

Supporting information

I₂-mediated oxidative cross coupling of 2- amino pyridine with terminal alkyne: Synthesis of Imidazo[1,2-*a*]pyridine under mild Condition

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Shibpur, Howrah-711103 (WB), India

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A. X-ray crystallographic data:

Single crystals of **3f** were obtained by slow evaporation from a solution of ethyl acetate and hexane (3:7). Data were collected using Bruker D8-Venture diffractometer. Further information were available from Cambridge crystallographic data centre (UK) with CCDC deposition number **1876401**.

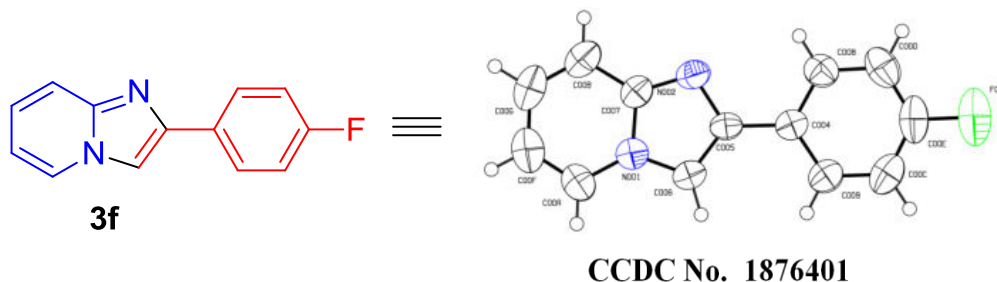


Figure 1: ORTEP diagram of the compound **3f** with at 50 % probability level

Table 2: Crystallographic data and structure refinement parameter for 3f

Identification code : 3f			
Molecular Weight : 212.0750			
Bond precision :	C-C = 0.0033 Å	Wavelength = 0.71073	
Cell :	a= 11.6177(10) alpha=90	b=14.1889(12) beta=91.299(2)	c=6.2178(5) gamma=90
Temperature :	293 K		
	Calculated	Reported	
Volume	1024.69(15)	1024.69(15)	
Space group	P 21/c	P 1 21/c 1	
Hall group	: -P 2ybc	-P 2ybc	
Moiety formula	C ₁₃ H ₉ F N ₂	C ₁₃ H ₉ F N ₂	
Sum formula	C ₁₃ H ₉ F N ₂	C ₁₃ H ₉ F N ₂	
Mr	212.22	212.22	
Dx,g cm ⁻³	1.376	1.376	
Z	4	4	
Mu (mm ⁻¹)	0.096	0.096	
F000	440.0	440.0	
F000'	440.20		

h,k,lmax	13,16,7	13,16,7
Nref	1834	1824
Tmin,Tmax	0.976,0.981	0.475,0.745
Tmin'	0.976	

Correction method= # Reported T Limits: Tmin=0.475 Tmax=0.745 AbsCorr = MULTI-SCAN

Data completeness= 0.995
R(reflections)= 0.0511(1245)

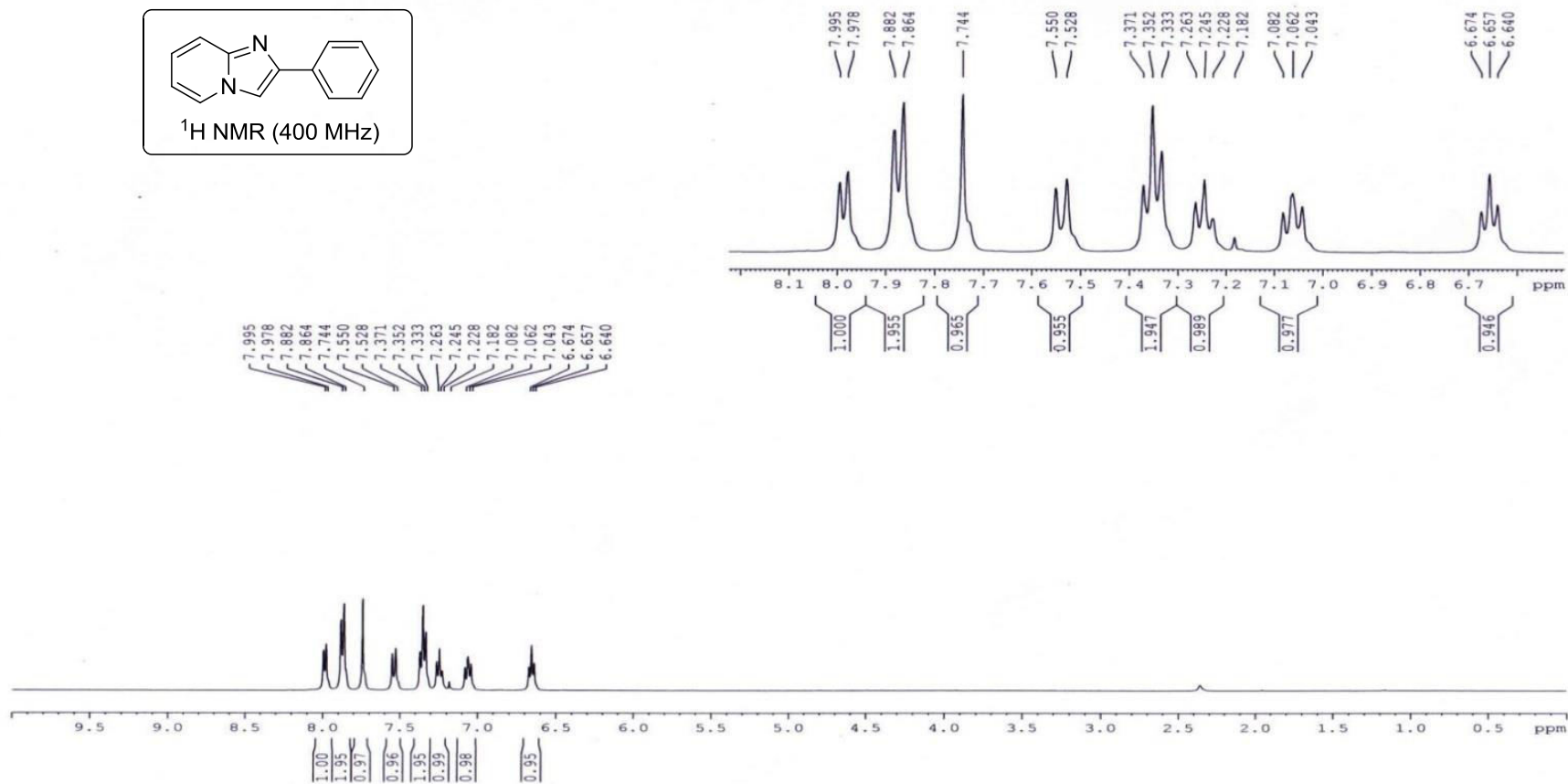
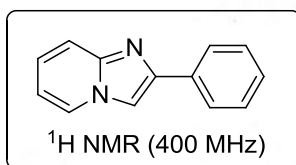
Theta(max)= 25.124
wR2(reflections)= 0.1266(1824)

S = 1.033

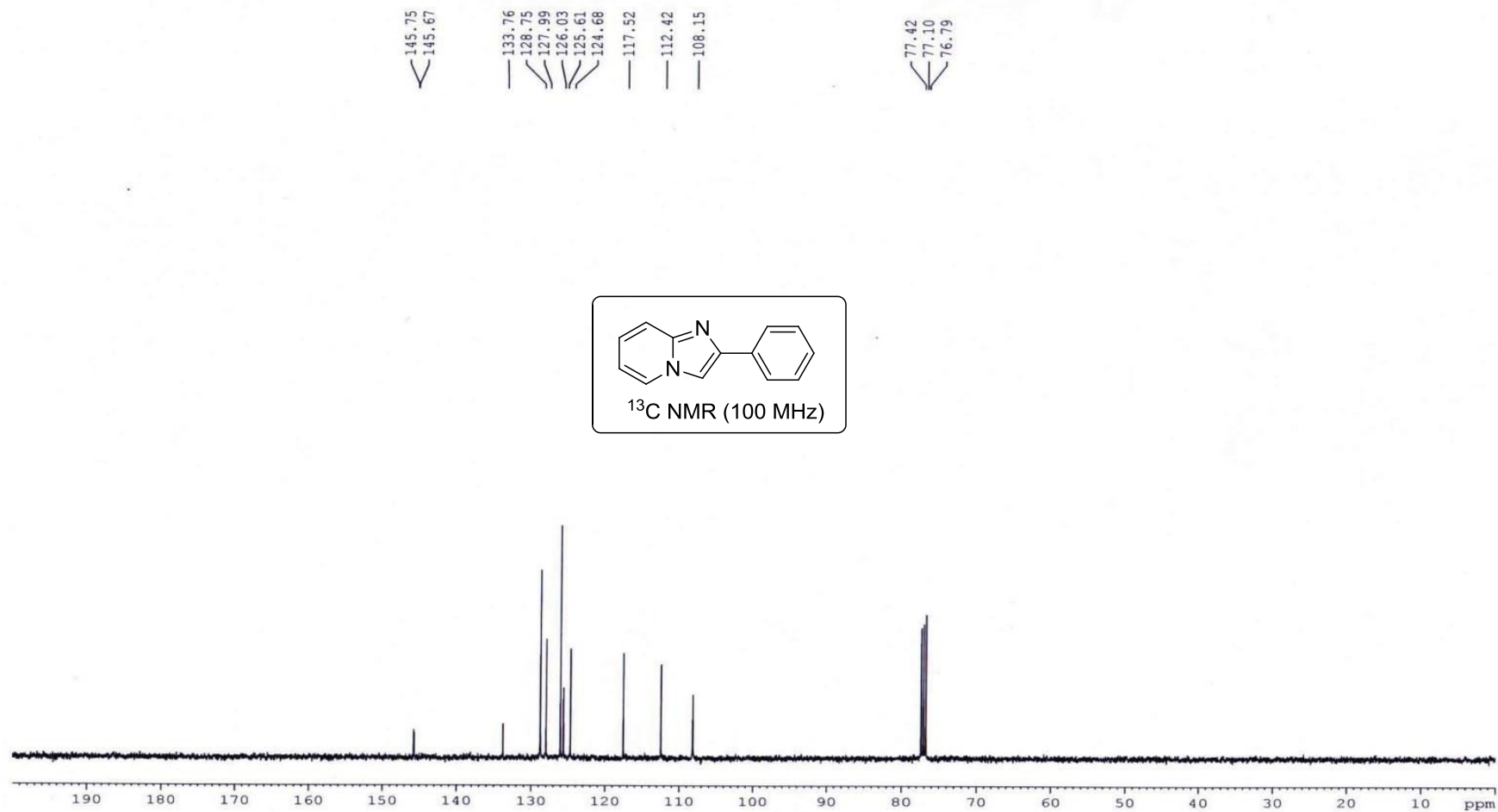
Npar= 145

B. Copies of ^1H NMR and ^{13}C NMR compounds:

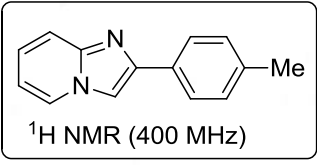
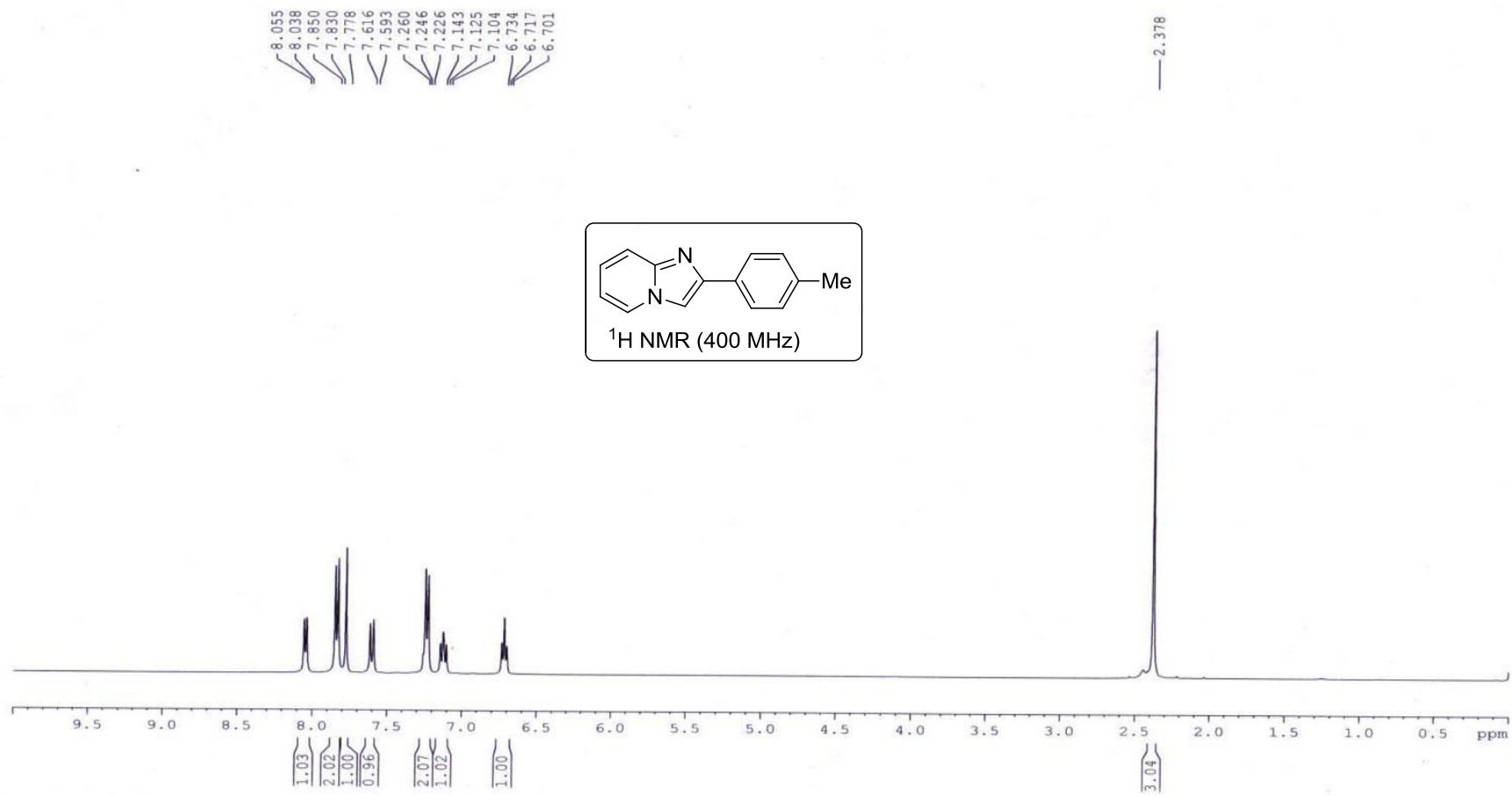
2-phenylimidazo[1,2-a]pyridine(3a) :



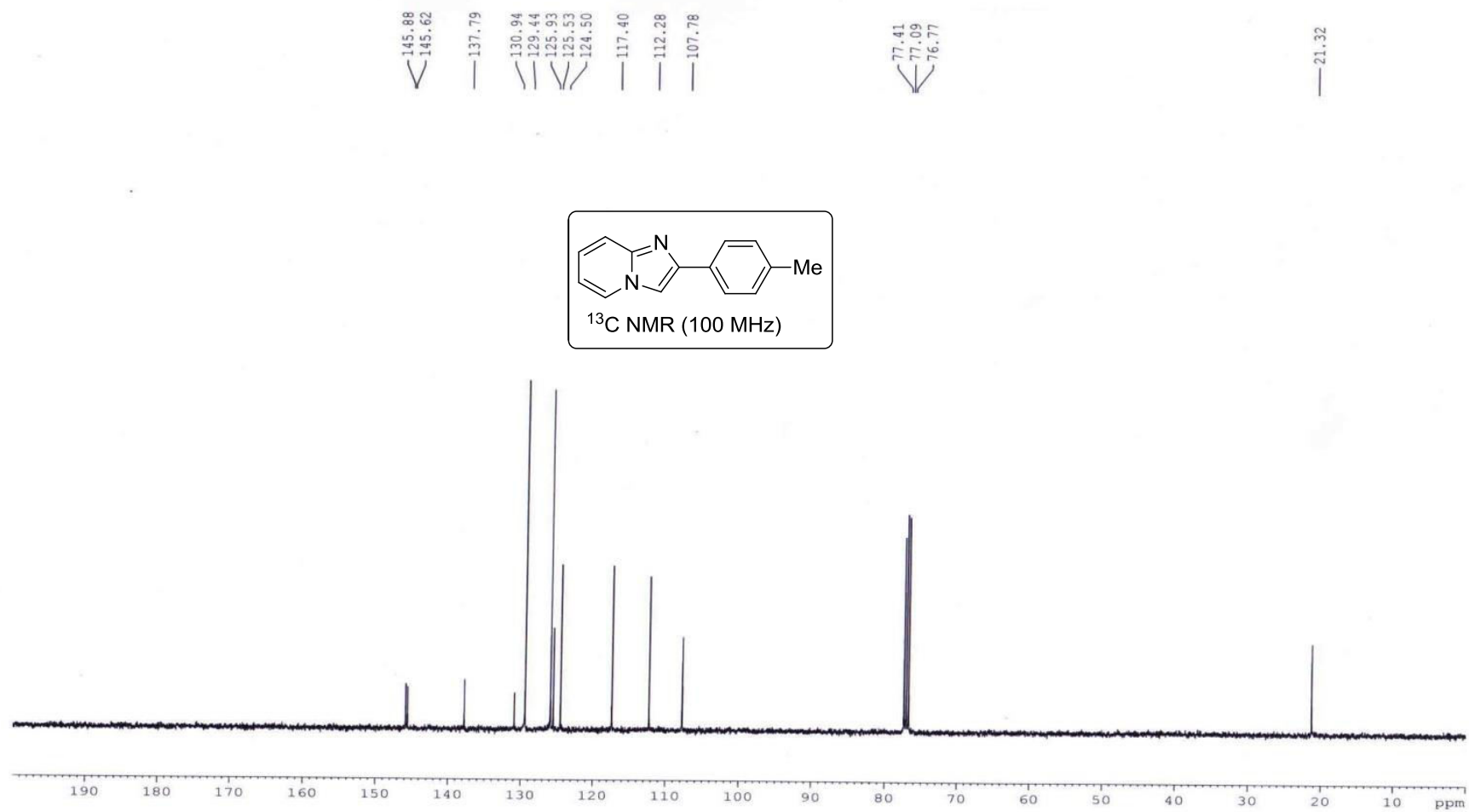
2-phenylimidazo[1,2-a]pyridine(3a) :



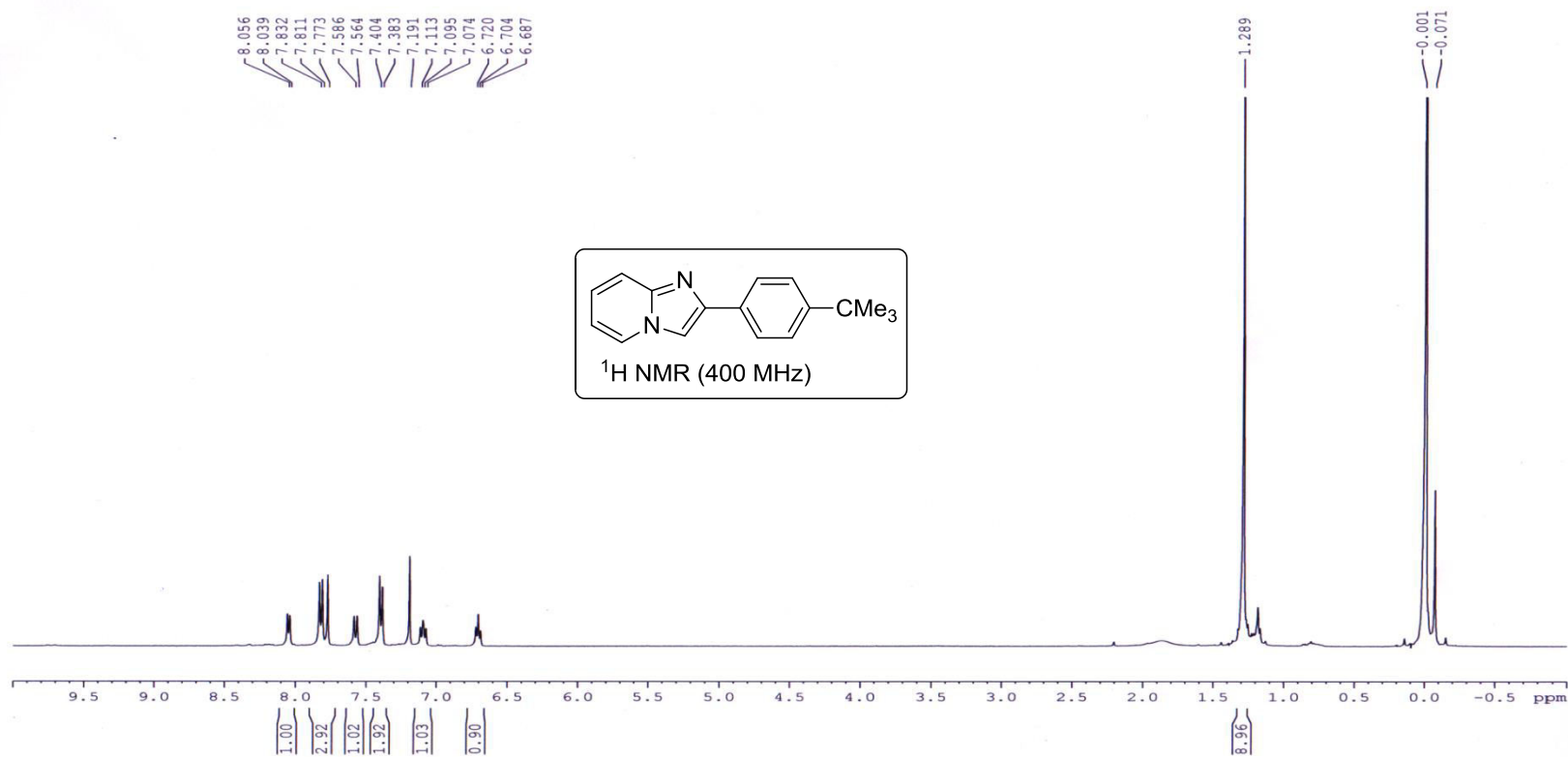
2-(p-tolyl)imidazo[1,2-a]pyridine (3b) :



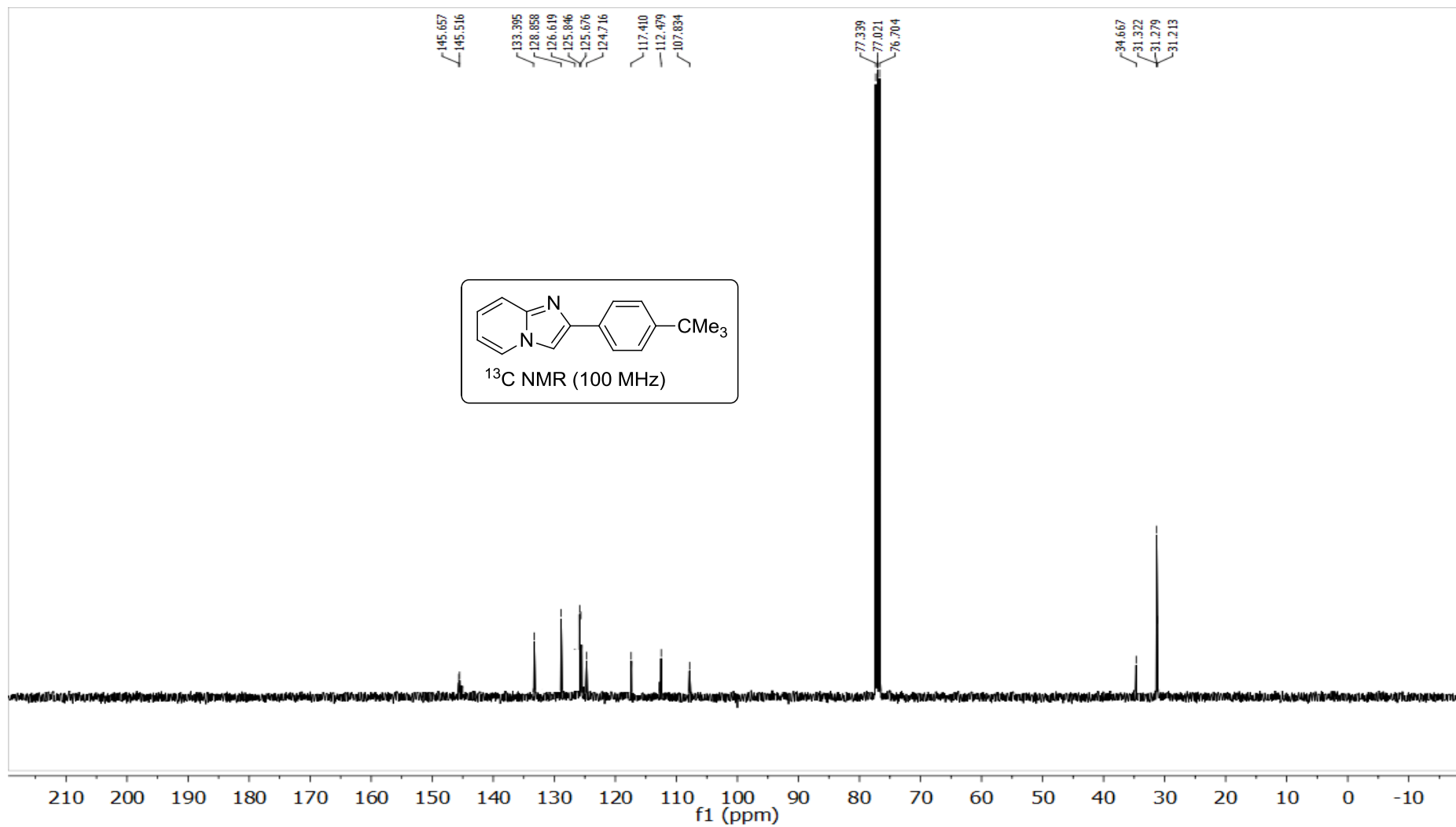
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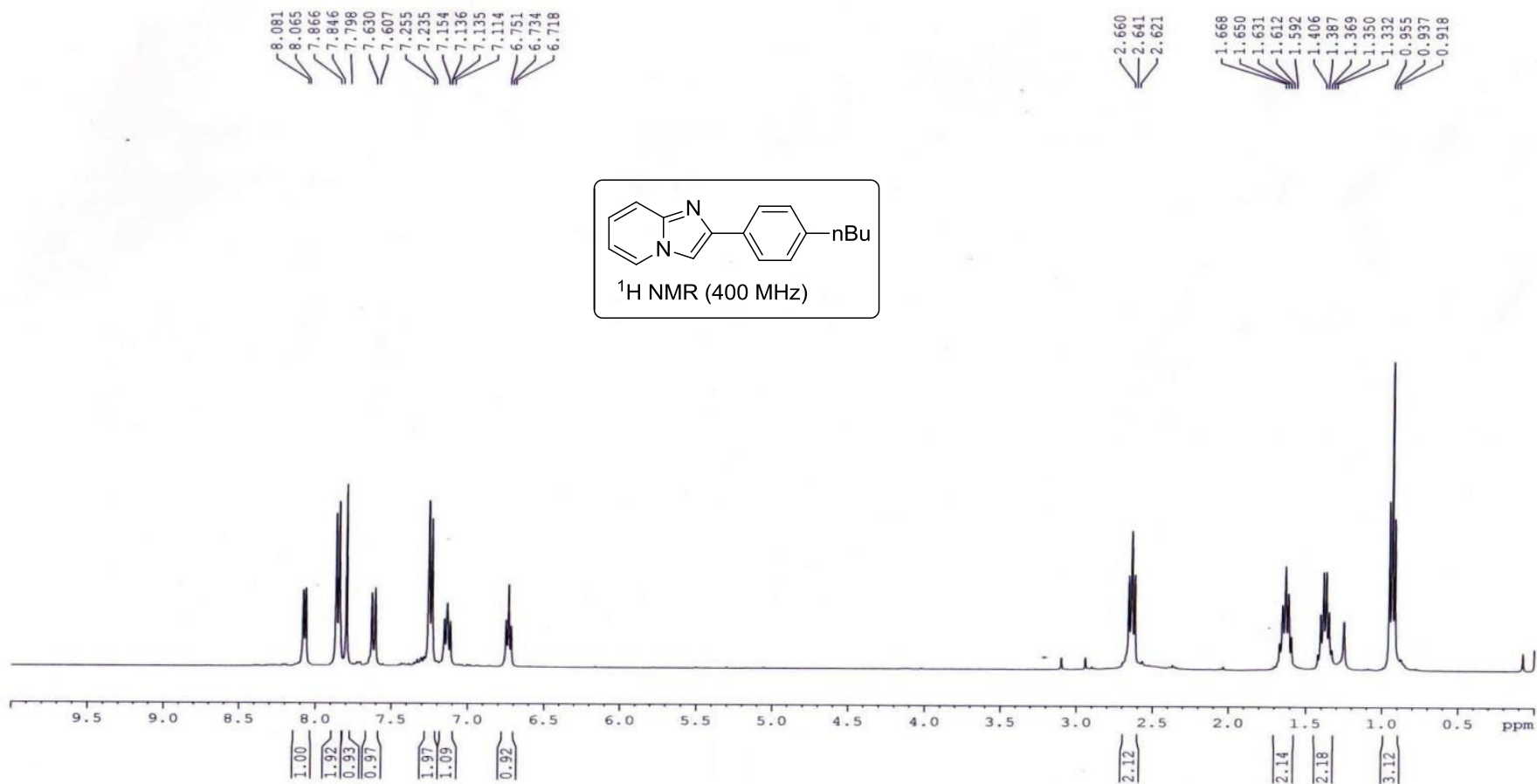
2-(4-(tert-butyl)phenyl)imidazo[1,2-a]pyridine (3c) :



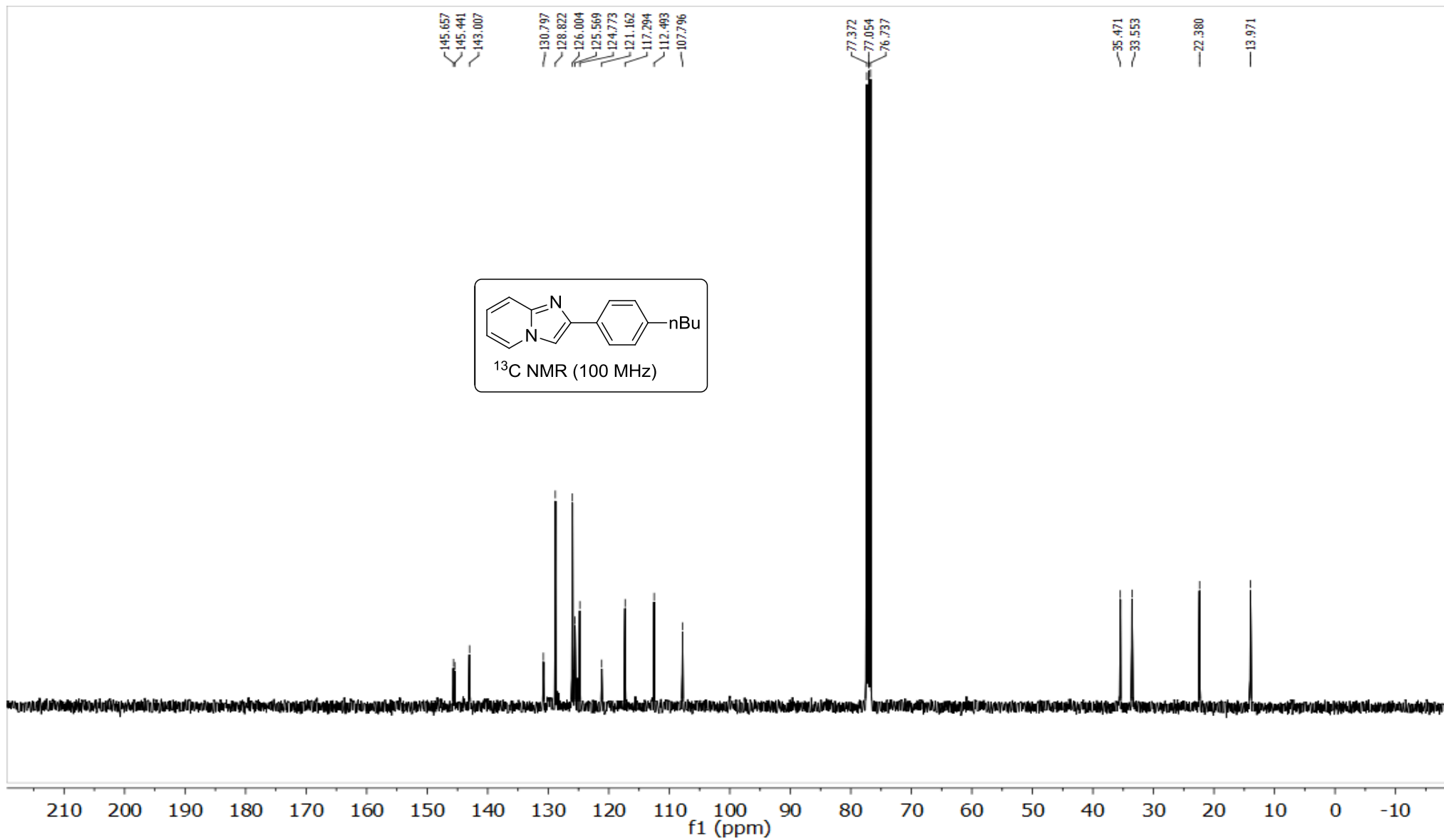
2-(4-(tert-butyl)phenyl)imidazo[1,2-a]pyridine (3c) :



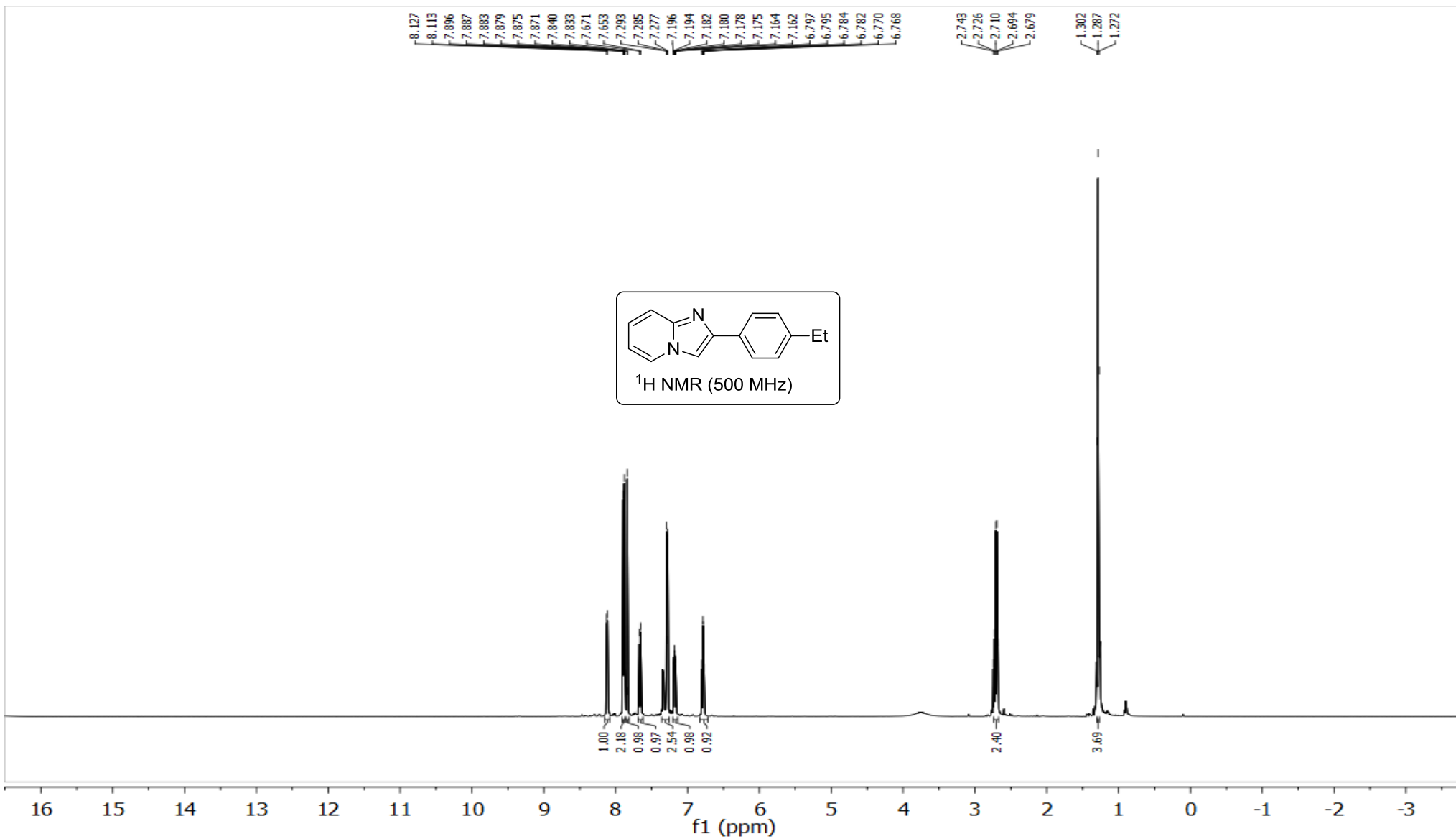
2-(4-butylphenyl)imidazo[1,2-a]pyridine (3d) :



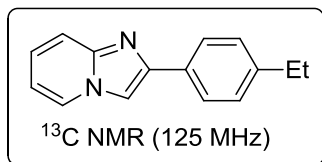
2-(4-butylphenyl)imidazo[1,2-a]pyridine (3d) :



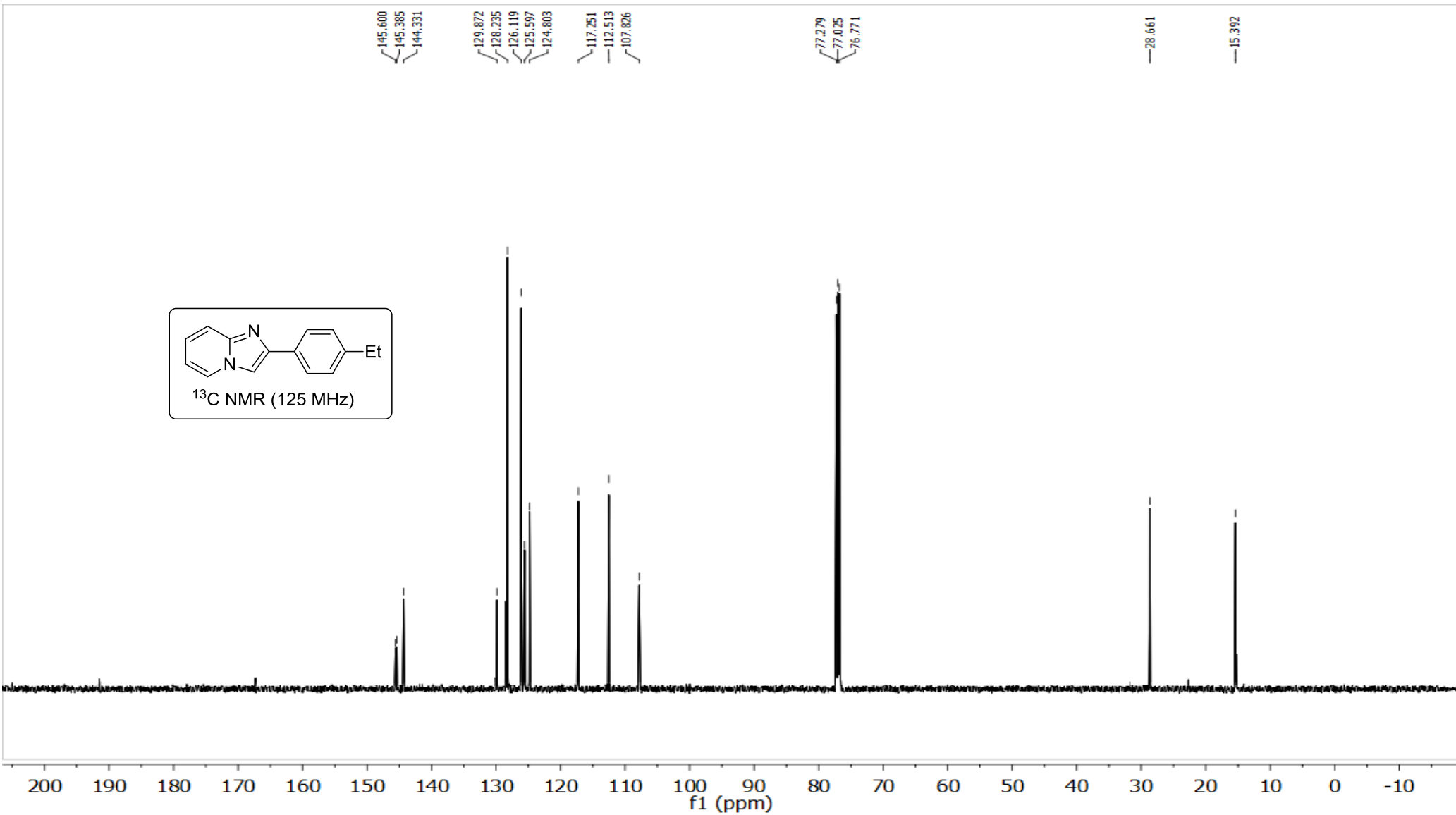
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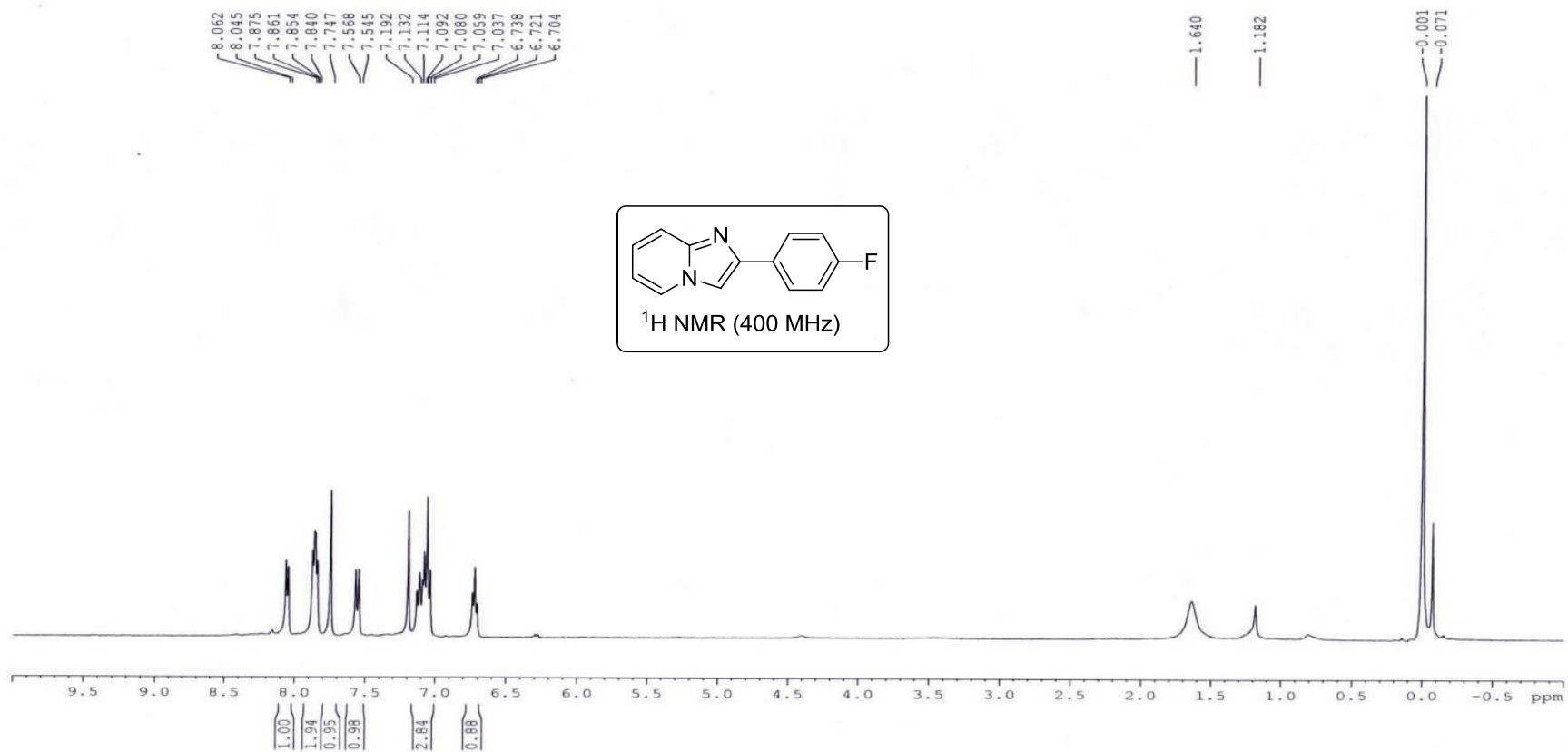
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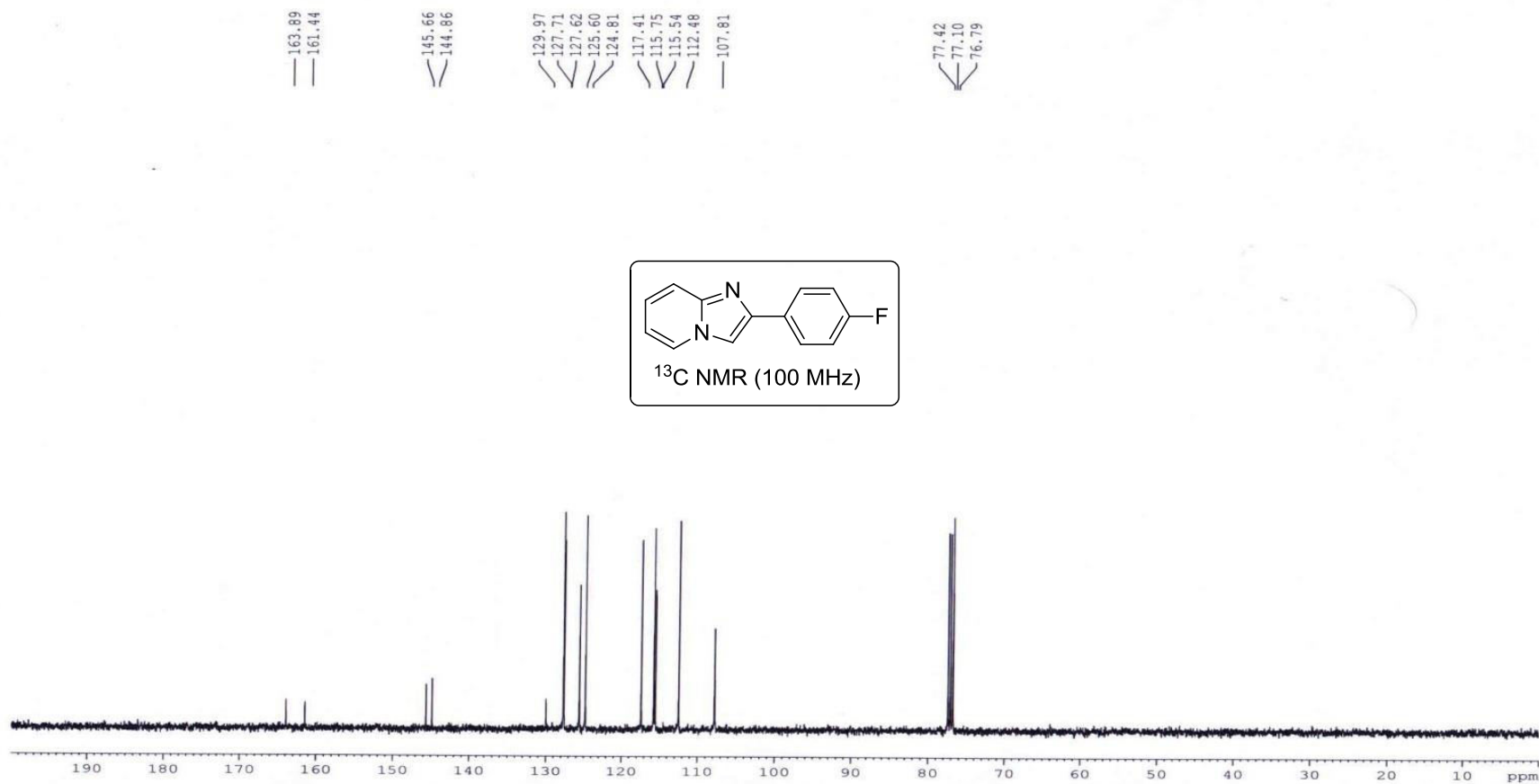
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128.235
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28.661
15.392



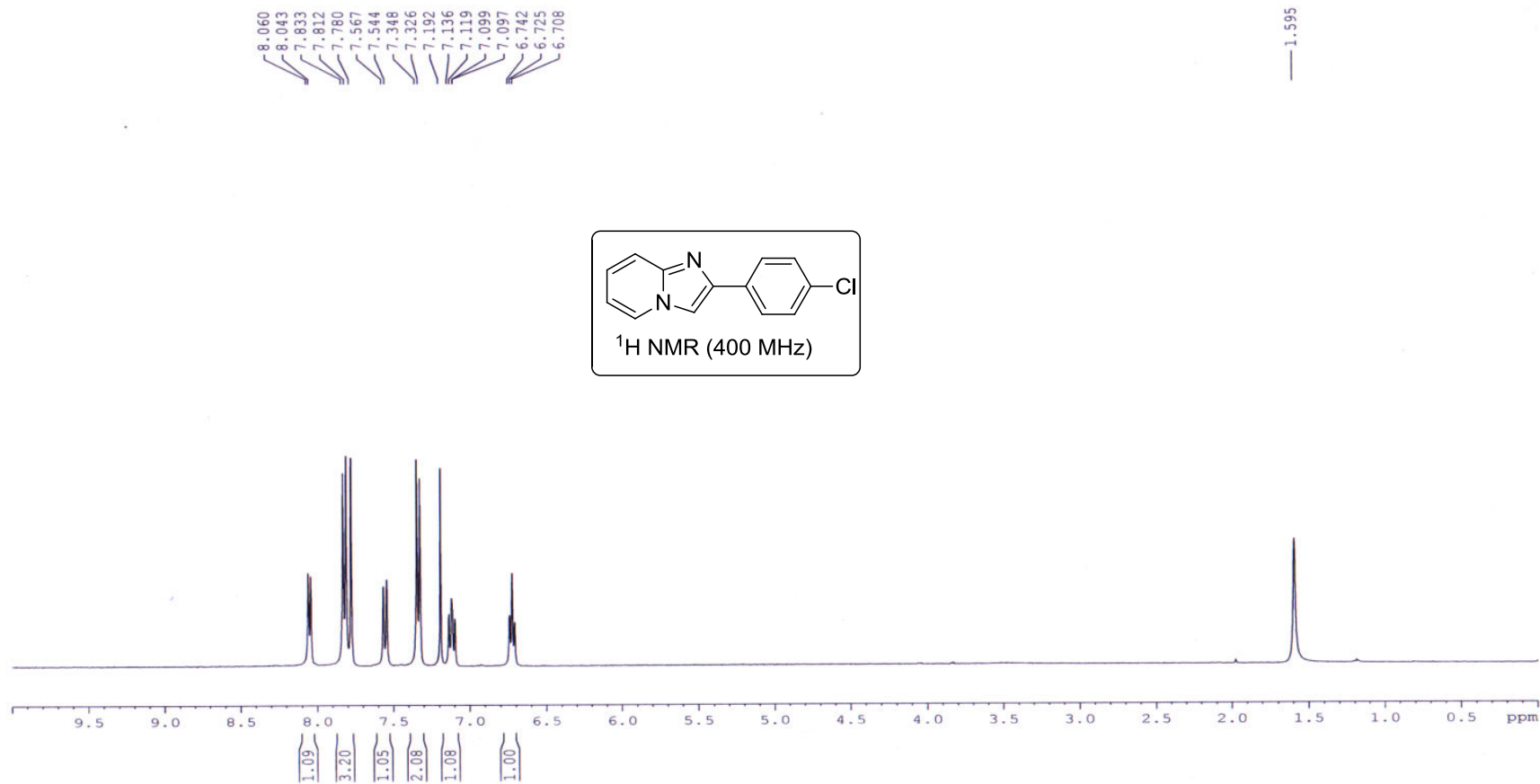
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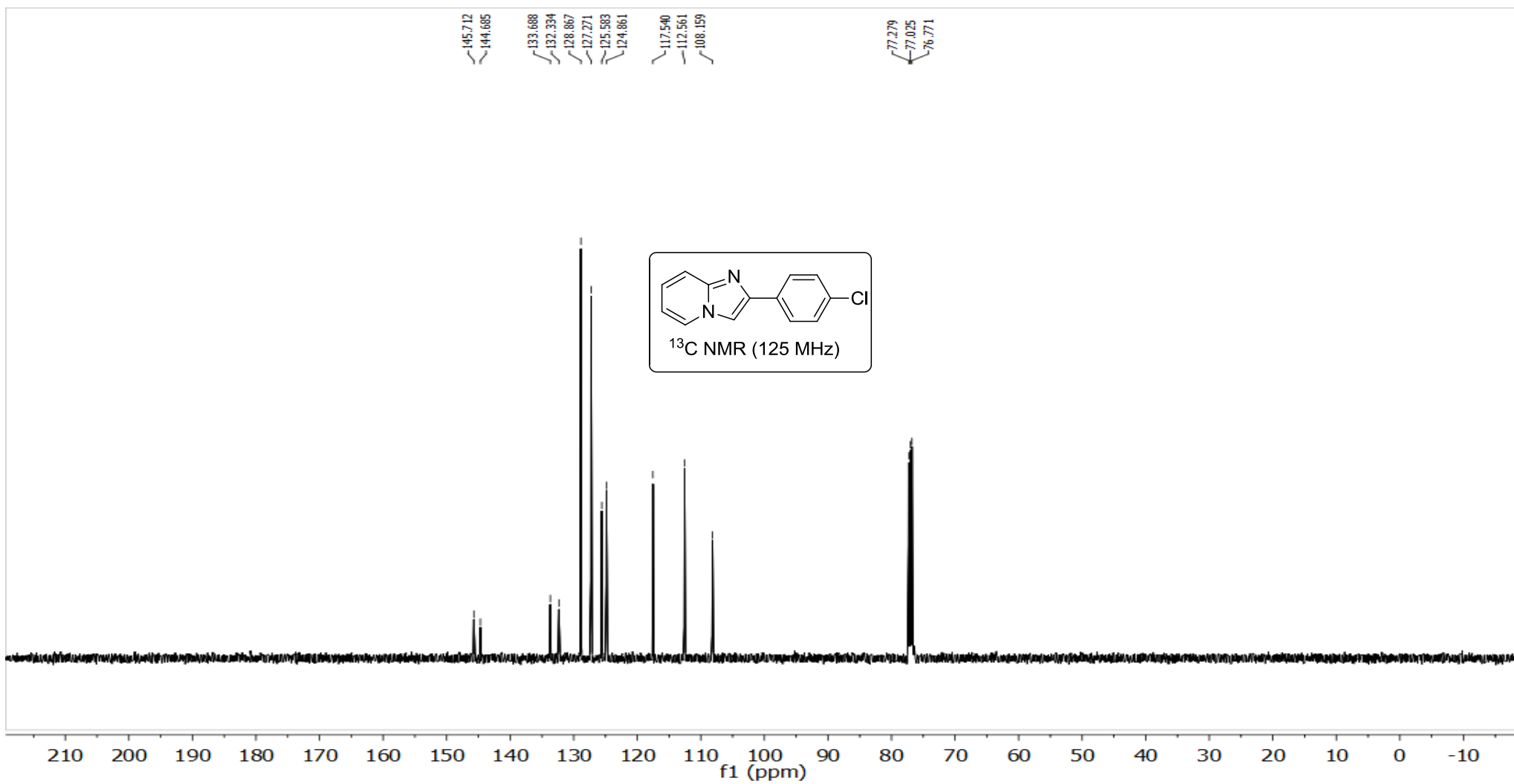
2-(4-fluorophenyl)imidazo[1,2-*a*]pyridine (3f) :



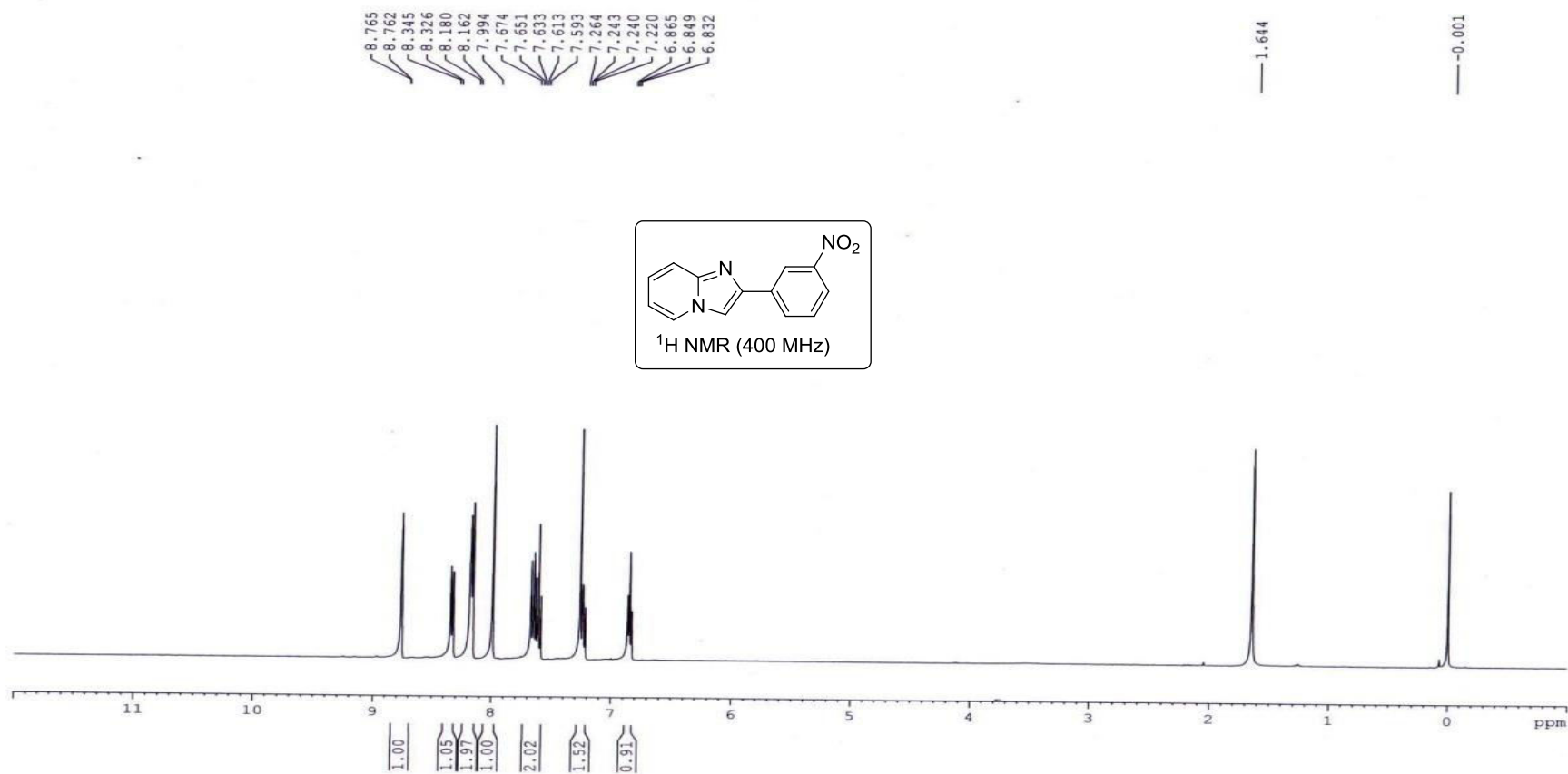
2-(4-chlorophenyl)imidazo[1,2-a]pyridine (3g) :



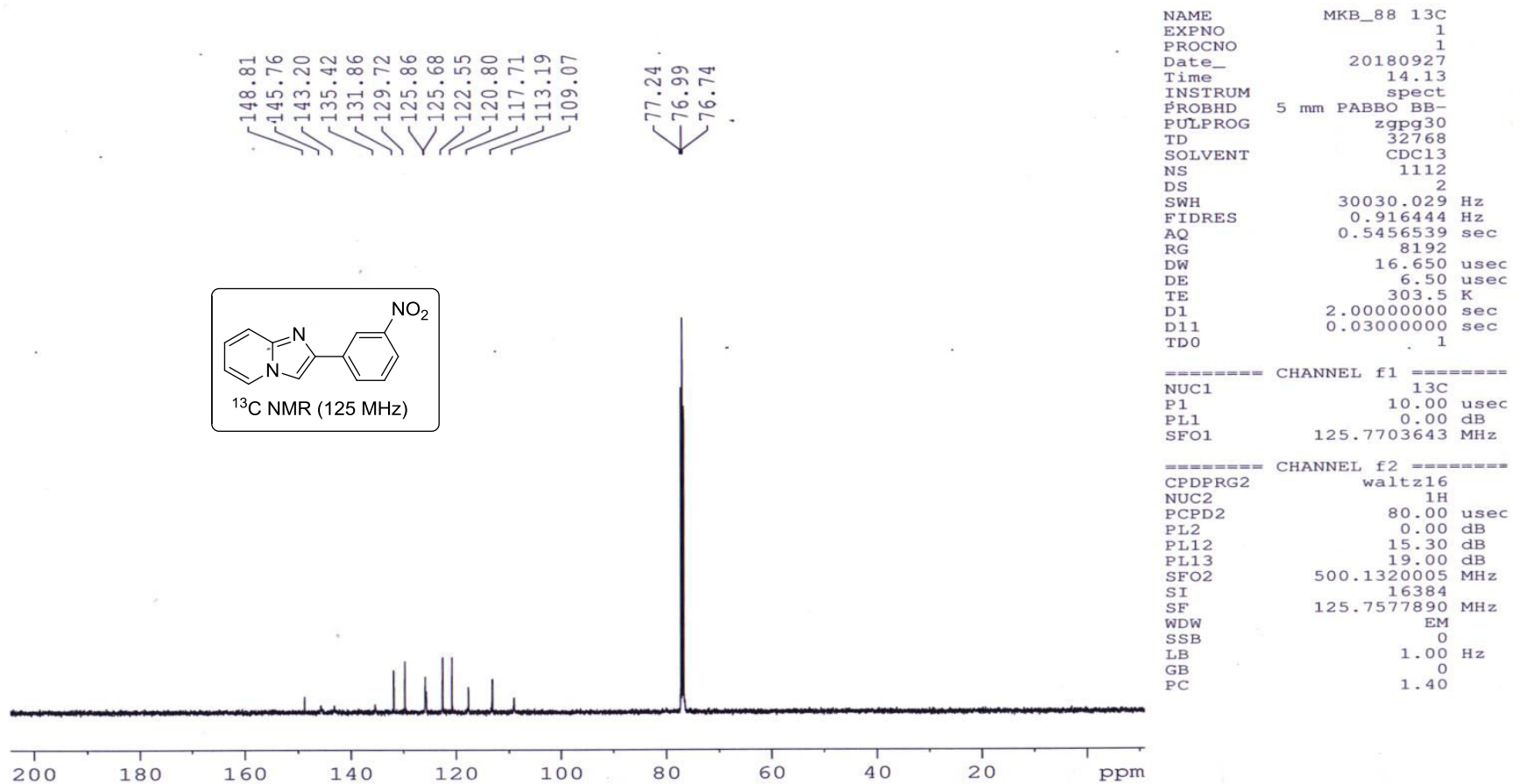
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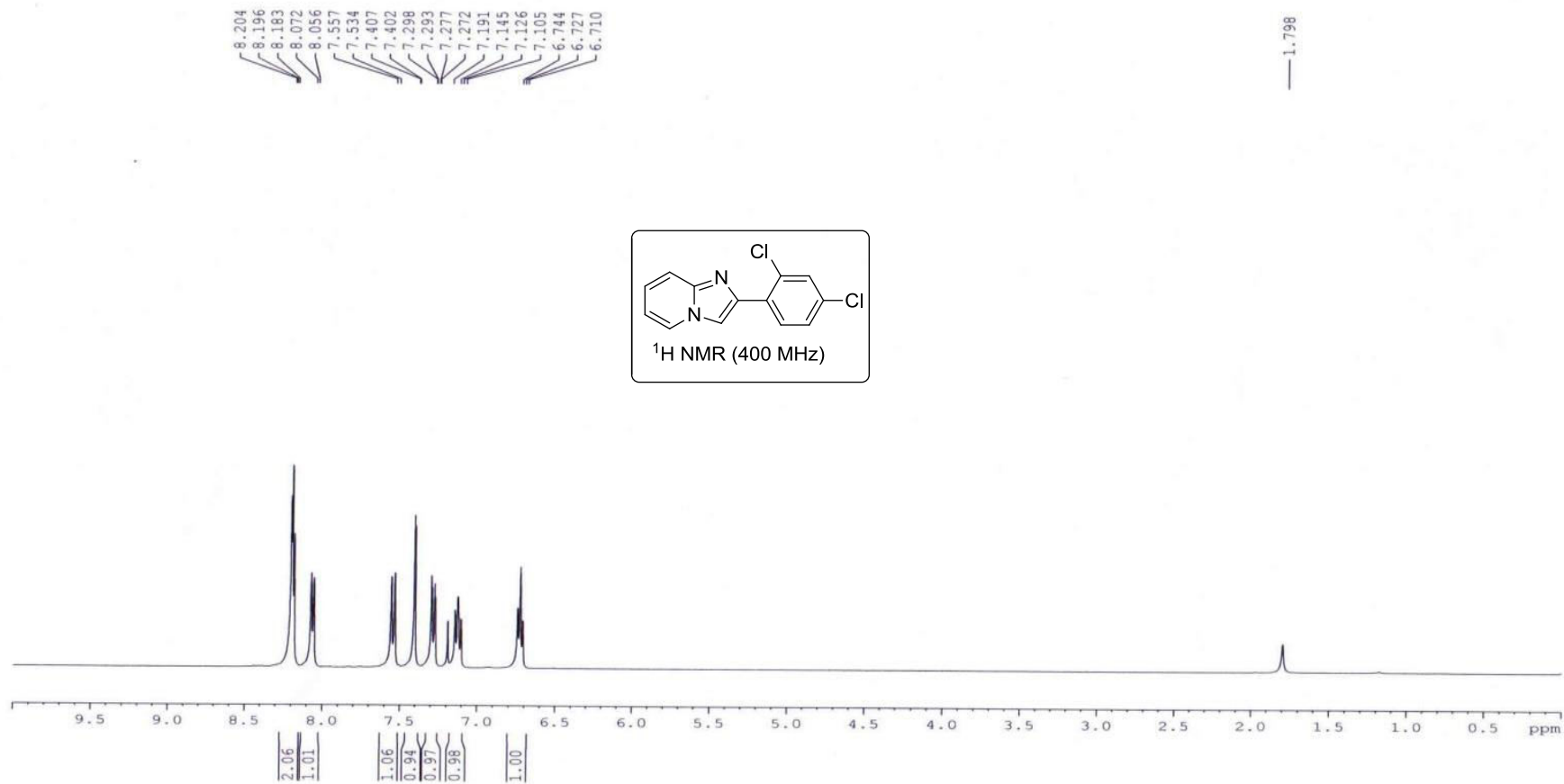
2-(3-nitrophenyl)imidazo[1,2-a]pyridine (3h) :



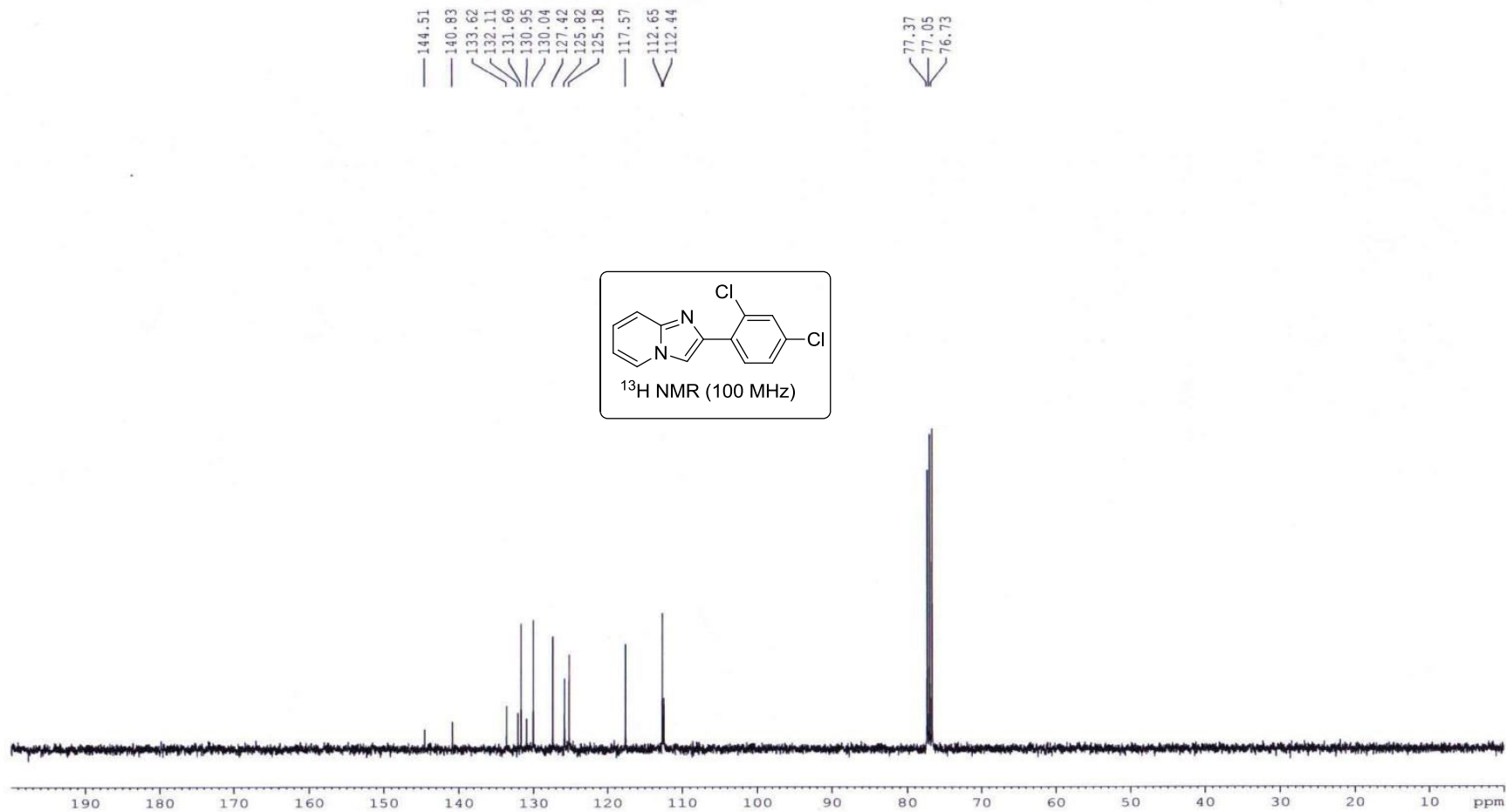
2-(3-nitrophenyl)imidazo[1,2-a]pyridine (3h) :



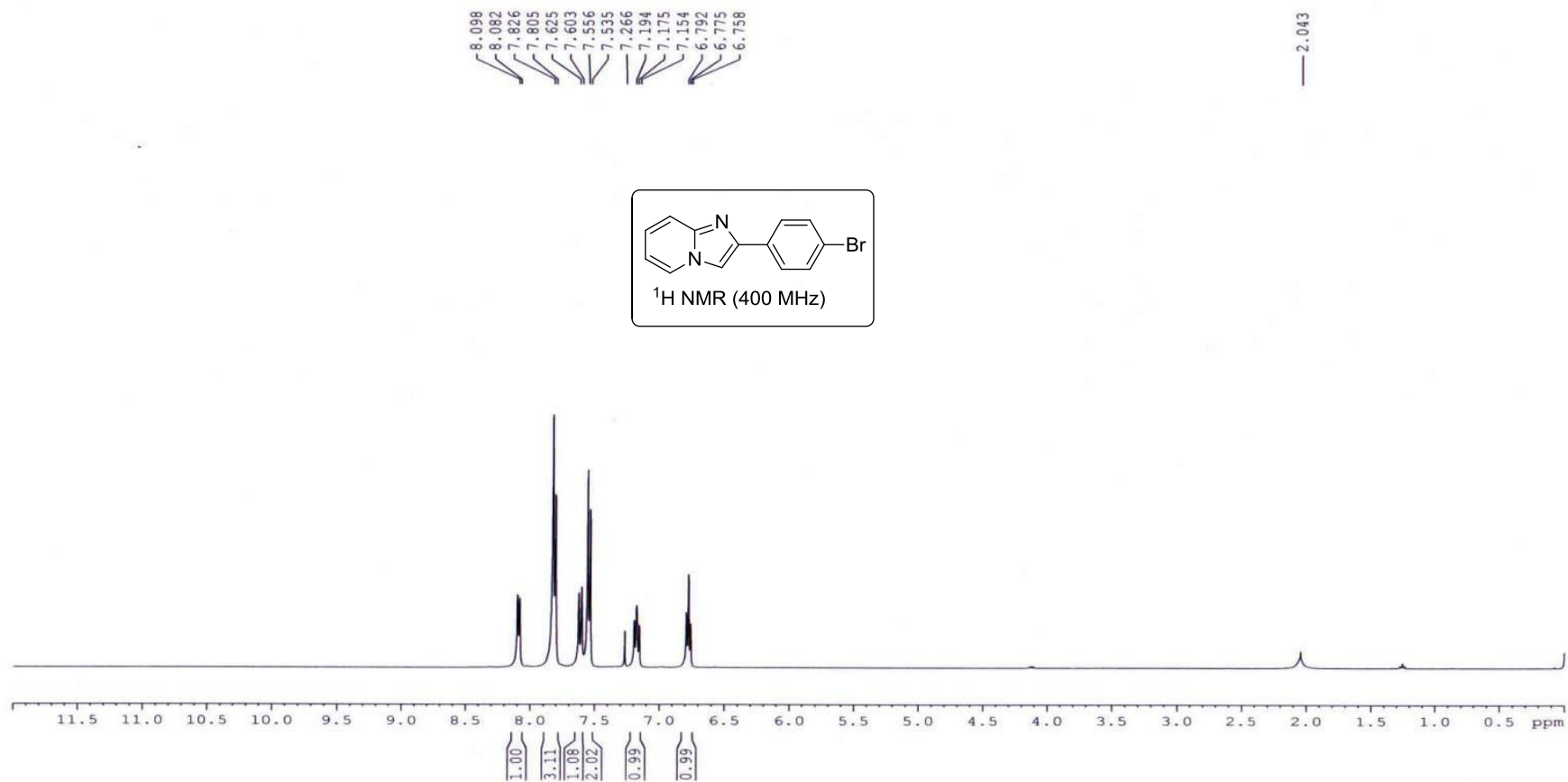
2-(3,4-dichlorophenyl)imidazo[1,2-a]pyridine (3i) :



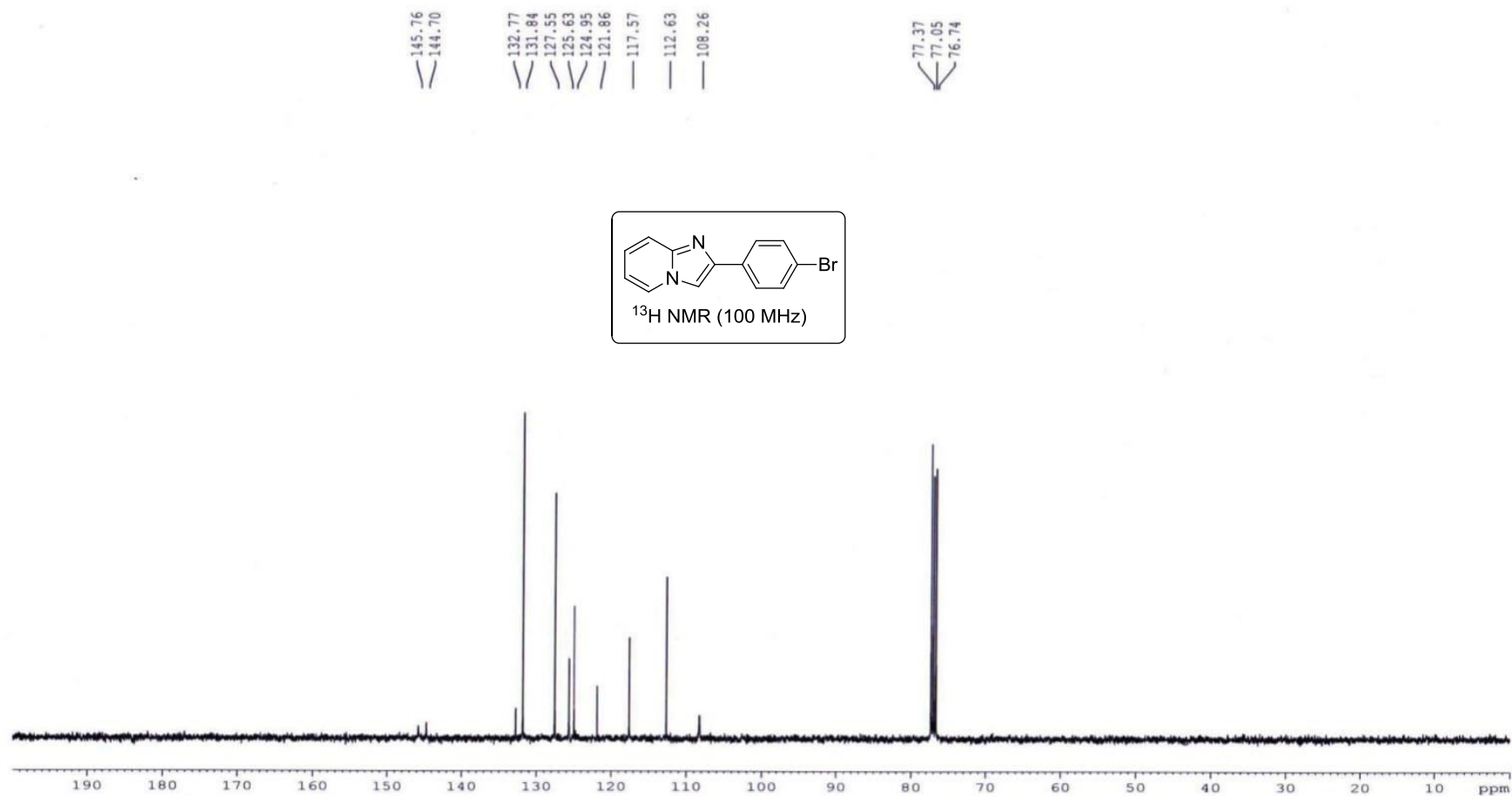
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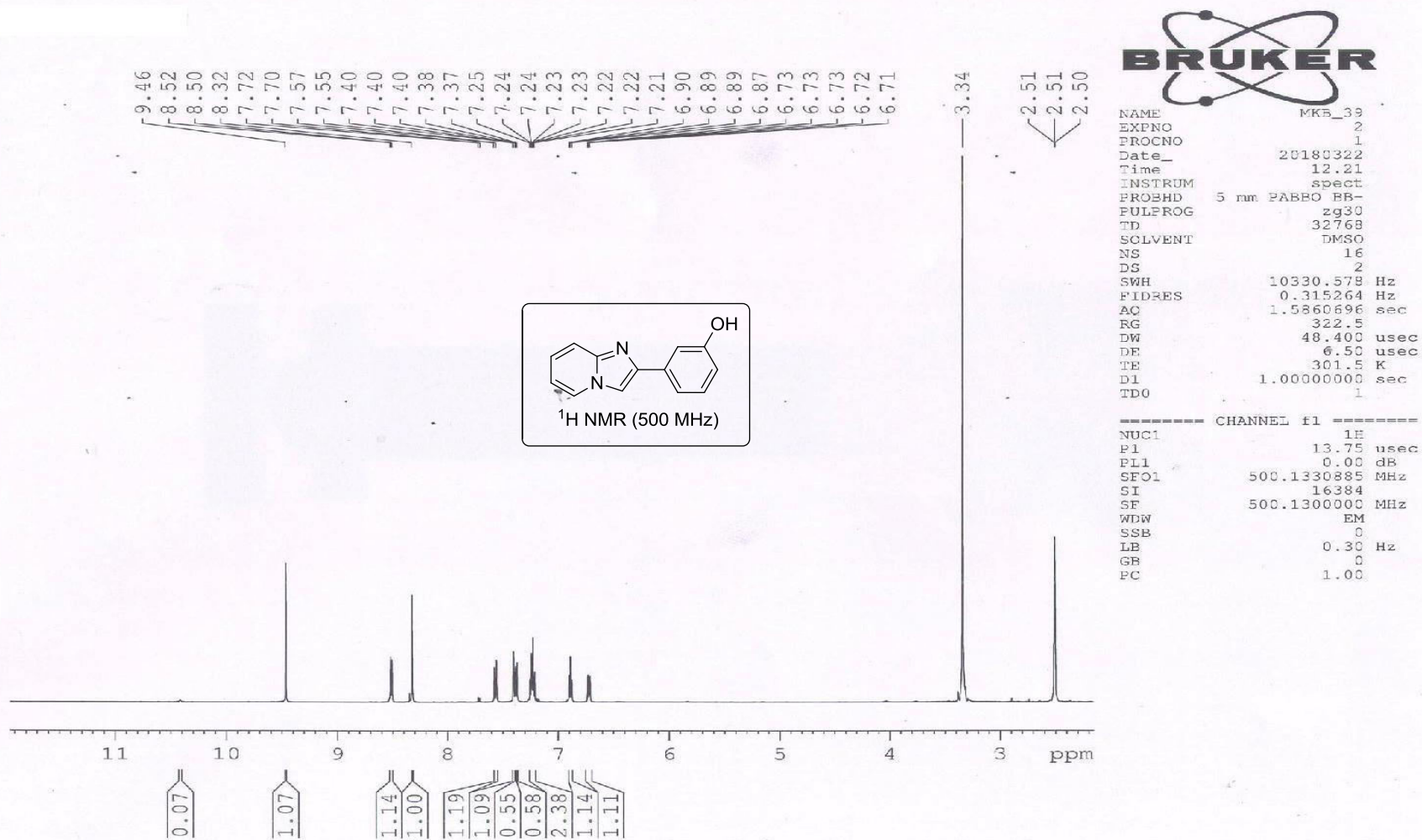
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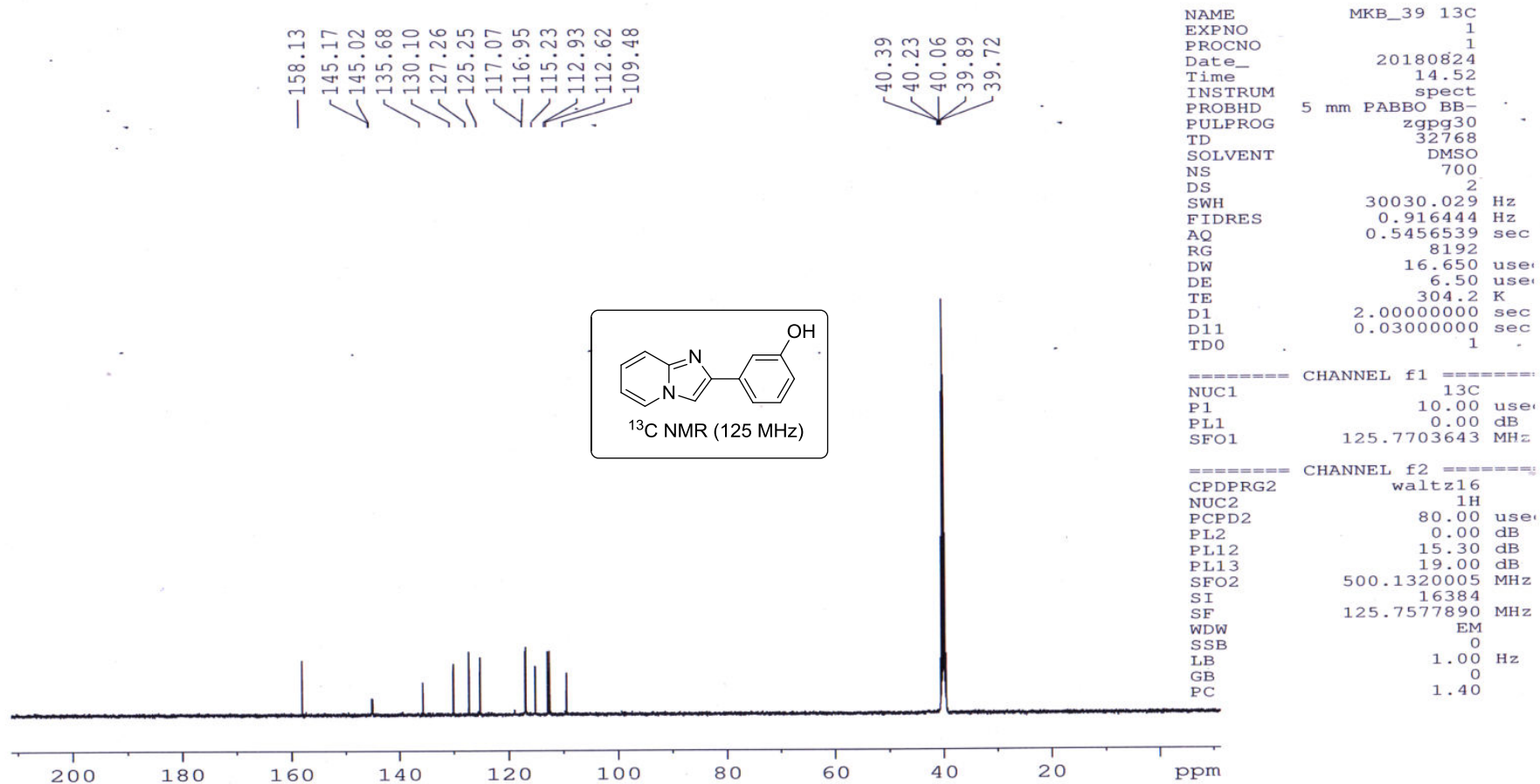
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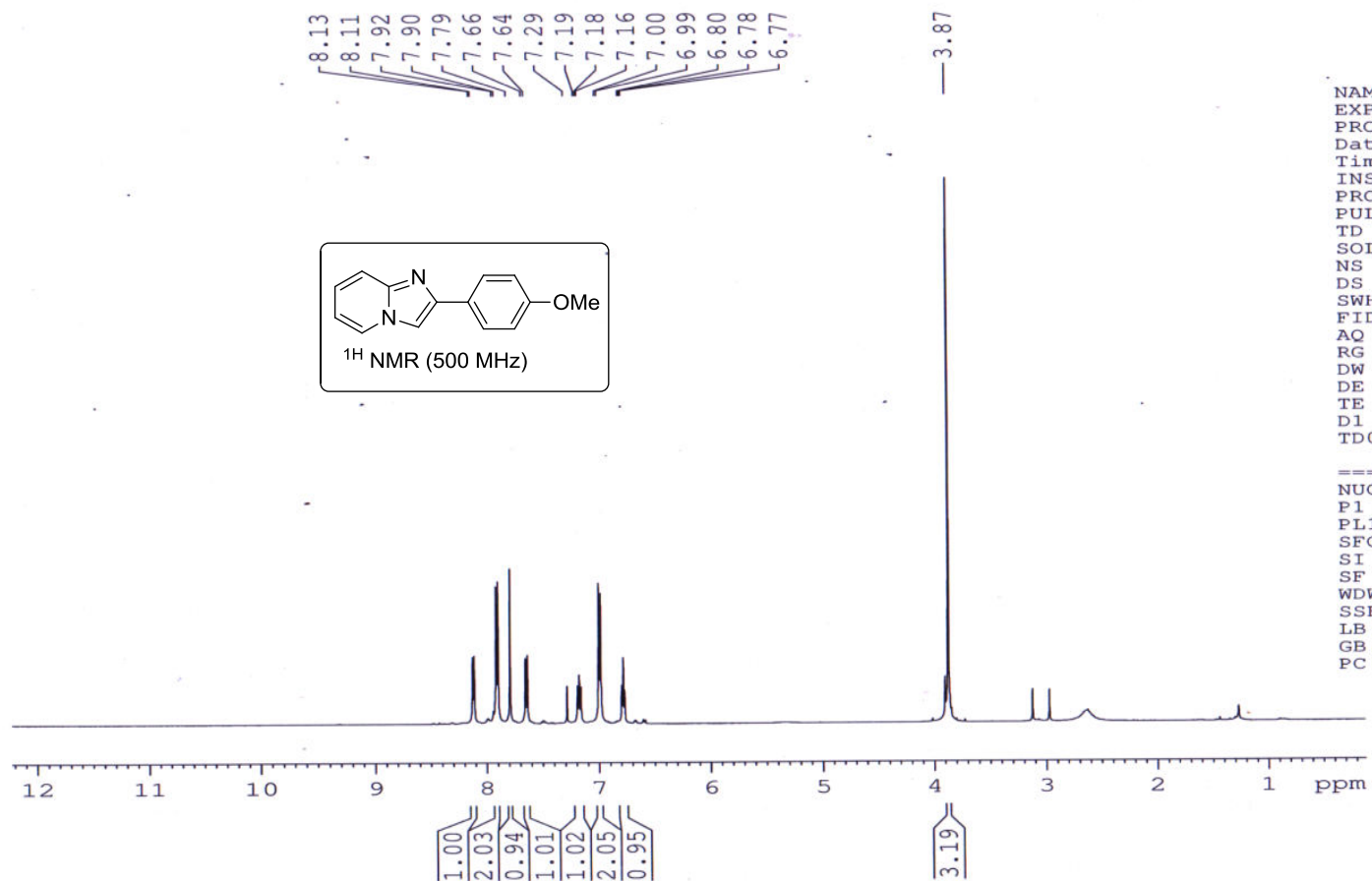
3-(imidazo[1,2-a]pyridin-2-yl)phenol (3k) :



3-(imidazo[1,2-a]pyridin-2-yl)phenol (3k) :



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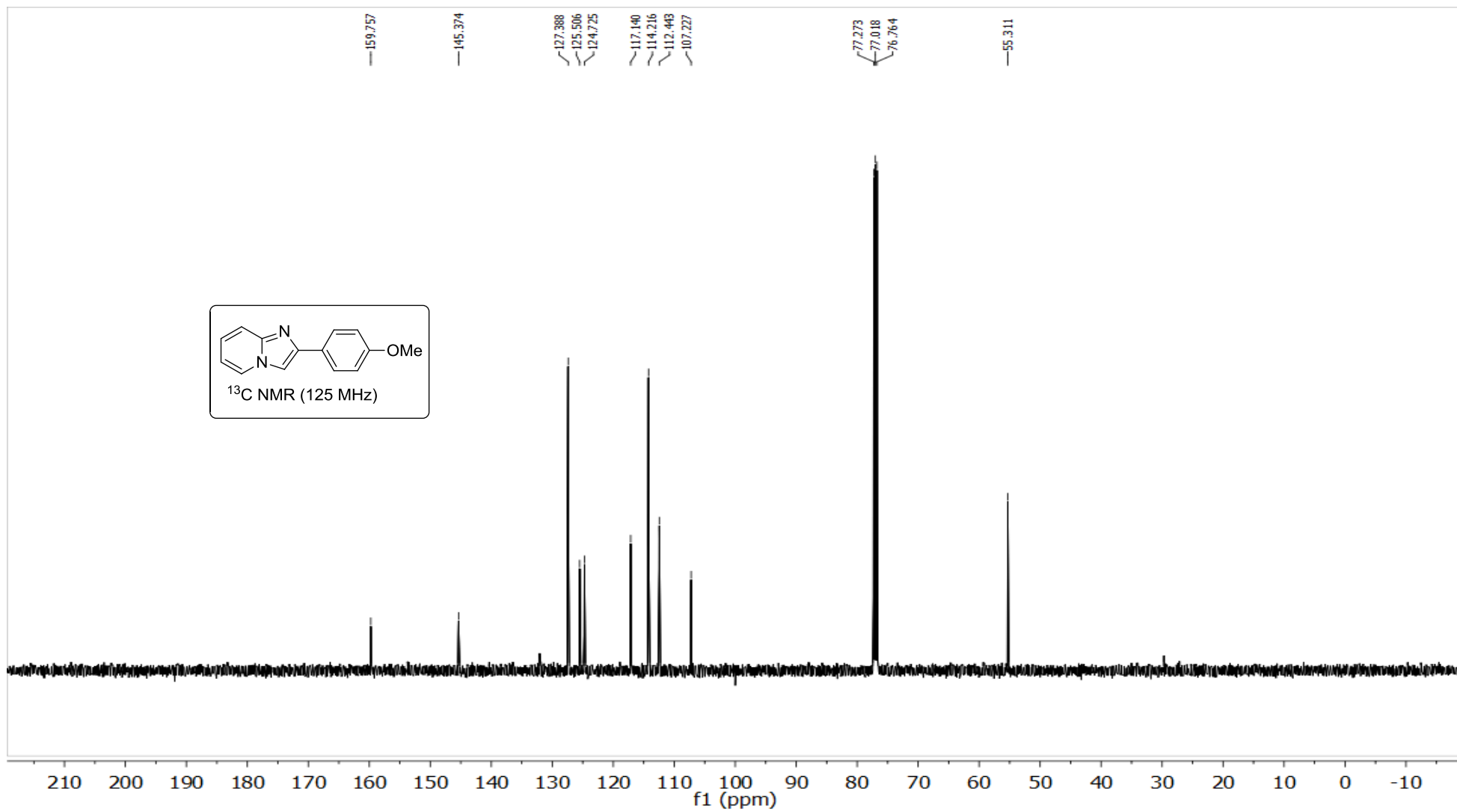


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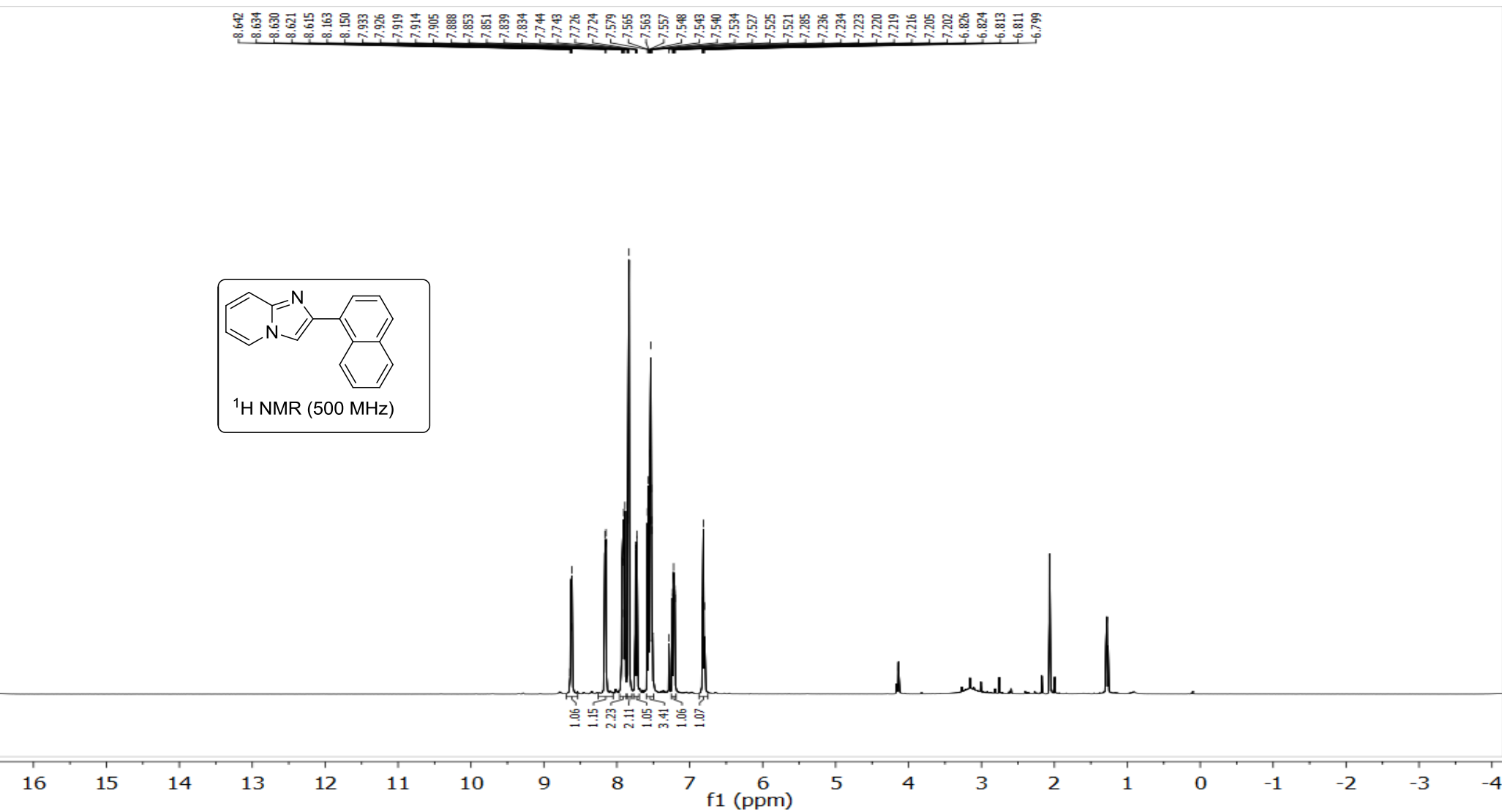
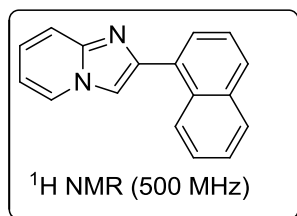
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DW            48.400 use
DE            6.50 use
TE            300.1 K
D1            1.00000000 sec
TD0           1

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SFO1          500.1330885 MHz
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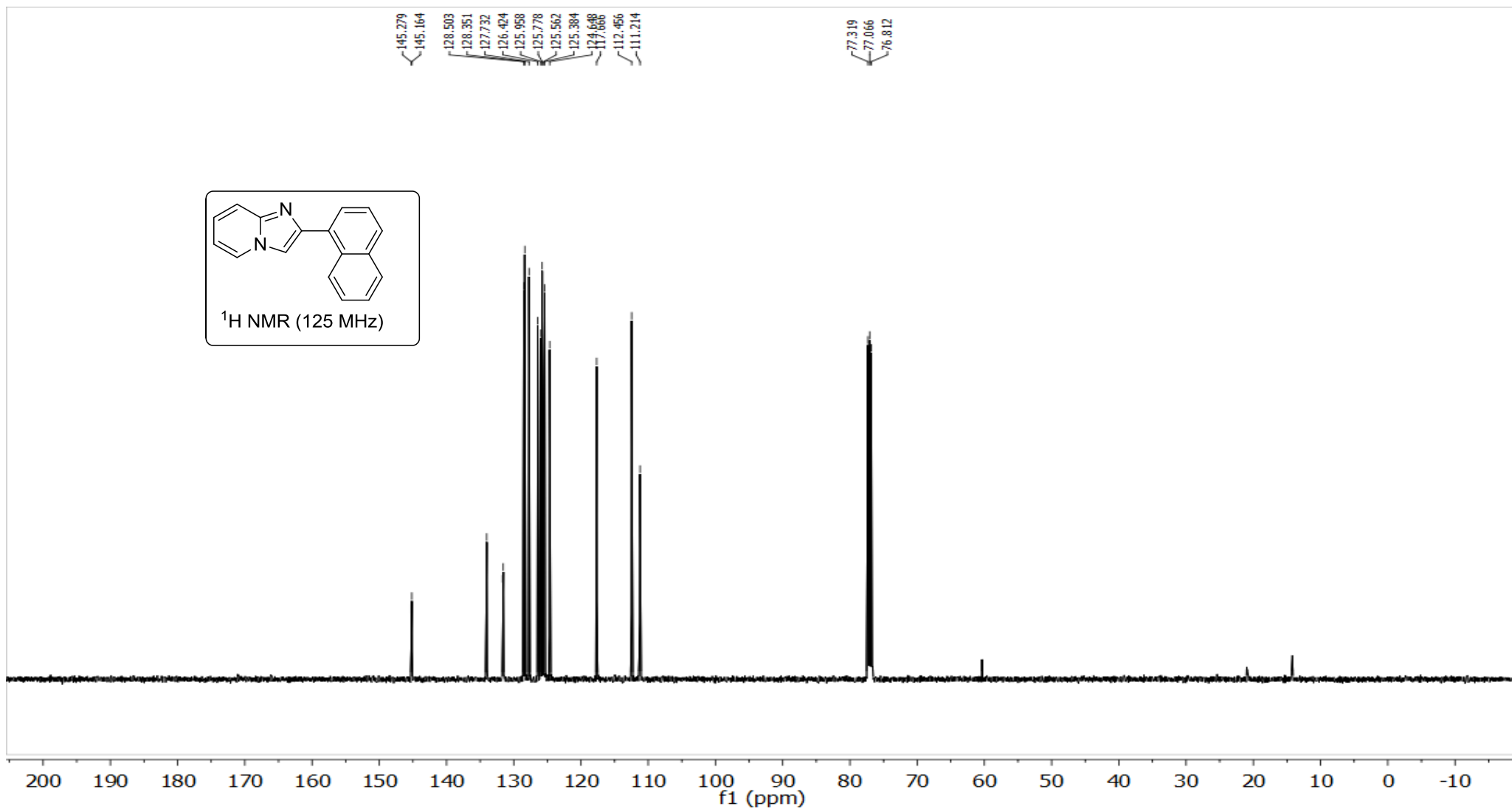
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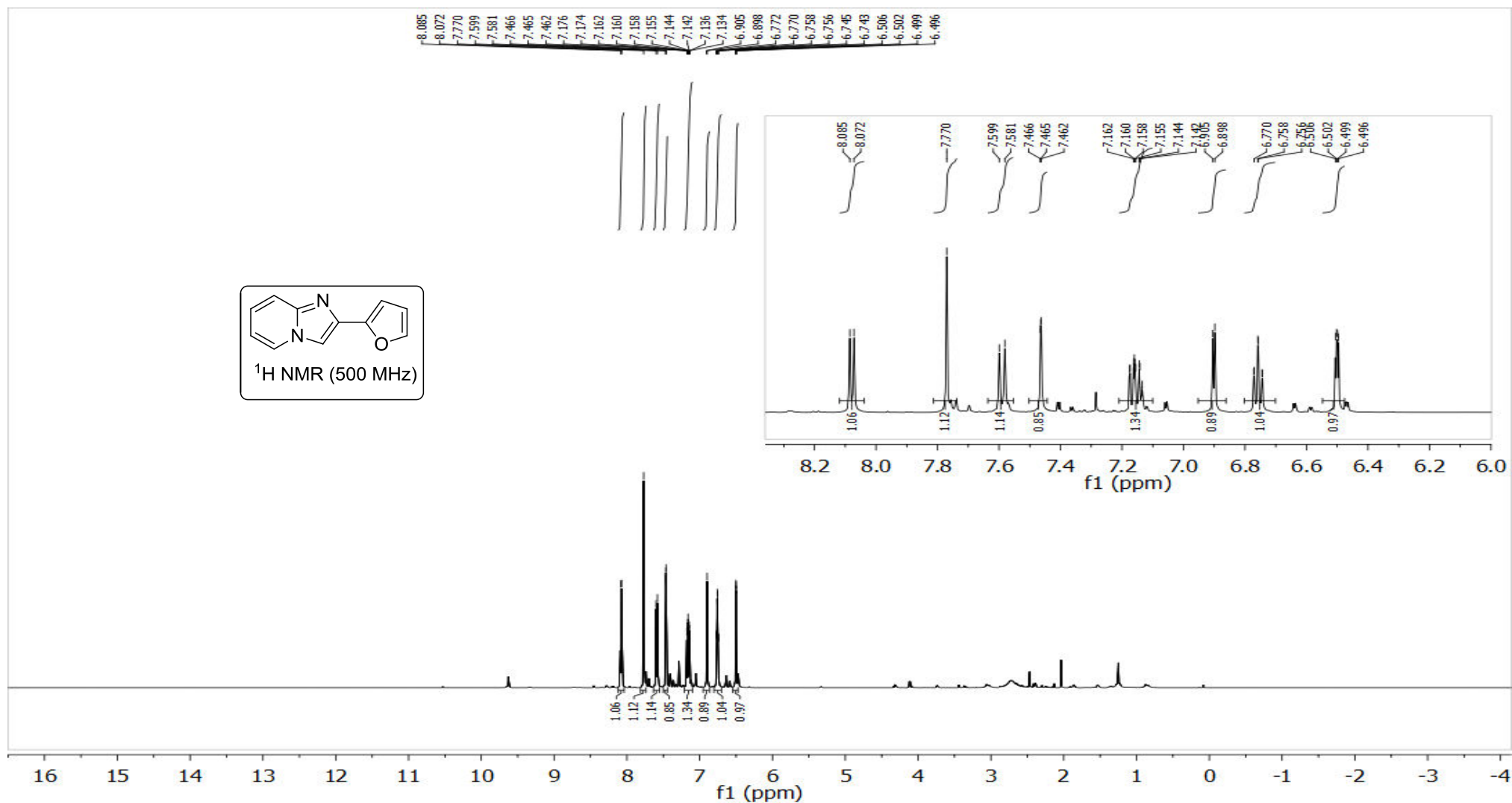
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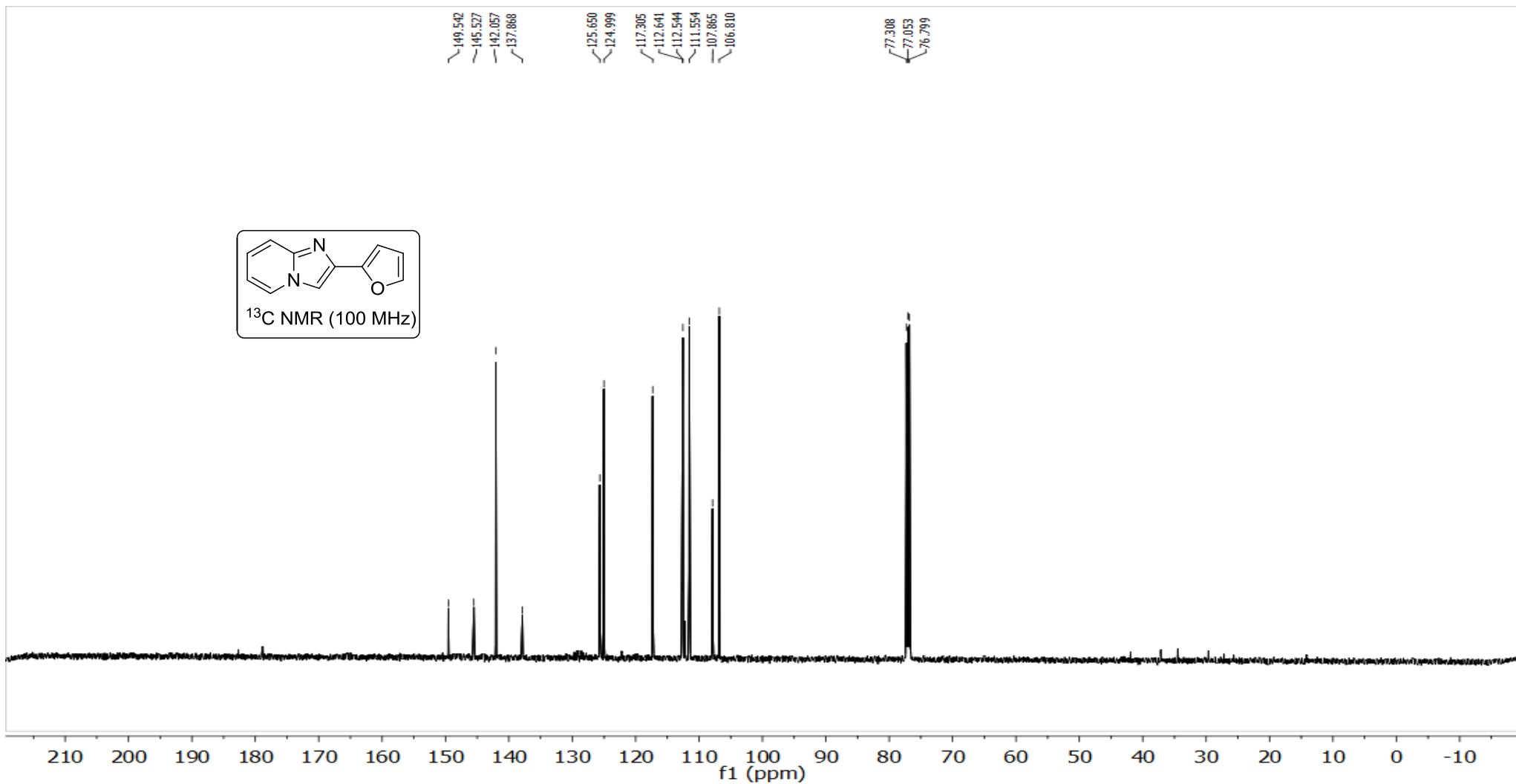
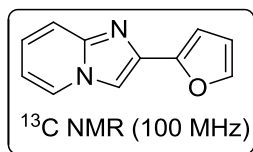
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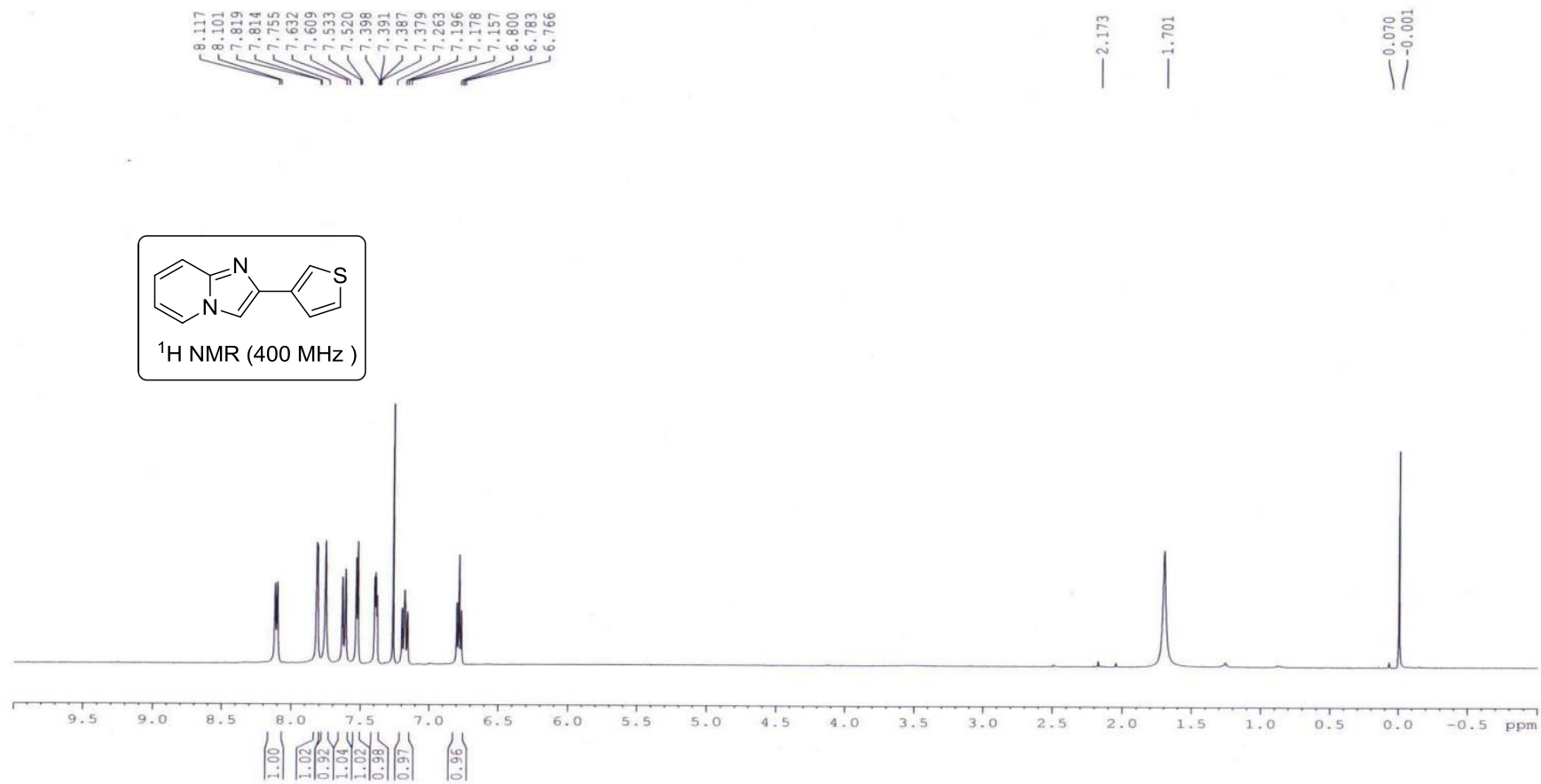
2-(furan-2-yl)imidazo[1,2-a]pyridine (3n) :



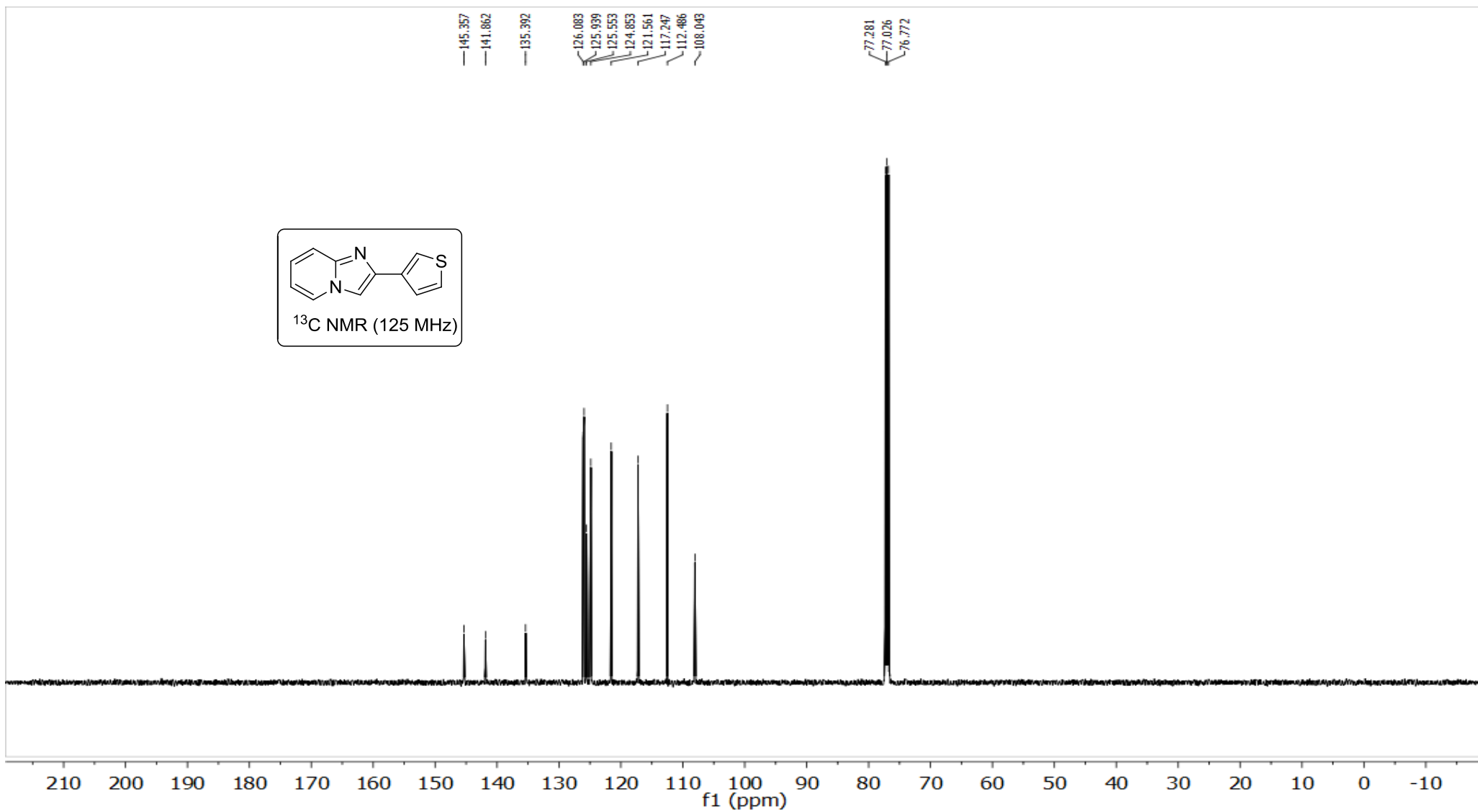
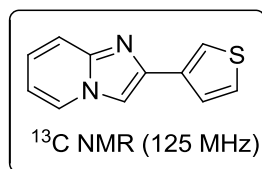
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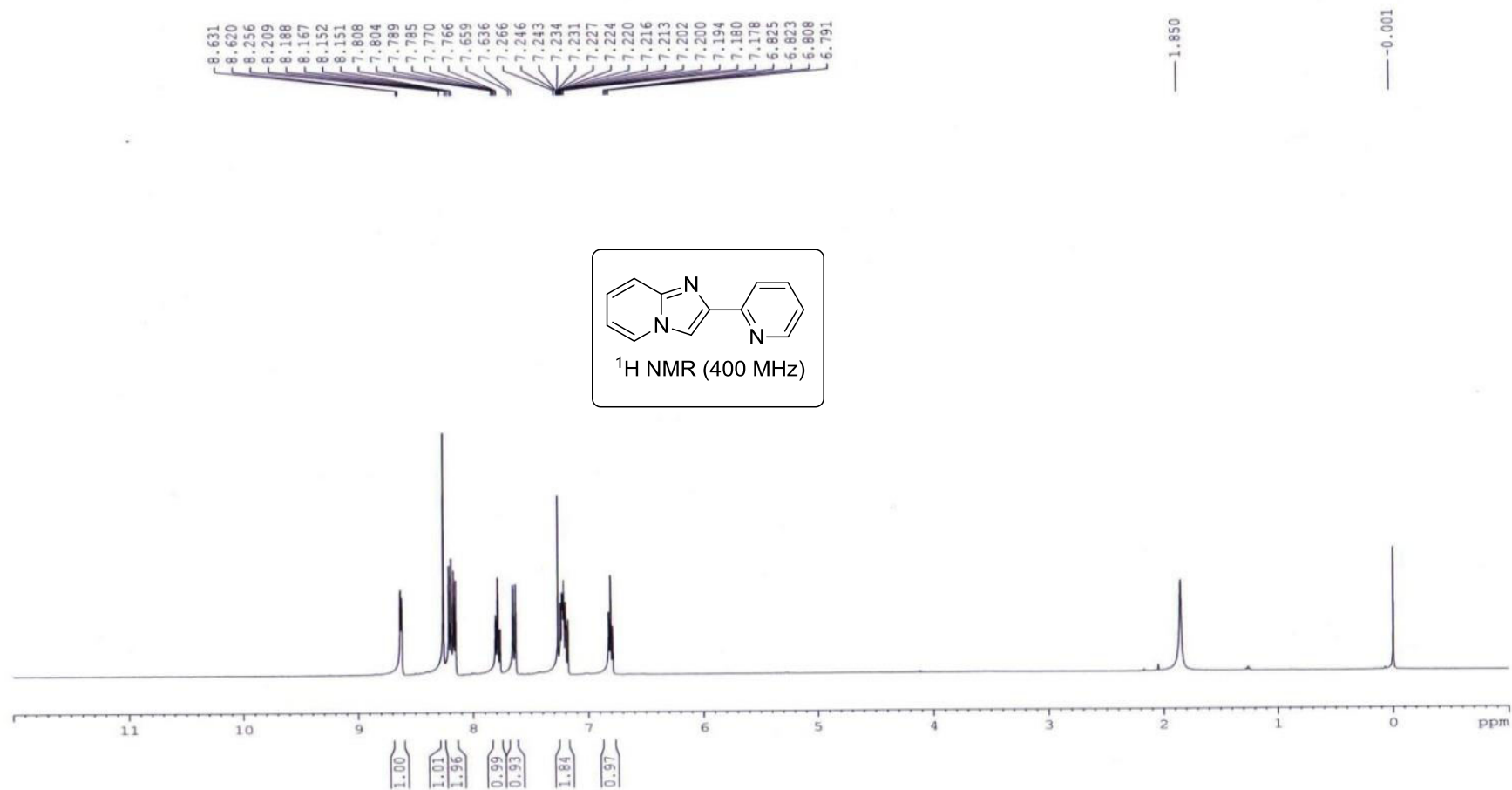
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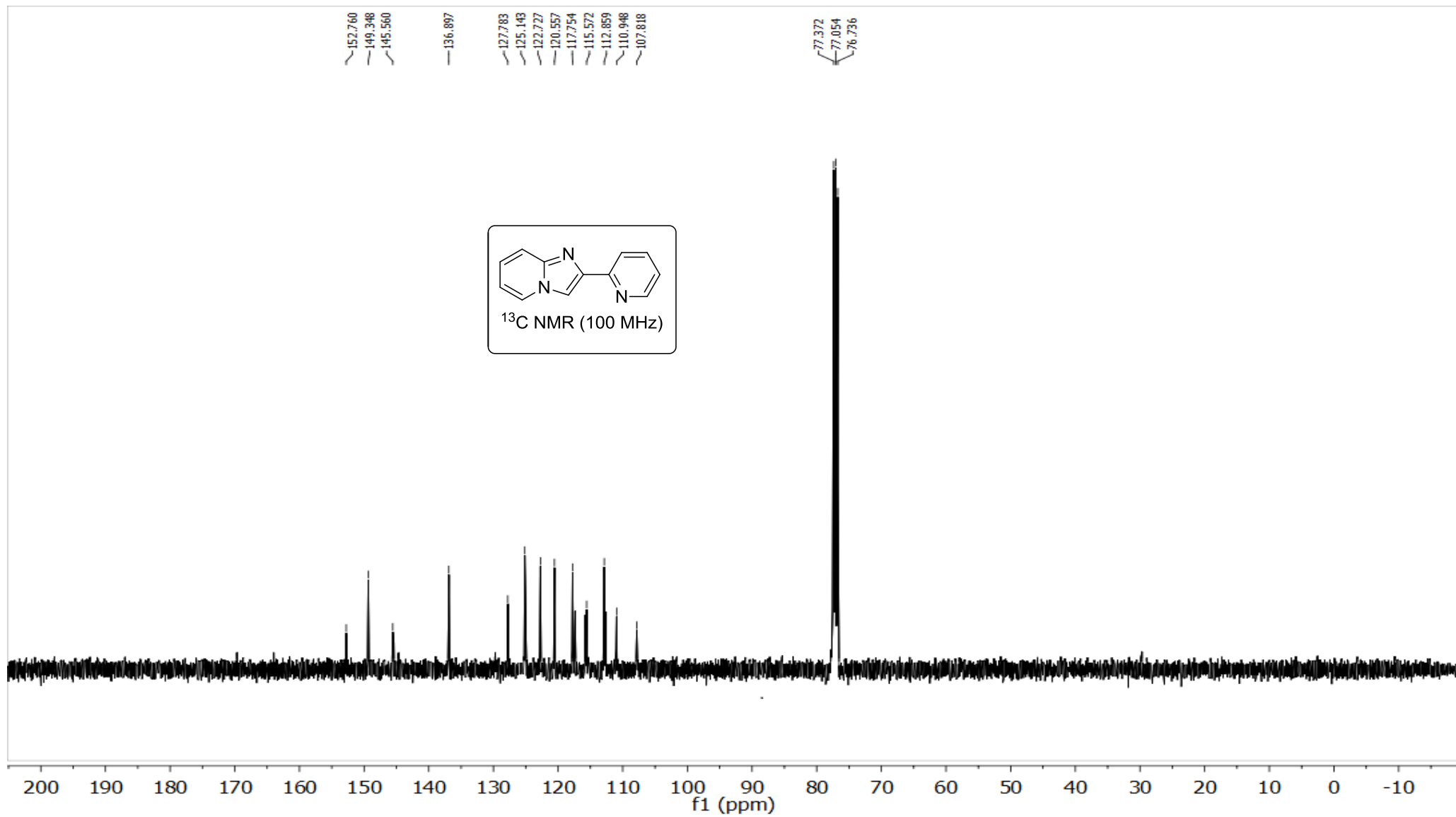
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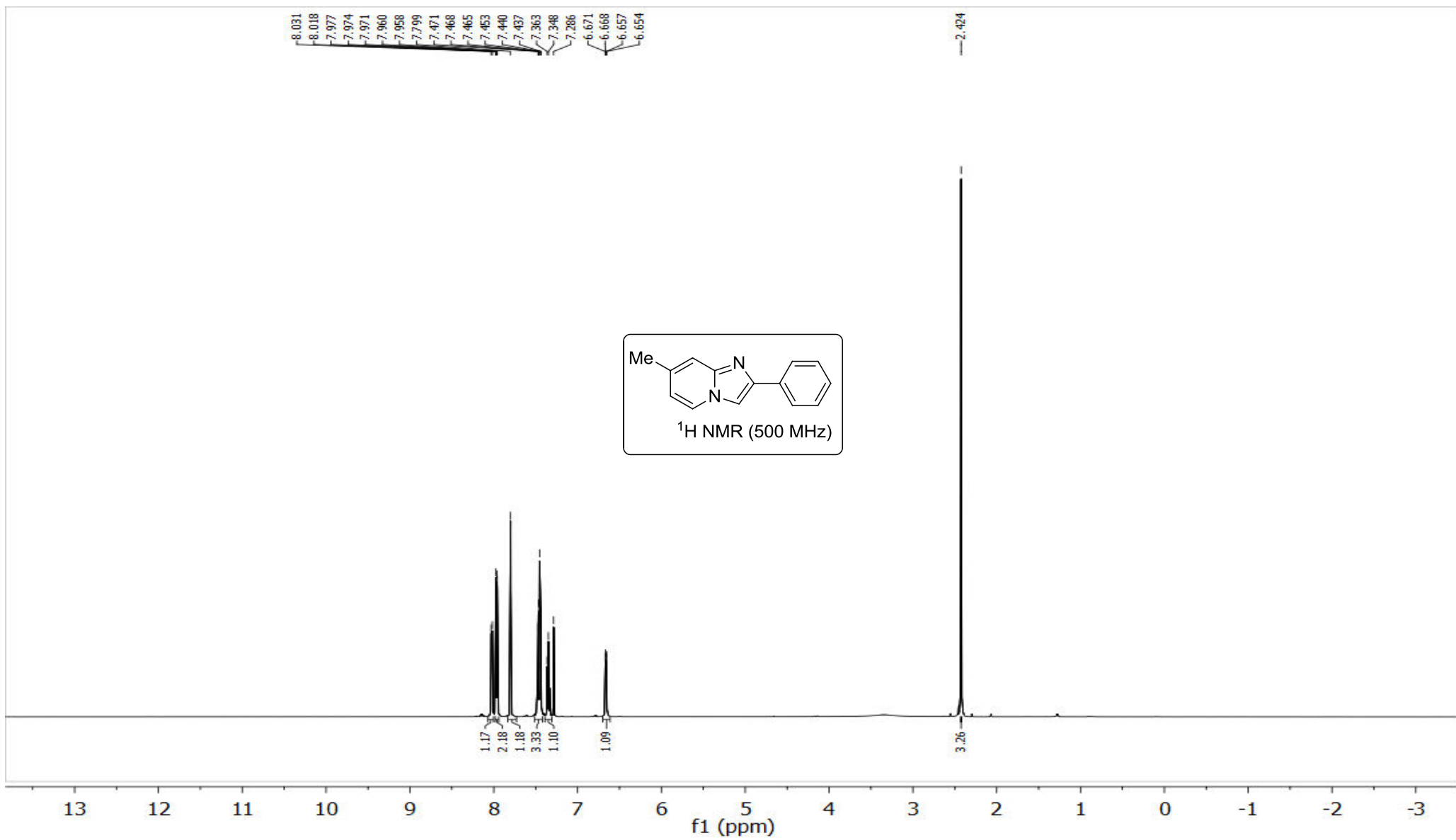
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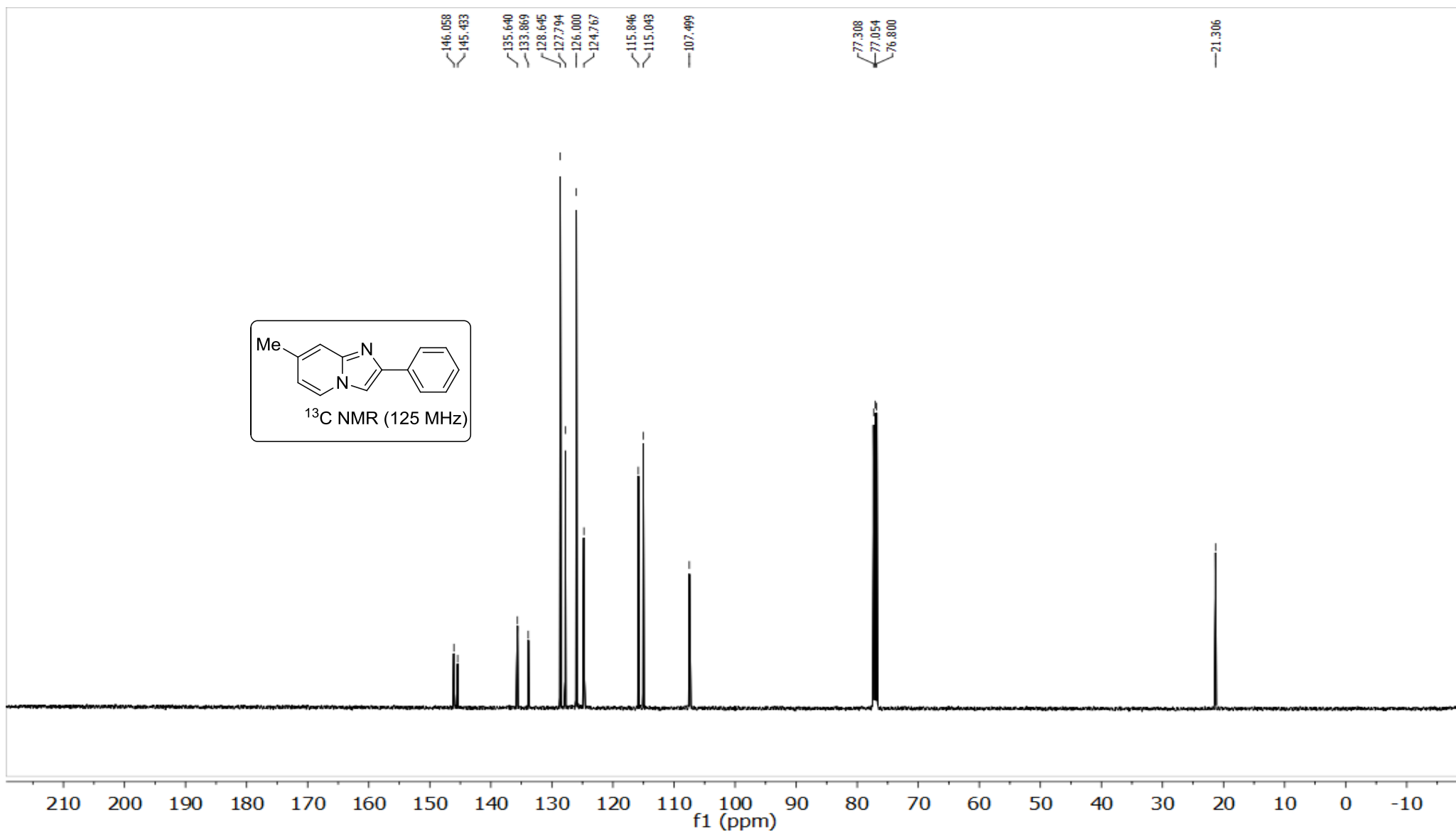
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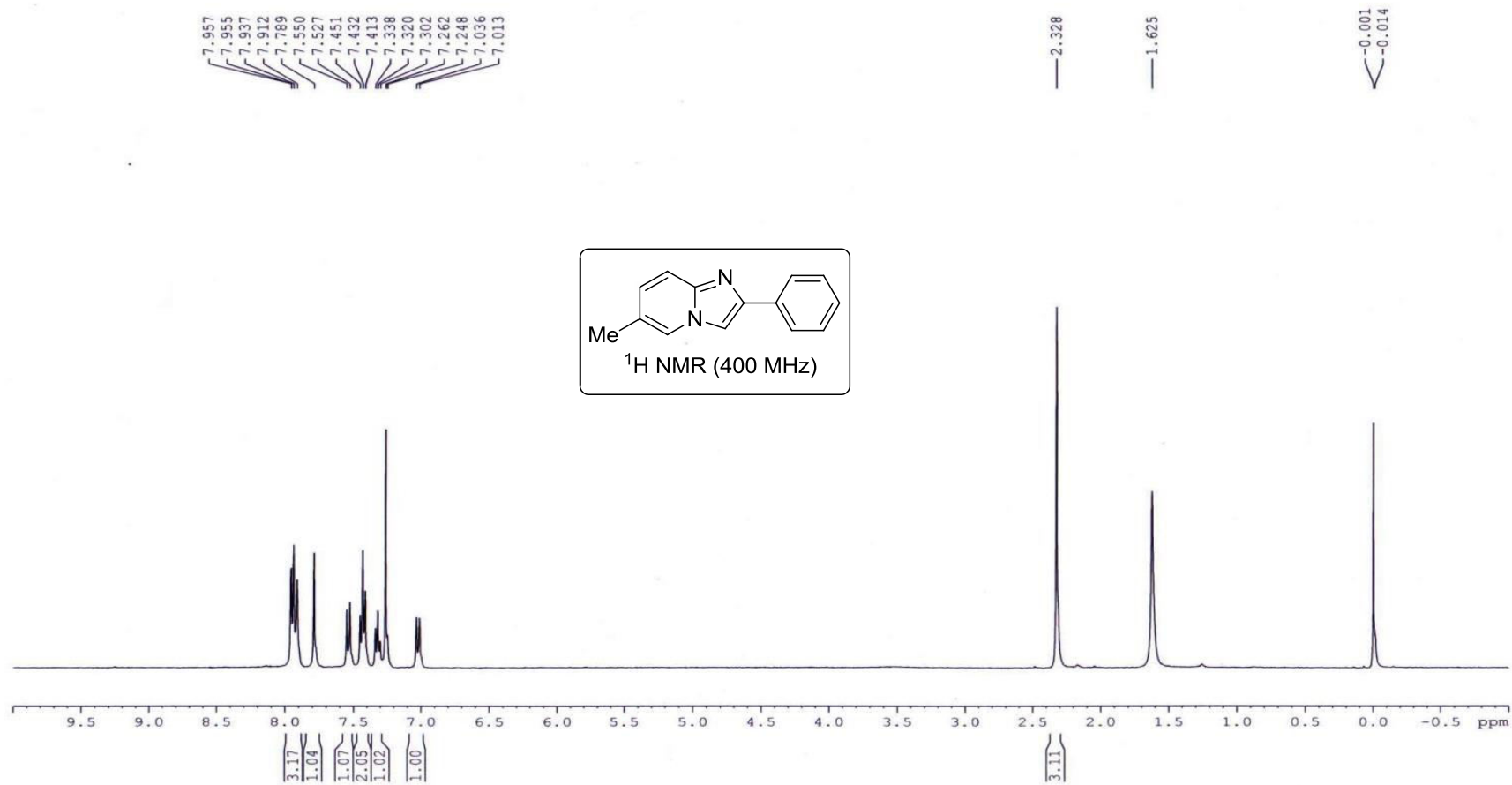
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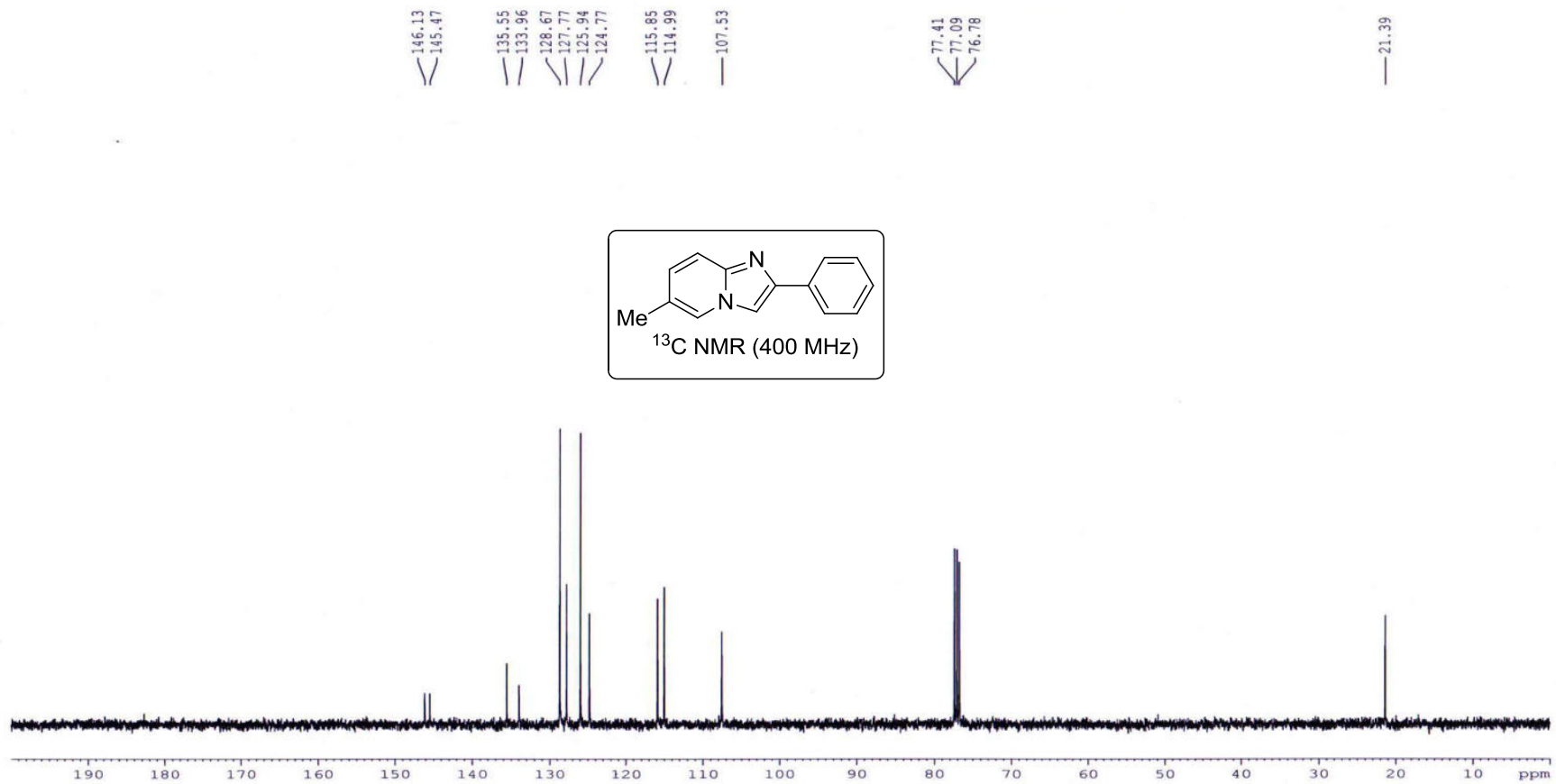
7-methyl-2-phenylimidazo[1,2-a]pyridine(3q) :



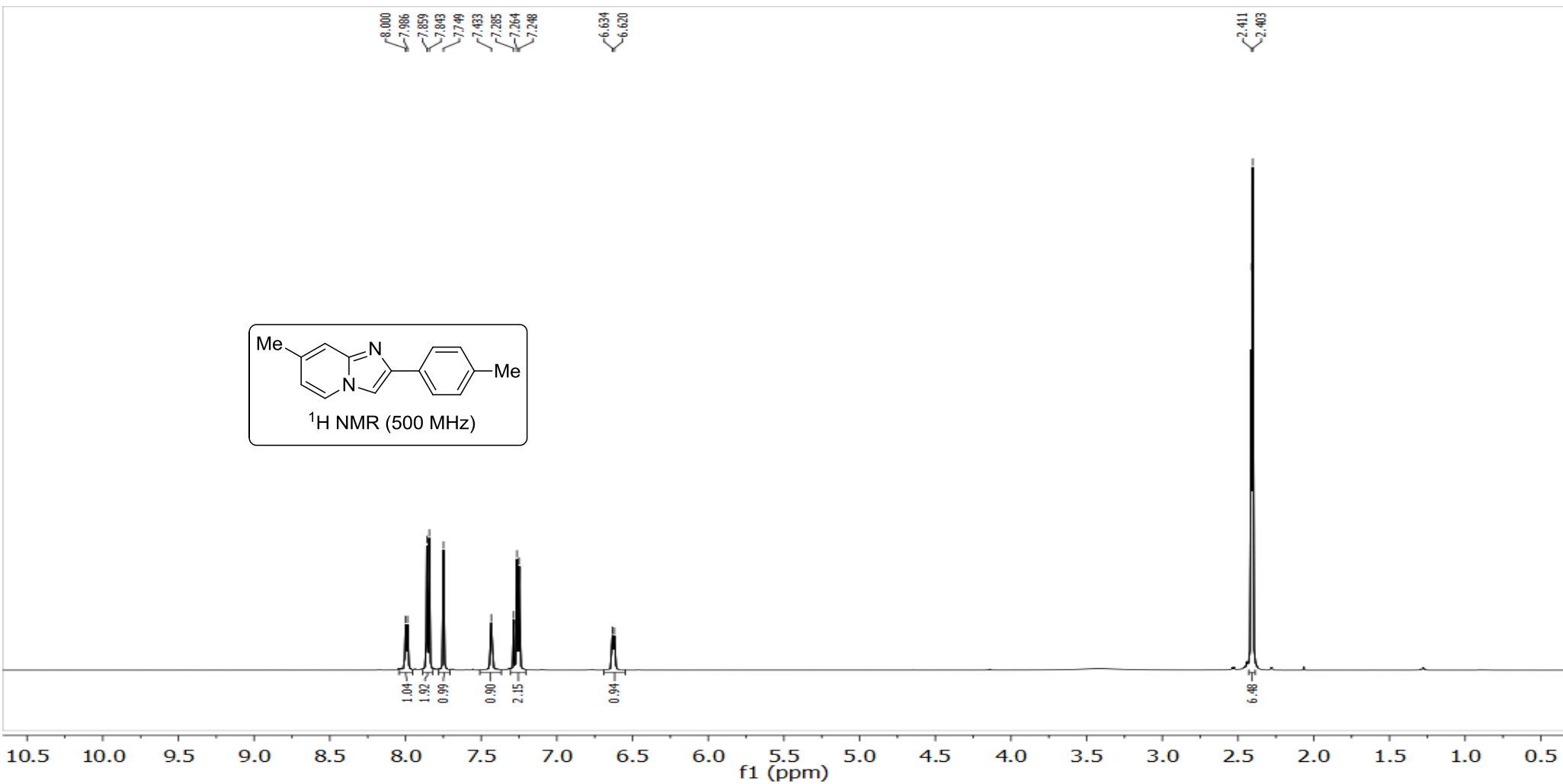
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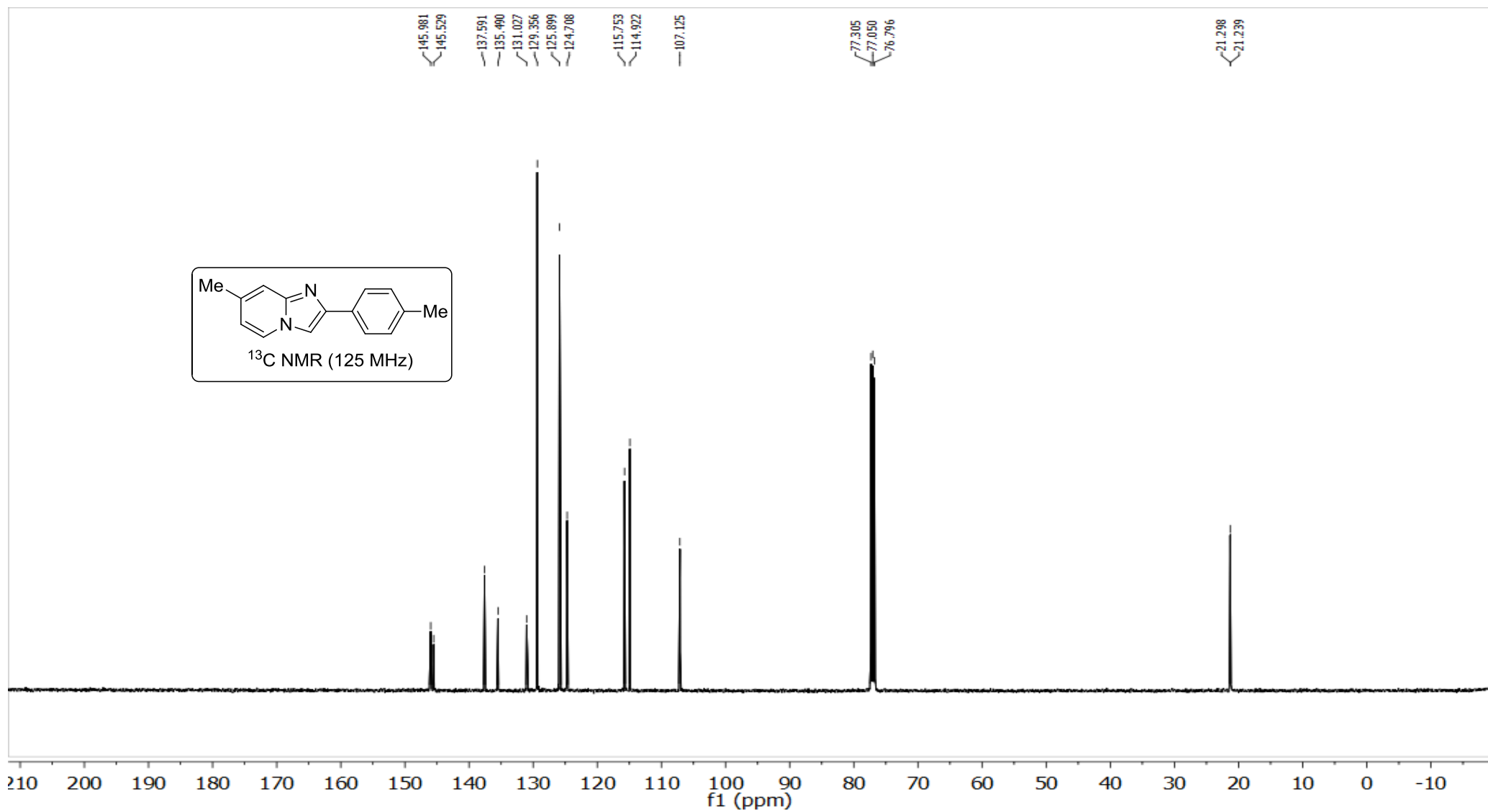
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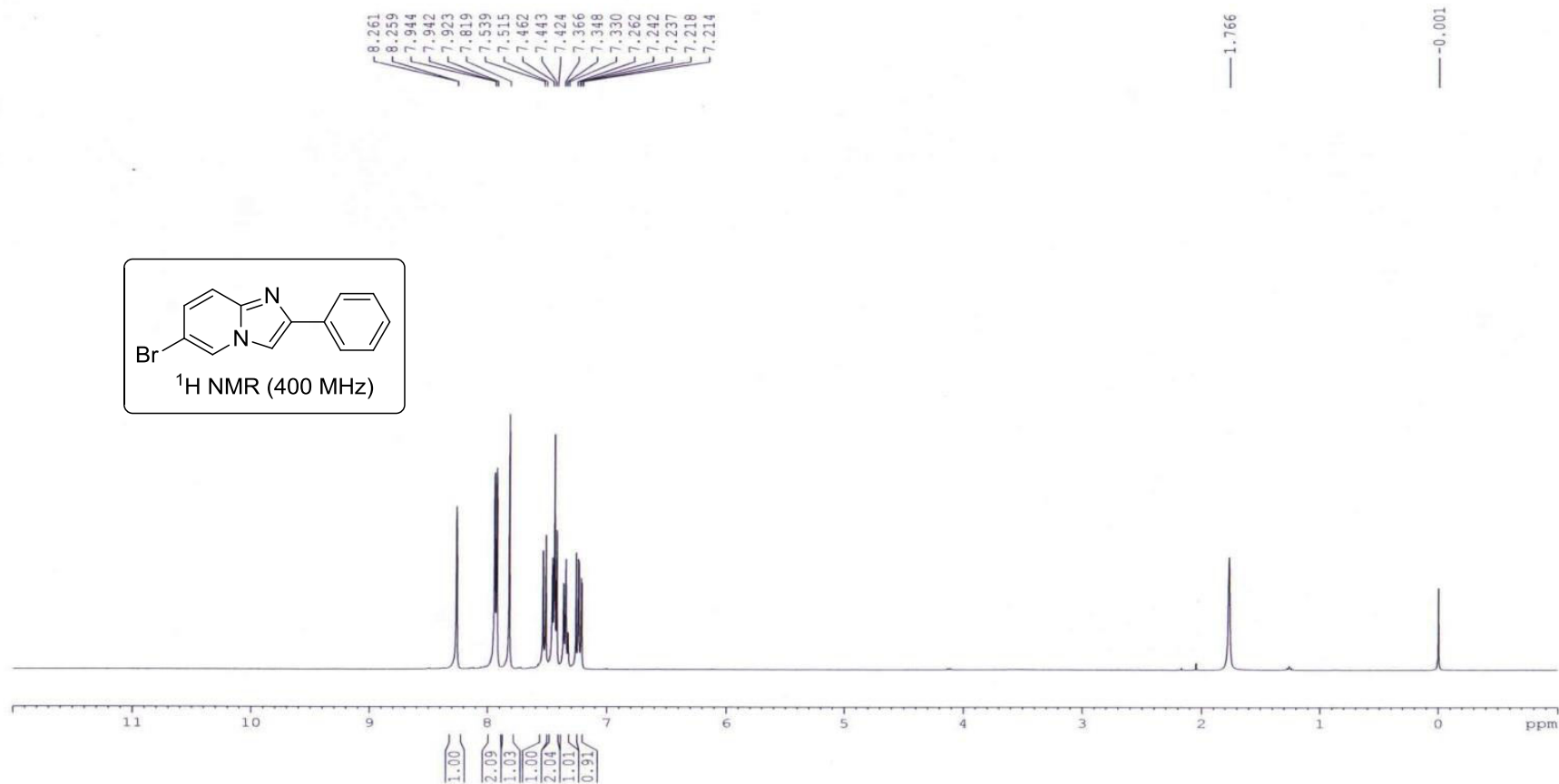
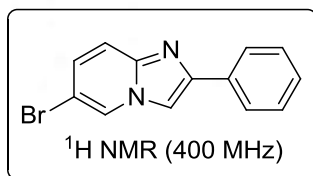
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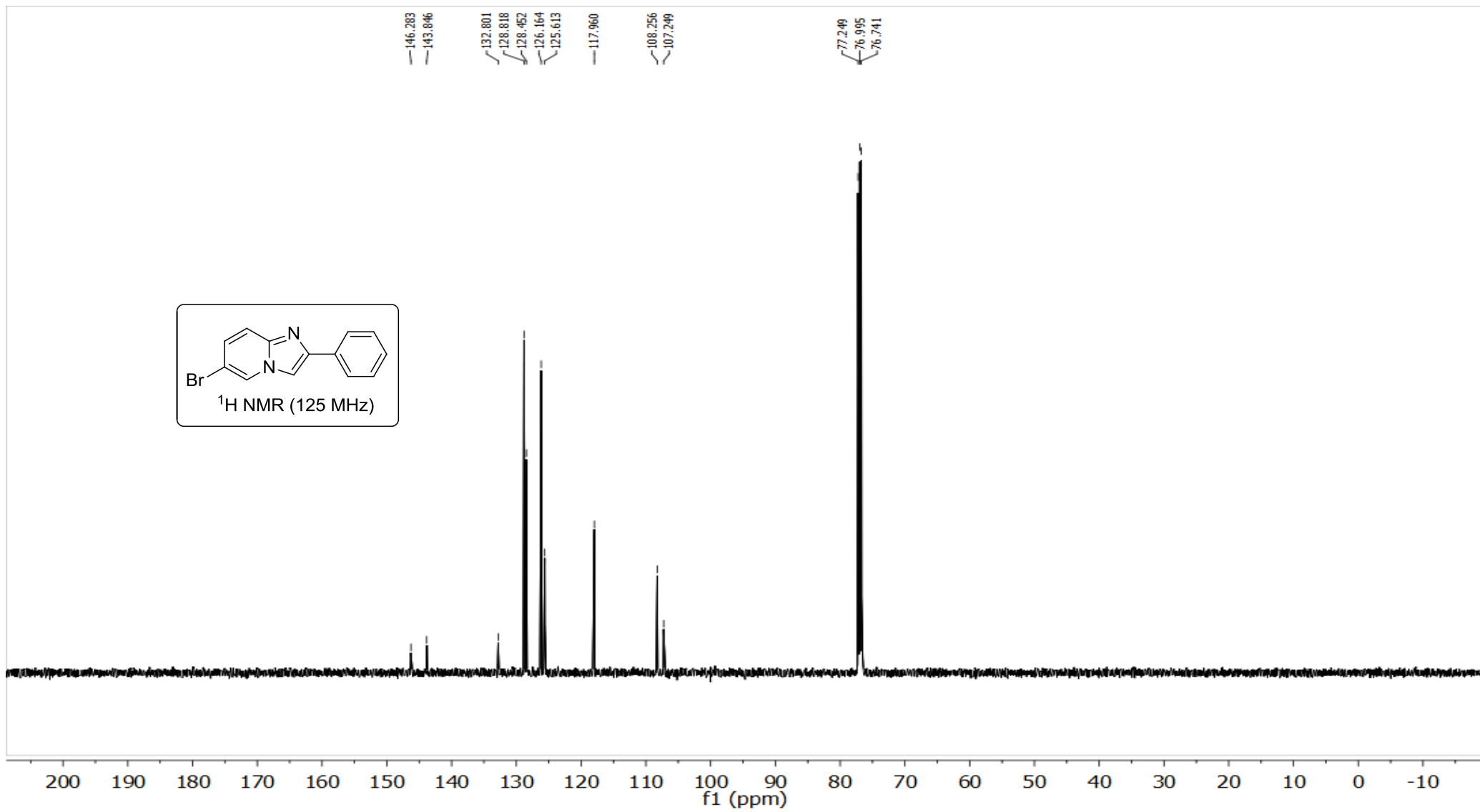
7-methyl-2-(p-tolyl)imidazo[1,2-a]pyridine(3s) :



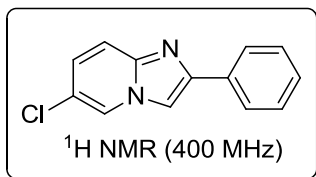
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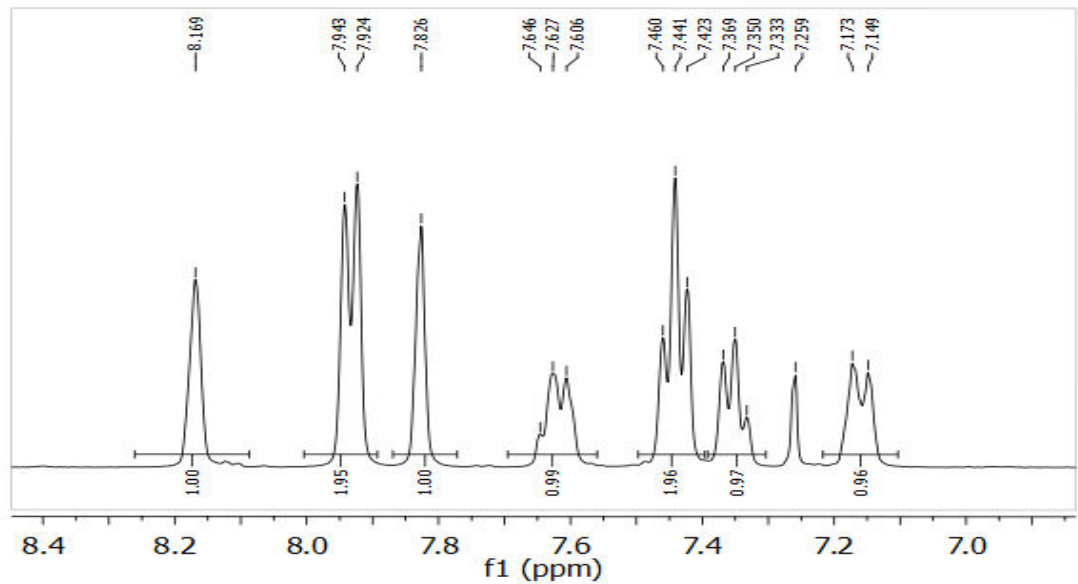
6-bromo-2-phenylimidazo[1,2-a]pyridine(3t) :



6-chloro-2-phenylimidazo[1,2-a]pyridine (3u) :



8.169
7.943
7.924
7.826
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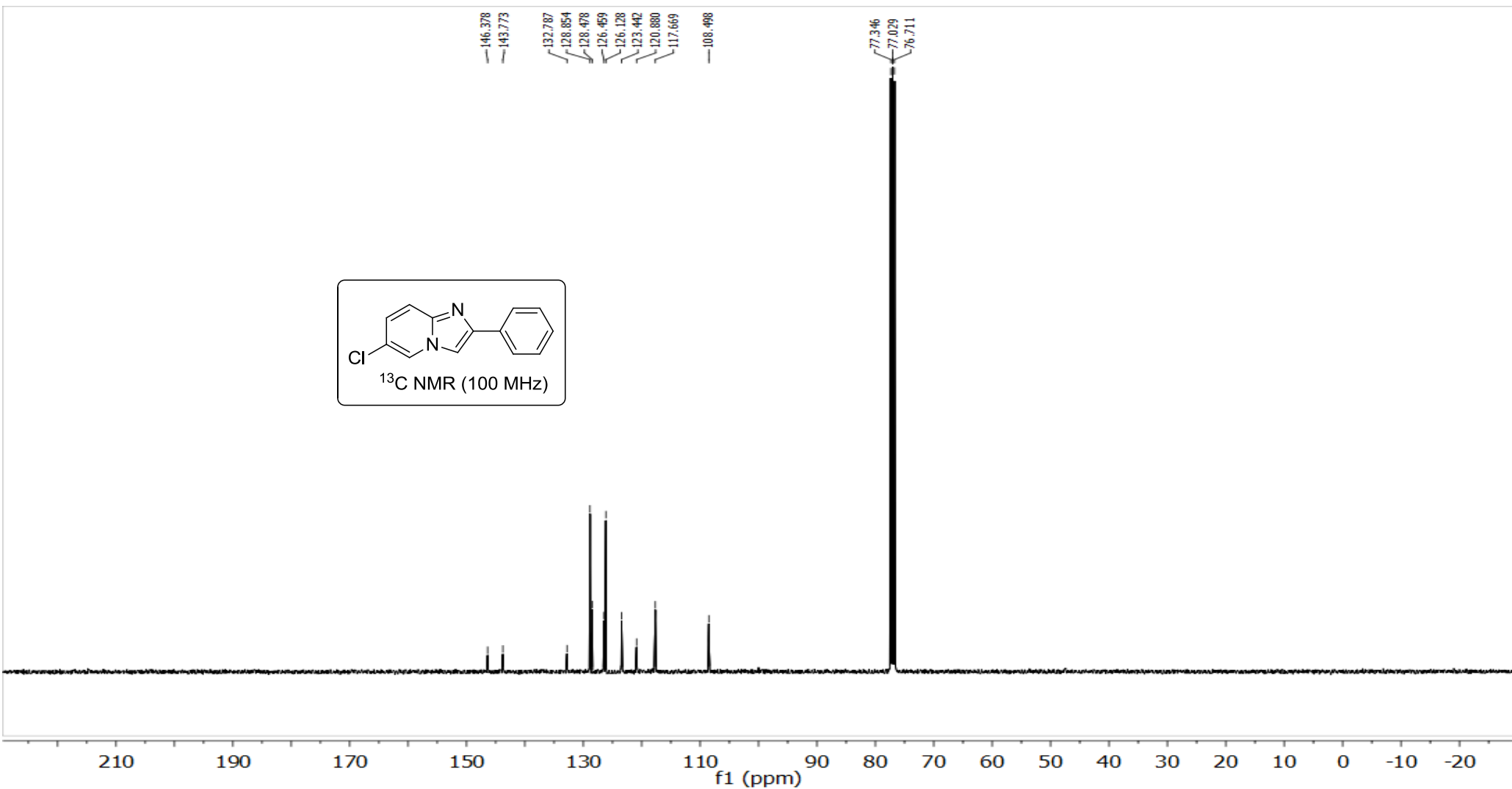
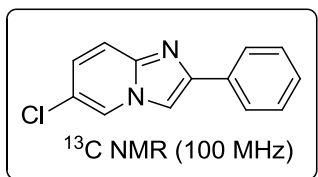


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1.00
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0.97
0.96

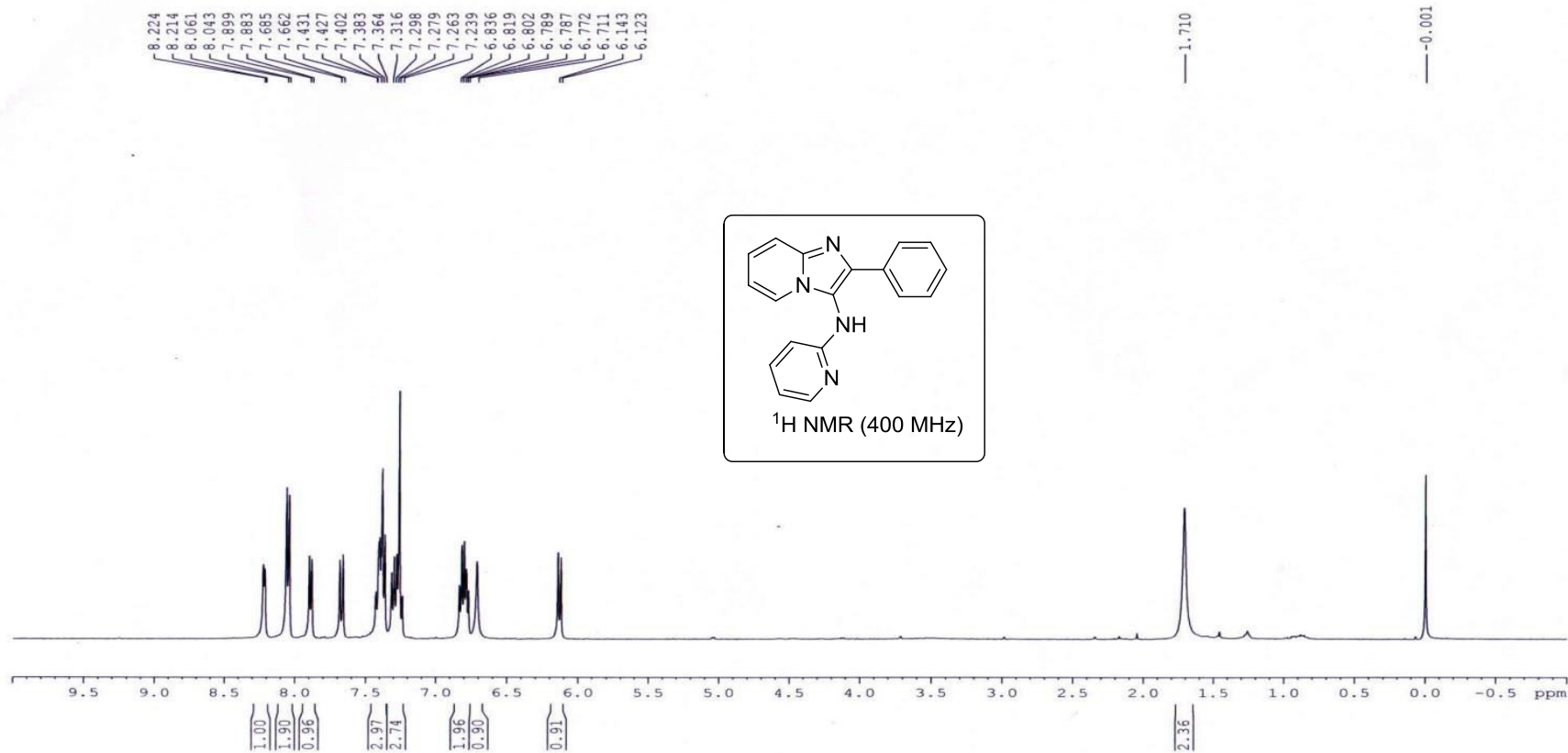
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f1 (ppm)

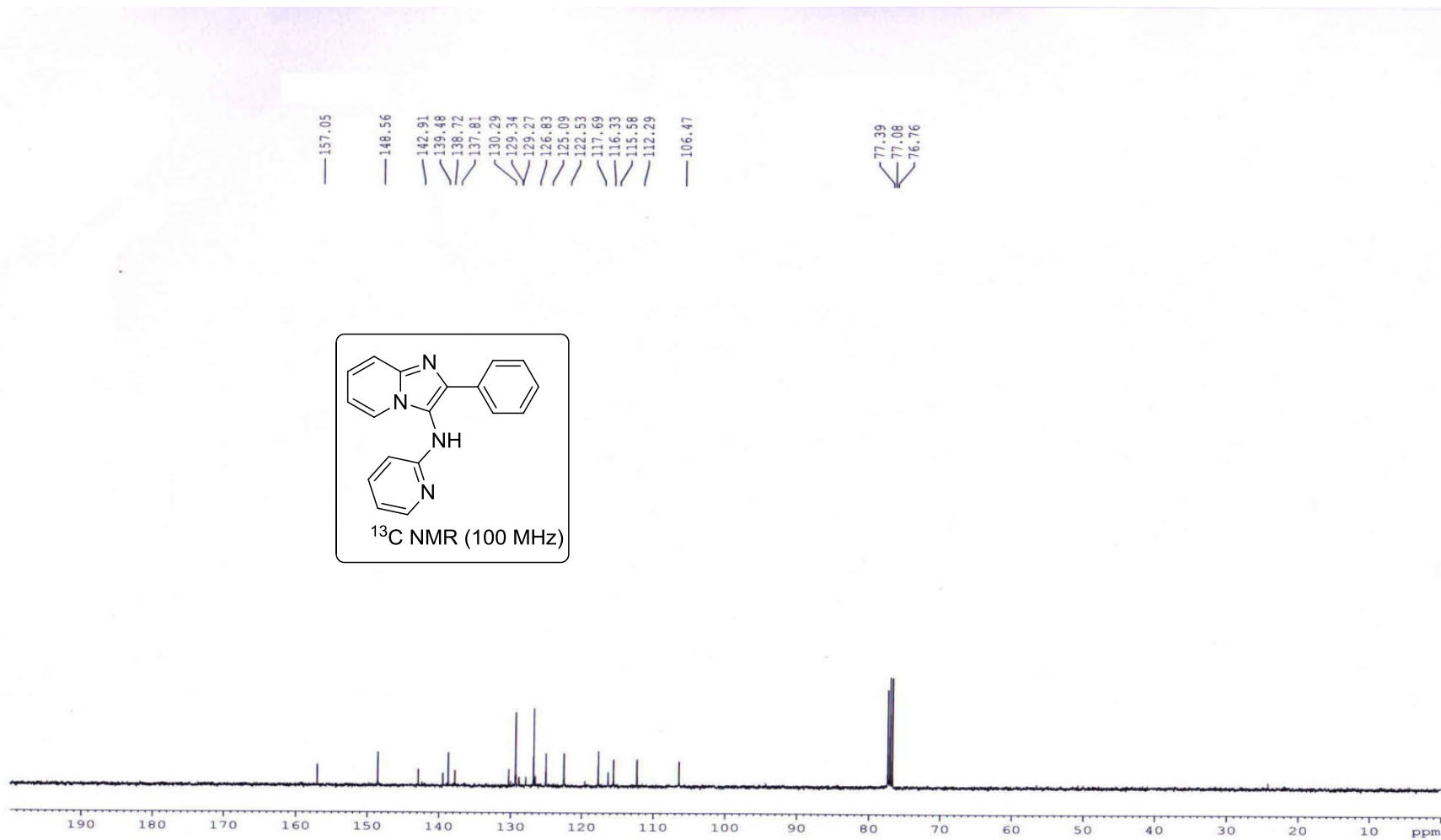
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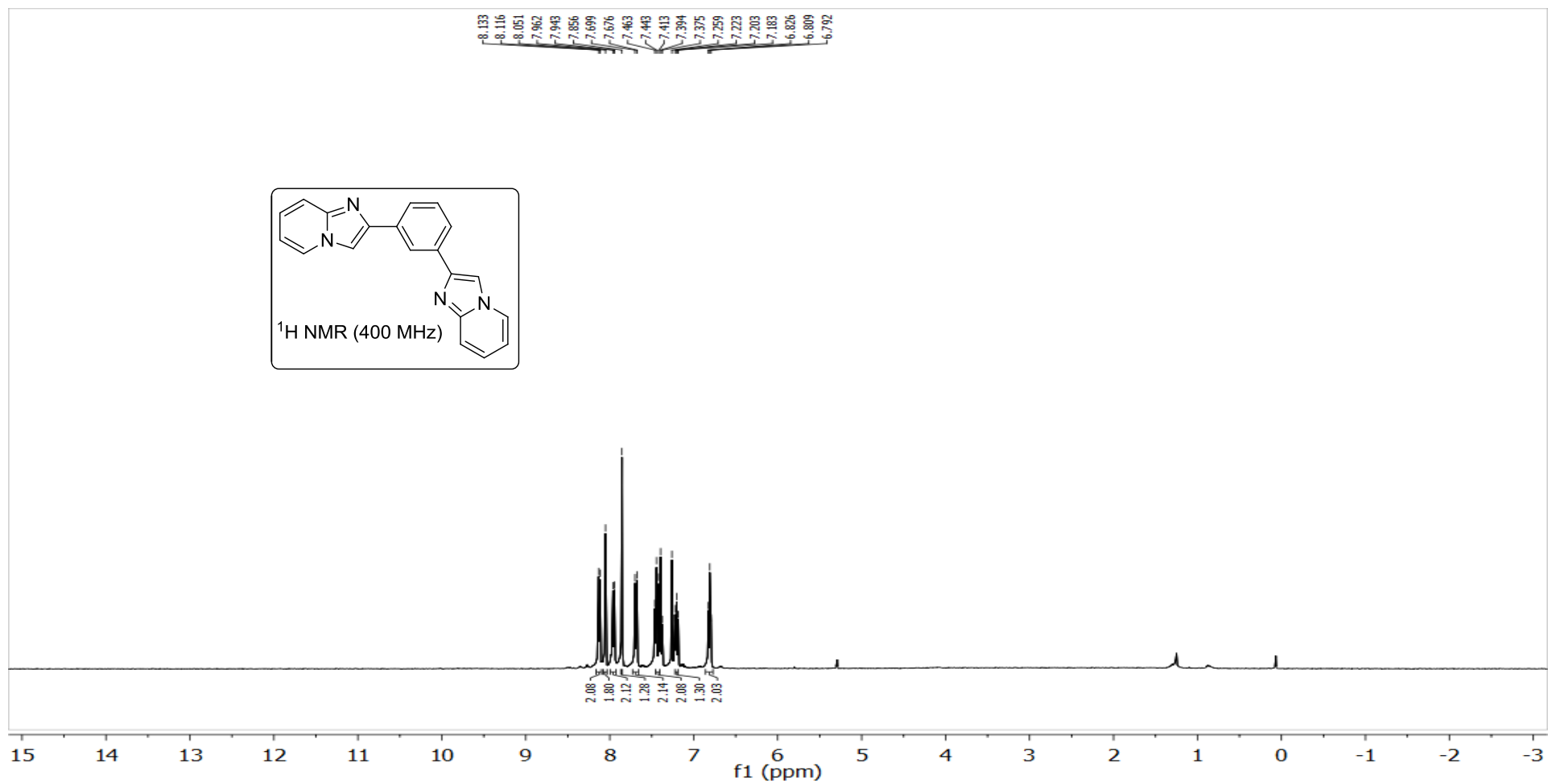
2-phenyl-N-(pyridin-2-yl)imidazo[1,2-a]pyridin-3-amine (4a) :



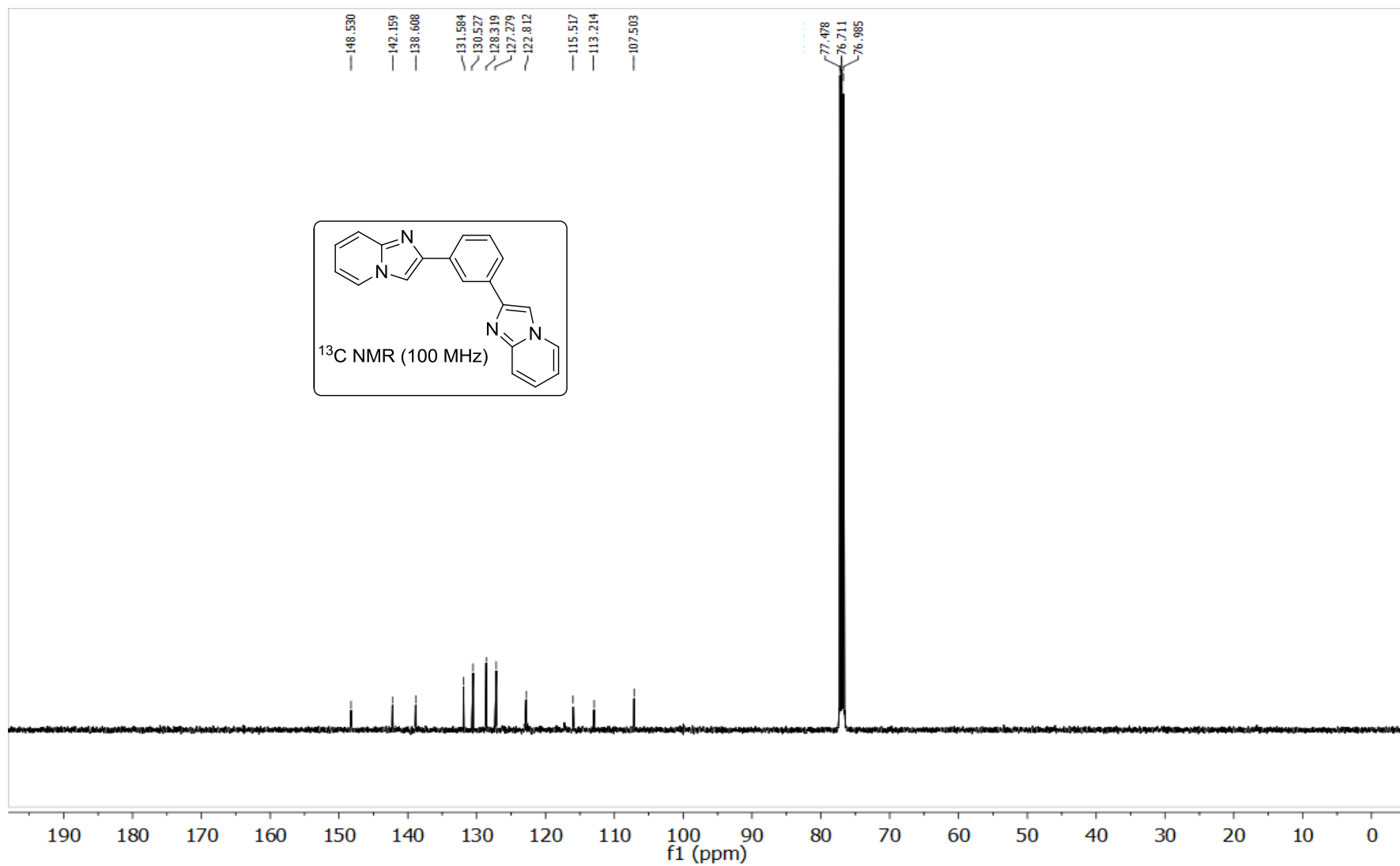
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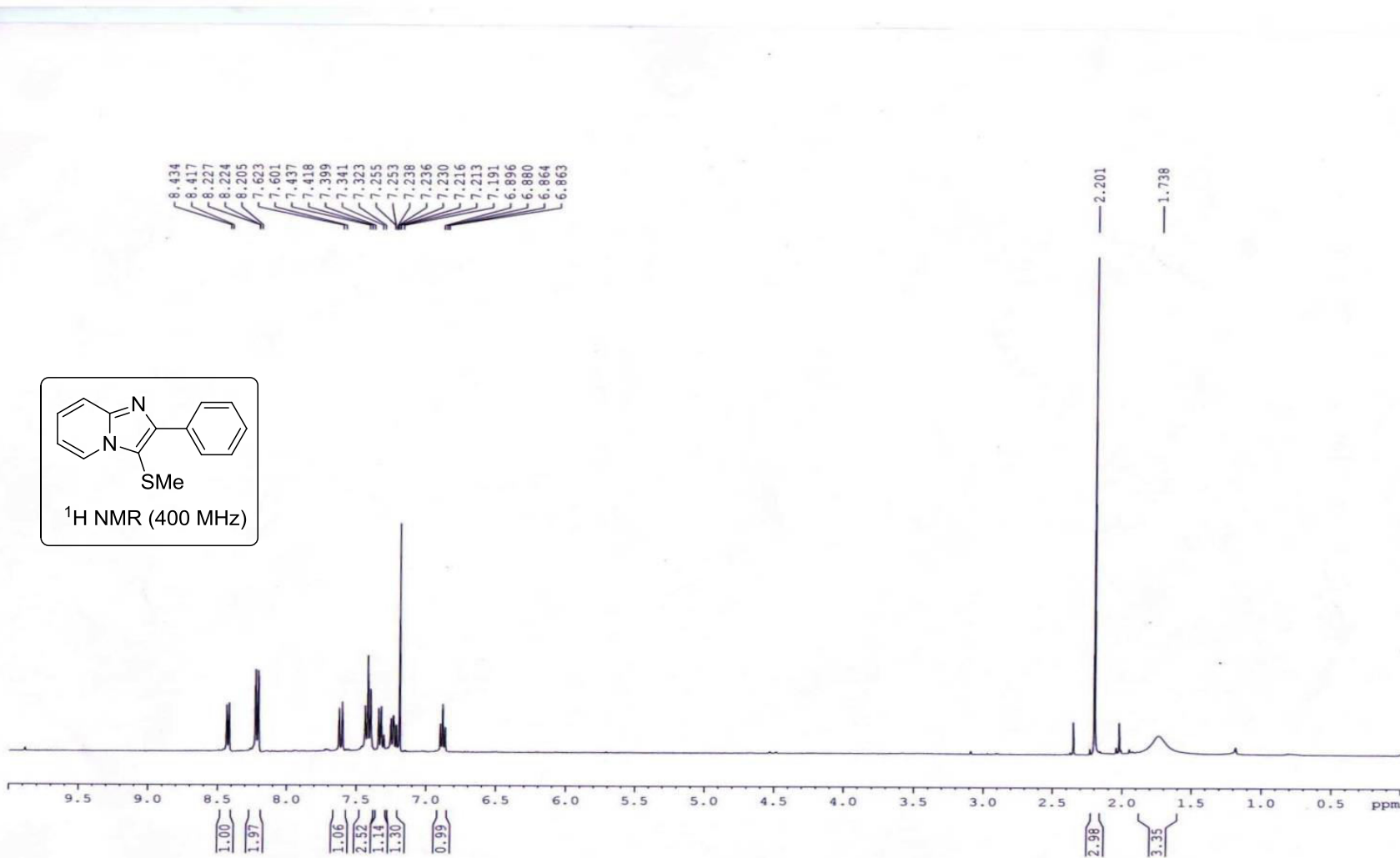
3-(3-(imidazo[1,2-a]pyridin-2-yl)phenyl)imidazo[1,2-a]pyridine(6) :



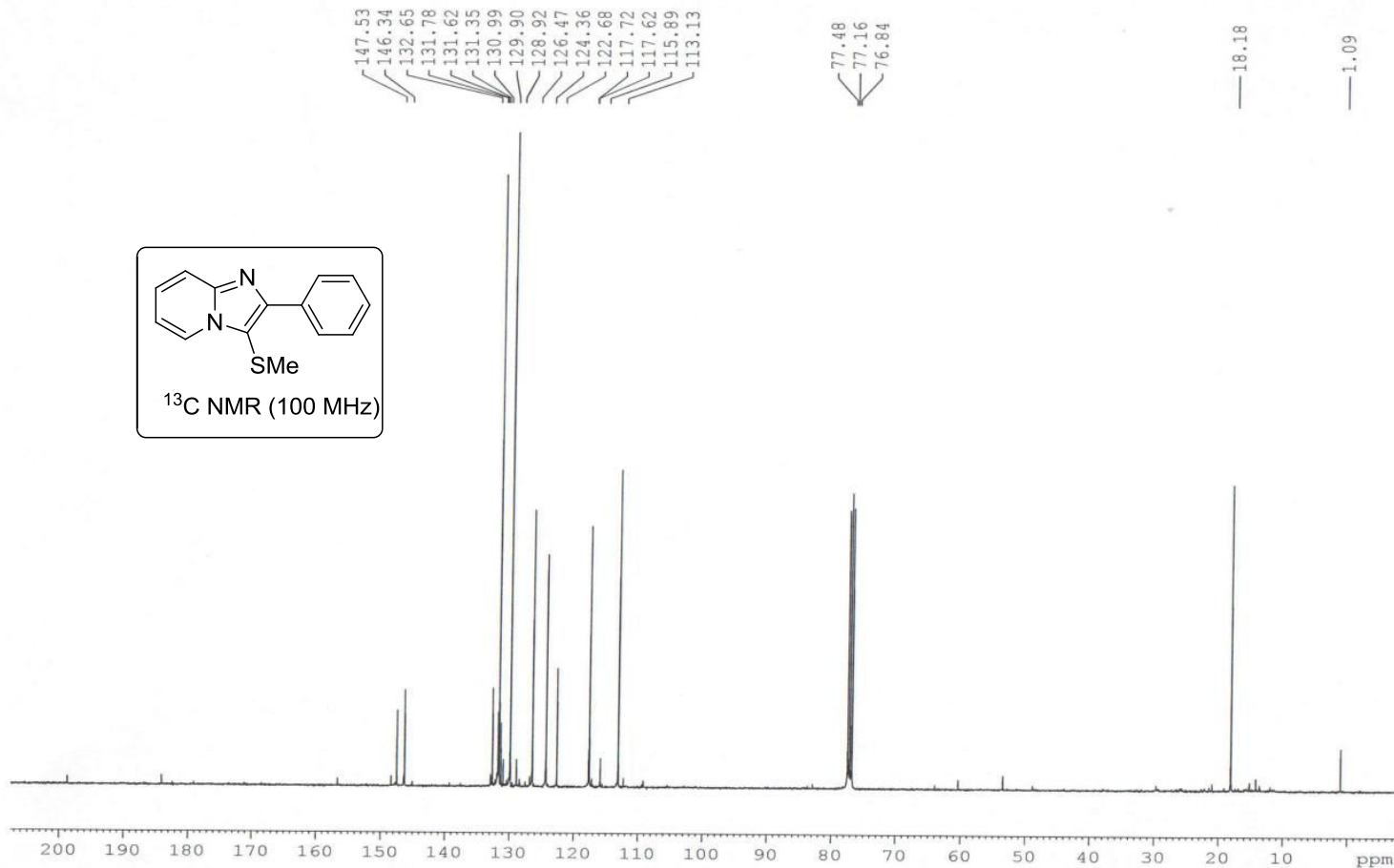
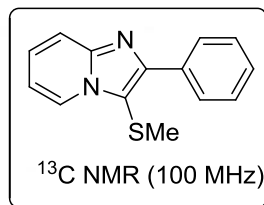
3-(3-(imidazo[1,2-*a*]pyridin-2-yl)phenyl)imidazo[1,2-*a*]pyridine(6) :



3-(methylthio)-2-phenylimidazo[1,2-a]pyridine(7a) :



3-(methylthio)-2-phenylimidazo[1,2-a]pyridine(7a) :



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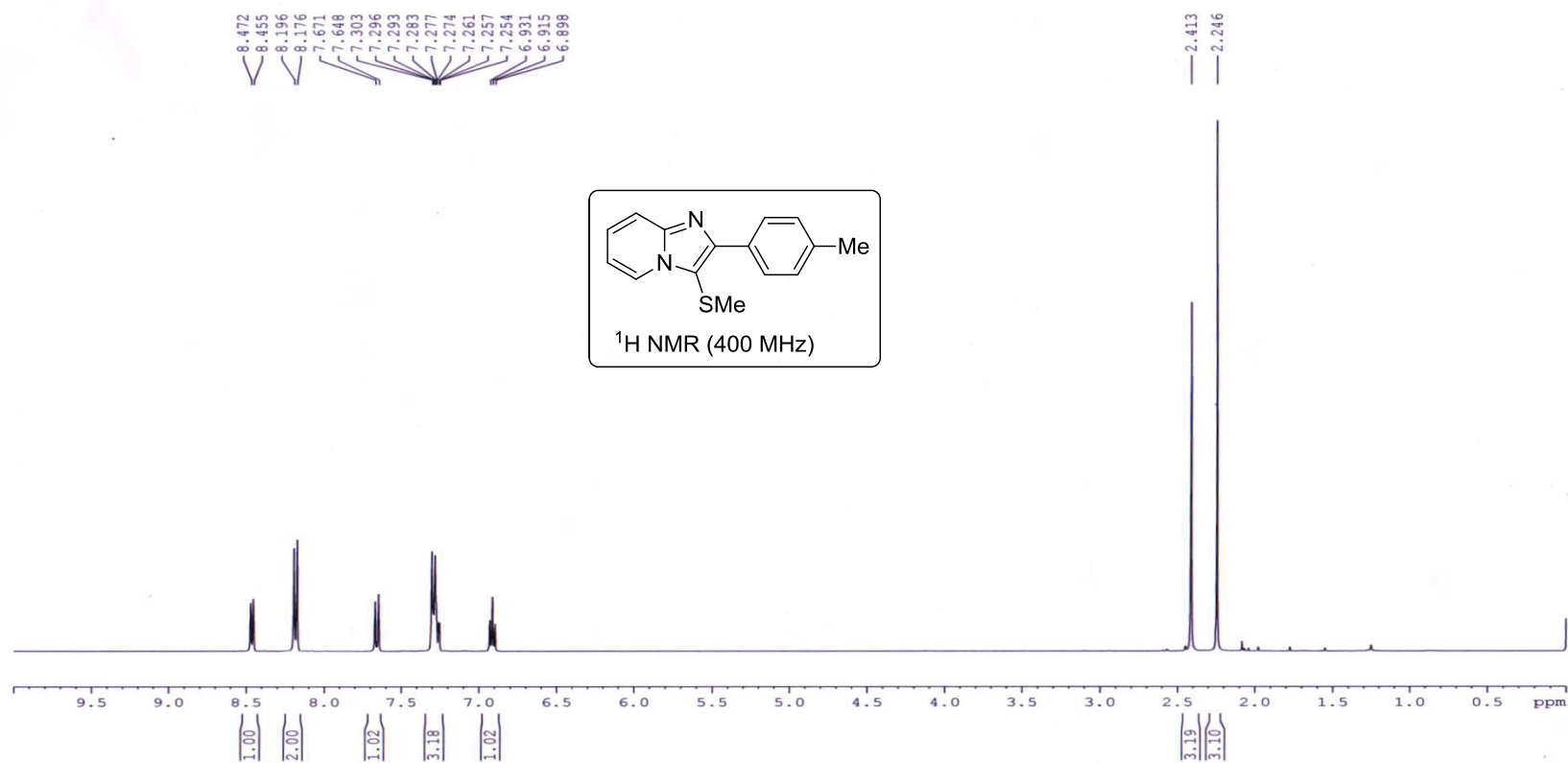
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 AQ 0.6815744 sec
 RG 203
 DW 20.800 usec
 DE 6.50 usec
 TE 0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

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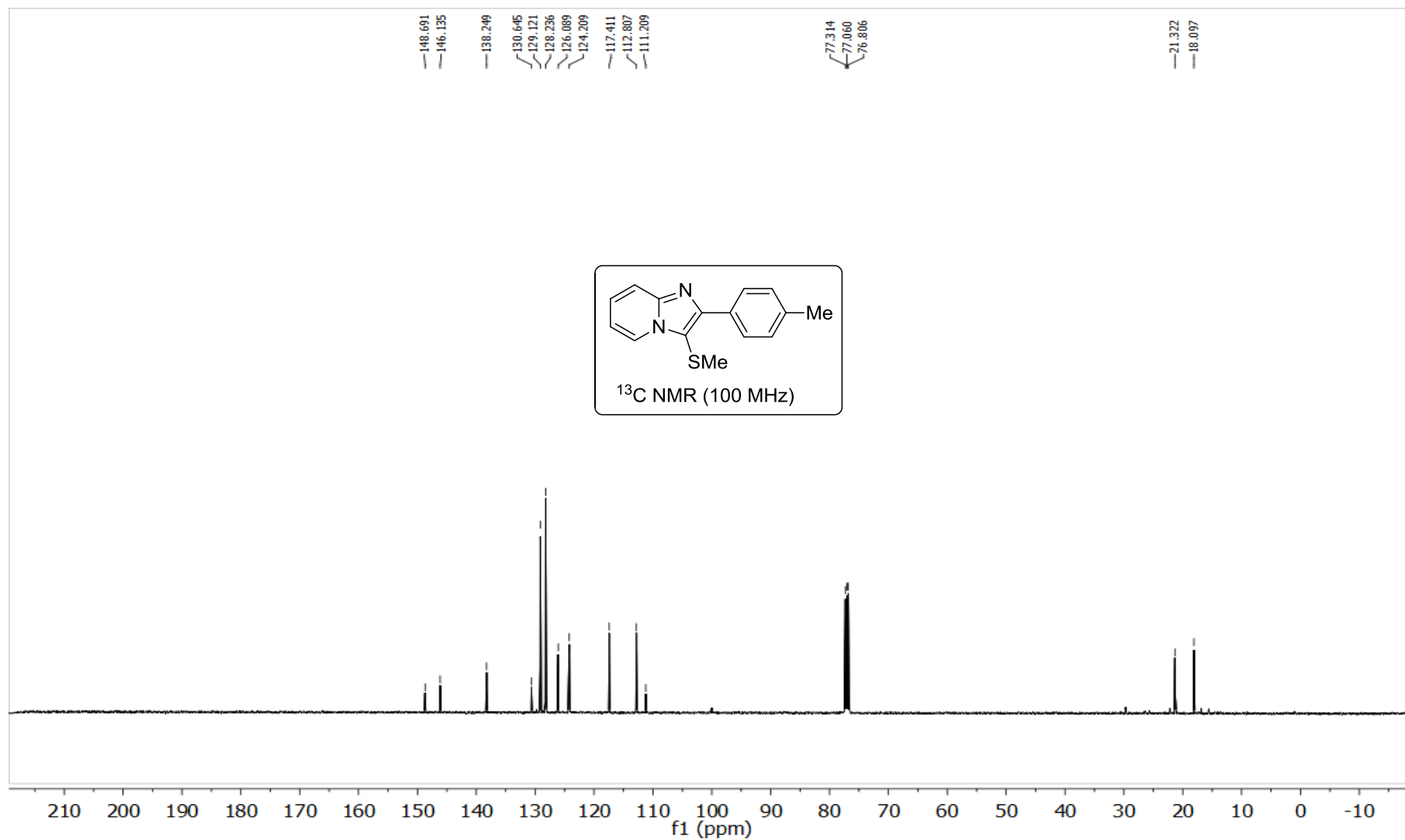
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 PLW12 0.36736000 W
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F2 - Processing parameters
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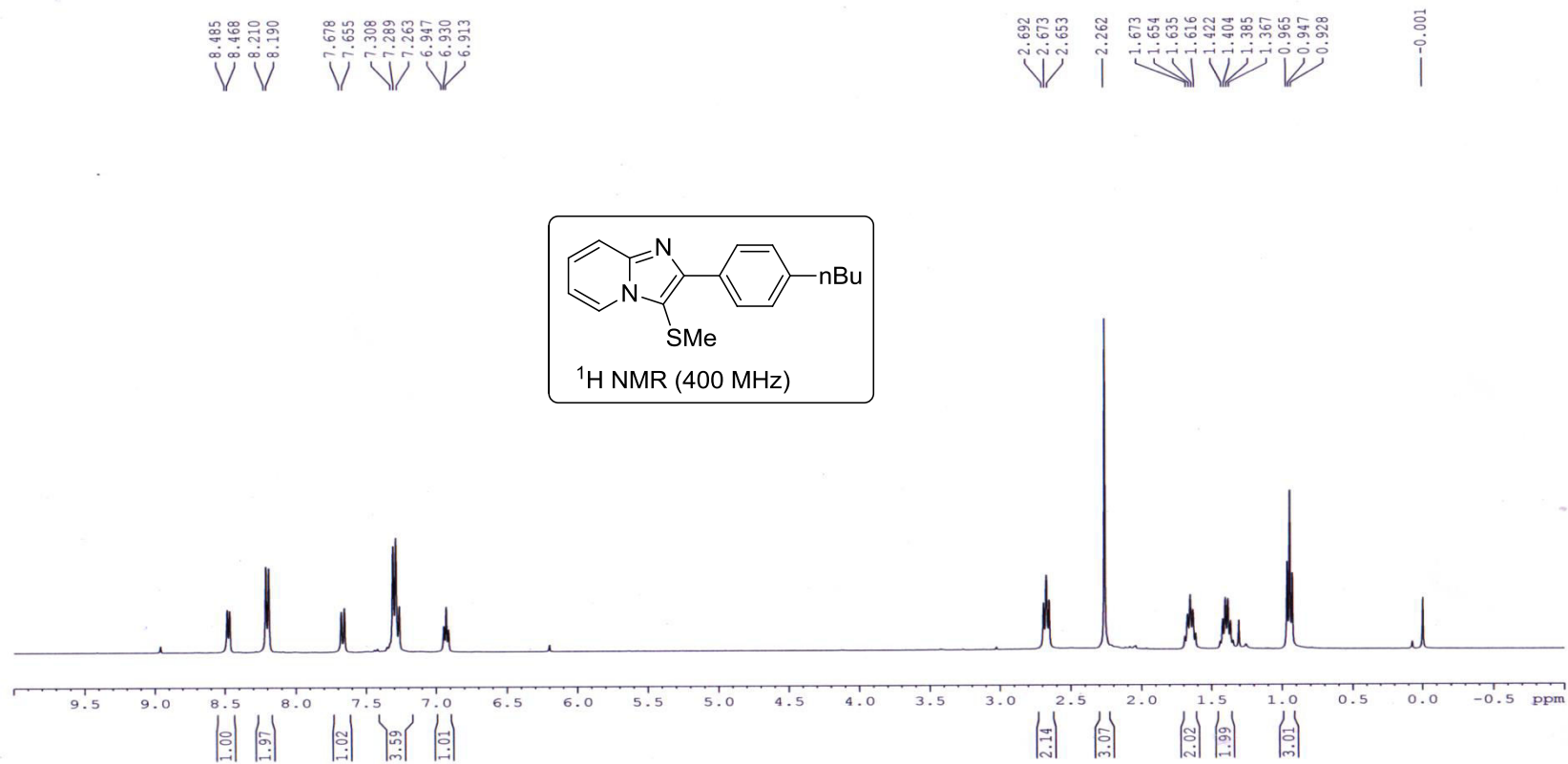
3-(methylthio)-2-(p-tolyl)imidazo[1,2-a]pyridine(7b) :



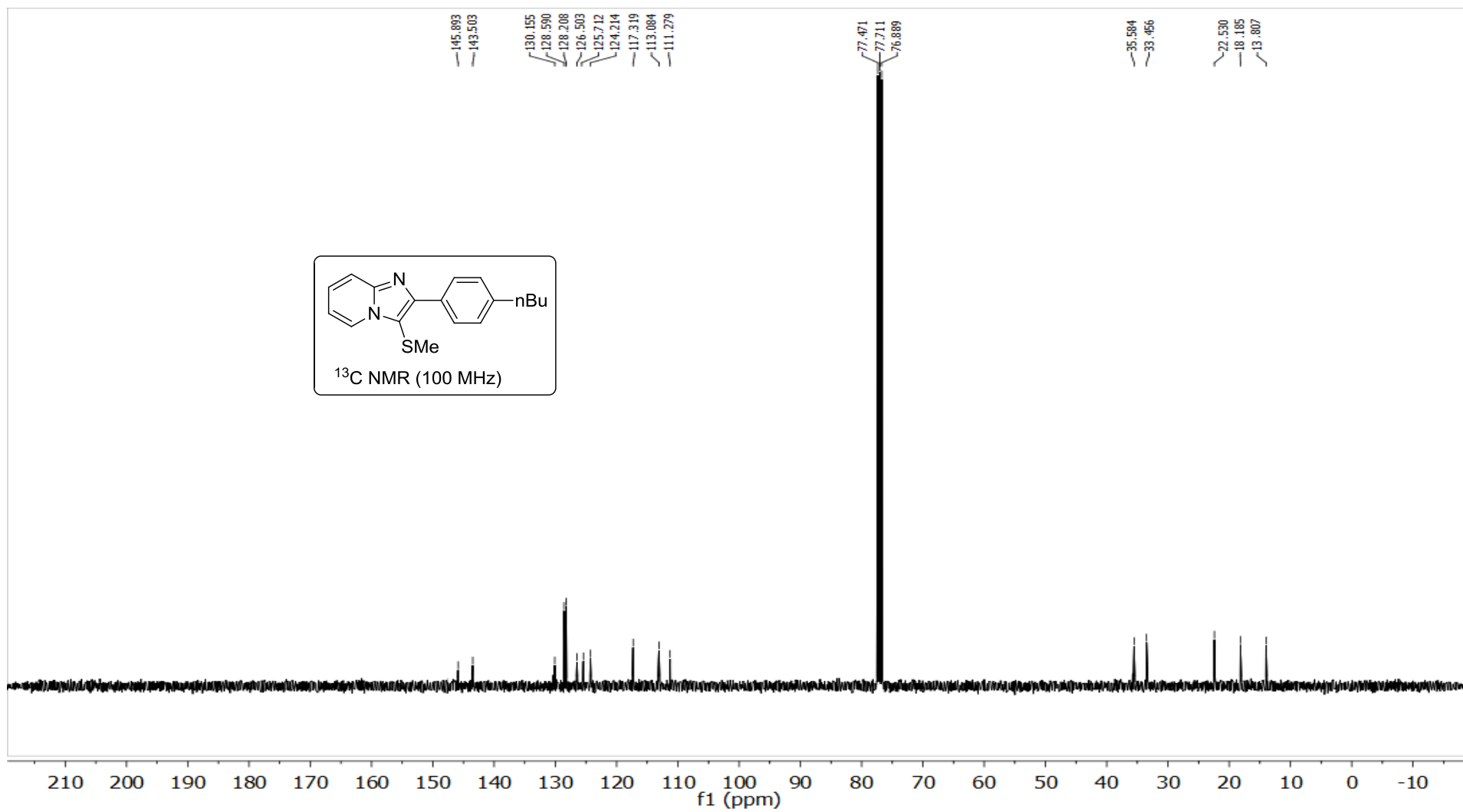
3-(methylthio)-2-(p-tolyl)imidazo[1,2-a]pyridine(7b) :



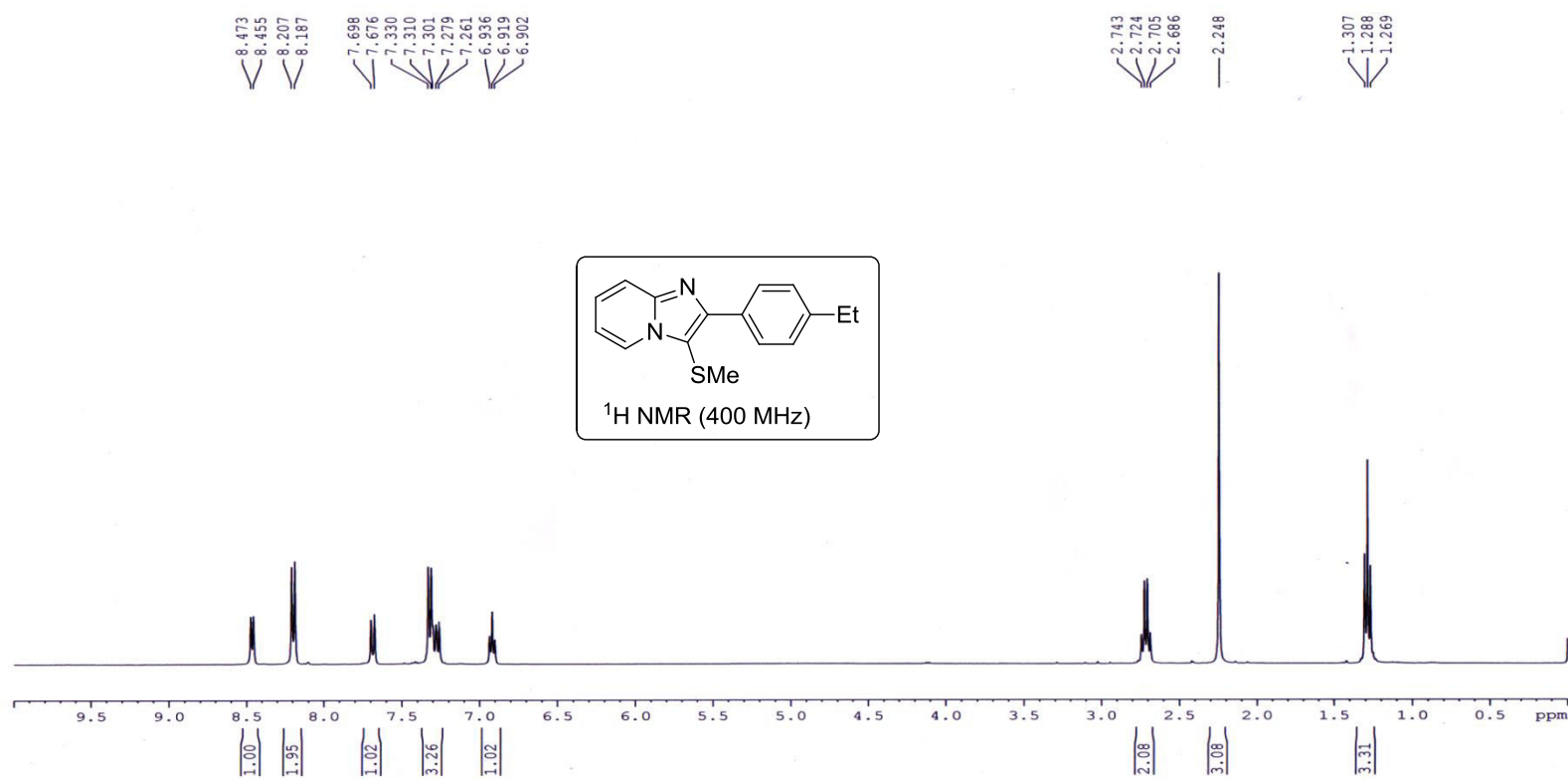
2-(4-butylphenyl)-3-(methylthio)imidazo[1,2-a]pyridine (7c) :



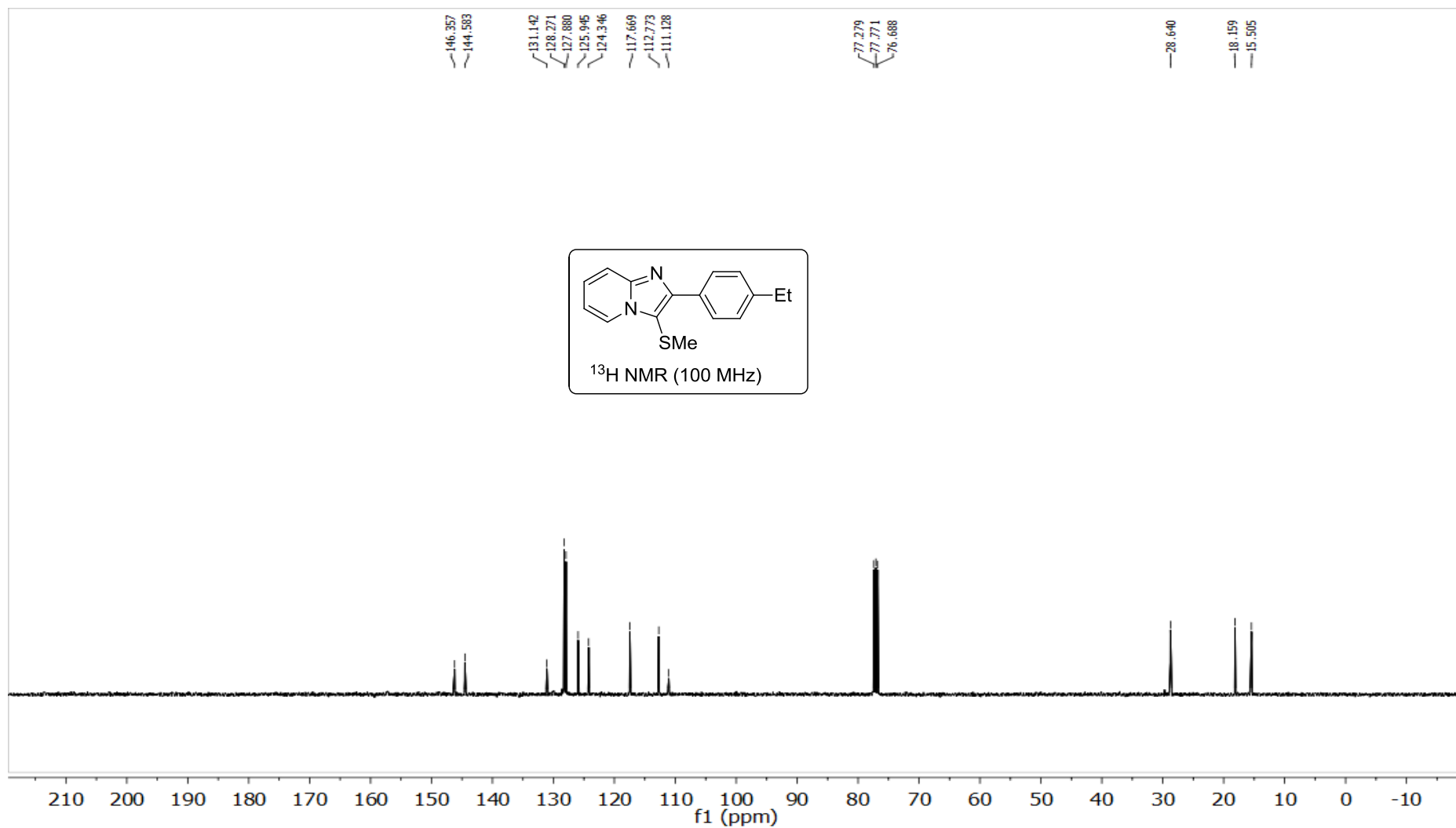
2-(4-butylphenyl)-3-(methylthio)imidazo[1,2-a]pyridine (7c) :



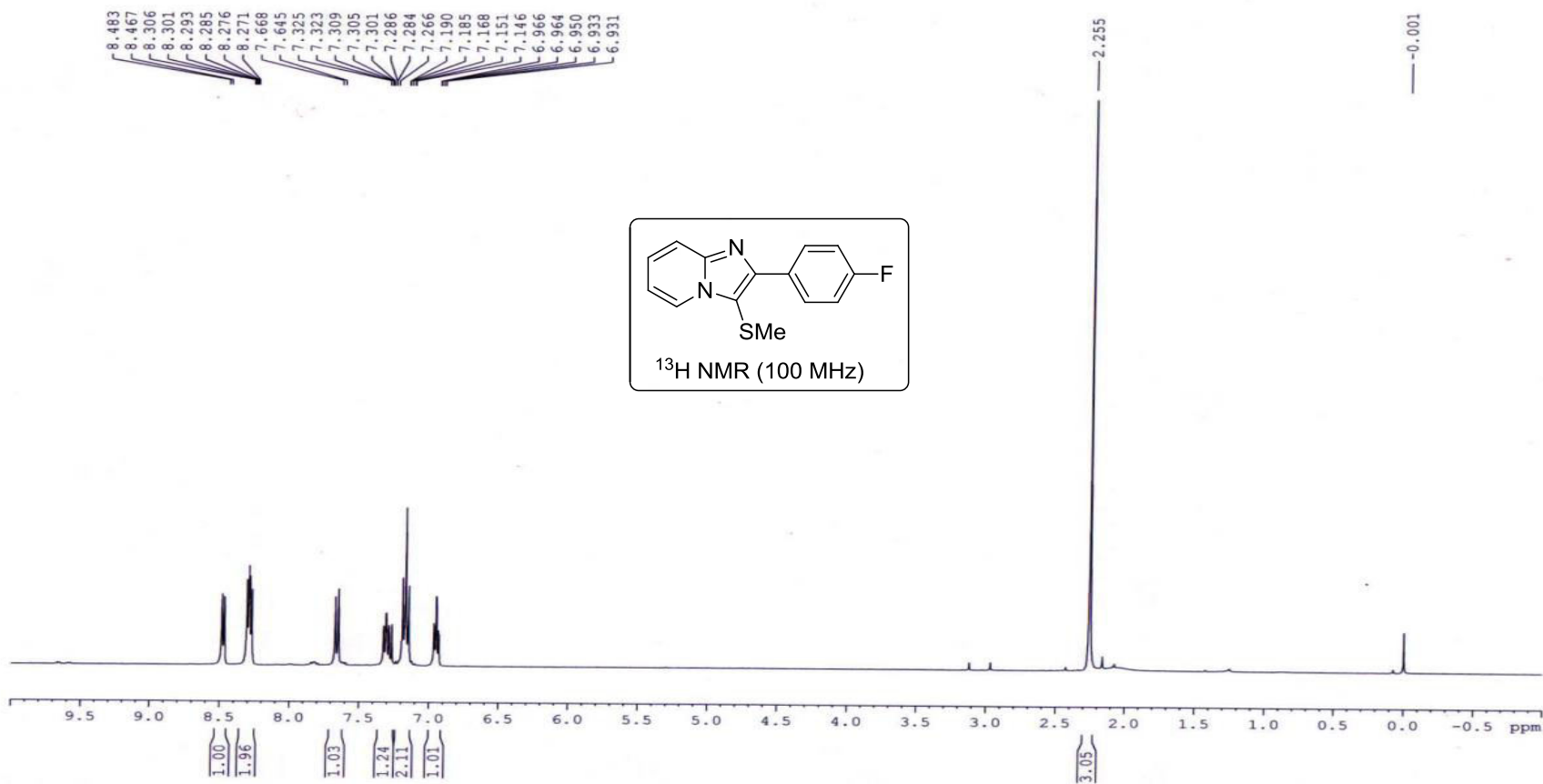
2-(4-ethylphenyl)-3-(methylthio)imidazo[1,2-a]pyridine(7d) :



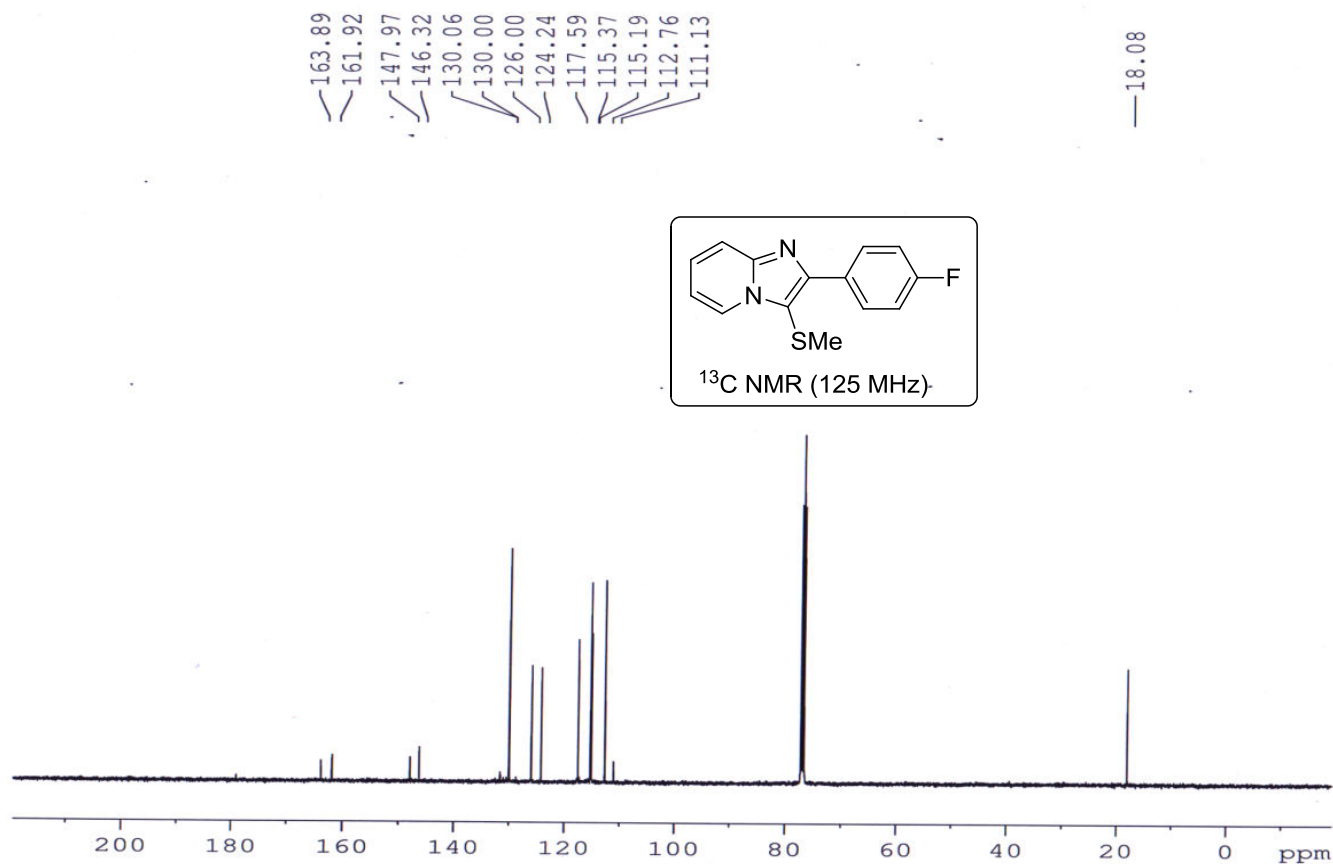
2-(4-ethylphenyl)-3-(methylthio)imidazo[1,2-a]pyridine(7d) :



2-(4-fluorophenyl)-3-(methylthio)imidazo[1,2-a]pyridine(7e) :



2-(4-fluorophenyl)-3-(methylthio)imidazo[1,2-a]pyridine(7e) :



```

NAME MRB_101 13C
EXPNO 1
PROCNO 1
Date_ 20180824
Time_ 13.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 1200
DS 2
SWH 30030.029 Hz
FIDRES 0.916444 Hz
AQ 0.5456539 se
RG 8192
DW 16.650 us
DE 6.50 us
TE 305.1 K
D1 2.00000000 se
D11 0.03000000 se
TDO 1
  
```

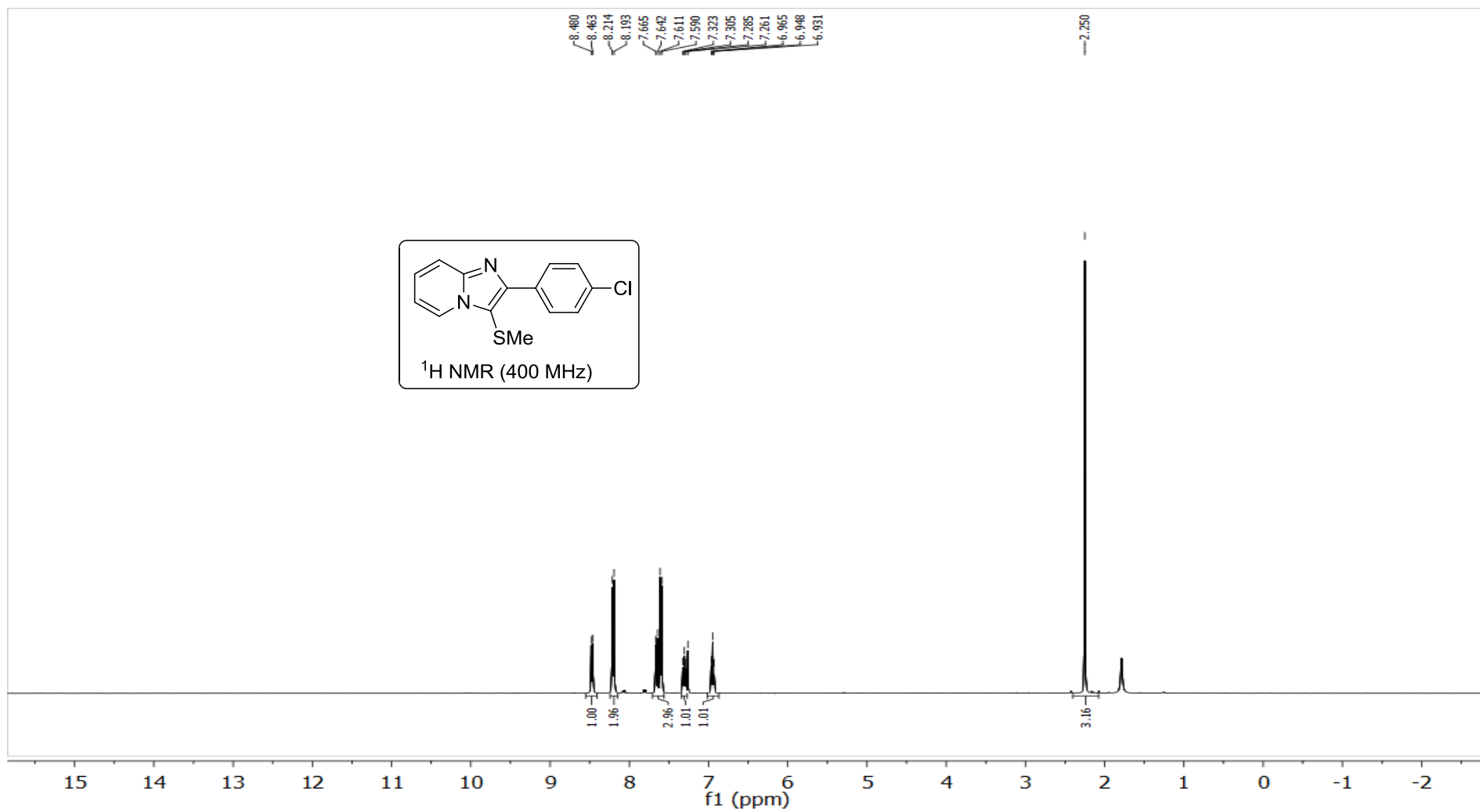
```

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 us
PL1 0.00 dB
SFO1 125.7703643 MH
  
```

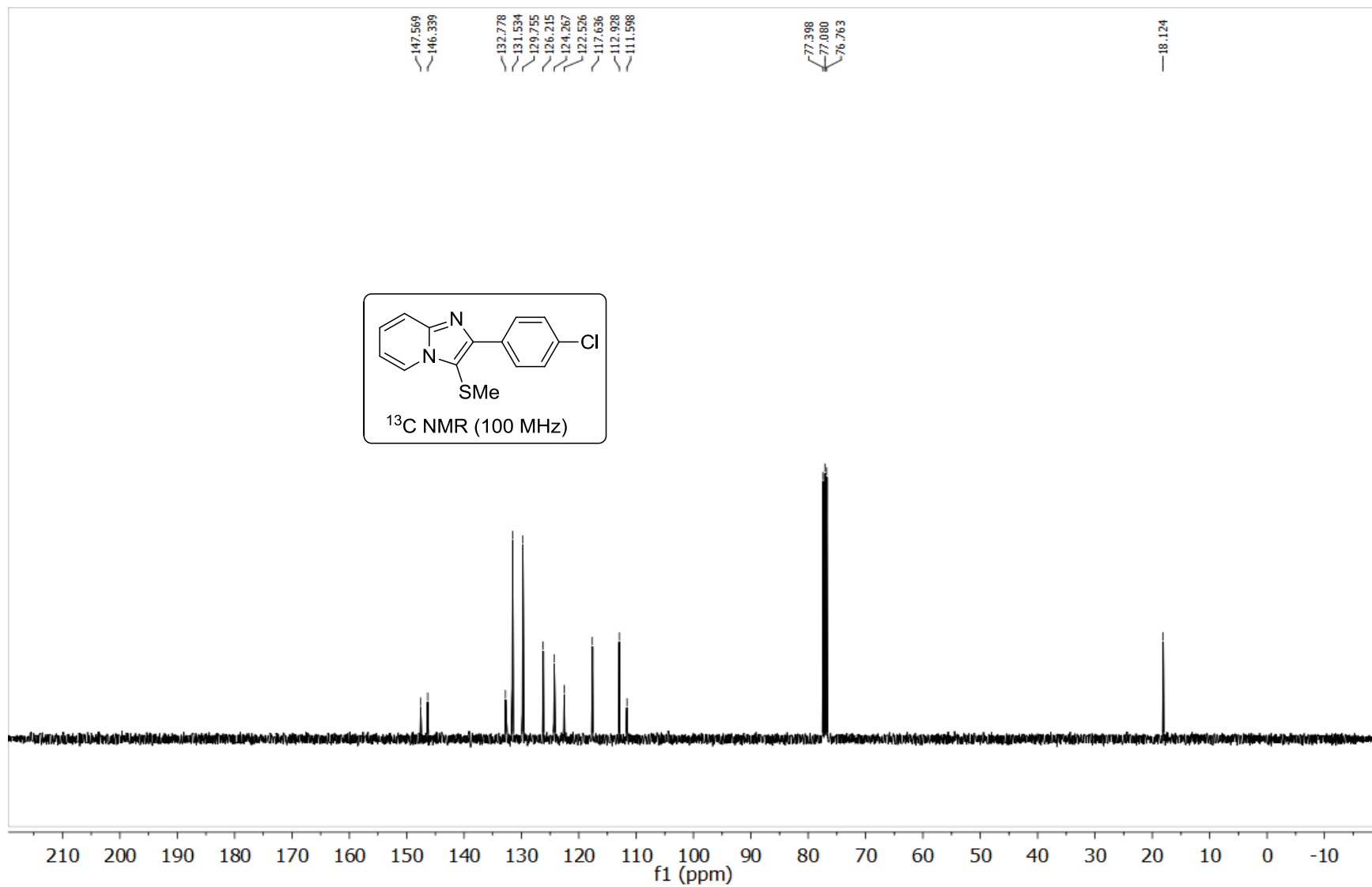
```

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PL2 0.00 dB
PL12 15.30 dB
PL13 19.00 dB
SFO2 500.1320005 MH
SI 16384
SF 125.7577890 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
  
```

2-(4-chlorophenyl)-3-(methylthio)imidazo[1,2-a]pyridine (7f) :



2-(4-chlorophenyl)-3-(methylthio)imidazo[1,2-a]pyridine (7f) :

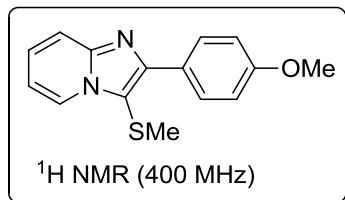


2-(4-methoxyphenyl)-3-(methylthio)imidazo[1,2-a]pyridine(7g) :

8.464
8.447
8.248
8.227
7.683
7.661
7.303
7.284
7.263
7.021
6.999
6.939
6.922
6.906

3.859

2.237



1.03

2.09

1.18

2.12

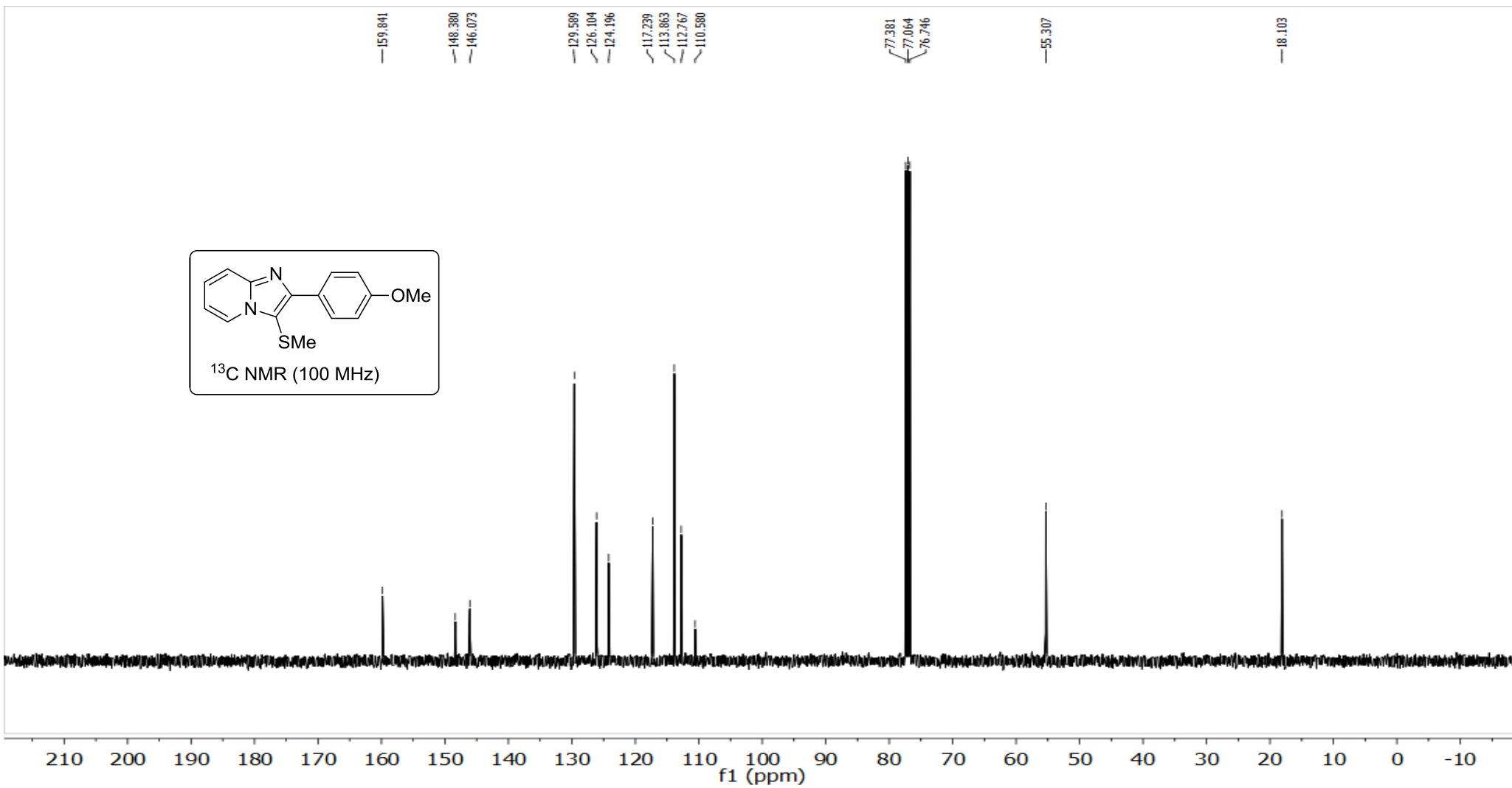
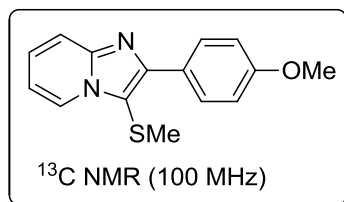
1.08

3.05

3.11

11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0 -2.0
f1 (ppm)

2-(4-methoxyphenyl)-3-(methylthio)imidazo[1,2-a]pyridine(7g) :



2-(4-(tert-butyl)phenyl)-3-(methylthio)imidazo[1,2-a]pyridine (7h):

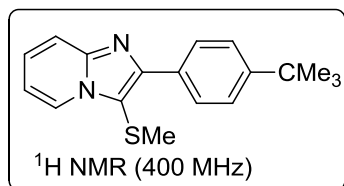
8.479
8.475
8.472
8.456
8.453
8.449
8.228
8.222
8.206
8.200
7.961
7.957
7.934
7.929
7.665
7.635
7.503
7.497
7.474
7.451
7.447
7.295
7.291
7.273
7.268
7.266
6.937
6.934
6.915
6.911
6.892
6.888

2.256

1.833

1.358

-0.017



1.00
1.30
1.31
1.26
3.03
1.18
1.01

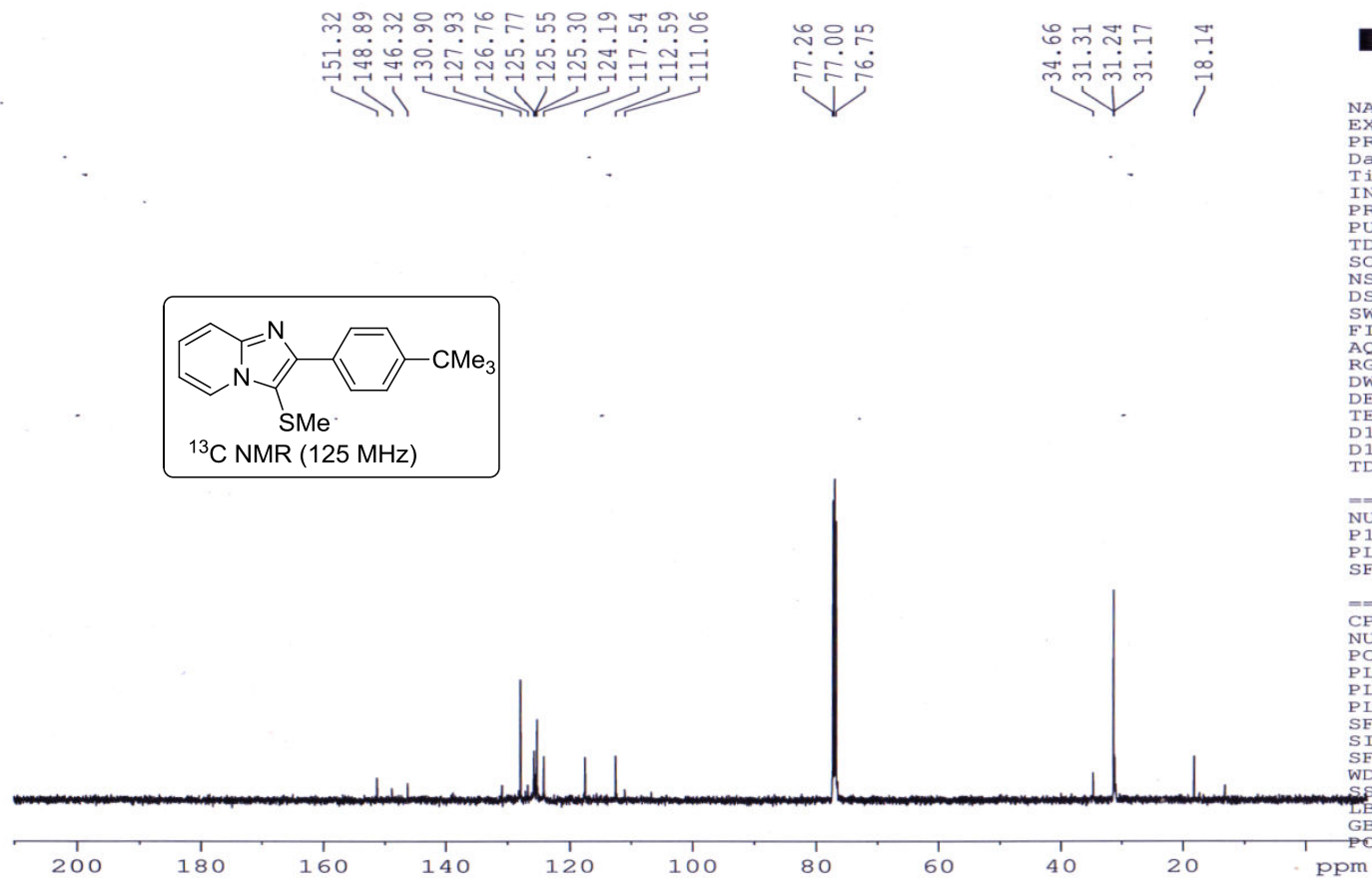
3.22

9.22

10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

2-(4-(tert-butyl)phenyl)-3-(methylthio)imidazo[1,2-a]pyridine (7h):



```

NAME          MKB-99
EXPNO         1
PROCNO        1
Date_         20180807
Time          11.35
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            512
DS            2
SWH           30030.029 Hz
FIDRES        0.916444 Hz
AQ            0.5456539 se
RG            8192
DW            16.650 us
DE            6.50 us
TE            304.5 K
D1            2.00000000 se
D11           0.03000000 se
TD0           1
    
```

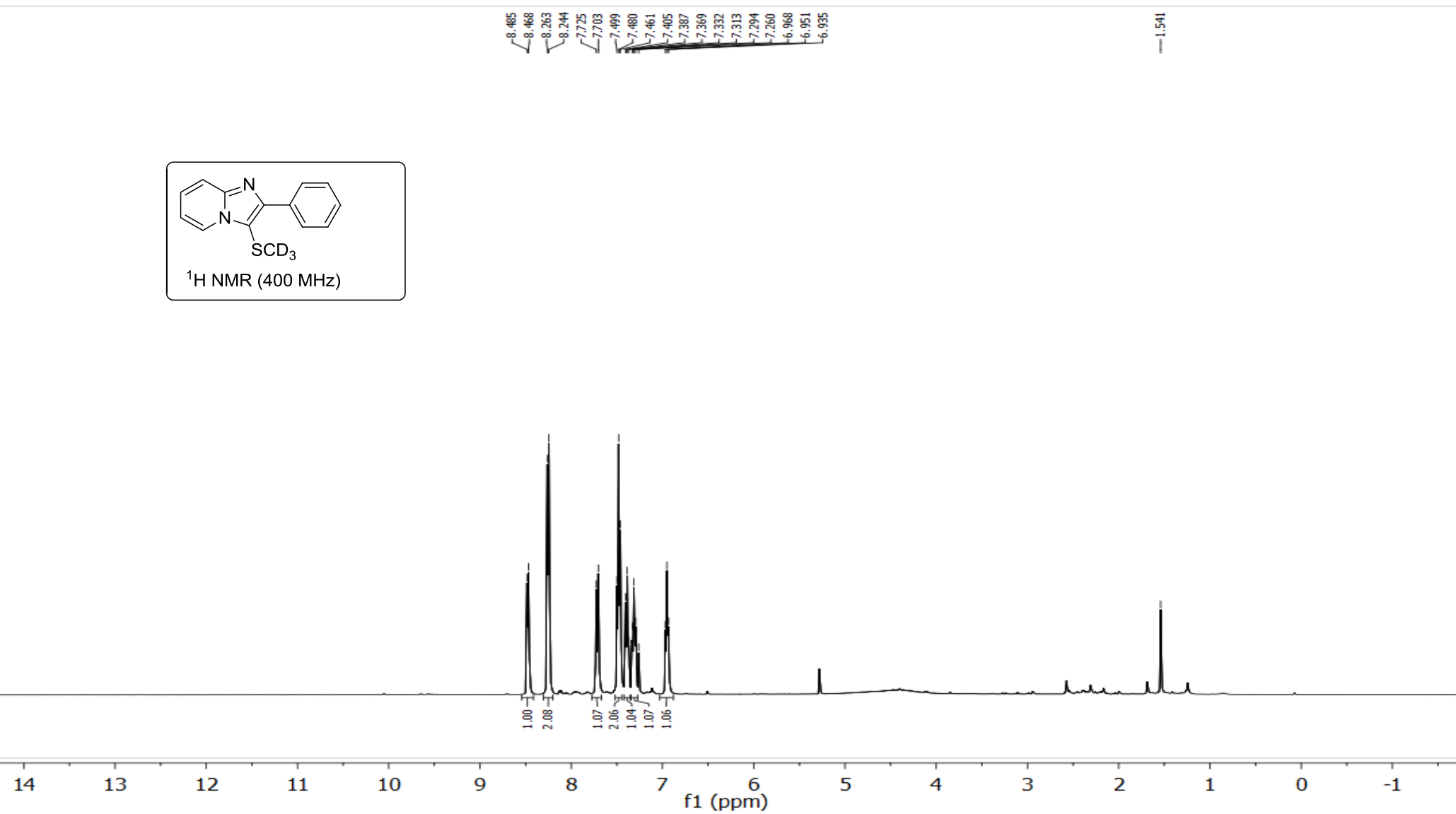
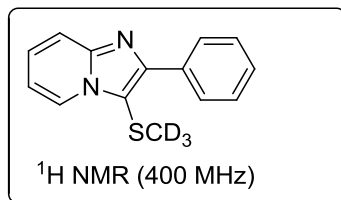
```

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 us
PL1           0.00 dB
SFO1          125.7703643 MH
    
```

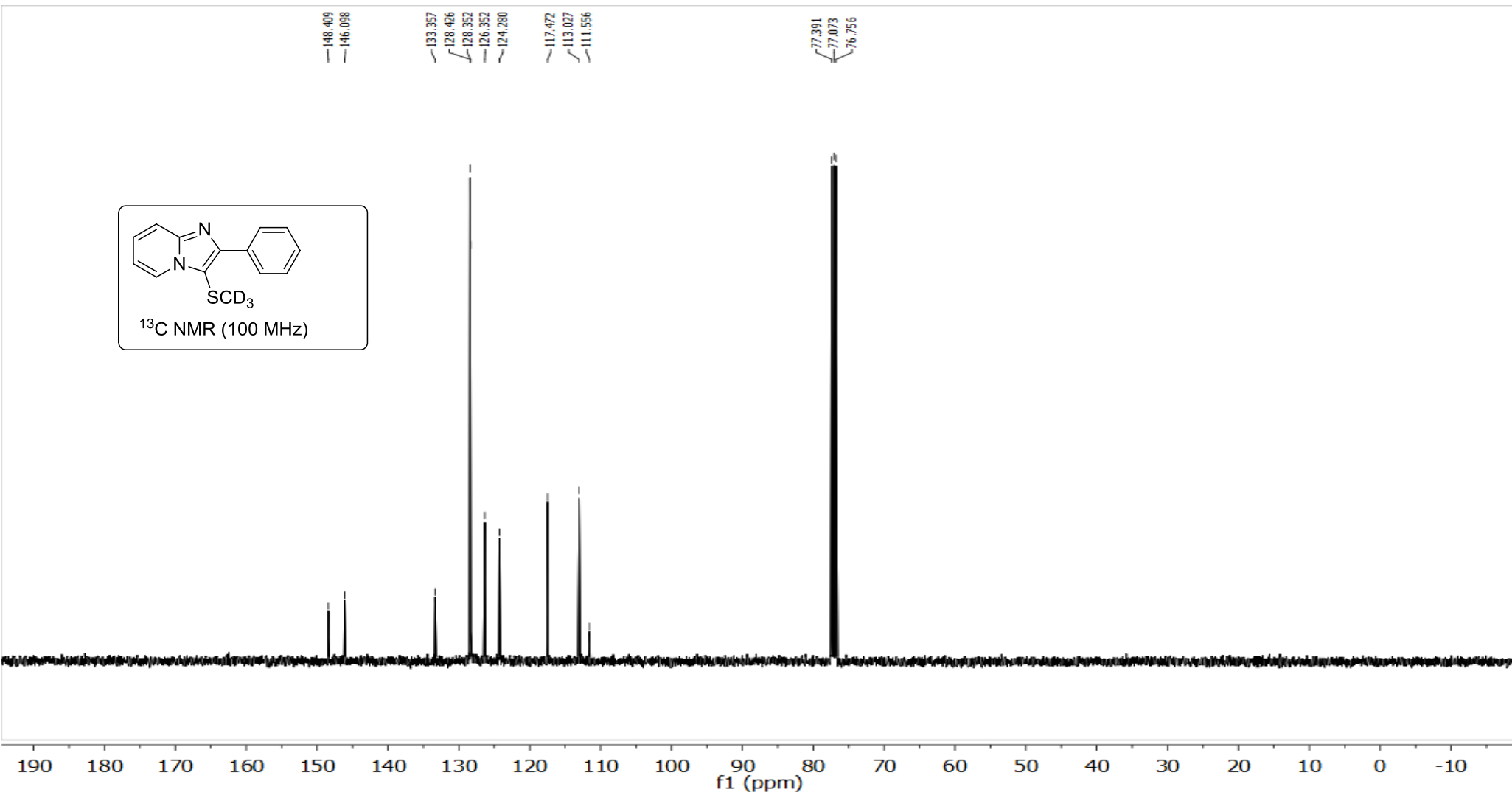
```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 us
PL2           0.00 dB
PL12          15.30 dB
PL13          19.00 dB
SFO2          500.1320005 MH
SI            16384
SF            125.7577890 MH
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

3-(d₃-methylthio)-2-phenylimidazo[1,2-a]pyridine (7i) :

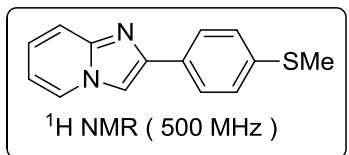


3-(d₃-methylthio)-2-phenylimidazo[1,2-a]pyridine (7i)



2-(4-(methylthio)phenyl)imidazo[1,2-a]pyridine (9):

8.133
8.120
7.905
7.901
7.891
7.888
7.843
7.690
7.672
7.335
7.332
7.322
7.318
7.285
7.228
7.226
7.214
7.212
7.210
7.208
7.196
7.194
6.828
6.827
6.815
6.813
6.801
6.800



1.11
2.14
1.09
1.07
1.99
1.02
1.00

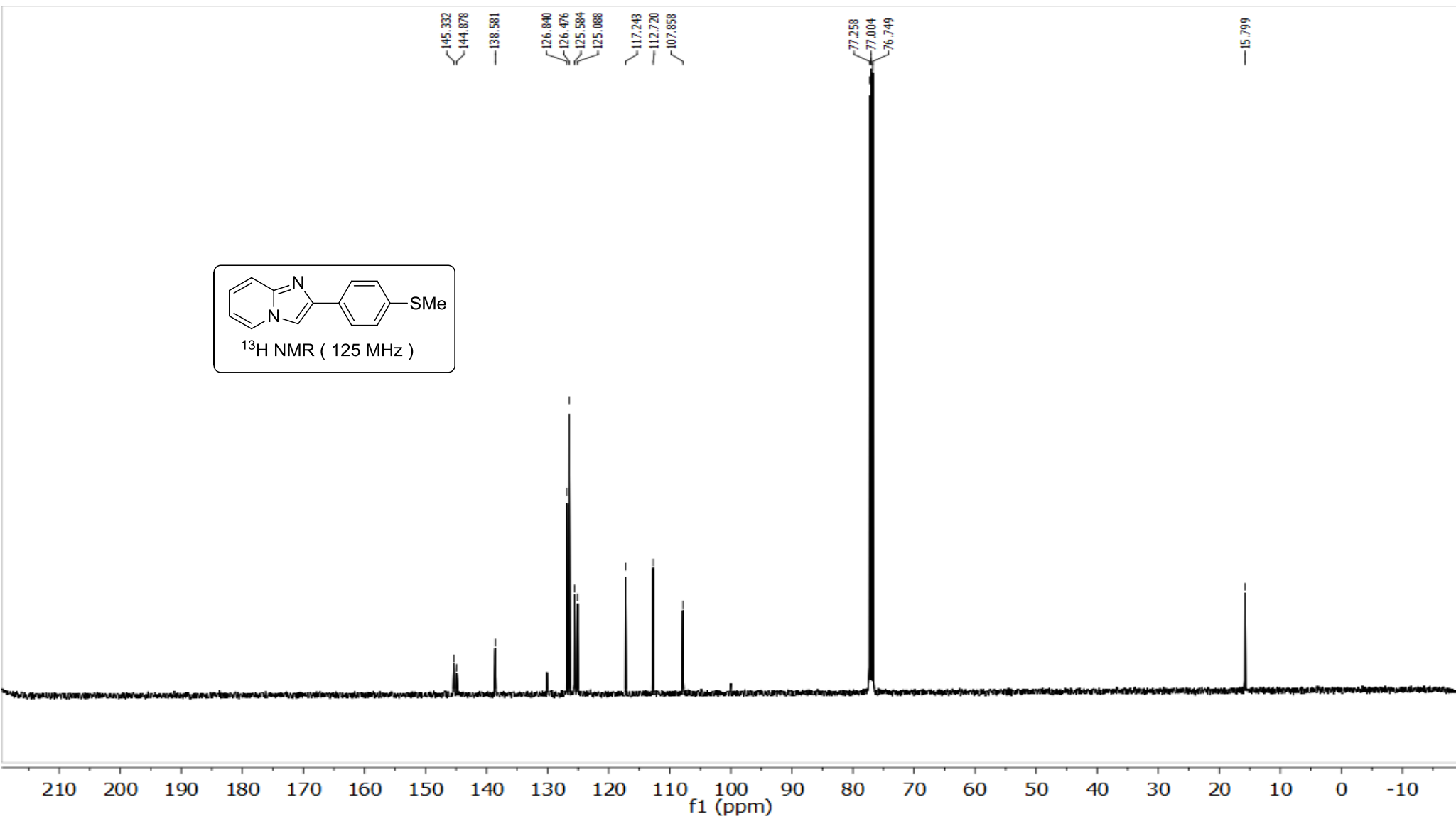
3.28

2.537

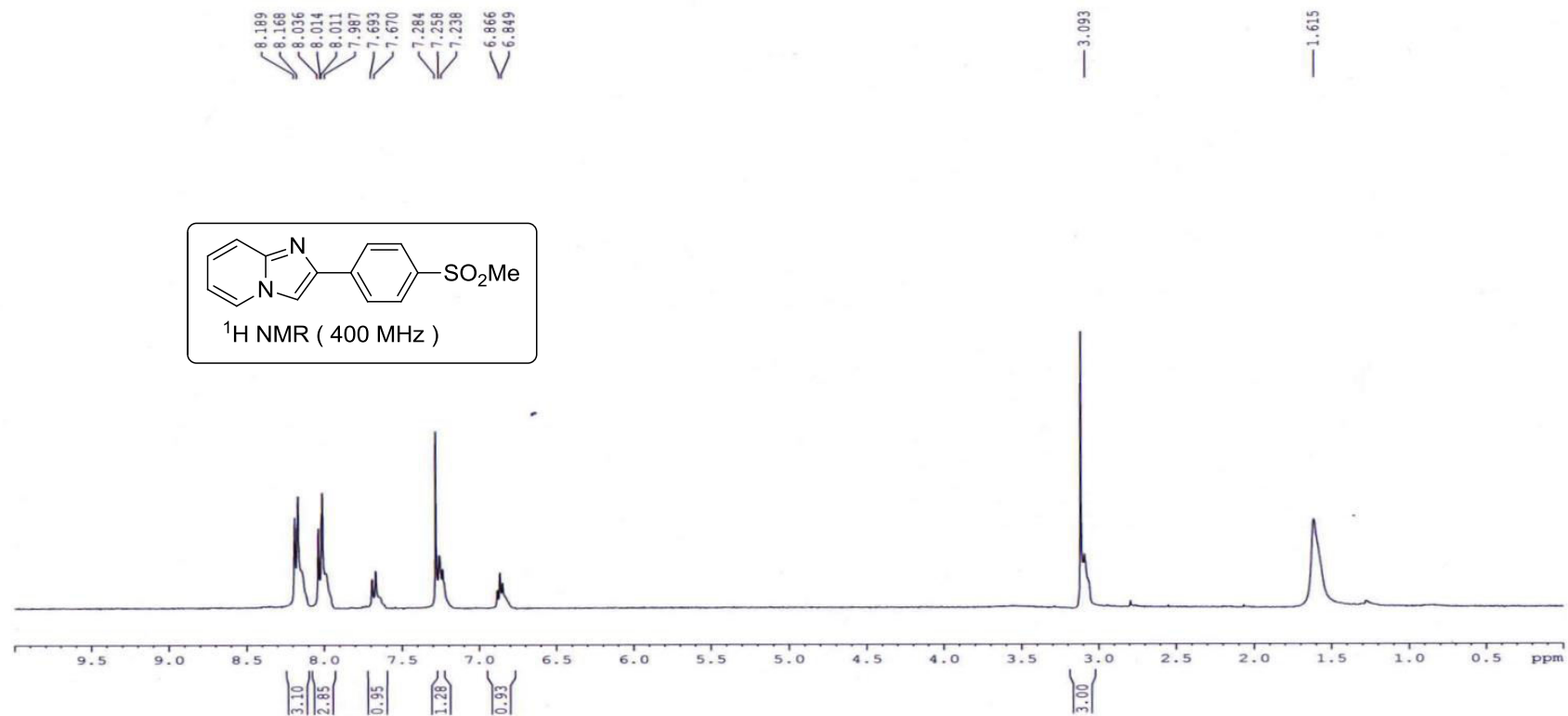
13.0 12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0

f1 (ppm)

2-(4- (methylthio)phenyl)imidazo[1,2-a]pyridine (9) :



2-(4-(methylsulfonyl)phenyl)imidazo[1,2-a]pyridine (10) :



2-(4-(methylsulfonyl)phenyl)imidazo[1,2-a]pyridine (10) :

