

## Synthesis of Unsymmetrical 2,4-Diaryl Substituted and 3-Bromo-2-(4-bromoalkoxy)-Derived Pyranocoumarins

Velu Saravanan,<sup>a</sup> Pin-Hui Lin,<sup>b</sup> and Ding-Yah Yang<sup>\*a,b</sup>

<sup>a</sup>Department of Chemistry, Tunghai University

<sup>b</sup>Graduate Program for Biomedical and Materials Science, Tunghai University  
No. 1727, Sec. 4, Taiwan Boulevard, Xitun District, Taichung 407224, Taiwan

\*Corresponding author; Email: [yang@thu.edu.tw](mailto:yang@thu.edu.tw)

## Supporting Information

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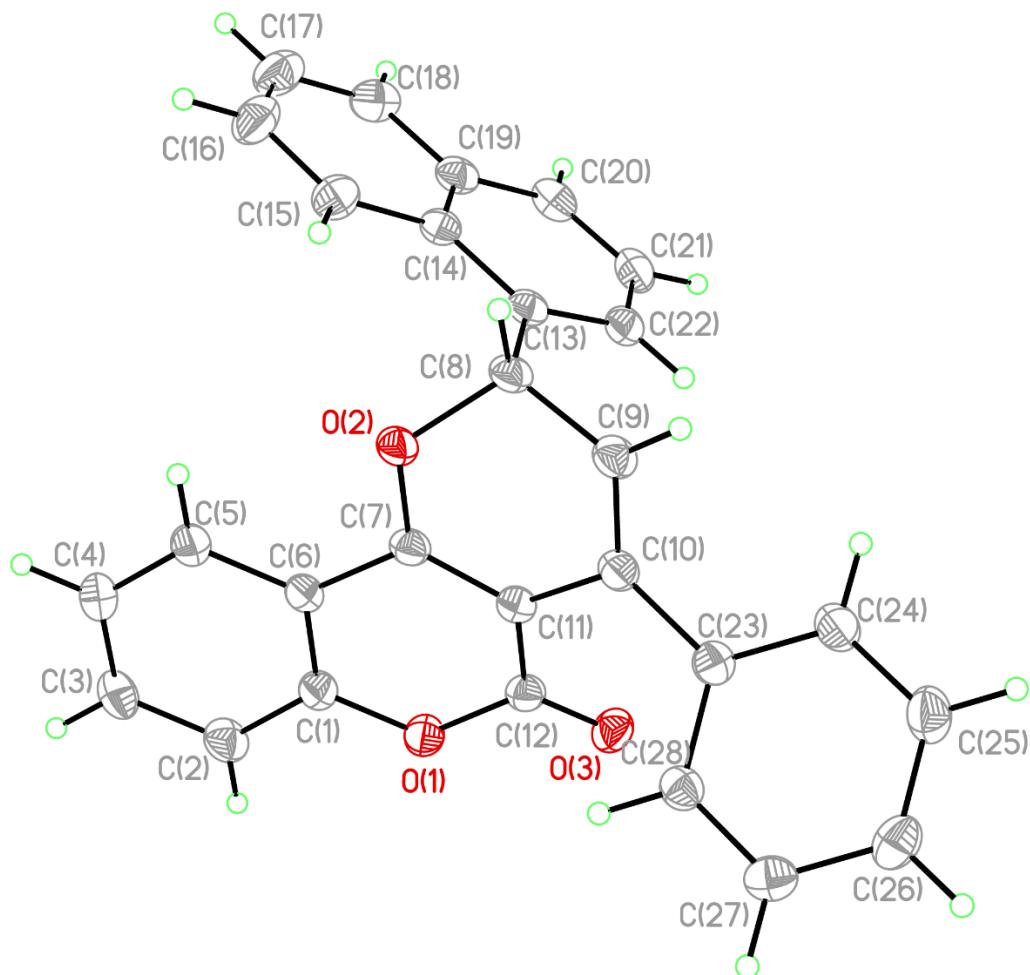
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## Instrumentation

Melting points were determined on a Mel-Temp melting point apparatus in open capillaries and are uncorrected. Infrared (IR) spectra were recorded using 1725XFT-IR spectrophotometer. High resolution mass spectra (HRMS) were obtained on a Thermo Fisher Scientific Finnigan MAT95XL spectrometer using magnetic sector analyzer.  $^1\text{H}$  NMR (400 MHz) and  $^{13}\text{C}$  NMR (100) spectra were recorded on a Bruker 400 spectrometer. Chemical shifts were reported in parts per million on the scale relative to an internal standard (tetramethylsilane, or appropriate solvent peaks) with coupling constants given in hertz.  $^1\text{H}$  NMR multiplicity data are denoted by s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet). Analytical thin-layer chromatography (TLC) was carried out on Merck silica gel 60G-254 plates (25 mm) and developed with the solvents mentioned. Visualization was accomplished by using portable UV light, ninhydrin spray, or iodine chamber. Flash chromatography was performed in columns of various diameters with Merck silica gel (230–400 mesh ASTM 9385 kieselgel 60H) by elution with the solvent systems. Solvents, unless otherwise specified, were reagent grade and distilled once prior to use. All new compounds exhibited satisfactory spectroscopic and analytical data.

X-ray crystallographic data of compound **1k** (CCDC-2376165)

Single crystal of **1k** was obtained by slow evaporation from a mixture of dichloromethane and n-hexane at 25 °C. Single-crystal X-ray data were collected at 150 K on a Bruker APEX-II CCD diffractometer using graphite-monochromated Mo KR radiation ( $\lambda = 0.71073\text{\AA}$ ). The crystal structures were solved by using SHELXS-97 and the structures were refined using SHELXL-97 2014. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were fixed at geometrically calculated positions and were refined using riding model.



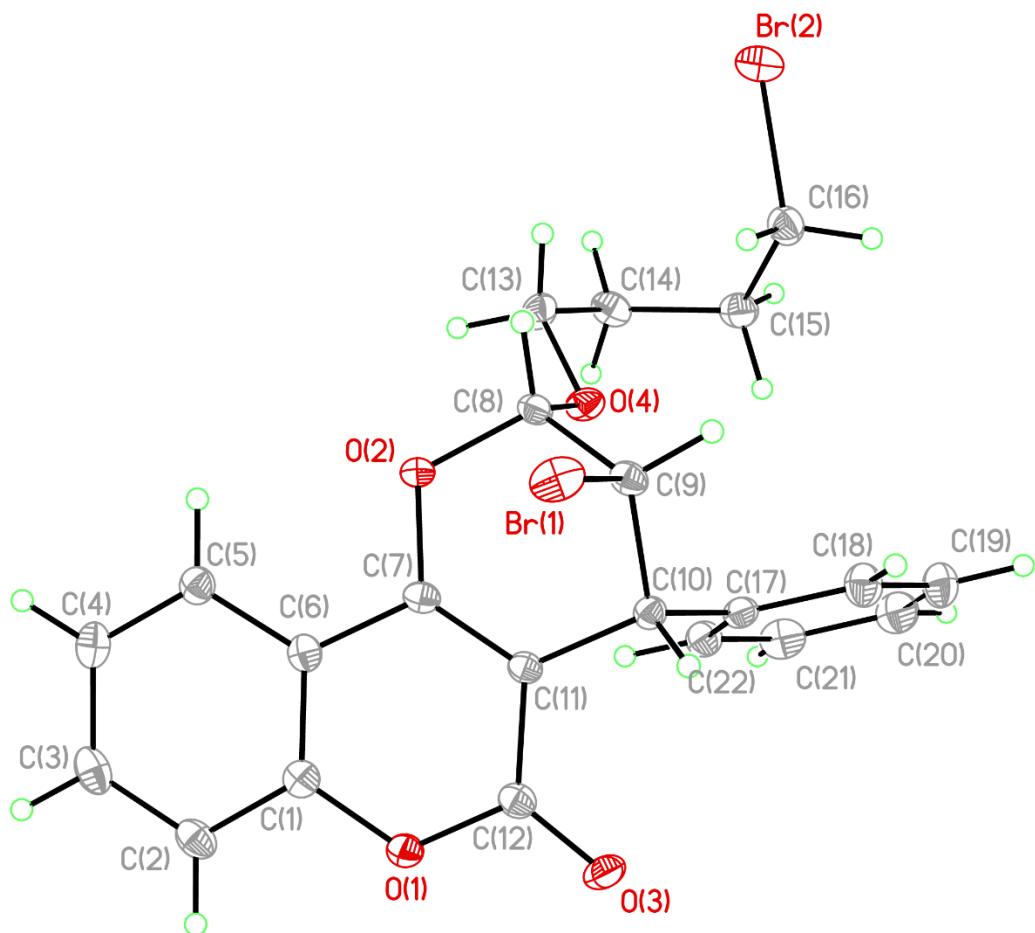
**Figure S1.** ORTEP diagram of compound **1k**. The ellipsoid contour probability levels: 50%

**Table S1.** Crystal data and structure refinement of compound **1k**

Identification code	VS214
Empirical formula	C <sub>28</sub> H <sub>18</sub> O <sub>3</sub>
Formula weight	402.42
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	a = 9.8884(5) Å      α = 100.494(2)°. b = 10.0147(5) Å      β = 105.227(2)°. c = 12.1140(7) Å      γ = 115.046(2)°.
Volume	987.89(9) Å <sup>3</sup>
Z	2
Density (calculated)	1.353 Mg/m <sup>3</sup>
Absorption coefficient	0.087 mm <sup>-1</sup>
F(000)	420
Crystal size	0.320 x 0.250 x 0.170 mm <sup>3</sup>
Theta range for data collection	3.137 to 27.895°.
Index ranges	-12<=h<=12, -13<=k<=13, -15<=l<=15
Reflections collected	19093
Independent reflections	4693 [R(int) = 0.0282]
Completeness to theta = 25.242°	99.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7456 and 0.6959
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	4693 / 0 / 280
Goodness-of-fit on F <sup>2</sup>	1.037
Final R indices [I>2sigma(I)]	R1 = 0.0492, wR2 = 0.1311
R indices (all data)	R1 = 0.0563, wR2 = 0.1377
Extinction coefficient	n/a
Largest diff. peak and hole	1.252 and -0.231 e.Å <sup>-3</sup>

X-ray crystallographic data of compound **5a** (CCDC-2361150)

Single crystal of **5a** was obtained by slow evaporation from a mixture of dichloromethane and *n*-hexane at 25 °C. Single-crystal X-ray data were collected at 150 K on a Bruker APEX-II CCD diffractometer using graphite-monochromated Mo KR radiation ( $\lambda = 0.71073\text{\AA}$ ). The crystal structures were solved by using SHELXS-97 and the structures were refined using SHELXL-97 2014. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were fixed at geometrically calculated positions and were refined using riding model.

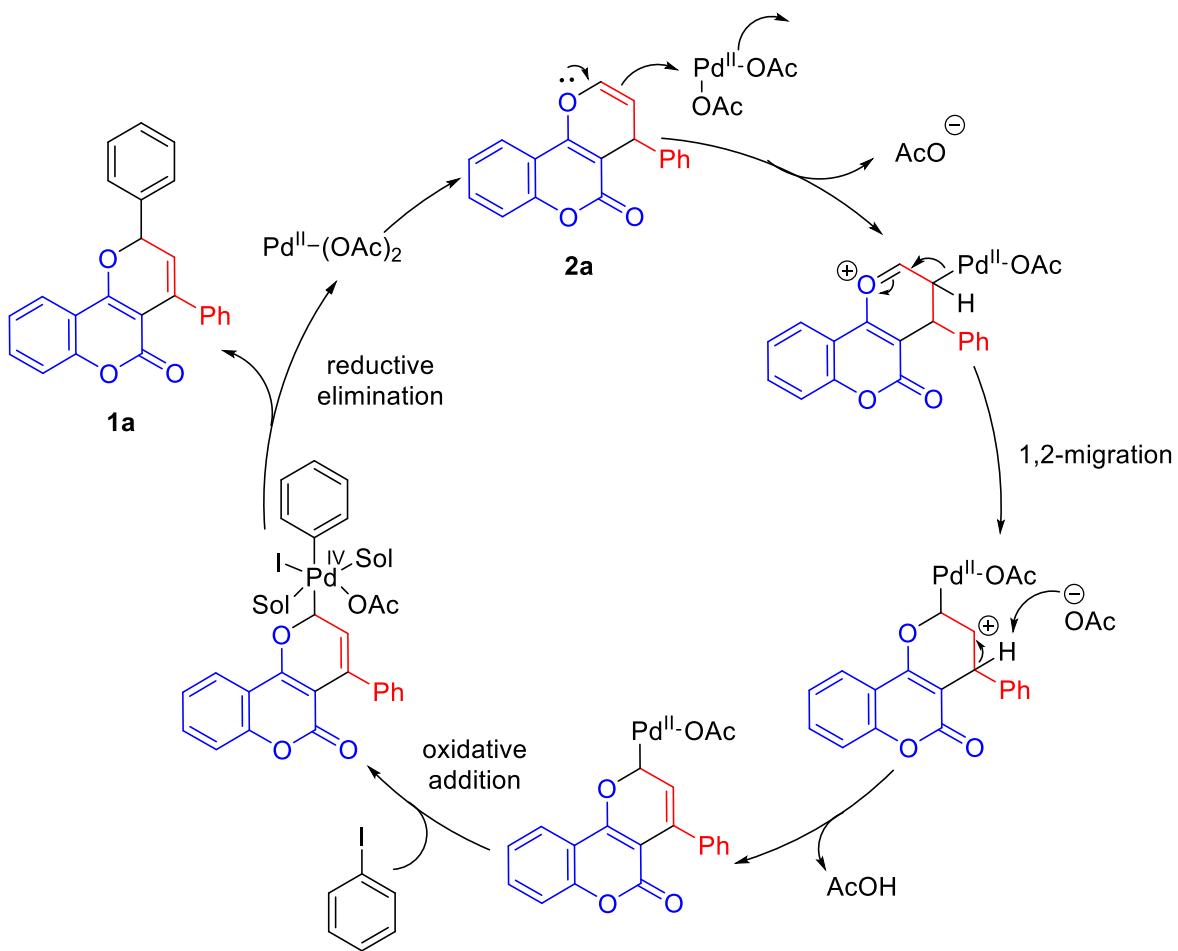


**Figure S2.** ORTEP diagram of compound **5a**. The ellipsoid contour probability levels: 50%

**Table S2.** Crystal data and structure refinement of compound **5a**.

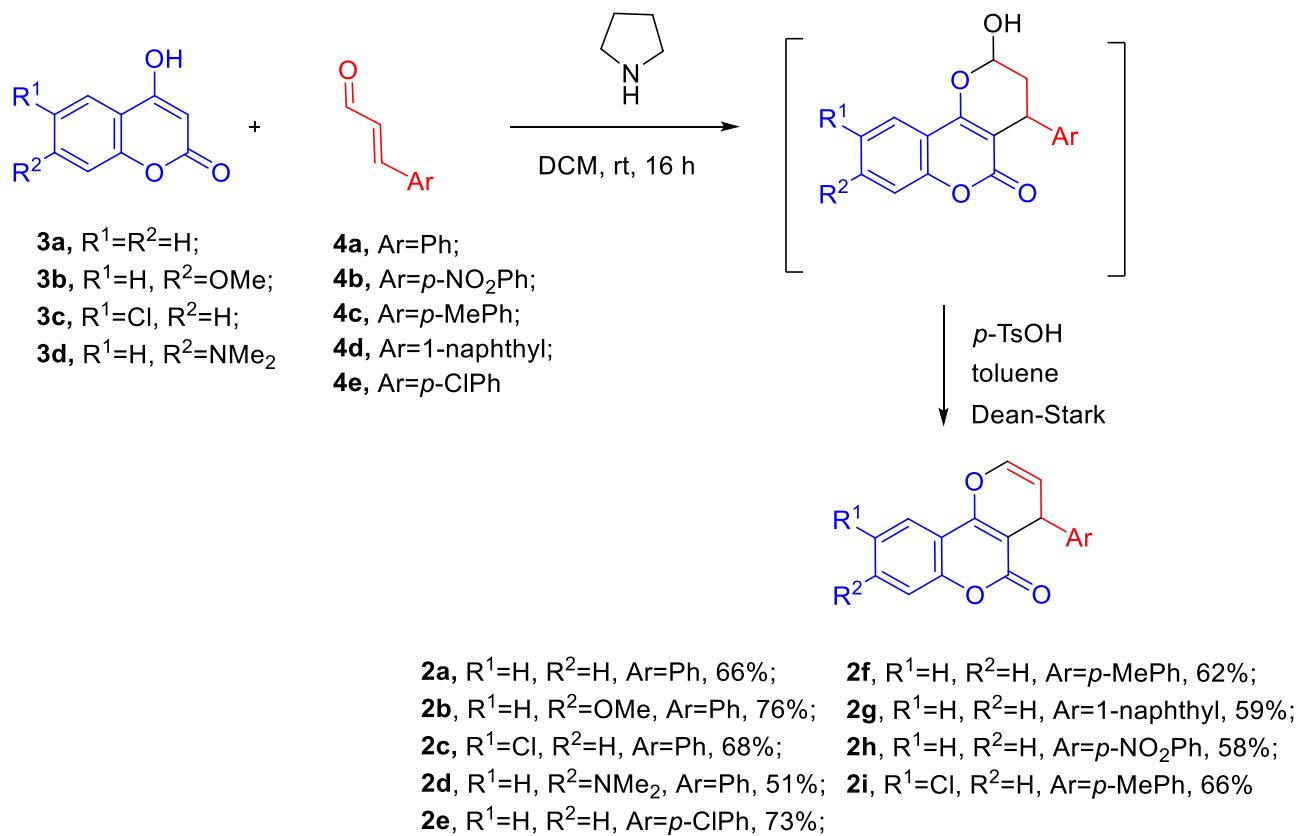
Identification code	JNS099
Empirical formula	C22 H20 Br2 O4
Formula weight	508.20
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2 <sub>1</sub> /n
Unit cell dimensions	a = 8.8245(4) Å b = 20.8267(8) Å c = 10.7464(5) Å
Volume	1938.03(15) Å <sup>3</sup>
Z	4
Density (calculated)	1.742 Mg/m <sup>3</sup>
Absorption coefficient	4.210 mm <sup>-1</sup>
F(000)	1016
Crystal size	0.350 x 0.300 x 0.150 mm <sup>3</sup>
Theta range for data collection	2.910 to 27.889°.
Index ranges	-11<=h<=11, -27<=k<=27, -14<=l<=14
Reflections collected	43618
Independent reflections	4618 [R(int) = 0.0598]
Completeness to theta = 25.242°	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7456 and 0.5528
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	4618 / 0 / 253
Goodness-of-fit on F <sup>2</sup>	1.030
Final R indices [I>2sigma(I)]	R1 = 0.0248, wR2 = 0.0650
R indices (all data)	R1 = 0.0348, wR2 = 0.0693
Extinction coefficient	n/a
Largest diff. peak and hole	0.372 and -0.694 e.Å <sup>-3</sup>

Proposed mechanism for Heck coupling reaction



Scheme S1. Proposed mechanism for formation of **1a** from **2a**.

Synthesis of 4-aryl-2*H*,5*H*-pyrano[3,2-*c*]chromen-5-ones **2a–i**.



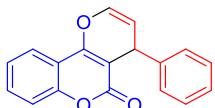
**Scheme S2.** Synthesis of 4-aryl-2*H*,5*H*-pyrano[3,2-*c*]chromen-5-ones **2a–i**.

General procedure for preparation of compounds **2a–i**.

To a solution of  $\alpha,\beta$ -unsaturated aldehyde **4** (2.2 mmol, 1.1 equiv.) and pyrrolidine (0.2 equiv.) in methylene chloride (10 mL) was added 4-hydroxycoumarin **3** (2.0 mmol, 1.0 equiv.) at 0 °C. The reaction mixture was warmed to room temperature. The mixture was then stirred at that temperature for 3–24 h. After consumption of the 4-hydroxycoumarin (monitored by TLC), the mixture was concentrated *in vacuum*. The residue was then redissolved in toluene and was added *p*-TsOH (2.4 mmol, 1.2 equiv.). The resulting mixture was then refluxed under a Dean-Stark trap for overnight. After completion of the reaction (monitored by TLC), the reaction mixture was cooled to room temperature, and the solvent was concentrated under reduced pressure. The residue was then diluted with ethyl acetate (20 mL), washed with water and brine. The organic layer was then dried over anhydrous MgSO<sub>4</sub> and concentrated under reduced pressure to provide the crude product, which was further purified by column chromatography to obtain the desired compound **2**.

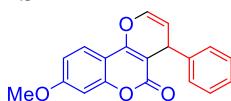
Characterization data of prepared compounds

**2a**



White solid; 364 mg; yield 66%; R<sub>f</sub> = 0.62 (20% EtOAc/hexanes); mp 146–148 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.83 (d, J = 8.0 Hz, 1H), 7.54 (td, J = 8.4, 1.6 Hz, 1H), 7.39 (d, J = 7.2 Hz, 2H), 7.35–7.28 (m, 4H), 7.22 (t, J = 7.2 Hz, 1H), 6.79 (d, J = 6.0 Hz, 1H), 5.35 (dd, J = 6.0, 4.4 Hz, 1H), 4.54 (d, J = 4.4 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.4, 155.6, 152.5, 143.5, 138.0, 131.9, 128.5 (2C), 128.3 (2C), 127.1, 124.0, 122.7, 116.6, 114.3, 108.8, 103.7, 35.3; IR ν<sub>max</sub> (KBr) 3027, 1721, 1623, 1395, 1229, 1011, 752, 699 cm<sup>-1</sup>.

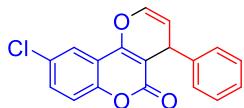
**2b**



White solid; 465 mg; yield 76%; R<sub>f</sub> = 0.45 (20% EtOAc/hexanes); mp 140–142 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.71 (d, J = 8.8 Hz, 1H), 7.38 (d, J = 7.2 Hz, 2H), 7.31 (t, J = 7.2 Hz, 2H), 7.22 (tt, J = 7.2, 1.6 Hz, 1H), 6.88 (dd, J = 8.8, 2.4 Hz, 1H), 6.78 (d, J = 2.4 Hz, 1H), 6.75 (dd, J = 6.0, 0.8 Hz, 1H), 5.33 (dd, J = 6.0, 4.4 Hz, 1H), 4.50 (d, J = 4.4 Hz, 1H), 3.86 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 162.8, 161.8, 156.0, 154.3, 143.7, 137.9, 128.4 (2C), 128.3 (2C), 127.0, 123.7, 112.3, 108.9, 107.4, 100.9, 100.4, 55.7, 35.1; IR ν<sub>max</sub> (KBr) 3026, 1719, 1618, 1399, 1233, 1157, 1031, 752,

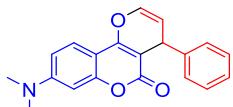
698 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>19</sub>H<sub>14</sub>O<sub>4</sub>, 306.0892; found, 306.0887.

**2c**



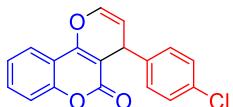
White solid; 422 mg; yield 68%; R<sub>f</sub> = 0.61 (20% EtOAc/hexanes); mp 144–146 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.80 (d, J = 2.4 Hz, 1H), 7.47 (dd, J = 8.8, 2.4 Hz, 1H), 7.38–7.35 (m, 2H), 7.34–7.30 (m, 2H), 7.25–7.21 (m, 2H), 6.79 (d, J = 6.0 Hz, 1H), 5.36 (dd, J = 6.0, 4.8 Hz, 1H), 4.53 (d, J = 4.8 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 160.8, 154.5, 150.9, 143.1, 138.0, 131.9, 129.6, 128.5 (2C), 128.4 (2C), 127.3, 122.3, 118.1, 115.4, 108.8, 104.5, 35.2; IR ν<sub>max</sub> (KBr) 3028, 1723, 1622, 1486, 1228, 1012, 838, 753, 700 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>18</sub>H<sub>11</sub>ClO<sub>3</sub>, 310.0397; found, 310.0389.

**2d**



Light pink solid; 325 mg; yield 51%; R<sub>f</sub> = 0.41 (20% EtOAc/hexanes); mp 194–196 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.60 (d, J = 8.8 Hz, 1H), 7.37 (dd, J = 7.2, 1.2 Hz, 2H), 7.30 (t, J = 7.2 Hz, 2H), 7.20 (t, J = 7.2, 1.2 Hz, 1H), 6.72 (dd, J = 6.0, 0.8 Hz, 1H), 6.64 (dd, J = 8.8, 2.4 Hz, 1H), 6.47 (d, J = 2.4 Hz, 1H), 5.30 (dd, J = 6.0, 4.4 Hz, 1H), 4.47 (d, J = 4.4 Hz, 1H), 3.04 (s, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 162.4, 156.6, 154.6, 152.9, 144.3, 137.9, 128.3 (2C), 128.2 (2C), 126.8, 123.3, 108.9, 108.8, 102.9, 98.6, 97.7, 40.1 (2C), 35.1; IR ν<sub>max</sub> (KBr) 2919, 1712, 1620, 1527, 1403, 1231, 1169, 1020, 752, 698 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>20</sub>H<sub>17</sub>NO<sub>3</sub>, 319.1208; found, 319.1202.

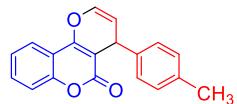
**2e**



White solid; 453 mg; yield 73%; R<sub>f</sub> = 0.61 (20% EtOAc/hexanes); mp 168–170 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.82 (dd, J = 8.4, 1.6 Hz, 1H), 7.55 (td, J = 8.4, 1.6 Hz, 1H), 7.35–7.33 (m, 1H), 7.33–7.28 (m, 4H), 7.28–7.25 (m, 1H), 6.80 (d, J = 6.0 Hz, 1H), 5.32 (t, J = 6.0, 4.4 Hz, 1H), 4.52 (d, J = 4.4 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.4, 155.7, 152.5, 142.0, 138.3, 132.9, 132.1, 129.8 (2C), 128.6 (2C), 124.1, 122.7, 116.7, 114.1, 108.4, 103.3, 34.7; IR ν<sub>max</sub> (KBr) 2923, 1723, 1621, 1492, 1392, 1229,

1044, 1011, 845, 756 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>18</sub>H<sub>11</sub>ClO<sub>3</sub>, 310.0397; found, 310.0399.

**2f**



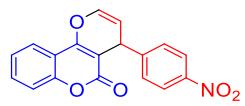
White solid; 360 mg; yield 62%; R<sub>f</sub> = 0.64 (20% EtOAc/hexanes); mp 142–144 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.82 (d, J = 8.0 Hz, 1H), 7.53 (t, J = 8.0, 1.2 Hz, 1H), 7.34–7.24 (m, 4H), 7.12 (d, J = 8.0 Hz, 2H), 6.77 (d, J = 6.0 Hz, 1H), 5.34 (dd, J = 6.0, 4.4 Hz, 1H), 4.50 (d, J = 4.4 Hz, 1H), 2.31 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.5, 155.5, 152.5, 140.7, 137.9, 136.8, 131.8, 129.2 (2C), 128.2 (2C), 124.0, 122.6, 116.6, 114.3, 109.0, 103.8, 34.8, 21.0; IR ν<sub>max</sub> (KBr) 2922, 1722, 1622, 1393, 1228, 1207, 1112, 1042, 1010, 758 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>19</sub>H<sub>14</sub>O<sub>3</sub>, 290.0943; found, 290.0945.

**2g**



White solid; 385 mg; yield 59%; R<sub>f</sub> = 0.57 (20% EtOAc/hexanes); mp 164–166 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 8.32 (d, J = 8.4 Hz, 1H), 7.89 (d, J = 8.0 Hz, 2H), 7.75 (d, J = 8.0 Hz, 1H), 7.63–7.55 (m, 2H), 7.52 (t, J = 7.2 Hz, 1H), 7.43–7.33 (m, 3H), 7.30 (d, J = 7.2 Hz, 1H), 6.69 (dd, J = 5.6, 0.8 Hz, 1H), 5.48 (dd, J = 5.6, 4.4 Hz, 1H), 5.39 (d, J = 4.4 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.4, 156.8, 152.6, 140.2, 137.6, 133.9, 132.0, 130.7, 128.9, 127.6, 126.4, 125.71 (2C), 125.67, 124.1, 122.8, 122.7, 116.8, 114.2, 108.9, 103.0, 30.7; IR ν<sub>max</sub> (KBr) 2923, 1722, 1621, 1397, 1229, 1040, 1010, 769, 757 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>22</sub>H<sub>14</sub>O<sub>3</sub>, 326.0943; found, 326.0648.

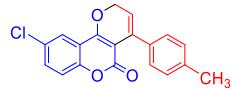
**2h**



White solid; 372 mg; yield 58%; R<sub>f</sub> = 0.38 (20% EtOAc/hexanes); mp 196–198 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 8.16 (d, J = 8.8 Hz, 2H), 7.84 (dd, J = 8.0, 1.2 Hz, 1H), 7.56 (d, J = 8.4 Hz, 2H), 7.58 (td, J = 7.2, 1.2 Hz, 1H), 7.31 (d, J = 8.8 Hz, 1H), 7.35 (t, J = 8.0, 0.4 Hz, 1H), 6.85 (dd, J = 6.0, 0.8 Hz, 1H), 5.32 (dd, J = 6.0, 4.4 Hz, 1H), 4.67 (d, J = 4.4 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.3, 156.1, 152.6, 150.5, 147.0, 138.9, 132.5, 129.4 (2C), 124.3, 123.8 (2C), 122.8, 116.8, 113.9, 107.5, 102.4, 35.3; IR ν<sub>max</sub>

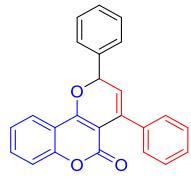
(KBr) 3077, 1720, 1621, 1518, 1395, 1347, 1231, 1013, 833, 752 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>18</sub>H<sub>11</sub>NO<sub>5</sub>, 321.0637; found, 321.0641.

**2i**



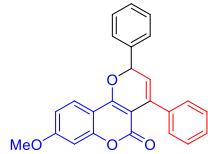
White solid; 428 mg; yield 66%; R<sub>f</sub> = 0.67 (20% EtOAc/hexanes); mp 154–156 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.79 (d, J = 2.4 Hz, 1H), 7.47 (dd, J = 8.8, 2.4 Hz, 1H), 7.25 (d, J = 8.0 Hz, 2H), 7.23 (d, J = 8.8 Hz, 1H), 7.13 (d, J = 8.0 Hz, 2H), 6.77 (d, J = 6.0 Hz, 1H), 5.34 (dd, J = 6.0, 4.4 Hz, 1H), 4.48 (d, J = 4.4 Hz, 1H), 2.31 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 160.9, 154.4, 150.8, 140.3, 137.9, 137.0, 131.8, 129.6, 129.2 (2C), 128.2 (2C), 122.3, 118.1, 115.4, 109.0, 104.7, 34.8, 21.0; IR ν<sub>max</sub> (KBr) 2921, 1723, 1622, 1421, 1385, 1227, 1204, 1010, 819 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>19</sub>H<sub>13</sub>ClO<sub>3</sub>, 324.0553; found, 324.0557.

**1a**

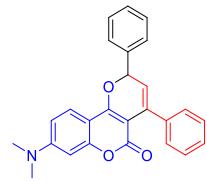


Pale yellow solid; 33 mg; yield 68%; R<sub>f</sub> = 0.63 (30% EtOAc/hexanes); mp 158–160 °C (Lit.<sup>1</sup> 158–160 °C); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.86 (dd, J = 8.0, 1.2 Hz, 1H), 7.59–7.51 (m, 3H), 7.46–7.38 (m, 3H), 7.38–7.33 (m, 5H), 7.30 (d, J = 8.4 Hz, 1H), 7.29–7.24 (m, 1H), 6.15 (d, J = 4.4 Hz, 1H), 5.77 (d, J = 4.4 Hz, 1H).

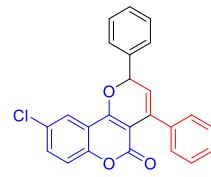
**1b**



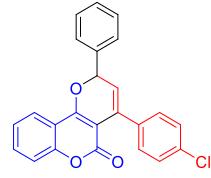
Light brown solid; 32 mg; yield 61%; R<sub>f</sub> = 0.65 (20% EtOAc/hexanes); mp 126–128 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.75 (d, J = 8.8 Hz, 1H), 7.56 (d, J = 8.0 Hz, 2H), 7.45–7.38 (m, 3H), 7.37–7.32 (m, 5H), 6.83 (dd, J = 8.8, 2.4 Hz, 1H), 6.78 (d, J = 2.4 Hz, 1H), 6.10 (d, J = 4.0 Hz, 1H), 5.71 (d, J = 4.0 Hz, 1H), 3.86 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 163.6, 161.9, 159.0, 155.5, 138.4, 138.0, 135.3, 129.1, 128.8 (2C), 127.8 (2C), 127.7, 127.4 (3C), 124.4, 119.2, 112.5, 108.3, 100.3 (2C), 78.7, 55.7; IR ν<sub>max</sub> (KBr) 2925, 1724, 1617, 1405, 1285, 1160, 1028, 755, 699 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>25</sub>H<sub>18</sub>O<sub>4</sub>, 382.1205; found, 382.1198.

**1c**

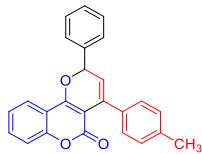
Pink solid; 41 mg; yield 76%;  $R_f = 0.39$  (20% EtOAc/hexanes); mp 128–130 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.65 (d,  $J = 8.8$  Hz, 1H), 7.56 (dd,  $J = 8.0$ , 1.6 Hz, 2H), 7.44–7.31 (m, 8H), 6.59 (dd,  $J = 8.8$ , 2.4 Hz, 1H), 6.47 (d,  $J = 2.4$  Hz, 1H), 6.04 (d,  $J = 4.0$  Hz, 1H), 5.64 (d,  $J = 4.0$  Hz, 1H), 3.06 (s, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  162.6, 159.6, 155.9, 153.5, 138.9, 138.4, 135.7, 128.9, 128.7 (2C), 127.7 (2C), 127.5, 127.42 (2C), 127.38 (2C), 124.2, 118.1, 108.7, 103.8, 98.2, 97.5, 78.4, 40.1 (2C); IR  $\nu_{max}$  (KBr) 2924, 1711, 1618, 1578, 1513, 1408, 1175, 755, 698  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{26}\text{H}_{21}\text{NO}_3$ , 395.1521; found, 395.1530.

**1d**

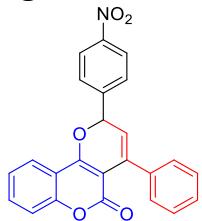
Pale yellow solid; 33 mg; yield 62%;  $R_f = 0.58$  (20% EtOAc/hexanes); mp 198–200 °C (Lit.<sup>2</sup> 203–205 °C);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.81 (d,  $J = 2.4$  Hz, 1H), 7.58–7.56 (m, 2H), 7.48 (dd,  $J = 8.8$ , 2.4 Hz, 1H), 7.46–7.41 (m, 3H), 7.39–7.35 (m, 3H), 7.35–7.31 (m, 2H), 7.25 (d,  $J = 9.2$  Hz, 1H), 6.16 (d,  $J = 4.0$  Hz, 1H), 5.79 (d,  $J = 4.0$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  160.1, 158.1, 151.9, 137.8, 137.5, 134.9, 132.5, 129.5 (3C), 129.0 (2C), 128.0, 127.9, 127.6 (2C), 127.4 (2C), 122.7, 120.7, 118.1, 116.3, 103.3, 79.0; IR  $\nu_{max}$  (KBr) 3029, 1729, 1630, 1552, 1484, 1393, 1118, 996, 755, 698  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{24}\text{H}_{15}\text{ClO}_3$ , 386.0710; found, 386.0720.

**1e**

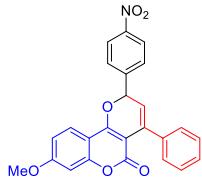
Pale yellow solid; 39 mg; yield 73%;  $R_f = 0.57$  (20% EtOAc/hexanes); mp 190–192 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.86 (dd,  $J = 8.0$ , 1.2 Hz, 1H), 7.58–7.52 (m, 3H), 7.46–7.39 (m, 3H), 7.33 (d,  $J = 8.4$  Hz, 2H), 7.31–7.24 (m, 4H), 6.14 (d,  $J = 4.4$  Hz, 1H), 5.75 (d,  $J = 4.4$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.5, 158.6, 153.5, 138.0, 136.3, 134.1, 133.7, 132.8, 129.3, 128.9 (2C), 128.8 (2C), 128.1 (2C), 127.4 (2C), 124.0, 123.3, 120.5, 116.6, 115.0, 102.3, 78.7; IR  $\nu_{max}$  (KBr) 2920, 1723, 1629, 1609, 1552, 1491, 1405, 990, 756  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{24}\text{H}_{15}\text{ClO}_3$ , 386.0710; found, 386.0712.

**1f**

White solid; 36 mg; yield 72%;  $R_f = 0.59$  (20% EtOAc/hexanes); mp 142–144 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.85 (dd,  $J = 7.6, 1.2$  Hz, 1H), 7.68–7.51 (m, 3H), 7.45–7.38 (m, 3H), 7.33–7.22 (m, 4H), 7.18 (d,  $J = 8.0$  Hz, 2H), 6.13 (d,  $J = 4.4$  Hz, 1H), 5.75 (d,  $J = 4.4$  Hz, 1H), 2.38 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.3, 158.7, 153.5, 138.2, 137.6, 135.1, 134.9, 132.5, 129.2, 128.8 (2C), 128.7 (2C), 127.5 (2C), 127.2 (2C), 123.9, 123.2, 119.7, 116.6, 115.2, 102.9, 78.7, 21.3; IR  $\nu_{max}$  (KBr) 2922, 1727, 1628, 1609, 1552, 1491, 1397, 1107, 988, 756  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{25}\text{H}_{18}\text{O}_3$ , 366.1256; found, 366.1246.

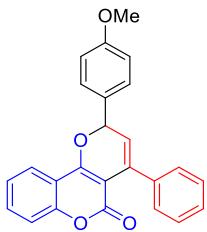
**1g**

Brown solid; 30 mg; yield 54%;  $R_f = 0.66$  (30% EtOAc/hexanes); mp 192–194 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  8.29 (d,  $J = 8.8$  Hz, 2H), 7.87 (dd,  $J = 8.0, 1.2$  Hz, 1H), 7.74 (d,  $J = 8.4$  Hz, 2H), 7.59 (td,  $J = 8.4, 1.6$  Hz, 1H), 7.40–7.35 (m, 3H), 7.35–7.29 (m, 4H), 6.24 (d,  $J = 4.4$  Hz, 1H), 5.75 (d,  $J = 4.4$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.0, 158.2, 153.6, 148.2, 145.1, 137.3, 136.1, 133.0, 128.1, 127.94 (2C), 127.89 (2C), 127.3 (2C), 124.14, 124.07 (2C), 123.0, 118.8, 116.7, 114.7, 103.0, 77.2; IR  $\nu_{max}$  (KBr) 2920, 1725, 1632, 1523, 1348, 754, 699  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{24}\text{H}_{15}\text{NO}_5$ , 397.0950; found, 397.0959.

**1h**

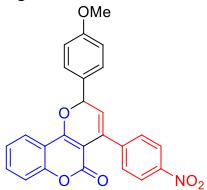
Brown solid; 31 mg; yield 53%;  $R_f = 0.33$  (20% EtOAc/hexanes); mp 174–176 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  8.28 (d,  $J = 8.4$  Hz, 2H), 7.76 (d,  $J = 8.8$  Hz, 1H), 7.73 (d,  $J = 8.4$  Hz, 2H), 7.38–7.30 (m, 5H), 6.87 (dd,  $J = 8.8, 2.4$  Hz, 1H), 6.80 (d,  $J = 2.4$  Hz, 1H), 6.19 (d,  $J = 4.4$  Hz, 1H), 5.68 (d,  $J = 4.4$  Hz, 1H), 3.88 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  163.9, 161.6, 158.7, 155.6, 148.2, 145.4, 137.4, 136.3, 128.1, 127.93 (2C), 127.88 (2C), 127.3 (2C), 124.2, 124.1 (2C), 117.7, 112.8, 107.9, 100.5, 100.4, 77.1, 55.8; IR  $\nu_{max}$  (KBr) 3365, 1727, 1615, 1529, 1402, 1161, 756  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{25}\text{H}_{17}\text{NO}_6$ , 427.1056; found, 427.1050.

**1i**



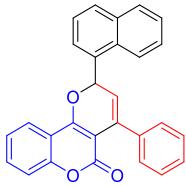
Pale yellow solid; 27 mg; yield 52%;  $R_f = 0.29$  (30% EtOAc/hexanes); mp 122–124 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.83 (dd,  $J = 8.0, 1.2$  Hz, 1H), 7.55–7.48 (m, 3H), 7.40–7.32 (m, 5H), 7.29 (d,  $J = 8.4$  Hz, 1H), 7.23 (d,  $J = 7.6$  Hz, 1H), 6.93 (d,  $J = 8.8$  Hz, 2H), 6.10 (d,  $J = 4.4$  Hz, 1H), 5.76 (d,  $J = 4.4$  Hz, 1H), 3.82 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.2, 160.4, 158.8, 153.5, 137.9, 135.1, 132.5, 129.9, 129.4 (2C), 127.9, 127.8 (2C), 127.4 (2C), 123.8, 123.3, 120.2, 116.5, 115.3, 114.2 (2C), 102.6, 78.5, 55.3; IR  $\nu_{max}$  (KBr) 2925, 1737, 1614, 1585, 1483, 1457, 1364, 1033  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{25}\text{H}_{18}\text{O}_4$ , 382.1205; found, 382.1199.

**1j**



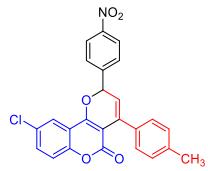
Brown solid; 43 mg; yield 73%;  $R_f = 0.33$  (30% EtOAc/hexanes); mp 156–158 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  8.22 (d,  $J = 8.8$  Hz, 2H), 7.84 (d,  $J = 7.6$  Hz, 1H), 7.56 (t,  $J = 7.6$  Hz, 1H), 7.48 (d,  $J = 8.4$  Hz, 4H), 7.33–7.24 (m, 2H), 6.95 (d,  $J = 8.4$  Hz, 2H), 6.16 (d,  $J = 4.4$  Hz, 1H), 5.82 (d,  $J = 4.0$  Hz, 1H), 3.82 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.7, 160.6, 158.8, 153.5, 147.3, 144.9, 133.4, 133.0, 129.4, 129.3 (2C), 128.4 (2C), 124.2, 123.4, 123.2 (2C), 122.0, 116.7, 115.0, 114.4 (2C), 101.6, 78.5, 55.3; IR  $\nu_{max}$  (KBr) 3441, 1717, 1609, 1515, 1345, 1252, 1175, 1033, 755  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{25}\text{H}_{17}\text{NO}_6$ , 427.1056; found, 427.1046.

