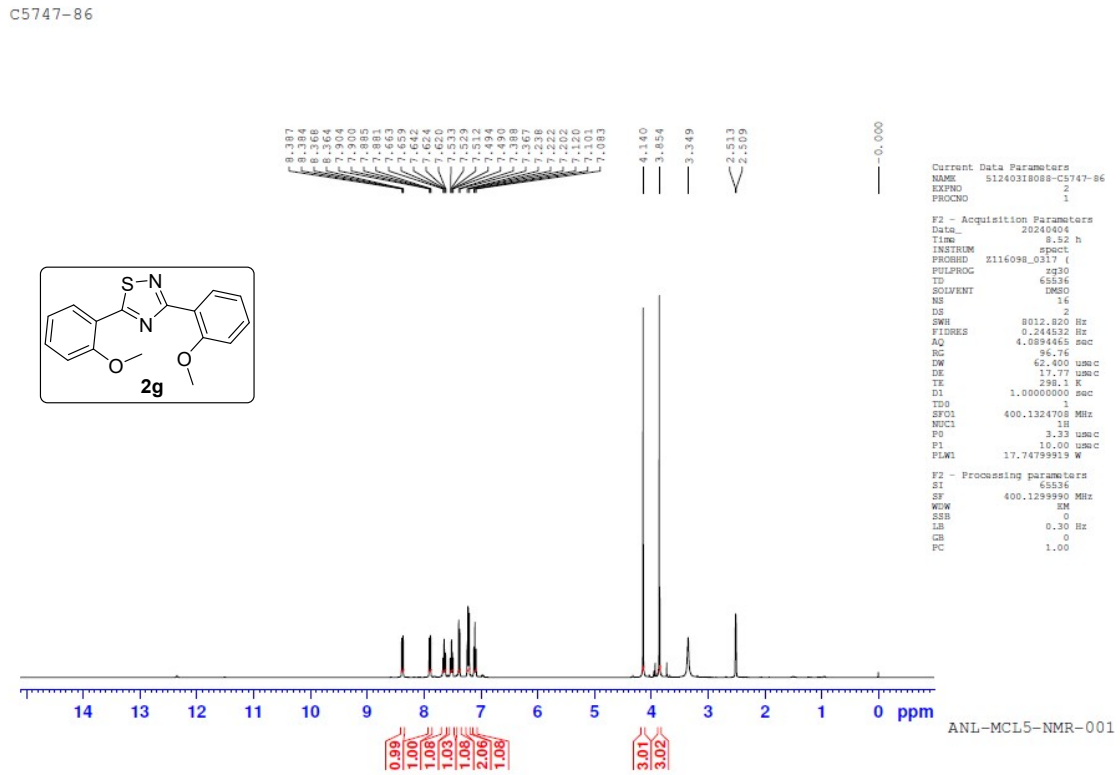

ANL-MCL5-NMR-007

## LCMS spectrum of Compound (2f)

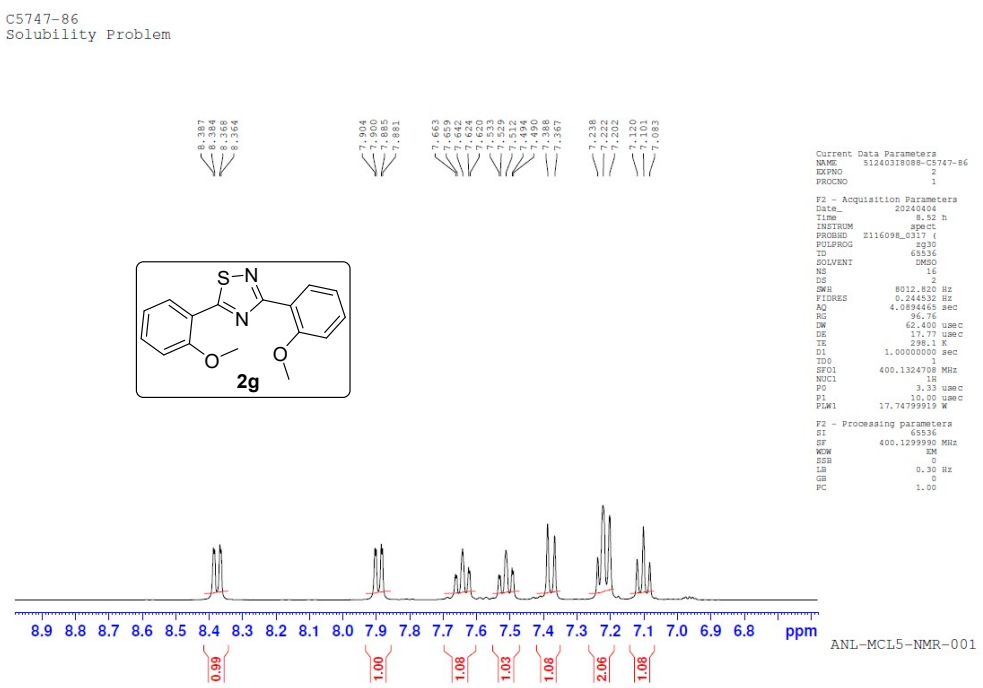



ARG-FMC-1008-237-p-2
Aragen Life Sciences Private Limited
Discovery Chemistry Analytical Services
CONFIDENTIAL
ANL-MCL5-LCMS-040
03-Jul-202509:02:56
512507A493P--A 235 (7.036)
1: Scan ES+
3.39e5
512507A493P--A 211 (6.315)
1: Scan ES+
3.87e6
512507A493P--A 183 (5.474)
1: Scan ES+
7.75e6
512507A493P--A 152 (4.543)
1: Scan ES+
5.65e6
512507A493P--A 101 (3.012)
1: Scan ES+
4.32e5
m/z
MeOOC
COOMe
2f

## $^1$H NMR spectrum (400 MHz) of Compound (2g) in DMSO-$d_6$

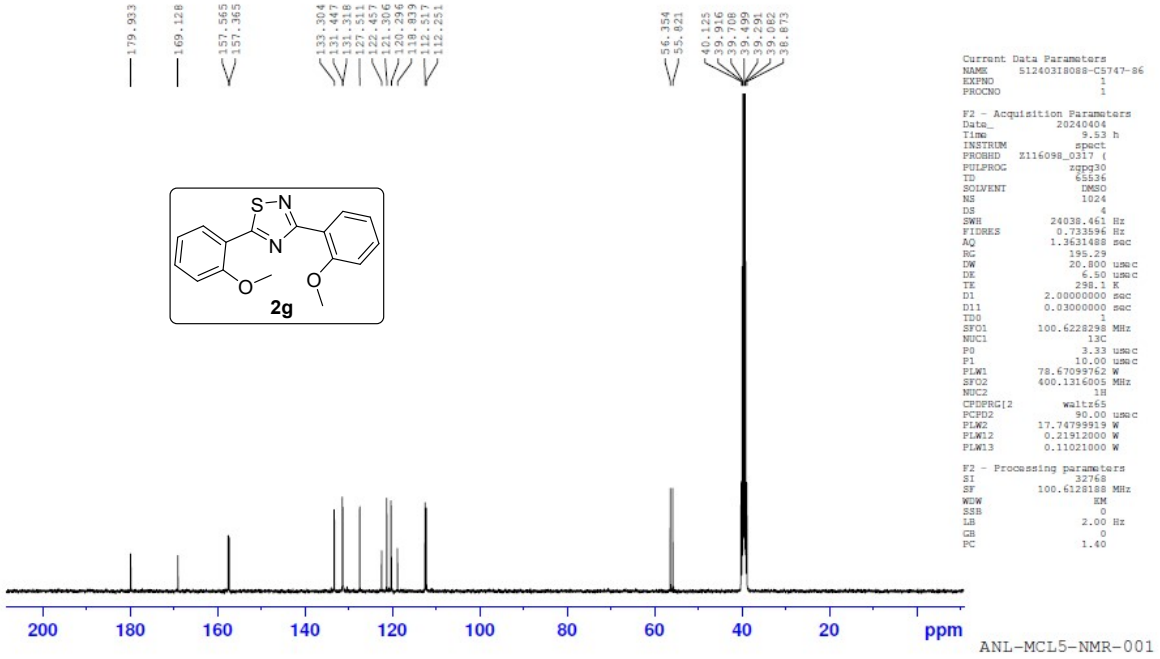



## $^1$H NMR spectrum (400 MHz) of Compound (2g) in DMSO-$d_{6\text{-Expanssion}}$



## $^{13}$C NMR spectrum (100 MHz) of Compound (2g) in DMSO-$d_6$

C5747-86




Current Data Parameters

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|---|---|
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| EXPNO | 1 |
| PROCNO | 1 |

F2 - Acquisition Parameters

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|---|---|
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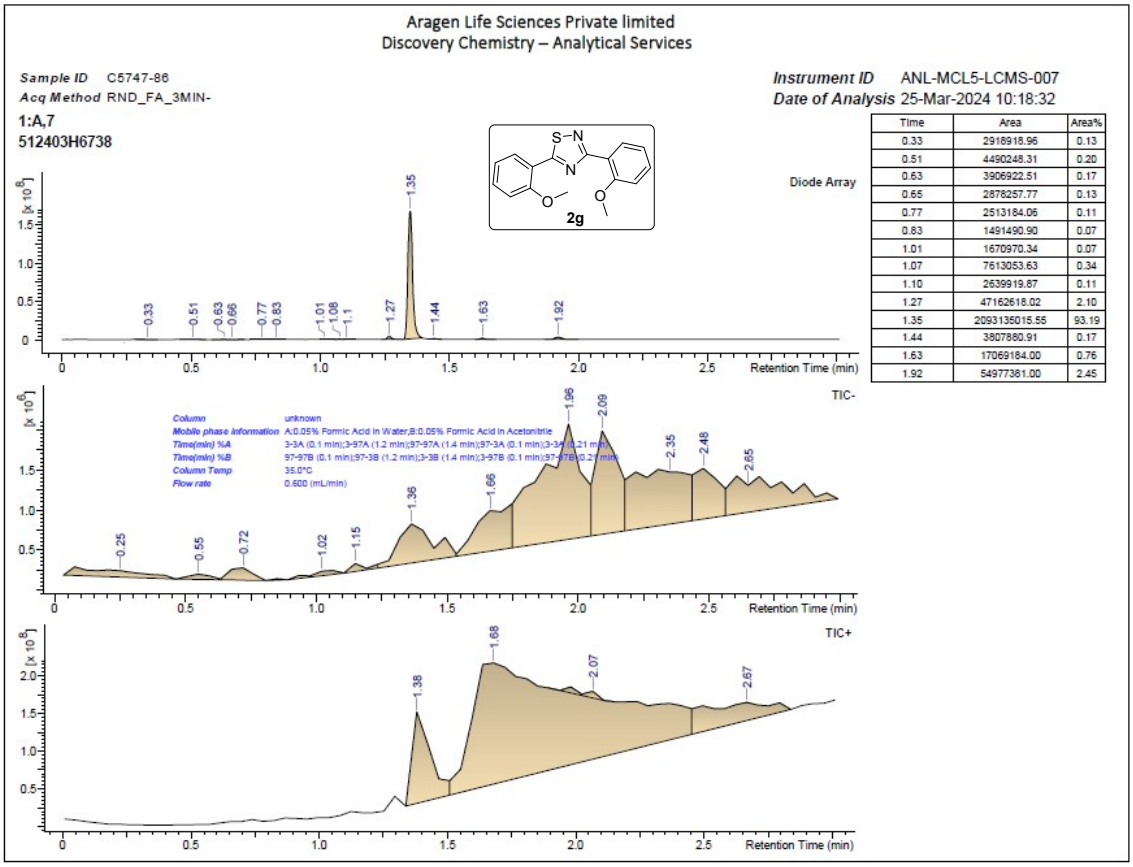
| Time | 9.53 h |
| INSTRUM | spect |
| PROBHD | Z116098_0317 ( |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | DMSO |
| NS | 1024 |
| DS | 4 |
| SWH | 24038.461 Hz |
| FIDRES | 0.733596 Hz |
| AQ | 1.3631488 sec |
| RG | 195.29 |
| DW | 20.800 usec |
| DE | 6.50 usec |
| TE | 298.1 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |
| SFO1 | 100.6228298 MHz |
| NUC1 | 13C |
| P0 | 3.33 usec |
| P1 | 10.00 usec |
| PLW1 | 78.67099762 W |
| SFO2 | 400.1316005 MHz |
| NUC2 | 1H |
| CPDPRG[2 | waltz65 |
| PCPD2 | 90.00 usec |
| PLW2 | 17.74799919 W |
| PLW12 | 0.21912000 W |
| PLW13 | 0.11021000 W |

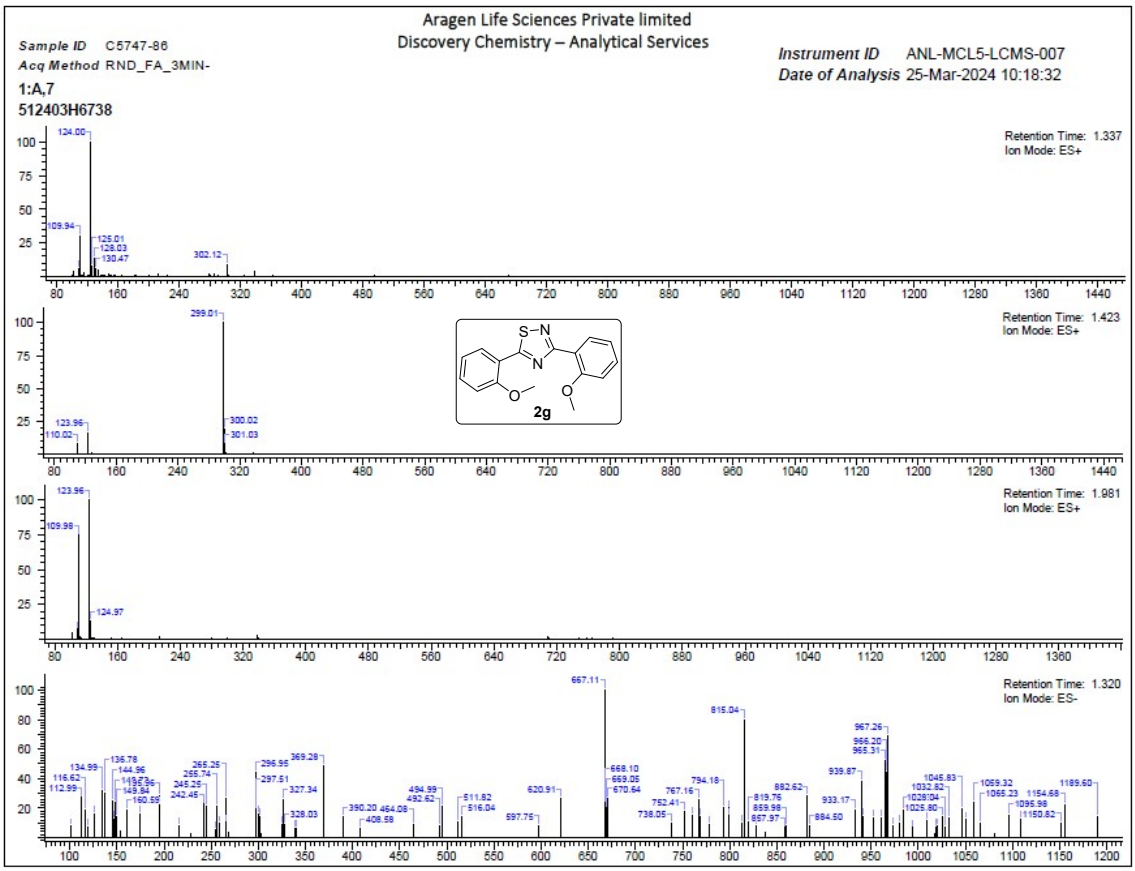
F2 - Processing parameters

| | |
|---|---|
| SI | 32768 |
| SF | 100.6128188 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 2.00 Hz |
| GB | 0 |
| PC | 1.40 |

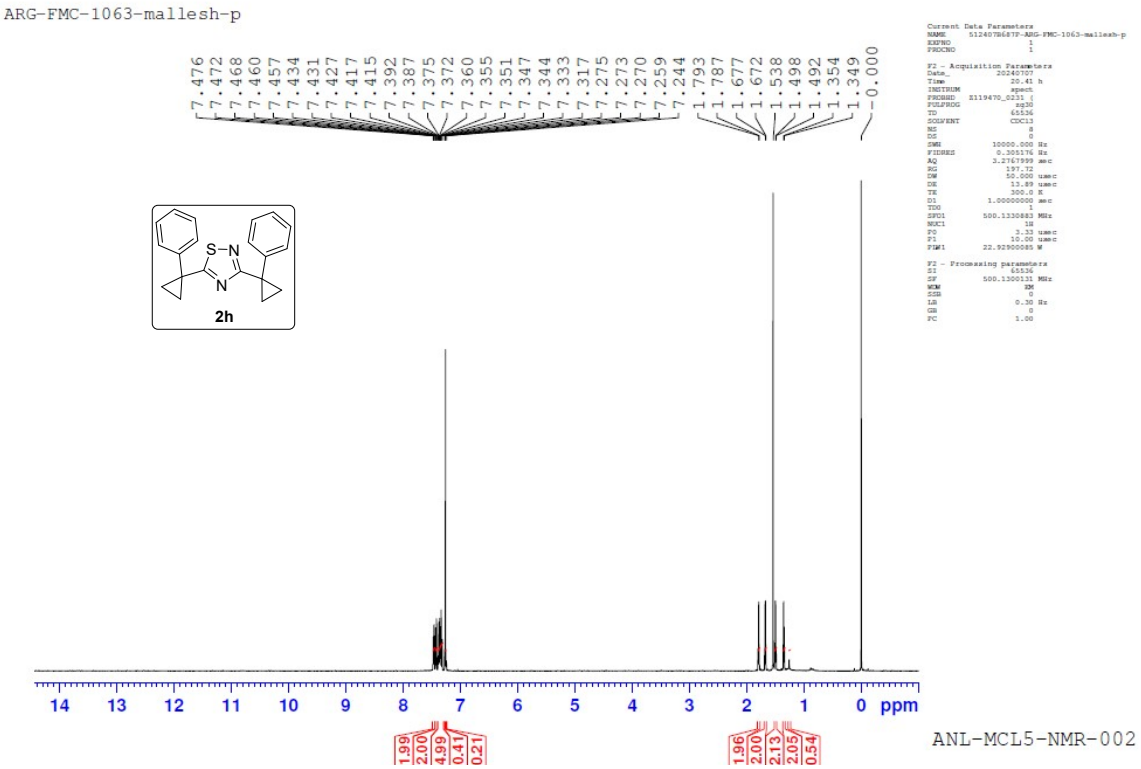
## LCMS spectrum of Compound (2g)



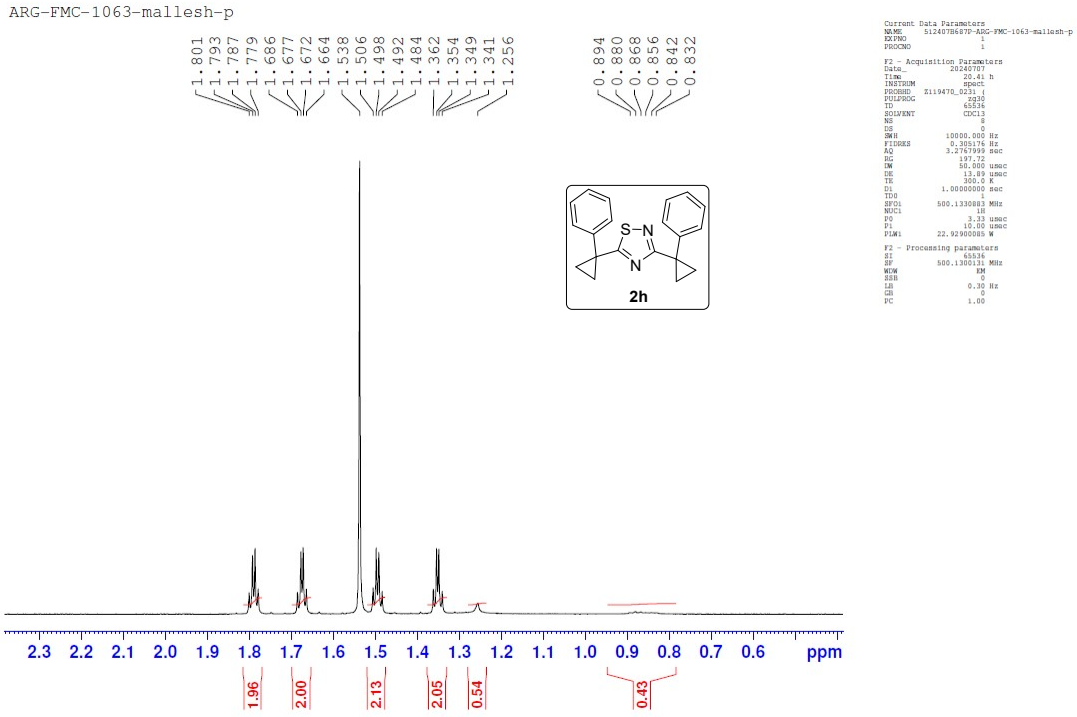

| Time | Area | Area% |
|---|---|---|
| 0.33 | 2918918.96 | 0.13 |
| 0.51 | 4490248.31 | 0.20 |
| 0.63 | 3906922.51 | 0.17 |
| 0.65 | 2878257.77 | 0.13 |
| 0.77 | 2513184.06 | 0.11 |
| 0.83 | 1491490.90 | 0.07 |
| 1.01 | 1670970.34 | 0.07 |
| 1.07 | 7613053.63 | 0.34 |
| 1.10 | 2639919.87 | 0.11 |
| 1.27 | 47162618.02 | 2.10 |
| 1.35 | 2093135015.55 | 93.19 |
| 1.44 | 3807880.91 | 0.17 |
| 1.63 | 17069184.00 | 0.76 |
| 1.92 | 54977381.00 | 2.45 |



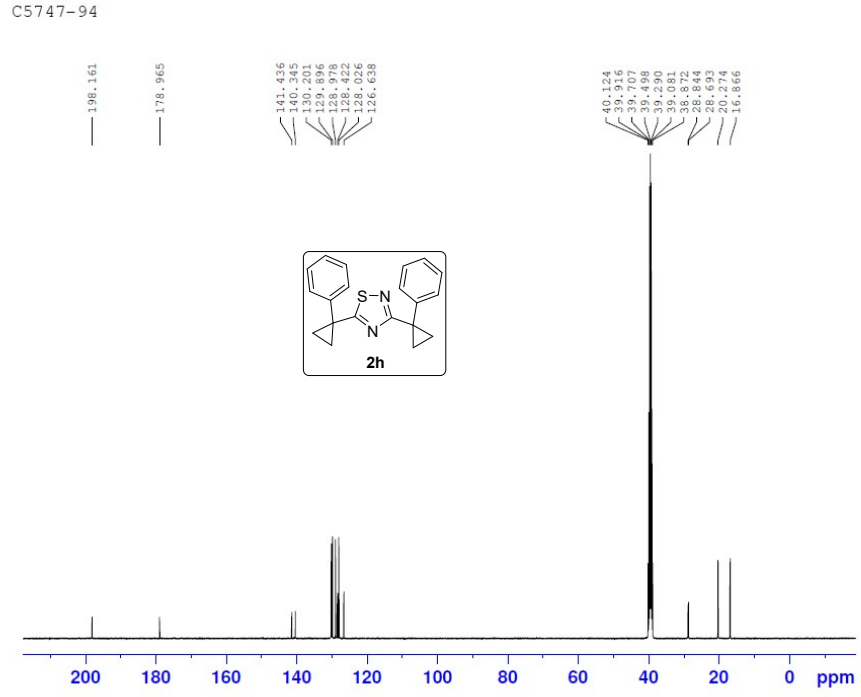
**$^{1}$H NMR spectrum (500 MHz) of Compound (2h) in $CDCl_3$**




**$^{1}$H NMR spectrum (500 MHz) of Compound (2h) in $CDCl_3$-Expansion**




**$^{13}$C NMR spectrum (100 MHz) of Compound (2h) in DMSO-$d_6$**

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EXPNO                        2
PROCNO                       1

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Time                 19.58 h
INSTRUM  Avance Neo Nanobay
PROBHD   Z163739_0485 (
PULPROG              zgpg30
TD                    65536
SOLVENT                DMSO
NS                     2000
DS                        4
SWH               23809.523 Hz
FIDRES             0.726609 Hz
AQ                1.3762560 sec
RG                     71.8
DW                   21.000 usec
DE                    6.50 usec
TE                  298.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1
SFO1            100.6228298 MHz
NUC1                    13C
P0                    2.67 usec
P1                    8.00 usec
PLW1            94.24700165 W
SFO2            400.1316005 MHz
NUC2                    1H
CPDPRG[2          waltz65
PCPD2                90.00 usec
PLW2            23.84199905 W
PLW12            0.18838000 W
PLW13            0.09475400 W

F2 - Processing parameters
SI                    32768
SF              100.6128200 MHz
WDW                     EM
SSB                      0
LB                    2.00 Hz
GB                       0
PC                    1.40
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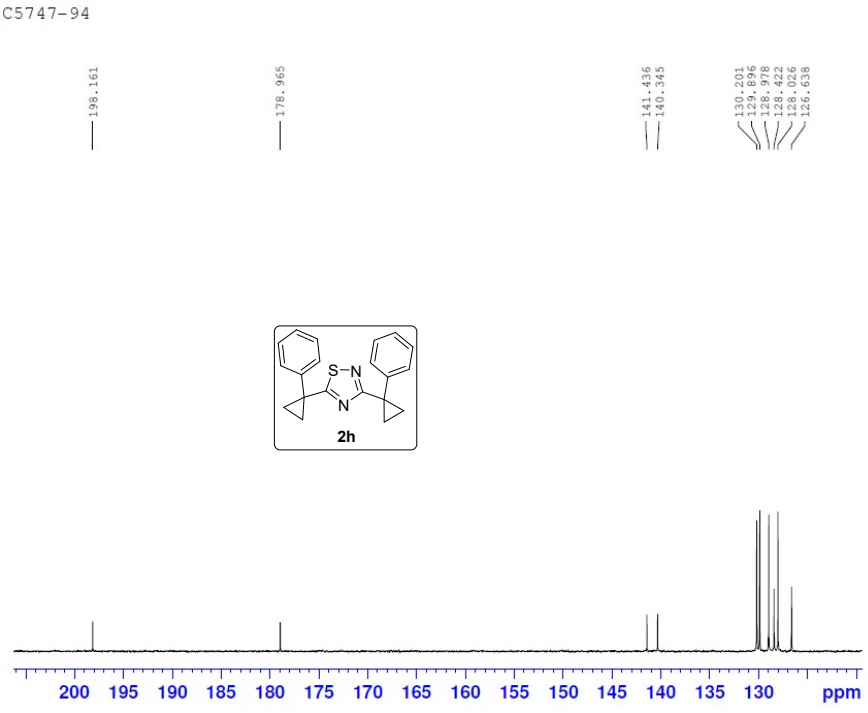


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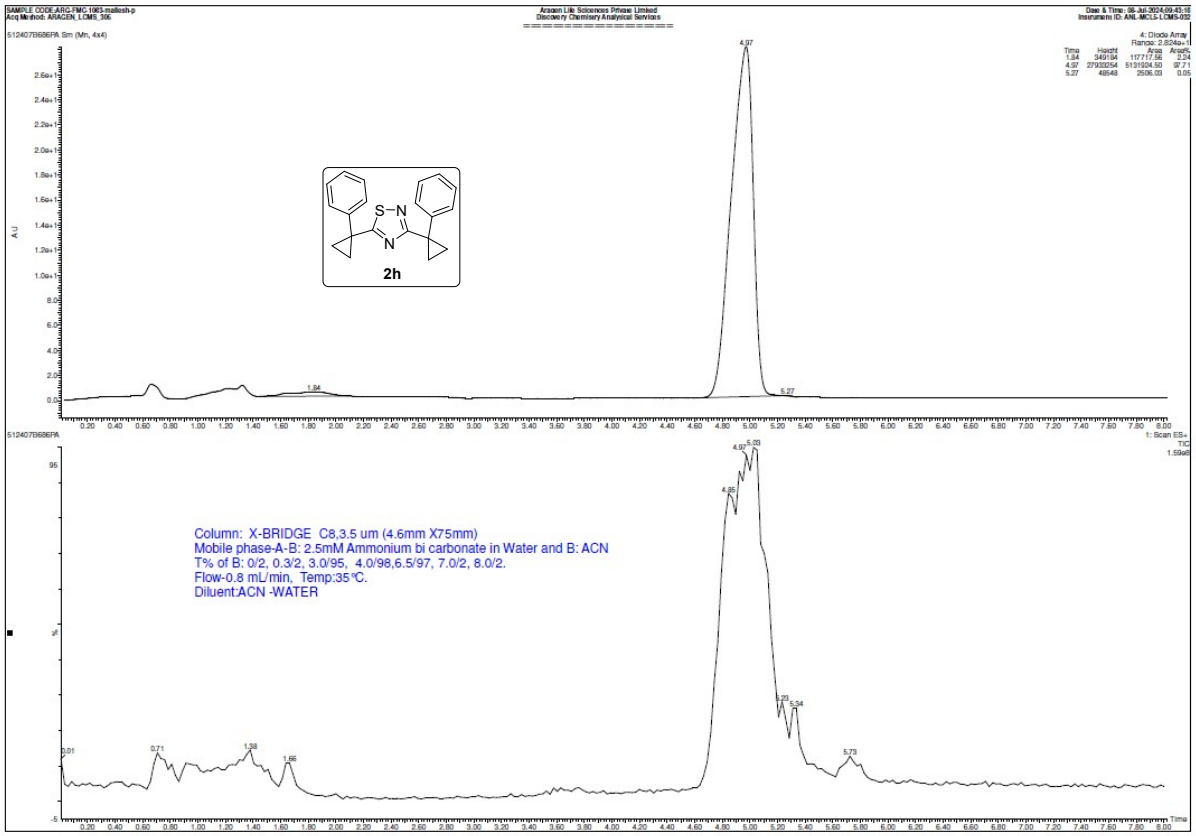
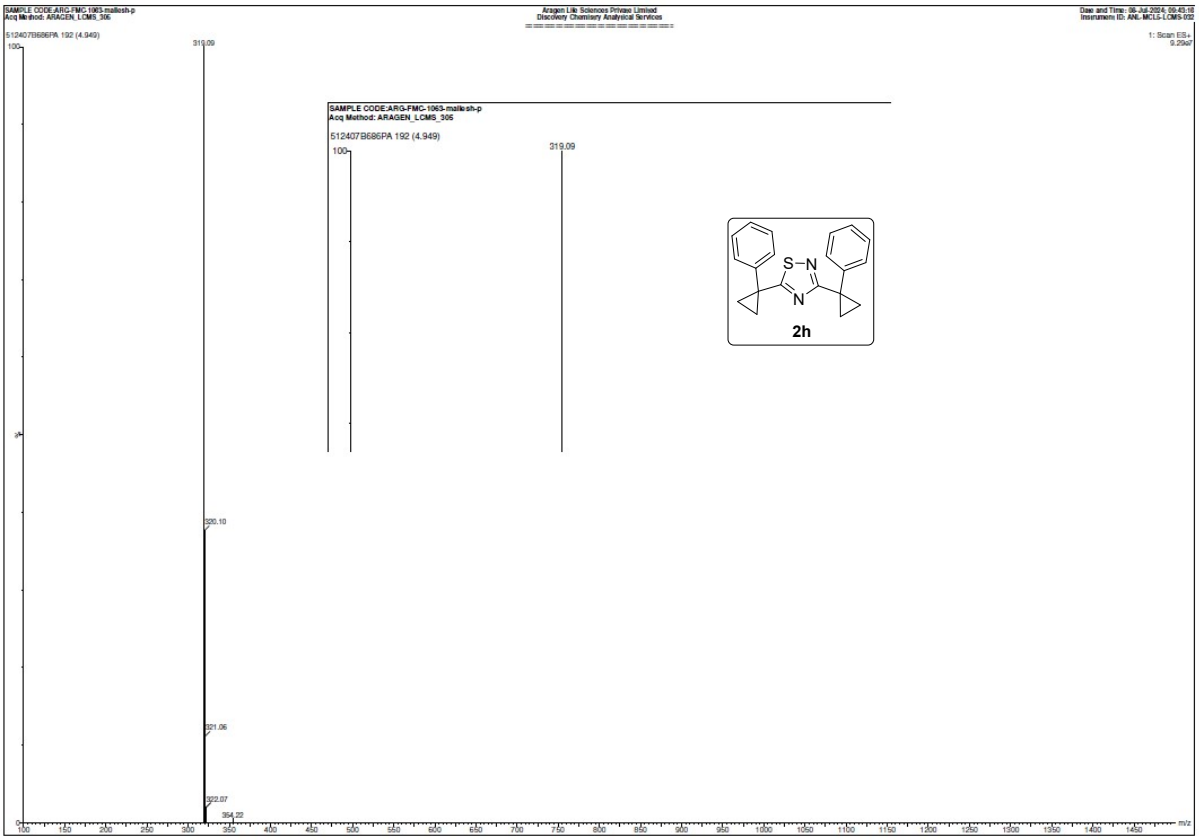
Current Data Parameters
NAME      512506B0566-C5747-94
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_              20250706
Time                 19.58 h
INSTRUM  Avance Neo Nanobay
PROBHD   Z163739_0485 (
PULPROG              zgpg30
TD                    65536
SOLVENT                DMSO
NS                     2000
DS                        4
SWH               23809.523 Hz
FIDRES             0.726609 Hz
AQ                1.3762560 sec
RG                     71.8
DW                   21.000 usec
DE                    6.50 usec
TE                  298.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1
SFO1            100.6228298 MHz
NUC1                    13C
P0                    2.67 usec
P1                    8.00 usec
PLW1            94.24700165 W
SFO2            400.1316005 MHz
NUC2                    1H
CPDPRG[2          waltz65
PCPD2                90.00 usec
PLW2            23.84199905 W
PLW12            0.18838000 W
PLW13            0.09475400 W

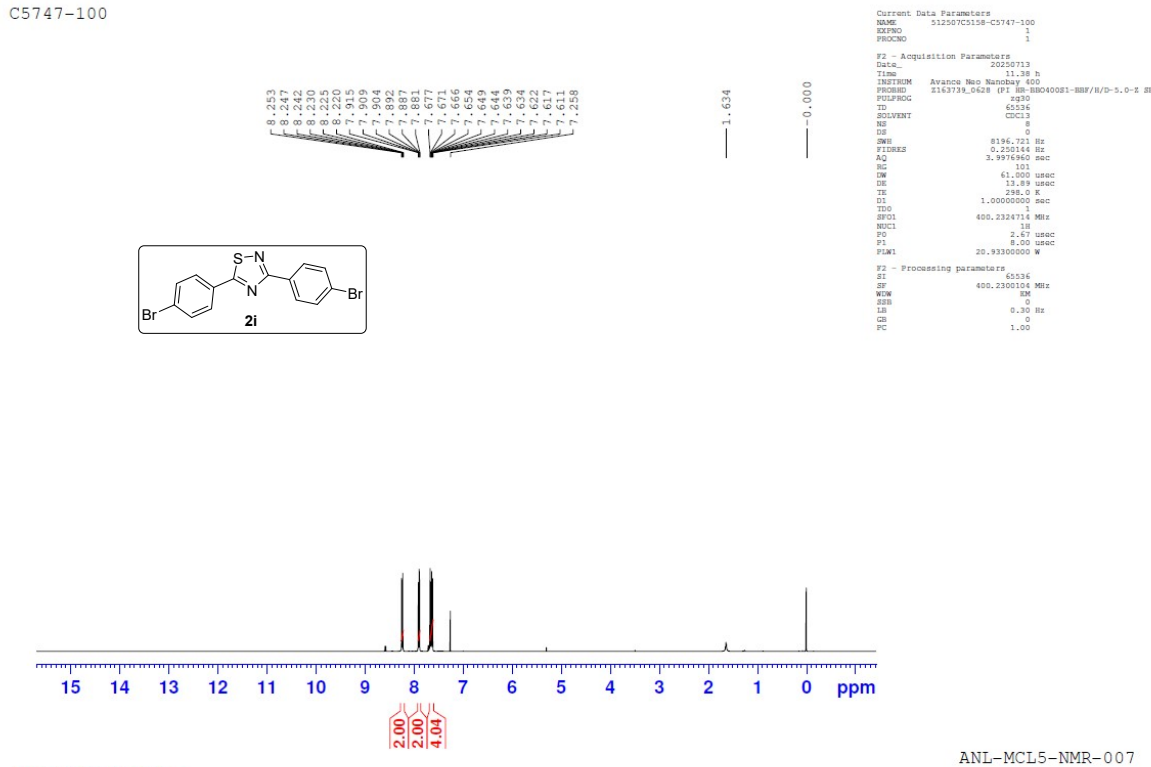
F2 - Processing parameters
SI                    32768
SF              100.6128200 MHz
WDW                     EM
SSB                      0
LB                    2.00 Hz
GB                       0
PC                    1.40
```

**LCMS spectrum of Compound (2h)**

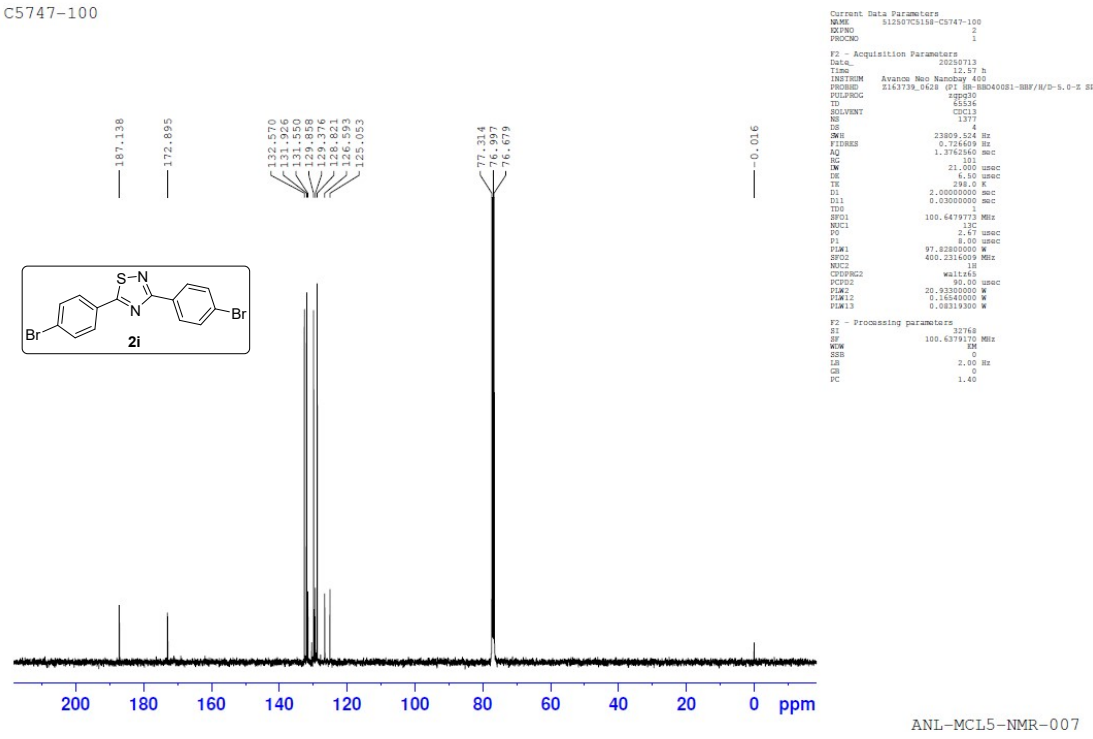

Aragen Life Sciences Private Limited
Discovery Chemistry Analytical Services
2h
Column: X-BRIDGE C8,3.5 um (4.6mm X75mm)
Mobile phase-A-B: 2.5mM Ammonium bi carbonate in Water and B: ACN
T% of B: 0/2, 0.3/2, 3.0/95, 4.0/98,6.5/97, 7.0/2, 8.0/2.
Flow-0.8 mL/min, Temp:35°C.
Diluent:ACN -WATER


Aragen Life Sciences Private Limited
Discovery Chemistry Analytical Services
SAMPLE CODE:ARG-FMC-1065-mallesh-p
Acq Method: ARAGEN_LCMS_30S
512407B686PA 192 (4.949)
319.09
320.10
321.06
322.07
2h

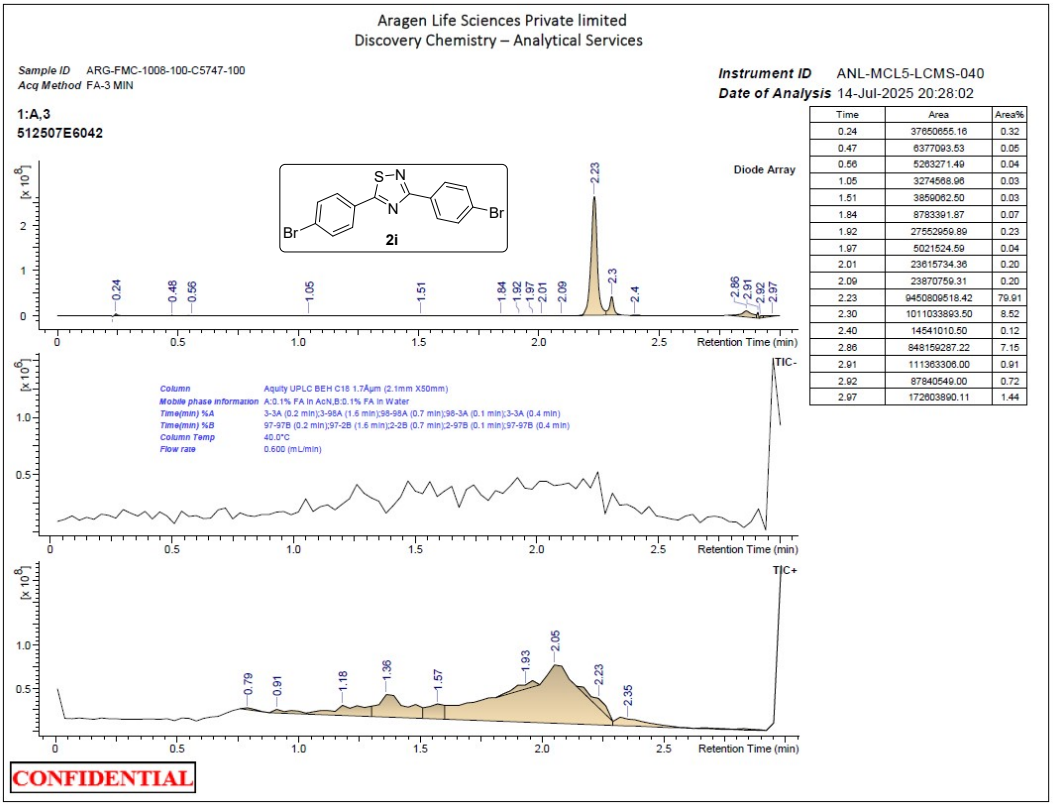
## $^{1}$H NMR spectrum (400 MHz) of Compound (2i) in $CDCl_3$



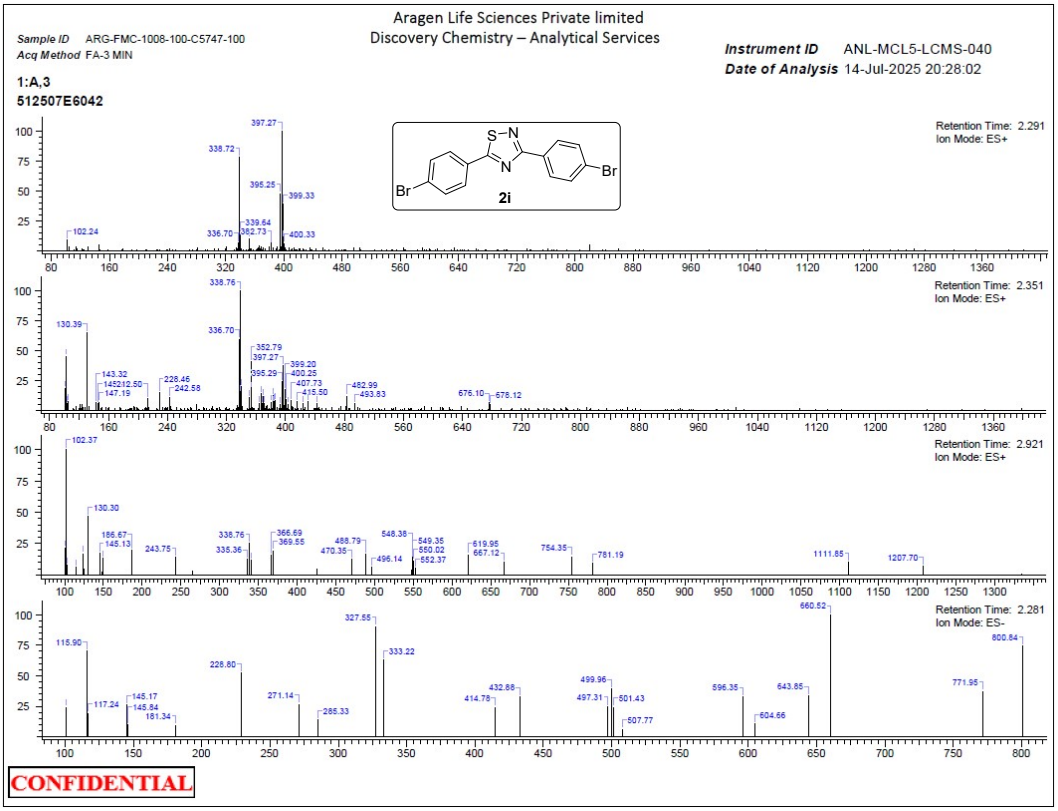

## $^{13}$C NMR spectrum (100 MHz) of Compound (2i) in DMSO-$d_6$



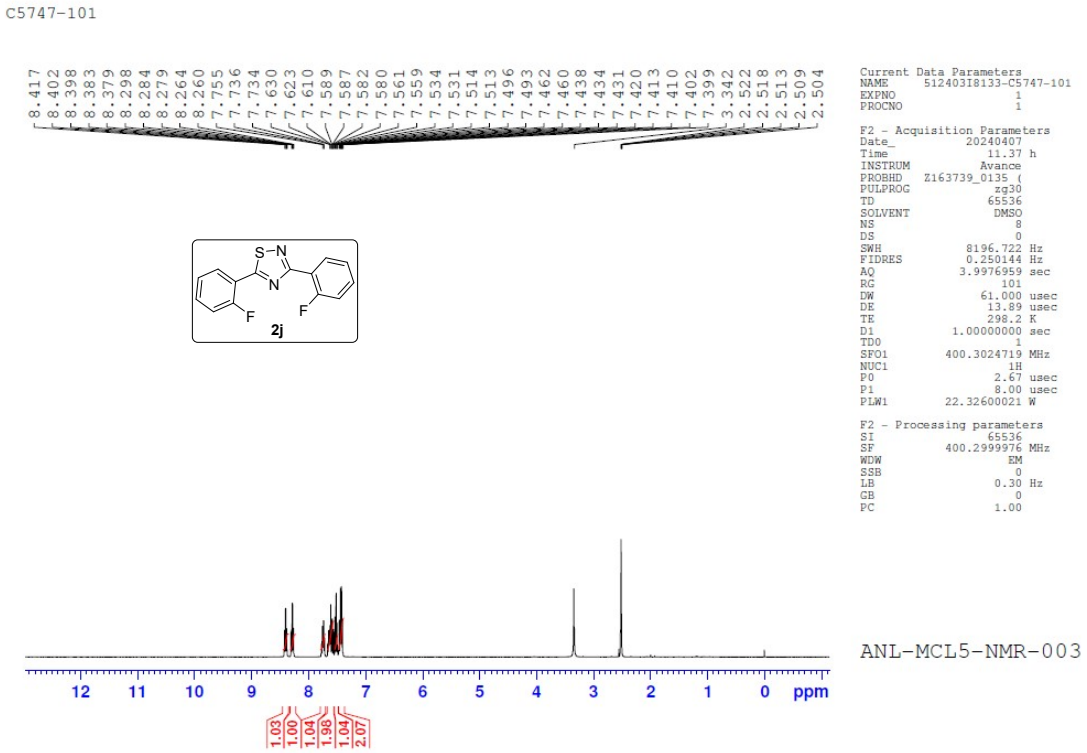
## LCMS spectrum of Compound (2i)




| Time | Area | Area% |
|---|---|---|
| 0.24 | 37650655.16 | 0.32 |
| 0.47 | 6377093.53 | 0.05 |
| 0.56 | 5263271.49 | 0.04 |
| 1.05 | 3274568.96 | 0.03 |
| 1.51 | 3859062.50 | 0.03 |
| 1.84 | 8783391.87 | 0.07 |
| 1.92 | 27552959.89 | 0.23 |
| 1.97 | 5021524.59 | 0.04 |
| 2.01 | 23615734.36 | 0.20 |
| 2.09 | 23870759.31 | 0.20 |
| 2.23 | 9450809518.42 | 79.91 |
| 2.30 | 1011033893.50 | 8.52 |
| 2.40 | 14541010.50 | 0.12 |
| 2.86 | 848159287.22 | 7.15 |
| 2.91 | 111363306.00 | 0.91 |
| 2.92 | 87840549.00 | 0.72 |
| 2.97 | 172603890.11 | 1.44 |

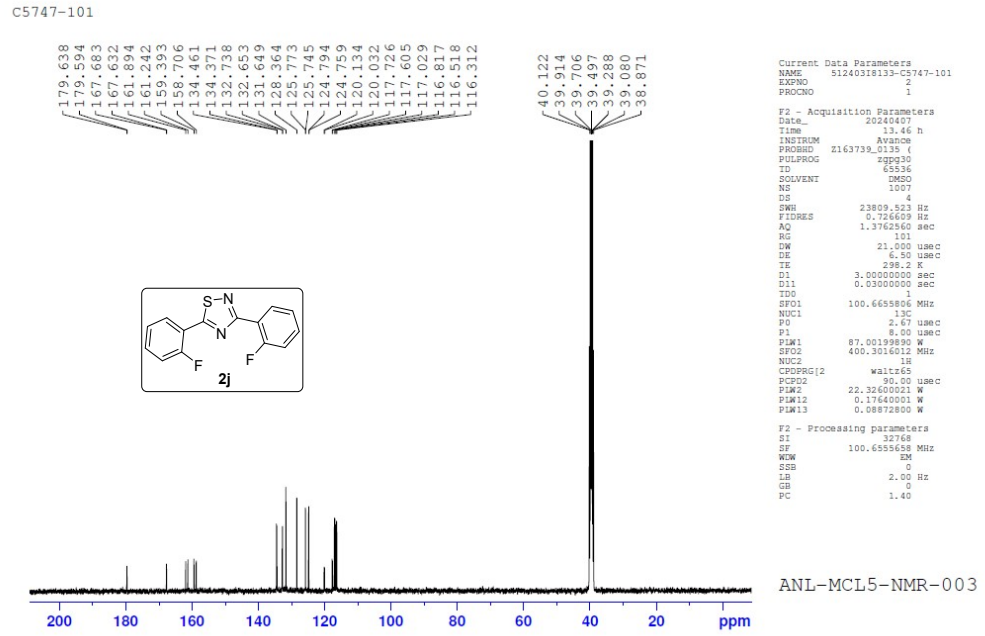


## $^1$H NMR spectrum (400 MHz) of Compound (2j) in DMSO-$d_6$

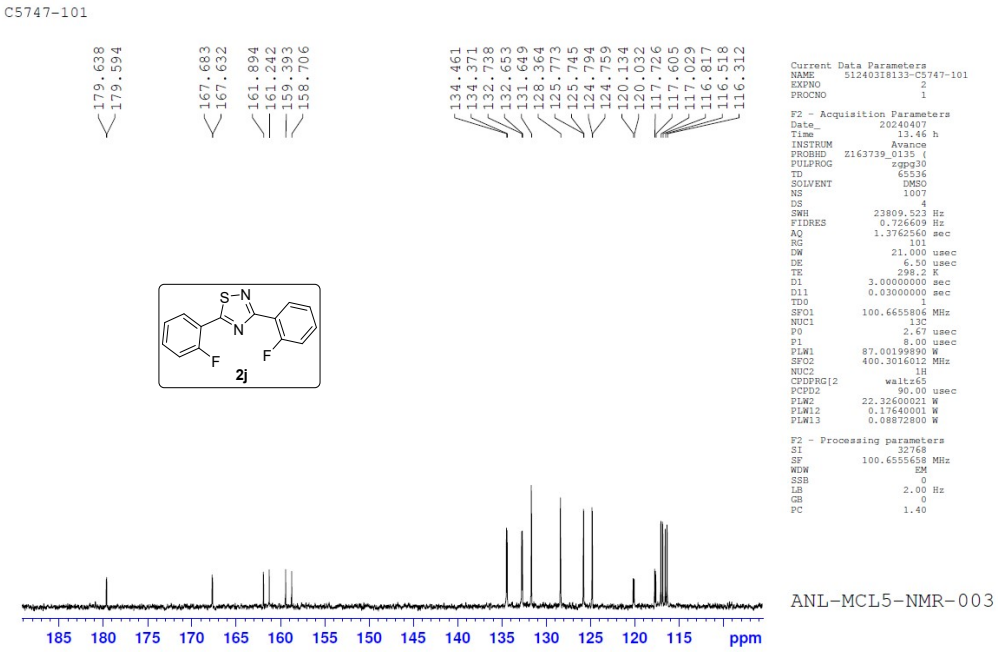



13

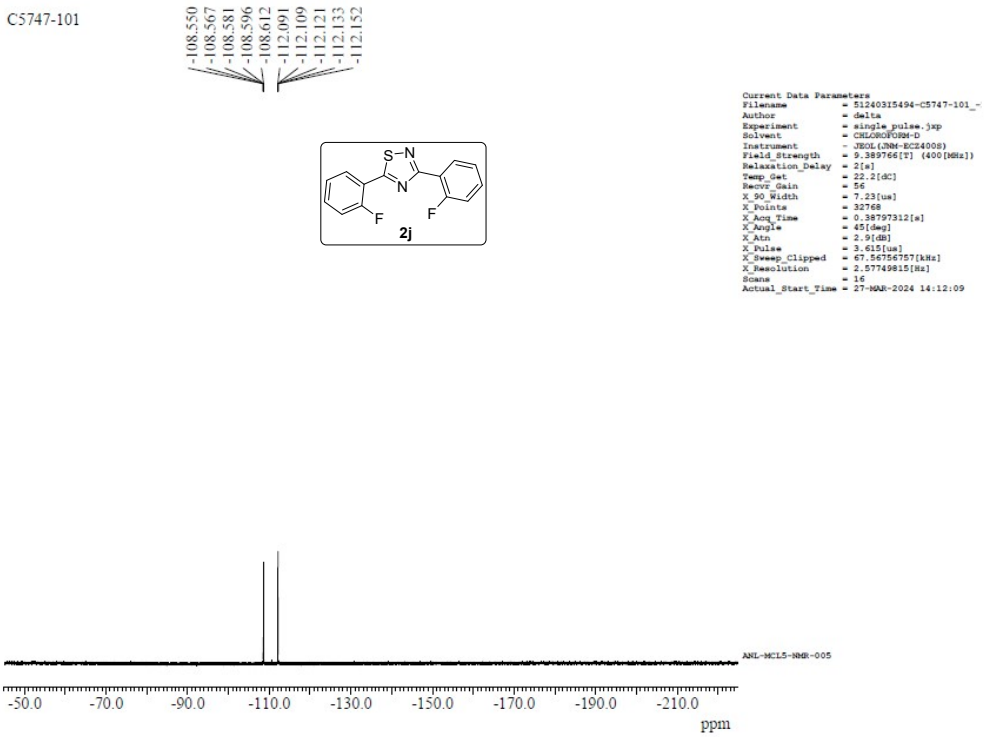
## C NMR spectrum (100 MHz) of Compound (2j) in DMSO-$d_6$



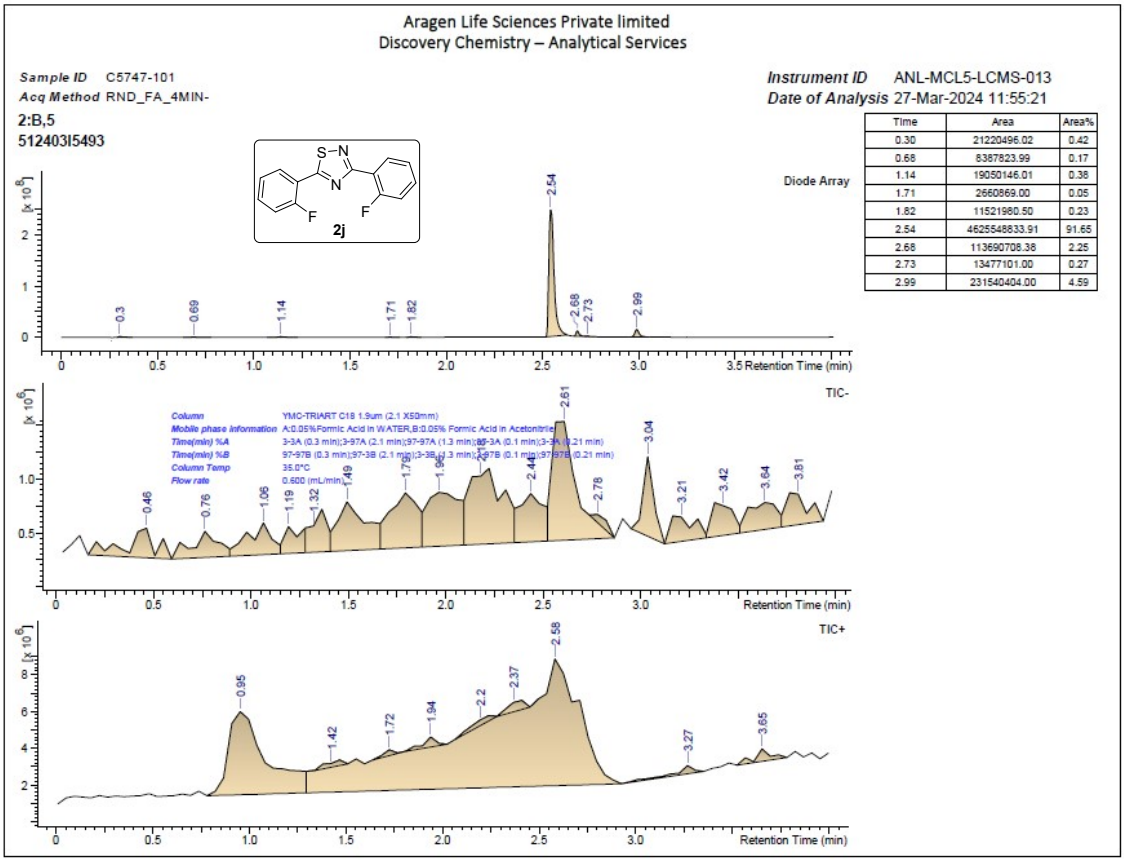
## $^{13}$C NMR spectrum (100 MHz) of Compound (2j) in DMSO-$d_6$ *(Expansion)*



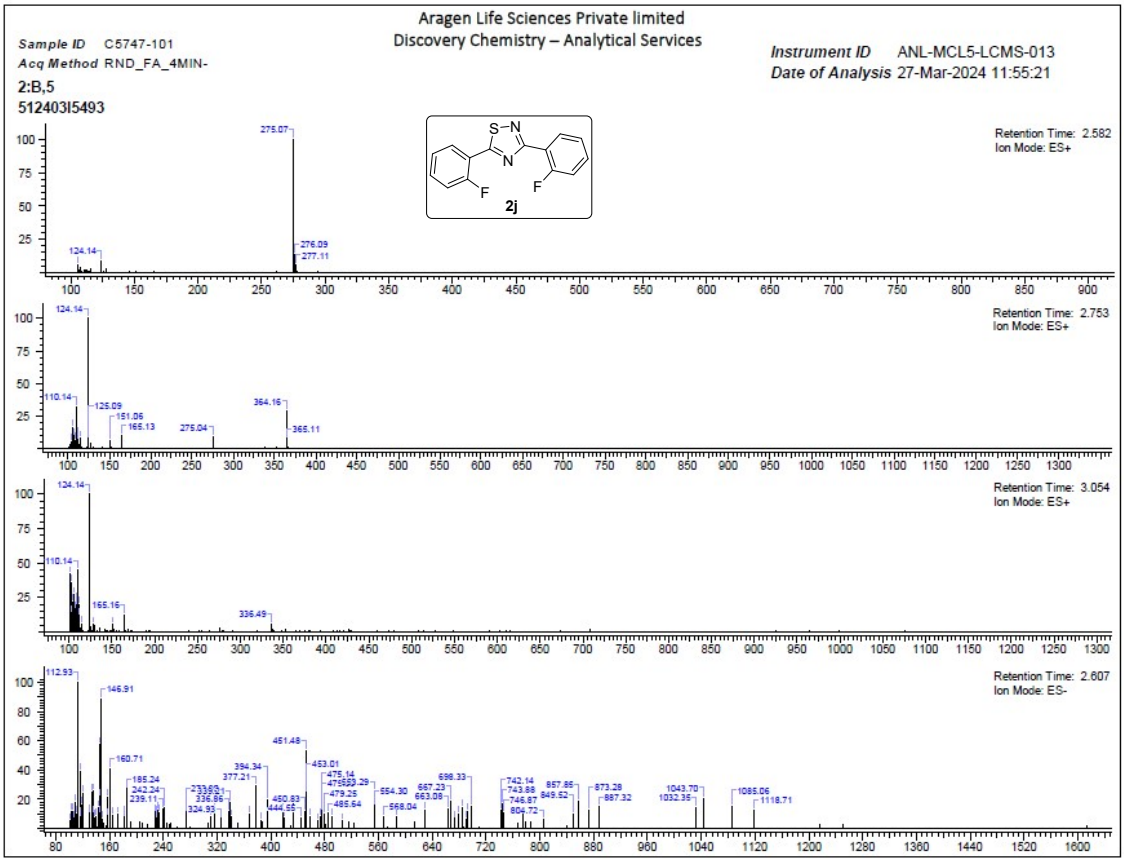

## 19-F NMR spectrum (400 MHz) of Compound (2j) in CDCl3



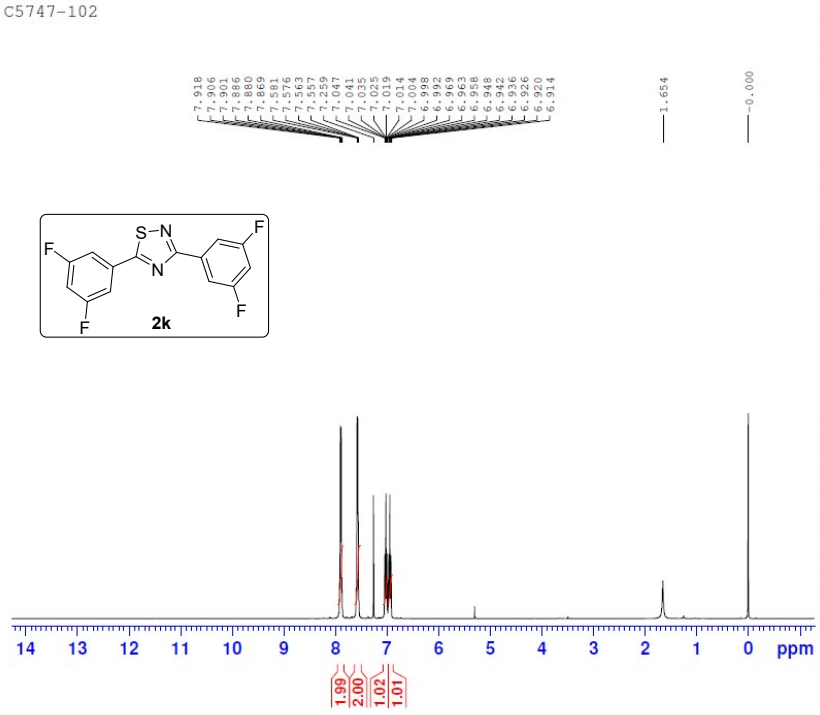
## LCMS spectrum of Compound (2j)




| Time | Area | Area% |
|---|---|---|
| 0.30 | 21220496.02 | 0.42 |
| 0.68 | 8387823.99 | 0.17 |
| 1.14 | 19050146.01 | 0.38 |
| 1.71 | 2660869.00 | 0.05 |
| 1.82 | 11521980.50 | 0.23 |
| 2.54 | 4625548833.91 | 91.65 |
| 2.68 | 113690708.38 | 2.25 |
| 2.73 | 13477101.00 | 0.27 |
| 2.99 | 231540404.00 | 4.59 |



## $^{1}$H NMR spectrum (400 MHz) of Compound (2k) in $CDCl_3$

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EXPNO                 1
PROCNO                1

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INSTRUM           Avance
PROBHD    Z163739_0686 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                   101
DW                61.000 usec
DE                13.89 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1          400.6324739 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          23.67700005 W

F2 - Processing parameters
SI                 65536
SF            400.6300101 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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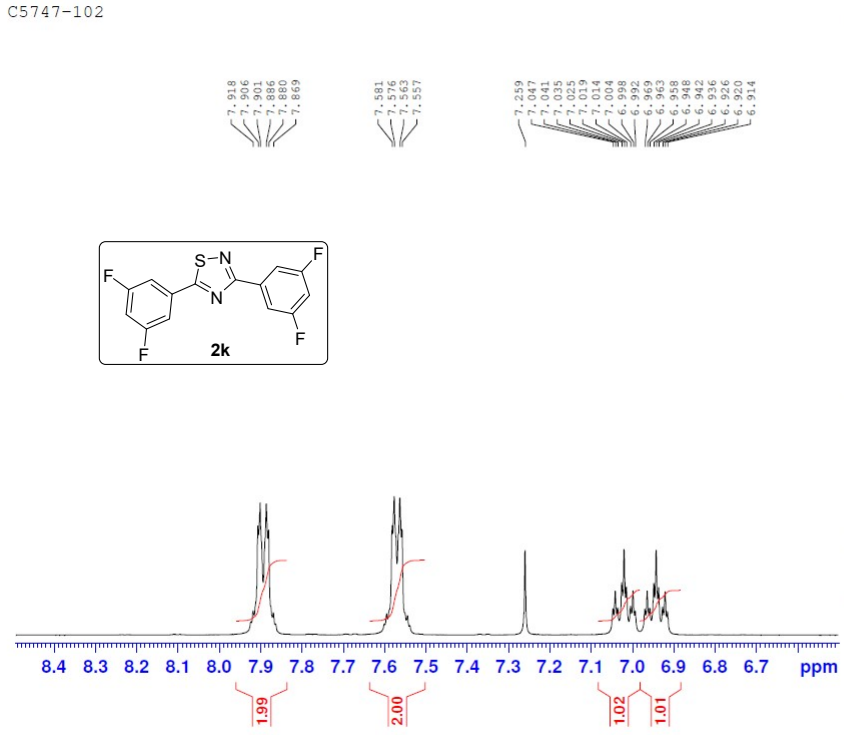
ANL-MCL5-NMR-010

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Current Data Parameters
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EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
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INSTRUM           Avance
PROBHD    Z163739_0686 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                   101
DW                61.000 usec
DE                13.89 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1          400.6324739 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          23.67700005 W

F2 - Processing parameters
SI                 65536
SF            400.6300101 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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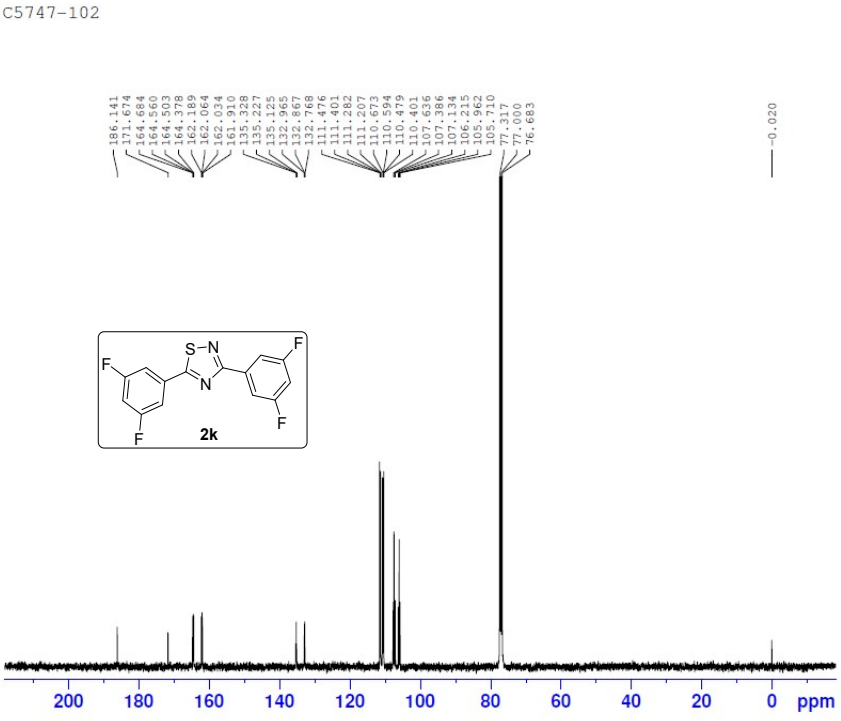
ANL-MCL5-NMR-010

## $^{13}C$ NMR spectrum (100 MHz) of Compound (2k) in $CDCl_3$





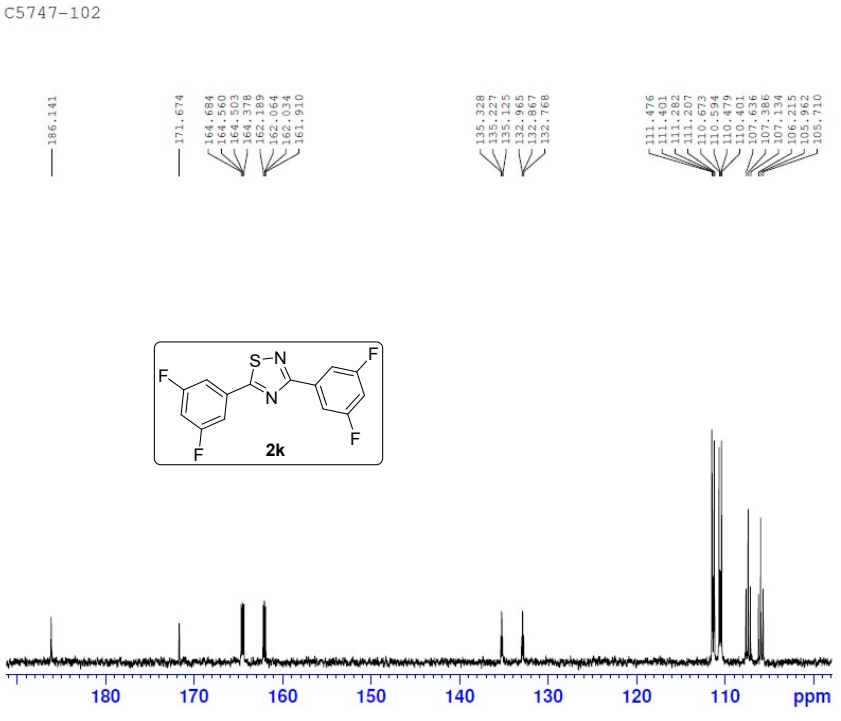
Current Data Parameters
NAME 512507C5161-C5747-102
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20250713
Time 11.36 h
INSTRUM Avance
PROBHD Z163739_0686 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1500
DS 4
SWH 23809.523 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 101
DW 21.000 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.7485675 MHz
NUC1 13C
P0 2.67 usec
P1 8.00 usec
PLW1 94.71600342 W
SFO2 400.6316025 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 90.00 usec
PLW2 23.67700005 W
PLW12 0.18708000 W
PLW13 0.09409900 W

F2 - Processing parameters
SI 32768
SF 100.7384954 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40







Current Data Parameters
NAME 512507C5161-C5747-102
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20250713
Time 11.36 h
INSTRUM Avance
PROBHD Z163739_0686 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1500
DS 4
SWH 23809.523 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 101
DW 21.000 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.7485675 MHz
NUC1 13C
P0 2.67 usec
P1 8.00 usec
PLW1 94.71600342 W
SFO2 400.6316025 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 90.00 usec
PLW2 23.67700005 W
PLW12 0.18708000 W
PLW13 0.09409900 W

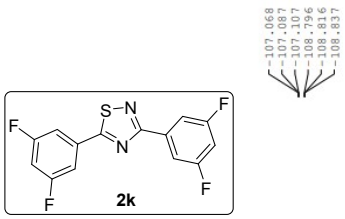
F2 - Processing parameters
SI 32768
SF 100.7384954 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40






## 19-F NMR spectrum (400 MHz) of Compound (2k) in CDCl3

C5747-102







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Current Data Parameters
NAME      512507C5161-C5747-102
EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
Date_          20250713
Time             17.05 h
INSTRUM          Avance
PROBHD   Z163739_0686 (
PULPROG             zg
TD              131072
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           90909.094 Hz
FIDRES         1.387163 Hz
AQ            0.7208960 sec
RG                  101
DW                5.500 usec
DE                6.50 usec
TE               298.2 K
D1           1.00000000 sec
TD0                  1
SFO1        376.9311394 MHz
NUC1               19F
P1                12.00 usec
PLW1        33.36899948 W

F2 - Processing parameters
SI                65536
SF          376.9688363 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.00
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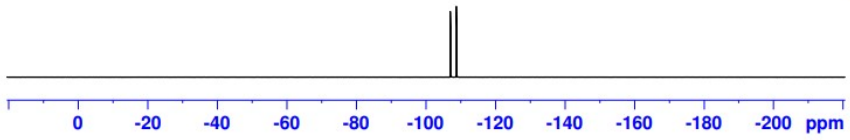
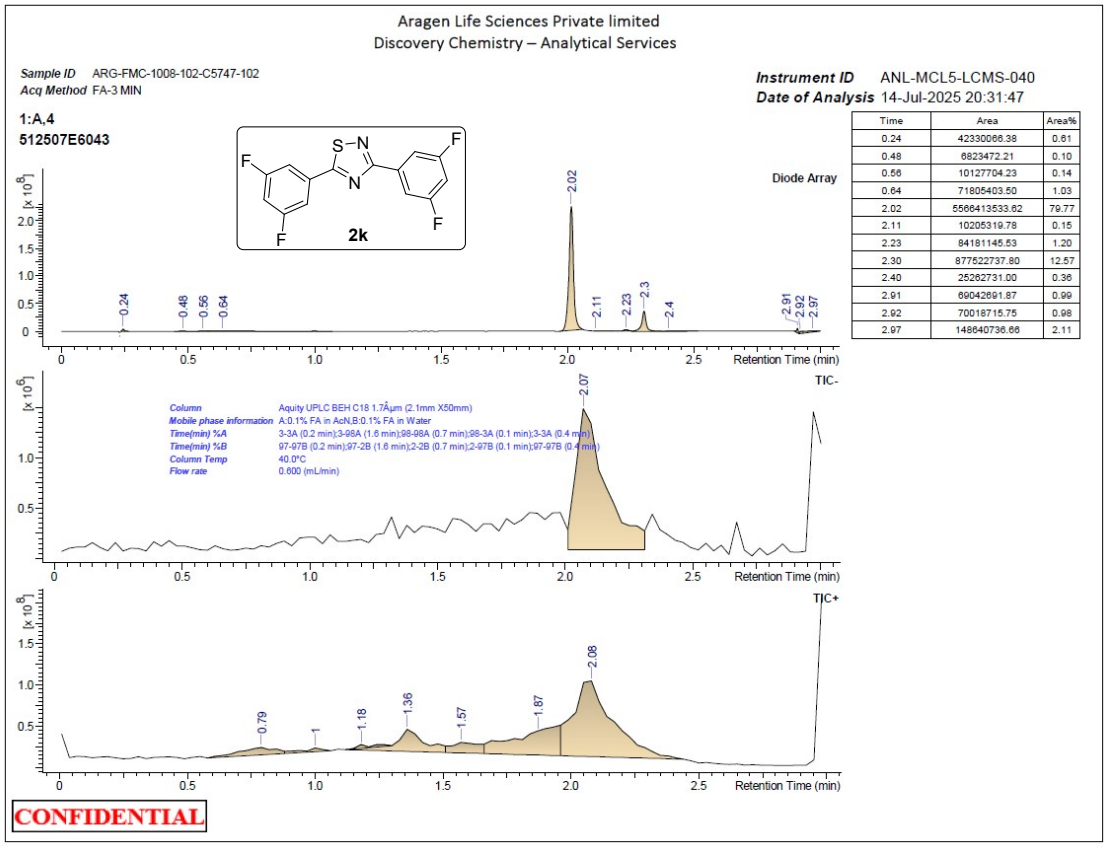


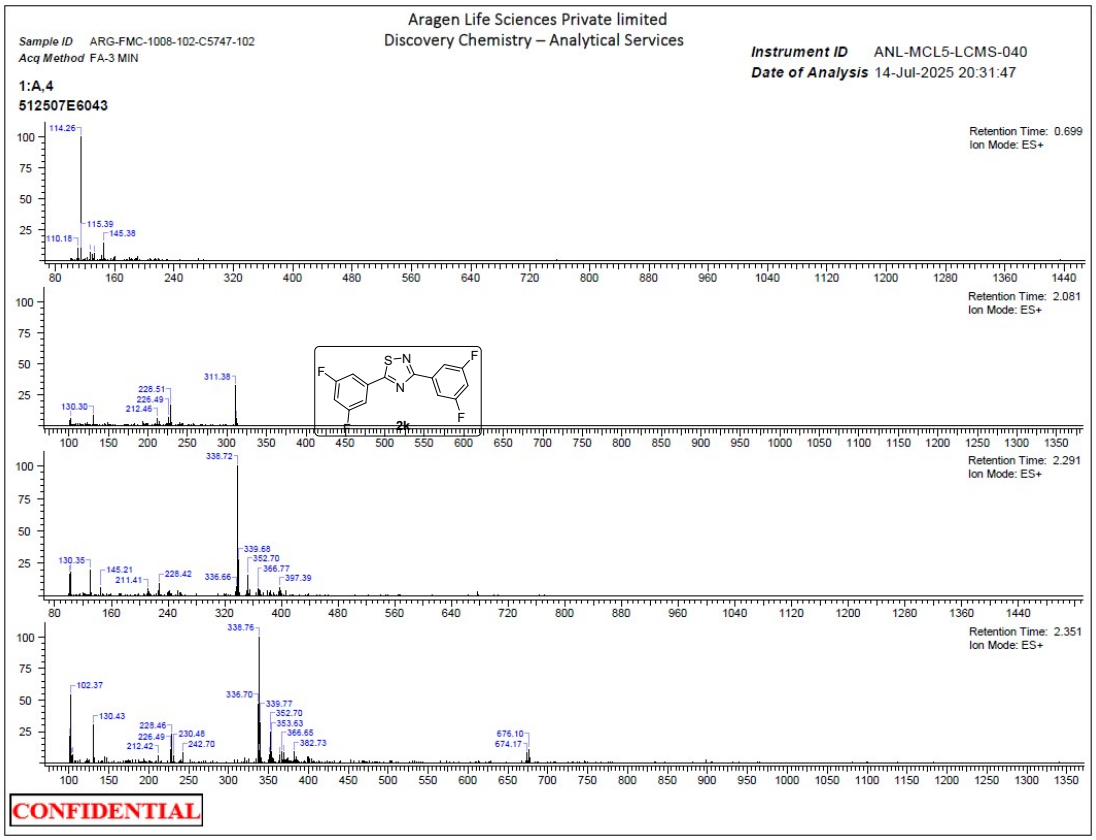

ANL-MCL5-NMR-010



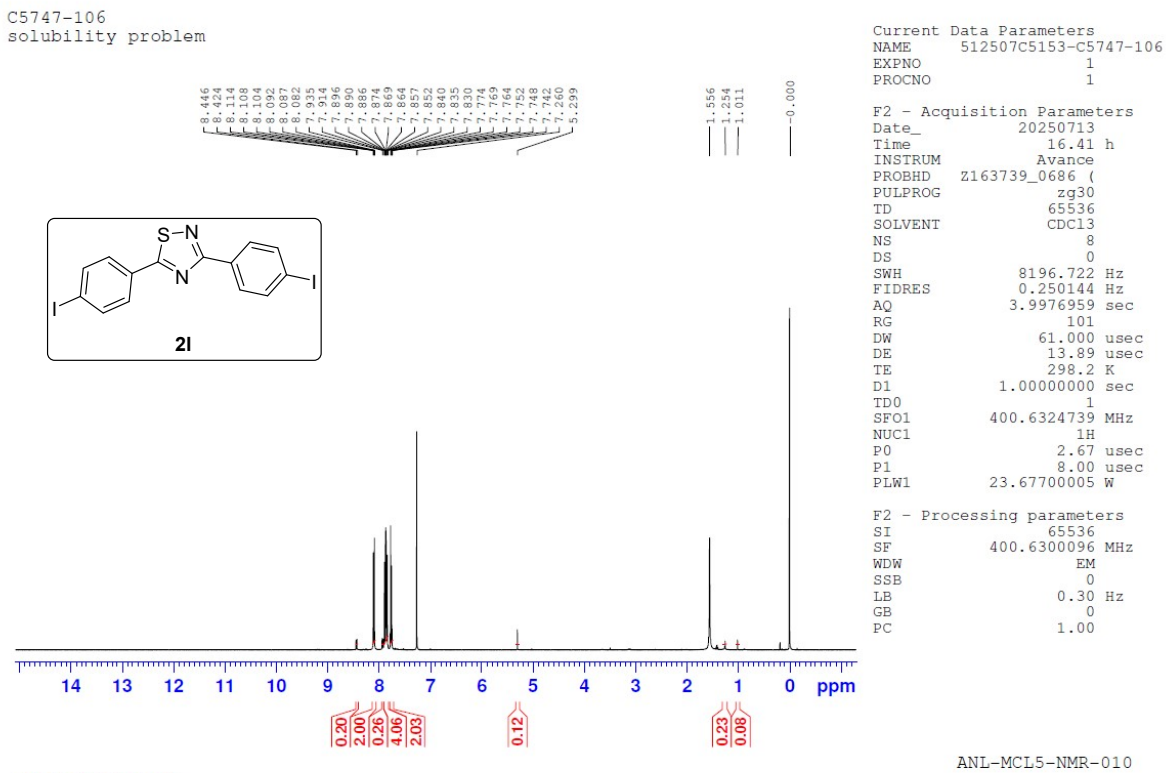
**LCMS spectrum of Compound (2k)**




| Time | Area | Area% |
|---|---|---|
| 0.24 | 42330066.38 | 0.61 |
| 0.48 | 6823472.21 | 0.10 |
| 0.56 | 10127704.23 | 0.14 |
| 0.64 | 71805403.50 | 1.03 |
| 2.02 | 5566413533.62 | 79.77 |
| 2.11 | 10205319.78 | 0.15 |
| 2.23 | 84181145.53 | 1.20 |
| 2.30 | 877522737.80 | 12.57 |
| 2.40 | 25262731.00 | 0.36 |
| 2.91 | 69042691.87 | 0.99 |
| 2.92 | 70018715.75 | 0.98 |
| 2.97 | 148640736.66 | 2.11 |



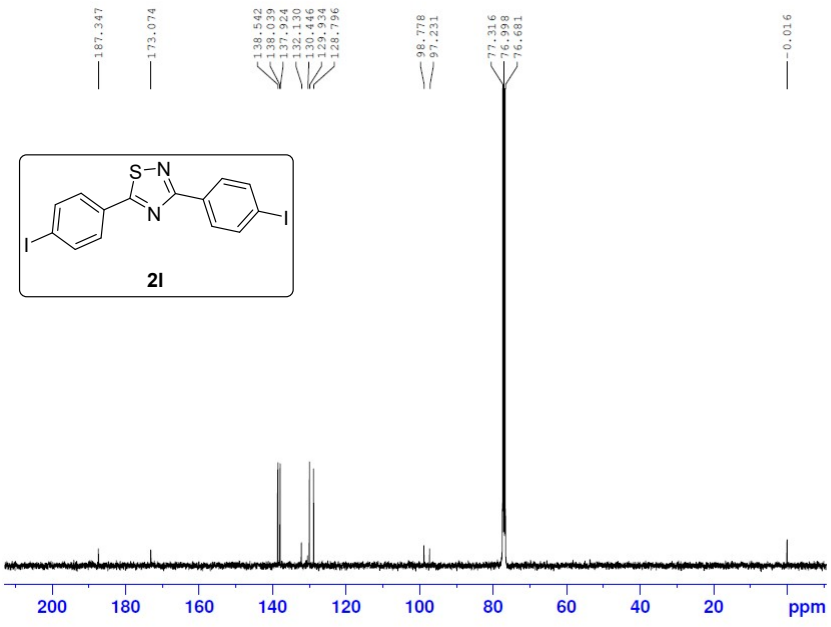

**$^{1}$H NMR spectrum (400 MHz) of Compound (2l) in $CDCl_3$**






**$^{13}$C NMR spectrum (100 MHz) of Compound (2l) in $CDCl_3$**

C5747-106
solubility problem




```
Current Data Parameters
NAME      512507C5153-C5747-106
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_           20250713
Time              21.42 h
INSTRUM           Avance
PROBHD   Z163739_0686 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2000
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                  101
DW               21.000 usec
DE                6.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1         100.7485675 MHz
NUC1                13C
P0                 2.67 usec
P1                 8.00 usec
PLW1         94.71600342 W
SFO2         400.6316025 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         23.67700005 W
PLW12         0.18708000 W
PLW13         0.09409900 W

F2 - Processing parameters
SI                 32768
SF           100.7384957 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.40
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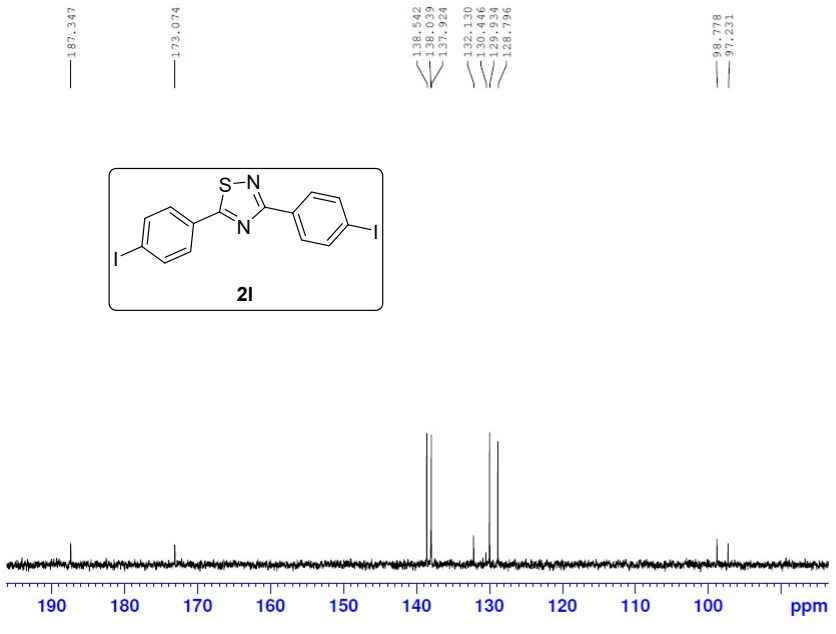
ANL-MCL5-NMR-010

CONFIDENTIAL

C5747-106
solubility problem

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Current Data Parameters
NAME      512507C5153-C5747-106
EXPNO                 3
PROCNO                1

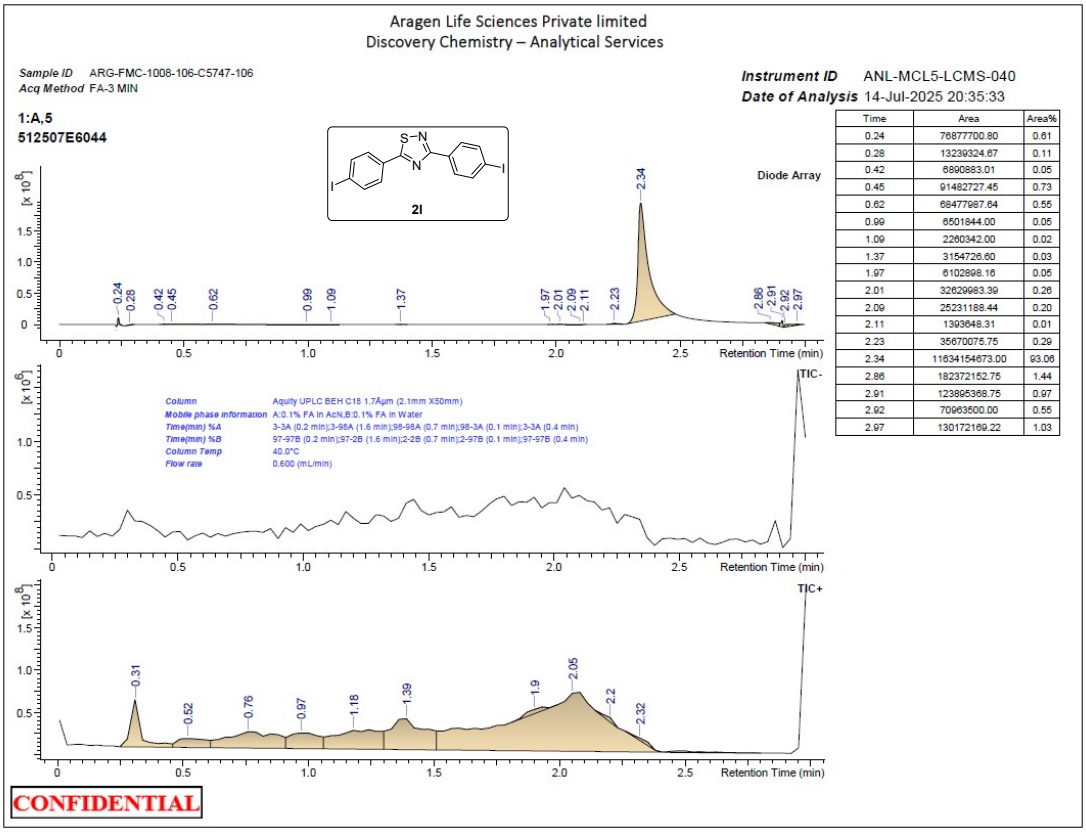
F2 - Acquisition Parameters
Date_           20250713
Time              21.42 h
INSTRUM           Avance
PROBHD   Z163739_0686 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2000
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                  101
DW               21.000 usec
DE                6.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1         100.7485675 MHz
NUC1                13C
P0                 2.67 usec
P1                 8.00 usec
PLW1         94.71600342 W
SFO2         400.6316025 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         23.67700005 W
PLW12         0.18708000 W
PLW13         0.09409900 W

F2 - Processing parameters
SI                 32768
SF           100.7384957 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.40
```

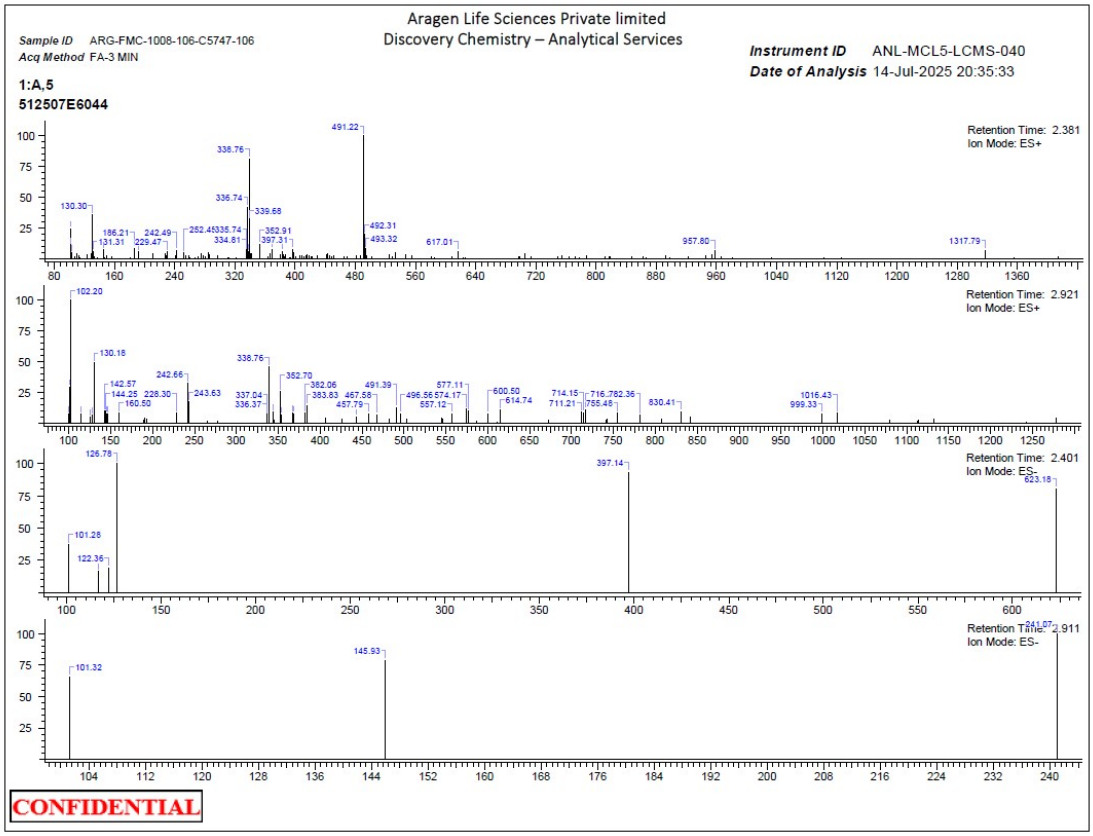
ANL-MCL5-NMR-010

CONFIDENTIAL

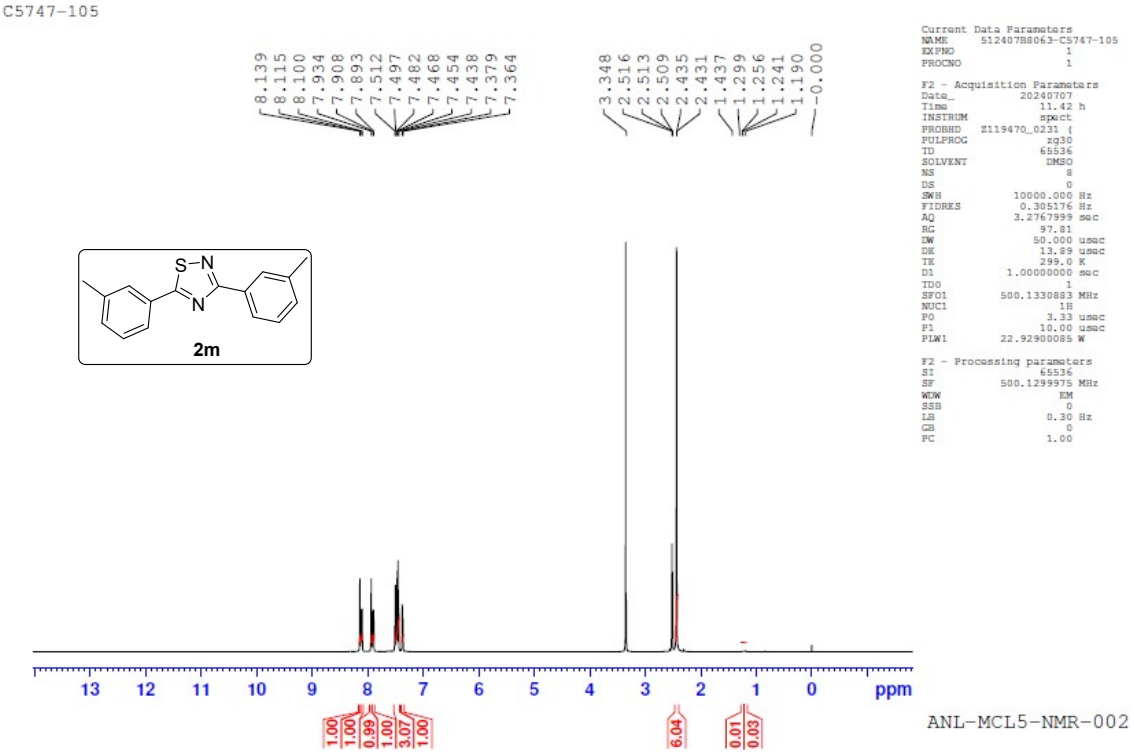
## LCMS spectrum of Compound (2l)




| Time | Area | Area% |
|---|---|---|
| 0.24 | 76877700.80 | 0.61 |
| 0.28 | 13239324.67 | 0.11 |
| 0.42 | 6890883.01 | 0.05 |
| 0.45 | 91482727.45 | 0.73 |
| 0.62 | 68477987.64 | 0.55 |
| 0.99 | 6501844.00 | 0.05 |
| 1.09 | 2260342.00 | 0.02 |
| 1.37 | 3154726.60 | 0.03 |
| 1.97 | 6102898.16 | 0.05 |
| 2.01 | 32629983.39 | 0.26 |
| 2.09 | 25231188.44 | 0.20 |
| 2.11 | 1393648.31 | 0.01 |
| 2.23 | 35670075.75 | 0.29 |
| 2.34 | 11634154673.00 | 93.06 |
| 2.86 | 182372152.75 | 1.44 |
| 2.91 | 123895368.75 | 0.97 |
| 2.92 | 70963500.00 | 0.55 |
| 2.97 | 130172169.22 | 1.03 |




## $^{1}$H NMR spectrum (500 MHz) of Compound (2m) in DMSO-$d_6$



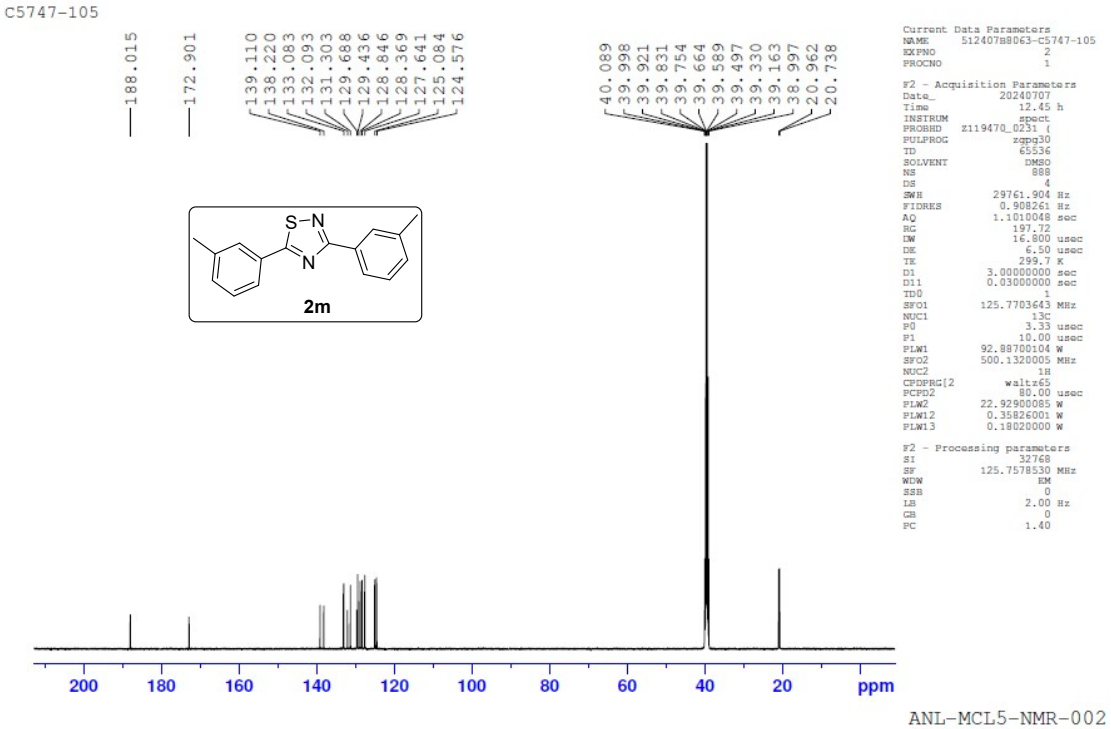


Current Data Parameters
NAME 512407B8063-C5747-105
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240707
Time 11.42 h
INSTRUM spect
PROBHD Z119470_0231 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 97.81
DW 50.000 usec
DE 13.89 usec
TE 299.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.92900085 W

F2 - Processing parameters
SI 65536
SF 500.1299975 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


**$^{13}$C NMR spectrum (125 MHz) of Compound (2m) in DMSO-$d_6$**



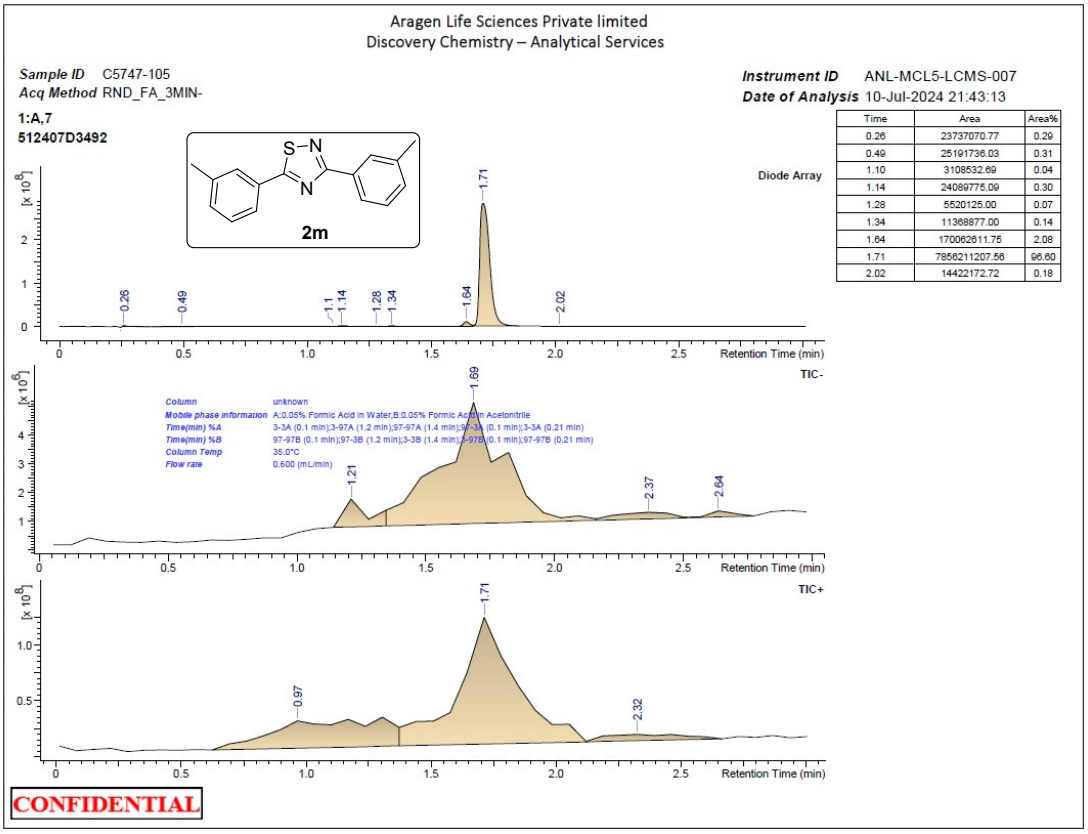


Current Data Parameters
NAME 512407B8063-C5747-105
EXPNO 2
PROCNO 1

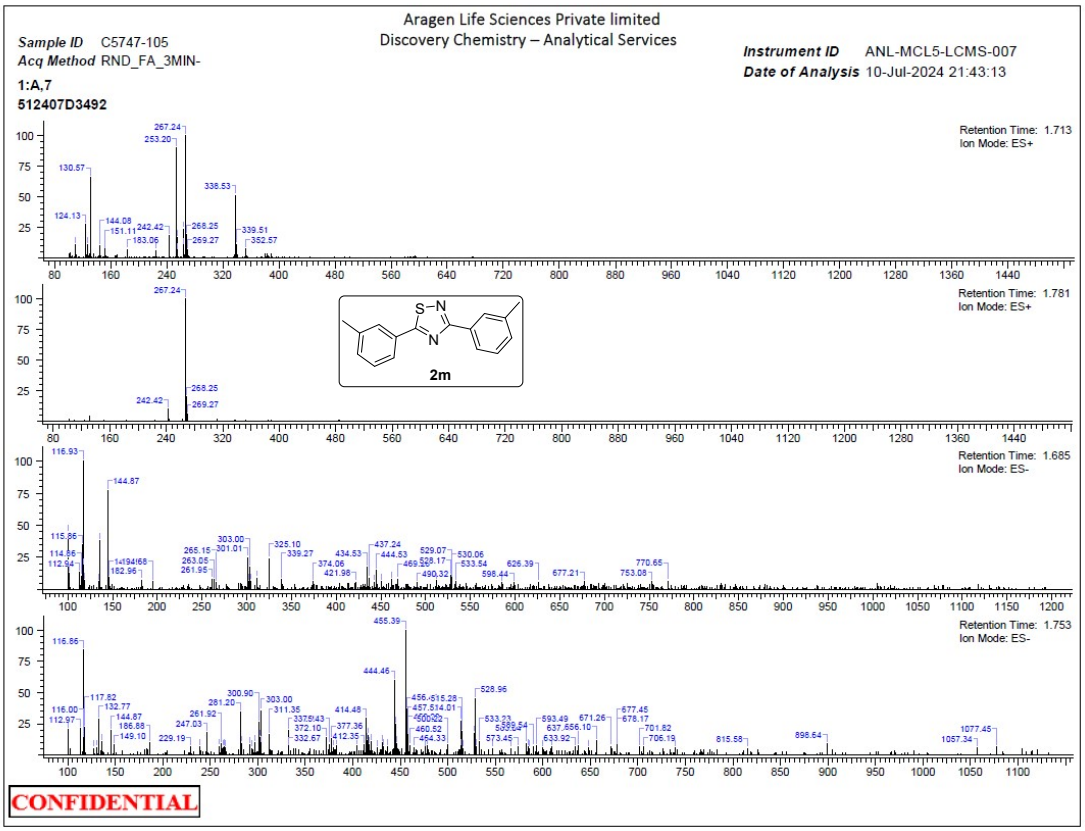
F2 - Acquisition Parameters
Date_ 20240707
Time 12.45 h
INSTRUM spect
PROBHD Z119470_0231 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 888
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 197.72
DW 16.800 usec
DE 6.50 usec
TE 299.7 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 92.88700104 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 80.00 usec
PLW2 22.92900085 W
PLW12 0.35826001 W
PLW13 0.18020000 W

F2 - Processing parameters
SI 32768
SF 125.7578530 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

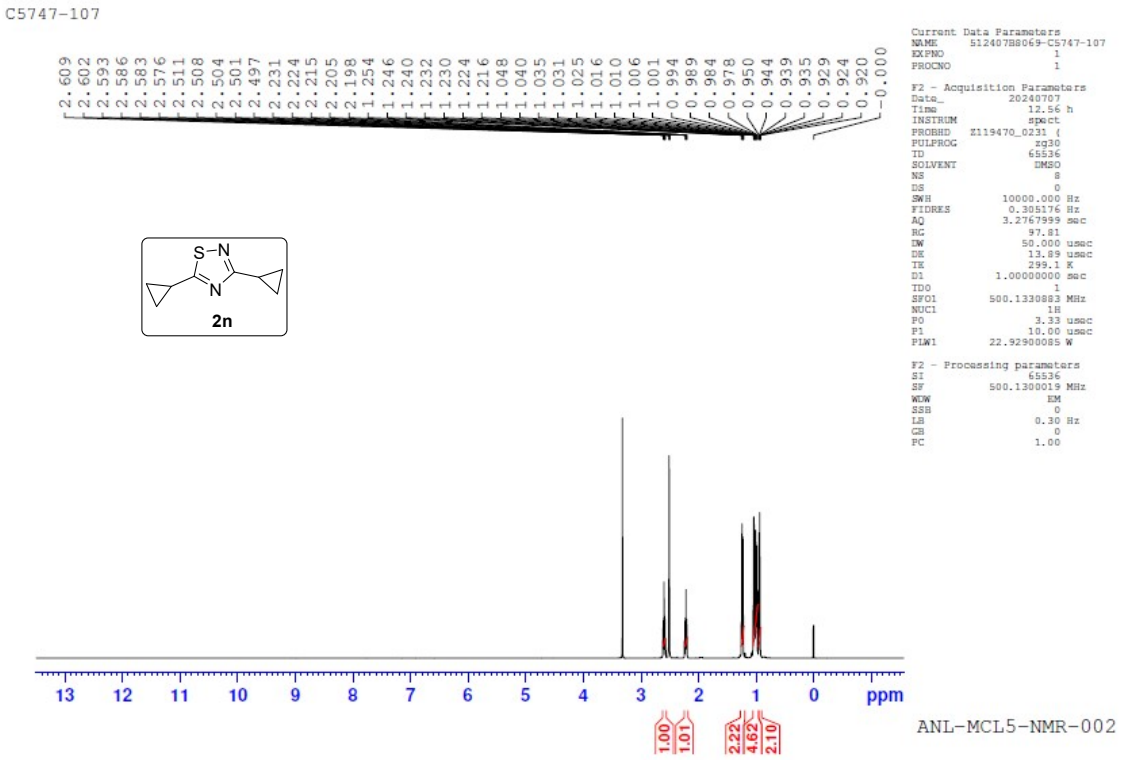

**LCMS spectrum of Compound (2m)**



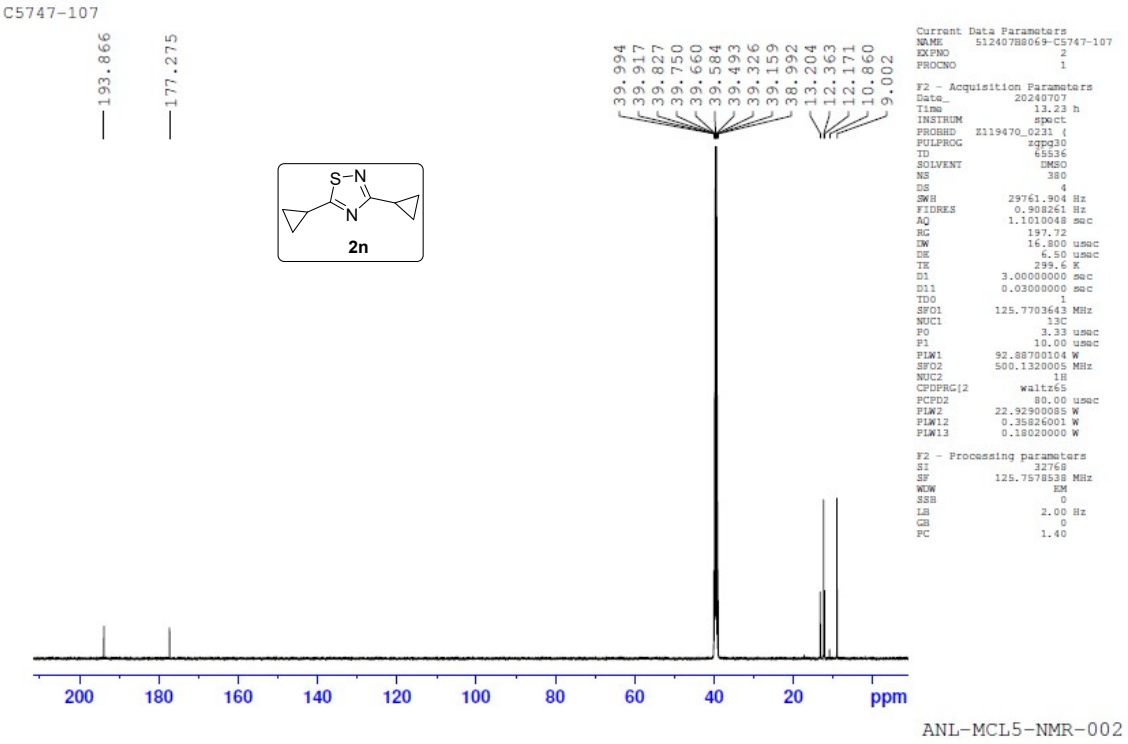

| Time | Area | Area% |
|---|---|---|
| 0.26 | 23737070.77 | 0.29 |
| 0.49 | 25191736.03 | 0.31 |
| 1.10 | 3108532.69 | 0.04 |
| 1.14 | 24089775.09 | 0.30 |
| 1.28 | 5520125.00 | 0.07 |
| 1.34 | 11368877.00 | 0.14 |
| 1.64 | 170062611.75 | 2.08 |
| 1.71 | 7856211207.56 | 96.60 |
| 2.02 | 14422172.72 | 0.18 |




**$^1$H NMR spectrum (500 MHz) of Compound (2n) in DMSO-$d_6$**

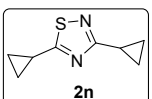


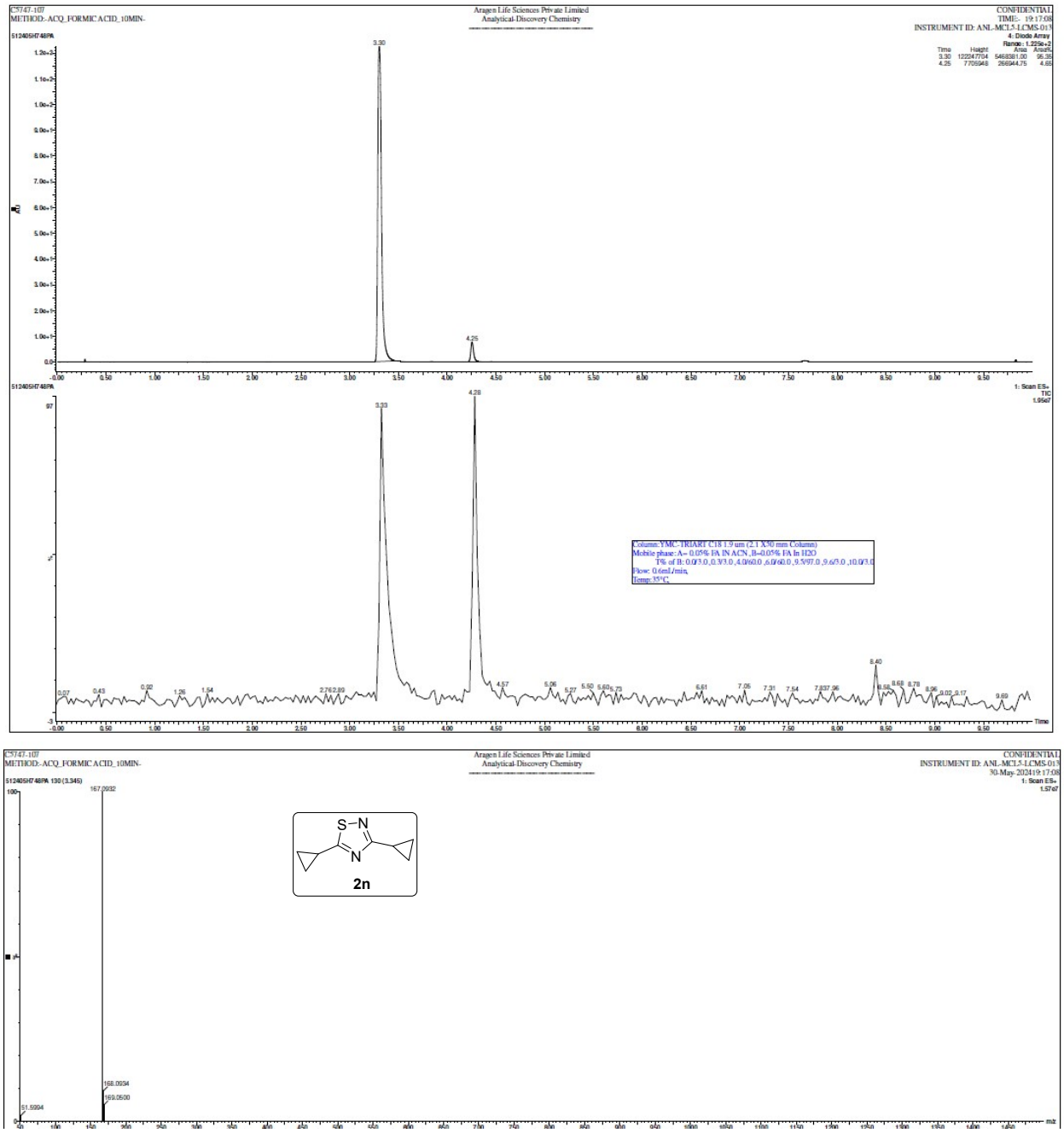

**$^{13}$C NMR spectrum (100 MHz) of Compound (2n) in DMSO-$d_6$**




**LCMS spectrum of Compound (2n)**



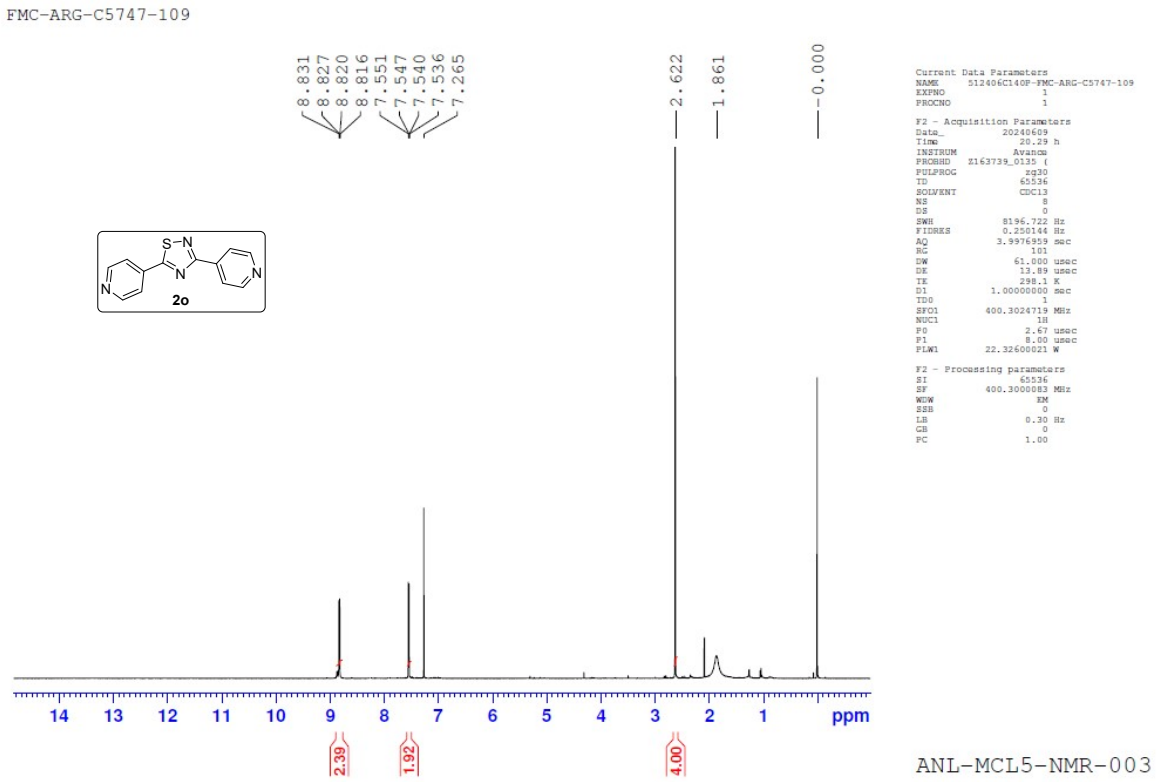




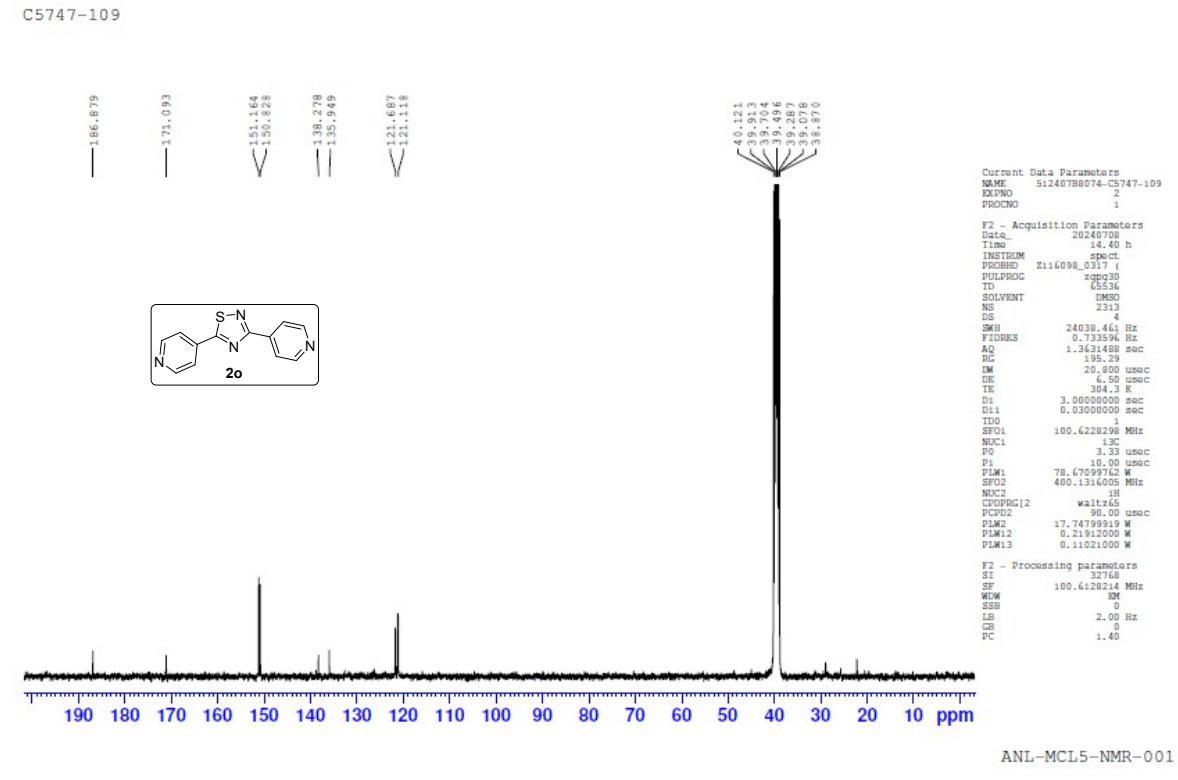




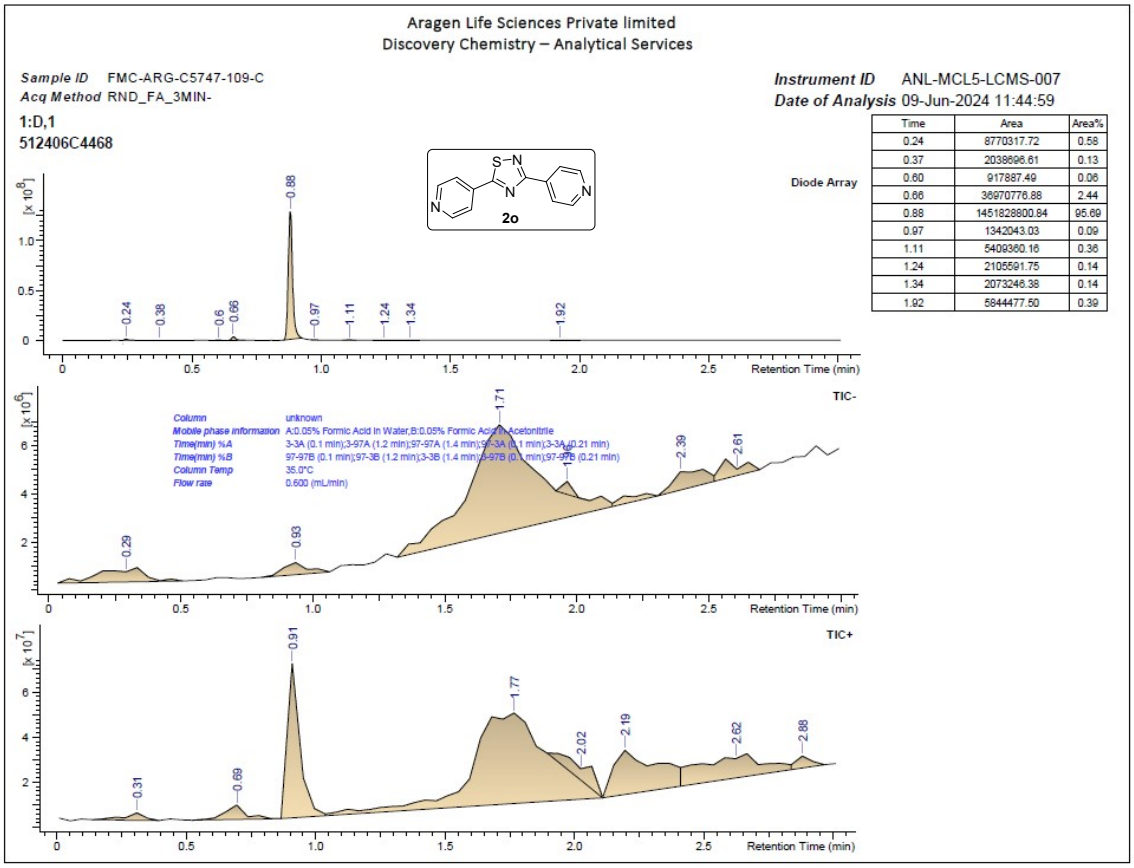
