*Electronic Supporting Information*

# DMSO-assisted environmentally benign synthesis of benzo[*c*]-chromeno[4,3,2-*gh*]phenanthridines by remote oxidative hetero cross-coupling cyclization and aromatization reaction

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**General information and methods**

Melting points were determined on a melting point apparatus and are uncorrected. $^{1}H$ and $^{13}C$ NMR spectra were recorded on 400, 500, and 600 MHz and 100, 125, and 150 MHz NMR spectrometers. TMS was used as an internal reference; chemical shifts (δ scale) are reported in parts per million(ppm). $^{1}H$ NMR spectra are reported in the order: multiplicity, coupling constant (J value) in hertz (Hz), and no. of protons; signals were characterized as s (singlet), d (doublet), t (triplet), m (multiplet), and bs (broad). IR spectra were recorded on an IR spectrophotometer. HRMS spectra were recorded using ESI and APCI (TOF) mode. The crystal structure was determined using a single crystal XRD diffractometer.

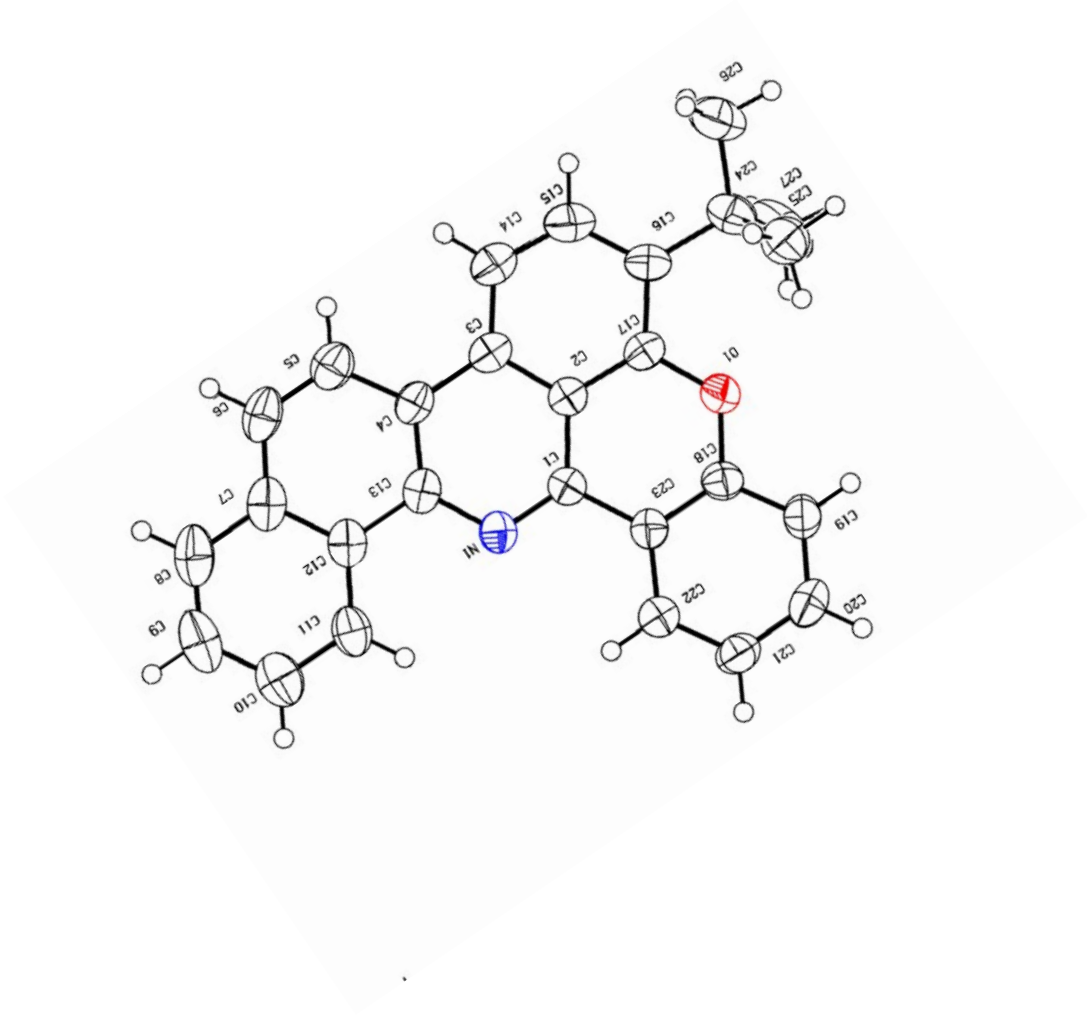
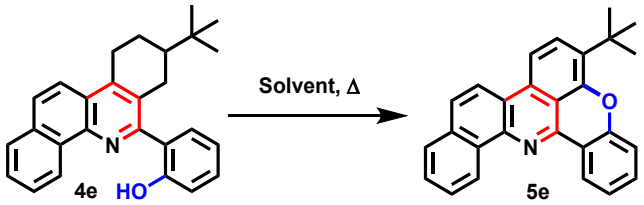



Figure S1. ORTEP diagrams of compound **5e**

Table S1. Crystal data and structure refinement for compound **5e**

| **Entry** | **Identification code** | **Compound 5e** |
|---|---|---|
| 01 | Empirical formula | $C_{27}H_{25}NO_2$ |
| 02 | Formula weight | 395.48 |
| 03 | Temperature | 296 K |
| 04 | Wavelength | 0.71073 |
| 05 | Radiation type | Mo K\a |
| 06 | Radiation system | Fine-focus sealed tube |
| 07 | Crystal system | Monoclinic |
| 08 | Space group | P 21/n |
| 09 | Cell length | a=8.9415 (9)<br>b=20.8156 (11)<br>c=11.0217 (8) |
| 10 | Cell angle | α=90<br>β=109.915 (9)<br>γ=90 |
| 11 | Cell volume | 1928.7 (3) |
| 12 | Density | 1.293 |
| 13 | Completeness to theta | 100 |
| 14 | Absorption correction | multi-scan |
| 15 | Refinement method | Full-matrix least-squares on F2 |
| 16 | Index ranges | -10<=h<=10,<br>-24<=k<=24,<br>-13<=l<=13 |
| 17 | Reflection number | 3415 |
| 18 | Theta range | 25.049 |
| 19 | Cell formula units Z | 4 |
| 20 | CCDC no | 2153056 |

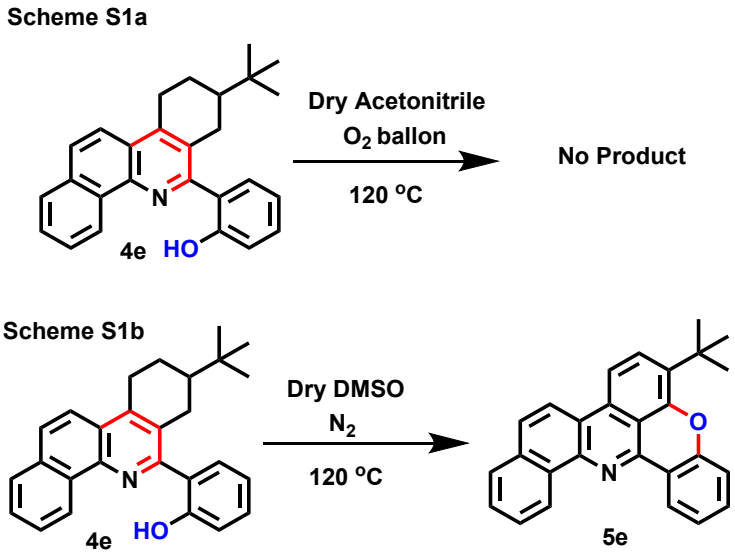
Table S2. Optimization of reaction conditions[a,b]




| Entry | Solvent | Temperature | Time (h) | % Yield[b] (**5e**) |
|---|---|---|---|---|
| 1. | DMSO | 25°C | 24 | NR |
| 2. | DMSO | 50°C | 24 | NR |
| 3. | DMSO | 70°C | 24 | NR |
| 4. | DMSO | 80°C | 24 | NR |
| 5. | DMSO | 90°C | 24 | 10 |
| 6. | DMSO | 100°C | 15 | 30 |
| 7. | DMSO | 110°C | 9 | 54 |
| 8. | **DMSO** | **120°C** | **6** | **70** |
| 9. | DMSO | 130°C | 6 | 72 |
| 10. | Neat | 120°C | 24 | NR |
| 11. | Toluene | 110°C | 24 | NR |
| 12. | $H_2O$ | 100°C | 24 | NR |
| 13. | DMF | 120°C | 6 | 30 |

[a]All reactions were carried out with 2-(8-(*tert*-butyl)-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (**4e**) in 1 mL solvent. [b]Isolated yield. NR. No reaction.

**Scheme S1. Control experiments**




Reactions was carried out with 2-(8-(*tert*-butyl)-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (**4e**) (0.20 mmol) derivatives in 1 mL DMSO solvent at 120 °C.

## Experimental Section

**General procedure for synthesis of 2-(7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)-phenol derivatives 4.**

Into a dry 25 mL single necked round-bottomed flask, 1-naphthylamine **1** (143 mg, 1.0 mmol), aromatic aldehyde **2** (1.0 mmol), and cyclohexanone derivatives **3** (154 mg, 1.0 mmol) were dissolved in acetonitrile (5.0 mL). A catalytic amount of (±)-camphor-10-sulfonic acid (CSA) (0.023 g, 0.10 mmol) was added and the reaction mixture was allowed to stir at reflux condition on preheated oil bath. The progress of reaction was monitored by TLC time to time in an interval of 30 minutes. After completion of the reaction, the reaction mixture was allowed to cool at room temperature. Precipitate, thus obtained was filtered through Buchner funnel, washed with acetonitrile (3 x 2.0 mL), air dried and recrystallized with acetone to obtain desired product **4**.

**General procedure for synthesis of benzo[*c*]chromeno[4,3,2-*gh*]phenanthridine derivatives 5.**

Compound **4** (0.20 mmol) was dissolved in DMSO (1.0 mL) and taken into a 10 mL single necked-round bottomed flask. The resultant solution was stirred at preheated oil bath (120 °C) in air atmosphere. After completion of reaction (monitored on TLC), the reaction mixture was cooled to room temperature. Distilled water (10 mL) was added, extracted with ethyl acetate (3 x 10 mL), washed with distilled water (3 x 10 mL), dried over $Na_2SO_4$, filtered, and evaporated on rotary evaporator under reduced pressure. Crude was purified through silica gel (60-120 mesh) column chromatography with hexane to obtain pure product **5**.

**2-(7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4a).**Yield 71% (231 mg), white solid, mp 176 °C; $^1$H NMR (400 MHz, Chloroform-*d*) δ 12.44 (s, 1H), 9.14 (d, *J* = 8.0 Hz, 1H), 7.94 – 7.85 (m, 3H), 7.76 – 7.68 (m, 2H), 7.64 (d, *J* = 6.5 Hz, 1H), 7.35 (t, *J* = 7.7 Hz, 1H), 7.20 (d, *J* = 7.3 Hz, 1H), 6.98 (t, *J* = 7.5 Hz, 1H), 3.31 (t, *J* = 6.6 Hz, 2H), 3.08 (t, *J* = 5.9 Hz, 2H), 2.08 – 2.02 (m, 2H), 1.80 – 1.76 (m, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 157.5, 156.0, 145.1, 141.4, 133.3, 130.7, 130.6, 130.5, 130.3, 128.2, 128.0, 127.9, 127.6, 124.4, 124.0, 122.6, 120.4, 118.5, 117.9, 30.1, 26.7, 23.0, 22.5. IR (KBr)$\nu_{max}$: 2957, 1614, 1251 cm$^{-1}$; HRMS (ESI) Calcd for $C_{23}H_{20}NO$ 326.1540 (M + H$^+$); Found 326.1555.

**2-(9-methyl-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4b).** Yield 72% (244 mg), white solid, mp 178–180 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 12.54 (s, 1H), 9.09 (d, *J* = 8.1 Hz, 1H), 7.90 (d, *J* = 7.7 Hz, 1H), 7.88 – 7.85 (m, 1H), 7.81 (d, *J* = 8.8 Hz, 1H), 7.72 (t, *J* = 7.4 Hz, 1H), 7.69 – 7.65 (m, 2H), 7.36 (t, *J* = 7.7 Hz, 1H), 7.18 (d, *J* = 8.2 Hz, 1H), 6.97 (t, *J* = 7.5 Hz, 1H), 3.45 (d, *J* = 17.7 Hz, 1H), 3.16 (t, *J* = 13.1 Hz, 1H), 3.08 (d, *J* = 16.2 Hz, 1H), 2.7 – 2.67 (m, 1H), 2.04 (s, 1H), 1.98 (d, *J* = 13.9 Hz, 1H), 1.26 (d, *J* = 11.4 Hz, 1H), 1.22 (d, *J* = 6.5 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 157.5, 155.9, 145.1, 141.4, 133.3, 130.6, 130.3, 130.0, 128.1, 128.0, 127.8, 127.6, 124.2, 124.0, 122.6, 120.4, 118.5, 117.9, 35.5, 31.2, 30.1, 28.8, 22.3. IR (KBr)$\nu_{max}$: 2956, 1617, 1251 cm$^{-1}$; HRMS (ESI) Calcd for $C_{24}H_{22}NO$ 340.1696 (M + H$^+$); Found 340.1696.

**2-(8-methyl-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4c).** Yield 72% (244 mg), white solid, mp 212–214 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 12.39 (s, 1H), 9.11 – 9.08 (m, 1H), 7.91 (d, *J* = 7.6 Hz, 1H), 7.84 (q, *J* = 9.1 Hz, 2H), 7.72 (t, *J* = 7.4 Hz, 1H), 7.70 – 7.67 (m, 1H), 7.65 (d, *J* = 7.7 Hz, 1H), 7.36 (t, *J* = 7.7 Hz, 1H), 7.19 (d, *J* = 8.1 Hz, 1H), 6.98 (t,

*J* = 7.5 Hz, 1H), 3.44 – 3.40 (m, 1H), 3.19 (dt, *J* = 17.8, 8.8 Hz, 1H), 3.06 (d, *J* = 16.2 Hz, 1H), 2.75 (dd, *J* = 16.0, 10.5 Hz, 1H), 2.13 – 2.09 (m, 1H), 1.70 (s, 1H), 1.63 – 1.56 (m, 1H), 1.11 (d, *J* = 6.4 Hz, 3H).$^{13}$C NMR (125 MHz, $CDCl_3$) δ 157.4, 155.8, 145.0, 141.2, 133.3, 130.7, 130.4, 130.1, 128.7, 128.2, 128.0, 128.0, 127.6, 124.2, 124.0, 122.5, 120.5, 118.6, 118.0, 38.2, 30.5, 29.2, 26.9, 21.7. IR (KBr)$\nu_{max}$: 2958, 1614, 1250 $cm^{-1}$; HRMS (ESI) Calcd for $C_{24}H_{22}NO$ 340.1696 ($M + H^+$); Found 340.1705.

**2-(8-ethyl-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4d).** Yield 74% (261 mg), solid, white solid, mp 164 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 12.57 (s, 1H), 9.09 (d, *J* = 8.0 Hz, 1H), 7.92 – 7.80 (m, 3H), 7.70 (m, 3H), 7.36 (t, *J* = 7.6 Hz, 1H), 7.19 (d, *J* = 8.1 Hz, 1H), 6.98 (t, *J* = 7.4 Hz, 1H), 3.44 (d, *J* = 17.8 Hz, 1H), 3.21 – 3.18 (m, 1H), 3.12 (d, *J* = 16.4 Hz, 1H), 2.79 – 2.75 (m, 1H), 2.19 (s, 1H), 1.57 (s, 2H), 1.46 – 1.43 (m, 2H), 0.97 (t, *J* = 6.7 Hz, 3H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 157.6, 156.0, 145.0, 141.4, 133.3, 130.7, 130.6, 130.3, 130.0, 128.1, 128.0, 127.9, 127.6, 124.2, 124.0, 122.6, 120.5, 118.5, 117.9, 36.4, 35.9, 28.8, 28.1, 26.8, 11.6. IR (KBr)$\nu_{max}$: 2956, 1614, 1252 $cm^{-1}$; HRMS (ESI) Calcd for $C_{25}H_{24}NO$ 354.1853 ($M + H^+$); Found 354.1841.

**2-(8-(*tert*-butyl)-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4e).** Yield 71% (270 mg), white solid, mp 230 °C; $^1$H NMR (400 MHz, Chloroform-*d*) δ 12.65 (s, 1H), 9.19 (d, *J* = 7.9 Hz, 1H), 7.93 – 7.83 (m, 3H), 7.74 – 7.67 (m, 3H), 7.36 (t, *J* = 8.5 Hz, 1H), 7.19 (d, *J* = 7.1 Hz, 1H), 6.99 (t, *J* = 7.0 Hz, 1H), 3.54 (d, *J* = 23.8 Hz, 1H), 3.23 – 3.13 (m, 1H), 3.08 (dt, *J* = 16.3, 2.8 Hz, 1H), 2.91 – 2.84 (m, 1H), 2.27 – 2.23 (m, 1H), 1.63 – 1.58 (m, 1H), 1.30 (t, *J* = 11.5 Hz, 1H), 0.98 (s, 9H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 157.6, 156.2, 145.0, 141.3, 133.3, 130.8, 130.7, 130.6, 130.2, 128.1, 128.1, 127.9, 127.6, 124.1, 124.0, 122.6, 120.6, 118.5, 118.0, 44.8, 32.5, 31.7, 28.2, 27.5, 23.9. IR (KBr)$\nu_{max}$: 2957, 1620, 1248 $cm^{-1}$; HRMS (ESI) Calcd for $C_{27}H_{28}NO$ 382.2166 ($M + H^+$); Found 382.2174.

**4-chloro-2-(9-methyl-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4f).** Yield 71% (264 mg), white solid, mp 200–202 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 12.51 (s, 1H), 9.04 (t, *J* = 6.4 Hz, 1H), 7.92 – 7.82 (m, 3H), 7.70 (dq, *J* = 14.8, 7.2 Hz, 2H), 7.64 – 7.63 (m, 1H), 7.29 (dd, *J* = 8.7, 2.2 Hz, 1H), 7.10 (d, *J* = 8.8 Hz, 1H), 3.46 (t, *J* = 17.4 Hz, 1H), 3.16 – 3.05 (m, 2H), 2.76 – 2.67 (m, 1H), 2.03 (s, 2H), 1.29 – 1.24 (m, 1H), 1.22 (d, *J* = 6.6 Hz, 3H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 156.3, 154.6, 145.5, 141.5, 133.4, 130.6, 130.3, 129.9, 129.8, 128.3, 128.2, 128.1, 127.7, 124.5, 123.9, 123.7, 123.2, 120.4, 119.3, 35.5, 31.2, 29.9, 28.7, 22.3.

IR (KBr)$\nu_{max}$: 2958, 1615, 1251 $cm^{-1}$; HRMS (ESI) Calcd for $C_{24}H_{21}ClNO$ 374.1307 ($M + H^+$); Found 374.1308.

**4-chloro-2-(8-methyl-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4g).** Yield 71% (264 mg), white solid, mp 189–191 °C; $^1$H NMR (400 MHz, Chloroform-*d*) δ 12.39 (s, 1H), 9.02 (d, *J* = 7.8 Hz, 1H), 7.91 (d, *J* = 7.5 Hz, 1H), 7.81 (s, 2H), 7.75 – 7.67 (m, 2H), 7.59 (d, *J* = 2.4 Hz, 1H), 7.30 (dd, *J* = 8.7, 2.4 Hz, 1H), 7.11 (d, *J* = 8.7 Hz, 1H), 3.34 (dd, *J* = 18.1, 4.4 Hz, 1H), 3.13 (dt, *J* = 17.7, 8.5 Hz, 1H), 3.01 (d, *J* = 16.1 Hz, 1H), 2.68 (dd, *J* = 16.1, 10.3 Hz, 1H), 2.10 – 2.05 (m, 1H), 1.71 – 1.67 (m, 1H), 1.53 (dp, *J* = 17.6, 6.5 Hz, 1H), 1.11 (d, *J* = 6.5 Hz, 3H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 156.1, 154.5, 145.3, 141.2, 133.4, 130.4, 129.9, 129.8, 128.4, 128.3, 128.1, 127.8, 124.5, 123.9, 123.7, 123.4, 120.4, 119.3, 37.9, 30.3, 29.0, 26.8, 21.6. IR (KBr)$\nu_{max}$: 2957, 1614, 1251 $cm^{-1}$; HRMS (ESI) Calcd for $C_{24}H_{21}ClNO$ 374.1306 ($M + H^+$); Found 374.1305.

**4-chloro-2-(8-ethyl-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4h).** Yield 71% (274 mg), white solid, mp 166–168 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 12.18 (s, 1H), 9.03 (d, *J* = 7.6 Hz, 1H), 7.91 (d, *J* = 7.2 Hz, 1H), 7.82 (s, 2H), 7.71 (t, *J* = 8.3 Hz, 2H), 7.59 (s, 1H), 7.29 (d, *J* = 8.6 Hz, 1H), 7.11 (d, *J* = 8.3 Hz, 1H), 3.34 (d, *J* = 17.6 Hz, 1H), 3.14 – 3.09 (m, 1H), 3.04 (d, *J* = 16.1 Hz, 1H), 2.70 – 2.67 (m, *J* = 15.6 Hz, 1H), 2.13 (s, 1H), 1.48 – 1.44 (m, 2H), 0.96 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 156.0, 154.4, 141.0, 133.3, 130.5, 130.1, 129.8, 128.8, 128.4, 128.3, 128.1, 127.8, 126.2, 124.5, 123.9, 123.6, 123.4, 120.4, 119.3, 35.8, 35.6, 28.6, 27.7, 26.8, 11.5. IR (KBr)$\nu_{max}$: 2958, 1617, 1253 $cm^{-1}$; HRMS (ESI) Calcd for $C_{25}H_{23}ClNO$ 388.1463 ($M + H^+$); Found 388.1462.

**4-chloro-2-(8-*iso*propyl-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4i).** Yield 70% (280 mg), white solid, mp 211–213 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.38 (d, *J* = 7.6 Hz, 1H), 7.89 (d, *J* = 4.2 Hz, 1H), 7.81 (d, *J* = 9.1 Hz, 1H), 7.74 (d, *J* = 7.2 Hz, 1H), 7.69 – 7.63 (m, 2H), 7.55 – 7.52 (m, 2H), 7.49 – 7.46 (m, 1H), 3.53 (dd, *J* = 12.5, 5 Hz, 1H), 3.10 (td, *J* = 11.7, 6.4 Hz, 1H), 2.88 (d, *J* = 16.3 Hz, 1H), 2.71 – 2.66 (m, 1H), 2.24 – 2.20 (m, 1H), 1.51 (dq, *J* = 12.3, 6.9 Hz, 1H), 1.42 (d, *J* = 5.2 Hz, 1H), 1.40 – 1.37 (m, 1H), 0.93 (s, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 158.6, 143.4, 142.2, 141.7, 133.1, 132.2, 130.5, 129.6, 129.5, 129.4, 128.3, 128.1, 128.0, 127.5, 127.1, 126.7, 125.1, 124.0, 120.7, 44.6, 32.4, 30.4, 27.6, 27.4, 23.9. IR (KBr)$\nu_{max}$: 2957, 1614, 1251 $cm^{-1}$; HRMS (ESI) Calcd for $C_{26}H_{25}ClNO$ 402.1620 ($M + H^+$); Found 402.1627.

**2-(8-(*tert*-butyl)-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)-4-chlorophenol (4j).** Yield 71% (295 mg), white solid, mp 175 °C; $^1$H NMR (600 MHz, Chloroform-*d*) δ 12.71 (s, 1H), 9.04 (d, *J* = 8.1 Hz, 1H), 7.93 (d, *J* = 7.7 Hz, 1H), 7.87 – 7.83 (m, 2H), 7.73 (t, *J* = 7.0 Hz, 1H), 7.70 (t, *J* = 6.9 Hz, 1H), 7.66 (d, *J* = 2.3 Hz, 1H), 7.30 (dd, *J* = 8.7, 6.6 Hz, 1H), 7.11 (d, *J* = 8.7 Hz, 1H), 3.49 (d, *J* = 17.7 Hz, 1H), 3.16 – 3.10 (m, 1H), 3.04 (d, *J* = 15.8 Hz, 1H), 2.84 – 2.79 (m, 1H), 2.22 (s, 1H), 1.51 (d, *J* = 6.7 Hz, 1H), 1.28 (t, *J* = 12.1 Hz, 1H), 0.98 (s, 9H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ 156.4, 154.9, 145.4, 141.3, 133.4, 130.7, 130.5, 130.4, 129.8, 128.4, 128.3, 128.2, 127.8, 124.4, 123.9, 123.6, 123.3, 120.6, 119.4, 44.7, 32.6, 31.5, 28.2, 27.6, 23.8. IR (KBr)$\nu_{max}$: 2958, 1614, 1248 cm$^{-1}$; HRMS (ESI) Calcd for $C_{27}H_{27}ClNO$ 416.1776 (M + H$^+$); Found 416.1785.

**4-chloro-2-(8-phenyl-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4k).** Yield 71% (309 mg), green solid, mp 175 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 12.20 (s, 1H), 9.06 (d, *J* = 8.0 Hz, 1H), 7.93 (d, *J* = 7.4 Hz, 1H), 7.84 (s, 2H), 7.75 – 7.71 (m, 2H), 7.53 (s, 1H), 7.33 (d, *J* = 7.1 Hz, 2H), 7.24 (d, *J* = 5.4 Hz, 4H), 7.06 (d, *J* = 8.5 Hz, 1H), 3.44 (d, *J* = 16.9 Hz, 1H), 3.28 (d, *J* = 8.3 Hz, 1H), 3.19 (s, 2H), 2.77 (s, 1H), 2.30 (s, 1H), 2.03 (s, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 155.9, 154.7, 145.2, 144.8, 141.7, 133.4, 130.5, 130.4, 129.8, 129.7, 128.8, 128.5, 128.4, 128.2, 127.8, 127.0, 126.7, 124.4, 124.0, 123.7, 123.5, 120.5, 119.3, 40.5, 37.4, 29.5, 27.5. IR (KBr)$\nu_{max}$: 2948, 1614, 1255 cm$^{-1}$; HRMS (ESI) Calcd for $C_{29}H_{23}ClNO$ 436.1463 (M + H$^+$); Found 436.1472.

**4-bromo-2-(9-methyl-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4l).** Yield 71% (296 mg), pale yellow solid, mp 268–270 °C; $^1$H NMR (400 MHz, DMSO-$d_6$) δ 9.36 (d, *J* = 9.1 Hz, 1H), 8.29 (d, *J* = 8 Hz, 1H), 8.00 – 7.94 (m, 2H), 7.71 – 7.69 (m, 2H), 7.33 (d, *J* = 7.9 Hz, 2H), 7.21 (d, *J* = 8.7 Hz, 1H), 3.03 – 2.99 (m, 2H), 2.91 – 2.85 (m, 1H), 2.72 – 2.71 (m, 1H), 2.20 – 2.10 (m, 2H), 1.57 (dq, *J* = 16.3, 5.9, 5.3 Hz, 1H), 1.38 (d, *J* = 6.5 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ 155.1, 151.3, 142.4, 139.4, 134.8, 133.7, 132.7, 131.8, 130.5, 130.3, 128.7, 128.6, 128.4, 126.0, 125.8, 124.1, 119.8, 119.3, 110.6, 35.8, 27.7, 26.9, 21.7, 21.1. IR (KBr)$\nu_{max}$: 2958, 2925, 1610, cm$^{-1}$; HRMS (ESI) Calcd for $C_{24}H_{21}BrNO$ 418.0801 (M + H$^+$); Found 418.0806 and 420.0787.

**4-bromo-2-(8-methyl-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4m).** Yield 71% (296 mg), pale yellow solid, mp 228–230 °C; $^1$H NMR (400 MHz, DMSO-$d_6$) δ 9.37 (d, *J* = 8.6 Hz, 1H), 8.30 – 8.26 (m, 2H), 8.00 – 7.94 (m, 2H), 7.76 – 7.71 (m, 2H), 7.34 (d, *J* = 7.9

Hz, 1H), 7.23 (d, *J* = 8.1 Hz, 1H), 3.45 – 3.36 (m, 1H), 2.88 (dd, *J* = 16.7, 3.8 Hz, 1H), 2.72 (s, 1H), 2.62 (dd, *J* = 16.8, 10.6 Hz, 1H), 2.26 – 2.24 (m, 1H), 2.02 – 2.00 (m, 1H), 1.74 – 1.64 (m, 1H), 1.22 (d, *J* = 6.4 Hz, 3H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 155.8, 133.9, 133.9, 133.8, 133.4, 132.6, 132.6, 130.7, 129.0, 128.9, 128.8, 128.2, 126.2, 124.8, 124.1, 120.1, 111.1, 36.9, 30.0, 28.7, 27.0, 21.5. IR (KBr)$\nu_{max}$: 2956, 1613, 1251 cm$^{-1}$; HRMS (ESI) Calcd for $C_{24}H_{21}BrNO$ 418.0801 (M + H$^+$); Found 418.0812 and 420.0796.

**4-bromo-2-(8-ethyl-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4n).** Yield 71% (306 mg), pale yellow solid, mp 158–160 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 12.57 (s, 1H), 9.03 (d, *J* = 7.9 Hz, 1H), 7.92 (d, *J* = 7.5 Hz, 1H), 7.85 (q, *J* = 9.0 Hz, 2H), 7.77 (s, 1H), 7.71 (dt, *J* = 16.6, 6.8 Hz, 2H), 7.44 (d, *J* = 8.6 Hz, 1H), 7.07 (d, *J* = 8.7 Hz, 1H), 3.43 (d, *J* = 18.2 Hz, 1H), 3.22 – 3.15 (m, 1H), 3.09 (d, *J* = 16.0 Hz, 1H), 2.76 – 2.71 (m, 1H), 2.18 (s, 1H), 1.51 – 1.44 (m, 2H), 0.98 (t, *J* = 7.1 Hz, 3H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 156.8, 154.6, 145.4, 141.4, 133.4, 133.2, 132.7, 130.5, 129.9, 128.3, 128.3, 128.1, 127.7, 124.5, 124.2, 123.9, 120.5, 119.8, 110.3, 36.1, 35.8, 28.6, 27.9, 26.8, 11.6. IR (KBr)$\nu_{max}$: 2957, 1617, 1250 cm$^{-1}$; HRMS (ESI) Calcd for $C_{25}H_{23}BrNO$ 432.0958 (M + H$^+$); Found 432.0958 and 434.0940.

**4-bromo-2-(8-(*tert*-butyl)-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4o).** Yield 71% (326 mg), pale yellow solid, mp 170 °C; $^1$H NMR (600 MHz, Chloroform-*d*) δ 12.72 (s, 1H), 9.02 (d, *J* = 8.0 Hz, 1H), 7.92 (d, *J* = 7.4 Hz, 1H), 7.83 – 7.79 (m, 3H), 7.73 (t, *J* = 7.5 Hz, 1H), 7.69 (t, *J* = 7.3 Hz, 1H), 7.43 (d, *J* = 6.4 Hz, 1H), 7.05 (d, *J* = 8.7 Hz, 1H), 3.41 (d, *J* = 17.6 Hz, 1H), 3.11 – 3.05 (m, 1H), 3.01 (d, *J* = 15.8 Hz, 1H), 2.78 – 2.74 (m, 1H), 2.19 – 2.16 (m, 1H), 1.44 (dd, *J* = 12.1, 5.5 Hz, 1H), 1.29 – 1.22 (m, 1H), 0.97 (s, 9H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ 156.8, 154.6, 145.3, 141.1, 133.3, 133.2, 132.7, 130.6, 130.4, 128.3, 128.2, 128.1, 127.8, 124.3, 124.0, 123.8, 120.5, 119.8, 110.2, 44.6, 32.5, 31.4, 28.1, 27.5, 23.6. IR (KBr)$\nu_{max}$: 2959, 1614, 1250 cm$^{-1}$; HRMS (ESI) Calcd for $C_{27}H_{27}BrNO$ 460.1271 (M + H$^+$); Found 460.1275 and 462.1258.

**2-(8-(*tert*-butyl)-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)-4,6-dichlorophenol (4p).** Yield 70% (314 mg), pale yellow solid, mp 180 °C; $^1$H NMR (400 MHz, Chloroform-*d*) δ 13.72 (s, 1H), 9.01 (d, *J* = 7.9 Hz, 1H), 7.91 (d, *J* = 7.6 Hz, 1H), 7.82 – 7.68 (m, 4H), 7.56 (d, *J* = 2.1 Hz, 1H), 7.44 (d, *J* = 2.2 Hz, 1H), 3.34 (d, *J* = 23.2 Hz, 1H), 3.07 – 2.98 (m, 1H), 2.93 (d, *J* = 15.8 Hz, 1H), 2.70 – 2.63 (m, 1H), 2.15 – 2.12 (m, 1H), 1.35 (dt, *J* = 12.2, 6.1 Hz, 1H), 1.19 (t, *J* = 11.7 Hz, 1H), 0.94 (s, 9H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 154.1, 152.9, 145.6, 140.8, 133.3,

130.7, 130.4, 130.1, 128.6, 128.5, 128.4, 128.2, 128.0, 124.5, 123.9, 123.8, 123.4, 122.8, 120.3, 44.5, 32.4, 31.4, 28.1, 27.4, 23.5. IR (KBr)$\nu_{max}$: 2957, 1614, 1251 $cm^{-1}$; HRMS (ESI) Calcd for $C_{27}H_{26}Cl_2NO$ 450.1386 ($M + H^+$); Found 450.1425.

**2-(8-ethyl-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)-5-methoxyphenol (4q).** Yield 71% (271 mg), white solid, mp 204–206 °C; $^1H$ NMR (500 MHz, Chloroform-*d*) δ 13.53 (s, 1H), 9.04 (d, *J* = 8.1 Hz, 1H), 7.90 (d, *J* = 7.8 Hz, 1H), 7.85 (d, *J* = 9.1 Hz, 1H), 7.79 (d, *J* = 9.1 Hz, 1H), 7.72 (t, *J* = 7.4 Hz, 1H), 7.67 (t, *J* = 7.3 Hz, 1H), 7.62 (d, *J* = 8.7 Hz, 1H), 6.72 (s, 1H), 6.55 (d, *J* = 10.3 Hz, 1H), 3.89 (s, 3H), 3.42 (d, *J* = 22.7 Hz, 1H), 3.20 – 3.15 (m, 1H), 3.11 (d, *J* = 16.5 Hz, 1H), 2.74 (dd, *J* = 15.9, 8.9 Hz, 1H), 2.19 – 2.16 (m, 1H), 1.55 – 1.41 (m, 4H), 0.97 (t, *J* = 6.8 Hz, 3H). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 161.6, 160.0, 156.2, 144.8, 141.0, 133.3, 131.4, 130.4, 129.6, 128.0, 128.0, 127.5, 127.4, 123.9, 123.7, 120.5, 115.2, 105.3, 102.3, 55.4, 36.7, 36.0, 28.8, 28.0, 26.9, 11.6. IR (KBr)$\nu_{max}$: 2950, 1620, 1251 $cm^{-1}$; HRMS (ESI) Calcd for $C_{26}H_{26}NO_2$ 384.1959 ($M + H^+$); Found 384.1958.

**2-(8-(*tert*-butyl)-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)-5-methoxyphenol (4r).** Yield 71% (292 mg), white solid, mp 162–164 °C; $^1H$ NMR (600 MHz, Chloroform-*d*) δ 13.35 (s, 1H), 8.88 (d, *J* = 7.9 Hz, 1H), 7.79 (d, *J* = 7.7 Hz, 1H), 7.77 (d, *J* = 9.1 Hz, 1H), 7.70 (d, *J* = 9.0 Hz, 1H), 7.56 (dd, *J* = 17.6, 7.6 Hz, 2H), 7.48 (d, *J* = 8.7 Hz, 1H), 6.56 (s, 1H), 6.42 (d, *J* = 8.6 Hz, 1H), 3.75 (s, 3H), 3.41 (d, *J* = 12.5 Hz, 1H), 3.05 – 3.02 (m, 1H), 2.91 (d, *J* = 16.0 Hz, 1H), 2.44 (s, 1H), 2.12 – 2.09 (m, 1H), 1.44 – 1.41 (m, 1H), 1.16 (t, *J* = 11.8 Hz, 1H), 0.84 (s, 9H). $^{13}C$ NMR (150 MHz, $CDCl_3$) δ 161.4, 159.6, 156.1, 152.7, 143.0, 141.8, 139.2, 136.6, 133.2, 131.0, 130.4, 128.0, 127.4, 127.4, 123.6, 123.6, 120.4, 105.2, 102.2, 55.3, 44.6, 32.3, 31.6, 28.1, 27.3, 23.6. IR (KBr)$\nu_{max}$: 2957, 1620, 1251 $cm^{-1}$; HRMS (ESI) Calcd for $C_{28}H_{30}NO_2$ 412.2272 ($M + H^+$); Found 412.2329.

**2-(12-bromo-8-(*tert*-butyl)-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol (4s).** Yield 69% (317 mg), yellow solid, mp 162–164 °C; $^1H$ NMR (400 MHz, DMSO-$d_6$) δ 9.23 (d, J = 7.6 Hz, 1H), 8.16 – 8.15 (m, 3H), 7.82 (p, J = 6.6 Hz, 2H), 7.43 (d, J = 7.4 Hz, 1H), 7.10 – 7.06 (m, 1H), 7.04 (d, J = 7.6 Hz, 1H), 3.30 – 3.15 (m, 2H), 2.69 (d, J = 18.0 Hz, 1H), 2.58 (d, J = 10.2 Hz, 1H), 2.18 (d, J = 8.6 Hz, 1H), 1.49 – 1.41 (m, 2H), 0.87 (s, 9H). $^{13}C$ NMR (100 MHz, DMSO) δ 155.3, 135.9, 133.6, 133.2, 132.9, 131.6, 131.1, 130.5, 129.7, 129.3, 129.0, 128.1, 125.2, 124.4, 124.4, 121.2, 120.2, 119.6, 116.3, 43.3, 32.5, 28.1, 27.4, 27.3, 23.3. IR (KBr)$\nu_{max}$:

2957, 1620, 1251 $cm^{-1}$; HRMS (ESI) Calcd for $C_{27}H_{27}BrNO$ 460.1271 (M + $H^+$); Found 460.1279, 462.1259.

**2-(12-bromo-8-(*tert*-butyl)-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)-4-chlorophenol (4t).** Yield 65% (320 mg), yellow solid, mp 174–176 °C; $^1$H NMR (400 MHz, DMSO-$d_6$) δ 9.20 – 9.18 (m, 1H), 8.35 – 8.32 (m, 1H), 8.26 (d, *J* = 8.0 Hz, 1H), 8.08 (s, 1H), 7.79 – 7.78 (d, *J* = 3.8 Hz, 1H), 7.41 (d, *J* = 3.8 Hz, 1H), 7.08 (d, *J* = 9.4 Hz, 1H), 7.03 (d, *J* = 8.7 Hz, 1H), 3.07 (d, *J* = 14.1 Hz, 2H), 2.71 (d, *J* = 16.1 Hz, 2H), 2.21 – 2.19 (m, 1H), 1.48 – 1.42 (m, 2H), 0.90 (s, 9H). $^{13}$C NMR (101 MHz, $CDCl_3$) δ 157.6, 156.2, 151.9, 145.0, 140.9, 132.1, 130.9, 130.7, 130.1, 127.7, 124.3, 123.4, 122.5, 121.4, 119.6, 118.6, 118.0, 116.5, 112.0, 44.7, 32.5, 31.7, 28.2, 27.5, 23.8. IR (KBr)$\nu_{max}$: 2957, 1620, 1251 $cm^{-1}$; HRMS (ESI) Calcd for $C_{27}H_{26}BrClNO$ 494.0881 (M + $H^+$); Found 494.0903, 496.0886.

**benzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5a).** Yield 60% (38 mg), solid, light green, mp 200–202 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.55 (d, *J* = 8.2 Hz, 1H), 8.91 (d, *J* = 7.8 Hz, 1H), 8.40 (d, *J* = 8.9 Hz, 1H), 8.20 (d, *J* = 8.2 Hz, 1H), 7.94 (dd, *J* = 13.3, 8.5 Hz, 2H), 7.83 (t, *J* = 8.1 Hz, 1H), 7.76 (t, *J* = 7.5 Hz, 1H), 7.68 (t, *J* = 7.4 Hz, 1H), 7.54 (t, *J* = 7.7 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.33 (dd, *J* = 8.0, 4.3 Hz, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 154.3, 153.1, 146.5, 142.4, 134.4, 133.9, 132.2, 132.1, 131.9, 127.7, 127.6, 126.8, 126.8, 125.3, 125.3, 124.2, 122.1, 120.4, 120.2, 117.3, 115.5, 114.9, 111.2. IR (KBr)$\nu_{max}$: 2925, 1575, 1251 $cm^{-1}$; HRMS (ESI) Calcd for $C_{23}H_{14}NO$ 320.1070 (M + $H^+$); Found 320.1069.

**8-methylbenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5b).** Yield 65% (43 mg), solid, light green, mp 220–222 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.51 (d, *J* = 8.2 Hz, 1H), 8.85 (d, *J* = 7.8 Hz, 1H), 8.32 (d, *J* = 8.8 Hz, 1H), 7.92 (d, *J* = 4.6 Hz, 2H), 7.87 (d, *J* = 8.8 Hz, 1H), 7.73 (t, *J* = 7.5 Hz, 1H), 7.66 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.7 Hz, 1H), 7.35 (t, *J* = 7.5 Hz, 1H), 7.28 (d, *J* = 8.3 Hz, 1H), 7.11 (s, 1H), 2.62 (s, 3H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 154.3, 152.9, 146.3, 142.8, 142.6, 134.3, 133.9, 132.3, 131.9, 127.7, 127.5, 126.6, 126.5, 125.3, 125.3, 124.0, 122.1, 120.4, 119.9, 117.2, 114.9, 113.7, 112.4, 22.8. IR (KBr)$\nu_{max}$: 2930, 1580, 1258 $cm^{-1}$; HRMS (ESI) Calcd for $C_{24}H_{16}NO$ 334.1227 (M + $H^+$); Found 334.1229.

**9-methylbenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5c).** Yield 67% (45 mg), solid, light green, mp 196–198 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.54 (d, *J* = 8.2 Hz, 1H), 8.91 (d, *J* = 7.8 Hz, 1H), 8.38 (d, *J* = 8.9 Hz, 1H), 8.12 (d, *J* = 8.3 Hz, 1H), 7.94 (d, *J* = 7.9 Hz, 1H), 7.91

(d, *J* = 8.9 Hz, 1H), 7.75 (t, *J* = 7.1 Hz, 1H), 7.71 (d, *J* = 8.3 Hz, 1H), 7.67 (t, *J* = 7.4 Hz, 1H), 7.54 (t, *J* = 8.5 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 2H), 2.58 (s, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 154.4, 150.3, 146.4, 141.7, 134.2, 133.7, 132.5, 132.2, 132.0, 127.7, 127.3, 126.7, 125.3, 125.2, 124.1, 122.1, 120.6, 120.3, 117.3, 115.4, 114.3, 29.8. IR (KBr)$\nu_{max}$: 2924, 1550, 1259 $cm^{-1}$; HRMS (ESI) Calcd for $C_{24}H_{16}NO$ 334.1227 ($M + H^+$); Found 334.1221.

**9-ethylbenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5d).** Yield 68% (47 mg), solid, light green, mp 214–216 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.54 (d, *J* = 8.3 Hz, 1H), 8.91 (d, *J* = 7.7 Hz, 1H), 8.39 (d, *J* = 8.9 Hz, 1H), 8.16 (d, *J* = 8.3 Hz, 1H), 7.94 (d, *J* = 7.9 Hz, 1H), 7.91 (d, *J* = 8.8 Hz, 1H), 7.75 (d, *J* = 8.3 Hz, 2H), 7.66 (t, *J* = 7.4 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.37 (t, *J* = 9.0 Hz, 2H), 3.01 (q, *J* = 7.6 Hz, 2H), 1.40 (t, *J* = 7.6 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ 154.4, 149.9, 146.5, 141.7, 133.7, 132.7, 132.5, 132.2, 132.0, 127.7, 127.3, 126.7, 126.7, 126.6, 125.3, 125.1, 124.1, 122.1, 120.4, 120.3, 117.3, 115.5, 114.6, 23.0, 14.3. IR (KBr)$\nu_{max}$: 2918, 1560, 1258 $cm^{-1}$; HRMS (ESI) Calcd for $C_{25}H_{18}NO$ 348.1383 ($M + H^+$); Found 348.1396.

**9-(*tert*-butyl)benzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5e).** Yield 72% (54 mg), solid, light green, mp 220–222°C; $^1$H NMR (600 MHz, Chloroform-*d*) δ 9.54 (d, *J* = 8.2 Hz, 1H), 8.92 (d, *J* = 8.1 Hz, 1H), 8.40 (d, *J* = 8.9 Hz, 1H), 8.15 (d, *J* = 8.7 Hz, 1H), 7.94 (d, *J* = 7.9 Hz, 1H), 7.91 (d, *J* = 8.5 Hz, 2H), 7.75 (t, *J* = 7.5 Hz, 1H), 7.67 (t, *J* = 6.8 Hz, 1H), 7.56 – 7.54 (m, 1H), 7.39 (t, *J* = 7.3 Hz, 2H), 1.62 (s, 9H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ 153.9, 150.7, 146.5, 141.5, 133.7, 132.6, 132.4, 132.1, 131.9, 130.2, 127.7, 127.3, 126.7, 126.6, 125.3, 125.1, 124.1, 121.9, 120.3, 120.1, 117.1, 116.0, 114.4, 35.1, 29.8. IR (KBr)$\nu_{max}$: 2920, 1540, 1248 $cm^{-1}$; HRMS (ESI) Calcd for $C_{27}H_{22}NO$ 376.1696 ($M + H^+$); Found 376.1705.

**13-chloro-8-methylbenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5f).** Yield 70% (51 mg), solid, light green, mp 208–210 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.49 (d, *J* = 8.3 Hz, 1H), 8.78 (d, *J* = 2.6 Hz, 1H), 8.33 (d, *J* = 8.9 Hz, 1H), 7.96 – 7.93 (m, 2H), 7.90 (d, *J* = 8.9 Hz, 1H), 7.76 (ddd, *J* = 8.2, 7.0, 1.2 Hz, 1H), 7.70 – 7.66 (m, 1H), 7.44 (dd, *J* = 8.7, 2.6 Hz, 1H), 7.22 (d, *J* = 8.8 Hz, 1H), 7.12 (s, 1H), 2.64 (s, 3H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 152.7, 152.6, 145.1, 143.1, 142.5, 134.3, 133.9, 132.2, 131.8, 129.6, 127.7, 127.7, 127.0, 126.9, 125.3, 124.7, 123.4, 120.3, 120.2, 118.8, 115.2, 113.6, 112.5, 22.9. IR (KBr)$\nu_{max}$: 2927, 1557, 1241 $cm^{-1}$; HRMS (ESI) Calcd for $C_{24}H_{15}ClO$ 368.0837 ($M + H^+$); Found 368.0836.

**13-chloro-9-methylbenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5g).** Yield 71% (52 mg), solid, light green, mp 204–206 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.39 (d, *J* = 8.2 Hz, 1H), 8.66 (s, 1H), 8.23 (d, *J* = 8.8 Hz, 1H), 7.95 (d, *J* = 8.2 Hz, 1H), 7.90 (d, *J* = 7.7 Hz, 1H), 7.84 (d, *J* = 8.8 Hz, 1H), 7.75 – 7.72 (m, 1H), 7.66 (td, *J* = 7.5, 6.9, 1.2 Hz, 1H), 7.56 (d, *J* = 8.2 Hz, 1H), 7.39 (dd, *J* = 8.7, 2.6 Hz, 1H), 7.16 (d, *J* = 8.7 Hz, 1H), 2.45 (s, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ 152.6, 149.7, 144.9, 141.3, 134.1, 133.5, 132.2, 132.0, 131.7, 129.4, 127.7, 127.4, 126.9, 126.7, 125.0, 124.6, 123.1, 120.6, 120.4, 120.1, 118.7, 114.9, 114.4, 15.6. IR (KBr)$\nu_{max}$: 2927, 1570, 1260 cm$^{-1}$; HRMS (ESI) Calcd for $C_{24}H_{15}ClNO$ 368.0837 (M + H$^+$); Found 368.0835.

**13-chloro-9-ethylbenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5h).** Yield 71% (53 mg), solid, light green, mp 164–166 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.32 (d, *J* = 8.2 Hz, 1H), 8.58 (d, *J* = 2.6 Hz, 1H), 8.17 (d, *J* = 8.9 Hz, 1H), 7.91 (d, *J* = 8.4 Hz, 1H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.79 (d, *J* = 8.8 Hz, 1H), 7.72 – 7.69 (m, 1H), 7.65 – 7.62 (m, 1H), 7.55 (d, *J* = 8.3 Hz, 1H), 7.35 (dd, *J* = 8.7, 2.6 Hz, 1H), 7.11 (d, *J* = 8.7 Hz, 1H), 2.83 (t, *J* = 7.6 Hz, 2H), 1.34 (t, *J* = 7.6 Hz, 3H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 152.6, 149.3, 144.9, 141.4, 133.6, 132.4, 132.2, 132.0, 131.5, 129.4, 127.6, 127.3, 126.9, 126.7, 126.5, 125.1, 124.6, 123.2, 120.3, 120.0, 118.6, 114.9, 114.6, 22.7, 14.1. IR (KBr)$\nu_{max}$: 2927, 1575, 1248 cm$^{-1}$; HRMS (ESI) Calcd for $C_{25}H_{17}ClNO$ 382.0994 (M + H$^+$); Found 382.0993.

**13-chloro-9-*iso*propylbenzo[c]chromeno[4,3,2-*gh*]phenanthridine (5i).** Yield 73% (58 mg), solid, light green, mp 214–216 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.47 (d, *J* = 8.2 Hz, 1H), 8.78 (d, *J* = 2.4 Hz, 1H), 8.33 (d, *J* = 8.9 Hz, 1H), 8.15 (d, *J* = 8.5 Hz, 1H), 7.92 (d, *J* = 7.9 Hz, 1H), 7.89 (d, *J* = 8.9 Hz, 1H), 7.80 (d, *J* = 8.5 Hz, 1H), 7.76 (d, *J* = 7.5 Hz, 1H), 7.67 (t, *J* = 7.4 Hz, 1H), 7.44 (dd, *J* = 8.7, 2.4 Hz, 1H), 7.25 (s, 1H), 3.68 (p, *J* = 6.9 Hz, 1H), 1.42 (d, *J* = 6.9 Hz, 6H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 152.8, 148.9, 145.3, 141.6, 133.7, 132.3, 132.1, 131.8, 131.1, 129.9, 129.6, 127.7, 127.5, 127.1, 126.8, 125.2, 124.8, 123.4, 120.5, 120.2, 118.8, 115.3, 115.1, 27.0, 22.7. IR (KBr)$\nu_{max}$: 2918, 1570, 1245 cm$^{-1}$; HRMS (ESI) Calcd for $C_{26}H_{19}ClNO$ 396.1150 (M + H$^+$); Found 396.1145.

**9-(*tert*-butyl)-13-chlorobenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5j).** Yield 75% (61 mg), solid, light green, mp 245–247 °C; $^1$H NMR (600 MHz, Chloroform-*d*) δ 9.43 (d, *J* = 8.2 Hz, 1H), 8.75 (d, *J* = 2.4 Hz, 1H), 8.29 (d, *J* = 8.8 Hz, 1H), 8.05 (d, *J* = 8.6 Hz, 1H), 7.90 (d, *J* = 7.9 Hz, 1H), 7.85 (dd, *J* = 12.3, 8.7 Hz, 2H), 7.74 (t, *J* = 7.4 Hz, 1H), 7.65 (t, *J* = 7.3 Hz, 1H), 7.44

(d, *J* = 8.7 Hz, 1H), 7.28 (d, *J* = 8.7 Hz, 1H), 1.61 (s, 9H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ 152.3, 150.3, 145.1, 141.3, 133.6, 132.5, 132.3, 131.9, 131.7, 130.3, 129.5, 127.7, 127.4, 127.0, 126.8, 125.1, 124.7, 123.1, 120.2, 120.1, 118.5, 115.6, 114.6, 35.0, 29.9. IR (KBr)$\nu_{max}$: 2929, 1550, 1251 $cm^{-1}$; HRMS (ESI) Calcd for $C_{27}H_{21}ClNO$ 410.1307 ($M + H^+$); Found 410.1312.

**13-chloro-9-phenylbenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5k).** Yield 75% (64 mg), solid, light green, mp 246–248 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.49 (d, *J* = 8.2 Hz, 1H), 8.79 (d, *J* = 2.5 Hz, 1H), 8.37 (d, *J* = 8.9 Hz, 1H), 8.23 (s, 1H), 7.93 (d, *J* = 9.0 Hz, 2H), 7.90 (d, *J* = 8.5 Hz, 1H), 7.79 – 7.75 (m, 3H), 7.69 (t, *J* = 7.0 Hz, 1H), 7.55 (t, *J* = 7.5 Hz, 2H), 7.46 (t, *J* = 7.5 Hz, 1H), 7.42 (dd, *J* = 8.7, 2.6 Hz, 1H), 7.16 (d, *J* = 8.8 Hz, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 152.4, 148.9, 136.8, 133.8, 133.8, 133.6, 132.0, 131.9, 129.8, 129.6, 128.6, 127.8, 127.3, 127.0, 125.2, 124.7, 124.5, 123.3, 120.3, 120.2, 119.0, 115.5, 115.4. IR (KBr)$\nu_{max}$: 2927, 1528, 1260 $cm^{-1}$; HRMS (ESI) Calcd for $C_{29}H_{17}ClNO$ 430.0994 ($M + H^+$); Found 430.0996.

**13-bromo-8-methylbenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5l).** Yield 69% (57 mg), solid, light green, mp 243–245 °C; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.46 (d, *J* = 8.2 Hz, 1H), 8.89 (d, *J* = 2.3 Hz, 1H), 8.30 (d, *J* = 8.9 Hz, 1H), 7.92 (s, 1H), 7.87 (s, 1H), 7.76 (t, *J* = 7.4 Hz, 2H), 7.68 (t, *J* = 7.5 Hz, 1H), 7.58 – 7.55 (m, 1H), 7.13 (d, *J* = 8.8 Hz, 1H), 7.09 (s, 1H), 2.62 (s, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ 153.0, 152.4, 144.8, 143.0, 142.2, 134.5, 134.1, 133.7, 132.0, 127.7, 127.7, 127.6, 126.9, 126.8, 125.2, 123.7, 120.2, 120.0, 119.0, 116.9, 115.2, 113.4, 112.5, 22.9. IR (KBr)$\nu_{max}$: 2937, 1520, 1260 $cm^{-1}$; HRMS (ESI) Calcd for $C_{24}H_{15}BrNO$ 412.0332 ($M + H^+$); Found 412.0324 and 414.0493.

**13-bromo-9-methylbenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5m).** Yield 70% (58 mg), solid, light green, mp 244–246 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.32 (d, *J* = 8.2 Hz, 1H), 8.72 (d, *J* = 2.4 Hz, 1H), 8.15 (d, *J* = 8.8 Hz, 1H), 7.87 (t, *J* = 7.4 Hz, 2H), 7.79 (d, *J* = 8.8 Hz, 1H), 7.73 – 7.70 (m, 1H), 7.66 – 7.63 (m, 1H), 7.49 (d, *J* = 8.6 Hz, 2H), 7.04 (d, *J* = 8.7 Hz, 1H), 2.40 (s, 3H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 153.1, 149.7, 144.6, 141.4, 134.4, 134.1, 133.6, 132.2, 132.0, 127.6, 127.4, 126.9, 126.7, 125.1, 123.6, 120.5, 120.4, 120.0, 119.0, 116.9, 114.9, 114.4, 15.5. IR (KBr)$\nu_{max}$: 2930, 1570, 1250 $cm^{-1}$; HRMS (ESI) Calcd for $C_{24}H_{15}BrNO$ 412.0332 ($M + H^+$); Found 412.0332 and 414.0371.

**13-bromo-9-ethylbenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5n).** Yield 70% (60 mg), solid, light green, mp 247–249 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.46 (d, *J* = 8.3 Hz, 1H),

8.91 (s, 1H), 8.32 (d, *J* = 8.9 Hz, 1H), 8.10 (d, *J* = 8.3 Hz, 1H), 7.93 (d, *J* = 7.9 Hz, 1H), 7.89 (d, *J* = 8.9 Hz, 1H), 7.76 (t, *J* = 7.6 Hz, 1H), 7.70 – 7.66 (m, 2H), 7.58 (d, *J* = 8.7 Hz, 1H), 7.18 (d, *J* = 8.7 Hz, 1H), 2.95 (q, *J* = 7.6 Hz, 2H), 1.37 (t, *J* = 7.6 Hz, 3H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 153.3, 149.5, 145.0, 141.6, 134.6, 133.7, 132.8, 132.4, 132.1, 127.8, 127.7, 127.5, 127.1, 126.8, 126.8, 125.2, 123.8, 120.5, 120.1, 119.1, 117.0, 115.2, 114.9, 22.8, 14.3. IR (KBr)$\nu_{max}$: 2929, 1570, 1240 cm$^{-1}$; HRMS (ESI) Calcd for $C_{25}H_{17}BrNO$ 426.0489 (M + H$^+$); Found 426.0489 and 428.0471.

**13-bromo-9-(*tert*-butyl)benzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5o).** Yield 72% (65 mg), solid, light green, mp 250 °C; $^1$H NMR (600 MHz, Chloroform-*d*) δ 9.45 (d, *J* = 8.2 Hz, 1H), 8.92 (s, 1H), 8.31 (d, *J* = 8.8 Hz, 1H), 8.08 (d, *J* = 8.5 Hz, 1H), 7.91 (d, *J* = 7.9 Hz, 1H), 7.87 (dd, *J* = 13.2, 8.7 Hz, 2H), 7.75 (t, *J* = 7.5 Hz, 1H), 7.66 (t, *J* = 7.3 Hz, 1H), 7.59 (d, *J* = 8.6 Hz, 1H), 7.23 (d, *J* = 8.6 Hz, 1H), 1.61 (s, 9H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ 152.8, 150.3, 145.0, 141.3, 134.6, 133.7, 132.5, 132.4, 131.9, 130.4, 127.8, 127.7, 127.5, 127.0, 126.8, 125.1, 123.6, 120.2, 120.1, 118.9, 117.0, 115.7, 114.7, 35.0, 29.9. IR (KBr)$\nu_{max}$: 2921, 1550, 1260 cm$^{-1}$; HRMS (ESI) Calcd for $C_{27}H_{21}BrNO$ 454.0802 (M + H$^+$); Found 454.0807.

**9-(*tert*-butyl)-11,13-dichlorobenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5p).** Yield 75% (66 mg), solid, light green, mp 254–256 °C; $^1$H NMR (600 MHz, Chloroform-*d*) δ 9.43 (d, *J* = 8.1 Hz, 1H), 8.70 (s, 1H), 8.33 (d, *J* = 8.8 Hz, 1H), 8.14 (d, *J* = 8.6 Hz, 1H), 7.92 (t, *J* = 8.7 Hz, 3H), 7.76 (t, *J* = 7.3 Hz, 1H), 7.67 (t, *J* = 7.2 Hz, 1H), 7.56 (s, 1H), 1.66 (s, 9H). $^{13}$C NMR (150 MHz, $CDCl_3$) δ 149.7, 148.7, 144.4, 141.3, 133.7, 133.1, 132.5, 131.9, 131.7, 130.6, 129.2, 127.7, 127.6, 127.4, 126.9, 125.0, 124.3, 123.3, 123.1, 120.4, 120.1, 115.5, 115.3, 35.1, 29.5. IR (KBr)$\nu_{max}$: 2919, 1540, 1250 cm$^{-1}$; HRMS (ESI) Calcd for $C_{27}H_{20}Cl_2NO$ 444.0917 (M + H$^+$); Found 444.1122.

**9-ethyl-13-methoxybenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5q).** Yield 68% (51 mg), solid, light green, mp 224 °C; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.50 (d, *J* = 8.2 Hz, 1H), 8.79 (d, *J* = 8.8 Hz, 1H), 8.36 (d, *J* = 8.9 Hz, 1H), 8.12 (d, *J* = 8.4 Hz, 1H), 7.93 (d, *J* = 7.9 Hz, 1H), 7.87 (d, *J* = 8.9 Hz, 1H), 7.72 (dd, *J* = 13.8, 8.1 Hz, 2H), 7.65 (t, *J* = 7.9 Hz, 1H), 6.95 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.83 (d, *J* = 2.4 Hz, 1H), 3.95 (s, 3H), 2.99 (q, *J* = 7.6 Hz, 2H), 1.40 (t, *J* = 7.6 Hz, 3H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 163.1, 155.7, 150.1, 146.7, 141.9, 133.8, 132.4, 132.2, 127.7, 127.2, 126.6, 126.6, 126.5, 126.1, 125.2, 120.3, 119.8, 115.4, 115.1, 114.7, 112.3,

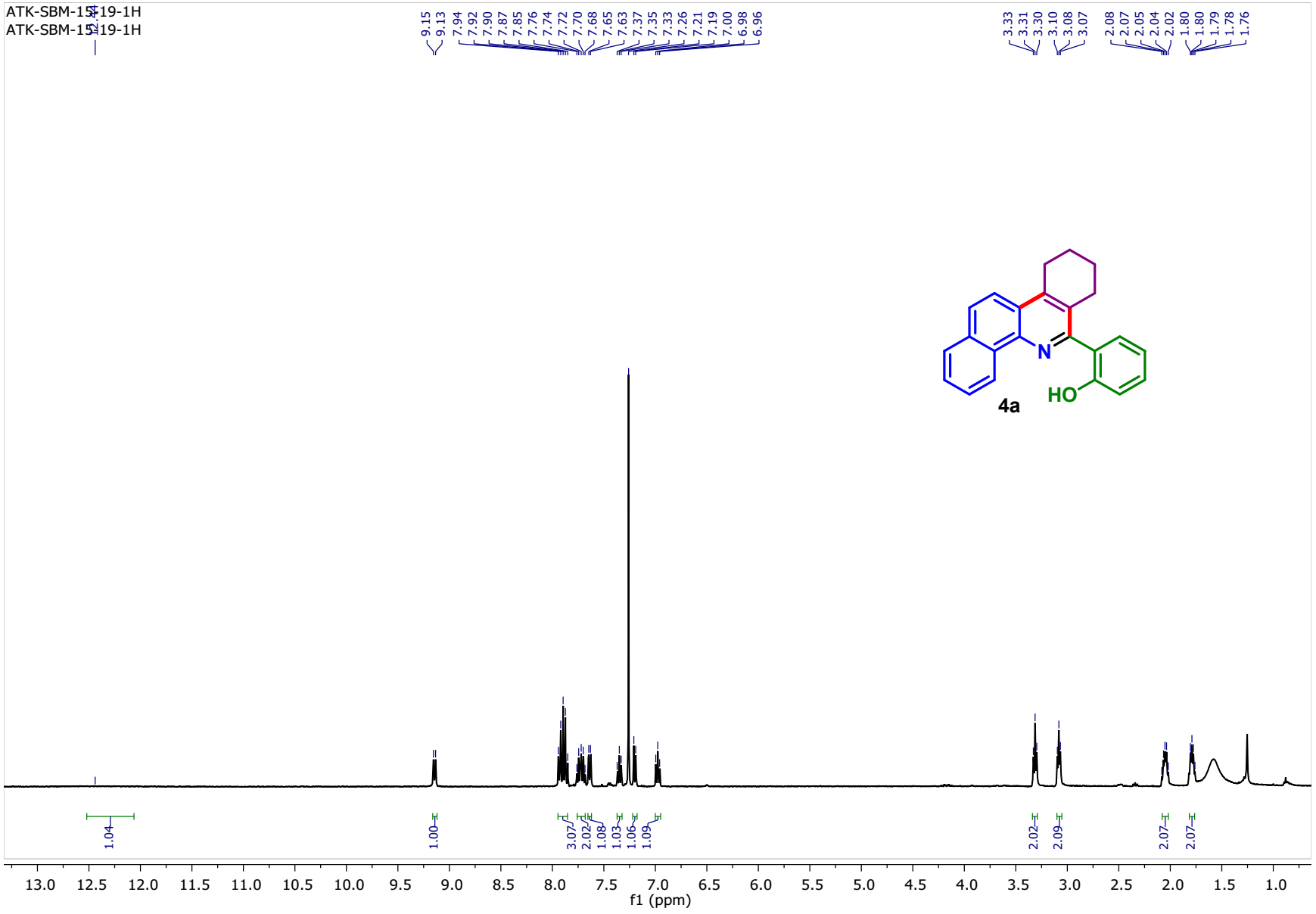
100.9, 55.8, 22.9, 14.3. IR (KBr)$\nu_{max}$: 2927, 1574, 1248 $cm^{-1}$; HRMS (ESI) Calcd for $C_{26}H_{20}NO_2$ 378.1489 ($M + H^+$); Found 378.1490.

**9-(*tert*-butyl)-13-methoxybenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5r).** Yield 67% (54 mg), solid, light green, mp 210–212 °C; $^1H$ NMR (600 MHz, Chloroform-*d*) δ 9.50 (d, *J* = 8.1 Hz, 1H), 8.81 (d, *J* = 8.7 Hz, 1H), 8.36 (d, *J* = 8.8 Hz, 1H), 8.12 (d, *J* = 8.5 Hz, 1H), 7.93 (d, *J* = 7.8 Hz, 1H), 7.87 (d, *J* = 8.6 Hz, 2H), 7.73 (t, *J* = 7.3 Hz, 1H), 7.65 (t, *J* = 7.3 Hz, 1H), 6.96 (d, *J* = 8.6 Hz, 1H), 6.83 (s, 1H), 3.96 (s, 3H), 1.62 (s, 9H). $^{13}C$ NMR (150 MHz, $CDCl_3$) δ 163.0, 155.2, 150.9, 146.7, 133.7, 132.6, 132.3, 131.9, 130.0, 127.7, 127.3, 126.6, 126.5, 126.0, 125.1, 120.3, 119.5, 115.5, 115.1, 114.5, 114.2, 112.3, 100.6, 55.8, 35.0, 29.9. IR (KBr)$\nu_{max}$: 2920, 1545, 1260 $cm^{-1}$; HRMS (ESI) Calcd for $C_{28}H_{24}NO_2$ 406.1802 ($M + H^+$); Found 406.1811.

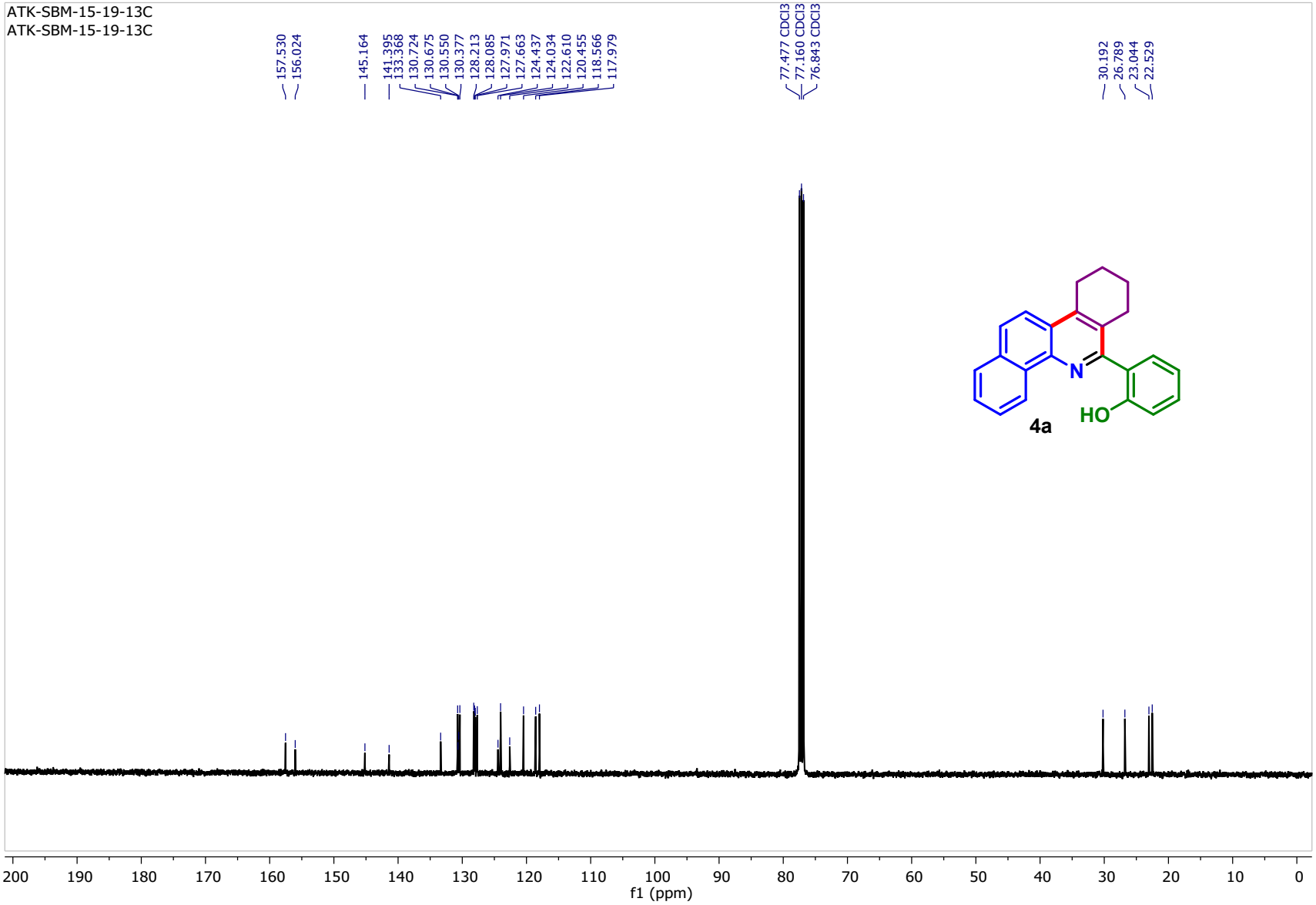
**5-bromo-9-(*tert*-butyl)benzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5s).** Yield 65% (59 mg), solid, light green, mp 228–230 °C; $^1H$ NMR (500 MHz, Chloroform-d) δ 9.55 (d, J = 9.2 Hz, 1H), 8.87 (d, J = 8.1 Hz, 1H), 8.68 (s, 1H), 8.32 (d, J = 9.0 Hz, 1H), 8.05 (d, J = 8.7 Hz, 1H), 7.90 (d, J = 8.6 Hz, 1H), 7.77 (t, J = 8.5 Hz, 2H), 7.55 (d, J = 8.4 Hz, 1H), 7.39 (d, J = 7.7 Hz, 2H), 1.63 (s, 9H). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 153.8, 150.6, 146.8, 141.0, 133.0, 132.9, 132.1, 131.9, 131.4, 130.4, 128.3, 127.2, 127.1, 125.3, 125.2, 124.2, 124.1, 121.5, 121.3, 120.5, 117.0, 115.9, 114.1, 35.0, 29.7. IR (KBr)$\nu_{max}$: 2930, 1540, 1270 $cm^{-1}$; HRMS (ESI) Calcd for $C_{27}H_{21}BrNO$ 454.0802 ($M + H^+$); Found 454.0807, and 456.0789.

**5-bromo-9-(*tert*-butyl)-13-chlorobenzo[*c*]chromeno[4,3,2-*gh*]phenanthridine (5t).** Yield 64% (62 mg), solid, light green, mp 245–247 °C; $^1H$ NMR (400 MHz, Chloroform-d) δ 9.46 (d, J = 8.1 Hz, 1H), 8.80 (d, J = 2.5 Hz, 1H), 8.36 (s, 1H), 8.17 (d, J = 8.8 Hz, 1H), 7.94 (t, J = 3.9 Hz, 2H), 7.92 (s, 1H), 7.77 (s, 1H), 7.74 (d, J = 2.4 Hz, 1H), 7.69 (d, J = 7.5 Hz, 1H), 1.69 (s, 9H). $^{13}C$ NMR (100 MHz, CDCl3) δ 149.8, 141.1, 134.6, 133.6, 133.0, 132.4, 131.9, 131.8, 130.6, 129.6, 127.6, 127.5, 127.4, 126.8, 124.9, 124.0, 120.3, 120.0, 115.3, 111.4, 35.0, 29.4. IR (KBr)$\nu_{max}$: 2920, 1540, 1265 $cm^{-1}$; HRMS (ESI) Calcd for $C_{27}H_{20}BrClNO$ 488.0412 ($M + H^+$); Found 488.0411, and 490.0425.

## $^{1}$H NMR Spectra of 4a

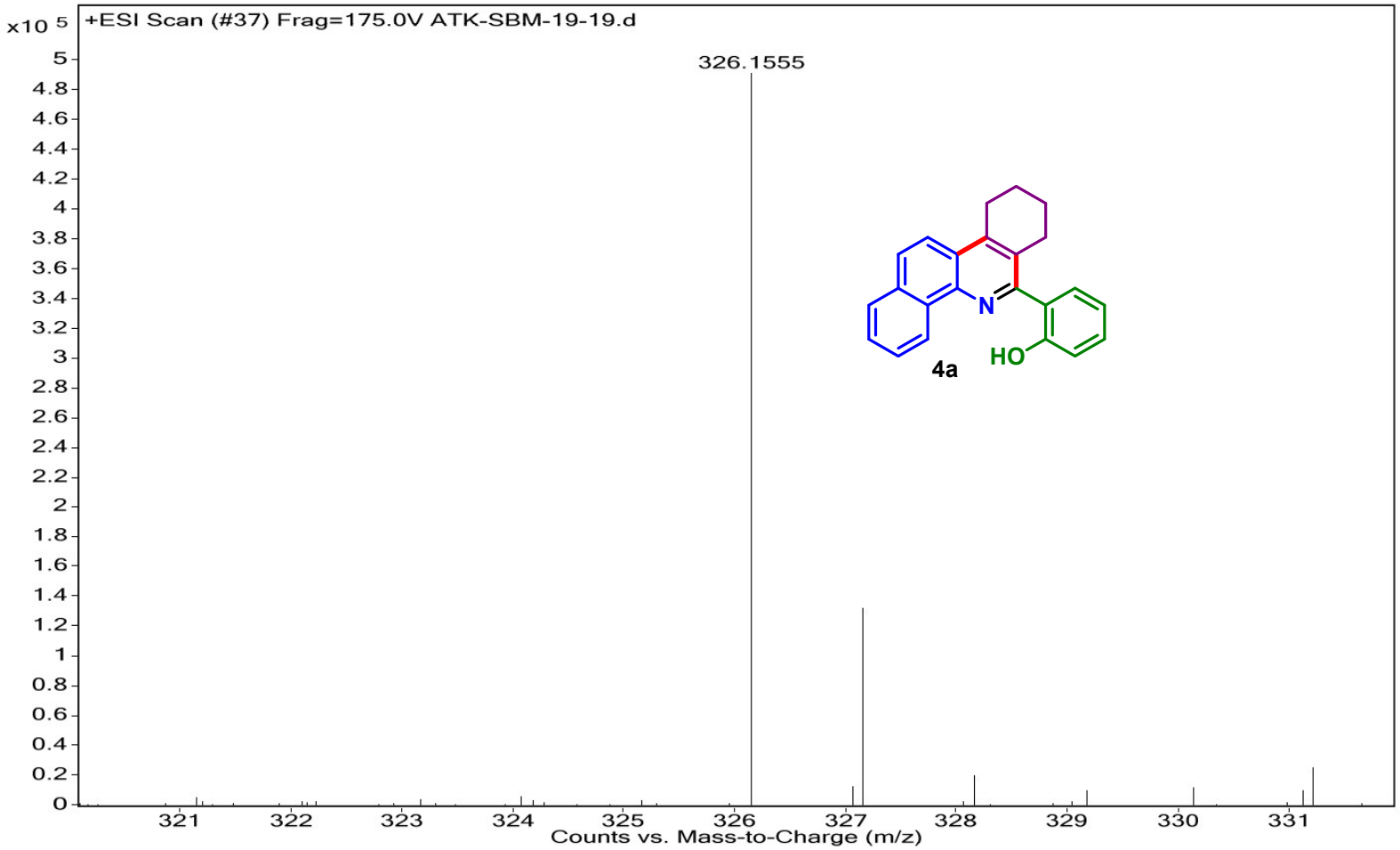


## $^{13}$C NMR Spectra of 4a

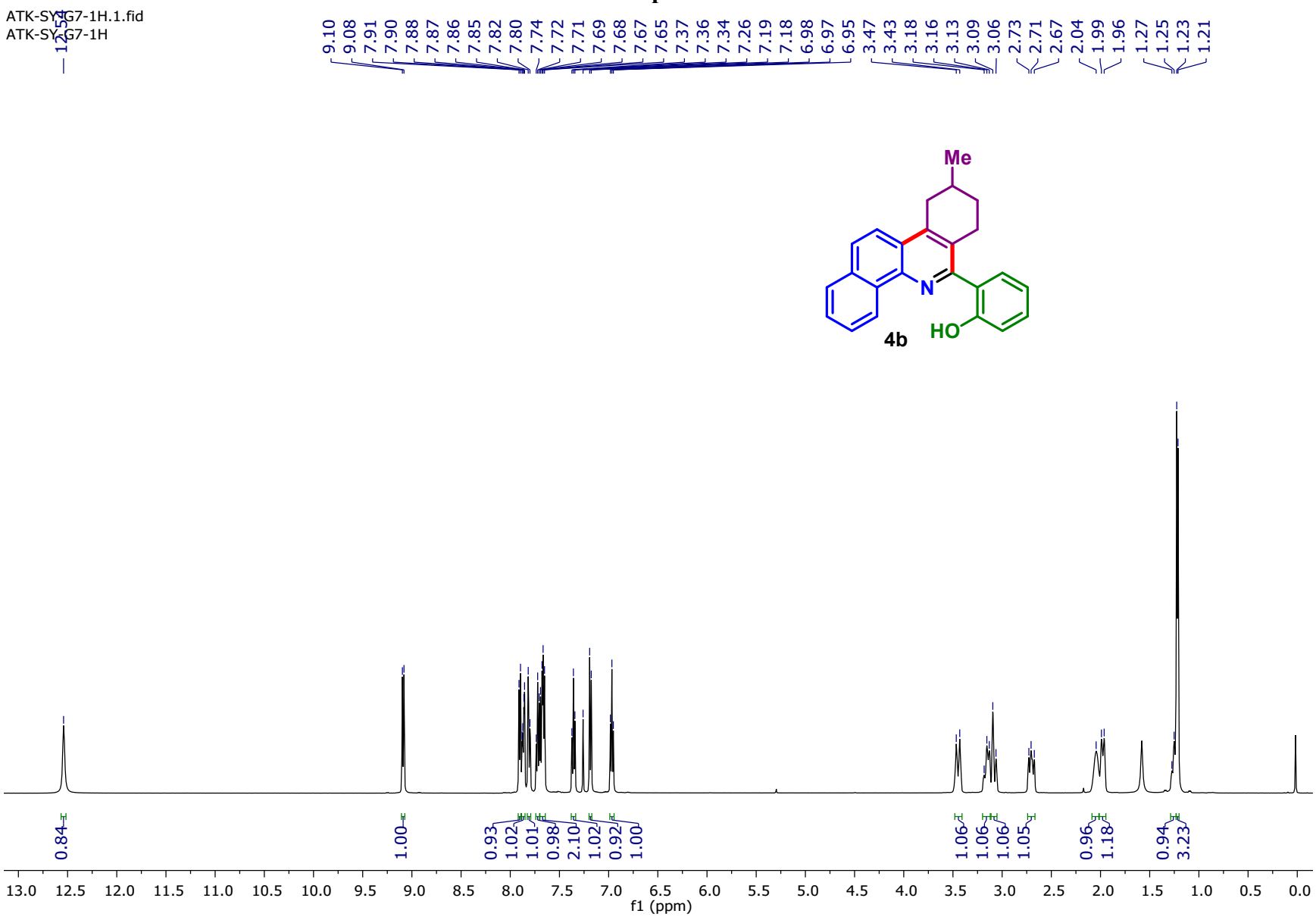


# HRMS Spectra of 4a

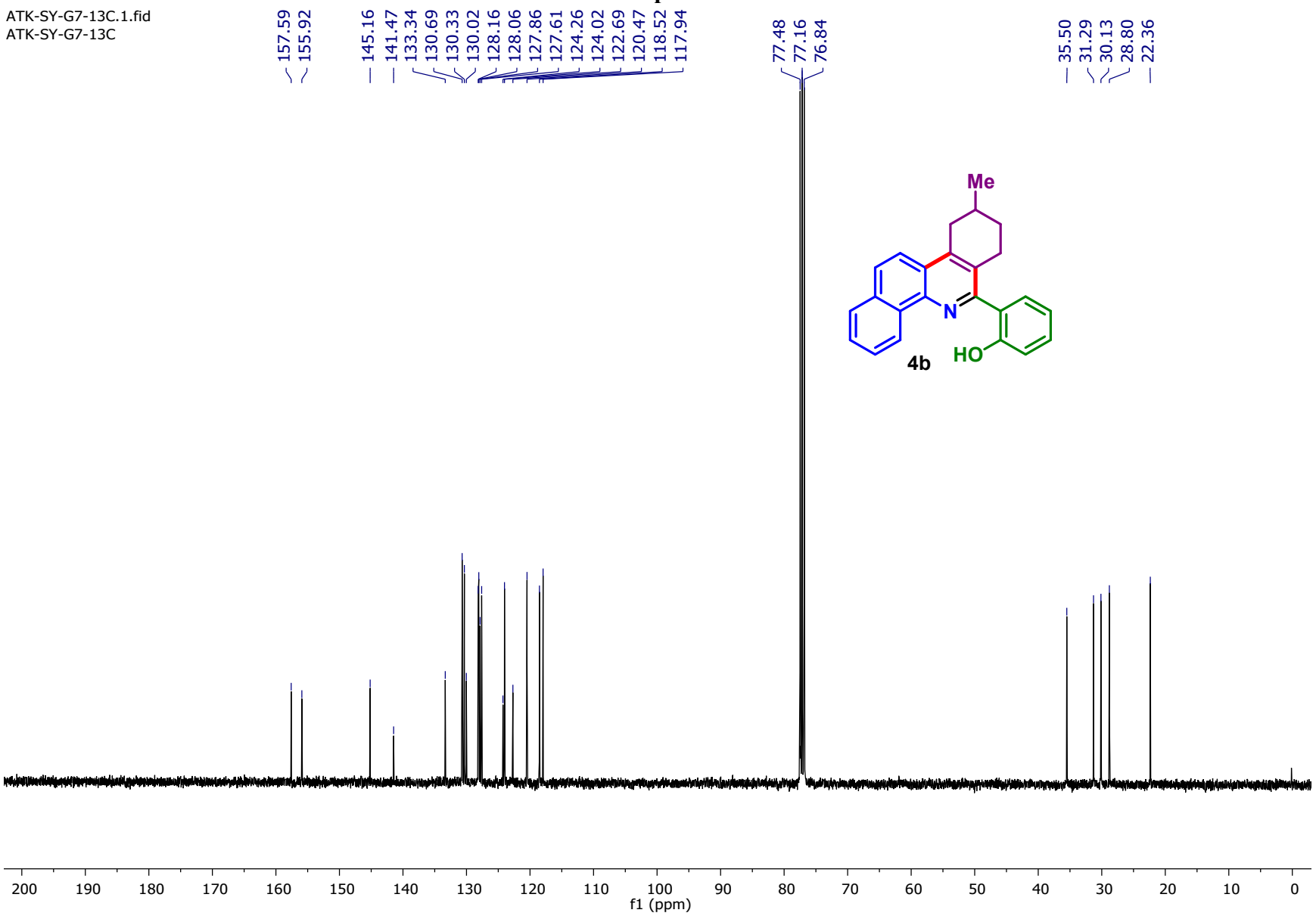
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Sample Name** | ATK-SBM-19-19 | **Position** | P1-C7 | **Instrument Name** | Instrument 1 | **User Name** | |
| **Inj Vol** | 20 | **InjPosition** | | **SampleType** | Sample | **IRM Calibration Status** | Success |
| **Data Filename** | ATK-SBM-19-19.d | **ACQ Method** | ESI ALS 100-600.m | **Comment** | | **Acquired Time** | 4/16/2019 4:42:25 PM |



## $^{1}$H NMR Spectra of 4b



## $^{13}$C NMR Spectra of 4b



# HRMS Spectra of 4b

| | | | | | |
|---|---|---|---|---|---|
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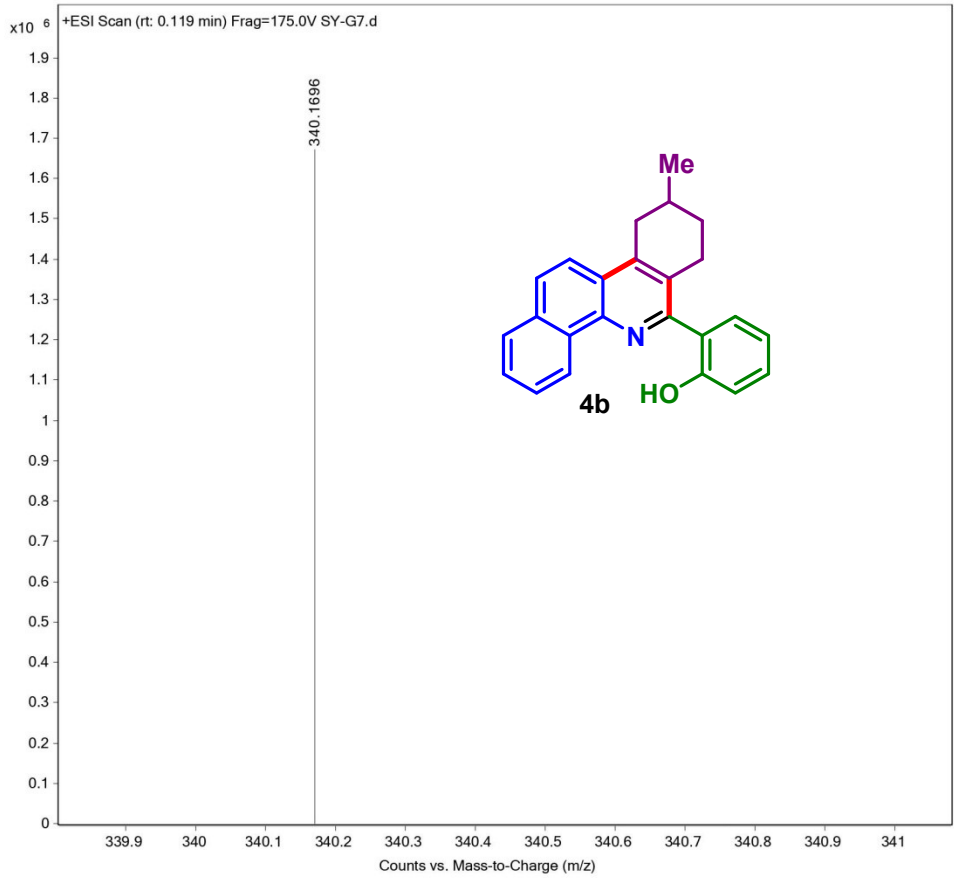
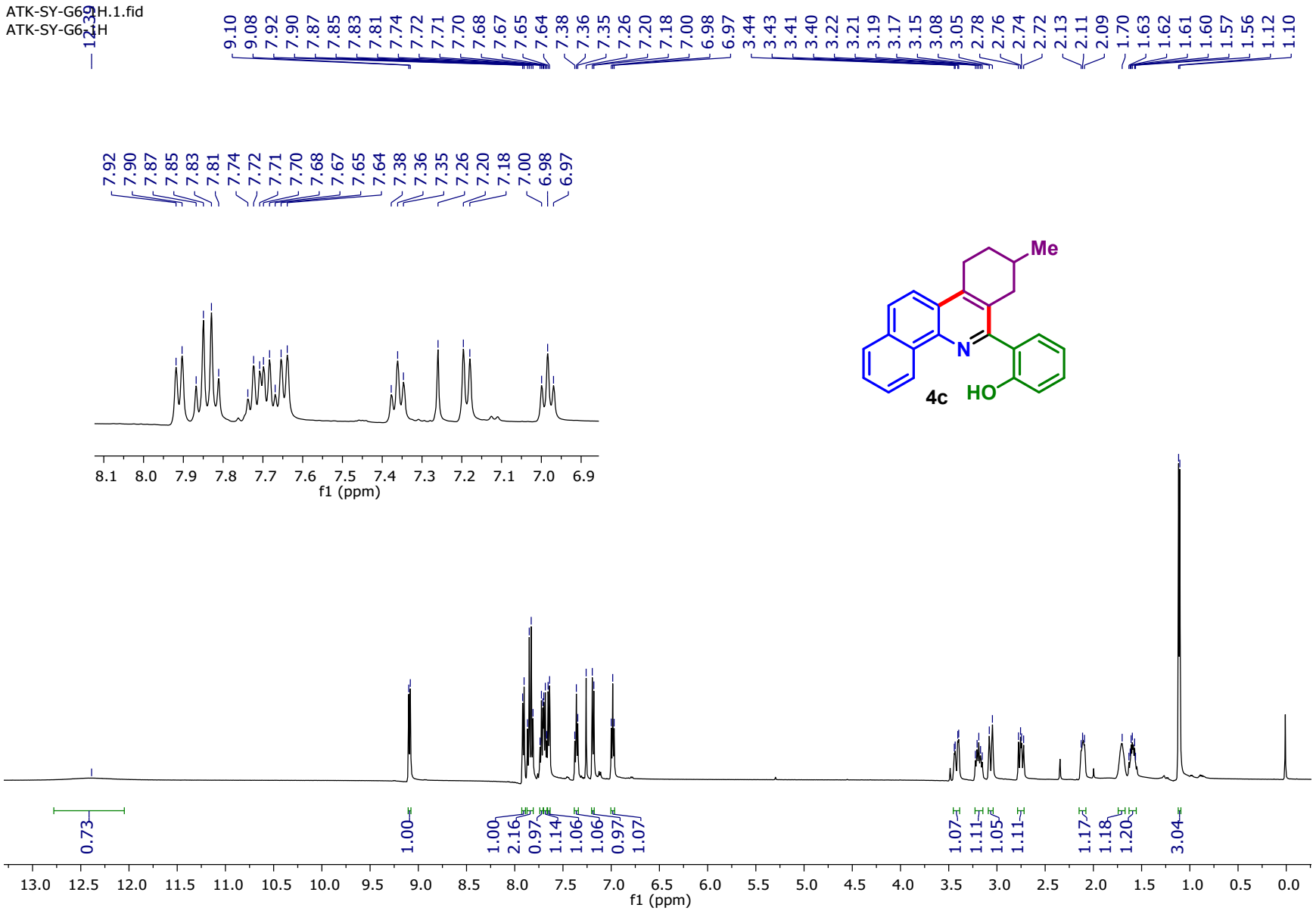
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-G7.d |
| **ACQ Method** | ESI ALS 100-500.m | **Comment** | | **Acquired Time** | 21-Apr-21 11:12:22 PM (UTC+05:30) |



## $^{1}$H NMR Spectra of 4c



## $^{13}$C NMR Spectra of 4c

ATK-SY-G6-13C.3.fid
ATK-SY-G6-13C

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130.41
130.11
128.71
128.26
128.08
128.00
127.69
124.29
124.05
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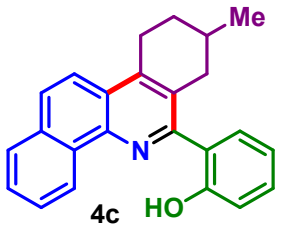
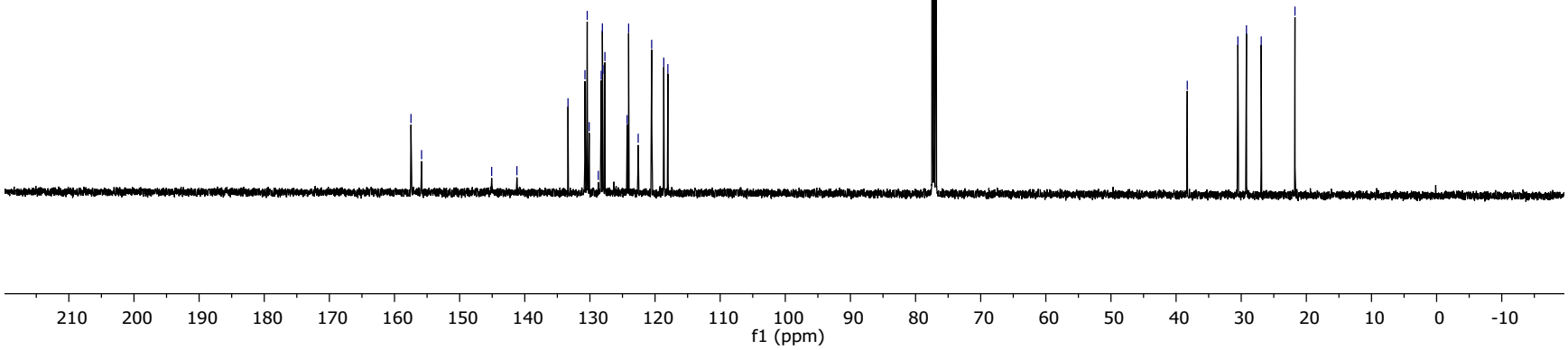
29.20
26.93
21.75




4c



# HRMS Spectra of 4c

| | | | | | |
|---|---|---|---|---|---|
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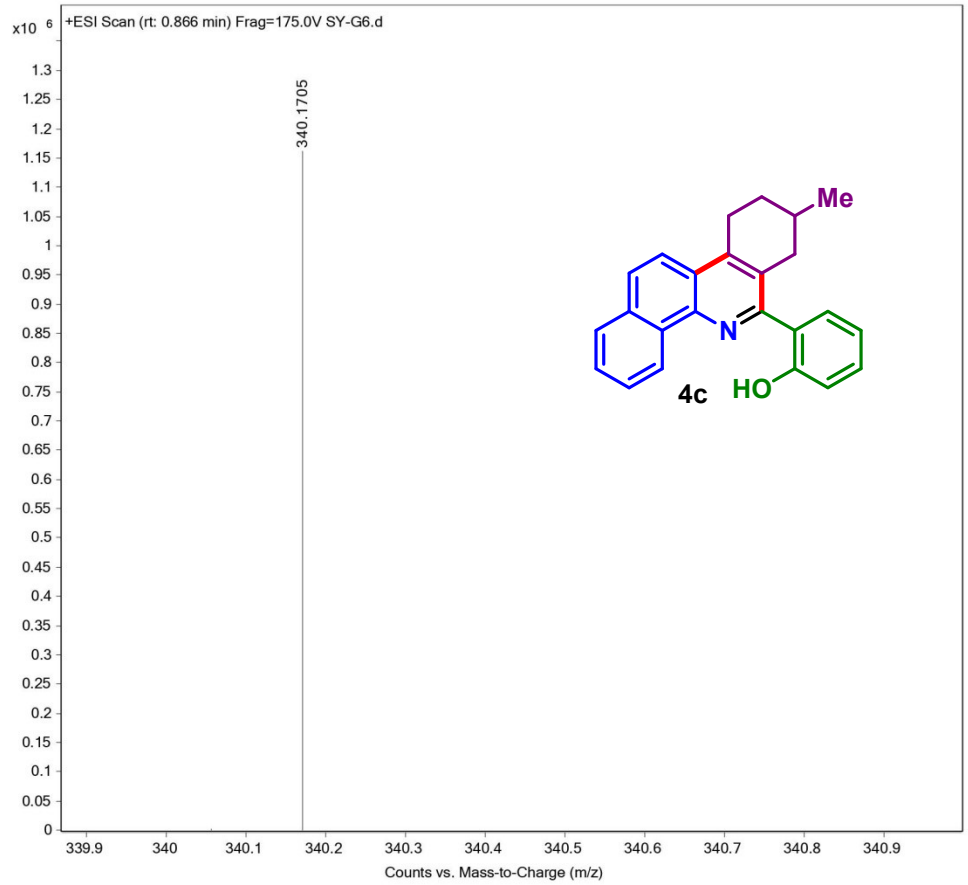
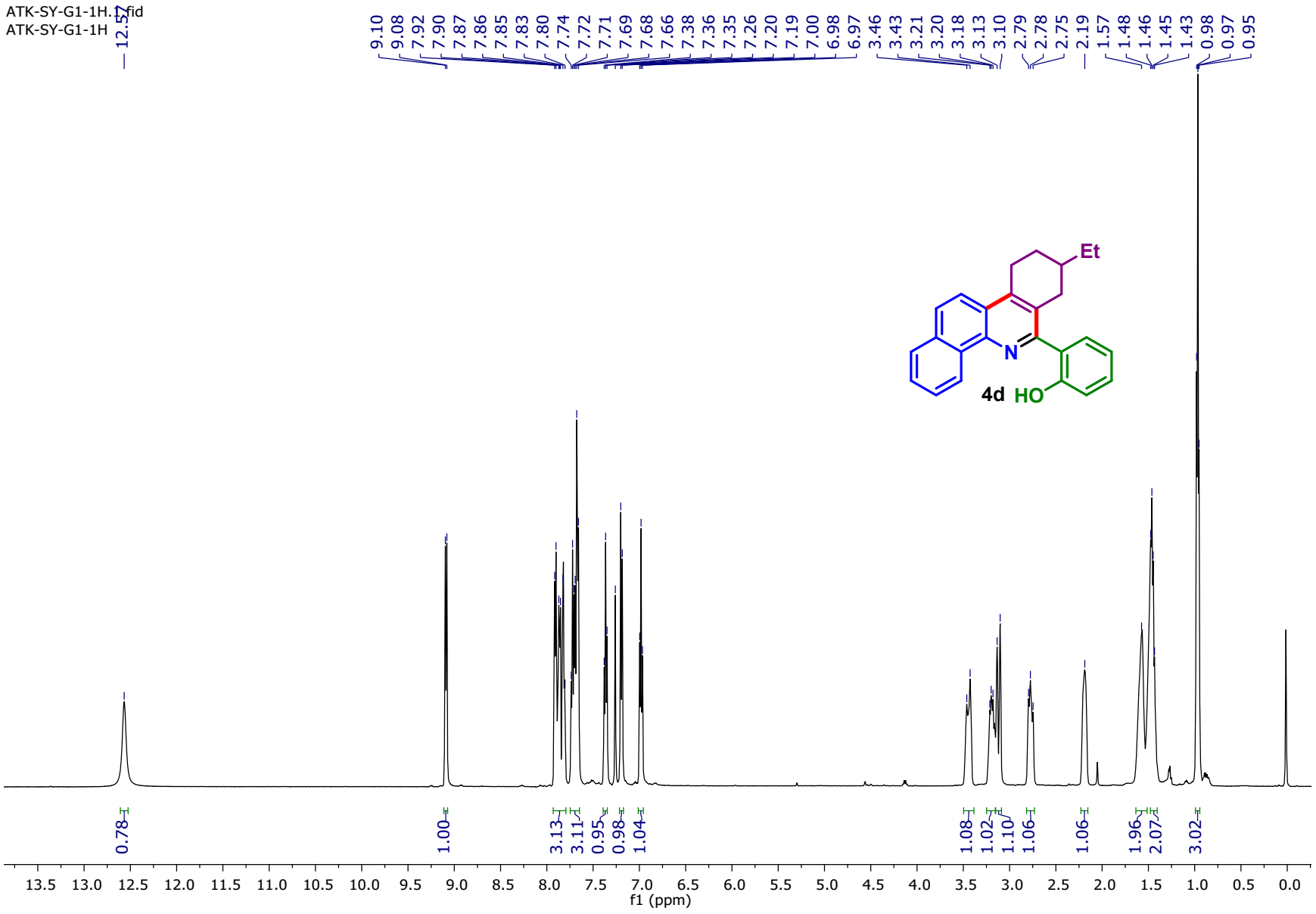
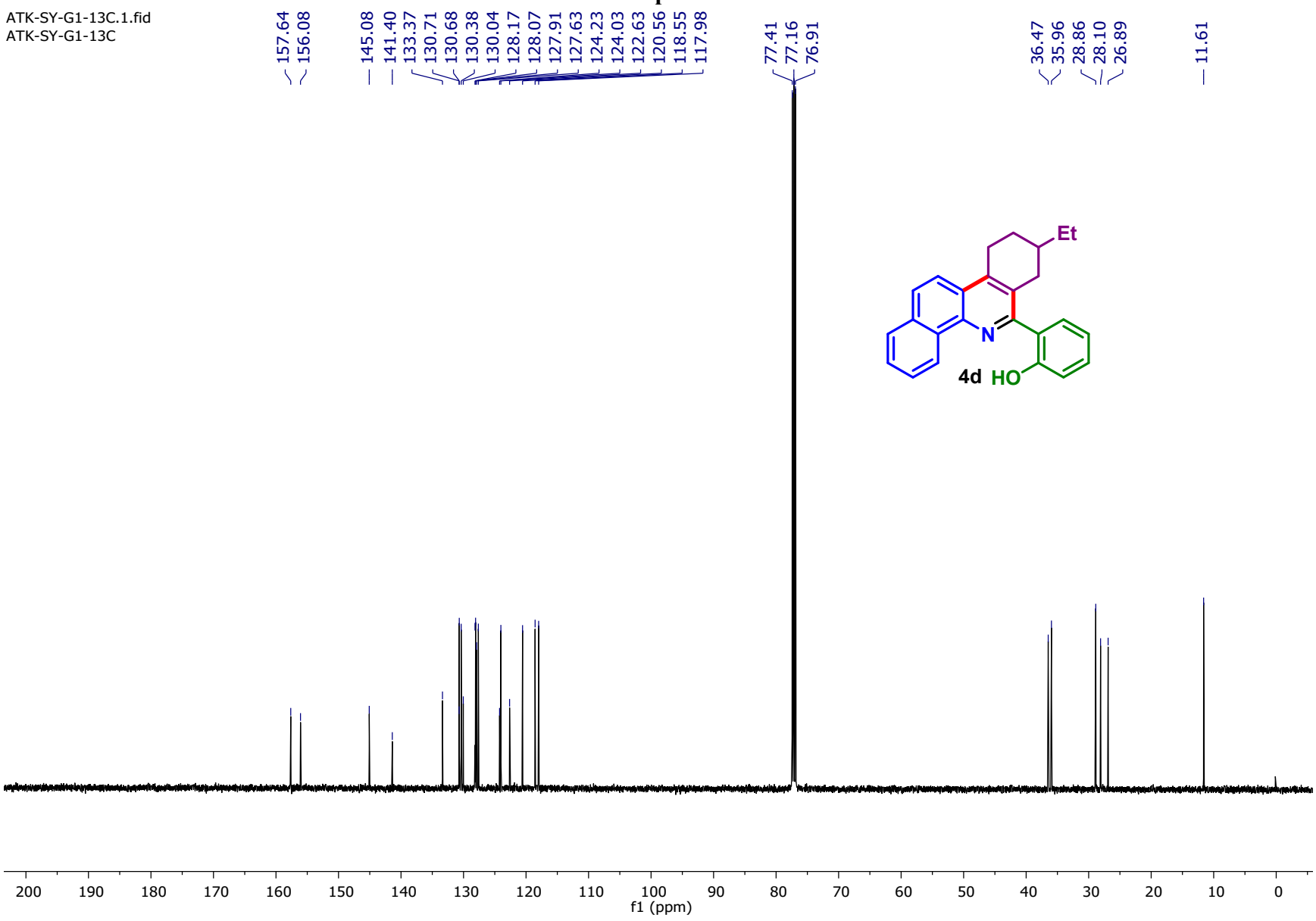
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-G6.d |
| **ACQ Method** | ESI ALS 100-500.m | **Comment** | | **Acquired Time** | 21-Apr-21 11:02:42 PM (UTC+05:30) |



## $^{1}$H NMR Spectra of 4d

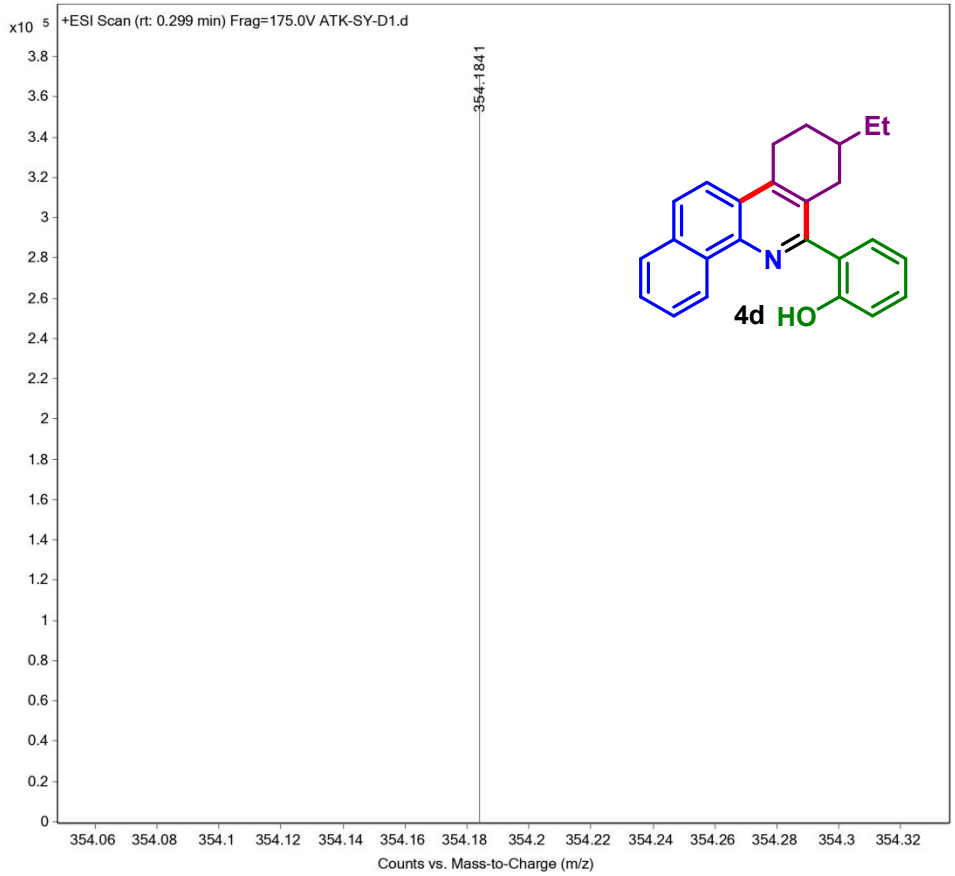


## $^{13}$C NMR Spectra of 4d

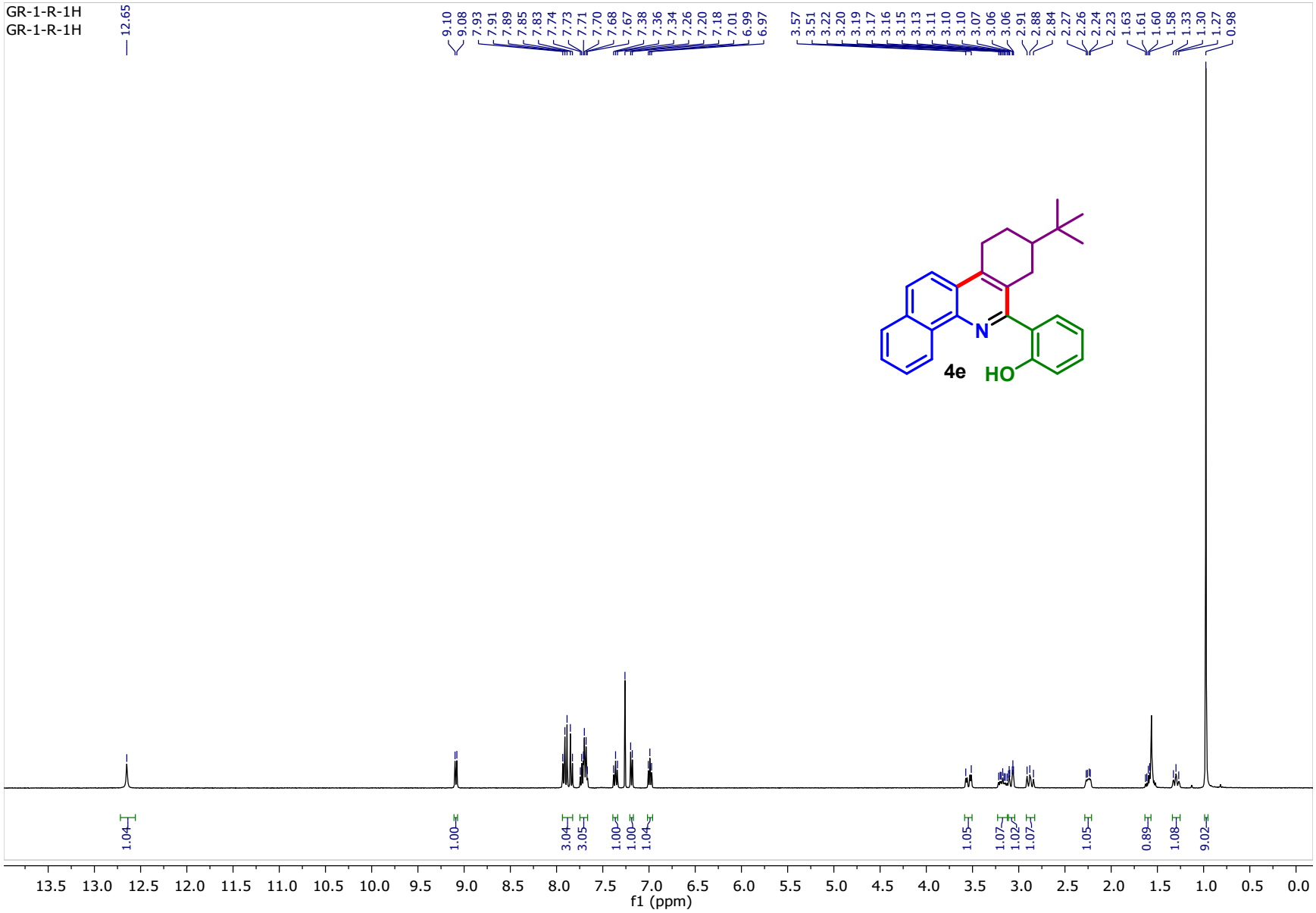


# HRMS Spectra of 4d

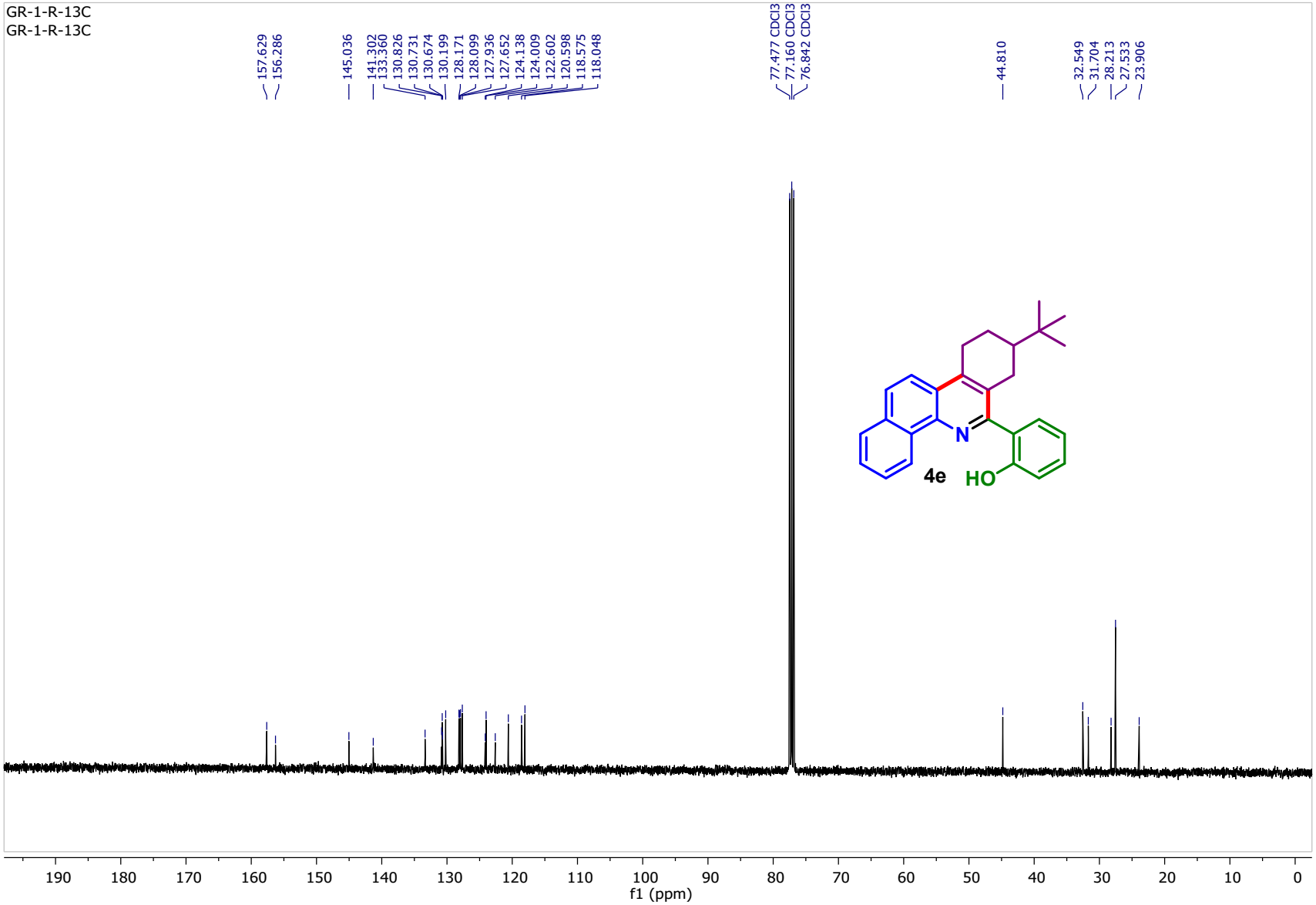
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | WASH | **Position** | P2-B5 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | ATK-SY-D1.d |
| **ACQ Method** | ESI ALS 100-1000.m | **Comment** | | **Acquired Time** | 08-Apr-21 05:09:48 PM (UTC+05:30) |



## $^{1}$H NMR Spectra of 4e

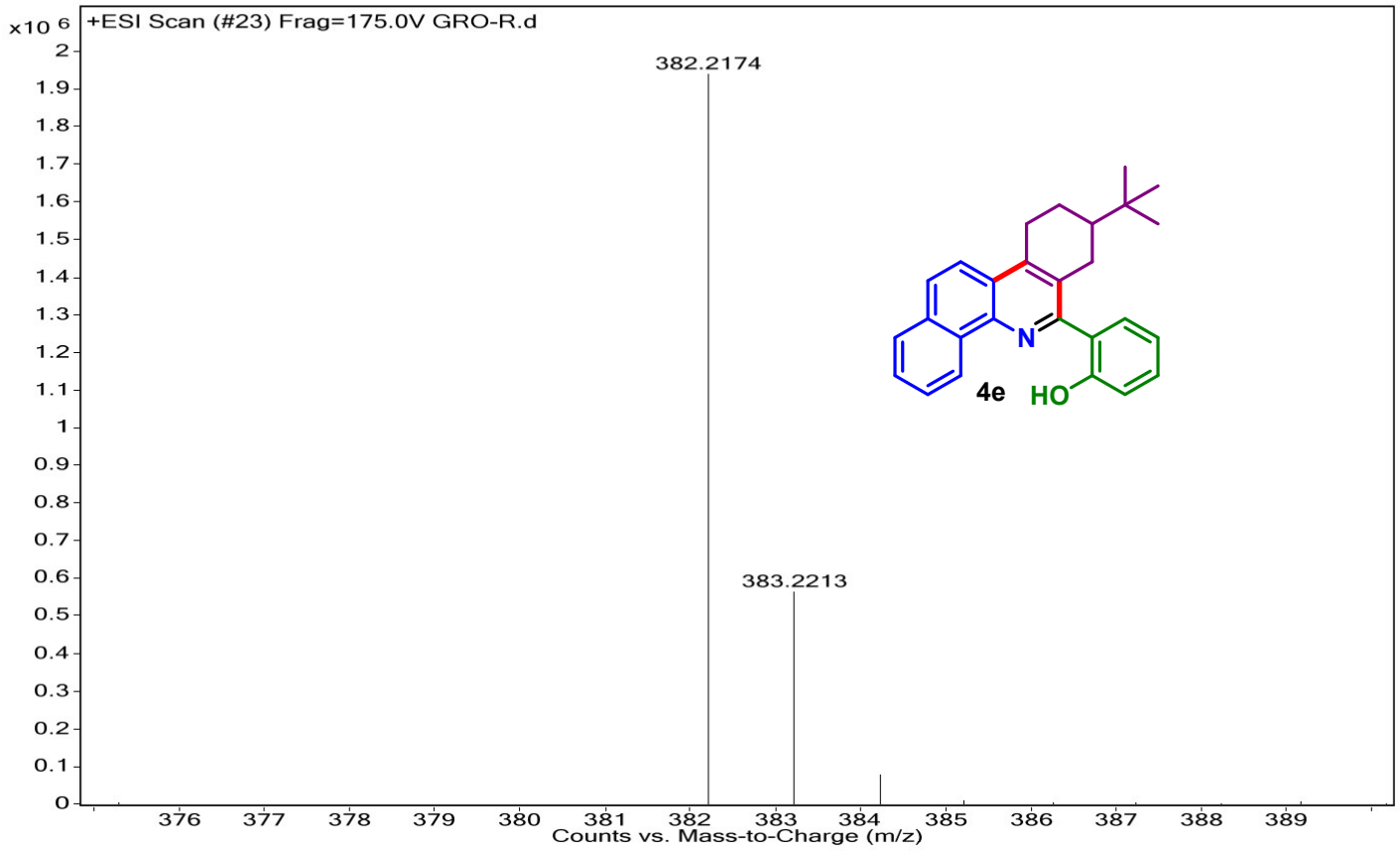


## $^{13}$C NMR Spectra of 4e

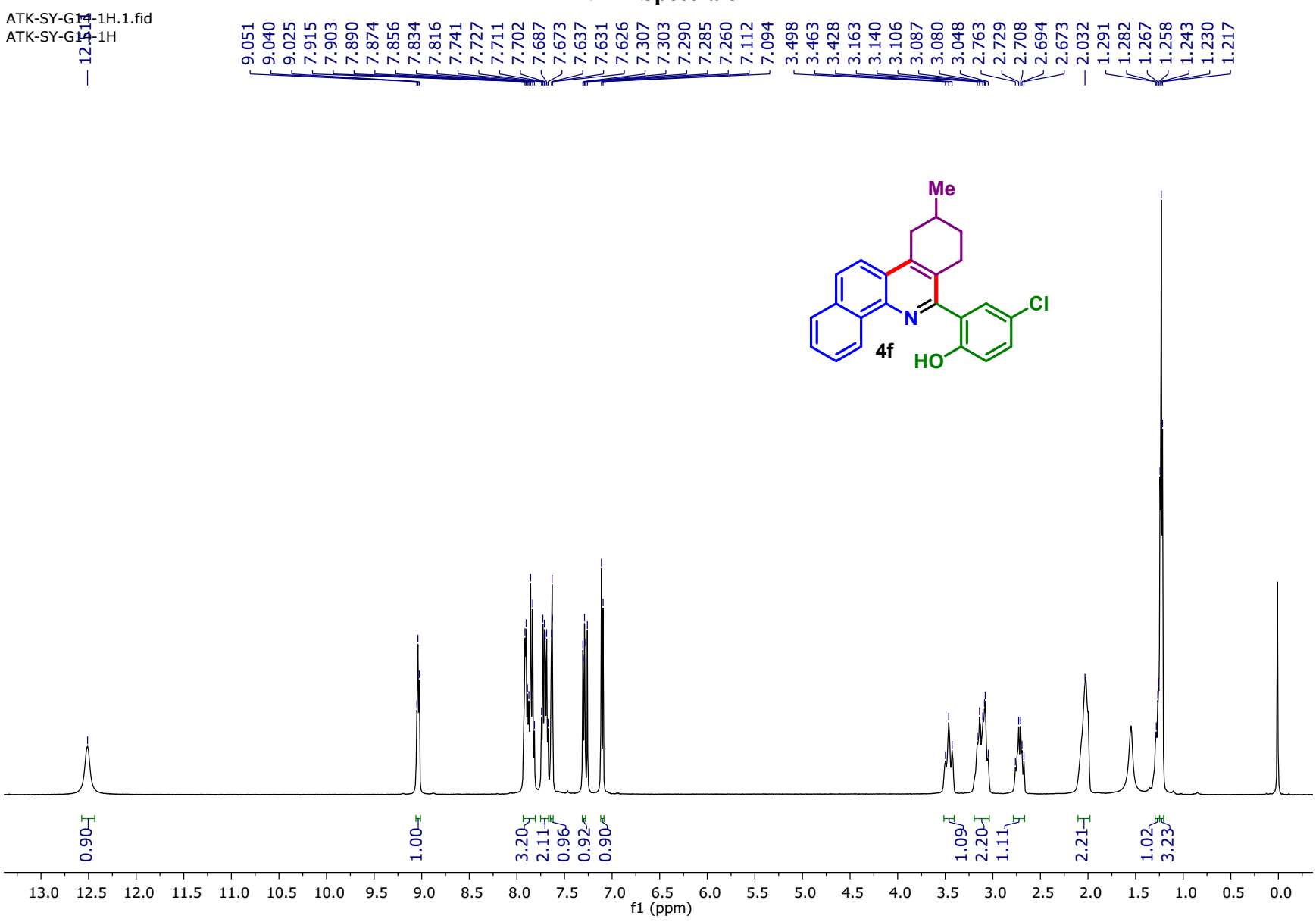


# HRMS Spectra of 4e

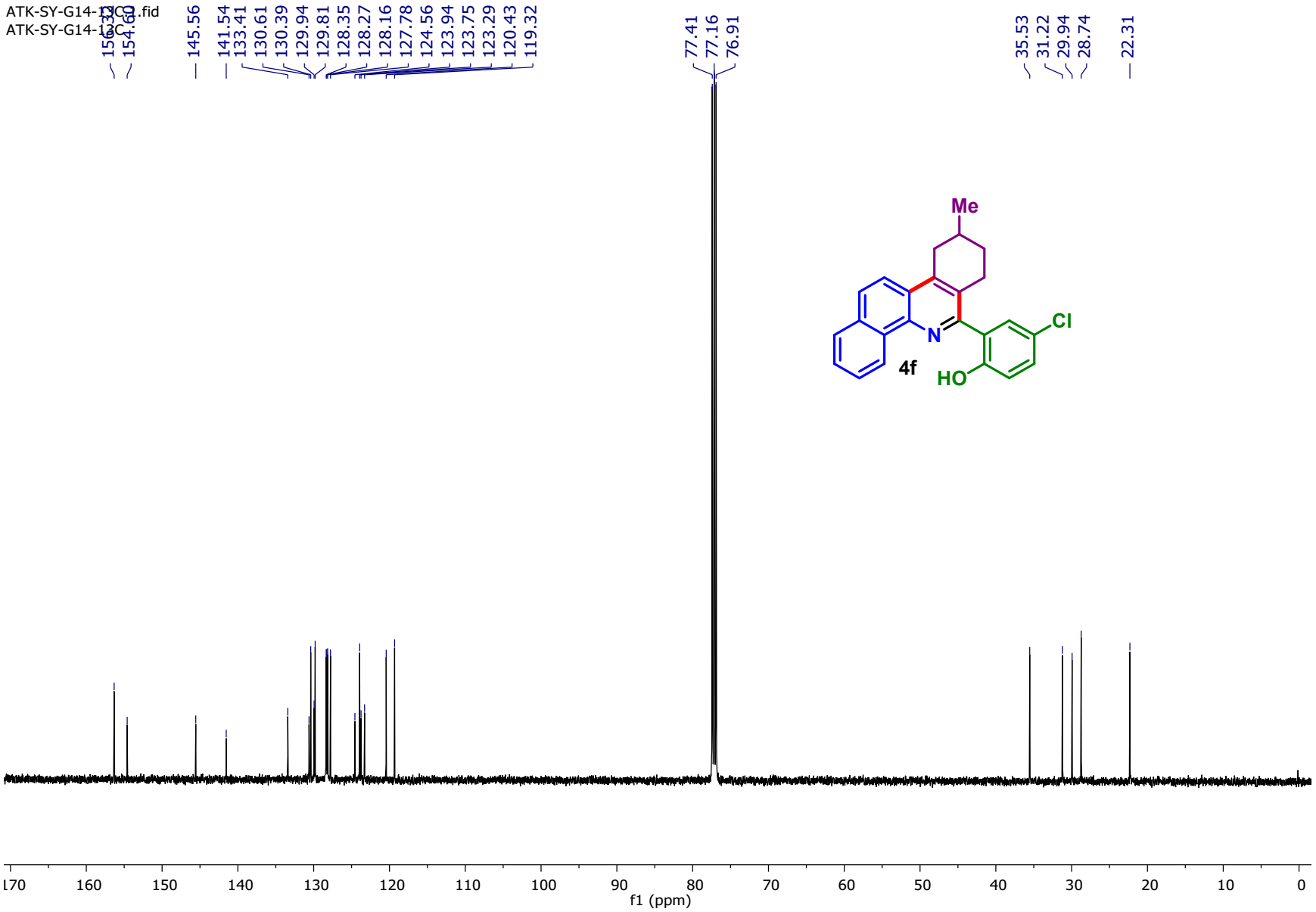
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Sample Name** | SAMPLE 3 | **Position** | P2-B1 | **Instrument Name** | Instrument 1 | **User Name** | |
| **Inj Vol** | 20 | **InjPosition** | | **SampleType** | Sample | **IRM Calibration Status** | Success |
| **Data Filename** | GRO-R.d | **ACQ Method** | ESI ALS 100-500.m | **Comment** | | **Acquired Time** | 12/26/2018 3:53:22 PM |