**1k**



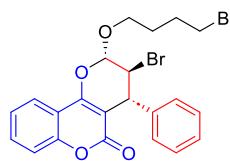
White solid; 35 mg; yield 63%;  $R_f = 0.53$  (20% EtOAc/hexanes); mp 184–186 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  8.38 (d,  $J = 8.4$  Hz, 1H), 7.92 (t,  $J = 9.2$  Hz, 2H), 7.81 (d,  $J = 7.2$  Hz, 1H), 7.75 (dd,  $J = 7.6, 1.2$  Hz, 1H), 7.64 (t,  $J = 7.2$  Hz, 1H), 7.57 (t,  $J = 7.2$  Hz, 1H), 7.53–7.47 (m, 2H), 7.43–7.35 (m, 5H), 7.28 (d,  $J = 8.4$  Hz, 1H), 7.18 (t,  $J = 7.2$  Hz, 1H), 6.90 (d,  $J = 4.4$  Hz, 1H), 5.87 (d,  $J = 4.4$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.6, 158.7, 153.5, 137.8, 135.8, 134.2, 132.5, 132.4, 131.0, 130.1, 129.1 (2C), 127.91 (2C), 127.88, 127.4, 126.8, 126.4, 126.0, 125.1, 123.8, 123.5, 123.3, 119.8, 116.5, 115.2, 102.9, 76.1; IR  $\nu_{max}$  (KBr) 3016, 1727, 1629, 1609, 1553, 1491, 1400, 1107, 753, 699  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{28}\text{H}_{18}\text{O}_3$ , 402.1256; found, 402.1250.

**11**



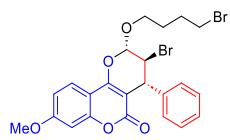
White solid; 37 mg; yield 61%;  $R_f = 0.49$  (20% EtOAc/hexanes); mp 182–184 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  8.31 (d,  $J = 8.8$  Hz, 2H), 7.80 (d,  $J = 2.4$  Hz, 1H), 7.73 (d,  $J = 8.8$  Hz, 2H), 7.52 (dd,  $J = 8.8, 2.4$  Hz, 1H), 7.27 (d,  $J = 8.8$  Hz, 1H), 7.2 (dd,  $J = 8.4, 6.0$  Hz, 4H), 6.23 (d,  $J = 4.4$  Hz, 1H), 5.74 (d,  $J = 4.4$  Hz, 1H), 2.38 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  159.8, 157.7, 151.9, 148.4, 144.8, 138.2, 135.8, 134.0, 132.9, 129.7, 128.8 (2C), 128.1 (2C), 127.2 (2C), 124.2 (2C), 122.5, 118.8, 118.2, 115.9, 103.8, 77.5, 21.3; IR  $\nu_{max}$  (KBr) 2921, 1730, 1630, 1551, 1522, 1348, 1118, 998, 820, 755  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{25}\text{H}_{16}\text{ClNO}_5$ , 445.0717; found, 445.0715.

**5a**



White solid; 44 mg; yield 63%;  $R_f = 0.58$  (20% EtOAc/hexanes); mp 118–120 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.91 (dd,  $J = 7.6, 1.2$  Hz, 1H), 7.61 (td,  $J = 8.8, 1.2$  Hz, 1H), 7.40 (d,  $J = 8.8$  Hz, 1H), 7.36 (td,  $J = 8.8, 0.8$  Hz, 1H), 7.29–7.18 (m, 5H), 5.52 (d,  $J = 2.4$  Hz, 1H), 4.58 (t,  $J = 2.4$  Hz, 1H), 4.52 (d,  $J = 2.4$  Hz, 1H), 3.90–3.85 (m, 1H), 3.56–3.50 (m, 1H), 3.17 (t,  $J = 6.0$  Hz, 2H), 1.60–1.40 (m, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.4, 157.2, 152.9, 139.6, 132.2, 128.2, 127.8 (2C), 127.1 (2C), 124.1, 122.7, 116.8, 115.0, 101.0 (2C), 69.2, 47.3, 44.0, 33.1, 28.8, 27.8; IR  $\nu_{max}$  (KBr) 2930, 1719, 1634, 1611, 1493, 1400, 1126, 1079, 953, 756  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{22}\text{H}_{20}\text{Br}_2\text{O}_4$ , 505.9728; found, 505.9722.

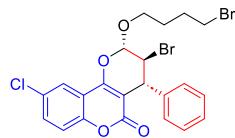
**5b**



White solid; 48 mg; yield 65%;  $R_f = 0.41$  (20% EtOAc/hexanes); mp 130–132 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.80 (d,  $J = 8.8$  Hz, 1H), 7.30–7.17 (m, 5H), 6.92 (dd,  $J = 8.8, 2.4$  Hz, 1H), 6.87 (d,  $J = 2.4$  Hz, 1H), 5.48 (d,  $J = 2.4$  Hz, 1H), 4.56 (t,  $J = 2.4$  Hz, 1H), 4.48 (d,  $J = 1.6$  Hz, 1H), 3.89 (s, 3H), 3.87–3.80 (m, 1H), 3.56–3.45 (m, 1H), 3.17 (t,  $J = 5.6$  Hz, 2H), 1.60–1.40 (m, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  163.1, 161.8, 157.6, 154.6, 139.8, 128.1 (2C), 127.7 (2C), 126.9, 123.7, 112.3, 108.1, 100.8, 100.5, 97.8, 69.1, 55.7, 47.6, 43.9, 33.1, 28.7, 27.8; IR  $\nu_{max}$  (KBr) 2925, 1721, 1635,

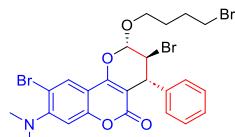
1574, 1484, 1097, 952, 756  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for C<sub>23</sub>H<sub>22</sub>Br<sub>2</sub>O<sub>5</sub>, 535.9834; found, 535.9843.

**5c**



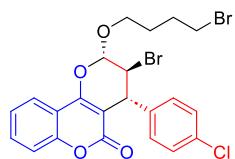
White solid; 58 mg; yield 78%; R<sub>f</sub> = 0.58 (20% EtOAc/hexanes); mp 154–156 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.88 (d, J = 2.4 Hz, 1H), 7.55 (dd, J = 8.8, 2.4 Hz, 1H), 7.34 (d, J = 8.8 Hz, 1H), 7.30–7.18 (m, 5H), 5.53 (d, J = 2.0 Hz, 1H), 4.59 (t, J = 2.4 Hz, 1H), 4.51 (d, J = 2.0 Hz, 1H), 3.91–3.85 (m, 1H), 3.57–3.49 (m, 1H), 3.21–3.10 (m, 2H), 1.55–1.43 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 160.9, 156.2, 151.3, 139.2, 132.2, 129.7, 128.3 (2C), 127.7 (2C), 127.2, 122.3, 118.3, 116.1, 101.6, 101.1, 69.4, 46.9, 43.9, 33.1, 28.8, 27.8; IR ν<sub>max</sub> (KBr) 3434, 1720, 1636, 1574, 1483, 1097, 952, 755, 719  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for C<sub>22</sub>H<sub>19</sub>Br<sub>2</sub>ClO<sub>4</sub>, 539.9339; found, 539.9332.

**5d**



White solid; 51 mg; yield 58%; R<sub>f</sub> = 0.60 (20% EtOAc/hexanes); mp 118–120 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 8.03 (s, 1H), 7.28–7.17 (m, 5H), 6.98 (s, 1H), 5.49 (d, J = 2.0 Hz, 1H), 4.57 (t, J = 2.4 Hz, 1H), 4.48 (d, J = 1.6 Hz, 1H), 3.90–3.84 (m, 1H), 3.55–3.48 (m, 1H), 3.19–3.14 (m, 2H), 2.92 (s, 6H), 1.54–1.45 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.6, 156.6, 155.5, 153.2, 139.7, 128.3 (2C), 127.8 (2C), 127.7, 127.1, 112.7, 110.0, 107.7, 101.0, 98.9, 69.3, 47.3, 43.9, 43.8 (2C), 33.3, 28.9, 27.9; IR ν<sub>max</sub> (KBr) 2923, 1718, 1626, 1608, 1396, 1135, 1081, 954, 754  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for C<sub>24</sub>H<sub>24</sub>Br<sub>3</sub>NO<sub>4</sub>, 636.9255; found, 636.9255.

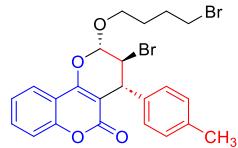
**5e**



White solid; 57 mg; yield 76%; R<sub>f</sub> = 0.56 (20% EtOAc/hexanes); mp 112–114 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.92 (d, J = 8.8 Hz, 1H), 7.62 (td, J = 8.4, 1.2 Hz, 1H), 7.40 (d, J = 8.4 Hz, 1H), 7.37 (t, J = 7.6 Hz, 1H), 7.25 (d, J = 8.8 Hz, 2H), 7.16 (d, J = 8.4 Hz, 2H), 5.52 (d, J = 2.4 Hz, 1H), 4.51 (t, J = 2.4 Hz, 1H), 4.48 (s, 1H), 3.93–3.83 (m, 1H), 3.60–3.51 (m, 1H), 3.25–3.17 (m, 2H), 1.58–1.46 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.4, 157.4, 152.9, 138.2, 132.8, 132.4, 129.2 (2C), 128.4 (2C), 124.2,

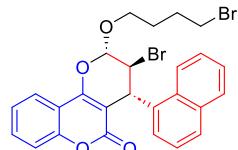
122.7, 116.9, 114.8, 100.9, 100.3, 69.4, 46.8, 43.5, 33.0, 28.9, 27.9; IR  $\nu_{max}$  (KBr) 2922, 1717, 1634, 1491, 1412, 1126, 1091, 1078, 955, 759 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>22</sub>H<sub>19</sub>Br<sub>2</sub>ClO<sub>4</sub>, 539.9339; found, 539.9334.

**5f**



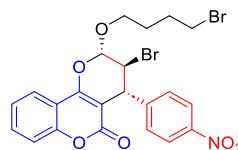
White solid; 56 mg; yield 78%; R<sub>f</sub> = 0.60 (20% EtOAc/hexanes); mp 136–138 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.91 (d, J = 6.8 Hz, 1H), 7.60 (td, J = 8.4, 1.2 Hz, 1H), 7.39 (d, J = 8.8 Hz, 1H), 7.36 (t, J = 8.8 Hz, 1H), 7.08 (q, J = 8.0 Hz, 4H), 5.51 (d, J = 2.4 Hz, 1H), 4.55 (d, J = 2.4 Hz, 1H), 4.48 (s, 1H), 3.92–3.84 (m, 1H), 3.59–3.49 (m, 1H), 3.22–3.12 (m, 2H), 2.30 (s, 3H), 3.92–3.84 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.4, 157.1, 152.9, 136.7, 136.6, 132.2, 129.0 (2C), 127.7 (2C), 124.1, 122.7, 116.8, 115.0, 100.99, 101.0, 69.3, 47.6, 43.8, 33.1, 28.9, 27.9, 21.1; IR  $\nu_{max}$  (KBr) 3429, 1717, 1639, 1611, 1398, 1327, 1126, 1078, 955, 759 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>23</sub>H<sub>22</sub>Br<sub>2</sub>O<sub>4</sub>, 519.9885; found, 519.9878.

**5g**



White solid; 47 mg; yield 61%; R<sub>f</sub> = 0.54 (20% EtOAc/hexanes); mp 158–160 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 8.14 (d, J = 8.8 Hz, 1H), 7.98 (dd, J = 8.0, 1.2 Hz, 1H), 7.92 (d, J = 8.0 Hz, 1H), 7.76 (d, J = 8.0 Hz, 1H), 7.68–7.61 (m, 2H), 7.54 (t, J = 7.6 Hz, 1H), 7.43 (d, J = 8.8 Hz, 1H), 7.40 (t, J = 8.0 Hz, 1H), 7.29 (t, J = 8.0 Hz, 1H), 7.19 (d, J = 7.2 Hz, 1H), 5.56 (s, 1H), 5.20 (s, 1H), 4.73 (s, 1H), 3.86–3.78 (m, 1H), 3.48–3.38 (m, 1H), 3.10 (t, J = 6.0 Hz, 2H), 1.48–1.34 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.5, 157.5, 153.0, 134.6, 134.2, 132.3, 130.9, 129.5, 128.1, 126.8, 125.5, 124.9 (2C), 124.2, 122.7, 122.4, 116.9, 115.1, 100.7, 100.4, 69.2, 45.1, 40.5, 33.0, 28.8, 27.8; IR  $\nu_{max}$  (KBr) 3419, 1717, 1635, 1611, 1399, 1126, 1077, 955, 754 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>26</sub>H<sub>22</sub>Br<sub>2</sub>O<sub>4</sub>, 555.9885; found, 555.9892.

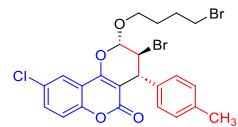
**5h**



White solid; 58 mg; yield 77%; R<sub>f</sub> = 0.36 (20% EtOAc/hexanes); mp 158–160 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 8.15 (d, J = 8.8 Hz, 2H), 7.93 (dd, J = 8.0, 1.2 Hz, 1H), 7.65 (td, J = 8.4, 1.2 Hz, 1H), 7.37–7.44 (m, 4H), 5.55

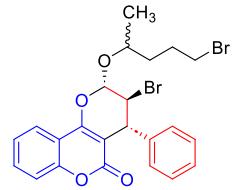
(d,  $J = 2.4$  Hz, 1H), 4.57 (d,  $J = 2.4$  Hz, 1H), 4.53 (t,  $J = 2.4$  Hz, 1H), 3.92–3.84 (m, 1H), 3.62–3.54 (m, 1H), 3.18 (t,  $J = 6.0$  Hz, 2H), 1.56–1.43 (m, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.3, 157.8, 152.9, 147.2, 146.9, 132.7, 128.8 (2C), 124.4, 123.5 (2C), 122.8, 116.9, 114.6, 100.6, 99.4, 69.6, 45.7, 43.8, 32.7, 28.8, 27.8; IR  $\nu_{max}$  (KBr) 2922, 1713, 1634, 1609, 1519, 1399, 1347, 1126, 1078, 953, 754  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{22}\text{H}_{19}\text{Br}_2\text{NO}_6$ , 550.9579; found, 550.9573.

### 5i

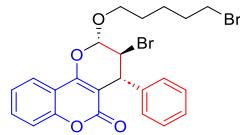


White solid; 59 mg; yield 77%;  $R_f = 0.62$  (20% EtOAc/hexanes); mp 122–124 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.88 (d,  $J = 2.4$  Hz, 1H), 7.54 (dd,  $J = 8.8, 2.4$  Hz, 1H), 7.33 (d,  $J = 9.2$  Hz, 1H), 7.08 (s, 4H), 5.52 (d,  $J = 2.4$  Hz, 1H), 4.55 (t,  $J = 2.4$  Hz, 1H), 4.47 (d,  $J = 2.4$  Hz, 1H), 3.94–3.85 (m, 1H), 3.50–3.58 (m, 1H), 3.11–3.22 (m, 2H), 2.30 (s, 3H), 1.60–1.48 (m, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  160.9, 156.1, 151.3, 136.8, 136.3, 132.2, 129.7, 129.0 (2C), 127.7 (2C), 122.3, 118.4, 116.2, 101.9, 101.2, 69.5, 47.2, 43.8, 33.0, 29.0, 28.0, 21.1; IR  $\nu_{max}$  (KBr) 2922, 1721, 1635, 1486, 1422, 1097, 1073, 954, 914, 756  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{23}\text{H}_{21}\text{Br}_2\text{ClO}_4$ , 553.9495; found, 553.9490.

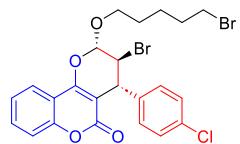
### 5j



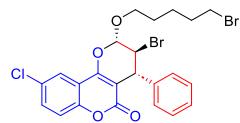
White solid; 51 mg; yield 71% (two inseparable isomers);  $R_f = 0.58$  (20% EtOAc/hexanes); mp 122–124 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz) (peaks overlapped for both isomers)  $\delta$  7.92 (d,  $J = 8.0$  Hz, 1H), 7.60 (d,  $J = 8.0$  Hz, 1H), 7.39 (d,  $J = 8.4$  Hz, 1H), 7.36 (t,  $J = 7.6$  Hz, 1H), 7.30–7.19 (m, 5H), 5.52 (d,  $J = 2.4$  Hz, 1H), 4.59 (t,  $J = 2.4$  Hz, 1H), 4.52 (d,  $J = 2.4$  Hz, 1H), 3.93–3.81 (m, 2H), 3.59–3.48 (m, 1H), 1.65–1.31 (m, 7H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) isomer one  $\delta$  161.4, 157.2, 152.9, 139.7, 139.6, 132.2, 128.3, 128.2, 127.8, 127.1, 127.0, 124.1, 122.7, 116.8, 115.0, 100.9, 69.5, 51.2, 47.2, 44.0, 37.1, 27.8, 26.4; isomer two  $\delta$  161.4, 157.2, 152.9, 139.7, 139.6, 132.2, 128.3, 128.2, 127.8, 127.1, 127.0, 124.1, 122.7, 116.8, 115.0, 101.0, 100.7, 69.6, 50.9, 47.3, 44.1, 27.5, 26.3; IR  $\nu_{max}$  (KBr) 2925, 1719, 1634, 1611, 1399, 1126, 1083, 953, 756  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{23}\text{H}_{22}\text{Br}_2\text{O}_4$ , 519.9885; found, 519.9886.

**5k**

White solid; 52 mg; yield 72%;  $R_f = 0.60$  (20% EtOAc/hexanes); mp 122–124 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.92 (dd,  $J = 8.0, 1.2$  Hz, 1H), 7.61 (td,  $J = 8.4, 1.2$  Hz, 1H), 7.39 (d,  $J = 8.4$  Hz, 1H), 7.37 (t,  $J = 8.0$  Hz, 1H), 7.29–7.17 (m, 5H), 5.52 (d,  $J = 2.8$  Hz, 1H), 4.58 (t,  $J = 2.8$  Hz, 1H), 4.52 (d,  $J = 2.4$  Hz, 1H), 3.90–3.80 (m, 1H), 3.55–3.45 (m, 1H), 3.23 (t,  $J = 6.8$  Hz, 2H), 1.70–1.00 (m, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.5, 157.3, 152.9, 139.6, 132.2, 128.2 (2C), 127.9 (2C), 127.0, 124.1, 122.7, 116.8, 115.0, 101.0, 100.7, 69.9, 47.4, 44.1, 33.4, 32.1, 28.3, 24.4; IR  $\nu_{max}$  (KBr) 3445, 1718, 1634, 1611, 1492, 1400, 1082, 758, 697  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{23}\text{H}_{22}\text{Br}_2\text{O}_4$ , 519.9885; found, 519.9877.

**5l**

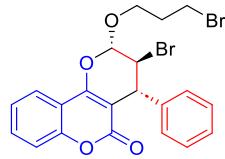
White solid; 53 mg; yield 69%;  $R_f = 0.58$  (20% EtOAc/hexanes); mp 116–118 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.92 (d,  $J = 8.0$  Hz, 1H), 7.61 (td,  $J = 8.4, 1.2$  Hz, 1H), 7.40 (d,  $J = 8.4$  Hz, 1H), 7.37 (t,  $J = 7.6$  Hz, 1H), 7.24 (d,  $J = 8.4$  Hz, 2H), 7.17 (d,  $J = 8.4$  Hz, 2H), 5.52 (d,  $J = 2.4$  Hz, 1H), 4.51 (t,  $J = 2.4$  Hz, 1H), 4.47 (s, 1H), 3.90–3.79 (m, 1H), 3.59–3.47 (m, 1H), 3.34–3.19 (m, 2H), 1.68 (quintet,  $J = 7.2$  Hz, 2H), 1.43–1.33 (m, 2H), 1.26–1.07 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.4, 157.4, 153.0, 138.3, 132.8, 132.4, 129.4 (2C), 128.3 (2C), 124.2, 122.8, 116.9, 114.9, 100.9, 100.3, 70.2, 46.8, 43.5, 33.4, 32.2, 28.5, 24.5; IR  $\nu_{max}$  (KBr) 3448, 1714, 1634, 1611, 1491, 1327, 1083, 760  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{23}\text{H}_{21}\text{Br}_2\text{ClO}_4$ , 553.9495; found, 553.9492.

**5m**

White solid; 56 mg; yield 73%;  $R_f = 0.58$  (20% EtOAc/hexanes); mp 120–122 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.88 (d,  $J = 2.8$  Hz, 1H), 7.55 (dd,  $J = 8.8, 2.4$  Hz, 1H), 7.34 (d,  $J = 8.8$  Hz, 1H), 7.29–7.18 (m, 5H), 5.53 (d,  $J = 2.0$  Hz, 1H), 4.58 (t,  $J = 2.4$  Hz, 1H), 4.51 (s, 1H), 3.89–3.81 (m, 1H), 3.55–3.47 (m, 1H), 3.29–3.19 (m, 2H), 1.70–1.61 (m, 2H), 1.40–1.29 (m, 2H), 1.23–1.03 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  160.9, 156.2, 151.3, 139.3, 132.2, 129.7, 128.2 (2C), 127.9 (2C), 127.1, 122.3, 118.3,

116.2, 101.7, 101.2, 70.1, 47.0, 44.1, 33.4, 32.2, 28.4, 24.4; IR  $\nu_{max}$  (KBr) 2938, 1721, 1636, 1574, 1483, 1422, 1097, 1071, 952, 755 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>23</sub>H<sub>21</sub>Br<sub>2</sub>ClO<sub>4</sub>, 553.9495; found, 553.9498.

**5n**



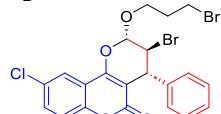
White solid; 26 mg; yield 38%; R<sub>f</sub> = 0.56 (20% EtOAc/hexanes); mp 204–206 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.94 (dd, J = 8.0, 1.2 Hz, 1H), 7.61 (td, J = 8.4, 1.2 Hz, 1H), 7.40 (d, J = 8.8 Hz, 1H), 7.36 (t, J = 7.2, 0.8 Hz, 1H), 7.30–7.20 (m, 5H), 5.55 (d, J = 2.4 Hz, 1H), 4.60 (t, J = 2.4 Hz, 1H), 4.53 (d, J = 2.4 Hz, 1H), 4.02–3.96 (m, 1H), 3.68–3.62 (m, 1H), 3.05–2.98 (m, 1H), 2.95–2.88 (m, 1H), 1.82 (quintet, J = 6.0 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.5, 157.3, 153.0, 139.7, 132.3, 128.3 (2C), 127.8 (2C), 127.2, 124.2, 122.8, 116.9, 115.0, 101.0, 100.7, 67.3, 47.2, 43.9, 31.9, 29.7; IR  $\nu_{max}$  (KBr) 3445, 1718, 1634, 1399, 1089, 1041, 758, 699 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>21</sub>H<sub>18</sub>Br<sub>2</sub>O<sub>4</sub>, 491.9572; found, 491.9581.

**5o**



White viscous liquid; 28 mg; yield 37%; R<sub>f</sub> = 0.53 (20% EtOAc/hexanes); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.60 (t, J = 8.4, 1.2 Hz, 1H), 7.39 (d, J = 7.6 Hz, 1H), 7.36 (t, J = 7.6 Hz, 2H), 7.29–7.18 (m, 5H), 5.53 (d, J = 2.4 Hz, 1H), 4.58 (t, J = 2.4 Hz, 1H), 4.52 (d, J = 2.4 Hz, 1H), 3.95–3.89 (m, 1H), 3.64–3.56 (m, 1H), 3.40 (t, J = 6.4 Hz, 2H), 3.35 (t, J = 6.4 Hz, 2H), 3.18–3.01 (m, 2H), 1.97 (quintet, J = 6.0 Hz, 2H), 1.64–1.51 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.5, 157.3, 152.9, 139.7, 132.2, 128.2 (2C), 127.9 (2C), 127.0, 124.1, 122.8, 116.8, 115.0, 101.0, 100.8, 67.9, 67.0, 66.6, 47.4, 44.1, 32.5, 30.5, 29.4; IR  $\nu_{max}$  (KBr) 2923, 1719, 1639, 1611, 1493, 1399, 1127, 1090, 955, 759 cm<sup>-1</sup>; HRMS (EI) m/z [M<sup>+</sup>] calcd for C<sub>24</sub>H<sub>24</sub>Br<sub>2</sub>O<sub>5</sub>, 549.9990; found, 549.9995.

**5p**



White solid; 32 mg; yield 44%; R<sub>f</sub> = 0.57 (20% EtOAc/hexanes); mp 208–210 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.91 (d, J = 2.4 Hz, 1H), 7.55 (dd, J = 8.8, 2.4 Hz, 1H), 7.34 (d, J = 9.2 Hz, 1H), 7.30–7.17 (m, 5H), 5.55 (d, J = 1.6 Hz, 1H), 4.60 (t, J = 2.0 Hz, 1H), 4.52 (s, 1H), 4.02–4.00 (m, 1H),

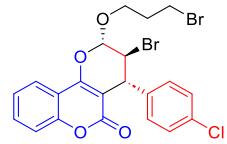
3.68–3.61 (m, 1H), 3.02–2.95 (m, 1H), 2.93–2.86 (m, 1H), 1.85–1.78 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  160.9, 156.2, 151.3, 139.2, 132.3, 129.7, 128.3 (2C), 127.7 (2C), 127.2, 122.4, 118.3, 116.3, 101.5, 101.0, 67.4, 46.7, 43.8, 31.8, 29.6; IR  $\nu_{max}$  (KBr) 2920, 1720, 1636, 1483, 1096, 1071, 954, 756  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{21}\text{H}_{17}\text{Br}_2\text{ClO}_4$ , 525.9182; found, 525.9190.

**5q**



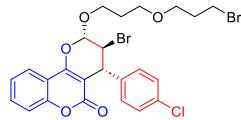
White viscous liquid; 27 mg; yield 33%;  $R_f = 0.54$  (20% EtOAc/hexanes);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.89 (d,  $J = 2.4$  Hz, 1H), 7.55 (dd,  $J = 8.8$ , 2.4 Hz, 1H), 7.33 (d,  $J = 8.8$  Hz, 1H), 7.29–7.18 (m, 5H), 5.54 (d,  $J = 2.4$  Hz, 1H), 4.58 (t,  $J = 2.4$  Hz, 1H), 4.51 (d,  $J = 1.6$  Hz, 1H), 3.98–3.88 (m, 1H), 3.65–3.56 (m, 1H), 3.42 (t,  $J = 6.4$  Hz, 2H), 3.36 (t,  $J = 6.4$  Hz, 2H), 3.17–3.09 (m, 1H), 3.08–3.00 (m, 1H), 1.99 (quintet,  $J = 6.0$  Hz, 2H), 1.62–1.56 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  160.9, 156.3, 151.3, 139.3, 132.2, 129.7, 128.3 (2C), 127.9 (2C), 127.2, 122.4, 118.3, 116.2, 101.6, 101.2, 67.9, 67.2, 66.5, 47.0, 44.1, 32.5, 30.6, 29.4; IR  $\nu_{max}$  (KBr) 2921, 1723, 1635, 1485, 1422, 1305, 1117, 1011, 953, 756  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{24}\text{H}_{23}\text{Br}_2\text{ClO}_5$ , 583.9601; found, 583.9594.

**5r**



White solid; 26 mg; yield 36%;  $R_f = 0.57$  (20% EtOAc/hexanes); mp 168–170 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.94 (d,  $J = 8.0$  Hz, 1H), 7.62 (t,  $J = 7.6$  Hz, 1H), 7.40 (d,  $J = 8.4$  Hz, 1H), 7.37 (t,  $J = 7.6$  Hz, 1H), 7.25 (d,  $J = 8.4$  Hz, 2H), 7.17 (d,  $J = 8.4$  Hz, 2H), 5.55 (d,  $J = 2.4$  Hz, 1H), 4.52 (d,  $J = 2.4$  Hz, 1H), 4.48 (s, 1H), 4.06–3.94 (m, 1H), 3.75–3.64 (m, 1H), 3.14–3.05 (m, 1H), 3.04–2.95 (m, 1H), 1.86 (quintet,  $J = 6.0$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.4, 157.4, 152.9, 138.2, 132.9, 132.5, 129.2 (2C), 128.4 (2C), 124.3, 122.8, 116.9, 114.8, 100.9, 100.2, 67.5, 46.6, 43.4, 31.8, 29.5; IR  $\nu_{max}$  (KBr) 2921, 1714, 1636, 1610, 1491, 1412, 1126, 1091, 1075, 921, 759  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{21}\text{H}_{17}\text{Br}_2\text{ClO}_4$ , 525.9182; found, 525.9193.

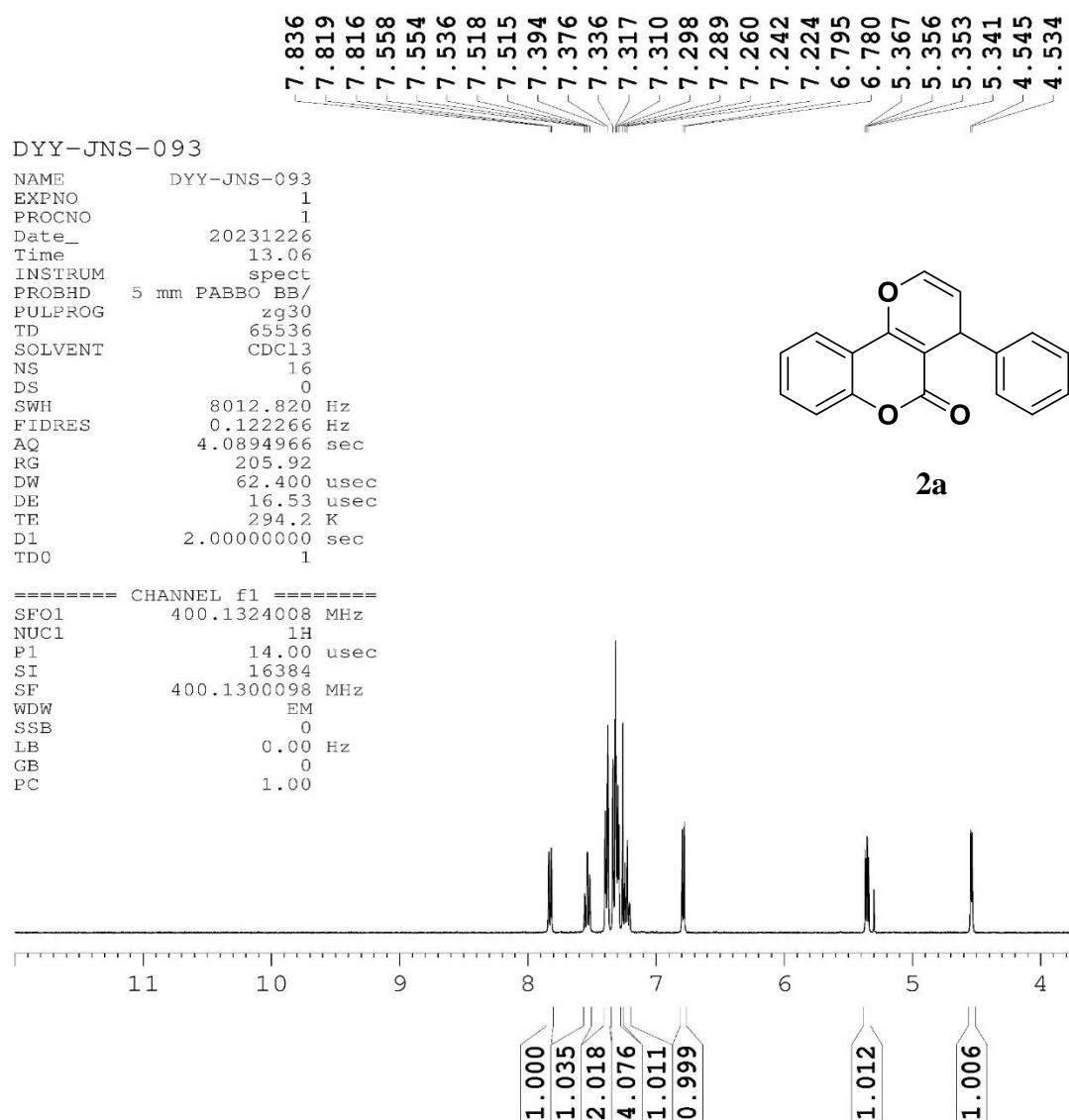
**5s**



White viscous liquid; 27 mg; yield 34%;  $R_f = 0.54$  (20% EtOAc/hexanes);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.93 (d,  $J = 7.6$  Hz, 1H), 7.61 (td,  $J = 8.0, 0.8$  Hz, 1H), 7.40 (d,  $J = 7.6$  Hz, 1H), 7.37 (t,  $J = 7.2$  Hz, 1H), 7.26–7.21 (m, 2H), 7.20–7.15 (m, 2H), 5.54 (d,  $J = 2.4$  Hz, 1H), 4.51 (t,  $J = 2.4$  Hz, 1H), 4.47 (s, 1H), 3.97–3.86 (m, 1H), 3.68–3.56 (m, 1H), 3.44–3.34 (m, 4H), 3.20–3.12 (m, 1H), 3.12–3.03 (m, 1H), 1.99 (quintet,  $J = 6.4$  Hz, 2H), 1.67–1.53 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.4, 157.5, 153.0, 138.3, 132.8, 132.4, 129.4 (2C), 128.4 (2C), 124.2, 122.8, 116.9, 114.9, 100.9, 100.3, 68.0, 67.2, 66.6, 46.8, 43.6, 32.6, 30.5, 29.5; IR  $\nu_{max}$  (KBr) 2870, 1719, 1634, 1491, 1122, 1074, 955, 760  $\text{cm}^{-1}$ ; HRMS (EI) m/z [M $^+$ ] calcd for  $\text{C}_{24}\text{H}_{23}\text{Br}_2\text{ClO}_5$ , 583.9601; found, 583.9597.

## References

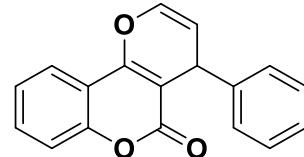
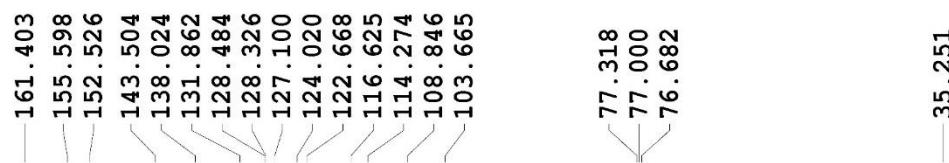
1. Y. Chen, Y. Wang, R. Zhong and J. Li, *J. Org. Chem.* 2020, **85**, 10638–10647.
2. D. Cheng, L. Wu, H. Lv, X. Xu and J. Yan, *J. Org. Chem.*, 2017, **82**, 1610–1617.



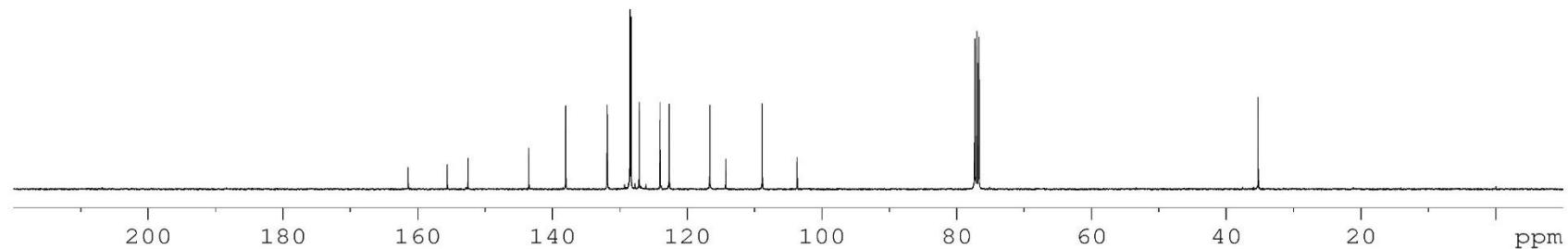
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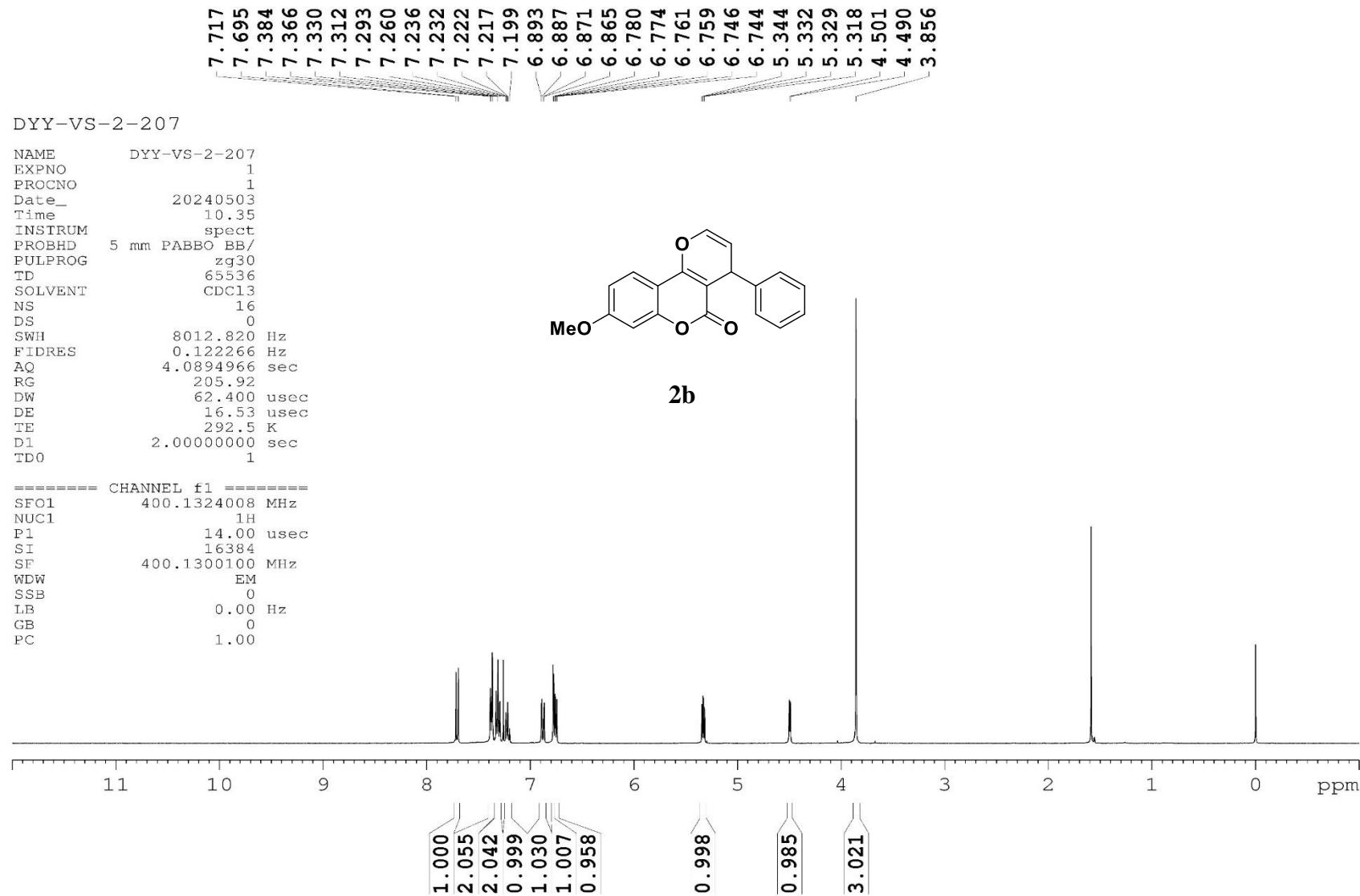
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DS 0  
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FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 297.7 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TD0 1

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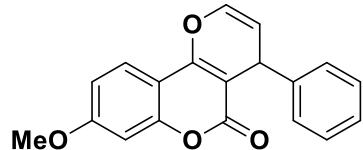
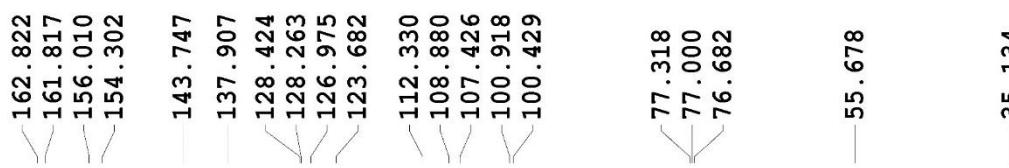
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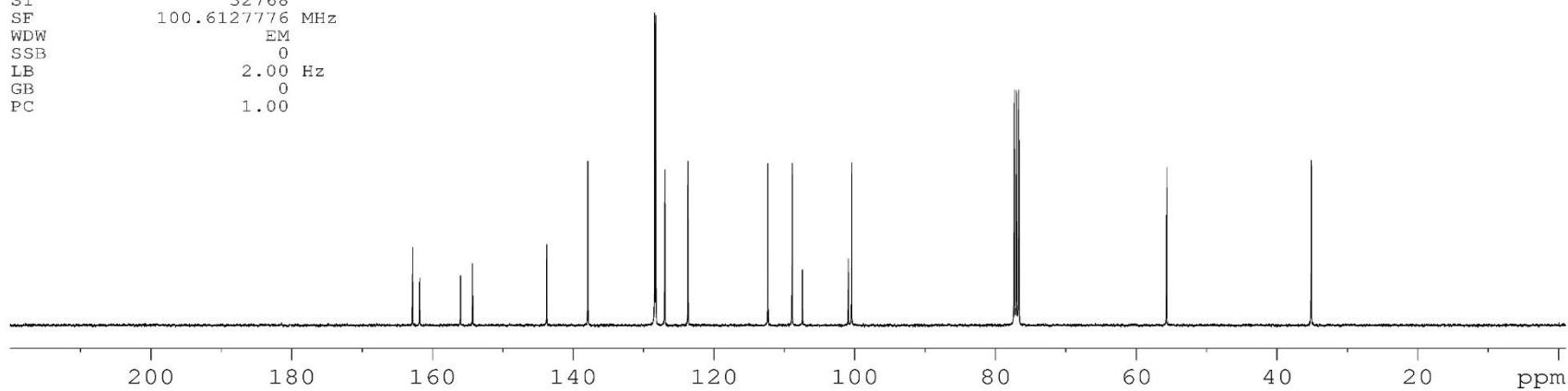


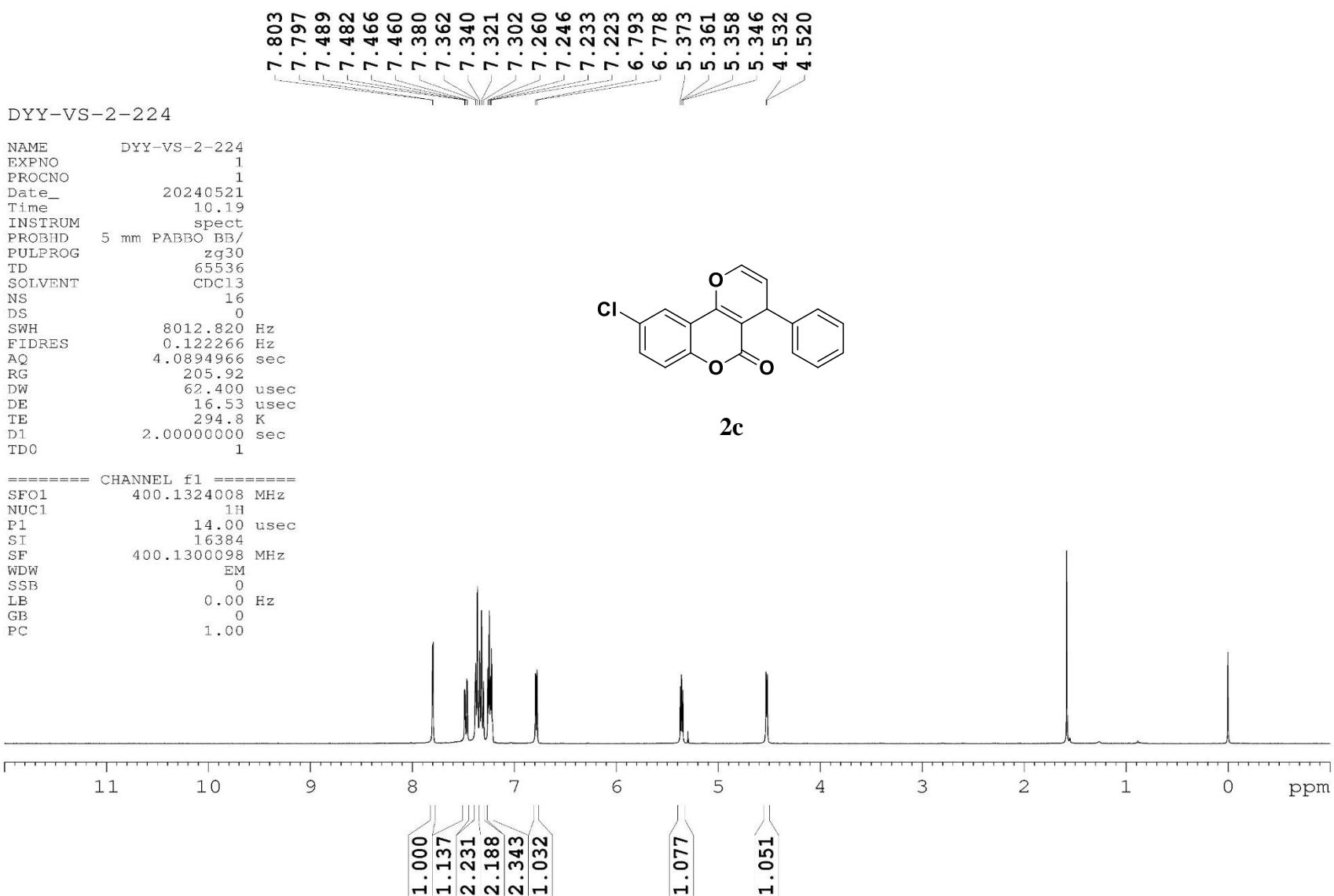
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TD 65536  
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**2b**

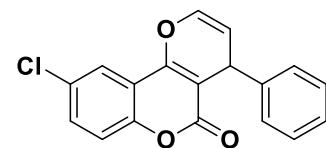
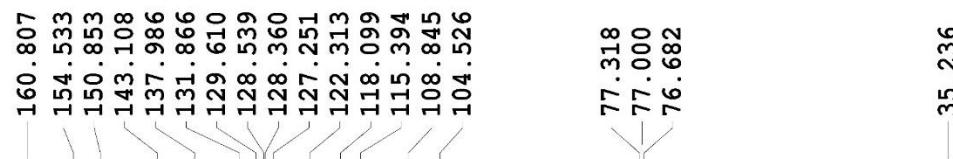




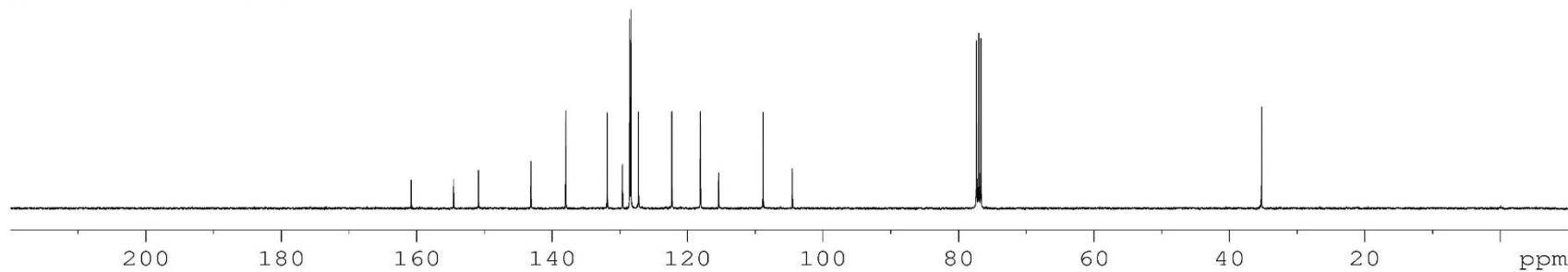
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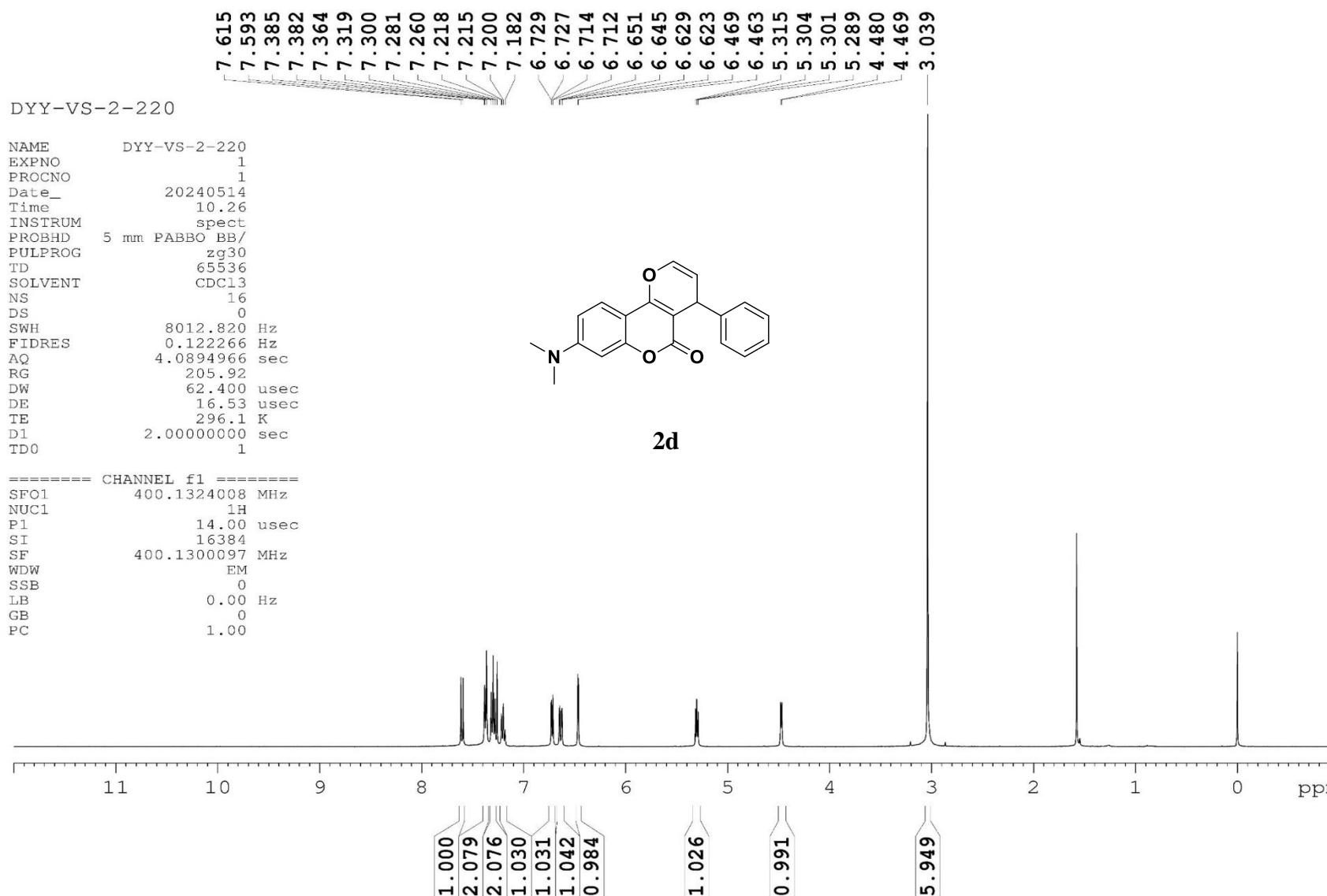
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SOLVENT CDC13  
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FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
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DE 6.50 usec  
TE 297.0 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TDO 1

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2c

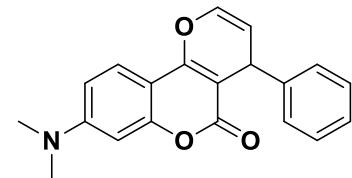
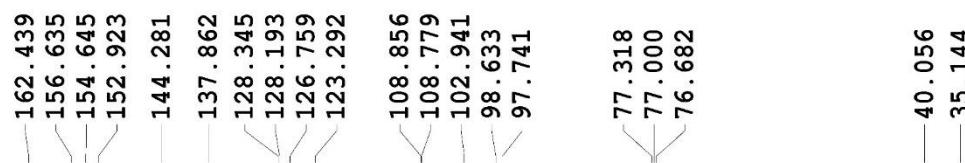




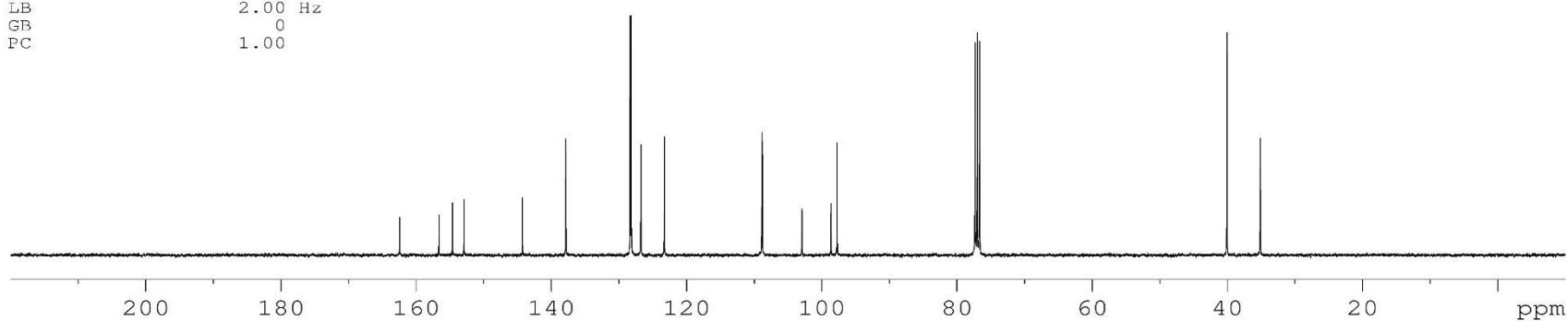
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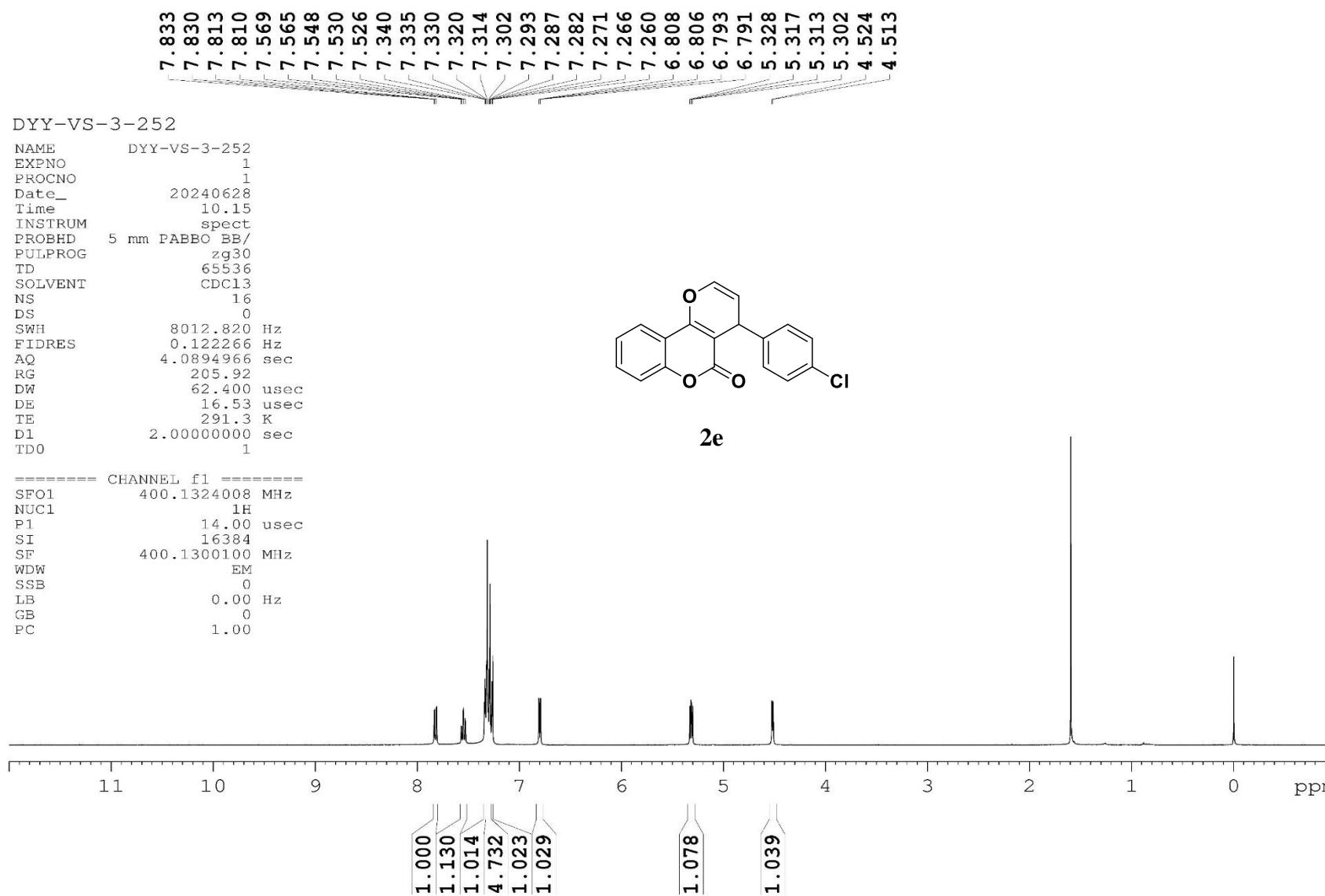
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NS 467  
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FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 297.6 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TDO 1

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NUC1 13C  
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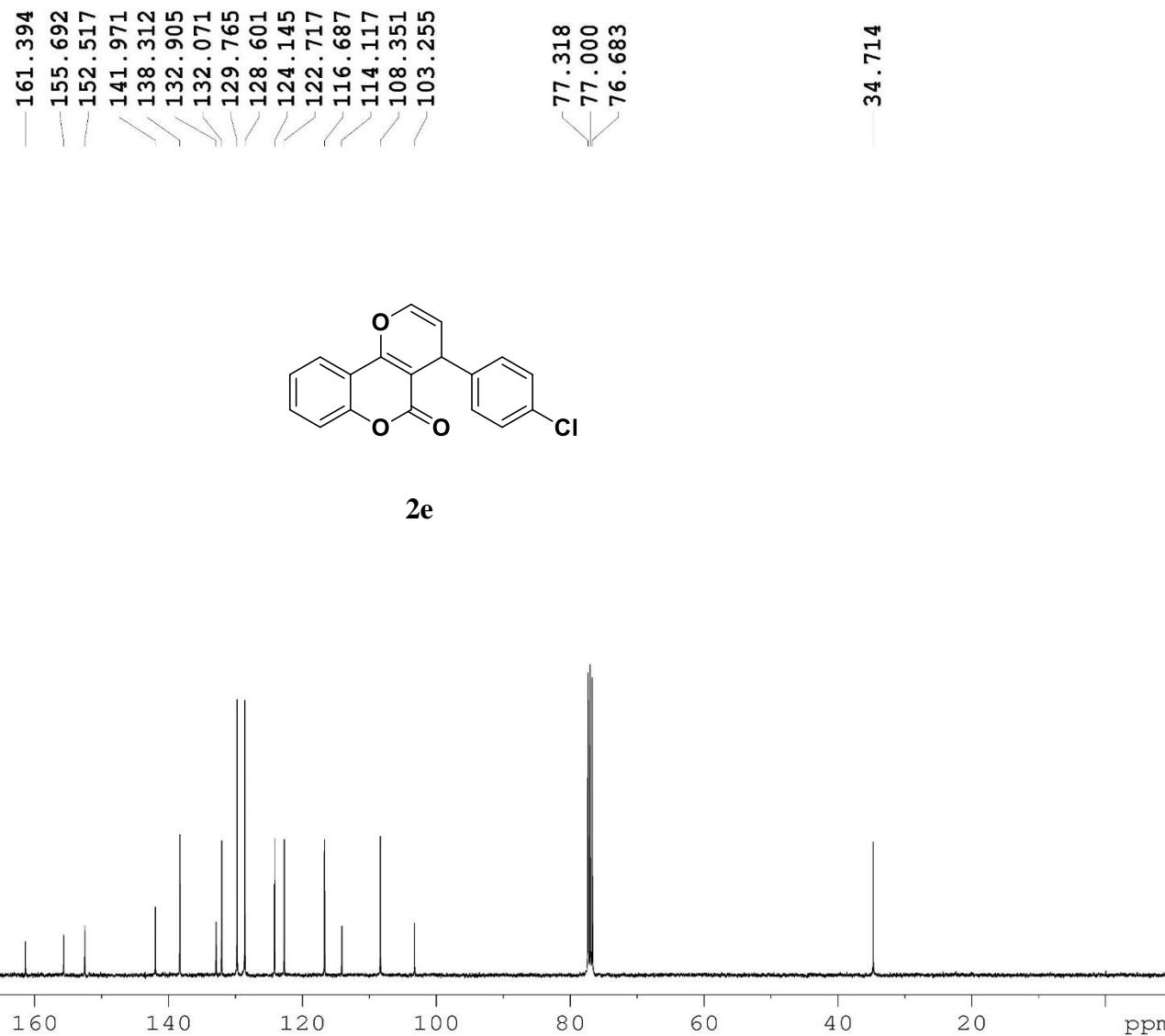
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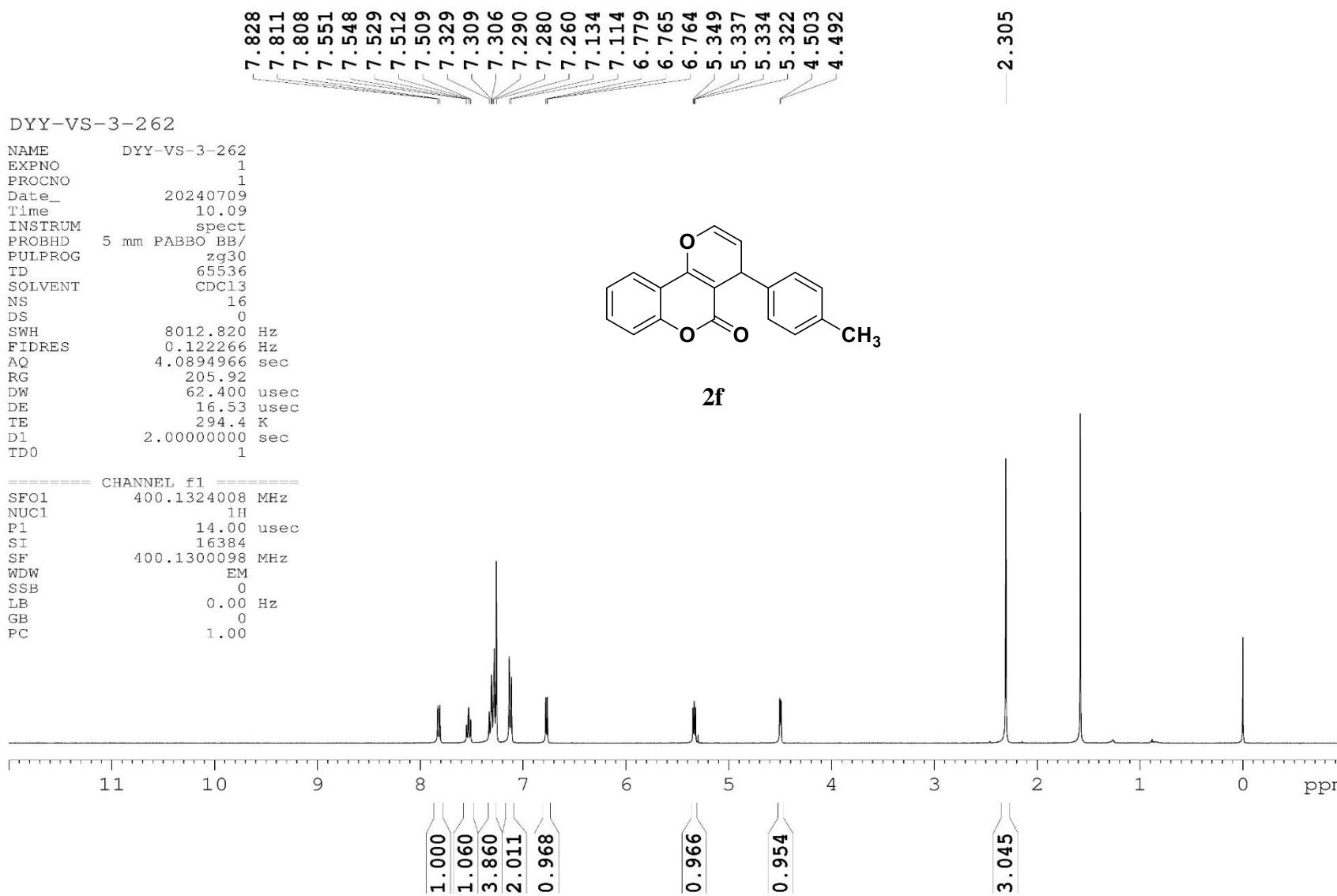


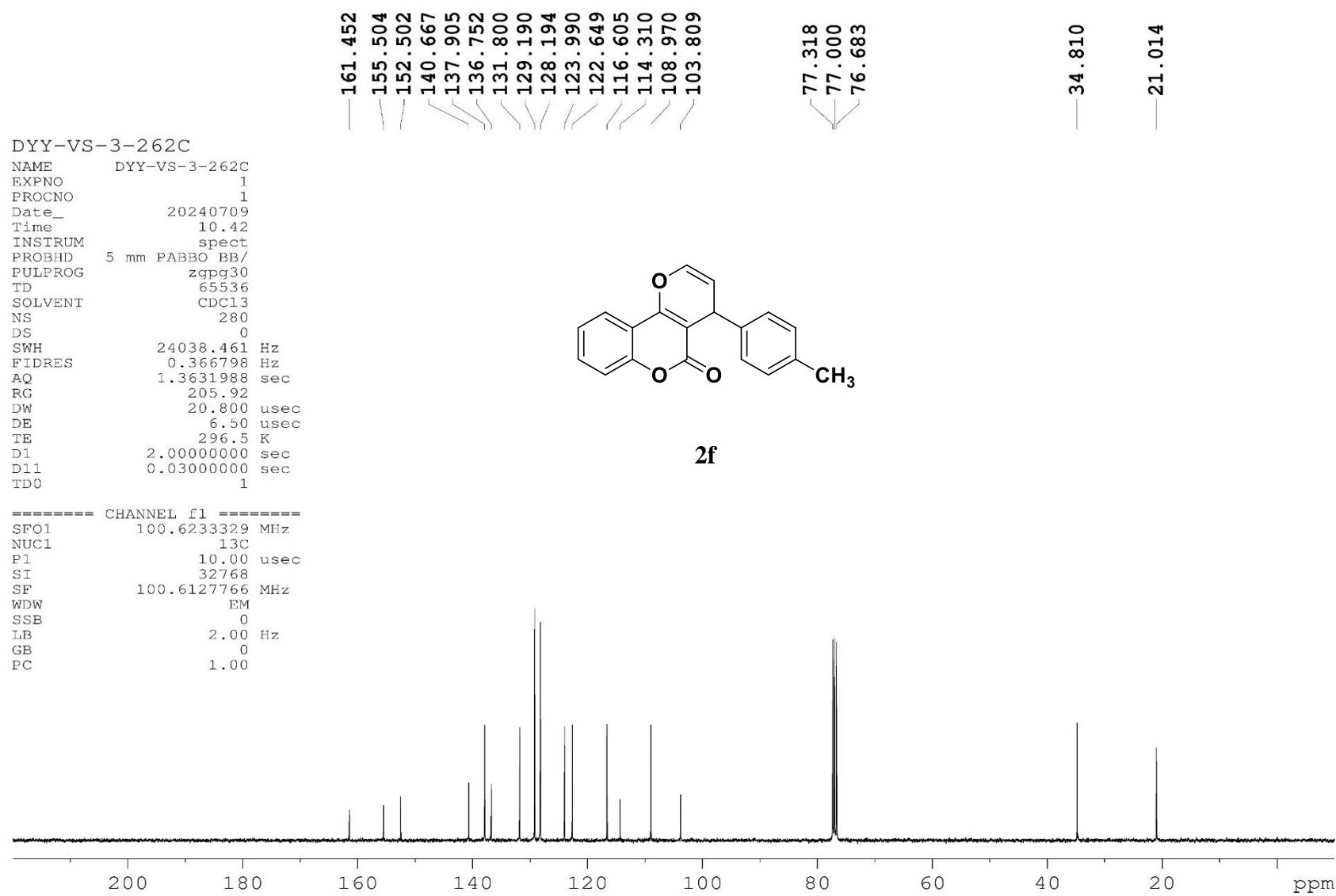


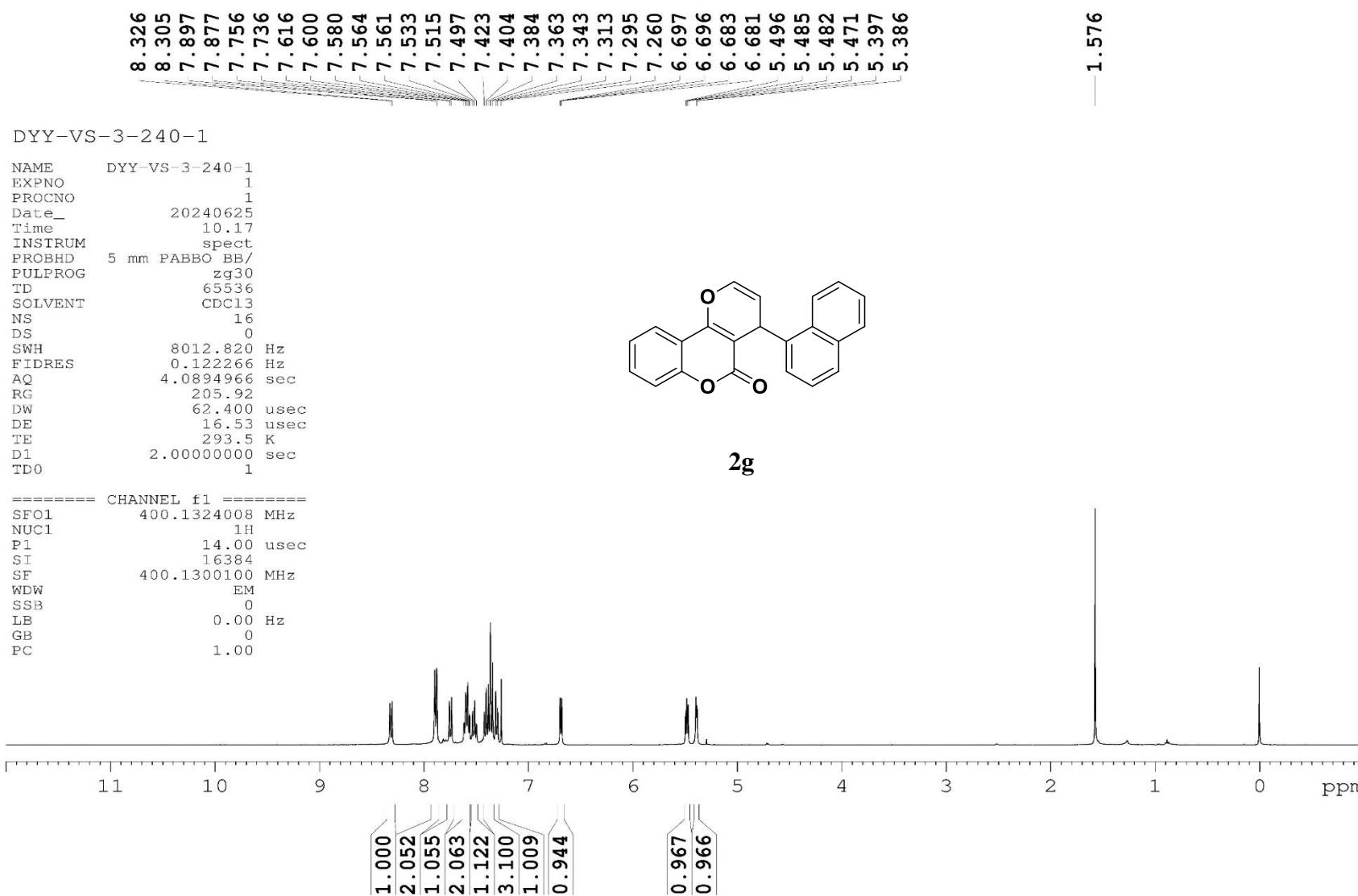
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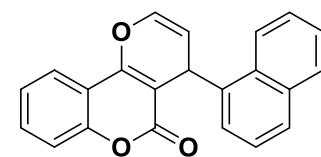
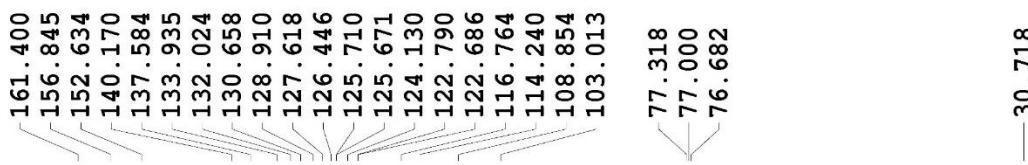




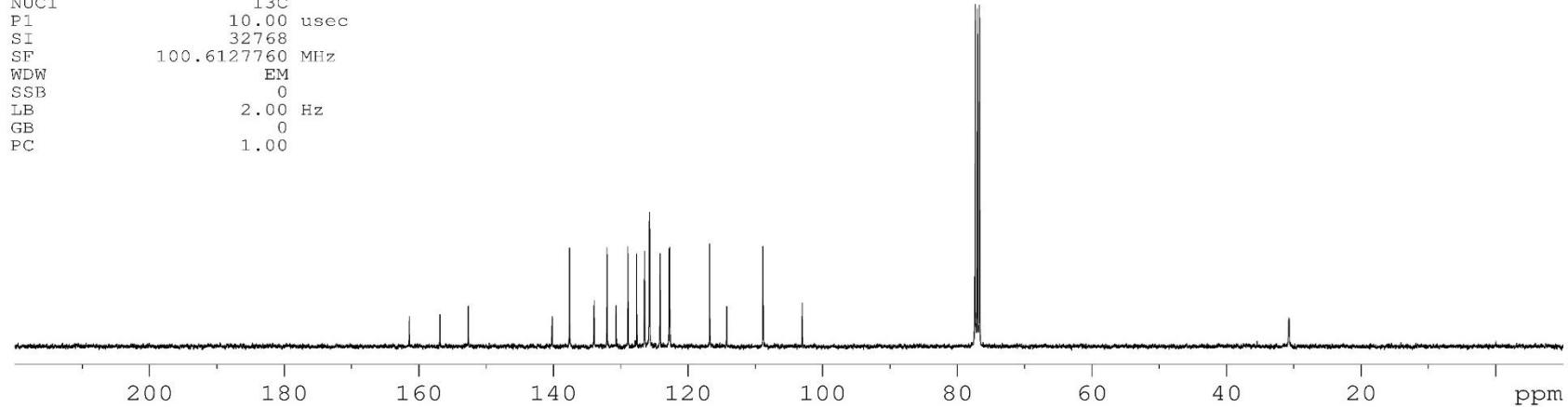
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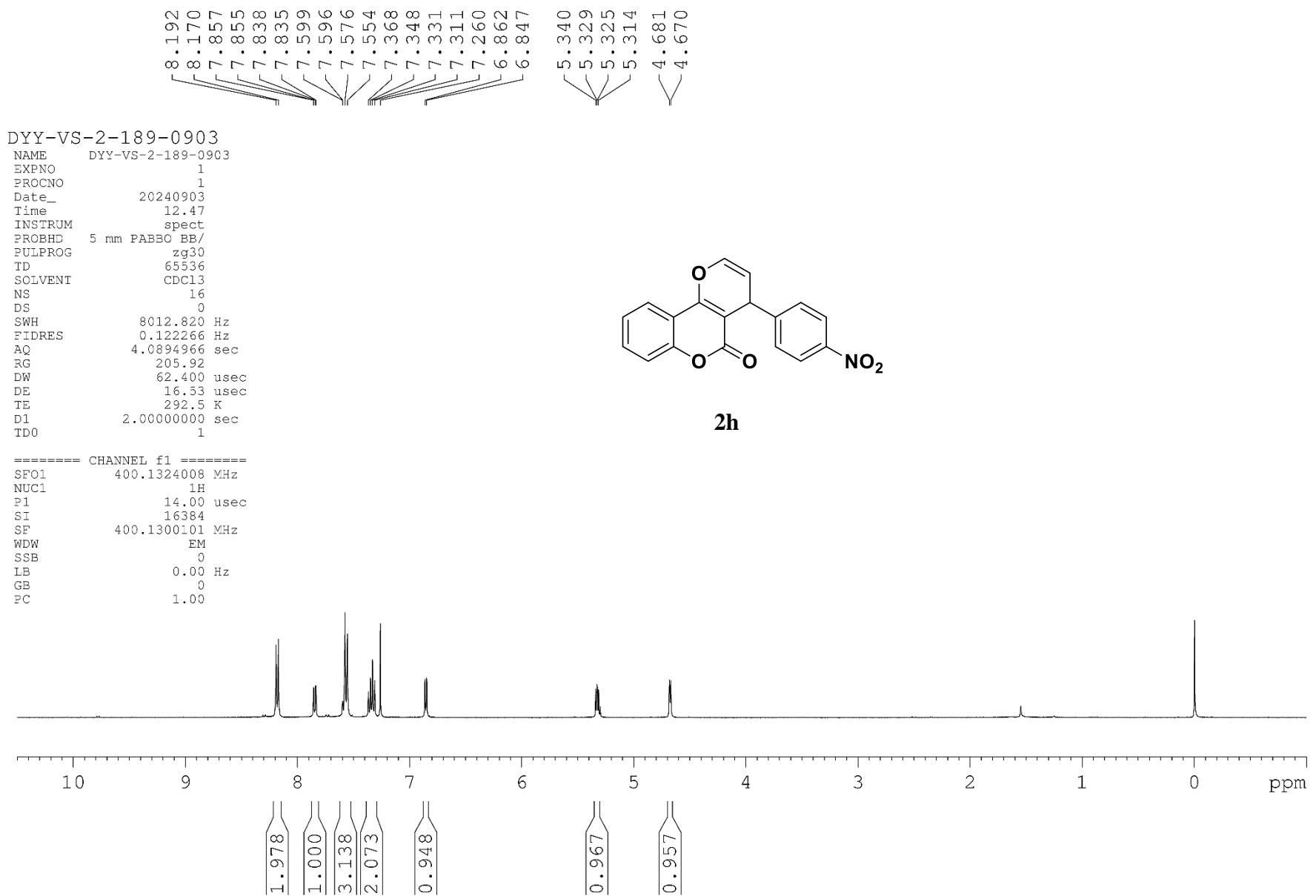
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AQ 1.3631988 sec  
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DW 20.800 usec  
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D1 2.0000000 sec  
D11 0.03000000 sec  
TD0 1

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NUC1 13C  
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**2g**

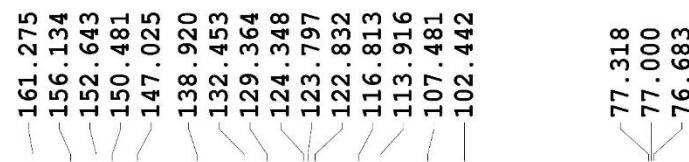




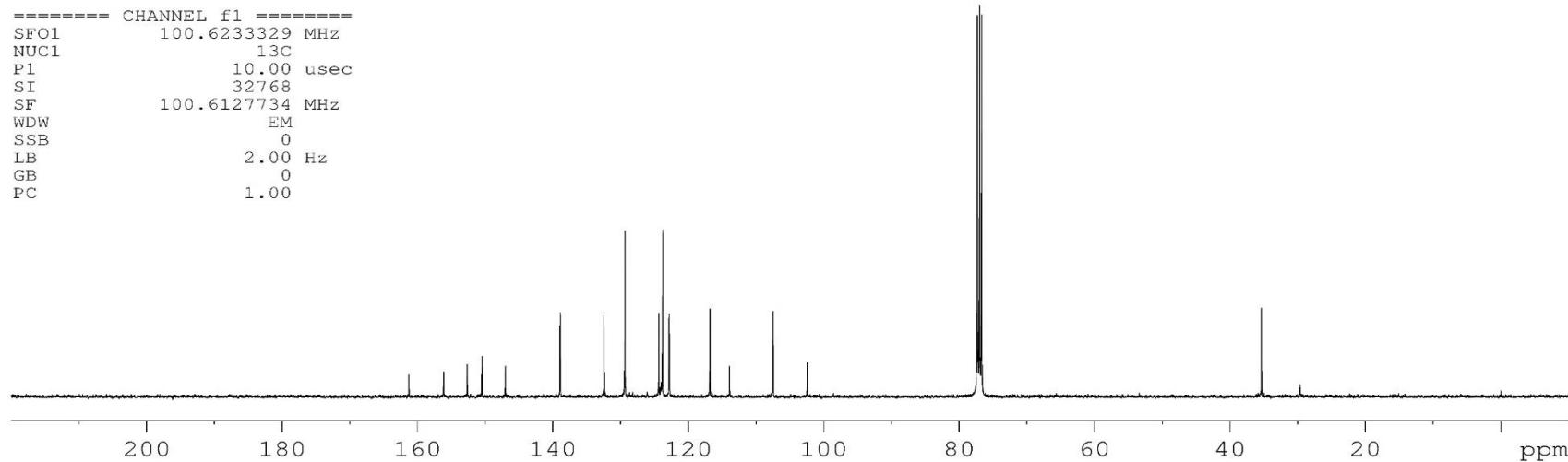
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AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
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TE 297.1 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TDO 1

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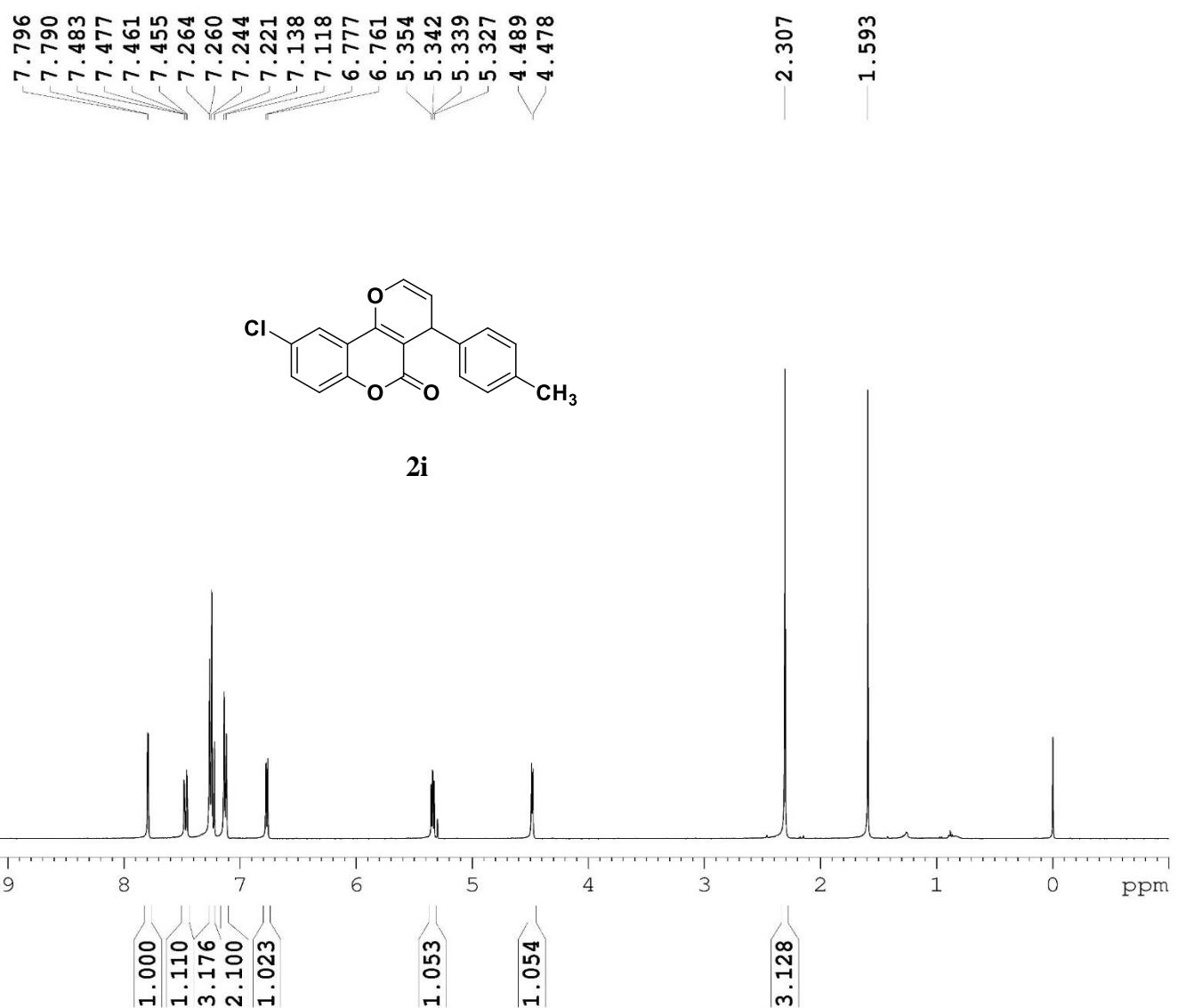
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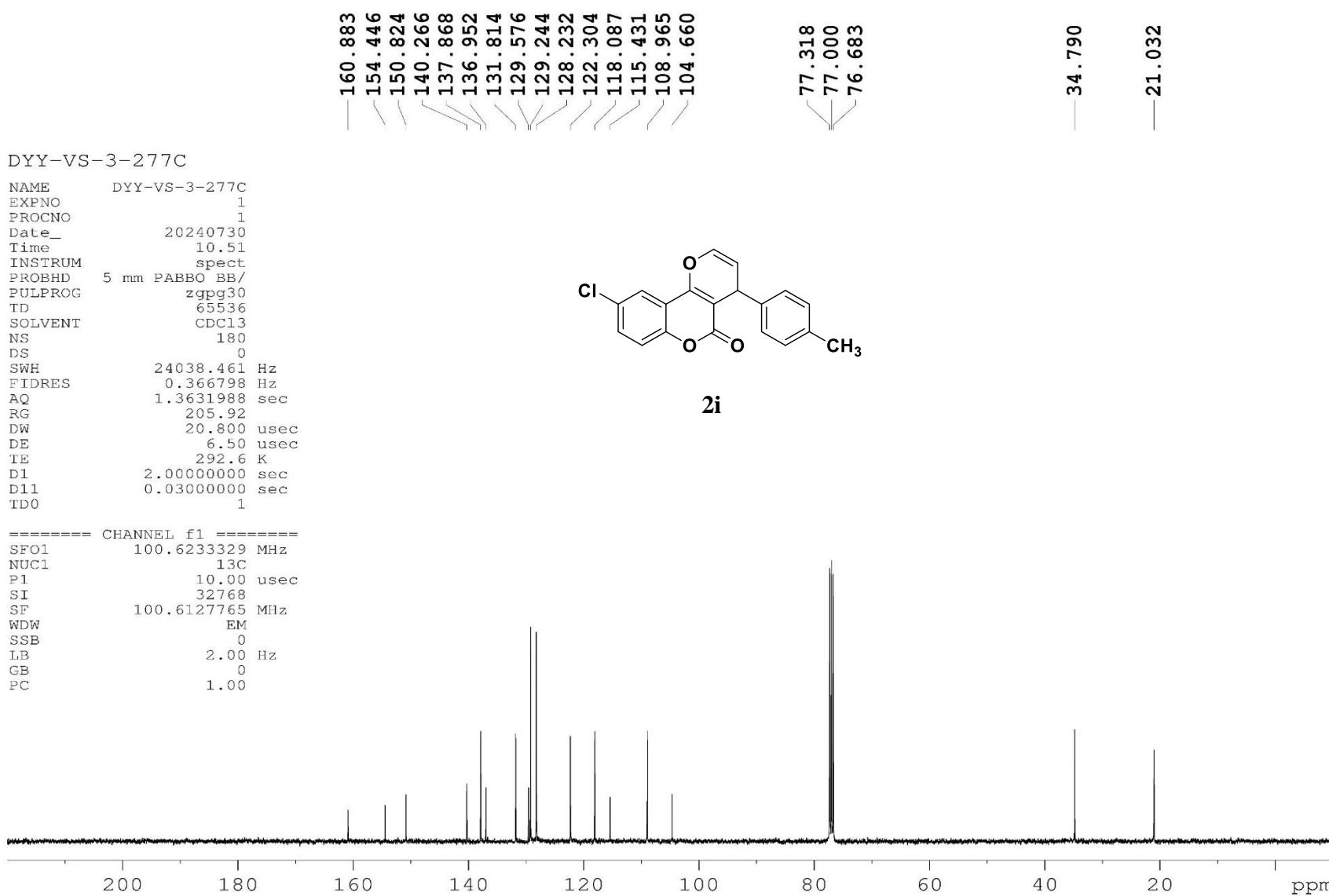


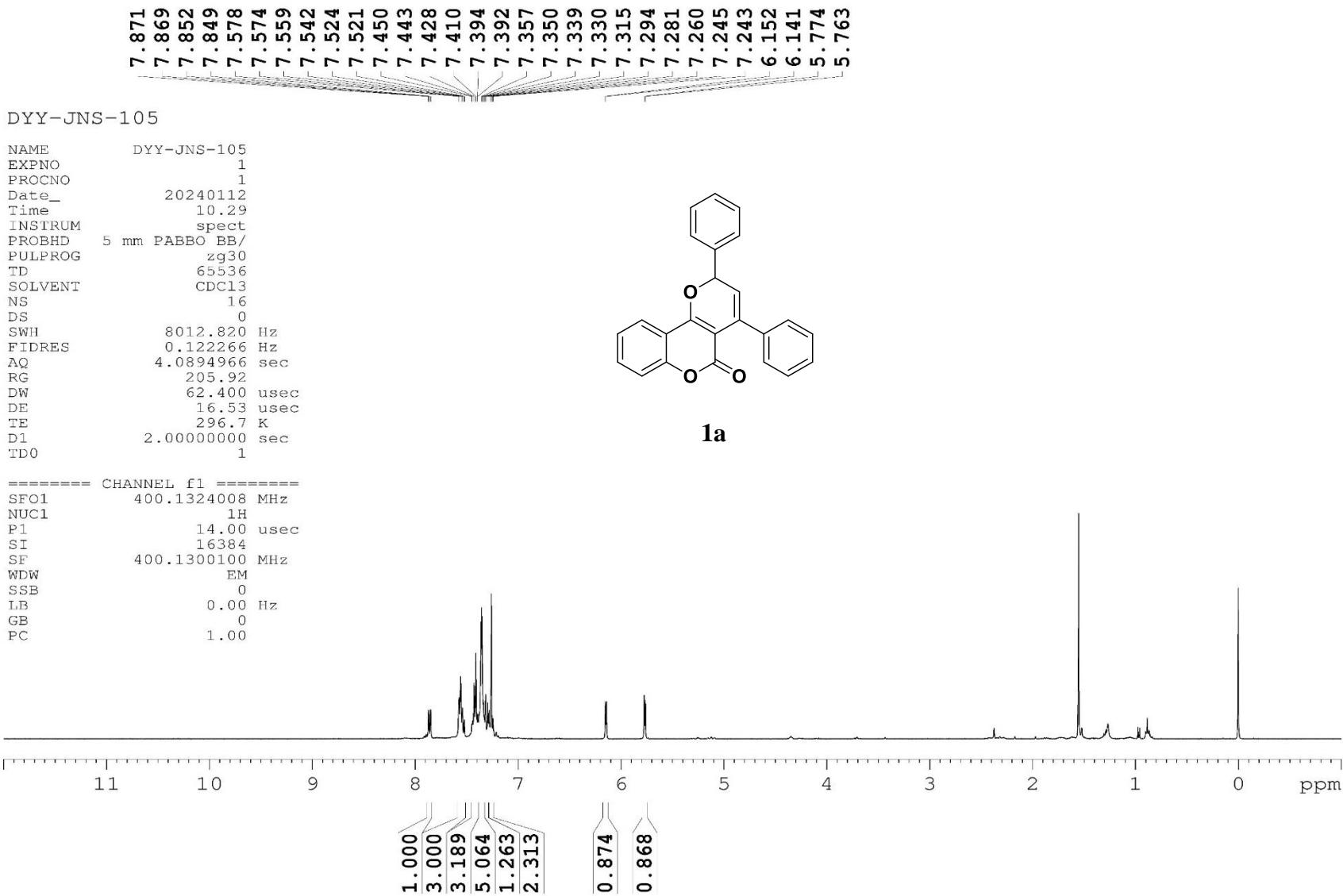
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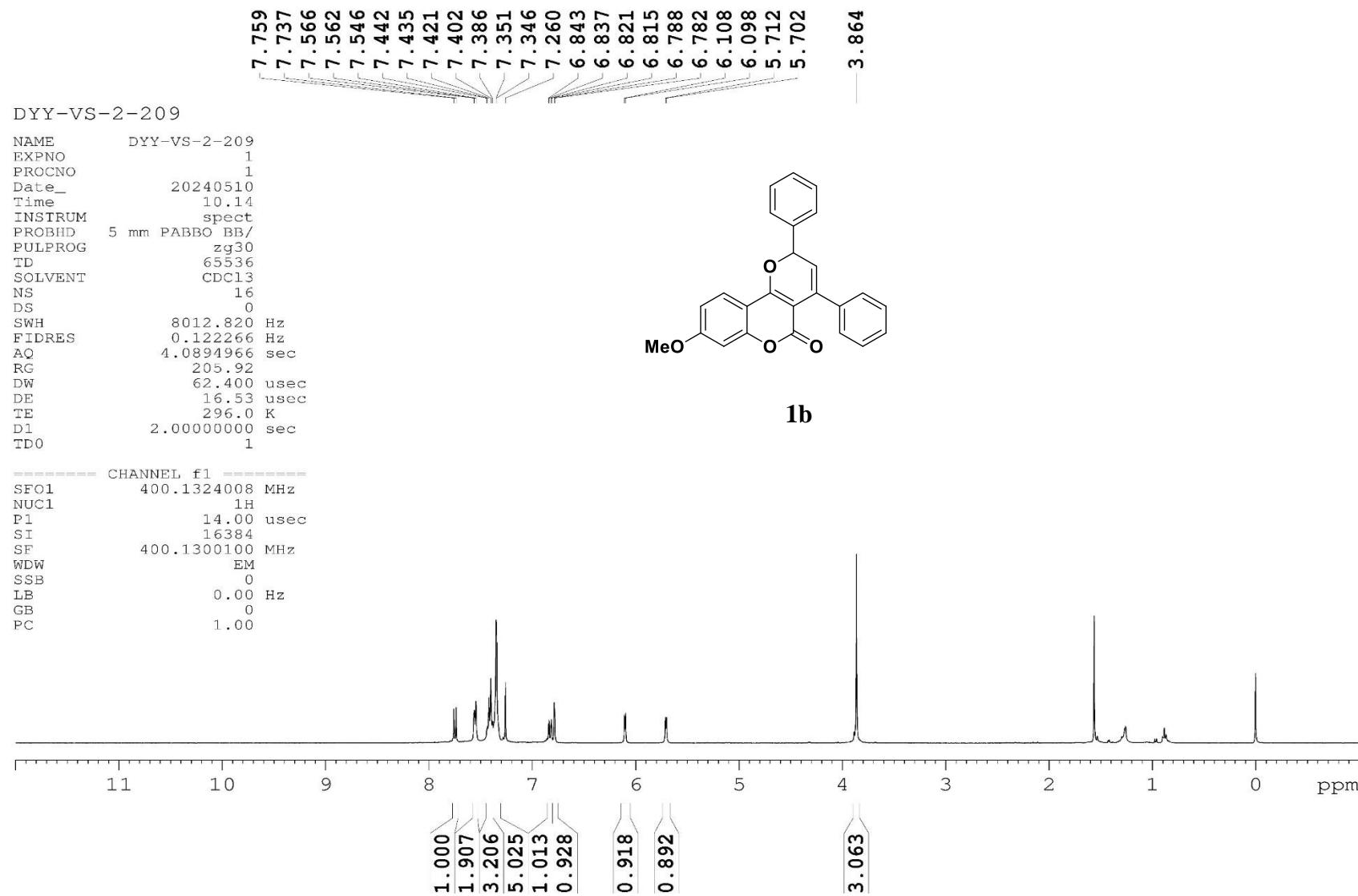
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NS 16  
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FIDRES 0.122266 Hz  
AQ 4.0894966 sec  
RG 205.92  
DW 62.400 usec  
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TE 291.5 K  
D1 2.0000000 sec  
TD0 1

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NUC1 1H  
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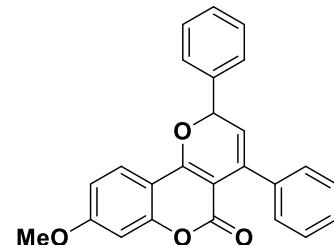
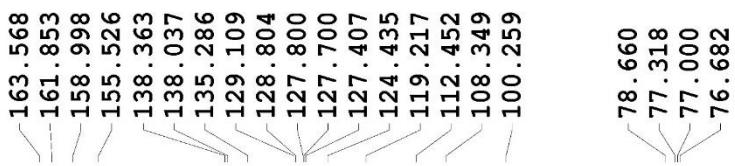




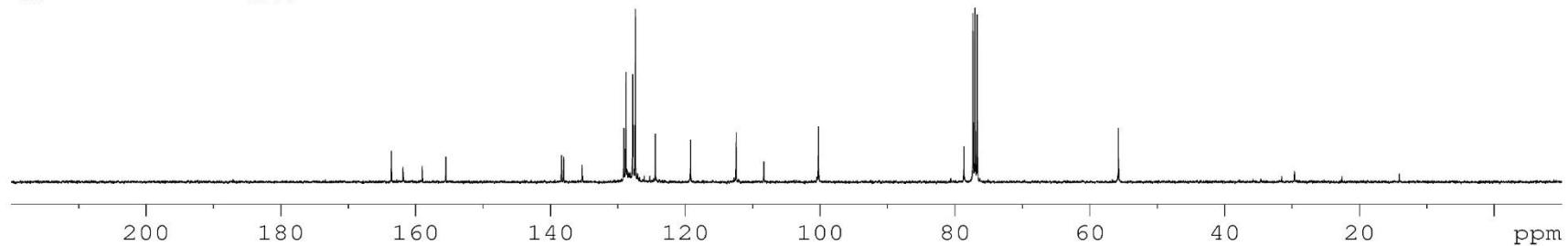
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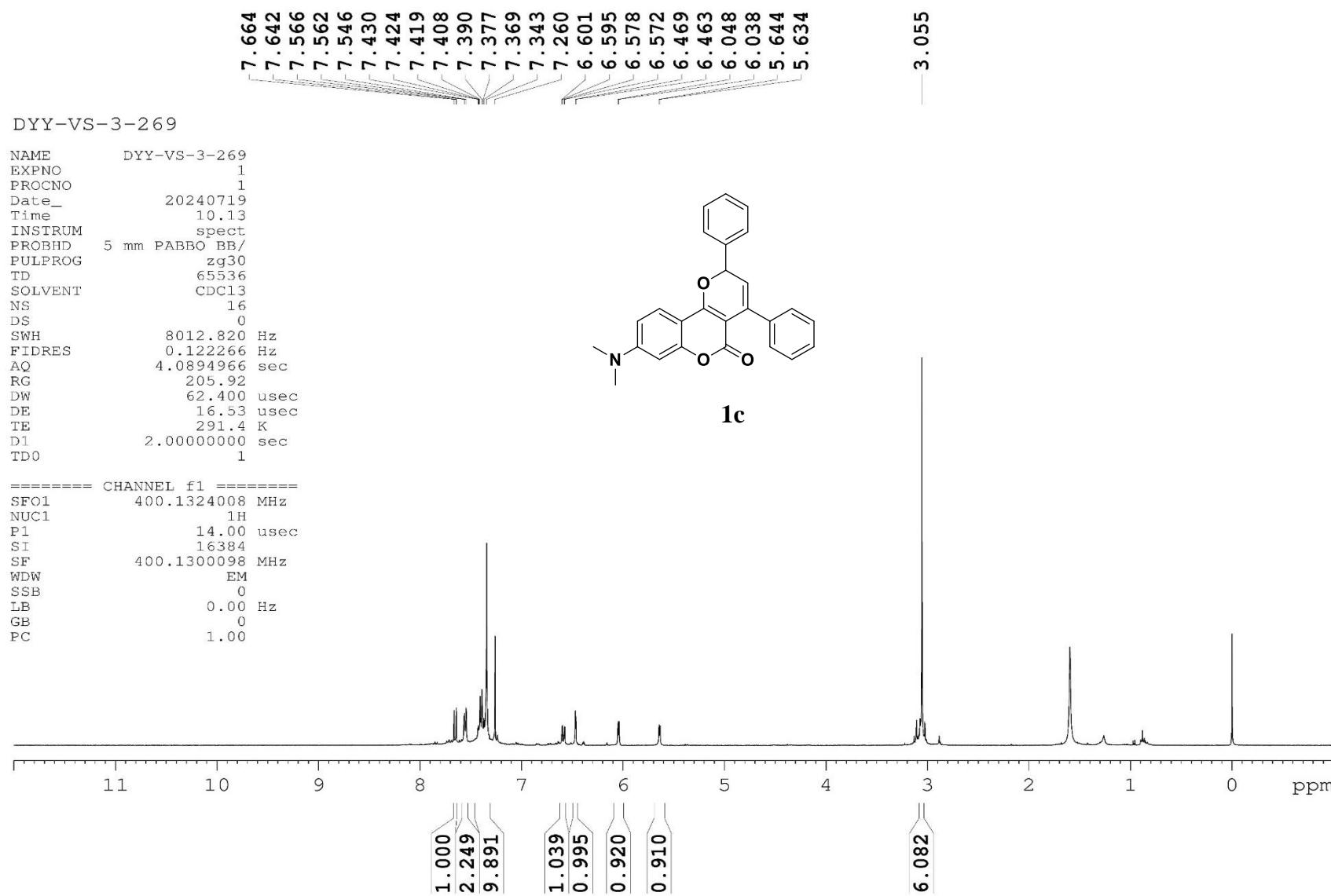
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AQ 1.3631988 sec  
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DE 6.50 usec  
TE 297.5 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TD0 1

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NUC1 <sup>13</sup>C  
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**1b**





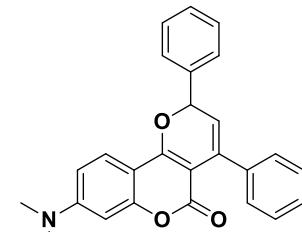
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NS 500  
DS 0  
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FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
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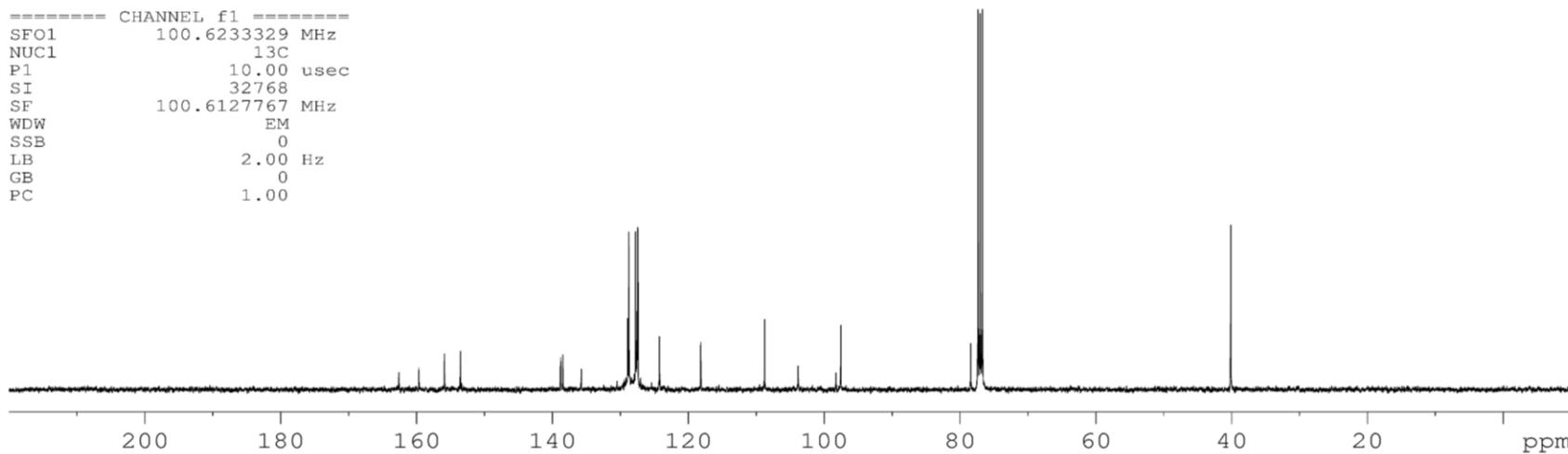
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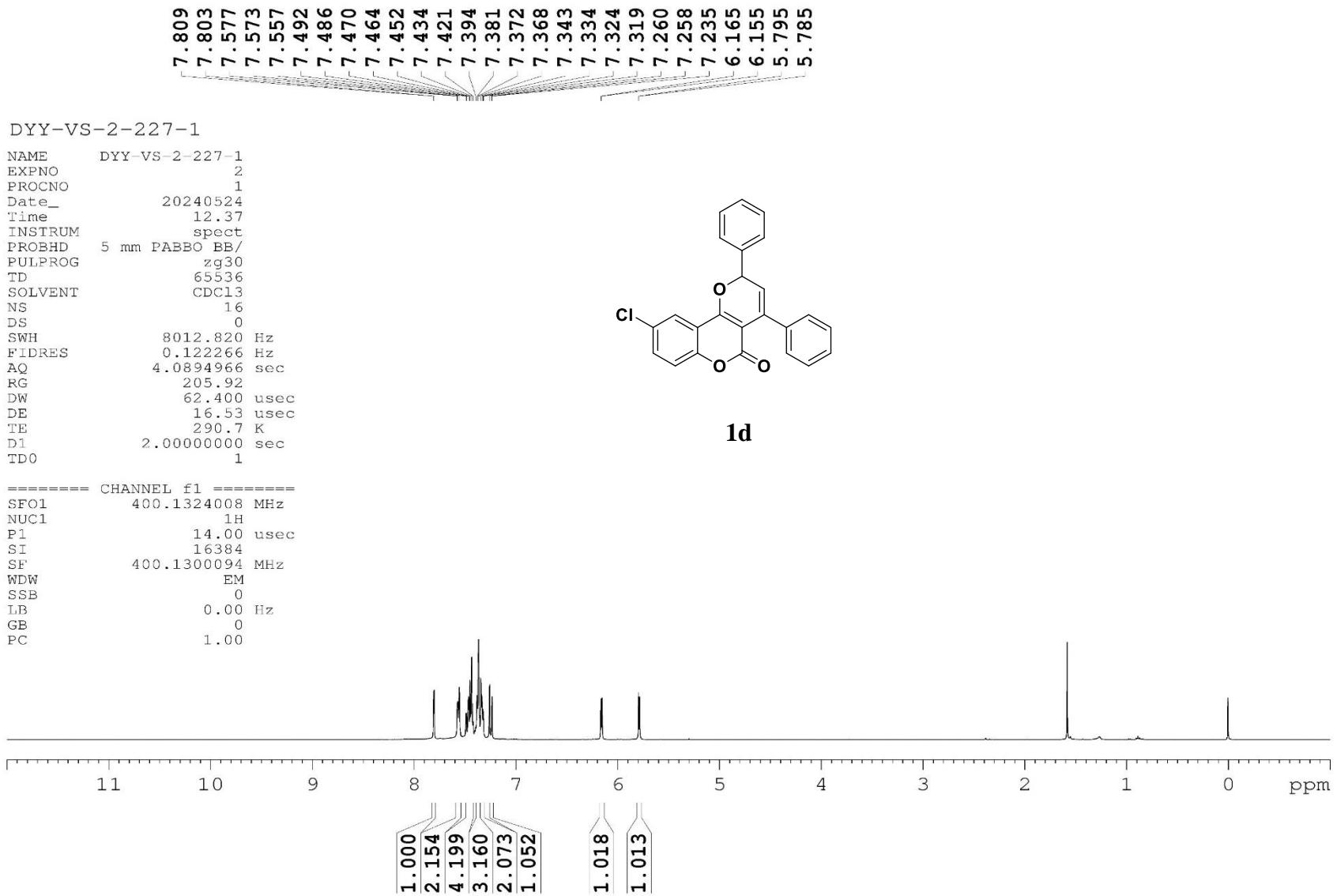


— 40.117 —



**1c**

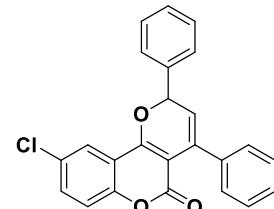
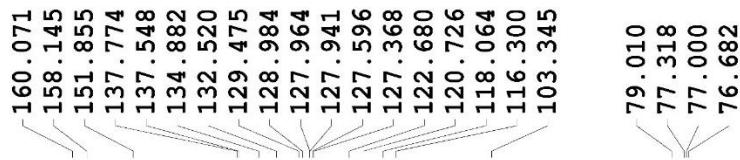




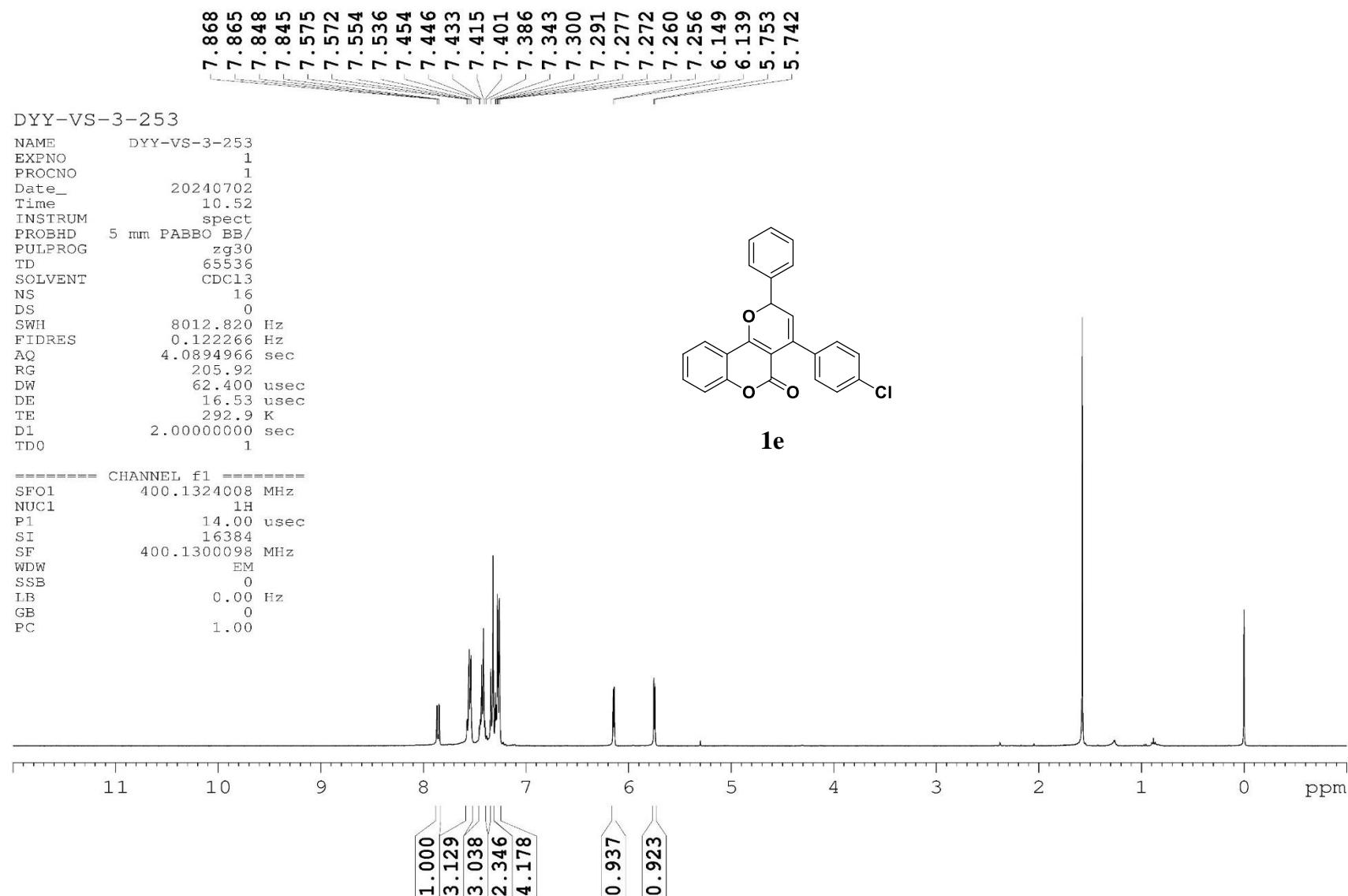
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TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 2000  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 291.8 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127742 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



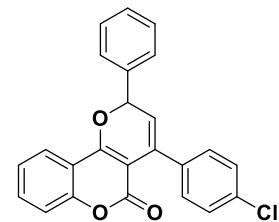
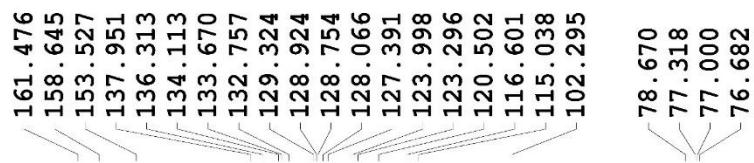
**1d**



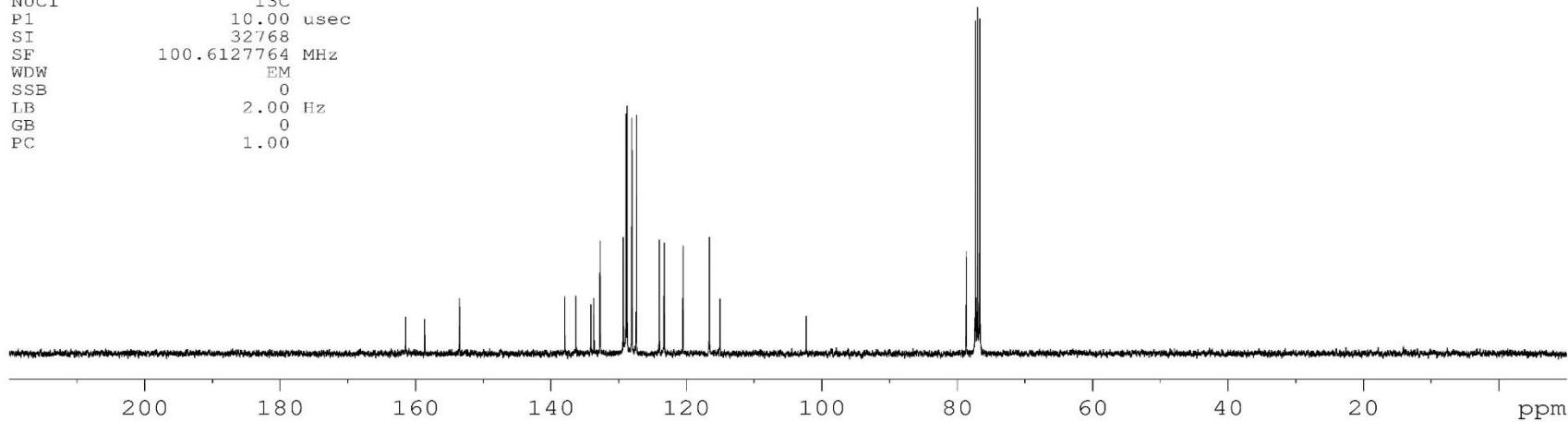
DYY-VS-3-253C

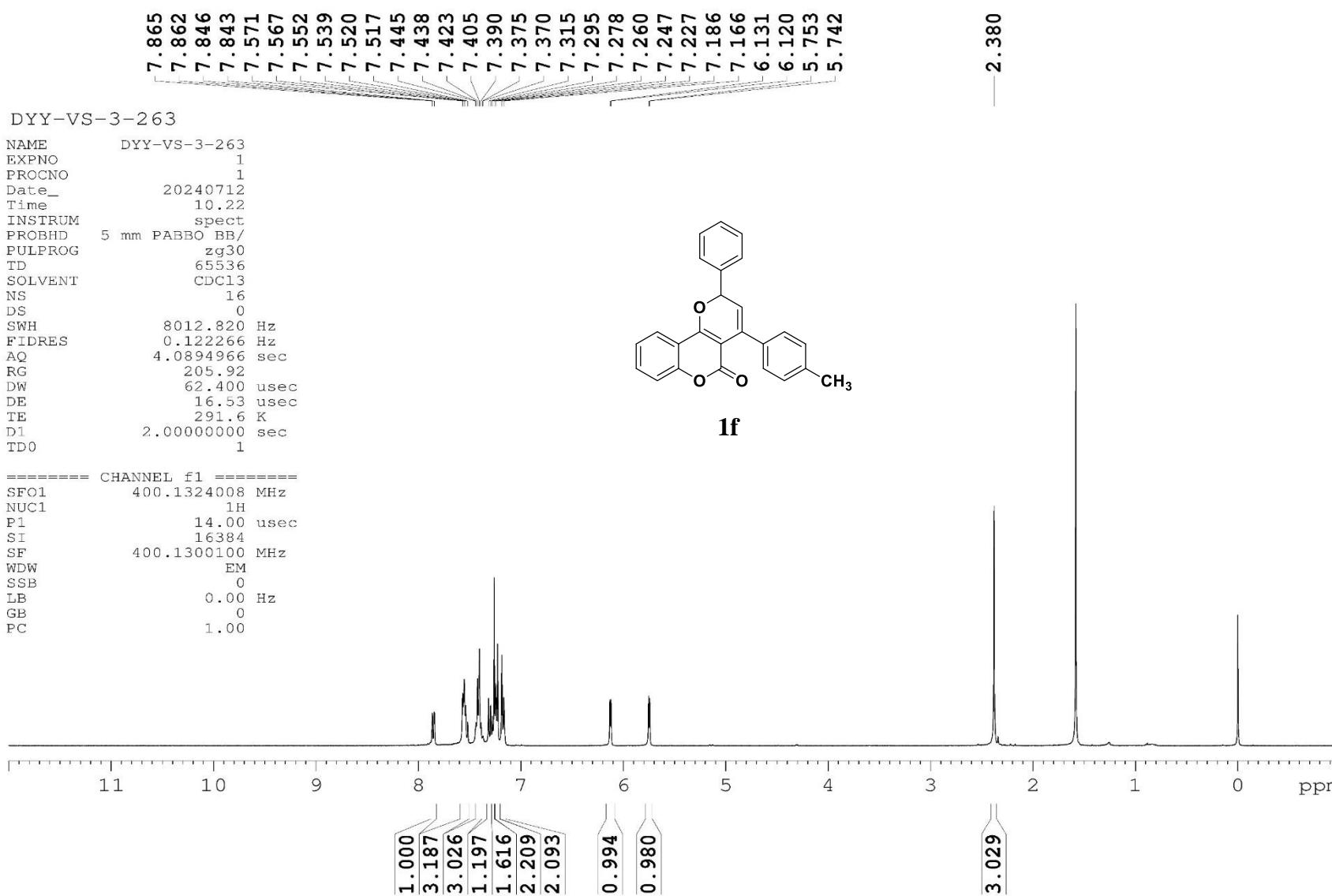
NAME DYY-VS-3-253C  
EXPNO 1  
PROCNO 1  
Date 20240702  
Time 11.07  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 210  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 294.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127764 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



**1e**

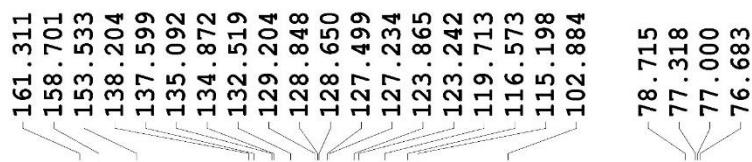




DYY-VS-3-263C

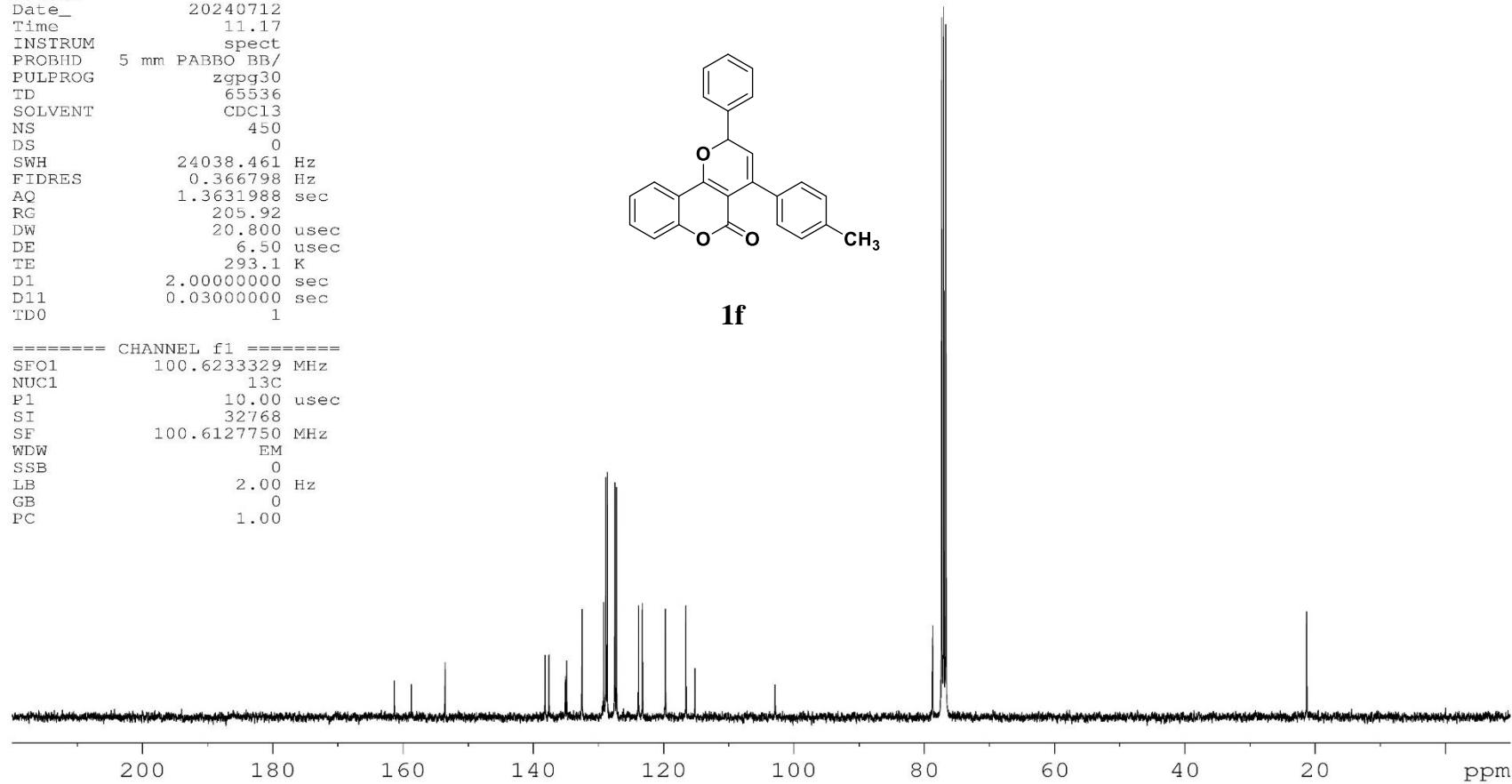
NAME DYY-VS-3-263C  
EXPNO 1  
PROCNO 1  
Date\_ 20240712  
Time 11.17  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 450  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 293.1 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TD0 1

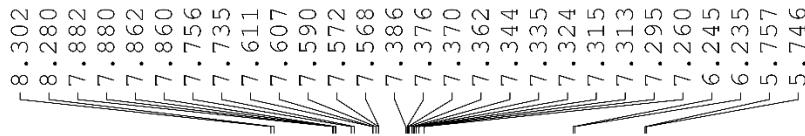
===== CHANNEL f1 ======  
SFO1 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127750 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



—21.293

**1f**

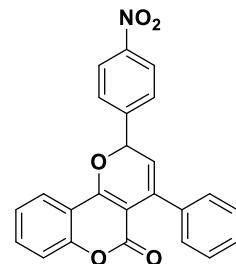




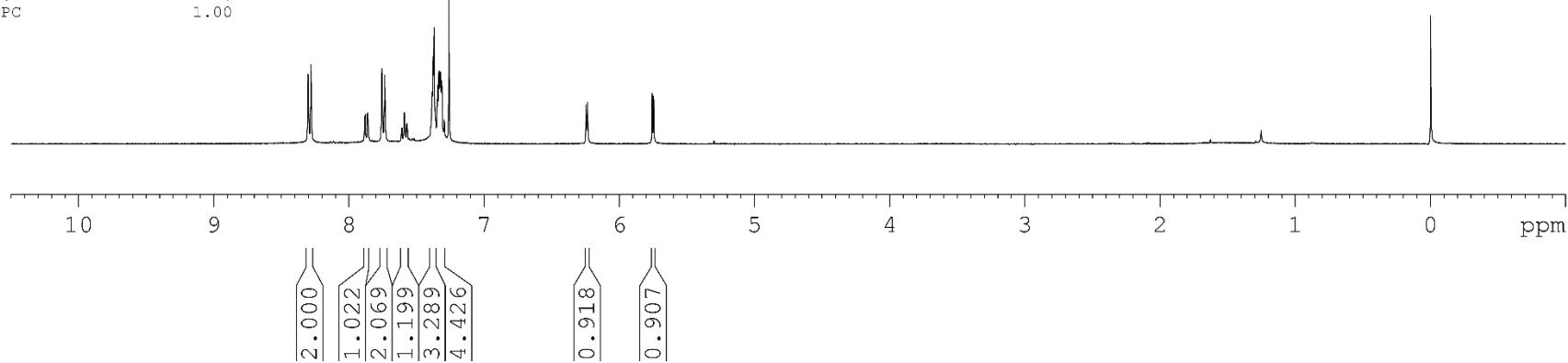
DYY-JNS-146-1-0903

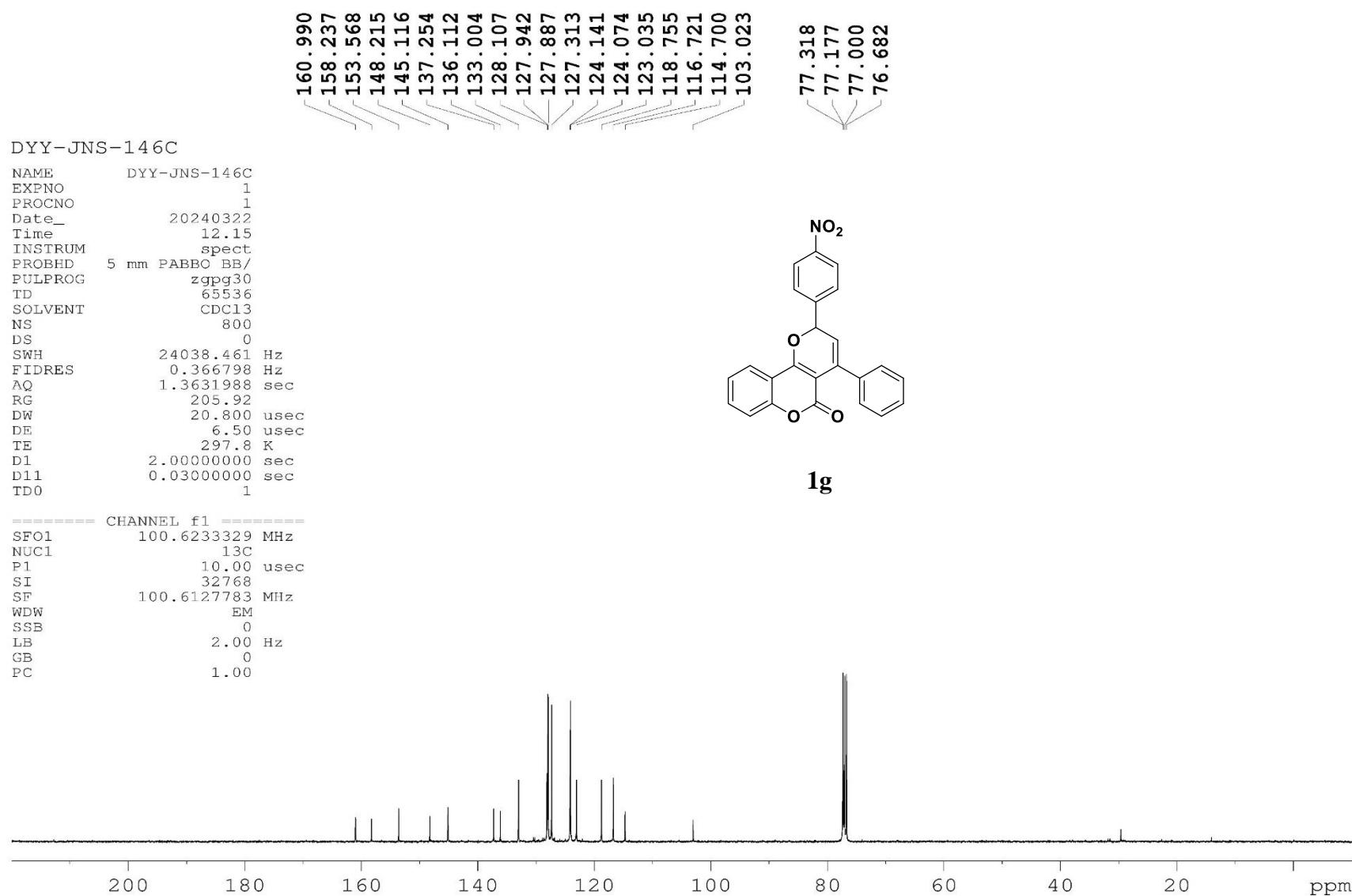
NAME DYY-JNS-146-1-0903  
EXPNO 1  
PROCNO 1  
Date\_ 20240903  
Time 13.01  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 16  
DS 0  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894966 sec  
RG 205.92  
DW 62.400 usec  
DE 16.53 usec  
TE 292.0 K  
D1 2.0000000 sec  
TD0 1

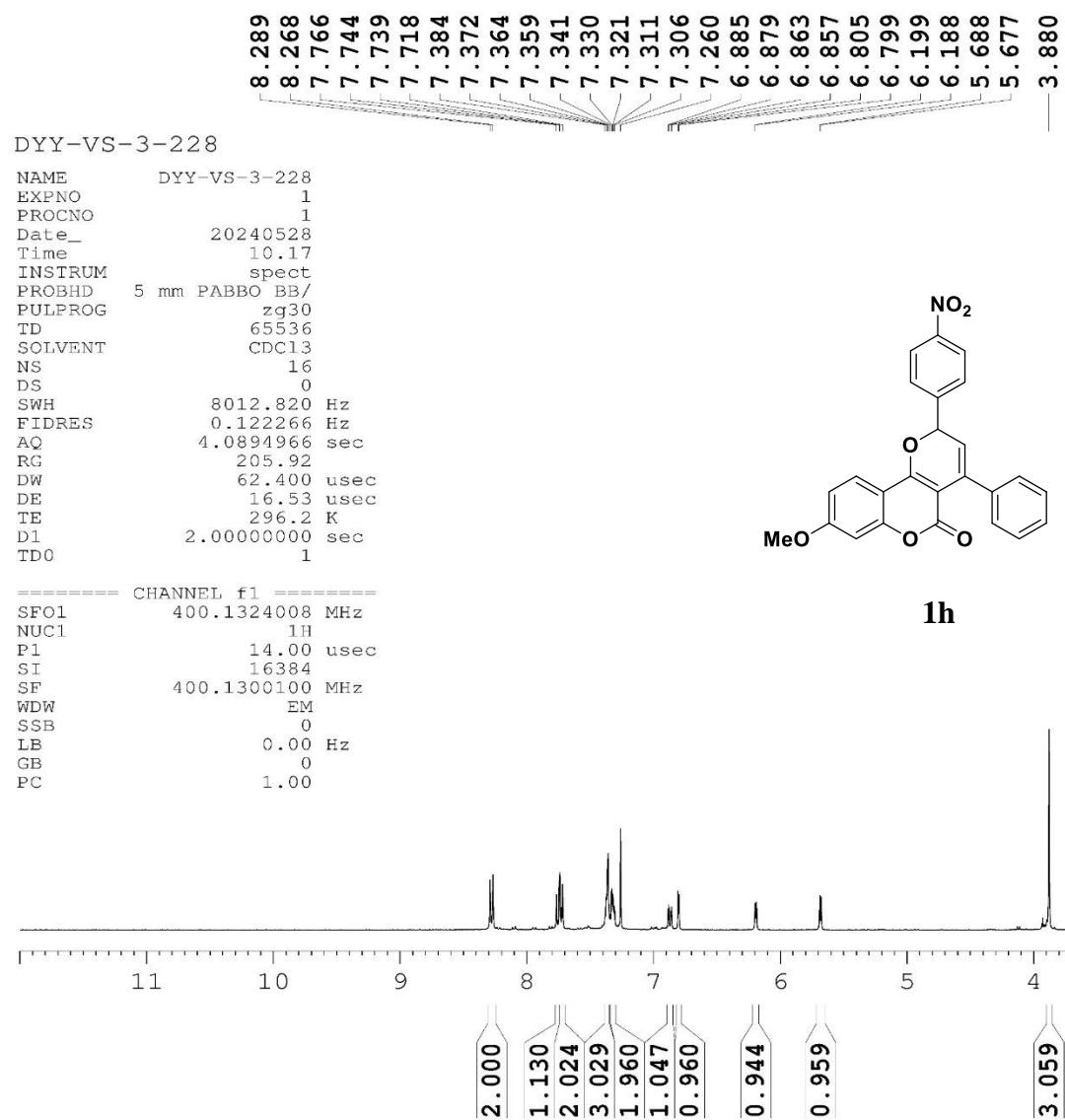
===== CHANNEL f1 ======  
SFO1 400.1324008 MHz  
NUC1 1H  
P1 14.00 usec  
SI 16384  
SF 400.1300101 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00



**1g**

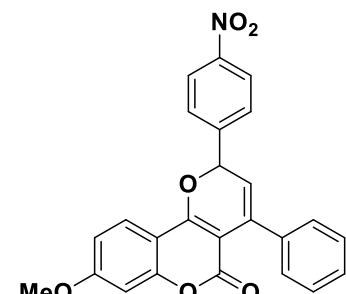
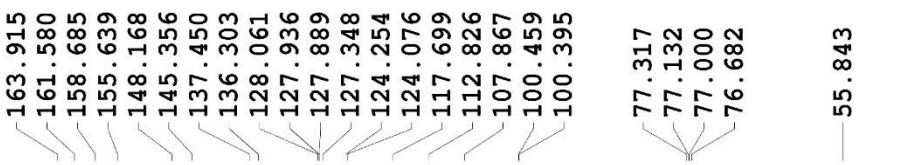




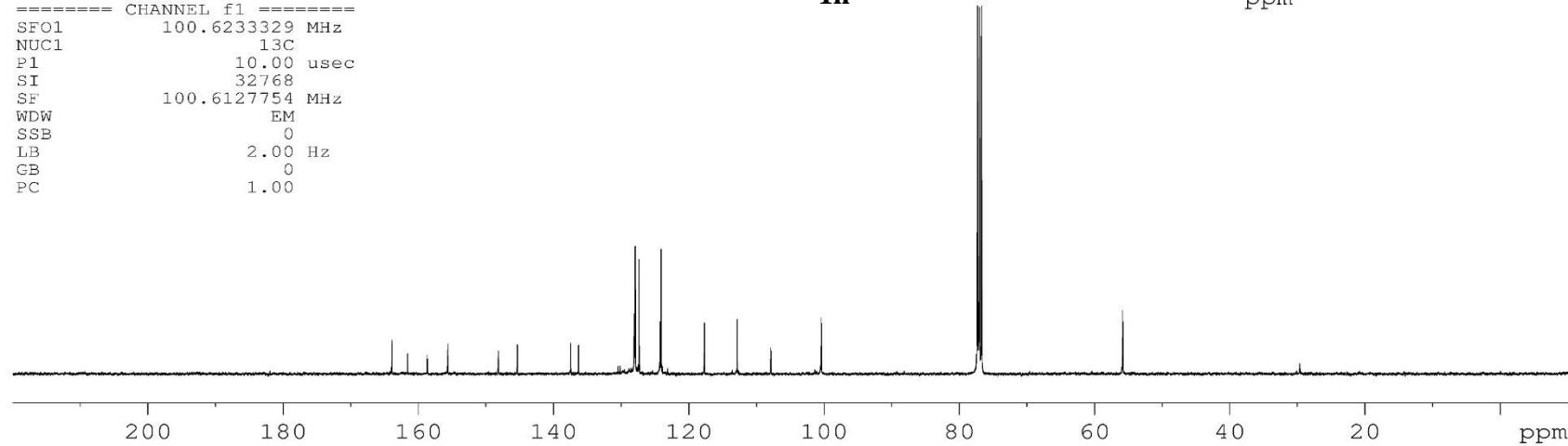
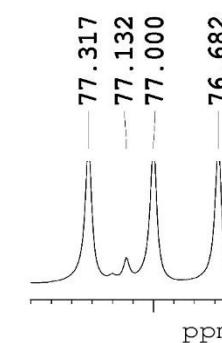


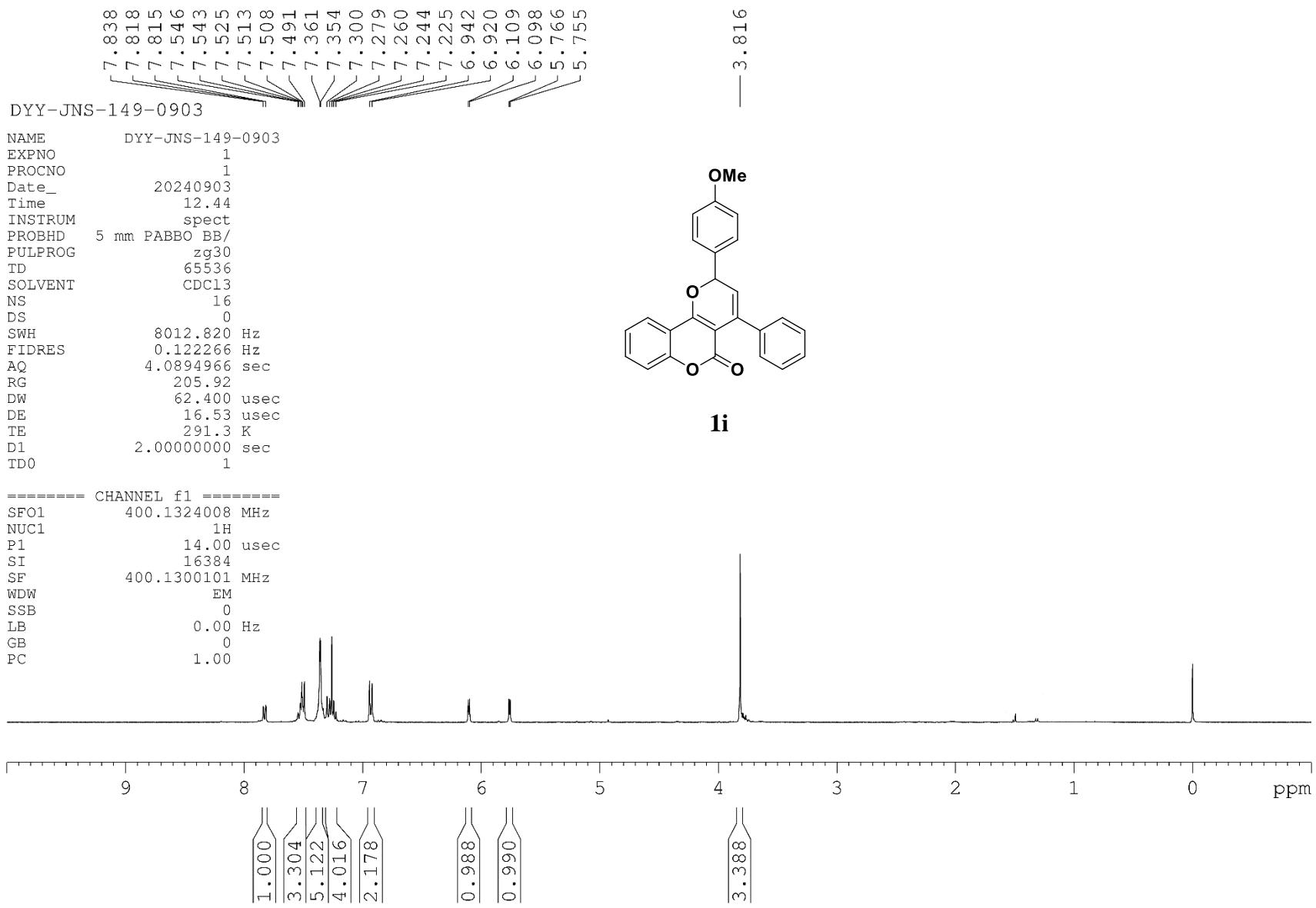
DYY-VS-3-228C

NAME DYY-VS-3-228C  
EXPNO 1  
PROCNO 1  
Date\_ 20240531  
Time 12.36  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgppg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 1200  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 293.1 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TDO 1  
===== CHANNEL f1 ======  
SF01 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127754 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



55.843

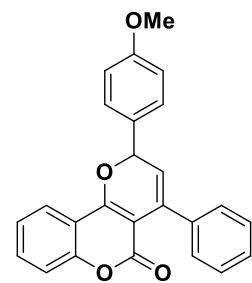
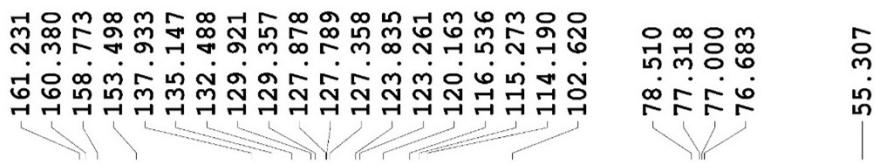




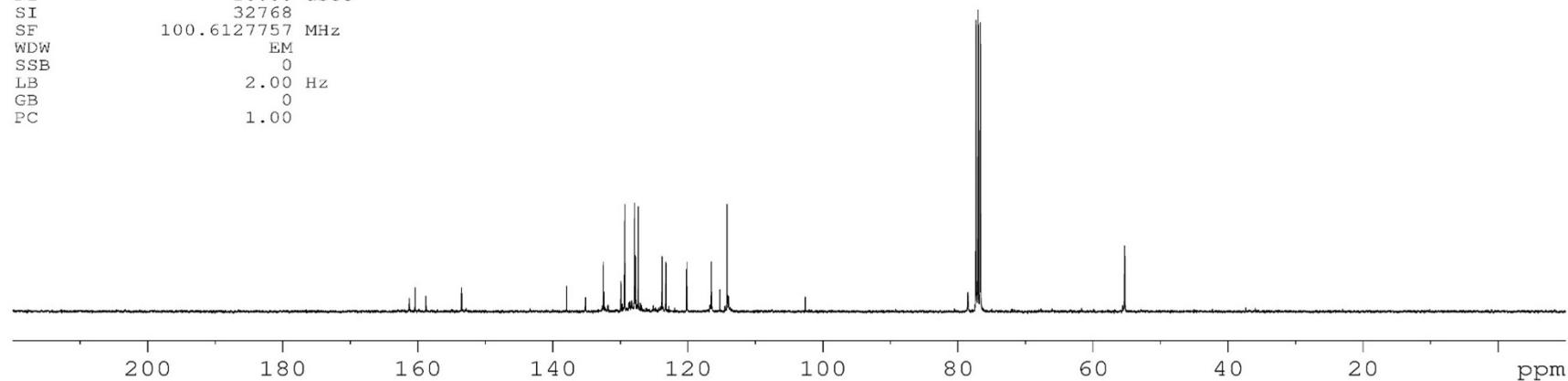
DYY-JNS-149C

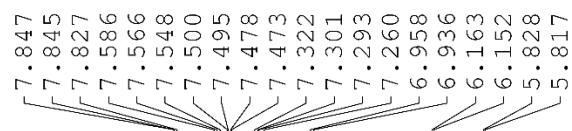
NAME DYY-JNS-149C  
EXPNO 1  
PROCNO 1  
Date\_ 20240326  
Time 11.17  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 32768  
SOLVENT CDC13  
NS 1500  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.733596 Hz  
AQ 0.6816244 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 292.1 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 13C  
P1 10.00 usec  
SI 32768  
SF 100.6127757 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



**1i**

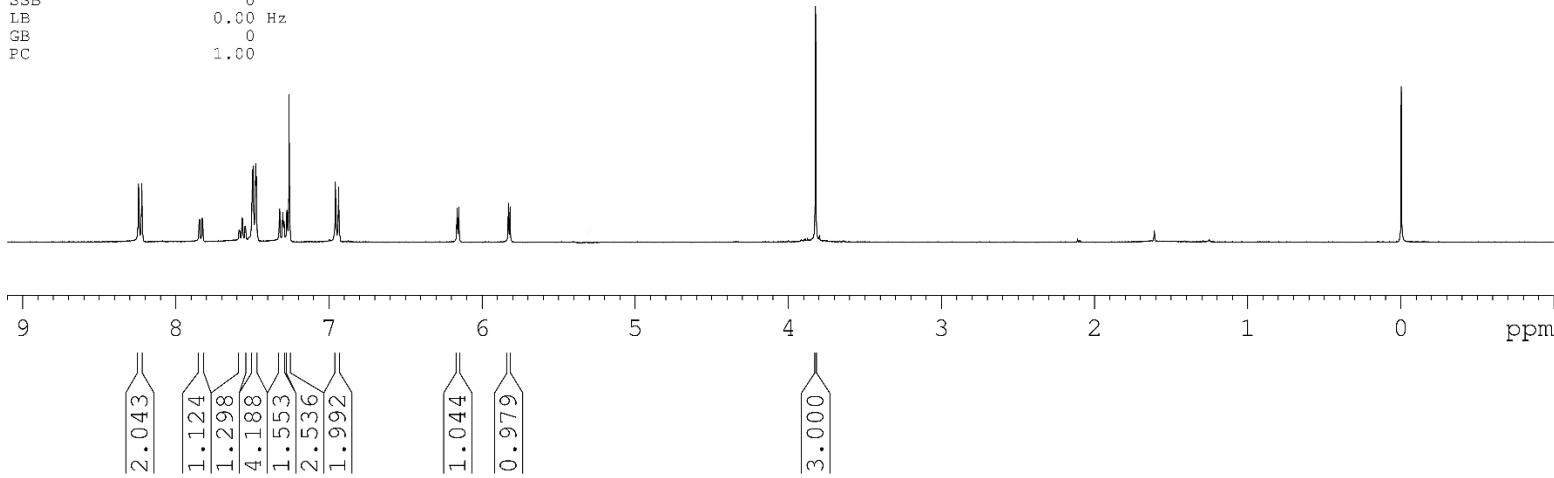
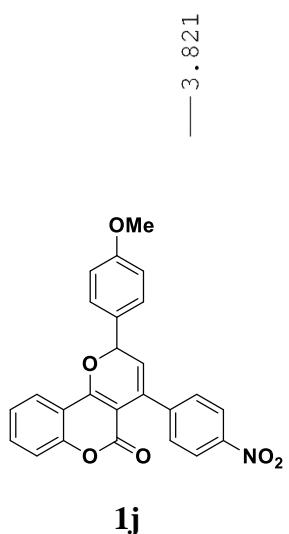


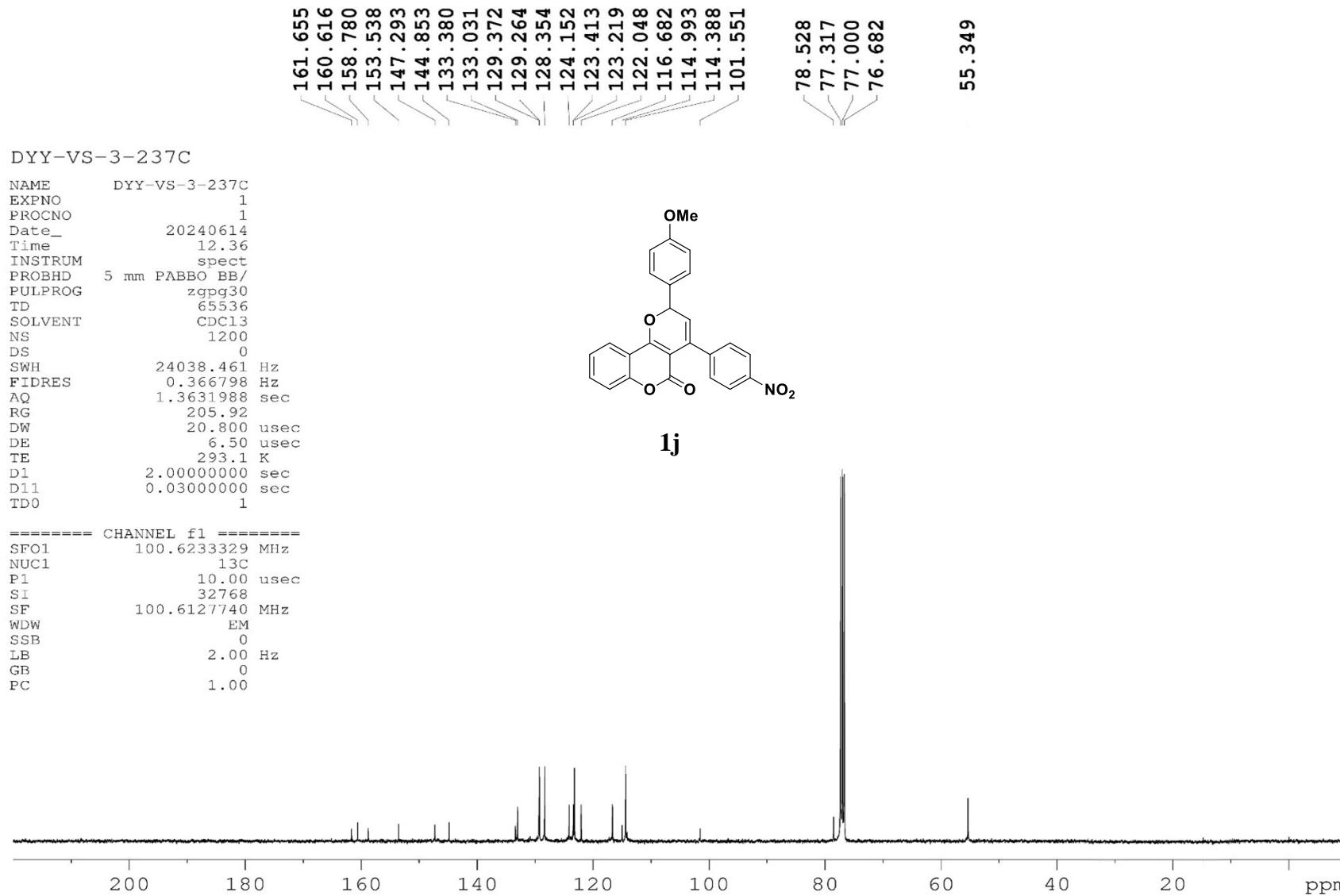


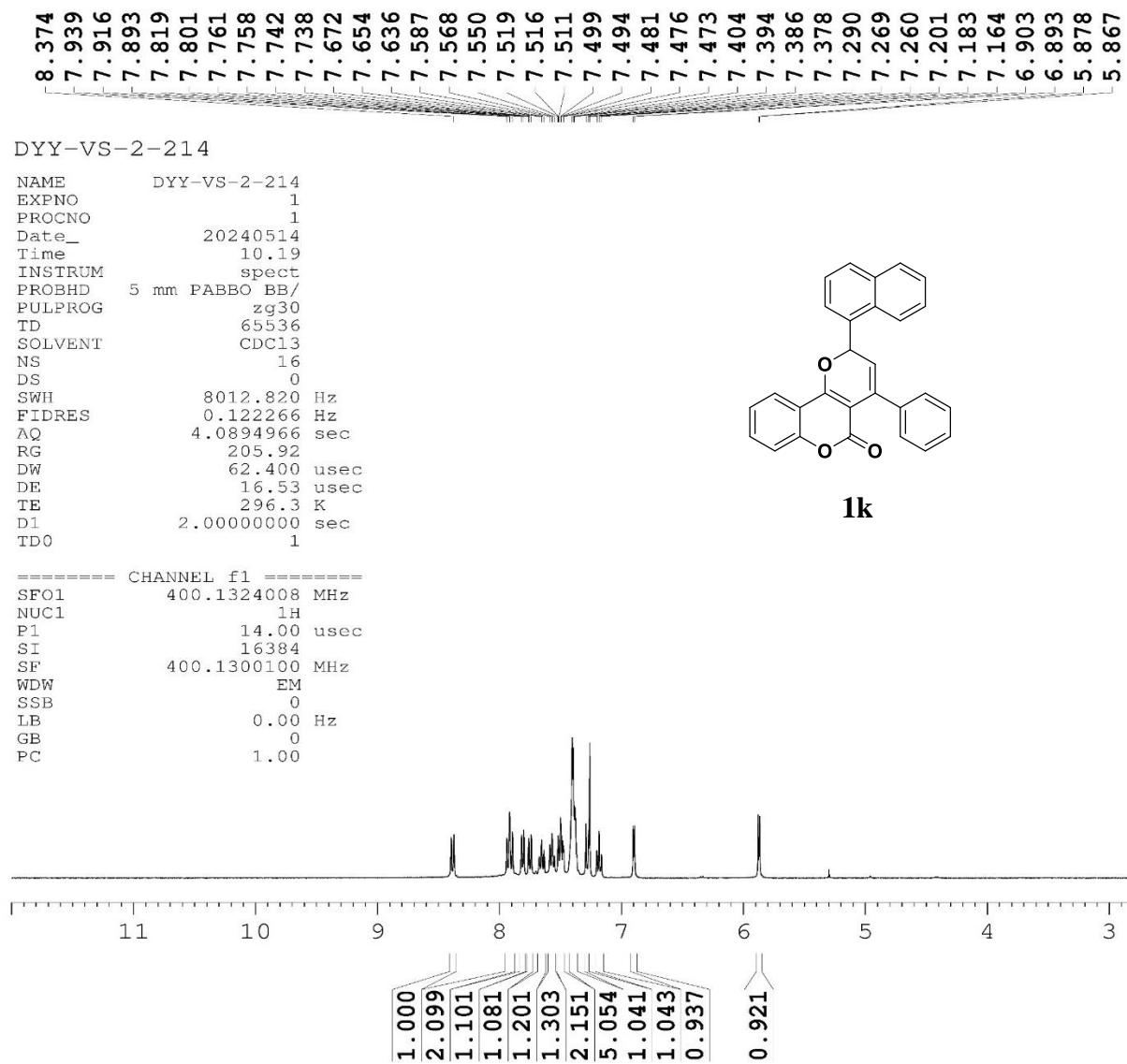
DYY-VS-3-237-0903

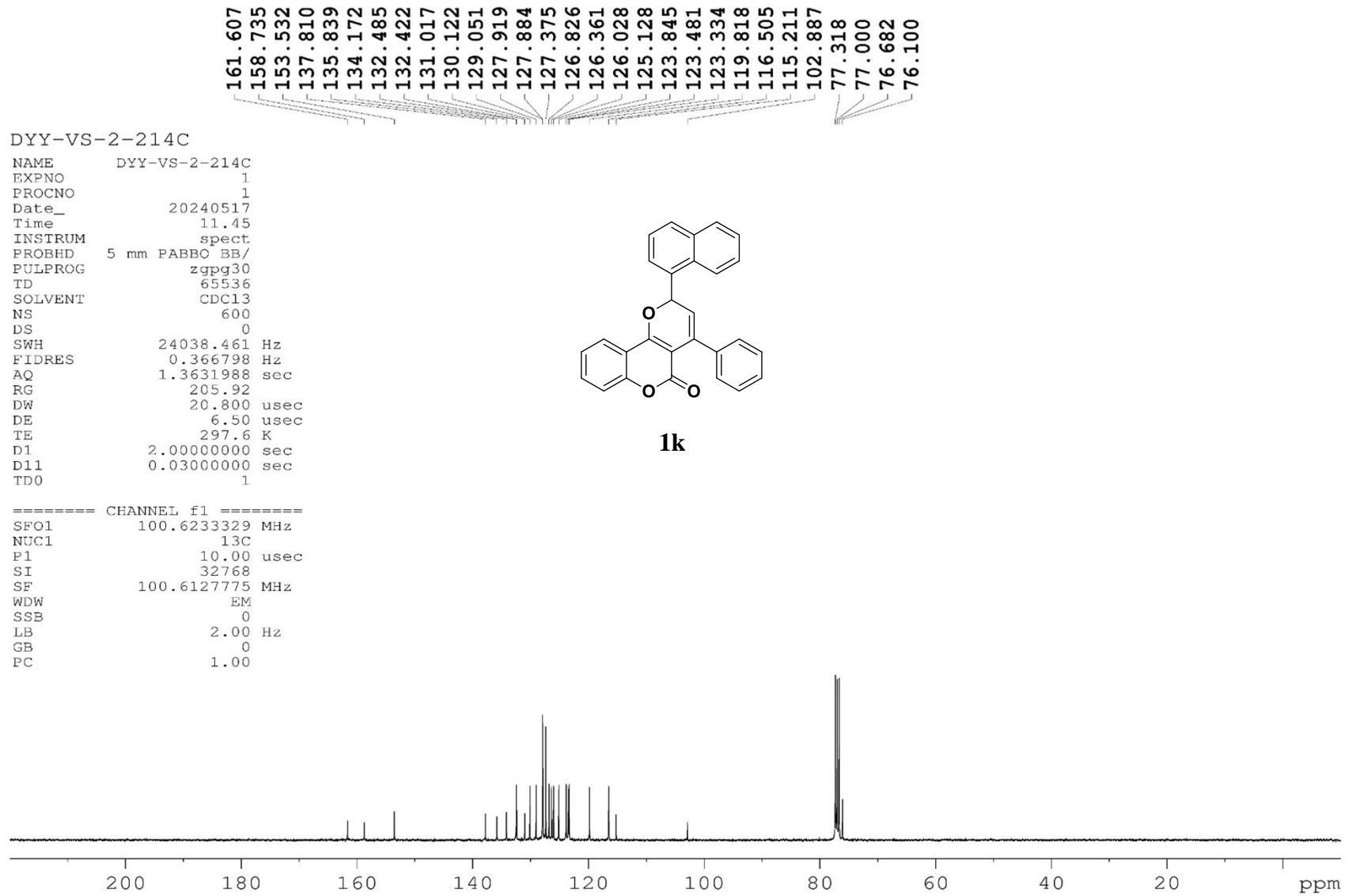
NAME DYY-VS-3-237-0903  
 EXPNO 1  
 PROCHNO 1  
 Date 20240903  
 Time 13.19  
 INSTRUM spect  
 PROBHDL 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.122266 Hz  
 AQ 4.0894966 sec  
 RG 205.92  
 DW 62.400 usec  
 DE 16.53 usec  
 TE 291.6 K  
 D1 2.0000000 sec  
 TDO 1

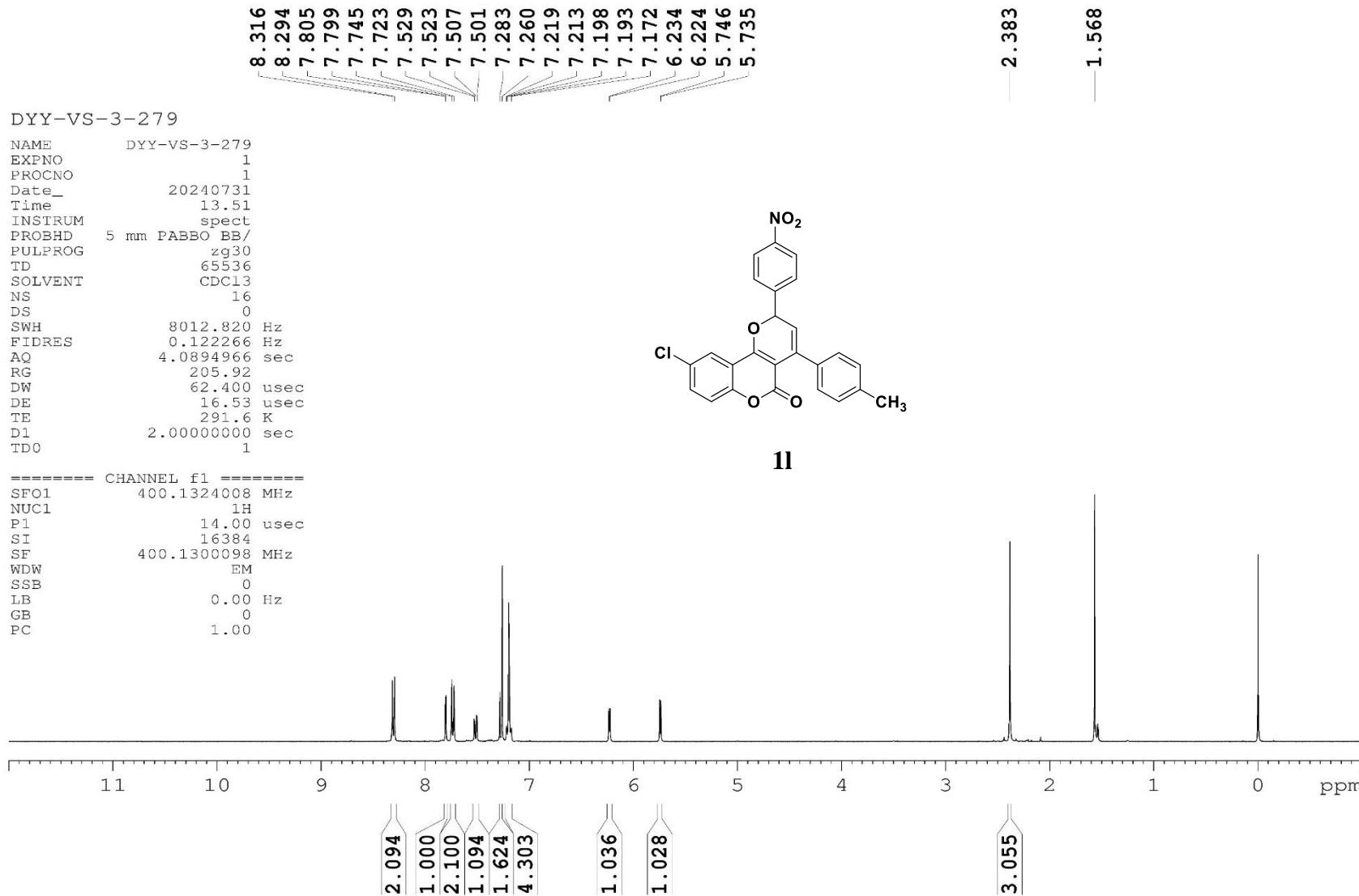
----- CHANNEL f1 -----  
 SFO1 400.1324008 MHz  
 NUC1 1H  
 P1 14.00 usec  
 SI 16384  
 SF 400.1300100 MHz  
 WDW EM  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00







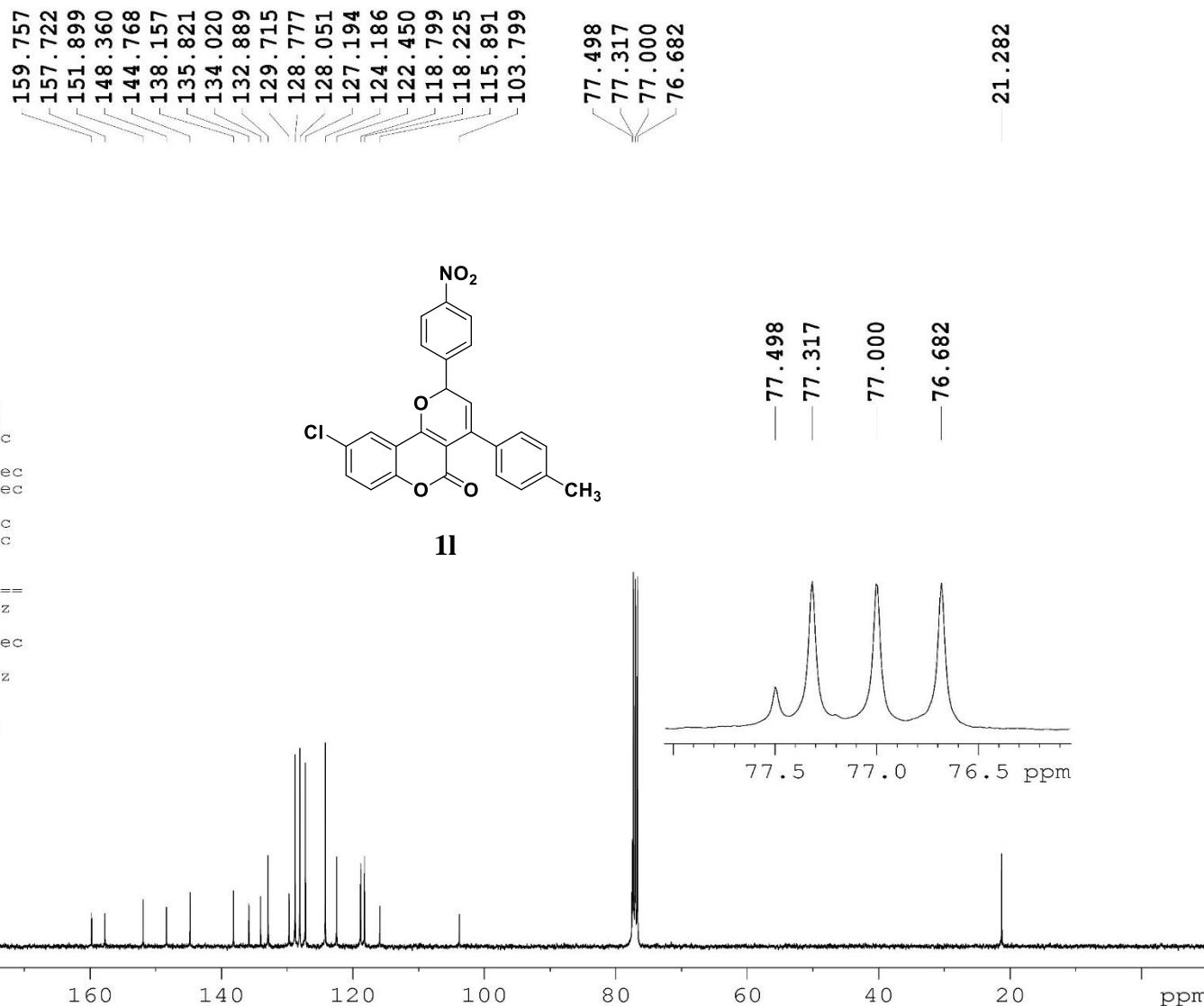


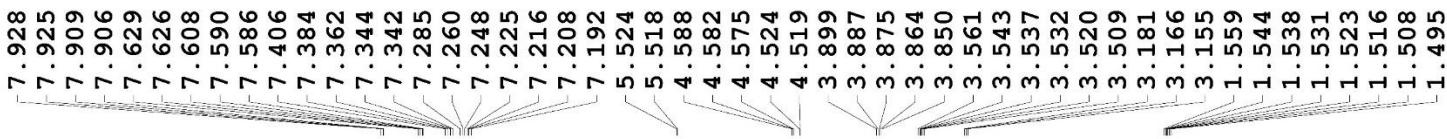


DYY-VS-3-279C

NAME DYY-VS-3-279C  
EXPNO 1  
PROCNO 1  
Date\_ 20240731  
Time 20.29  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 772  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 296.9 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127746 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00





DYY-VS-2-195-1

```

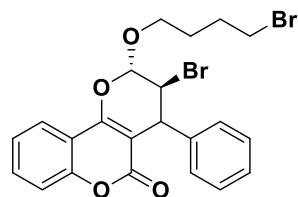
NAME      DYY-VS-2-195-1
EXPNO         1
PROCNO        1
Date_ 20240423
Time   10.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD      65536
SOLVENT  CDCl3
NS       16
DS        0
SWH     8012.820 Hz
FIDRES    0.122266 Hz
AQ      4.0894966 sec
RG      205.92
DW      62.400 usec
DE      16.53 usec
TE      296.0 K
D1      2.00000000 sec
TDO          1

```

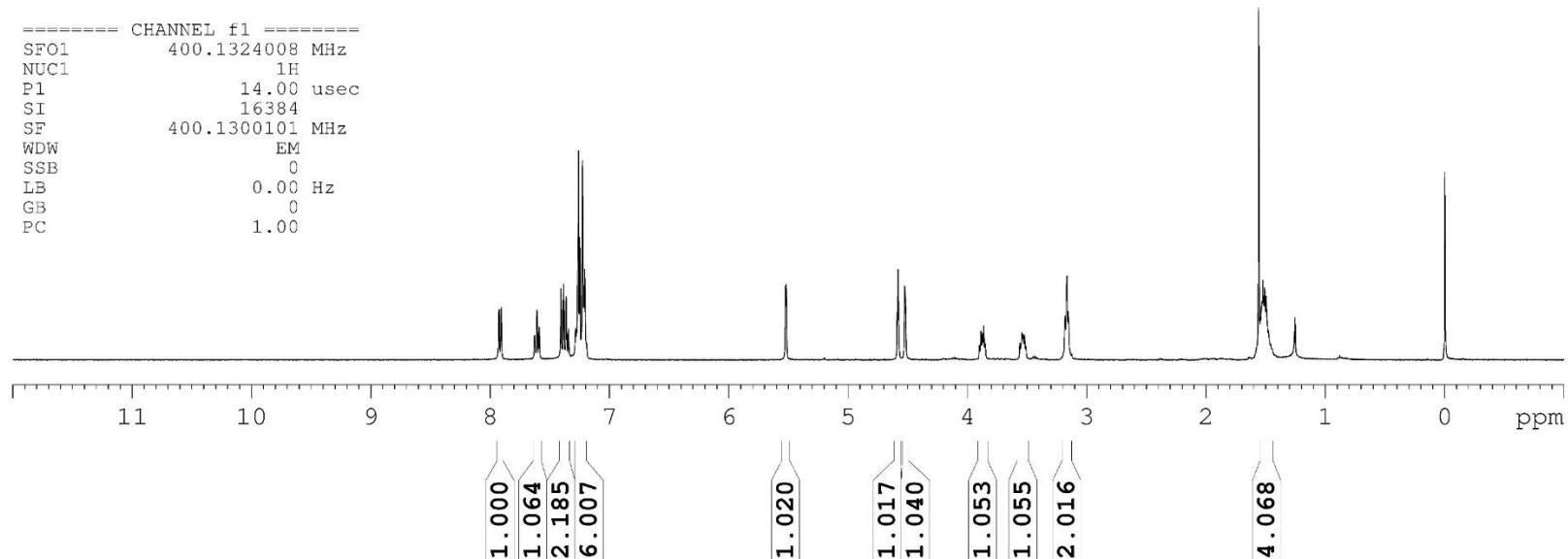
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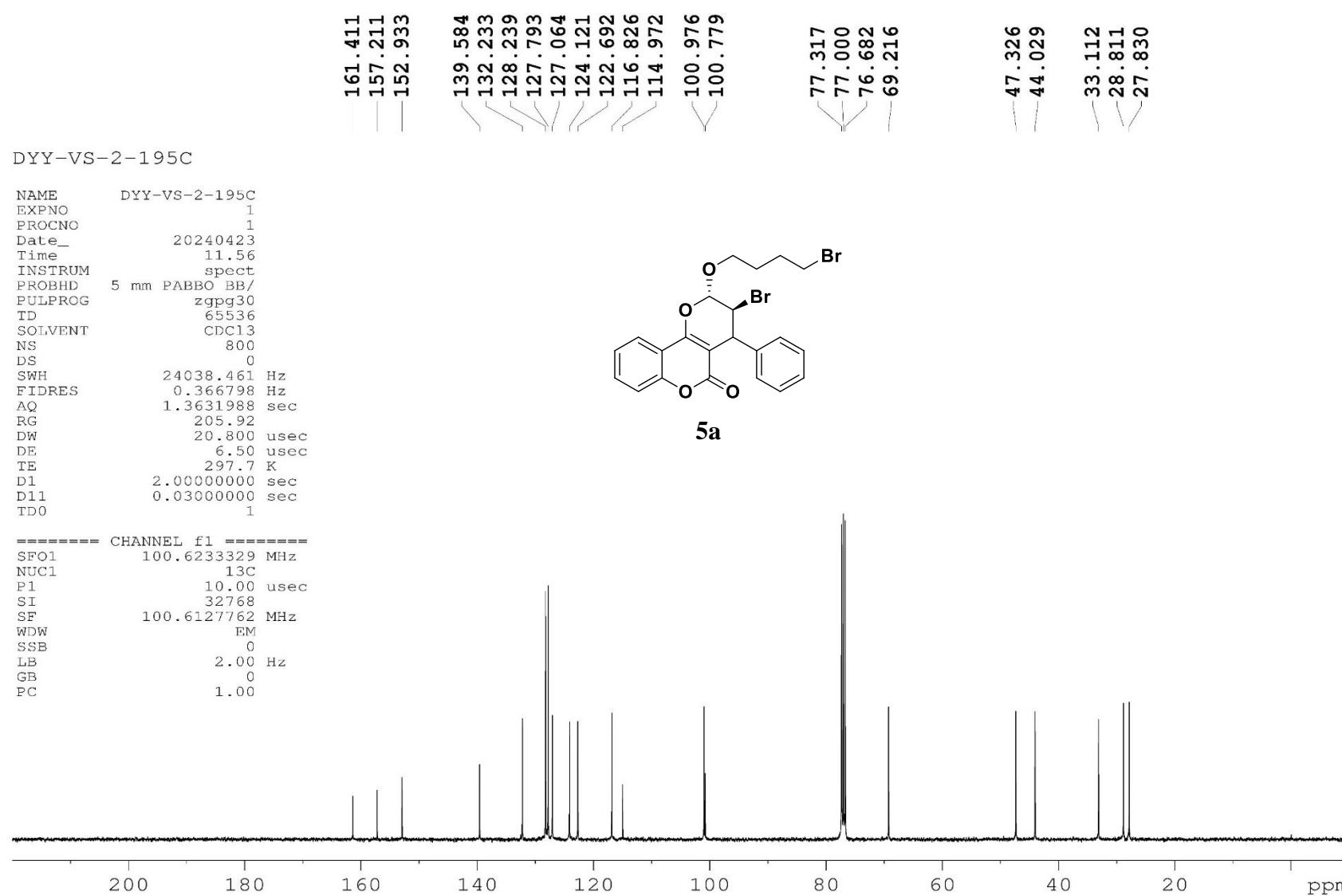
----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1           1H
P1        14.00 usec
SI        16384
SF      400.1300101 MHz
WDW        EM
SSB        0
LB        0.00 Hz
GB        0
PC        1.00

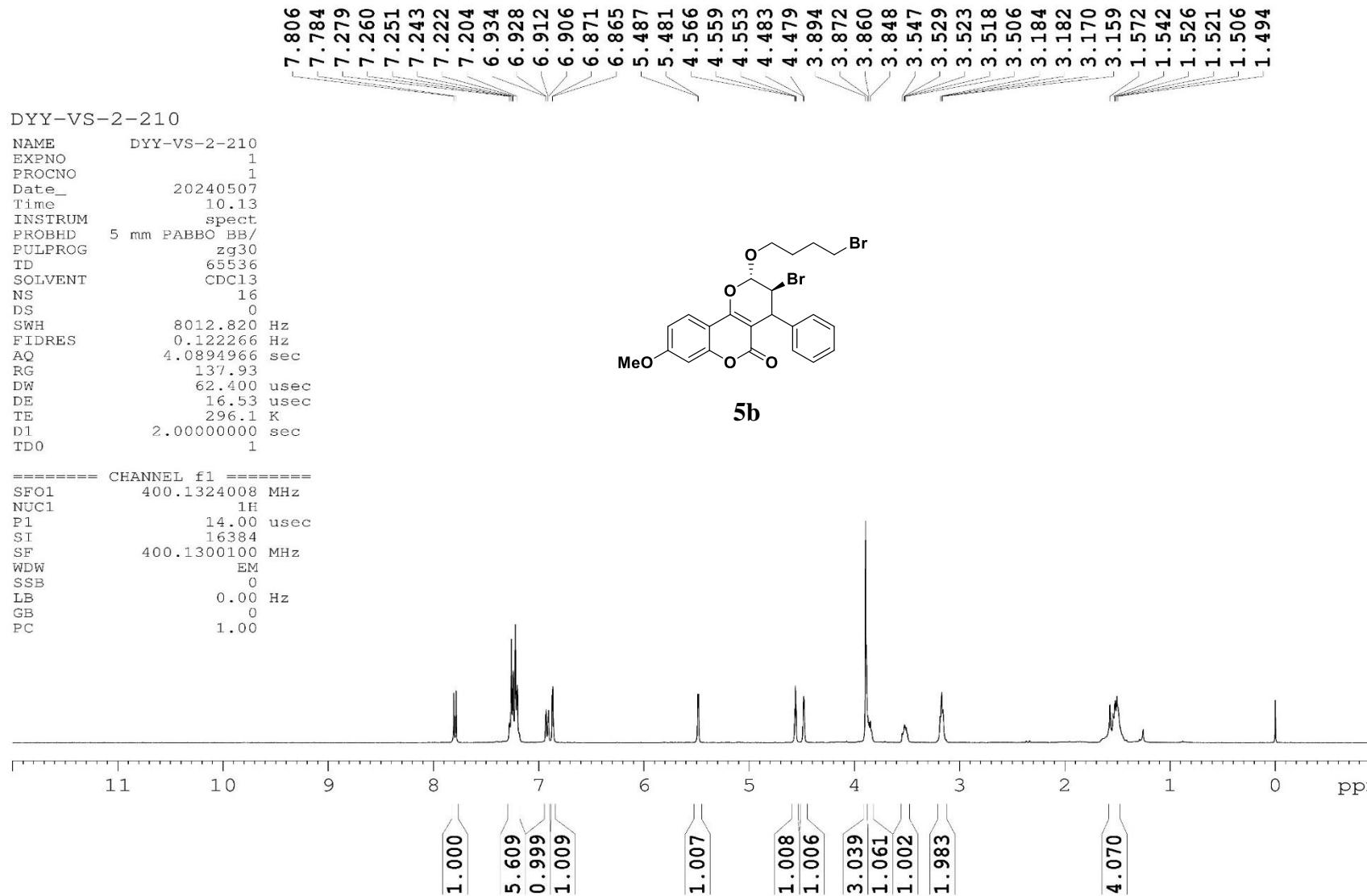
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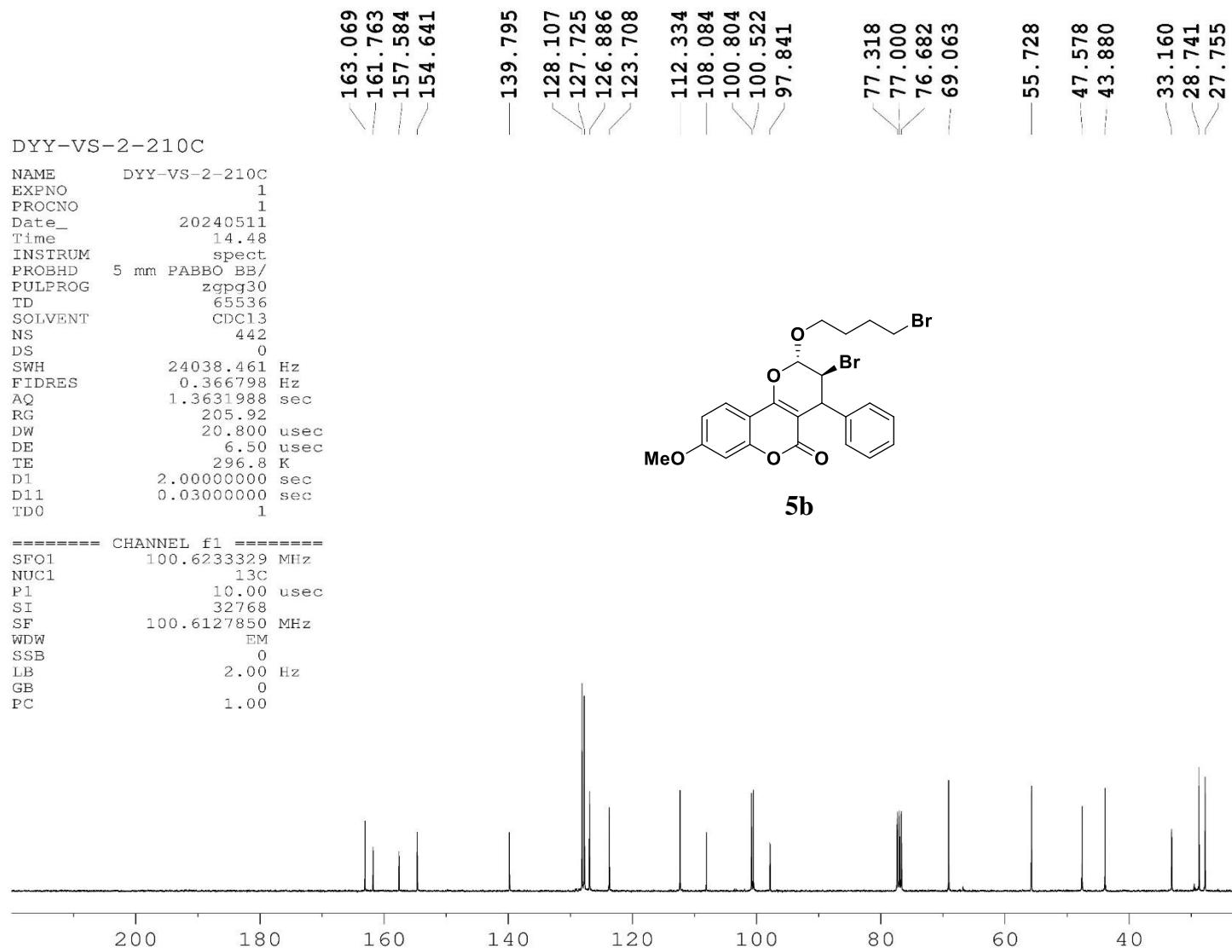


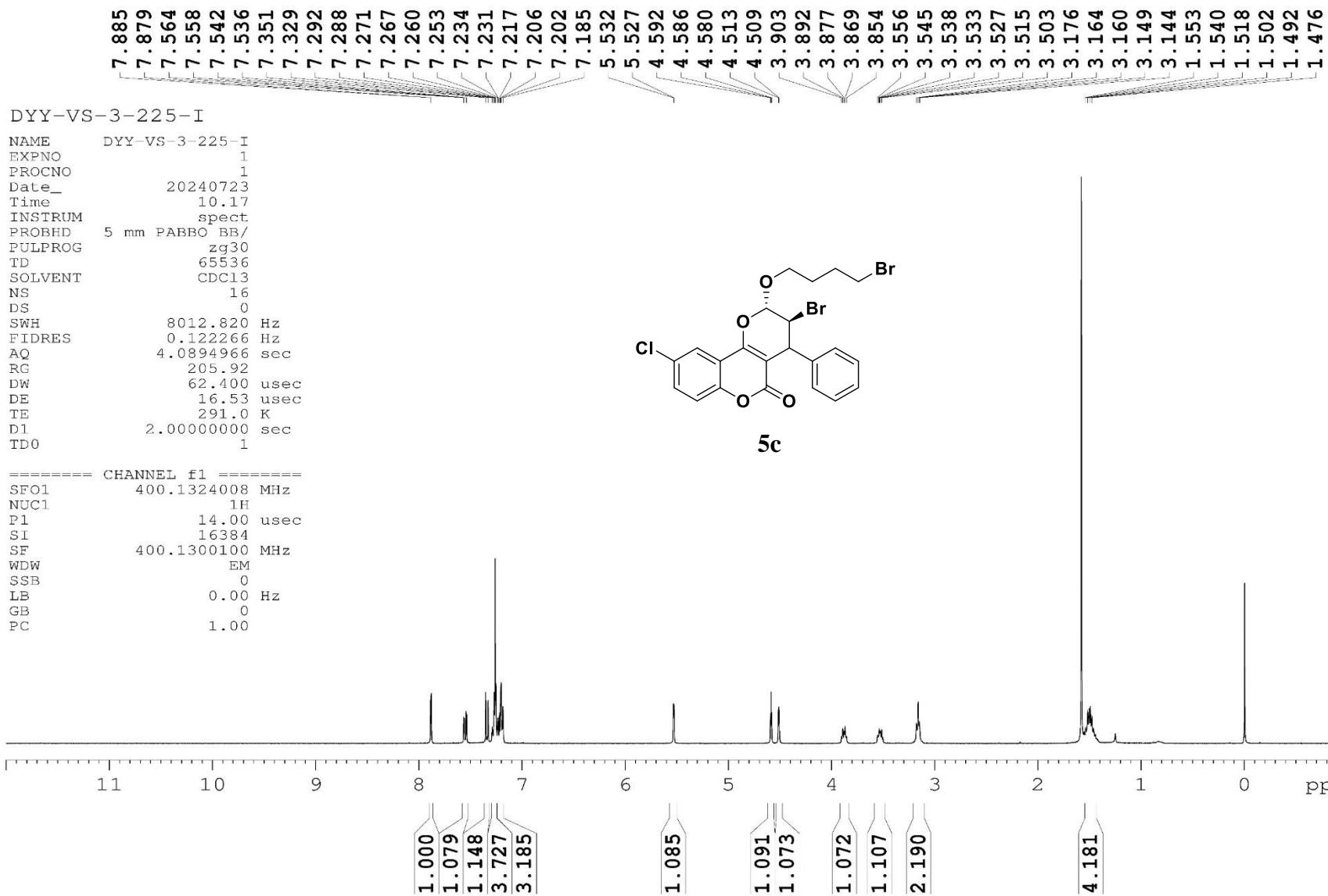
**5a**







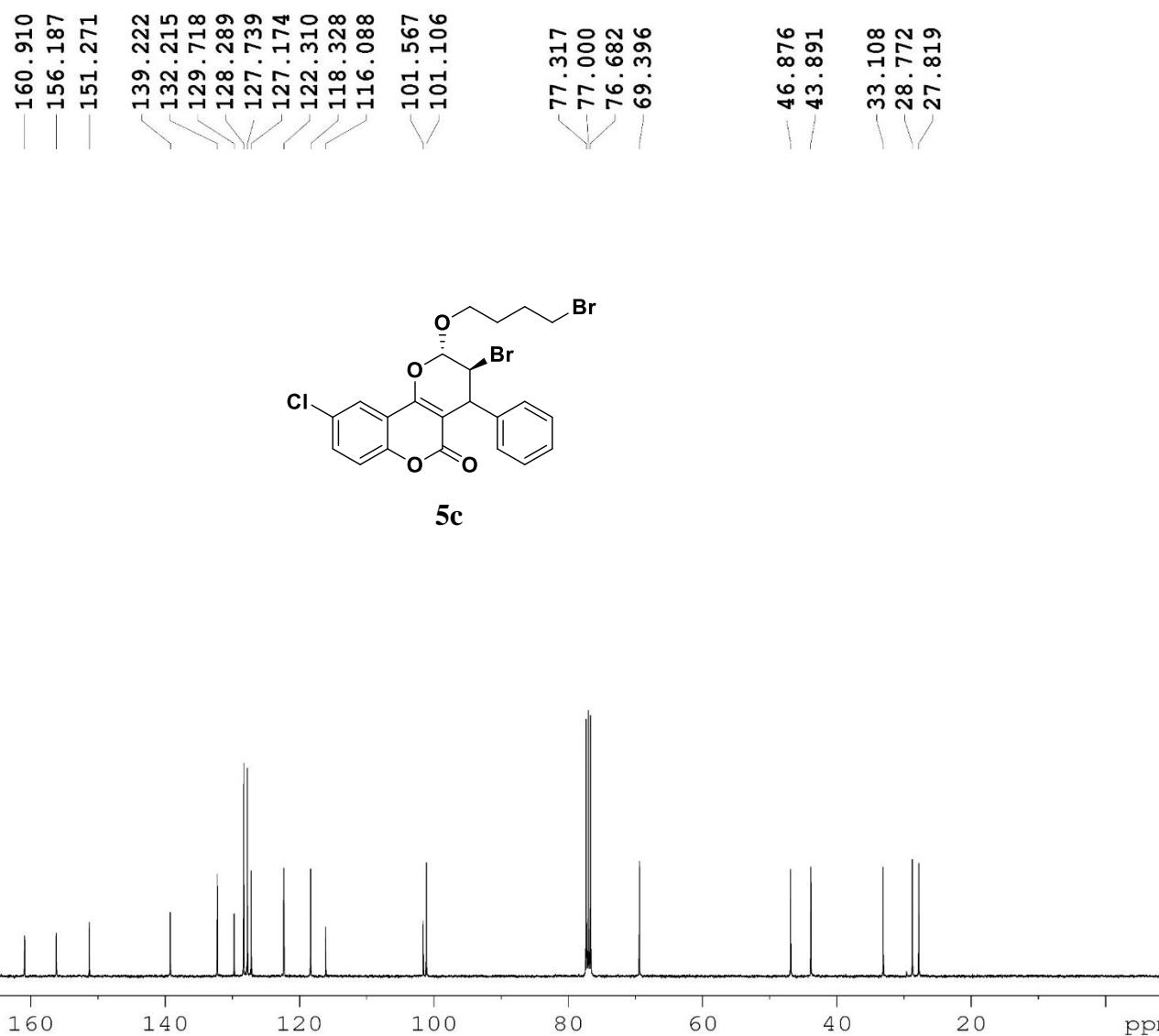




DYY-VS-3-225C

NAME DYY-VS-3-225C  
EXPNO 1  
PROCNO 1  
Date 20240723  
Time 11.12  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 900  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 292.4 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TDO 1

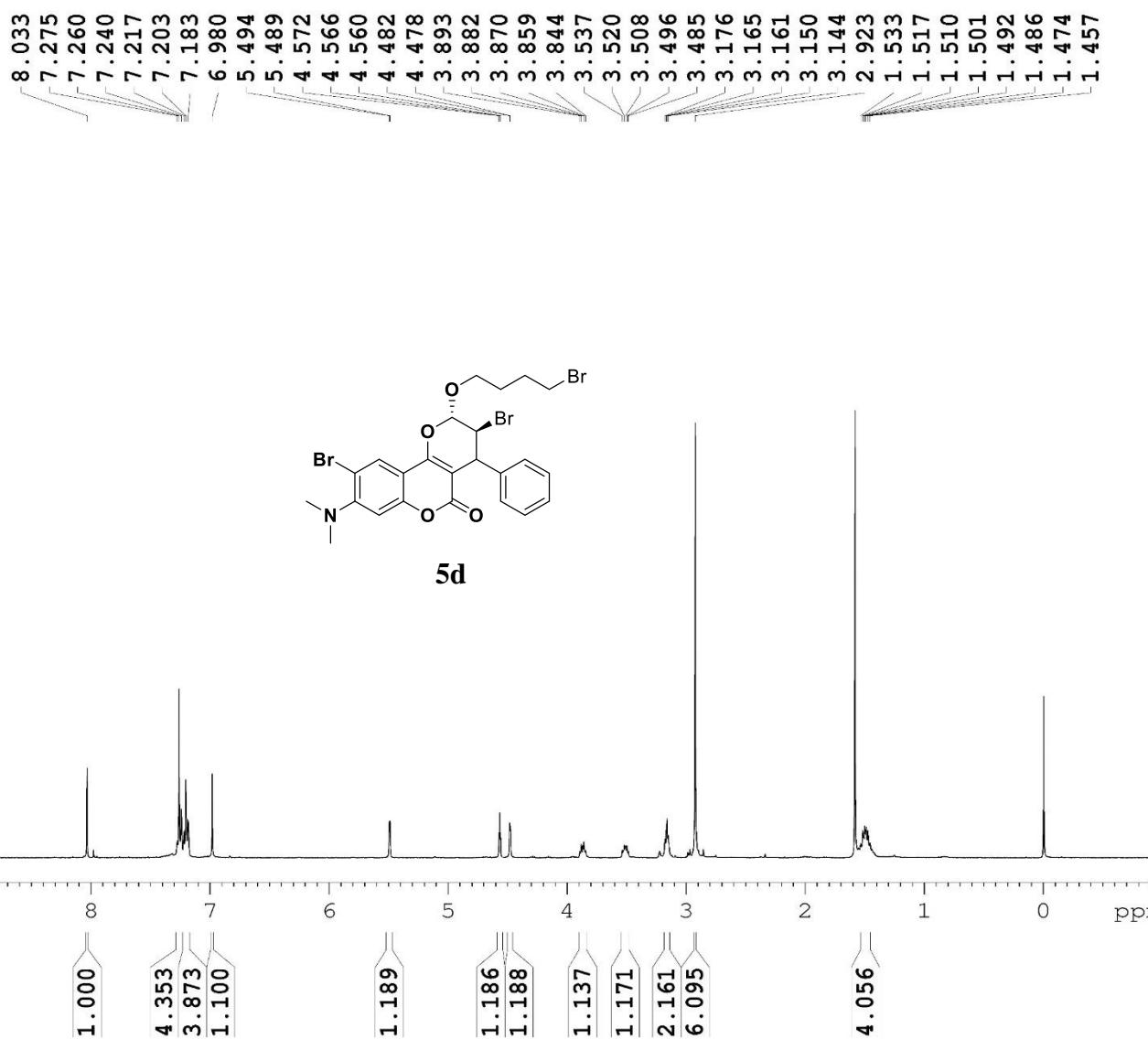
===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127770 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



DYY-VS-3-246-1

NAME DYY-VS-3-246-1  
EXPNO 1  
PROCNO 1  
Date 20240628  
Time 10.22  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 16  
DS 0  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894966 sec  
RG 205.92  
DW 62.400 usec  
DE 16.53 usec  
TE 291.3 K  
D1 2.0000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 400.1324008 MHz  
NUC1 1H  
P1 14.00 usec  
SI 16384  
SF 400.1300099 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00



DYY-VS-3-246-1C

NAME DYY-VS-3-246-1C  
EXPNO 1  
PROCNO 1  
Date\_ 20240628  
Time 11.09  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 199  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 292.8 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127685 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00

161.571  
156.625  
155.462  
153.168

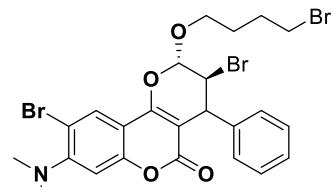
139.707  
128.301  
127.841  
127.747  
127.119

112.717  
110.018  
107.694  
101.035  
98.850

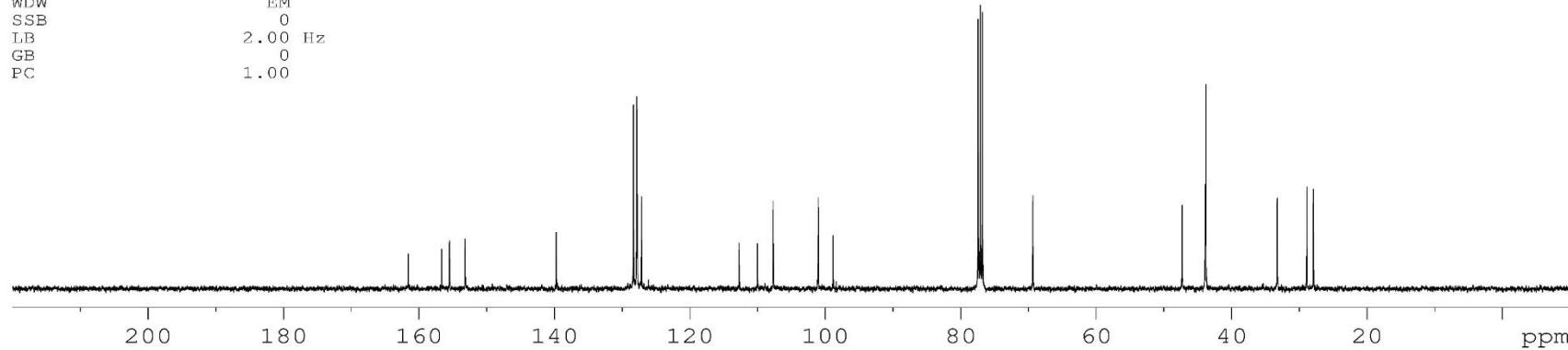
77.424  
77.106  
76.789  
69.351

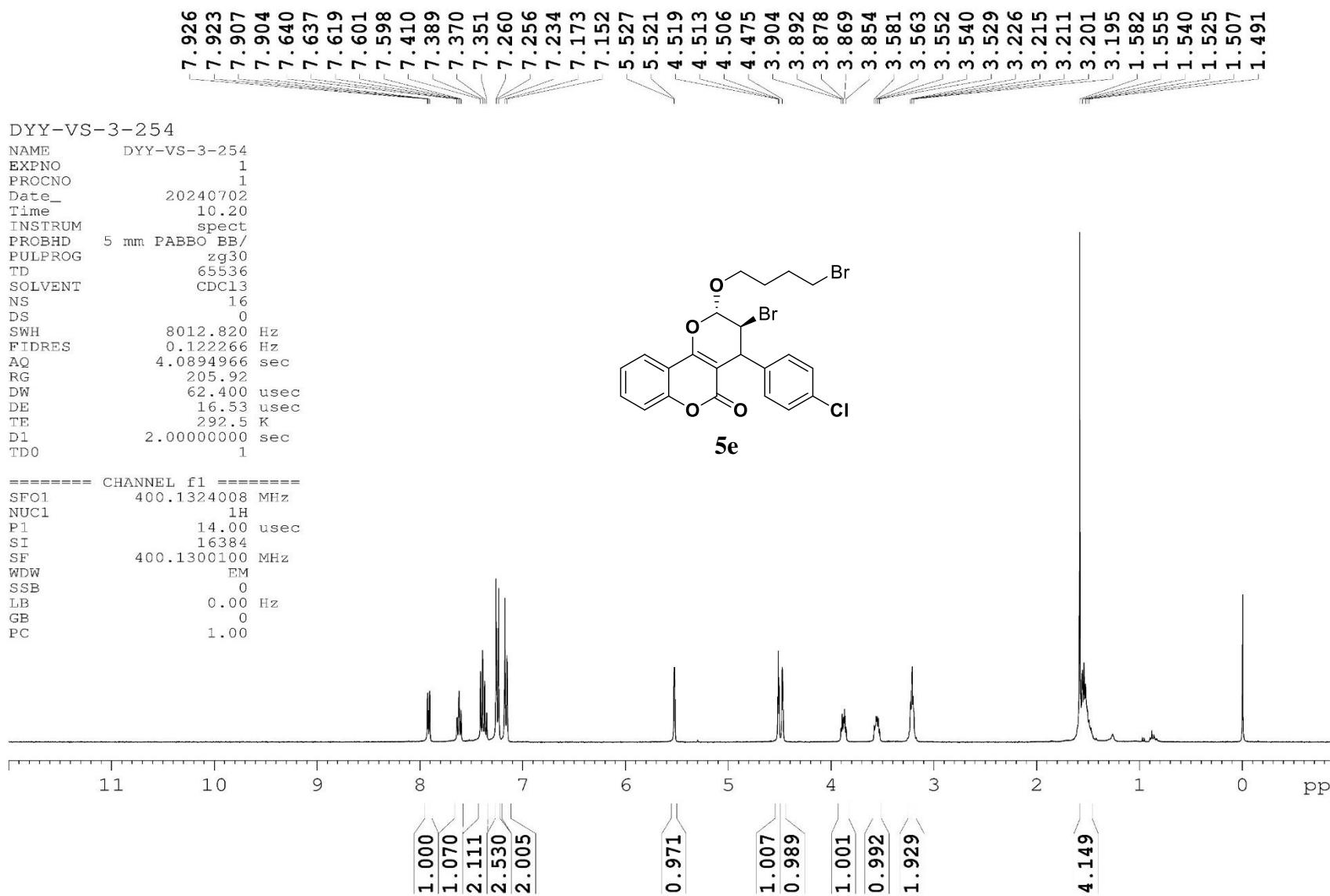
47.311  
43.880  
43.793

33.260  
28.878  
27.919



**5d**

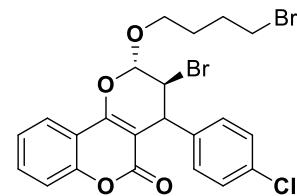
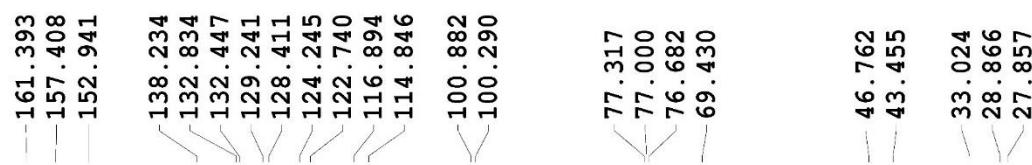




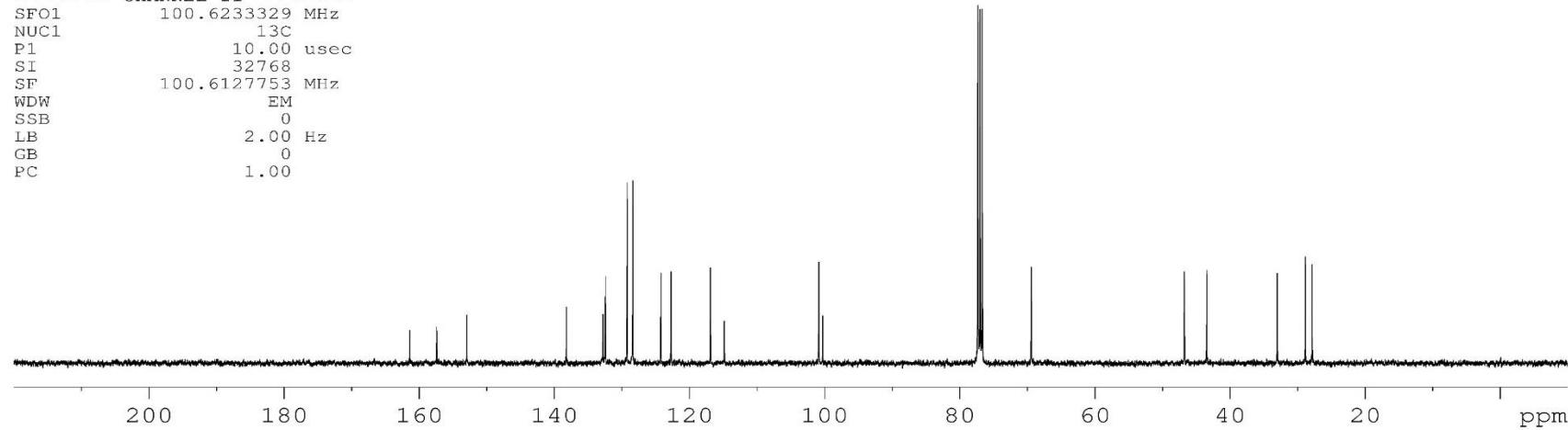
DYY-VS-3-254C

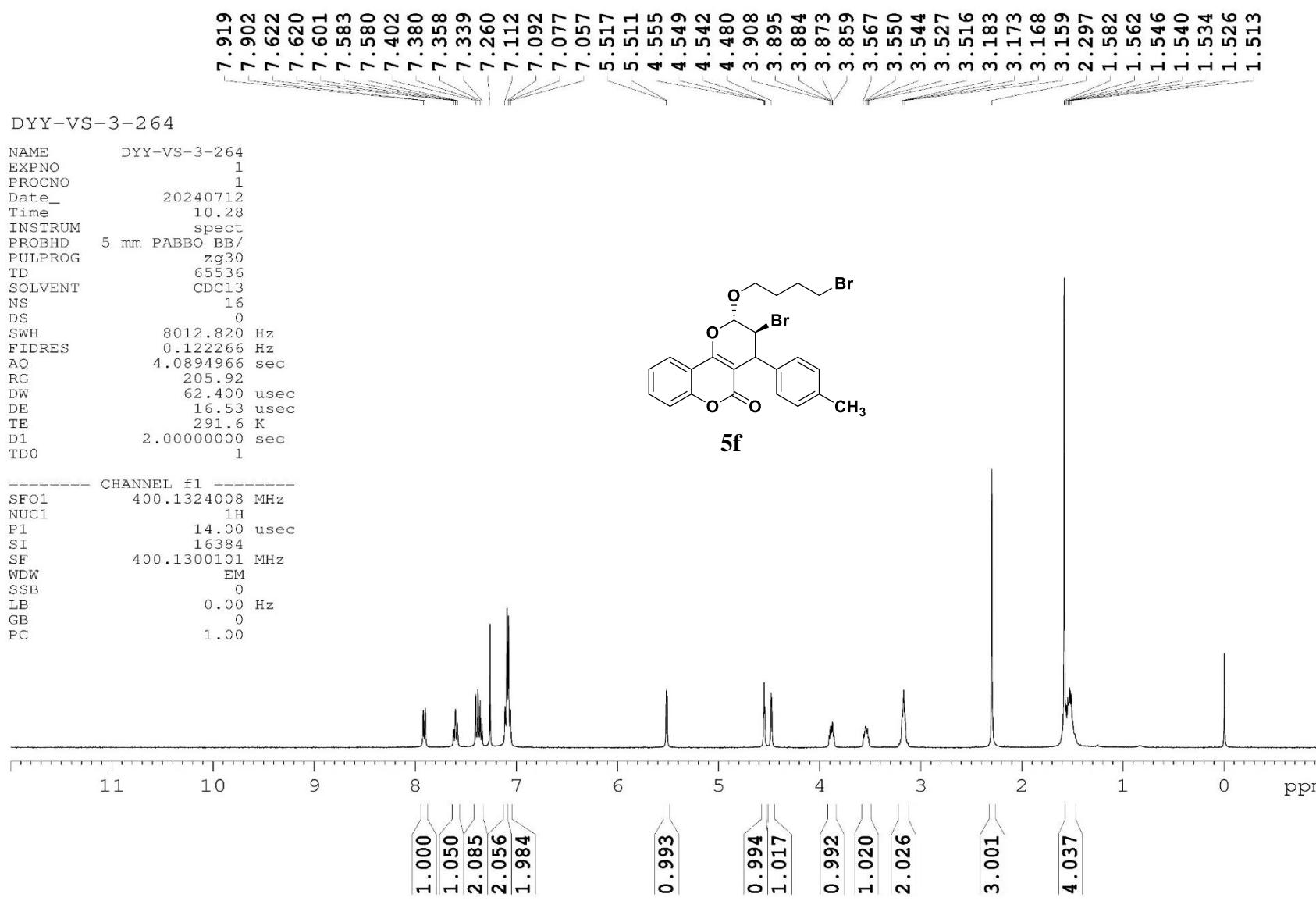
NAME DYY-VS-3-254C  
EXPNO 1  
PROCNO 1  
Date\_ 20240702  
Time 10.44  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 293  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 294.0 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127753 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



**5e**

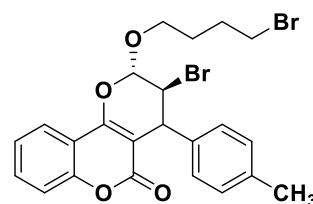
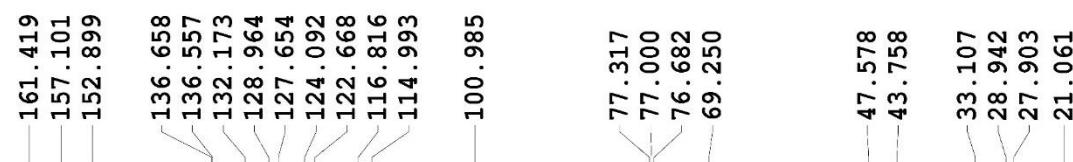




