Dienaminodioate based multicomponent reactions with post-benzylic oxidative transformations mediated by DDQ

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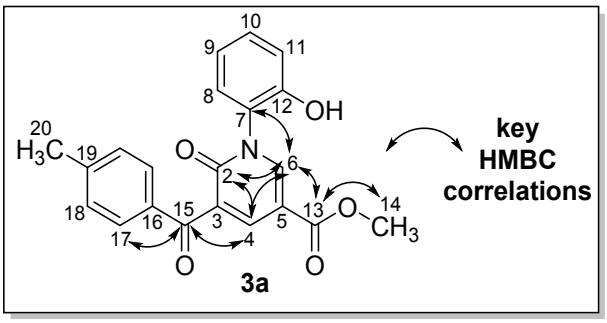
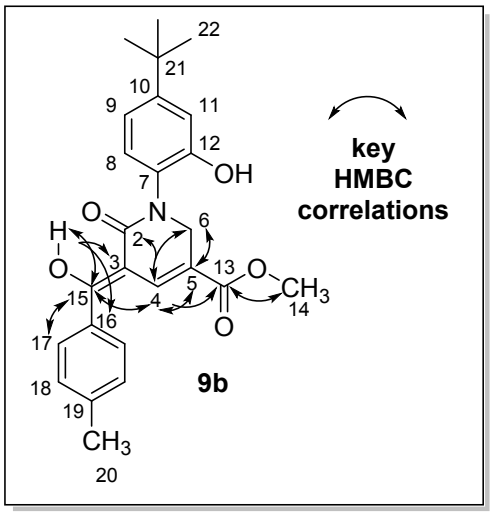
1. Structural confirmation of compounds 3a, 9b and 9a



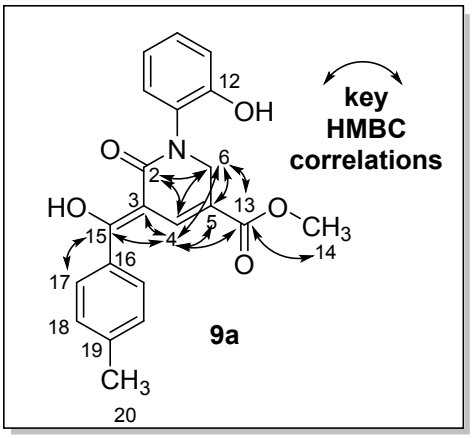
The molecular formula of compound **3a** determined as $C_{21}H_{17}NO_5$ by HR-ESI-MS (14 degrees of unsaturation) along with ^1H and ^{13}C NMR data was in agreement with the desired 2-pyridone product.^{13}C NMR showed the presence of ketone carbonyl at 192.5 ppm (C15), ester and amide carbonyls. ^1H NMR spectra indicated the presence of a D_2O exchangeable proton (C12-OH) at 7.50 ppm. A loss of one methoxy group with concomitant loss of one ester carbonyl from 1,2-DHP in the ^1H and ^{13}C NMRs of compound **3**, and appearance of one ketone, amide carbonyls in its ^{13}C NMR corroborate 2-pyridone formation. Presence of COSY correlations between H11 and H10; H9 and H8; and H17 and H18 indicate the role of both the phenyl units as mere substituents without undergoing any change in the synthetic transformation. Key HMBC correlations confirm the presence of 2-pyridone core unit and its connectivity with ester, ketone and two phenyl units, Table S1 contains the entire list of HMBC correlations. At the outset, presence of HMBC correlations from H14 (OCH_3) to ester carbonyl C13 (δ_H 163.8), H20 (C-CH_3) to C19 (4°), C18 confirms the positions of the two methyl groups. HMBC correlation from H18 to C16 confirm its 4°carbon position. The most shielded aromatic proton H11 (δ_H 6.85, ortho to OH) have HMBC correlations with C9, C7 (4°), C12 (4°); and HMBC correlations from H9 to C11, C7 (4°); H8 to C7 (4°), C12 (4°); and H10 to C12 (4°) confirm the annotation of phenolic ring with C12 as the most deshielded 4° carbon (δ_C 151.8) as it is directly attached to OH and the next 4°carbon C7 (δ_C 128.5) attached to N.

Turning on to the key HMBC correlations, firstly, correlation between H17 to C15, C19 point toward the connectivity of *p*-tolyl group to ketone carbonyl. Secondly, presence of HMBC correlations from H4 to C15, C2 (amide carbonyl δ_H 160.7), C6 confirms its position in 2-pyridone. H6 (δ_H 8.38) is the most downfield proton than H4 (δ_H 8.29), similar to its position in the corresponding 1,2-DHP, due to its presence ortho to N(CO) atom. HMBC correlation from H6 to C13, C2, C4 and C7 confirms the 2-pyridone core of compound **3**. Even though, no HMBC correlations exist for two 4° carbons (C3 and C5) but large difference in their chemical shifts helped in identifying C3 and C5 at δ_C 130.0 and 110.8 ppm, respectively.



The molecular formula of compound **9b** determined as $C_{25}H_{27}NO_5$ by HR-ESI-MS (14 degrees of unsaturation) along with ^{1}H and ^{13}C NMR data was in agreement with the reduced 2-pyridone product. ^{13}C NMR showed the loss of ketone carbonyl peak and appearance of a new peak at δ_C 175.2 ppm (C15) along with ester and amide carbonyls. ^{1}H NMR spectra indicated the presence of two D_2O exchangeable protons at 5.84 and 15.10 ppm. Presence of the most downfield proton at δ_H15.10 ppm arises due to the presence of an enolic OH in strong hydrogen bond with the oxygen atom of amide carbonyl. Interestingly, a two proton signal at δ_H 4.72 ppm (C6), confirmed by DEPT as CH_2, indicates a 1,4- or 1,6- reduction product. Hence, HMBC correlations play a key role in elucidation of this reduction product, Table S1 contains the entire list of HMBC correlations.

Presence of COSY correlation between H17 and H18 indicate that the phenyl unit is unaltered during reduction. In addition, HMBC correlations from H20 (C-CH_3) to C19 (4°), C18 and H17 to C19 (4°), C15 (4°) point toward the connectivity of *p*-tolyl group to C15 the enolic center bearing hydroxyl group and Cα. Further confirmation of enolic center includes HMBC correlations from enolic-OH to C15, C16 (4°), C3 (Cα). The upfield shift of C3 to 98.6 ppm clearly indicates the characteristic nucleophilic Cα center of an enol. HMBC correlations from H14 (OCH_3) to ester carbonyl C13, and other correlations (Table S1) pertaining to the phenolic ring with *tert*-Bu substituent provides evidence that they are unaltered during reduction. The most downfield proton H4 (δ_H 7.59) corresponding to the pyridine core exhibited HMBC correlations with C2 (amide carbonyl), C13, C15, C5 (4° Cα to ester carbonyl) and C6, thus, confirming the position of C4 in the reduced product of 2-pyridone. This and presence of HMBC correlation from H6 to C5 altogether indicates that compound **9b** is a formal 1,6-reduction product of its corresponding 2-pyridone.



Promising HMBC correlations were observed in compound **9a** to reaffirm the formal 1,6-reduction structure from its corresponding 2-pyridone. The HMBC correlations include H14 to C13; H17 to H15; H6 to C2, C4, C5, C13; and H4 to C2, C3, C15, C5, C13, C6.

2. Table S1: ^{1}H, ^{13}C and HMBC correlations of compounds 3a and 9b

	Compound 3a			Compound 9b		
Carbon	^{13}C ppm	^{1}H ppm	HMBC	^{13}C ppm	^{1}H ppm	HMBC
2	160.79			169.20		
3	130.06			98.62		
4	140.94	8.29, d (2.5)	15, 2, 6	134.30	7.59, s	6, 5, 13, 2, 15
5	110.86			114.58		
6	146.18	8.38, d (2.5)	13, 2, 4, 7	51.77	4.72, bs	5
7	128.58			128.65		
8	126.88	7.18, d (8)	12, 7	119.05	7.05, d (8)	7, 10
9	121.07	6.97, td (8,1)	7, 11	126.57	7.33, d (8)	21, 11
10	131.04	7.19, td (8,1)	12	145.18		
11	119.45	6.85, d (8)	9, 7, 12	122.29	7.29, m (1)	21, 9, 12
12	151.85			148.59		
13	163.88			165.31		
14	52.48	3.88, s	13	51.83	3.76, s	13
15	192.51			175.20		
16	133.91			130.75		
17	129.81	7.78, d (8)	15, 19, 17	129.01	7.51, d (8)	15, 19, 17
18	129.34	7.24, d (8)	20, 16, 18	129.27	7.32, d (8)	20
19	144.66			141.75		
20	21.75	2.38, s	18, 19	21.57	2.46, s	19, 18
21				34.36		
22				31.47	1.34, s	10, 21
C12-OH		7.50, bs			5.84, bs	
C15-OH					15.10, bs	15, 16, 3

3. HMBC correlations of **3a**

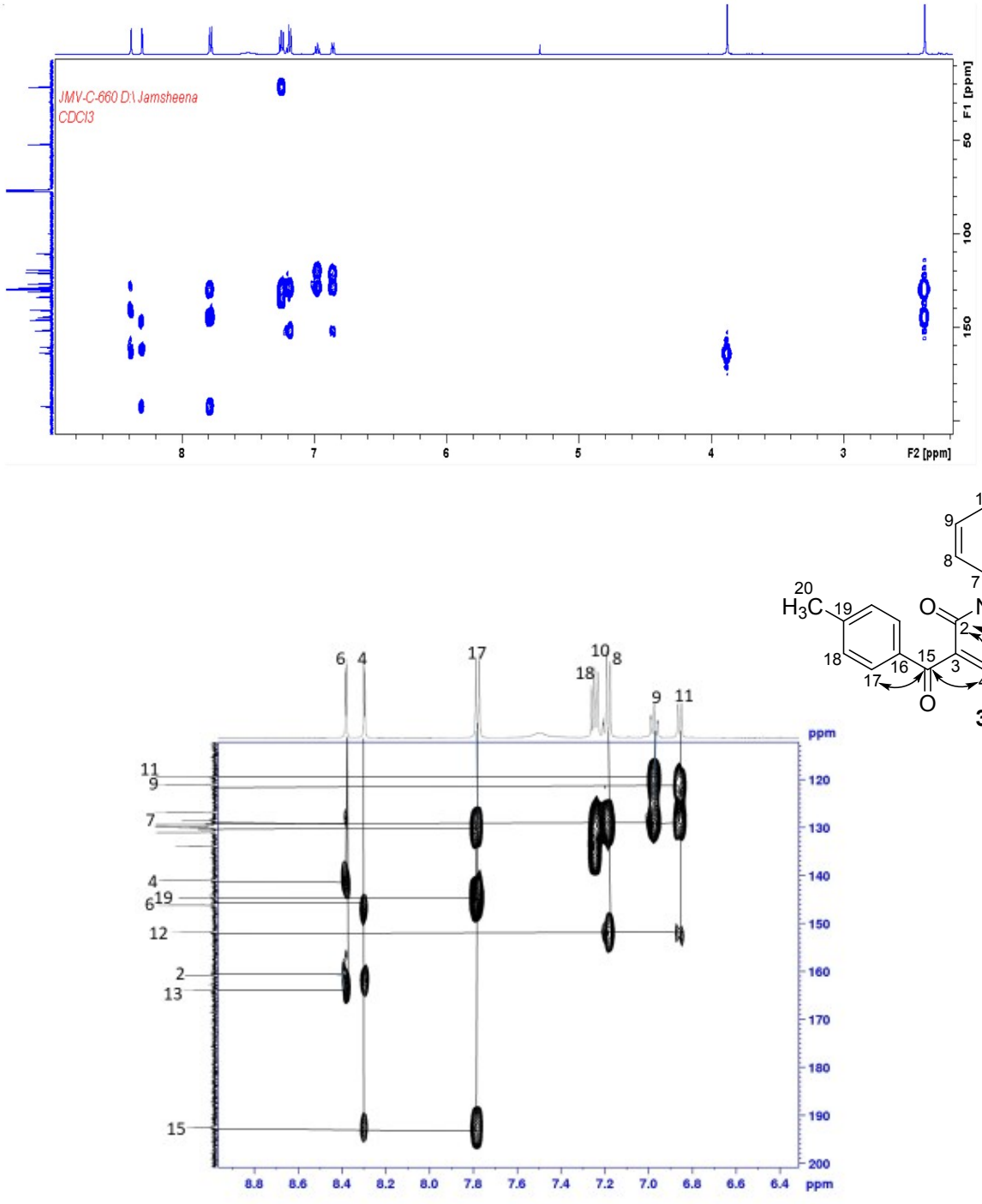


Figure S3a: Key HMBC correlations

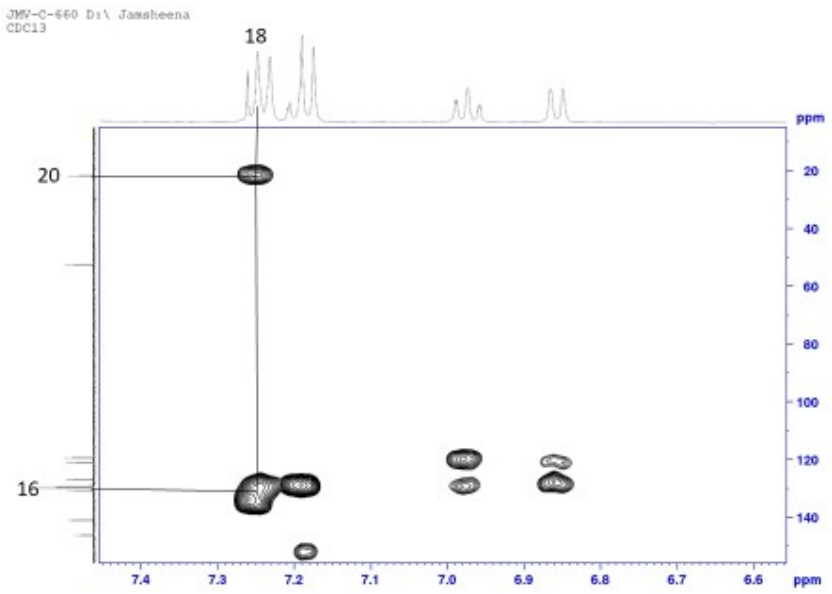


Figure S3b: Expanded HMBC spectra of 3a showing correlations of H18 with C16

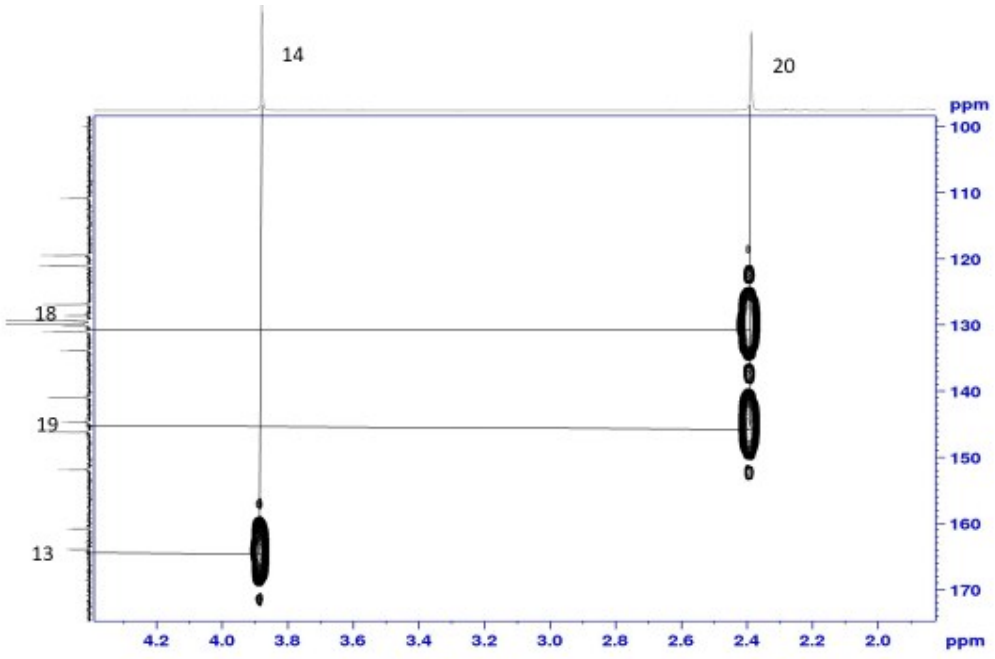
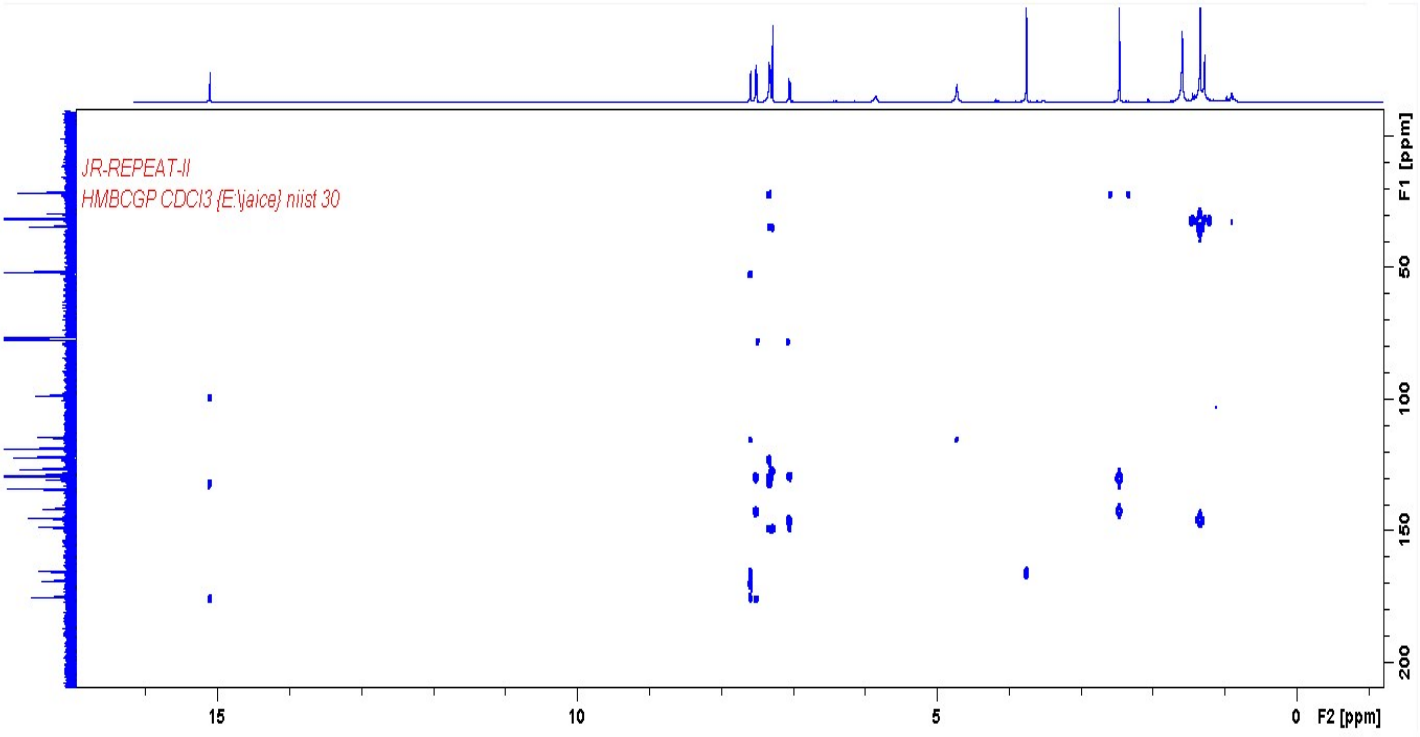


Figure S3c: Expanded HMBC spectra of 3a showing correlations of H14 and H20

4. HMBC correlations of **9b**



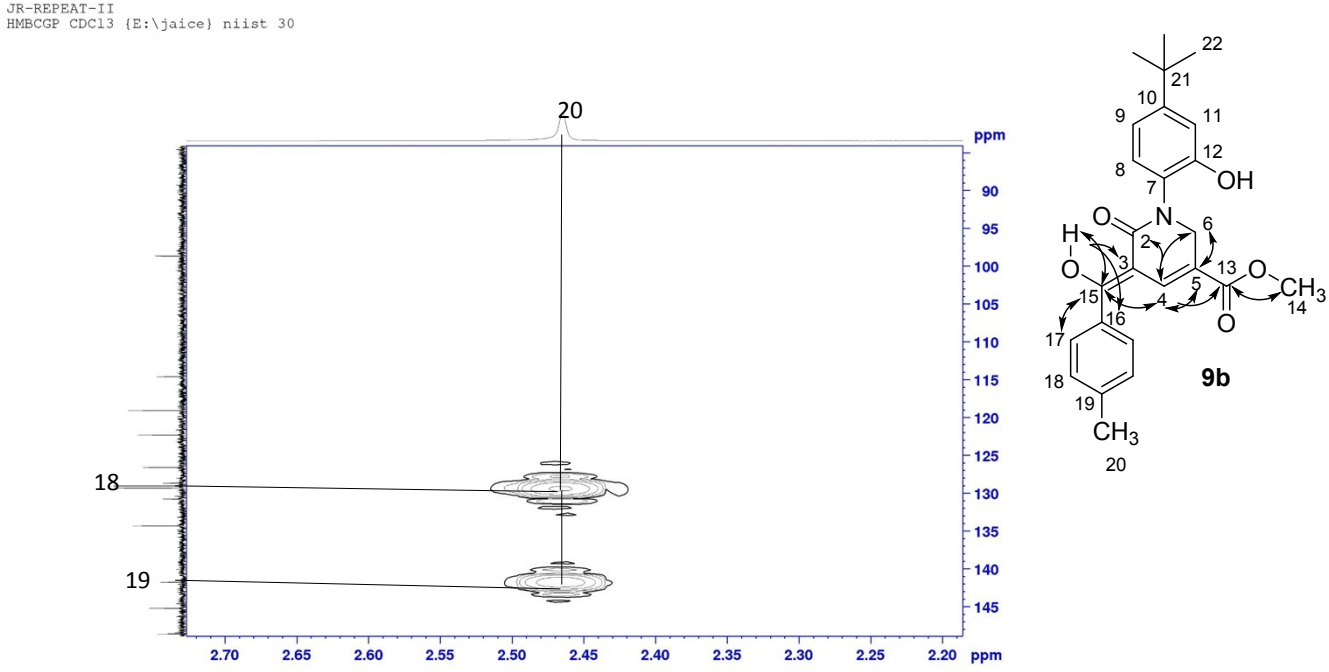


Figure S4a: Expanded HMBC spectra of 9b showing correlations of H20 with C18 and C19

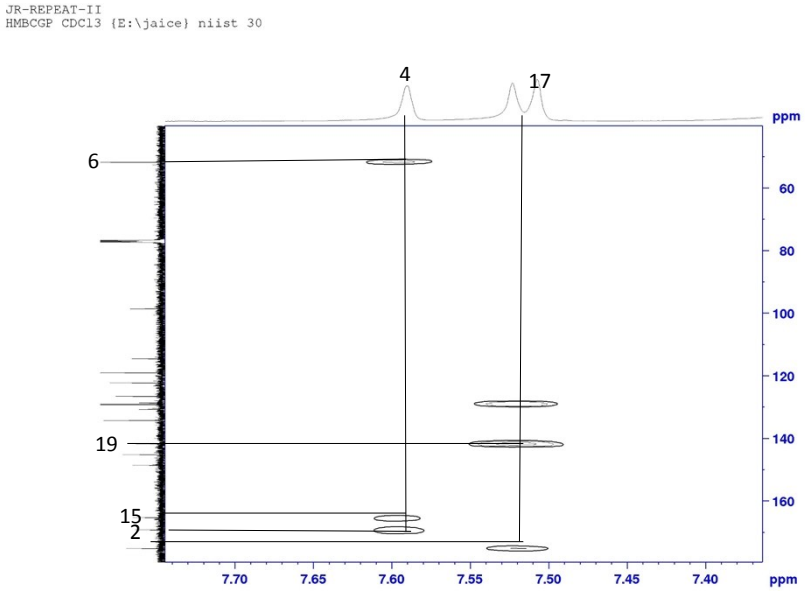


Figure S4b: Expanded HMBC spectra of 9b showing correlations of H4 and H17

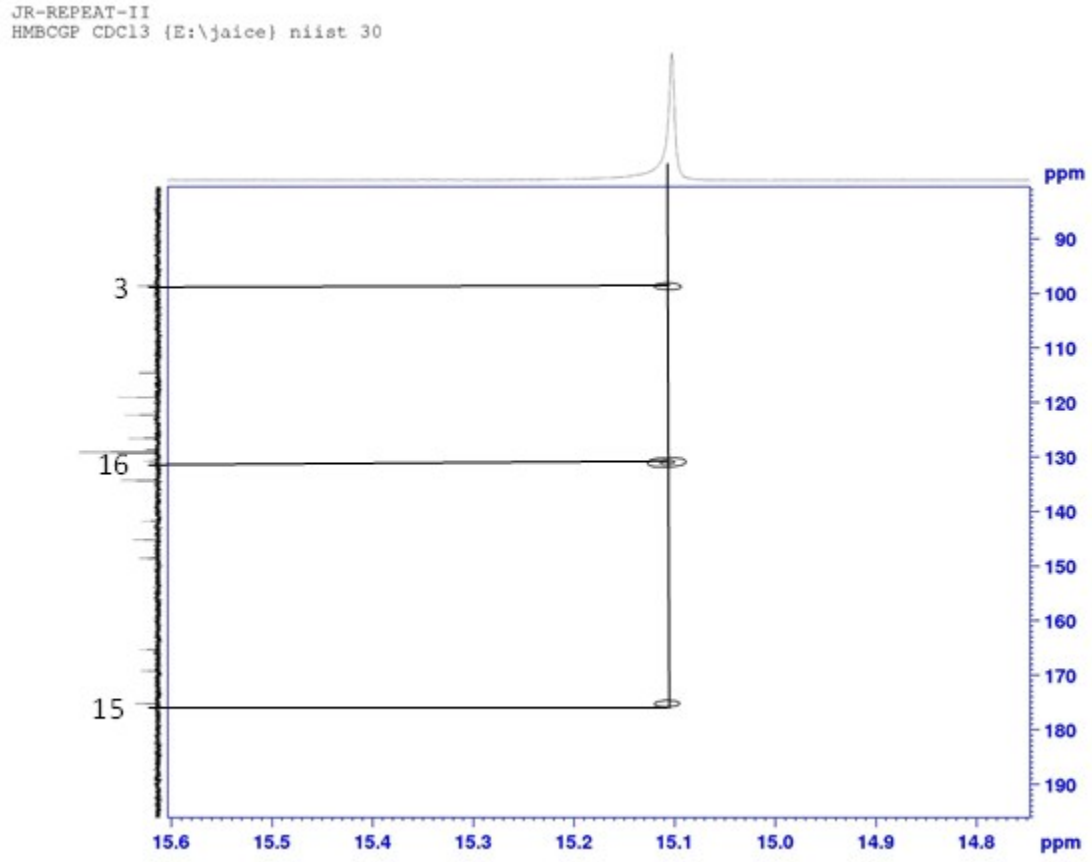
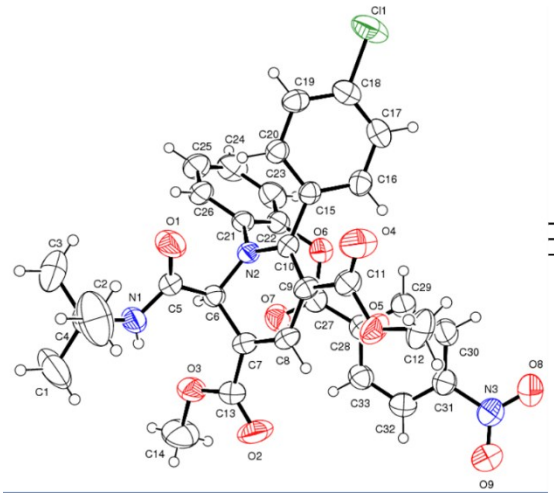


Figure S4c: Expanded HMBC spectra of 9b showing correlations of enolic-OH

5. Crystal information of para-nitroester derivative of 1,2-DHP 4c and benzo[*d*]oxazole appended biaryl 8b

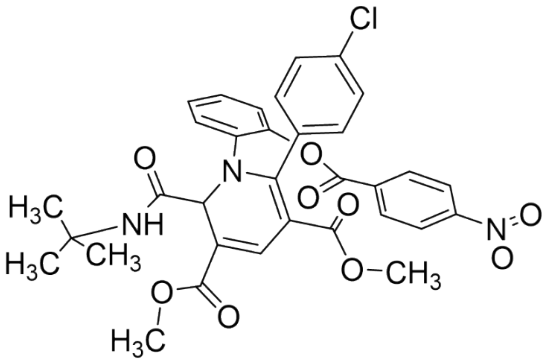
CCDC 1884531 (**para-nitroester derivative of 4c** (JR637)) and 1884529 (**8b** (JR749)) contain the crystallographic data for this manuscript. These data are available free of charge from the Cambridge Crystallographic Data Centre. Both the crystals were obtained from slow evaporation of the solvent system that was used for column purification i.e. 30:70 (EtOAc/Hexane).

ORTEP-40%

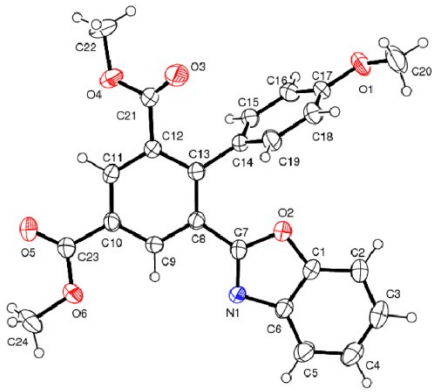


CCDC 1884531

Chemical Formula: $C_{33}H_{30}ClN_3O_9$



ORTEP-40%



CCDC 1884529

Chemical Formula: $C_{24}H_{19}NO_6$

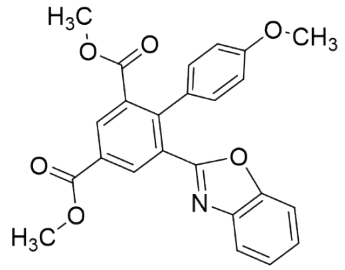


Table 1. Crystal data and structure refinement for jr637.

Identification code	JR637	
Empirical formula	C33 H30 Cl N3 O9	
Formula weight	648.05	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.3935(6) Å	α= 102.592(3)°.
	b = 10.3136(7) Å	β= 95.138(4)°.
	c = 17.8351(12) Å	γ = 101.978(3)°.
Volume	1633.08(19) $Å^3$	
Z	2	
Density (calculated)	1.318 Mg/m^3	
Absorption coefficient	0.175 mm^{-1}	
F(000)	676	
Crystal size	0.150 x 0.100 x 0.100 mm^3	
Theta range for data collection	3.018 to 24.999°.	
Index ranges	-11<=h<=11, -12<=k<=12, -21<=l<=21	
Reflections collected	46502	
Independent reflections	5738 [R(int) = 0.0555]	
Completeness to theta = 24.999°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7457 and 0.7151	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5738 / 0 / 420	
Goodness-of-fit on F^2	1.080	
Final R indices [I>2sigma(I)]	R1 = 0.0498, wR2 = 0.1267	
R indices (all data)	R1 = 0.0758, wR2 = 0.1504	
Extinction coefficient	0.032(3)	
Largest diff. peak and hole	0.349 and -0.232 $e.Å^{-3}$	

Table 1. Crystal data and structure refinement for jr749.

Identification code	JR749	
Empirical formula	C24 H19 N O6	
Formula weight	417.40	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.2622(7) Å	α= 83.258(4)°.
	b = 10.8562(10) Å	β= 82.820(4)°.
	c = 11.4554(8) Å	γ = 84.193(4)°.
Volume	1008.49(15) $Å^3$	
Z	2	
Density (calculated)	1.375 Mg/m^3	
Absorption coefficient	0.100 mm^{-1}	
F(000)	436	
Crystal size	0.200 x 0.150 x 0.150 mm^3	
Theta range for data collection	2.474 to 24.997°.	
Index ranges	-9<=h<=9, -12<=k<=12, -13<=l<=13	
Reflections collected	15132	
Independent reflections	3551 [R(int) = 0.0343]	
Completeness to theta = 24.997°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7453 and 0.6961	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3551 / 1 / 284	
Goodness-of-fit on F^2	1.051	
Final R indices [I>2sigma(I)]	R1 = 0.0433, wR2 = 0.1025	
R indices (all data)	R1 = 0.0828, wR2 = 0.1287	
Extinction coefficient	0.008(2)	
Largest diff. peak and hole	0.242 and -0.195 $e.Å^{-3}$	

6. Characterization data of 1,2-DHPs (1a-1za, 1f′′)

Dimethyl-1-(2-hydroxyphenyl)-2-(*p*-tolyl)-1,2-dihydropyridine-3,5-dicarboxylate (1a): Yield (146.2 mg, 87%). ^{1}H NMR ($CDCl_3$): δ 2.31 (s, 3H), 3.71 (s, 3H), 3.79 (s, 3H), 5.92 (s, 1H), 6.81 (t, *J* = 7.5 Hz, 1H), 6.87 (dd, *J* = 8.0, 1.5 Hz, 1H), 6.92 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.05 (d, *J* = 1.0 Hz, 2H), 7.16 (m, 3H), 7.73 (s, 1H), 7.88 (d, *J* = 1.0 Hz, 1H); ^{13}C{1H} NMR ($CDCl_3$): δ 14.2, 21.2, 51.3, 51.7, 60.5, 62.2, 99.1, 113.7, 117.3, 120.9, 127.2, 128.1, 129.2, 129.4, 131.3, 132.2 137.9, 138.5, 150.1, 150.7, 166.5, 166.6; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{22}NO_5$ 380.1498; Found 380.1503.

Dimethyl-1-(2-hydroxyphenyl)-2-phenyl-1,2-dihydropyridine-3,5-dicarboxylate (1b): Yield (108.7 mg, 67%) yellow solid. Mp 170-172 °C. ^{1}H NMR ($CDCl_3$): δ 3.62 (s, 3H), 3.71 (s, 3H), 5.89 (s, 1H), 6.30 (bs, 1H), 6.70 (t, *J* = 7.5 Hz, 1H), 6.76 (dd, *J* = 6.5, 1.0 Hz, 1H), 6.82 (d, *J* = 8.0 Hz, 1H), 7.05 (td, *J* = 7.0, 1.5 Hz, 1H), 7.17 (m, 2H), 7.19 (m, 2H), 7.66 (s, 1H), 7.80 (s, 1H); ^{13}C{1H} NMR ($CDCl_3$): δ 51.3, 51.7, 55.2, 61.9, 99.0, 113.7, 113.9, 117.3, 121.0, 128.2, 128.6, 129.2, 131.2, 132.1, 133.2, 150.0, 150.7, 159.8, 166.5, 166.6; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{20}NO_5$ 366.1341; Found 366.1340.

Dimethyl-2-(4-fluorophenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1c): Yield (106.3 mg, 63%) yellow solid. Mp 192-195 °C. ^{1}H NMR ($CDCl_3$): δ 3.63 (s, 3H), 3.71 (s, 3H), 5.61 (bs, 1H), 5.82 (s, 1H), 6.75 (m, 2H), 6.84 (m, 3H), 7.10 (m, 1H), 7.11 (m, 2H), 7.62 (s, 1H), 7.80 (d, *J* = 1.0 Hz, 1H); ^{13}C{1H} NMR ($CDCl_3$): δ 51.4, 51.8, 61.6, 98.8, 113.4, 115.3, 117.2, 117.3, 120.9, 121.3, 127.9, 129.1, 130.9, 132.5, 136.6, 150.3, 150.6, 162.8 (d, *J* = 247 Hz), 166.5, 166.7; ^{19}F NMR ($CDCl_3$): δ -112.97; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{19}FNO_5$ 384.1247; Found 384.1255.

Dimethyl-2-(4-bromophenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1d): Yield (121.2 mg, 62%). ^{1}H NMR ($CDCl_3$): δ 3.63 (s, 3H), 3.71 (s, 3H), 5.93 (s, 1H), 6.71 (m, 3H), 6.81 (d, *J* = 8.0 Hz, 1H), 7.04 (t, *J* = 7.0 Hz, 1H), 7.10 (m, 4H), 7.69 (s, 1H), 7.79 (s, 1H); ^{13}C{1H} NMR ($CDCl_3$): δ 51.5, 51.8, 61.6, 98.9, 113.2, 117.3, 121.0, 127.8, 128.72, 128.74, 129.2, 130.8, 132.6, 134.4, 139.1, 150.4, 150.5, 166.5, 166.7; HR-ESI-MS *m/z*: $[M]^+$ Calcd for $C_{21}H_{18}BrNNaO_5$ 466.0266; Found 466.0268.

Dimethyl-2-(4-chlorophenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1e): Yield (105.6mg, 60%) yellow solid. Mp 176-178 °C. ^{1}H NMR ($CDCl_3$): δ 3.63 (s, 3H), 3.71 (s, 3H), 5.86 (s, 1H), 6.74 (m, 2H), 6.82 (d, *J* = 8.0 Hz, 1H), 7.07 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8.5 Hz, 2H), 7.65 (s, 1H), 7.78 (d, *J* = 1.0 Hz, 1H); ^{13}C{1H} NMR ($CDCl_3$):

δ 51.4, 51.8, 61.8, 99.3, 113.4, 117.3, 121.2, 128.0, 128.7, 128.8, 129.3, 130.9, 132.4, 134.5, 139.1, 150.1, 150.4, 166.3, 166.5; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{19}ClNO_5$ 400.0952; Found 400.0011.

Dimethyl-2-(4-cyanophenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1f): Yield (135.2 mg, 78%) yellow solid. Mp 177-179 °C. ^{1}H NMR ($CDCl_3$): δ 3.73 (s, 3H), 3.81 (s, 3H), 6.10 (s, 3H), 6.84 (m, 2H), 6.92 (d, *J* = 8.0 Hz, 1H), 7.16 (td, *J* = 8.5, 1.5 Hz, 1H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.81 (s, 1H), 7.88 (s, 1H); ^{13}C{1H} NMR ($CDCl_3$): δ 51.5, 51.9, 61.9, 99.1, 112.2, 112.7, 117.3, 118.5, 121.0, 127.4, 128.0, 129.3, 130.7, 132.4, 132.9, 145.3, 150.6, 166.2, 166.6; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{19}N_2O_5$ 391.1294; Found 391.1308.

Dimethyl-1-(2-hydroxyphenyl)-2-(4-methoxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1g): Yield (124.7 mg, 71%) yellow solid. Mp 169-171 °C. ^{1}H NMR ($CDCl_3$): δ 3.71 (s, 3H), 3.78 (s, 3H), 3.79 (s, 3H), 5.85 (s, 1H), 6.78 (d, *J* = 8.5 Hz, 1H), 6.85 (m, 2H), 6.92 (d, *J* = 8.0 Hz, 1H), 7.19 (m, 3H), 7.69 (s, 1H), 7.88 (s, 1H); ^{13}C{1H} NMR ($CDCl_3$): δ 51.2, 51.6, 55.2, 62.2, 99.4, 114.1, 117.3, 121.3, 128.4, 128.6, 129.4, 131.3, 131.9, 133.2, 149.6, 150.7, 160.0, 166.2, 166.3; HR-ESI-MS *m/z*: $[M-H]^+$ Calcd for $C_{22}H_{20}NO_6$ 394.1291; Found 394.1306.

Dimethyl-2-(4-(*tert*-butyl)phenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1h): Yield (135.4 mg, 73%) yellow solid. Mp 186-189 °C. ^{1}H NMR ($CDCl_3$): δ 1.19 (s, 9H), 3.63 (s, 3H), 3.70 (s, 3H), 5.79 (s, 1H), 6.74 (t, *J* = 7.5 Hz, 1H), 6.82 (m, 2H), 7.08 (td, *J* = 8.5, 1.5 Hz, 1H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.18 (m, 3H), 7.63 (s, 1H), 7.79 (d, *J* = 1.0 Hz, 1H); ^{13}C {1H}NMR ($CDCl_3$): δ 31.3, 34.6, 51.3, 51.6, 62.3, 99.9, 113.7, 117.3, 121.2, 125.7, 126.7, 128.2, 129.3, 131.6, 132.0, 137.6, 149.7, 150.7, 151.7, 166.3, 166.4; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{25}H_{28}NO_5$ 422.1967; Found 422.1987.

Dimethyl-2-(3-chlorophenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1i): Yield (123.1 mg, 70%). ^{1}H NMR ($CDCl_3$): δ 3.62 (s, 3H), 3.69 (s, 3H), 5.96 (s, 1H), 6.66 (t, *J* = 7.5 Hz, 1H), 6.72 (dd, *J* = 7.5, 1.5 Hz, 1H), 6.82 (d, *J* = 8.0 Hz, 1H), 7.04 (m, 3H), 7.12 (m, 1H), 7.70 (s, 1H), 7.77 (d, *J* = 1.0 Hz, 1H); ^{13}C{1H} NMR ($CDCl_3$): δ 51.3, 51.7, 61.6, 98.7, 112.8, 117.1, 120.5, 125.5, 127.4, 127.8, 128.5, 129.1, 129.7, 131.0, 132.6, 134.4, 142.7, 150.5, 150.8, 166.4, 166.6; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{19}ClNO_5$ 400.0952; Found 400.0962.

Dimethyl-2-(3-fluorophenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1j): Yield (124.8 mg, 74%). ^{1}H NMR ($CDCl_3$): δ 3.64 (s, 3H), 3.71 (s, 3H), 5.90 (t, *J* = 9.5 Hz, 1H), 6.83 (d, *J* = 8.0 Hz, 1H), 6.88 (t, *J* = 8.5 Hz, 1H), 6.95 (m, 2H), 7.10 (m, 2H), 7.19 (s, 1H), 7.67 (s, 1H), 7.80 (s, 1H); ^{13}C{1H} NMR ($CDCl_3$): δ 51.4, 51.8, 61.8, 99.2, 113.1, 114.2, 114.3, 115.4, 115.6, 117.3, 121.0, 123.0, 127.8, 129.3, 130.1, 130.9, 132.6, 143.0, 150.3, 150.4, 162.95 (d, *J* = 247 Hz), 166.5, 166.6; ^{19}F NMR ($CDCl_3$): δ -112.1; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{19}FNO_5$ 384.1247; Found 384.1255.

Dimethyl-2-(2-fluorophenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1k): Yield (138.3 mg, 82%) yellow solid. Mp 173-175 °C. ^{1}H NMR ($CDCl_3$): δ 3.60 (s, 3H), 3.70 (s, 3H), 6.35 (s, 1H), 6.44 (bs, 1H), 6.70 (m, 2H), 6.77 (t, *J* = 9.0 Hz, 1H), 6.83 (d, *J* = 8.0 Hz, 1H), 7.00 (t, *J* = 7.5 Hz, 1H), 7.05 (td, *J* = 9.0, 2.0 Hz, 1H), 7.14 (m, 1H), 7.48 (td, *J* = 7.5, 1.5 Hz, 1H), 7.60 (s, 1H), 7.84 (d, *J* = 1.0 Hz, 1H); ^{13}C{1H} NMR ($CDCl_3$): δ 51.3, 51.7, 55.3, 98.8, 112.8, 115.2, 115.3, 117.1, 121.0, 124.7, 128.0, 129.4, 130.0, 130.4, 130.8, 133.0, 150.3, 150.7, 158.1-160.0 (d, *J* = 249 Hz), 166.0, 166.5; ^{19}F NMR ($CDCl_3$): δ -118.8; HR-ESI-MS *m/z*: $[M+Na]^+$ Calcd for $C_{21}H_{18}FNNaO_5$ 406.1067; Found 406.1076.

Dimethyl-2-(2-bromophenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1l): Yield (154.4 mg, 79%). ^{1}H NMR ($CDCl_3$): δ 3.59 (s, 3H), 3.70 (s, 3H), 6.44 (s, 1H), 6.59 (d, *J* = 8.0 Hz, 1H), 6.67 (t, *J* = 7.5 Hz, 1H), 6.85 (d, *J* = 8.0 Hz, 1H), 7.02 (t, *J* = 7.5 Hz, 1H), 7.09 (t, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 7.5 Hz, 1H), 7.26 (d, *J* = 8.0 Hz, 1H), 7.52 (s, 1H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.84 (s, 1H); ^{13}C{1H} NMR ($CDCl_3$): δ 51.4, 51.7, 60.6, 98.2, 114.4, 117.2, 120.9, 122.6, 128.5, 128.8, 129.8, 130.1, 130.4, 132.5, 132.8, 140.5, 150.5, 151.2, 166.0, 166.5; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{19}BrNO_5$ 444.0447: Found 444.0459.

Dimethyl-2-(4-chloro-2-fluorophenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1n): Yield (161.8 mg, 88%) yellow solid. Mp 175-177 °C. ^{1}H NMR ($CDCl_3$): δ 3.60 (s, 3H), 3.70 (s, 3H), 6.37 (s, 1H), 6.70 (m, 3H), 6.79 (m, 2H), 6.97 (d, *J* = 8.5 Hz, 1H), 7.03 (t, *J* = 8.0 Hz, 1H), 7.39 (t, *J* = 8.0 Hz, 1H), 7.61 (s, 1H), 7.83 (s, 1H); ^{13}C{1H} NMR ($CDCl_3$): δ 51.4, 51.8, 54.9, 98.4, 112.2, 115.9, 116.1, 117.1, 120.8, 125.0, 126.5, 127.6, 129.4, 130.5, 131.0, 133.4, 135.1, 135.2, 150.6, 150.8, 157.9-159.9 (d, *J* = 253 Hz), 166.2, 166.6; ^{19}F NMR ($CDCl_3$): δ -115.9; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{18}ClFNO_5$ 418.0858; Found 418.0859.

Dimethyl-2-(4-fluoro-3-methoxyphenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1o): Yield (71.0 mg, 39%) yellow solid. Mp 197-199 °C. ^{1}H NMR ($CDCl_3$): δ

3.61 (s, 3H), 3.64 (s, 3H), 3.70 (s, 3H), 5.84 (s, 1H), 6.04 (bs, 1H), 6.69 (dd, *J* = 8.0, 1.5 Hz, 1H), 6.76 (m, 3H), 6.85 (m, 2H), 7.09 (td, *J* = 8.5, 2.0 Hz, 1H), 7.63 (s, 1H), 7.81 (s, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 51.4, 51.8, 61.8, 99.3, 113.0, 117.3, 121.1, 125.5, 127.4, 127.9, 128.8, 129.4, 129.8, 130.9, 132.7, 134.6, 142.4, 150.2, 150.4, 160.0 (d, *J* = 204 Hz), 166.4, 166.5; ^{19}F NMR ($CDCl_3$): δ -134.7; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{21}FNO_6$ 414.1353; Found 414.1358.

Dimethyl-2-(2,4-dimethoxyphenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-di-carboxylate (1p): Yield (92.5 mg, 50%). ^1H NMR ($CDCl_3$): δ 3.25 (s, 3H), 3.58 (s, 3H), 3.68 (d, *J* = 1.5 Hz, 6H), 5.23 (s, 1H), 6.18 (d, *J* = 2.5 Hz, 1H), 6.27 (s, 1H), 6.34 (dd, *J* = 8.5, 2.5 Hz, 1H), 6.69 (t, *J* = 7.5 Hz, 1H), 6.75 (dd, *J* = 8.0, 1.5 Hz, 1H), 6.83 (d, *J* = 7.5 Hz, 1H), 7.04 (td, *J* = 8.0, 1.5 Hz, 1H), 7.26 (d, *J* = 8.5 Hz, 1H), 7.47 (s, 1H), 7.81 (d, *J* = 8.0 Hz, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 51.2, 51.5, 55.2, 55.3, 98.6, 98.7, 105.0, 113.3, 116.9, 120.6, 121.9, 128.0, 129.1, 130.4, 132.7, 132.7, 149.8, 151.3, 157.6, 161.2, 166.2, 166.5; HR-ESI-MS *m/z*: $[M]^+$ Calcd for $C_{23}H_{23}NO_7$ 425.1475; Found 425.1446.

Dimethyl-2-(2,5-dimethoxyphenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1q): Yield (83.0 mg, 44%). ^1H NMR ($CDCl_3$): δ 3.26 (s, 3H), 3.59 (s, 3H), 3.64 (s, 3H), 3.68 (s, 3H), 6.33 (s, 1H), 6.59 (d, *J* = 9.0 Hz, 1H), 6.69 (m, 2H), 6.78 (dd, *J* = 7.5, 1.0 Hz, 1H), 6.82 (d, *J* = 8.0 Hz, 1H), 6.94 (d, *J* = 3.0 Hz, 1H), 7.04 (td, *J* = 8.0, 1.0 Hz, 1H), 7.47 (s, 1H), 7.81 (d, *J* = 1.0 Hz, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 51.2, 51.5, 55.7, 56.1, 56.5, 98.8, 112.6, 113.4, 114.3, 115.7, 117.0, 120.7, 127.9, 129.2, 130.0, 131.5, 132.8, 150.0, 150.7, 151.4, 154.0, 166.0, 166.4; HR-ESI-MS *m/z*: $[M]^+$ Calcd for $C_{23}H_{23}NO_7$ 425.1475; Found 425.1438.

Dimethyl-2-(3,5-dimethylphenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1r): Yield (90 mg, 52%) yellow solid. Mp 216-219 °C. ^1H NMR ($CDCl_3$): δ 2.05 (s, 6H), 3.64 (s, 3H), 3.71 (s, 3H), 5.96 (s, 1H), 6.66 (t, *J* = 7.0 Hz, 1H), 6.71 (d, *J* = 7.5 Hz, 1H), 6.75 (s, 2H), 6.79 (d, *J* = 2.0 Hz, 1H), 6.98 (t, *J* = 7.5 Hz, 1H), 7.15 (bs, 1H), 7.66 (s, 1H), 7.82 (s, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 21.2, 51.3, 51.8, 62.1, 98.7, 113.2, 117.2, 120.7, 125.1, 128.3, 129.1, 130.5, 131.1, 132.7, 138.2, 140.6, 150.4, 150.7, 166.6, 166.9; HR-ESI-MS *m/z*: $[M+Na]^+$ Calcd for $C_{23}H_{23}NNaO_5$ 416.1474; Found 416.1488.

Dimethyl-1-(2-hydroxyphenyl)-2-(3,4,5-trimethoxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1s): Yield (92.2 mg, 46%). ^1H NMR ($CDCl_3$): δ 3.66 (s, 6H), 3.74 (s, 3H), 3.78 (s, 3H), 3.80 (s, 3H), 5.98 (s, 1H), 6.44 (s, 2H), 6.80 (t, *J* = 8.0 Hz, 1H), 6.88 (dd, *J* = 7.5, 1.5 Hz, 1H), 6.93 (d, *J* = 8.0 Hz, 1H), 7.12 (td, *J* = 8.0, 1.0 Hz, 1H), 7.77 (s, 1H), 7.89 (d, *J* = 1.0

Hz, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 51.4, 51.8, 56.0, 60.8, 62.4, 98.6, 104.4, 113.0, 117.3, 120.8, 127.9, 129.1, 131.2, 132.5, 135.8, 138.1, 150.2, 150.8, 153.2, 166.6, 166.7; HR-ESI-MS *m/z*: $[M+Na]^+$ Calcd for $C_{24}H_{25}NNaO_8$ 478.1478; Found 478.1482.

Dimethyl-2-(4-bromophenyl)-1-(4-(*tert*-butyl)-2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1t): Yield (182.0 mg, 83%). ^1H NMR ($CDCl_3$): δ 1.06 (s, 9H), 3.61 (s, 3H), 3.70 (s, 3H), 5.79 (s, 1H), 6.49 (bs, 1H), 6.59 (d, *J* = 2.0 Hz, 1H), 6.74 (d, *J* = 8.5 Hz, 1H), 7.04 (m, 3H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.65 (s, 1H), 7.79 (s, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 31.2, 34.0, 51.4, 51.7, 62.2, 98.8, 113.1, 116.7, 122.7, 125.3, 126.2, 129.2, 130.1, 131.6, 132.6, 139.7, 144.2, 148.0, 150.3, 166.3, 166.6; HR-ESI-MS *m/z*: $[M+Na]^+$ Calcd for $C_{25}H_{26}BrNNaO_5$ 522.0892; Found 522.0906.

Dimethyl-1-(4-(*tert*-butyl)-2-hydroxyphenyl)-2-(*p*-tolyl)-1,2-dihydropyridine-3,5-dicarboxylate (1u): Yield (162.0 mg, 85%). ^1H NMR ($CDCl_3$): δ 1.07 (s, 9H), 2.22 (s, 3H), 3.60 (s, 3H), 3.70 (s, 3H), 4.98 (bs, 1H), 5.64 (s, 1H), 6.65 (d, *J* = 2.0 Hz, 1H), 6.76 (d, *J* = 3.5 Hz, 1H), 6.97 (d, *J* = 2.0 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 2H), 7.11 (dd, *J* = 8.5, 2.5 Hz, 1H) 7.55 (s, 1H), 7.80 (d, *J* = 1.0 Hz, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 21.2, 31.2, 34.1, 51.2, 51.6, 62.9, 99.1, 113.8, 116.7, 125.7, 126.4, 127.3, 129.4, 130.6, 132.1, 138.0, 138.7, 144.3, 148.2, 149.9, 166.2, 166.4; HR-ESI-MS *m/z*: $[M+Na]^+$ Calcd for $C_{26}H_{29}NNaO_5$ 458.1943; Found 458.1955.

Dimethyl-1-(5-chloro-2-hydroxyphenyl)-2-(4-chlorophenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1v): Yield (168.5 mg, 88%). ^1H NMR ($CDCl_3$): δ 3.63 (s, 3H), 3.72 (s, 3H), 5.94 (s, 1H), 6.76 (d, *J* = 8.5 Hz, 1H), 6.81 (d, *J* = 2.5 Hz, 1H), 6.99 (dd, *J* = 9.0, 2.5 Hz, 1H), 7.11 (m, 4H), 7.65 (s, 1H), 7.76 (s, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 51.6, 51.9, 61.5, 99.6, 113.8, 118.3, 125.3, 127.3, 128.6, 128.9, 129.0, 131.6, 132.3, 134.7, 138.6, 149.4, 149.7, 166.4, 166.6; HR-ESI-MS *m/z*: $[M+Na]^+$ Calcd for $C_{21}H_{17}Cl_2NNaO_5$ 456.0381; Found 456.0390.

Dimethyl-1-(5-chloro-2-hydroxyphenyl)-2-(*p*-tolyl)-1,2-dihydropyridine-3,5-dicarboxylate (1w): Yield (148.0 mg, 81%). ^1H NMR ($CDCl_3$): δ 2.23 (s, 3H), 3.62 (s, 3H), 3.71 (s, 3H), 5.84 (s, 1H), 6.58 (bs, 1H), 6.75 (d, *J* = 9.0 Hz, 1H), 6.82 (d, *J* = 2.0 Hz, 1H), 6.97 (d, *J* = 7.5 Hz, 2H), 7.00 (dd, *J* = 9.0, 2.5 Hz, 1H), 7.06 (d, *J* = 8.0 Hz, 2H), 7.59 (s, 1H), 7.76 (s, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 21.2, 29.7, 51.4, 51.7, 62.2, 100.0, 114.3, 118.4, 125.4, 127.0, 127.8, 129.1, 129.6, 131.9, 132.1, 137.5, 138.9, 149.2, 149.6, 166.32, 166.38; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{21}ClNO_5$ 414.1108; Found 414.1118.

Dimethyl-2-(2-bromophenyl)-1-(5-chloro-2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1x): Yield (129.5 mg, 62%). ^1H NMR ($CDCl_3$): δ 3.59 (s, 3H), 3.71 (s, 3H), 5.23

(s, 1H), 6.43 (s, 1H), 6.49 (bs, 1H), 6.65 (d, *J* = 2.5 Hz, 1H), 6.80 (d, *J* = 8.5 Hz, 1H), 7.05 (m, 2H), 7.25 (t, *J* = 7.0 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.47 (s, 1H), 7.68 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.82 (d, *J* = 0.5 Hz, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 51.4, 51.8, 60.5, 98.9, 114.9, 118.3, 122.6, 125.2, 128.5, 128.7, 129.6, 130.30, 130.32, 131.2, 132.5, 132.7, 140.0, 149.7, 150.1, 165.9, 166.3; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{18}BrClNO_5$ 478.0057; Found 478.0066.

Dimethyl-2-(2-bromophenyl)-1-(4-chloro-2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1y): Yield (178.0 mg, 86%). ^1H NMR ($CDCl_3$): δ 3.59 (s, 3H), 3.71 (s, 3H), 6.39 (s, 1H), 6.51 (d, *J* = 8.5 Hz, 1H), 6.65 (dd, *J* = 8.5, 2.0 Hz, 1H), 6.89 (d, *J* = 2.5 Hz, 1H), 7.03 (td, *J* = 8.0, 1.5 Hz, 1H), 7.24 (t, *J* = 7.5 Hz, 1H), 7.29 (d, *J* = 8 Hz, 1H), 7.46 (s, 1H), 7.69 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.82 (d, *J* = 1.0 Hz, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 51.4, 51.8, 60.5, 98.6, 114.7, 117.5, 121.1, 122.7, 128.6, 129.5, 130.2, 130.4, 132.6, 132.7, 134.8, 140.2, 150.1, 152.1, 166.0, 166.4; HR-ESI-MS *m/z*: $[M+Na]^+$ Calcd for $C_{21}H_{17}BrClNNaO_5$ 499.9876; Found 499.9887.

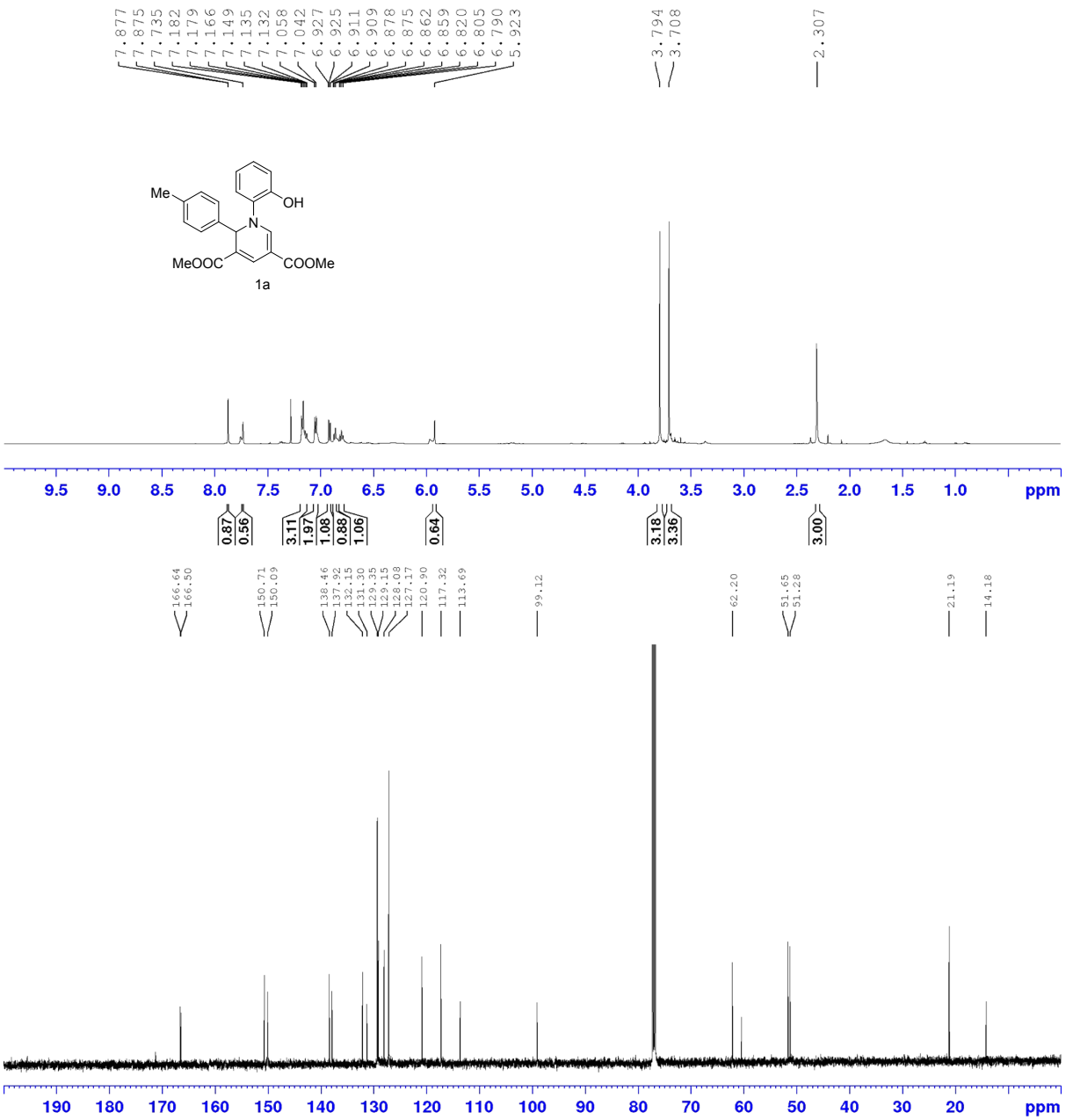
Dimethyl-2-(furan-2-yl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1z): Yield (143.0 mg, 92%) yellow solid. Mp 177-179 °C. ^1H NMR ($CDCl_3$): δ 3.67 (s, 3H), 3.70 (s, 3H), 5.98 (s, 1H), 6.08 (d, *J* = 3.0 Hz, 1H), 6.18 (m, 1H), 6.80 (t, *J* = 7.5 Hz, 1H), 6.87 (d, *J* = 8.0 Hz, 1H), 6.94 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.11 (td, *J* = 8.0, 1.0 Hz, 1H), 7.22 (d, *J* = 1.0 Hz, 1H), 7.61 (s, 1H), 7.80 (d, *J* = 0.5 Hz, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 51.3, 51.8, 55.5, 100.9, 108.7, 110.6, 110.7, 117.3, 121.2, 127.9, 129.4, 131.4, 133.1, 142.7, 149.2, 150.7, 152.8, 166.0, 166.3; HR-ESI-MS *m/z*: $[M]^+$ Calcd for $C_{19}H_{17}NO_6$ 355.1056; Found 355.1033.

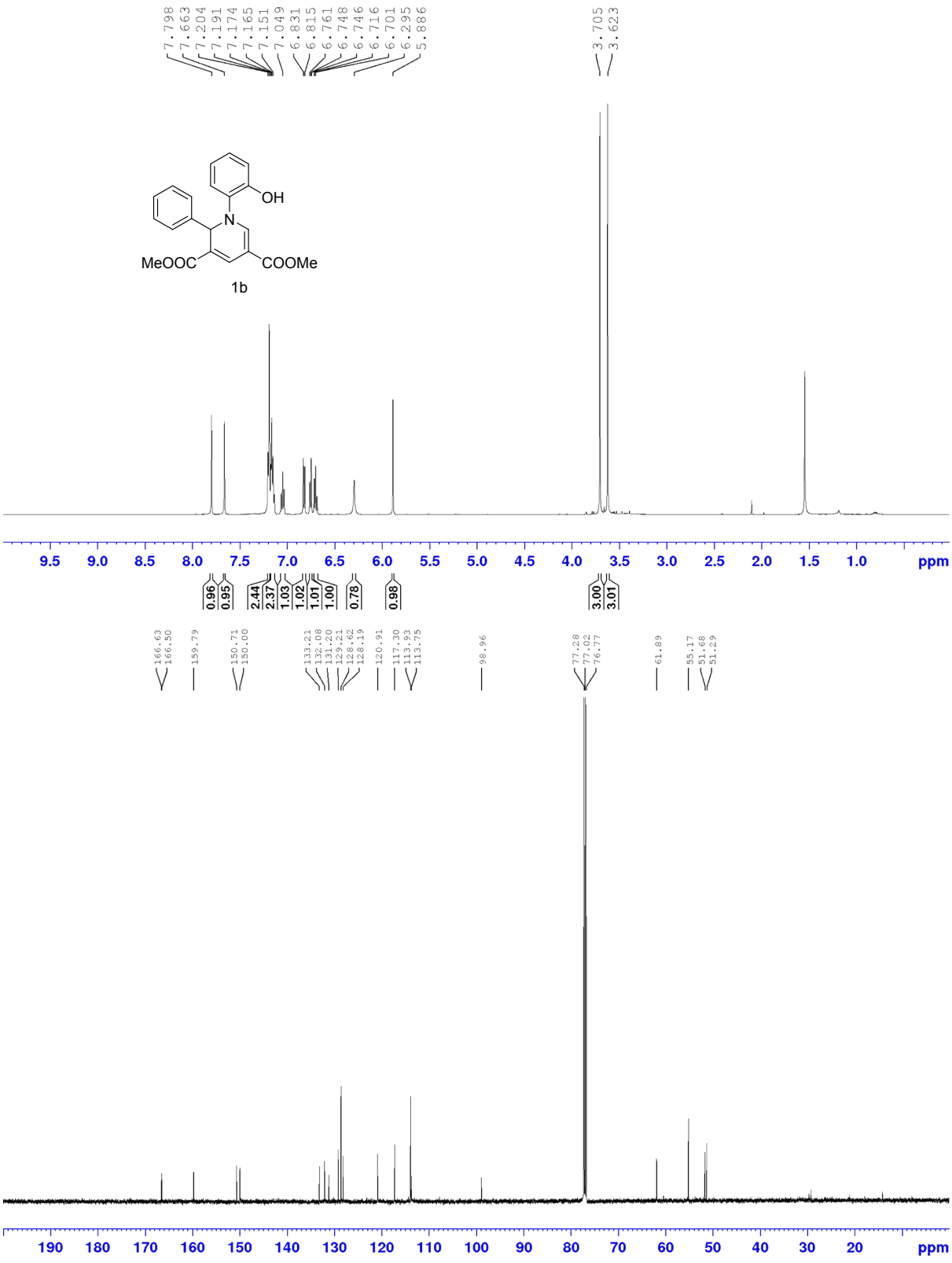
Dimethyl-1-(2-hydroxyphenyl)-2-(thiophen-2-yl)-1,2-dihydropyridine-3,5-dicarboxylate (1za): Yield (141.0 mg, 86%). ^1H NMR ($CDCl_3$): δ 3.67 (s, 3H), 3.70 (s, 3H), 6.02 (s, 1H), 6.42 (m ,1H), 6.50 (d, *J* = 3.5 Hz, 1H), 6.80 (t, *J* = 7.5 Hz, 1H), 6.95 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.12 (m, 1H), 7.19 (s, 1H), 7.59 (s, 1H), 7.75 (bs, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 51.3, 51.7, 57.7, 100.7, 113.7, 117.3, 121.4, 125.0, 126.1, 128.3, 129.5, 130.3, 131.2, 131.8, 140.7, 148.6, 150.6, 166.0, 166.1; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{19}H_{18}NO_5S$ 372.0906; Found 372.0880.

Dimethyl-2-(3,4-dichlorophenyl)-1-(2-hydroxyphenyl)-1,2-dihydropyridine-3,5-dicarboxylate (1f′′ – 1,2-DHP substrate for 4f): Yield (71.5 mg, 37%). ^1H NMR ($CDCl_3$): δ 3.74 (s, 3H), 3.81 (s, 3H), 5.98 (s, 1H), 6.17 (bs, 1H), 6.87 (m, 2H), 6.92 (d, *J* = 8.5 Hz, 1H), 7.18 (t, *J* = 7.0 Hz, 1H), 7.29 (m, 2H), 7.37 (s, 1H), 7.76 (s, 1H), 7.89 (s, 1H); $^{13}C\{1H\}$ NMR ($CDCl_3$): δ 51.4, 51.8, 61.4, 99.5, 113.0, 117.2, 121.5, 126.7, 128.0, 129.2, 129.5, 130.5, 132.6, 132.7,

132.8, 140.6, 142.8, 149.9, 150.1, 166.0, 167.1; HR-ESI-MS *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{18}Cl_2NO_5$ 434.0562; Found 434.0573.

7. NMR spectra of 1,2-DHPs

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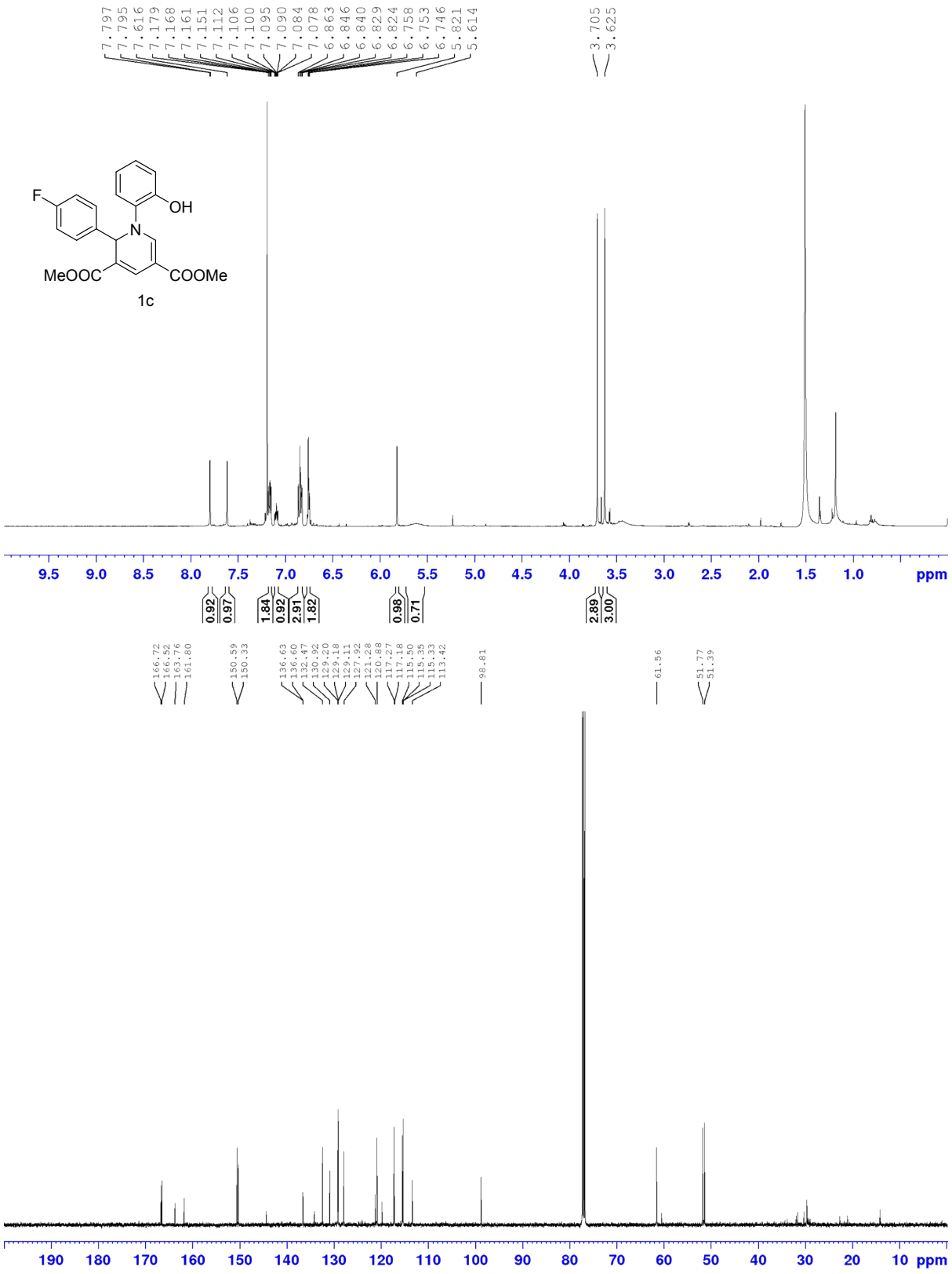
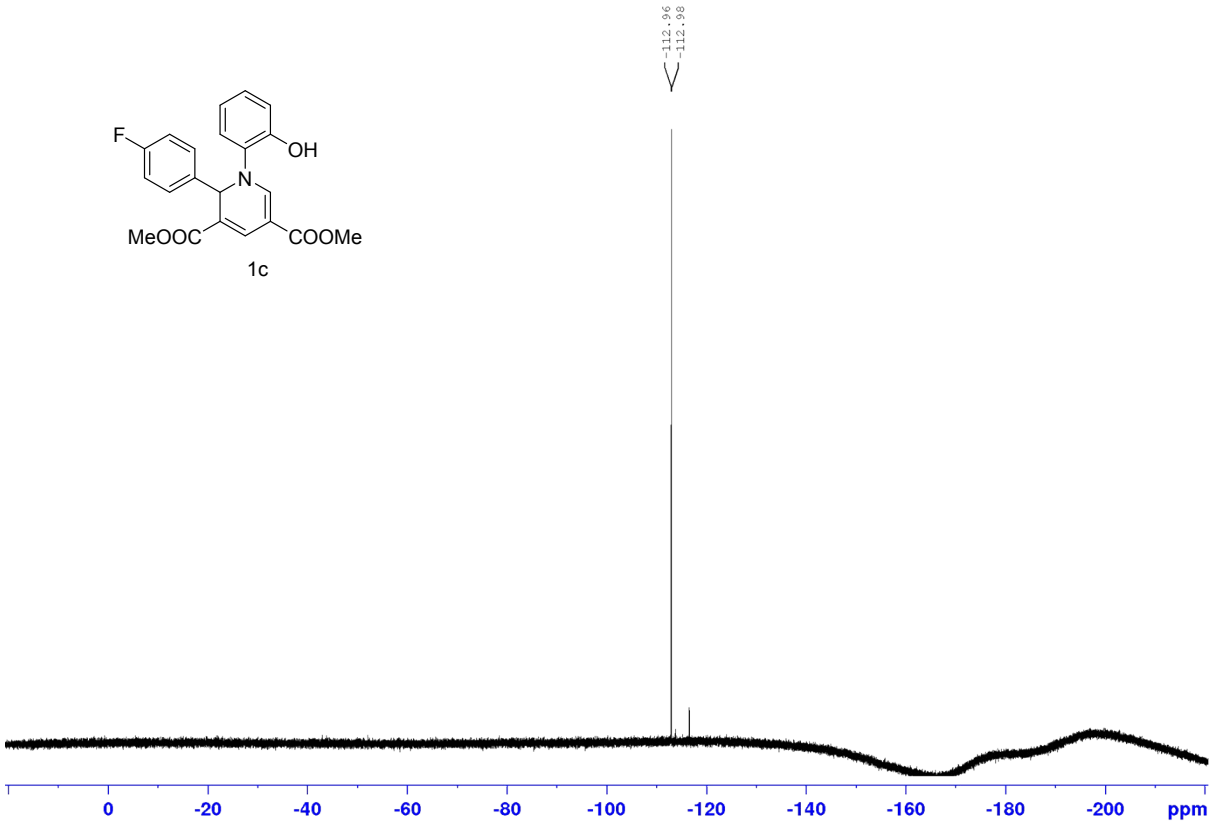
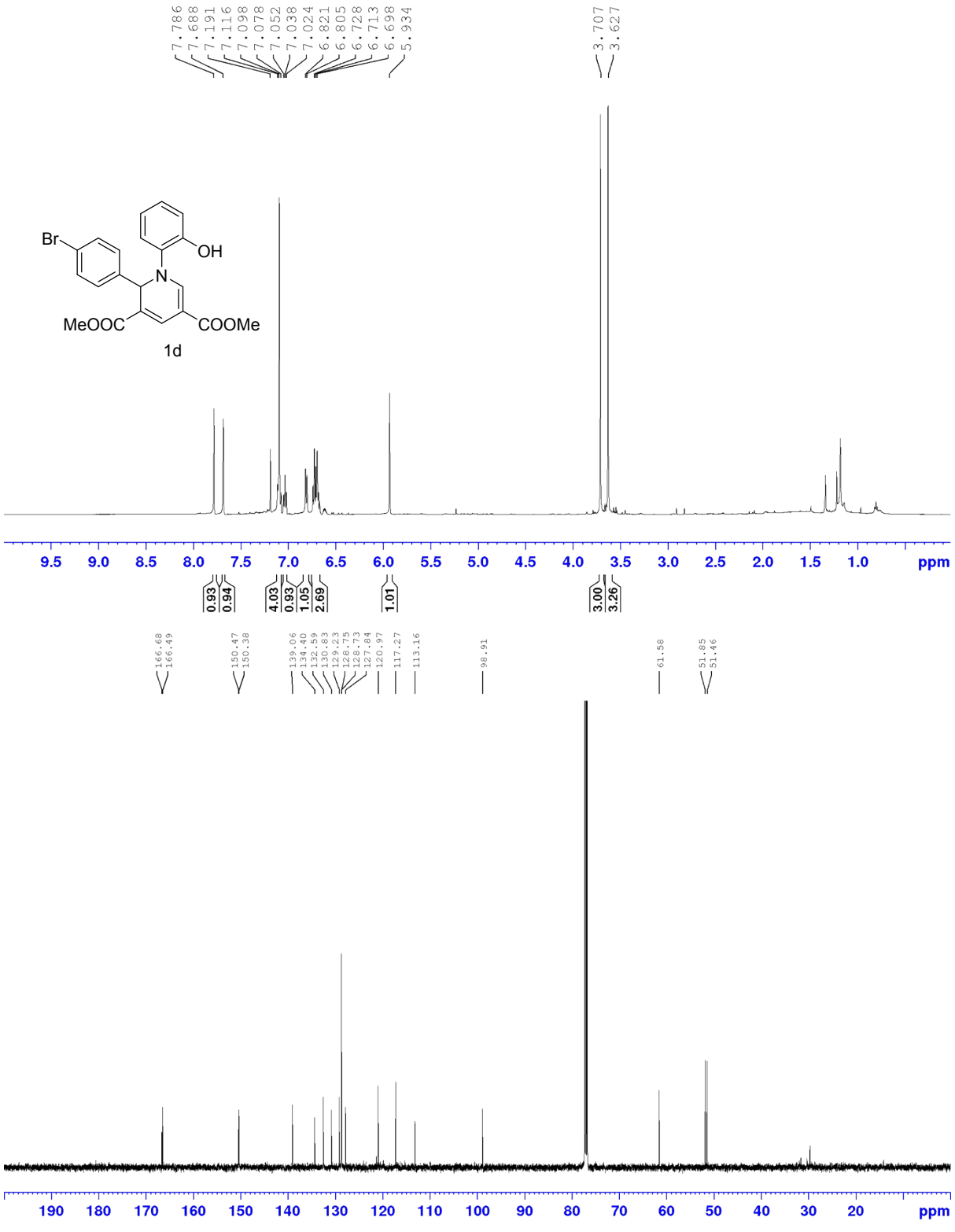
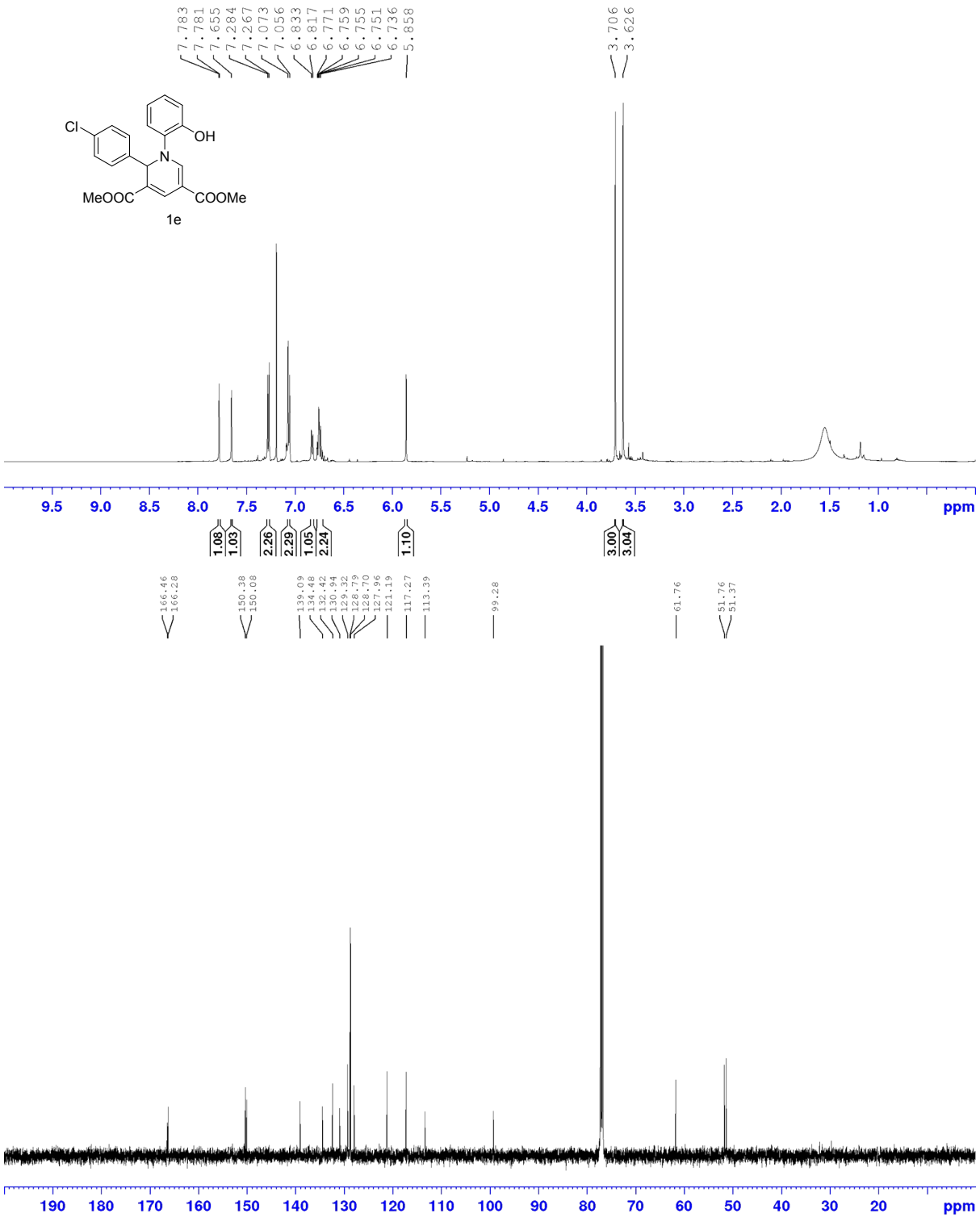
7.663
7.204
7.191
7.174
7.165
7.151
7.049
6.831
6.815
6.761
6.748
6.746
6.716
6.701
6.295
5.886
3.705
3.623
OH
N
MeOOC
COOMe
1b
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.96 0.95 2.44 2.37 1.03 1.02 1.01 1.00 0.78 0.98 3.00 3.01
166.63
166.50
159.79
150.71
150.00
133.21
132.08
131.20
129.21
128.62
128.19
120.91
117.30
113.93
113.75
98.96
77.28
77.02
76.77
61.89
55.17
51.68
51.29
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

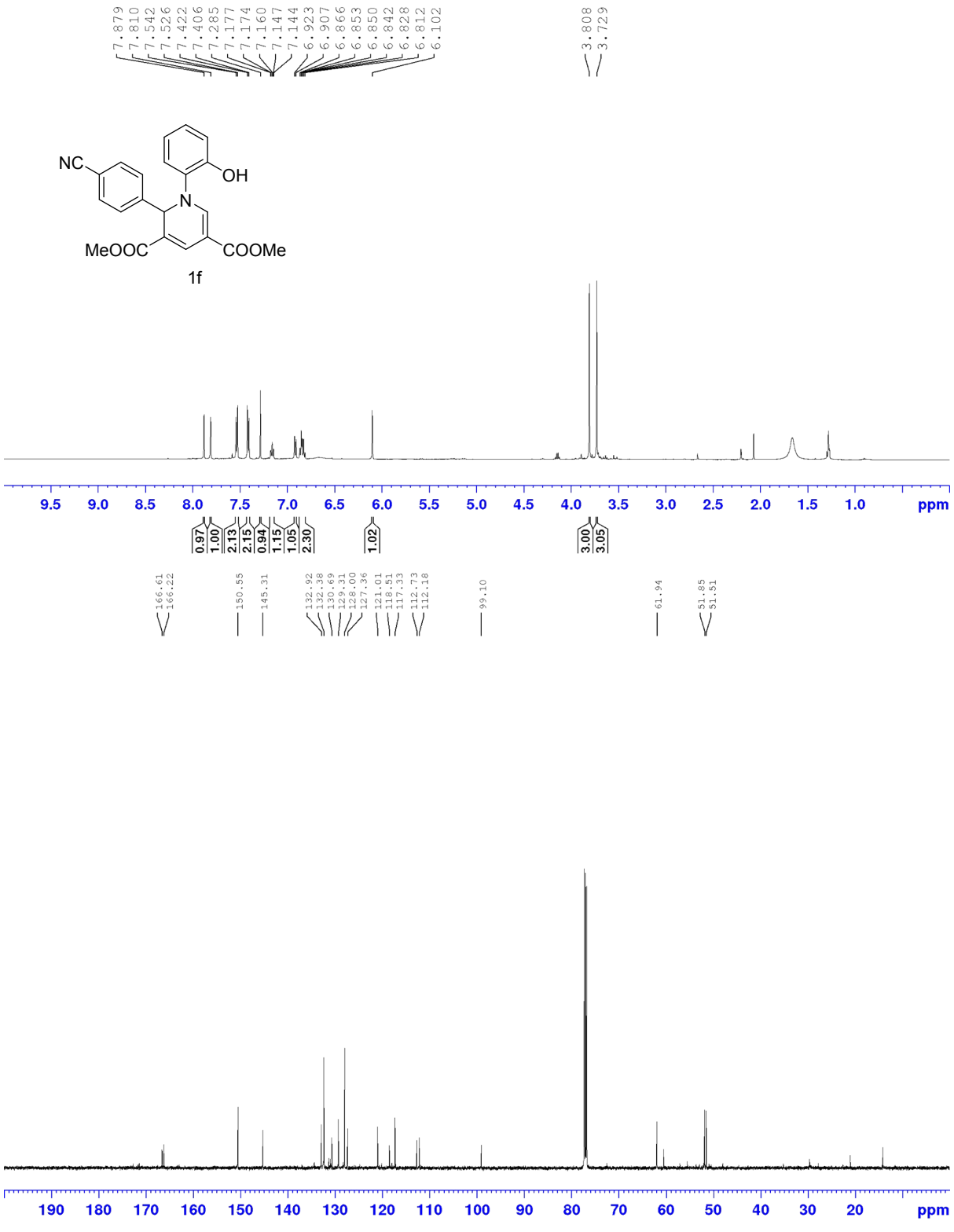

7.797
7.795
7.616
7.179
7.168
7.161
7.151
7.112
7.106
7.100
7.095
7.090
7.084
7.078
6.863
6.846
6.840
6.829
6.824
6.758
6.753
6.746
5.821
5.614
3.705
3.625
F
OH
N
MeOOC
COOMe
1c
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.92
0.97
1.84
0.92
2.91
1.82
0.98
0.71
2.89
3.00
166.72
166.52
163.76
161.80
150.59
150.33
136.63
136.60
132.47
130.92
129.20
129.18
129.11
127.92
121.28
120.88
117.27
117.18
115.50
115.35
115.33
113.42
98.81
61.56
51.77
51.39
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

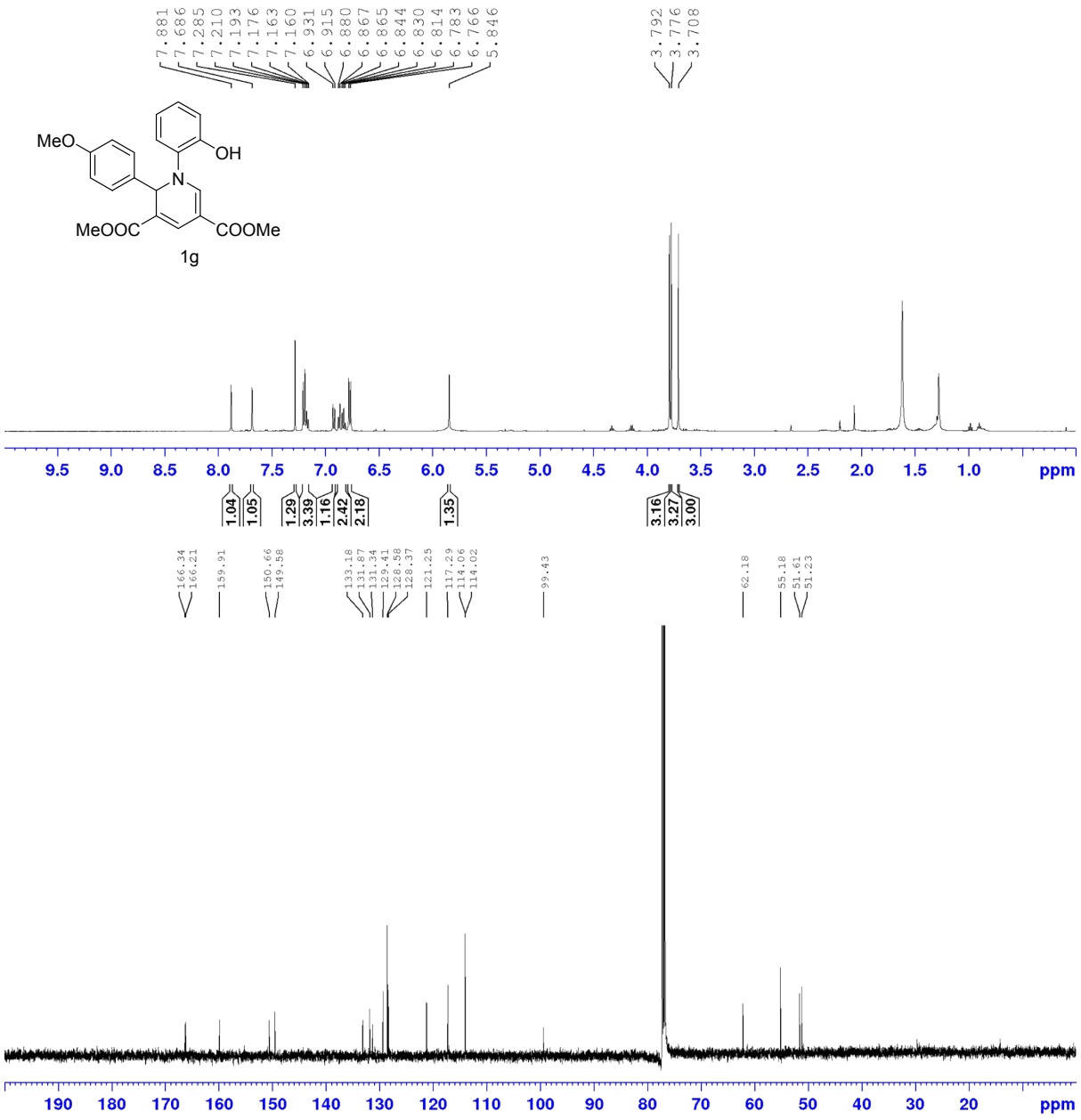
JR-3C
F19CPD CDCl3 {E:\jaice} niist 21

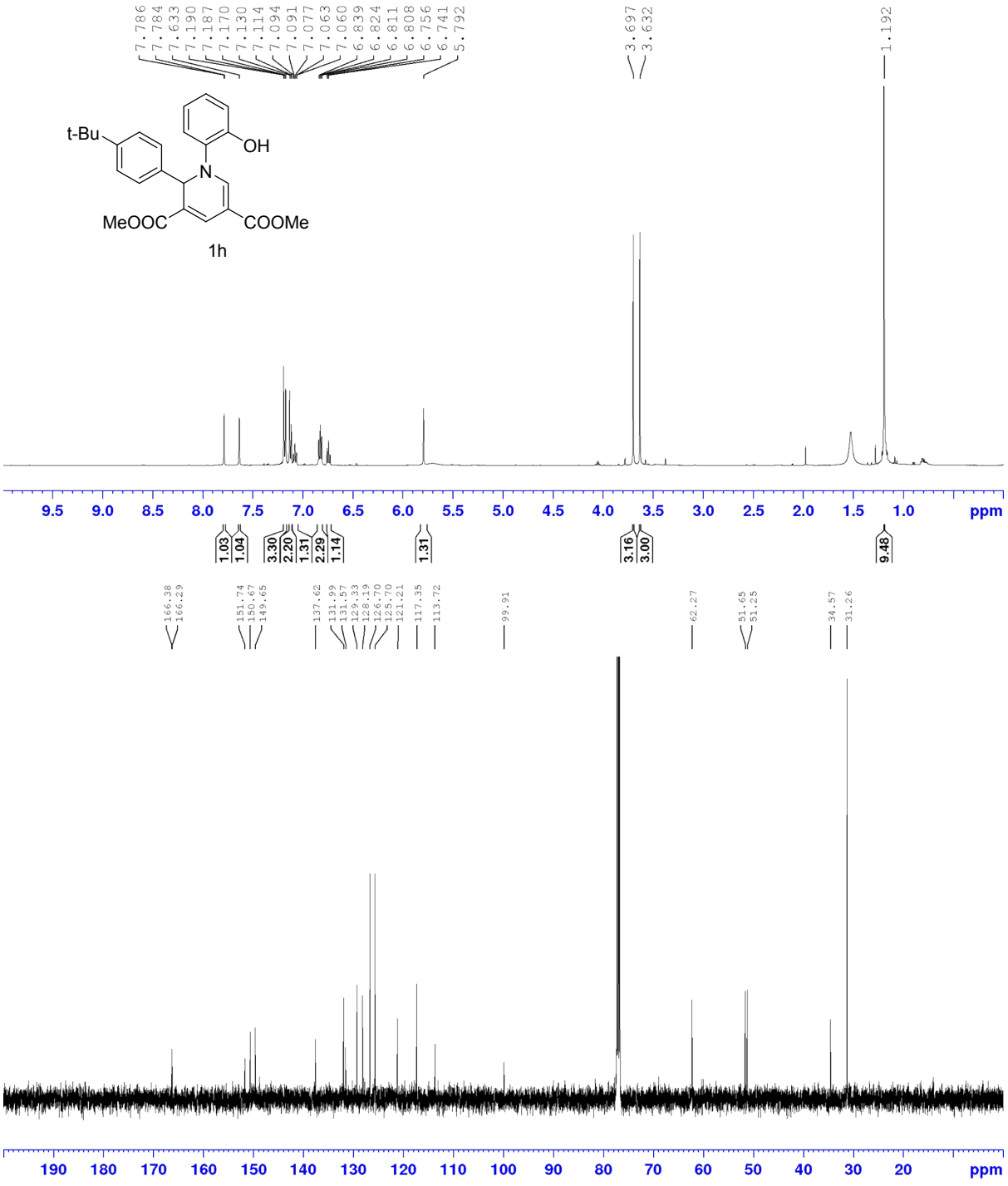


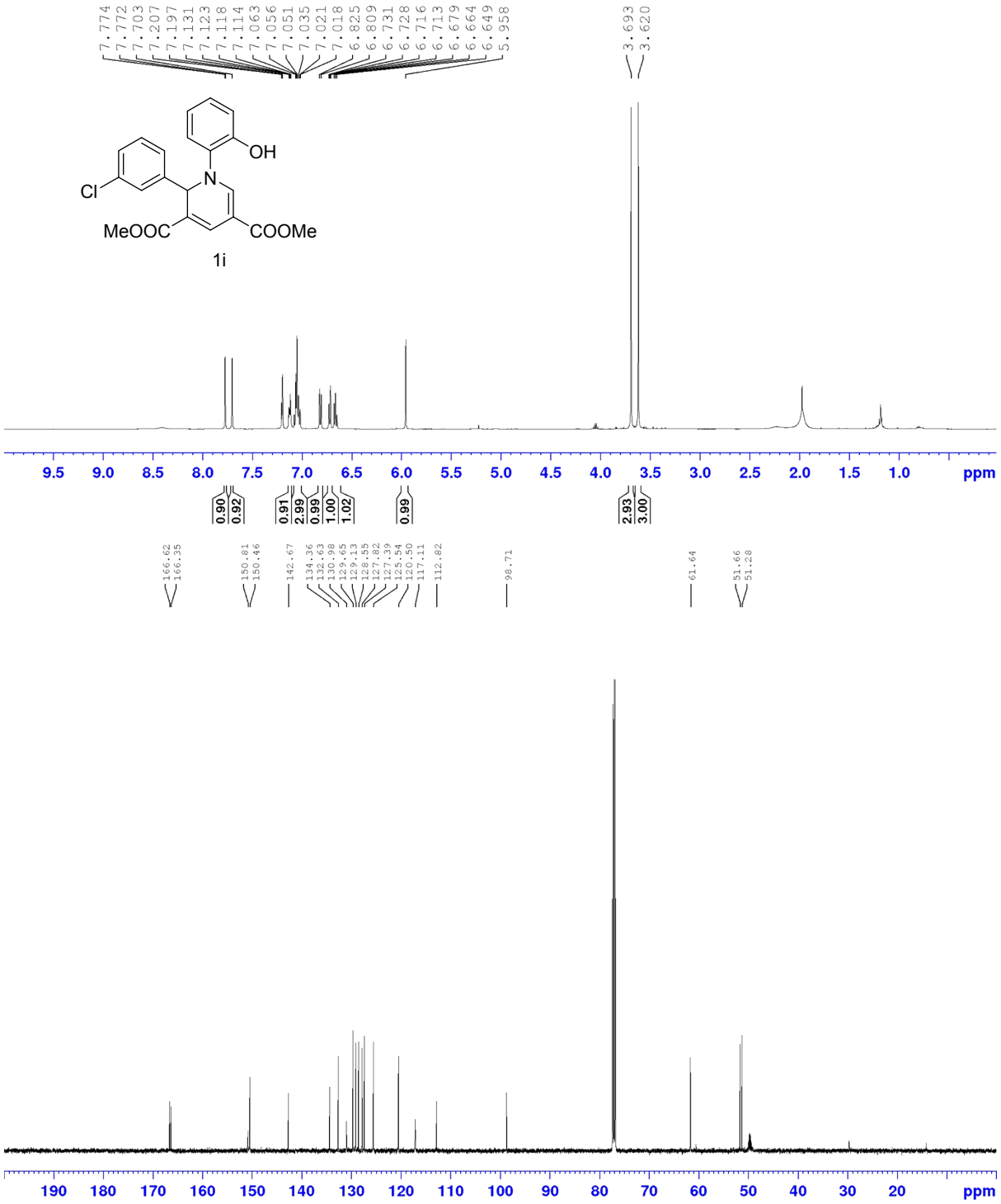

7.786
7.688
7.191
7.116
7.098
7.078
7.052
7.038
7.024
6.821
6.805
6.728
6.713
6.698
5.934
3.707
3.627
Br
OH
N
MeOOC
COOMe
1d
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.93
0.94
4.03
0.93
1.05
2.69
1.01
3.00
3.26
166.68
166.49
150.47
150.38
139.06
134.40
132.59
130.83
129.23
128.75
128.73
127.84
120.97
117.27
113.16
98.91
61.58
51.85
51.46
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

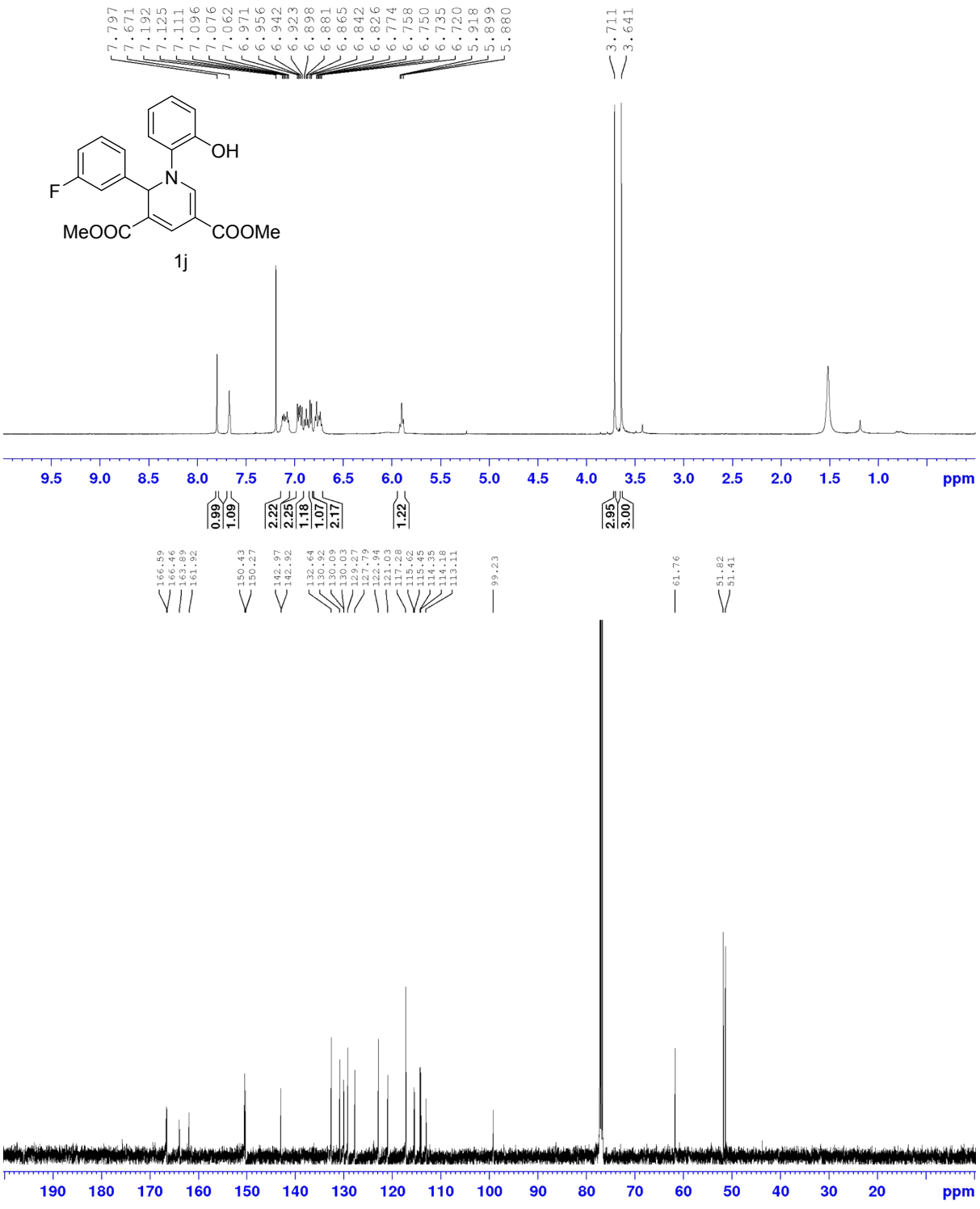

7.783
7.781
7.655
7.284
7.267
7.073
7.056
6.833
6.817
6.771
6.759
6.755
6.751
6.736
5.858
3.706
3.626
Cl
OH
N
MeOOC
COOMe
1e
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.08
1.03
2.26
2.29
1.05
2.24
1.10
3.00
3.04
166.46
166.28
150.38
150.08
139.09
134.48
132.42
130.94
129.32
128.79
128.70
127.96
121.19
117.27
113.39
99.28
61.76
51.76
51.37
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.879
7.810
7.542
7.526
7.422
7.406
7.285
7.177
7.174
7.160
7.147
7.144
6.923
6.907
6.866
6.853
6.850
6.842
6.828
6.812
6.102
3.808
3.729
NC
OH
N
MeOOC
COOMe
1f
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 ppm
0.97 1.00 2.13 2.15 0.94 1.15 1.05 2.30 1.02 3.00 3.05
166.61
166.22
150.55
145.31
132.92
132.38
130.69
129.31
128.00
127.36
121.01
118.51
117.33
112.73
112.18
99.10
61.94
51.85
51.51
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

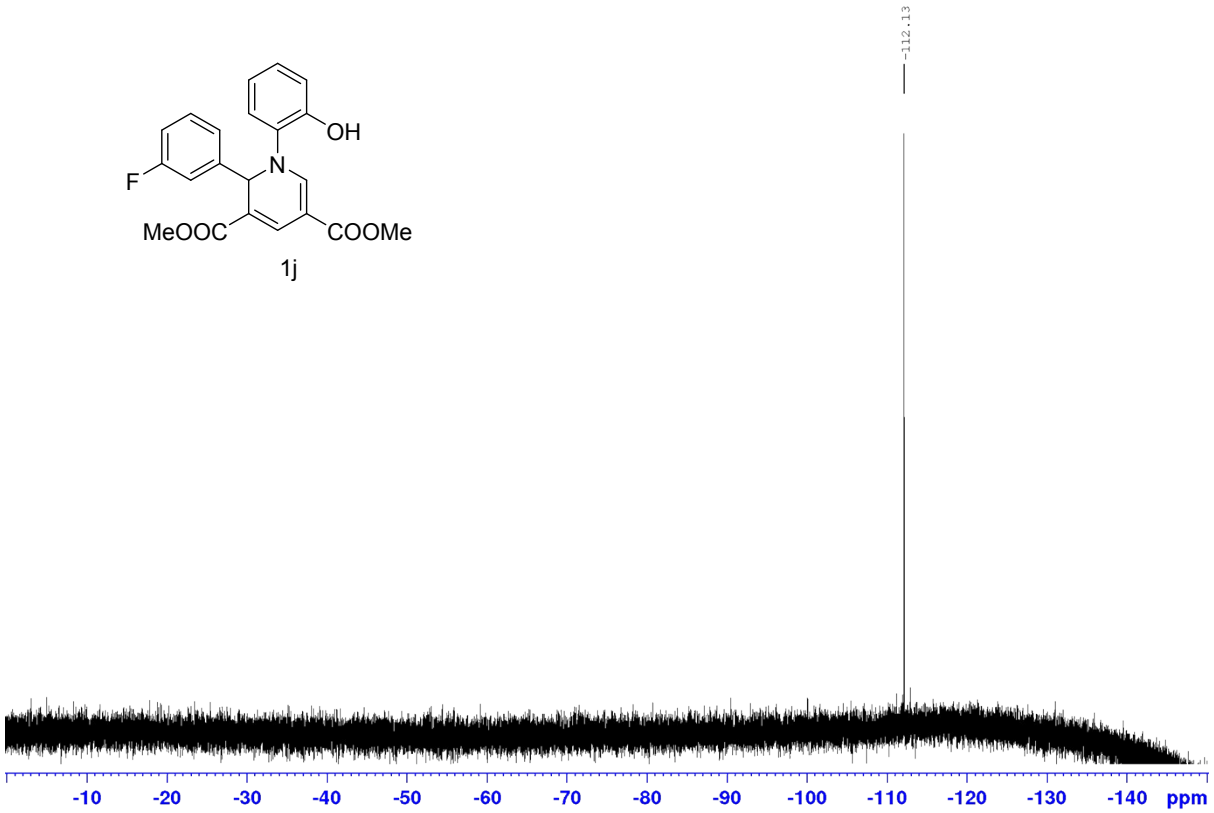

7.881
7.686
7.285
7.210
7.193
7.176
7.163
7.160
6.931
6.915
6.880
6.867
6.865
6.844
6.830
6.814
6.783
6.766
5.846
3.792
3.776
3.708
MeO
OH
N
MeOOC
COOMe
1g
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.04
1.05
1.29
3.39
1.16
2.42
2.18
1.35
3.16
3.27
3.00
166.34
166.21
159.91
150.66
149.58
133.18
131.87
131.34
129.41
128.58
128.37
121.25
117.29
114.06
114.02
99.43
62.18
55.18
51.61
51.23
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.786
7.784
7.633
7.190
7.187
7.170
7.130
7.114
7.094
7.091
7.077
7.063
7.060
6.839
6.824
6.811
6.808
6.756
6.741
5.792
3.697
3.632
1.192
t-Bu
OH
N
MeOOC
COOMe
1h
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 ppm
1.03
1.04
3.30
2.20
1.31
2.29
1.14
1.31
3.16
3.00
9.48
166.38
166.29
151.74
150.67
149.65
137.62
131.99
131.57
129.33
128.19
126.70
125.70
121.21
117.35
113.72
99.91
62.27
51.65
51.25
34.57
31.26
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

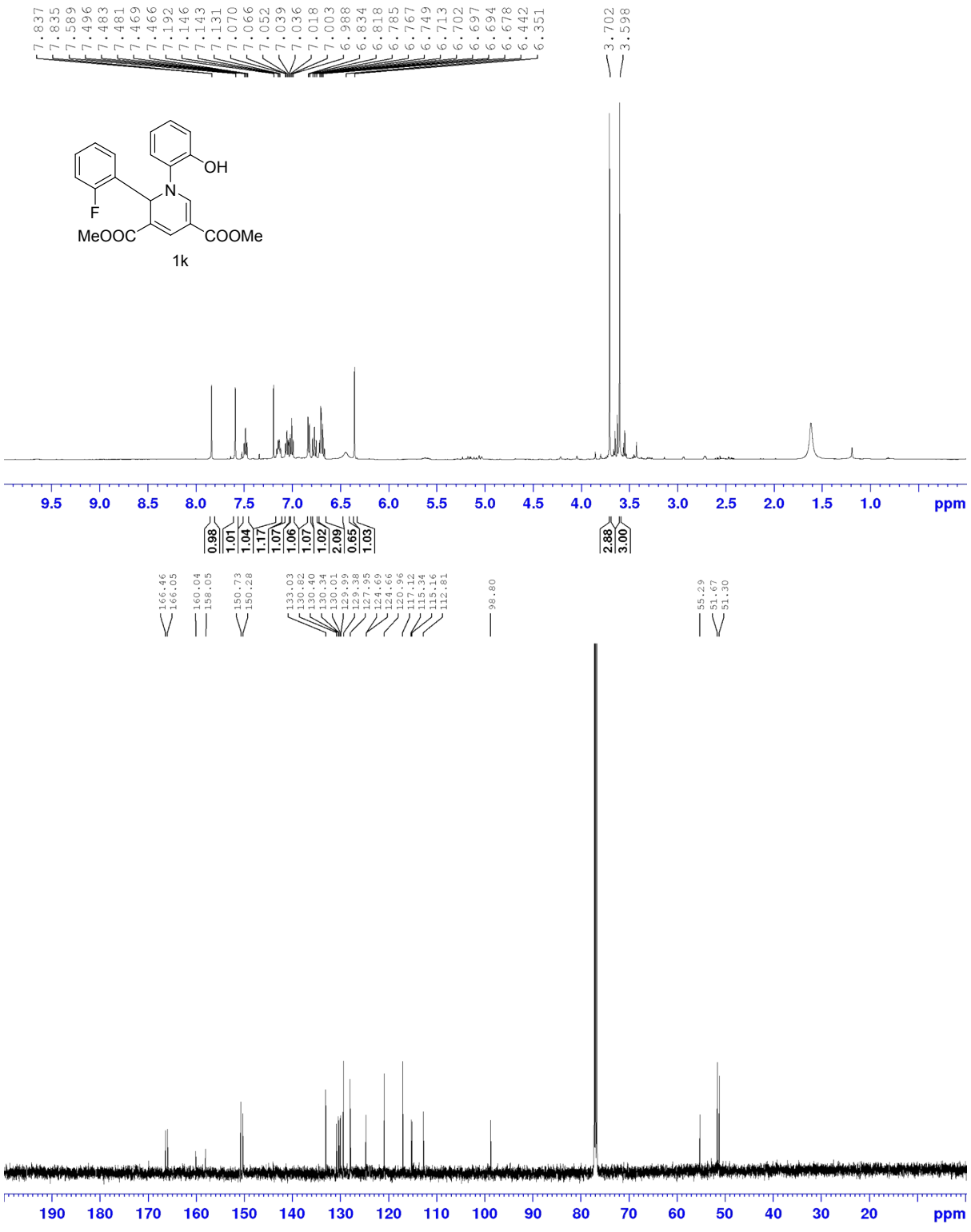

7.774
7.772
7.703
7.207
7.197
7.131
7.123
7.118
7.114
7.063
7.056
7.051
7.035
7.021
7.018
6.825
6.809
6.731
6.728
6.716
6.713
6.679
6.664
6.649
5.958
3.693
3.620
OH
N
Cl
MeOOC
COOMe
1i
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.90 0.92 0.91 2.99 0.99 1.00 1.02 0.99 2.93 3.00
166.62
166.35
150.81
150.46
142.67
134.36
132.63
130.98
129.65
129.13
128.85
127.82
127.39
125.54
120.50
117.11
112.82
98.71
61.64
51.66
51.28
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.797
7.671
7.192
7.125
7.111
7.096
7.076
7.062
6.971
6.956
6.942
6.923
6.898
6.881
6.865
6.842
6.826
6.774
6.758
6.750
6.735
6.720
5.918
5.899
5.880
3.711
3.641
OH
N
F
MeOOC
COOMe
1j
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 ppm
0.99 1.09 2.22 2.25 1.18 1.07 2.17 1.22 2.95 3.00
166.59
166.46
163.89
161.92
150.43
150.27
142.97
142.92
132.64
130.92
130.09
130.03
129.27
127.79
122.94
121.03
117.28
115.62
115.45
114.35
114.18
113.11
99.23
61.76
51.82
51.41
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

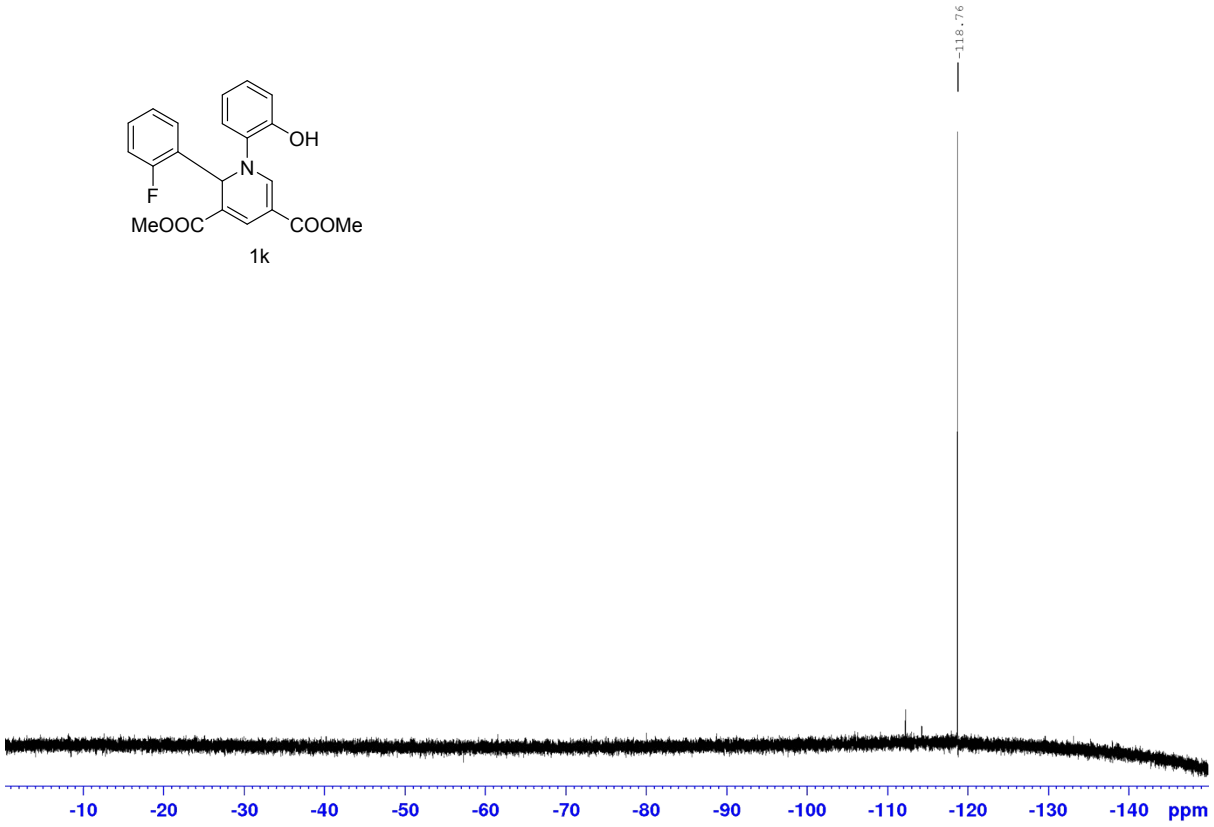
JR-3J-F
F19CPD CDCl3 {E:\jaice} niist 22

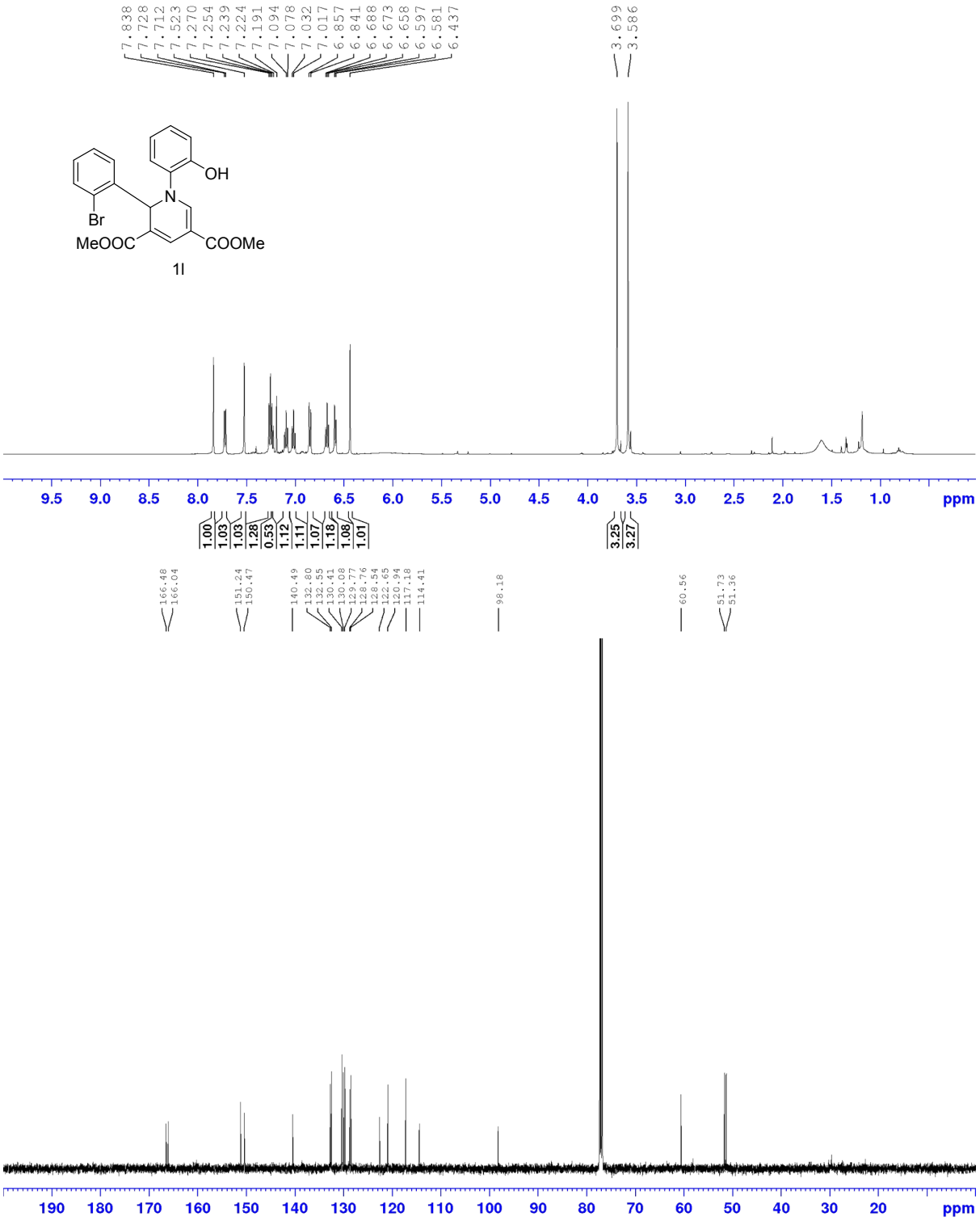


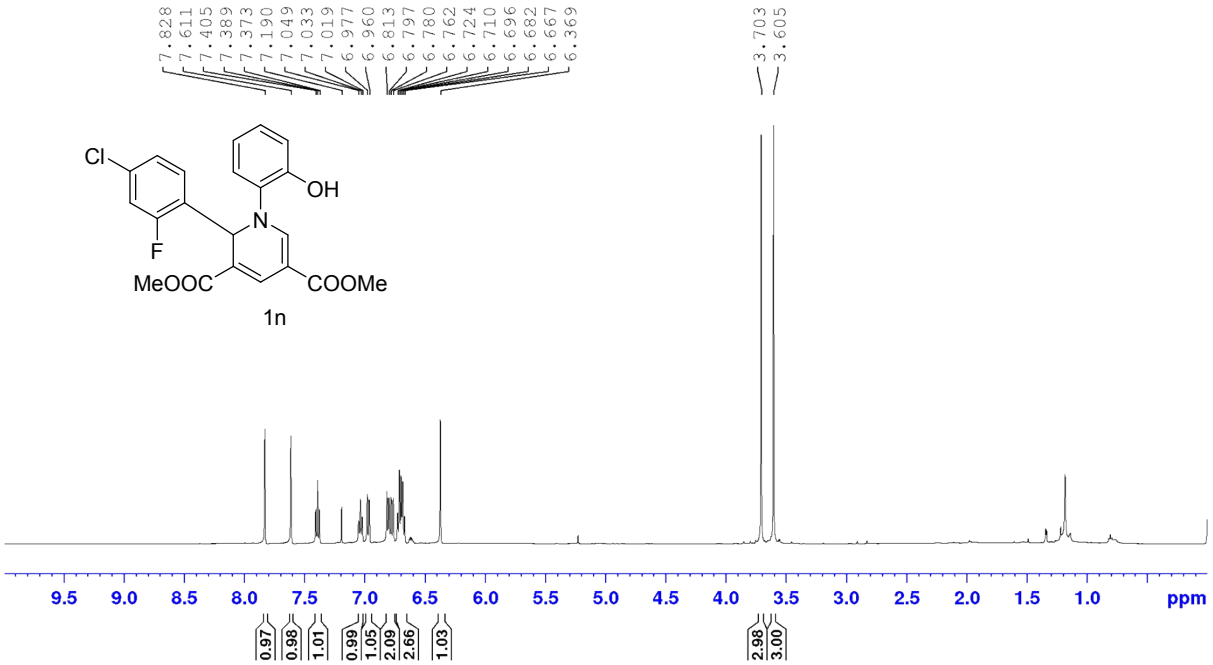
1j

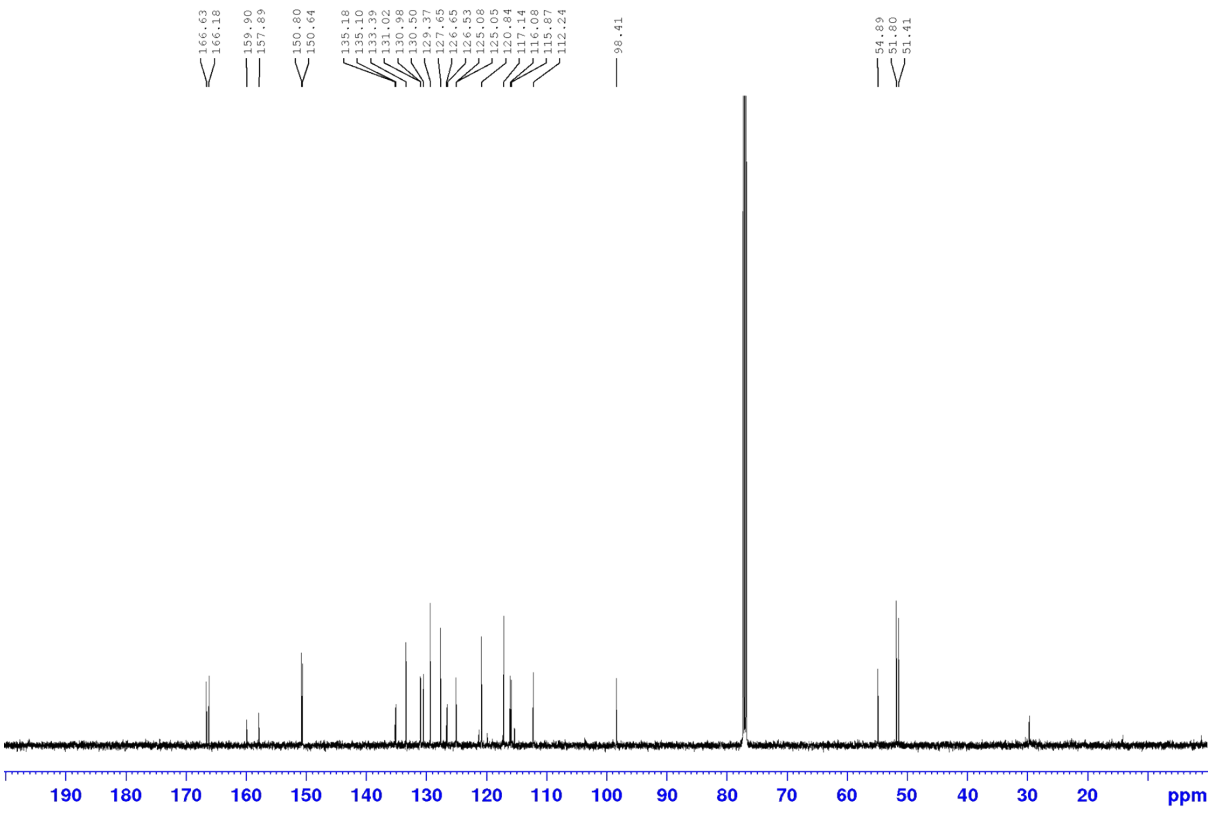

7.837
7.835
7.589
7.496
7.483
7.481
7.469
7.466
7.192
7.146
7.143
7.131
7.070
7.066
7.052
7.039
7.036
7.018
7.003
6.988
6.834
6.818
6.785
6.767
6.749
6.713
6.702
6.697
6.694
6.678
6.442
6.351
3.702
3.598
OH
N
F
MeOOC
COOMe
1k
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.98 1.01 1.04 1.17 1.07 1.06 1.07 1.02 2.09 0.65 1.03
2.88 3.00
166.46
166.05
160.04
158.05
150.73
150.28
133.03
130.82
130.40
130.34
130.01
129.99
129.38
127.95
124.69
124.66
120.96
117.12
115.34
115.16
112.81
98.80
55.29
51.67
51.30
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

JR-3K
F19CPD CDCl3 {E:\jaice} niist 38

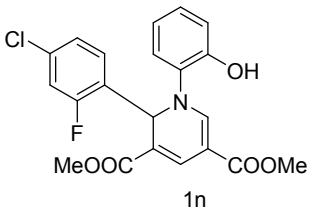



7.838
7.728
7.712
7.523
7.270
7.254
7.239
7.224
7.191
7.094
7.078
7.032
7.017
6.857
6.841
6.688
6.673
6.658
6.597
6.581
6.437
3.699
3.586
OH
N
Br
MeOOC
COOMe
1l
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00 1.03 1.03 1.28 0.53 1.12 1.11 1.07 1.18 1.08 1.01
3.25 3.27
166.48
166.04
151.24
150.47
140.49
132.80
132.55
130.41
130.08
129.77
128.76
128.54
122.65
120.94
117.18
114.41
98.18
60.56
51.73
51.36
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

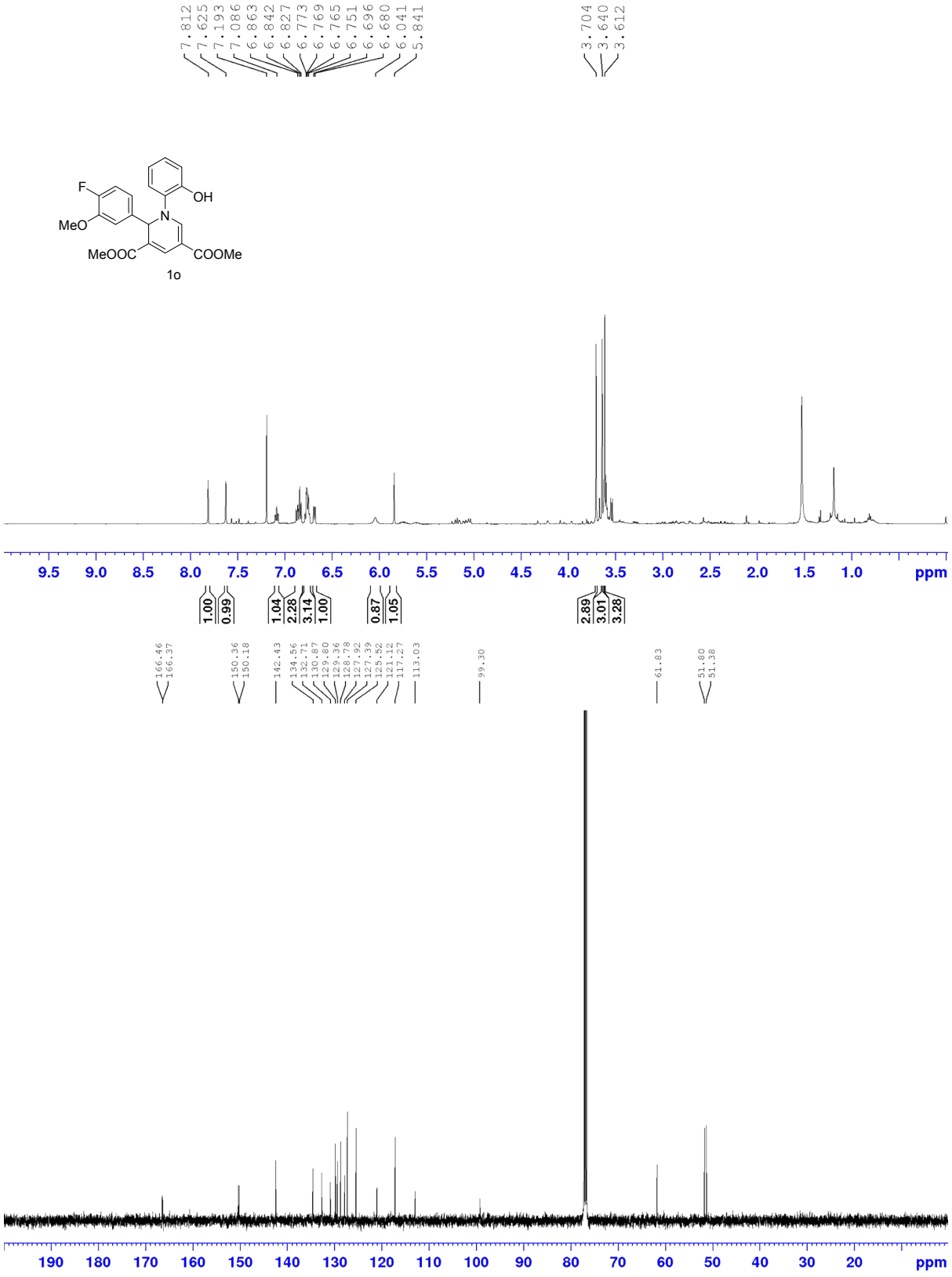

7.828
7.611
7.405
7.389
7.373
7.190
7.049
7.033
7.019
6.977
6.960
6.813
6.797
6.780
6.762
6.724
6.710
6.696
6.682
6.667
6.369
3.703
3.605
Cl
OH
N
F
MeOOC
COOMe
1n
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.97
0.98
1.01
0.99
1.05
2.09
2.66
1.03
2.98
3.00


166.63
166.18
159.90
157.89
150.80
150.64
135.18
135.10
133.39
131.02
130.98
130.50
129.37
127.65
126.65
126.53
125.08
125.05
120.84
117.14
116.08
115.87
112.24
98.41
54.89
51.80
51.41
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

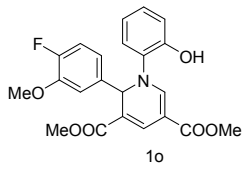
JR-3n
F19CPD CDCl3 {E:\jaice} niist 34



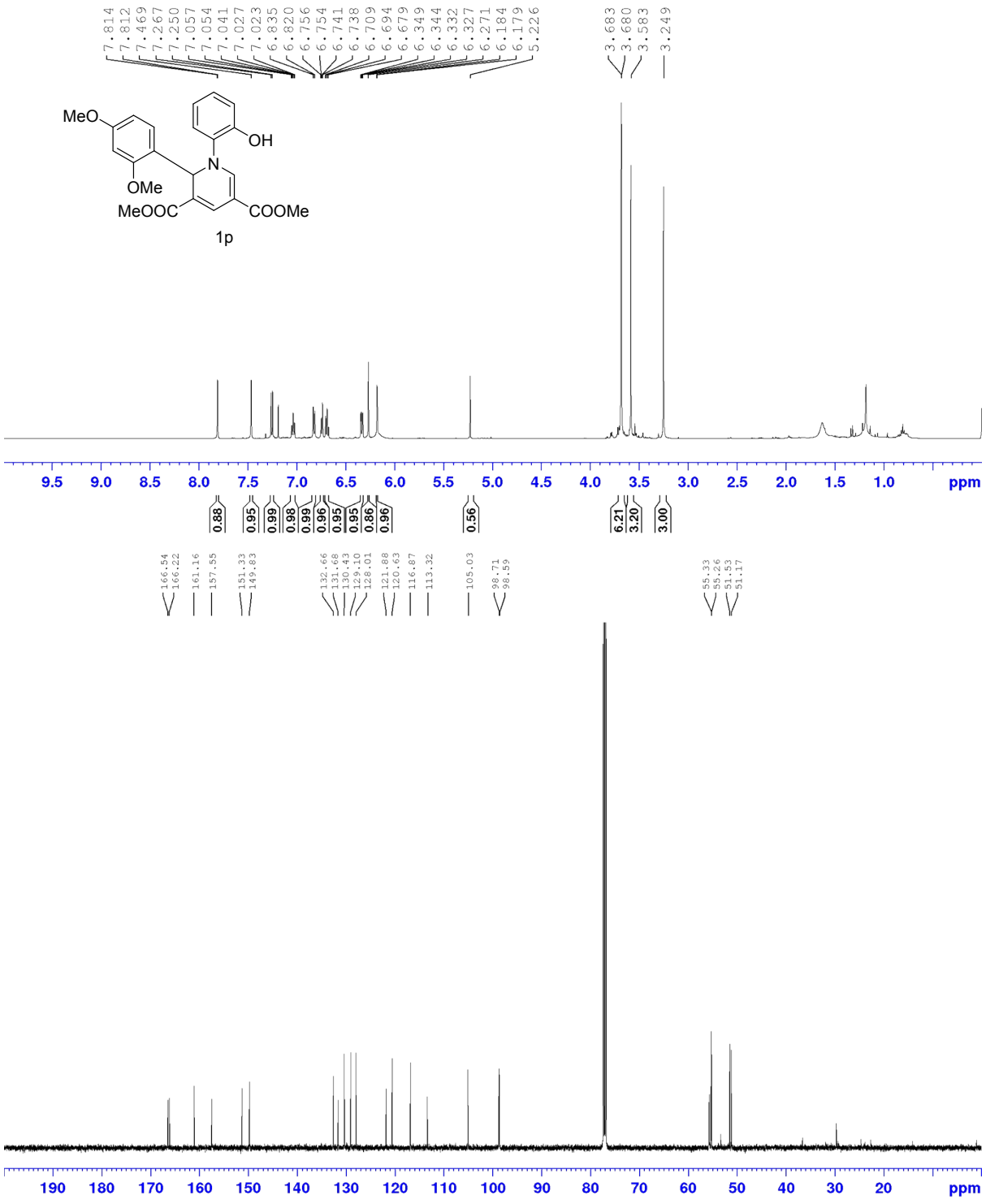
1n

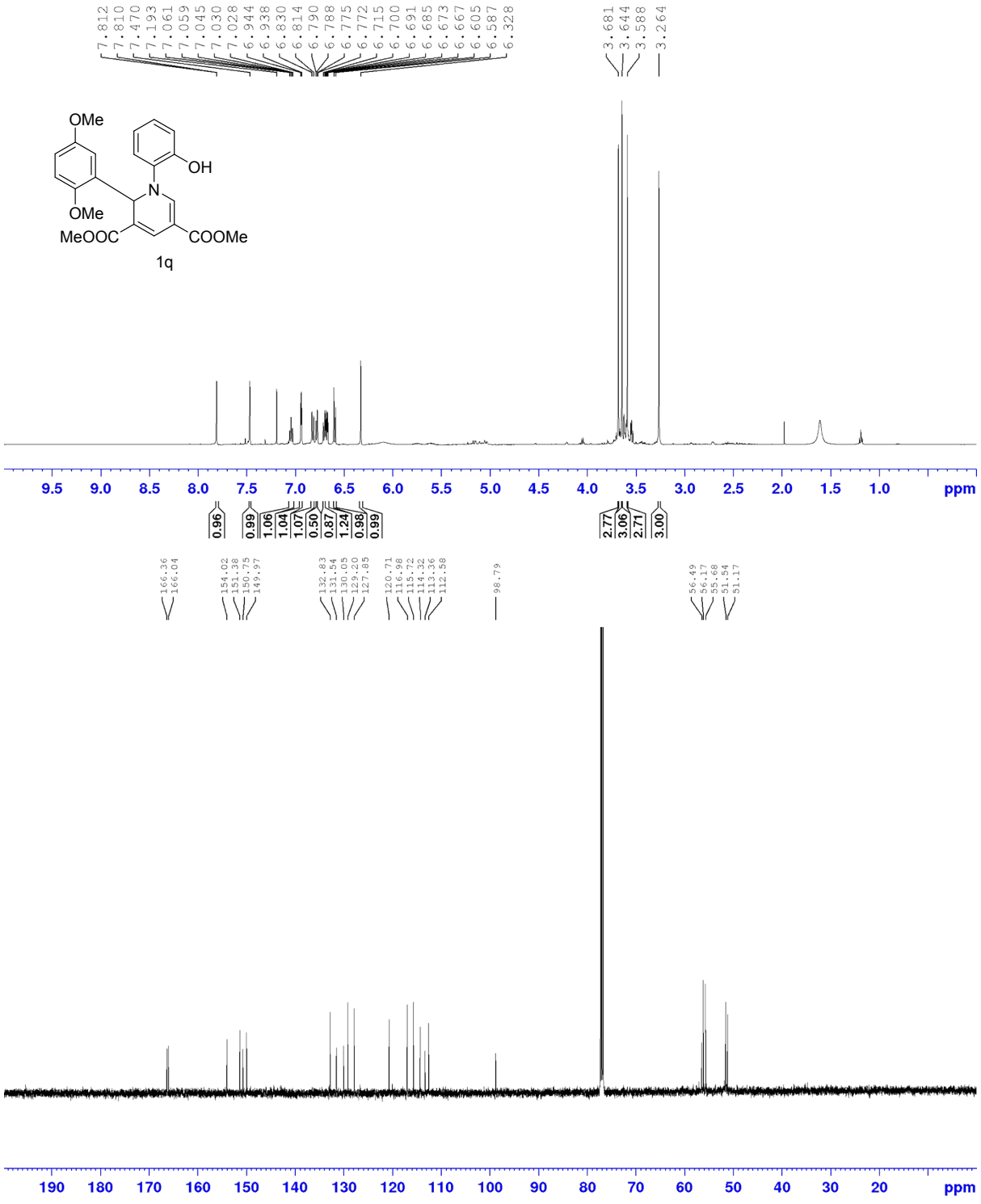

7.812
7.625
7.193
7.086
6.863
6.842
6.827
6.773
6.769
6.765
6.751
6.696
6.680
6.041
5.841
3.704
3.640
3.612
F
OH
N
MeO
MeOOC
COOMe
1o
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00 0.99 1.04 2.28 3.14 1.00 0.87 1.05 2.89 3.01 3.28
166.46
166.37
150.36
150.18
142.43
134.56
132.71
130.87
129.80
128.66
128.78
127.92
127.39
125.52
121.12
117.27
113.03
99.30
61.83
51.80
51.38
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

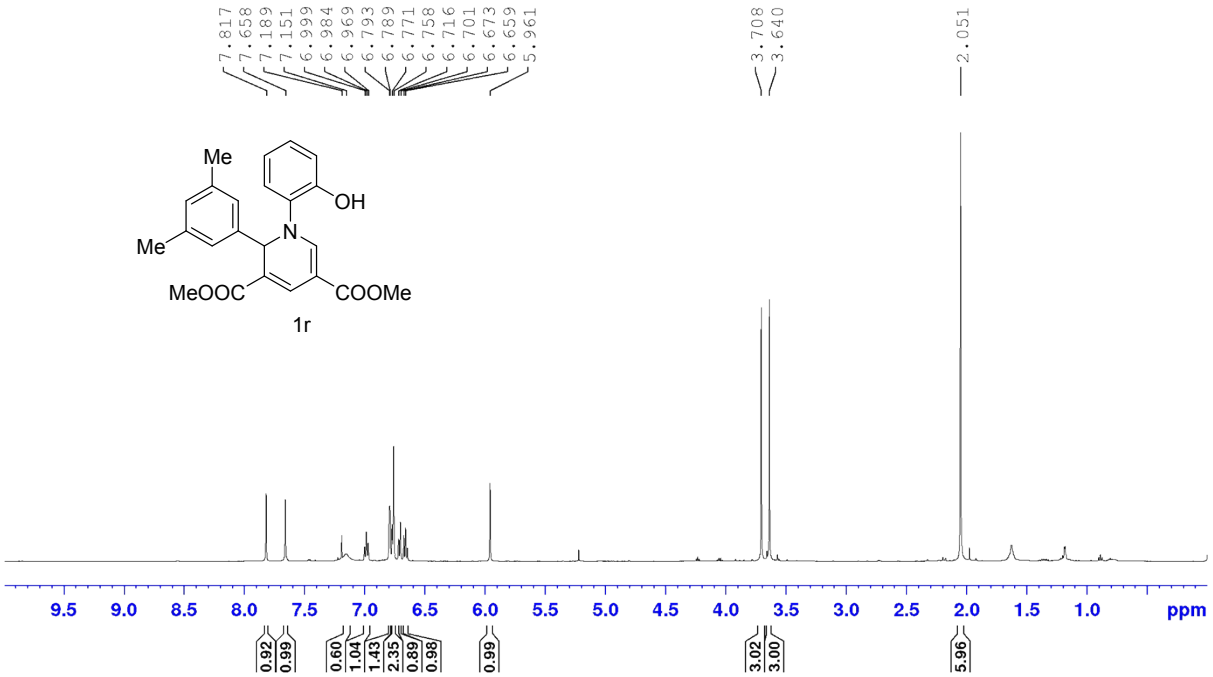
JR-30
F19CPD CDCl3 {E:\jaice} niist 24



1o


7.814
7.812
7.469
7.267
7.250
7.057
7.054
7.041
7.027
7.023
6.835
6.820
6.756
6.754
6.741
6.738
6.709
6.694
6.679
6.349
6.344
6.332
6.327
6.271
6.184
6.179
5.226
3.683
3.680
3.583
3.249
MeO
OH
N
OMe
MeOOC
COOMe
1p
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 ppm
0.88
0.95
0.99
0.98
0.99
0.96
0.95
0.95
0.86
0.96
0.56
6.21
3.20
3.00
166.54
166.22
161.16
157.55
151.33
149.83
132.66
131.68
130.43
129.10
128.01
121.88
120.63
116.87
113.32
105.03
98.71
98.59
55.33
55.26
51.53
51.17
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.812
7.810
7.470
7.193
7.061
7.059
7.045
7.030
7.028
6.944
6.938
6.830
6.814
6.790
6.788
6.775
6.772
6.715
6.700
6.691
6.685
6.673
6.667
6.605
6.587
6.328
3.681
3.644
3.588
3.264
OMe
OH
N
OMe
MeOOC
COOMe
1q
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 ppm
0.96
0.99
1.06
1.04
1.07
0.50
0.87
1.24
0.98
0.99
2.77
3.06
2.71
3.00
166.36
166.04
154.02
151.38
150.75
149.97
132.83
131.54
130.05
129.20
127.85
120.71
116.98
115.72
114.32
113.36
112.58
98.79
56.49
56.17
55.68
51.54
51.17
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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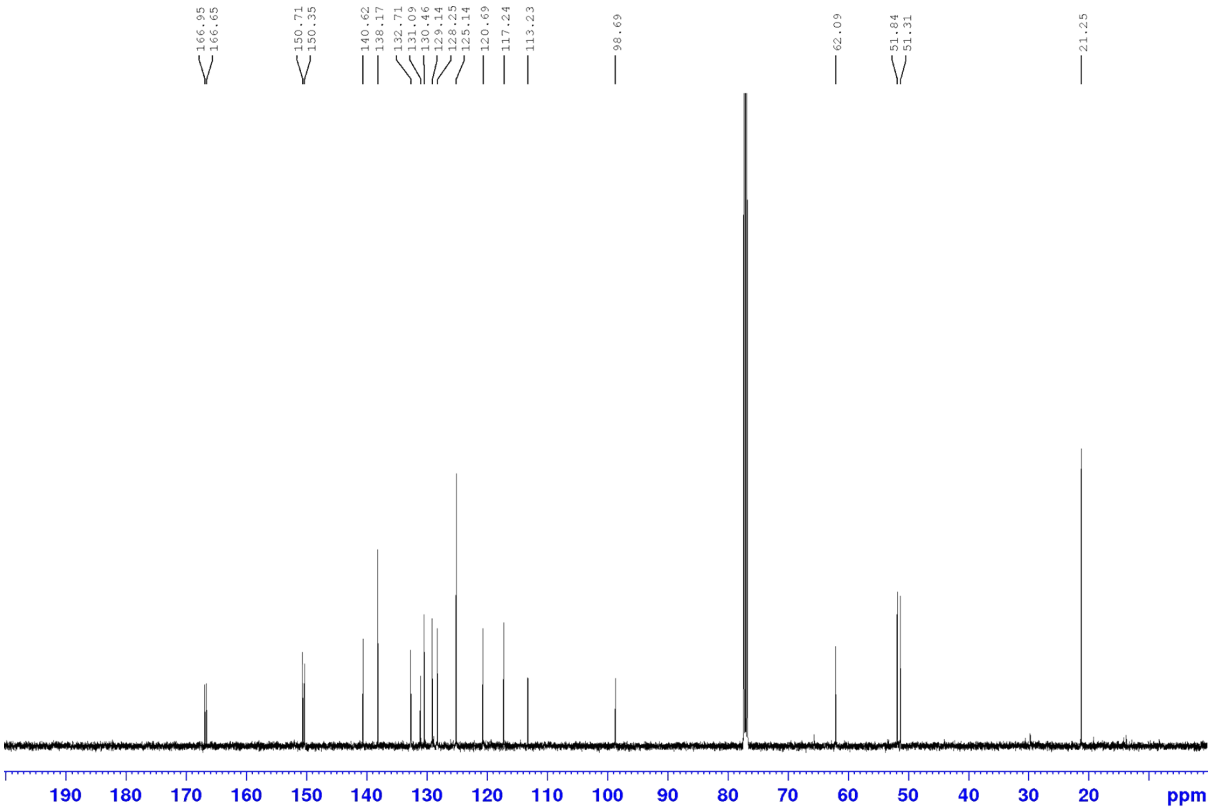
7.658
7.189
7.151
6.999
6.984
6.969
6.793
6.789
6.771
6.758
6.716
6.701
6.673
6.659
5.961
3.708
3.640
2.051
Me
OH
N
Me
MeOOC
COOMe
1r
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.92
0.99
0.60
1.04
1.43
2.35
0.89
0.98
0.99
3.02
3.00
5.96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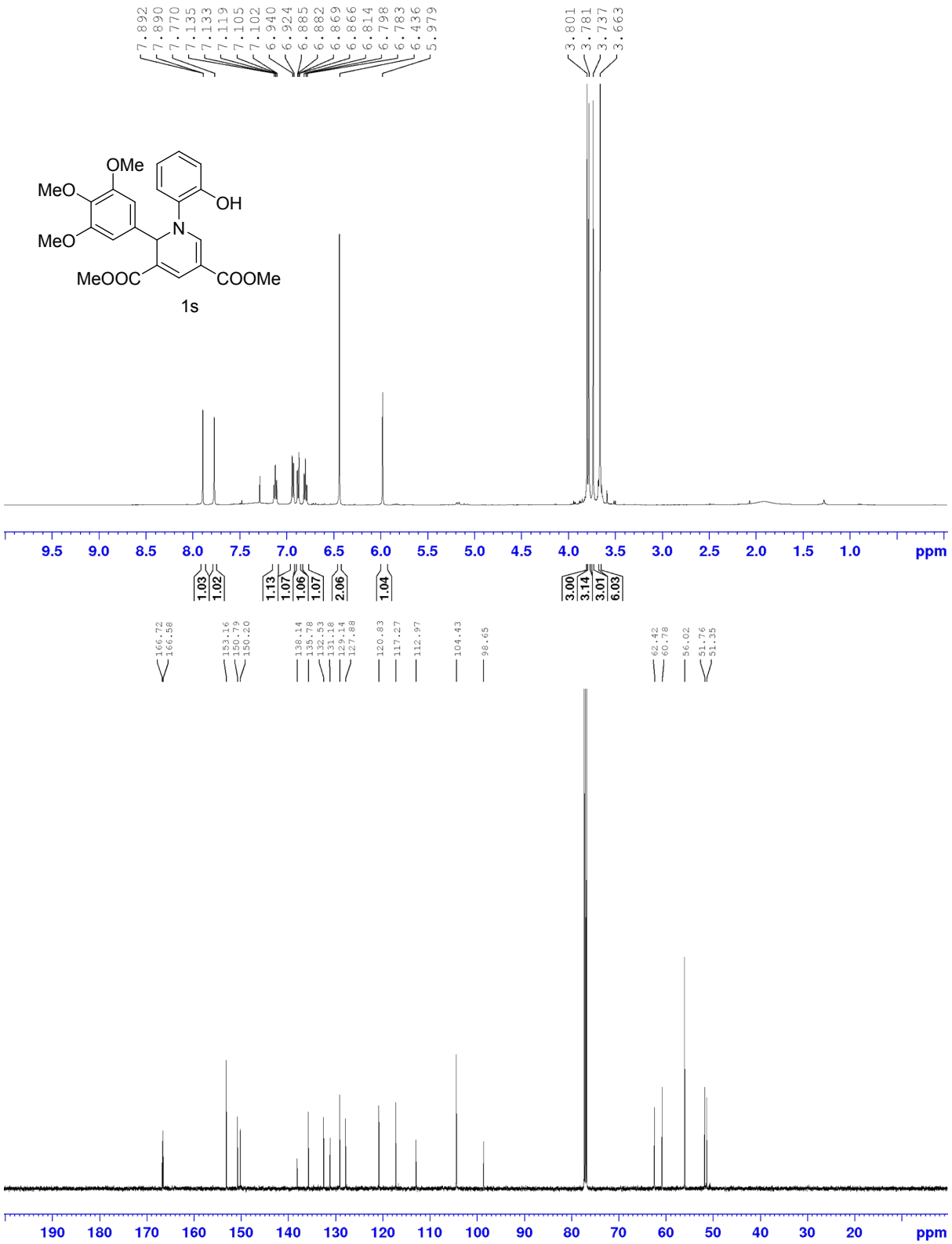
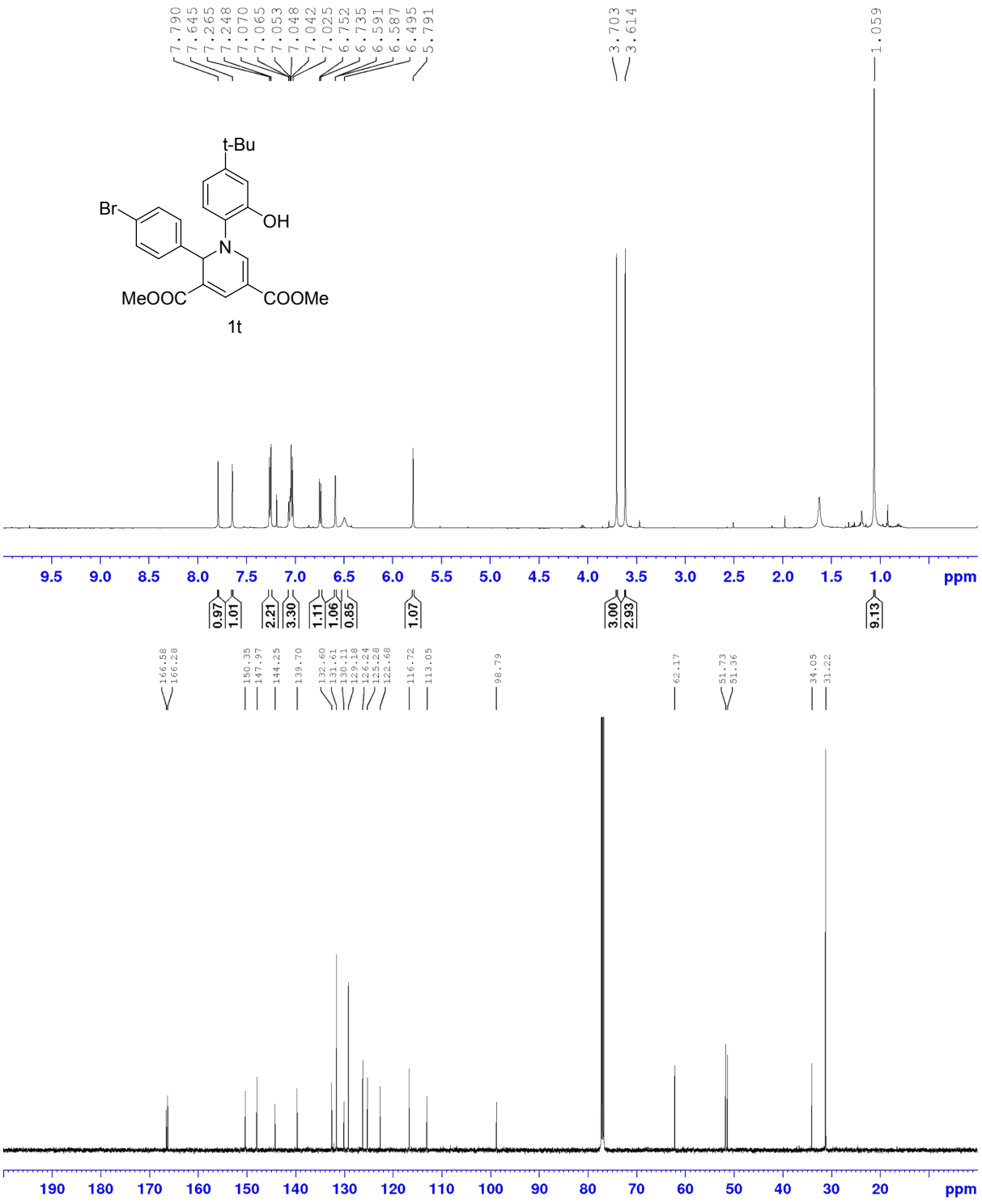
166.65
150.71
150.35
140.62
138.17
132.71
131.09
130.46
129.14
128.25
125.14
120.69
117.24
113.23
98.69
62.09
51.84
51.31
21.25
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.892
7.890
7.770
7.135
7.133
7.119
7.105
7.102
6.940
6.924
6.885
6.882
6.869
6.866
6.814
6.798
6.783
6.436
5.979
3.801
3.781
3.737
3.663
OMe
MeO
MeO
OH
N
MeOOC
COOMe
1s
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 ppm
1.03
1.02
1.13
1.07
1.06
1.07
2.06
1.04
3.00
3.14
3.01
6.03
166.72
166.58
153.16
150.79
150.20
138.14
135.78
132.53
131.18
129.14
127.88
120.83
117.27
112.97
104.43
98.65
62.42
60.78
56.02
51.76
51.35
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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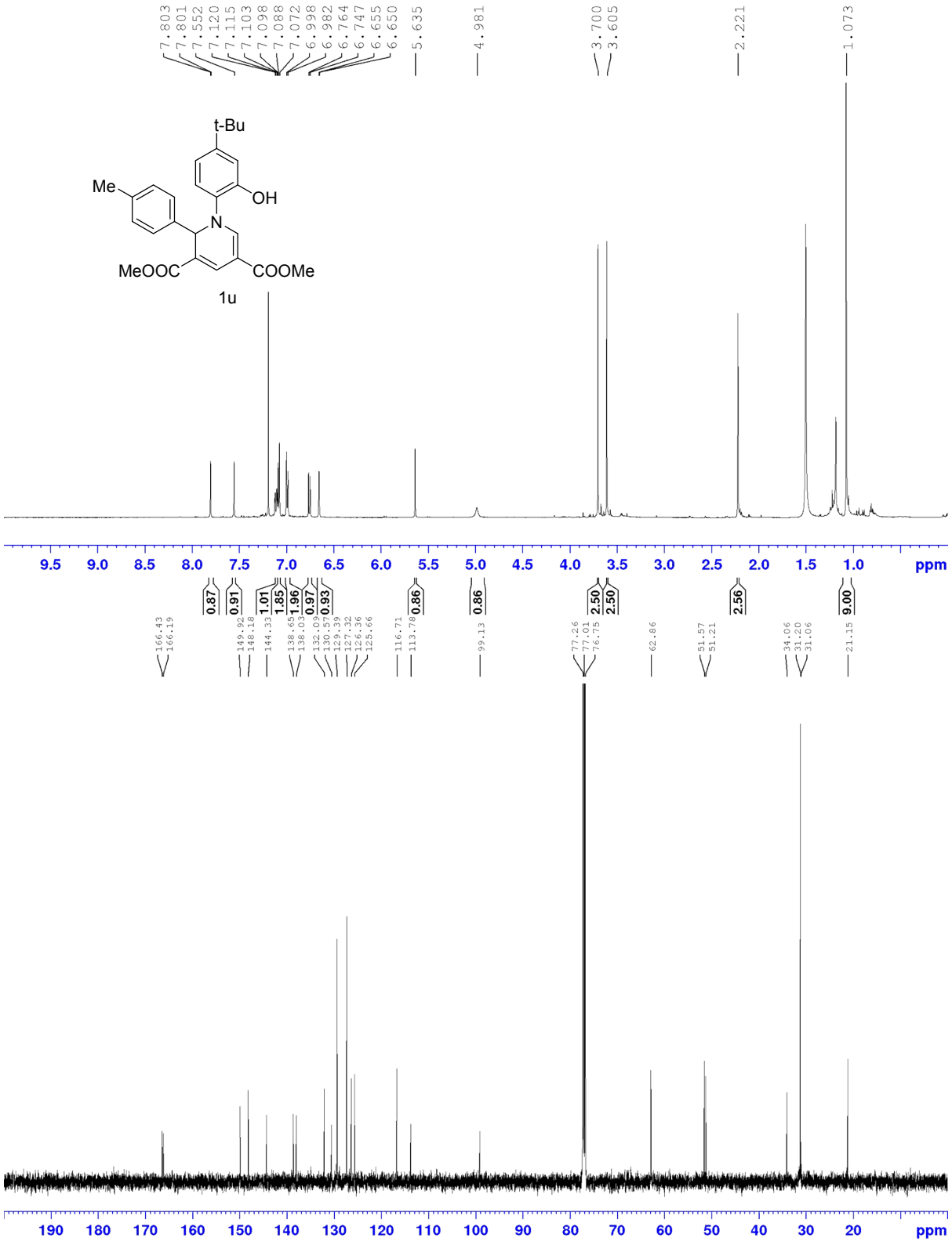
7.645
7.265
7.248
7.070
7.065
7.053
7.048
7.042
7.025
6.752
6.735
6.591
6.587
6.495
5.791
3.703
3.614
1.059
t-Bu
Br
OH
N
MeOOC
COOMe
1t
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.97
1.01
2.21
3.30
1.11
1.06
0.85
1.07
3.00
2.93
9.13
166.58
166.28
150.35
147.97
144.25
139.70
132.60
131.61
130.11
129.18
126.24
125.28
122.68
116.72
113.05
98.79
62.17
51.73
51.36
34.05
31.22
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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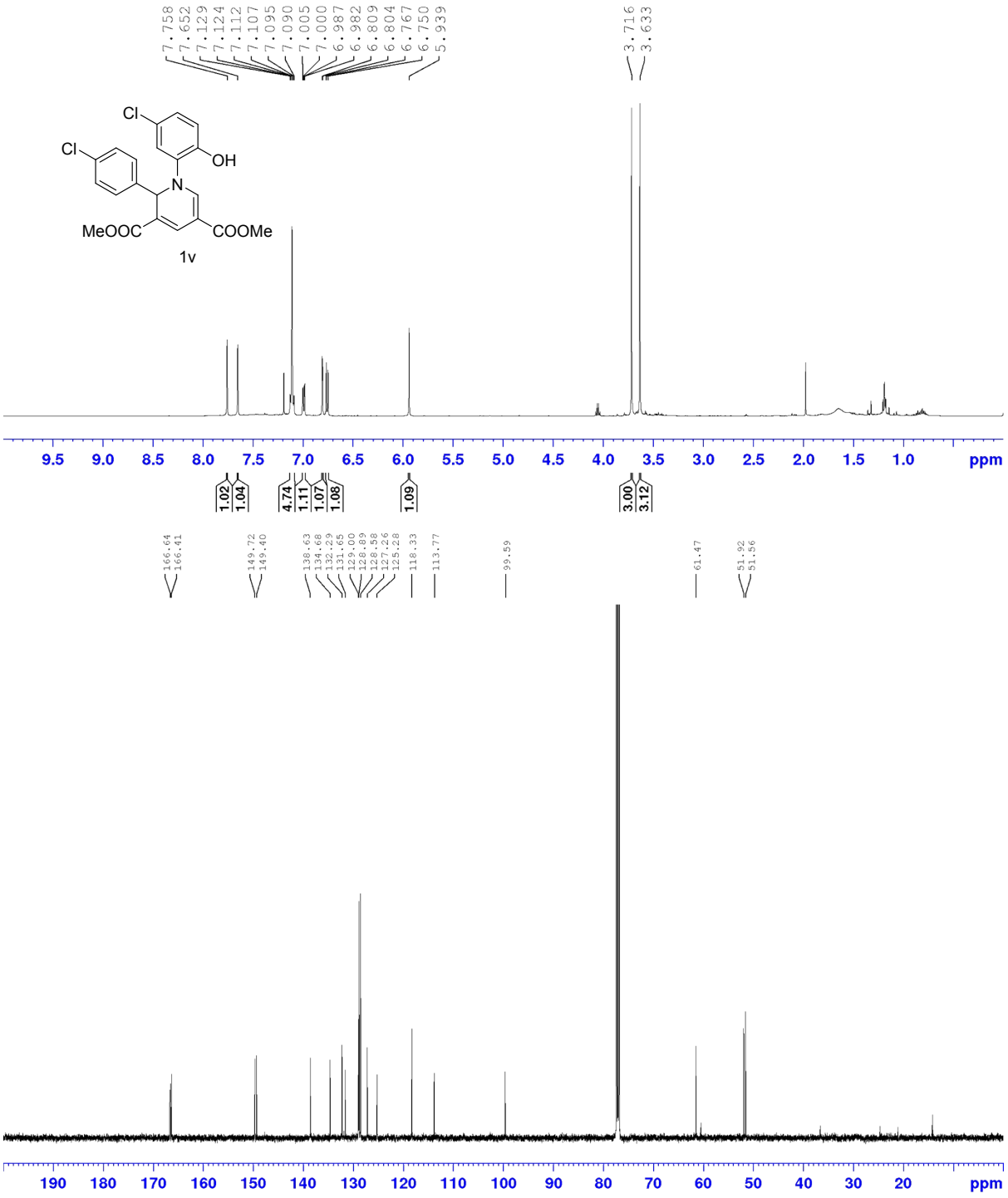
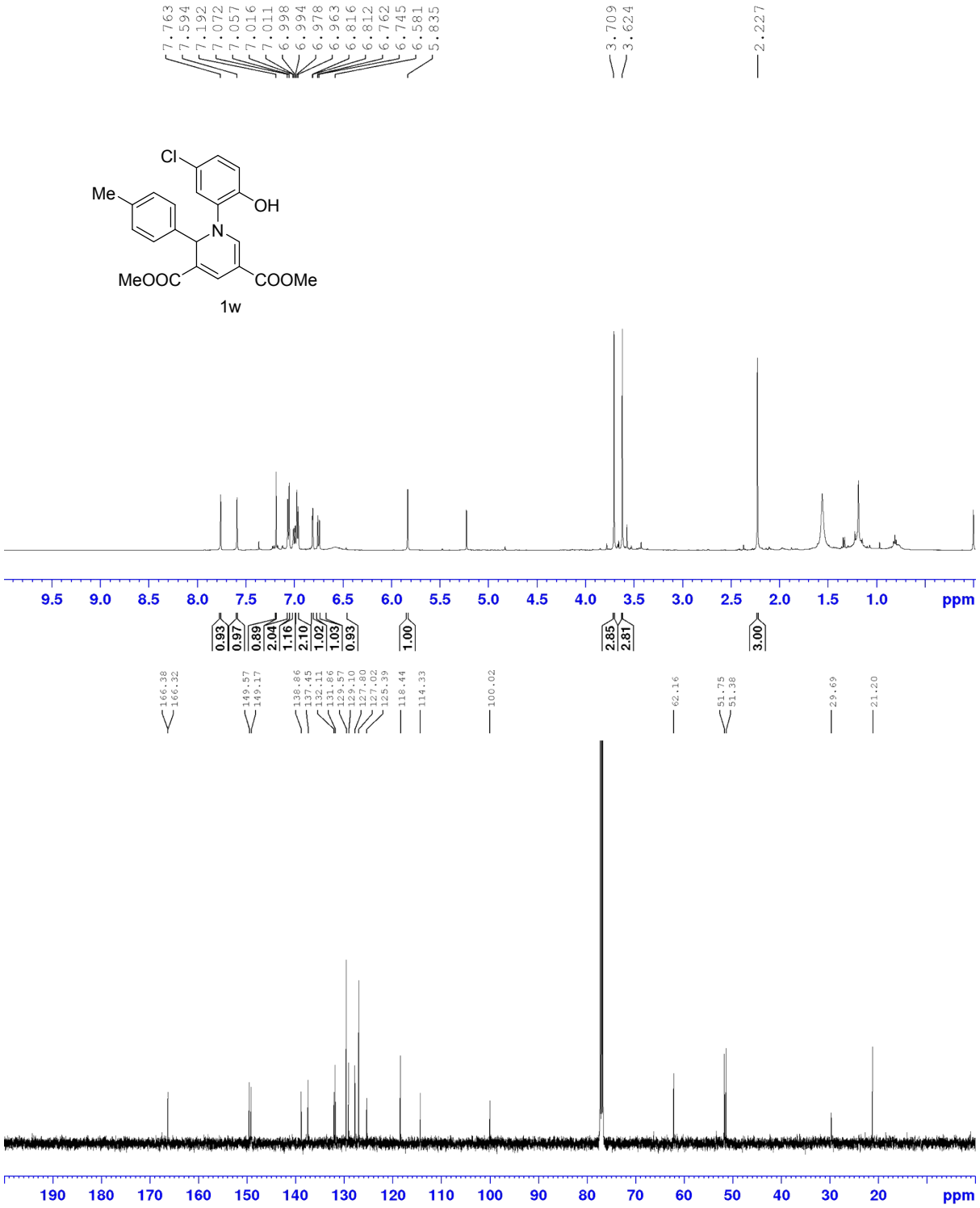
7.801
7.552
7.120
7.115
7.103
7.098
7.088
7.072
6.998
6.982
6.764
6.747
6.655
6.650
5.635
4.981
3.700
3.605
2.221
1.073
t-Bu
Me
OH
N
MeOOC
COOMe
1u
ppm
0.87
0.91
1.01
1.85
1.96
0.97
0.93
0.86
0.86
2.50
2.50
2.56
9.00
166.43
166.19
149.92
148.18
144.33
138.65
138.03
132.09
130.57
129.39
127.32
126.36
125.66
116.71
113.78
99.13
77.26
77.01
76.75
62.86
51.57
51.21
34.06
31.20
31.06
21.15
ppm


7.758
7.652
7.129
7.124
7.112
7.107
7.095
7.090
7.005
7.000
6.987
6.982
6.809
6.804
6.767
6.750
5.939
3.716
3.633
Cl
Cl
OH
N
MeOOC
COOMe
1v
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 ppm
1.02
1.04
4.74
1.11
1.07
1.08
1.09
3.00
3.12
166.64
166.41
149.72
149.40
138.63
134.68
132.29
131.65
129.00
128.89
128.58
127.26
125.28
118.33
113.77
99.59
61.47
51.92
51.56
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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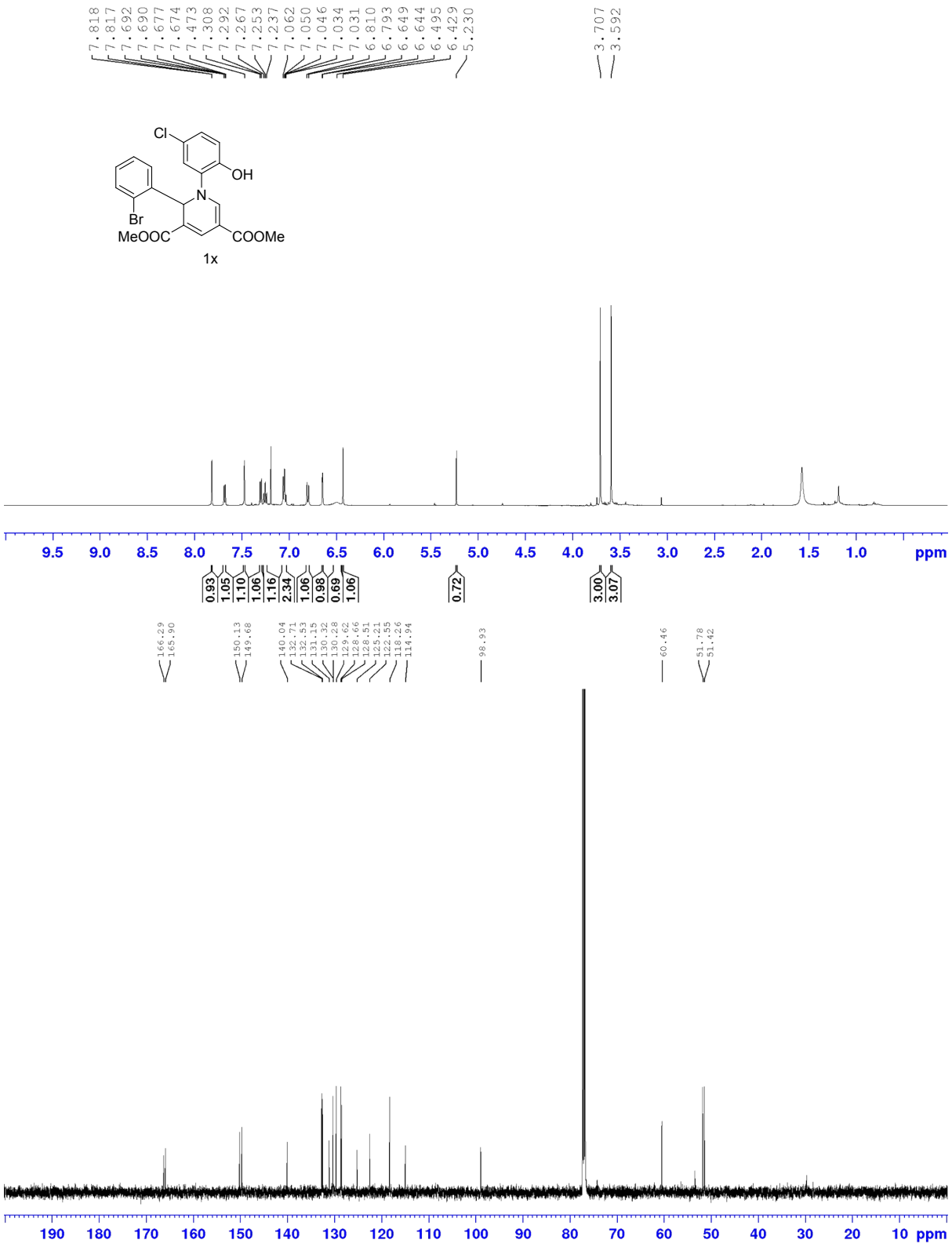
7.594
7.192
7.072
7.057
7.016
7.011
6.998
6.994
6.978
6.963
6.816
6.812
6.762
6.745
6.581
5.835
3.709
3.624
2.227
Cl
OH
Me
N
MeOOC
COOMe
1w
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.93 0.97 0.89 2.04 1.16 2.10 1.02 1.03 0.93 1.00 2.85 2.81 3.00
166.38
166.32
149.57
149.17
138.86
137.45
132.11
131.86
129.57
129.10
127.80
127.02
125.39
118.44
114.33
100.02
62.16
51.75
51.38
29.69
21.20
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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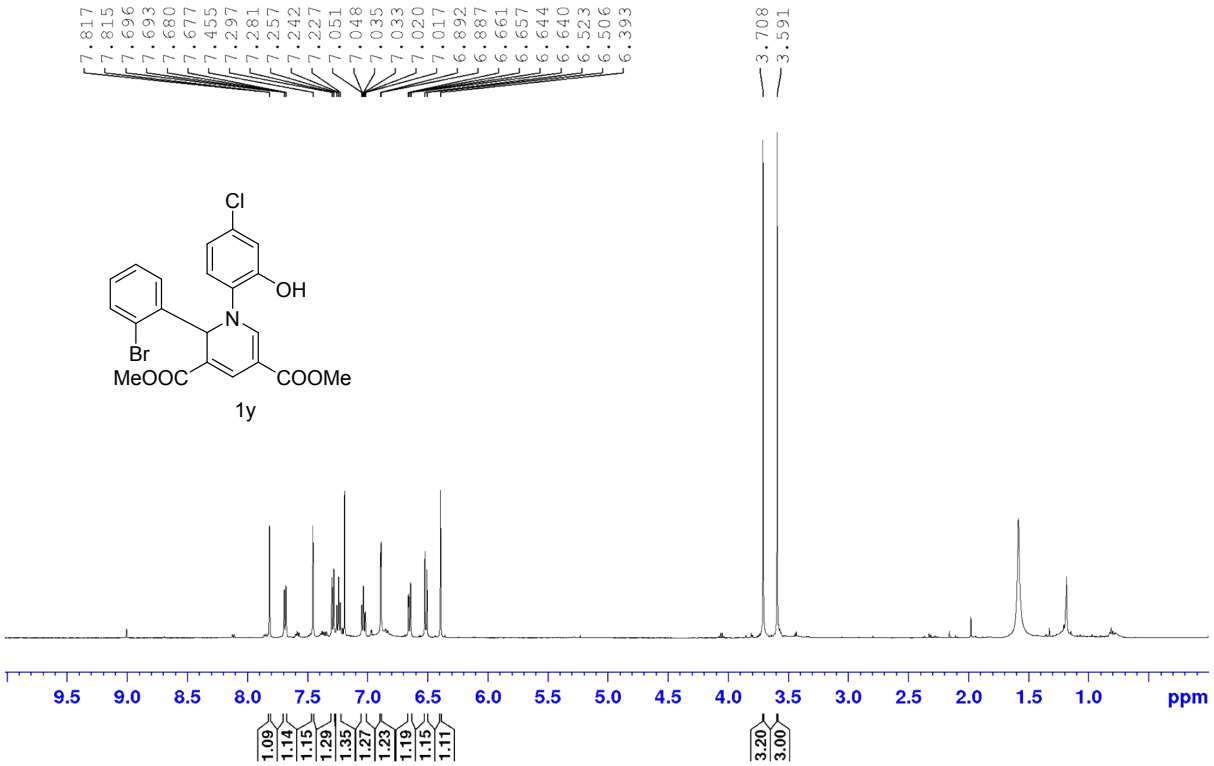
7.692
7.690
7.677
7.674
7.473
7.308
7.292
7.267
7.253
7.237
7.062
7.050
7.046
7.034
7.031
6.810
6.793
6.649
6.644
6.495
6.429
5.230
3.707
3.592
Cl
OH
N
Br
MeOOC
COOMe
1x
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.93 1.05 1.10 1.06 1.16 2.34 1.06 0.98 0.69 1.06
0.72
3.00 3.07
166.29
165.90
150.13
149.68
140.04
132.71
132.53
131.15
130.32
130.28
129.62
128.66
128.51
125.21
122.55
118.26
114.94
98.93
60.46
51.78
51.42
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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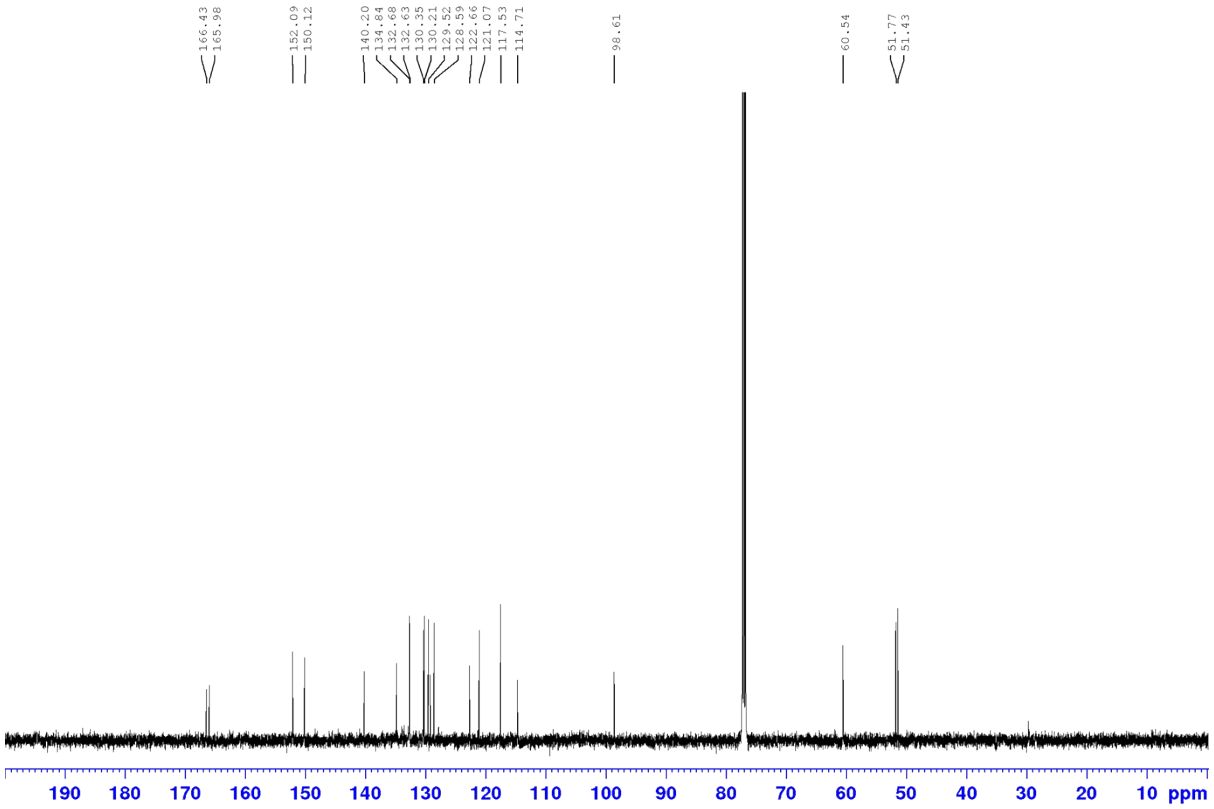
7.815
7.696
7.693
7.680
7.677
7.455
7.297
7.281
7.257
7.242
7.227
7.051
7.048
7.035
7.033
7.020
7.017
6.892
6.887
6.661
6.657
6.644
6.640
6.523
6.506
6.393
3.708
3.591
Cl
OH
N
Br
MeOOC
COOMe
1y
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.09 1.14 1.15 1.29 1.35 1.27 1.23 1.19 1.15 1.11
3.20 3.00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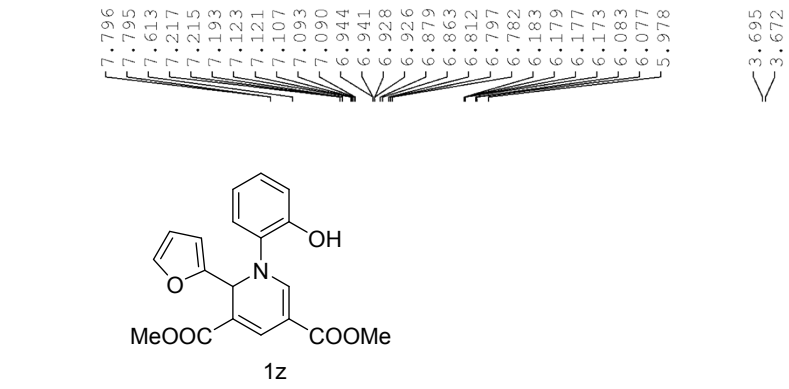
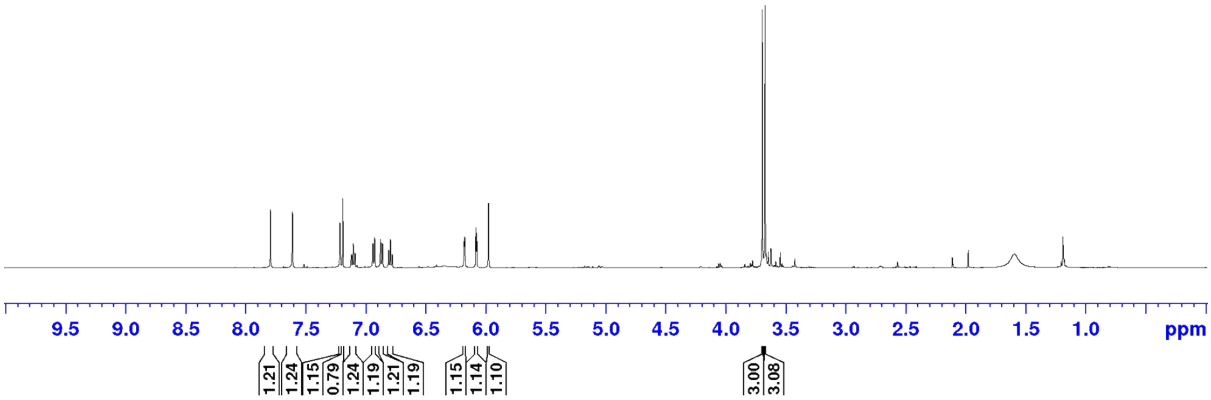
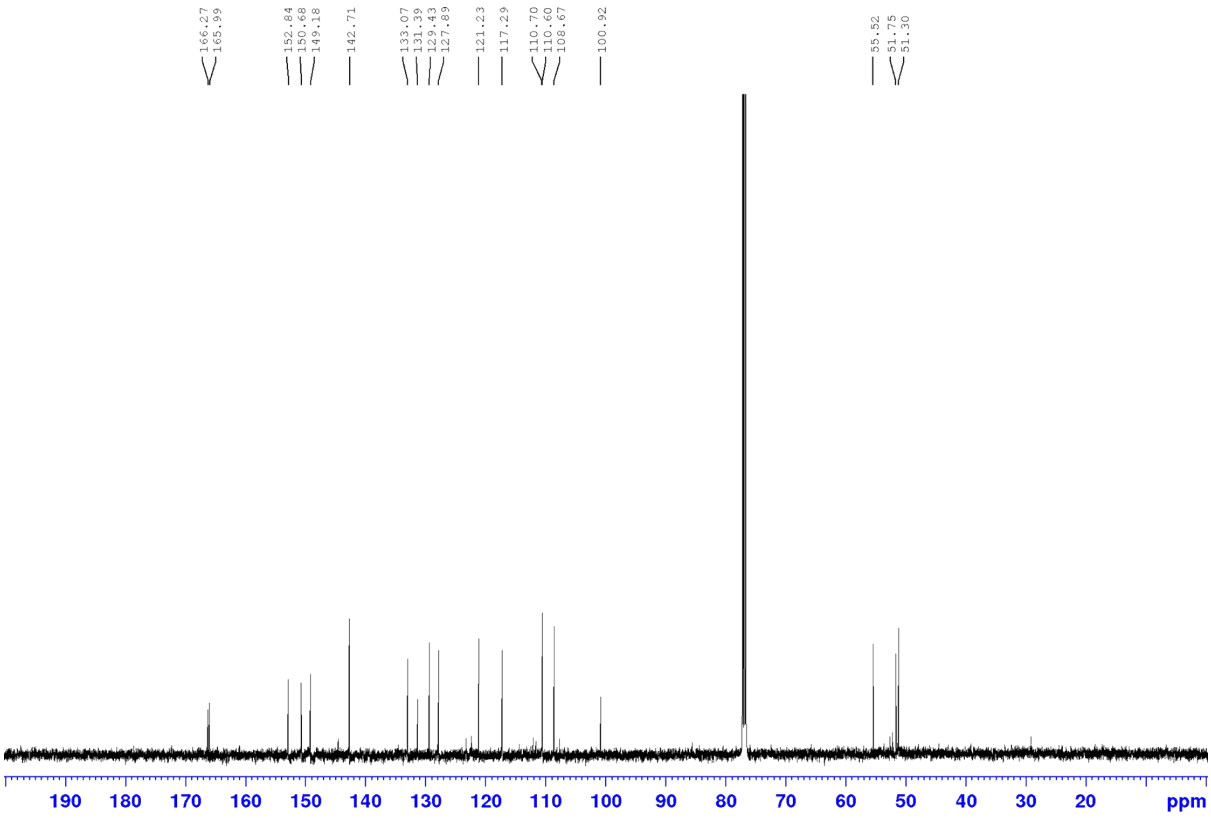
165.98
152.09
150.12
140.20
134.84
132.68
132.63
130.35
130.21
129.52
128.59
122.66
121.07
117.53
114.71
98.61
60.54
51.77
51.43
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

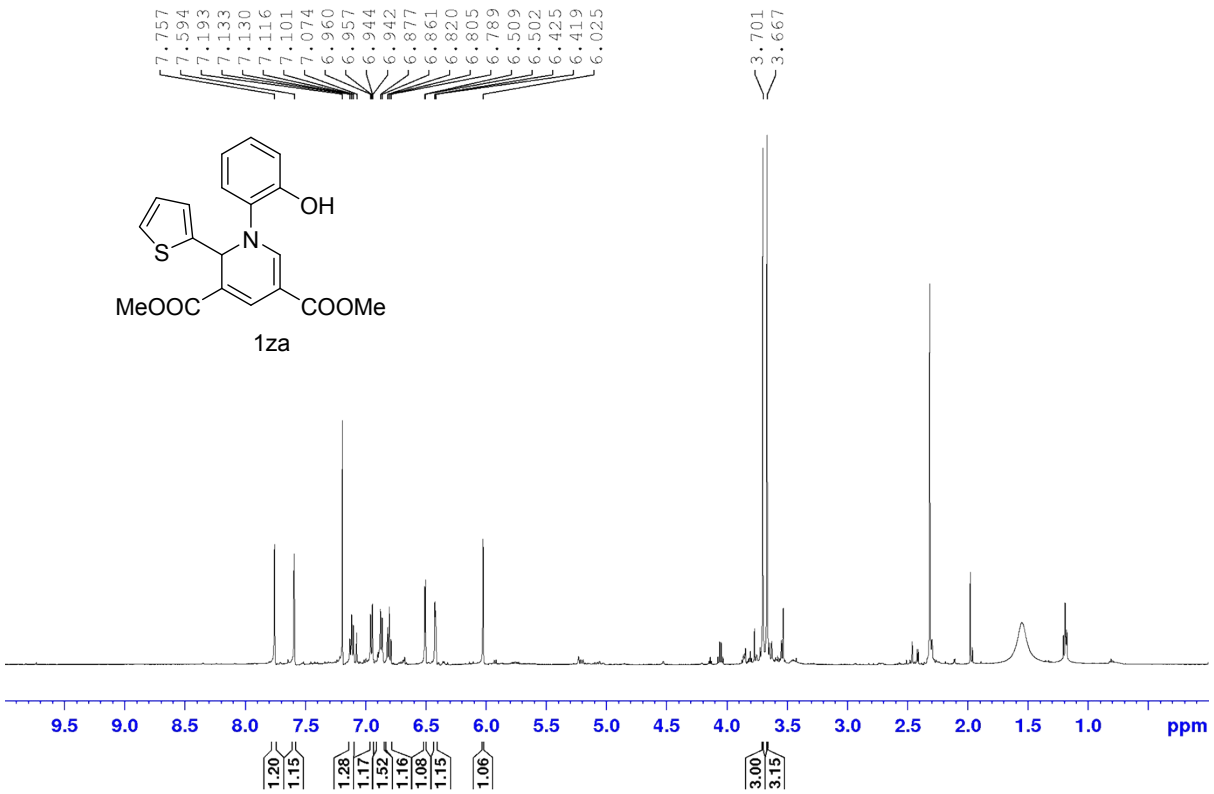

7.796
7.795
7.613
7.217
7.215
7.193
7.123
7.121
7.107
7.093
7.090
6.944
6.941
6.928
6.926
6.879
6.863
6.812
6.797
6.782
6.183
6.179
6.177
6.173
6.083
6.077
5.978
3.695
3.672

OH
N
O
MeOOC
COOMe

1z


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
ppm
1.21
1.24
1.15
0.79
1.24
1.19
1.21
1.19
1.15
1.14
1.10
3.00
3.08


166.27
165.99
152.84
150.68
149.18
142.71
133.07
131.39
129.43
127.89
121.23
117.29
110.70
110.60
108.67
100.92
55.52
51.75
51.30
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


7.757
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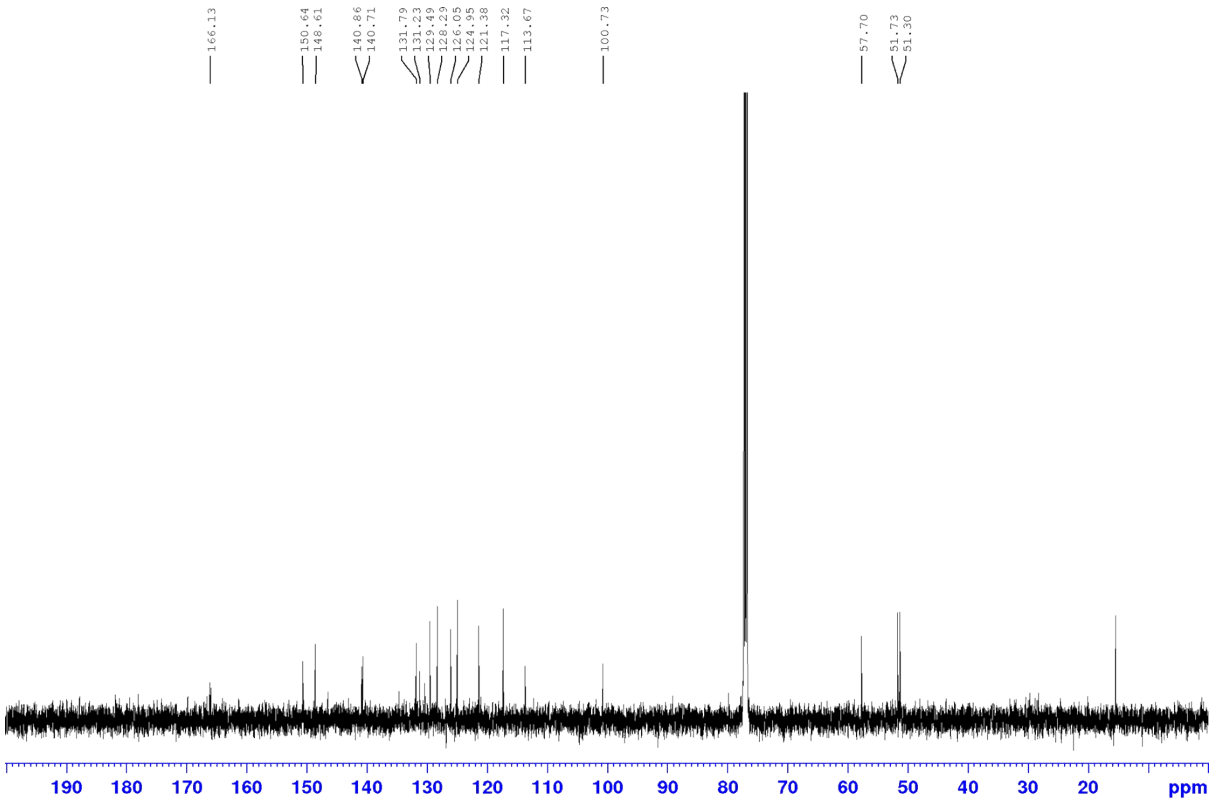
7.133
7.130
7.116
7.101
7.074
6.960
6.957
6.944
6.942
6.877
6.861
6.820
6.805
6.789
6.509
6.502
6.425
6.419
6.025
3.701
3.667
OH
N
S
MeOOC
COOMe
1za
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.20 1.15 1.28 1.17 1.52 1.16 1.08 1.15 1.06 3.00 3.15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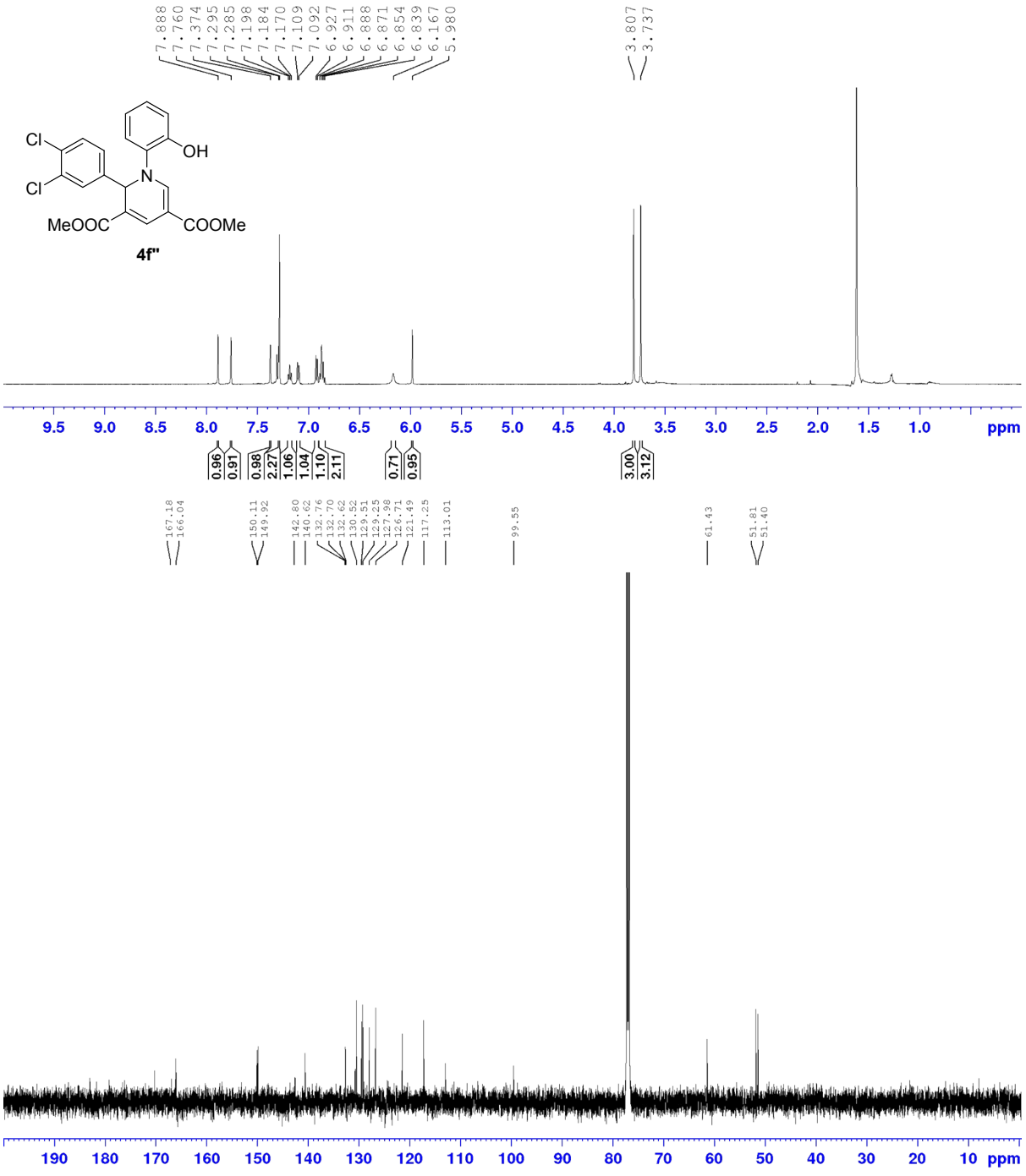
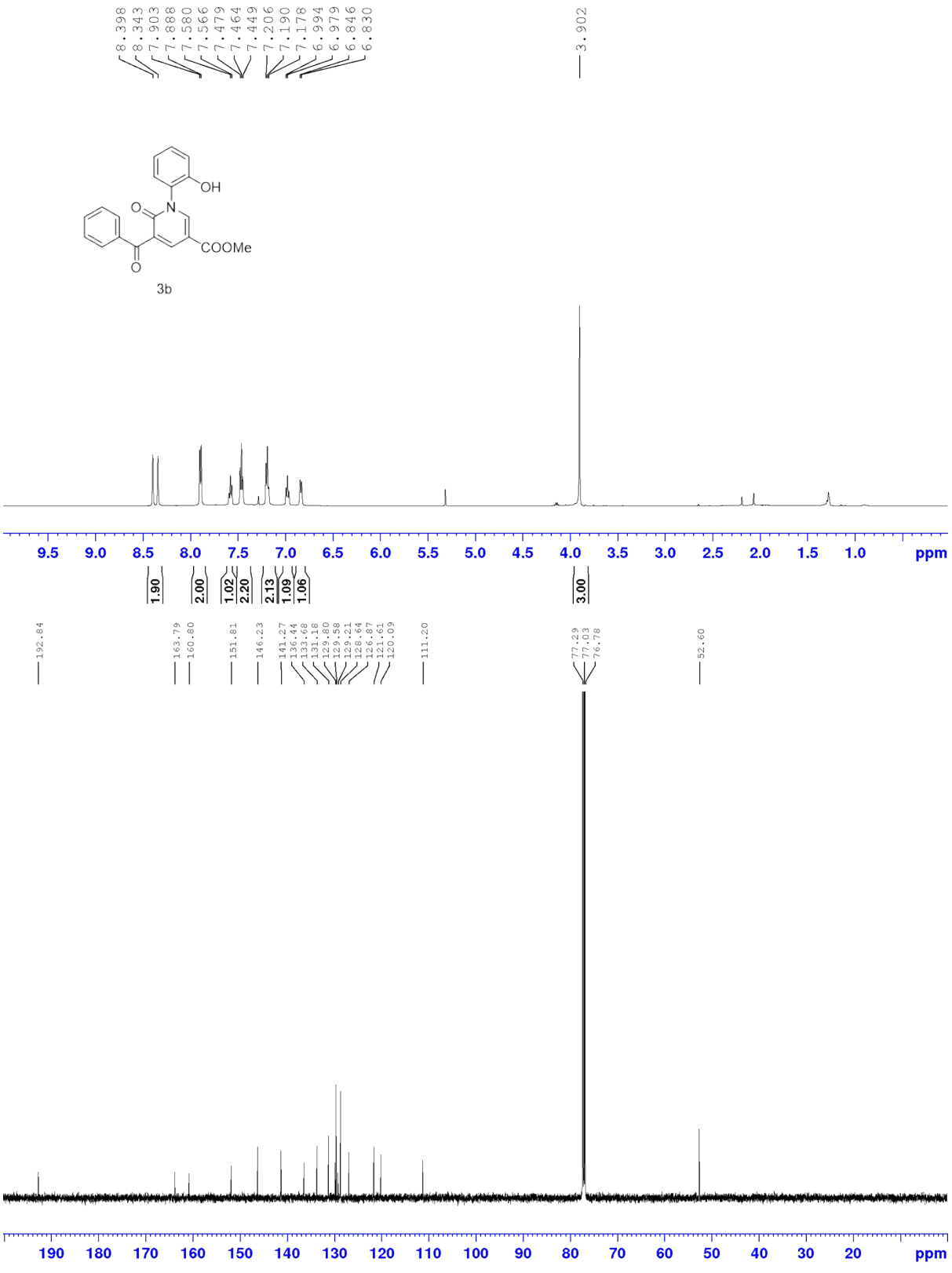
150.64
148.61
140.86
140.71
131.79
131.23
129.49
128.29
126.05
124.95
121.38
117.32
113.67
100.73
57.70
51.73
51.30
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.888
7.760
7.374
7.295
7.285
7.198
7.184
7.170
7.109
7.092
6.927
6.911
6.888
6.871
6.854
6.839
6.167
5.980
3.807
3.737
Cl
Cl
OH
N
MeOOC
COOMe
4f''
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 ppm
0.96
0.91
0.98
2.27
1.06
1.04
1.10
2.11
0.71
0.95
3.00
3.12
167.18
166.04
150.11
149.92
142.80
140.62
132.76
132.70
132.62
130.52
129.51
129.25
127.98
126.71
121.49
117.25
113.01
99.55
61.43
51.81
51.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


8.468
8.346
7.808
7.792
7.362
7.347
7.331
7.286
7.273
7.255
7.240
7.102
7.086
7.071
7.056
7.039
3.924
2.427
OH
Me
O
N
COOMe
O
3a
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 ppm
1.01
0.91
1.86
0.96
2.15
2.03
2.98
3.00
192.39
163.80
160.88
151.83
145.96
144.77
140.94
133.86
131.15
130.16
129.77
129.50
129.38
126.84
121.73
120.32
111.32
77.29
77.03
76.78
52.59
21.81
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.464
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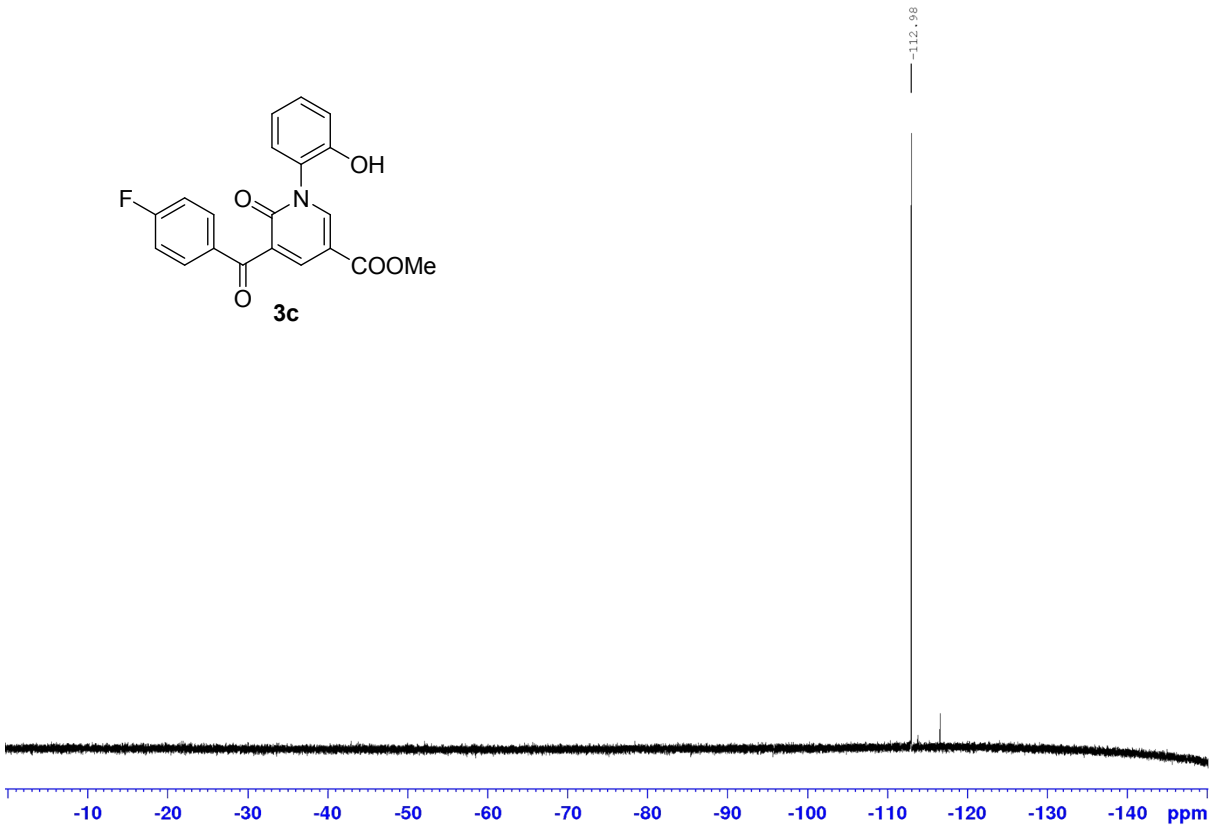
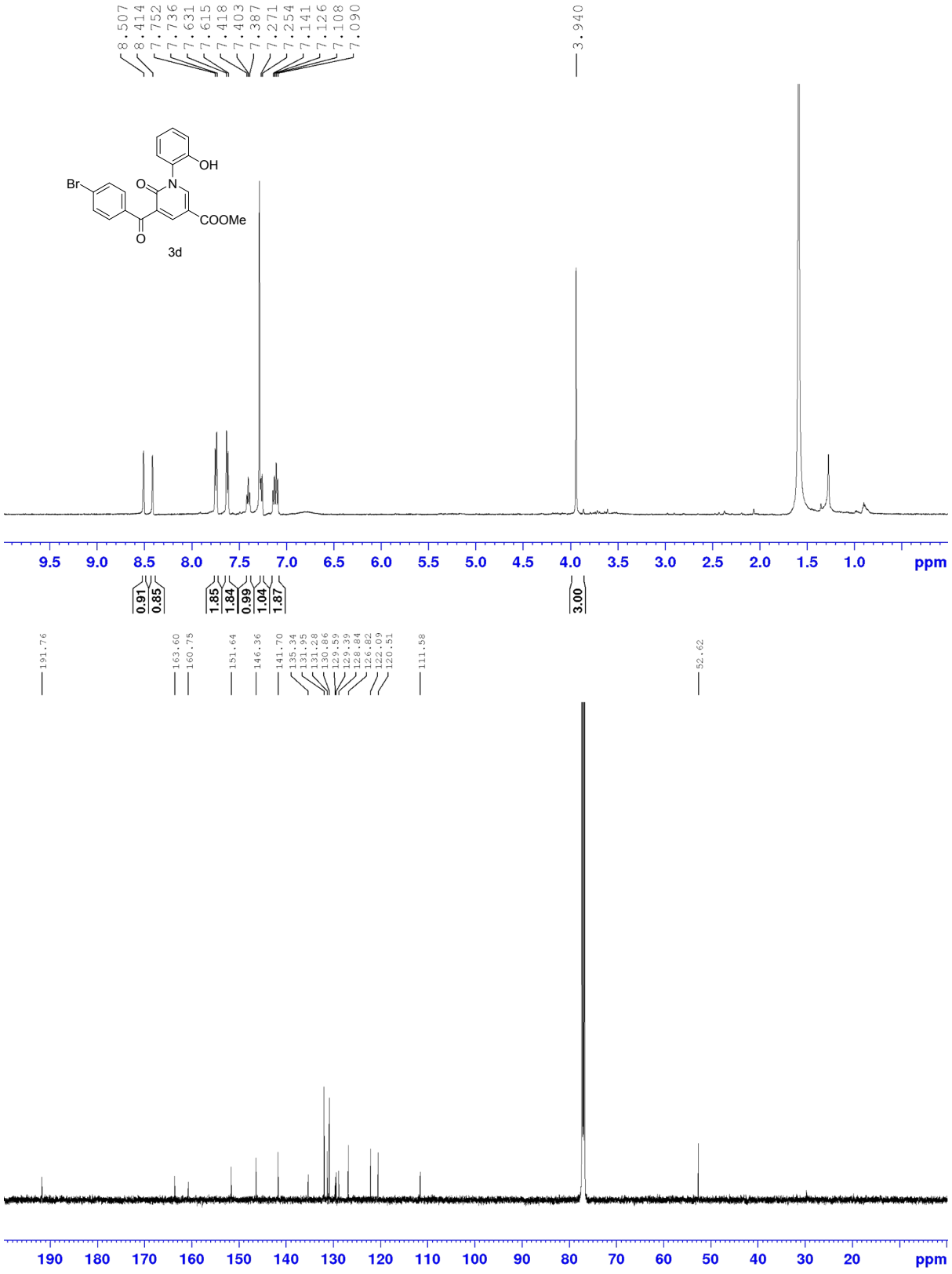
7.206
7.190
7.178
6.994
6.979
6.846
6.830
3.902
OH
N
O
COOMe
O
3b
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.90
2.00
1.02
2.20
2.13
1.09
1.06
3.00
192.84
163.79
160.80
151.81
146.23
141.27
136.44
133.68
131.18
129.80
129.58
129.21
128.64
126.87
121.61
120.09
111.20
77.29
77.03
76.78
52.60
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


8.475
8.402
7.837
7.820
7.449
7.433
7.317
7.302
7.284
7.248
7.233
7.090
7.075
6.981
6.965
3.930
OH
F
O
N
COOMe
O
3c
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 ppm
0.94
0.88
1.91
1.87
1.03
1.00
1.34
0.99
3.00
191.57
163.63
160.74
151.66
146.36
141.62
140.07
134.91
131.24
130.81
129.45
128.96
126.81
121.89
120.26
111.47
77.25
76.99
76.74
52.59
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


-112.98
OH
F
O
N
COOMe
O
3c
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
ppm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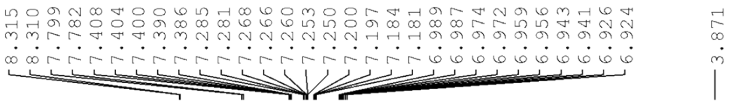
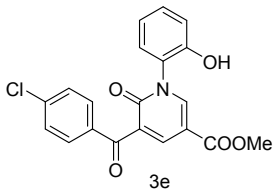
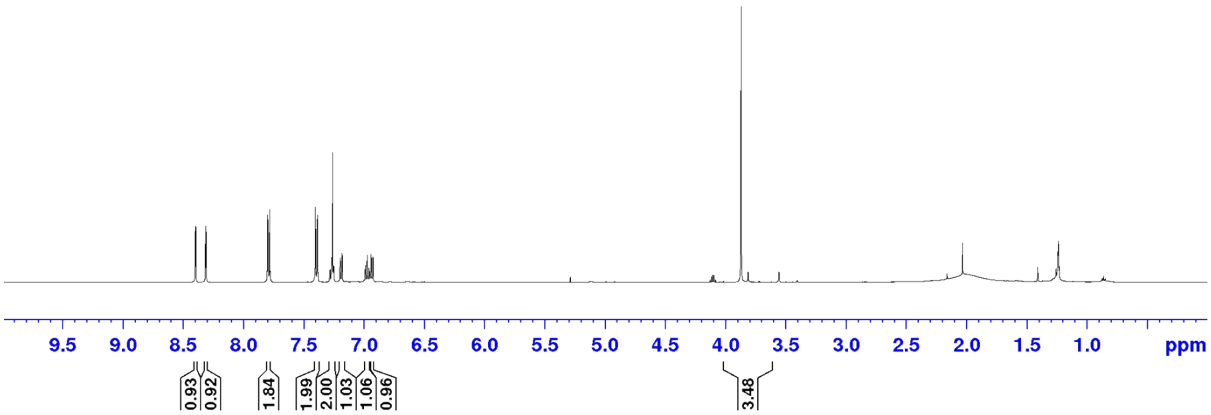
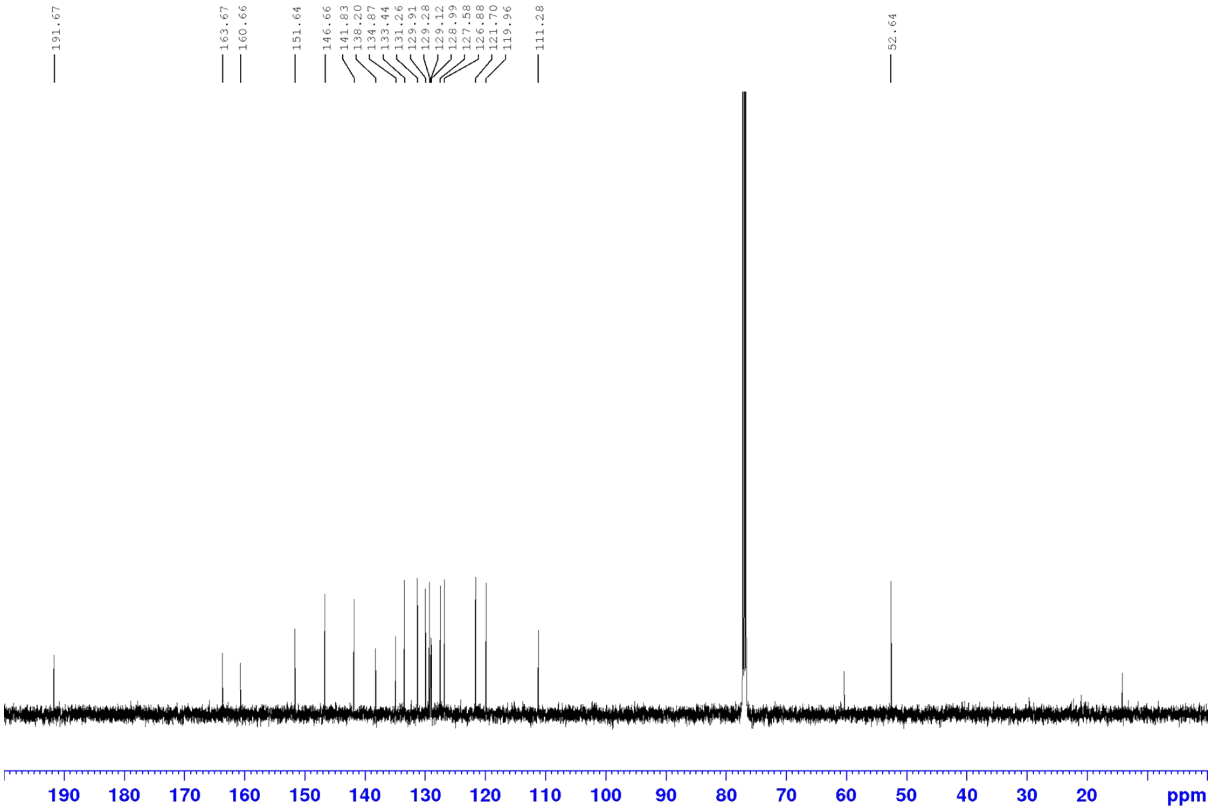
8.414
7.752
7.736
7.631
7.615
7.418
7.403
7.387
7.271
7.254
7.141
7.126
7.108
7.090
3.940
OH
Br
O
N
COOMe
O
3d
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.91
0.85
1.85
1.84
0.99
1.04
1.87
3.00
191.76
163.60
160.75
151.64
146.36
141.70
135.34
131.95
131.28
130.86
129.59
129.39
128.84
126.82
122.09
120.51
111.58
52.62
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

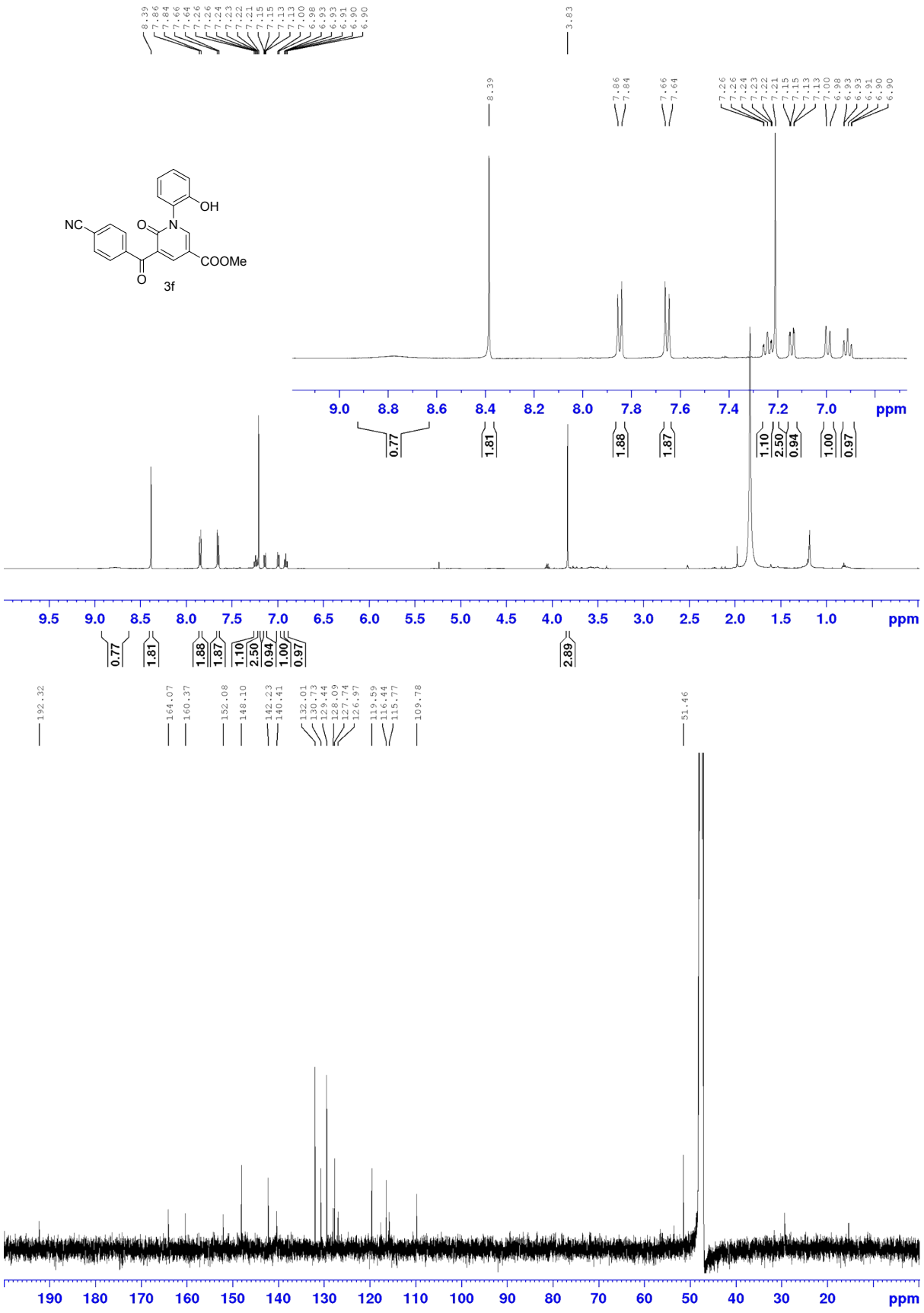

8.315
8.310
7.799
7.782
7.408
7.404
7.400
7.390
7.386
7.285
7.281
7.268
7.266
7.260
7.253
7.250
7.200
7.197
7.184
7.181
6.989
6.987
6.974
6.972
6.959
6.956
6.943
6.941
6.926
6.924
3.871


OH
Cl
O
N
COOMe
O

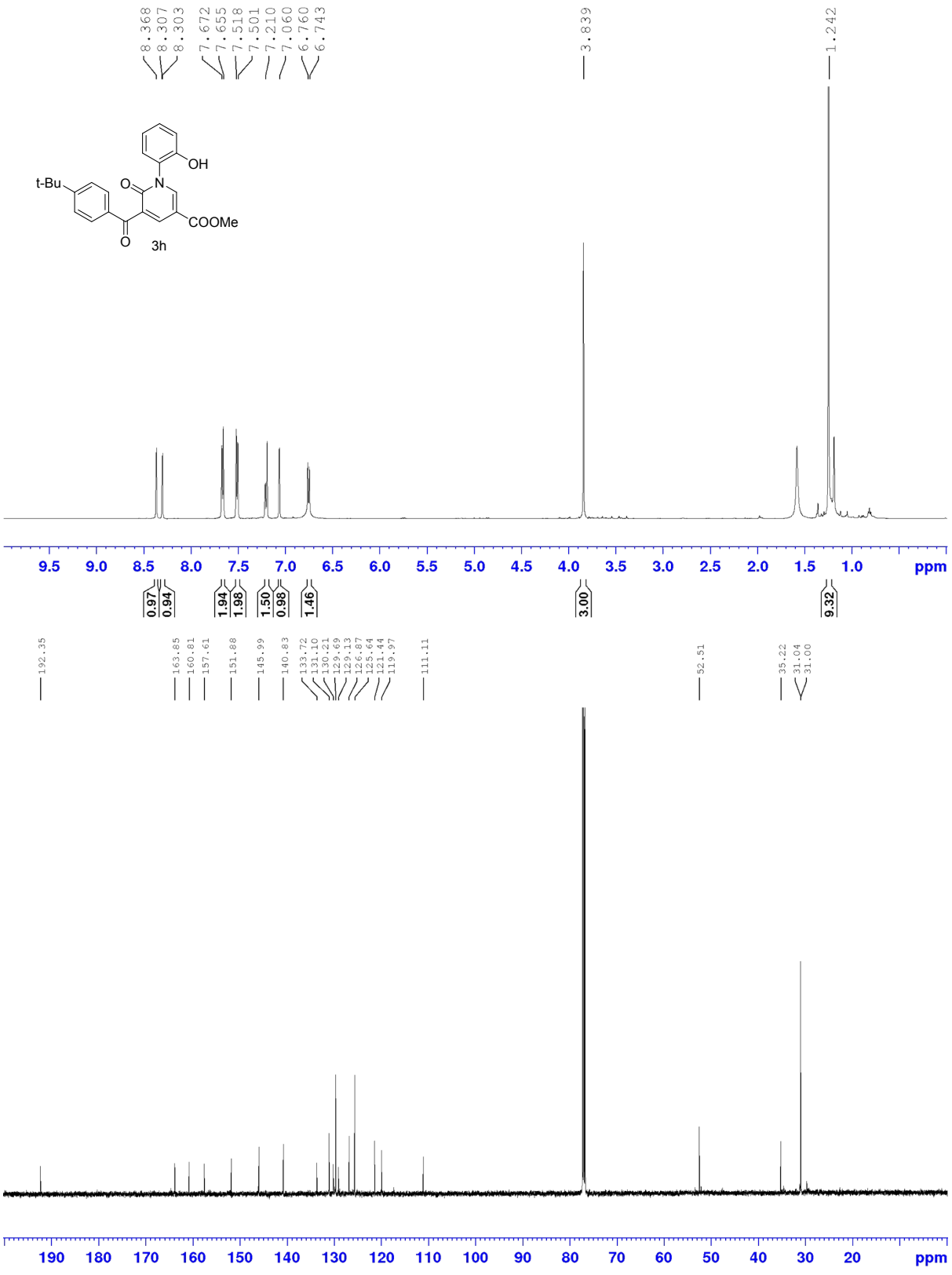
3e

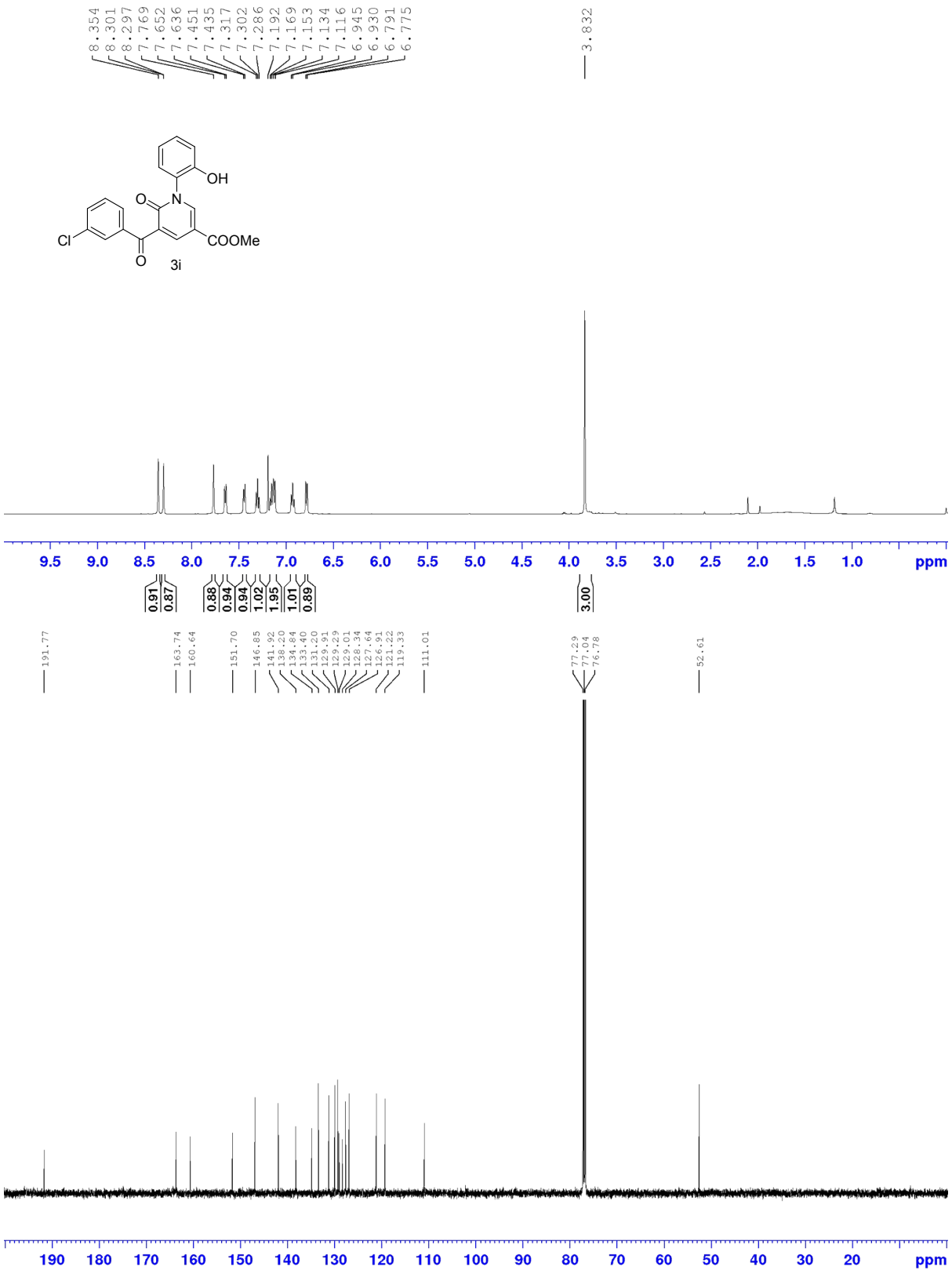

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.93
0.92
1.84
1.99
2.00
1.03
1.06
0.96
3.48

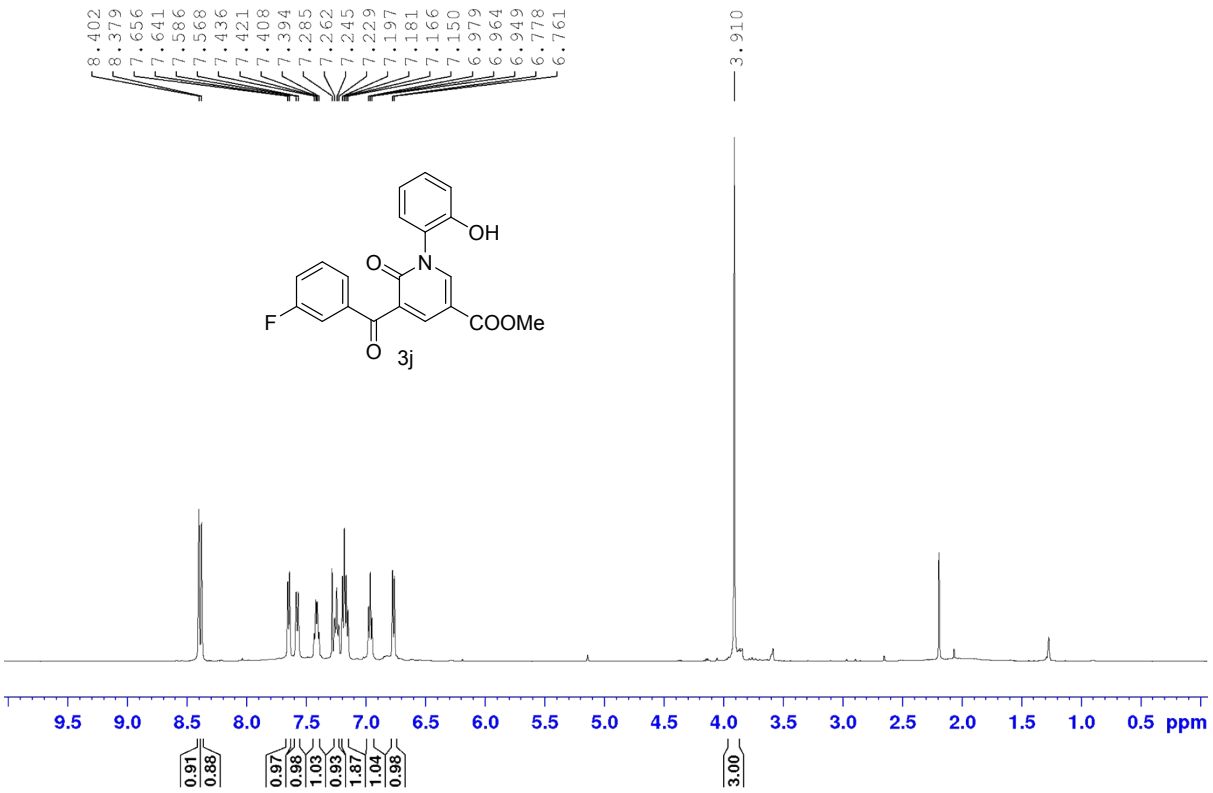

191.67
163.67
160.66
151.64
146.66
141.83
138.20
134.87
134.44
131.26
129.91
129.28
129.12
128.99
127.58
126.88
121.70
119.96
111.28
52.64
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

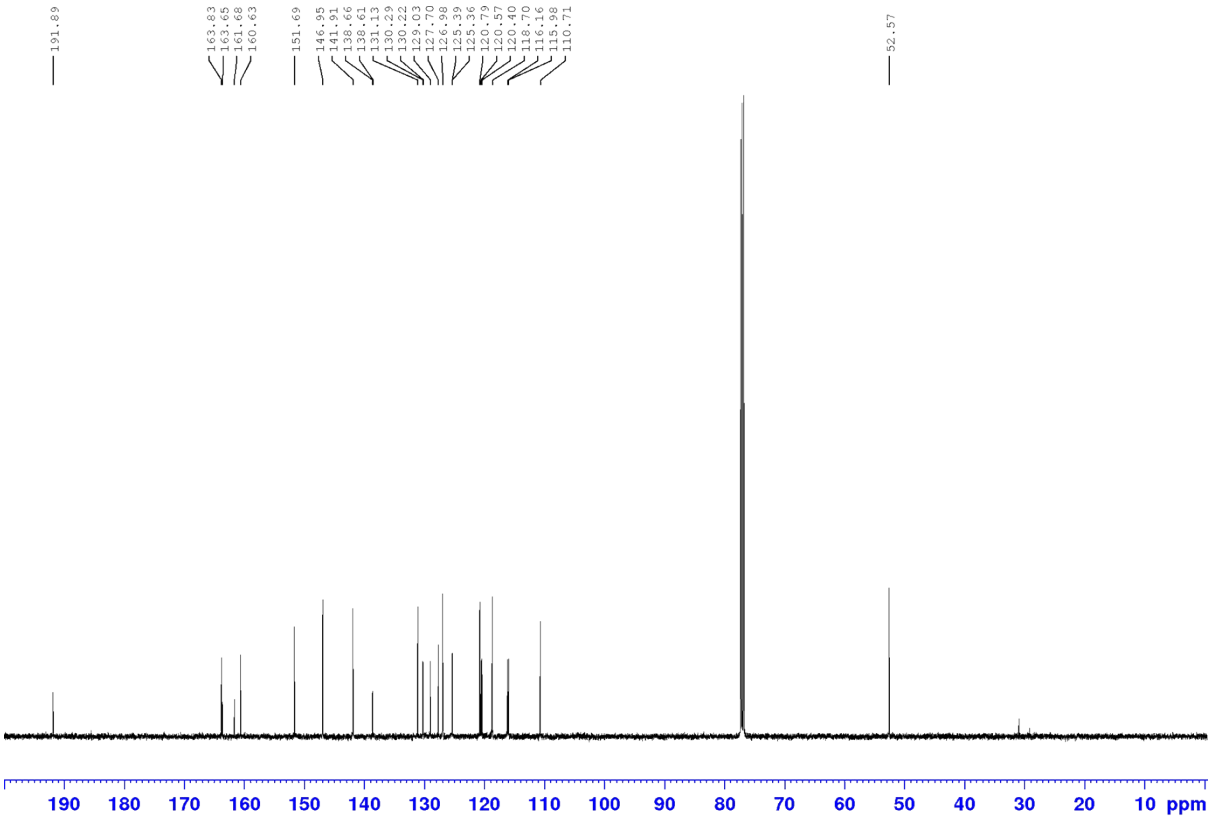

8.39
7.86
7.84
7.66
7.64
7.26
7.24
7.23
7.22
7.21
7.15
7.13
7.00
6.98
6.93
6.91
6.90
3.83
OH
NC
O
N
COOMe
O
3f
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
ppm
0.77
1.81
1.88
1.87
1.10
2.50
0.94
1.00
0.97
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
0.77
1.81
1.88
1.87
1.10
2.50
0.94
1.00
0.97
2.89
192.32
164.07
160.37
152.08
148.10
142.23
140.41
132.01
130.73
129.44
128.09
127.74
126.97
119.59
116.44
115.77
109.78
51.46
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

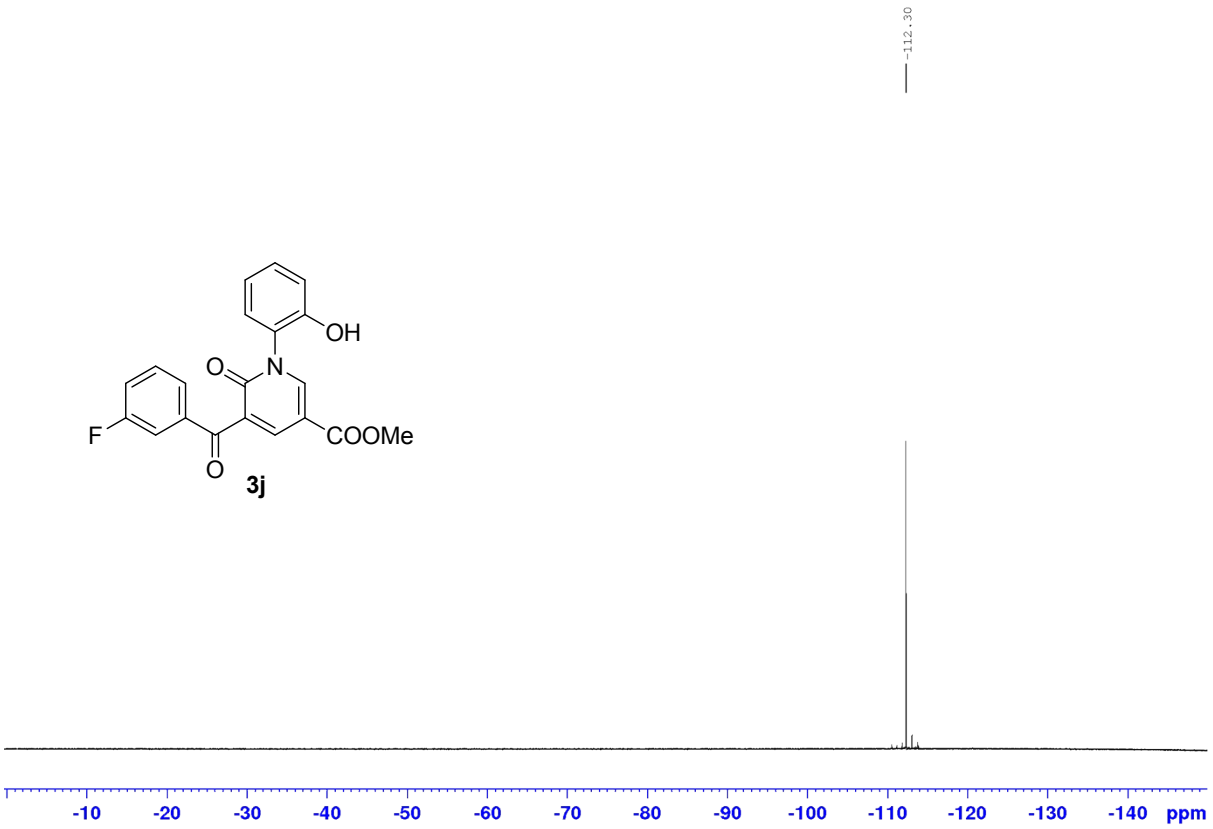

8.374
8.281
8.280
7.901
7.884
7.283
7.199
7.183
7.166
6.983
6.968
6.935
6.918
6.852
6.836
3.890
3.842
OH
MeO
O
N
COOMe
O
3g
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.96 0.96 1.98 2.05 1.20 2.01 1.14 2.87 3.00
191.47
164.13
163.96
160.76
151.89
146.10
140.64
132.25
131.00
130.22
129.22
128.22
126.92
120.77
119.07
113.94
110.69
55.52
52.47
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

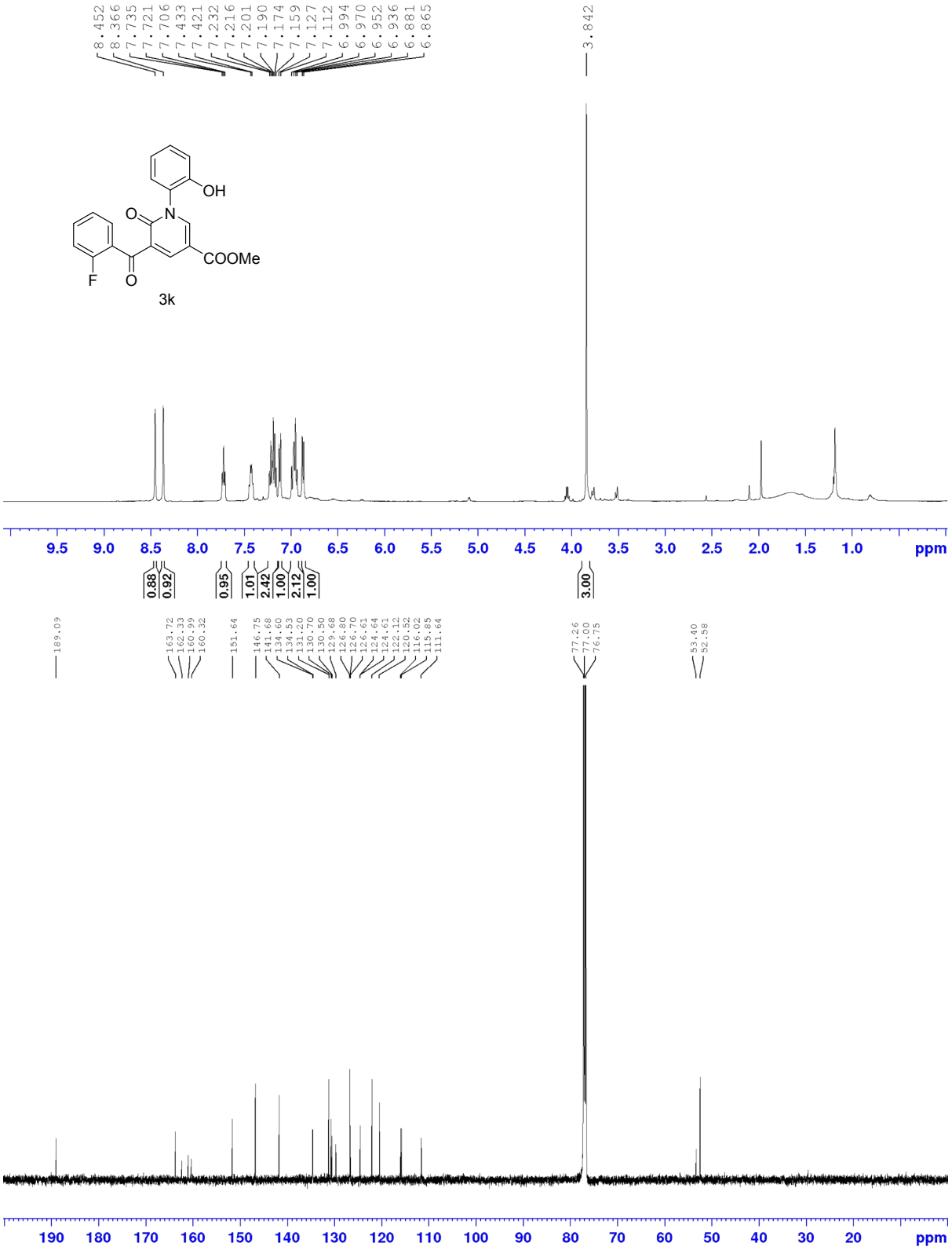

8.368
8.307
8.303
7.672
7.655
7.518
7.501
7.210
7.060
6.760
6.743
3.839
1.242
OH
t-Bu
O
N
COOMe
O
3h
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.97
0.94
1.94
1.98
1.50
0.98
1.46
3.00
9.32
192.35
163.85
160.81
157.61
151.88
145.99
140.83
133.72
131.10
130.21
129.69
129.13
126.87
125.64
121.44
119.97
111.11
52.51
35.22
31.04
31.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

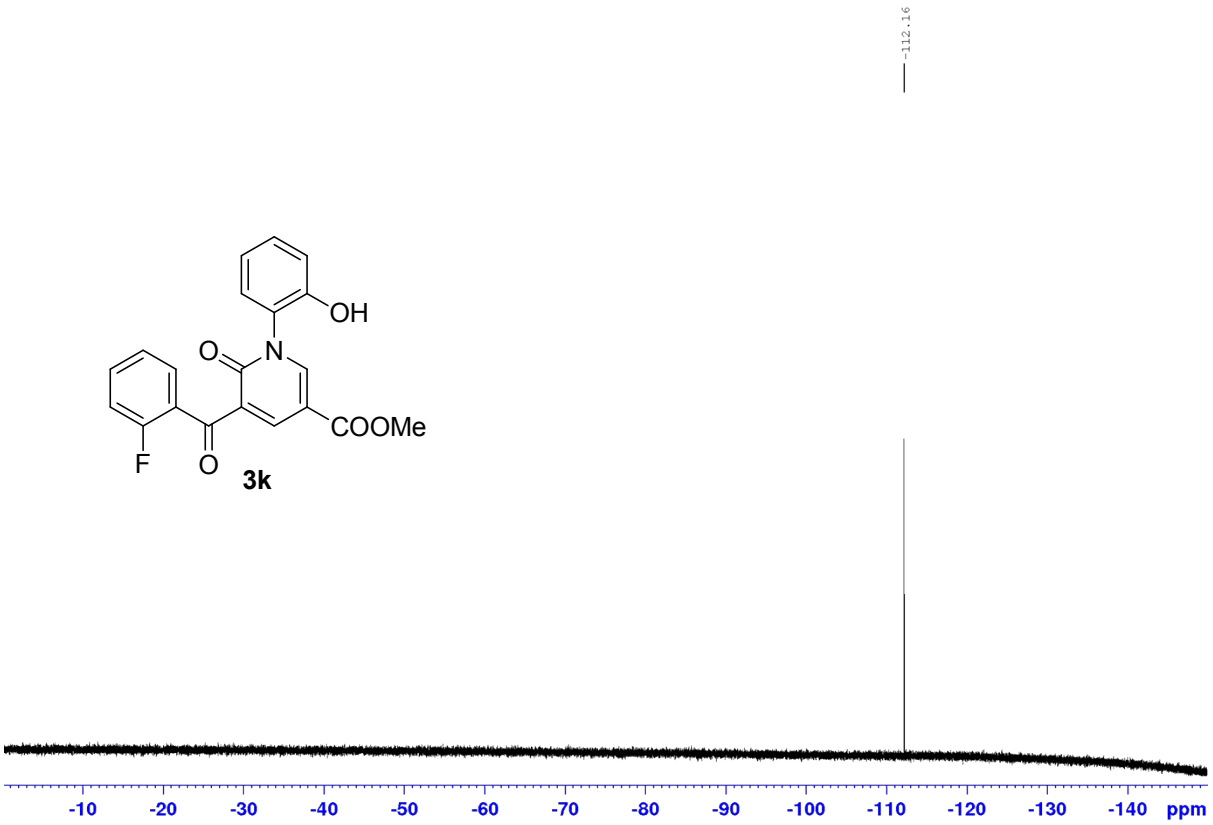

8.354
8.301
8.297
7.769
7.652
7.636
7.451
7.435
7.317
7.302
7.286
7.192
7.169
7.153
7.134
7.116
6.945
6.930
6.791
6.775
3.832
OH
N
O
Cl
COOMe
O
3i
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.91 0.87 0.88 0.94 0.94 1.02 1.95 1.01 0.89 3.00
191.77
163.74
160.64
151.70
146.85
141.92
138.20
134.84
133.40
131.20
129.21
129.01
128.34
127.64
126.91
121.22
119.33
111.01
77.29
77.04
76.78
52.61
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

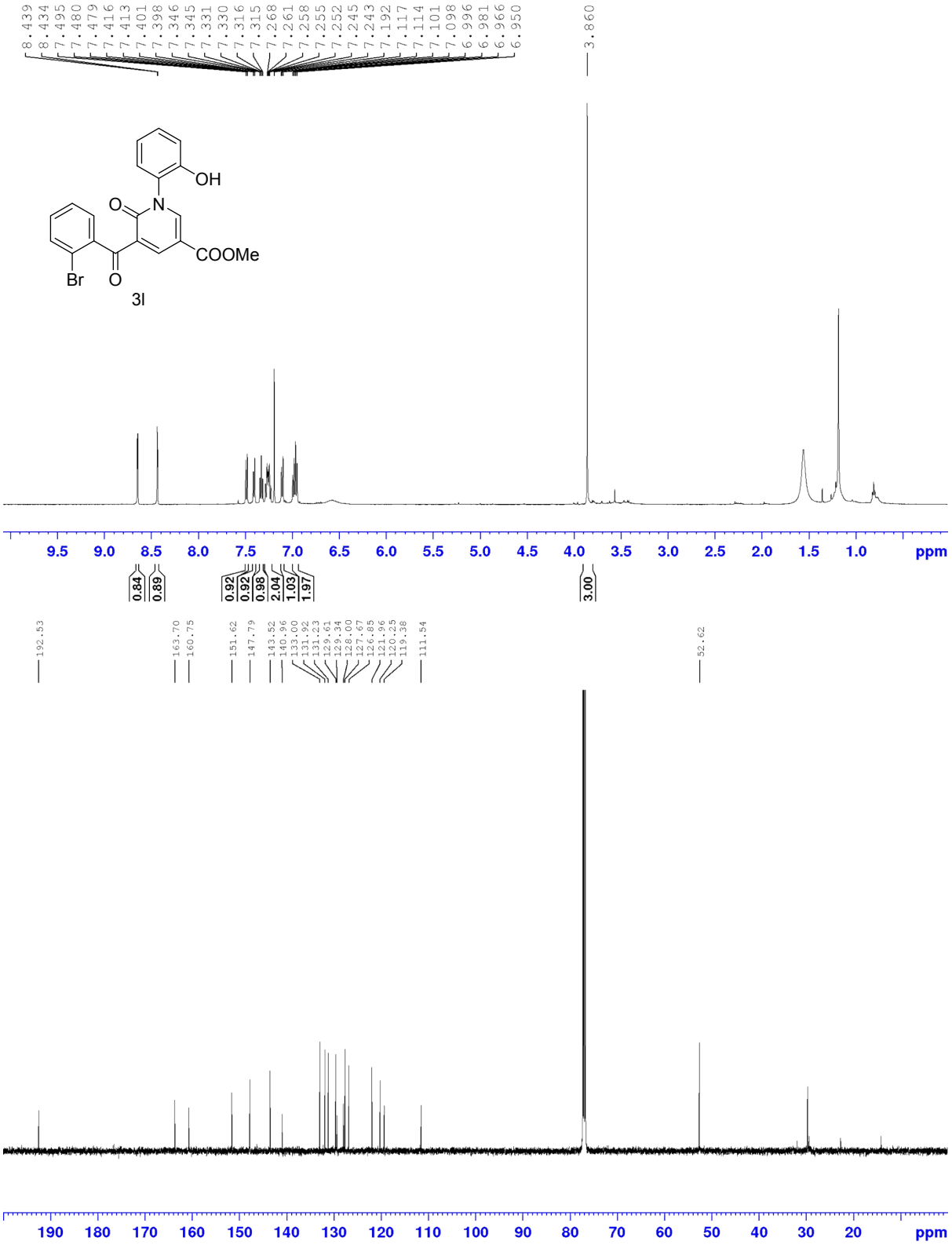

8.402
8.379
7.656
7.641
7.586
7.568
7.436
7.421
7.408
7.394
7.285
7.262
7.245
7.229
7.197
7.181
7.166
7.150
6.979
6.964
6.949
6.778
6.761
3.910
OH
O
N
F
COOMe
O
3j
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.91
0.88
0.97
0.98
1.03
0.93
1.87
1.04
0.98
3.00

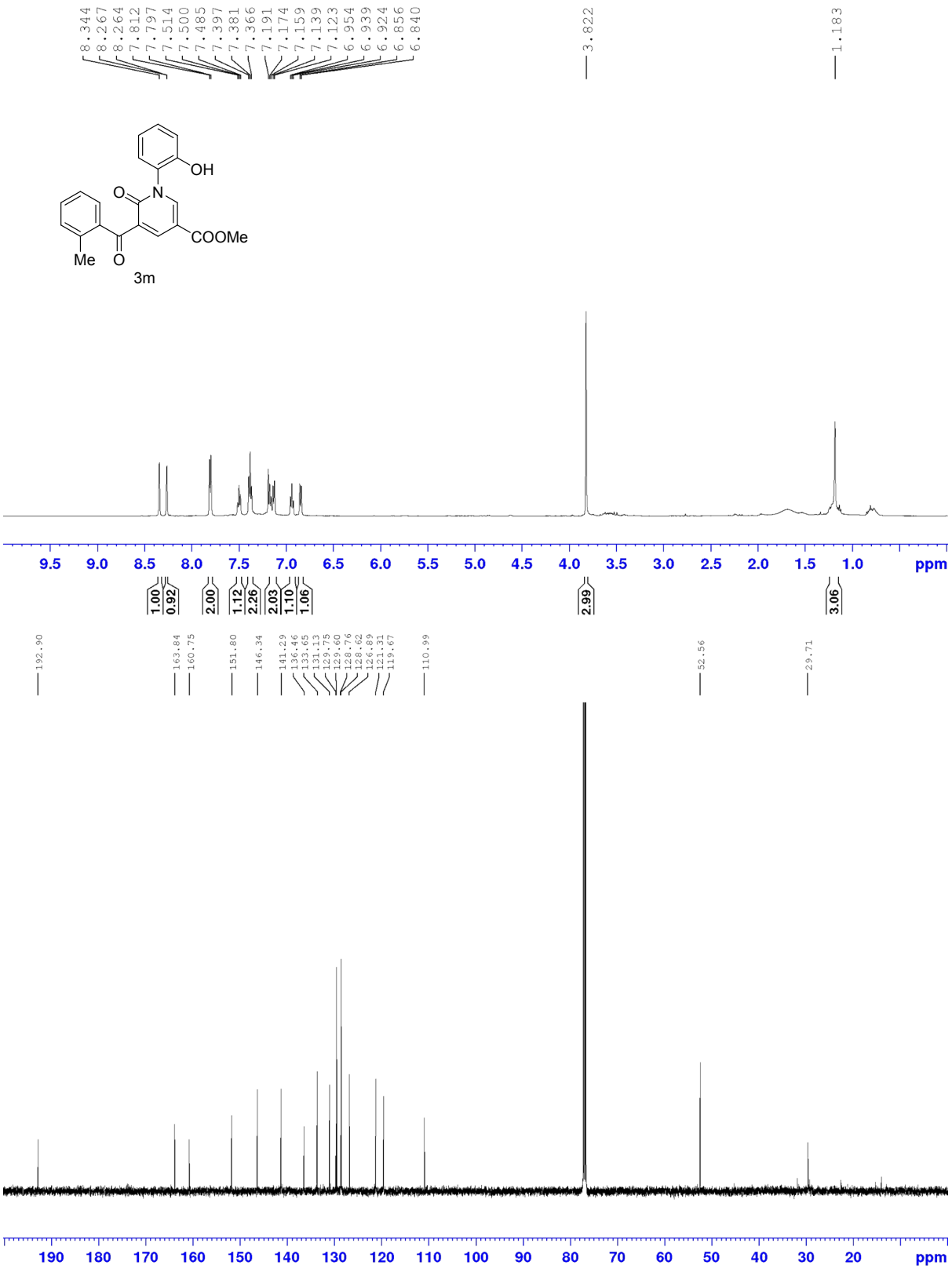

191.89
163.83
163.65
161.68
160.63
151.69
146.95
141.91
138.66
138.61
131.13
130.29
130.22
129.03
127.70
126.98
125.39
125.36
120.79
120.57
120.40
118.70
116.16
115.98
110.71
52.57
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

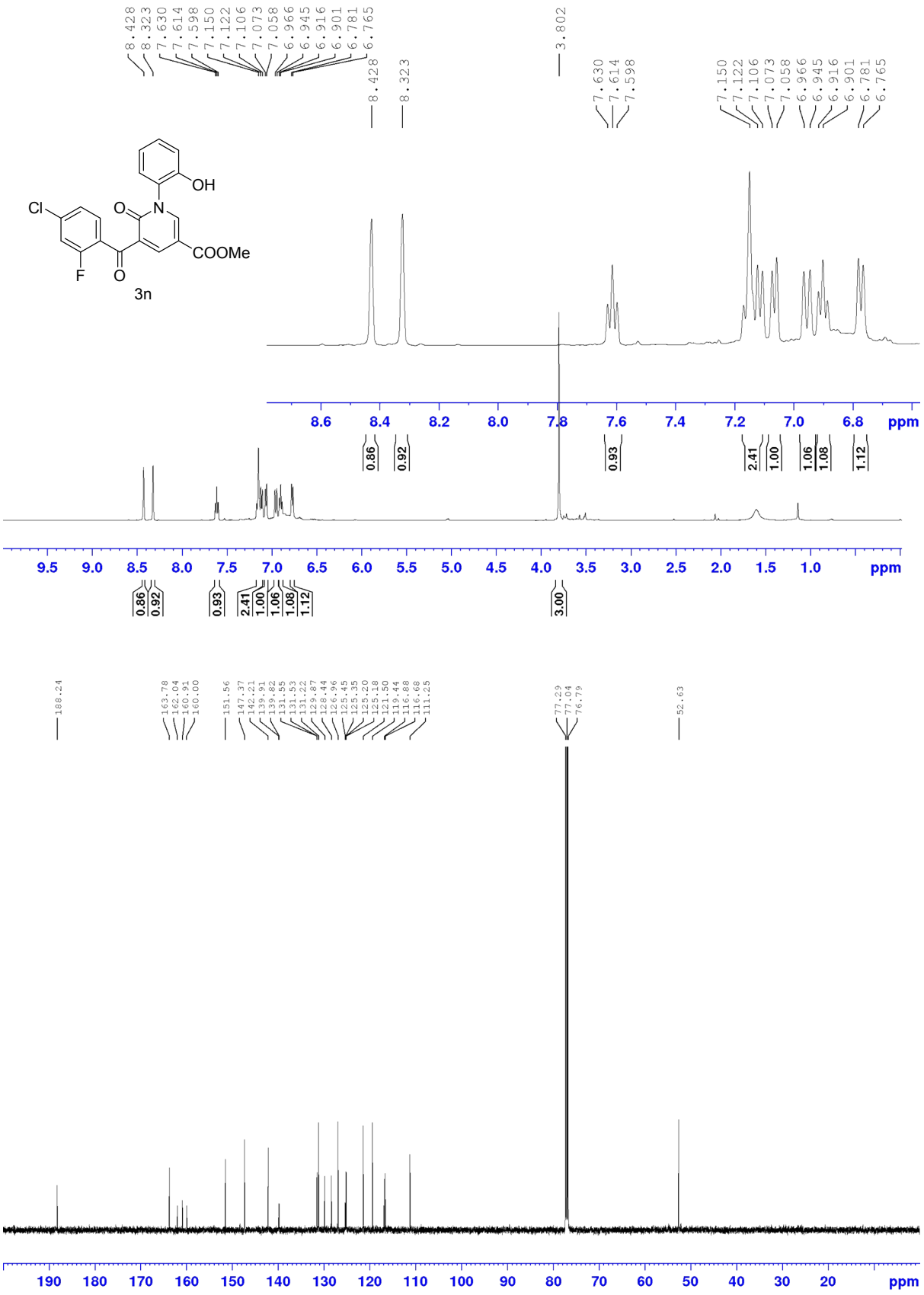

-112.30
OH
O
N
F
COOMe
O
3j
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
ppm

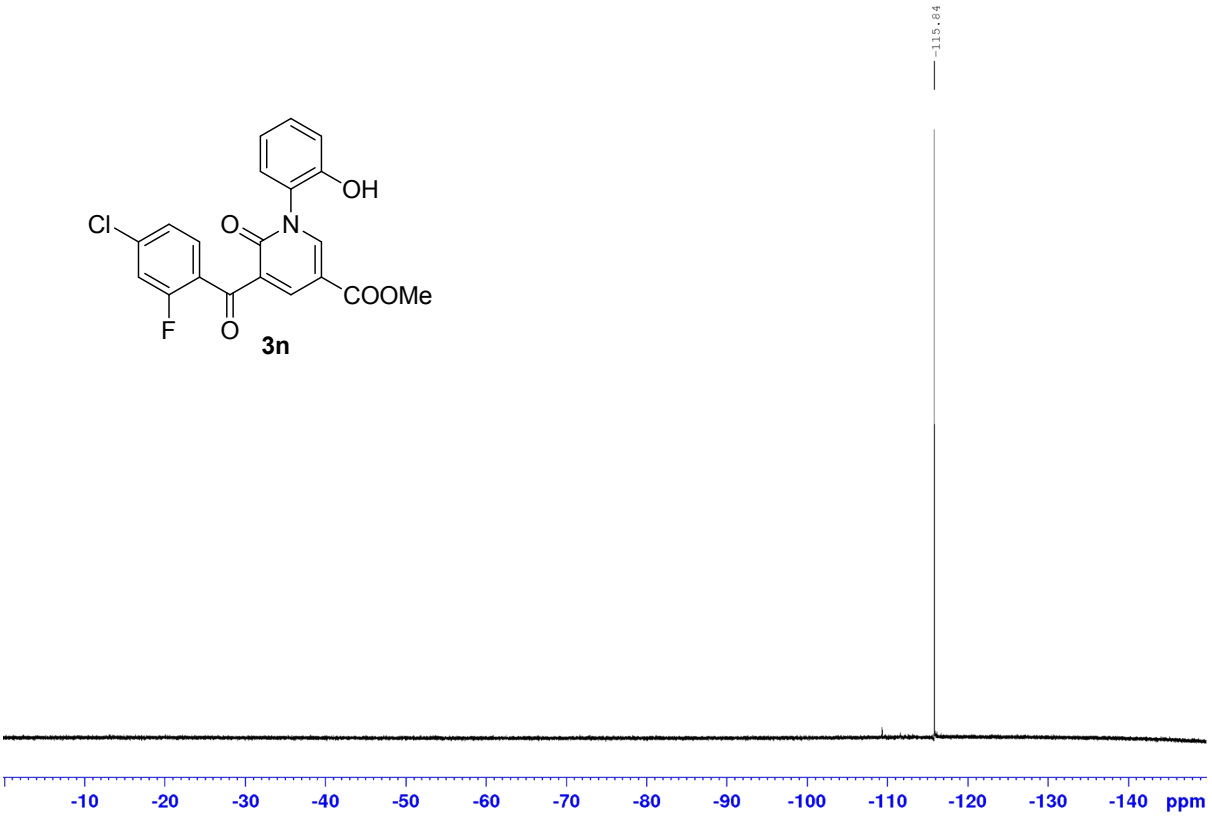

8.452
8.366
7.735
7.721
7.706
7.433
7.421
7.232
7.216
7.201
7.190
7.174
7.159
7.127
7.112
6.994
6.970
6.952
6.936
6.881
6.865
3.842
OH
O
N
COOMe
F
O
3k
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.88 0.92 0.95 1.01 2.42 1.00 2.12 1.00 3.00
189.09
163.72
162.33
160.99
160.32
151.64
146.75
141.68
134.60
134.53
131.20
130.70
130.50
129.68
126.80
126.70
126.61
124.64
124.61
122.12
120.52
116.02
115.85
111.64
77.26
77.00
76.75
53.40
52.58
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


-112.16
OH
O
N
COOMe
F
O
3k
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
ppm

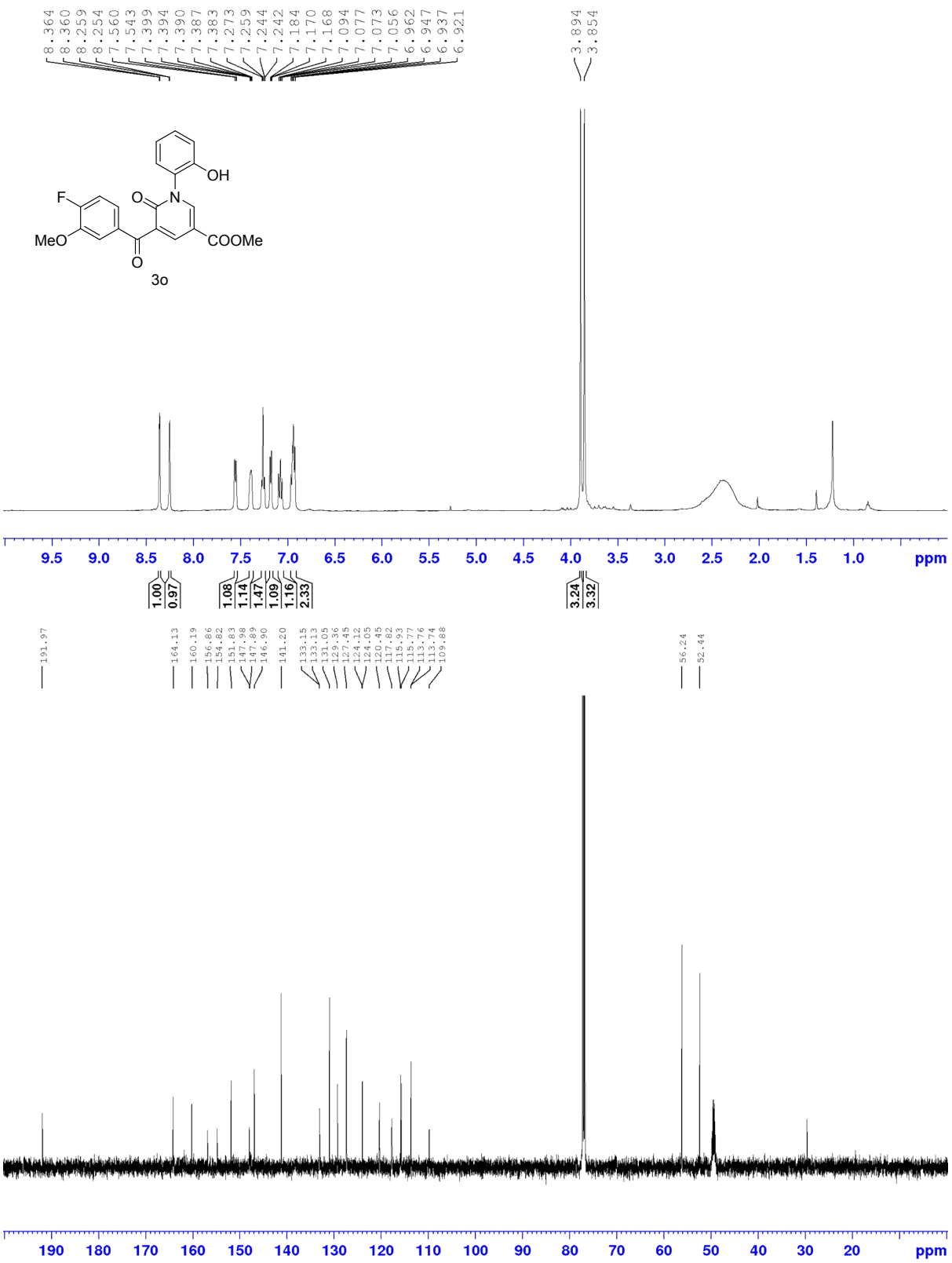

8.439
8.434
7.495
7.480
7.479
7.416
7.413
7.401
7.398
7.346
7.345
7.331
7.330
7.316
7.315
7.268
7.261
7.258
7.255
7.252
7.245
7.243
7.192
7.117
7.114
7.101
7.098
6.996
6.981
6.966
6.950
3.860
OH
O
N
COOMe
Br
O
3l
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.84
0.89
0.92
0.92
0.98
2.04
1.03
1.97
3.00
192.53
163.70
160.75
151.62
147.79
143.52
140.96
133.00
131.92
131.23
129.61
129.34
128.00
127.67
126.85
121.96
120.25
119.38
111.54
52.62
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

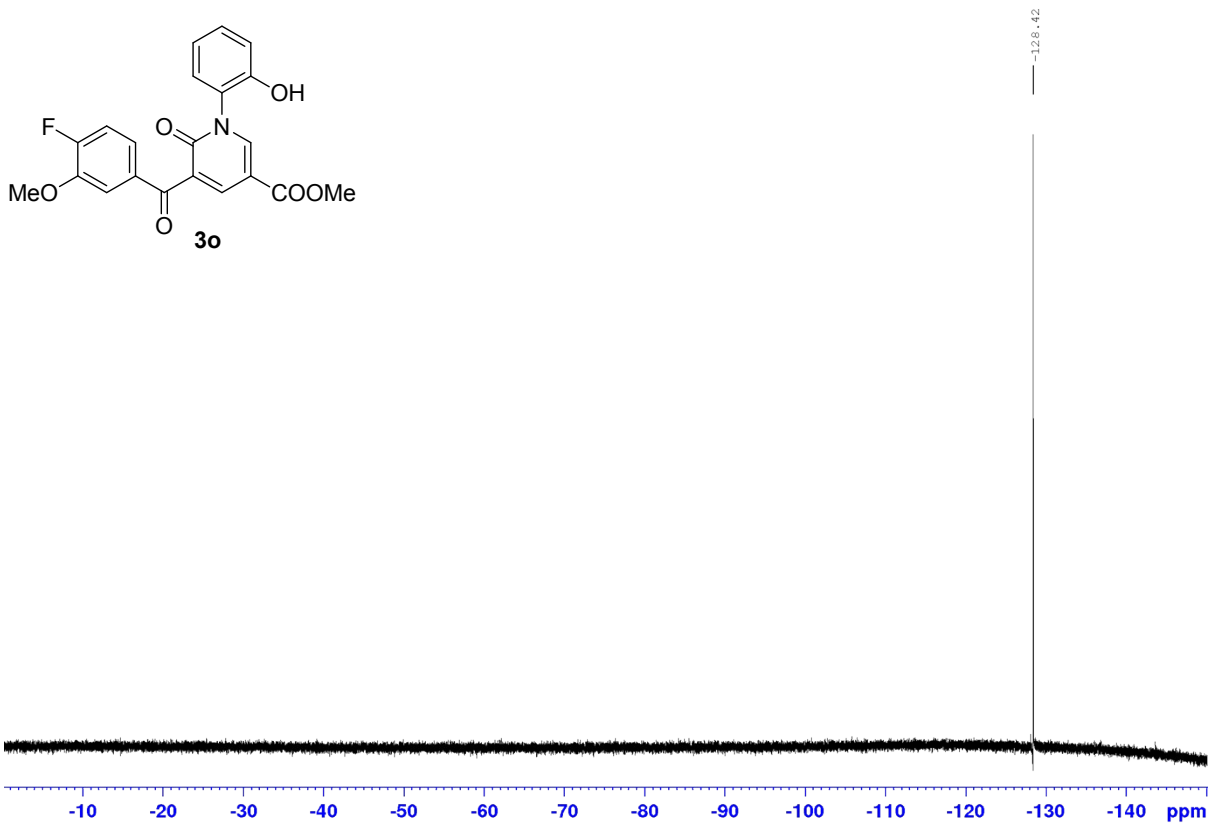

8.344
8.267
8.264
7.812
7.797
7.514
7.500
7.485
7.397
7.381
7.366
7.191
7.174
7.159
7.139
7.123
6.954
6.939
6.924
6.856
6.840
3.822
1.183
OH
O
N
COOMe
Me
O
3m
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00 0.92 2.00 1.12 2.26 2.03 1.10 1.06 2.99 3.06
192.90
163.84
160.75
151.80
146.34
141.29
136.46
133.65
131.13
129.75
129.60
128.76
128.62
126.89
121.31
119.67
110.99
52.56
29.71
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


8.428
8.323
7.630
7.614
7.598
7.150
7.122
7.106
7.073
7.058
6.966
6.945
6.916
6.901
6.781
6.765
3.802
OH
Cl
O
N
COOMe
F
O
3n
ppm
0.86
0.92
0.93
2.41
1.00
1.06
1.08
1.12
3.00
188.24
163.78
162.04
160.91
160.00
151.56
147.37
140.21
139.81
139.82
131.55
131.53
131.22
129.87
128.44
126.96
125.45
125.35
125.20
125.18
121.50
119.44
116.88
116.68
111.25
77.29
77.04
76.79
52.63

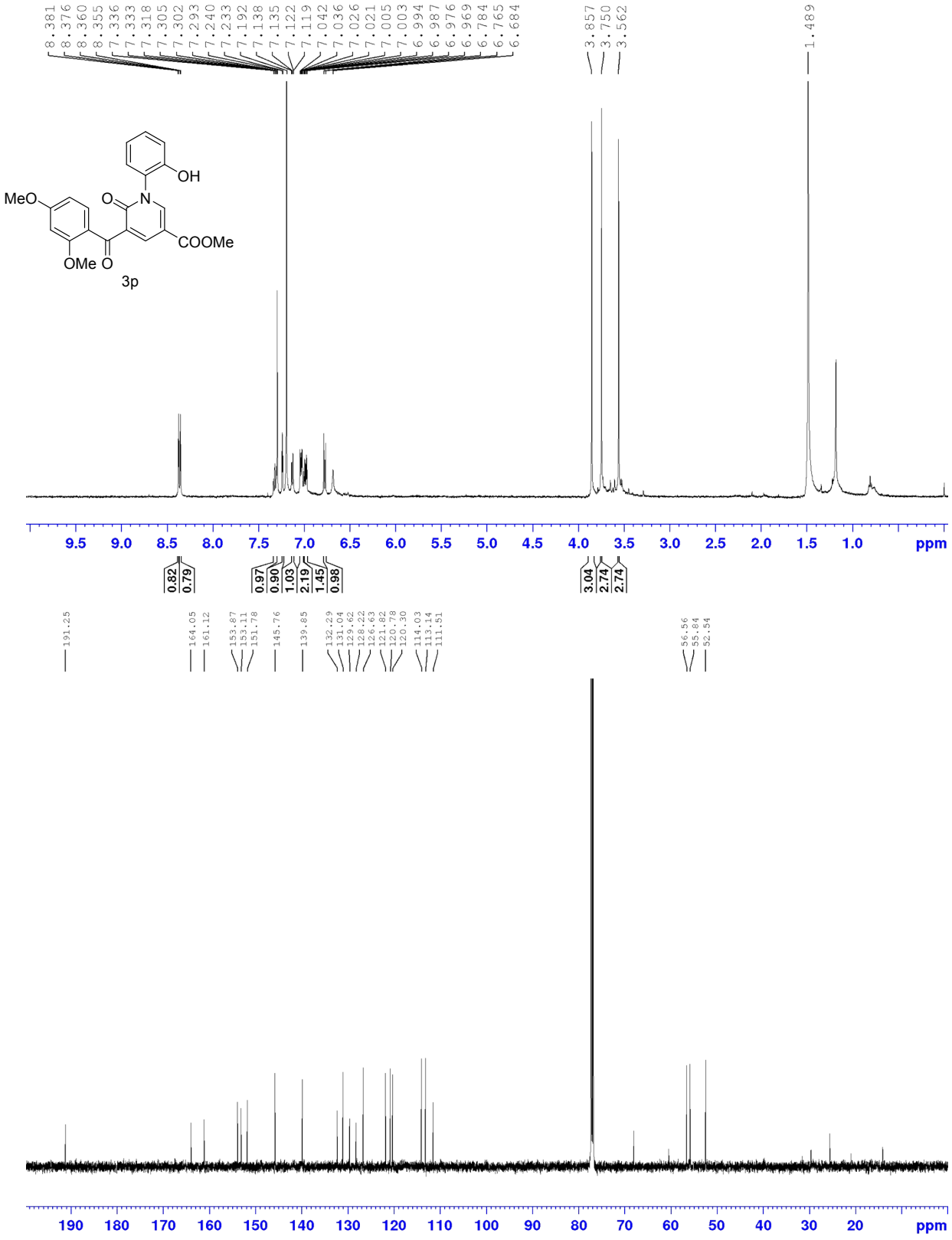

-115.84
OH
Cl
O
N
COOMe
F
O
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
ppm

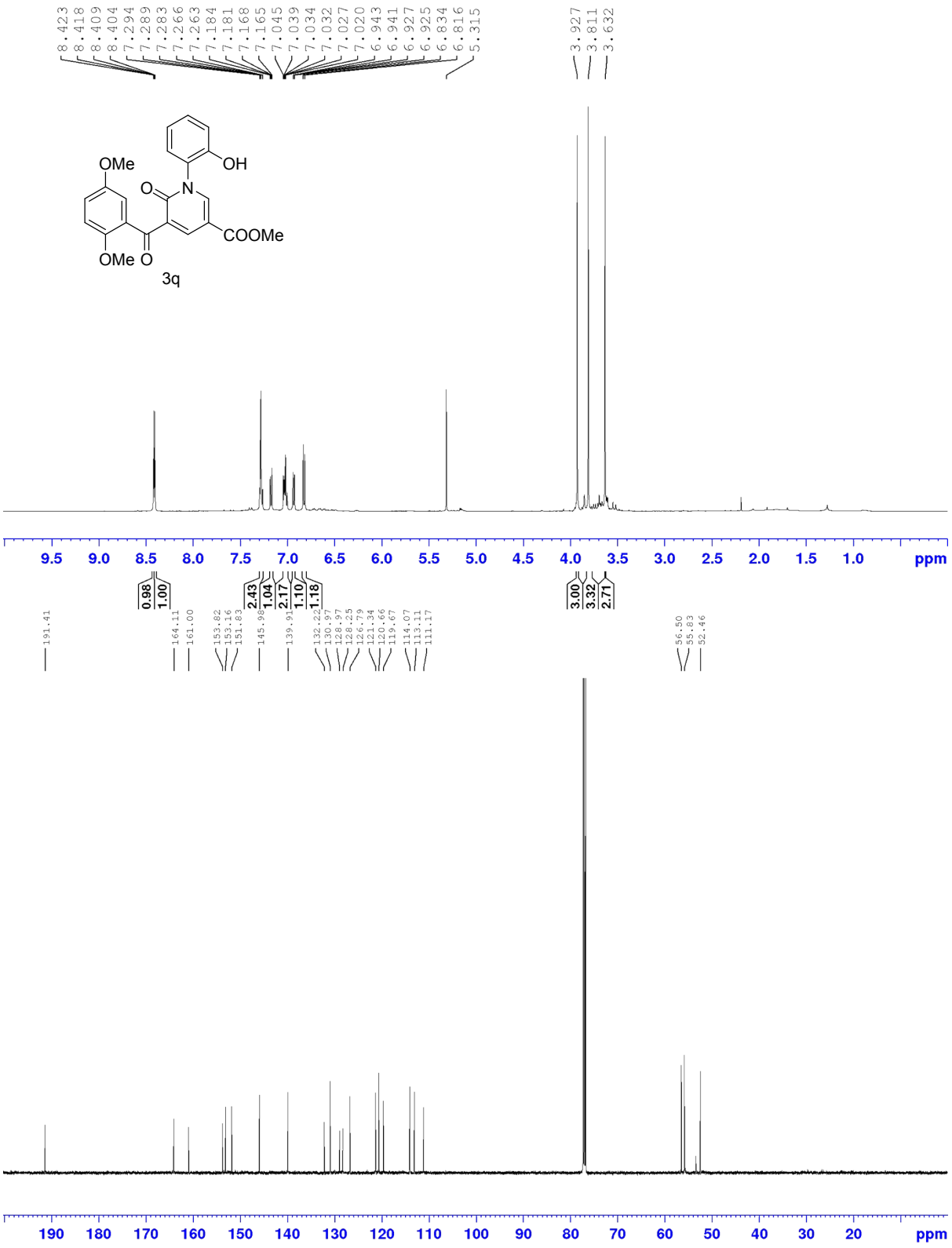
3n

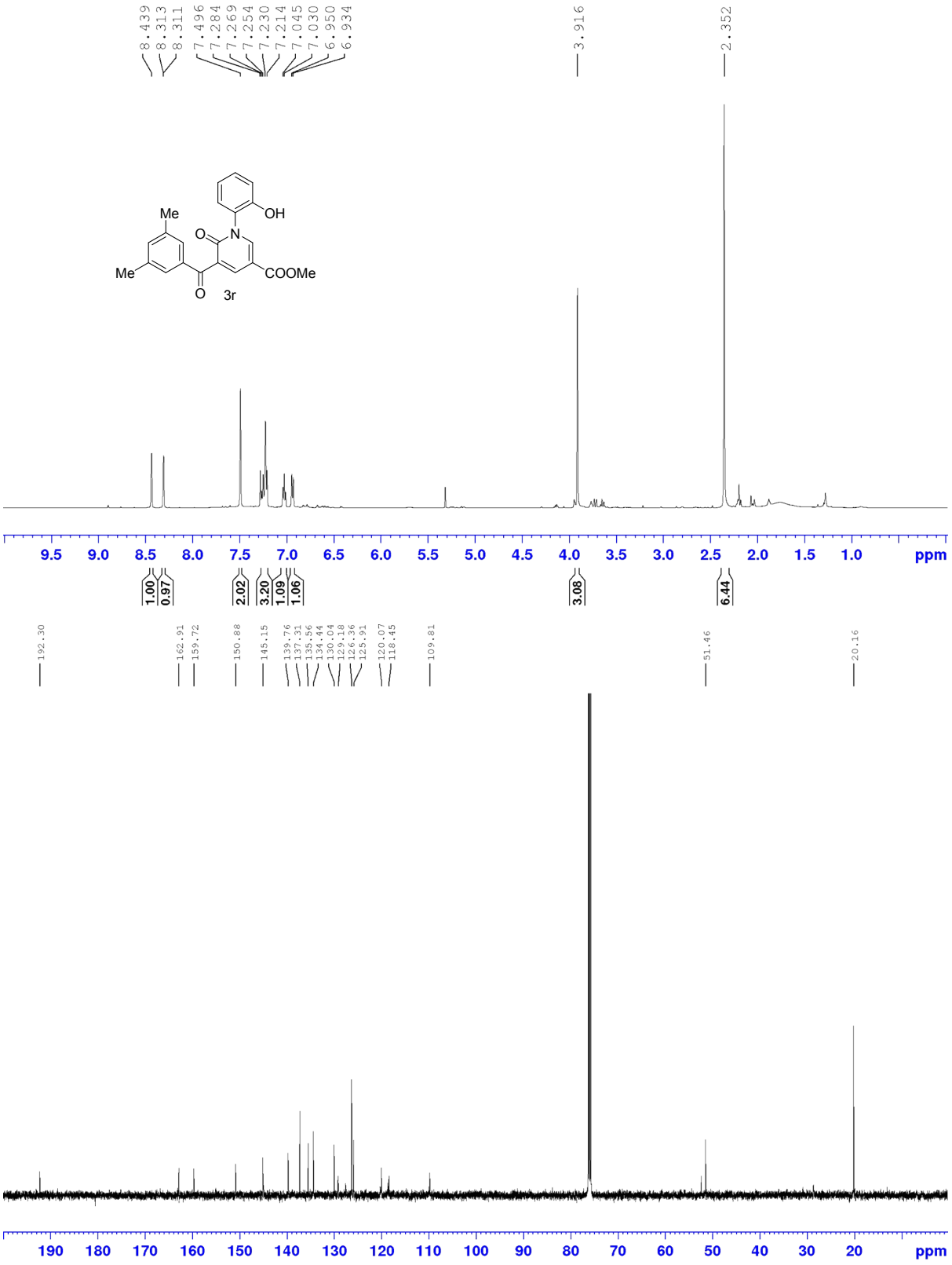

8.364
8.360
8.259
8.254
7.560
7.543
7.399
7.394
7.390
7.387
7.383
7.273
7.259
7.244
7.242
7.184
7.170
7.168
7.094
7.077
7.073
7.056
6.962
6.947
6.937
6.921
3.894
3.854
OH
F
O
N
MeO
COOMe
O
3o
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00 0.97 1.08 1.14 1.47 1.09 1.16 2.33 3.24 3.32
191.97
164.13
160.19
156.86
154.82
151.83
147.98
147.89
146.90
141.20
133.15
133.13
131.05
129.36
127.45
124.12
124.05
120.45
117.82
115.93
115.77
113.76
113.74
109.88
56.24
52.44
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


OH
F
O
N
MeO
COOMe
O
-128.42
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
ppm

3o


8.381
8.376
8.360
8.355
7.336
7.333
7.318
7.305
7.302
7.293
7.240
7.233
7.192
7.138
7.135
7.122
7.119
7.042
7.036
7.026
7.021
7.005
7.003
6.994
6.987
6.976
6.969
6.784
6.765
6.684
3.857
3.750
3.562
1.489
OH
MeO
O
N
COOMe
OMe O
3p
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 ppm
0.82 0.79 0.97 0.90 1.03 2.19 1.45 0.98 3.04 2.74 2.74
191.25
164.05
161.12
153.87
153.11
151.78
145.76
139.85
132.29
131.04
129.62
128.22
122.63
121.82
120.78
120.30
114.03
113.14
111.51
56.56
55.84
52.54
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


8.423
8.418
8.409
8.404
7.294
7.289
7.283
7.266
7.263
7.184
7.181
7.168
7.165
7.045
7.039
7.034
7.032
7.027
7.020
6.943
6.941
6.927
6.925
6.834
6.816
5.315
3.927
3.811
3.632
OMe
OH
O
N
COOMe
OMe O
3q
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 ppm
0.98
1.00
2.43
1.04
2.17
1.10
1.18
3.00
3.32
2.71
191.41
164.11
161.00
153.82
153.16
151.83
145.98
139.91
132.22
130.97
128.97
128.25
126.79
121.34
120.66
119.67
114.07
113.11
111.17
56.50
55.83
52.46
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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8.313
8.311
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7.269
7.254
7.230
7.214
7.045
7.030
6.950
6.934
3.916
2.352
Me
OH
N
O
Me
COOMe
O
3r
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 ppm
1.00
0.97
2.02
3.20
1.09
1.06
3.08
6.44
192.30
162.91
159.72
150.88
145.15
139.76
137.31
135.56
134.44
130.04
129.18
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109.81
51.46
20.16
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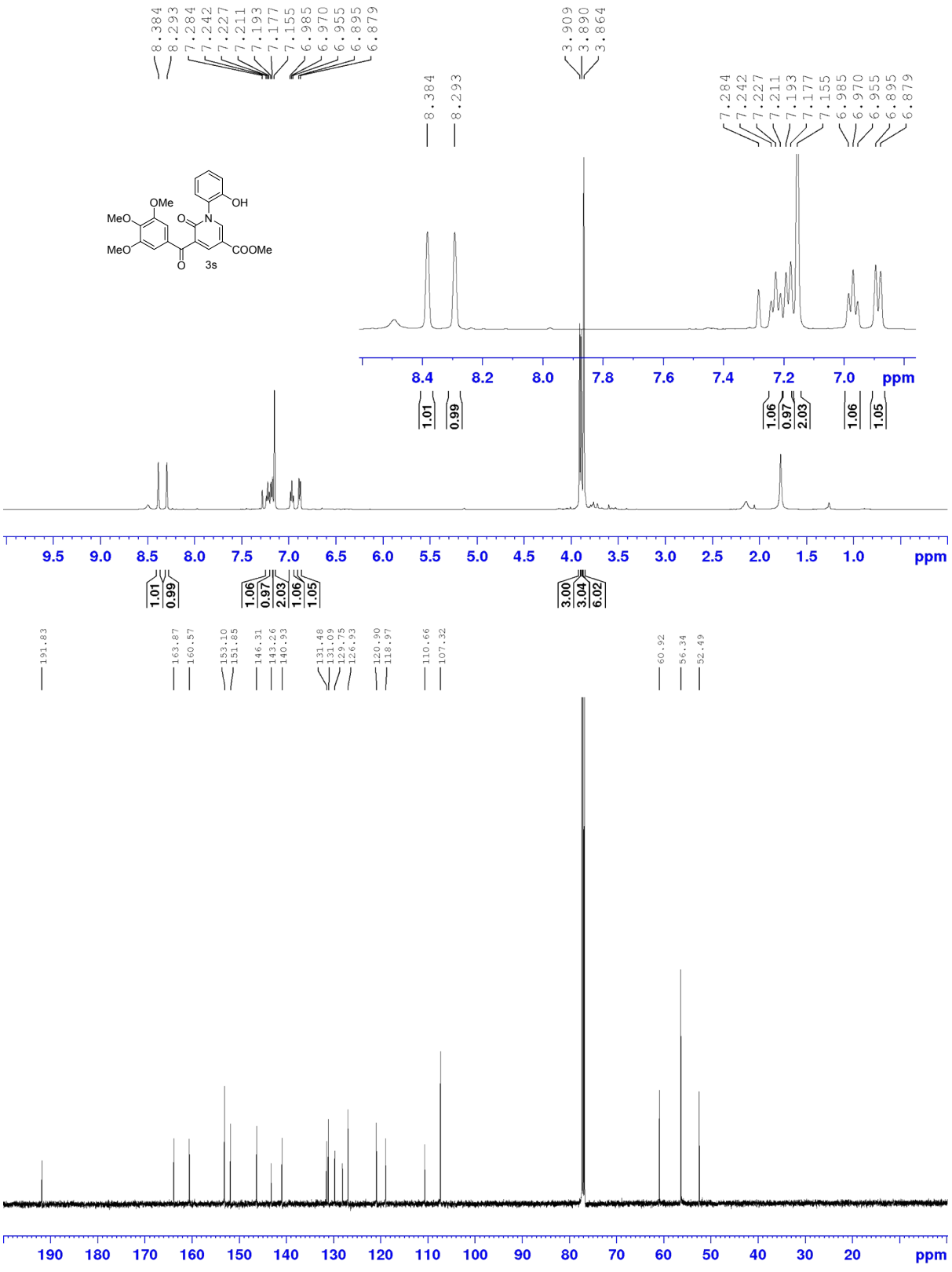
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7.284
7.242
7.227
7.211
7.193
7.177
7.155
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6.970
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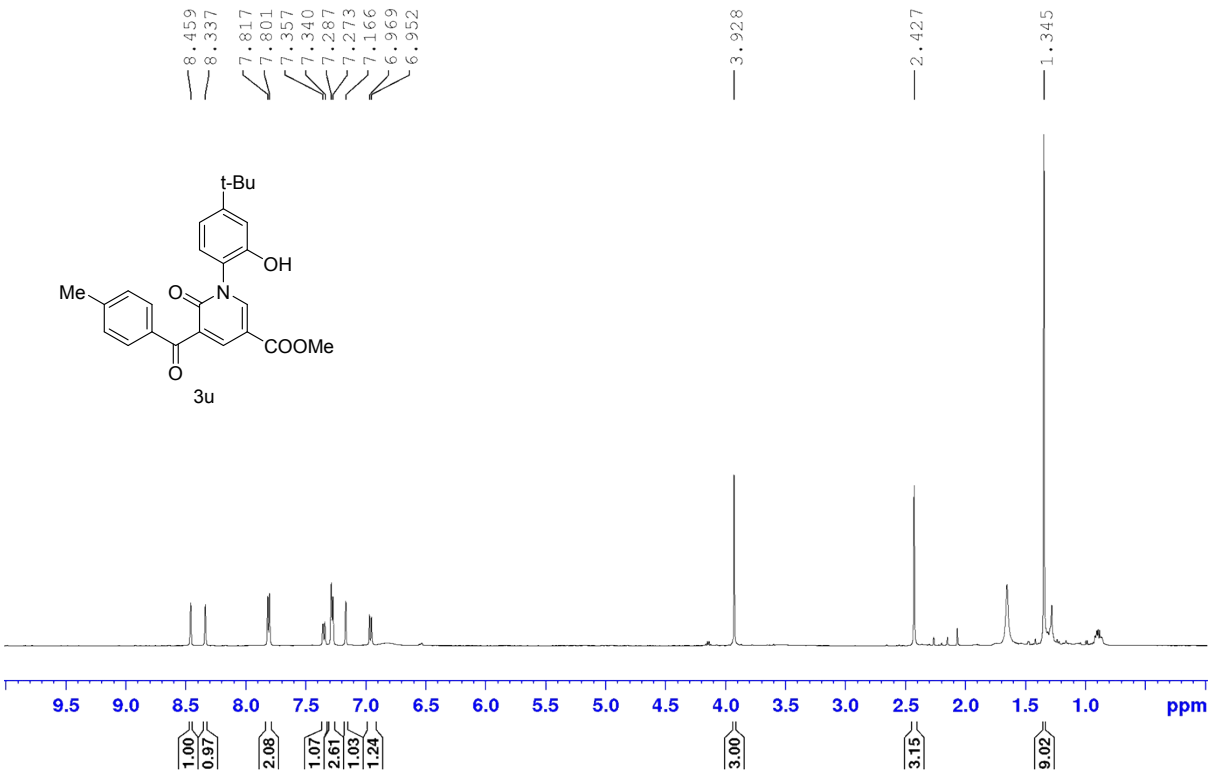
6.895
6.879
3.909
3.890
3.864
OMe
OH
MeO
O
N
MeO
COOMe
O
3s
1.01
0.99
1.06
0.97
2.03
1.06
1.05
3.00
3.04
6.02
ppm
191.83
163.87
160.57
153.10
151.85
146.31
143.26
140.93
131.48
131.09
129.75
126.93
120.90
118.97
110.66
107.32
60.92
56.34
52.49


8.393
8.310
7.668
7.652
7.530
7.514
7.281
7.264
7.191
7.076
6.867
6.850
3.847
1.254
t-Bu
OH
Br
O
N
COOMe
O
3t
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 ppm
0.96
0.90
1.94
1.90
1.00
0.96
1.01
3.00
8.61
191.96
163.79
160.77
149.12
146.65
144.95
141.71
135.34
131.94
130.96
129.17
128.76
128.32
128.10
123.39
119.25
111.15
52.61
34.34
31.44
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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7.817
7.801
7.357
7.340
7.287
7.273
7.166
6.969
6.952
3.928
2.427
1.345
t-Bu
OH
Me
O
N
COOMe
O
3u
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 ppm
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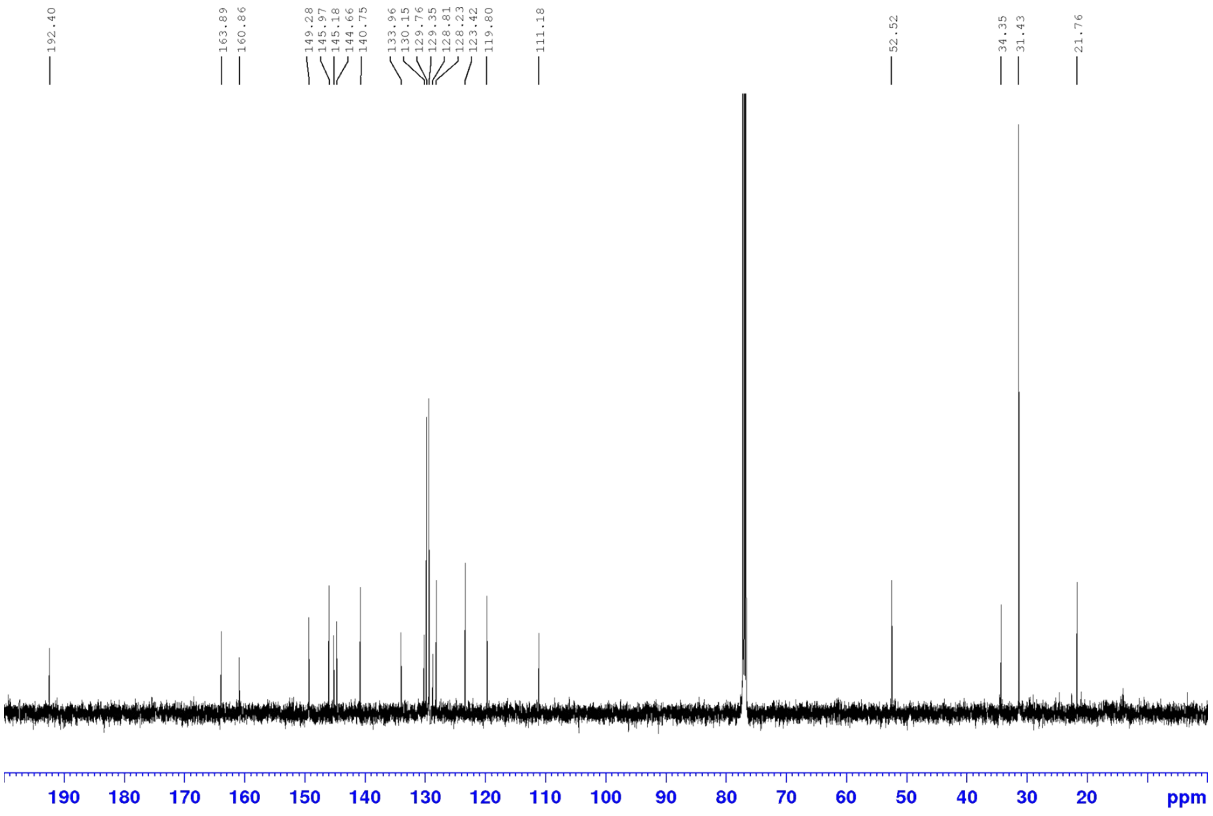
0.97
2.08
1.07
2.61
1.03
1.24
3.00
3.15
9.02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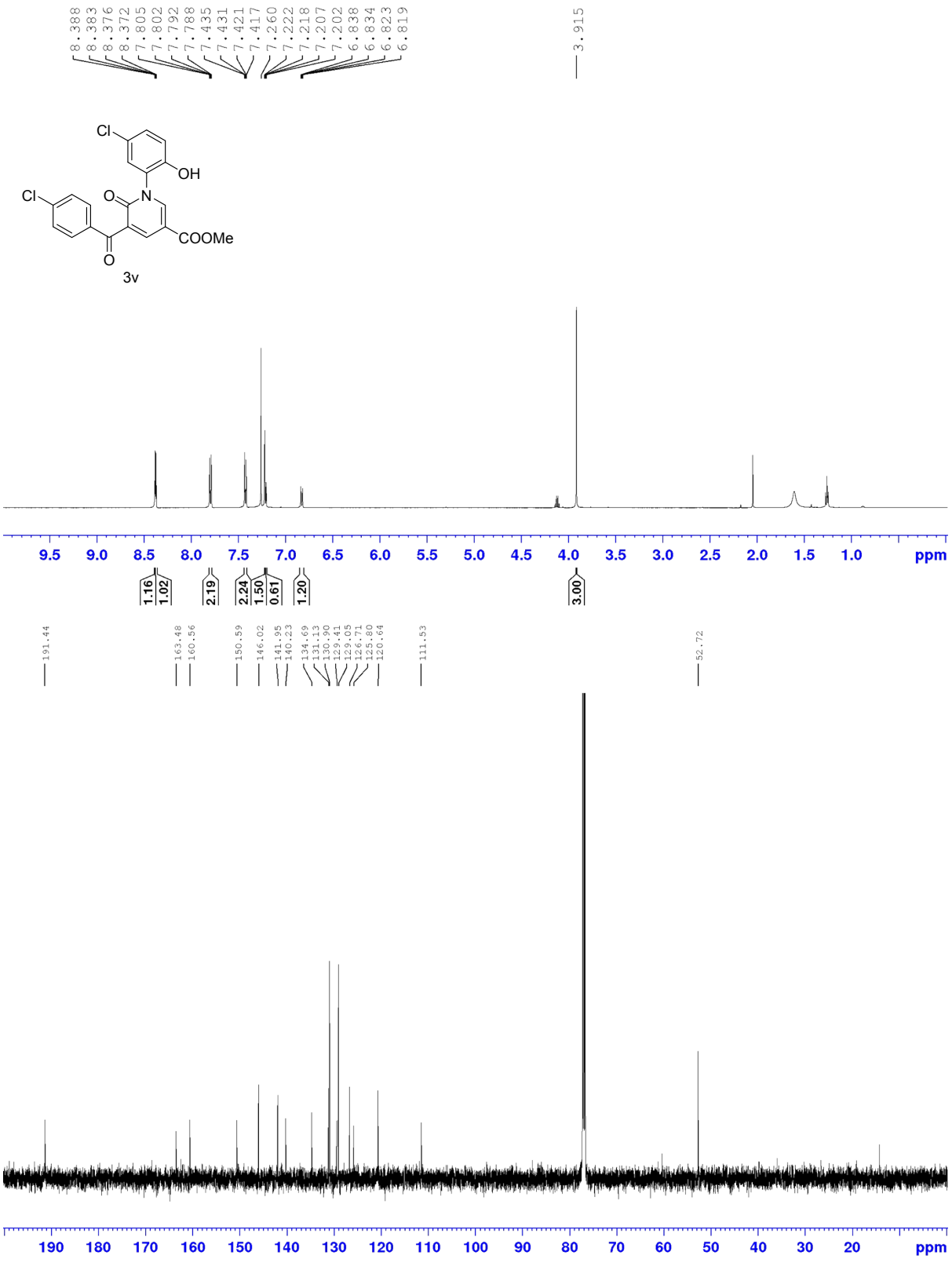
163.89
160.86
149.28
145.97
145.18
144.66
140.75
133.96
130.15
129.76
129.35
128.81
128.23
123.42
119.80
111.18
52.52
34.35
31.43
21.76
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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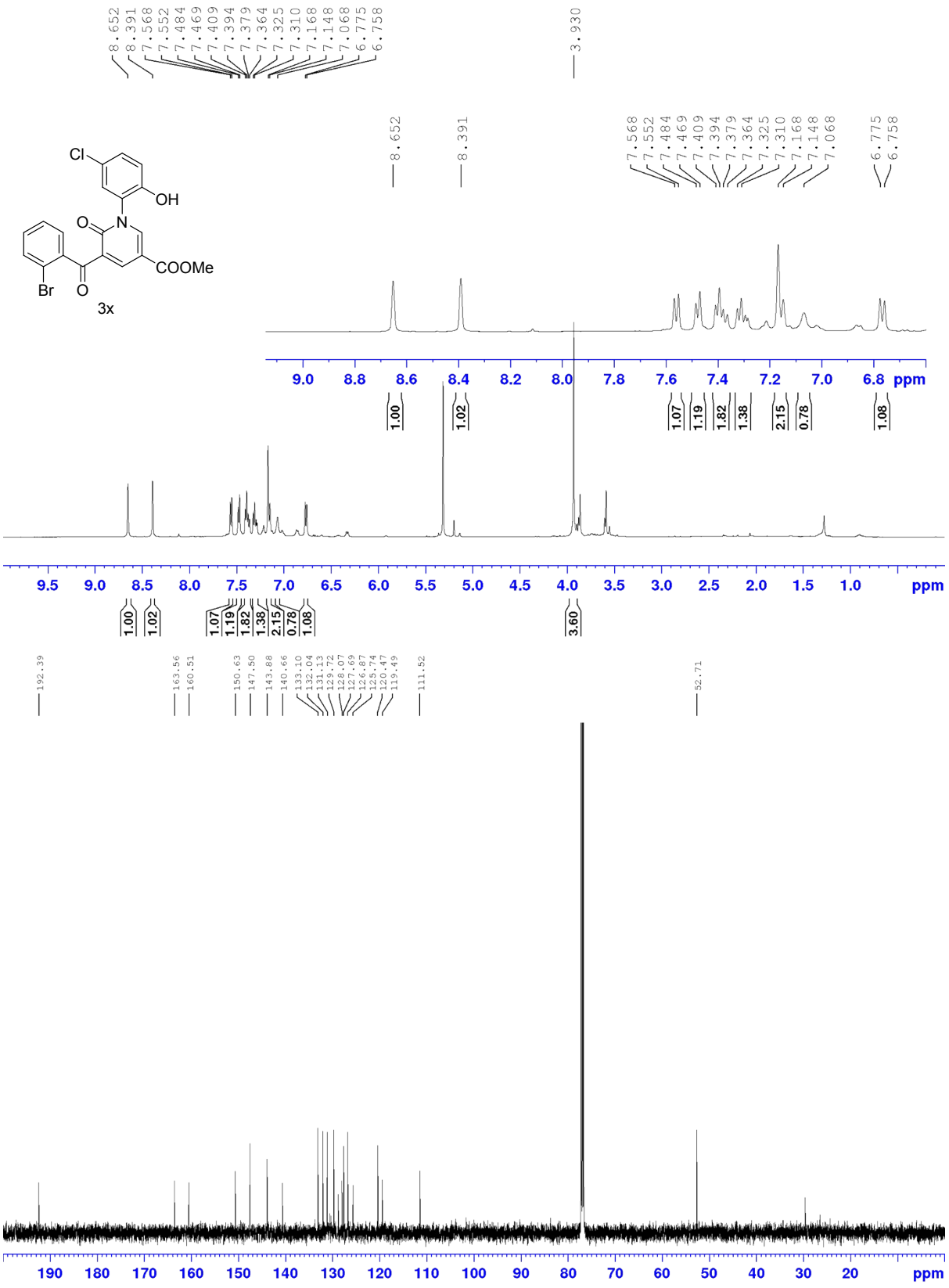
8.376
8.372
7.805
7.802
7.792
7.788
7.435
7.431
7.421
7.417
7.260
7.222
7.218
7.207
7.202
6.838
6.834
6.823
6.819
3.915
Cl
OH
Cl
O
N
COOMe
O
3v
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.16
1.02
2.19
2.24
1.50
0.61
1.20
3.00
191.44
163.48
160.56
150.59
146.02
141.95
140.23
134.69
131.13
130.90
129.41
129.05
126.71
125.60
120.64
111.53
52.72
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


8.308
7.802
7.786
7.280
7.263
7.190
7.087
7.070
6.726
6.708
3.895
2.412
Cl
OH
Me
O
N
COOMe
O
3w
ppm
1.76
1.84
2.20
0.84
1.07
0.96
3.00
2.85
192.33
163.69
160.56
150.78
145.86
144.86
141.24
133.79
130.96
130.01
129.87
129.41
128.39
126.85
125.04
119.95
110.97
52.55
21.76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7.568
7.552
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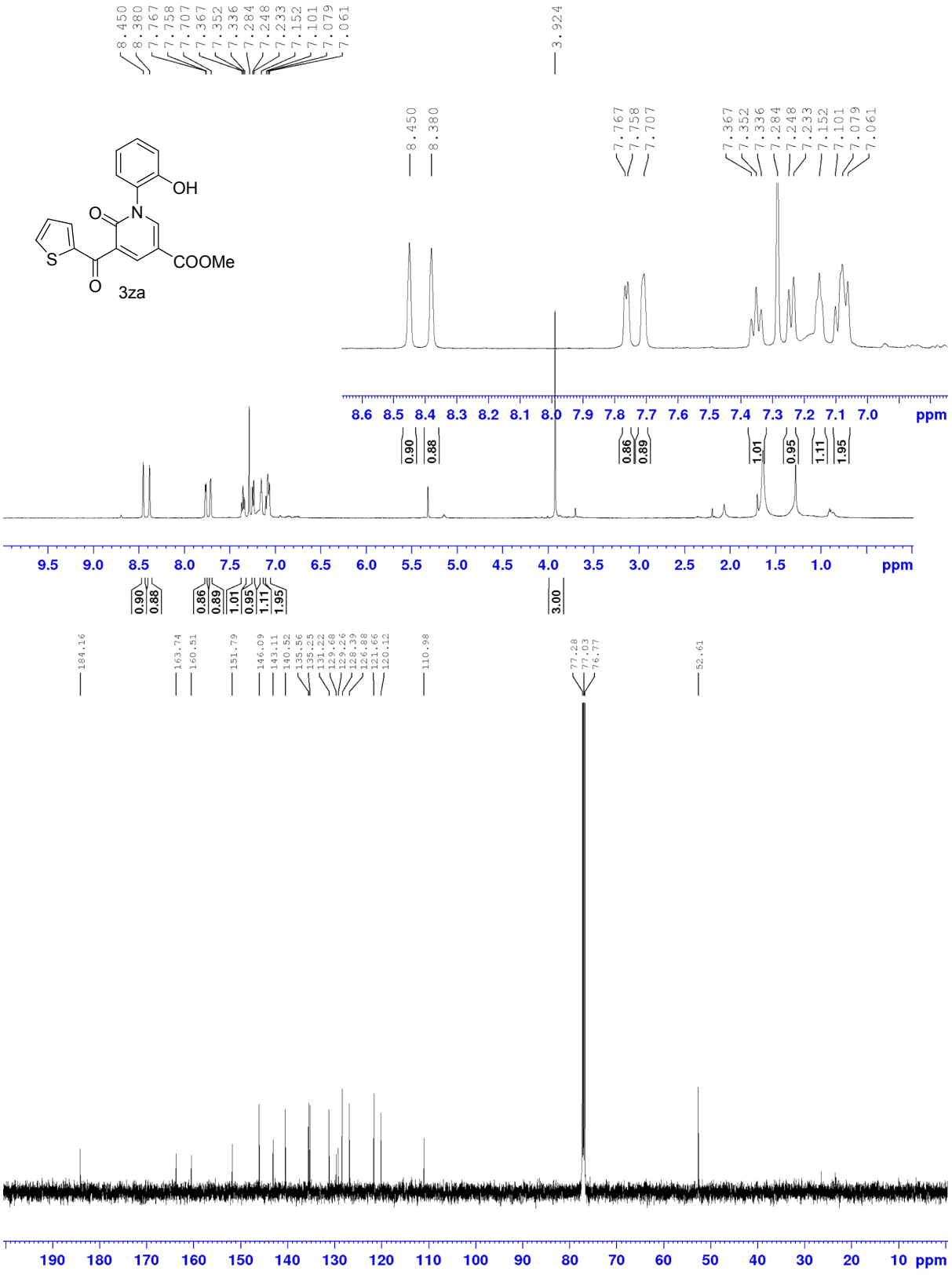
7.469
7.409
7.394
7.379
7.364
7.325
7.310
7.168
7.148
7.068
6.775
6.758
3.930
Cl
OH
O
N
COOMe
Br
O
3x
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
ppm
1.00
1.02
1.07
1.19
1.82
1.38
2.15
0.78
1.08
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.00
1.02
1.07
1.19
1.82
1.38
2.15
0.78
1.08
3.60
192.39
163.56
160.51
150.63
147.50
143.88
140.66
133.10
132.04
131.13
129.72
128.07
127.69
126.87
125.74
120.47
119.49
111.52
52.71
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


8.544
8.539
8.289
8.284
7.456
7.440
7.384
7.381
7.369
7.366
7.301
7.286
7.271
7.213
7.210
7.197
7.195
7.002
6.985
6.849
6.845
6.832
6.828
6.665
6.661
3.828
Cl
OH
N
O
COOMe
Br
O
3y
ppm
1.00
0.95
1.10
1.09
1.18
1.18
1.06
0.99
0.94
3.05
192.56
163.76
160.59
152.39
147.97
143.86
140.62
136.43
133.10
131.94
129.65
127.86
127.66
121.07
119.47
118.73
111.01
52.61


8.427
8.422
8.397
8.392
7.665
7.354
7.336
7.329
7.284
7.250
7.247
7.233
7.231
7.071
7.065
7.063
7.057
7.049
7.047
7.043
6.588
6.585
6.581
6.578
3.909
OH
N
O
O
O
COOMe
3z
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 ppm
0.91
0.86
0.95
1.91
0.92
1.92
0.97
3.00
179.25
163.74
160.42
152.02
151.77
147.64
146.35
141.12
131.20
129.24
128.94
126.94
121.69
120.55
120.06
112.68
110.97
52.56
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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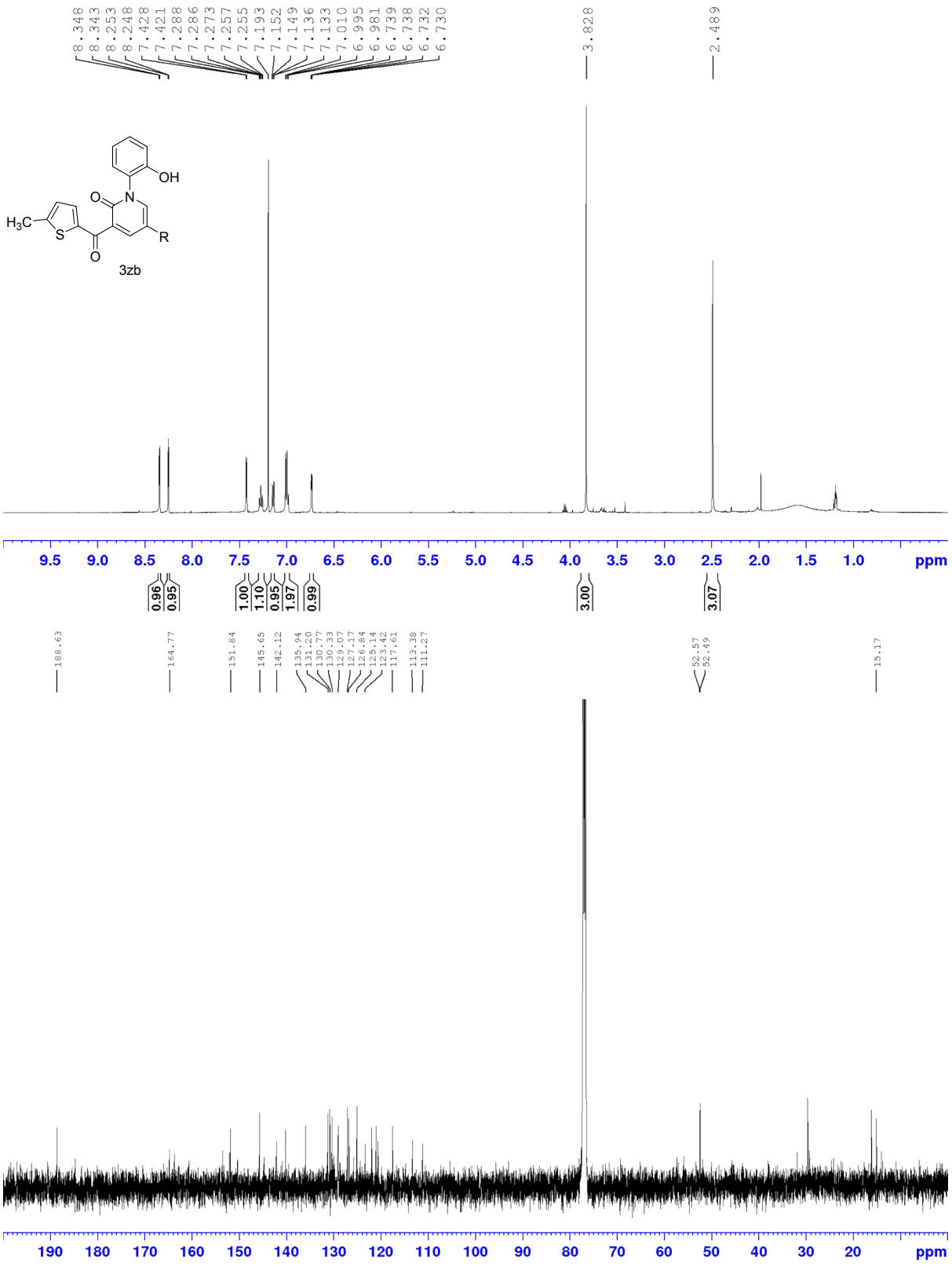
7.707
7.367
7.352
7.336
7.284
7.248
7.233
7.152
7.101
7.079
7.061
3.924
OH
N
O
S
O
COOMe
3za
0.90
0.88
0.86
0.89
1.01
0.95
1.11
1.95
3.00
ppm
184.16
163.74
160.51
151.79
146.09
143.11
140.52
135.56
135.25
131.22
129.68
129.26
128.39
126.88
121.66
120.12
110.98
77.28
77.03
76.77
52.61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8.343
8.253
8.248
7.428
7.421
7.288
7.286
7.273
7.257
7.255
7.193
7.152
7.149
7.136
7.133
7.010
6.995
6.981
6.739
6.738
6.732
6.730
3.828
2.489
OH
O
N
H3C
S
R
O
3zb
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 ppm
0.96
0.95
1.00
1.10
0.95
1.97
0.99
3.00
3.07
188.63
164.77
151.84
145.65
142.12
135.94
131.20
130.77
130.33
129.07
127.17
126.84
125.14
123.42
117.61
113.38
111.27
52.57
52.49
15.17
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


11.330
8.137
7.284
7.016
7.001
6.845
6.829
6.472
6.455
6.231
5.269
3.853
3.511
2.254
1.343
Me
OH
O
N
N
H
MeOOC
COOMe
4a
ppm
1.05
0.96
3.19
1.12
1.90
0.90
0.97
2.95
3.00
3.21
9.13
172.52
166.38
166.25
159.84
154.81
140.00
136.45
131.46
131.31
129.72
129.57
118.71
117.80
106.77
106.07
63.73
52.54
51.19
28.40
21.45
ppm


11.190
8.090
7.261
7.012
7.007
6.995
6.991
6.982
6.978
6.879
6.873
6.811
6.794
6.476
6.472
6.459
6.446
6.443
6.263
5.250
3.841
3.492
1.324
F
OH
O
N
H
N
MeOOC
COOMe
4b
ppm
1.10
1.12
1.42
1.89
1.64
2.15
1.18
1.08
3.34
3.00
9.47
172.46
166.31
166.07
162.47
160.27
158.64
154.85
136.12
131.15
130.38
130.04
129.46
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106.65
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76.76
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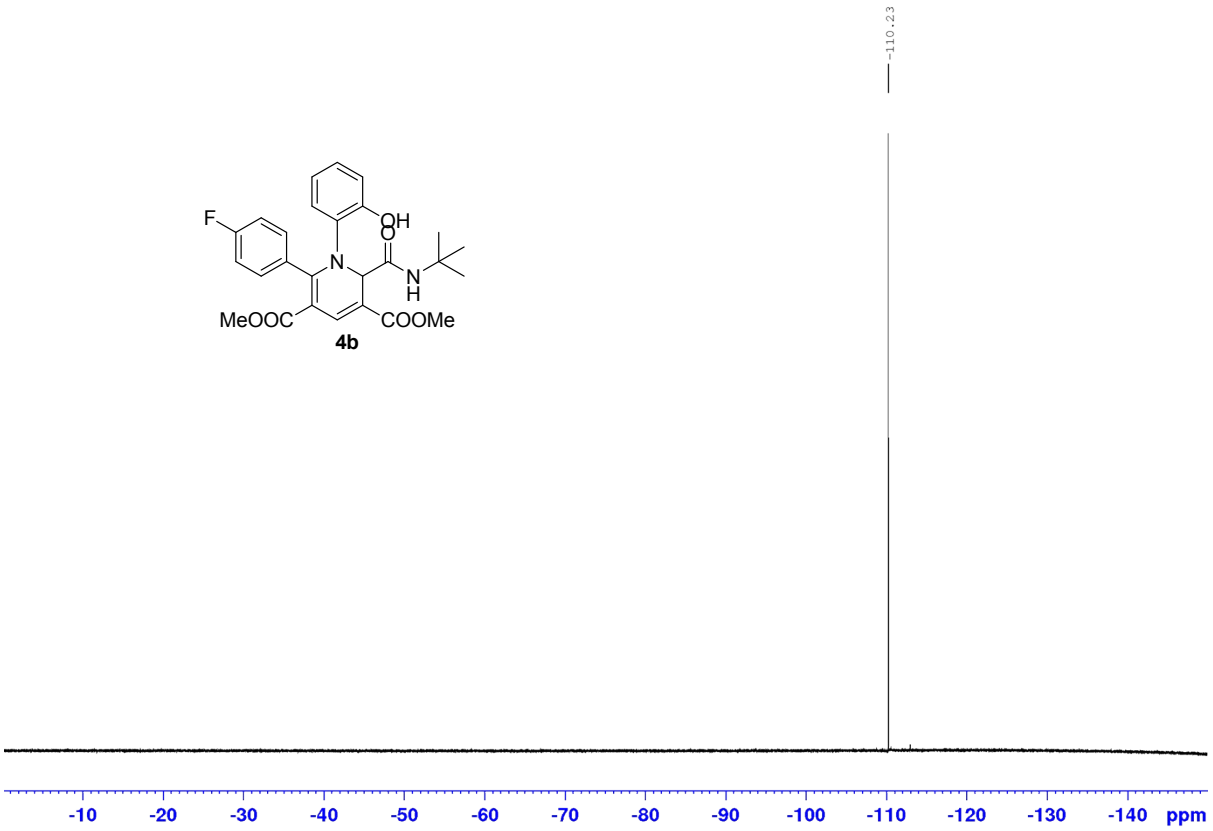
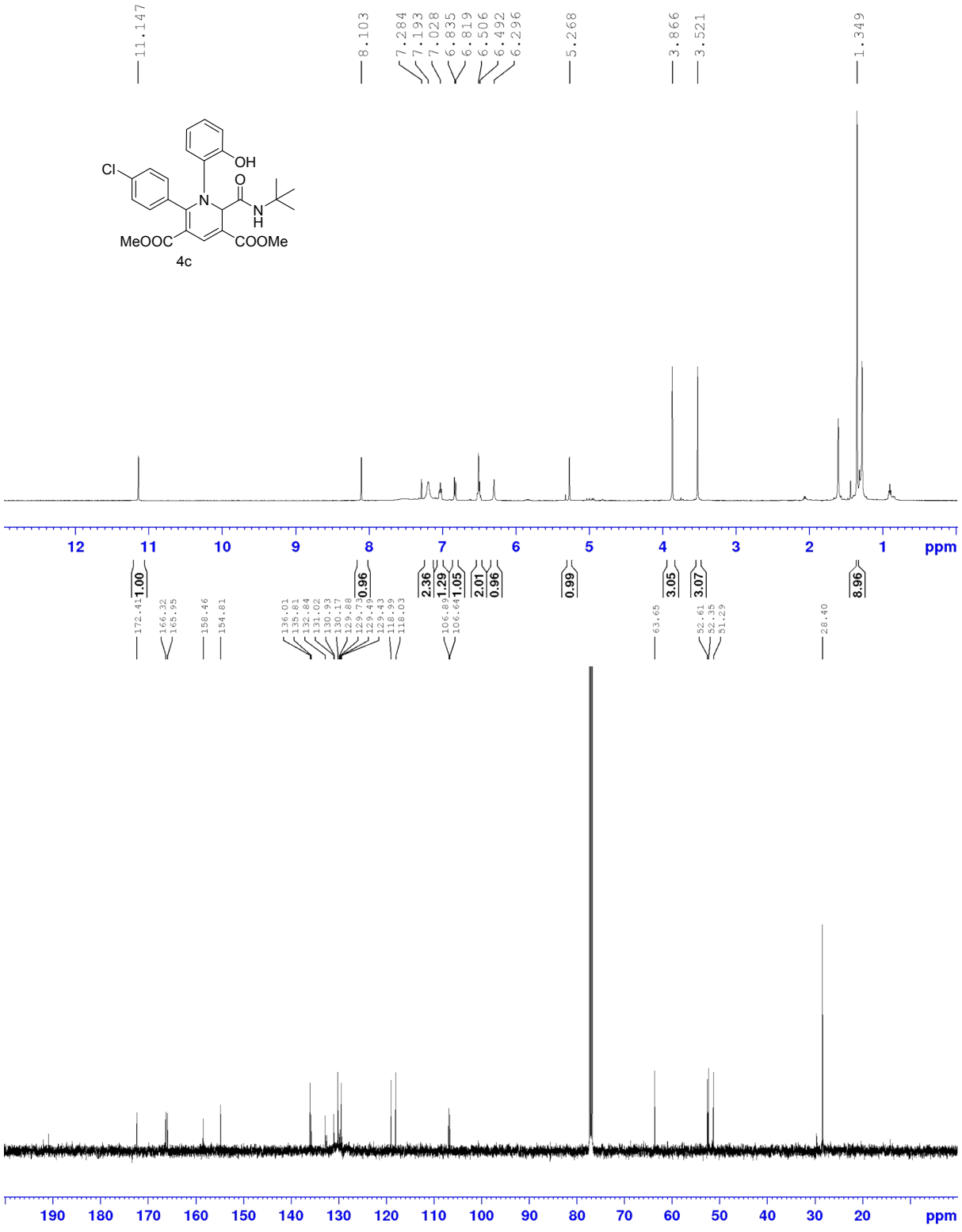
52.29
51.22
28.40


-110.23
F
OH
O
N
N
H
MeOOC
COOMe
4b
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
ppm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7.193
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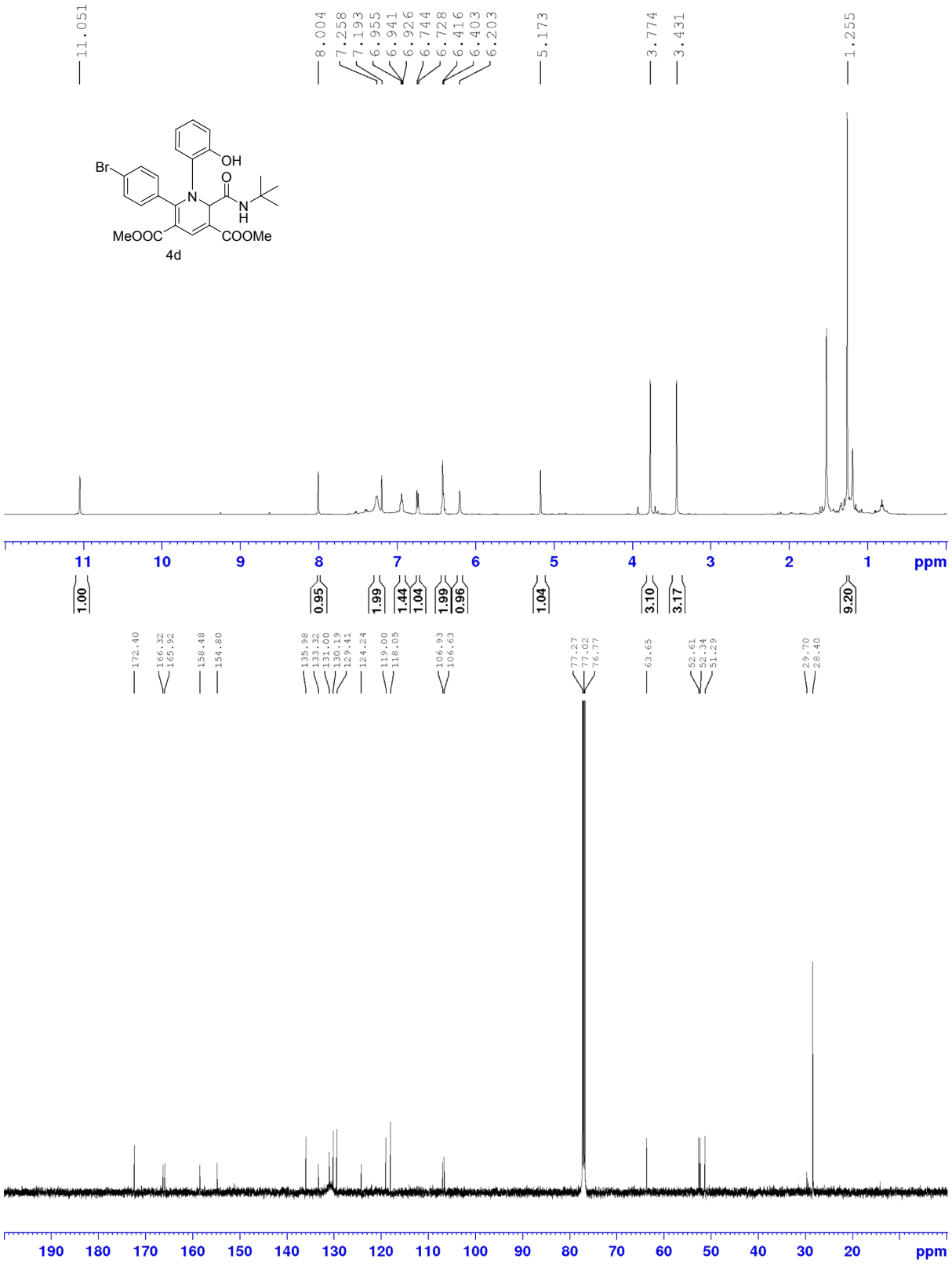
6.835
6.819
6.506
6.492
6.296
5.268
3.866
3.521
1.349
Cl
OH
O
N
N
H
MeOOC
COOMe
4c
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
0.96
2.36
1.29
1.05
2.01
0.96
0.99
3.05
3.07
8.96
172.41
166.32
165.95
158.46
154.81
136.01
135.81
132.84
131.02
130.93
130.17
129.88
129.73
129.49
129.43
118.99
118.03
106.89
106.64
63.65
52.61
52.35
51.29
28.40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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6.416
6.403
6.203
5.173
3.774
3.431
1.255
Br
OH
O
N
N
H
MeOOC
COOMe
4d
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
0.95
1.99
1.44
1.04
1.99
0.96
1.04
3.10
3.17
9.20
172.40
166.32
165.92
158.48
154.80
135.98
133.32
131.00
130.19
129.41
124.24
119.00
118.05
106.93
106.63
77.27
77.02
76.77
63.65
52.61
52.34
51.29
29.70
28.40
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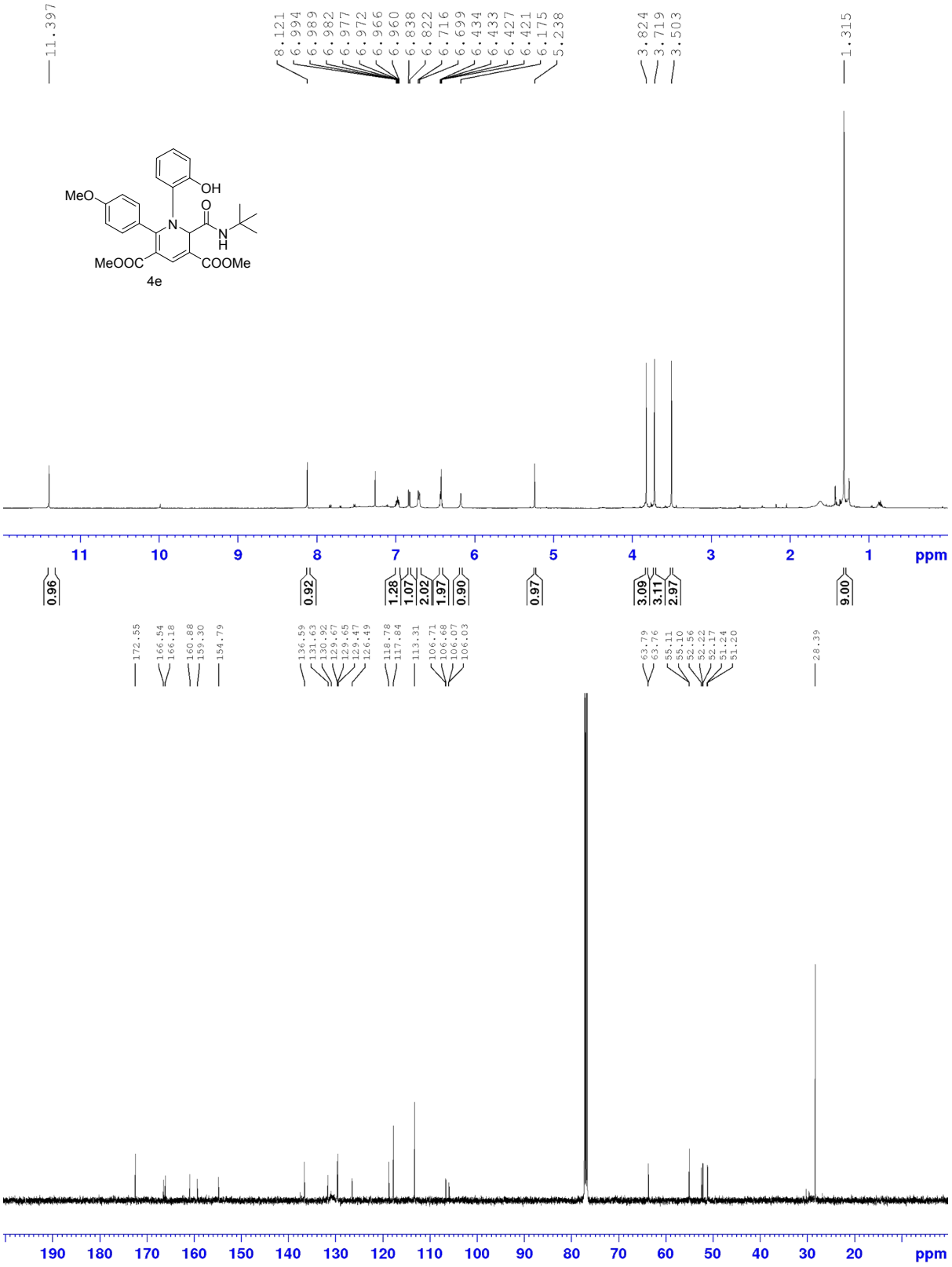
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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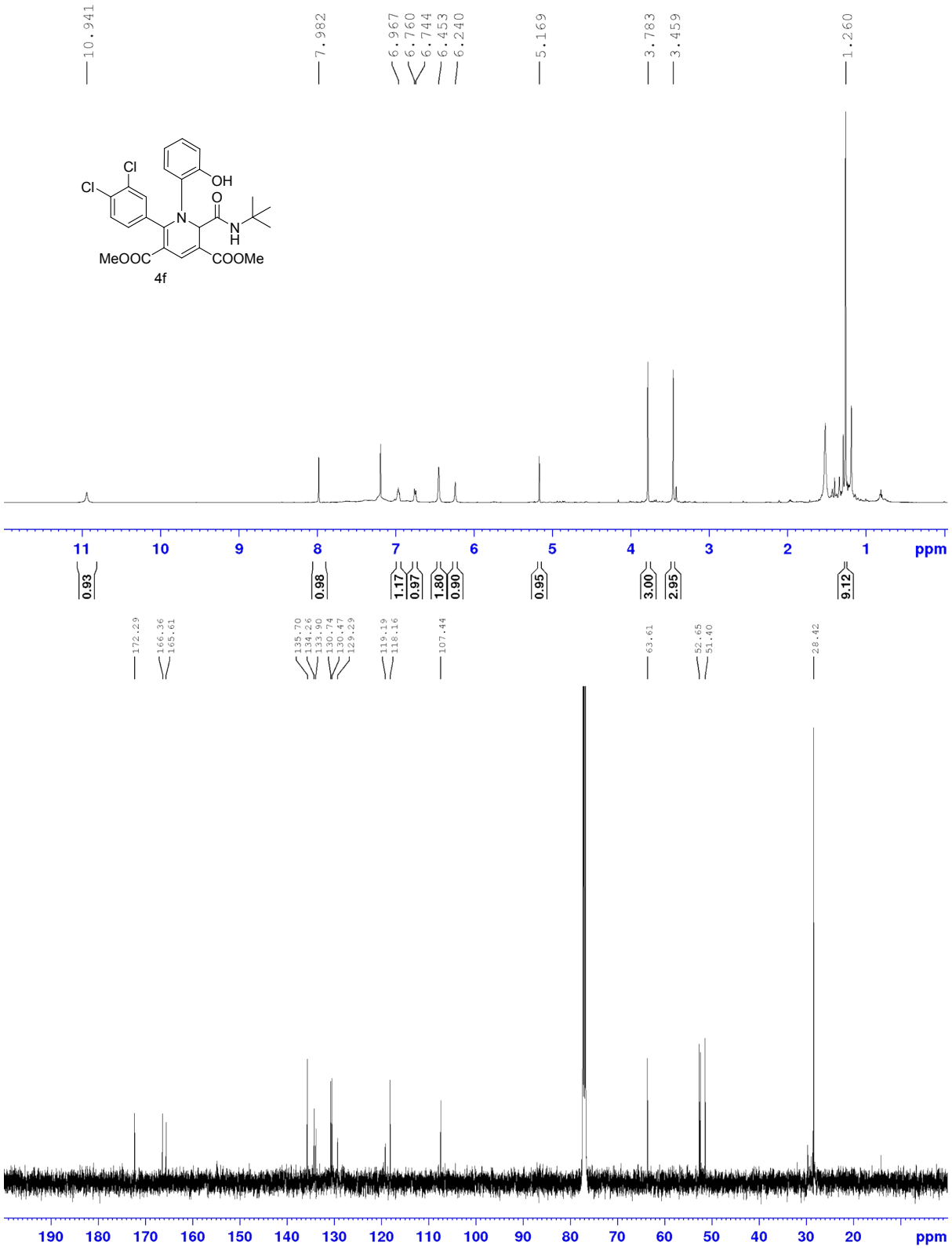
6.989
6.982
6.977
6.972
6.966
6.960
6.838
6.822
6.716
6.699
6.434
6.433
6.427
6.421
6.175
5.238
3.824
3.719
3.503
1.315
MeO
OH
O
N
N
H
MeOOC
COOMe
4e
11
10
9
8
7
6
5
4
3
2
1
ppm
0.96
0.92
1.28
1.07
2.02
1.97
0.90
0.97
3.09
3.11
2.97
9.00
172.55
166.54
166.18
160.88
159.30
154.79
136.59
131.63
130.92
129.67
129.65
129.47
126.49
118.78
117.84
113.31
106.71
106.68
106.07
106.03
63.79
63.76
55.11
55.10
52.56
52.22
52.17
51.24
51.20
28.39
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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6.760
6.744
6.453
6.240
5.169
3.783
3.459
1.260
Cl
Cl
OH
O
N
N
H
MeOOC
COOMe
4f
11
10
9
8
7
6
5
4
3
2
1
ppm
0.93
0.98
1.17
0.97
1.80
0.90
0.95
3.00
2.95
9.12
172.29
166.36
165.61
135.70
134.26
133.90
130.74
130.47
129.29
119.19
118.16
107.44
63.61
52.65
51.40
28.42
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180
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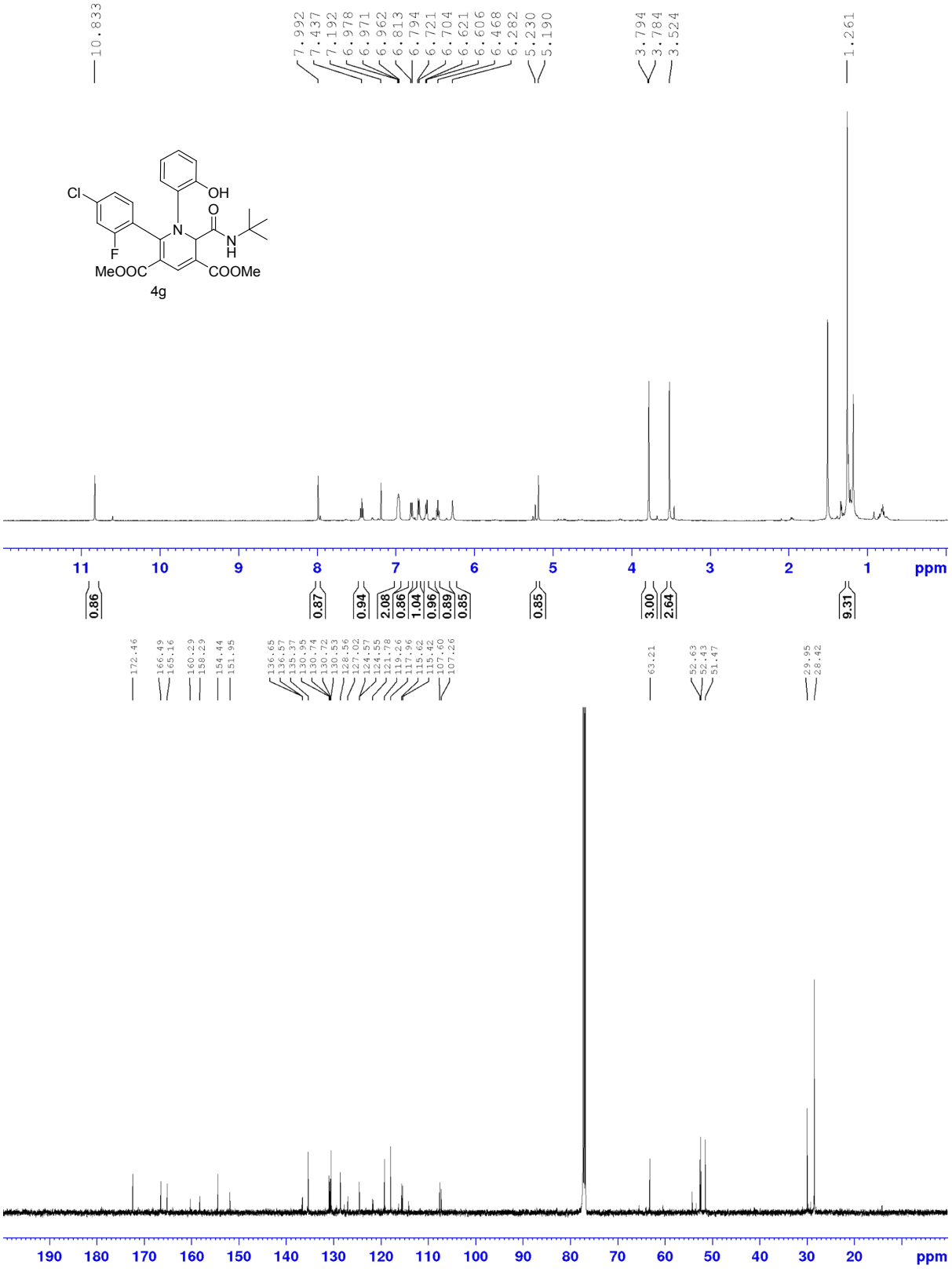
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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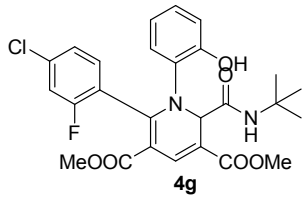
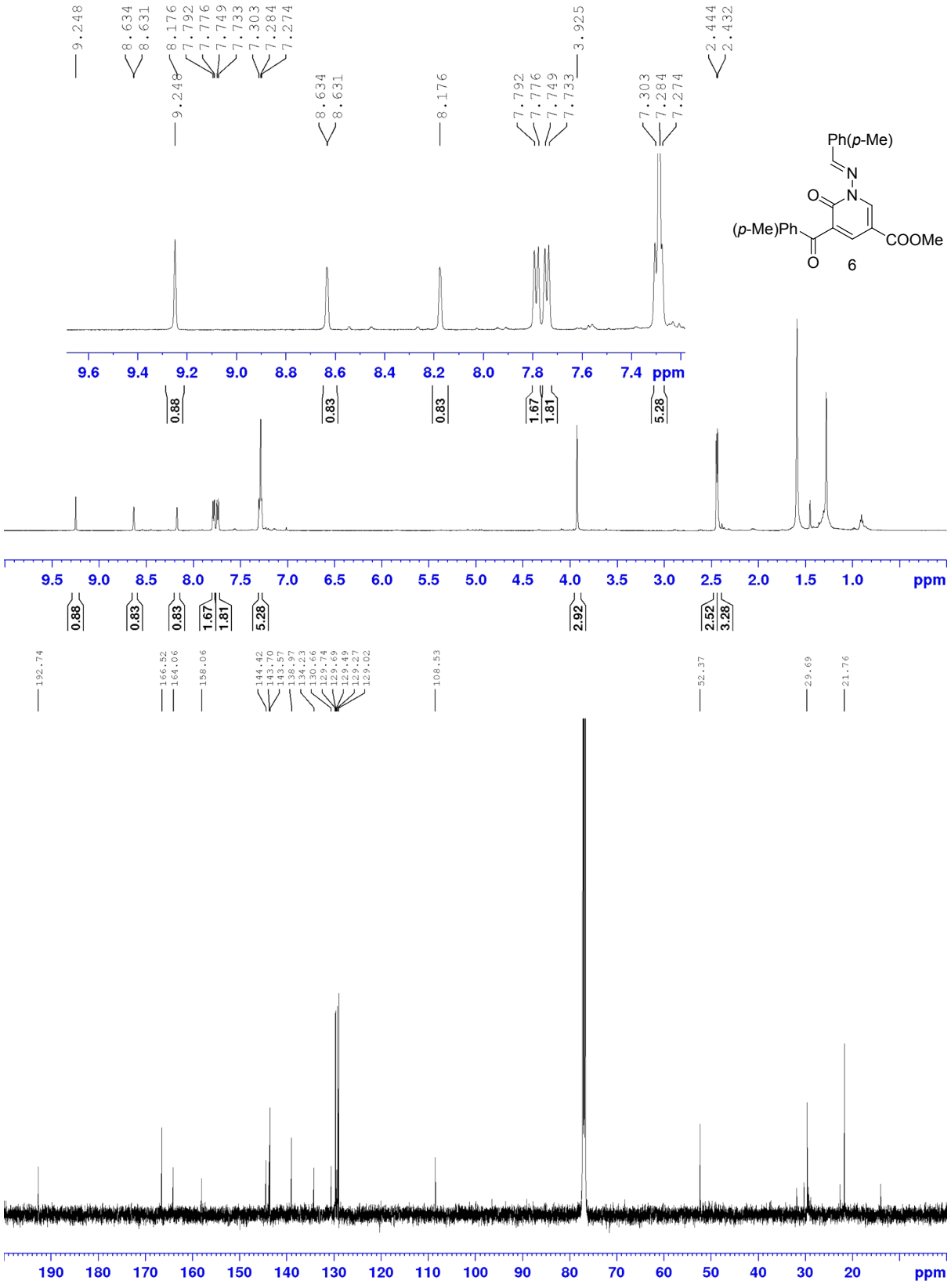
7.437
7.192
6.978
6.971
6.962
6.813
6.794
6.721
6.704
6.621
6.606
6.468
6.282
5.230
5.190
3.794
3.784
3.524
1.261
Cl
OH
O
N
N
H
F
MeOOC
COOMe
4g
11
10
9
8
7
6
5
4
3
2
1
ppm
0.86
0.87
0.94
2.08
0.86
1.04
0.96
0.89
0.85
0.85
3.00
2.64
9.31
172.46
166.49
165.16
160.29
158.29
154.44
151.95
136.65
136.57
135.37
130.95
130.74
130.72
130.53
128.56
127.02
124.57
124.55
121.78
119.26
117.96
115.62
115.42
107.60
107.26
63.21
52.63
52.43
51.47
29.95
28.42
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

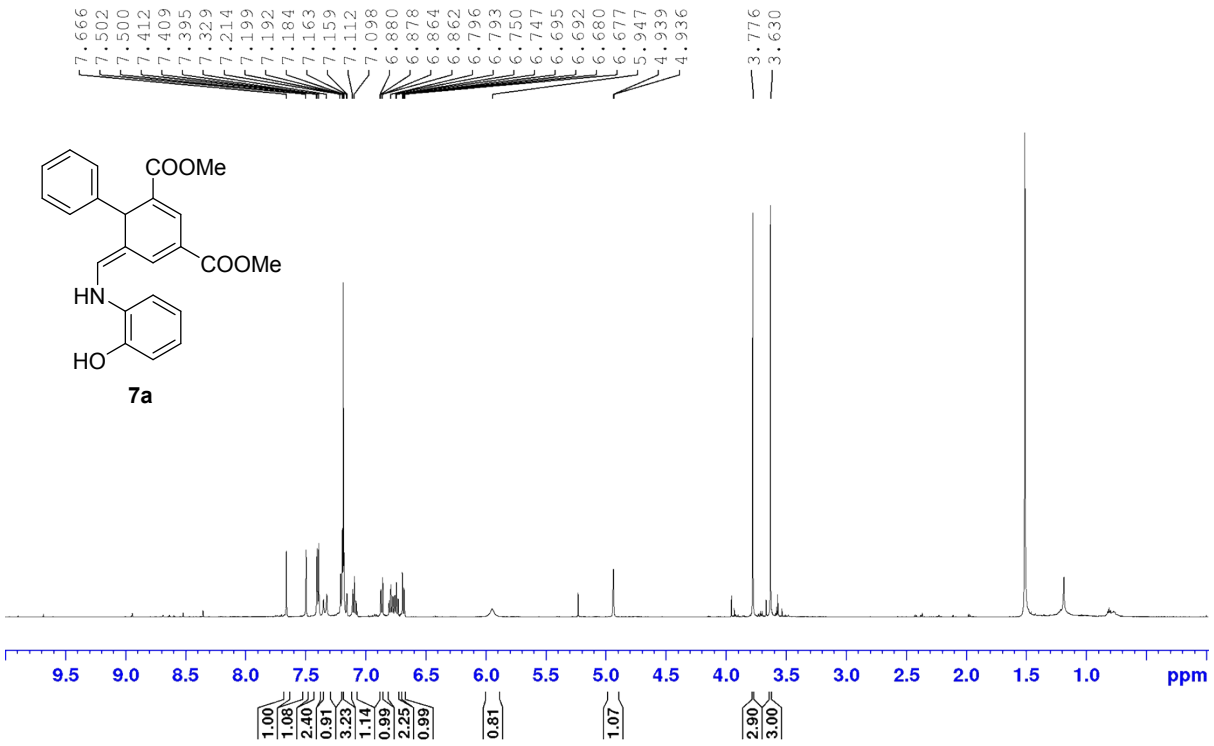

Cl
OH
O
N
N
H
F
MeOOC
COOMe

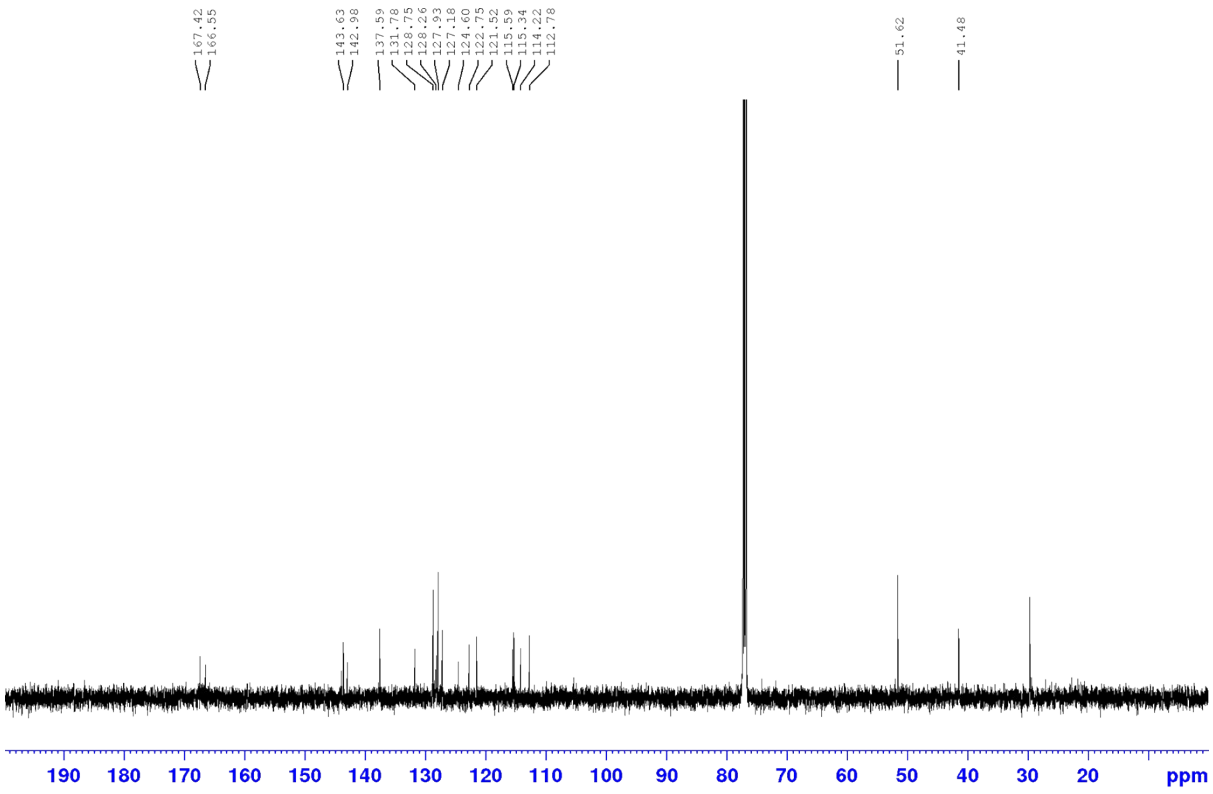
4g

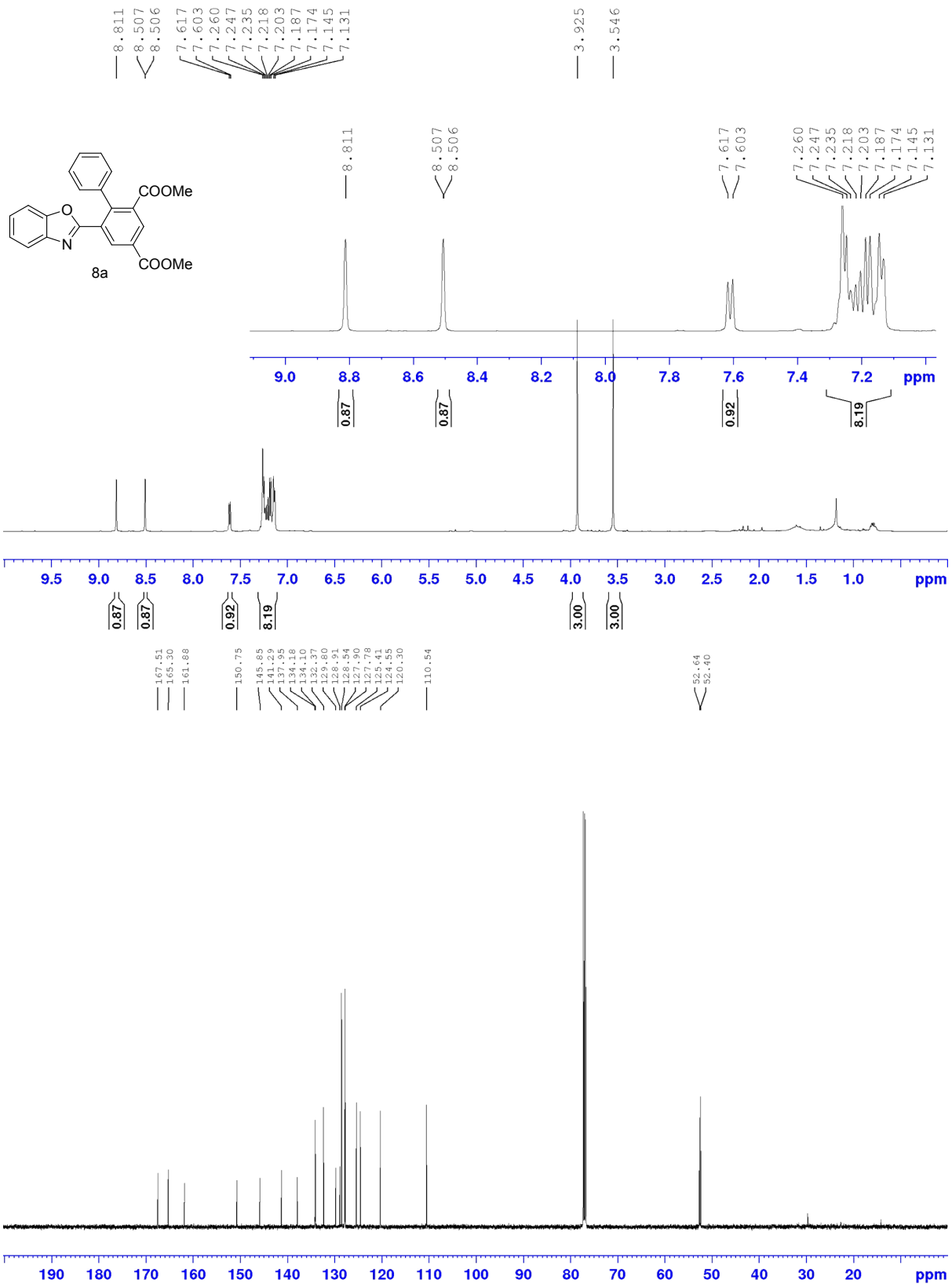
-111.99
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
ppm

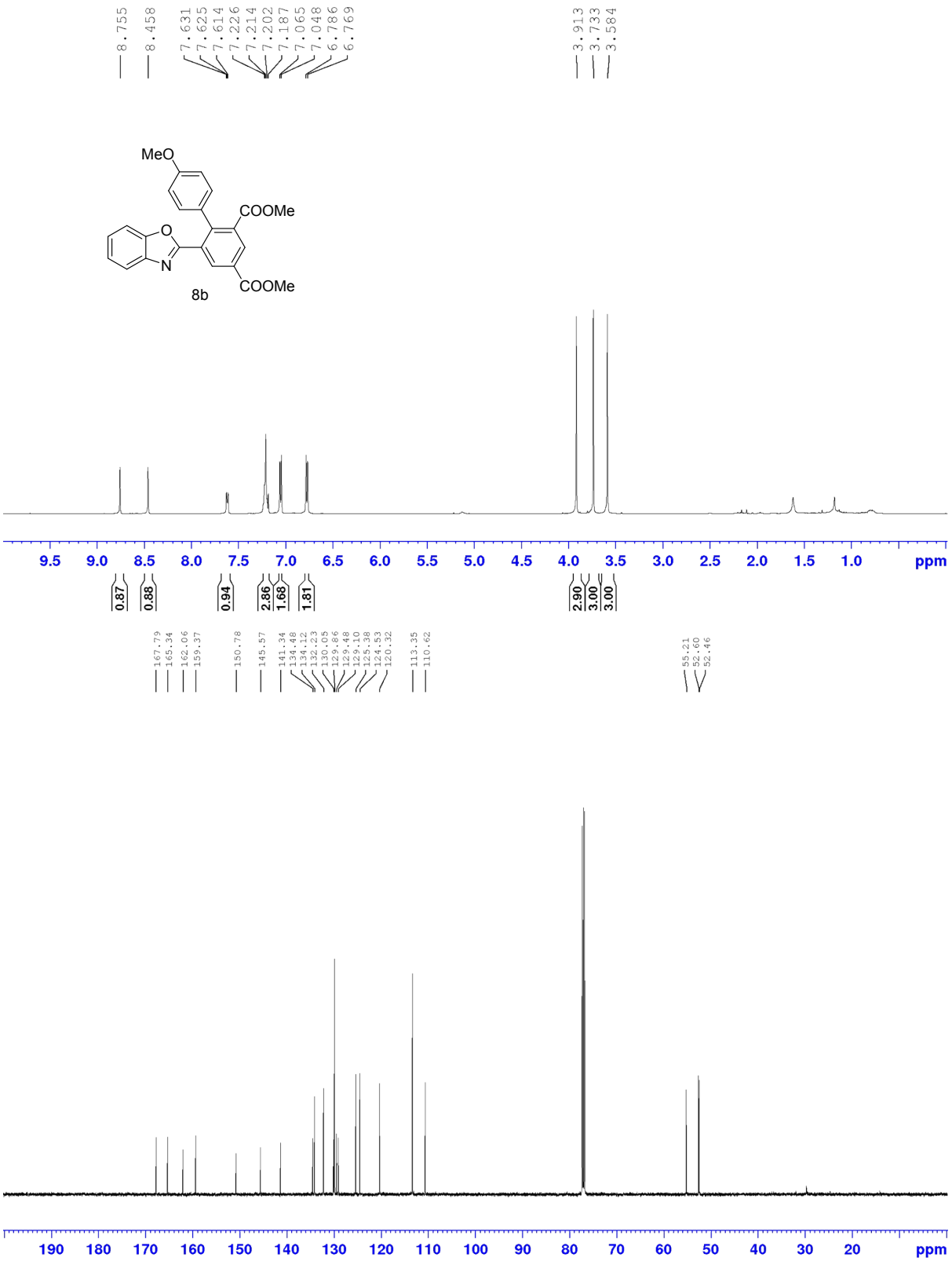

11.290
7.996
7.192
7.146
7.132
6.908
6.893
6.890
6.683
6.666
6.425
6.422
6.185
5.126
3.794
3.441
1.253
Cl
Cl
OH
O
N
N
H
MeOOC
COOMe
4h
11
10
9
8
7
6
5
4
3
2
1
ppm
0.90
0.96
1.98
1.00
0.95
0.91
0.88
0.98
3.00
2.95
8.91
172.23
166.16
165.74
157.71
153.81
136.15
135.85
132.44
131.39
130.27
129.03
122.87
119.19
107.53
107.31
77.28
77.02
76.77
63.44
52.75
52.45
51.39
29.70
28.37
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

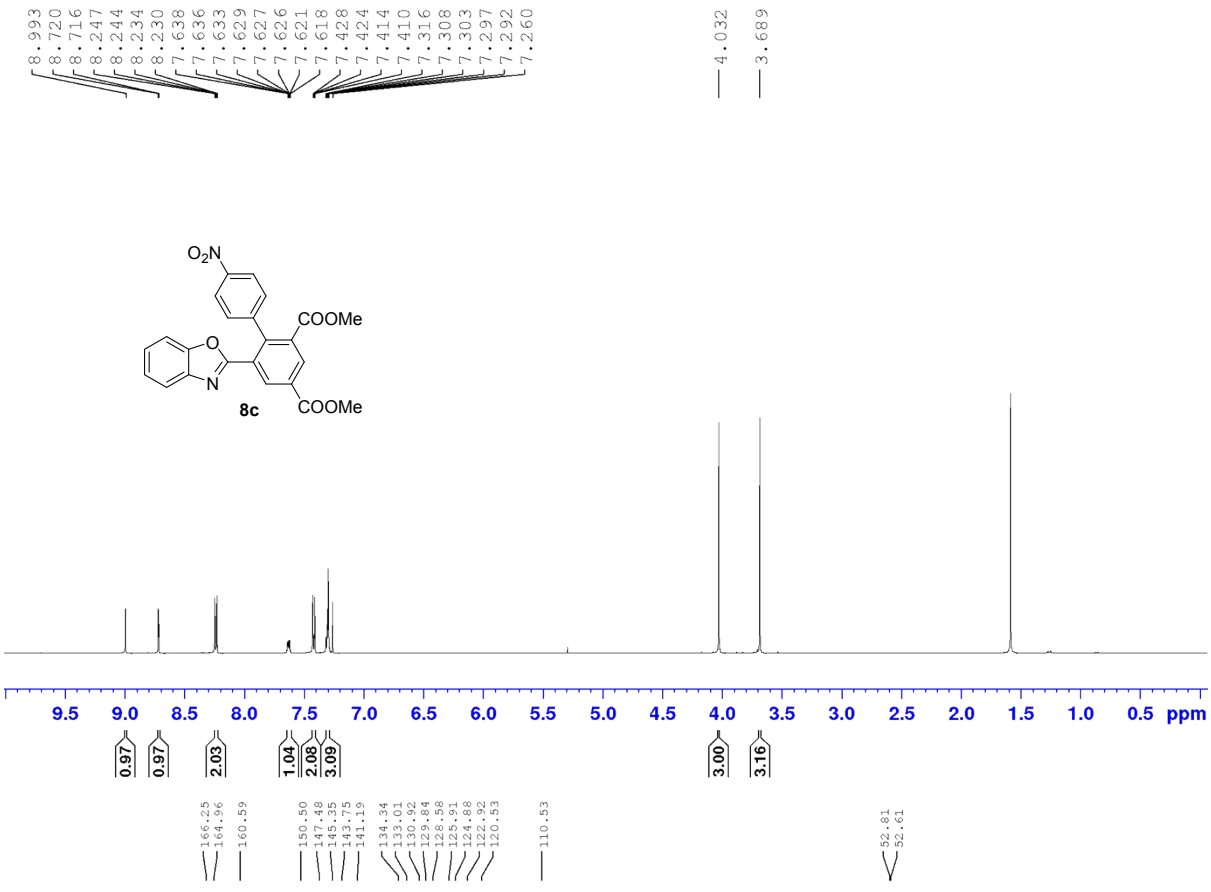

9.248
8.634
8.631
8.176
7.792
7.776
7.749
7.733
7.303
7.284
7.274
3.925
2.444
2.432
Ph(p-Me)
N
O
N
(p-Me)Ph
COOMe
O
6
9.6 9.4 9.2 9.0 8.8 8.6 8.4 8.2 8.0 7.8 7.6 7.4 ppm
0.88 0.83 0.83 1.67 1.81 5.28
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.88 0.83 0.83 1.67 1.81 5.28 2.92 2.52 3.28
192.74
166.52
164.06
158.06
144.42
143.70
143.57
138.97
134.23
130.66
129.74
129.69
129.49
129.27
129.02
108.53
52.37
29.69
21.76
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

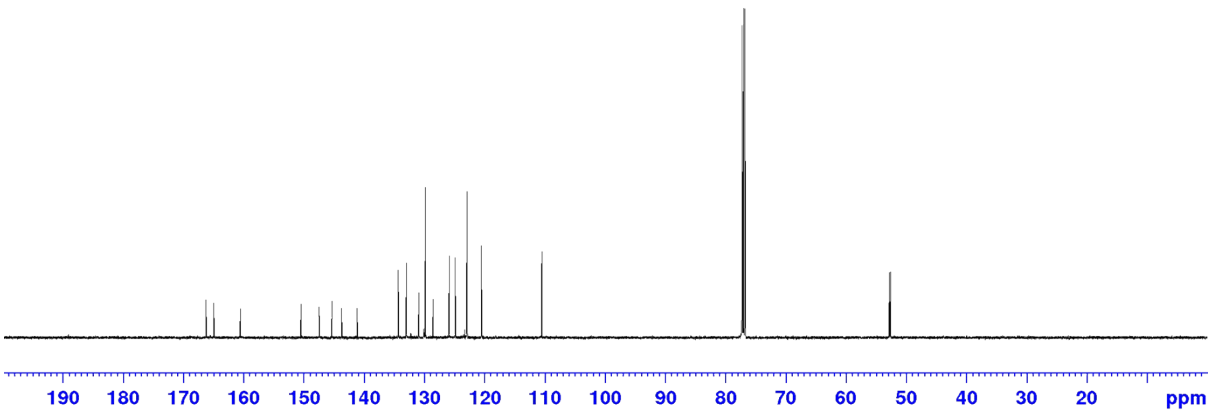

7.666
7.502
7.500
7.412
7.409
7.395
7.329
7.214
7.199
7.192
7.184
7.163
7.159
7.112
7.098
6.880
6.878
6.864
6.862
6.796
6.793
6.750
6.747
6.695
6.692
6.680
6.677
5.947
4.939
4.936
3.776
3.630
COOMe
COOMe
HN
HO
7a
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00 1.08 2.40 0.91 3.23 1.14 0.99 2.25 0.99 0.81 1.07 2.90 3.00

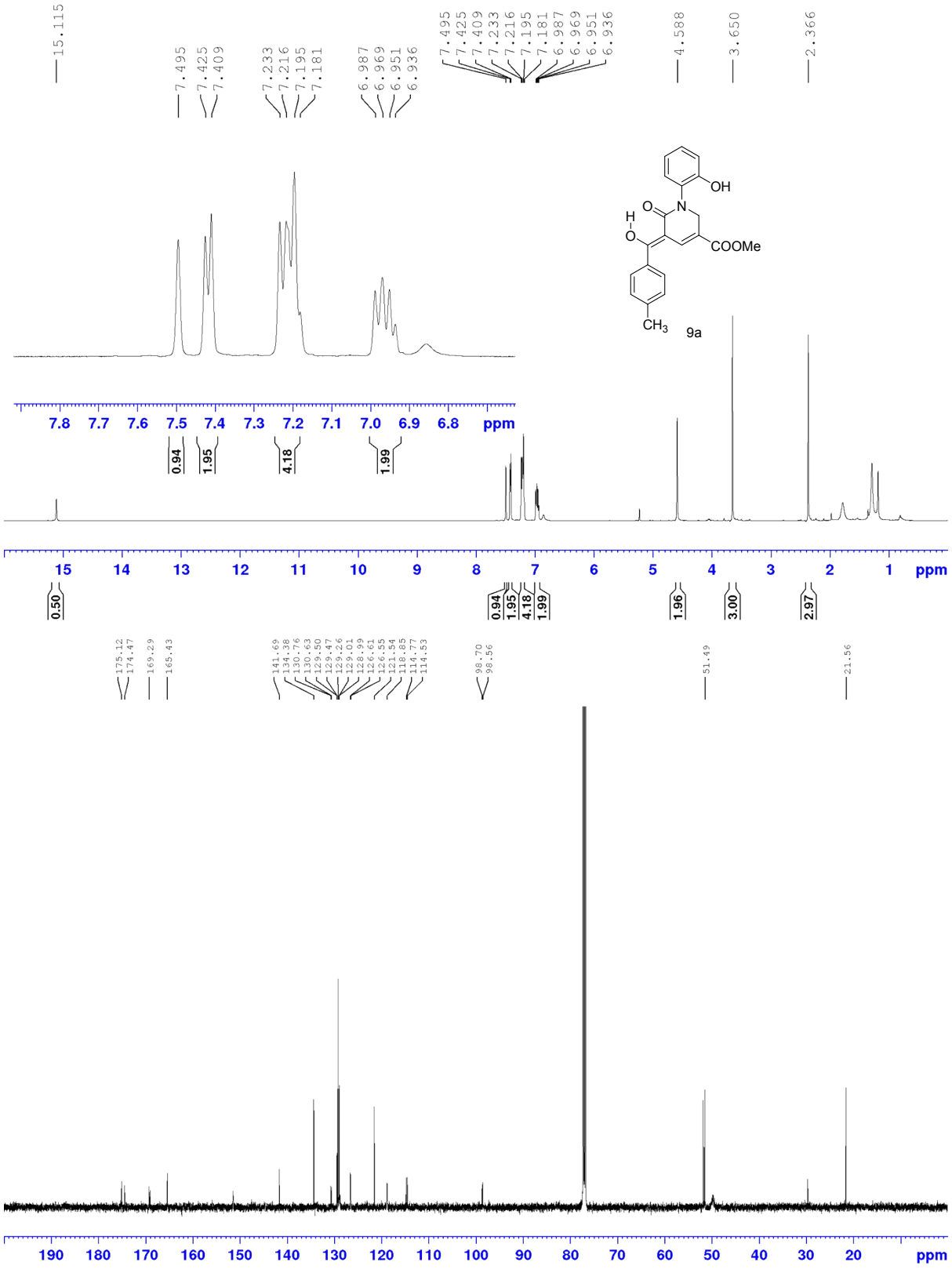

167.42
166.55
143.63
142.98
137.59
131.78
128.75
128.26
127.93
127.18
124.60
122.75
121.52
115.59
115.34
114.22
112.78
51.62
41.48
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

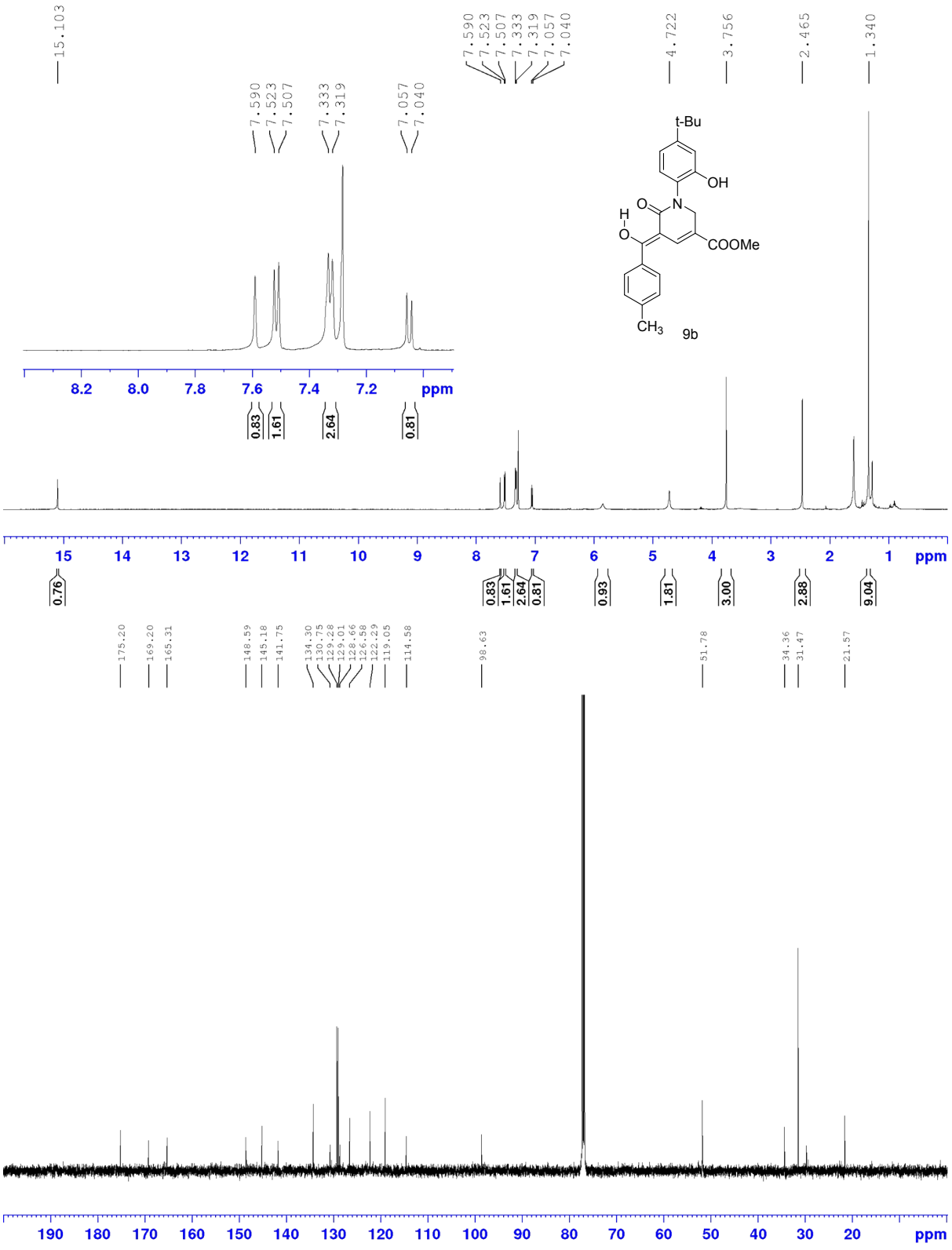

8.811
8.507
8.506
7.617
7.603
7.260
7.247
7.235
7.218
7.203
7.187
7.174
7.145
7.131
3.925
3.546
COOMe
COOMe
O
N
8a
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
ppm
0.87
0.87
0.92
8.19
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
0.87
0.87
0.92
8.19
3.00
3.00
167.51
165.30
161.88
150.75
145.85
141.29
137.95
134.18
134.10
132.37
132.30
128.91
128.54
127.90
127.78
125.41
124.55
120.30
110.54
52.64
52.40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


8.755
8.458
7.631
7.625
7.614
7.226
7.214
7.202
7.187
7.065
7.048
6.786
6.769
3.913
3.733
3.584
MeO
COOMe
O
N
COOMe
8b
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 ppm
0.87
0.88
0.94
2.86
1.68
1.81
2.90
3.00
3.00
167.79
165.34
162.06
159.37
150.78
145.57
141.34
134.48
134.12
132.23
130.05
129.86
129.48
129.10
125.38
124.53
120.32
113.35
110.62
55.21
52.60
52.46
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


8.993
8.720
8.716
8.247
8.244
8.234
8.230
7.638
7.636
7.633
7.629
7.627
7.626
7.621
7.618
7.428
7.424
7.414
7.410
7.316
7.308
7.303
7.297
7.292
7.260
4.032
3.689
O2N
COOMe
COOMe
O
N
8c
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
0.97
0.97
2.03
1.04
2.08
3.09
3.00
3.16


166.25
164.96
160.59
150.50
147.48
145.35
143.75
141.19
134.34
133.01
130.92
129.84
128.58
125.91
124.88
122.92
120.53
110.53
52.81
52.61
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


15.115
7.495
7.425
7.409
7.233
7.216
7.195
7.181
6.987
6.969
6.951
6.936
4.588
3.650
2.366
OH
O
N
H
O
COOMe
CH3
9a
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 ppm
0.94
1.95
4.18
1.99
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.50
0.94
1.95
4.18
1.99
1.96
3.00
2.97
175.12
174.47
169.29
165.43
141.69
134.38
130.76
130.63
129.50
129.47
129.26
129.01
128.99
126.61
126.55
121.54
118.85
114.77
114.53
98.70
98.56
51.49
21.56
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


15.103
7.590
7.523
7.507
7.333
7.319
7.057
7.040
4.722
3.756
2.465
1.340
t-Bu
OH
N
O
H
O
COOMe
CH3
9b
8.2
8.0
7.8
7.6
7.4
7.2
ppm
0.83
1.61
2.64
0.81
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.76
0.83
1.61
2.64
0.81
0.93
1.81
3.00
2.88
9.04
175.20
169.20
165.31
148.59
145.18
141.75
134.30
130.75
129.28
129.01
128.66
126.58
122.29
119.05
114.58
98.63
51.78
34.36
31.47
21.57
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm