Supporting Information

Palladium Meets Copper: One-Pot Tandem Synthesis of Pyrido Fused Heterocycle via Sonogashira conjoined Electrophilic Cyclization

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X-ray crystallographic studies

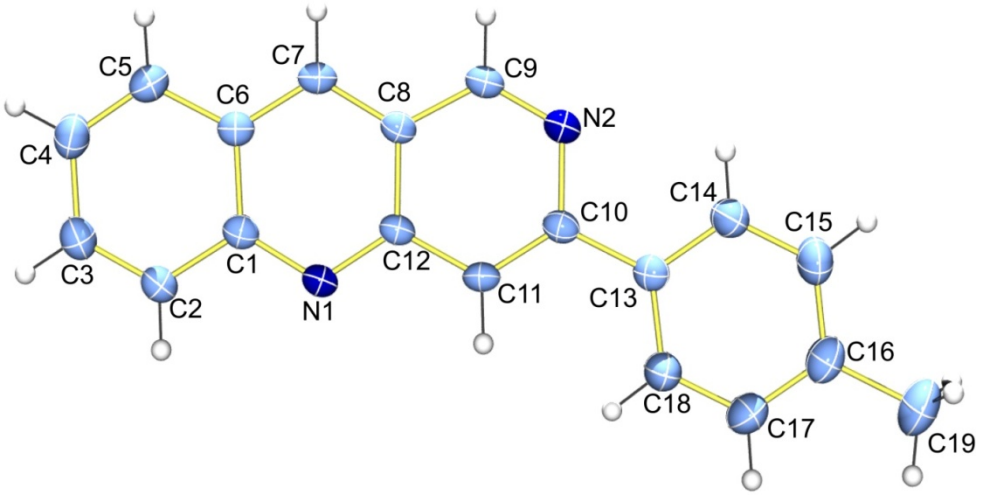


Figure 1. ORTEP drawing of compound **4b** drawn at 50% probability level

The crystal of compound **4b** (CCDC No: **1486060**) was generated in CH_2Cl_2/Hexane. The intensity data for SON-387 was collected on an Bruker Kappa Apex-CCD diffractometer equipped with graphite monochromatic Mo-Kα radiation (λ = 0.71073 Å) at 100(2) K[1].A multi-scan correction was applied. The structure was solved by the direct methods using SIR-92 and refined by full-matrix least-squares refinement techniques on F^2 using SHELXL97[2]. The hydrogen atoms were placed into the calculated positions and included in the last cycles of the refinement. All calculations were done using Wingx software package[3].

Table 1. Crystal data collection and structure refinement parameters for **4b**

	Compound **4b**
Empirical formula	$C_{19}H_{14}N_2$
Formula weight	270.32
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P\,2_1/c$
A	13.739(5)Å
B	5.908(5)Å
C	17.319(5)Å
α (°)	90.000(5)°
β (°)	91.208(5)°
γ (°)	90.000(5)°
Volume	1405.5(14) $Å^3$
Z	4
Density (calculated)	1.278 Mg/m^3
Absorption coefficient	0.076 mm^{-1}
F(000)	568
Crystal size	0.20 x 0.19 x 0.17 mm^3
Theta range for data collection	3.64 to 25.00°
Index ranges	$-16 \le h \le 16$, $-7 \le k \le 7$, $-20 \le l \le 20$
Reflections collected	15507
Independent reflections	2465 [*R*(int) = 0.0390]
Completeness to theta = 25.00°	99.8 %
Absorption correction	Multi-scan
Max. and min. transmission	0.9872 and 0.9850
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2465 / 0 / 190
Goodness-of-fit on F^2	1.038
Final *R* indices [I>2sigma(I)][a, b]	$R_1 = 0.0514$, $wR_2 = 0.1235$
R indices (all data)	$R_1 = 0.0823$, $wR_2 = 0.1365$
Largest diff. peak and hole	0.168 and -0.149e.$Å^{-3}$

[a] $R = \sum(\|Fo| - |Fc|)/\sum|Fo|$; [b] $wR = \{\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]\}^{1/2}$

References

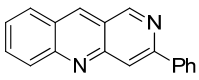
1. a) *SMART: Bruker Molecular Analysis Research Tool*, version 5.618. Bruker Analytical X-ray System, **2000**. b) *SAINT-NT*, version 6.45, Bruker Analytical X-Ray System, **2003**. c) *SHELXTL-NT*, version 6.10, Bruker Analytical X-ray System, **2000**.

2. G. M. Sheldrick, Acta Cryst., **2008**, A64, 112-122.

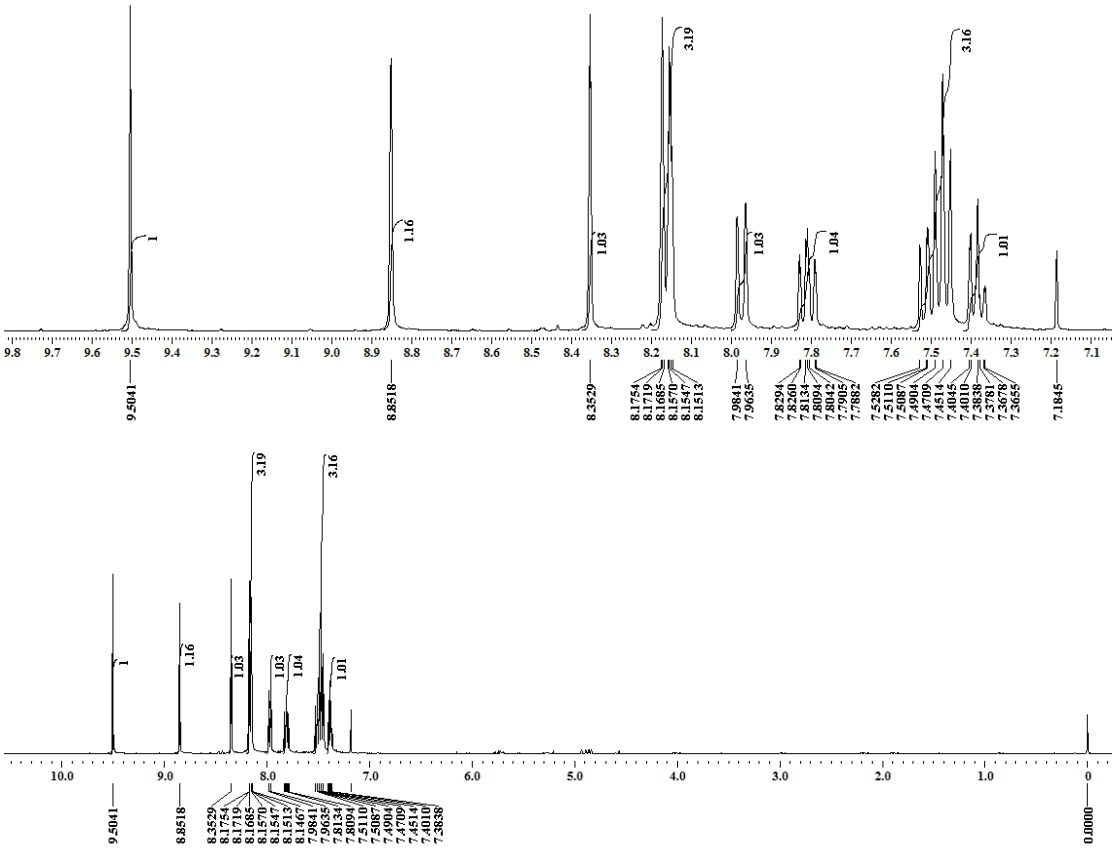
3. L. J. Farrugia, WinGX Version 1.80.05, *An integrated system of Windows Programs for the Solution, Refinement and Analysis of Single Crystal X-Ray Diffraction Data;* Department of Chemistry, University of Glasgow (**1997-2009**).

Copies of ^{1}H NMR, ^{13}C NMR and HRMS

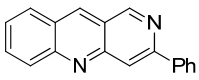
^{1}H NMR



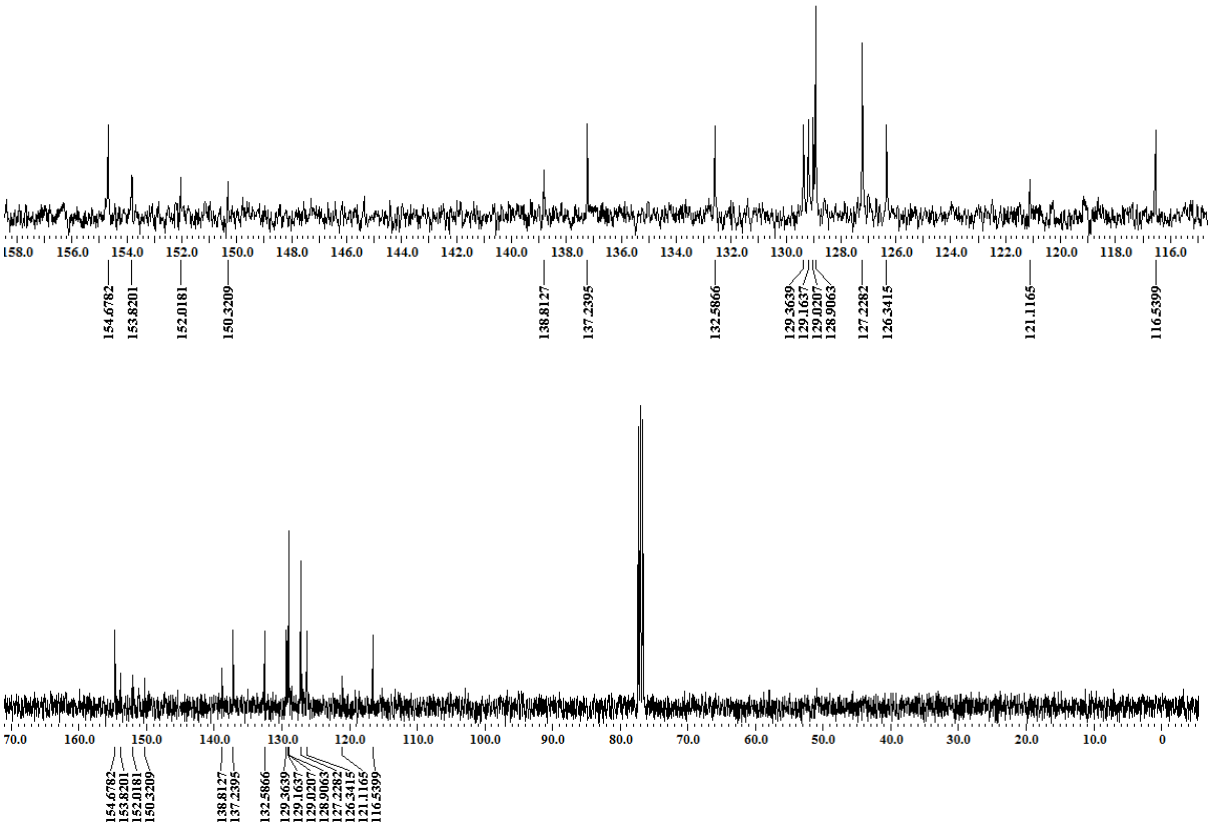
3-Phenylbenzo[*b*][1,6]naphthyridine (4a)



^{13}C NMR



3-Phenylbenzo[*b*][1,6]naphthyridine (4a)



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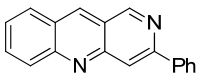

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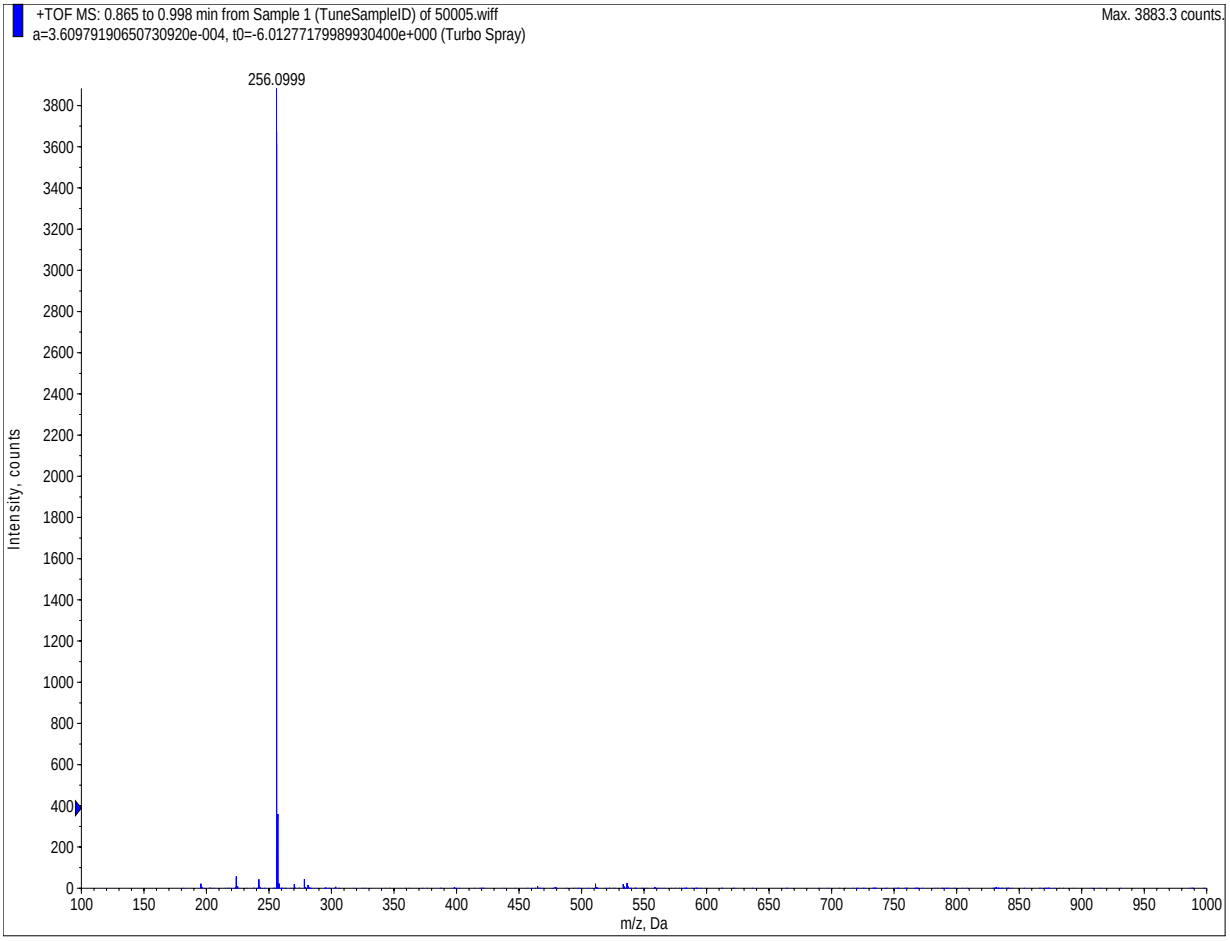

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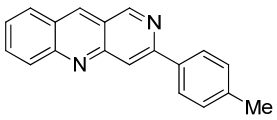

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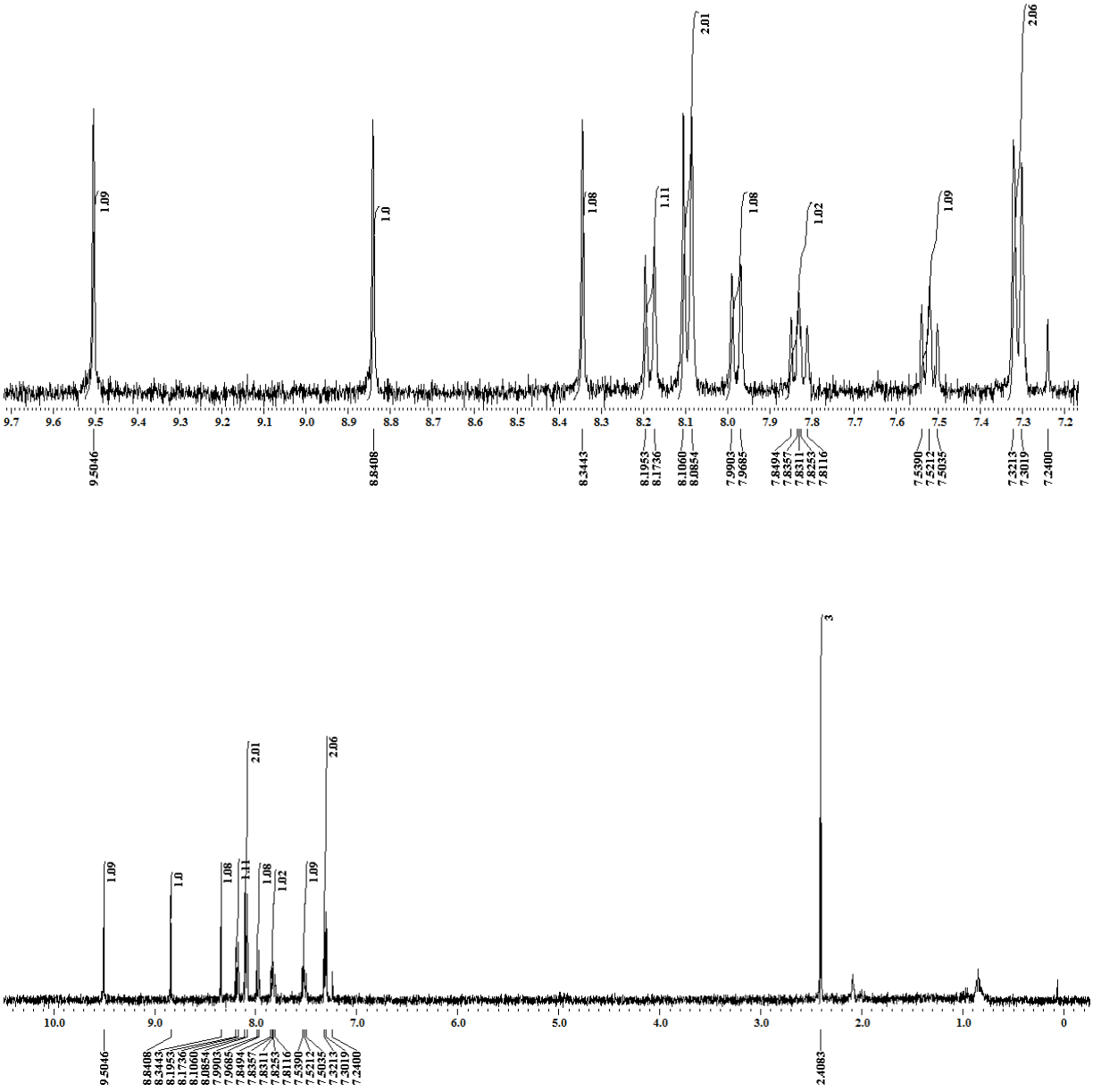

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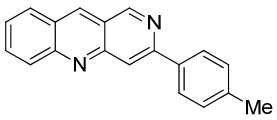

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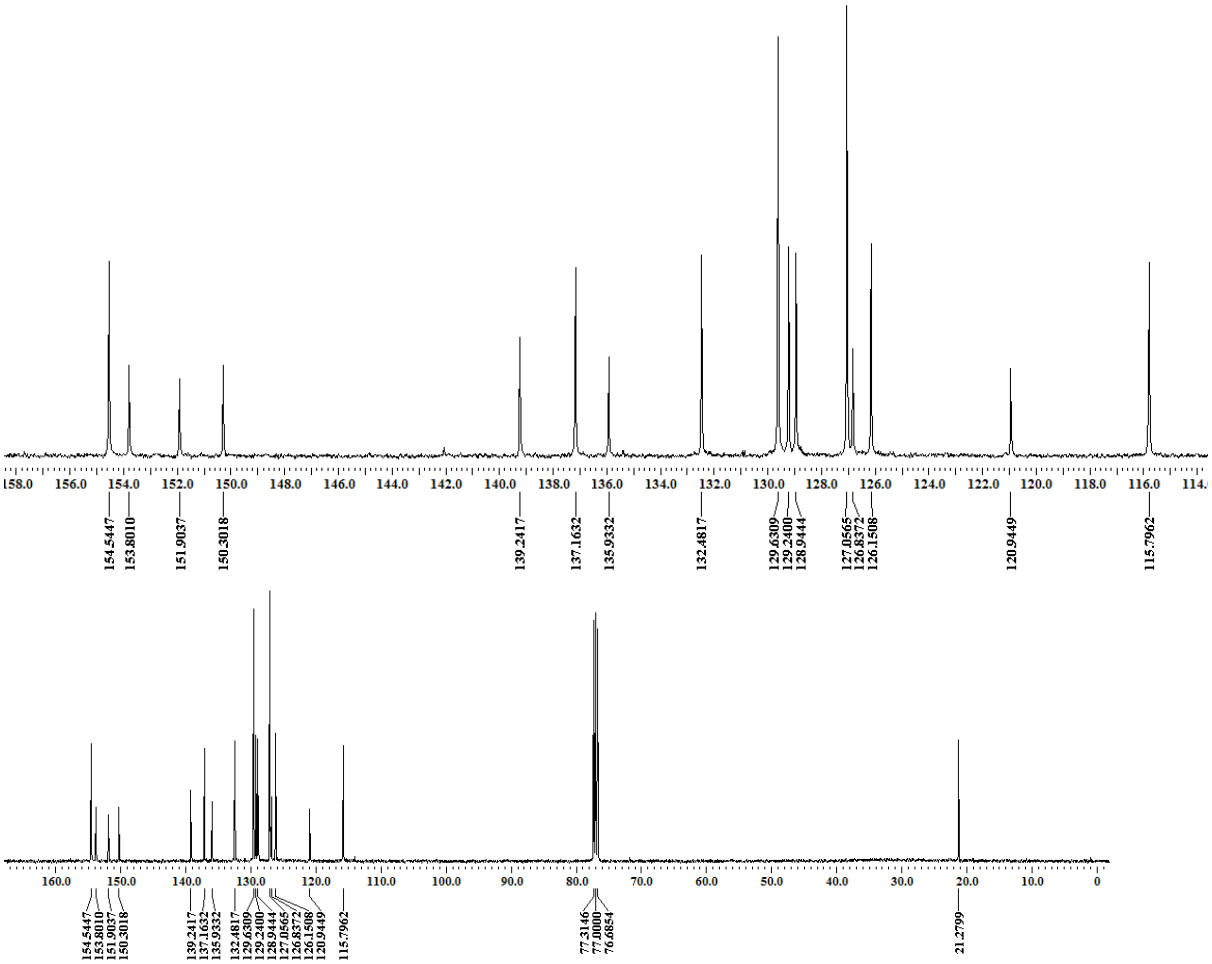

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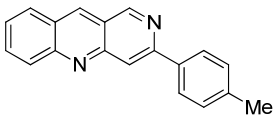

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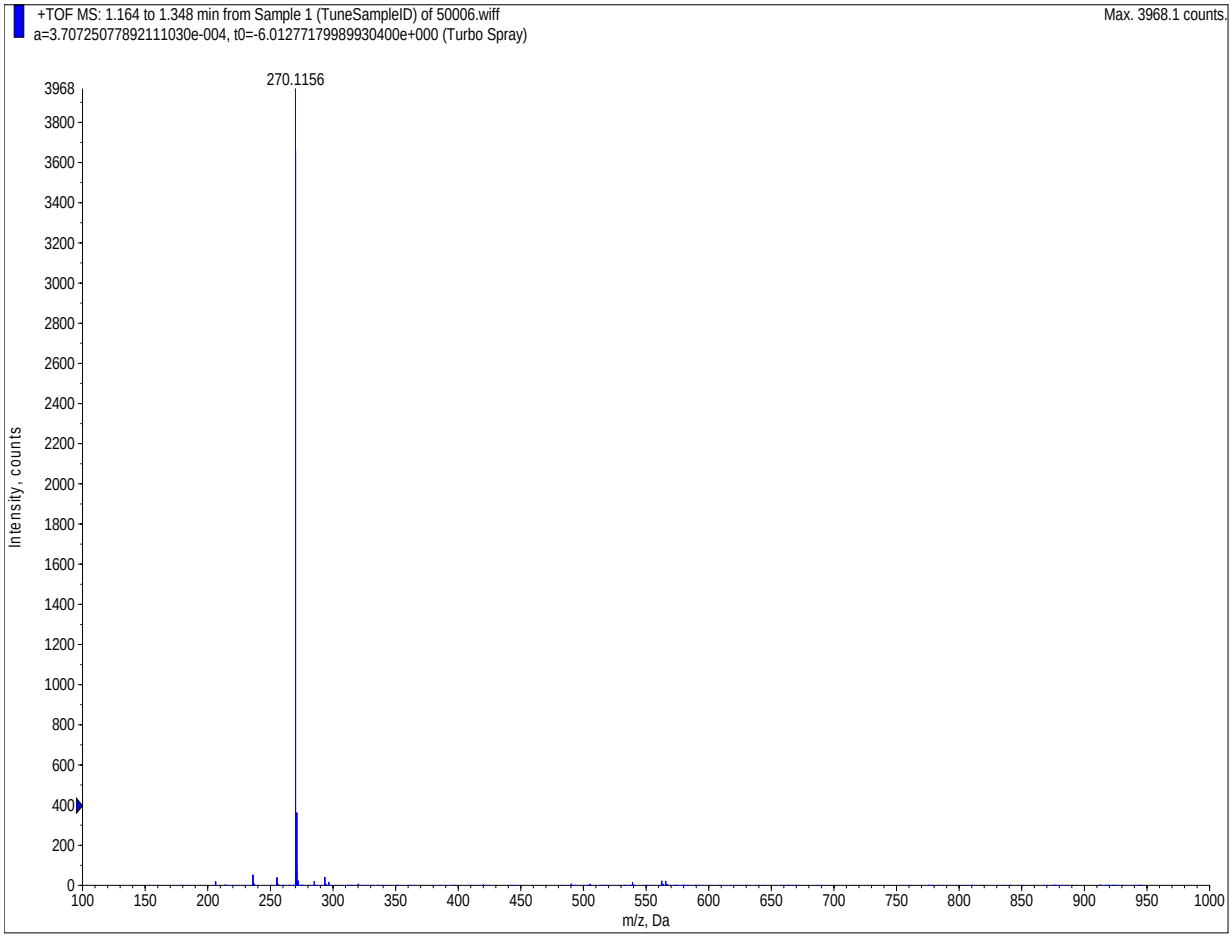

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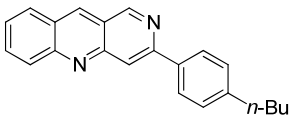

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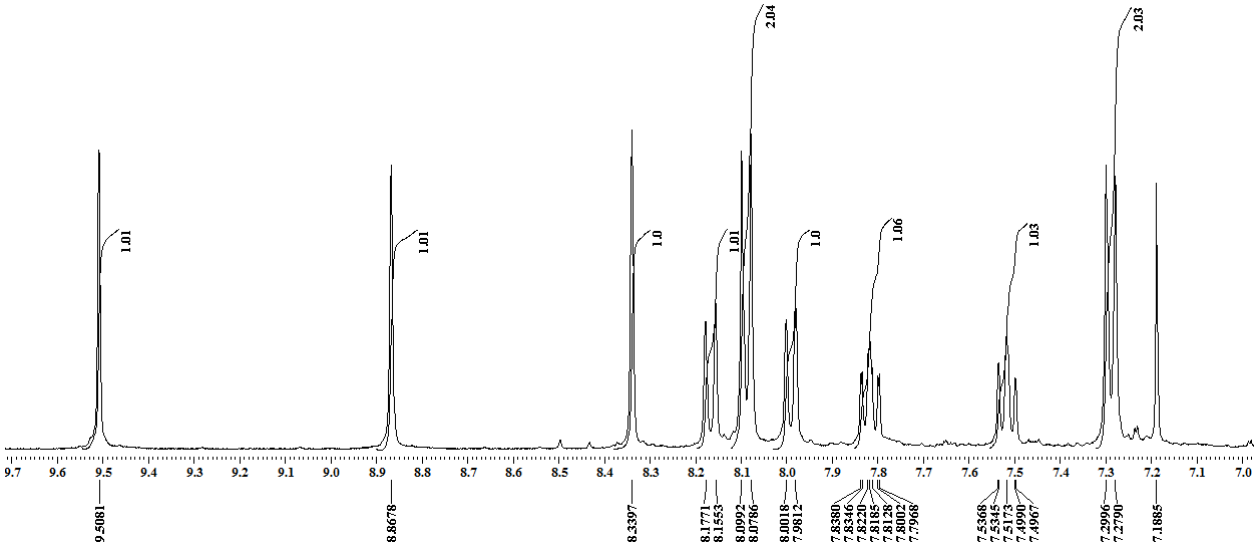

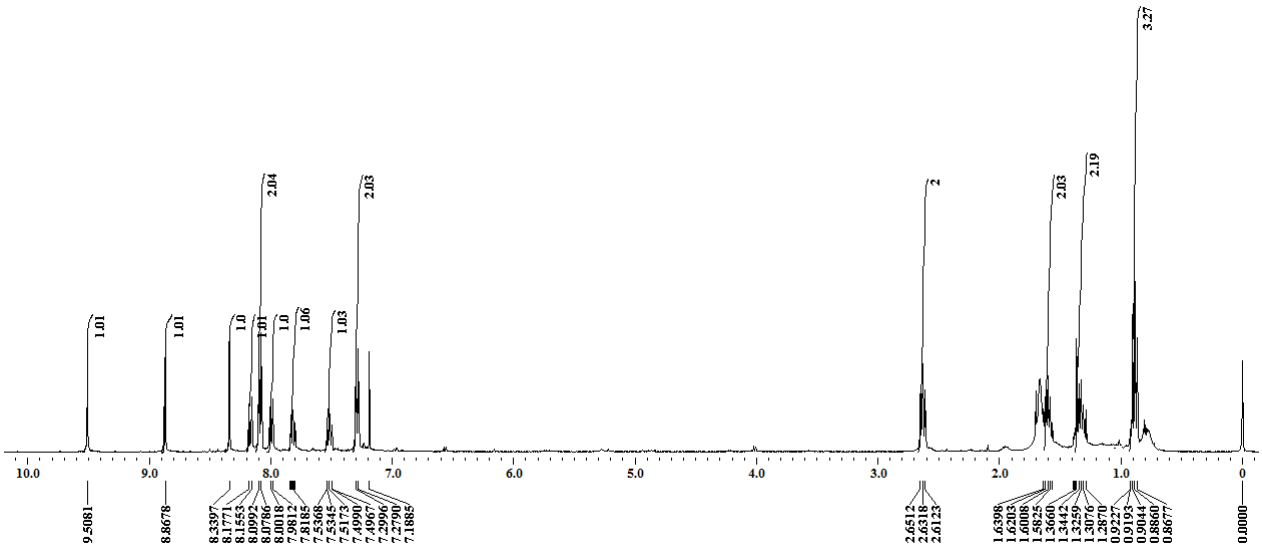

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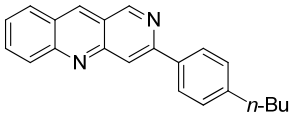

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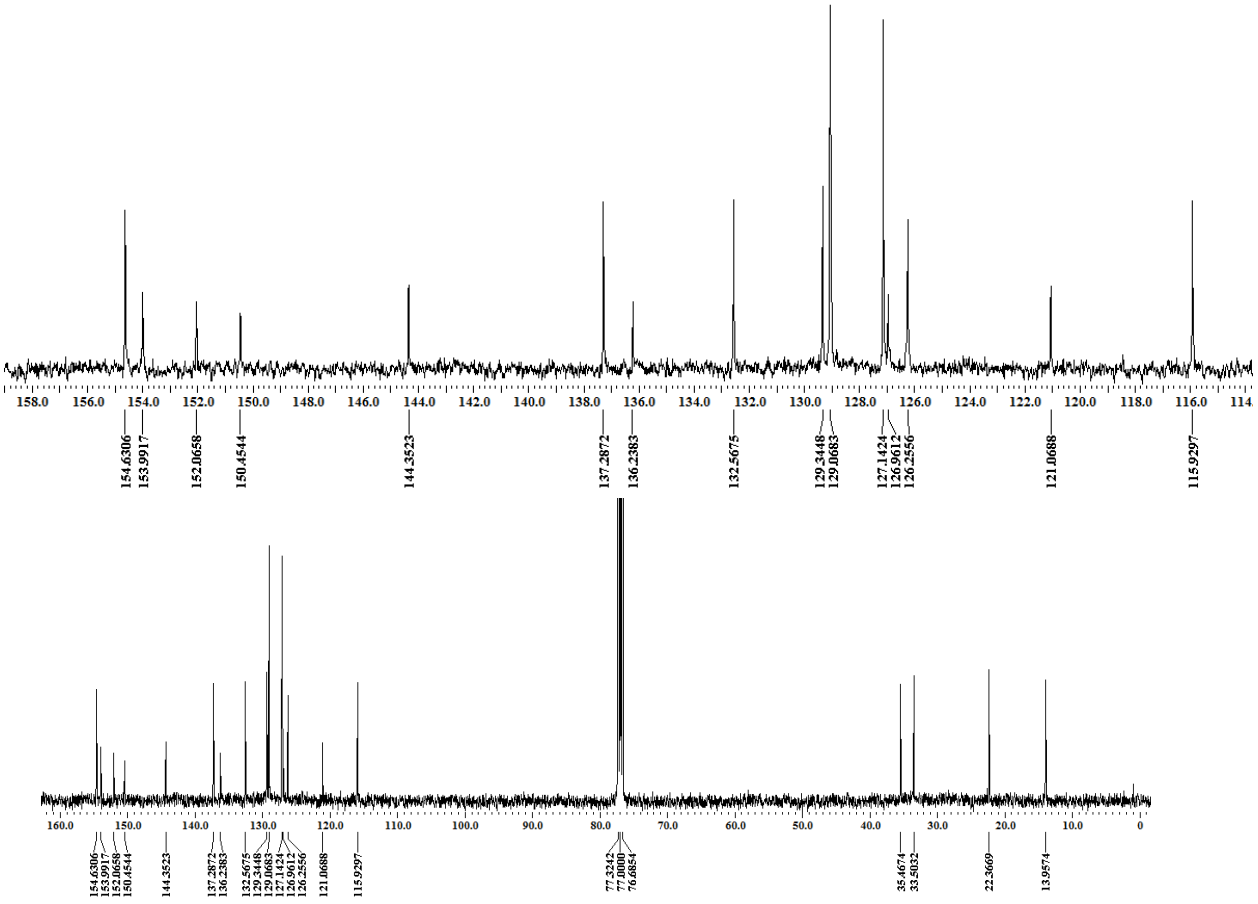

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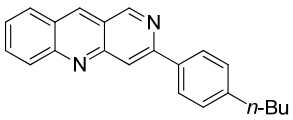

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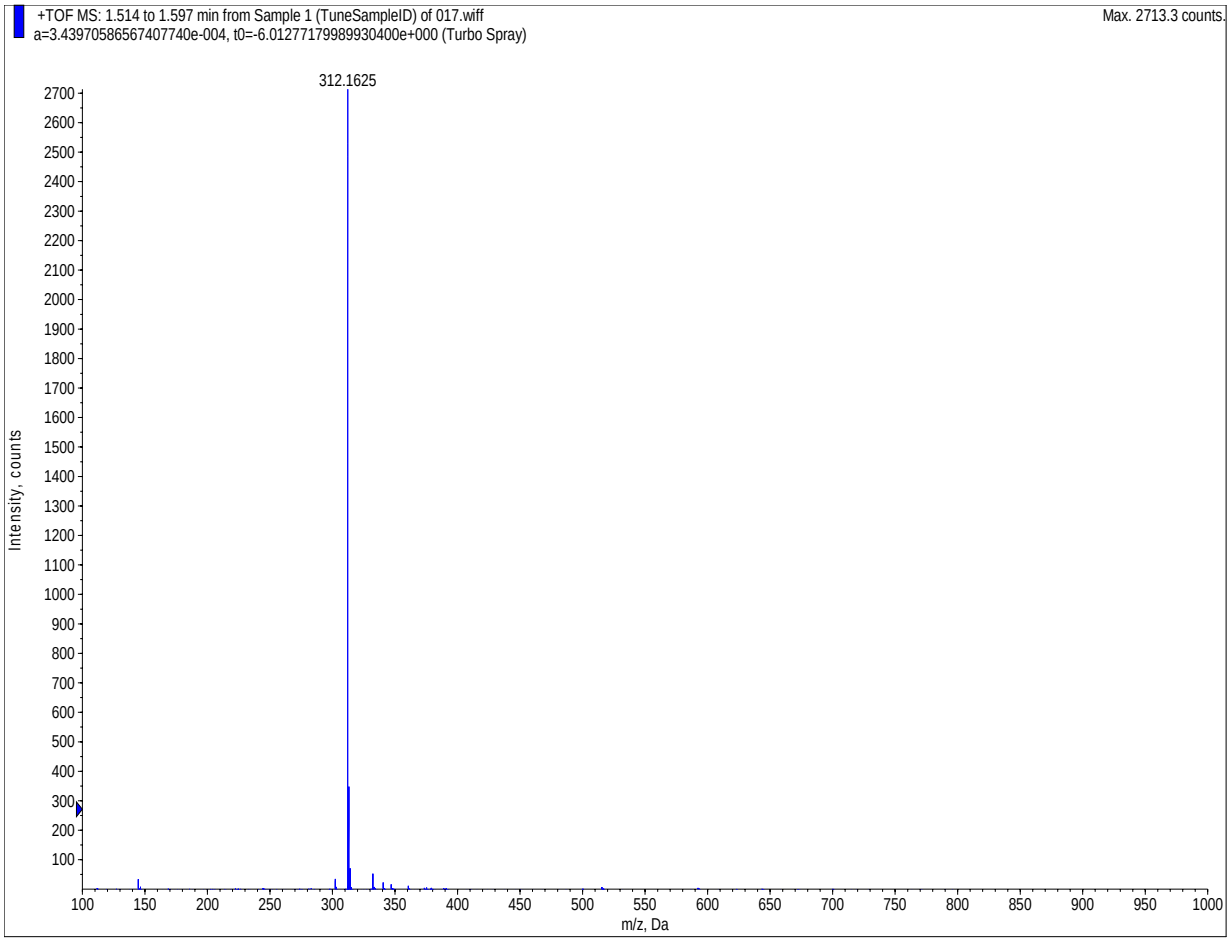

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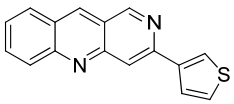

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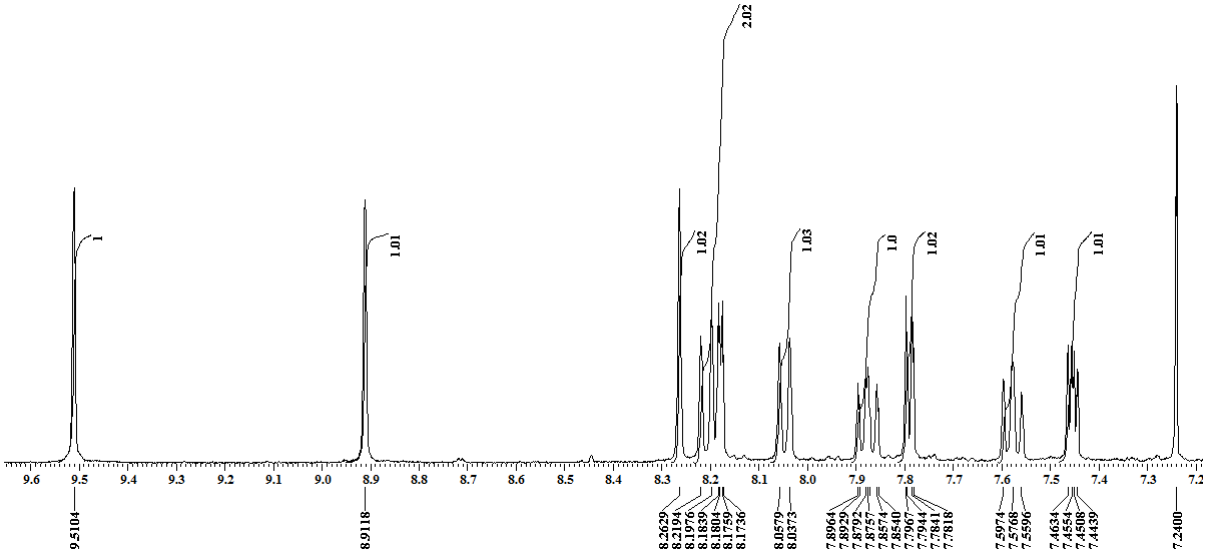

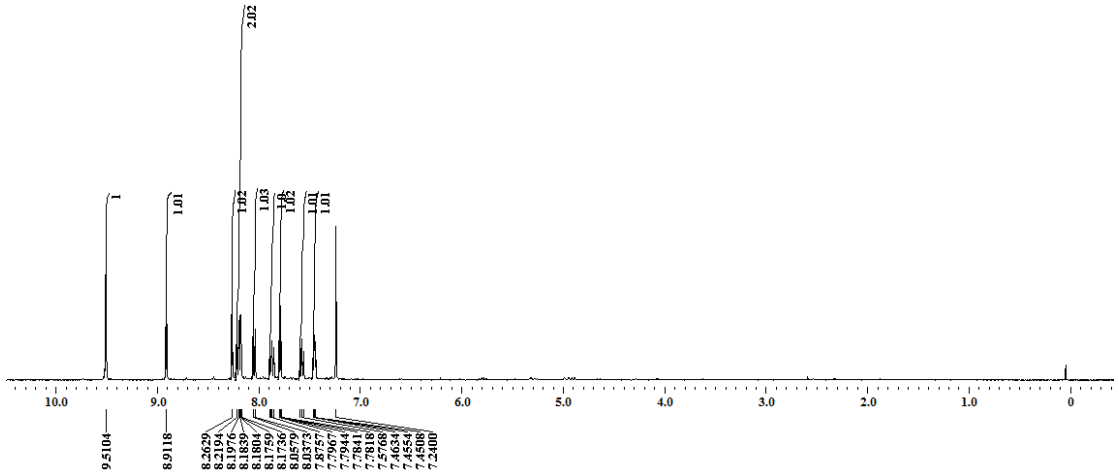

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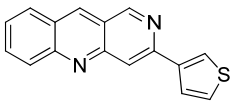

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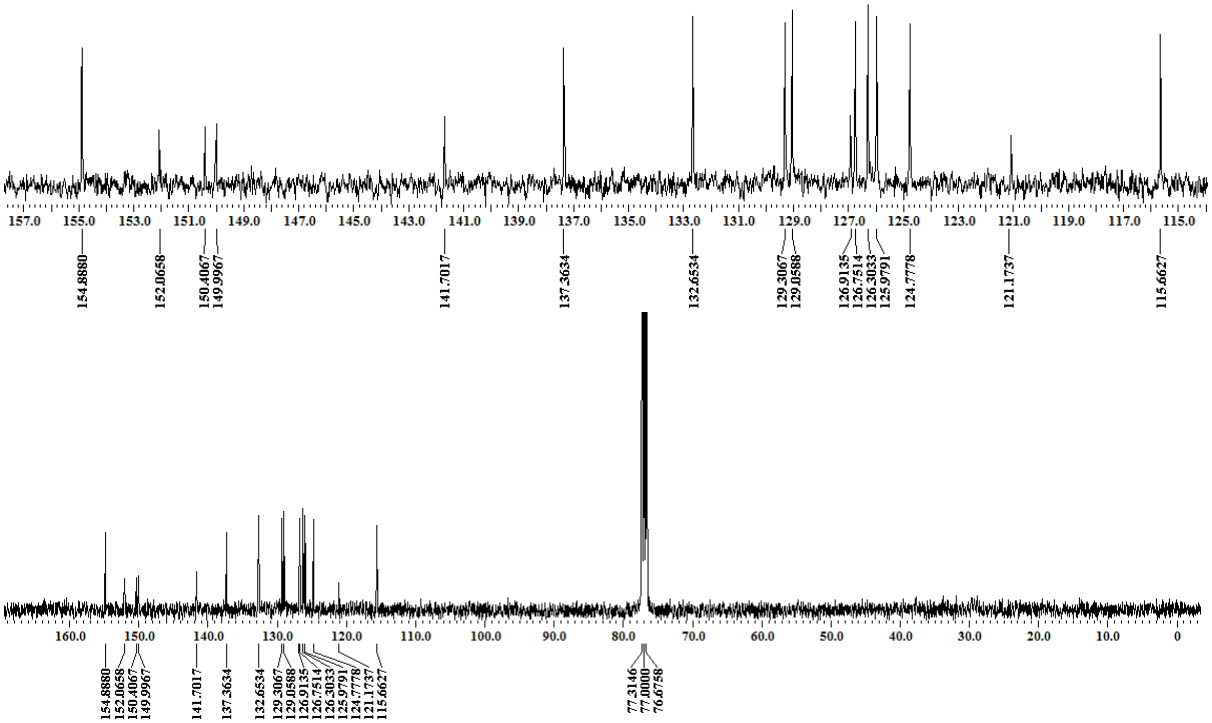

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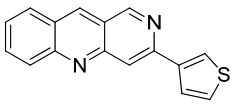

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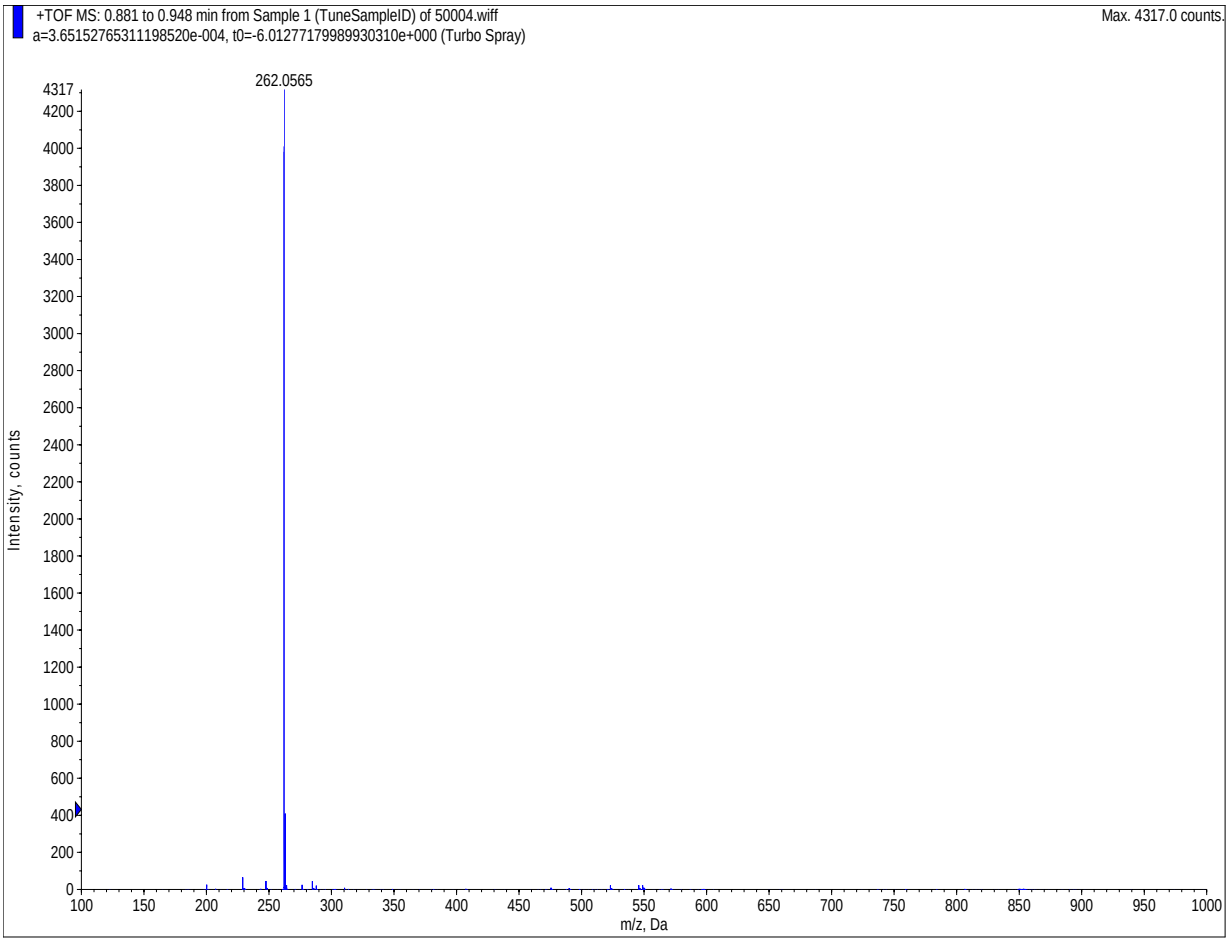

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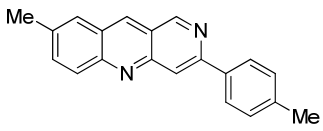

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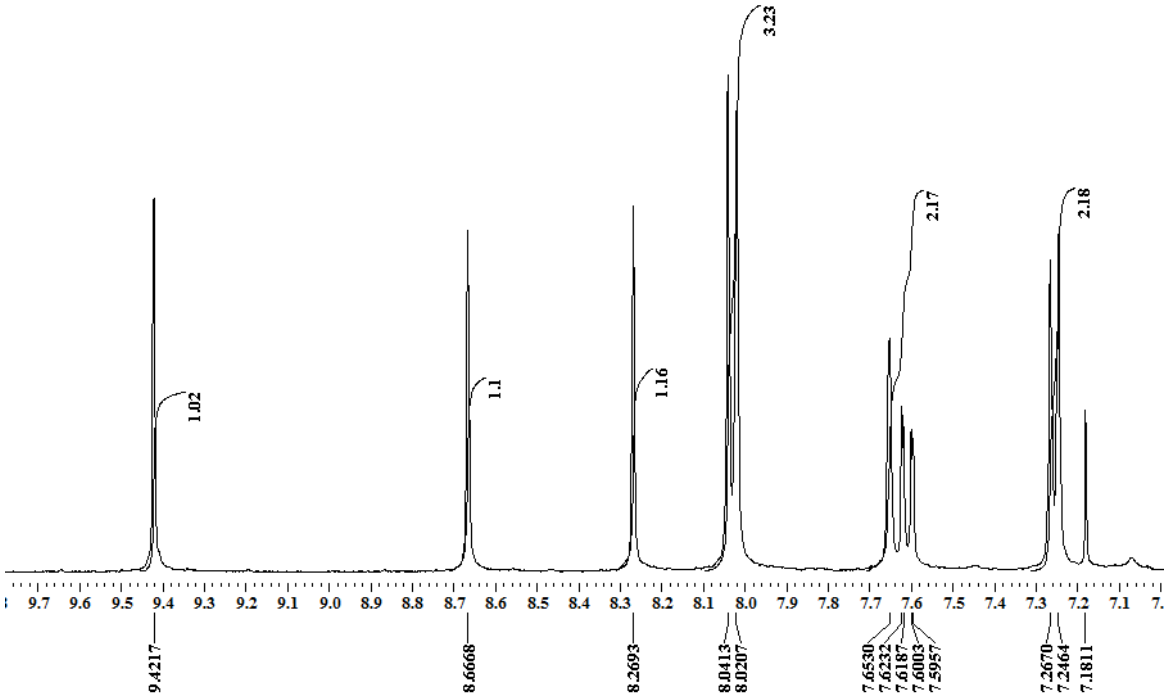

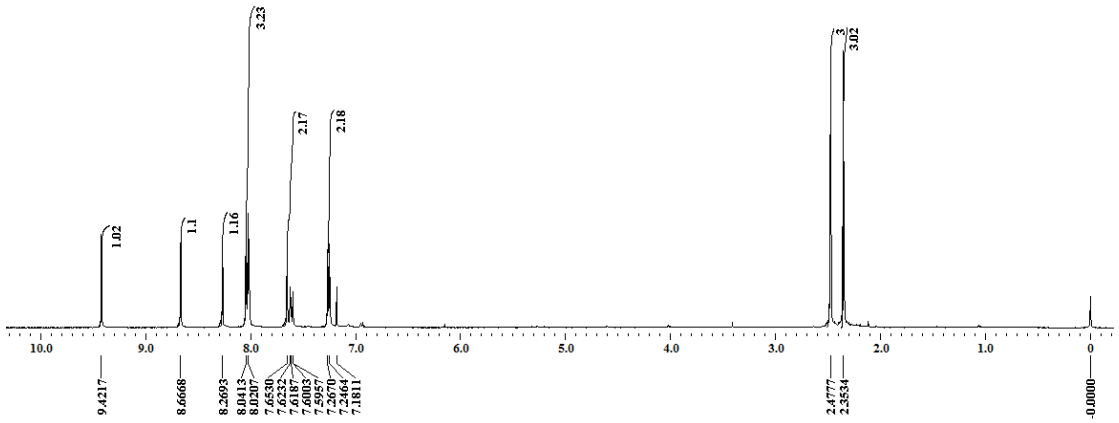

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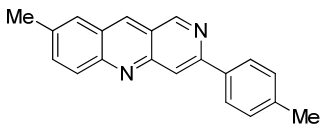
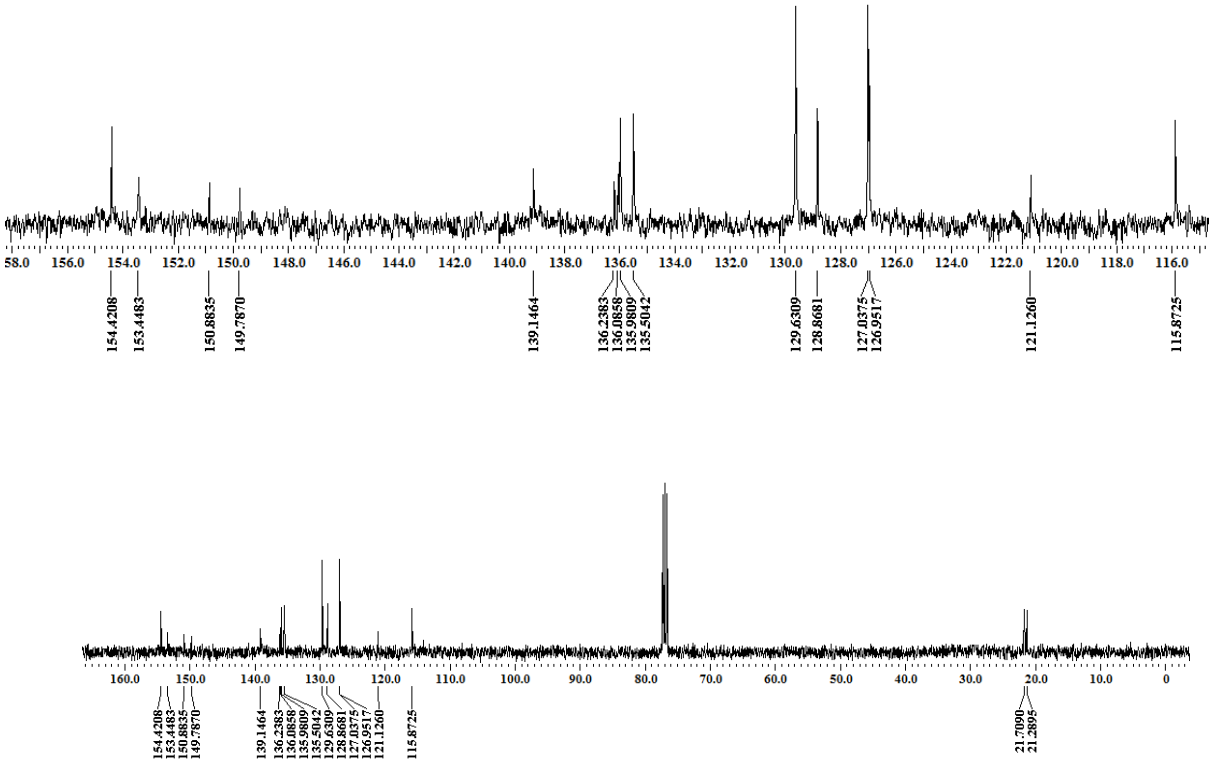


8-Methyl-3-(*p*-tolyl)benzo[*b*][1,6]naphthyridine (4e)



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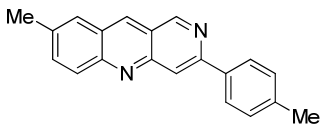

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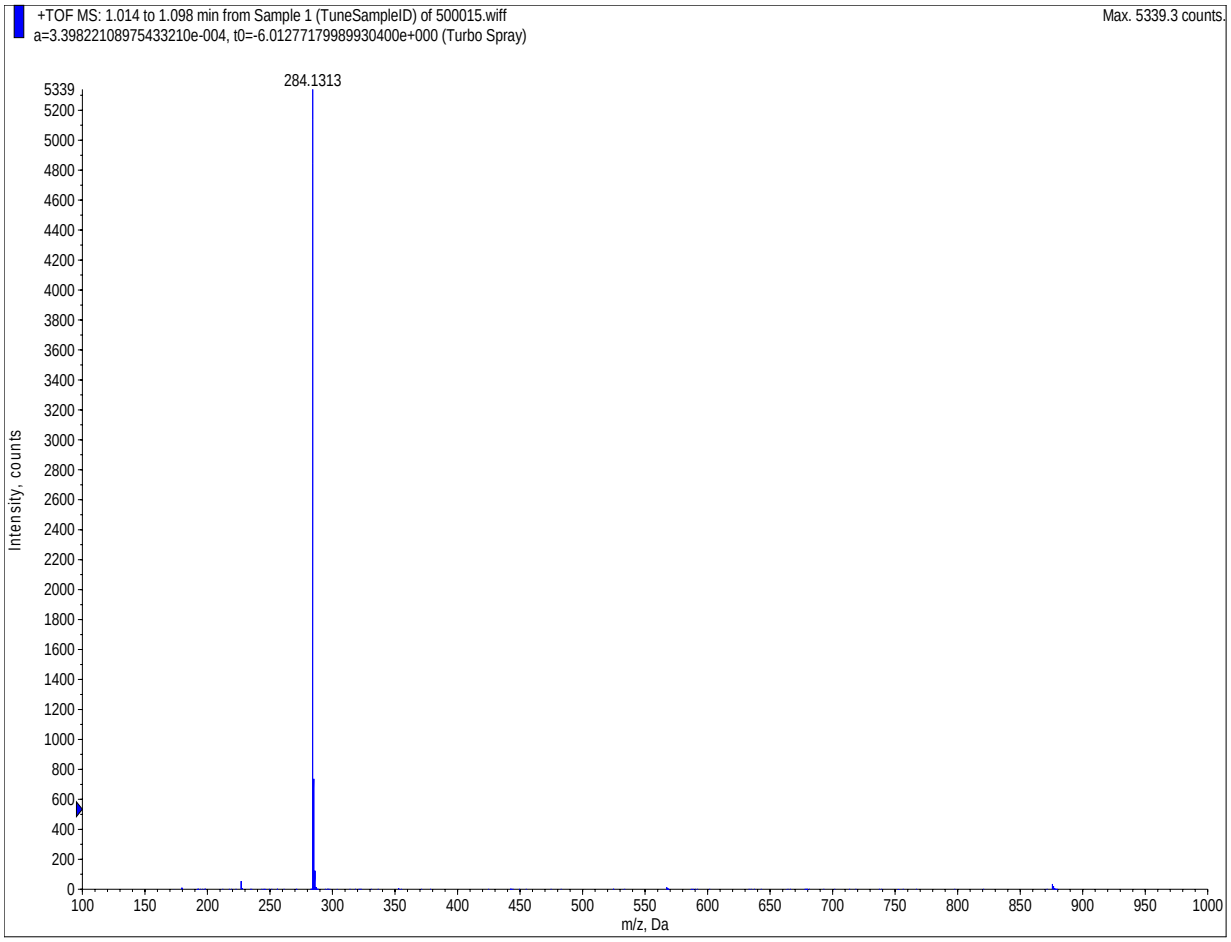

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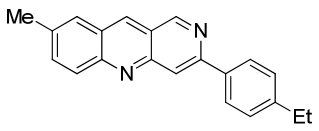

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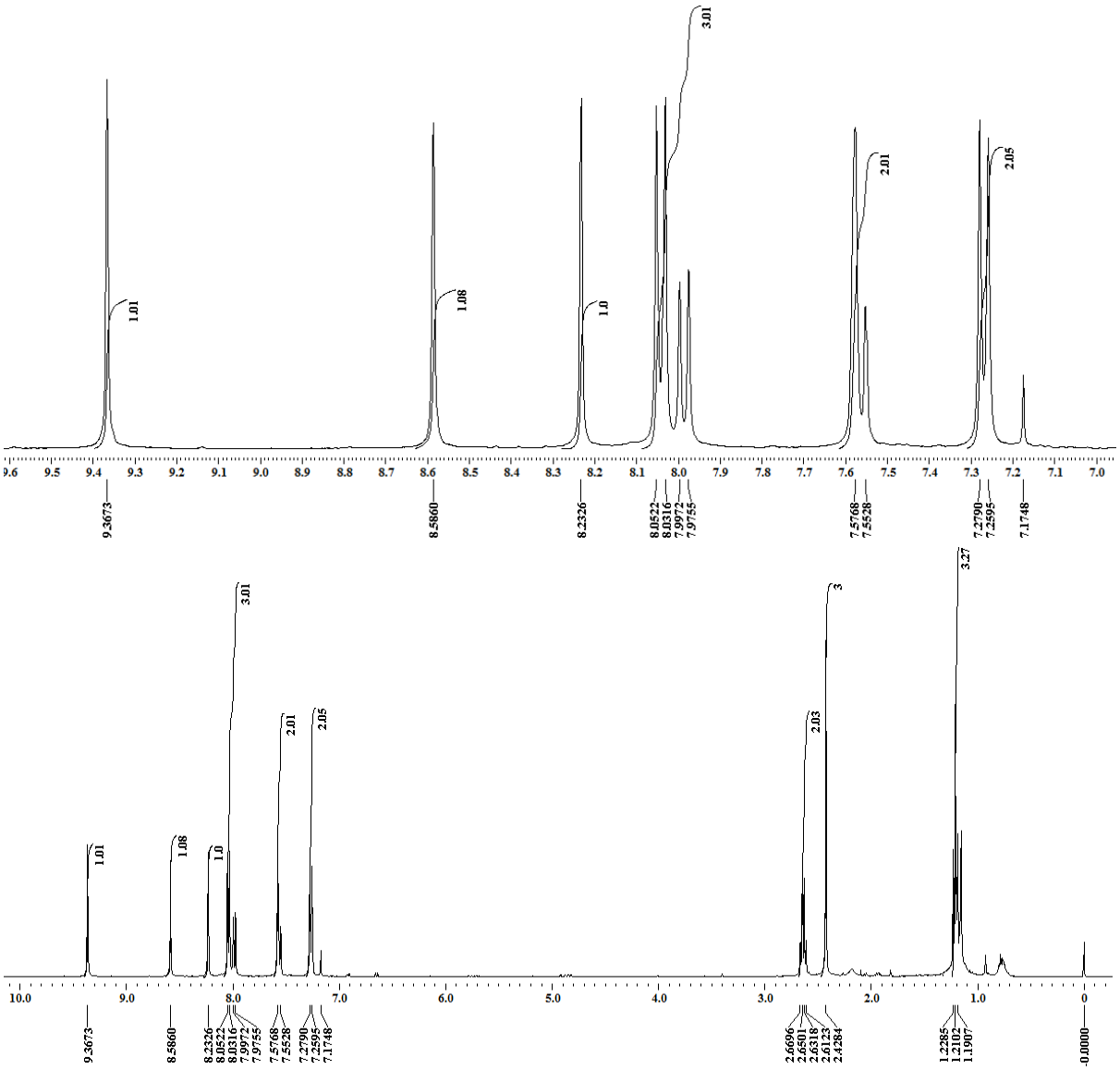

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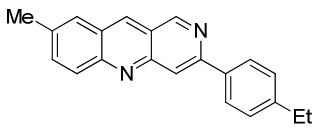

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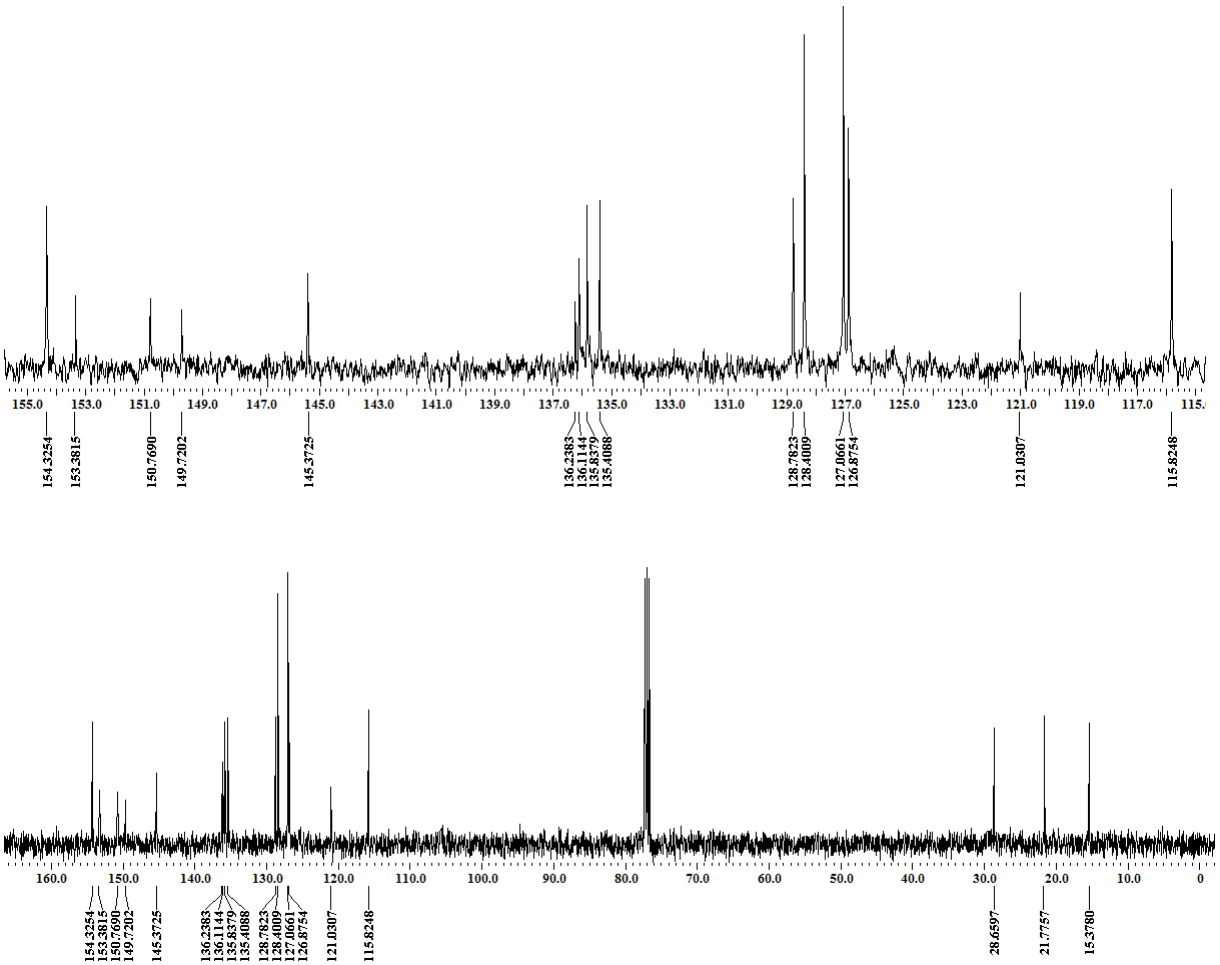

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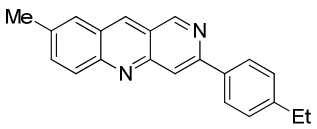

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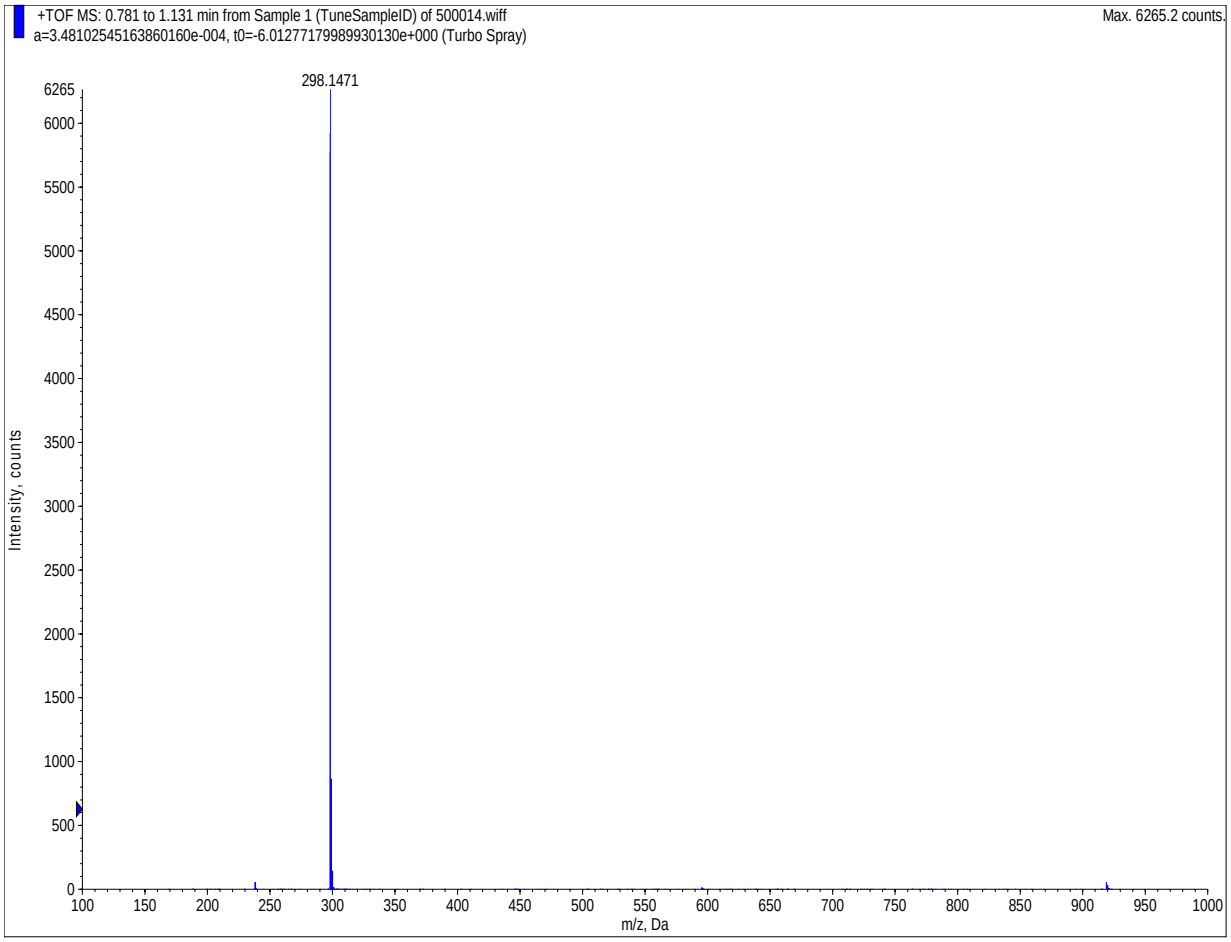

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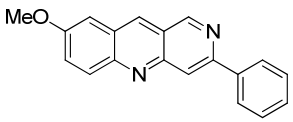

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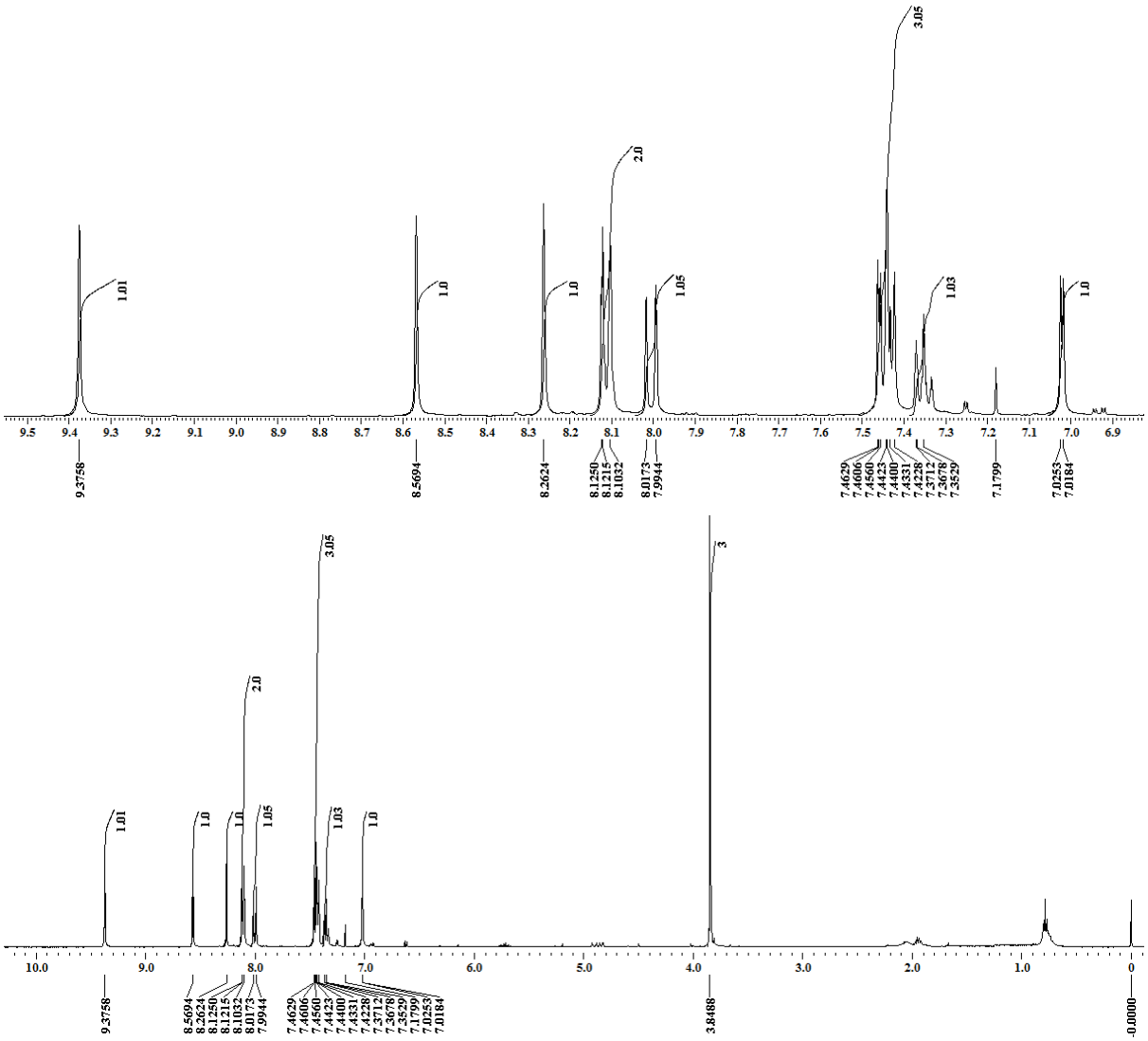

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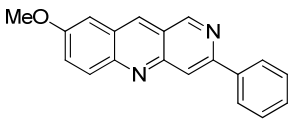

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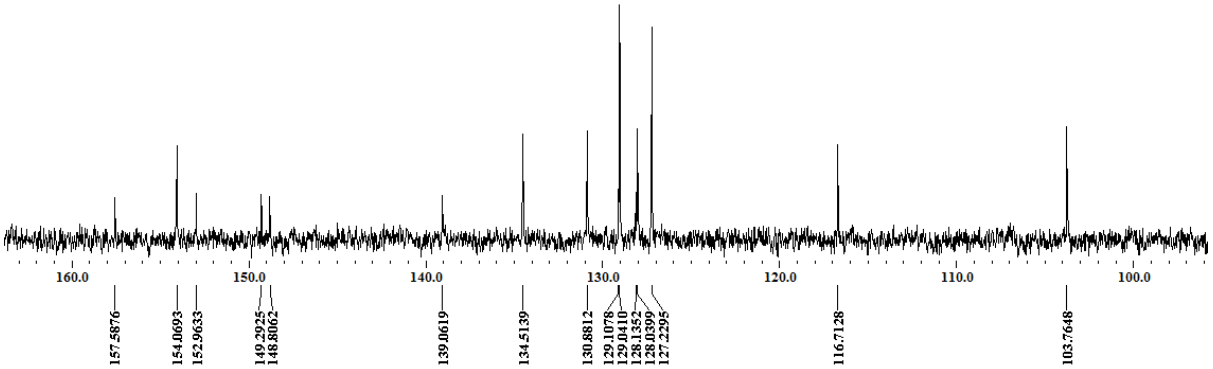

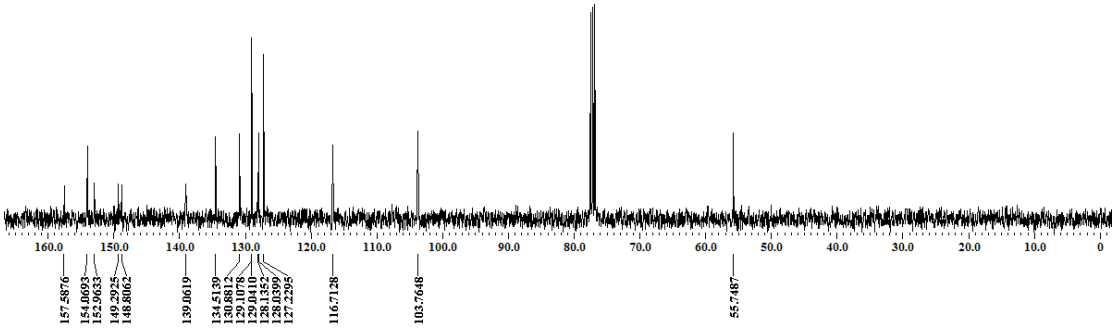

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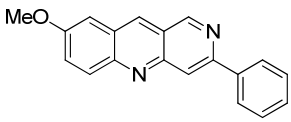

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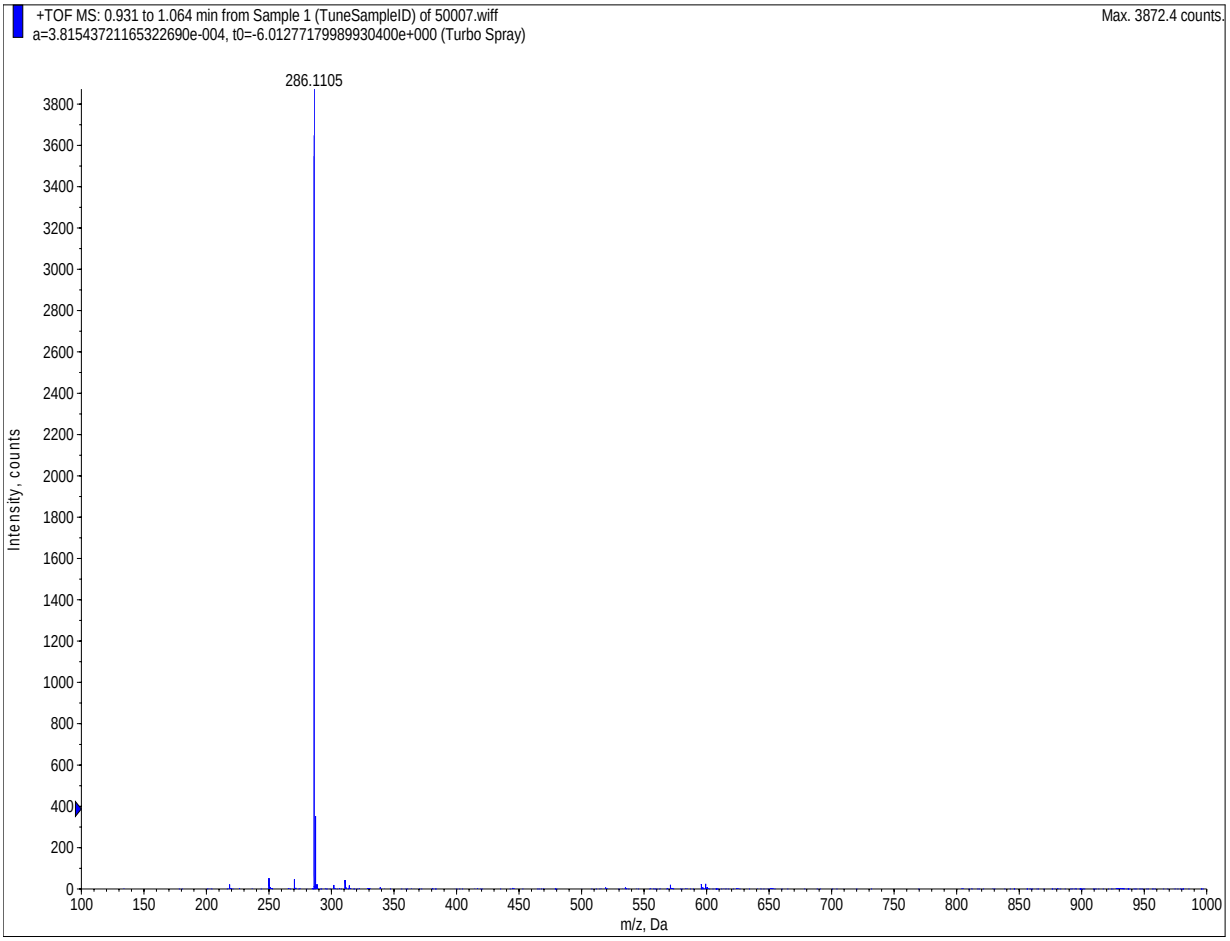
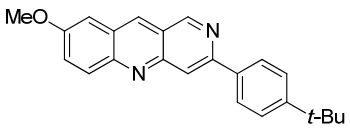
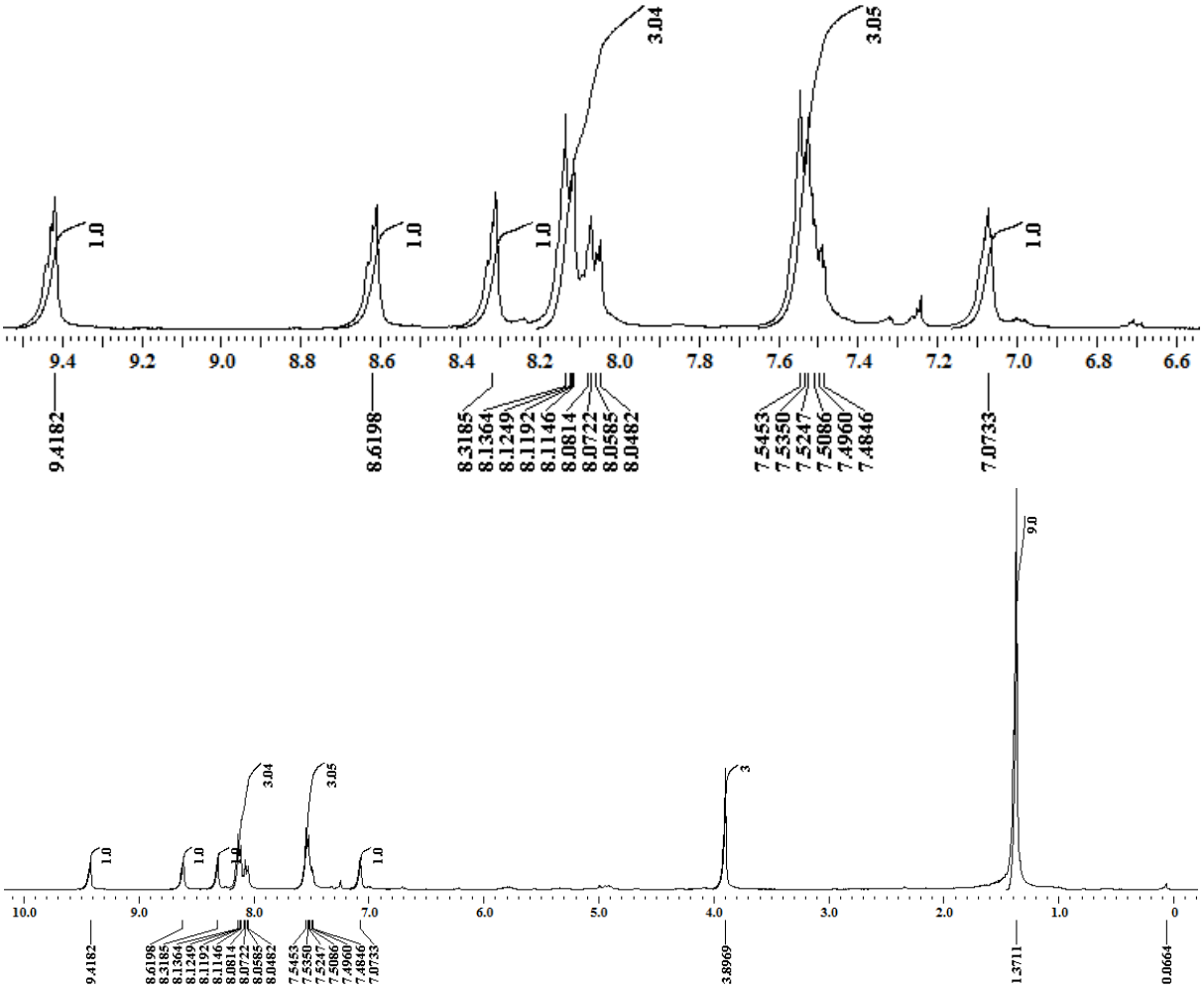


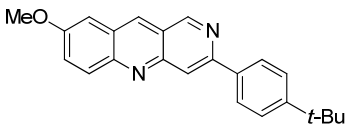
^{1}H NMR



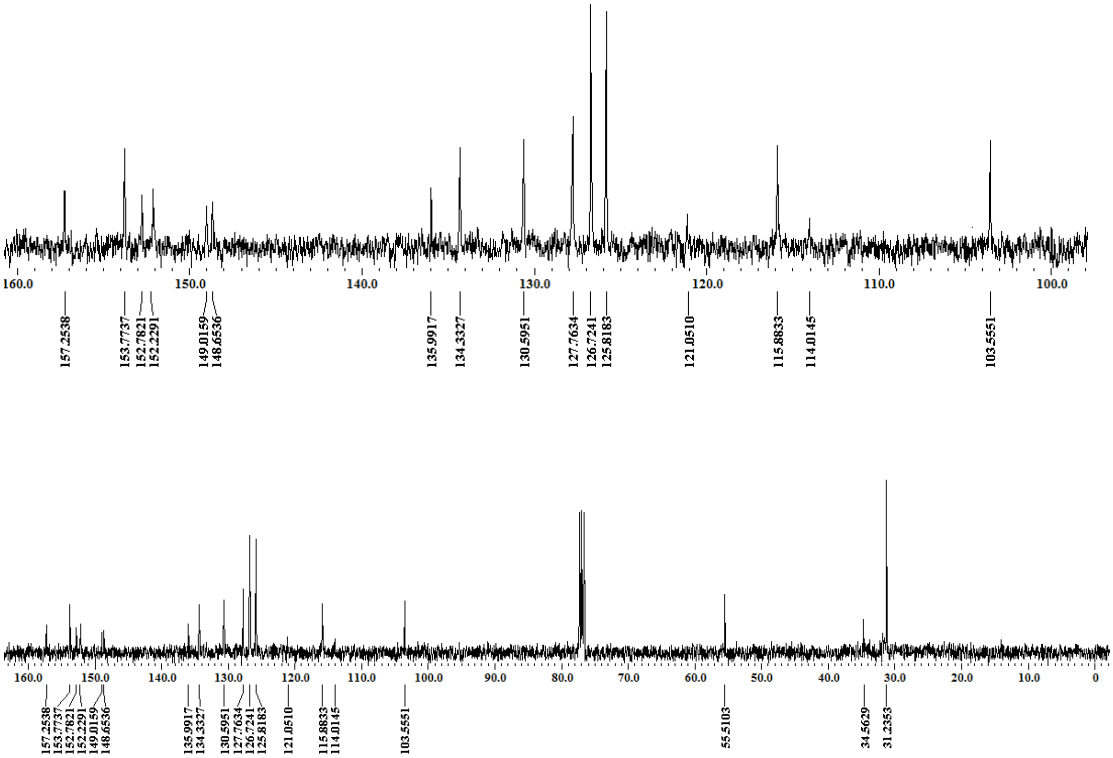
3-(4-(*t*-Butyl)phenyl)-8-methoxybenzo[*b*][1,6]naphthyridine (4h)



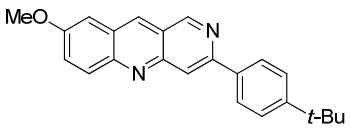
^{13}C NMR



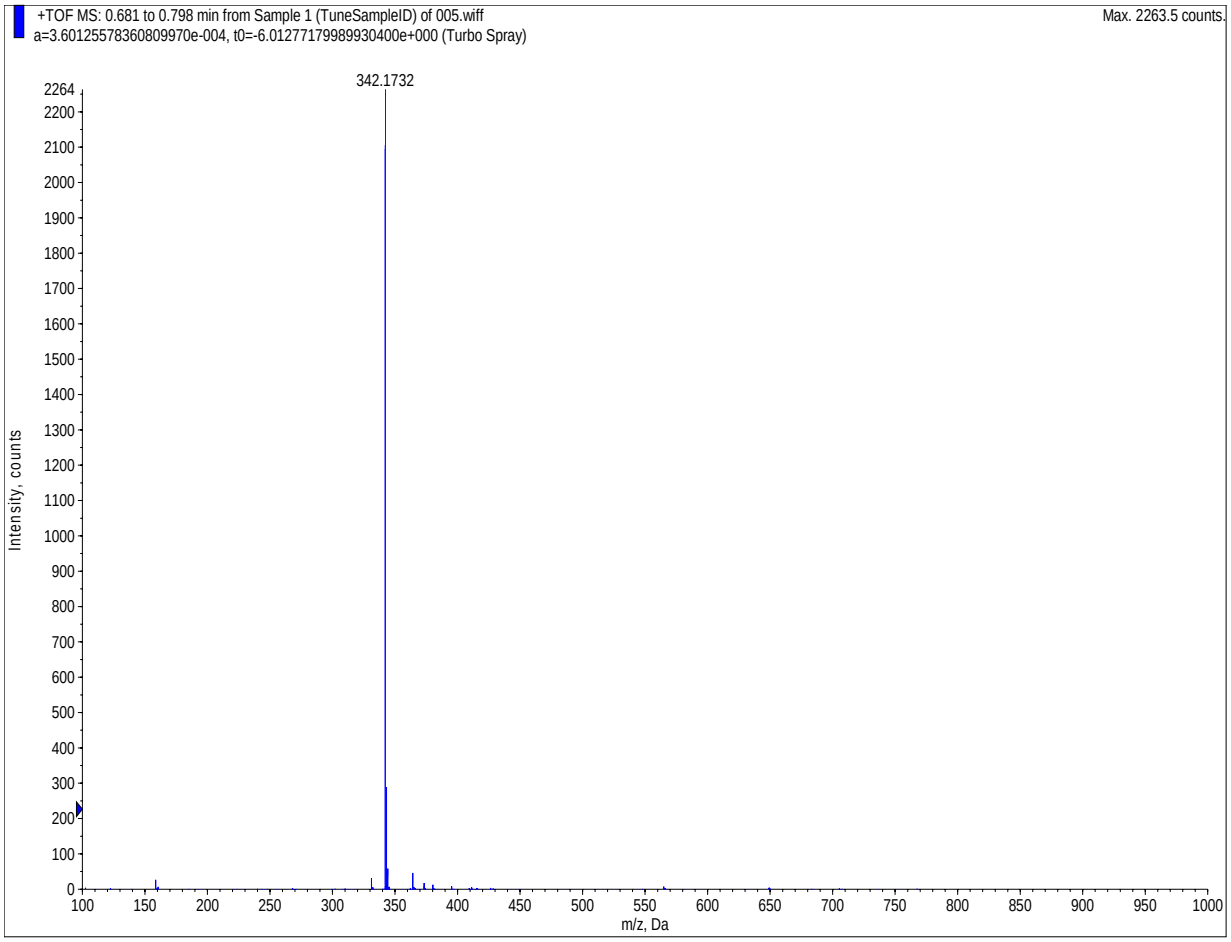
3-(4-(*t*-Butyl)phenyl)-8-methoxybenzo[*b*][1,6]naphthyridine (4h)



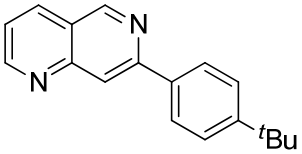
HRMS



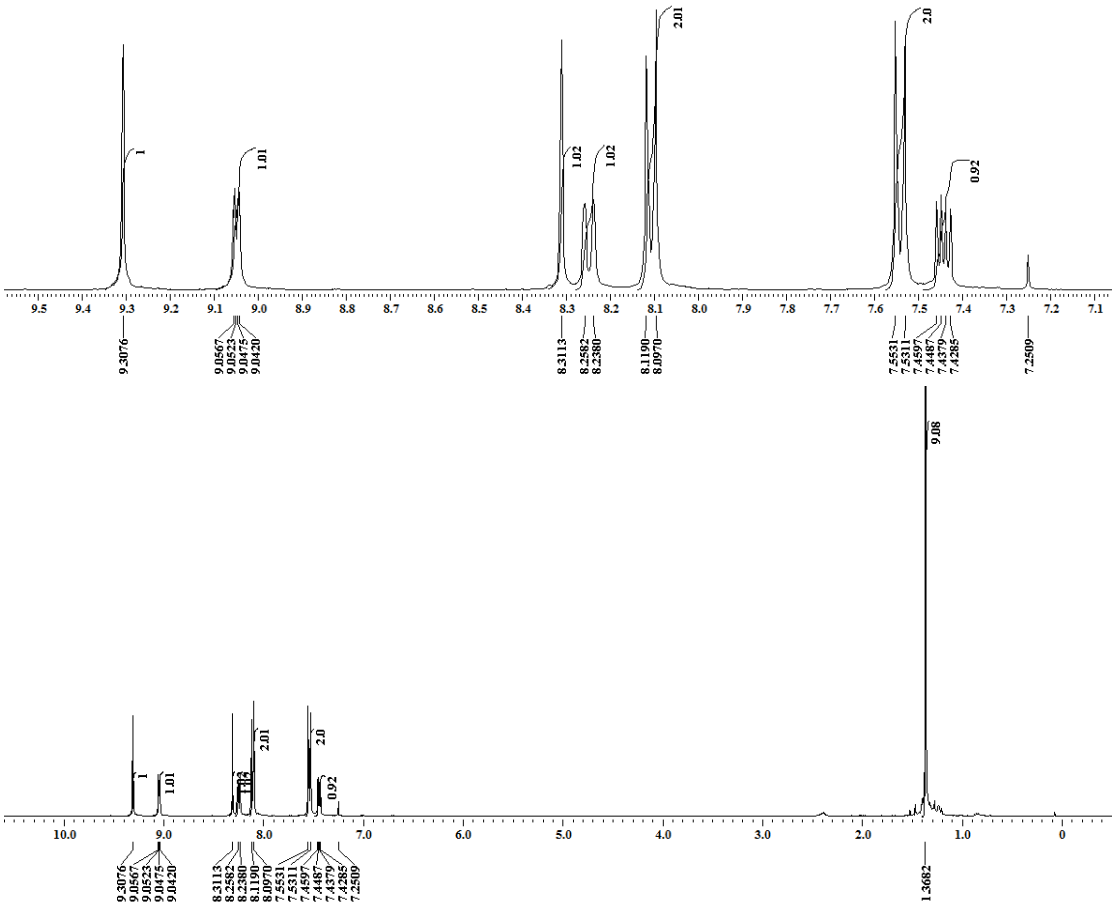
3-(4-(*t*-Butyl)phenyl)-8-methoxybenzo[*b*][1,6]naphthyridine (4h)



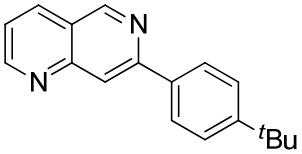
^{1}H NMR



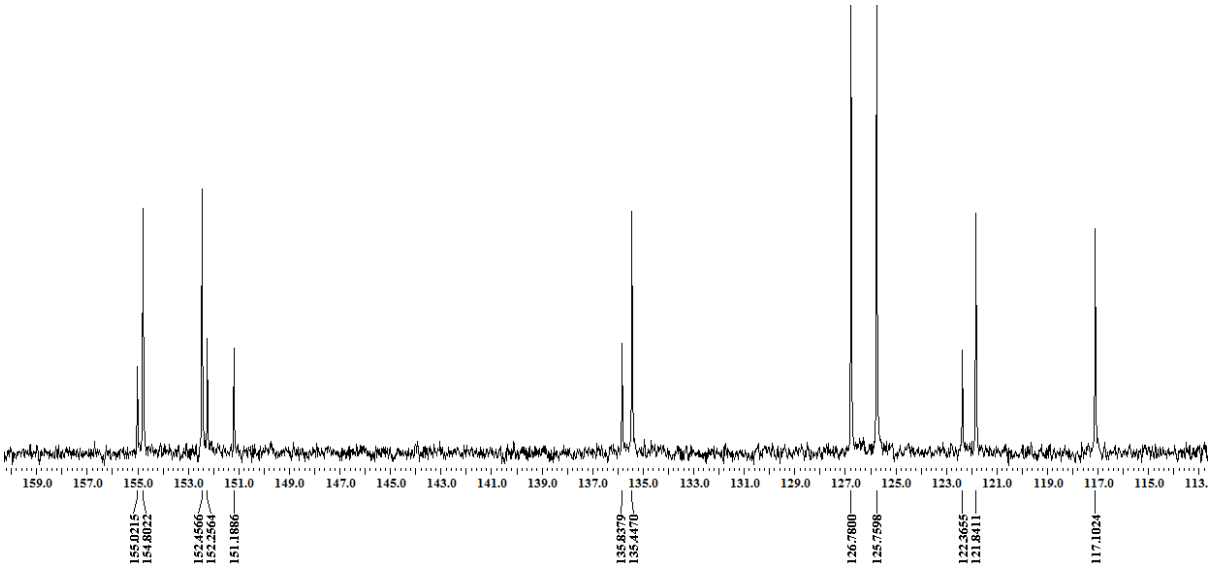
7-(4-(Tert-butyl)phenyl)-1,6-naphthyridine (4i)

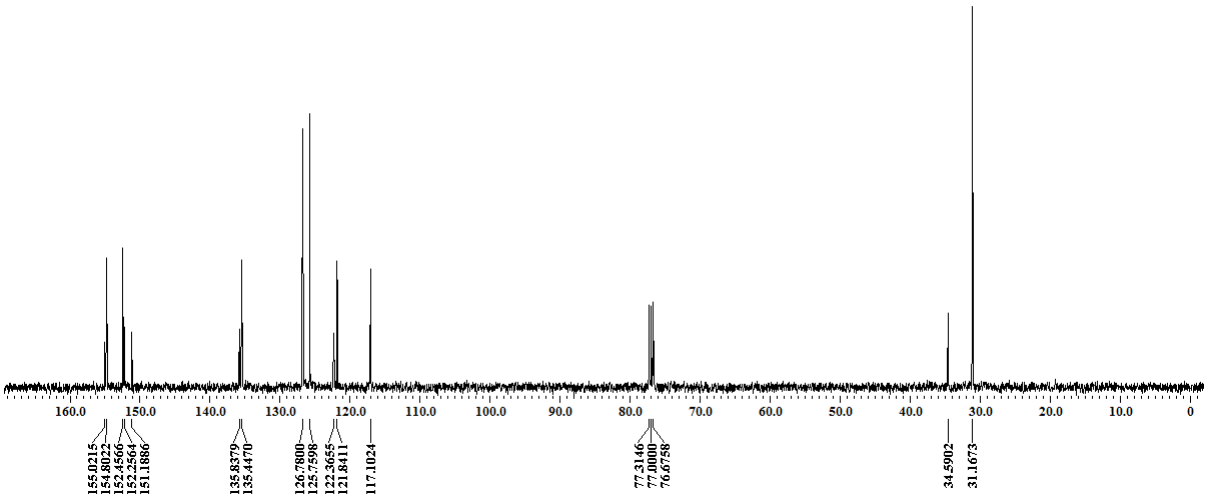


^{13}C NMR

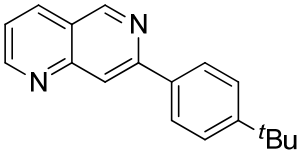


7-(4-(Tert-butyl)phenyl)-1,6-naphthyridine (4i)

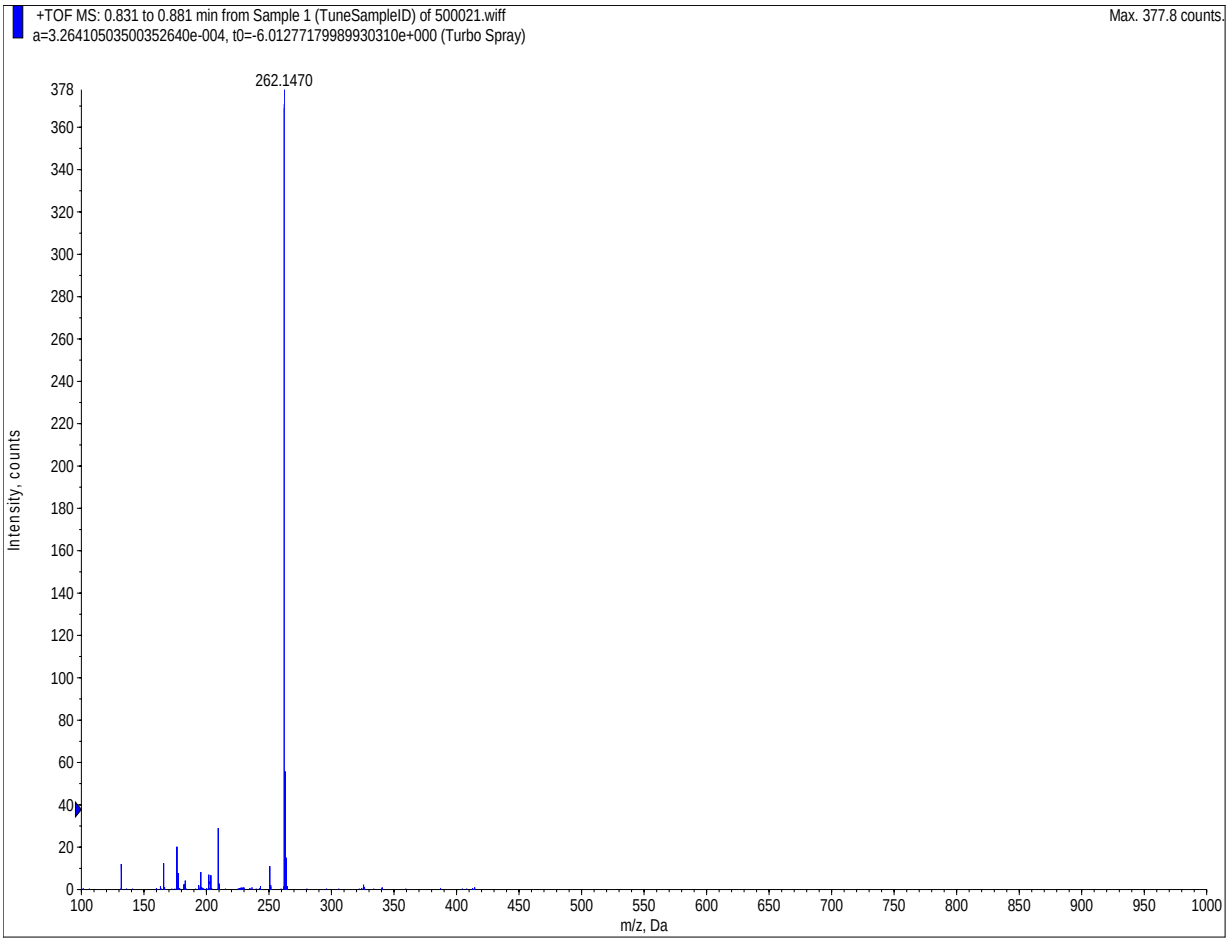




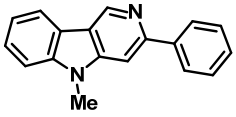
HRMS



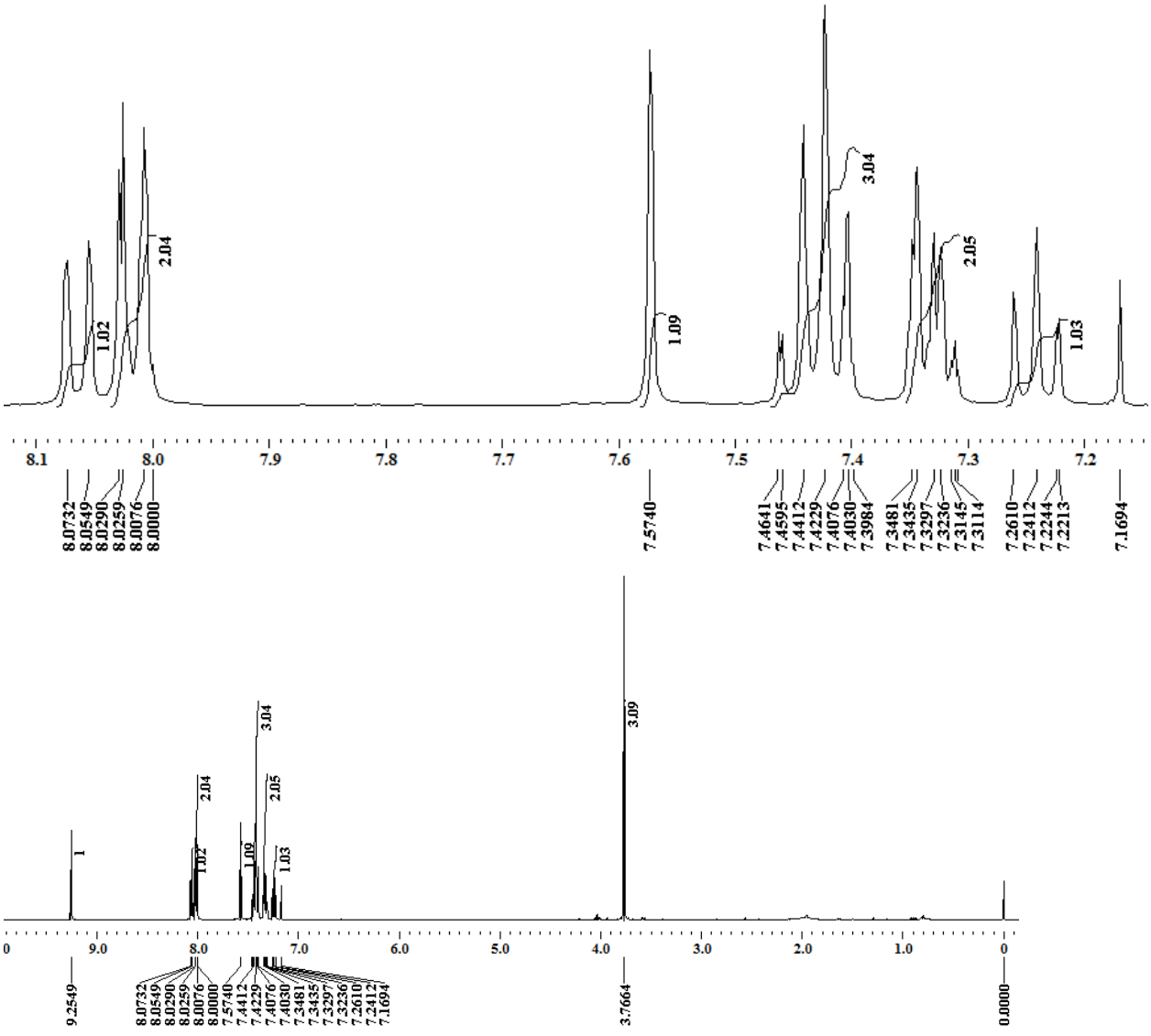
7-(4-(Tert-butyl)phenyl)-1,6-naphthyridine (4i)



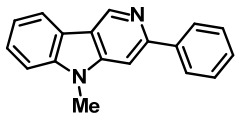
^{1}H NMR



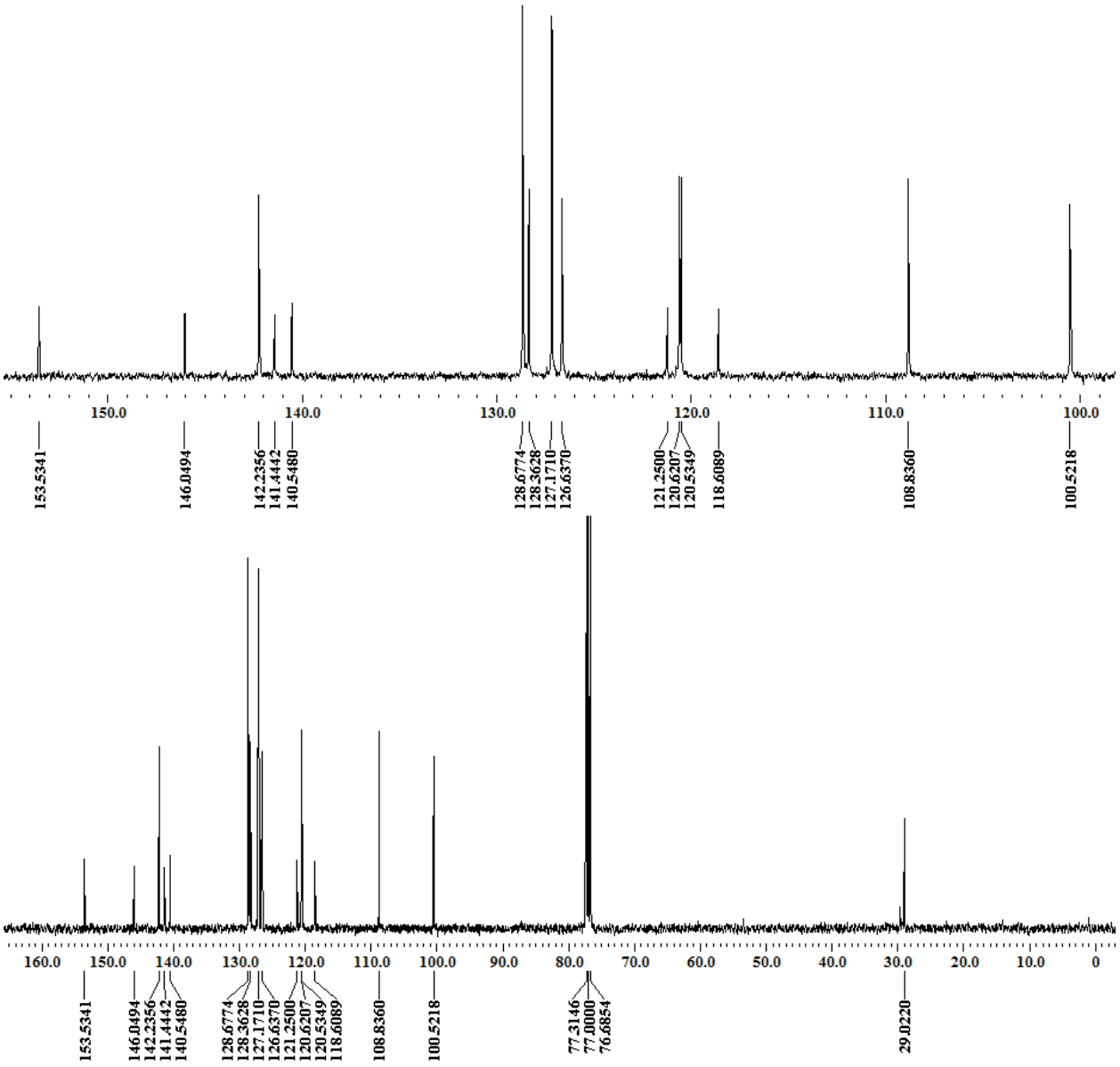
5-Methyl-3-phenyl-5*H*-pyrido[4,3-*b*]indole (6a)



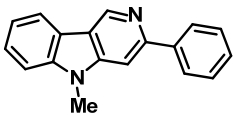
^{13}C NMR



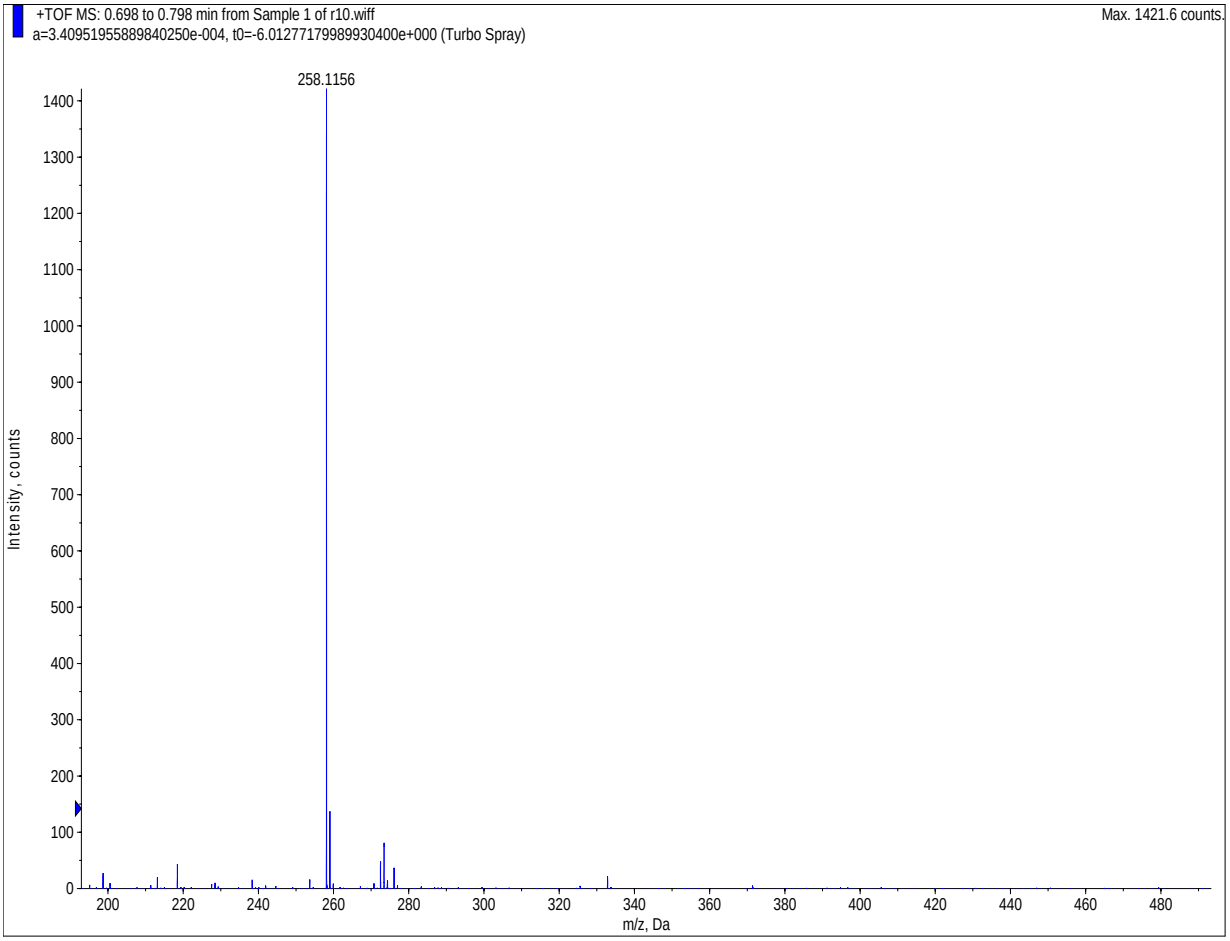
5-methyl-3-phenyl-5*H*-pyrido[4,3-*b*]indole (6a)



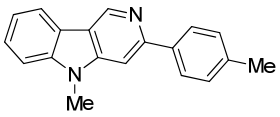
HRMS



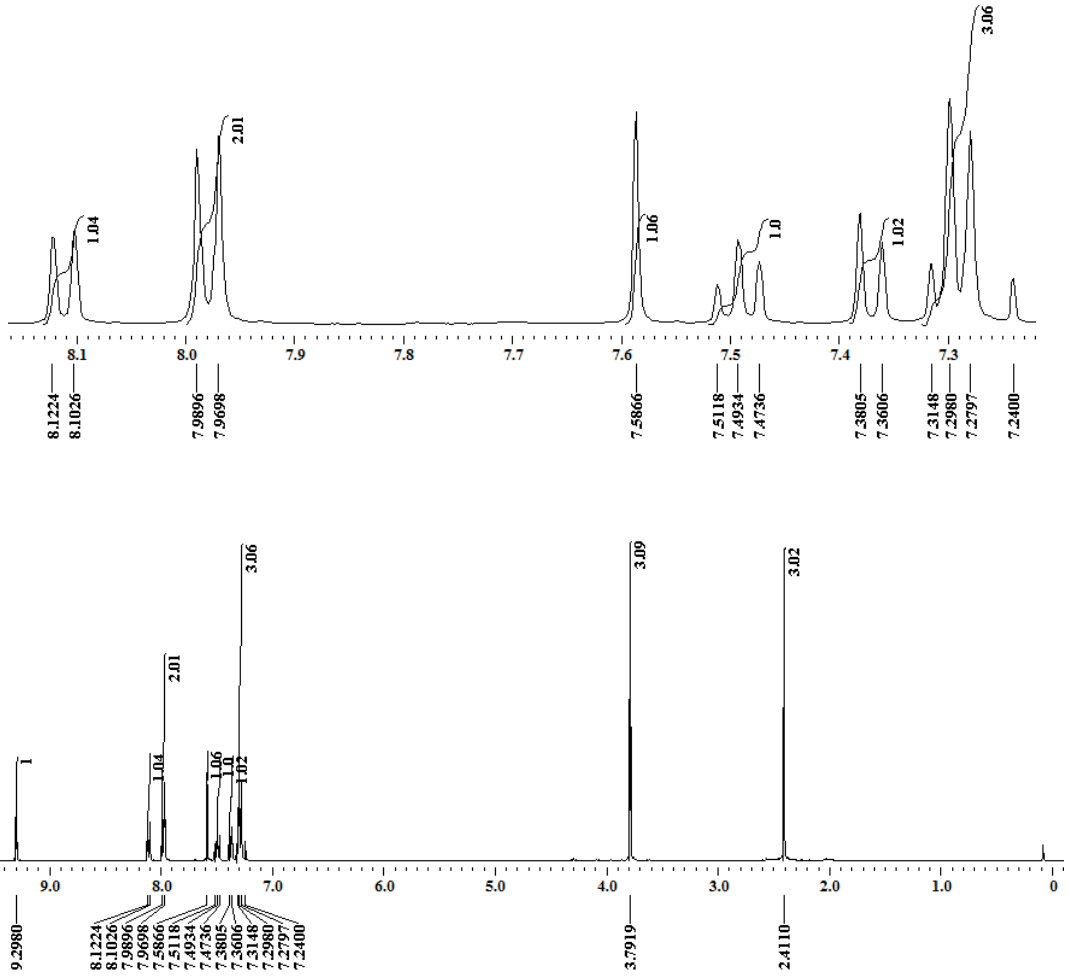
5-methyl-3-phenyl-5*H*-pyrido[4,3-*b*]indole (6a)



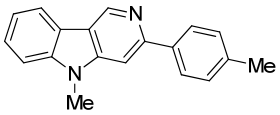
^{1}H NMR



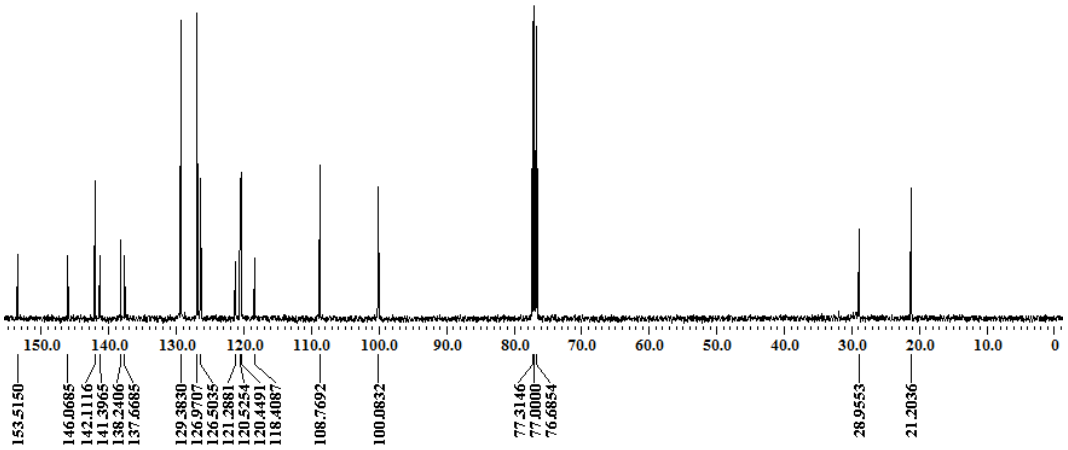
5-Methyl-3-(*p*-tolyl)-5*H*-pyrido[4,3-*b*]indole (6b)



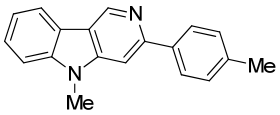
^{13}C NMR



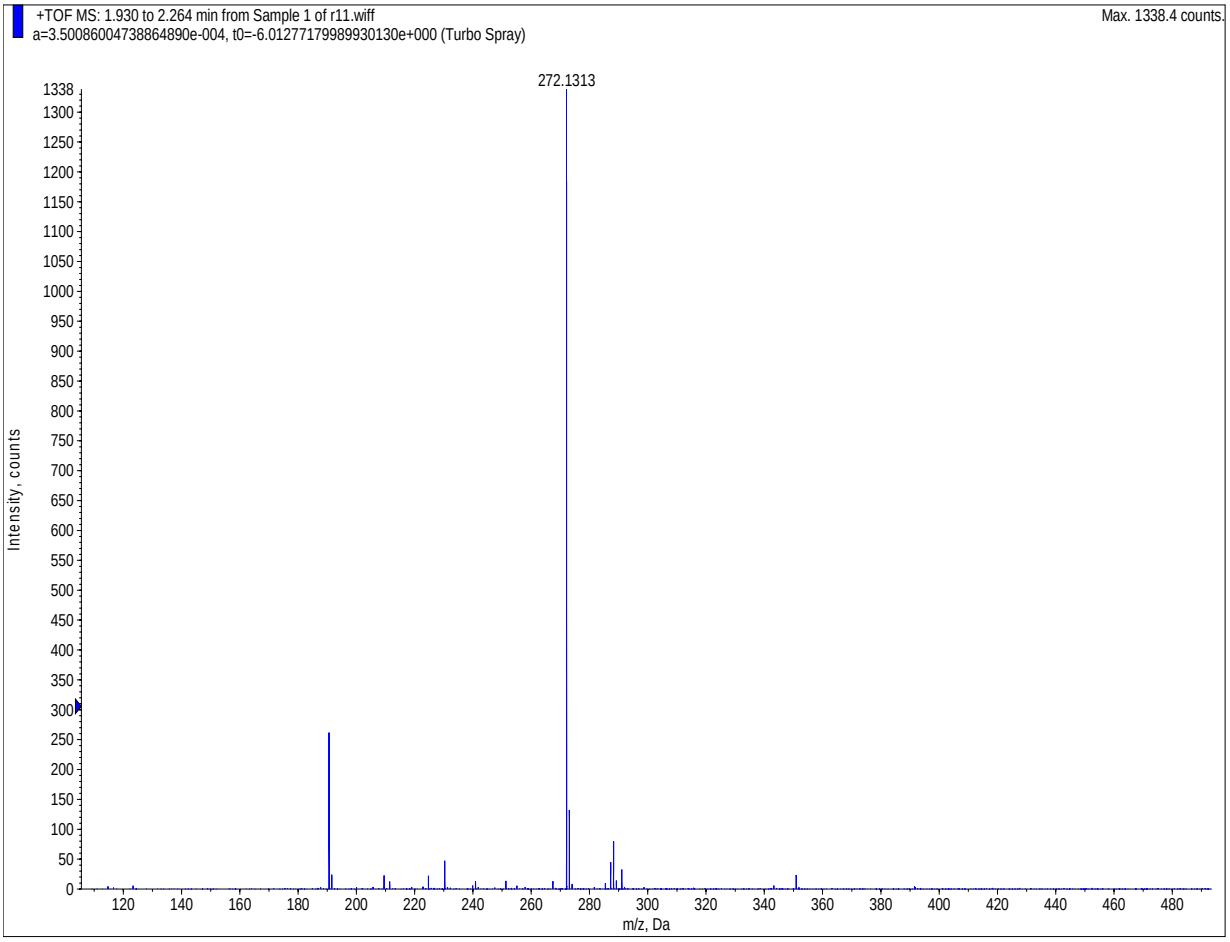
5-Methyl-3-(*p*-tolyl)-5*H*-pyrido[4,3-*b*]indole (6b)



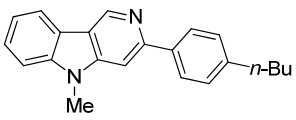
HRMS



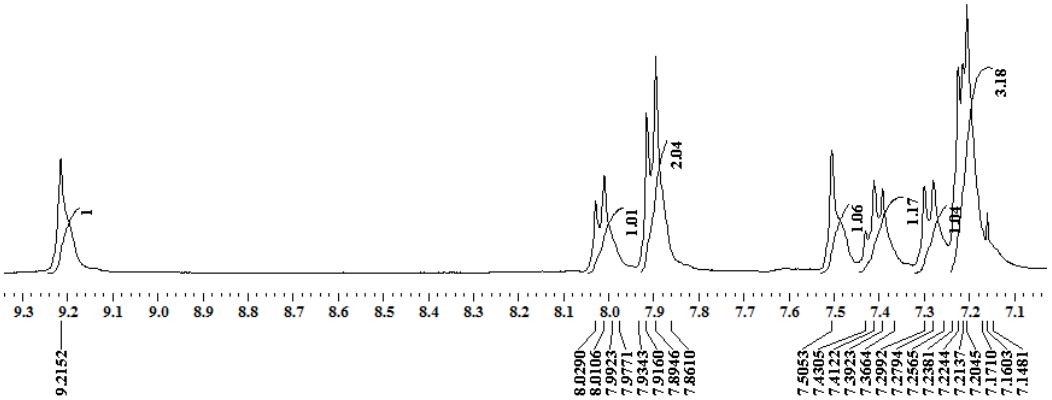
5-Methyl-3-(*p*-tolyl)-5*H*-pyrido[4,3-*b*]indole (6b)

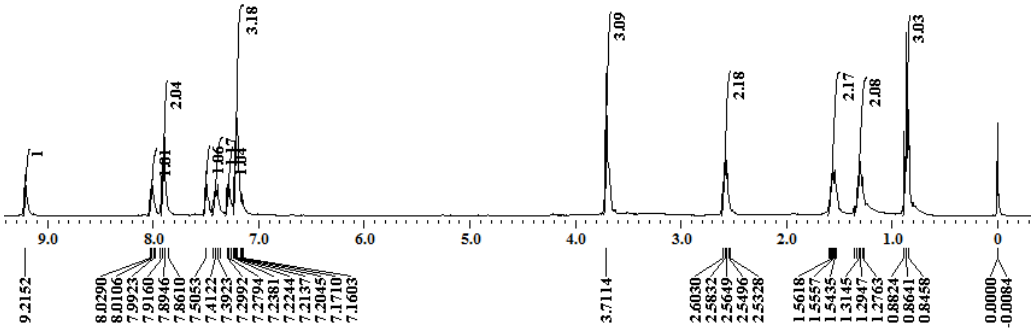


^{1}H NMR

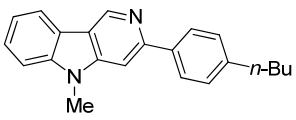


3-(4-Butylphenyl)-5-methyl-5*H*-pyrido[4,3-*b*]indole (6c)

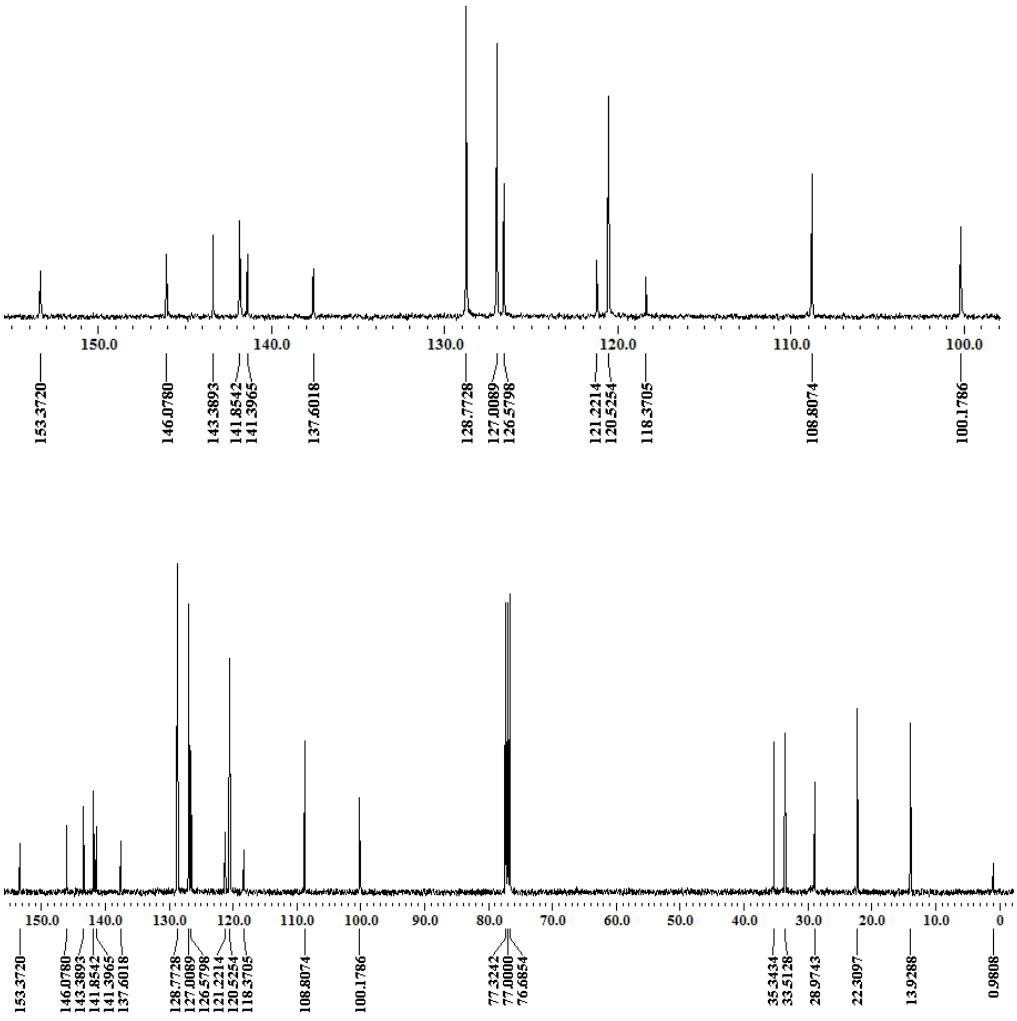




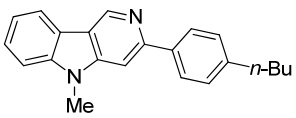
^{13}C NMR



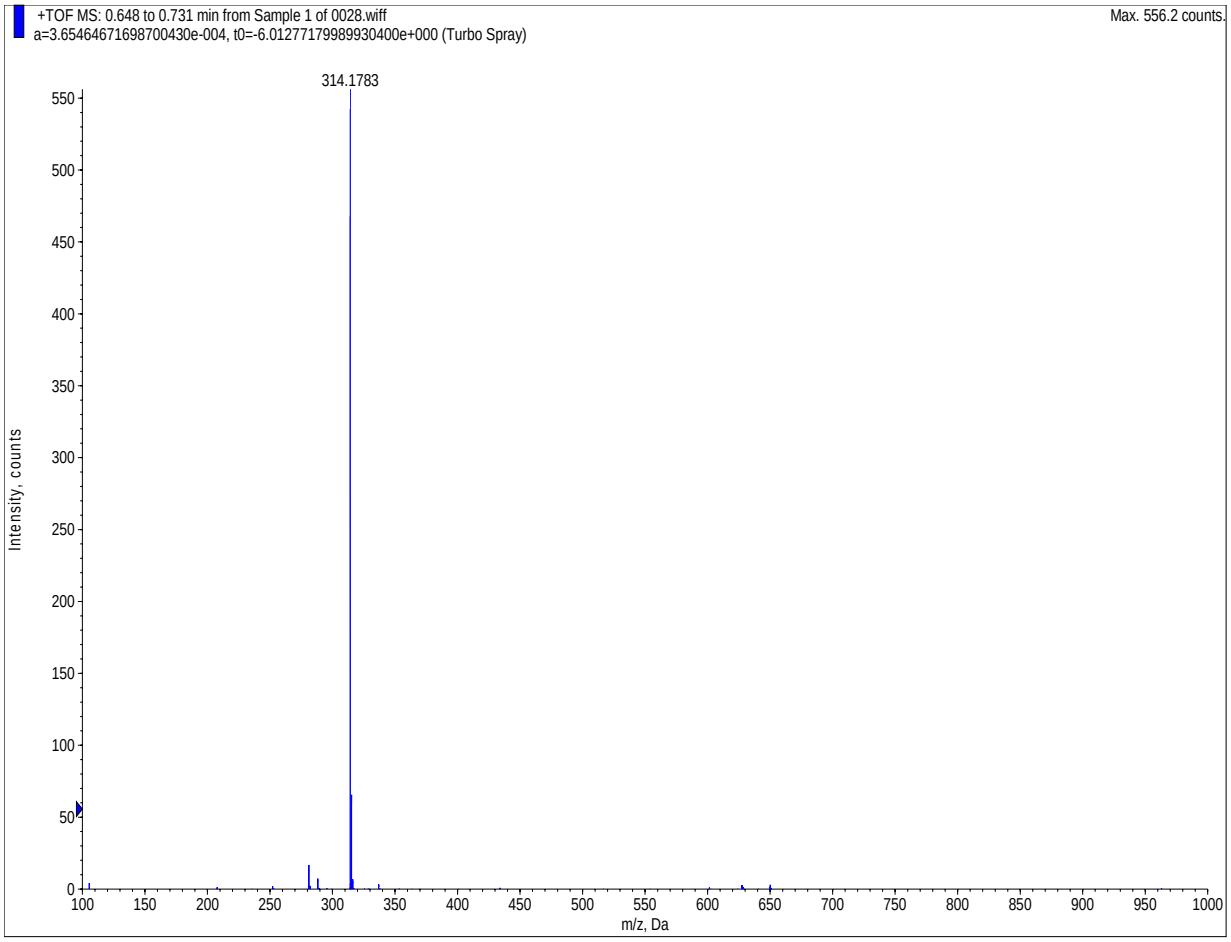
3-(4-Butylphenyl)-5-methyl-5*H*-pyrido[4,3-*b*]indole (6c)



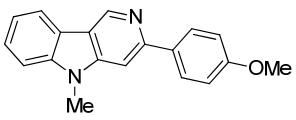
HRMS



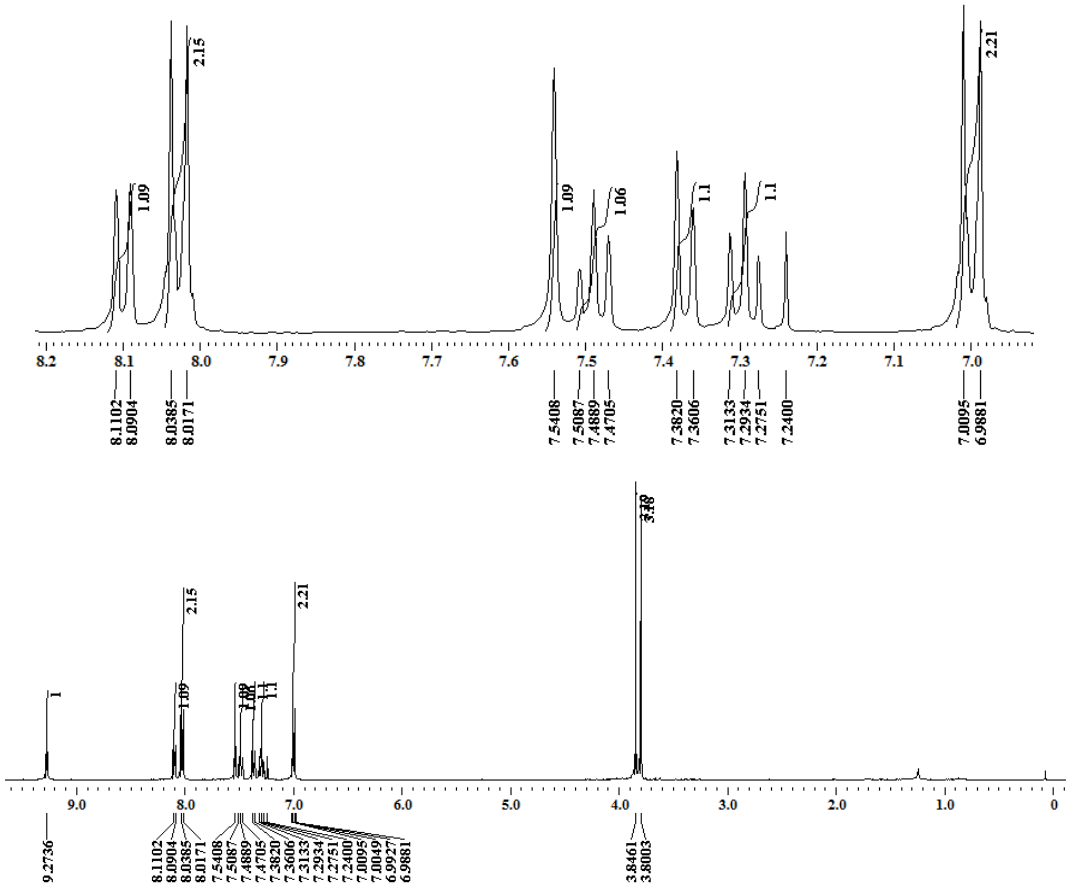
3-(4-Butylphenyl)-5-methyl-5*H*-pyrido[4,3-*b*]indole (6c)



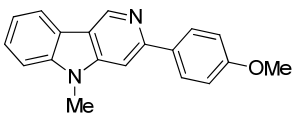
^{1}H NMR



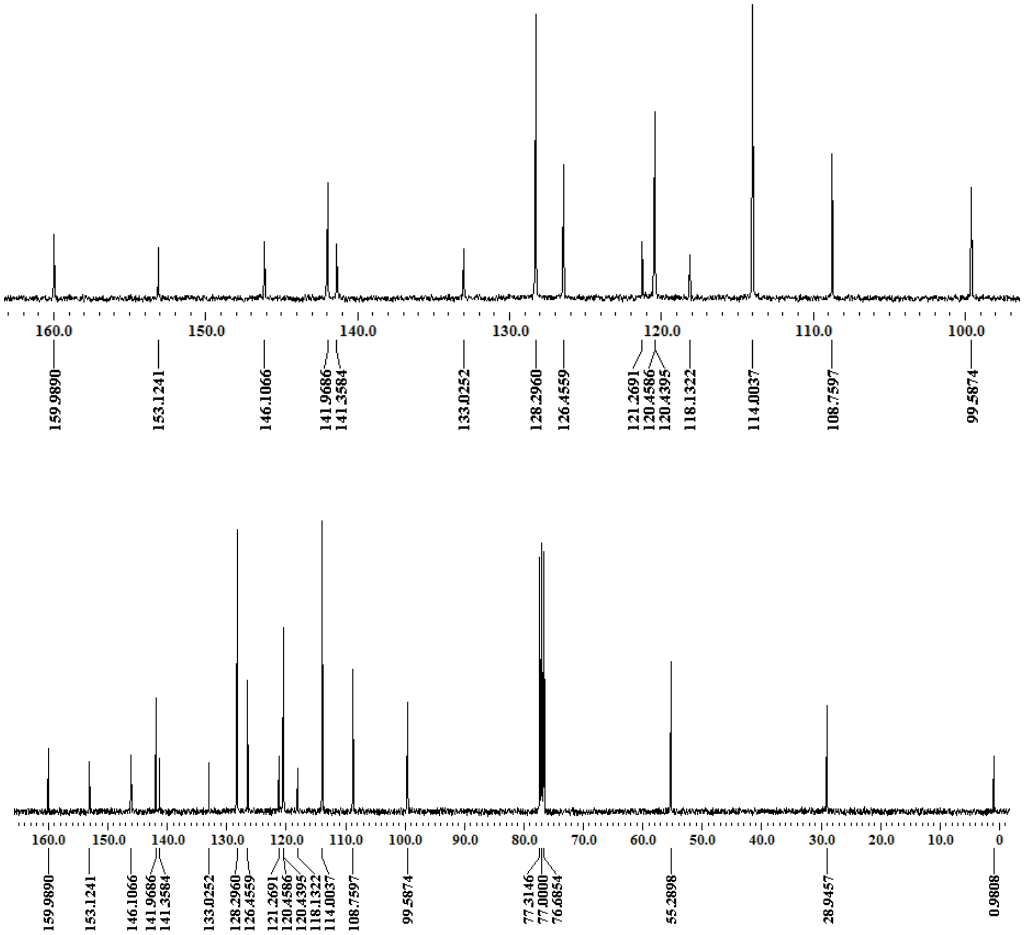
3-(4-Methoxyphenyl)-5-methyl-5*H*-pyrido[4,3-*b*]indole (6d)



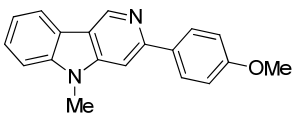
^{13}C NMR



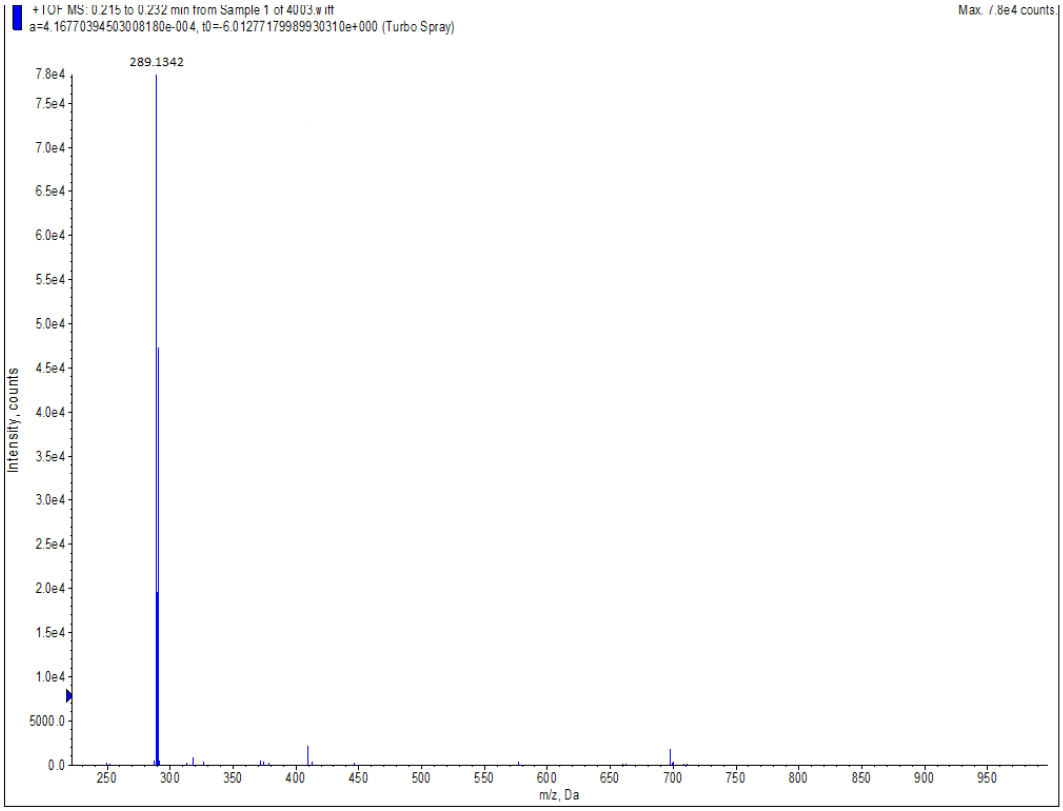
3-(4-Methoxyphenyl)-5-methyl-5*H*-pyrido[4,3-*b*]indole (6d)



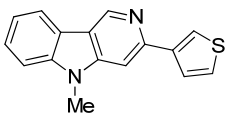
HRMS



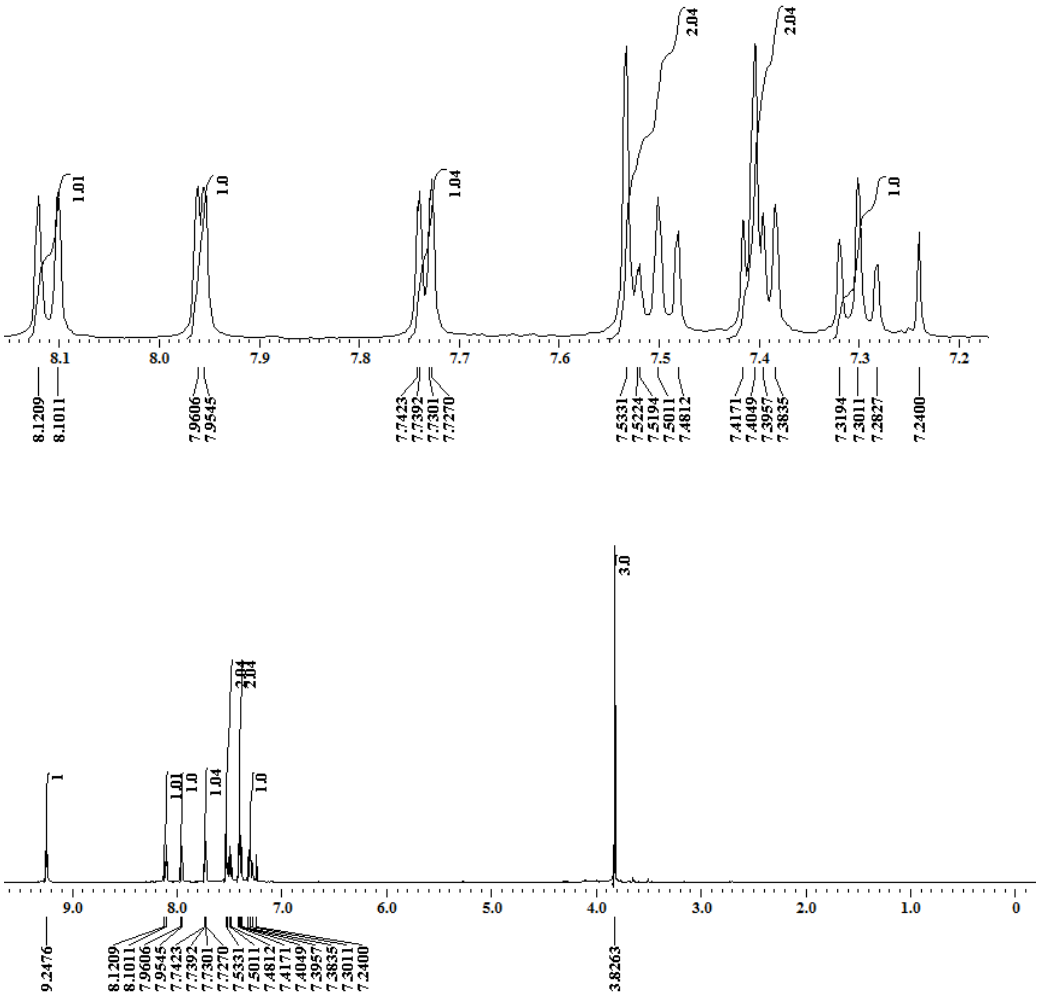
3-(4-Methoxyphenyl)-5-methyl-5*H*-pyrido[4,3-*b*]indole (6d)



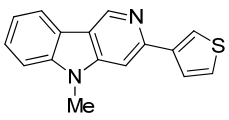
^{1}H NMR



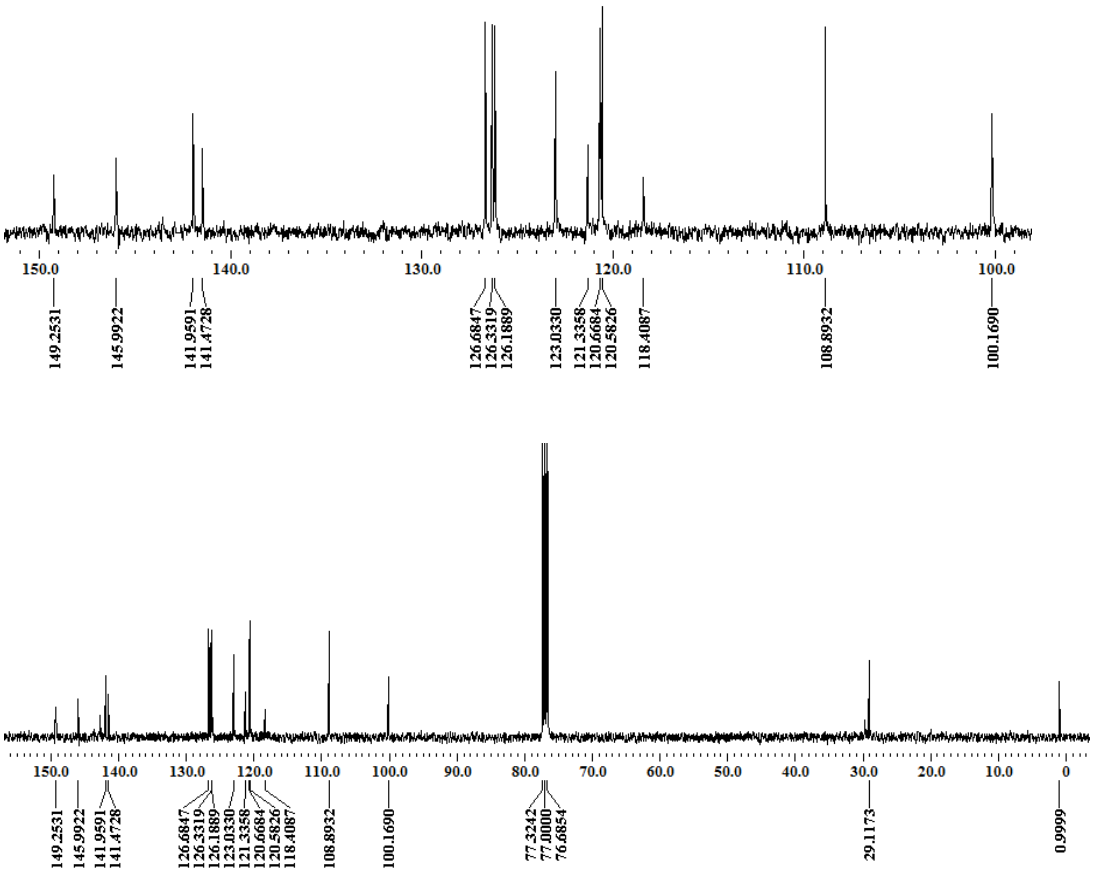
5-Methyl-3-(thiophen-3-yl)-5*H*-pyrido[4,3-*b*]indole (6e)



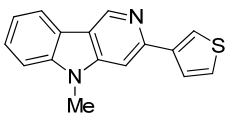
^{13}C NMR



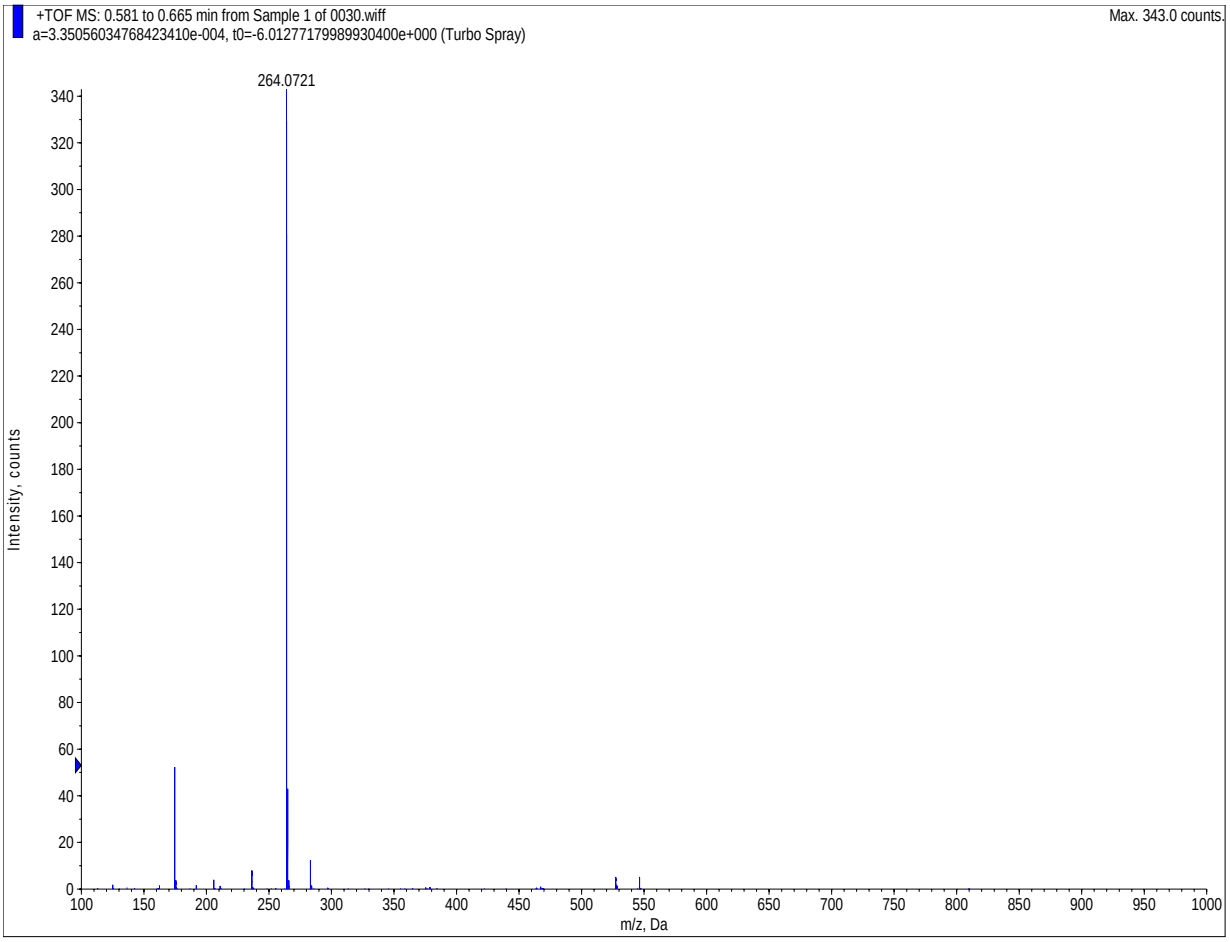
5-Methyl-3-(thiophen-3-yl)-5*H*-pyrido[4,3-*b*]indole (6e)



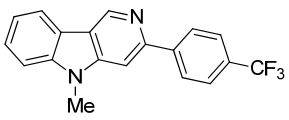
HRMS



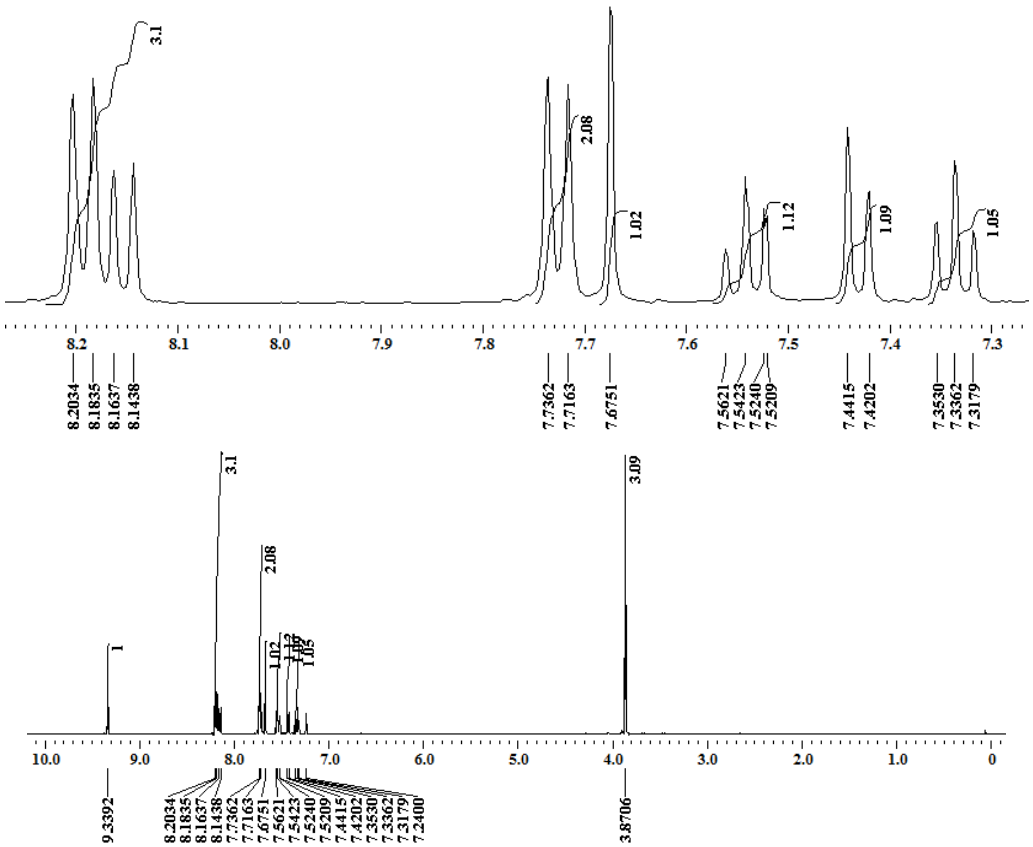
5-Methyl-3-(thiophen-3-yl)-5*H*-pyrido[4,3-*b*]indole (6e)



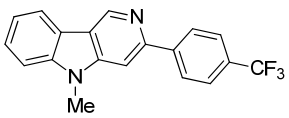
^{1}H NMR



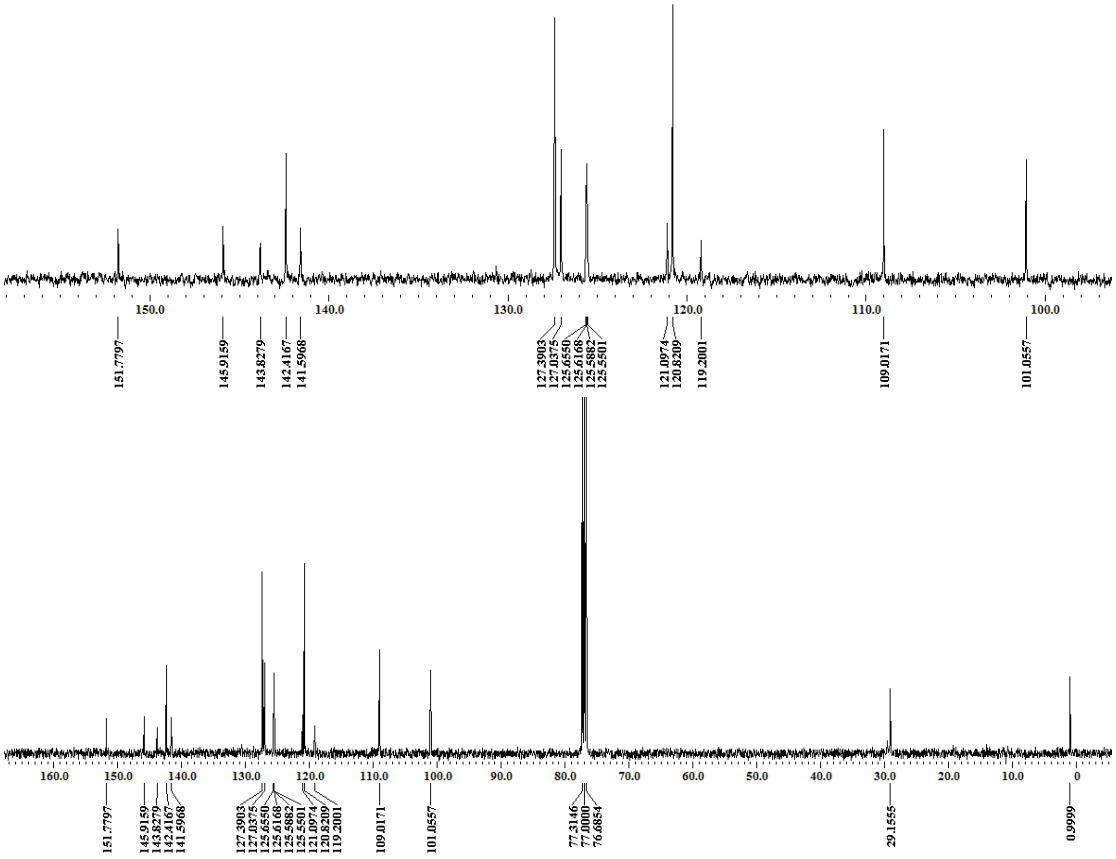
5-Methyl-3-(4-(trifluoromethyl)phenyl)-5*H*-pyrido[4,3-*b*]indole (6f)



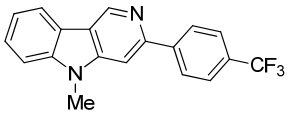
^{13}C NMR



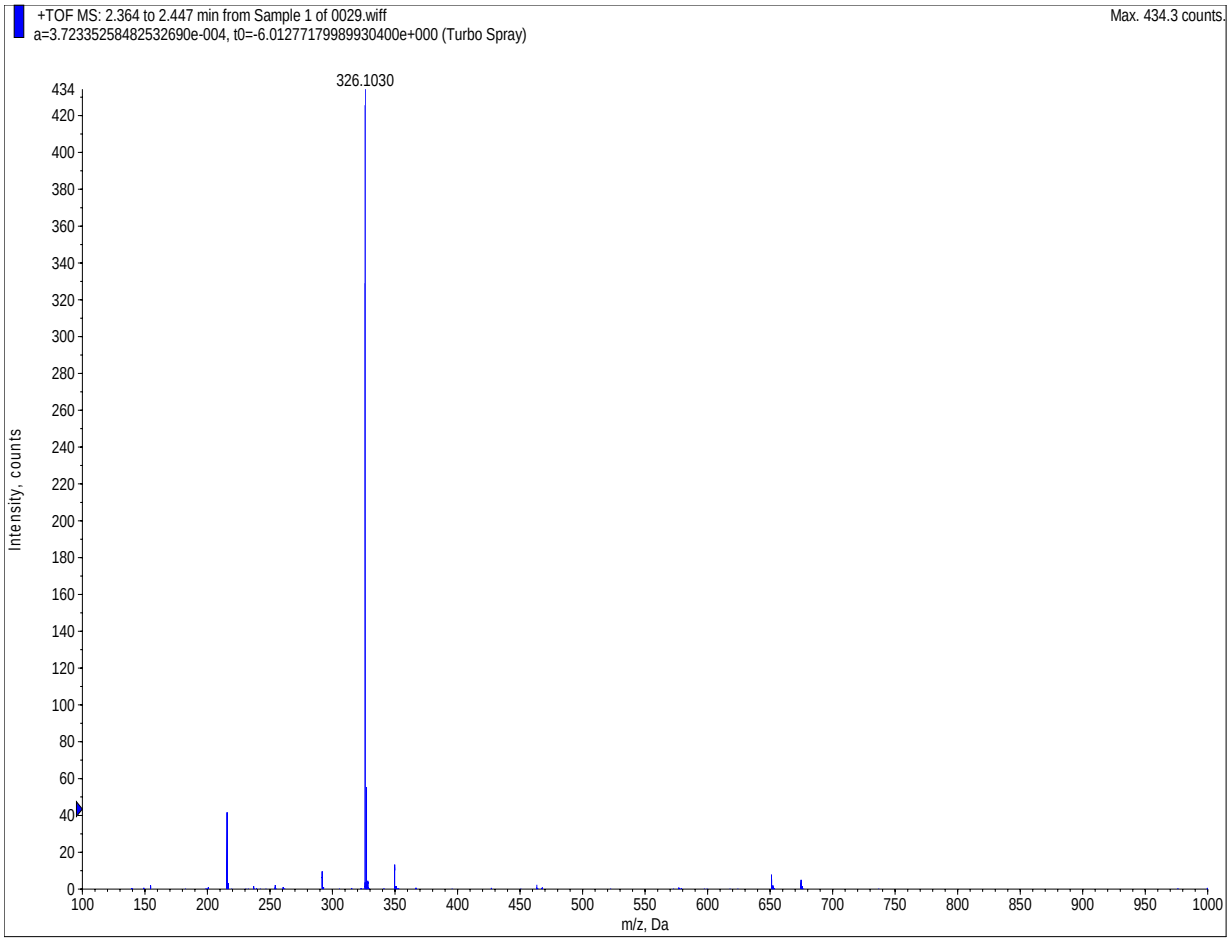
5-Methyl-3-(4-(trifluoromethyl)phenyl)-5*H*-pyrido[4,3-*b*]indole (6f)



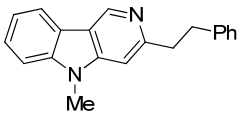
HRMS



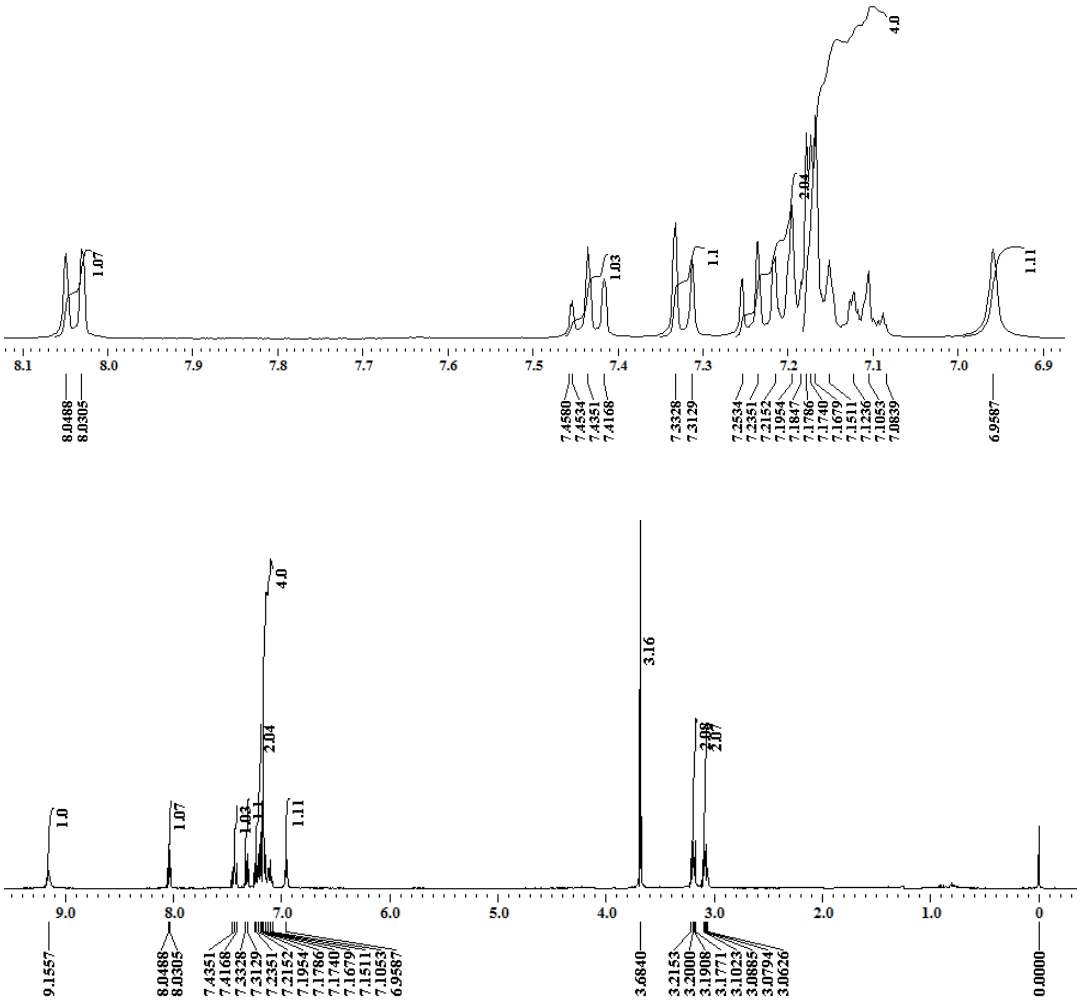
5-Methyl-3-(4-(trifluoromethyl)phenyl)-5*H*-pyrido[4,3-*b*]indole (6f)



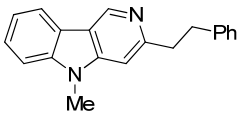
^{1}H NMR



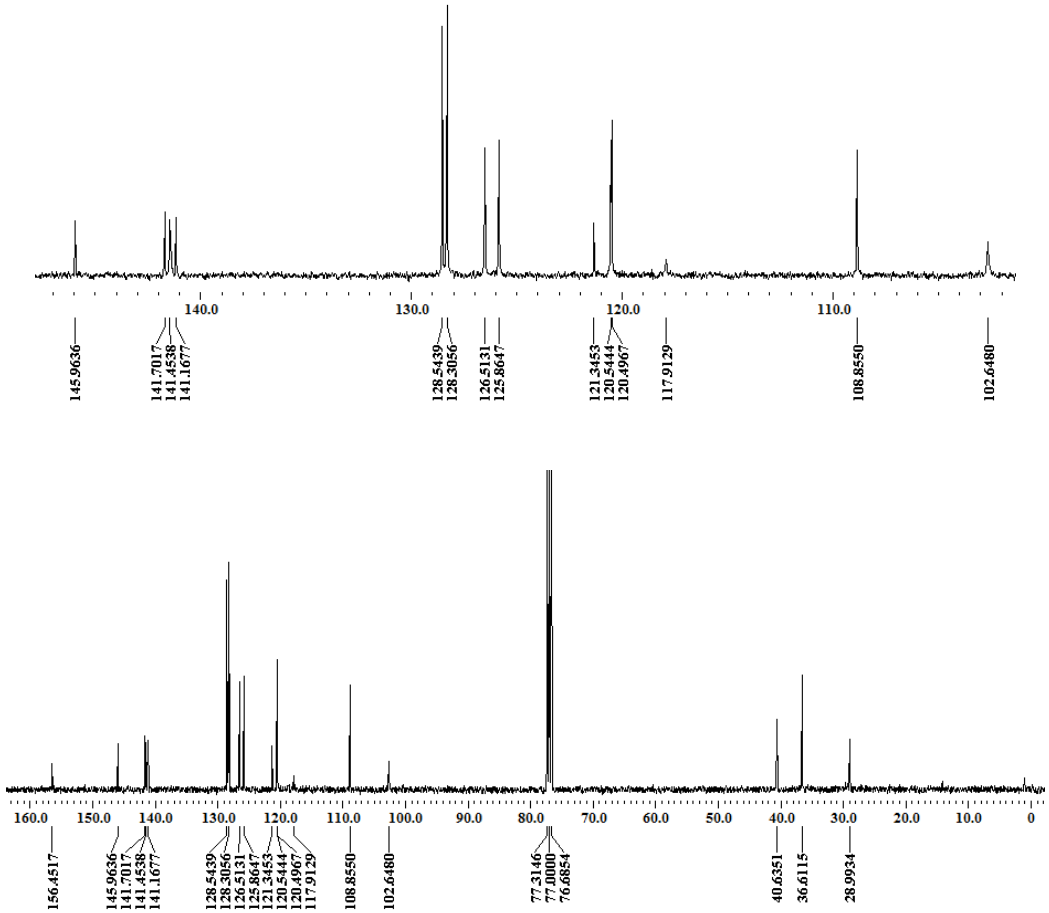
5-Methyl-3-phenethyl-5*H*-pyrido[4,3-*b*]indole (6g)



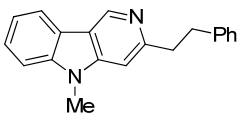
^{13}C NMR



5-Methyl-3-phenethyl-5*H*-pyrido[4,3-*b*]indole (6g)



HRMS



5-Methyl-3-phenethyl-5*H*-pyrido[4,3-*b*]indole (6g)

Qualitative Compound Report

Data File	CCH-14.d	**Sample Name**	CCH-14
Sample Type	Sample	**Position**	P1-C2
Instrument Name	Instrument 1	**User Name**	SMILY
Acq Method	29.10.2014.m	**Acquired Time**	21-03-2016 15:24:26
IRM Calibration Status	Success	**DA Method**	Default.m
Comment			

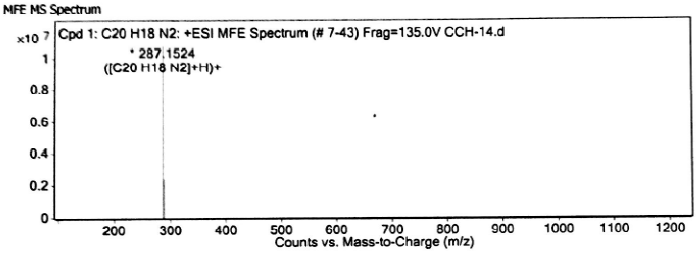
Sample Group	**Info.**
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)

Compound Table

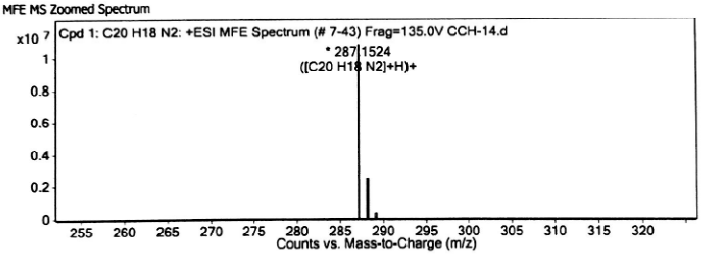
Compound Label	RT	Mass	Formula	MFG Formula	MFG Diff (ppm)	DB Formula
Cpd 1: C20 H18 N2	11	286.1451	C20 H18 N2	C20 H18 N2	6.53	C20 H18 N2

Compound Label	*m/z*	RT	Algorithm	Mass
Cpd 1: C20 H18 N2	287.1524	11	Find by Molecular Feature	286.1451

MFE MS Spectrum



MFE MS Zoomed Spectrum

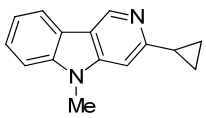


MS Spectrum Peak List

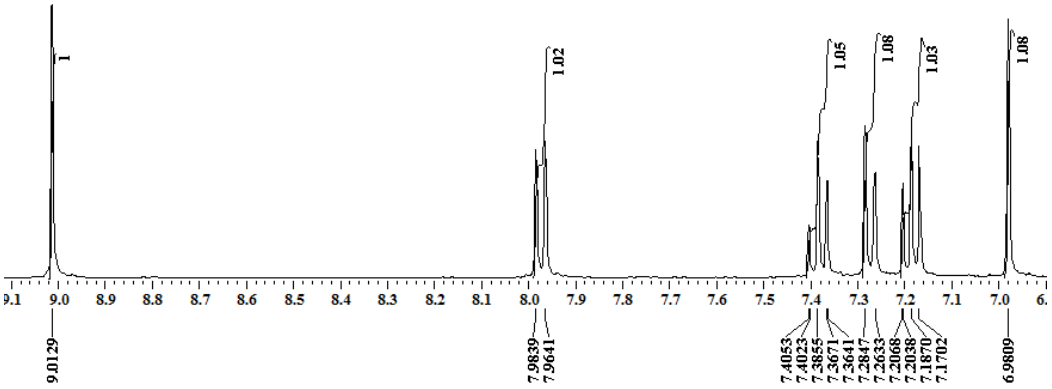
m/z	z	Abund	Formula	Ion
287.1524	1	10766272	C20 H18 N2	(M+H)+
288.1558	1	2399926.84	C20 H18 N2	(M+H)+
289.1588	1	246813.7	C20 H18 N2	(M+H)+
290.1615	1	18230.1	C20 H18 N2	(M+H)+
291.1564	1	2226.24	C20 H18 N2	(M+H)+

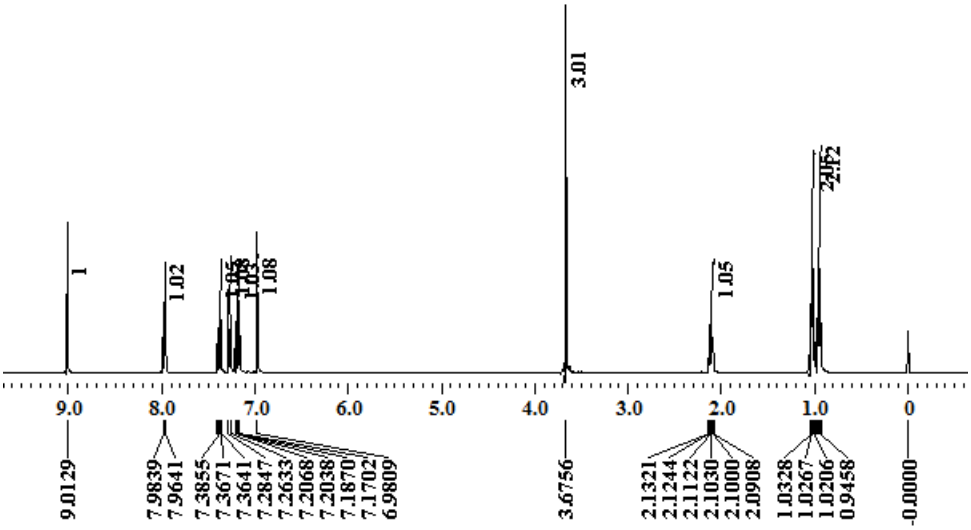
--- End Of Report ---

^{1}H NMR

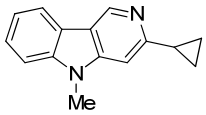


3-Cyclopropyl-5-methyl-5*H*-pyrido[4,3-*b*]indole (6h)

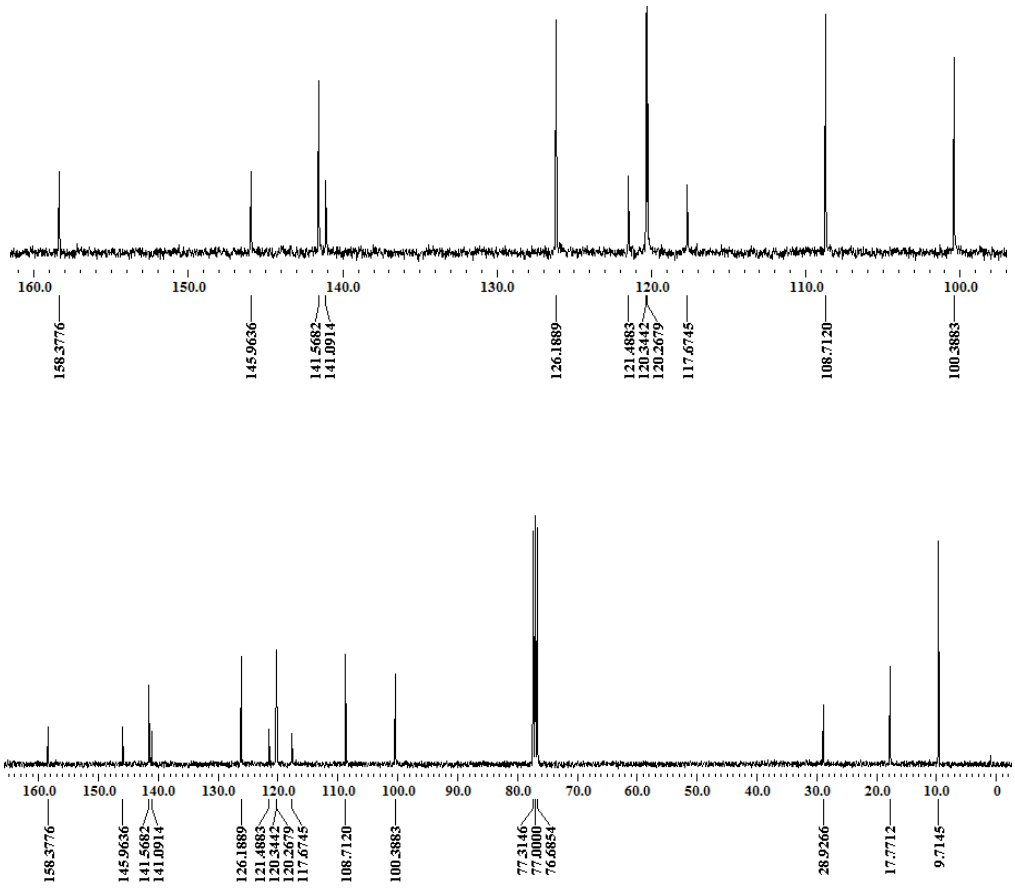




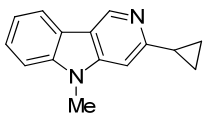
^{13}C NMR



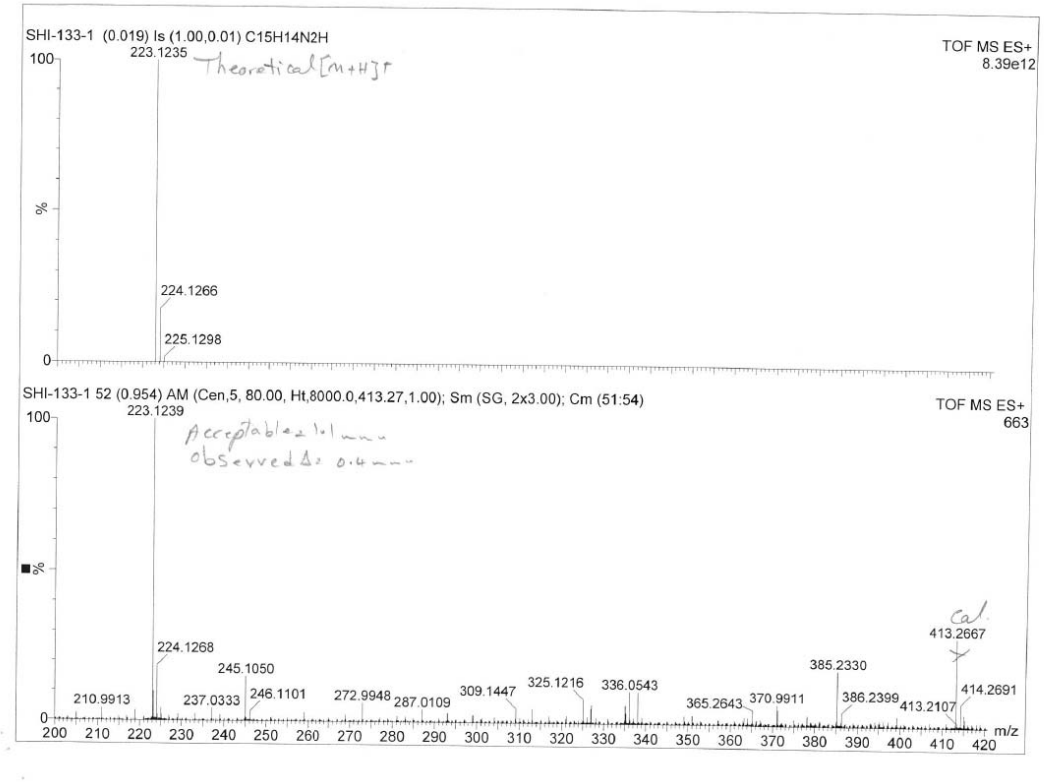
3-Cyclopropyl-5-methyl-5*H*-pyrido[4,3-*b*]indole (6h)



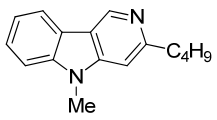
HRMS



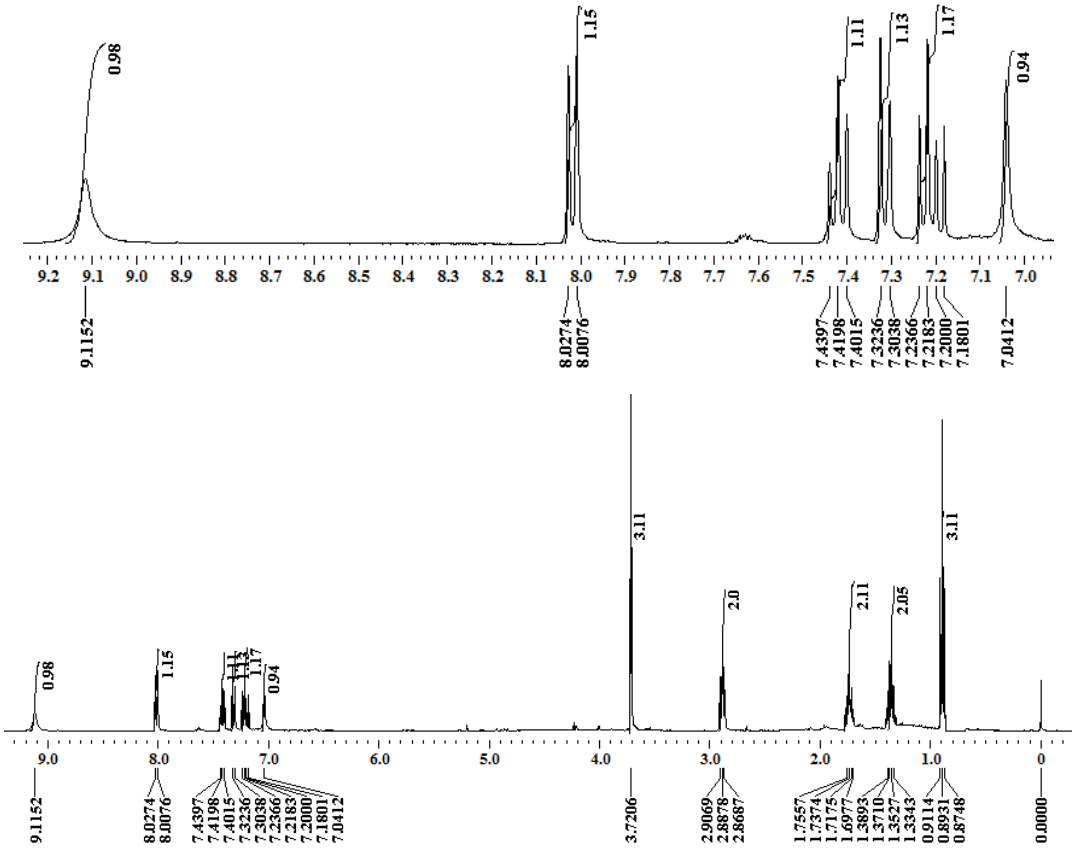
3-Cyclopropyl-5-methyl-5*H*-pyrido[4,3-*b*]indole (6h)



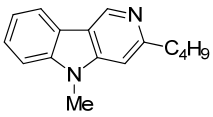
^{1}H NMR



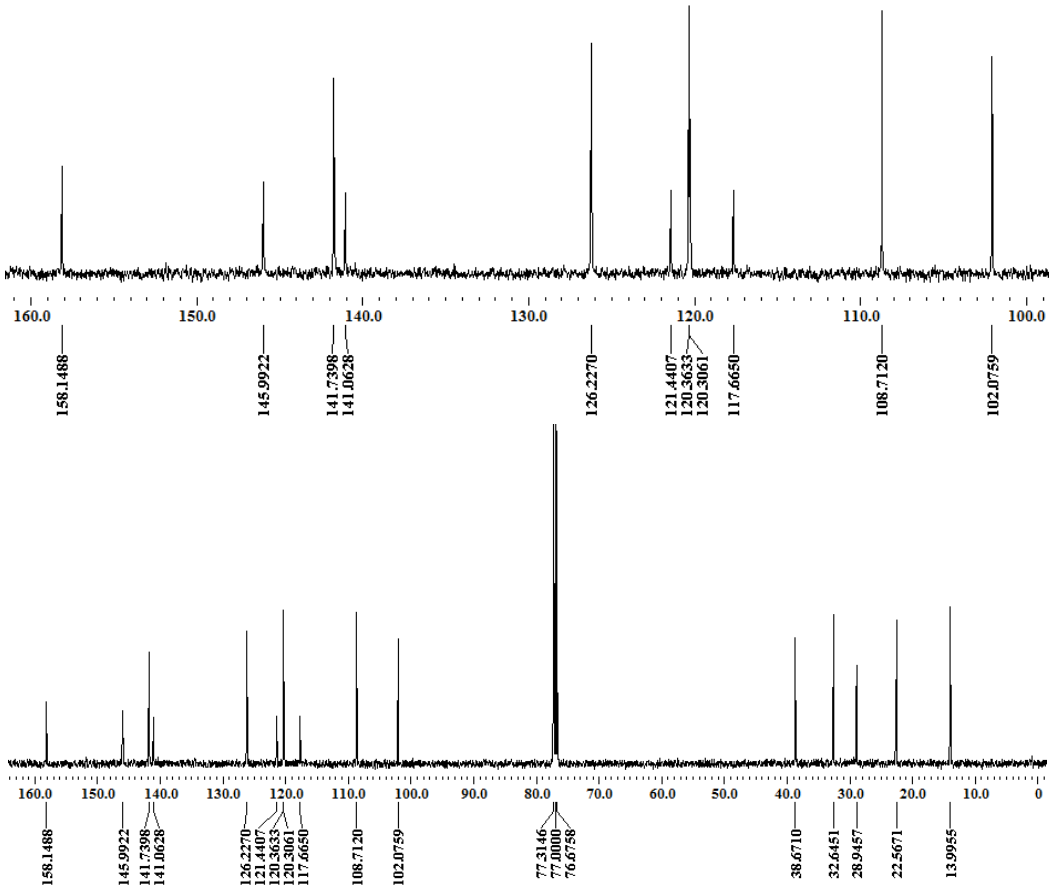
3-Butyl-5-methyl-5*H*-pyrido[4,3-*b*]indole (6i)



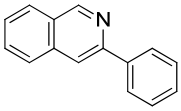
^{13}C NMR



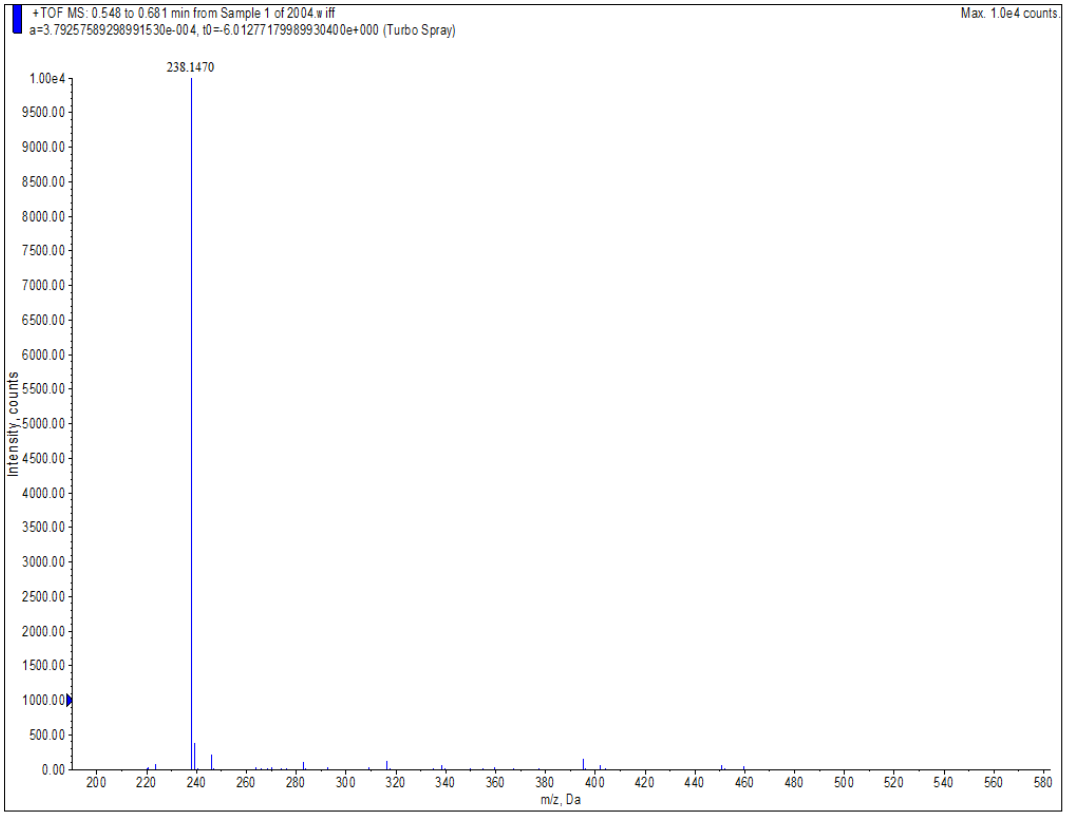
3-Butyl-5-methyl-5*H*-pyrido[4,3-*b*]indole (6i)



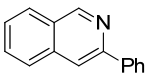
HRMS



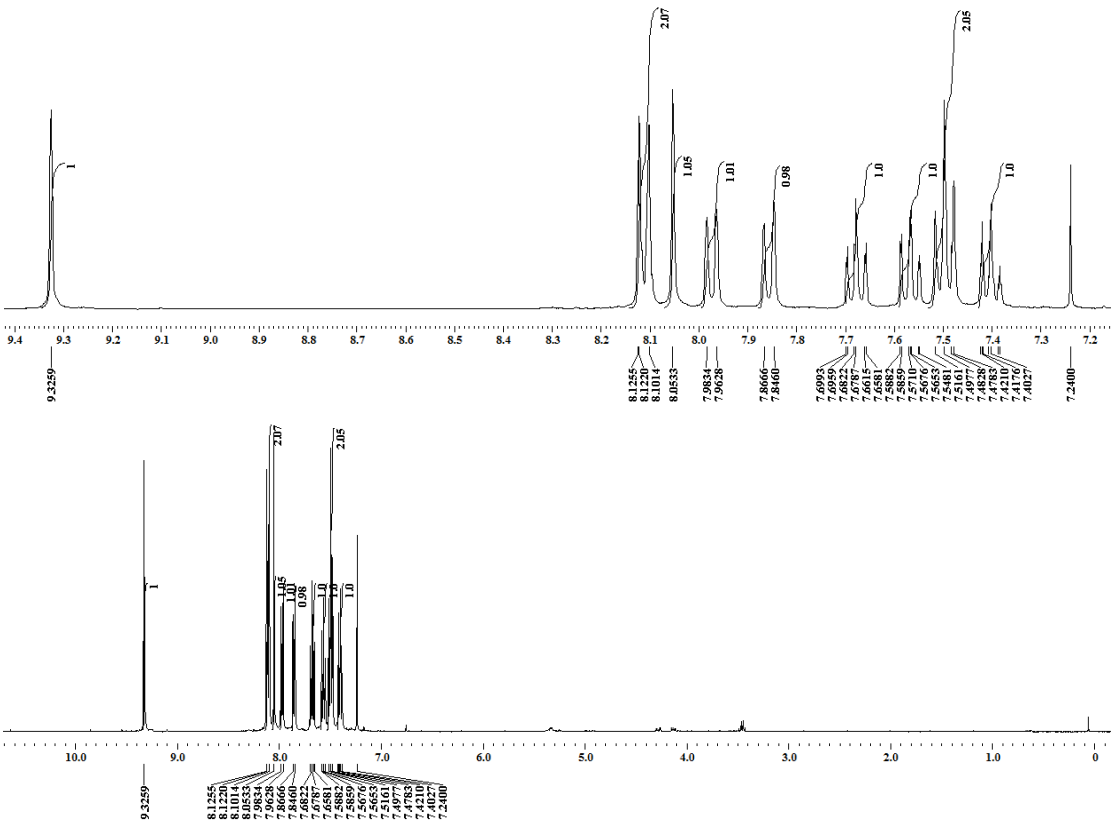
3-Butyl-5-methyl-5*H*-pyrido[4,3-*b*]indole (6i)



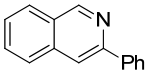
^{1}H NMR



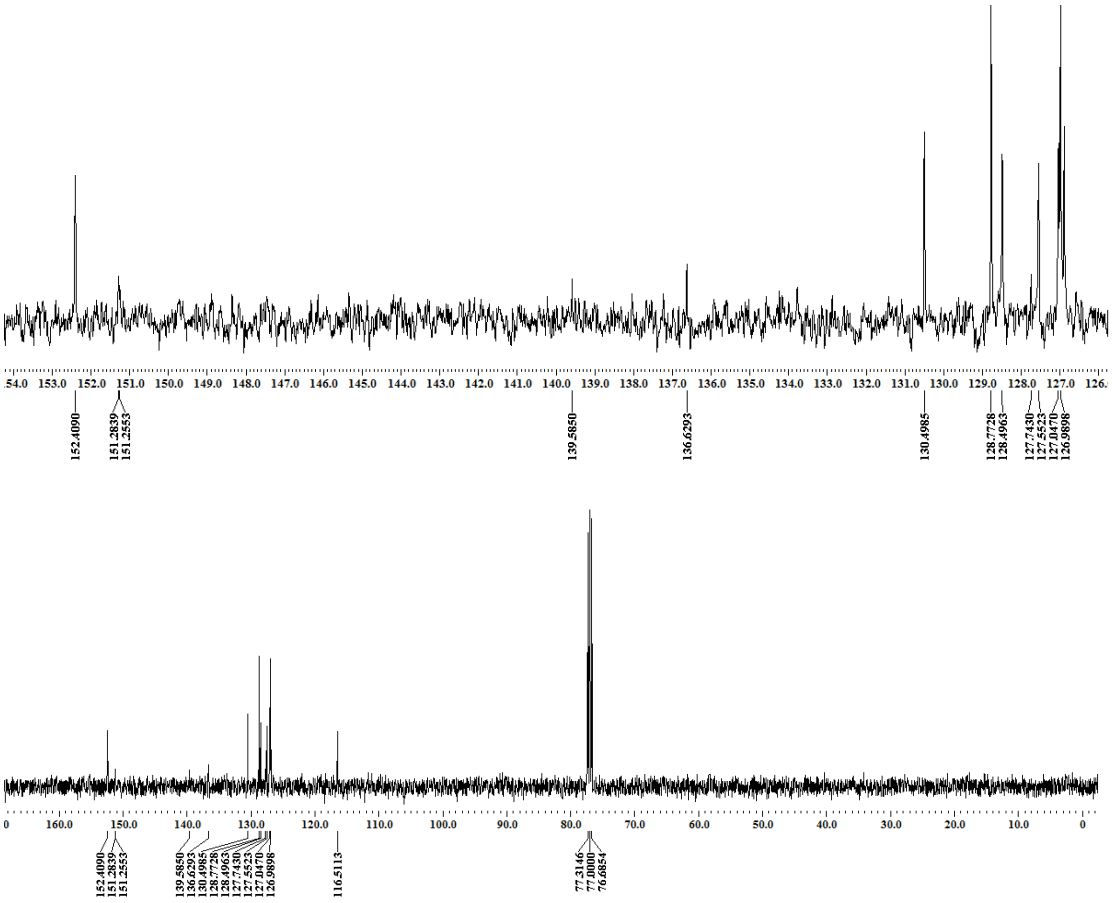
3-Phenylisoquinoline (8a)



^{13}C NMR



3-Phenylisoquinoline (8a)



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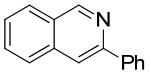
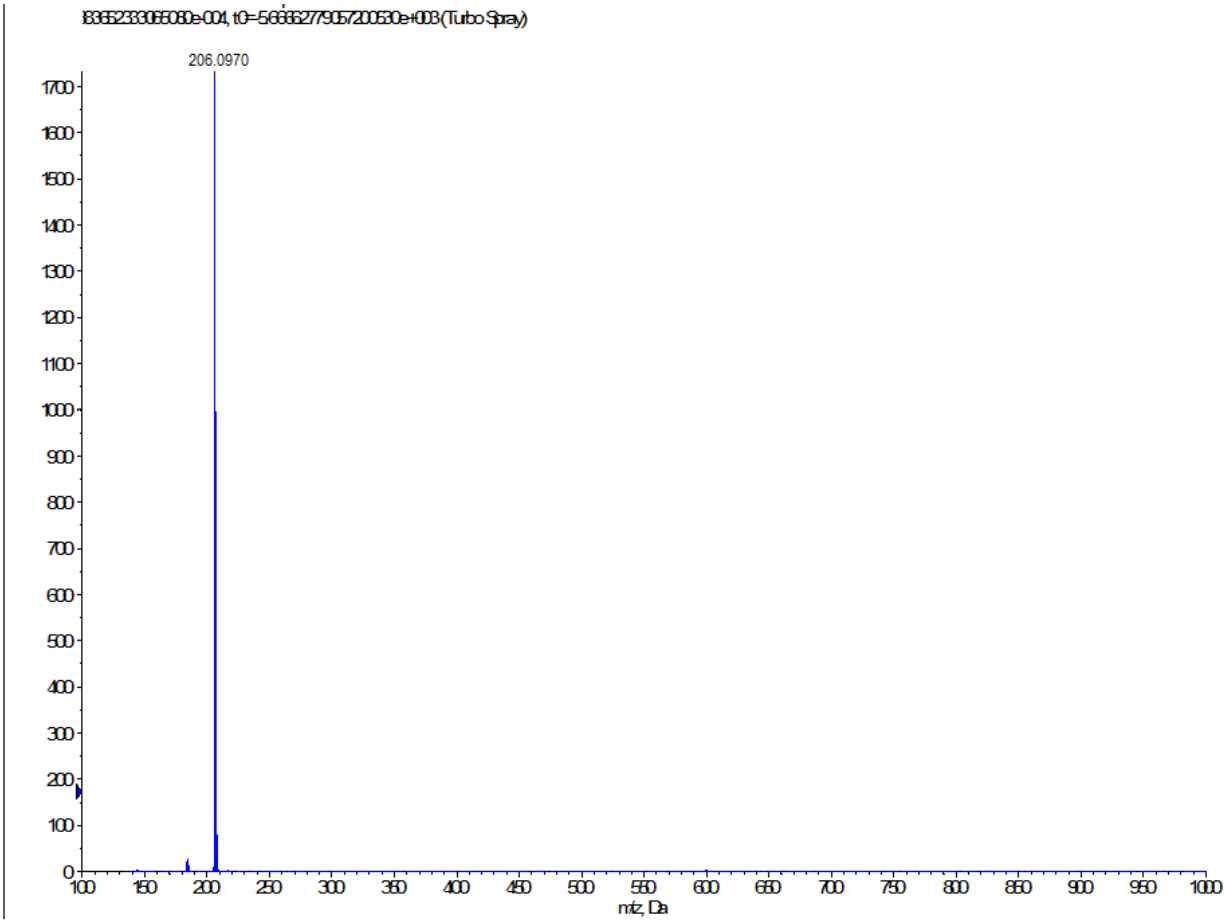
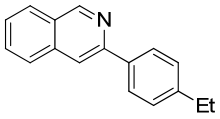


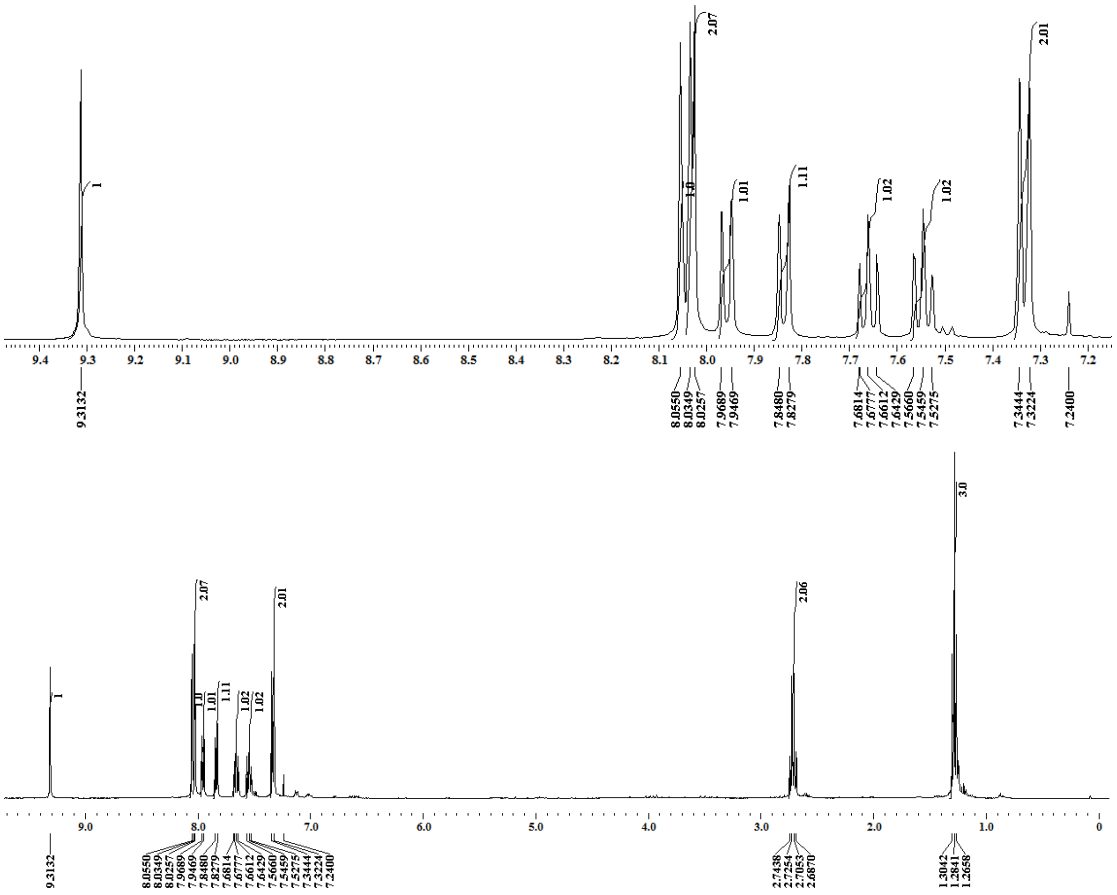
3-Phenylisoquinoline (8a)



^{1}H NMR



3-(4-Ethylphenyl)isoquinoline (8b)



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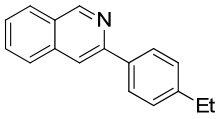
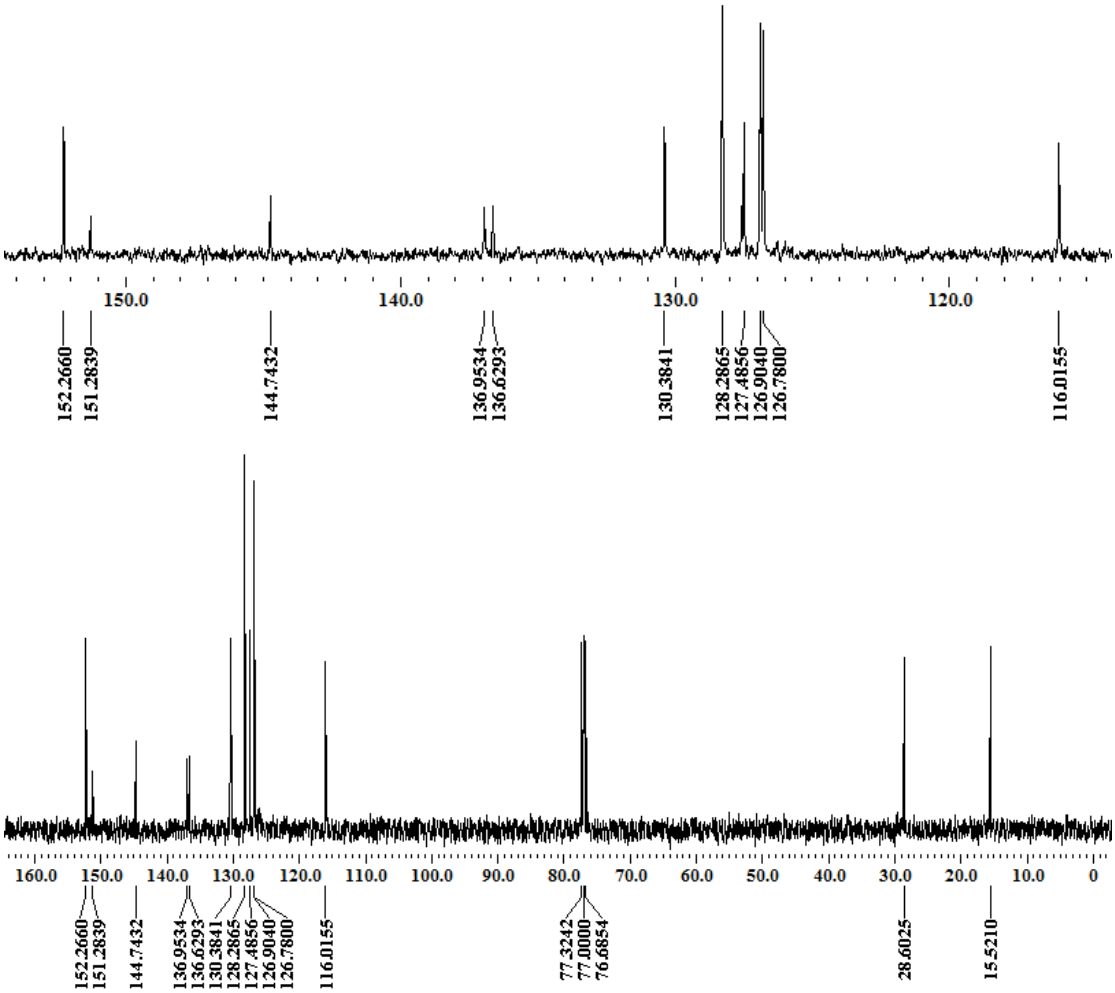


3-(4-Ethylphenyl)isoquinoline (8b)



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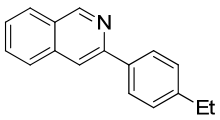
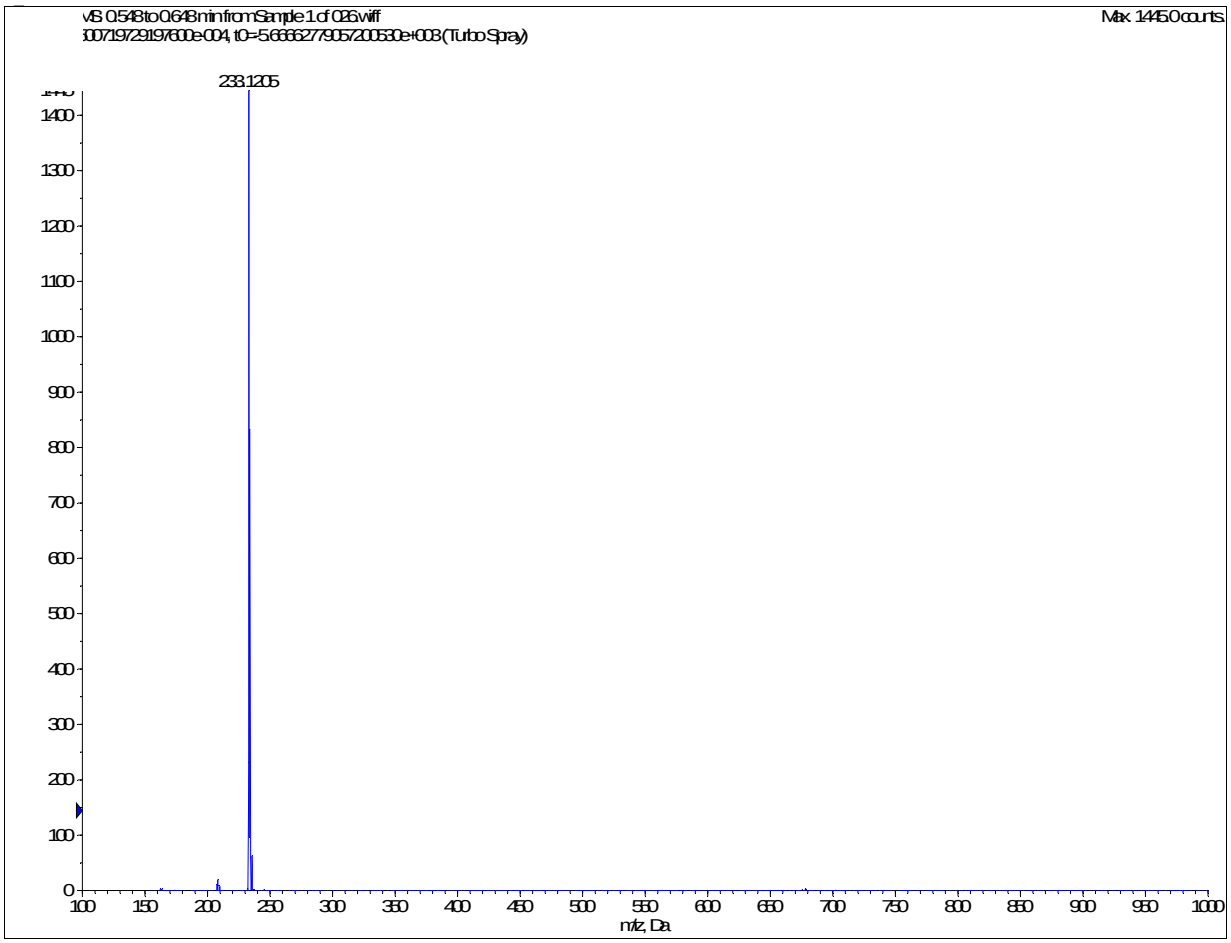
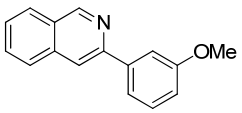


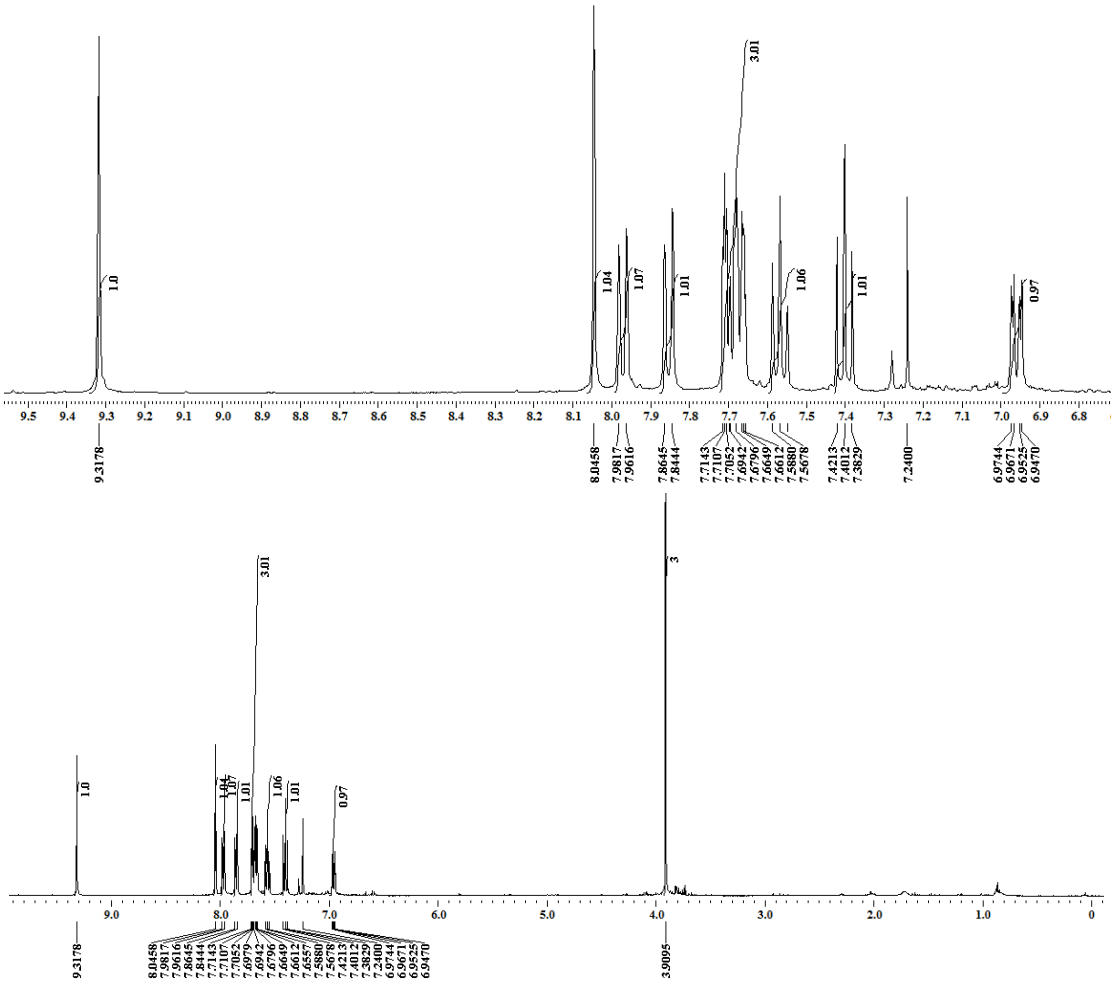
3-(4-Ethylphenyl)isoquinoline (8b)



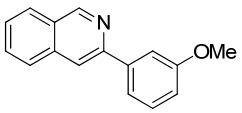
^{1}H NMR



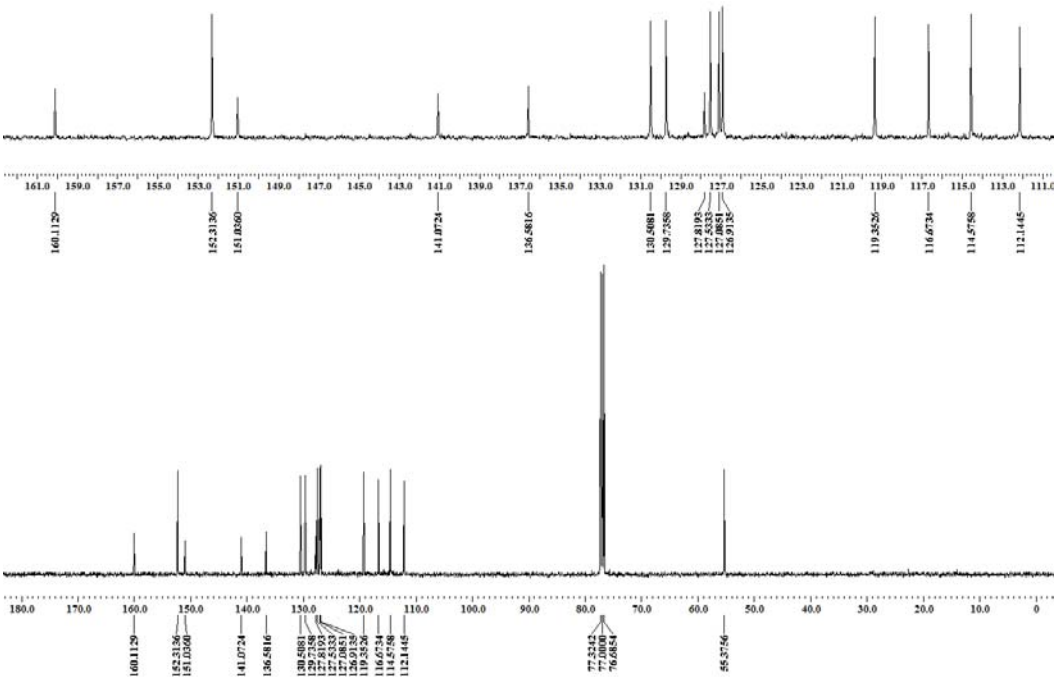
3-(3-Methoxyphenyl)isoquinoline (8c)



^{13}C NMR



3-(3-Methoxyphenyl)isoquinoline (8c)



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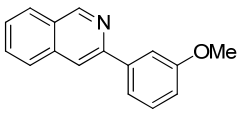
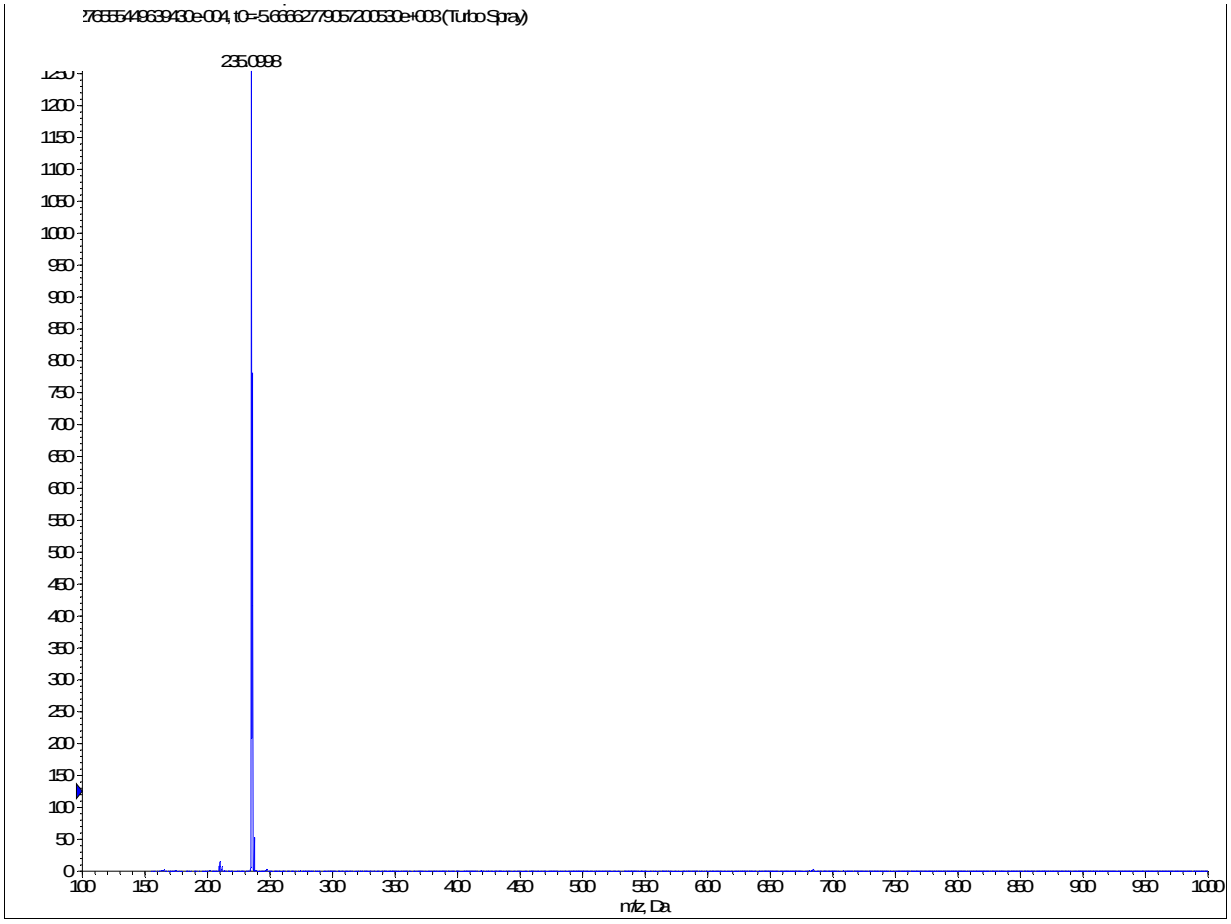
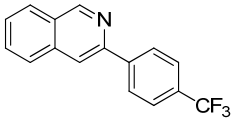


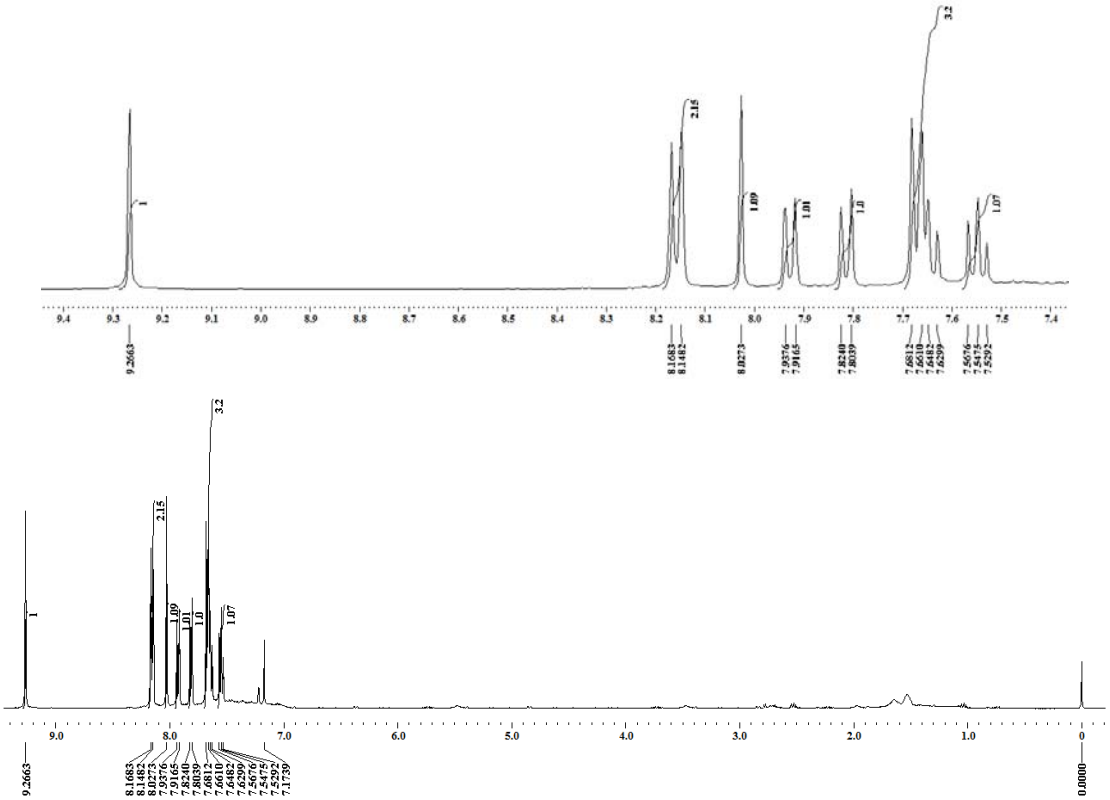
3-(3-Methoxyphenyl)isoquinoline (8c)



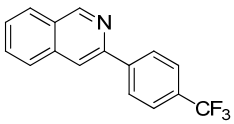
^{1}H NMR



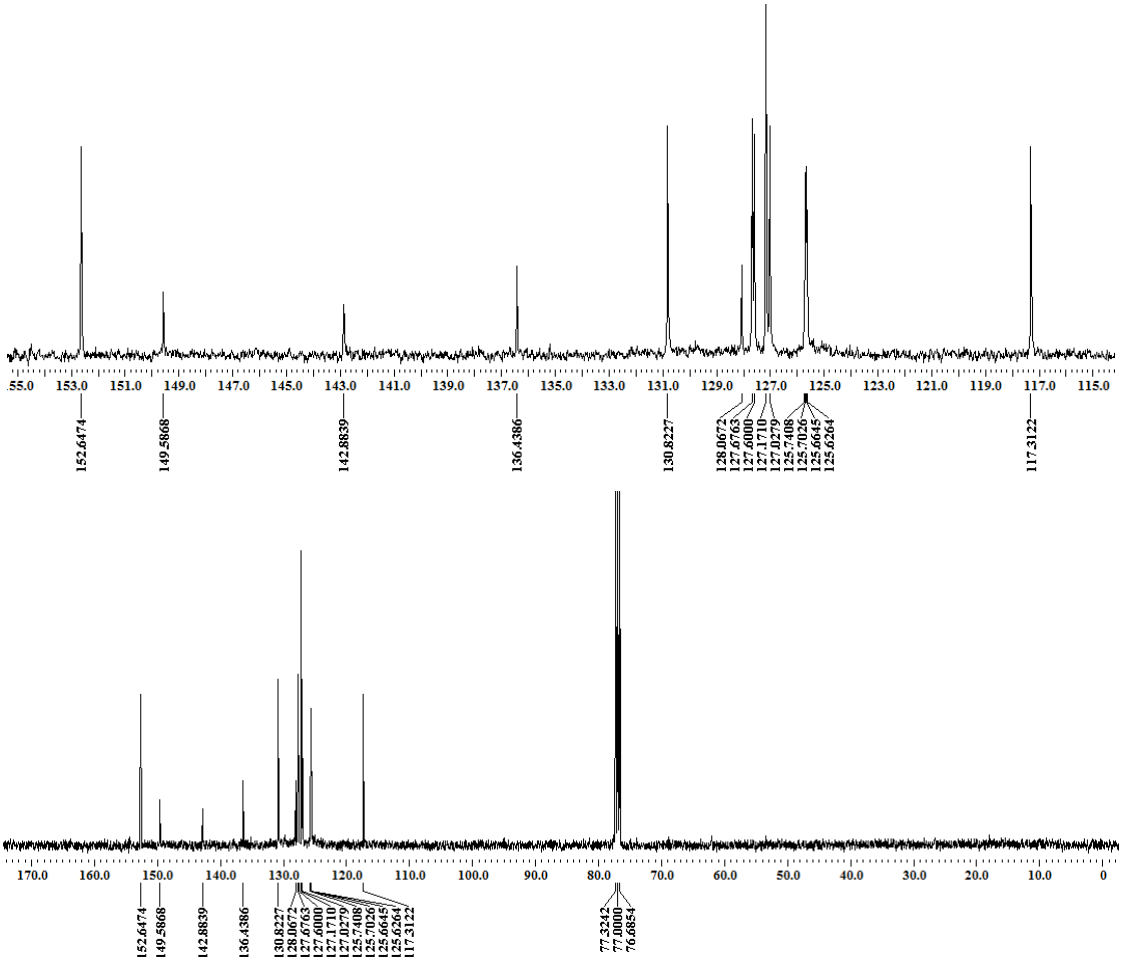
3-(4-(Trifluoromethyl)phenyl)isoquinoline (8d)



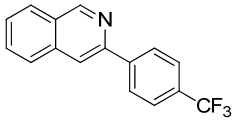
^{13}C NMR



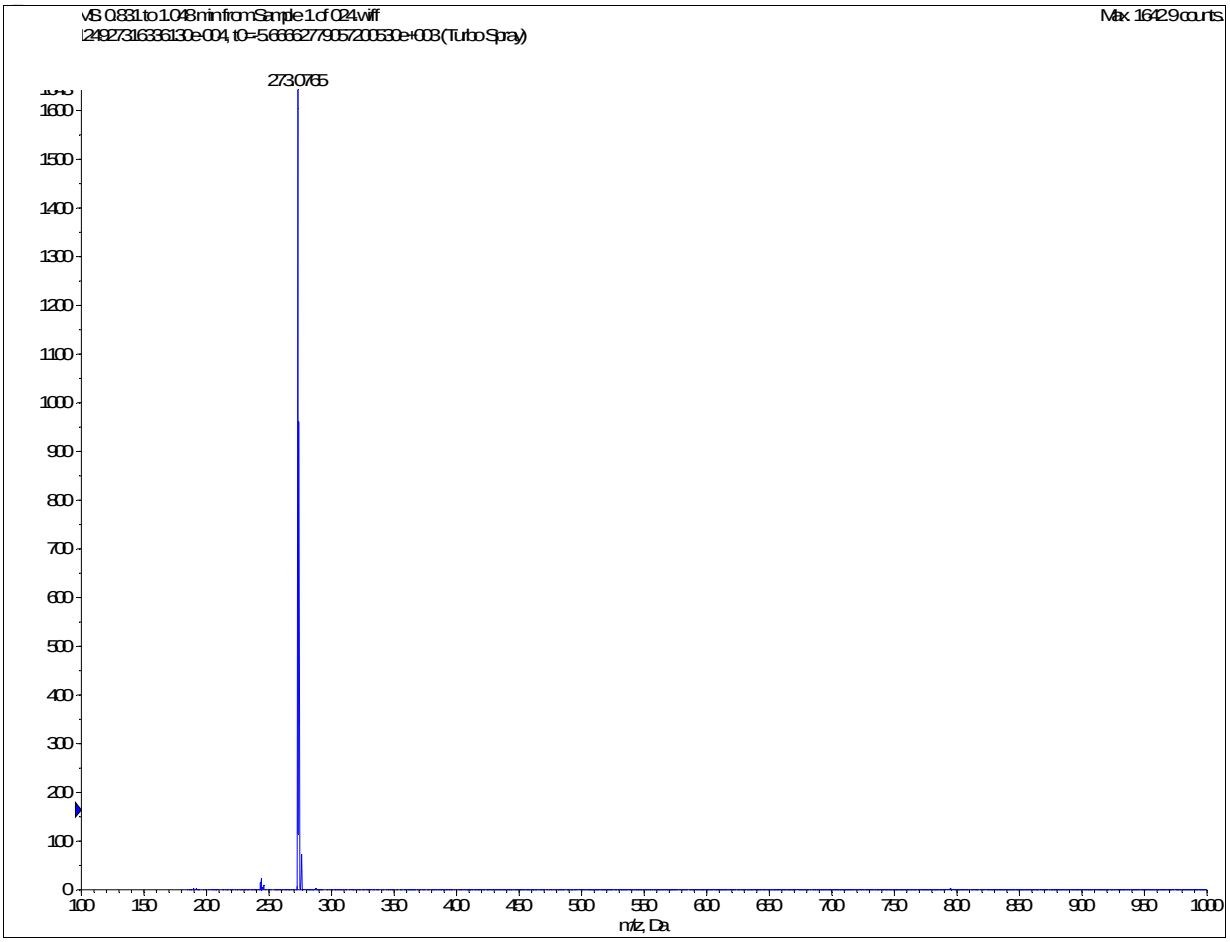
3-(4-(Trifluoromethyl)phenyl)isoquinoline (8d)



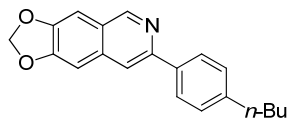
HRMS



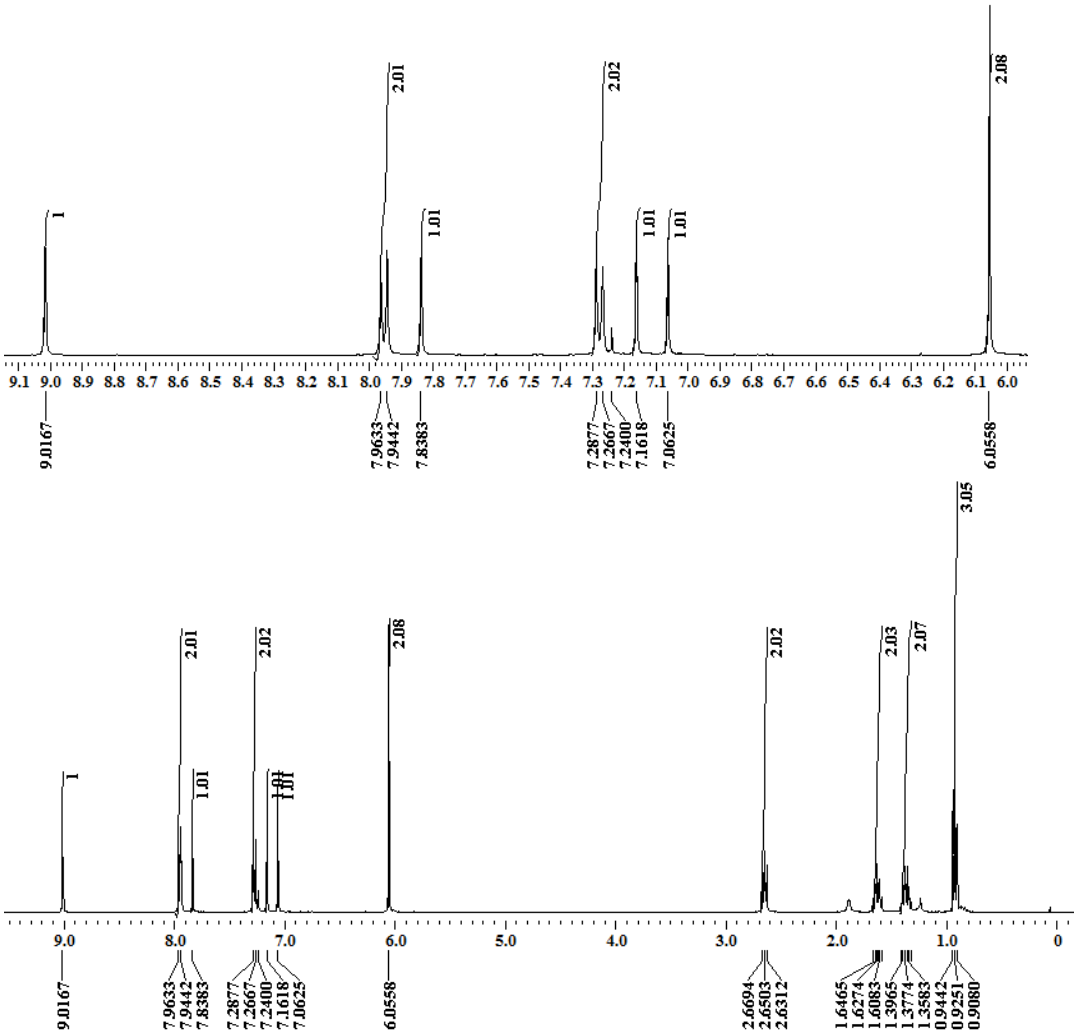
3-(4-(Trifluoromethyl)phenyl)isoquinoline (8d)



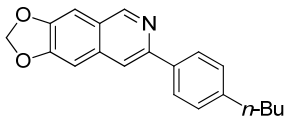
^{1}H NMR



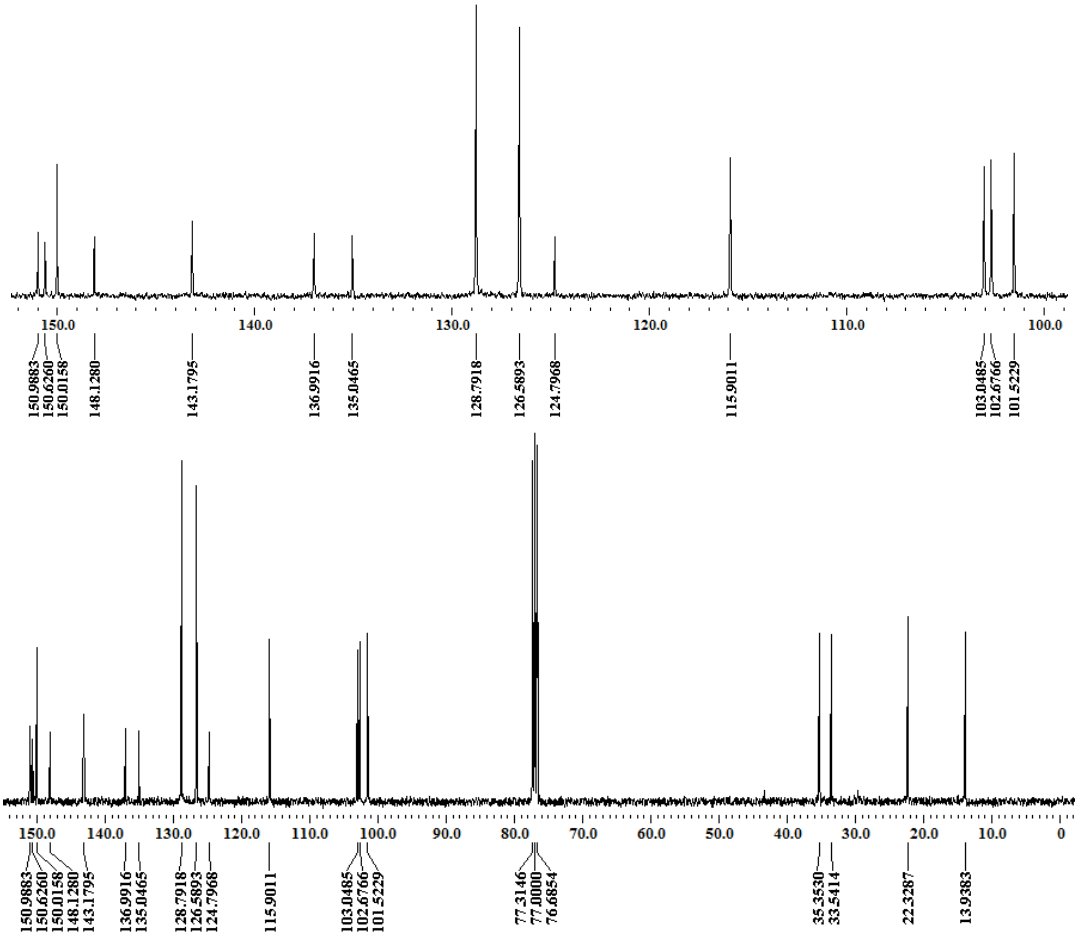
7-(4-Butylphenyl)-[1,3]dioxolo[4,5-*g*]isoquinoline (8e)



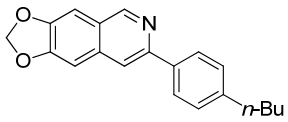
^{13}C NMR



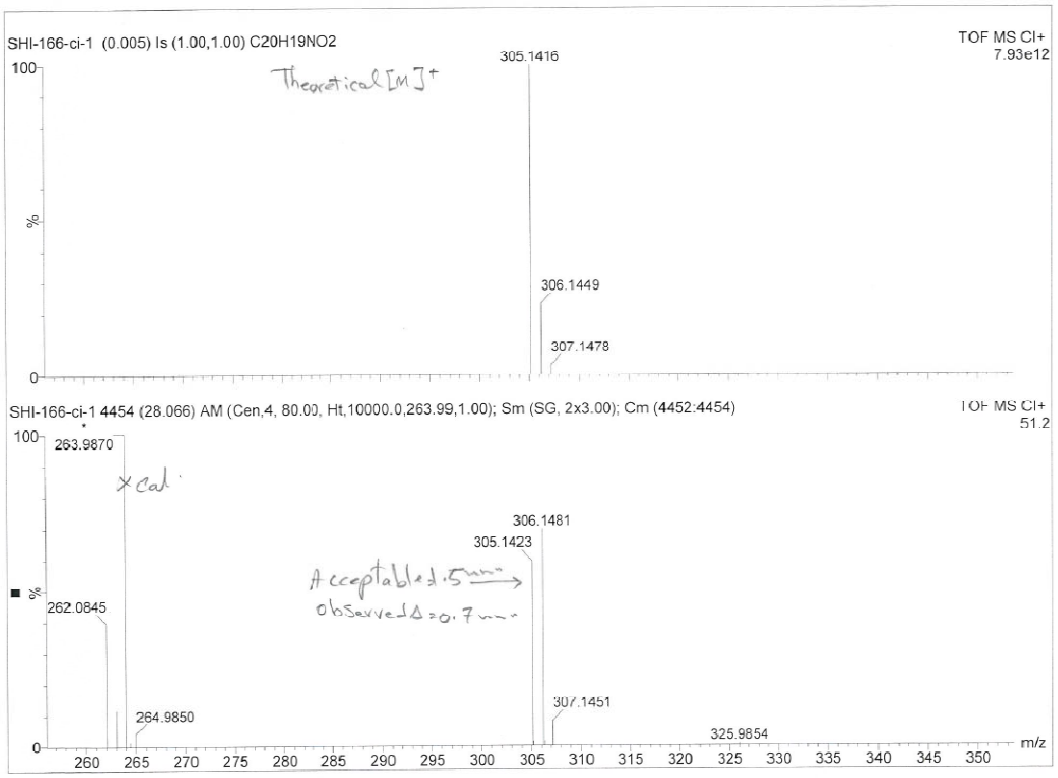
7-(4-Butylphenyl)-[1,3]dioxolo[4,5-*g*]isoquinoline (8e)



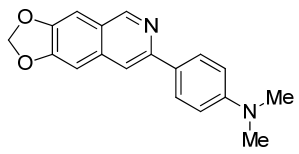
HRMS



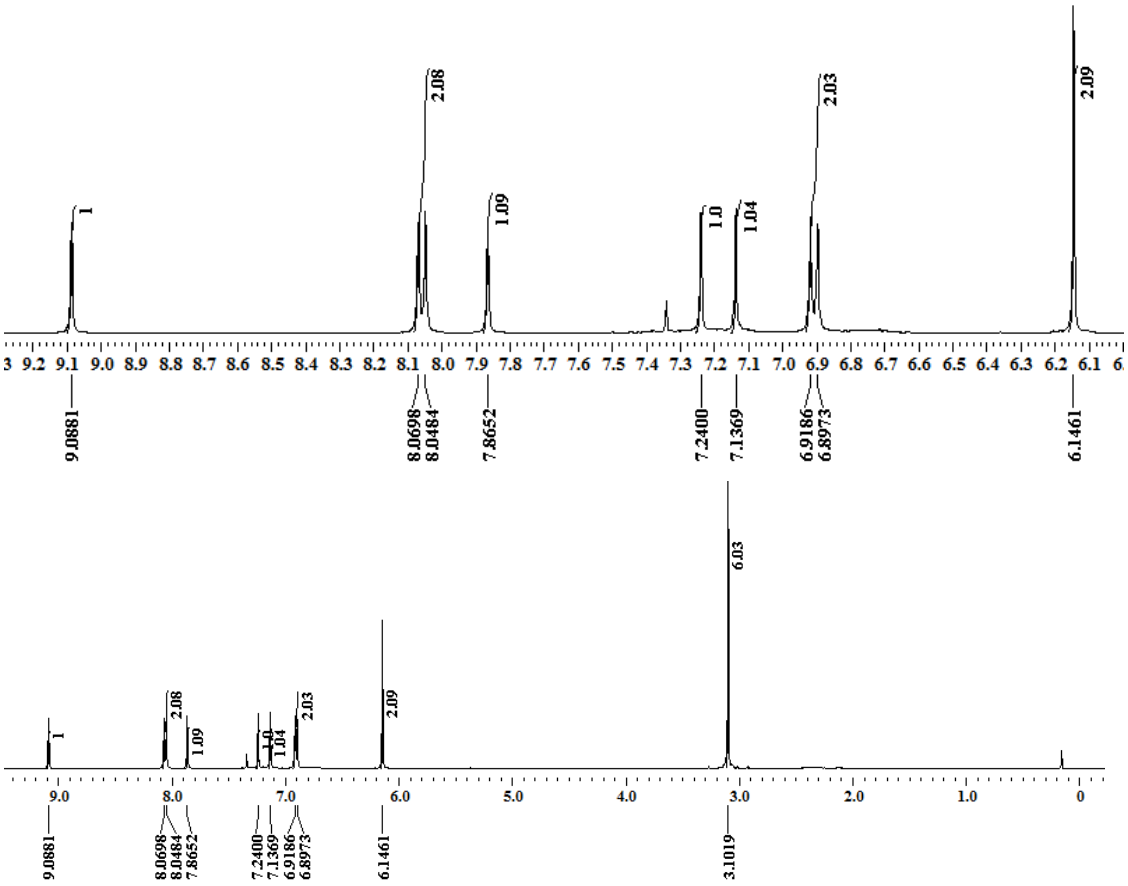
7-(4-Butylphenyl)-[1,3]dioxolo[4,5-*g*]isoquinoline (8e)



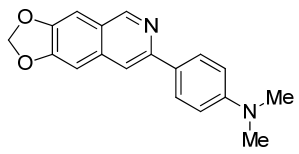
^{1}H NMR



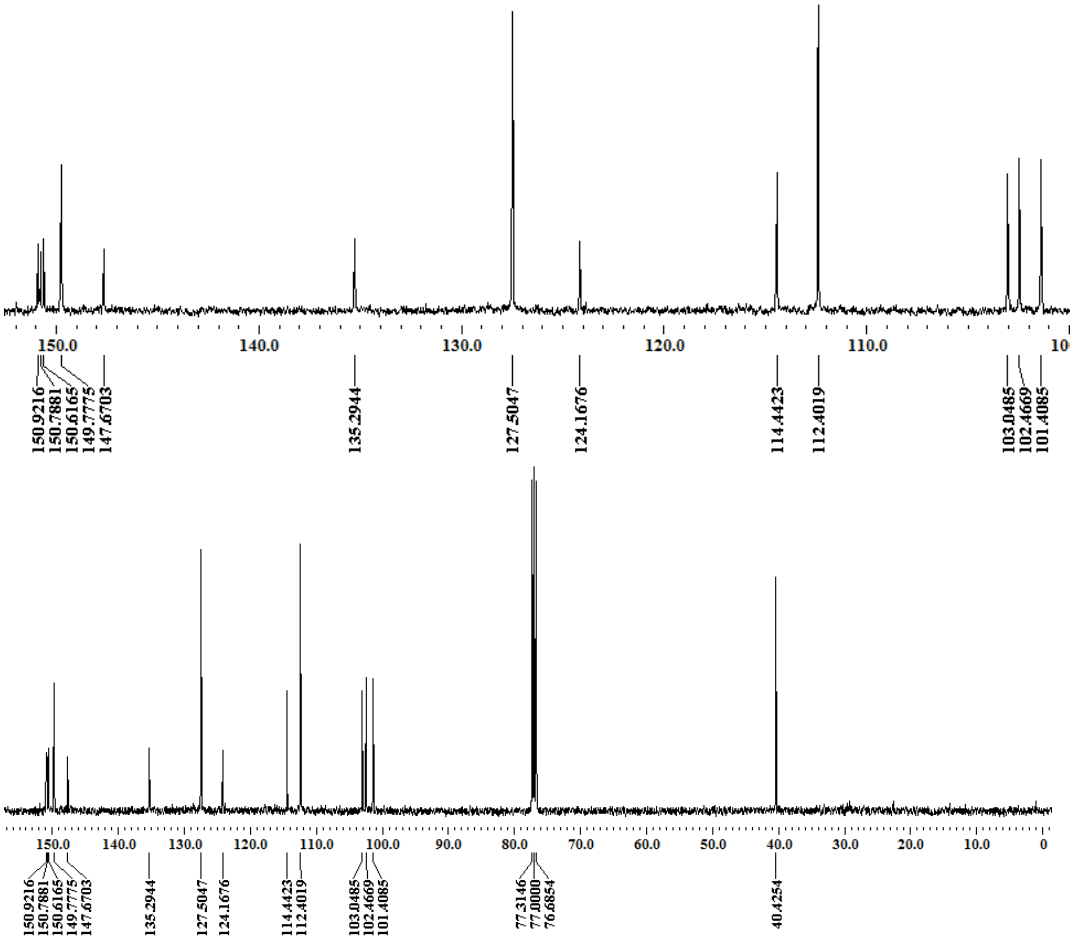
4-([1,3]Dioxolo[4,5-*g*]isoquinolin-7-yl)-N,N-dimethylaniline (8f)



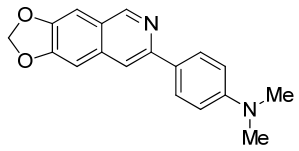
^{13}C NMR



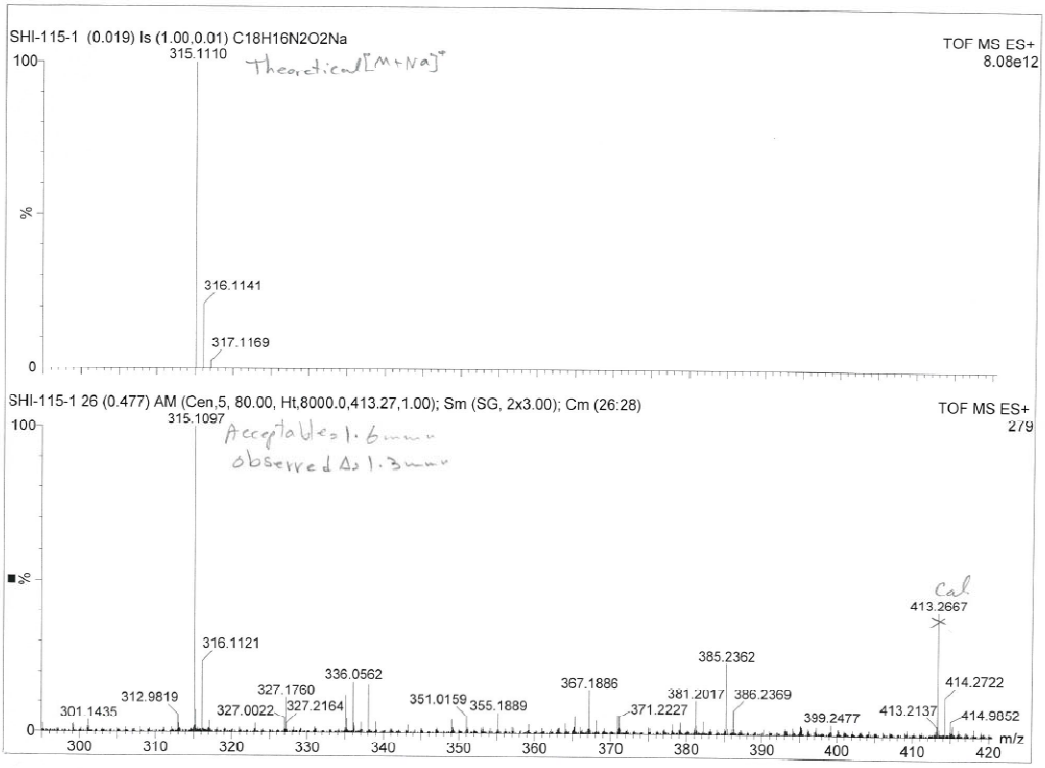
4-([1,3]Dioxolo[4,5-*g*]isoquinolin-7-yl)-N,N-dimethylaniline (8f)



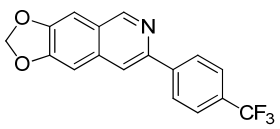
HRMS



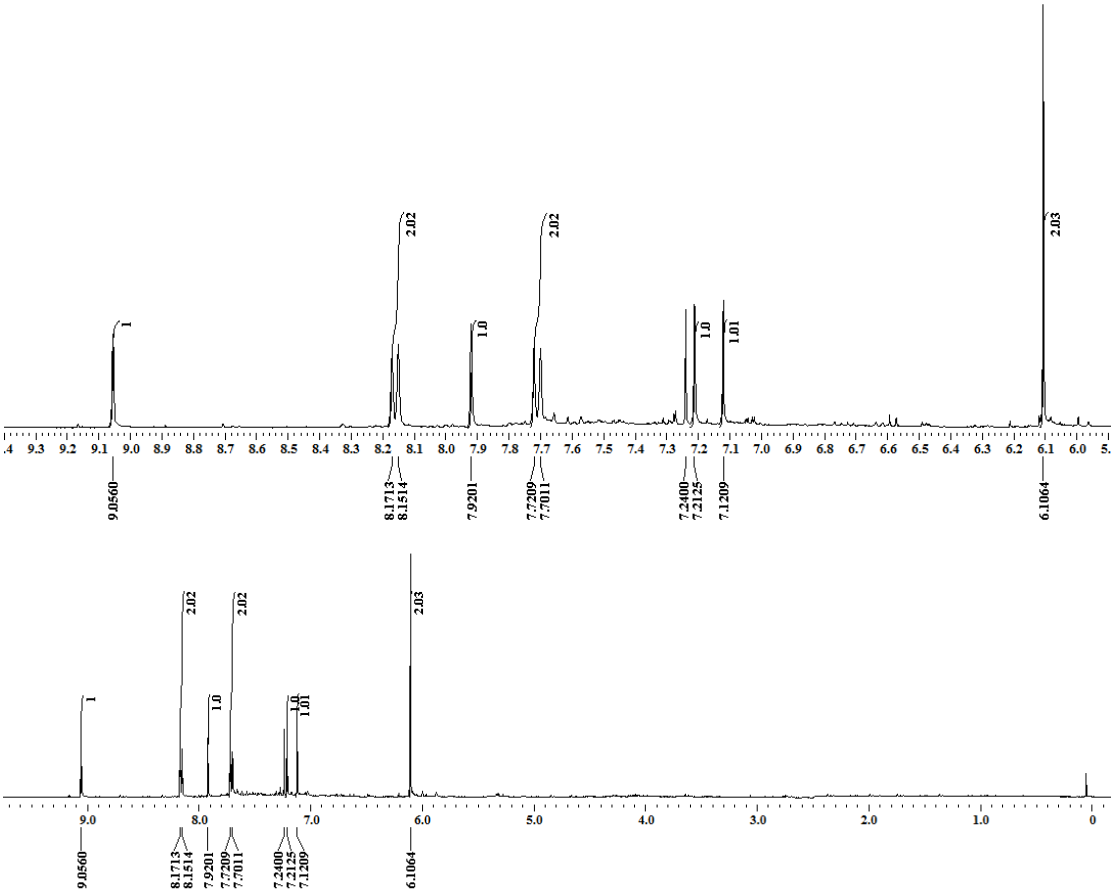
4-([1,3]Dioxolo[4,5-*g*]isoquinolin-7-yl)-N,N-dimethylaniline (8f)



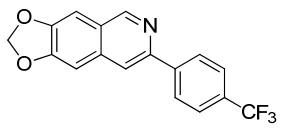
^{1}H NMR



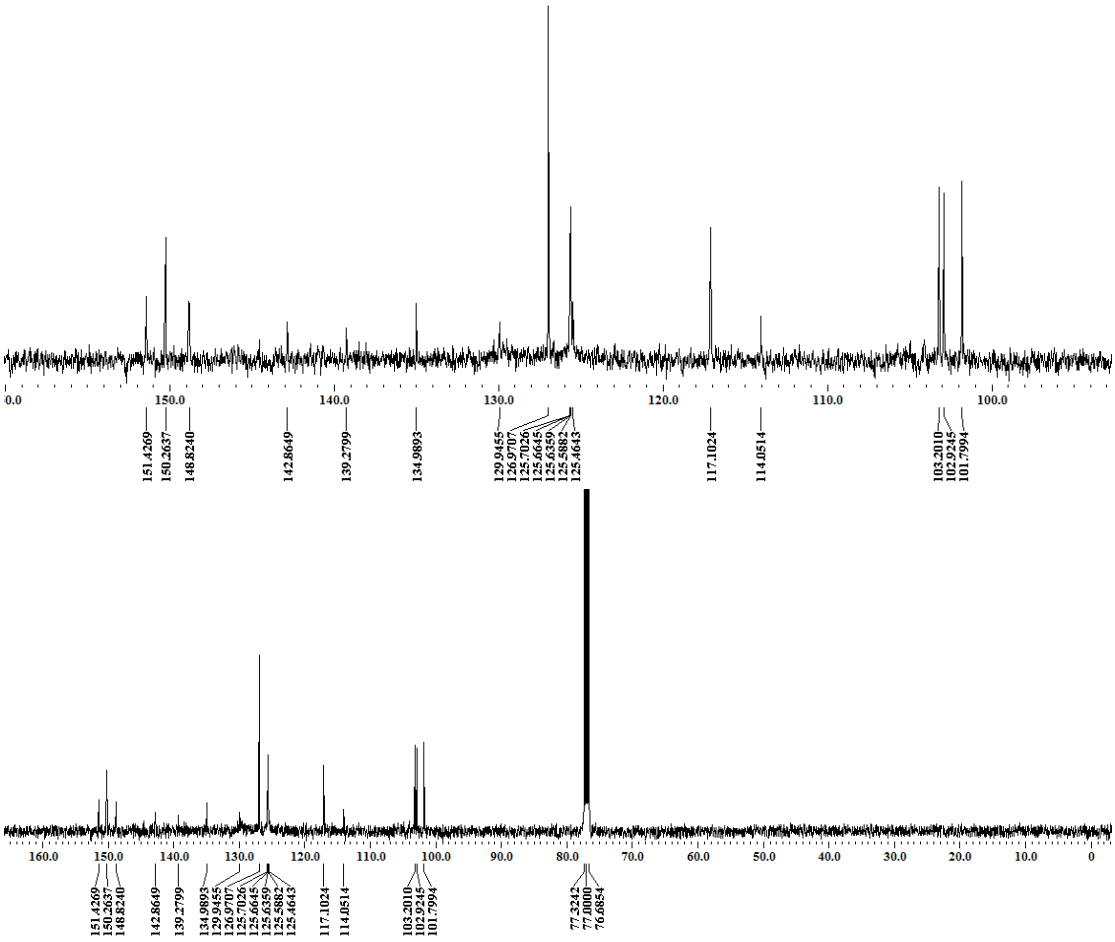
7-(4-(Trifluoromethyl)phenyl)-[1,3]dioxolo[4,5-g]isoquinoline (8g)



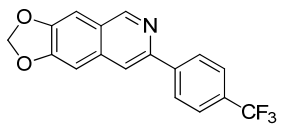
^{13}C NMR



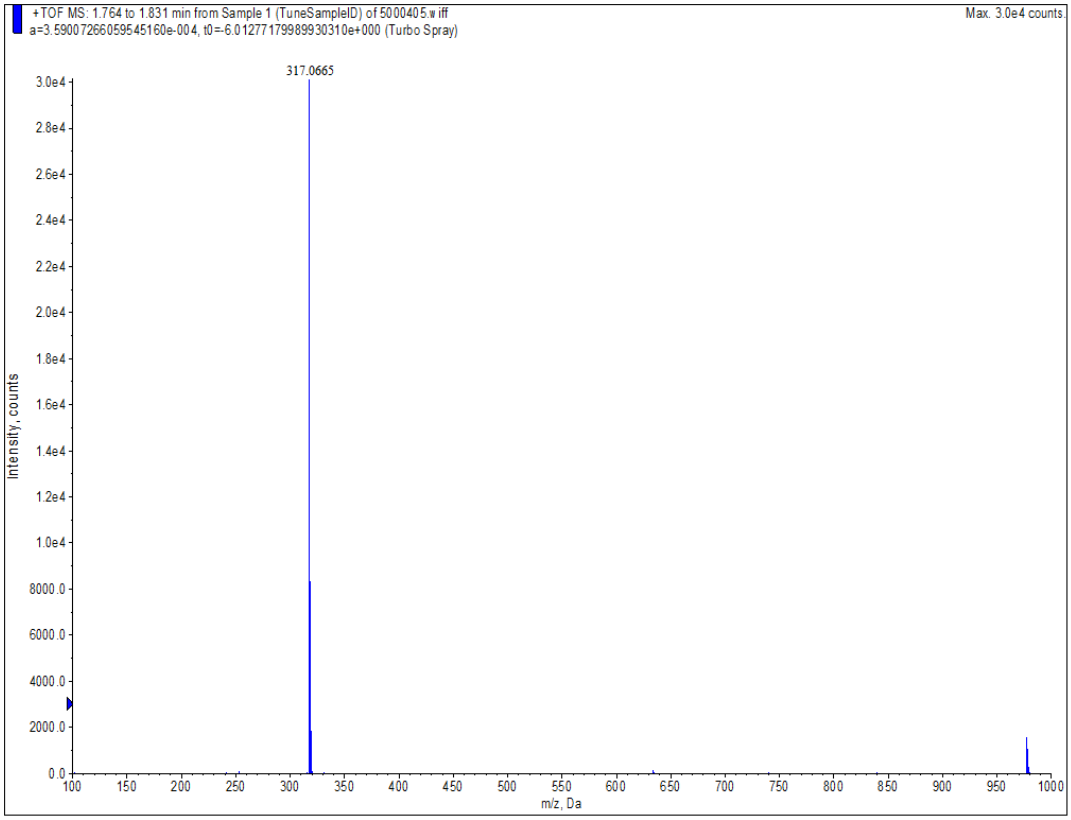
7-(4-(Trifluoromethyl)phenyl)-[1,3]dioxolo[4,5-g]isoquinoline (8g)



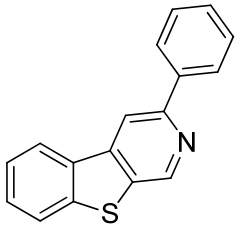
HRMS



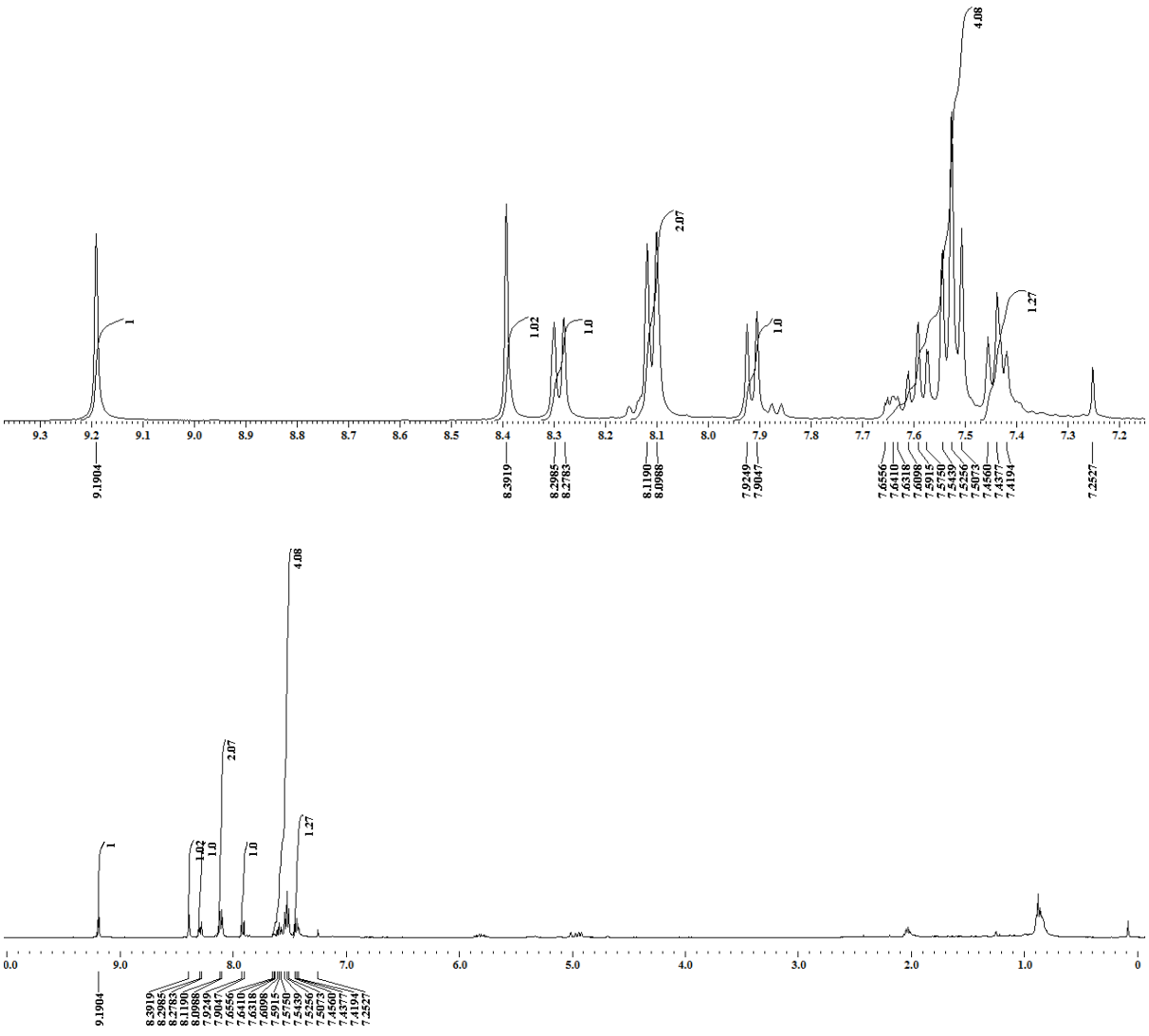
7-(4-(Trifluoromethyl)phenyl)-[1,3]dioxolo[4,5-g]isoquinoline (8g)



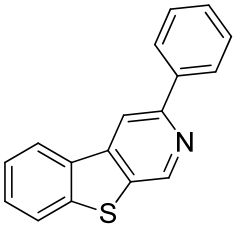
^{1}H NMR



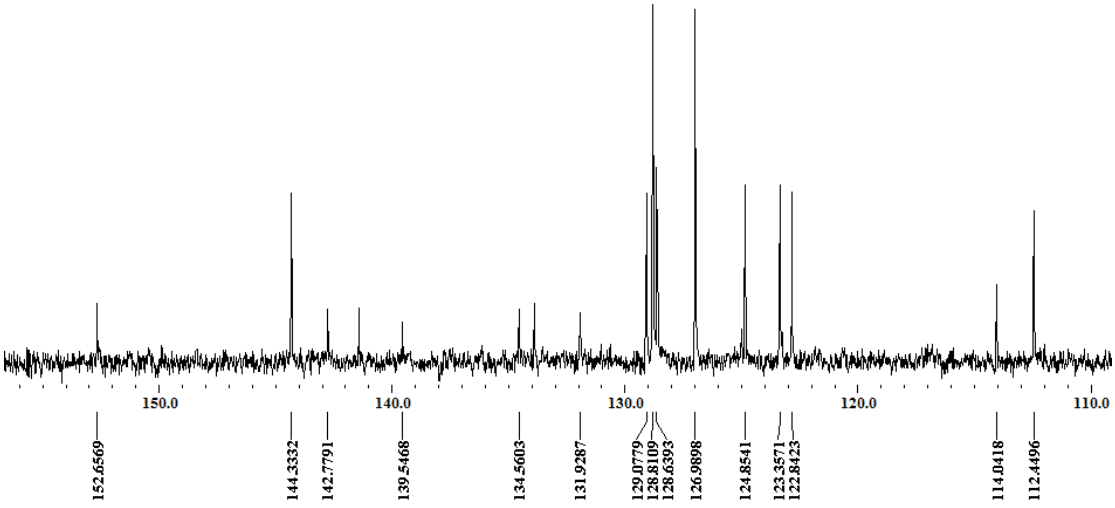
3-Phenylbenzo[4,5]thieno[2,3-*c*]pyridine (10a)

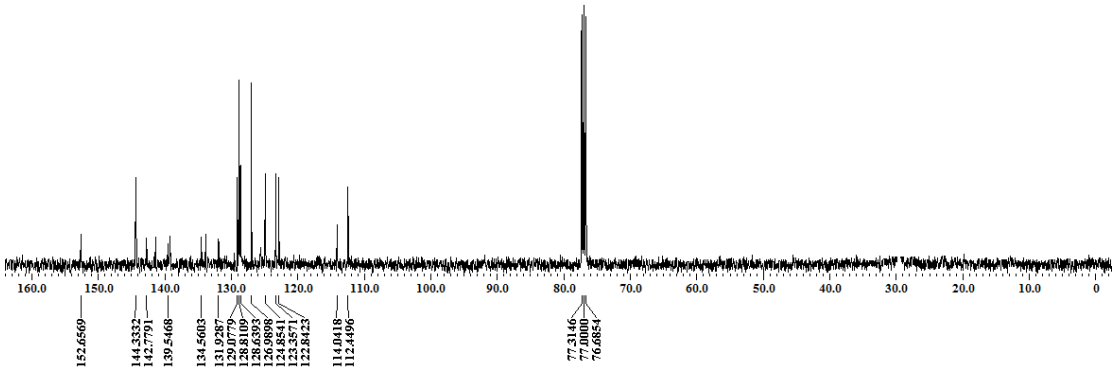


^{13}C NMR

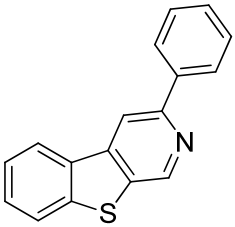


3-Phenylbenzo[4,5]thieno[2,3-*c*]pyridine (10a)

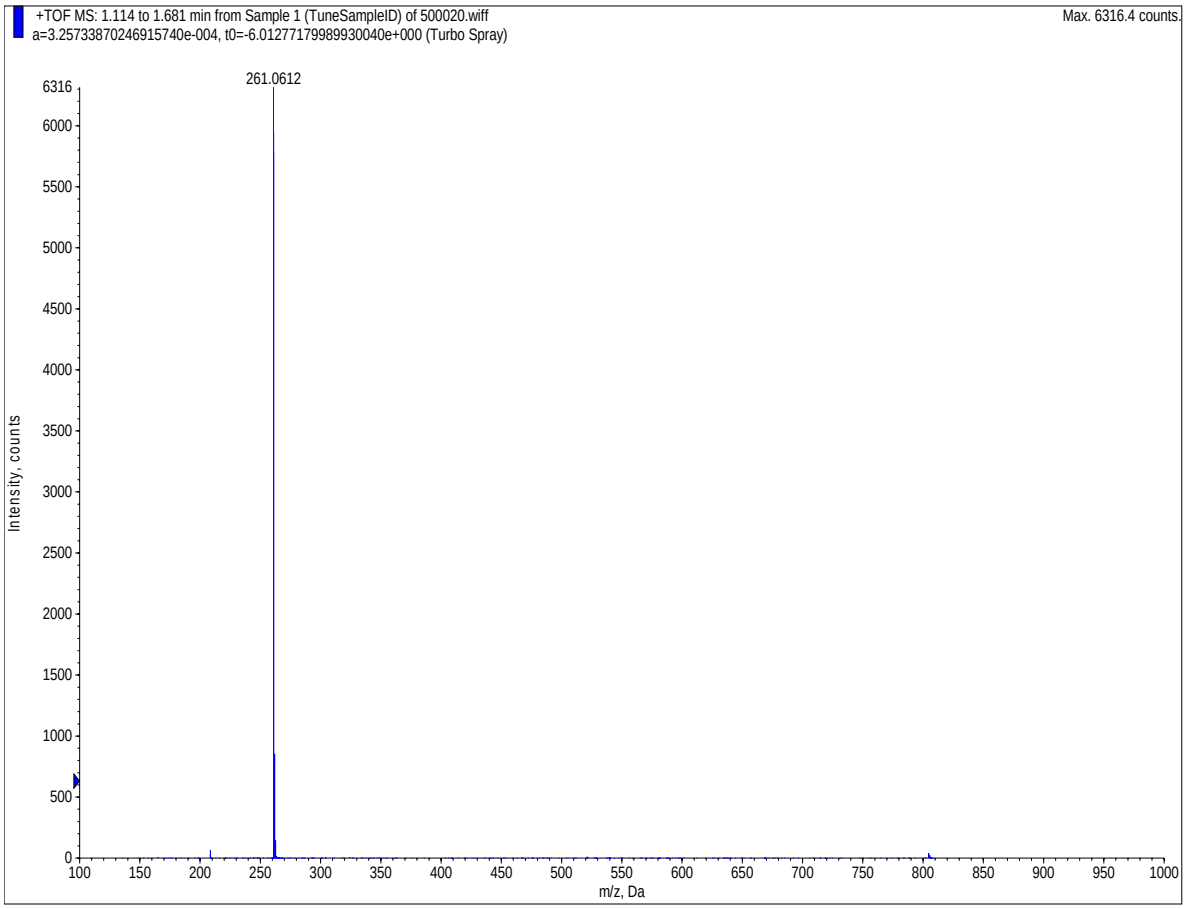




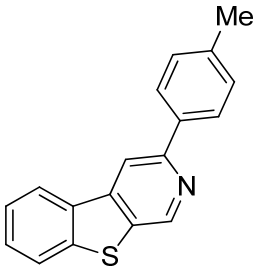
HRMS



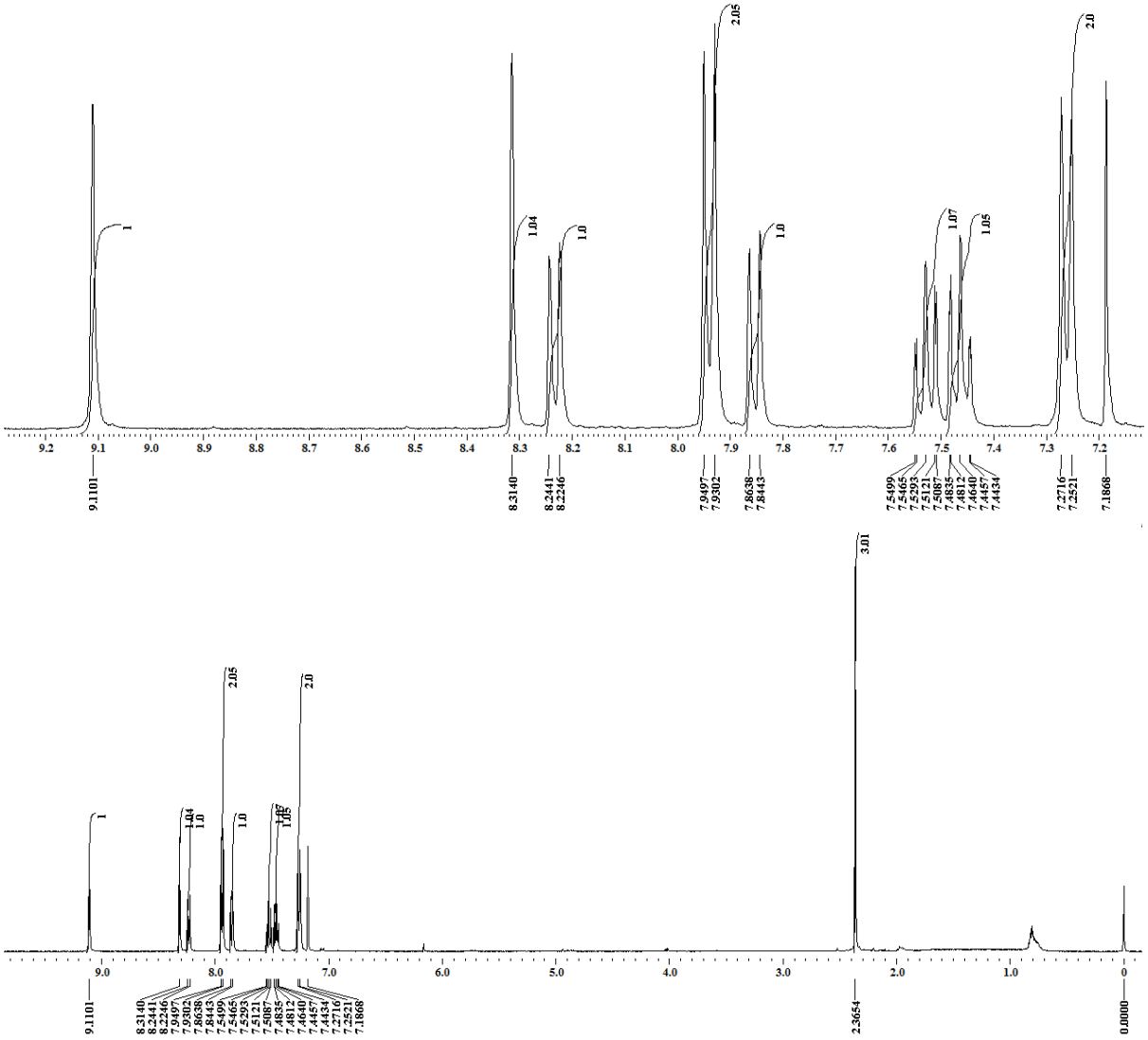
3-Phenylbenzo[4,5]thieno[2,3-*c*]pyridine (10a)



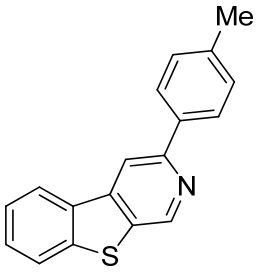
^{1}H NMR



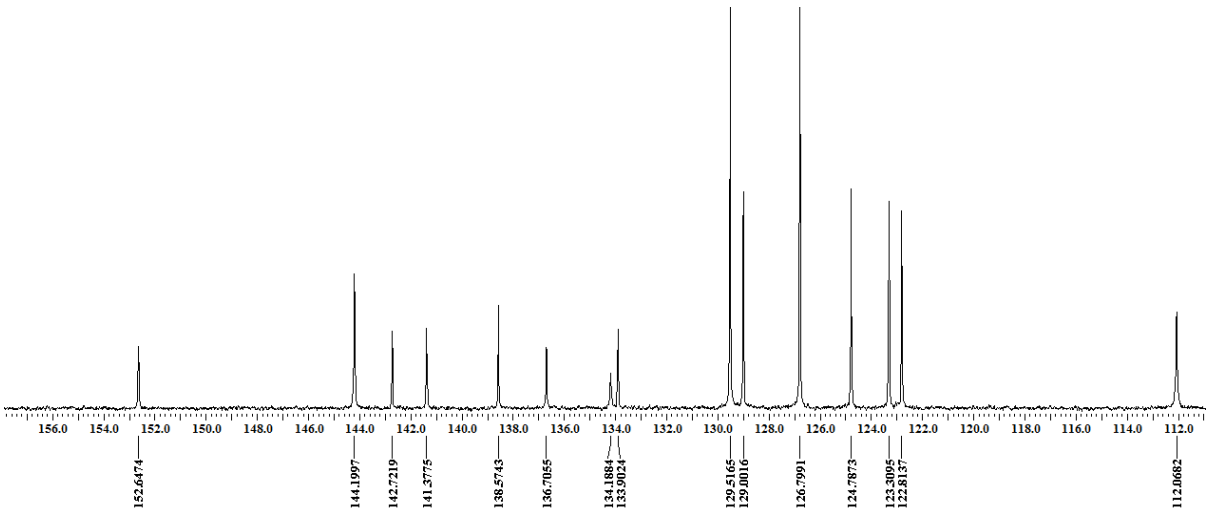
3-(*p*-Tolyl)benzo[4,5]thieno[2,3-*c*]pyridine (10b)

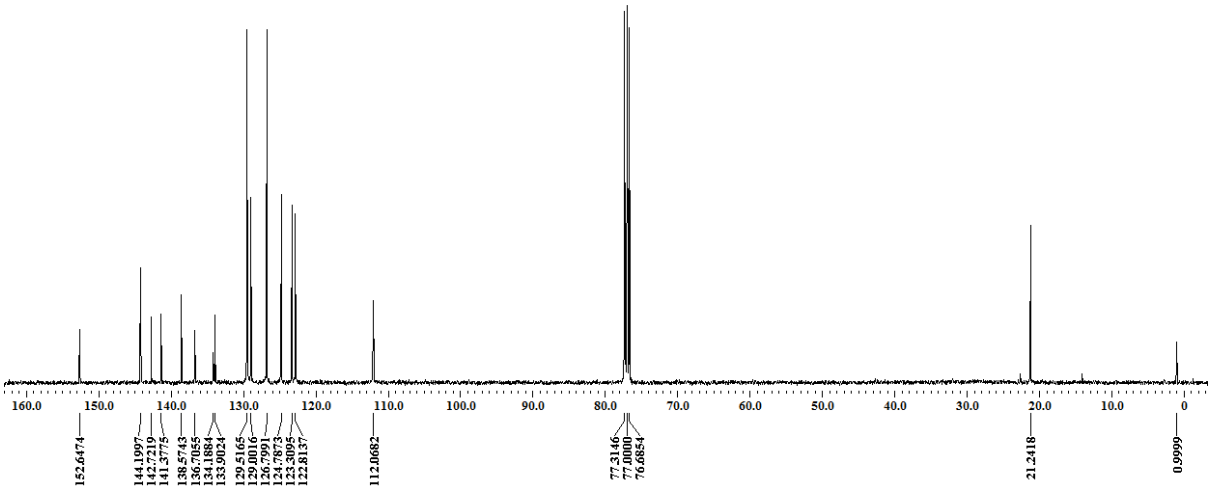


^{13}C NMR

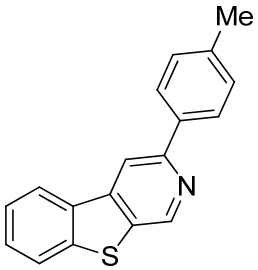


3-(*p*-Tolyl)benzo[4,5]thieno[2,3-*c*]pyridine (10b)

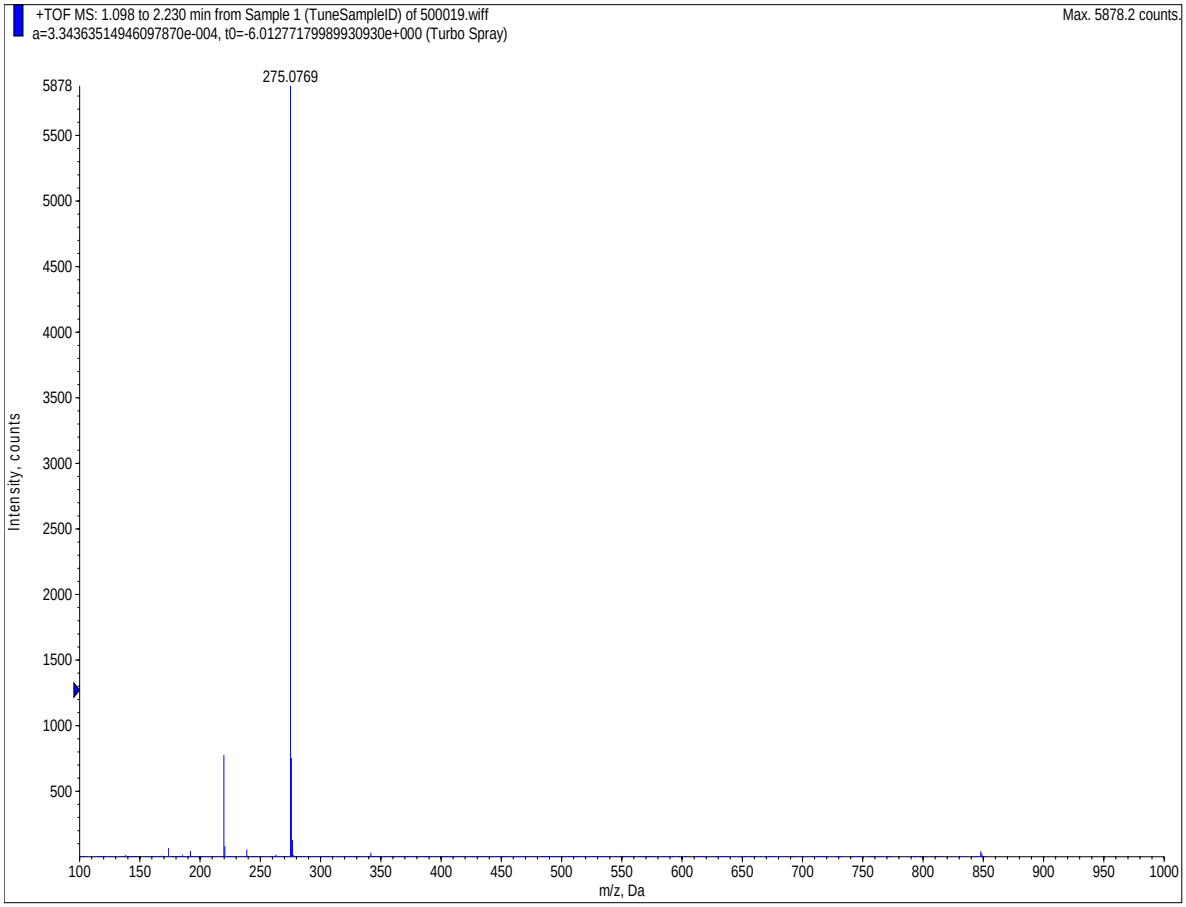




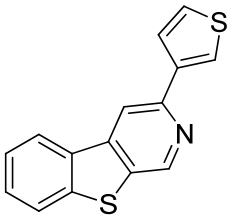
HRMS



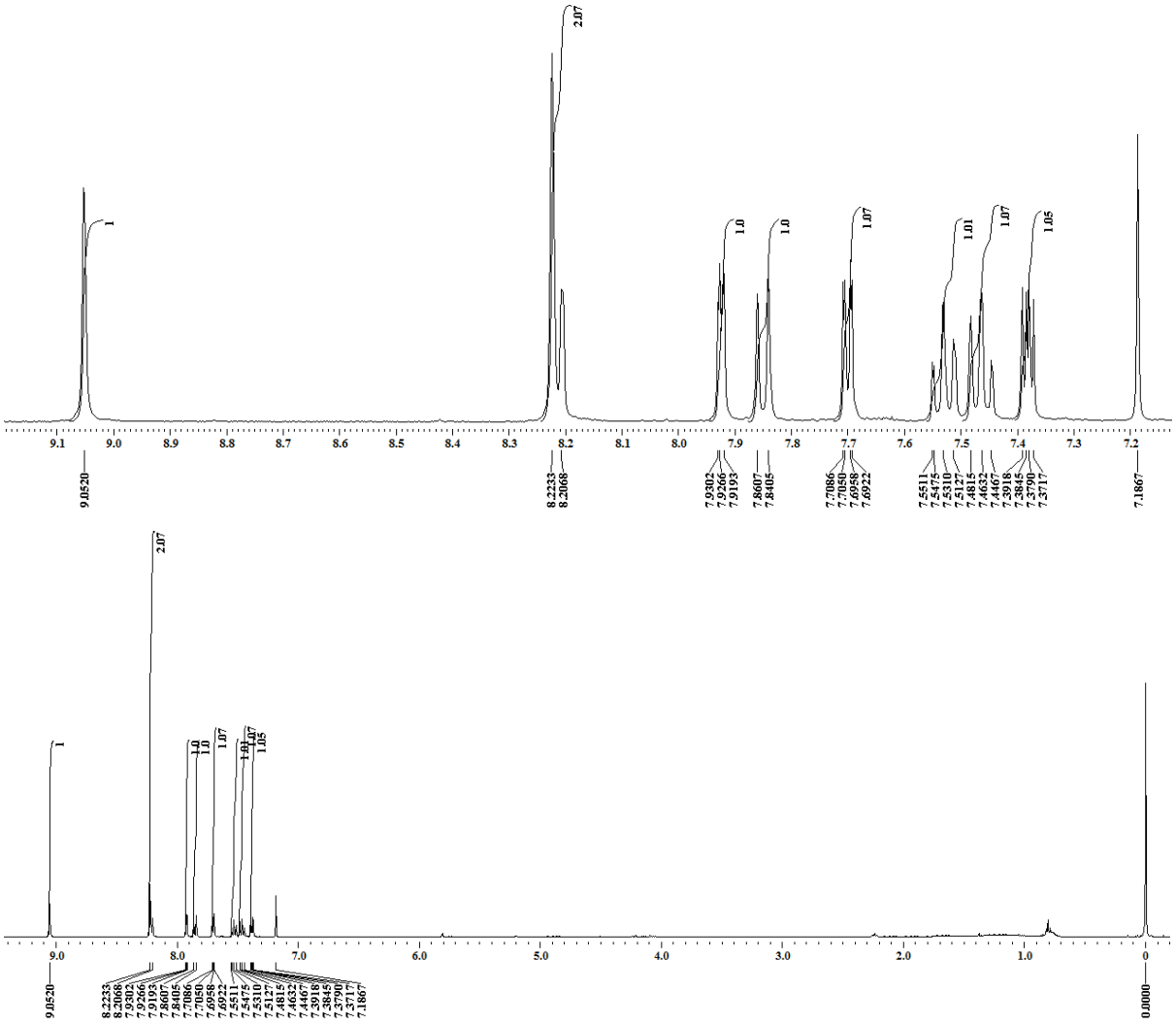
3-(*p*-Tolyl)benzo[4,5]thieno[2,3-*c*]pyridine (10b)



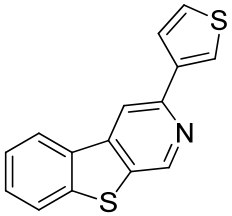
^{1}H NMR



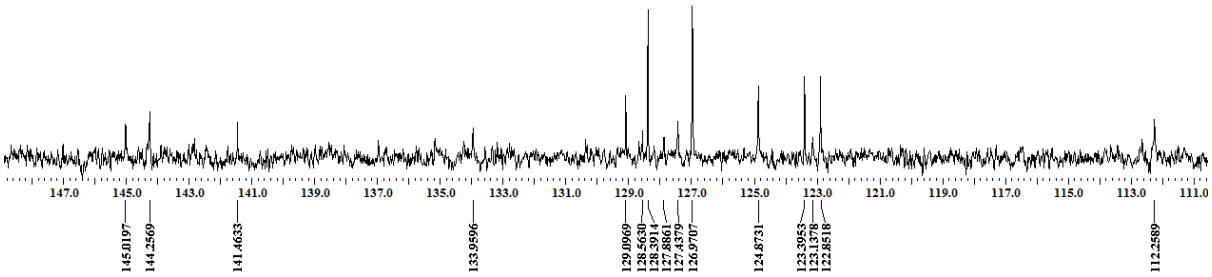
3-(Thiophen-3-yl)benzo[4,5]thieno[2,3-*c*]pyridine (10c)

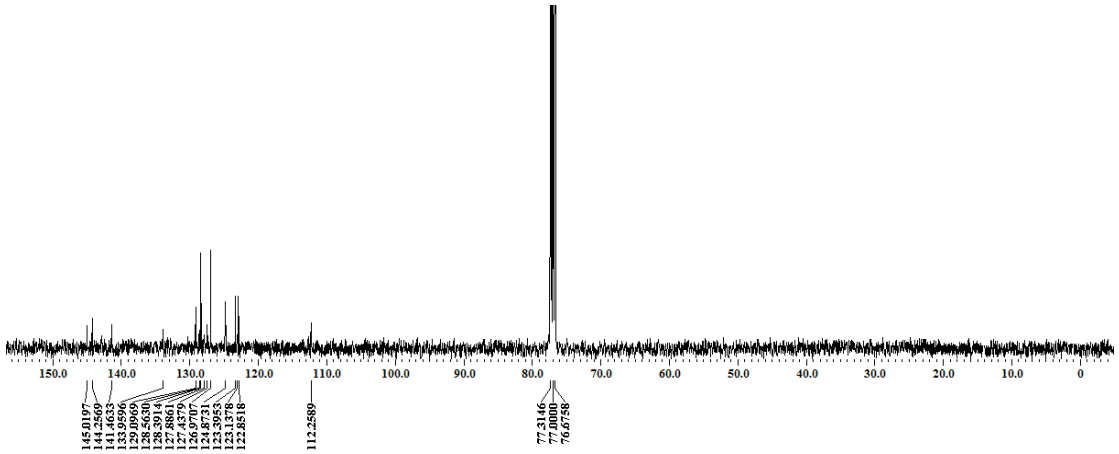


^{13}C NMR

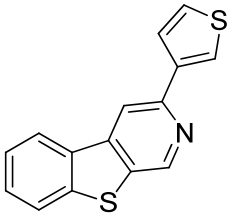


3-(Thiophen-3-yl)benzo[4,5]thieno[2,3-*c*]pyridine (10c)

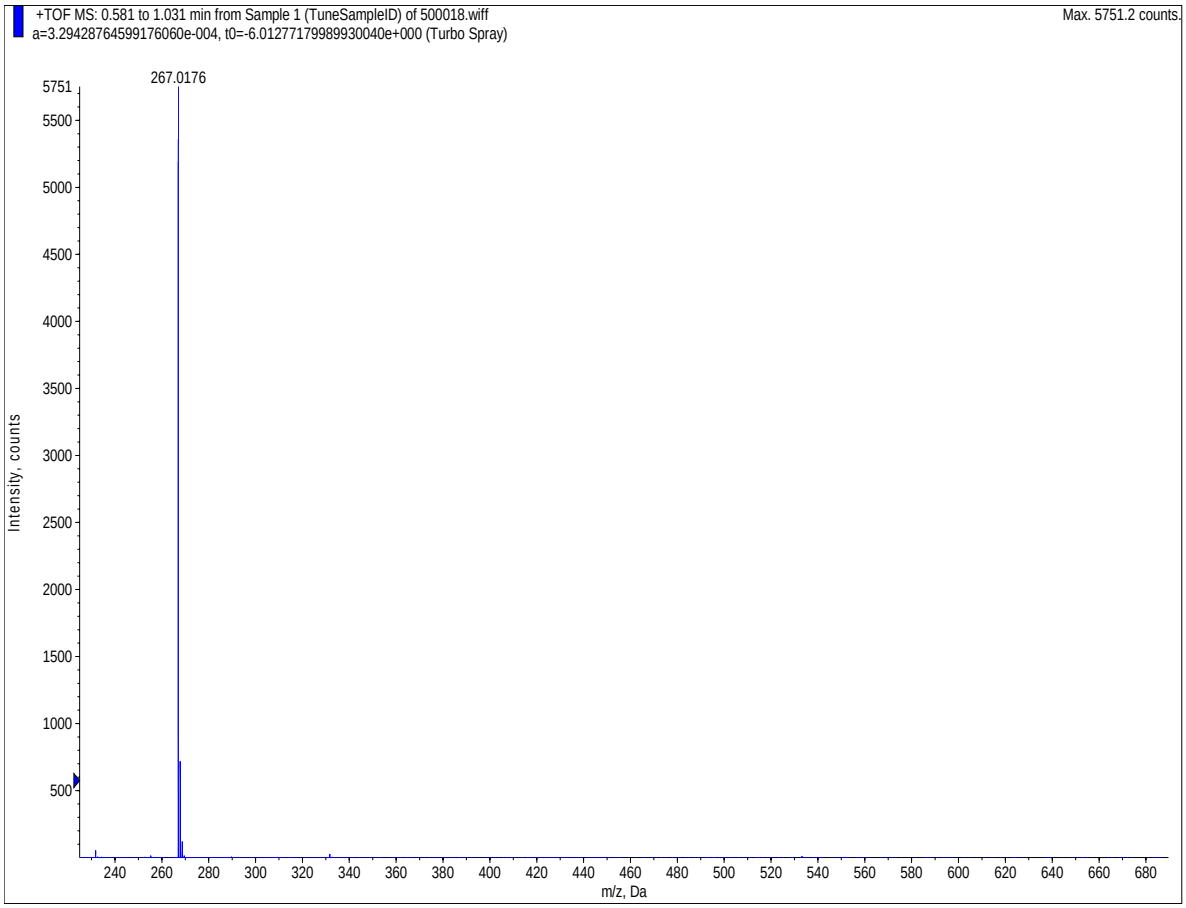




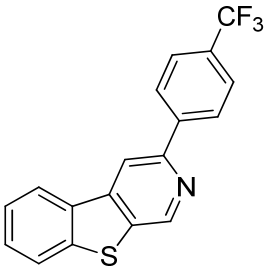
HRMS



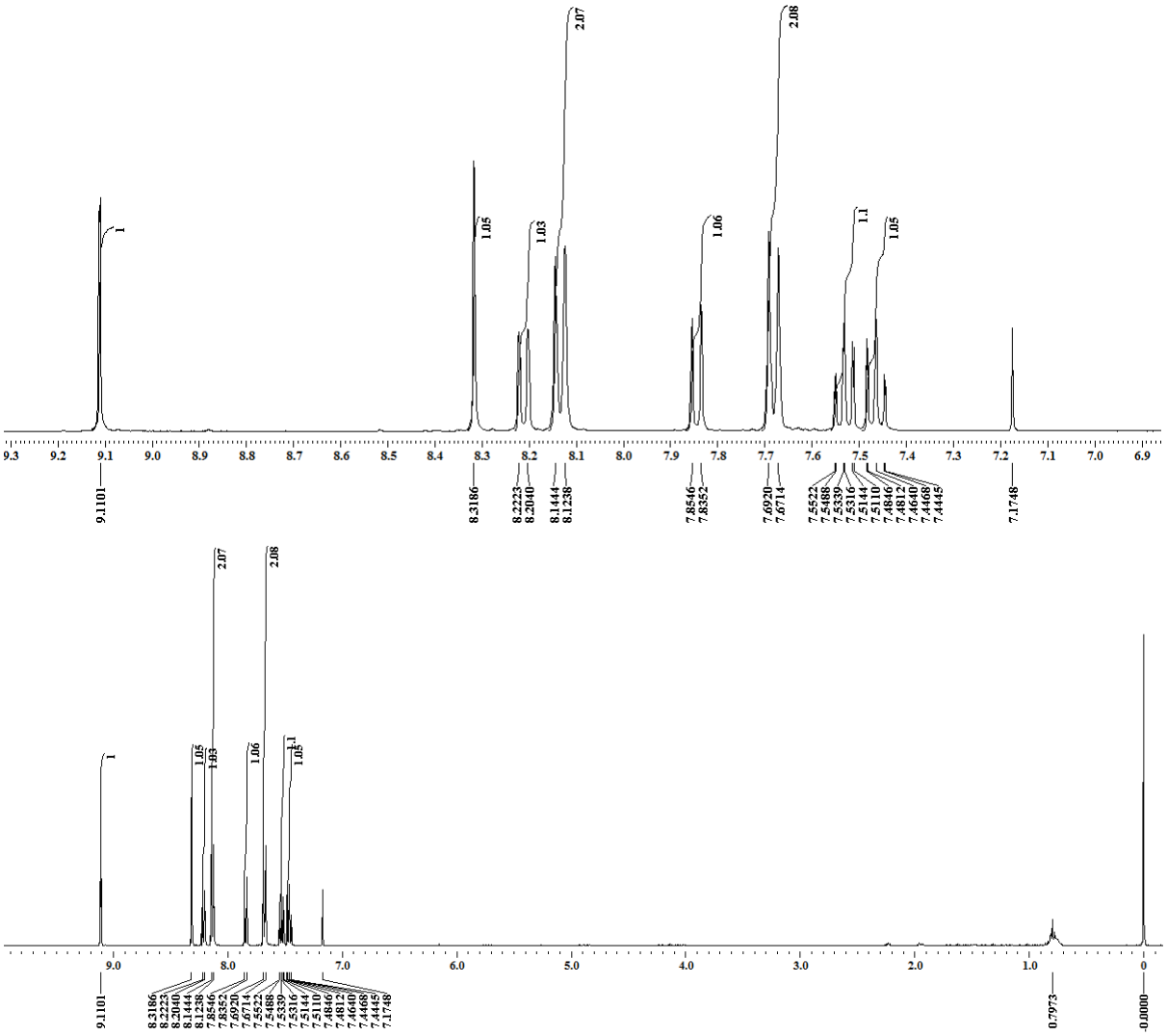
3-(Thiophen-3-yl)benzo[4,5]thieno[2,3-*c*]pyridine (10c)



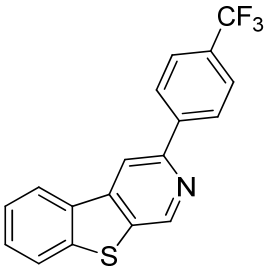
^{1}H NMR



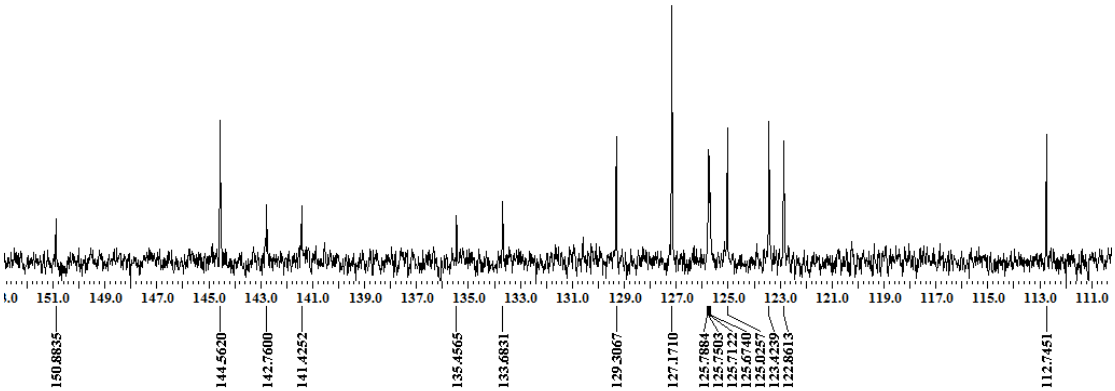
3-(4-(Trifluoromethyl)phenyl)benzo[4,5]thieno[2,3-*c*]pyridine(10d)

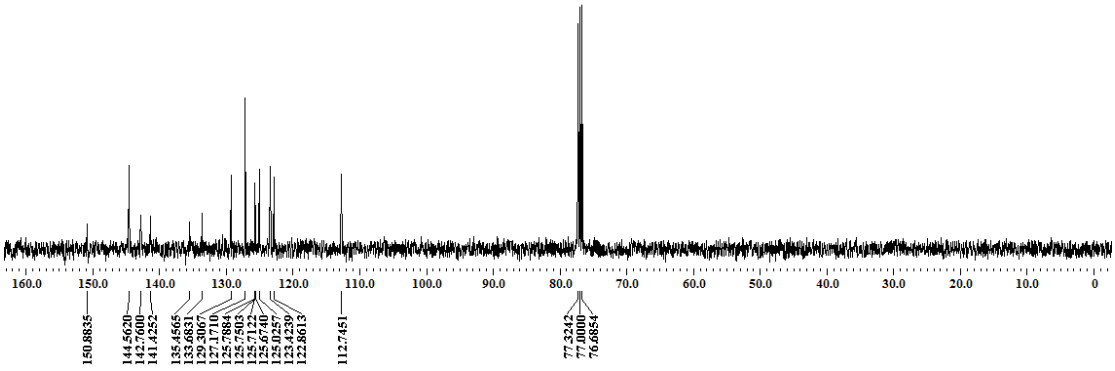


^{13}C NMR



3-(4-(Trifluoromethyl)phenyl)benzo[4,5]thieno[2,3-*c*]pyridine(10d)





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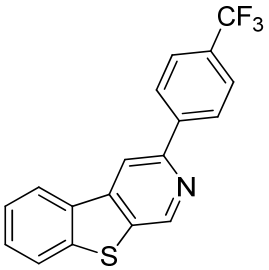
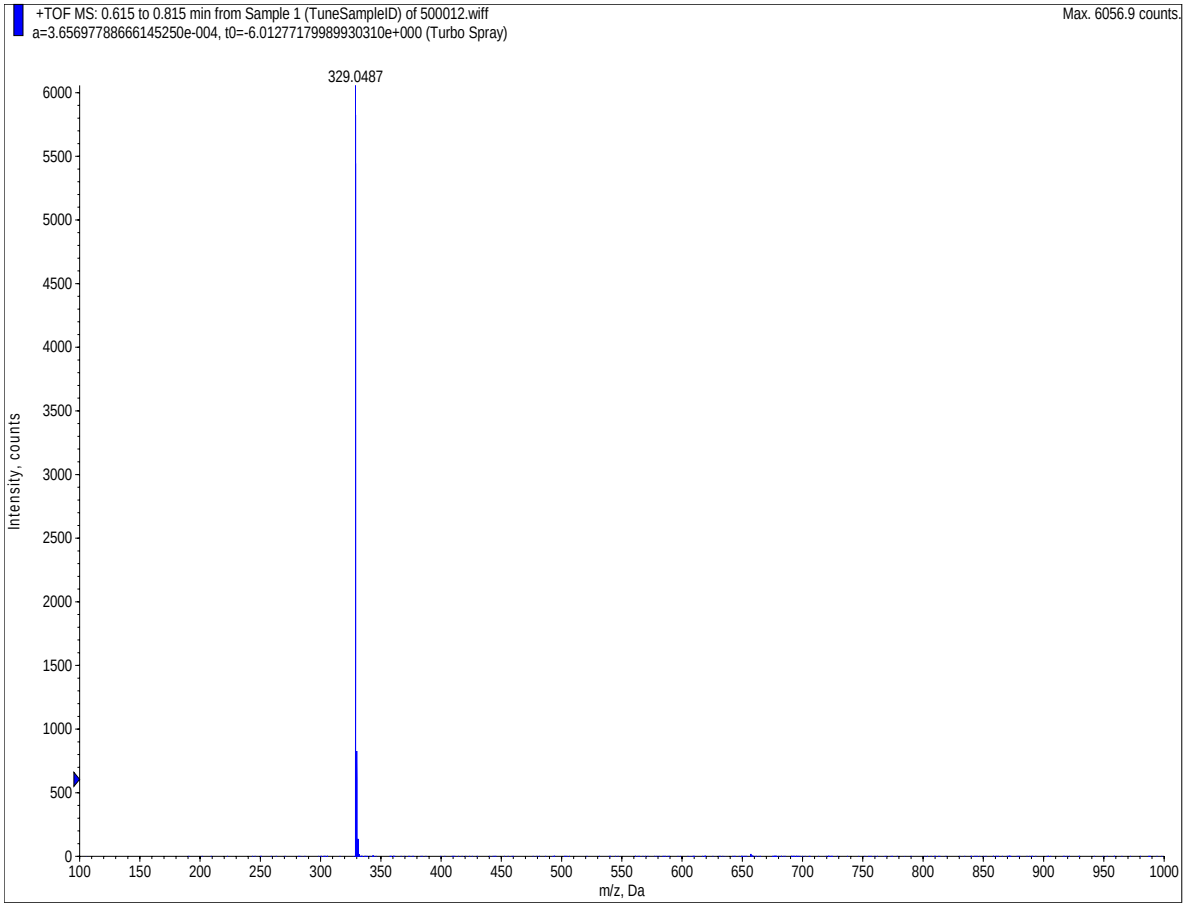
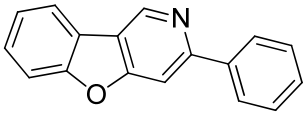


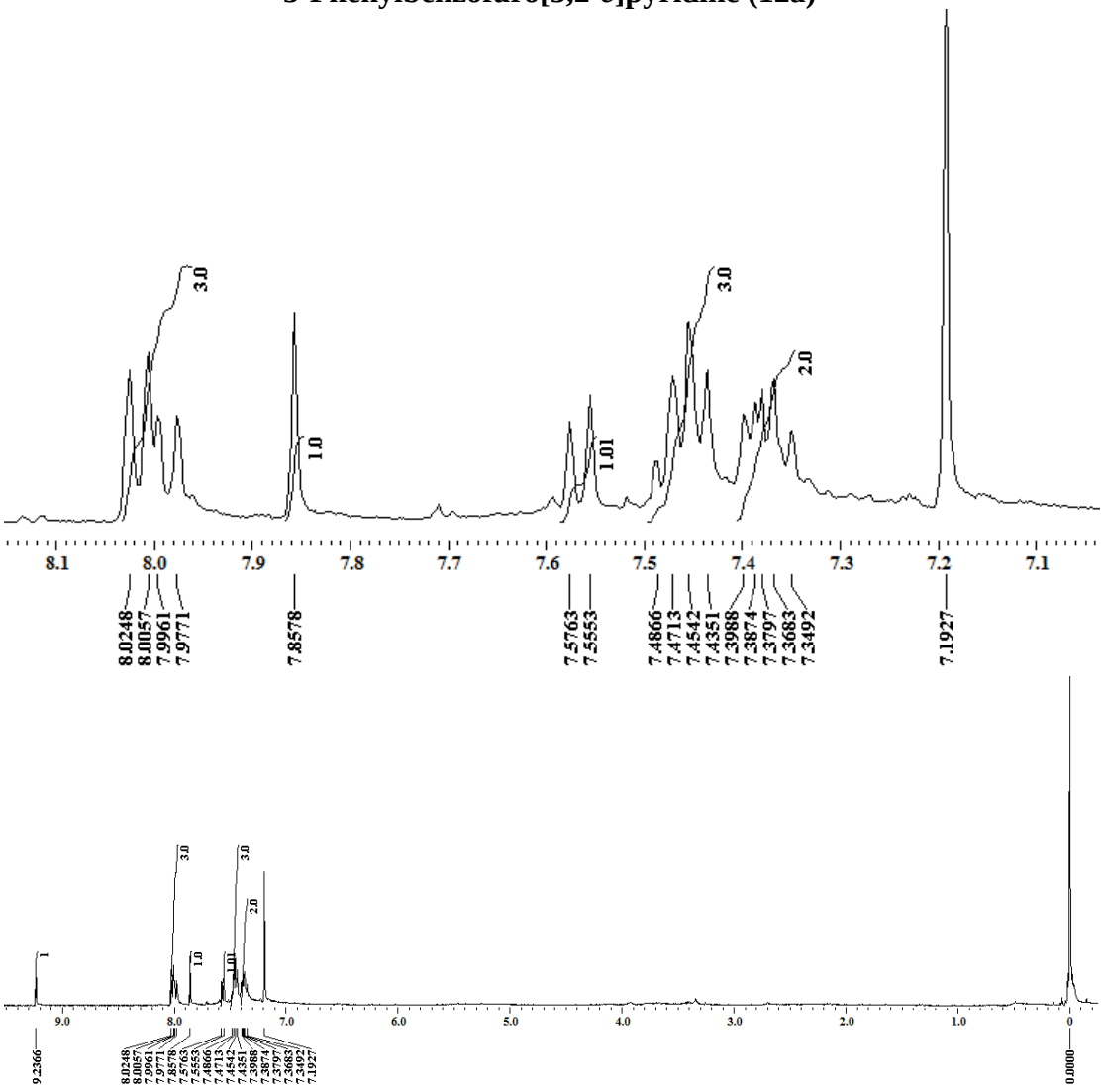
3-(4-(Trifluoromethyl)phenyl)benzo[4,5]thieno[2,3-*c*]pyridine(10d)



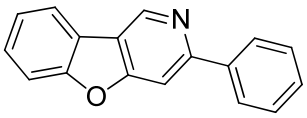
^{1}H NMR



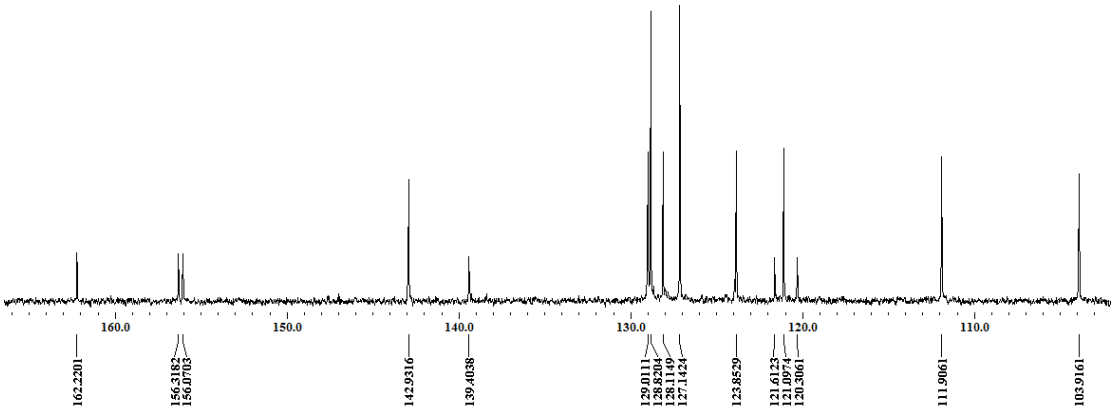
3-Phenylbenzofuro[3,2-*c*]pyridine (12a)

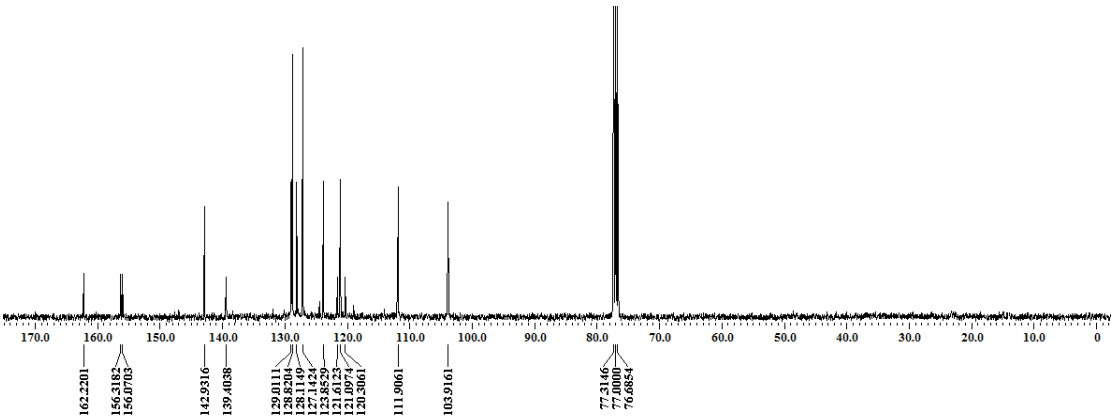


^{13}C NMR

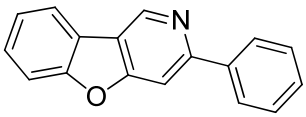


3-Phenylbenzofuro[3,2-*c*]pyridine (12a)

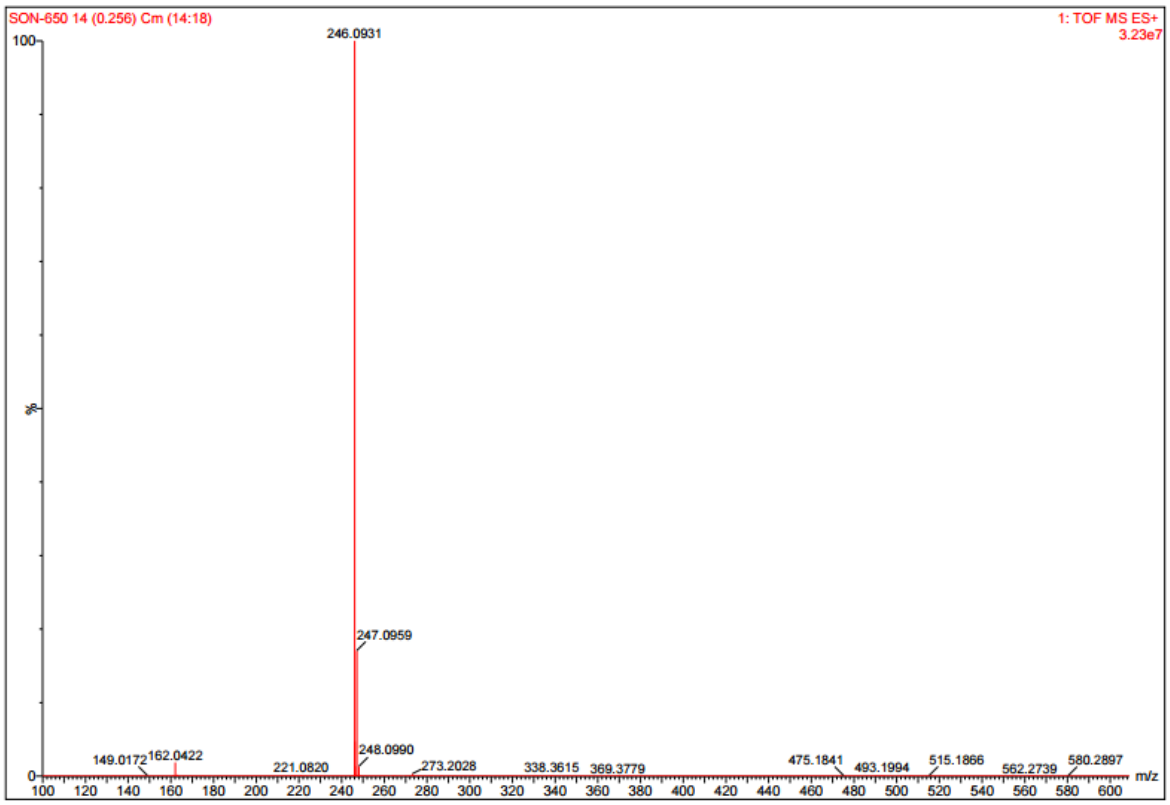




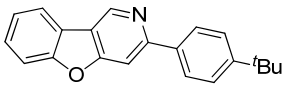
HRMS



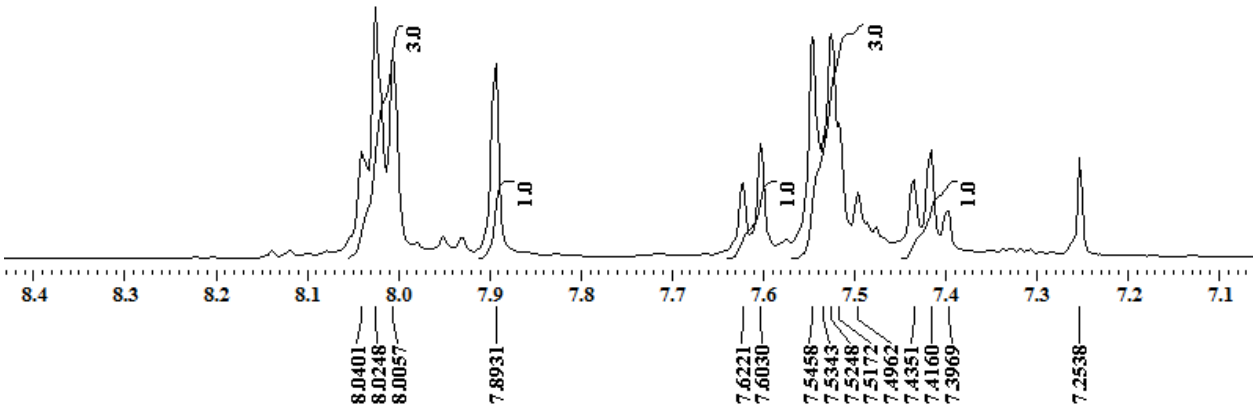
3-Phenylbenzofuro[3,2-*c*]pyridine (12a)

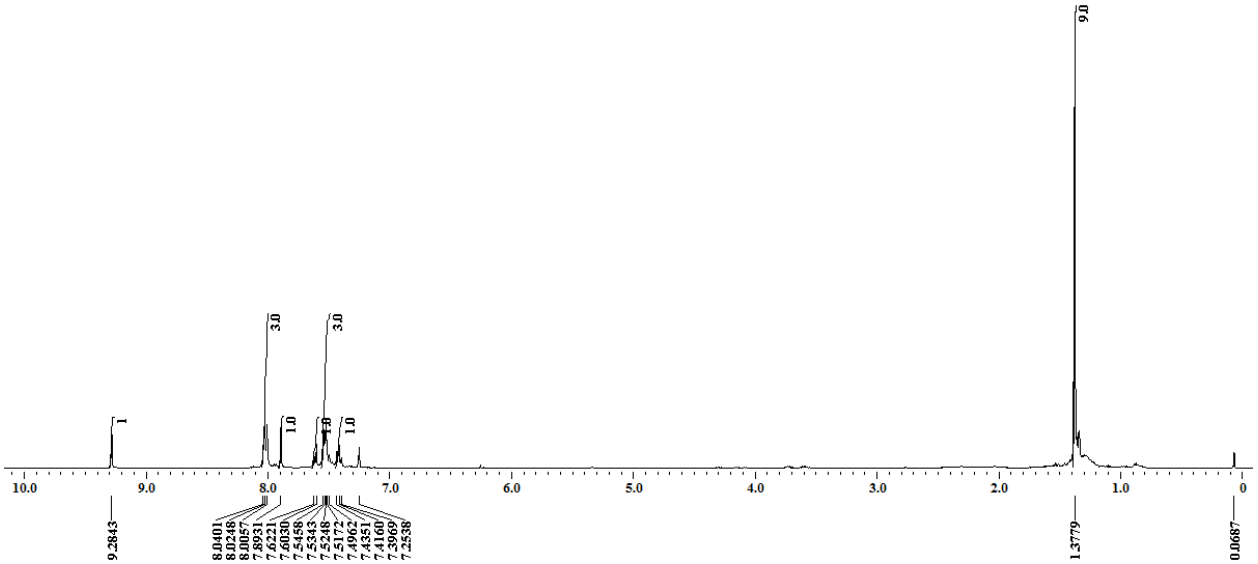


^{1}H NMR

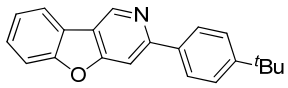


3-(4-(Tert-butyl)phenyl)benzofuro[3,2-*c*]pyridine (12b)

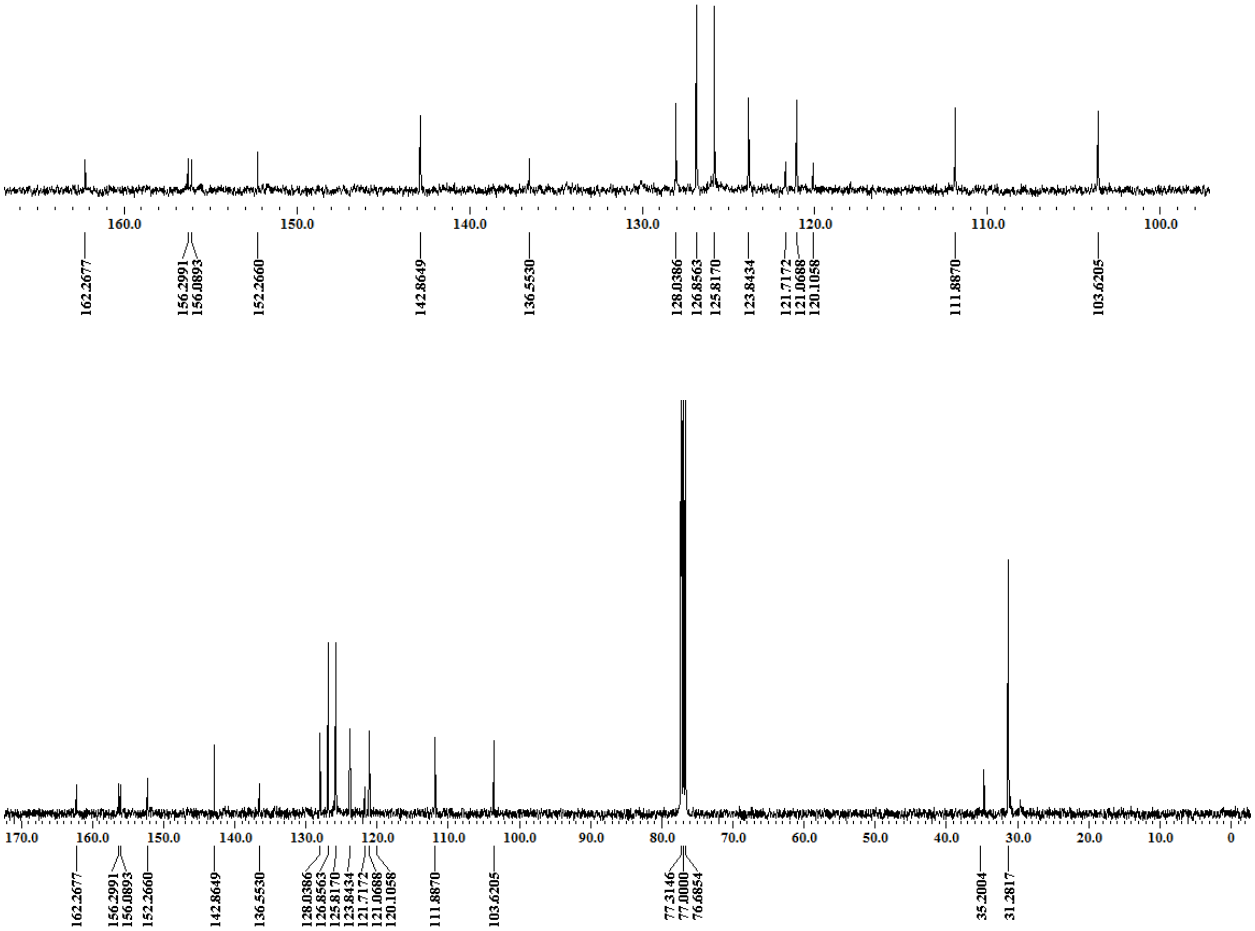




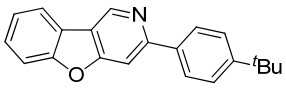
^{13}C NMR



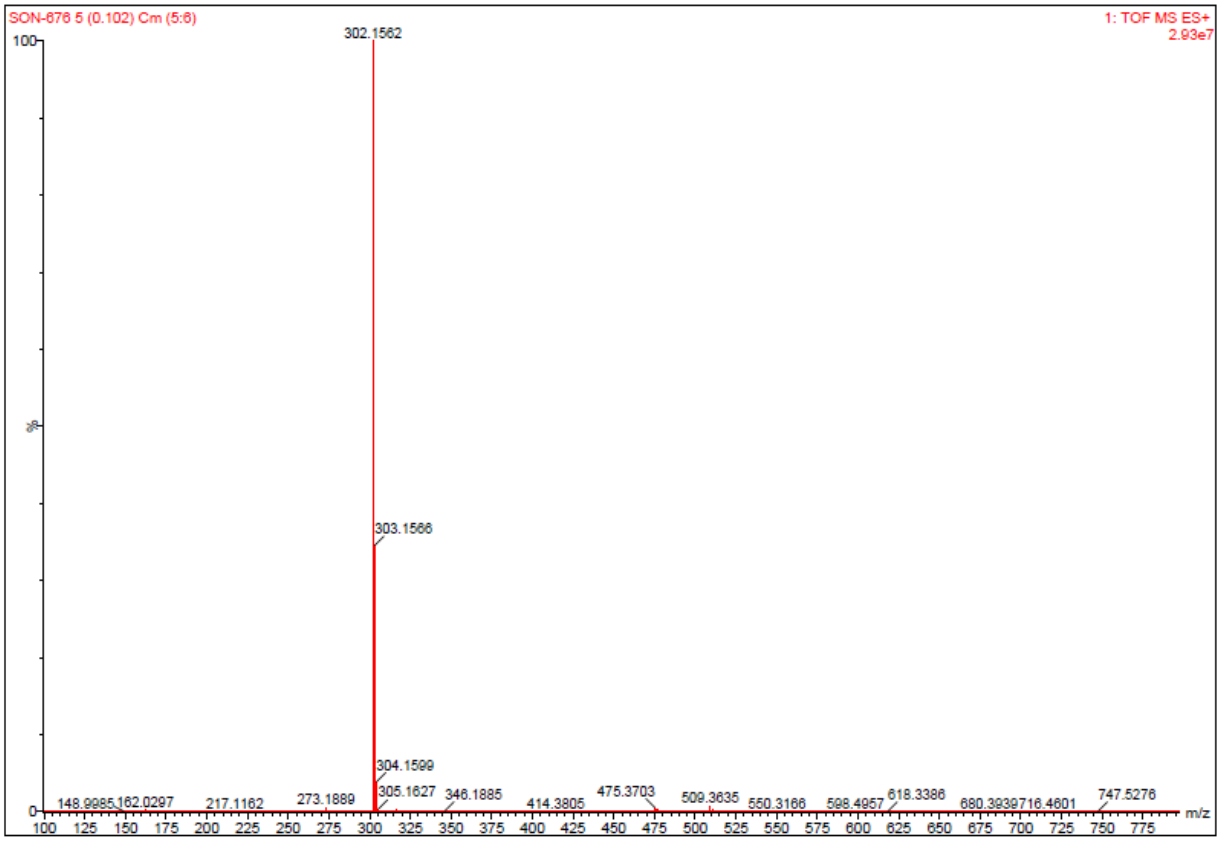
3-(4-(Tert-butyl)phenyl)benzofuro[3,2-*c*]pyridine (12b)



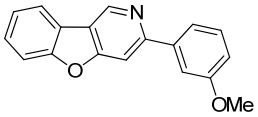
HRMS



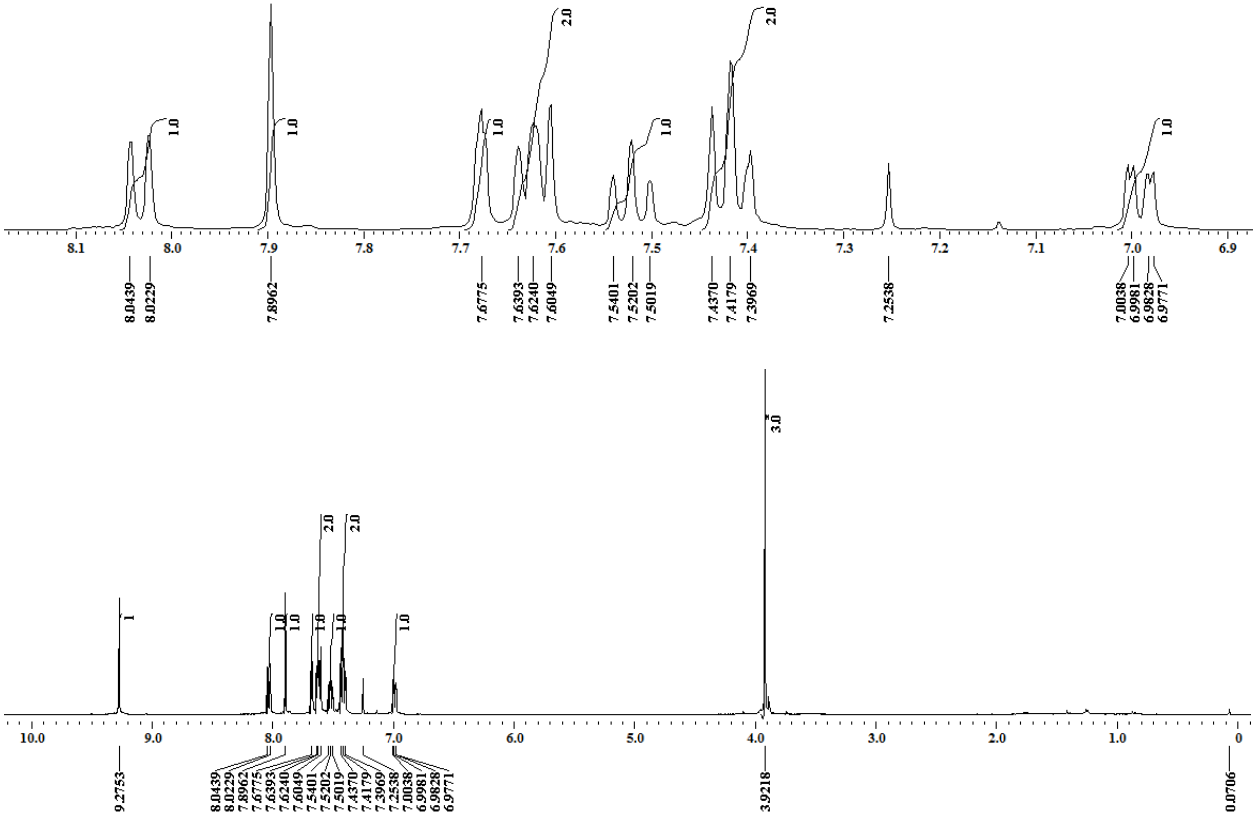
3-(4-(Tert-butyl)phenyl)benzofuro[3,2-*c*]pyridine (12b)



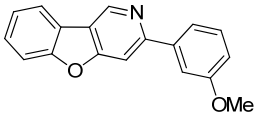
^{1}H NMR



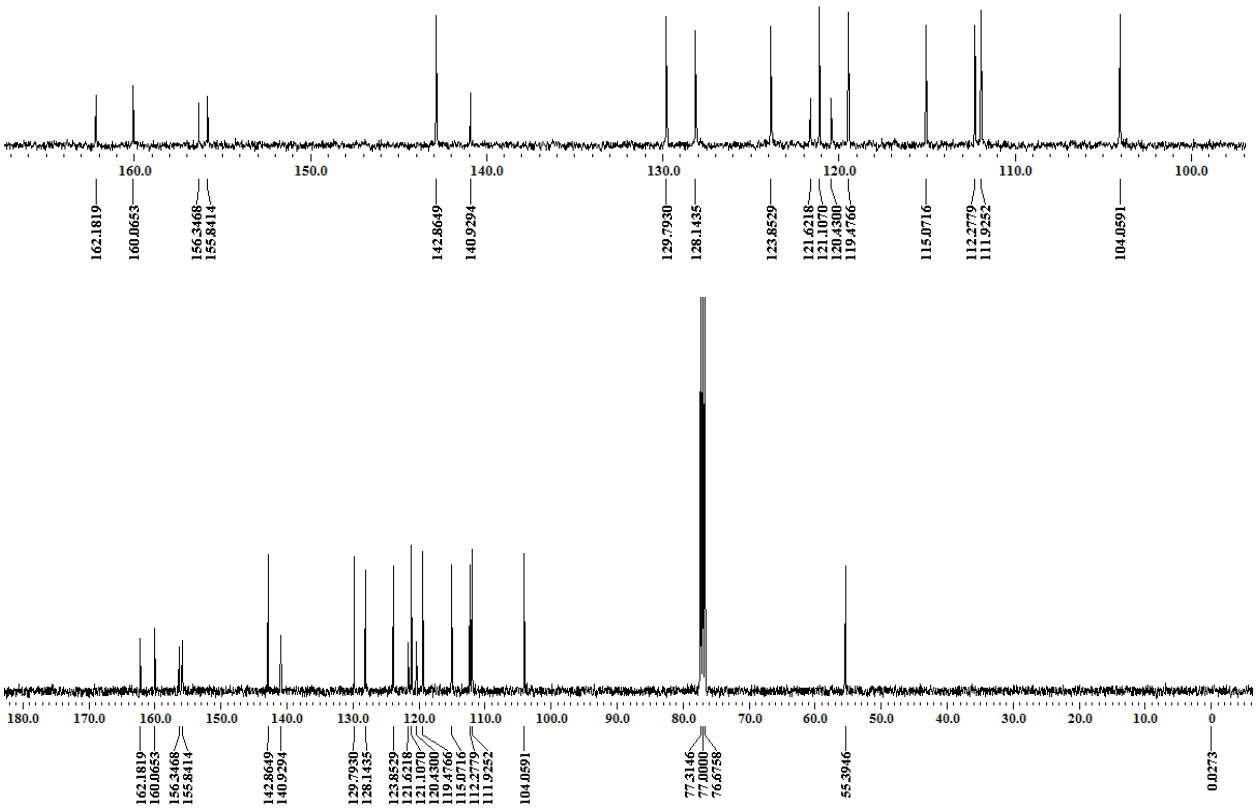
3-(3-Methoxyphenyl)benzofuro[3,2-*c*]pyridine (12c)



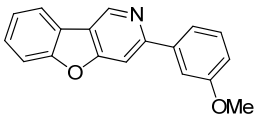
^{13}C NMR



3-(3-Methoxyphenyl)benzofuro[3,2-*c*]pyridine (12c)

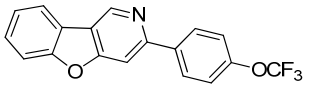


HRMS

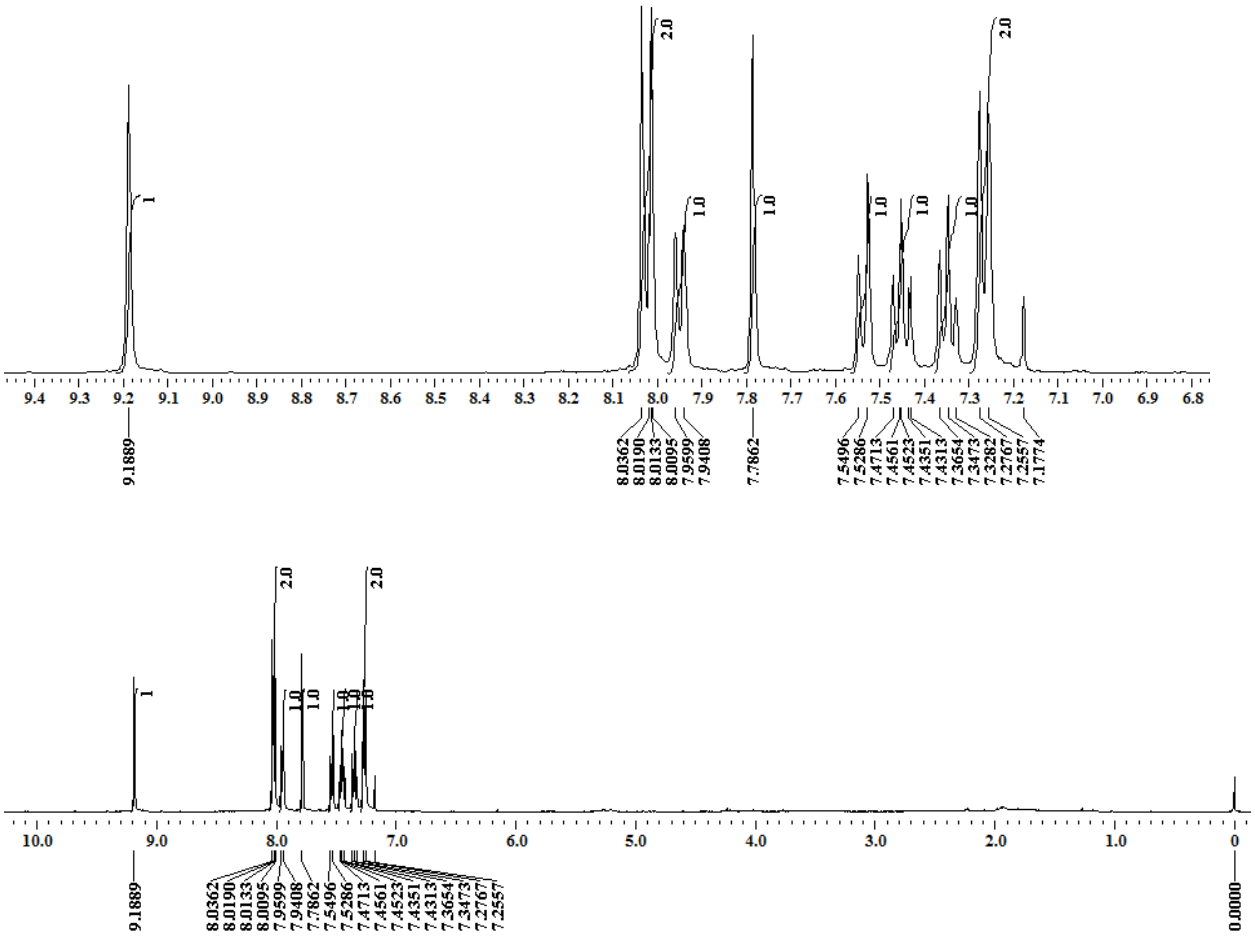


3-(3-Methoxyphenyl)benzofuro[3,2-*c*]pyridine (12c)

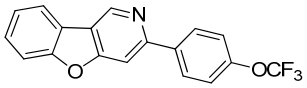
^{1}HNMR



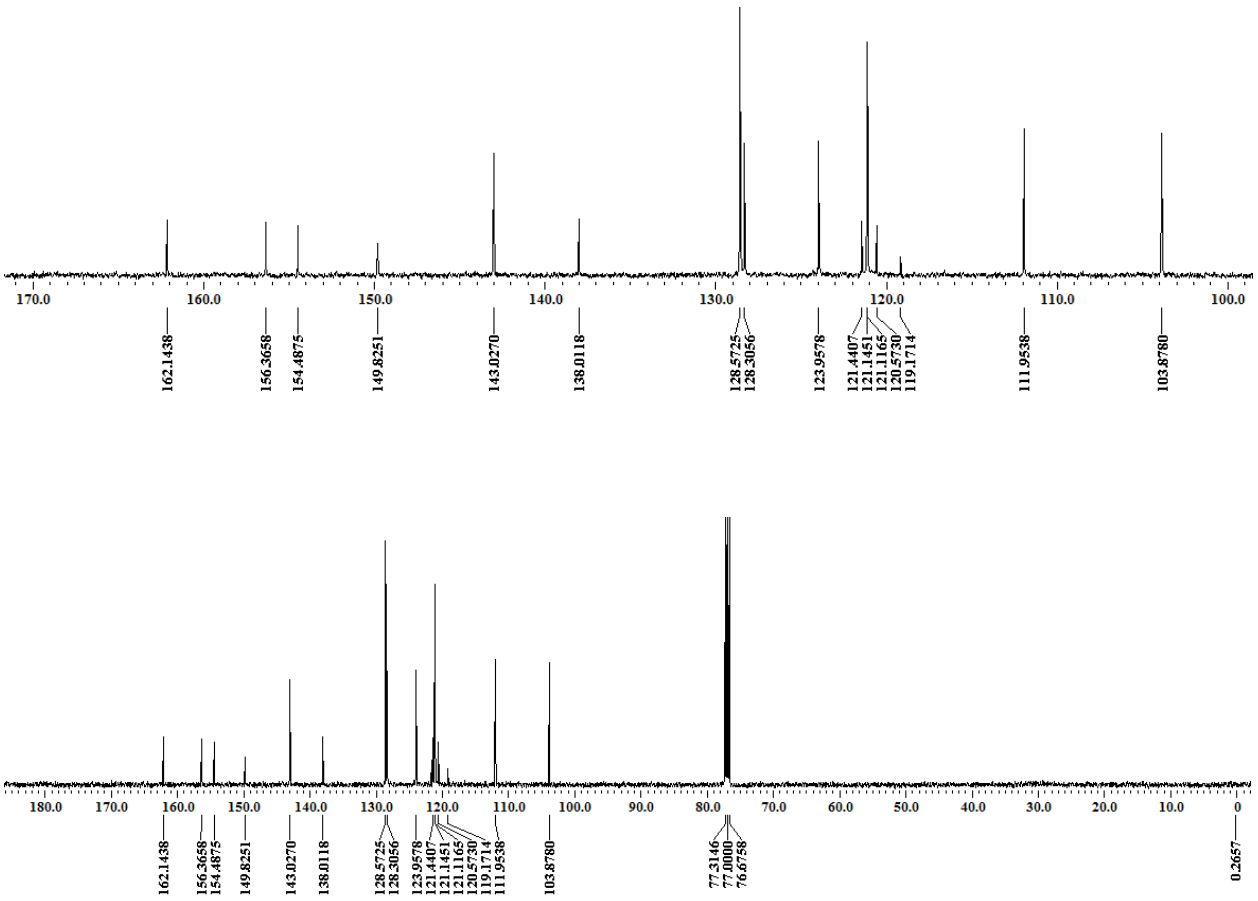
3-(4-(Trifluoromethoxy)phenyl)benzofuro[3,2-*c*]pyridine (12d)



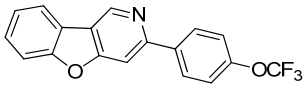
^{13}C NMR



3-(4-(Trifluoromethoxy)phenyl)benzofuro[3,2-*c*]pyridine (12d)



HRMS



3-(4-(Trifluoromethoxy)phenyl)benzofuro[3,2-*c*]pyridine (12d)