Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

One-step synthesis of 5,6-diaryl pyridine-2(1*H*)-thiones from isoflavones.

Juanjuan Wang, Zunting Zhang*, Wenli Wang, Fangfang Liu

Key Laboratory of the Ministry of Education for Medicinal Resources and Natural Pharmaceutical Chemistry, National Engineering Laboratory for Resource Development of Endangered Crude Drugs in Northwest of China, and School of Chemistry and Chemical Engineering, Shaanxi Normal University, Xi'an 710062, P.R. China

e-mail: zhangzt@snnu.edu.cn

Supporting Information

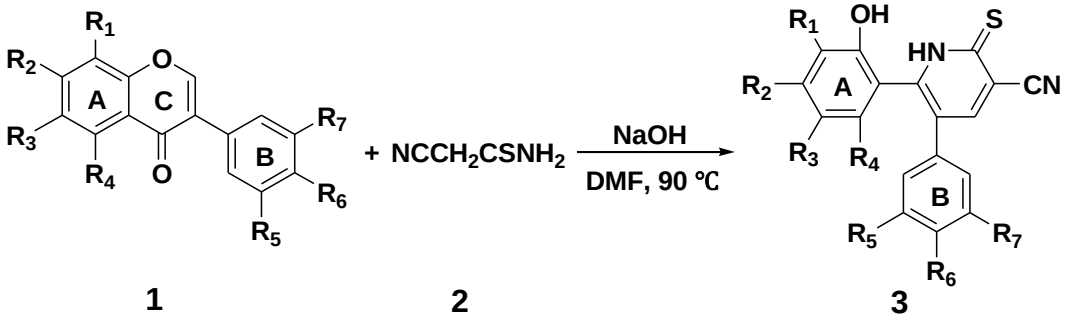
Table of Content

General Information:

^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded in DMSO-d_6 solution, using tetramethylsilane (TMS) as an internal standard unless otherwise noted. HRMS spectra were recorded using ESI technique and IR spectra were recorded in KBr pellet. Melting points were measured on micro melting point apparatus and are uncorrected. TLC was performed on Merck kieselgel 60-F_{254} and products were detected with 254 nm light. Merck kieselgel 60 (230-400 mesh) was used for column chromatography.

General Procedure for the Synthesis of 3-Cyano-5,6 Pyridine-2(1*H*)-thiones (3a-u):

The corresponding isoflavones **1a-u** (1 mmol), cyanothioacetamide **2** (200 mg, 2 mmol), and solid NaOH (140 mg, 3.5 mmol) were reacted in DMF at 90 ℃(10 mL) for 5-7 h. The progress of the reactions was monitored by TLC till the disappearance of **1a-u**. The reaction mixture was poured into water (40 mL) and acidified with 2% HCl/H_2O to the neutral pH. The precipitated yellow solid was filtered off and was purified on silica gel column ($CHCl_3$/MeOH, 100:1) to give the corresponding products **3a-u** as colorless amorphous powders.



1 **2** **3**

Crystal and structure refinement data for 3a dimmer(crystallized from acetone)

X-Ray Crystallography. The crystallographic data of compound 3a dimmer is collected in Table 1. Details of the structure investigations are available on request from the Cambridge Crystallographic Data Centre, on quoting the depository numbers.

X-ray crystal structure of 3a dimmer(crystallized from acetone)

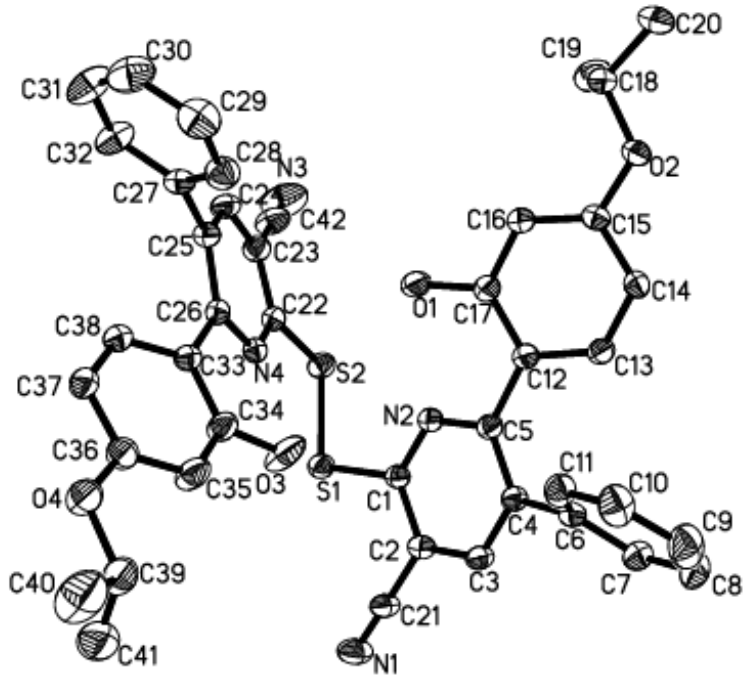
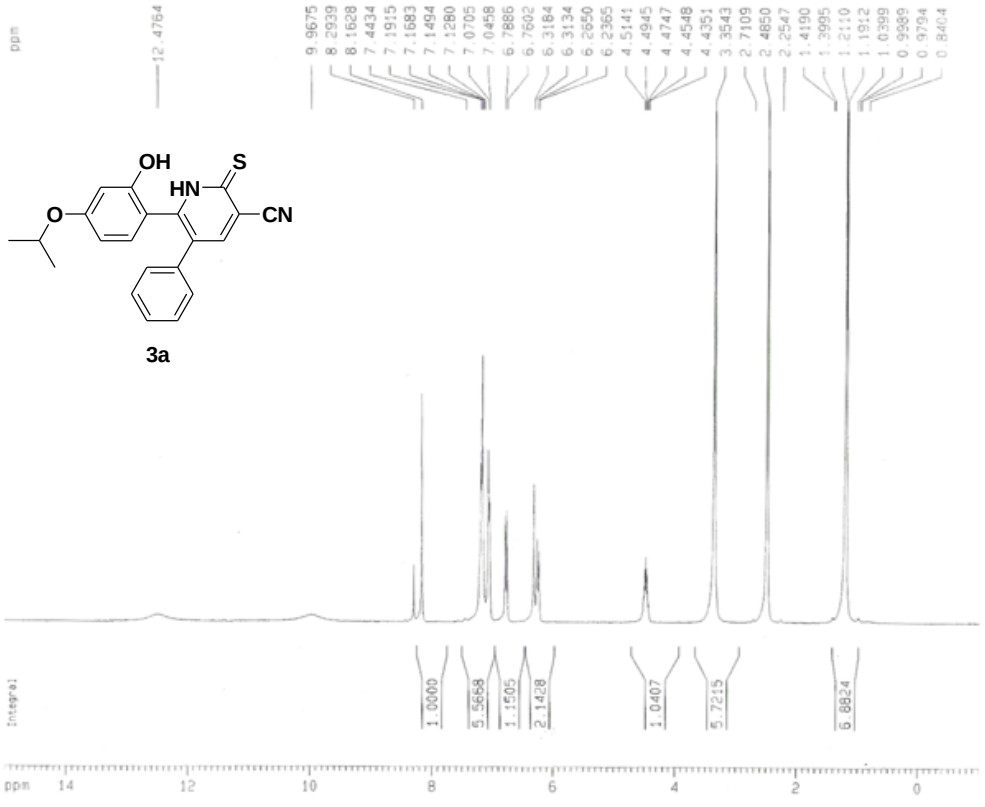


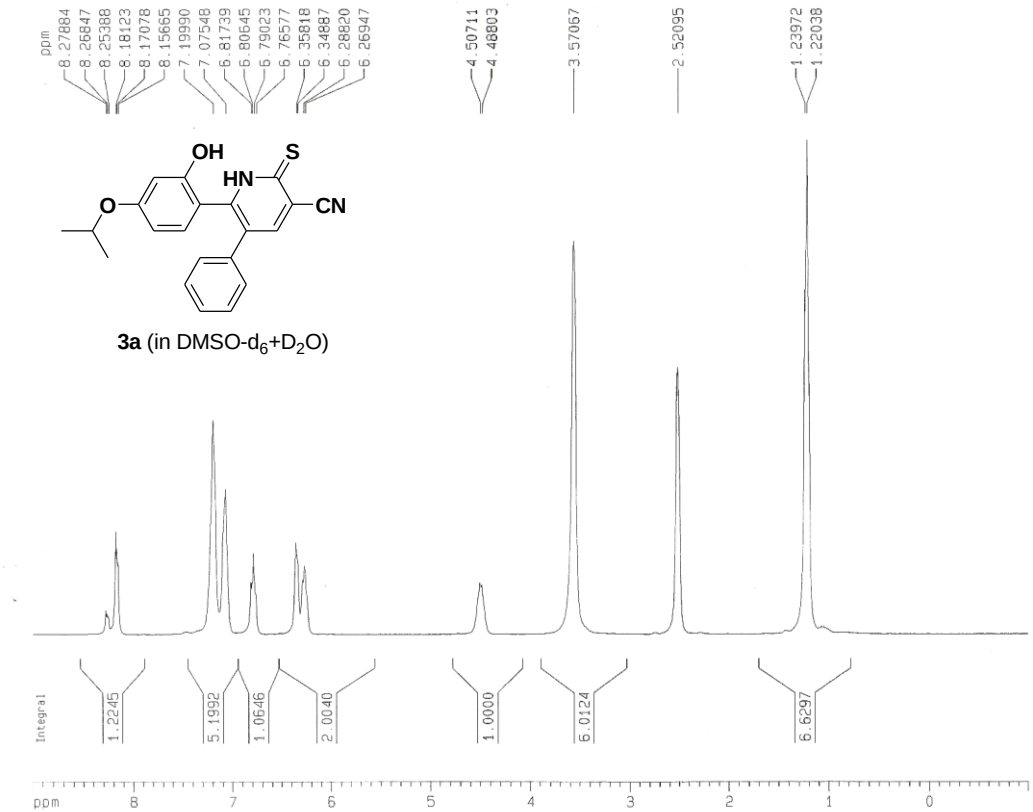
Table 1 The crystallographic data of compound **3a** dimmer

Compound 3a dimmer	**Data**
Empirical formula	$C_{21}H_{17}N_2O_2S$
Formula weight	361.43
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 11.5891(16) Å, b = 13.0949(18)Å
	c = 14.337(3) Å, β = 98 .964(3)°
Volume	1860.8(6)Å^3
Z, Density (calculated)	4, 1.290 Mg/m^3
Absorption coefficient	0.191 mm^{-1}
F(000)	892
Crystal size	0.38×0.30×0.27 mm
θ range for data collection	1.52-25.10°
Limiting indices	$-12 \le h \le 13$, $-15 \le k \le 15$, $-17 \le l \le 12$
Reflections collected / unique	9425 / 6555 [R(int) = 0.0230]
Completeness to θ = 25.10	99.0 %
Absorption correction	None
Max. and min. transmission	0.9511 and 0.9311
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6555 / 0 / 475
Goodness-of-fit on F^2	1.029
Final R indices [I > 2sigma(I)]	$R1$ = 0.0503, $\omega R2$ = 0.1388
R indices (all data)	$R1$ = 0.0741, $\omega R2$ = 0.1698
Extinction coefficient	0.005(3)
Largest diff. peak and hole	0.375 and -0.353 e.A^{-3}

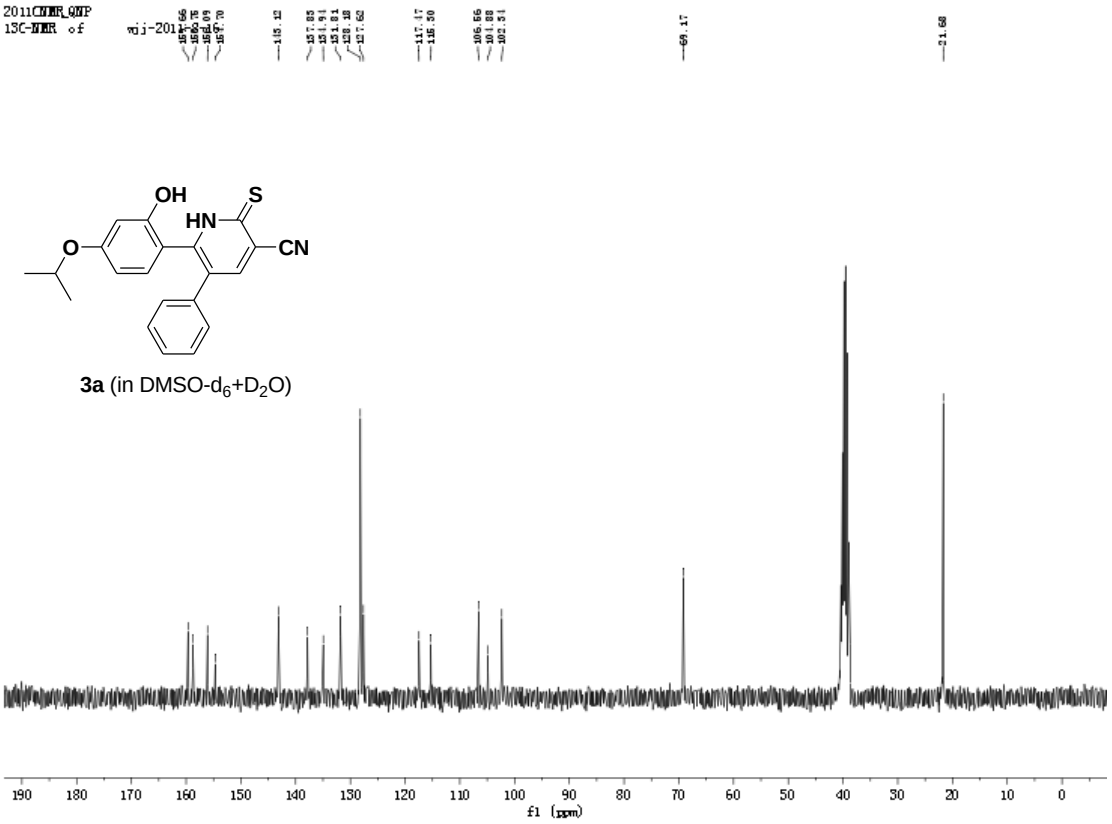
1H and 13C NMR spectra:



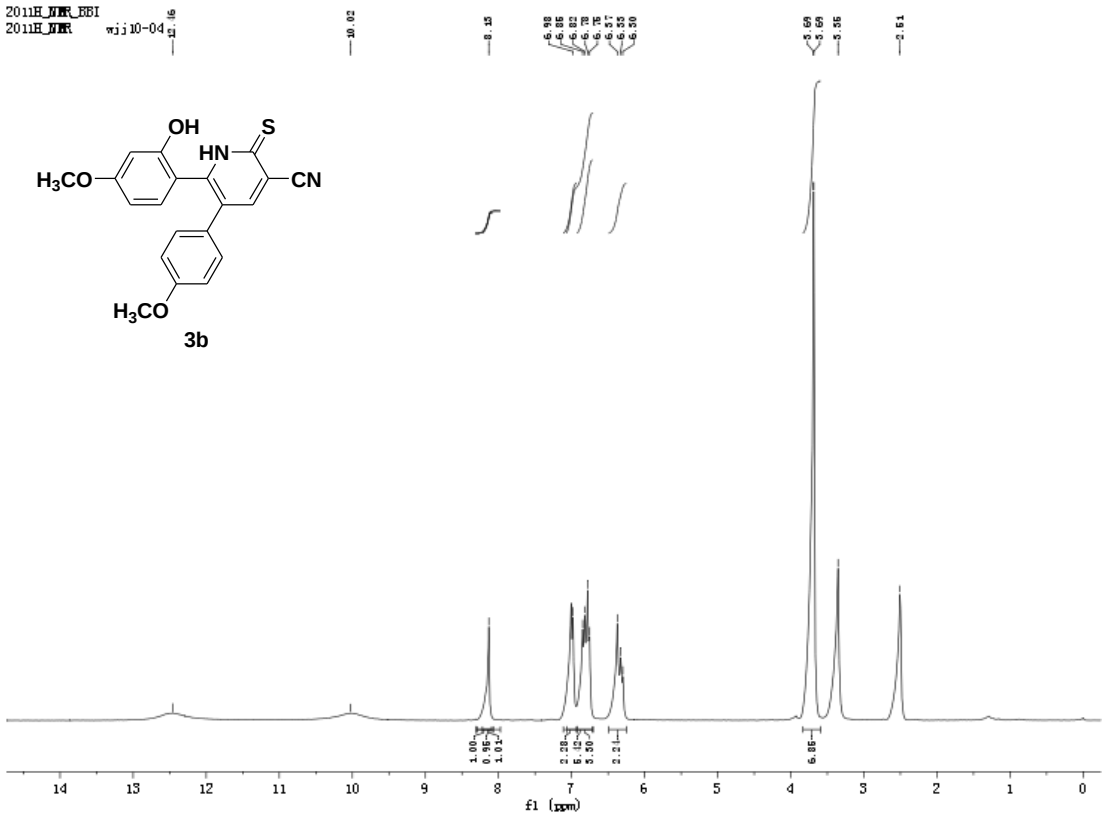
3a

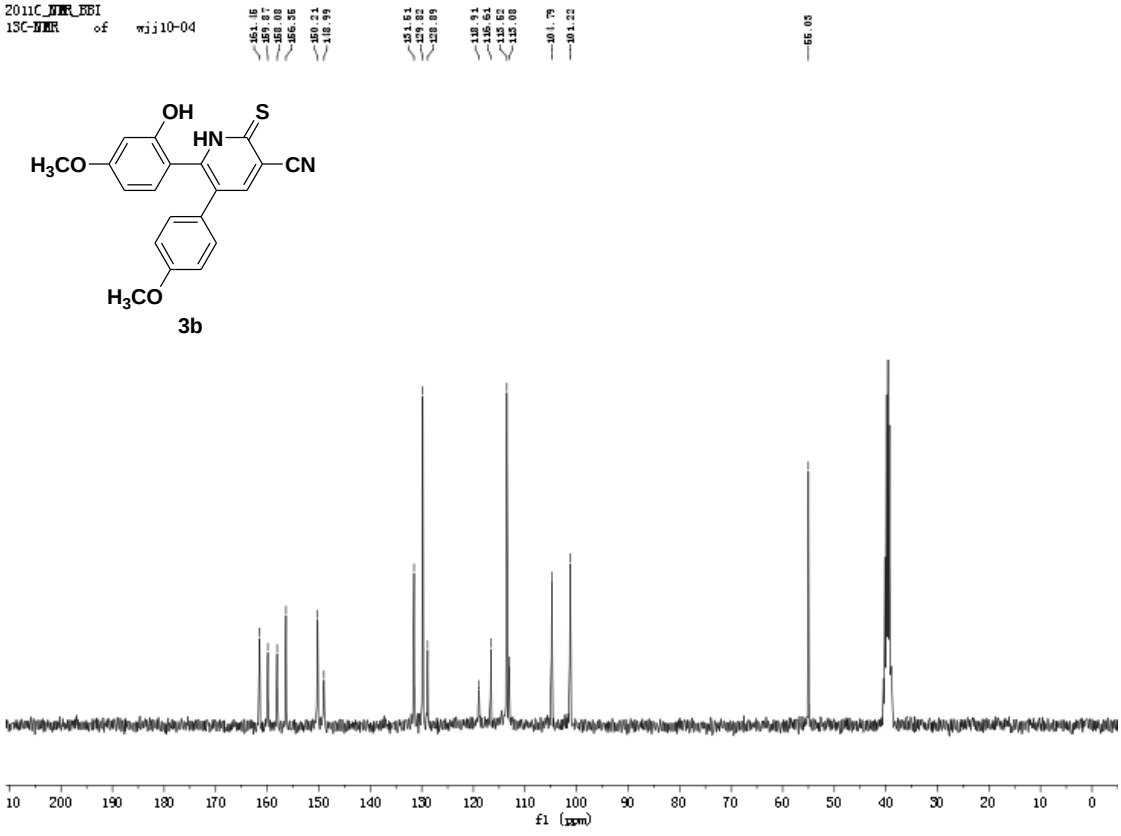


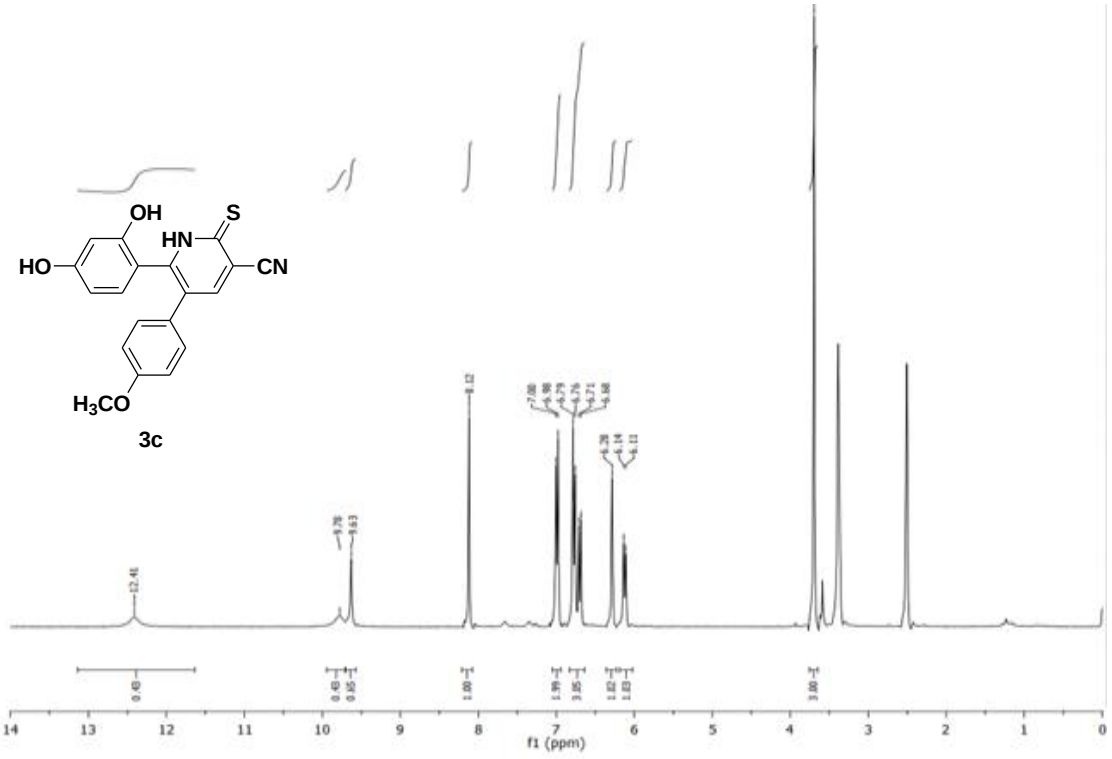
3a (in DMSO-d_6+D_2O)



3a (in DMSO-d_6+D_2O)



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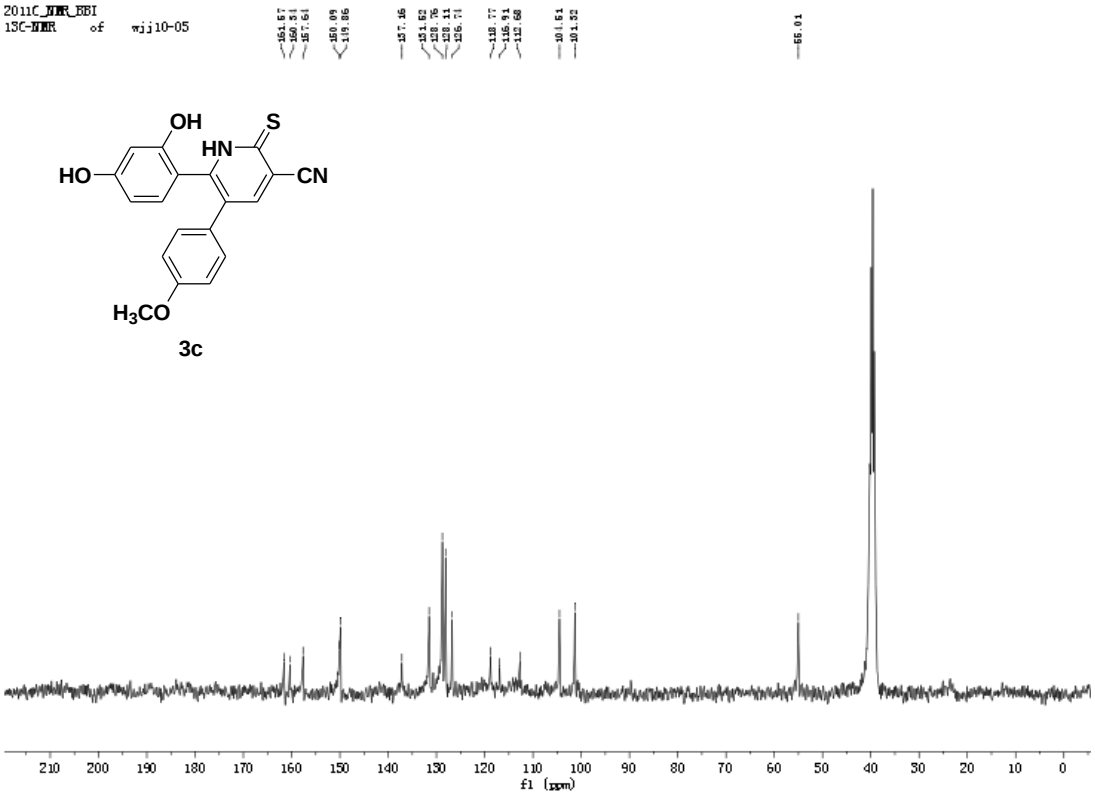
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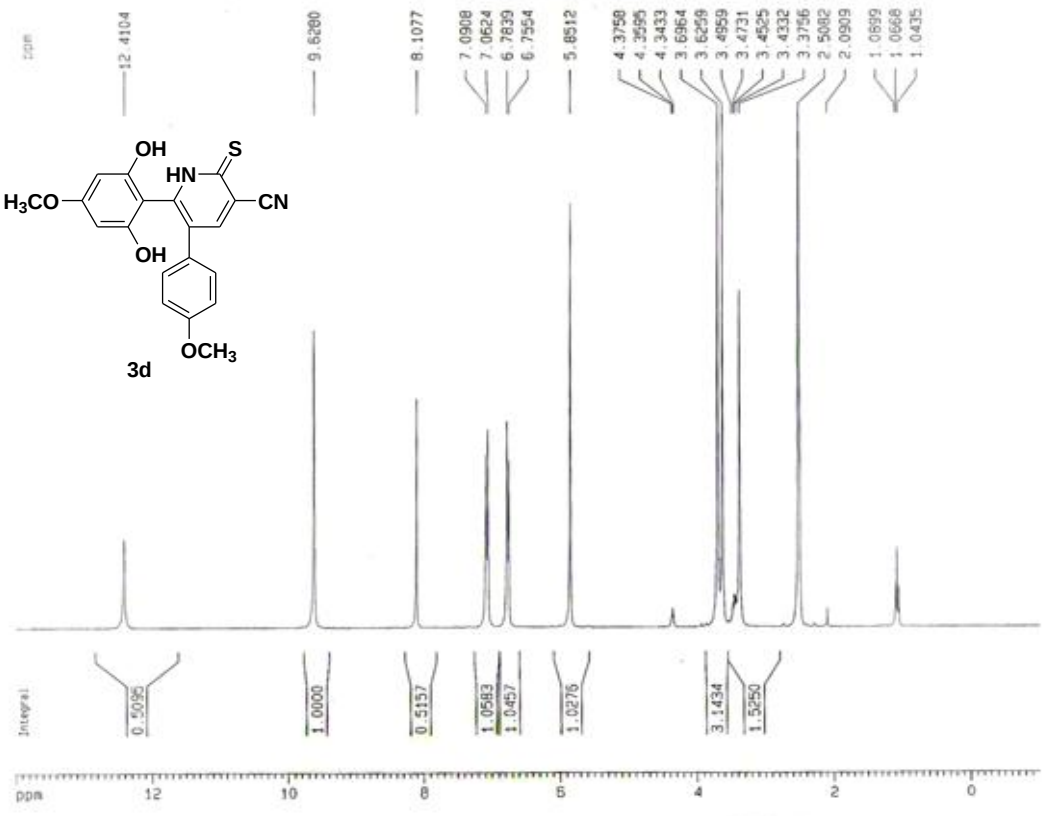

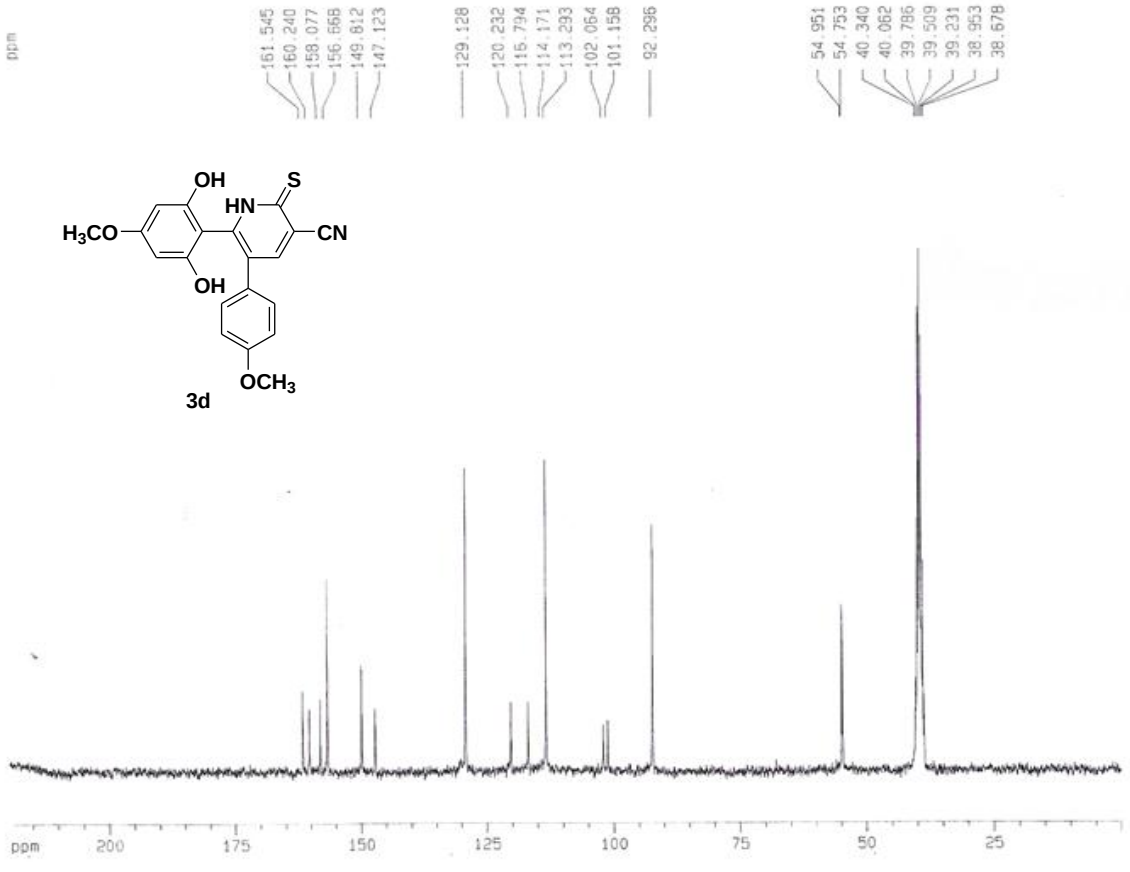

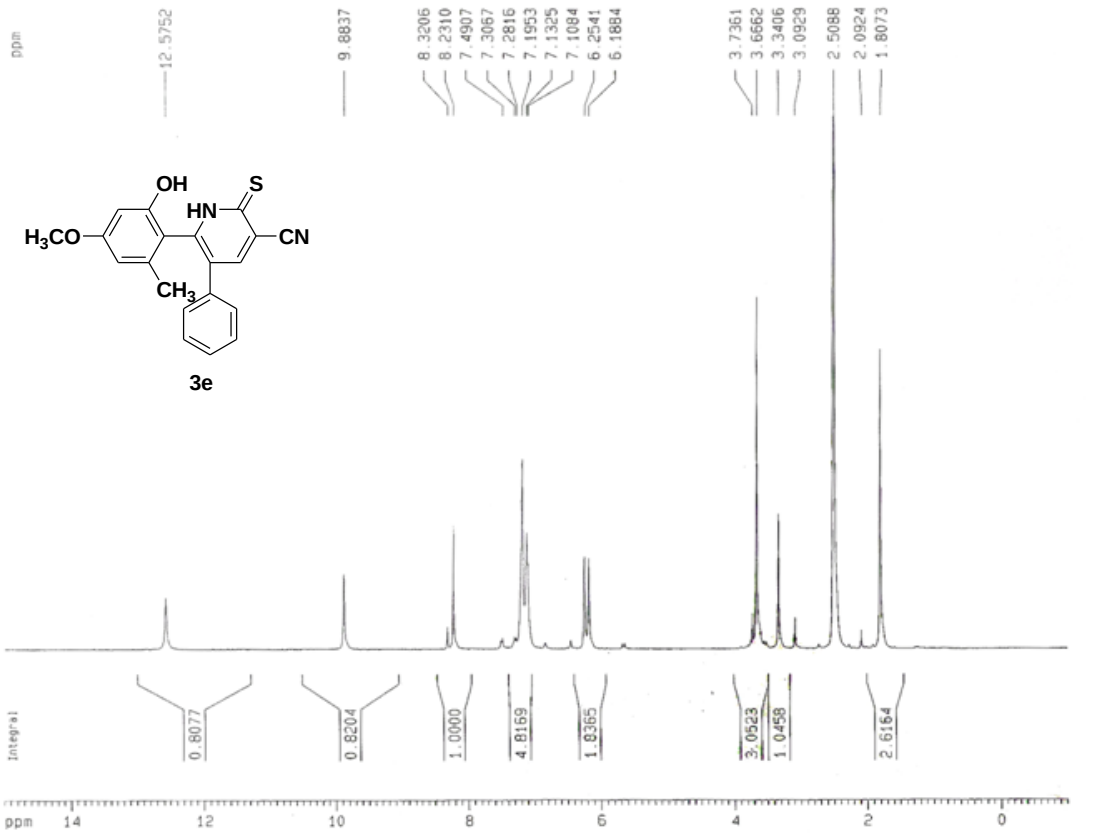
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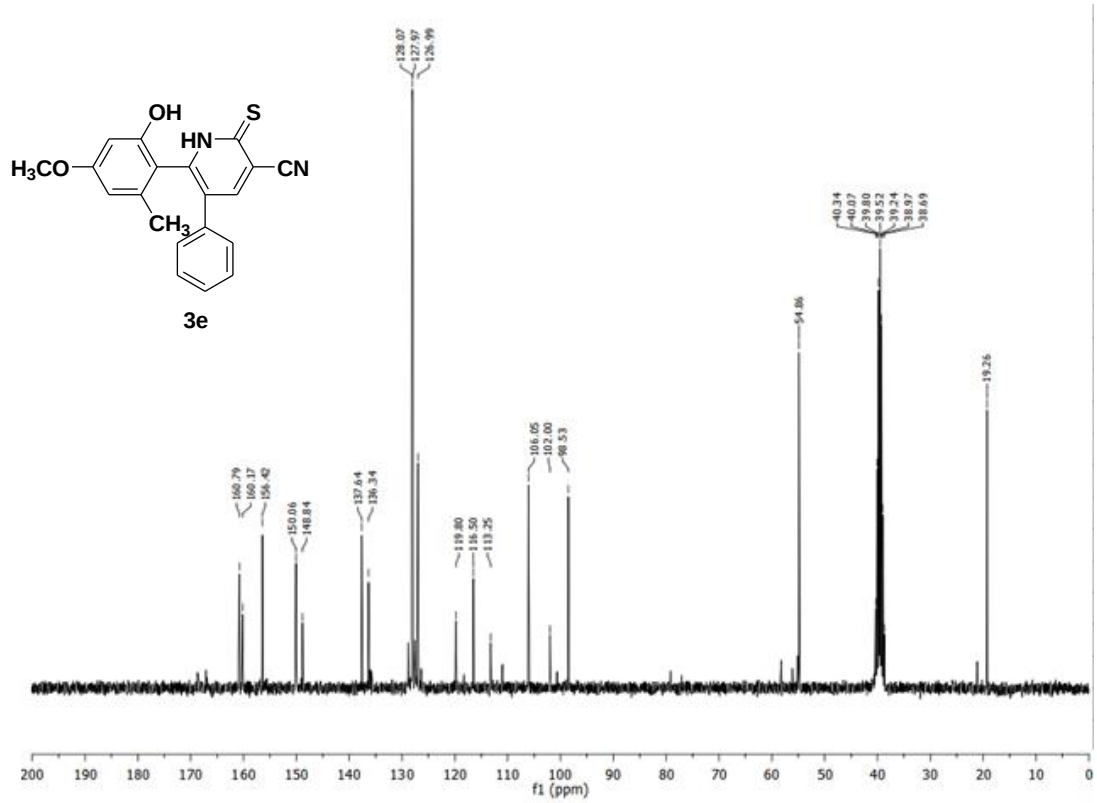
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40.07
39.80
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39.24
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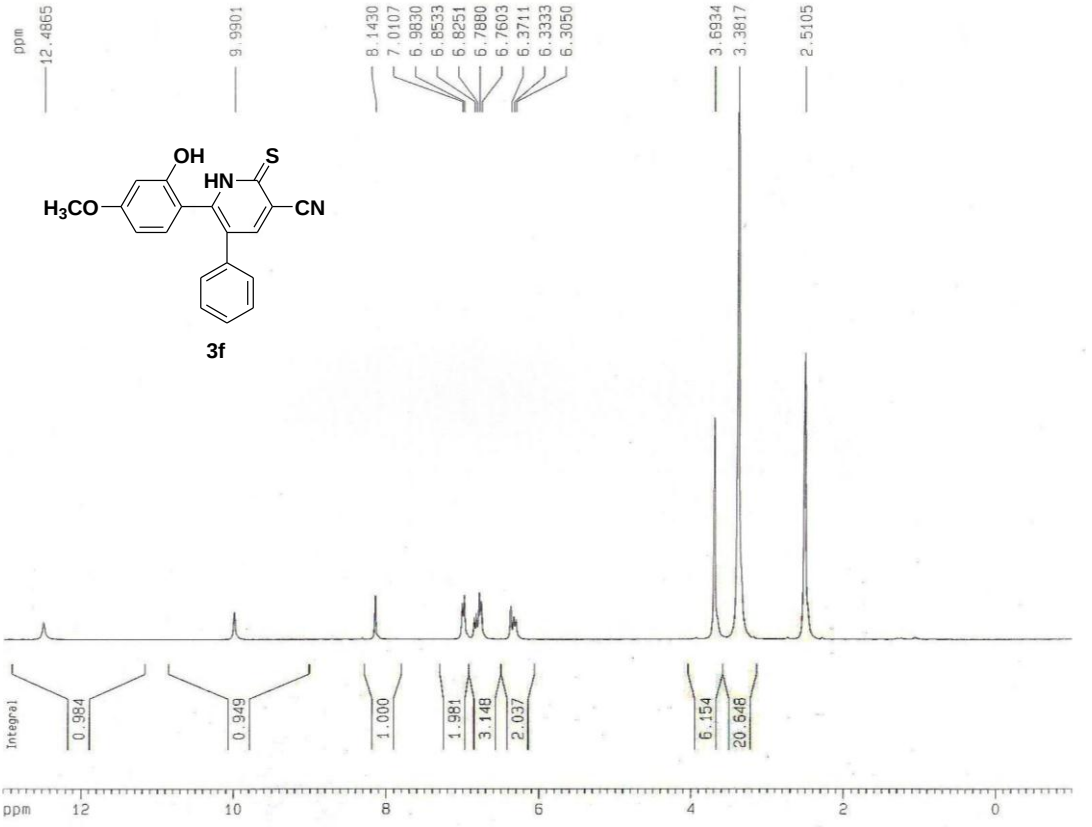

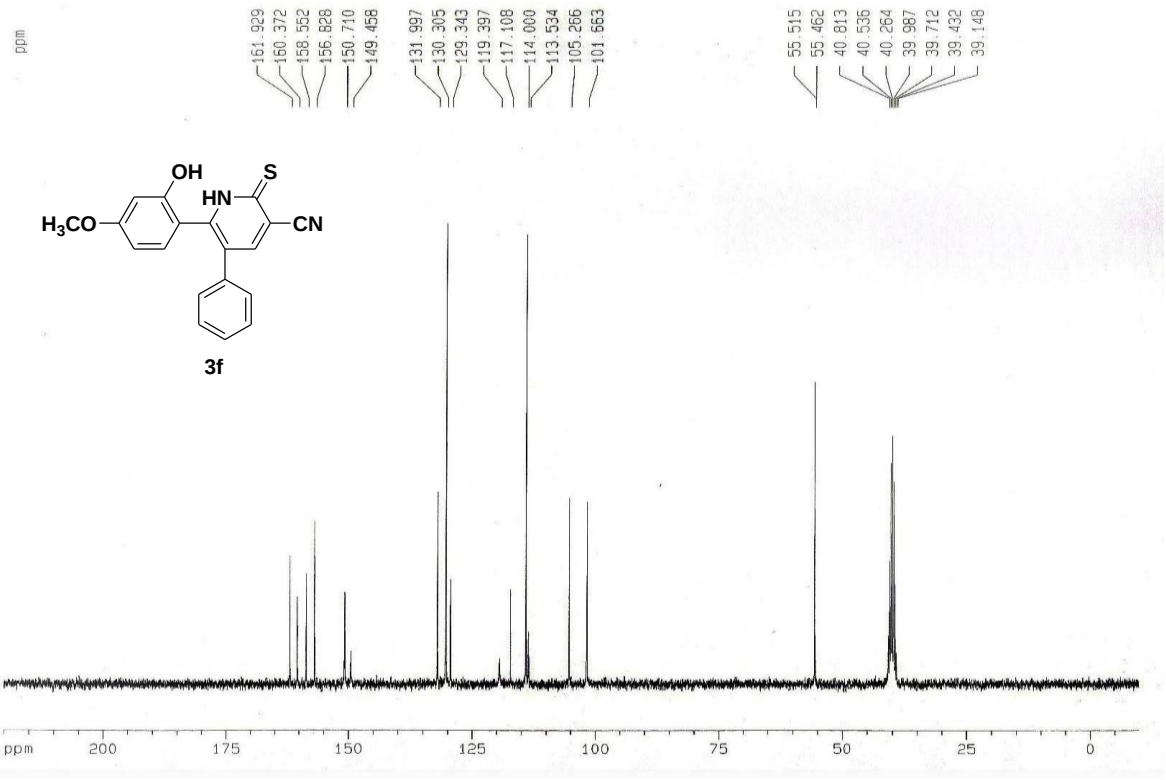

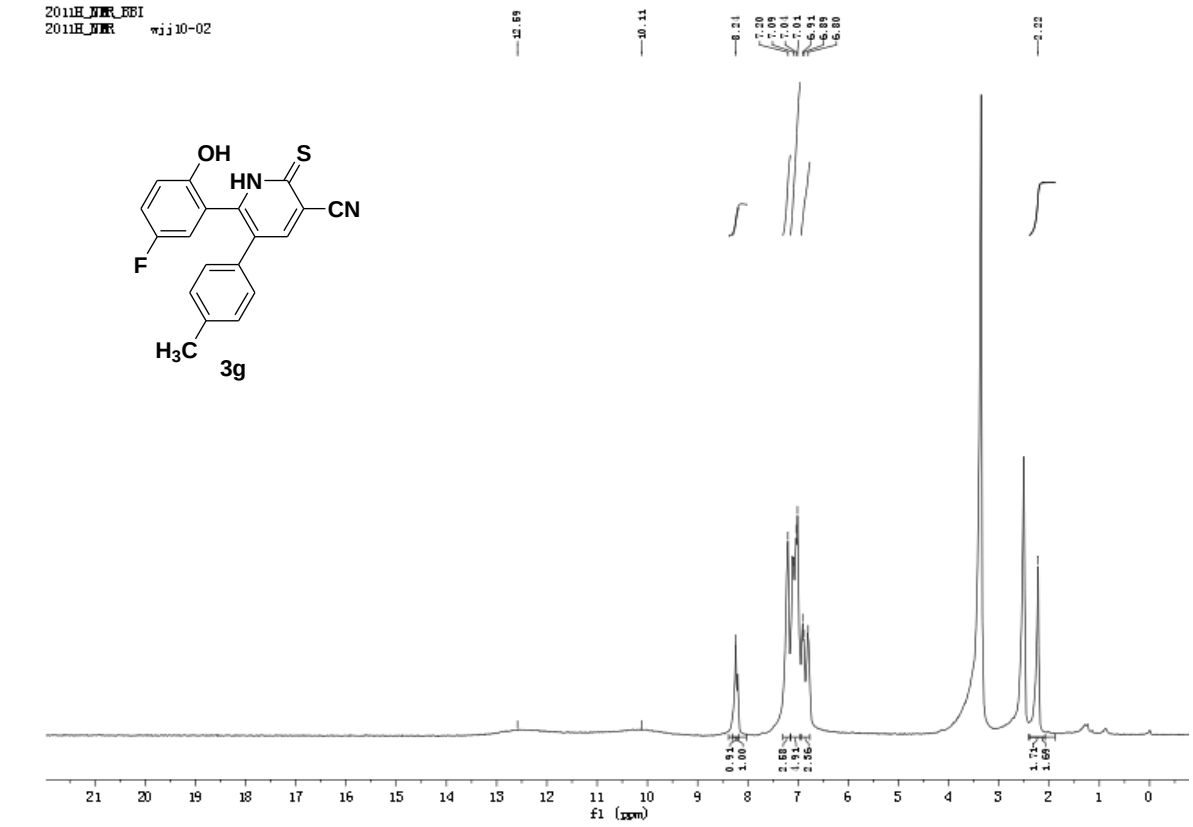
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F
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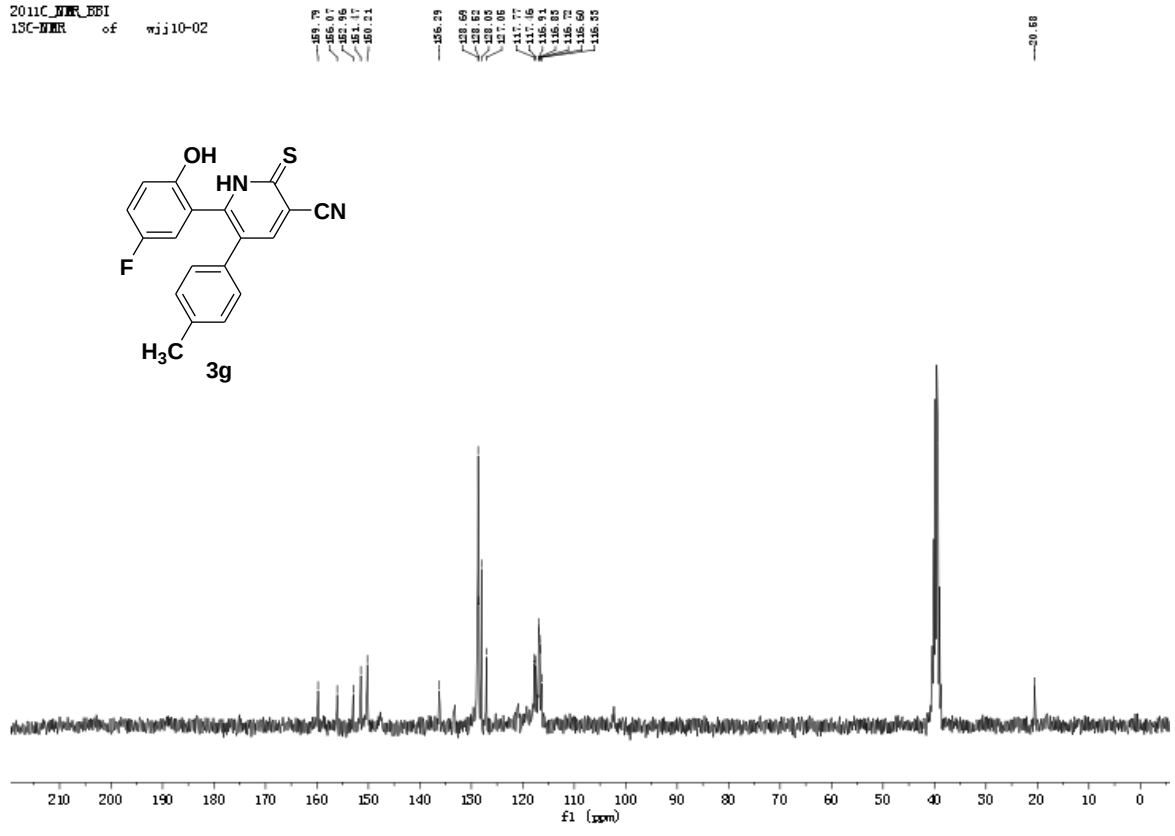
f1 (ppm)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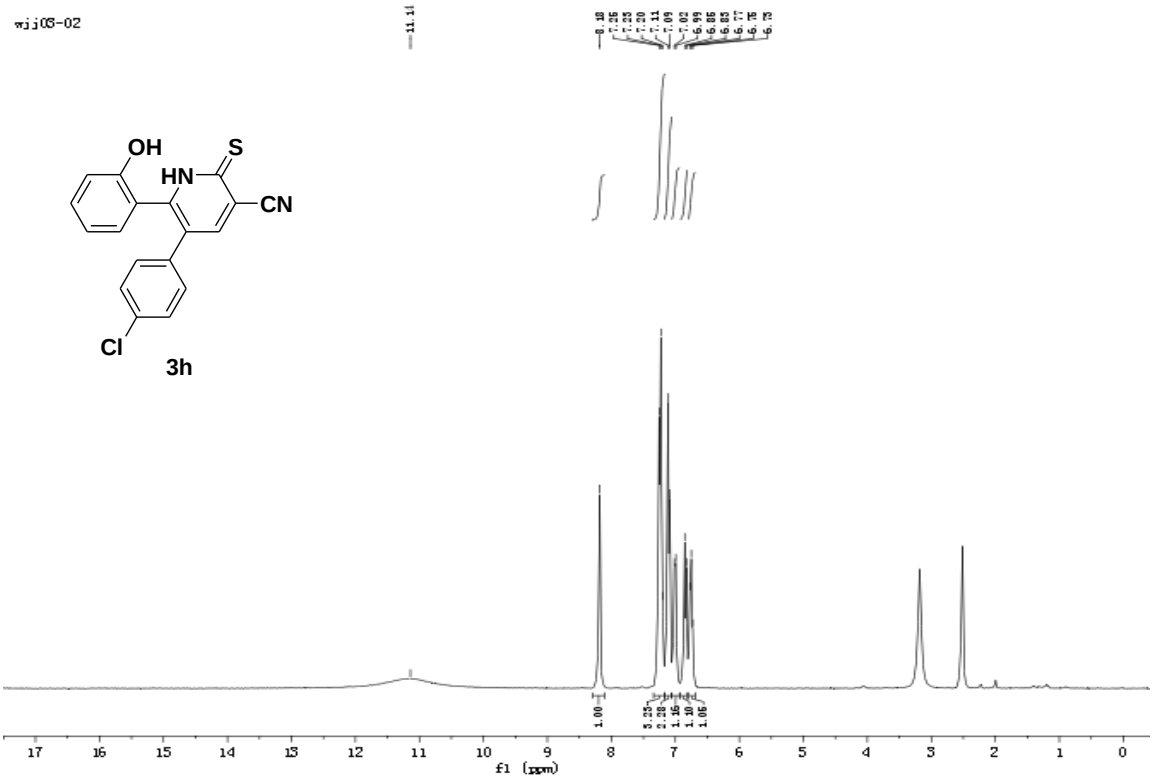
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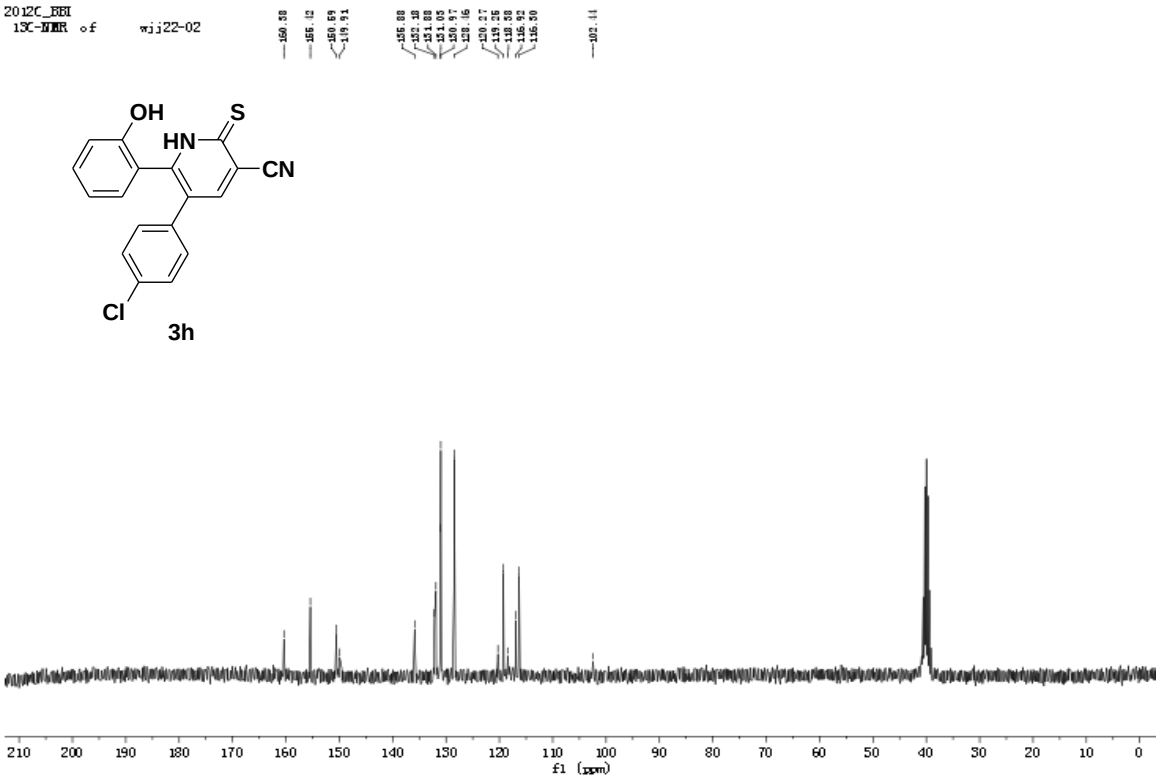
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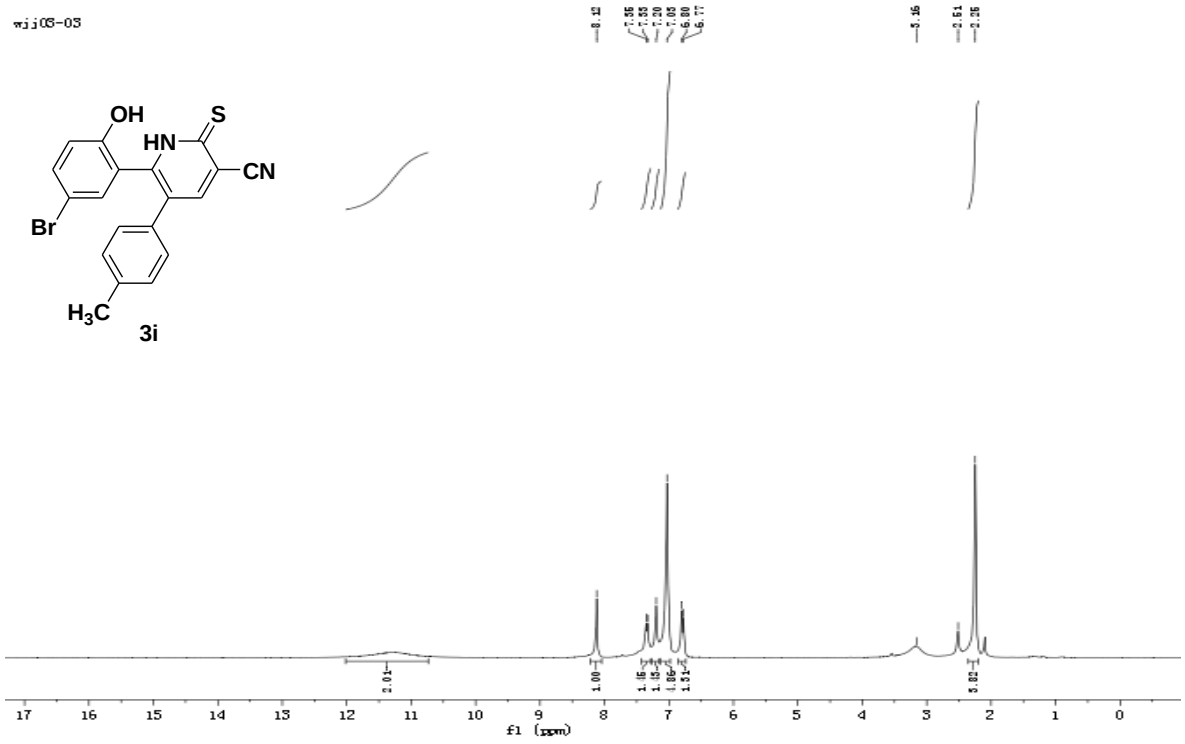
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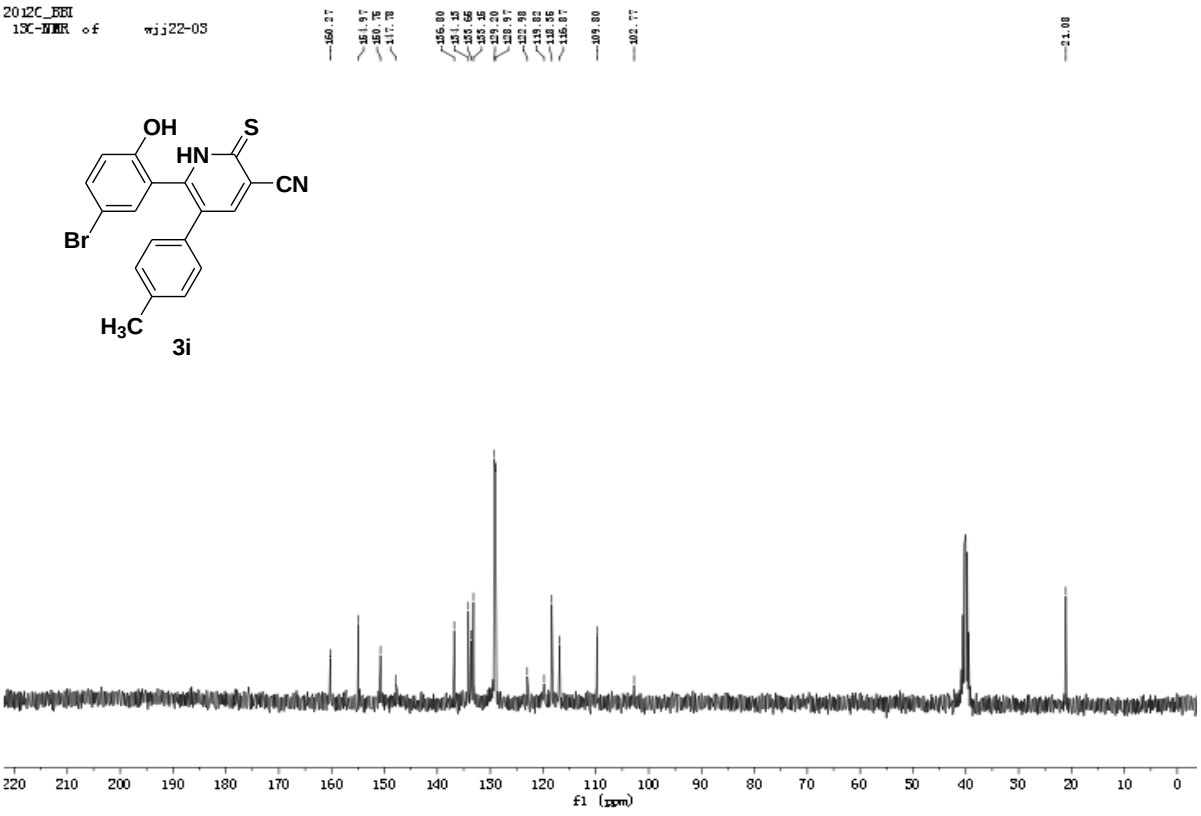
3i
f1 (ppm)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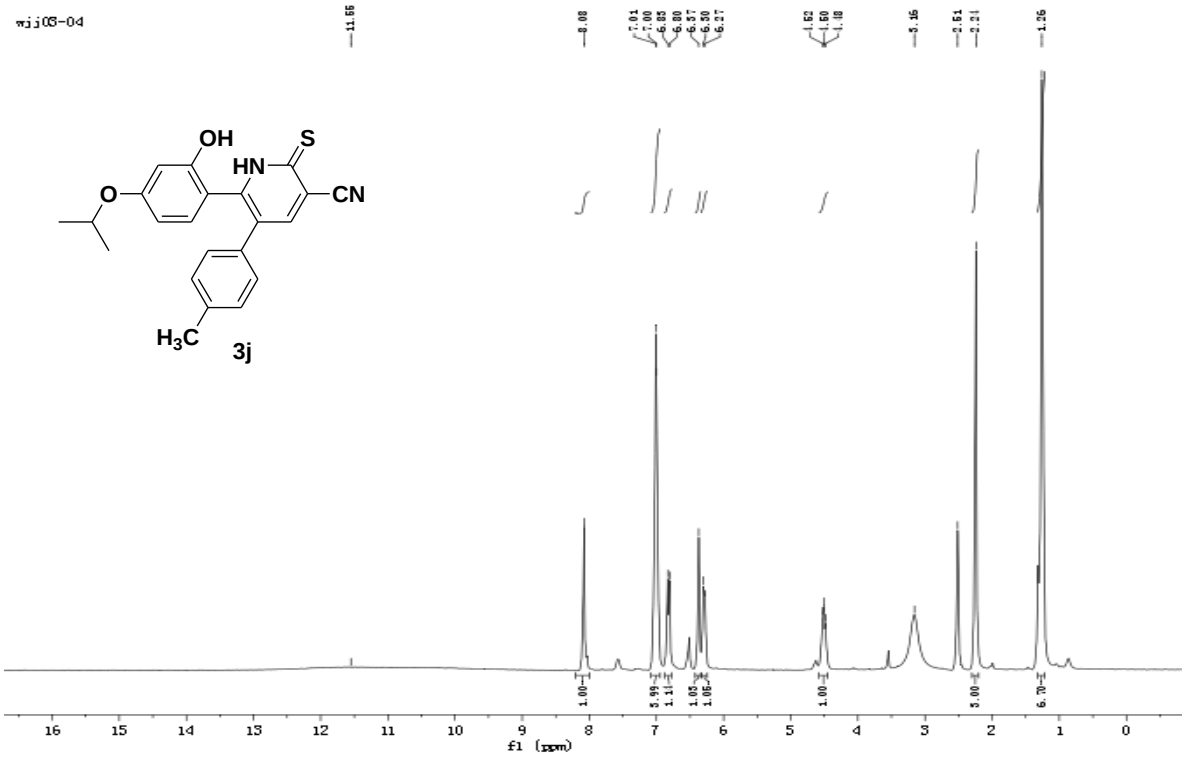
f1 (ppm)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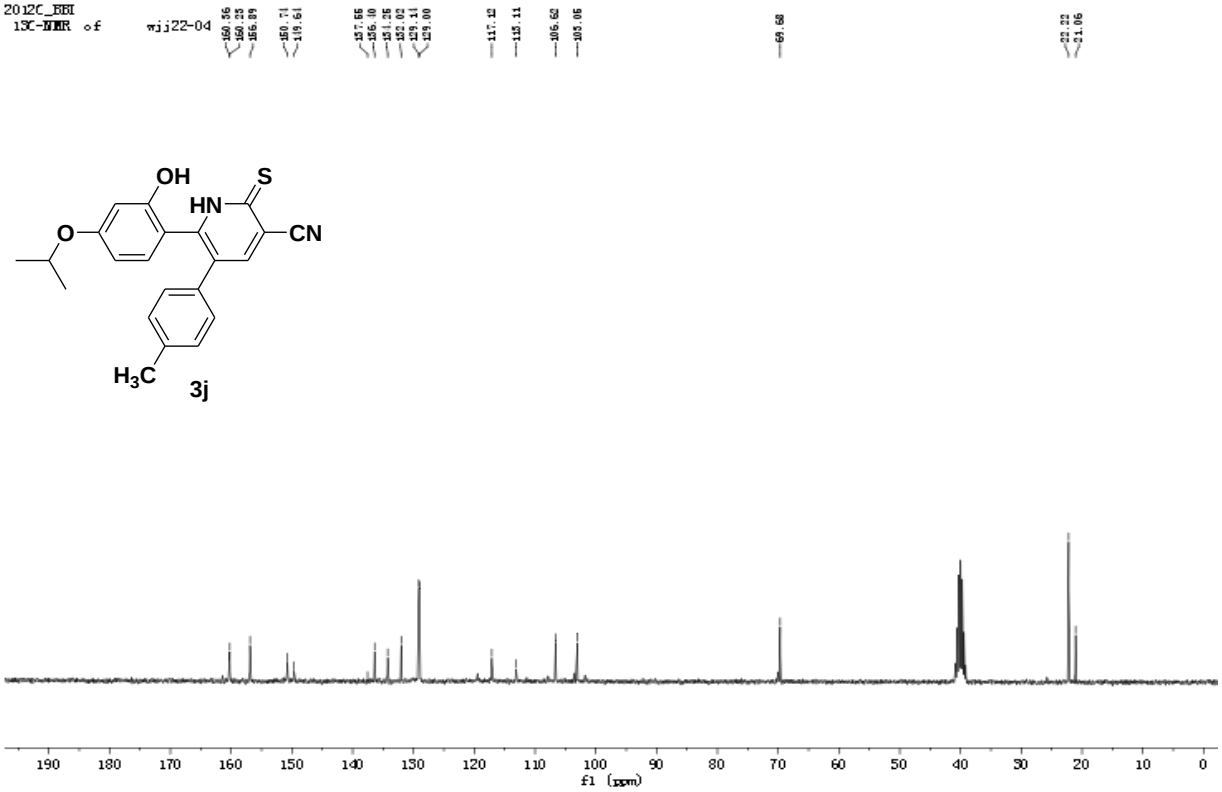
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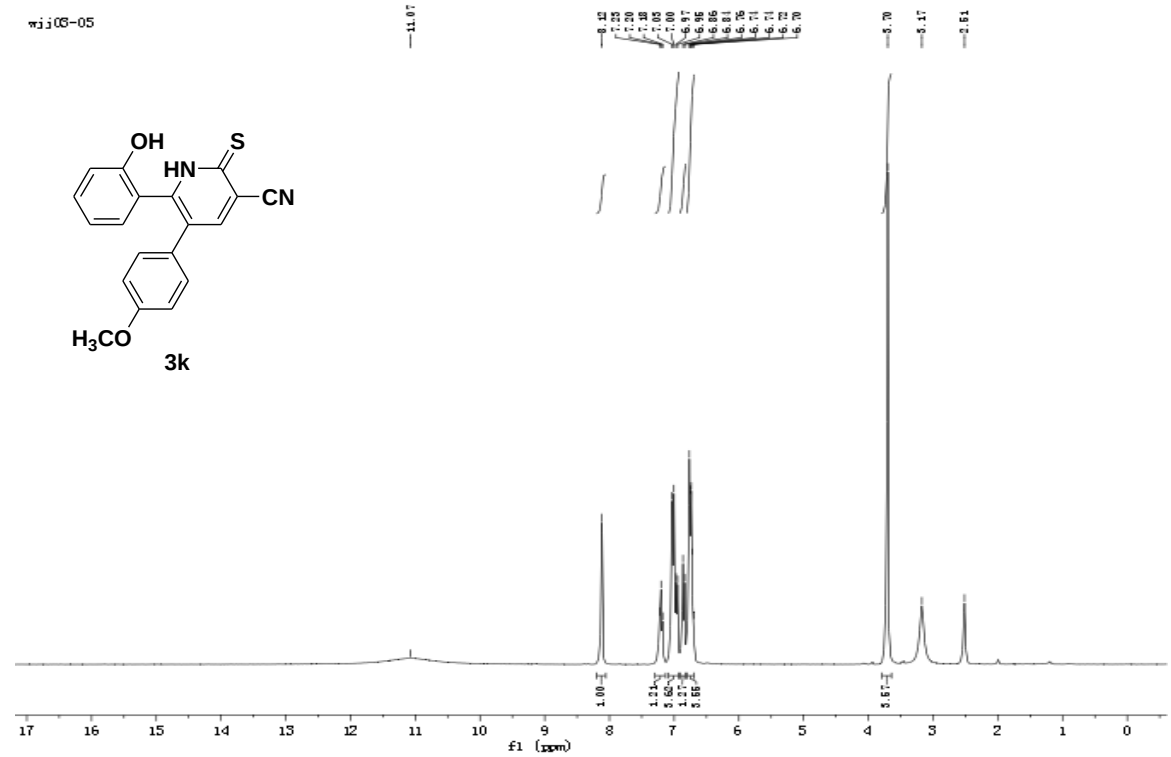
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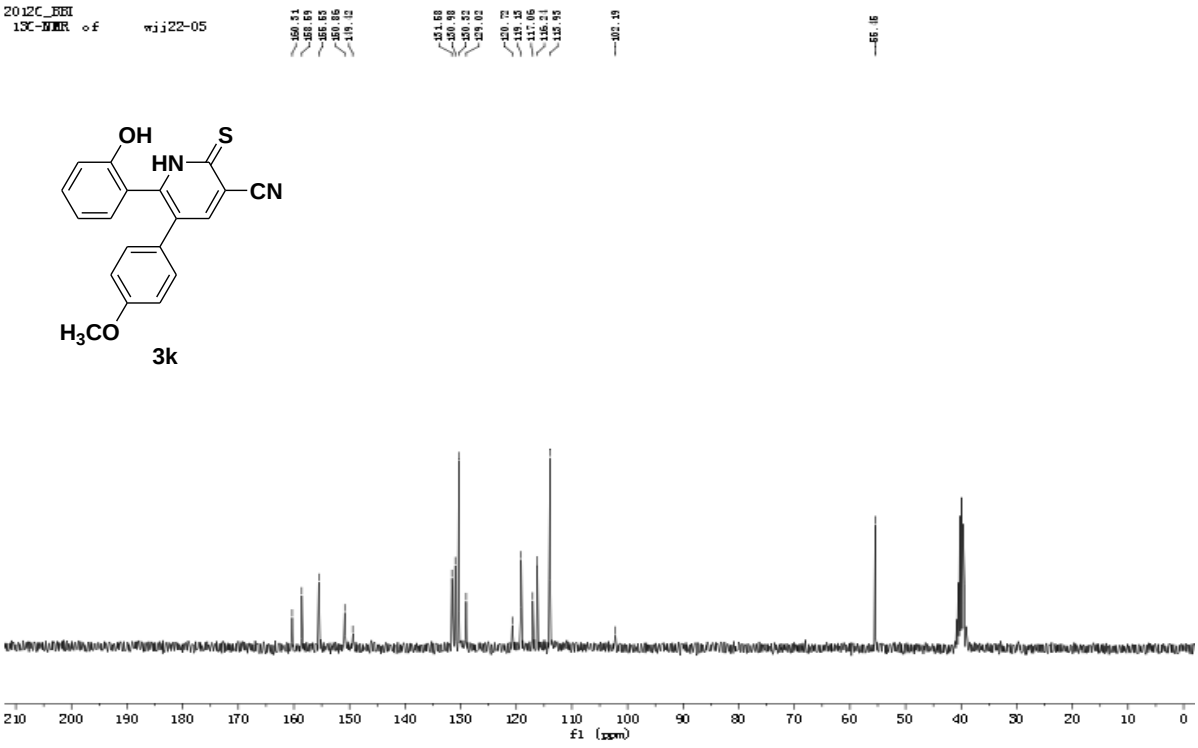
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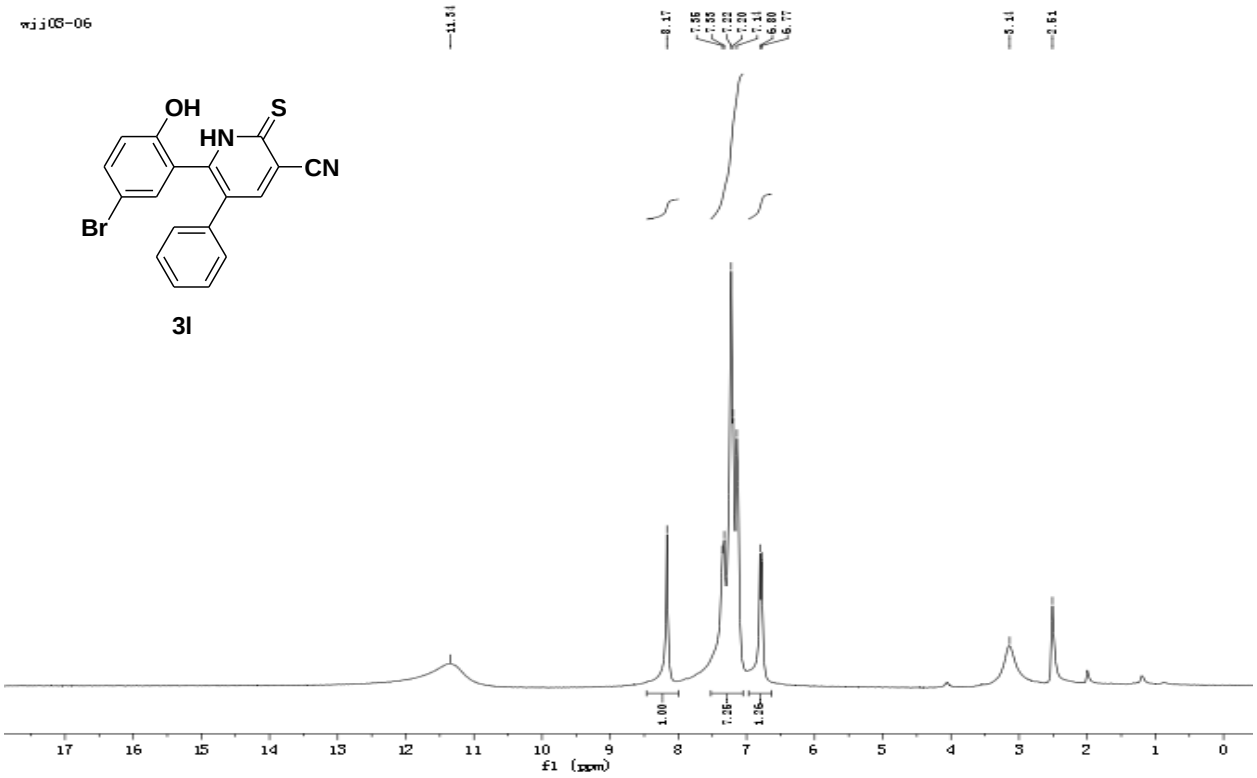
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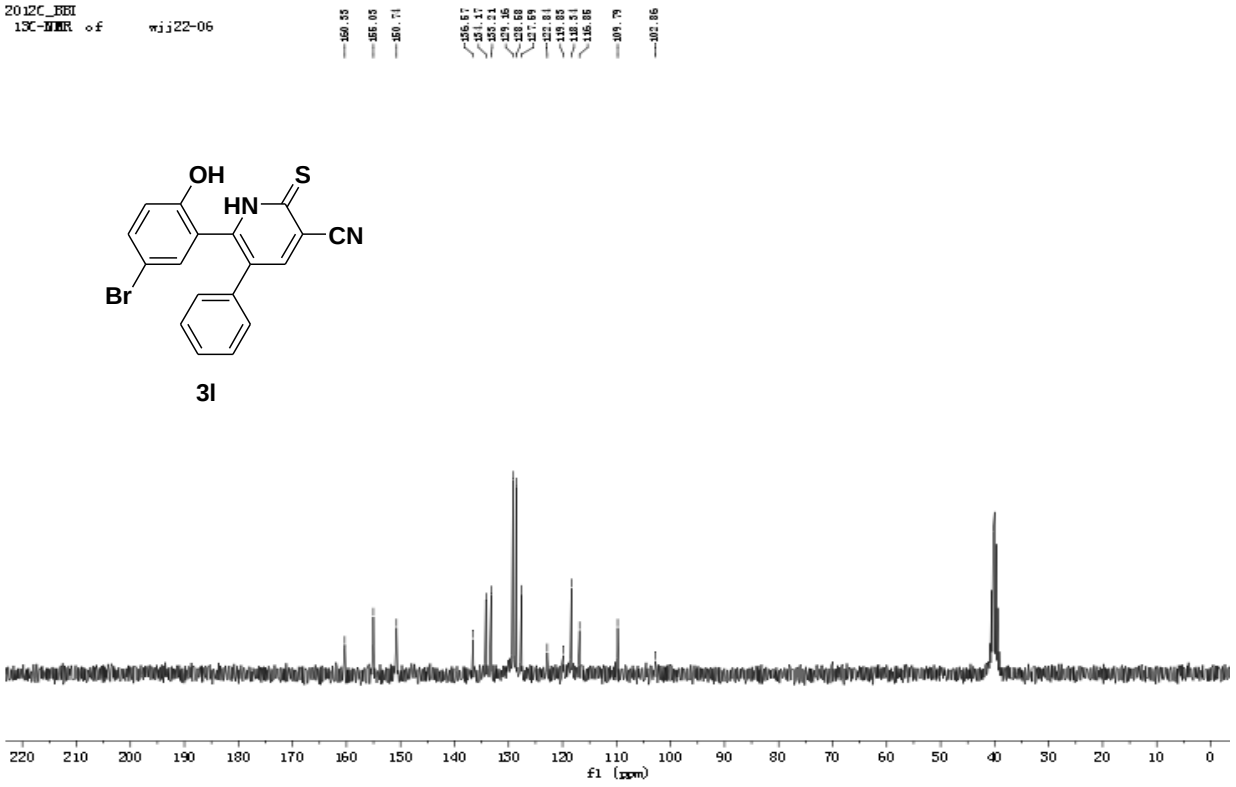
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S
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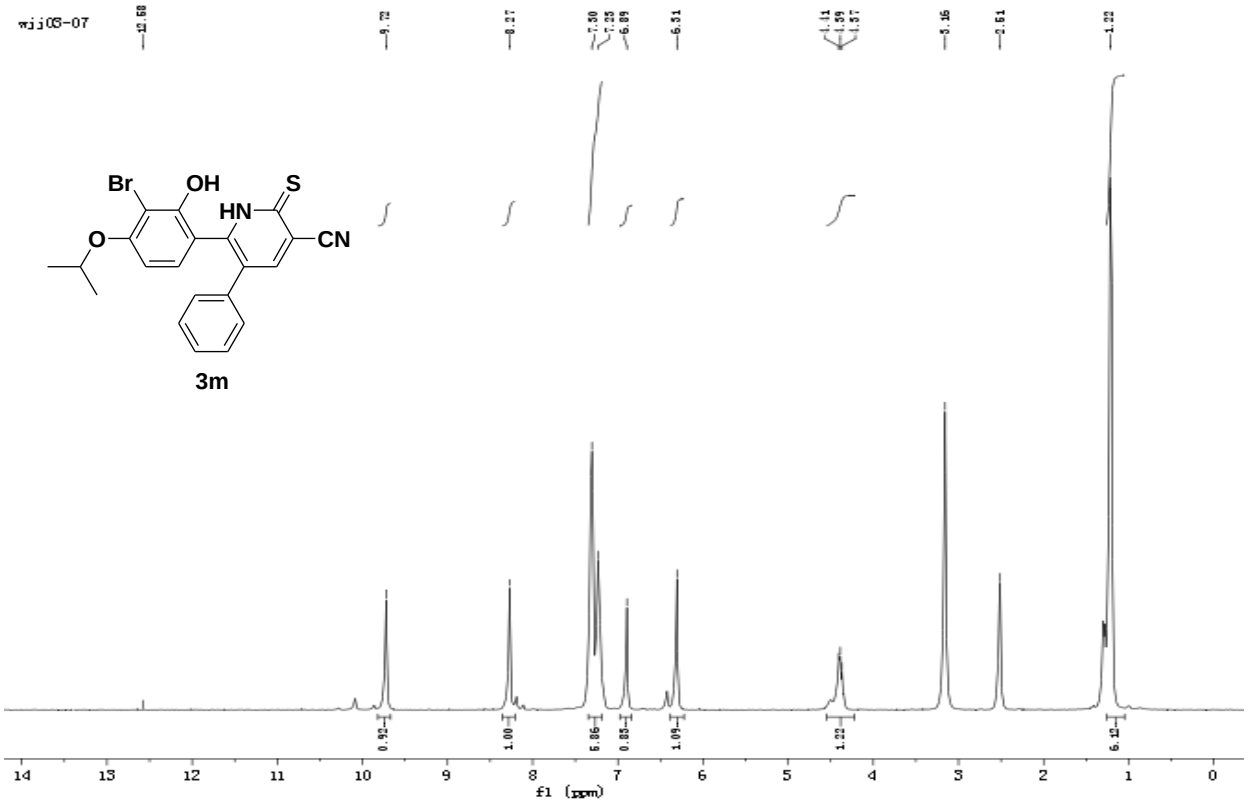
f1 (ppm)

3l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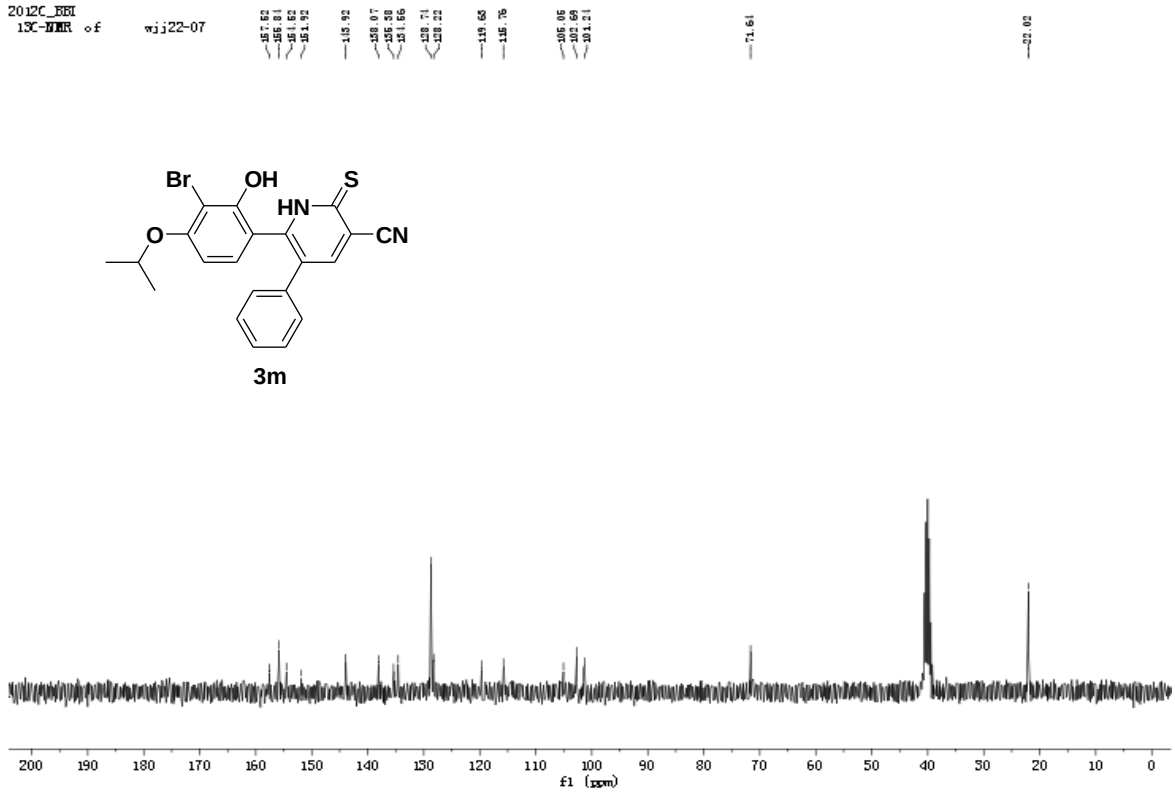
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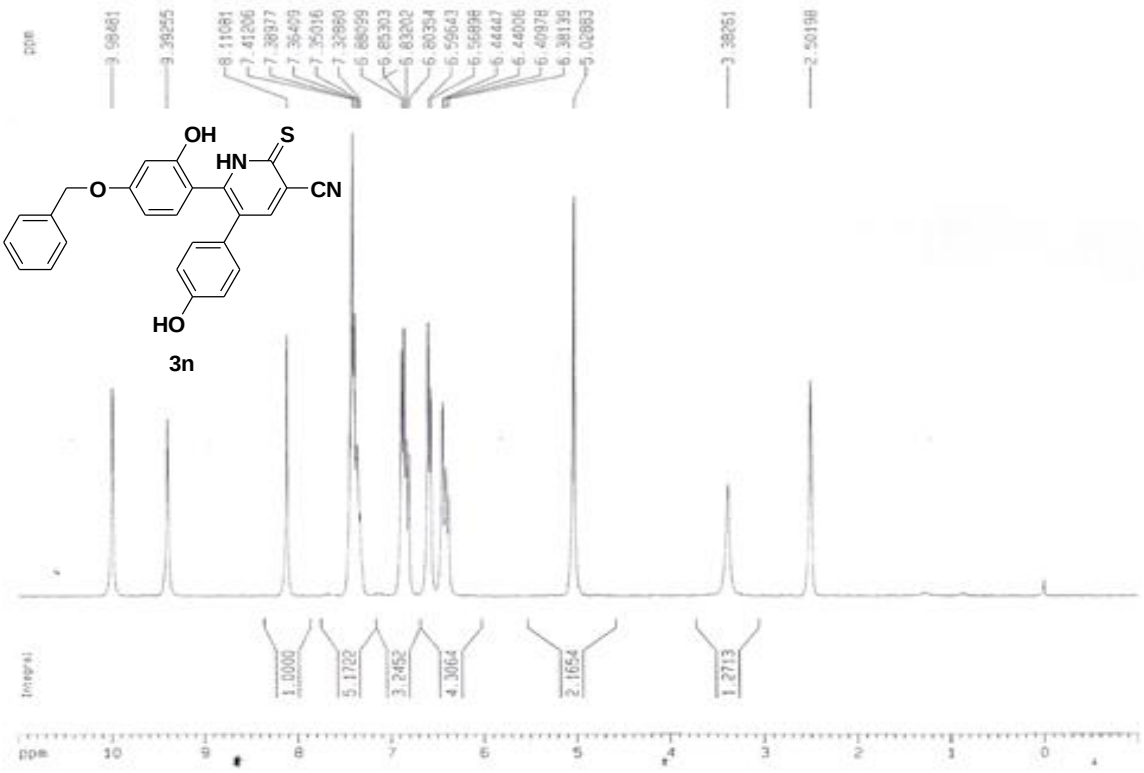

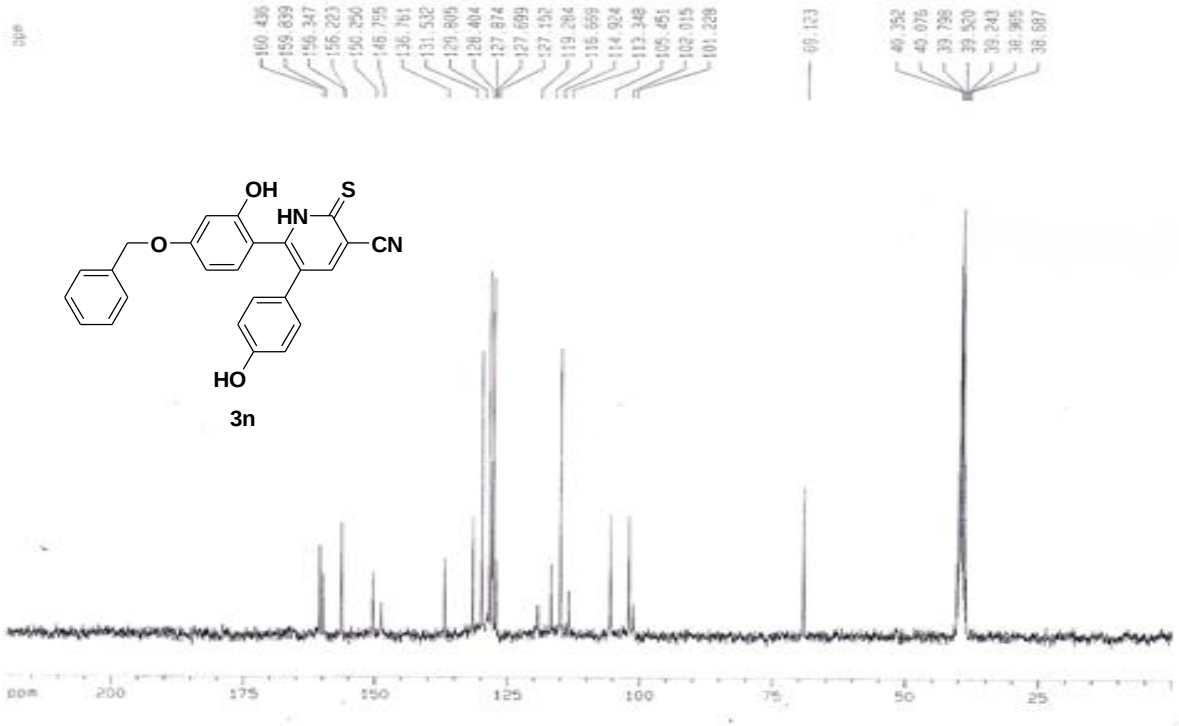

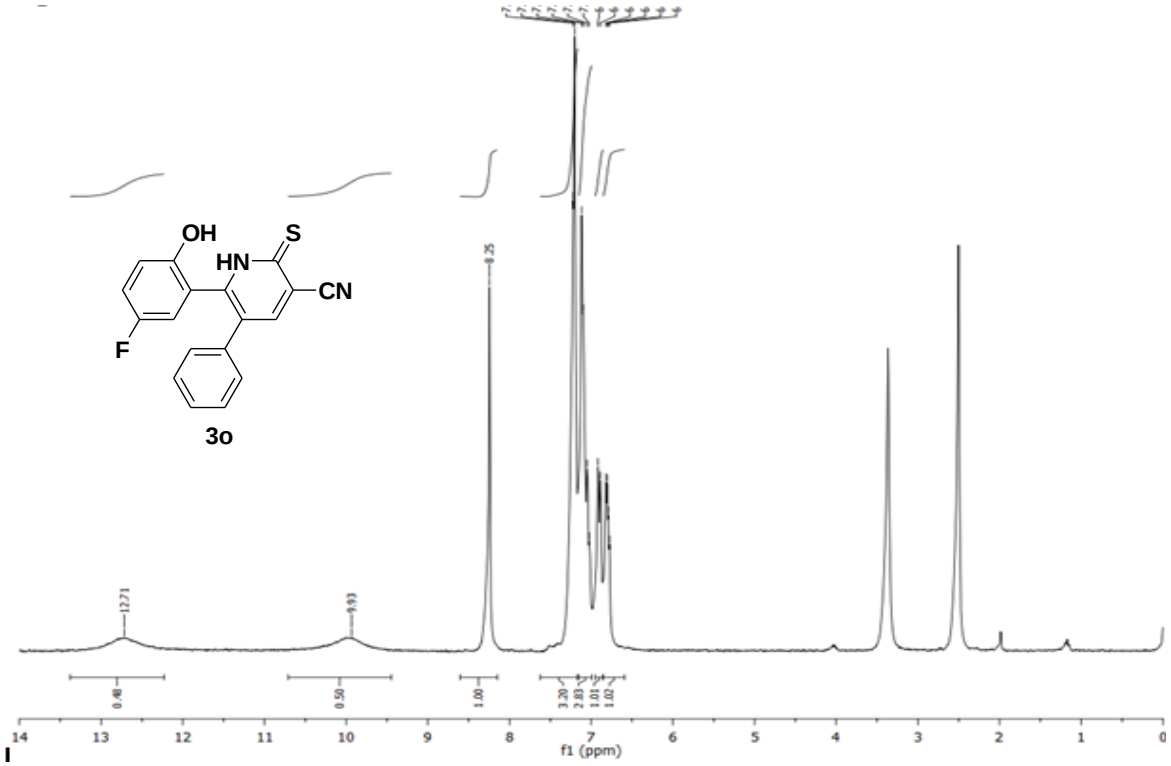
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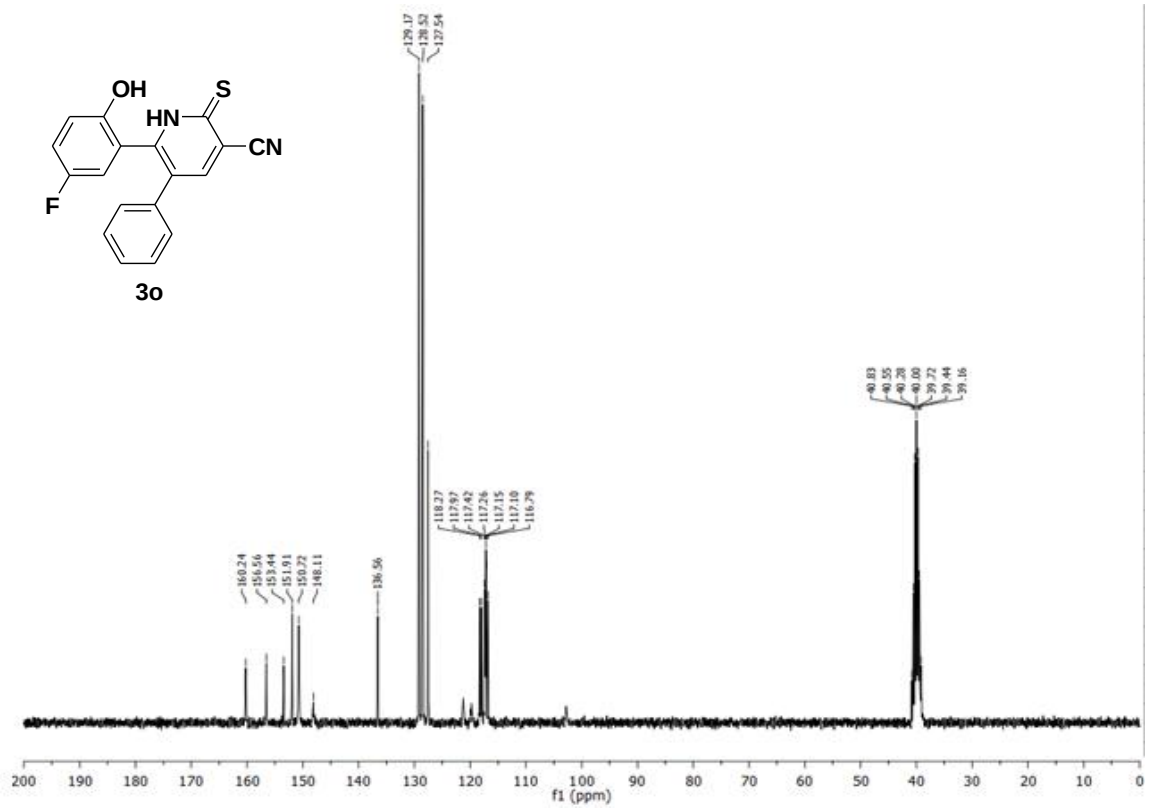
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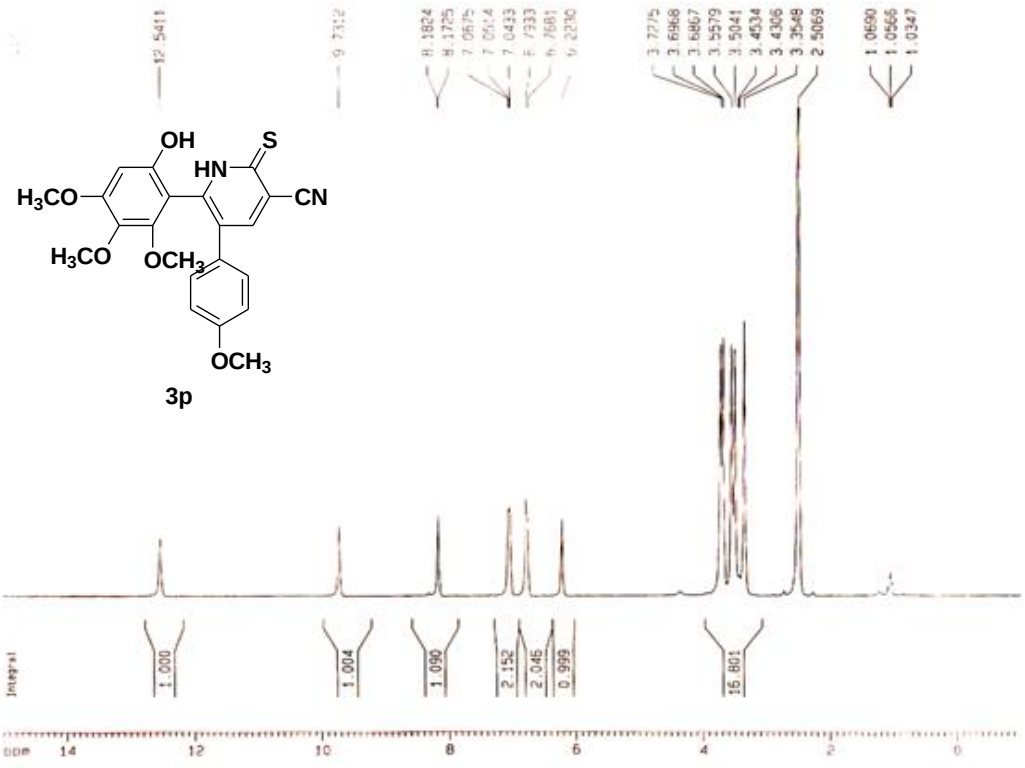

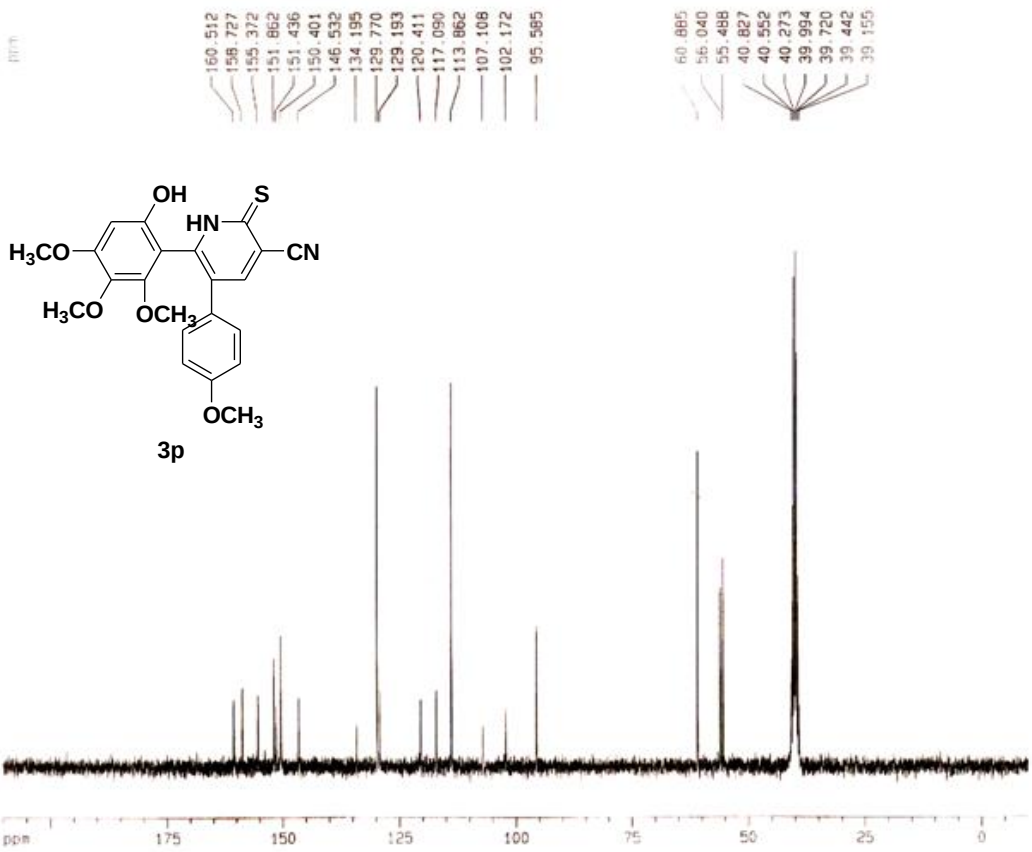

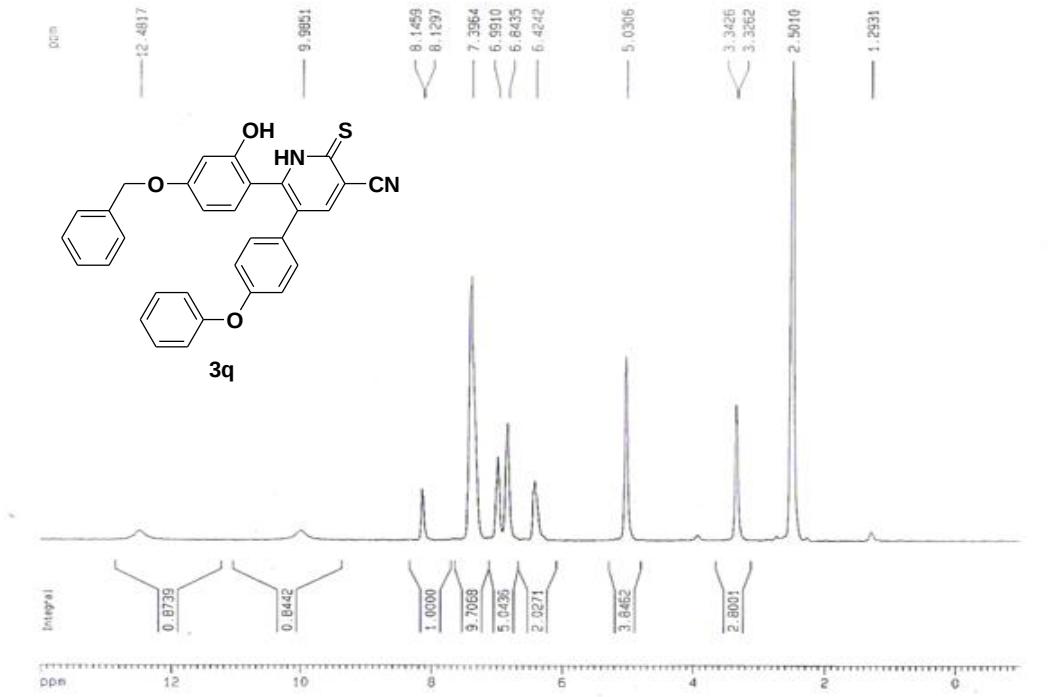

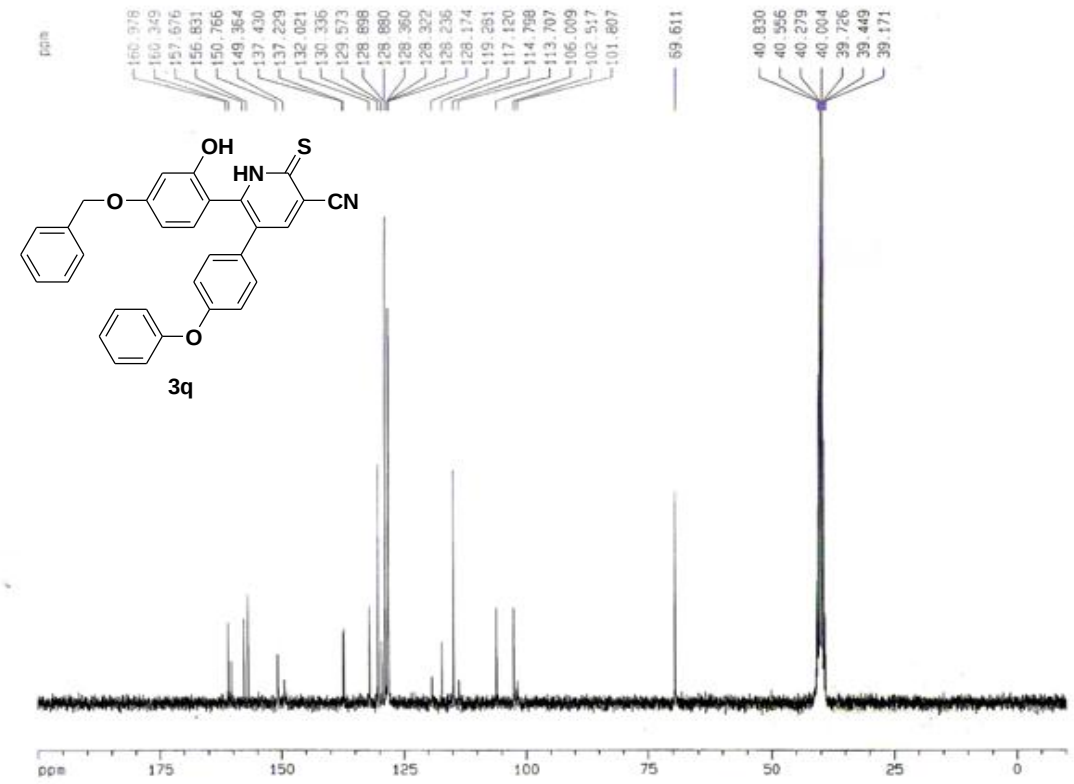

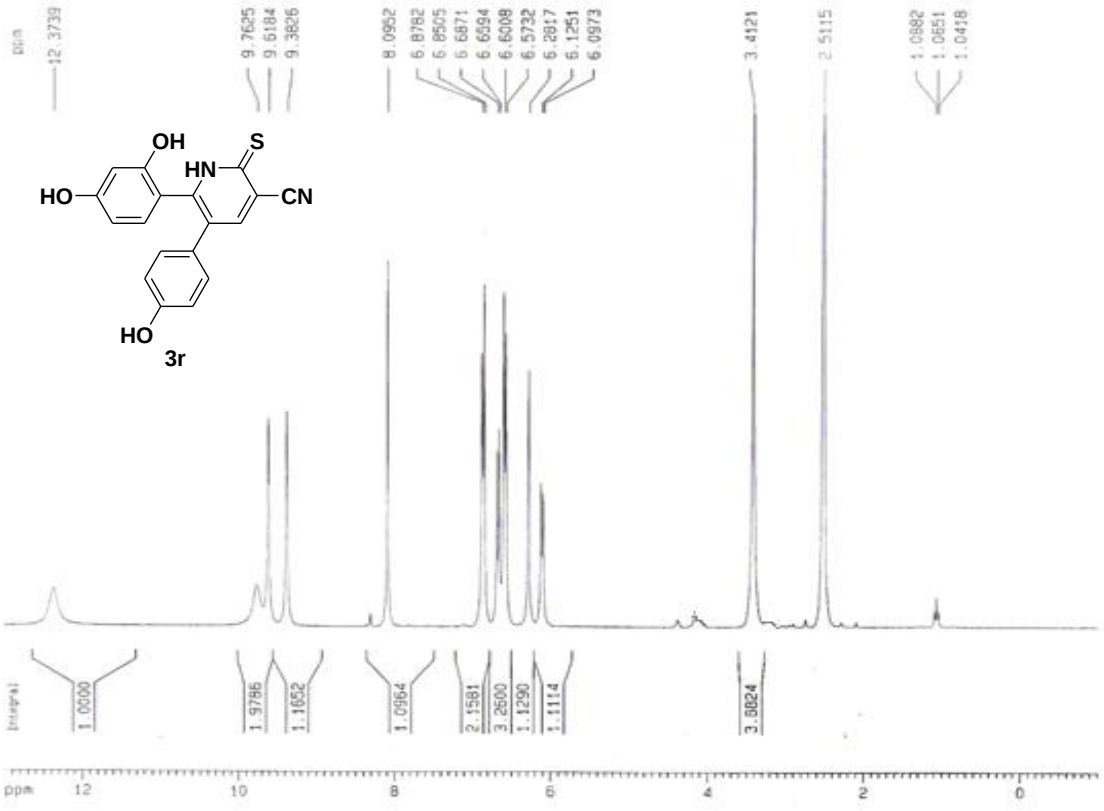
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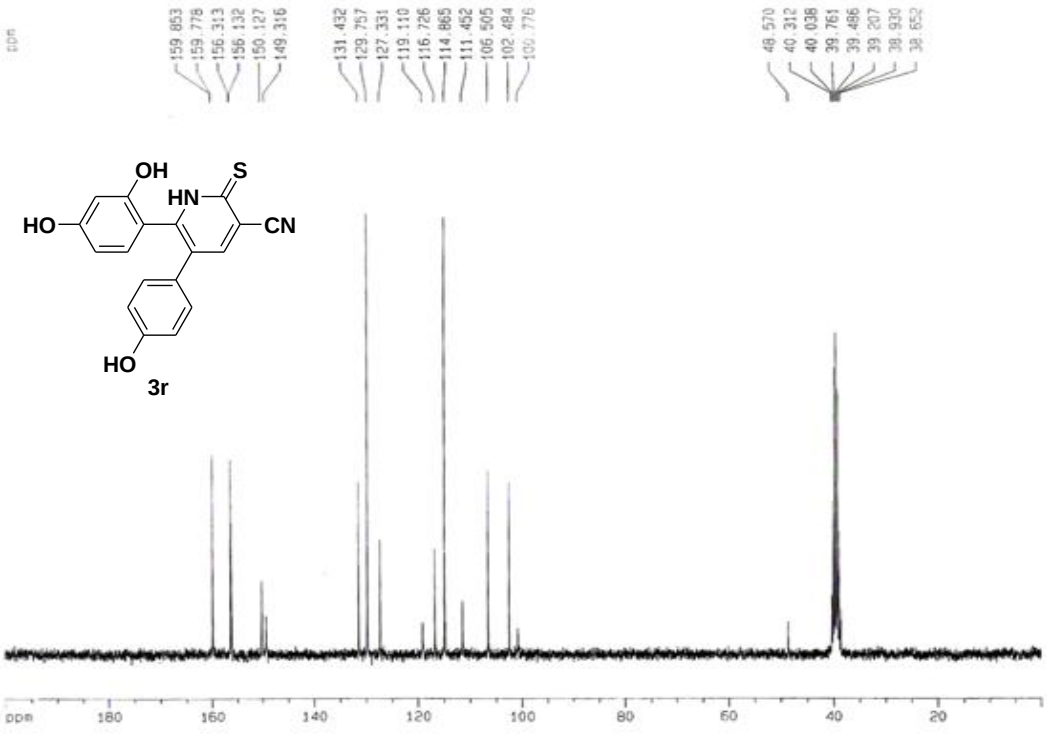
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102.484
48.570
40.312
40.038
39.761
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39.207
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HO
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3r
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140
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40
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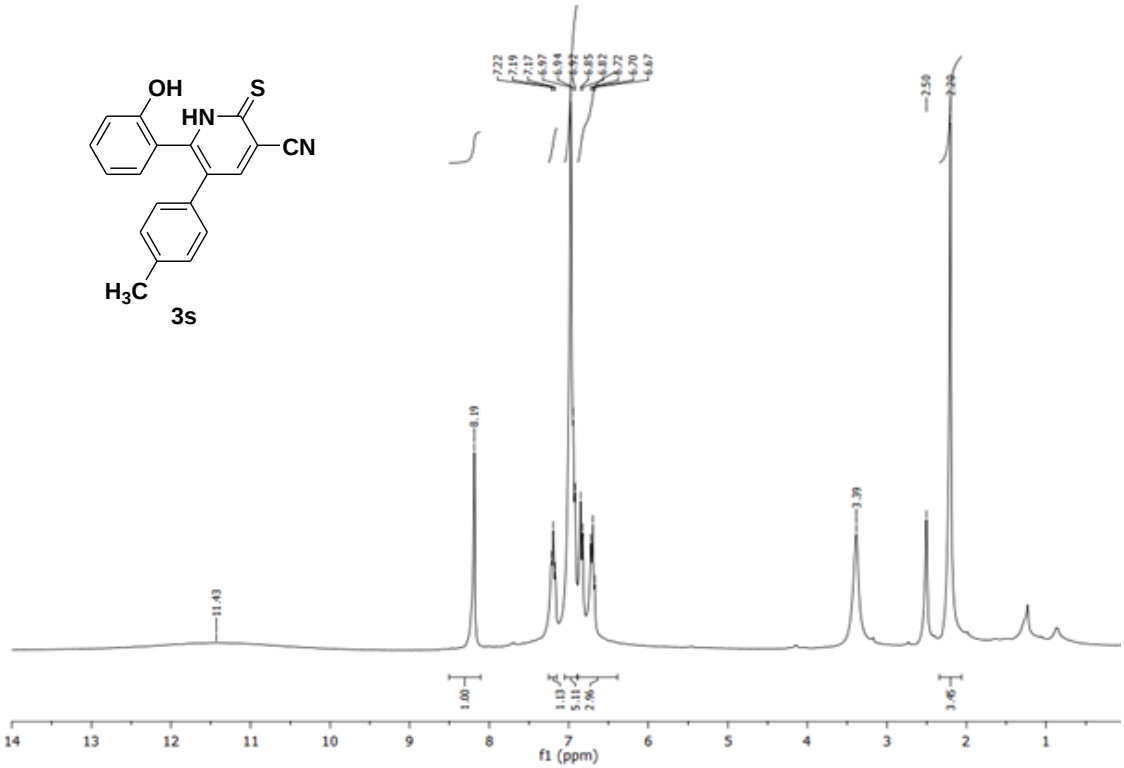

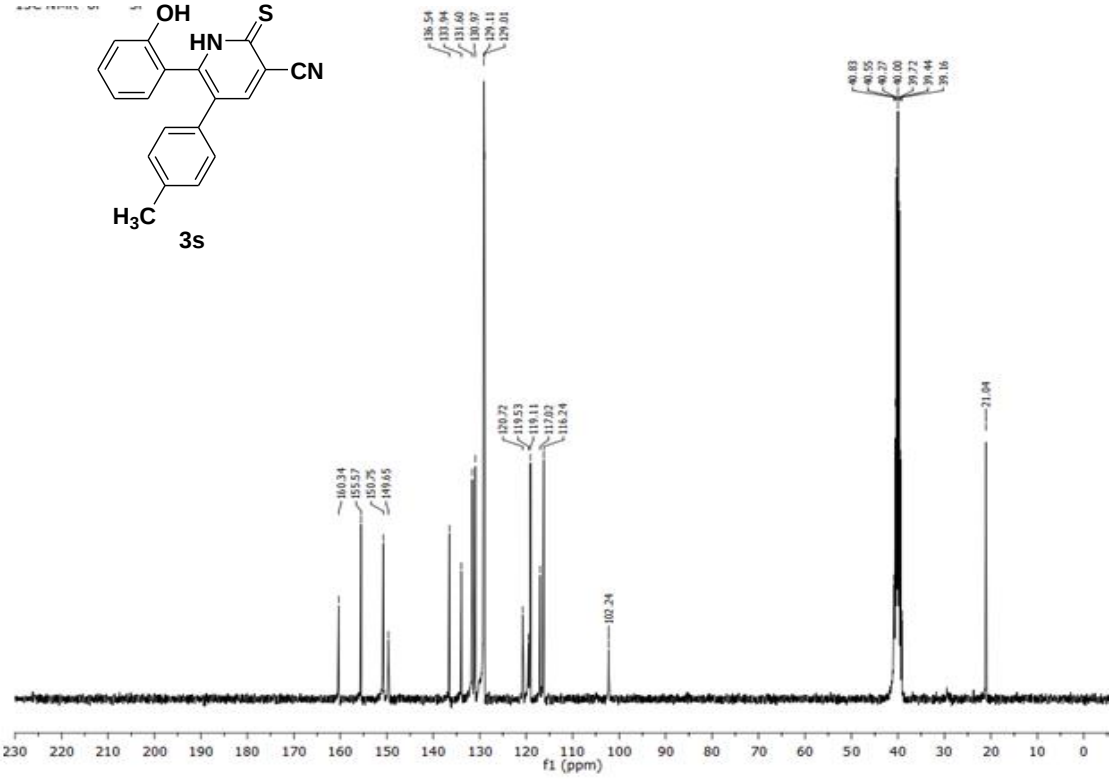

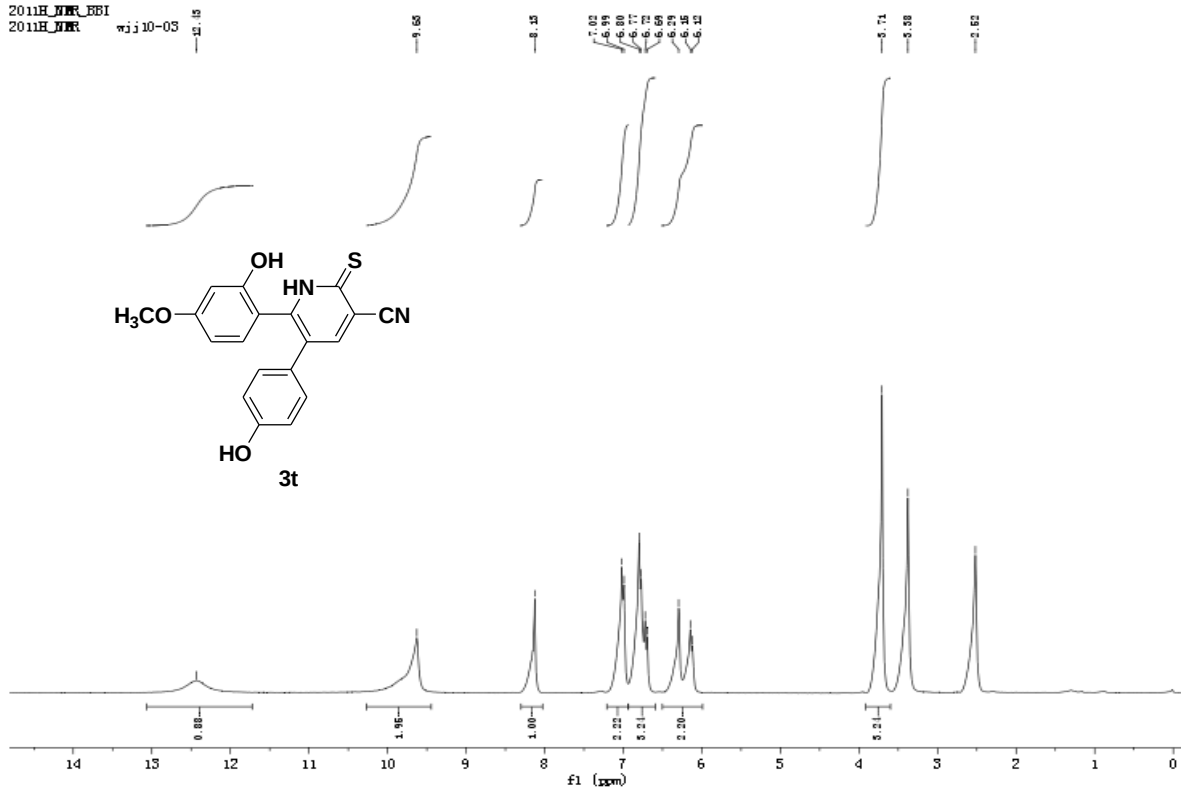
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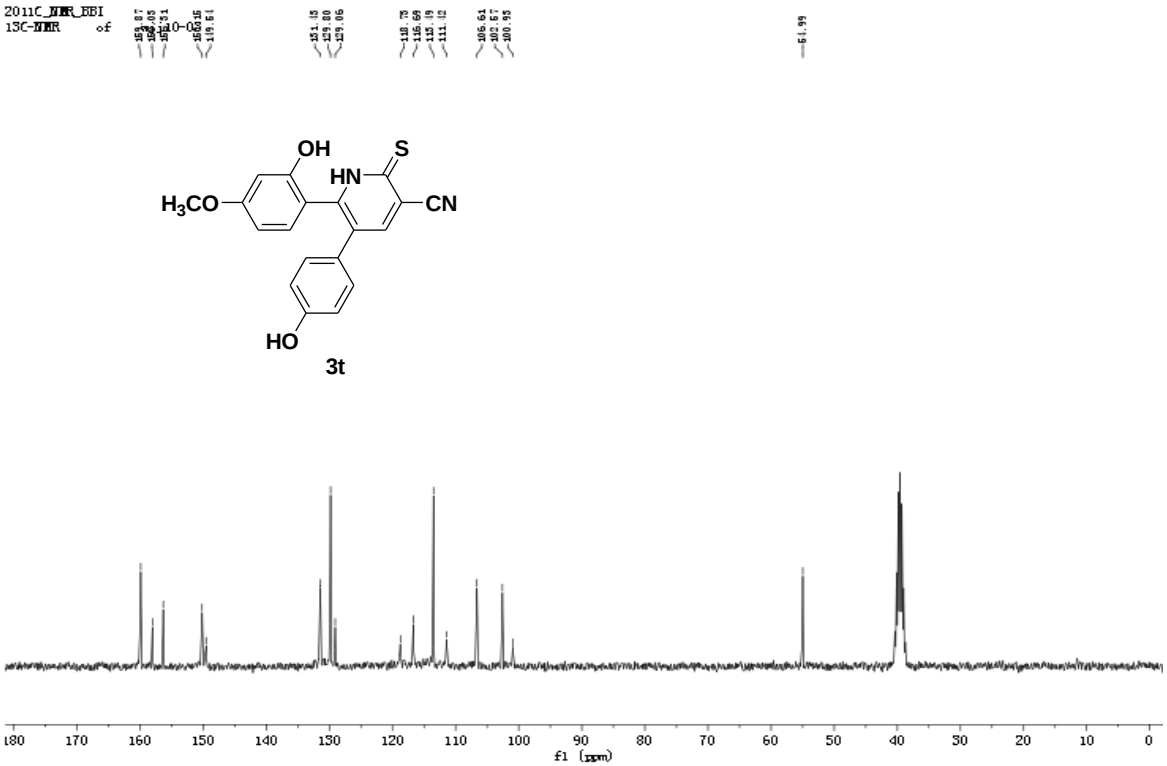
HN
H3CO
CN
HO
3t
f1 (ppm)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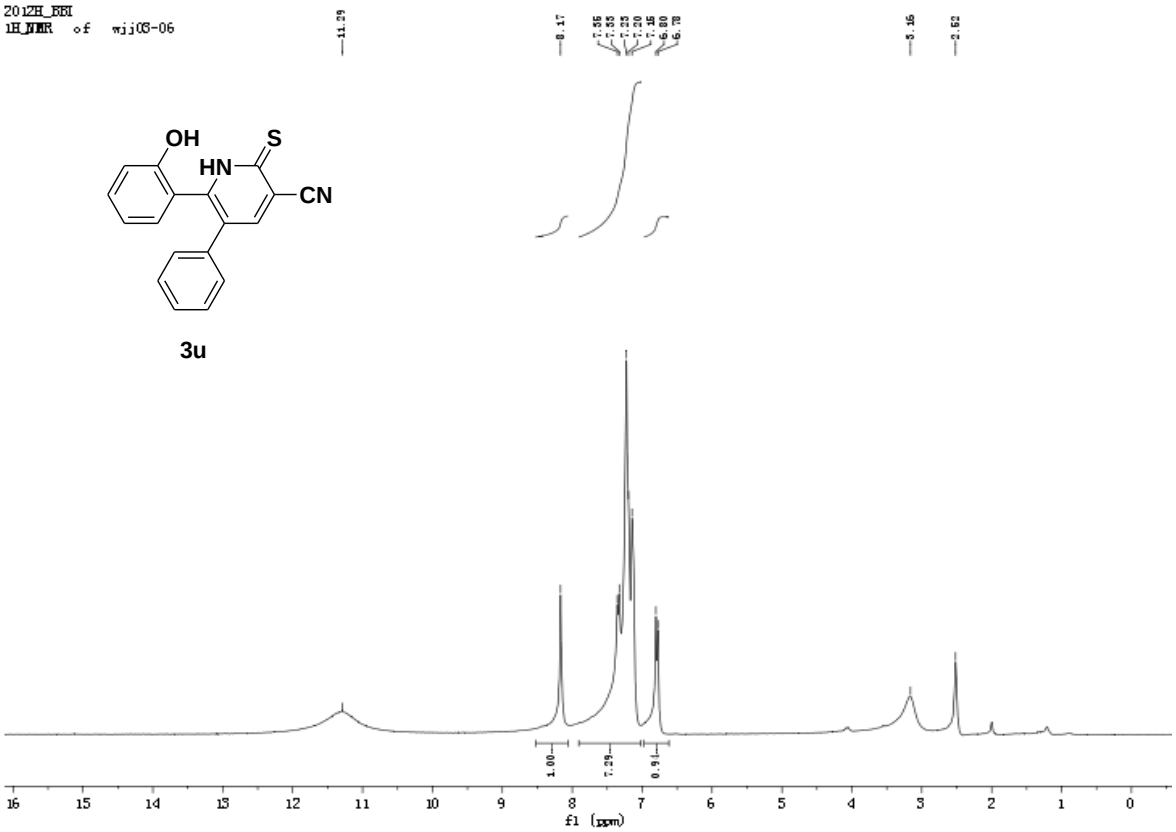
f1 (ppm)



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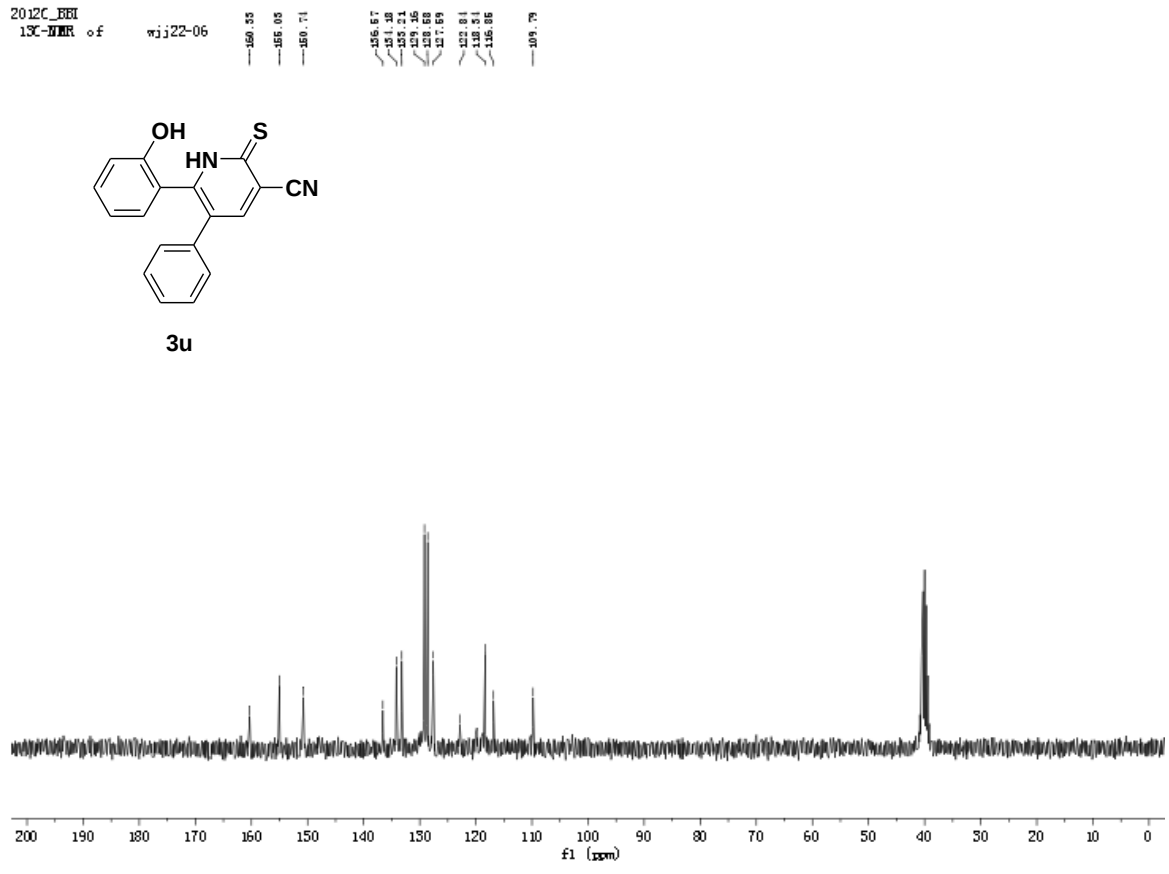

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