Electronic Supplementary Material (ESI) for New Journal of Chemistry.

SUPPORTING INFORMATION

Novel construction of diversely functionalized *N*-heteroaryl-2-pyridones *via* copper(II)-catalyzed [3+2+1] annulation

Muhammad Saeed Akhtar,[a] Jae-Jin, Shim,[a] Sung Hong Kim,[b] and Yong Rok Lee*[a]

[a]School of Chemical Engineering, Yeungnam University, Gyeongsan 712-749, Republic of Korea

[b]Analysis Research Division, Daegu Center, Korea Basic Science Institute, Daegu 702-701, Republic of Korea

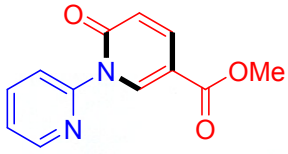
E-mail: yrlee@yu.ac.kr

Phone: 82-53-810-2529; Fax: 82-53-810-4631

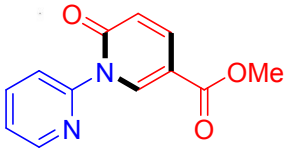
TABLE OF CONTENTS

^{1}H NMR and ^{13}C NMR Spectra of Synthesized Compounds 3, 4 and 5

Methyl 2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3a)

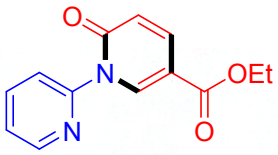


^{1}H NMR of **3a**
600 MHz, $CDCl_3$

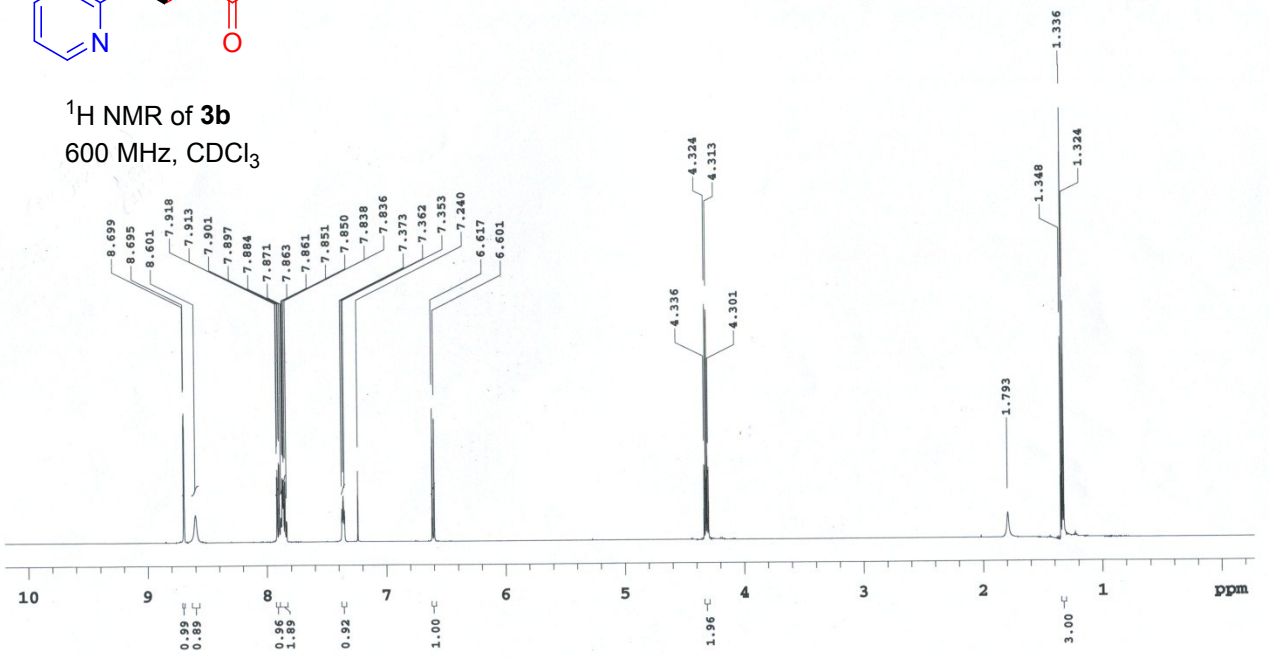


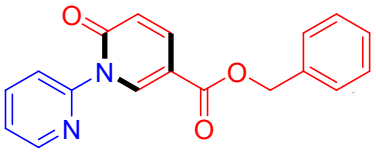
^{13}C NMR of **3a**
150 MHz, $CDCl_3$

Ethyl 2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3b)



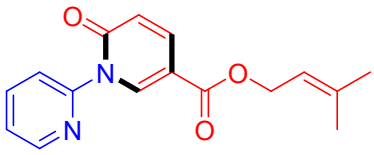
^{1}H NMR of **3b**
600 MHz, $CDCl_3$



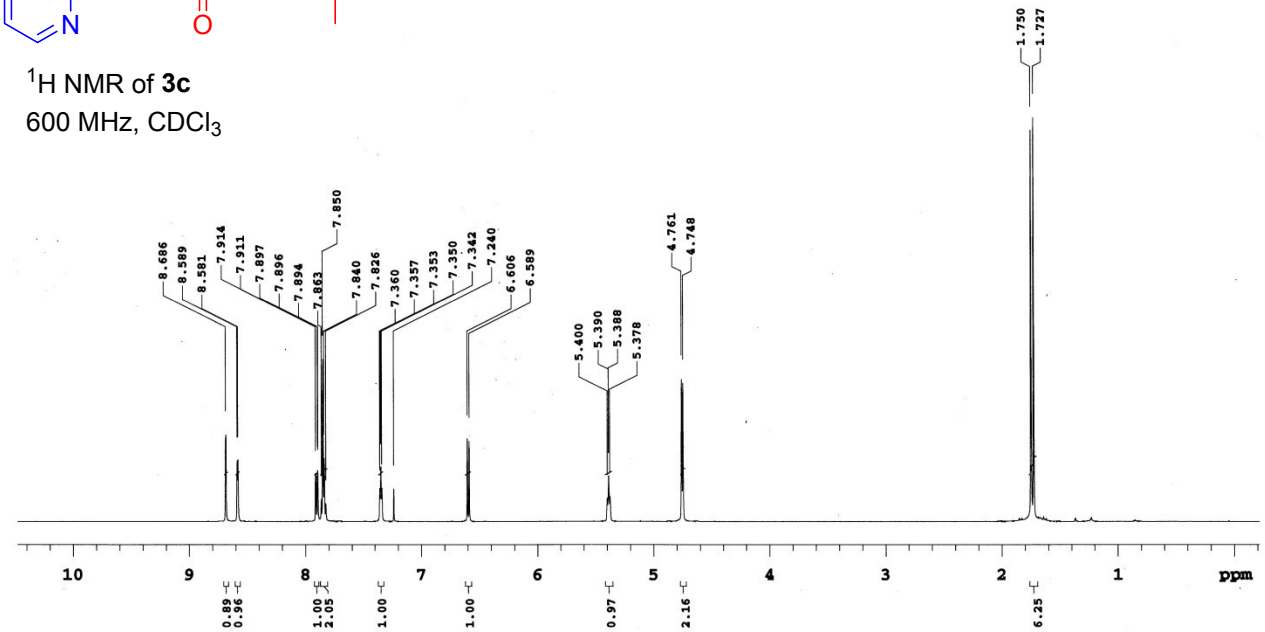


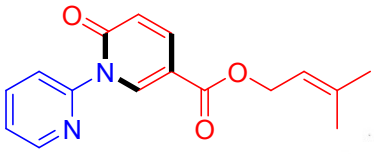
^{13}C NMR of **3d**
150 MHz, $CDCl_3$

3-Methylbut-2-en-1-yl 2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3c)



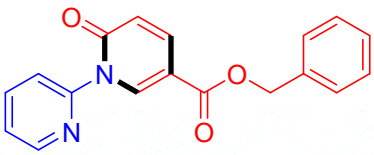
^{1}H NMR of **3c**
600 MHz, $CDCl_3$



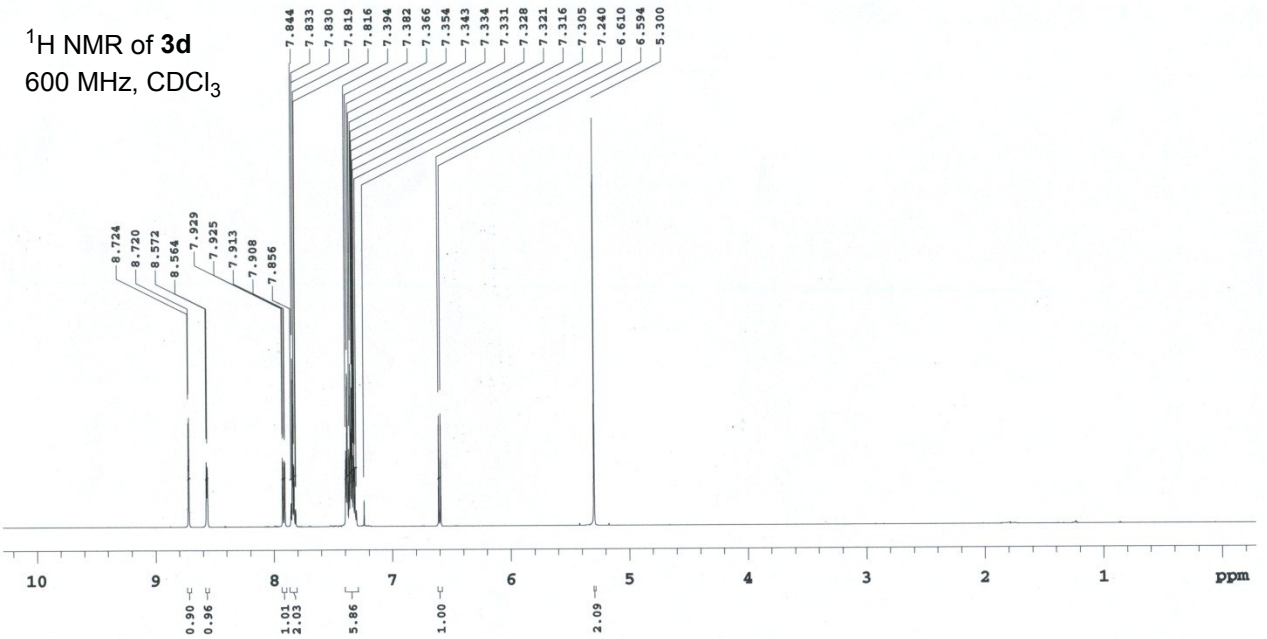


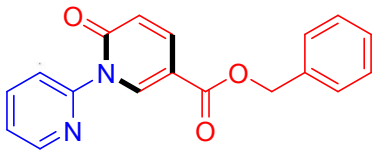
^{13}C NMR of **3c**
150 MHz, $CDCl_3$

Benzyl 2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3d)



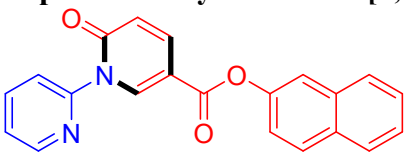
^{1}H NMR of **3d**
600 MHz, $CDCl_3$



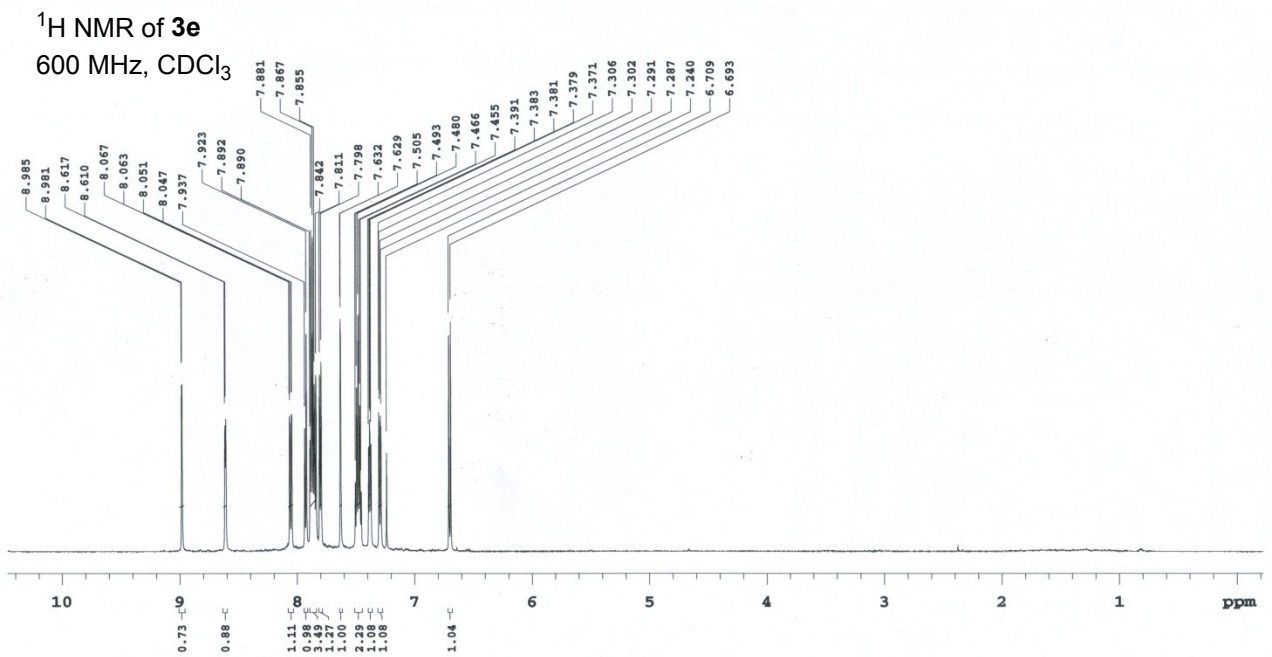


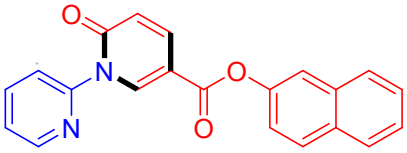
^{13}C NMR of **3d**
150 MHz, $CDCl_3$

Naphthalen-2-yl 2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3e)



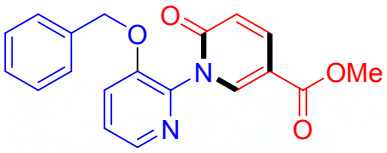
^{1}H NMR of **3e**
600 MHz, $CDCl_3$



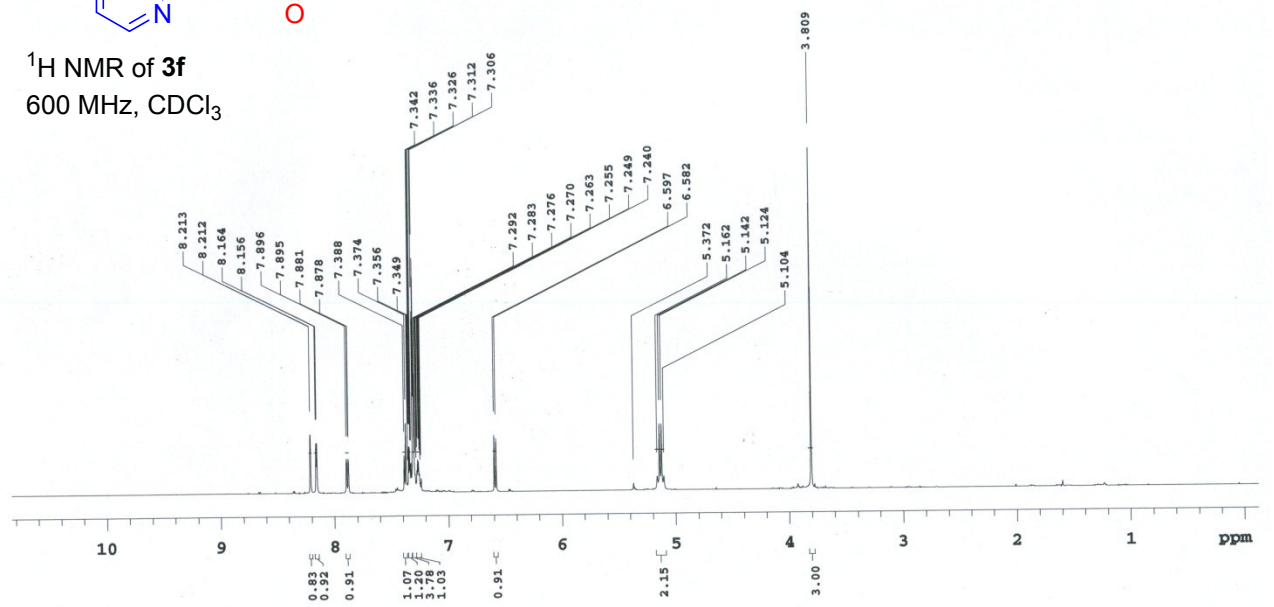


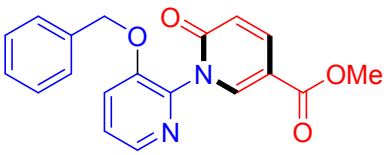
^{13}C NMR of **3e**
150 MHz, $CDCl_3$

Methyl 4'-(benzyloxy)-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3f)

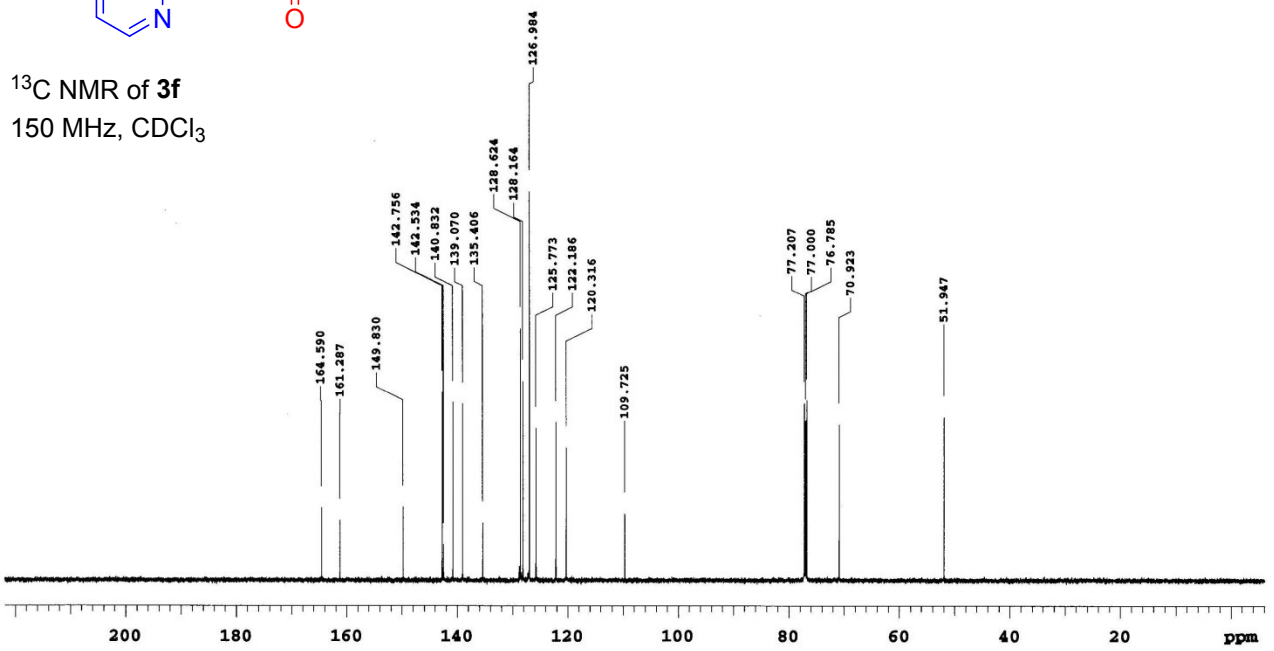


^{1}H NMR of **3f**
600 MHz, $CDCl_3$

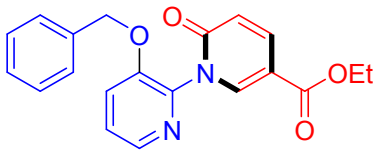




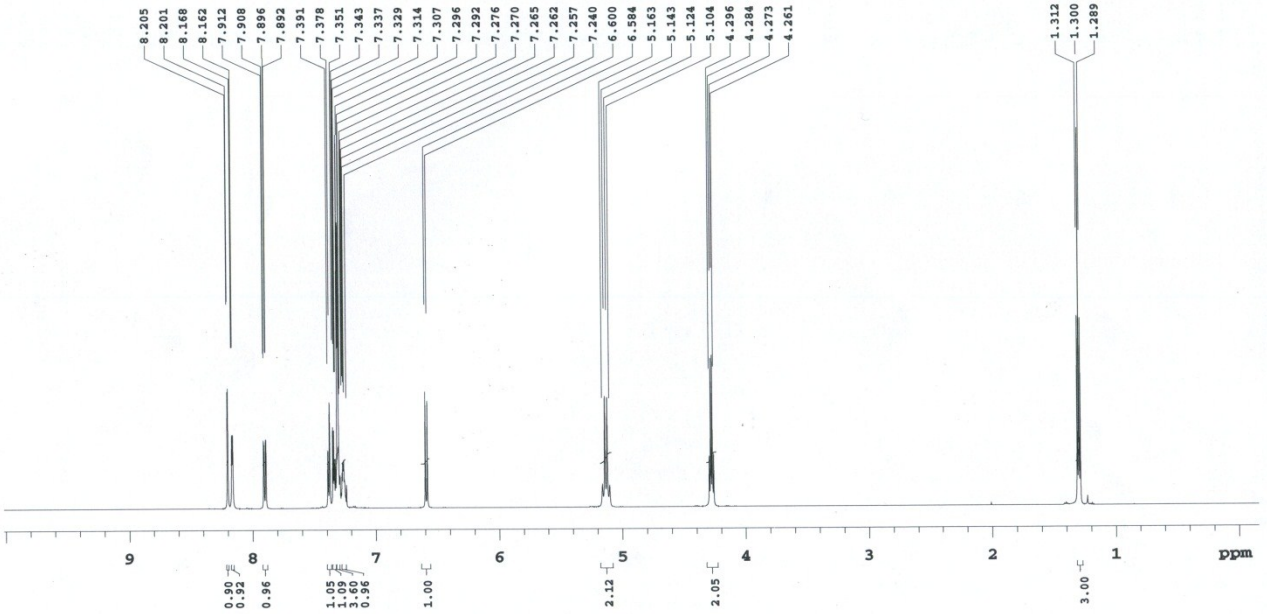
^{13}C NMR of **3f**
150 MHz, $CDCl_3$

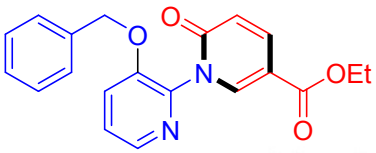


Ethyl 4'-(benzyloxy)-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3g)

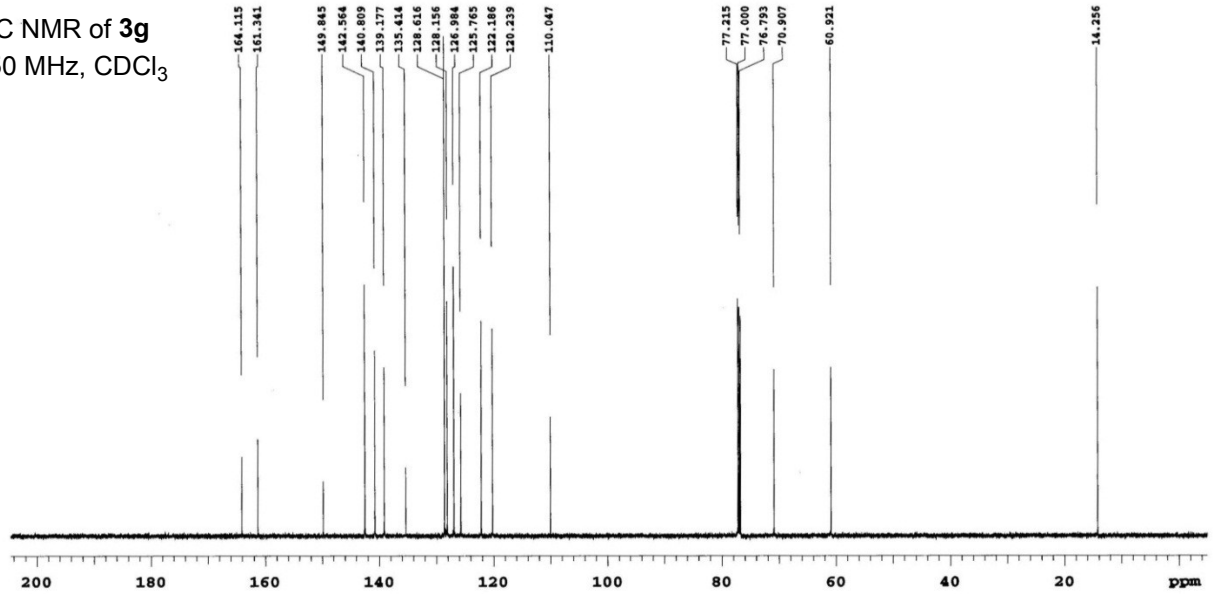


^{1}H NMR of **3g**
600 MHz, $CDCl_3$

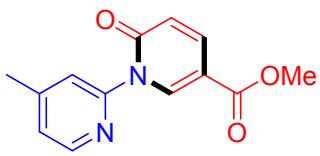




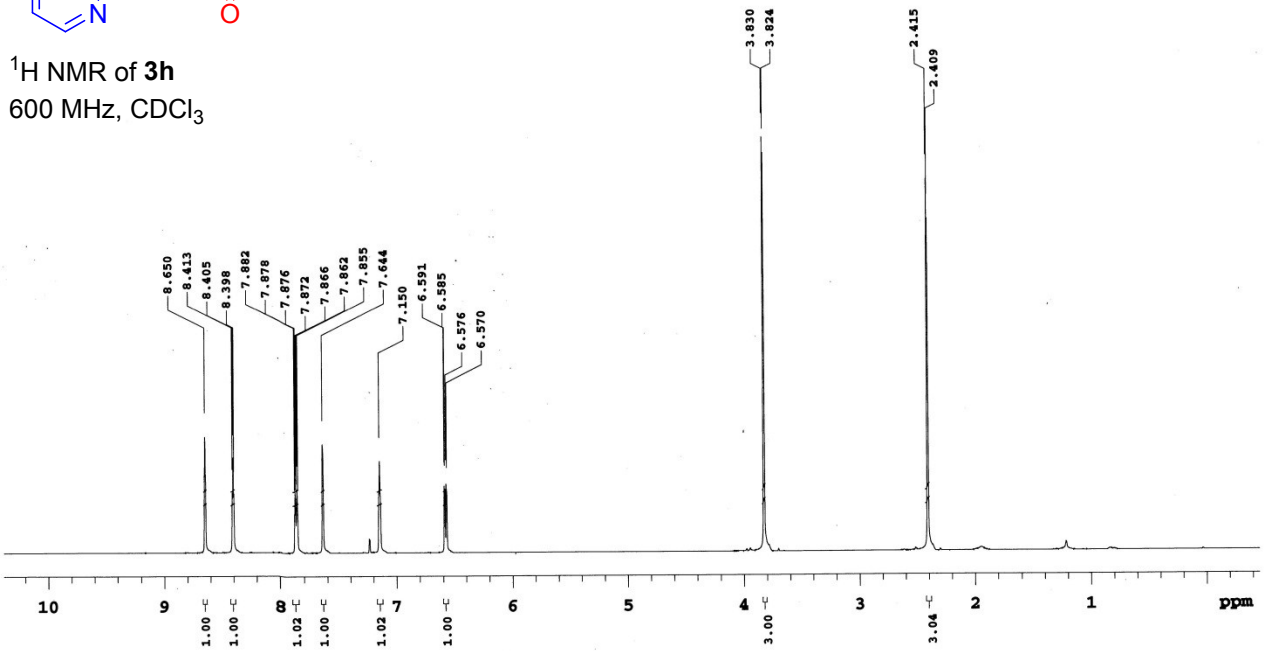
^{13}C NMR of **3g**
150 MHz, $CDCl_3$

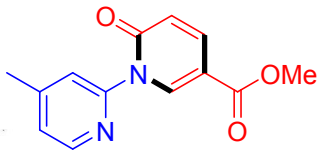


Methyl 4'-methyl-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3h)

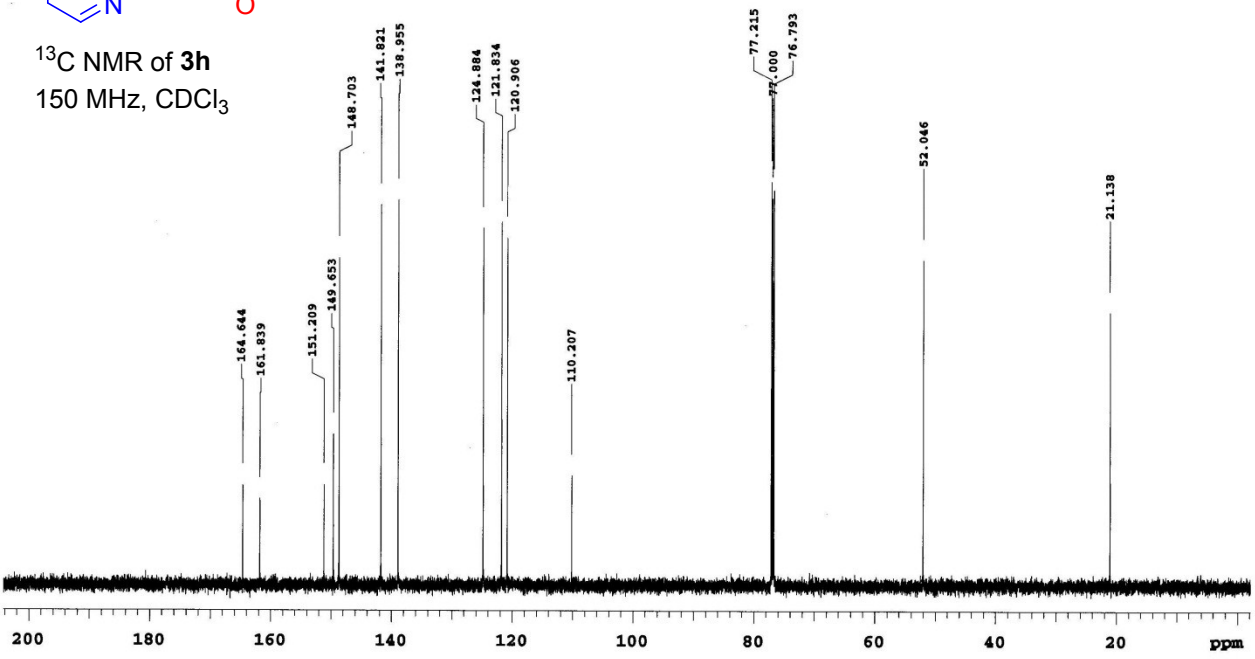


^{1}H NMR of **3h**
600 MHz, $CDCl_3$

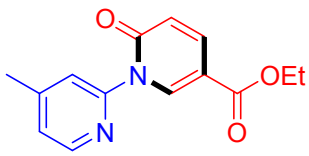




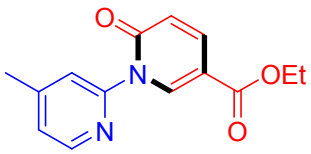
^{13}C NMR of **3h**
150 MHz, $CDCl_3$



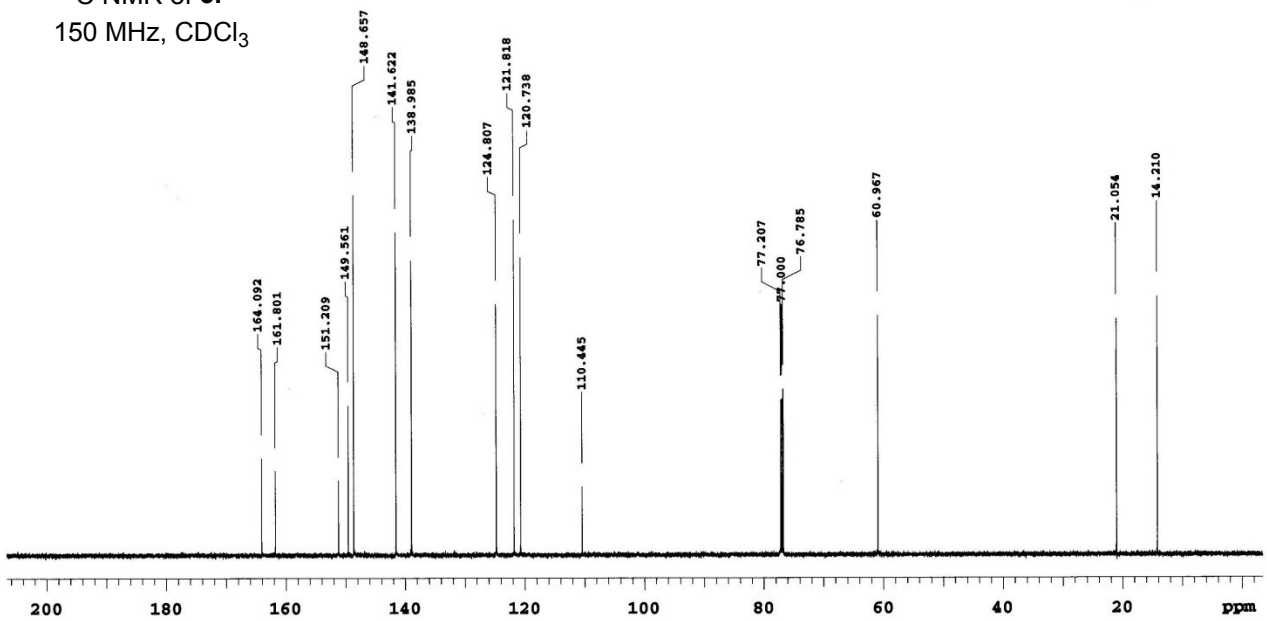
Ethyl 4'-methyl-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3i)



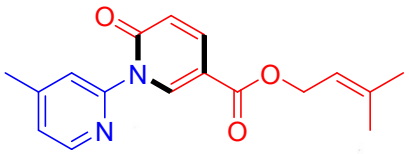
^{1}H NMR of **3i**
600 MHz, $CDCl_3$



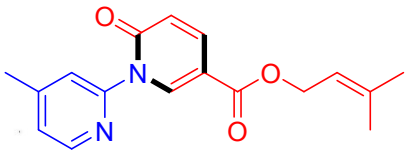
^{13}C NMR of **3i**
150 MHz, $CDCl_3$



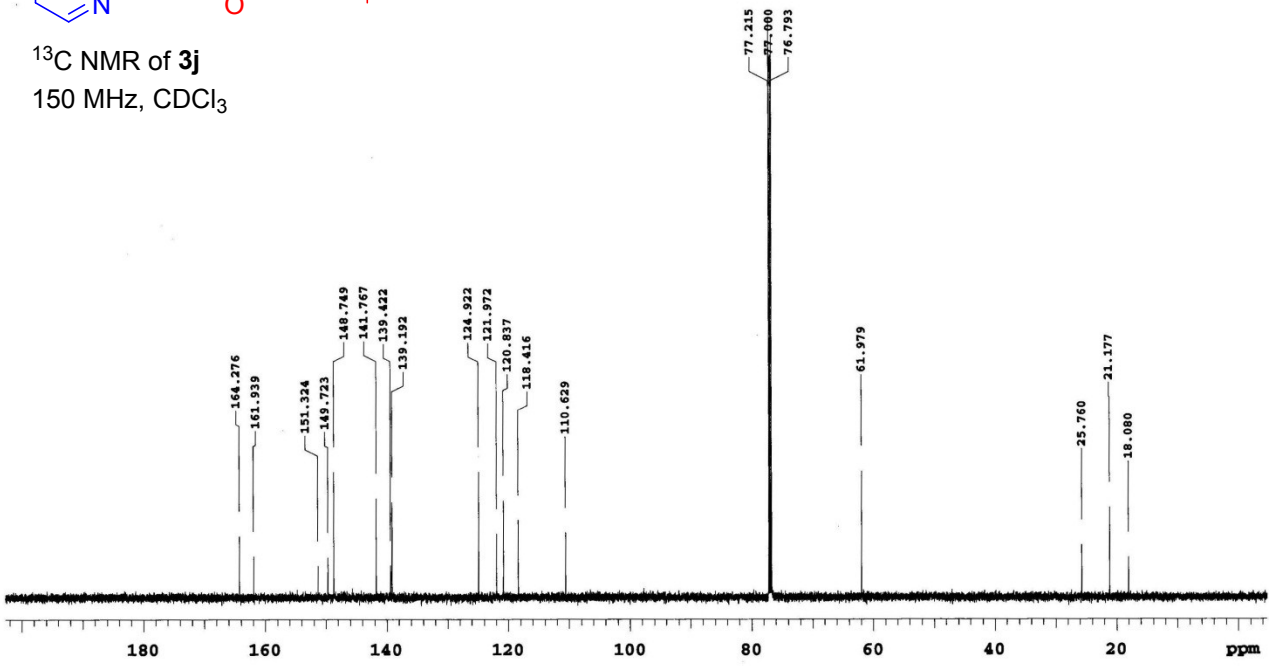
3-Methylbut-2-en-1-yl 4'-methyl-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3j)



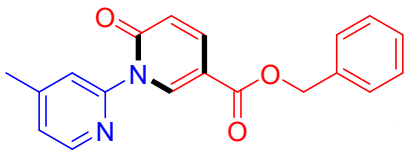
^{1}H NMR of **3j**
600 MHz, $CDCl_3$



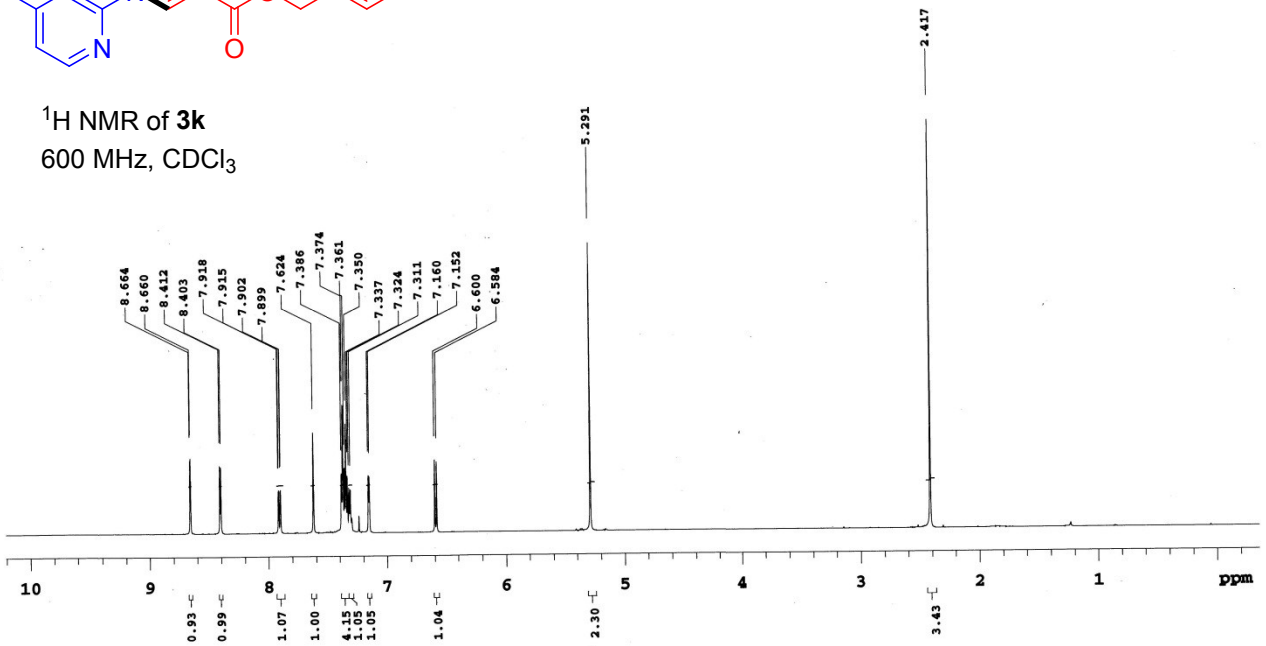
^{13}C NMR of **3j**
150 MHz, $CDCl_3$

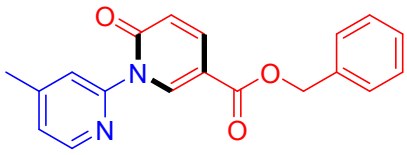


Benzyl 4'-methyl-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3k)

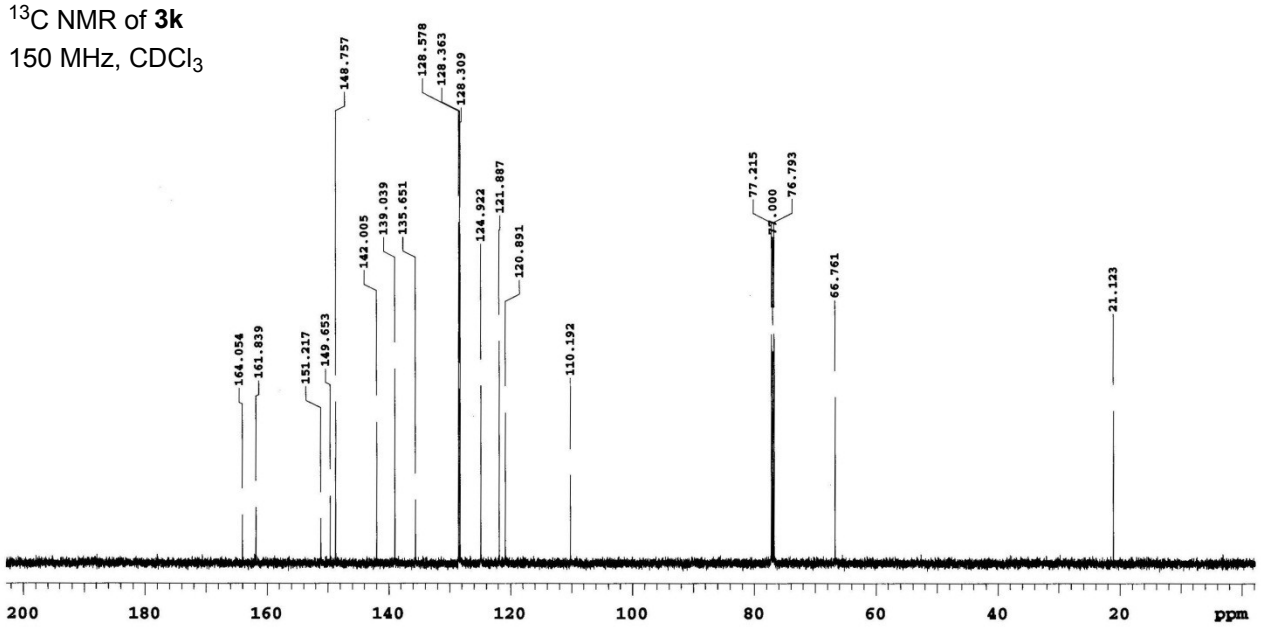


^{1}H NMR of **3k**
600 MHz, $CDCl_3$

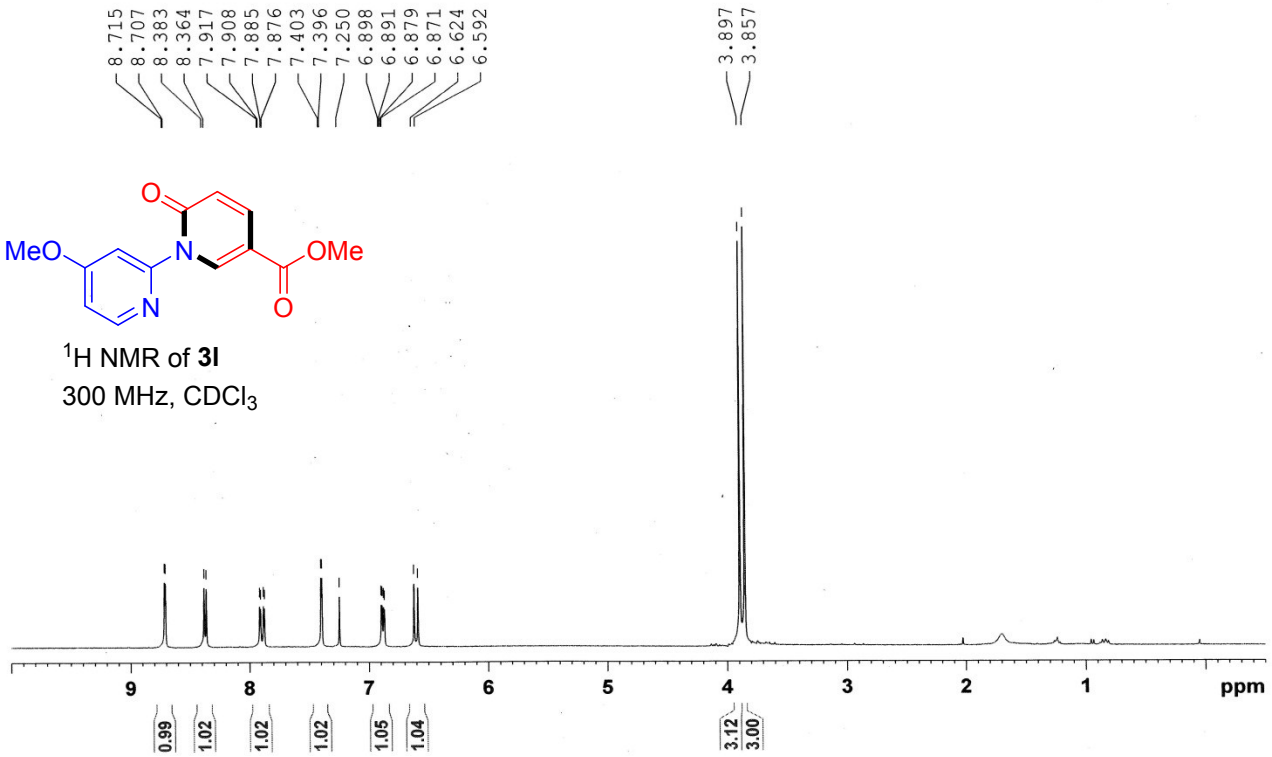




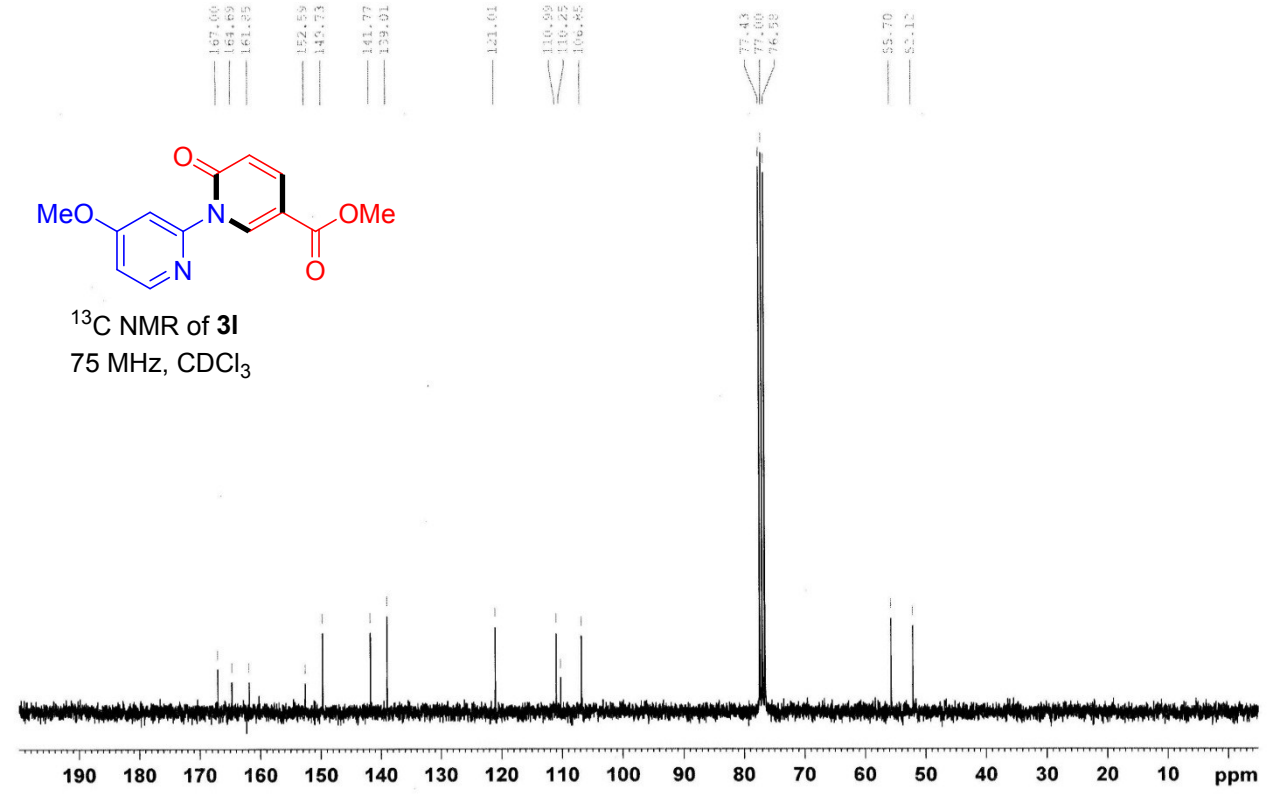
^{13}C NMR of **3k**
150 MHz, $CDCl_3$



Methyl 4'-methoxy-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3l)