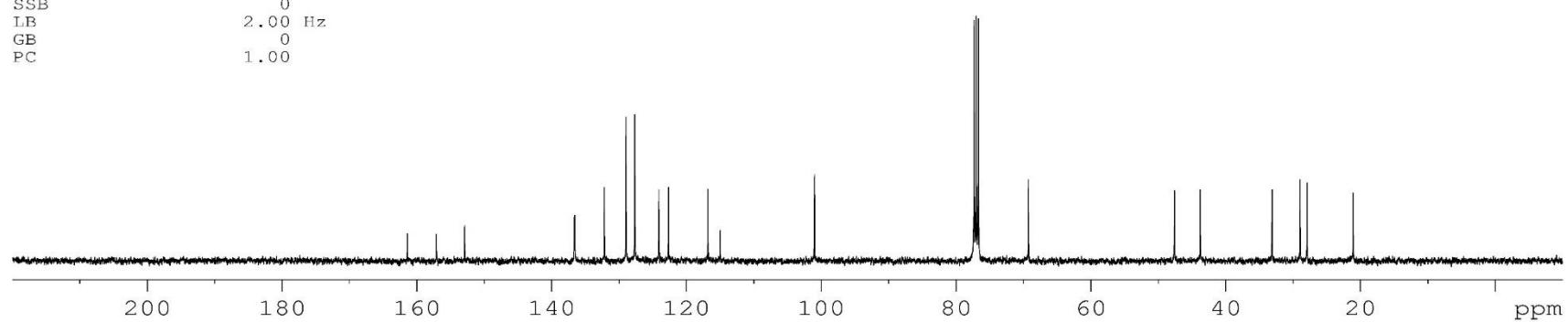
DYY-VS-3-264C

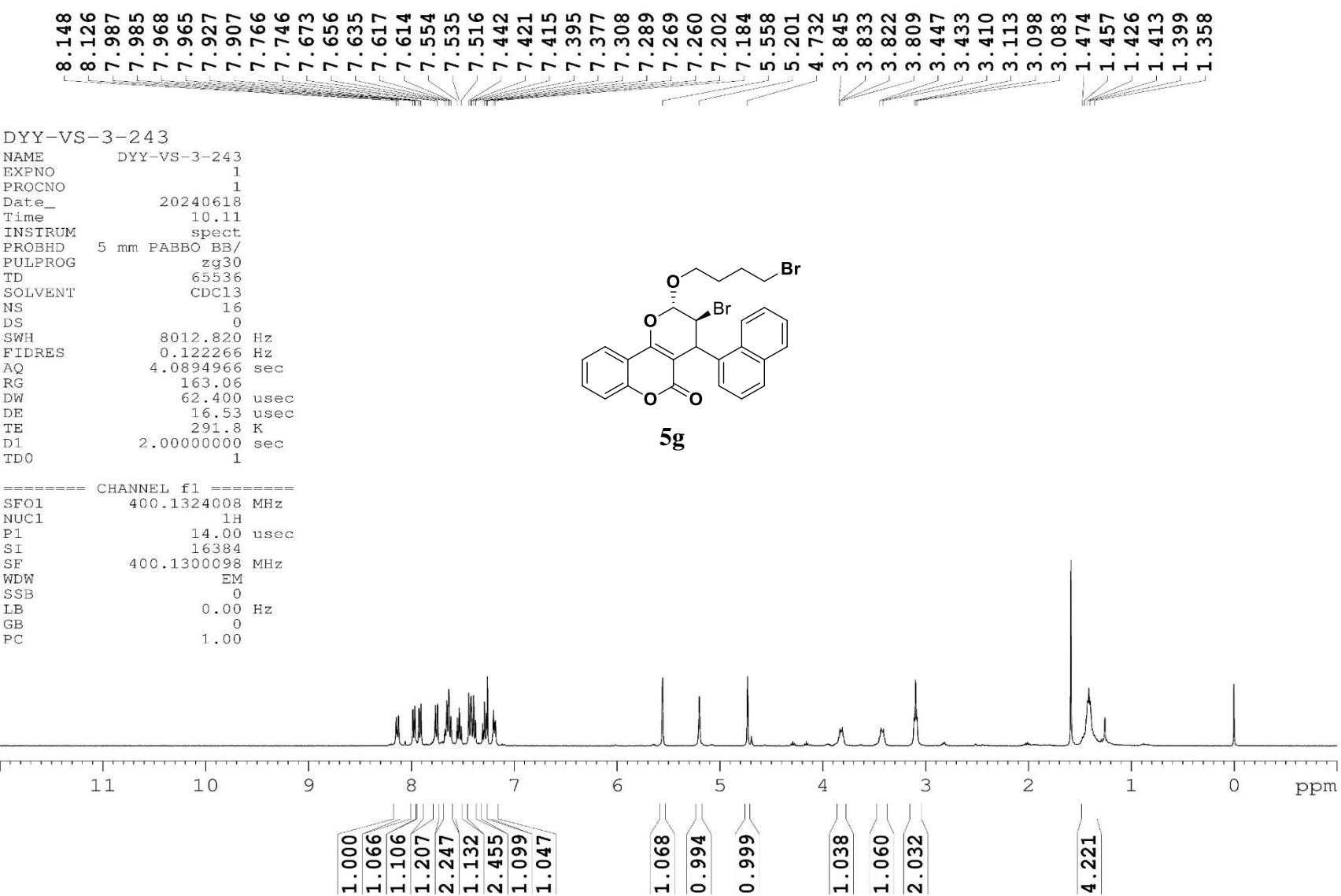
NAME DYY-VS-3-264C  
EXPNO 1  
PROCNO 1  
Date\_ 20240712  
Time 11.30  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 204  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 293.1 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 13C  
P1 10.00 usec  
SI 32768  
SF 100.6127769 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



**5f**

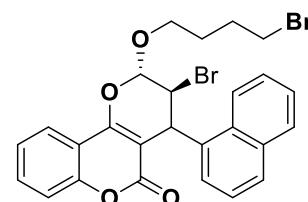
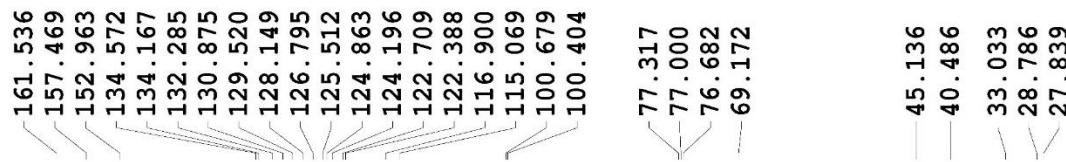




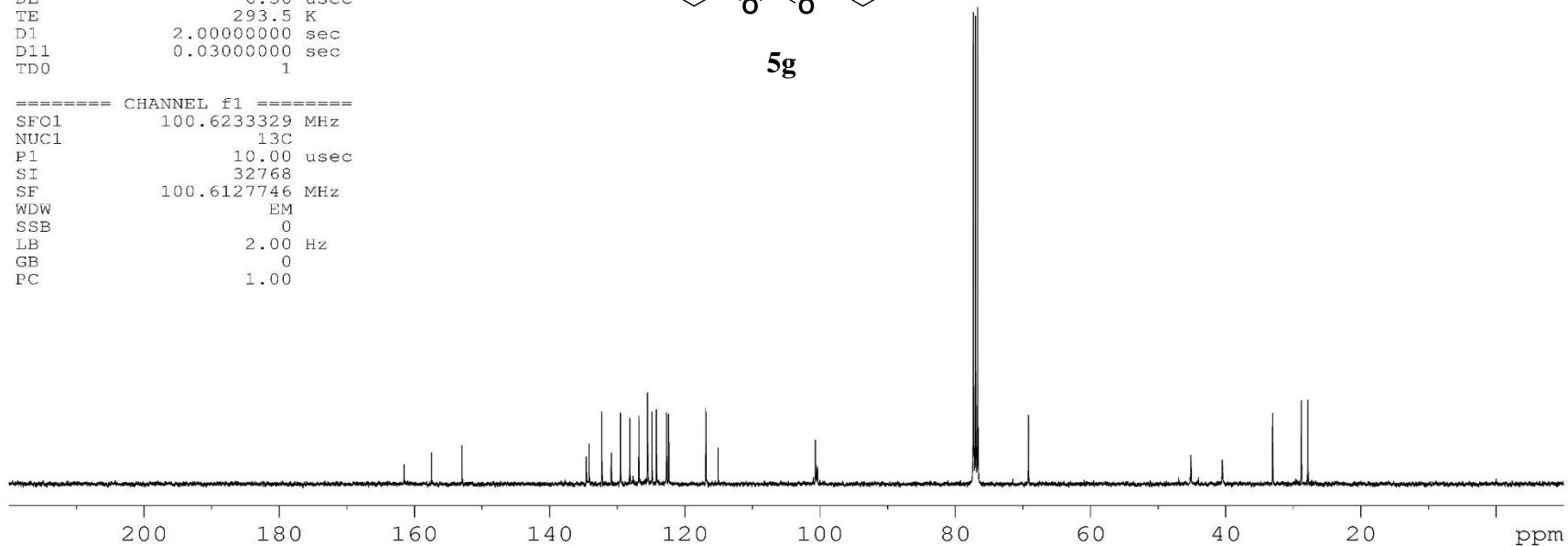
DYY-VS-243C

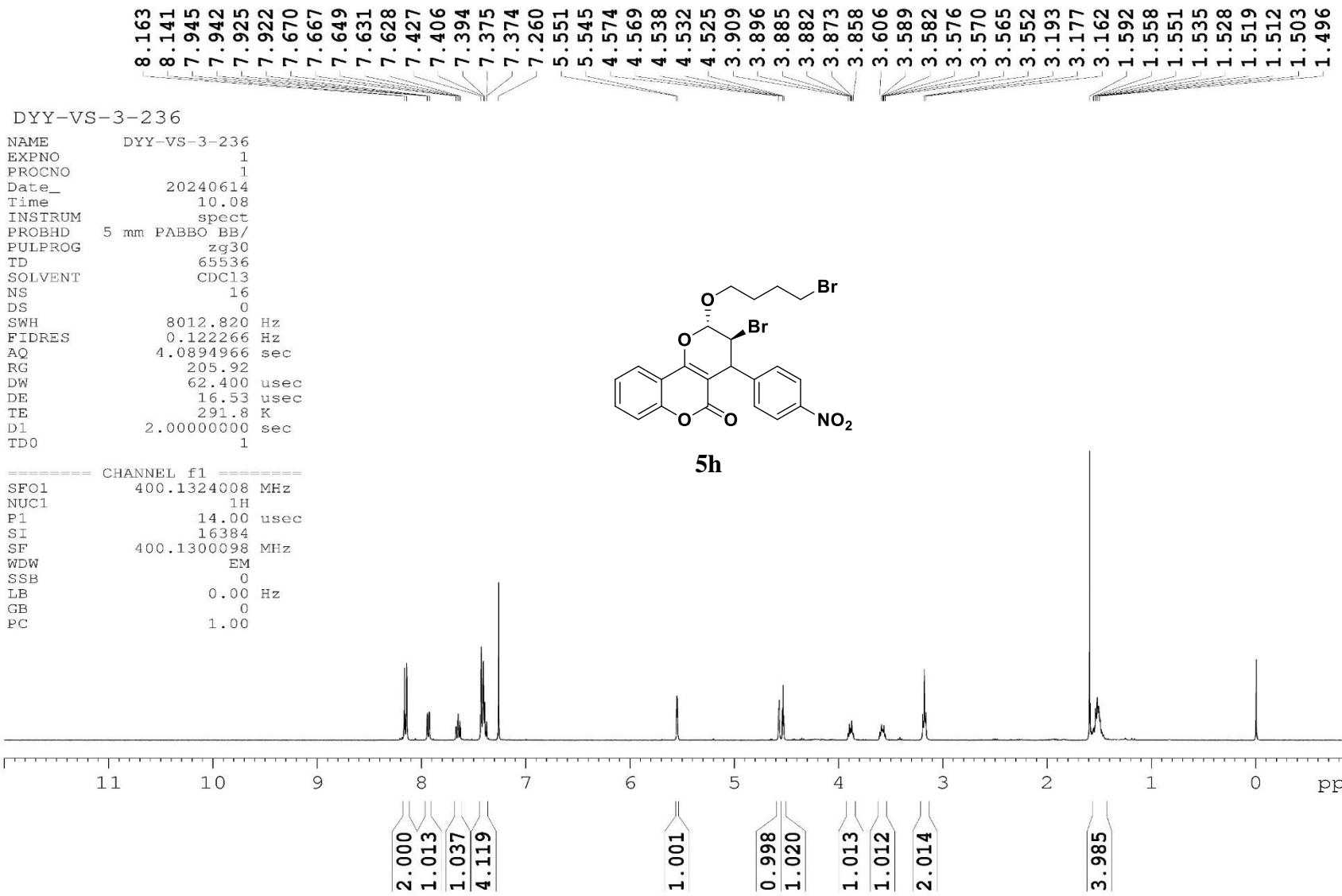
NAME DYY-VS-3-243C  
EXPNO 1  
PROCNO 1  
Date\_ 20240618  
Time 11.40  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 600  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 293.5 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TDO 1

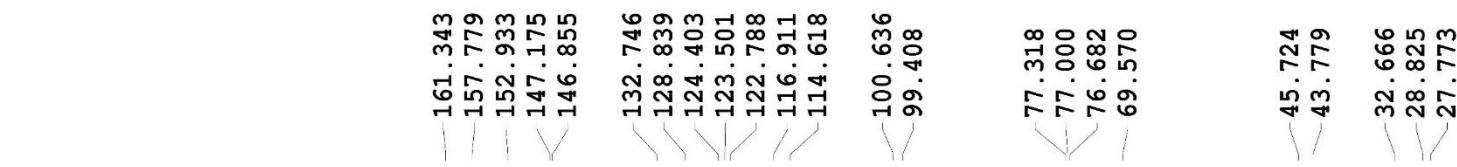
===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 13C  
P1 10.00 usec  
SI 32768  
SF 100.6127746 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



5g



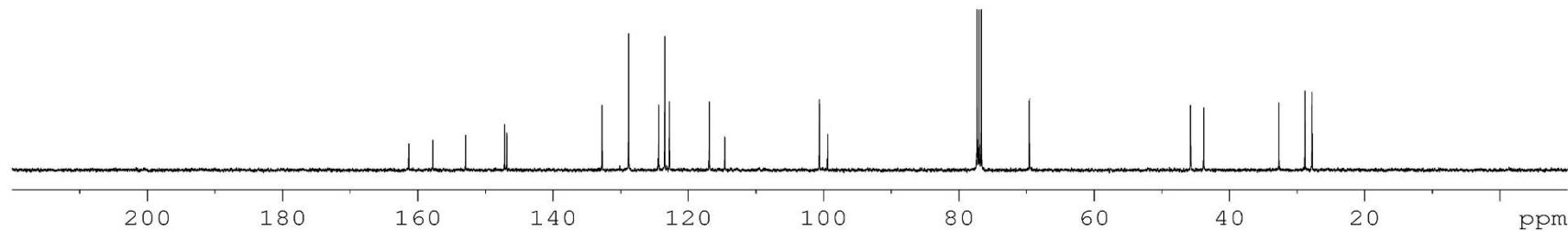




DYY-VS-3-236C

NAME DYY-VS-3-236C  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20240614  
 Time 11.17  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 135  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631988 sec  
 RG 205.92  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 292.3 K  
 D1 2.0000000 sec  
 D11 0.03000000 sec  
 TDO 1

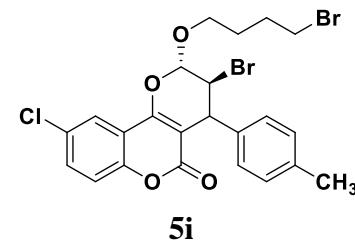
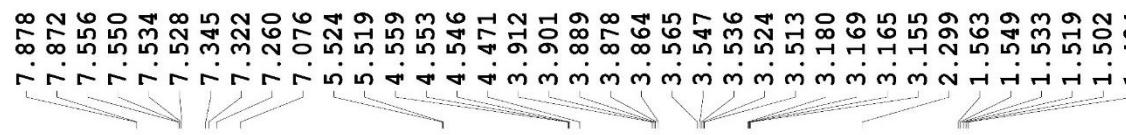
===== CHANNEL f1 =====  
 SFO1 100.6233329 MHz  
 NUC1 13C  
 P1 10.00 usec  
 SI 32768  
 SF 100.6127803 MHz  
 WDW EM  
 SSB 0  
 LB 2.00 Hz  
 GB 0  
 PC 1.00



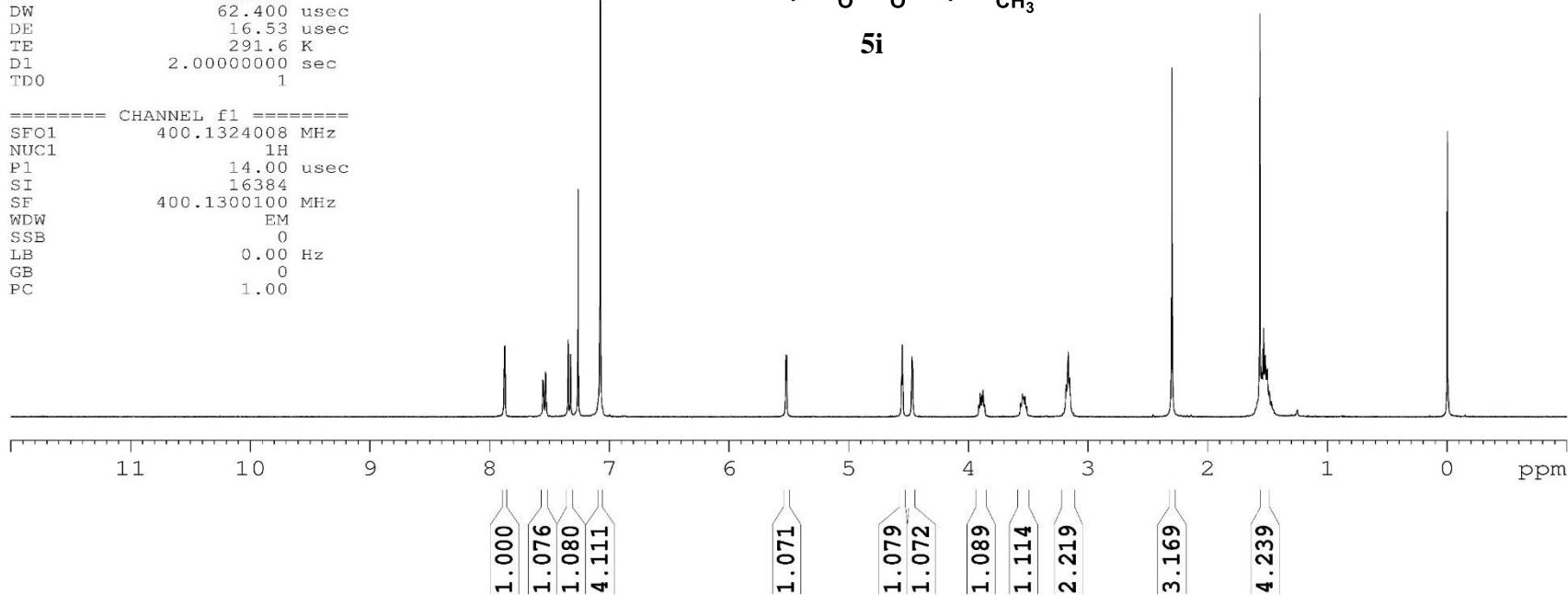
DYY-VS-3-278

NAME DYY-VS-3-278  
EXPNO 1  
PROCNO 1  
Date 20240731  
Time 13.46  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 8012.820 Hz  
FIDRES 0.122266 Hz  
AQ 4.0894966 sec  
RG 205.92  
DW 62.400 usec  
DE 16.53 usec  
TE 291.6 K  
D1 2.0000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 400.1324008 MHz  
NUC1 1H  
P1 14.00 usec  
SI 16384  
SF 400.1300100 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00



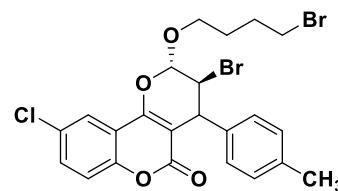
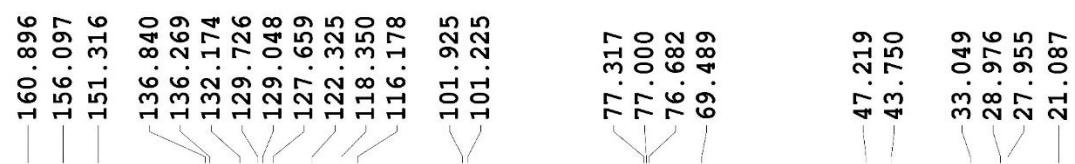
5i



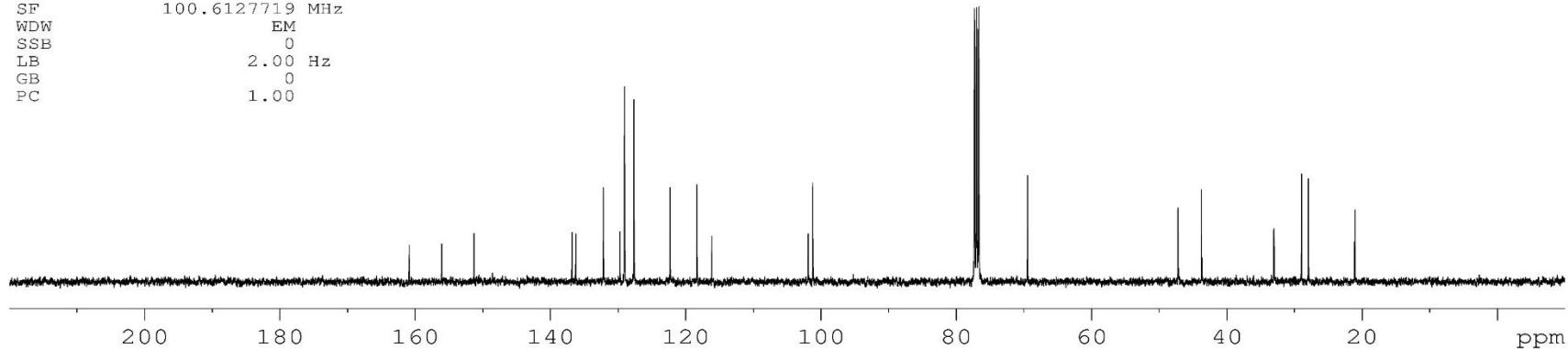
DYY-VS-3-278C

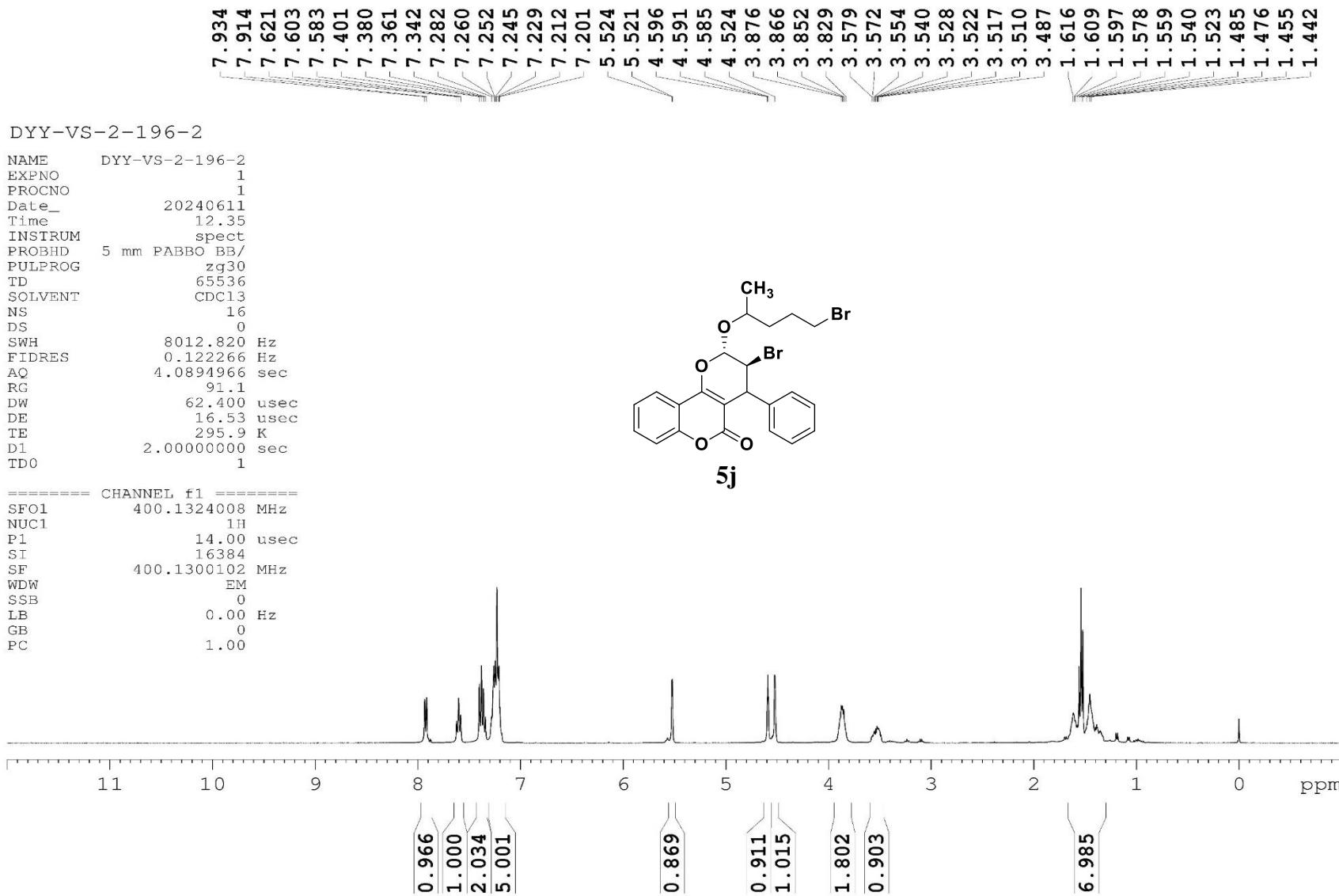
NAME DYY-VS-3-278C  
EXPNO 1  
PROCNO 1  
Date\_ 20240731  
Time 19.42  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 200  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 296.8 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127719 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



**5i**

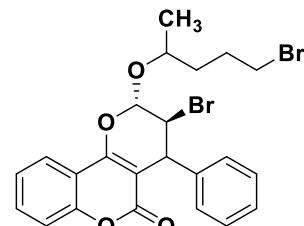
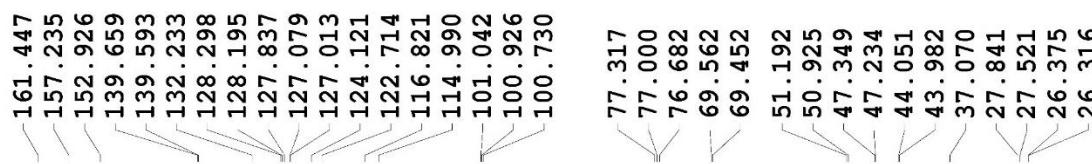




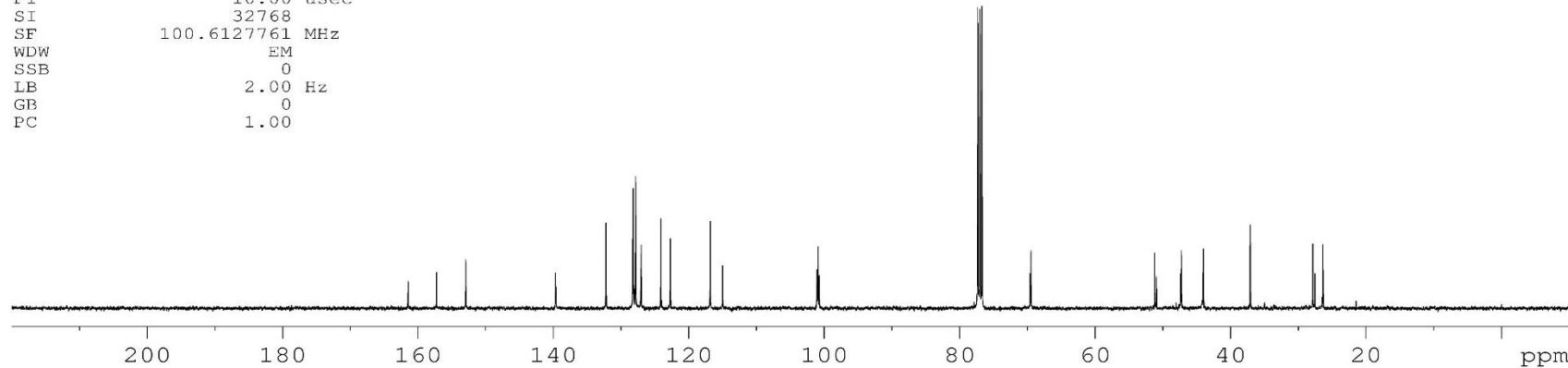
DYY-VS-2-196C

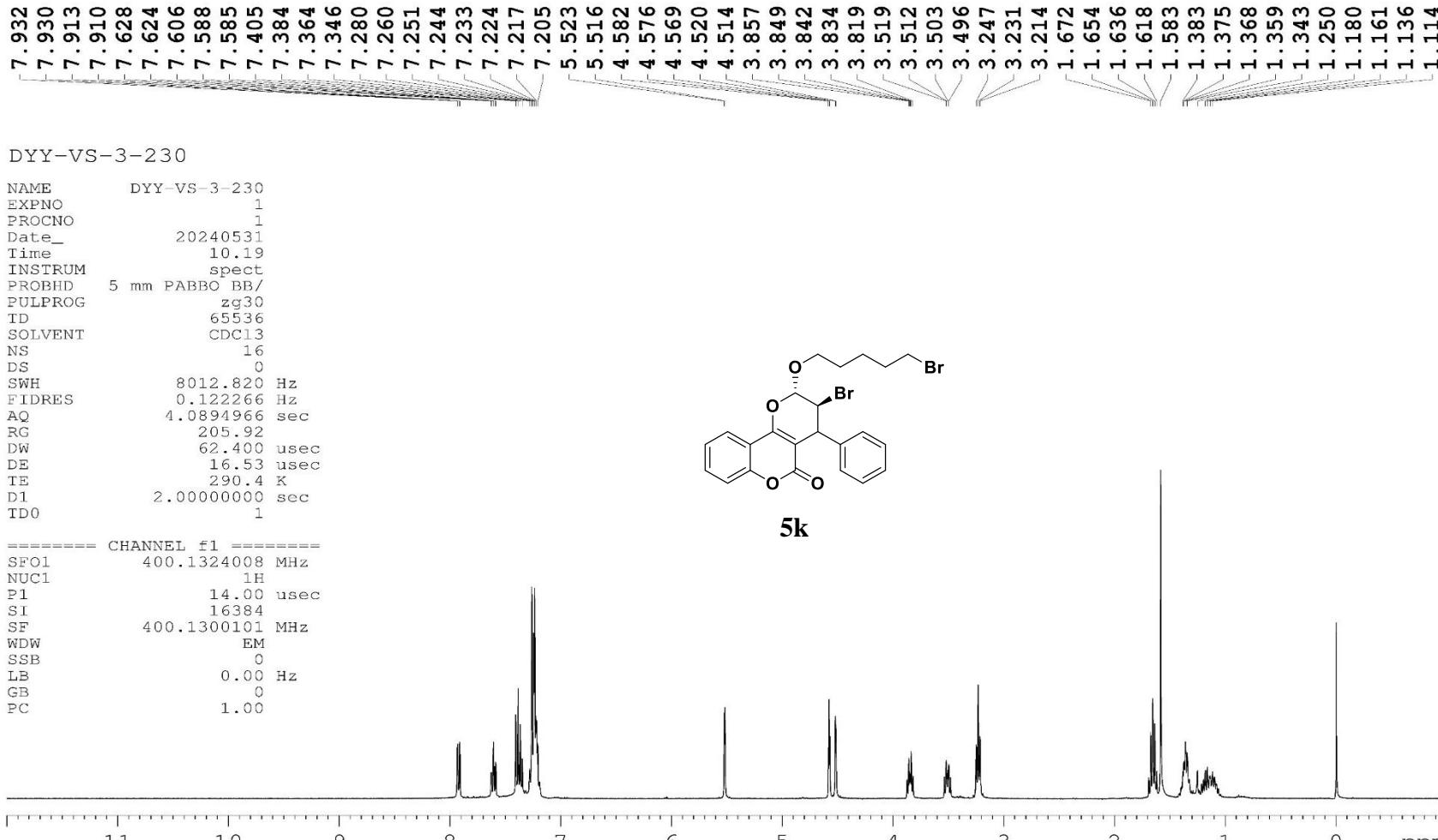
NAME DYY-VS-2-196C  
EXPNO 1  
PROCNO 1  
Date\_ 20240611  
Time 12.32  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 500  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 296.9 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TDO 1

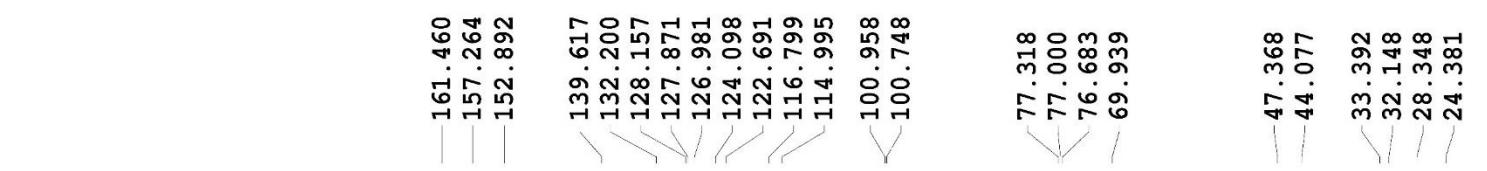
===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127761 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



5j



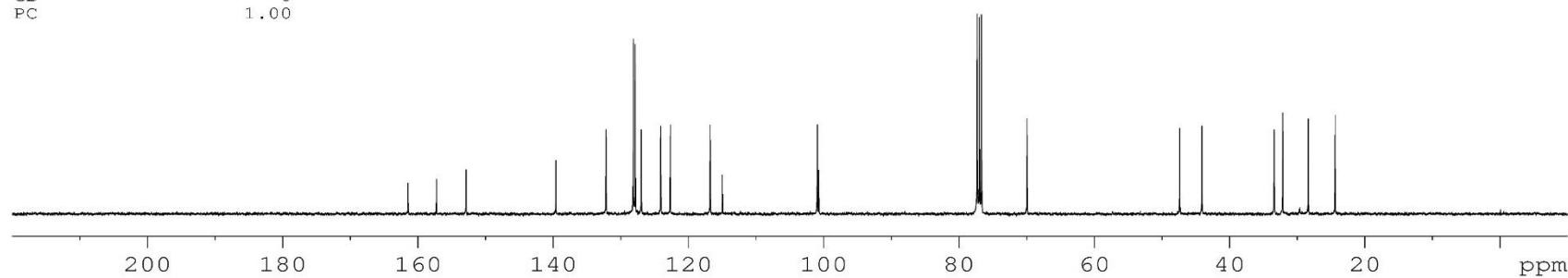


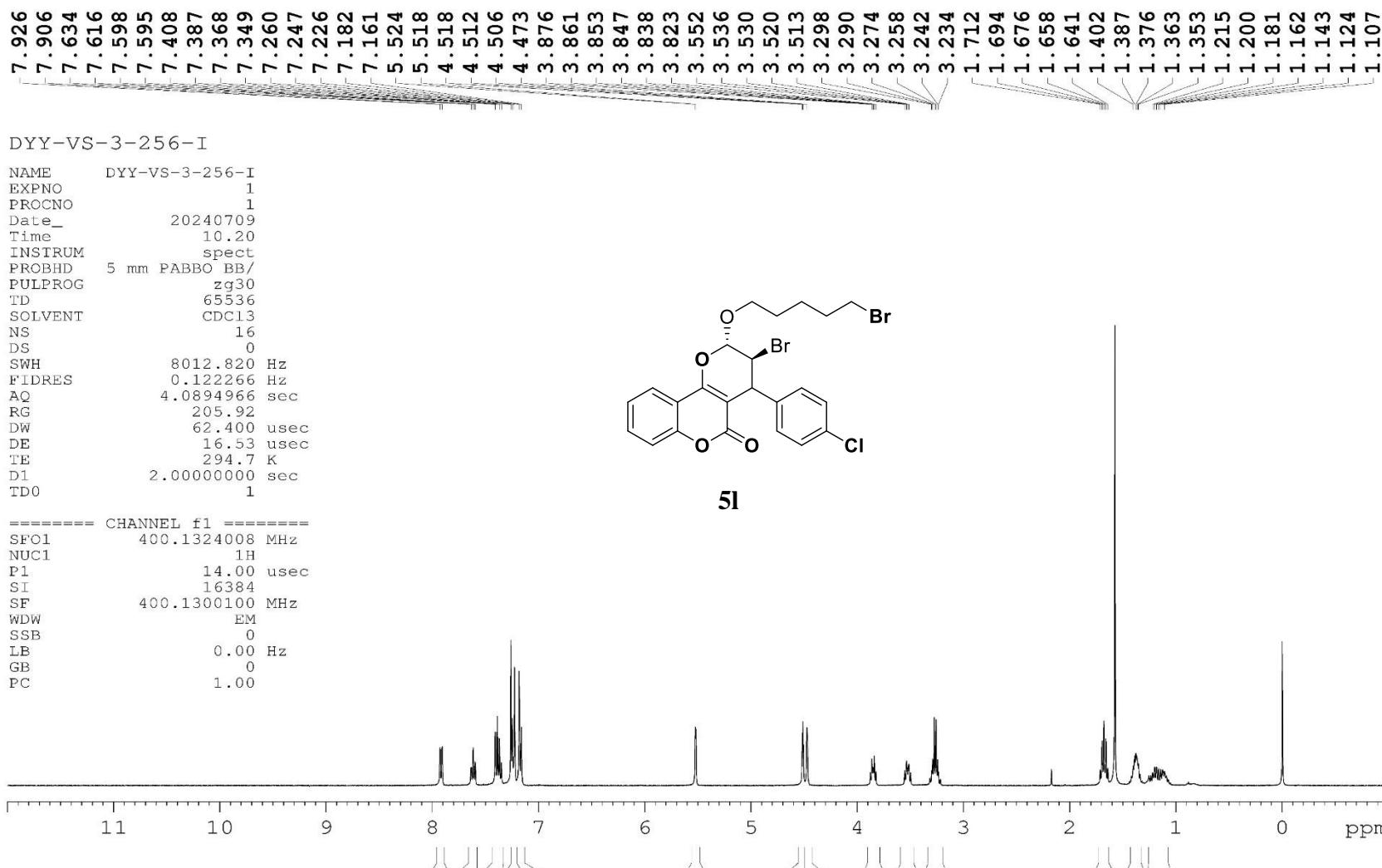


DYY-VS-3-230C

NAME DYY-VS-3-230C  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20240531  
 Time 11.23  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 500  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631988 sec  
 RG 205.92  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 292.4 K  
 D1 2.0000000 sec  
 D11 0.03000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 SFO1 100.6233329 MHz  
 NUC1 13C  
 P1 10.00 usec  
 SI 32768  
 SF 100.6127789 MHz  
 WDW EM  
 SSB 0  
 LB 2.00 Hz  
 GB 0  
 PC 1.00



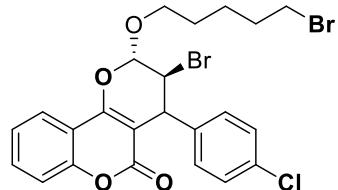
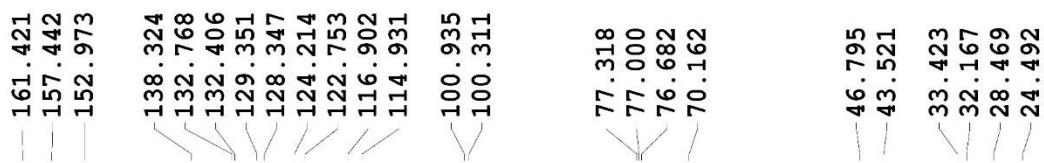


DYY-VS-3-256-IC

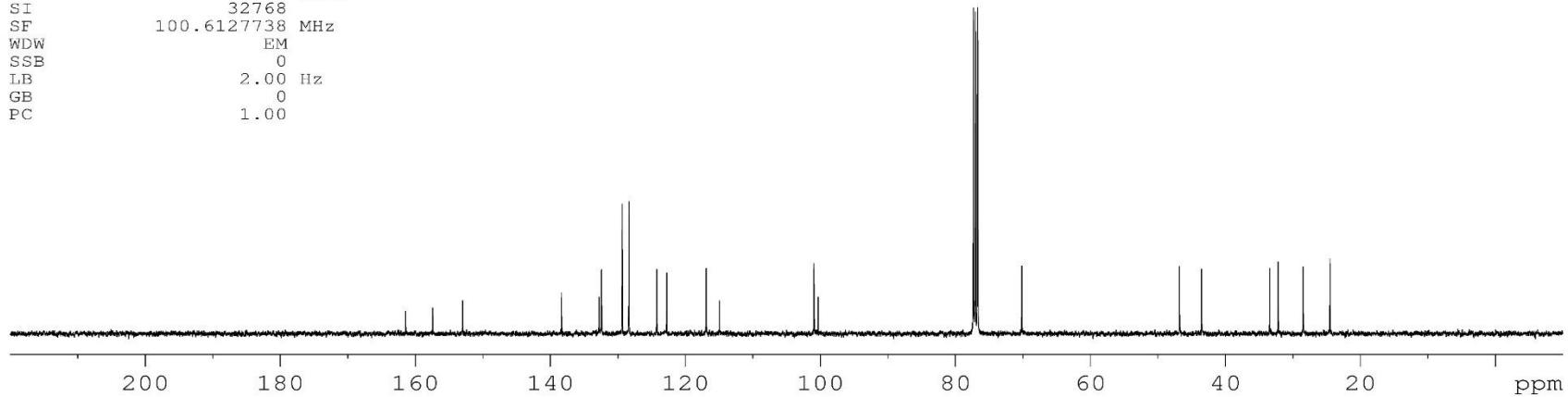
NAME DYY-VS-3-256-IC  
EXPNO 1  
PROCNO 1  
Date\_ 20240709  
Time 10.57  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 275  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 295.8 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TDO 1

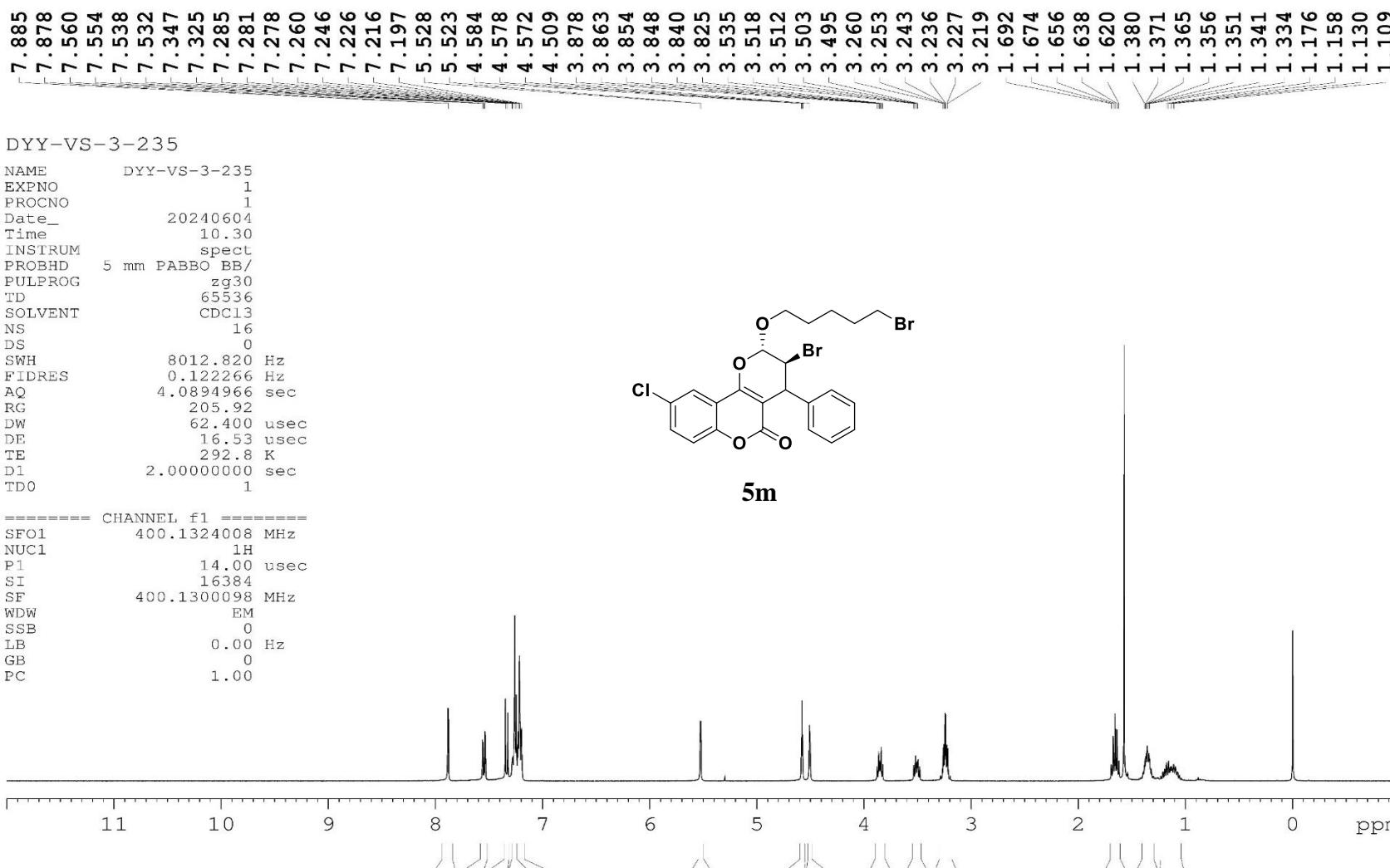
===== CHANNEL f1 =====

SFO1 100.6233329 MHz  
NUC1 13C  
P1 10.00 usec  
SI 32768  
SF 100.6127738 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



**5l**

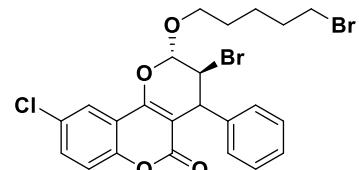
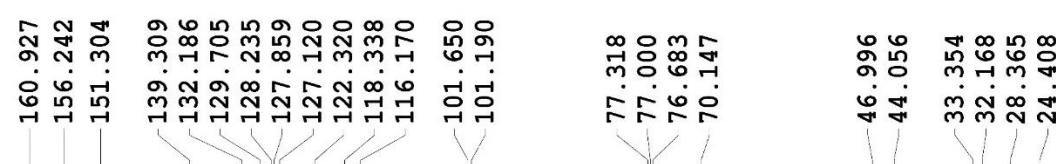




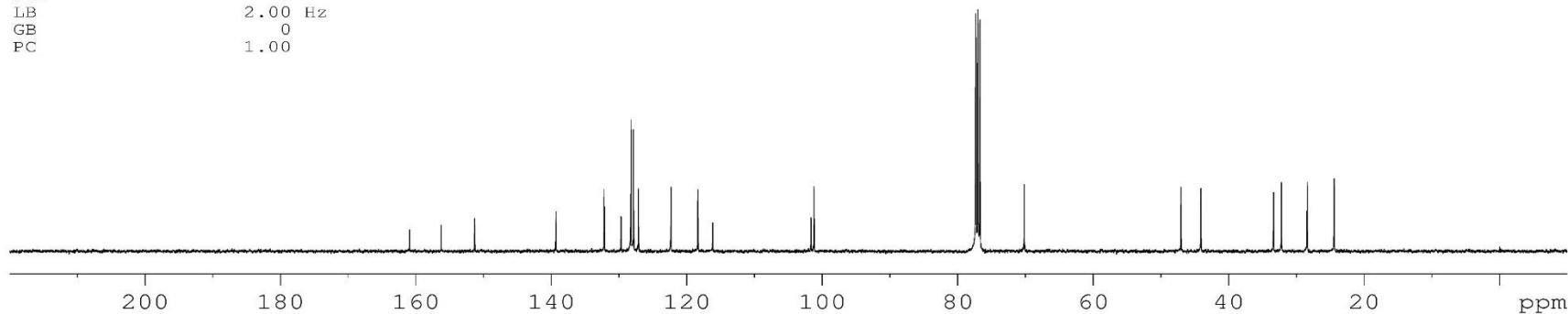
DYY-VS-3-235C

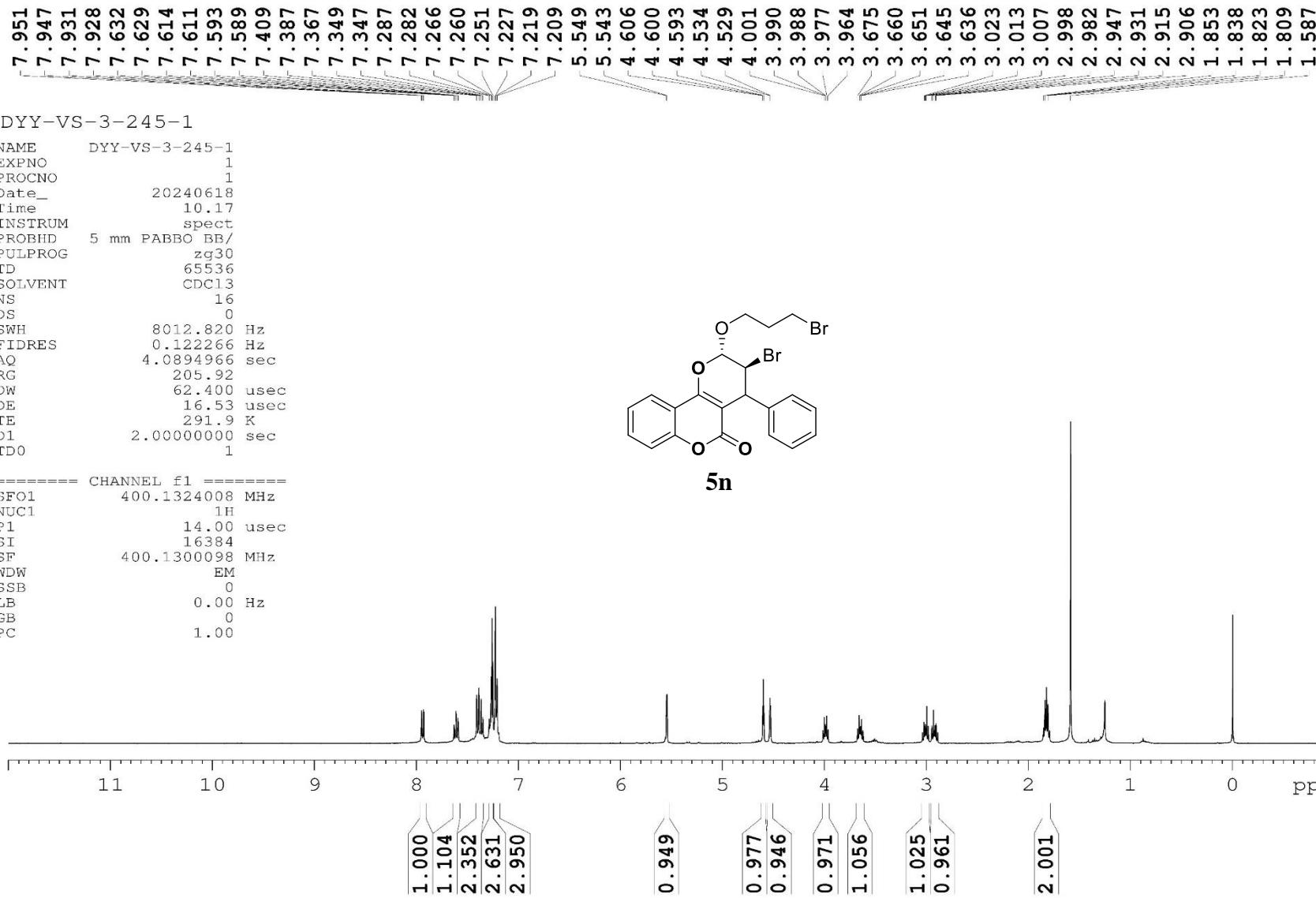
NAME DYY-VS-3-235C  
EXPNO 1  
PROCNO 1  
Date\_ 20240604  
Time 11.27  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 600  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 295.3 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TD0 1

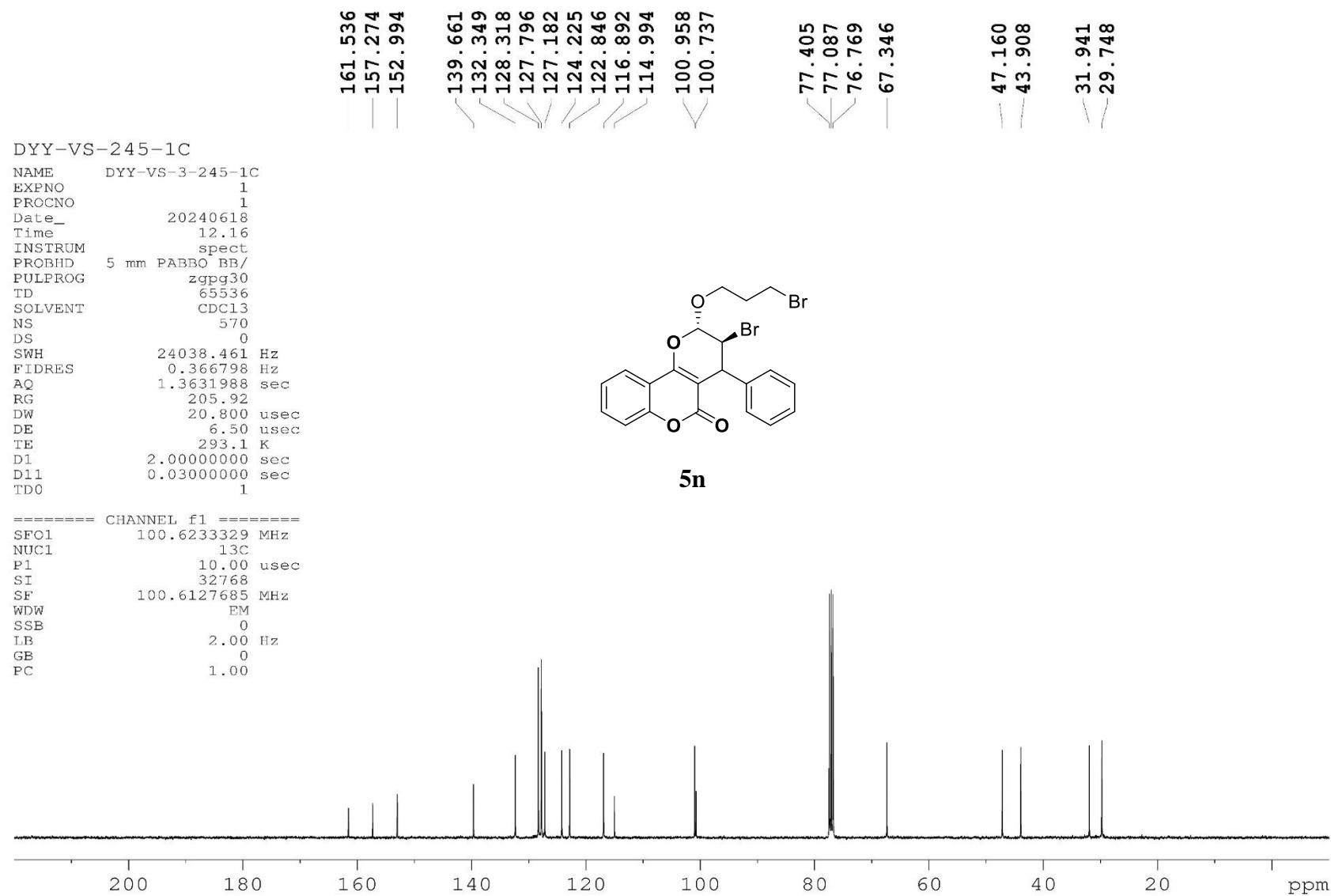
===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 13C  
P1 10.00 usec  
SI 32768  
SF 100.6127751 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00

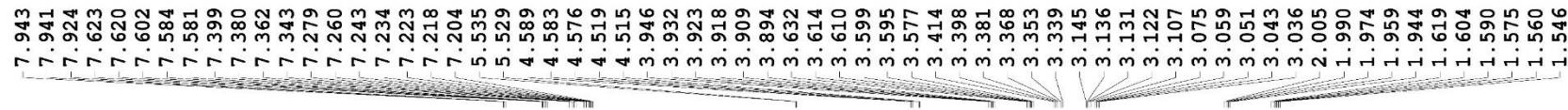


**5m**









DYY-VS-3-245B

```

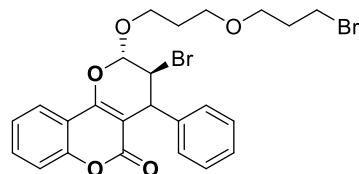
NAME      DYY-VS-3-245B
EXPNO     1
PROCNO    1
Date_     20240719
Time      11.13
INSTRUM   spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD        65536
SOLVENT   CDCl3
NS        16
DS        0
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ        4.0894966 sec
RG        55.28
DW        62.400 usec
DE        16.53 usec
TE        291.5 K
D1        2.00000000 sec
TDO      1

```

```

===== CHANNEL f1 =====
SFO1      400.1324008 MHz
NUC1       1H
P1        14.00 usec
SI        16384
SF        400.1300099 MHz
WDW       EM
SSB       0
LB        0.00 Hz
GB        0
PC        1.00

```



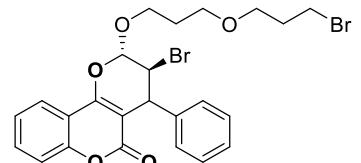
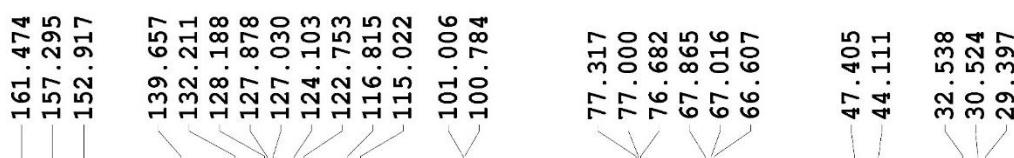
**50**



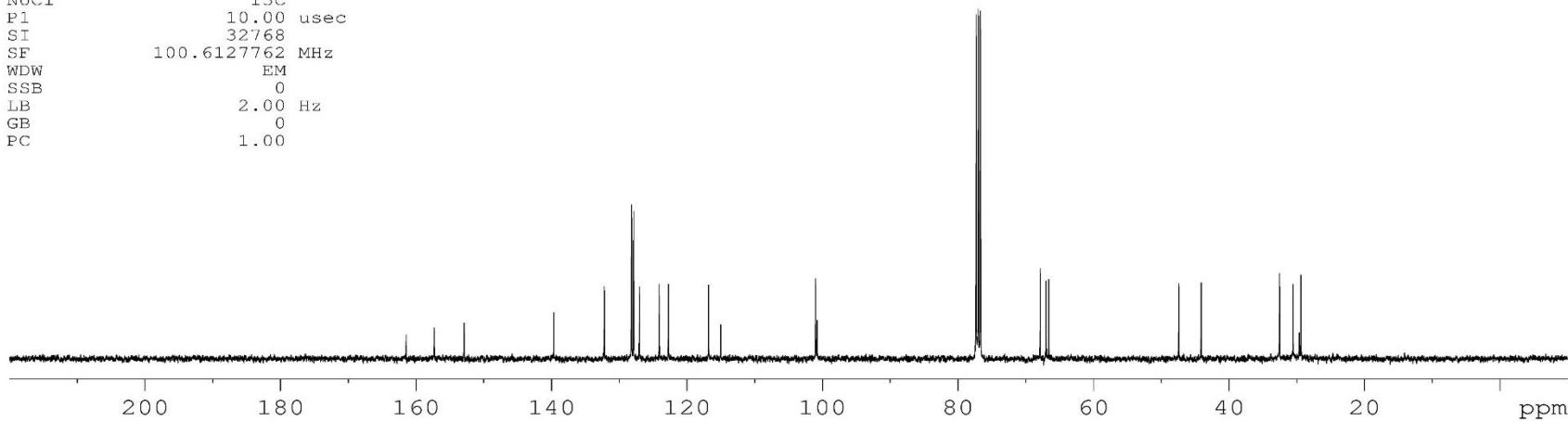
DYY-VS-3-245B-C

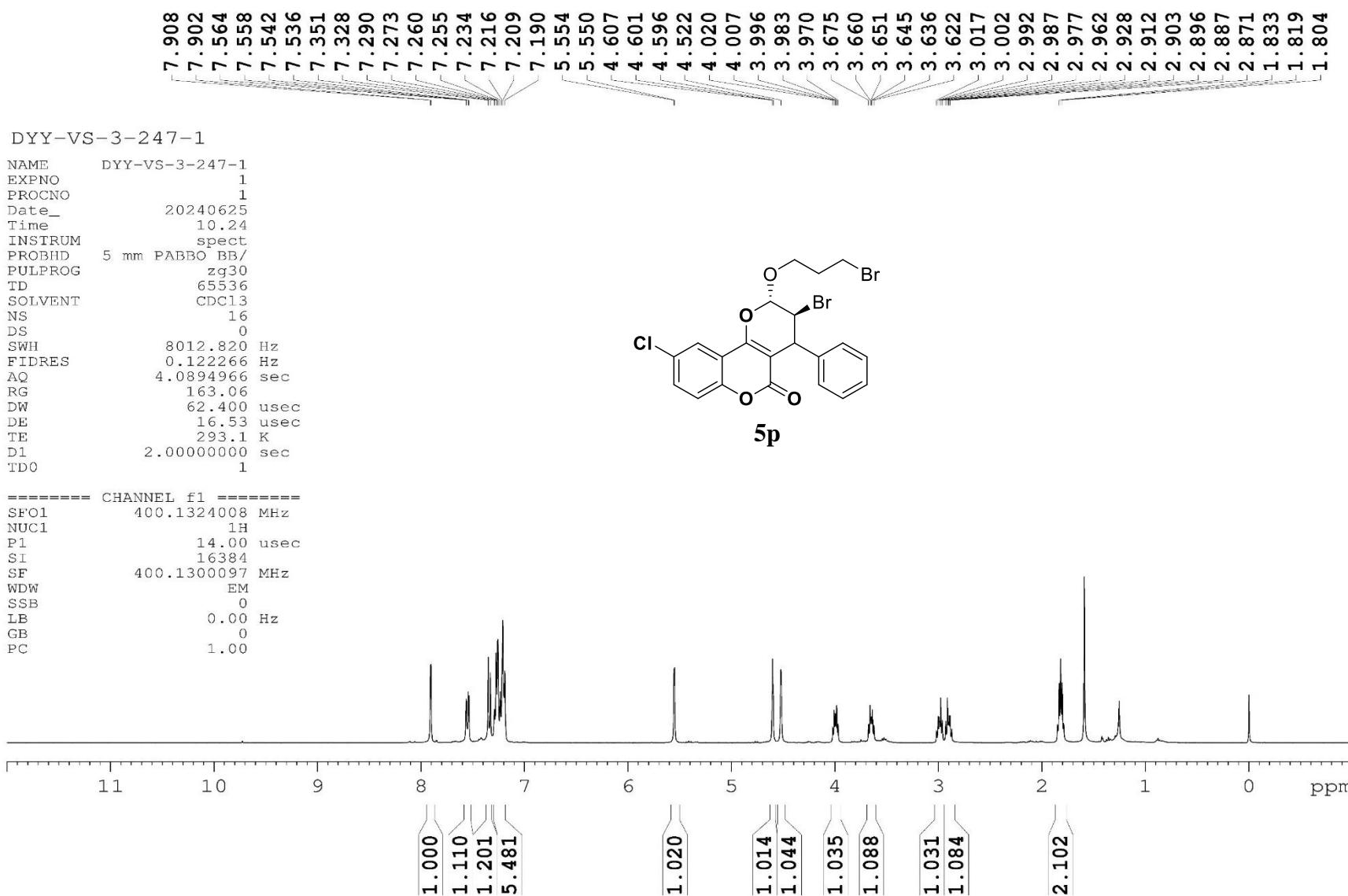
NAME DYY-VS-3-245B-C  
EXPNO 1  
PROCNO 1  
Date\_ 20240719  
Time 11.03  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 180  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 292.5 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127762 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00



**50**

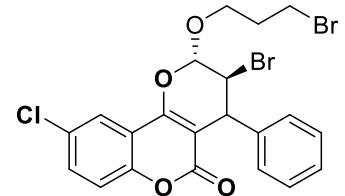
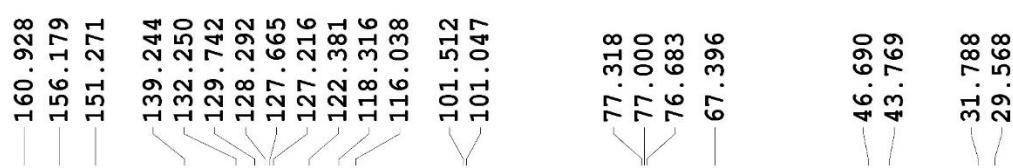




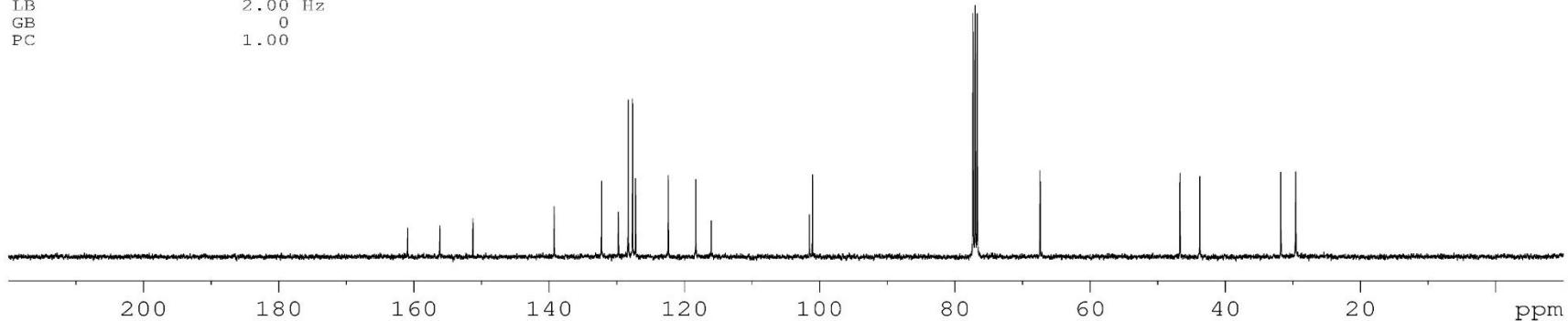
DYY-VS-3-247-1C

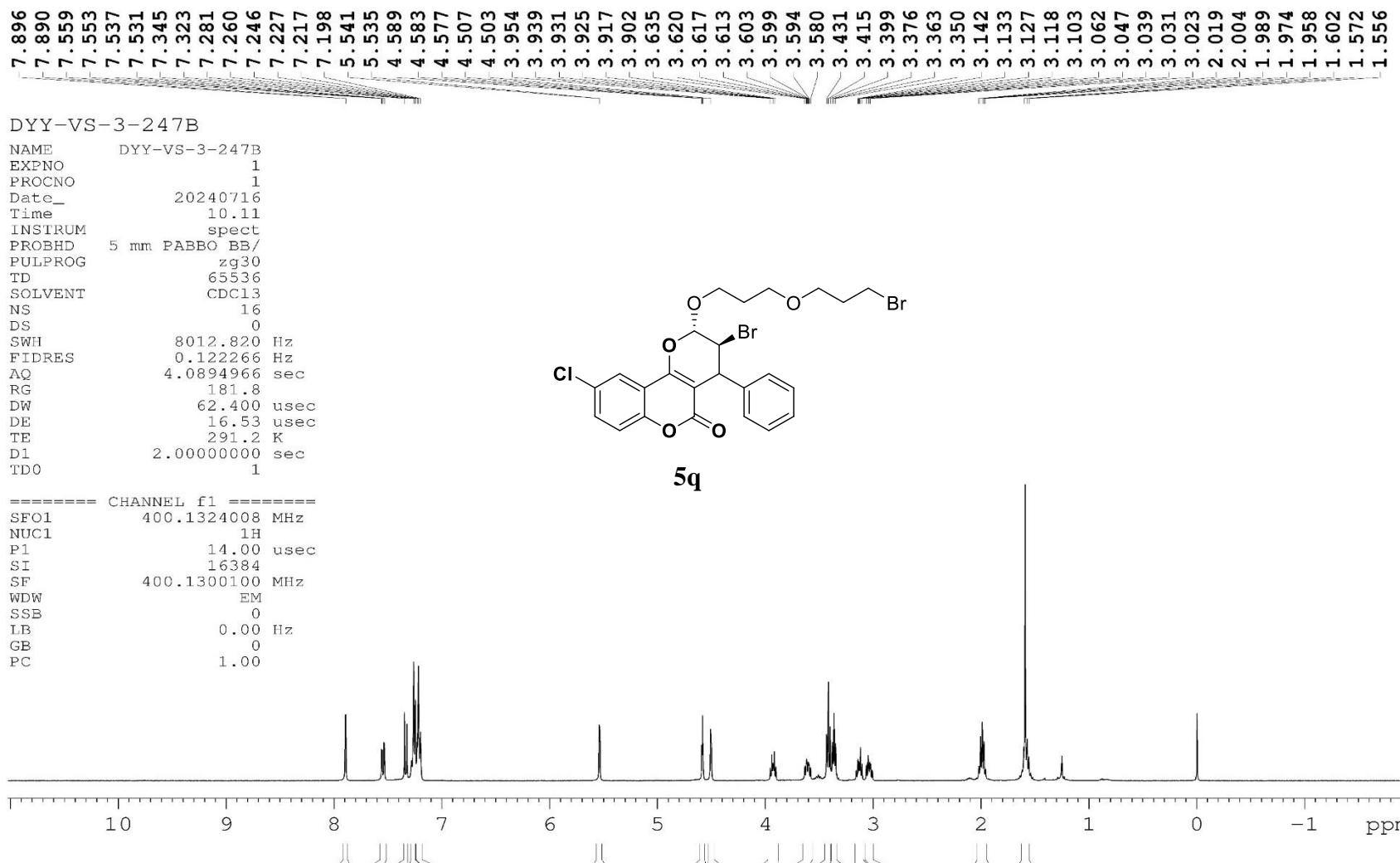
NAME DYY-VS-3-247-1C  
EXPNO 1  
PROCNO 1  
Date 20240625  
Time 11.10  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 202  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 293.5 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TDO 1

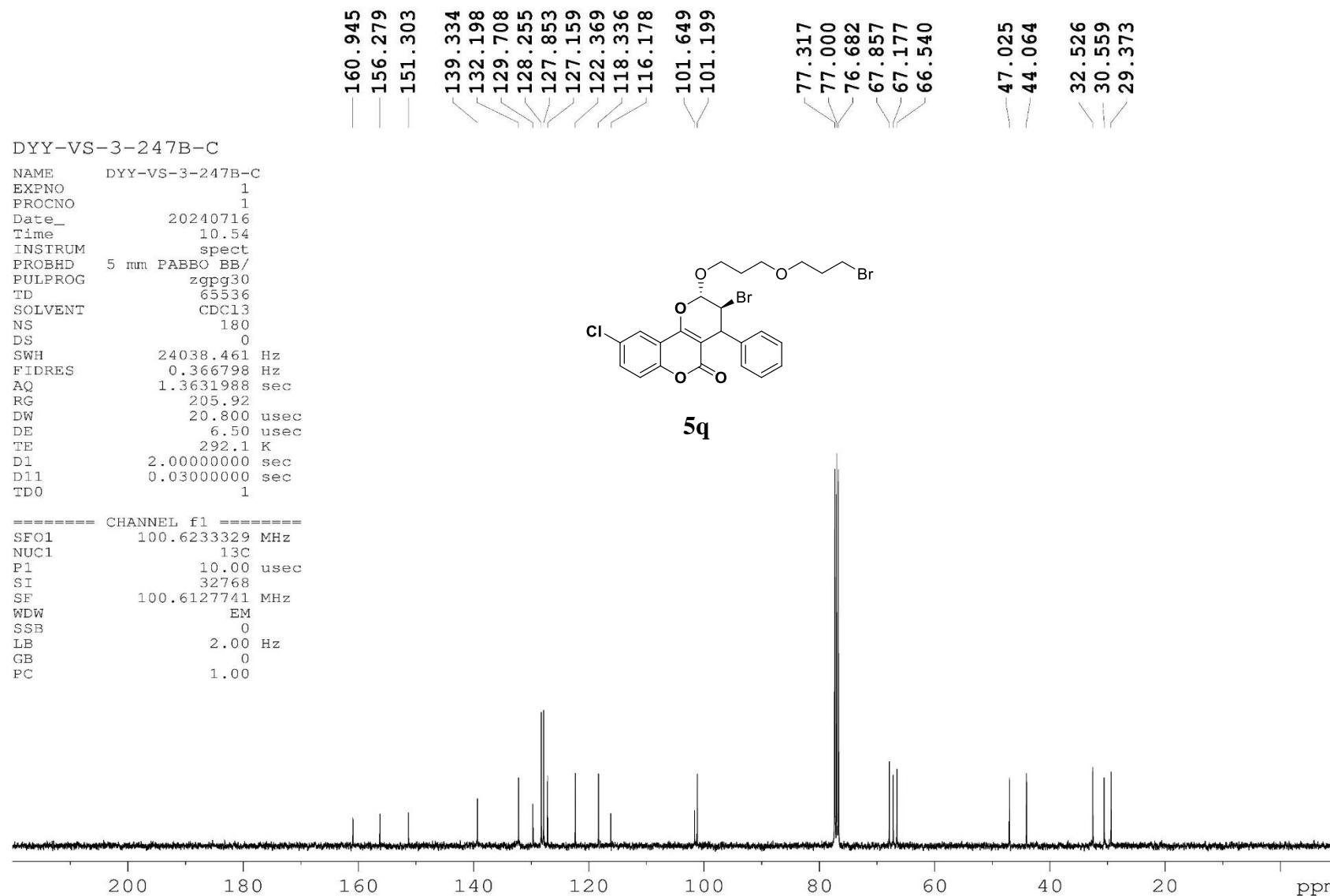
===== CHANNEL f1 =====  
SFO1 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127764 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00

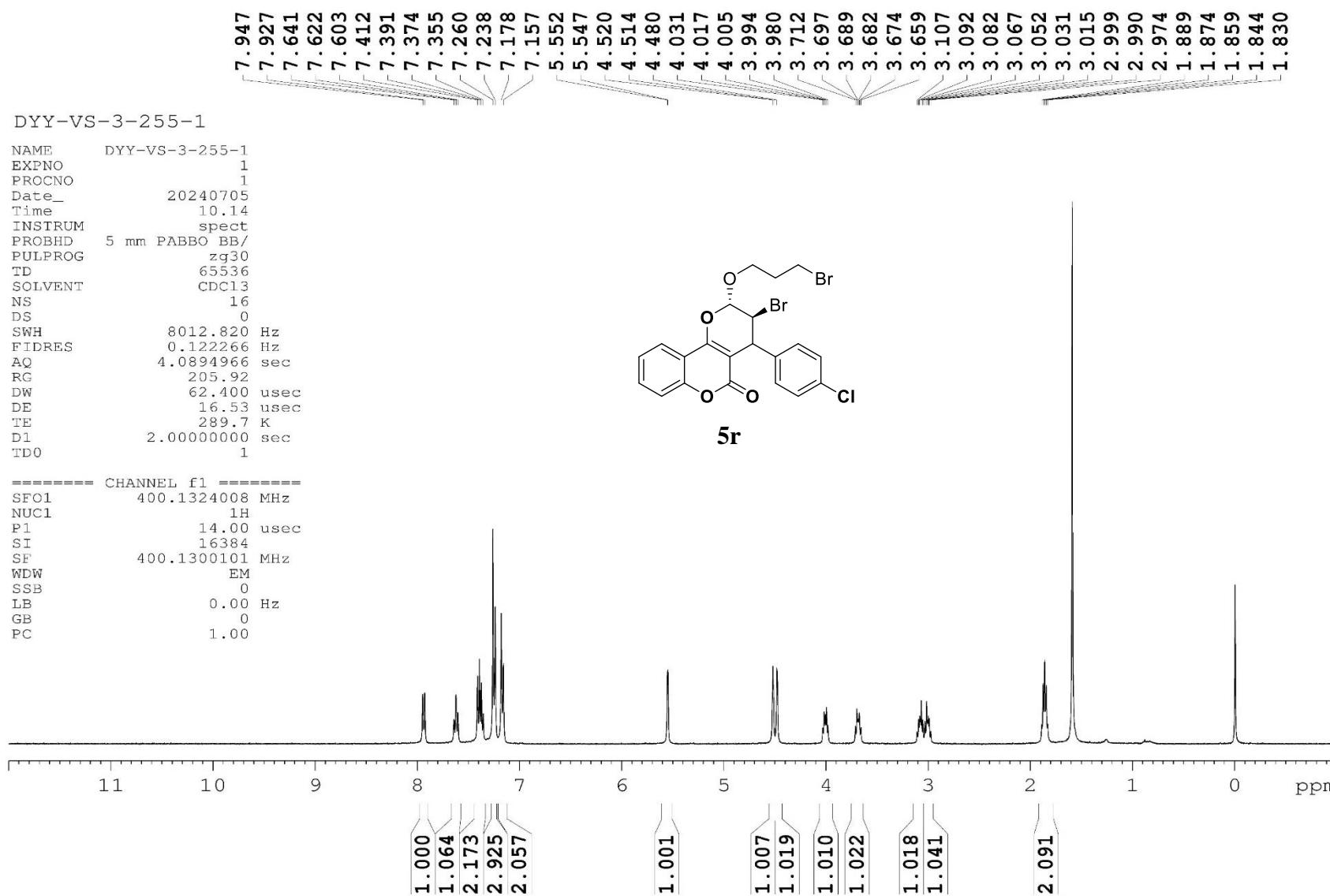


**5p**



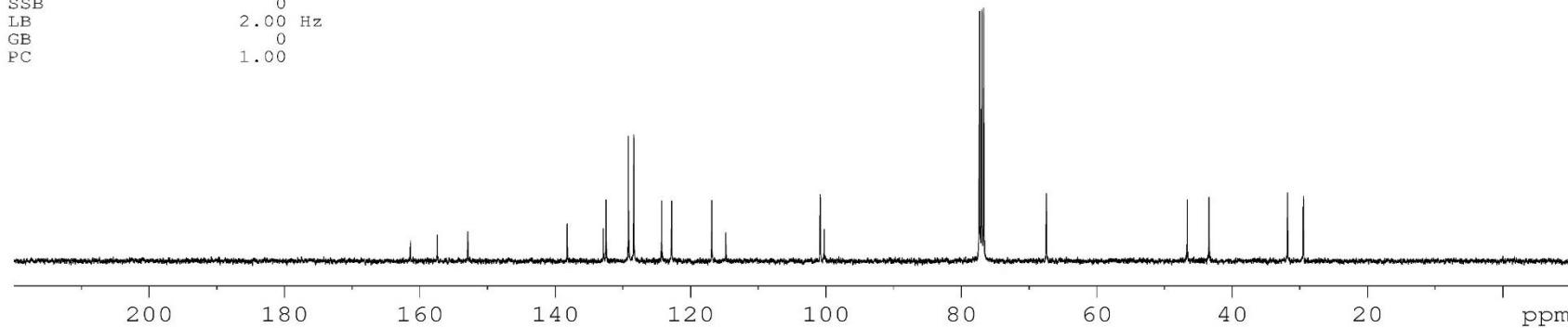
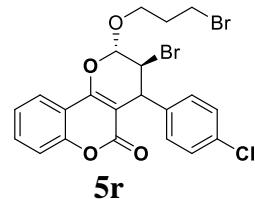
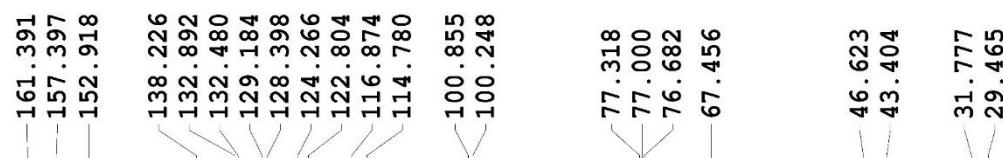






DYY-VS-3-255-1C

NAME DYY-VS-3-255-1C  
EXPNO 1  
PROCNO 1  
Date\_ 20240705  
Time 11.01  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 300  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 291.2 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TD0 1  
===== CHANNEL f1 ======  
SFO1 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127761 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00

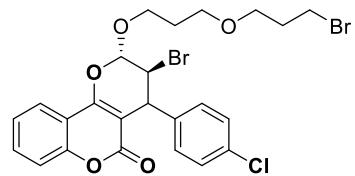


7.940  
 7.939  
 7.921  
 7.635  
 7.633  
 7.614  
 7.596  
 7.594  
 7.405  
 7.386  
 7.369  
 7.351  
 7.260  
 7.226  
 7.247  
 7.186  
 7.165  
 5.539  
 5.533  
 4.520  
 4.514  
 4.508  
 4.471  
 3.933  
 3.925  
 3.919  
 3.910  
 3.896  
 3.636  
 3.621  
 3.425  
 3.409  
 3.392  
 3.375  
 3.361  
 3.169  
 3.160  
 3.131  
 3.110  
 3.095  
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 3.072  
 2.008  
 1.993  
 1.978  
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 1.631  
 1.615  
 1.594  
 1.568

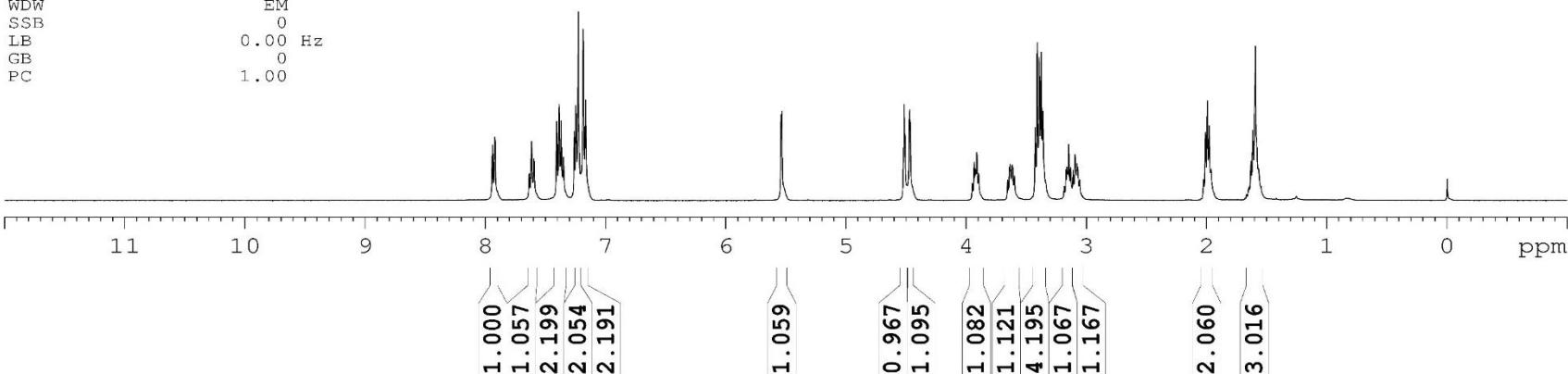
DYY-VS-3-255B-1

NAME DYY-VS-3-255B-1  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20240716  
 Time 11.59  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDC13  
 NS 16  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.122266 Hz  
 AQ 4.0894966 sec  
 RG 163.06  
 DW 62.400 usec  
 DE 16.53 usec  
 TE 291.4 K  
 D1 2.0000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 SFO1 400.1324008 MHz  
 NUC1 1H  
 P1 14.00 usec  
 SI 16384  
 SF 400.1300101 MHz  
 WDW EM  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00



**5s**



DYY-VS-3-255B-C

NAME DYY-VS-3-255B-C  
EXPNO 1  
PROCNO 1  
Date\_ 20240716  
Time 11.08  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 900  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 205.92  
DW 20.800 usec  
DE 6.50 usec  
TE 292.3 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TDO 1  
===== CHANNEL f1 =====  
SF01 100.6233329 MHz  
NUC1 <sup>13</sup>C  
P1 10.00 usec  
SI 32768  
SF 100.6127736 MHz  
WDW EM  
SSB 0  
LB 2.00 Hz  
GB 0  
PC 1.00

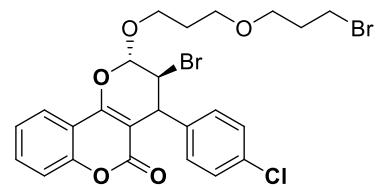
— 161.439  
— 157.473  
— 152.958

138.340  
132.784  
132.419  
129.353  
128.358  
124.222  
122.805  
116.896  
114.925

77.318  
77.000  
76.683  
67.952  
67.223  
66.617

46.819  
43.556

32.551  
30.529  
29.455



**5s**

