Light/Inductive Effect Induced Isomerization of Chromeno-5-methyl-2,6,9-trioxabicyclo[3.3.1]nonadienes

Sameer Vyasamudri[a] and Ding-Yah Yang*[a,b]

[a]*Department of Chemistry, Tunghai University,*

[b]*Graduate Program for Biomedical and Materials Science, Tunghai University*

No. 1727, Sec. 4, Taiwan Boulevard, Xitun District, Taichung 40704, Taiwan,

Republic of China

*Corresponding author; Tel: 886-4-2359-7613; Fax: 886-4-2359-0426;

Email: yang@thu.edu.tw

Supporting Information

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1. General Information

Melting points were determined on a Mel-Temp melting point apparatus in open capillaries and are uncorrected. High resolution mass spectra (HRMS) were obtained on a Thermo Fisher Scientific Finnigan MAT95XL spectrometer using a magnetic sector analyzer. Single-crystal structures were determined with a Bruker AXS SMART-1000 X-ray single-crystal diffractometer. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on a Bruker 400 spectrometer. Chemical shifts were reported in parts per million on the δ scale relative to an internal standard (tetramethylsilane, or appropriate solvent peaks) with coupling constants given in hertz. ^{1}H NMR multiplicity data are denoted by s (singlet), d (doublet), t (triplet), q (quartet), and m (multiplet). Photochemical reactions were performed using Rayonet reactor (PR-2000). UV-vis spectroscopy was recorded using Shimadzu UV-Spectrophotometer (UV-1800). Analytical thin-layer chromatography (TLC) was carried out on Merck silica gel 60G-254 plates (25 mm) and developed with the solvents mentioned. Solvents, unless otherwise specified, were reagent grade and distilled once prior to use. All new compounds exhibited satisfactory spectroscopic and analytical data. Commercially available appropriately substituted 4-hydroxycoumarins were purchased and used as received.

2. Supporting information for single crystal X-ray of **4a**.

Compound **4a** was dissolved in a mixture of CH_2Cl_2 and hexanes (1:1), this solution was set aside for slow evaporation to get colourless needle like crystals. Single-crystal X-ray data for these crystals were collected at 150 K on a Bruker APEX-II CCD diffractometer using graphite-monochromated Mo KR radiation (λ = 0.71073A°). 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.

Molecular structure of **4a** with atomic displacement shown at 50% probability. Crystallographic data, CCDC-1952549, can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request.cif.

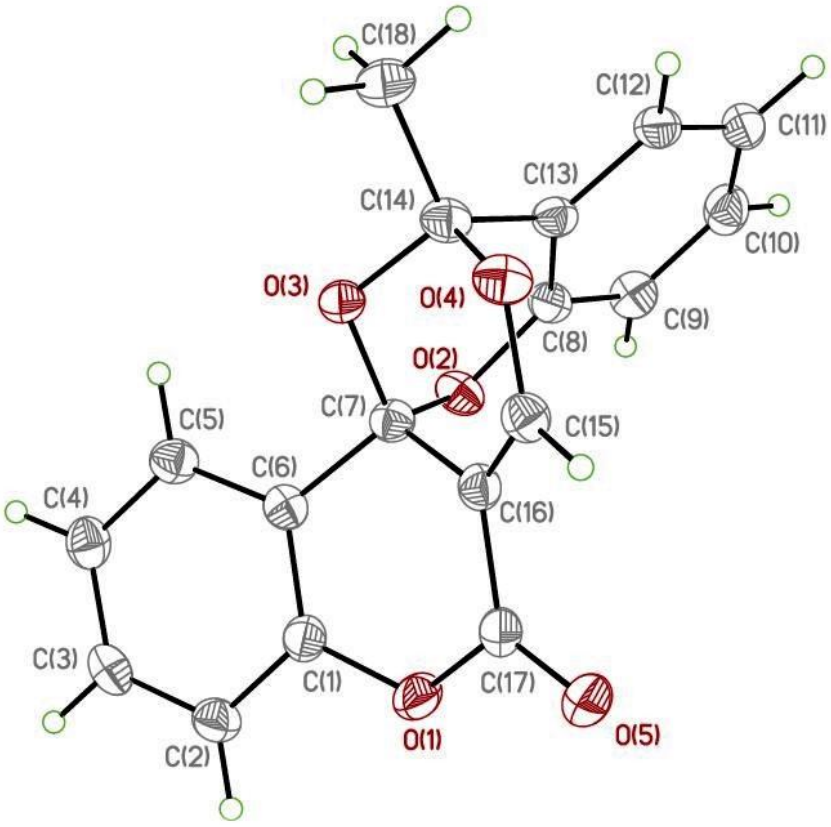


Table S2. Crystal data and structure refinement for **4a**.

Identification code	**4a**	
Empirical formula	C18 H12 O5	
Formula weight	308.28	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 10.6500(3) Å	α = 90°.
	b = 17.8886(5) Å	β = 101.6871(12)°.
	c = 7.6179(2) Å	γ = 90°.
Volume	1421.23(7) Å3	
Z	4	
Density (calculated)	1.441 Mg/m3	
Absorption coefficient	0.106 mm-1	
F(000)	640	
Crystal size	0.400 x 0.350 x 0.150 mm3	
Theta range for data collection	3.000 to 26.403°.	
Index ranges	-13<=h<=13, -22<=k<=22, -9<=l<=9	
Reflections collected	22834	
Independent reflections	2908 [R(int) = 0.0317]	
Completeness to theta = 25.242°	99.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9281 and 0.8623	
Refinement method	Full-matrix least-squares on F2	
Data / restraints / parameters	2908 / 0 / 208	
Goodness-of-fit on F2	1.033	
Final R indices [I>2sigma(I)]	R1 = 0.0336, wR2 = 0.0892	
R indices (all data)	R1 = 0.0405, wR2 = 0.0963	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.268 and -0.211 e.Å-3	

Supporting information for single crystal X-ray of **5a**.

Compound **5a** was dissolved in a mixture of CH_2Cl_2 and MeOH (3:1), this solution was set aside for slow evaporation to get colourless cubic crystals. Single-crystal X-ray data for these crystals were collected at 150 K on a Bruker APEX-II CCD diffractometer using graphite-monochromated Mo KR radiation (λ = 0.71073A°). 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.

Molecular structure of **5a** with atomic displacement shown at 50% probability. Crystallographic data, CCDC-1960547, can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request.cif.

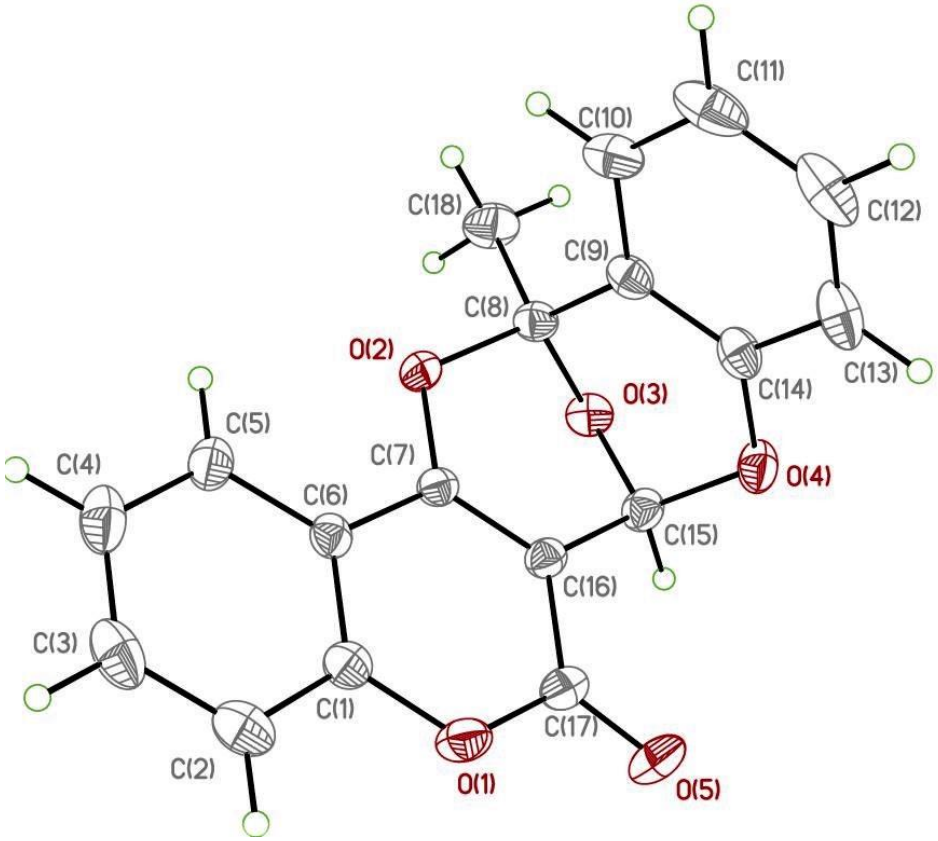
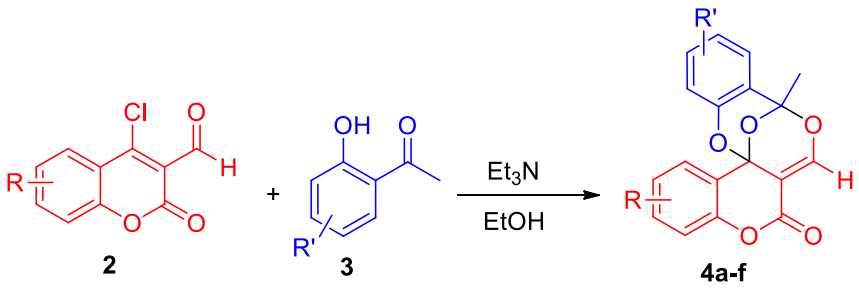


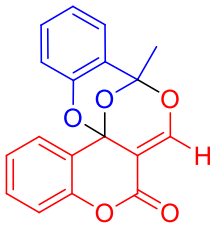
Table S3. Crystal data and structure refinement for **5a**.

Identification code	**5a**	
Empirical formula	C18 H12 O5	
Formula weight	308.28	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 7.6581(2) Å	α = 90°.
	b = 17.9590(6) Å	β = 92.0863(11)°.
	c = 10.1131(3) Å	γ = 90°.
Volume	1389.95(7) $Å^3$	
Z	4	
Density (calculated)	1.473 Mg/m^3	
Absorption coefficient	0.108 mm^{-1}	
F(000)	640	
Crystal size	0.430 x 0.370 x 0.340 mm^3	
Theta range for data collection	3.471 to 27.901°.	
Index ranges	-10<=h<=10, -23<=k<=23, -13<=l<=13	
Reflections collected	23177	
Independent reflections	3292 [R(int) = 0.0331]	
Completeness to theta = 25.242°	99.2 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9281 and 0.8713	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3292 / 0 / 208	
Goodness-of-fit on F^2	1.029	
Final R indices [I>2sigma(I)]	R1 = 0.0376, wR2 = 0.1126	
R indices (all data)	R1 = 0.0424, wR2 = 0.1195	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.324 and -0.259 $e.Å^{-3}$	

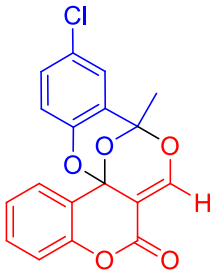
3. General Synthetic Procedure and spectroscopic data of **4a**–**f:**



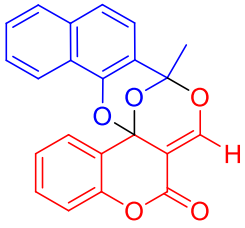
To a solution of appropriately substituted 4-chloro-3-formylcoumarin **2** (0.24 mmol) in EtOH (2 mL) was added appropriately substituted *o*-hydroxyacetophenones **3** (0.24 mmol) and triethylamine (0.24 mmol) at room temperature. The solution was then stirred at room temperature for two to six hours. The resulting precipitate was filtered, washed sequentially with ethanol, hexanes: dichloromethane (9:1), and dried under vacuum to obtain the desired product **4a**–**f**.



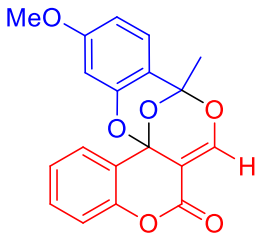
9-Methyl-9,14*a*-epoxybenzo[7,8][1,5]dioxocino[3,2-*c*]chromen-6(9*H*)-one (**4a**); Off-white solid; R_f = 0.50 (30% EtOAc/hexanes); 55 mg; yield 74%; mp 156–158 °C (lit,[1] 153–154 °C); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.80–7.78 (m, 1H), 7.79 (s, 1H), 7.50 (td, *J* = 8.4, 1.6 Hz, 1H), 7.38 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.35–7.28 (m, 2H), 7.21 (t, *J* = 8.4 Hz, 1H), 7.08 (t, *J* = 7.6 Hz, 1H), 6.88 (d, *J* = 8.0 Hz, 1H), 2.09 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 161.0, 158.0, 150.9, 150.1, 132.0, 131.4, 125.7, 125.4, 124.9, 122.4, 120.6, 120.3, 117.6, 117.2, 103.8, 99.6, 90.5, 23.8; HRMS (EI) m/z calcd for $C_{18}H_{12}O_5$ [M^+] 308.0685, found 308.0686.



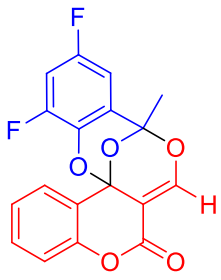
11-Chloro-9-methyl-9,14*a*-epoxybenzo[7,8][1,5]dioxocino[3,2-*c*]chromen-6(9*H*)-one (**4b**); Off-white solid; R_f = 0.55 (30% EtOAc/hexanes); 53 mg; yield 65%; mp 175–177 °C (lit,[1] 178–179 °C); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.79 (s, 1H), 7.77 (d, *J* = 7.6 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 1H), 7.37 (d, *J* = 2.4 Hz, 1H), 7.31–7.26 (m, 2H), 7.21 (d, *J* = 8.4 Hz, 1H), 6.83 (d, *J* = 8.4 Hz, 1H), 2.08 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 160.7, 157.8, 151.0, 148.8, 132.3, 131.6, 127.5, 125.8, 125.5, 125.1, 122.1, 120.0, 118.9, 117.7, 103.9, 99.1, 90.8, 23.9; HRMS (EI) m/z calcd for $C_{18}H_{11}ClO_5$ [M^+] 342.0295, found 342.0294.



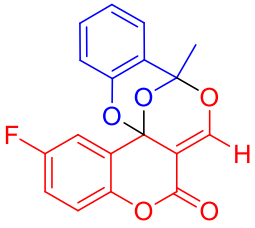
9-Methyl-9,16*a*-epoxynaphtho[2',1':7,8][1,5]dioxocino[3,2-*c*]chromen-6(9*H*)-one (**4c**); Off-white solid; R_f = 0.50 (30% EtOAc/hexanes); 59 mg; yield 69%; mp 181–183 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.01 (d, *J* = 8.4 Hz, 1H), 7.86 (d, *J* = 7.6 Hz, 1H), 7.82 (s, 1H), 7.79 (d, *J* = 8.4 Hz, 1H), 7.56–7.51 (m, 3H), 7.44 (t, *J* = 7.6 Hz, 1H), 7.40 (d, *J* = 8.4 Hz, 1H), 7.33 (t, *J* = 7.6 Hz, 1H), 7.26 (d, *J* = 8.4 Hz, 1H), 2.16 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 161.0, 158.1, 151.1, 146.4, 134.8, 132.0, 127.8, 127.6, 126.4, 125.6, 124.9, 123.9, 122.2, 122.1, 121.7, 120.4, 117.6, 114.4, 103.9, 99.9, 90.9, 23.7; HRMS (EI) m/z calcd for $C_{22}H_{14}O_5$ [M^+] 358.0841, found 358.0836.



12-Methoxy-9-methyl-9,14*a*-epoxybenzo[7,8][1,5]dioxocino[3,2-*c*]chromen-6(9*H*)-one (**4d**); Off-white solid; R_f = 0.45 (30% EtOAc/hexanes); 47 mg; yield 58%; mp 172–174 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.79–7.77 (m, 2H), 7.49 (td, *J* = 8.4, 1.6 Hz, 1H), 7.30–7.26 (m, 2H), 7.20 (d, *J* = 8.4 Hz, 1H), 6.63 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.38 (d, *J* = 2.4 Hz, 1H) 3.75 (s, 3H), 2.06 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 162.0, 161.1, 158.1, 151.5, 151.0, 132.0, 126.8, 125.4, 125.0, 120.5, 117.6, 113.0, 110.2, 103.7, 101.7, 100.0, 90.8, 55.6, 24.0; HRMS (EI) m/z calcd for $C_{19}H_{14}O_6$ [M^+] 338.0790, found 338.0784.

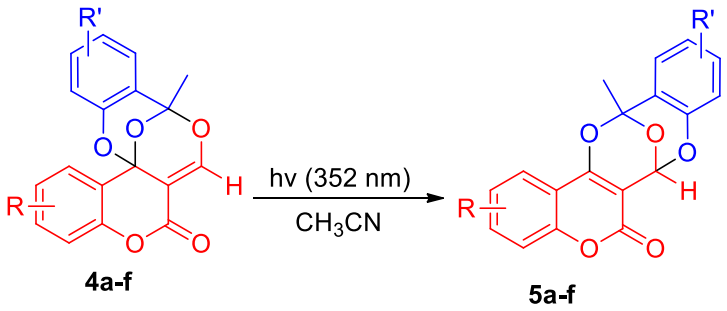


11,13-Difluoro-9-methyl-9,14*a*-epoxybenzo[7,8][1,5]dioxocino[3,2-*c*]chromen-6(9*H*)-one (**4e**); Off-white solid; R_f = 0.55 (30% EtOAc/hexanes); 55 mg; yield 67%; mp 175–177 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.80 (s, 1H), 7.77 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.51 (td, *J* = 8.0, 1.6 Hz, 1H), 7.29 (td, *J* = 8.0, 0.8 Hz, 1H), 7.20 (dd, *J* = 8.0, 0.8 Hz, 1H), 6.95–6.90 (m, 2H), 2.07 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 160.6, 157.8, 156.5 (dd, J_{C-F} = 242.8, 10.2 Hz, 1C), 151.1, 151.0 (dd, J_{C-F} = 250, 10.2 Hz, 1C) 135.7 (dd, J_{C-F} = 12.7, 3.0 Hz, 1C), 132.4, 125.6, 125.1, 123.1 (dd, J_{C-F} = 8.3, 3.0 Hz, 1C), 119.4, 117.8, 107.4 (dd, J_{C-F} = 24.0, 4.0 Hz, 1C), 106.9 (dd, J_{C-F} = 26.8, 21.2 Hz, 1C), 103.8, 98.7, 91.0, 23.9; HRMS (EI) m/z calcd for $C_{18}H_{10}F_2O_5$ [M^+] 344.0496, found 344.0496.

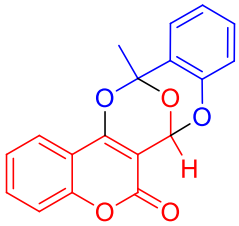


2-Fluoro-9-methyl-9,14*a*-epoxybenzo[7,8][1,5]dioxocino[3,2-*c*]chromen-6(9*H*)-one (**4f**); Off-white solid; R_f = 0.50 (30% EtOAc/hexanes); 35 mg; yield 45%; mp 178–180 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.80 (s, 1H), 7.47 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.38 (d, *J* = 7.6 Hz, 1H), 7.34 (t, *J* = 7.6, 1H), 7.20–7.18 (m, 2H), 7.09 (t, *J* = 7.6 Hz, 1H), 6.88 (d, *J* = 7.6 Hz, 1H), 2.09 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 160.8, 159.3 (d, J_{C-F} = 244, 1C), 158.4, 149.9, 147.1 (d, J_{C-F} = 9.6 Hz, 1C), 131.7, 125.9, 122.7, 121.8 (d, J_{C-F} = 8.0 Hz, 1C), 120.5, 119.3 (d, J_{C-F} = 8.2 Hz, 1C), 119.1 (d, J_{C-F} = 23.9 Hz, 1C), 117.3, 112.2 (d, J_{C-F} = 25.1 Hz, 1C), 103.3, 99.8, 90.3, 23.9; HRMS (EI) m/z calcd for $C_{18}H_{11}FO_5$ [M^+] 326.0591, 326.0583.

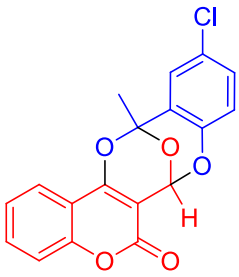
4. General Synthetic Procedure and spectroscopic data of **5a**–**f:**



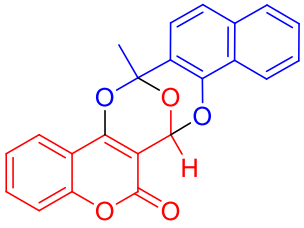
To a solution of **4** (0.065 mmol) in 20 mL of acetonitrile was taken in a quartz tube, and was irradiated with UV light (352 nm) in a Rayonet reactor (PR-2000). The reaction was monitored by TLC. After the completion of the reaction about 40 min, the solvent was evaporated under vacuum to obtain the photogenerated product **5a**–**f**.



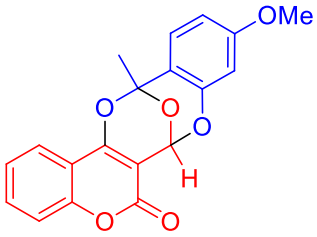
13-Methyl-7,13-dihydro-6*H*-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-*c*]chromen-6-one (**5a**); Off-white solid; R_f = 0.55 (30% EtOAc/hexanes); 19.8 mg; yield 99%; mp 162–164 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.79 (d, *J* = 8.0 Hz, 1H), 7.55 (t, *J* = 8.0 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.32–7.26 (m, 3H), 7.04–6.99 (m, 2H), 6.42 (s, 1H), 2.13 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 160.9, 160.0, 153.7, 150.0, 133.4, 131.5, 126.0, 124.3, 123.1, 122.2, 121.1, 117.6, 117.2, 114.1, 99.6, 98.5, 87.8, 24.3; HRMS (EI) m/z calcd for $C_{18}H_{12}O_5$ [M^+] 308.0685, 308.0677.



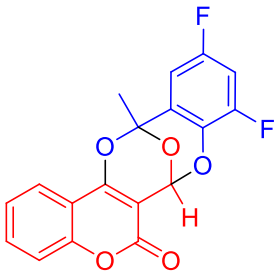
11-Chloro-13-methyl-7,13-dihydro-6*H*-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-*c*]chromen-6-one (**5b**); Off-white solid; R_f = 0.50 (30% EtOAc/hexanes); 21.6 mg; yield 98%; mp 180–182 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.80 (d, *J* = 7.6 Hz, 1H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.35 (d, *J* = 2.4 Hz, 1H), 7.32–7.29 (m, 2H), 7.27–7.24 (m, 2H), 6.98 (d, *J* = 8.8 Hz, 1H), 6.42 (s, 1H), 2.11 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 160.8, 159.8, 153.7, 148.6, 133.6, 131.6, 127.2, 125.8, 124.5, 123.1, 122.4, 119.2, 117.2, 113.8, 99.3, 97.9, 87.8, 24.2; HRMS (EI) m/z calcd for $C_{18}H_{11}ClO_5$ [M^+] 342.0295, found 342.0300.



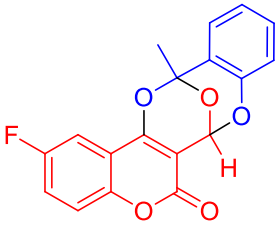
15-Methyl-7,15-dihydro-6*H*-7,15-epoxynaphtho[1',2':6,7][1,5]dioxocino[3,2-*c*]chromen-6-one (**5c**); Off-white solid; R_f = 0.45 (30% EtOAc/hexanes); 21.8 mg; yield 95%; mp 178–180 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.30 (s, 1H), 7.80–7.73 (m, 2H), 7.51–7.46 (m, 3H), 7.44–7.38 (m, 2H), 7.25–7.20 (m, 2H), 6.64 (s, 1H), 2.19 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 161.0, 159.9, 153.6, 146.4, 134.7, 133.3, 127.9, 127.5, 126.6, 124.3, 124.2, 123.0, 122.5, 121.9 (2C), 117.1, 114.7, 114.0, 99.7, 98.7, 88.1, 24.1; HRMS (EI) m/z calcd for $C_{22}H_{14}O_5$ [M^+] 358.0841, found 358.0848.



10-Methoxy-13-methyl-7,13-dihydro-6*H*-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-*c*]chromen-6-one (**5d**); Off-white solid; R_f = 0.40 (30% EtOAc/hexanes); 20.8 mg; yield 96%; mp 169–171 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.78 (td, *J* = 8.4, 2.0 Hz, 1H), 7.54 (td, *J* = 8.4, 1.2 Hz, 1H), 7.28–7.24 (m, 4H), 6.56 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.52 (d, *J* = 2.4 Hz, 1H), 6.39 (s, 1H), 3.74 (s, 3H), 2.10 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 161.9, 160.9, 160.1, 153.6, 151.3, 133.3, 126.8, 124.3, 123.0, 117.1, 114.2, 113.3, 110.0, 101.2, 99.5, 98.8, 87.9, 55.5, 24.3; HRMS (EI) m/z calcd for $C_{19}H_{14}O_6$ [M^+] 338.0790, found 338.0780.

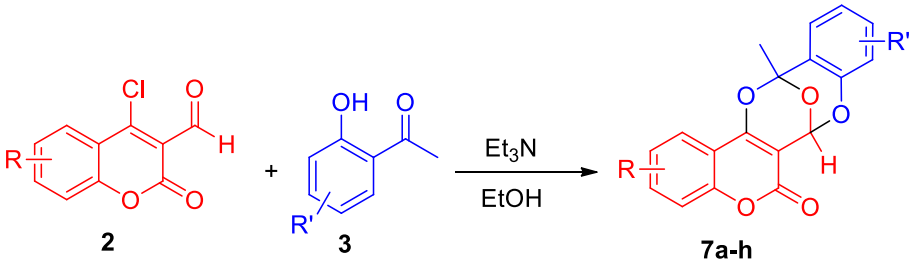


9,11-Difluoro-13-methyl-7,13-dihydro-6*H*-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-*c*]chromen-6-one (**5e**); Off-white solid; R_f = 0.50 (30% EtOAc/hexanes); 21.8 mg; yield 99%; mp 181–183 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.78 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.59 (td, *J* = 8.4, 1.6 Hz, 1H), 7.32–7.27 (m, 2H), 6.93–6.86 (m, 2H), 6.50 (s, 1H), 2.11 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 160.7, 159.4, 156.5 (dd, J_{C-F} = 242.5, 10.1 Hz, 1C), 153.7, 151.0 (dd, J_{C-F} = 252, 11.8 Hz, 1C), 135.5 (d, J_{C-F} = 9.6 Hz, 1C), 133.8, 124.5, 123.4 (d, J_{C-F} = 8.2 Hz, 1C), 123.0, 117.3, 113.7, 107.4 (dd, J_{C-F} = 23.8, 4.0 Hz, 1C), 106.9 (dd, J_{C-F} = 26.8, 21.1 Hz, 1C), 99.0, 97.4, 87.9, 24.2; HRMS (EI) m/z calcd for $C_{18}H_{10}F_2O_5$ [M^+] 344.0496, found 344.0491.

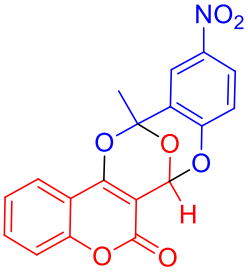


2-Fluoro-13-methyl-7,13-dihydro-6*H*-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-*c*]chromen-6-one (**5f**); Off-white solid; R_f = 0.45 (30% EtOAc/hexanes); 20.6 mg; yield 98%; mp 175–177 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.45 (d, *J* = 7.6 Hz, 1H), 7.39 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.31 (td, *J* = 8.0, 1.2 Hz, 1H), 7.27–7.25 (m, 2H), 7.04–7.00 (m, 2H), 6.41 (s, 1H), 2.13 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 160.2, 160.1, 159.6, 158.8 (d, J_{C-F} = 243.7 Hz, 1C), 149.8 (d, J_{C-F} = 7.6 Hz, 1C), 131.6, 126.0, 122.3, 120.9 (d, J_{C-F} = 24.3 Hz, 1C), 120.8, 118.9 (d, J_{C-F} = 8.2 Hz, 1C), 117.6, 115.0 (d, J_{C-F} = 9.1 Hz, 1C), 108.8 (d, J_{C-F} = 25.2 Hz, 1C), 100.3, 98.9, 87.6, 24.2; HRMS (EI) m/z calcd for $C_{18}H_{11}FO_5$ [M^+] 326.0591, found 326.0598.

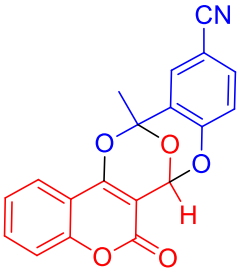
5. General Synthetic Procedure and spectroscopic data of **7a**–**h:**



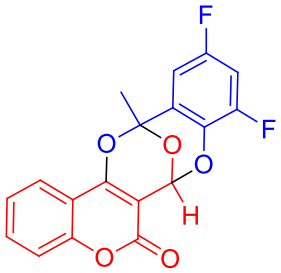
To a solution of appropriately substituted 4-chloro-3-formylcoumarin **2** (0.24 mmol) in EtOH (2 mL) was sequentially added appropriately substituted *o*-hydroxyacetophenones **3** (0.24 mmol) and triethylamine (0.24 mmol) at room temperature. The mixture was then stirred at room temperature for 12 hours. The resulting precipitate was filtered, washed with ethanol and henxanes: dichloromethane (9:1), and dried under vacuum to obtain the desired product **7a**–**h**.



13-Methyl-11-nitro-7,13-dihydro-6*H*-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-*c*]chromen-6-one (**7a**); Off-white solid; R_f = 0.50 (30% EtOAc/hexanes); 52.5 mg; yield 62%; mp 196–198 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.36 (d, *J* = 2.8 Hz, 1H), 8.21 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.87 (d, *J* = 7.6 Hz, 1H), 7.60 (t, *J* = 7.6 Hz, 1H), 7.35–7.30 (m, 2H), 7.16 (d, *J* = 8.8 Hz, 1H), 6.53 (s, 1H), 2.20 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 160.8, 159.5, 155.3, 153.8, 142.4, 134.0, 127.1, 124.7, 123.2, 122.7, 121.8, 118.8, 117.3, 113.5, 99.0, 97.7, 88.3, 24.3; HRMS (EI) m/z calcd for $C_{18}H_{11}NO_7$ [M^+] 353.0536, found 353.0538.

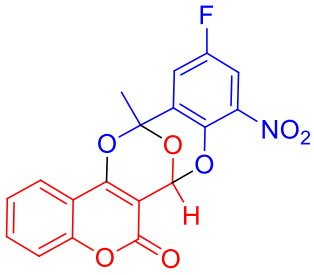


13-Methyl-6-oxo-7,13-dihydro-6*H*-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-*c*]chromene-11-carbonitrile (**7b**); Off-white solid; R_f = 0.45 (30% EtOAc/hexanes); 51 mg; yield 64%; mp 191–193 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.80 (d, *J* = 7.6 Hz, 1H), 7.73 (s, 1H), 7.62–7.57 (m, 2H), 7.35–7.30 (m, 2H), 7.13 (d, *J* = 8.4 Hz, 1H), 6.49 (s, 1H), 2.15 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 160.8, 159.5, 153.7, 153.6, 135.0, 133.9, 130.9, 124.7, 123.1, 122.5, 119.1, 118.2, 117.3, 113.6, 106.0, 99.1, 97.5, 88.1, 24.2; HRMS (EI) m/z calcd for $C_{19}H_{11}NO_5$ [M^+] 333.0637, found 333.0645.

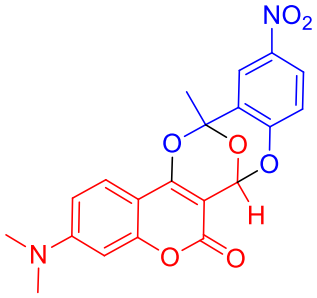


9,11-Difluoro-13-methyl-7,13-dihydro-6*H*-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-*c*]chromen-6-one (**7c**); Off-white solid; R_f = 0.50 (30% EtOAc/hexanes); 52 mg; yield 63%; mp 181–183 ^{o}C;

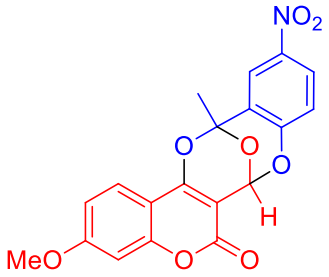
[NOTE: Compound **7c is same as **5e**, for spectral detail look for **5e**]



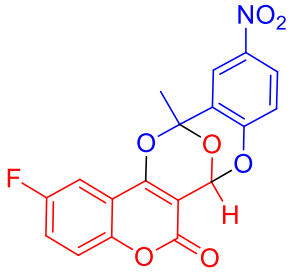
11-Fluoro-13-methyl-9-nitro-7,13-dihydro-6*H*-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-*c*]chromen-6-one (**7d**); Off-white solid; R_f = 0.55 (30% EtOAc/hexanes); 61.2 mg; yield 69%; mp 199–201 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.78 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.73 (dd, *J* = 7.6, 3.2 Hz, 1H), 7.61 (td, *J* = 8.4, 1.6 Hz 1H), 7.41 (dd, *J* = 7.6, 3.2 Hz 1H), 7.34–7.30 (m, 2H), 6.60 (s, 1H), 2.16 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 160.8, 158.9, 155.4 (d, J_{C-F} = 245.5 Hz, 1C), 153.9, 141.3, 134.1, 125.4, 124.6, 123.0, 119.2, 118.2 (d, J_{C-F} = 23.9 Hz, 1C), 117.4, 115.4 (d, J_{C-F} = 27.2 Hz, 1C), 113.3, 98.8, 97.2, 88.4, 24.5; HRMS (EI) m/z calcd for $C_{18}H_{10}FNO_7$ [M^+] 371.0441, found 371.0439.



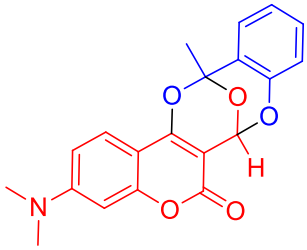
3-(Dimethylamino)-13-methyl-11-nitro-7,13-dihydro-6*H*-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-*c*]chromen-6-one (**7e**); Light yellow solid; R_f = 0.40 (40% EtOAc/hexanes); 43 mg; yield 45%; mp 201–203 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.34 (d, *J* = 2.8 Hz, 1H), 8.19 (dd, *J* = 9.2, 2.8 Hz, 1H), 7.57 (d, *J* = 8.8 Hz, 1H), 7.14 (d, *J* = 9.2 Hz, 1H), 6.61 (dd, *J* = 9.2, 2.4 Hz, 2H), 6.50 (s, 1H), 6.42 (d, *J* = 2.4 Hz, 1H), 3.05 (s, 6H), 2.15 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 161.4, 160.7, 156.1, 155.6, 154.4, 142.2, 126.8, 124.0, 122.7, 122.3, 118.8, 109.3, 101.7, 97.9, 97.1, 93.8, 89.0, 40.3 (2C), 24.4; HRMS (EI) m/z calcd for $C_{20}H_{16}N_2O_7$ [M^+] 396.0958, found 396.0964.



3-methoxy-13-methyl-11-nitro-7,13-dihydro-6H-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-c]chromen-6-one (**7f**); White solid; R_f = 0.55 (40% EtOAc/hexanes); 47 mg; yield 51%; mp 200–202 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.35 (d, *J* = 2.4 Hz, 1H), 8.20 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.70 (d, *J* = 8.8, Hz 1H), 7.15 (d, *J* = 9.2, Hz 1H), 6.87 (dd, *J* = 9.2, 2.4 Hz 1H) 6.76 (d, *J* = 2.4 Hz, 1H) 6.50 (s, 1H), 3.86 (s, 3H), 2.18 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 164.6, 161.1, 159.9, 155.8, 155.4, 142.3, 127.0, 124.3, 122.7, 121.9, 118.8, 113.1, 106.5, 101.0, 97.5, 96.3, 88.6, 56.1, 24.3; HRMS (EI) m/z calcd for $C_{19}H_{13}NO_8$ [M^+] 383.0641, found 371.0647.



2-Fluoro-13-methyl-11-nitro-7,13-dihydro-6*H*-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-*c*]chromen-6-one (**7g**); Off-white solid; R_f = 0.50 (30% EtOAc/hexanes); 54.5 mg; yield 61%; mp 175–177 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.37 (s, 1H), 8.22 (d, *J* = 9.2 Hz, 1H), 7.49 (d, *J* = 7.2 Hz, 1H), 7.31 (d, *J* = 4.4 Hz, 2H), 7.17 (d, 9.2 Hz, 1H), 6.52 (s, 1H), 2.21 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 160.2, 160.1, 160.0 (d, $J_{C–F}$ = 242.6 Hz, 1C), 155.1, 149.9, 142.5, 127.2, 122.7, 121.6 (d, $J_{C–F}$ = 24.4 Hz, 1C), 121.5, 119.1 (d, $J_{C–F}$ = 8.0 Hz, 1C), 118.8, 114.4 (d, $J_{C–F}$ = 8.9 Hz, 1C), 109.0 (d, $J_{C–F}$ = 25.3 Hz, 1C), 99.8, 98.0, 88.1, 24.2; HRMS (EI) m/z calcd for $C_{18}H_{18}FNO_7$ [M^+] 371.0441, found 371.0436.

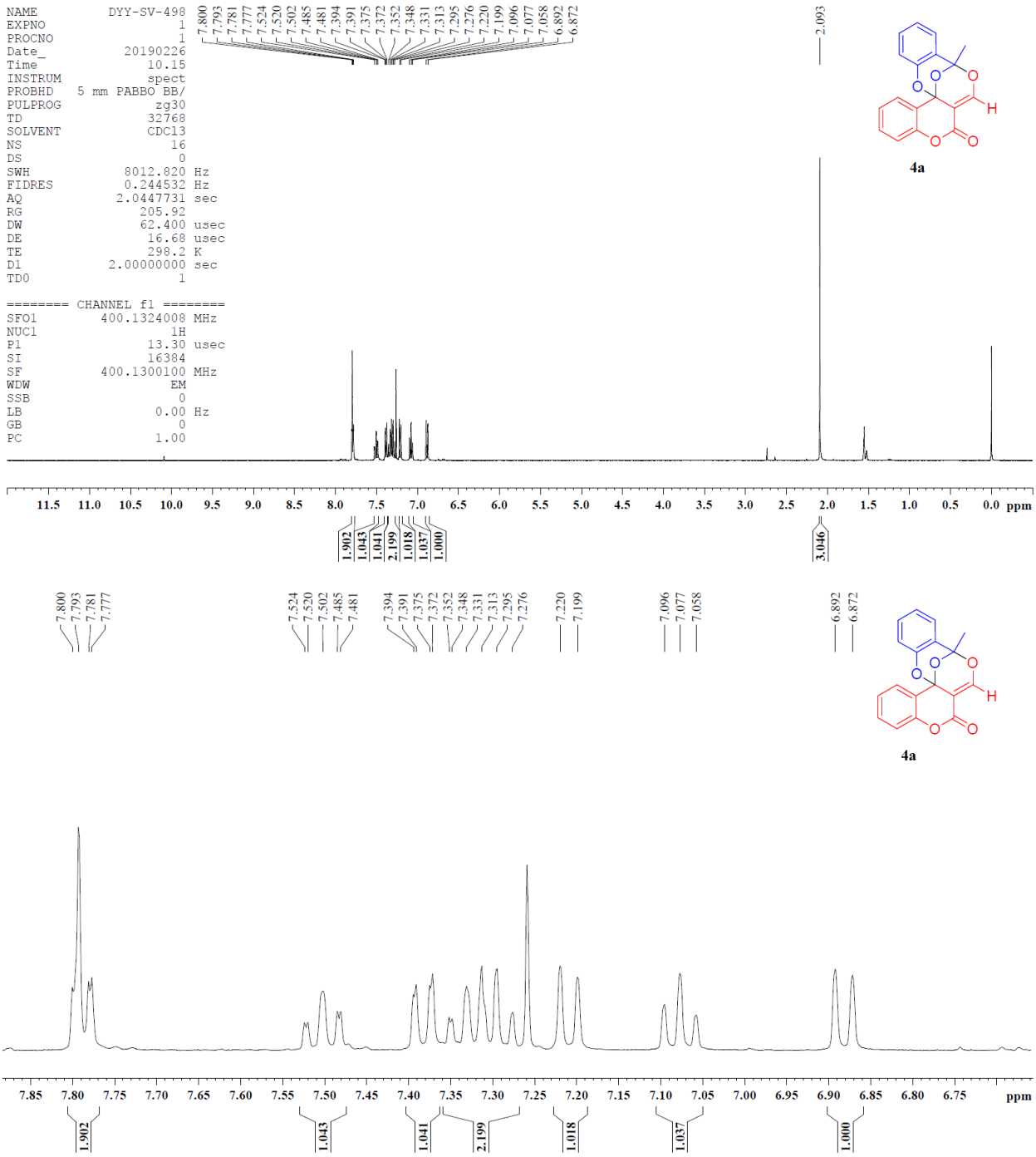


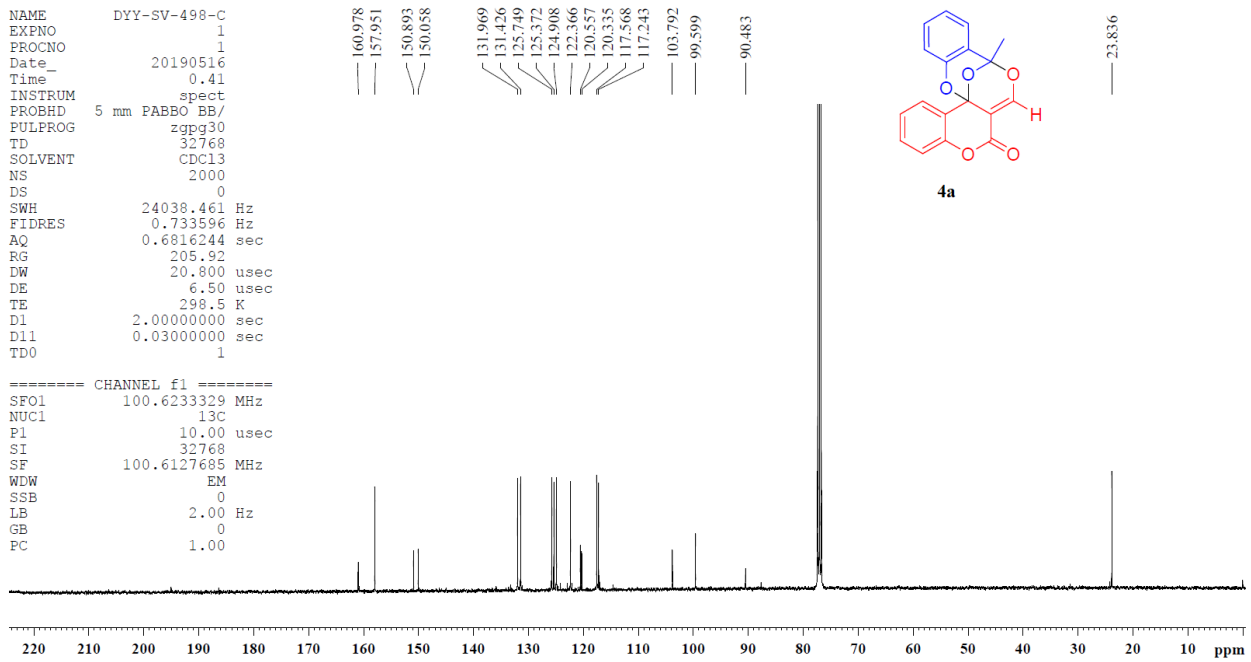
3-(Dimethylamino)-13-methyl-7,13-dihydro-6*H*-7,13-epoxybenzo[6,7][1,5]dioxocino[3,2-*c*]chromen-6-one (**7h**); Off-white solid; R_f = 0.45 (40% EtOAc/hexanes); 46 mg; yield 54%; mp 187–189 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.55 (d, *J* = 8.8 Hz, 1H), 7.36 (d, *J* = 7.6 Hz, 1H), 7.29–7.26 (m, 1H), 7.02–6.96 (m, 2H), 6.57 (d, *J* = 7.2 Hz, 1H), 6.40 (s, 1H), 6.42 (d, *J* = 2.4 Hz, 1H), 3.02 (s, 6H), 2.08 (s, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz) δ 161.6, 161.1, 155.9, 154.0, 150.2, 131.1, 125.9, 123.8, 121.8, 121.7, 117.7, 109.0, 102.6, 97.9 (2C), 94.6, 88.4, 40.3 (2C), 24.5; HRMS (EI) m/z calcd for $C_{20}H_{17}NO_5$ [M^+] 351.1107, found 351.1104.

6. Reference:

1. Jaggavarapu, S. R.; Kamalakaran, A. S.; Gayatri, G.; Shukla, M.; Dorai, K.; Gaddamanugu, G. *Tetrahedron* **2013**, *69*, 2142–2149.

7. Copies of ^{1}H and ^{13}C NMR spectra for **5a-f**, **6a-f**, and **7a-f**

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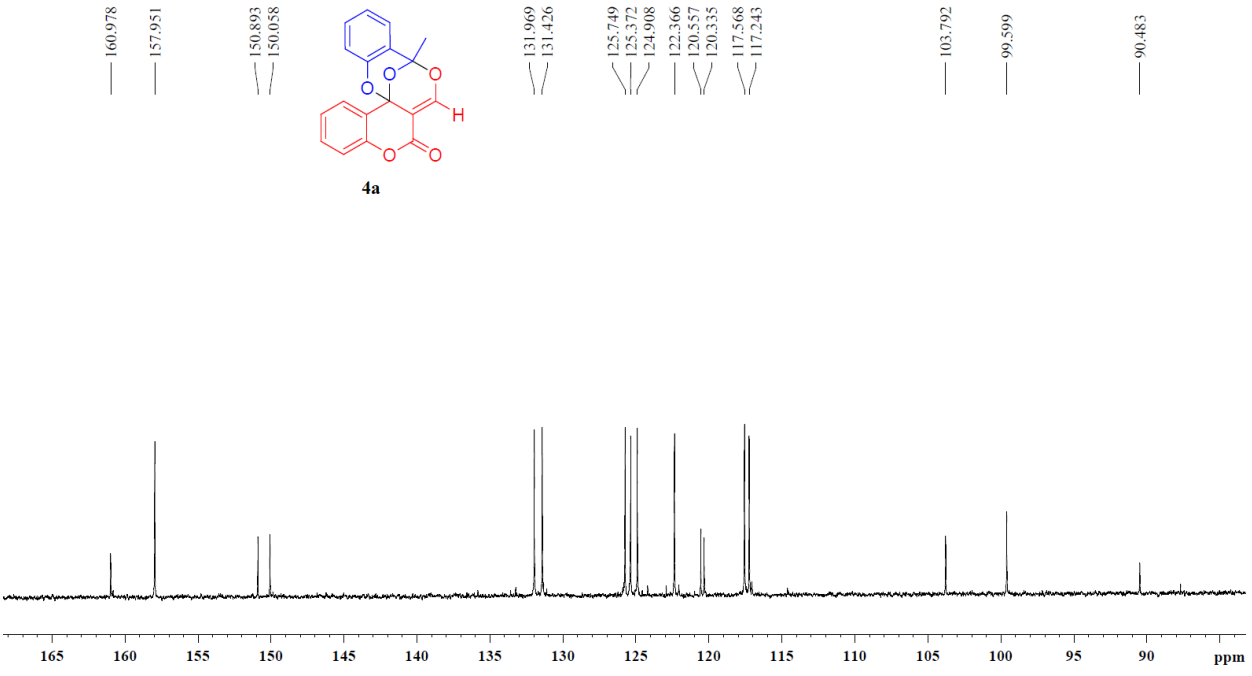
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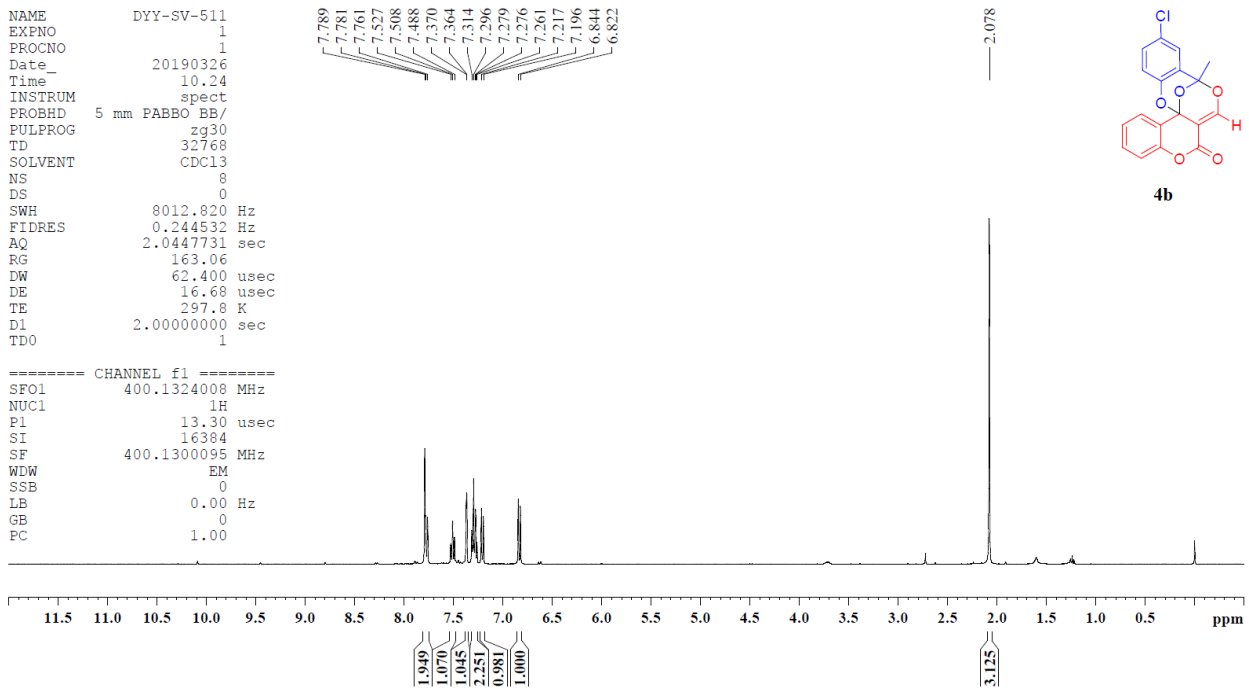
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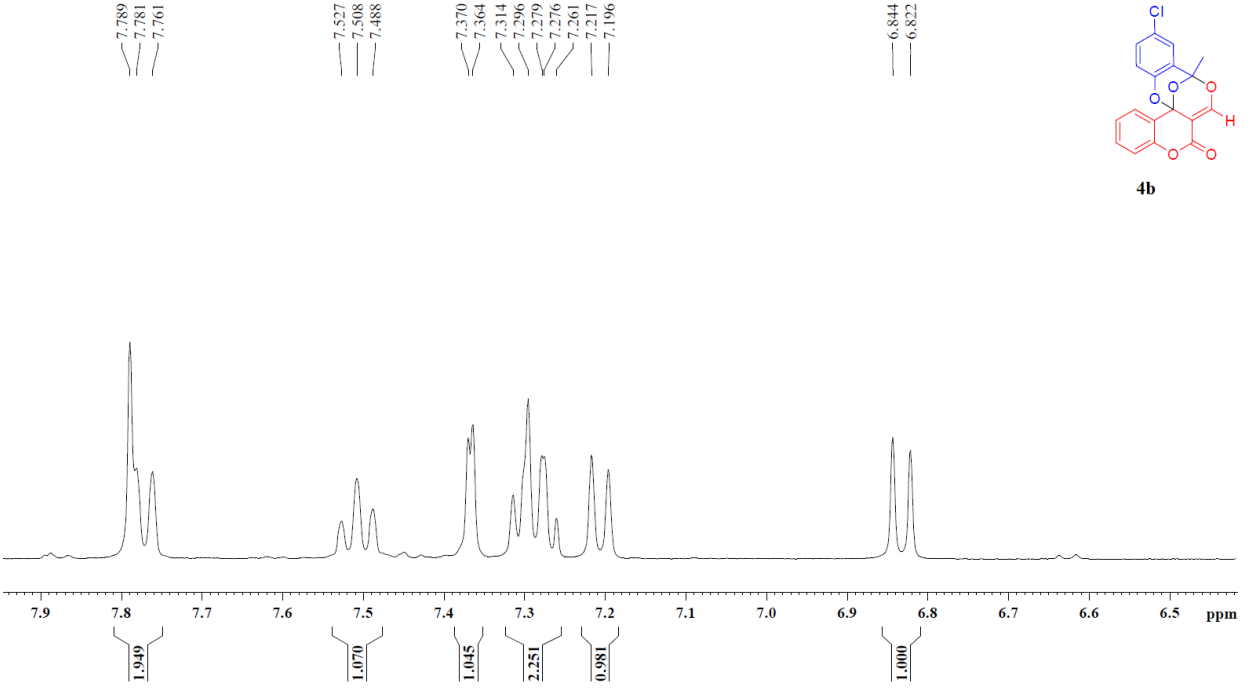
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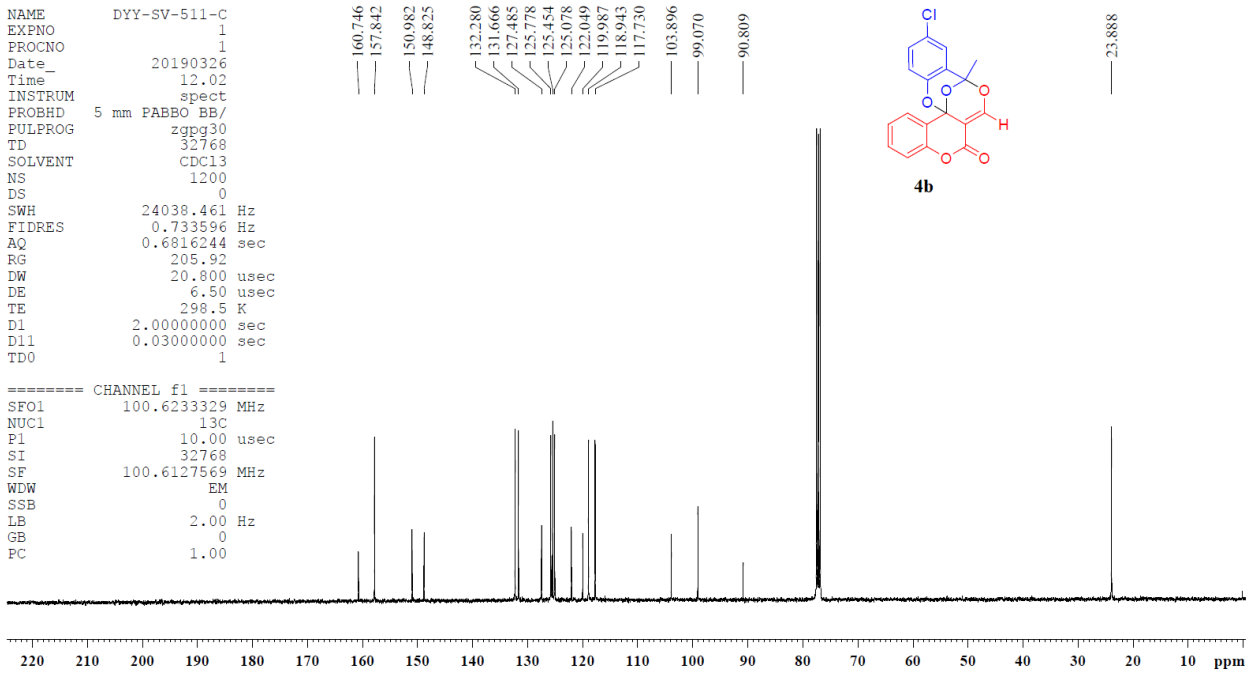
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H
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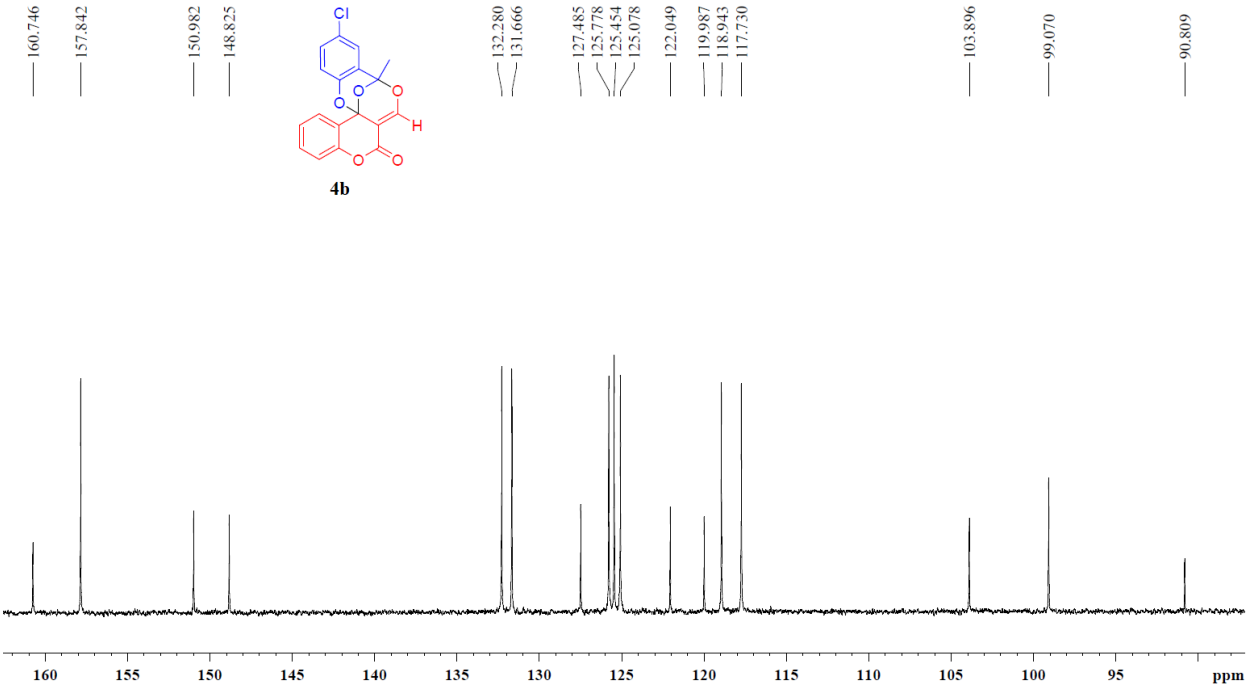
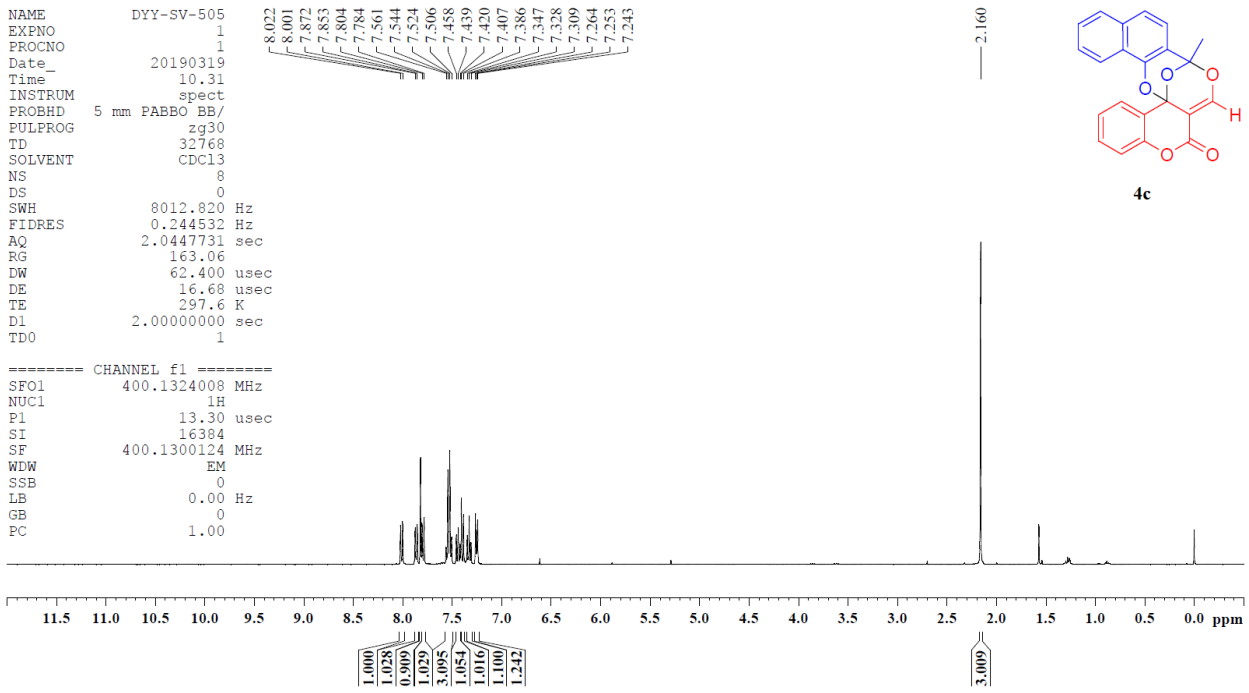
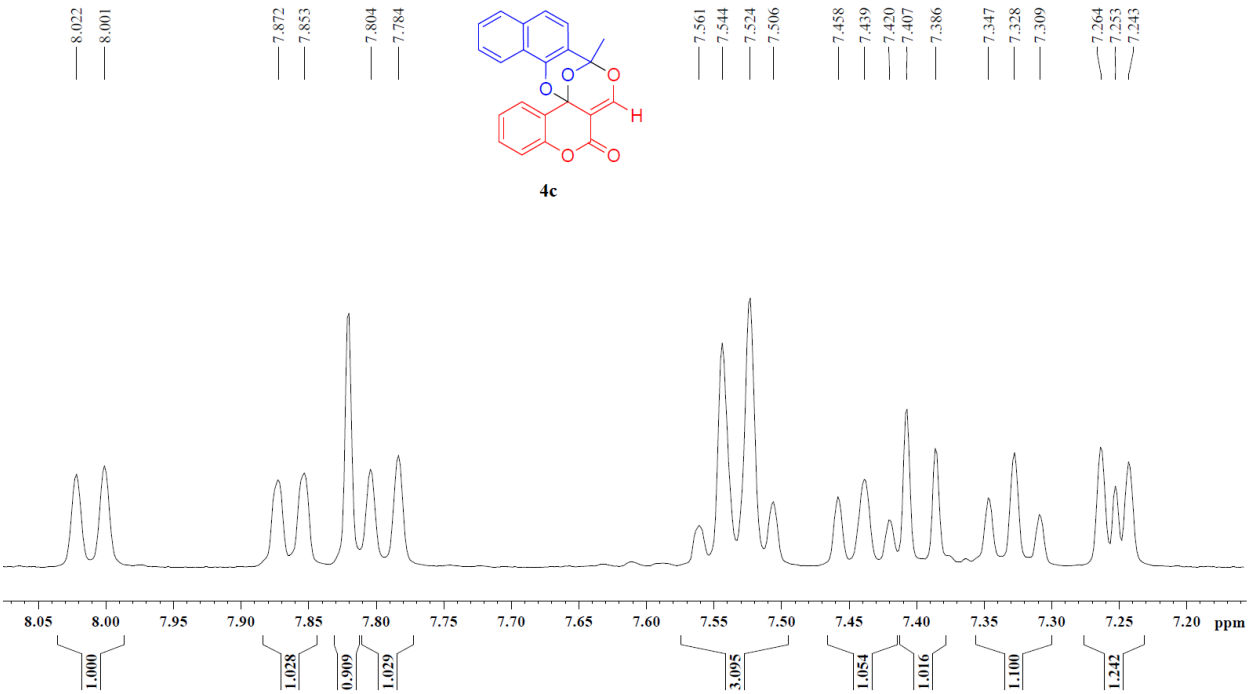
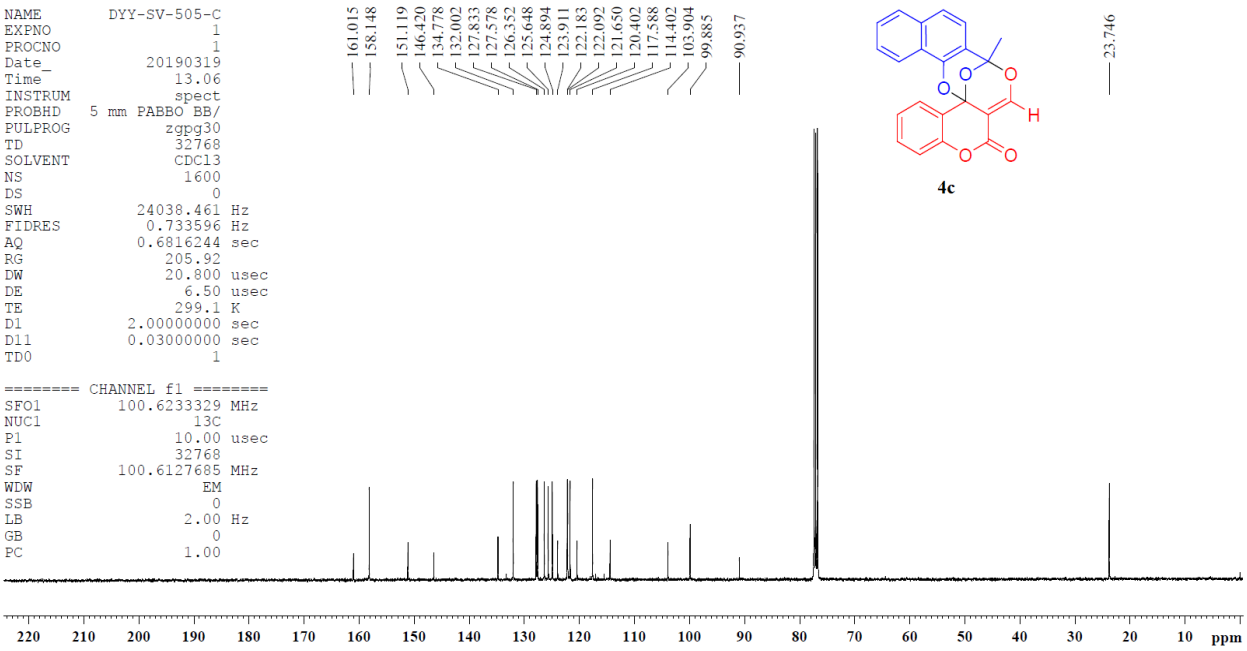
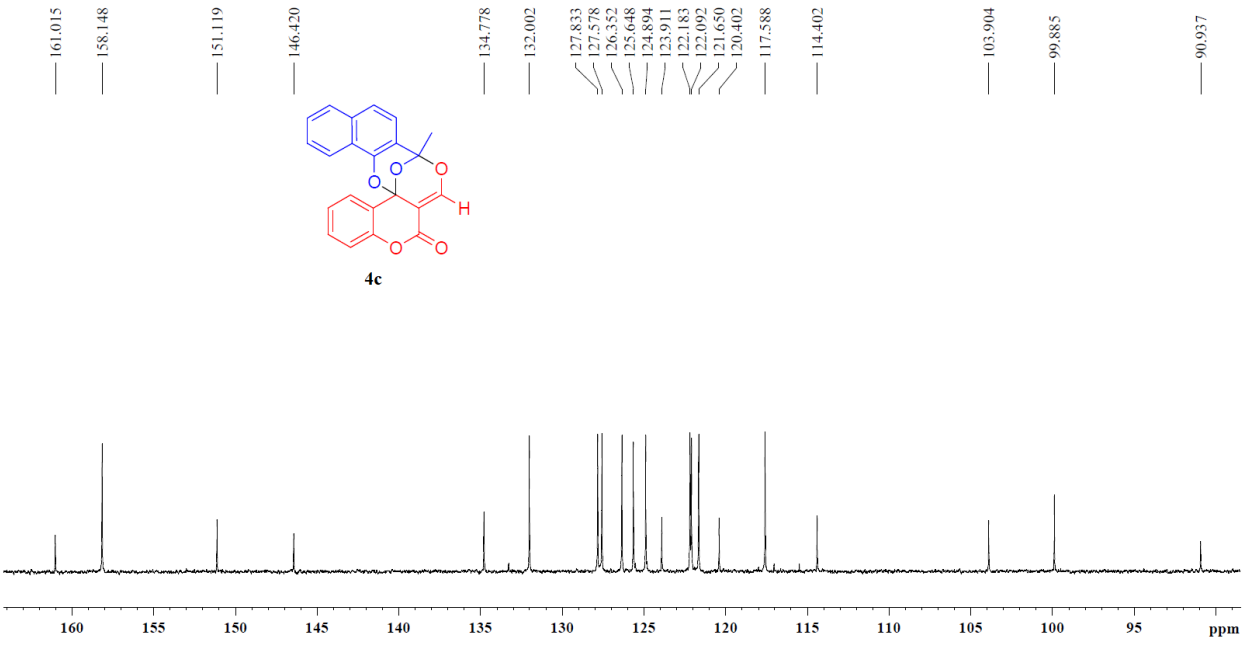
O
4b
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

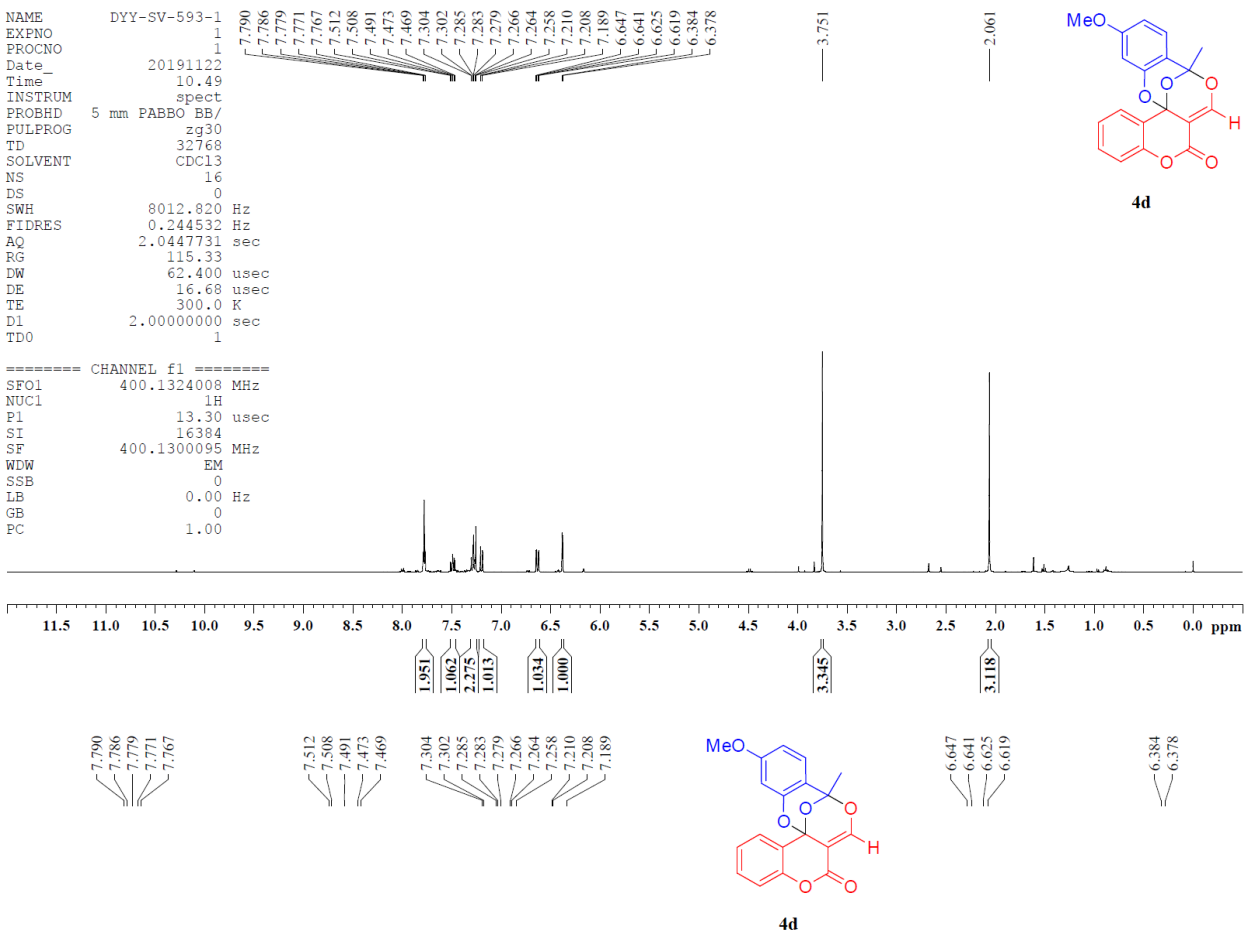

160.746
157.842
150.982
148.825
132.280
131.666
127.485
125.778
125.454
125.078
122.049
119.987
118.943
117.730
103.896
99.070
90.809
Cl
O
O
H
O
O
4b
160 155 150 145 140 135 130 125 120 115 110 105 100 95 ppm


NAME DYY-SV-505
EXPNO 1
PROCNO 1
Date_ 20190319
Time 10.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 163.06
DW 62.400 usec
DE 16.68 usec
TE 297.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300124 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.022
8.001
7.872
7.853
7.804
7.784
7.561
7.544
7.524
7.506
7.458
7.439
7.420
7.407
7.386
7.347
7.328
7.309
7.264
7.253
7.243
2.160
4c
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.000
1.028
0.909
1.029
3.095
1.054
1.016
1.100
1.242
3.009

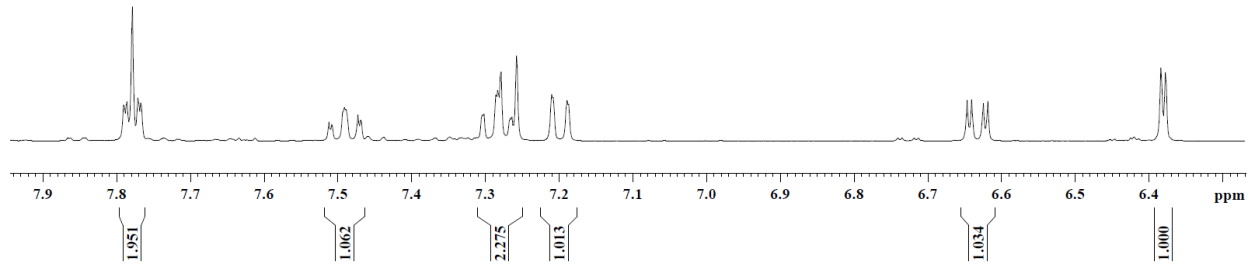

8.022
8.001
7.872
7.853
7.804
7.784
7.561
7.544
7.524
7.506
7.458
7.439
7.420
7.407
7.386
7.347
7.328
7.309
7.264
7.253
7.243
4c
8.05 8.00 7.95 7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 ppm
1.000
1.028
0.909
1.029
3.095
1.054
1.016
1.100
1.242

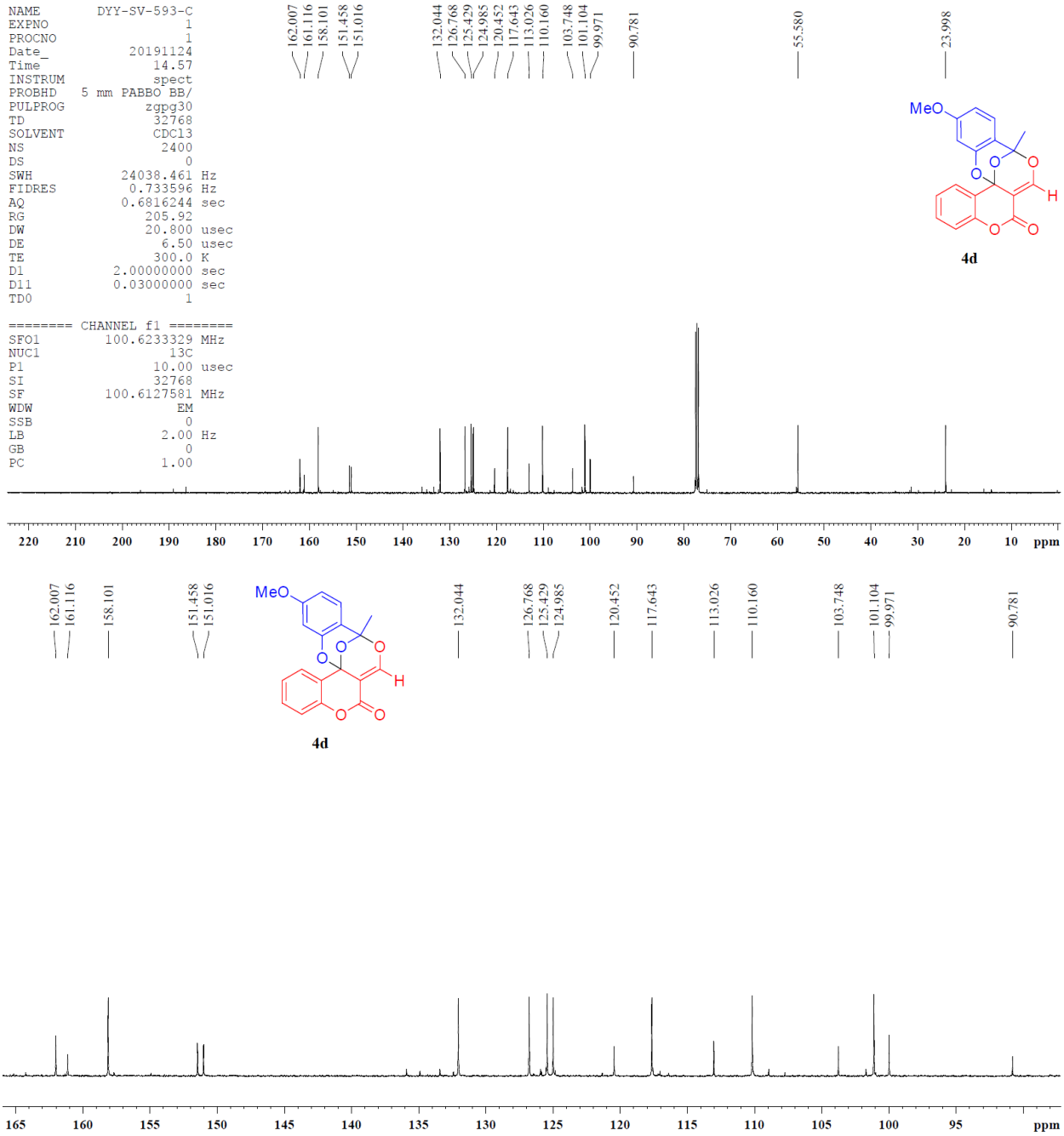

NAME DYY-SV-505-C
EXPNO 1
PROCNO 1
Date_ 20190319
Time 13.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1600
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 205.92
DW 20.800 usec
DE 6.50 usec
TE 299.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
161.015
158.148
151.119
146.420
134.778
132.002
127.833
127.578
126.352
125.648
124.894
123.911
122.183
122.092
121.650
120.402
117.588
114.402
103.904
99.885
90.937
23.746
4c
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


161.015
158.148
151.119
146.420
134.778
132.002
127.833
127.578
126.352
125.648
124.894
123.911
122.183
122.092
121.650
120.402
117.588
114.402
103.904
99.885
90.937
4c
160 155 150 145 140 135 130 125 120 115 110 105 100 95 ppm

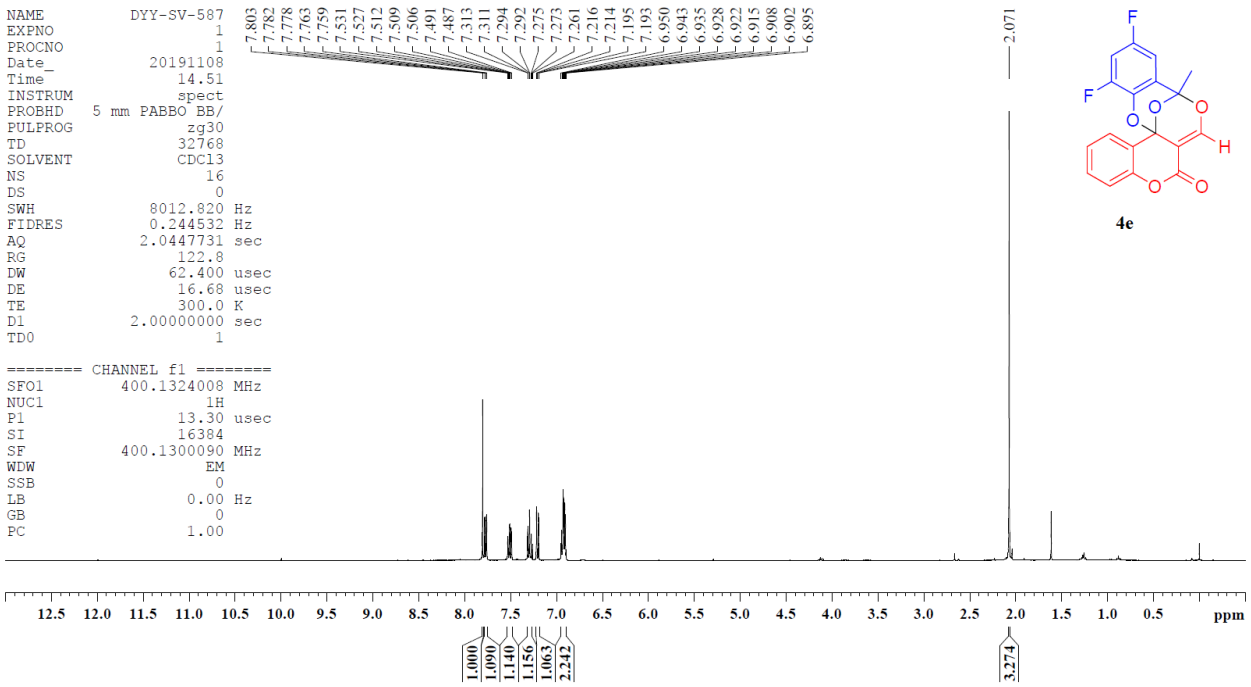

NAME DYY-SV-593-1
EXPNO 1
PROCNO 1
Date_ 20191122
Time 10.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 115.33
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.790
7.786
7.779
7.771
7.767
7.512
7.508
7.491
7.473
7.469
7.304
7.302
7.285
7.283
7.279
7.266
7.264
7.258
7.210
7.208
7.189
6.647
6.641
6.625
6.619
6.384
6.378
3.751
2.061
MeO
O
O
O
H
O
O
4d
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.951
1.062
2.275
1.013
1.034
1.000
3.345
3.118

7.790
7.786
7.779
7.771
7.767
7.512
7.508
7.491
7.473
7.469
7.304
7.302
7.285
7.283
7.279
7.266
7.264
7.258
7.210
7.208
7.189
6.647
6.641
6.625
6.619
6.384
6.378
MeO
O
O
O
H
O
O
4d

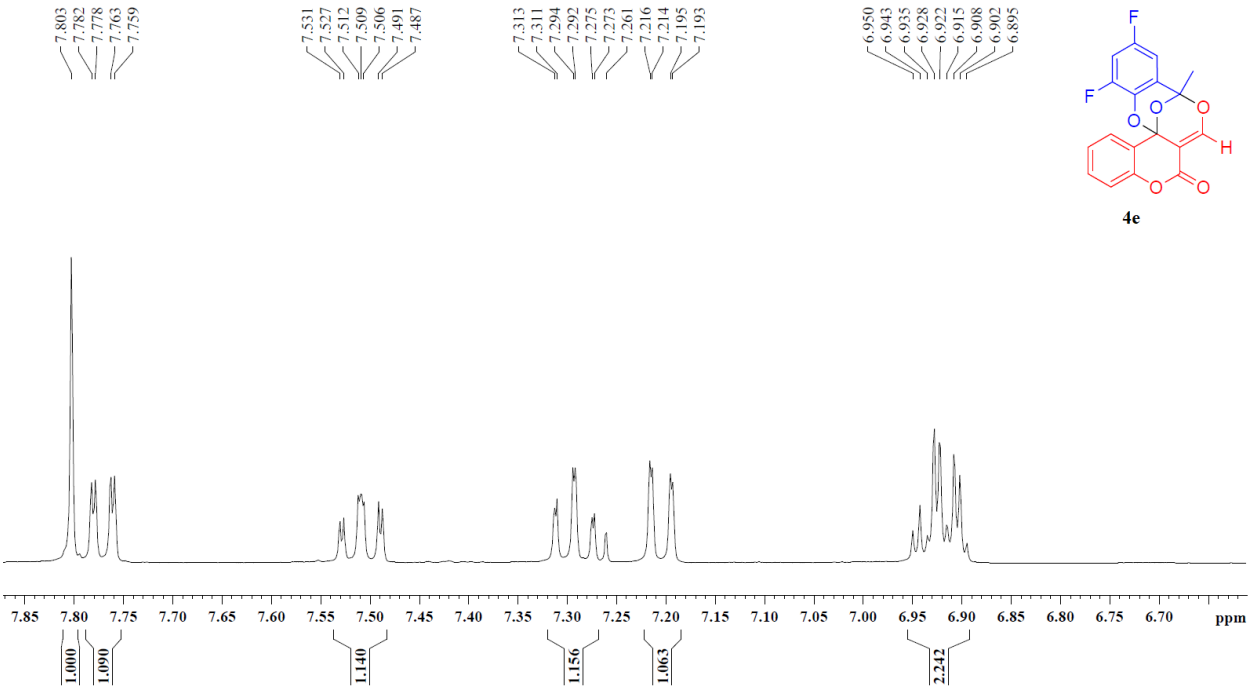

7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 ppm
1.951
1.062
2.275
1.013
1.034
1.000


NAME DYY-SV-593-C
EXPNO 1
PROCNO 1
Date_ 20191124
Time 14.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2400
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 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127581 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
162.007
161.116
158.101
151.458
151.016
132.044
126.768
125.429
124.985
120.452
117.643
113.026
110.160
103.748
101.104
99.971
90.781
55.580
23.998
MeO
O
O
O
H
O
O
4d
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

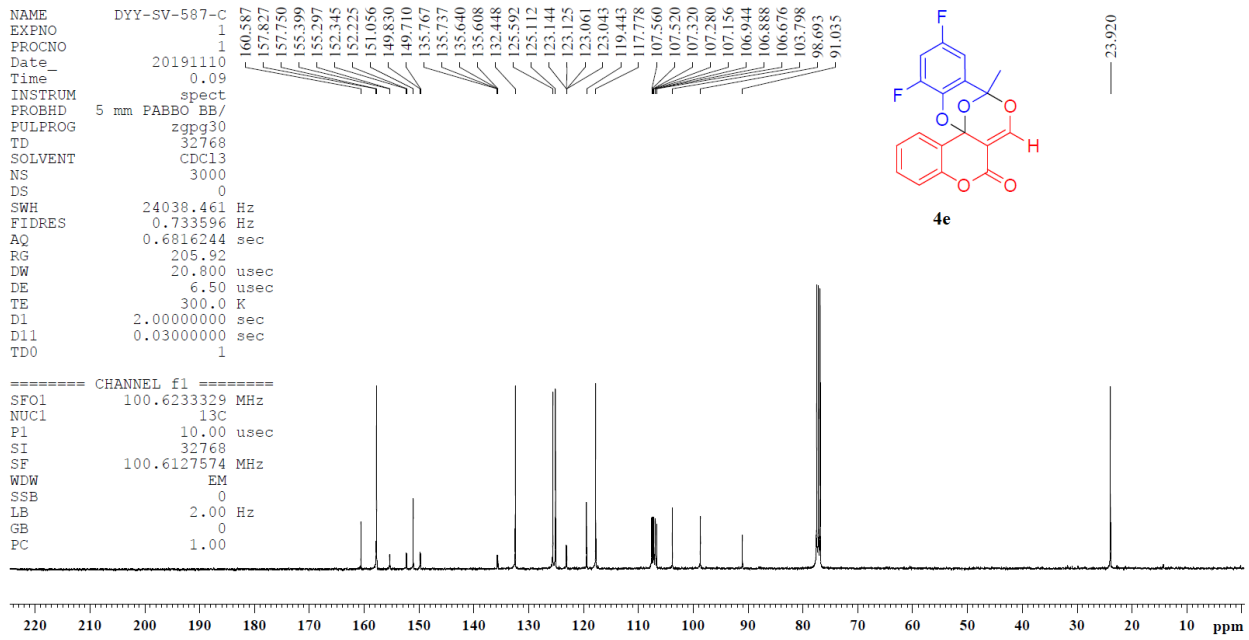
162.007
161.116
158.101
151.458
151.016
132.044
126.768
125.429
124.985
120.452
117.643
113.026
110.160
103.748
101.104
99.971
90.781
MeO
O
O
O
H
O
O
4d
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 ppm