**$^{1}$H NMR spectrum (400 MHz) of Compound (2o) in DMSO-$d_6$**

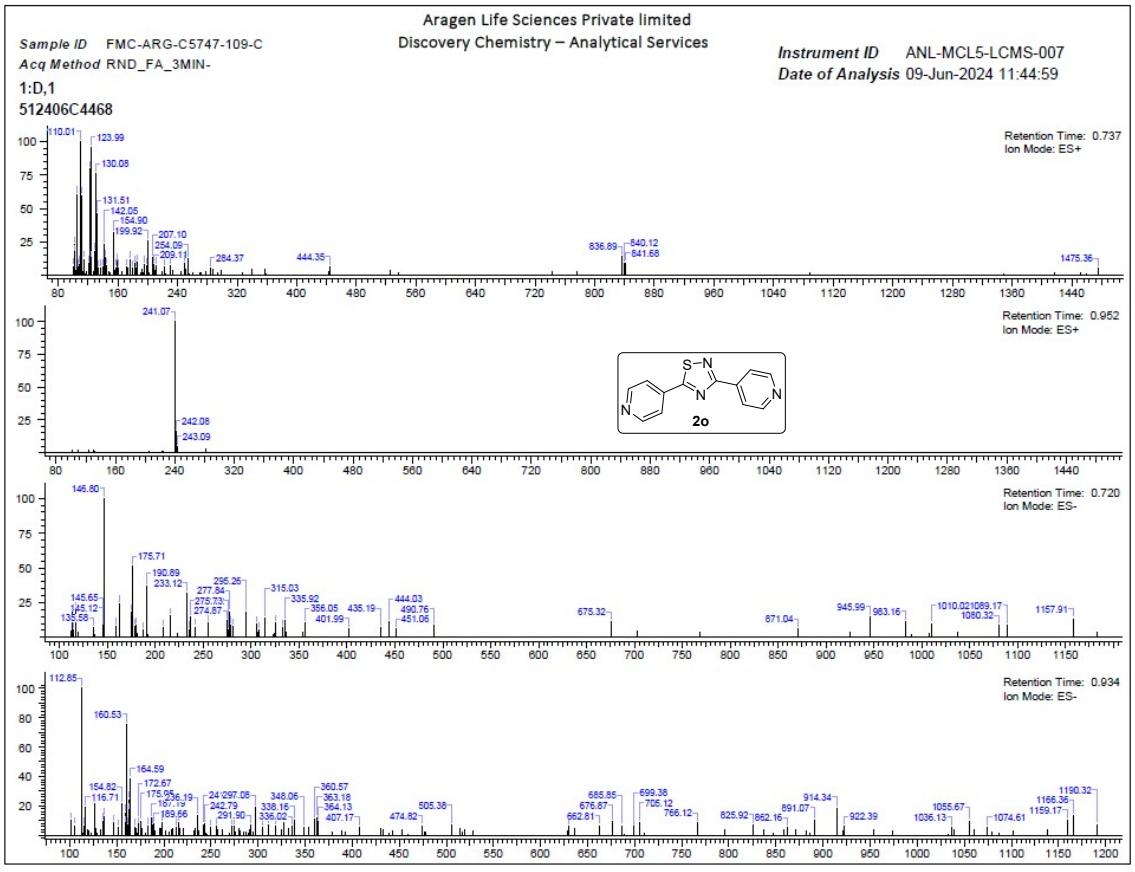



**$^{13}$C NMR spectrum (100 MHz) of Compound (2o) in DMSO-$d_6$**

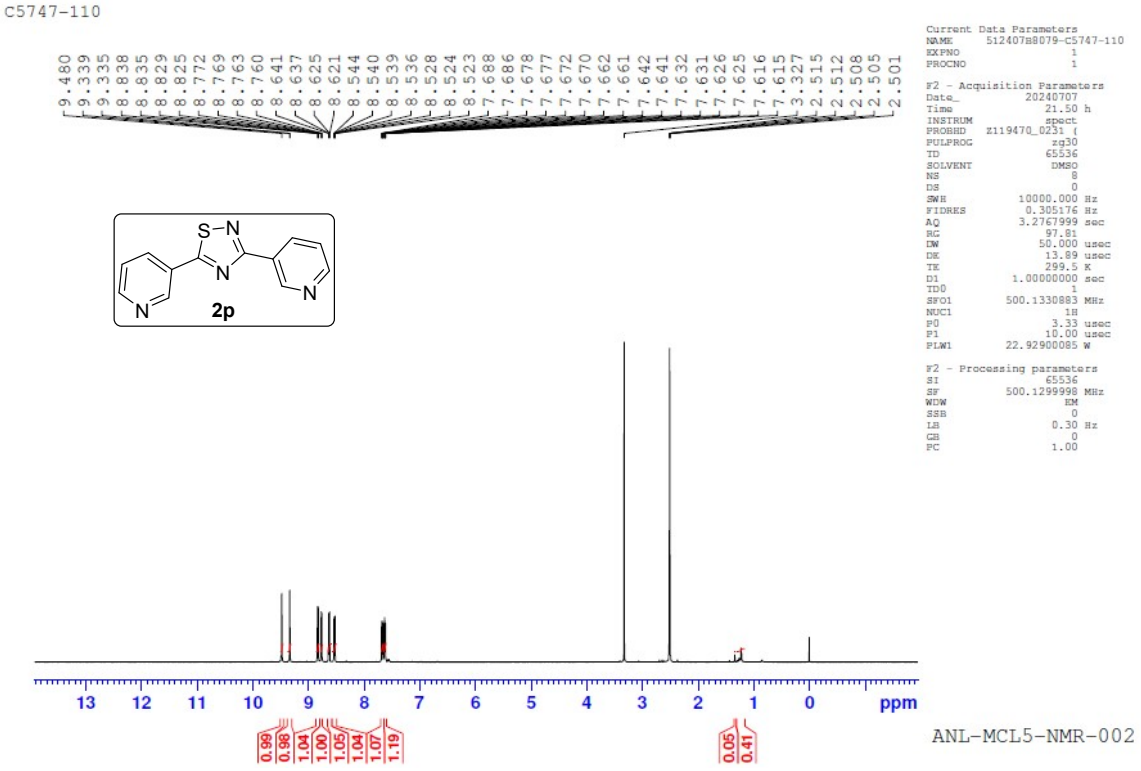



**LCMS spectrum of Compound (2o)**

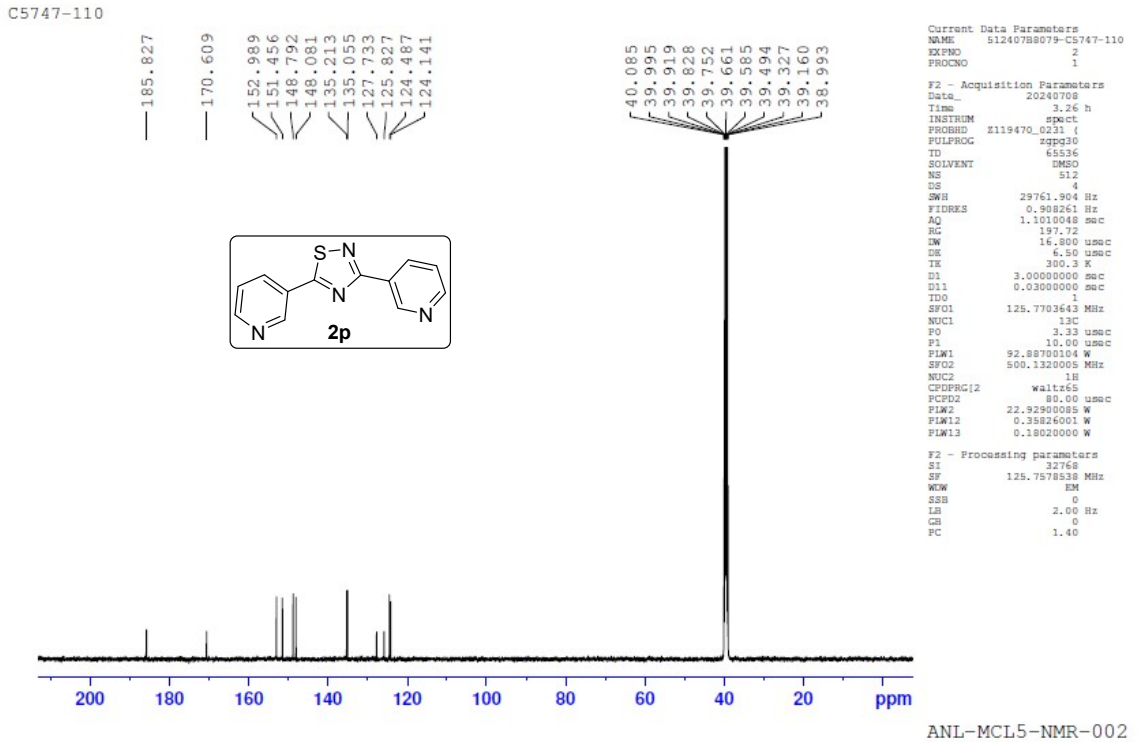


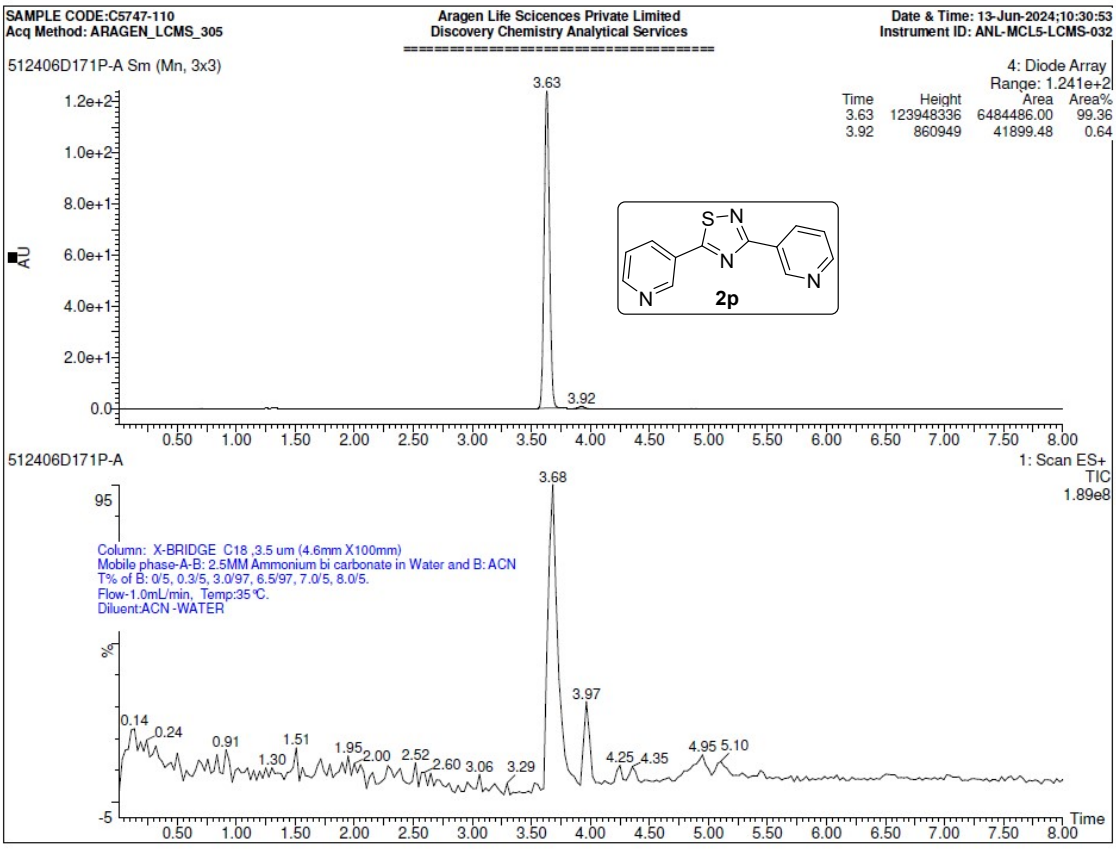

**$^{1}$H NMR spectrum (500 MHz) of Compound (2p) in DMSO-$d_6$**

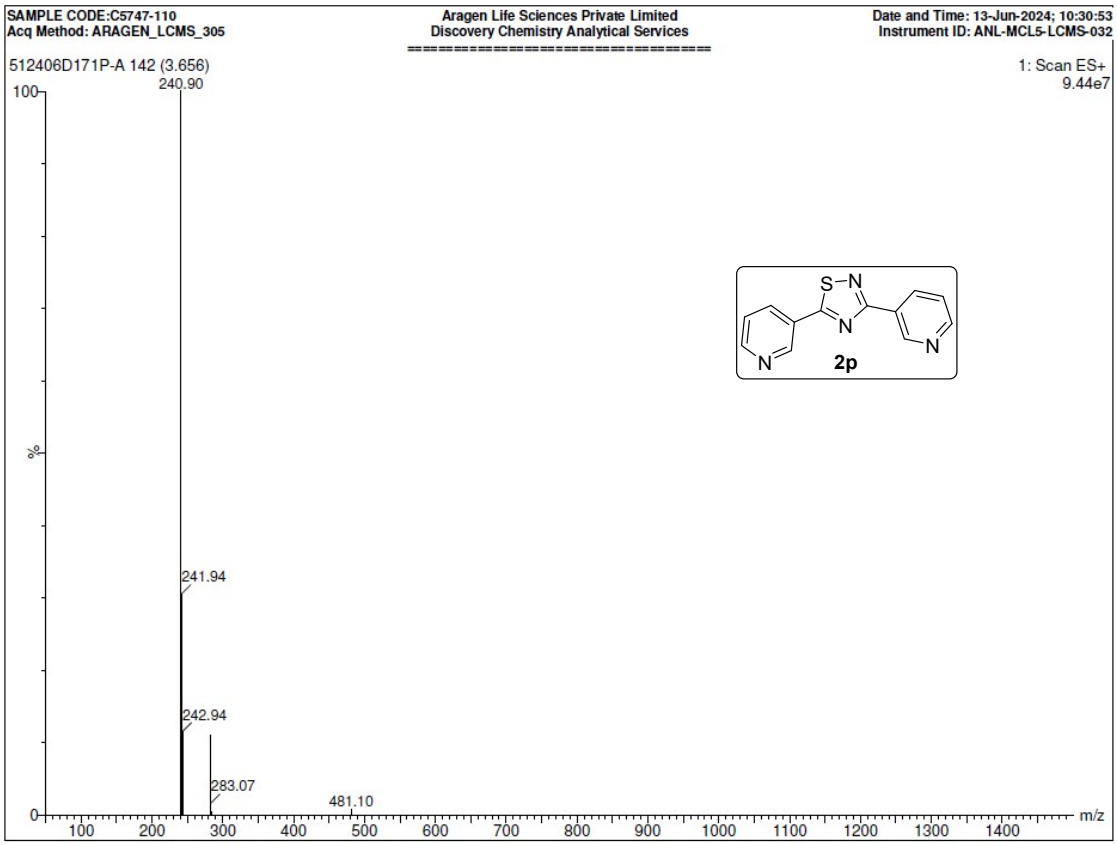



**$^{13}$C NMR spectrum (125 MHz) of Compound (2p) in DMSO-$d_6$**

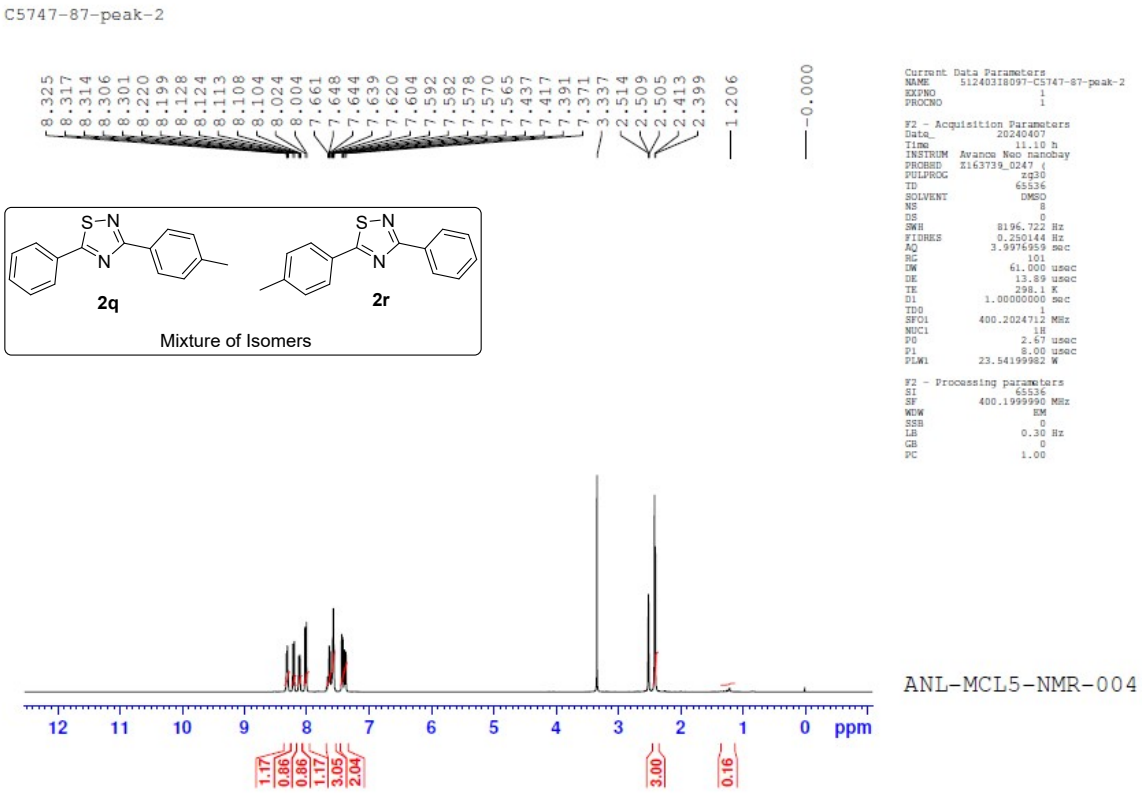



**LCMS spectrum of Compound (2p)**

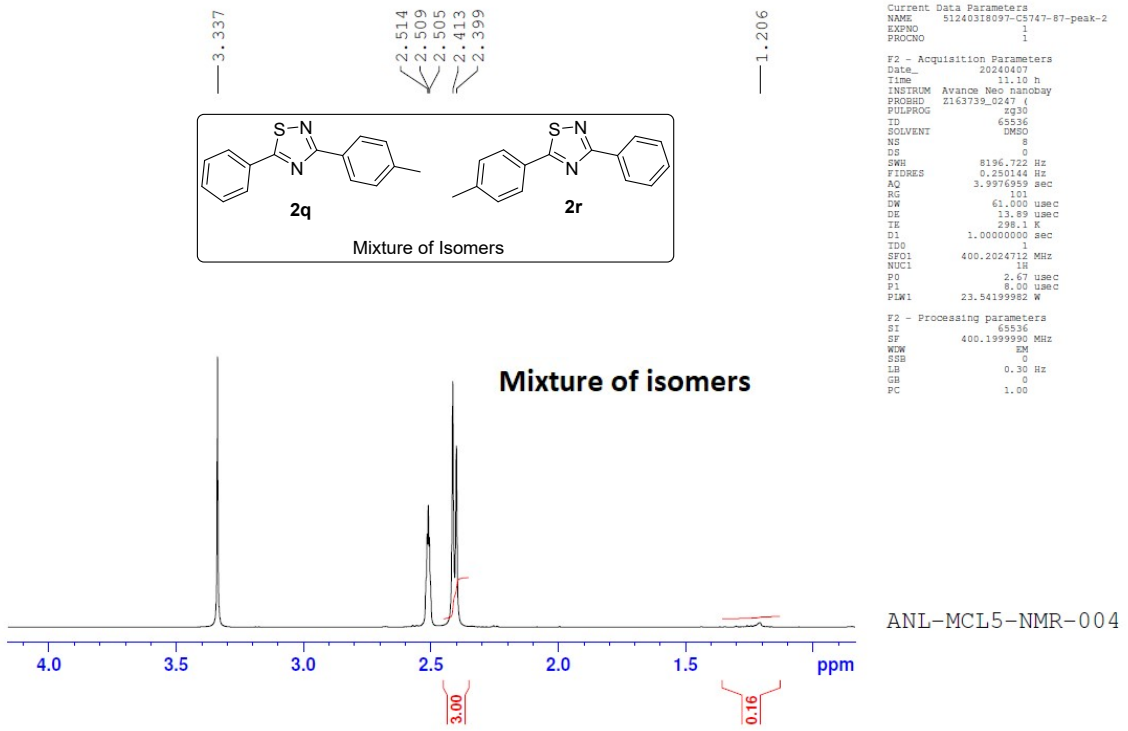

SAMPLE CODE:C5747-110
Acq Method: ARAGEN_LCMS_305
Aragen Life Sciences Private Limited
Discovery Chemistry Analytical Services
Date & Time: 13-Jun-2024;10:30:53
Instrument ID: ANL-MCL5-LCMS-032
512406D171P-A Sm (Mn, 3x3)
4: Diode Array
Range: 1.241e+2
Time Height Area Area%
3.63 123948336 6484486.00 99.36
3.92 860949 41899.48 0.64
3.63
3.92
AU
2p
512406D171P-A
1: Scan ES+
TIC
1.89e8
3.68
Column: X-BRIDGE C18 ,3.5 um (4.6mm X100mm)
Mobile phase-A-B: 2.5MM Ammonium bi carbonate in Water and B: ACN
T% of B: 0/5, 0.3/5, 3.0/97, 6.5/97, 7.0/5, 8.0/5.
Flow-1.0mL/min, Temp:35°C.
Diluent:ACN -WATER
3.97
Time


SAMPLE CODE:C5747-110
Acq Method: ARAGEN_LCMS_305
Aragen Life Sciences Private Limited
Discovery Chemistry Analytical Services
Date and Time: 13-Jun-2024; 10:30:53
Instrument ID: ANL-MCL5-LCMS-032
512406D171P-A 142 (3.656)
1: Scan ES+
9.44e7
240.90
241.94
242.94
283.07
481.10
2p
m/z

## $^1$H NMR spectrum (400 MHz) of Compound (2q+2r) in DMSO-$d_6$



C5747-87-peak-2





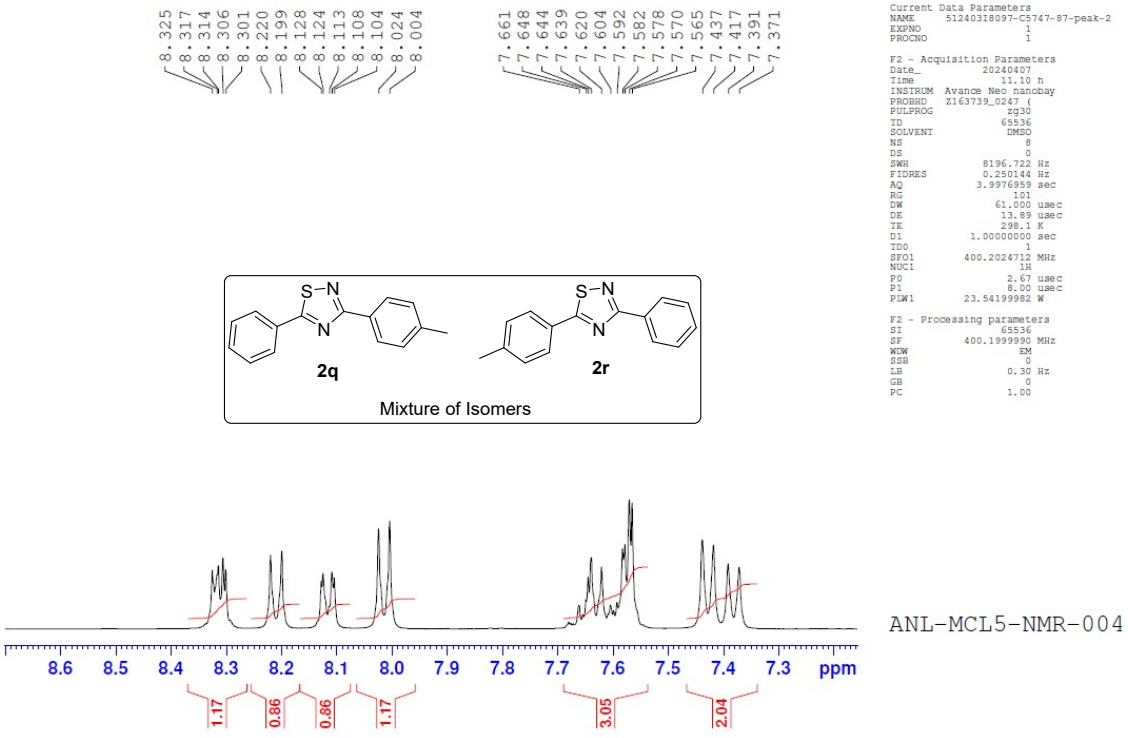
Current Data Parameters
NAME 512403I8097-C5747-87-peak-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240407
Time 11.10 h
INSTRUM Avance Neo nanobay
PROBHD Z163739_0247 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 8196.722 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.89 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.2024712 MHz
NUC1 1H
P0 2.67 usec
P1 8.00 usec
PLW1 23.54199982 W

F2 - Processing parameters
SI 65536
SF 400.1999990 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


ANL-MCL5-NMR-004

C5747-87-peak-2





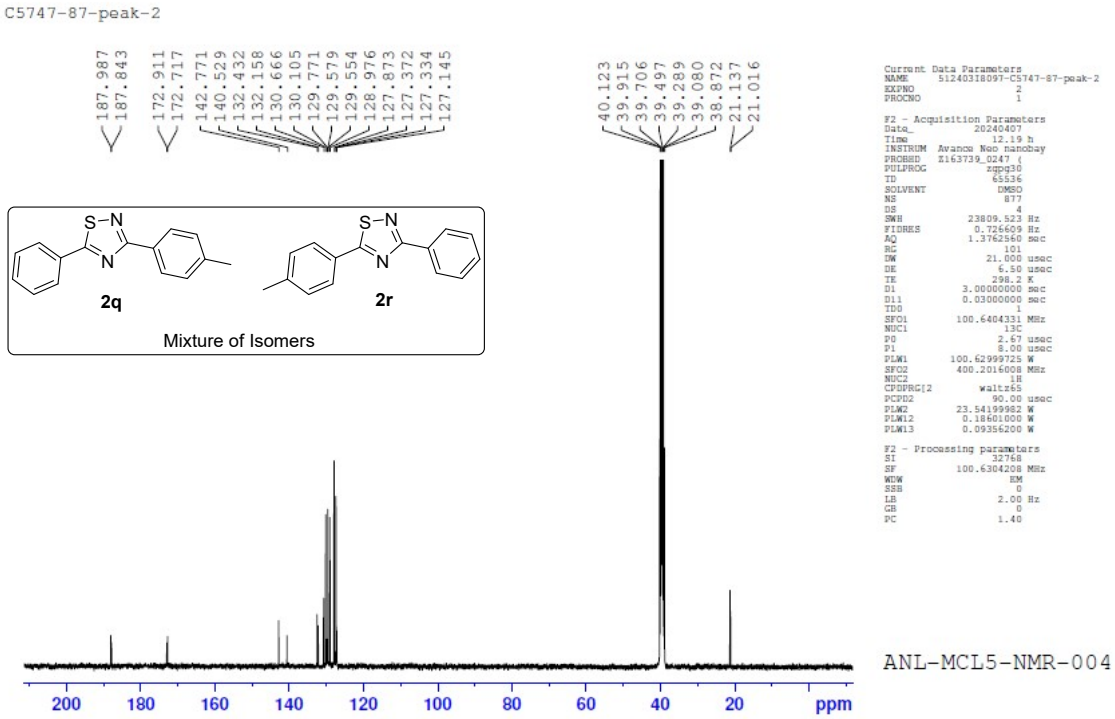
Current Data Parameters
NAME 512403I8097-C5747-87-peak-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240407
Time 11.10 h
INSTRUM Avance Neo nanobay
PROBHD Z163739_0247 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 8196.722 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.89 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.2024712 MHz
NUC1 1H
P0 2.67 usec
P1 8.00 usec
PLW1 23.54199982 W

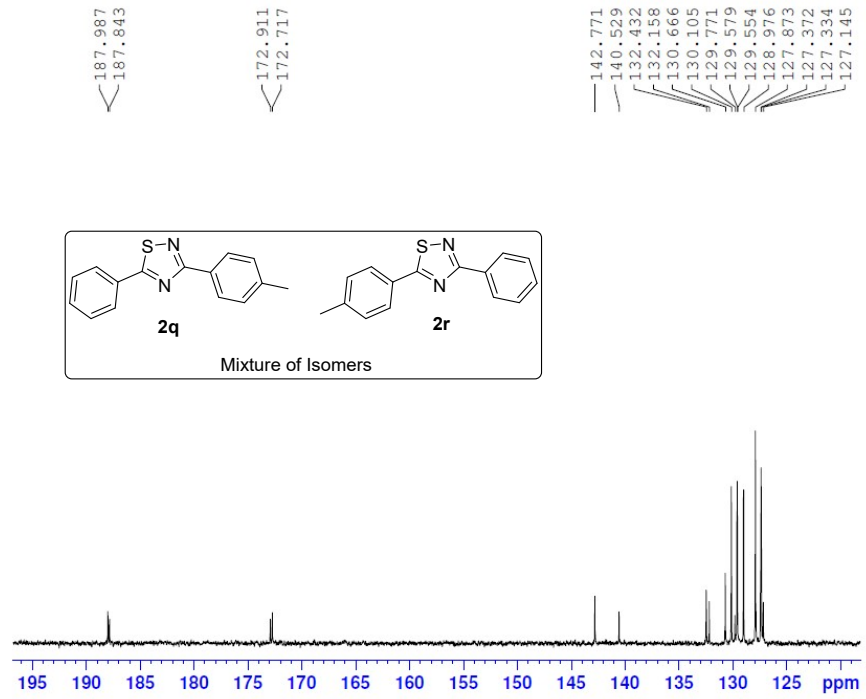
F2 - Processing parameters
SI 65536
SF 400.1999990 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


ANL-MCL5-NMR-004

## $^{13}$C NMR spectrum (100 MHz) of Compound (2q+2r) in DMSO-$d_6$



C5747-87-peak-2



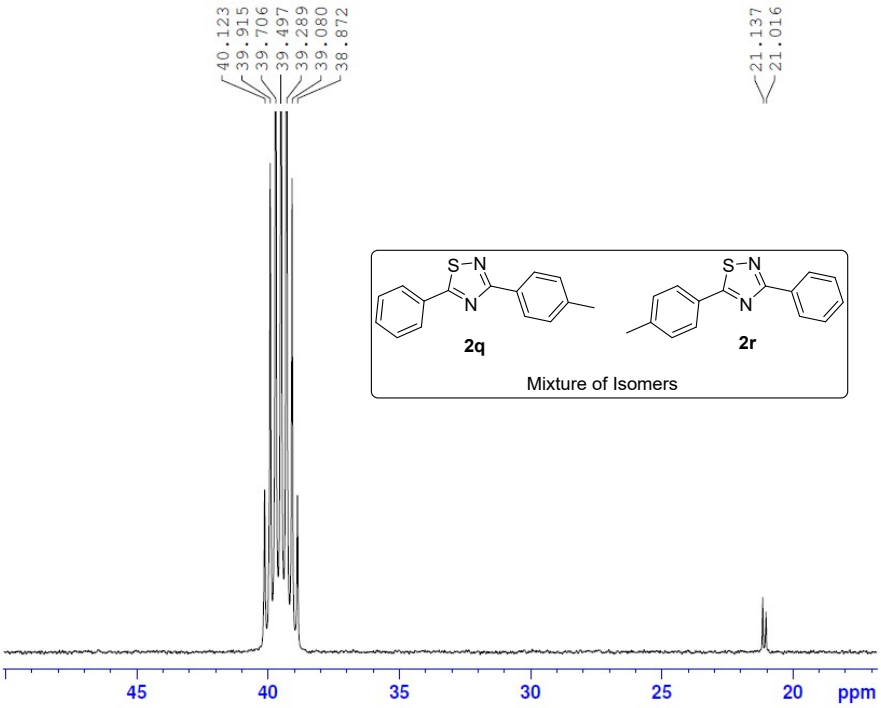

```
Current Data Parameters
NAME      51240318097-C5747-87-peak-2
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20240407
Time              12.19 h
INSTRUM  Avance Neo nanobay
PROBHD   Z163739_0247 (
PULPROG           zgpg30
TD                65536
SOLVENT            DMSO
NS                  877
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                  101
DW               21.000 usec
DE                6.50 usec
TE                298.2 K
D1           3.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6404331 MHz
NUC1                13C
P0                 2.67 usec
P1                 8.00 usec
PLW1       100.62999725 W
SFO2       400.2016008 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2        23.54199982 W
PLW12        0.18601000 W
PLW13        0.09356200 W

F2 - Processing parameters
SI                32768
SF          100.6304208 MHz
WDW                 EM
SSB                  0
LB                2.00 Hz
GB                   0
PC                1.40
```

ANL-MCL5-NMR-004

C5747-87-peak-2



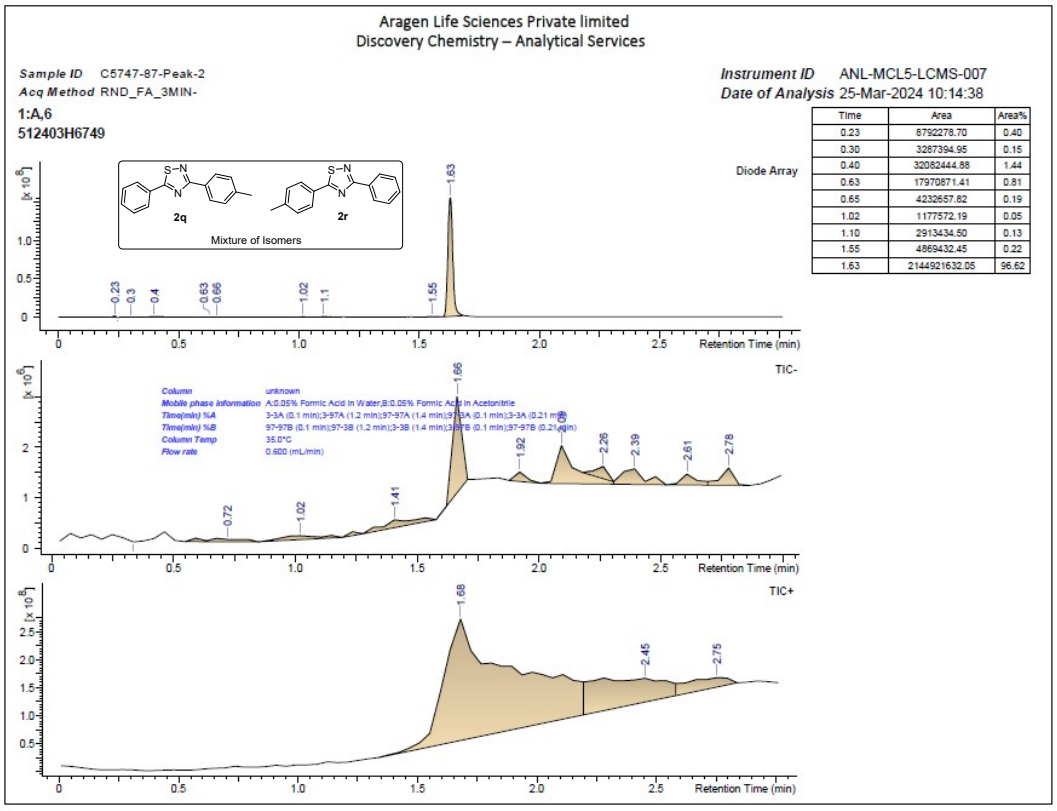

```
Current Data Parameters
NAME      51240318097-C5747-87-peak-2
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20240407
Time              12.19 h
INSTRUM  Avance Neo nanobay
PROBHD   Z163739_0247 (
PULPROG           zgpg30
TD                65536
SOLVENT            DMSO
NS                  877
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                  101
DW               21.000 usec
DE                6.50 usec
TE                298.2 K
D1           3.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6404331 MHz
NUC1                13C
P0                 2.67 usec
P1                 8.00 usec
PLW1       100.62999725 W
SFO2       400.2016008 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2        23.54199982 W
PLW12        0.18601000 W
PLW13        0.09356200 W

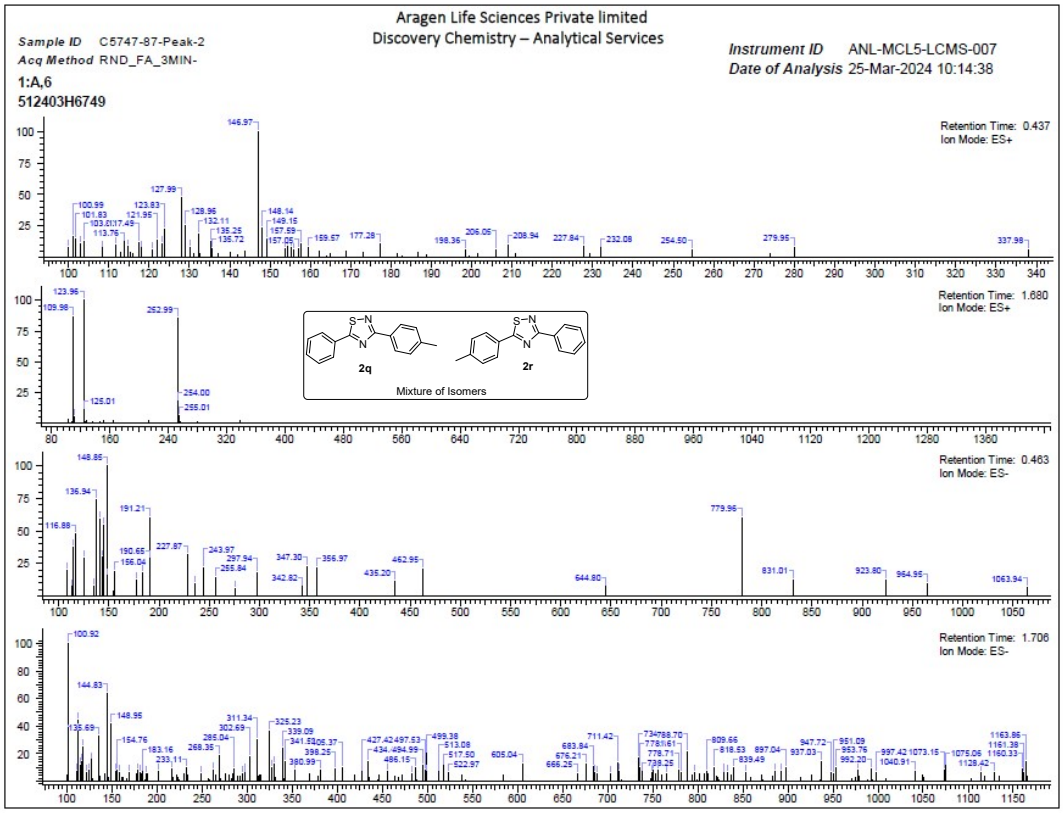
F2 - Processing parameters
SI                32768
SF          100.6304208 MHz
WDW                 EM
SSB                  0
LB                2.00 Hz
GB                   0
PC                1.40
```

ANL-MCL5-NMR-004

## LCMS spectrum of Compound (2q+2r)




| Time | Area | Area% |
|---|---|---|
| 0.23 | 8792278.70 | 0.40 |
| 0.30 | 3287394.95 | 0.15 |
| 0.40 | 32082444.88 | 1.44 |
| 0.63 | 17970871.41 | 0.81 |
| 0.65 | 4232657.82 | 0.19 |
| 1.02 | 1177572.19 | 0.05 |
| 1.10 | 2913434.50 | 0.13 |
| 1.55 | 4869432.45 | 0.22 |
| 1.63 | 2144921632.05 | 96.62 |



**Chiral HPLC spectrum of Compound (2q+2r)**

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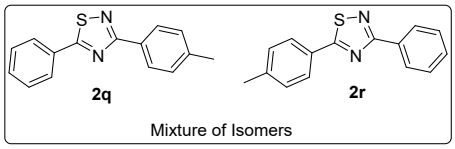
| | | |
|---|---|---|
| Sample Name | : | ARG-FMC-1008-c5747-87-Peak-2 |
| Date | : | 20.08.2025 18:58 |
| Method | : | 3g_40%_A1- 10 Mins |
| Injection Volume (µl) | : | 5 |
| Vial Position | : | F3 |
| Column Name | : | CHIRALCEL OJ-H (4.6*250MM) 5µ |
| Co-Solvent | : | 0.5% DEA IN METHANOL |
| Co-Solvent % | : | 40 |
| Temperature (°C) | : | 30 |
| Flow (mL/min) | : | 3 |
| Back Pressure (bar) | : | 100 |
| Detector (nm) | : | 254 |
| Instrument Name | : | ANL-MCL5-SFC-026 |

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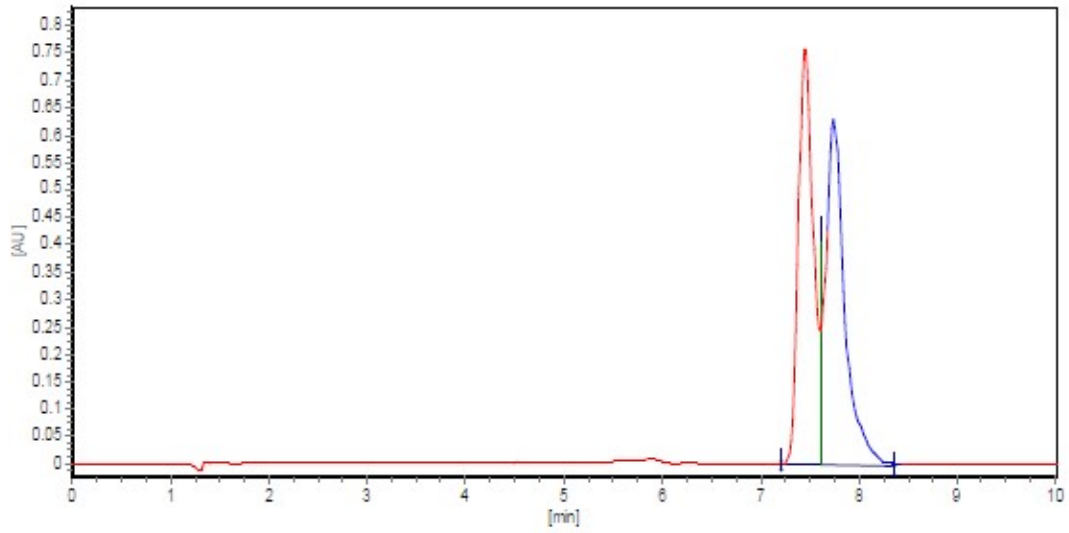
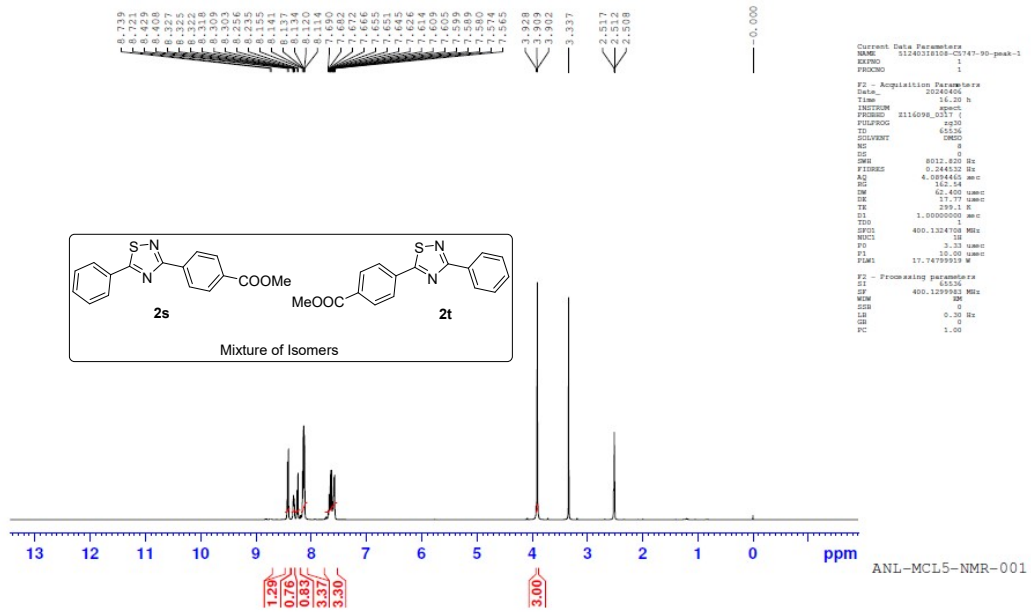


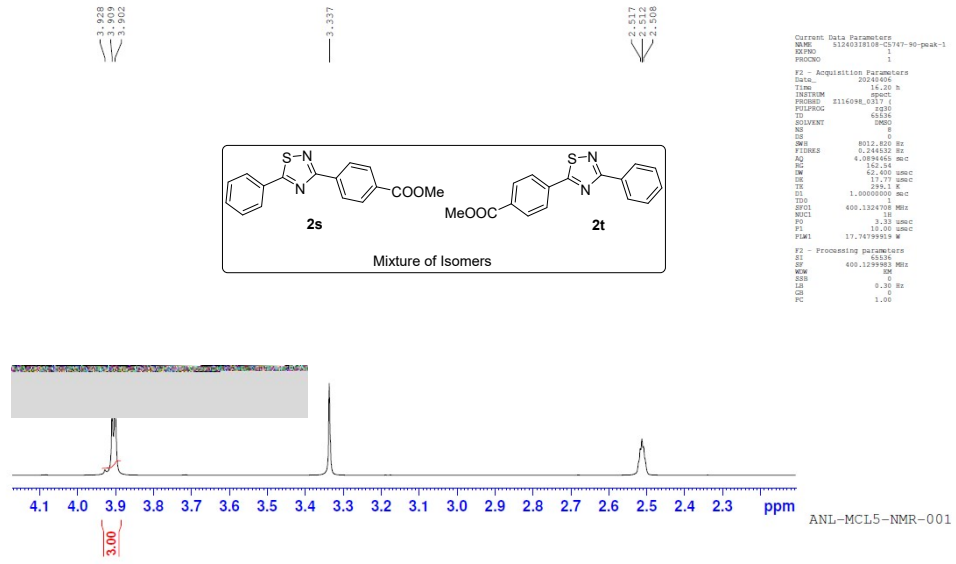

| Peak No. | RT | Area | % Area |
|---|---|---|---|
| 1 | 7.4 | 9496467 | 56.08 |
| 2 | 7.68 | 7437184 | 43.92 |

## $^{1}$H NMR spectrum (400 MHz) of Compound (2s+2t) in DMSO-$d_6$

C5747-90-peak-1

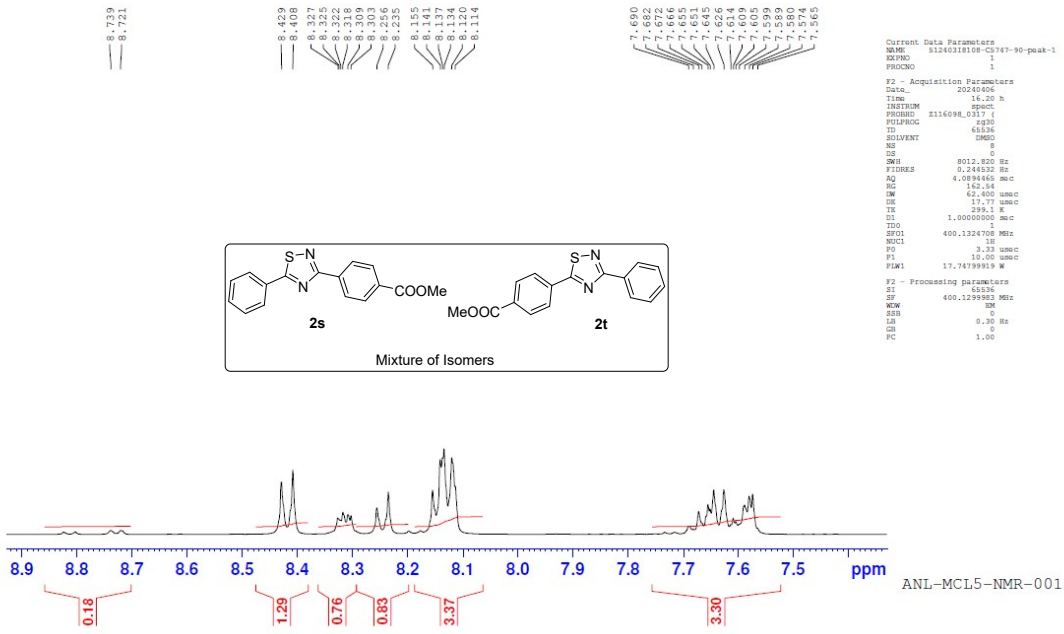


C5747-90-peak-1



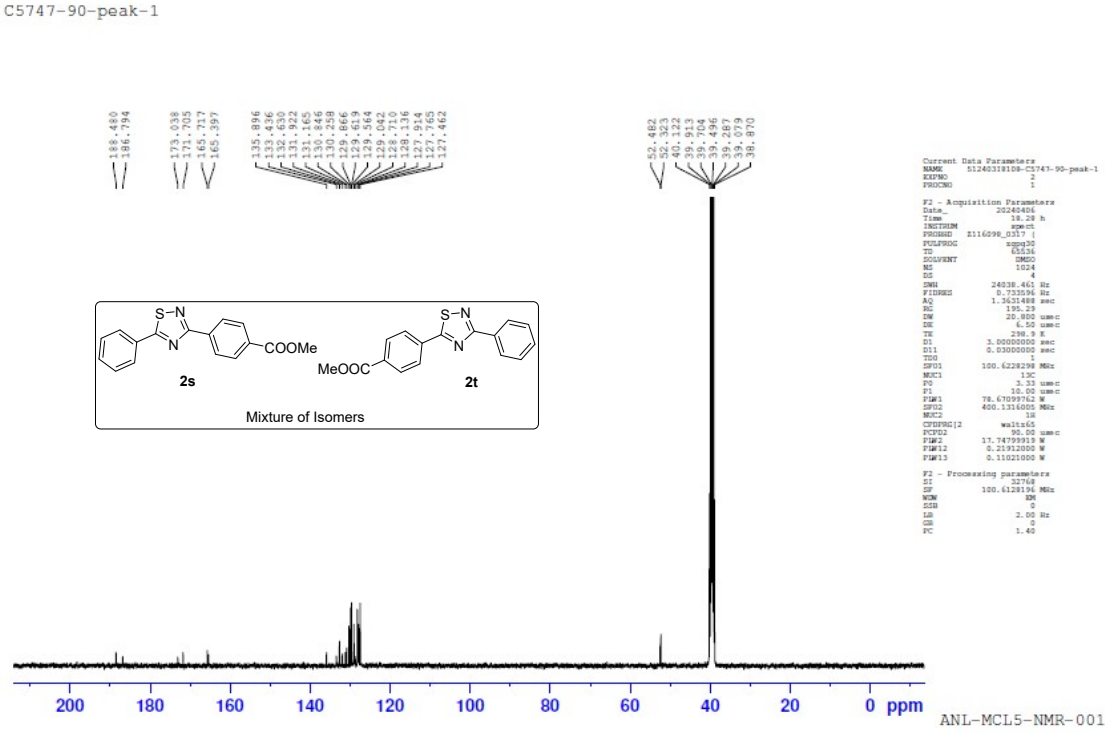

ANL-MCL5-NMR-001

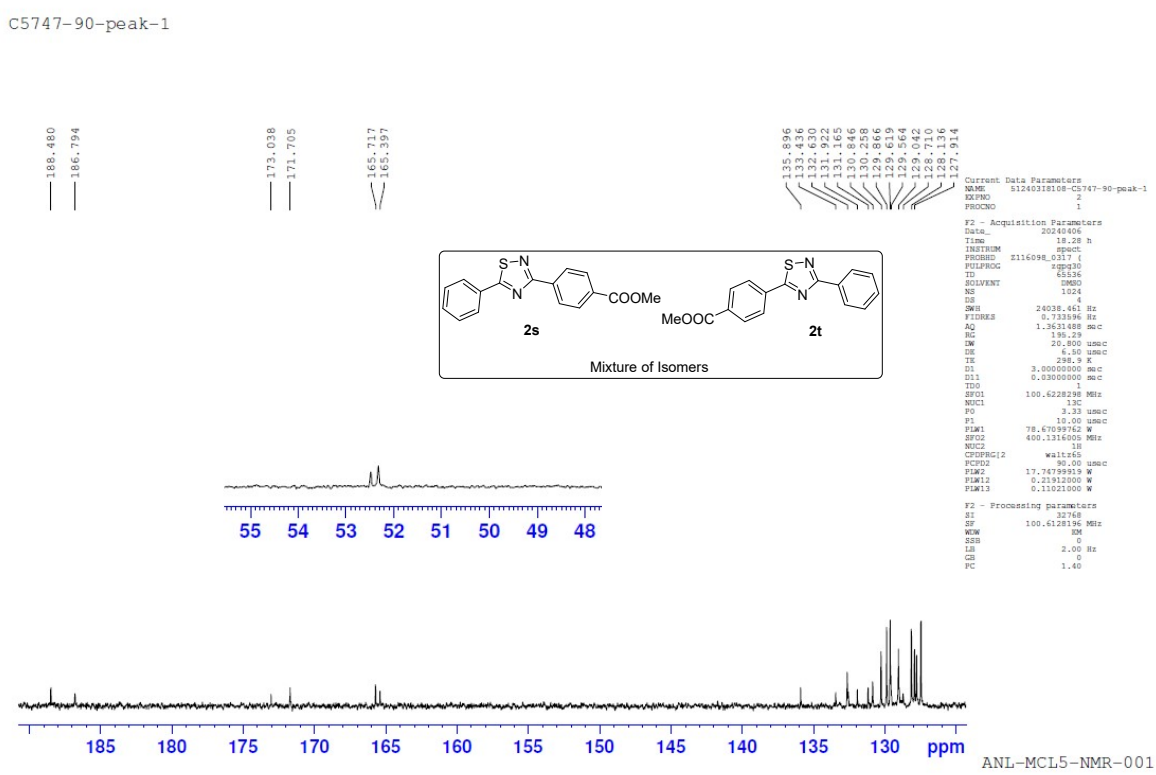
C5747-90-peak-1

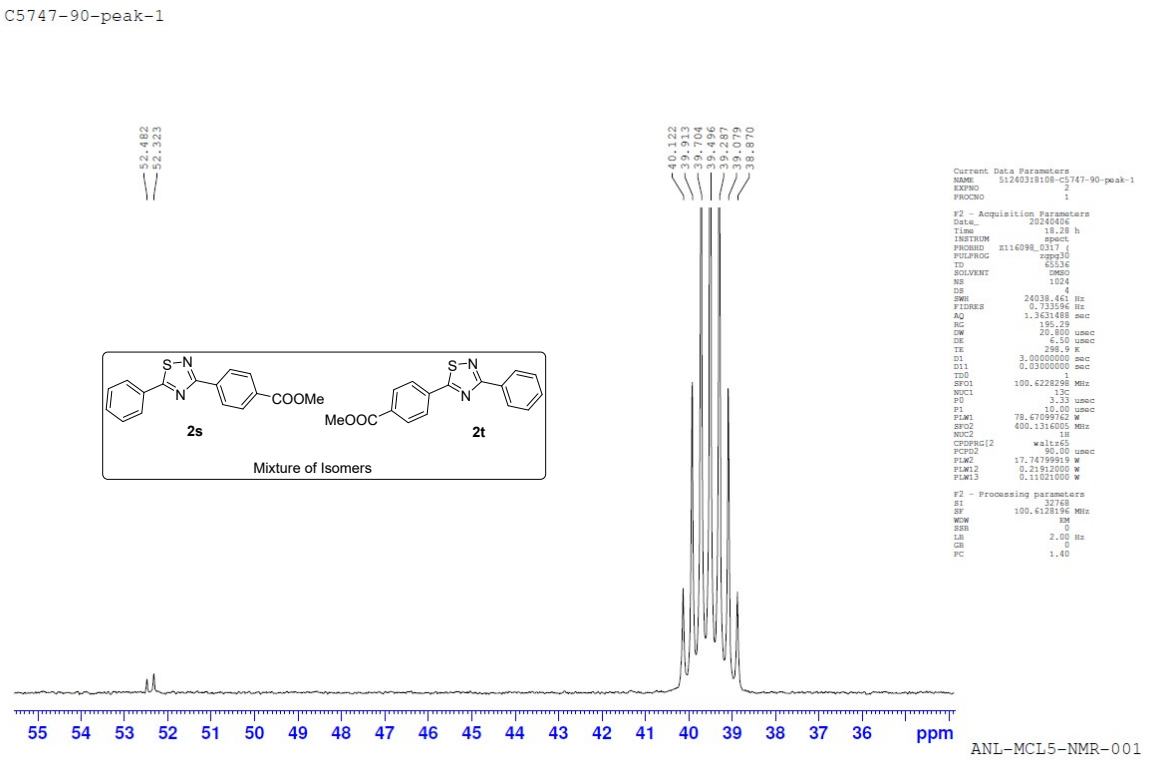



ANL-MCL5-NMR-001

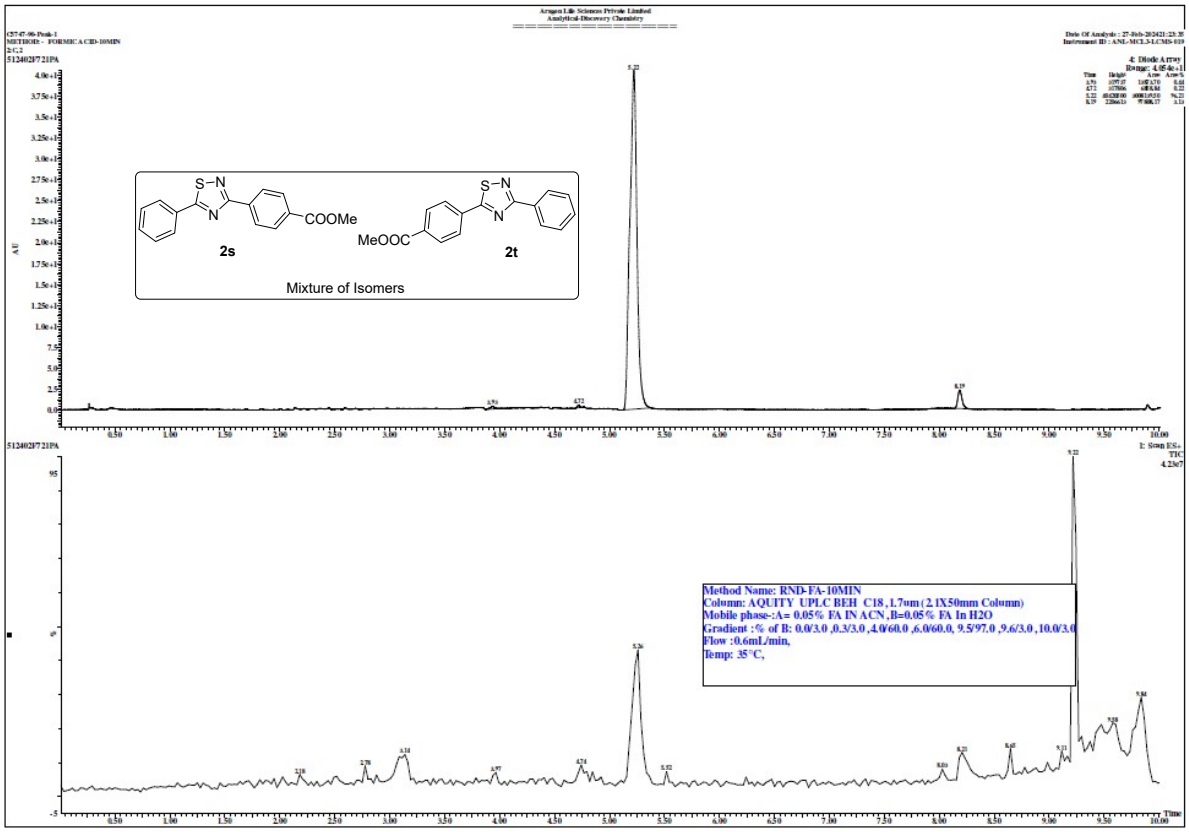
## $^{13}C$ NMR spectrum (100 MHz) of Compound (2s+2t) in DMSO-$d_6$

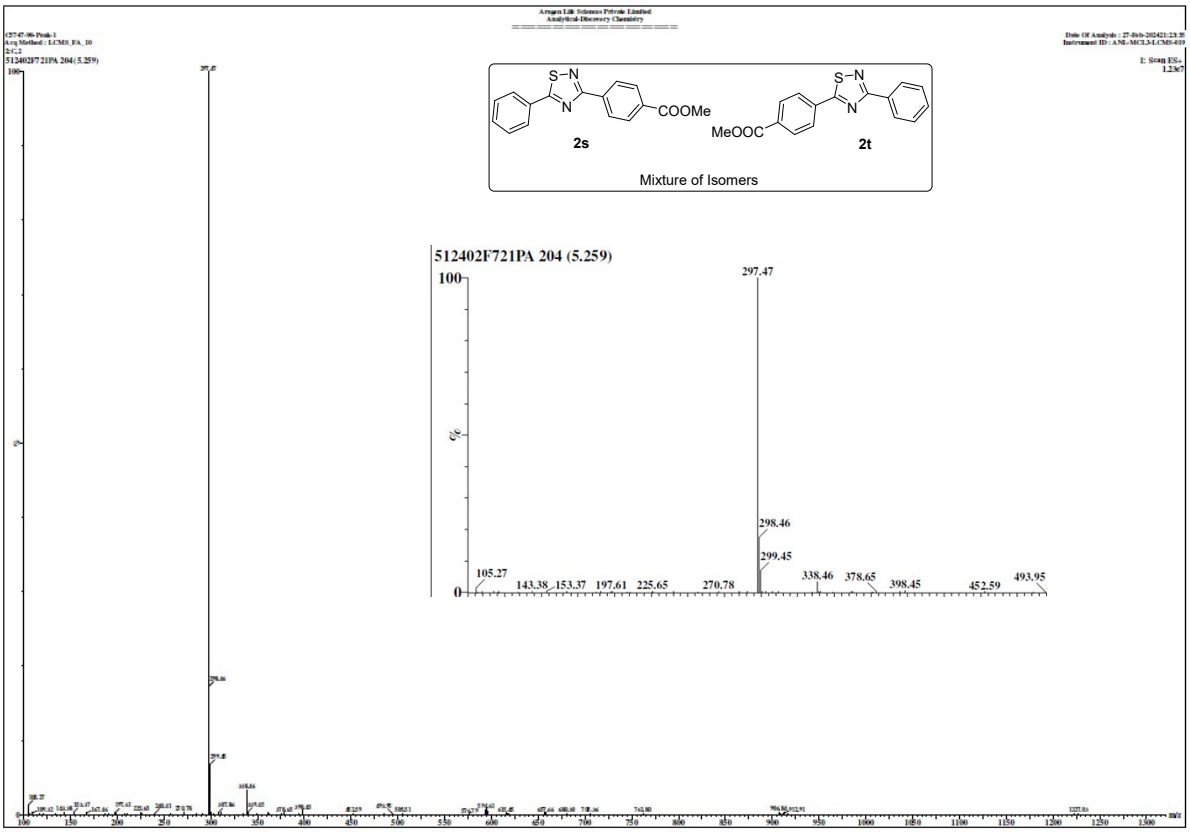





**LCMS spectrum of Compound (2s+2t)**




**Chiral HPLC spectrum of Compound (2s+2t)**

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SFC Report

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| | | |
|---|---|---|
| Sample Name | : | ARG-FMC-1008-C5747-90-Peak-1 |
| Date | : | 20.08.2025 18:58 |
| Method | : | 3g_40%_A1- 10 Mins |
| Injection Volume (µl) | : | 5 |
| Vial Position | : | F4 |
| Column Name | : | (R,R) WHELK-01 (4.6*250MM) 5µ |
| Co-Solvent | : | 0.5% DEA IN METHANOL |
| Co-Solvent % | : | 40 |
| Temperature (°C) | : | 30 |
| Flow (mL/min) | : | 3 |
| Back Pressure (bar) | : | 100 |
| Detector (nm) | : | 254 |
| Instrument Name | : | ANL-MCL5-SFC-026 |

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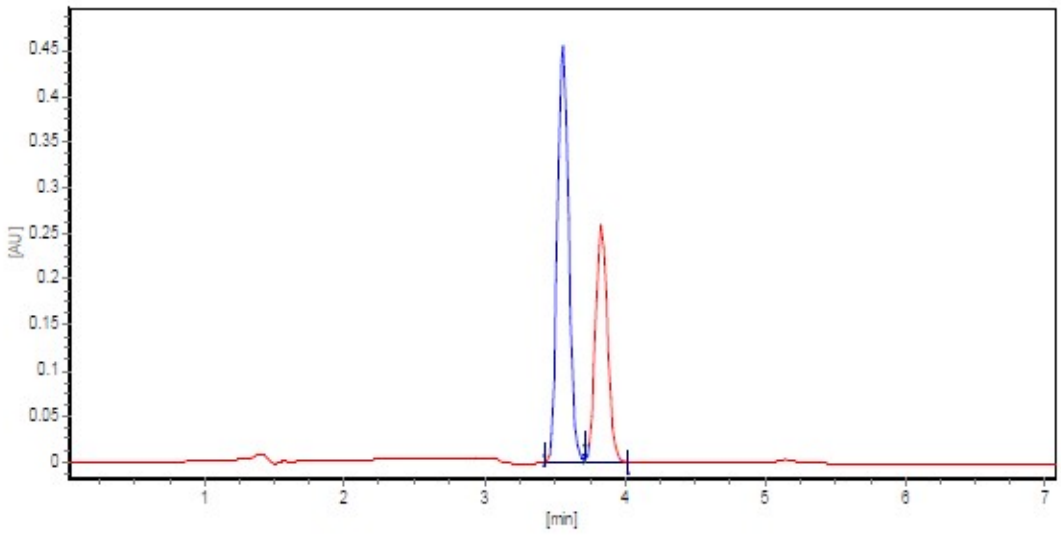


| Peak No. | RT | Area | % Area |
|---|---|---|---|
| 1 | 3.53 | 2547296 | 62.68 |
| 2 | 3.8 | 1516964 | 37.32 |