Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Green synthetic approach to diverse 2-pyridones for their exceptional UV shielding functions

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Spectrophotometric Measurements

Absorbance spectra of the tested compounds were analyzed at room temperature (298 K) on a UV/vis spectrophotometer (Optizen UV-3200). Tested compounds and oxybenzone (Aldrich, analytical standard, ≥ 98% pure) were prepared in an ethanol solvent (Sigma-Aldrich, ≥99.8% pure) at a concentration of 50 μM. The data were corrected for solvent background by the instrument's calibration using the solvent as a blank. The absorption spectra of samples in solution were obtained in the range of 200-450 nm (for the measurements and determinations of typical parameters for sun protection materials: UVA/UVB, 290-400) using 1 cm quartz cell, and ethanol as a blank. The absorption data were obtained in the range of 200-450/290-400, every 1 nm, and 3 determinations were made at each point, followed by the data analysis. Critical wavelength is defined as the wavelength λ_c where the area under the spectrum from 290 nm (the approximate lower wavelength limit of terrestrial sunlight) to λ_c, is 90% of the integral of the absorbance spectrum from 290 to 400 nm. Similar to the critical wavelength, the UVA/UVB-ratio is also a reduction of the complete spectral information to one number, characterizing in some way the shape of the spectrum in terms of the amount of UVA-coverage in relation to the amount of UVB-coverage. The *in vitro* SPF is used for rating the protection strength of sun protection materials.[S1]

References

S1. (*a*) E. A. Dutra, D. A. Goncalves da Costa e Oliveira, E. R. M. Kedor-Hackmann and M. I. R. M. Santoro, *Rev. Bras. Cienc. Farm.*, 2004, **40**, 381; (*b*) J. Akrman, L. Kubac, H. Bendova, D. Jirova and K. Kejlova, *Int. J. Cosmet. Sci.*, 2009, **31**, 119; (*c*) A. P. Schuch, M. C. S. Moraes, T. Yagura and C. F. M. Menck, *Environ. Sci. Technol.*, 2014, **48**, 11584.

^{1}H NMR and ^{13}C NMR Spectra of 2-Pyridones

Compound-**4a**
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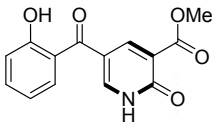

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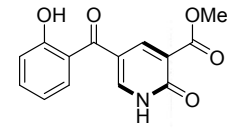

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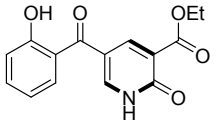

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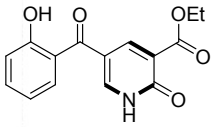

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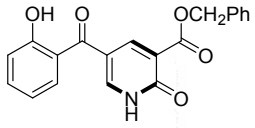

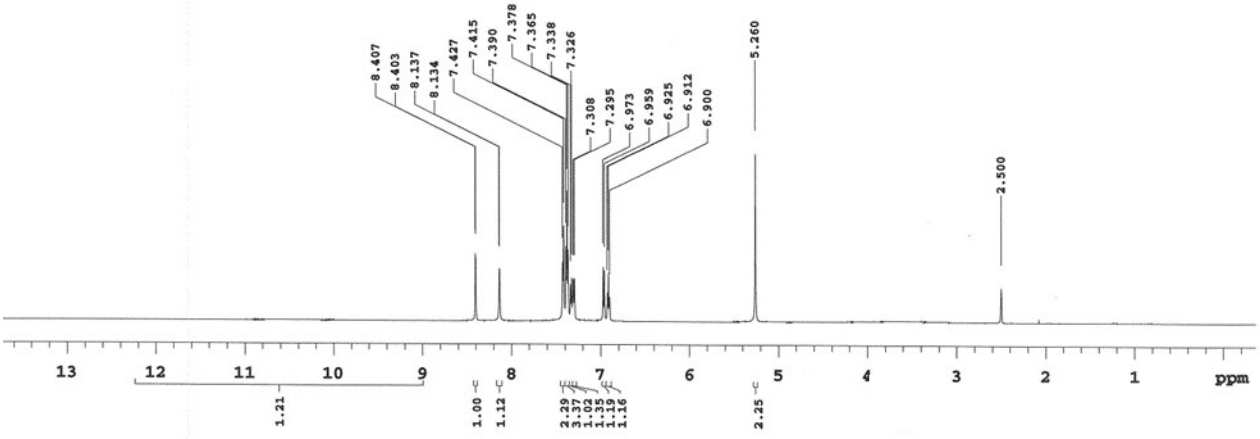

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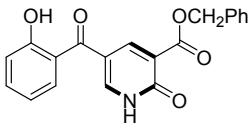

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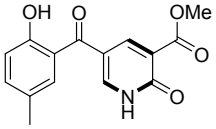

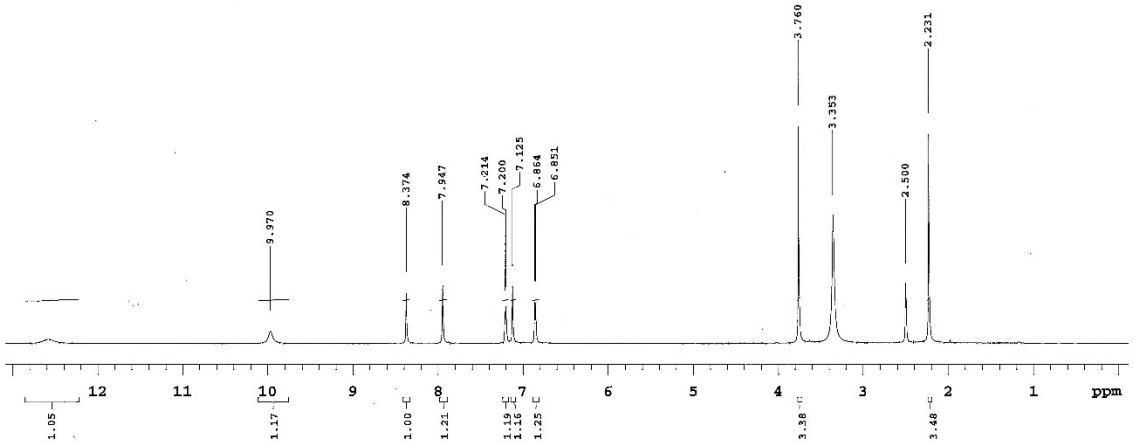

Compound-**4d**

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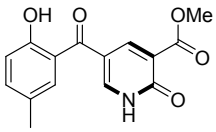

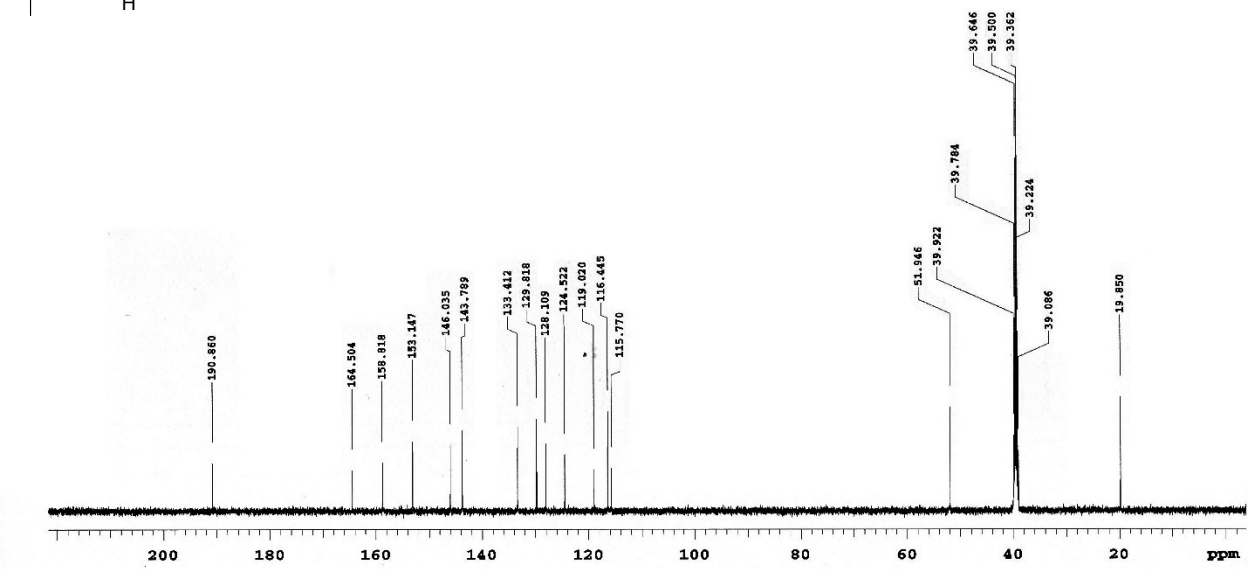

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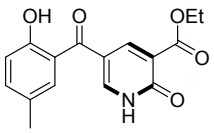

Compound-**4e**

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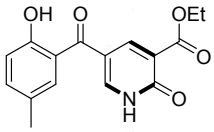

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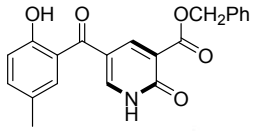

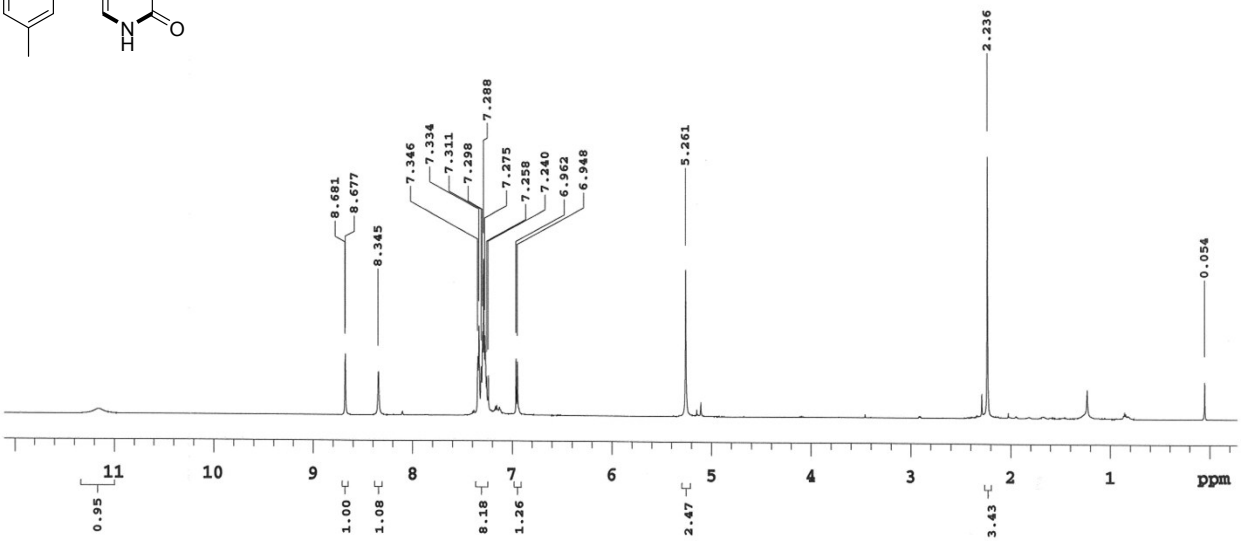

Compound-**4f**

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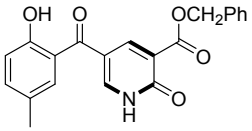

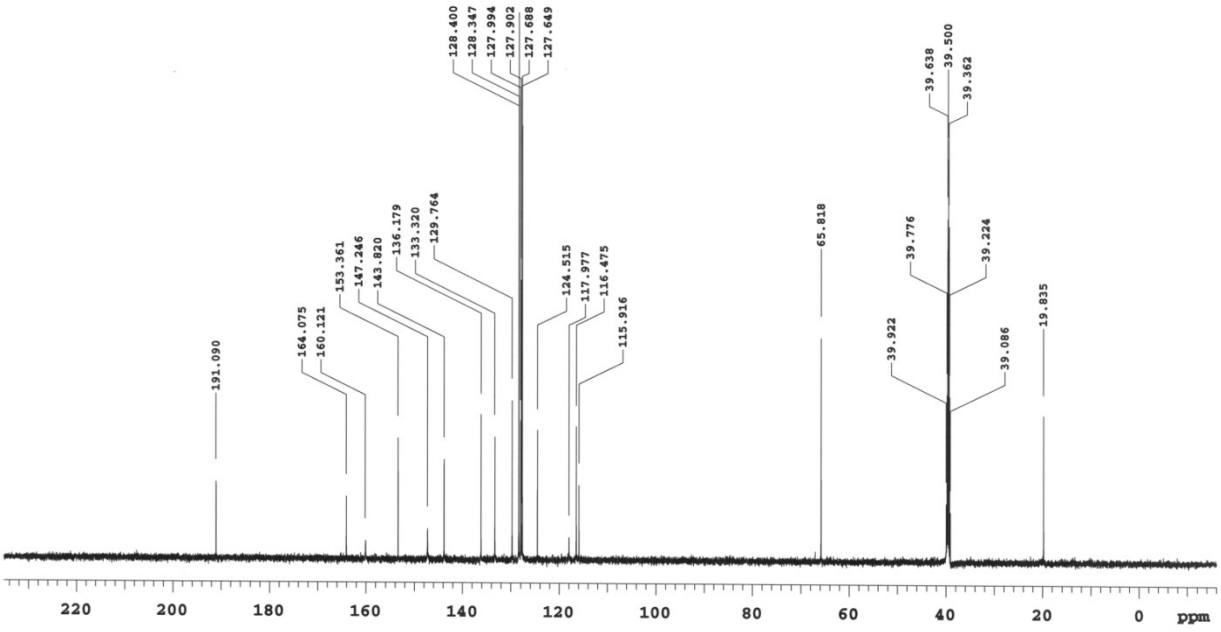

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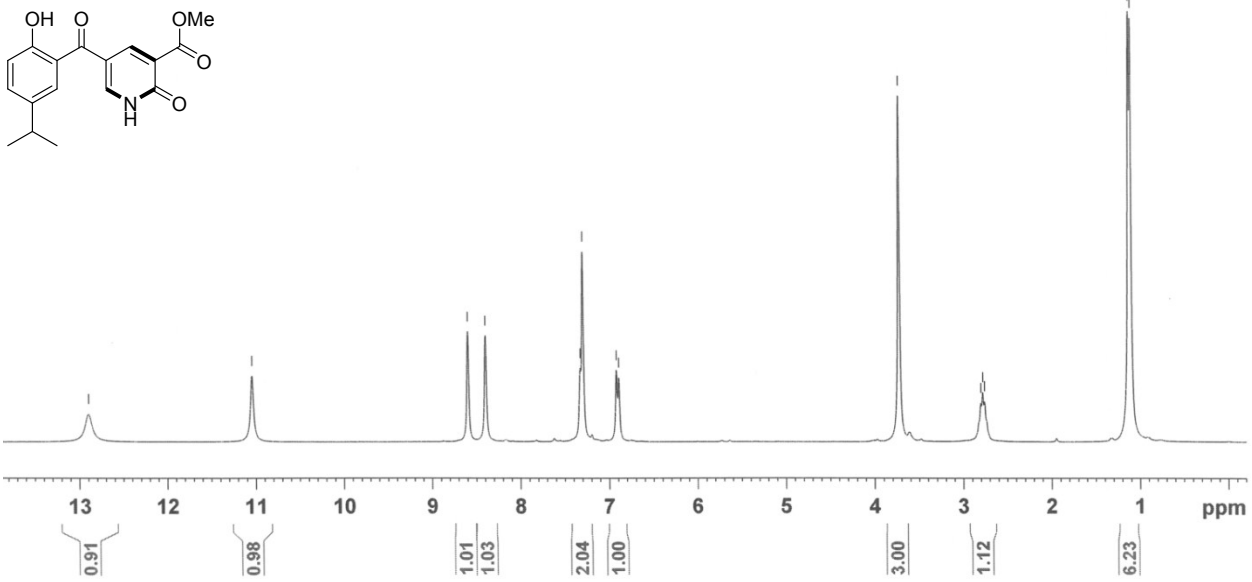

Compound-**4g**
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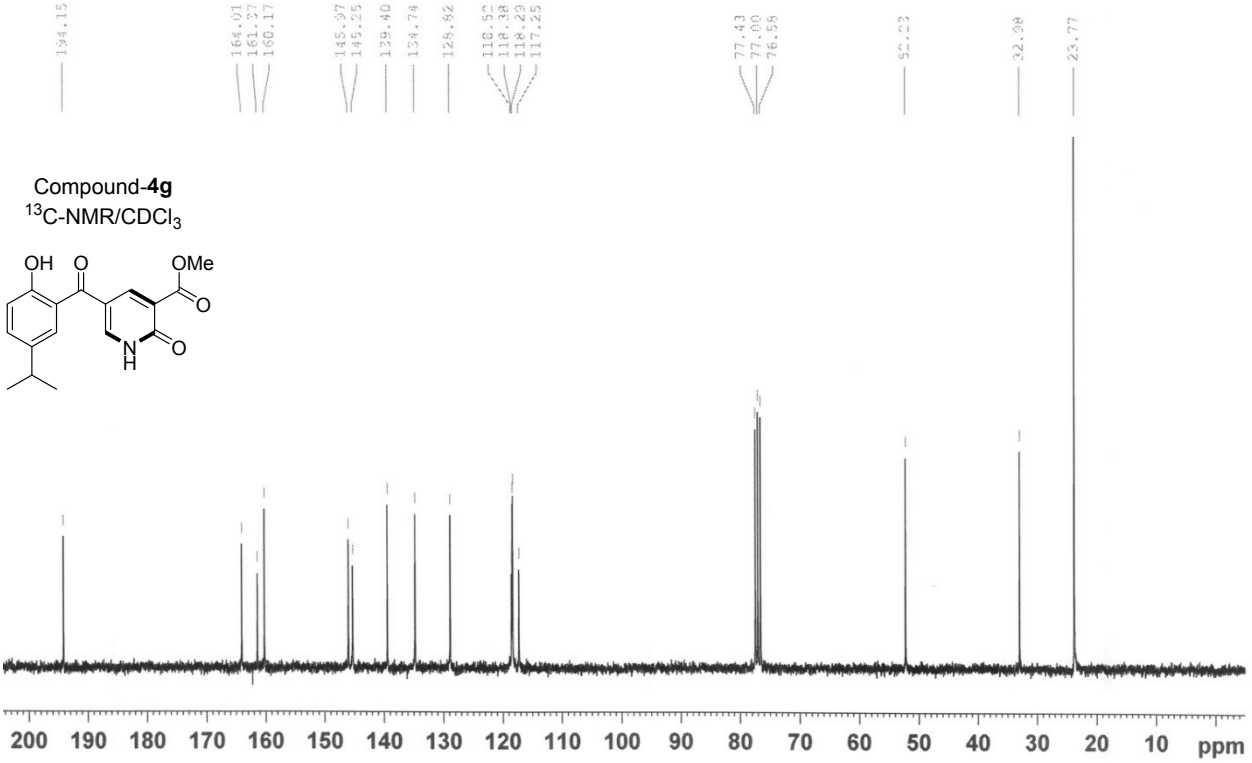

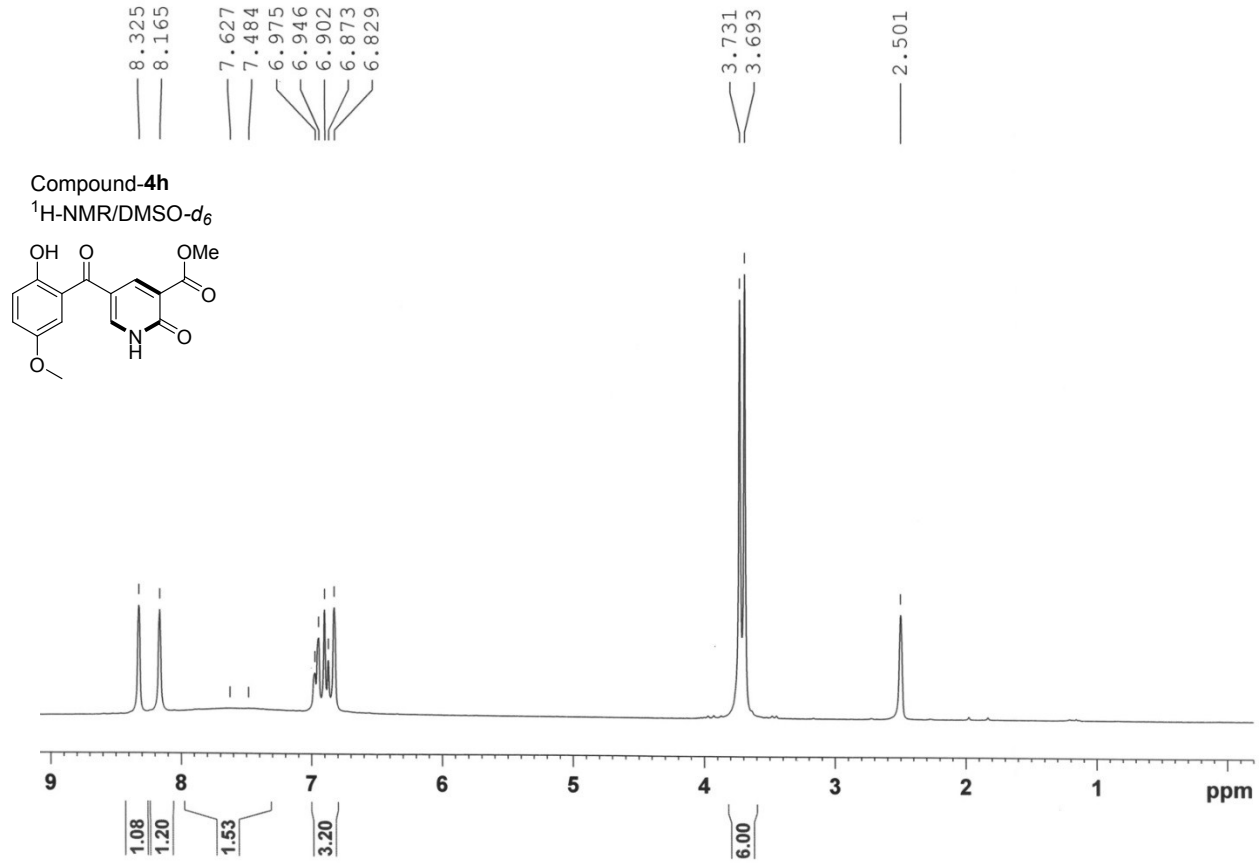
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7.627
7.484
6.975
6.946
6.902
6.873
6.829
3.731
3.693
2.501
Compound-4h
1H-NMR/DMSO-d6
OH
O
OMe
O
N
H
O
O
9
8
7
6
5
4
3
2
1
ppm
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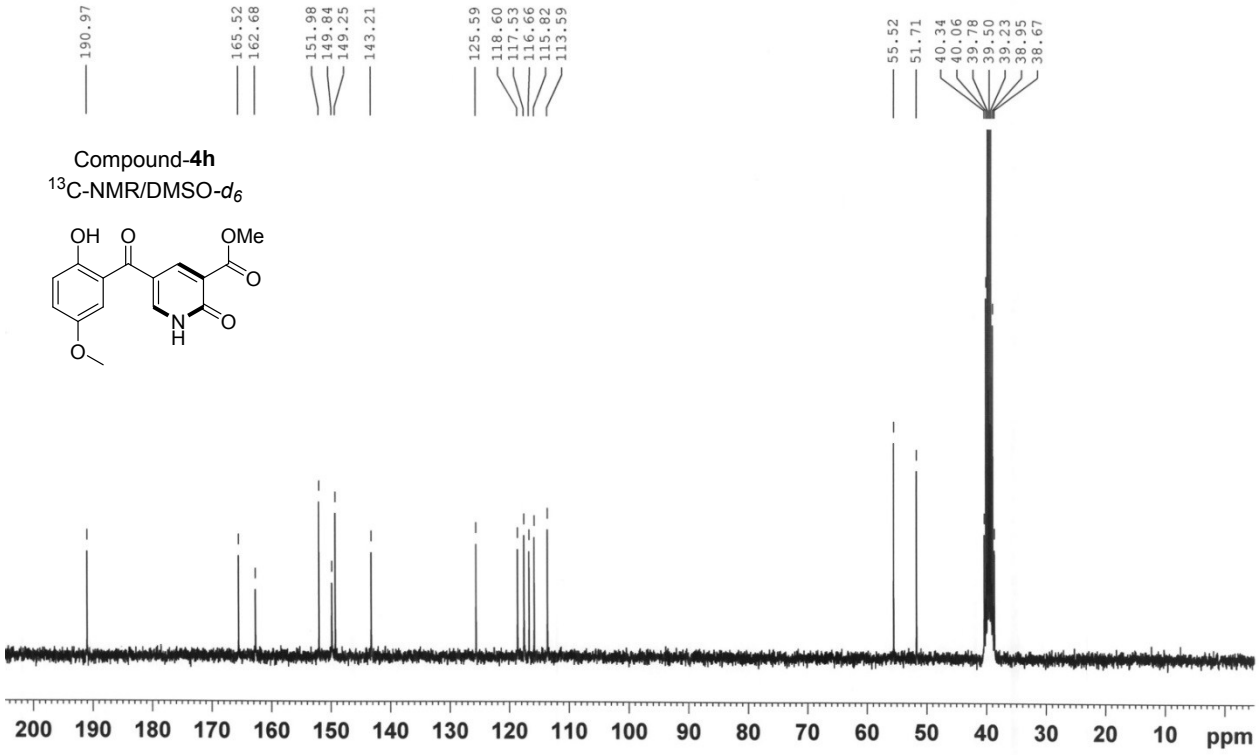
1.20
1.53
3.20
6.00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55.52
51.71
40.34
40.06
39.78
39.50
39.23
38.95
38.67
Compound-4h
13C-NMR/DMSO-d6
OH
O
OMe
O
N
H
O
O
200
190
180
170
160
150
140
130
120
110
100
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80
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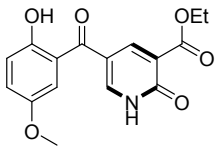
10
ppm

Compound-**4i**

^{1}H-NMR/DMSO-d_6



Compound-**4i**

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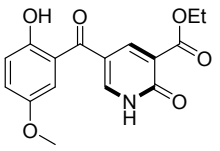

Compound-**4j**

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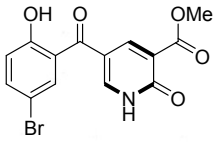

Compound-**4j**

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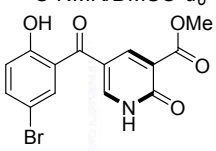

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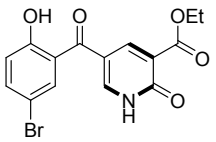

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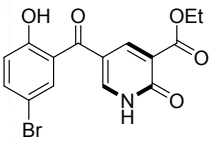

Compound-**4l**

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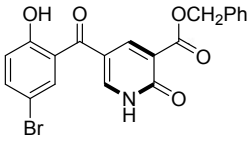

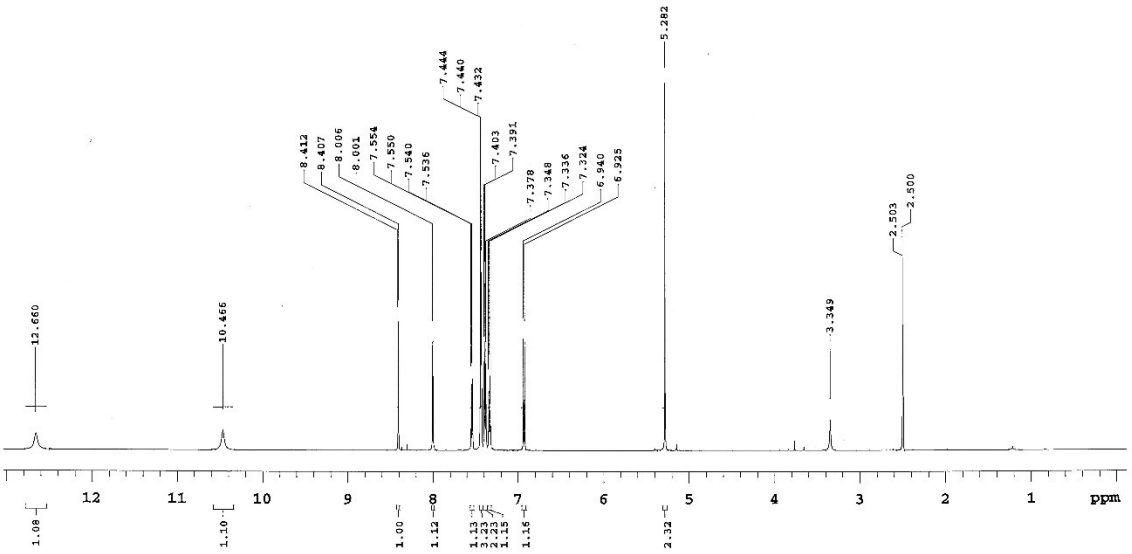

Compound-**4l**

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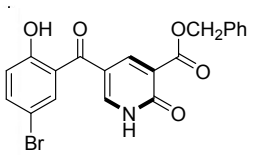

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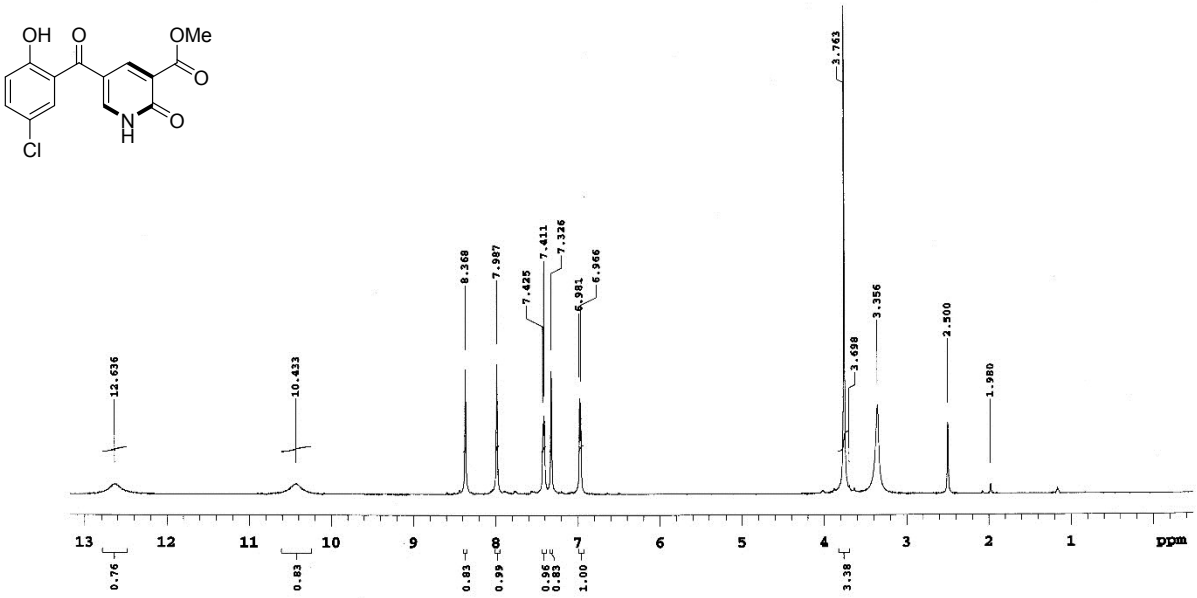

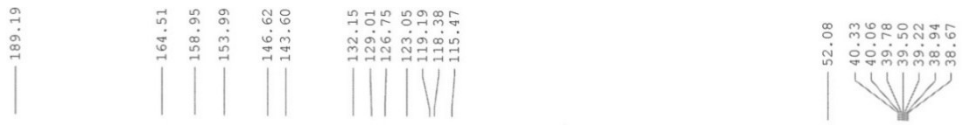

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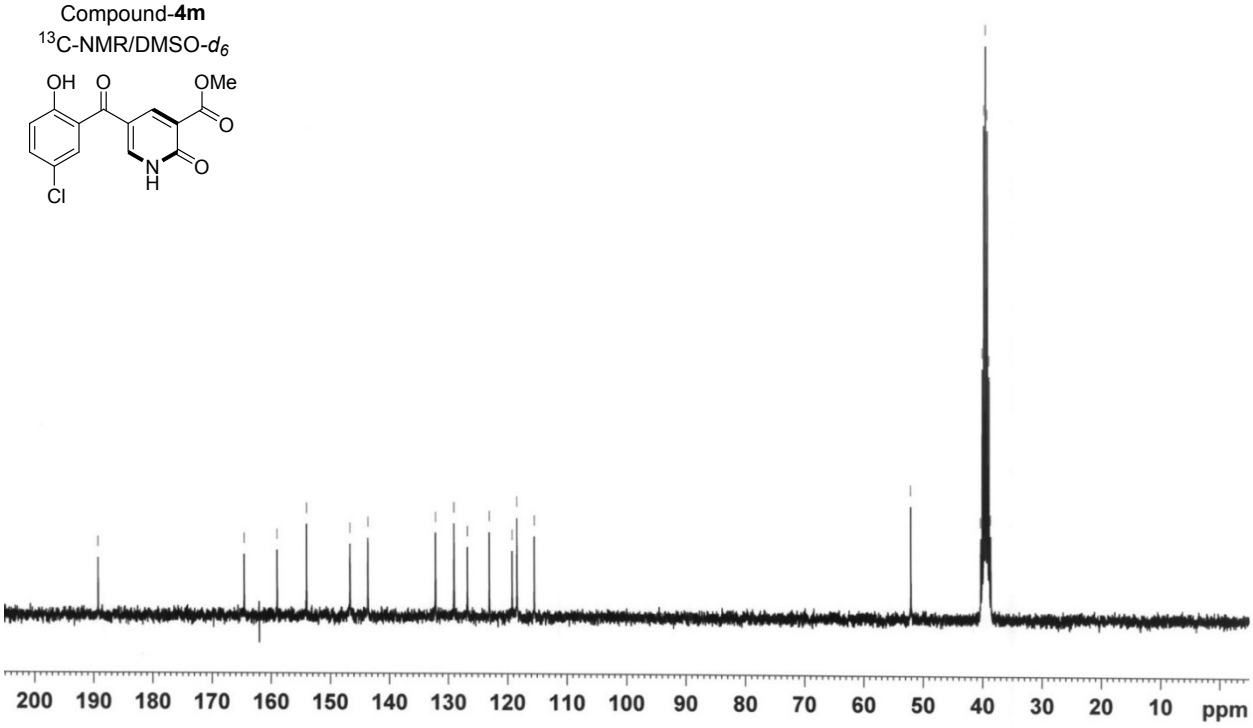

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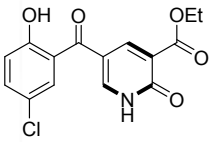

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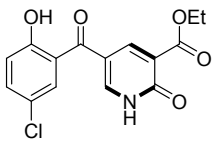

Compound-**4o**

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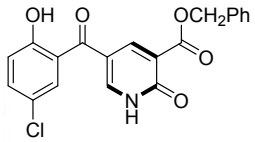

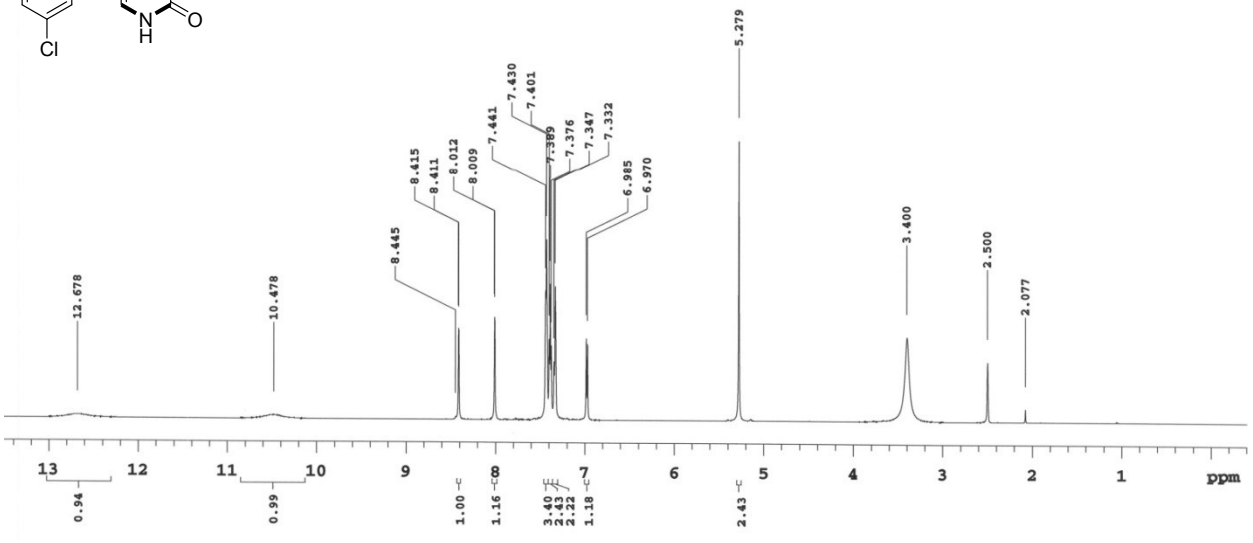

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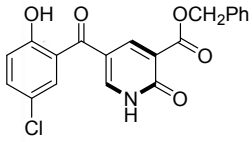

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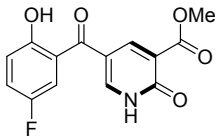

Compound-**4p**

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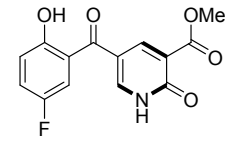

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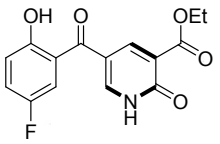

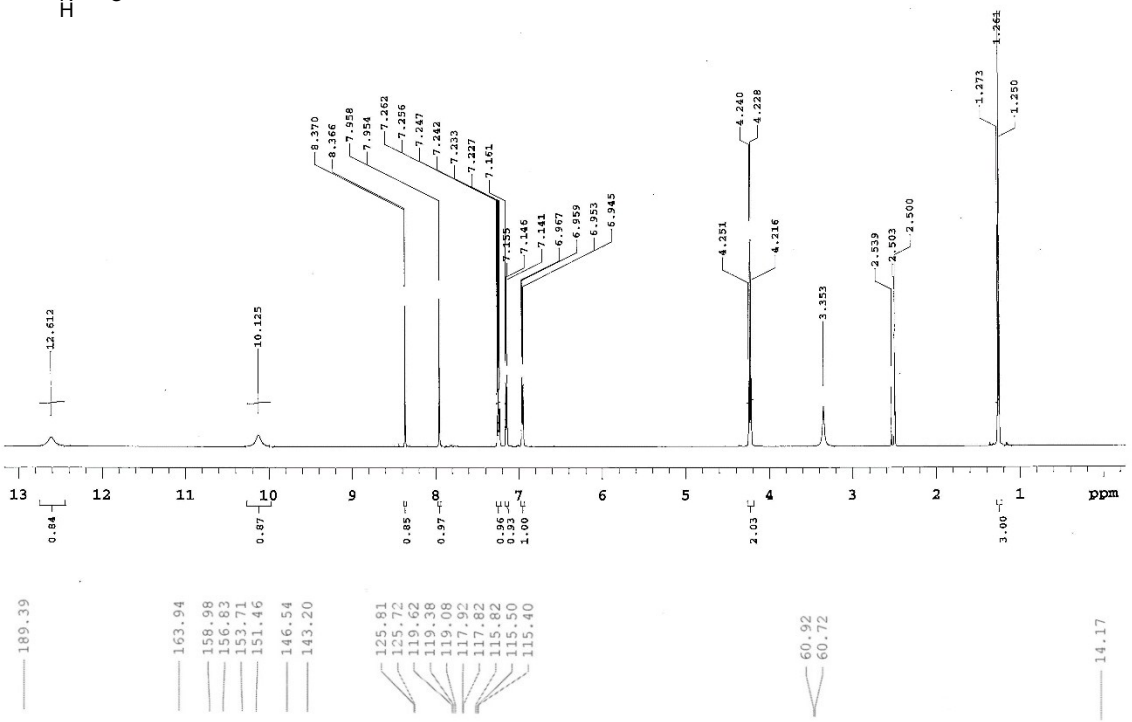

Compound-**4q**
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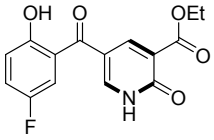

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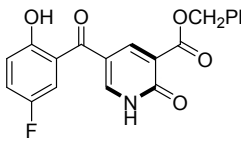
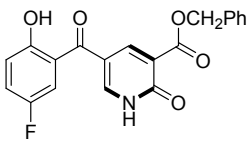
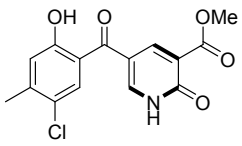


Compound-**4r**
^{13}C-NMR/DMSO-d_6



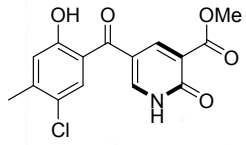
Compound-**4s**

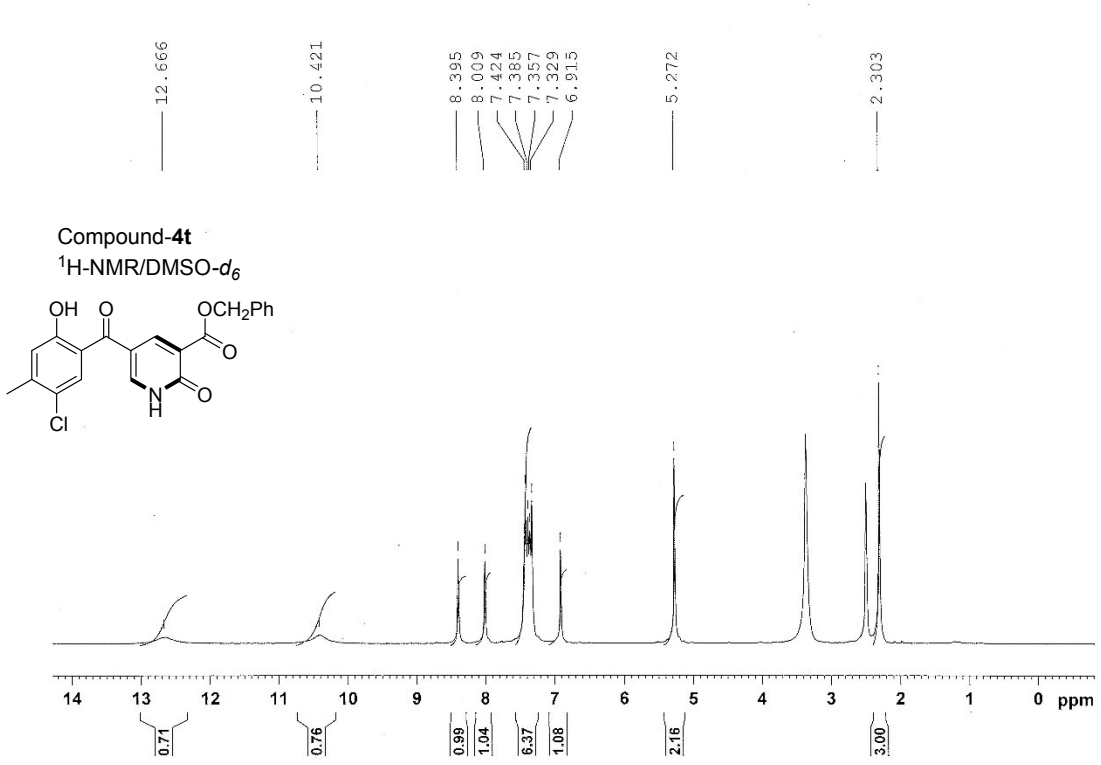
^{1}H-NMR/DMSO-d_6

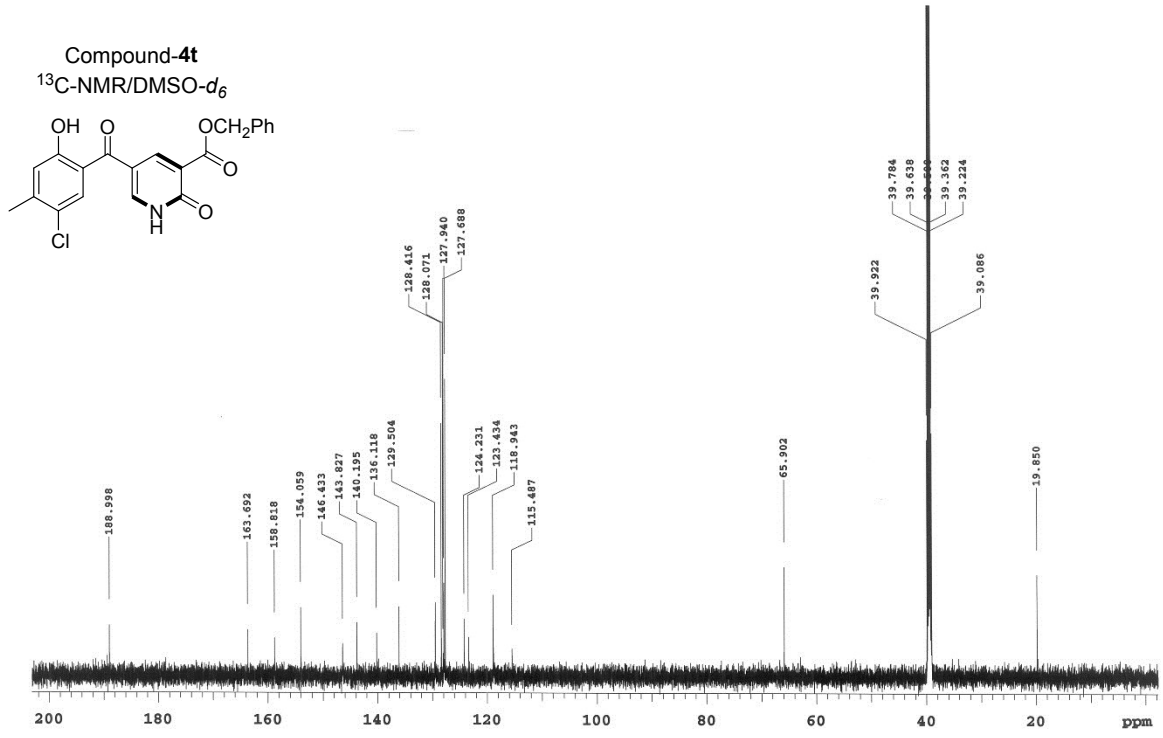


Compound-**4s**

^{13}C-NMR/DMSO-d_6

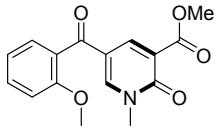


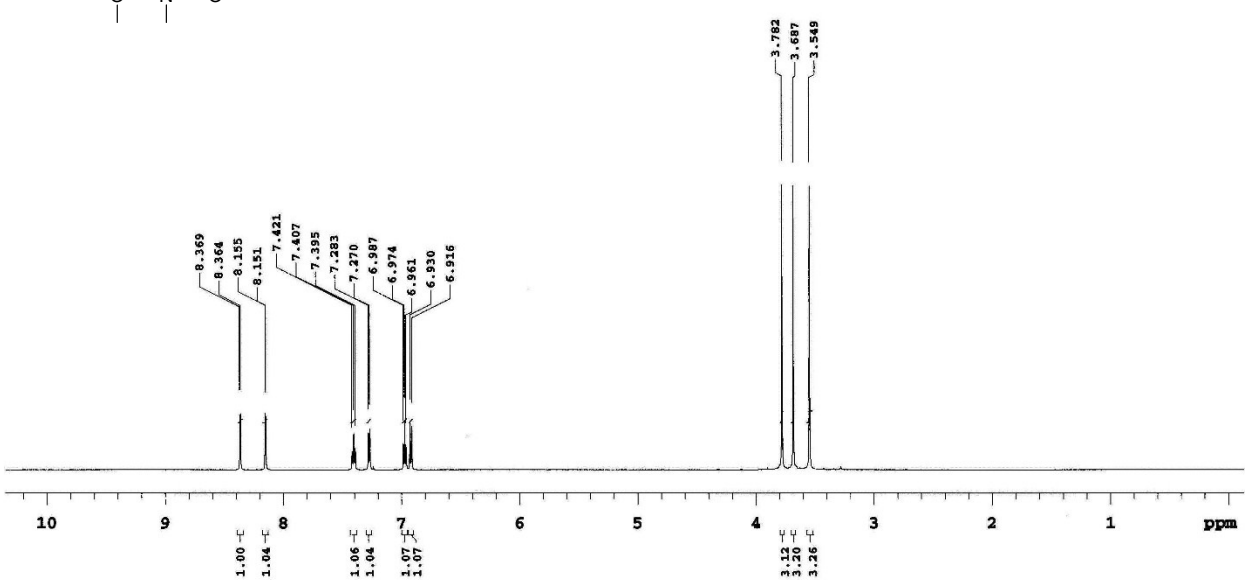

12.666
10.421
8.395
8.009
7.424
7.385
7.357
7.329
6.915
5.272
2.303
Compound-4t
1H-NMR/DMSO-d6
OH
O
OCH2Ph
O
N
H
O
Cl
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.71
0.76
0.99
1.04
6.37
1.08
2.16
3.00


Compound-4t
13C-NMR/DMSO-d6
OH
O
OCH2Ph
O
N
H
O
Cl
188.998
163.692
158.818
154.059
146.433
143.827
140.195
136.118
129.504
128.416
128.071
127.940
127.688
124.231
123.434
118.943
115.487
65.902
39.922
39.784
39.638
39.362
39.224
39.086
19.850
200
180
160
140
120
100
80
60
40
20
ppm

Compound-**5**

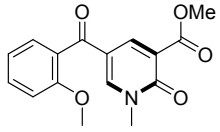
^{1}H-NMR/$CDCl_3$

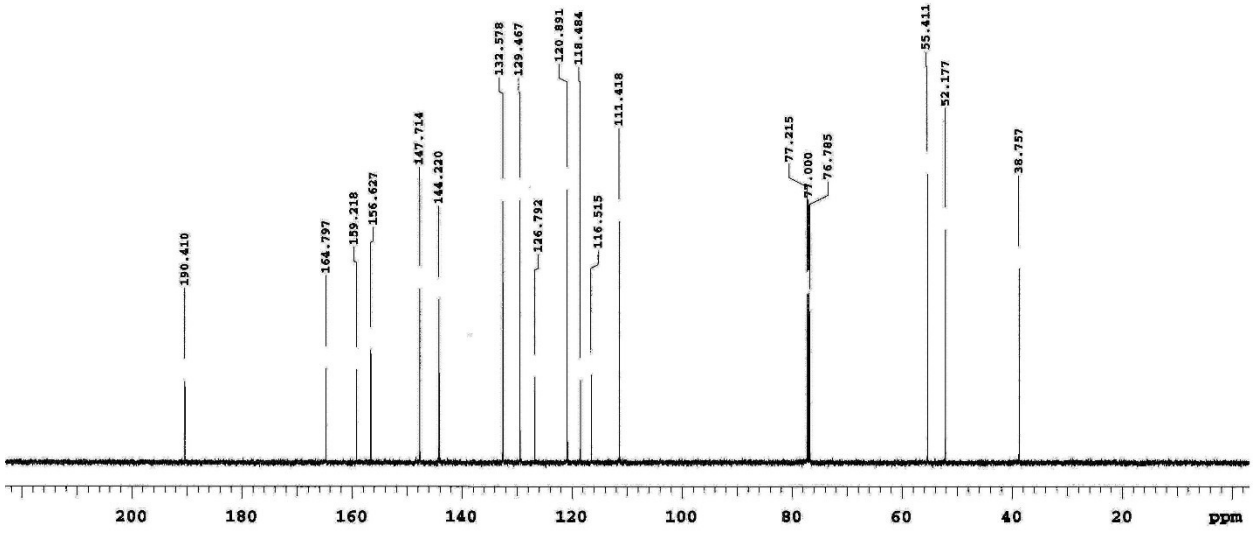




Compound-**5**

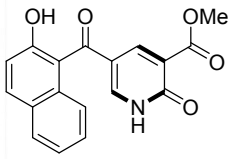
^{13}C-NMR/$CDCl_3$





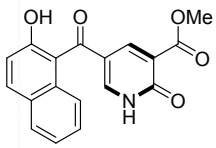
Compound-**6a**

^{1}H-NMR/DMSO-d_6



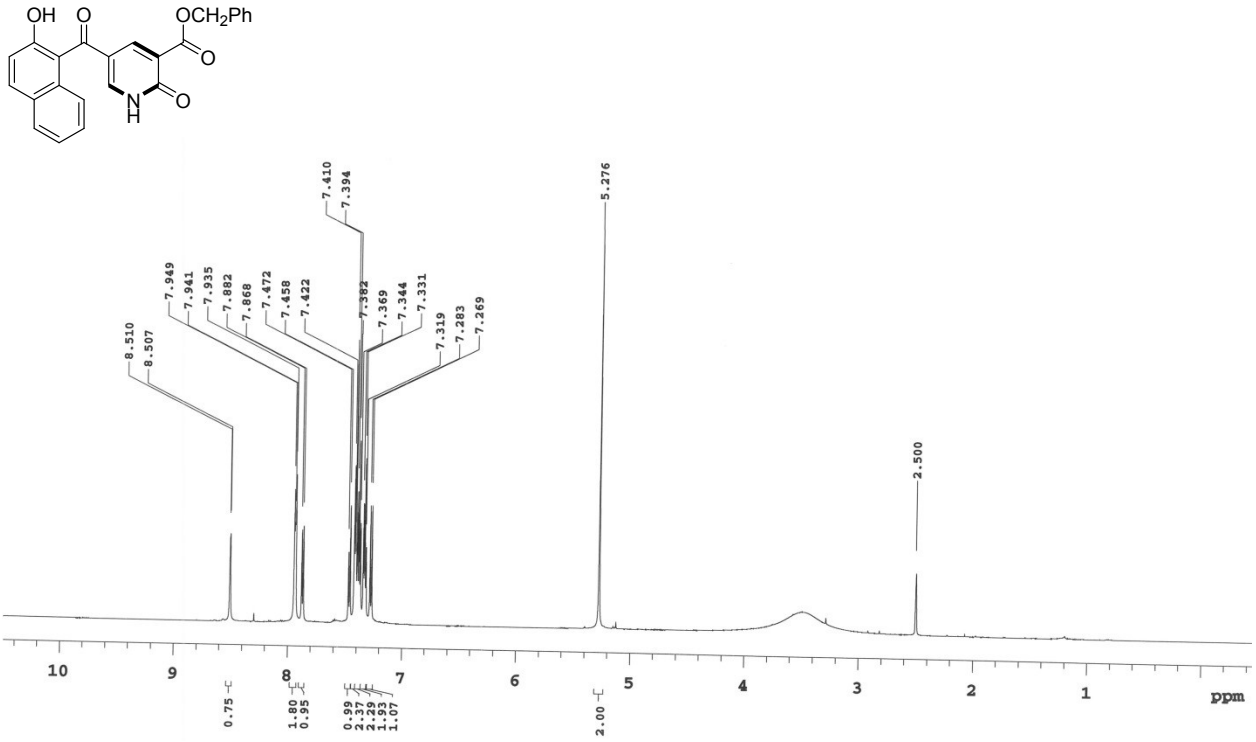
Compound-**6a**

^{13}C-NMR/DMSO-d_6



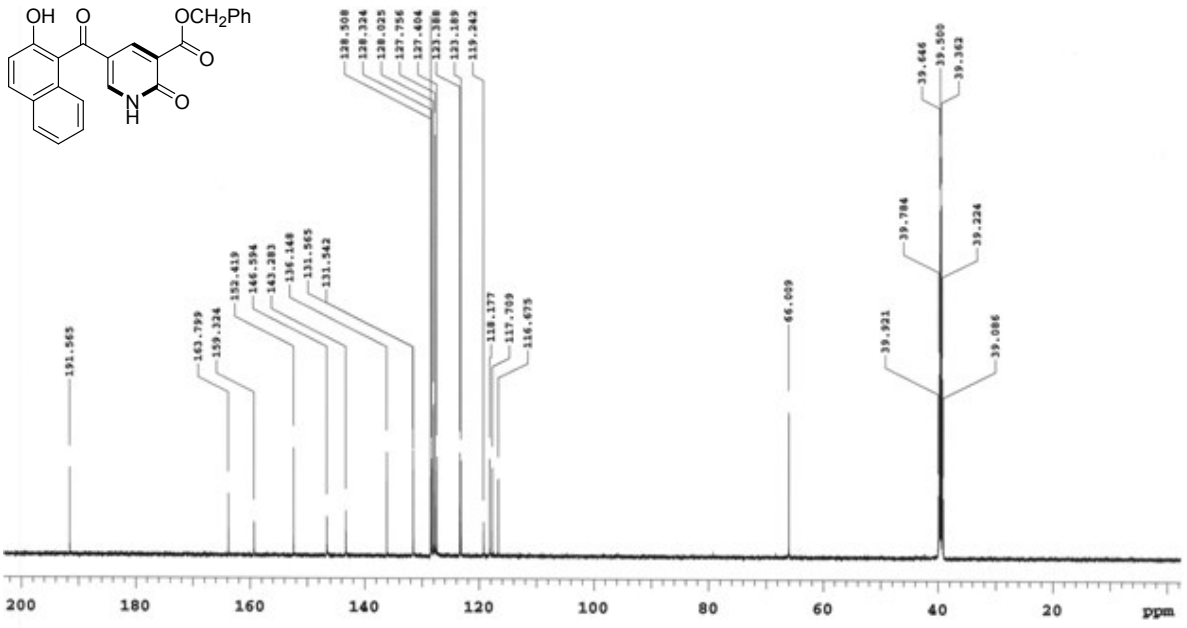
Compound-**6b**

^{1}H-NMR/DMSO-d_6



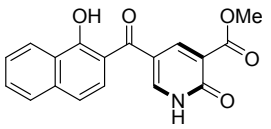
Compound-**6b**

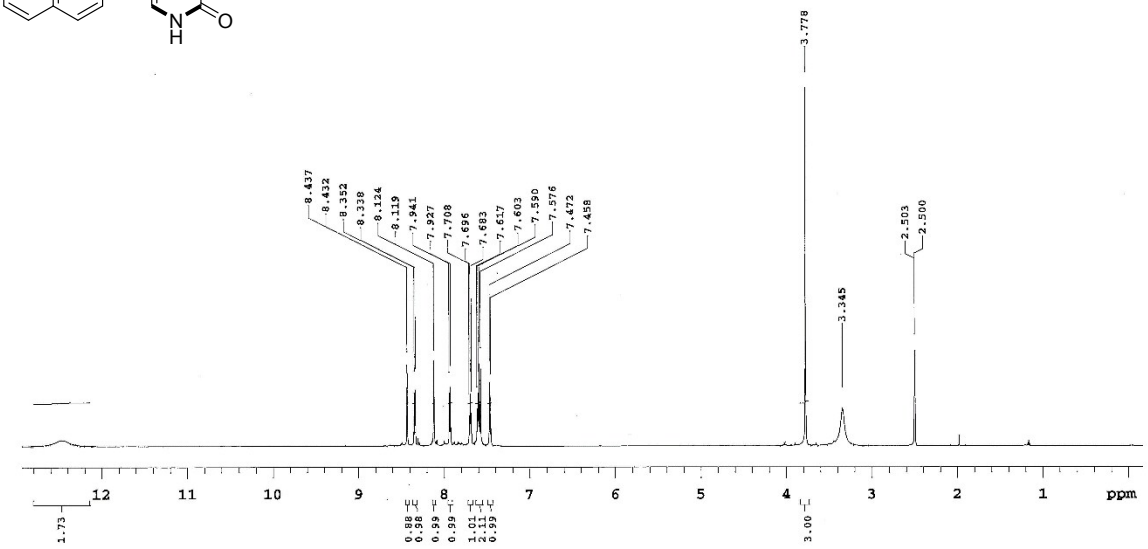
^{13}C-NMR/DMSO-d_6



Compound-**6c**

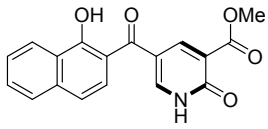
^{1}H-NMR/DMSO-d_6





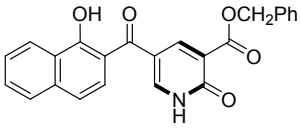
Compound-**6c**

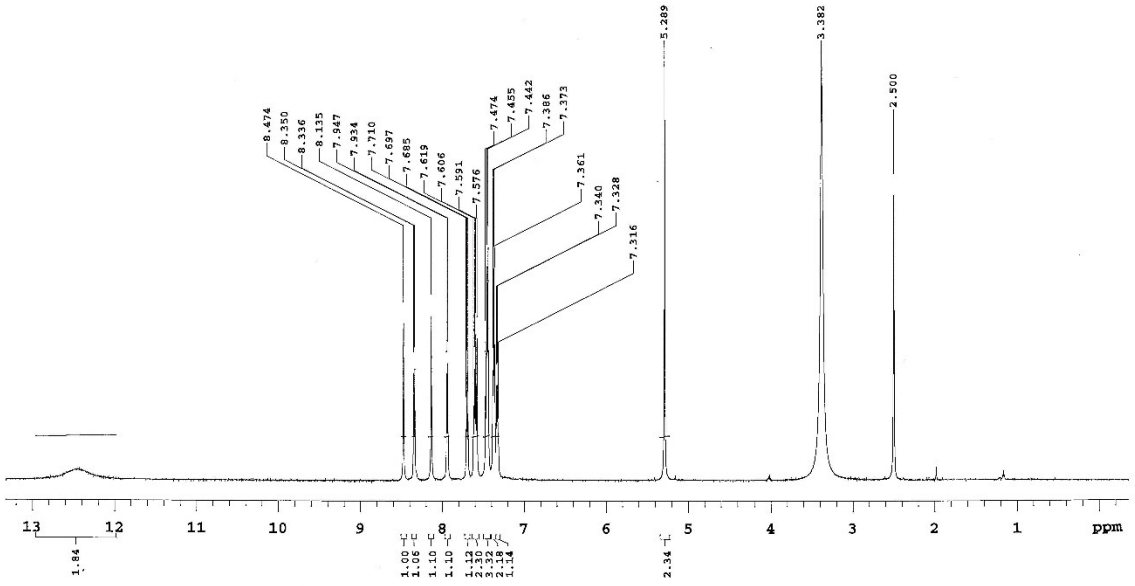
^{13}C-NMR/DMSO-d_6



Compound-**6d**

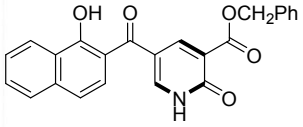
^{1}H-NMR/DMSO-d_6



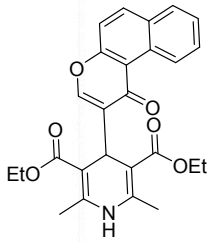


Compound-**6d**

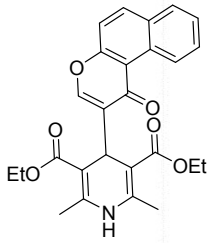
^{13}C-NMR/DMSO-d_6

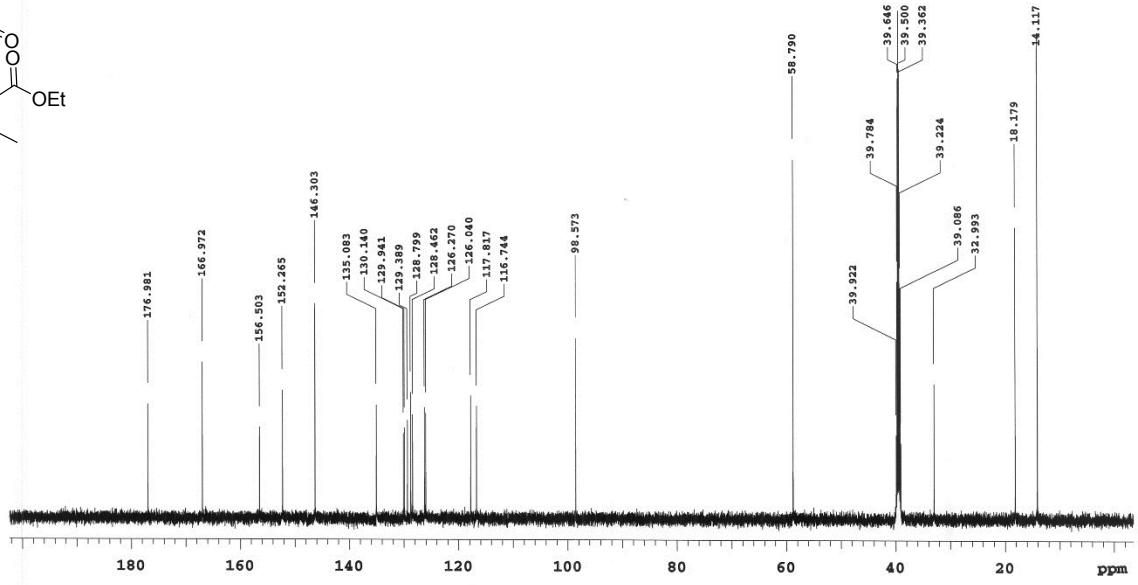


Compound-**10**
^{1}H-NMR/DMSO



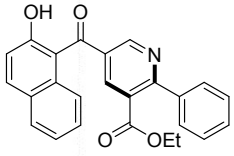
Compound-**10**
^{13}C-NMR/DMSO

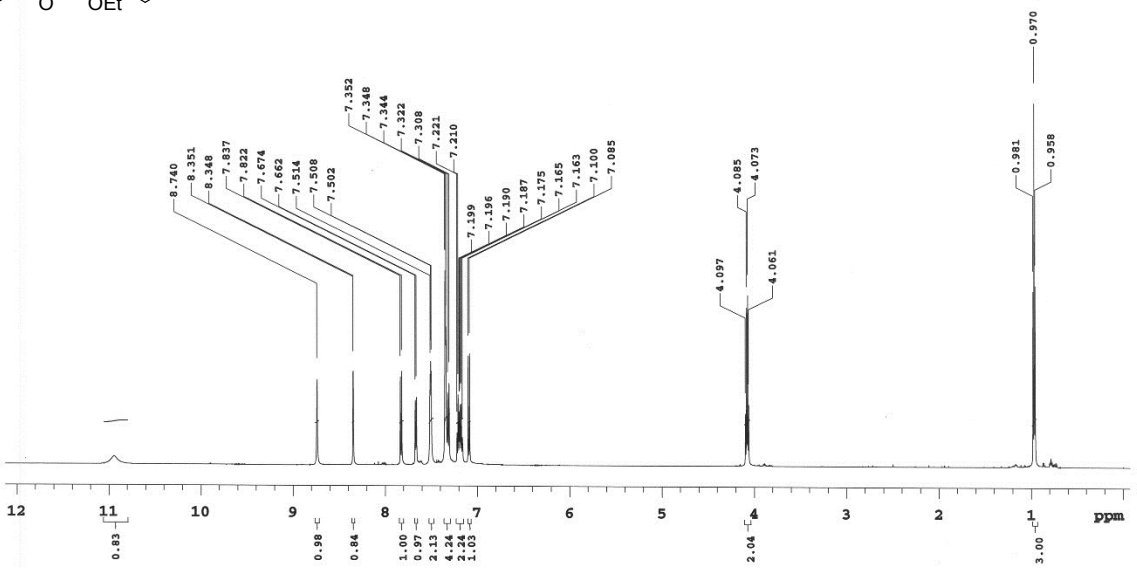




Compound-**11**

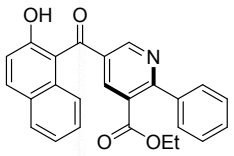
^{1}H-NMR/$CDCl_3$

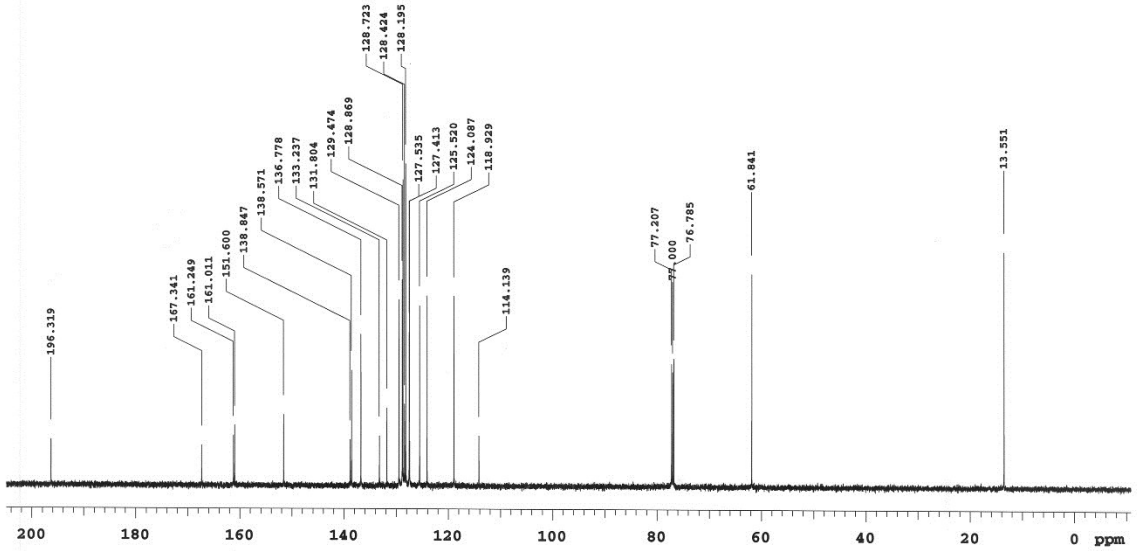




Compound-**11**

^{13}C-NMR/$CDCl_3$





X-Ray crystallographic structure and data of compound 5: Empirical Formula- $C_{16}H_{15}NO_5$, M = 301.29, Monoclinic, Space group P_{bca}, a = 8.3195(4) Å, b = 13.6469 (6) Å, c = 12.8427 (5) Å, V = 1458.10(11) Å^3, Z = 4, T = 223(2) K, ρ_{calcd} = 1.372 Mg/m^3, $2\Theta_{max.}$ = 25.89^0, Refinement of 202 parameters on 2807 independent reflections out of 46442 collected reflections (R_{int} = 0.1156) led to R_1 = 0.0616 [I >2σ(I)], wR_2 = 0.1659 (all data) and S = 1.043 with the largest difference peak and hole of 0.701 and -0.471 e. Å^{-3} respectively. The crystal structure has been deposited at the Cambridge Crystallographic Data Centre (CCDC 1495313). The data can be obtained free of charge via the Internet at www.ccdc.cam.ac.uk/data_request/cif.

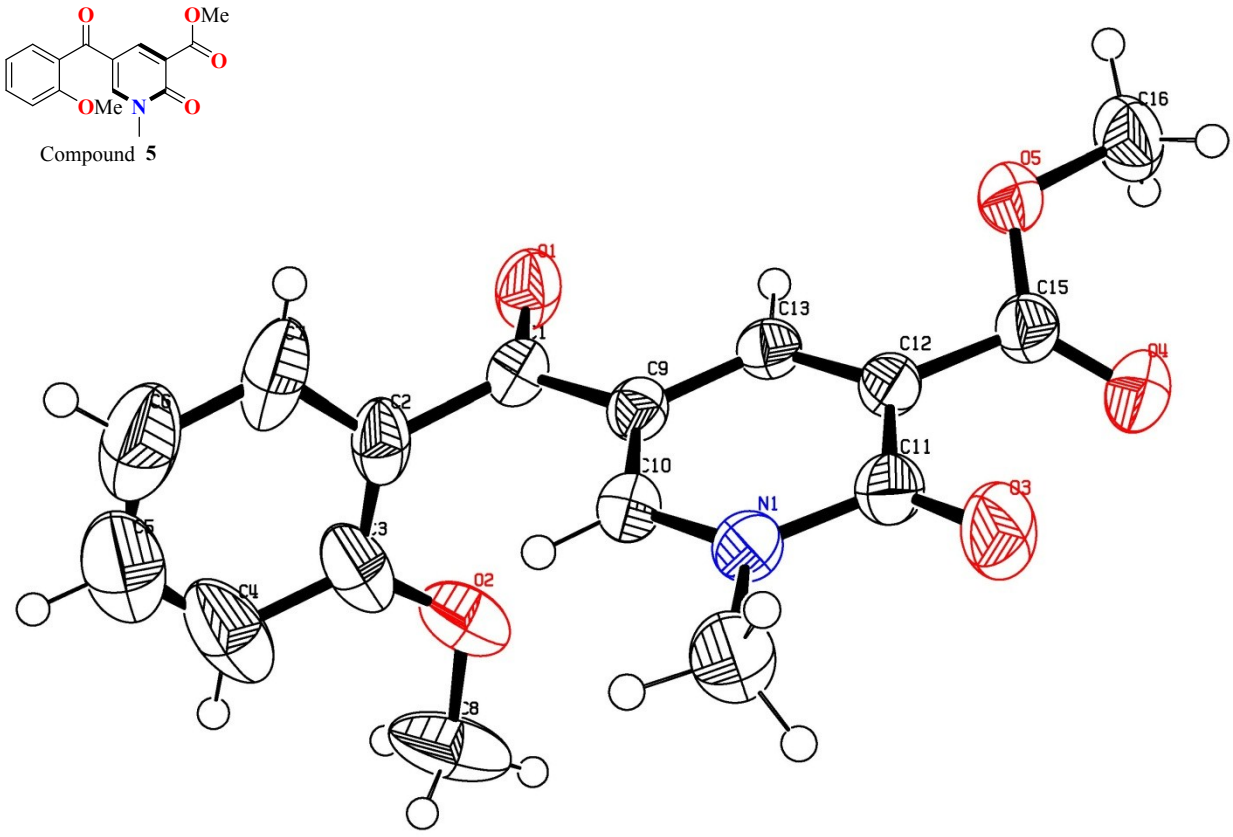


Fig. S1 X-Ray Structure of Compound **5.**

X-Ray Data of Compound 5

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

Identification code	**5**	
Empirical formula	$C_{16} H_{15} N O_5$	
Formula weight	301.29	
Temperature	223(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 8.3195(4) Å	α= 90°.
	b = 13.6469(6) Å	β= 90°.
	c = 12.8427(5) Å	γ = 90°.
Volume	1458.10(11) $Å^3$	
Z	4	
Density (calculated)	1.372 Mg/m^3	
Absorption coefficient	0.103 mm^{-1}	
F(000)	632	
Crystal size	0.24 x 0.20 x 0.19 mm^3	
Theta range for data collection	2.18 to 25.89°.	
Index ranges	-10<=h<=10, -16<=k<=16, -15<=l<=15	
Reflections collected	46442	
Independent reflections	2807 [R(int) = 0.1156]	
Completeness to theta = 25.89°	99.3 %	
Absorption correction	None	
Max. and min. transmission	0.9807 and 0.9757	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2807 / 12 / 202	
Goodness-of-fit on F^2	1.043	
Final R indices [I>2sigma(I)]	R1 = 0.0616, wR2 = 0.1555	
R indices (all data)	R1 = 0.0769, wR2 = 0.1659	
Largest diff. peak and hole	0.701 and -0.471 $e.Å^{-3}$	

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) or **5**. U (eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	9294(3)	10043(2)	8707(2)	57(1)
C(1)	8280(3)	9437(2)	8493(2)	40(1)
C(2)	8205(4)	9015(3)	7417(2)	52(1)
C(3)	8944(4)	8121(3)	7210(3)	66(1)
C(4)	8970(6)	7743(4)	6201(4)	99(2)
C(5)	8216(7)	8271(5)	5439(4)	101(2)
C(6)	7440(7)	9129(5)	5616(4)	97(2)
C(7)	7443(5)	9533(4)	6630(3)	79(1)
O(2)	9630(3)	7677(2)	8049(2)	70(1)
C(8)	10296(5)	6718(3)	7907(5)	94(2)
C(9)	7091(3)	9110(2)	9267(2)	37(1)
C(10)	5914(3)	8451(2)	9030(2)	40(1)
N(1)	4828(3)	8150(2)	9740(2)	43(1)
C(11)	4848(4)	8456(2)	10791(2)	45(1)
C(12)	6102(3)	9151(2)	11036(2)	39(1)
C(13)	7153(3)	9466(2)	10295(2)	38(1)
C(14)	3539(5)	7483(3)	9426(3)	65(1)
O(3)	3827(3)	8144(2)	11388(2)	70(1)
C(15)	6291(4)	9476(2)	12132(2)	44(1)
O(4)	6062(4)	8981(2)	12892(2)	66(1)
O(5)	6809(3)	10399(2)	12167(2)	51(1)
C(16)	7185(5)	10782(3)	13184(3)	64(1)

Table S3. Bond lengths [Å] and angles [°] for **5**.

O(1)-C(1)	1.212(4)
C(1)-C(9)	1.471(4)
C(1)-C(2)	1.499(4)
C(2)-C(7)	1.387(5)
C(2)-C(3)	1.392(6)
C(3)-O(2)	1.362(5)
C(3)-C(4)	1.394(5)
C(4)-C(5)	1.368(8)
C(4)-H(4)	0.9400
C(5)-C(6)	1.357(8)
C(5)-H(5)	0.9400
C(6)-C(7)	1.413(7)
C(6)-H(6)	0.9400
C(7)-H(7)	0.9400
O(2)-C(8)	1.432(5)
C(8)-H(8A)	0.9700
C(8)-H(8B)	0.9700
C(8)-H(8C)	0.9700
C(9)-C(10)	1.365(4)
C(9)-C(13)	1.408(4)
C(10)-N(1)	1.348(4)
C(10)-H(10)	0.9400
N(1)-C(11)	1.413(4)
N(1)-C(14)	1.463(4)
C(11)-O(3)	1.221(4)
C(11)-C(12)	1.444(4)
C(12)-C(13)	1.362(4)
C(12)-C(15)	1.485(4)
C(13)-H(13)	0.9400
C(14)-H(14A)	0.9700
C(14)-H(14B)	0.9700
C(14)-H(14C)	0.9700
C(15)-O(4)	1.202(4)
C(15)-O(5)	1.332(4)

O(5)-C(16)	1.441(4)
C(16)-H(16A)	0.9700
C(16)-H(16B)	0.9700
C(16)-H(16C)	0.9700

O(1)-C(1)-C(9)	121.4(3)
O(1)-C(1)-C(2)	120.0(3)
C(9)-C(1)-C(2)	118.6(3)
C(7)-C(2)-C(3)	120.6(4)
C(7)-C(2)-C(1)	119.7(4)
C(3)-C(2)-C(1)	119.7(3)
O(2)-C(3)-C(2)	115.1(3)
O(2)-C(3)-C(4)	124.4(5)
C(2)-C(3)-C(4)	120.6(5)
C(5)-C(4)-C(3)	117.6(6)
C(5)-C(4)-H(4)	121.2
C(3)-C(4)-H(4)	121.2
C(6)-C(5)-C(4)	123.6(5)
C(6)-C(5)-H(5)	118.2
C(4)-C(5)-H(5)	118.2
C(5)-C(6)-C(7)	119.3(5)
C(5)-C(6)-H(6)	120.3
C(7)-C(6)-H(6)	120.3
C(2)-C(7)-C(6)	118.3(5)
C(2)-C(7)-H(7)	120.9
C(6)-C(7)-H(7)	120.9
C(3)-O(2)-C(8)	117.9(4)
O(2)-C(8)-H(8A)	109.5
O(2)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
O(2)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(10)-C(9)-C(13)	117.6(3)
C(10)-C(9)-C(1)	122.1(3)
C(13)-C(9)-C(1)	120.3(3)

N(1)-C(10)-C(9)	122.0(3)
N(1)-C(10)-H(10)	119.0
C(9)-C(10)-H(10)	119.0
C(10)-N(1)-C(11)	123.3(3)
C(10)-N(1)-C(14)	119.6(3)
C(11)-N(1)-C(14)	117.2(3)
O(3)-C(11)-N(1)	119.3(3)
O(3)-C(11)-C(12)	126.5(3)
N(1)-C(11)-C(12)	114.2(2)
C(13)-C(12)-C(11)	121.3(3)
C(13)-C(12)-C(15)	120.0(3)
C(11)-C(12)-C(15)	118.6(3)
C(12)-C(13)-C(9)	121.5(3)
C(12)-C(13)-H(13)	119.3
C(9)-C(13)-H(13)	119.3
N(1)-C(14)-H(14A)	109.5
N(1)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
N(1)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
O(4)-C(15)-O(5)	123.7(3)
O(4)-C(15)-C(12)	125.8(3)
O(5)-C(15)-C(12)	110.4(2)
C(15)-O(5)-C(16)	116.3(3)
O(5)-C(16)-H(16A)	109.5
O(5)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
O(5)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table S4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 332. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	60(1)	66(2)	45(1)	3(1)	1(1)	-24(1)
C(1)	38(2)	44(2)	39(2)	7(1)	-1(1)	-2(1)
C(2)	48(2)	74(2)	34(2)	1(2)	5(1)	-24(2)
C(3)	56(2)	86(3)	54(2)	-28(2)	19(2)	-34(2)
C(4)	99(3)	121(4)	77(3)	-47(3)	37(3)	-71(3)
C(5)	111(3)	126(3)	65(2)	-13(2)	12(2)	-70(3)
C(6)	104(3)	124(3)	63(2)	26(2)	-13(2)	-53(2)
C(7)	79(3)	114(4)	45(2)	26(2)	-12(2)	-43(3)
O(2)	65(2)	61(2)	84(2)	-27(1)	5(1)	4(1)
C(8)	57(2)	60(2)	164(5)	-47(3)	29(3)	-6(2)
C(9)	36(1)	38(2)	36(2)	4(1)	1(1)	3(1)
C(10)	40(2)	43(2)	37(2)	3(1)	1(1)	1(1)
N(1)	41(1)	43(1)	45(1)	1(1)	5(1)	-7(1)
C(11)	45(2)	44(2)	46(2)	1(1)	10(1)	0(1)
C(12)	41(2)	37(2)	38(2)	2(1)	4(1)	4(1)
C(13)	38(2)	35(1)	40(2)	2(1)	-1(1)	2(1)
C(14)	61(2)	71(2)	61(2)	-8(2)	8(2)	-28(2)
O(3)	68(2)	81(2)	60(2)	-7(1)	28(1)	-26(1)
C(15)	47(2)	46(2)	40(2)	2(1)	6(1)	4(1)
O(4)	96(2)	62(2)	40(1)	8(1)	8(1)	-8(1)
O(5)	65(1)	49(1)	38(1)	-3(1)	2(1)	-5(1)
C(16)	79(3)	71(2)	44(2)	-13(2)	2(2)	-14(2)

Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **5.**

	x	y	z	U(eq)
H(4)	9486	7147	6051	119
H(5)	8238	8026	4755	121
H(6)	6906	9453	5070	116
H(7)	6941	10136	6767	95
H(8A)	9493	6291	7601	141
H(8B)	10627	6456	8576	141
H(8C)	11220	6757	7448	141
H(10)	5859	8198	8350	48
H(13)	7938	9932	10475	45
H(14A)	3670	7313	8698	97
H(14B)	2508	7802	9525	97
H(14C)	3585	6894	9846	97
H(16A)	6271	10691	13642	97
H(16B)	7431	11475	13129	97
H(16C)	8108	10437	13465	97

Table S6. Torsion angles [°] for **5**.

O(1)-C(1)-C(2)-C(7)	-81.4(4)
C(9)-C(1)-C(2)-C(7)	97.8(4)
O(1)-C(1)-C(2)-C(3)	96.5(4)
C(9)-C(1)-C(2)-C(3)	-84.3(4)
C(7)-C(2)-C(3)-O(2)	-179.1(3)
C(1)-C(2)-C(3)-O(2)	3.0(4)
C(7)-C(2)-C(3)-C(4)	1.7(5)
C(1)-C(2)-C(3)-C(4)	-176.2(3)
O(2)-C(3)-C(4)-C(5)	179.5(4)
C(2)-C(3)-C(4)-C(5)	-1.4(6)
C(3)-C(4)-C(5)-C(6)	-0.7(7)
C(4)-C(5)-C(6)-C(7)	2.4(7)
C(3)-C(2)-C(7)-C(6)	-0.1(5)
C(1)-C(2)-C(7)-C(6)	177.9(3)
C(5)-C(6)-C(7)-C(2)	-1.9(6)
C(2)-C(3)-O(2)-C(8)	175.0(3)
C(4)-C(3)-O(2)-C(8)	-5.9(5)
O(1)-C(1)-C(9)-C(10)	178.1(3)
C(2)-C(1)-C(9)-C(10)	-1.1(4)
O(1)-C(1)-C(9)-C(13)	-2.8(4)
C(2)-C(1)-C(9)-C(13)	178.0(3)
C(13)-C(9)-C(10)-N(1)	0.7(4)
C(1)-C(9)-C(10)-N(1)	179.8(3)
C(9)-C(10)-N(1)-C(11)	-2.6(4)
C(9)-C(10)-N(1)-C(14)	176.6(3)
C(10)-N(1)-C(11)-O(3)	-179.5(3)
C(14)-N(1)-C(11)-O(3)	1.3(5)
C(10)-N(1)-C(11)-C(12)	2.2(4)
C(14)-N(1)-C(11)-C(12)	-177.0(3)
O(3)-C(11)-C(12)-C(13)	-178.3(3)
N(1)-C(11)-C(12)-C(13)	-0.1(4)
O(3)-C(11)-C(12)-C(15)	5.3(5)
N(1)-C(11)-C(12)-C(15)	-176.5(3)
C(11)-C(12)-C(13)-C(9)	-1.7(4)

C(15)-C(12)-C(13)-C(9)	174.7(3)
C(10)-C(9)-C(13)-C(12)	1.4(4)
C(1)-C(9)-C(13)-C(12)	-177.8(3)
C(13)-C(12)-C(15)-O(4)	-142.0(3)
C(11)-C(12)-C(15)-O(4)	34.4(5)
C(13)-C(12)-C(15)-O(5)	35.9(4)
C(11)-C(12)-C(15)-O(5)	-147.6(3)
O(4)-C(15)-O(5)-C(16)	3.5(5)
C(12)-C(15)-O(5)-C(16)	-174.5(3)

Symmetry transformations used to generate equivalent atoms: