Supplementary information

Copper-catalyzed alkynyldisulfuration of arynes: an efficient access to unsymmetrical disulfides

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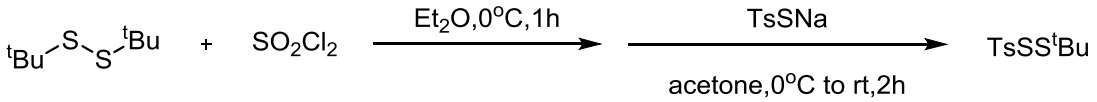
Content

1. General information

Unless otherwise noted, all the reagents were obtained commercially and used without further purification and reactions were monitored by TLC. Analytic grade solvents for the chromatography were used as received. All NMR spectra were recorded on Bruker-500 MHz and 400 MHz spectrometer. HRMS were measured on the Q-TOF6510 instruments.

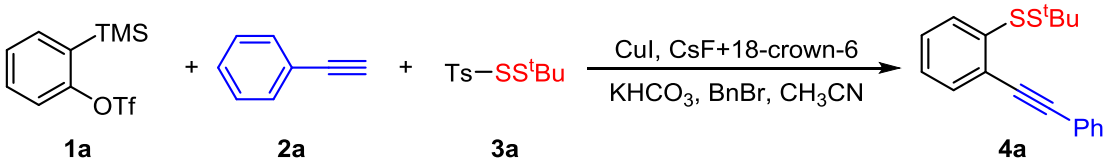
2. Synthesis of the starting materials

Procedure for the synthesis of 3a



SO_2Cl_2 (10 mmol) was added slowly to a solution of $^tBuSS^tBu$ (10 mmol) in Et_2O (40 mL) at 0 ℃ and the mixture was then stirred at 0°C for 1 h. Then a solution of TsSNa (20 mmol) in acetone (50 mL) was added dropwise at 0 ℃ and the reaction was then stirred at room temperature for 2 h. The precipitate was filtered and evaporated under reduced pressure. The residue was purified by column chromatography.

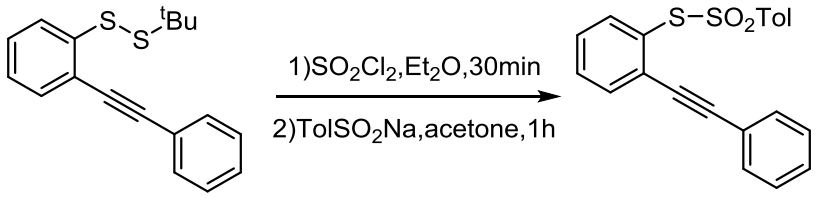
3. Procedure for the synthesis of 4a



1a (0.2 mmol), **2a** (0.3 mmol) and BnBr (0.4 mmol) were added to a mixture of CuI (10 mol%), KHCO3 (0.4 mmol), CsF (0.6 mmol), 18-crown-6 (0.6 mmol), **3a** (0.4 mmol) and 4Å MS in CH_3CN under N_2 atmosphere. Then the mixture was stirred at 40 ℃ for overnight. The precipitate was filtered and evaporated under reduced pressure. The residue was purified by column chromatography.

4. Procedure for the synthesis of 5，6，8 and 10

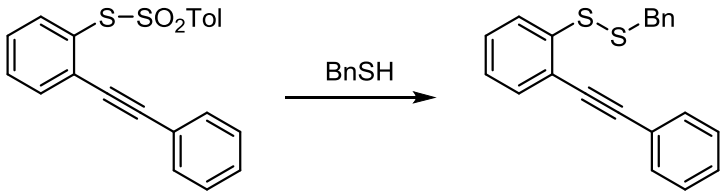
Procedure for the synthesis of 5



4a **5**

SO_2Cl_2 (1.5 mmol) was added slowly to a solution of **4a** (1 mmol) in Et_2O (5 mL) at -40 ℃ and the mixture was then stirred at -40 ℃ for 1 h. Then a solution of TsSNa (4 mmol) in 10 mL of acetone was added dropwise at -40 ℃ and the reaction was then stirred at room temperature for 2 h. The mixture was filtered and evaporated under reduced pressure. The residue was purified by column chromatography.

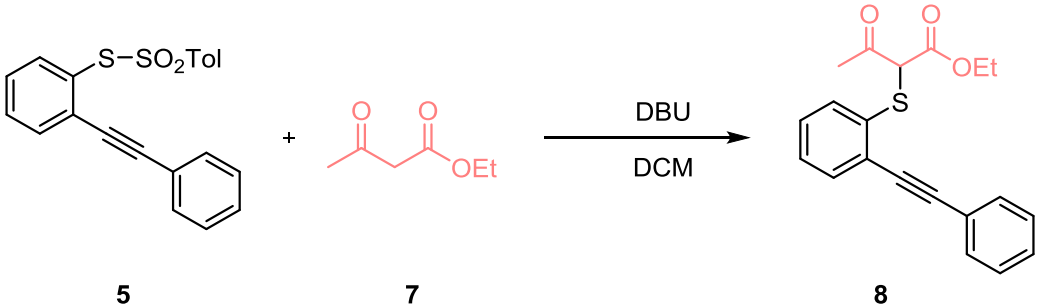
Procedure for the synthesis of 6



5 **6**

BnSH (0.25 mmol) was added to a solution **5** (0.1 mmol) in EtOH, and the mixture was stirred at 40 ℃ for 30 min. The mixture was evaporated under reduced pressure. The residue was purified by column chromatography.

Procedure for the synthesis of 8

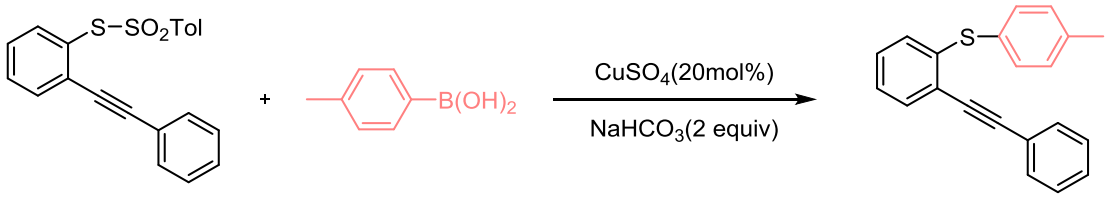


5 **7** **8**

7 (0.1 mmol) and DBU (0.15 mmol) were added to **5** (0.2 mmol) in DCM (1 mL),

and the mixture was stirred at 30 ℃ for overnight. The mixture was evaporated under reduced pressure. The residue was purified by column chromatography.

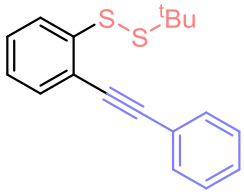
Procedure for the synthesis of 10



5 **9** **10**

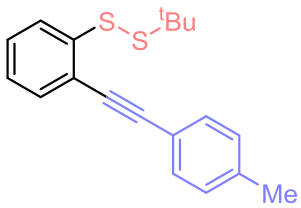
$CuSO_4$ (20 mol%), $NaHCO_3$ (0.2 mmol), **9** (0.2 mmol) and **5** (0.1 mmol) were added in CH_3OH (1 mL) under N_2 atmosphere, and the mixture was stirred at 60 ℃ for overnight. The mixture was evaporated under reduced pressure. The residue was purified by column chromatography.

5. Characterization Data



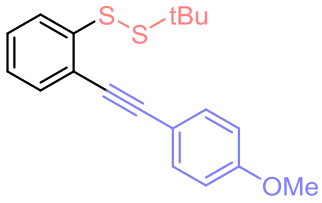
4a

4a, 85% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.85 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.62 – 7.57 (m, 2H), 7.45 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.38 – 7.28 (m, 4H), 7.14 (td, *J* = 7.5, 1.2 Hz, 1H), 1.34 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 141.40, 132.20, 131.67, 128.61, 128.55, 128.41, 126.26, 125.73, 123.16, 121.18, 96.30, 86.55, 49.36, 29.94. IR (KBr) 3058, 2962, 2920, 2858, 2217, 1597, 1490, 1454, 1362, 1159, 1032, 753, 690, 552. HRMS exact mass calcd for $C_{18}H_{18}S_2$ $[M+H]^+$ requires m/z 299.0923, found m/z 299.0922.



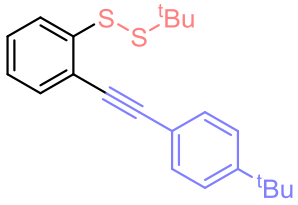
4b

4b, 81% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.84 (d, *J* = 8.0 Hz, 1H), 7.49 (d, *J* = 8.1 Hz, 2H), 7.44 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.32 – 7.27 (m, 1H), 7.19 – 7.11 (m, 3H), 2.37 (s, 3H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 141.24, 138.72, 132.10, 131.56, 129.18, 128.43, 126.19, 125.70, 121.37, 120.08, 96.55, 85.93, 49.33, 29.94, 21.61. IR (KBr) 2962, 2920, 2212, 1584, 1508, 1456, 1362, 1159, 816, 756, 521. HRMS exact mass calcd for $C_{19}H_{20}S_2$ $[M+H]^+$ requires m/z 313.1079, found m/z 313.1081.



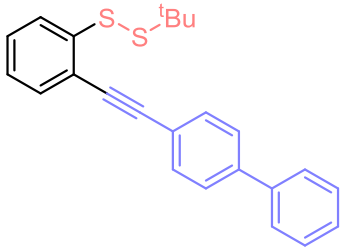
4c

4c, 76% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.84 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.56 – 7.51 (m, 2H), 7.43 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.31 – 7.26 (m, 1H), 7.13 (td, *J* = 7.5, 1.2 Hz, 1H), 6.92 – 6.87 (m, 2H), 3.83 (s, 3H), 1.34 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 159.87, 141.05, 133.13, 131.97, 128.25, 126.16, 125.68, 121.50, 115.26, 114.06, 96.41, 85.31, 55.35, 49.31, 29.93. IR (KBr) 2962, 2214, 1735, 1607, 1508, 1456, 1363, 1287, 1248, 1172, 1034, 831, 754, 539. HRMS exact mass calcd for $C_{19}H_{20}OS_2$ $[M+H]^+$ requires m/z 329.1028, found m/z 329.1029.



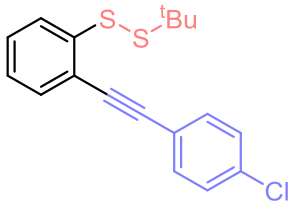
4d

4d, 76% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.84 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.57 – 7.51 (m, 2H), 7.44 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.41 – 7.37 (m, 2H), 7.32 – 7.28 (m, 1H), 7.14 (td, *J* = 7.5, 1.2 Hz, 1H), 1.34 (s, 9H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 151.84, 141.30, 132.09, 131.39, 128.40, 126.18, 125.65, 125.40, 121.36, 120.11, 96.48, 85.88, 49.31, 34.85, 31.20, 29.91. IR (KBr) 2964, 2217, 1584, 1456, 1363, 1269, 1165, 834, 753, 563. HRMS exact mass calcd for $C_{22}H_{26}S_2$ $[M+H]^+$ requires m/z 355.1549, found m/z 355.1546.



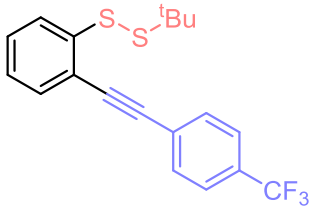
4e

4e, 77% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.86 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.68 – 7.65 (m, 2H), 7.63 – 7.58 (m, 4H), 7.49 – 7.43 (m, 3H), 7.38 – 7.34 (m, 1H), 7.33 – 7.29 (m, 1H), 7.15 (td, *J* = 7.5, 1.2 Hz, 1H), 1.35 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 141.41, 141.26, 140.40, 132.19, 132.08, 128.91, 128.62, 127.70, 127.09, 127.08, 126.27, 125.75, 122.04, 121.20, 96.23, 87.24, 49.38, 29.94. IR (KBr) 2962, 2214, 1579, 1486, 1362, 1264, 1159, 1007, 841, 766, 696, 558. HRMS exact mass calcd for $C_{24}H_{22}S_2$ $[M+H]^+$ requires m/z 375.1236, found m/z 375.1237.



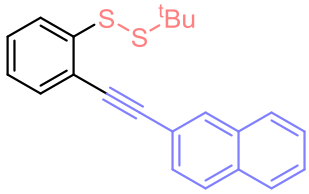
4f

4f, 61% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.85 (dd, *J* = 8.2, 1.2 Hz, 1H), 7.54 – 7.49 (m, 2H), 7.44 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.36 – 7.29 (m, 3H), 7.15 (td, *J* = 7.5, 1.2 Hz, 1H), 1.34 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 141.45, 134.56, 132.84, 132.20, 128.81, 128.76, 126.31, 125.78, 121.64, 120.86, 95.09, 87.48, 49.38, 29.92. IR (KBr) 3058, 2956, 1581, 1493, 1362, 1162, 1092, 1014, 828, 756, 519. HRMS exact mass calcd for $C_{18}H_{17}ClS_2$ $[M+H]^+$ requires m/z 333.0533, found m/z 333.0533.



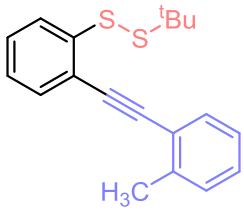
4g

4g, 80% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.87 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.69 (d, *J* = 8.7 Hz, 2H), 7.62 (d, *J* = 8.2 Hz, 2H), 7.47 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.35 (dd, *J* = 15.5, 1.4 Hz, 1H), 7.17 (td, *J* = 7.5, 1.2 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-d) δ 141.77, 132.38, 131.83, 129.16, 126.41, 125.83, 125.33 (q, *J* = 3.8 Hz), 120.49, 94.68, 88.84, 49.43, 29.90. ^{19}F NMR (471 MHz, Chloroform-d) δ -62.77. IR (KBr) 2923, 1614, 1459, 1326, 1131, 1066, 839, 756.HRMS exact mass calcd for $C_{19}H_{18}F_3S_2$ $[M+H]^+$ requires m/z 367.0797, found m/z 367.0797.



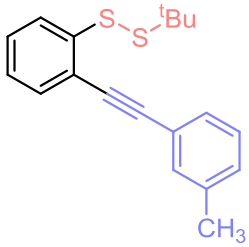
4h

4h, 80% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 8.11 (s, 1H), 7.87 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.82 (dd, *J* = 6.7, 3.8 Hz, 3H), 7.64 (dd, *J* = 8.4, 1.5 Hz, 1H), 7.54 – 7.46 (m, 3H), 7.35 – 7.30 (m, 1H), 7.19 – 7.13 (m, 1H), 1.36 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 141.45, 133.05, 132.98, 132.27, 131.54, 128.66, 128.40, 128.08, 127.92, 127.84, 126.81, 126.62, 126.24, 125.77, 121.18, 120.45, 96.74, 86.90, 49.41, 29.96. IR (KBr) 3058, 2962, 1597, 1454, 1362, 1165, 857, 818, 750, 698, 474. HRMS exact mass calcd for $C_{22}H_{20}S_2$ $[M+H]^+$ requires m/z 349.1079, found m/z 349.1077.



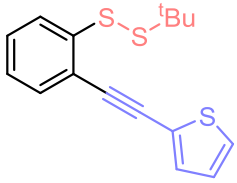
4i

4i, 85% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.86 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.56 (d, *J* = 7.4 Hz, 1H), 7.47 (dt, *J* = 7.6, 1.4 Hz, 1H), 7.30 (tt, *J* = 7.9, 1.4 Hz, 1H), 7.26 – 7.23 (m, 2H), 7.18 (dd, *J* = 7.8, 4.3 Hz, 1H), 7.14 (td, *J* = 7.5, 1.2 Hz, 1H), 2.59 (s, 3H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 141.17, 140.42, 132.32, 132.08, 129.57, 128.59, 128.52, 126.11, 125.68, 125.63, 122.96, 121.29, 95.34, 90.33, 49.38, 29.92, 21.11. IR (KBr) 3058, 2959, 2212, 1581, 1456, 1363, 1050, 1034, 753, 566. HRMS exact mass calcd for $C_{19}H_{20}S_2$ $[M+H]^+$ requires m/z 313.1079, found m/z 313.1080.



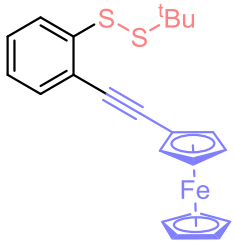
4j

4j, 74% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.85 (d, *J* = 8.1 Hz, 1H), 7.46 – 7.38 (m, 3H), 7.32 – 7.28 (m, 1H), 7.25 (t, *J* = 8.1 Hz, 1H), 7.18 – 7.11 (m, 2H), 2.36 (s, 3H), 1.34 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 141.34, 138.08, 132.20, 129.47, 128.78, 128.54, 128.31, 126.17, 125.70, 122.95, 121.23, 96.52, 86.18, 49.35, 29.94, 21.31. IR (KBr) 3058, 2962, 2204, 1601, 1456, 1363, 1163, 1034, 784, 756, 688, 560. HRMS exact mass calcd for $C_{19}H_{20}S_2$ $[M+H]^+$ requires m/z 313.1079, found m/z 313.1079.



4k

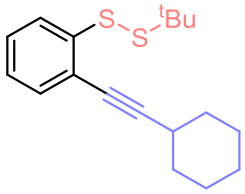
4k, 80% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.84 (d, *J* = 8.1 Hz, 1H), 7.42 (d, *J* = 1.4 Hz, 1H), 7.35 – 7.28 (m, 3H), 7.17 – 7.12 (m, 1H), 7.03 (dd, *J* = 5.2, 3.6 Hz, 1H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 141.34, 132.20, 132.03, 128.75, 127.74, 127.20, 126.44, 125.79, 123.04, 120.95, 90.20, 89.42, 49.40, 29.92. IR (KBr) 2962, 2199, 1582, 1459, 1363, 1214, 1165, 1034, 855, 756, 703, 568. HRMS exact mass calcd for $C_{16}H_{16}S_3$ $[M+H]^+$ requires m/z 305.0487, found m/z 305.0485.



4l

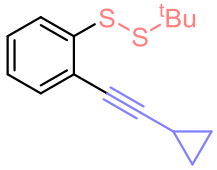
4l, 77% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.83 (dt, *J* = 8.1, 1.3 Hz, 1H), 7.40 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.32 – 7.26 (m, 1H), 7.12 (td, *J* = 7.4, 1.1 Hz, 1H), 4.57 (t, *J* = 1.9 Hz, 2H), 4.29 (s, 5H), 4.26 (d, *J* = 1.9 Hz, 2H), 1.36 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 140.81,

131.99, 128.02, 126.03, 125.65, 121.76, 95.73, 82.63, 71.61, 70.14, 69.04, 64.85, 49.25, 29.95. IR (KBr) 2959, 2206, 1443, 1363, 1165, 1106, 1034, 823, 756, 498. HRMS exact mass calcd for $C_{22}H_{22}FeS_2$ $[M+H]^+$ requires m/z 407.0585, found m/z 407.0533.



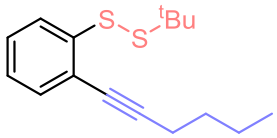
4m

4m, 66% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.79 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.32 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.27 – 7.22 (m, 1H), 7.07 (td, *J* = 7.4, 1.2 Hz, 1H), 2.78 – 2.66 (m, 1H), 1.91 (dd, *J* = 12.0, 7.2 Hz, 2H), 1.86 – 1.78 (m, 2H), 1.63 (dt, *J* = 13.0, 8.9 Hz, 2H), 1.45 – 1.35 (m, 4H), 1.32 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 140.96, 132.02, 127.77, 125.79, 125.44, 121.77, 101.86, 49.17, 32.52, 29.88, 26.00, 24.72. IR (KBr) 2925, 2222, 1584, 1461, 1363, 1264, 1167, 1034, 756. HRMS exact mass calcd for $C_{18}H_{24}S_2$ $[M+H]^+$ requires m/z 305.1392, found m/z 305.1392.



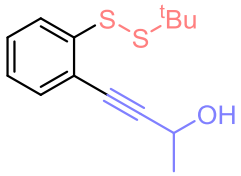
4n

4n, 53% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.77 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.30 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.25 – 7.21 (m, 1H), 7.06 (td, *J* = 7.5, 1.2 Hz, 1H), 1.56 – 1.51 (m, 1H), 1.32 (s, 9H), 0.95 – 0.87 (m, 4H). ^{13}C NMR (126 MHz, Chloroform-d) δ 140.45, 131.65, 127.26, 125.27, 124.98, 121.12, 100.51, 72.30, 48.67, 29.39, 8.49. IR (KBr) 2959, 2227, 1584, 1461, 1365, 1162, 1029, 951, 756. HRMS exact mass calcd for $C_{15}H_{18}S_2$ $[M+H]^+$ requires m/z 263.0923, found m/z 263.0922.



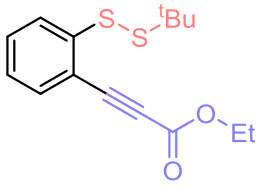
4o

4o, 66% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.79 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.31 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.25 (td, *J* = 7.8, 1.5 Hz, 1H), 7.07 (td, *J* = 7.5, 1.2 Hz, 1H), 2.51 (t, *J* = 6.9 Hz, 2H), 1.68 – 1.61 (m, 2H), 1.59 – 1.51 (m, 2H), 1.32 (s, 9H), 0.96 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, Chloroform-d) δ 140.85, 132.14, 127.81, 125.71, 125.44, 121.70, 97.93, 49.17, 30.74, 29.87, 22.02, 19.41, 13.66. IR (KBr) 2956, 2863, 2229, 1584, 1460, 1362, 1162, 1033, 750. HRMS exact mass calcd for $C_{16}H_{22}S_2$ $[M+H]^+$ requires m/z 279.1236, found m/z 279.1235.



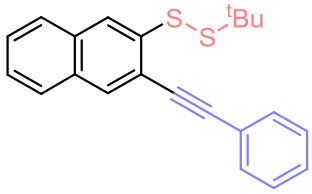
4p

4p, 75% yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.81 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.36 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.32 – 7.28 (m, 1H), 7.11 (td, *J* = 7.5, 1.2 Hz, 1H), 4.88 – 4.80 (m, 1H), 2.08 (d, *J* = 5.4 Hz, 1H), 1.61 (d, *J* = 6.6 Hz, 3H), 1.32 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 141.29, 132.36, 128.75, 126.04, 125.64, 120.34, 97.96, 81.10, 59.04, 49.33, 29.88, 24.37. IR (KBr) 2925, 1594, 1455, 1316, 1154, 1029, 933, 758, 695, 599, 547, 513. HRMS exact mass calcd for $C_{14}H_{18}S_2$ $[M+H]^+$ requires m/z 267.0872, found m/z 267.0836.



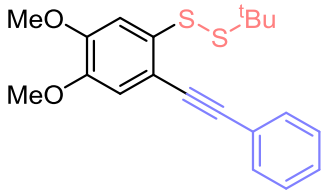
4q

4q, 56%yield. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.87 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.51 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.43 – 7.38 (m, 1H), 7.16 (td, *J* = 7.5, 1.1 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 1.37 (t, *J* = 7.2 Hz, 3H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 153.97, 143.73, 133.95, 133.00, 130.79, 128.59, 126.51, 125.83, 86.76, 82.75, 62.23, 49.64, 29.86, 14.14. IR (KBr) 2962, 2209, 1712, 1458, 1365, 1295, 1248, 1191, 1021, 758. HRMS exact mass calcd for $C_{22}H_{20}S_2$ $[M+H]^+$ requires m/z 295.0821, found m/z 295.0825.



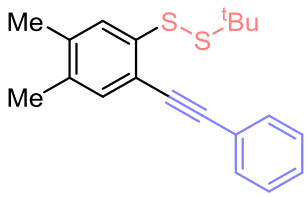
4r

4r, 73%yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 8.24 (s, 1H), 8.02 (s, 1H), 7.79 (dd, *J* = 14.5, 8.1 Hz, 2H), 7.67 – 7.63 (m, 2H), 7.51 – 7.47 (m, 1H), 7.44 (td, *J* = 7.5, 6.8, 1.3 Hz, 1H), 7.42 – 7.37 (m, 3H), 1.39 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 137.53, 133.11, 132.22, 131.72, 131.54, 128.58, 128.42, 127.50, 127.22, 127.06, 126.12, 124.81, 123.12, 119.60, 95.84, 86.78, 49.61, 30.04. IR (KBr) 2920, 1493, 1362, 1162, 887, 756, 690, 474. HRMS exact mass calcd for $C_{22}H_{20}S_2$ $[M+H]^+$ requires m/z 349.1079, found m/z 349.1080.



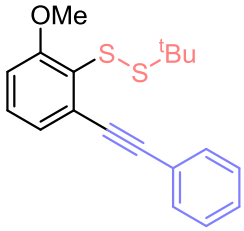
4s

4s, 68%yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.58 (dd, *J* = 7.8, 1.7 Hz, 2H), 7.38 – 7.33 (m, 4H), 6.96 (s, 1H), 3.95 (s, 3H), 3.89 (s, 3H), 1.34 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 149.85, 147.69, 133.69, 131.48, 128.38, 128.26, 123.36, 114.41, 113.99, 110.86, 94.51, 86.91, 56.15, 56.10, 49.26, 29.98. IR (KBr) 2923, 1592, 1495, 1248, 1211, 1045, 862, 758, 691. HRMS exact mass calcd for $C_{20}H_{22}O_2S_2$ $[M+H]^+$ requires m/z 359.1134, found m/z 359.1133.



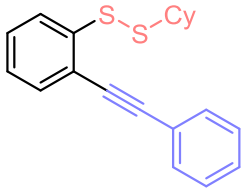
4t

4t, 72%yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.60 – 7.54 (m, 3H), 7.38 – 7.31 (m, 3H), 7.25 (s, 1H), 2.29 (s, 3H), 2.22 (s, 3H), 1.34 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 137.99, 137.91, 134.78, 133.17, 131.56, 128.33, 128.23, 123.46, 119.05, 95.00, 86.99, 49.21, 29.93, 19.94, 19.07. IR (KBr) 2923, 1595, 1490, 1451, 1362, 1167, 1021, 886, 761, 688. HRMS exact mass calcd for $C_{20}H_{22}S_2$ $[M+H]^+$ requires m/z 327.1236, found m/z 327.1235.



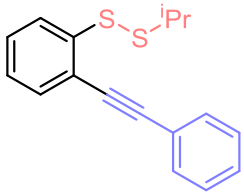
4u

4u, 50%yield. ^{1}H NMR (500 MHz, Chloroform-d) δ 7.61 (dd, *J* = 7.8, 1.8 Hz, 2H), 7.39 – 7.33 (m, 3H), 7.27 – 7.24 (m, 1H), 7.18 (dd, *J* = 7.6, 1.2 Hz, 1H), 6.87 (dd, *J* = 8.3, 1.2 Hz, 1H), 3.93 (s, 3H), 1.34 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-d) δ 159.79, 131.71, 129.76, 129.03, 128.99, 128.47, 128.37, 125.13, 123.33, 111.47, 95.51, 88.40, 56.26, 48.30, 29.88. IR (KBr) 2959, 1560, 1491, 1461, 1261, 1068, 735. HRMS exact mass calcd for $C_{19}H_{20}OS_2$ $[M+H]^+$ requires m/z 329.1028, found m/z 329.1028.



4v

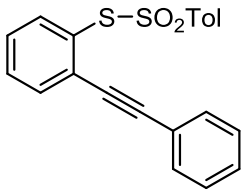
4v, 45%yield. ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.86 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.64 – 7.59 (m, 2H), 7.49 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.42 – 7.34 (m, 4H), 7.19 (td, *J* = 7.5, 1.2 Hz, 1H), 2.85 (tt, *J* = 10.9, 3.7 Hz, 1H), 2.13 – 2.03 (m, 2H), 1.84 – 1.74 (m, 2H), 1.65 – 1.59 (m, 1H), 1.49 – 1.37 (m, 2H), 1.31 – 1.24 (m, 4H). ^{13}C NMR (101 MHz, Chloroform-d) δ 141.07, 132.25, 131.65, 128.72, 128.53, 128.37, 125.61, 125.55, 123.06, 121.00, 96.36, 86.38, 49.88, 32.75, 26.07, 25.55. IR (KBr) 2925, 2850, 1491, 1454, 1266, 737. HRMS exact mass calcd for C20H20S2 [M+H]+ requires m/z 325.1079, found m/z 325.1079.



4w

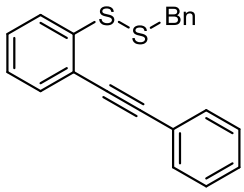
4w, 65%yield. ^{1}H NMR (400 MHz, Chloroform-d) δ 7.82 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.61 – 7.57 (m,

2H), 7.47 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.37 – 7.31 (m, 4H), 7.16 (td, *J* = 7.5, 1.2 Hz, 1H), 3.08 (h, *J* = 6.8 Hz, 1H), 1.33 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-d) δ 140.85, 132.30, 131.65, 128.73, 128.55, 128.38, 125.83, 125.77, 123.07, 121.24, 96.34, 86.39, 41.60, 22.54. IR (KBr) 2923, 1490, 1456, 1245, 1154, 1050, 756, 689. HRMS exact mass calcd for $C_{17}H_{16}S_2$ $[M+H]^+$ requires m/z 285.0766, found m/z 285.0768.



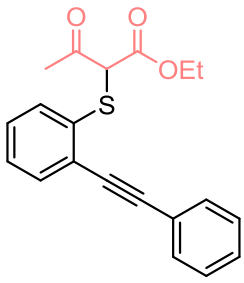
5

5, 80% yield. ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.75 (dd, *J* = 7.5, 1.7 Hz, 1H), 7.55 – 7.35 (m, 10H), 7.04 (d, *J* = 7.8 Hz, 2H), 2.25 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 144.60, 141.20, 138.54, 132.86, 131.67, 131.32, 129.87, 129.66, 129.50, 129.00, 128.68, 128.23, 127.41, 122.74, 94.29, 86.67, 21.53. IR (KBr) 3061, 2219, 1592, 1490, 1331, 1146, 1076, 811, 756, 691, 654, 578, 521. HRMS exact mass calcd for $C_{21}H_{16}O_2S_2$ $[M+H]^+$ requires m/z 365.0664, found m/z 365.0666.



6

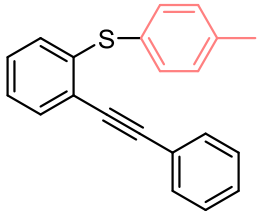
6,82% yield. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.67 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.59 – 7.55 (m, 2H), 7.47 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.37 – 7.22 (m, 9H), 7.16 (td, *J* = 7.5, 1.2 Hz, 1H), 3.96 (s, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 139.63, 136.56, 132.34, 131.66, 129.35, 128.75, 128.59, 128.58, 128.35, 127.59, 126.03, 125.94, 123.00, 121.64, 96.42, 86.42, 43.54. IR (KBr) 2917, 1493, 1456, 1266, 756, 696. HRMS exact mass calcd for $C_{21}H_{16}S_2$ $[M+H]^+$ requires m/z 333.0766, found m/z 333.0765.



8

8, 80% yield. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 13.89 (d, *J* = 0.8 Hz, 1H), 7.54 – 7.51 (m, 2H), 7.40 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.31 – 7.25 (m, 3H), 7.17 – 7.11 (m, 1H), 7.01 (td, *J* = 7.5, 1.2 Hz, 1H), 6.84 (dd, *J* = 8.0, 1.2 Hz, 1H), 4.15 (q, *J* = 7.1 Hz, 2H), 2.25 (d, *J* = 0.7 Hz, 3H), 1.12 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 184.05, 171.96, 140.37, 131.40, 130.61, 127.58, 127.38, 127.30, 123.29, 122.50, 122.20, 119.05, 94.90, 90.03, 85.68, 60.65, 19.95, 13.06. IR (KBr) 2923, 2852, 1714,

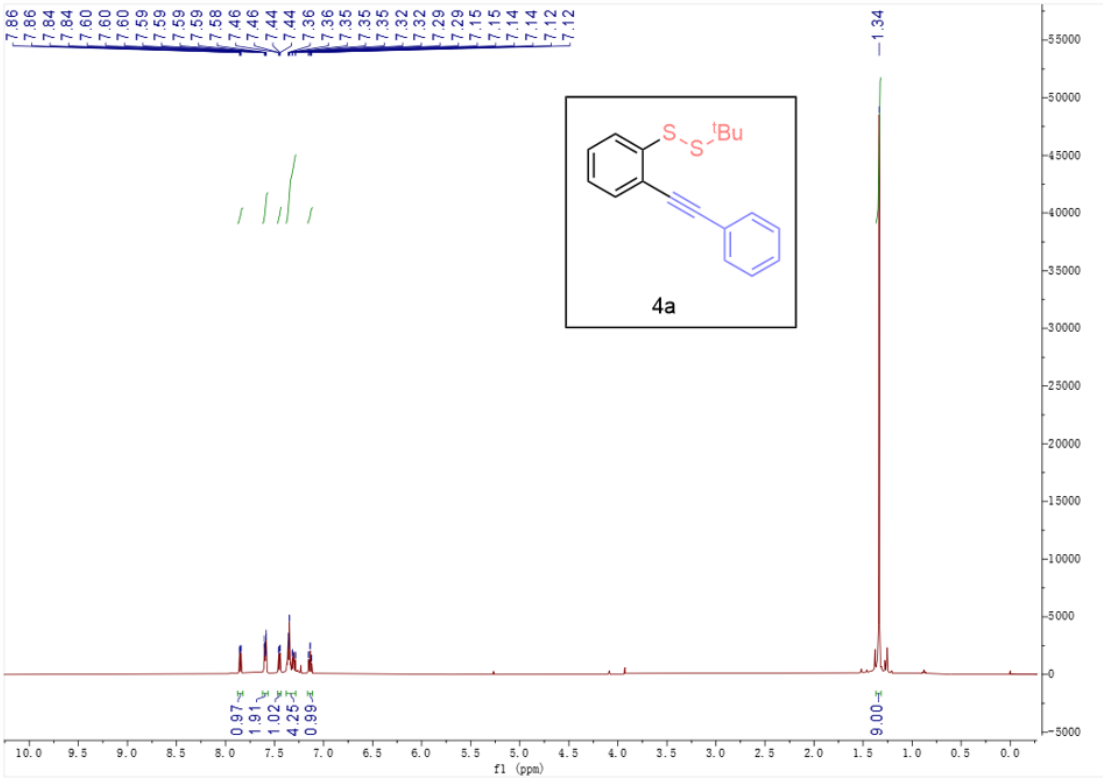
1594, 1461, 1378, 1250, 755, 737, 690. HRMS exact mass calcd for $C_{20}H_{18}O_3S$ $[M+H]^+$ requires m/z 339.1049, found m/z 339.1049.

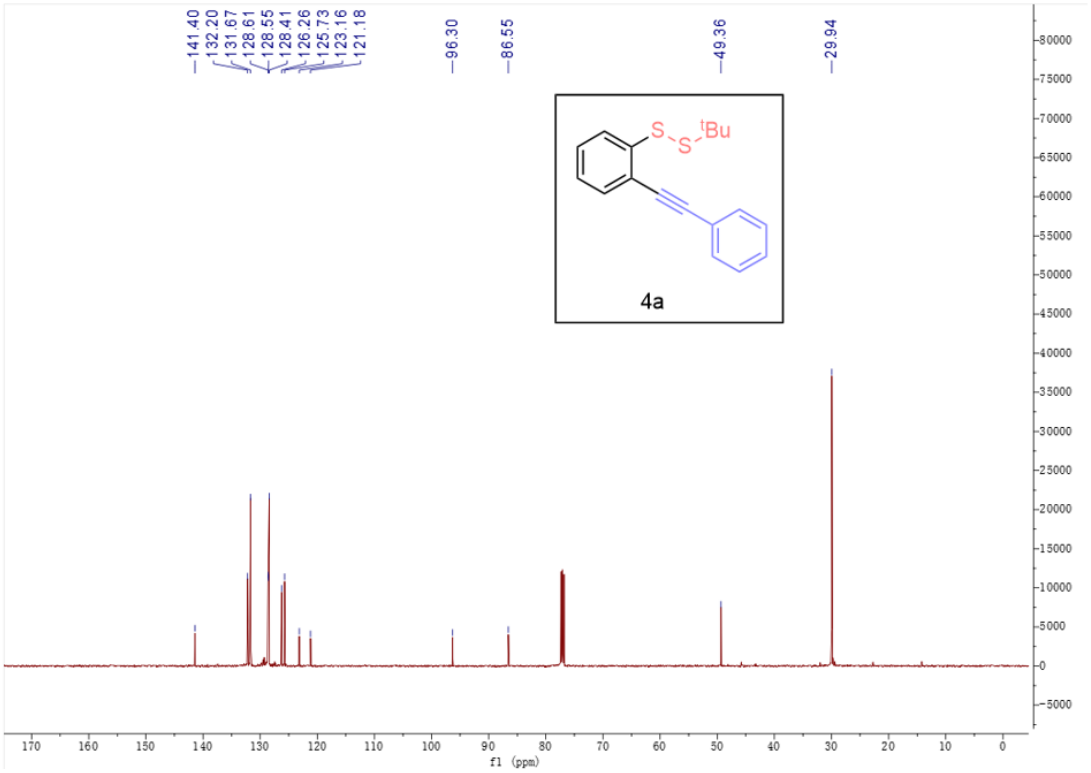


10

10, 67% yield. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 – 7.53 (m, 2H), 7.52 – 7.48 (m, 1H), 7.44 – 7.39 (m, 2H), 7.36 – 7.31 (m, 3H), 7.21 – 7.18 (m, 2H), 7.15 – 7.08 (m, 2H), 6.91 – 6.88 (m, 1H), 2.37 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 141.52, 138.60, 134.18, 132.40, 131.69, 130.36, 129.15, 128.69, 128.41, 128.31, 127.38, 125.25, 123.19, 121.82, 95.68, 87.11, 21.26. IR (KBr) 3055, 2917, 2852, 1598, 1490, 1459, 1433, 1060, 808, 750, 690, 555, 513. HRMS exact mass calcd for $C_{21}H_{16}S$ $[M+H]^+$ requires m/z 301.1045, found m/z 301.1045.

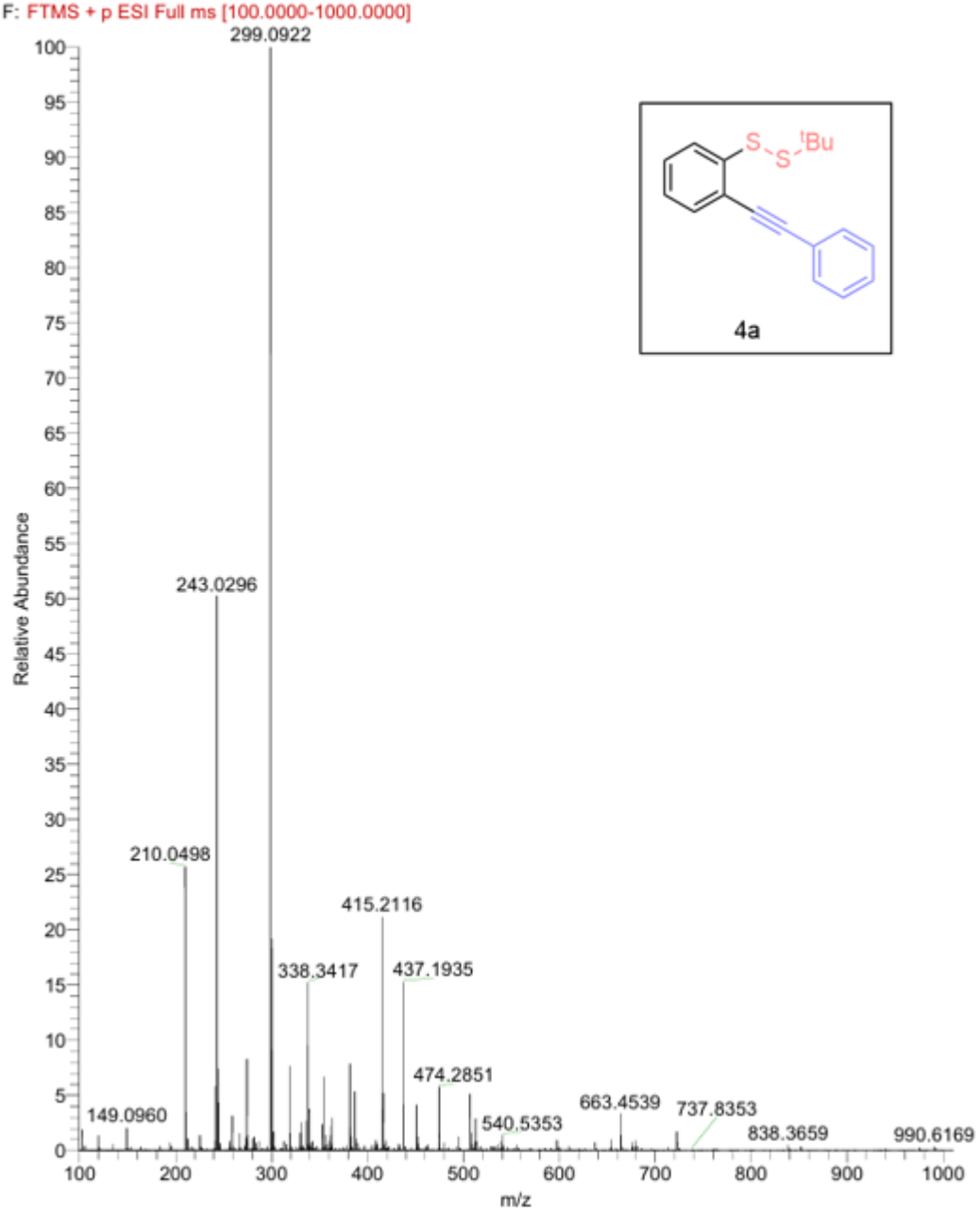
6. Spectra for the products

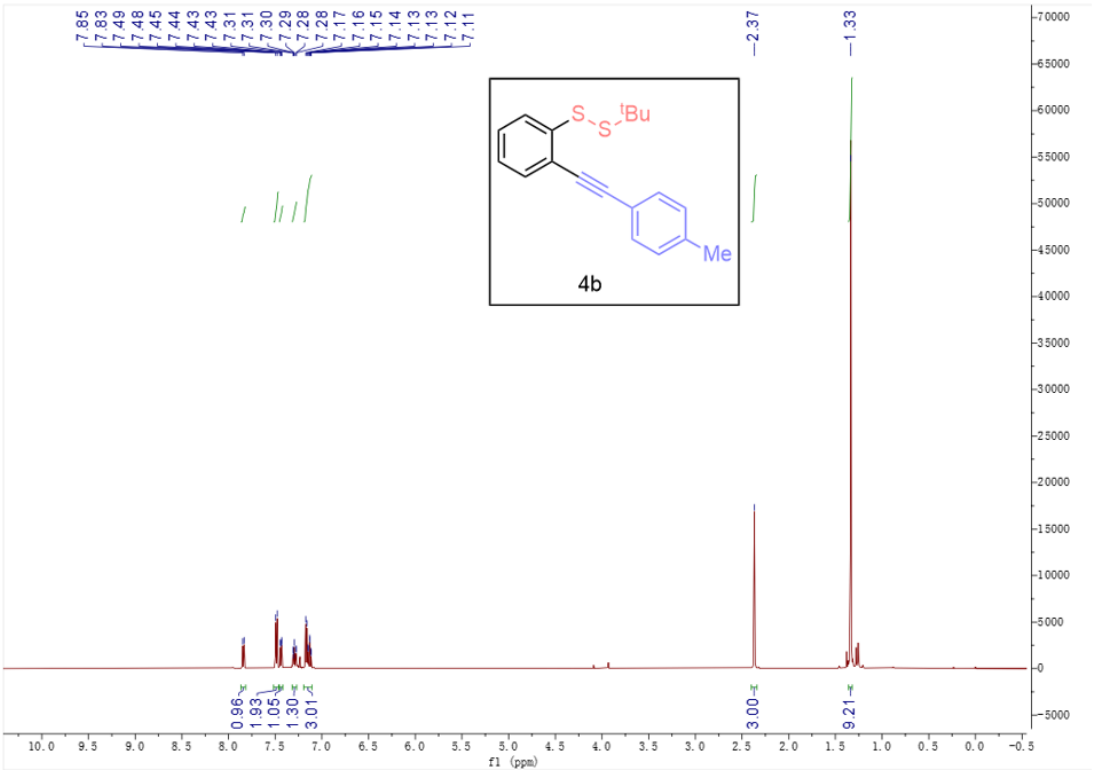


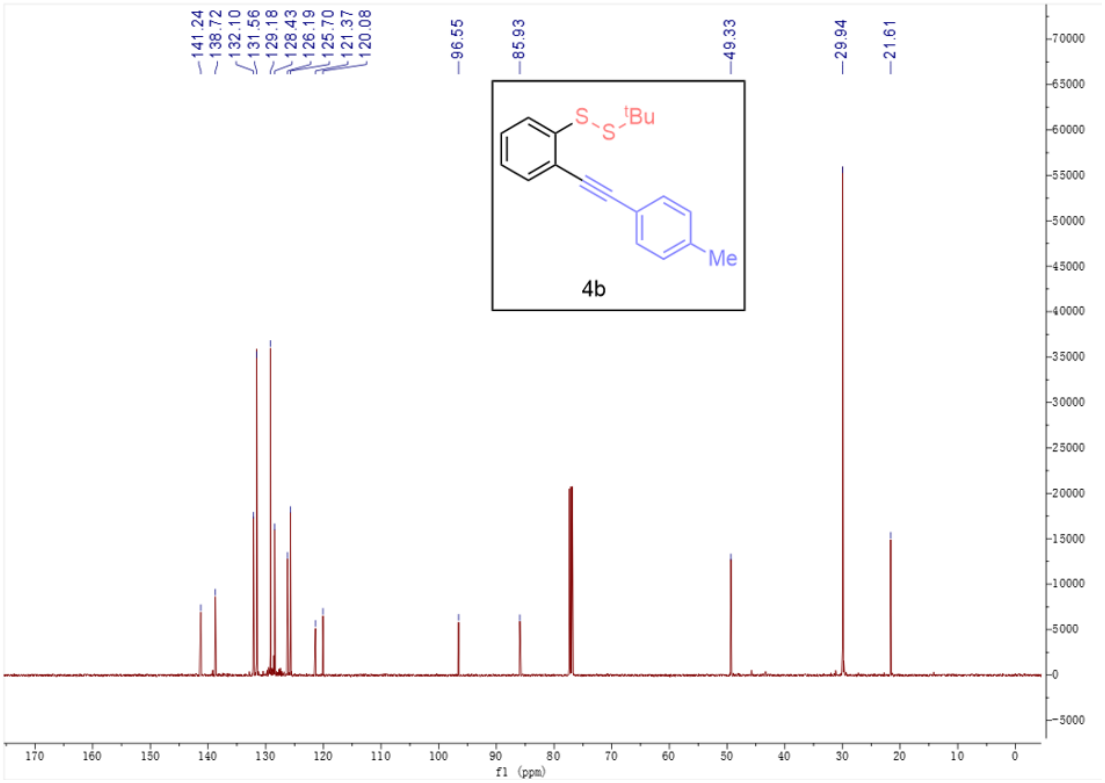

141.40
132.20
131.67
128.61
128.55
128.41
126.26
125.73
123.16
121.18
96.30
86.55
49.36
29.94
S
S
tBu
4a
f1 (ppm)

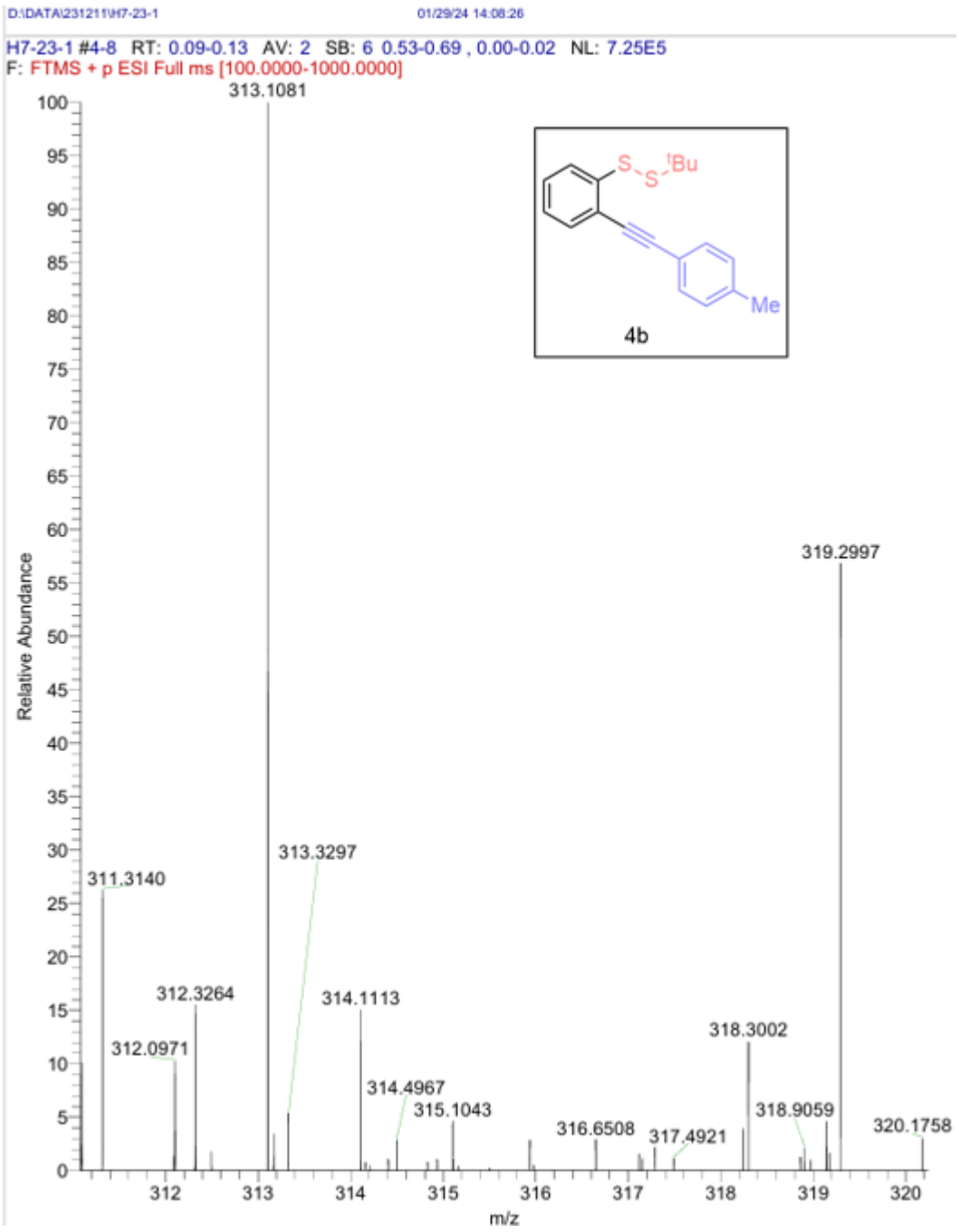
D:\DATA\230922\SYM-H7-17-1A 12/05/23 16:29:59

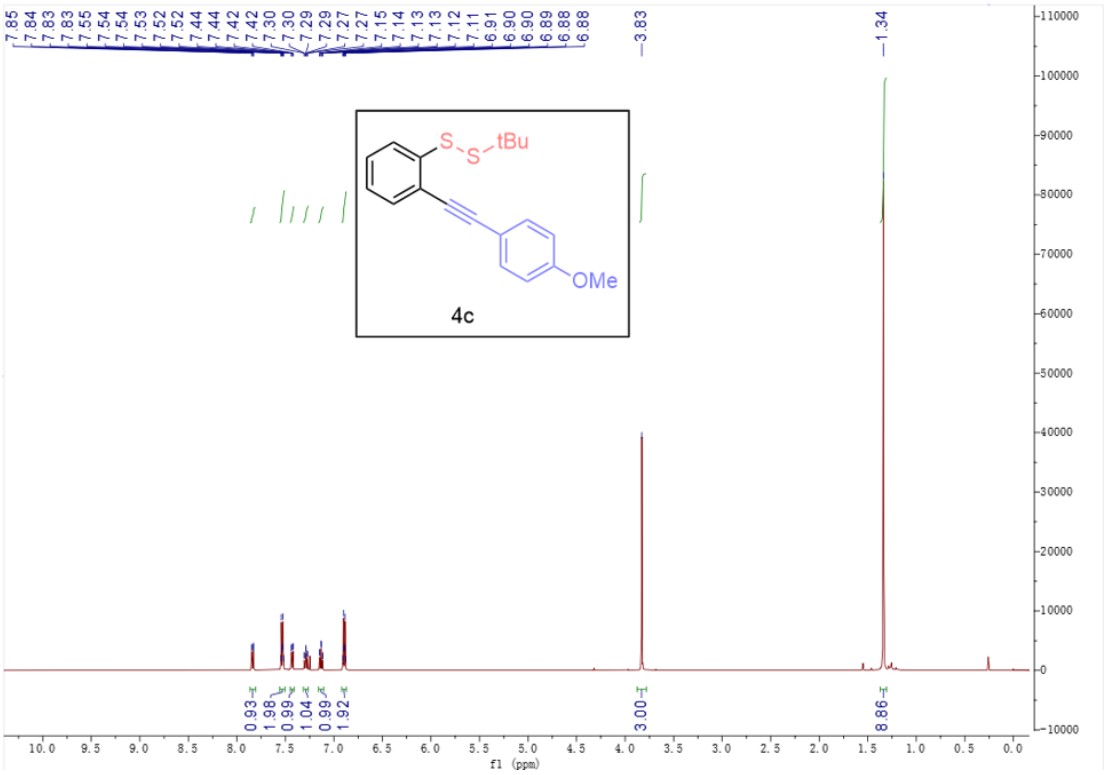
SYM-H7-17-1A #4-11 RT: 0.09-0.21 AV: 4 NL: 2.70E8
F: FTMS + p ESI Full ms [100.0000-1000.0000]

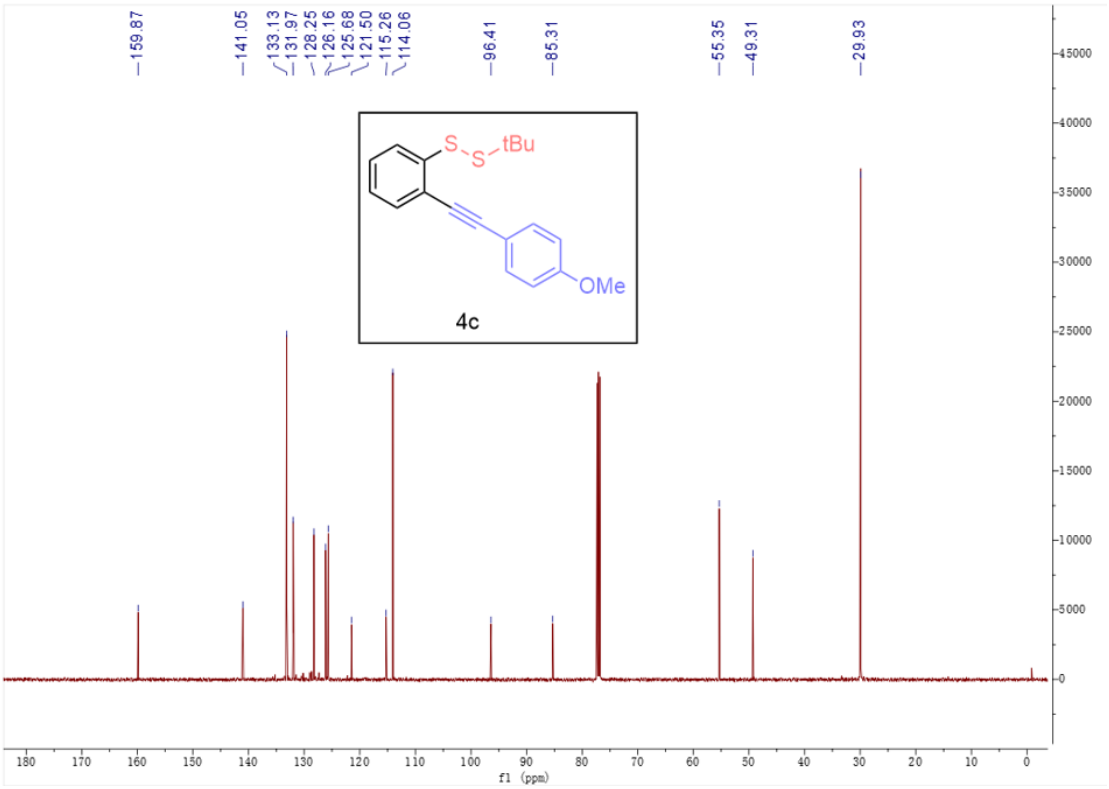


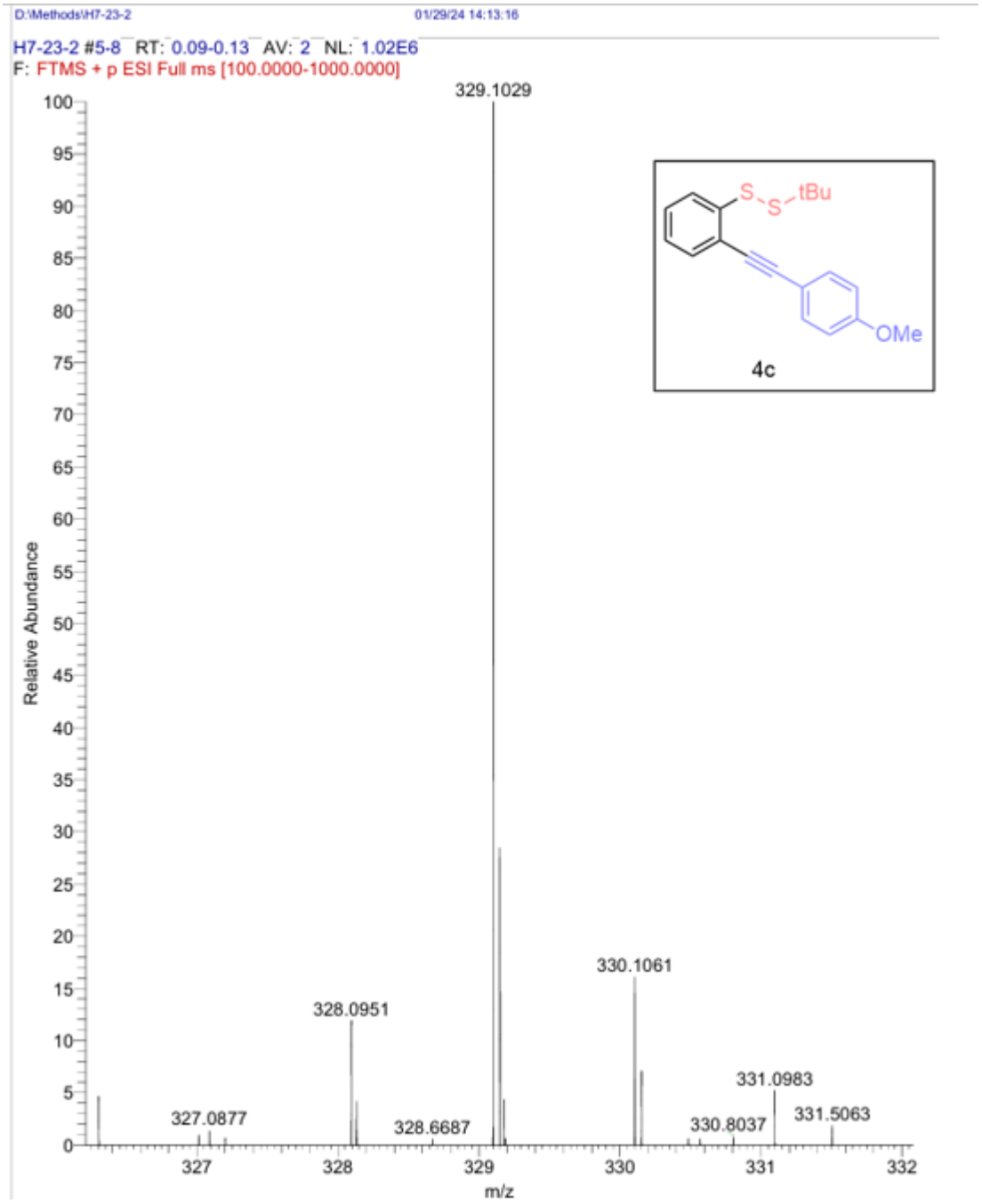

7.85
7.83
7.49
7.48
7.45
7.44
7.43
7.43
7.31
7.31
7.30
7.29
7.28
7.28
7.17
7.16
7.15
7.14
7.13
7.13
7.12
7.11
2.37
1.33
S
S
tBu
Me
4b
0.96
1.93
1.05
1.30
3.01
3.00
9.21
f1 (ppm)

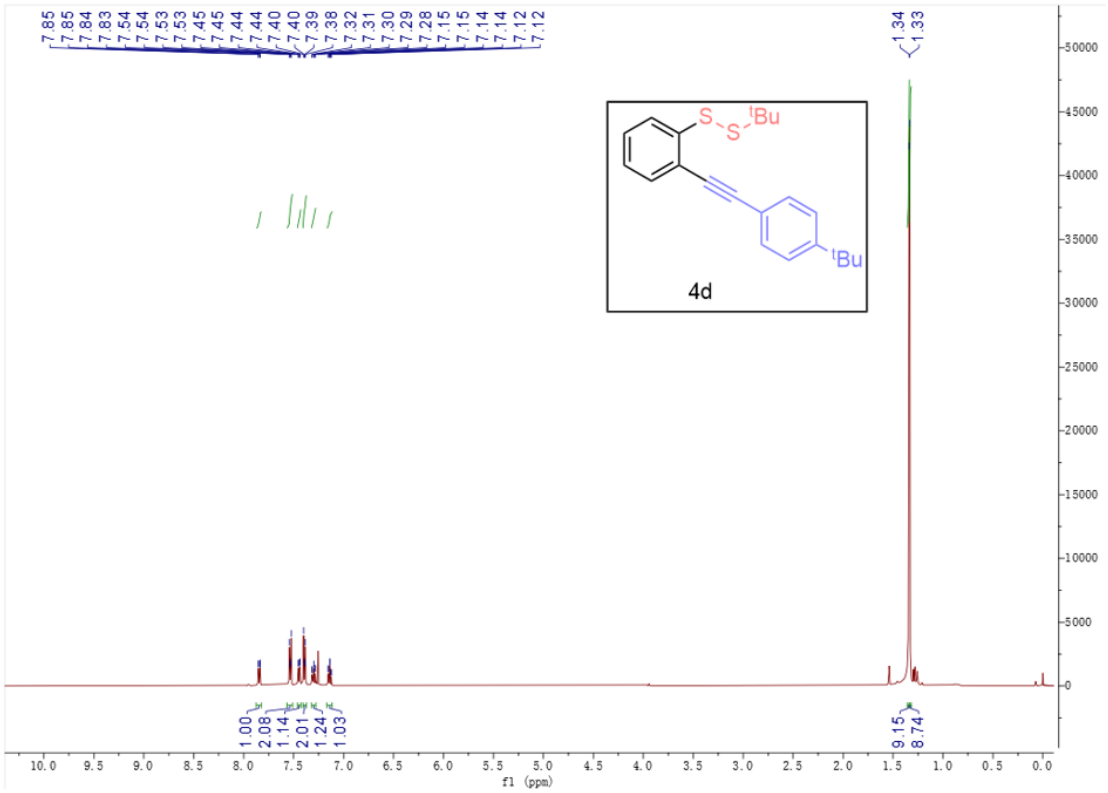

141.24
138.72
132.10
131.56
129.18
128.43
126.19
125.70
121.37
120.08
96.55
85.93
49.33
29.94
21.61
S
S
tBu
Me
4b
f1 (ppm)


D:\DATA\231211\H7-23-1
01/29/24 14:08:26
H7-23-1 #4-8 RT: 0.09-0.13 AV: 2 SB: 6 0.53-0.69 , 0.00-0.02 NL: 7.25E5
F: FTMS + p ESI Full ms [100.0000-1000.0000]
313.1081
319.2997
313.3297
311.3140
312.3264
314.1113
312.0971
318.3002
314.4967
315.1043
318.9059
316.6508
317.4921
320.1758
Relative Abundance
m/z
S
S
tBu
Me
4b


7.85
7.84
7.83
7.83
7.55
7.54
7.54
7.53
7.52
7.52
7.44
7.44
7.42
7.42
7.30
7.30
7.29
7.29
7.27
7.27
7.15
7.14
7.13
7.12
7.11
6.91
6.90
6.90
6.89
6.88
6.88
3.83
1.34
S
S
tBu
OMe
4c
0.93
1.98
0.99
1.04
0.99
1.92
3.00
8.86
f1 (ppm)


159.87
141.05
133.13
131.97
128.25
126.16
125.68
121.50
115.26
114.06
96.41
85.31
55.35
49.31
29.93
S
S
tBu
OMe
4c
f1 (ppm)


D:\Methods\H7-23-2
01/29/24 14:13:16
H7-23-2 #5-8 RT: 0.09-0.13 AV: 2 NL: 1.02E6
F: FTMS + p ESI Full ms [100.0000-1000.0000]
329.1029
330.1061
328.0951
331.0983
331.5063
327.0877
328.6687
330.8037
Relative Abundance
m/z
S
S
tBu
OMe
4c


7.85
7.85
7.84
7.83
7.54
7.54
7.53
7.53
7.45
7.45
7.44
7.44
7.40
7.40
7.39
7.38
7.32
7.31
7.30
7.29
7.28
7.15
7.15
7.14
7.14
7.12
7.12
1.34
1.33
S
S
tBu
tBu
4d
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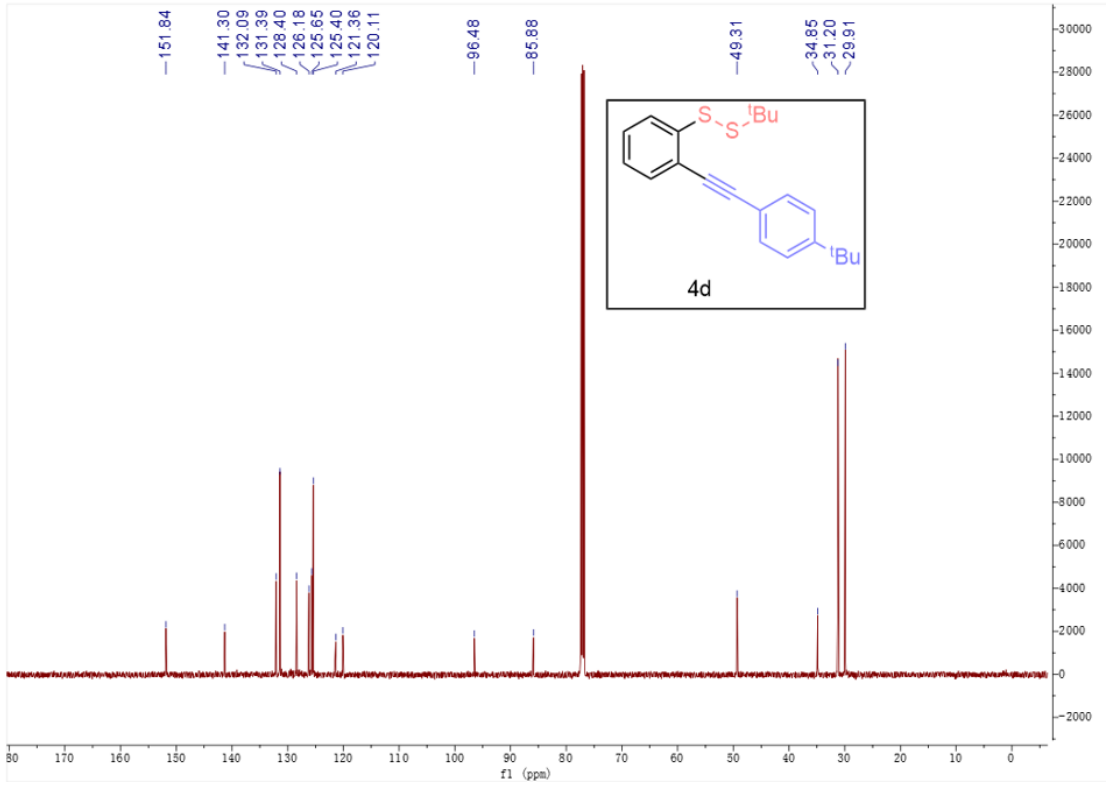
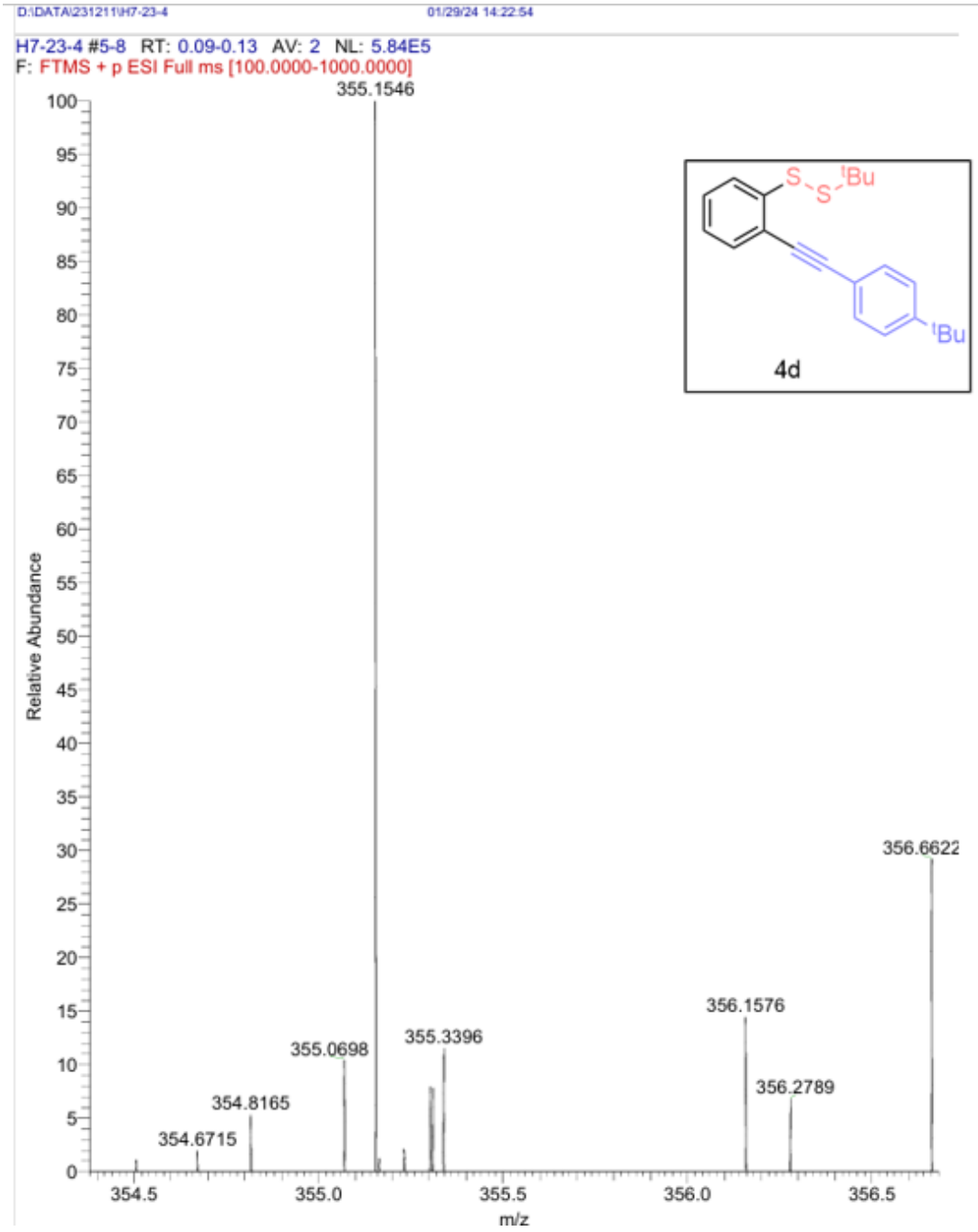
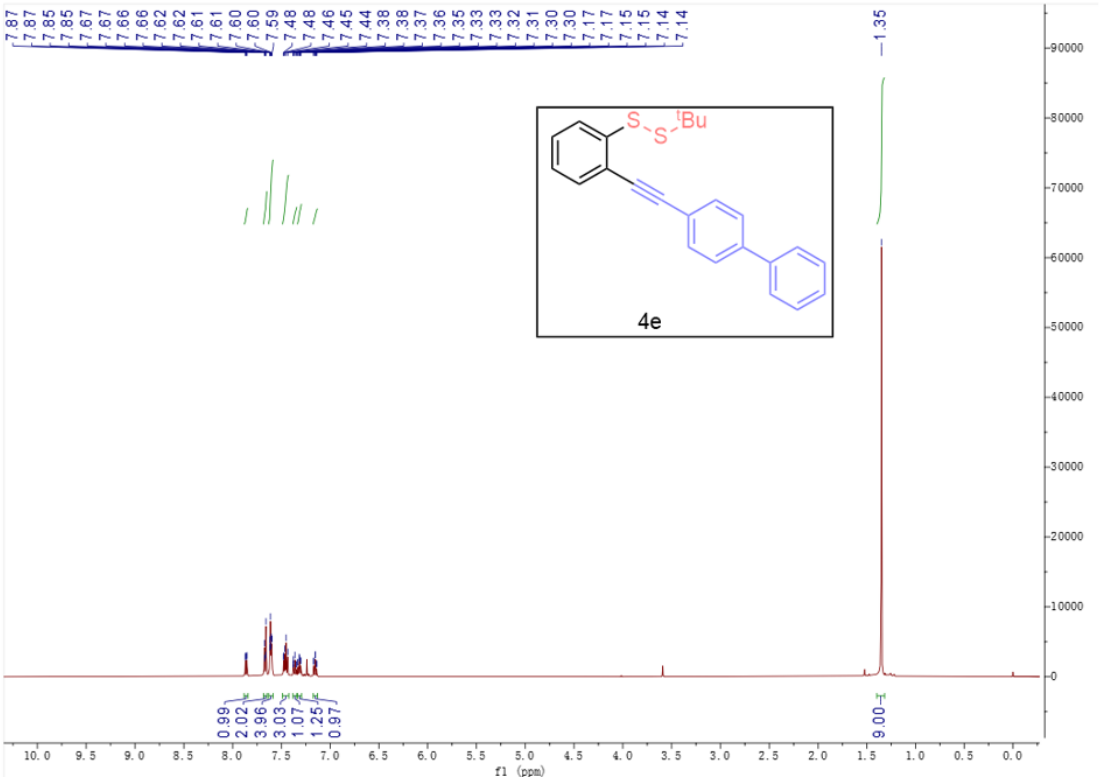
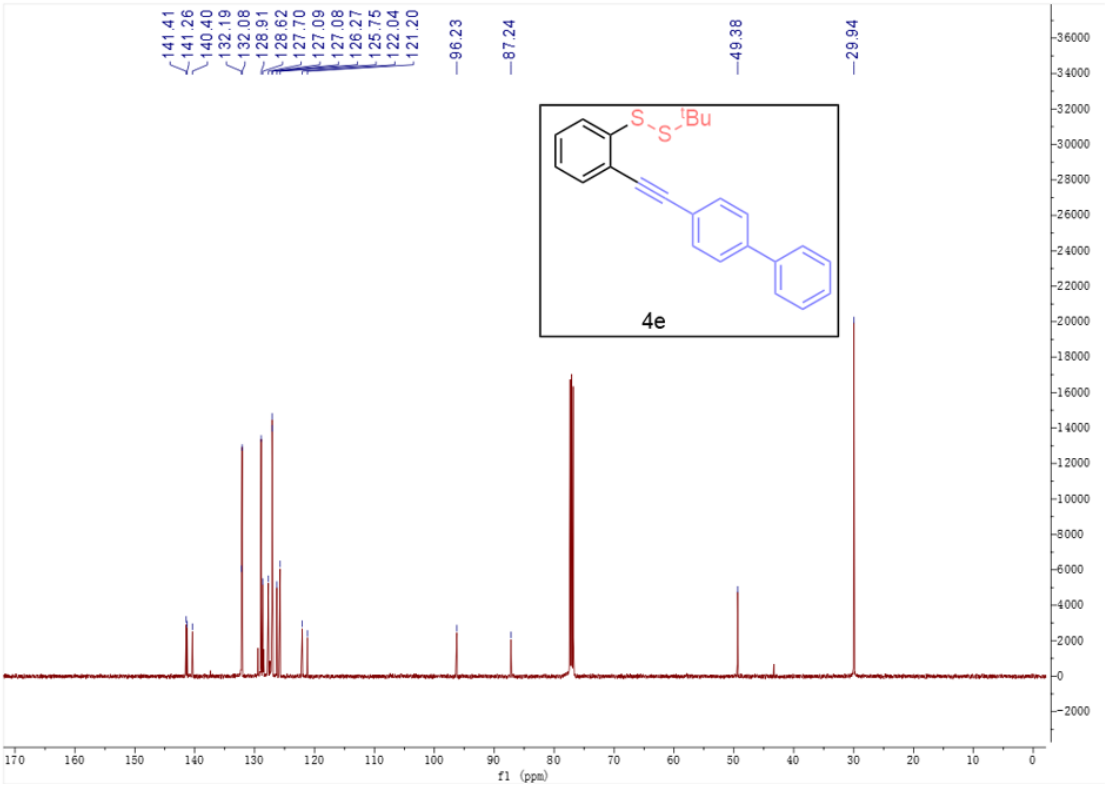
2.01
1.24
1.03
9.15
8.74
f1 (ppm)


151.84
141.30
132.09
131.39
128.40
126.18
125.65
125.40
121.36
120.11
96.48
85.88
49.31
34.85
31.20
29.91
S
S
tBu
tBu
4d
f1 (ppm)

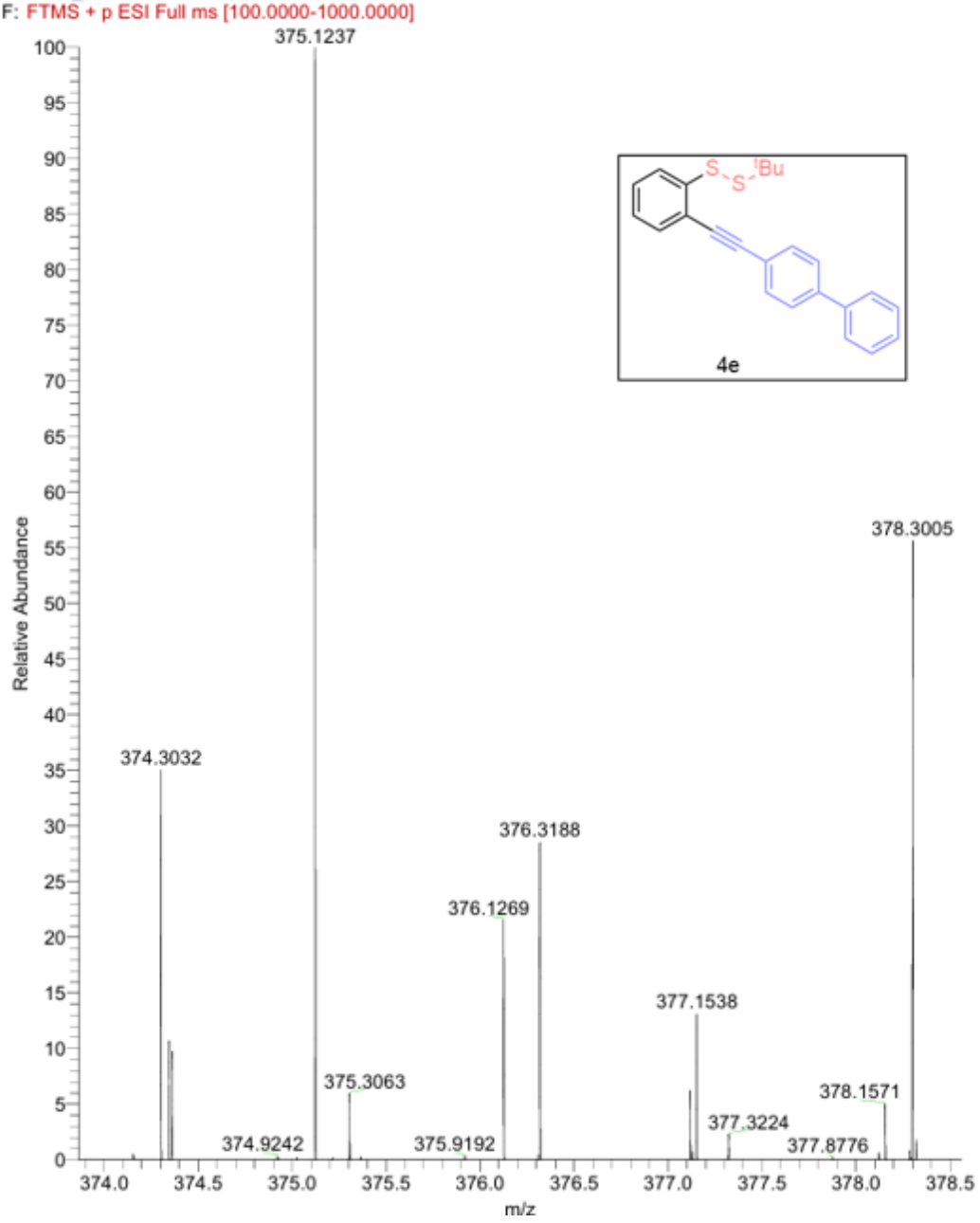

D:\DATA\231211\H7-23-4
01/29/24 14:22:54
H7-23-4 #5-8 RT: 0.09-0.13 AV: 2 NL: 5.84E5
F: FTMS + p ESI Full ms [100.0000-1000.0000]
355.1546
356.6622
356.1576
355.3396
355.0698
356.2789
354.8165
354.6715
Relative Abundance
m/z
4d

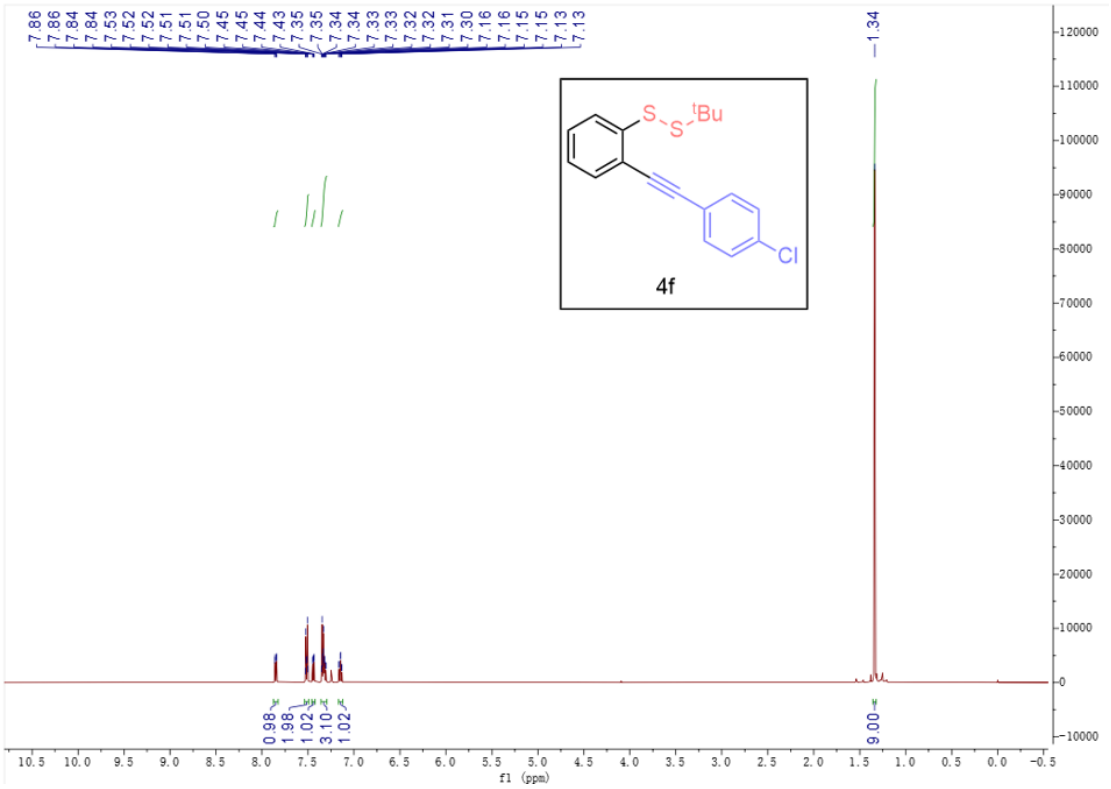

7.87
7.87
7.85
7.85
7.67
7.67
7.66
7.66
7.62
7.62
7.61
7.61
7.60
7.60
7.59
7.48
7.48
7.46
7.45
7.44
7.38
7.38
7.37
7.36
7.35
7.33
7.33
7.32
7.31
7.30
7.30
7.17
7.17
7.15
7.15
7.14
7.14
1.35
S
S
tBu
4e
0.99
2.02
3.96
3.03
1.07
1.25
0.97
9.00
f1 (ppm)

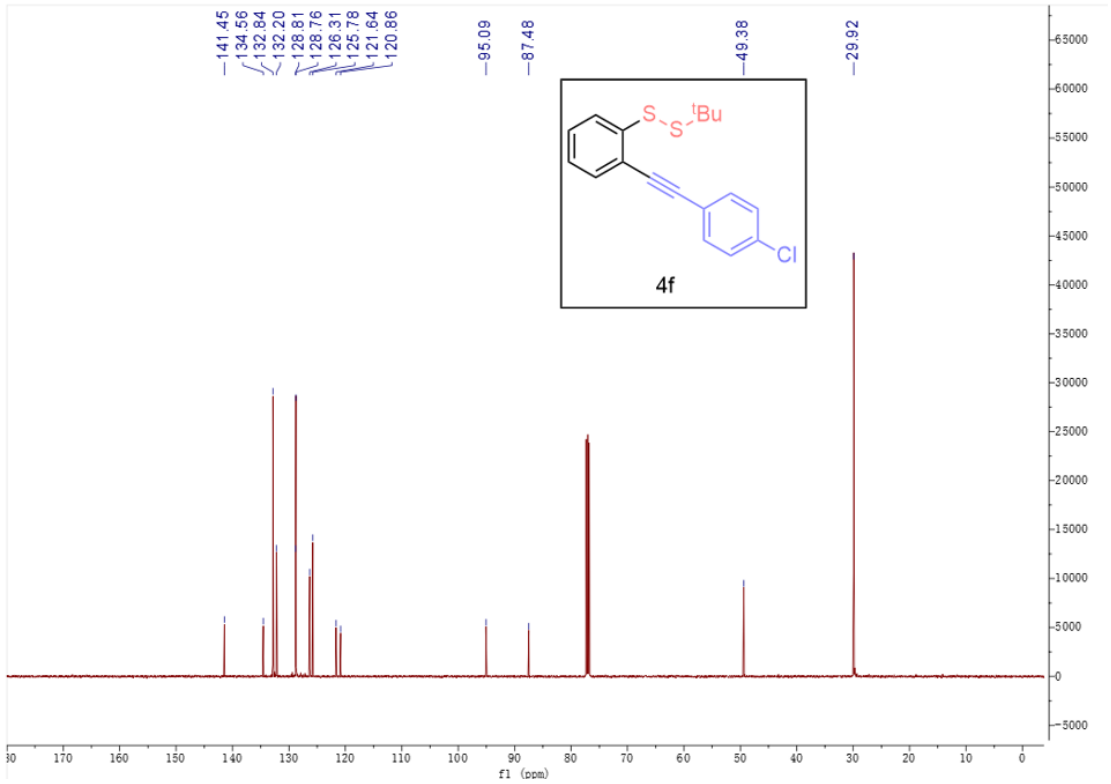

141.41
141.26
140.40
132.19
132.08
128.91
128.62
127.70
127.09
127.08
126.27
125.75
122.04
121.20
96.23
87.24
49.38
29.94
S
S
tBu
4e
f1 (ppm)

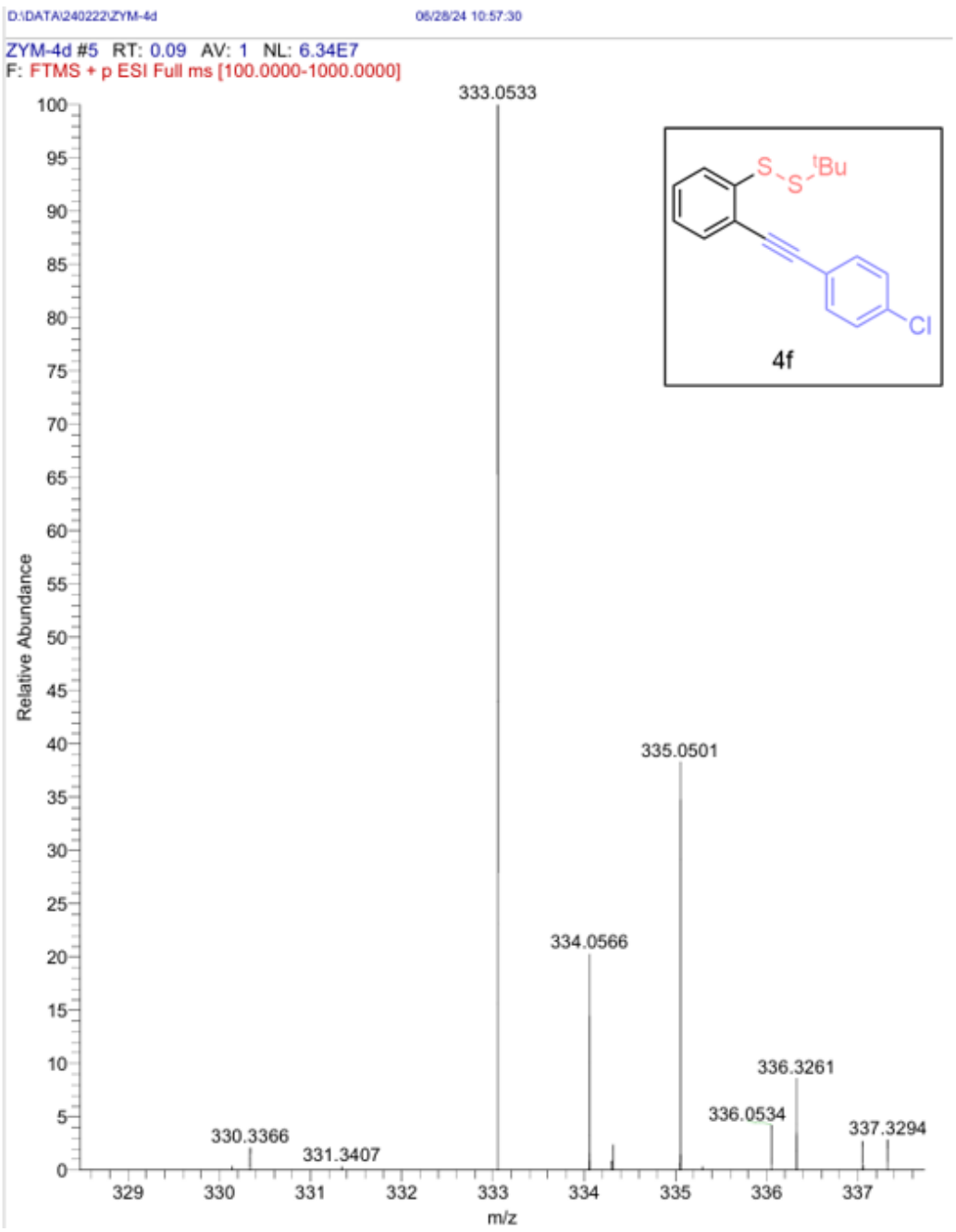
D:\DATA\231211\H7-24-9_20240304124854 03/04/24 12:49:45

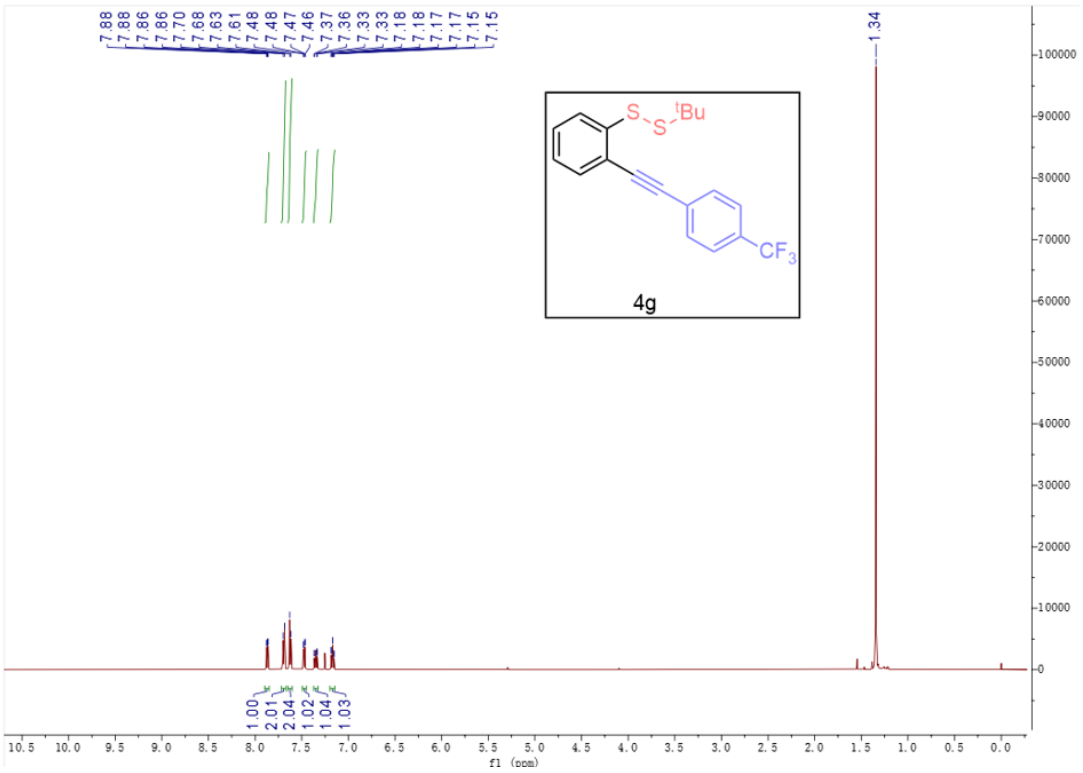
H7-24-9_20240304124854 #5-9 RT: 0.09-0.18 AV: 3 NL: 1.31E6
F: FTMS + p ESI Full ms [100.0000-1000.0000]

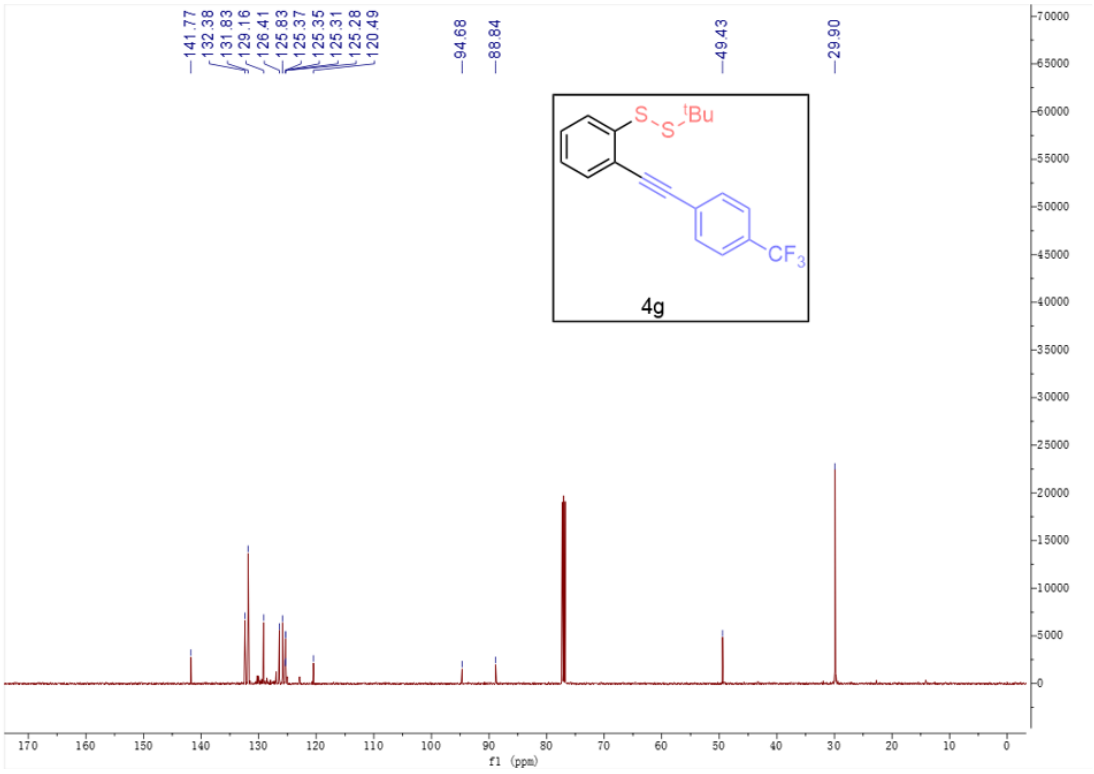


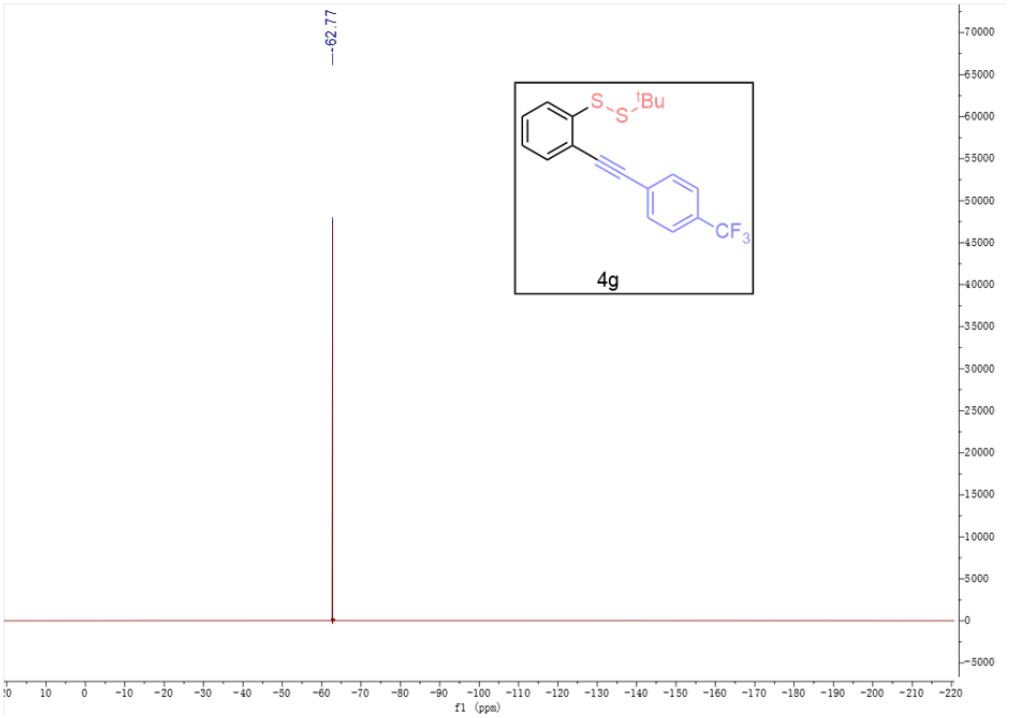

7.86
7.86
7.84
7.84
7.53
7.52
7.52
7.51
7.51
7.50
7.45
7.45
7.44
7.43
7.35
7.35
7.34
7.34
7.33
7.33
7.32
7.32
7.31
7.30
7.16
7.16
7.15
7.15
7.13
7.13
1.34
S
S
tBu
Cl
4f
0.98
1.98
1.02
3.10
1.02
9.00
f1 (ppm)

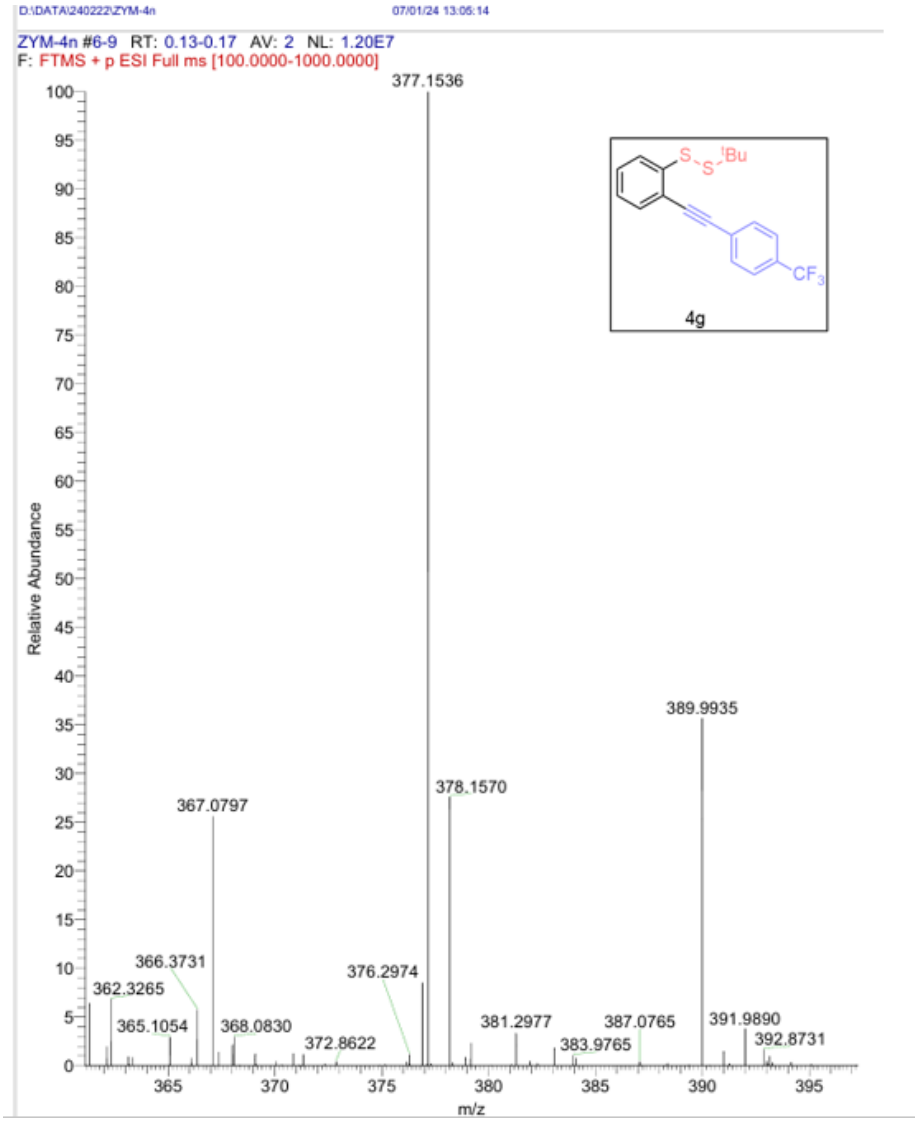

141.45
134.56
132.84
132.20
128.81
128.76
126.31
125.78
121.64
120.86
95.09
87.48
49.38
29.92
S
S
tBu
Cl
4f
f1 (ppm)

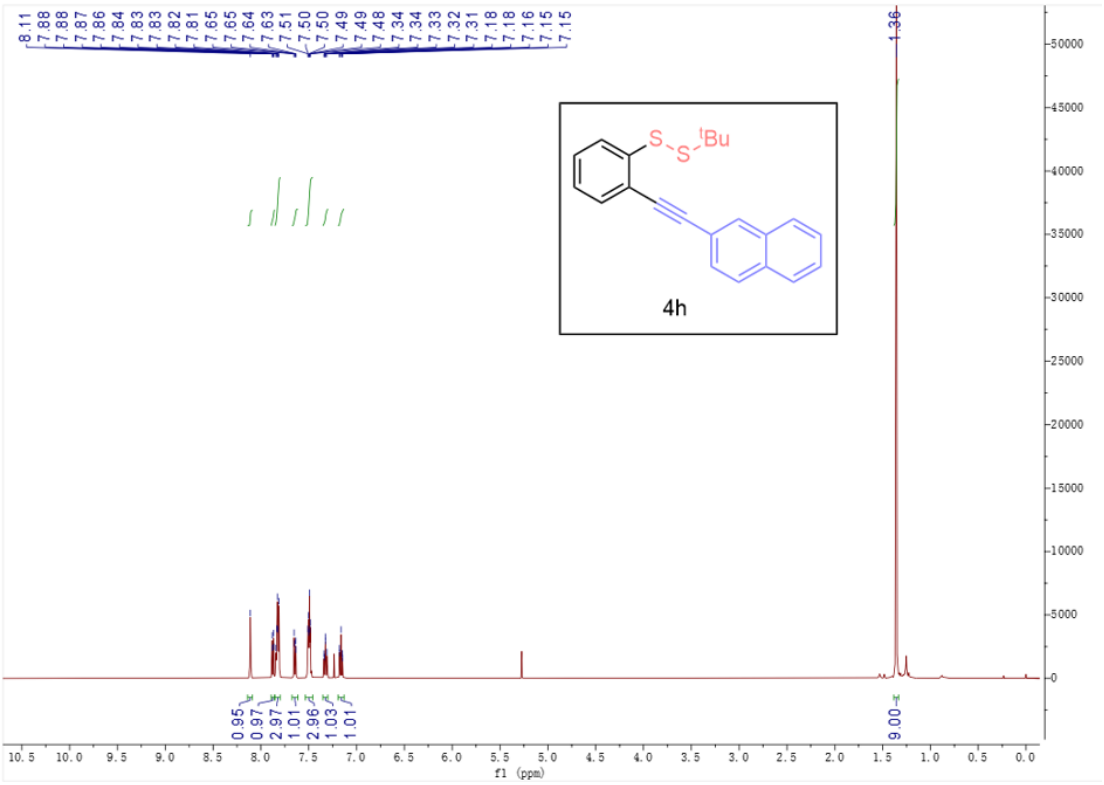

D:\DATA\240222\ZYM-4d
08/28/24 10:57:30
ZYM-4d #5 RT: 0.09 AV: 1 NL: 6.34E7
F: FTMS + p ESI Full ms [100.0000-1000.0000]
333.0533
335.0501
334.0566
336.3261
336.0534
337.3294
330.3366
331.3407
Relative Abundance
m/z
S
S
tBu
Cl
4f

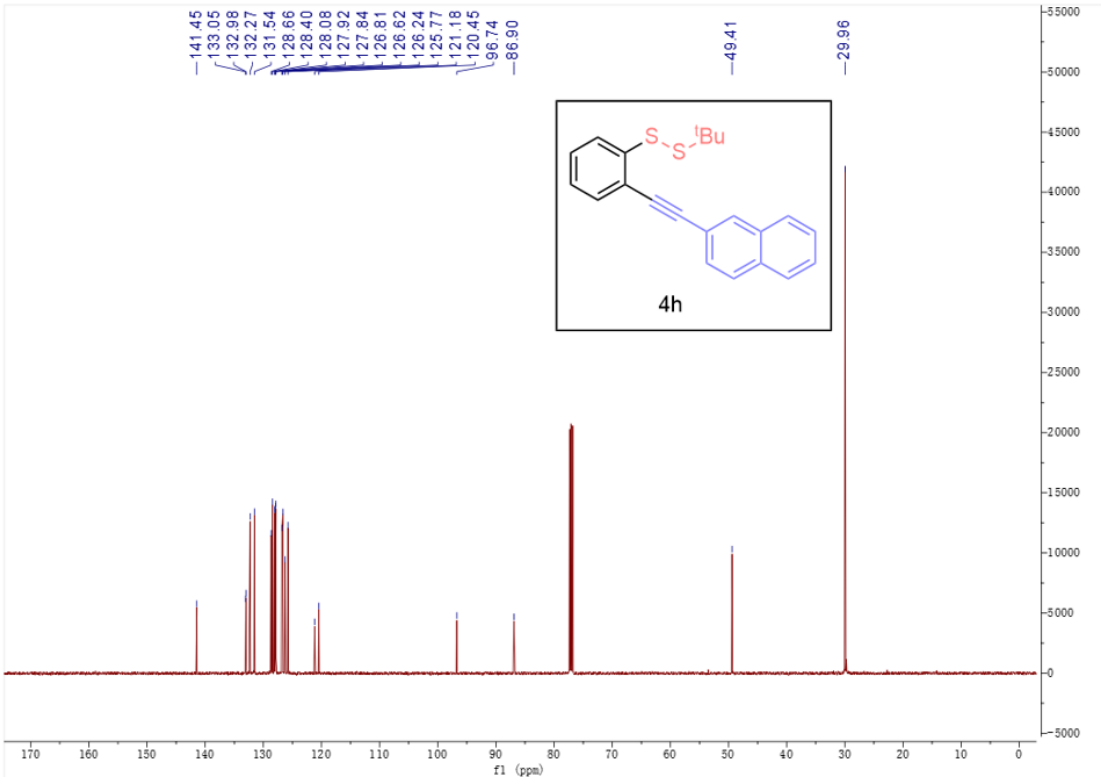

7.88
7.88
7.86
7.86
7.70
7.68
7.63
7.61
7.48
7.48
7.47
7.46
7.37
7.36
7.33
7.33
7.18
7.18
7.17
7.17
7.15
7.15
1.34
S
S
tBu
CF3
4g
1.00
2.01
2.04
1.02
1.04
1.03
f1 (ppm)

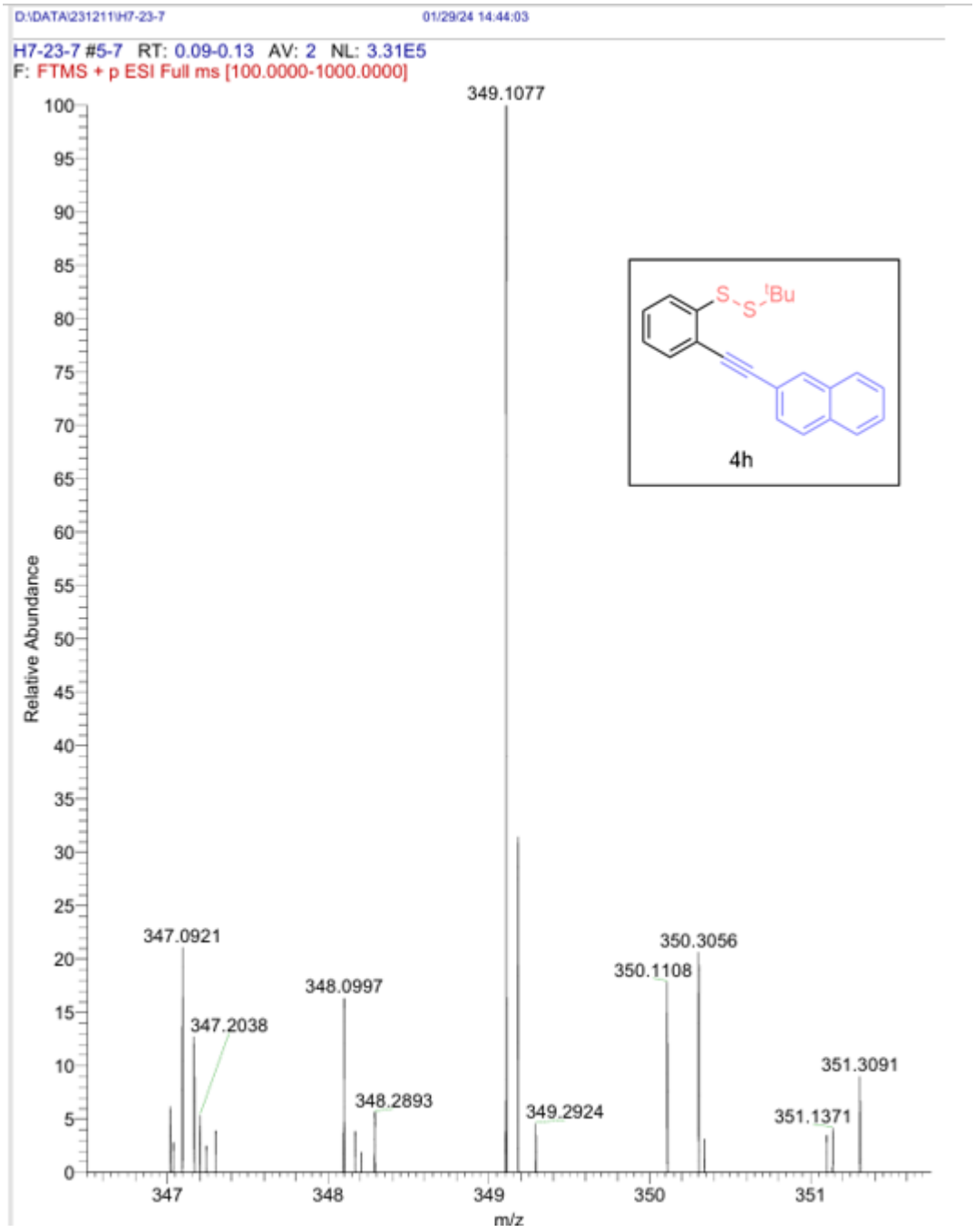

141.77
132.38
131.83
129.16
126.41
125.83
125.37
125.35
125.31
125.28
120.49
94.68
88.84
49.43
29.90
S
S
tBu
CF3
4g
f1 (ppm)

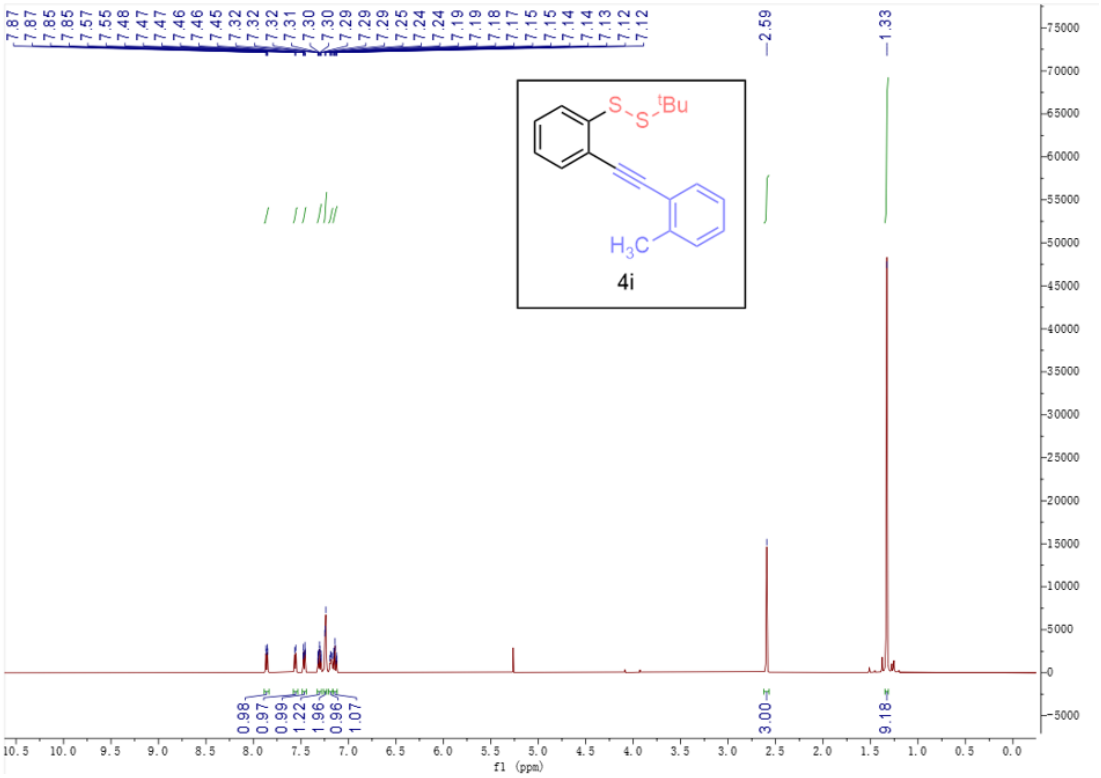

62.77
S
S
tBu
CF3
4g
f1 (ppm)

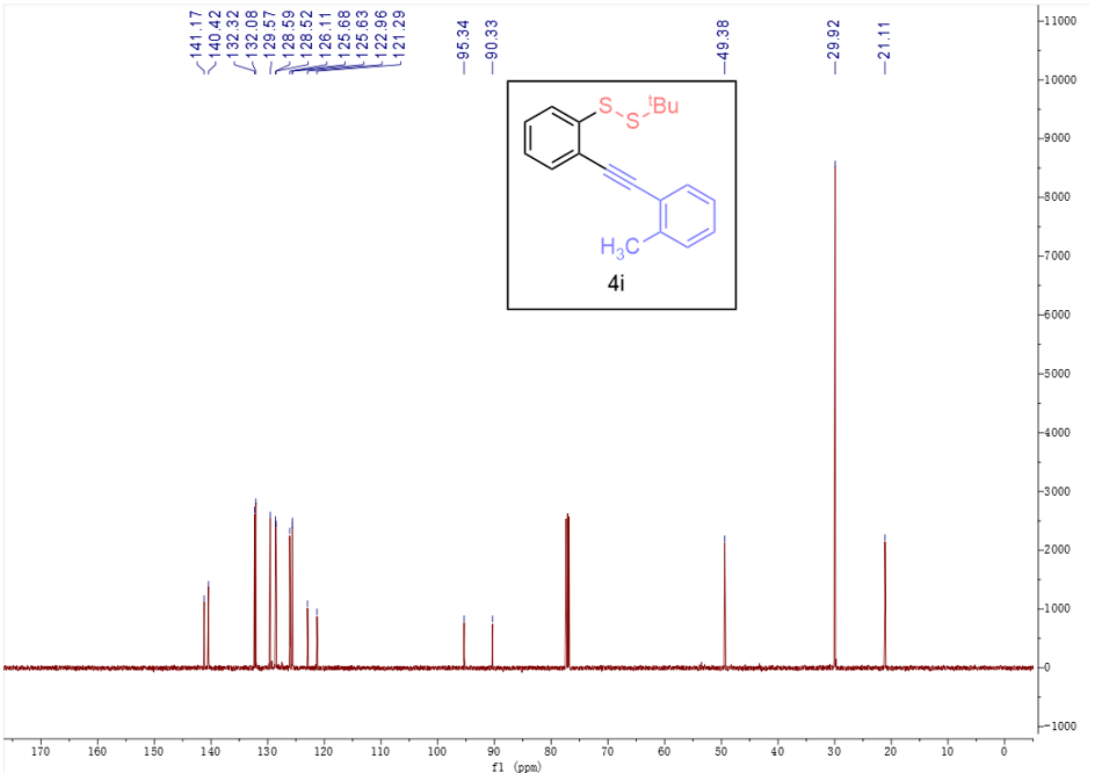

D:\DATA\240222\ZYM-4n
07/01/24 13:05:14
ZYM-4n #6-9 RT: 0.13-0.17 AV: 2 NL: 1.20E7
F: FTMS + p ESI Full ms [100.0000-1000.0000]
377.1536
389.9935
378.1570
367.0797
366.3731
376.2974
362.3265
365.1054
368.0830
372.8622
381.2977
387.0765
383.9765
391.9890
392.8731
Relative Abundance
m/z
4g


8.11
7.88
7.88
7.87
7.86
7.84
7.83
7.83
7.82
7.81
7.65
7.64
7.63
7.51
7.50
7.50
7.49
7.49
7.48
7.34
7.34
7.33
7.32
7.31
7.18
7.18
7.16
7.15
7.15
1.36
S
S
tBu
4h
0.95
0.97
2.97
1.01
2.96
1.03
1.01
9.00
f1 (ppm)


141.45
133.05
132.98
132.27
131.54
128.66
128.40
128.08
127.92
127.84
126.81
126.62
126.24
125.77
121.18
120.45
96.74
86.90
49.41
29.96
S
S
tBu
4h
f1 (ppm)

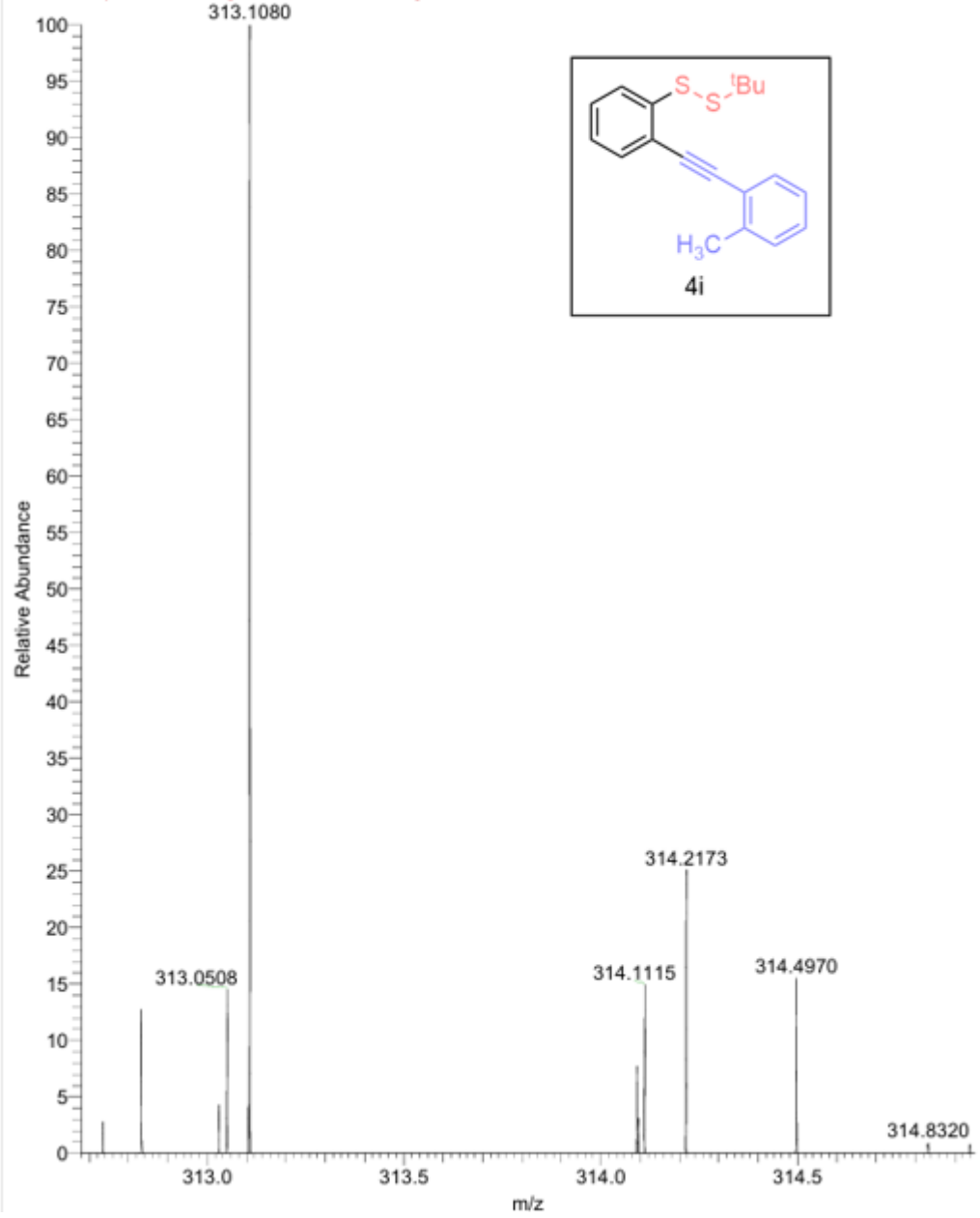

D:\DATA\231211\H7-23-7
01/29/24 14:44:03
H7-23-7 #5-7 RT: 0.09-0.13 AV: 2 NL: 3.31E5
F: FTMS + p ESI Full ms [100.0000-1000.0000]
349.1077
347.0921
347.2038
348.0997
348.2893
349.2924
350.3056
350.1108
351.3091
351.1371
S
S
tBu
4h
Relative Abundance
m/z
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
347
348
349
350
351

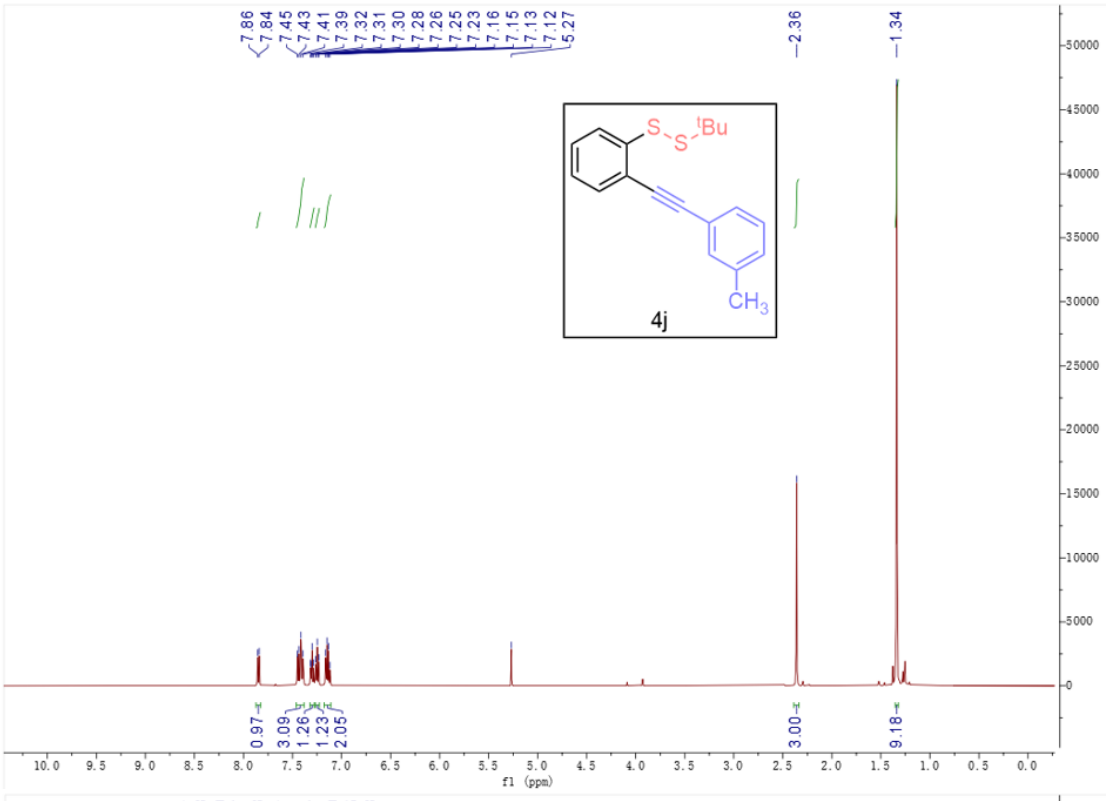

7.87
7.87
7.85
7.85
7.57
7.55
7.48
7.47
7.47
7.46
7.46
7.45
7.32
7.32
7.32
7.31
7.30
7.30
7.29
7.29
7.29
7.25
7.24
7.24
7.19
7.19
7.18
7.17
7.15
7.15
7.14
7.14
7.13
7.12
7.12
2.59
1.33
S
S
tBu
H3C
4i
0.98
0.97
0.99
1.22
1.96
0.96
1.07
3.00
9.18
f1 (ppm)

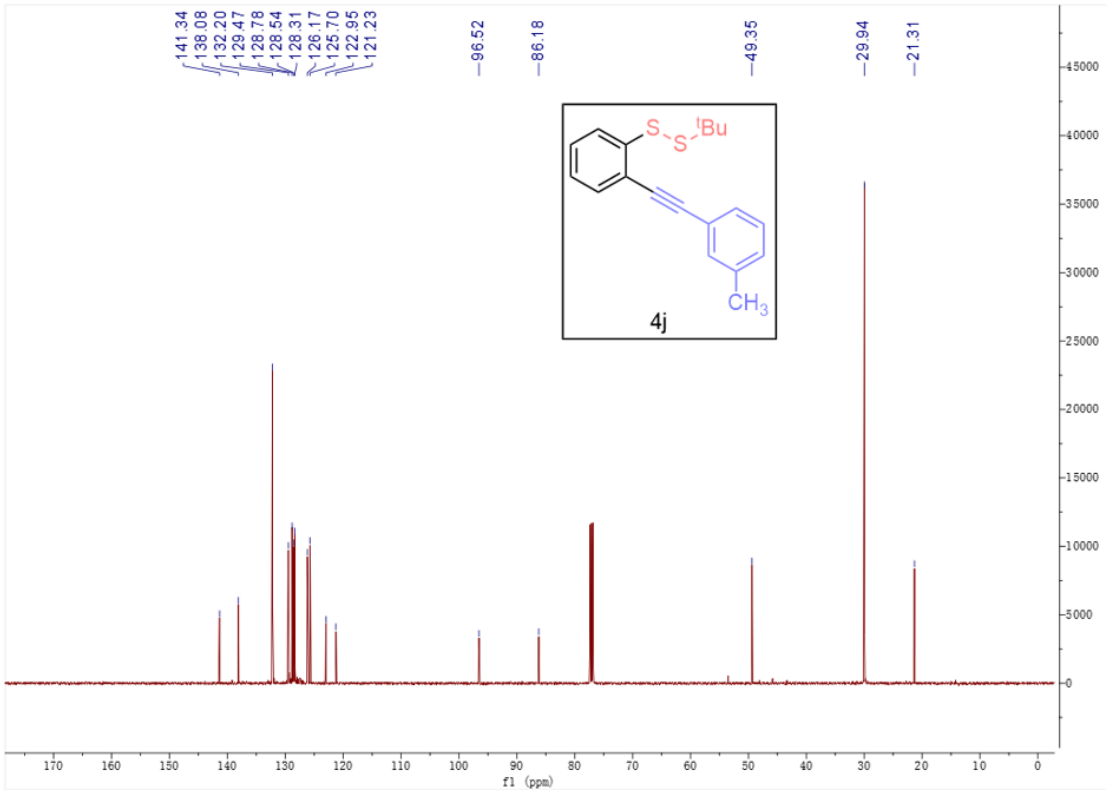

141.17
140.42
132.32
132.08
129.57
128.59
128.52
126.11
125.68
125.63
122.96
121.29
95.34
90.33
49.38
29.92
21.11
S
S
tBu
H3C
4i
f1 (ppm)

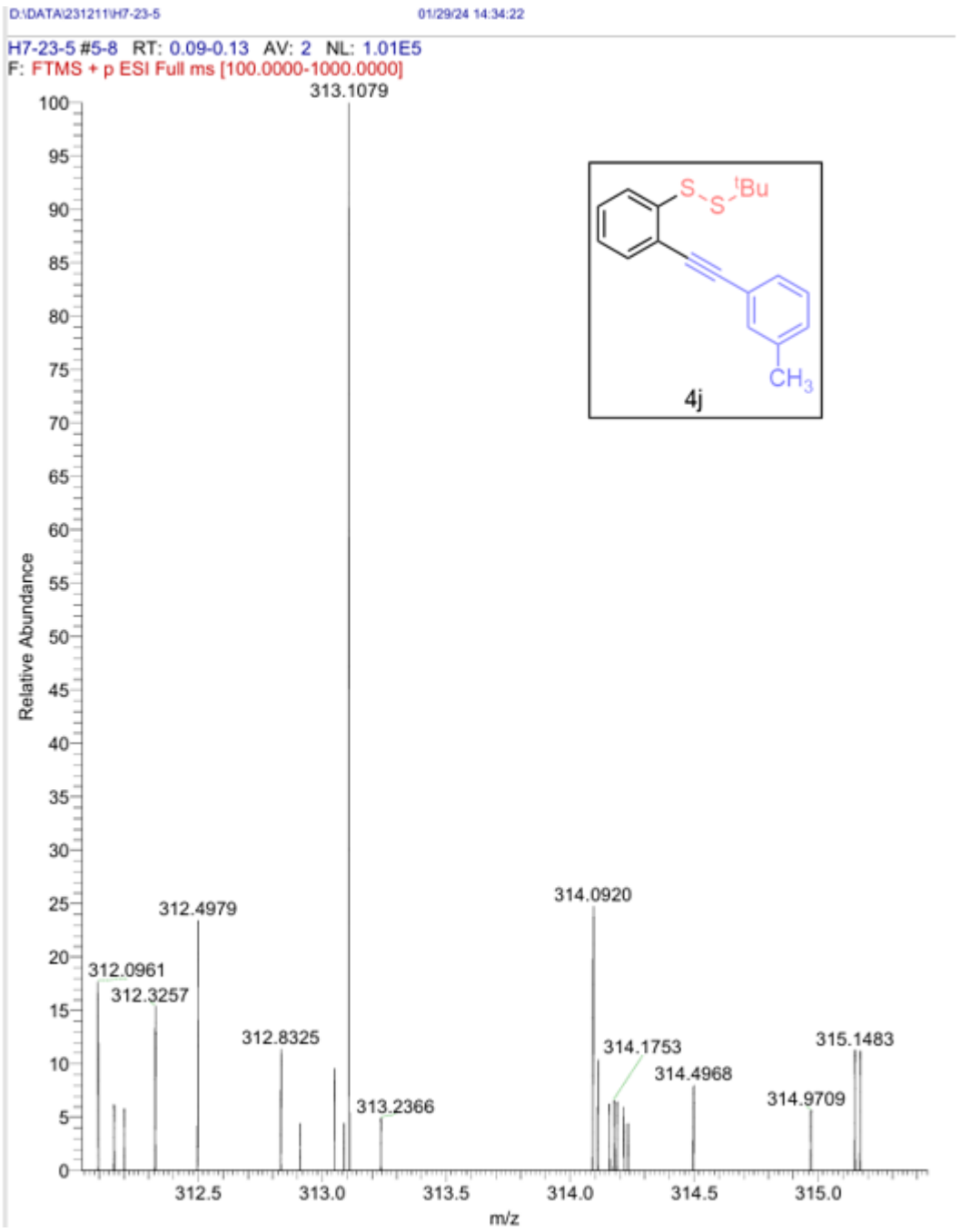
D:\DATA\231211\H7-23-6 01/29/24 14:39:13

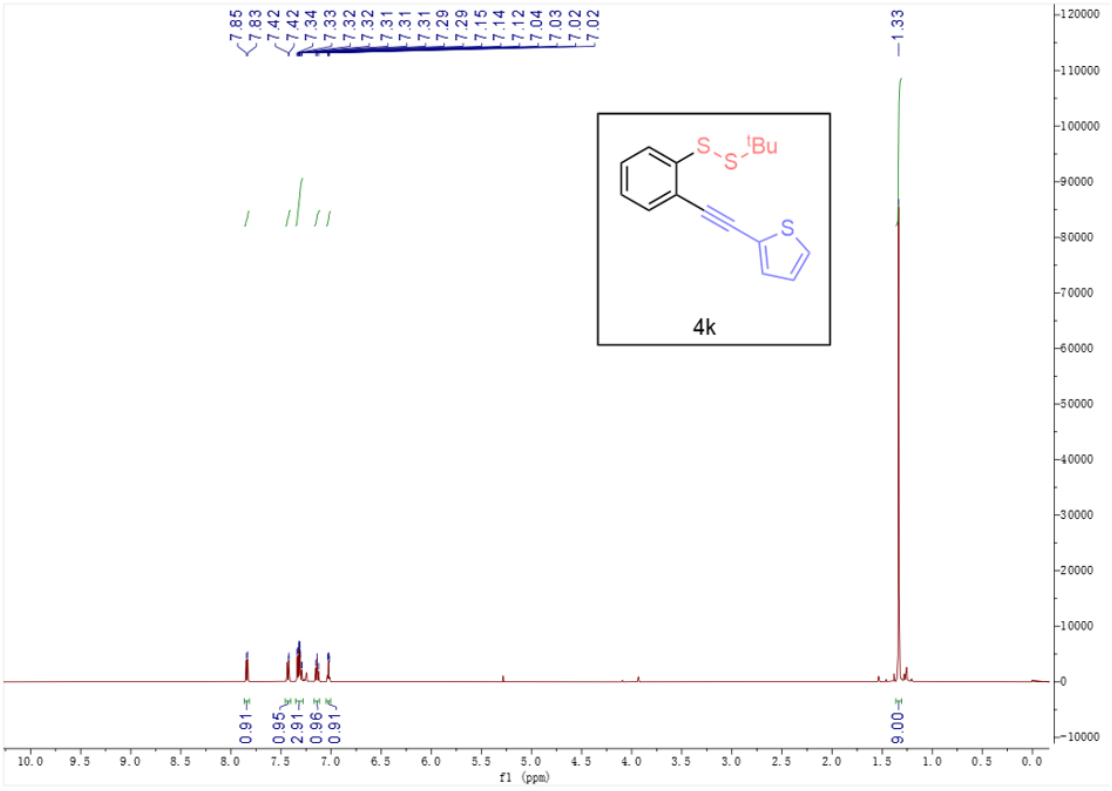
H7-23-6 #5-9 RT: 0.09-0.16 AV: 3 NL: 1.52E5
F: FTMS + p ESI Full ms [100.0000-1000.0000]

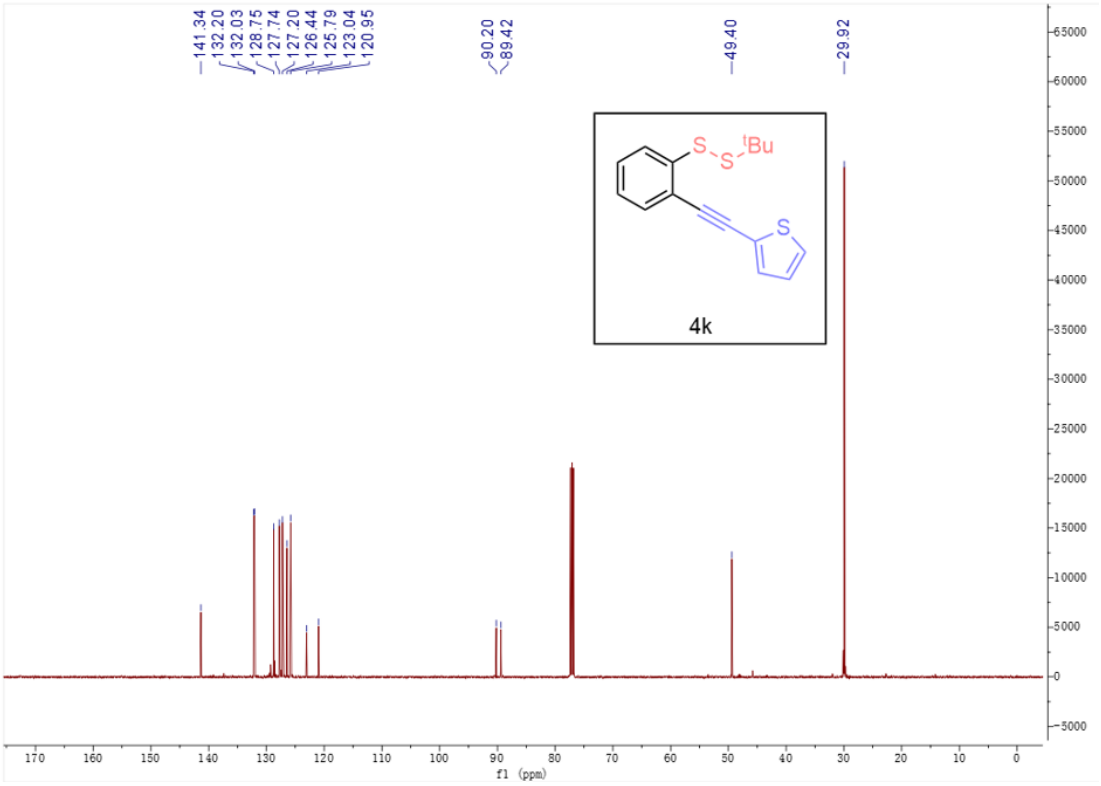



7.86
7.84
7.45
7.43
7.41
7.39
7.32
7.31
7.30
7.28
7.26
7.25
7.23
7.16
7.15
7.13
7.12
5.27
2.36
1.34
S
S
tBu
CH3
4j
0.97
3.09
1.26
1.23
2.05
3.00
9.18
f1 (ppm)


141.34
138.08
132.20
129.47
128.78
128.54
128.31
126.17
125.70
122.95
121.23
96.52
86.18
49.35
29.94
21.31
S
S
tBu
CH3
4j
f1 (ppm)

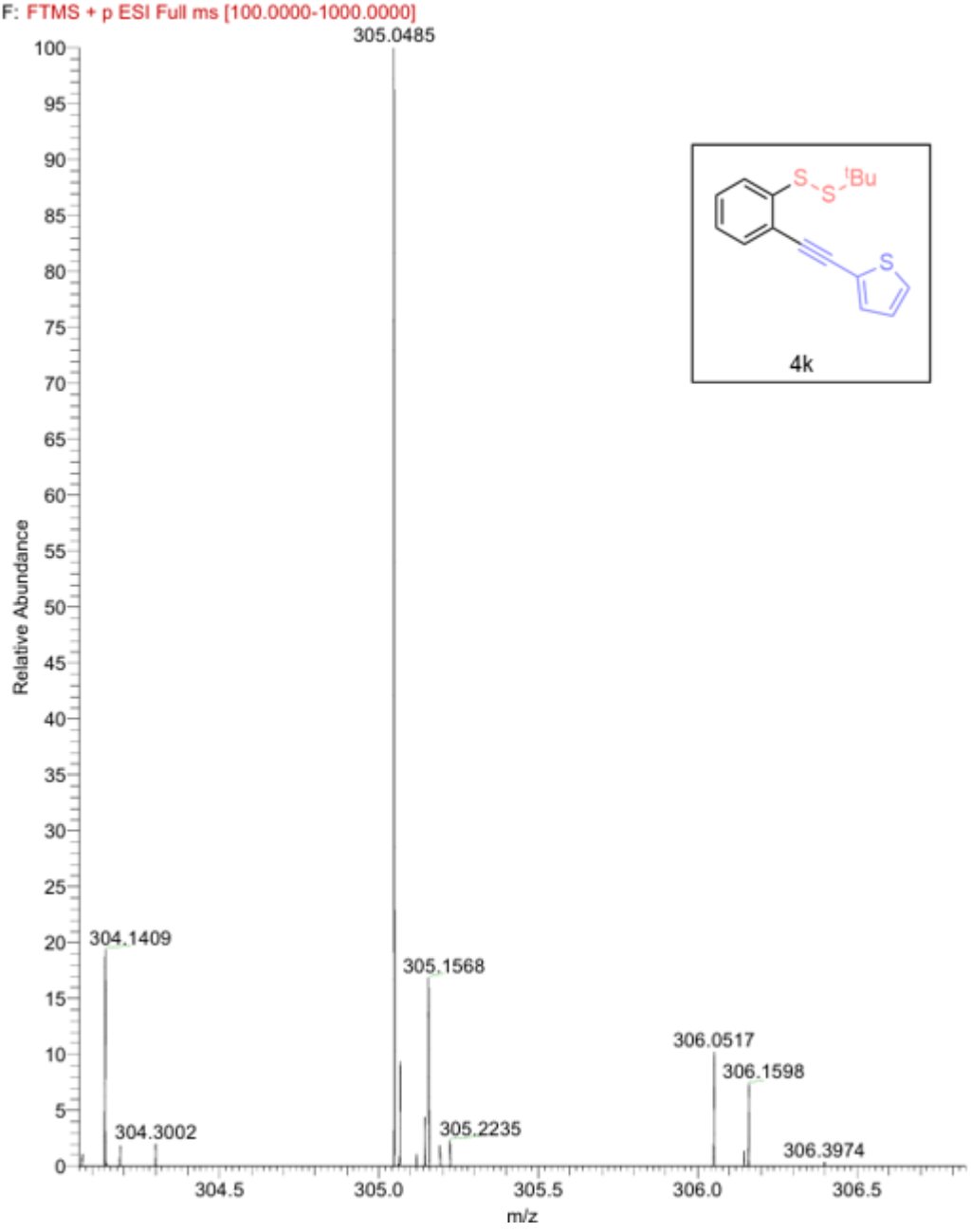

D:\DATA\231211\H7-23-5
01/29/24 14:34:22
H7-23-5 #5-8 RT: 0.09-0.13 AV: 2 NL: 1.01E5
F: FTMS + p ESI Full ms [100.0000-1000.0000]
313.1079
312.4979
312.0961
312.3257
312.8325
313.2366
314.0920
314.1753
314.4968
314.9709
315.1483
Relative Abundance
m/z
4j

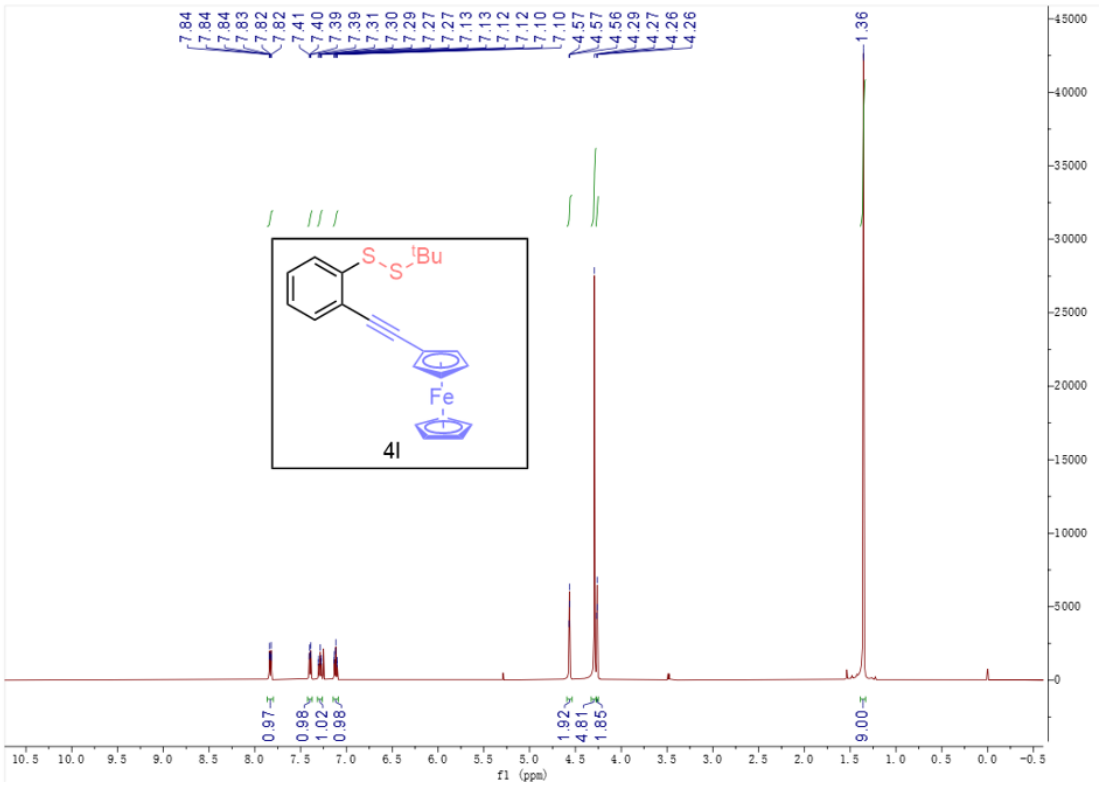

7.85
7.83
7.42
7.42
7.34
7.33
7.32
7.32
7.31
7.31
7.31
7.29
7.29
7.15
7.14
7.12
7.04
7.03
7.02
7.02
1.33
0.91
0.95
2.91
0.96
0.91
9.00
S
S
tBu
S
4k
f1 (ppm)

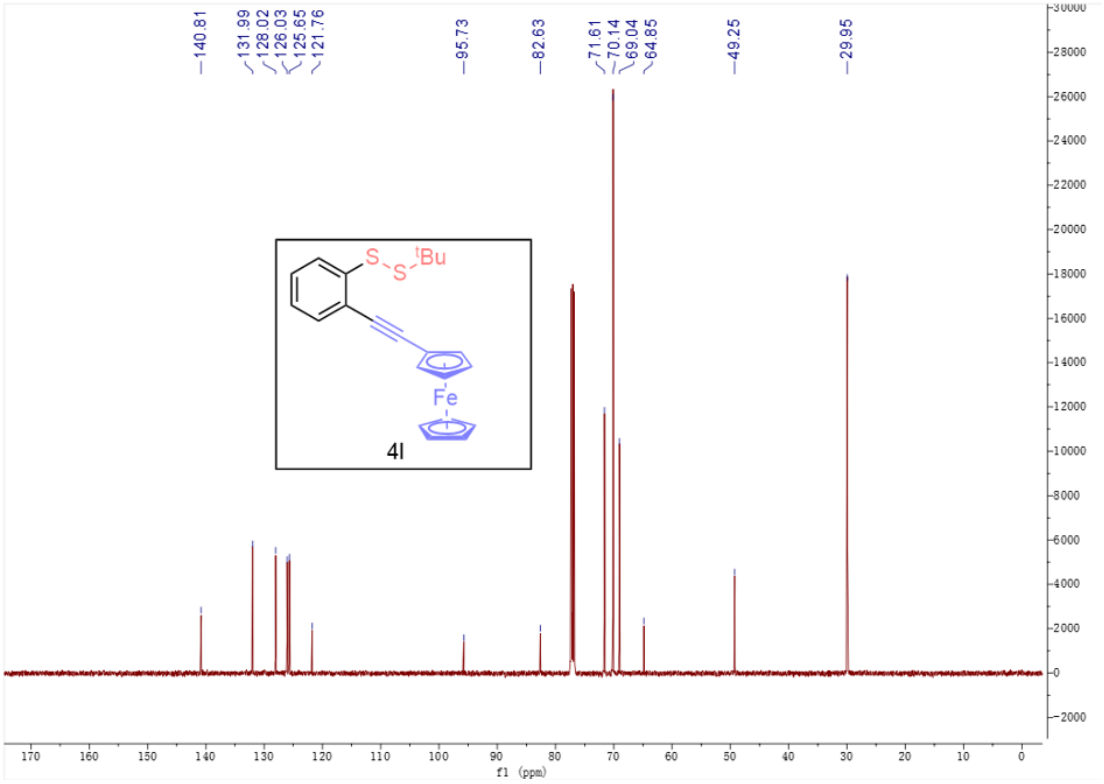

141.34
132.20
132.03
128.75
127.74
127.20
126.44
125.79
123.04
120.95
90.20
89.42
49.40
29.92
S
S
tBu
S
4k
f1 (ppm)

D:\DATA\231211\H7-23-9 01/29/24 14:48:53

H7-23-9 #4-9 RT: 0.09-0.16 AV: 3 NL: 4.66E5
F: FTMS + p ESI Full ms [100.0000-1000.0000]