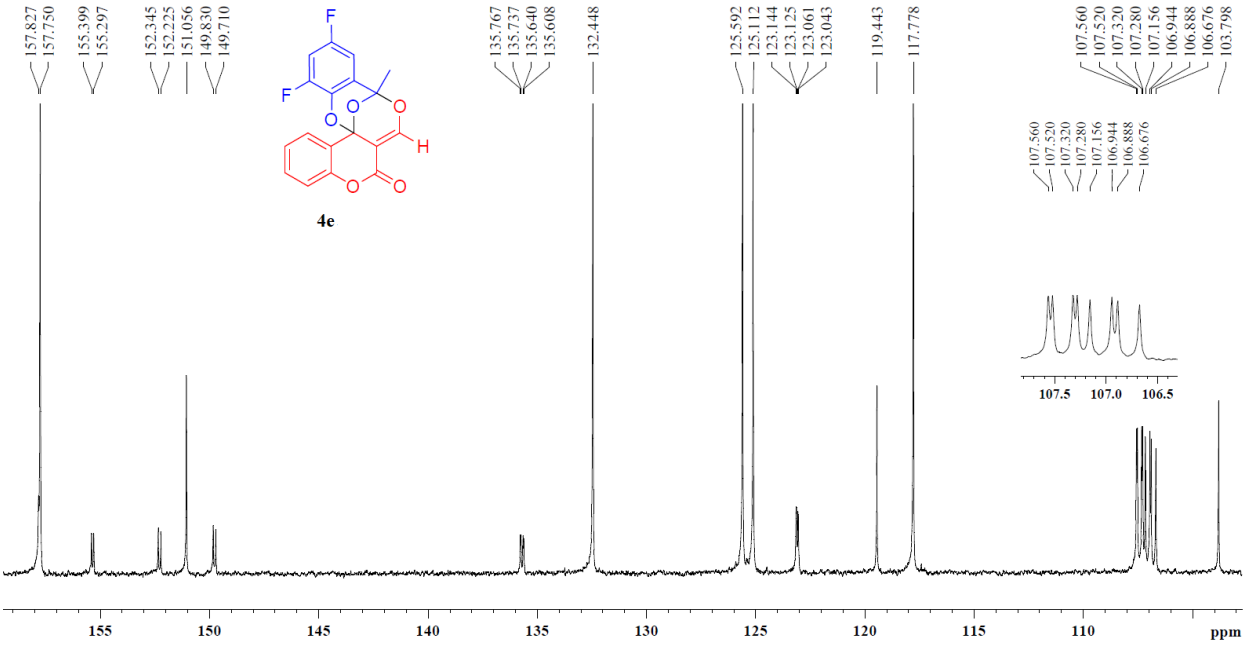

NAME DYY-SV-587
EXPNO 1
PROCNO 1
Date_ 20191108
Time 14.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 122.8
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.803 7.782 7.778 7.763 7.759 7.531 7.527 7.512 7.509 7.506 7.491 7.487 7.313 7.311 7.294 7.292 7.275 7.273 7.261 7.216 7.214 7.195 7.193 6.950 6.943 6.935 6.928 6.922 6.915 6.908 6.902 6.895
2.071
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.000 1.090 1.140 1.156 1.063 2.242
3.274
F F O O O H O O

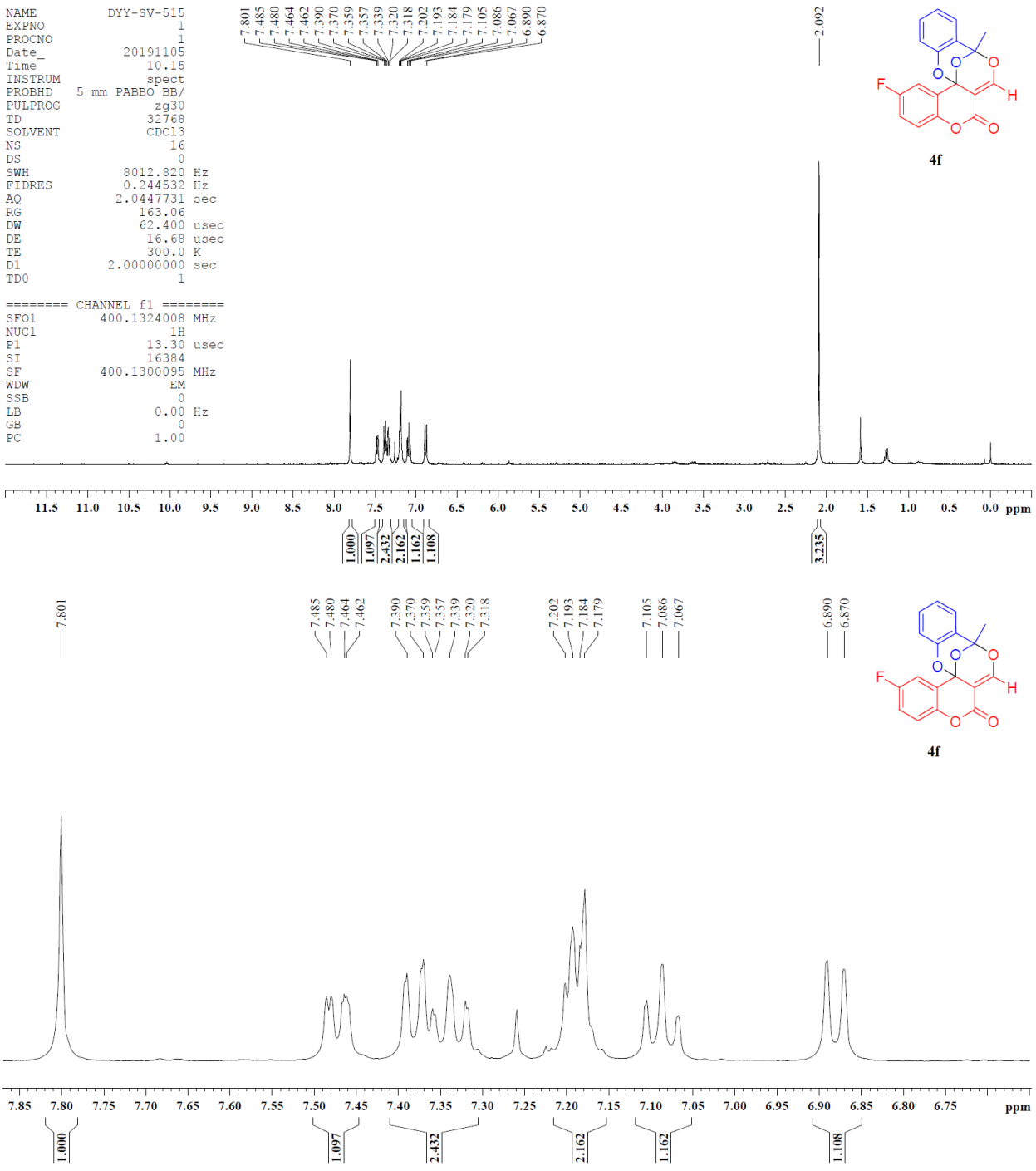
4e


7.803 7.782 7.778 7.763 7.759
7.531 7.527 7.512 7.509 7.506 7.491 7.487
7.313 7.311 7.294 7.292 7.275 7.273 7.261 7.216 7.214 7.195 7.193
6.950 6.943 6.935 6.928 6.922 6.915 6.908 6.902 6.895
7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70 ppm
1.000 1.090 1.140 1.156 1.063 2.242
F F O O O H O O

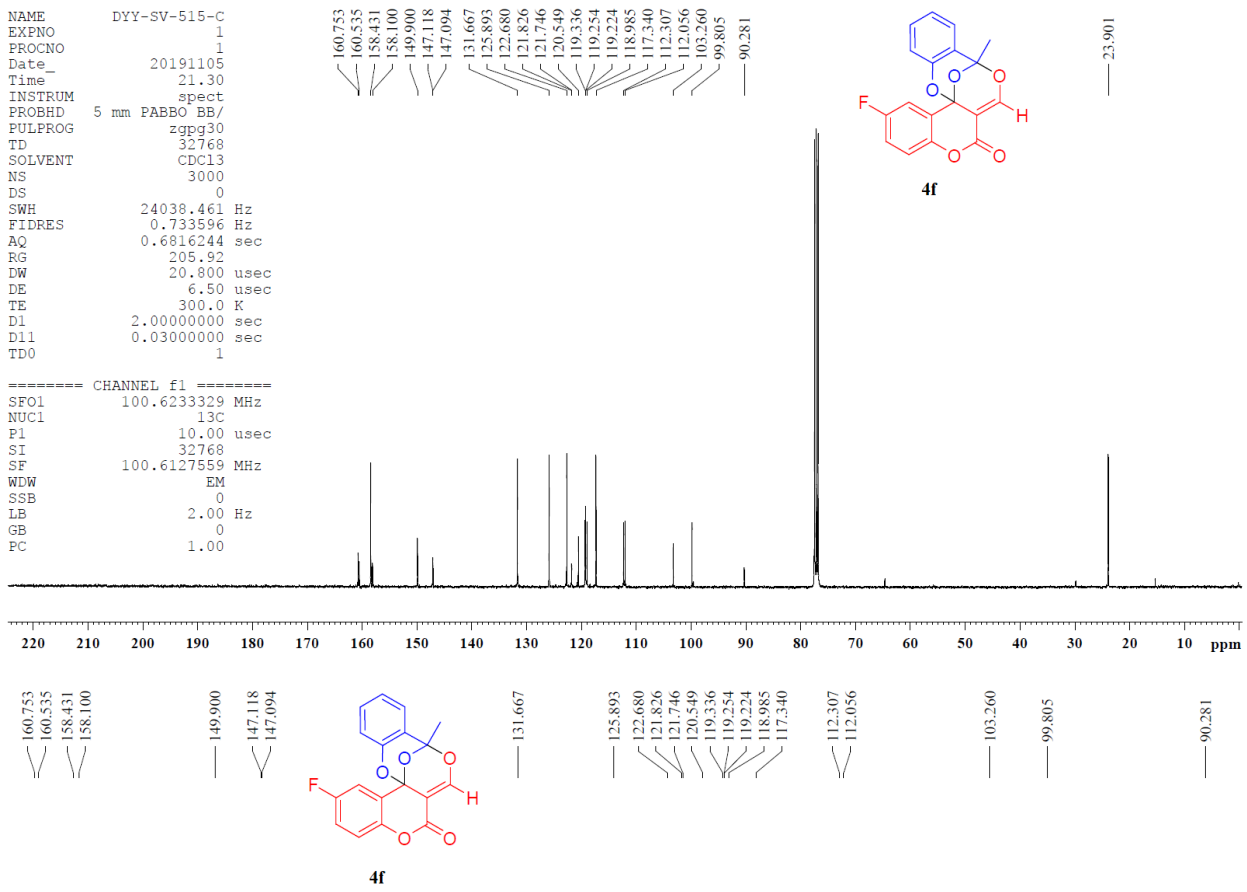
4e

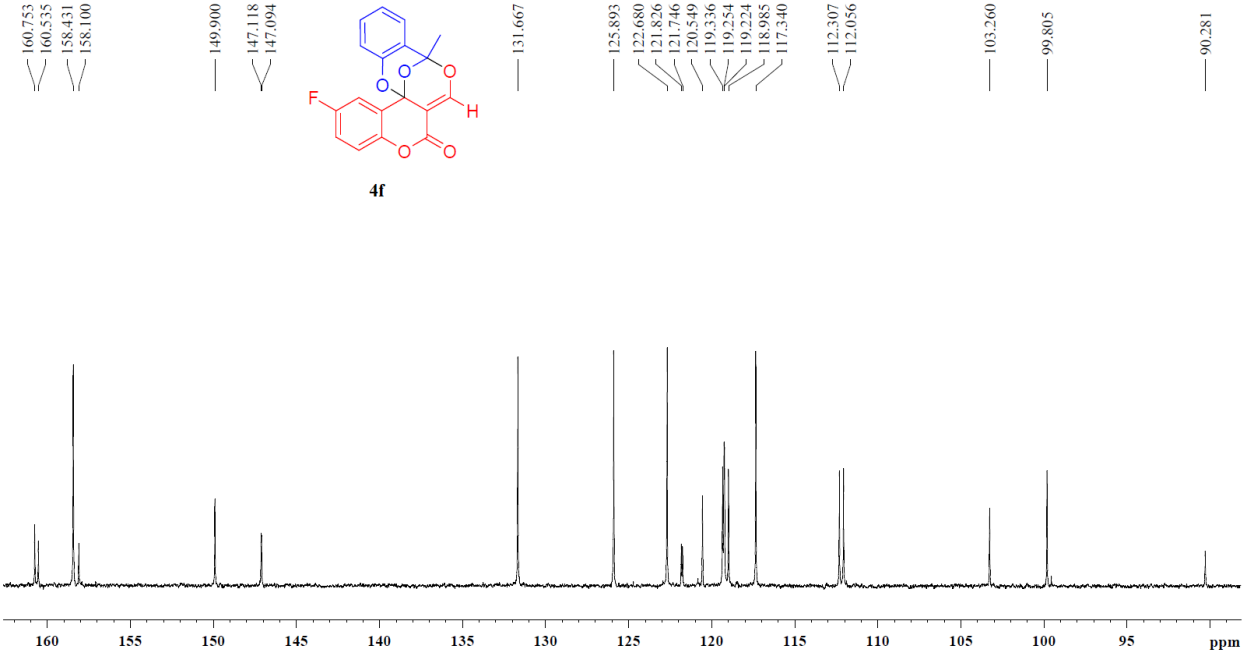

NAME DYY-SV-587-C
EXPNO 1
PROCNO 1
Date_ 20191110
Time 0.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3000
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 205.92
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127574 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
160.587
157.827
157.750
155.399
155.297
152.345
152.225
151.056
149.830
149.710
135.767
135.737
135.640
135.608
132.448
125.592
125.112
123.144
123.125
123.061
123.043
119.443
117.778
107.560
107.520
107.320
107.280
107.156
106.944
106.888
106.676
103.798
98.693
91.035
23.920
4e
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

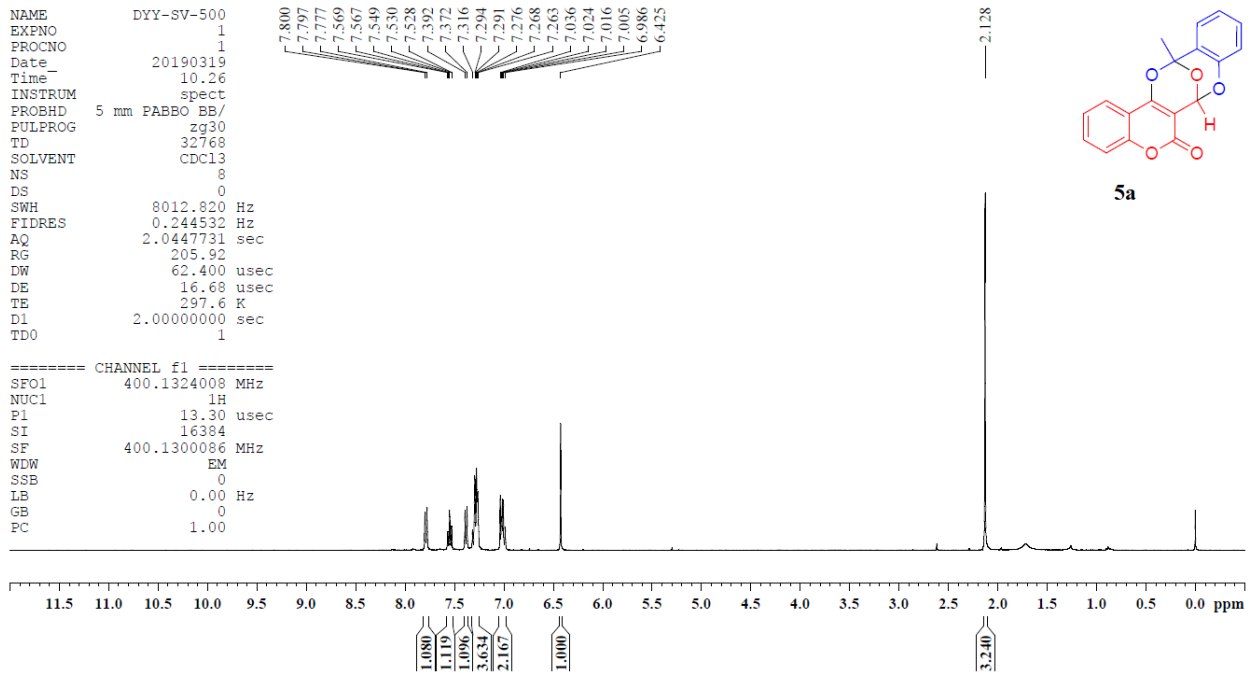

157.827
157.750
155.399
155.297
152.345
152.225
151.056
149.830
149.710
135.767
135.737
135.640
135.608
132.448
125.592
125.112
123.144
123.125
123.061
123.043
119.443
117.778
107.560
107.520
107.320
107.280
107.156
106.944
106.888
106.676
103.798
4e
107.5 107.0 106.5
155 150 145 140 135 130 125 120 115 110 ppm

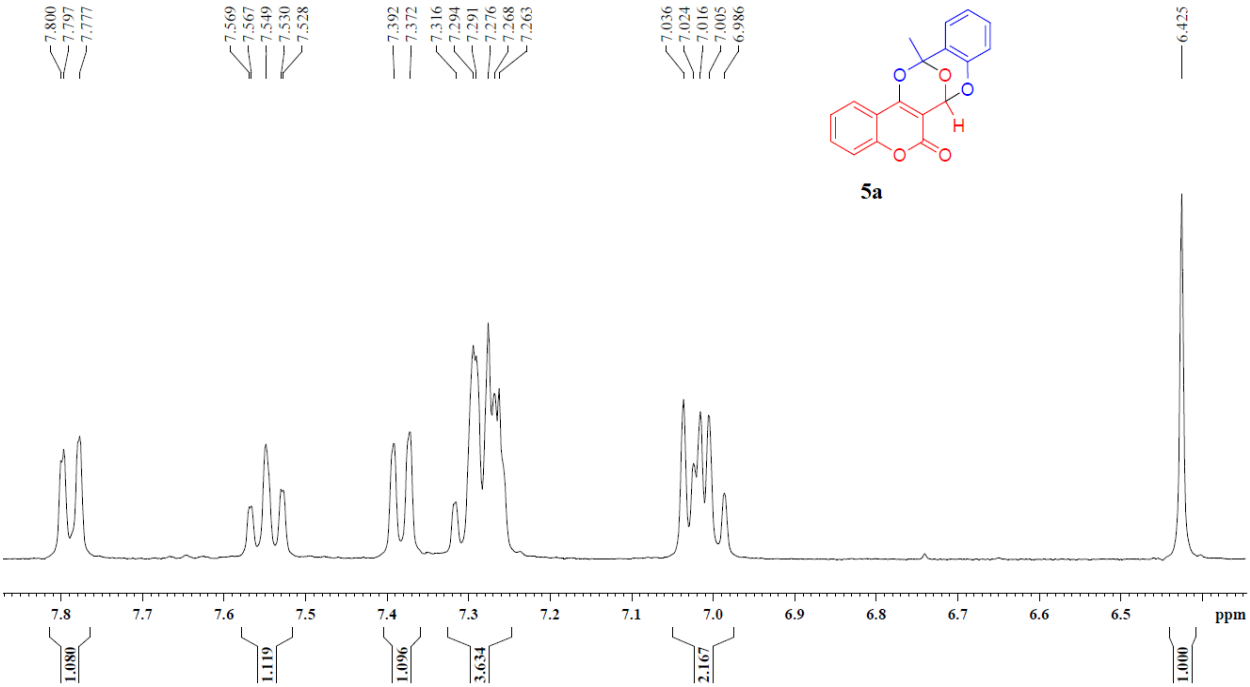

NAME DYY-SV-515
EXPNO 1
PROCNO 1
Date_ 20191105
Time 10.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 163.06
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.801
7.485
7.480
7.464
7.462
7.390
7.370
7.359
7.357
7.339
7.320
7.318
7.202
7.193
7.184
7.179
7.105
7.086
7.067
6.890
6.870
2.092
4f
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.000
1.097
2.432
2.162
1.162
1.108
3.235

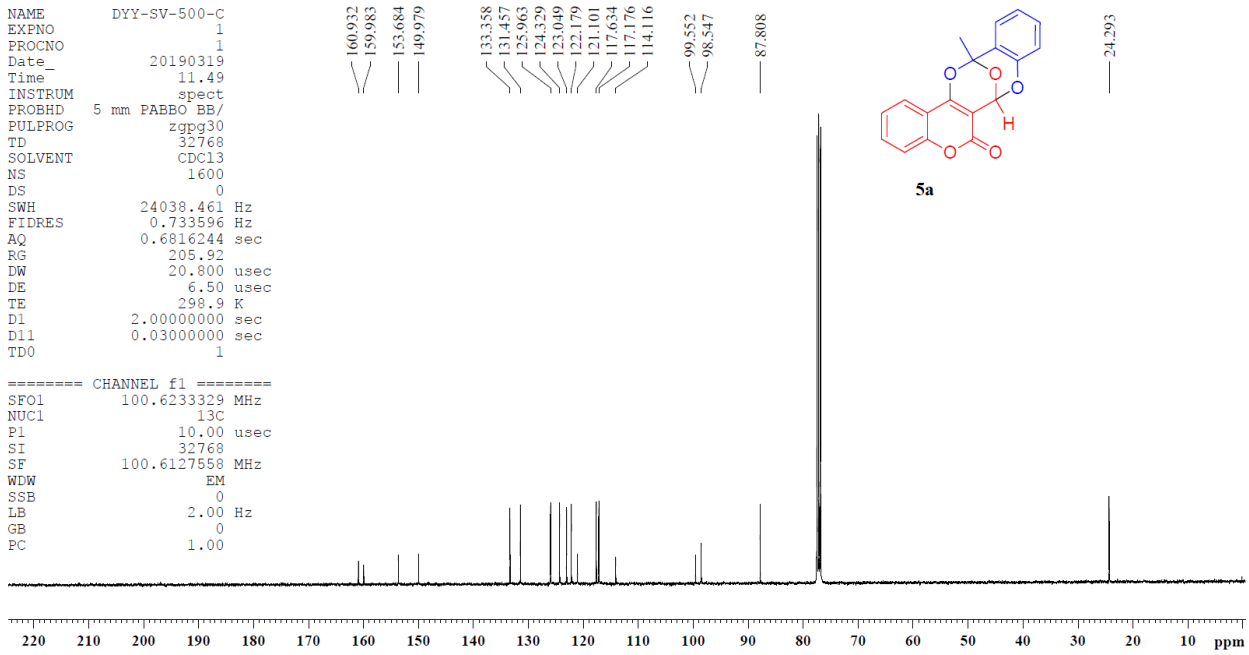
7.801
7.485
7.480
7.464
7.462
7.390
7.370
7.359
7.357
7.339
7.320
7.318
7.202
7.193
7.184
7.179
7.105
7.086
7.067
6.890
6.870
4f
7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 ppm
1.000
1.097
2.432
2.162
1.162
1.108

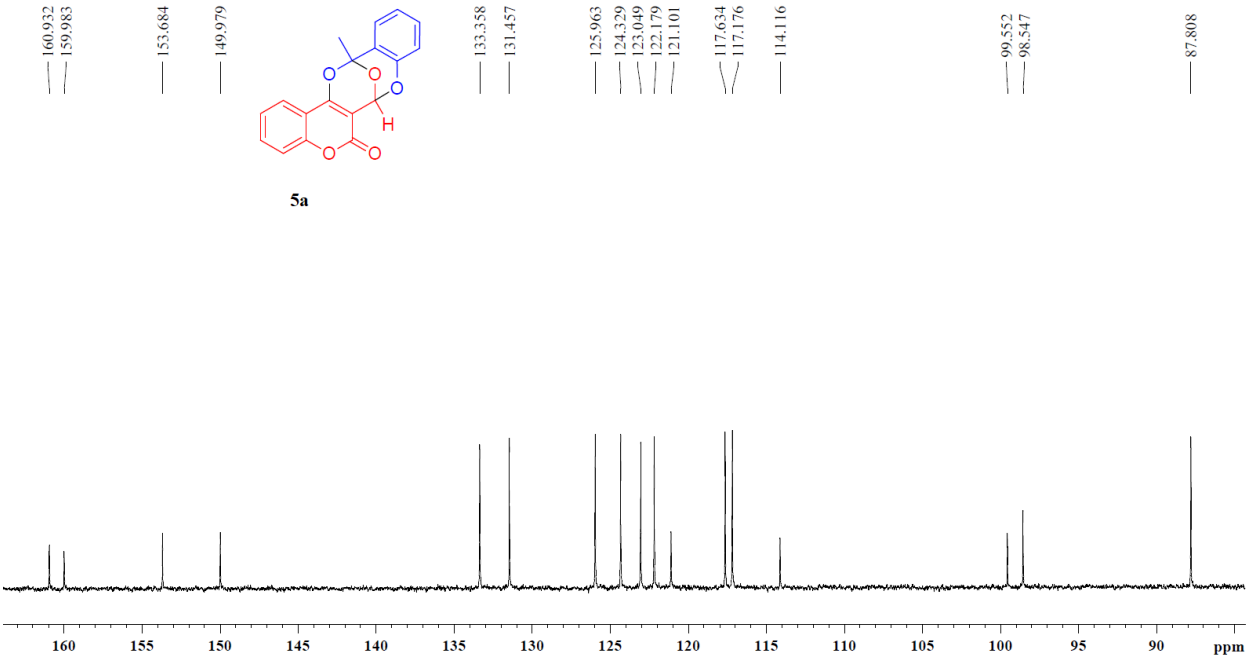

NAME DYY-SV-515-C
EXPNO 1
PROCNO 1
Date_ 20191105
Time 21.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3000
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 205.92
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127559 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
160.753
160.535
158.431
158.100
149.900
147.118
147.094
131.667
125.893
122.680
121.826
121.746
120.549
119.336
119.254
119.224
118.985
117.340
112.307
112.056
103.260
99.805
90.281
23.901
F
H
O
4f
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

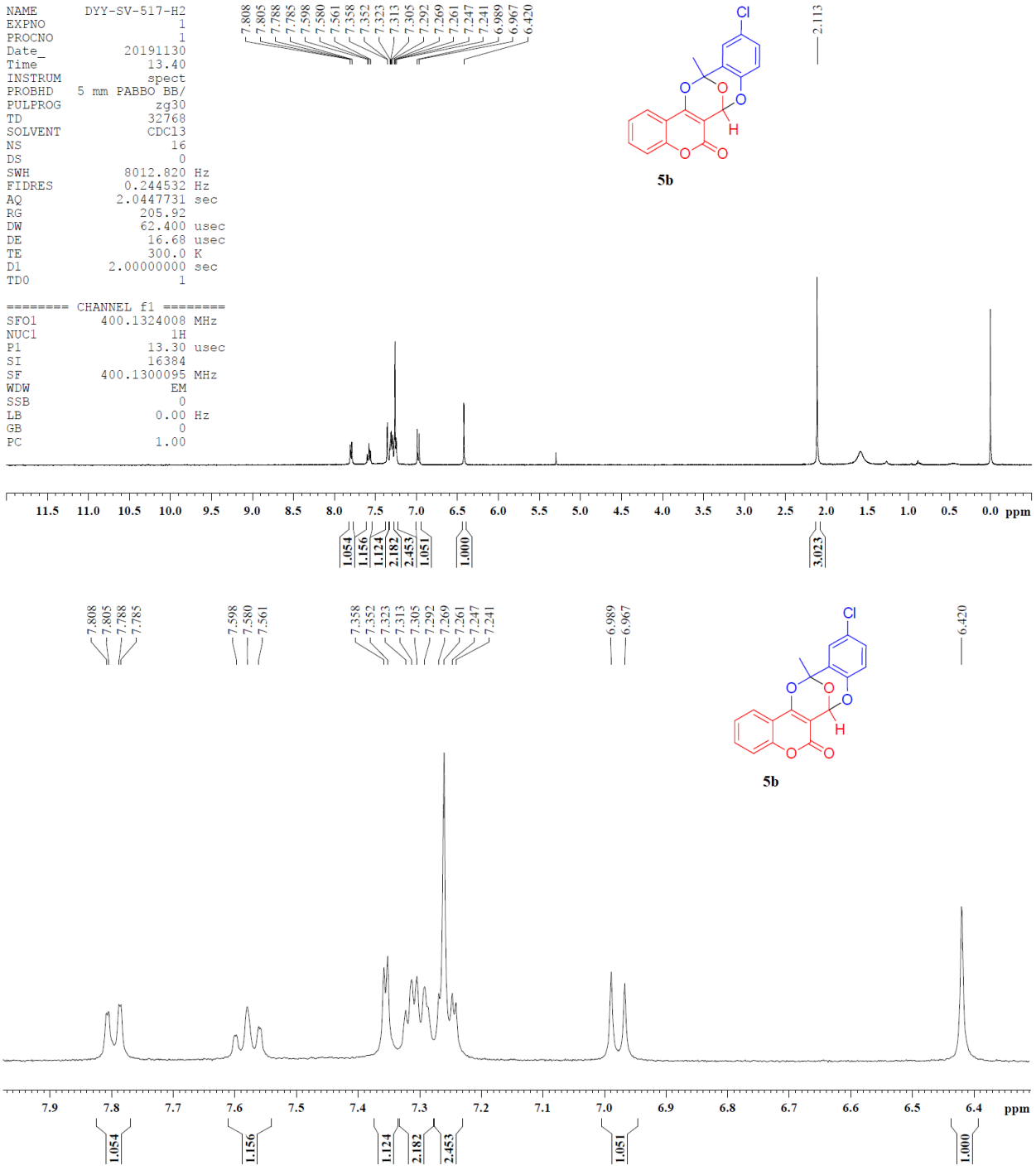

160.753
160.535
158.431
158.100
149.900
147.118
147.094
131.667
125.893
122.680
121.826
121.746
120.549
119.336
119.254
119.224
118.985
117.340
112.307
112.056
103.260
99.805
90.281
F
H
O
4f
160 155 150 145 140 135 130 125 120 115 110 105 100 95 ppm

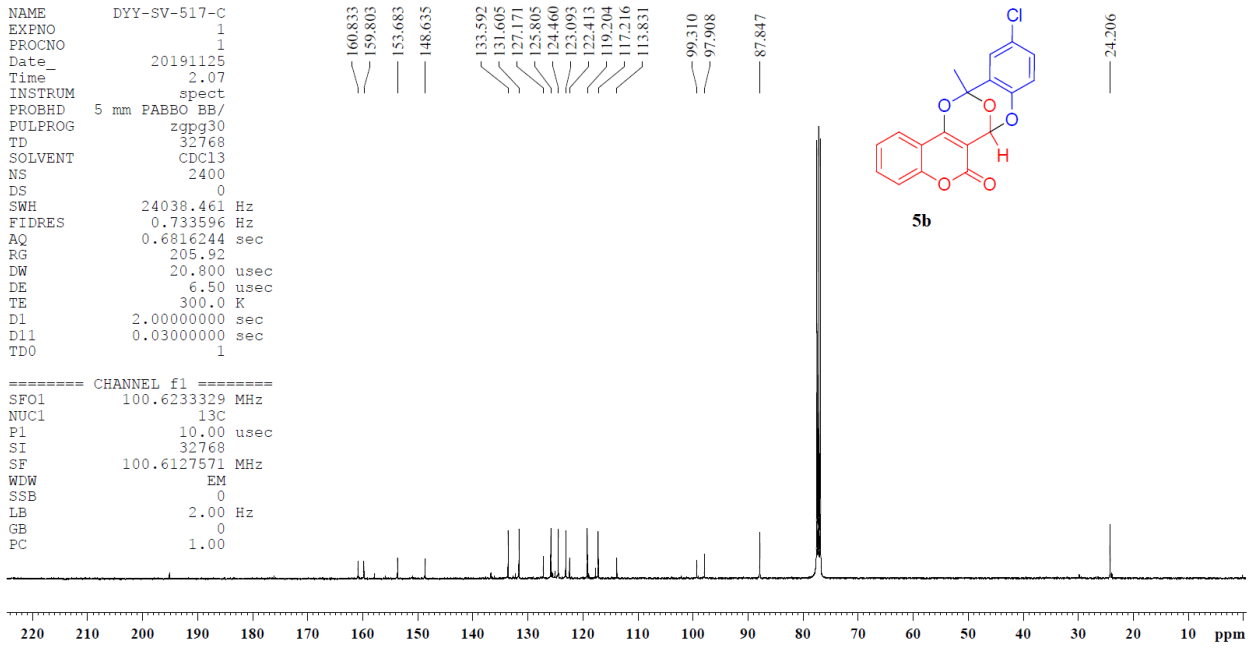

NAME DYY-SV-500
EXPNO 1
PROCNO 1
Date_ 20190319
Time 10.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 205.92
DW 62.400 usec
DE 16.68 usec
TE 297.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.800
7.797
7.777
7.569
7.567
7.549
7.530
7.528
7.392
7.372
7.316
7.294
7.291
7.276
7.268
7.263
7.036
7.024
7.016
7.005
6.986
6.425
2.128
5a
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.080
1.119
1.096
3.634
2.167
1.000
3.240


7.800
7.797
7.777
7.569
7.567
7.549
7.530
7.528
7.392
7.372
7.316
7.294
7.291
7.276
7.268
7.263
7.036
7.024
7.016
7.005
6.986
6.425
5a
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 ppm
1.080
1.119
1.096
3.634
2.167
1.000

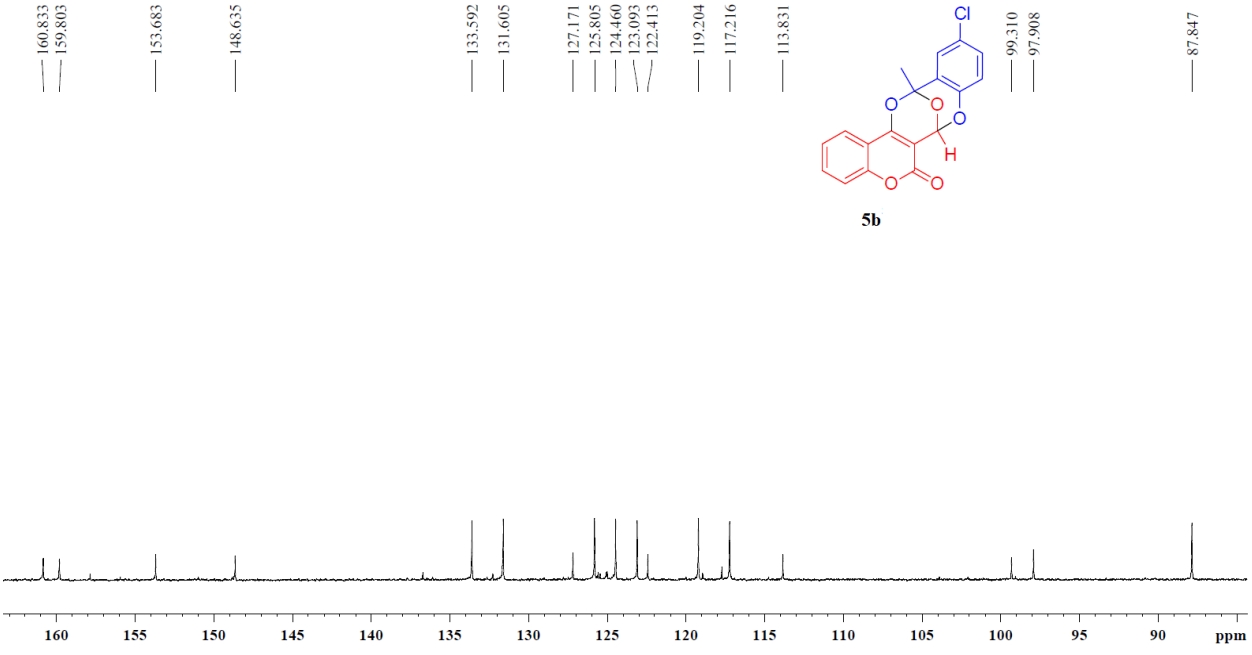

NAME DYY-SV-500-C
EXPNO 1
PROCNO 1
Date_ 20190319
Time 11.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1600
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 205.92
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127558 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
160.932
159.983
153.684
149.979
133.358
131.457
125.963
124.329
123.049
122.179
121.101
117.634
117.176
114.116
99.552
98.547
87.808
24.293
5a
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


160.932
159.983
153.684
149.979
133.358
131.457
125.963
124.329
123.049
122.179
121.101
117.634
117.176
114.116
99.552
98.547
87.808
5a
160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 ppm

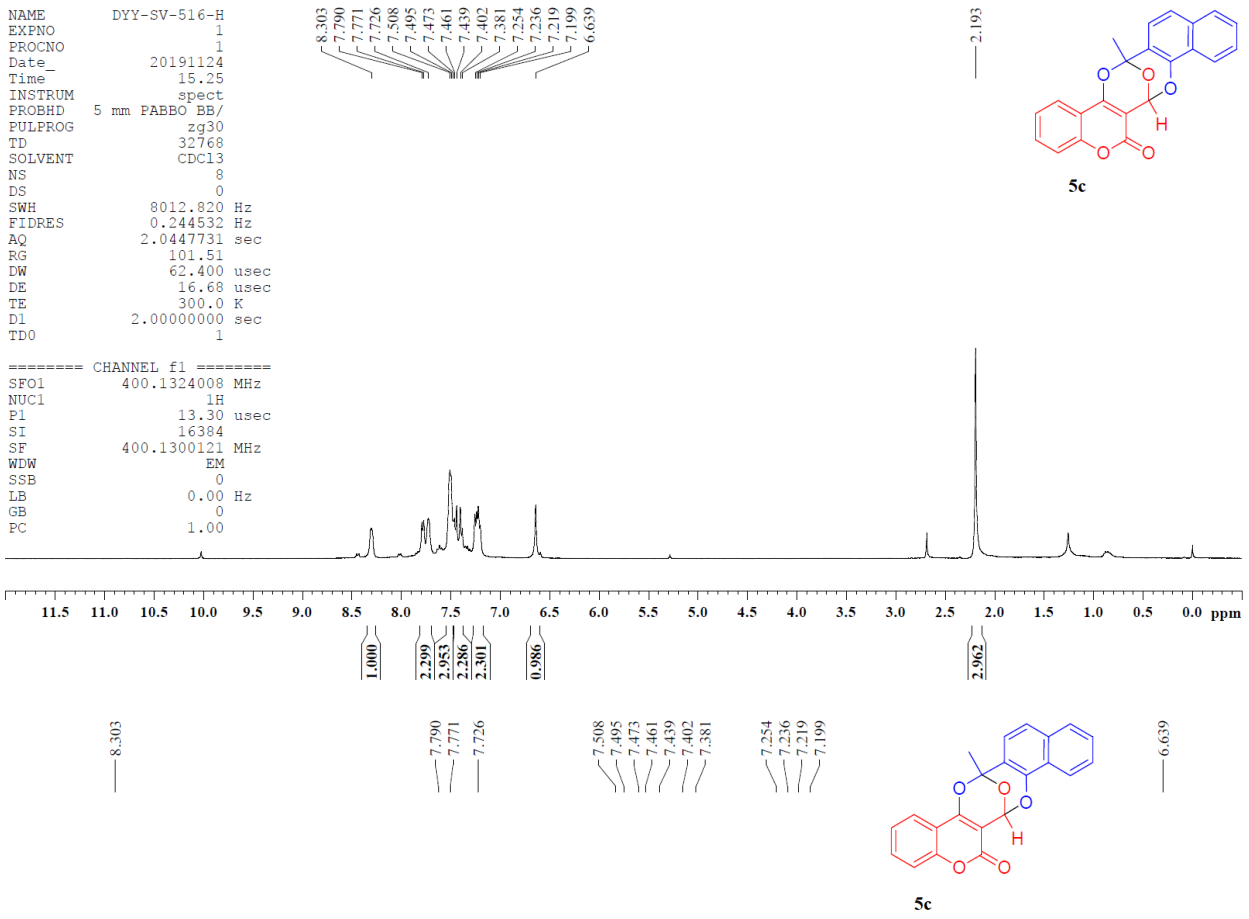

NAME DYY-SV-517-H2
EXPNO 1
PROCNO 1
Date_ 20191130
Time 13.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 205.92
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.808
7.805
7.788
7.785
7.598
7.580
7.561
7.358
7.352
7.323
7.313
7.305
7.292
7.269
7.261
7.247
7.241
6.989
6.967
6.420
2.113
Cl
O
O
O
H
O
O
5b
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.054
1.156
1.124
2.182
2.453
1.051
1.000
3.023
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 ppm
1.054
1.156
1.124
2.182
2.453
1.051
1.000

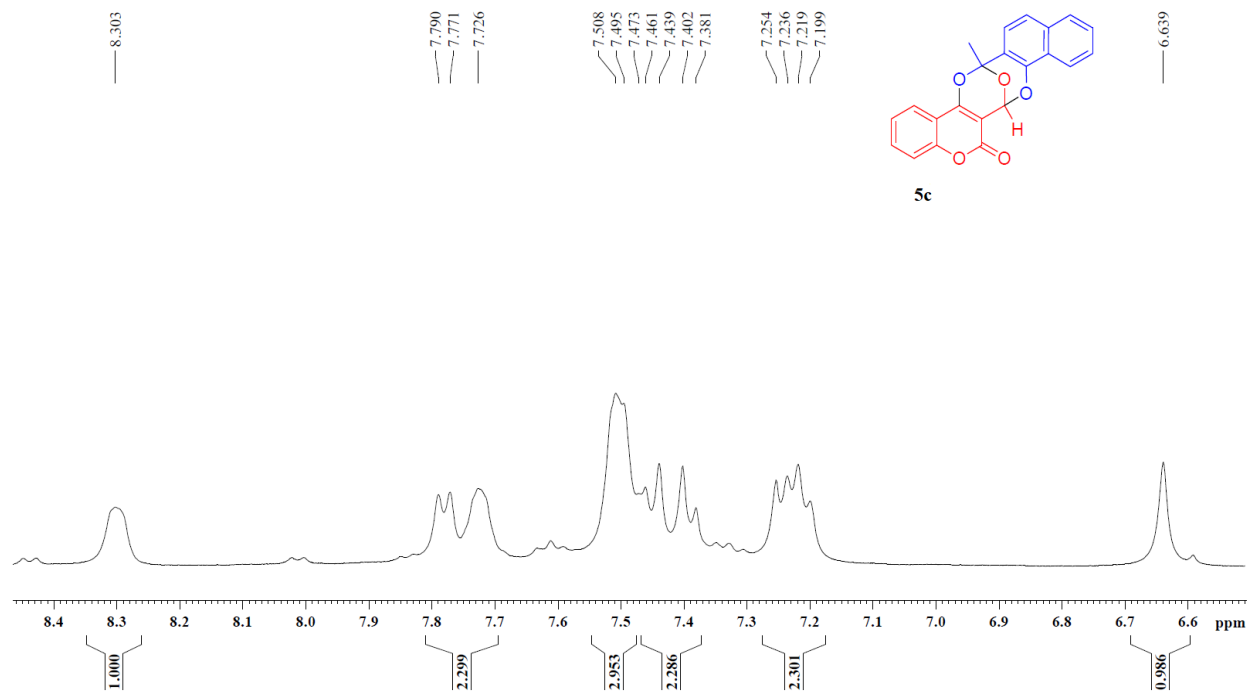

NAME DYY-SV-517-C
EXPNO 1
PROCNO 1
Date_ 20191125
Time 2.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2400
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 205.92
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127571 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
160.833
159.803
153.683
148.635
133.592
131.605
127.171
125.805
124.460
123.093
122.413
119.204
117.216
113.831
99.310
97.908
87.847
24.206
Cl
O
O
O
H
O
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

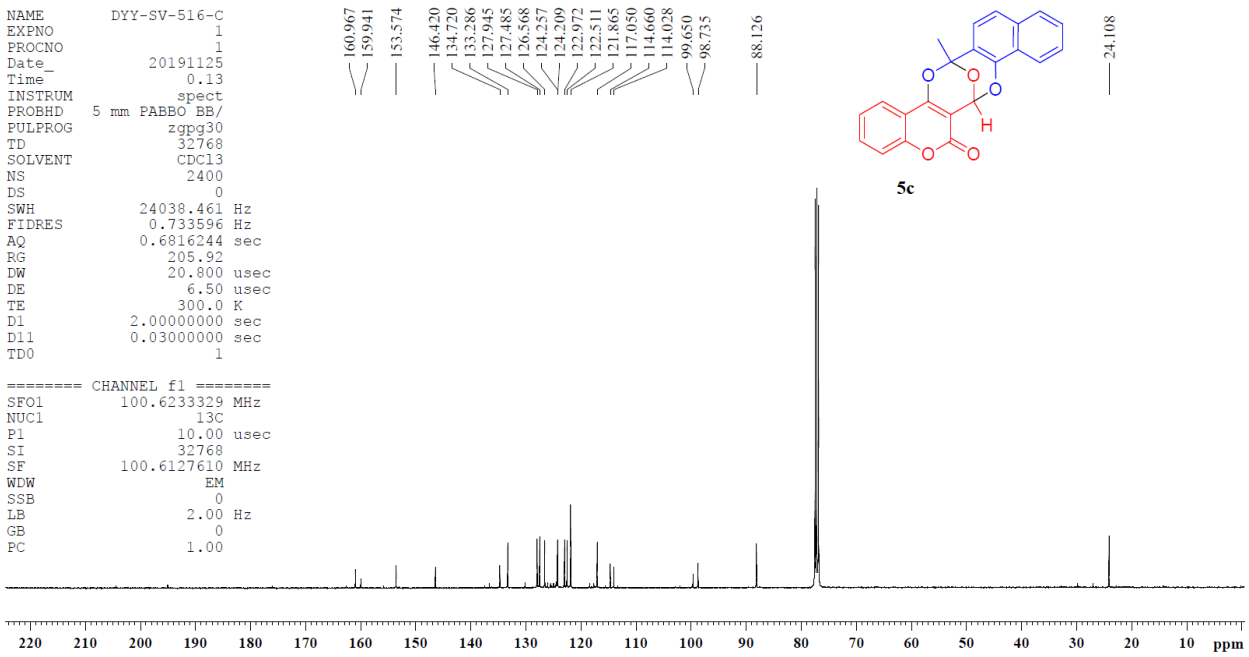
5b

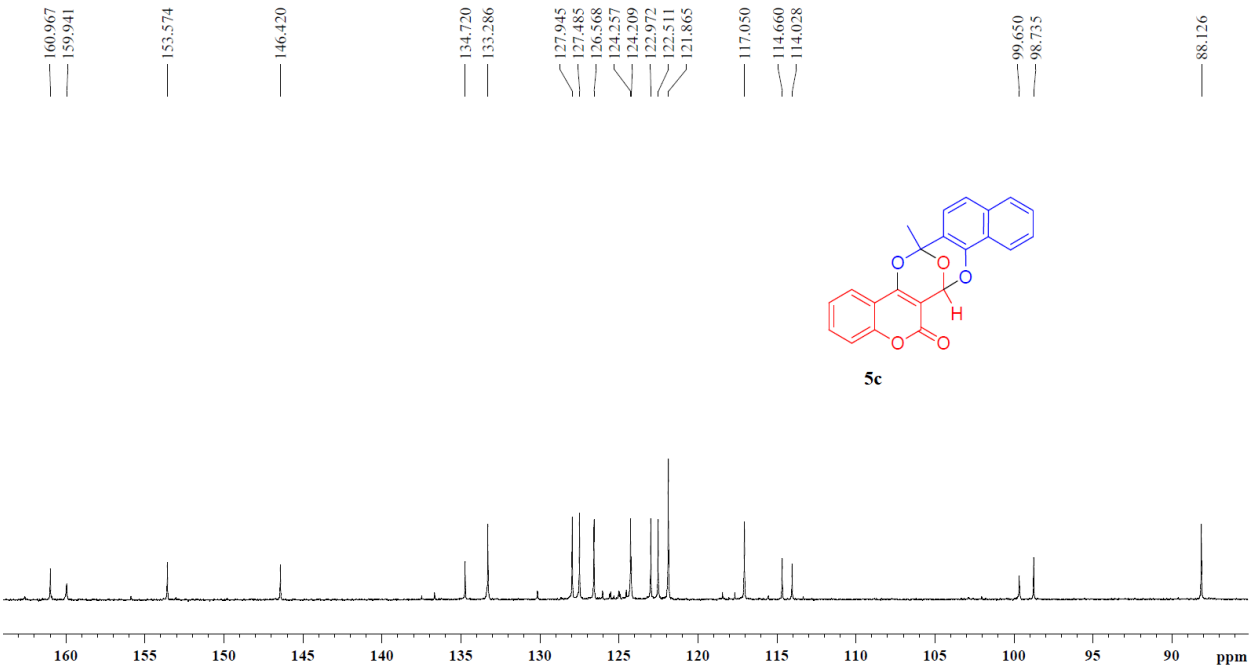

160.833
159.803
153.683
148.635
133.592
131.605
127.171
125.805
124.460
123.093
122.413
119.204
117.216
113.831
99.310
97.908
87.847
Cl
O
O
O
H
O
O
160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 ppm

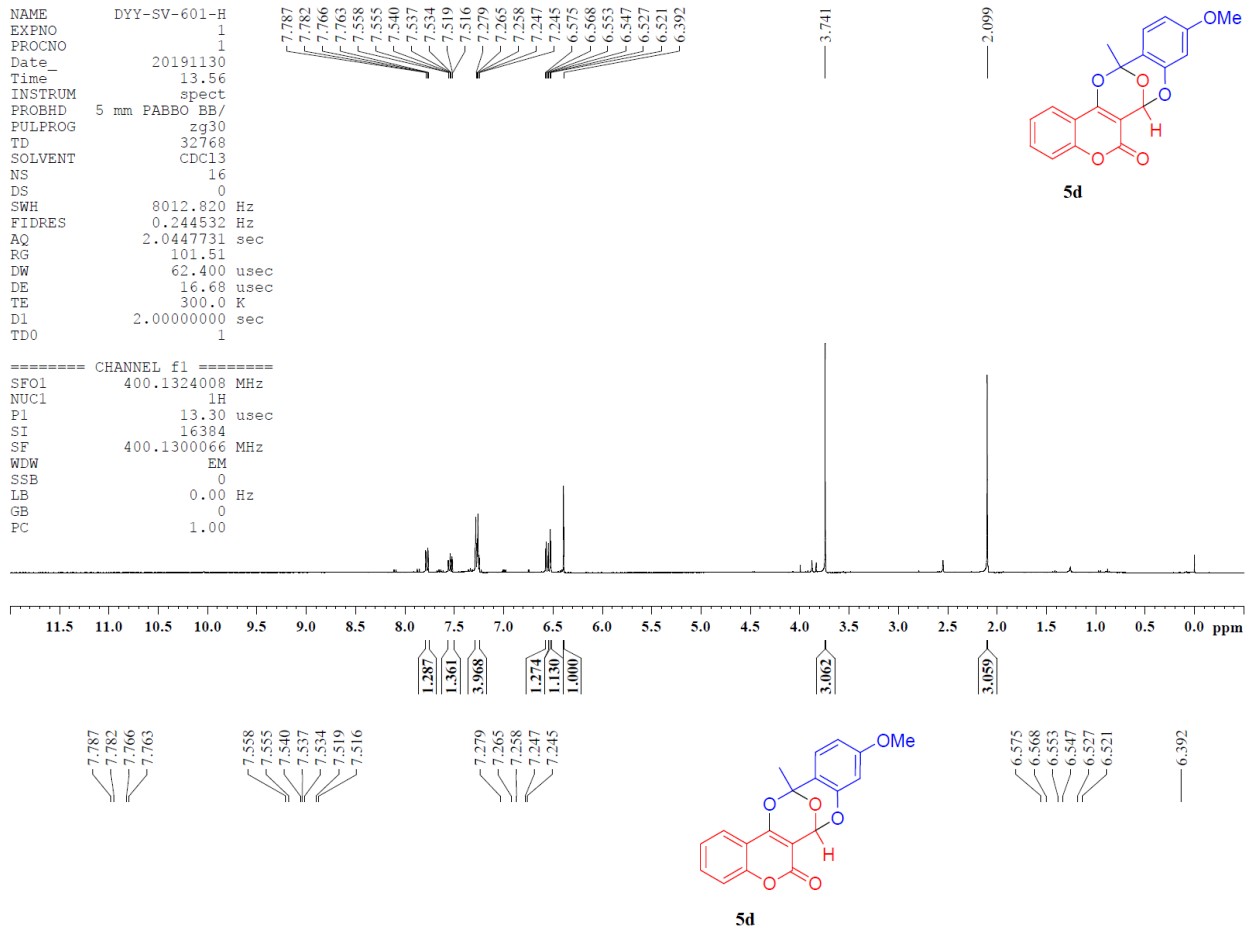
5b


NAME DYY-SV-516-H
EXPNO 1
PROCNO 1
Date_ 20191124
Time 15.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 101.51
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300121 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.303
7.790
7.771
7.726
7.508
7.495
7.473
7.461
7.439
7.402
7.381
7.254
7.236
7.219
7.199
6.639
2.193
5c
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.000
2.299
2.953
2.286
2.301
0.986
2.962


8.303
7.790
7.771
7.726
7.508
7.495
7.473
7.461
7.439
7.402
7.381
7.254
7.236
7.219
7.199
6.639
5c
8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 ppm
1.000
2.299
2.953
2.286
2.301
0.986


NAME DYY-SV-516-C
EXPNO 1
PROCNO 1
Date_ 20191125
Time 0.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2400
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 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127610 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
160.967
159.941
153.574
146.420
134.720
133.286
127.945
127.485
126.568
124.257
124.209
122.972
122.511
121.865
117.050
114.660
114.028
99.650
98.735
88.126
24.108
5c
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

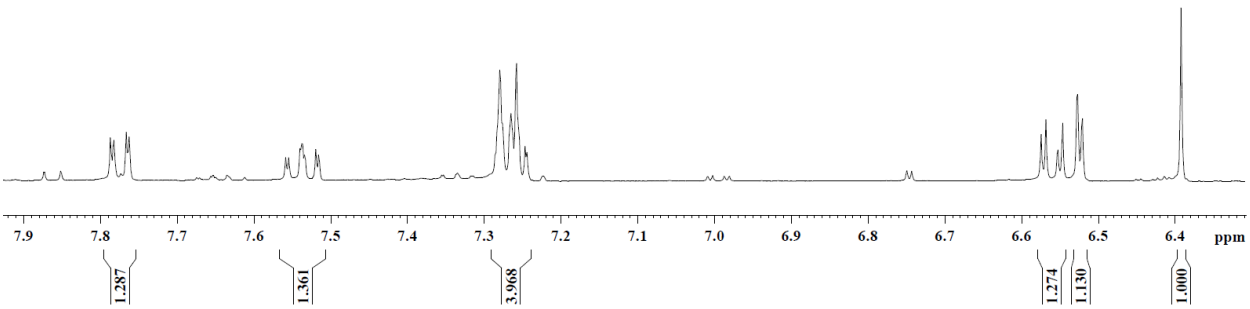

160.967
159.941
153.574
146.420
134.720
133.286
127.945
127.485
126.568
124.257
124.209
122.972
122.511
121.865
117.050
114.660
114.028
99.650
98.735
88.126
5c
160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 ppm

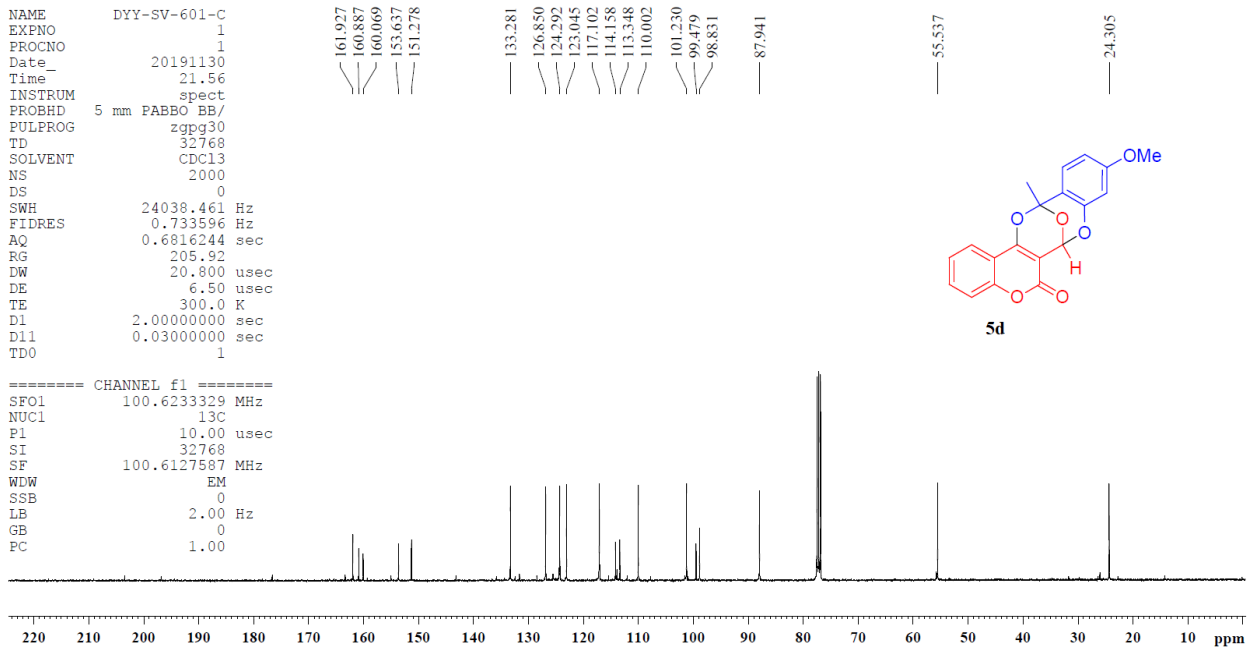

NAME DYY-SV-601-H
EXPNO 1
PROCNO 1
Date_ 20191130
Time 13.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 101.51
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300066 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.787
7.782
7.766
7.763
7.558
7.555
7.540
7.537
7.534
7.519
7.516
7.279
7.265
7.258
7.247
7.245
6.575
6.568
6.553
6.547
6.527
6.521
6.392
3.741
2.099
OMe
O
O
O
H
O
O
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.287
1.361
3.968
1.274
1.130
1.000
3.062
3.059