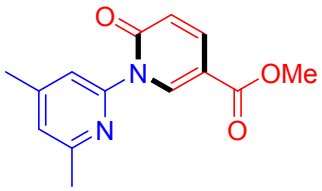


^{1}H NMR of **3l**
300 MHz, $CDCl_3$

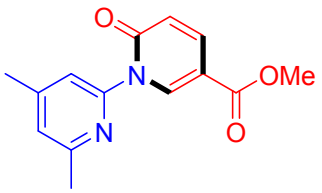


^{13}C NMR of **3l**
75 MHz, $CDCl_3$

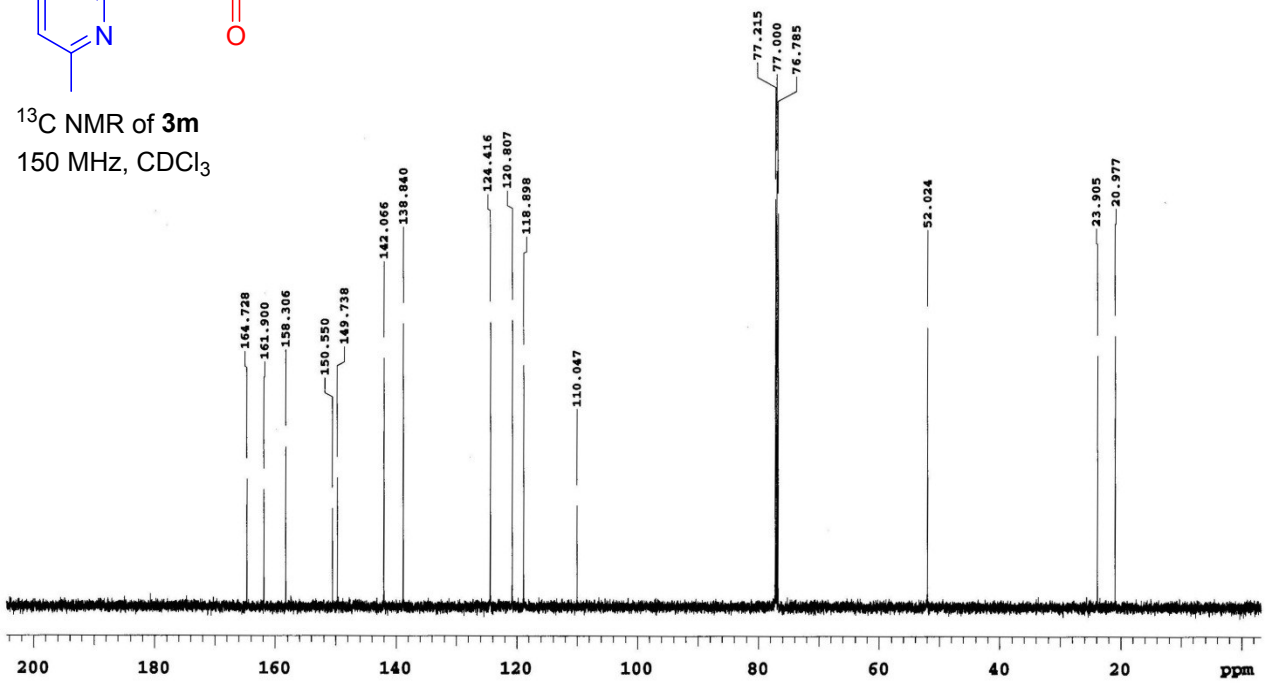
Methyl 4',6'-dimethyl-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3m)



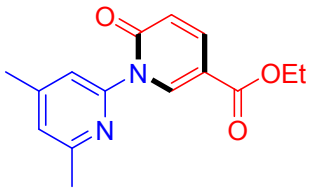
^{1}H NMR of **3m**
600 MHz, $CDCl_3$



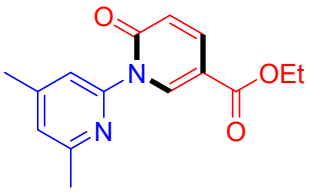
^{13}C NMR of **3m**
150 MHz, $CDCl_3$



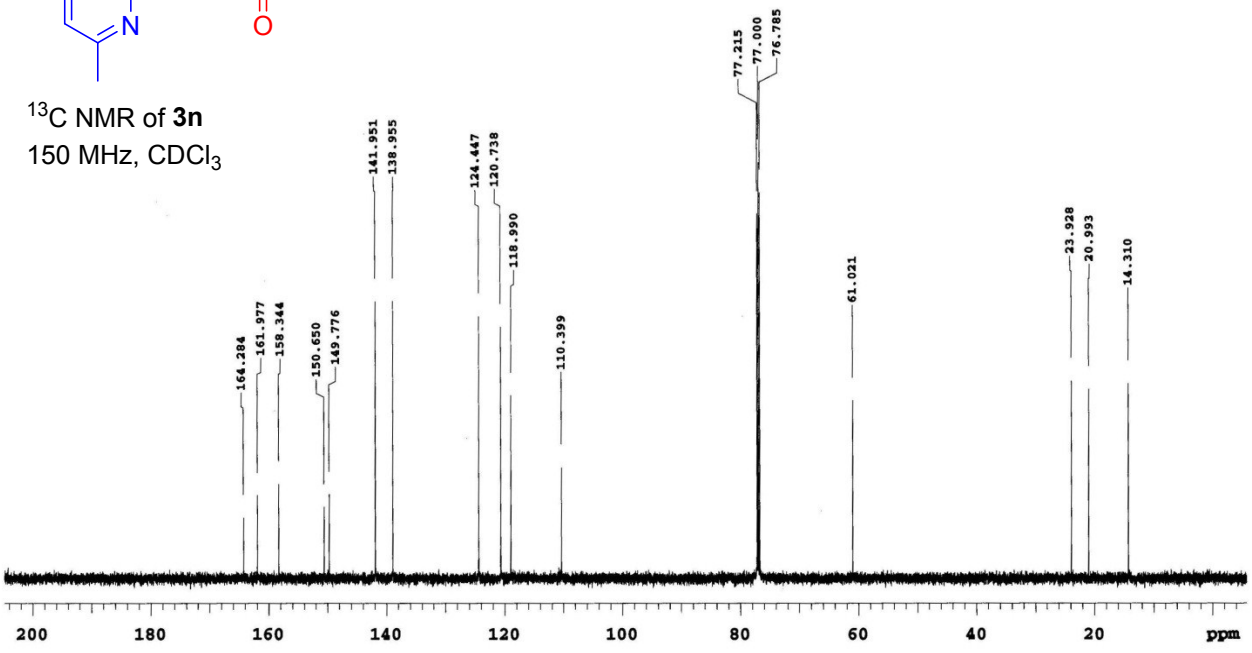
Ethyl 4',6'-dimethyl-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3n)



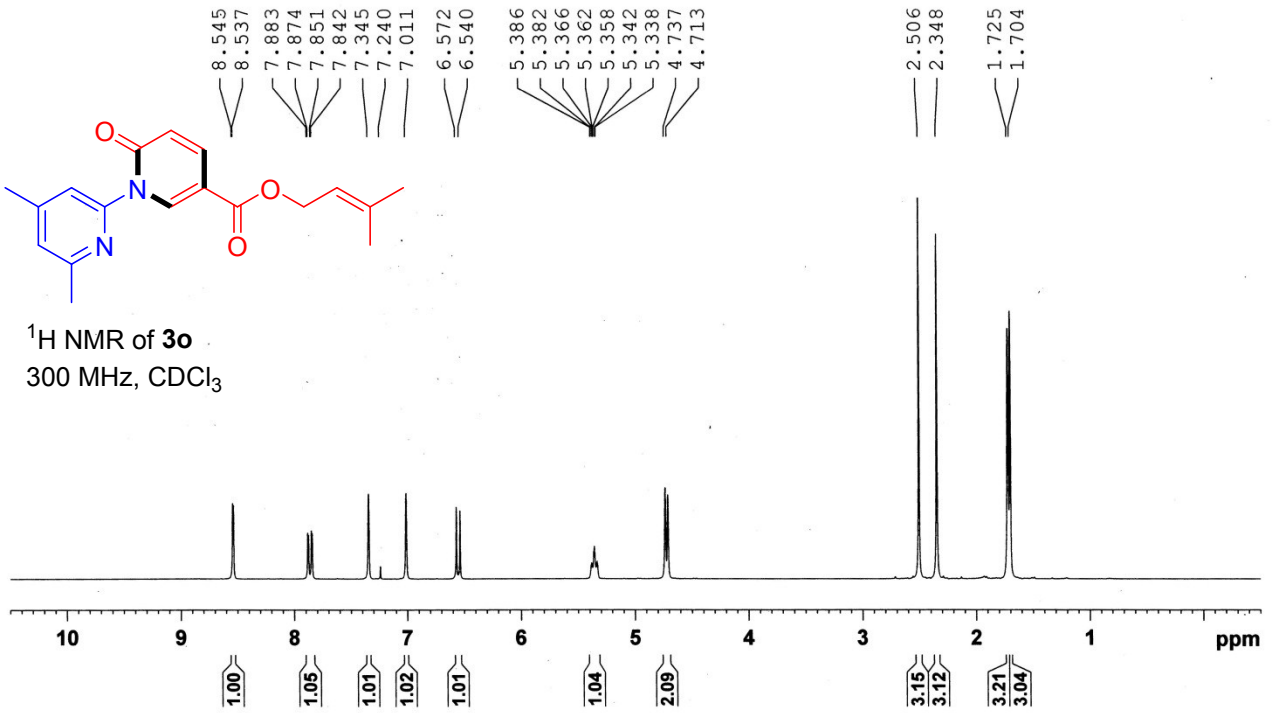
^{1}H NMR of **3n**
600 MHz, $CDCl_3$



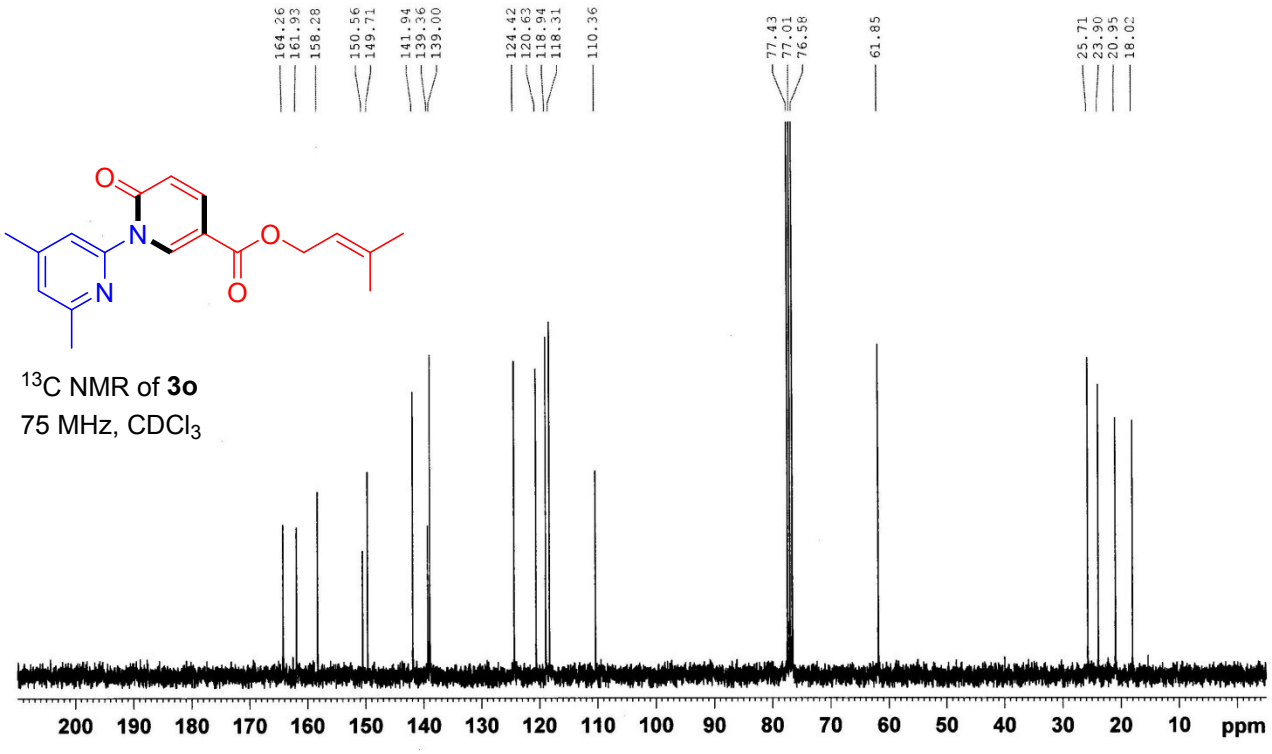
^{13}C NMR of **3n**
150 MHz, $CDCl_3$



3-Methylbut-2-en-1-yl 4',6'-dimethyl-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3o)

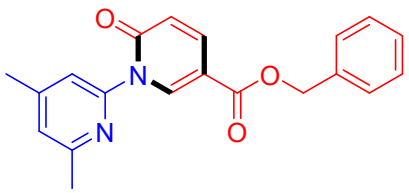


^{1}H NMR of **3o**
300 MHz, $CDCl_3$

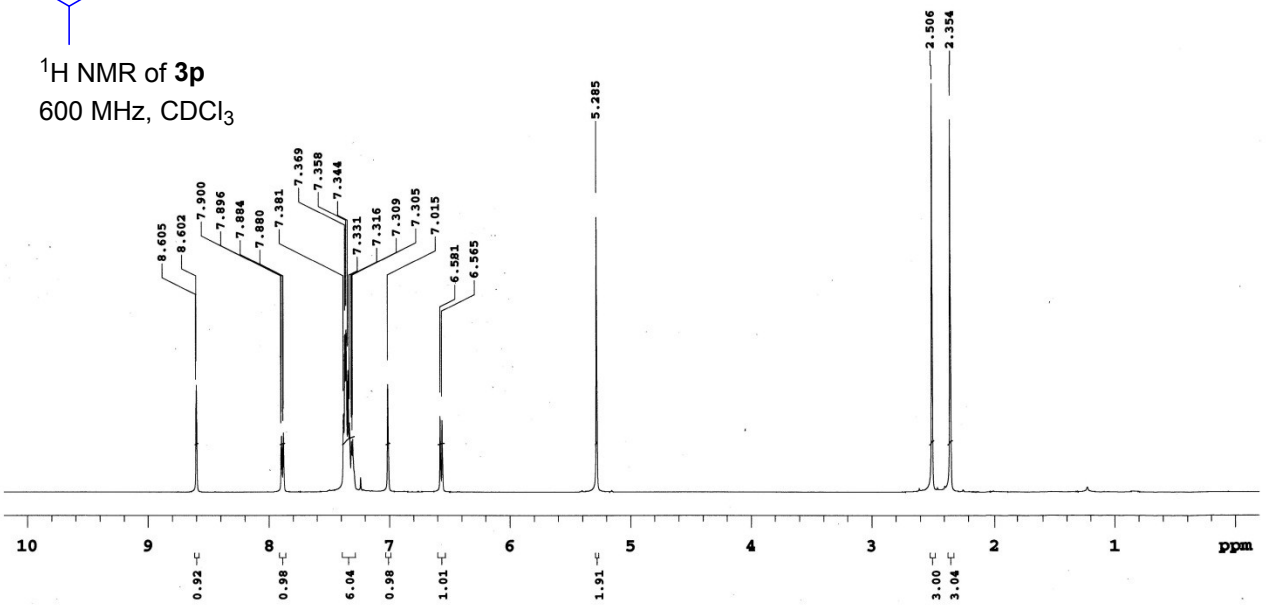


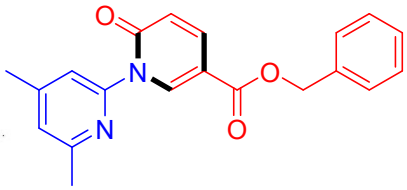
^{13}C NMR of **3o**
75 MHz, $CDCl_3$

Benzyl 4',6'-dimethyl-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3p)

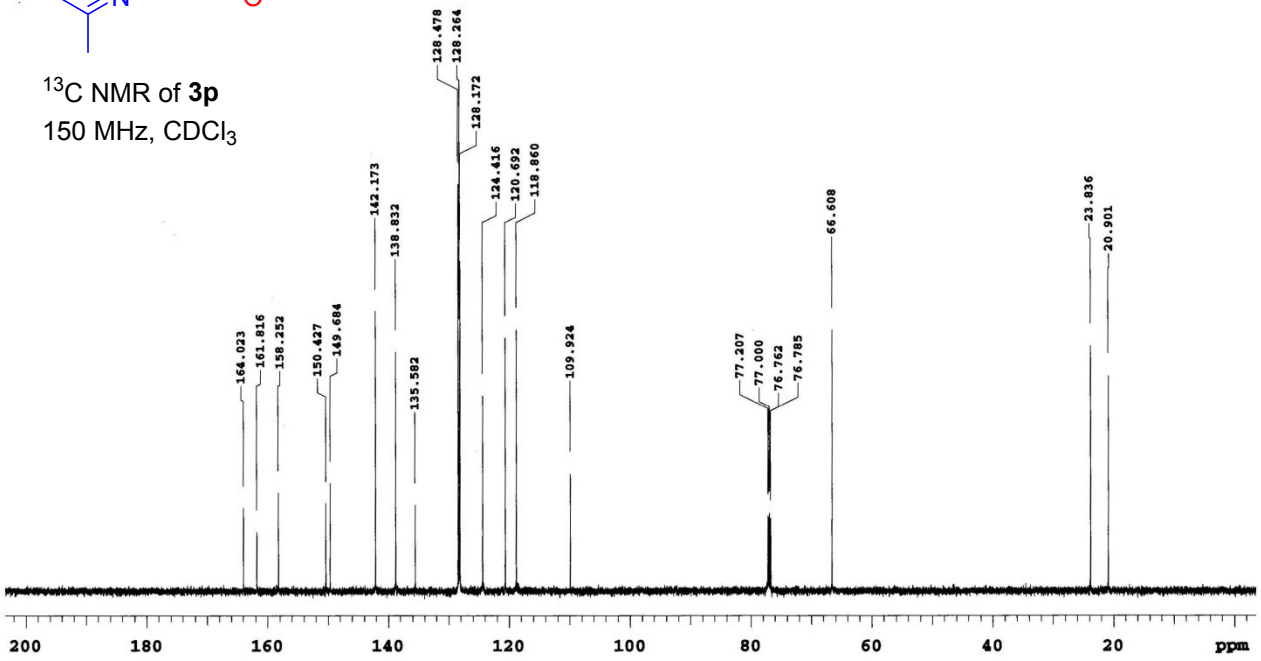


^{1}H NMR of **3p**
600 MHz, $CDCl_3$

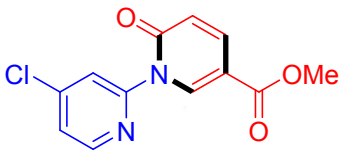




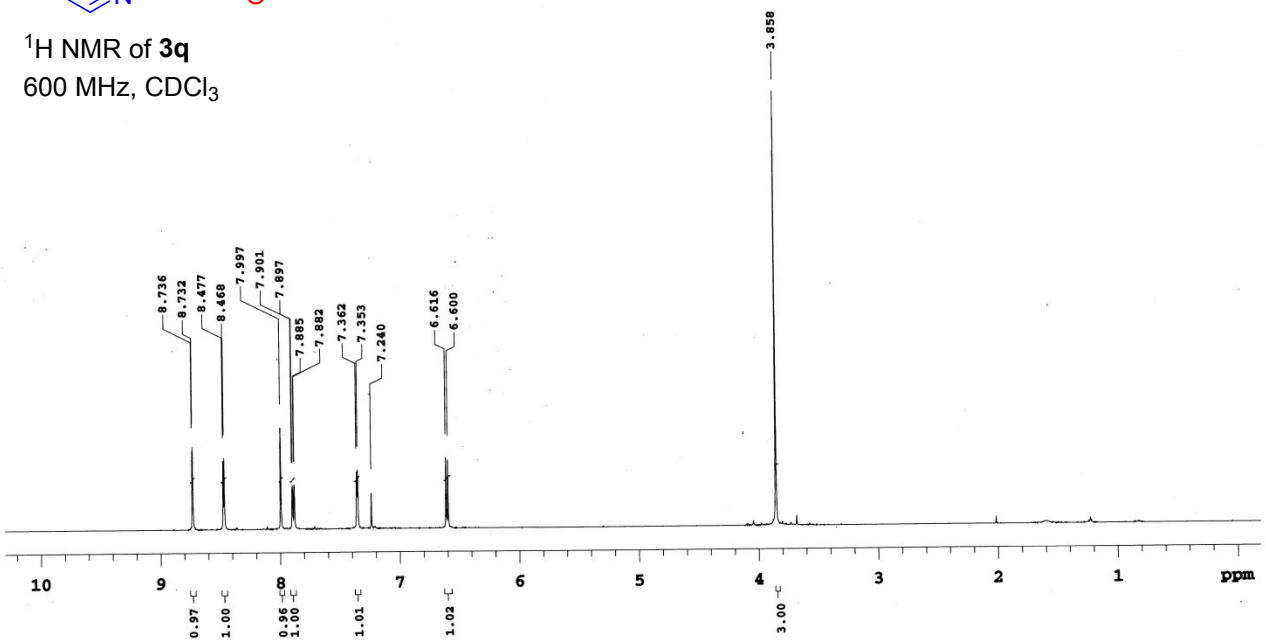
^{13}C NMR of **3p**
150 MHz, $CDCl_3$

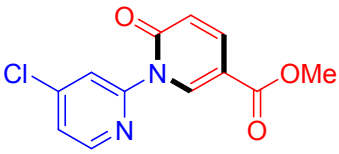


Methyl 4'-chloro-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3q)