## $^{1}$H NMR Spectra of 4f

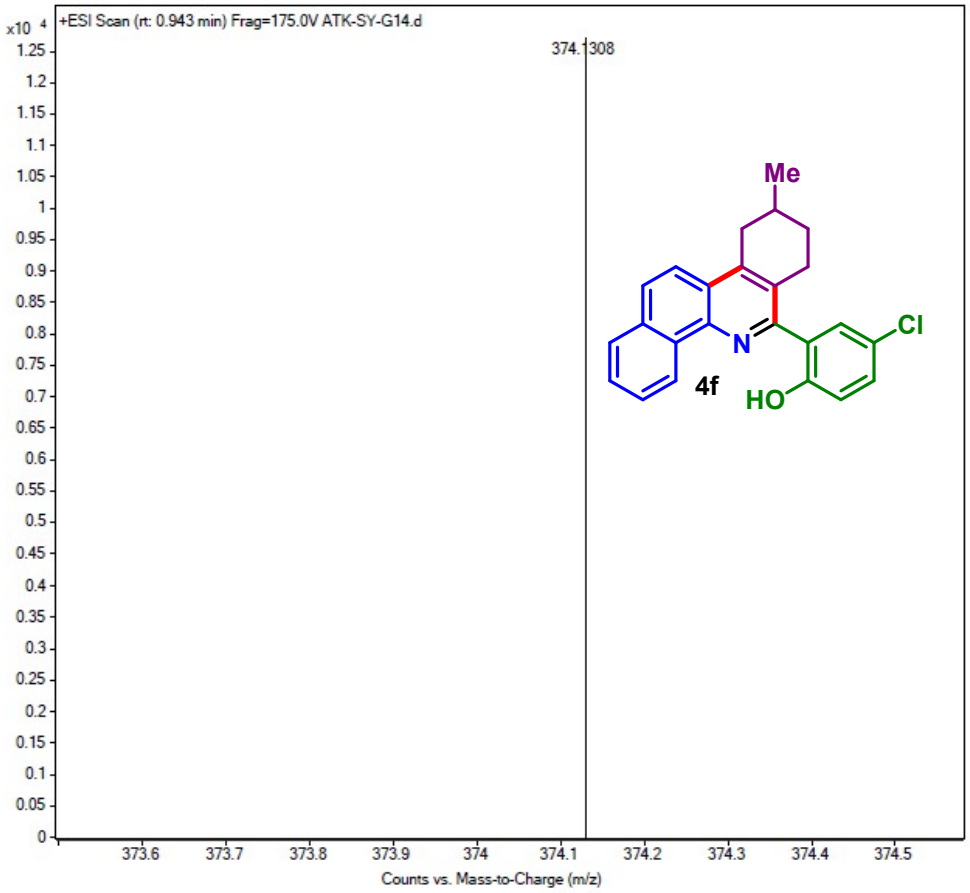


## $^{13}$C NMR Spectra of 4f

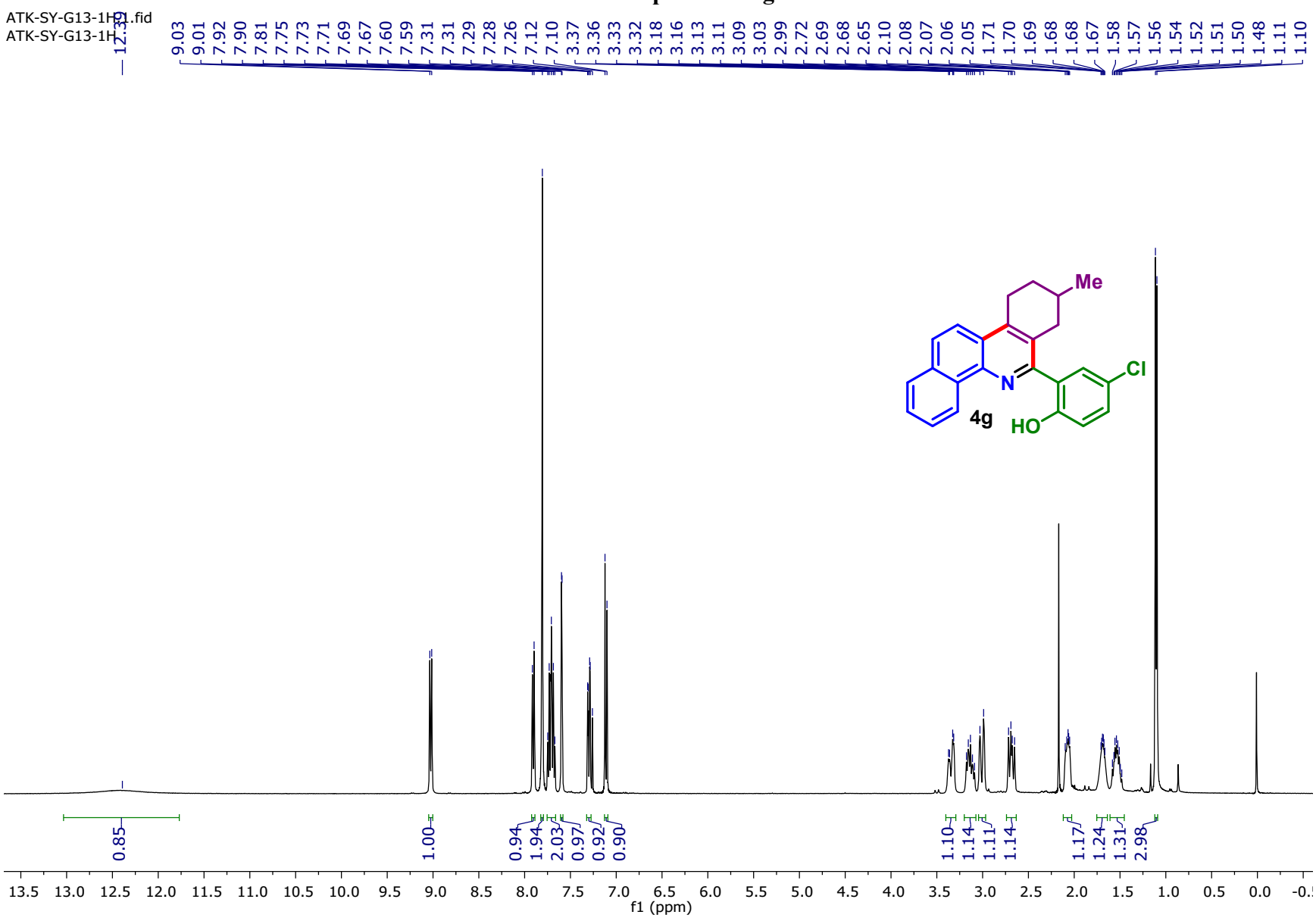


# HRMS Spectra of 4f

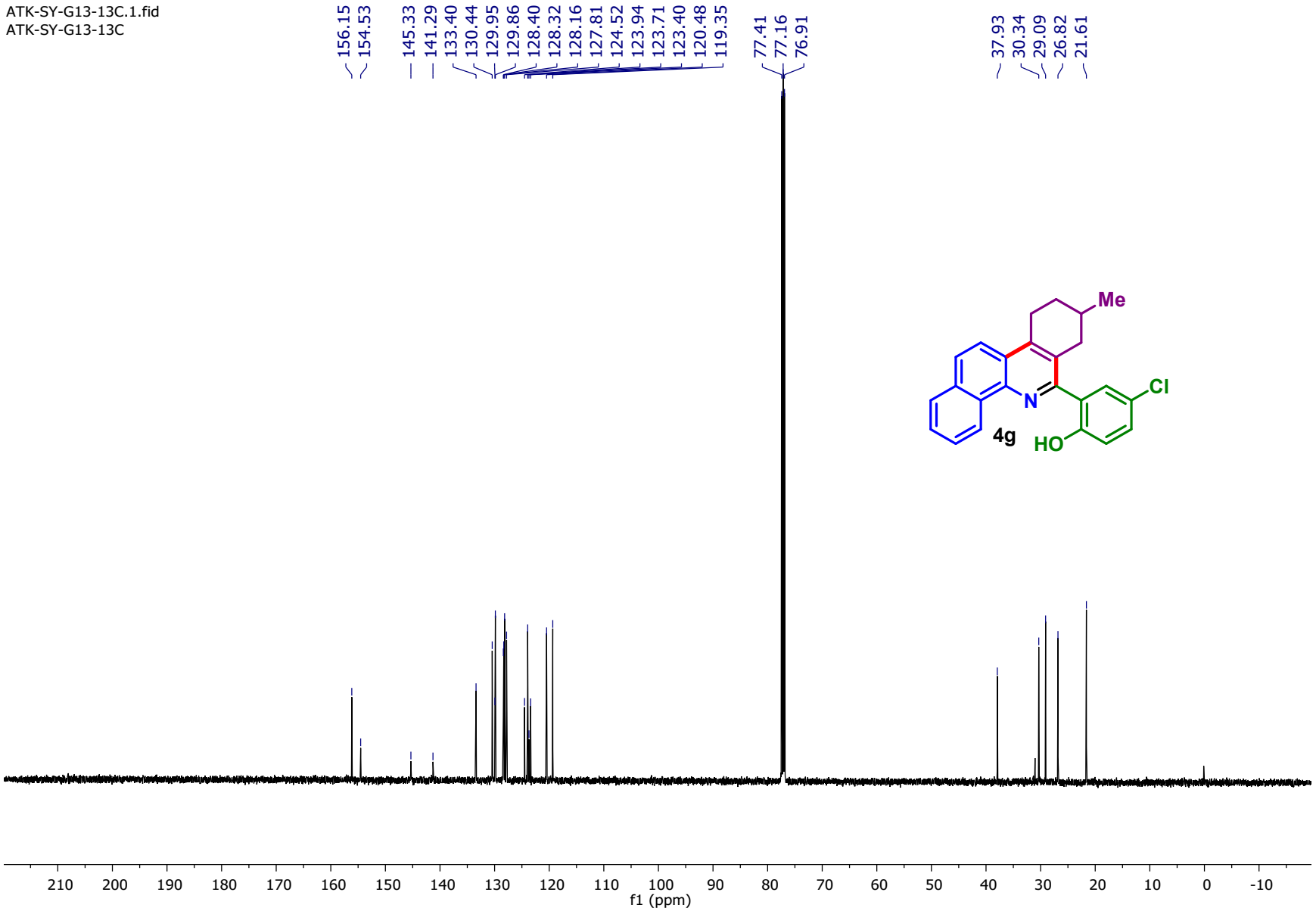
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SAMPLE | **Position** | P2-C4 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | ATK-SY-G14.d |
| **ACQ Method** | ESI ALS 200-600.m | **Comment** | | **Acquired Time** | 06-Sep-21 07:56:16 PM (UTC+05:30) |



## $^{1}$H NMR Spectra of 4g

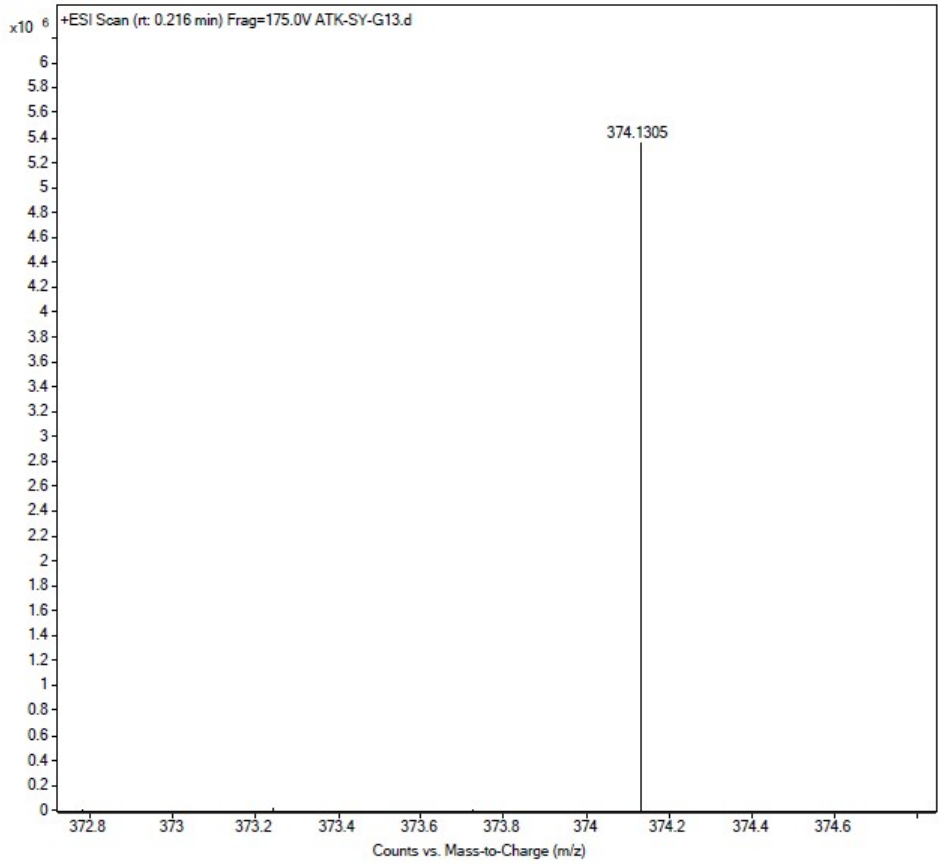


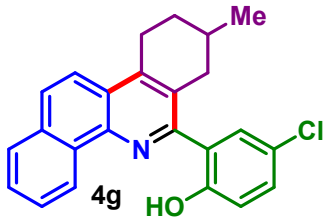
## $^{13}$C NMR Spectra of 4g



# HRMS Spectra of 4g

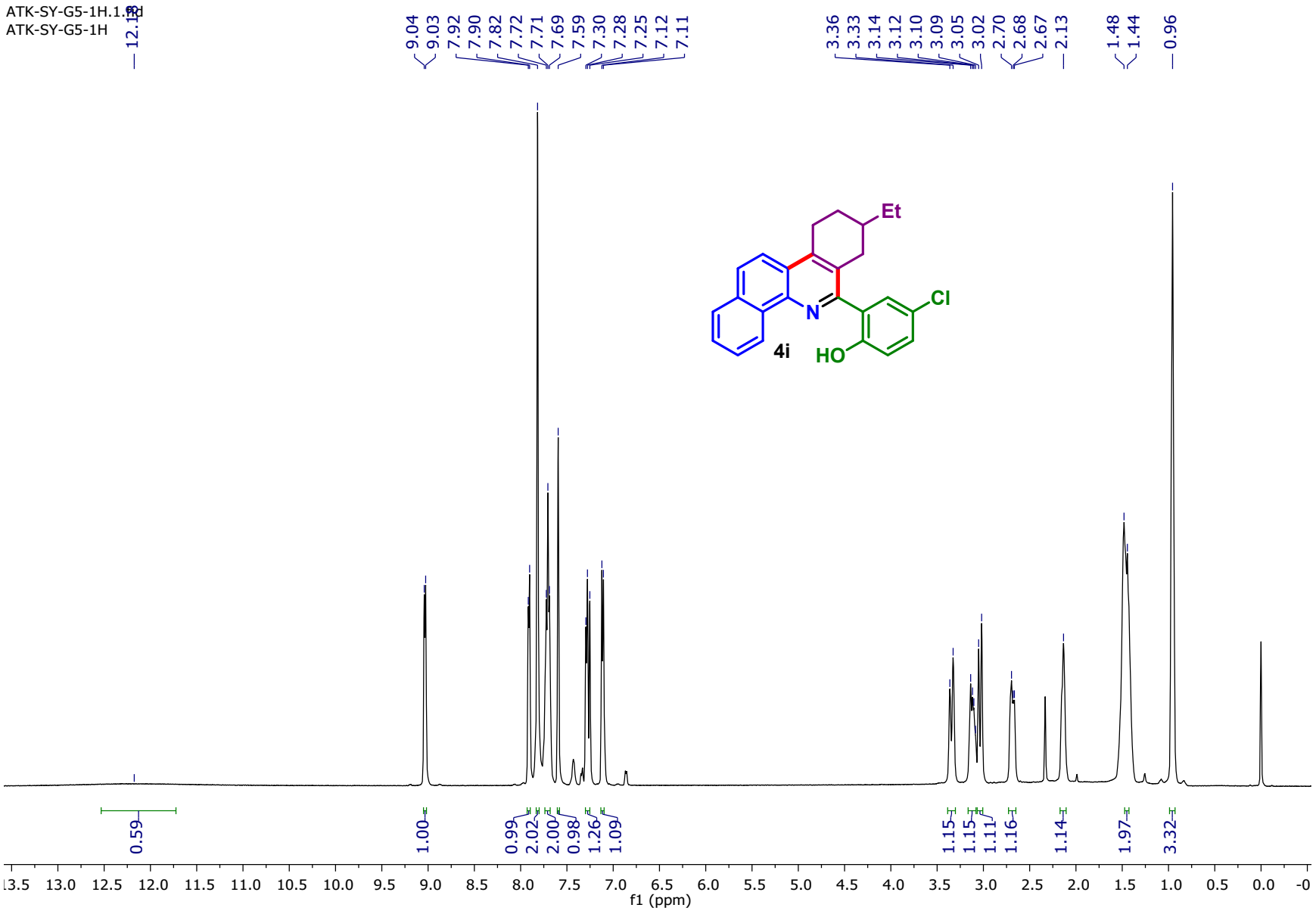
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SAMPLE | **Position** | P2-C3 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | ATK-SY-G13.d |
| **ACQ Method** | ESI ALS 200-600.m | **Comment** | | **Acquired Time** | 06-Sep-21 07:50:40 PM (UTC+05:30) |



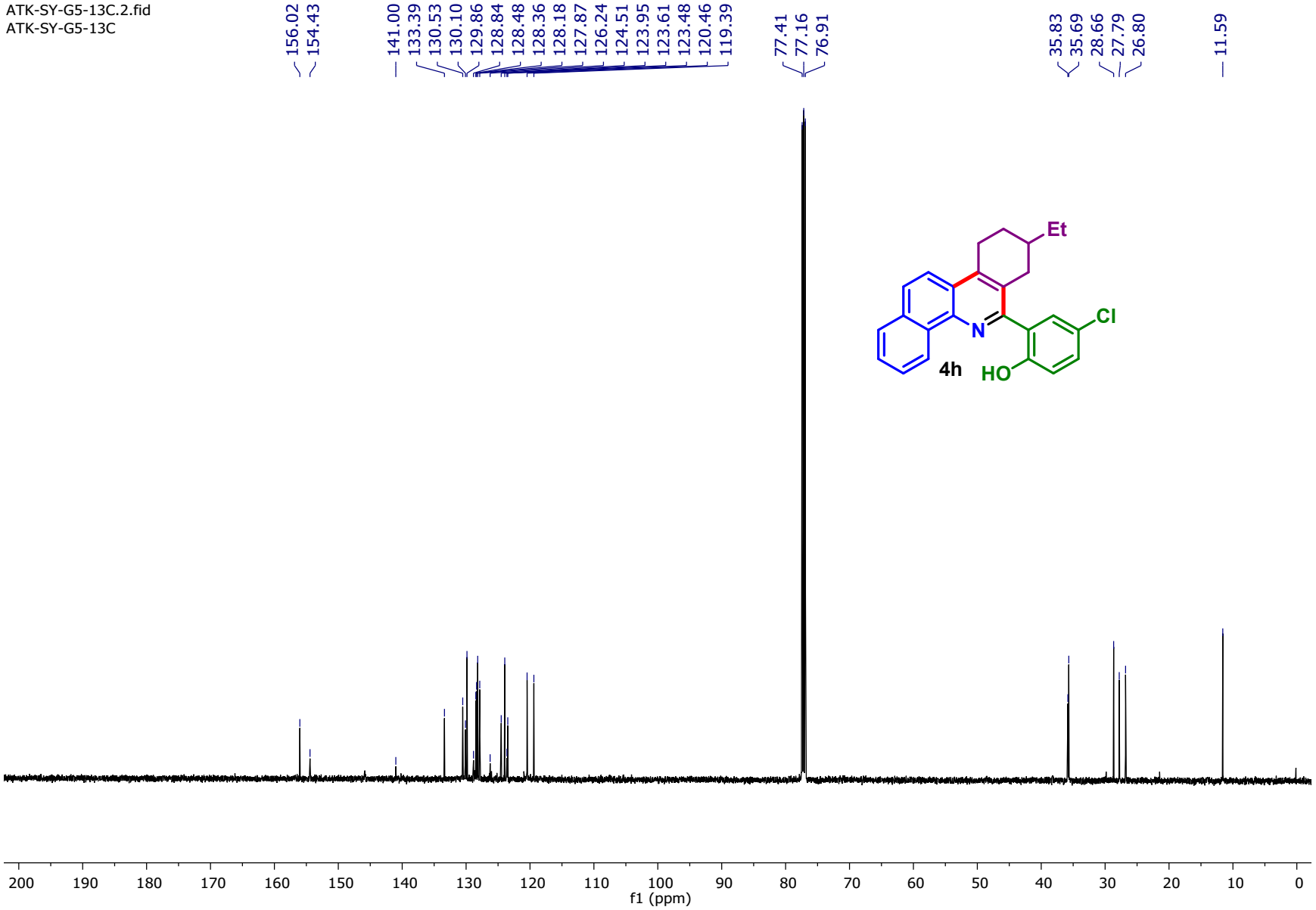

4g

## $^1$H NMR Spectra of 4h



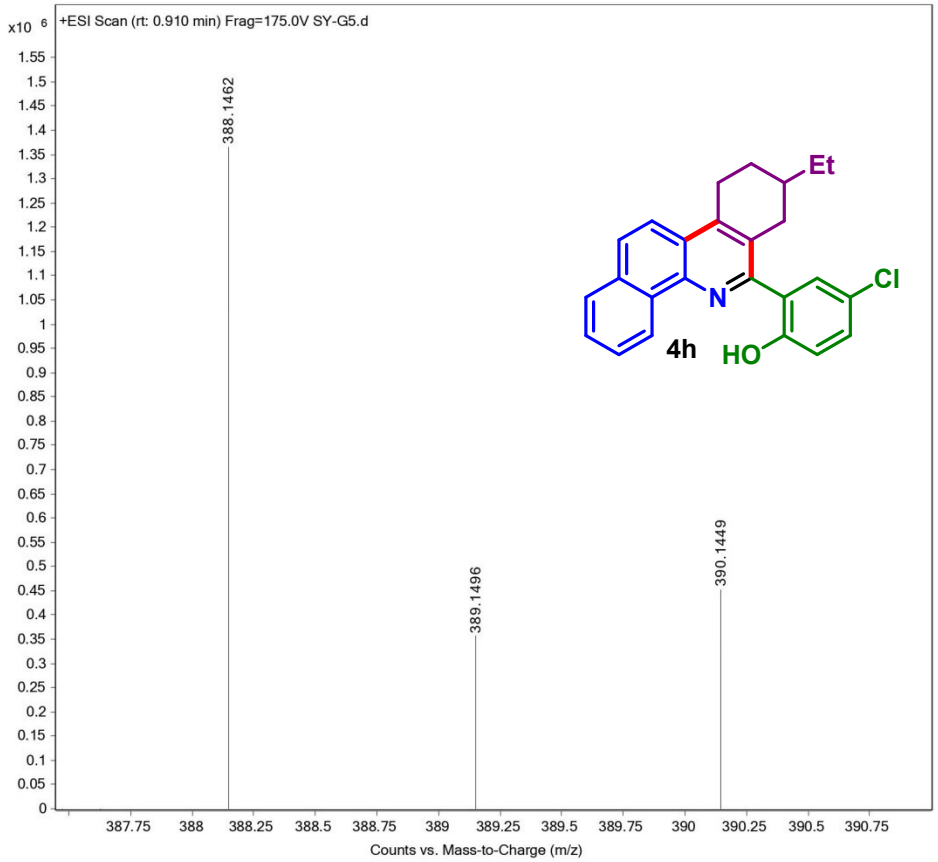
## $^{13}$C NMR Spectra of 4h

ATK-SY-G5-13C.2.fid
ATK-SY-G5-13C

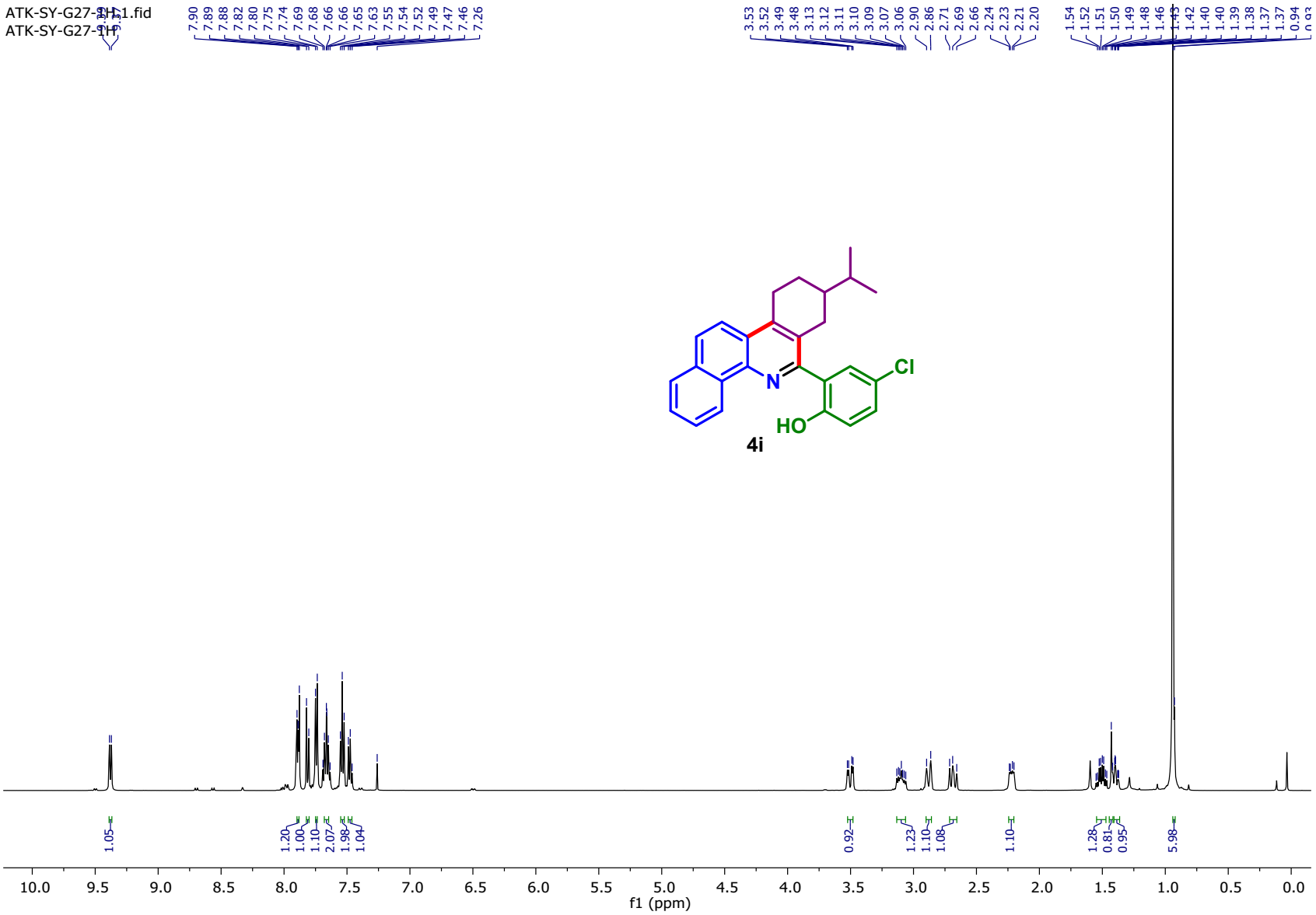


# HRMS Spectra of 4h

| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SAMPLE 19 | **Position** | P1-B7 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-G5.d |
| **ACQ Method** | ESI ALS 100-500.m | **Comment** | | **Acquired Time** | 21-Apr-21 10:53:01 PM (UTC+05:30) |



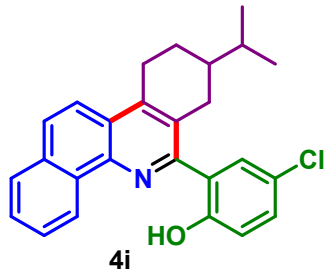
## $^{1}$H NMR Spectra of 4i



## $^{13}$C NMR Spectra of 4i

ATK-SY-G27-13C.8.fid
ATK-SY-G27-13C

158.60
143.42
142.27
141.75
133.11
132.26
130.56
129.66
129.57
129.49
128.37
128.14
128.03
127.56
127.18
126.77
125.10
124.06
120.76
77.48
77.16
76.84
44.61
32.49
30.46
27.65
27.40
23.95




**4i**

**HRMS Spectra of 4i**

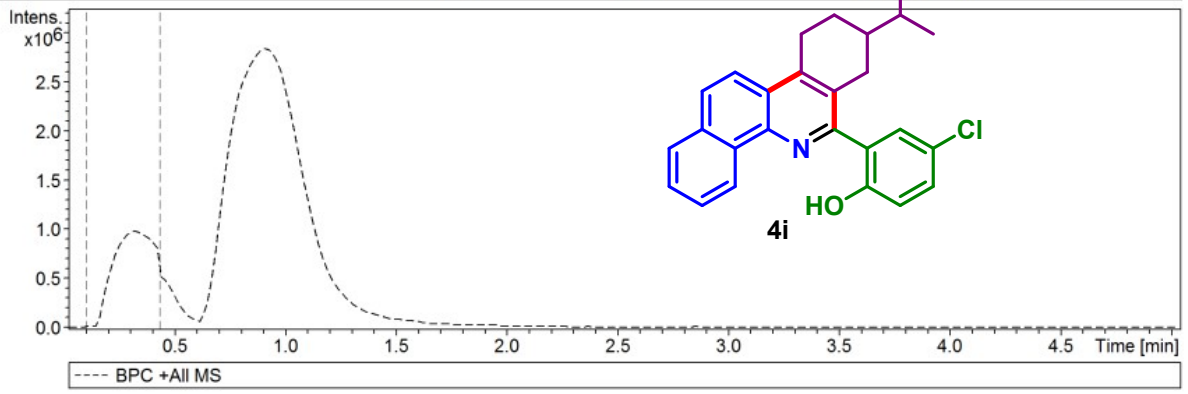
# Display Report

**Analysis Info**

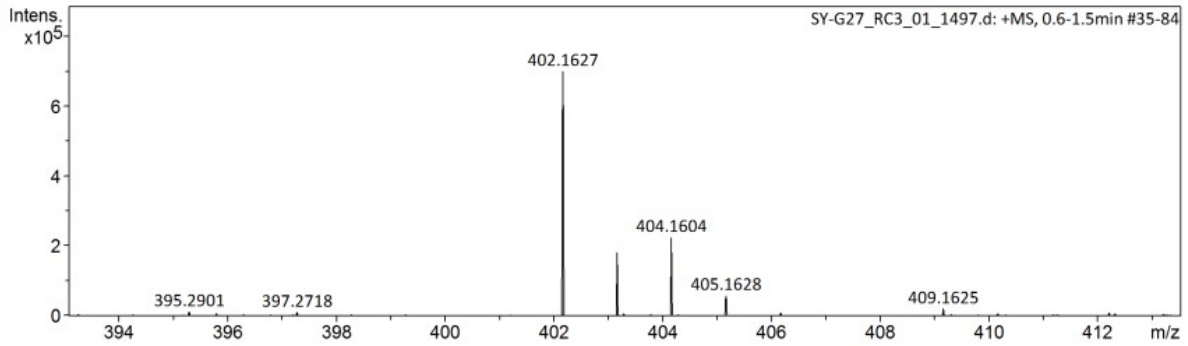
| | | | |
|---|---|---|---|
| Acquisition Date | 1/27/2022 1:46:48 PM | | |
| Analysis Name | D:\Data\user data\HPLC\DR LOKMAN\PRABHAS\SY-G27_RC3_01_1497.d | | |
| Method | low mass bruker.m | Operator | vidhi |
| Sample Name | SY-G27 | Instrument | impact HD 1819696.00197 |
| Comment | | | |

**Acquisition Parameter**

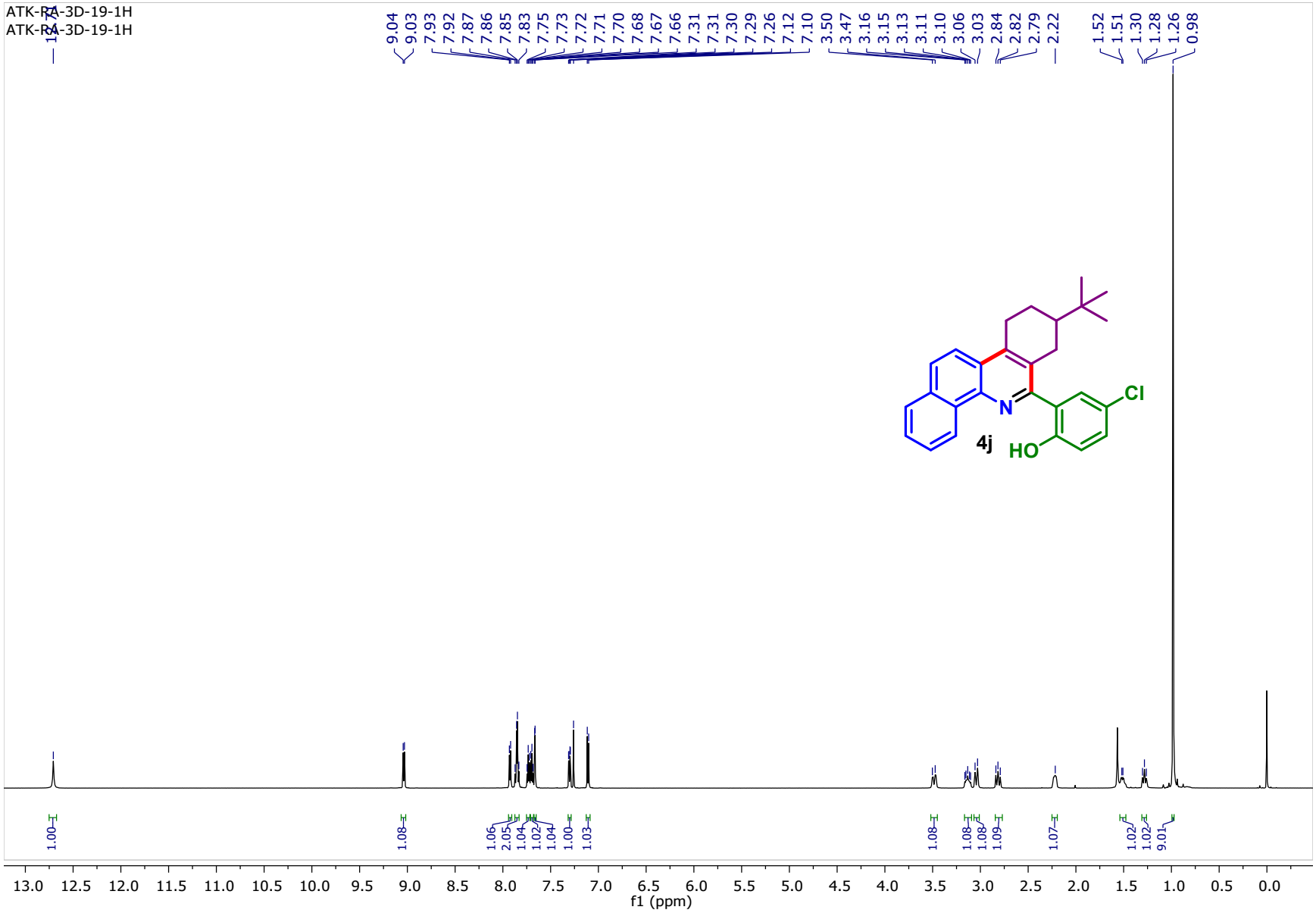
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.8 Bar |
| Focus | Active | Set Capillary | 4500 V | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 6.0 l/min |
| Scan End | 1500 m/z | Set Charging Voltage | 2000 V | Set Divert Valve | Waste |
| | | Set Corona | 0 nA | Set APCI Heater | 0 °C |



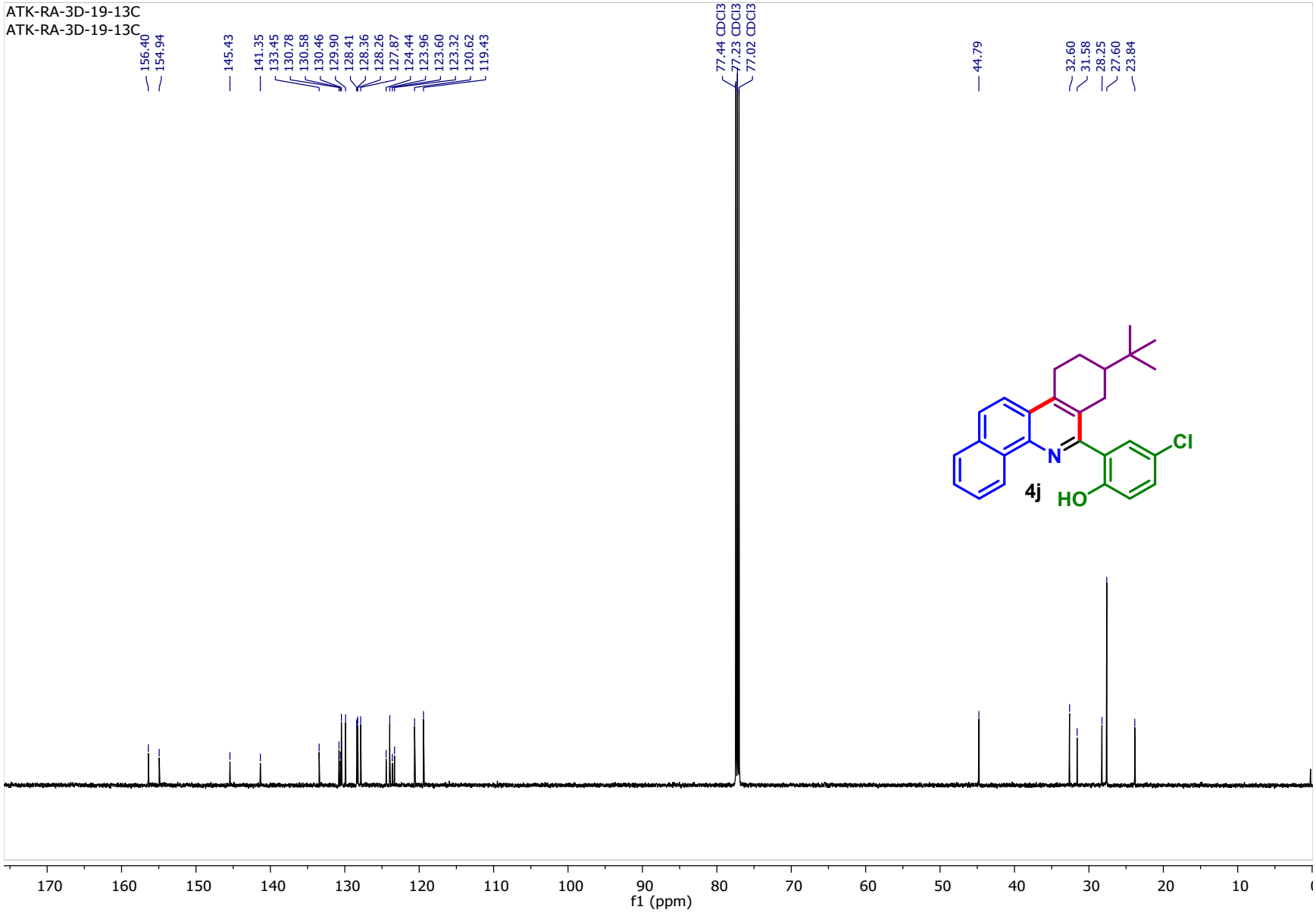

4i



## $^{1}$H NMR Spectra of 4j

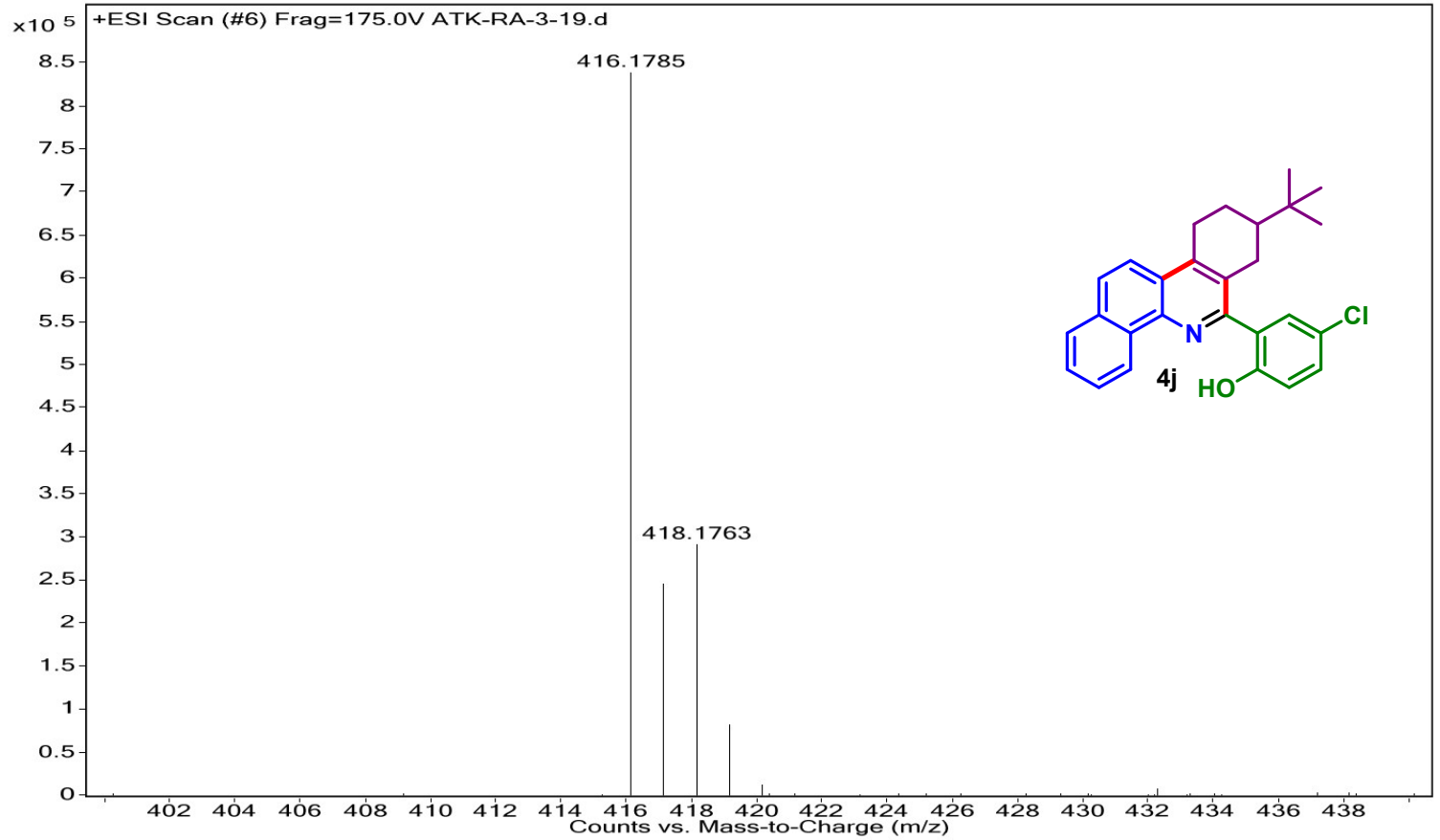


## $^{13}$C NMR Spectra of 4j

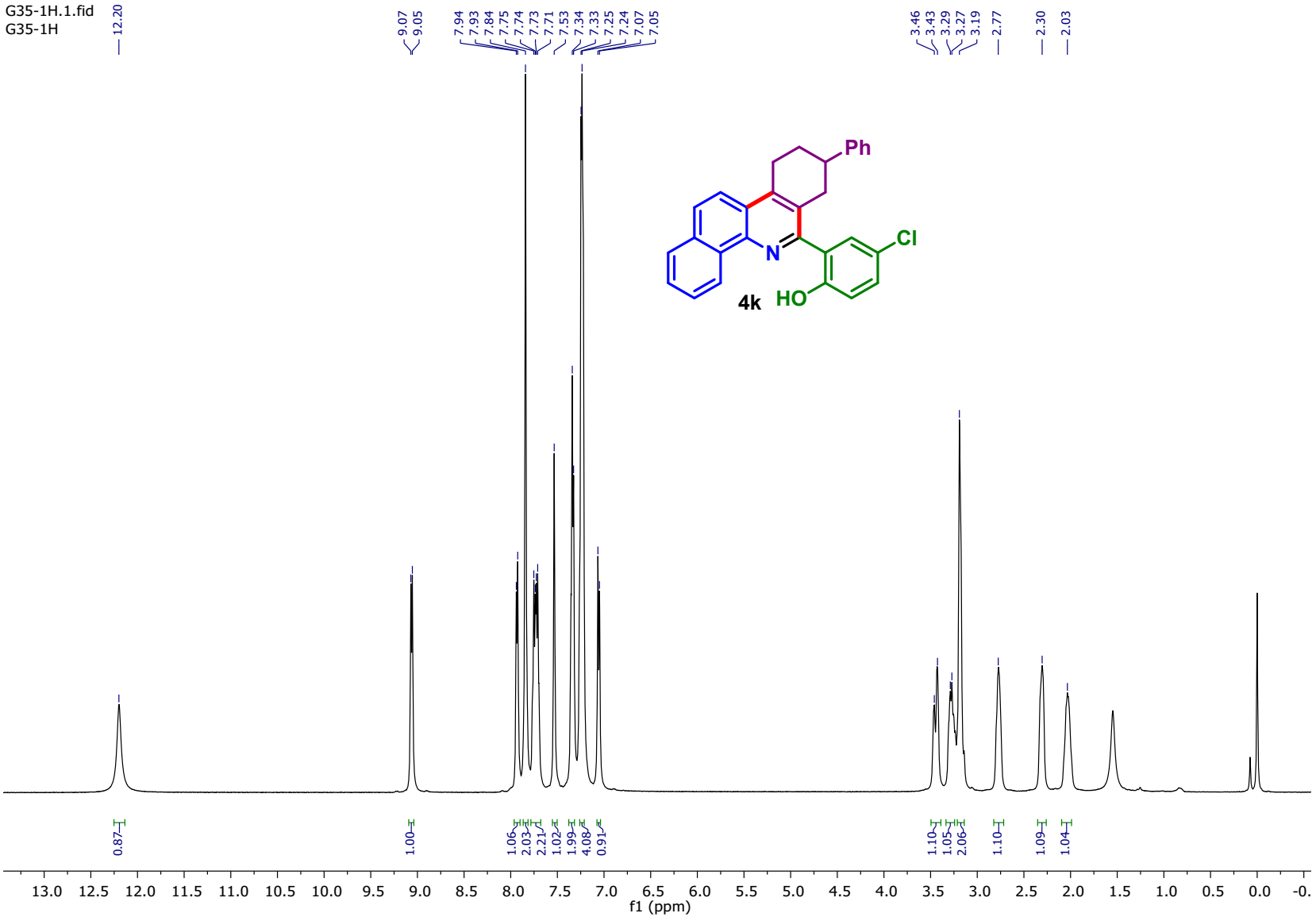


# HRMS Spectra of 4j

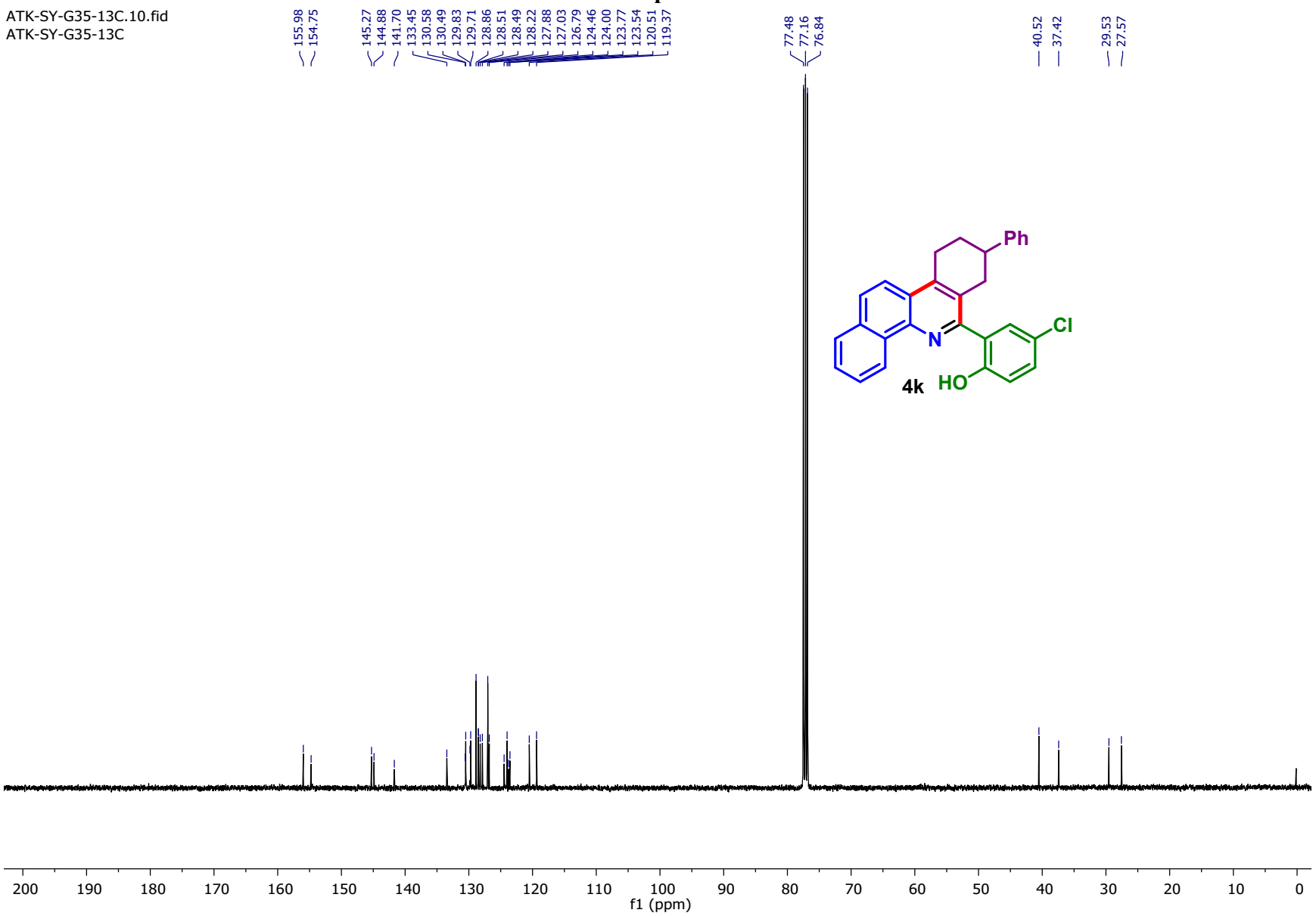
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Sample Name** | SAMPLE | **Position** | P2-B7 | **Instrument Name** | Instrument 1 | **User Name** | |
| **Inj Vol** | 20 | **InjPosition** | | **SampleType** | Sample | **IRM Calibration Status** | Success |
| **Data Filename** | ATK-RA-3-19.d | **ACQ Method** | ESI ALS 100-600.m | **Comment** | | **Acquired Time** | 2/5/2020 4:55:27 PM |



## $^1$H NMR Spectra of 4k



## $^{13}$C NMR Spectra of 4k



## HRMS Spectra of 4k

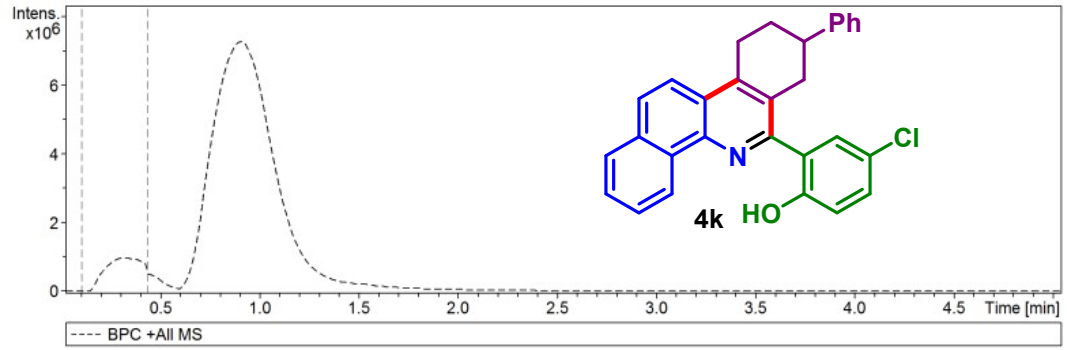
# Display Report

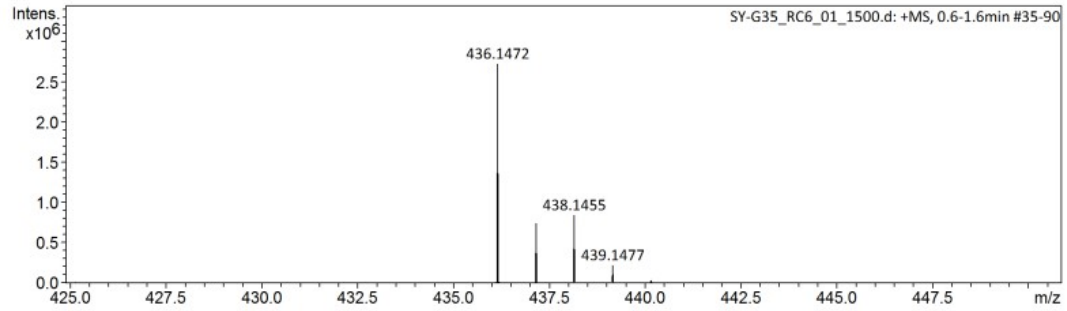
**Analysis Info**

| | | | | | |
|---|---|---|---|---|---|
| Analysis Name | D:\Data\user data\HPLC\DR LOKMAN\PRABHAS\SY-G35_RC6_01_1500.d | | | Acquisition Date | 1/27/2022 2:06:17 PM |
| Method | low mass bruker.m | | | Operator | vidhi |
| Sample Name | SY-G35 | | | Instrument | impact HD 1819696.00197 |
| Comment | | | | | |

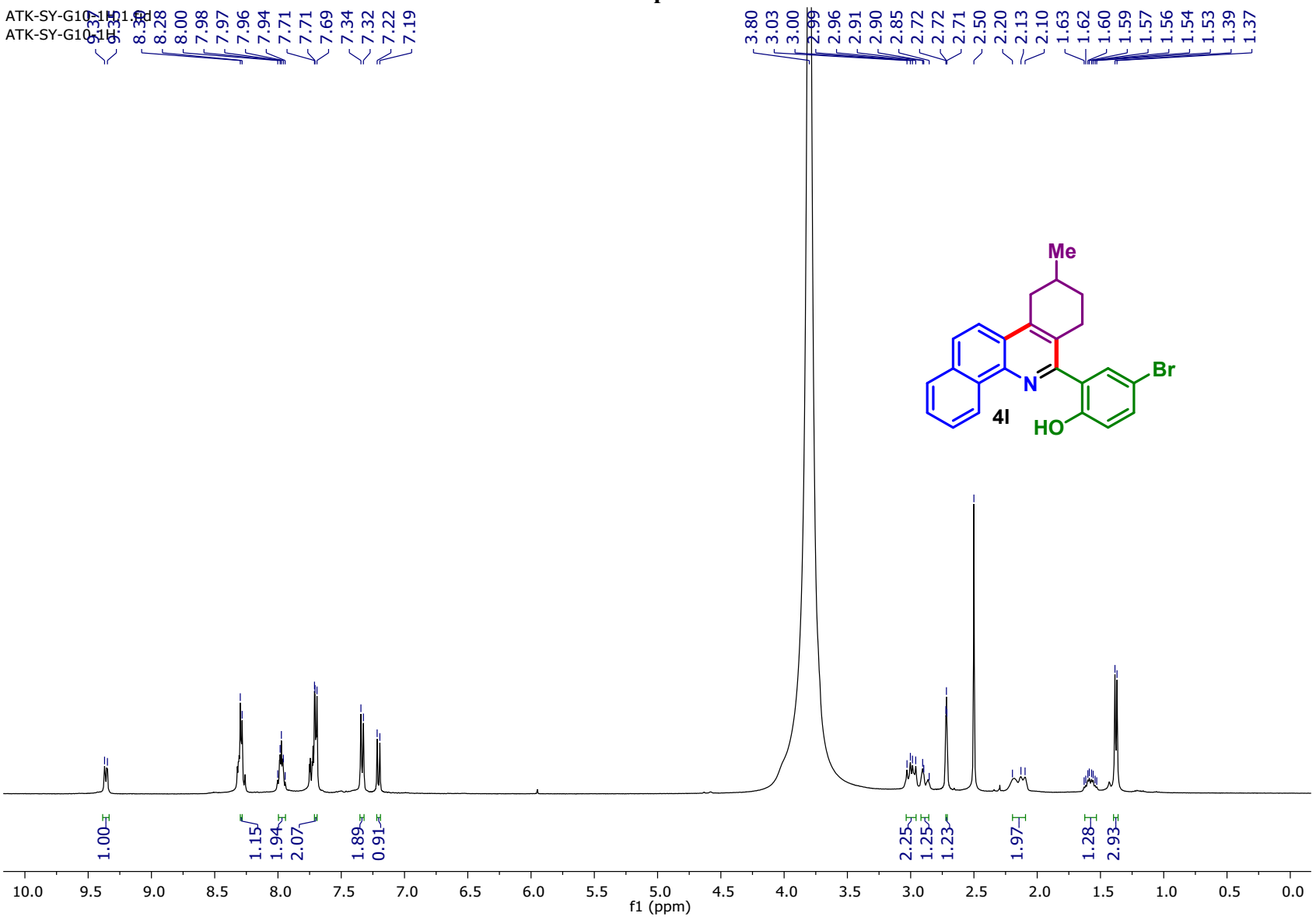
**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.8 Bar |
| Focus | Active | Set Capillary | 4500 V | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 6.0 l/min |
| Scan End | 1500 m/z | Set Charging Voltage | 2000 V | Set Divert Valve | Waste |
| | | Set Corona | 0 nA | Set APCI Heater | 0 °C |

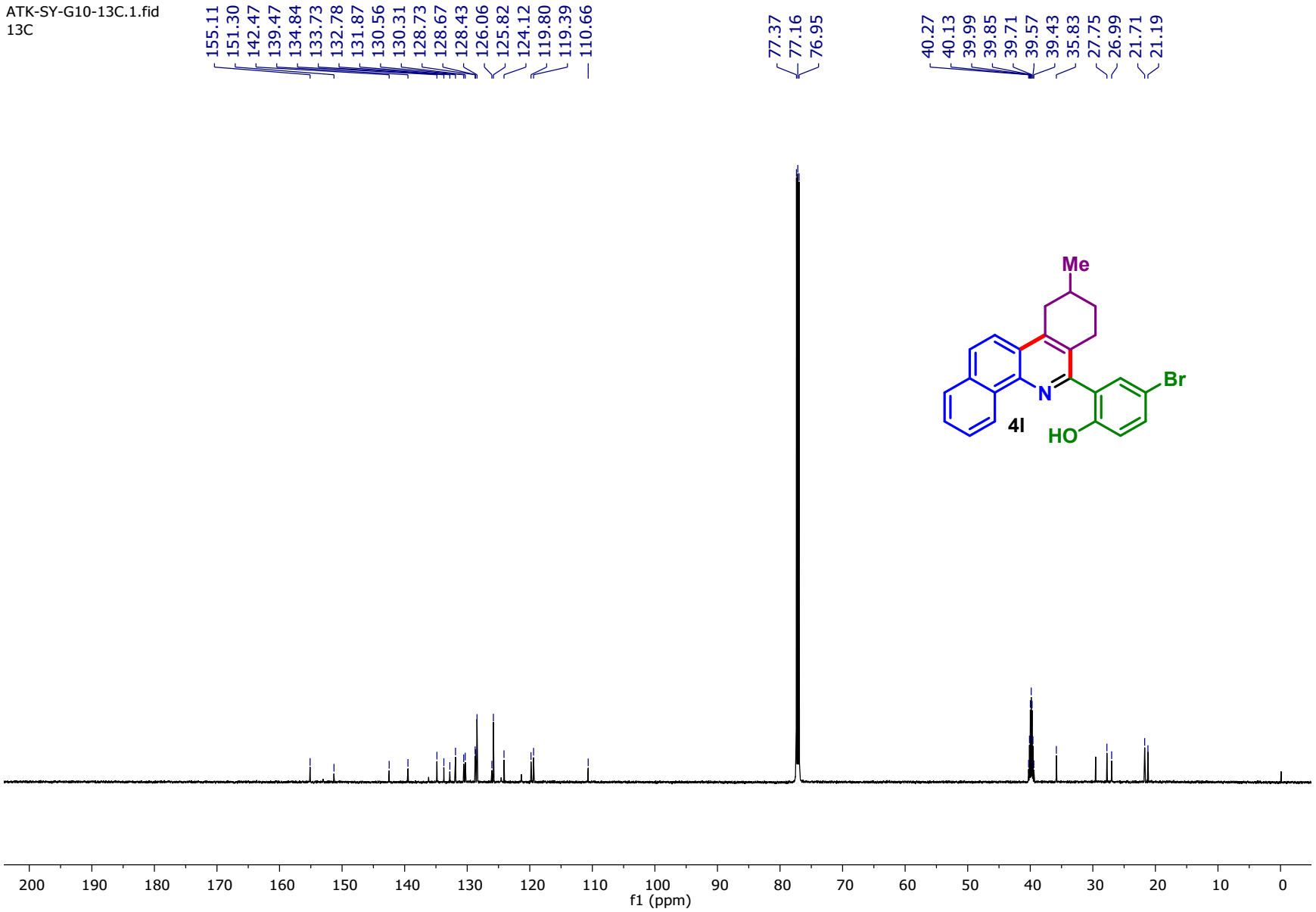


## $^{1}$H NMR Spectra of 4l

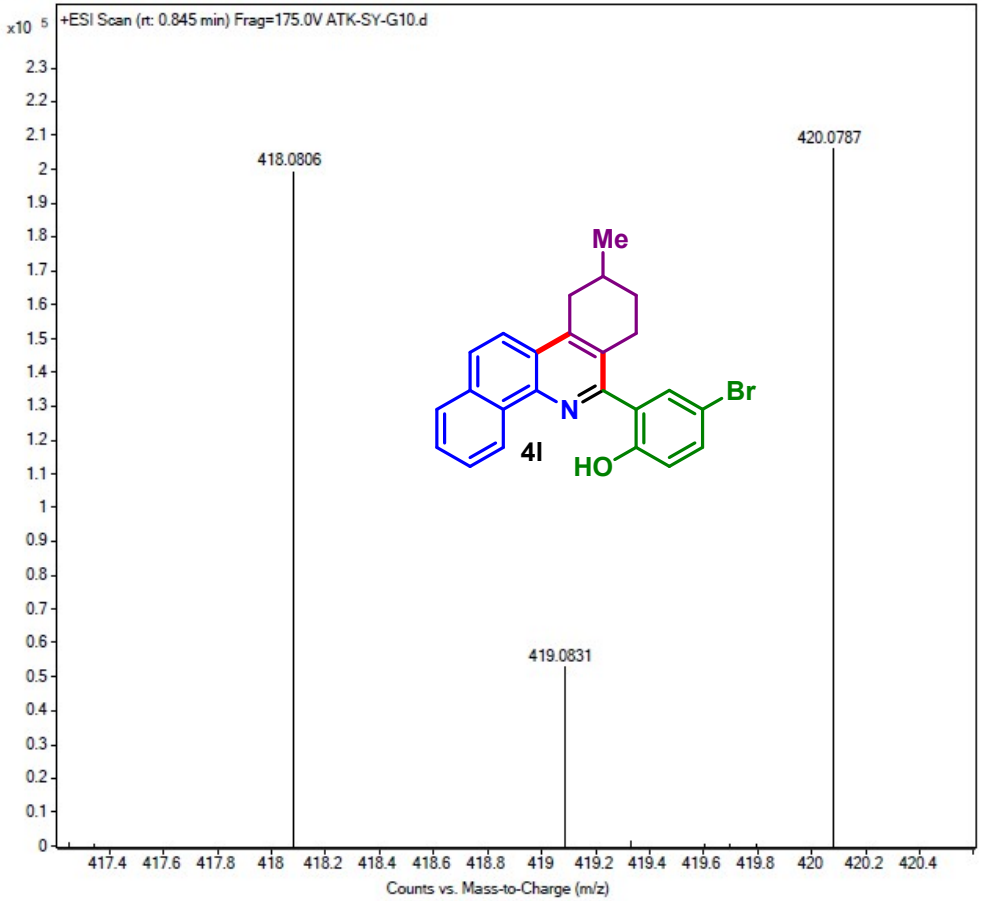


## $^{13}$C NMR Spectra of 4l

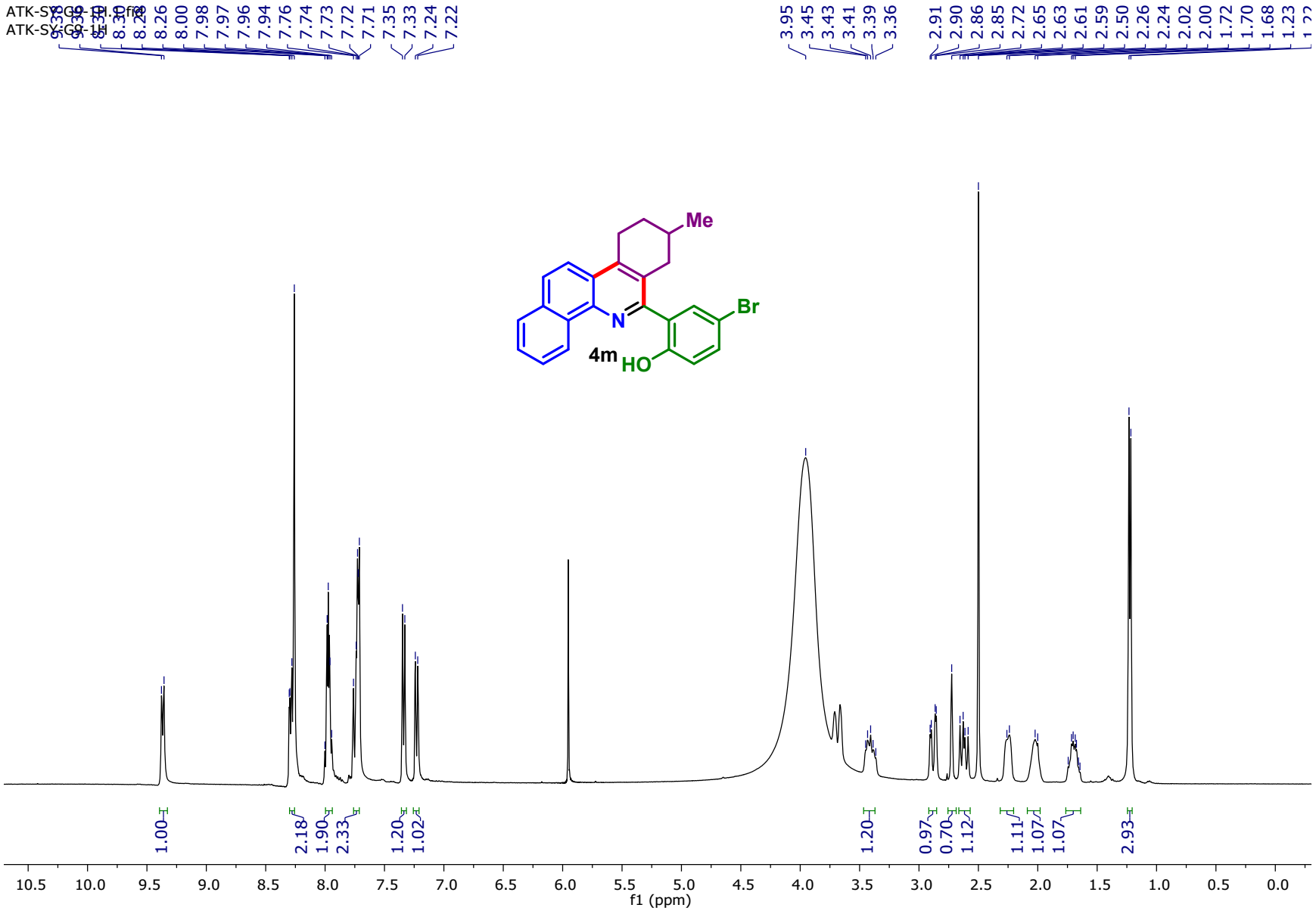


# HRMS Spectra of 4l

| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SAMPLE | **Position** | P2-B11 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | ATK-SY-G10.d |
| **ACQ Method** | ESI ALS 200-600.m | **Comment** | | **Acquired Time** | 06-Sep-21 07:39:30 PM (UTC+05:30) |



## $^{1}$H NMR Spectra of 4m



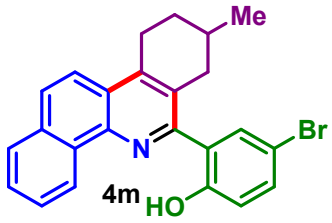
## $^{13}$C NMR Spectra of 4m

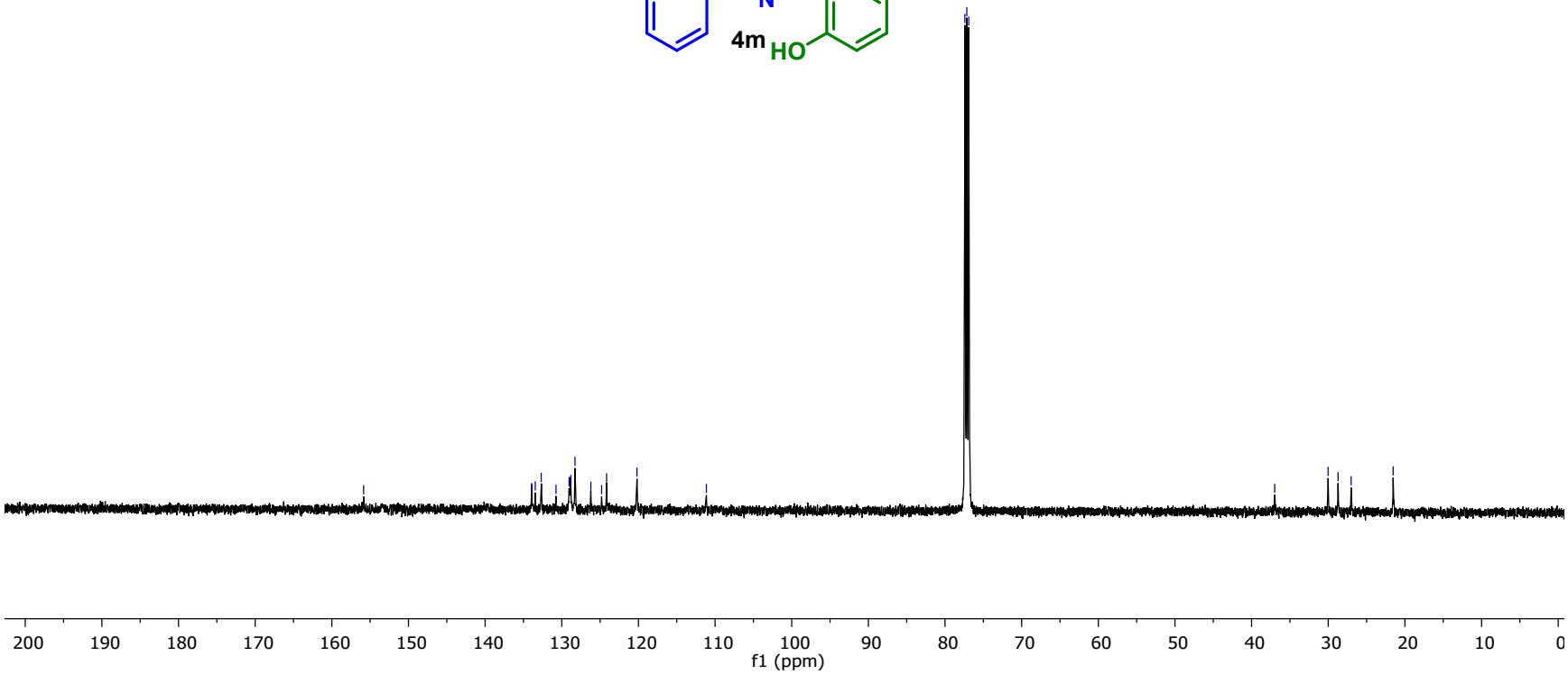
ATK-SY-G9-13C.3.fid
ATK-SY-G9-13C

155.85 133.96 133.91 133.89 133.46 132.68 132.64 130.75 129.04 128.94 128.81 128.28 126.21 124.80 124.15 120.19 111.13

77.41 77.16 76.91

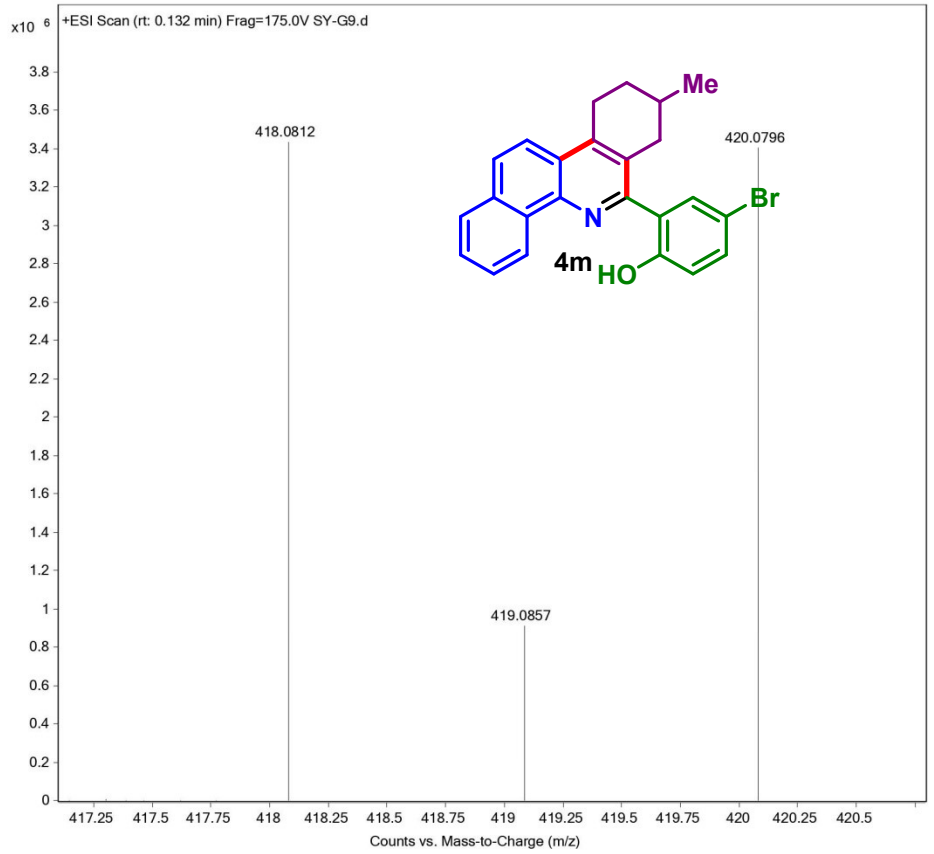
36.99 30.02 28.72 27.01 21.53



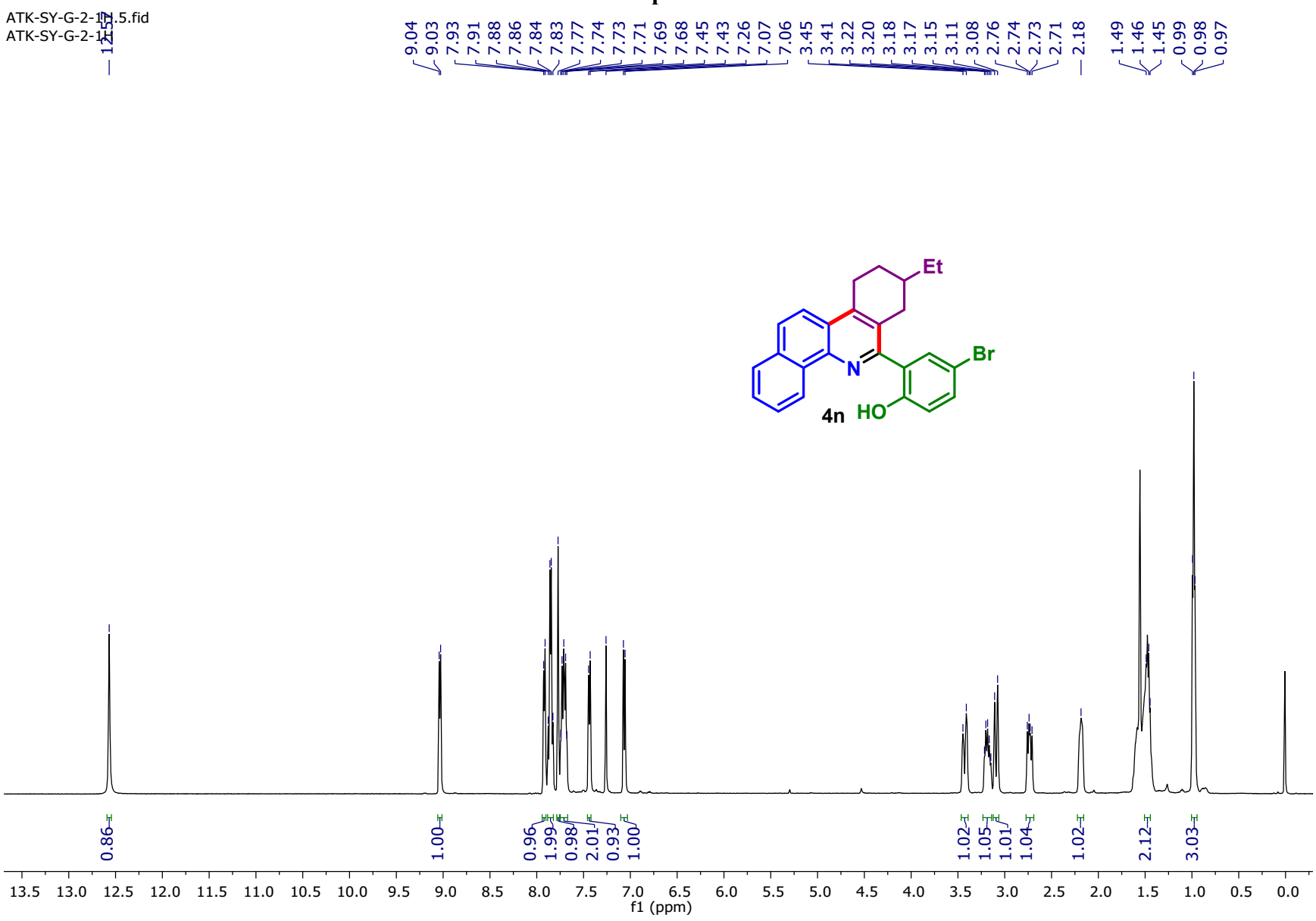



# HRMS Spectra of 4m

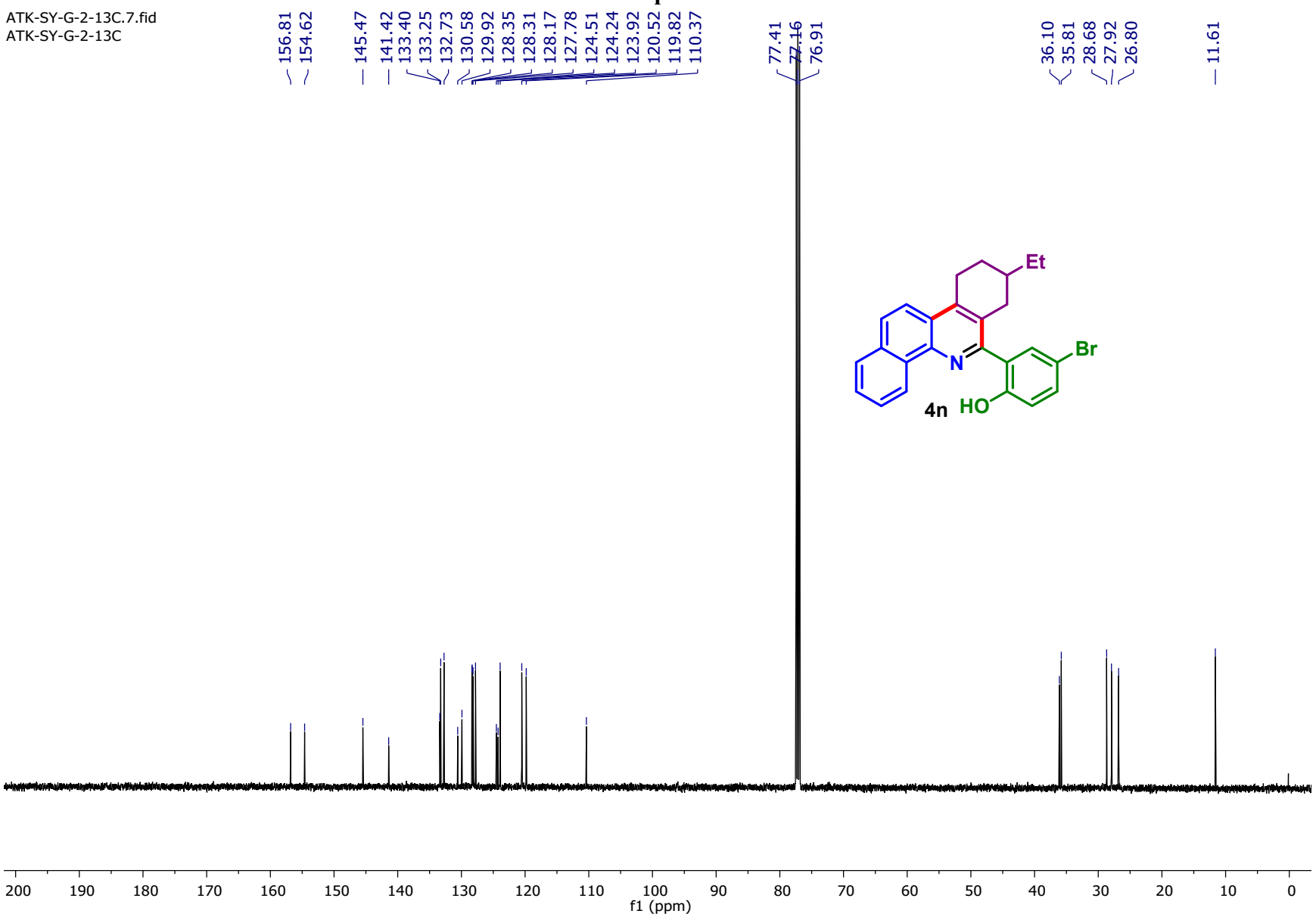
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SY-G9 | **Position** | P1-B8 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-G9.d |
| **ACQ Method** | ESI ALS 100-1000.m | **Comment** | | **Acquired Time** | 01-Sep-21 11:07:17 AM (UTC+05:30) |



## $^1$H NMR Spectra of 4n

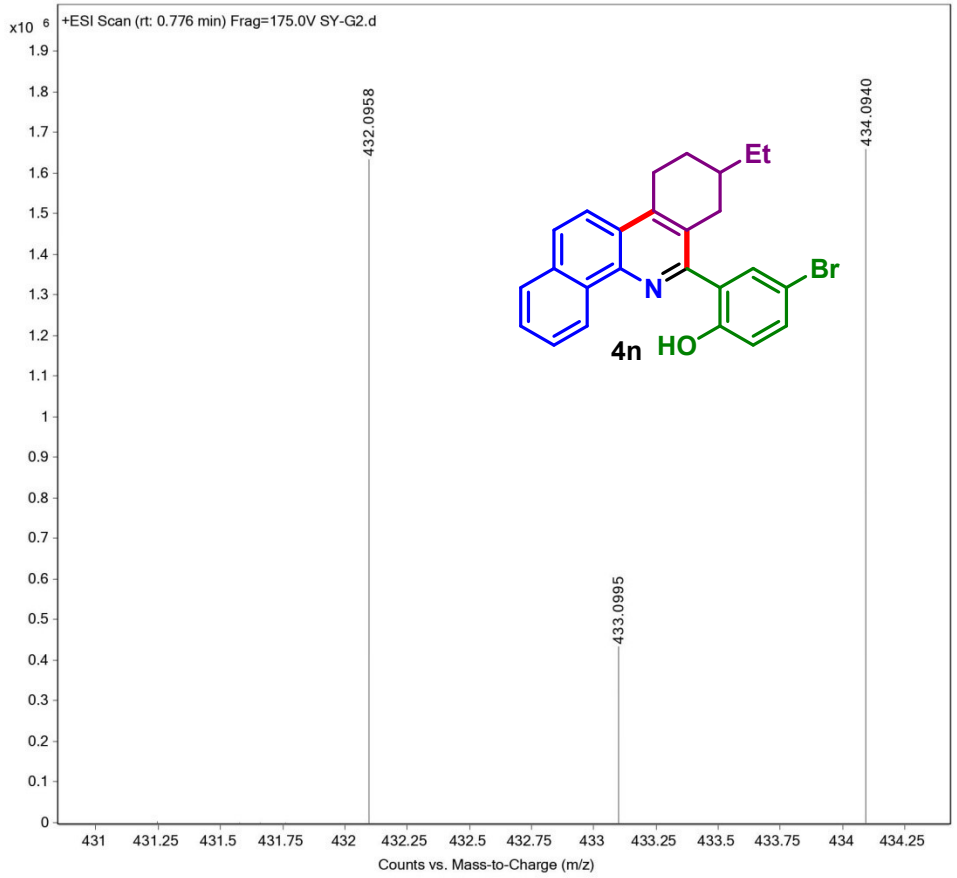


## $^{13}$C NMR Spectra of 4n

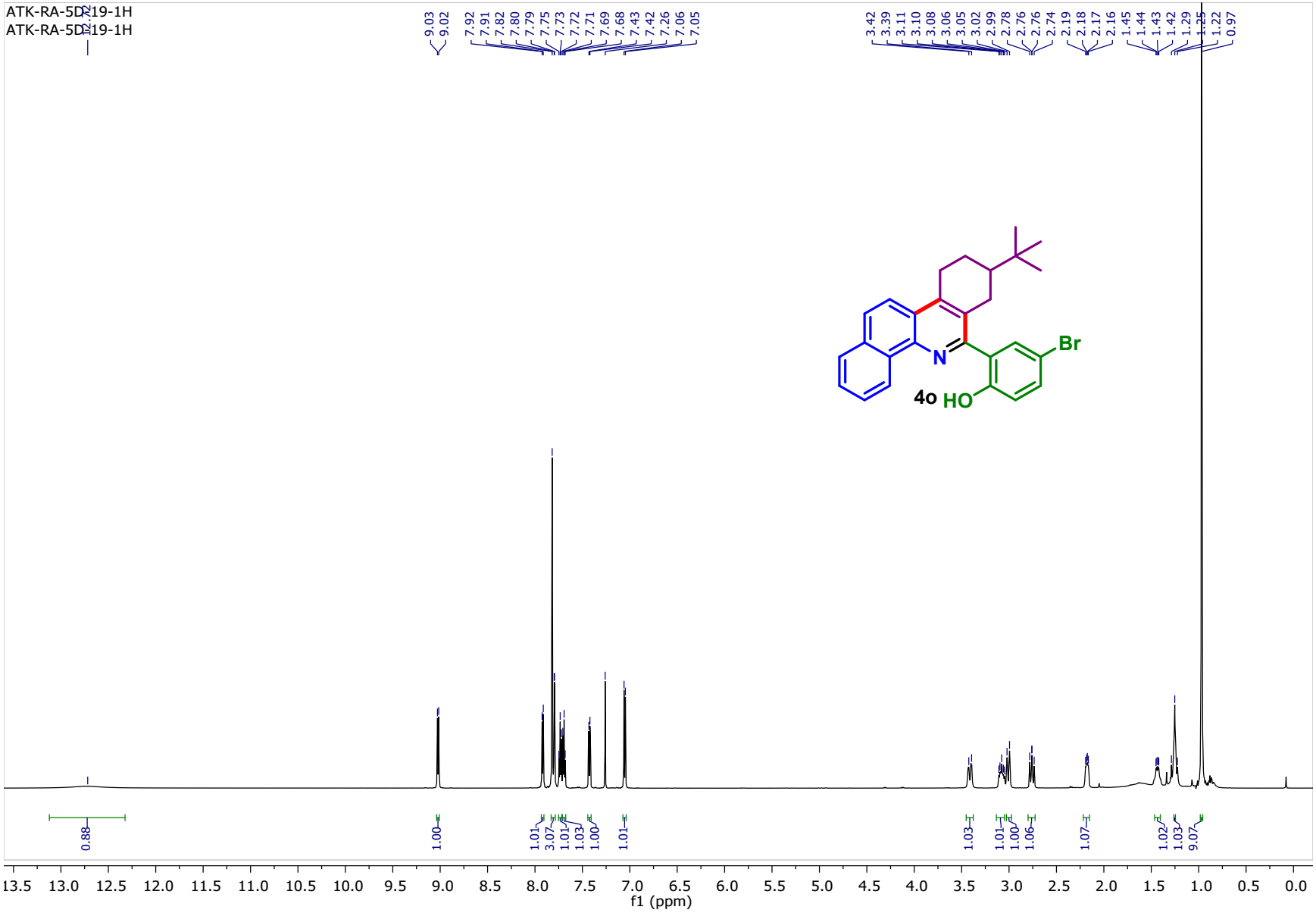


# HRMS Spectra of 4n

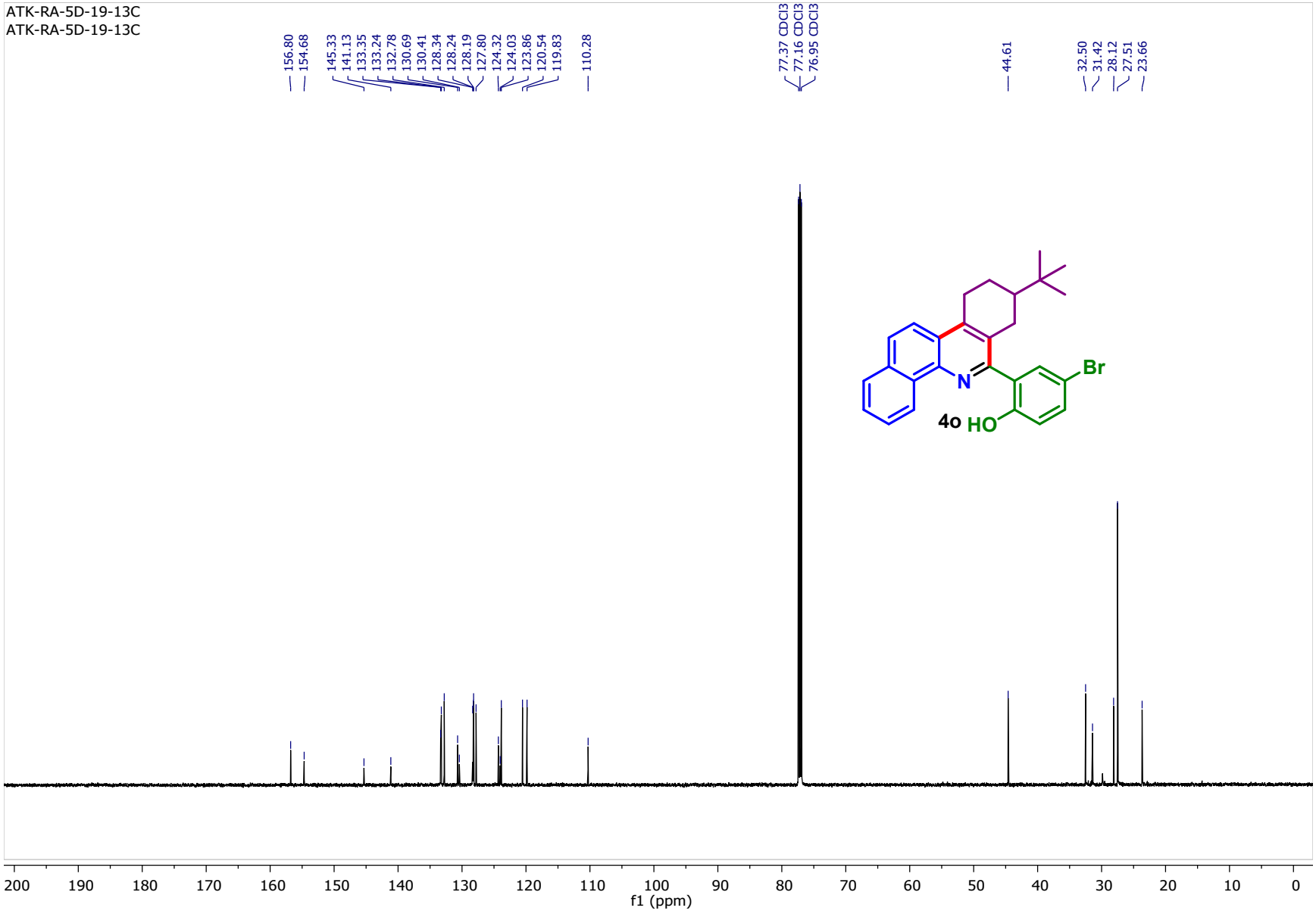
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SAMPLE 17 | **Position** | P1-B5 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-G2.d |
| **ACQ Method** | ESI ALS 100-500.m | **Comment** | | **Acquired Time** | 21-Apr-21 10:35:35 PM (UTC+05:30) |



## $^{1}$H NMR Spectra of 4o

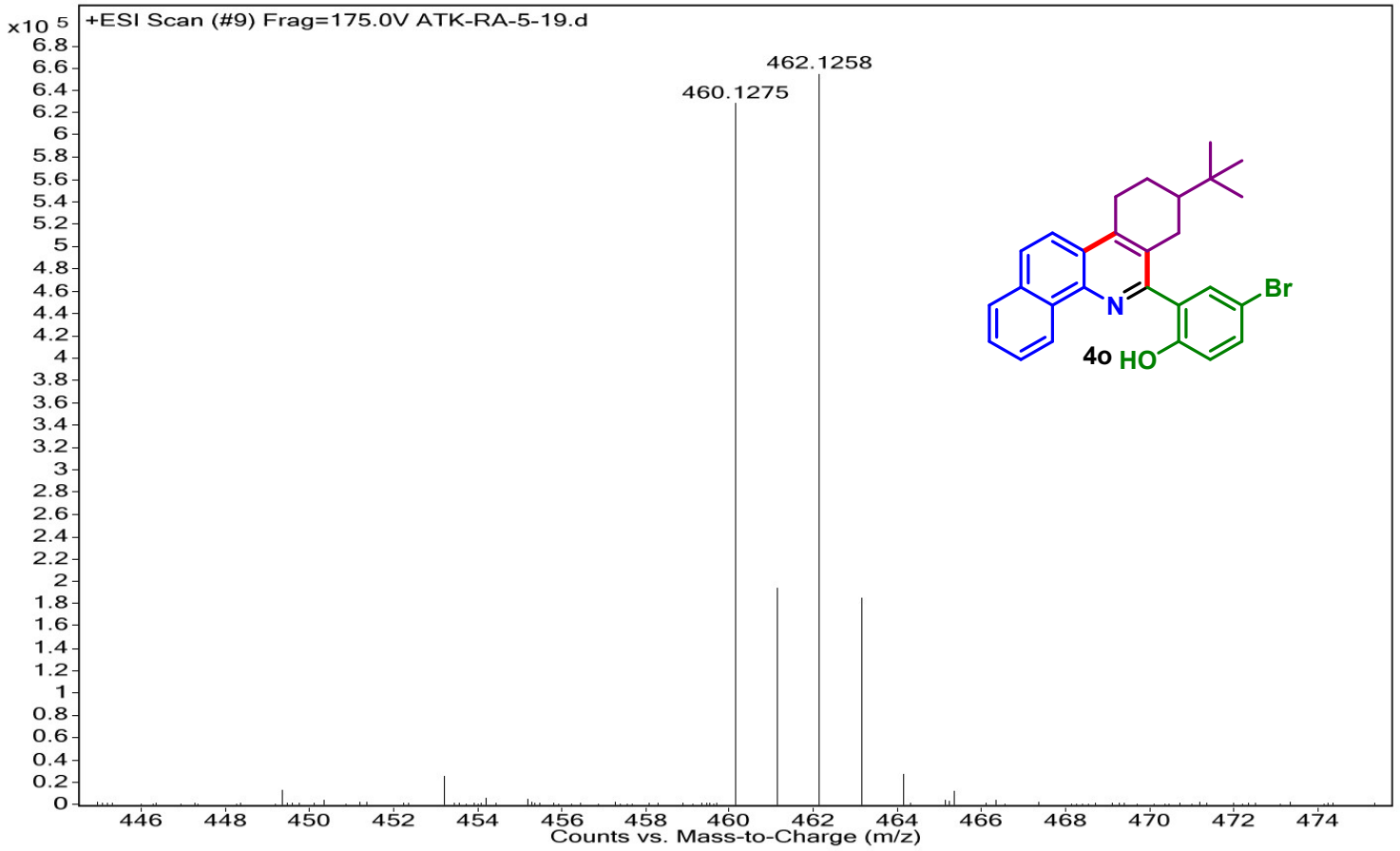


## $^{13}$C NMR Spectra of 4o

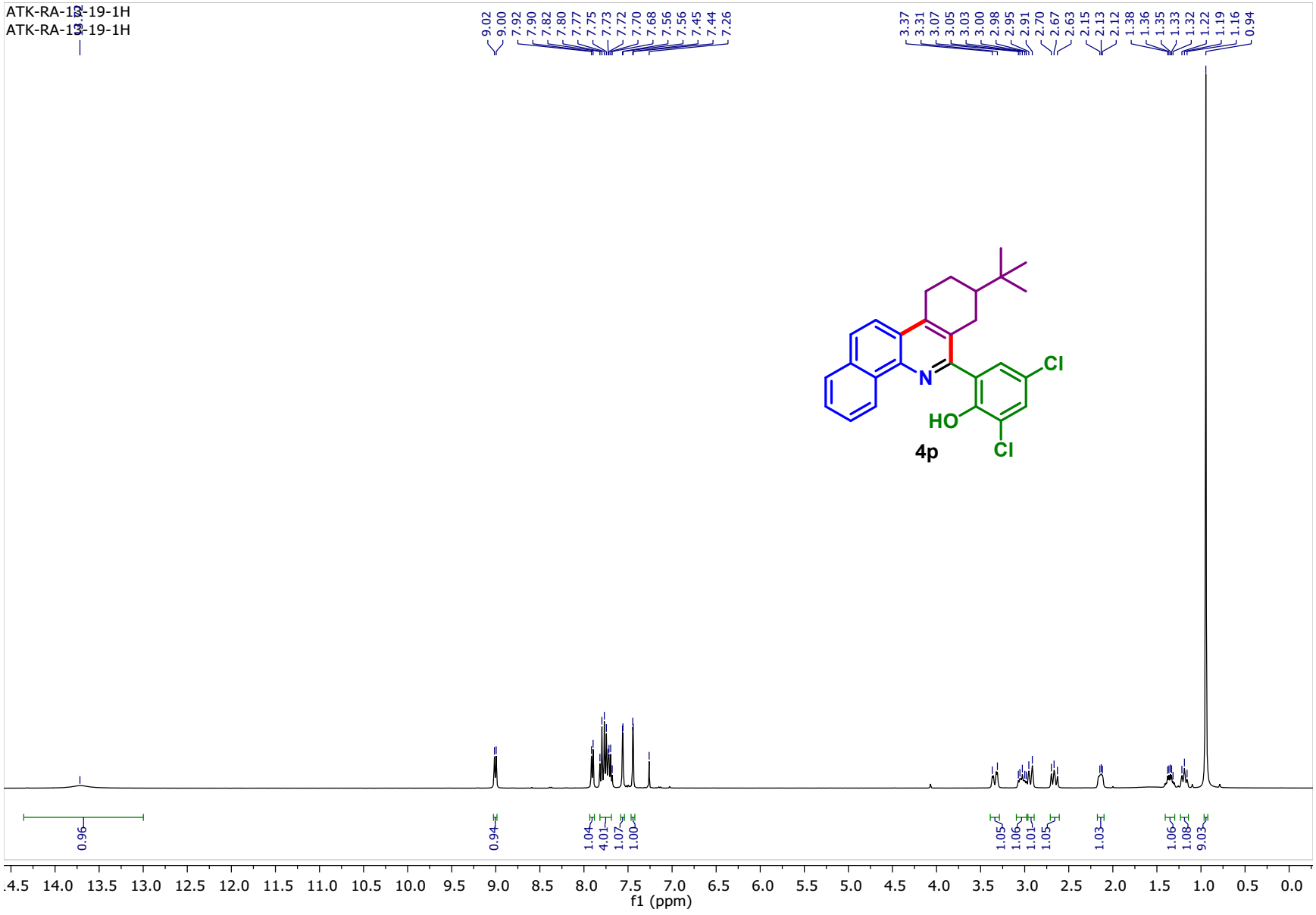


# HRMS Spectra of 4o

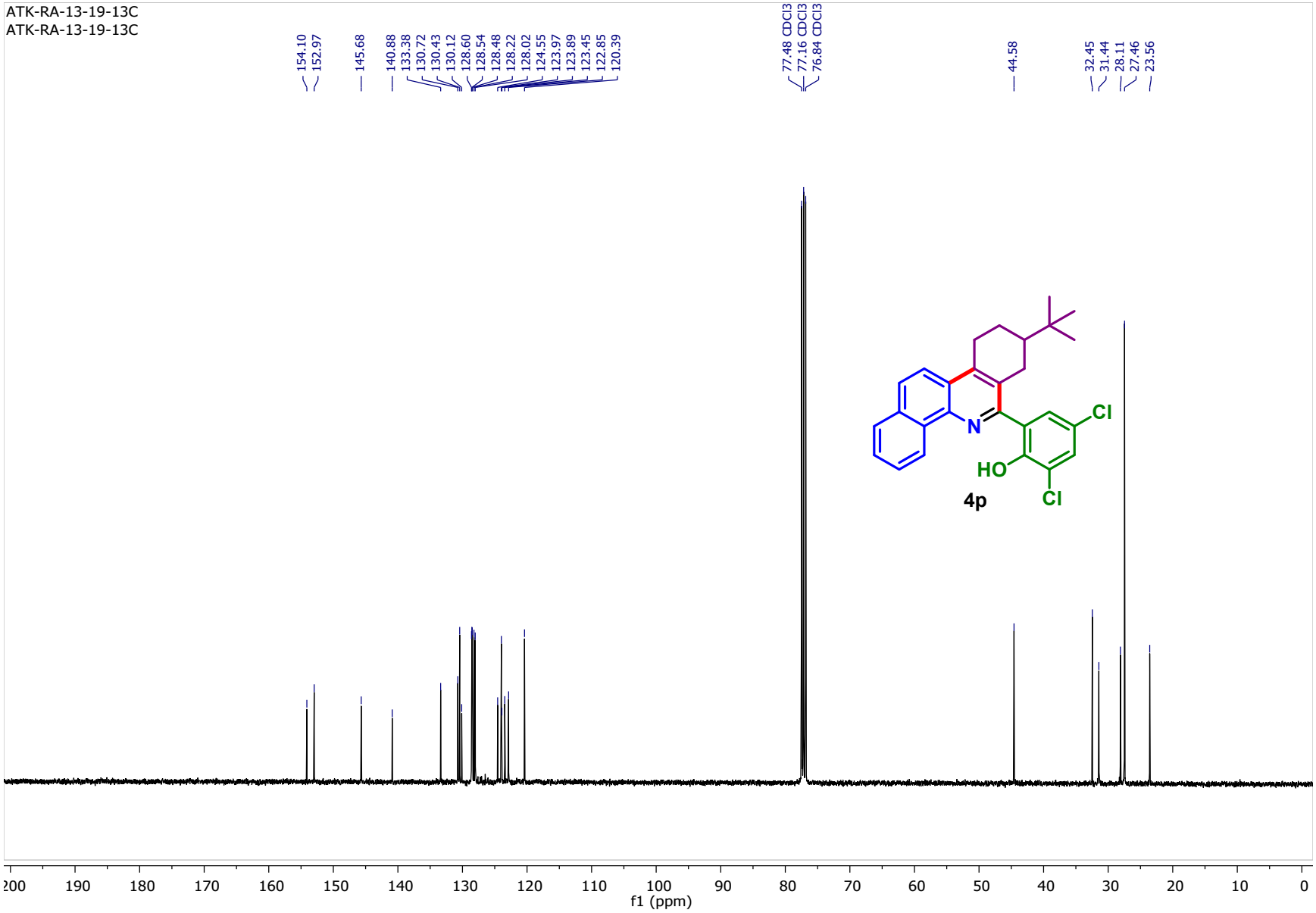
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Sample Name** | SAMPLE | **Position** | P2-B8 | **Instrument Name** | Instrument 1 | **User Name** | |
| **Inj Vol** | 20 | **InjPosition** | | **SampleType** | Sample | **IRM Calibration Status** | Success |
| **Data Filename** | ATK-RA-5-19.d | **ACQ Method** | ESI ALS 100-600.m | **Comment** | | **Acquired Time** | 2/5/2020 4:57:15 PM |



## $^{1}$H NMR Spectra of 4p

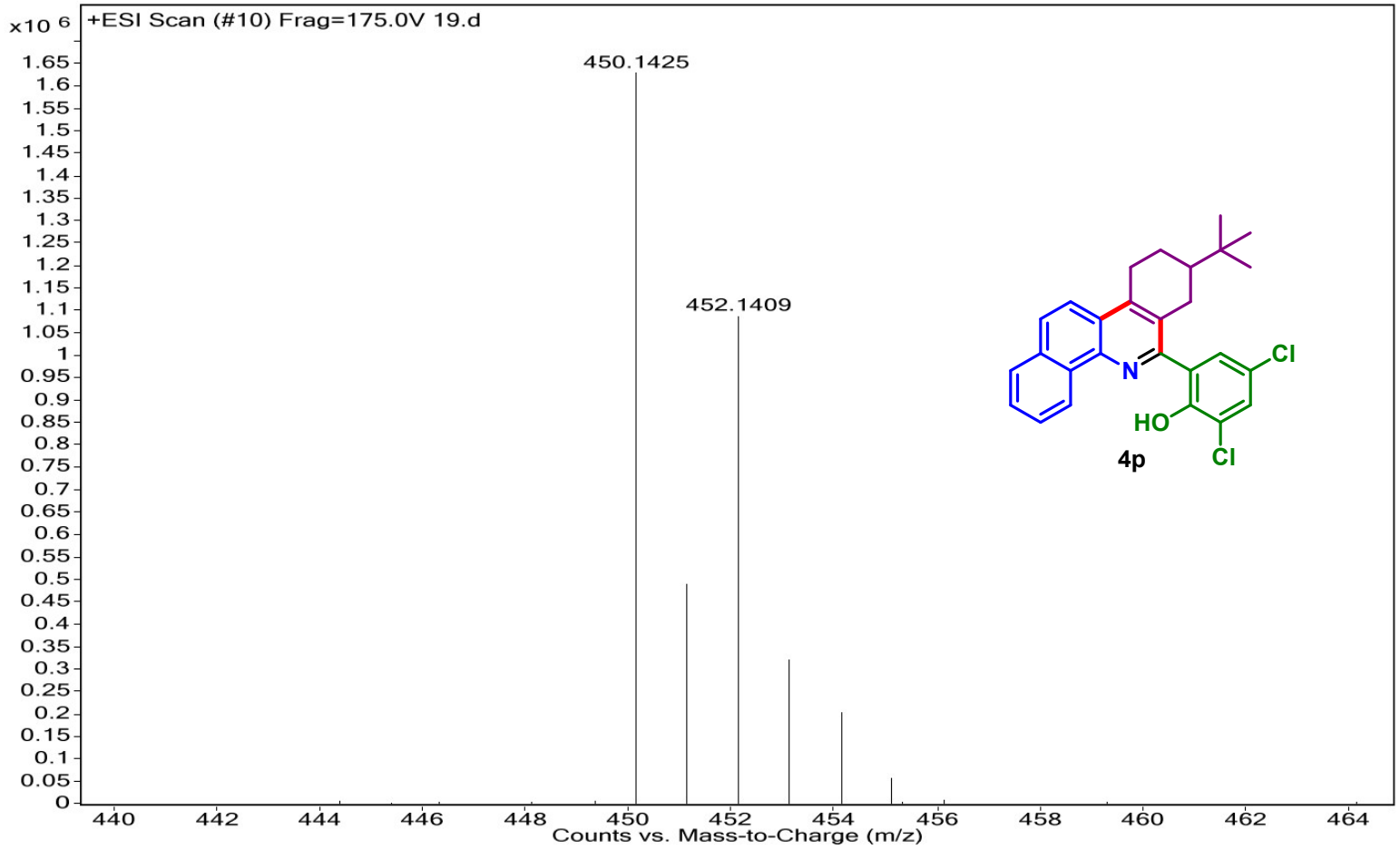


## $^{13}$C NMR Spectra of 4p

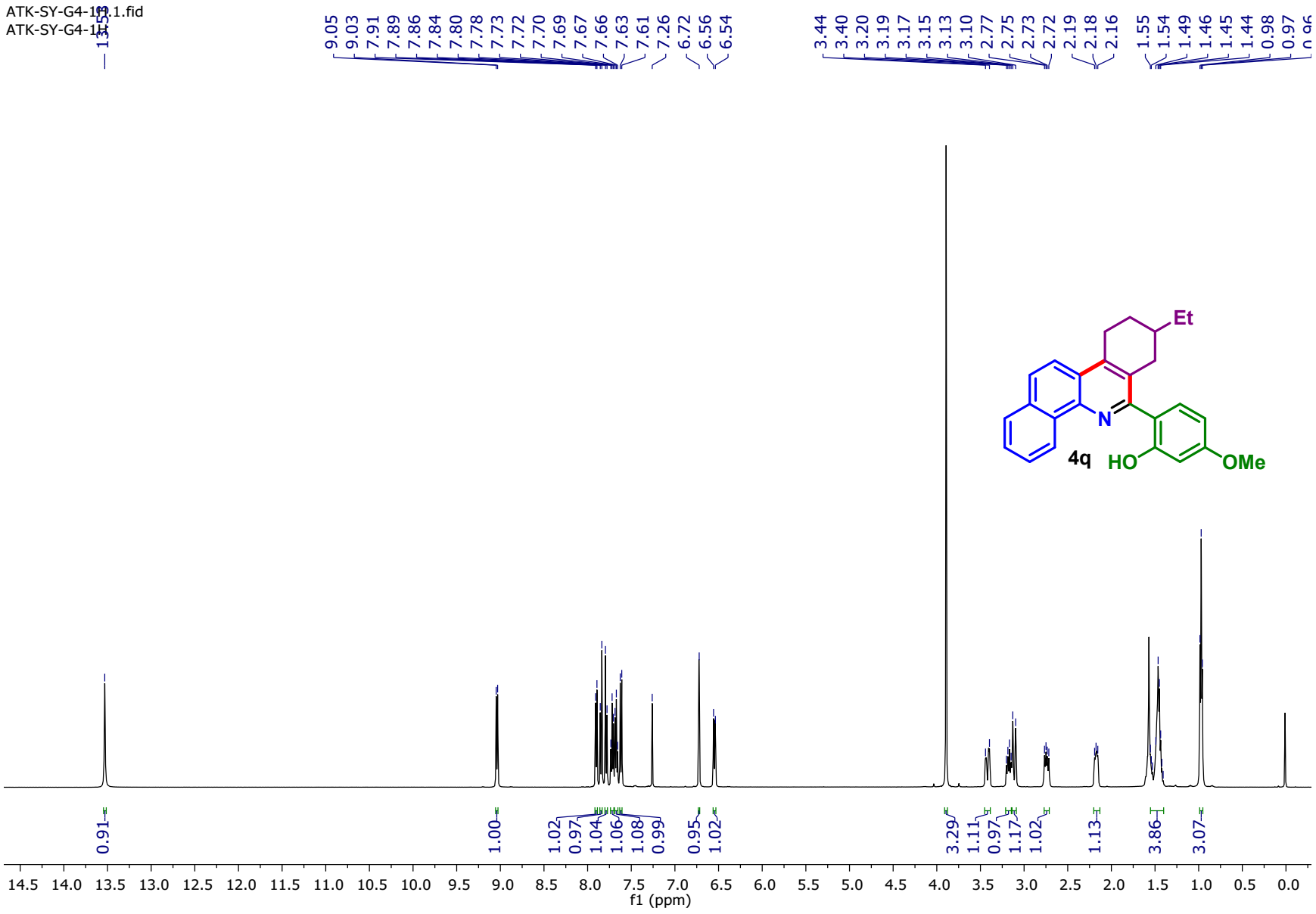


# HRMS Spectra of 4p

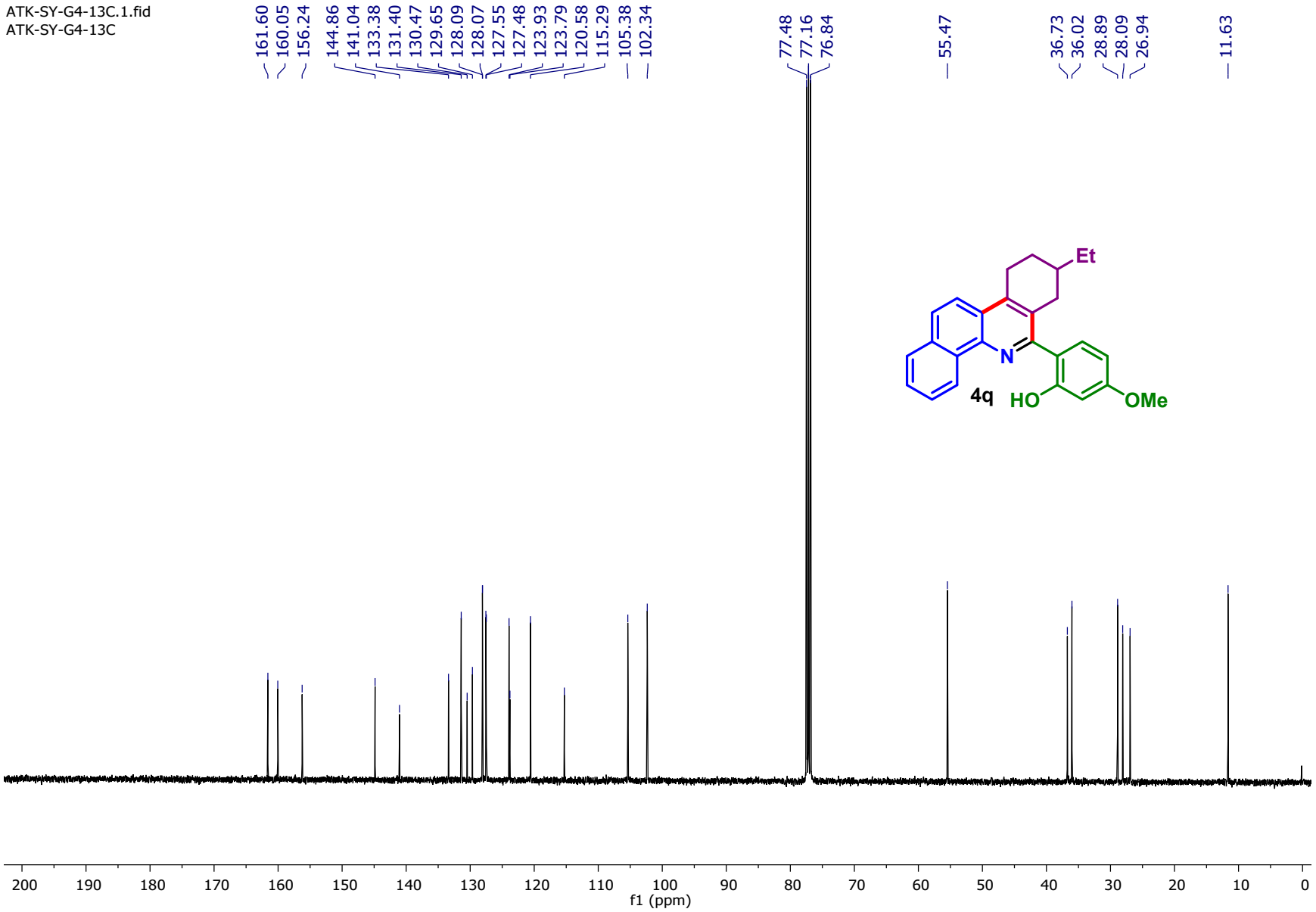
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Sample Name** | WASH | **Position** | P2-C1 | **Instrument Name** | Instrument 1 | **User Name** | |
| **Inj Vol** | 20 | **InjPosition** | | **SampleType** | Sample | **IRM Calibration Status** | Success |
| **Data Filename** | 19.d | **ACQ Method** | ESI ALS 100-600.m | **Comment** | | **Acquired Time** | 4/5/2019 11:24:24 AM |



## $^{1}$H NMR Spectra of 4q

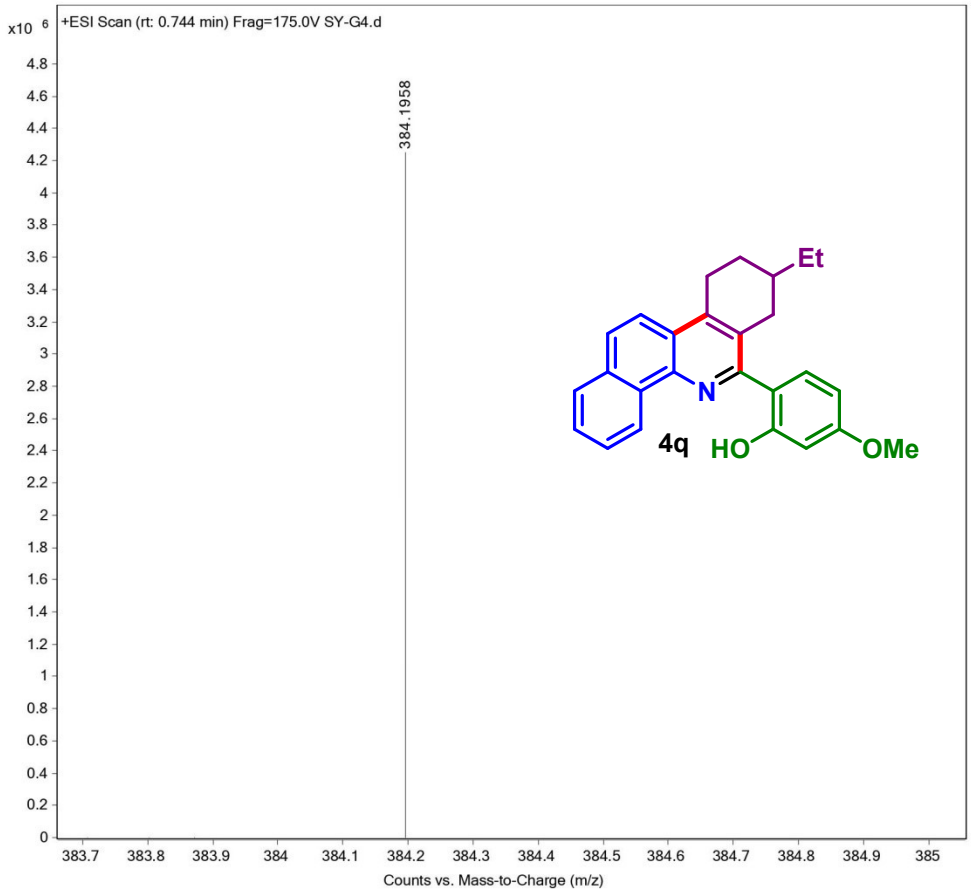


## $^{13}$C NMR Spectra of 4q

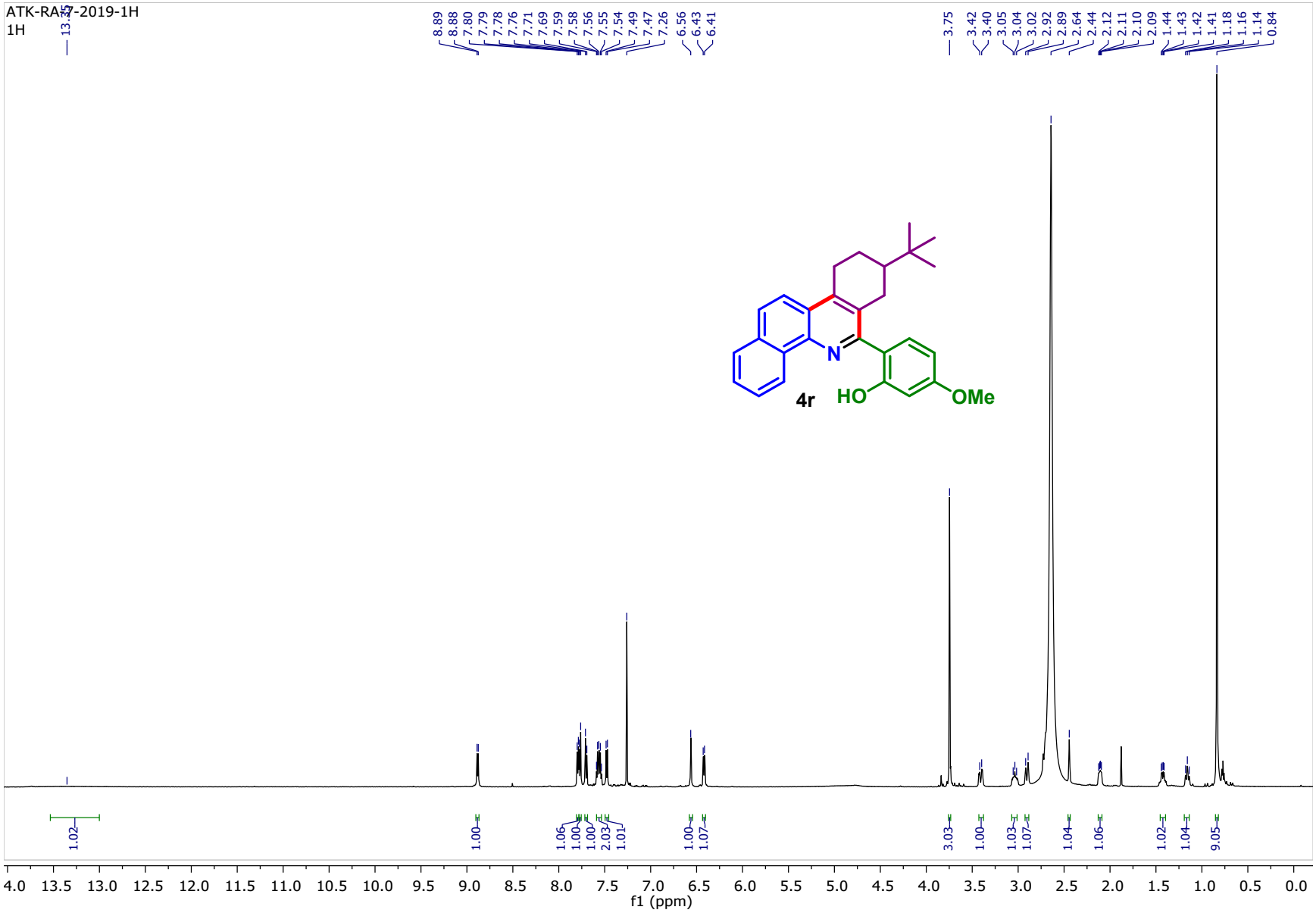


# HRMS Spectra of 4q

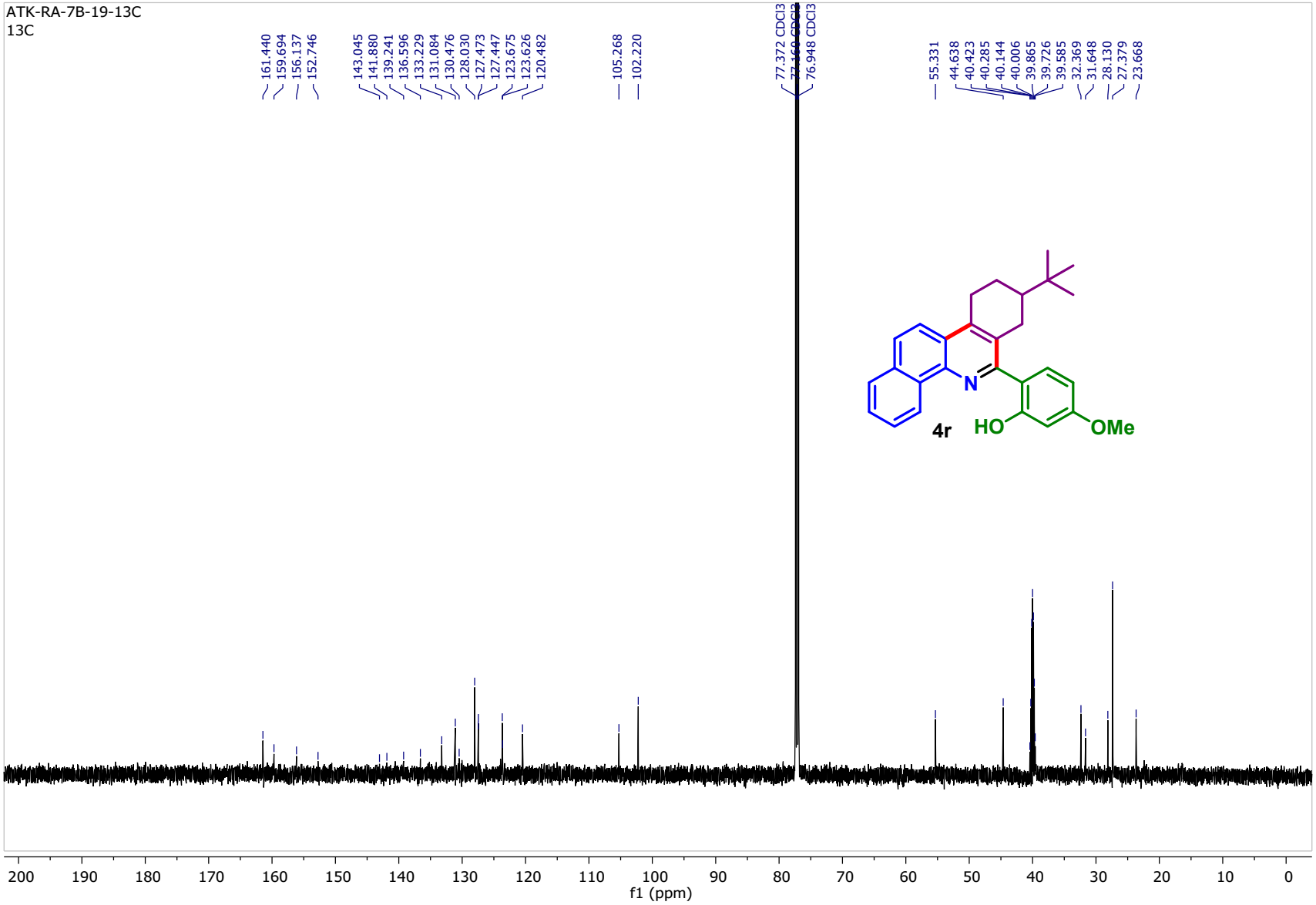
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SAMPLE 18 | **Position** | P1-B6 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-G4.d |
| **ACQ Method** | ESI ALS 100-500.m | **Comment** | | **Acquired Time** | 21-Apr-21 10:43:19 PM (UTC+05:30) |



## $^{1}$H NMR Spectra of 4r

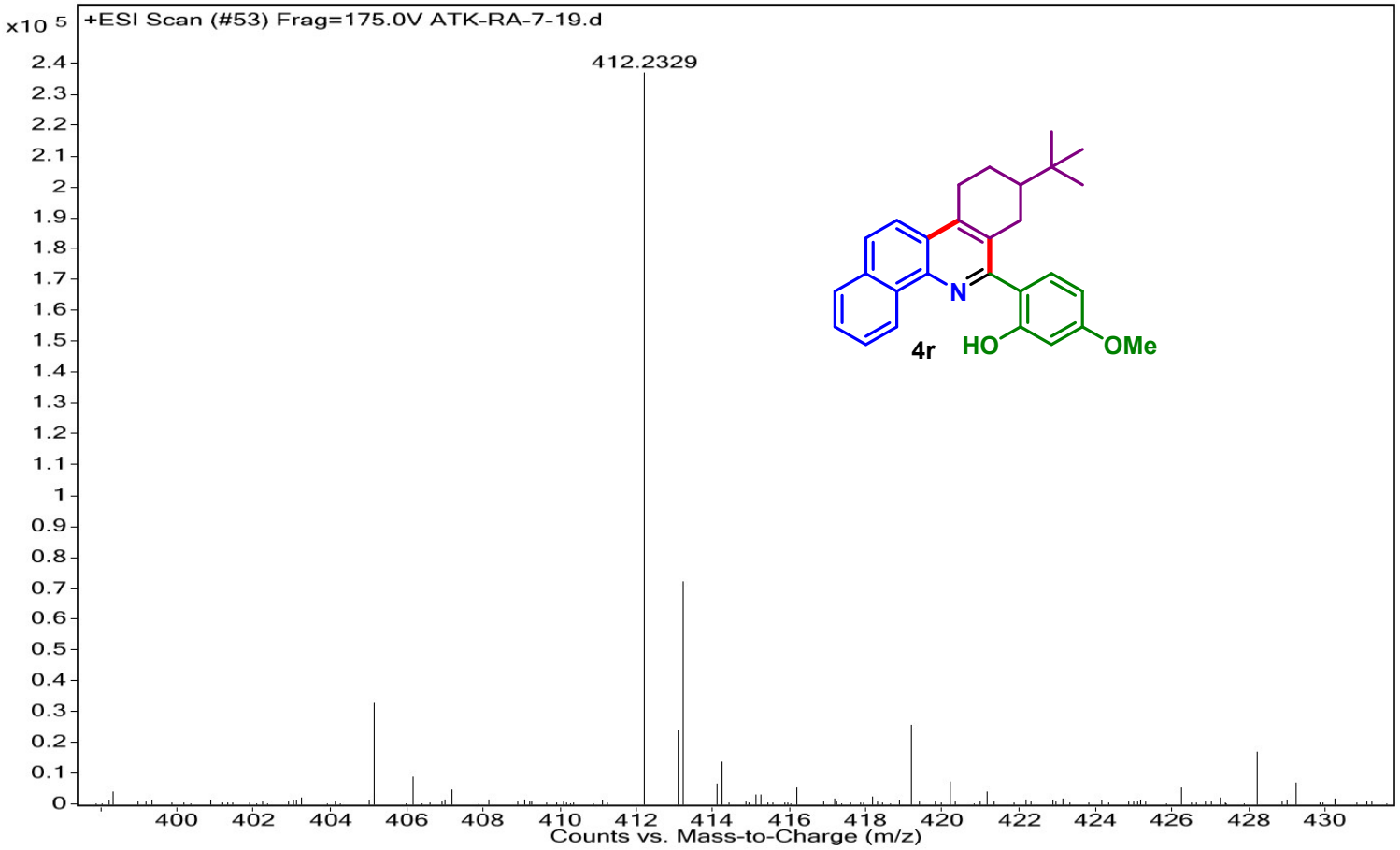


## $^{13}$C NMR Spectra of 4r

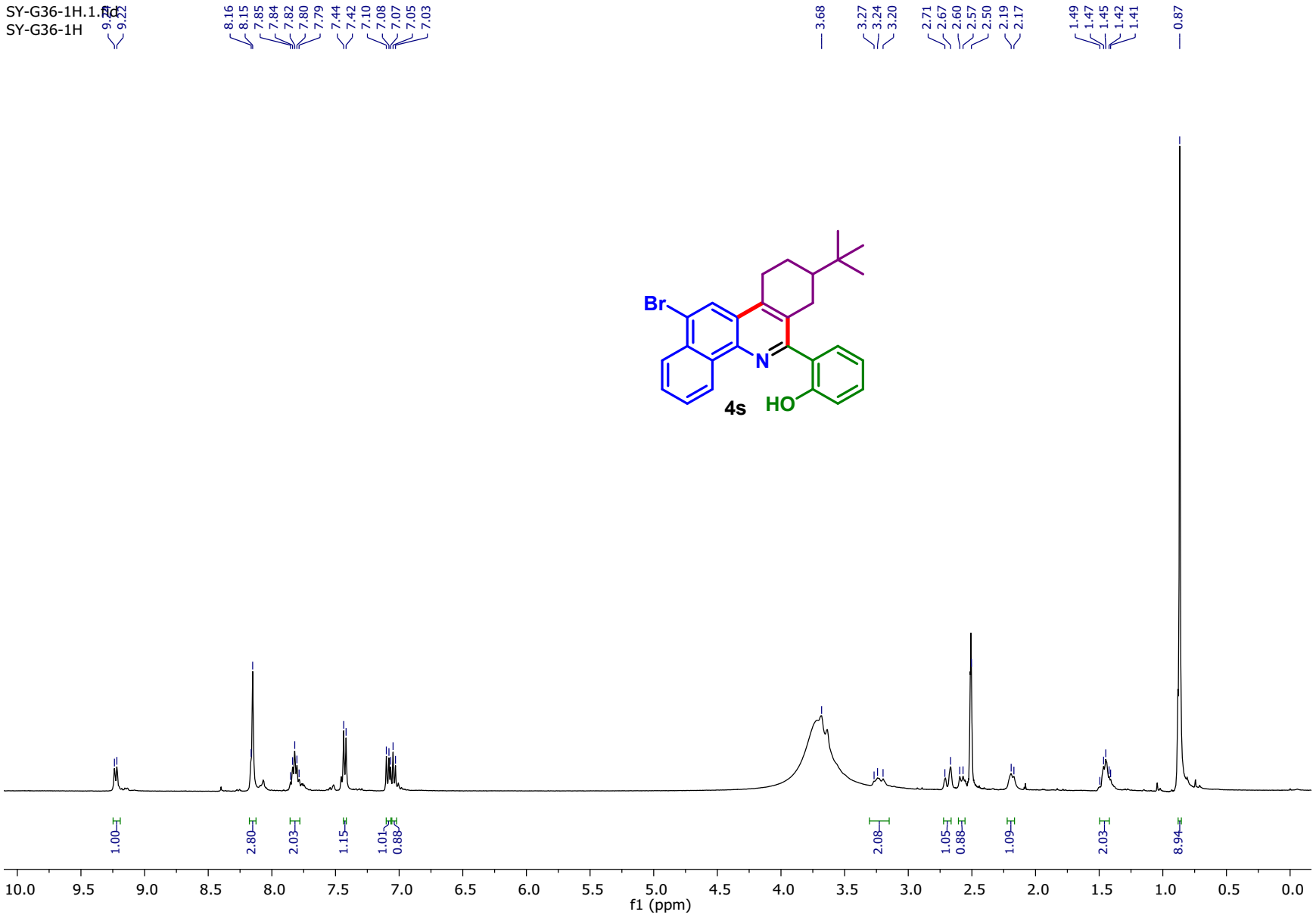


# HRMS Spectra of 4r

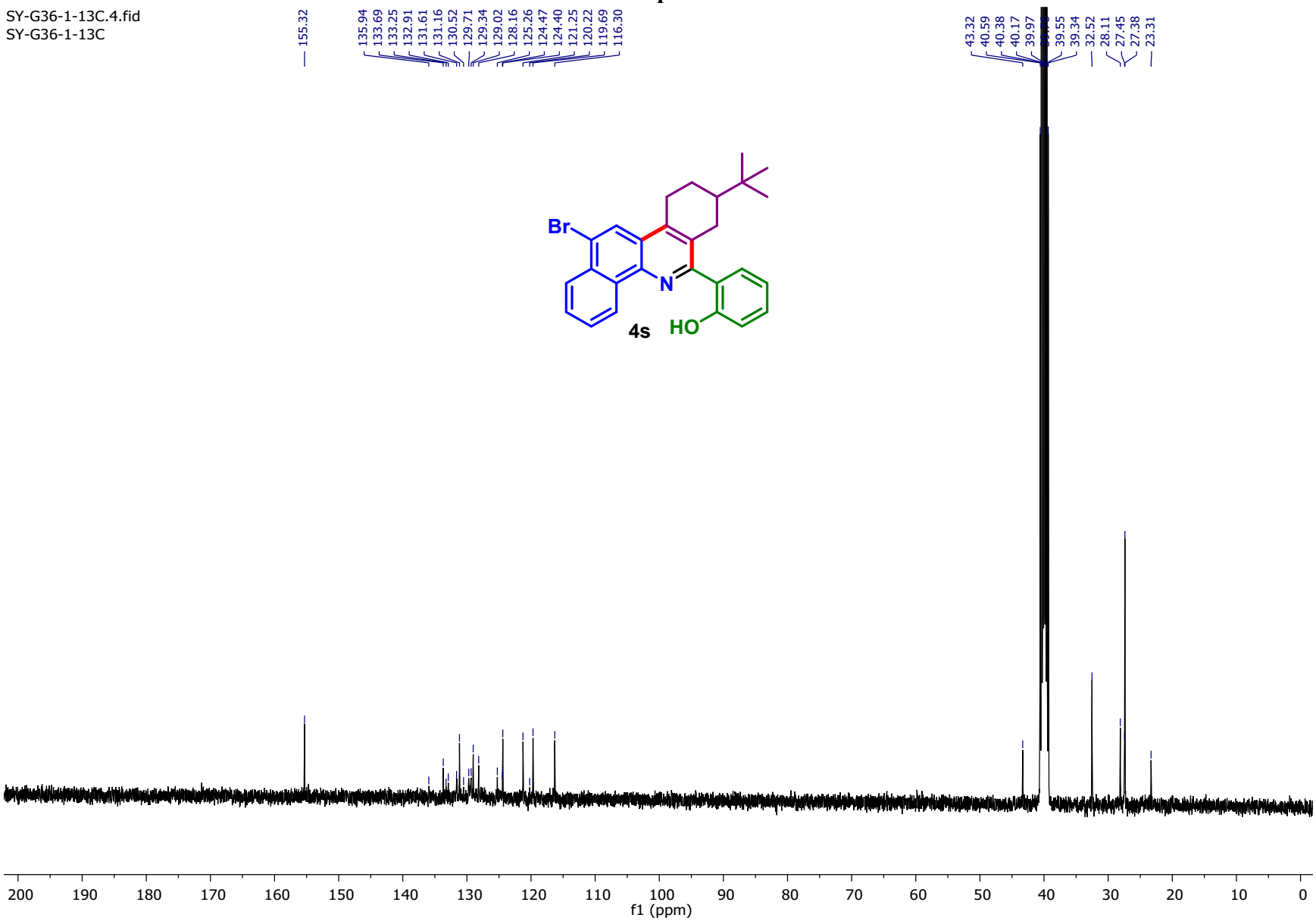
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Sample Name** | SAMPLE-6 | **Position** | P1-A7 | **Instrument Name** | Instrument 1 | **User Name** | |
| **Inj Vol** | 20 | **InjPosition** | | **SampleType** | Sample | **IRM Calibration Status** | Success |
| **Data Filename** | ATK-RA-7-19.d | **ACQ Method** | ESI ALS 100-600.m | **Comment** | | **Acquired Time** | 1/18/2019 4:13:51 PM |



## $^{1}$H NMR Spectra of 4s

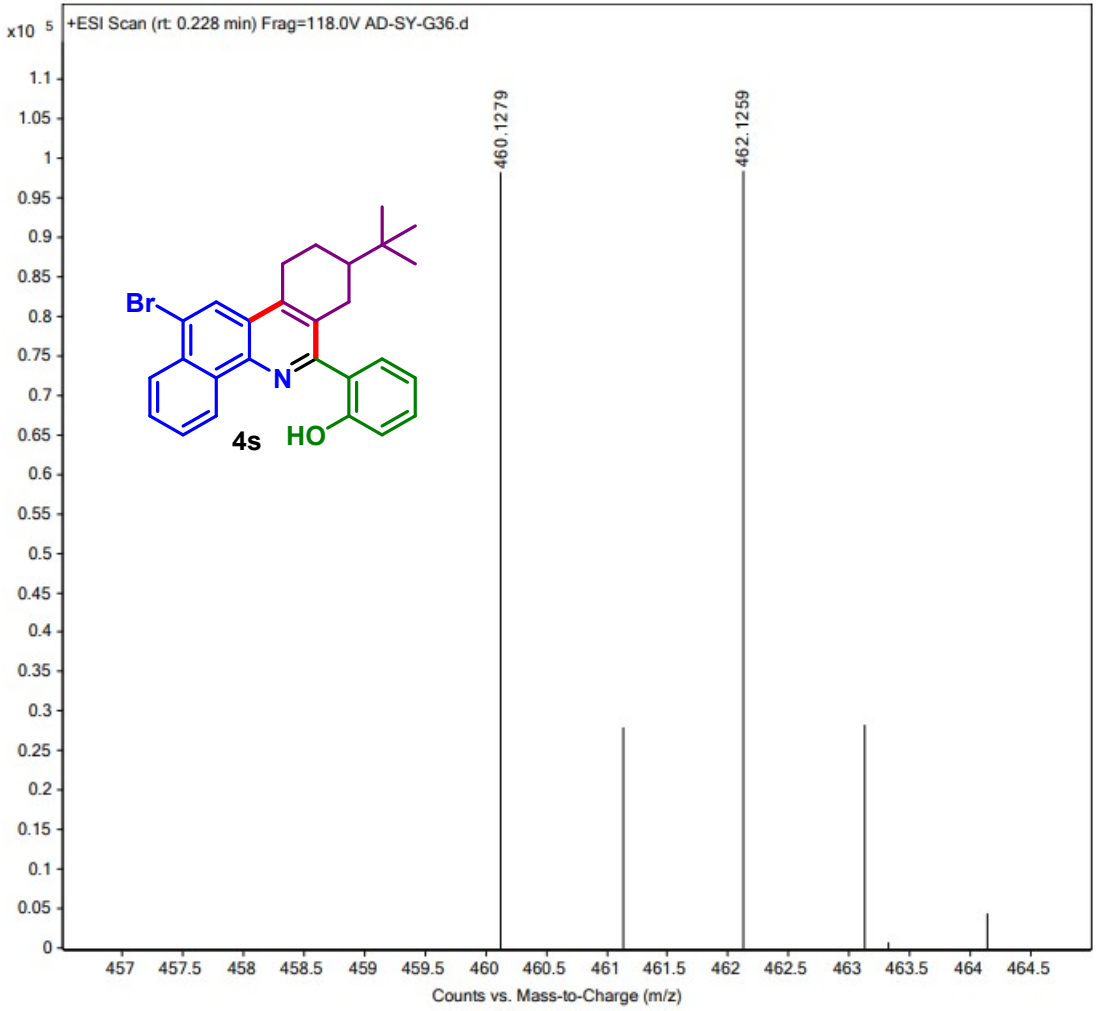


## $^{13}$C NMR Spectra of 4s

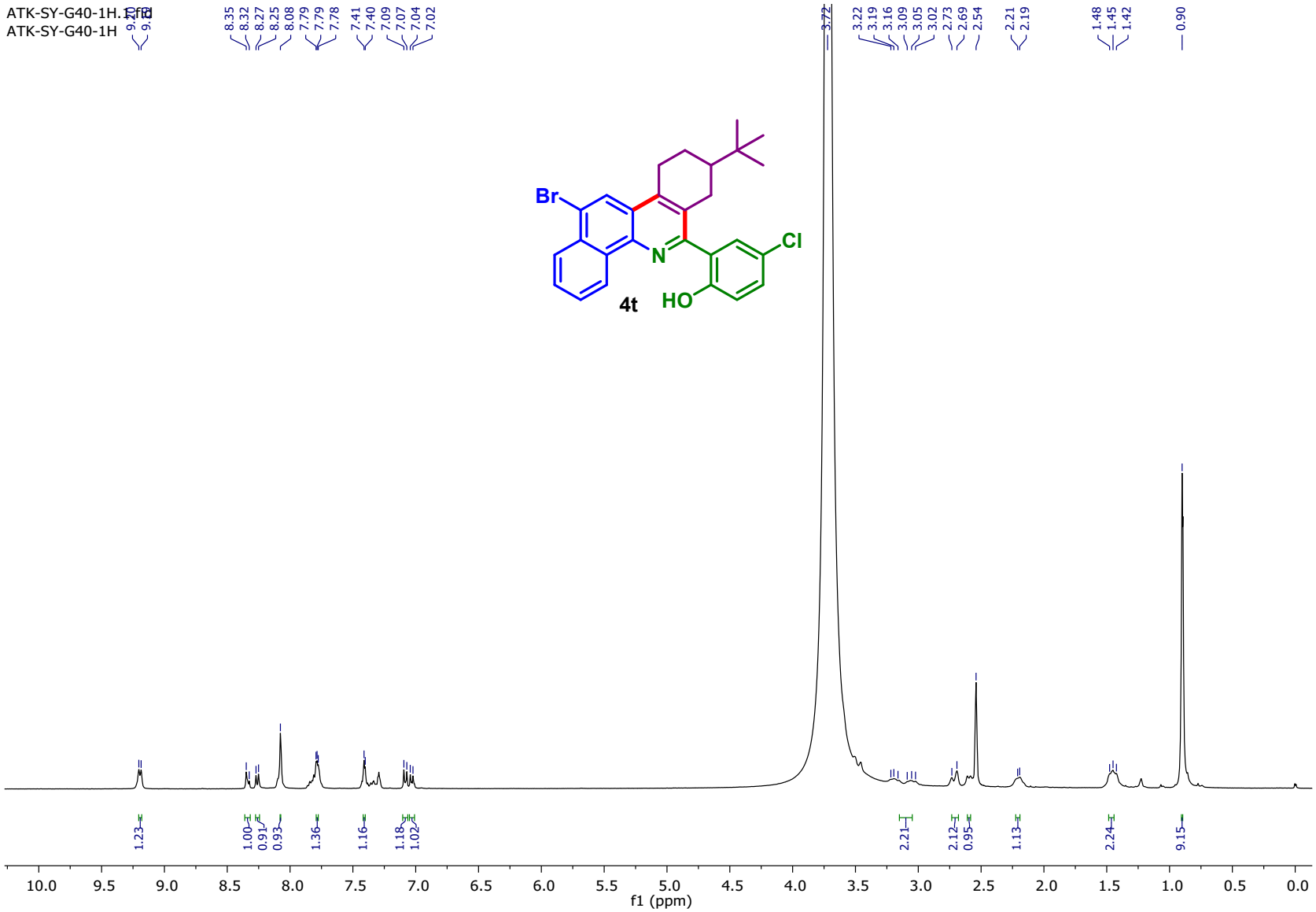


# HRMS Spectra of 4s

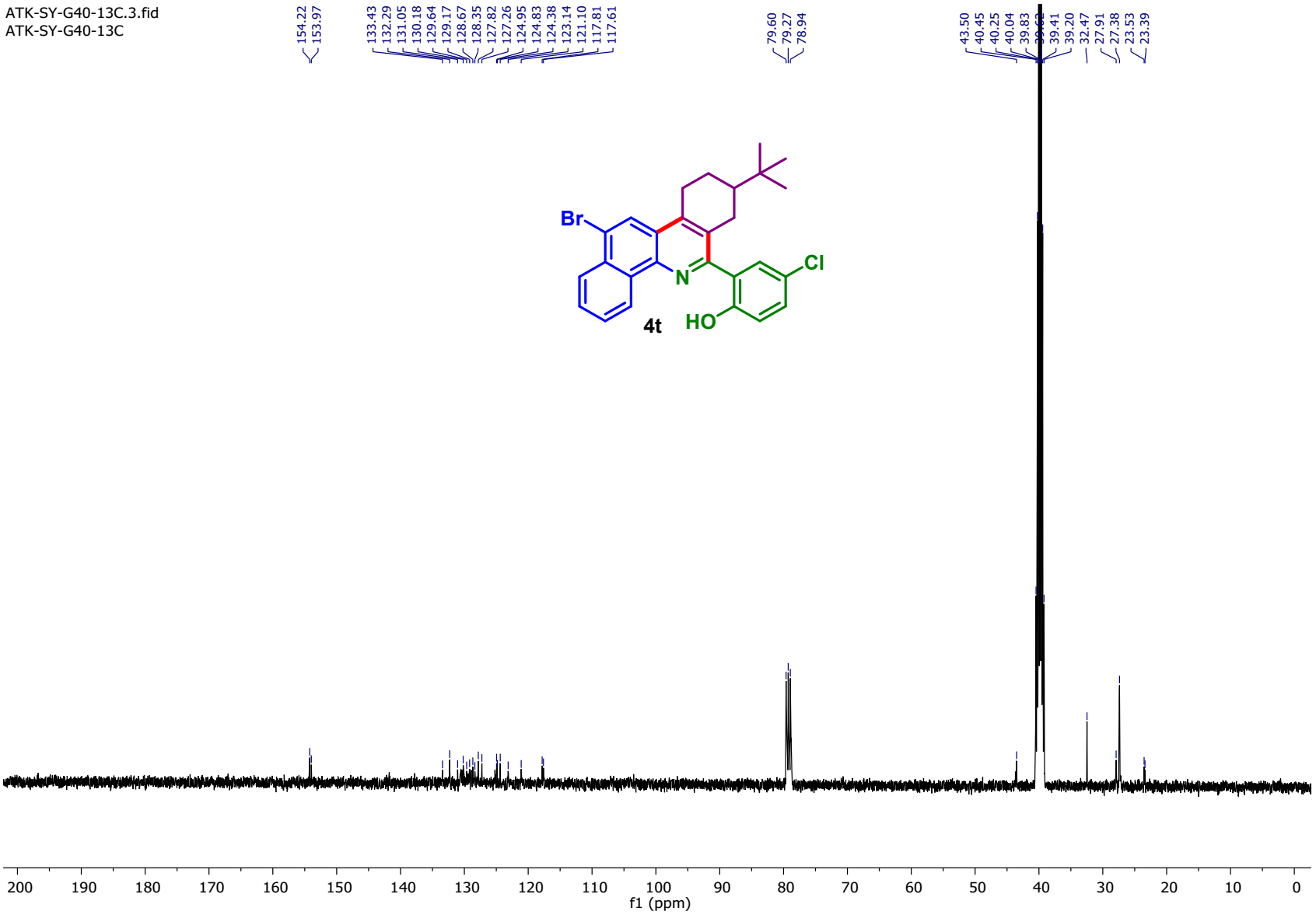
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | AD-SY-G36 | **Position** | Vial 21 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 0.1 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Some Ions Missed | **Data Filename** | AD-SY-G36.d |
| **ACQ Method** | Direct Mass-2017.m | **Comment** | | **Acquired Time** | 28-03-2022 21:00:07 (UTC+05:30) |



## $^{1}$H NMR Spectra of 4t

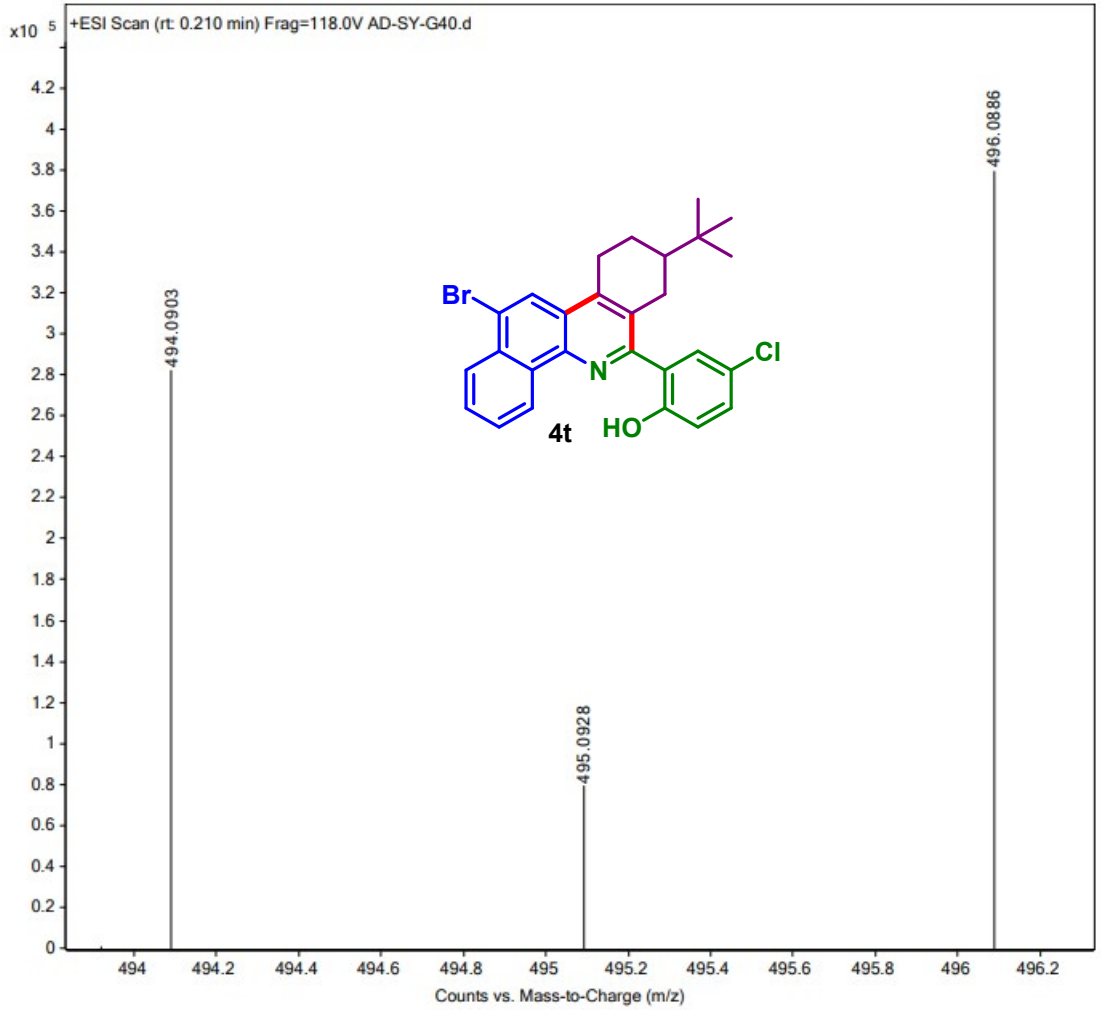


## $^{13}$C NMR Spectra of 4t

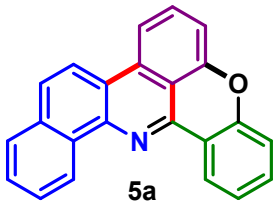


# HRMS Spectra of 4t

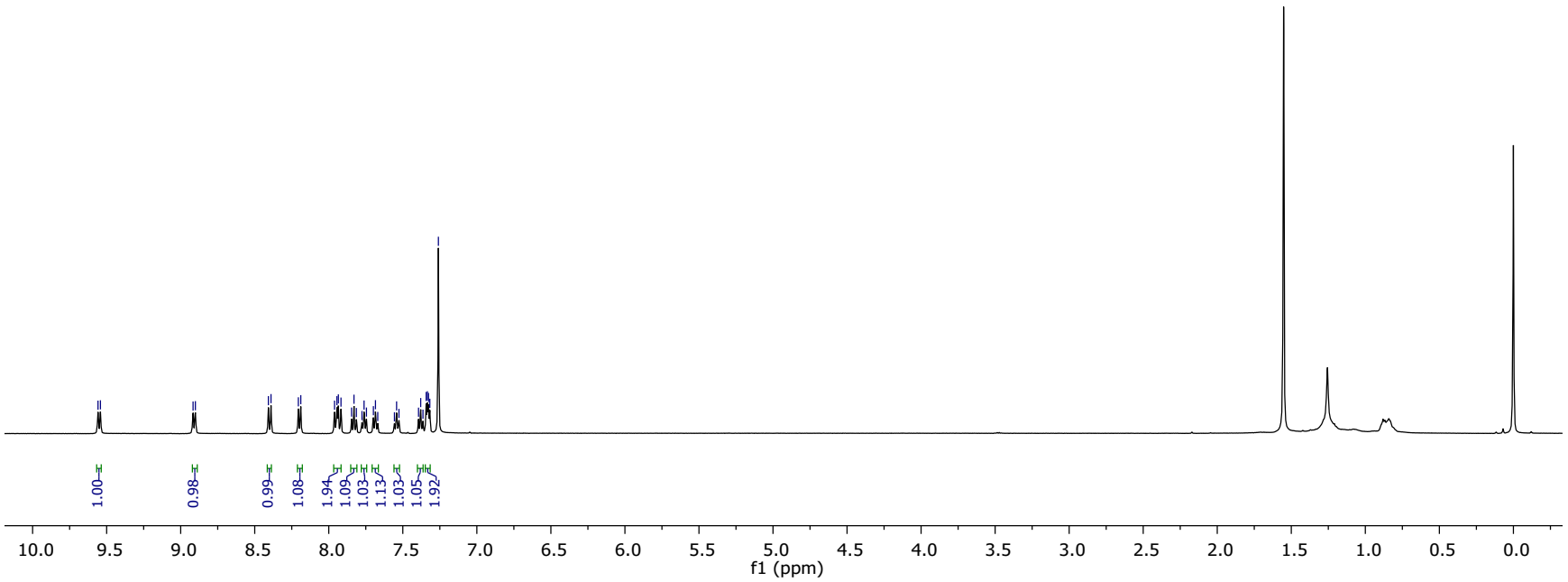
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | AD-SY-G40 | **Position** | Vial 23 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 0.1 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Some Ions Missed | **Data Filename** | AD-SY-G40.d |
| **ACQ Method** | Direct Mass-2017.m | **Comment** | | **Acquired Time** | 28-03-2022 21:07:19 (UTC+05:30) |



## $^{1}$H NMR Spectra of 5a

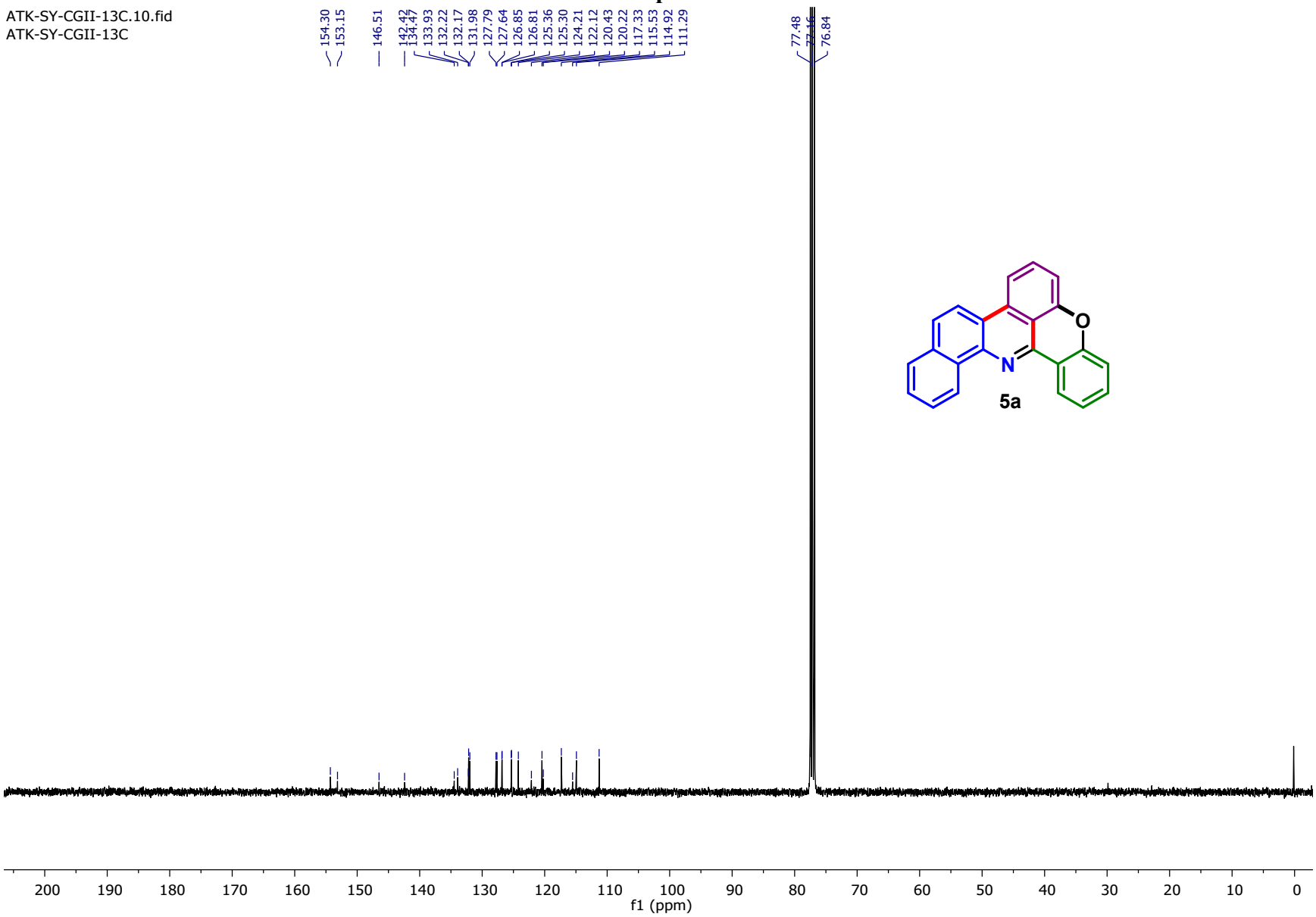




5a



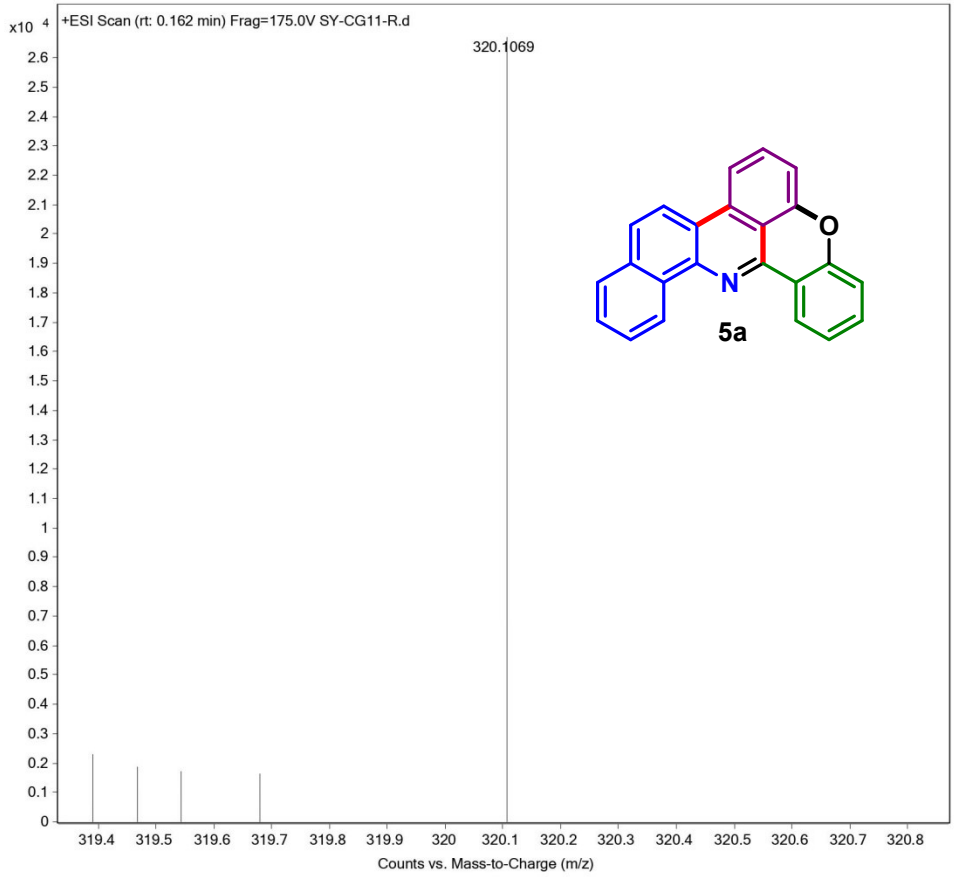
## $^{13}$C NMR Spectra of 5a

ATK-SY-CGII-13C.10.fid
ATK-SY-CGII-13C

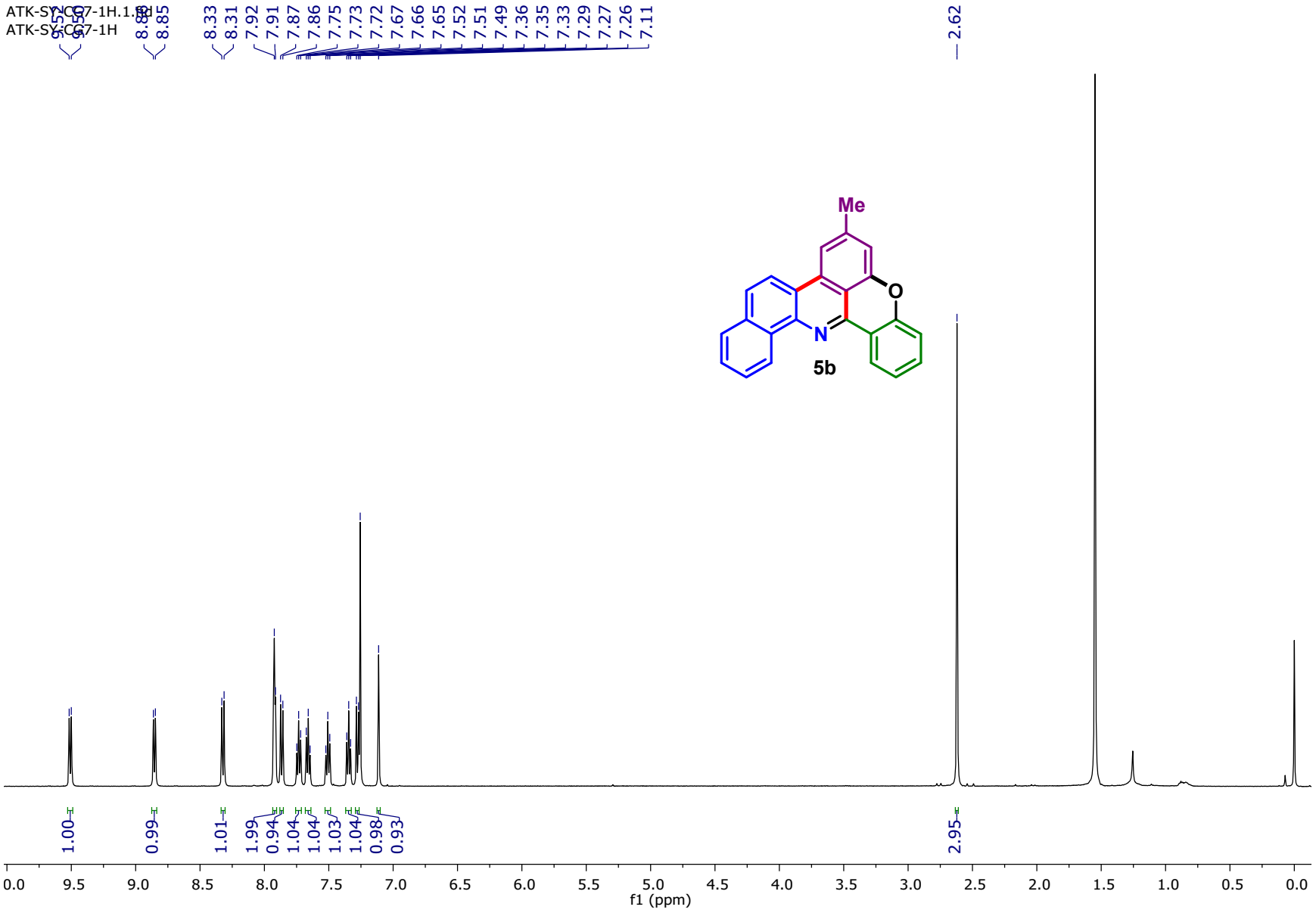


# HRMS Spectra of 5a

| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SAMPLE | **Position** | P2-C4 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-CG11-R.d |
| **ACQ Method** | ESI ALS 200-600.m | **Comment** | | **Acquired Time** | 30-Aug-21 04:35:33 PM (UTC+05:30) |

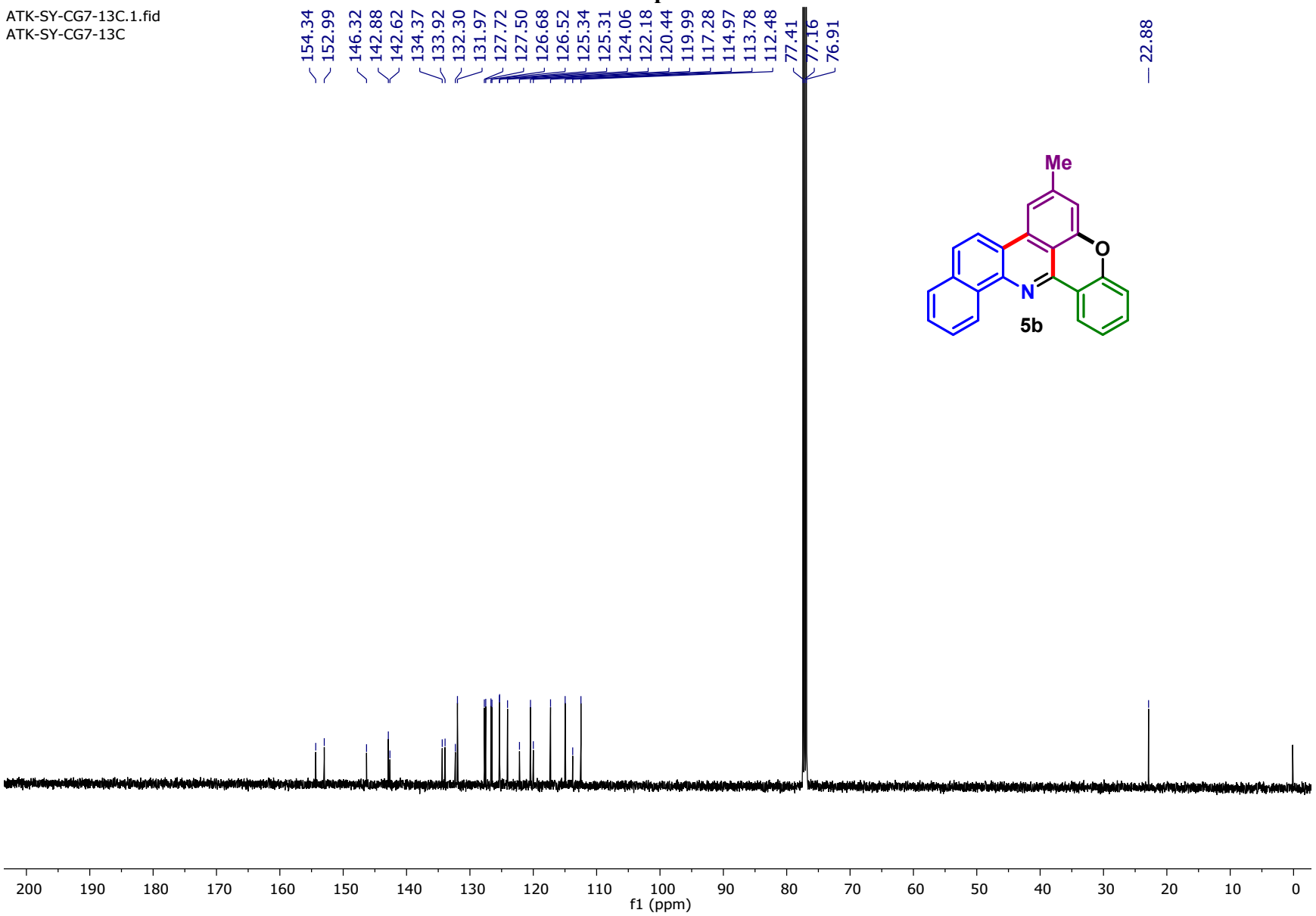


## $^{1}$H NMR Spectra of 5b



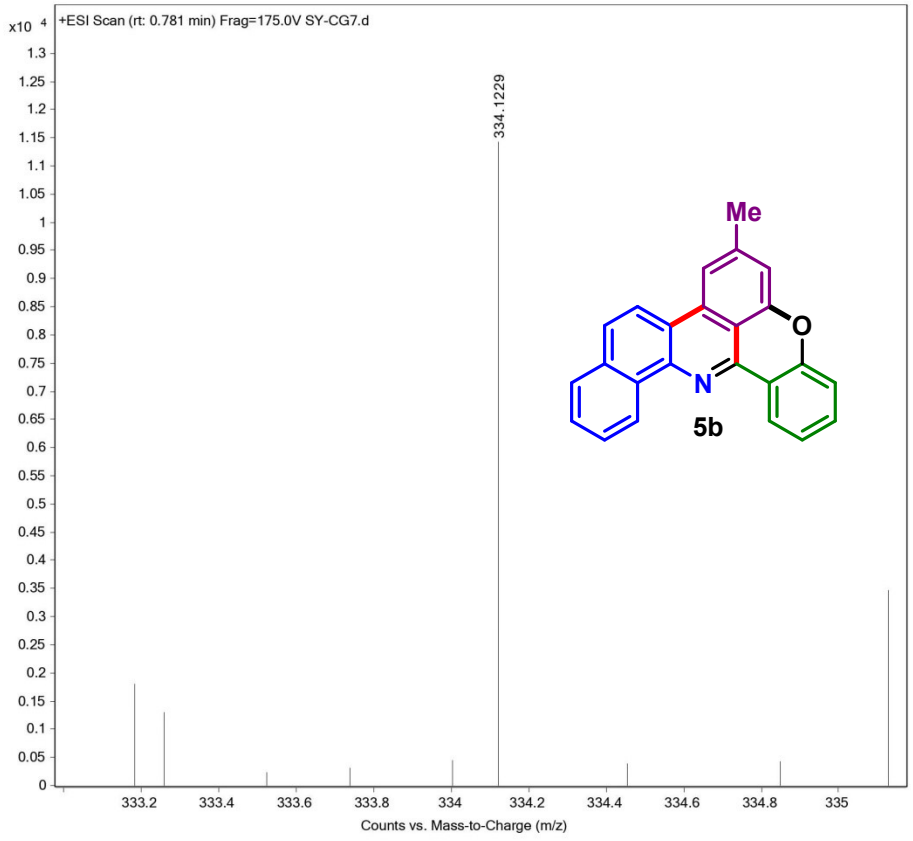
## $^{13}$C NMR Spectra of 5b

ATK-SY-CG7-13C.1.fid
ATK-SY-CG7-13C

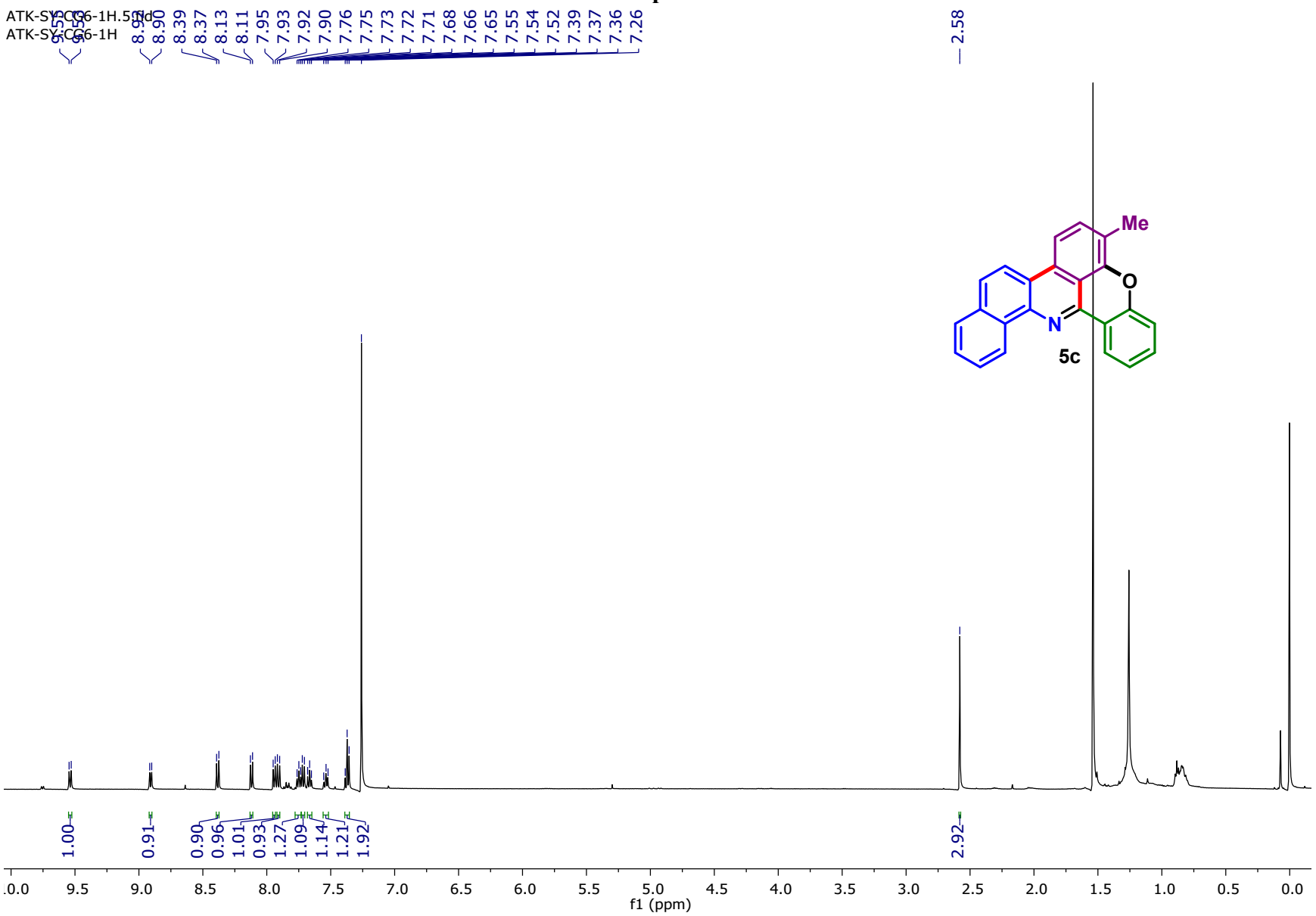


# HRMS Spectra of 5b

| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SY-CG7 | **Position** | P1-D5 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-CG7.d |
| **ACQ Method** | ESI ALS 100-1000.m | **Comment** | | **Acquired Time** | 05-May-21 04:43:07 PM (UTC+05:30) |

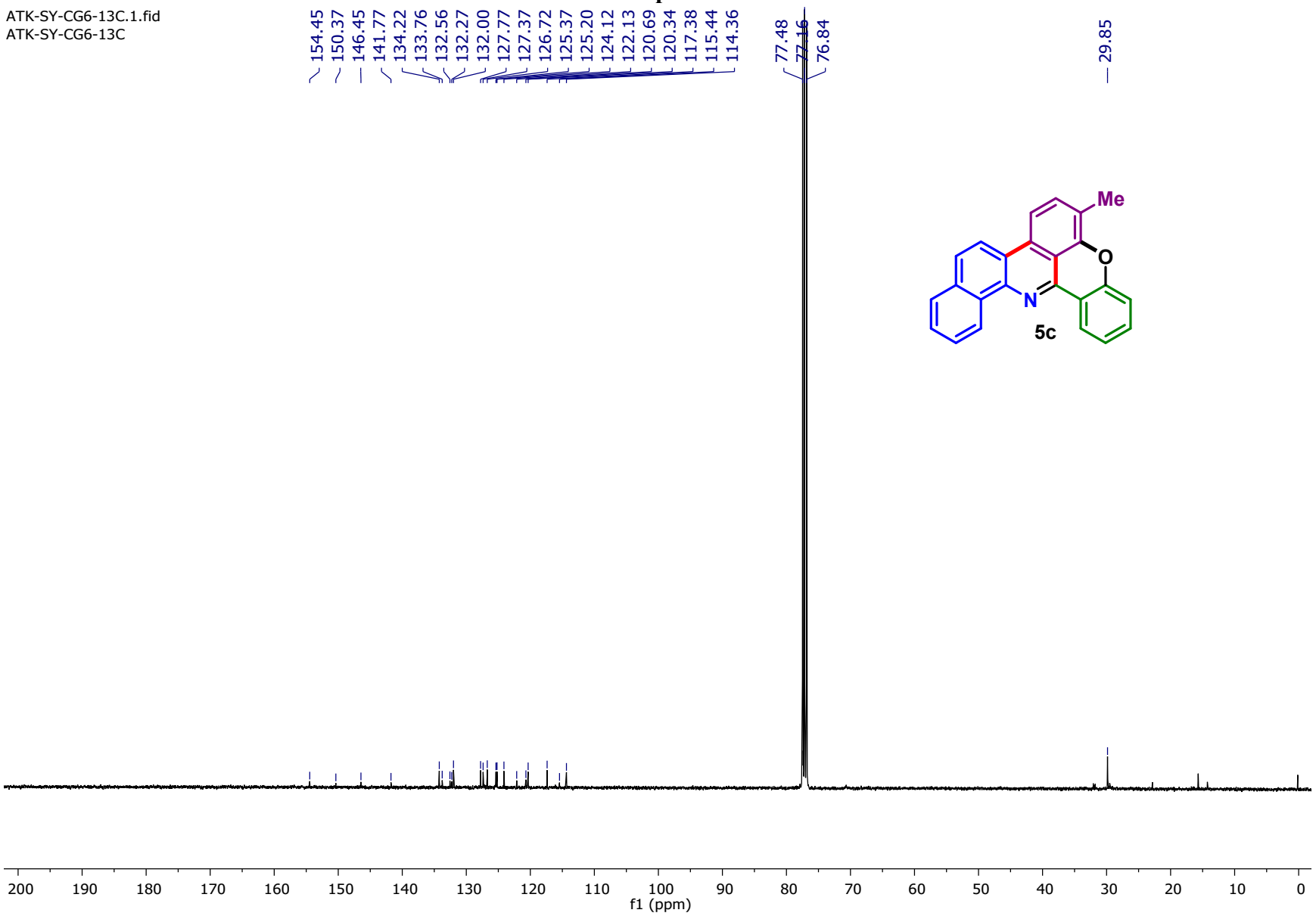


## $^{1}$H NMR Spectra of 5c



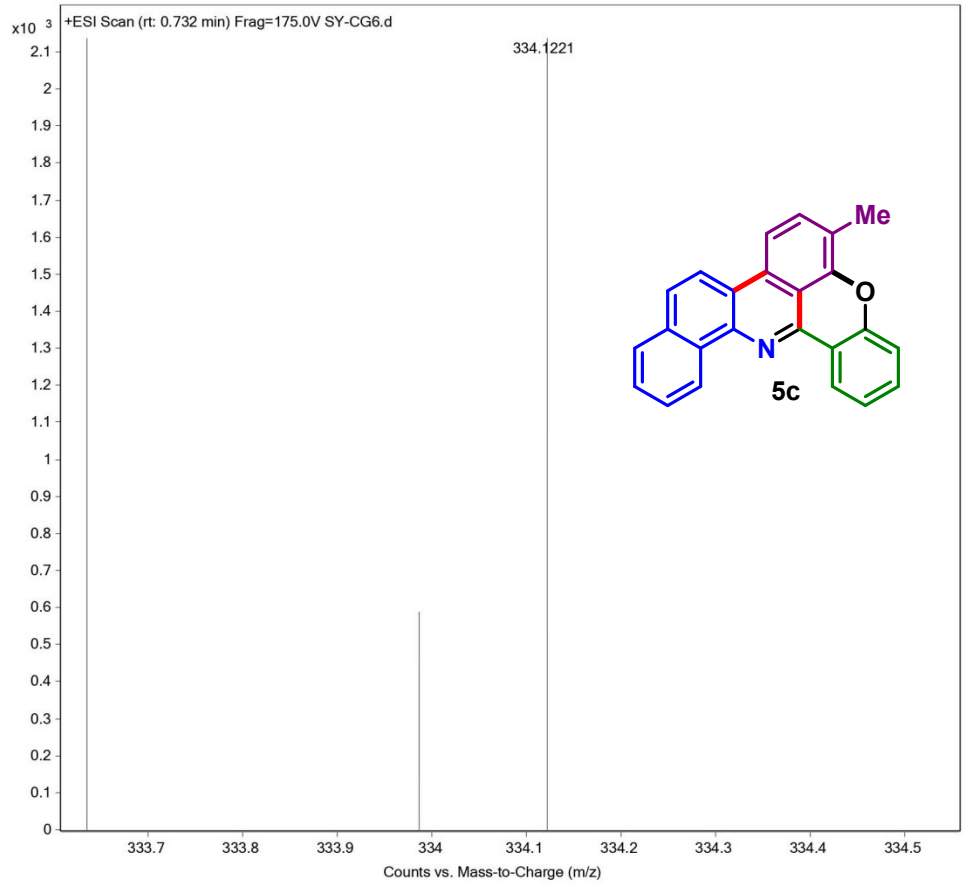
## $^{13}$C NMR Spectra of 5c

ATK-SY-CG6-13C.1.fid
ATK-SY-CG6-13C

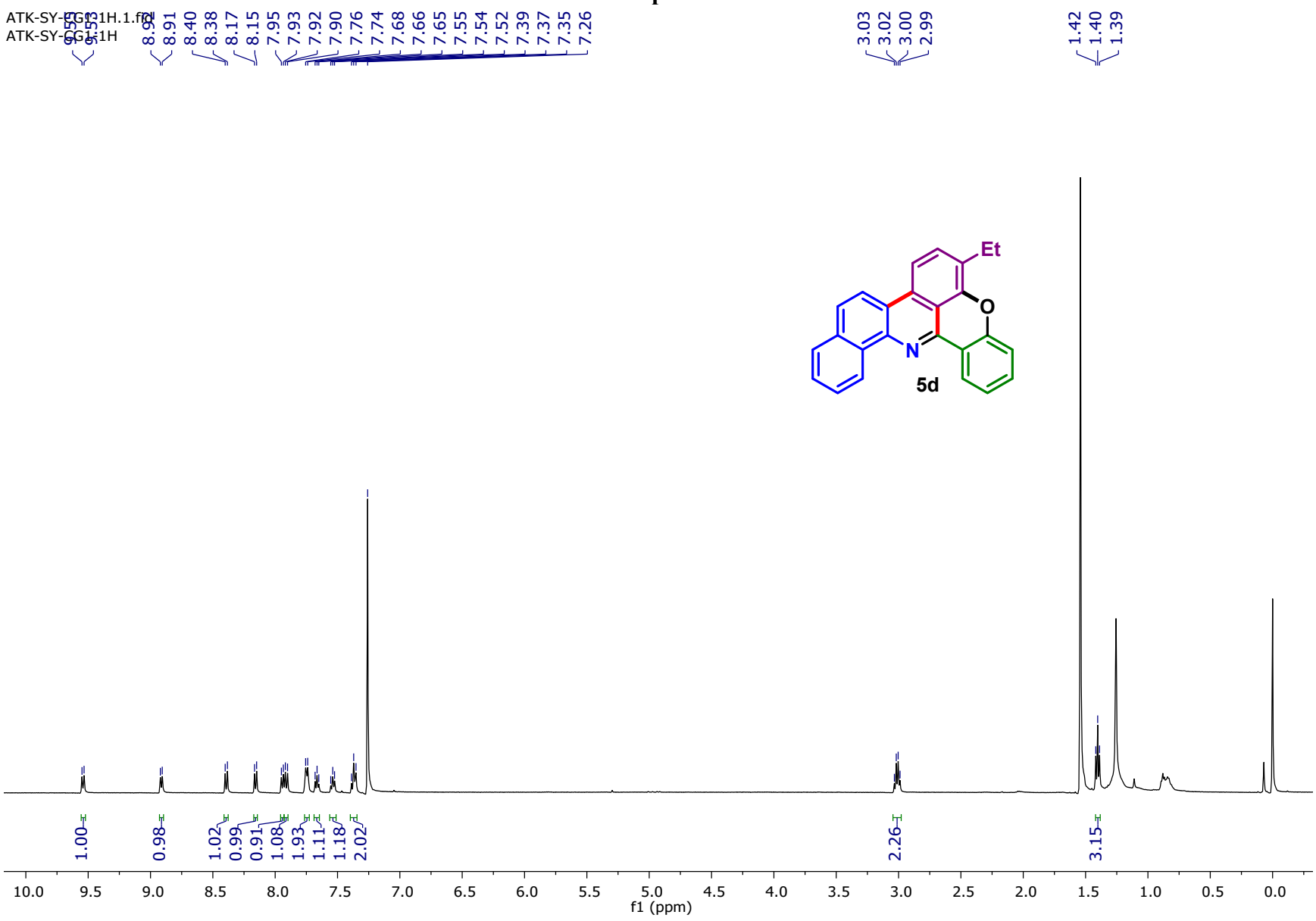


# HRMS Spectra of 5c

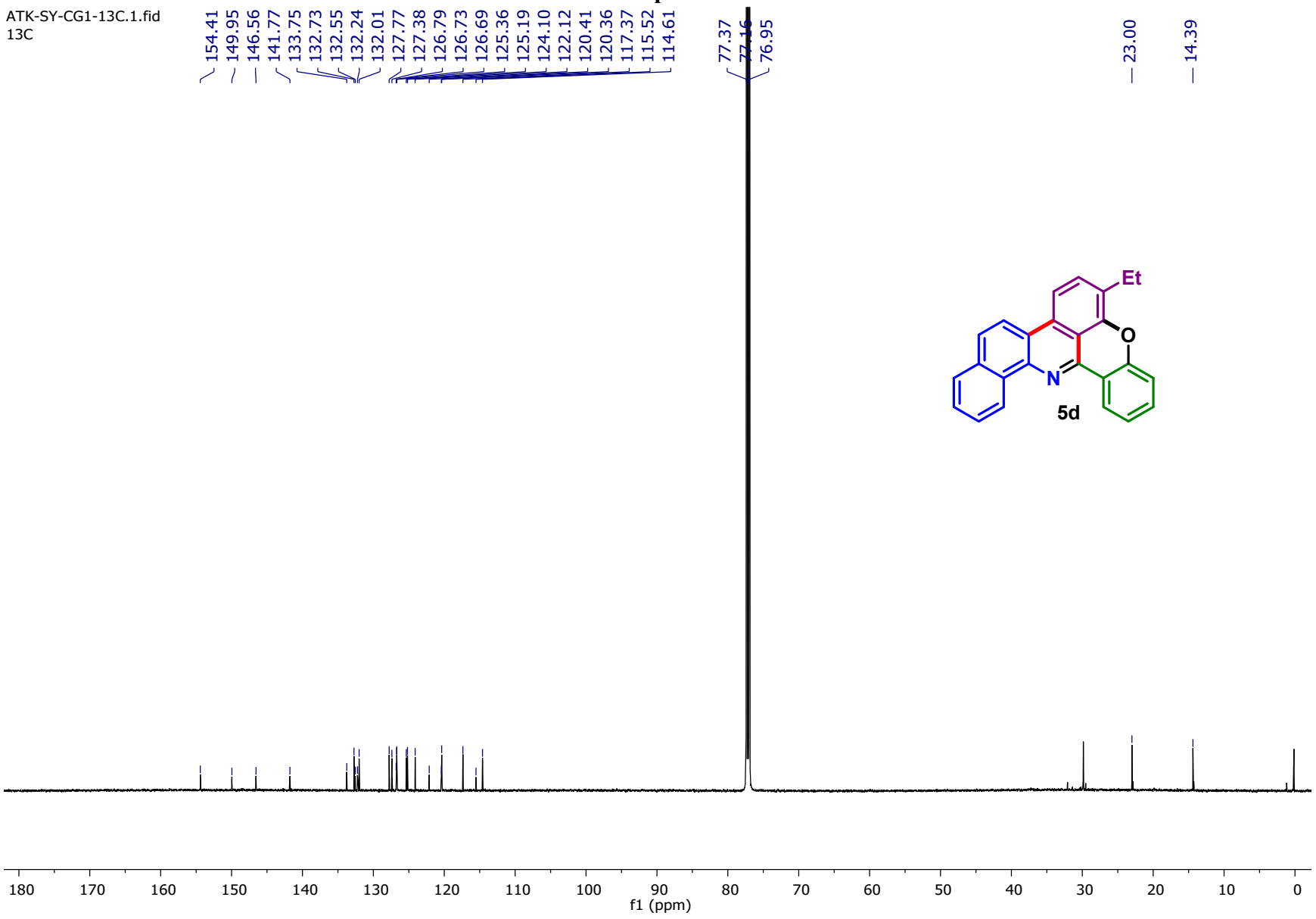
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | WASH | **Position** | P1-C7 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-CG6.d |
| **ACQ Method** | ESI ALS 100-600.m | **Comment** | | **Acquired Time** | 26-Aug-21 02:45:47 PM (UTC+05:30) |



## $^{1}$H NMR Spectra of 5d

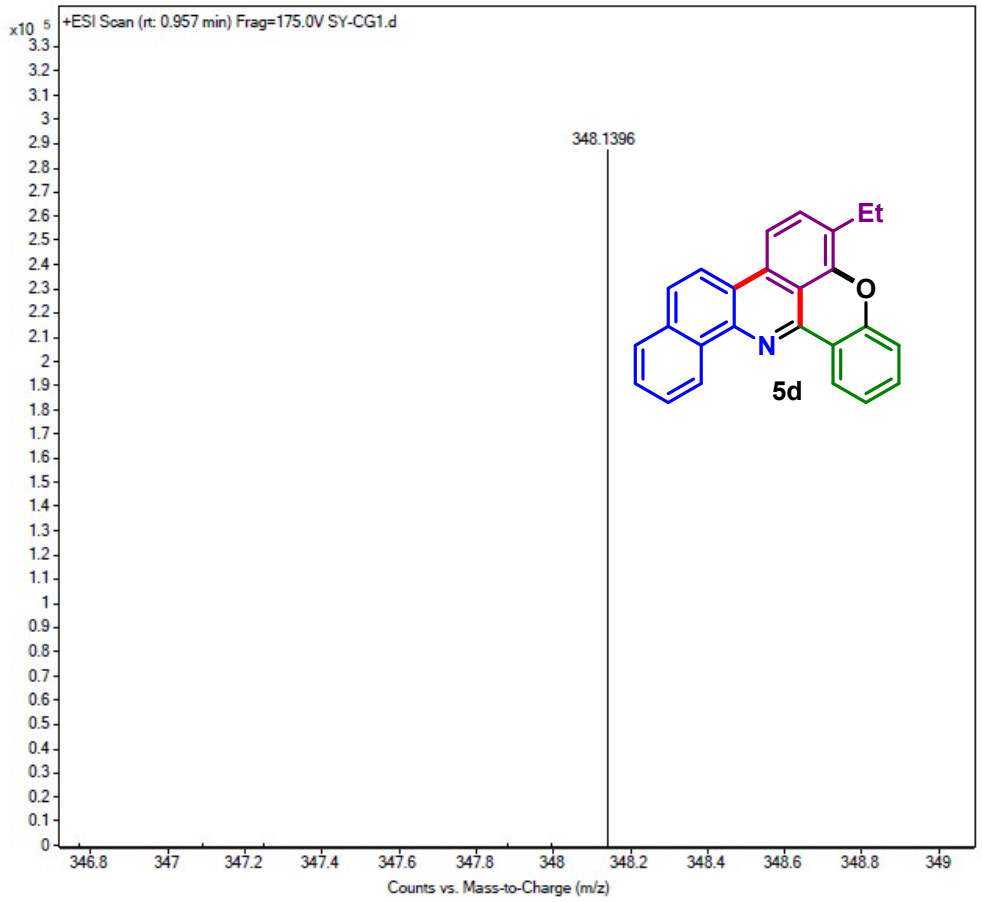


## $^{13}$C NMR Spectra of 5d

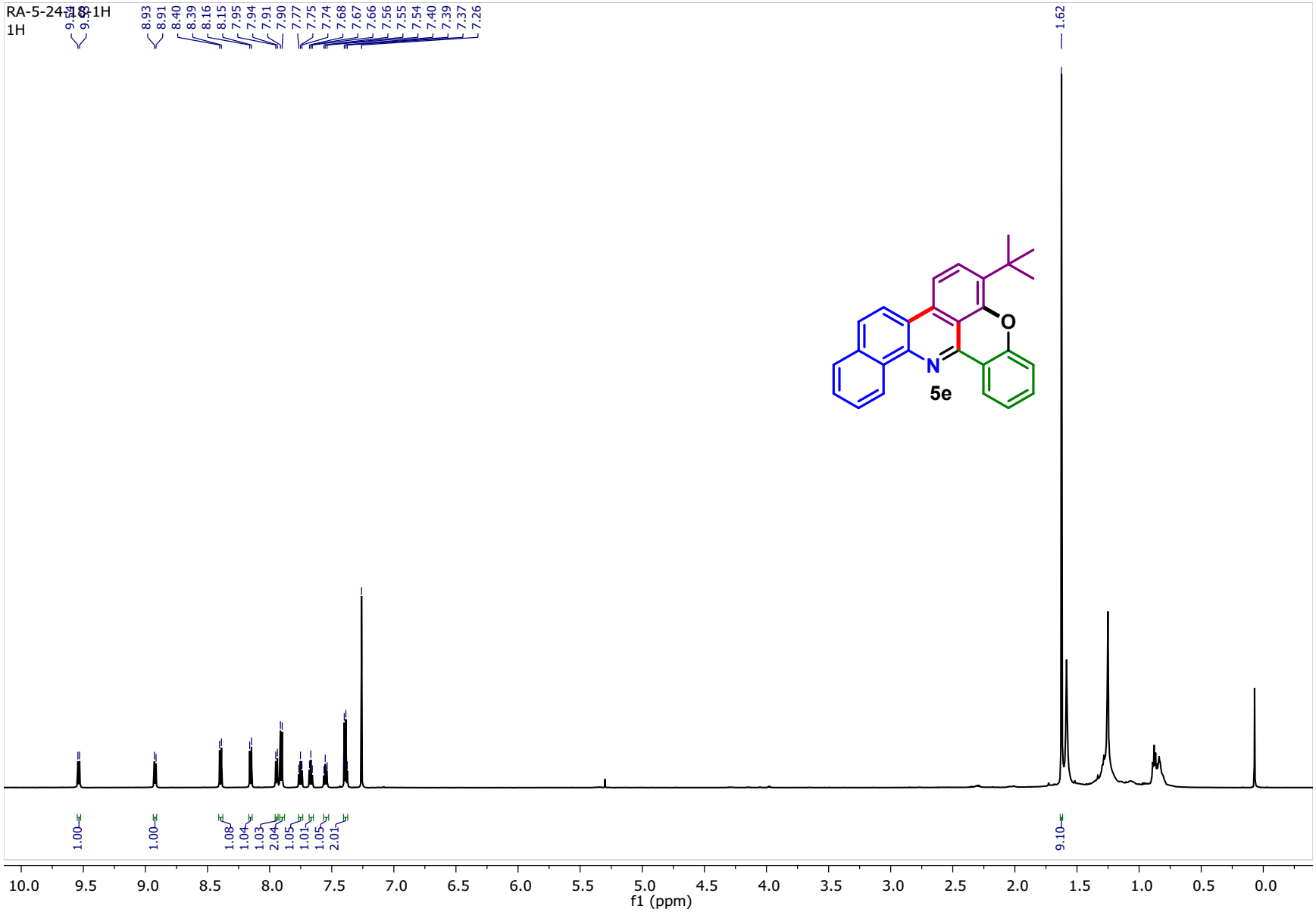


# HRMS Spectra of 5d

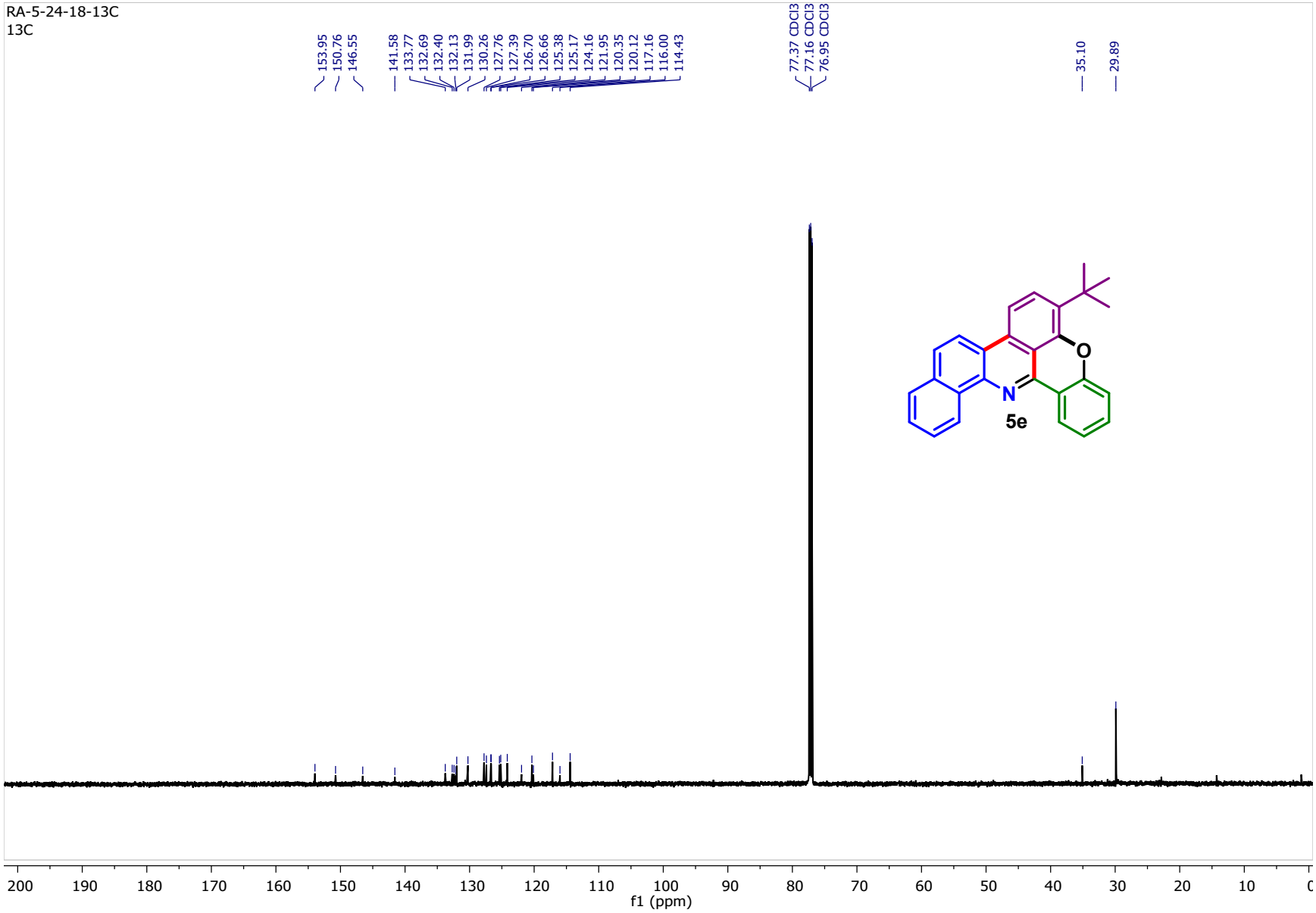
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SY-CG1 | **Position** | P1-D2 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-CG1.d |
| **ACQ Method** | ESI ALS 100-1000.m | **Comment** | | **Acquired Time** | 05-May-21 04:12:56 PM (UTC+05:30) |



## $^{1}$H NMR Spectra of 5e

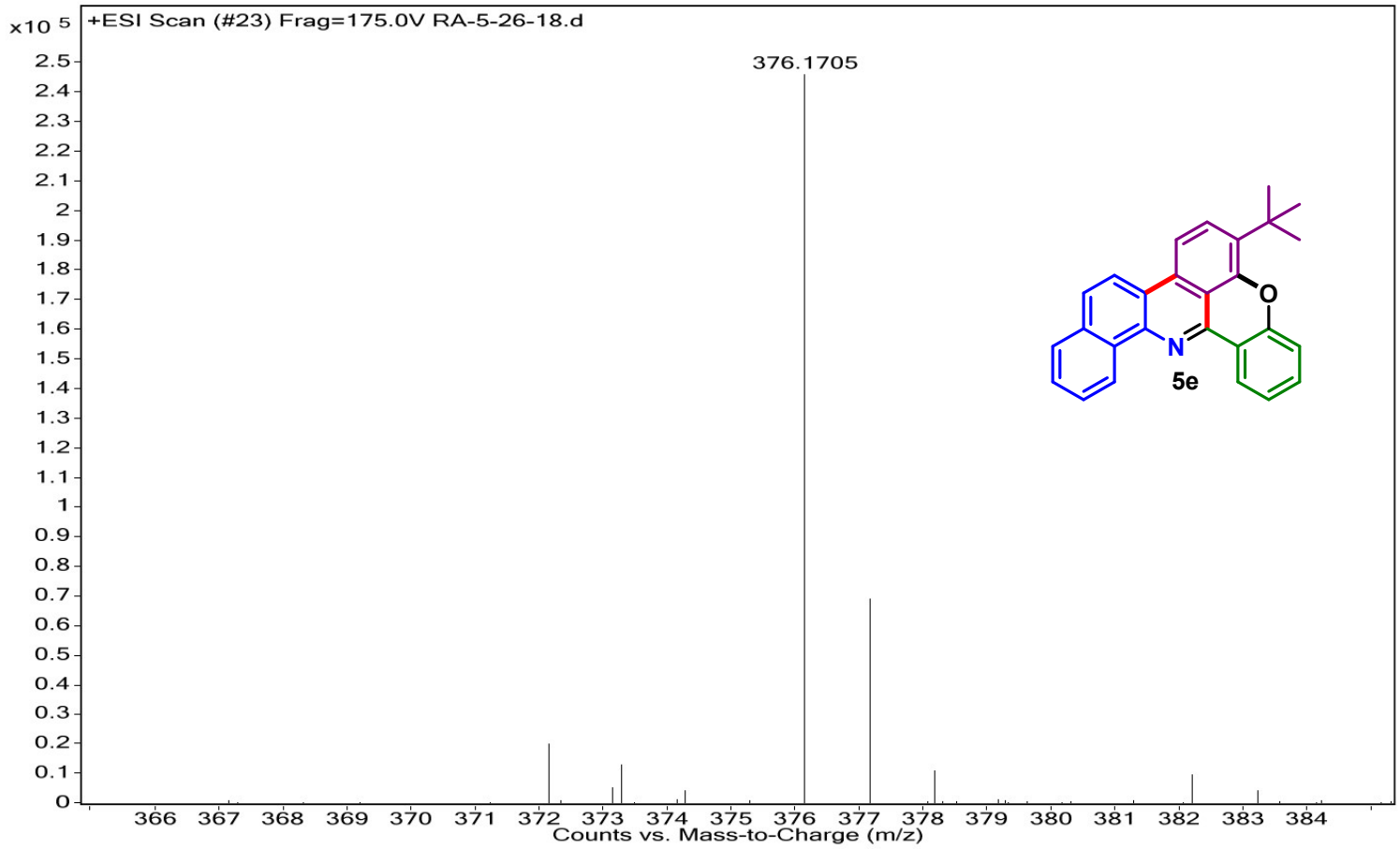


## $^{13}$C NMR Spectra of 5e

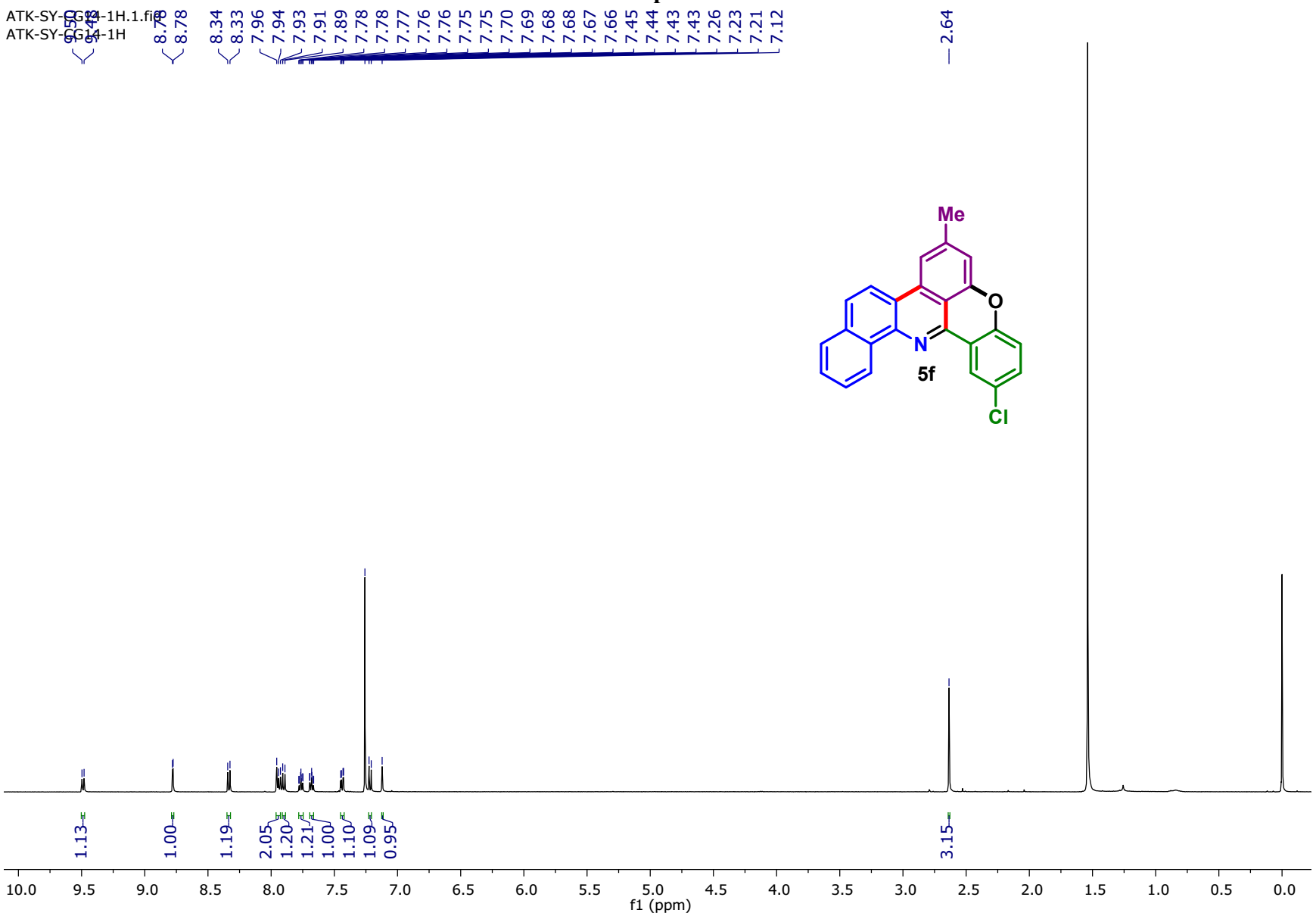


# HRMS Spectra of 5e

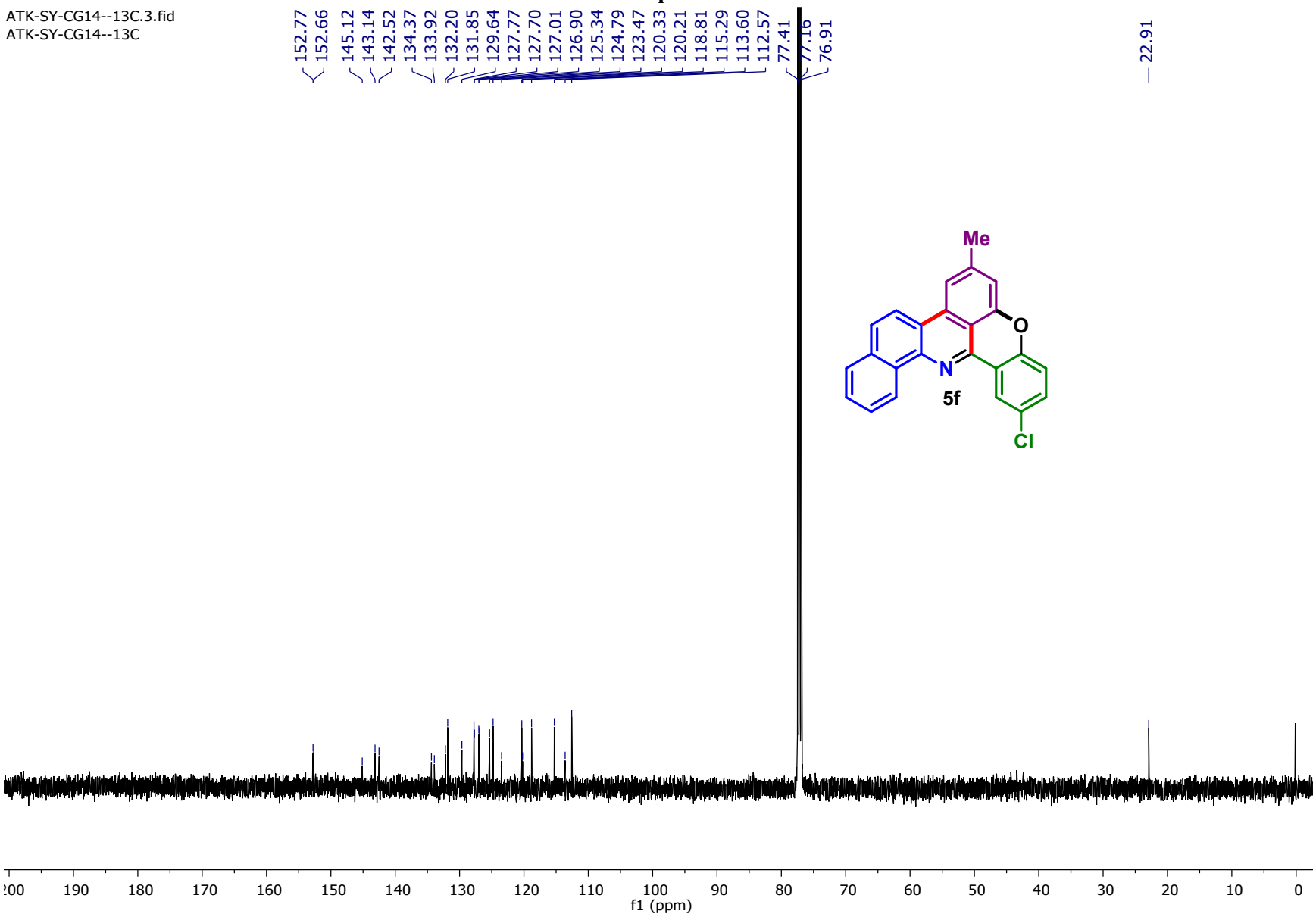
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Sample Name** | SAMPLE 6 | **Position** | P2-A5 | **Instrument Name** | Instrument 1 | **User Name** | |
| **Inj Vol** | 20 | **InjPosition** | | **SampleType** | Sample | **IRM Calibration Status** | Success |
| **Data Filename** | RA-5-26-18.d | **ACQ Method** | ESI ALS 100-600.m | **Comment** | | **Acquired Time** | 12/26/2018 4:29:26 PM |



## $^1$H NMR Spectra of 5f

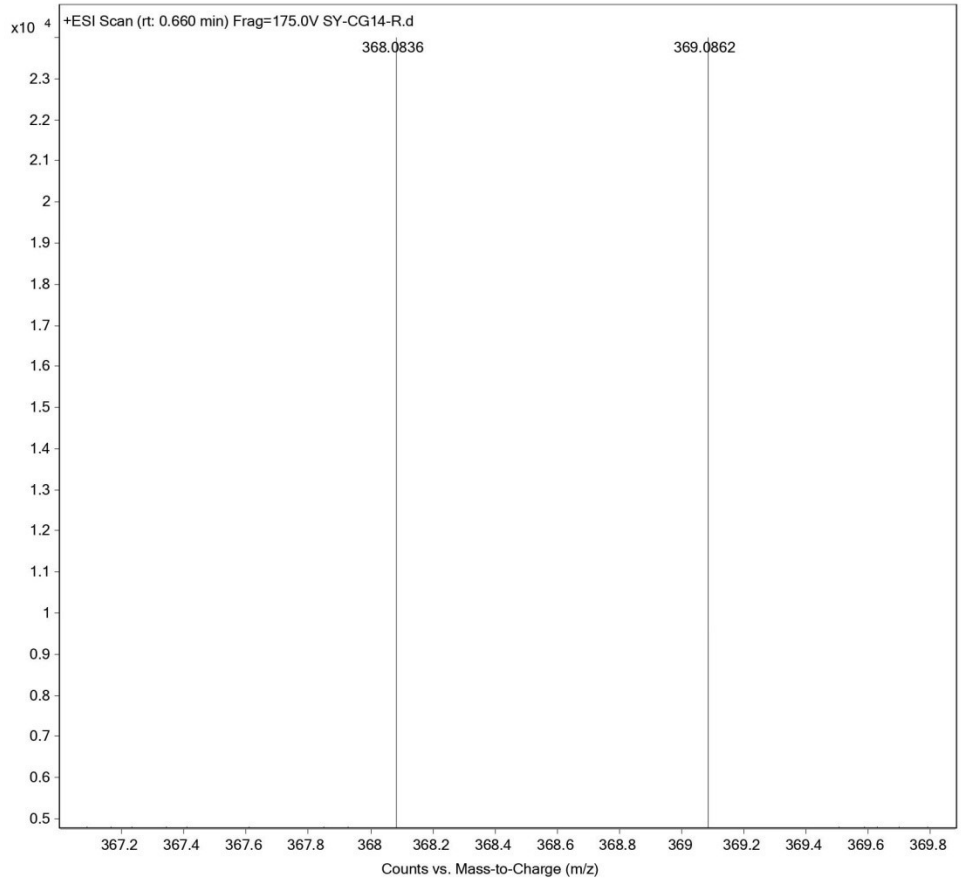


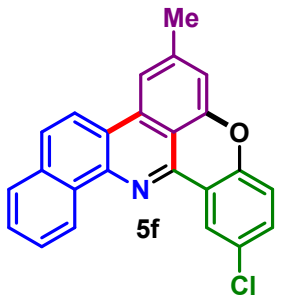
## $^{13}$C NMR Spectra of 5f



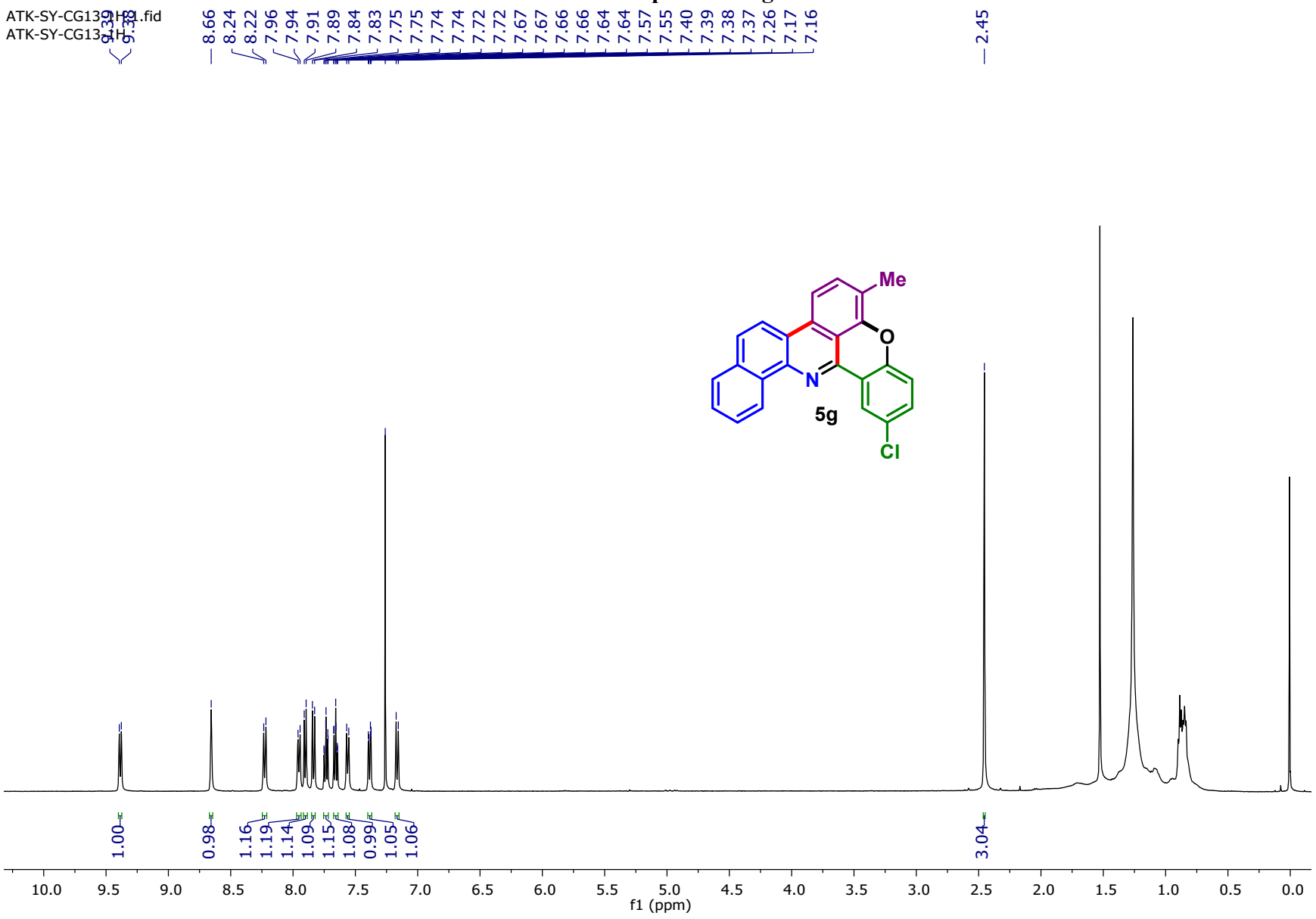
# HRMS Spectra of 5f

| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SAMPLE | **Position** | P2-C6 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-CG14-R.d |
| **ACQ Method** | ESI ALS 200-600.m | **Comment** | | **Acquired Time** | 30-Aug-21 04:54:25 PM (UTC+05:30) |

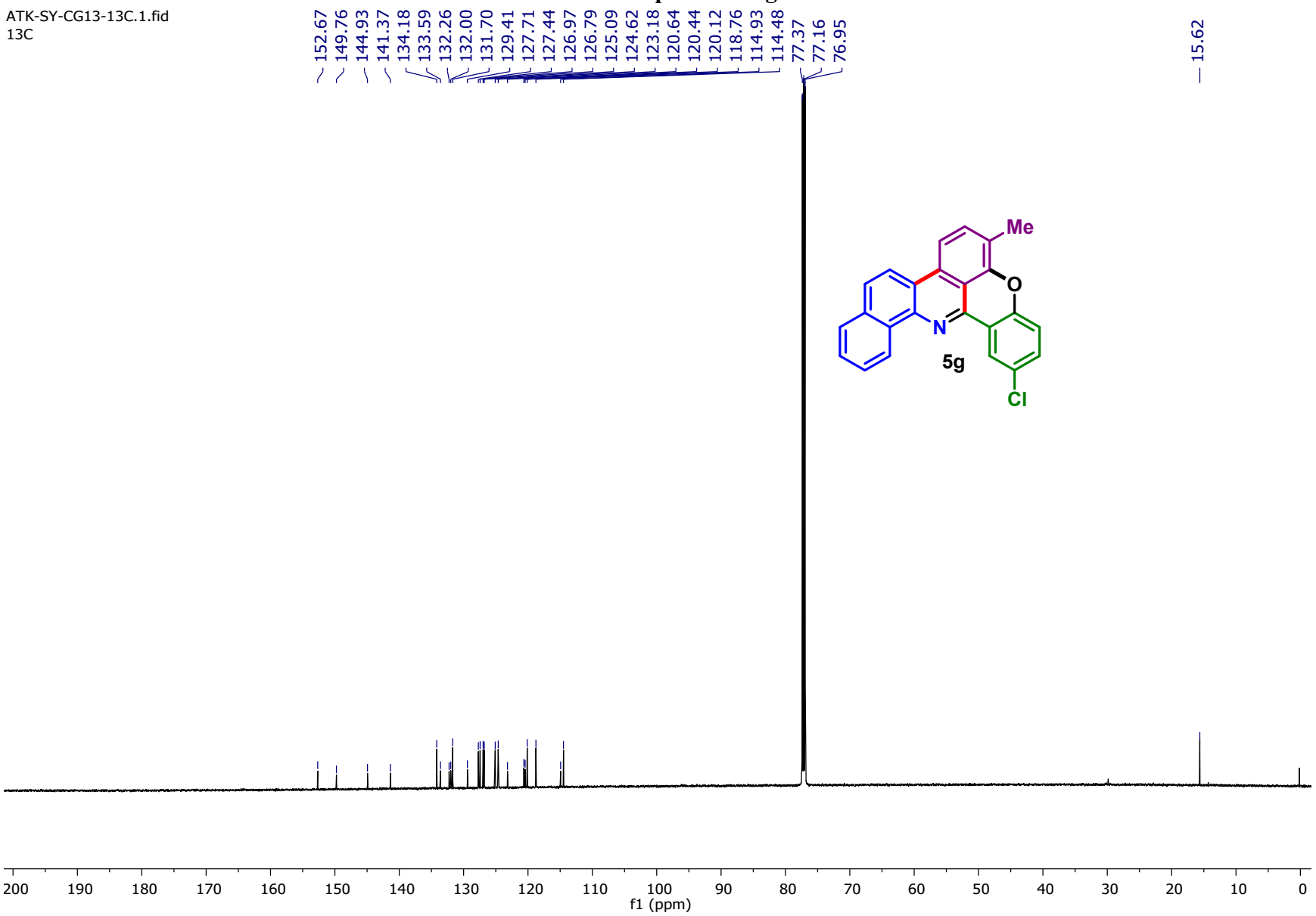


## $^{1}$H NMR Spectra of 5g

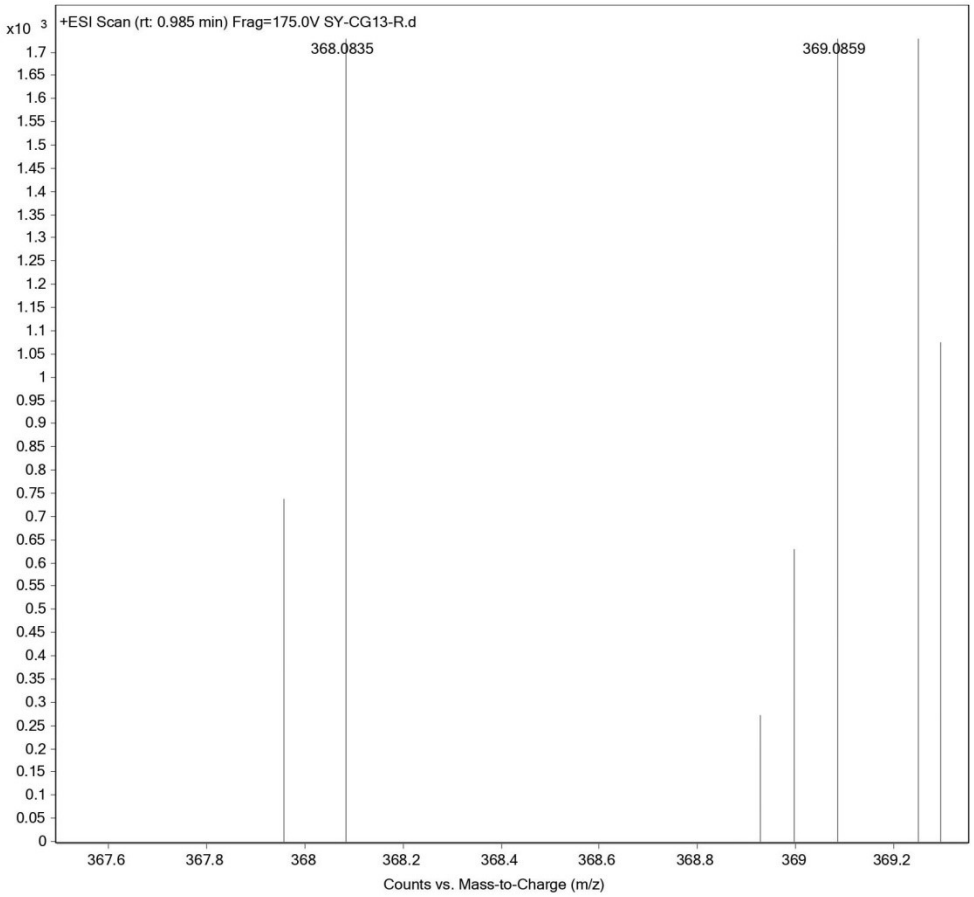


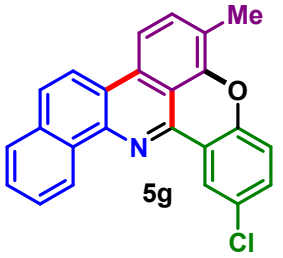
## $^{13}$C NMR Spectra of 5g



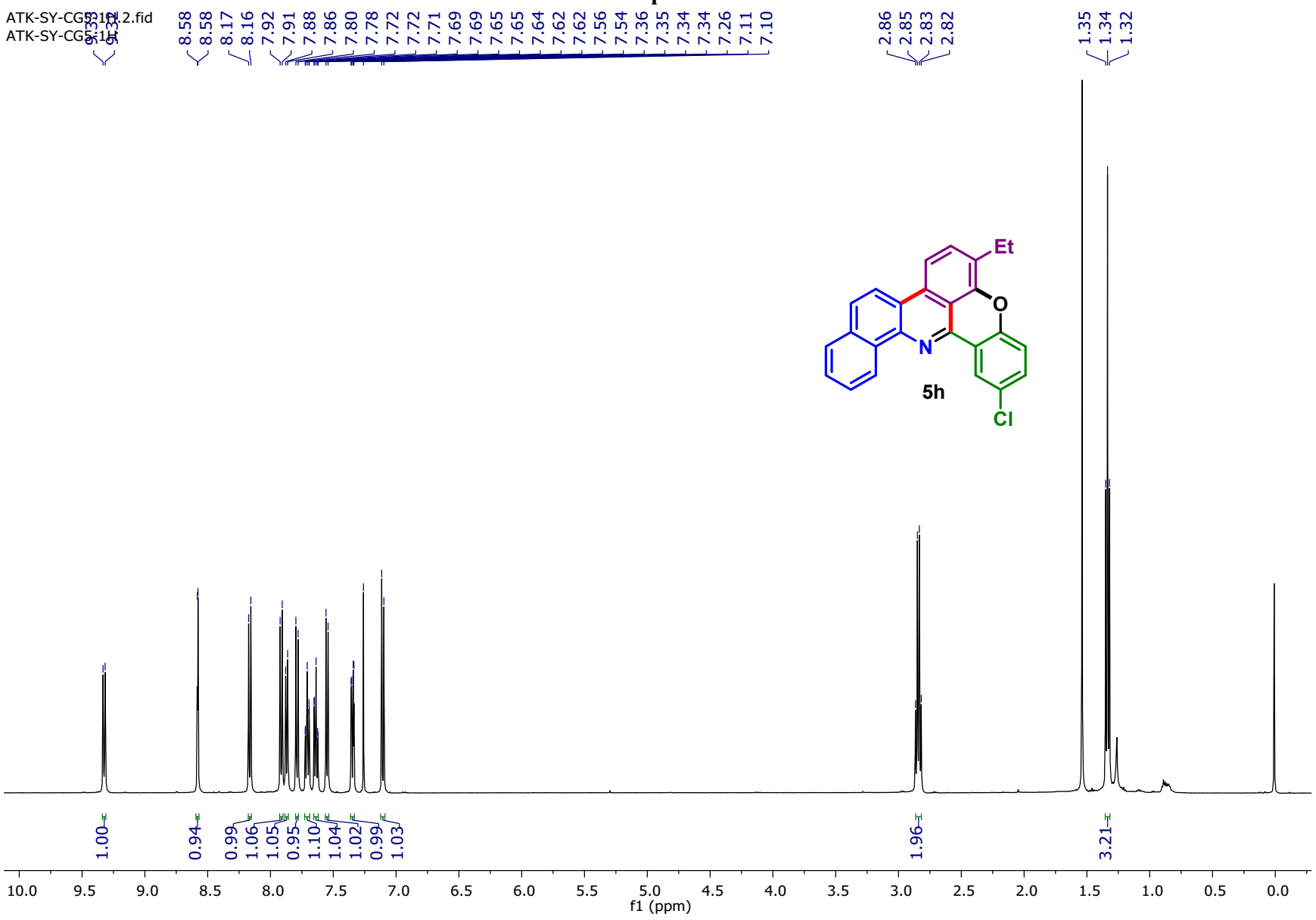
# HRMS Spectra of 5g

| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SAMPLE | **Position** | P2-C5 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-CG13-R.d |
| **ACQ Method** | ESI ALS 200-600.m | **Comment** | | **Acquired Time** | 30-Aug-21 04:44:57 PM (UTC+05:30) |

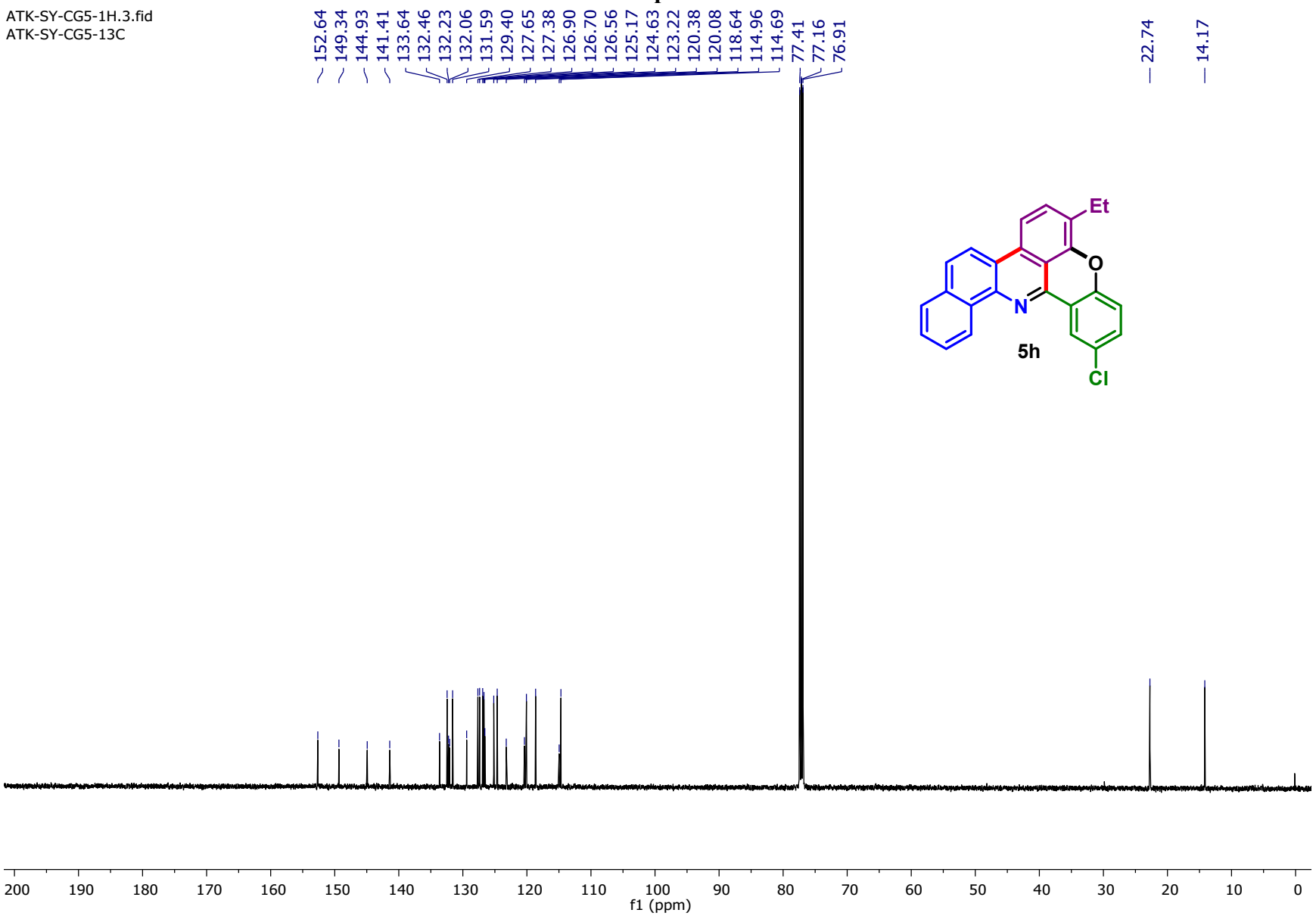


## $^1$H NMR Spectra of 5h

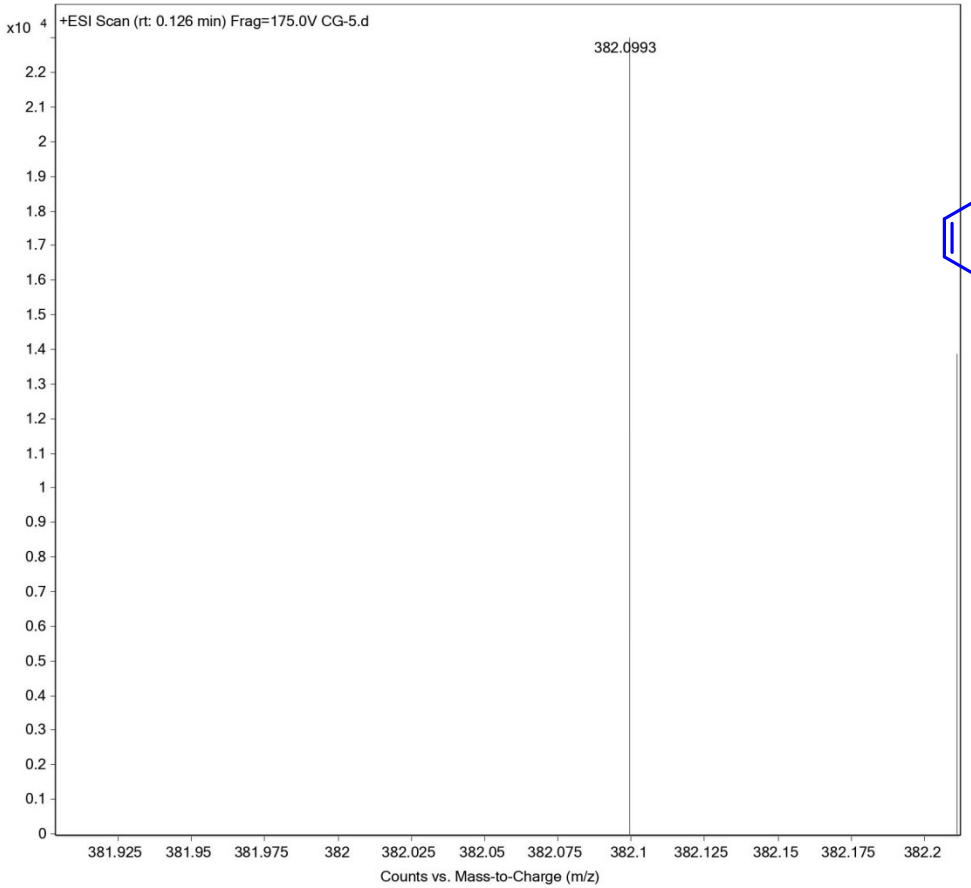


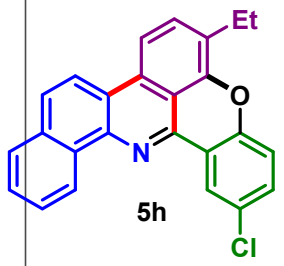
## $^{13}$C NMR Spectra of 5h



# HRMS Spectra of 5h

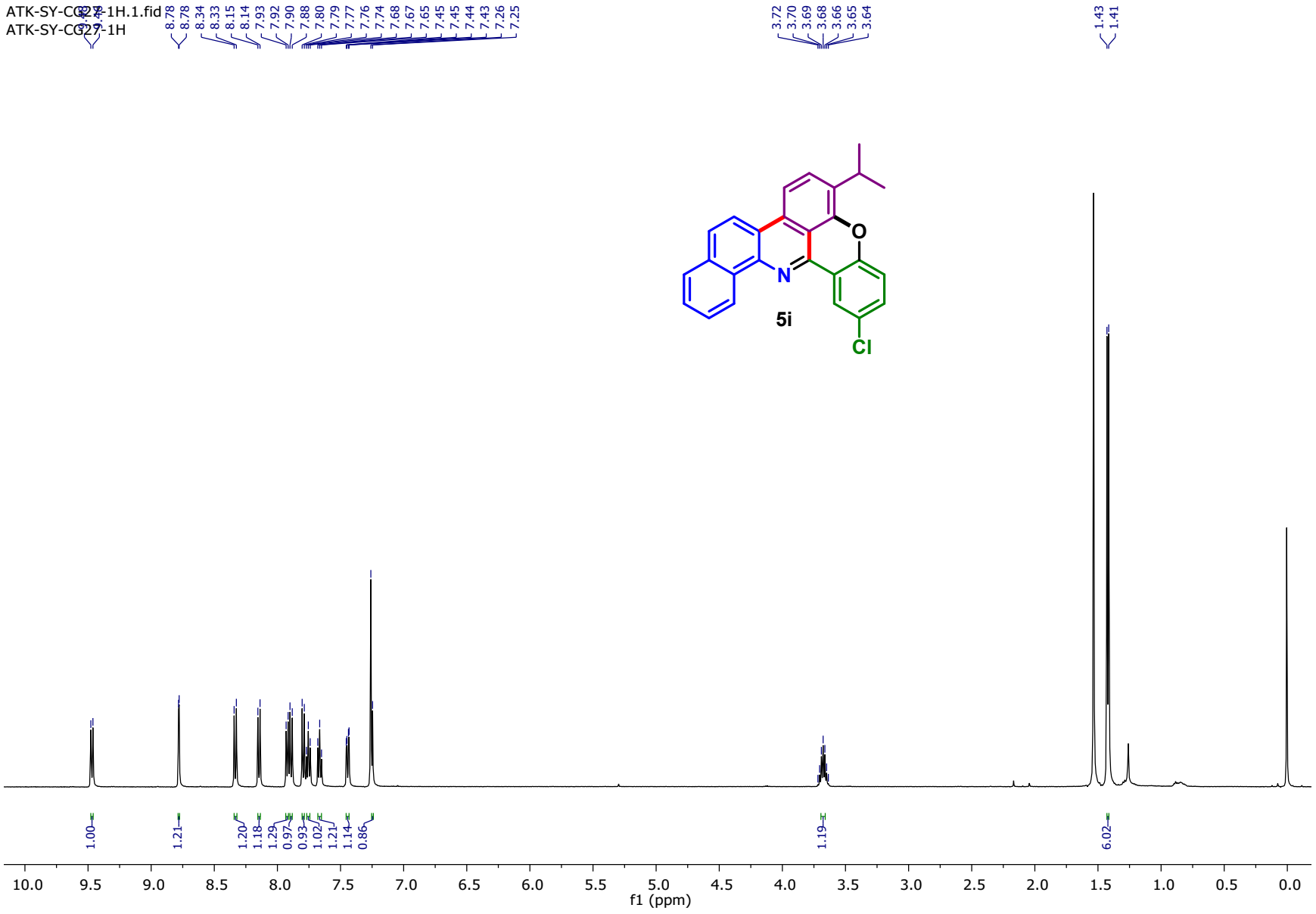
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | WASH | **Position** | P1-E5 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | CG-5.d |
| **ACQ Method** | ESI ALS 100-1000.m | **Comment** | | **Acquired Time** | 30-Aug-21 12:04:27 PM (UTC+05:30) |



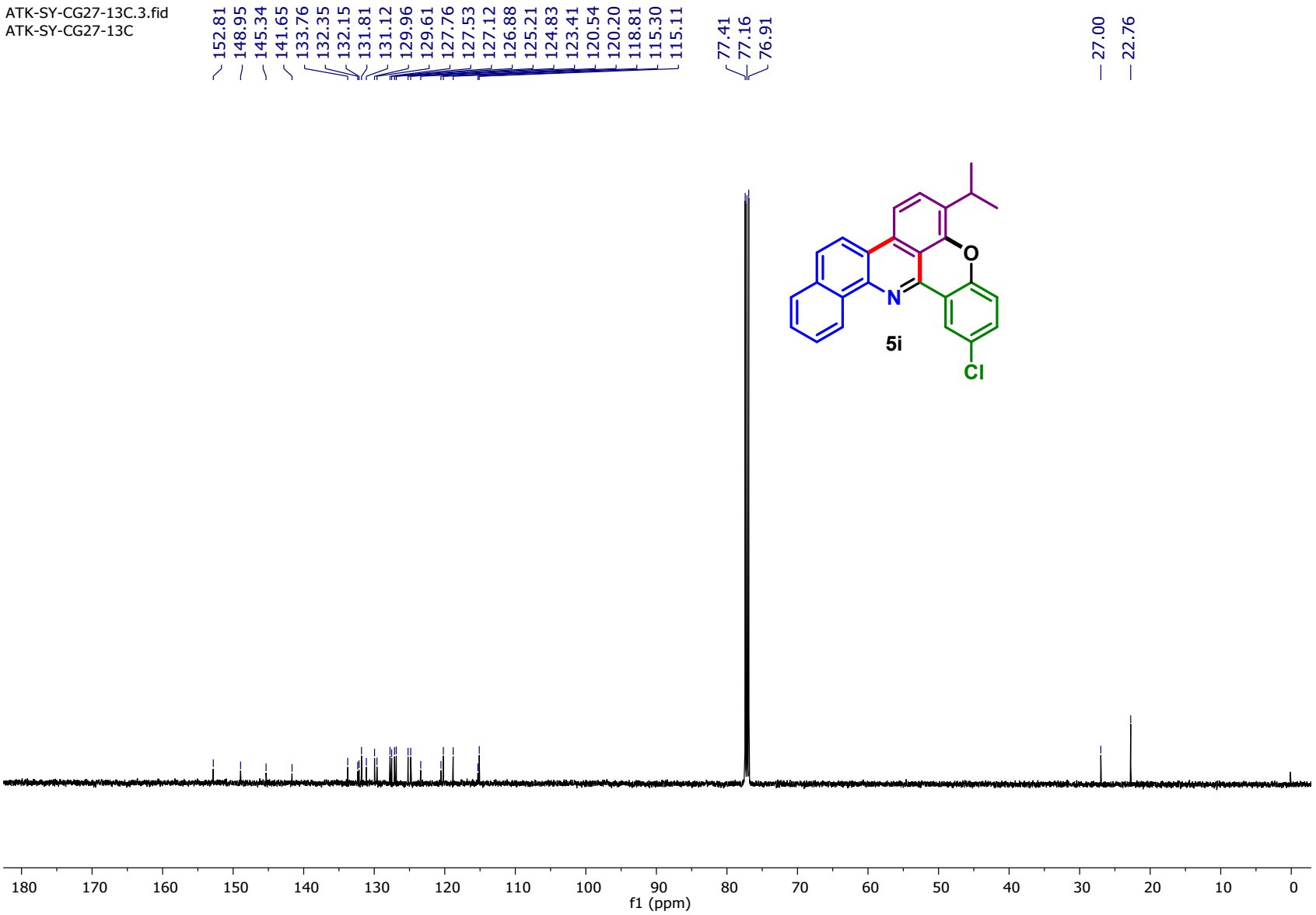




5h

## $^{1}$H NMR Spectra of 5i



## $^{13}$C NMR Spectra of 5i



**HRMS Spectra of 5i**

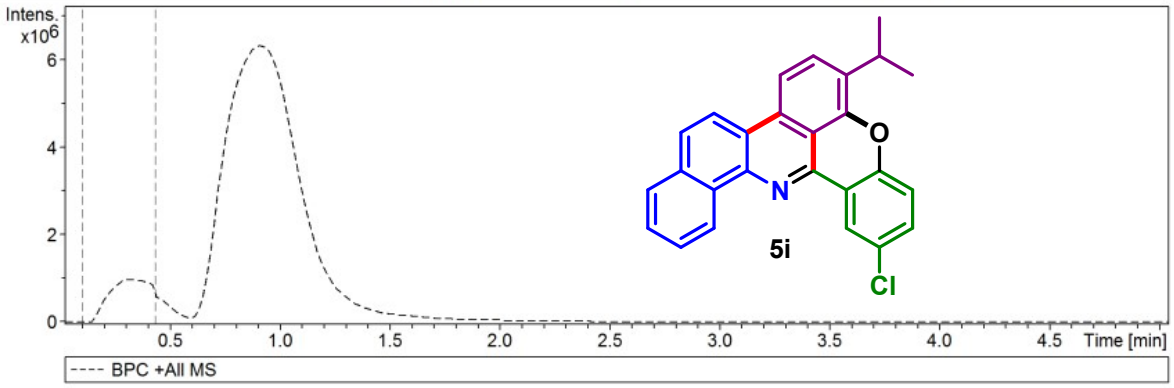
# Display Report

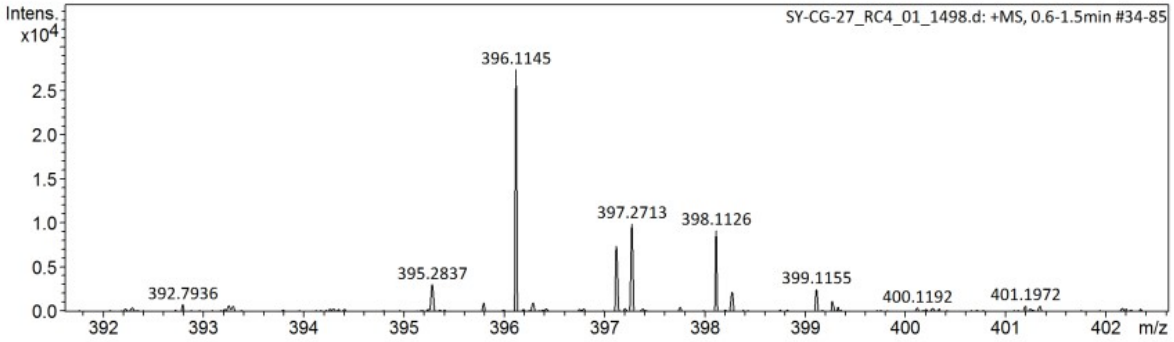
**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\user data\HPLC\DR LOKMAN\PRABHAS\SY-CG-27_RC4_01_1498.d | Acquisition Date | 1/27/2022 1:53:16 PM |
| Method | low mass bruker.m | Operator | vidhi |
| Sample Name | SY-CG-27 | Instrument | impact HD 1819696.00197 |
| Comment | | | |

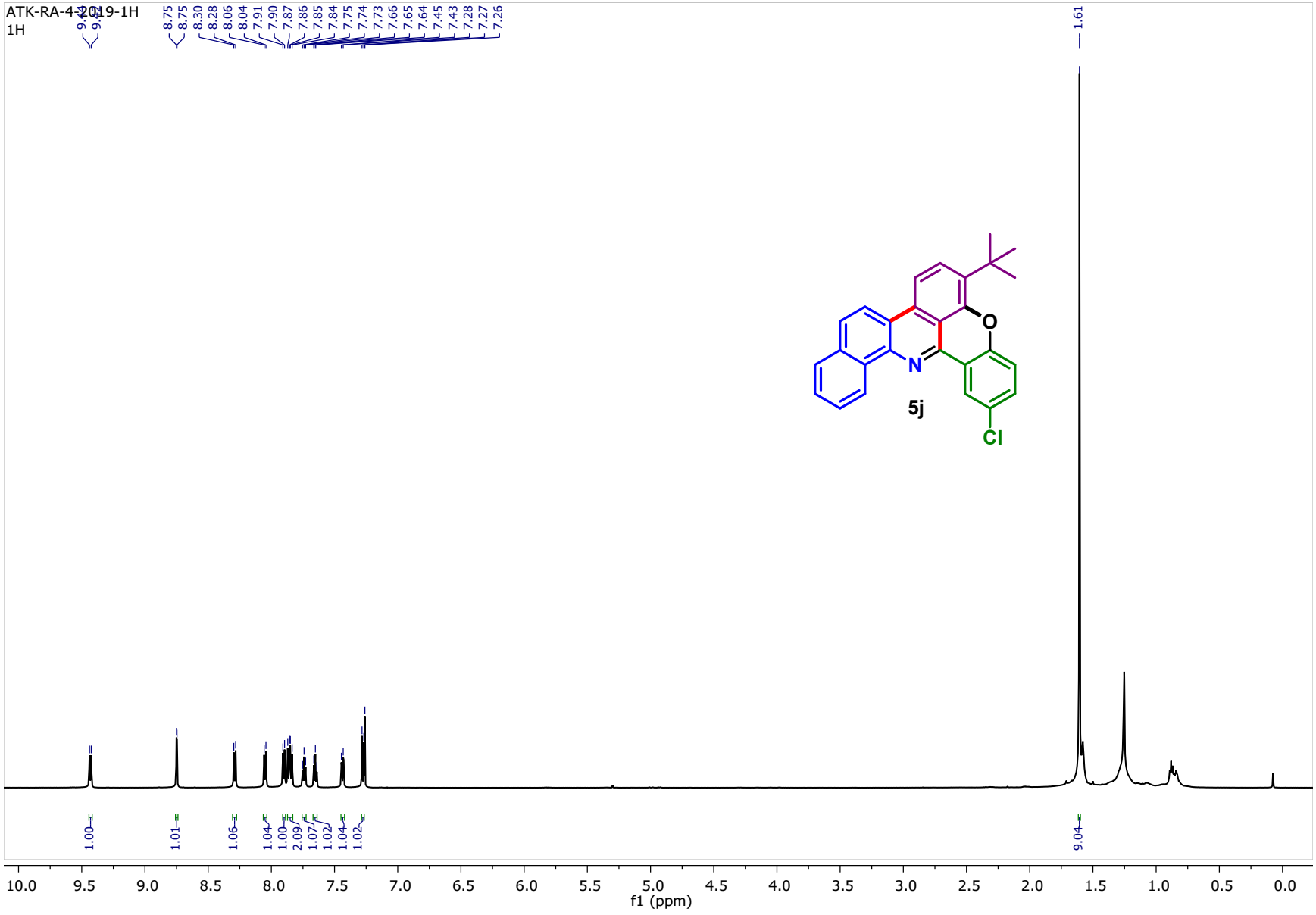
**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.8 Bar |
| Focus | Active | Set Capillary | 4500 V | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 6.0 l/min |
| Scan End | 1500 m/z | Set Charging Voltage | 2000 V | Set Divert Valve | Waste |
| | | Set Corona | 0 nA | Set APCI Heater | 0 °C |

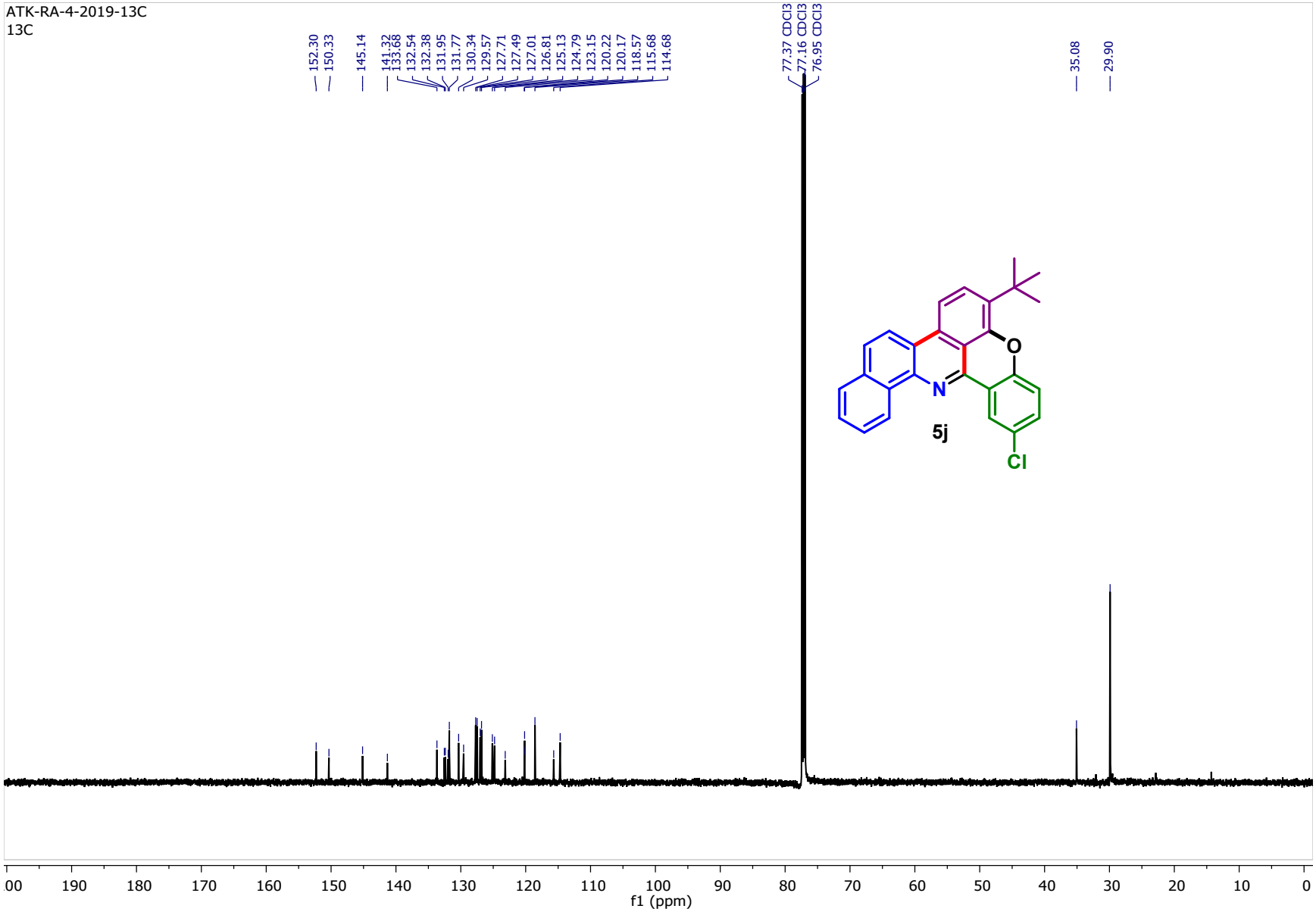


## $^{1}$H NMR Spectra of 5j

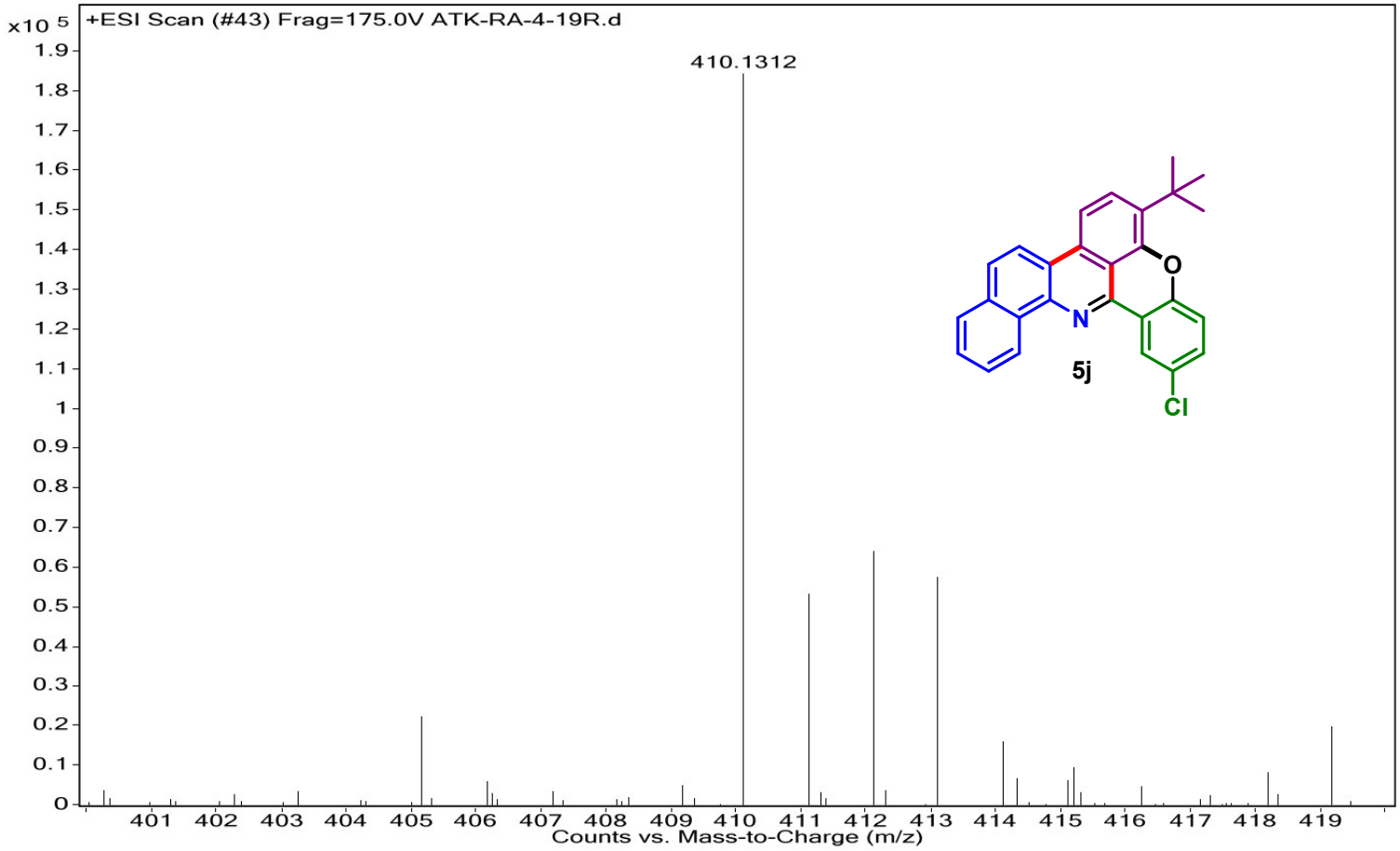


## $^{13}$C NMR Spectra of 5j

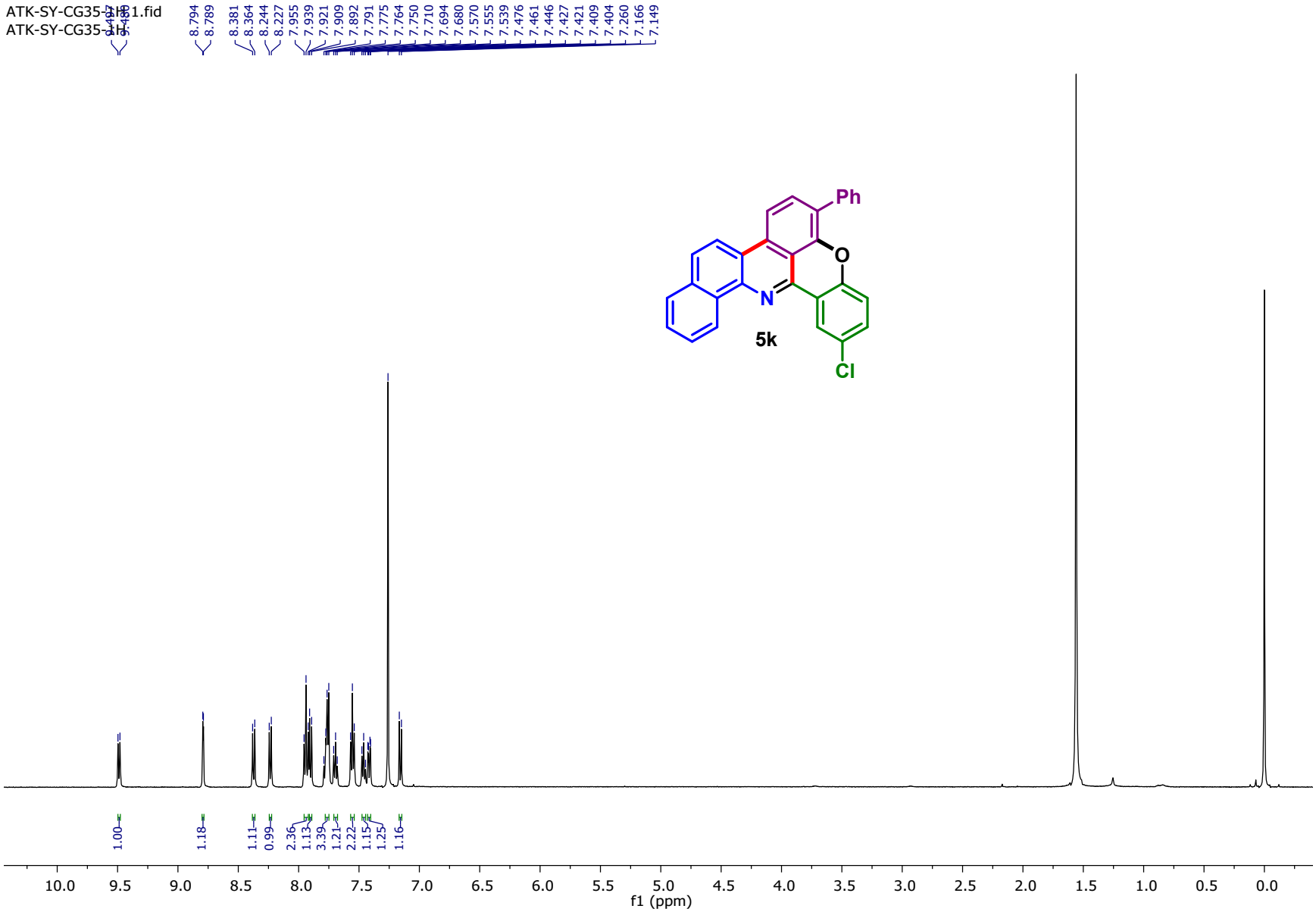


# HRMS Spectra of 5j

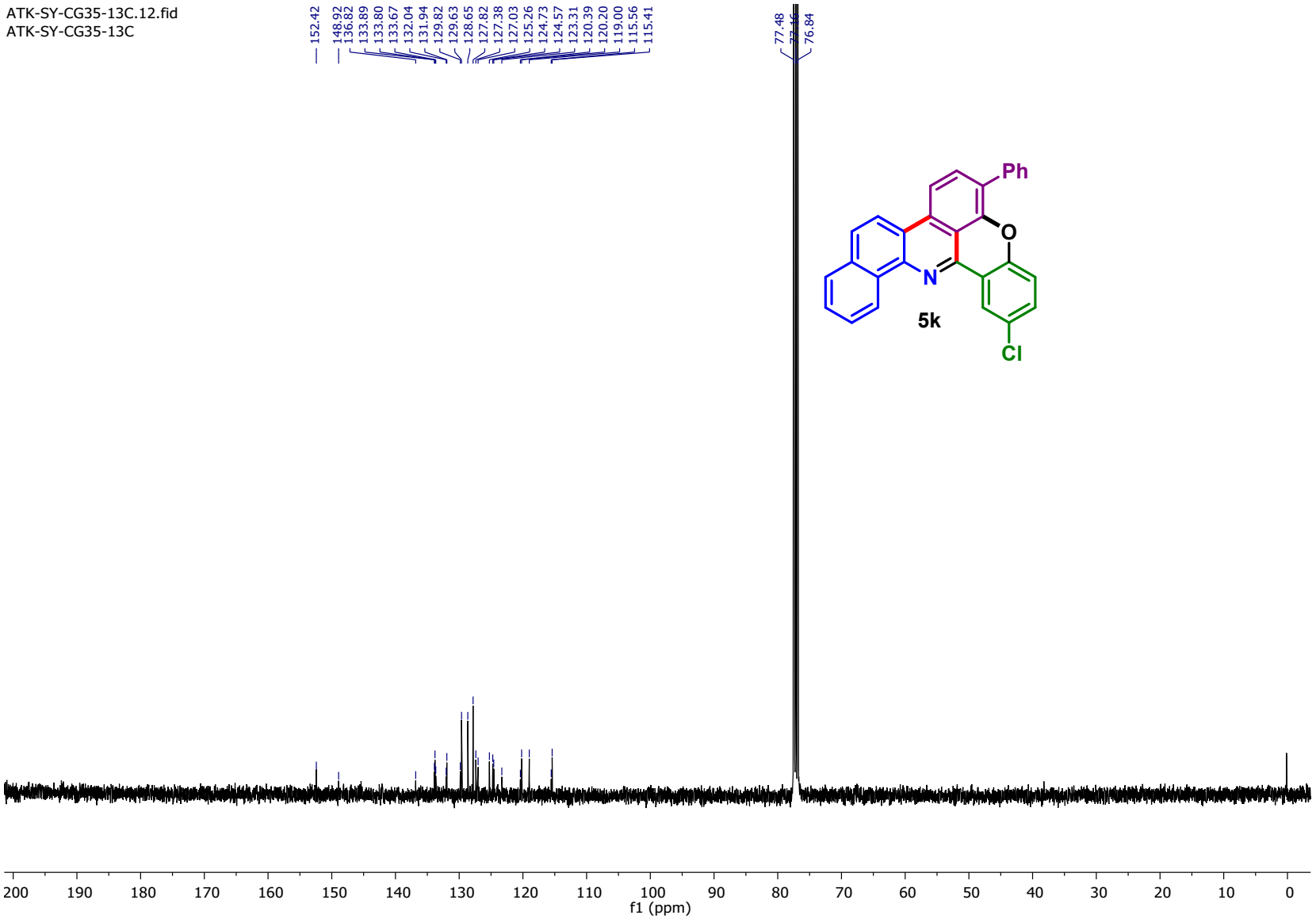
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Sample Name** | SAMPLE 1 | **Position** | P1-A2 | **Instrument Name** | Instrument 1 | **User Name** | |
| **Inj Vol** | 20 | **InjPosition** | | **SampleType** | Sample | **IRM Calibration Status** | Success |
| **Data Filename** | ATK-RA-4-19R.d | **ACQ Method** | ESI ALS 200-600.m | **Comment** | | **Acquired Time** | 1/21/2019 3:50:39 PM |



## $^{1}$H NMR Spectra of 5k



## $^{13}$C NMR Spectra of 5k



## HRMS Spectra of 5k

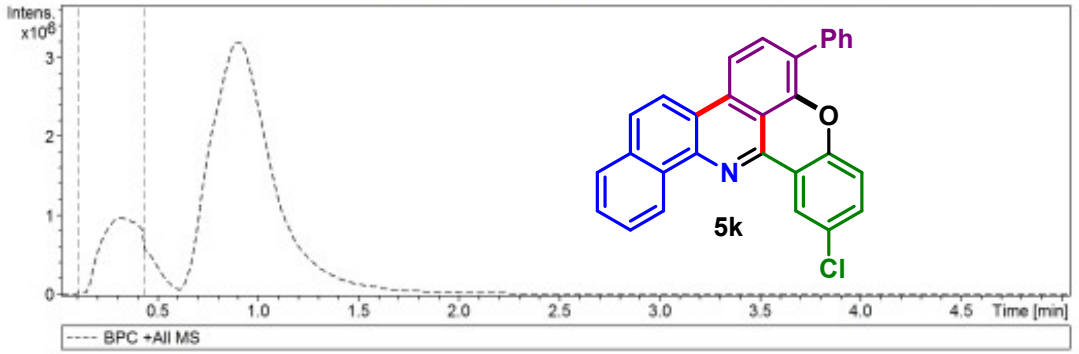
# Display Report

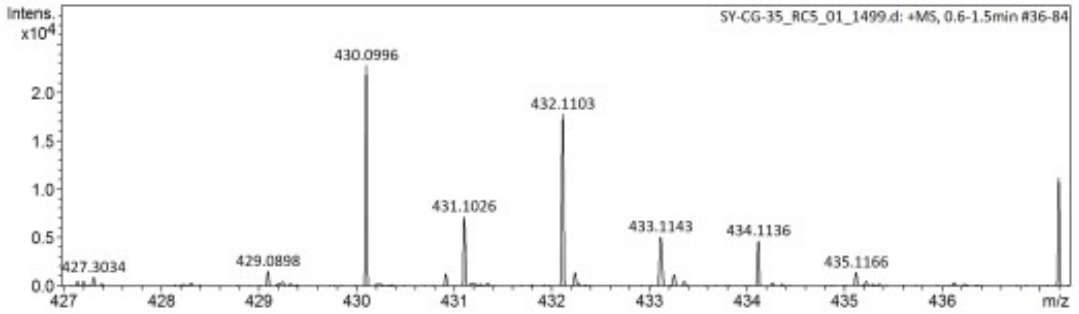
**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\user data\HPLC\DR LOKMAN\PRABHAS\SY-CG-35_RC5_01_1499.d | Acquisition Date | 1/27/2022 1:59:48 PM |
| Method | low mass bruker.m | Operator | vidhi |
| Sample Name | SY-CG-35 | Instrument | impact HD 1819696.00197 |
| Comment | | | |

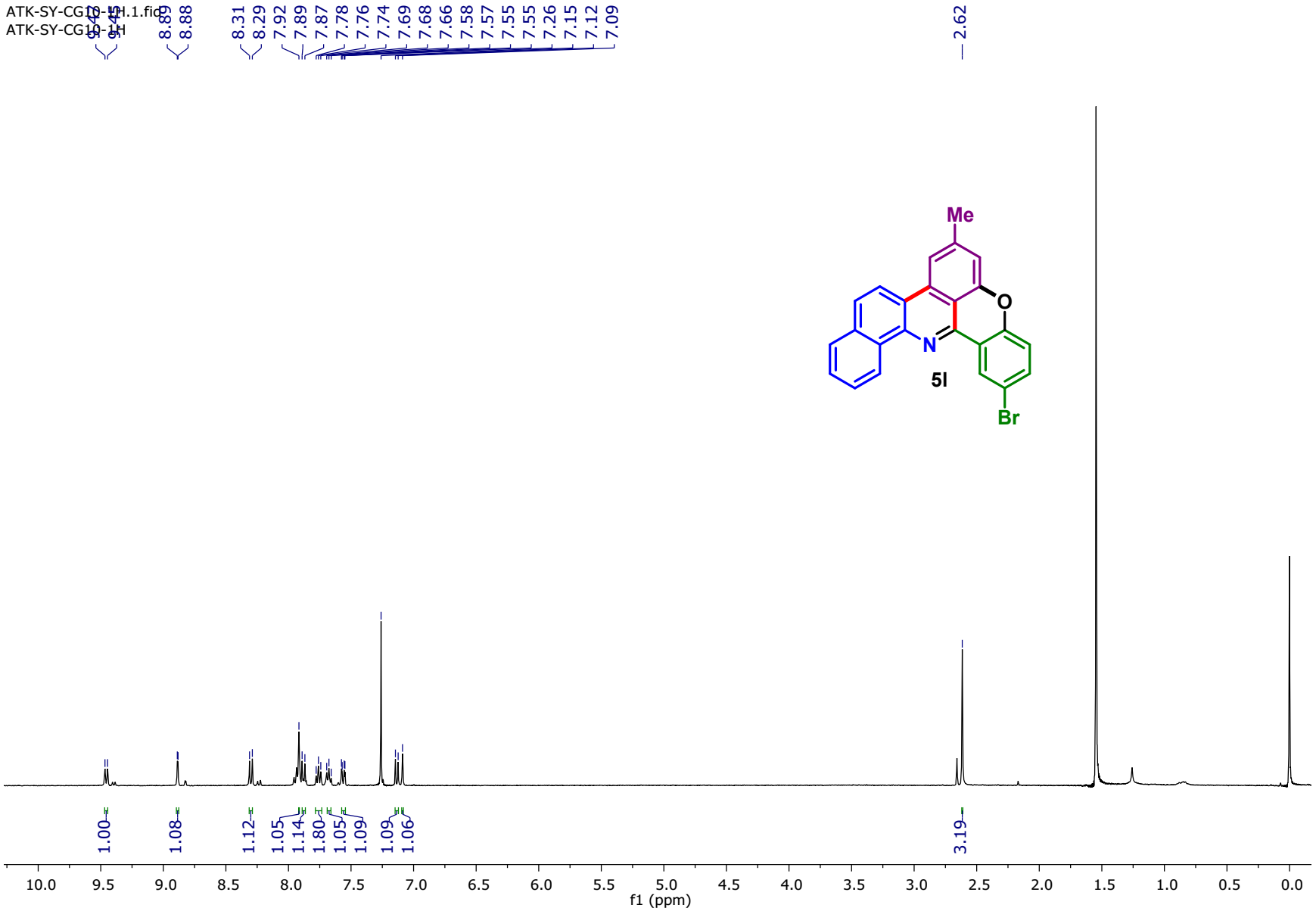
**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.8 Bar |
| Focus | Active | Set Capillary | 4500 V | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 6.0 l/min |
| Scan End | 1500 m/z | Set Charging Voltage | 2000 V | Set Divert Valve | Waste |
| | | Set Corona | 0 nA | Set APCI Heater | 0 °C |

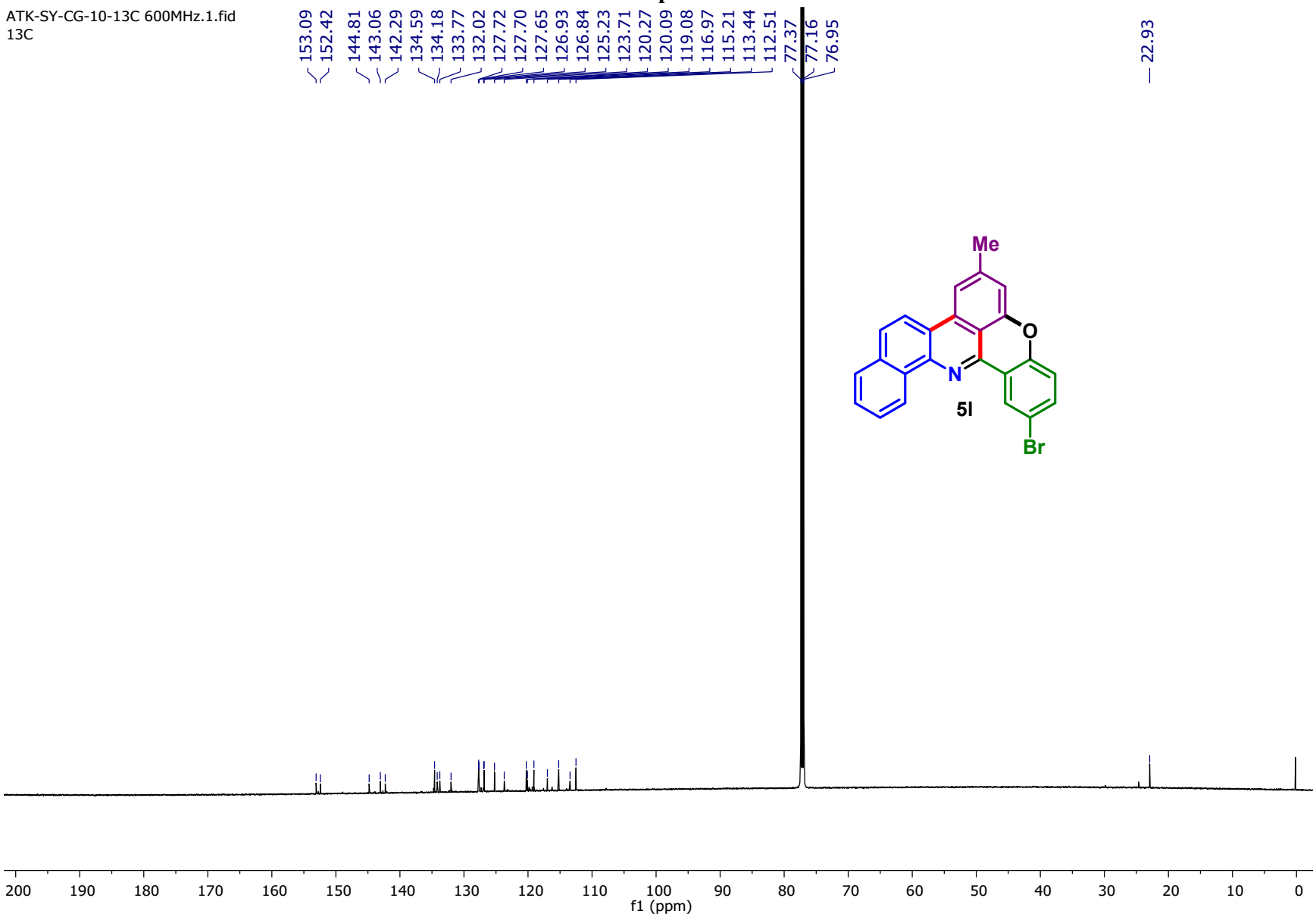


## $^{1}$H NMR Spectra of 5l

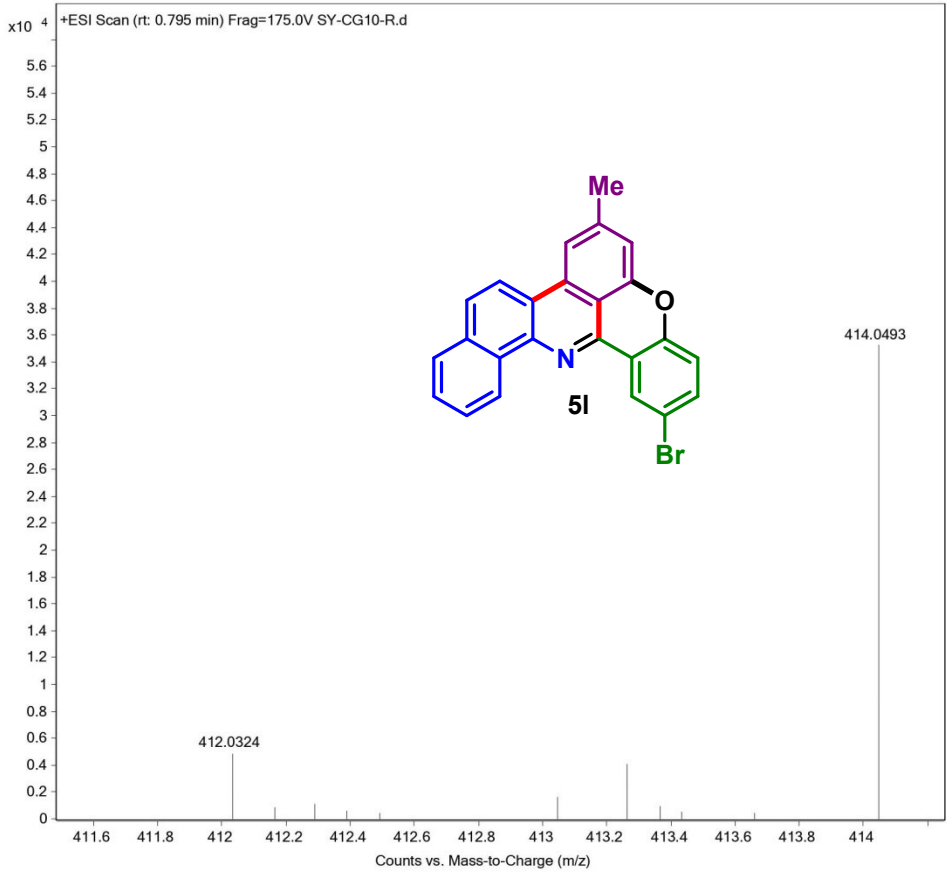


## $^{13}$C NMR Spectra of 5l

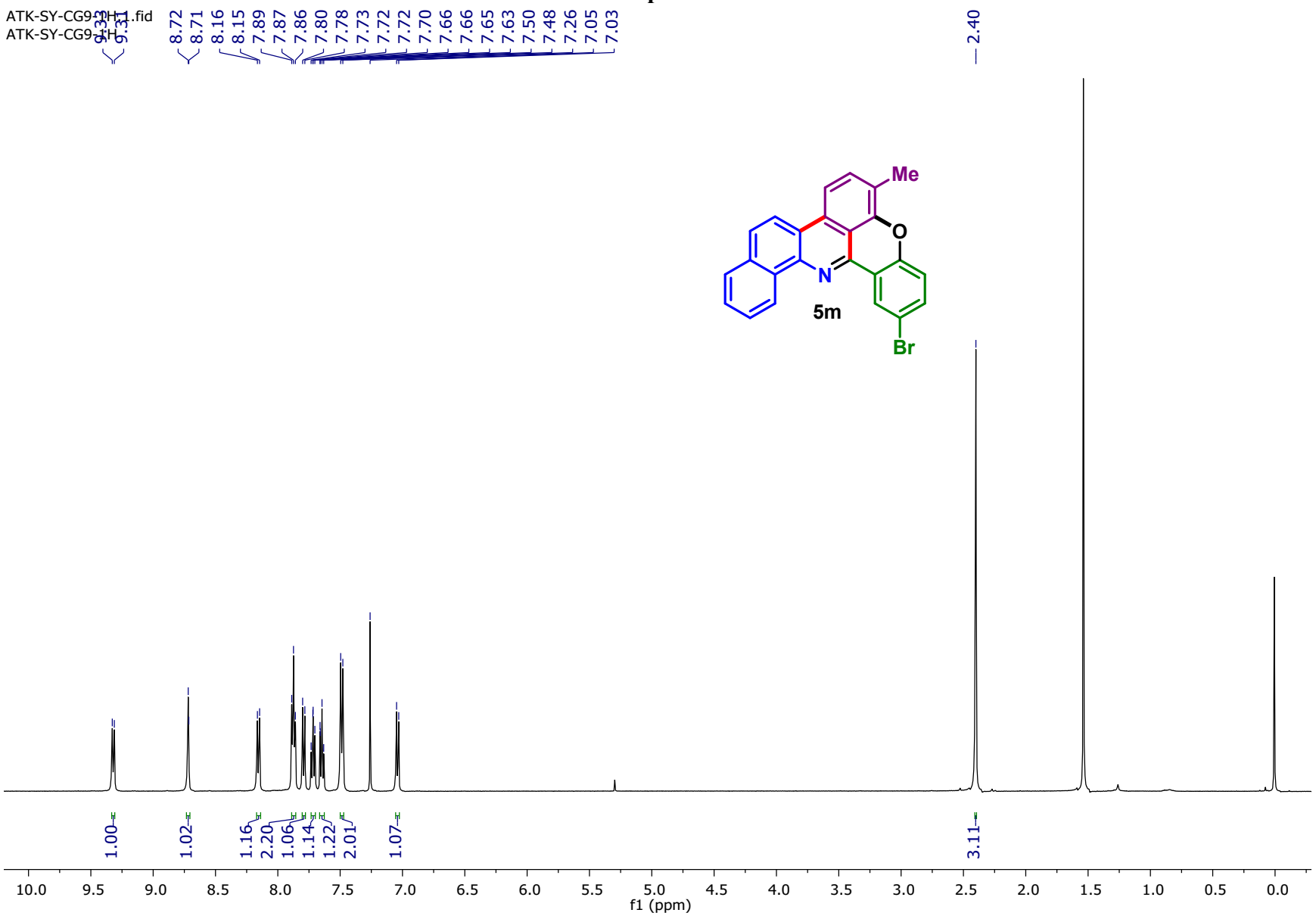


# HRMS Spectra of 5l

| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SAMPLE | **Position** | P2-C3 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-CG10-R.d |
| **ACQ Method** | ESI ALS 200-600.m | **Comment** | | **Acquired Time** | 30-Aug-21 04:26:19 PM (UTC+05:30) |



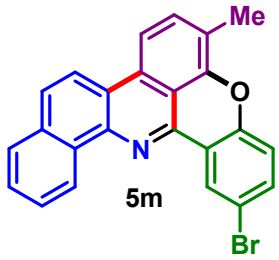
## $^{1}$H NMR Spectra of 5m



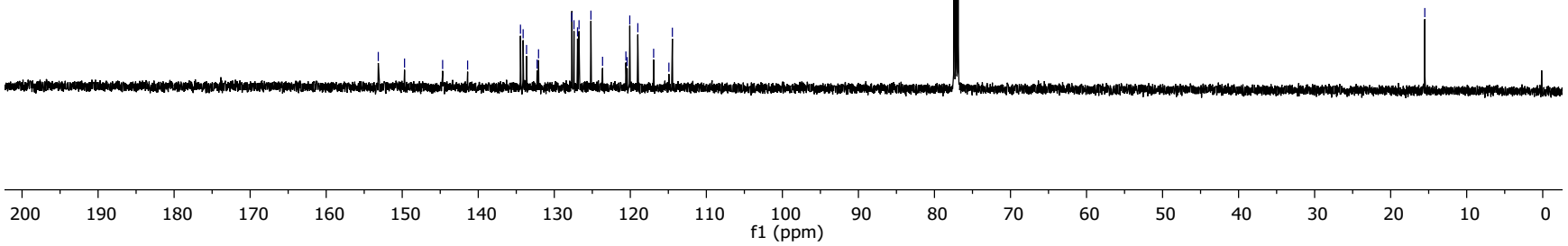
## $^{13}$C NMR Spectra of 5m

ATK-SY-CG9-13C.1.fid
ATK-SY-CG9-13C

153.15 149.70 144.69 141.40 134.45 134.10 133.65 132.26 132.07 127.69 127.41 126.94 126.75 125.18 123.65 120.58 120.42 120.07 119.01 116.93 114.93 114.47 77.41 77.16 76.91 15.51

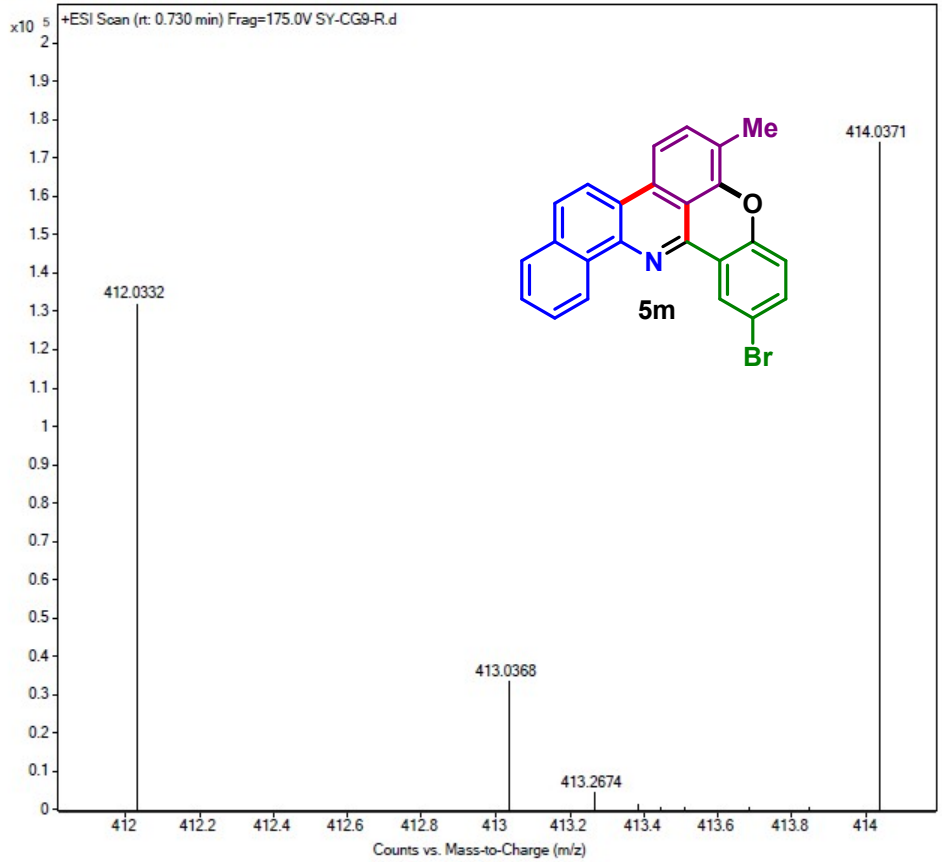



5m

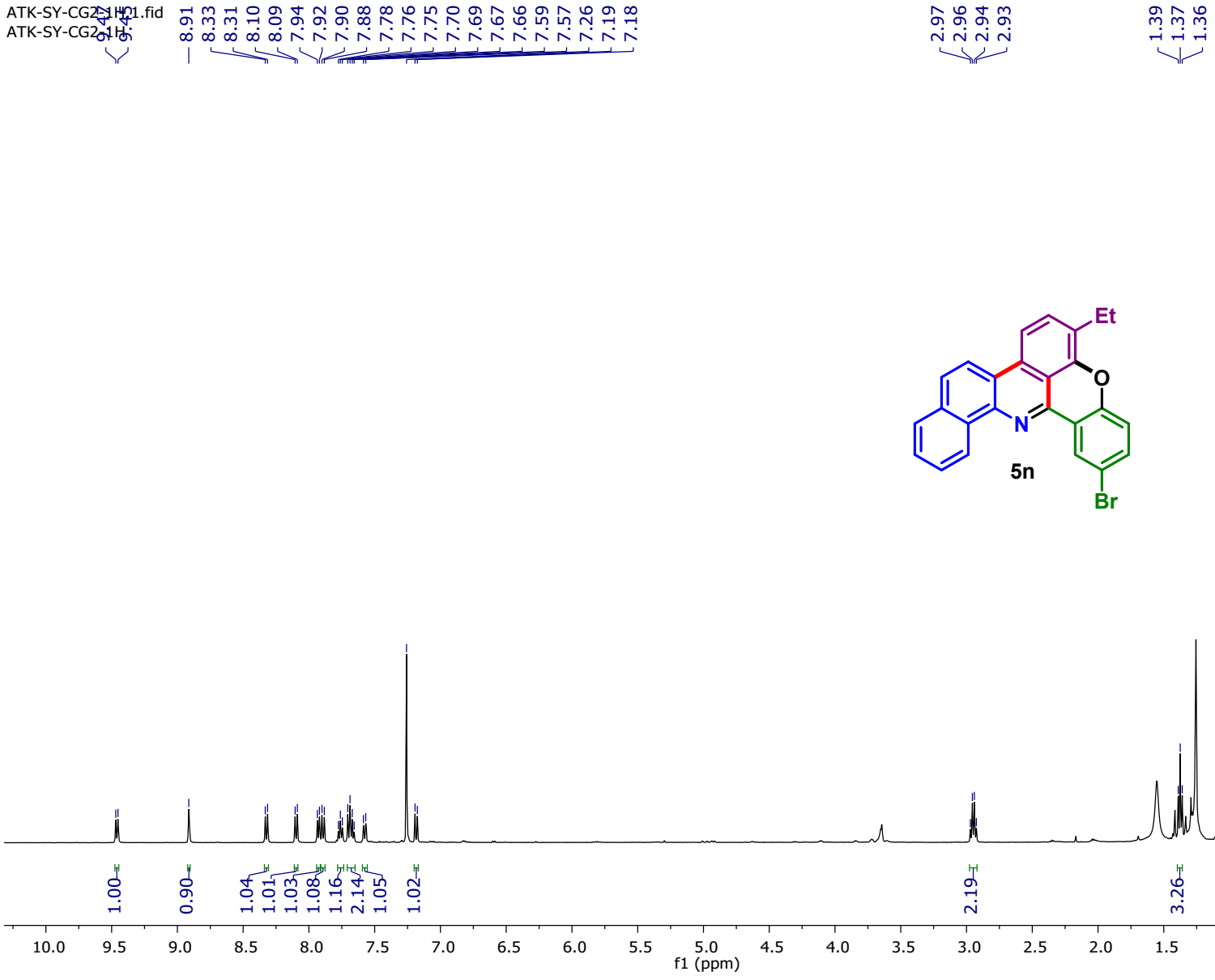


# HRMS Spectra of 5m

| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SAMPLE | **Position** | P2-C2 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-CG9-R.d |
| **ACQ Method** | ESI ALS 200-600.m | **Comment** | | **Acquired Time** | 30-Aug-21 04:17:00 PM (UTC+05:30) |



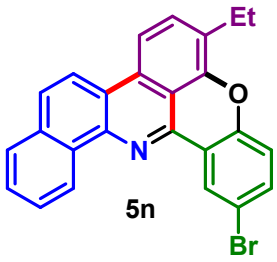
## $^{1}$H NMR Spectra of 5n



## $^{13}$C NMR Spectra of 5n

ATK-SY-CG2-13C.1.fid
ATK-SY-CG2-13C

153.30, 149.51, 145.07, 141.61, 134.63, 133.73, 132.80, 132.49, 132.14, 127.87, 127.77, 127.53, 127.13, 126.89, 126.83, 125.21, 123.84, 120.57, 120.19, 119.17, 117.05, 115.29, 114.92, 77.41, 77.16, 76.91, 22.87, 14.32

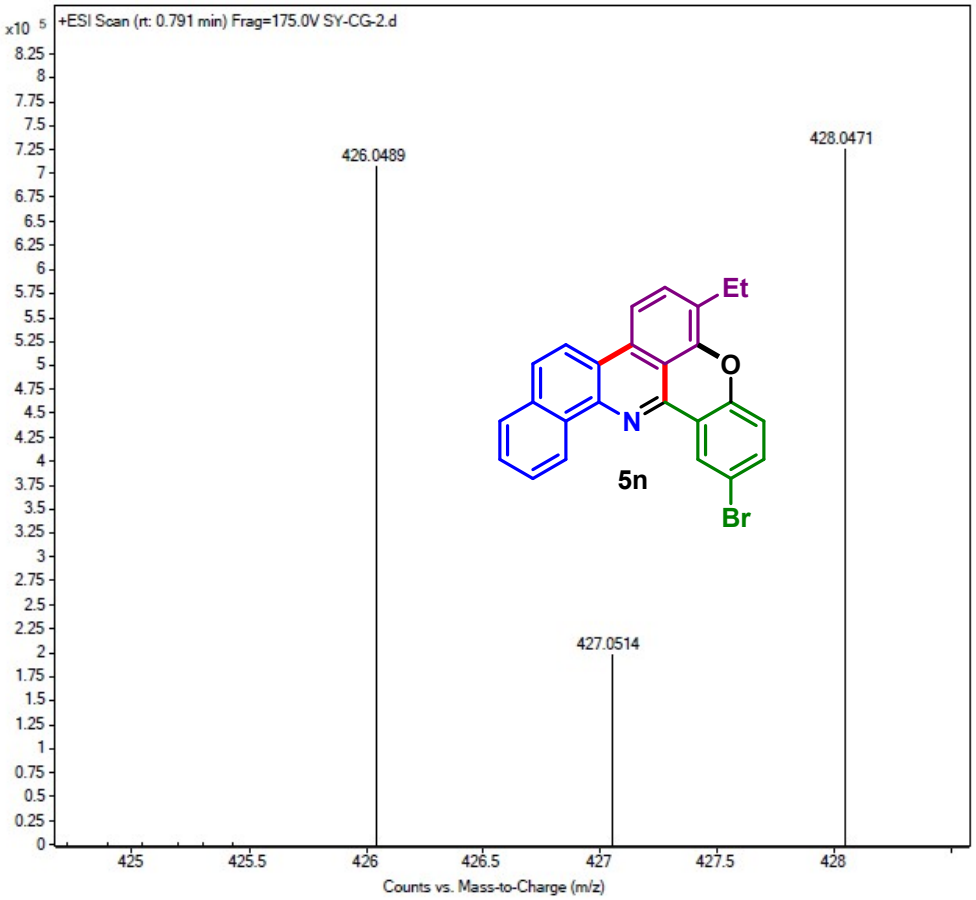



5n

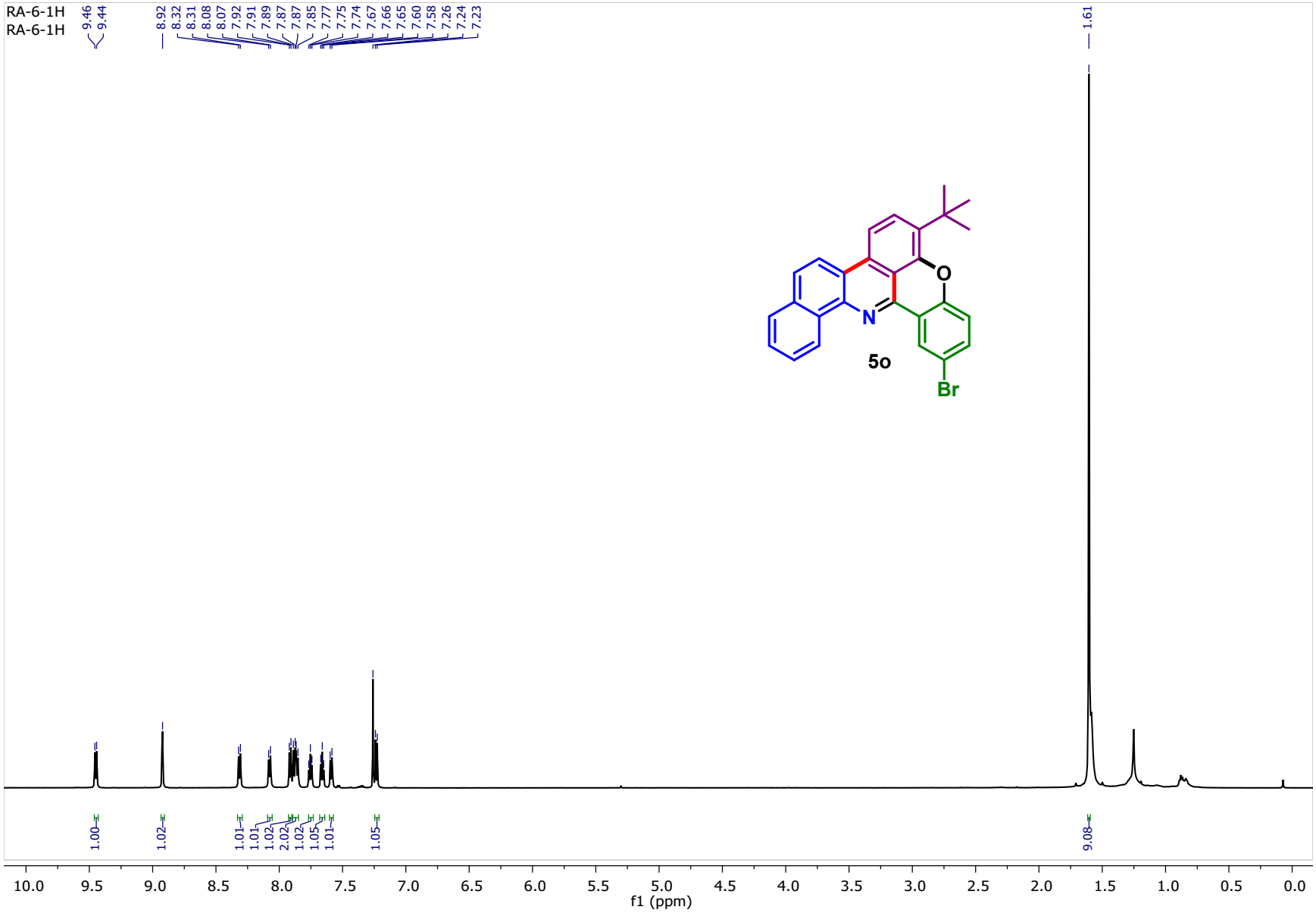
f1 (ppm)

# HRMS Spectra of 5n

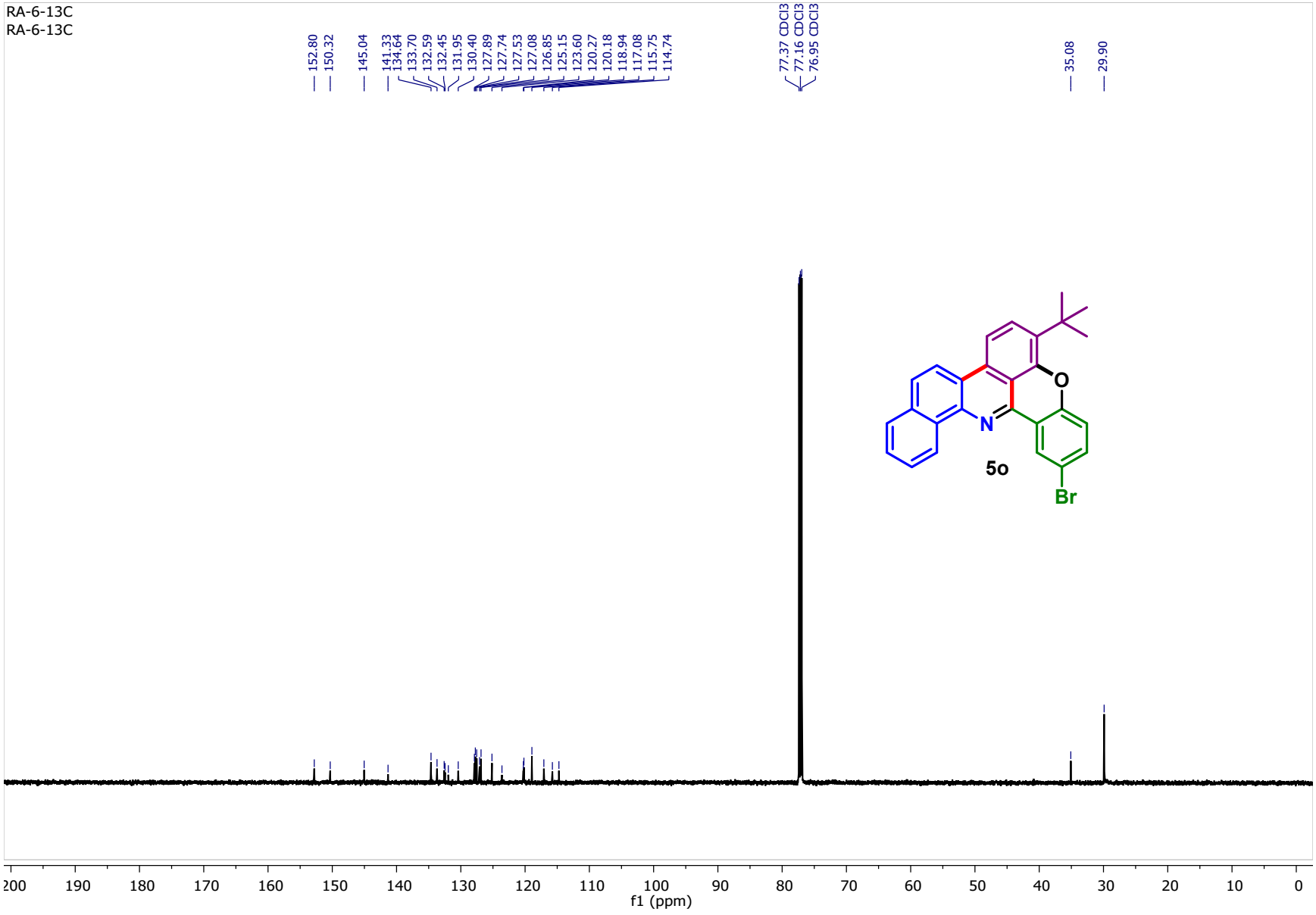
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | WASH | **Position** | P1-E4 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-CG-2.d |
| **ACQ Method** | ESI ALS 100-1000.m | **Comment** | | **Acquired Time** | 30-Aug-21 12:00:41 PM (UTC+05:30) |



## $^1$H NMR Spectra of 5o

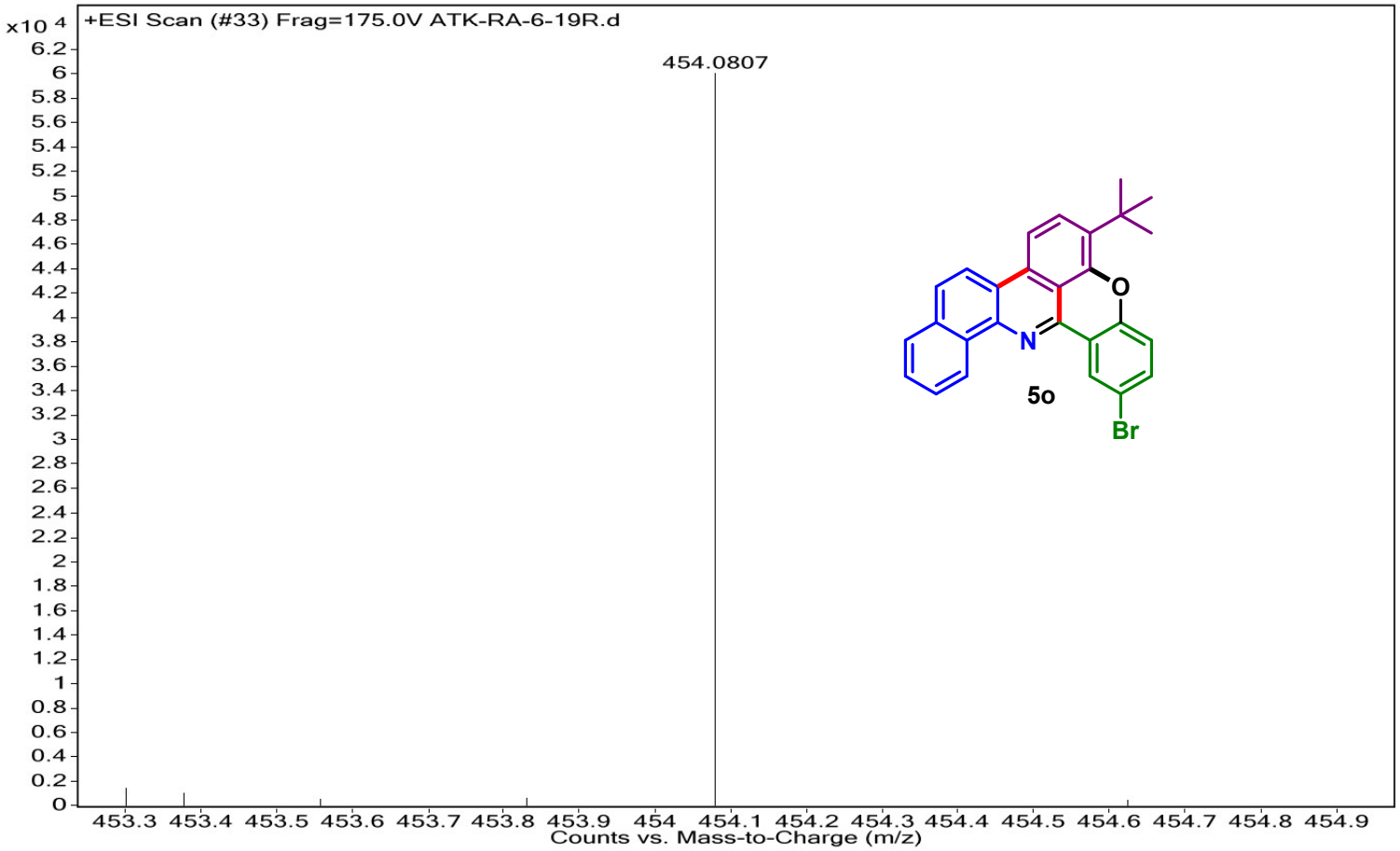


## $^{13}$C NMR Spectra of 5o

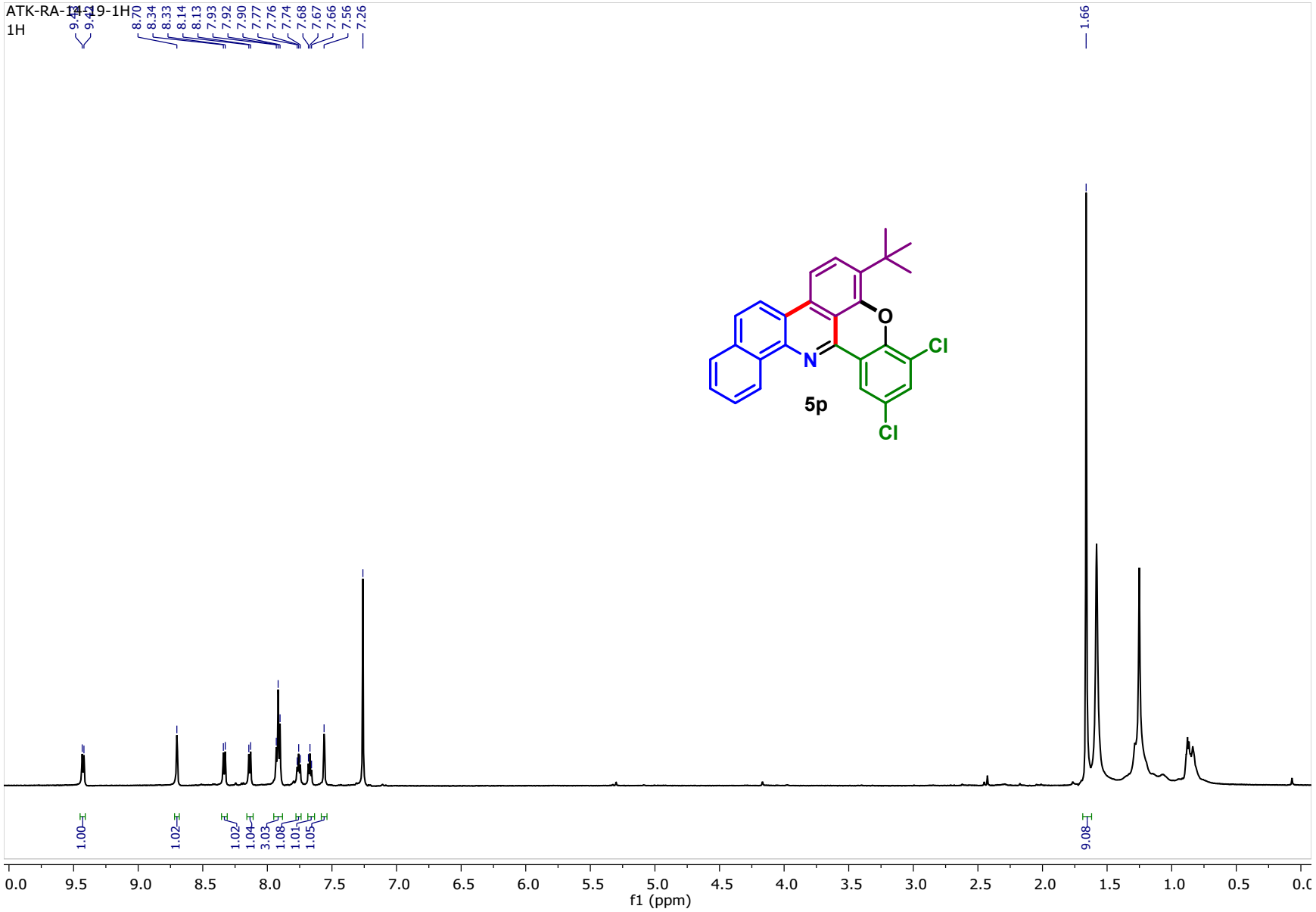


# HRMS Spectra of 5o

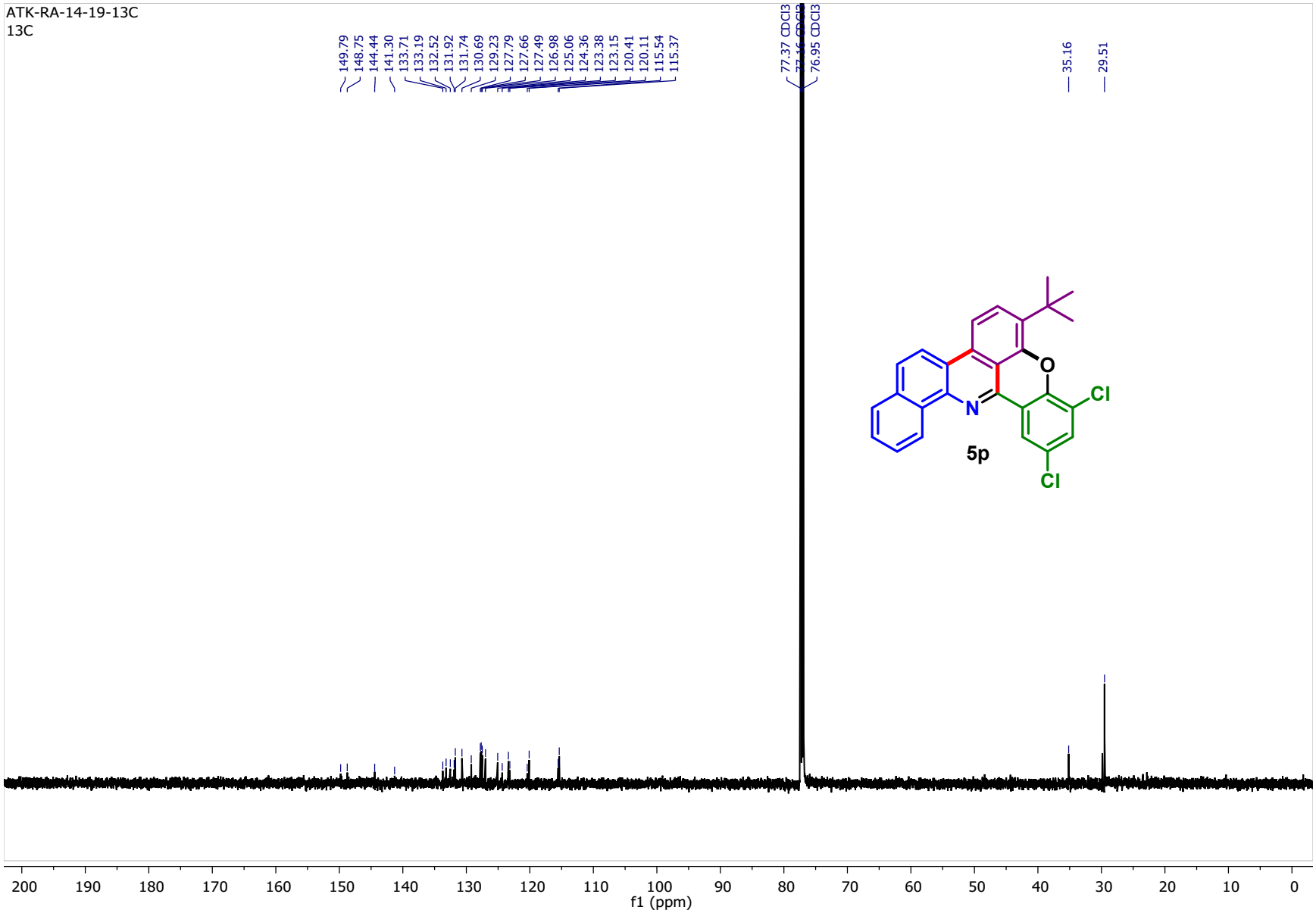
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Sample Name** | SAMPLE 2 | **Position** | P1-A3 | **Instrument Name** | Instrument 1 | **User Name** | |
| **Inj Vol** | 20 | **InjPosition** | | **SampleType** | Sample | **IRM Calibration Status** | Success |
| **Data Filename** | ATK-RA-6-19R.d | **ACQ Method** | ESI ALS 200-600.m | **Comment** | | **Acquired Time** | 1/21/2019 3:52:32 PM |



## $^{1}$H NMR Spectra of 5p

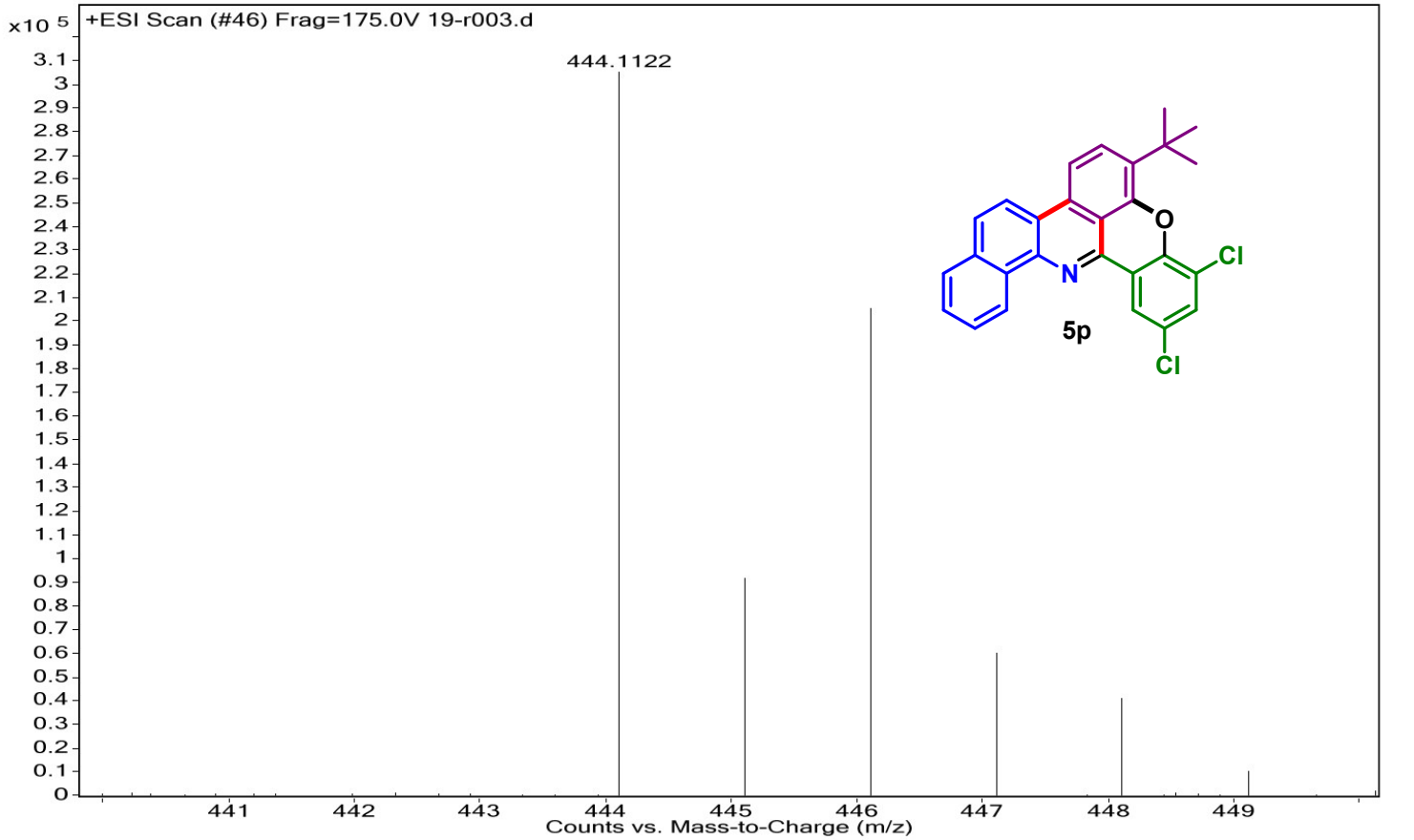


## $^{13}$C NMR Spectra of 5p

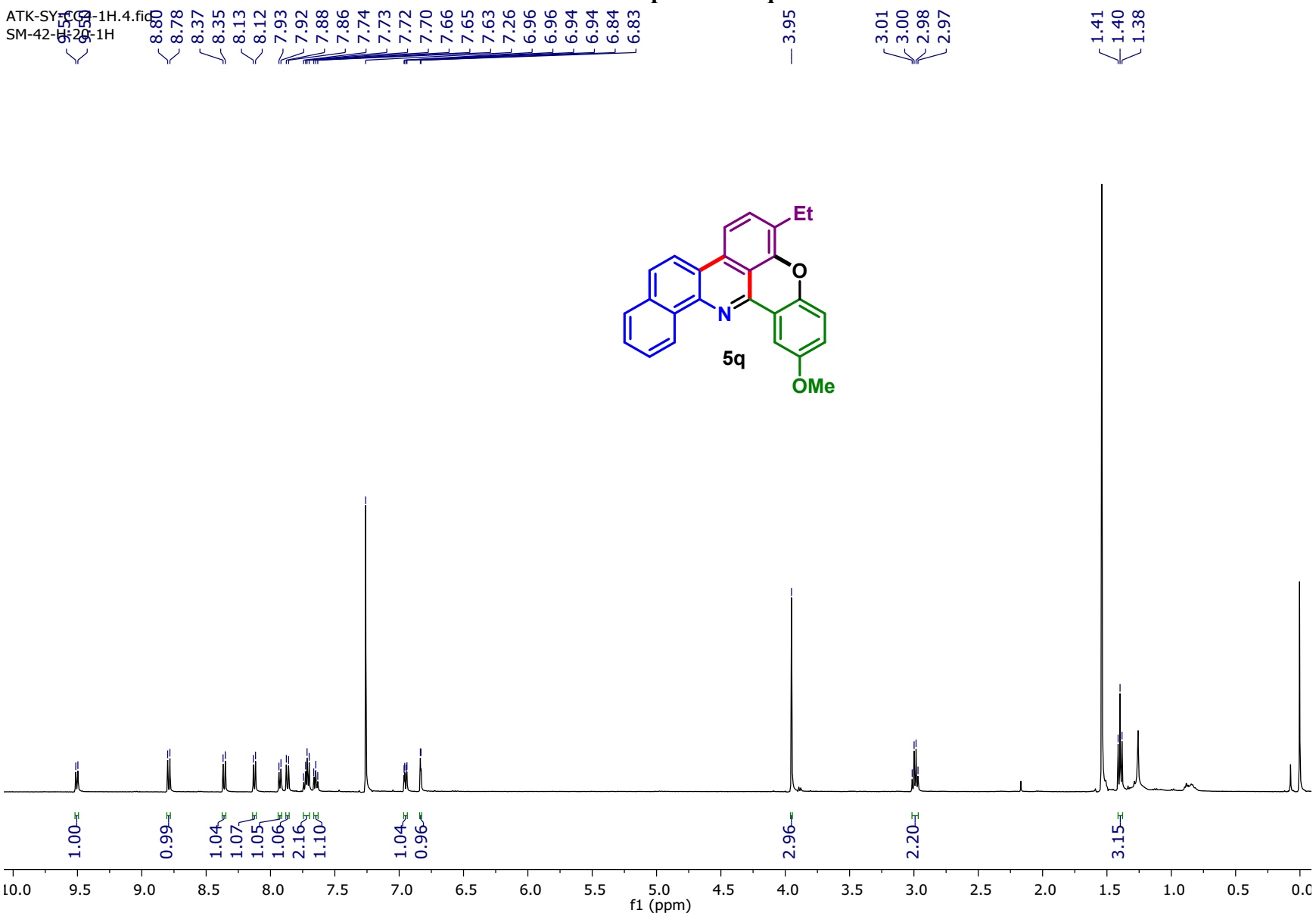


# HRMS Spectra of 5p

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Sample Name** | SAMPLE | **Position** | P1-A6 | **Instrument Name** | Instrument 1 | **User Name** | |
| **Inj Vol** | 20 | **InjPosition** | | **SampleType** | Sample | **IRM Calibration Status** | Success |
| **Data Filename** | 19-r003.d | **ACQ Method** | ESI ALS 100-600.m | **Comment** | | **Acquired Time** | 1/30/2020 8:21:01 PM |



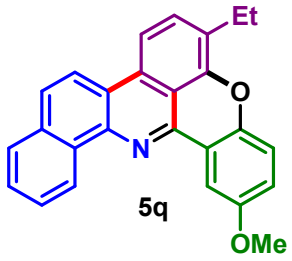
## $^1$H NMR Spectra of 5q



## $^{13}$C NMR Spectra of 5q

ATK-SY-CG4-13C.6.fid
ATK-SY-CG4-13C

163.13
155.74
150.17
146.74
141.95
133.82
132.49
132.20
127.73
127.28
126.69
126.62
126.57
126.11
125.22
120.38
119.89
115.43
115.14
114.71
112.30
100.99
77.41
77.16
76.91
55.83
22.97
14.39

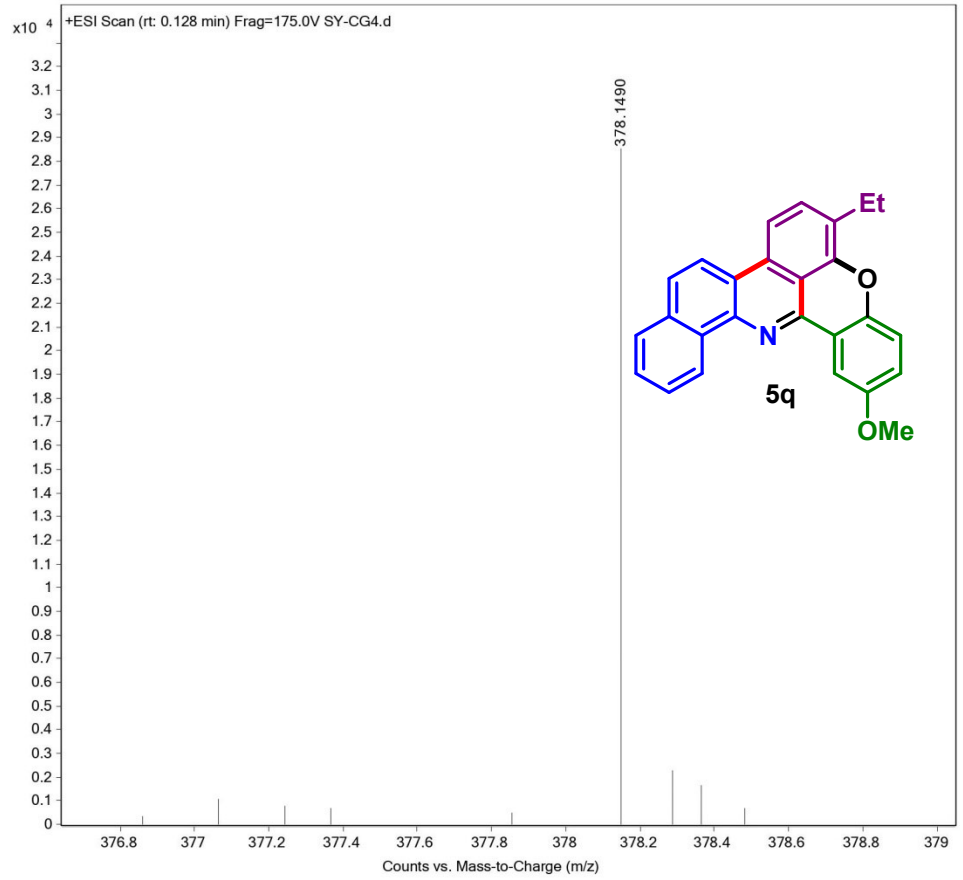



5q

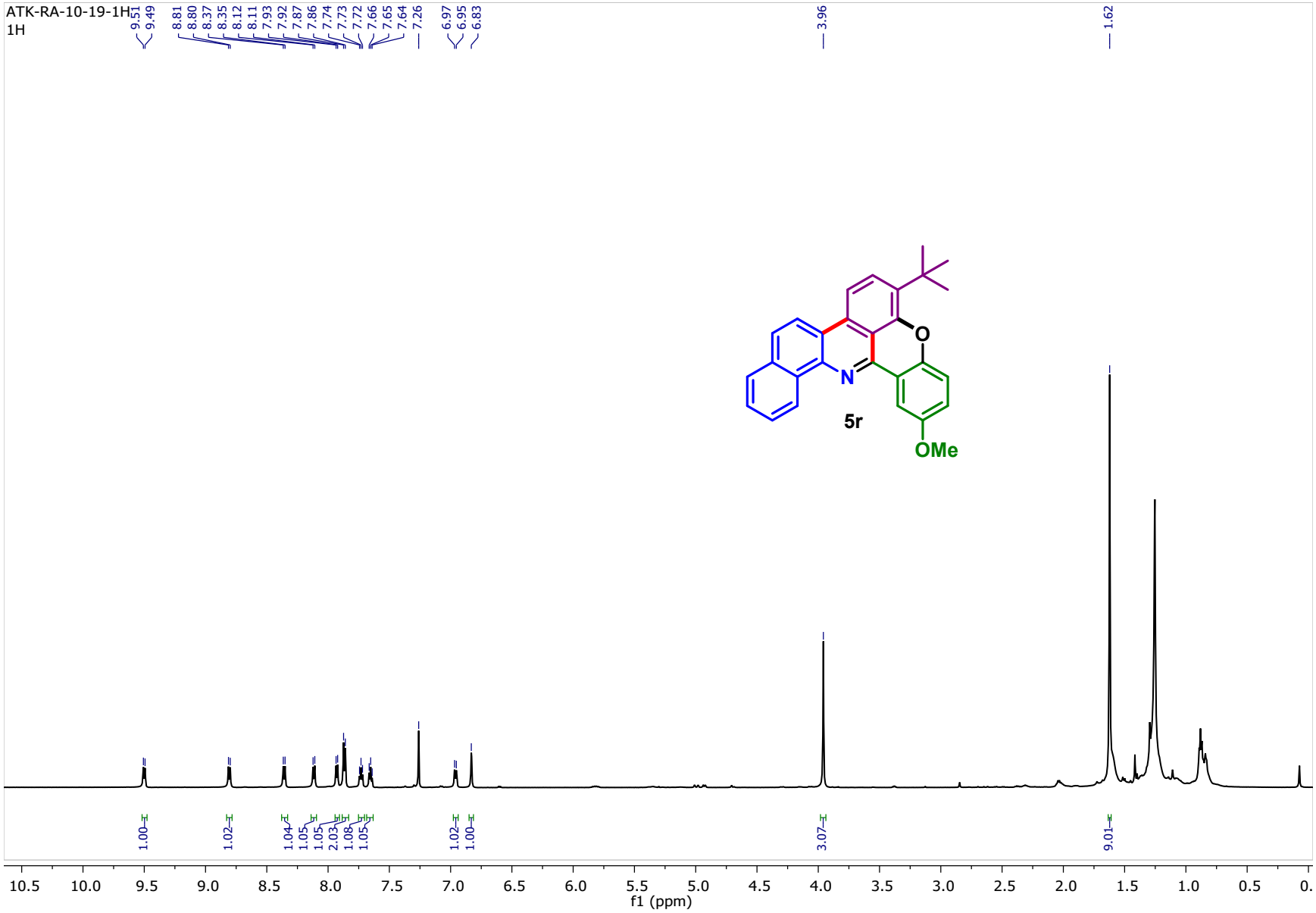
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

# HRMS Spectra of 5q

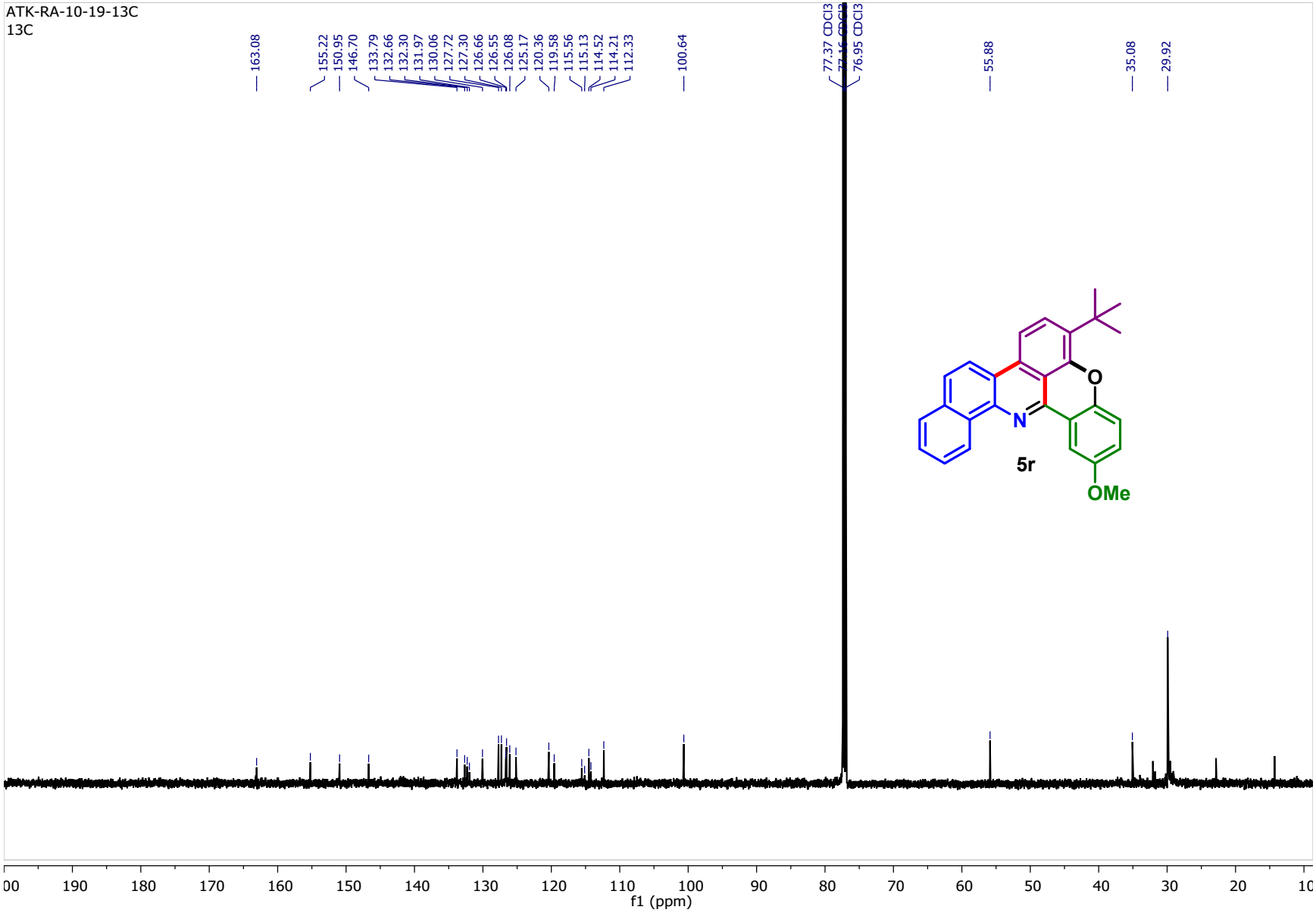
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SY-CG4 | **Position** | P1-D3 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 20 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | SY-CG4.d |
| **ACQ Method** | ESI ALS 100-1000.m | **Comment** | | **Acquired Time** | 05-May-21 04:22:21 PM (UTC+05:30) |



## $^{1}$H NMR Spectra of 5r

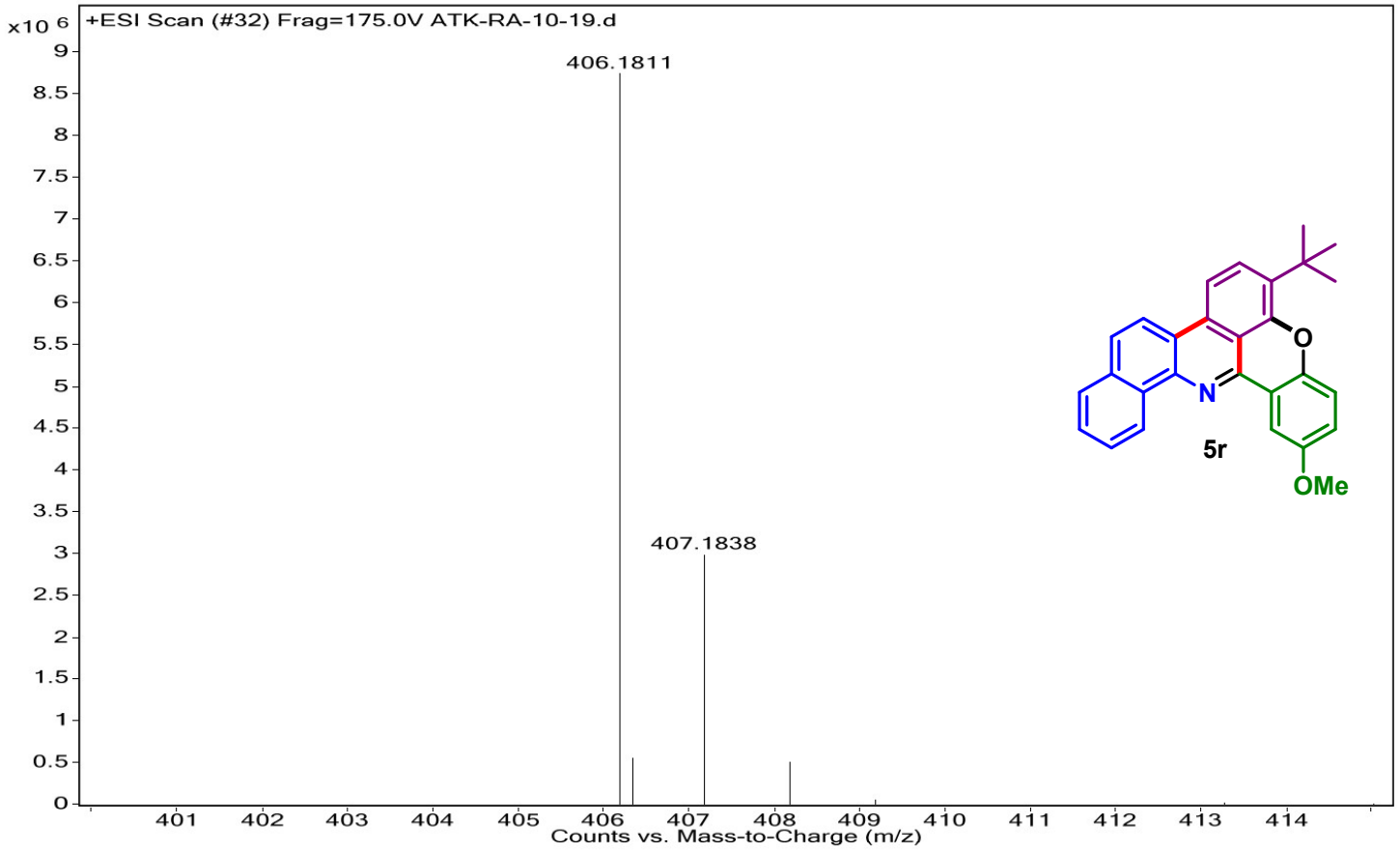


## $^{13}$C NMR Spectra of 5r

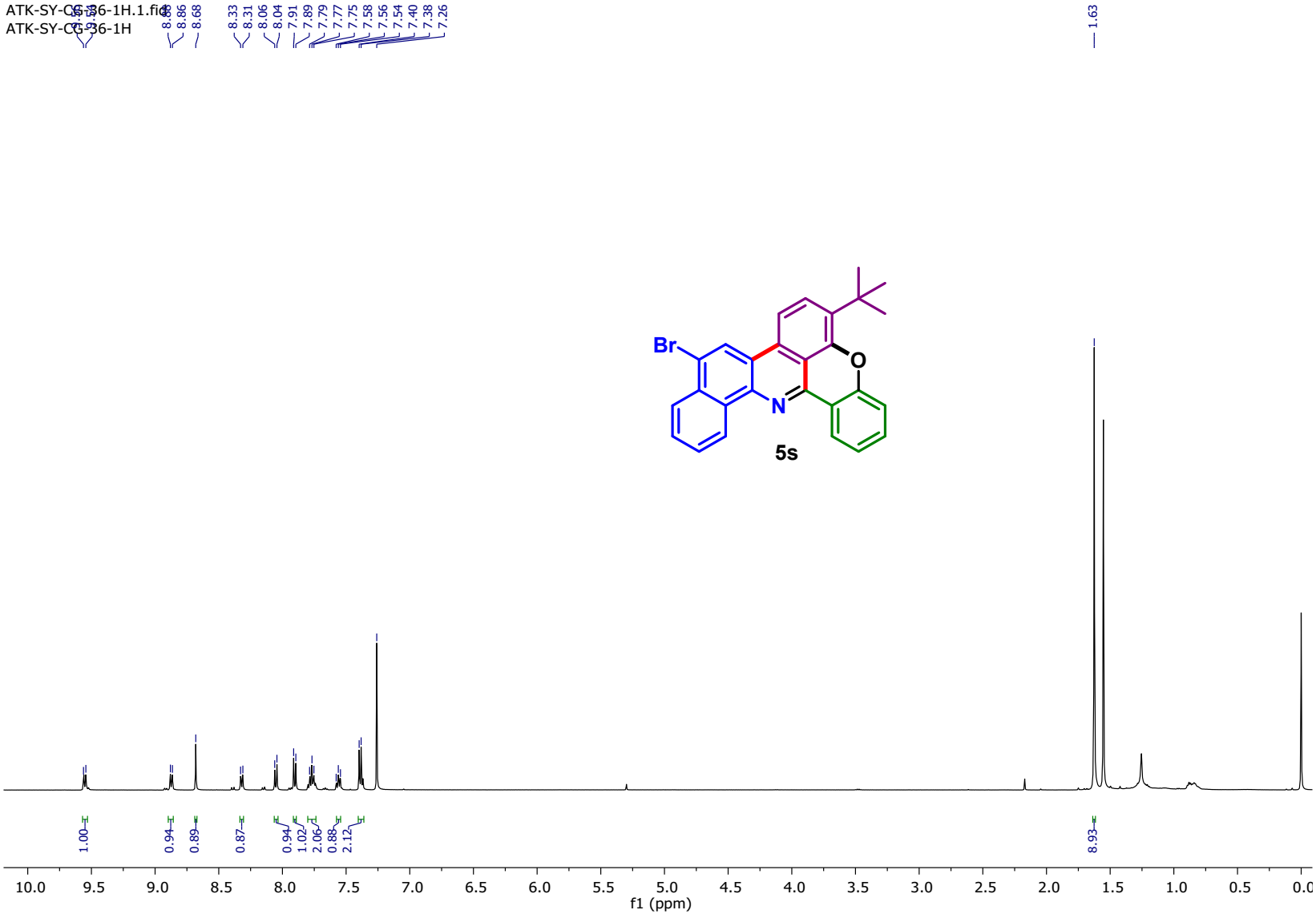


# HRMS Spectra of 5r

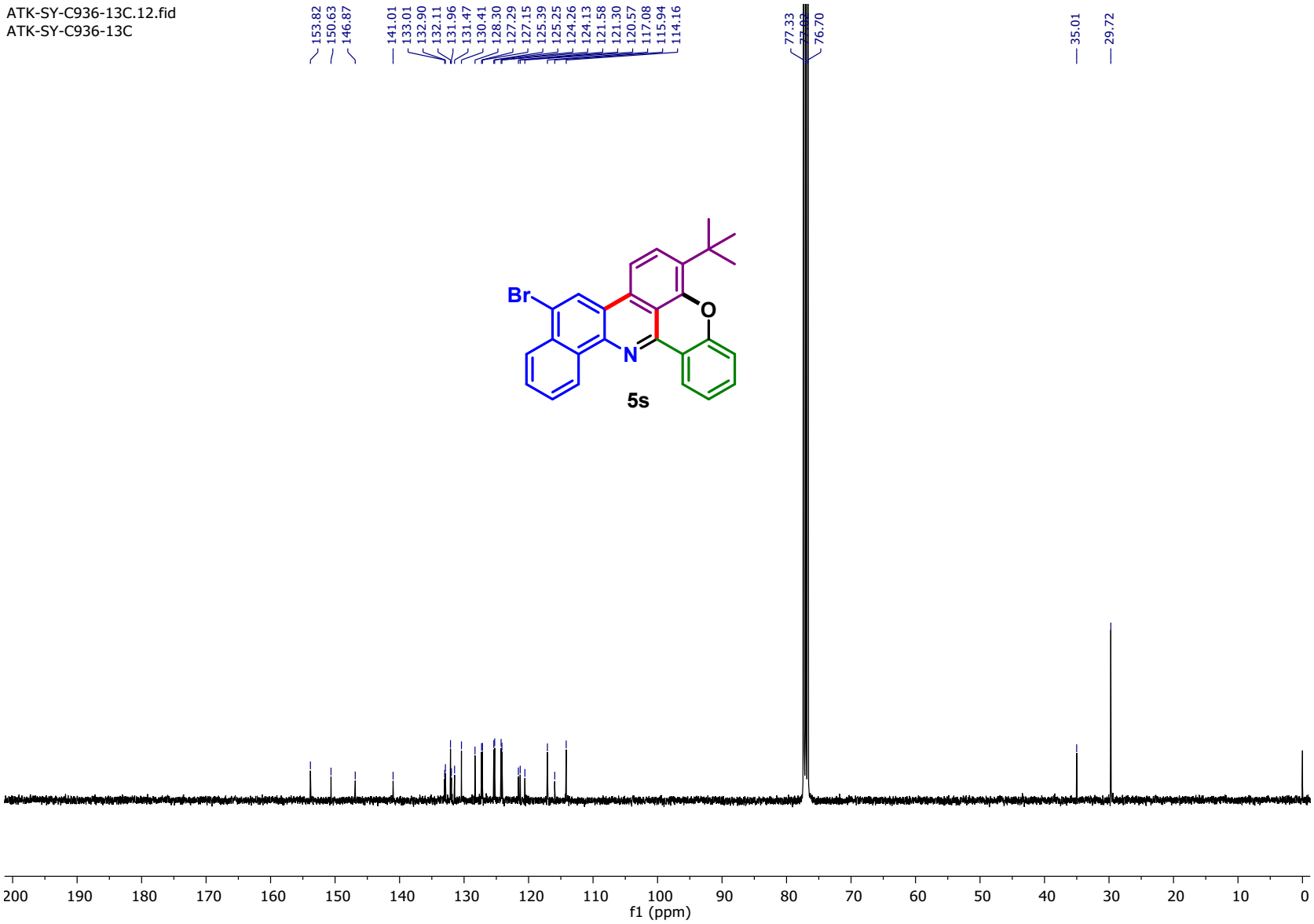
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Sample Name** | ATK-RA-10-19 | **Position** | P1-D7 | **Instrument Name** | Instrument 1 | **User Name** | |
| **Inj Vol** | 20 | **InjPosition** | | **SampleType** | Sample | **IRM Calibration Status** | Success |
| **Data Filename** | ATK-RA-10-19.d | **ACQ Method** | ESI ALS 100-600.m | **Comment** | | **Acquired Time** | 4/16/2019 5:11:09 PM |



# $^{1}$H NMR Spectra of 5s



## $^{13}$C NMR Spectra of 5s



**HRMS Spectra of 5s**

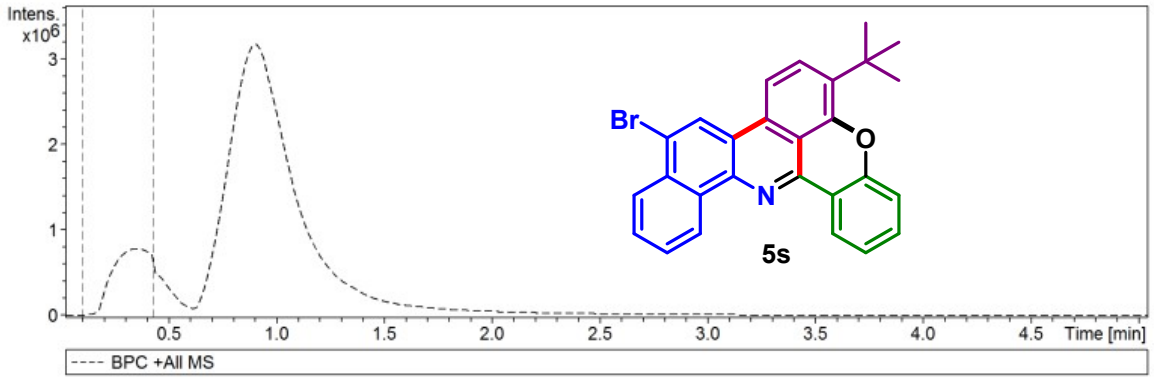
# Display Report

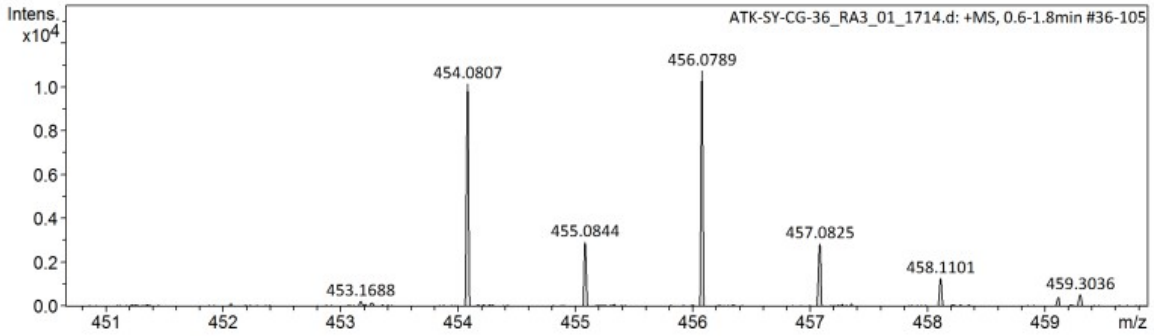
**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\user data\HPLC\DR LOKMAN\PRABHAS\ATK-23-3-22\ATK-SY-CG-36_RA3_01_1714.d | Acquisition Date | 3/23/2022 11:25:34 AM |
| Method | low mass bruker.m | Operator | vidhi |
| Sample Name | ATK-SY-CG-36 | Instrument | impact HD 1819696.00197 |
| Comment | | | |

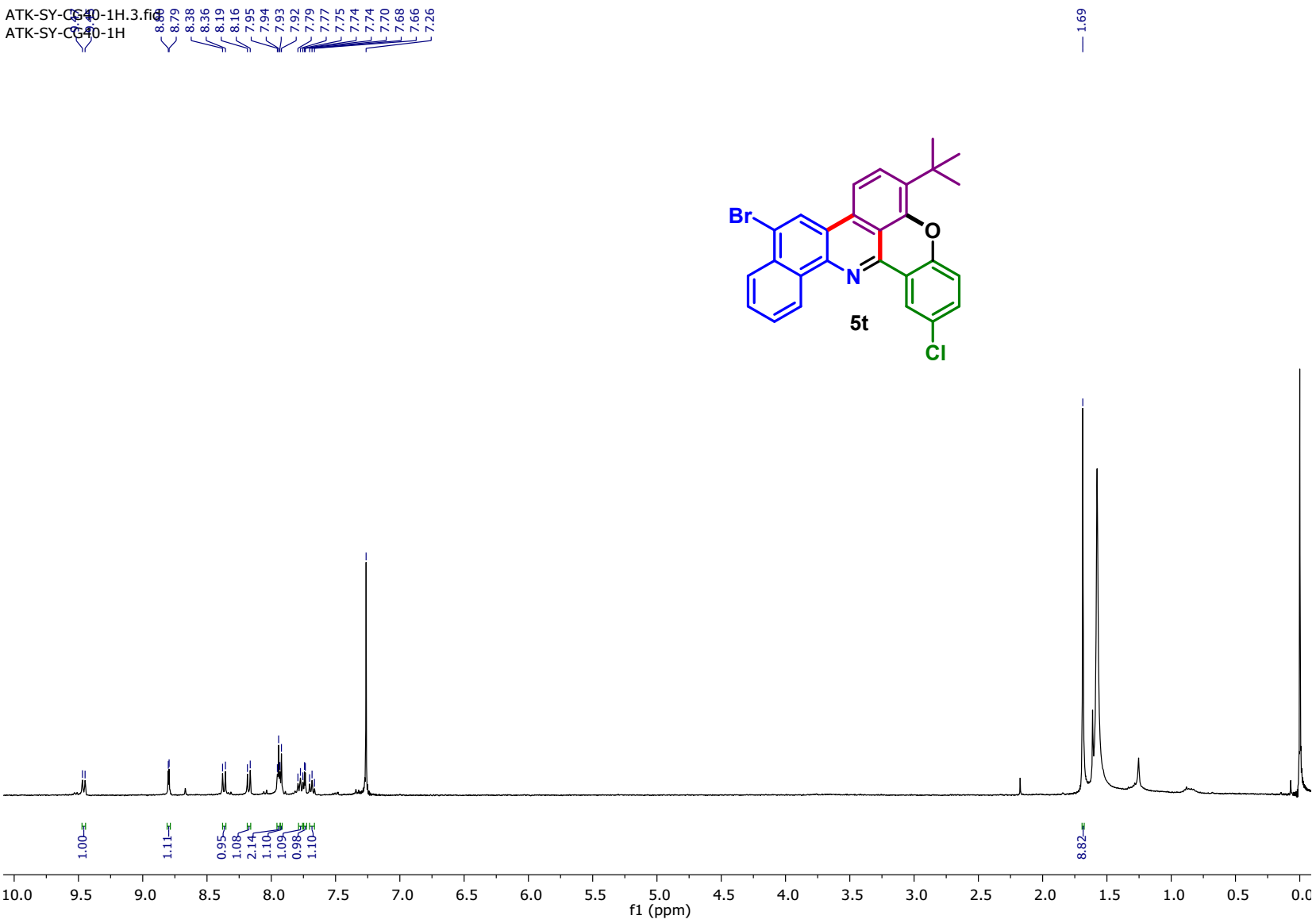
**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.8 Bar |
| Focus | Active | Set Capillary | 4500 V | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 6.0 l/min |
| Scan End | 1500 m/z | Set Charging Voltage | 2000 V | Set Divert Valve | Waste |
| | | Set Corona | 0 nA | Set APCI Heater | 0 °C |

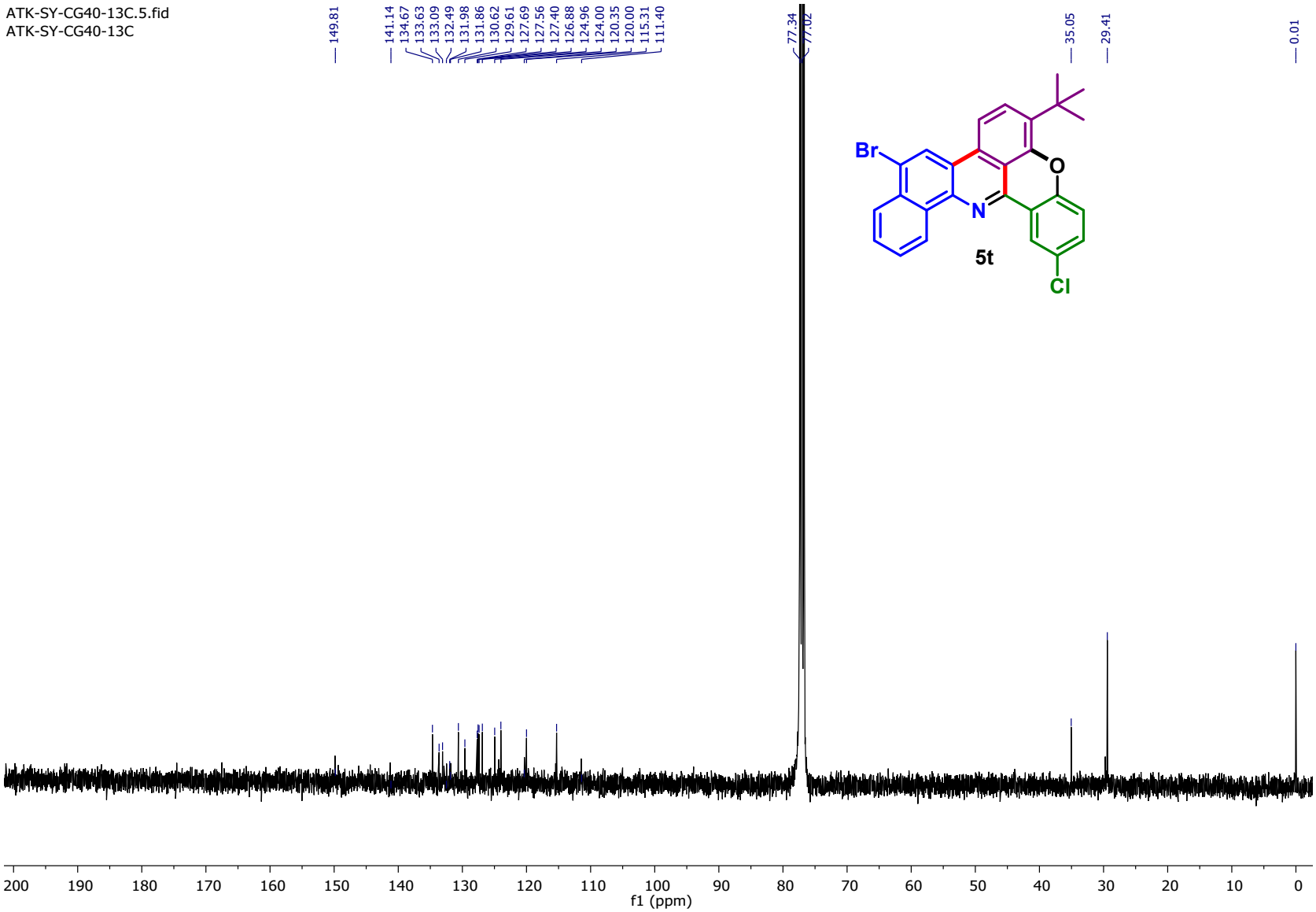


## $^{1}$H NMR Spectra of 5t

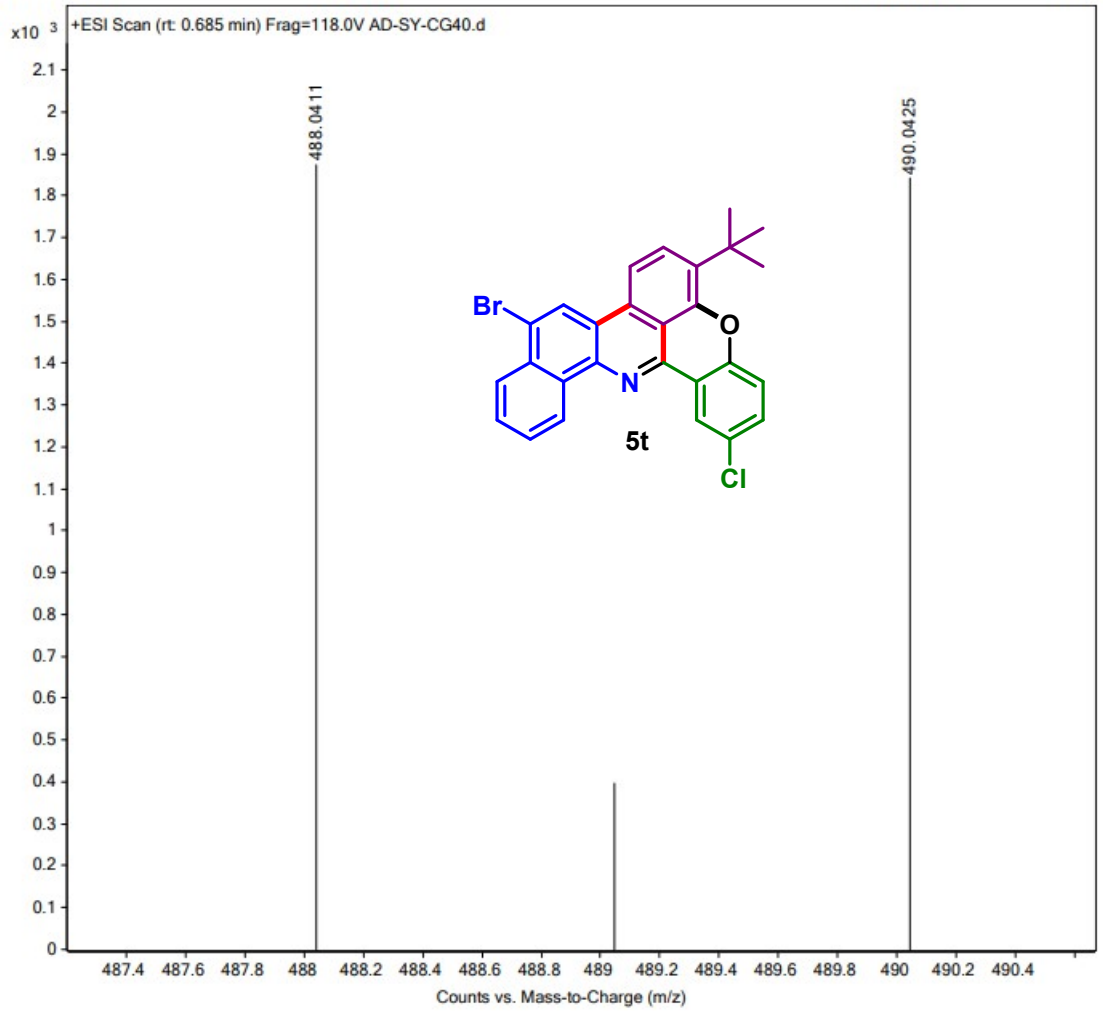


## $^{13}$C NMR Spectra of 5t



# HRMS Spectra of 5t

| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | AD-SY-CG40 | **Position** | Vial 24 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 0.1 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Some Ions Missed | **Data Filename** | AD-SY-CG40.d |
| **ACQ Method** | Direct Mass-2017.m | **Comment** | | **Acquired Time** | 28-03-2022 21:10:58 (UTC+05:30) |

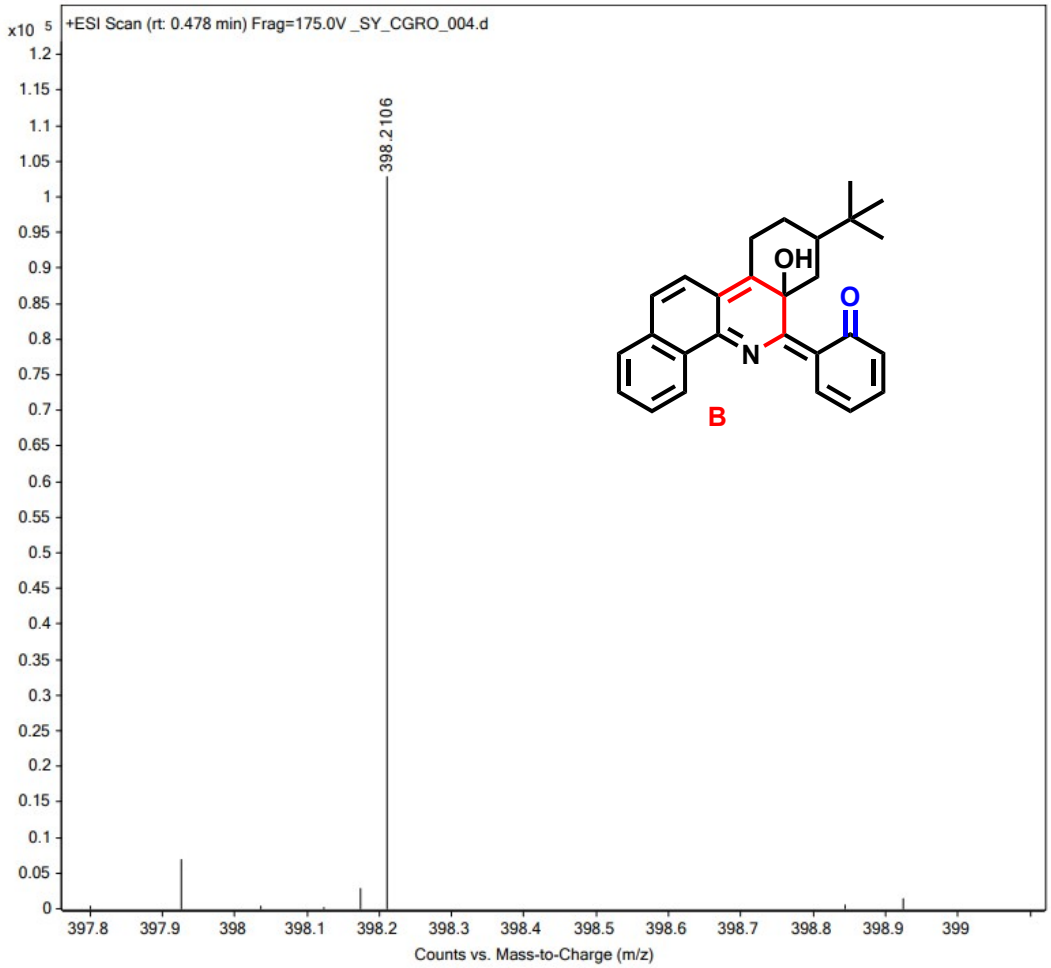


**Detection of intermediates in HRMS**

0.10 mmol (37 mg) of 2-(8-(*tert*-butyl)-7,8,9,10-tetrahydrobenzo[*c*]phenanthridin-6-yl)phenol **(4e)** was stirred in DMSO in a 10 mL r.b at 120 $^{o}$C temperature. After 2.5 h. the reaction mixture was subjected to ESI-MS mass experiment, and the intermediates **B**, **C**, **D**, **E**, **F**, **G**, **H**, **I** were detected by HRMS values. The observed m/e values are as follows: intermediate **B** - 398.2106 (expected 398.2115); intermediate **C, D, E** – 380.2000 (expected 380.2009); intermediate **F** – 396.1950 (expected 396.1959); intermediate **G, H** – 378.1853 (expected 378.1853); intermediate **I** – 394.1795 (expected 394.1802).

## HRMS Spectra of intermediate B

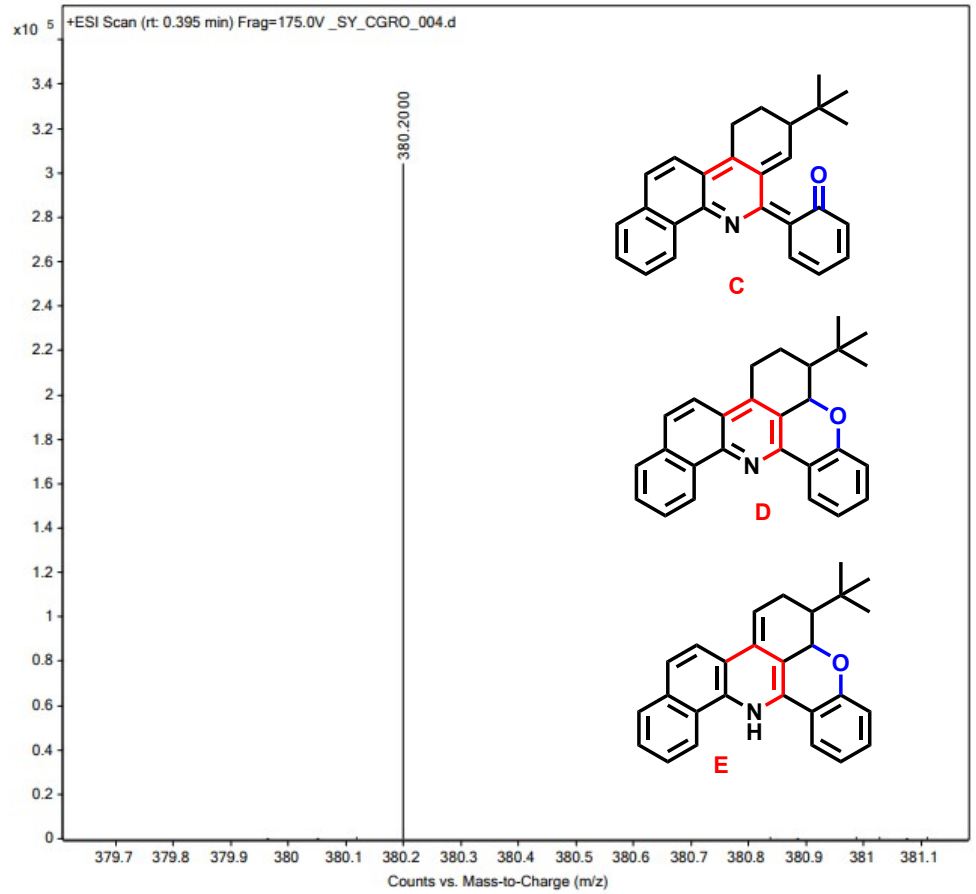
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SY_CGRO | **Position** | P1-A3 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 10 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | _SY_CGRO_004.d |
| **ACQ Method** | FULL SCAN-POSITIVE.m | **Comment** | | **Acquired Time** | 31-03-2022 21:48:53 (UTC+05:30) |




**Expected [M+H$^+$]** for $C_{27}H_{28}NO_2$: 398.2115. **Found**: 398.2106.

## HRMS Spectra of intermediate C or D or E

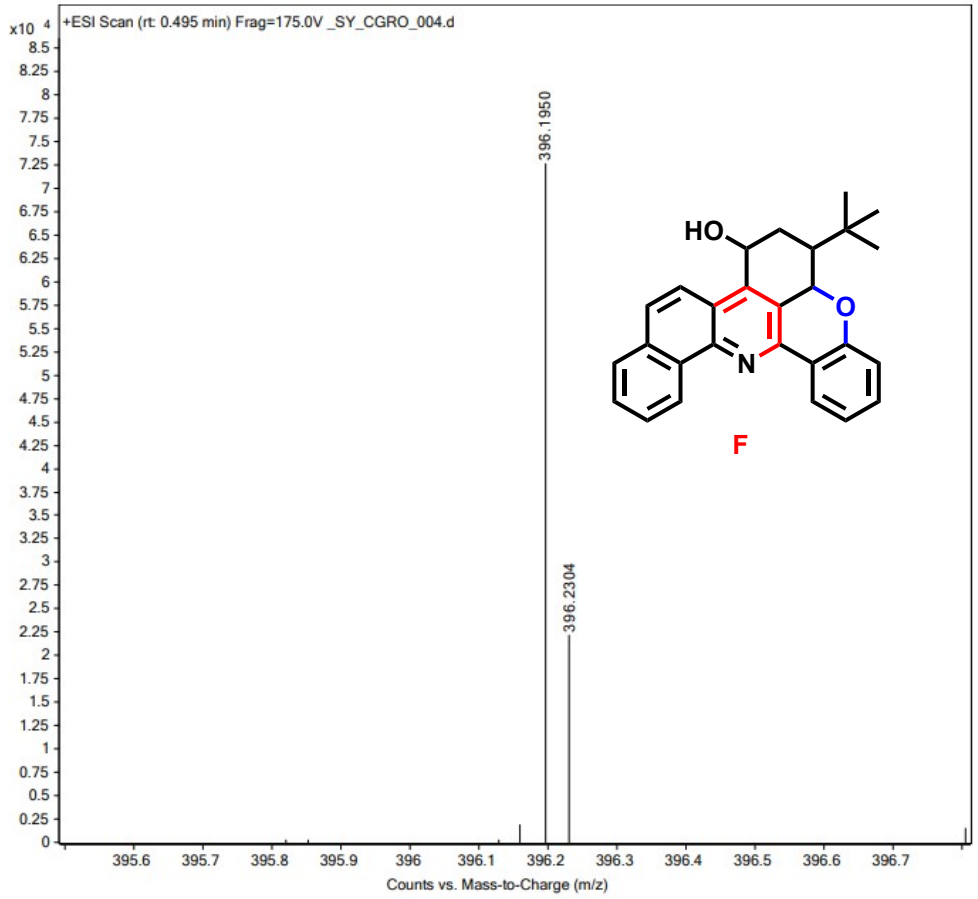
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SY_CGRO | **Position** | P1-A3 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 10 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | _SY_CGRO_004.d |
| **ACQ Method** | FULL SCAN-POSITIVE.m | **Comment** | | **Acquired Time** | 31-03-2022 21:48:53 (UTC+05:30) |




**Expected [M+H$^+$]** for $C_{27}H_{26}NO$: 380.2009. **Found**: 380.2000.

## HRMS Spectra of intermediate F

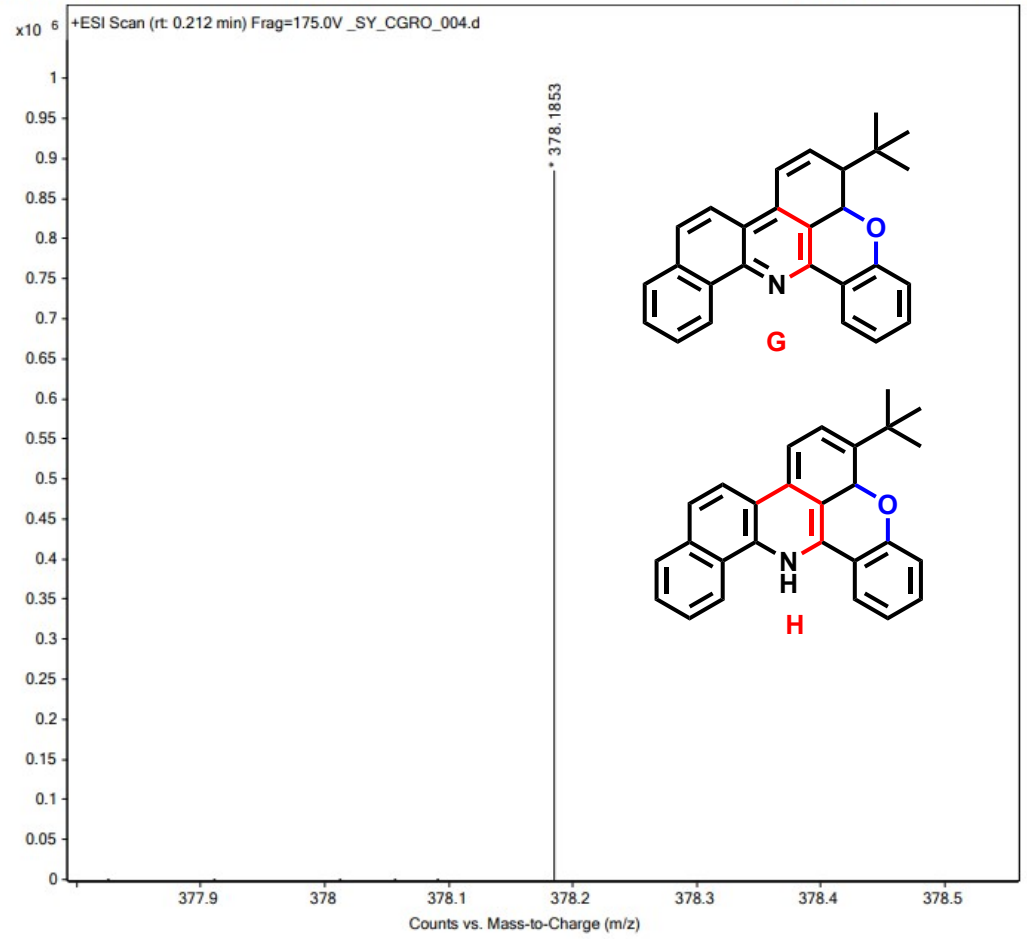
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SY_CGRO | **Position** | P1-A3 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 10 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | _SY_CGRO_004.d |
| **ACQ Method** | FULL SCAN-POSITIVE.m | **Comment** | | **Acquired Time** | 31-03-2022 21:48:53 (UTC+05:30) |




**Expected [M+H$^+$]** for $C_{27}H_{26}NO_2$: 396.1959. **Found**: 396.1950.

## HRMS Spectra of intermediate G or H

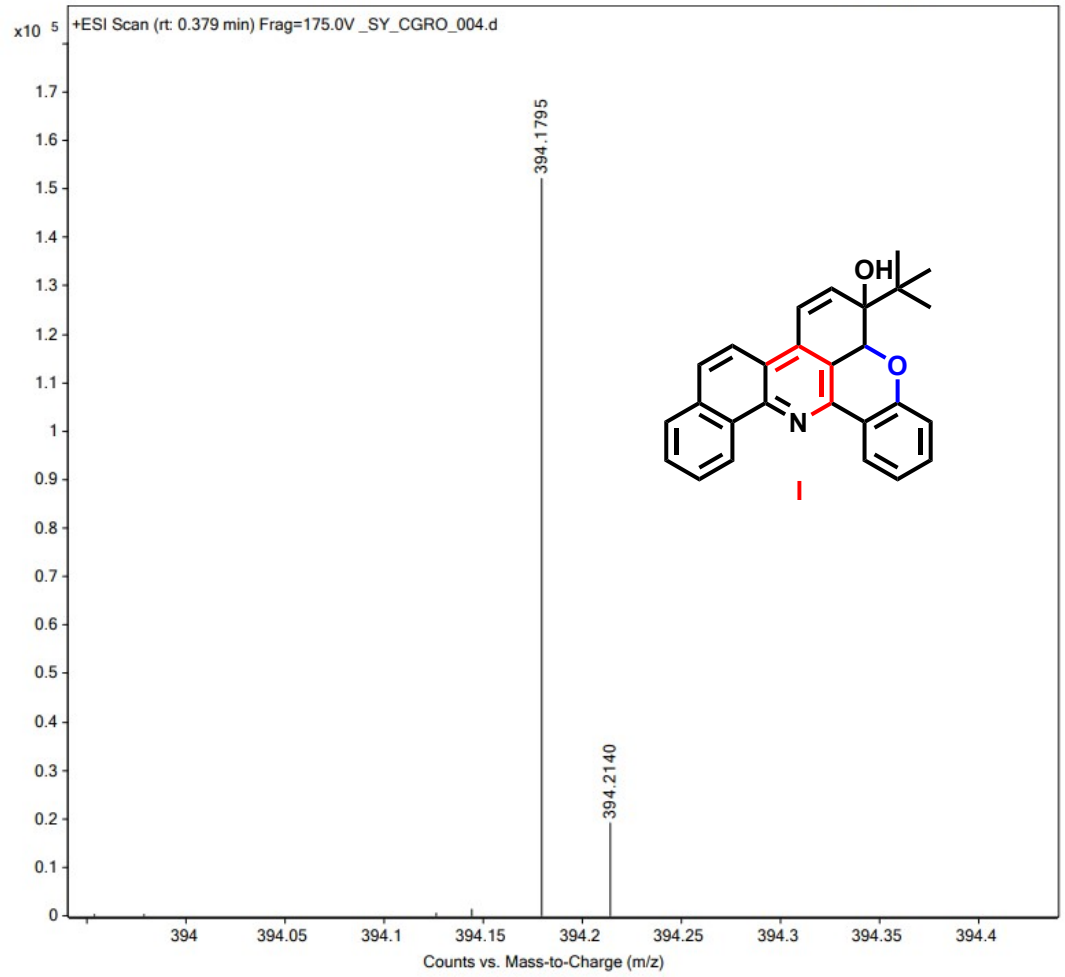
| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SY_CGRO | **Position** | P1-A3 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 10 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | _SY_CGRO_004.d |
| **ACQ Method** | FULL SCAN-POSITIVE.m | **Comment** | | **Acquired Time** | 31-03-2022 21:48:53 (UTC+05:30) |




**Expected [M+H$^+$]** for $C_{27}H_{24}NO$: 378.1853. **Found**: 378.1853.

## HRMS Spectra of intermediate I

| | | | | | |
|---|---|---|---|---|---|
| **Sample Name** | SY_CGRO | **Position** | P1-A3 | **Instrument Name** | Instrument 1 |
| **User Name** | | **Inj Vol** | 10 | **InjPosition** | |
| **Sample Type** | Sample | **IRM Calibration Status** | Success | **Data Filename** | _SY_CGRO_004.d |
| **ACQ Method** | FULL SCAN-POSITIVE.m | **Comment** | | **Acquired Time** | 31-03-2022 21:48:53 (UTC+05:30) |




**Expected [M+H$^+$]** for $C_{27}H_{24}NO_2$: 394.1802. **Found**: 394.1795.