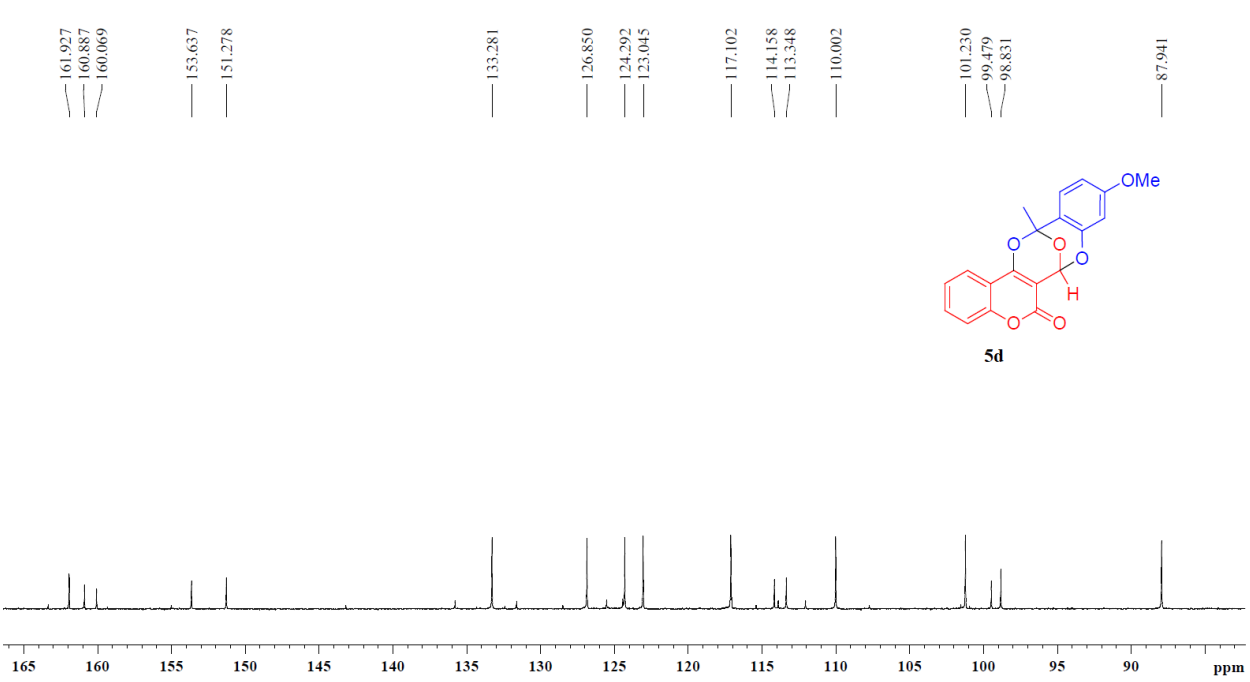
5d

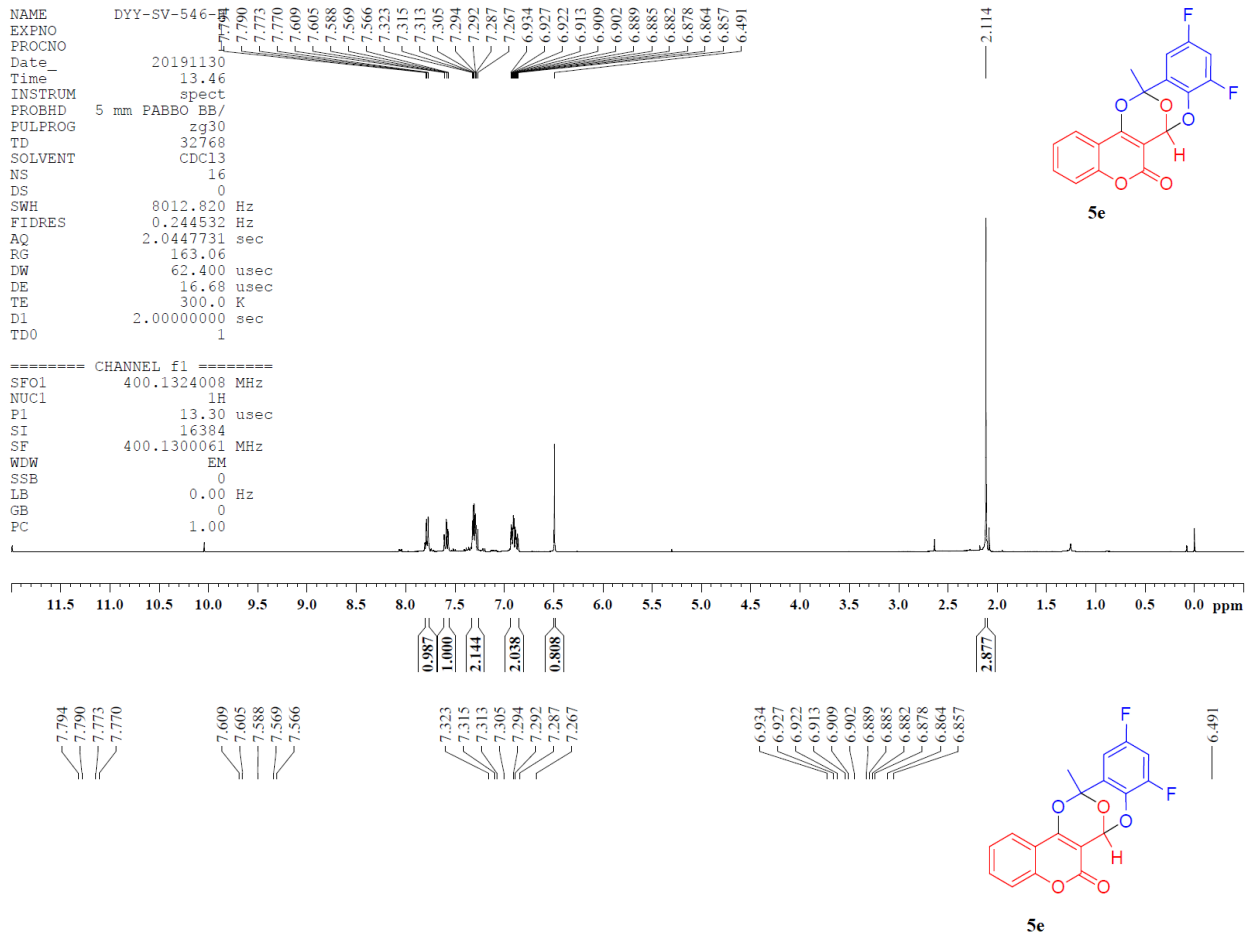
7.787
7.782
7.766
7.763
7.558
7.555
7.540
7.537
7.534
7.519
7.516
7.279
7.265
7.258
7.247
7.245
6.575
6.568
6.553
6.547
6.527
6.521
6.392
OMe
O
O
O
H
O
O

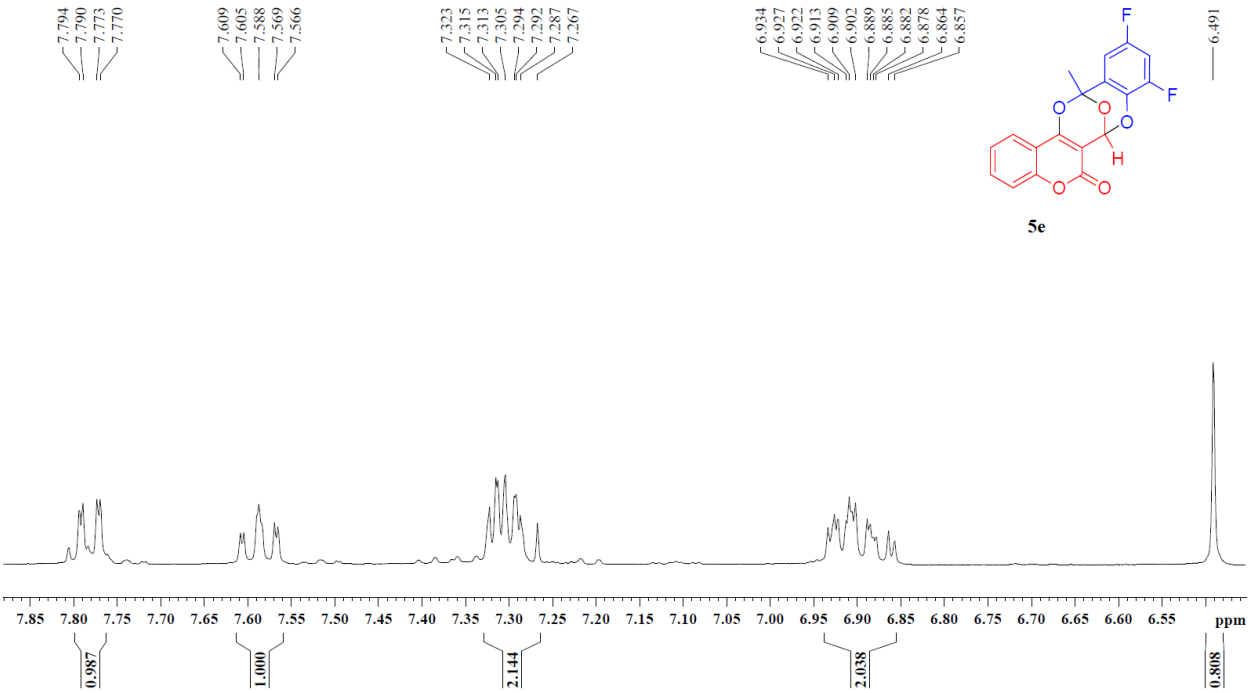
5d

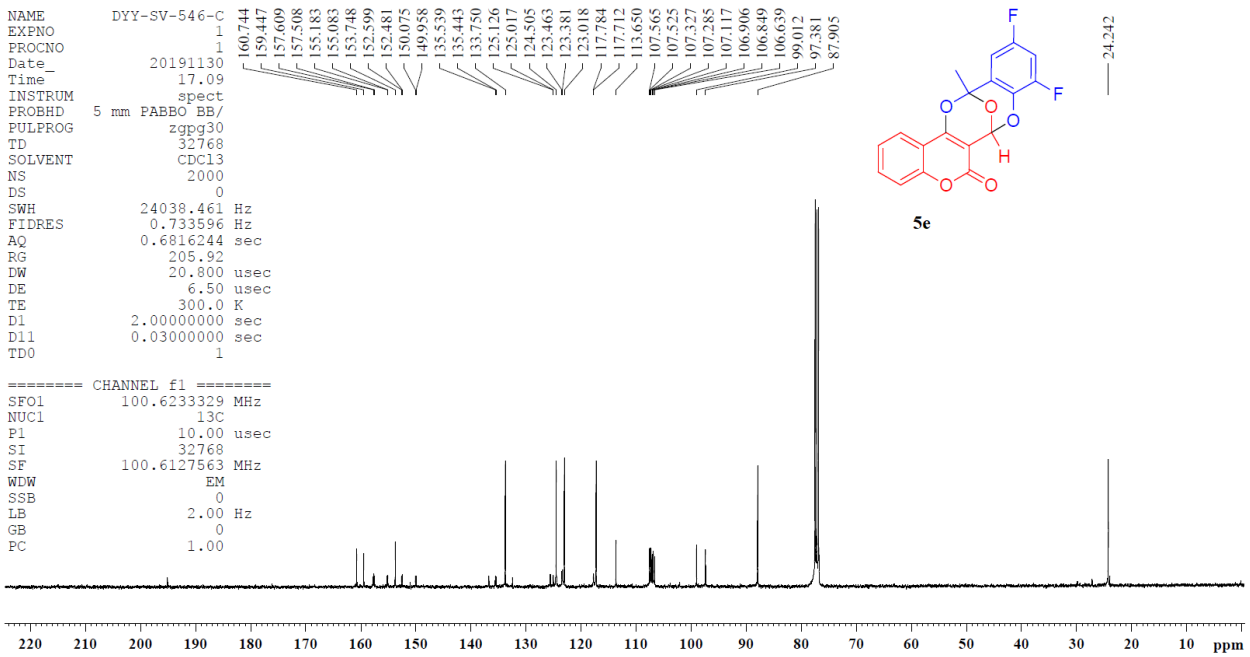

7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 ppm
1.287
1.361
3.968
1.274
1.130
1.000

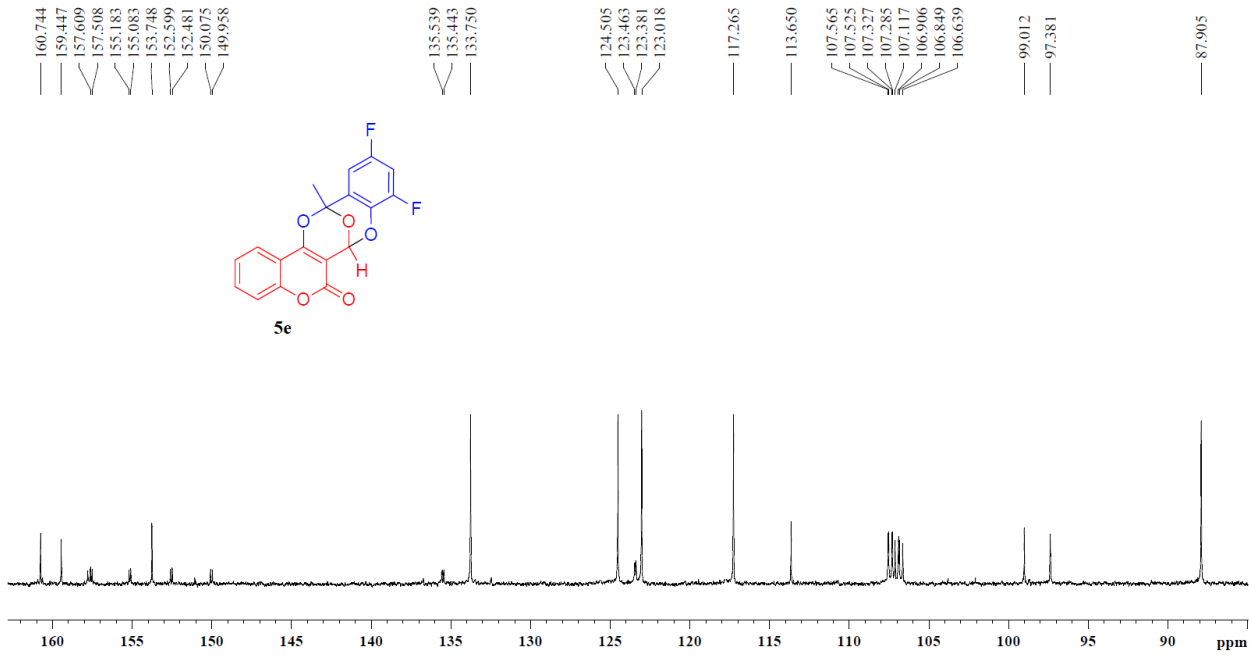

NAME DYY-SV-601-C
EXPNO 1
PROCNO 1
Date_ 20191130
Time 21.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2000
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 205.92
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127587 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
161.927
160.887
160.069
153.637
151.278
133.281
126.850
124.292
123.045
117.102
114.158
113.348
110.002
101.230
99.479
98.831
87.941
55.537
24.305
OMe
O
O
O
H
O
O
5d
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

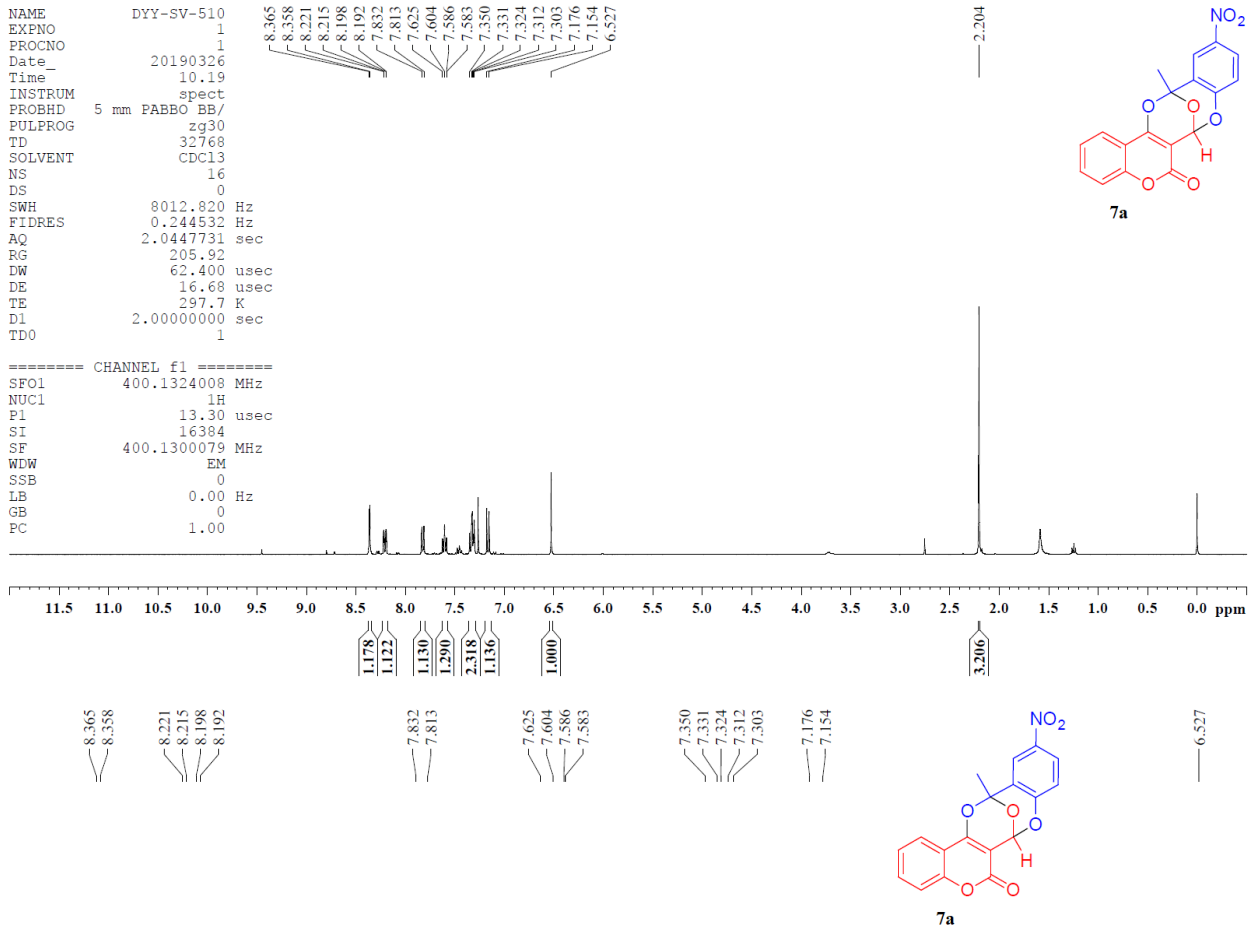

161.927
160.887
160.069
153.637
151.278
133.281
126.850
124.292
123.045
117.102
114.158
113.348
110.002
101.230
99.479
98.831
87.941
OMe
O
O
O
H
O
O
5d
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 ppm


NAME DYY-SV-546-H
EXPNO
PROCNO
Date_ 20191130
Time 13.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 163.06
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300061 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.794
7.790
7.773
7.770
7.609
7.605
7.588
7.569
7.566
7.323
7.315
7.313
7.305
7.294
7.292
7.287
7.267
6.934
6.927
6.922
6.913
6.909
6.902
6.889
6.885
6.882
6.878
6.864
6.857
6.491
2.114
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.987
1.000
2.144
2.038
0.808
2.877
F
F
O
O
O
H
O
O
5e

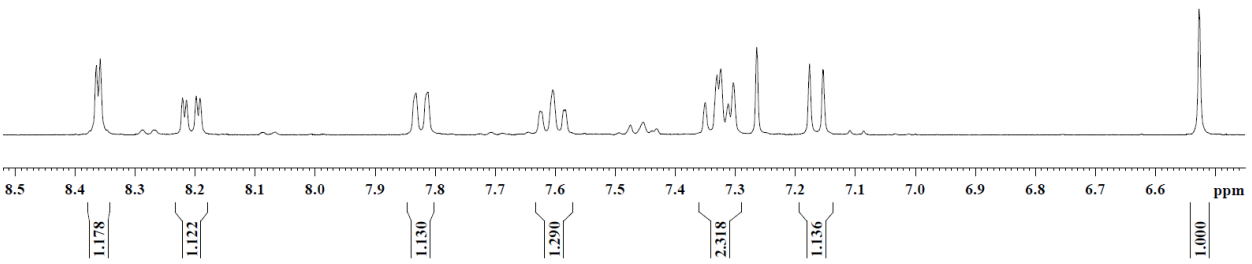

7.794
7.790
7.773
7.770
7.609
7.605
7.588
7.569
7.566
7.323
7.315
7.313
7.305
7.294
7.292
7.287
7.267
6.934
6.927
6.922
6.913
6.909
6.902
6.889
6.885
6.882
6.878
6.864
6.857
6.491
F
F
O
O
O
H
O
O
5e
7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70 6.65 6.60 6.55 ppm
0.987
1.000
2.144
2.038
0.808

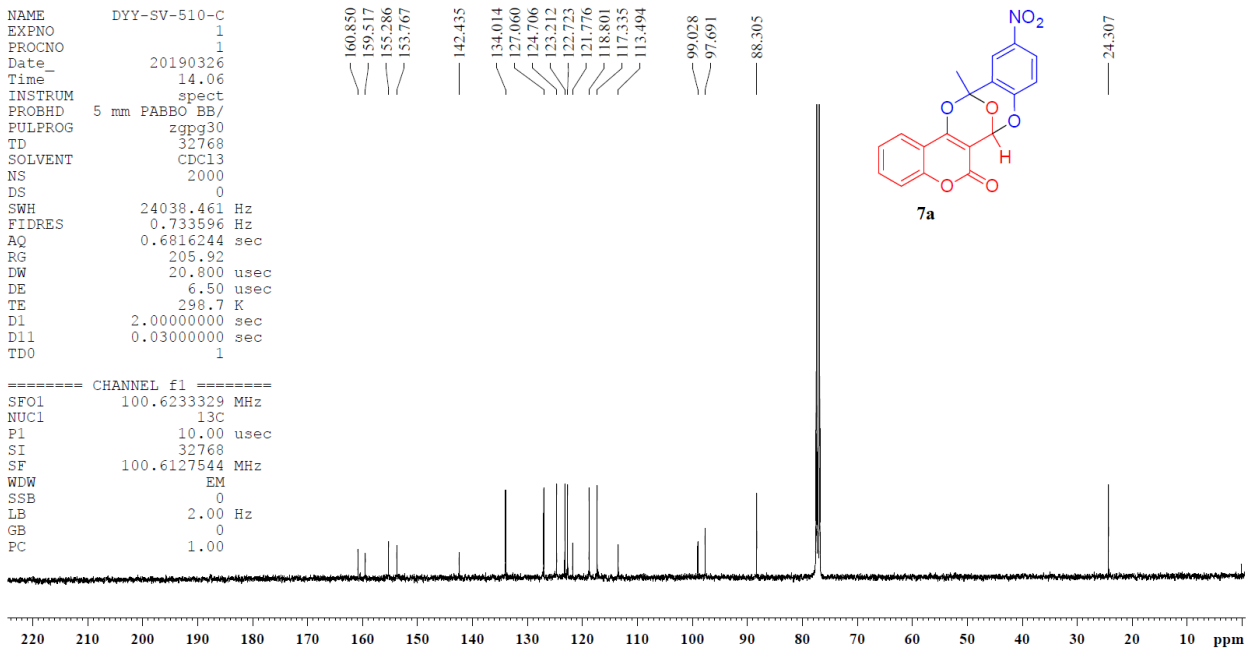

NAME DYY-SV-546-C
EXPNO 1
PROCNO 1
Date_ 20191130
Time 17.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2000
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 205.92
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127563 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
160.744
159.447
157.609
157.508
155.183
155.083
153.748
152.599
152.481
150.075
149.958
135.539
135.443
133.750
125.126
125.017
124.505
123.463
123.381
123.018
117.784
117.712
113.650
107.565
107.525
107.327
107.285
107.117
106.906
106.849
106.639
99.012
97.381
87.905
24.242
F
F
O
O
O
H
O
O
5e
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

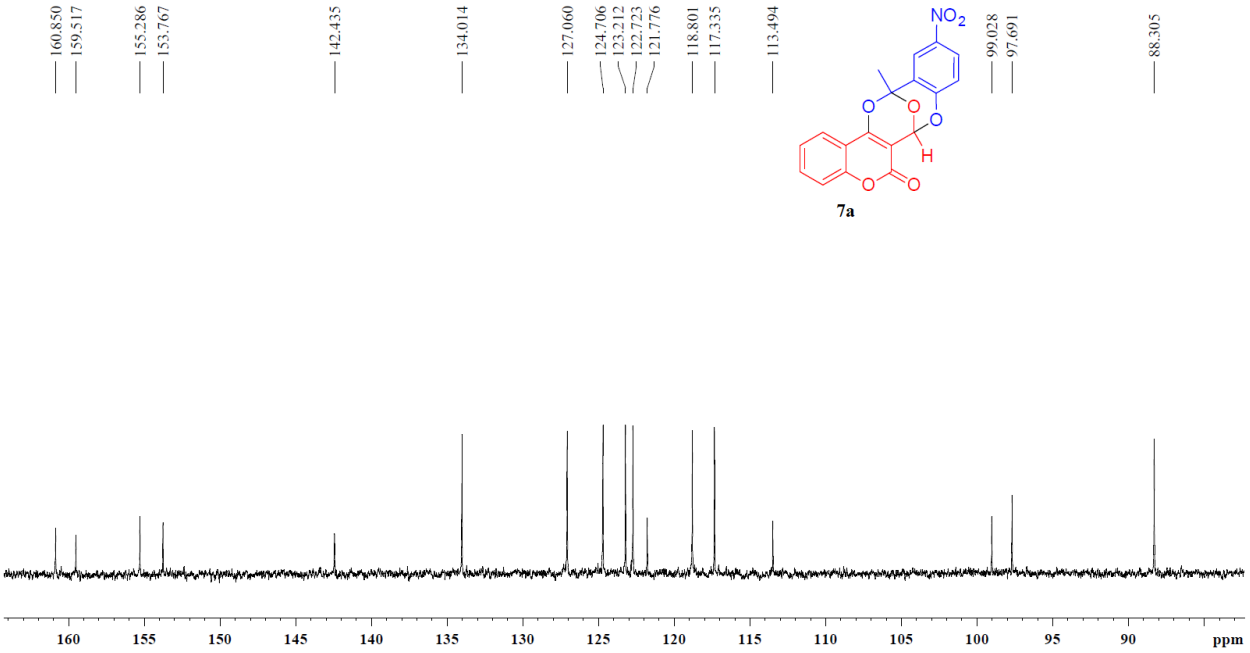

160.744
159.447
157.609
157.508
155.183
155.083
153.748
152.599
152.481
150.075
149.958
135.539
135.443
133.750
124.505
123.463
123.381
123.018
117.265
113.650
107.565
107.525
107.327
107.285
107.117
106.906
106.849
106.639
99.012
97.381
87.905
F
F
O
O
O
H
O
O
5e
160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 ppm

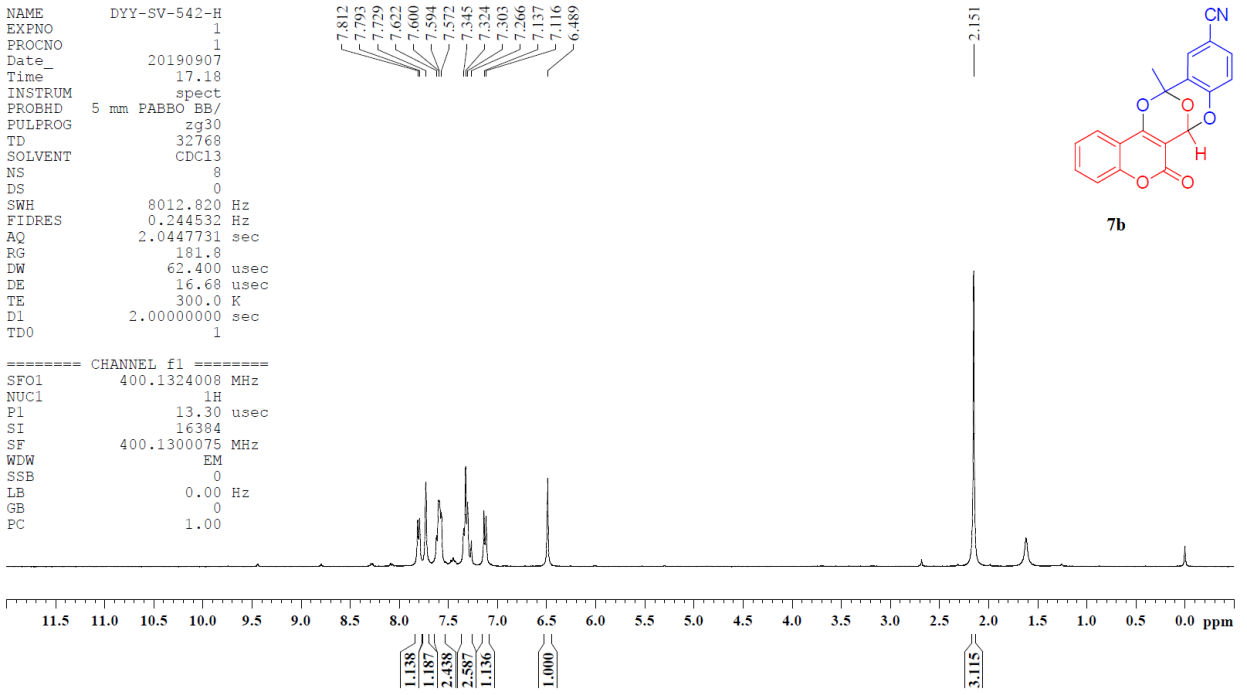

NAME DYY-SV-510
EXPNO 1
PROCNO 1
Date_ 20190326
Time 10.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 205.92
DW 62.400 usec
DE 16.68 usec
TE 297.7 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300079 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.365
8.358
8.221
8.215
8.198
8.192
7.832
7.813
7.625
7.604
7.586
7.583
7.350
7.331
7.324
7.312
7.303
7.176
7.154
6.527
2.204
NO2
7a
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.178
1.122
1.130
1.290
2.318
1.136
1.000
3.206

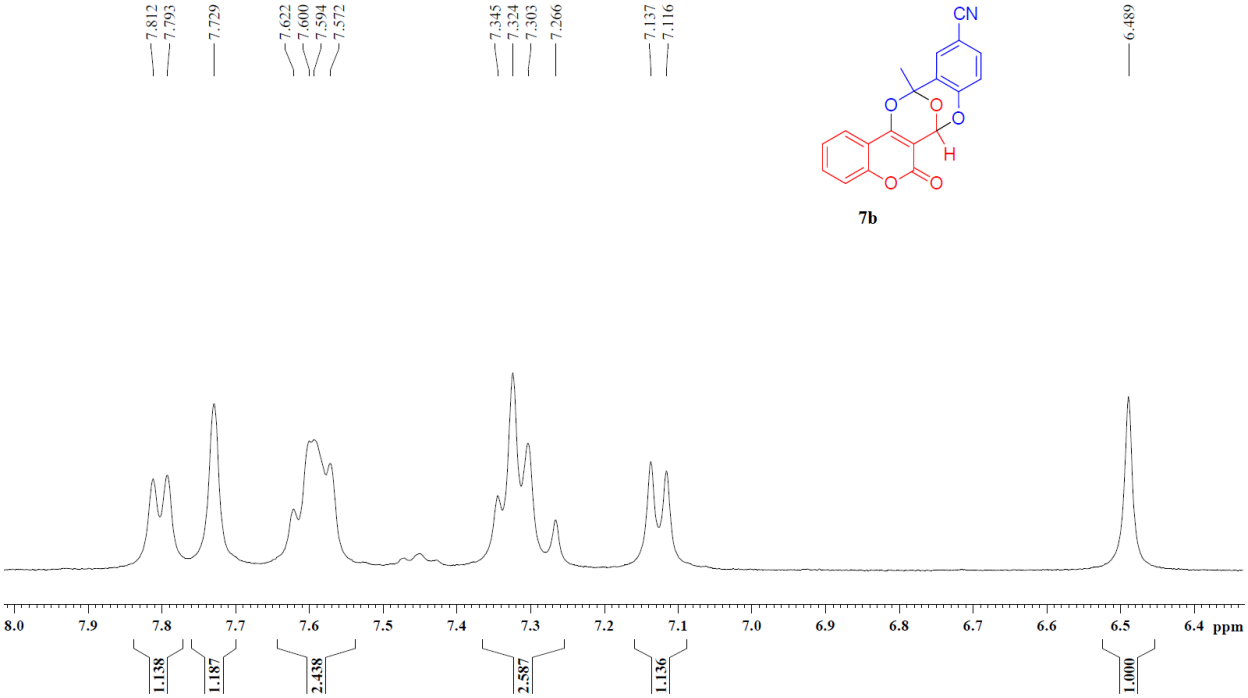
8.365
8.358
8.221
8.215
8.198
8.192
7.832
7.813
7.625
7.604
7.586
7.583
7.350
7.331
7.324
7.312
7.303
7.176
7.154
6.527
NO2
7a

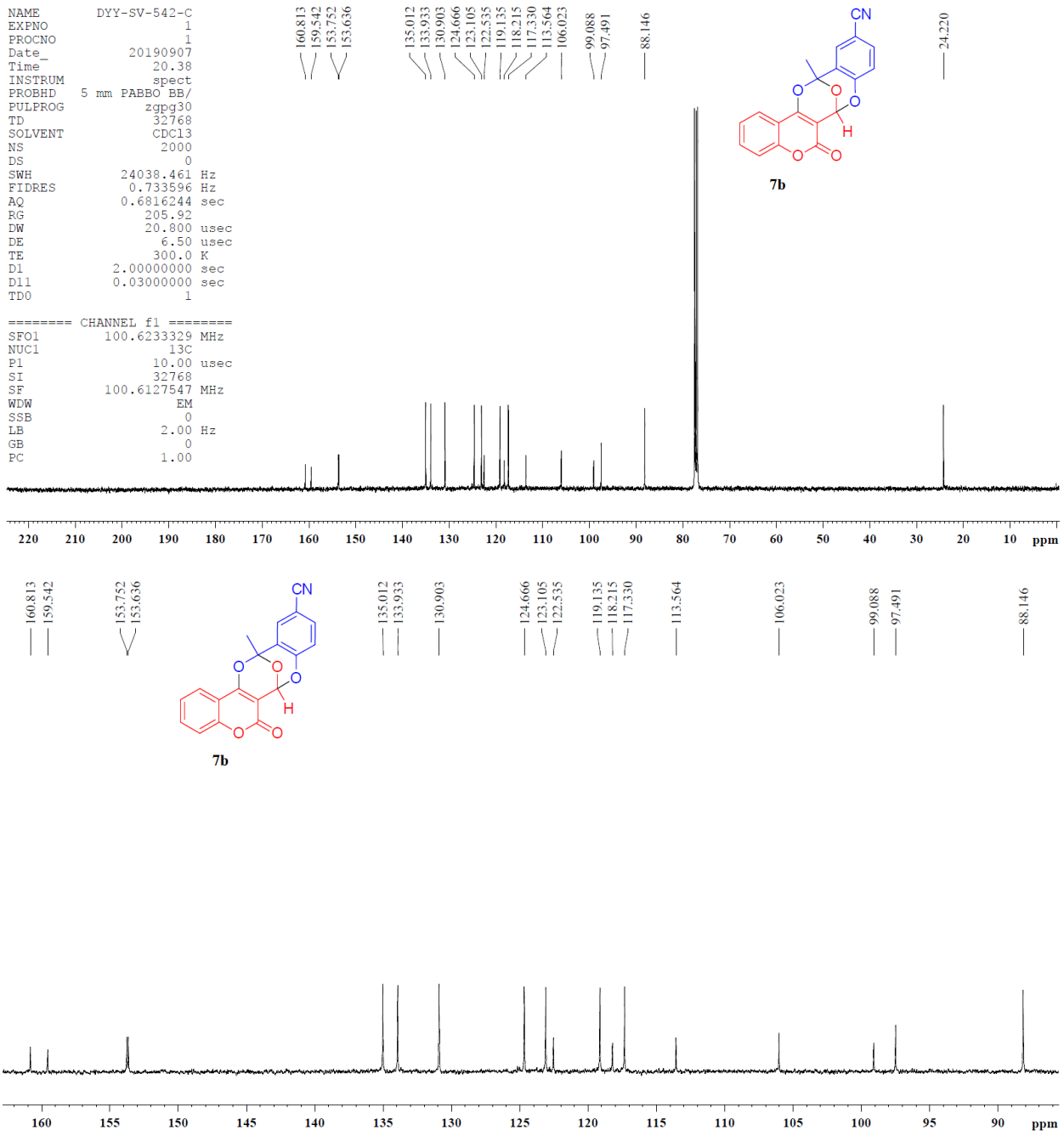

8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 ppm
1.178
1.122
1.130
1.290
2.318
1.136
1.000


NAME DYY-SV-510-C
EXPNO 1
PROCNO 1
Date_ 20190326
Time 14.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2000
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 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127544 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
160.850
159.517
155.286
153.767
142.435
134.014
127.060
124.706
123.212
122.723
121.776
118.801
117.335
113.494
99.028
97.691
88.305
24.307
NO2
O
O
O
H
O
O
7a
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

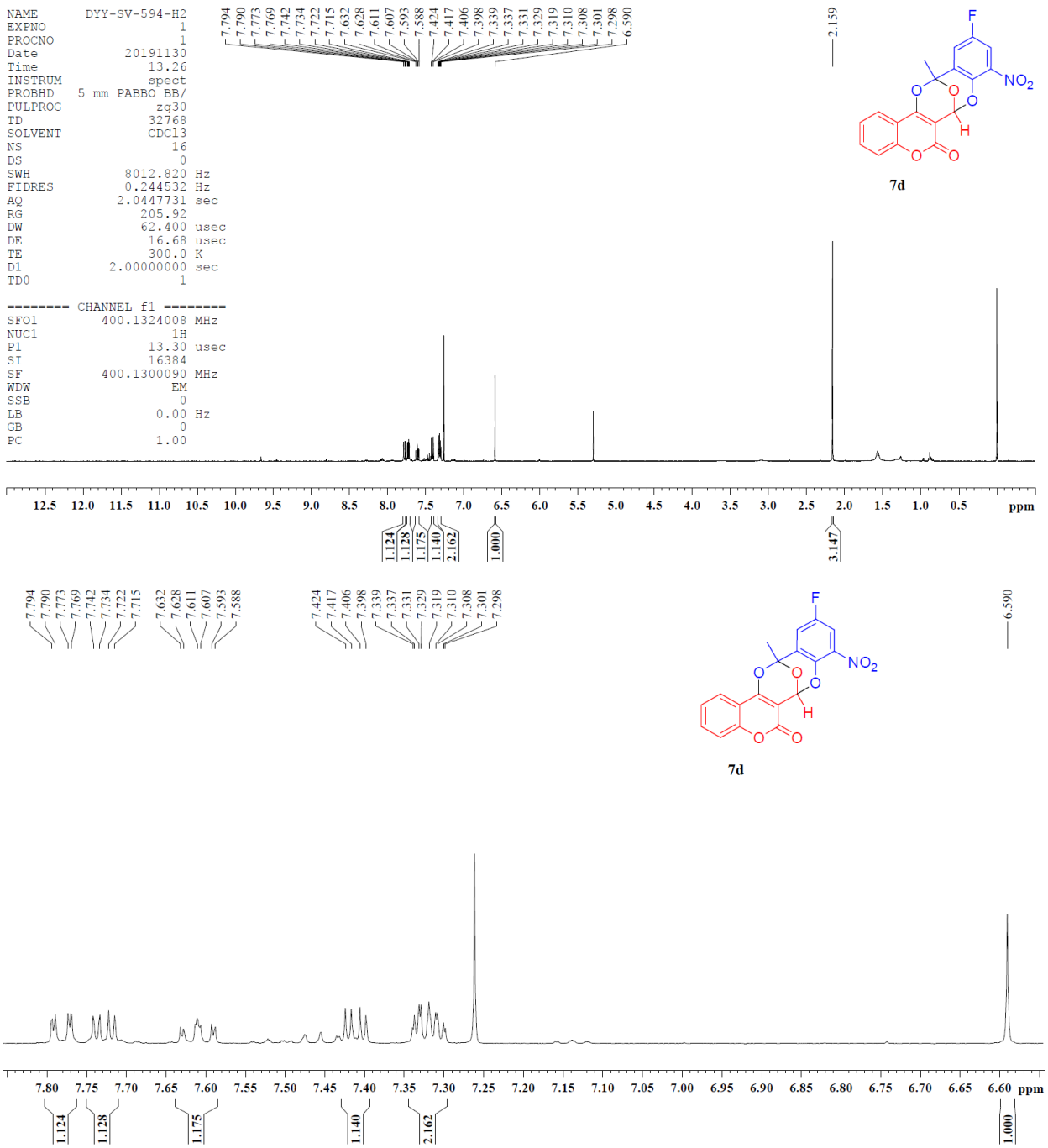

160.850
159.517
155.286
153.767
142.435
134.014
127.060
124.706
123.212
122.723
121.776
118.801
117.335
113.494
99.028
97.691
88.305
NO2
O
O
O
H
O
O
7a
160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 ppm

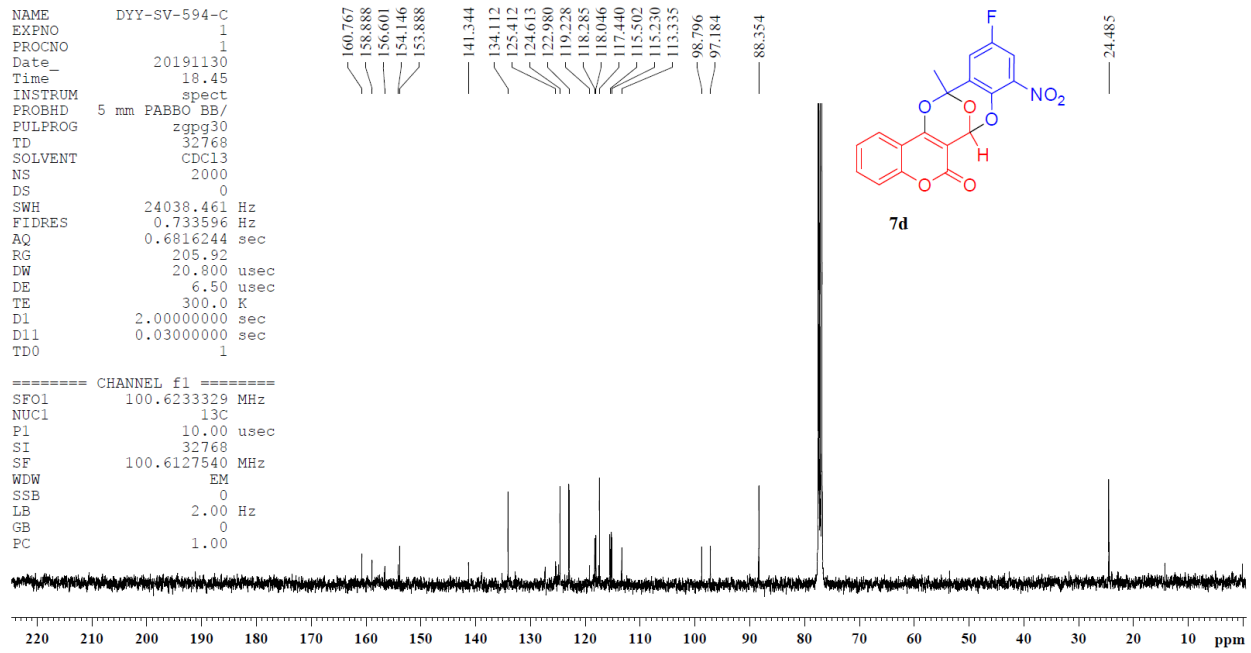

NAME DYY-SV-542-H
EXPNO 1
PROCNO 1
Date_ 20190907
Time 17.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 181.8
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300075 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.812
7.793
7.729
7.622
7.600
7.594
7.572
7.345
7.324
7.303
7.266
7.137
7.116
6.489
2.151
CN
O
O
O
H
O
O
7b
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.138
1.187
2.438
2.587
1.136
1.000
3.115


7.812
7.793
7.729
7.622
7.600
7.594
7.572
7.345
7.324
7.303
7.266
7.137
7.116
6.489
CN
O
O
O
H
O
O
7b
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 ppm
1.138
1.187
2.438
2.587
1.136
1.000

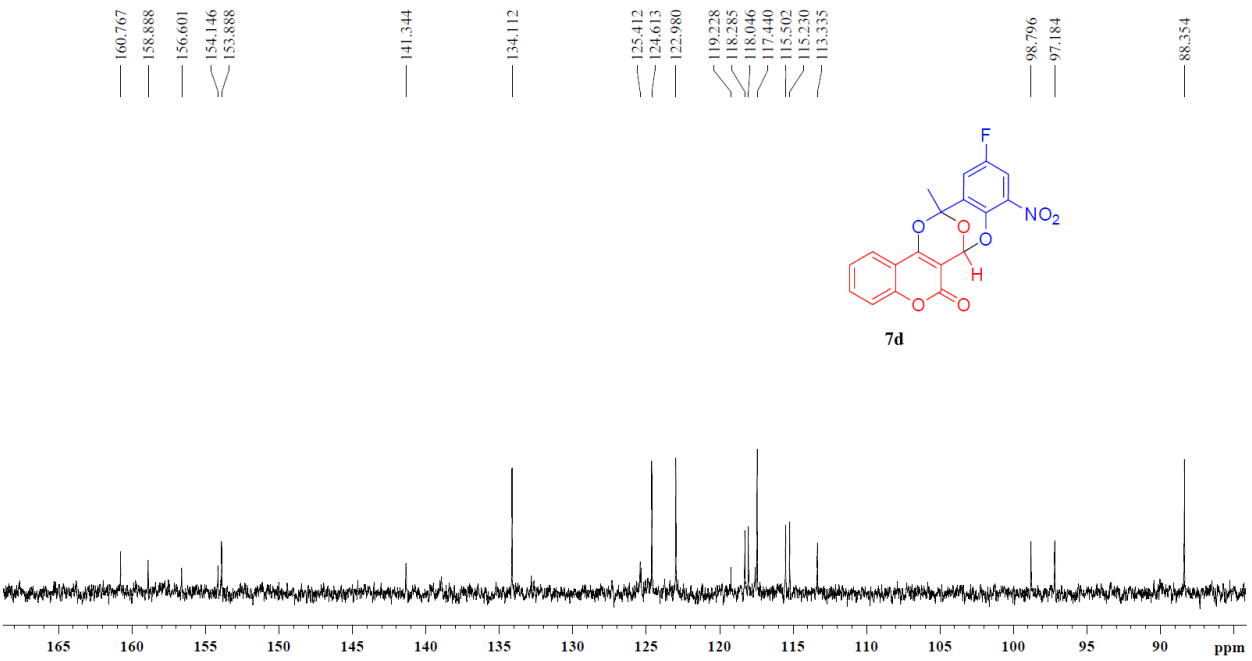

NAME DYY-SV-542-C
EXPNO 1
PROCNO 1
Date_ 20190907
Time 20.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2000
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 205.92
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127547 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
160.813
159.542
153.752
153.636
135.012
133.933
130.903
124.666
123.105
122.535
119.135
118.215
117.330
113.564
106.023
99.088
97.491
88.146
24.220
CN
O
O
O
H
O
O
7b
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
160.813
159.542
153.752
153.636
CN
135.012
133.933
130.903
124.666
123.105
122.535
119.135
118.215
117.330
113.564
106.023
99.088
97.491
88.146
O
O
O
H
O
O
7b
160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 ppm

[NOTE: **7c** = **5e**]

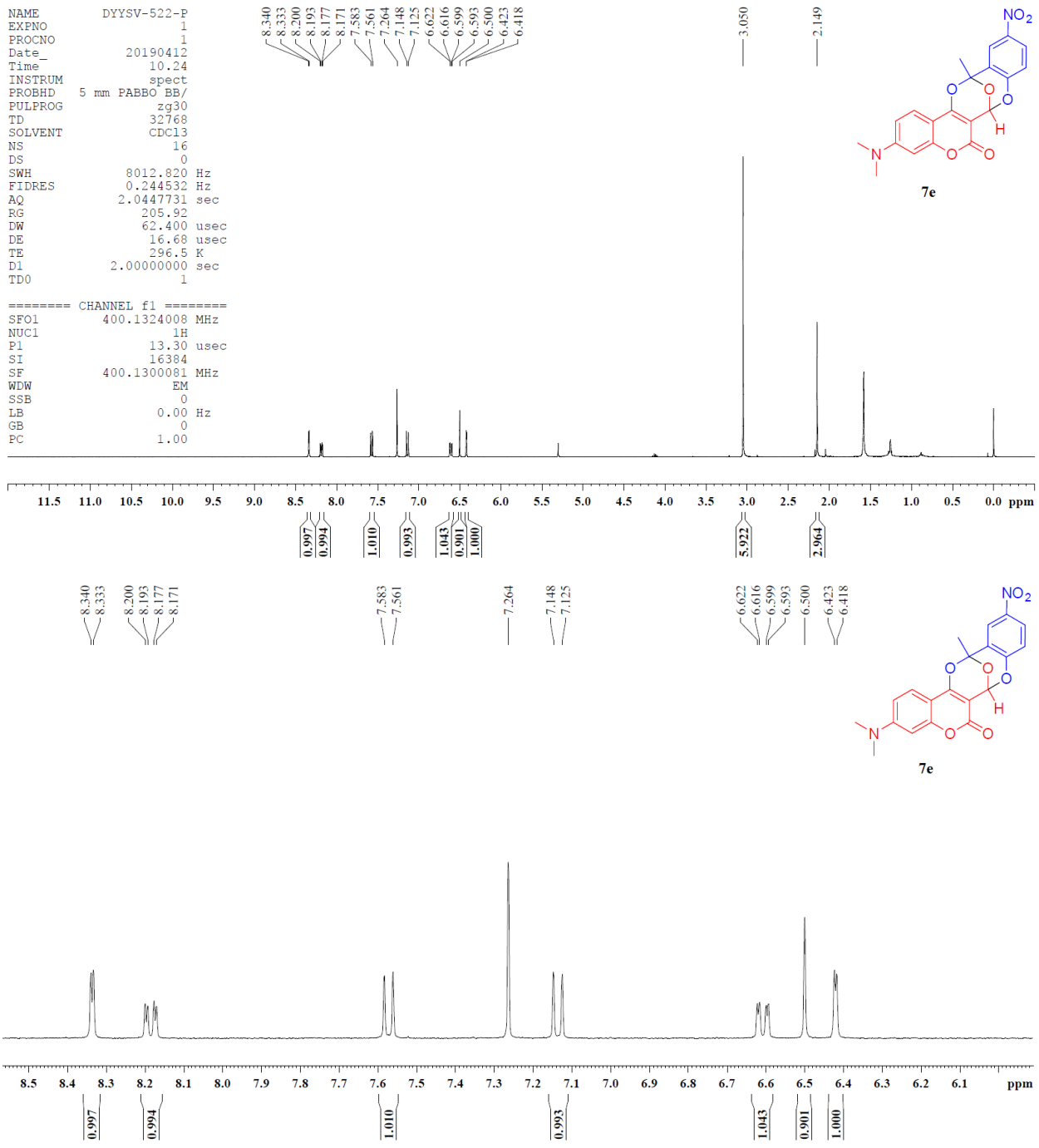

NAME DYY-SV-594-H2
EXPNO 1
PROCNO 1
Date_ 20191130
Time 13.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 205.92
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.794 7.790 7.773 7.769 7.742 7.734 7.722 7.715 7.632 7.628 7.611 7.607 7.593 7.588 7.424 7.417 7.406 7.398 7.339 7.337 7.331 7.329 7.319 7.310 7.308 7.301 7.298 6.590
2.159
F
NO2
O
O
O
H
O
O
7d
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.124 1.128 1.175 1.140 2.162 1.000 3.147
7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70 6.65 6.60 ppm
1.124 1.128 1.175 1.140 2.162 1.000


NAME DYY-SV-594-C
EXPNO 1
PROCNO 1
Date_ 20191130
Time 18.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2000
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 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127540 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
160.767
158.888
156.601
154.146
153.888
141.344
134.112
125.412
124.613
122.980
119.228
118.285
118.046
117.440
115.502
115.230
113.335
98.796
97.184
88.354
24.485
F
NO2
O
O
O
H
O
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

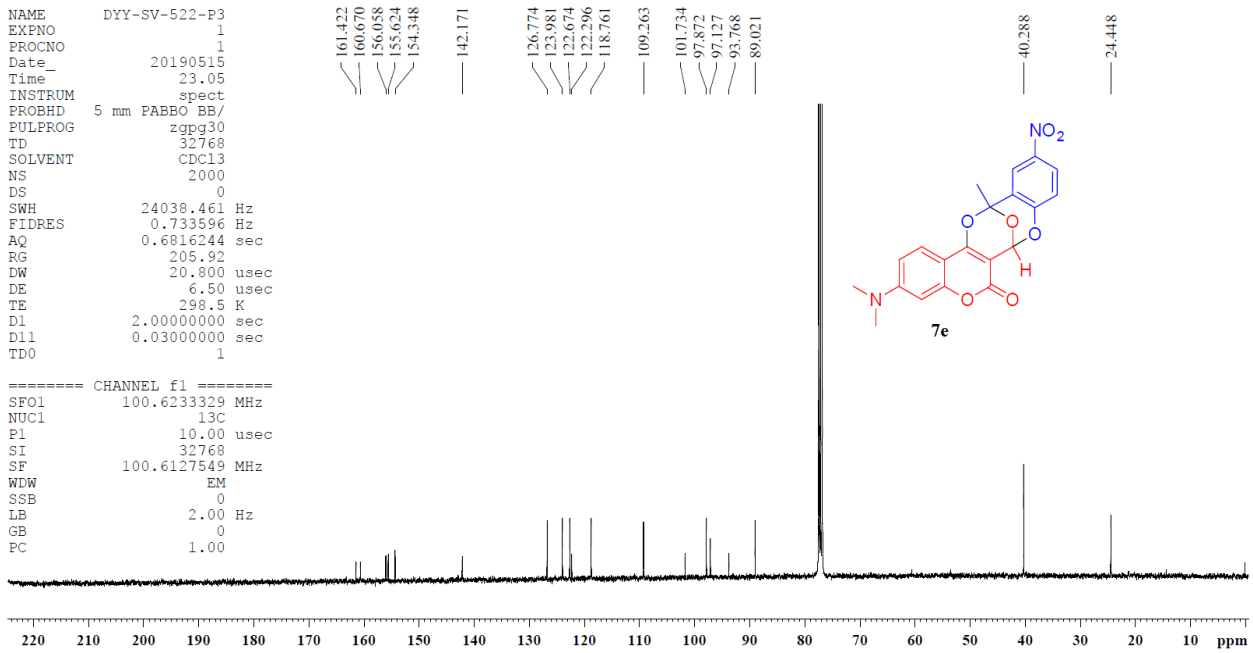
7d

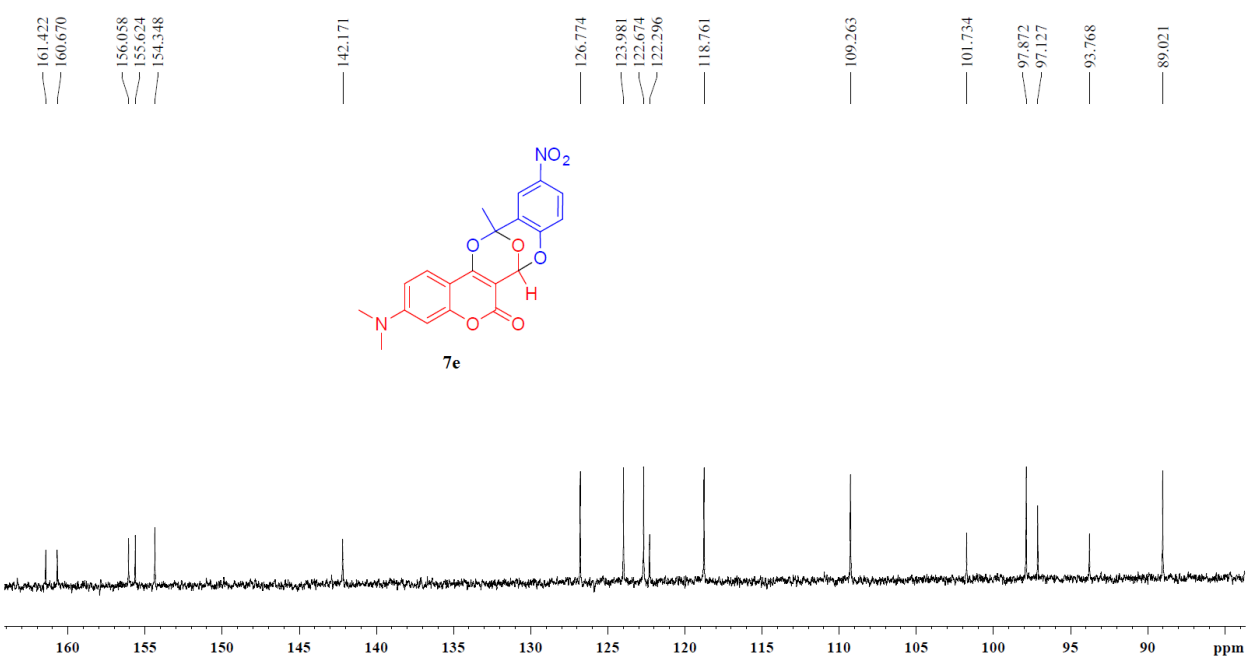

160.767
158.888
156.601
154.146
153.888
141.344
134.112
125.412
124.613
122.980
119.228
118.285
118.046
117.440
115.502
115.230
113.335
98.796
97.184
88.354
F
NO2
O
O
O
H
O
O
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 ppm

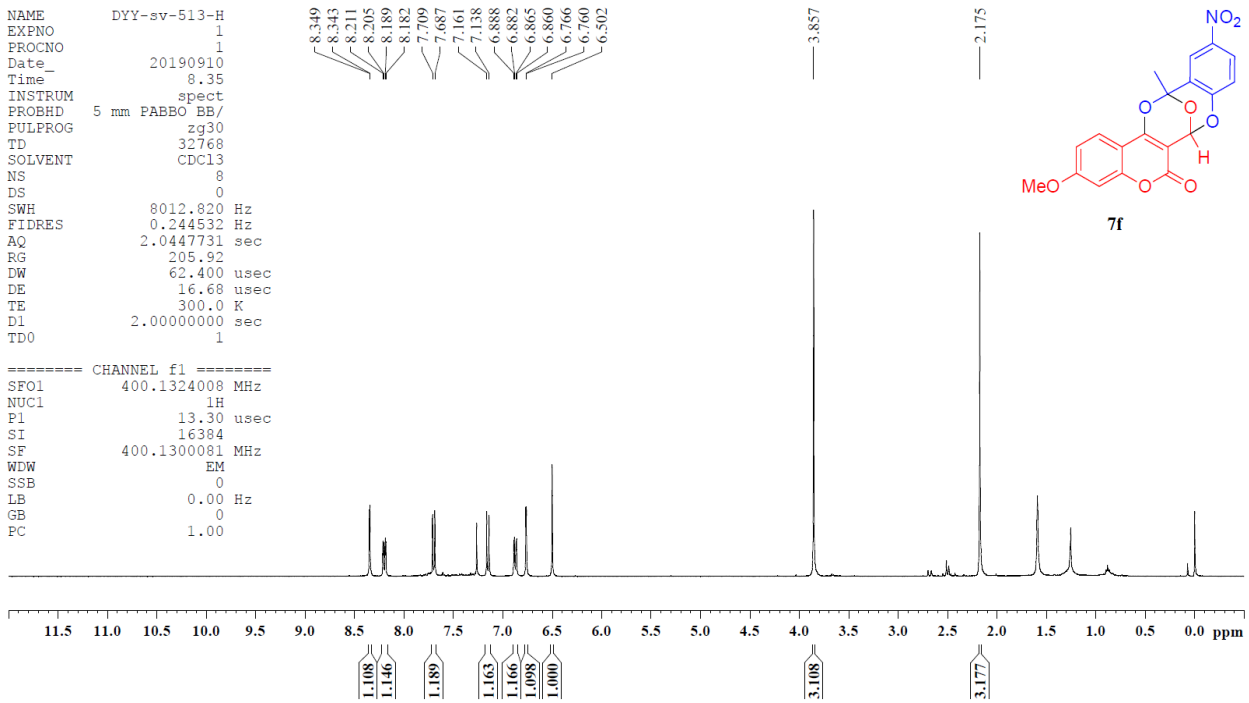
7d


NAME DYYSV-522-P
EXPNO 1
PROCNO 1
Date_ 20190412
Time 10.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 205.92
DW 62.400 usec
DE 16.68 usec
TE 296.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.340
8.333
8.200
8.193
8.177
8.171
7.583
7.561
7.264
7.148
7.125
6.622
6.616
6.599
6.593
6.500
6.423
6.418
3.050
2.149
NO2
O
O
O
H
N
O
O
7e
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.997
0.994
1.010
0.993
1.043
0.901
1.000
5.922
2.964

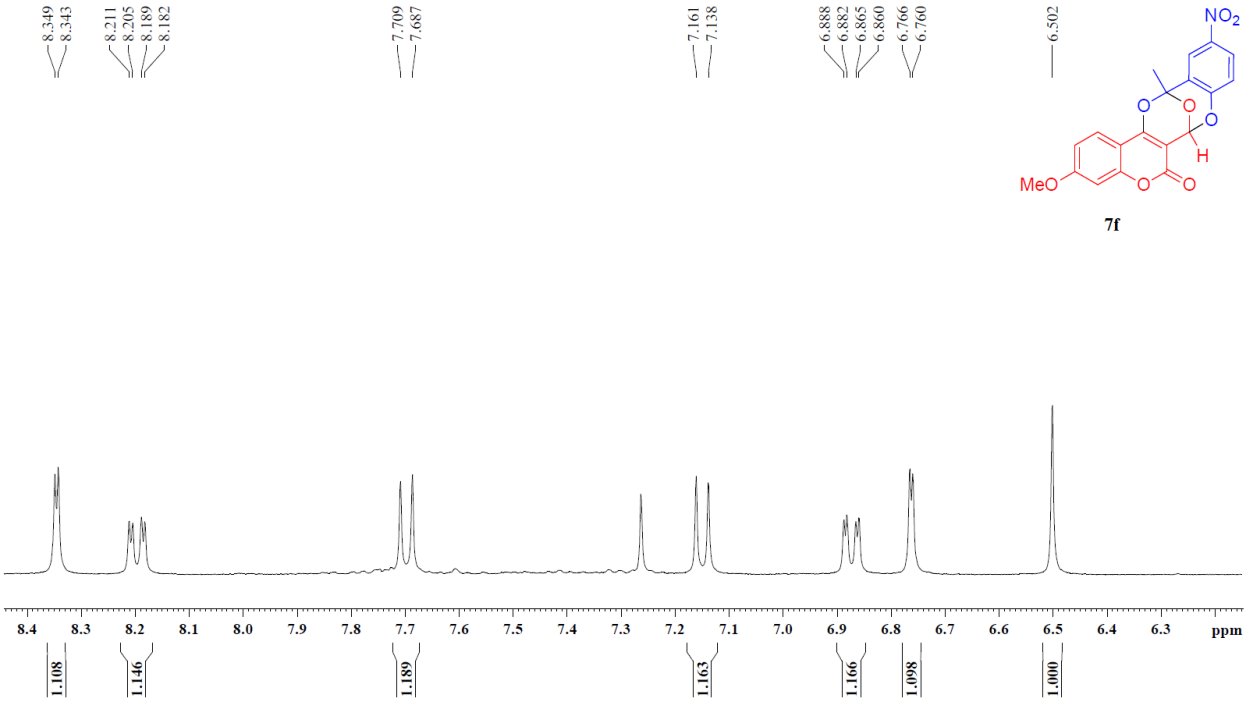
8.340
8.333
8.200
8.193
8.177
8.171
7.583
7.561
7.264
7.148
7.125
6.622
6.616
6.599
6.593
6.500
6.423
6.418
NO2
O
O
O
H
N
O
O
7e
8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1 ppm
0.997
0.994
1.010
0.993
1.043
0.901
1.000


NAME DYY-SV-522-P3
EXPNO 1
PROCNO 1
Date_ 20190515
Time 23.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2000
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 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127549 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
161.422
160.670
156.058
155.624
154.348
142.171
126.774
123.981
122.674
122.296
118.761
109.263
101.734
97.872
97.127
93.768
89.021
40.288
24.448
NO2
O
O
O
H
N
O
O
7e
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

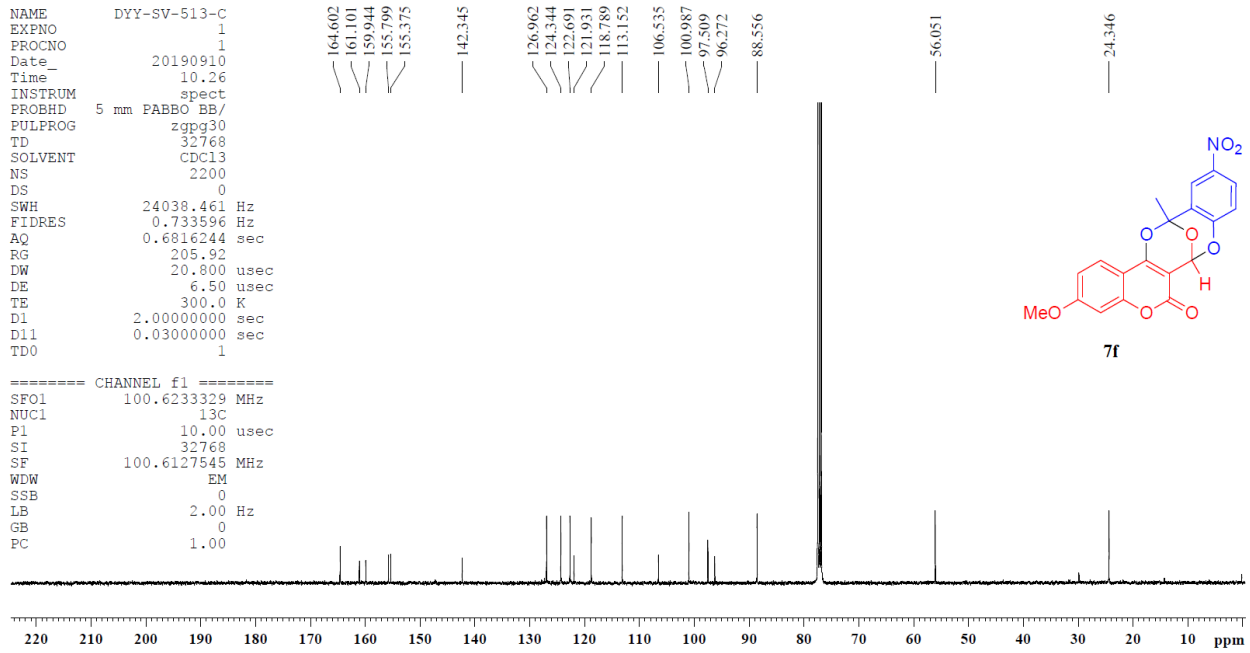

161.422
160.670
156.058
155.624
154.348
142.171
126.774
123.981
122.674
122.296
118.761
109.263
101.734
97.872
97.127
93.768
89.021
NO2
O
O
O
H
N
O
O
7e
160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 ppm


NAME DYY-sv-513-H
EXPNO 1
PROCNO 1
Date_ 20190910
Time 8.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 205.92
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.349
8.343
8.211
8.205
8.189
8.182
7.709
7.687
7.161
7.138
6.888
6.882
6.865
6.860
6.766
6.760
6.502
3.857
2.175
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.108
1.146
1.189
1.163
1.166
1.098
1.000
3.108
3.177
NO2
O
O
O
H
MeO
O
O

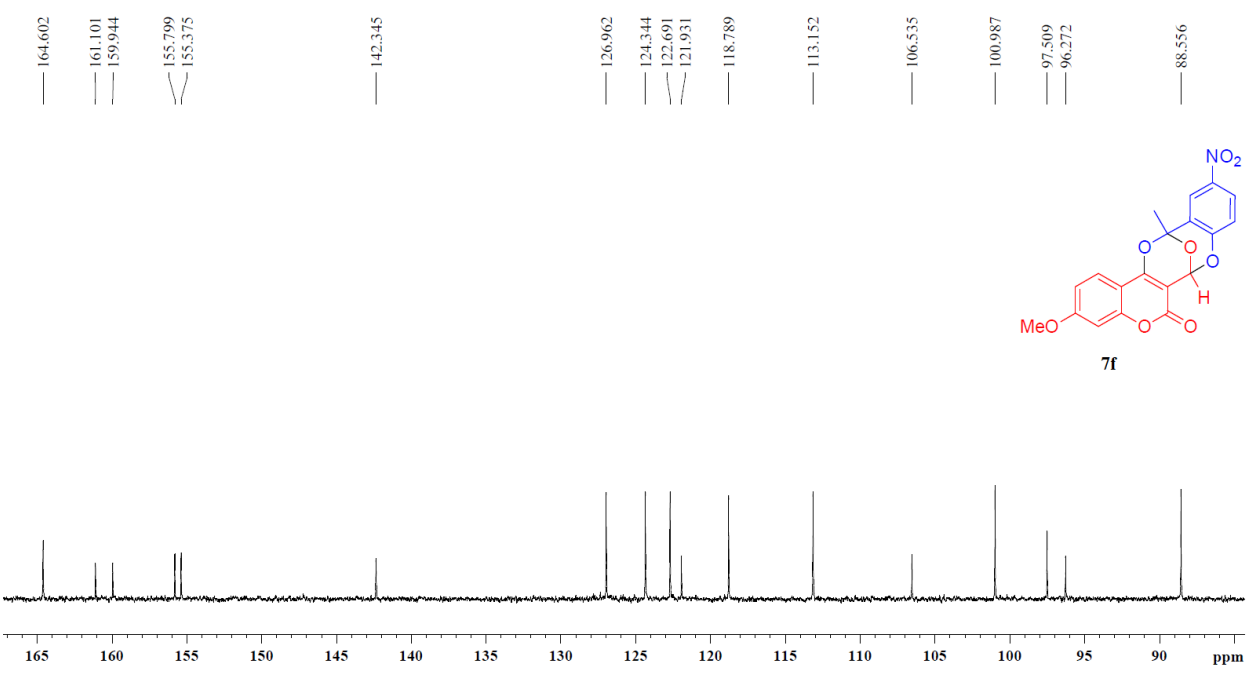
7f


8.349
8.343
8.211
8.205
8.189
8.182
7.709
7.687
7.161
7.138
6.888
6.882
6.865
6.860
6.766
6.760
6.502
8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 ppm
1.108
1.146
1.189
1.163
1.166
1.098
1.000
NO2
O
O
O
H
MeO
O
O

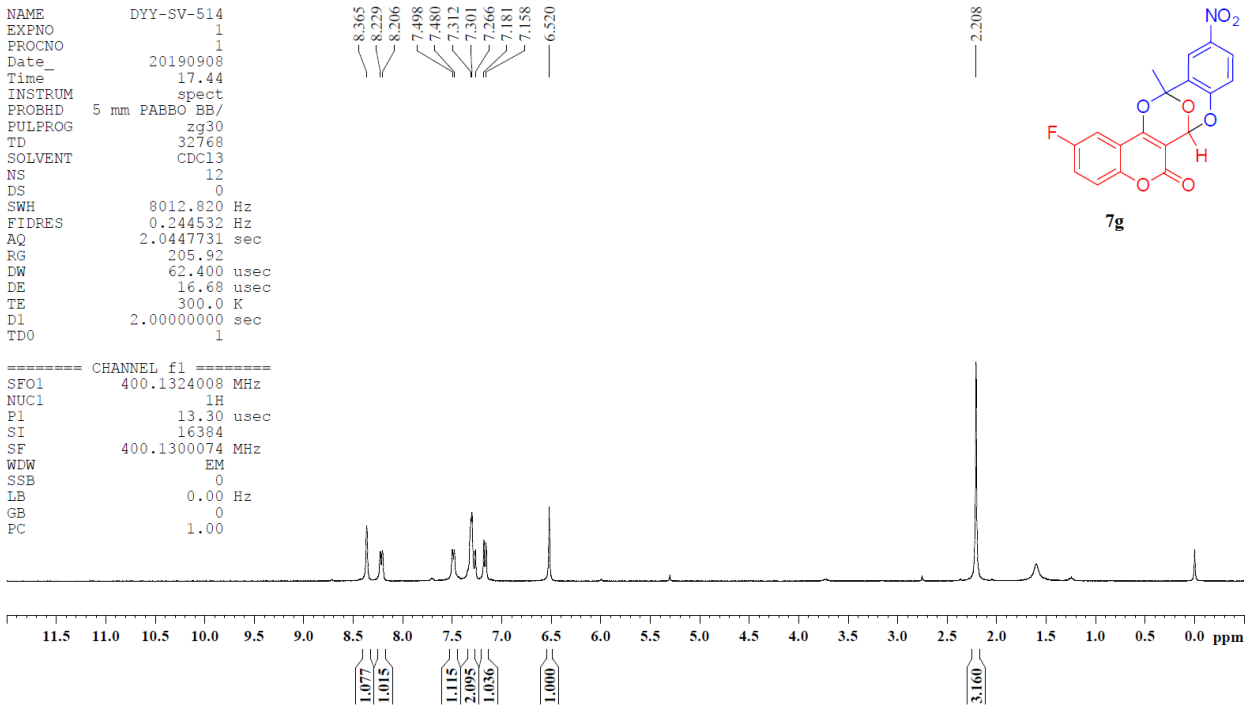
7f


NAME DYY-SV-513-C
EXPNO 1
PROCNO 1
Date_ 20190910
Time 10.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2200
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 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127545 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
164.602
161.101
159.944
155.799
155.375
142.345
126.962
124.344
122.691
121.931
118.789
113.152
106.535
100.987
97.509
96.272
88.556
56.051
24.346
NO2
MeO
H
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

7f

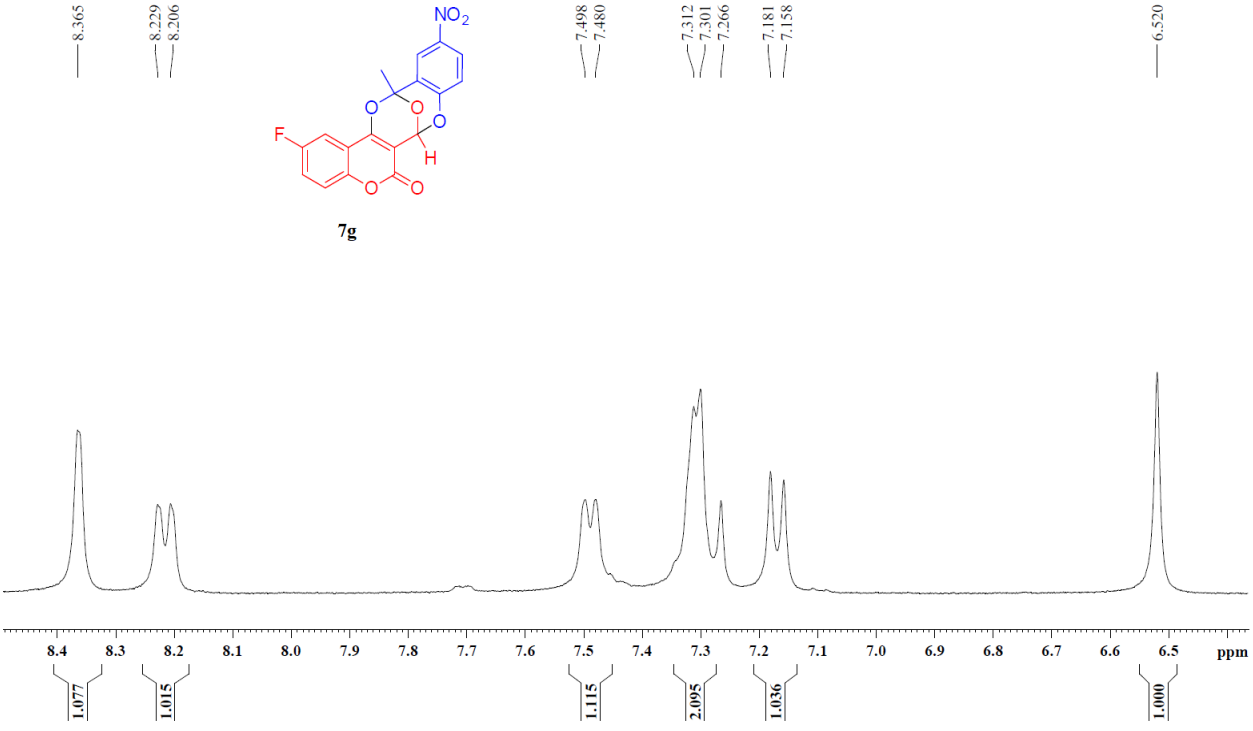

164.602
161.101
159.944
155.799
155.375
142.345
126.962
124.344
122.691
121.931
118.789
113.152
106.535
100.987
97.509
96.272
88.556
NO2
MeO
H
O
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 ppm

7f

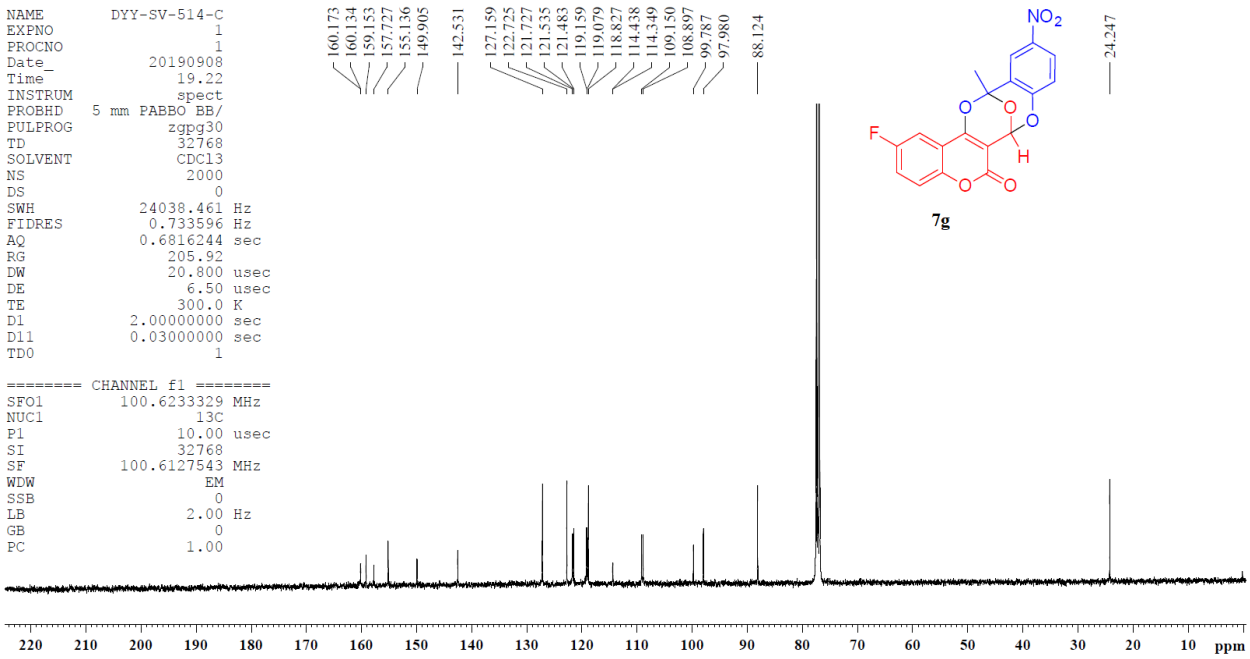

NAME DYY-SV-514
EXPNO 1
PROCNO 1
Date_ 20190908
Time 17.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 12
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 205.92
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300074 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.365
8.229
8.206
7.498
7.480
7.312
7.301
7.266
7.181
7.158
6.520
2.208
1.077
1.015
1.115
2.095
1.036
1.000
3.160
ppm

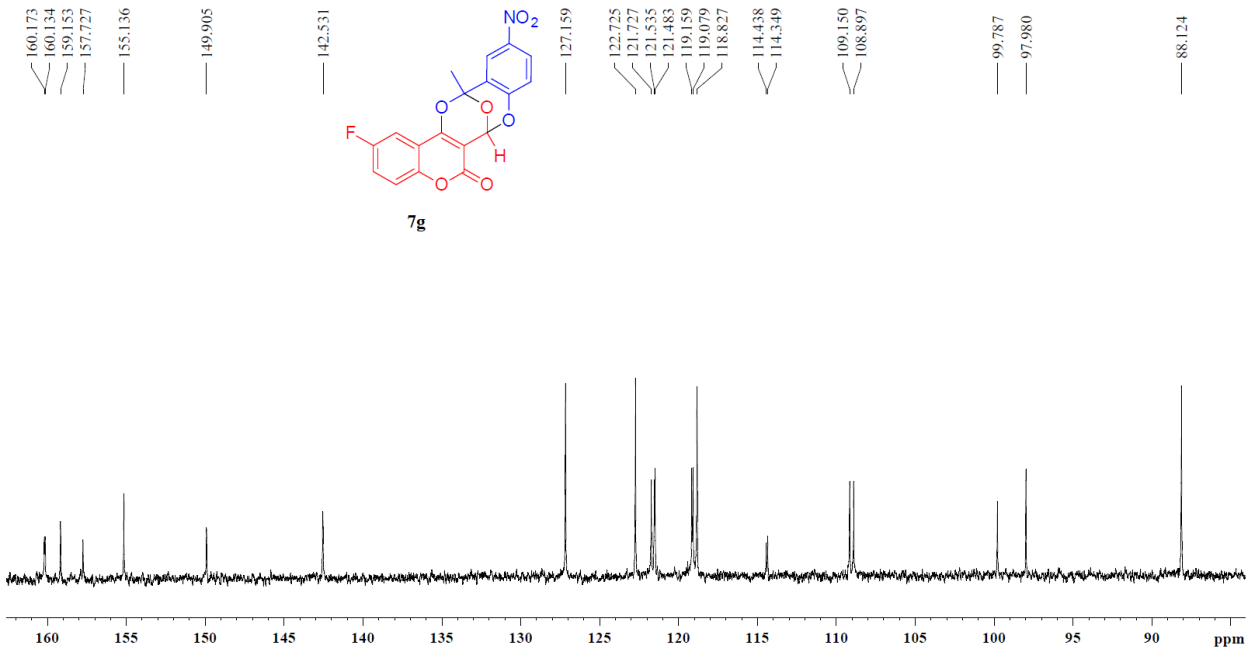
NO2
F
O
O
O
H
O
O

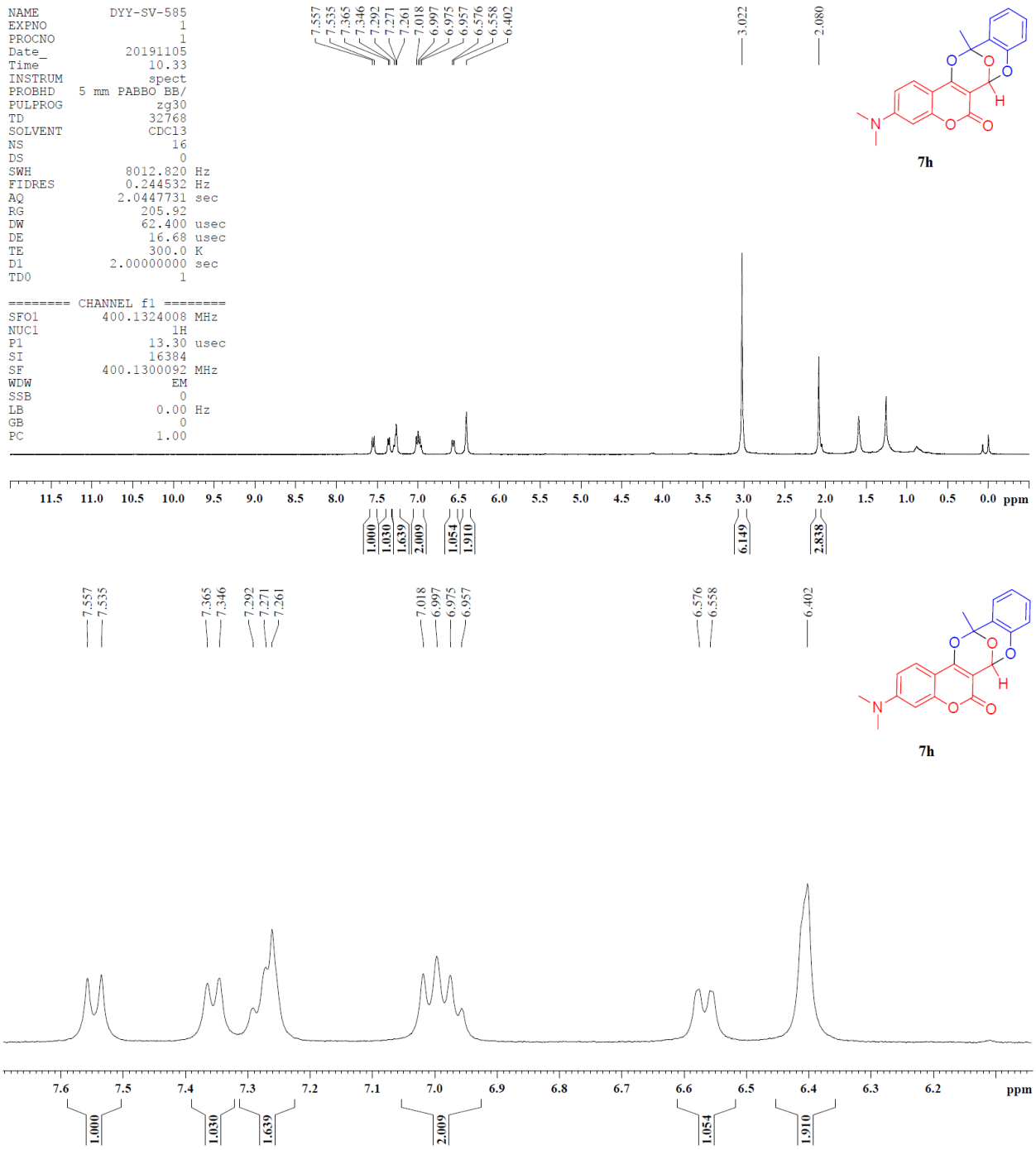
7g


8.365
8.229
8.206
7.498
7.480
7.312
7.301
7.266
7.181
7.158
6.520
NO2
F
O
O
O
H
O
O
1.077
1.015
1.115
2.095
1.036
1.000
ppm

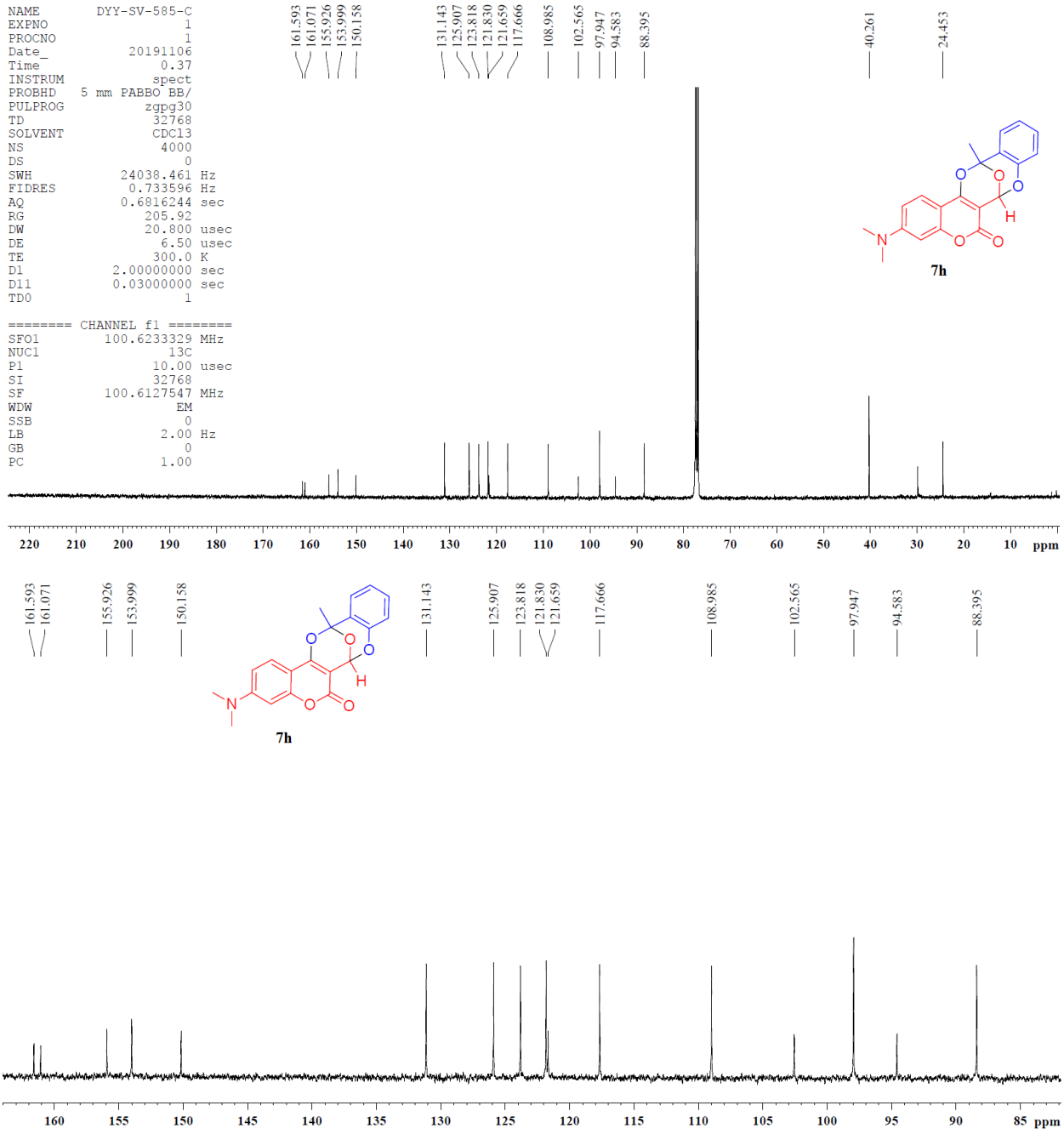
7g


NAME DYY-SV-514-C
EXPNO 1
PROCNO 1
Date_ 20190908
Time 19.22
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2000
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 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127543 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
160.173
160.134
159.153
157.727
155.136
149.905
142.531
127.159
122.725
121.727
121.535
121.483
119.159
119.079
118.827
114.438
114.349
109.150
108.897
99.787
97.980
88.124
24.247
NO2
F
O
O
O
H
O
O
7g
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


160.173
160.134
159.153
157.727
155.136
149.905
142.531
127.159
122.725
121.727
121.535
121.483
119.159
119.079
118.827
114.438
114.349
109.150
108.897
99.787
97.980
88.124
NO2
F
O
O
O
H
O
O
7g
160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 ppm


NAME DYY-SV-585
EXPNO 1
PROCNO 1
Date_ 20191105
Time 10.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 205.92
DW 62.400 usec
DE 16.68 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 13.30 usec
SI 16384
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.557
7.535
7.365
7.346
7.292
7.271
7.261
7.018
6.997
6.975
6.957
6.576
6.558
6.402
3.022
2.080
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.000
1.030
1.639
2.009
1.054
1.910
6.149
2.838
7h

7.557
7.535
7.365
7.346
7.292
7.271
7.261
7.018
6.997
6.975
6.957
6.576
6.558
6.402
7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 ppm
1.000
1.030
1.639
2.009
1.054
1.910
7h


NAME DYY-SV-585-C
EXPNO 1
PROCNO 1
Date_ 20191106
Time 0.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4000
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 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6233329 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127547 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
161.593
161.071
155.926
153.999
150.158
131.143
125.907
123.818
121.830
121.659
117.666
108.985
102.565
97.947
94.583
88.395
40.261
24.453
7h
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

161.593
161.071
155.926
153.999
150.158
131.143
125.907
123.818
121.830
121.659
117.666
108.985
102.565
97.947
94.583
88.395
7h
160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 ppm