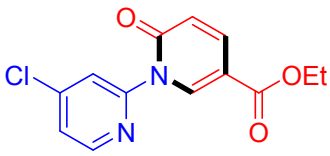
^{1}H NMR of **3q**
600 MHz, $CDCl_3$



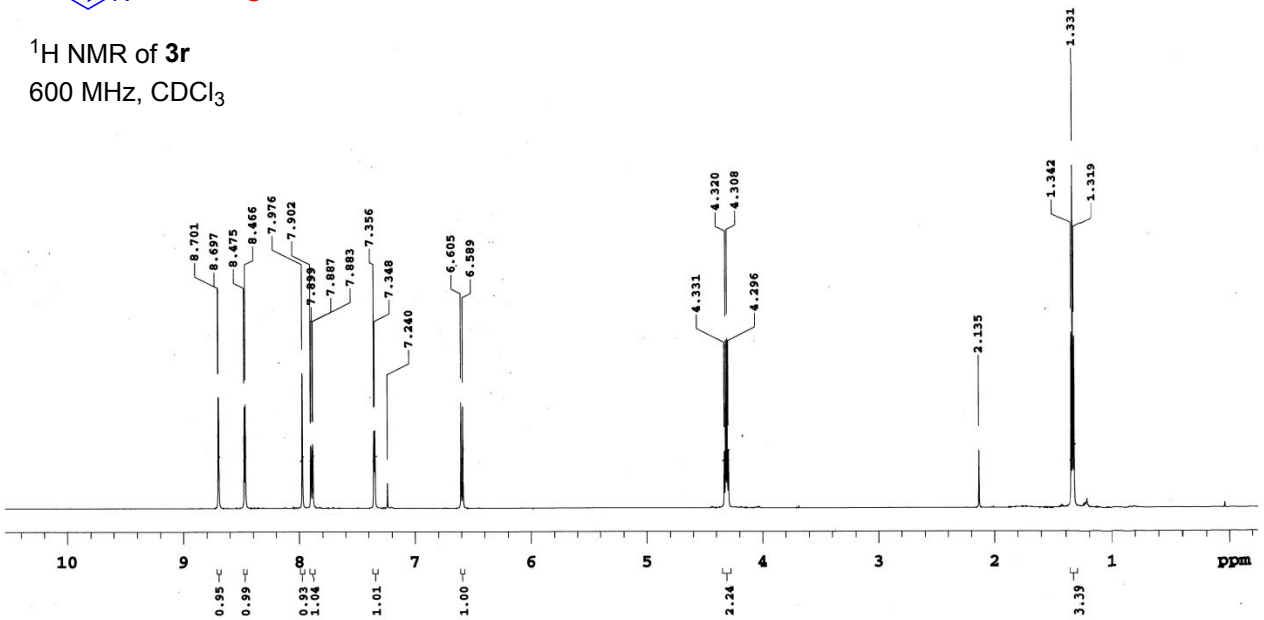


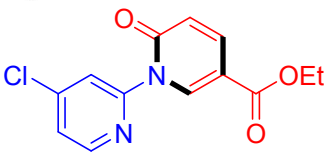
^{13}C NMR of **3q**
150 MHz, CDCl3

Ethyl 4'-chloro-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3r)



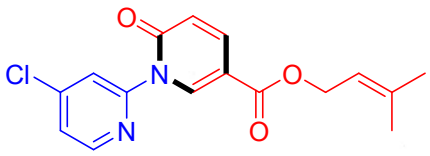
^{1}H NMR of **3r**
600 MHz, $CDCl_3$



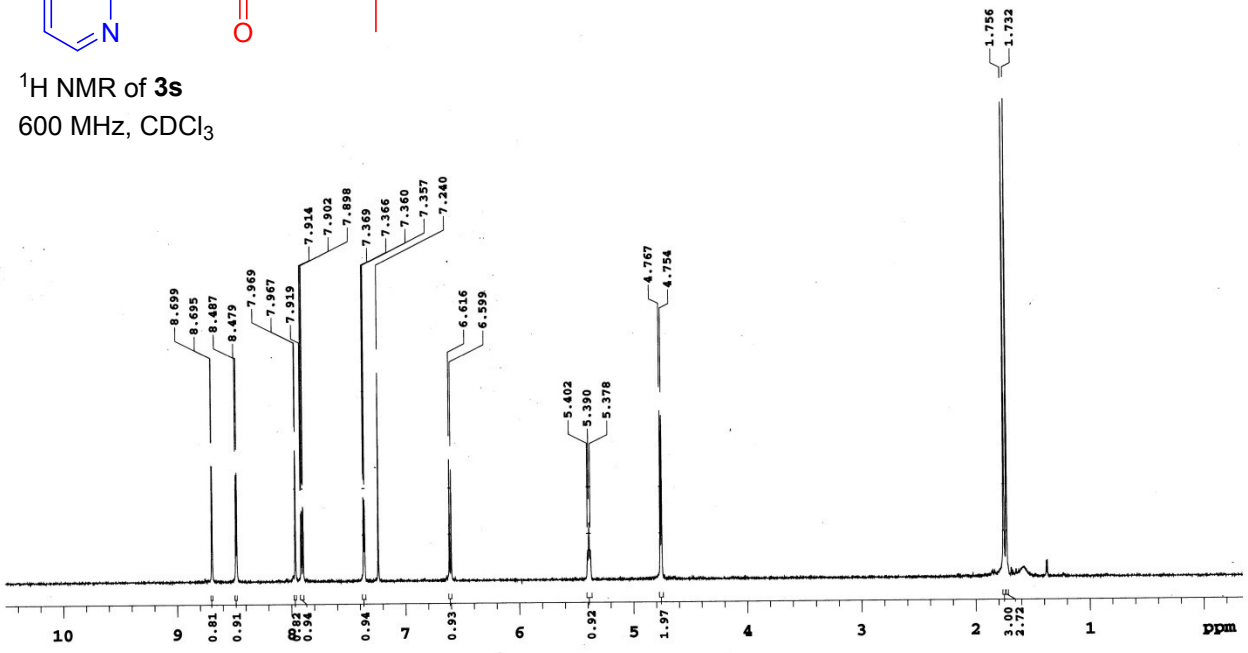


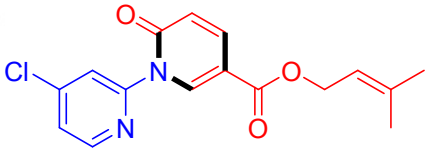
^{13}C NMR of **3r**
150 MHz, $CDCl_3$

3-Methylbut-2-en-1-yl 4'-chloro-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3s)



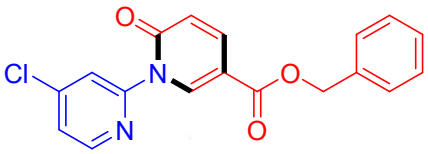
^{1}H NMR of **3s**
600 MHz, $CDCl_3$



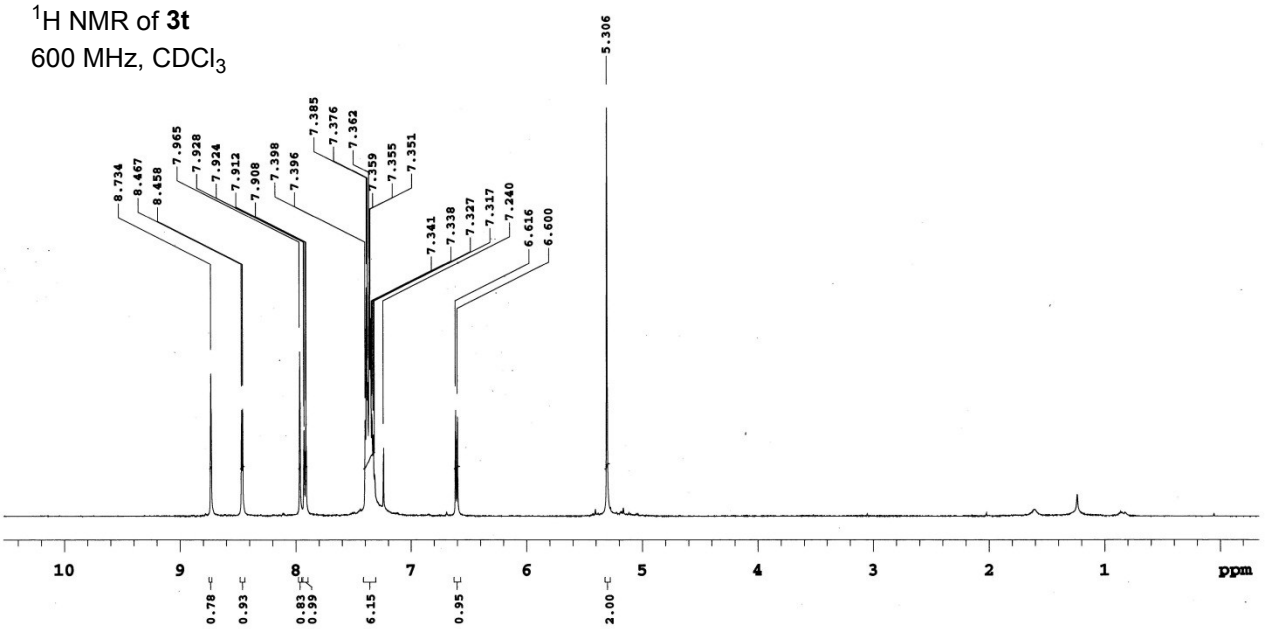


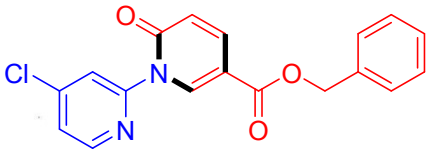
^{13}C NMR of **3s**
150 MHz, $CDCl_3$

Benzyl 4'-chloro-2-oxo-2*H*-[1,2'-bipyridine]-5-carboxylate (3t)

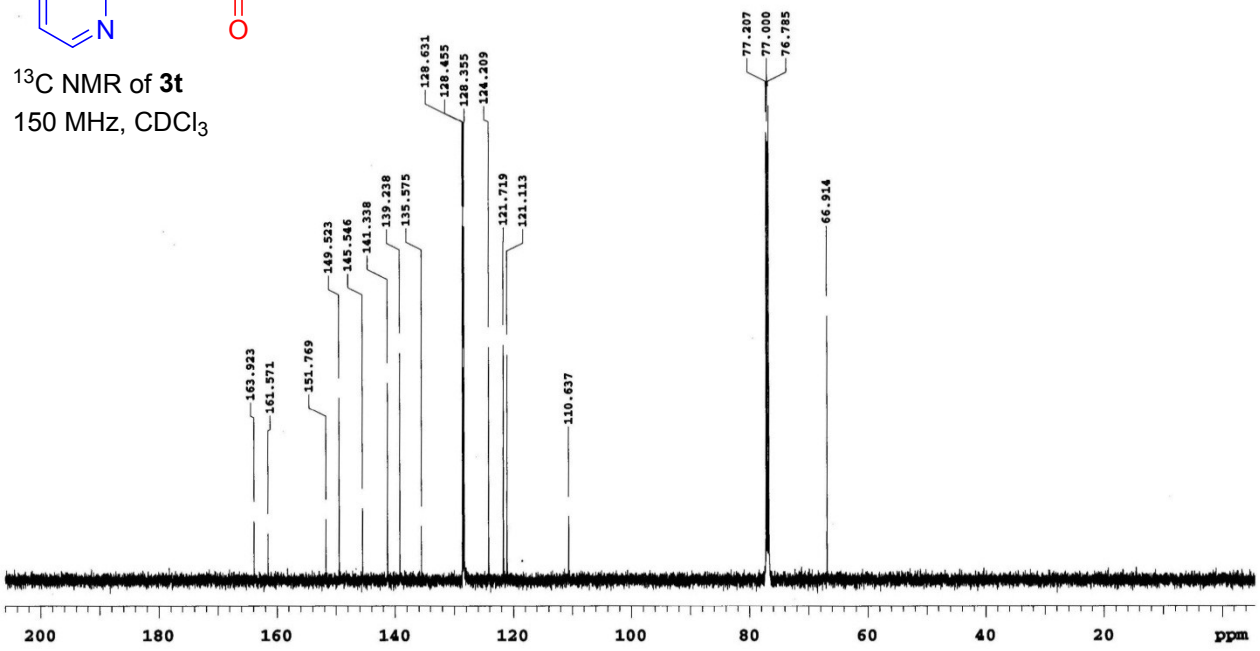


^{1}H NMR of **3t**
600 MHz, $CDCl_3$

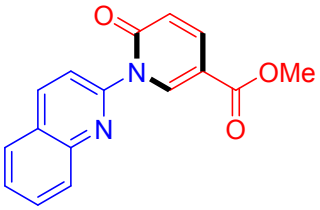




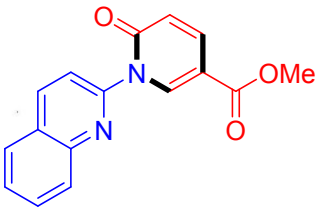
^{13}C NMR of **3t**
150 MHz, $CDCl_3$



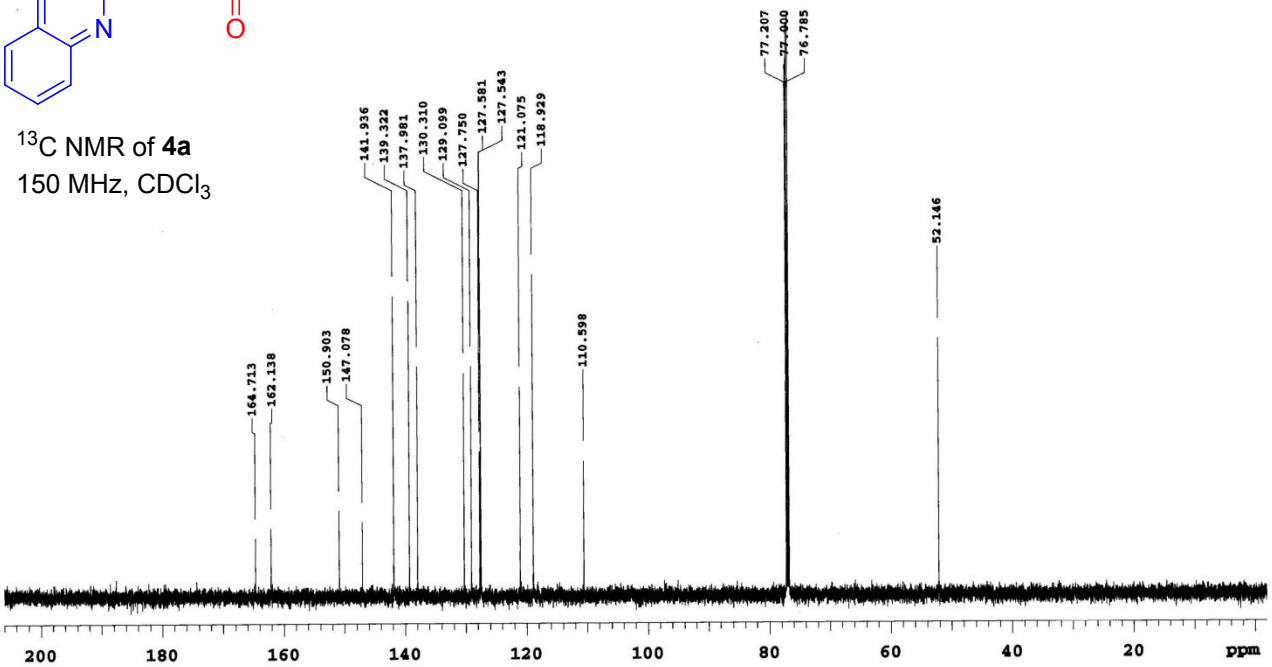
Methyl 6-oxo-1-(quinolin-2-yl)-1,6-dihydropyridine-3-carboxylate (4a)



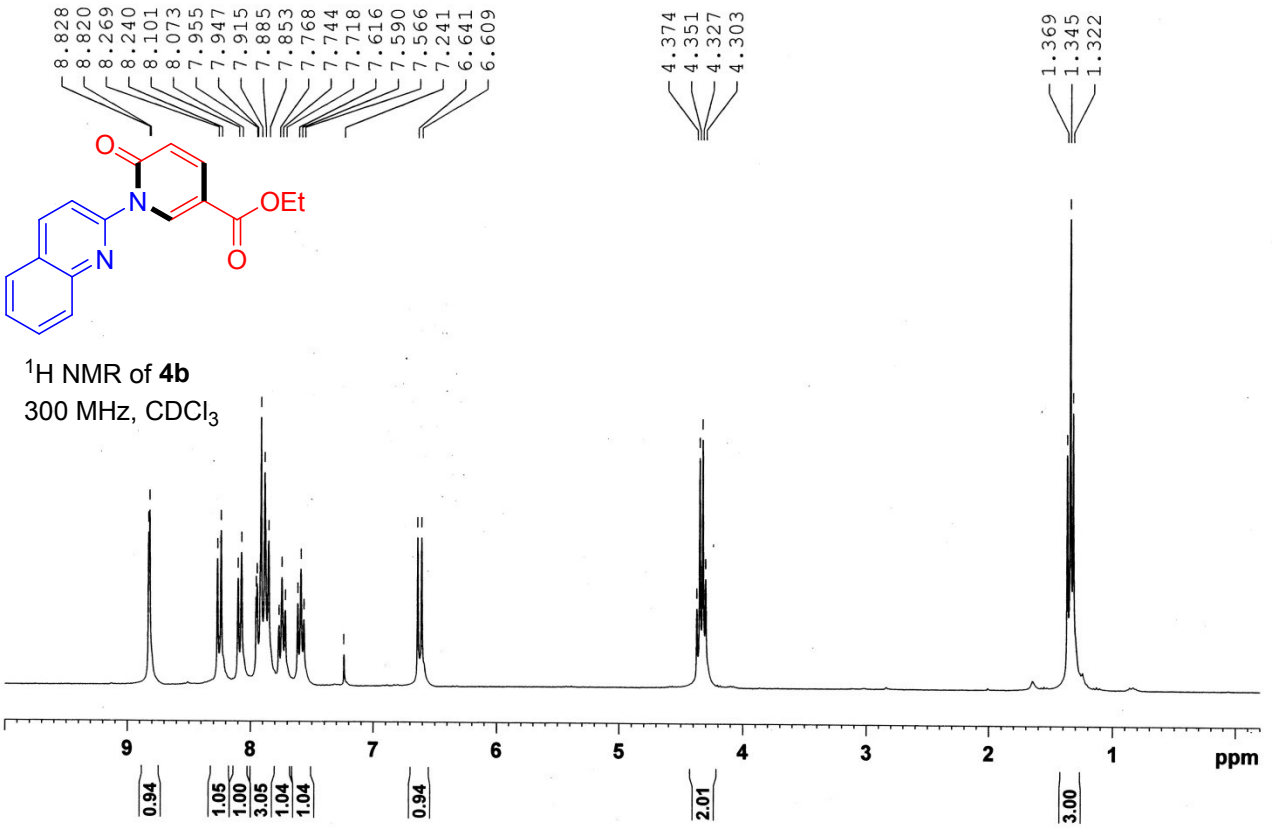
^{1}H NMR of **4a**
600 MHz, $CDCl_3$



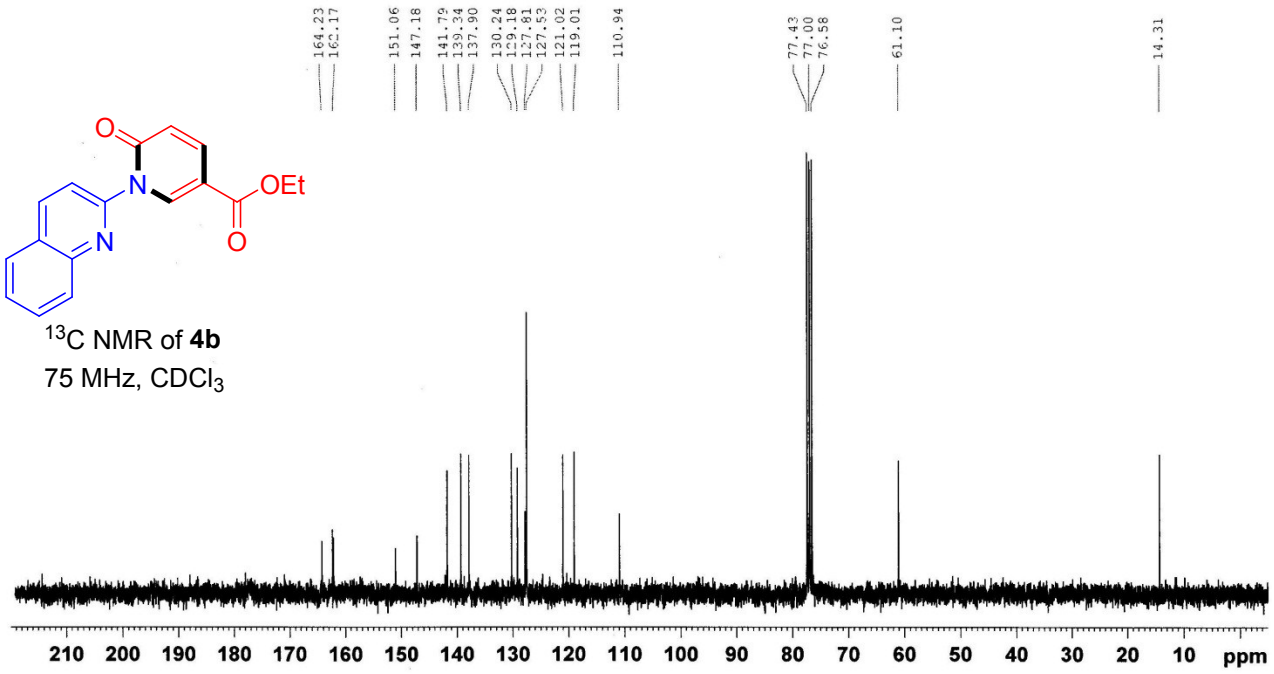
^{13}C NMR of **4a**
150 MHz, $CDCl_3$



Ethyl 6-oxo-1-(quinolin-2-yl)-1,6-dihydropyridine-3-carboxylate (4b)

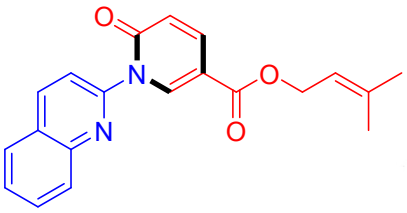


^{1}H NMR of **4b**
300 MHz, $CDCl_3$

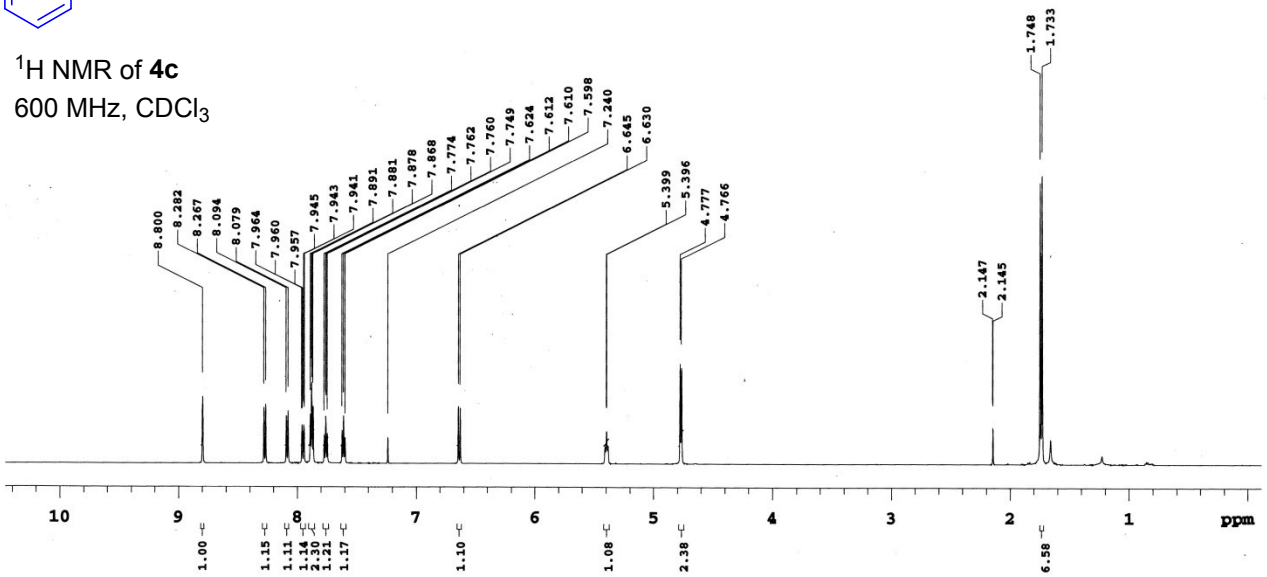


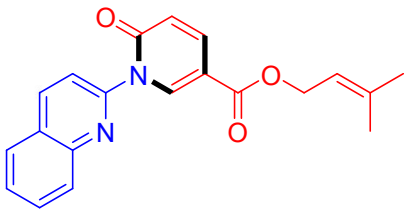
^{13}C NMR of **4b**
75 MHz, $CDCl_3$

3-Methylbut-2-en-1-yl 6-oxo-1-(quinolin-2-yl)-1,6-dihydropyridine-3-carboxylate (4c)