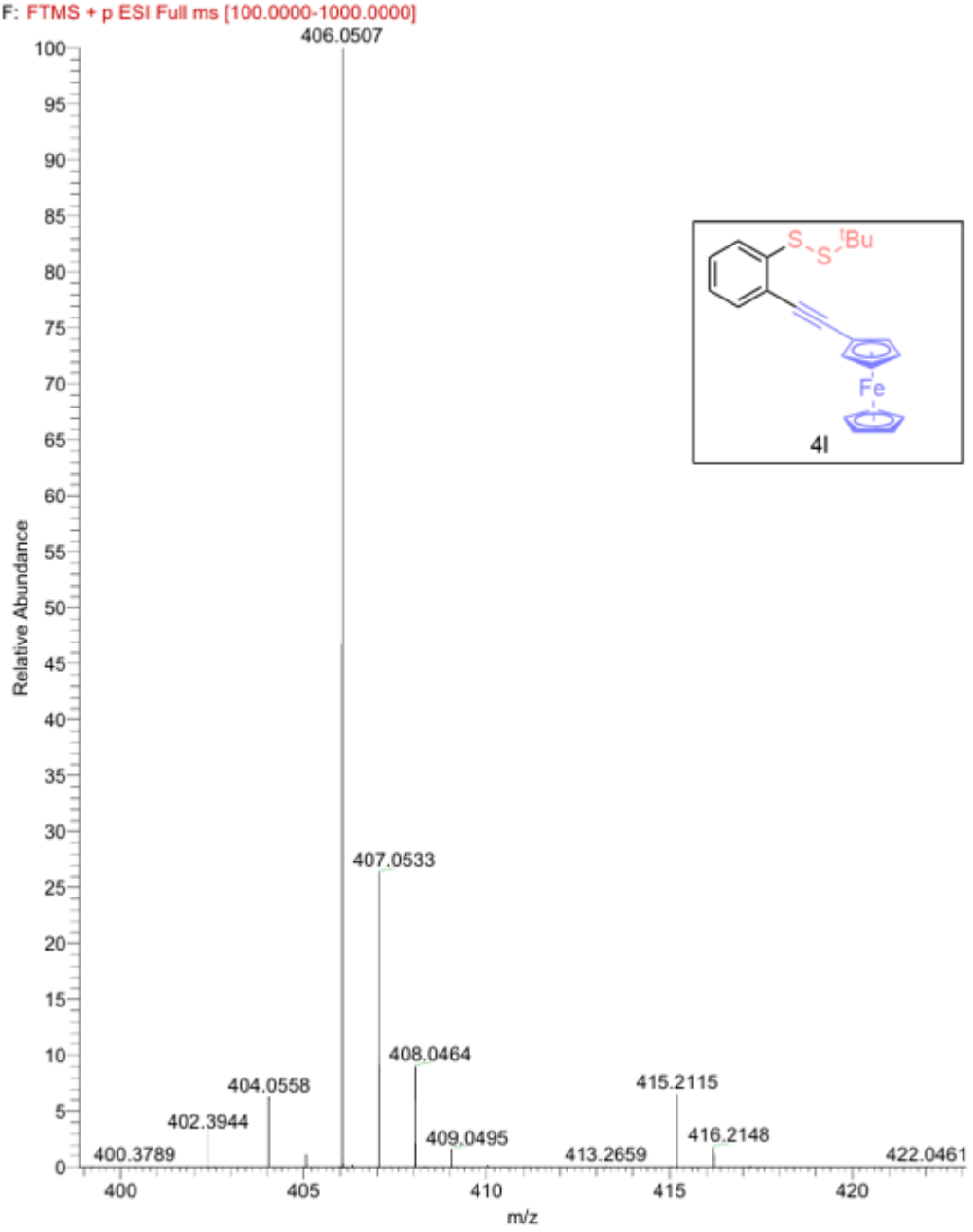



7.84
7.84
7.84
7.83
7.82
7.82
7.41
7.40
7.39
7.39
7.31
7.30
7.29
7.27
7.27
7.13
7.12
7.12
7.10
7.10
4.57
4.57
4.56
4.29
4.27
4.26
4.26
1.36
S
S
tBu
Fe
4l
0.97
0.98
1.02
0.98
1.92
4.81
1.85
9.00
f1 (ppm)

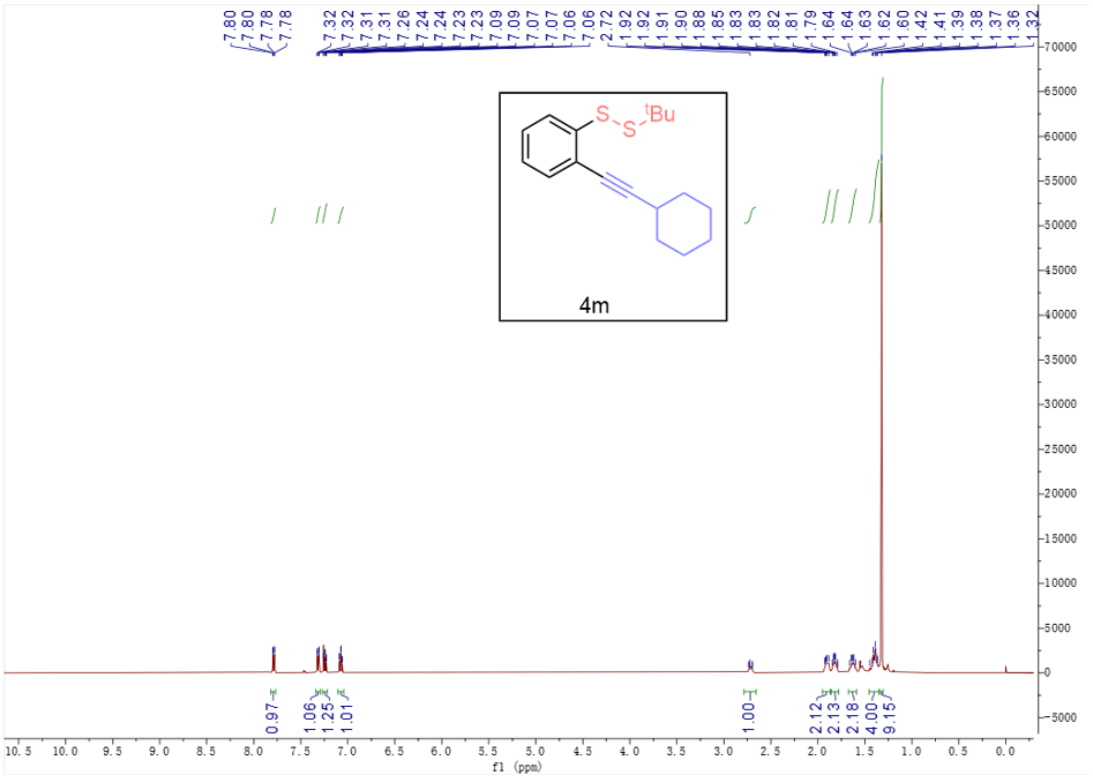

140.81
131.99
128.02
126.03
125.65
121.76
95.73
82.63
71.61
70.14
69.04
64.85
49.25
29.95
S
S
tBu
Fe
4l
f1 (ppm)

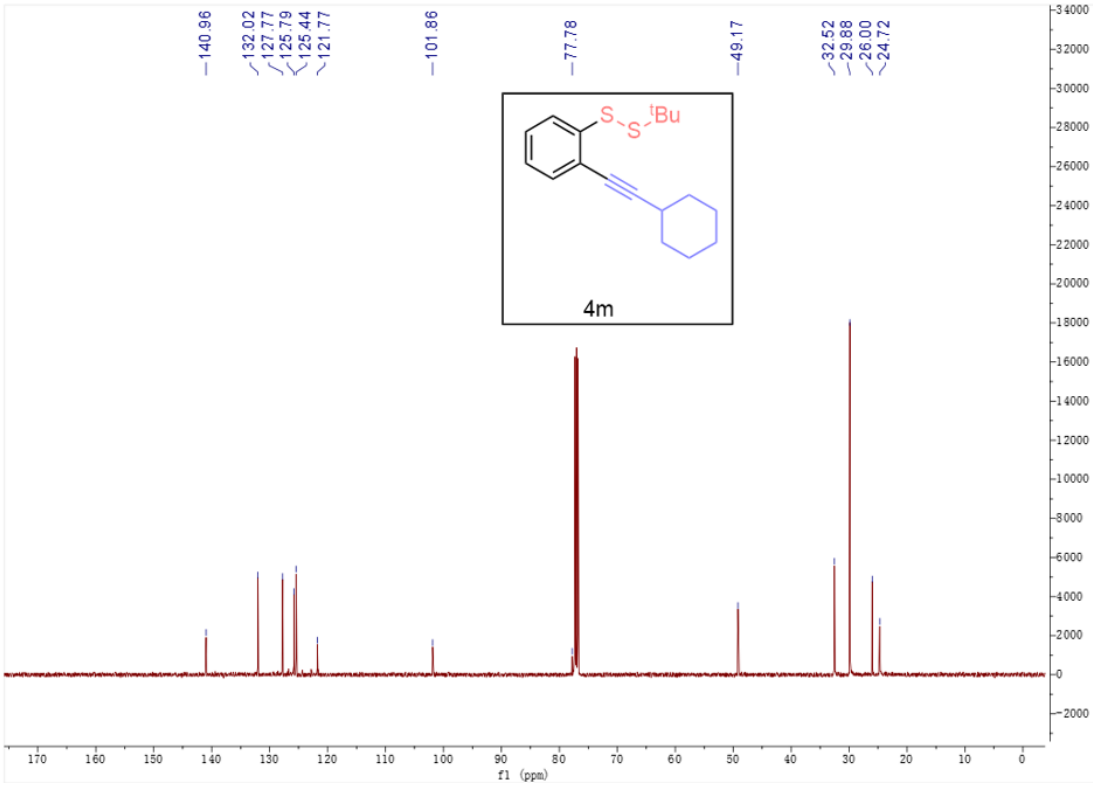
D:\DATA\240222\ZYM-4L 06/28/24 11:07:10

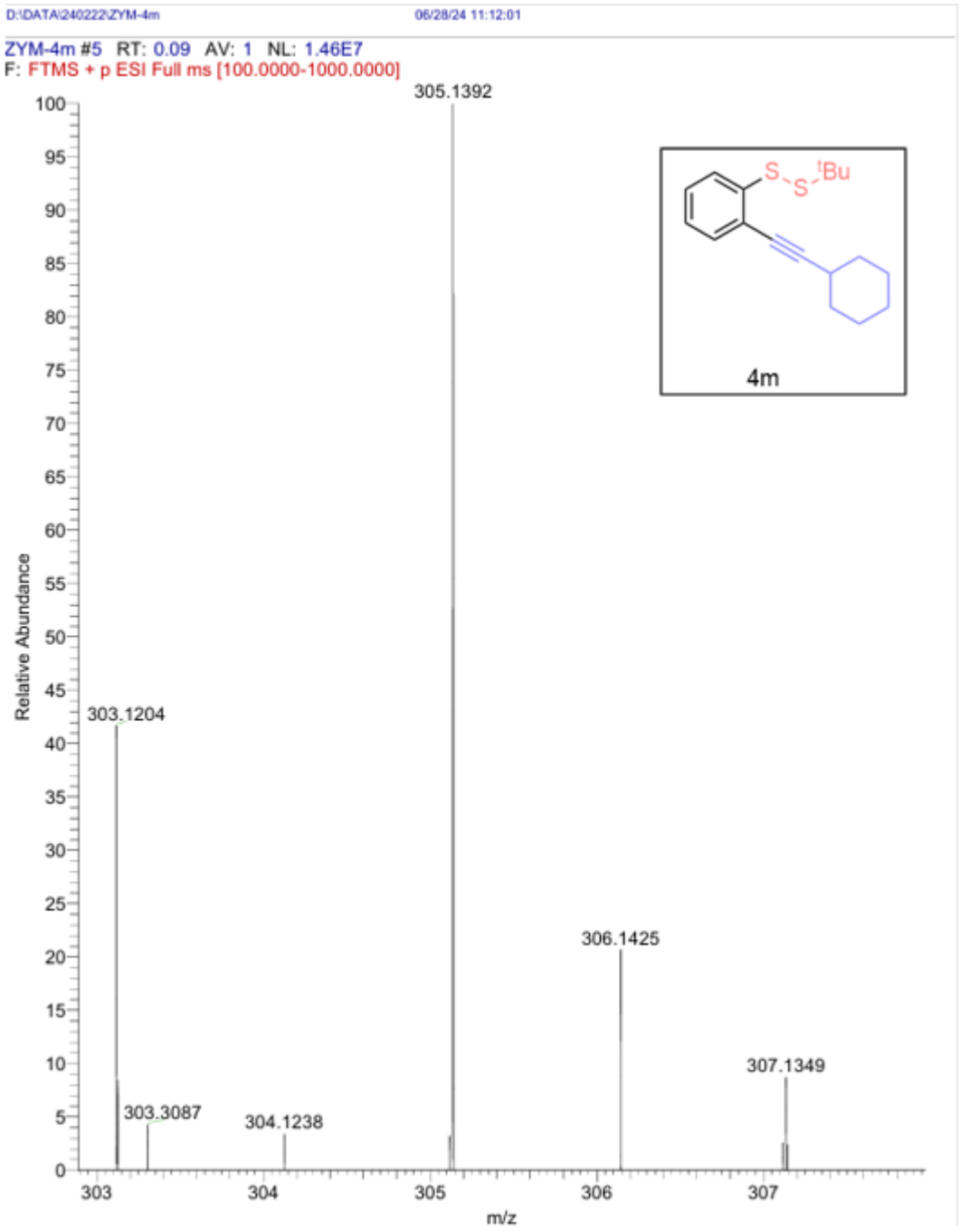
ZYM-4L #6 RT: 0.14 AV: 1 NL: 9.04E7
F: FTMS + p ESI Full ms [100.0000-1000.0000]

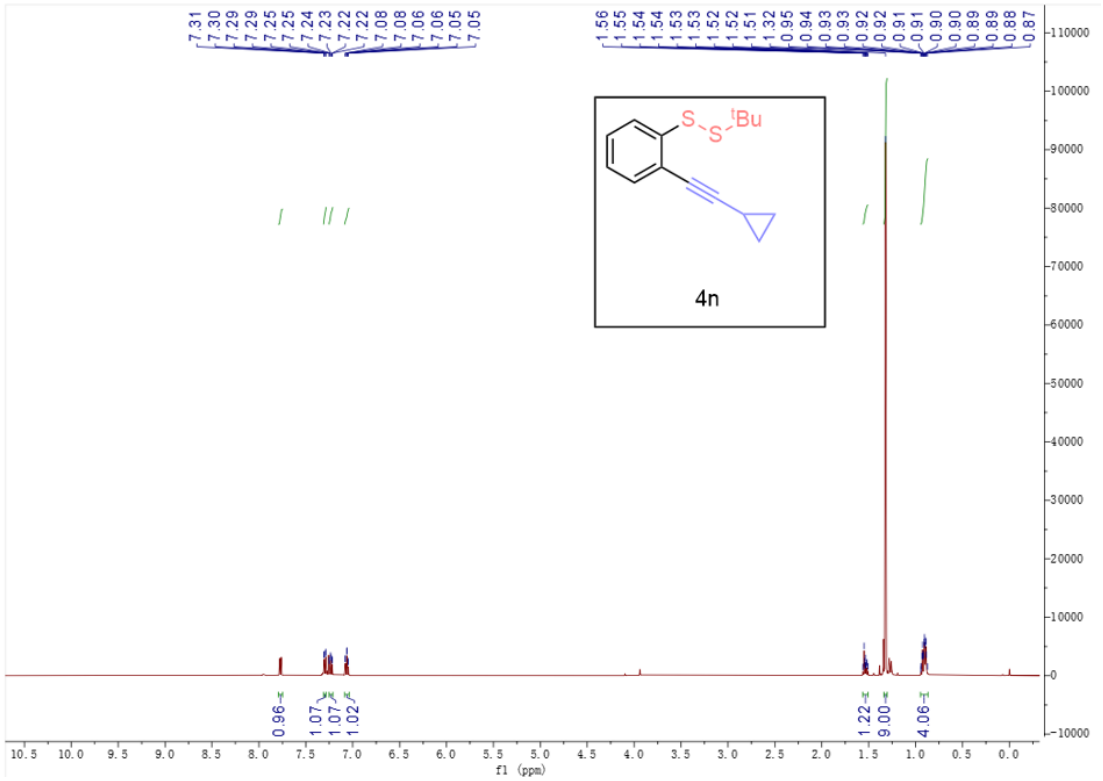


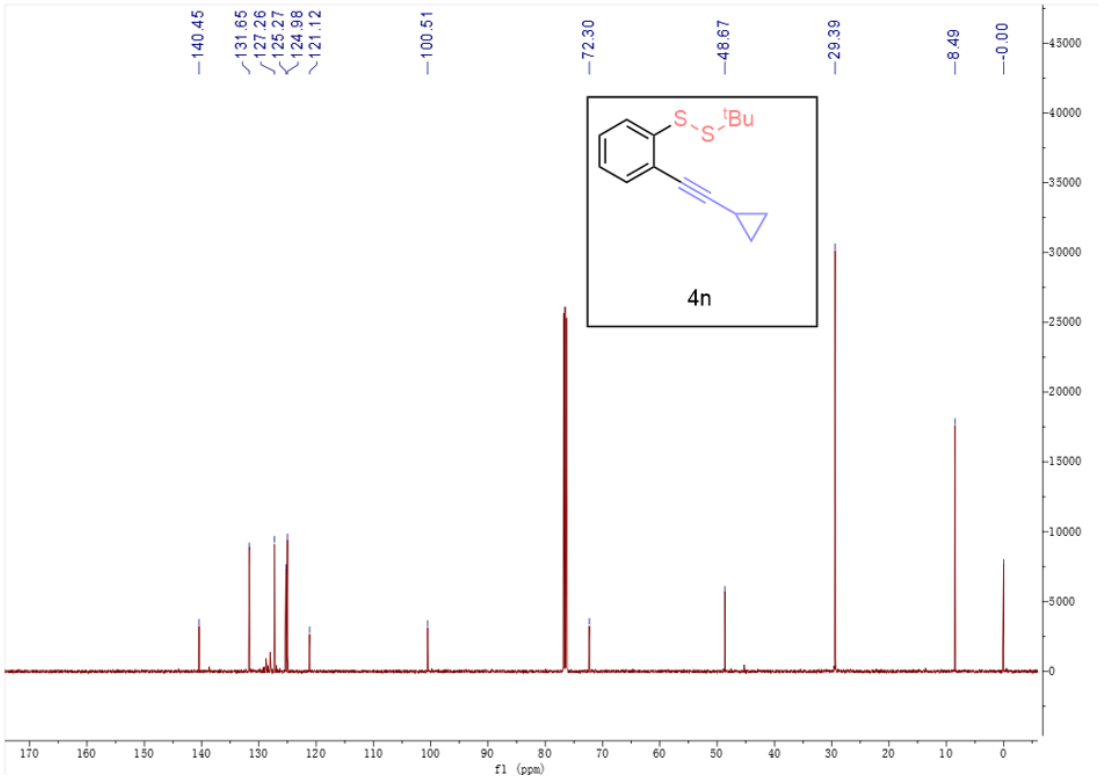
4l

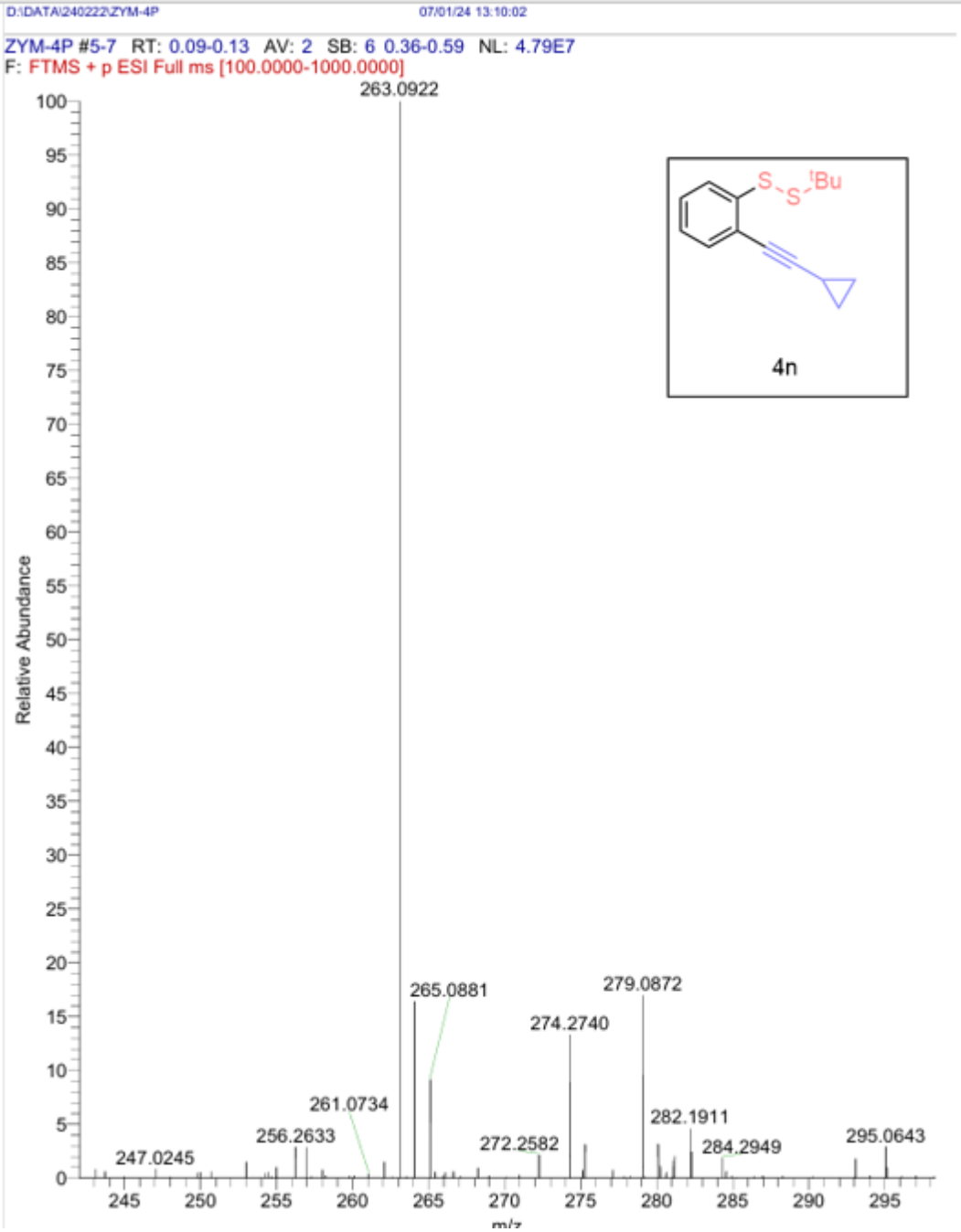

7.80
7.80
7.78
7.78
7.32
7.32
7.31
7.31
7.26
7.24
7.24
7.23
7.23
7.09
7.09
7.07
7.07
7.06
7.06
2.72
1.92
1.92
1.91
1.90
1.88
1.85
1.83
1.83
1.82
1.81
1.79
1.64
1.64
1.63
1.62
1.60
1.42
1.41
1.39
1.38
1.37
1.36
1.32
S
S
tBu
4m
0.97
1.06
1.25
1.01
1.00
2.12
2.13
2.18
4.00
9.15
f1 (ppm)

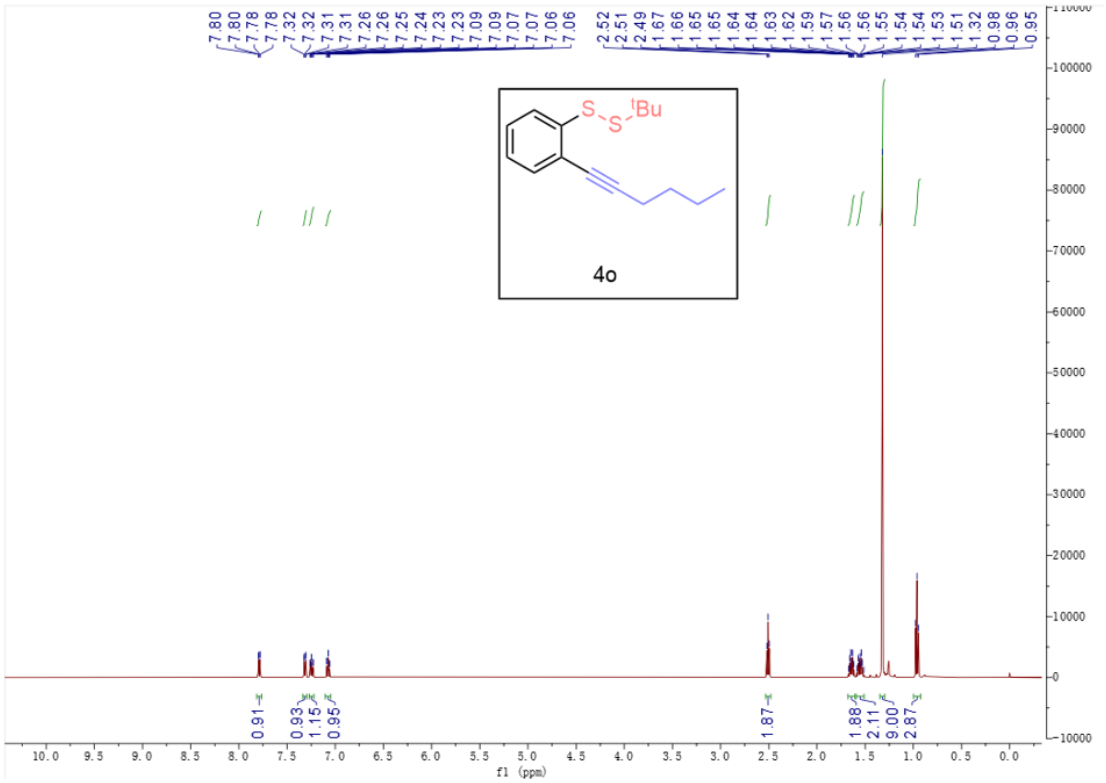

140.96
132.02
127.77
125.79
125.44
121.77
101.86
77.78
49.17
32.52
29.88
26.00
24.72
S
S
tBu
4m
f1 (ppm)

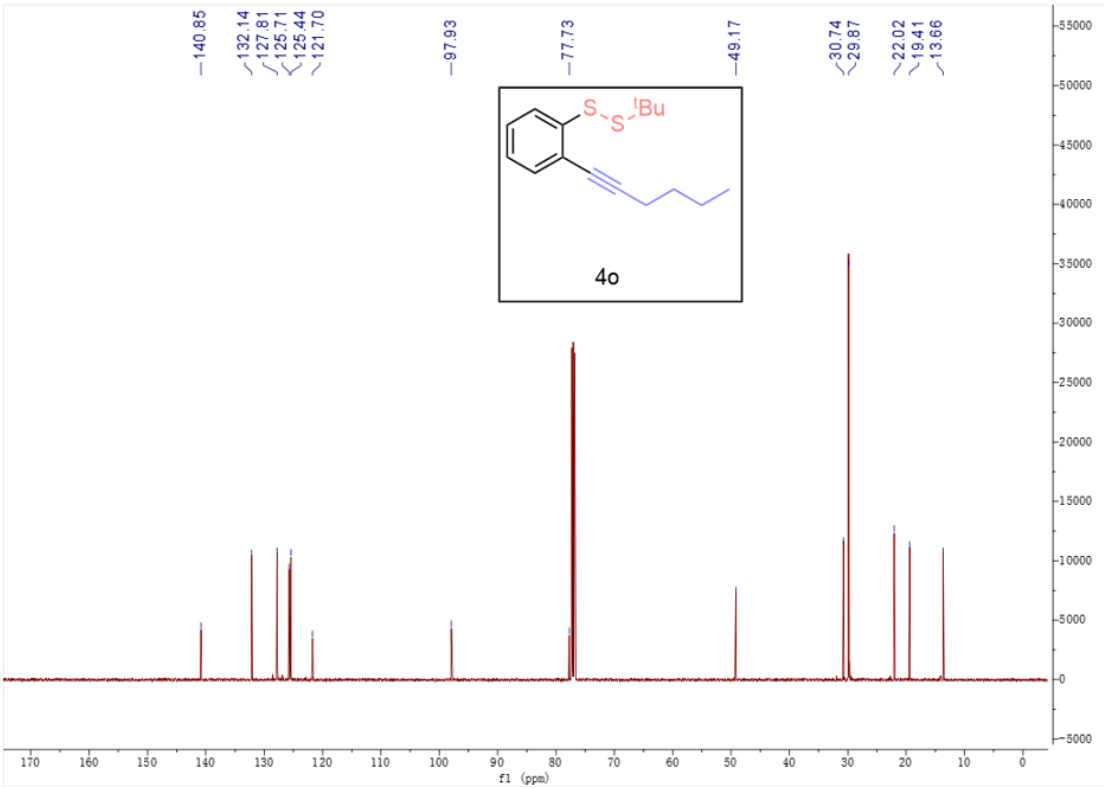

D:\DATA\240222\ZYM-4m
06/28/24 11:12:01
ZYM-4m #5 RT: 0.09 AV: 1 NL: 1.46E7
F: FTMS + p ESI Full ms [100.0000-1000.0000]
305.1392
303.1204
306.1425
307.1349
303.3087
304.1238
Relative Abundance
m/z
4m


7.31
7.30
7.29
7.29
7.25
7.25
7.24
7.23
7.22
7.22
7.08
7.08
7.06
7.06
7.05
7.05
1.56
1.55
1.54
1.54
1.53
1.53
1.52
1.52
1.51
1.32
0.95
0.94
0.93
0.93
0.92
0.92
0.91
0.91
0.90
0.90
0.89
0.89
0.88
0.87
S
S
tBu
4n
0.96
1.07
1.07
1.02
1.22
9.00
4.06
f1 (ppm)


140.45
131.65
127.26
125.27
124.98
121.12
100.51
72.30
48.67
29.39
8.49
0.00
S
S
tBu
4n
f1 (ppm)

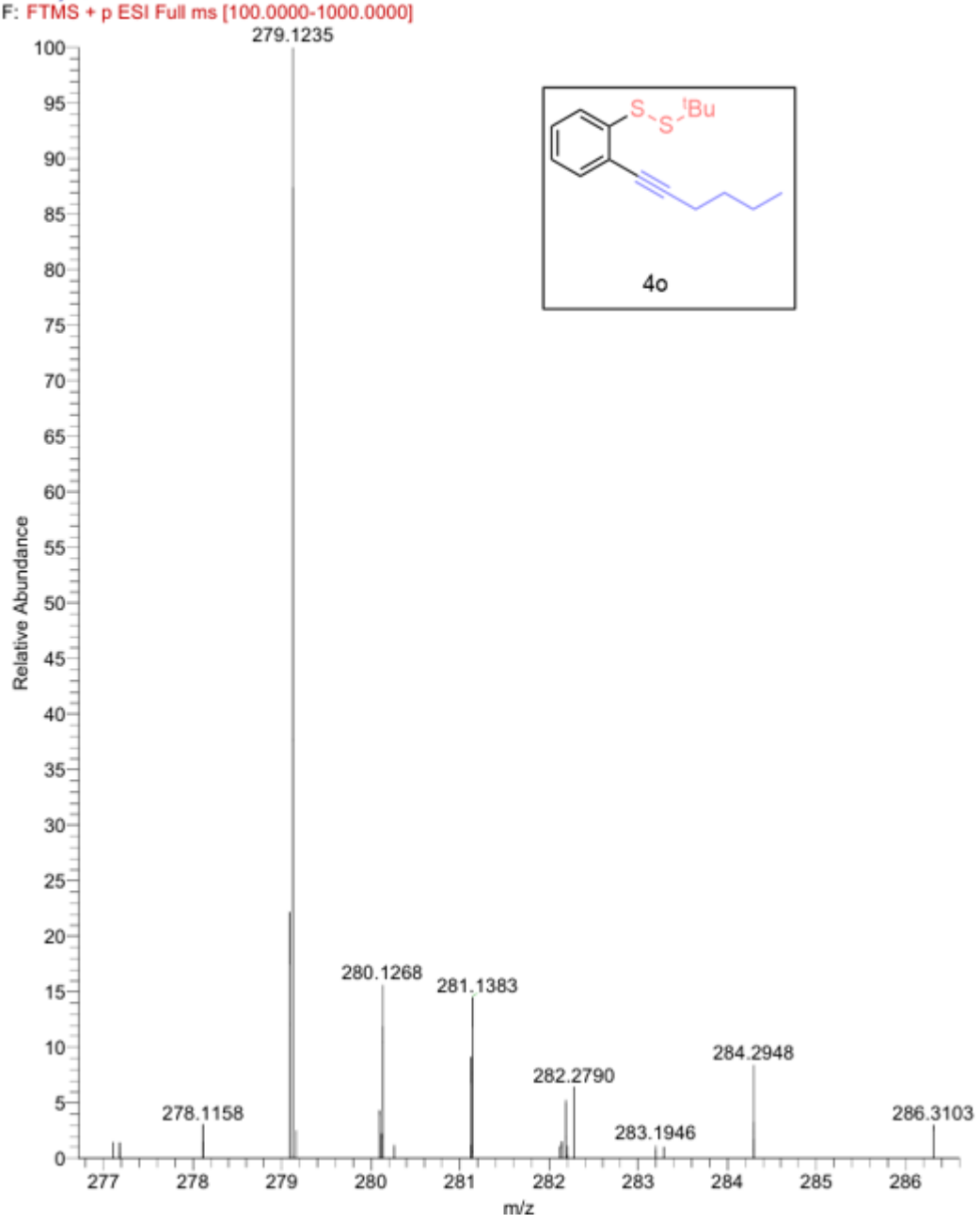

D:\DATA\240222\ZYM-4P
07/01/24 13:10:02
ZYM-4P #5-7 RT: 0.09-0.13 AV: 2 SB: 6 0.36-0.59 NL: 4.79E7
F: FTMS + p ESI Full ms [100.0000-1000.0000]
263.0922
265.0881
279.0872
274.2740
261.0734
282.1911
256.2633
272.2582
284.2949
295.0643
247.0245
Relative Abundance
m/z
S
S
tBu
4n

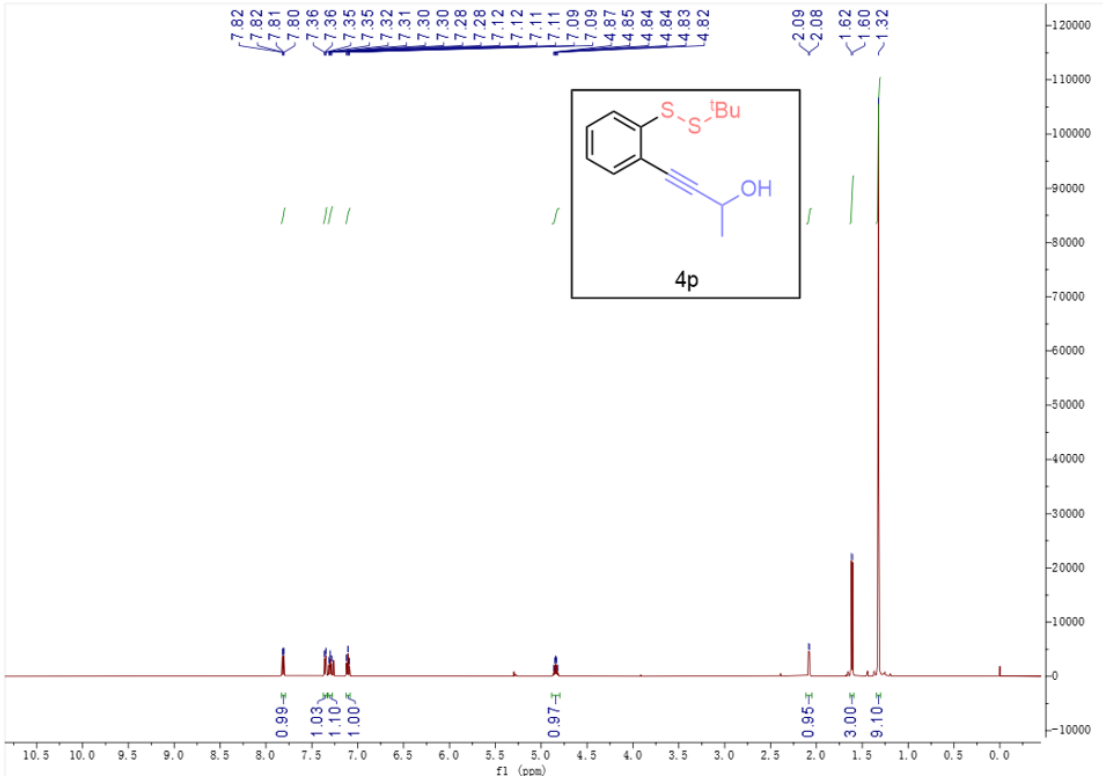

7.80
7.80
7.78
7.78
7.32
7.32
7.31
7.31
7.26
7.26
7.25
7.24
7.23
7.09
7.09
7.07
7.07
7.06
7.06
2.52
2.51
2.49
1.67
1.66
1.65
1.65
1.64
1.64
1.63
1.62
1.59
1.57
1.56
1.56
1.55
1.54
1.54
1.53
1.51
1.32
0.98
0.96
0.95
S
S
tBu
4o
0.91
0.93
1.15
0.95
1.87
1.88
2.11
9.00
2.87
f1 (ppm)

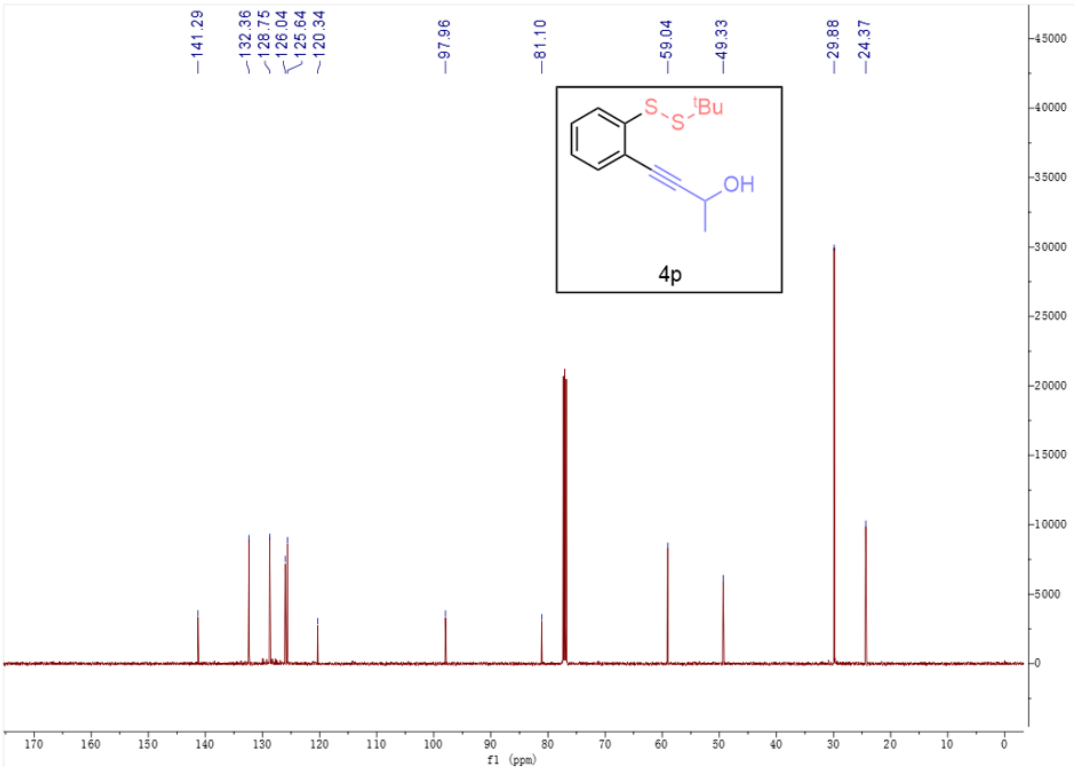

140.85
132.14
127.81
125.71
125.44
121.70
97.93
77.73
49.17
30.74
29.87
22.02
19.41
13.66
S
S
tBu
4o
f1 (ppm)

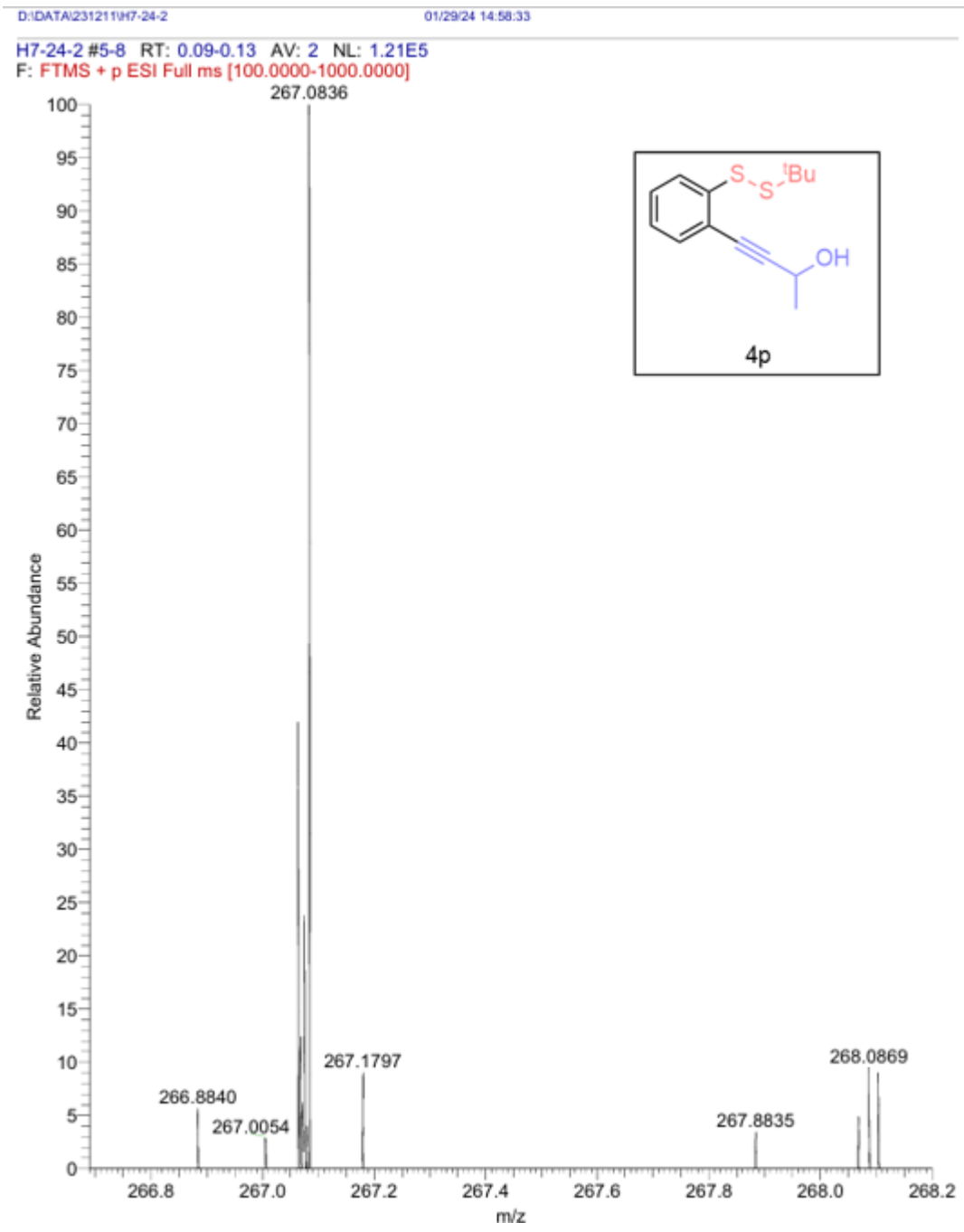
D:\DATA\240222\ZYM-4j 06/28/24 11:02:20

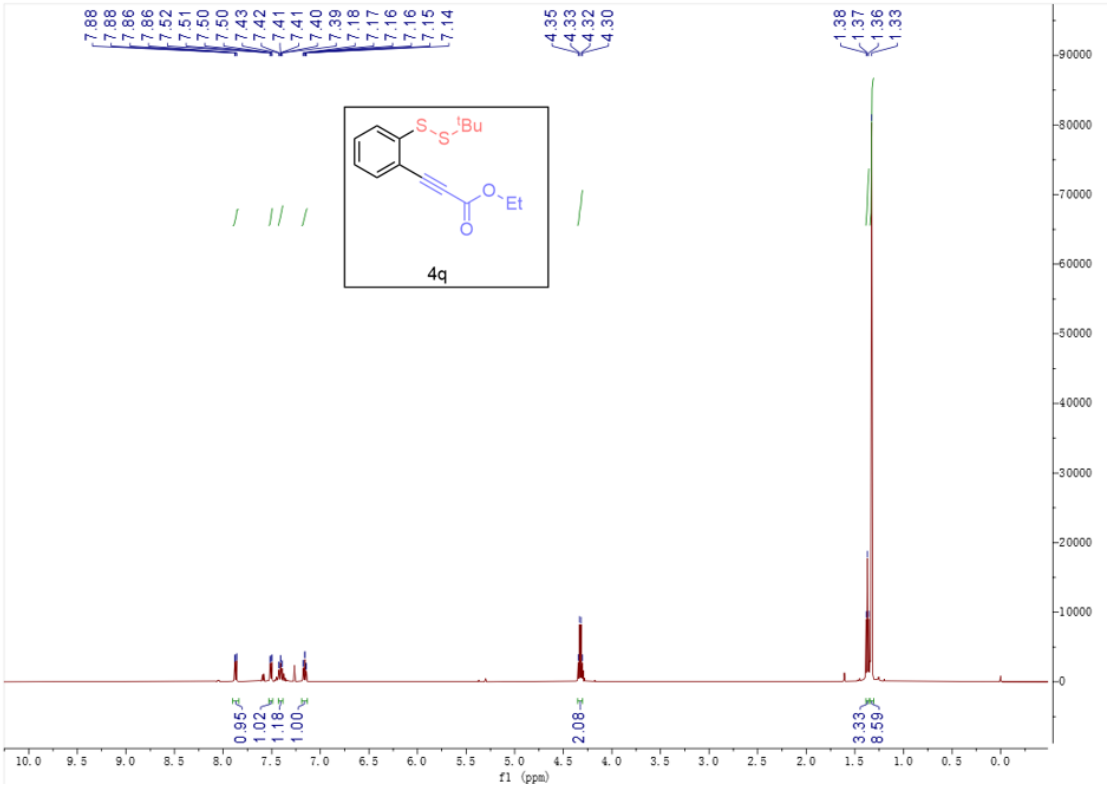
ZYM-4j #5 RT: 0.09 AV: 1 NL: 1.44E7
F: FTMS + p ESI Full ms [100.0000-1000.0000]

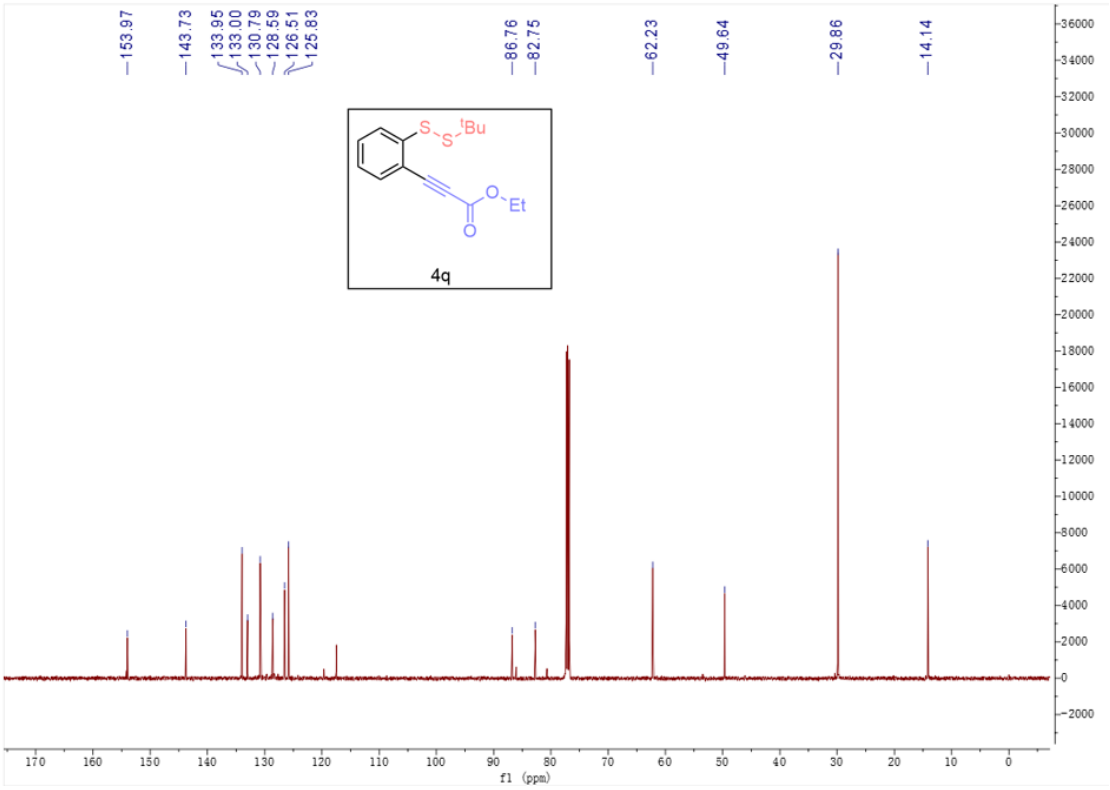


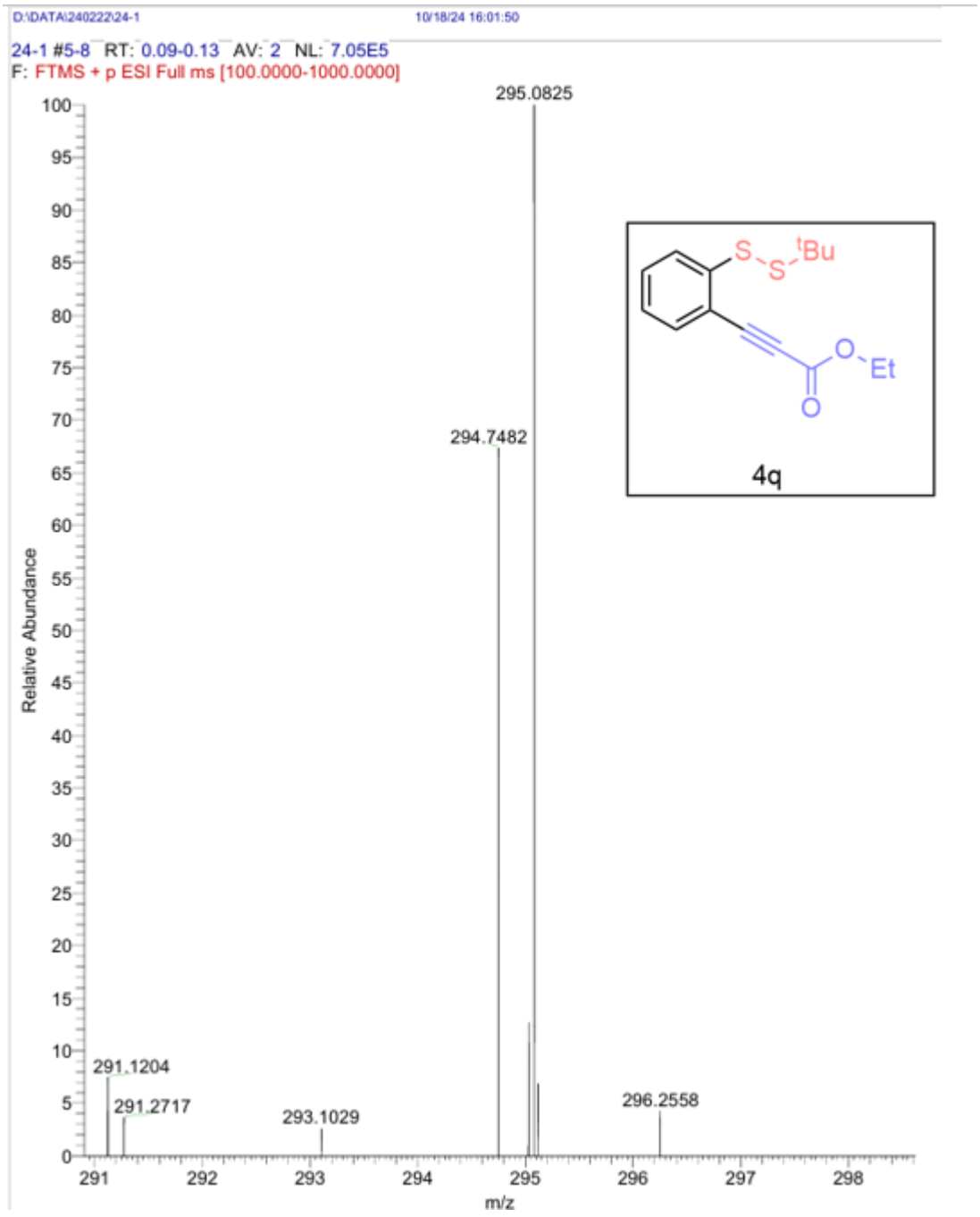

7.82
7.82
7.81
7.80
7.36
7.36
7.35
7.35
7.32
7.31
7.30
7.30
7.28
7.28
7.12
7.12
7.11
7.11
7.09
7.09
4.87
4.85
4.84
4.84
4.83
4.82
2.09
2.08
1.62
1.60
1.32
S
S
tBu
OH
4p
0.99
1.03
1.10
1.00
0.97
0.95
3.00
9.10
f1 (ppm)

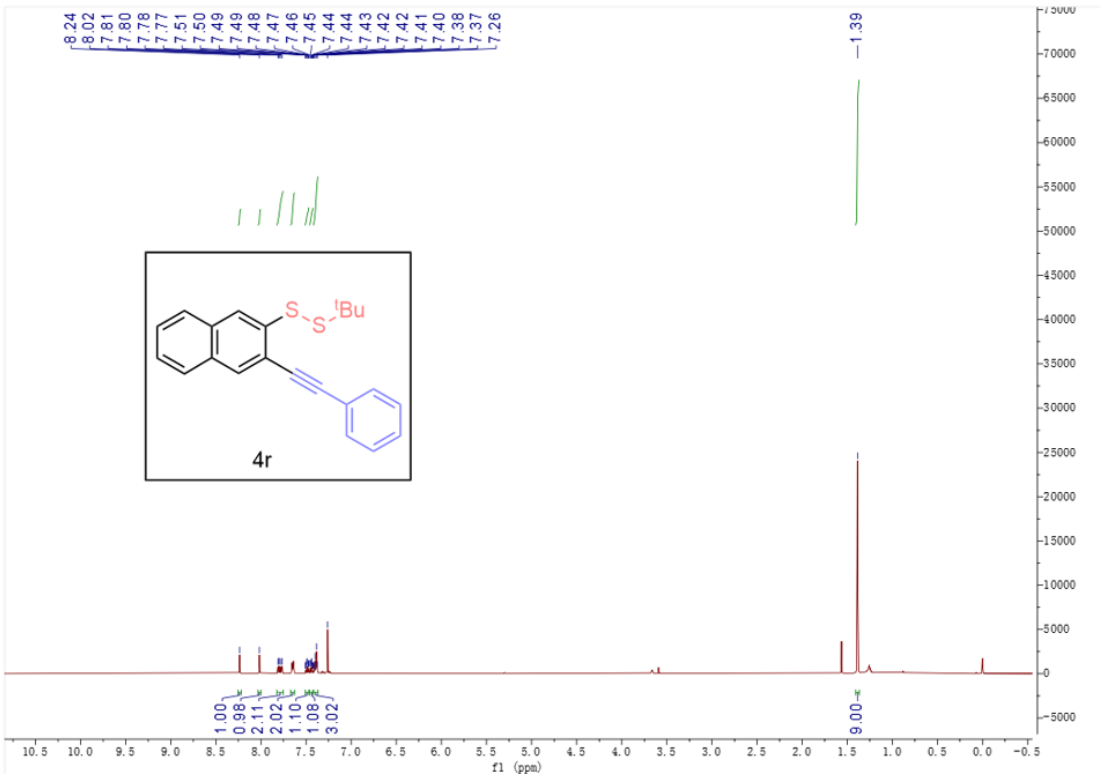

141.29
132.36
128.75
126.04
125.64
120.34
97.96
81.10
59.04
49.33
29.88
24.37
S
S
tBu
OH
4p
f1 (ppm)

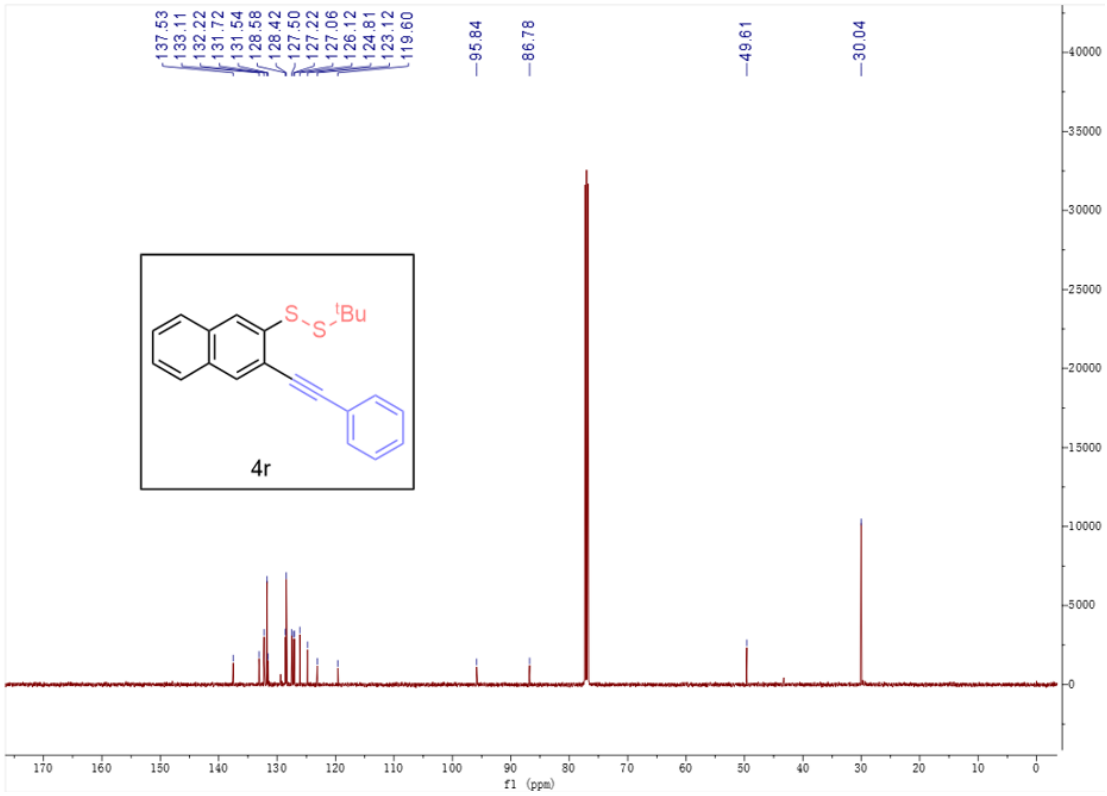

D:\DATA\231211\H7-24-2
01/29/24 14:58:33
H7-24-2 #5-8 RT: 0.09-0.13 AV: 2 NL: 1.21E5
F: FTMS + p ESI Full ms [100.0000-1000.0000]
267.0836
267.1797
268.0869
266.8840
267.0054
267.8835
S
S
tBu
OH
4p
Relative Abundance
m/z
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
266.8
267.0
267.2
267.4
267.6
267.8
268.0
268.2

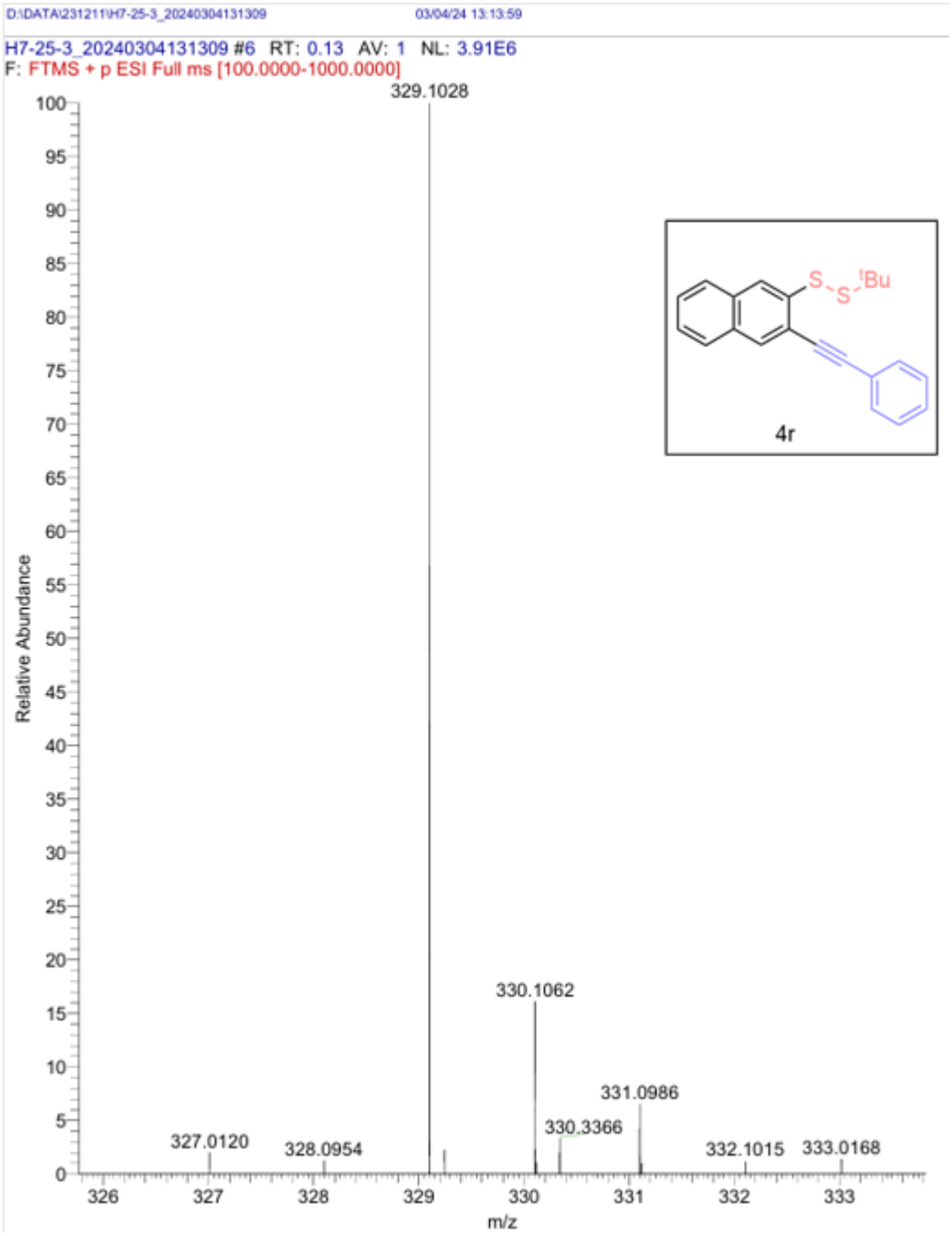

7.88
7.88
7.86
7.86
7.52
7.51
7.50
7.50
7.43
7.42
7.41
7.41
7.40
7.39
7.18
7.17
7.16
7.16
7.15
7.14
4.35
4.33
4.32
4.30
1.38
1.37
1.36
1.33
S
S
tBu
O
Et
O
4q
0.95
1.02
1.18
1.00
2.08
3.33
8.59
f1 (ppm)

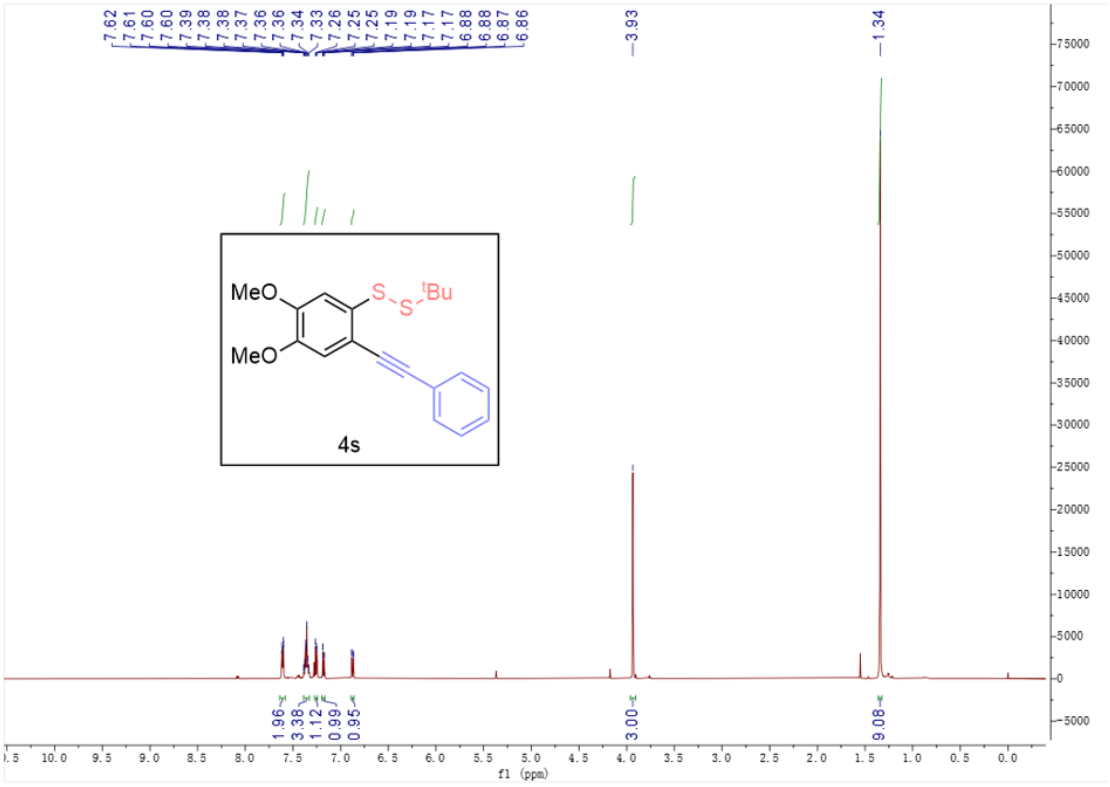

153.97
143.73
133.95
133.00
130.79
128.59
126.51
125.83
86.76
82.75
62.23
49.64
29.86
14.14
S
S
tBu
O
Et
O
4q
f1 (ppm)

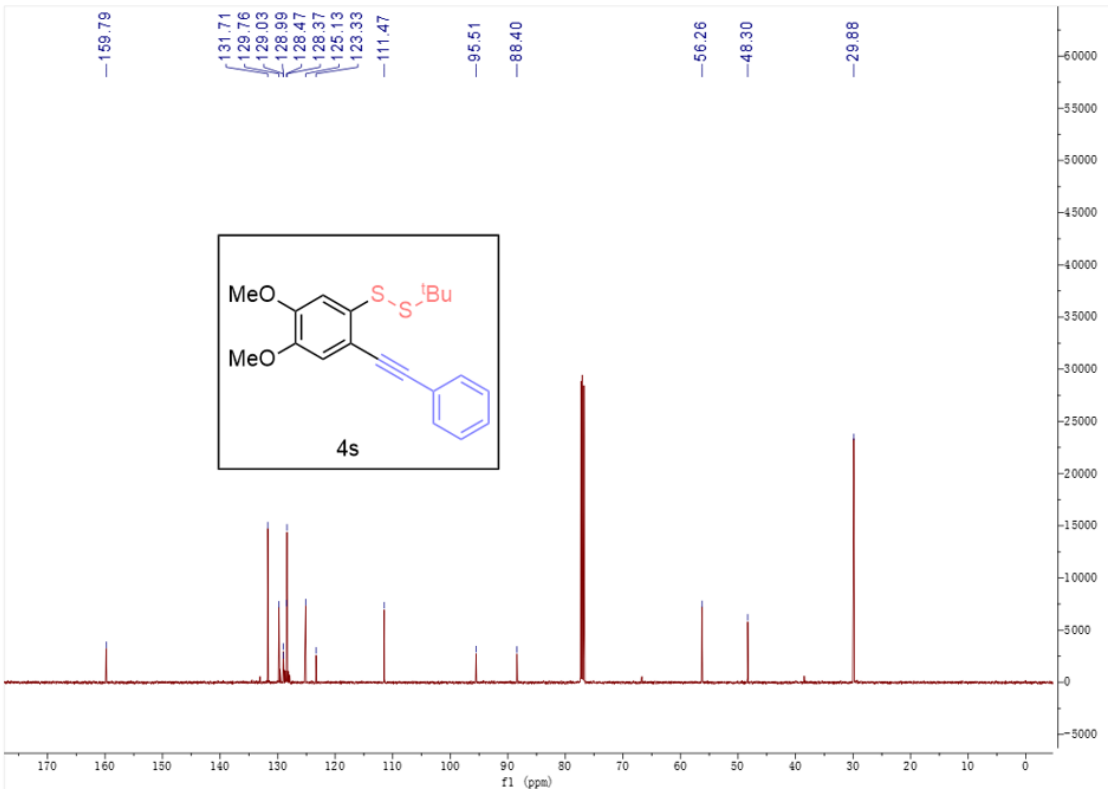

D:\DATA\240222\24-1
10/18/24 16:01:50
24-1 #5-8 RT: 0.09-0.13 AV: 2 NL: 7.05E5
F: FTMS + p ESI Full ms [100.0000-1000.0000]
295.0825
294.7482
291.1204
291.2717
293.1029
296.2558
Relative Abundance
m/z
S
S
tBu
O
Et
O
4q

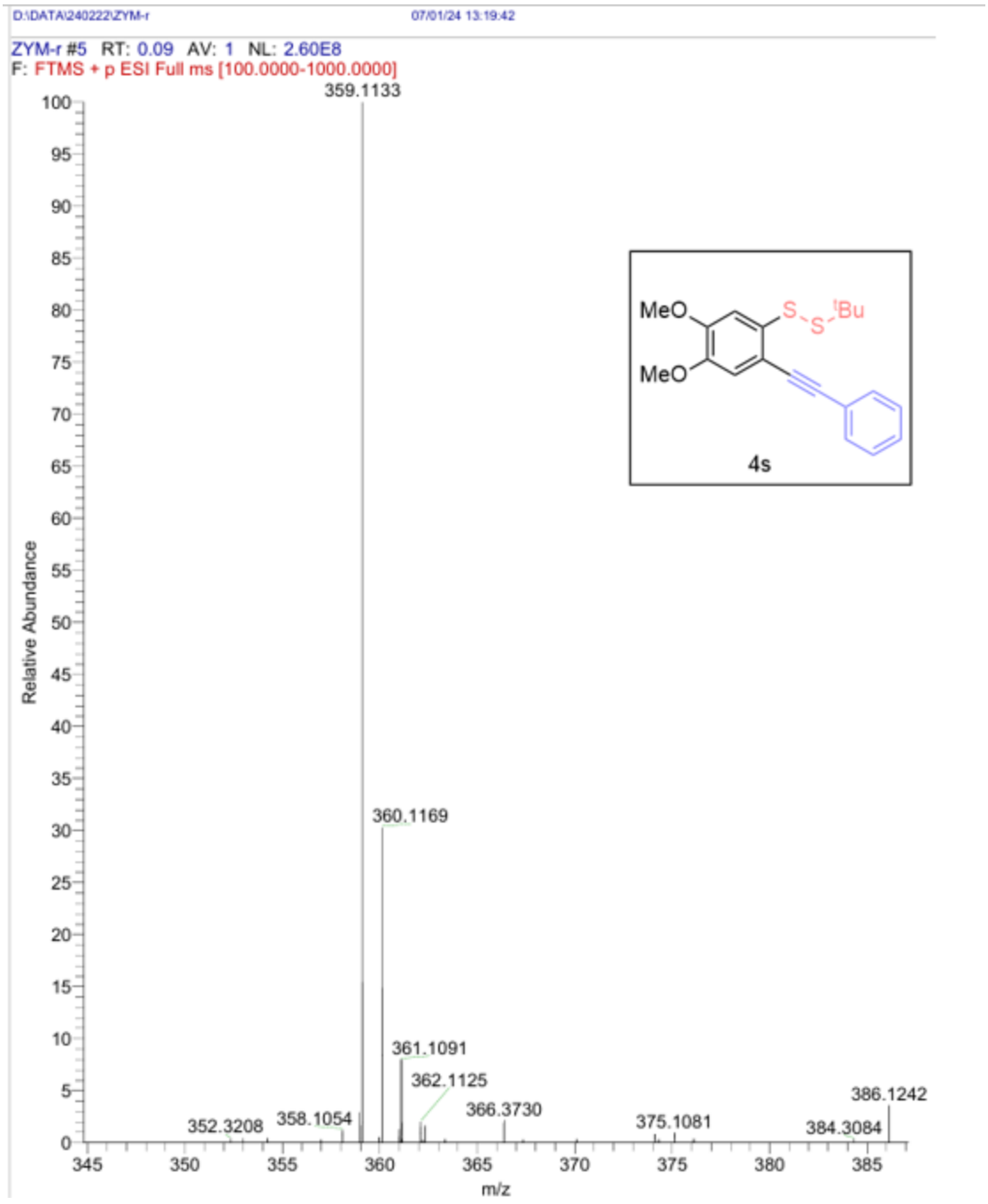

8.24
8.02
7.81
7.80
7.78
7.77
7.51
7.50
7.49
7.49
7.48
7.47
7.46
7.45
7.44
7.44
7.43
7.42
7.42
7.41
7.40
7.38
7.37
7.26
1.39
S
S
tBu
4r
1.00
0.98
2.11
2.02
1.10
1.08
3.02
9.00
f1 (ppm)

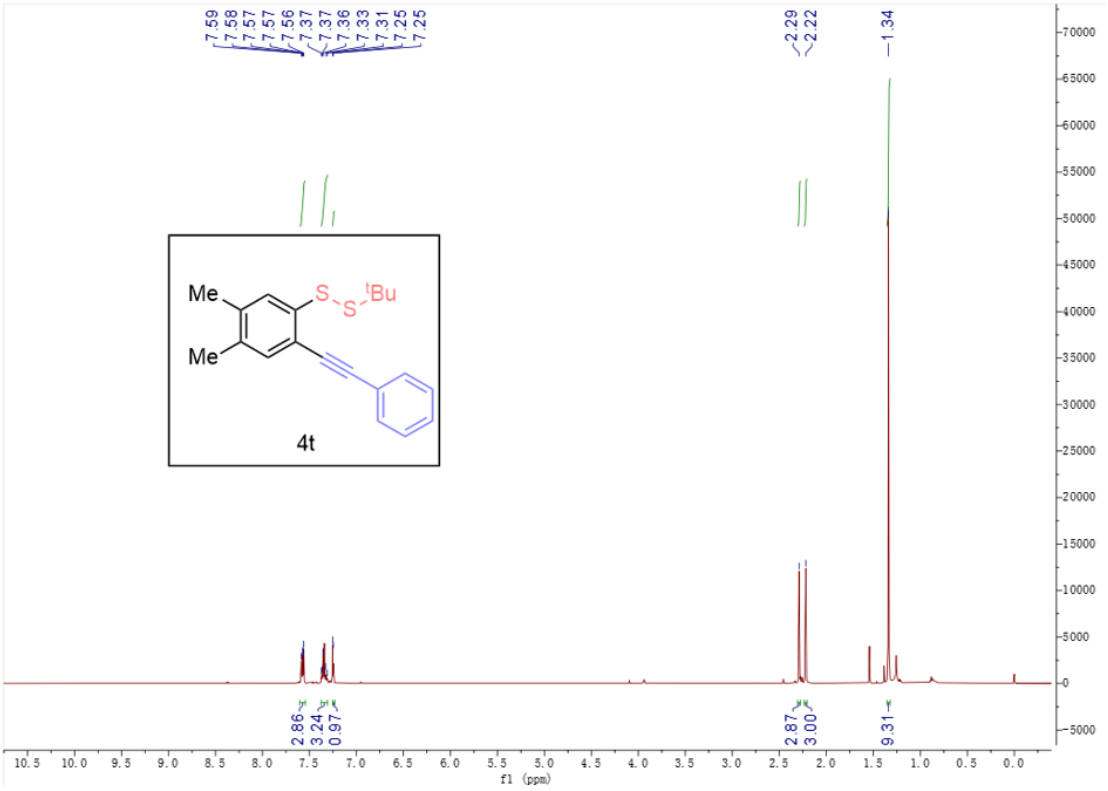

137.53
133.11
132.22
131.72
131.54
128.58
128.42
127.50
127.22
127.06
126.12
124.81
123.12
119.60
95.84
86.78
49.61
30.04
S
S
tBu
4r
f1 (ppm)

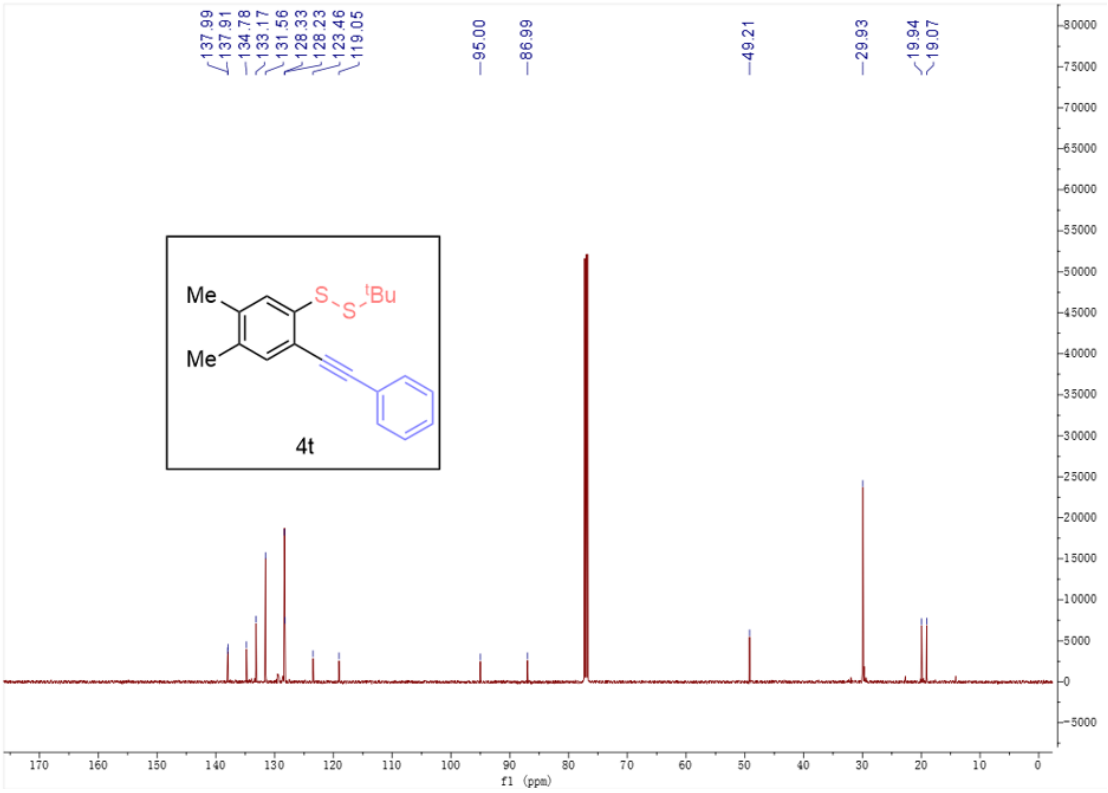

D:\DATA\231211\H7-25-3_20240304131309 03/04/24 13:13:59
H7-25-3_20240304131309 #6 RT: 0.13 AV: 1 NL: 3.91E6
F: FTMS + p ESI Full ms [100.0000-1000.0000]
329.1028
330.1062
331.0986
330.3366
327.0120
328.0954
332.1015
333.0168
Relative Abundance
m/z
4r

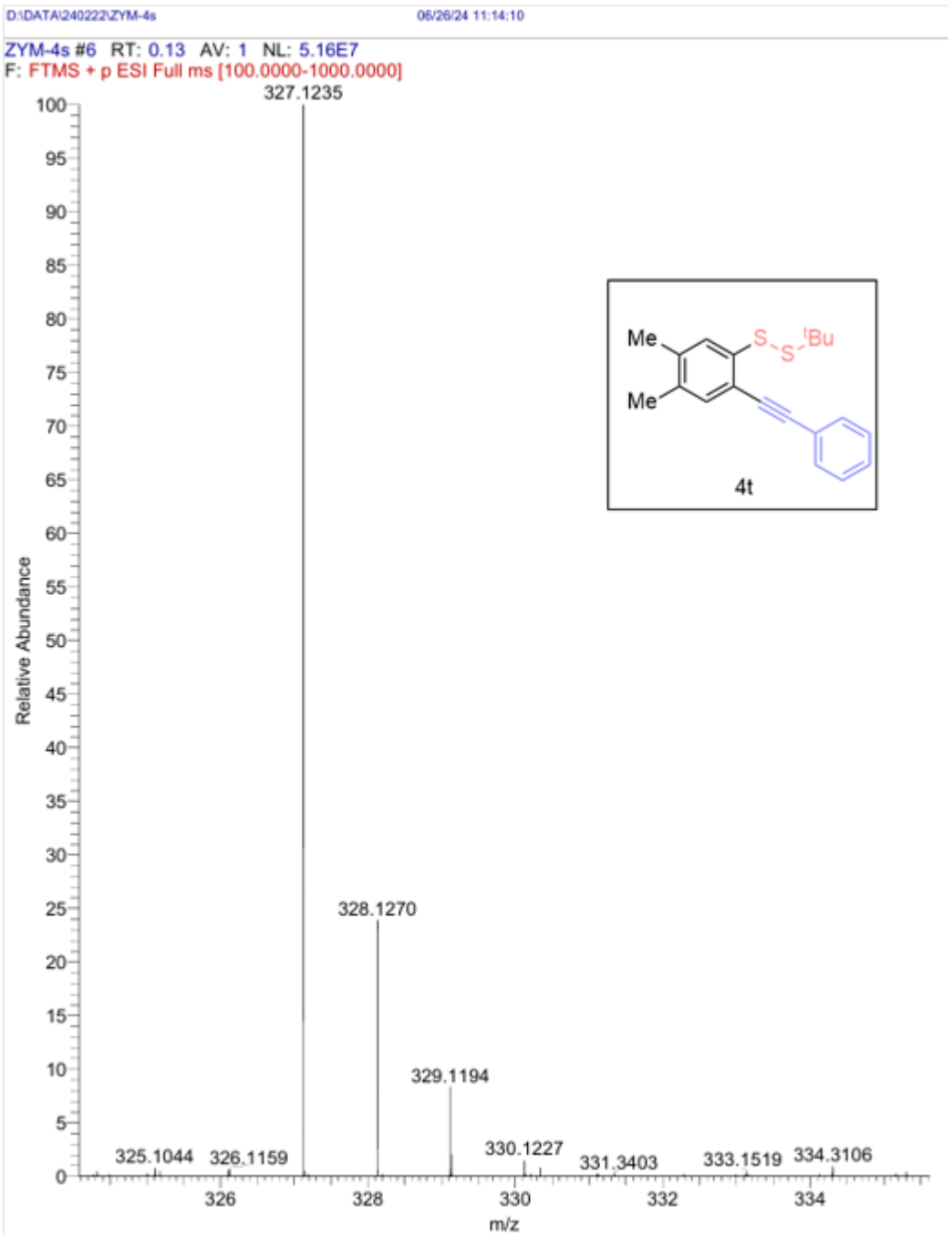

7.62
7.61
7.60
7.60
7.39
7.38
7.38
7.37
7.36
7.36
7.34
7.33
7.26
7.25
7.25
7.19
7.17
7.17
6.88
6.88
6.87
6.86
3.93
1.34
MeO
MeO
S
S
tBu
4s
1.96
3.38
1.12
0.99
0.95
3.00
9.08
f1 (ppm)

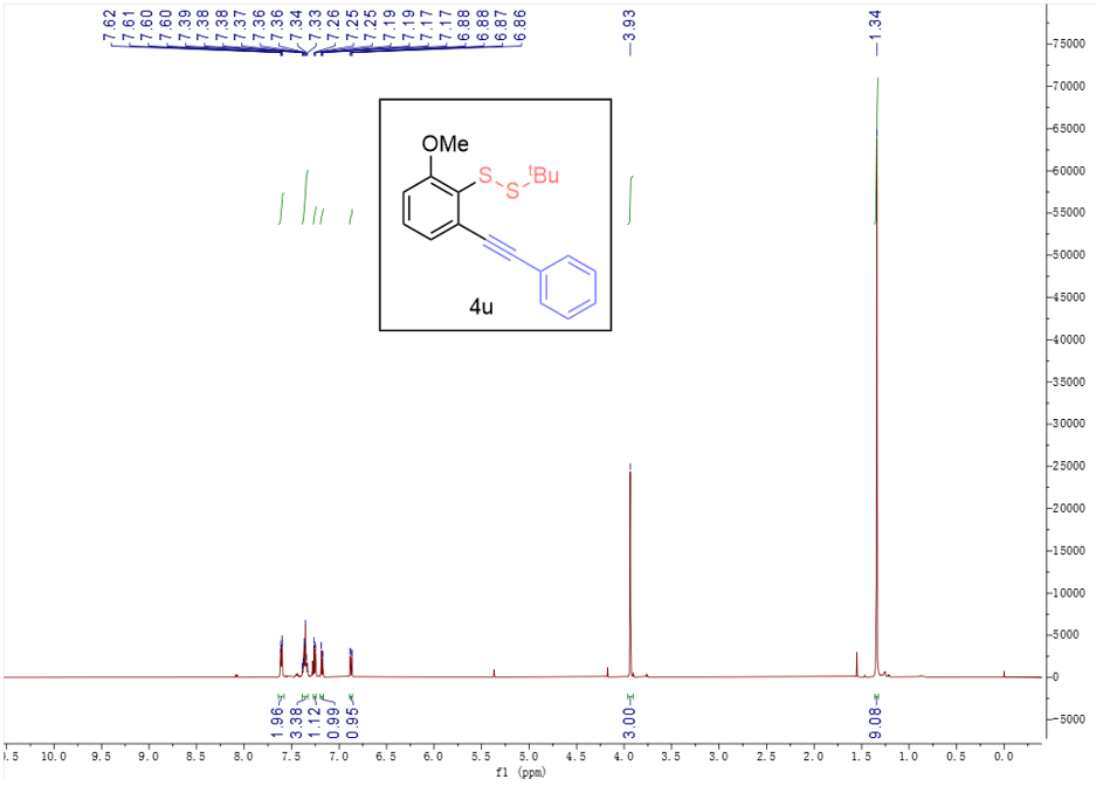

159.79
131.71
129.76
129.03
128.99
128.47
128.37
125.13
123.33
111.47
95.51
88.40
56.26
48.30
29.88
MeO
MeO
S
S
tBu
4s
f1 (ppm)

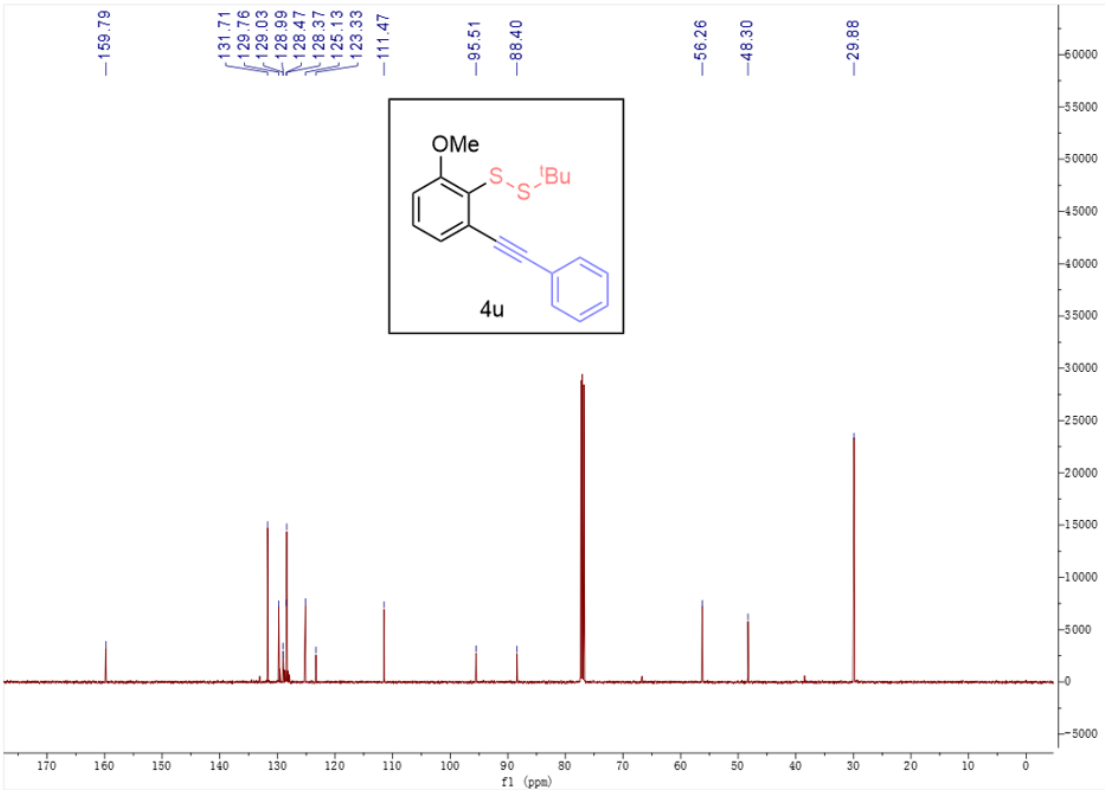

D:\DATA\240222\ZYM-r
07/01/24 13:19:42
ZYM-r #5 RT: 0.09 AV: 1 NL: 2.60E8
F: FTMS + p ESI Full ms [100.0000-1000.0000]
359.1133
360.1169
361.1091
362.1125
366.3730
352.3208
358.1054
375.1081
384.3084
386.1242
MeO
MeO
S
S
tBu
4s
Relative Abundance
m/z


7.59
7.58
7.57
7.57
7.56
7.37
7.37
7.36
7.33
7.31
7.25
7.25
2.29
2.22
1.34
Me
Me
S
S
tBu
4t
2.86
3.24
0.97
2.87
3.00
9.31
f1 (ppm)


137.99
137.91
134.78
133.17
131.56
128.33
128.23
123.46
119.05
95.00
86.99
49.21
29.93
19.94
19.07
Me
Me
S
S
tBu
4t
f1 (ppm)

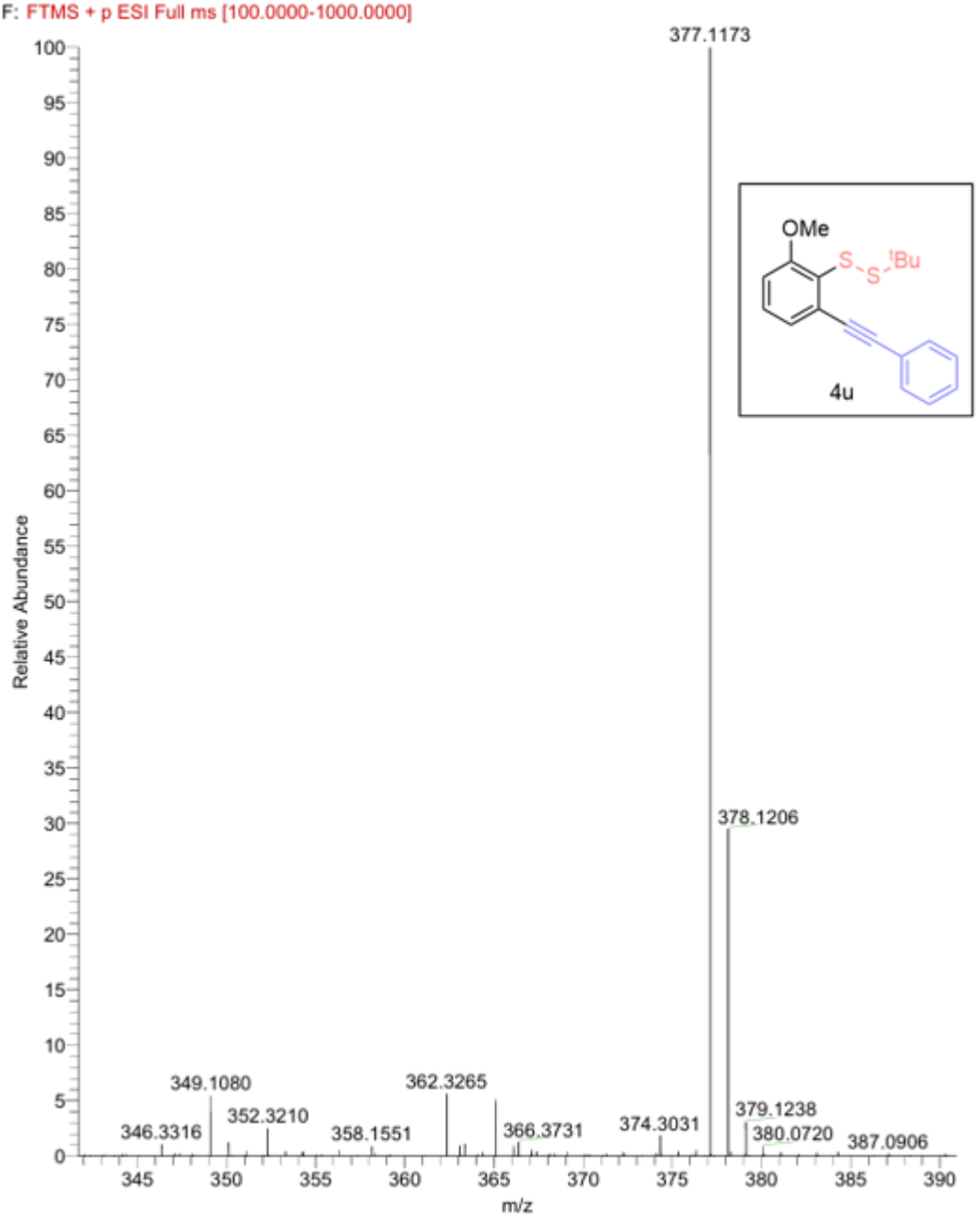

D:\DATA\240222\ZYM-4s
06/26/24 11:14:10
ZYM-4s #6 RT: 0.13 AV: 1 NL: 5.16E7
F: FTMS + p ESI Full ms [100.0000-1000.0000]
327.1235
328.1270
329.1194
325.1044
326.1159
330.1227
331.3403
333.1519
334.3106
Relative Abundance
m/z
Me
Me
S
S
tBu
4t

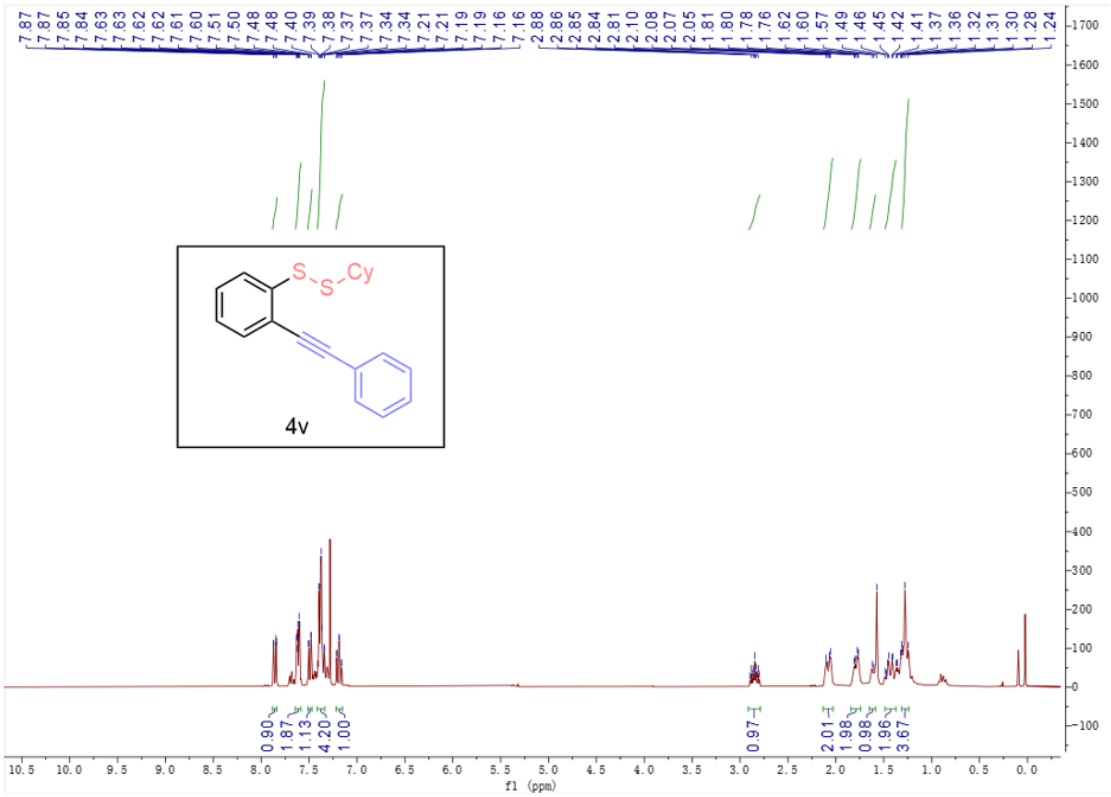

7.62
7.61
7.60
7.60
7.39
7.38
7.38
7.37
7.36
7.36
7.34
7.33
7.26
7.25
7.25
7.19
7.19
7.17
7.17
6.88
6.87
6.86
3.93
1.34
OMe
S
S
tBu
4u
1.96
3.38
1.12
0.99
0.95
3.00
9.08
f1 (ppm)

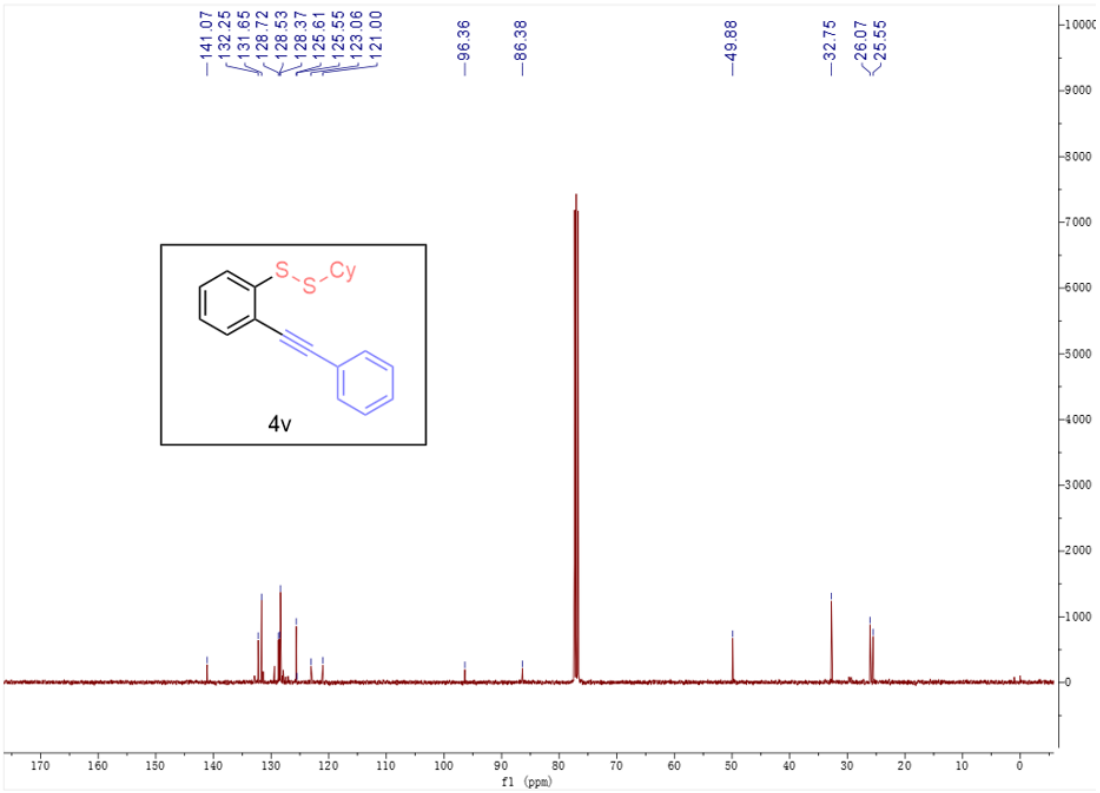

159.79
131.71
129.76
129.03
128.99
128.47
128.37
125.13
123.33
111.47
95.51
88.40
56.26
48.30
29.88
OMe
S
S
tBu
4u
f1 (ppm)

D:\DATA\240222\ZYM-4u 06/26/24 11:19:00

ZYM-4u #6 RT: 0.13 AV: 1 NL: 4.99E7
F: FTMS + p ESI Full ms [100.0000-1000.0000]

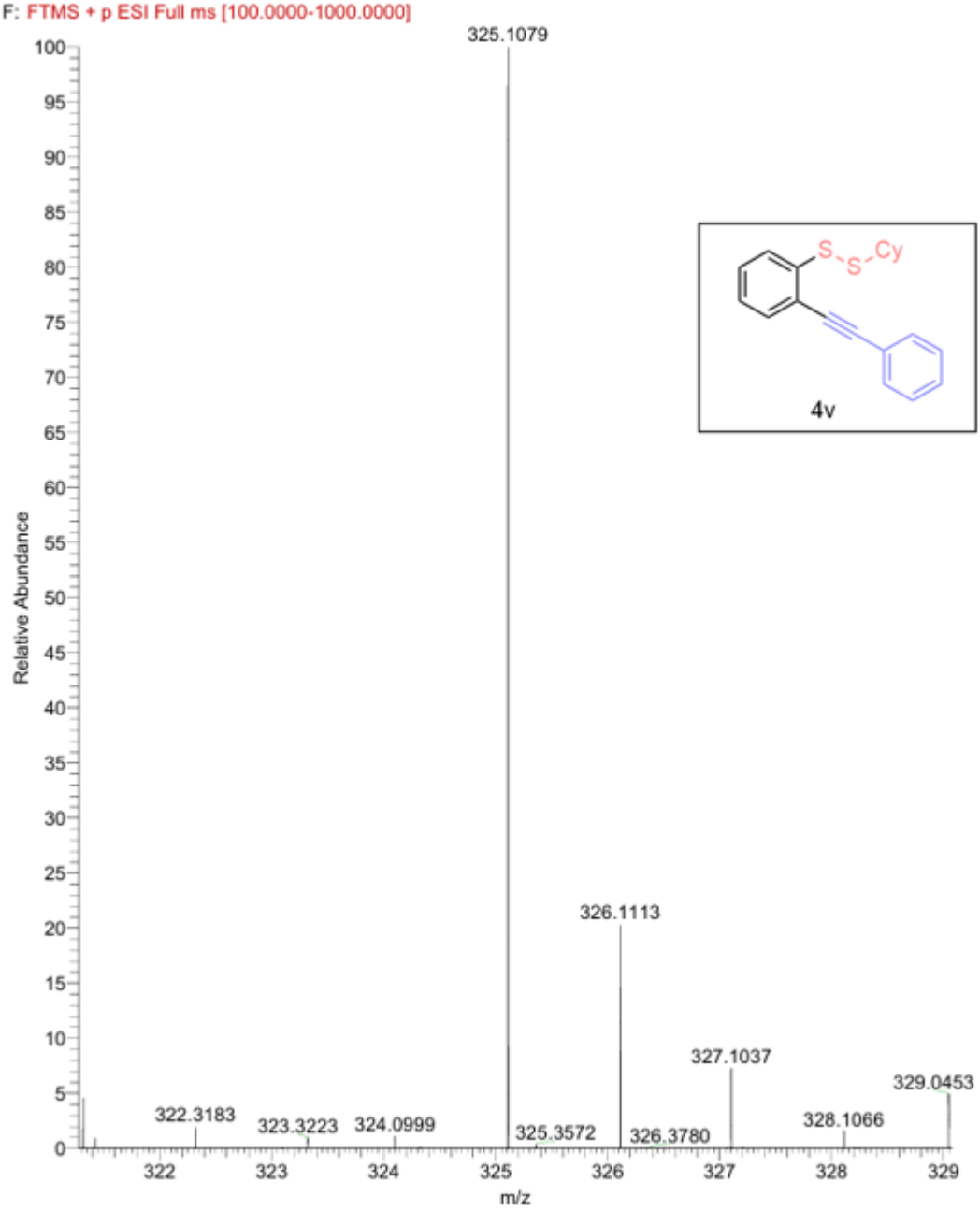


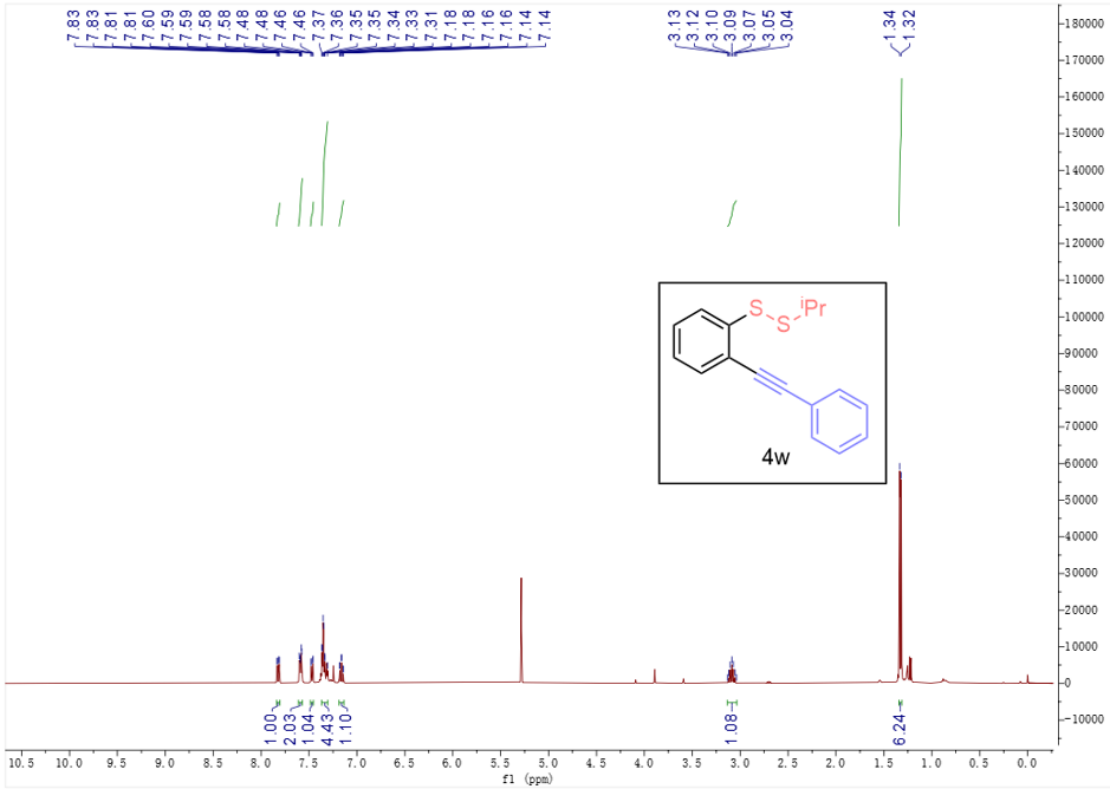

7.87 7.87 7.85 7.84 7.63 7.63 7.62 7.62 7.61 7.60 7.51 7.50 7.48 7.48 7.40 7.39 7.38 7.37 7.37 7.34 7.34 7.21 7.21 7.19 7.19 7.16 7.16
2.88 2.86 2.85 2.84 2.81 2.10 2.08 2.07 2.05 1.81 1.80 1.78 1.76 1.62 1.60 1.57 1.49 1.46 1.45 1.42 1.41 1.37 1.36 1.32 1.31 1.30 1.28 1.24
S S Cy
4v
0.90 1.87 1.13 4.20 1.00 0.97 2.01 1.98 0.98 1.96 3.67
f1 (ppm)

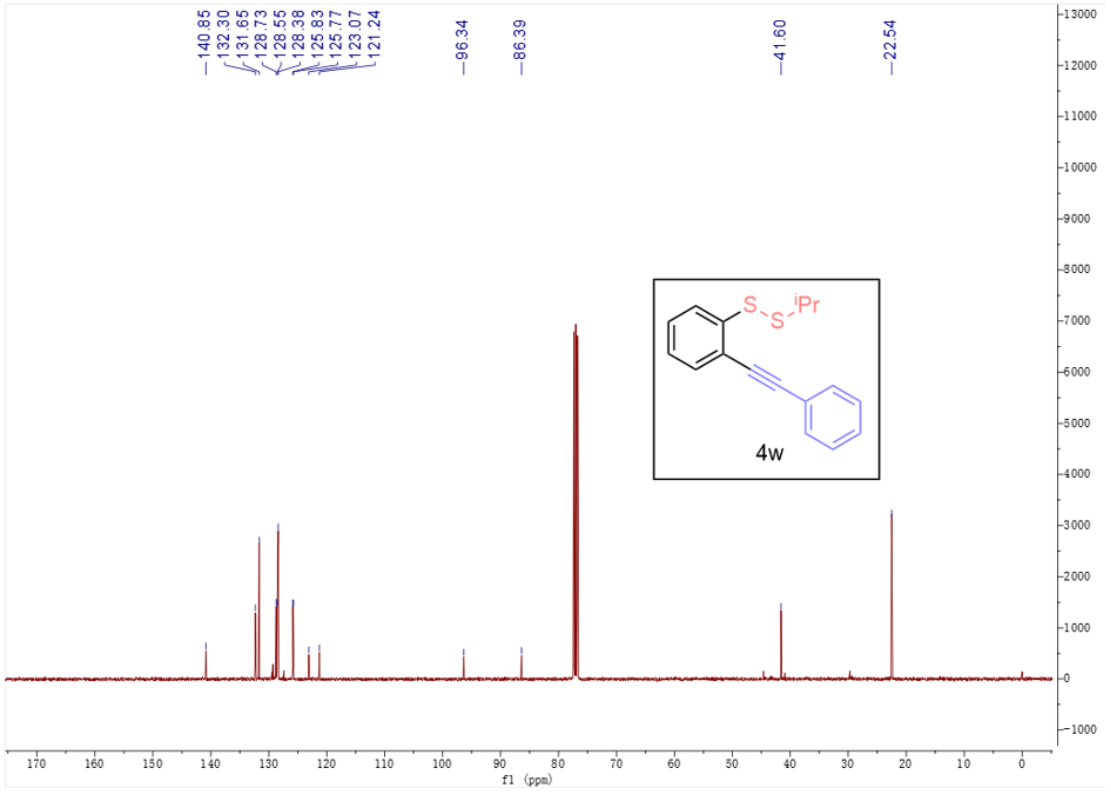

141.07 132.25 131.65 128.72 128.53 128.37 125.61 125.55 123.06 121.00 96.36 86.38 49.88 32.75 26.07 25.55
S S Cy
4v
f1 (ppm)

D:\DATA\240222\ZYM-10-6 05/10/24 16:19:24

ZYM-10-6 #5-8 RT: 0.08-0.12 AV: 2 NL: 3.57E6
F: FTMS + p ESI Full ms [100.0000-1000.0000]



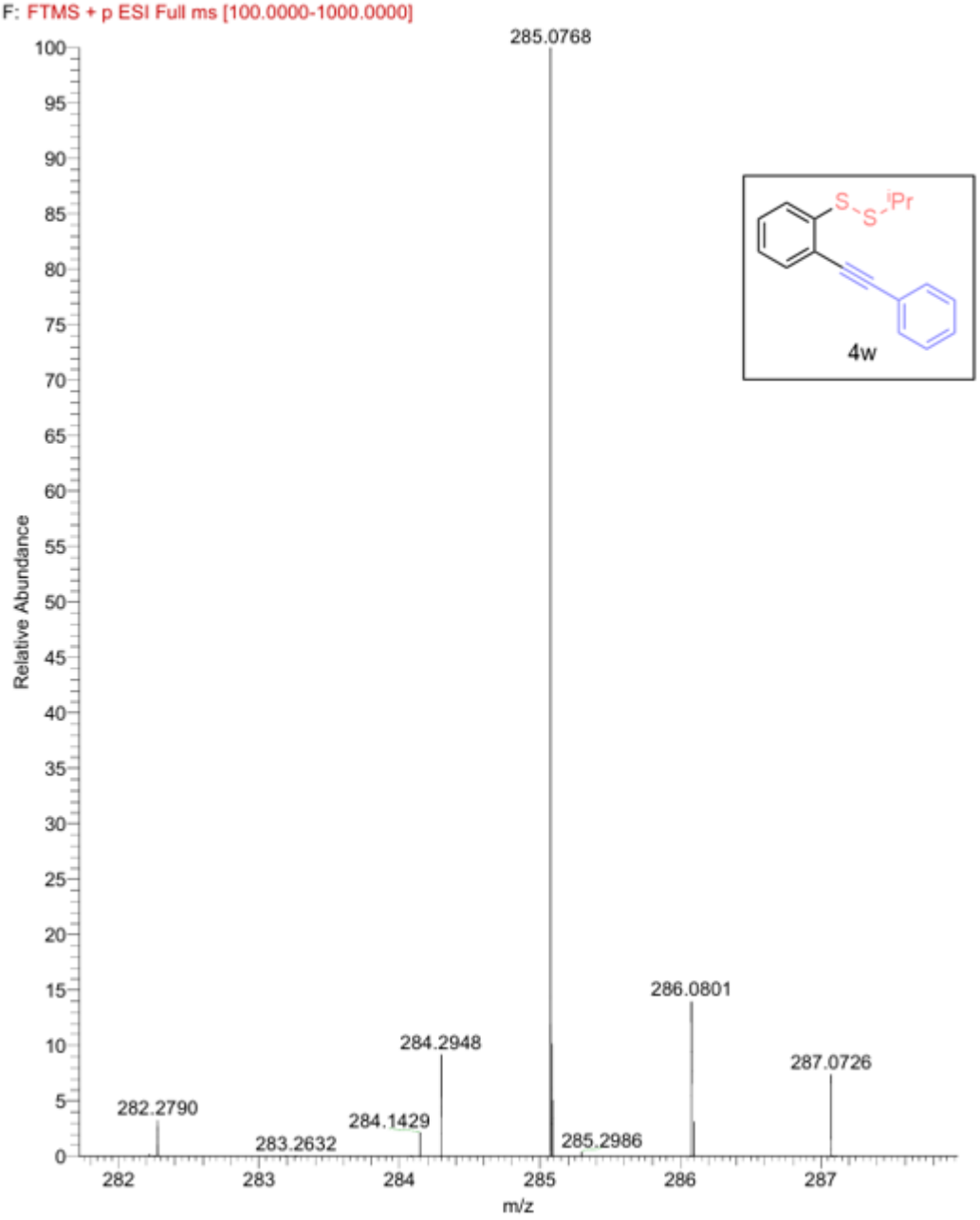

7.83
7.83
7.81
7.81
7.60
7.59
7.59
7.58
7.58
7.48
7.48
7.46
7.46
7.37
7.36
7.35
7.35
7.34
7.33
7.31
7.18
7.18
7.16
7.16
7.14
7.14
3.13
3.12
3.10
3.09
3.07
3.05
3.04
1.34
1.32
S
S
iPr
4w
1.00
2.03
1.04
4.43
1.10
1.08
6.24
f1 (ppm)

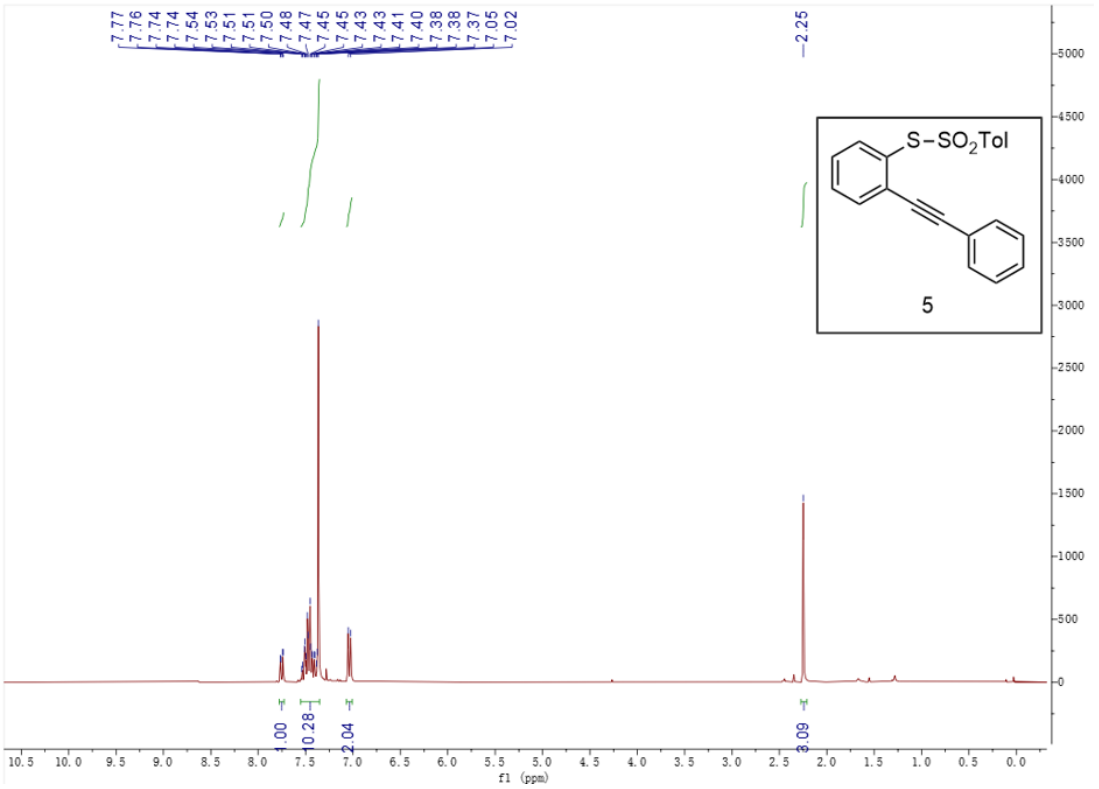

140.85
132.30
131.65
128.73
128.55
128.38
125.83
125.77
123.07
121.24
96.34
86.39
41.60
22.54
S
S
iPr
4w
f1 (ppm)

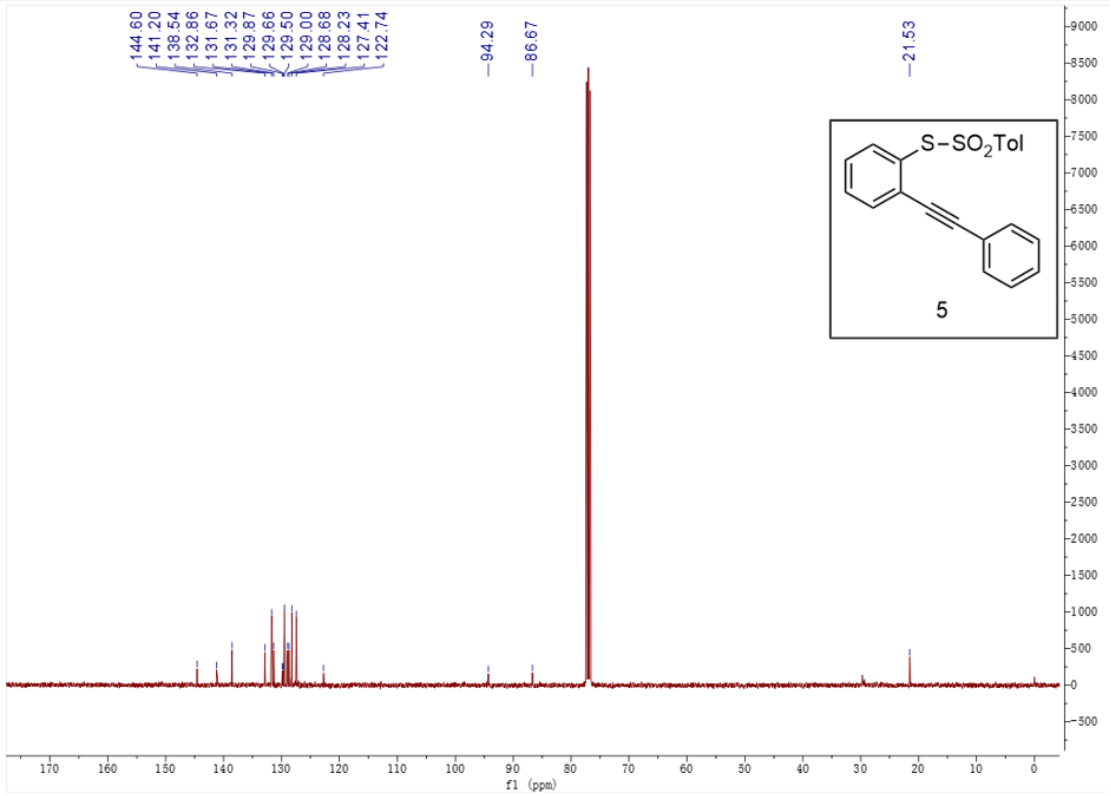
D:\DATA\240222\H7-29-2 05/10/24 16:16:59

H7-29-2 #4-7 RT: 0.08-0.12 AV: 2 NL: 4.58E6

F: FTMS + p ESI Full ms [100.0000-1000.0000]

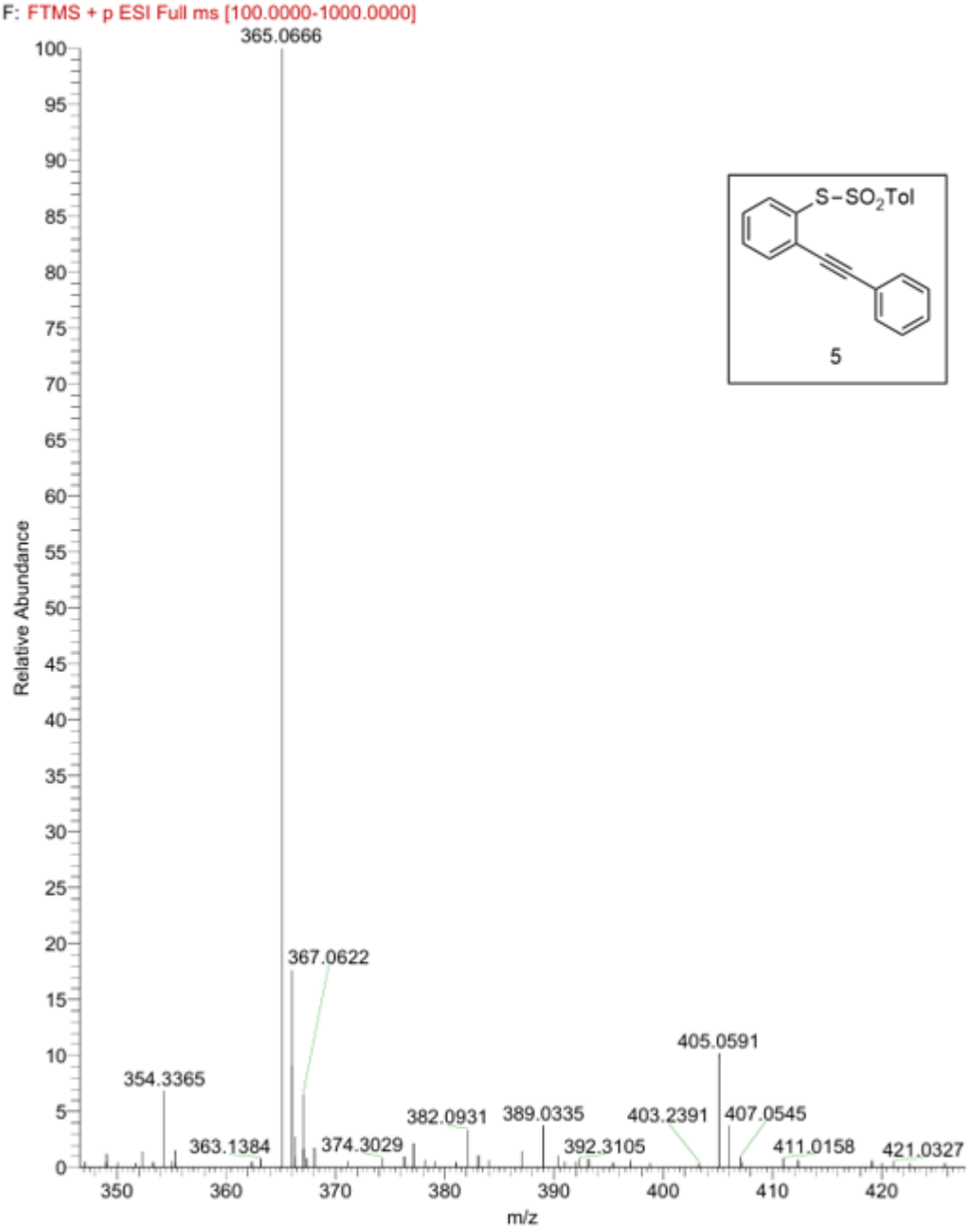


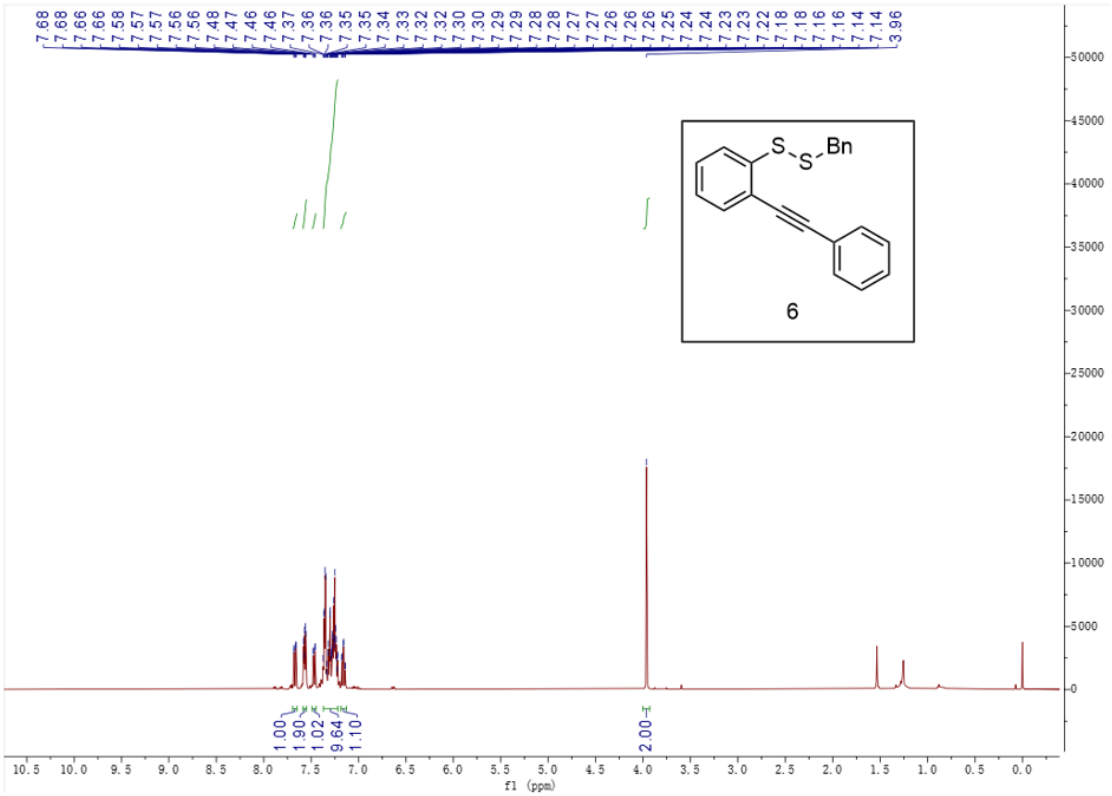

7.77
7.76
7.74
7.74
7.54
7.53
7.51
7.51
7.50
7.48
7.47
7.45
7.45
7.43
7.43
7.41
7.40
7.38
7.38
7.37
7.05
7.02
2.25
S−SO2Tol
5
1.00
10.28
2.04
3.09
f1 (ppm)

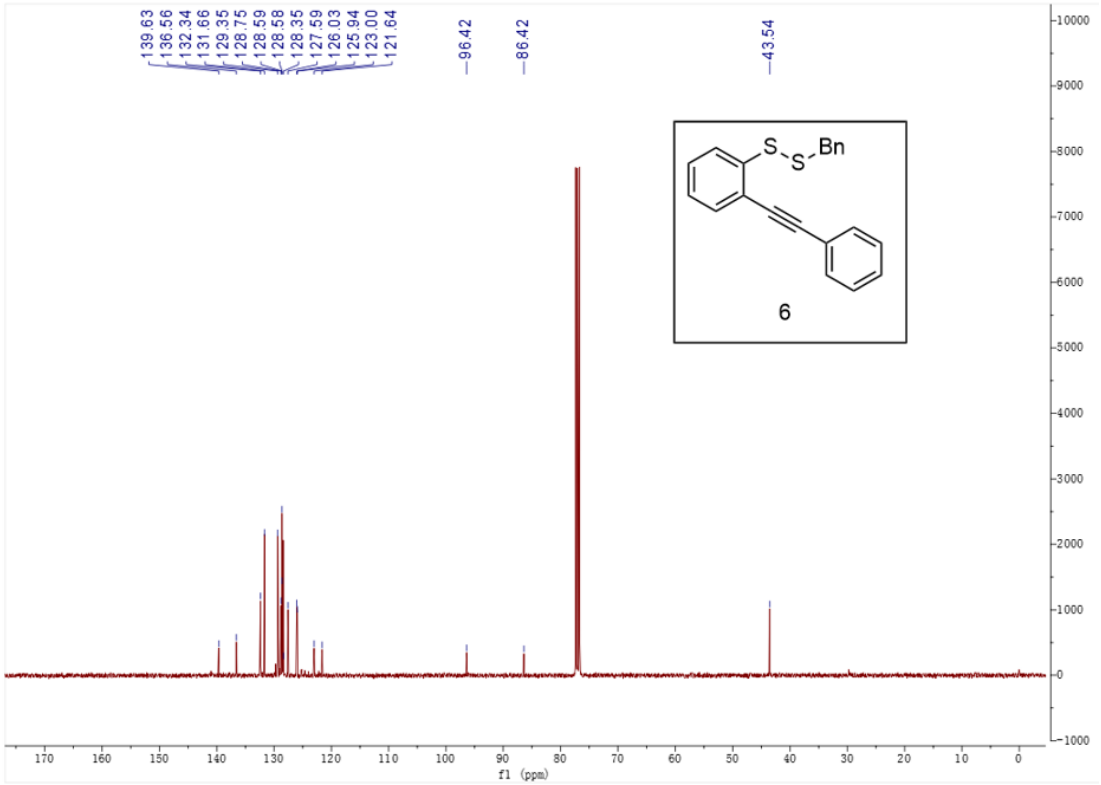

144.60
141.20
138.54
132.86
131.67
131.32
129.87
129.66
129.50
129.00
128.68
128.23
127.41
122.74
94.29
86.67
21.53
S−SO2Tol
5
f1 (ppm)

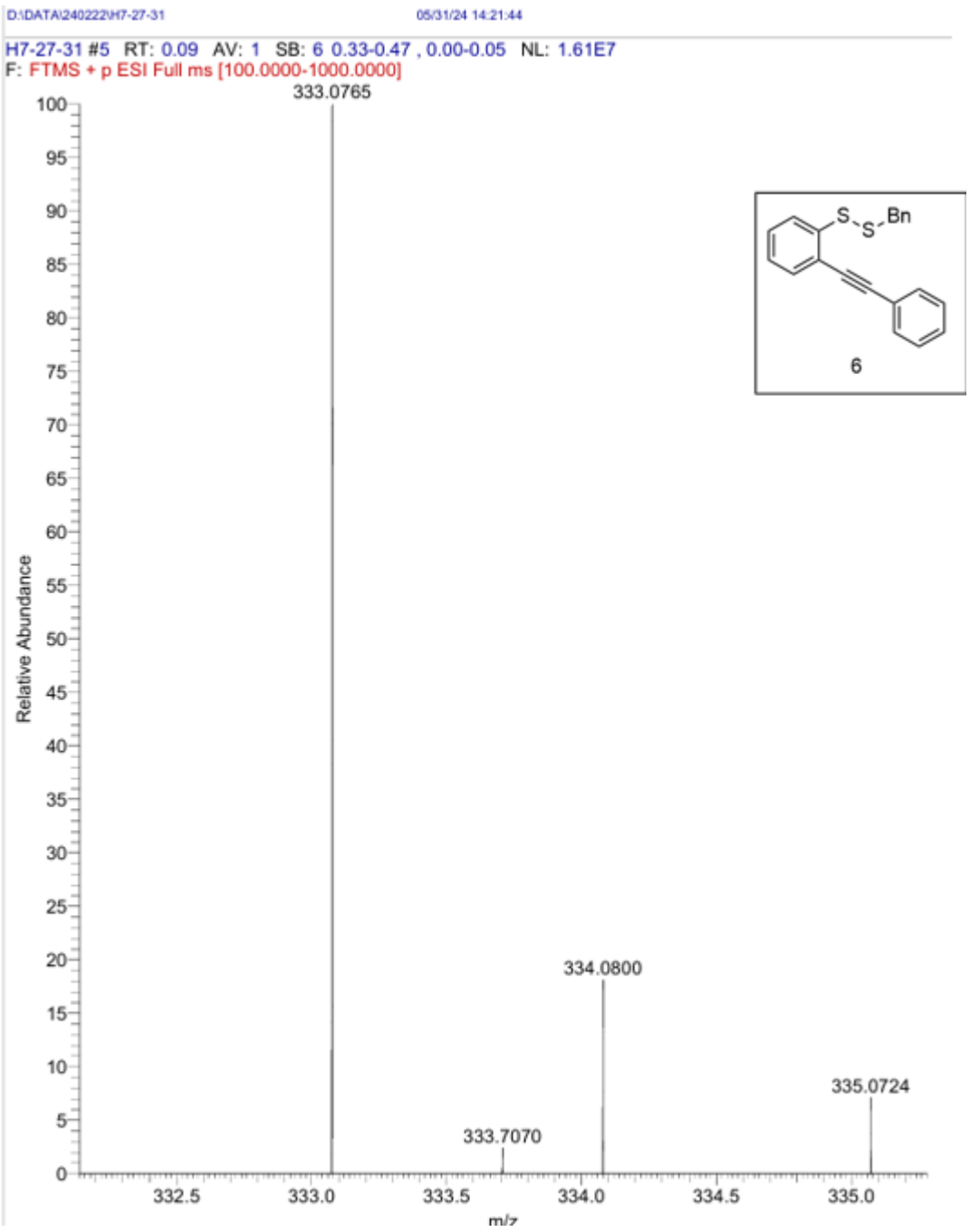
D:\DATA\240222\ZYM-21 05/31/24 14:07:25

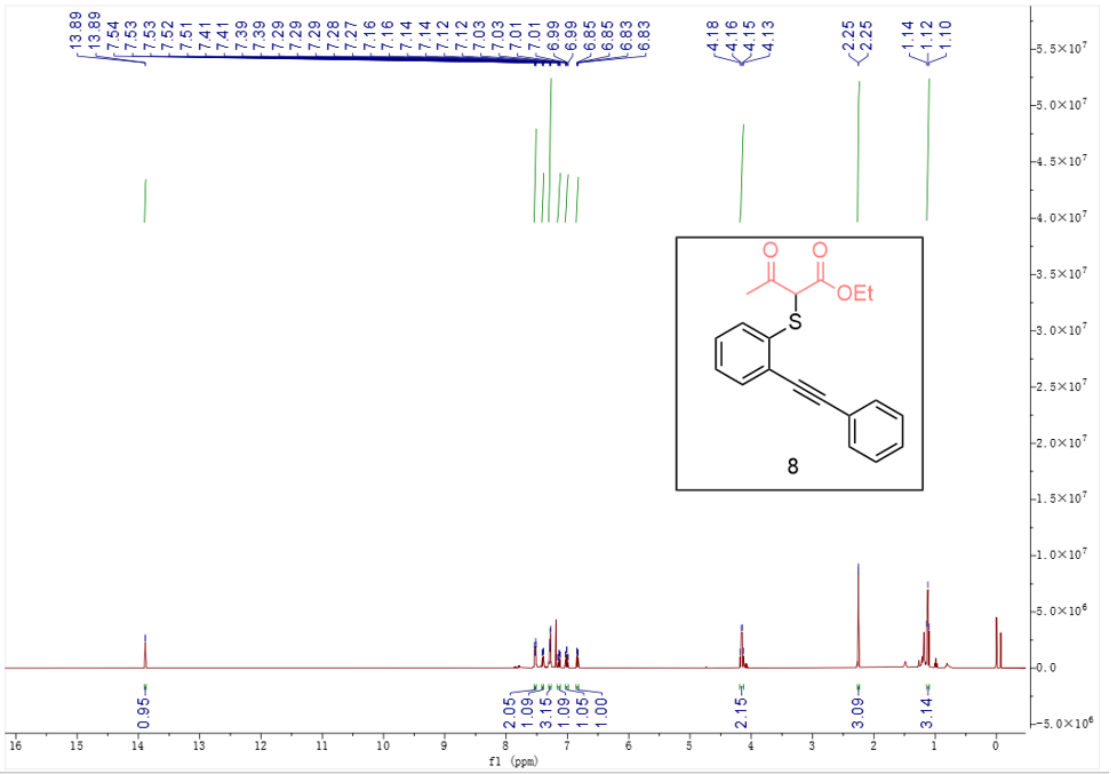
ZYM-21 #5 RT: 0.09 AV: 1 NL: 3.93E8
F: FTMS + p ESI Full ms [100.0000-1000.0000]

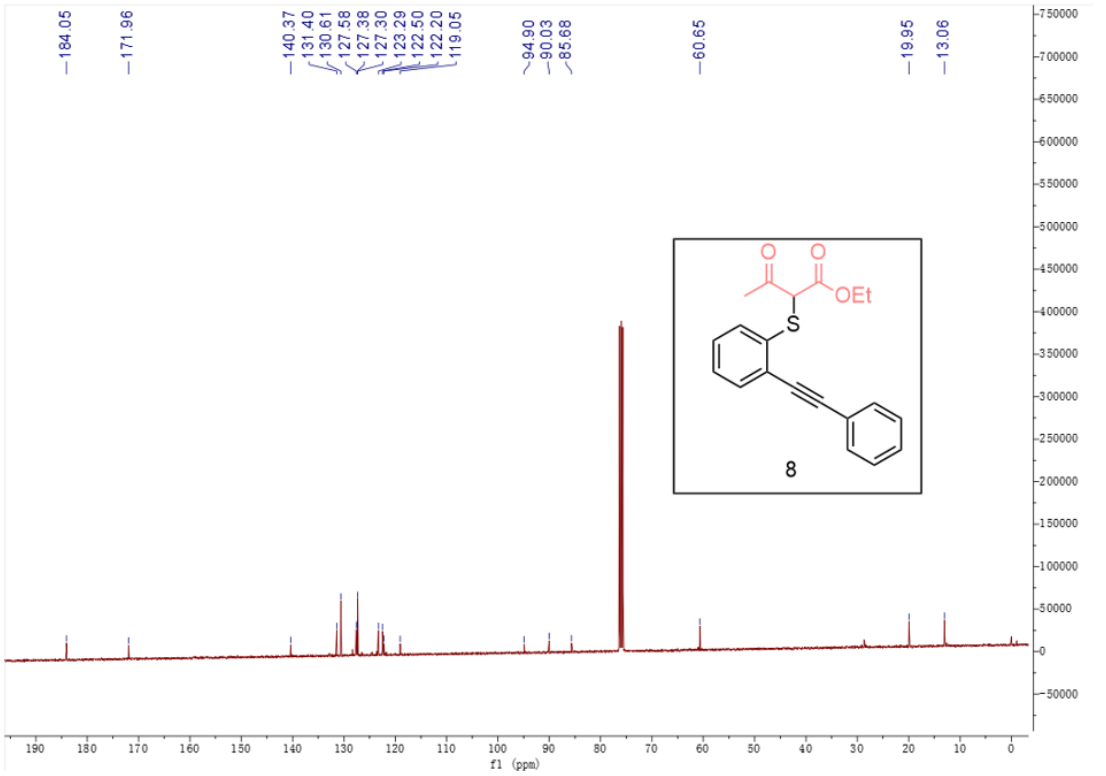


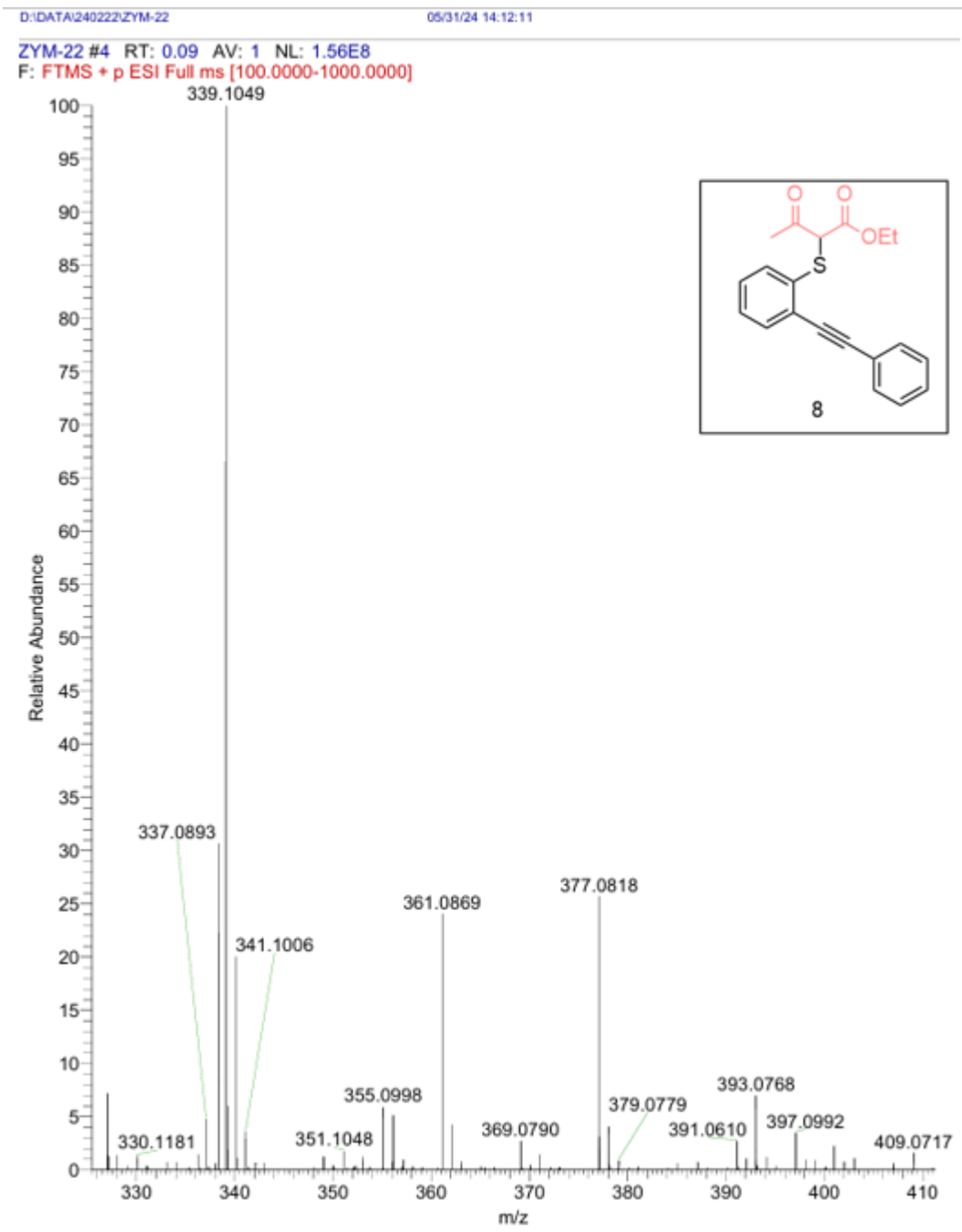

7.68
7.68
7.66
7.66
7.58
7.57
7.57
7.56
7.56
7.48
7.47
7.46
7.46
7.37
7.36
7.36
7.35
7.35
7.34
7.33
7.32
7.32
7.30
7.30
7.29
7.29
7.28
7.28
7.27
7.27
7.26
7.26
7.26
7.25
7.24
7.24
7.23
7.23
7.22
7.18
7.16
7.16
7.14
7.14
3.96
S
S
Bn
6
1.00
1.90
1.02
9.64
1.10
2.00
f1 (ppm)

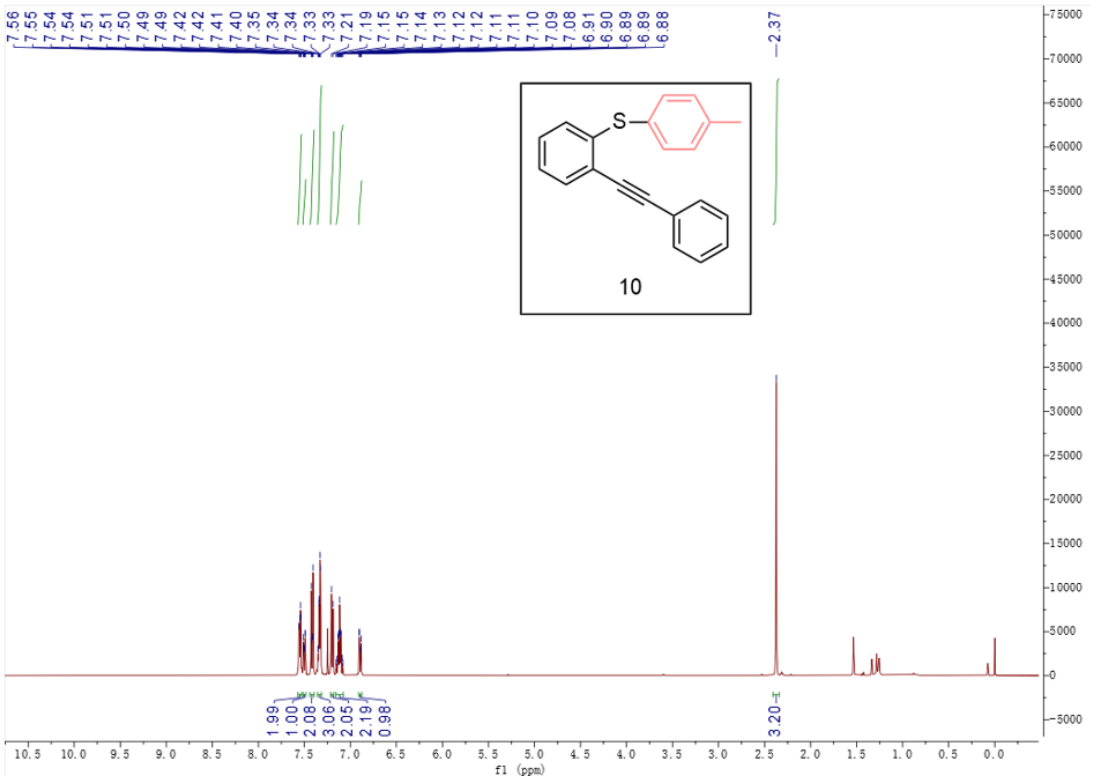

139.63
136.56
132.34
131.66
129.35
128.75
128.59
128.58
128.35
127.59
126.03
125.94
123.00
121.64
96.42
86.42
43.54
S
S
Bn
6
f1 (ppm)

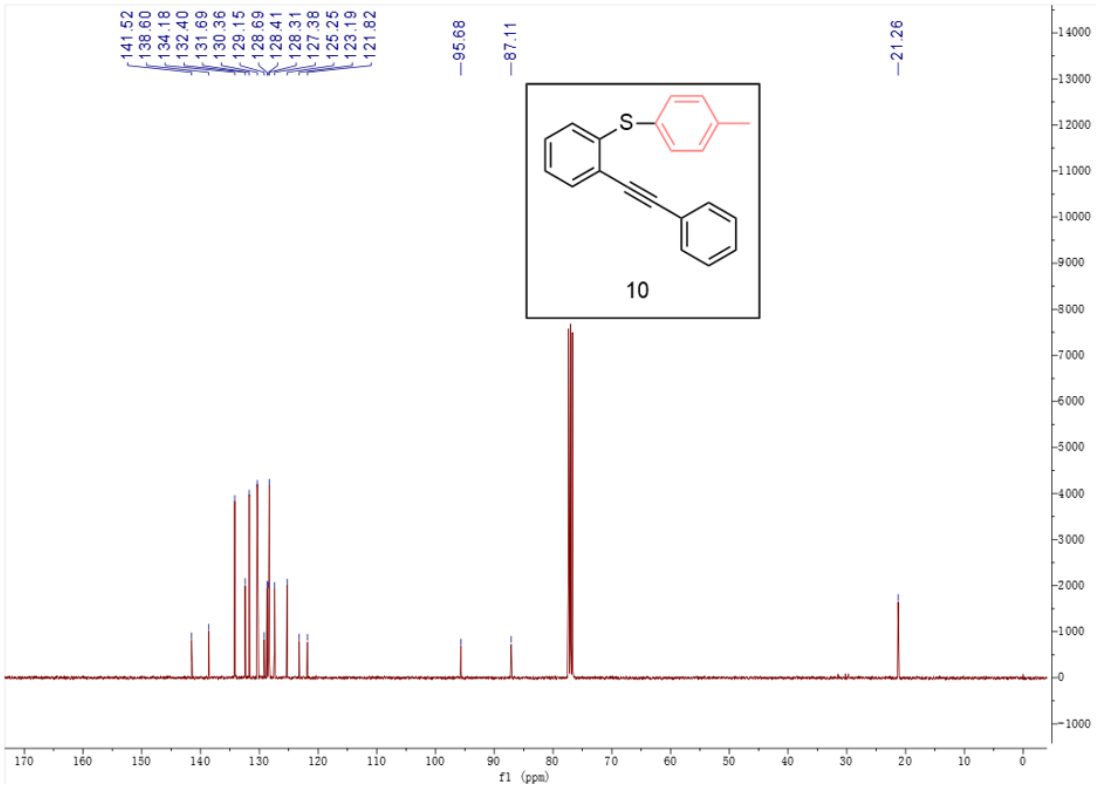

D:\DATA\240222\H7-27-31
05/31/24 14:21:44
H7-27-31 #5 RT: 0.09 AV: 1 SB: 6 0.33-0.47 , 0.00-0.05 NL: 1.61E7
F: FTMS + p ESI Full ms [100.0000-1000.0000]
333.0765
334.0800
335.0724
333.7070
Relative Abundance
m/z
S
S
Bn
6


13.89
13.89
7.54
7.53
7.53
7.52
7.51
7.41
7.41
7.39
7.39
7.29
7.29
7.29
7.28
7.27
7.16
7.16
7.14
7.14
7.12
7.12
7.03
7.03
7.01
7.01
6.99
6.99
6.85
6.85
6.83
6.83
4.18
4.16
4.15
4.13
2.25
2.25
1.14
1.12
1.10
OEt
S
8
0.95
2.05
1.09
3.15
1.09
1.05
1.00
2.15
3.09
3.14
f1 (ppm)


184.05
171.96
140.37
131.40
130.61
127.58
127.38
127.30
123.29
122.50
122.20
119.05
94.90
90.03
85.68
60.65
19.95
13.06
OEt
S
8
f1 (ppm)


D:\DATA\240222\ZYM-22
05/31/24 14:12:11
ZYM-22 #4 RT: 0.09 AV: 1 NL: 1.56E8
F: FTMS + p ESI Full ms [100.0000-1000.0000]
339.1049
337.0893
341.1006
361.0869
377.0818
355.0998
379.0779
393.0768
369.0790
391.0610
397.0992
330.1181
351.1048
409.0717
Relative Abundance
m/z
OEt
S
8


7.56
7.55
7.54
7.54
7.51
7.51
7.50
7.49
7.49
7.42
7.42
7.41
7.40
7.35
7.34
7.34
7.33
7.33
7.21
7.19
7.15
7.15
7.14
7.13
7.12
7.12
7.11
7.11
7.10
7.09
7.08
6.91
6.90
6.89
6.89
6.88
2.37
S
10
1.99
1.00
2.08
3.06
2.05
2.19
0.98
3.20
f1 (ppm)


141.52
138.60
134.18
132.40
131.69
130.36
129.15
128.69
128.41
128.31
127.38
125.25
123.19
121.82
95.68
87.11
21.26
S
10
f1 (ppm)

D:\DATA\240222\ZYM-23-2 05/31/24 14:16:57

ZYM-23-2 #5-7 RT: 0.09-0.13 AV: 2 NL: 1.30E8
F: FTMS + p ESI Full ms [100.0000-1000.0000]