

Supporting Information

Unexpected Synthesis of 4,4'-Bis(1,2,3-triazole)s from Calcium Carbide as an Acetylene Source via the Click Reaction

Rugang Fu^{*a}, Cheng Cao^a, Junke Wang^a, Fei Wen^b, Dong Qiu^a, Yingxiao Zong^a

^aCollege of Chemistry and Chemical Engineering, Key Laboratory of Hexi Corridor Resources Utilization of Gansu, Hexi University, Zhangye, Gansu 734000, P. R. China.

^bYellow River Basin Ecotope Integration of Industry and Education Research Institute, Lanzhou Resources & Environment Voc-Tech University, Lanzhou 730022, P. R. China.

E-mail: 382827314@qq.com

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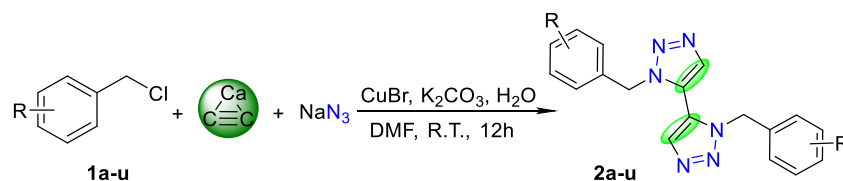
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1. General Information

^1H NMR and ^{13}C NMR spectra were recorded on Bruker-400 MB instrument using CDCl_3 as solvent and Me_4Si as internal standard. High-resolution mass spectra (HRMS) (ESI) were obtained with Bruker micrOTOF-QIII, Bruker Daltonics APEX II 47e and Orbitrap Elite mass spectrometer. Calcium carbide was purchased from Tianjin Chemical Company (purity: 72-82%), and ground into powder prior to use. Unless otherwise noted, the solvents and reagents were reagent grade and were used without any further purification. Column chromatographic separations were carried out on a flash chromatographic system using silica gel, and petroleum ether (60–90 °C) and ethyl acetate as eluent. For thin layer chromatography (TLC), silica gel plates precoated with GF-254 were used.

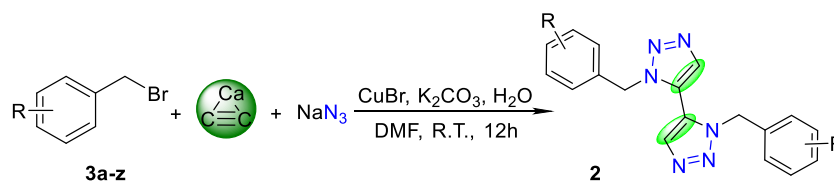
2. General procedure for the synthesis of 4,4'-Bis(1,2,3-triazole)s

2.1 The general procedure for the preparation of 4,4'-Bis(1,2,3-triazole)s from benzyl chlorides



Benzyl chlorides (1.0 mmol), calcium carbide (4.8 mmol, 0.3 g for 72-82% purity), sodium azide (3.0 mmol), cuprous bromide (0.1 mmol), potassium carbonate (2.0 mmol) and H_2O (6 mmol) in DMF (4 mL) and stirred at room temperature for 12 h. The reaction was monitored by TLC. After the reaction was complete, the resulting mixture was filtered to remove the solid, and the liquor was extracted with ethyl acetate (3×10 mL), and washed with saturated brine (3×10 mL). The resulting organic phase was dried with anhydrous sodium sulfate, and concentrated under reduced pressure. The crude product was purified by recrystallization using ethanol and petroleum ether as solvents or by column chromatography using petroleum ether and ethyl acetate as eluents to give the pure product.

2.2 The general procedure for the preparation of 4,4'-Bis(1,2,3-triazole)s from benzyl bromide

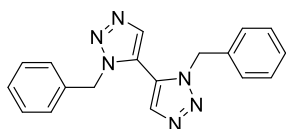


benzyl bromides (1.0 mmol), calcium carbide (4.8 mmol, 0.3 g for 72-82% purity),

sodium azide (3.0 mmol), cuprous bromide (0.1 mmol), potassium carbonate (2.0 mmol) and H₂O (6 mmol) in DMF (4 mL) and stirred at room temperature for 12 h. The reaction was monitored by TLC. After the reaction was complete, the resulting mixture was filtered to remove the solid, and the liquor was extracted with ethyl acetate (3×10 mL), and washed with saturated brine (3×10 mL). The resulting organic phase was dried with anhydrous sodium sulfate, and concentrated under reduced pressure. The crude product is purified by recrystallization purification using ethanol and petroleum ether as solvents or by column chromatography using petroleum ether and ethyl acetate as eluents to obtain a pure product.

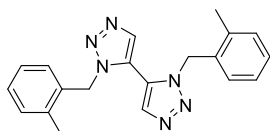
3. Analytical data of the products

1. 3,3'-dibenzyl-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2a)^[1]



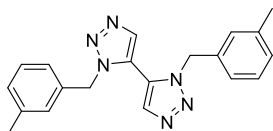
White solid (136 mg, 86% yield), m.p. 173–174 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.49 (s, 2H), 7.34 – 7.26 (m, 6H), 6.97 – 6.89 (m, 4H), 5.09 (s, 4H). ¹³C NMR (101 MHz, CDCl₃): δ 135.91, 134.21, 129.13, 128.84, 127.36, 123.67, 52.36. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₁₈H₁₆N₆Na 339.1329; found 339.1334.

2. 3,3'-bis(2-methylbenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2b)



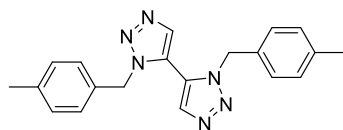
White solid (101 mg, 59% yield). m.p. 137–138 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.42 (s, 2H), 7.24 – 7.03 (m, 6H), 6.51 (d, *J* = 7.7 Hz, 2H), 5.05 (s, 4H), 2.09 (s, 6H). ¹³C NMR (101 MHz, CDCl₃): δ 135.91, 135.88, 132.37, 130.93, 128.89, 127.78, 126.60, 123.83, 50.33, 18.86. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₀H₂₀N₆Na 367.1642; found 367.1645.

2. 3,3'-bis(3-methylbenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2c)



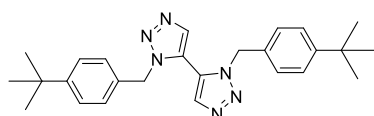
White solid (113 mg, 66% yield). m.p. 146–148 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.49 (s, 2H), 7.16 (t, *J* = 7.6 Hz, 2H), 7.10 (d, *J* = 7.7 Hz, 2H), 6.74 (d, *J* = 1.8 Hz, 2H), 6.72 – 6.67 (m, 2H), 5.05 (s, 4H), 2.27 (s, 6H). ¹³C NMR (101 MHz, CDCl₃): δ 139.02, 135.91, 134.12, 129.5, 128.99, 128.01, 124.37, 123.69, 52.37, 21.25. HRMS (ESI) m/z: [M+Na]⁺ Calcd for C₂₀H₂₀N₆Na 367.1642; found 367.1643.

4. 3,3'-bis(4-methylbenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2d)



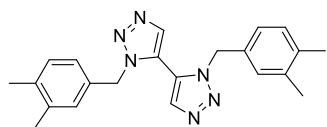
White solid (125 mg, 73% yield). m.p. 183–184 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.49 (s, 2H), 7.07 (d, *J* = 7.8 Hz, 4H), 6.81 (d, *J* = 7.9 Hz, 4H), 5.06 (s, 4H), 2.30 (s, 6H). ¹³C NMR (101 MHz, CDCl₃): δ 138.72, 135.89, 131.20, 129.71, 127.36, 123.65, 52.20, 21.10. HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₂₀H₂₁N₆ 345.1822; found 345.1821.

5. 3,3'-bis(4-(tert-butyl)benzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2e)



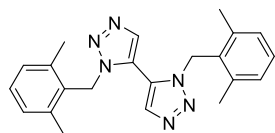
White solid (146 mg, 68% yield). m.p. 162–163 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.49 (s, 2H), 7.32 (d, *J* = 8.4 Hz, 4H), 6.88 (d, *J* = 8.3 Hz, 4H), 5.01 (s, 4H), 1.28 (s, 18H). ¹³C NMR (101 MHz, CDCl₃): δ 152.14, 136.00, 131.25, 127.21, 126.01, 123.66, 52.06, 34.65, 31.22. HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₆H₃₂N₆Na 451.2581; found 451.2597.

6. 3,3'-bis(3,4-dimethylbenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2f)



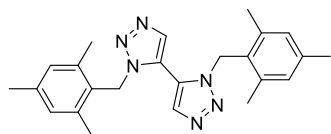
White solid (76 mg, 41% yield). m.p. 146–147 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.50 (s, 2H), 7.03 (d, *J* = 7.7 Hz, 2H), 6.77 – 6.56 (m, 4H), 5.03 (s, 4H), 2.20 (d, *J* = 14.3 Hz, 12H). ¹³C NMR (101 MHz, CDCl₃): δ 137.51, 137.34, 135.91, 131.57, 130.21, 128.60, 124.79, 123.68, 52.22, 19.65, 19.45. HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₂₂H₂₅N₆ 373.2135; found 373.2135.

7. 3,3'-bis(2,6-dimethylbenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2g)



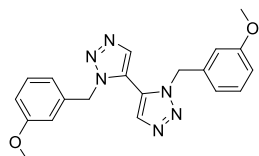
White solid (99 mg, 53% yield). m.p. 173–174 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.65 (s, 2H), 7.16 (t, *J* = 7.6 Hz, 2H), 7.01 (d, *J* = 7.6 Hz, 4H), 5.01 (s, 4H), 2.07 (s, 12H). ¹³C NMR (101 MHz, CDCl₃): δ 138.02, 135.99, 130.03, 129.21, 128.87, 123.61, 47.48, 19.90. HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₂₂H₂₅N₆ 373.2135; found 373.2135.

8. 3,3'-bis(2,4,6-trimethylbenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2h)



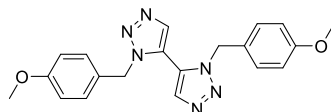
White solid (120 mg, 60% yield). m.p. 60–61 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.68 (s, 2H), 6.83 (s, 4H), 4.95 (s, 4H), 2.25 (s, 6H), 2.03 (s, 12H). ^{13}C NMR (101 MHz, CDCl_3): δ 138.92, 137.83, 135.94, 129.62, 127.06, 123.62, 47.28, 20.95, 19.71. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{24}\text{H}_{29}\text{N}_6$ 401.2448; found 401.2448.

9. 3,3'-bis(3-methoxybenzyl)-3H,3'H-4,4'-bi(1,2,3-triazole) (2i)



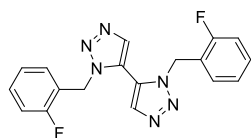
White solid (132 mg, 70% yield). m.p. 150–151 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.52 (s, 2H), 7.24 – 7.16 (m, 2H), 6.86 – 6.82 (m, 2H), 6.52 – 6.46 (m, 4H), 5.10 (s, 4H), 3.73 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3): δ 160.13, 135.90, 135.65, 130.22, 123.72, 119.40, 114.46, 112.74, 55.28, 52.31. HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{20}\text{N}_6\text{O}_2\text{Na}$ 399.1540; found 399.1543.

10. 3,3'-bis(4-methoxybenzyl)-3H,3'H-4,4'-bi(1,2,3-triazole) (2j)



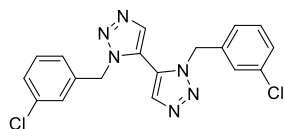
White solid (149 mg, 79% yield). m.p. 153–155 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.52 (s, 2H), 6.92 – 6.70 (m, 8H), 5.04 (s, 4H), 3.76 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3): δ 159.86, 135.93, 128.96, 126.07, 123.55, 114.37, 55.30, 51.99. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{20}\text{H}_{21}\text{N}_6\text{O}_2$ 377.1721; found 377.1720.

11. 3,3'-bis(2-fluorobenzyl)-3H,3'H-4,4'-bi(1,2,3-triazole) (2k)



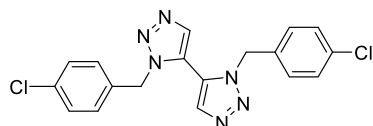
White solid (110 mg, 63% yield). m.p. 144–145 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.61 (s, 2H), 7.33 (tdd, $J = 7.5, 5.3, 1.8$ Hz, 2H), 7.12 – 7.01 (m, 4H), 6.96 (td, $J = 7.6, 1.7$ Hz, 2H), 5.30 (s, 4H). ^{13}C NMR (101 MHz, CDCl_3): δ 161.14, 135.57, 130.95, 130.87, 129.54, 129.52, 124.83, 124.79, 123.69, 121.44, 121.30, 115.98, 115.77, 45.90, 45.85. HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{18}\text{H}_{14}\text{F}_2\text{N}_6\text{Na}$ 375.1140; found 375.1138.

12. 3,3'-bis(3-chlorobenzyl)-3H,3'H-4,4'-bi(1,2,3-triazole) (2l)



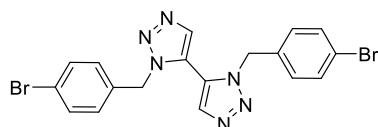
White solid (157 mg, 82% yield). m.p. 200–201 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.59 (s, 2H), 7.30 (ddd, *J* = 8.1, 2.1, 1.2 Hz, 2H), 7.23 (t, *J* = 7.8 Hz, 2H), 6.99 (t, *J* = 1.9 Hz, 2H), 6.81 (ddd, *J* = 7.6, 1.8, 1.0 Hz, 2H), 5.18 (s, 4H). ¹³C NMR (101 MHz, CDCl₃): δ 135.91, 135.75, 135.26, 130.46, 129.22, 127.38, 125.35, 123.61, 51.66. HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₈H₁₄Cl₂N₆Na 407.0549; found 407.0545.

13. 3,3'-bis(4-chlorobenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2m)



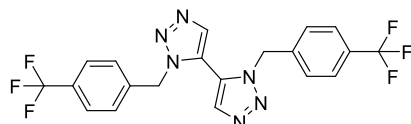
White solid (167 mg, 87% yield). m.p. 193–195 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.59 (s, 2H), 7.26 (d, *J* = 8.5 Hz, 4H), 6.88 (d, *J* = 8.4 Hz, 4H), 5.16 (s, 4H). ¹³C NMR (101 MHz, CDCl₃): δ 135.73, 135.07, 132.35, 129.39, 128.77, 123.55, 51.69. HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₁₈H₁₅Cl₂N₆ 385.0730; found 385.0730.

14. 3,3'-bis(4-bromobenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2n)



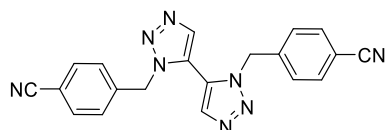
White solid (218 mg, 92% yield). m.p. 91–92 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, DMSO-*d*₆): δ 8.21 (s, 2H), 7.44 – 7.36 (m, 4H), 6.83 – 6.76 (m, 4H), 5.58 (s, 4H). ¹³C NMR (101 MHz, DMSO-*d*₆): δ 135.61, 134.64, 132.04, 129.82, 124.39, 121.80, 51.33. HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₁₈H₁₅Br₂N₆ 472.9719; found 472.9720.

15. 3,3'-bis(4-(trifluoromethyl)benzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2o)



White solid (74 mg, 33% yield). m.p. 161–162 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.60 (s, 2H), 7.56 (d, *J* = 8.1 Hz, 4H), 7.08 (d, *J* = 7.9 Hz, 4H), 5.28 (s, 4H). ¹³C NMR (101 MHz, CDCl₃): δ 137.81, 135.60, 131.48, 131.16, 127.63, 126.20, 124.89, 123.67, 122.19, 51.68. HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₂₀H₁₅F₆N₆ 453.1257; found 453.1257.

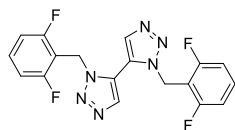
16. 4,4'-(3*H*,3'*H*-[4,4'-bi(1,2,3-triazole)]-3,3'-diylbis(methylene))dibenzonitrile (2p)



White solid (163 mg, 89% yield). m.p. 241–242 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, DMSO-*d*₆): δ 8.16 (s, 2H), 7.72 – 7.67 (m, 4H), 7.07 – 7.03 (m, 4H), 5.70 (s, 4H). ¹³C NMR (101 MHz, DMSO-*d*₆): δ 140.76,

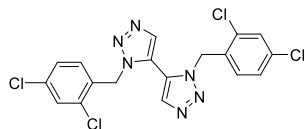
135.72, 133.07, 128.47, 124.46, 118.78, 111.37, 51.41. HRMS (ESI) m/z : $[M+Na]^+$ Calcd for $C_{20}H_{14}N_8Na$ 389.1234; found 389.1237.

17. 3,3'-bis(2,6-difluorobenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2q)



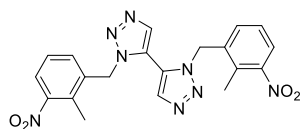
White solid (102 mg, 53% yield). m.p. 170–171 °C. Petroleum ether/EtOH as the recrystallization solvent. 1H NMR (400 MHz, $CDCl_3$): δ 7.75 (s, 2H), 7.34 (tt, J = 8.4, 6.4 Hz, 2H), 6.90 (t, J = 8.0 Hz, 4H), 5.34 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 162.55, 162.48, 160.05, 159.98, 135.53, 131.63, 131.53, 131.42, 123.66, 111.85, 111.80, 111.67, 111.63, 111.61, 110.20, 110.02, 109.84, 40.26, 40.22. HRMS (ESI) m/z : $[M+H]^+$ Calcd for $C_{18}H_{13}F_6N_6$ 389.1132; found 389.1132.

18. 3,3'-bis(2,4-dichlorobenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2r)



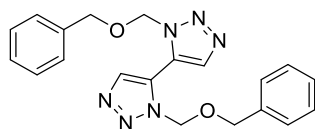
White solid (144 mg, 64% yield). m.p. 141–142 °C. Petroleum ether/EtOH as the recrystallization solvent. 1H NMR (400 MHz, $CDCl_3$): δ 7.63 (s, 2H), 7.40 (d, J = 2.1 Hz, 2H), 7.18 (dd, J = 8.4, 2.1 Hz, 2H), 6.77 (d, J = 8.3 Hz, 2H), 5.41 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 135.68, 135.38, 133.38, 130.35, 129.83, 129.72, 127.93, 123.82, 49.01. HRMS (ESI) m/z : $[M+H]^+$ Calcd for $C_{18}H_{13}Cl_4N_6$ 452.9950; found 452.9950.

19. 3,3'-bis(2-methyl-3-nitrobenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2s)



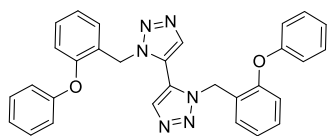
White solid (149 mg, 69% yield). m.p. 175–178 °C. Petroleum ether/EtOH as the recrystallization solvent. 1H NMR (400 MHz, $CDCl_3$): δ 7.73 (dd, J = 8.2, 1.2 Hz, 2H), 7.62 (s, 2H), 7.23 (t, J = 8.0 Hz, 2H), 6.73 (d, J = 7.7 Hz, 2H), 5.30 (s, 4H), 2.29 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.67, 135.63, 134.81, 131.52, 130.30, 127.22, 124.56, 123.80, 50.26, 14.52. HRMS (ESI) m/z : $[M+H]^+$ Calcd for $C_{20}H_{19}O_4N_8$ 435.1524; found 435.1524.

20. 3,3'-bis((benzyloxy)methyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2t)



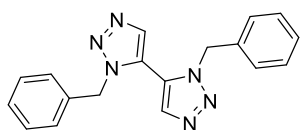
White solid (61 mg, 32% yield). m.p. 97–98 °C. Petroleum ether/EtOH as the recrystallization solvent. 1H NMR (400 MHz, $CDCl_3$): δ 8.26 (s, 2H), 7.38 – 7.30 (m, 10H), 5.73 (s, 4H), 4.64 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 135.64, 128.68, 128.45, 128.26, 128.23, 124.70, 76.04, 71.22. HRMS (ESI) m/z : $[M+H]^+$ Calcd for $C_{20}H_{21}O_2N_6$ 377.1721; found 377.1719.

21. 3,3'-bis(2-phenoxybenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2u)



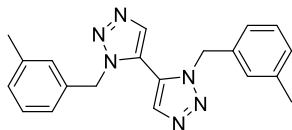
White solid (61 mg, 24% yield). m.p. 81–82 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.58 (s, 2H), 7.34 – 7.28 (m, 4H), 7.21 (ddd, *J* = 8.3, 7.3, 1.8 Hz, 2H), 7.13 – 7.08 (m, 2H), 6.97 (td, *J* = 7.5, 1.2 Hz, 2H), 6.89 (dd, *J* = 7.7, 1.7 Hz, 2H), 6.84 – 6.78 (m, 4H), 6.75 (dd, *J* = 8.2, 1.1 Hz, 2H), 5.32 (s, 4H). ¹³C NMR (101 MHz, CDCl₃): δ 155.95, 154.35, 135.43, 130.11, 130.03, 129.03, 125.09, 124.10, 124.02, 123.62, 118.94, 117.88, 47.02. HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₃₀H₂₄O₂N₆Na 523.1853; found 523.1859.

22. 3,3'-dibenzyl-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2a)



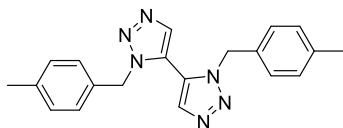
White solid (142 mg, 90% yield). m.p. 173–174 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.48 (s, 2H), 7.27 (d, *J* = 6.6 Hz, 6H), 6.91 (dd, *J* = 7.2, 2.2 Hz, 4H), 5.08 (s, 4H). ¹³C NMR (101 MHz, CDCl₃): δ 135.91, 134.21, 129.12, 128.82, 127.35, 123.67, 52.35.

23. 3,3'-bis(3-methylbenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2c)



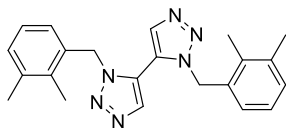
White solid (110 mg, 64% yield). m.p. 146–148 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.49 (s, 2H), 7.19 – 7.08 (m, 4H), 6.77 – 6.66 (m, 4H), 5.05 (s, 4H), 2.27 (s, 6H). ¹³C NMR (101 MHz, CDCl₃): δ 139.02, 135.91, 134.11, 129.55, 128.99, 128.01, 124.37, 123.70, 52.38, 21.25.

24. 3,3'-bis(4-methylbenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2d)



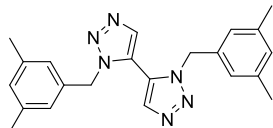
White solid (126 mg, 73% yield). m.p. 183–184 °C. Petroleum ether/EtOH as the recrystallization solvent. ¹H NMR (400 MHz, CDCl₃): δ 7.50 (s, 2H), 7.21 – 7.10 (m, 4H), 6.78 – 6.68 (m, 4H), 5.06 (s, 4H), 2.28 (s, 6H). ¹³C NMR (101 MHz, CDCl₃): δ 139.03, 134.13, 129.55, 129.00, 128.02, 124.38, 52.37, 21.25.

25. 3,3'-bis(2,3-dimethylbenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2v)



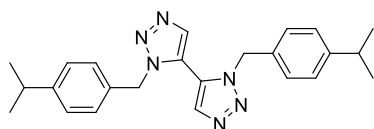
White solid (156 mg, 84% yield). m.p. 168–170 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): 7.42 (s, 2H), 7.12 (d, $J = 7.5$ Hz, 2H), 6.96 (t, $J = 7.6$ Hz, 2H), 6.30 (d, $J = 7.7$ Hz, 2H), 5.07 (s, 4H), 2.26 (s, 6H), 2.02 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3): δ 137.75, 135.85, 134.47, 132.29, 130.46, 126.03, 125.66, 123.89, 50.92, 20.44, 14.68. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{22}\text{H}_{25}\text{N}_6$ 373.2135; found 373.2134.

26. 3,3'-bis(3,5-dimethylbenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2w)



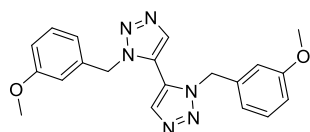
White solid (103 mg, 55% yield). m.p. 158–159 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.49 (s, 2H), 6.92 (s, 2H), 6.52 (s, 4H), 5.01 (s, 4H), 2.22 (s, 12H). ^{13}C NMR (101 MHz, CDCl_3): δ 138.84, 135.93, 134.07, 130.36, 125.08, 123.73, 52.36, 21.13. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{22}\text{H}_{25}\text{N}_6$ 373.2135; found 373.2135.

27. 3,3'-bis(4-isopropylbenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2x)



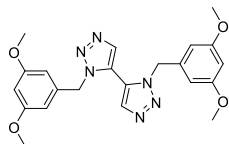
White solid (80 mg, 40% yield). m.p. 113–114 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.48 (s, 2H), 7.17 – 7.12 (m, 4H), 6.89 – 6.83 (m, 4H), 5.01 (s, 4H), 2.87 (p, $J = 6.9$ Hz, 2H), 1.20 (d, $J = 6.9$ Hz, 12H). ^{13}C NMR (101 MHz, CDCl_3): δ 149.81, 135.98, 131.59, 127.49, 127.13, 123.65, 52.16, 33.81, 23.86. HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{24}\text{H}_{28}\text{N}_6\text{Na}$ 423.2268; found 423.2269.

28. 3,3'-bis(3-methoxybenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2i)



White solid (122 mg, 65% yield). m.p. 147–149 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.52 (s, 2H), 7.19 (t, $J = 8.0$ Hz, 2H), 6.87 – 6.80 (m, 2H), 6.51 – 6.45 (m, 4H), 5.10 (s, 4H), 3.72 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3): δ 160.08, 135.91, 135.65, 130.23, 123.72, 119.38, 114.42, 112.70, 55.29, 52.30. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{20}\text{H}_{21}\text{N}_6\text{O}_2$ 377.1721; found 377.1720.

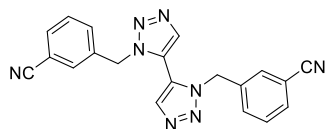
29. 3,3'-bis(3,5-dimethoxybenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2y)



White solid (70 mg, 32% yield). m.p. 104–105 °C. Petroleum ether/EtOH as the

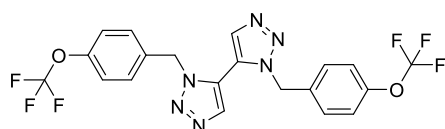
recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.56 (s, 2H), 6.36 (t, $J = 2.3$ Hz, 2H), 6.06 (d, $J = 2.2$ Hz, 4H), 5.10 (s, 4H), 3.70 (s, 12H). ^{13}C NMR (101 MHz, CDCl_3): δ 161.32, 136.33, 135.85, 123.78, 105.14, 100.63, 55.38, 52.40. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{22}\text{H}_{25}\text{N}_6\text{O}_4$ 437.1932; found 437.1941.

30. 3,3'-(3*H*,3'*H*-[4,4'-bi(1,2,3-triazole)]-3,3'-diylbis(methylene))dibenzonitrile (2z)



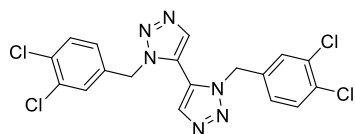
White solid (117 mg, 64% yield). m.p. 160–161 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 8.18 (s, 2H), 7.73 (d, $J = 8.1$ Hz, 2H), 7.50 – 7.39 (m, 4H), 7.20 (d, $J = 8.1$ Hz, 2H), 5.64 (s, 4H). ^{13}C NMR (101 MHz, $\text{DMSO}-d_6$): δ 136.81, 135.84, 132.55, 132.41, 131.44, 130.39, 124.44, 118.68, 112.15, 51.06. HRMS (ESI) m/z : $[\text{M}-\text{H}]^+$ Calcd for $\text{C}_{20}\text{H}_{13}\text{N}_8$ 365.1258; found 365.1266.

31. 3,3'-bis(4-(trifluoromethoxy)benzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2aa)



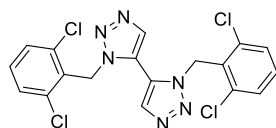
White solid (99 mg, 41% yield). m.p. 191–192 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.60 (s, 2H), 7.18 – 7.11 (m, 4H), 7.03 – 6.96 (m, 4H), 5.16 (s, 4H). ^{13}C NMR (101 MHz, CDCl_3): δ 149.50, 135.76, 132.57, 129.00, 123.57, 121.55, 119.01, 51.48. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{20}\text{H}_{15}\text{F}_6\text{N}_6\text{O}_2$ 485.1155; found 485.1155.

32. 3,3'-bis(3,4-dichlorobenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2ab)



White solid (67 mg, 30% yield). m.p. 178–179 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.66 (s, 2H), 7.36 (d, $J = 8.2$ Hz, 2H), 7.11 (d, $J = 2.1$ Hz, 2H), 6.78 (dd, $J = 8.3, 2.2$ Hz, 2H), 5.22 (s, 4H). ^{13}C NMR (101 MHz, CDCl_3): δ 135.58, 133.82, 133.61, 133.56, 131.16, 129.27, 126.48, 123.56, 51.08. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{18}\text{H}_{13}\text{Cl}_4\text{N}_6$ 452.9950; found 452.9950.

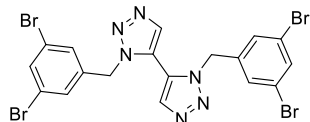
33. 3,3'-bis(2,6-dichlorobenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2ac)



White solid (144 mg, 64% yield). m.p. 202–204 °C. Petroleum ether/EtOH as the

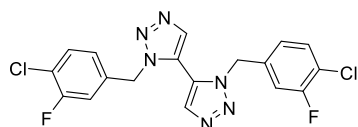
recrystallization solvent. ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 8.32 (s, 2H), 7.54 (d, J = 7.9 Hz, 4H), 7.46 (dd, J = 9.1, 7.2 Hz, 2H), 5.71 (s, 4H). ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$): δ 141.19, 140.07, 136.73, 135.19, 133.97, 129.66, 53.14. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{18}\text{H}_{13}\text{Cl}_4\text{N}_6$ 452.9950; found 452.9953.

34. 3,3'-bis(3,5-dibromobenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2ad)



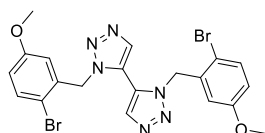
White solid (136 mg, 43% yield). m.p. 193–194 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 8.17 (s, 2H), 7.73 (t, J = 1.8 Hz, 2H), 7.16 (d, J = 1.7 Hz, 4H), 5.60 (s, 4H). ^{13}C NMR (101 MHz, CDCl_3): δ 144.20, 140.60, 138.51, 134.69, 129.17, 127.83, 55.27. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{18}\text{H}_{13}\text{Br}_4\text{N}_6$ 628.7930; found 628.7933.

35. 3,3'-bis(4-chloro-3-fluorobenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2ae)



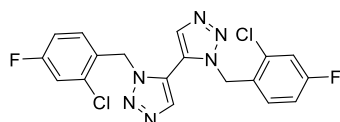
White solid (80 mg, 38% yield). m.p. 174–177 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.65 (s, 2H), 7.33 (t, J = 7.8 Hz, 2H), 6.82 (dd, J = 9.2, 2.1 Hz, 2H), 6.68 (dd, J = 8.3, 2.0 Hz, 2H), 5.25 (s, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.53, 157.02, 135.52, 134.49, 134.43, 131.47, 123.60, 123.57, 123.53, 122.19, 122.01, 115.74, 115.52, 51.23, 51.21. HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{18}\text{H}_{12}\text{Cl}_2\text{F}_2\text{N}_6\text{Na}$ 443.0361; found 443.0378.

36. 3,3'-bis(2-bromo-5-methoxybenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2af)



White solid (88 mg, 33% yield). m.p. 123–124 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.57 (s, 2H), 7.44 (d, J = 8.8 Hz, 2H), 6.74 (dd, J = 8.8, 3.0 Hz, 2H), 6.28 (d, J = 3.0 Hz, 2H), 5.38 (s, 4H), 3.68 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3): δ 159.47, 135.49, 134.50, 133.89, 123.95, 115.88, 114.40, 112.45, 55.50, 52.10. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{20}\text{H}_{19}\text{Br}_2\text{N}_6\text{O}_2$ 532.9931; found 532.9931.

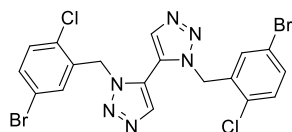
37. 3,3'-bis(2-chloro-4-fluorobenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2ag)



White solid (101 mg, 48% yield). m.p. 154–156 °C. Petroleum ether/EtOH as the recrystallization solvent. ^1H NMR (400 MHz, CDCl_3): δ 7.61 (s, 2H), 7.12 (dd, J = 8.2, 2.5 Hz, 2H), 6.97 – 6.83 (m, 4H), 5.39 (s, 4H). ^{13}C NMR (101 MHz, CDCl_3): δ 163.72,

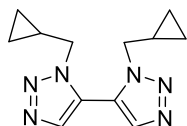
161.20, 135.42, 133.72, 133.62, 130.46, 130.38, 127.83, 127.80, 123.79, 117.57, 117.33, 115.08, 114.86, 48.96. HRMS (ESI) m/z : $[M+H]^+$ Calcd for $C_{18}H_{13}Cl_2F_2N_6$ 421.0541; found 421.0541.

38. 3,3'-bis(5-bromo-2-chlorobenzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2ah)



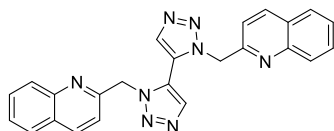
White solid (121 mg, 45% yield). m.p. 150–151 °C. Petroleum ether/EtOH as the recrystallization solvent. 1H NMR (400 MHz, $CDCl_3$): δ 7.64 (s, 2H), 7.41 (dd, J = 8.5, 2.4 Hz, 2H), 7.25 (d, J = 8.5 Hz, 2H), 6.97 (d, J = 2.3 Hz, 2H), 5.41 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 135.53, 133.75, 133.31, 131.50, 131.36, 123.79, 121.33, 48.98. HRMS (ESI) m/z : $[M+H]^+$ Calcd for $C_{18}H_{13}Cl_2Br_2N_6$ 540.8940; found 540.8940.

39. 3,3'-bis(cyclopropylmethyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2ai)



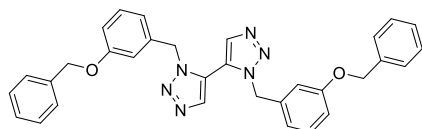
White solid (73 mg, 60% yield). m.p. 86–87 °C. Petroleum ether/EtOH as the recrystallization solvent. 1H NMR (400 MHz, $CDCl_3$): δ 7.85 (s, 2H), 4.14 (d, J = 7.2 Hz, 4H), 1.13 (tt, J = 7.6, 4.8 Hz, 2H), 0.60 – 0.54 (m, 4H), 0.31 – 0.24 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 135.01, 123.72, 53.49, 11.40, 4.34. HRMS (ESI) m/z : $[M+H]^+$ Calcd for $C_{12}H_{17}N_6$ 245.1509; found 245.1509.

40. 3,3'-bis(quinolin-2-ylmethyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2aj)



Yellow solid (117 mg, 56% yield). m.p. 150–152 °C. Petroleum ether/EtOH as the recrystallization solvent. 1H NMR (400 MHz, $CDCl_3$): δ 8.07 (d, J = 8.4 Hz, 2H), 8.02 (s, 2H), 7.98 (d, J = 8.6 Hz, 2H), 7.78 – 7.68 (m, 4H), 7.54 (ddd, J = 8.1, 6.8, 1.2 Hz, 2H), 7.23 (d, J = 8.5 Hz, 2H), 5.77 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 153.91, 147.56, 137.60, 135.52, 130.26, 129.23, 127.66, 127.40, 127.20, 125.10, 119.12, 54.11. HRMS (ESI) m/z : $[M+H]^+$ Calcd for $C_{24}H_{19}N_8$ 419.1727; found 419.1727.

41. 3,3'-bis(3-(benzyloxy)benzyl)-3*H*,3'*H*-4,4'-bi(1,2,3-triazole) (2ak)



White solid (97 mg, 37% yield). m.p. 97–98 °C. Petroleum ether/EtOH as the recrystallization solvent. 1H NMR (400 MHz, $CDCl_3$): δ 7.46 (s, 2H), 7.40 – 7.28 (m, 10H), 7.19 (t, J = 7.9 Hz, 2H), 6.91 (ddd, J = 8.4, 2.6, 0.9 Hz, 2H), 6.55 (t, J = 2.0 Hz, 2H), 6.48 (dt, J = 7.7, 1.2 Hz, 2H), 5.08 (s, 4H), 4.98 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 159.24, 136.44, 135.85, 135.75, 130.25, 128.63, 128.07, 127.44, 123.72,

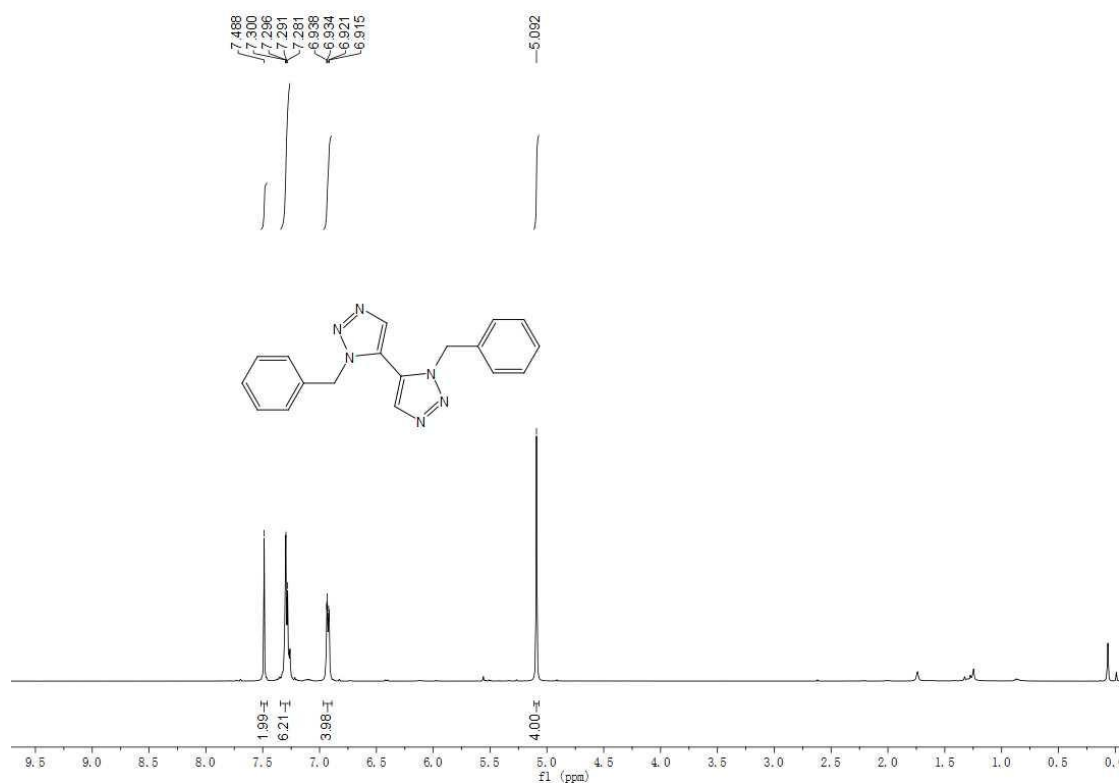
119.62, 115.38, 113.66, 70.00, 52.22. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{32}H_{29}N_6O_2$ 529.2347; found 529.2348.

4. References

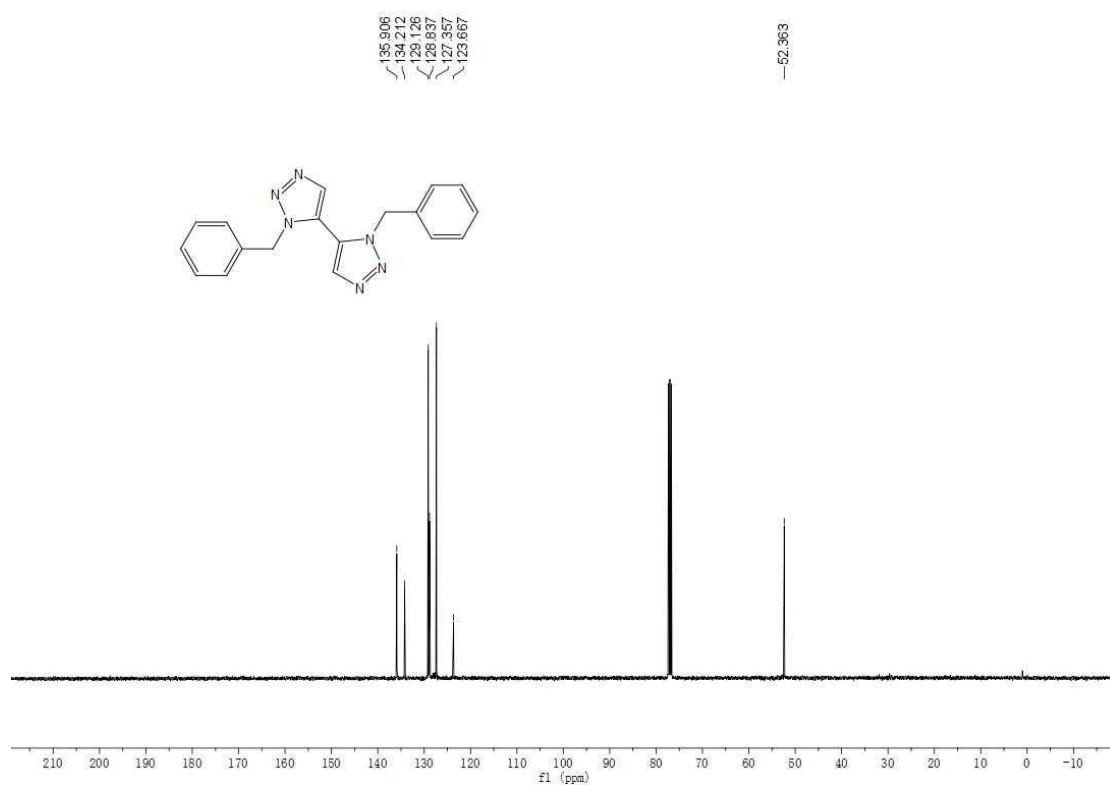
[1]. Angell, Y.; Burgess, K. Angew. Base Dependence in Copper-Catalyzed Huisgen Reactions: Efficient Formation of Bistriazoles, *Chem., Int. Ed.* 2007, 46, 3649-3651.

5. The ^1H NMR and ^{13}C NMR spectra for products

^1H and ^{13}C NMR spectra of compound **2a**

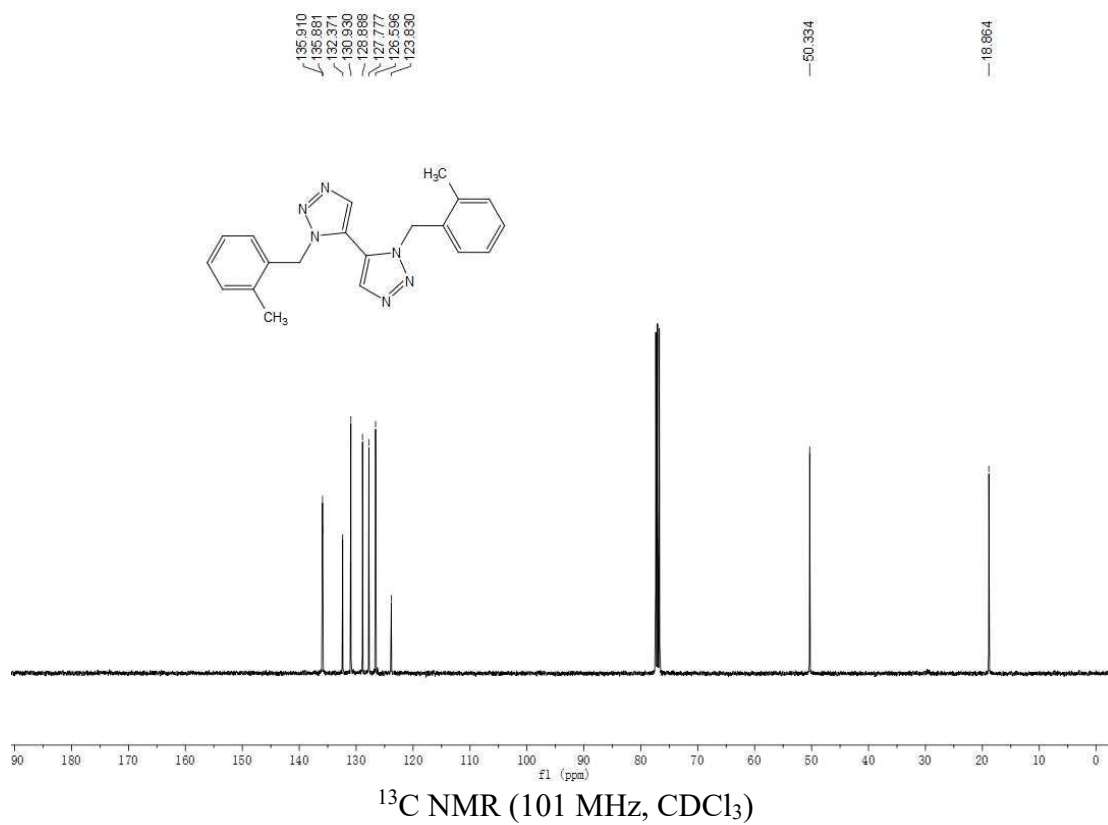
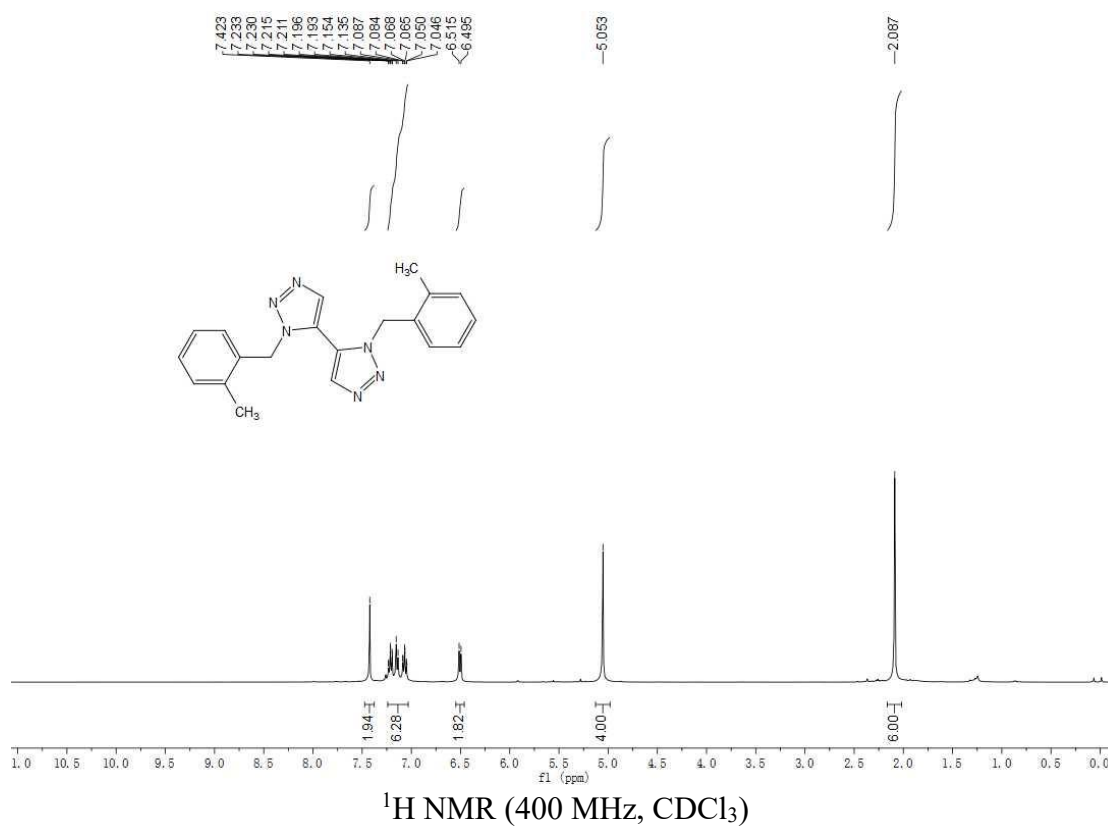


^1H NMR (400 MHz, CDCl_3)

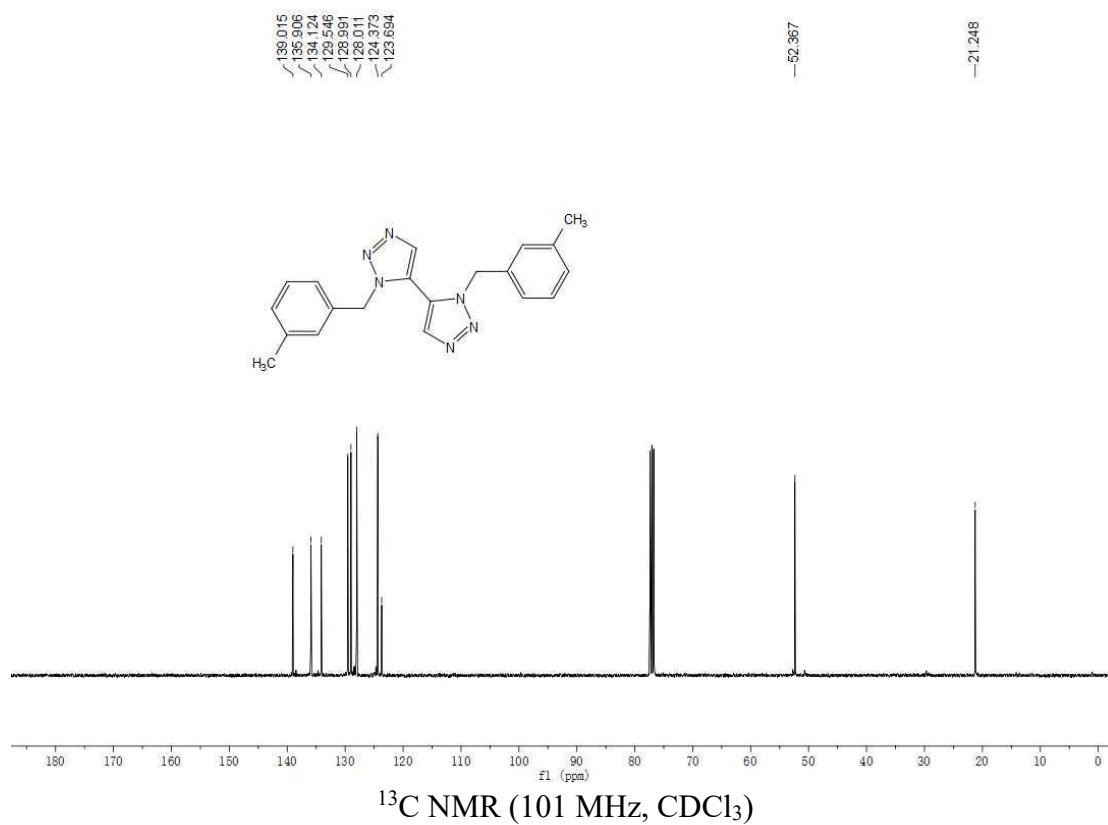
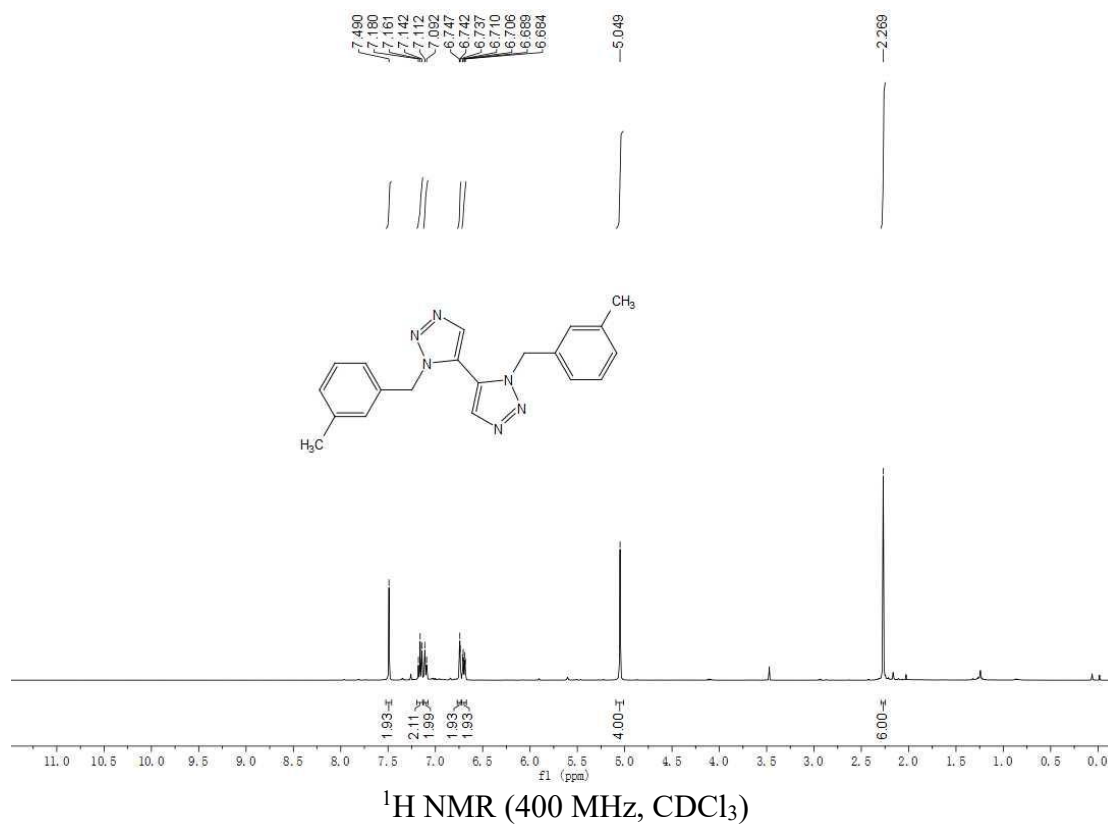


^{13}C NMR (101 MHz, CDCl_3)

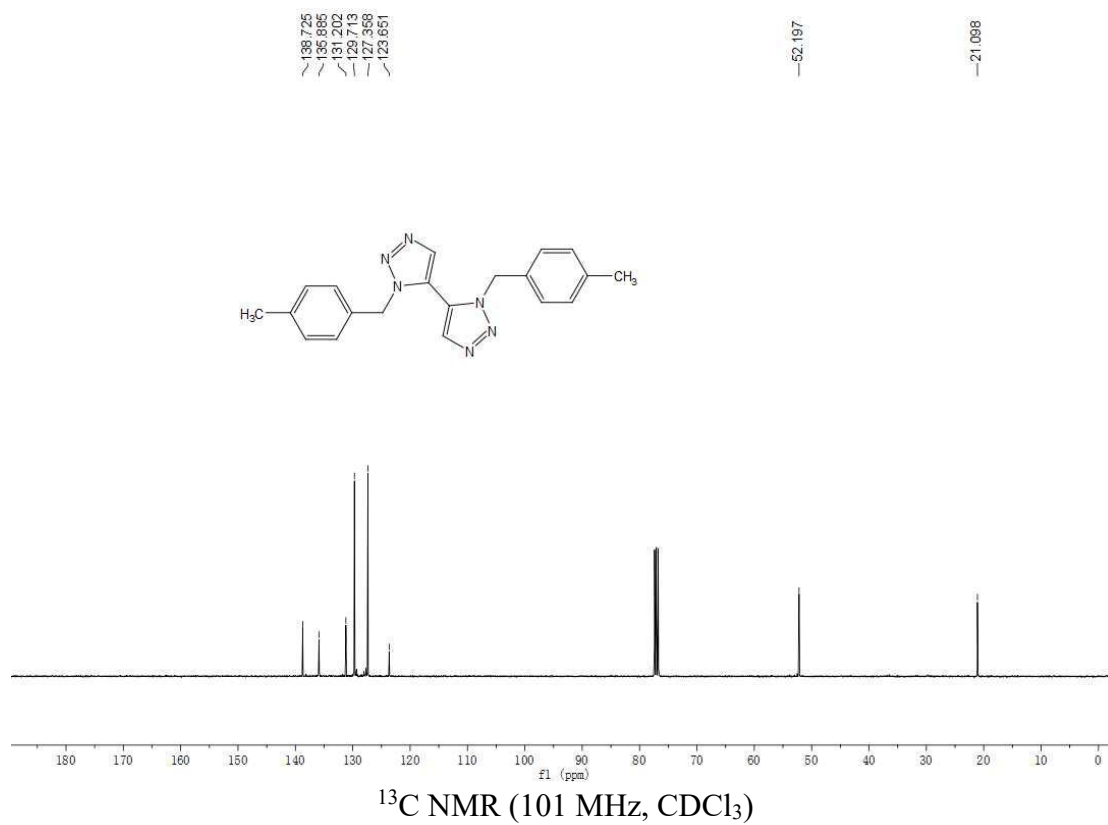
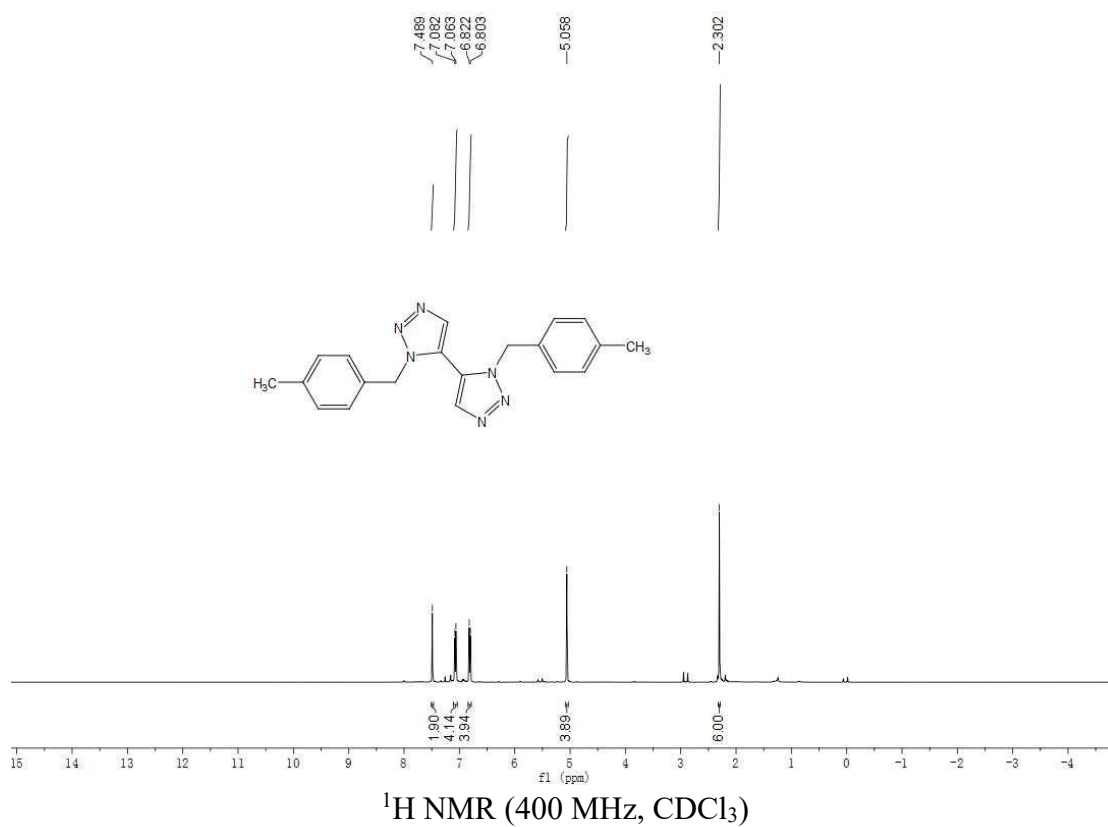
^1H and ^{13}C NMR spectra of compound **2b**



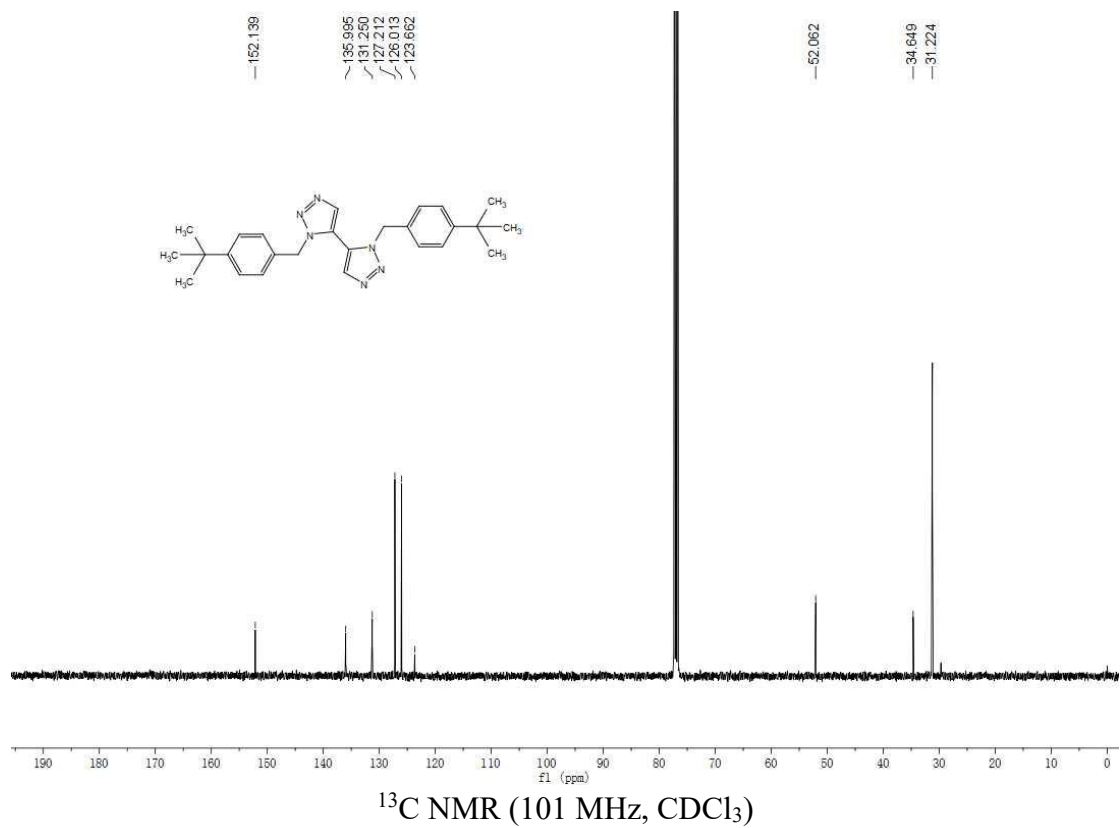
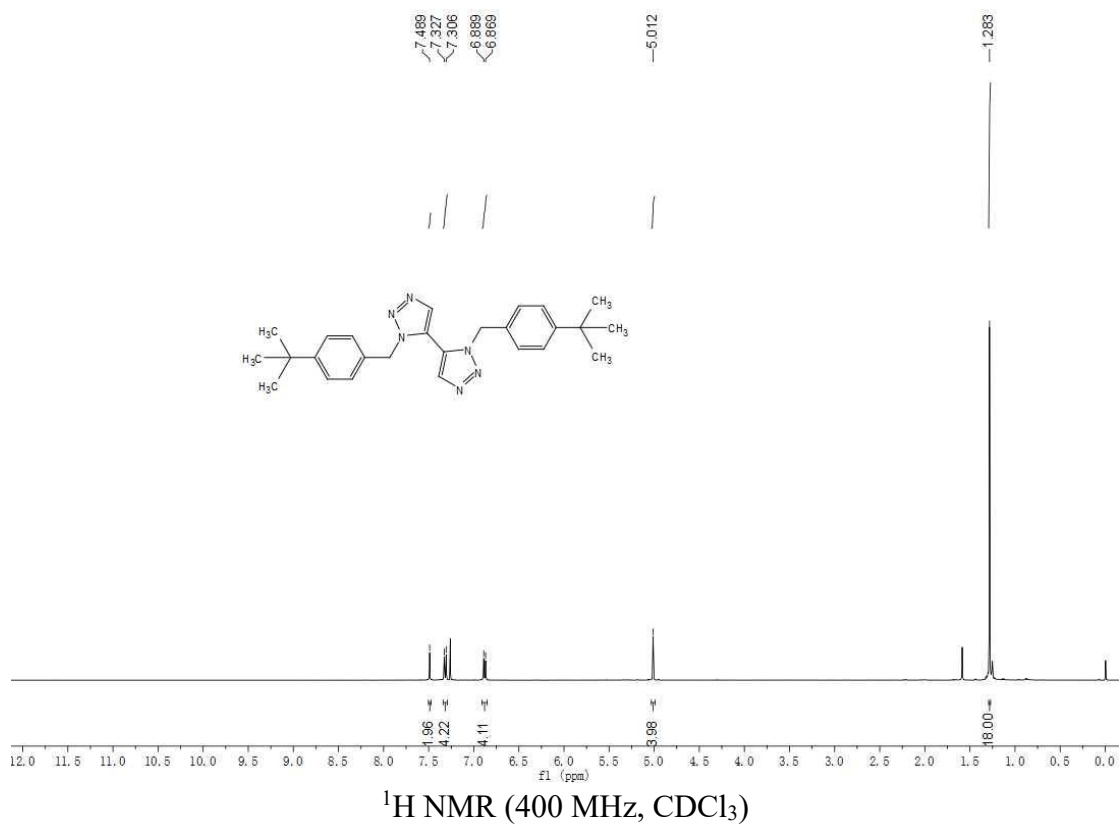
^1H and ^{13}C NMR spectra of compound **2c**



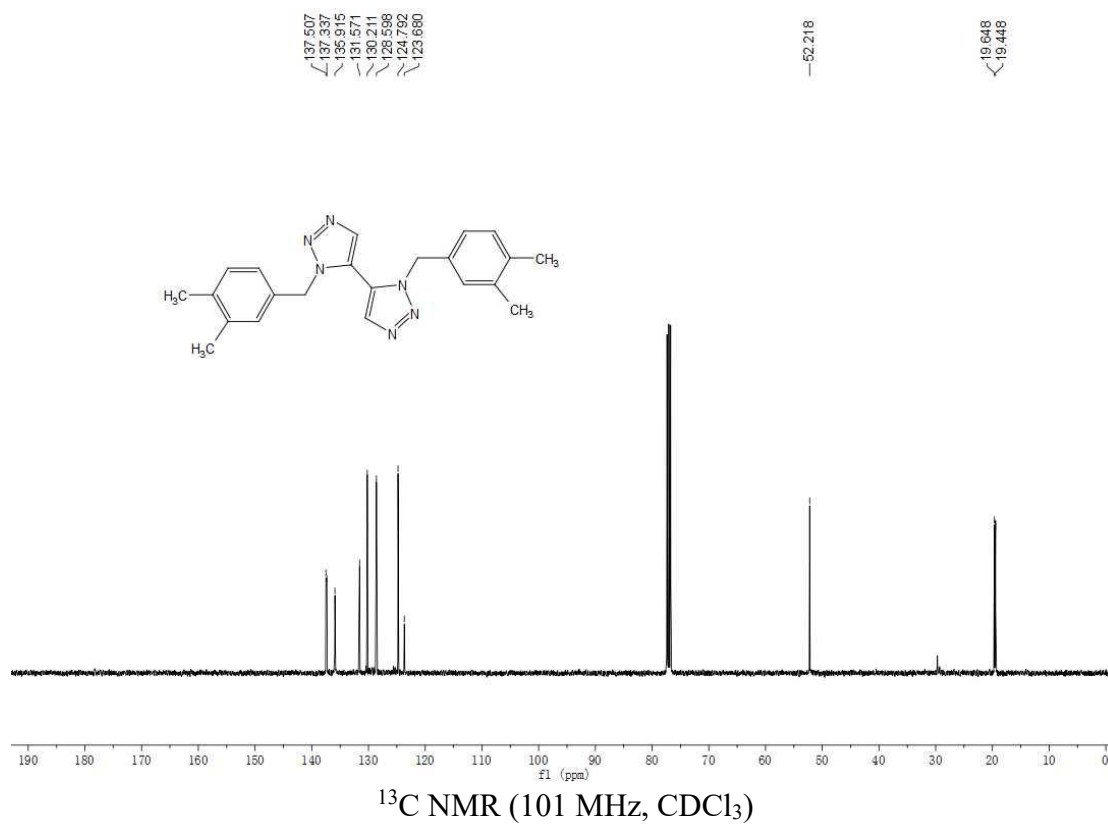
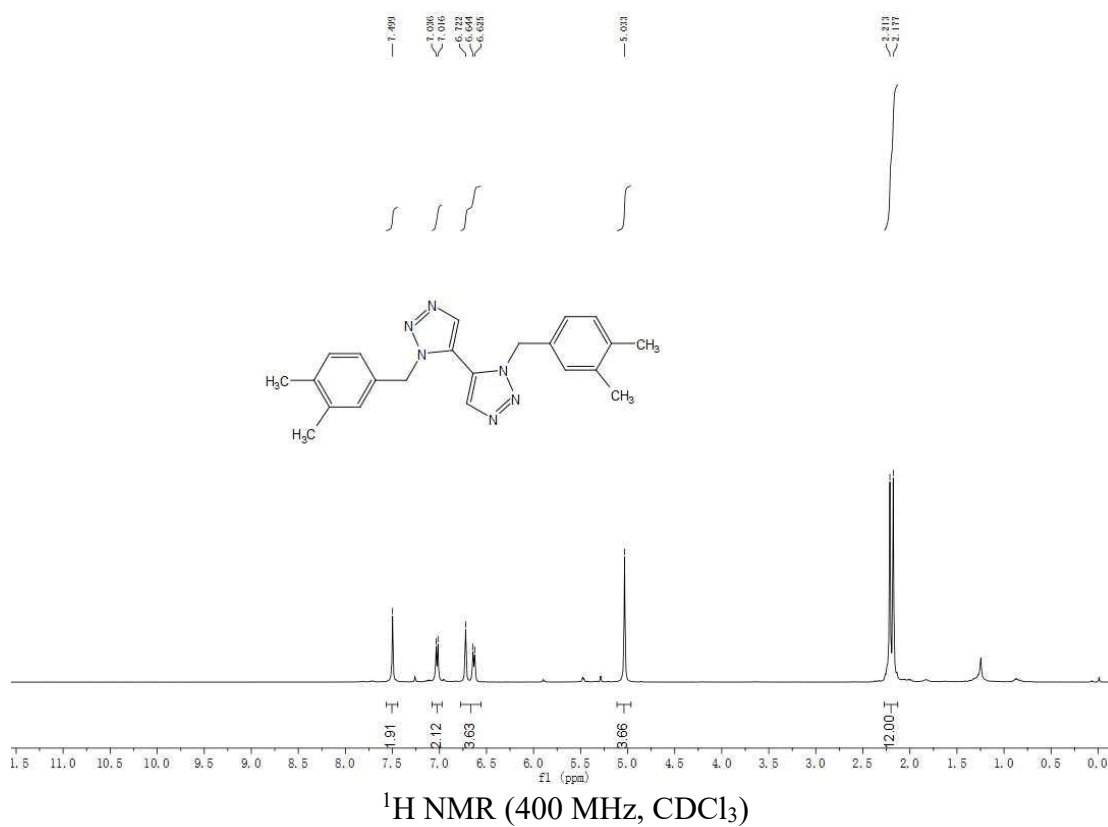
^1H and ^{13}C NMR spectra of compound **2d**



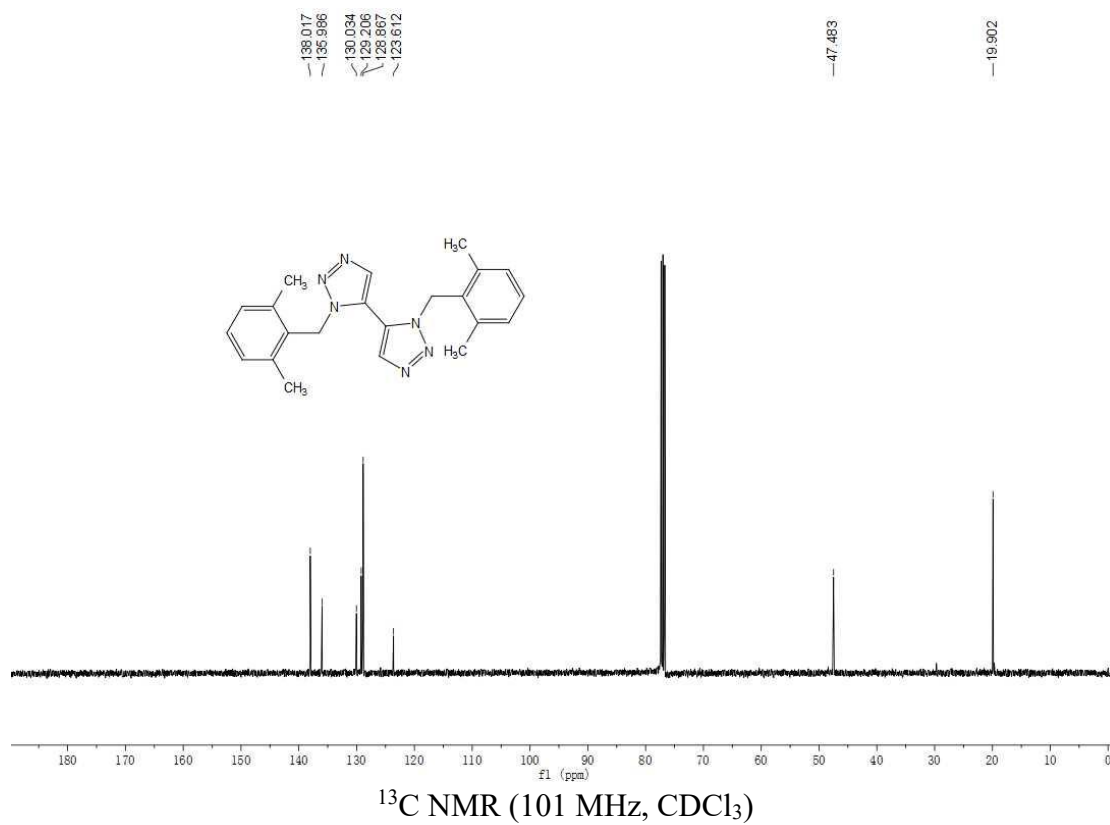
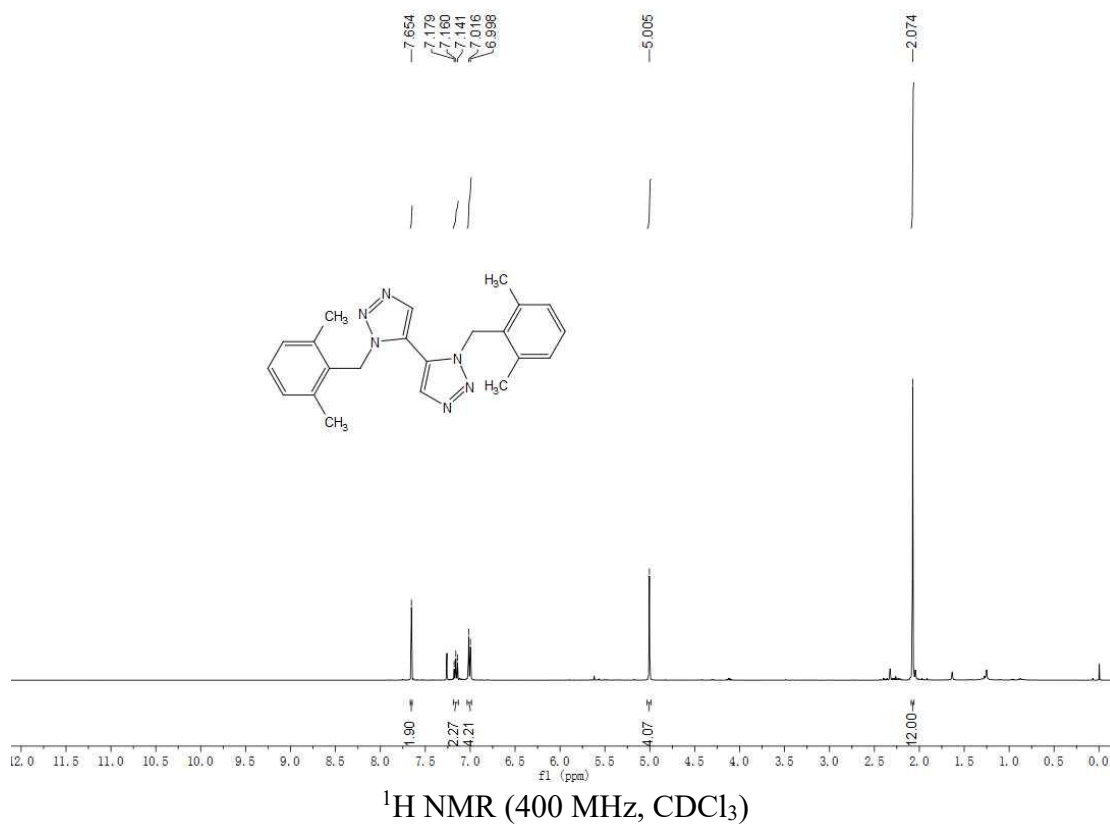
^1H and ^{13}C NMR spectra of compound **2e**



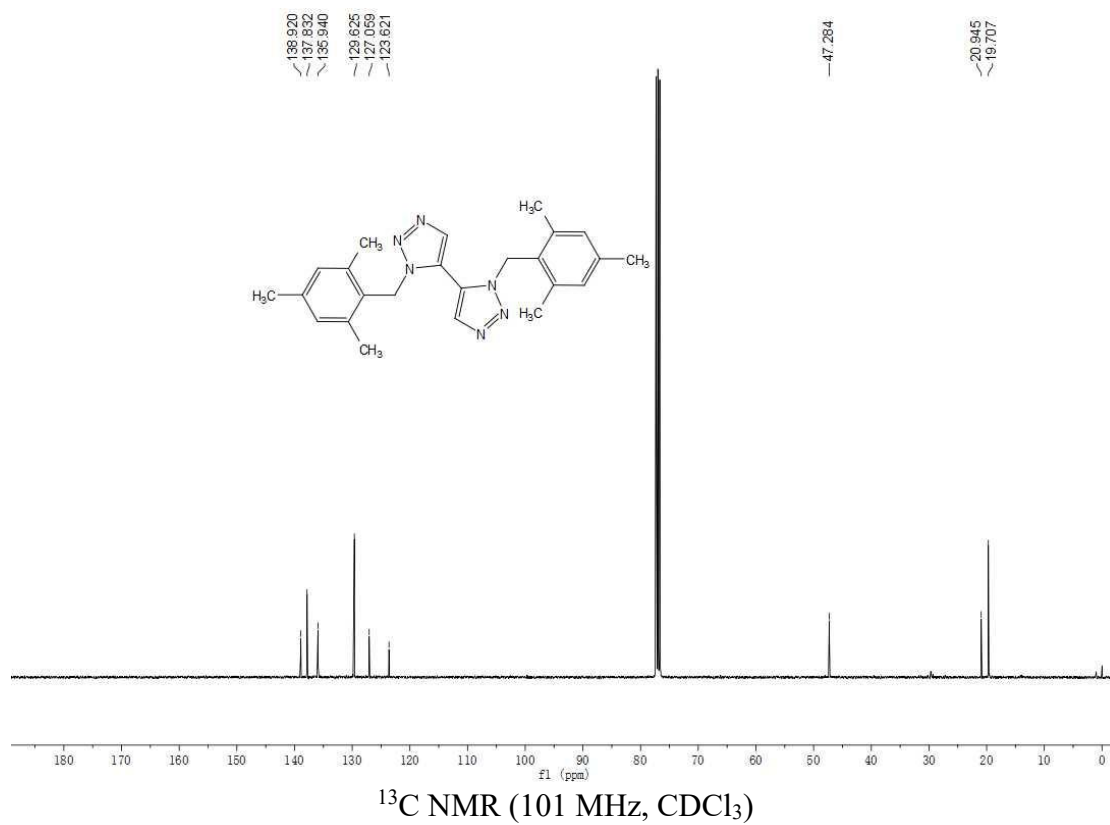
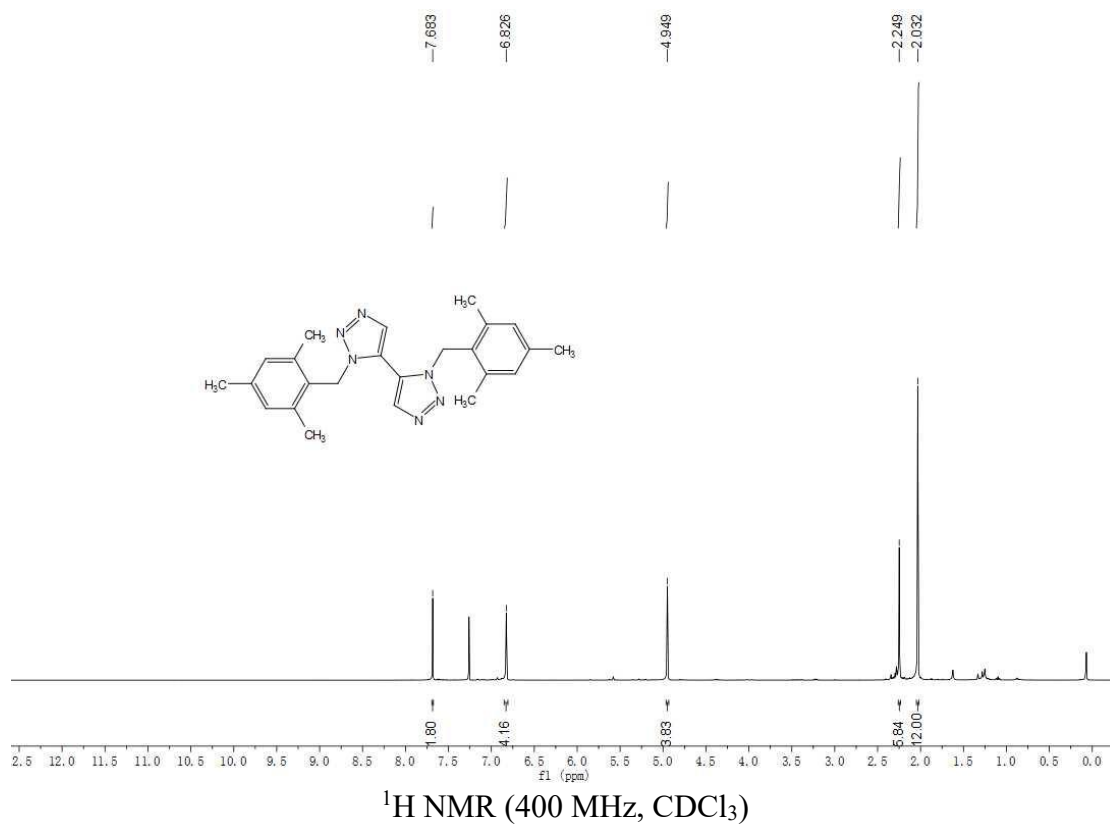
^1H and ^{13}C NMR spectra of compound **2f**



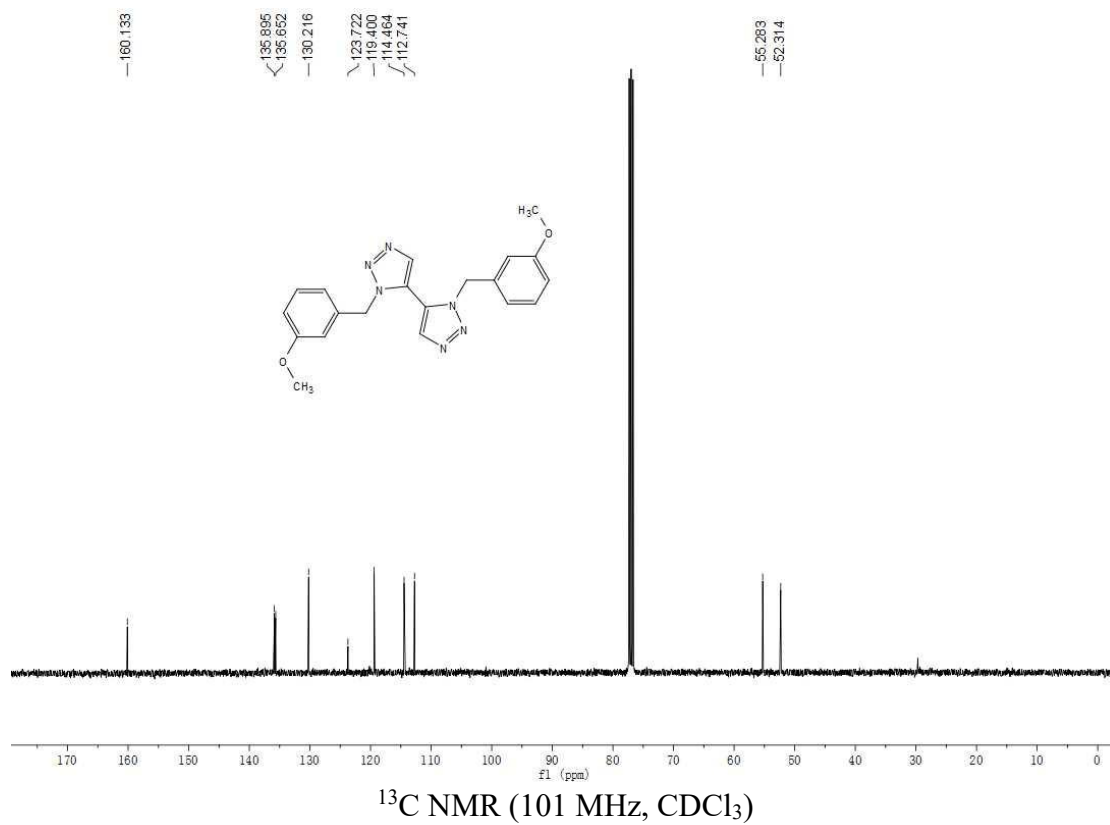
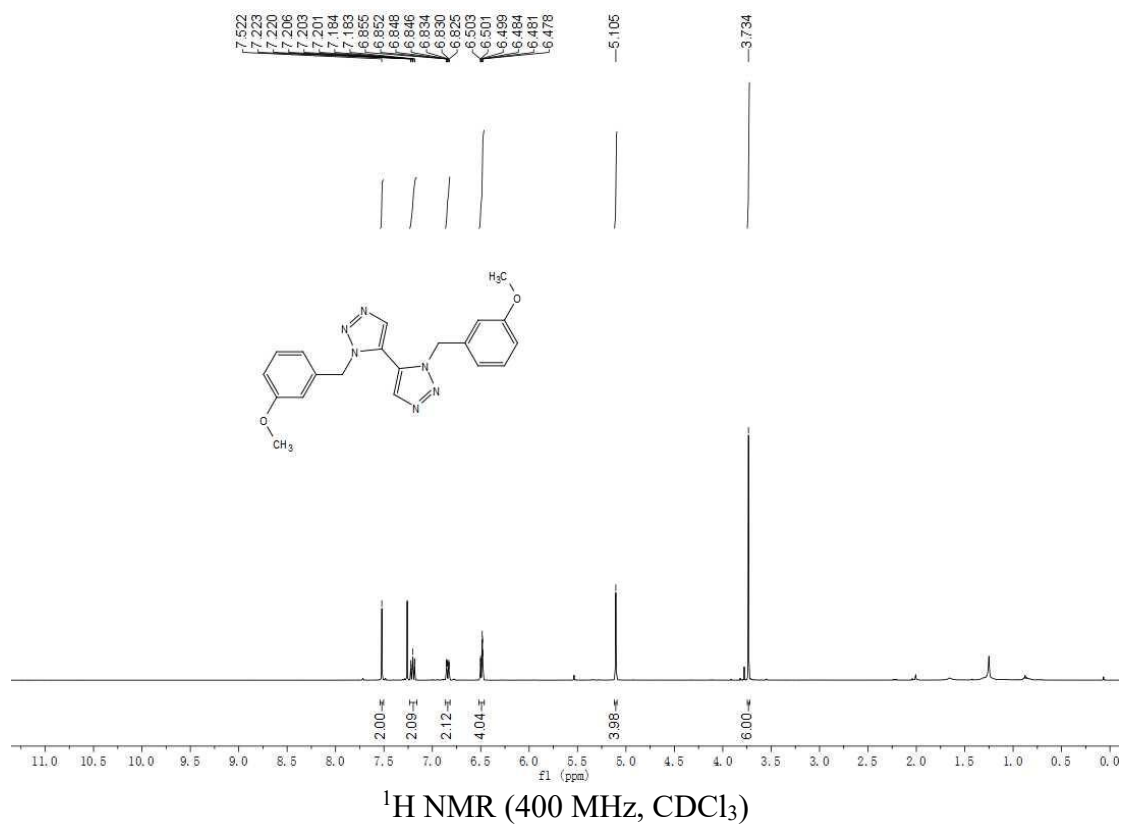
^1H and ^{13}C NMR spectra of compound **2g**



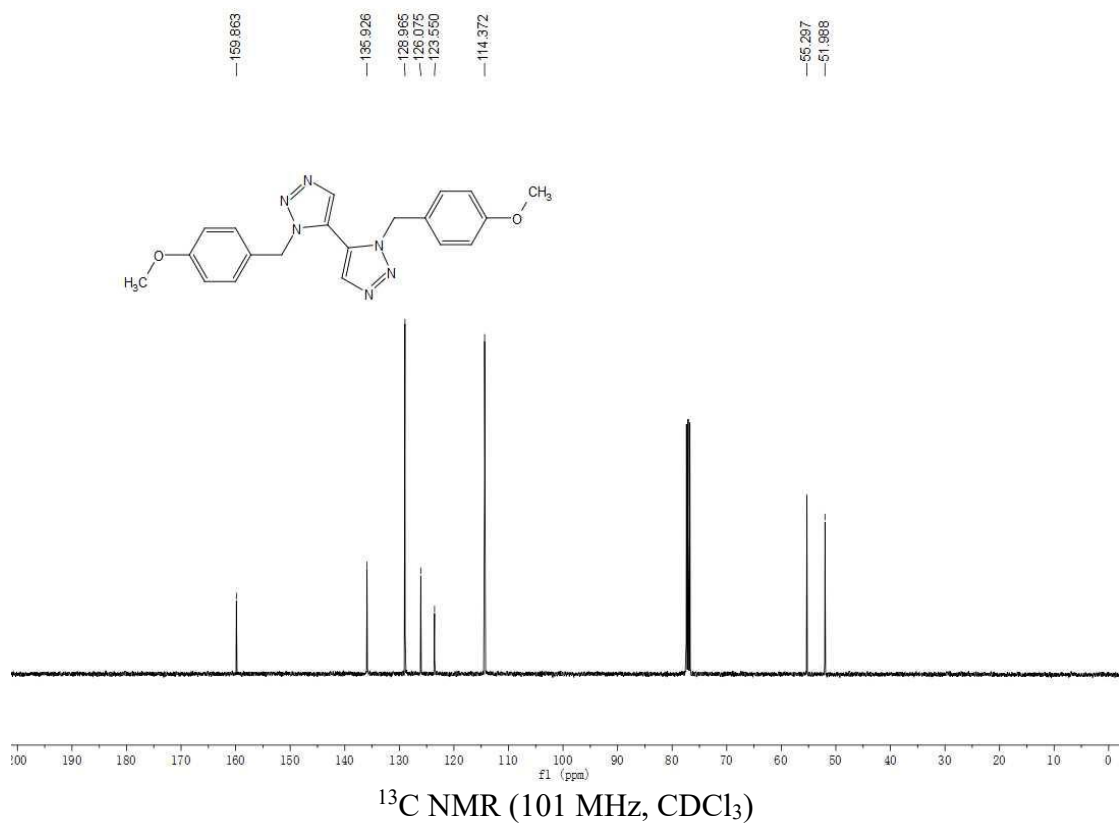
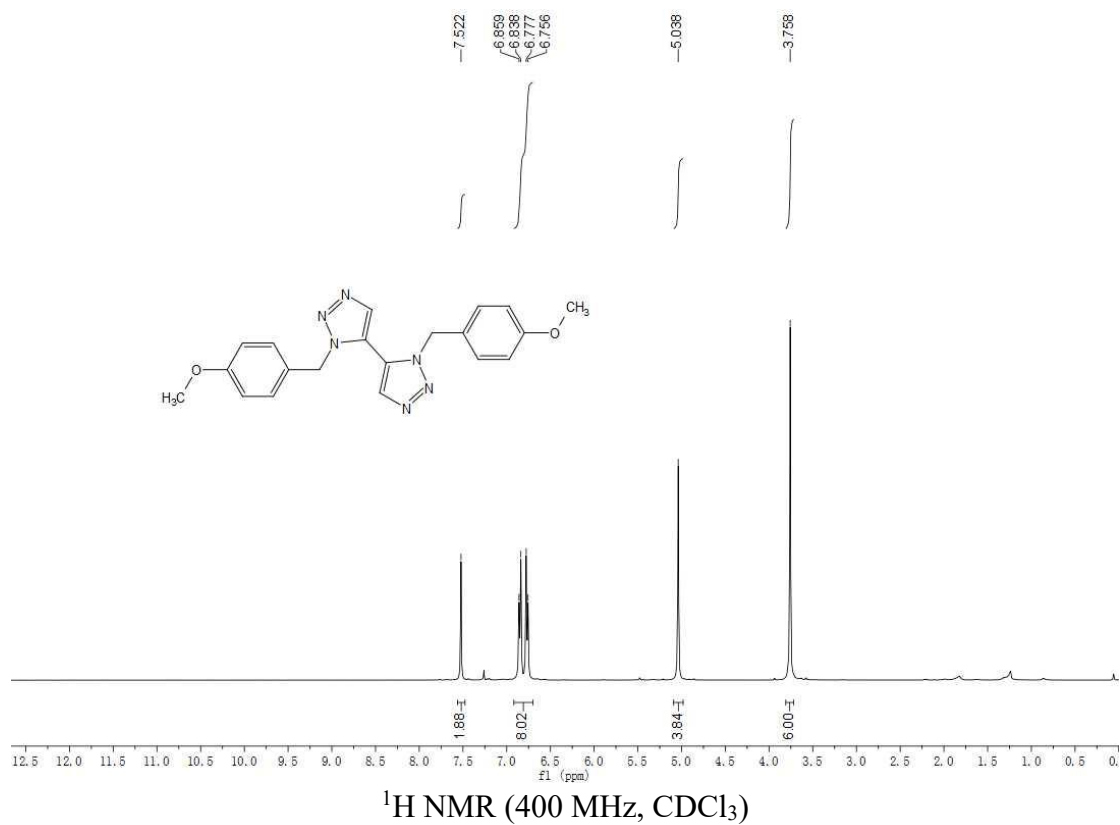
^1H and ^{13}C NMR spectra of compound **2h**



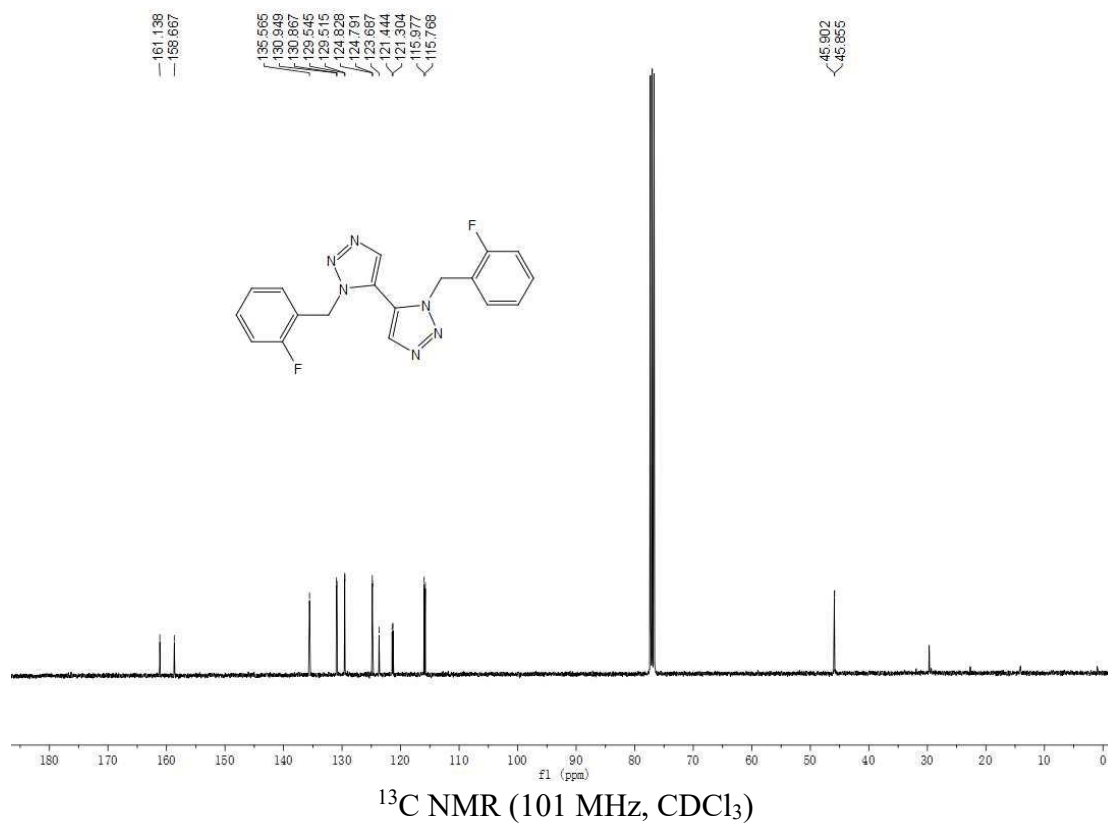
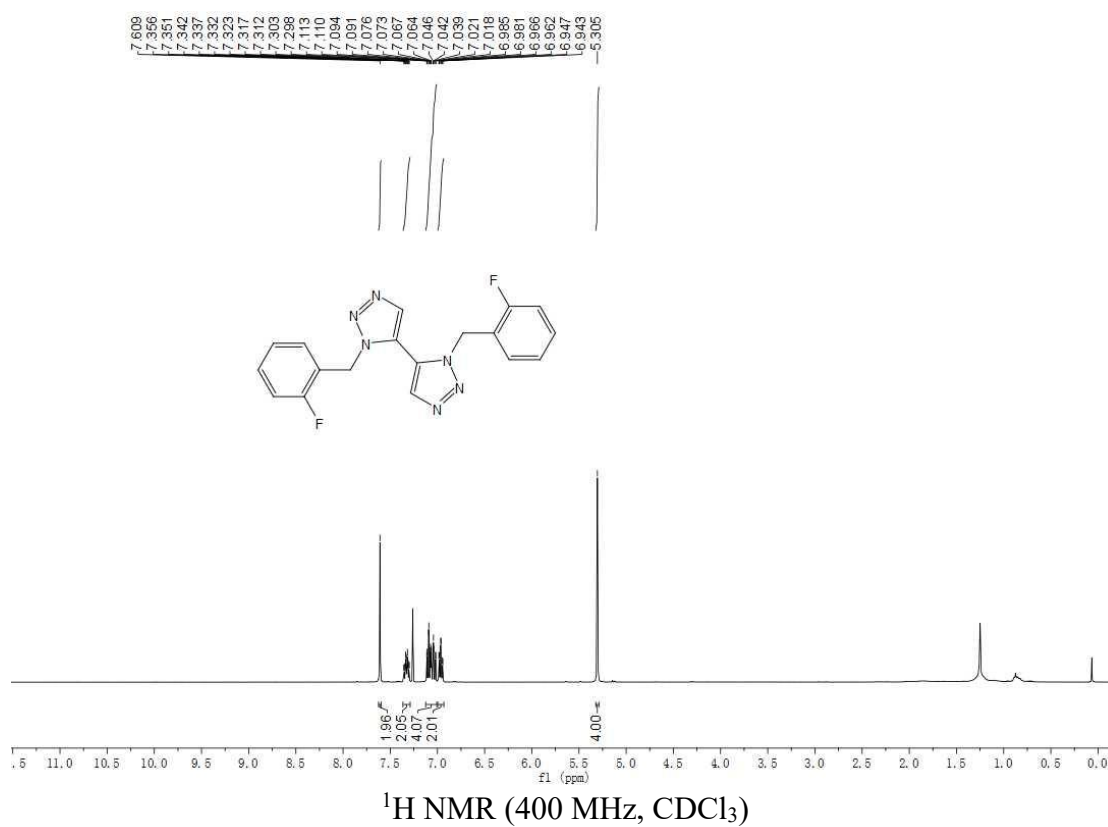
^1H and ^{13}C NMR spectra of compound **2i**



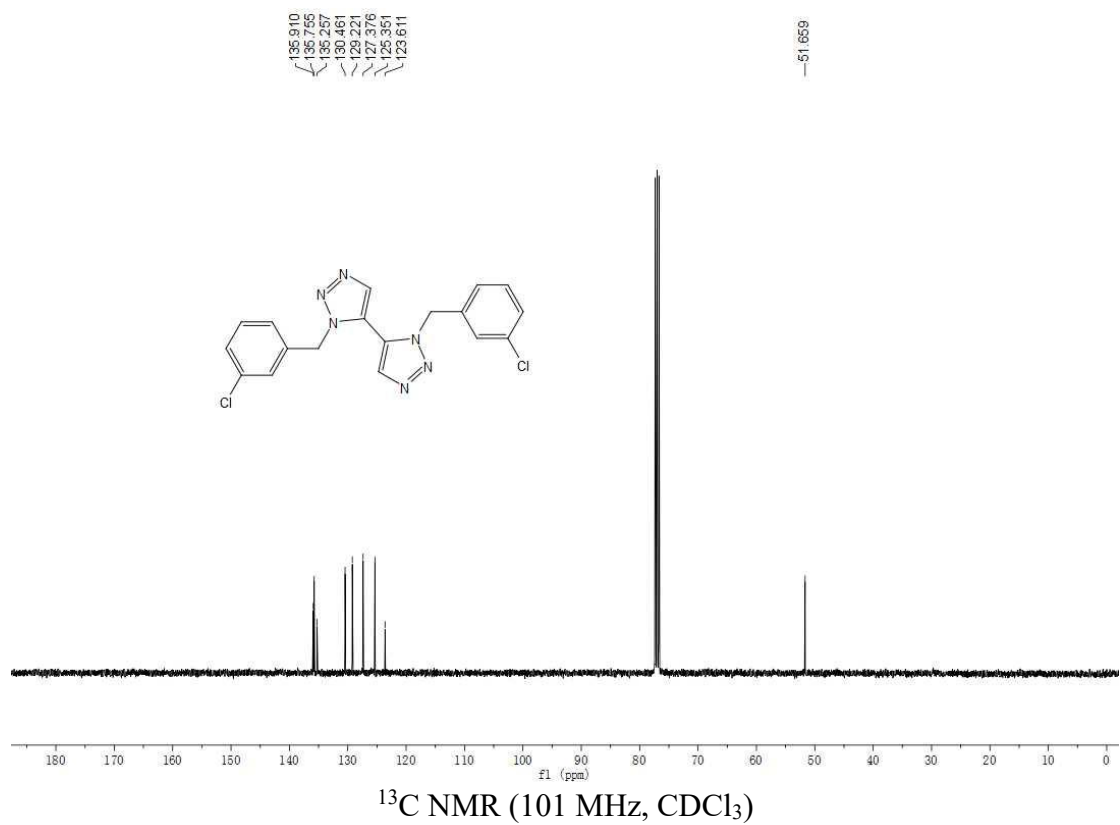
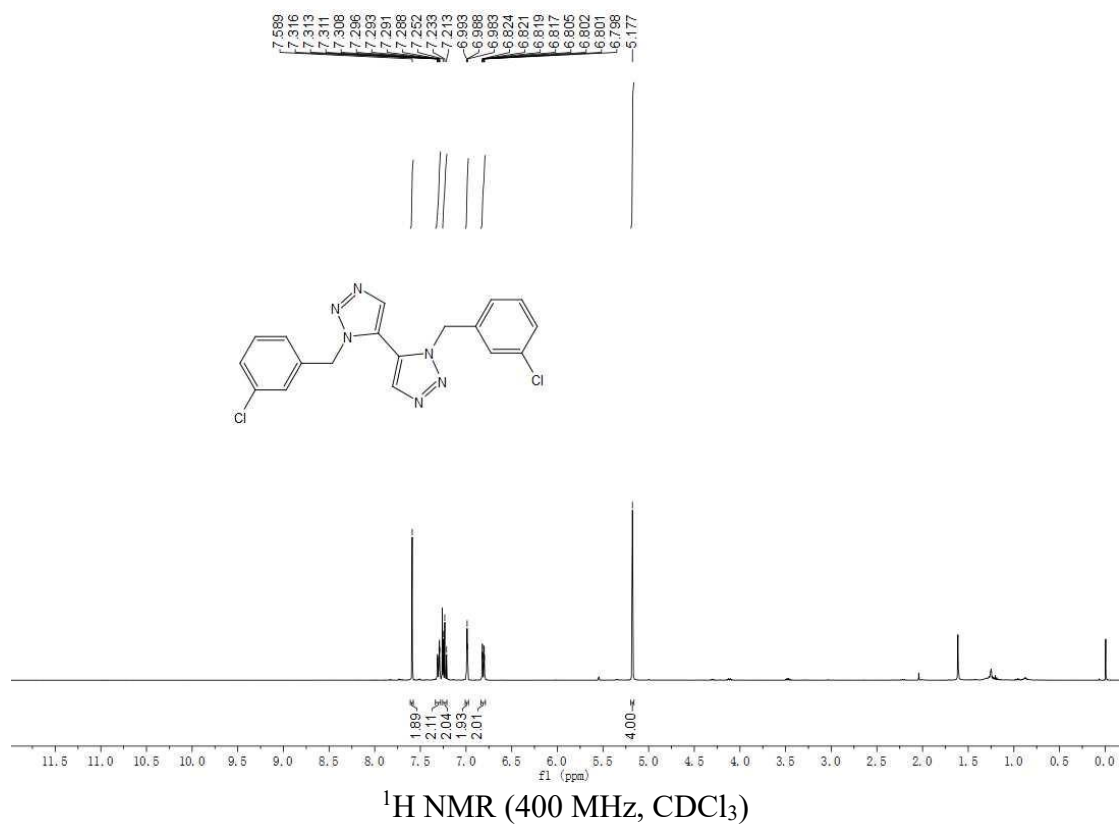
^1H and ^{13}C NMR spectra of compound **2j**



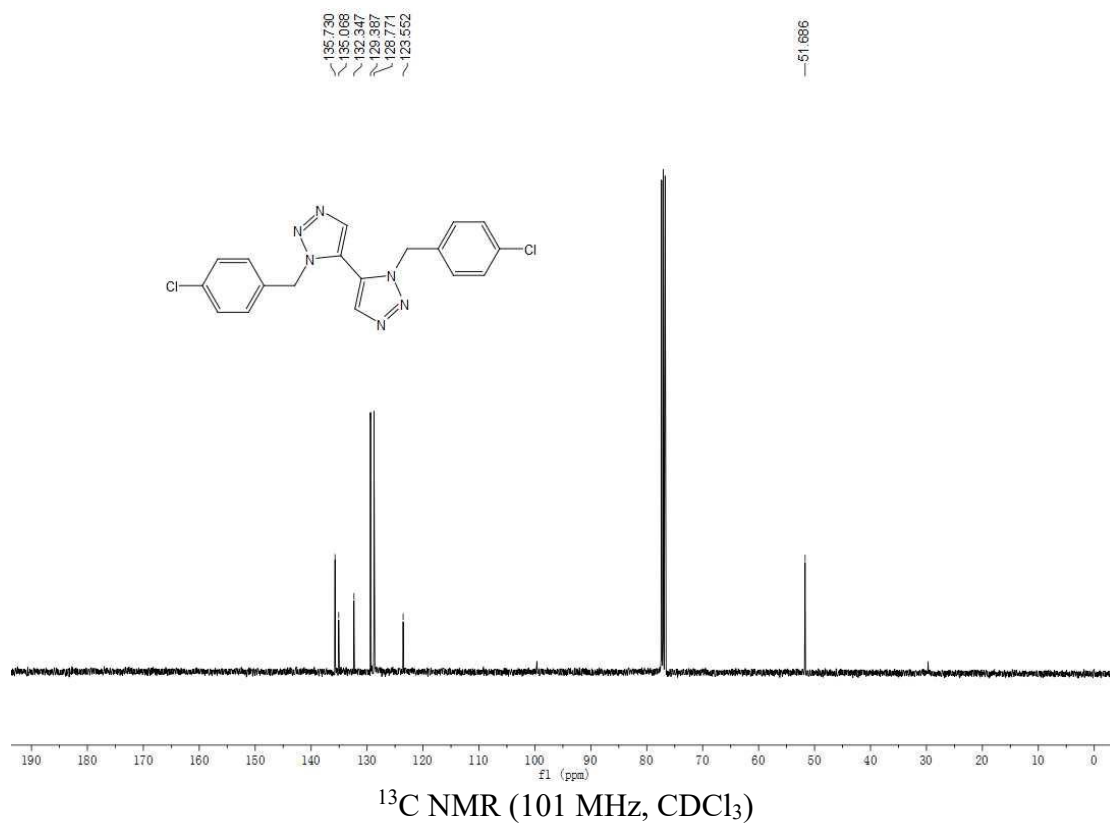
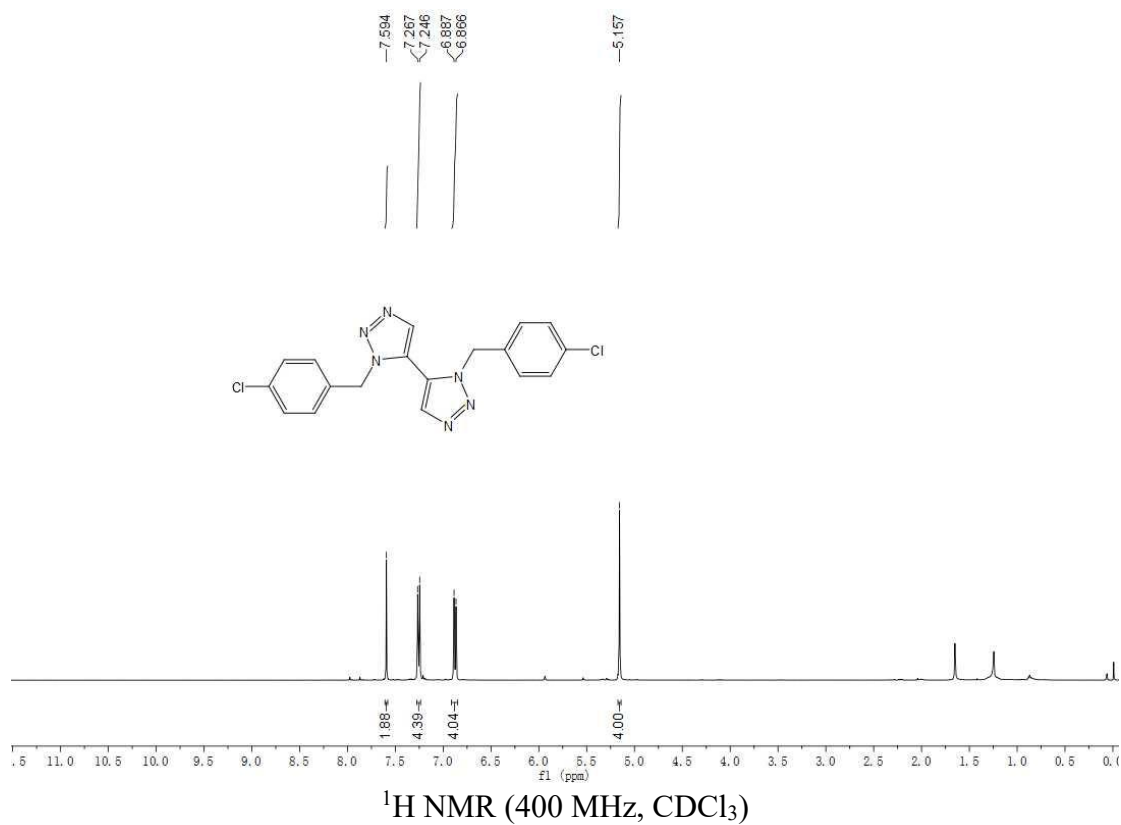
^1H and ^{13}C NMR spectra of compound **2k**



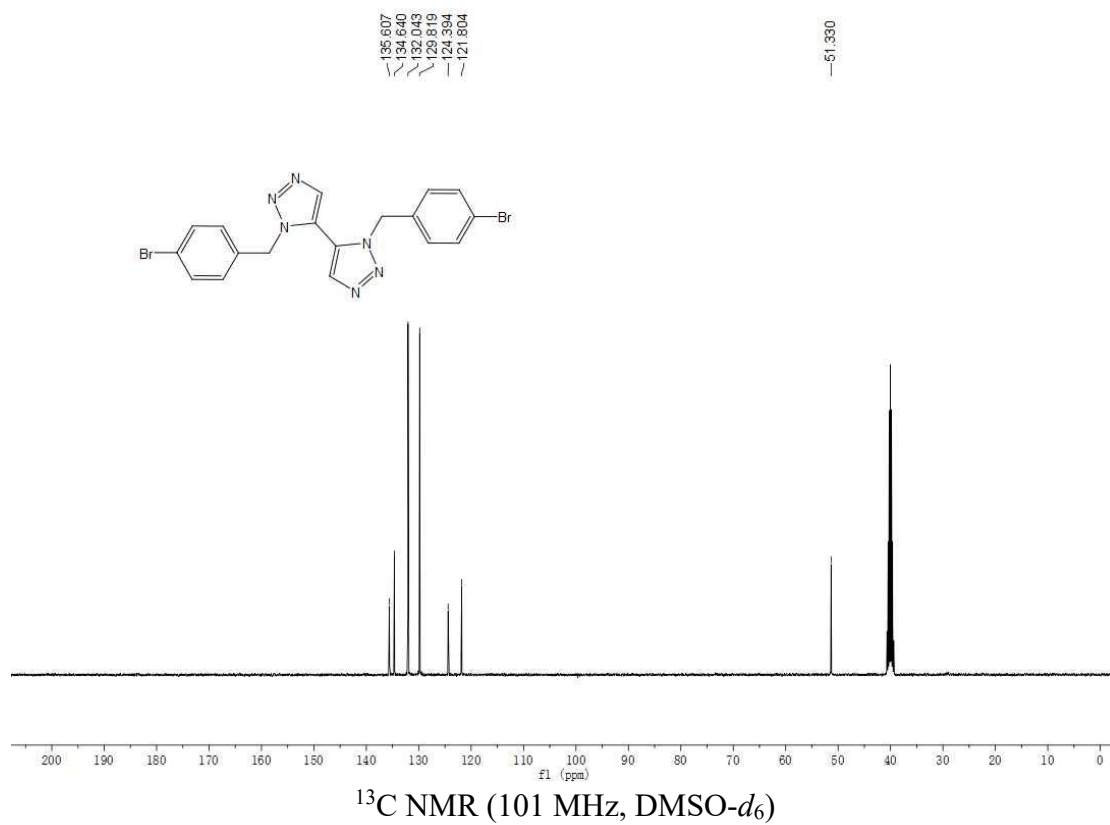
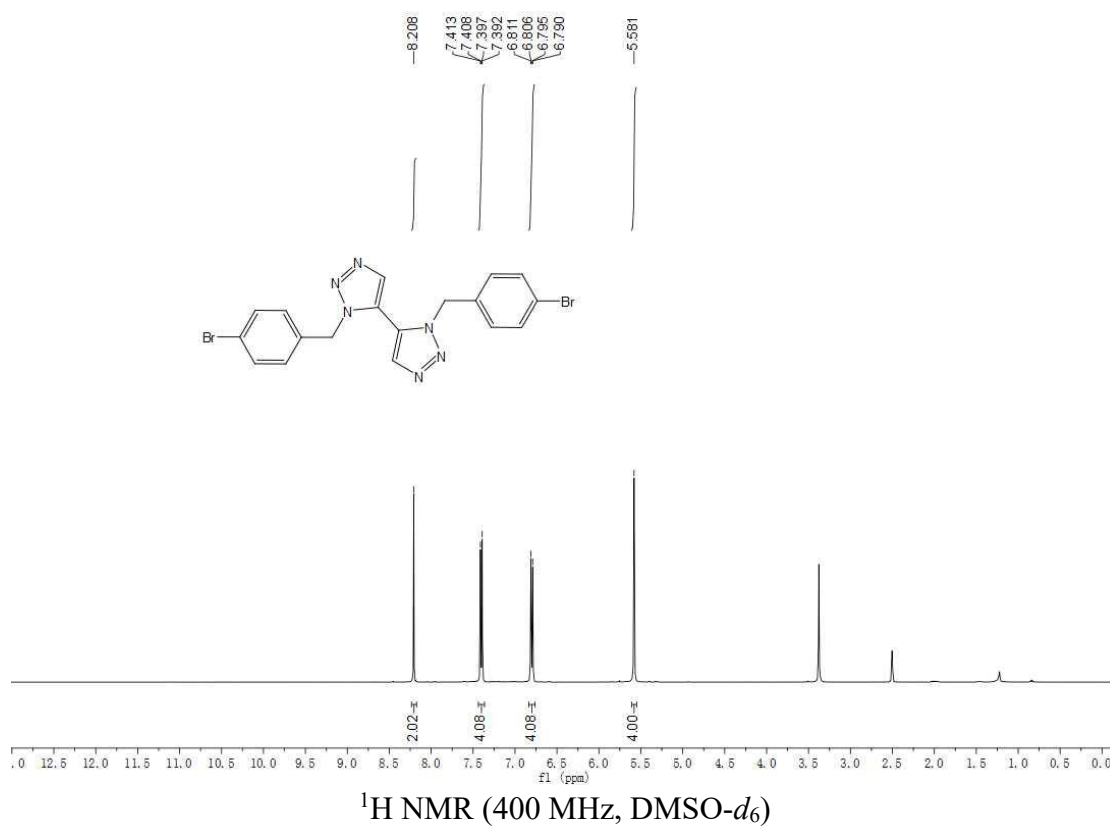
^1H and ^{13}C NMR spectra of compound **21**



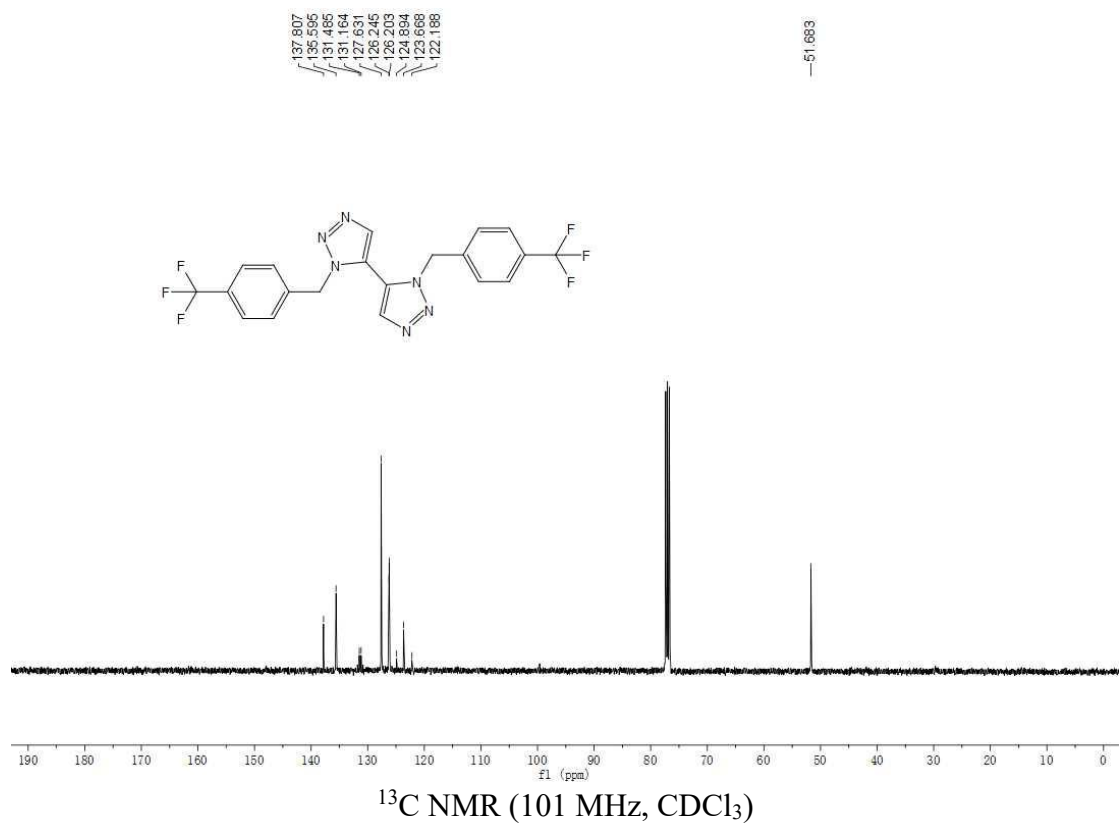
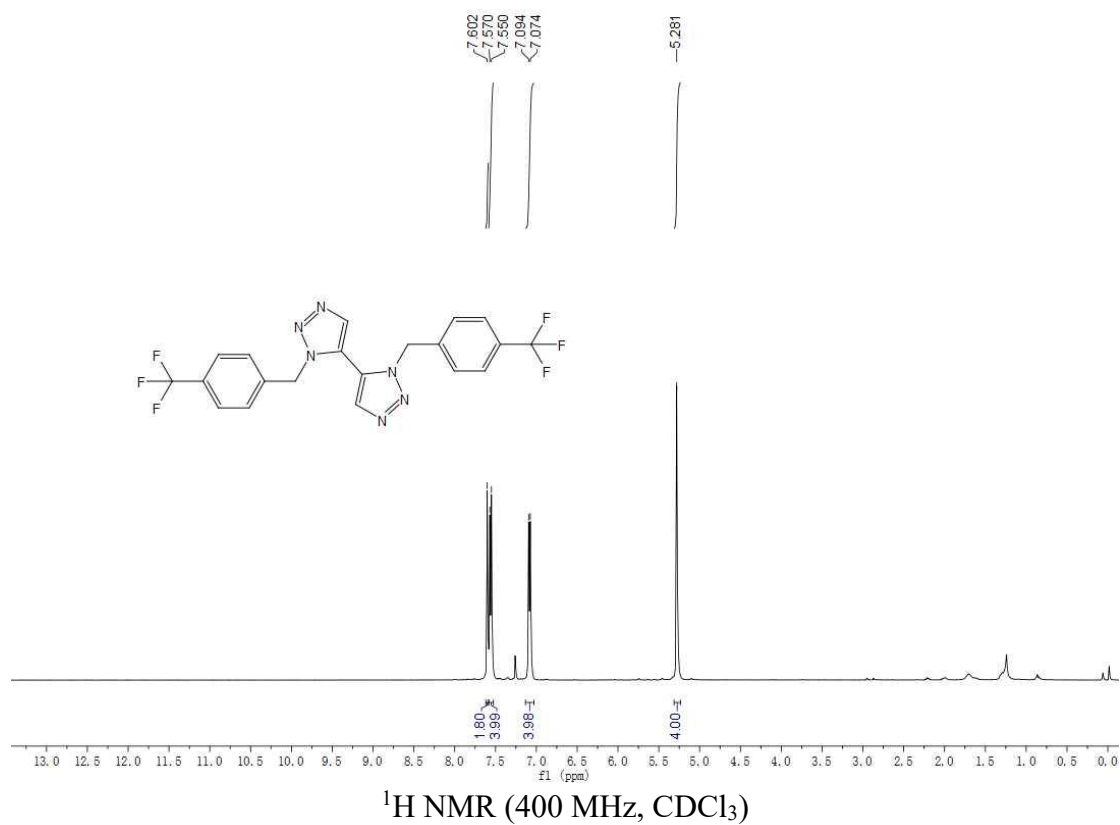
^1H and ^{13}C NMR spectra of compound **2m**



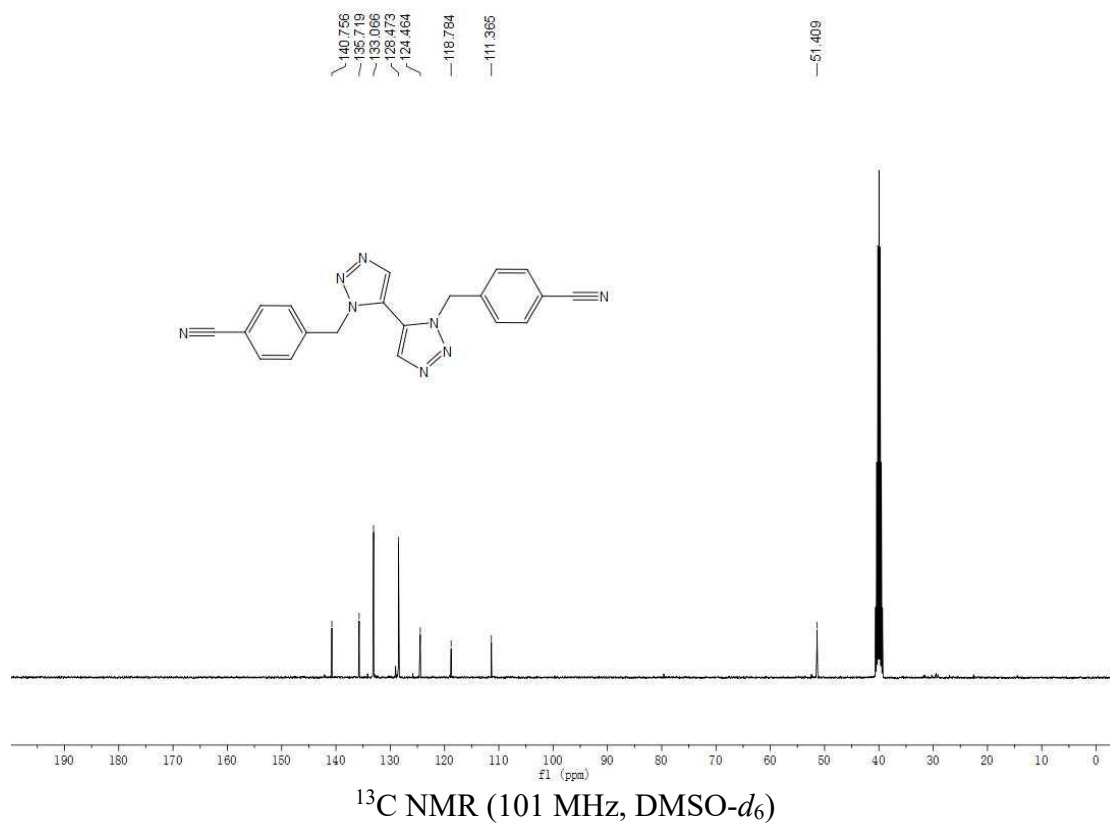
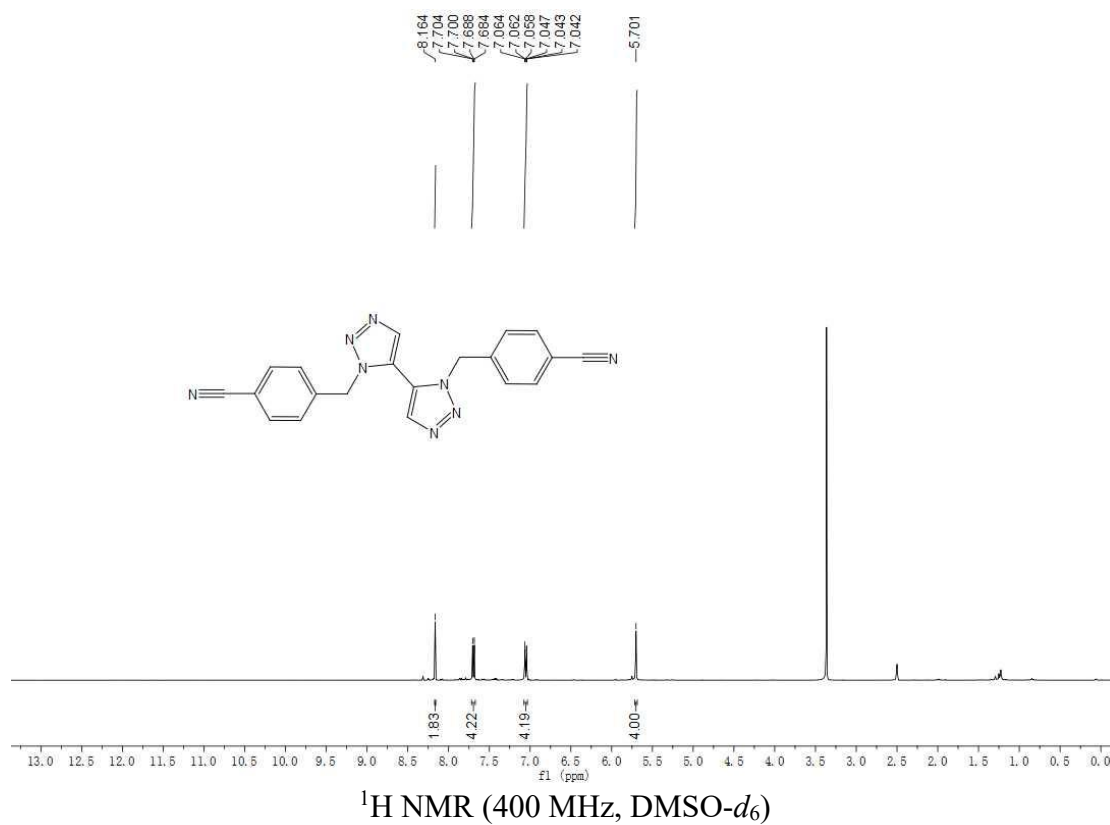
^1H and ^{13}C NMR spectra of compound **2n**



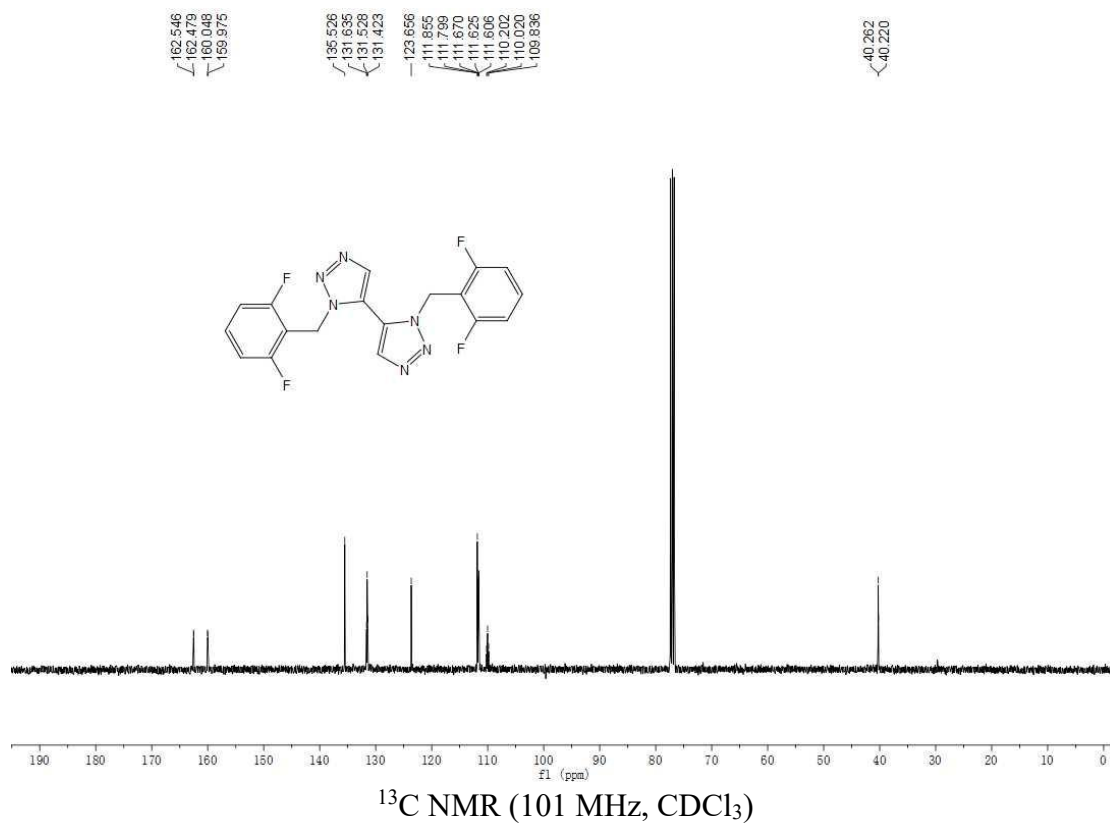
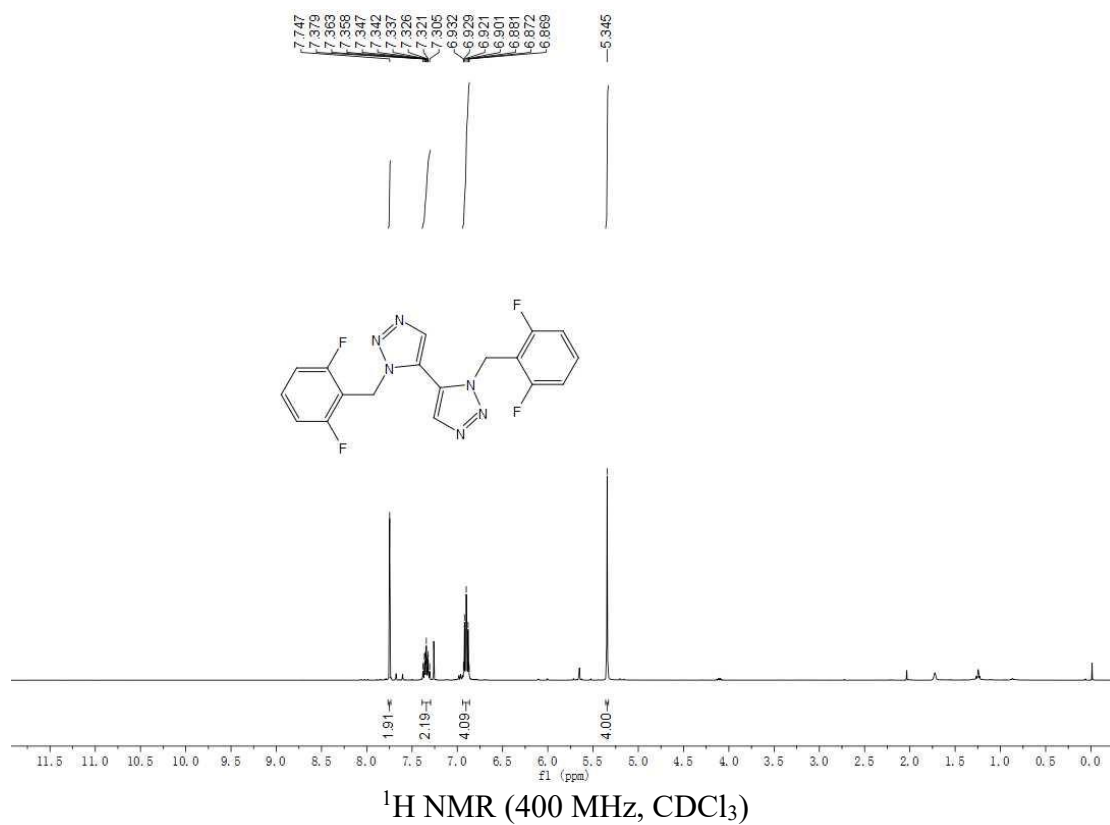
^1H and ^{13}C NMR spectra of compound **2o**



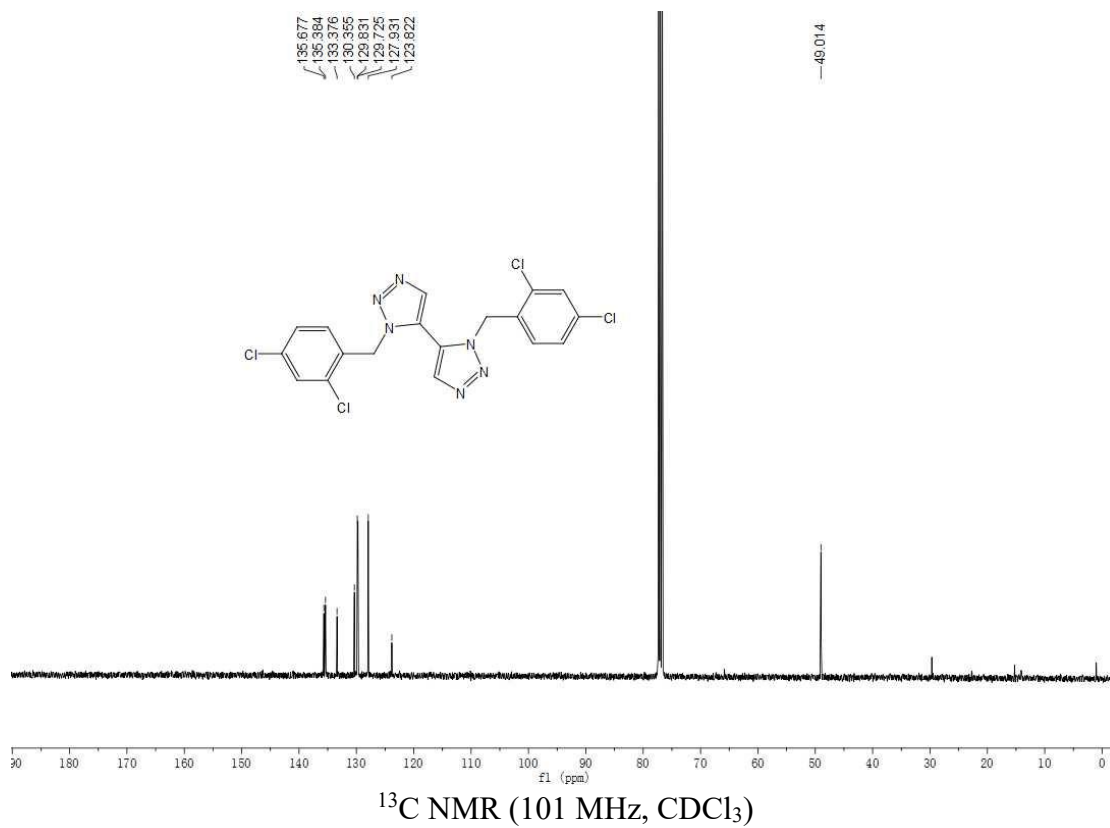
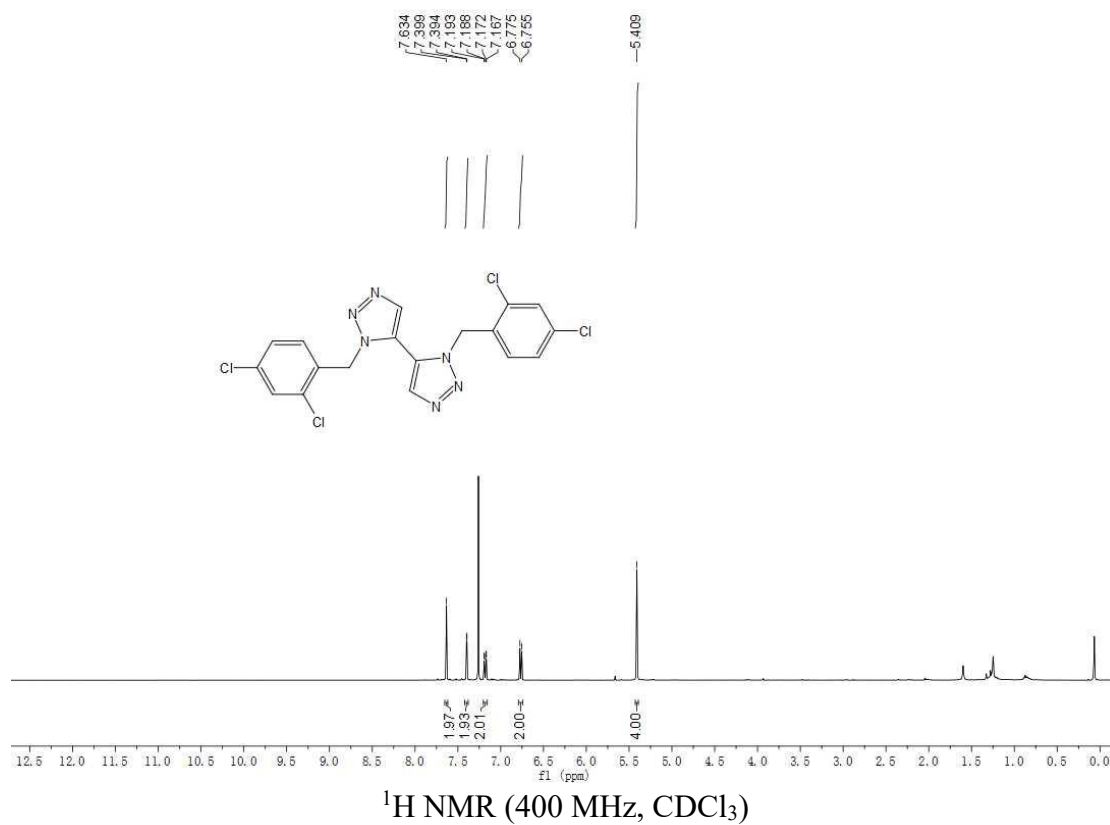
^1H and ^{13}C NMR spectra of compound **2p**



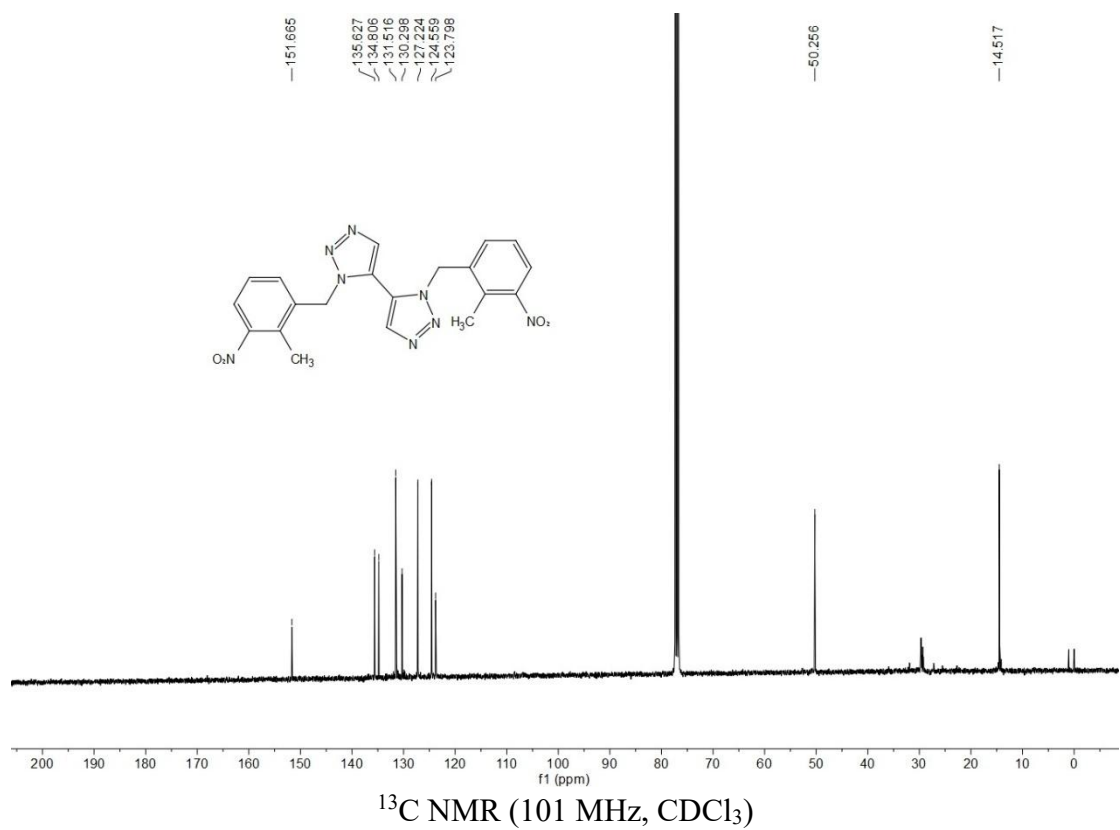
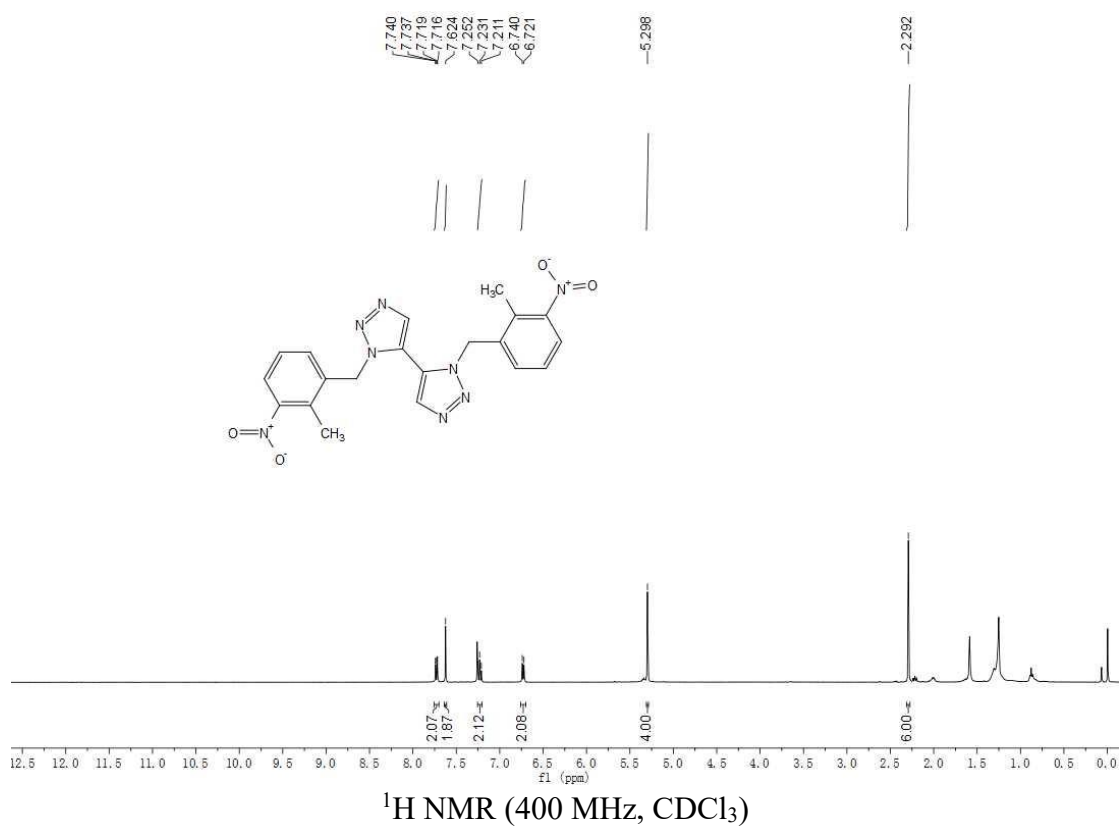
^1H and ^{13}C NMR spectra of compound **2q**



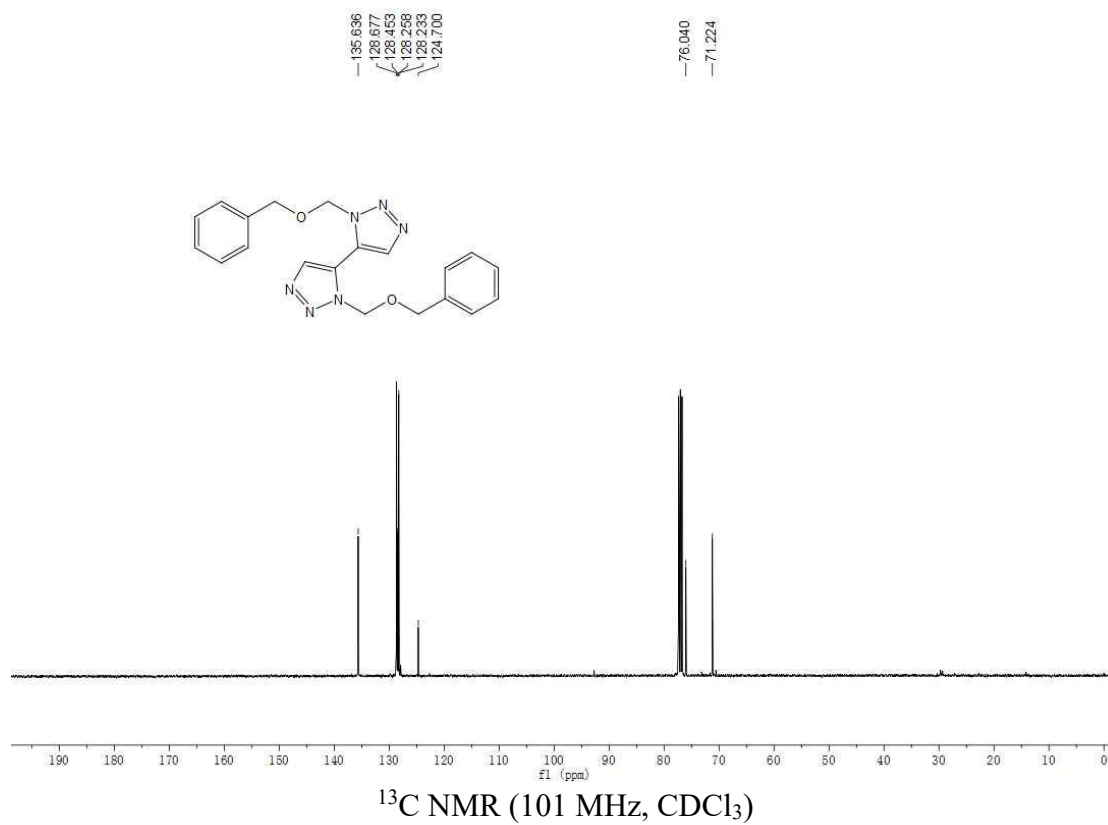
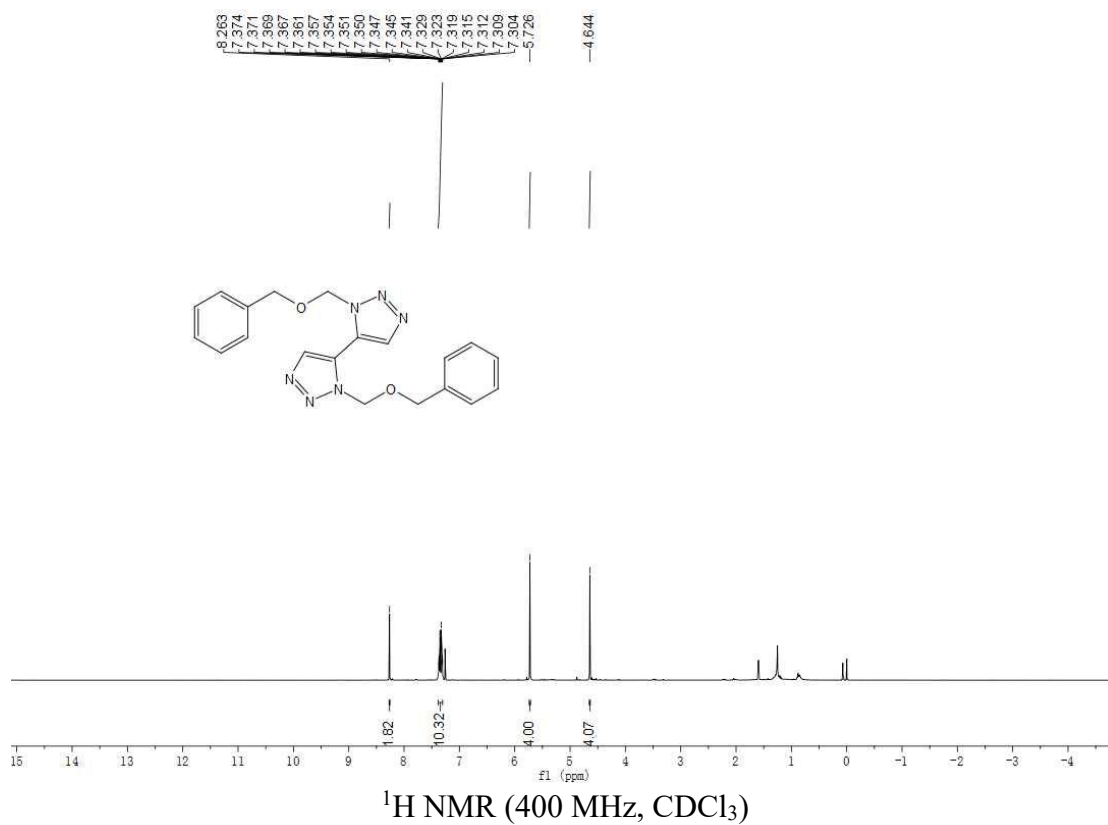
^1H and ^{13}C NMR spectra of compound **2r**



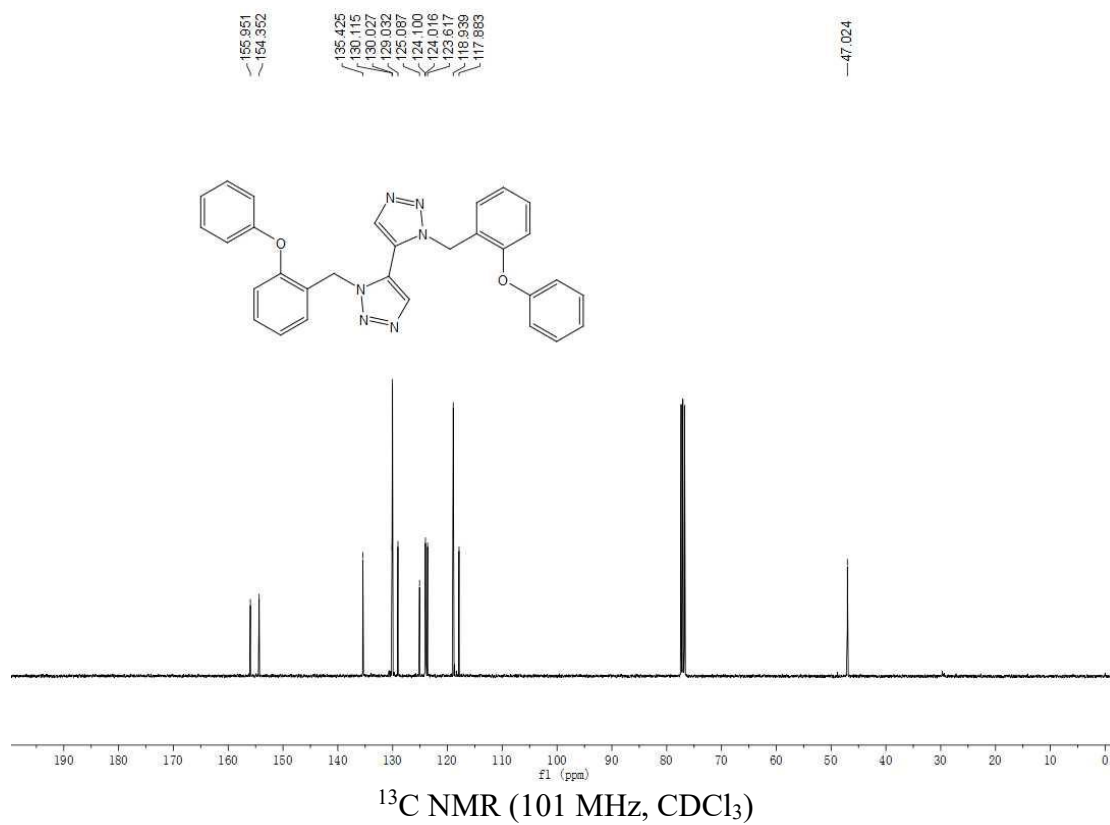
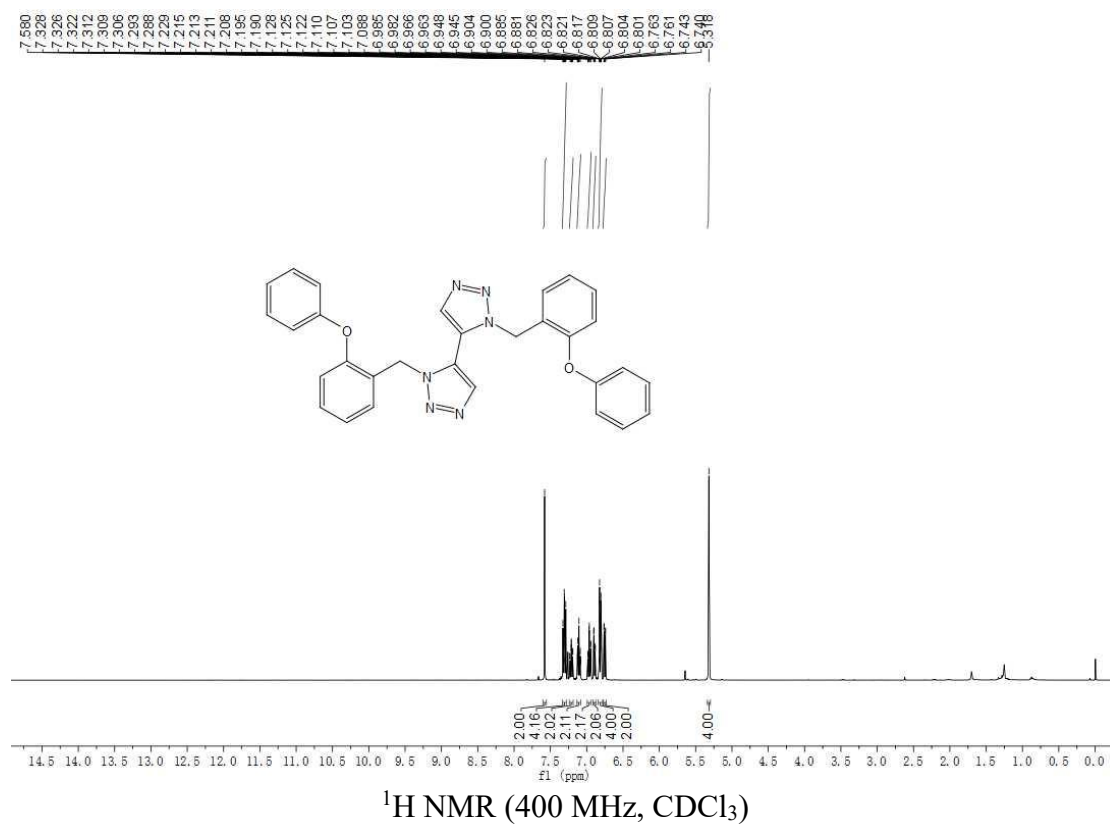
^1H and ^{13}C NMR spectra of compound **2s**



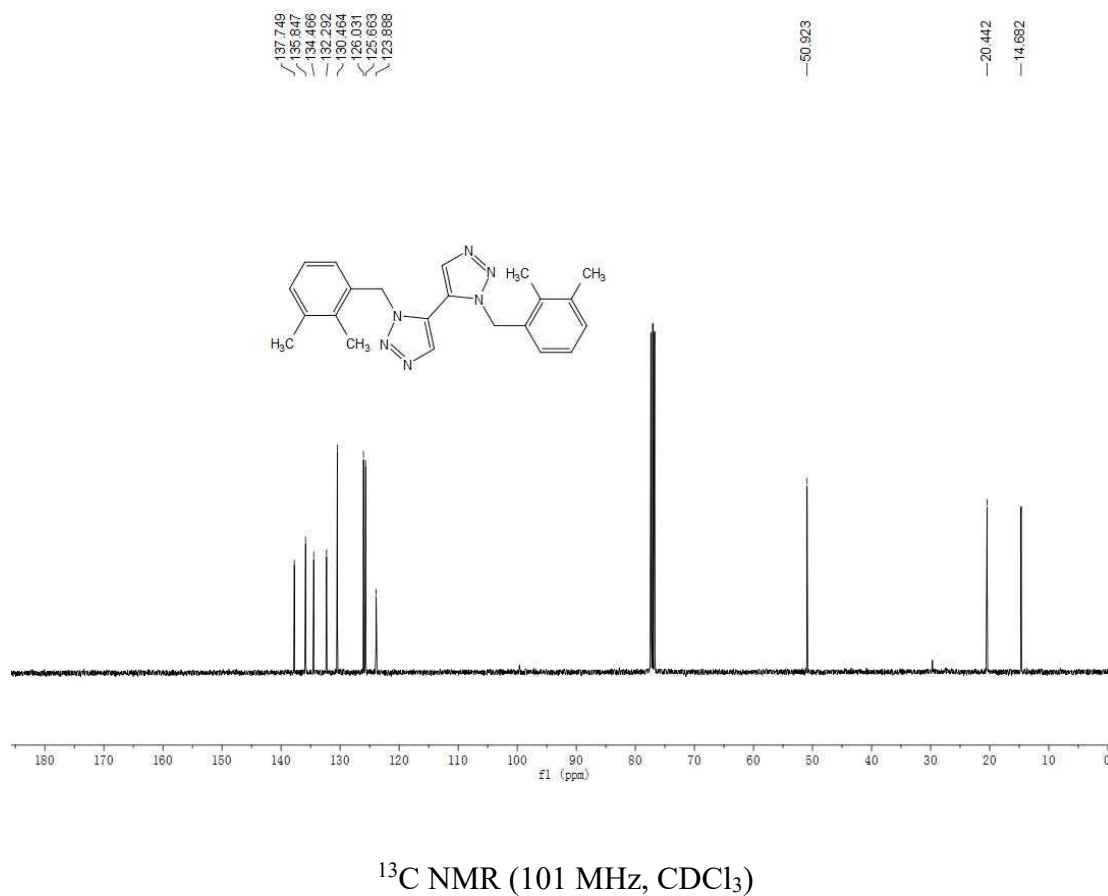
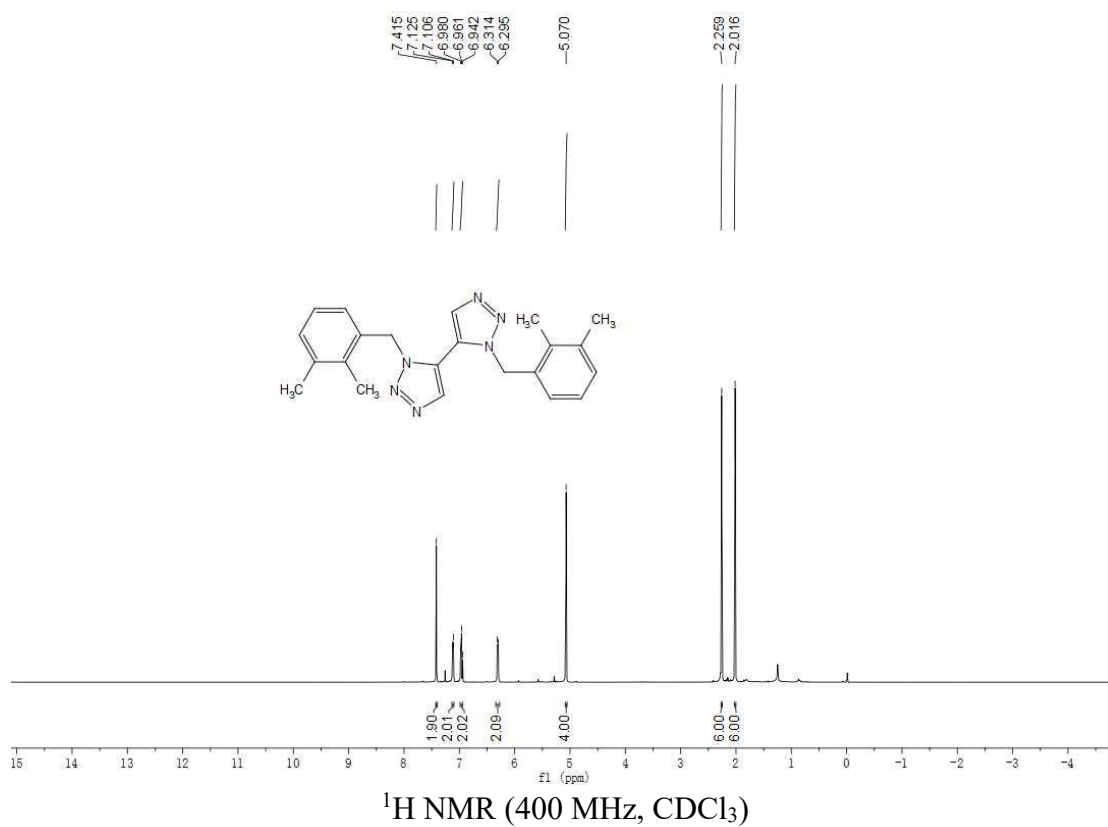
^1H and ^{13}C NMR spectra of compound **2t**



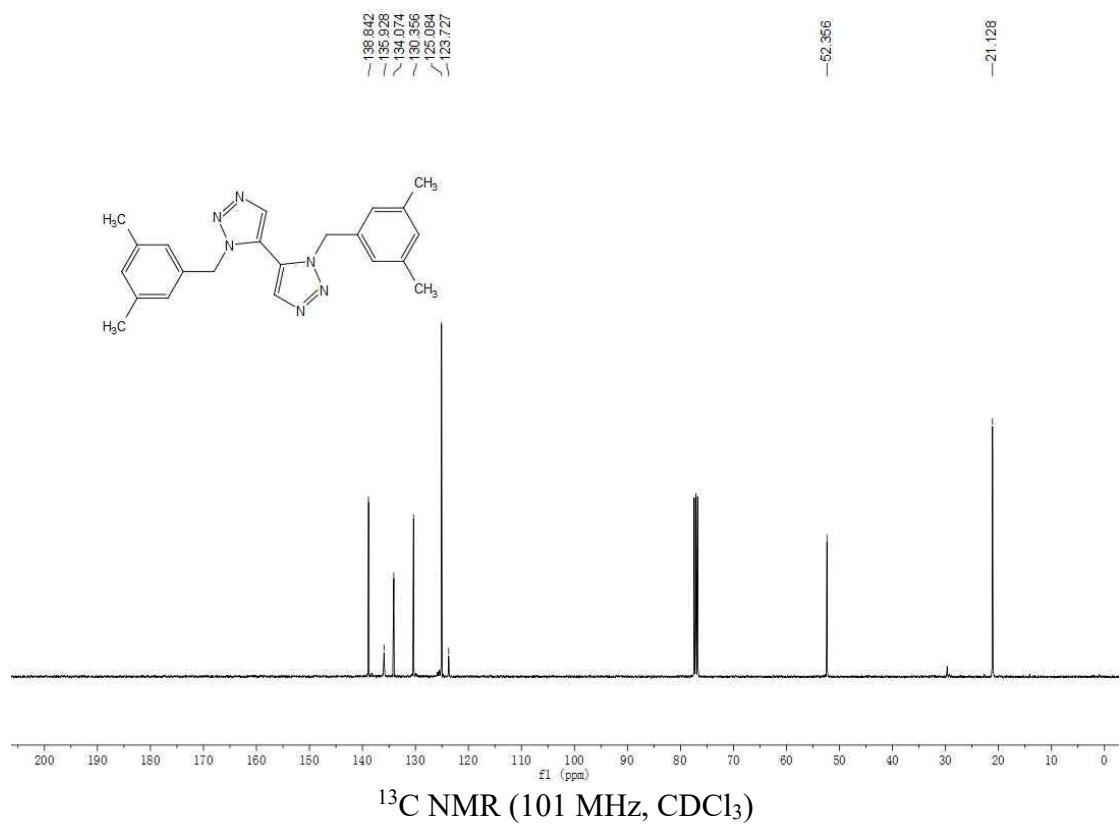
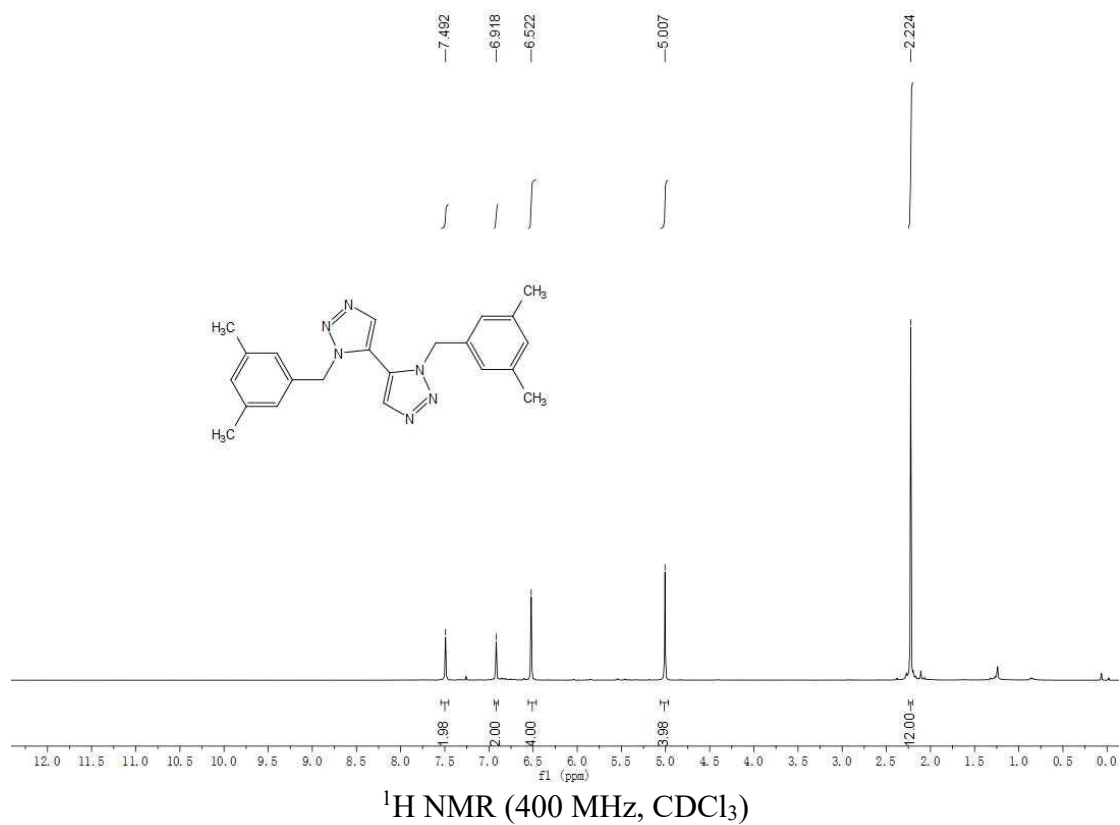
^1H and ^{13}C NMR spectra of compound **2u**



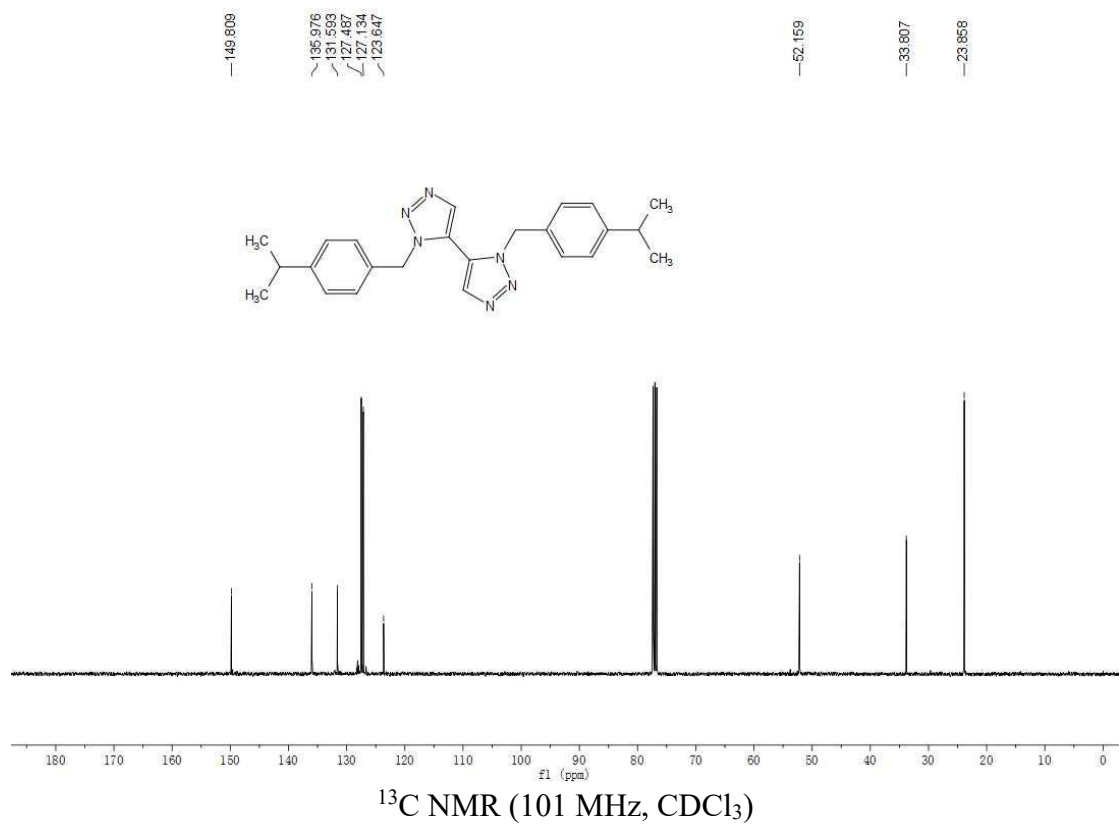
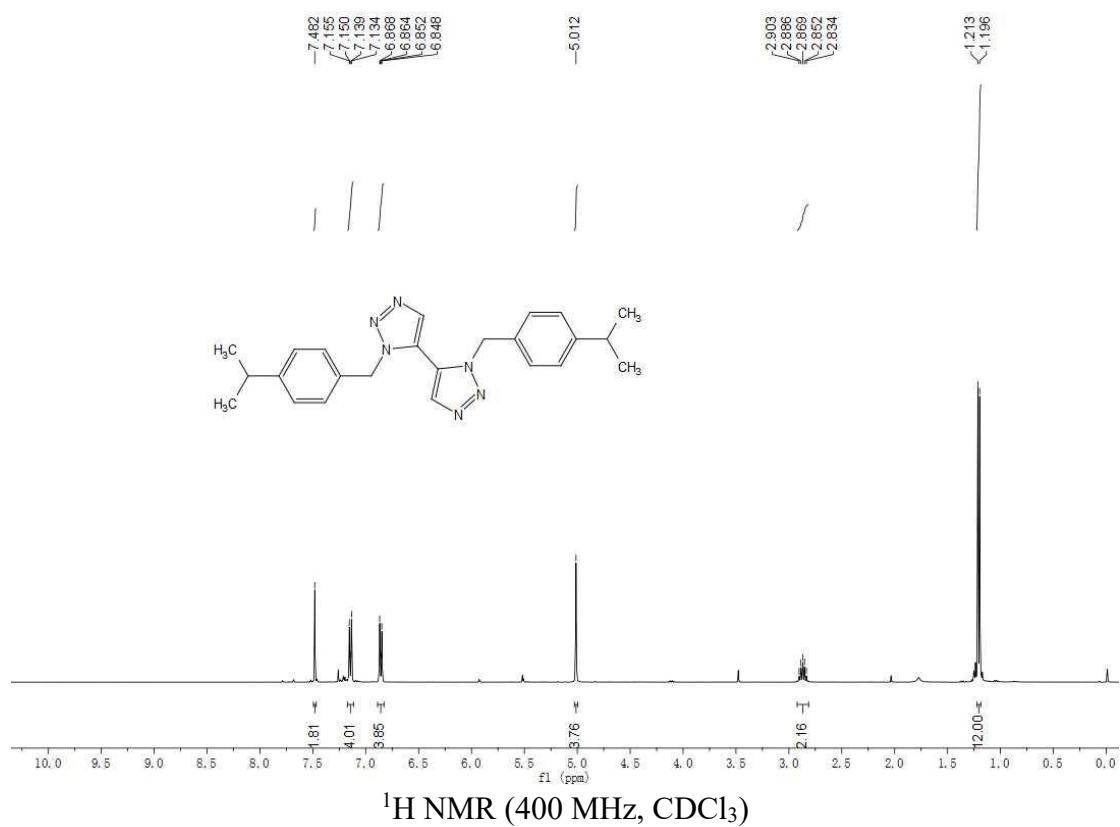
^1H and ^{13}C NMR spectra of compound **2v**



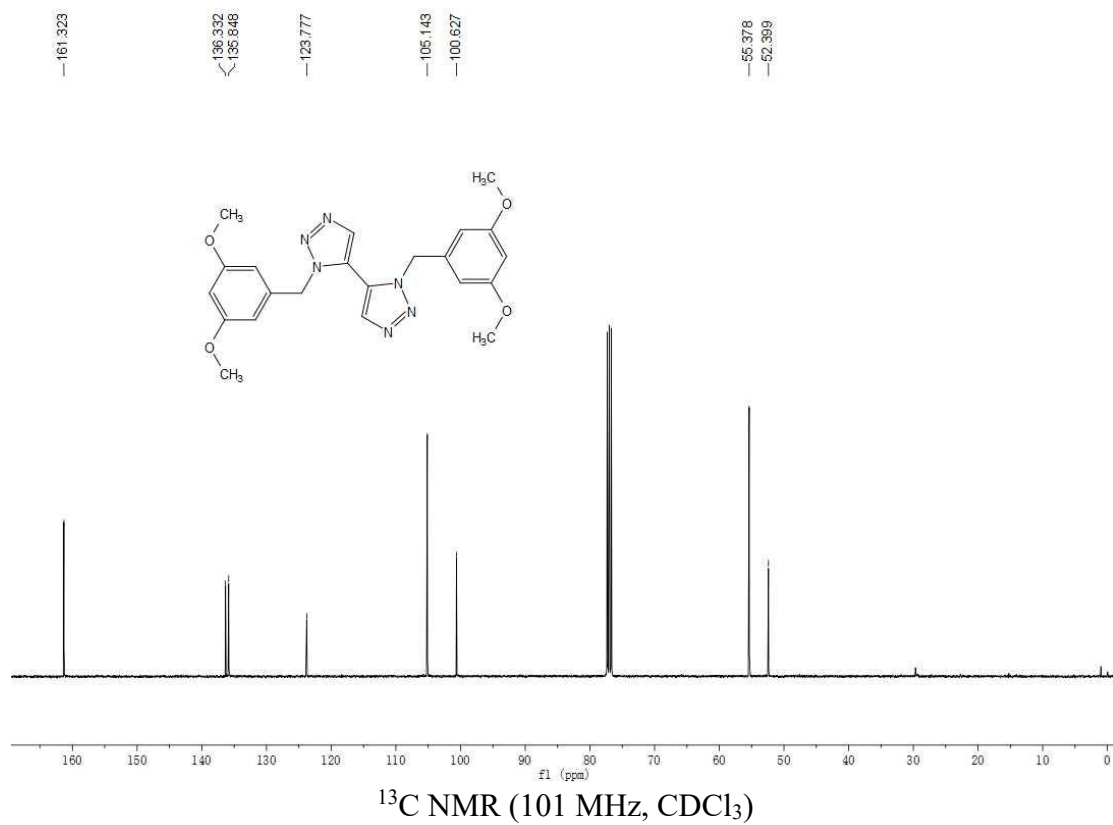
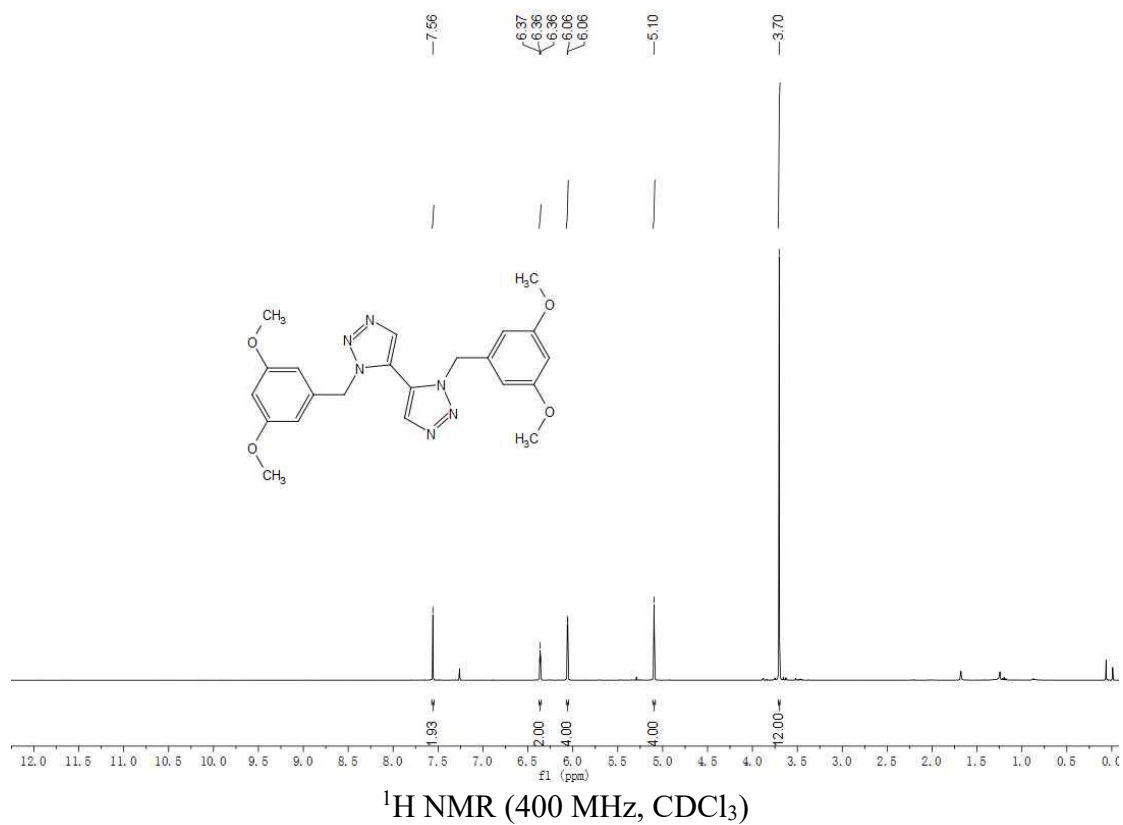
^1H and ^{13}C NMR spectra of compound **2w**



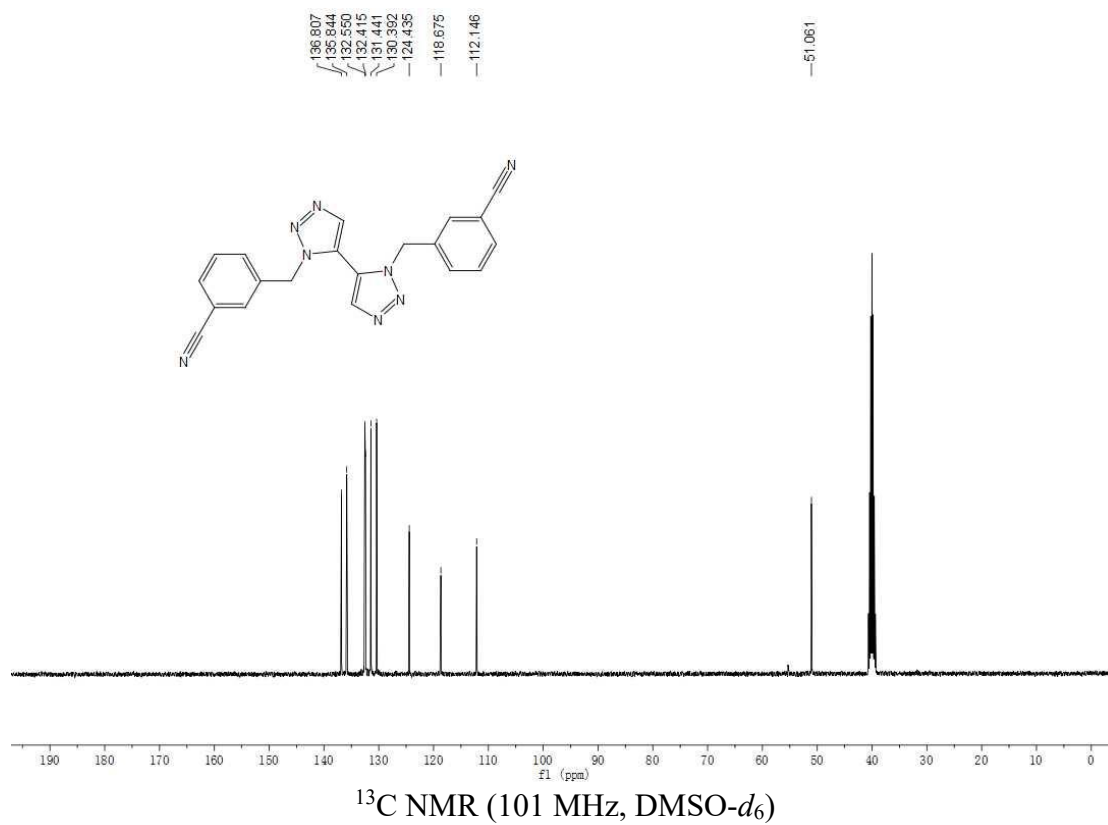
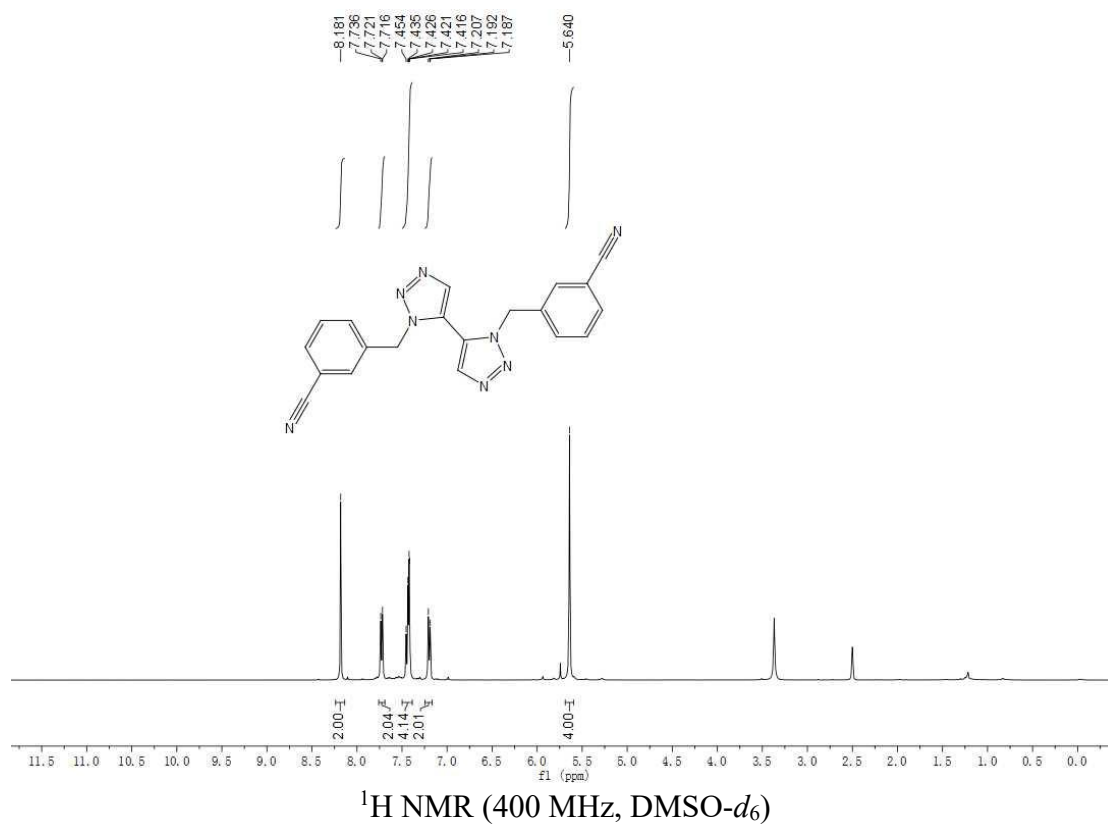
^1H and ^{13}C NMR spectra of compound **2x**



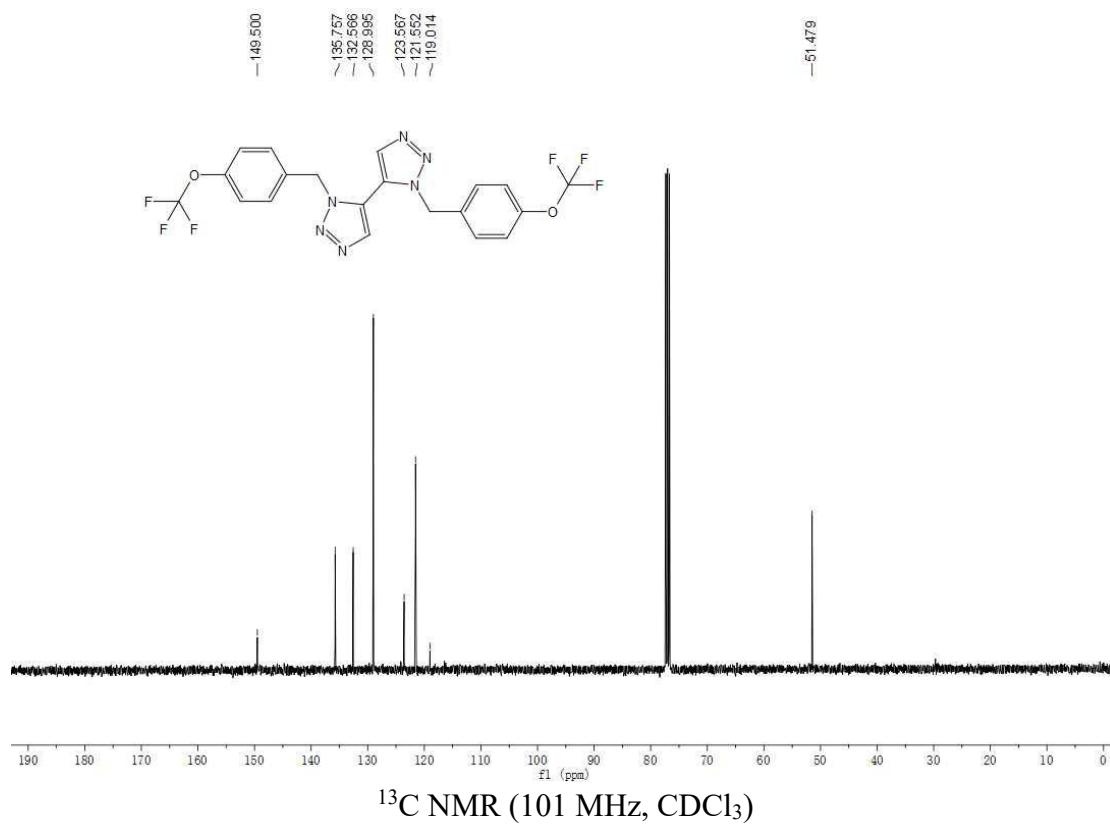
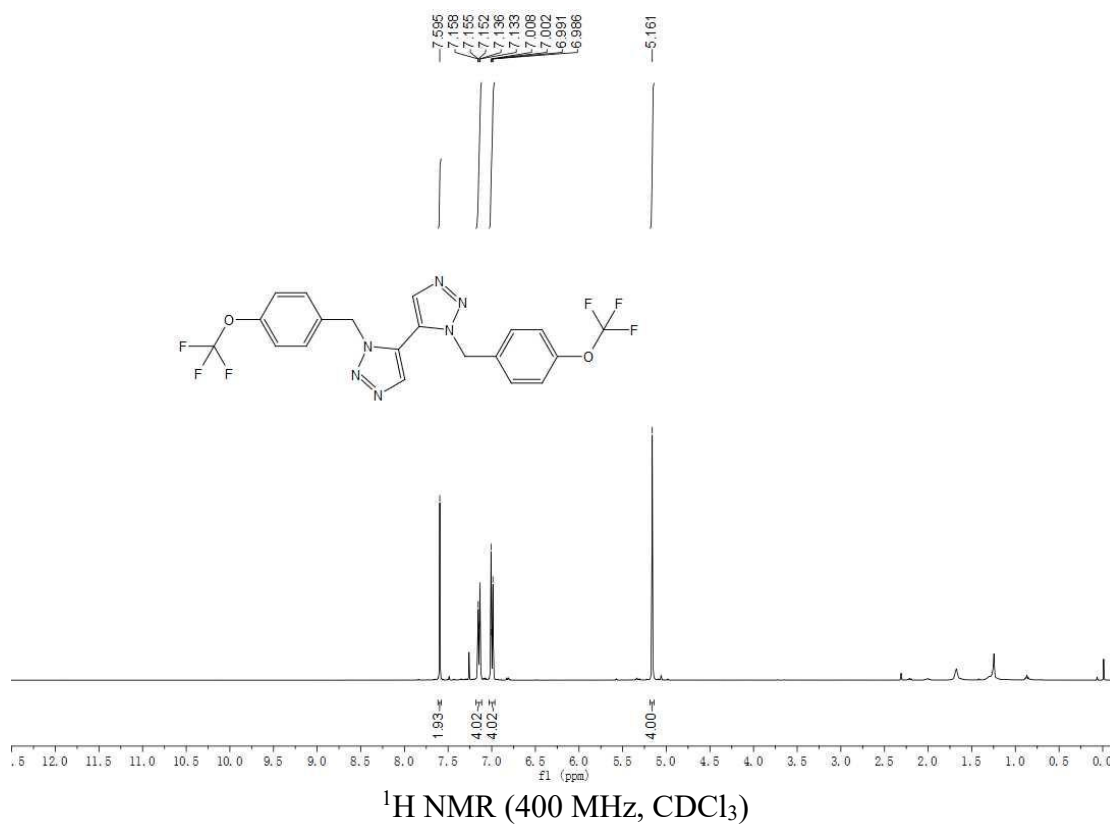
^1H and ^{13}C NMR spectra of compound **2y**



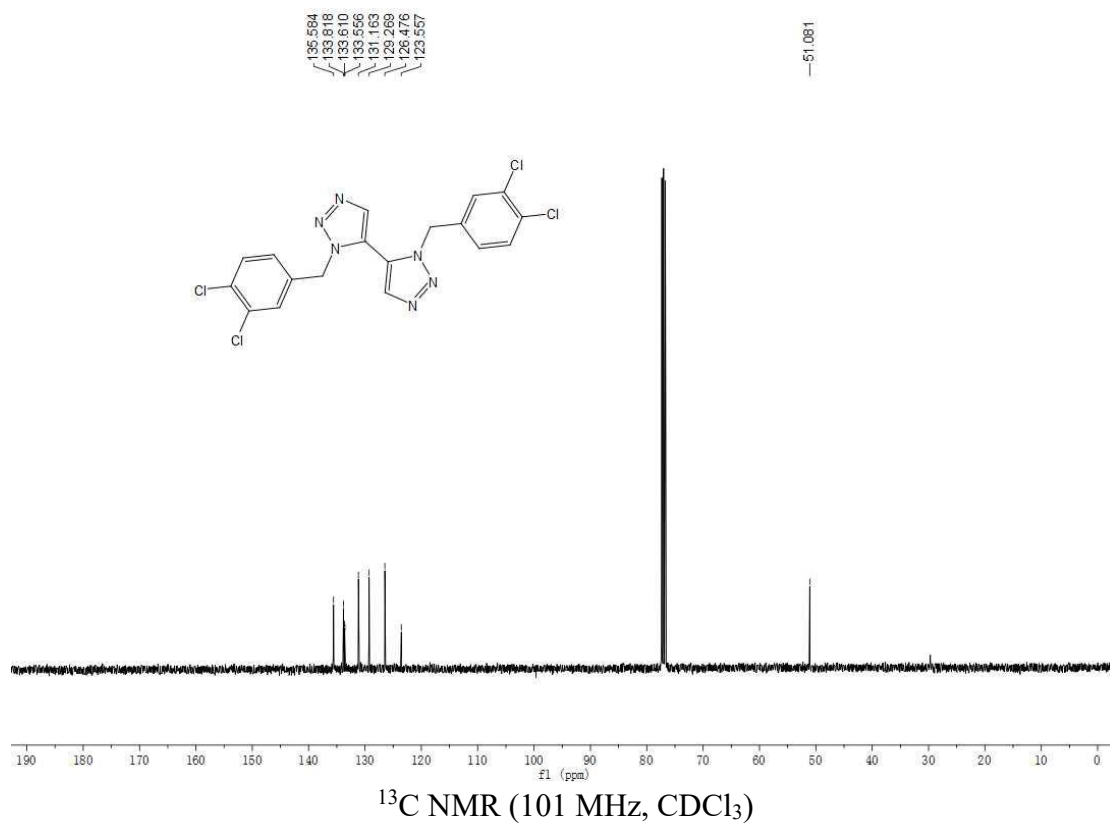
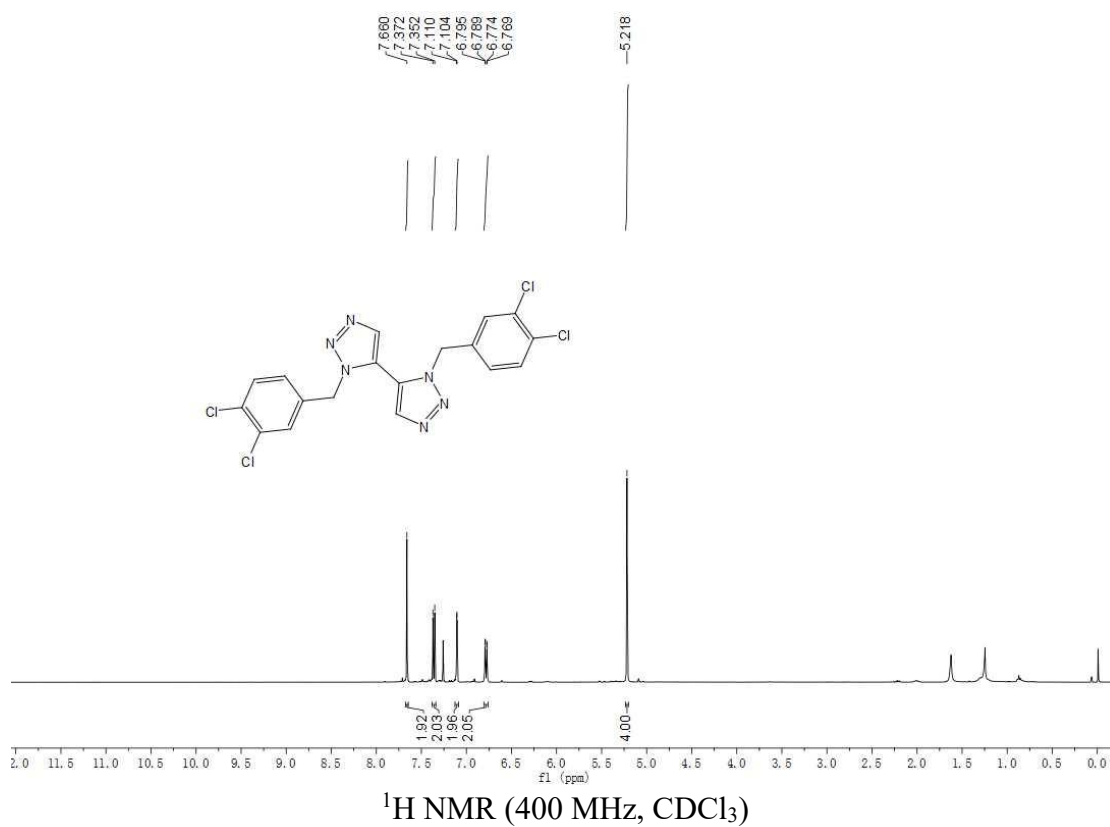
^1H and ^{13}C NMR spectra of compound **2z**



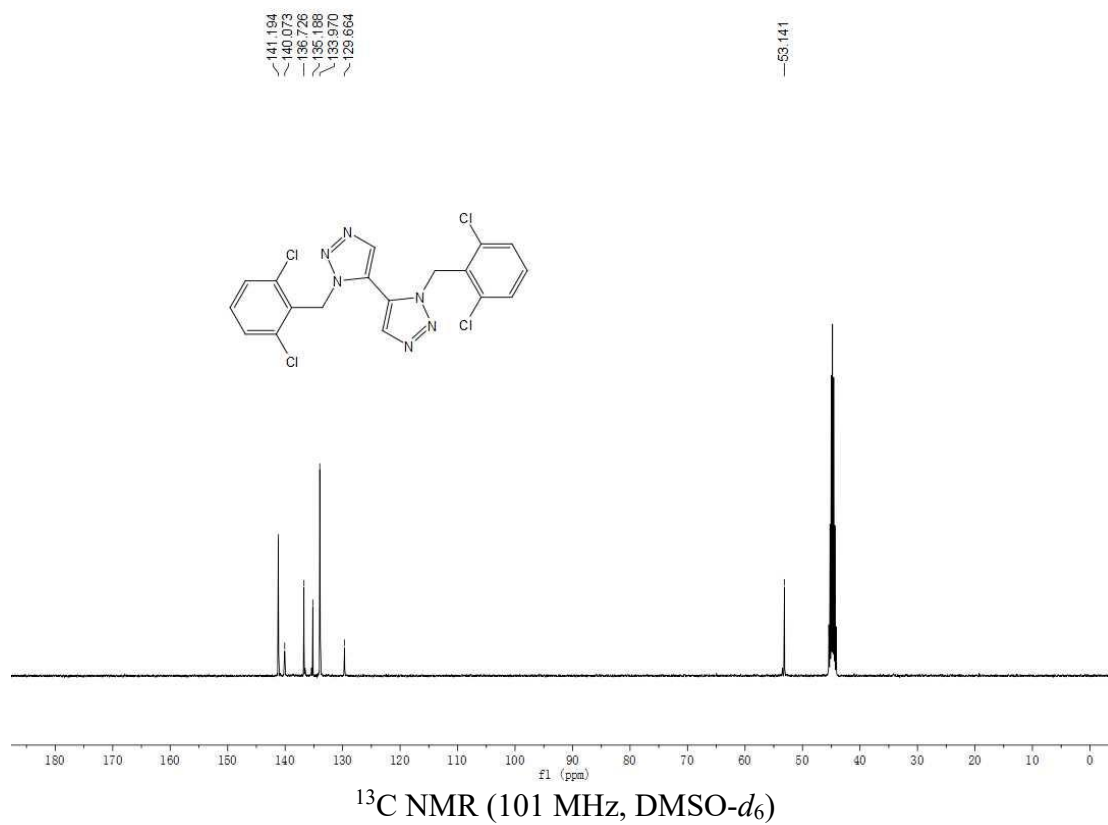
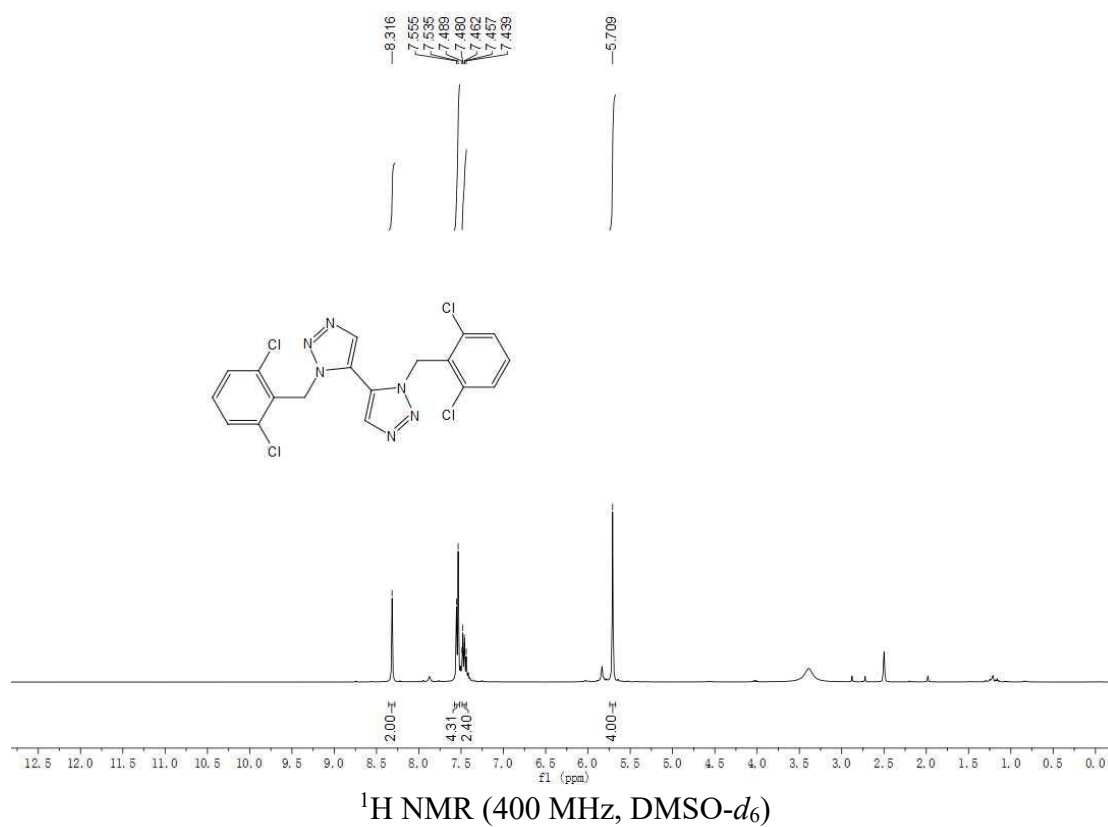
^1H and ^{13}C NMR spectra of compound **2aa**



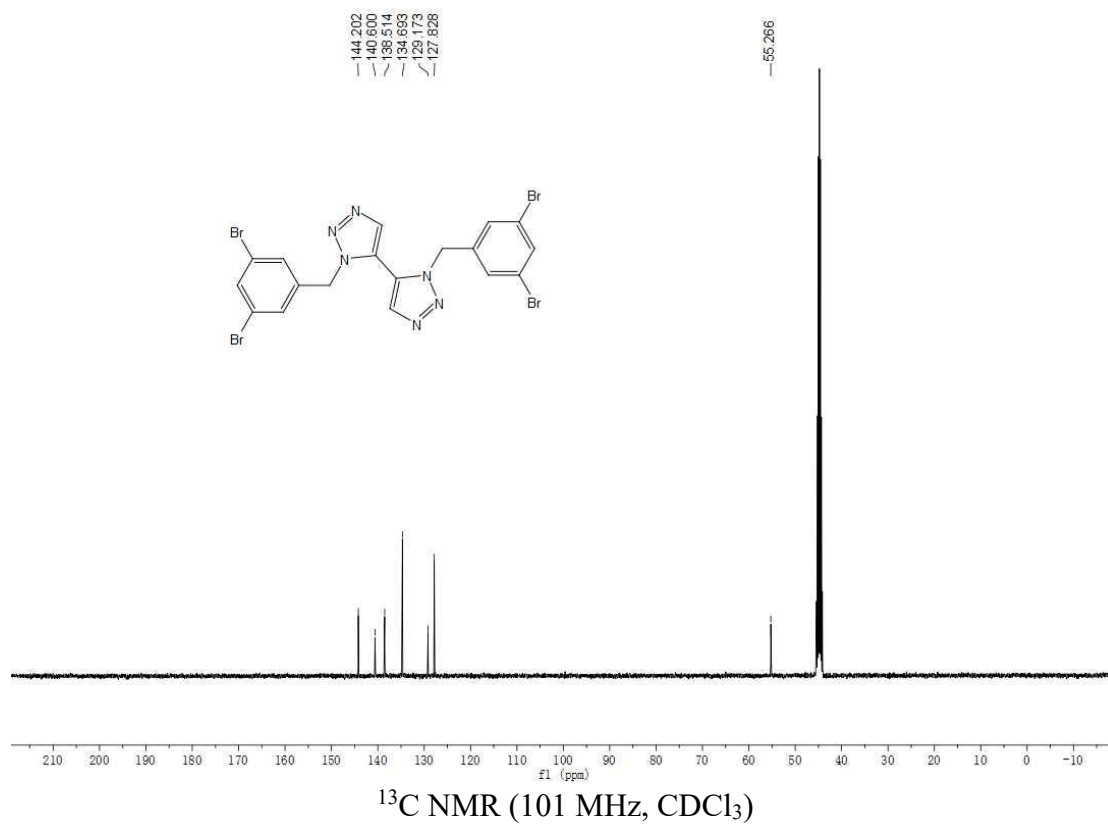
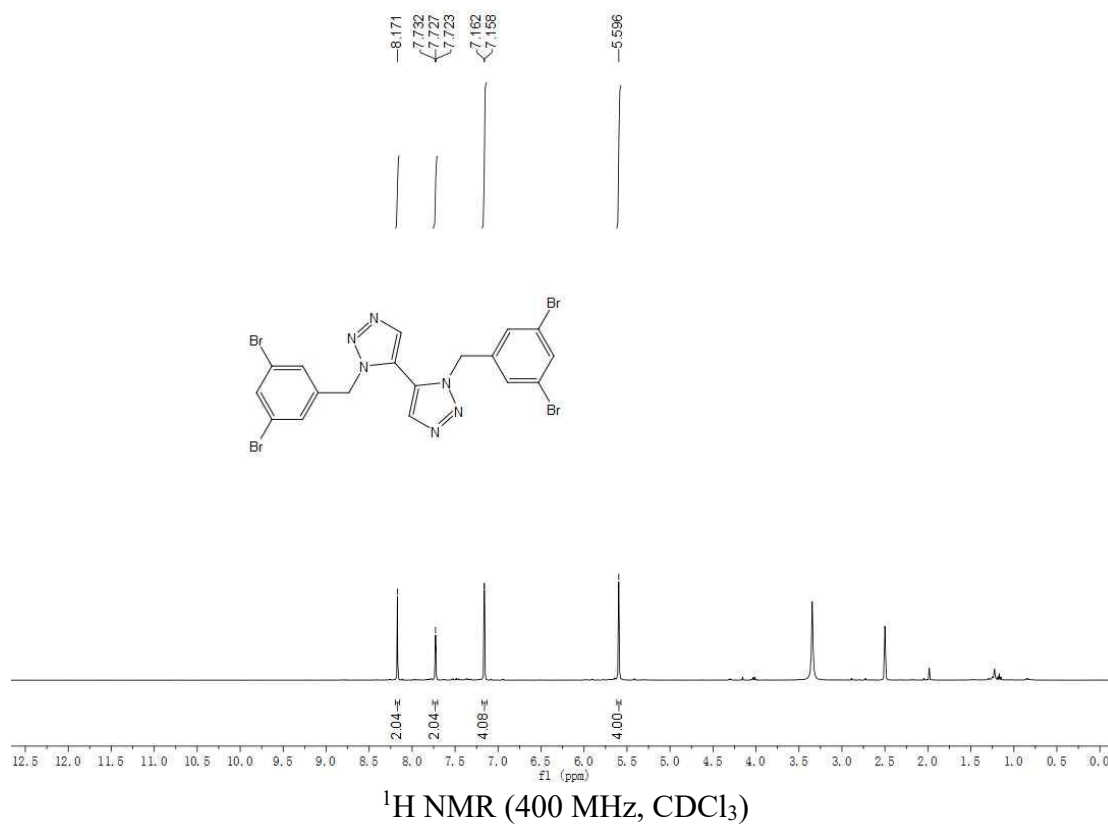
^1H and ^{13}C NMR spectra of compound **2ab**



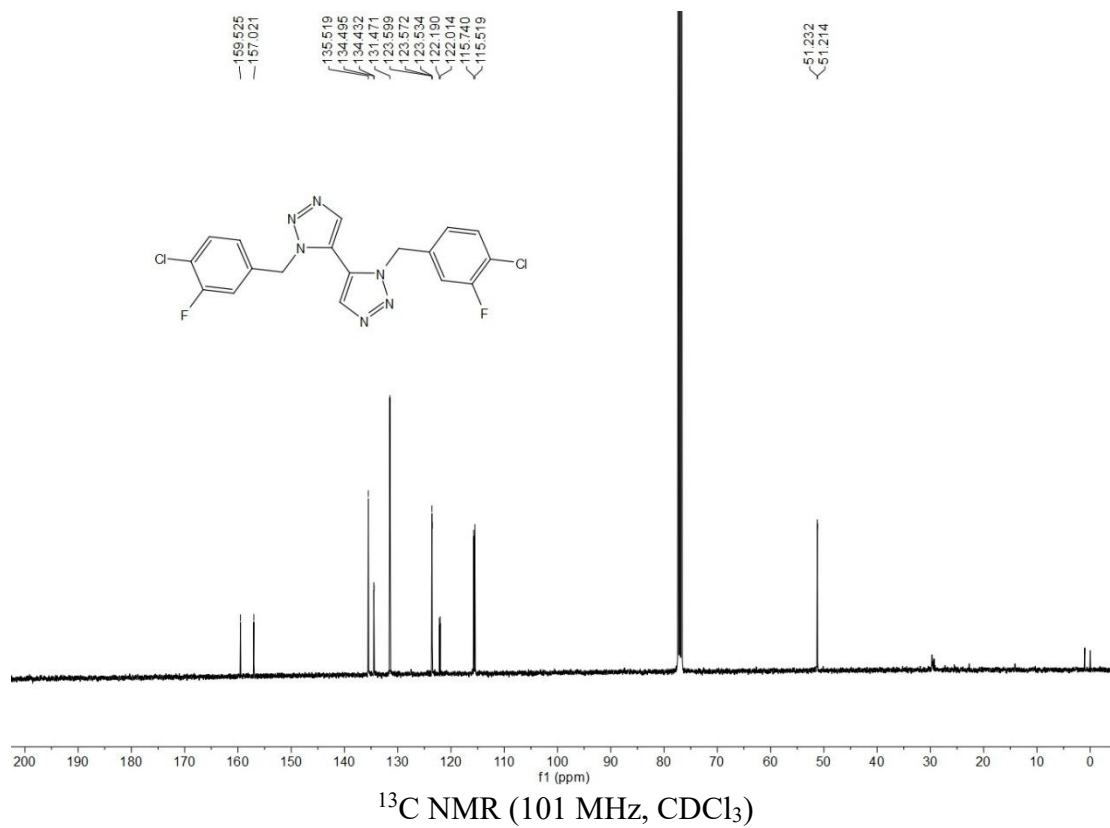
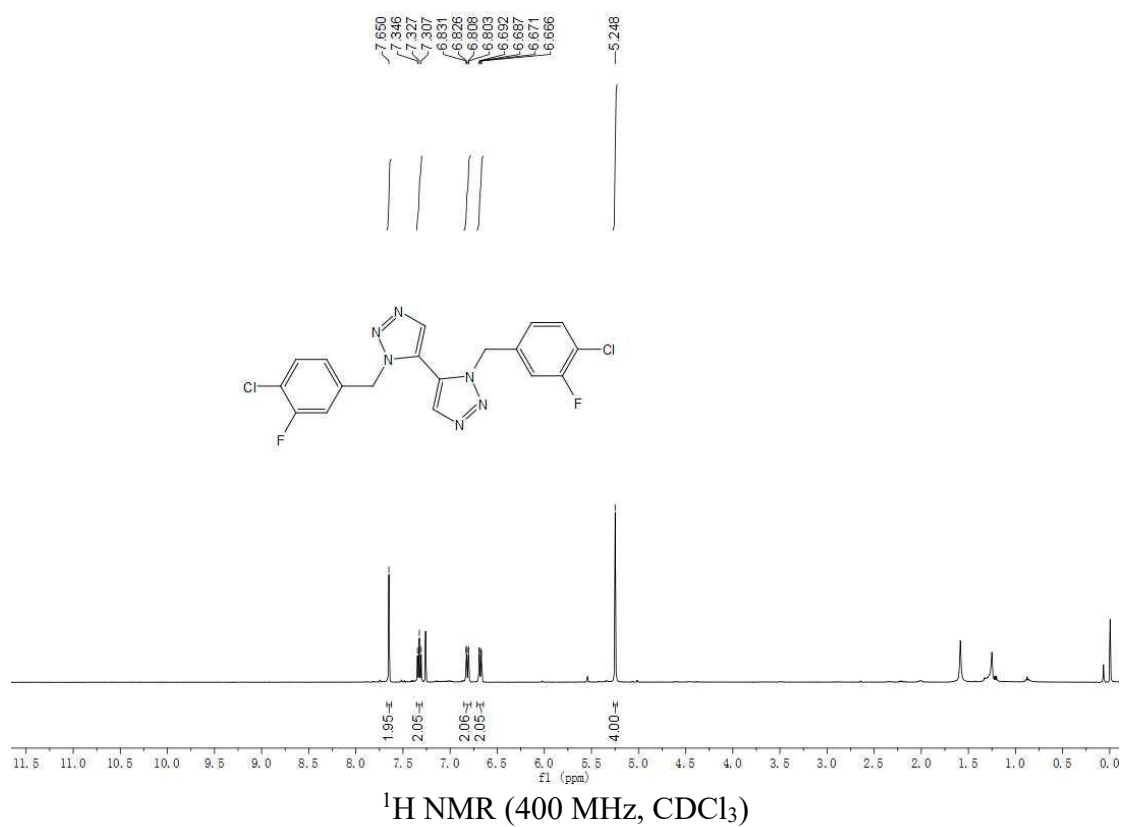
^1H and ^{13}C NMR spectra of compound **2ac**



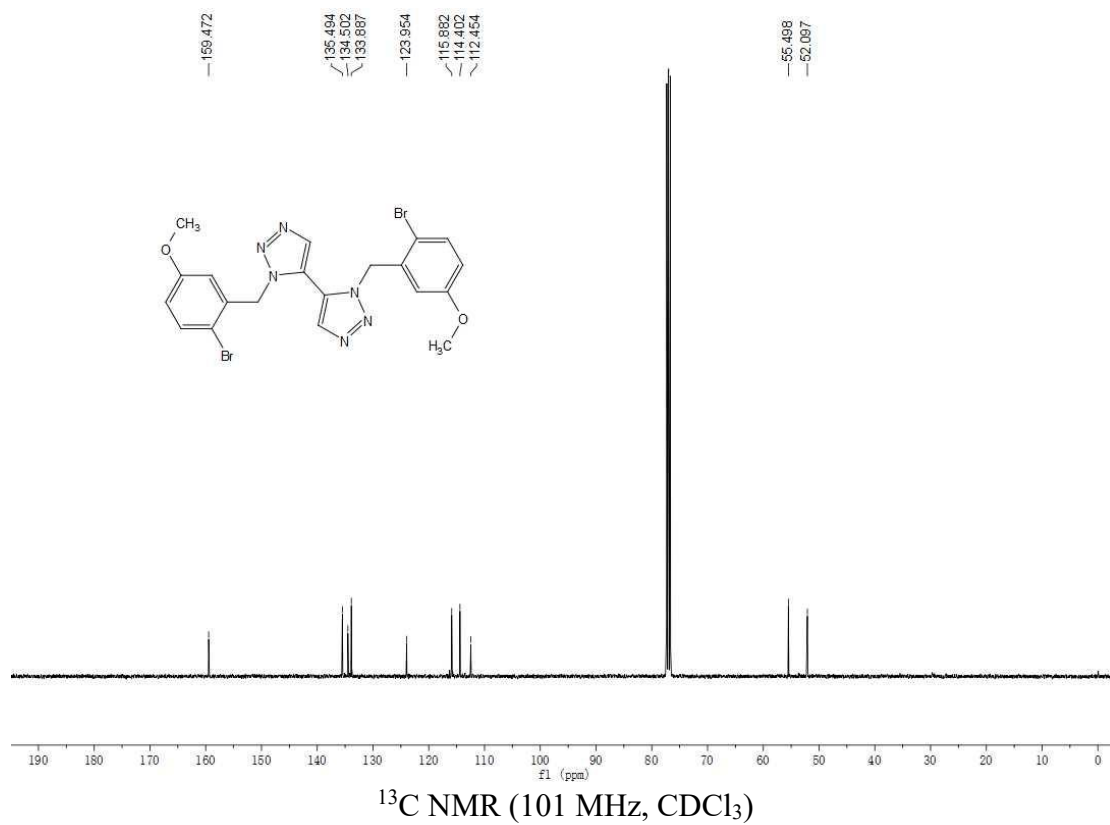
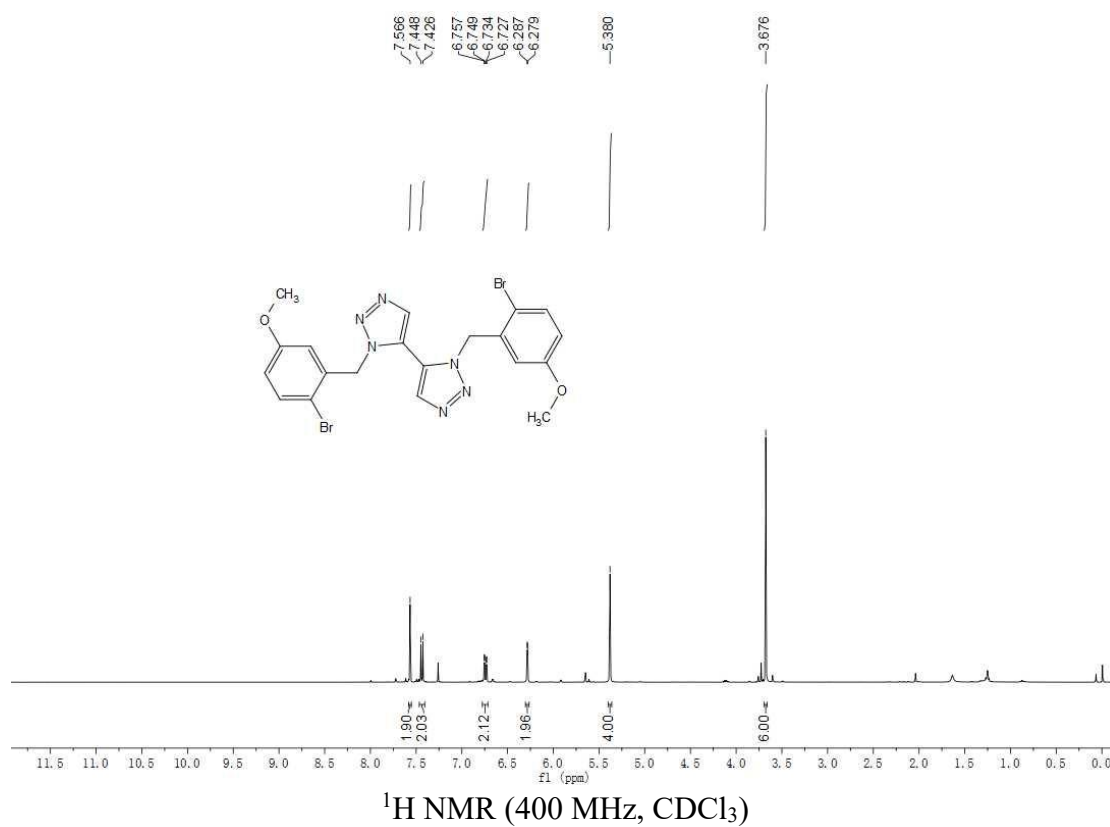
^1H and ^{13}C NMR spectra of compound **2ad**



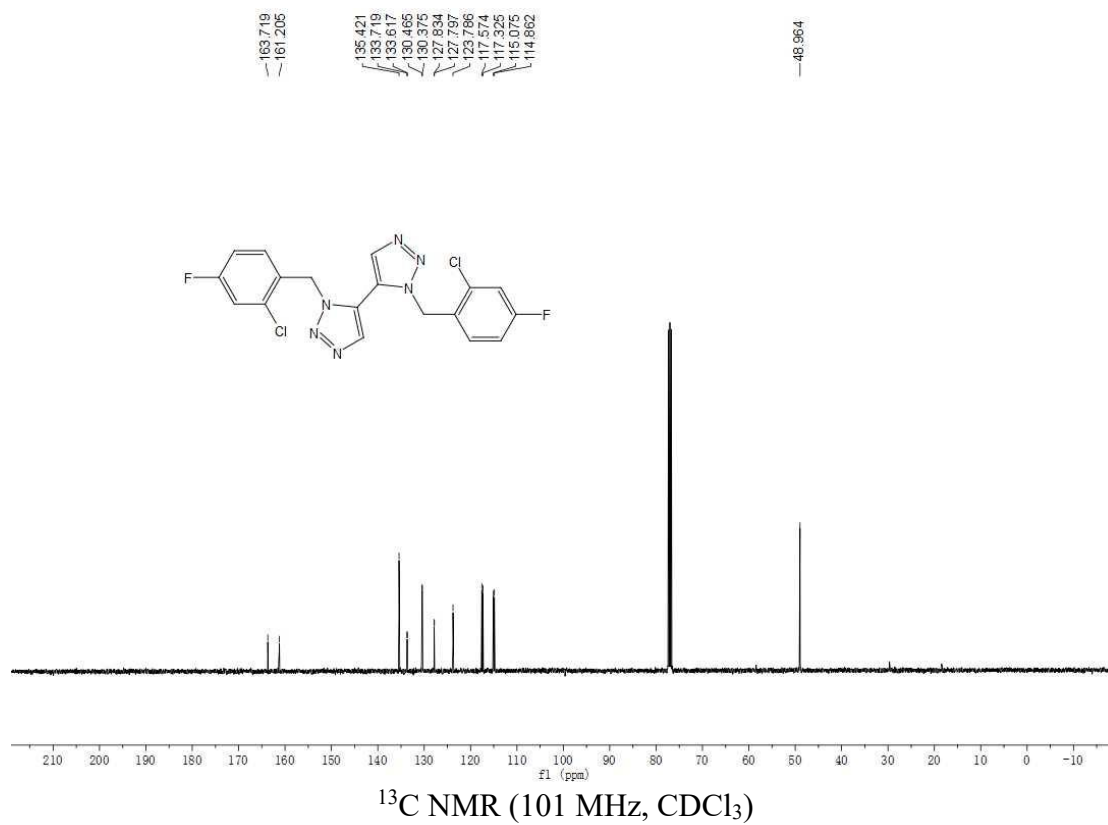
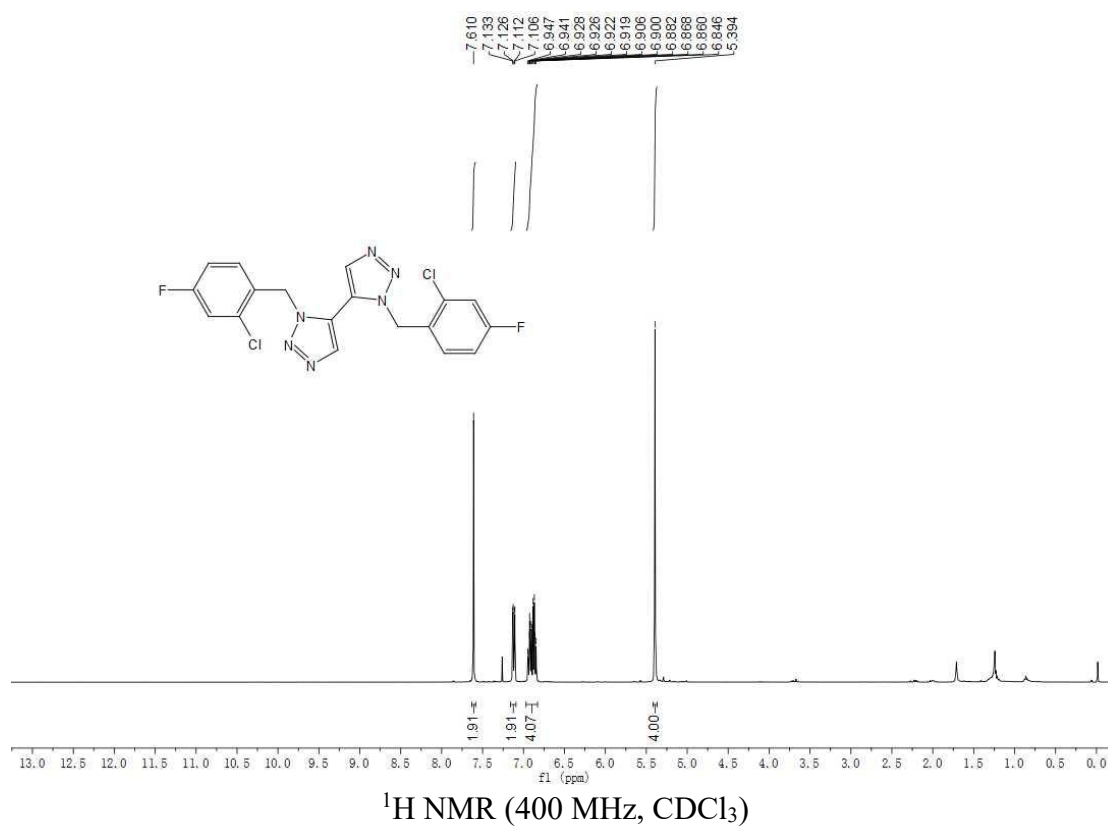
^1H and ^{13}C NMR spectra of compound **2ae**



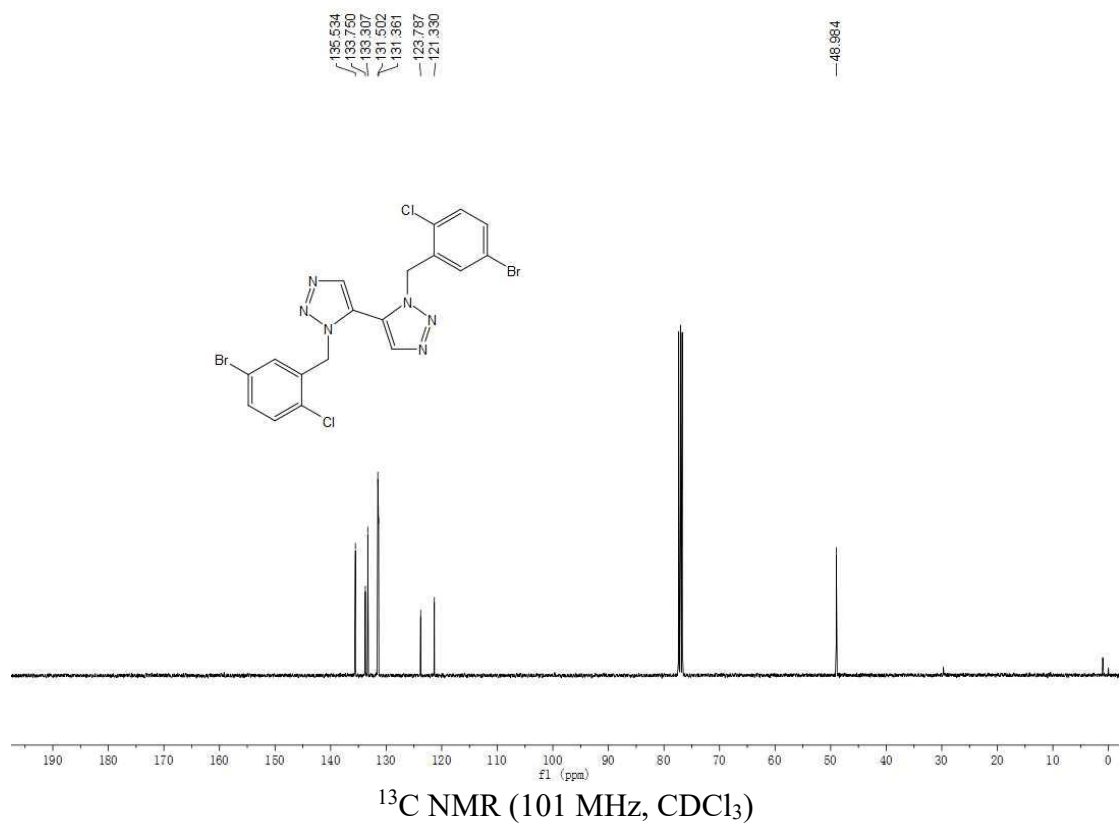
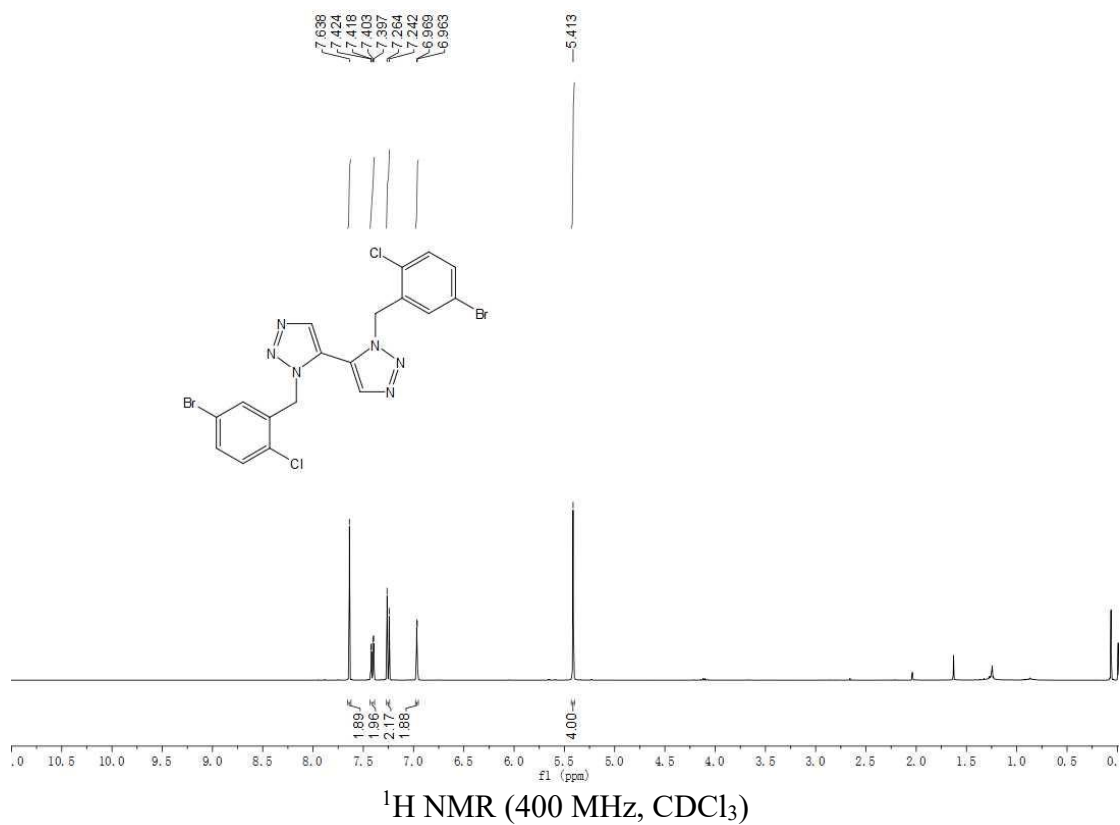
^1H and ^{13}C NMR spectra of compound **2af**



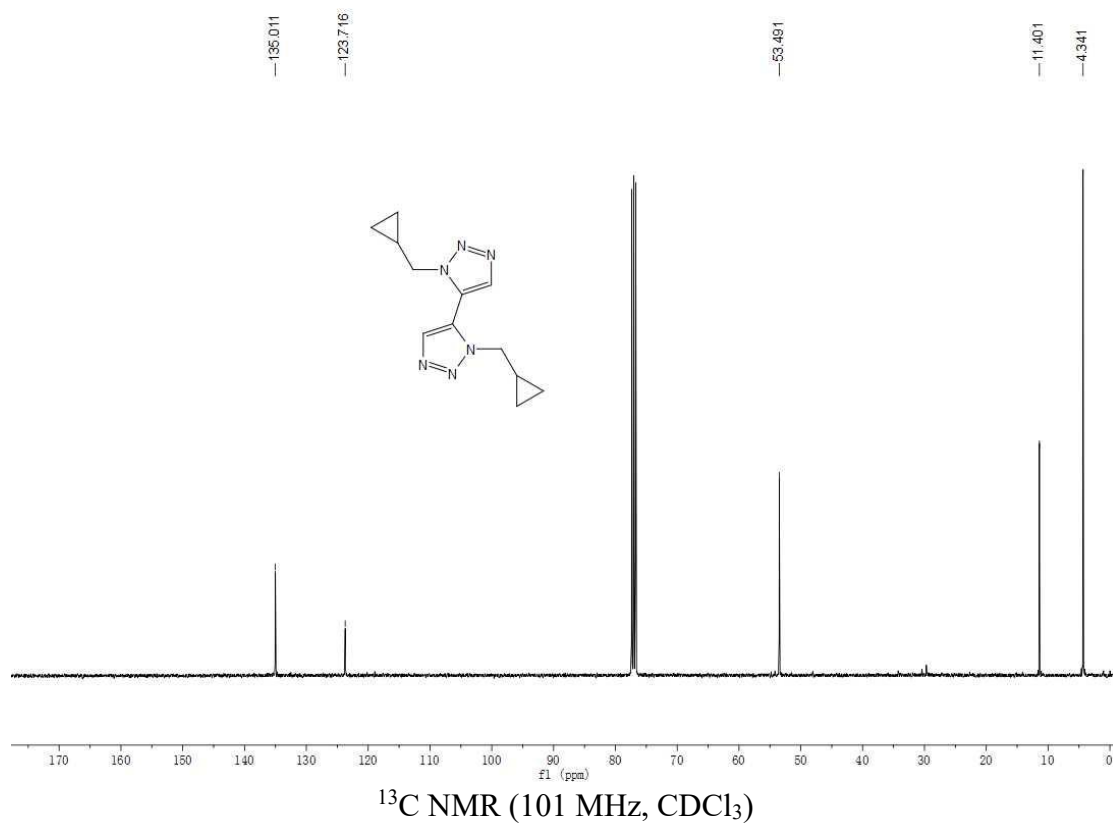
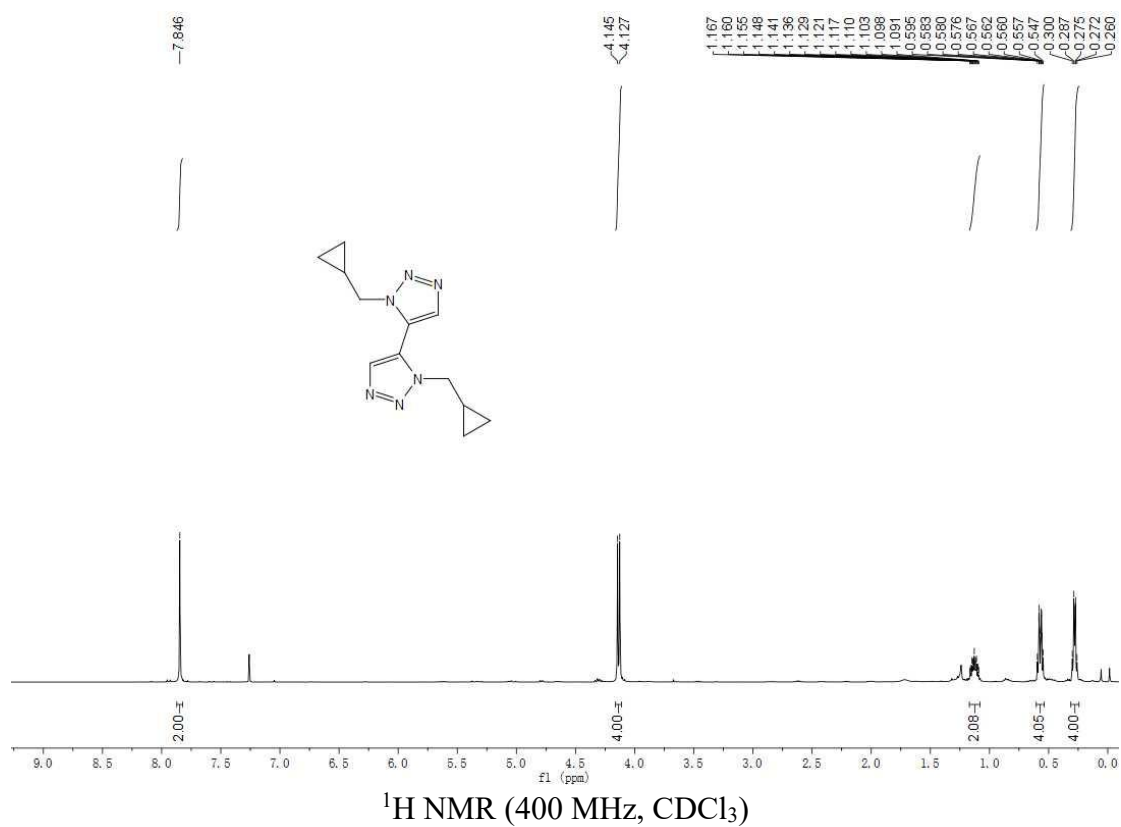
^1H and ^{13}C NMR spectra of compound **2ag**



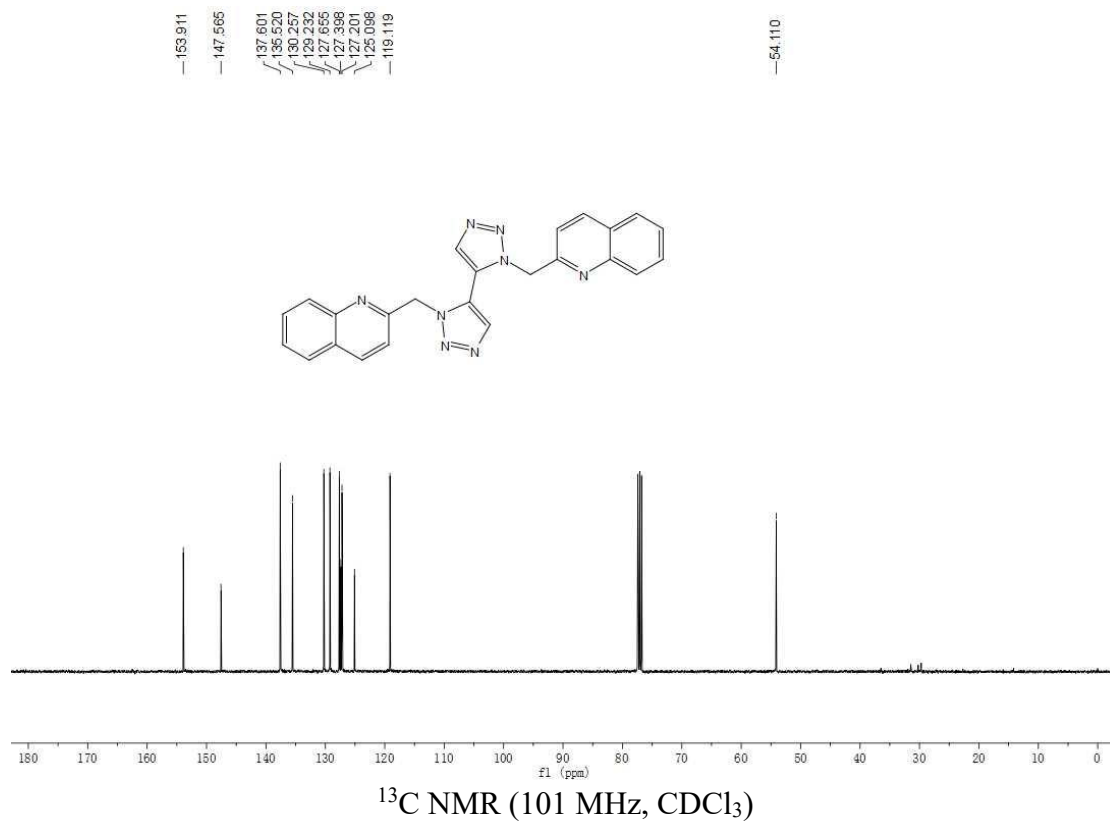
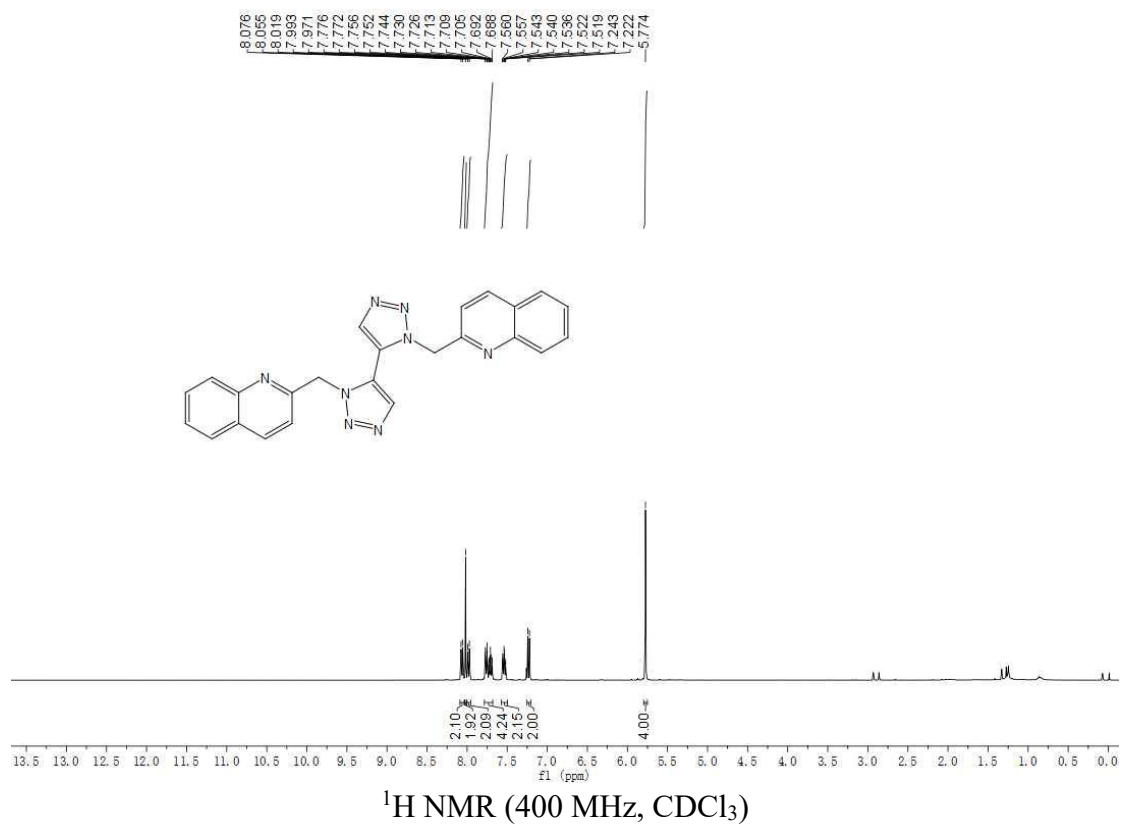
^1H and ^{13}C NMR spectra of compound **2ah**



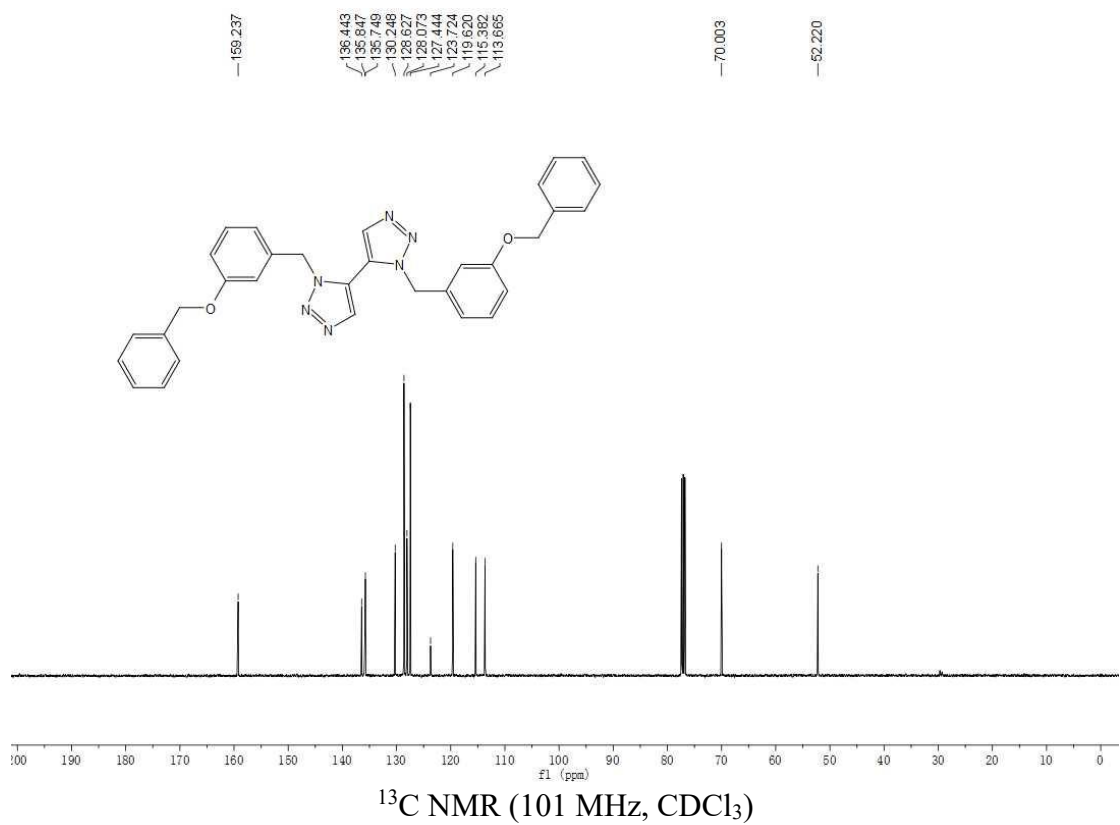
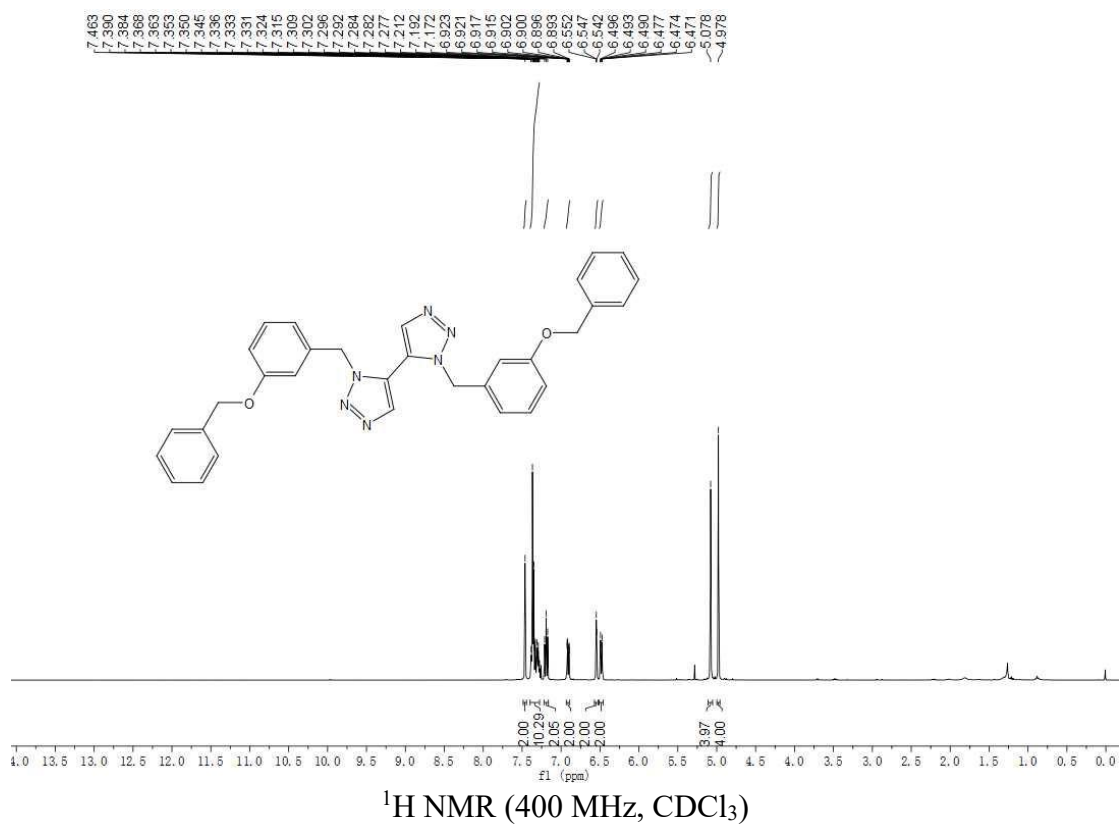
^1H and ^{13}C NMR spectra of compound **2ai**



^1H and ^{13}C NMR spectra of compound **2aj**

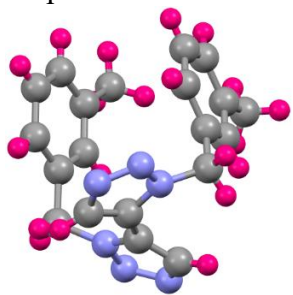


^1H and ^{13}C NMR spectra of compound **2ak**

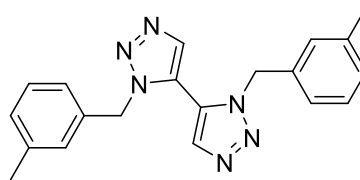


6. X-ray Information for 2c

Crystals suitable of **2c** for X-ray analysis could be successfully grown by slow volatilization in petroleum ether. CCDC: 2414231.



CCDC: 2414231



2c

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) ws_1

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: ws_1

Bond precision:	C-C = 0.0023 A	Wavelength=1.54184
Cell:	a=8.3739 (2)	b=13.2079 (3) c=32.2928 (8)
	alpha=90	beta=90 gamma=90
Temperature:	213 K	
	Calculated	Reported
Volume	3571.64 (15)	3571.64 (15)
Space group	P b c a	P b c a
Hall group	-P 2ac 2ab	-P 2ac 2ab
Moiety formula	C20 H20 N6	C20 H20 N6
Sum formula	C20 H20 N6	C20 H20 N6
Mr	344.42	344.42
Dx, g cm ⁻³	1.281	1.281
Z	8	8
Mu (mm ⁻¹)	0.638	0.638
F000	1456.0	1456.0
F000'	1460.00	
h, k, lmax	10, 15, 38	9, 15, 38
Nref	3180	3158
Tmin, Tmax	0.955, 0.962	0.850, 1.000
Tmin'	0.950	

Correction method= # Reported T Limits: Tmin=0.850 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.993 Theta(max)= 67.075

R(reflections)= 0.0416 (2561)	wR2(reflections)=
S = 1.048	0.1141 (3158)
Npar= 236	

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.



Alert level C

PLAT906_ALERT_3_C Large K Value in the Analysis of Variance	2.375 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.597	21 Report



Alert level G

PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary .	Please Do !
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still	70% Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity	3.4 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.	5 Info

- 0 **ALERT level A** = Most likely a serious problem - resolve or explain
- 0 **ALERT level B** = A potentially serious problem, consider carefully
- 2 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
- 4 **ALERT level G** = General information/check it is not something unexpected

- 1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 - 1 ALERT type 2 Indicator that the structure model may be wrong or deficient
 - 4 ALERT type 3 Indicator that the structure quality may be low
 - 0 ALERT type 4 Improvement, methodology, query or suggestion
 - 0 ALERT type 5 Informative message, check
-
-

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

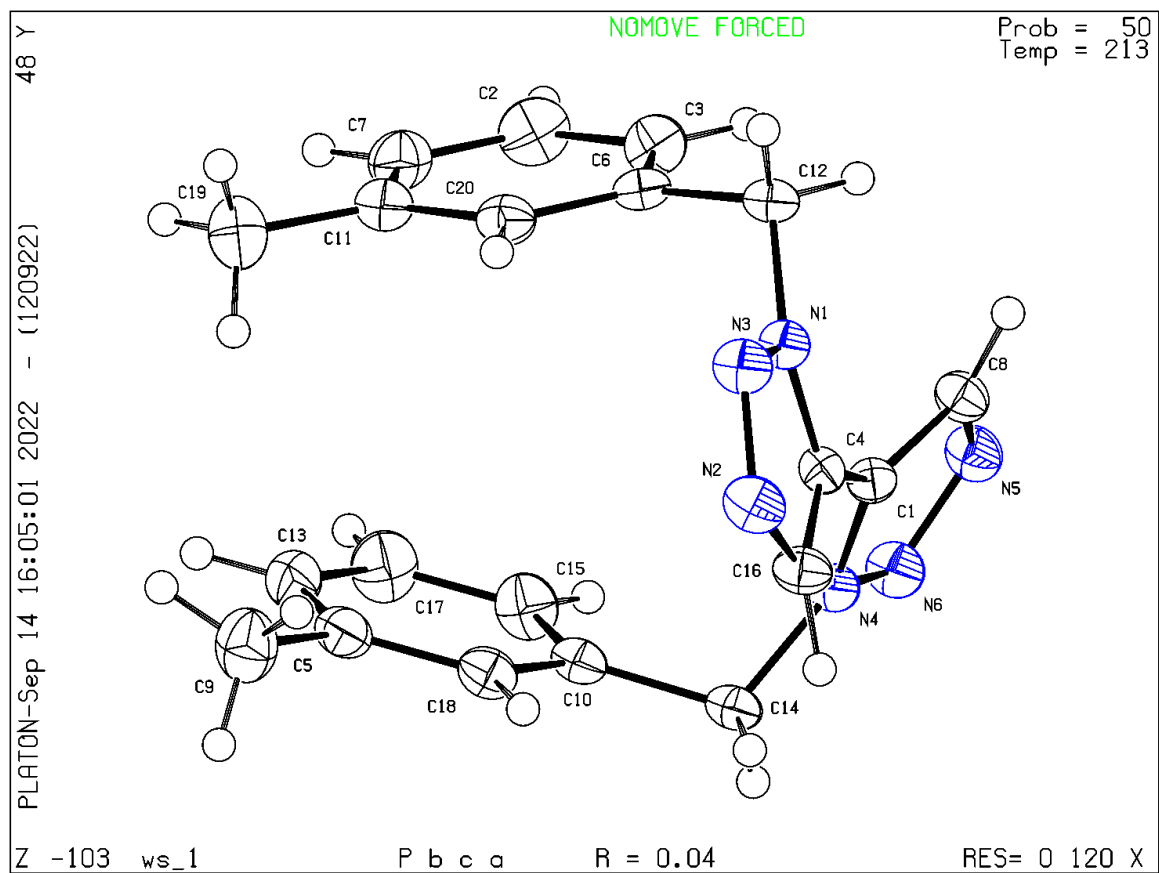
Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 12/09/2022; check.def file version of 09/08/2022

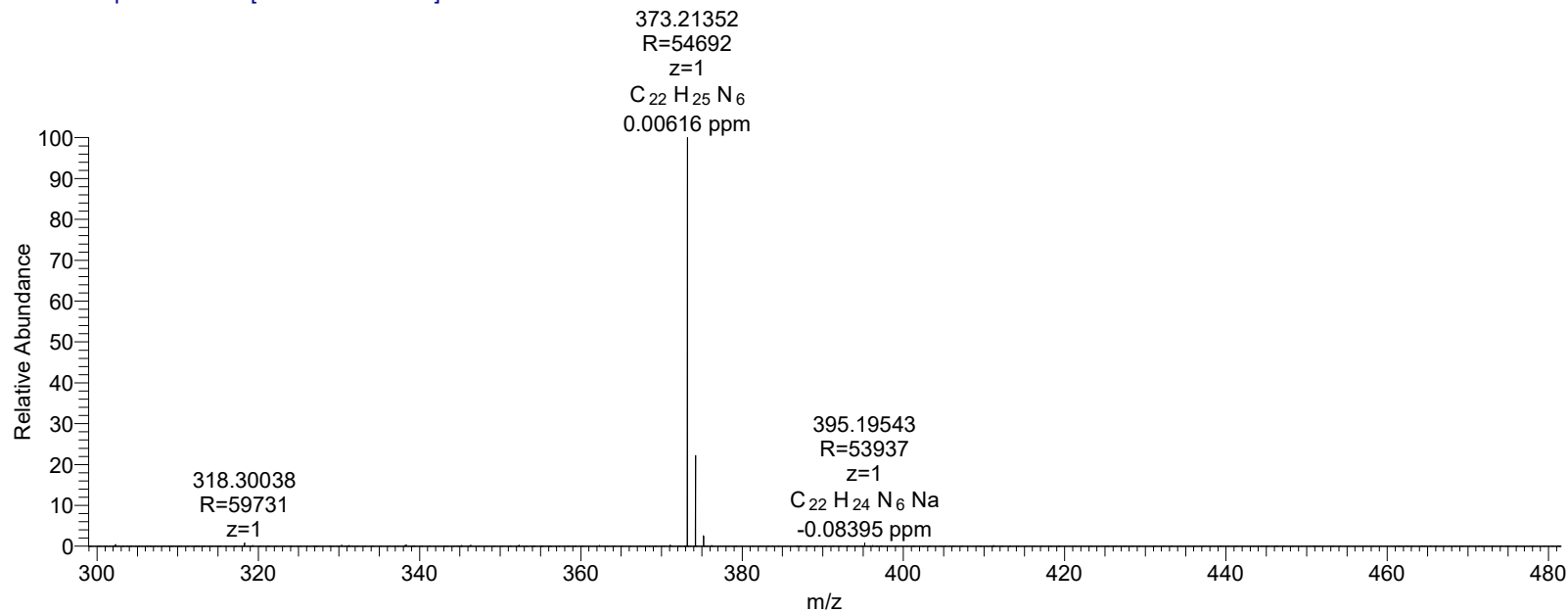


7. The HRMS for the representative products

Supplementary FDF files corresponding to the HRMS data for compounds (**2f**: FRG-1; **2g**: FRG-2; **2h**: FRG-3; **2j**: FRG-4; **2m**: FRG-5; **2n**: FRG-6; **2o**: FRG-7; **2q**: FRG-8; **2r**: FRG-9; **2s**: FRG-10; **2t**: FRG-11; **2v**: FRG-12; **2w**: FRG-13; **2i**: FRG-14; **2z**: FRG-15; **2aa**: FRG-16; **2ab**: FRG-17; **2ac**: FRG-18; **2ad**: FRG-19; **2af**: FRG-20; **2ag**: FRG-21; **2ah**: FRG-22; **2ai**: FRG-23; **2aj**: FRG-24; **2ak**: FRG-25) are provided below.

FRG-1-0812 #1-112 RT: 0.00423-0.49895 AV: 112 NL: 9.82E8

T: FTMS + p ESI Full ms [50.0000-750.0000]

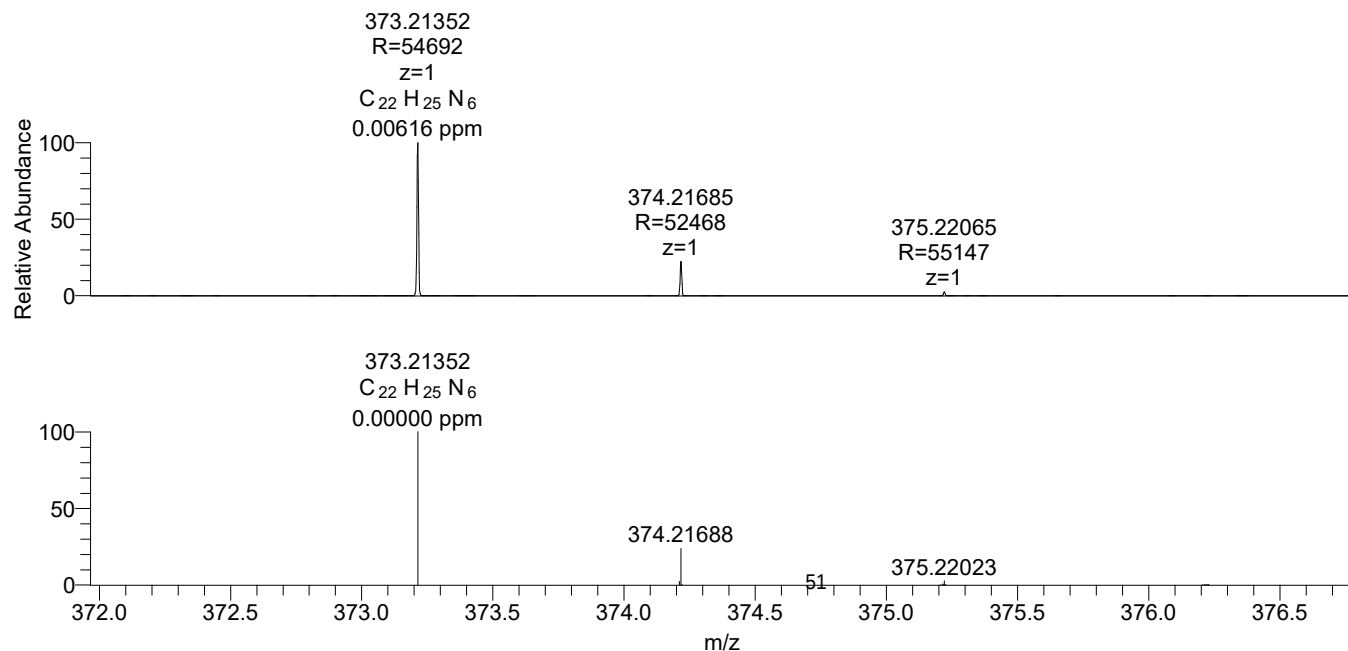


FRG-1-0812 #1-112 RT: 0.00423-0.49895 AV: 112

T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z = 317.85435-431.93967

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
318.30038	8199959.5	0.83	59730.63	1.00		
338.34181	3637443.8	0.37	56358.51	0.00		
352.32073	2620607.5	0.26	58197.94	1.00		
373.21352	990420864.0	100.00	54691.57	1.00	0.01	C ₂₂ H ₂₅ N ₆
374.21685	226033968.0	22.82	52468.08	1.00		
375.22065	24976568.0	2.52	55147.23	1.00		
395.19543	8302066.0	0.84	53937.15	1.00	-0.08	C ₂₂ H ₂₄ N ₆ Na

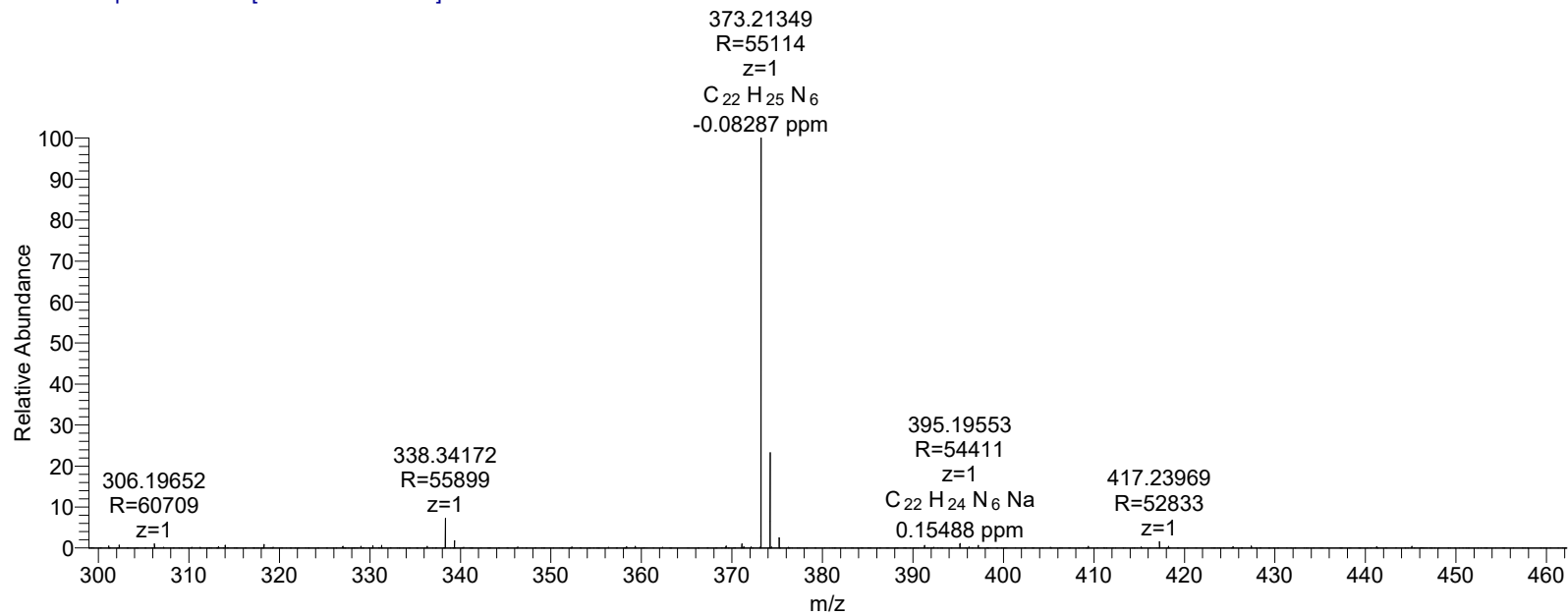


NL:
9.82E8
FRG-1-0812#1-112
RT: 0.00423-0.49895
AV: 112 T: FTMS + p
ESI Full ms
[50.0000-750.0000]

NL:
7.70E5
C₂₂H₂₄N₆+H:
C₂₂H₂₅N₆
pa Chrg 1

FRG-2-0812#90-141 RT: 0.40073-0.62804 AV: 52 NL: 2.28E8

T: FTMS + p ESI Full ms [50.0000-750.0000]

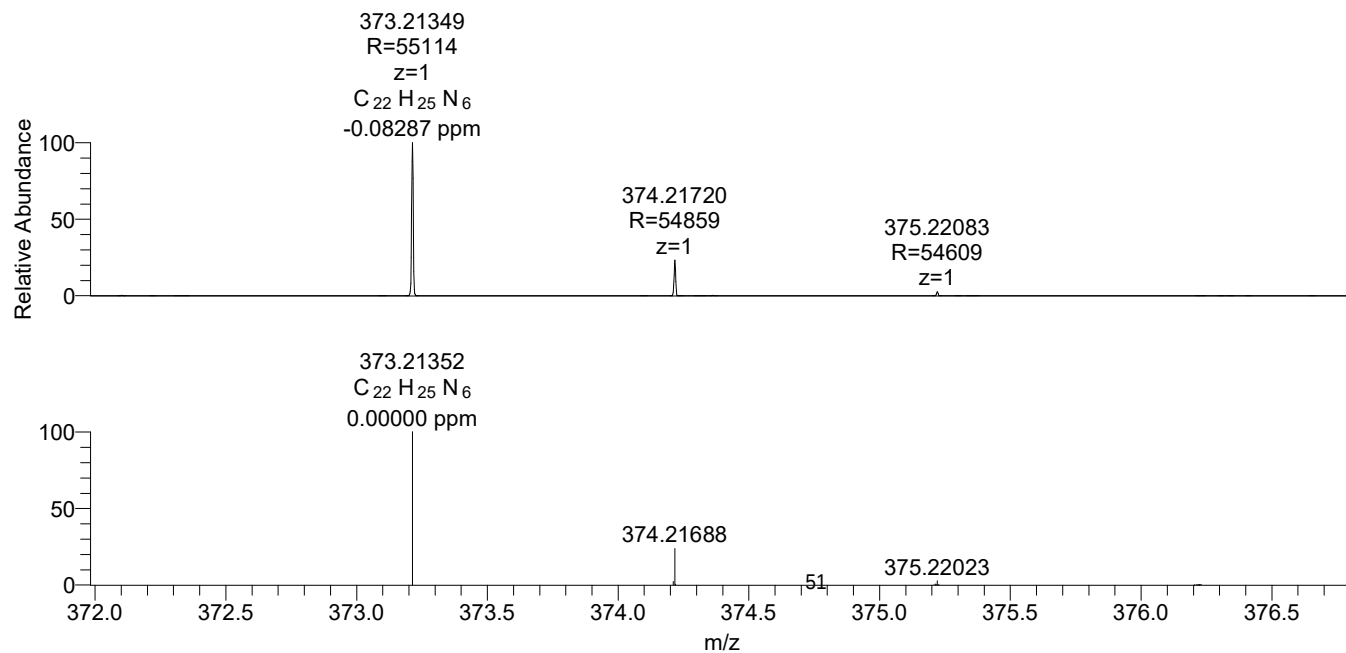


FRG-2-0812#90-141 RT: 0.40073-0.62804 AV: 52

T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z= 298.93937-462.23713

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
338.34172	17190718.0	7.50	55898.96	1.00	-0.08	$C_{22}H_{25}N_6$
339.34527	4154532.5	1.81	58656.69	1.00		
373.21349	229198832.0	100.00	55114.46	1.00		
374.21720	53638208.0	23.40	54859.07	1.00		
375.22083	5881374.0	2.57	54609.07	1.00		
395.19553	2474812.3	1.08	54411.45	1.00	0.15	$C_{22}H_{24}N_6Na$
417.23969	3668968.5	1.60	52832.60	1.00		

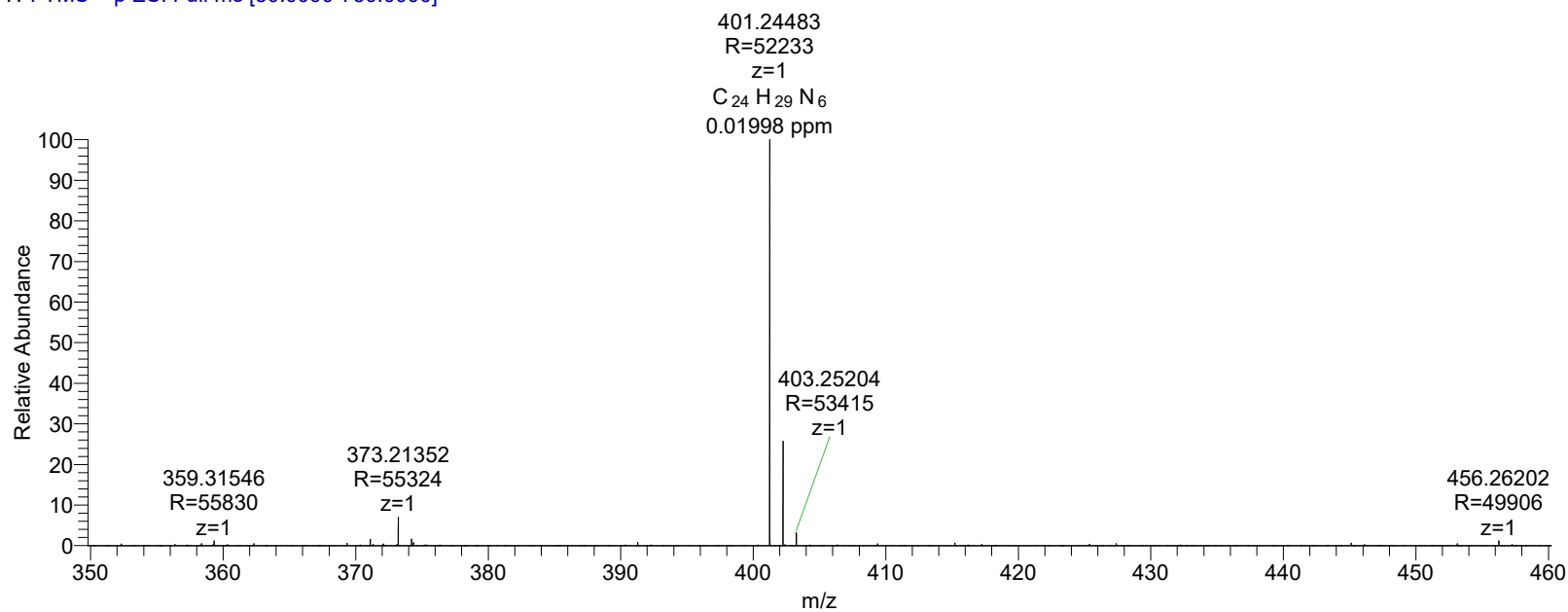


NL:
2.28E8
FRG-2-0812#90-141
RT: 0.40073-0.62804
AV: 52 T: FTMS + p
ESI Full ms
[50.0000-750.0000]

NL:
7.70E5
 $C_{22}H_{24}N_6 + H$:
 $C_{22}H_{25}N_6$
pa Chrg 1

FRG-3-0812 #44-135 RT: 0.19571-0.60129 AV: 92 NL: 1.73E8

T: FTMS + p ESI Full ms [50.0000-750.0000]

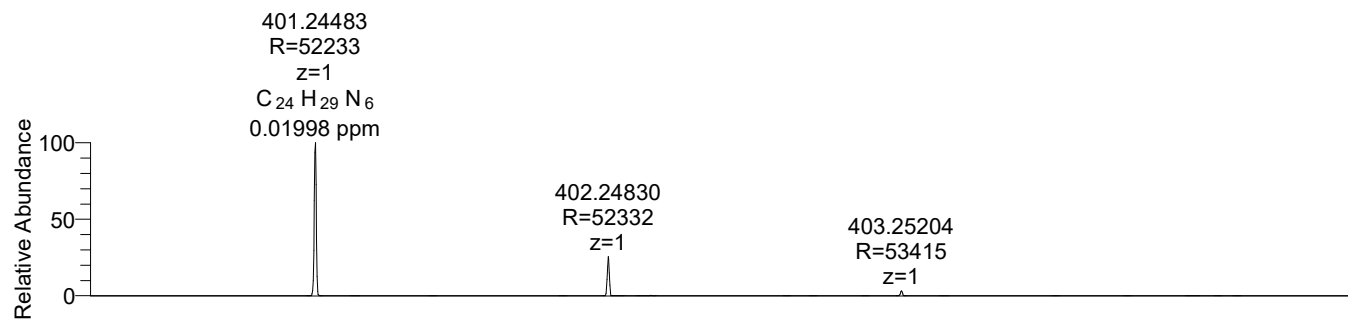


FRG-3-0812#44-135 RT: 0.19571-0.60129 AV: 92

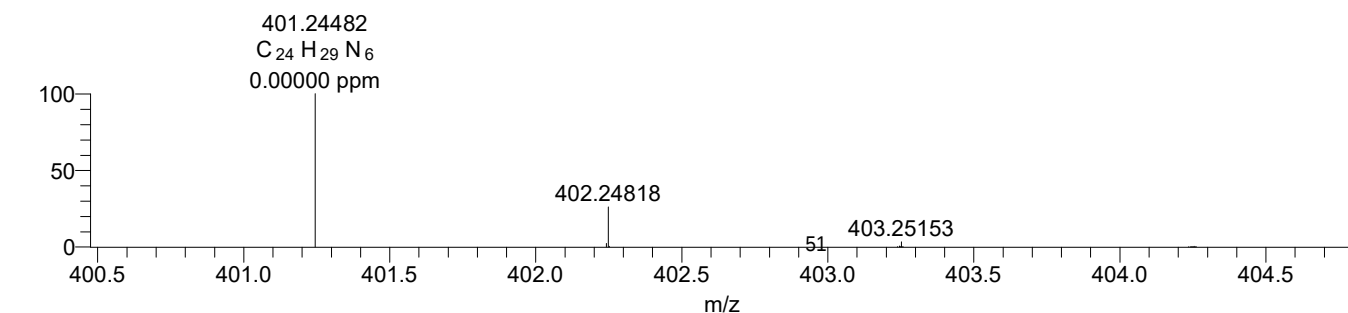
T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z= 349.79465-460.20981

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
359.31546	2068434.0	1.17	55829.93	1.00		
371.10126	2712430.3	1.54	56496.49	1.00		
373.21352	12240348.0	6.95	55323.98	1.00		
374.21729	2891343.8	1.64	55832.63	1.00		
401.24483	176227840.0	100.00	52233.32	1.00	0.02	C ₂₄ H ₂₉ N ₆
402.24830	45105316.0	25.59	52331.79	1.00		
403.25204	5588749.5	3.17	53414.92	1.00		



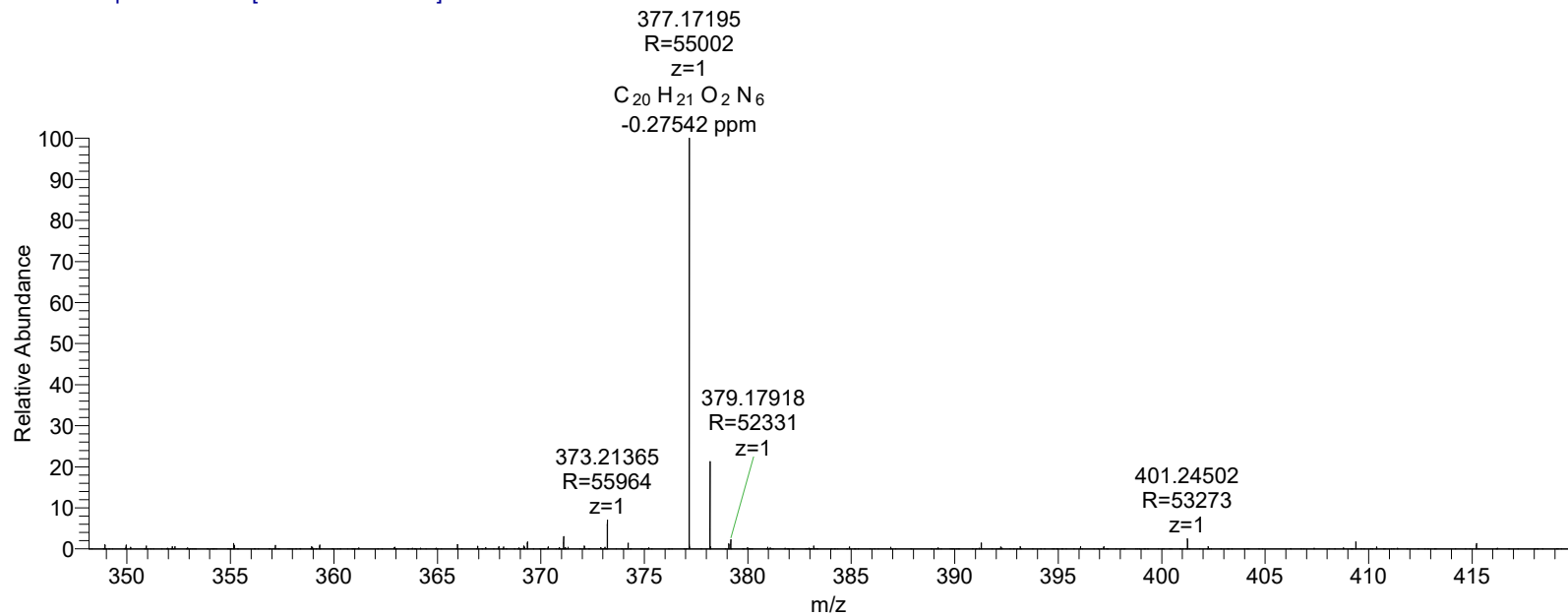
NL:
1.73E8
FRG-3-0812#44-135
RT: 0.19571-0.60129
AV: 92 T: FTMS + p
ESI Full ms
[50.0000-750.0000]



NL:
7.53E5
C₂₄H₂₈N₆+H:
C₂₄H₂₉N₆
pa Chrg 1

FRG-4-0812 #56-142 RT: 0.24919-0.63249 AV: 87 NL: 7.38E7

T: FTMS + p ESI Full ms [50.0000-750.0000]

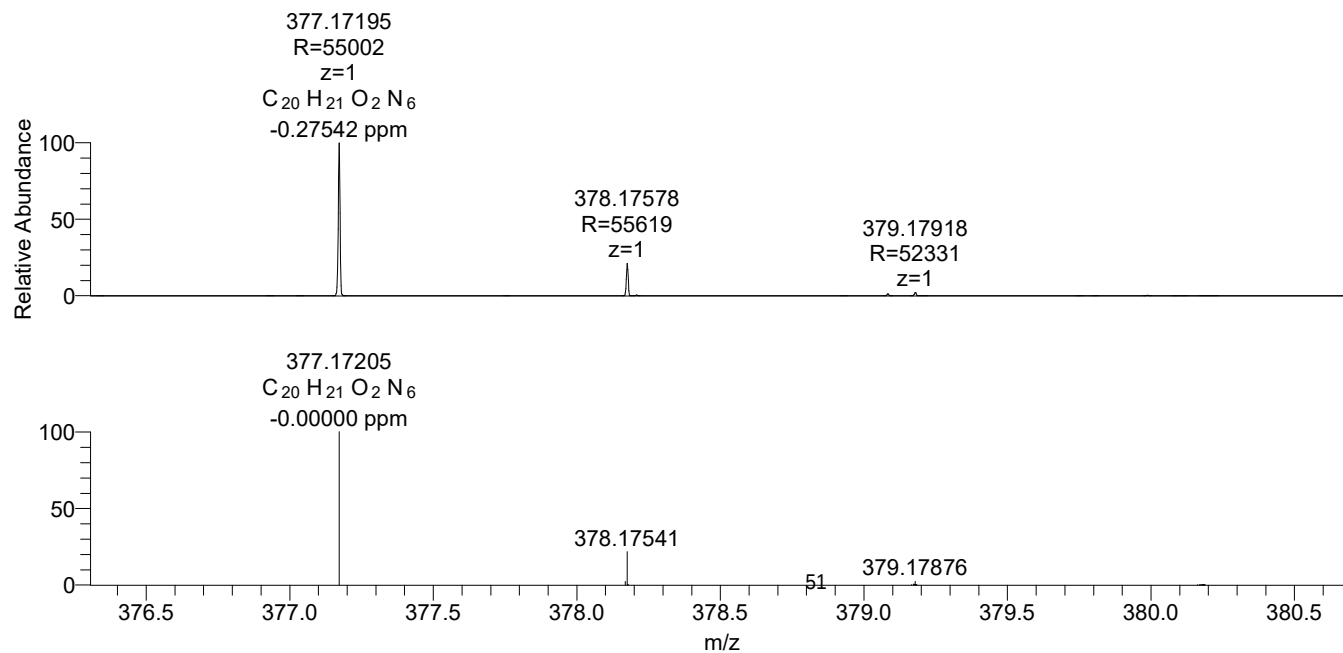


FRG-4-0812 #56-142 RT: 0.24919-0.63249 AV: 87

T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z= 348.16143-419.66654

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
371.10129	2217907.5	2.99	56333.18	1.00	-0.28	C ₂₀ H ₂₁ O ₂ N ₆
373.21365	5152954.5	6.96	55964.25	1.00		
377.17195	74087176.0	100.00	55001.74	1.00		
378.17578	15702278.0	21.19	55619.00	1.00		
379.17918	1689178.8	2.28	52330.95	1.00		
401.24502	1832513.1	2.47	53272.77	1.00		
409.38280	1302776.8	1.76	53439.93	1.00		

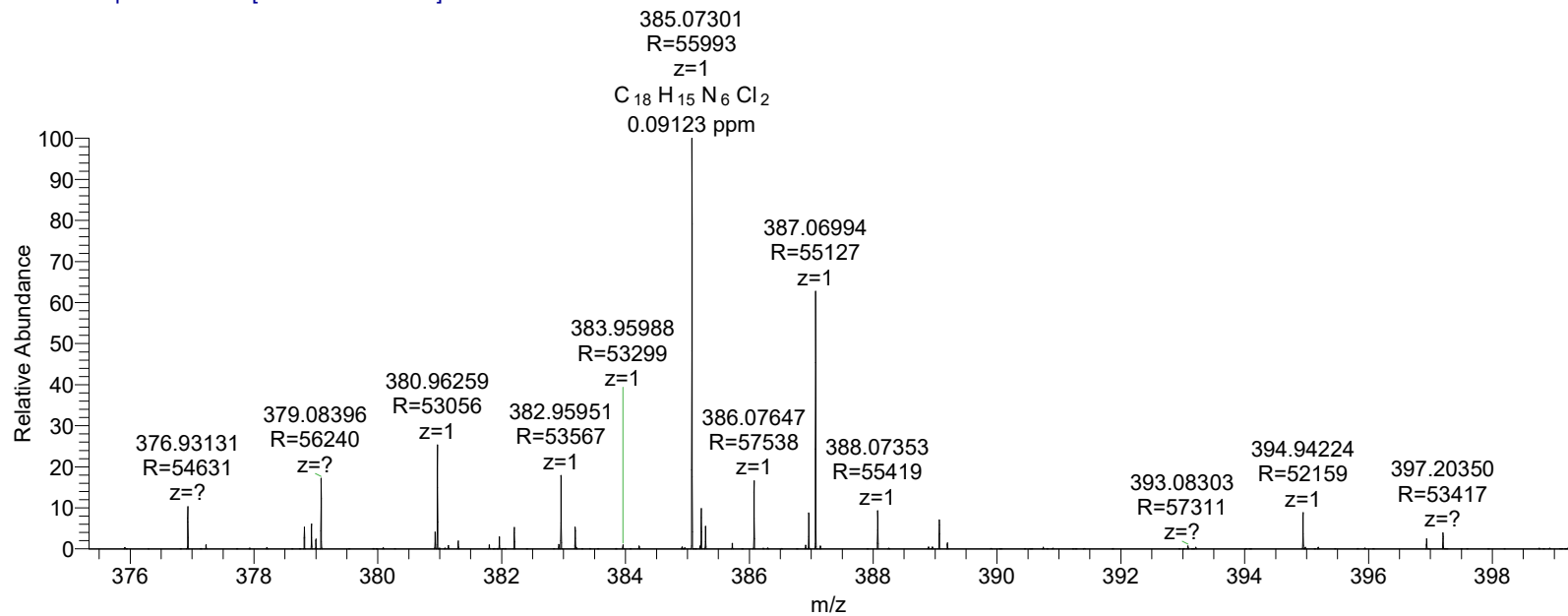


NL:
7.38E7
FRG-4-0812#56-142
RT: 0.24919-0.63249
AV: 87 T: FTMS + p
ESI Full ms
[50.0000-750.0000]

NL:
7.83E5
C₂₀H₂₀N₆O₂+H:
C₂₀H₂₁N₆O₂
pa Chrg 1

FRG-5-0812 #117-208 RT: 0.52108-0.92666 AV: 92 NL: 7.51E6

T: FTMS + p ESI Full ms [50.0000-750.0000]

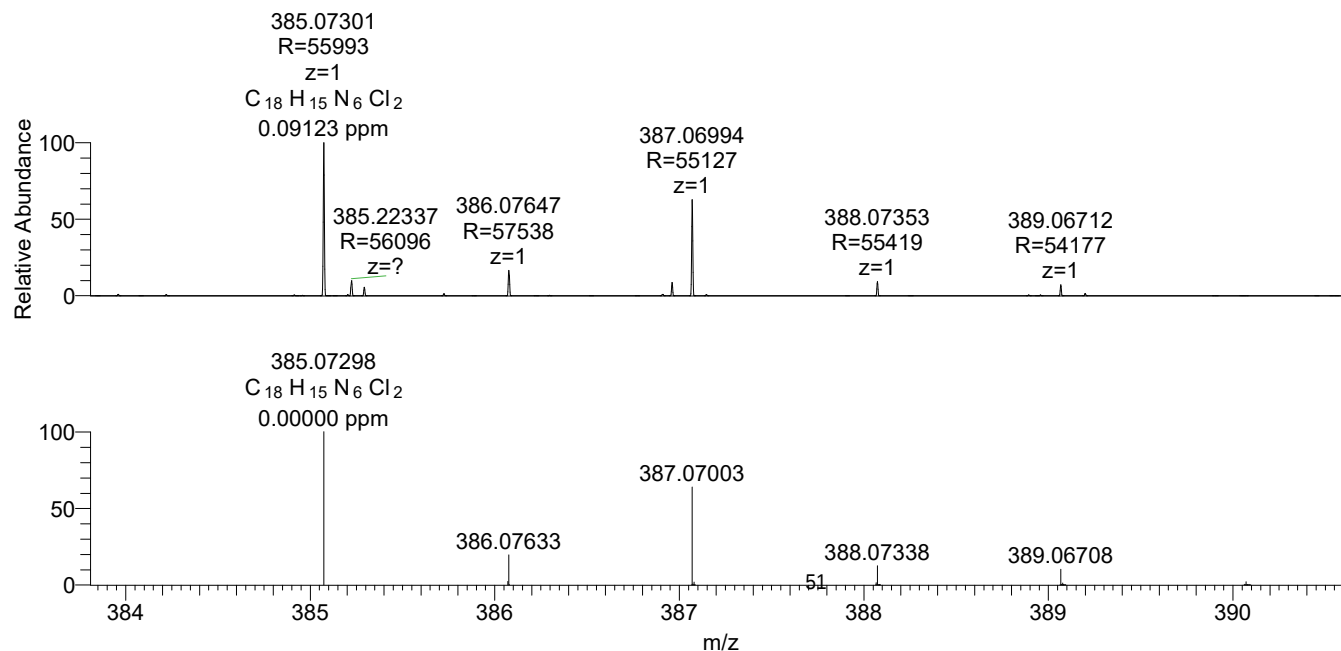


FRG-5-0812 #117-208 RT: 0.52108-0.92666 AV: 92

T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z = 375.33363-399.23270

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
376.93131	789057.8	10.50	54630.93	0.00		
379.08396	1316520.4	17.52	56240.27	0.00		
380.96259	1915085.1	25.49	53055.87	1.00		
382.95951	1386467.1	18.45	53566.64	1.00		
385.07301	7514476.0	100.00	55993.28	1.00	0.09	C ₁₈ H ₁₅ N ₆ Cl ₂
385.22337	743341.4	9.89	56095.99	0.00		
386.07647	1249378.0	16.63	57537.85	1.00		
387.06994	4760155.5	63.35	55127.21	1.00		
388.07353	701202.7	9.33	55419.13	1.00		

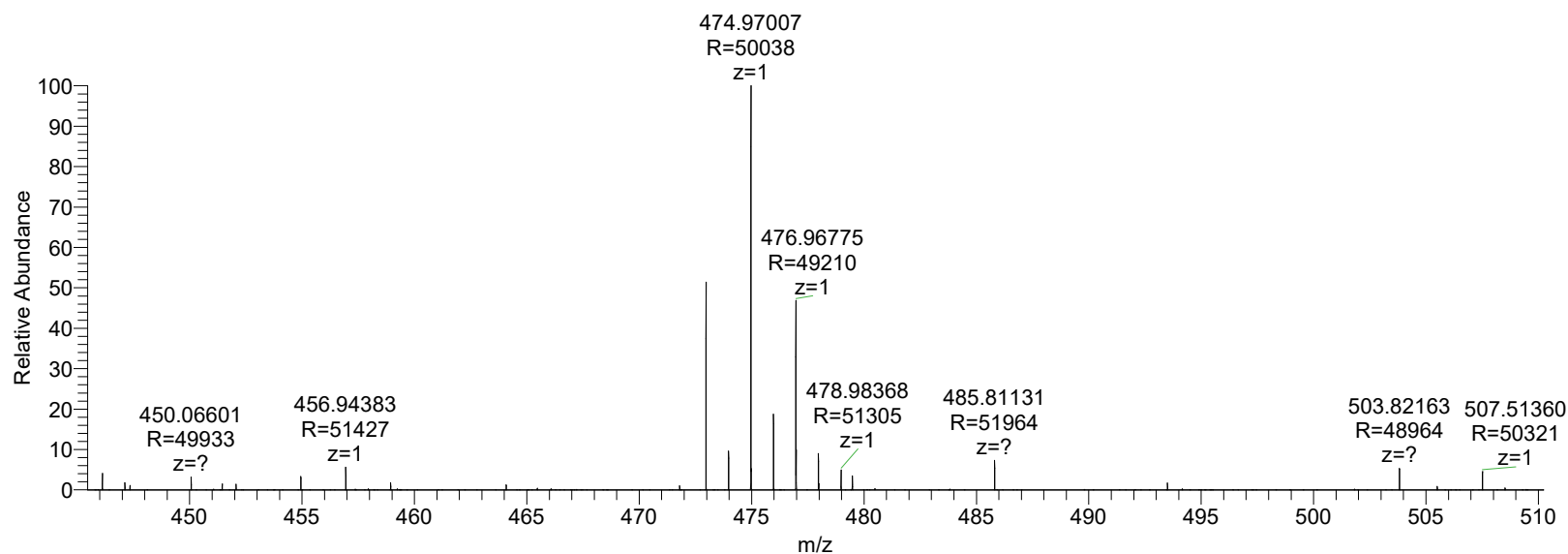


NL:
7.51E6
FRG-5-0812#117-208
RT: 0.52108-0.92666
AV: 92 T: FTMS + p
ESI Full ms
[50.0000-750.0000]

NL:
4.62E5
C₁₈ H₁₄ Cl₂ N₆ +H:
C₁₈ H₁₅ Cl₂ N₆
pa Chrg 1

FRG-6-0812 #63-139 RT: 0.28039-0.61912 AV: 77 NL: 8.81E6

T: FTMS + p ESI Full ms [50.0000-750.0000]

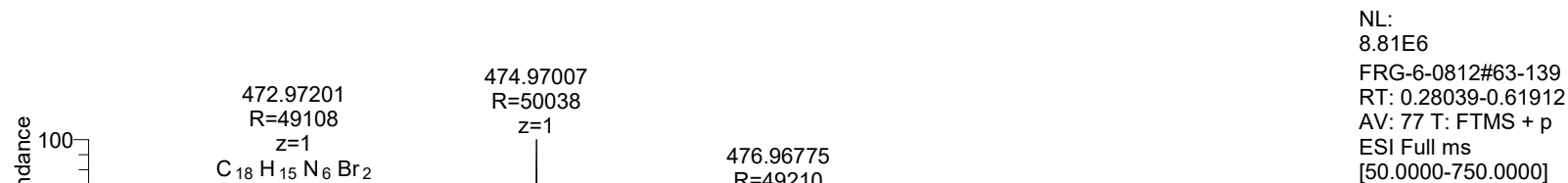


FRG-6-0812#63-139 RT: 0.28039-0.61912 AV: 77

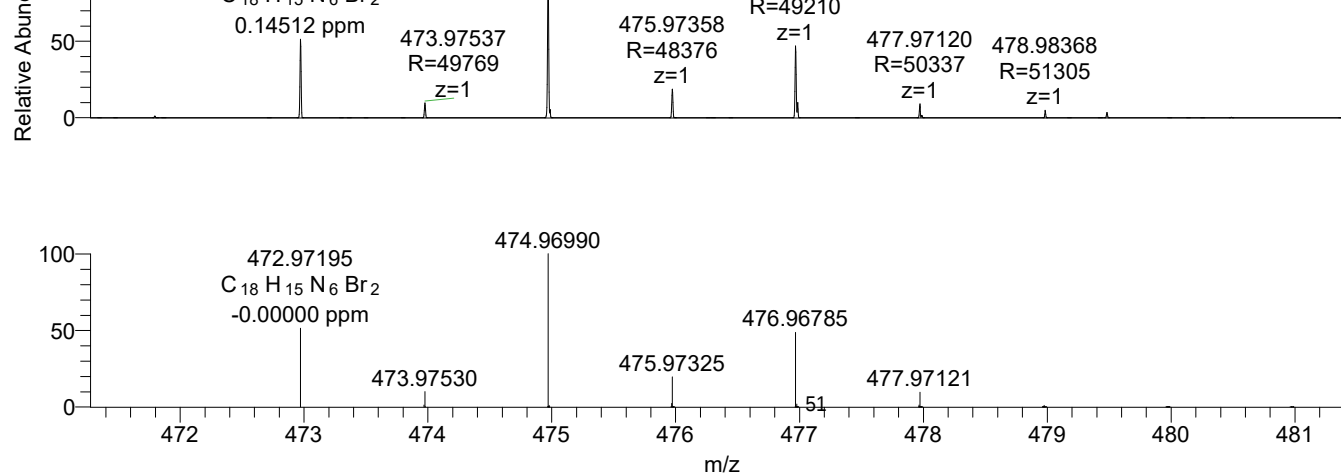
T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z = 445.45423-510.22813

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
456.94383	511330.7	5.80	51426.65	1.00	0.15	C ₁₈ H ₁₅ N ₆ Br ₂
472.97201	4579115.5	51.91	49107.52	1.00		
473.97537	850674.4	9.64	49769.39	1.00		
474.97007	8821356.0	100.00	50037.65	1.00		
475.97358	1654144.0	18.75	48375.97	1.00		
476.96775	4195722.5	47.56	49210.04	1.00		
476.98541	899727.3	10.20	50775.13	1.00		
477.97120	796205.9	9.03	50337.33	1.00		
485.81131	647518.8	7.34	51964.49	0.00		

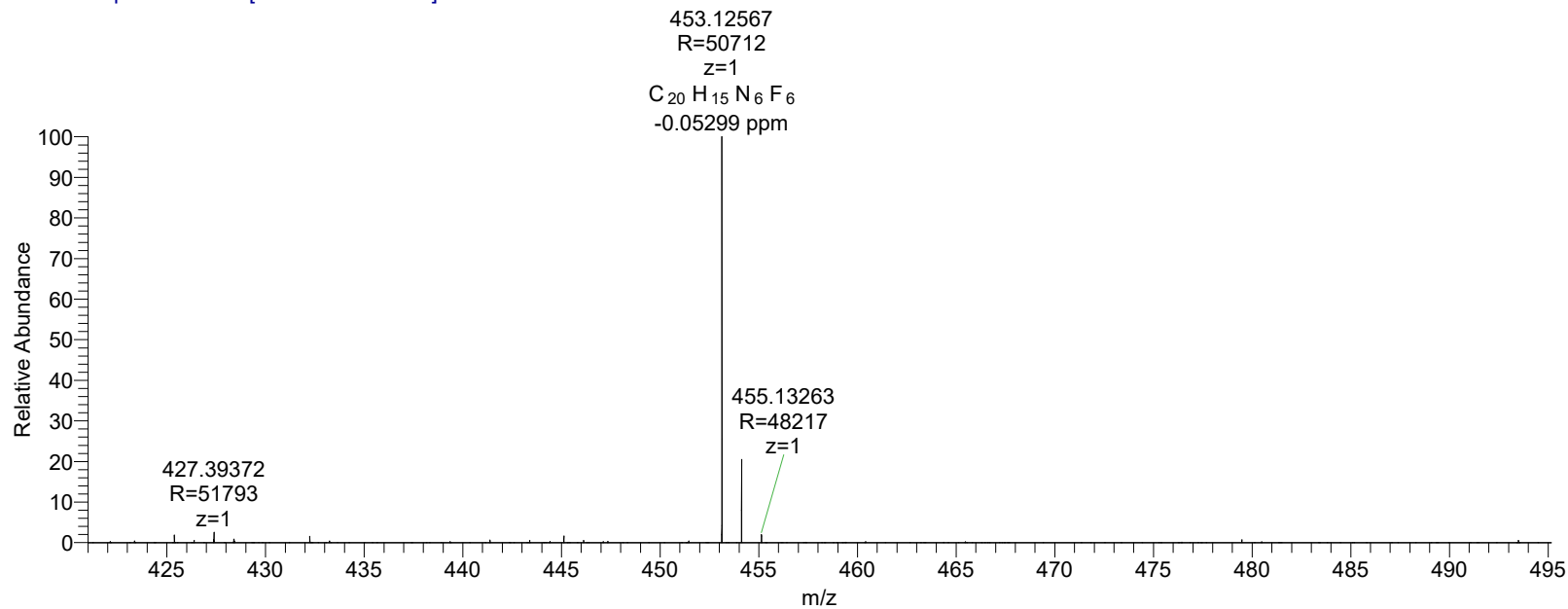


NL:
8.81E6
FRG-6-0812#63-139
RT: 0.28039-0.61912
AV: 77 T: FTMS + p
ESI Full ms
[50.0000-750.0000]



NL:
4.02E5
C₁₈ H₁₄ Br₂ N₆ +H:
C₁₈ H₁₅ Br₂ N₆
pa Chrg 1

FRG-7-0812#100-139 RT: 0.44530-0.61913 AV: 40 NL: 6.87E7
T: FTMS + p ESI Full ms [50.0000-750.0000]

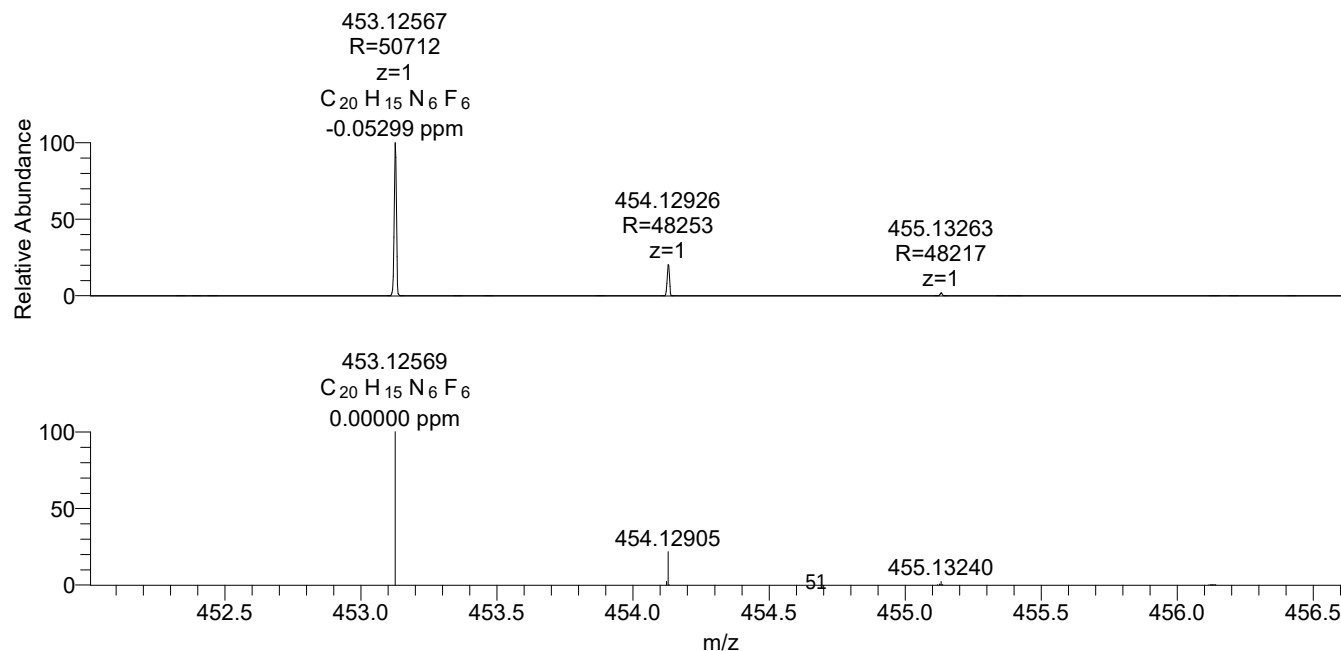


FRG-7-0812#100-139 RT: 0.44530-0.61913 AV: 40

T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z = 420.99750-495.15605

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
425.37773	1291575.0	1.86	52768.48	1.00	-0.05	C ₂₀ H ₁₅ N ₆ F ₆
427.39372	1761889.1	2.53	51793.36	1.00		
432.23832	1085301.9	1.56	51948.37	1.00		
445.12021	1127528.5	1.62	51580.66	1.00		
453.12567	69546696.0	100.00	50712.13	1.00		
454.12926	14359460.0	20.65	48252.86	1.00		
455.13263	1414631.3	2.03	48216.77	1.00		

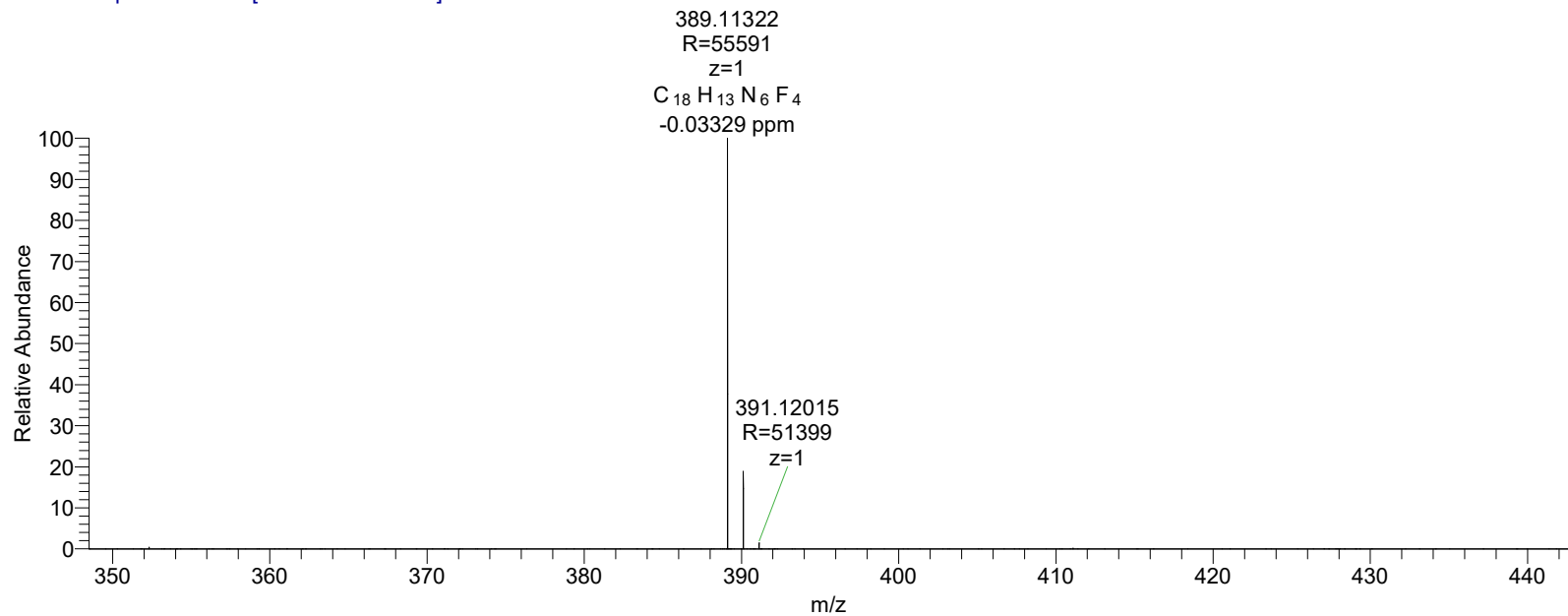


NL:
6.87E7
FRG-7-0812#100-139
RT: 0.44530-0.61913
AV: 40 T: FTMS + p
ESI Full ms
[50.0000-750.0000]

NL:
7.87E5
C₂₀H₁₄N₆F₆+H:
C₂₀H₁₅N₆F₆
pa Chrg 1

FRG-8-0812 #88-142 RT: 0.39182-0.63249 AV: 55 NL: 1.33E9

T: FTMS + p ESI Full ms [50.0000-750.0000]

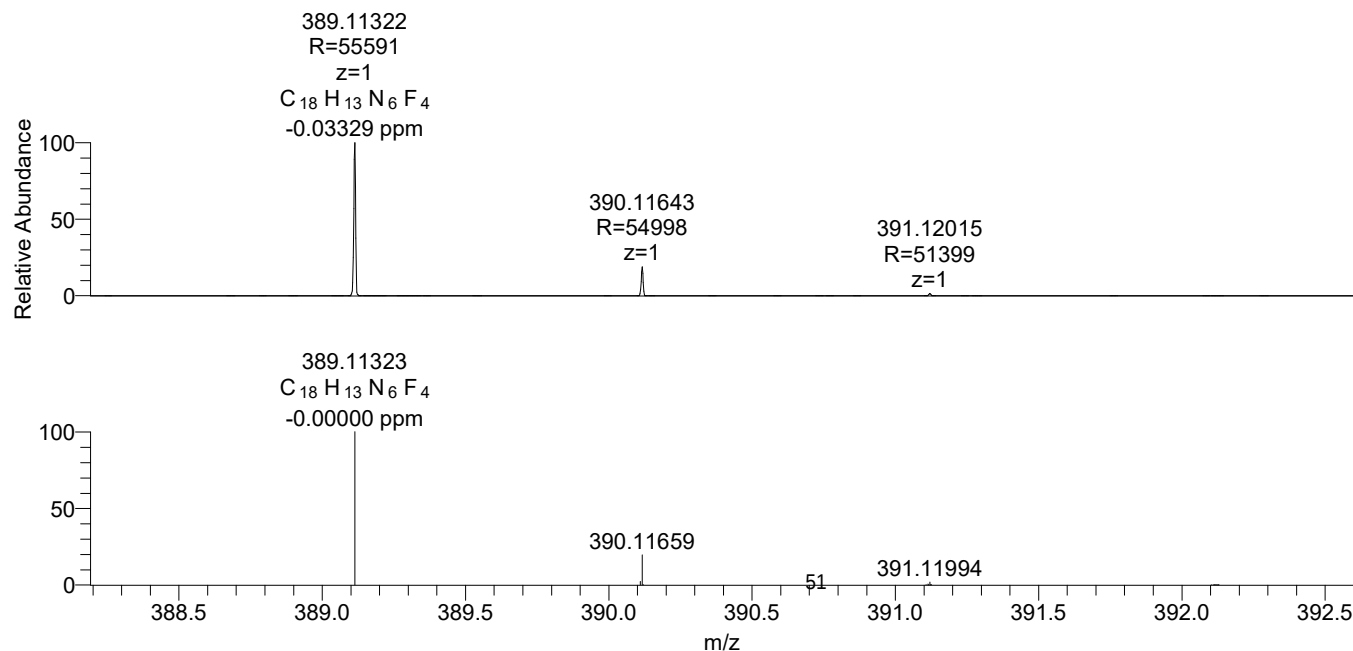


FRG-8-0812#88-142 RT: 0.39182-0.63249 AV: 55

T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z= 348.49355-442.61408

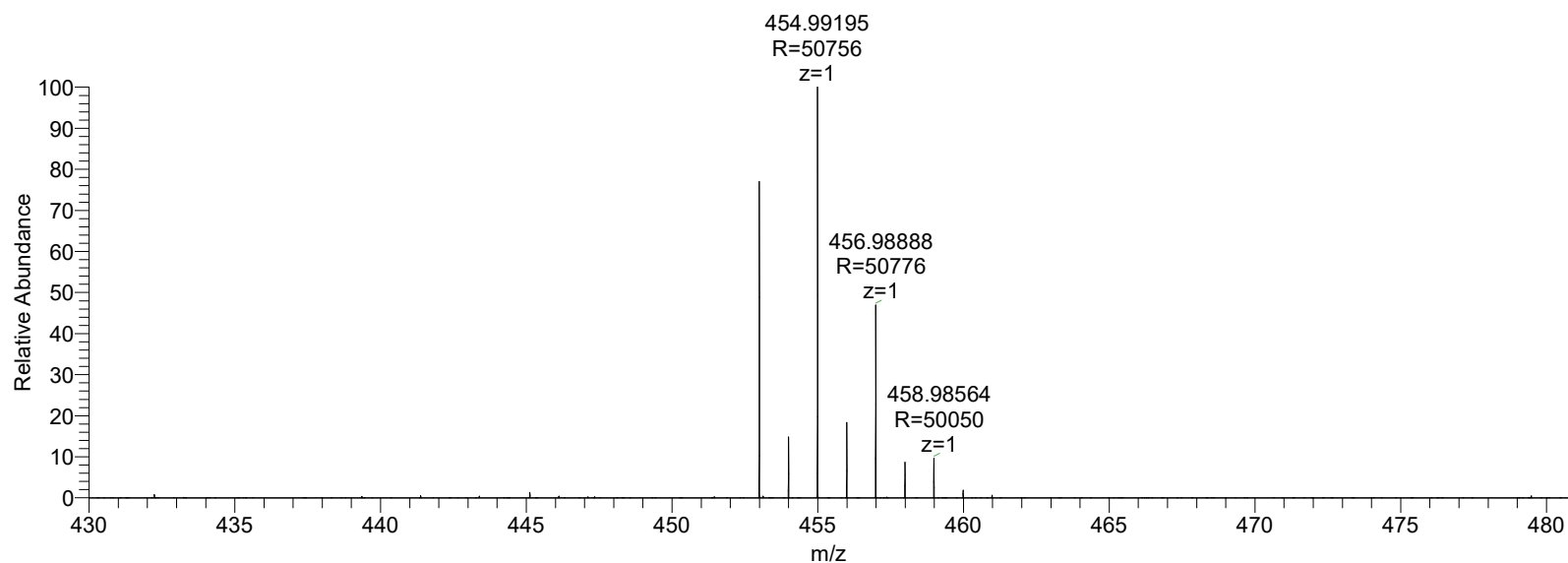
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
352.32101	5935939.5	0.44	56900.27	1.00		
361.10697	1905655.0	0.14	60723.84	1.00		
371.12285	1829279.9	0.14	56197.73	0.00		
389.11322	1348426496.0	100.00	55590.73	1.00	-0.03	C ₁₈ H ₁₃ N ₆ F ₄
390.11643	251899696.0	18.68	54998.40	1.00		
391.12015	20170778.0	1.50	51398.50	1.00		
411.09468	2979077.5	0.22	52131.71	1.00	-1.22	C ₁₈ H ₁₂ N ₆ F ₄ Na



NL:
1.33E9
FRG-8-0812#88-142
RT: 0.39182-0.63249
AV: 55 T: FTMS + p
ESI Full ms
[50.0000-750.0000]

NL:
8.05E5
C₁₈H₁₂F₄N₆+H:
C₁₈H₁₃F₄N₆
pa Chrg 1

FRG-9-0812#90-144 RT: 0.40073-0.64141 AV: 55 NL: 7.19E7
T: FTMS + p ESI Full ms [50.0000-750.0000]

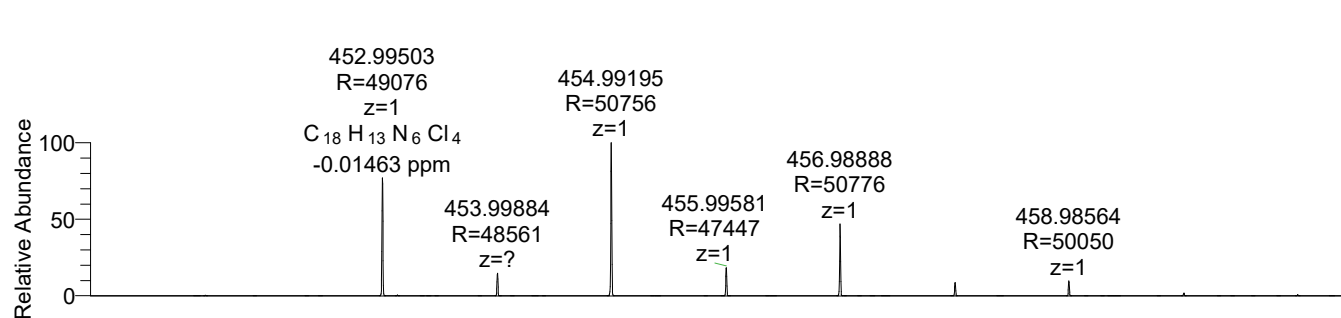


FRG-9-0812#90-144 RT: 0.40073-0.64141 AV: 55

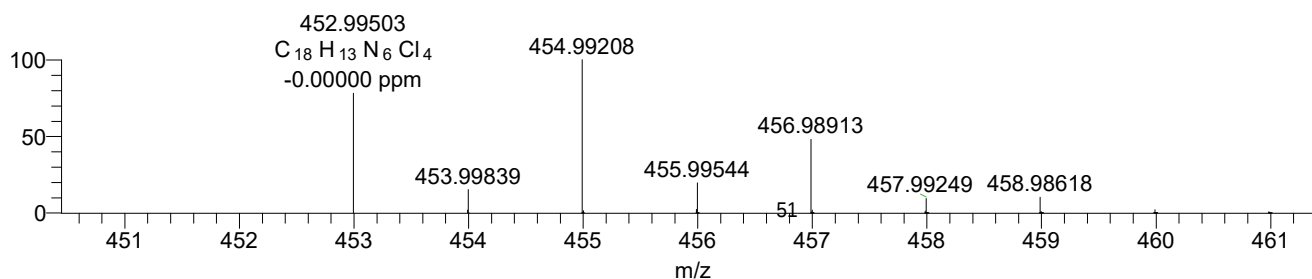
T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z = 415.72840-502.08098

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
452.99503	56490760.0	78.55	49076.46	1.00	-0.01	C ₁₈ H ₁₃ N ₆ Cl ₄
453.99884	10801415.0	15.02	48560.95	0.00		
454.99195	71916912.0	100.00	50756.17	1.00		
455.99581	13632009.0	18.96	47447.36	1.00		
456.98888	33766676.0	46.95	50776.11	1.00		
457.99276	6392242.0	8.89	47849.94	1.00		
458.98564	7032631.5	9.78	50049.68	1.00		



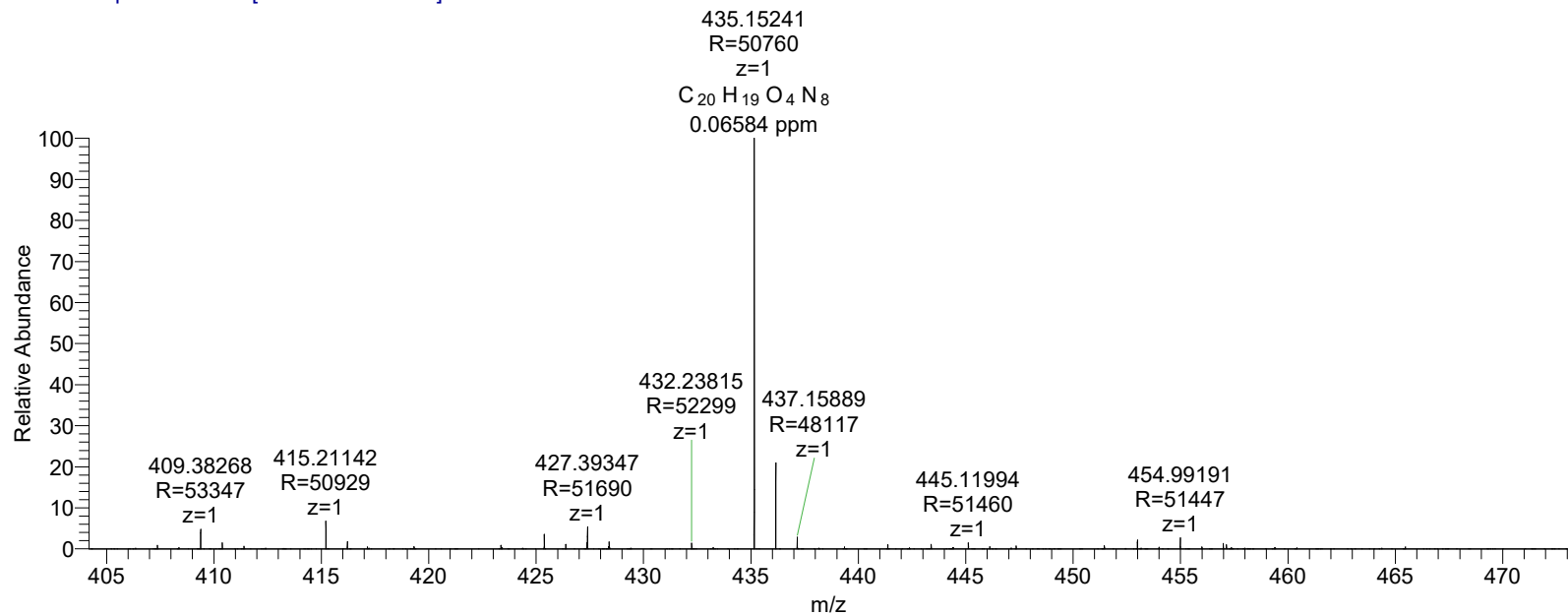
NL:
7.19E7
FRG-9-0812#90-144
RT: 0.40073-0.64141
AV: 55 T: FTMS + p
ESI Full ms
[50.0000-750.0000]



NL:
3.39E5
C₁₈H₁₂Cl₄N₆+H:
C₁₈H₁₃Cl₄N₆
pa Chrg 1

FRG-10-0812 #35-117 RT: 0.15576-0.52124 AV: 83 NL: 4.43E7

T: FTMS + p ESI Full ms [50.0000-750.0000]

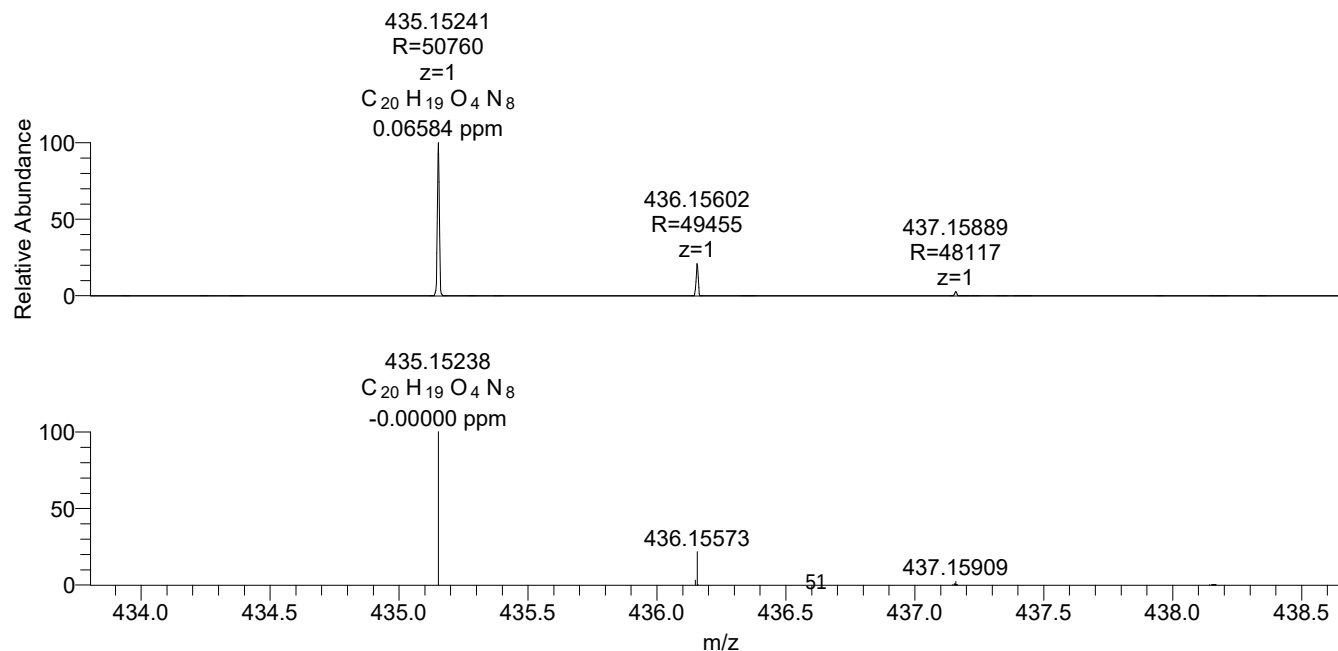


FRG-10-0812#35-117 RT: 0.15576-0.52124 AV: 83

T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z = 404.17860-473.07052

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
409.38268	2130707.8	4.75	53346.59	1.00	0.07	C ₂₀ H ₁₉ O ₄ N ₈
415.21142	3077380.0	6.86	50928.70	1.00		
425.37752	1592456.8	3.55	51841.43	1.00		
427.39347	2411760.3	5.38	51690.26	1.00		
435.15241	44853228.0	100.00	50759.51	1.00		
436.15602	9387036.0	20.93	49454.52	1.00		
437.15889	1267750.4	2.83	48117.18	1.00		
452.99488	979144.9	2.18	50540.57	1.00		
454.99191	1222773.1	2.73	51446.95	1.00		

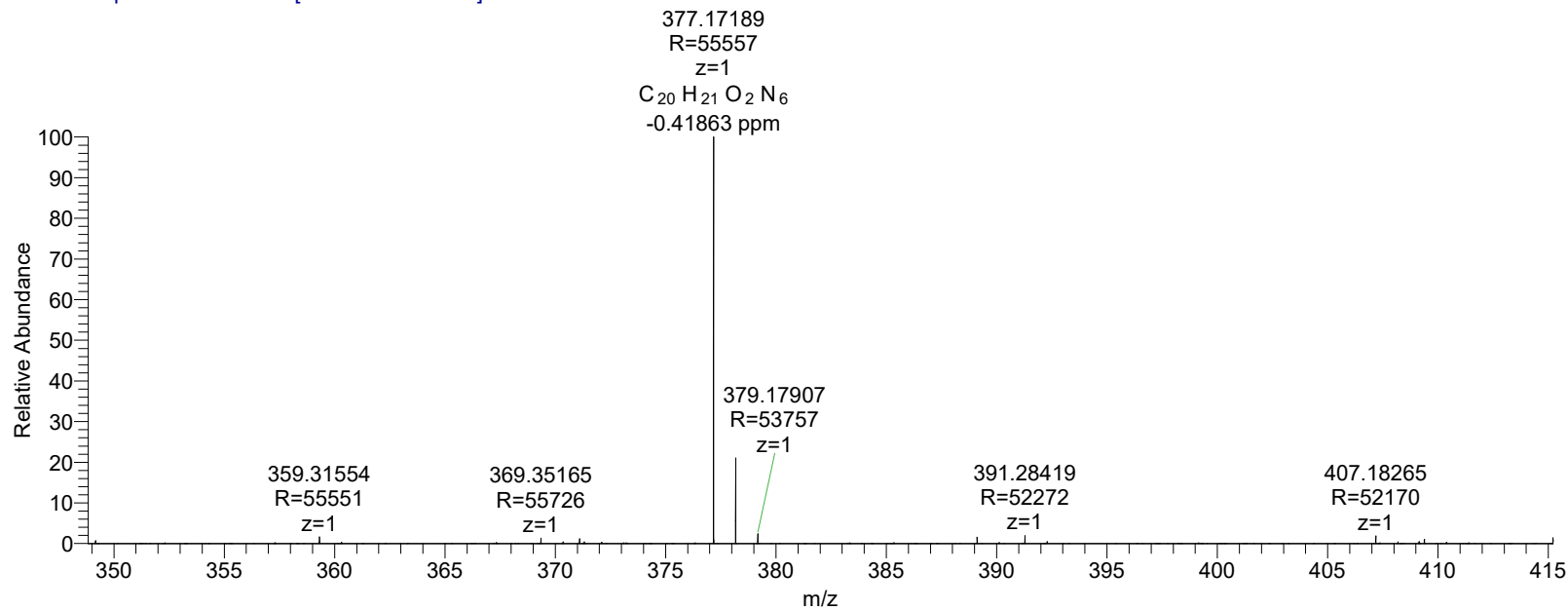


NL:
4.43E7
FRG-10-0812#35-117
RT: 0.15576-0.52124
AV: 83 T: FTMS + p
ESI Full ms
[50.0000-750.0000]

NL:
7.74E5
C₂₀H₁₈N₈O₄ +H:
C₂₀H₁₉N₈O₄
pa Chrg 1

FRG-11-0812 #101-138 RT: 0.44992-0.61483 AV: 38 NL: 1.72E8

T: FTMS + p ESI Full lock ms [50.0000-750.0000]

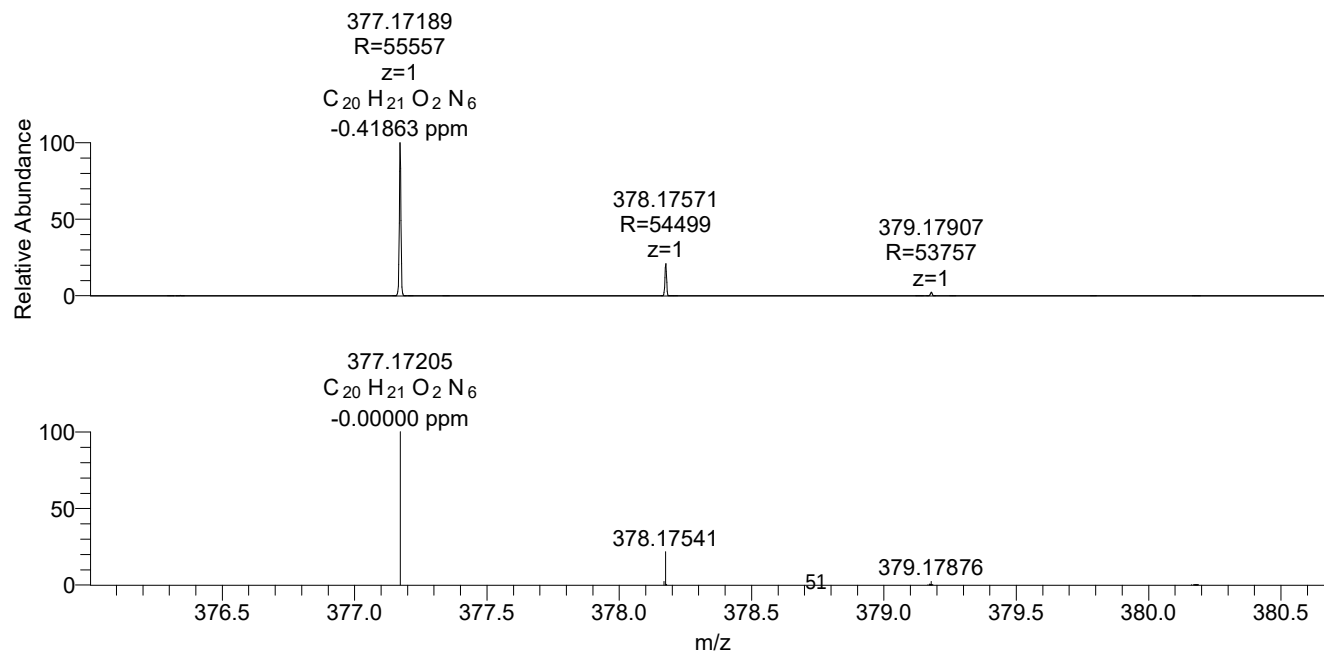


FRG-11-0812#101-138 RT: 0.44992-0.61483 AV: 38

T: FTMS + p ESI Full lock ms [50.0000-750.0000]

m/z = 348.82662-415.21460

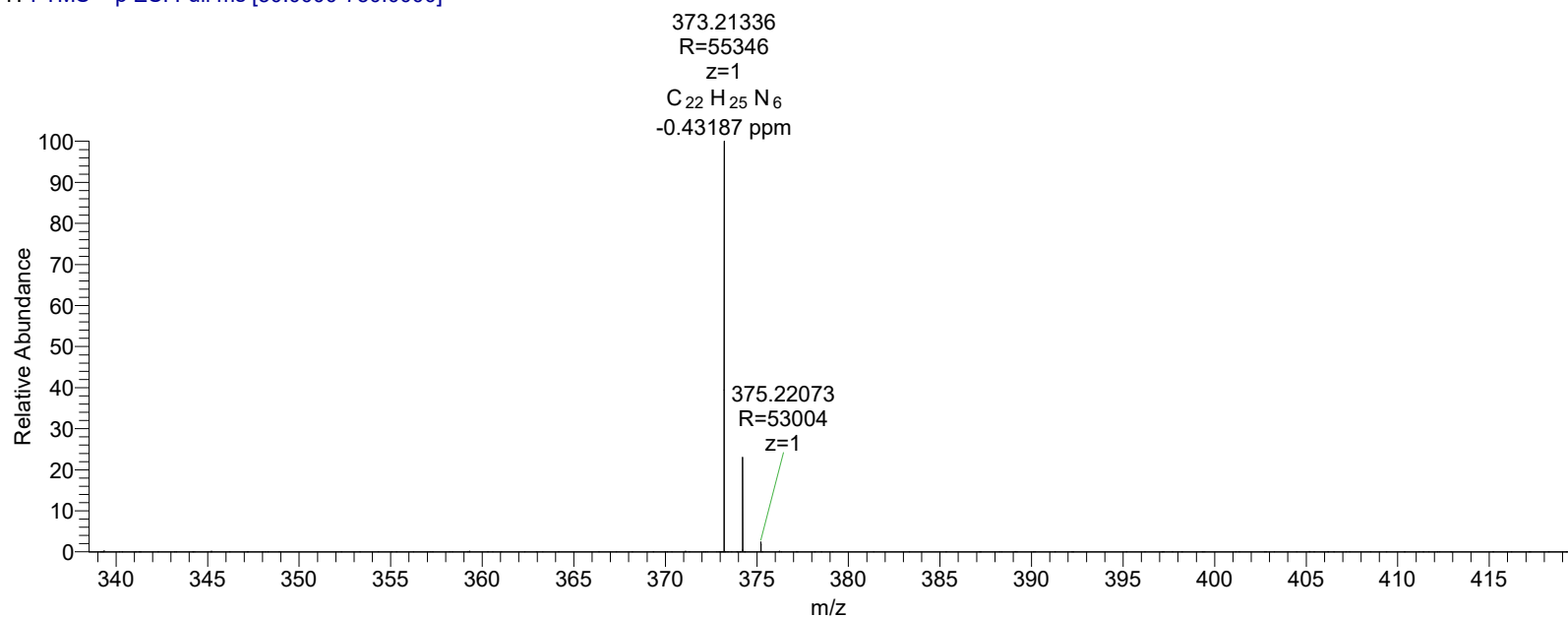
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
377.17189	171560528.0	100.00	55557.38	1.00	-0.42	C ₂₀ H ₂₁ O ₂ N ₆
378.17571	36309348.0	21.16	54498.68	1.00		
379.17907	4110610.3	2.40	53757.44	1.00		
391.28419	3506794.5	2.04	52272.00	1.00		
407.18265	3291352.8	1.92	52169.80	1.00		



NL:
1.72E8
FRG-11-0812#101-138
RT: 0.44992-0.61483
AV: 38 T: FTMS + p ESI
Full lock ms
[50.0000-750.0000]

NL:
7.83E5
C₂₀H₂₀N₆O₂+H:
C₂₀H₂₁N₆O₂
pa Chrg 1

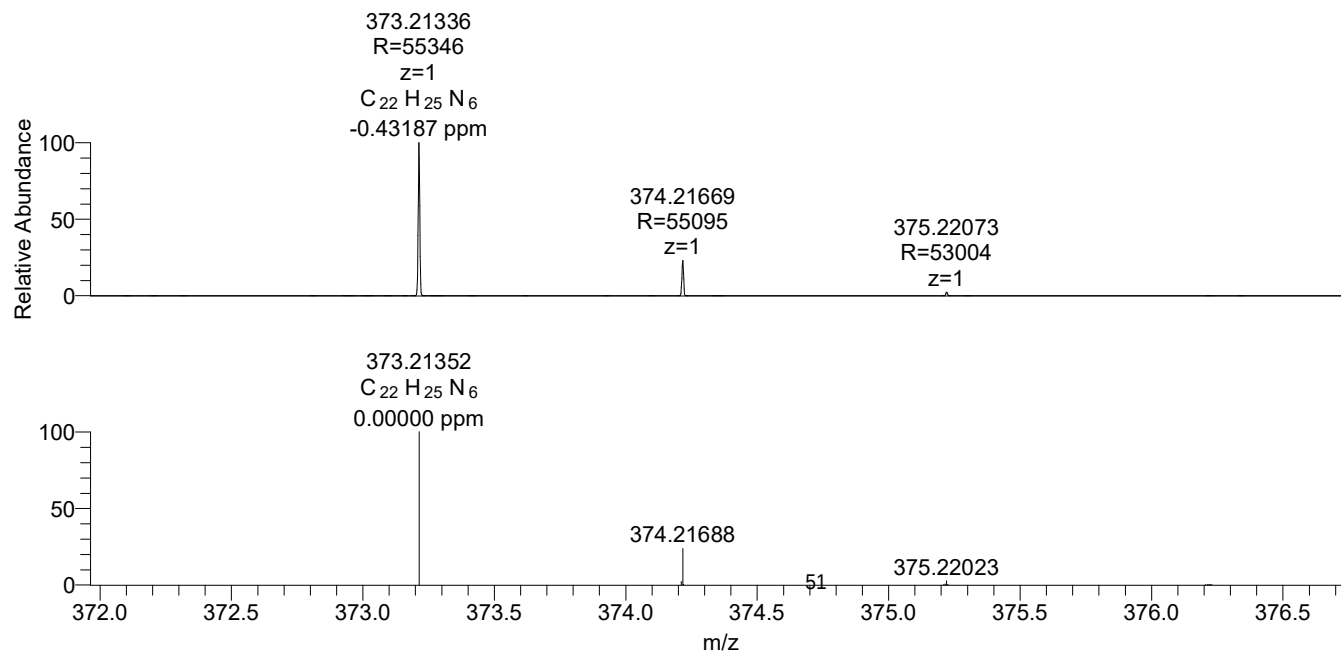
FRG-12-0812 #91-140 RT: 0.40535-0.62375 AV: 50 NL: 1.21E9
T: FTMS + p ESI Full ms [50.0000-750.0000]



FRG-12-0812#91-140 RT: 0.40535-0.62375 AV: 50
T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z= 338.51753-419.33520

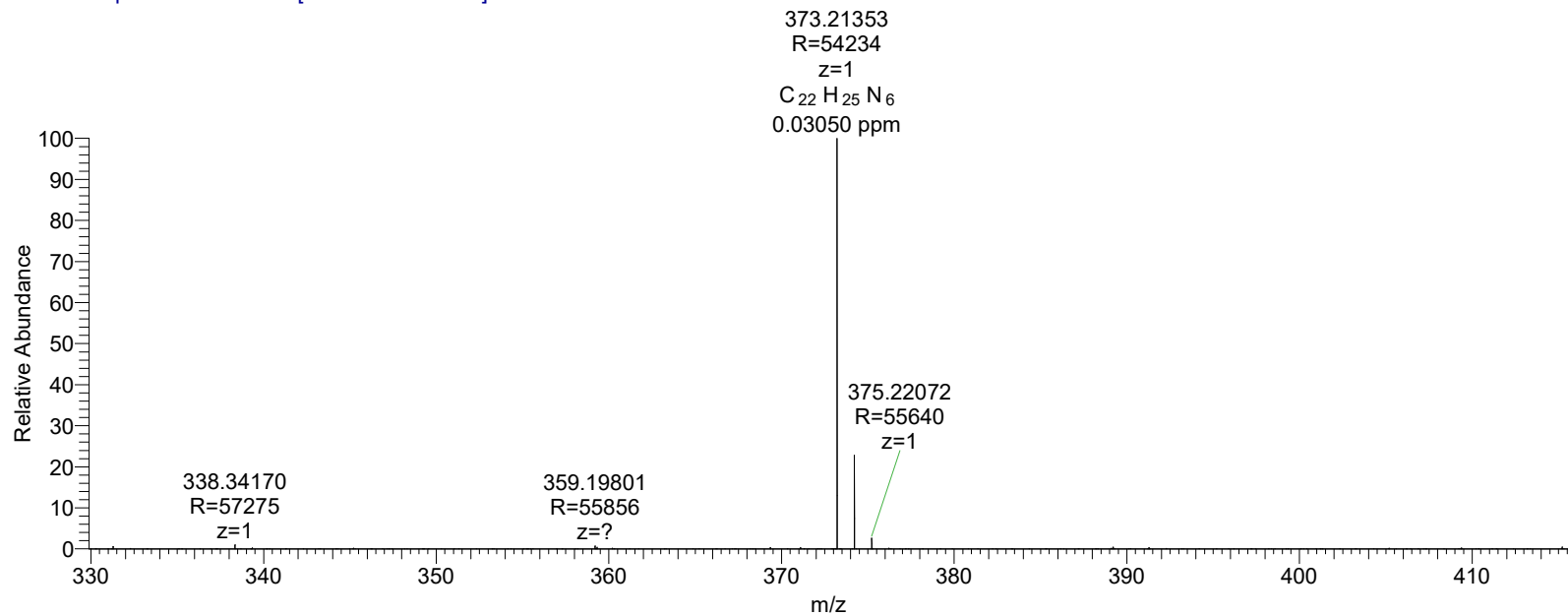
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
339.34504	3398622.3	0.28	56157.29	1.00	-0.43	C ₂₂ H ₂₅ N ₆
345.20747	2502963.3	0.20	56765.80	1.00		
359.31540	2246370.5	0.18	59568.80	1.00		
373.21336	1226886656.0	100.00	55345.56	1.00		
374.21669	280180864.0	22.84	55094.97	1.00		
375.22073	29588268.0	2.41	53003.95	1.00		
376.22454	2035046.0	0.17	52272.58	1.00		



NL:
1.21E9
FRG-12-0812#91-140
RT: 0.40535-0.62375
AV: 50 T: FTMS + p
ESI Full ms
[50.0000-750.0000]

NL:
7.70E5
C₂₂H₂₄N₆+H:
C₂₂H₂₅N₆
pa Chrg 1

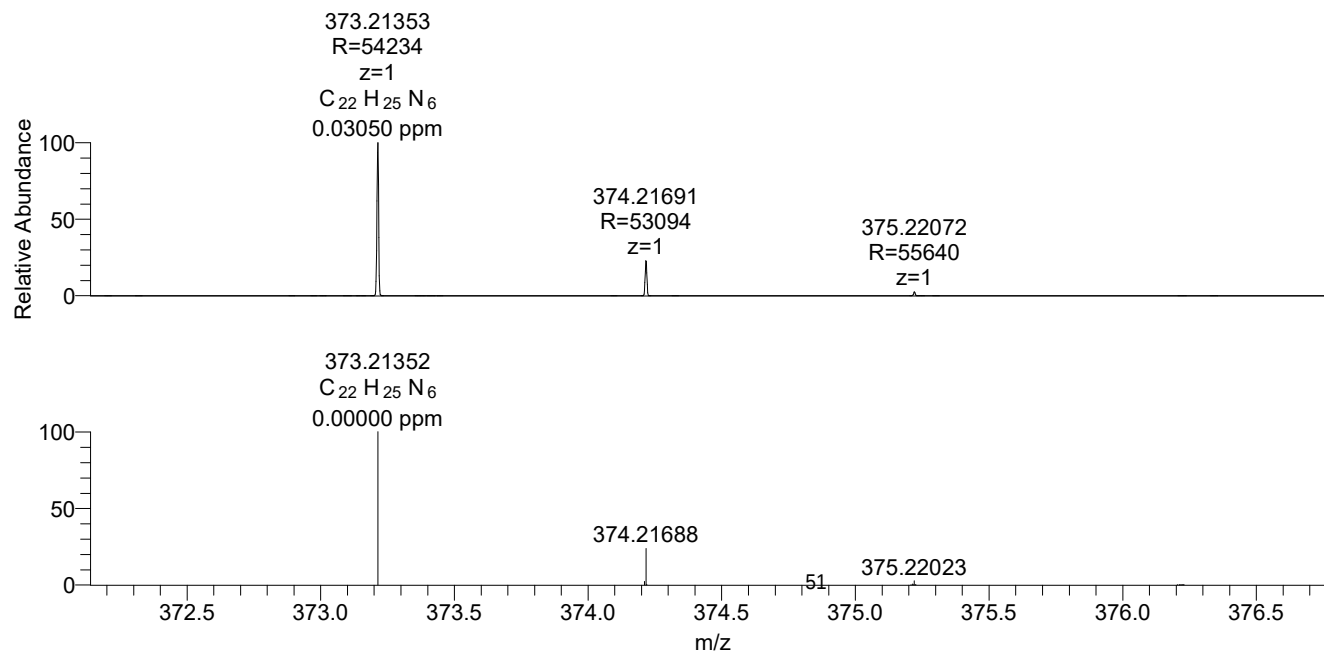
FRG-13-0812 #104-143 RT: 0.46313-0.63695 AV: 40 NL: 6.71E8
T: FTMS + p ESI Full lock ms [50.0000-750.0000]



FRG-13-0812 #104-143 RT: 0.46313-0.63695 AV: 40
T: FTMS + p ESI Full lock ms [50.0000-750.0000]

m/z = 328.54054-452.73826

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
331.28420	4134396.8	0.61	57550.72	1.00	0.03	C ₂₂ H ₂₅ N ₆
338.34170	7247042.0	1.06	57275.41	1.00		
359.19801	5040519.5	0.74	55855.93	0.00		
373.21353	681314752.0	100.00	54233.77	1.00		
374.21691	157715520.0	23.15	53093.59	1.00		
375.22072	17531518.0	2.57	55640.39	1.00		
440.23075	8646957.0	1.27	50089.94	1.00		

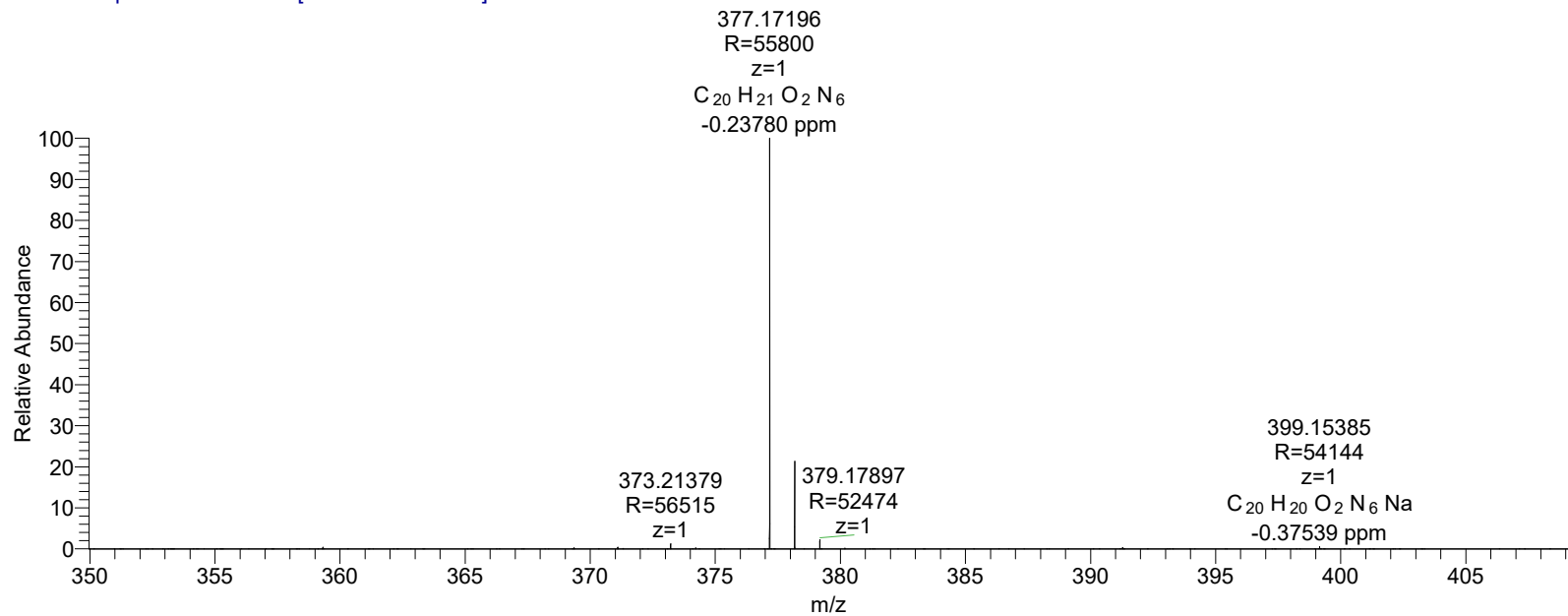


NL:
6.71E8
FRG-13-0812#104-143
RT: 0.46313-0.63695
AV: 40 T: FTMS + p ESI
Full lock ms
[50.0000-750.0000]

NL:
7.70E5
C₂₂ H₂₄ N₆ +H:
C₂₂ H₂₅ N₆
pa Chrg 1

FRG-14-0812#90-143 RT: 0.40090-0.63712 AV: 54 NL: 5.23E8

T: FTMS + p ESI Full lock ms [50.0000-750.0000]

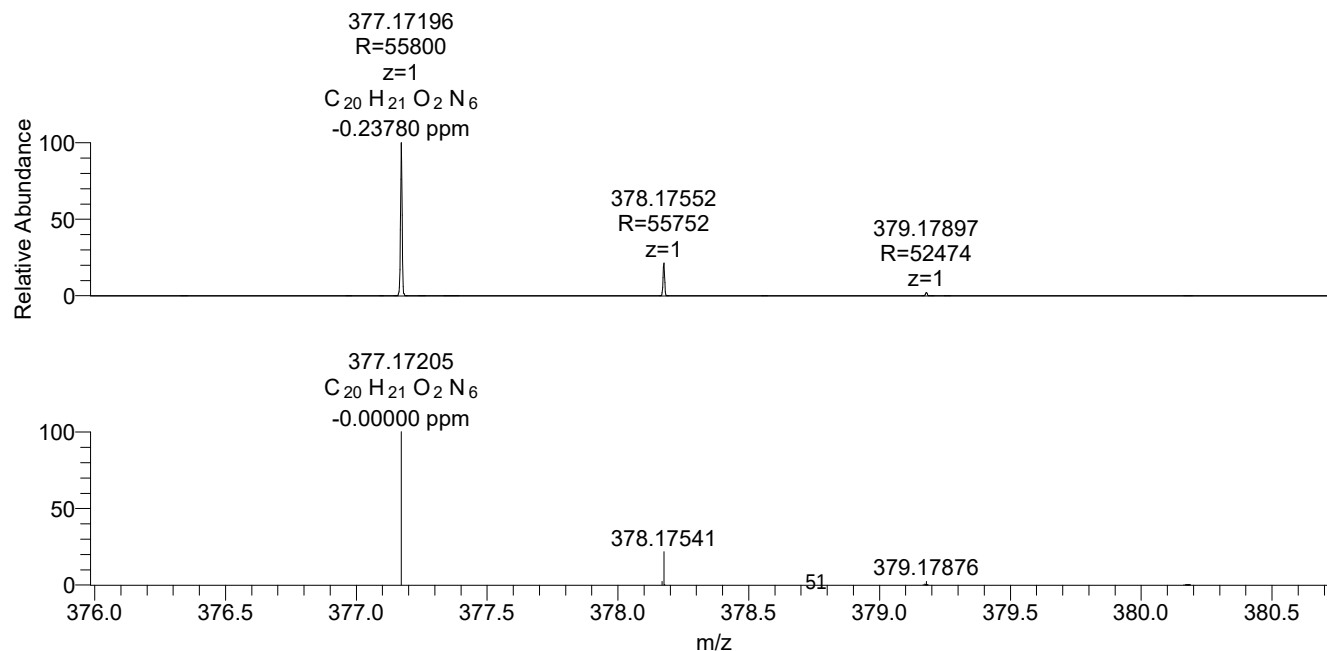


FRG-14-0812#90-143 RT: 0.40090-0.63712 AV: 54

T: FTMS + p ESI Full lock ms [50.0000-750.0000]

m/z= 349.95971-409.11366

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
359.31565	2307186.3	0.44	57027.27	1.00		
371.10139	2382765.0	0.45	55843.56	1.00		
373.21379	6628010.0	1.26	56514.50	1.00		
377.17196	524946592.0	100.00	55799.70	1.00	-0.24	C ₂₀ H ₂₁ O ₂ N ₆
378.17552	112278016.0	21.39	55751.68	1.00		
379.17897	11807291.0	2.25	52473.66	1.00		
399.15385	3193967.0	0.61	54143.61	1.00	-0.38	C ₂₀ H ₂₀ O ₂ N ₆ Na

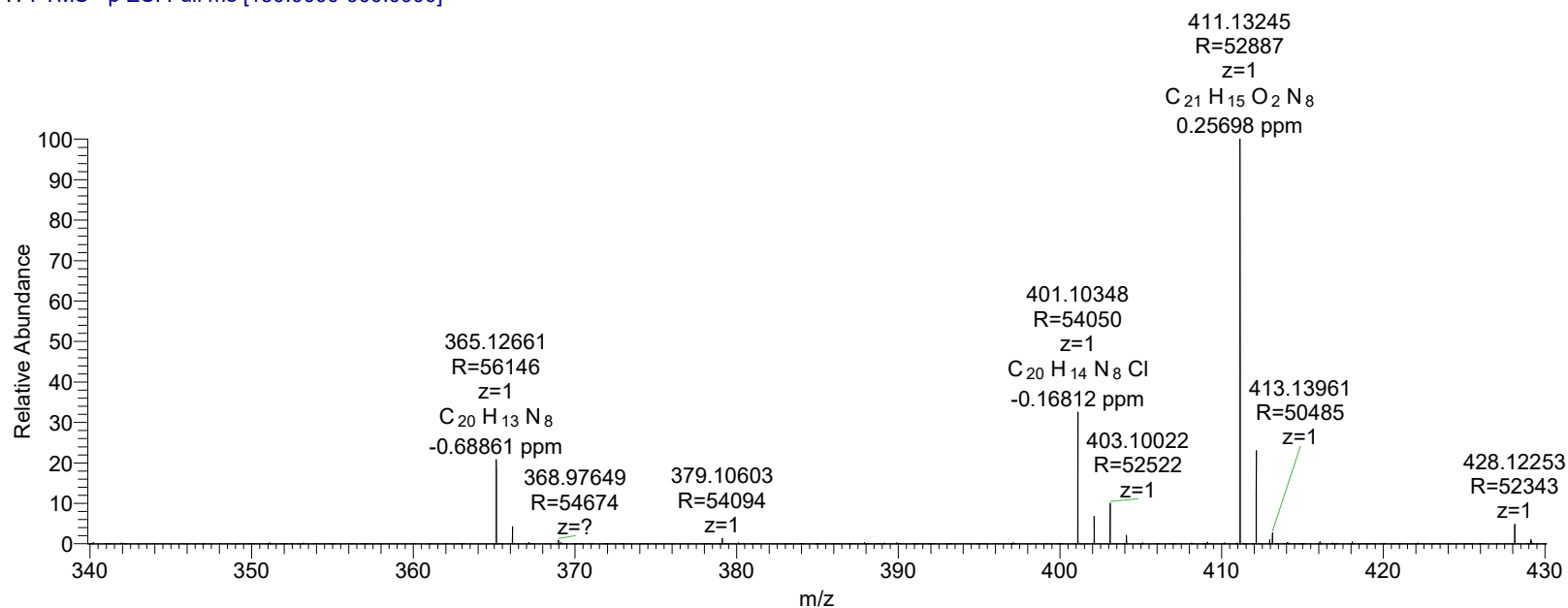


NL:
5.23E8
FRG-14-0812#90-143
RT: 0.40090-0.63712
AV: 54 T: FTMS + p ESI
Full lock ms
[50.0000-750.0000]

NL:
7.83E5
C₂₀H₂₀N₆O₂+H:
C₂₀H₂₁N₆O₂
pa Chrg 1

FRG-15-0813 #164-232 RT: 1.05727-1.36036 AV: 69 NL: 1.69E7

T: FTMS - p ESI Full ms [150.0000-900.0000]

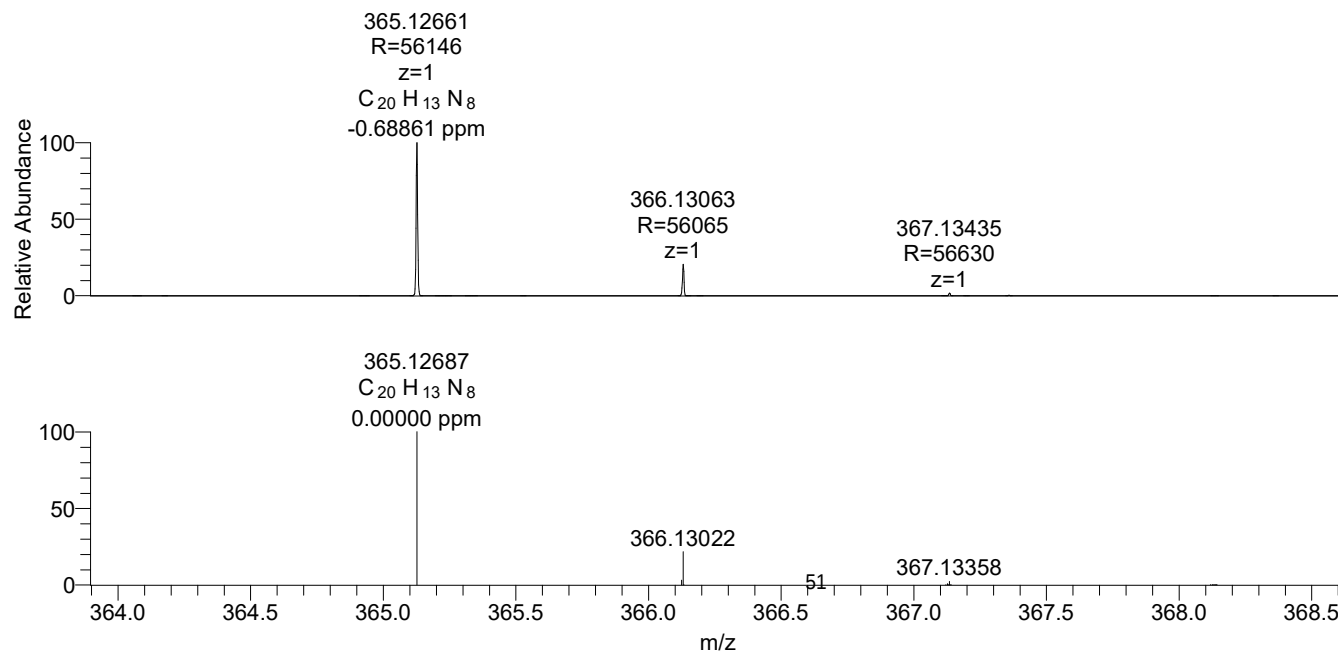


FRG-15-0813#164-232 RT: 1.05727-1.36036 AV: 69

T: FTMS - p ESI Full ms [150.0000-900.0000]

m/z= 339.84802-430.07991

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
365.12661	3519152.8	20.79	56146.37	1.00	-0.69	C ₂₀ H ₁₃ N ₈
366.13063	724118.9	4.28	56065.25	1.00		
401.10348	5493875.0	32.45	54049.54	1.00	-0.17	C ₂₀ H ₁₄ N ₈ Cl
402.10737	1155609.1	6.83	53281.16	1.00		
403.10022	1719388.4	10.16	52521.64	1.00		
411.13245	16929640.0	100.00	52886.67	1.00	0.26	C ₂₁ H ₁₅ O ₂ N ₈
412.13625	3895569.0	23.01	53290.93	1.00		
413.13961	457768.2	2.70	50484.61	1.00		
428.12253	824384.5	4.87	52342.58	1.00		

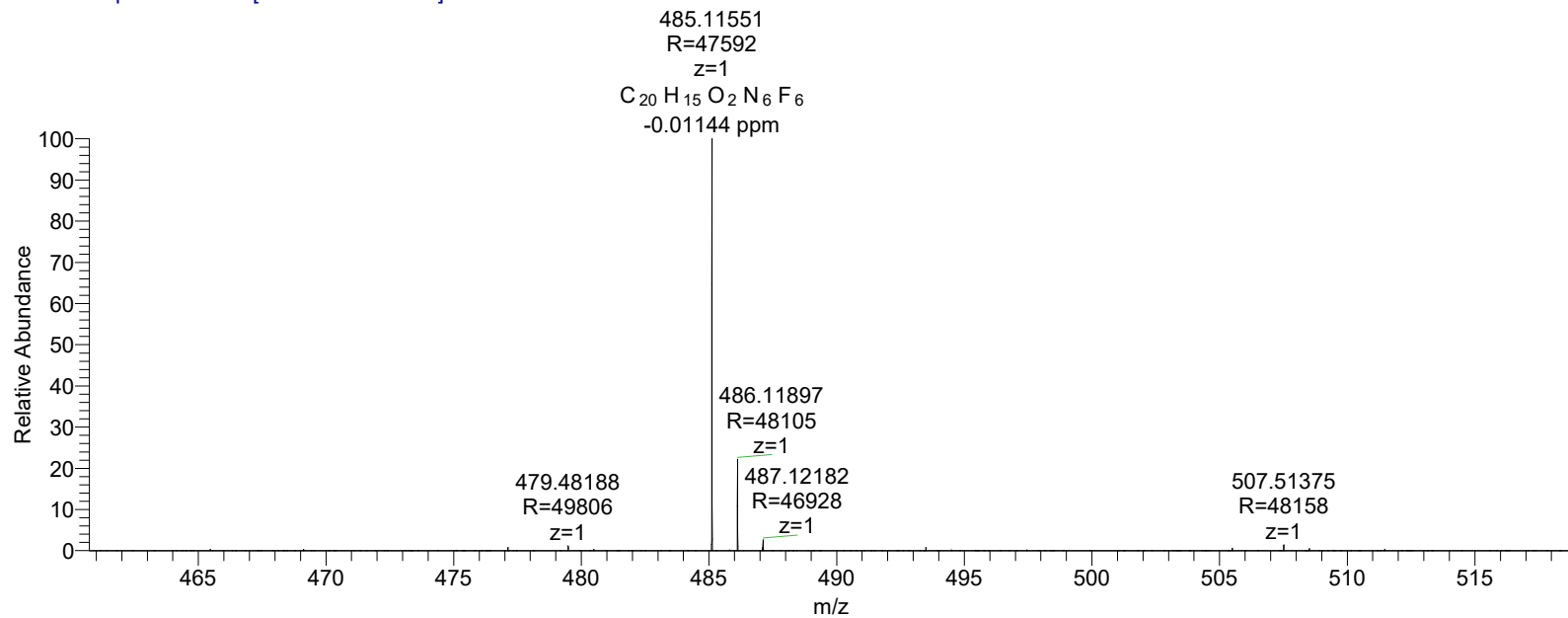


NL:
3.51E6
FRG-15-0813#164-232
RT: 1.05727-1.36036
AV: 69 T: FTMS - p ESI
Full ms
[150.0000-900.0000]

NL:
7.82E5
C₂₀H₁₃N₈:
C₂₀H₁₃N₈
pa Chrg -1

FRG-16-0812 #34-124 RT: 0.15114-0.55227 AV: 91 NL: 3.74E7

T: FTMS + p ESI Full ms [50.0000-750.0000]

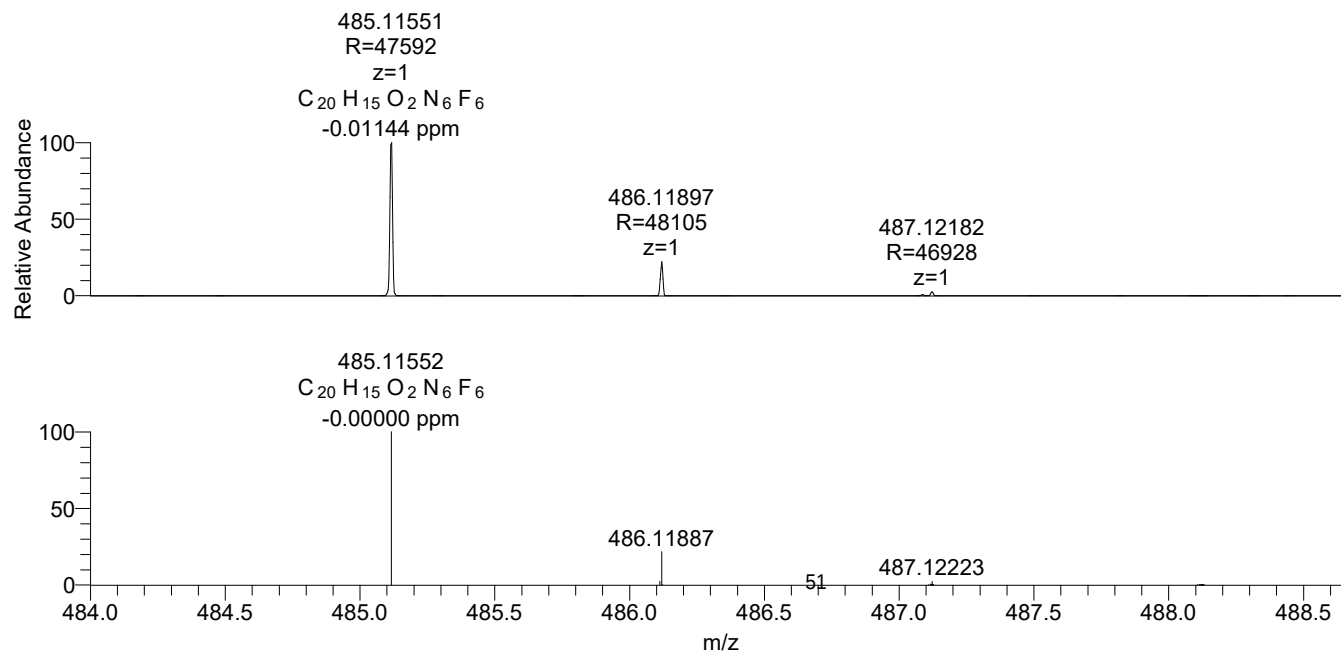


FRG-16-0812#34-124 RT: 0.15114-0.55227 AV: 91

T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z= 460.72274-518.86758

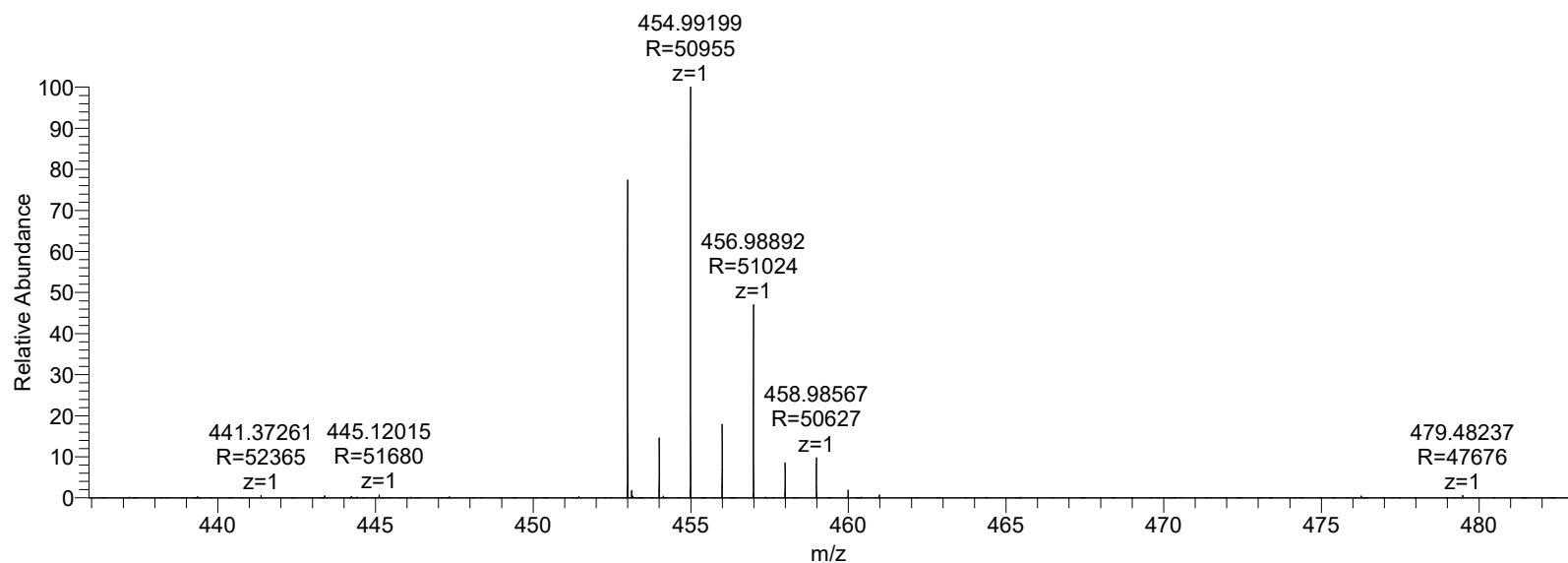
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
479.48188	458925.1	1.19	49805.91	1.00	-0.01	$C_{20}H_{15}O_2N_6F_6$
485.11551	38645320.0	100.00	47592.47	1.00		
486.11897	8323673.5	21.54	48104.86	1.00		
487.12182	995876.0	2.58	46927.53	1.00		
507.51375	531835.2	1.38	48158.02	1.00		



NL:
3.74E7
FRG-16-0812#34-124
RT: 0.15114-0.55227
AV: 91 T: FTMS + p
ESI Full ms
[50.0000-750.0000]

NL:
7.84E5
 $C_{20}H_{14}F_6N_6O_2 + H$
 $C_{20}H_{15}F_6N_6O_2$
pa Chrg 1

FRG-17-0812 #88-138 RT: 0.39182-0.61466 AV: 51 NL: 5.90E7
T: FTMS + p ESI Full ms [50.0000-750.0000]

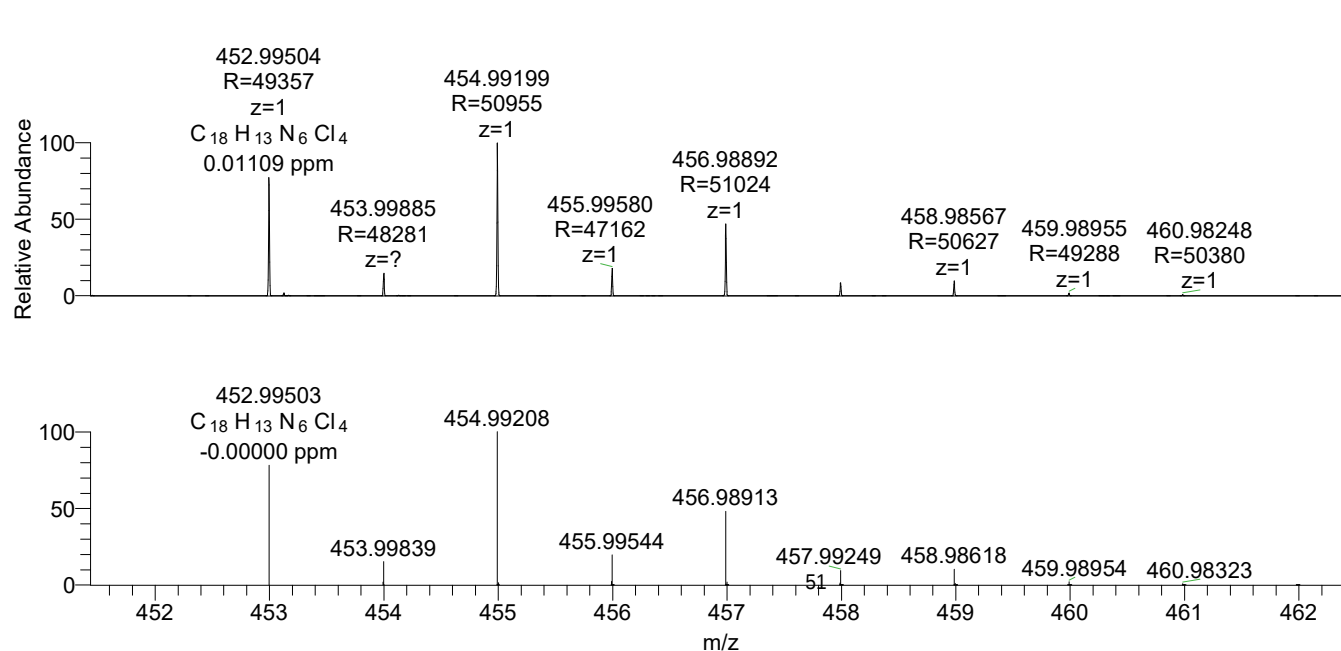


FRG-17-0812#88-138 RT: 0.39182-0.61466 AV: 51

T: FTMS + p ESI Full ms [50.0000-750.0000]

m/z = 435.91160-482.84386

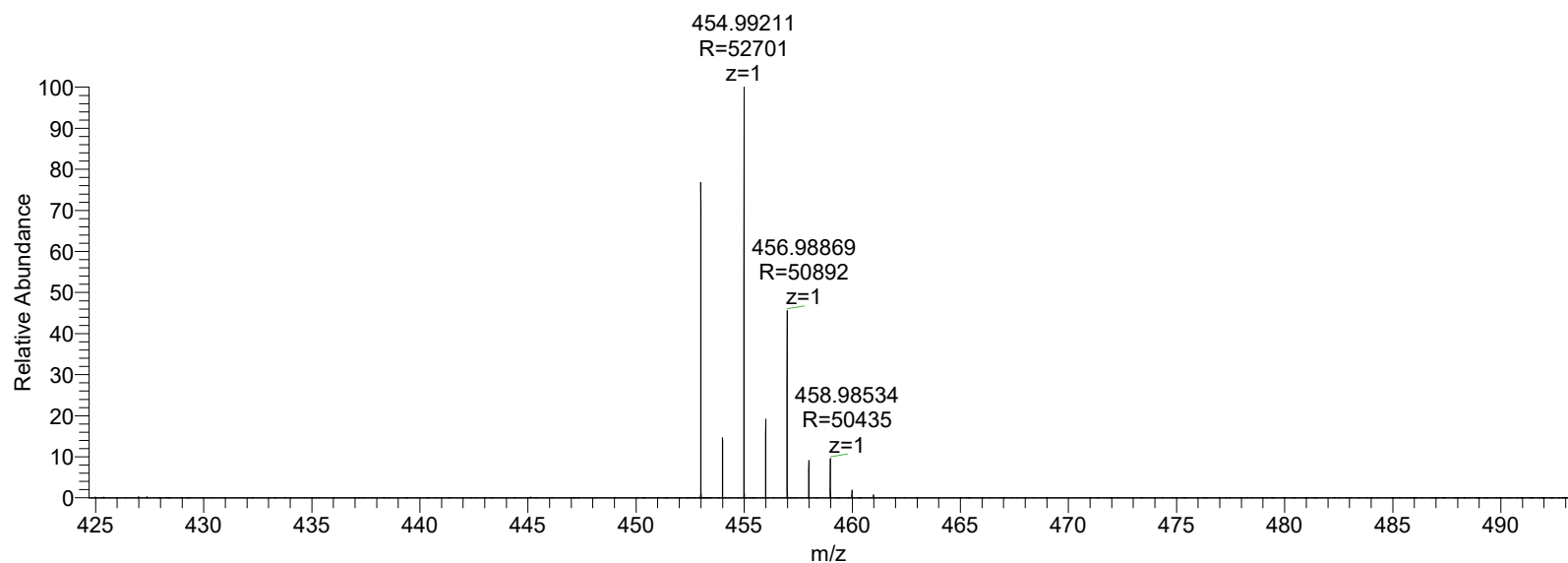
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
452.99504	46406568.0	78.69	49356.75	1.00	0.01	C ₁₈ H ₁₃ N ₆ Cl ₄
453.12562	1083604.9	1.84	48789.35	1.00		
453.99885	8767143.0	14.87	48280.71	0.00		
454.99199	58974152.0	100.00	50955.24	1.00		
455.99580	10938605.0	18.55	47161.52	1.00		
456.98892	27763572.0	47.08	51023.62	1.00		
457.99279	5142931.5	8.72	47637.54	1.00		
458.98567	5804767.0	9.84	50627.45	1.00		
459.98955	1094182.4	1.86	49287.75	1.00		



NL:
5.90E7
FRG-17-0812#88-138
RT: 0.39182-0.61466
AV: 51 T: FTMS + p
ESI Full ms
[50.0000-750.0000]

NL:
3.39E5
C₁₈H₁₂Cl₄N₆+H:
C₁₈H₁₃Cl₄N₆
pa Chrg 1

FRG-18-0812 #1-72 RT: 0.00407-0.32053 AV: 72 NL: 4.99E8
T: FTMS + p ESI Full ms [150.0000-750.0000]

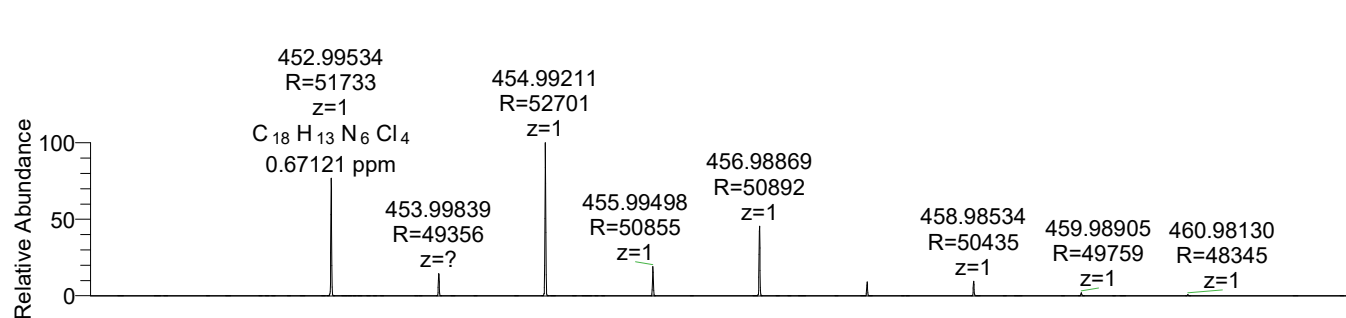


FRG-18-0812#1-72 RT: 0.00407-0.32053 AV: 72

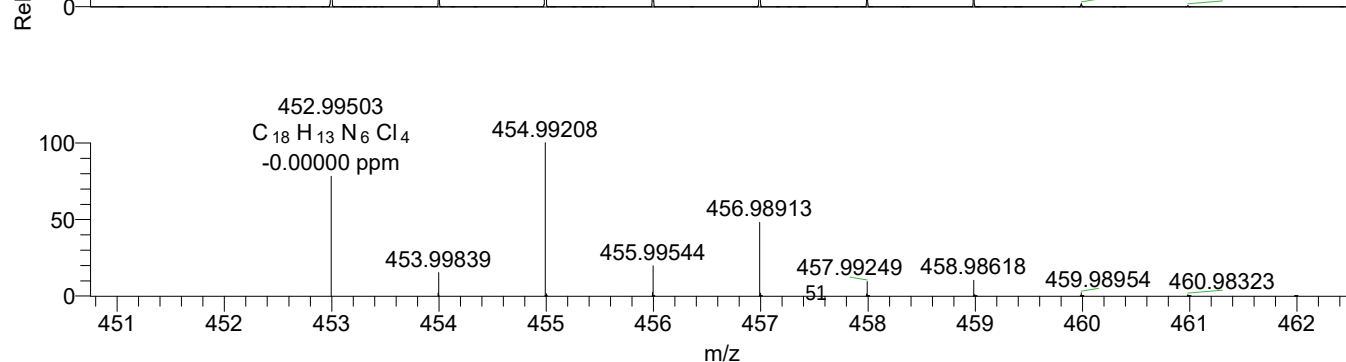
T: FTMS + p ESI Full ms [150.0000-750.0000]

m/z = 424.68972-493.13842

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
452.99534	389217344.0	77.76	51733.34	1.00	0.67	C ₁₈ H ₁₃ N ₆ Cl ₄
453.99839	74481576.0	14.88	49356.25	0.00		
454.99211	500554240.0	100.00	52701.42	1.00		
455.99498	95648288.0	19.11	50855.40	1.00		
456.98869	231596256.0	46.27	50891.77	1.00		
457.99192	45338800.0	9.06	50304.50	1.00		
458.98534	47740372.0	9.54	50435.32	1.00		
459.98905	9625369.0	1.92	49758.91	1.00		
460.98130	3577449.5	0.71	48344.50	1.00		



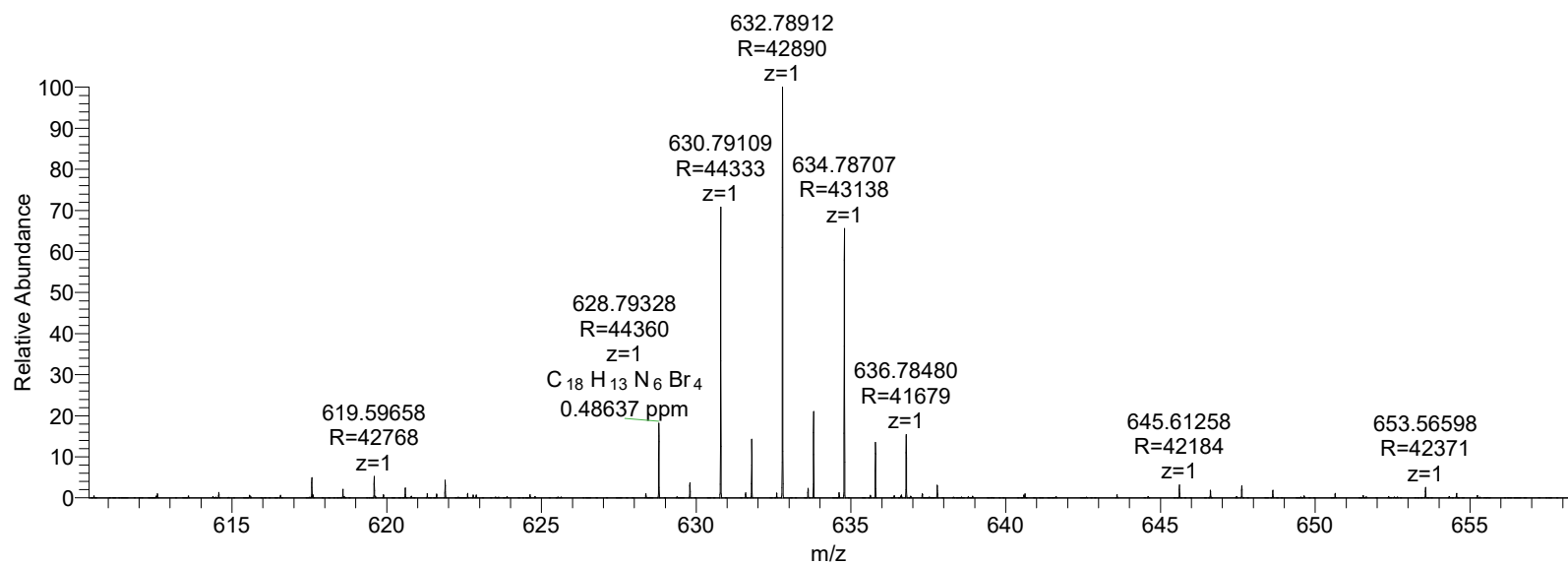
NL:
4.99E8
FRG-18-0812#1-72
RT: 0.00407-0.32053
AV: 72 T: FTMS + p
ESI Full ms
[150.0000-750.0000]



NL:
3.39E5
C₁₈H₁₂Cl₄N₆+H:
C₁₈H₁₃Cl₄N₆
pa Chrg 1

FRG-19-0812 #35-131 RT: 0.15578-0.58367 AV: 97 NL: 9.82E6

T: FTMS + p ESI Full ms [199.6000-900.0000]

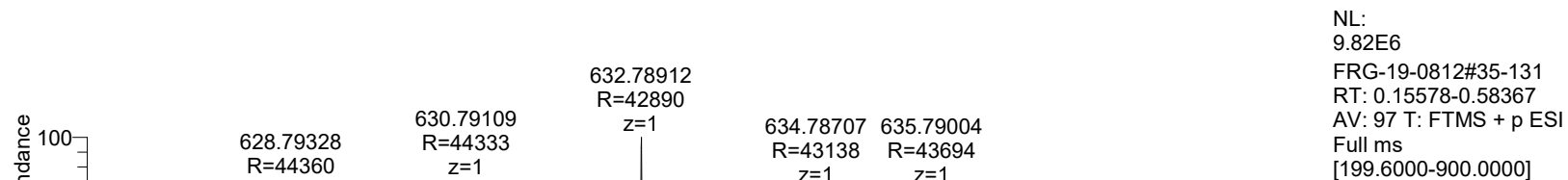


FRG-19-0812#35-131 RT: 0.15578-0.58367 AV: 97

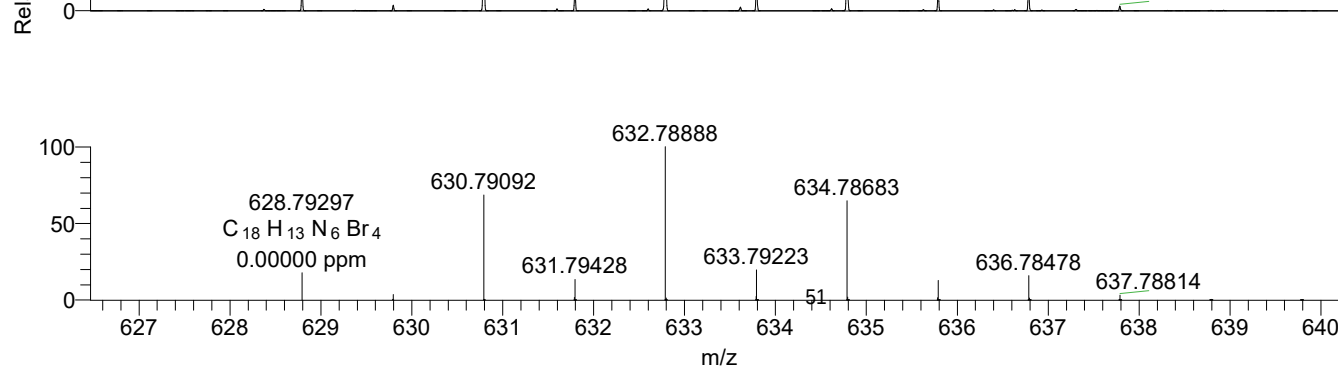
T: FTMS + p ESI Full ms [199.6000-900.0000]

m/z = 610.37695-658.19017

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
619.59658	529529.6	5.29	42767.77	1.00	0.49	C ₁₈ H ₁₃ N ₆ Br ₄
628.79328	1791044.0	17.88	44360.18	1.00		
630.79109	6956874.0	69.44	44332.73	1.00		
631.79436	1418314.1	14.16	43613.69	1.00		
632.78912	10018820.0	100.00	42889.79	1.00		
633.79203	2068909.6	20.65	43861.21	1.00		
634.78707	6500089.0	64.88	43138.05	1.00		
635.79004	1332273.8	13.30	43693.89	1.00		
636.78480	1557102.9	15.54	41679.38	1.00		

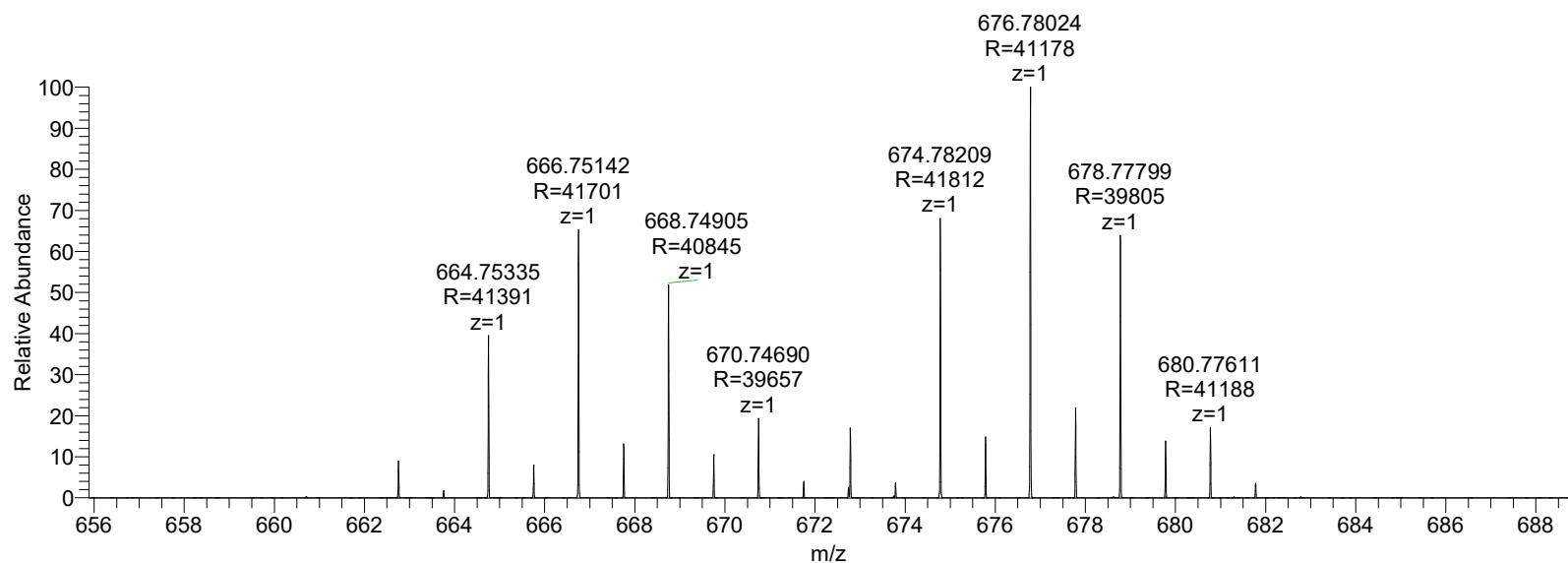


NL:
9.82E6
FRG-19-0812#35-131
RT: 0.15578-0.58367
AV: 97 T: FTMS + p ESI
Full ms
[199.6000-900.0000]



NL:
3.02E5
C₁₈ H₁₂ Br₄ N₆ +H:
C₁₈ H₁₃ Br₄ N₆
pa Chrg 1

FRG-19-0812 #155-249 RT: 0.94614-1.36511 AV: 95 NL: 5.30E5
T: FTMS - p ESI Full ms [199.6000-900.0000]

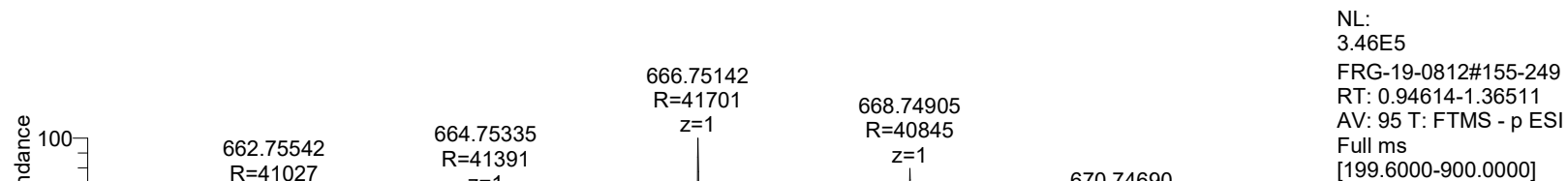


FRG-19-0812#155-249 RT: 0.94614-1.36511 AV: 95

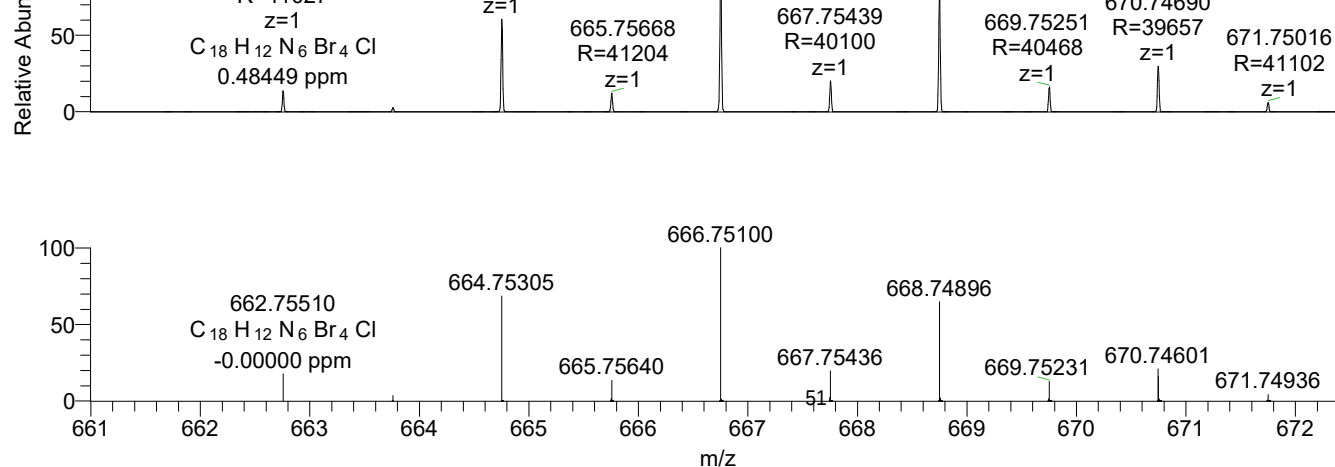
T: FTMS - p ESI Full ms [199.6000-900.0000]

m/z= 662.00257-673.50789

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
662.75542	48594.6	13.99	41026.61	1.00	0.48	C ₁₈ H ₁₂ N ₆ Br ₄ Cl
664.75335	210717.1	60.66	41391.06	1.00		
665.75668	42535.5	12.24	41203.68	1.00		
666.75142	347395.5	100.00	41700.55	1.00		
667.75439	71142.6	20.48	40100.37	1.00		
668.74905	278033.5	80.03	40844.80	1.00		
669.75251	56698.9	16.32	40467.63	1.00		
670.74690	106623.2	30.69	39657.38	1.00		
672.78426	90737.2	26.12	41612.05	1.00	0.55	C ₁₉ H ₁₃ O ₂ N ₆ Br ₄

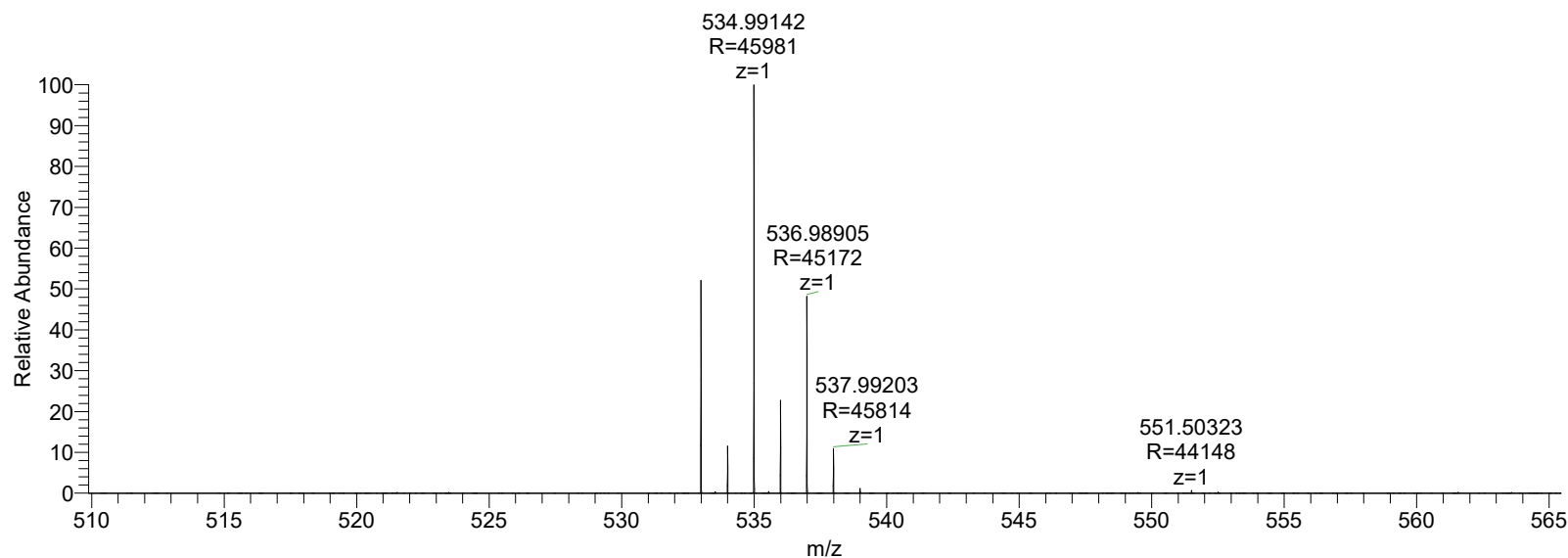


NL:
3.46E5
FRG-19-0812#155-249
RT: 0.94614-1.36511
AV: 95 T: FTMS - p ESI
Full ms
[199.6000-900.0000]



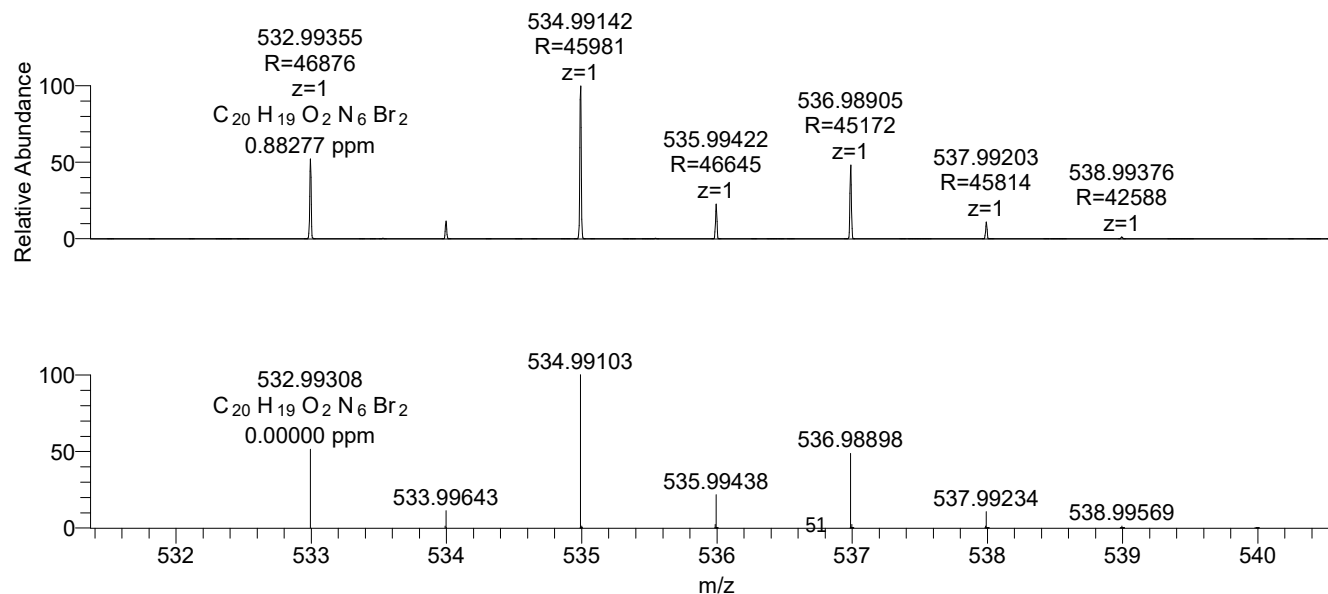
NL:
2.29E5
C₁₈H₁₂Br₄N₆Cl:
C₁₈H₁₂Br₄N₆Cl₁
pa Chrg -1

FRG-20-0813 #1-67 RT: 0.00424-0.29841 AV: 67 NL: 3.39E8
T: FTMS + p ESI Full ms [120.0000-900.0000]



FRG-20-0813#1-67 RT: 0.00424-0.29841 AV: 67
T: FTMS + p ESI Full ms [120.0000-900.0000]
m/z = 509.88084-565.43774

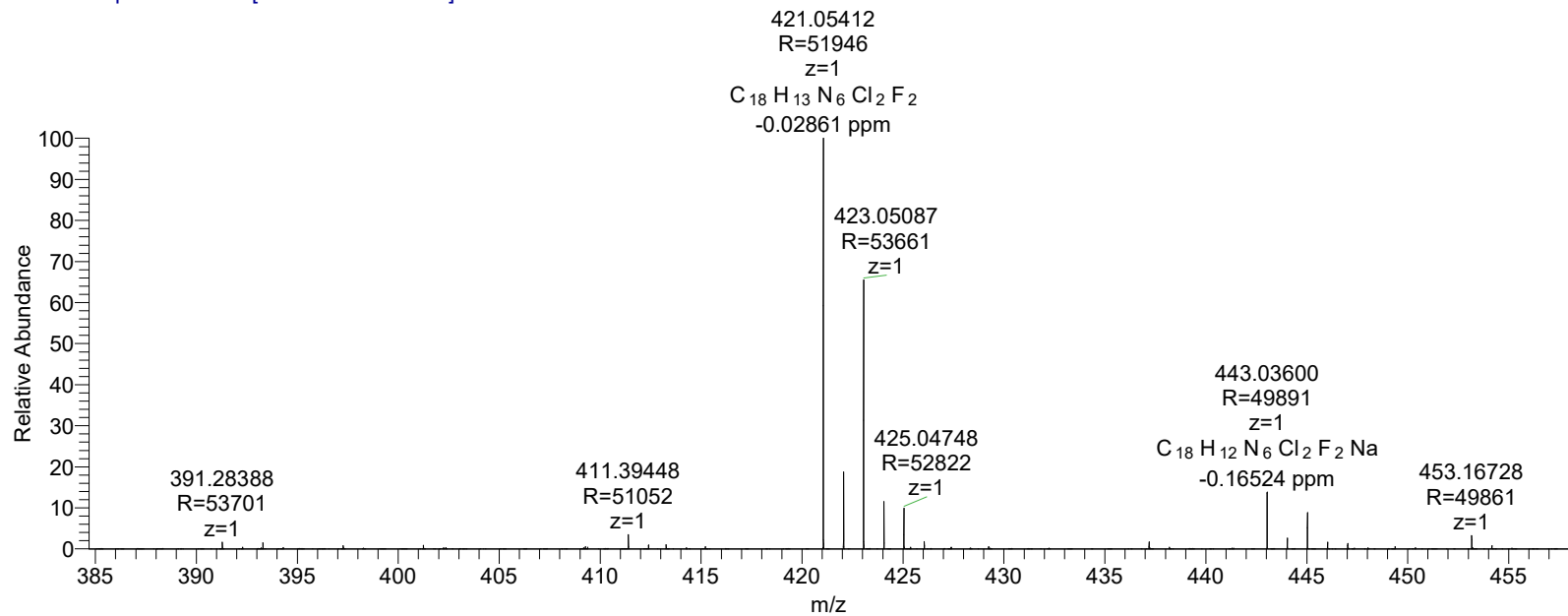
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
532.99355	177064528.0	51.63	46876.05	1.00	0.88	C ₂₀ H ₁₉ O ₂ N ₆ Br ₂
533.99660	39346520.0	11.47	46329.97	1.00		
534.99142	342933152.0	100.00	45980.63	1.00		
535.99422	77285992.0	22.54	46644.80	1.00		
536.98905	165912208.0	48.38	45171.51	1.00		



NL:
3.39E8
FRG-20-0813#1-67 RT:
0.00424-0.29841 AV:
67 T: FTMS + p ESI Full
ms [120.0000-900.0000]

NL:
3.92E5
C₂₀H₁₈Br₂N₆O₂+H:
C₂₀H₁₉Br₂N₆O₂
pa Chrg 1

FRG-21-0813 #56-137 RT: 0.24936-0.61038 AV: 82 NL: 3.80E8
T: FTMS + p ESI Full ms [100.0000-750.0000]

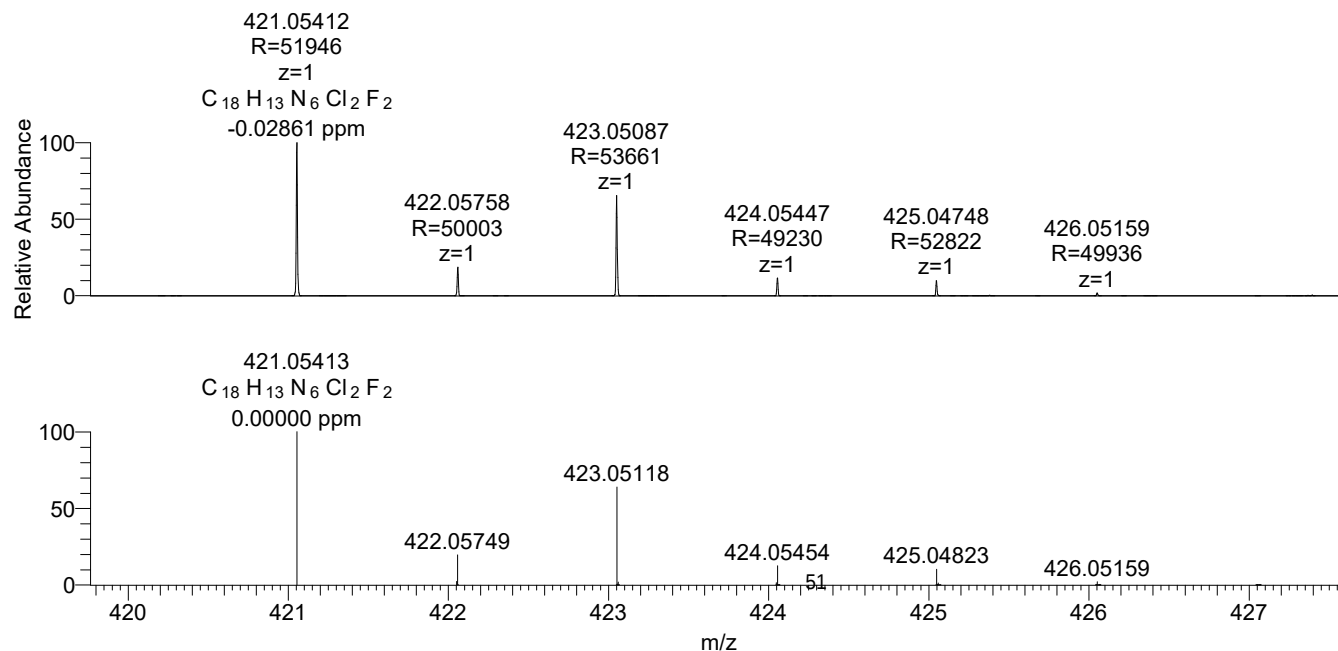


FRG-21-0813#56-137 RT: 0.24936-0.61038 AV: 82

T: FTMS + p ESI Full ms [100.0000-750.0000]

m/z = 384.67677-457.97167

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
411.39448	13494488.0	3.50	51052.38	1.00		
421.05412	385939840.0	100.00	51945.68	1.00	-0.03	$C_{18}H_{13}N_6Cl_2F_2$
422.05758	72422816.0	18.77	50003.50	1.00		
423.05087	248854336.0	64.48	53660.93	1.00		
424.05447	45255952.0	11.73	49229.61	1.00		
425.04748	38481948.0	9.97	52821.51	1.00		
443.03600	54118128.0	14.02	49890.52	1.00	-0.17	$C_{18}H_{12}N_6Cl_2F_2Na$
445.03294	34360360.0	8.90	51230.09	1.00		
453.16728	12779475.0	3.31	49861.33	1.00		

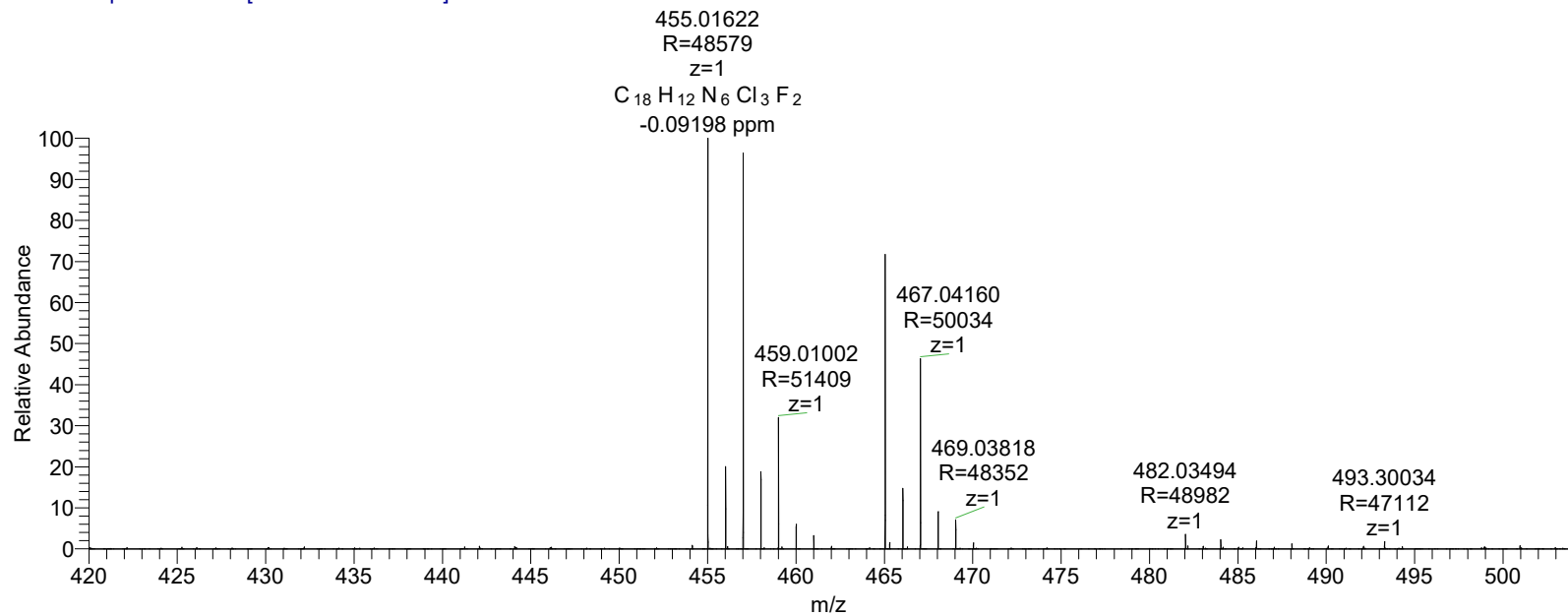


NL:
3.80E8
FRG-21-0813#56-137
RT: 0.24936-0.61038
AV: 82 T: FTMS + p ESI
Full ms
[100.0000-750.0000]

NL:
4.62E5
 $C_{18}H_{12}Cl_2F_2N_6+H$:
 $C_{18}H_{13}Cl_2F_2N_6$
pa Chrg 1

FRG-21-0813 #162-223 RT: 0.94986-1.22174 AV: 62 NL: 4.53E7

T: FTMS - p ESI Full ms [100.0000-750.0000]

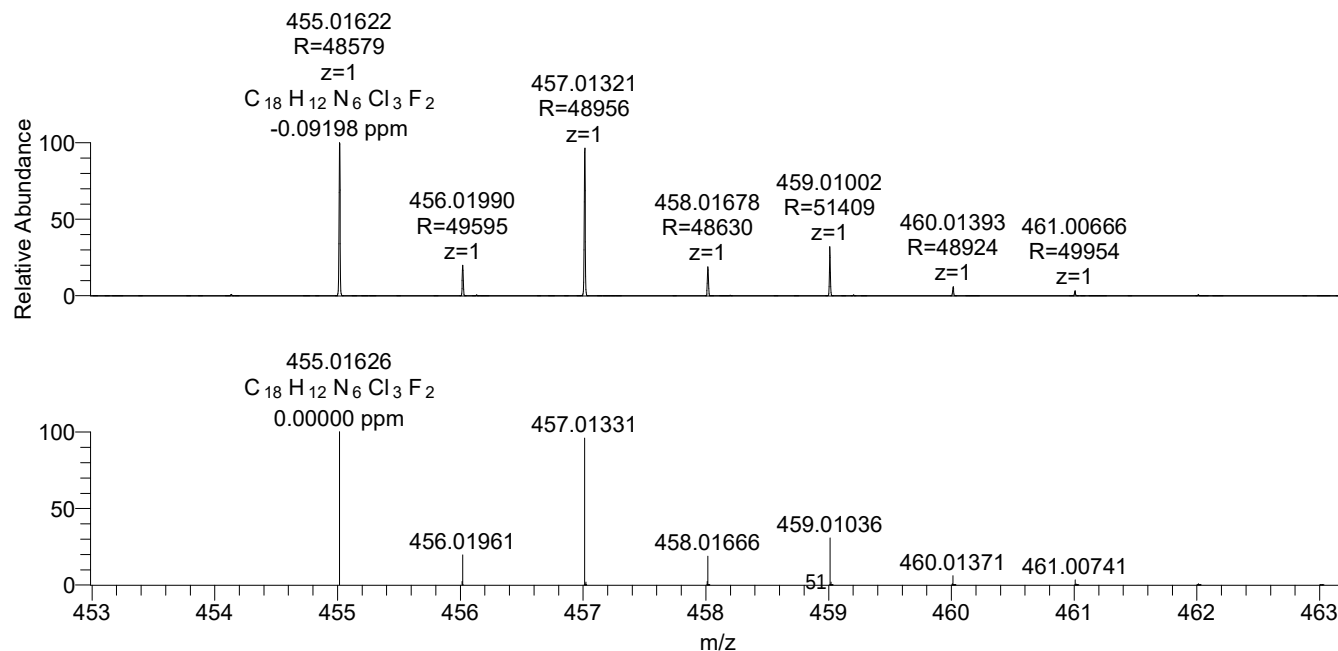


FRG-21-0813#162-223 RT: 0.94986-1.22174 AV: 62

T: FTMS - p ESI Full ms [100.0000-750.0000]

m/z = 419.99797-503.71422

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
455.01622	47062796.0	100.00	48579.24	1.00	-0.09	C ₁₈ H ₁₂ N ₆ Cl ₃ F ₂
456.01990	9087448.0	19.31	49595.46	1.00		
457.01321	44928648.0	95.47	48956.18	1.00		
458.01678	8603462.0	18.28	48629.99	1.00		
459.01002	14537227.0	30.89	51409.06	1.00		
465.04458	33229140.0	70.61	49204.10	1.00	-1.03	C ₁₉ H ₁₃ O ₂ N ₆ Cl ₂ F ₂
466.04837	6702257.0	14.24	48784.22	1.00		
467.04160	21388400.0	45.45	50033.74	1.00		
468.04536	4241757.0	9.01	46739.98	1.00		

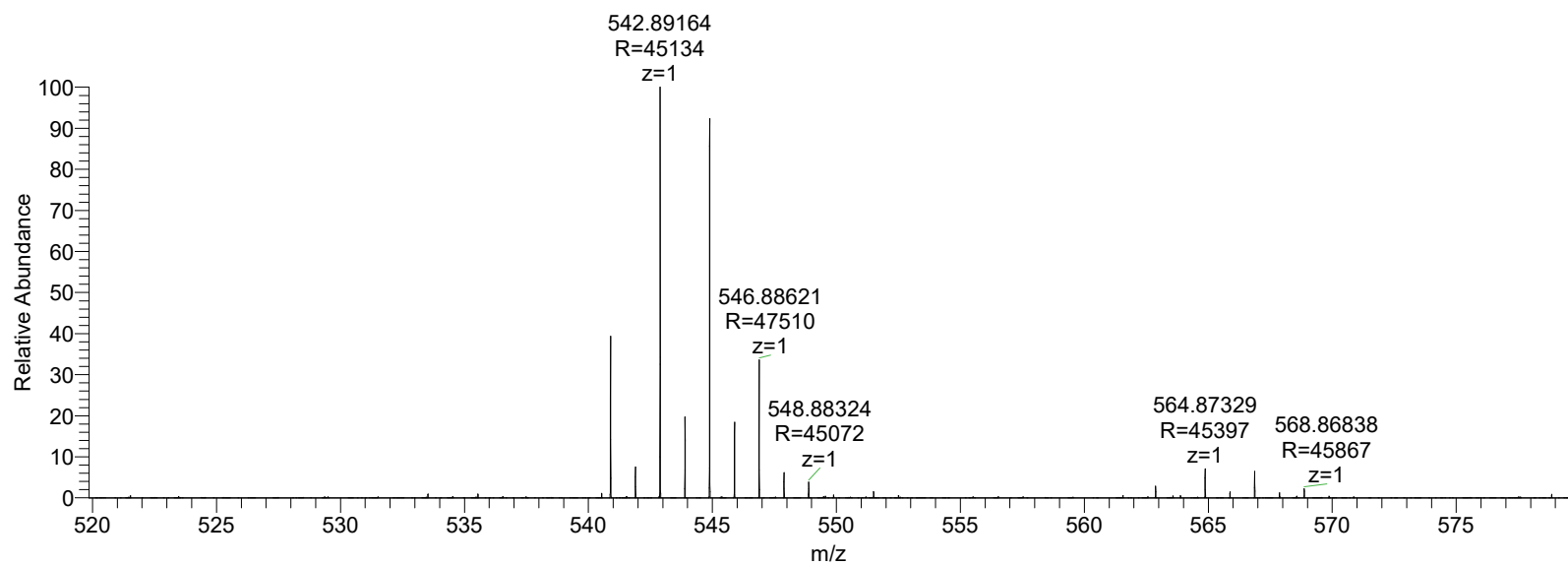


NL:
4.53E7
FRG-21-0813#162-223
RT: 0.94986-1.22174
AV: 62 T: FTMS - p ESI
Full ms
[100.0000-750.0000]

NL:
3.50E5
C₁₈H₁₂Cl₃F₂N₆:
C₁₈H₁₂Cl₃F₂N₆
pa Chrg -1

FRG-22-0813 #10-108 RT: 0.04434-0.48112 AV: 99 NL: 1.73E8

T: FTMS + p ESI Full ms [100.0000-750.0000]

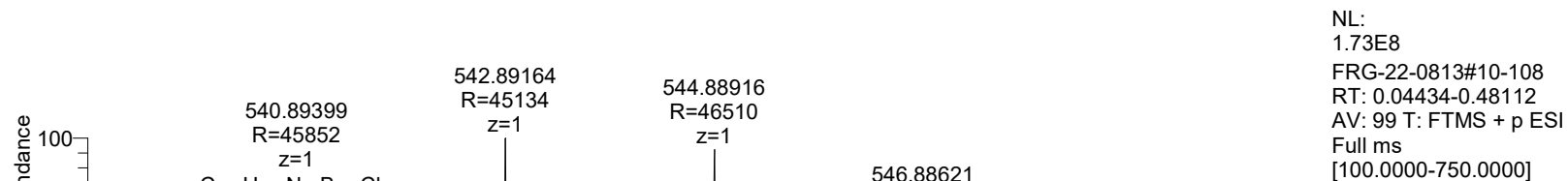


FRG-22-0813#10-108 RT: 0.04434-0.48112 AV: 99

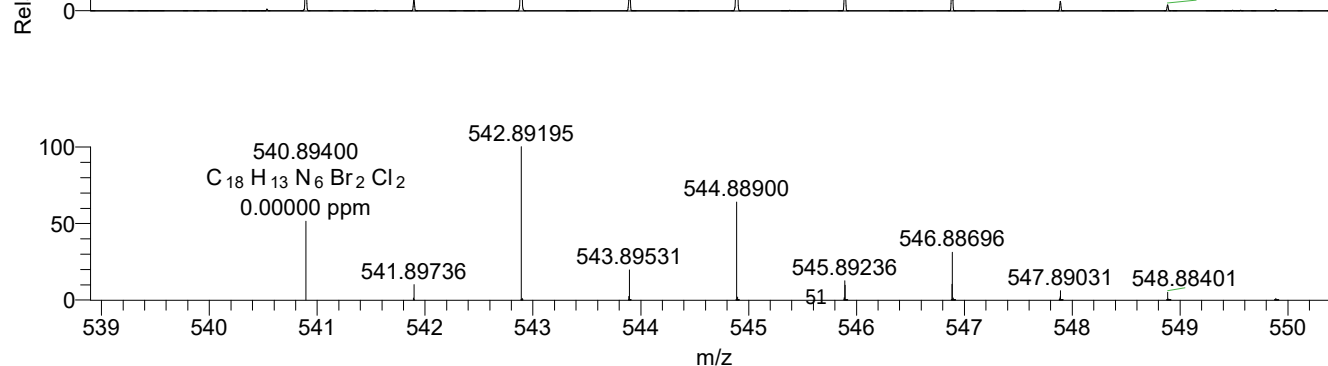
T: FTMS + p ESI Full ms [100.0000-750.0000]

m/z= 537.24619-561.33531

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
540.89399	69765568.0	38.90	45851.80	1.00	-0.02	C ₁₈ H ₁₃ N ₆ Br ₂ Cl ₂
541.89734	13519376.0	7.54	43966.14	1.00		
542.89164	179333888.0	100.00	45133.63	1.00		
543.89489	35232748.0	19.65	44023.21	1.00		
544.88916	162278976.0	90.49	46510.48	1.00		
545.89233	31934442.0	17.81	45855.45	1.00		
546.88621	58168124.0	32.44	47510.02	1.00		
547.88983	10949254.0	6.11	44043.18	1.00		
548.88324	6768901.0	3.77	45072.32	1.00		



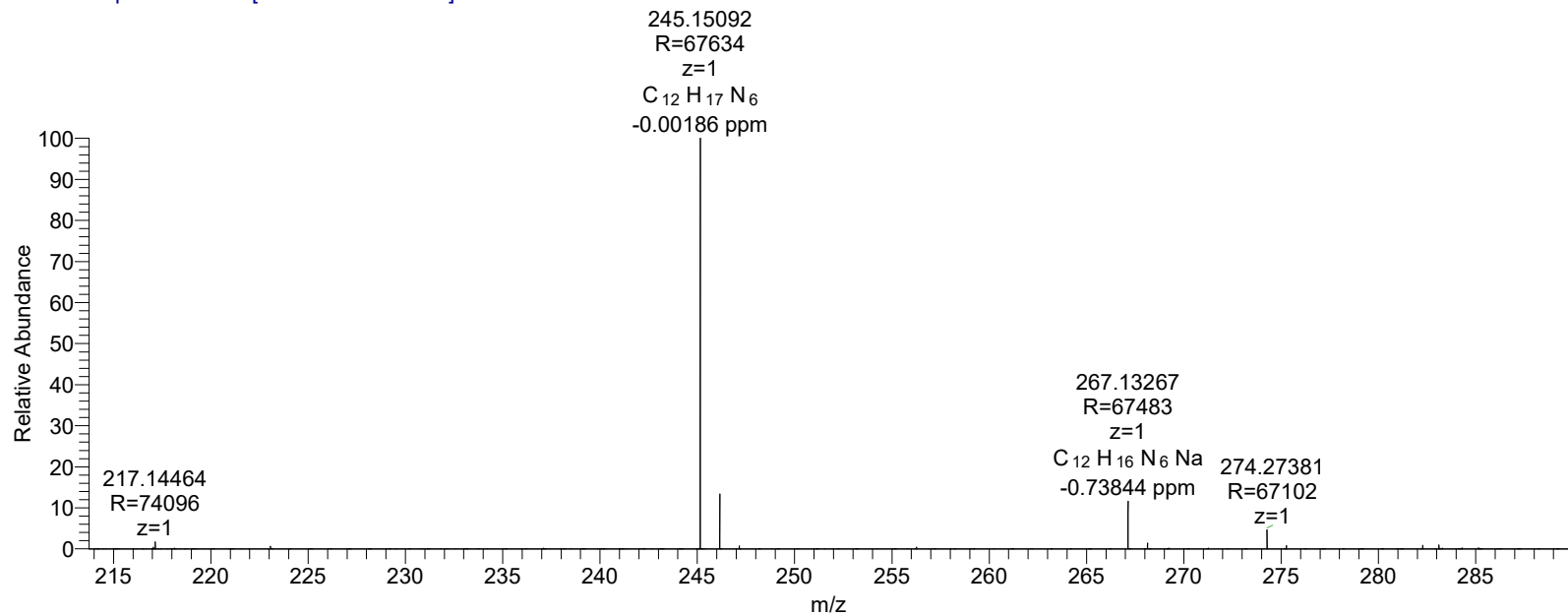
NL:
1.73E8
FRG-22-0813#10-108
RT: 0.04434-0.48112
AV: 99 T: FTMS + p ESI
Full ms
[100.0000-750.0000]



NL:
2.31E5
C₁₈H₁₂Br₂Cl₂N₆+H:
C₁₈H₁₃Br₂Cl₂N₆
pa Chrg 1

FRG-23-0813#70-139 RT: 0.31161-0.61915 AV: 70 NL: 6.05E8

T: FTMS + p ESI Full ms [150.0000-750.0000]

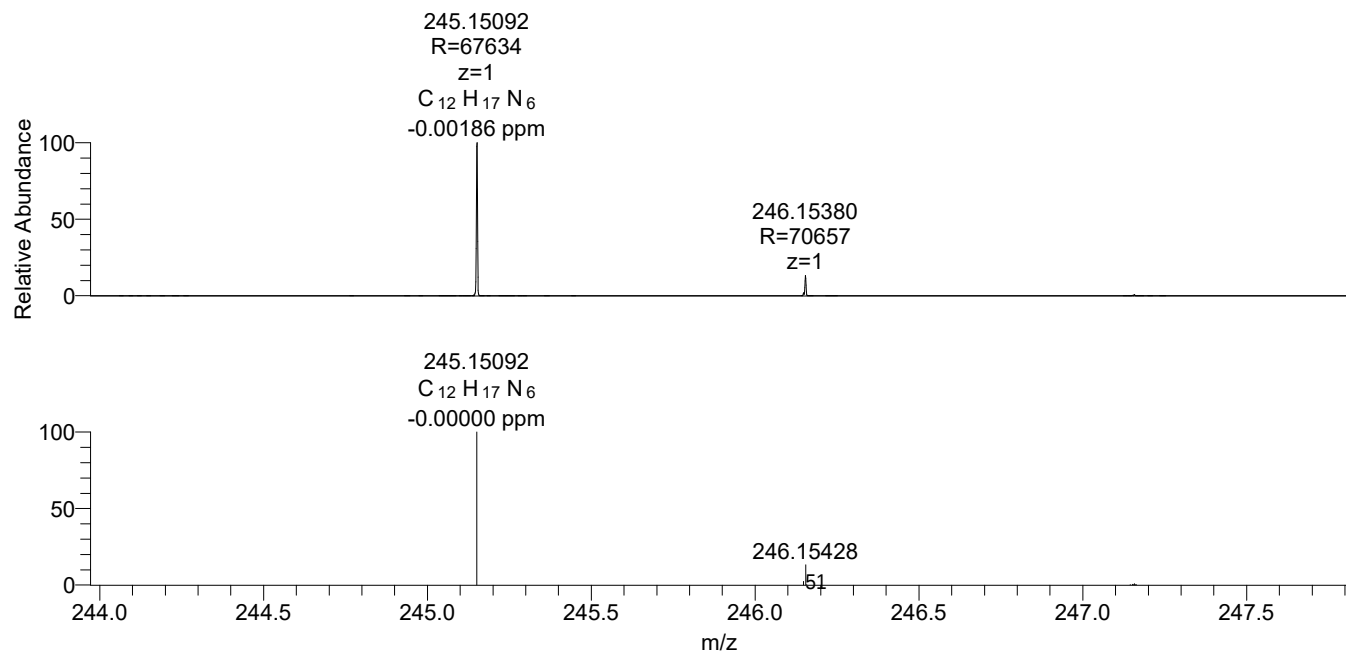


FRG-23-0813#70-139 RT: 0.31161-0.61915 AV: 70

T: FTMS + p ESI Full ms [150.0000-750.0000]

m/z = 213.73455-289.77878

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
217.14464	10415592.0	1.67	74096.06	1.00		
245.15092	622411008.0	100.00	67634.48	1.00	-0.00	$C_{12}H_{17}N_6$
246.14791	12021798.0	1.93	68983.27	1.00		
246.15380	81128824.0	13.03	70657.18	1.00		
267.13267	70605960.0	11.34	67482.65	1.00	-0.74	$C_{12}H_{16}N_6Na$
268.13603	8904766.0	1.43	66804.46	1.00		
274.27381	28321188.0	4.55	67101.64	1.00		

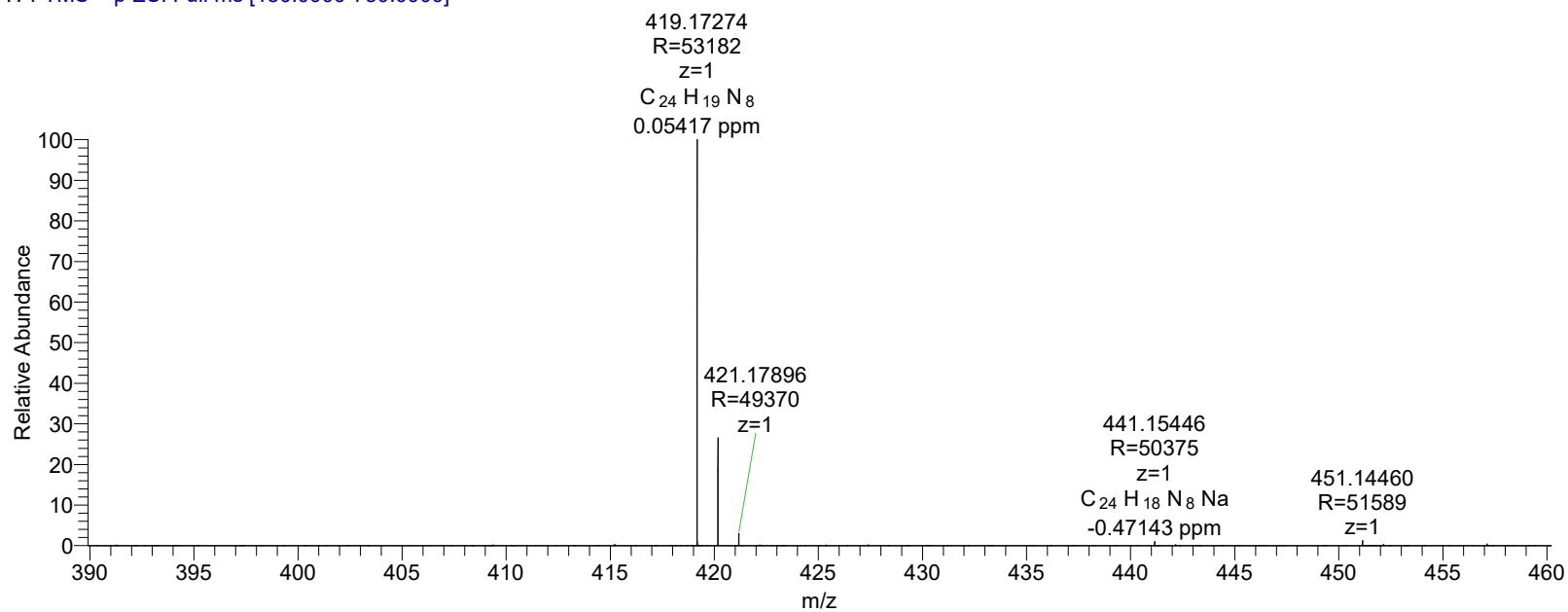


NL:
6.05E8
FRG-23-0813#70-139
RT: 0.31161-0.61915
AV: 70 T: FTMS + p ESI
Full ms
[150.0000-750.0000]

NL:
8.58E5
 $C_{12}H_{16}N_6 + H$:
 $C_{12}H_{17}N_6$
pa Chrg 1

FRG-24-0813 #33-105 RT: 0.14669-0.46761 AV: 73 NL: 7.32E8

T: FTMS + p ESI Full ms [150.0000-750.0000]

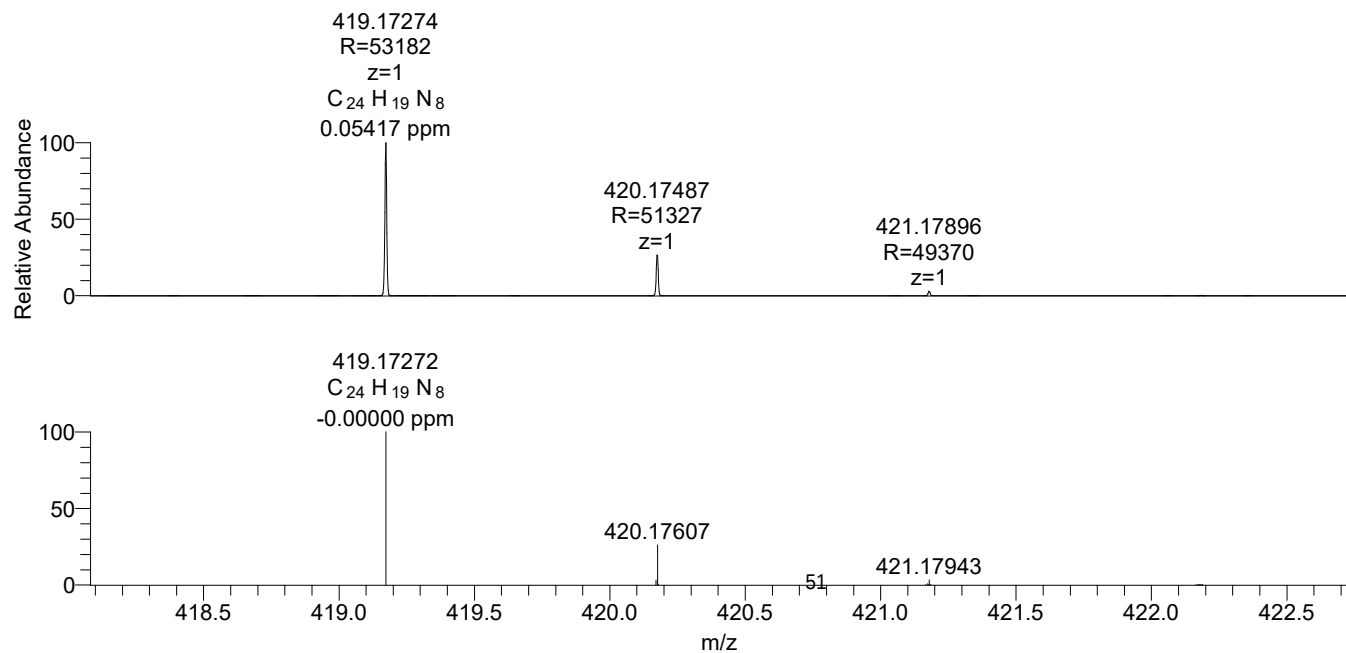


FRG-24-0813#33-105 RT: 0.14669-0.46761 AV: 73

T: FTMS + p ESI Full ms [150.0000-750.0000]

m/z = 389.90520-460.20358

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
419.17274	735636608.0	100.00	53182.27	1.00	0.05	C ₂₄ H ₁₉ N ₈
420.17487	201094160.0	27.34	51327.16	1.00		
421.17896	22176598.0	3.01	49370.30	1.00		
441.15446	7996309.0	1.09	50374.98	1.00	-0.47	C ₂₄ H ₁₈ N ₈ Na
451.14460	9099833.0	1.24	51589.00	1.00		
452.14827	2394434.8	0.33	51992.86	1.00		
457.12858	3200287.0	0.44	50538.99	1.00	-0.05	C ₂₄ H ₁₈ N ₈ K

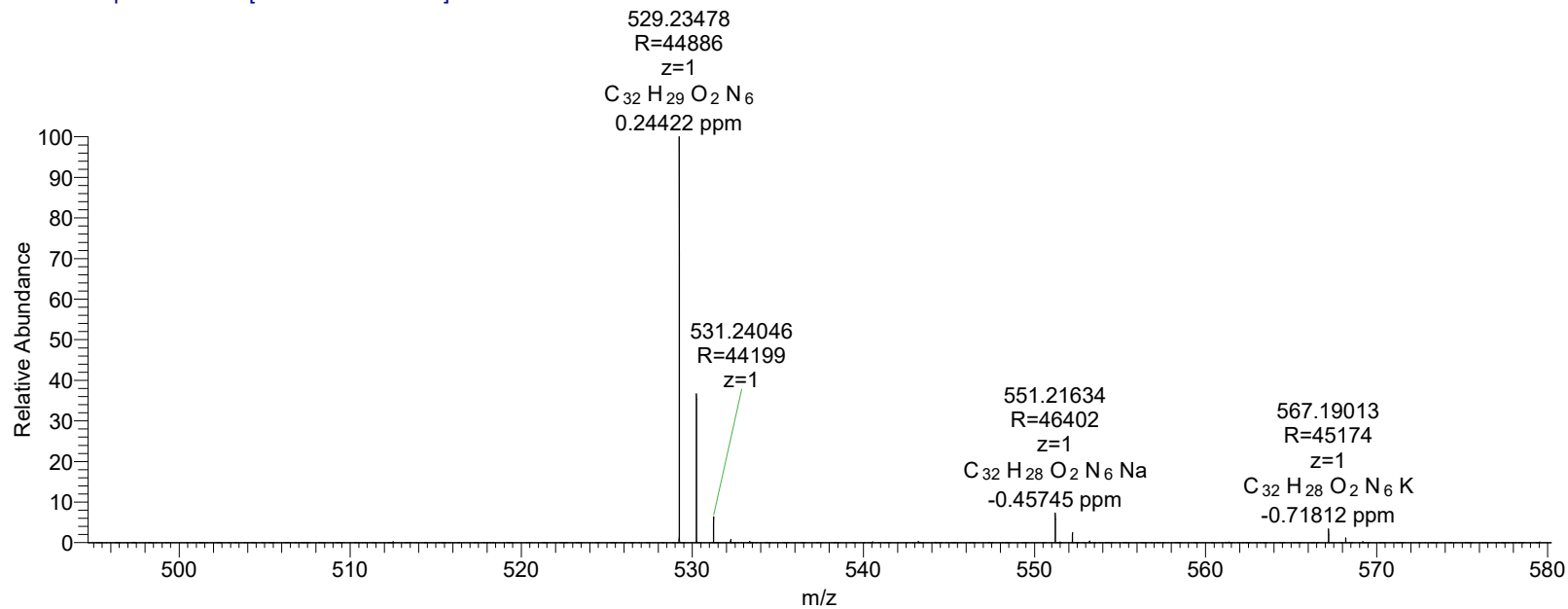


NL:
7.32E8
FRG-24-0813#33-105
RT: 0.14669-0.46761
AV: 73 T: FTMS + p ESI
Full ms
[150.0000-750.0000]

NL:
7.48E5
C₂₄H₁₈N₈ +H:
C₂₄H₁₉N₈
pa Chrg 1

FRG-25-0813 #1-90 RT: 0.00407-0.40075 AV: 90 NL: 5.32E8

T: FTMS + p ESI Full ms [150.0000-750.0000]

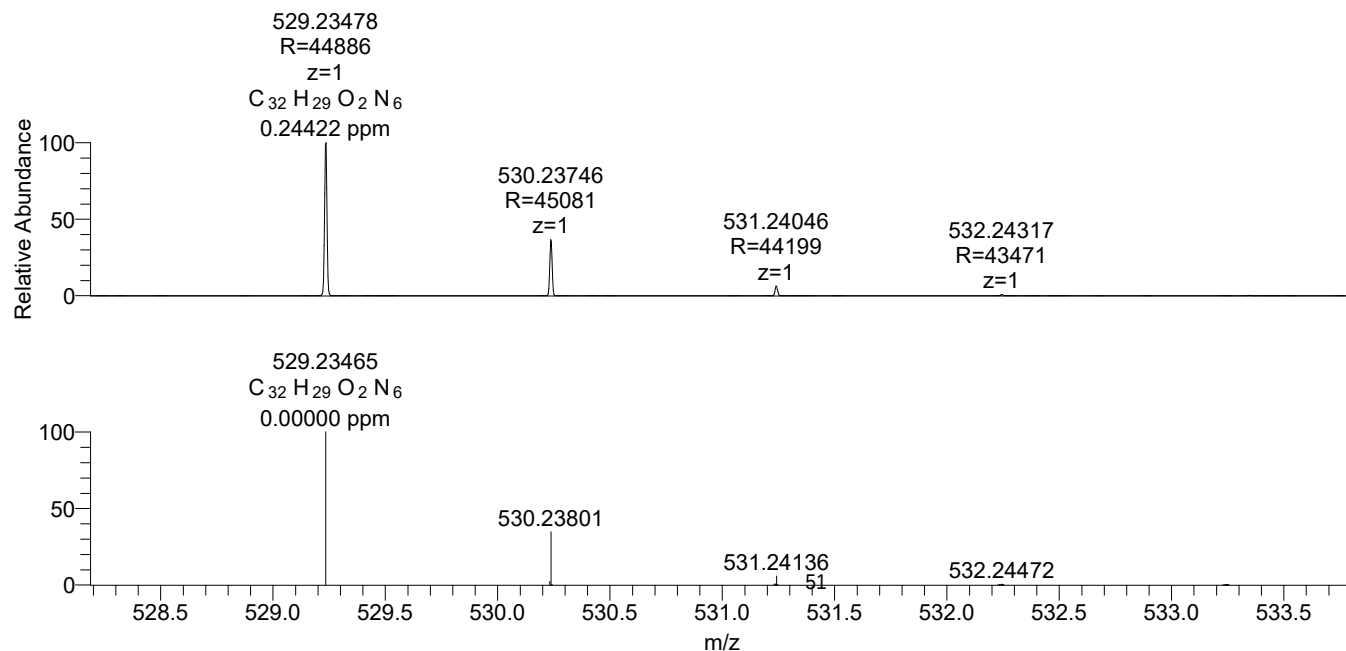


FRG-25-0813#1-90 RT: 0.00407-0.40075 AV: 90

T: FTMS + p ESI Full ms [150.0000-750.0000]

m/z = 494.66513-580.20304

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
529.23478	549321664.0	100.00	44886.16	1.00	0.24	C ₃₂ H ₂₉ O ₂ N ₆
530.23746	200181776.0	36.44	45081.15	1.00		
531.24046	34886752.0	6.35	44198.80	1.00		
532.24317	4206590.0	0.77	43470.64	1.00		
551.21634	39190168.0	7.13	46401.55	1.00	-0.46	C ₃₂ H ₂₈ O ₂ N ₆ Na
552.21970	13604597.0	2.48	43836.00	1.00		
553.22207	2407414.0	0.44	44606.48	1.00		
567.19013	18327288.0	3.34	45174.27	1.00	-0.72	C ₃₂ H ₂₈ O ₂ N ₆ K
568.19348	6428656.0	1.17	44967.75	1.00		



NL:
5.32E8
FRG-25-0813#1-90
RT: 0.00407-0.40075
AV: 90 T: FTMS + p
ESI Full ms
[150.0000-750.0000]

NL:
6.88E5
C₃₂H₂₈N₆O₂+H:
C₃₂H₂₉N₆O₂
pa Chrg 1