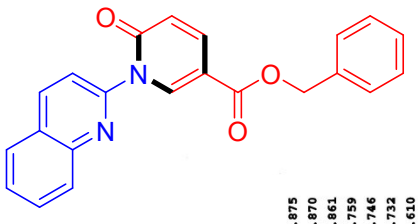
^{1}H NMR of **4c**
600 MHz, $CDCl_3$



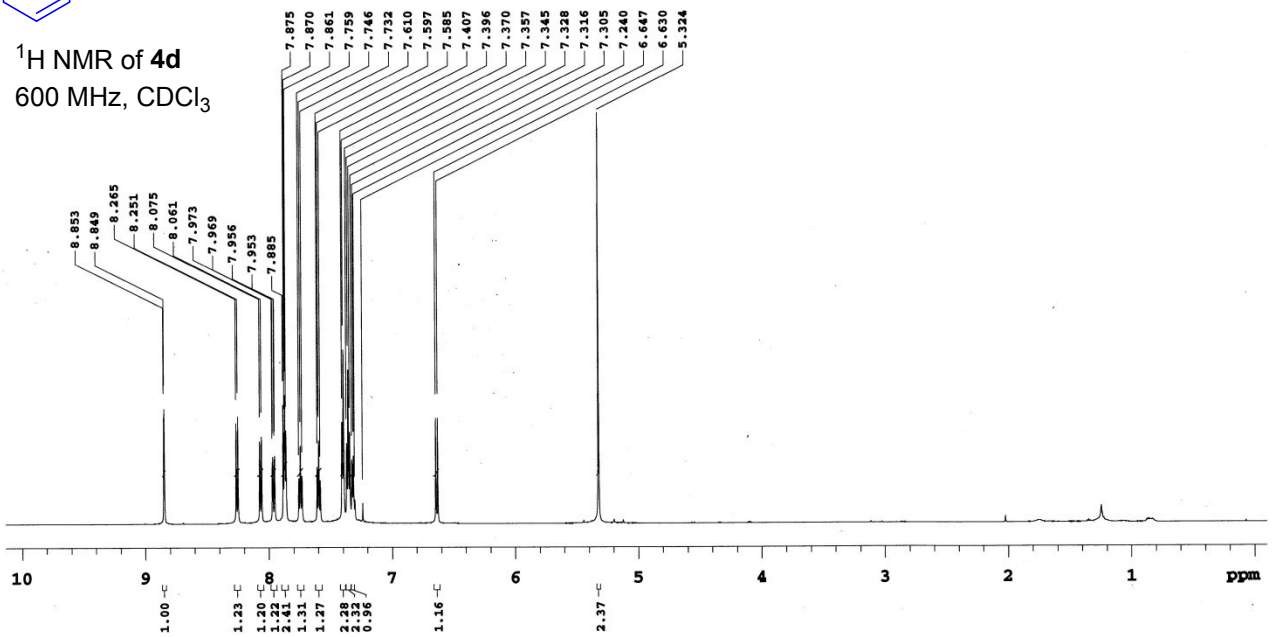


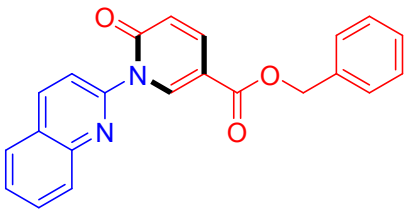
^{13}C NMR of **4c**
150 MHz, $CDCl_3$

Benzyl 6-oxo-1-(quinolin-2-yl)-1,6-dihydropyridine-3-carboxylate (4d)

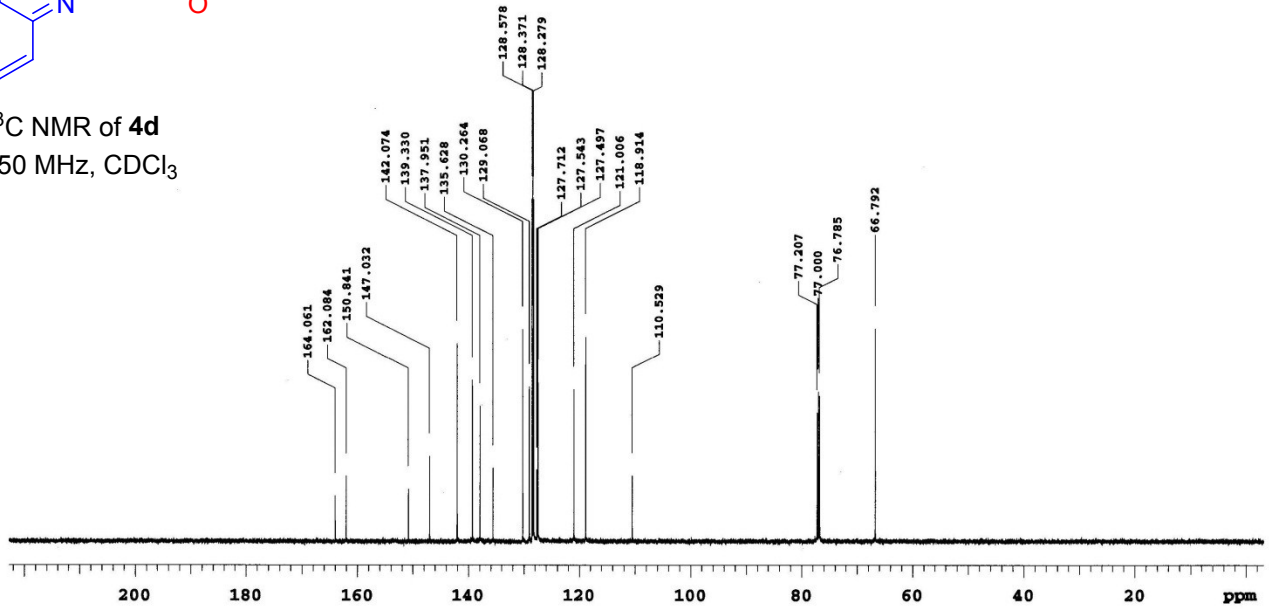


^{1}H NMR of **4d**
600 MHz, $CDCl_3$

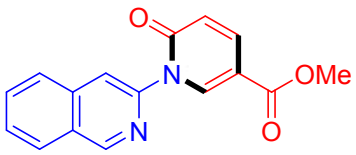




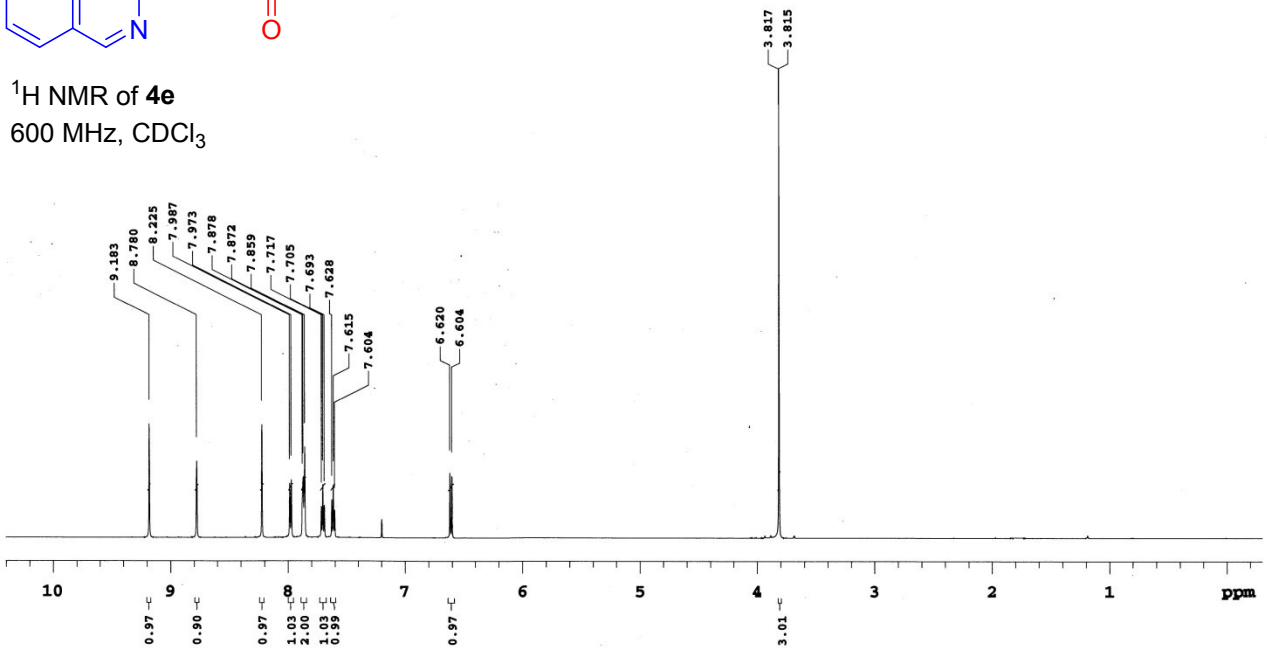
^{13}C NMR of **4d**
150 MHz, $CDCl_3$

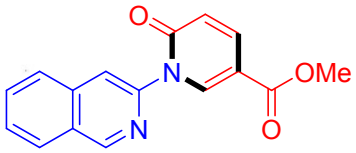


Methyl 1-(isoquinolin-3-yl)-6-oxo-1,6-dihydropyridine-3-carboxylate (4e)



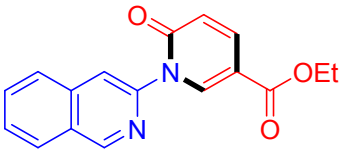
^{1}H NMR of **4e**
600 MHz, $CDCl_3$



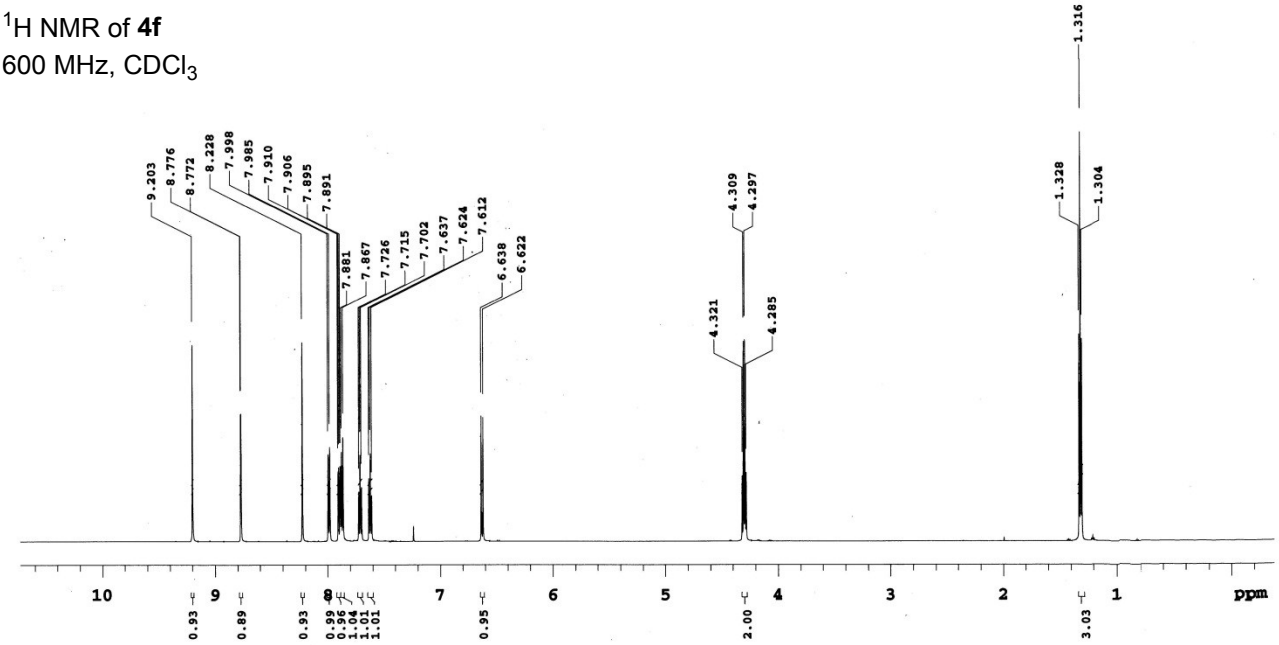


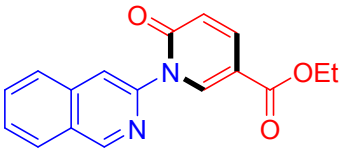
^{13}C NMR of **4e**
150 MHz, $CDCl_3$

Ethyl 1-(isoquinolin-3-yl)-6-oxo-1,6-dihydropyridine-3-carboxylate (4f)

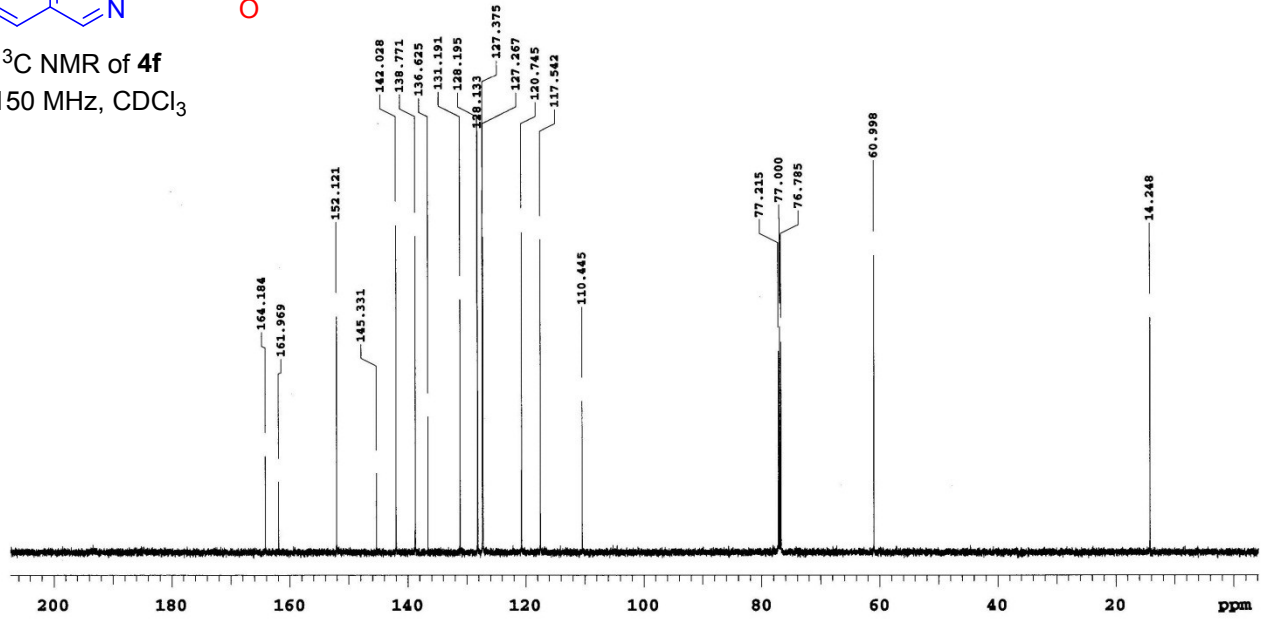


^{1}H NMR of **4f**
600 MHz, $CDCl_3$

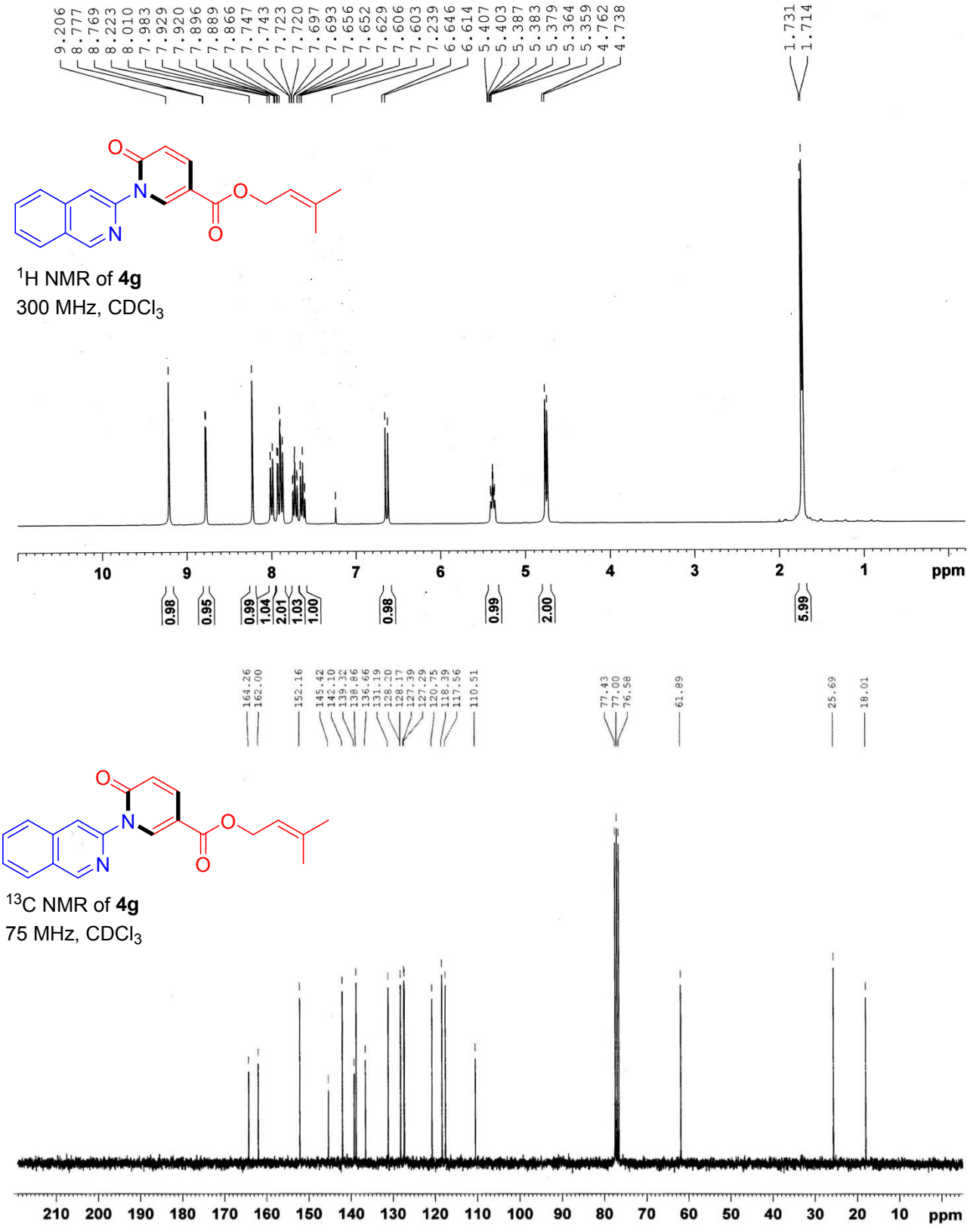




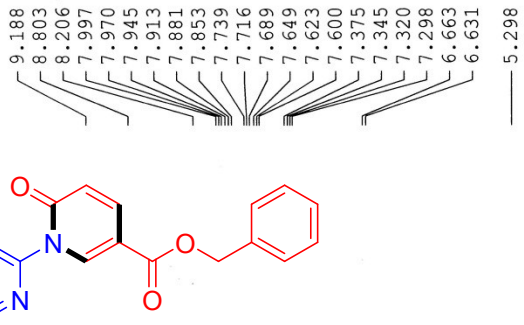
^{13}C NMR of **4f**
150 MHz, $CDCl_3$



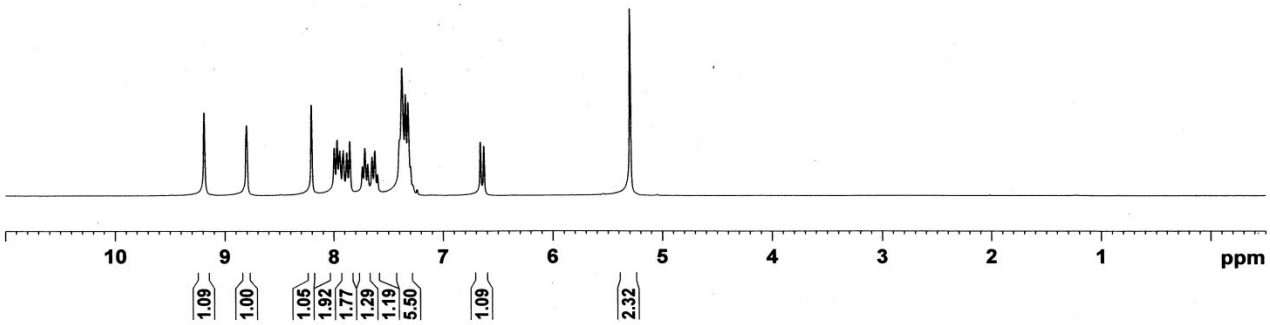
3-Methylbut-2-en-1-yl 1-(isoquinolin-3-yl)-6-oxo-1,6-dihydropyridine-3-carboxylate (4g)

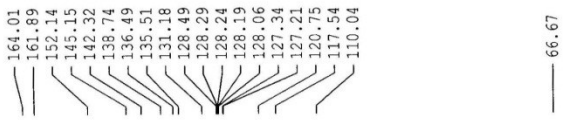


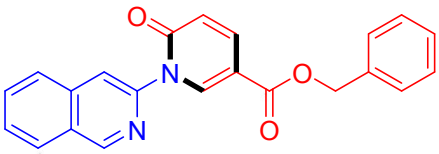
Benzyl 1-(isoquinolin-3-yl)-6-oxo-1,6-dihydropyridine-3-carboxylate (4h)



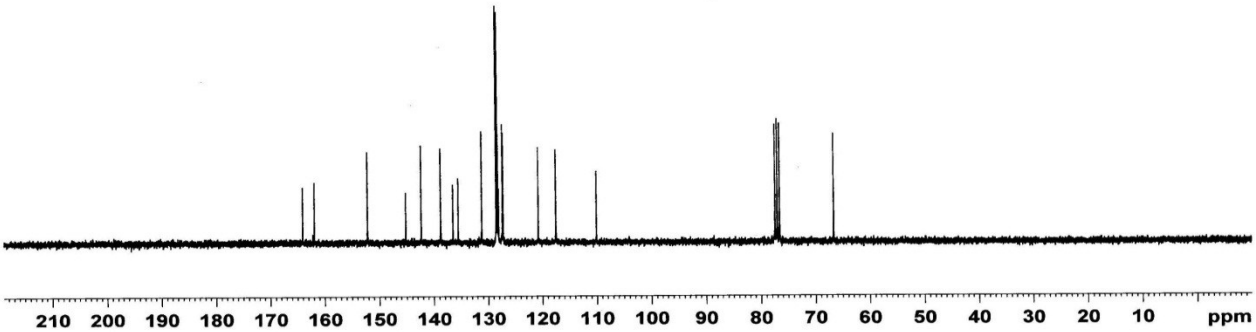
^{1}H NMR of **4h**
300 MHz, $CDCl_3$



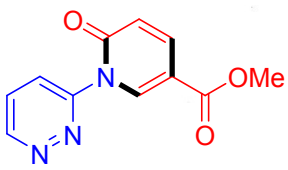




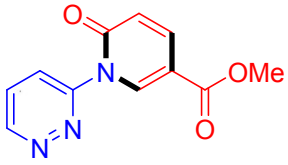
^{13}C NMR of **4h**
75 MHz, $CDCl_3$



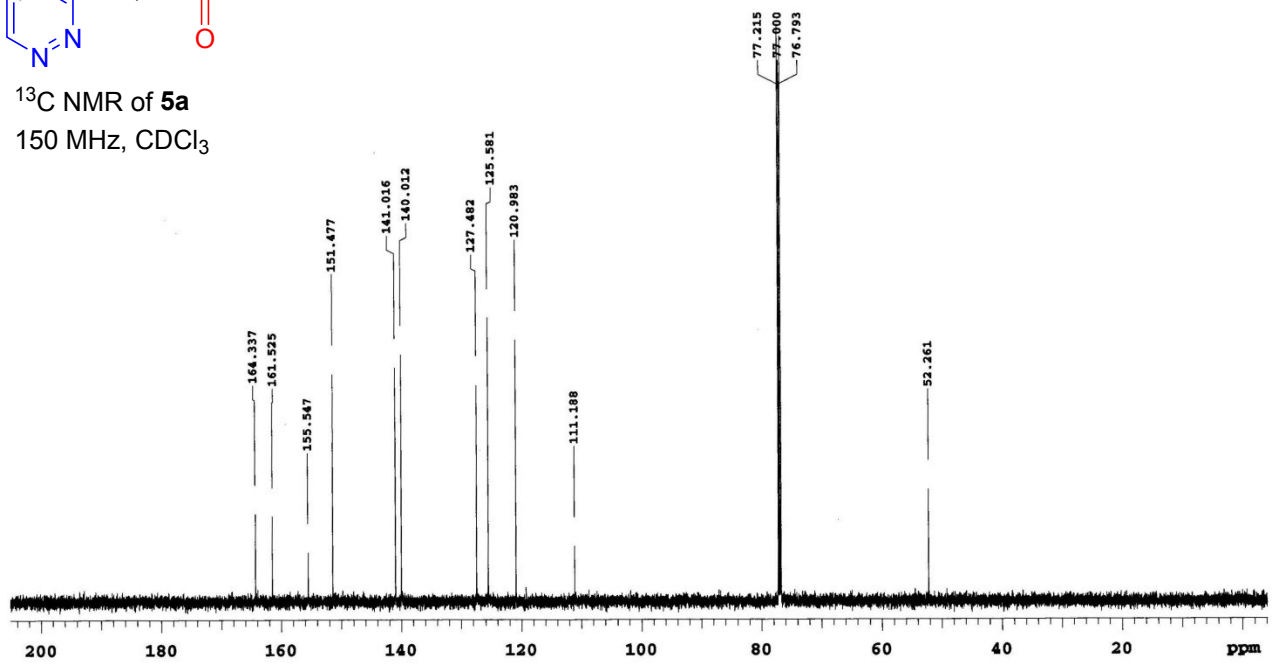
Methyl 6-oxo-1-(pyridazin-3-yl)-1,6-dihydropyridine-3-carboxylate (5a)



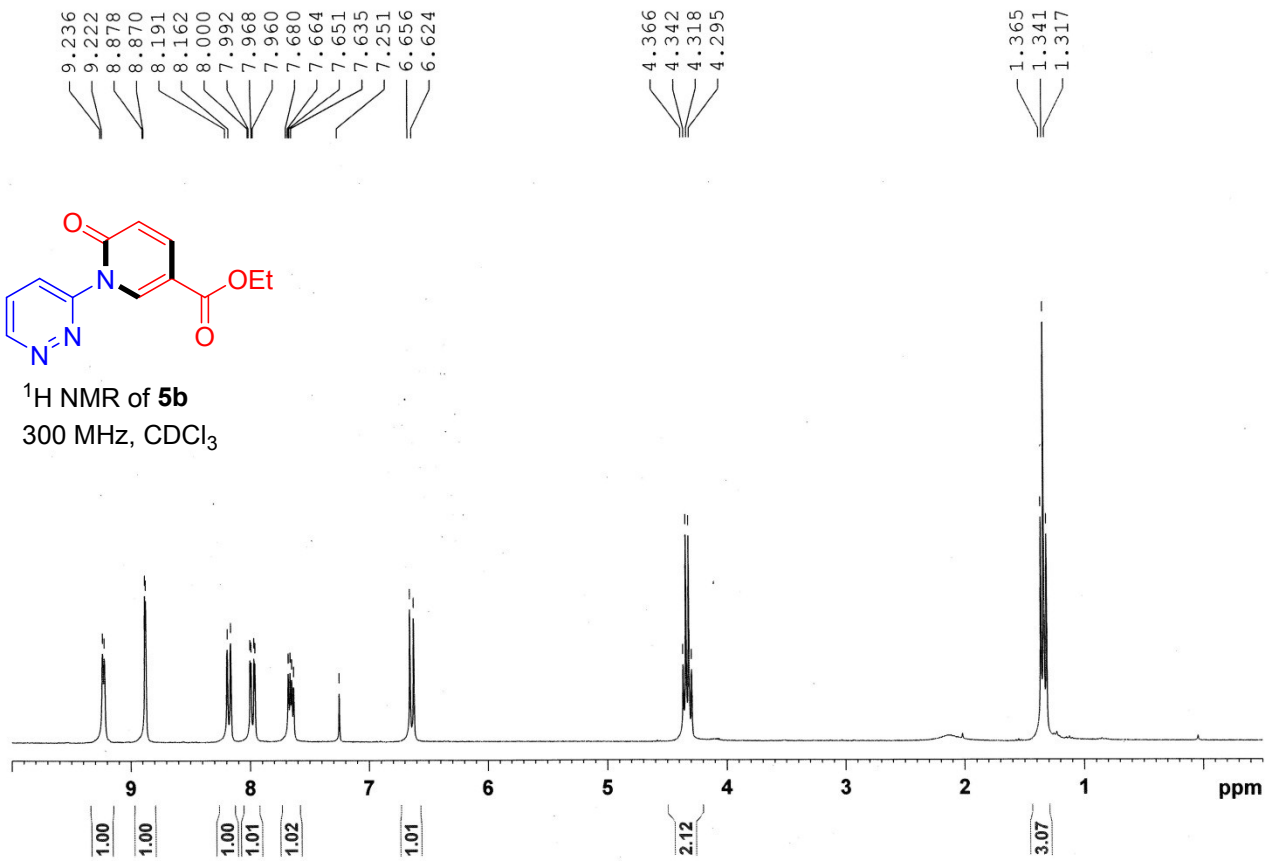
^{1}H NMR of **5a**
600 MHz, $CDCl_3$

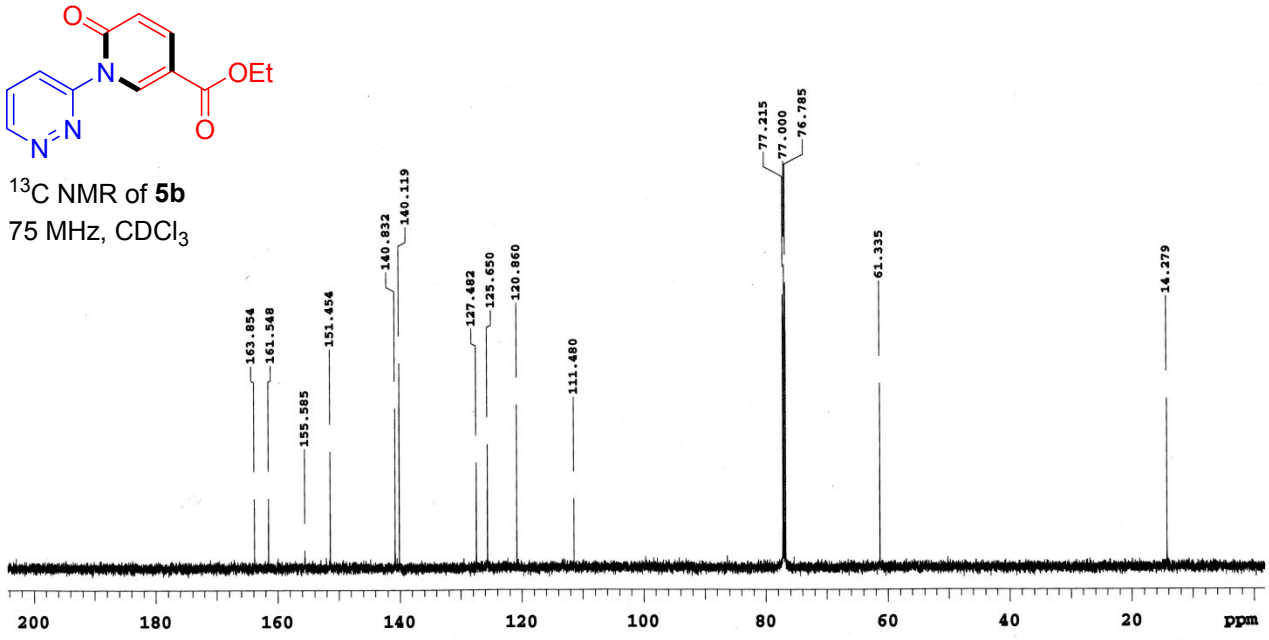


^{13}C NMR of **5a**
150 MHz, $CDCl_3$

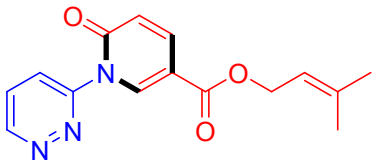


Ethyl 6-oxo-1-(pyridazin-3-yl)-1,6-dihydropyridine-3-carboxylate (5b)

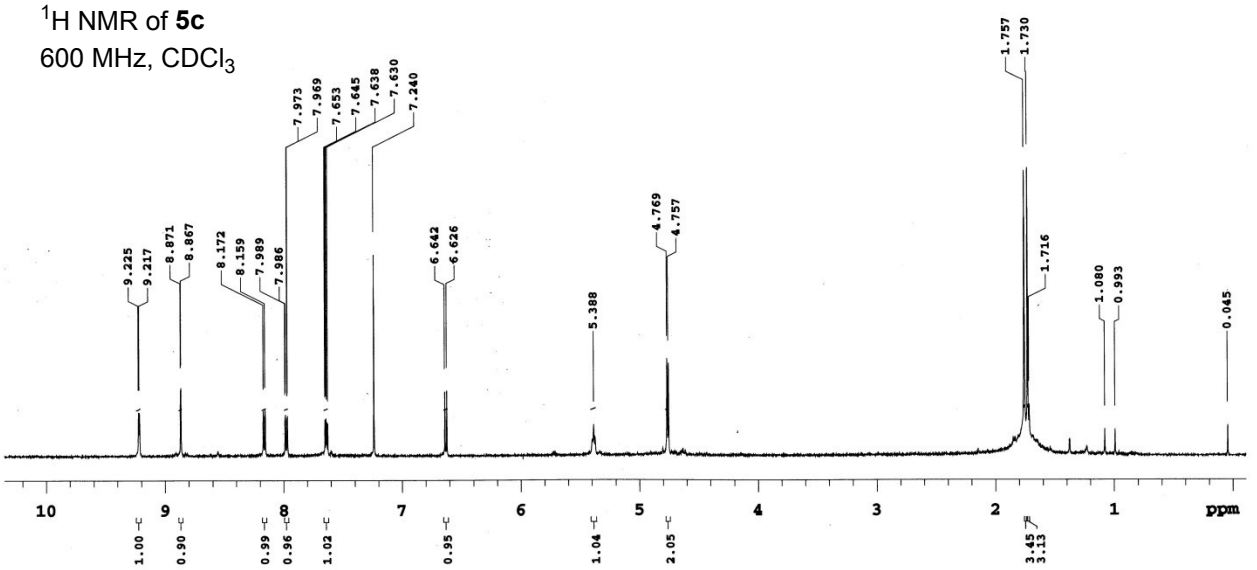


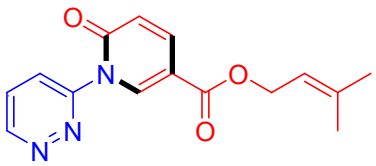


3-Methylbut-2-en-1-yl 6-oxo-1-(pyridazin-3-yl)-1,6-dihydropyridine-3-carboxylate (5c)

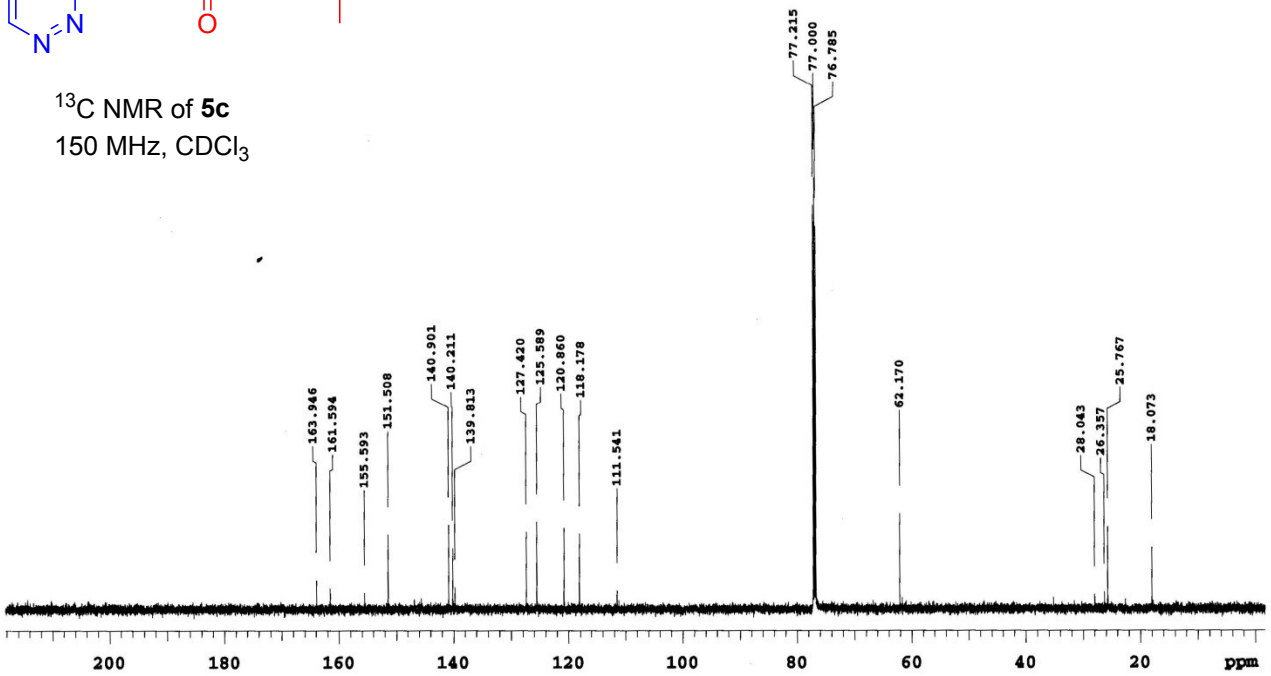


^{1}H NMR of **5c**
600 MHz, $CDCl_3$

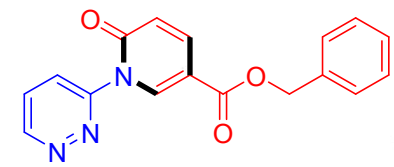




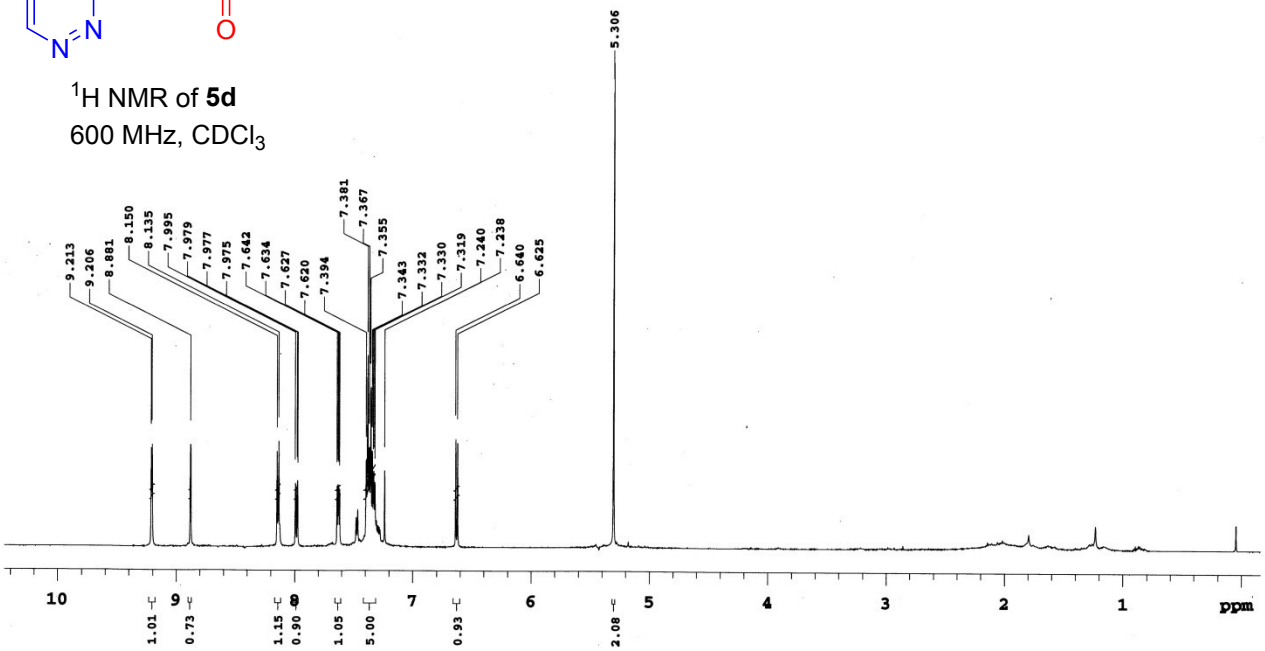
^{13}C NMR of **5c**
150 MHz, $CDCl_3$

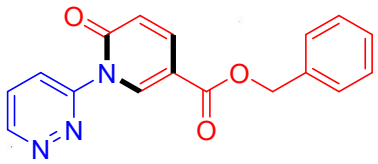


Benzyl 6-oxo-1-(pyridazin-3-yl)-1,6-dihydropyridine-3-carboxylate (5d)



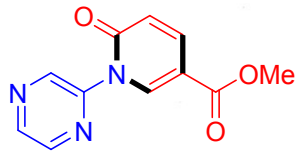
^{1}H NMR of **5d**
600 MHz, $CDCl_3$



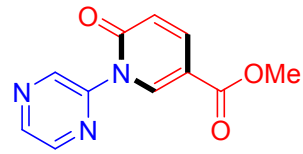


^{13}C NMR of **5d**
150 MHz, $CDCl_3$

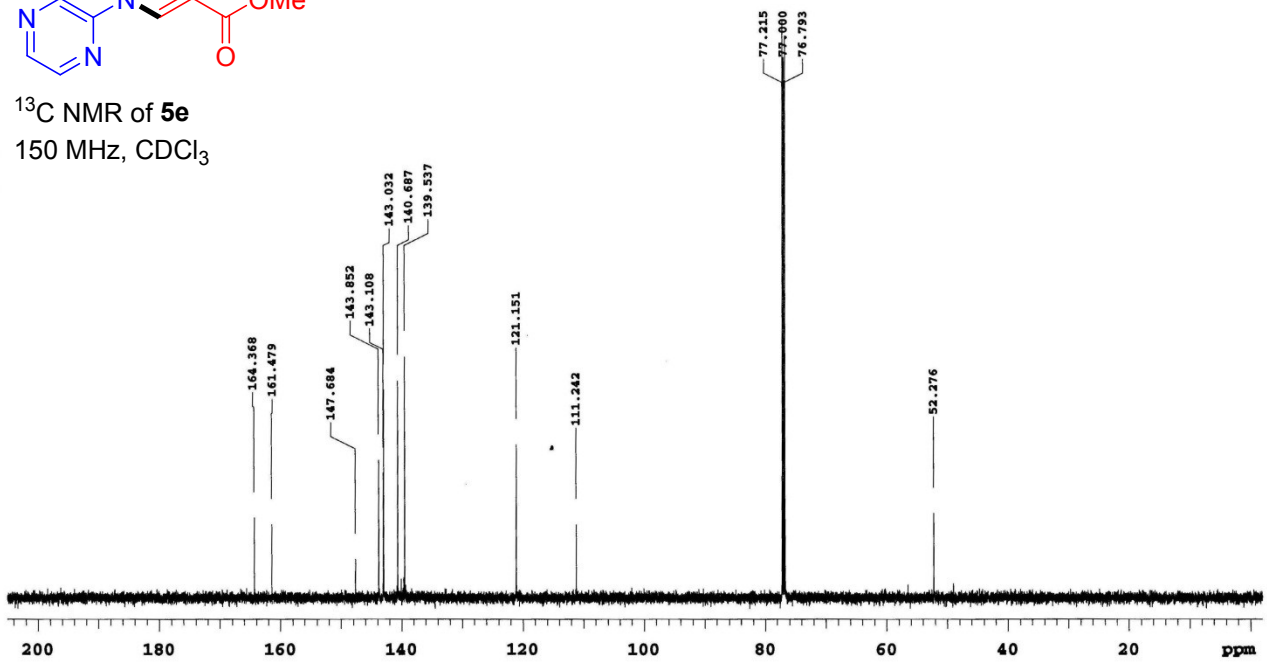
Methyl 6-oxo-1-(pyrazin-2-yl)-1,6-dihydropyridine-3-carboxylate (5e)



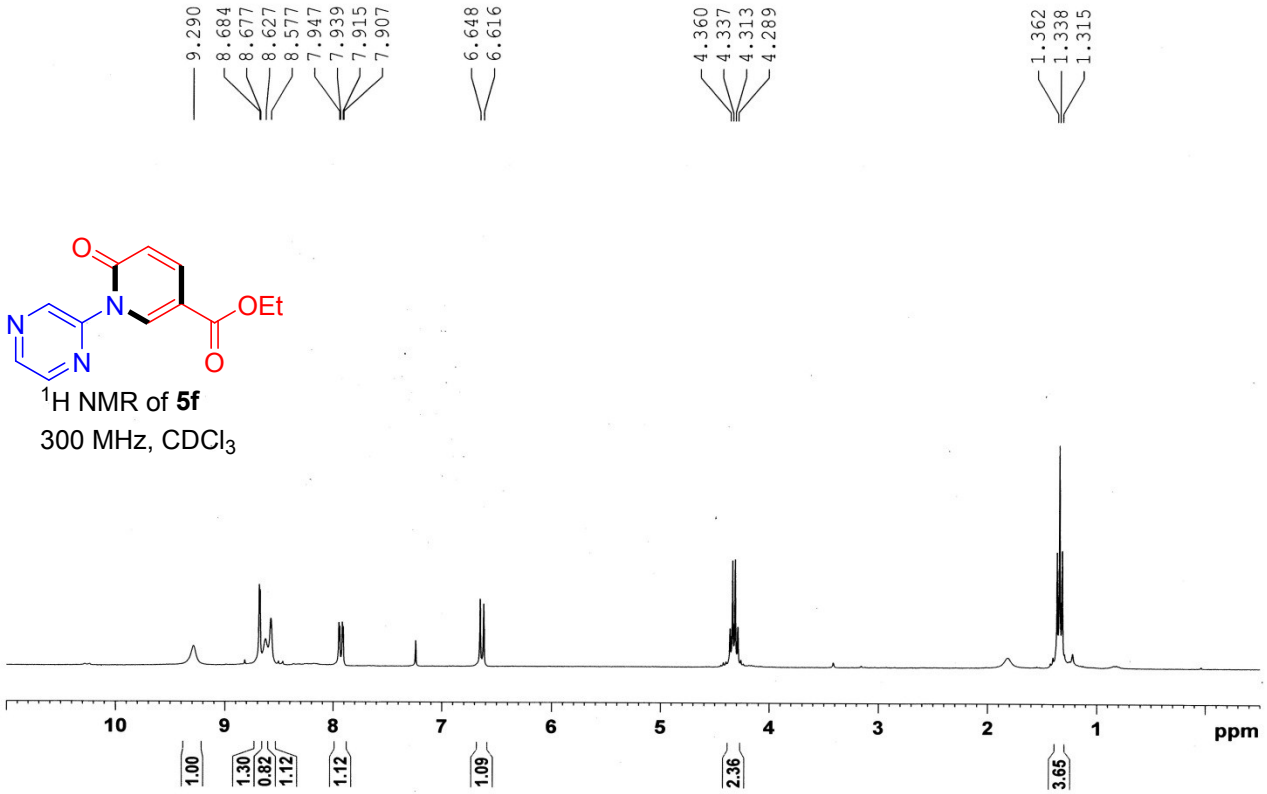
^{1}H NMR of **5e**
600 MHz, $CDCl_3$



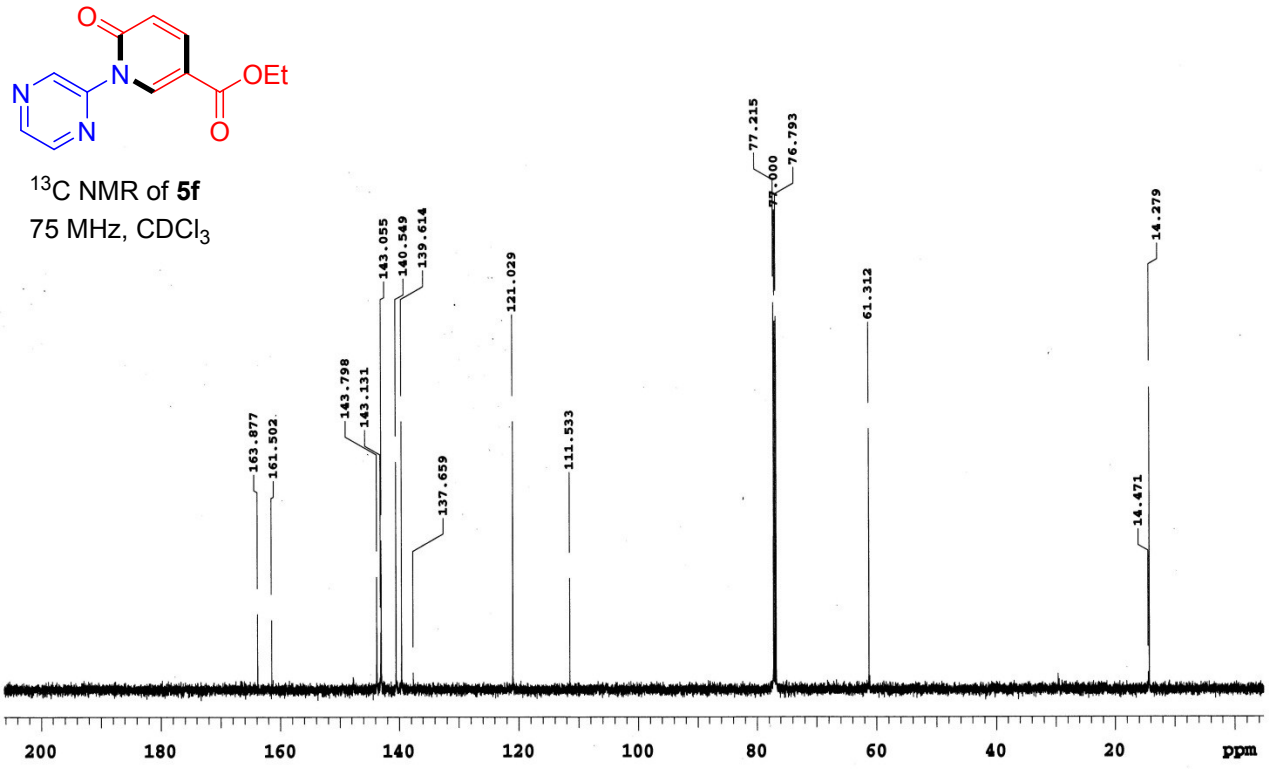
^{13}C NMR of **5e**
150 MHz, $CDCl_3$



Ethyl 6-oxo-1-(pyrazin-2-yl)-1,6-dihydropyridine-3-carboxylate (5f)

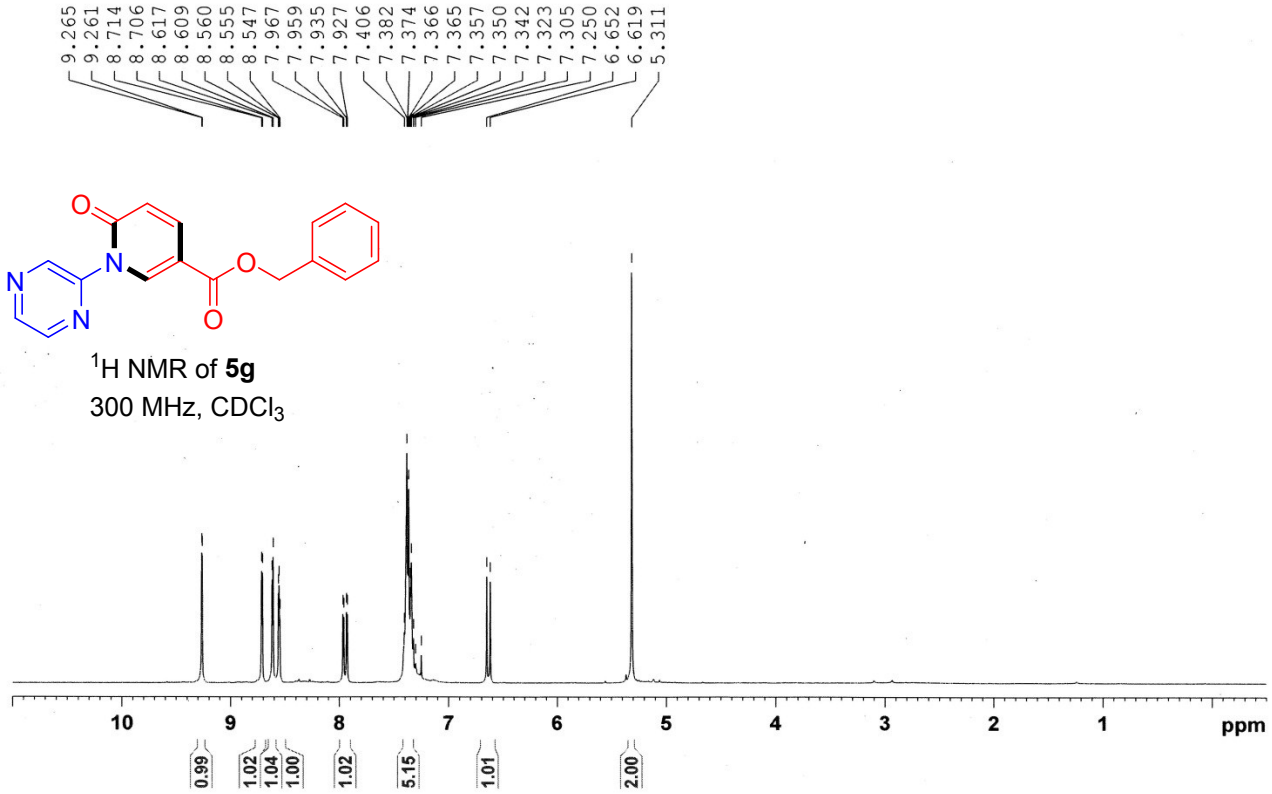


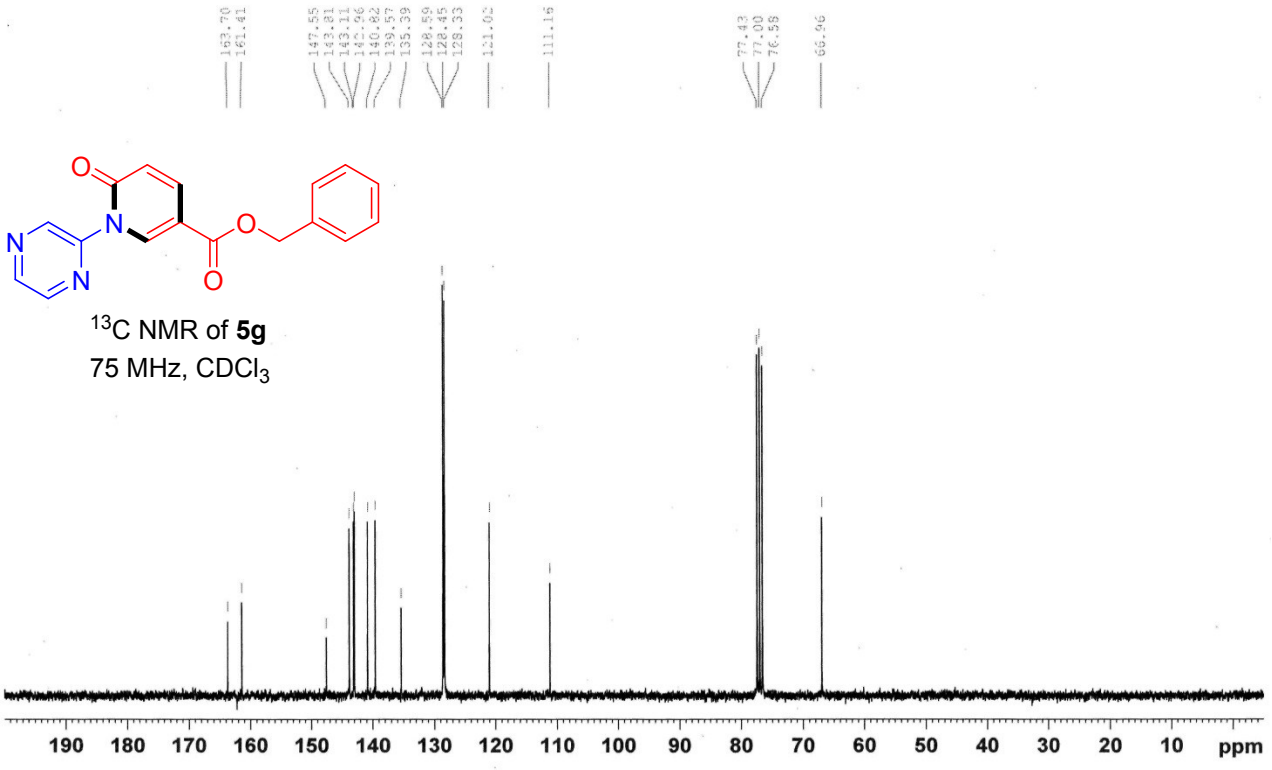
^{1}H NMR of **5f**
300 MHz, $CDCl_3$



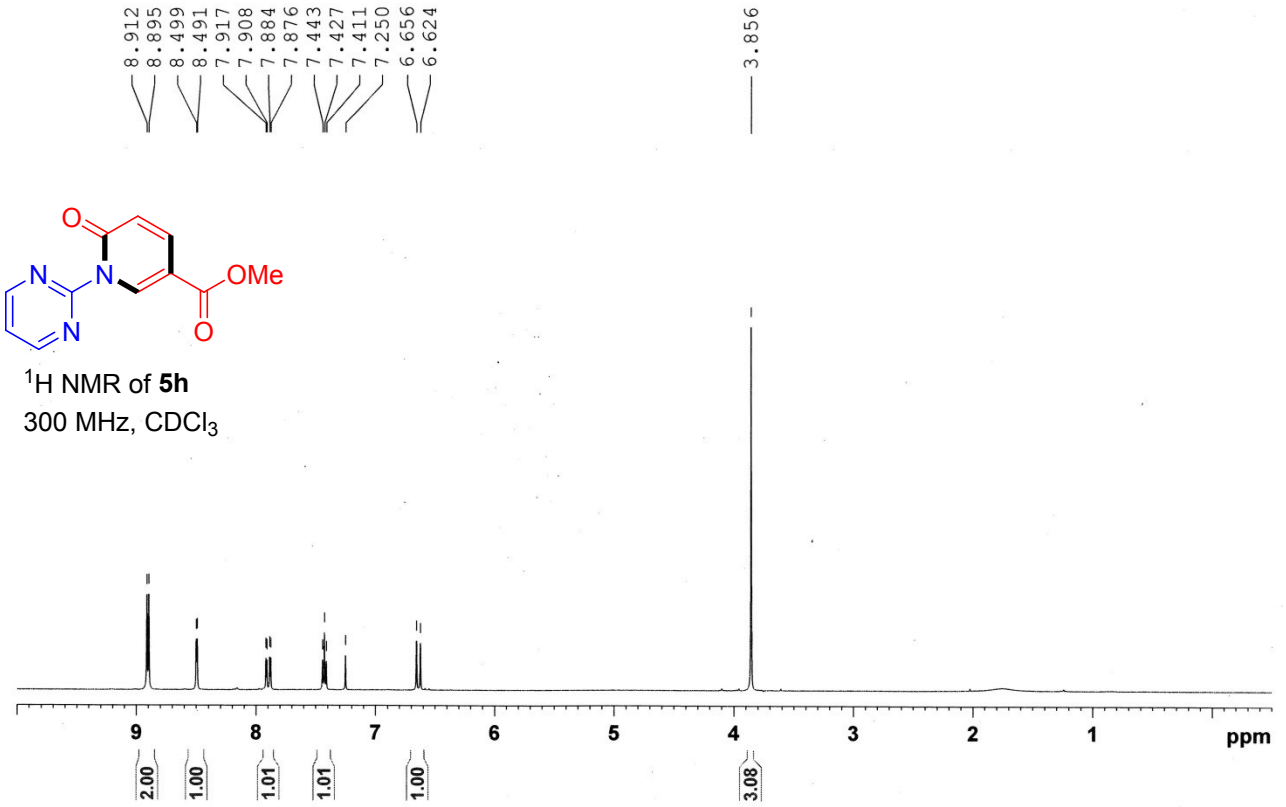
^{13}C NMR of **5f**
75 MHz, $CDCl_3$

Benzyl 6-oxo-1-(pyrazin-2-yl)-1,6-dihydropyridine-3-carboxylate (5g)

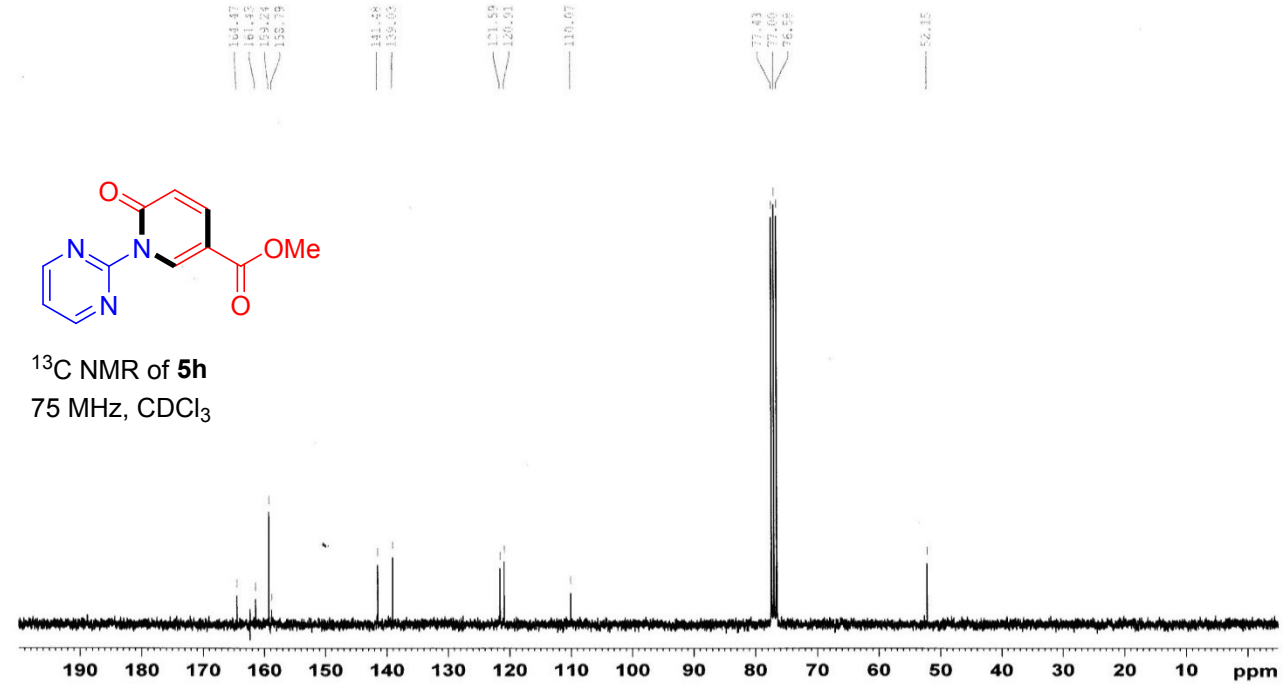




Methyl 6-oxo-1-(pyrimidin-2-yl)-1,6-dihydropyridine-3-carboxylate (5h)

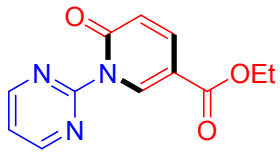


^{1}H NMR of **5h**
300 MHz, $CDCl_3$

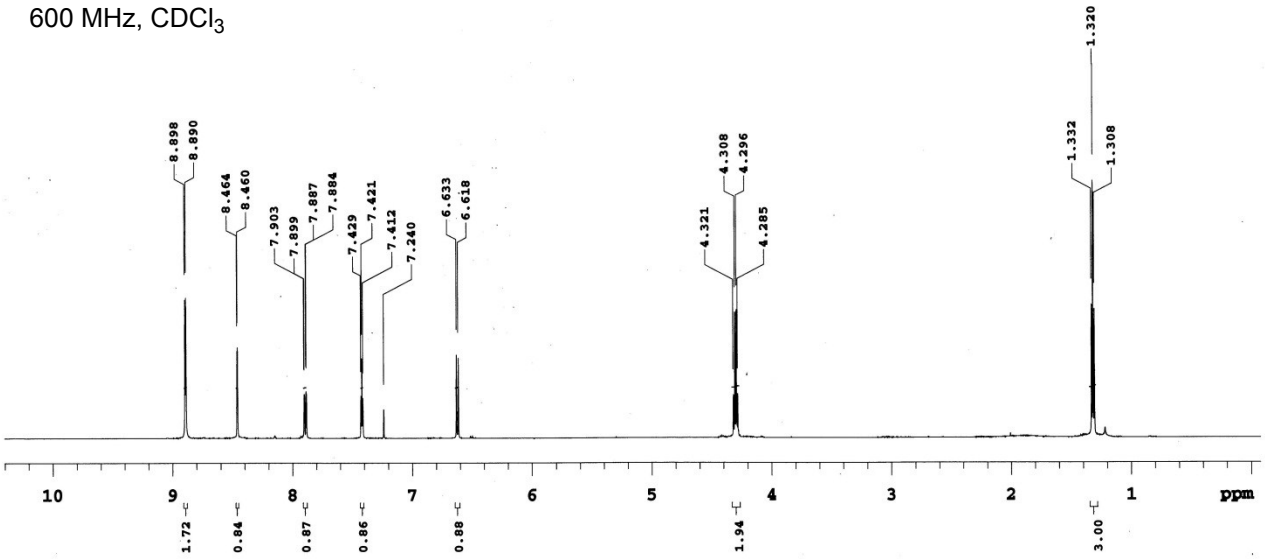


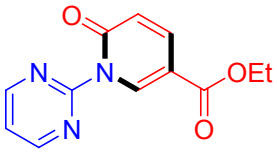
^{13}C NMR of **5h**
75 MHz, $CDCl_3$

Ethyl 6-oxo-1-(pyrimidin-2-yl)-1,6-dihydropyridine-3-carboxylate (5i)



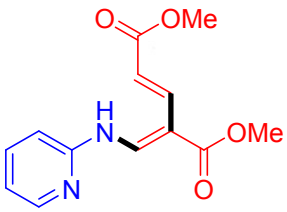
^{1}H NMR of **5i**
600 MHz, $CDCl_3$



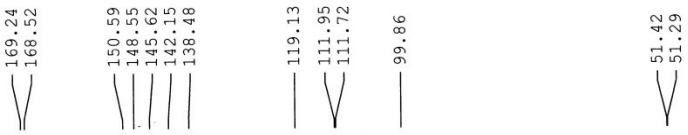


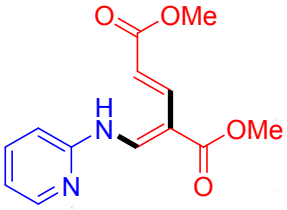
^{13}C NMR of **5i**
150 MHz, $CDCl_3$

dimethyl (2E,4E)-4-((pyridin-2-ylamino)methylene)pent-2-enedioate (6)

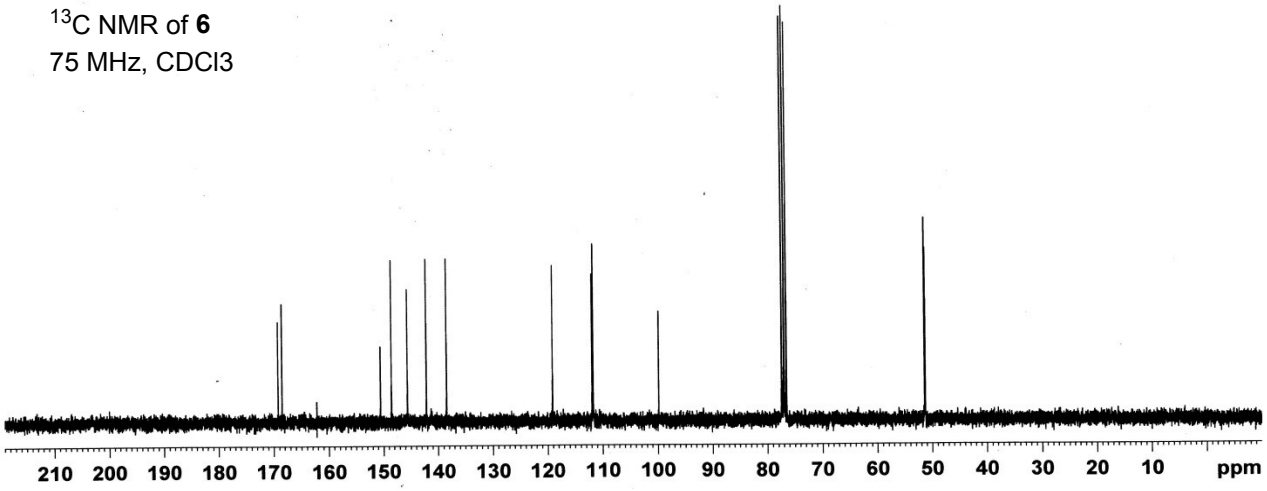


^{1}H NMR of **6**
600 MHz, CDCl3

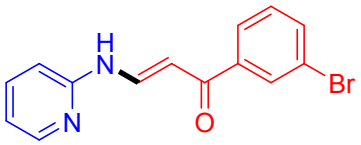




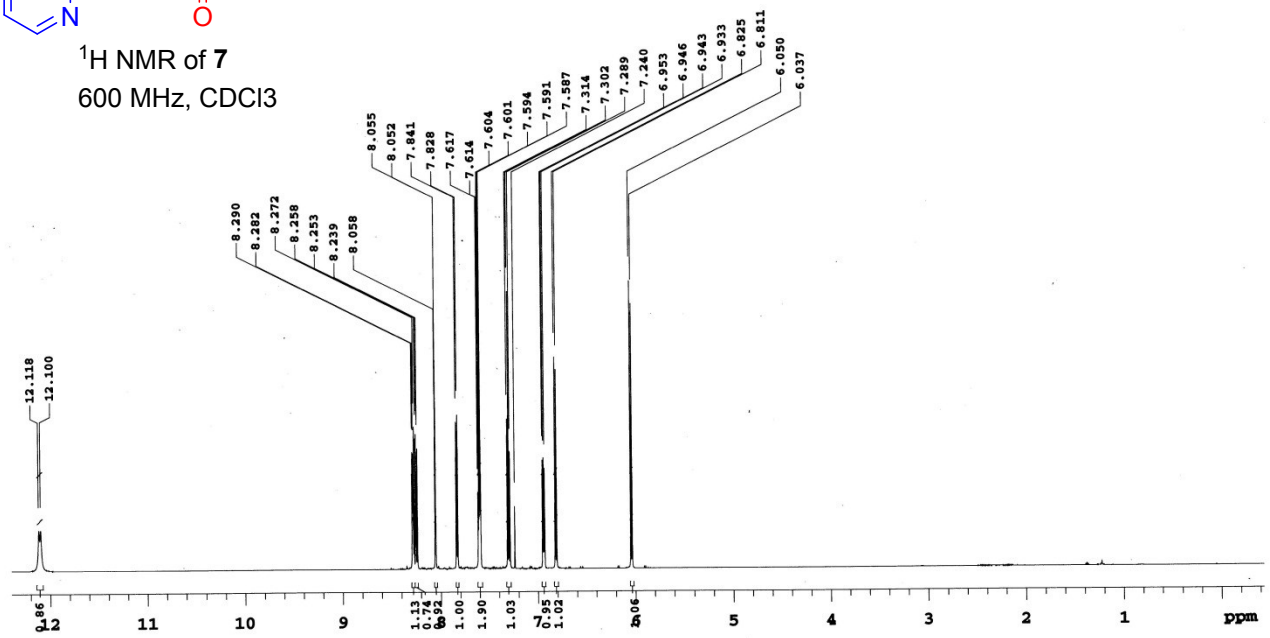
^{13}C NMR of **6**
75 MHz, CDCl3

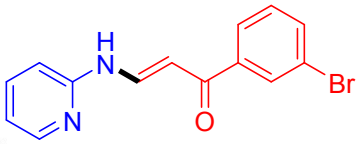


(E)-1-(3-bromophenyl)-3-(pyridin-2-ylamino)prop-2-en-1-one (7)

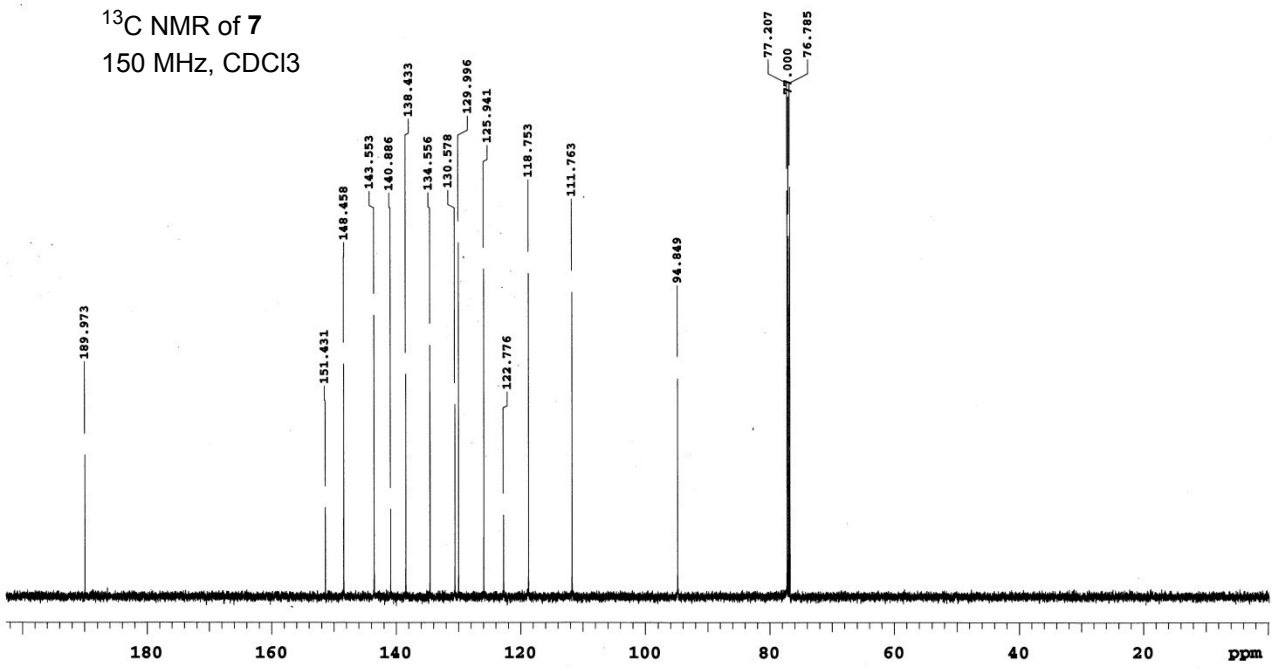


^{1}H NMR of **7**
600 MHz, CDCl3

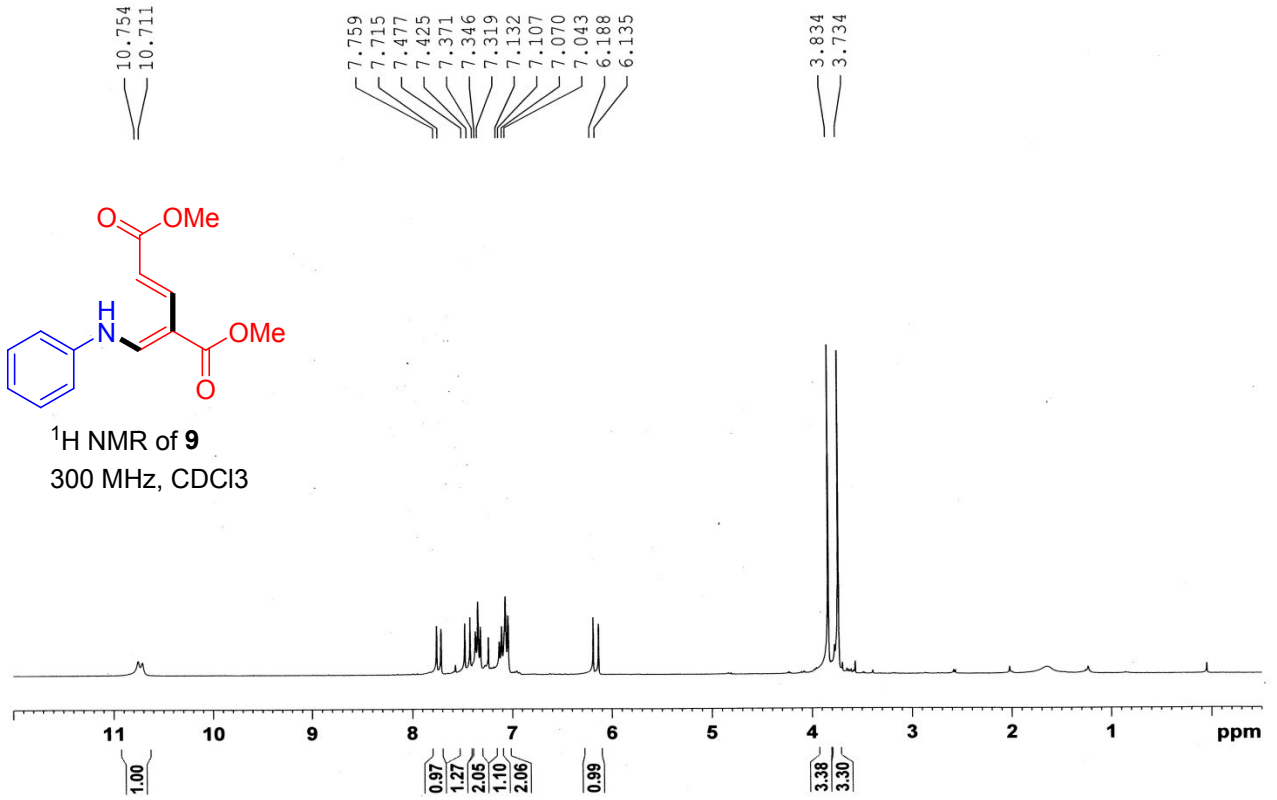




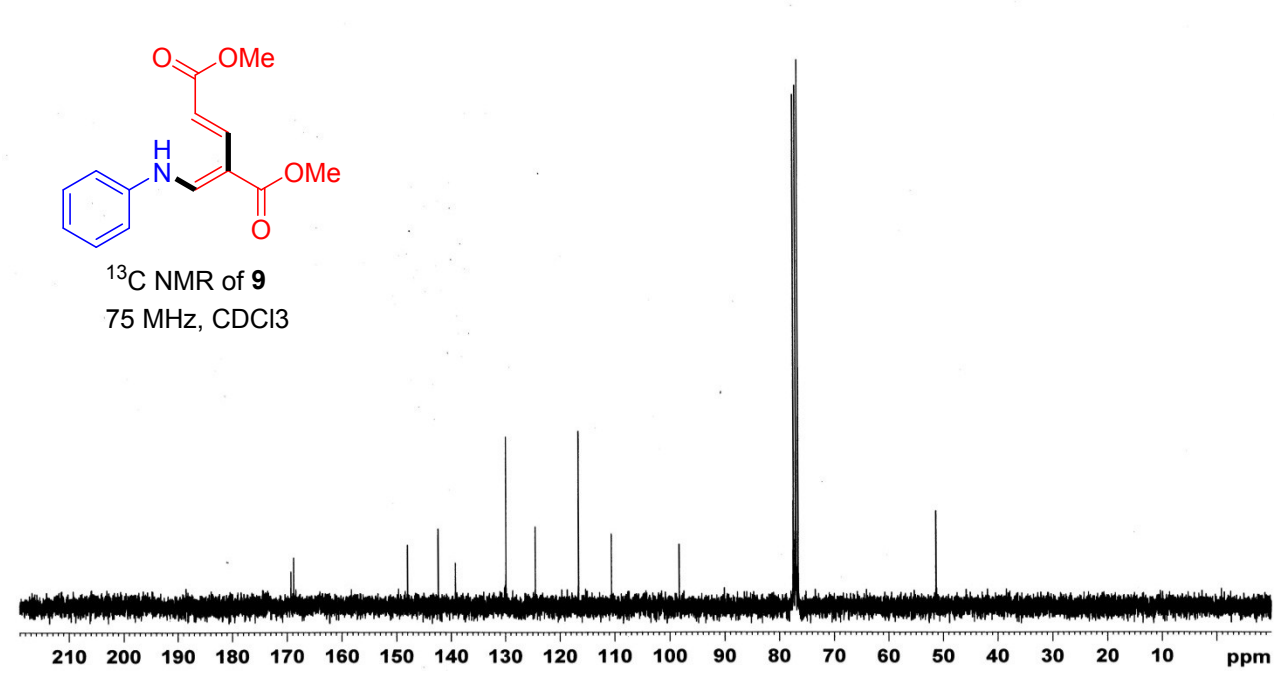
^{13}C NMR of **7**
150 MHz, CDCl3



dimethyl (2E,4E)-4-((phenylamino)methylene)pent-2-enedioate (9)



^{1}H NMR of **9**
300 MHz, CDCl3



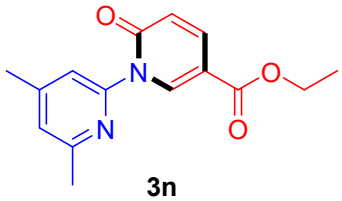
^{13}C NMR of **9**
75 MHz, CDCl3

X-Ray crystallographic structure and data of compound 3n:

A colorless block crystal (0.22 × 0.18 × 0.10 mm^3) was picked up with paraton oil and mounted on a Bruker D8 Venture PHOTON 100 CMOS diffractometer at the Korea Basic Science institute. The diffractometer was equipped with a graphite-monochromated Mo Kα (λ= 0.71073 Å) radiation source at -50 °C. Data collection and integration were performed with SMART APEX2 (Bruker, 2012) and SAINT (Bruker, 2012) [1]. Absorption correction was performed by multi-scan method implemented in SADABS [2]. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 using SHELXTL [3]. All the non-hydrogen atoms were refined anisotropically, and hydrogen atoms were added to their geometrically ideal positions.

1. SMART, SAINT and SADABS, Bruker AXS Inc., Madison, Wisconsin, USA, 2012.
2. G. M. Sheldrick, SADABS v 2.03, University of Göttingen, Germany, 2002.
3. SHELXTL v 6.10; Bruker AXS, Inc: Madison, Wisconsin, USA, 2000**.**

Empirical Formula $C_{15}H_{16}N_2O_3$, M = 272.30, Monoclinic, Space group $P_{21/n}$, a = 11.3734 (5) Å, b = 7.9651 (3) Å, c = 15.6500 (7) Å, V = 1413.23(10) $Å^3$, Z = 4, T = 223(2) K, ρ_{calcd} = 1.280 Mg/m^3, $2\Theta_{max.}$ = 28.31^0, Refinement of 184 parameters on 3517 independent reflections out of 37651 collected reflections (R_{int} = 0.0707) led to R_1 = 0.0459 [I >2σ(I)], wR_2 = 0.1283 (all data) and S = 1.038 with the largest difference peak and hole of 0.219 and -0.200 e. $Å^{-3}$ respectively. The crystal structure has been deposited at the Cambridge Crystallographic Data Centre (CCDC 1534635). The data can be obtained free of charge via the Internet at www.ccdc.cam.ac.uk/data_request/cif.



3n

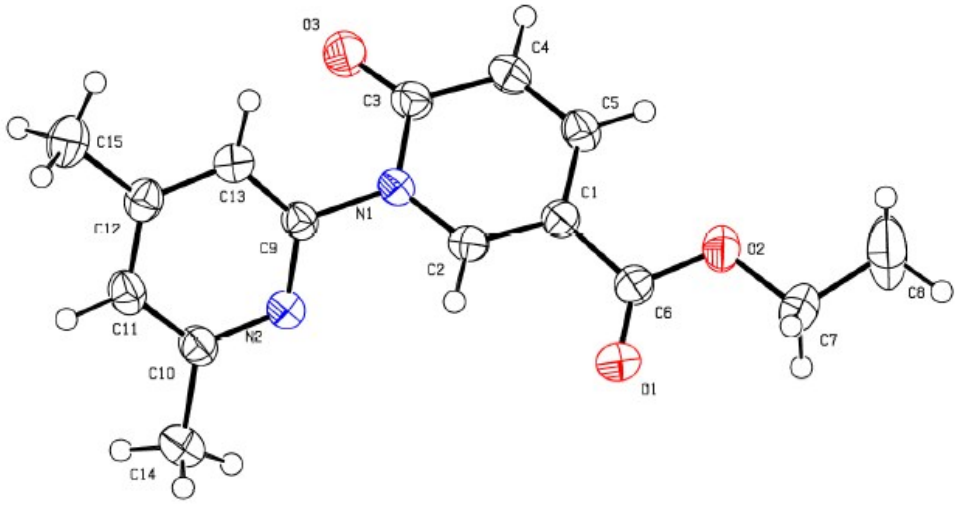


Figure S1. X-ray crystallographic structure of **3n** with 50% probability ellipsoids.

Table 1. Crystal data and structure refinement for **3n** (MS39).

Identification code	MS39	
Empirical formula	$C_{15}\ H_{16}\ N_2\ O_3$	
Formula weight	272.30	
Temperature	223(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 11.3734(5) Å	α= 90°.
	b = 7.9651(3) Å	β= 94.572(2)°.
	c = 15.6500(7) Å	γ = 90°.
Volume	1413.23(10) $Å^3$	
Z	4	
Density (calculated)	1.280 Mg/m^3	
Absorption coefficient	0.090 mm^{-1}	
F(000)	576	
Crystal size	0.22 x 0.18 x 0.10 mm^3	
Theta range for data collection	2.61 to 28.31°.	
Index ranges	-15<=h<=15, -10<=k<=10, -20<=l<=20	
Reflections collected	37651	
Independent reflections	3517 [R(int) = 0.0707]	
Completeness to theta = 28.31°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9910 and 0.9804	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3517 / 0 / 184	
Goodness-of-fit on F^2	1.038	
Final R indices [I>2sigma(I)]	R1 = 0.0459, wR2 = 0.1101	
R indices (all data)	R1 = 0.0763, wR2 = 0.1283	
Largest diff. peak and hole	0.219 and -0.200 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3n** (MS39). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3883(1)	250(2)	1316(1)	31(1)
C(2)	2828(1)	1038(2)	1305(1)	30(1)
N(1)	2646(1)	2586(2)	952(1)	30(1)
C(3)	3535(1)	3457(2)	550(1)	34(1)
C(4)	4657(1)	2617(2)	584(1)	38(1)
C(5)	4827(1)	1090(2)	947(1)	36(1)
C(6)	3992(1)	-1428(2)	1712(1)	33(1)
O(1)	3190(1)	-2189(1)	1996(1)	42(1)
O(2)	5098(1)	-2006(2)	1728(1)	41(1)
C(7)	5298(2)	-3661(2)	2100(1)	46(1)
C(8)	6582(2)	-3994(3)	2098(2)	79(1)
O(3)	3319(1)	4816(2)	206(1)	48(1)
C(9)	1475(1)	3310(2)	957(1)	29(1)
N(2)	613(1)	2277(2)	683(1)	32(1)
C(10)	-496(1)	2870(2)	695(1)	33(1)
C(11)	-719(1)	4471(2)	985(1)	36(1)
C(12)	196(1)	5519(2)	1280(1)	34(1)
C(13)	1338(1)	4912(2)	1262(1)	32(1)
C(14)	-1472(2)	1709(2)	382(1)	47(1)
C(15)	-27(2)	7256(2)	1609(1)	48(1)

Table 3. Bond lengths [Å] and angles [°] for **3n** (MS39).

C(1)-C(2)	1.354(2)
C(1)-C(5)	1.424(2)
C(1)-C(6)	1.474(2)
C(2)-N(1)	1.3602(19)
C(2)-H(2)	0.9400
N(1)-C(3)	1.4141(19)
N(1)-C(9)	1.4512(18)
C(3)-O(3)	1.225(2)
C(3)-C(4)	1.438(2)
C(4)-C(5)	1.350(2)
C(4)-H(4)	0.9400
C(5)-H(5)	0.9400
C(6)-O(1)	1.2086(19)
C(6)-O(2)	1.3387(19)
O(2)-C(7)	1.452(2)
C(7)-C(8)	1.485(3)
C(7)-H(7A)	0.9800
C(7)-H(7B)	0.9800
C(8)-H(8A)	0.9700
C(8)-H(8B)	0.9700
C(8)-H(8C)	0.9700
C(9)-N(2)	1.325(2)
C(9)-C(13)	1.375(2)
N(2)-C(10)	1.348(2)
C(10)-C(11)	1.384(2)
C(10)-C(14)	1.497(2)
C(11)-C(12)	1.384(2)
C(11)-H(11)	0.9400
C(12)-C(13)	1.388(2)
C(12)-C(15)	1.505(2)
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)-H(15A)	0.9700
C(15)-H(15B)	0.9700
C(15)-H(15C)	0.9700
C(2)-C(1)-C(5)	118.23(14)
C(2)-C(1)-C(6)	118.03(14)
C(5)-C(1)-C(6)	123.74(14)
C(1)-C(2)-N(1)	122.09(14)
C(1)-C(2)-H(2)	119.0
N(1)-C(2)-H(2)	119.0
C(2)-N(1)-C(3)	122.30(13)
C(2)-N(1)-C(9)	117.87(12)
C(3)-N(1)-C(9)	119.76(12)
O(3)-C(3)-N(1)	120.24(14)
O(3)-C(3)-C(4)	124.98(15)
N(1)-C(3)-C(4)	114.78(14)
C(5)-C(4)-C(3)	122.11(15)
C(5)-C(4)-H(4)	118.9
C(3)-C(4)-H(4)	118.9
C(4)-C(5)-C(1)	120.42(15)
C(4)-C(5)-H(5)	119.8
C(1)-C(5)-H(5)	119.8
O(1)-C(6)-O(2)	123.83(15)
O(1)-C(6)-C(1)	124.66(14)
O(2)-C(6)-C(1)	111.51(13)
C(6)-O(2)-C(7)	115.89(13)
O(2)-C(7)-C(8)	106.48(16)
O(2)-C(7)-H(7A)	110.4
C(8)-C(7)-H(7A)	110.4
O(2)-C(7)-H(7B)	110.4
C(8)-C(7)-H(7B)	110.4
H(7A)-C(7)-H(7B)	108.6
C(7)-C(8)-H(8A)	109.5
C(7)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(7)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5

H(8B)-C(8)-H(8C)	109.5
N(2)-C(9)-C(13)	125.90(14)
N(2)-C(9)-N(1)	114.02(13)
C(13)-C(9)-N(1)	120.03(13)
C(9)-N(2)-C(10)	116.57(13)
N(2)-C(10)-C(11)	121.56(15)
N(2)-C(10)-C(14)	116.69(15)
C(11)-C(10)-C(14)	121.75(15)
C(10)-C(11)-C(12)	120.81(15)
C(10)-C(11)-H(11)	119.6
C(12)-C(11)-H(11)	119.6
C(11)-C(12)-C(13)	117.59(15)
C(11)-C(12)-C(15)	121.69(15)
C(13)-C(12)-C(15)	120.72(15)
C(9)-C(13)-C(12)	117.56(15)
C(9)-C(13)-H(13)	121.2
C(12)-C(13)-H(13)	121.2
C(10)-C(14)-H(14A)	109.5
C(10)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(10)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(12)-C(15)-H(15A)	109.5
C(12)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(12)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3n** (MS39). 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}
C(1)	29(1)	30(1)	32(1)	-4(1)	3(1)	0(1)
C(2)	29(1)	30(1)	31(1)	-2(1)	4(1)	-4(1)
N(1)	25(1)	31(1)	35(1)	0(1)	4(1)	0(1)
C(3)	31(1)	36(1)	36(1)	2(1)	5(1)	-3(1)
C(4)	28(1)	42(1)	44(1)	3(1)	11(1)	-3(1)
C(5)	28(1)	39(1)	42(1)	-2(1)	7(1)	2(1)
C(6)	32(1)	31(1)	34(1)	-7(1)	1(1)	0(1)
O(1)	38(1)	33(1)	54(1)	1(1)	5(1)	-6(1)
O(2)	36(1)	37(1)	52(1)	5(1)	7(1)	8(1)
C(7)	53(1)	33(1)	52(1)	1(1)	1(1)	10(1)
C(8)	66(2)	86(2)	88(2)	35(1)	28(1)	42(1)
O(3)	39(1)	45(1)	61(1)	20(1)	12(1)	3(1)
C(9)	26(1)	33(1)	29(1)	2(1)	4(1)	1(1)
N(2)	28(1)	31(1)	36(1)	0(1)	2(1)	0(1)
C(10)	28(1)	37(1)	36(1)	3(1)	3(1)	1(1)
C(11)	30(1)	42(1)	37(1)	4(1)	7(1)	6(1)
C(12)	38(1)	33(1)	32(1)	1(1)	7(1)	4(1)
C(13)	33(1)	32(1)	33(1)	0(1)	3(1)	-2(1)
C(14)	32(1)	48(1)	61(1)	-2(1)	0(1)	-4(1)
C(15)	51(1)	40(1)	52(1)	-7(1)	9(1)	10(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **3n** (MS39).

	x	y	z	U(eq)
H(2)	2200	499	1550	36
H(4)	5292	3149	346	45
H(5)	5572	577	957	43
H(7A)	4846	-4509	1759	55
H(7B)	5055	-3688	2687	55
H(8A)	6821	-3880	1519	118
H(8B)	6751	-5125	2303	118
H(8C)	7015	-3195	2470	118
H(11)	-1502	4852	981	43
H(13)	1993	5572	1450	39
H(14A)	-1155	814	48	71
H(14B)	-2060	2328	26	71
H(14C)	-1833	1233	868	71
H(15A)	-352	7955	1141	71
H(15B)	709	7737	1851	71
H(15C)	-582	7194	2048	71

Table 6. Torsion angles [°] for **3n** (MS39).

C(5)-C(1)-C(2)-N(1)	0.4(2)
C(6)-C(1)-C(2)-N(1)	-179.84(13)
C(1)-C(2)-N(1)-C(3)	1.9(2)
C(1)-C(2)-N(1)-C(9)	178.80(14)
C(2)-N(1)-C(3)-O(3)	176.73(15)
C(9)-N(1)-C(3)-O(3)	-0.2(2)
C(2)-N(1)-C(3)-C(4)	-3.0(2)

C(9)-N(1)-C(3)-C(4)	-179.92(14)
O(3)-C(3)-C(4)-C(5)	-177.58(17)
N(1)-C(3)-C(4)-C(5)	2.2(2)
C(3)-C(4)-C(5)-C(1)	-0.1(3)
C(2)-C(1)-C(5)-C(4)	-1.2(2)
C(6)-C(1)-C(5)-C(4)	179.01(15)
C(2)-C(1)-C(6)-O(1)	3.4(2)
C(5)-C(1)-C(6)-O(1)	-176.81(15)
C(2)-C(1)-C(6)-O(2)	-175.88(13)
C(5)-C(1)-C(6)-O(2)	3.9(2)
O(1)-C(6)-O(2)-C(7)	1.2(2)
C(1)-C(6)-O(2)-C(7)	-179.52(13)
C(6)-O(2)-C(7)-C(8)	-176.65(17)
C(2)-N(1)-C(9)-N(2)	-48.33(18)
C(3)-N(1)-C(9)-N(2)	128.68(15)
C(2)-N(1)-C(9)-C(13)	129.25(15)
C(3)-N(1)-C(9)-C(13)	-53.74(19)
C(13)-C(9)-N(2)-C(10)	1.0(2)
N(1)-C(9)-N(2)-C(10)	178.38(13)
C(9)-N(2)-C(10)-C(11)	-0.4(2)
C(9)-N(2)-C(10)-C(14)	179.69(15)
N(2)-C(10)-C(11)-C(12)	-0.5(2)
C(14)-C(10)-C(11)-C(12)	179.39(16)
C(10)-C(11)-C(12)-C(13)	0.9(2)
C(10)-C(11)-C(12)-C(15)	-179.08(15)
N(2)-C(9)-C(13)-C(12)	-0.5(2)
N(1)-C(9)-C(13)-C(12)	-177.81(13)
C(11)-C(12)-C(13)-C(9)	-0.4(2)
C(15)-C(12)-C(13)-C(9)	179.57(15)

Symmetry transformations used to generate equivalent atoms: