Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Practical Oxazole Synthesis Mediated by Iodine from α-Bromoketones and Benzylamine Derivatives

(Supplementary Information)

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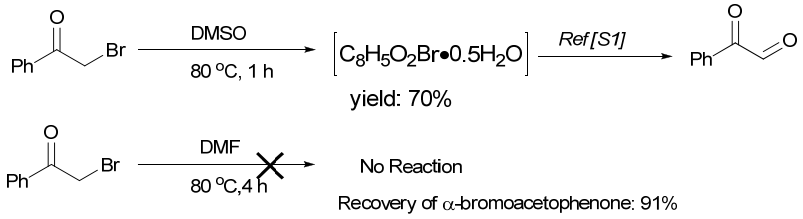
General Information

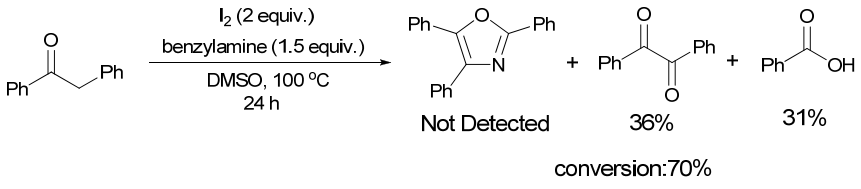
The ^{1}H NMR spectra were recorded at 400 MHz or 300 MHz and ^{13}C NMR spectra were measured at 100 MHz or 75 MHz using a Bruker AV400 instrument with $CDCl_3$ or DMSO-d_6 as the solvent. The chemical shifts (δ) were measured in ppm and with the solvents as references (For $CDCl_3$, ^{1}H: δ =7.26 ppm, ^{13}C: δ = 77.00 ppm; for DMSO-d_6, ^{1}H: δ =2.50 ppm, ^{13}C: δ = 39.43 ppm). The multiplicities of the signals are described using the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, br = broad. The known compounds were identified by the comparison of their NMR spectra with reported data in the literatures. The new compounds were characterized by NMR, IR, HRMS and melting point for solid samples. IR spectra were recorded on a FT-IR Bruker EQUINOX55 spectrometer and only major peaks are reported in cm^{-1}. High resolution mass spectral analyses (HR-MS) were performed on a high resolution ESI-FTICR mass spectrometer (Varian 7.0 T). Melting points were recorded on a RY-1 type apparatus. Optical rotations were obtained on a Perkin-Elmer 431 Polarimeter. All solvents were obtained from commercial sources and were purified according to standard procedures. Petroleum ether (PE), where used, had the boiling point range 60-90 ^{o}C.

Control Experiments

The oxidation test of DMSO and DMF



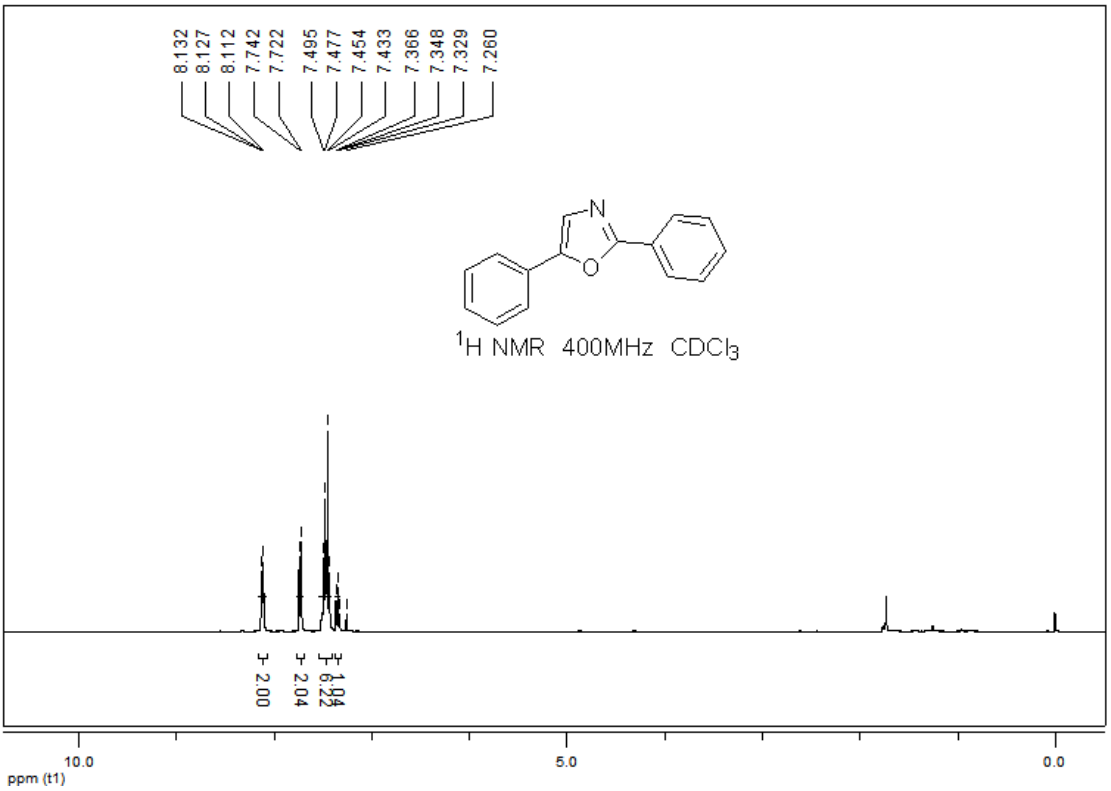
The examination of 2,4,5-triphenyl oxazole synthesis from α-iodo ketone generated in situ

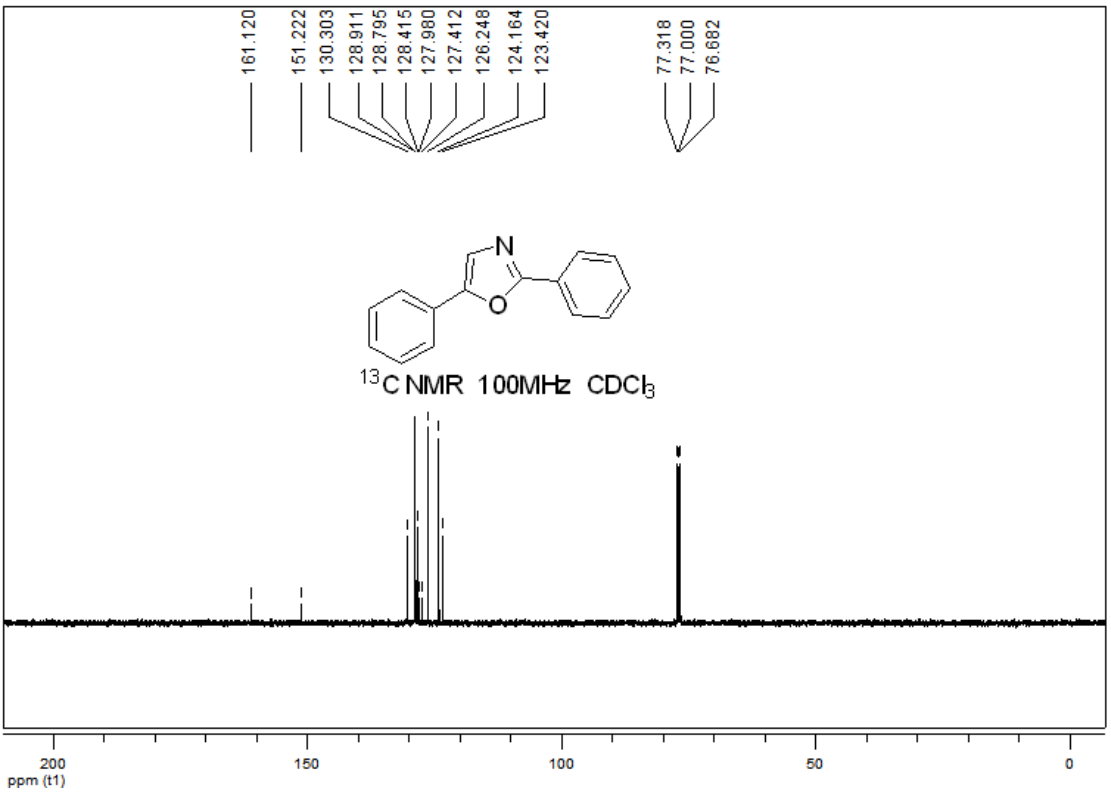


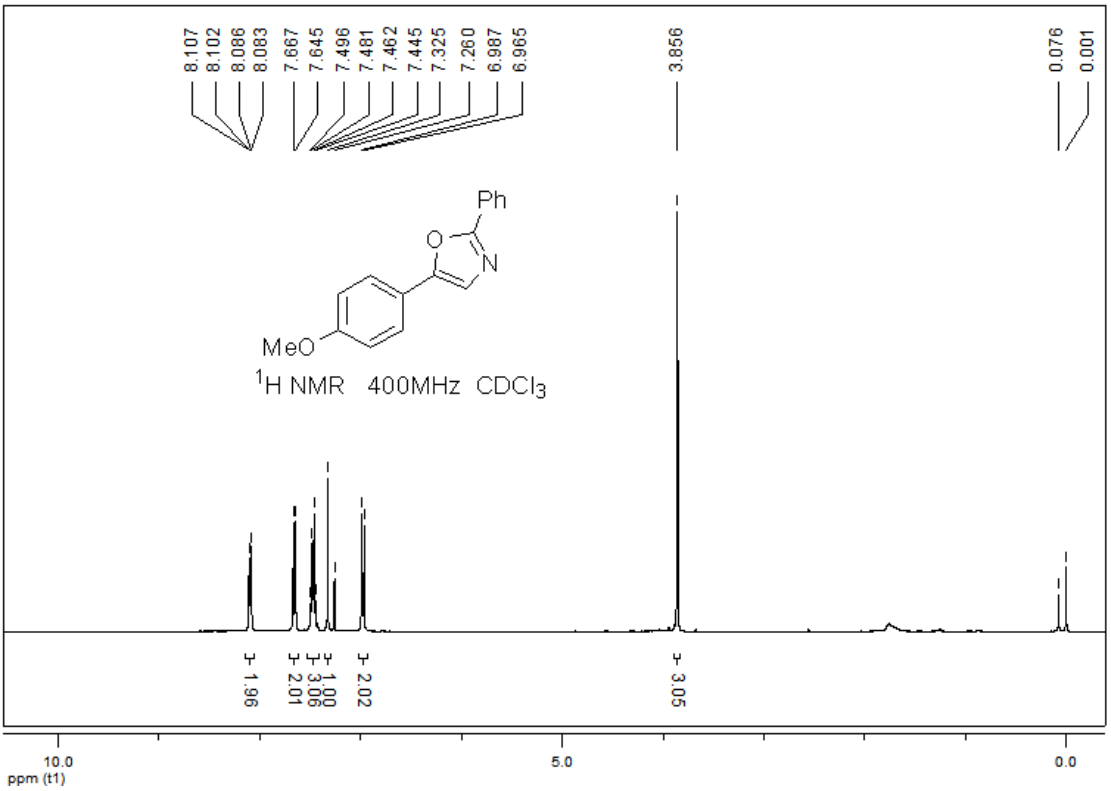
References

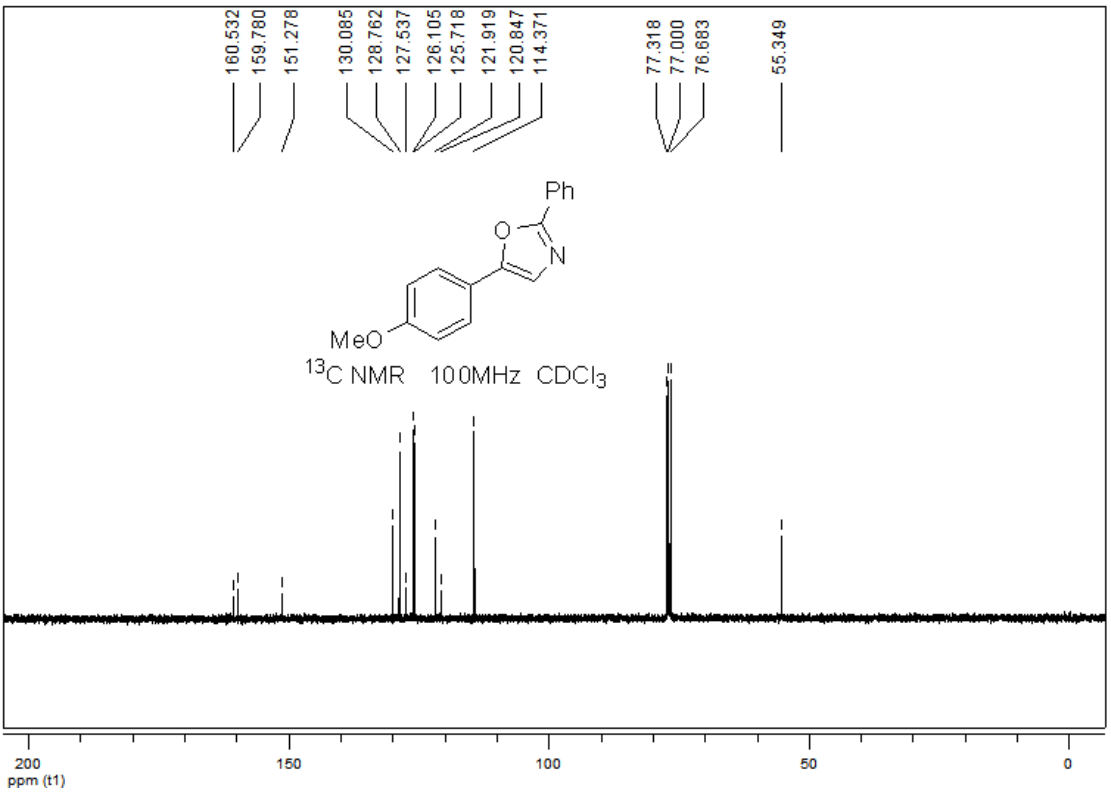
S1 N. Kornblum, J. W. Powers, G. J. Anderson, W. J. Jones, H. O. Larson, O. Levand and W. M. Weaver, *J. Am. Chem. Soc.*, 1957, **79**, 6562.

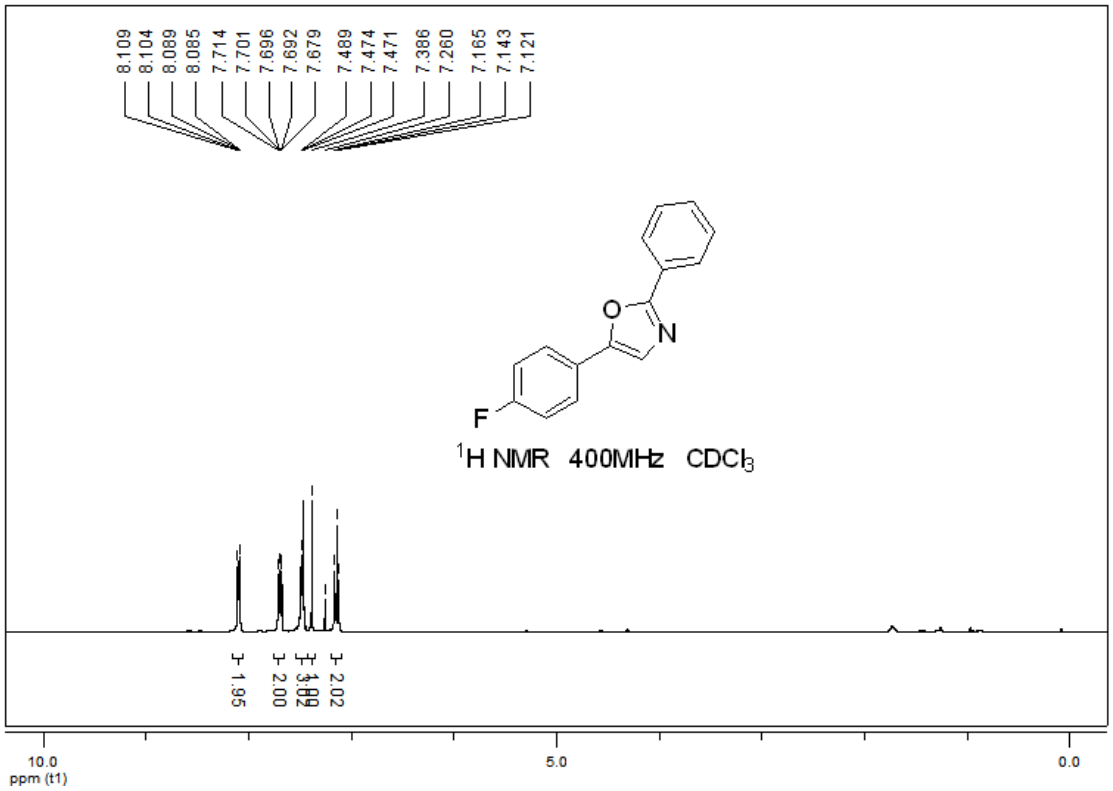
NMR Spectra of Products

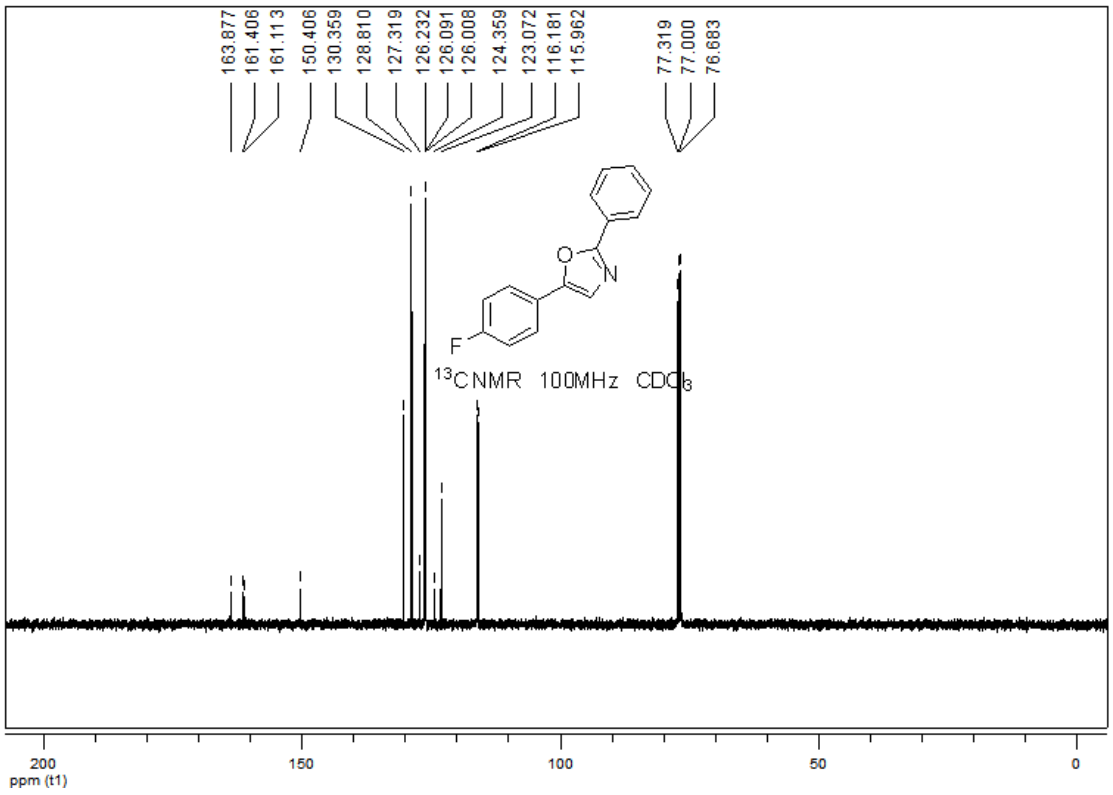


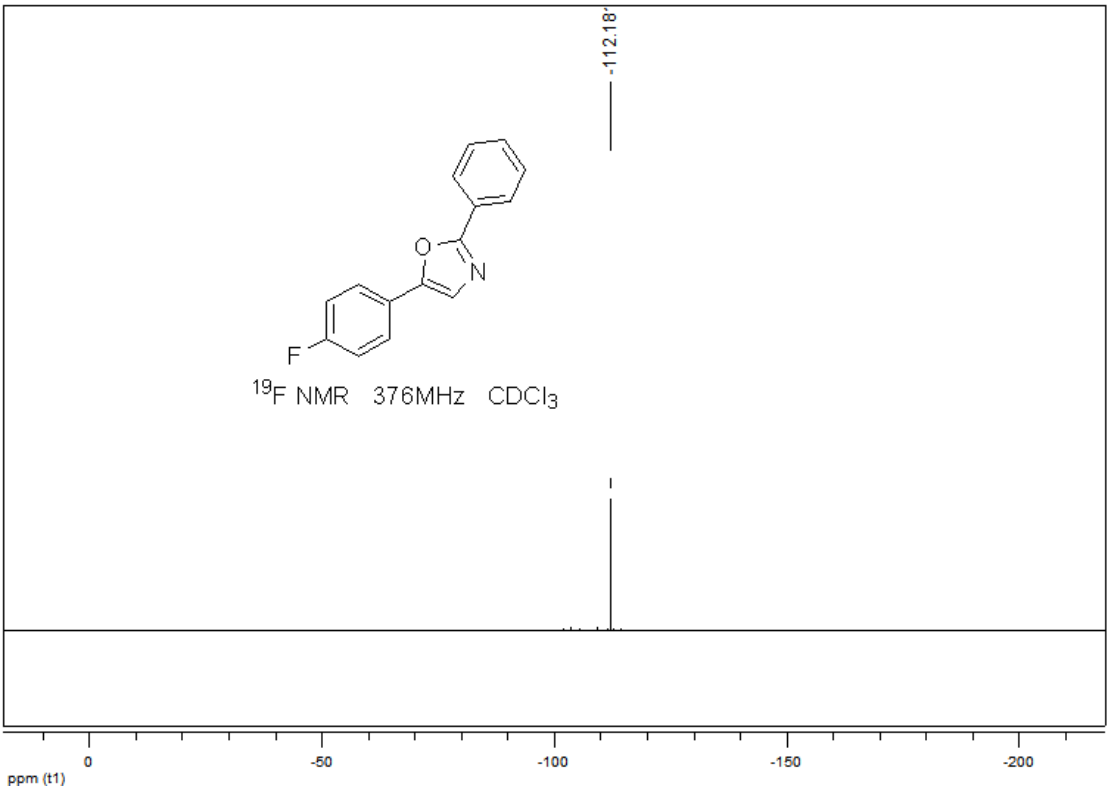


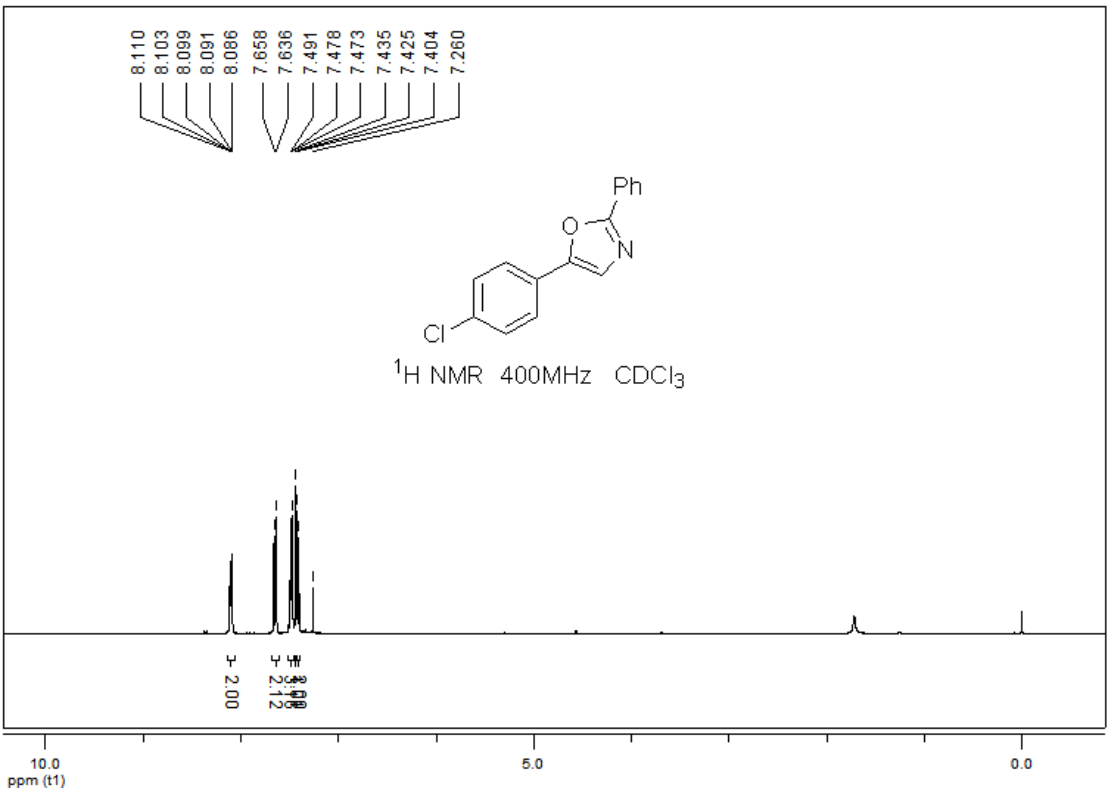


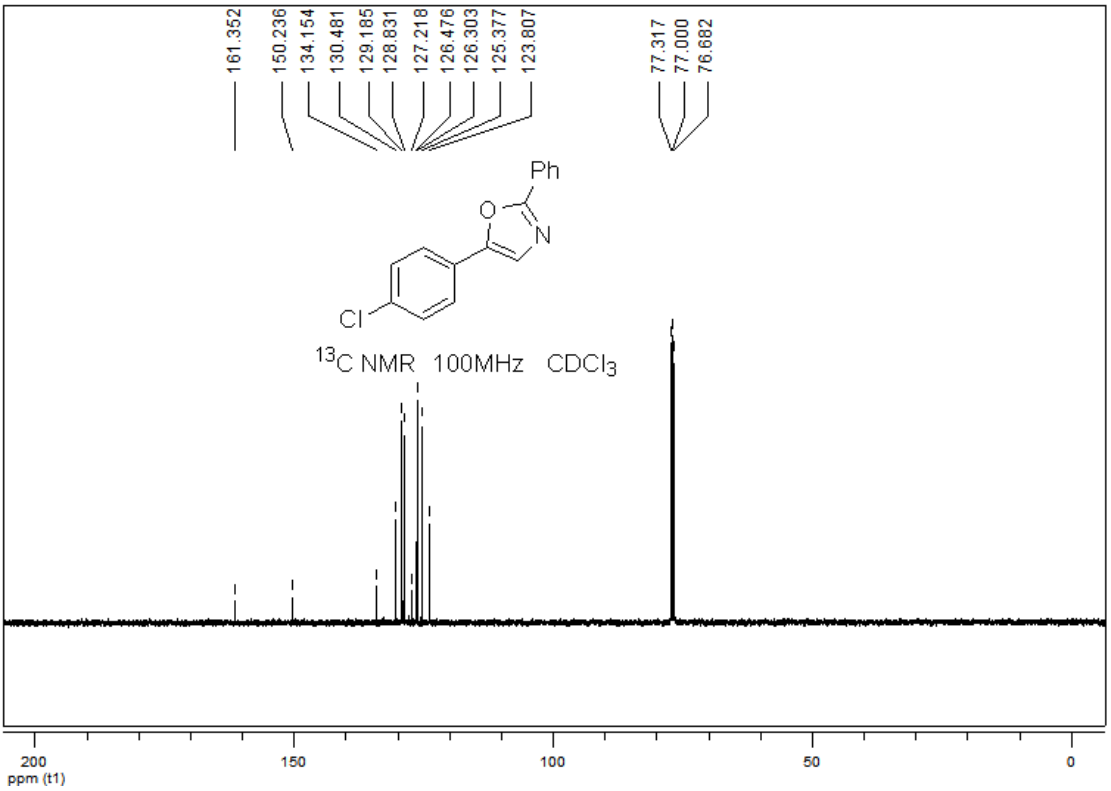


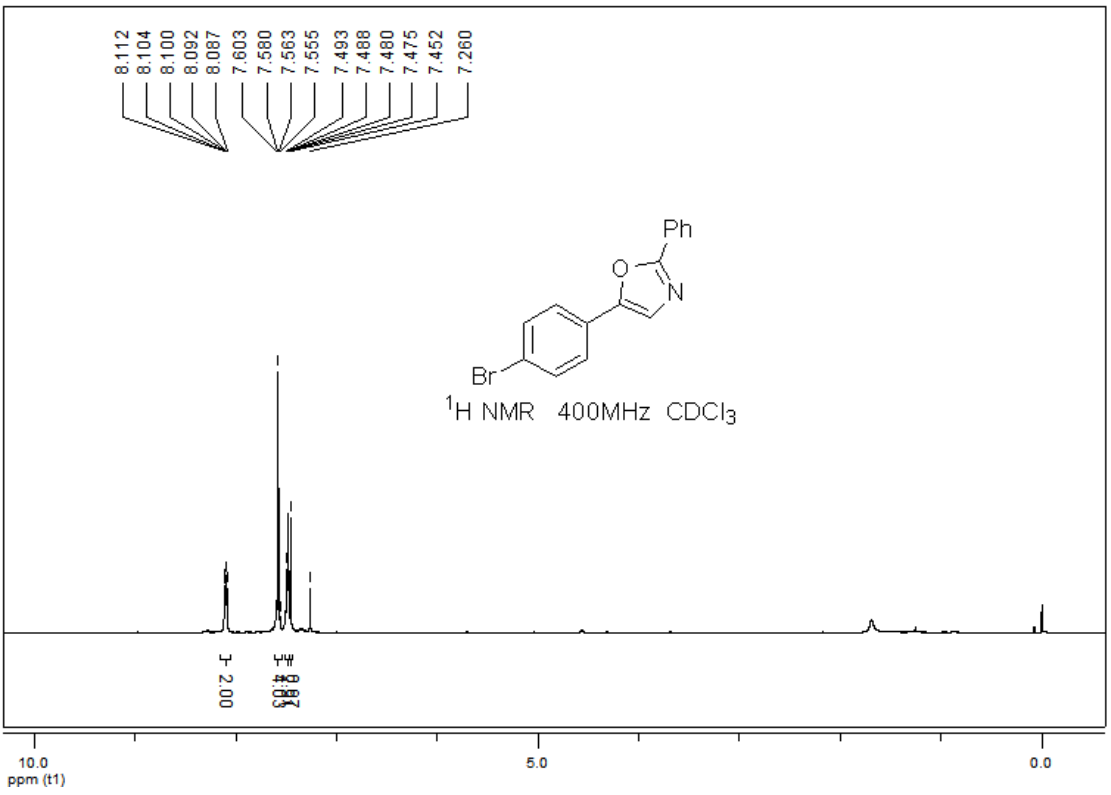


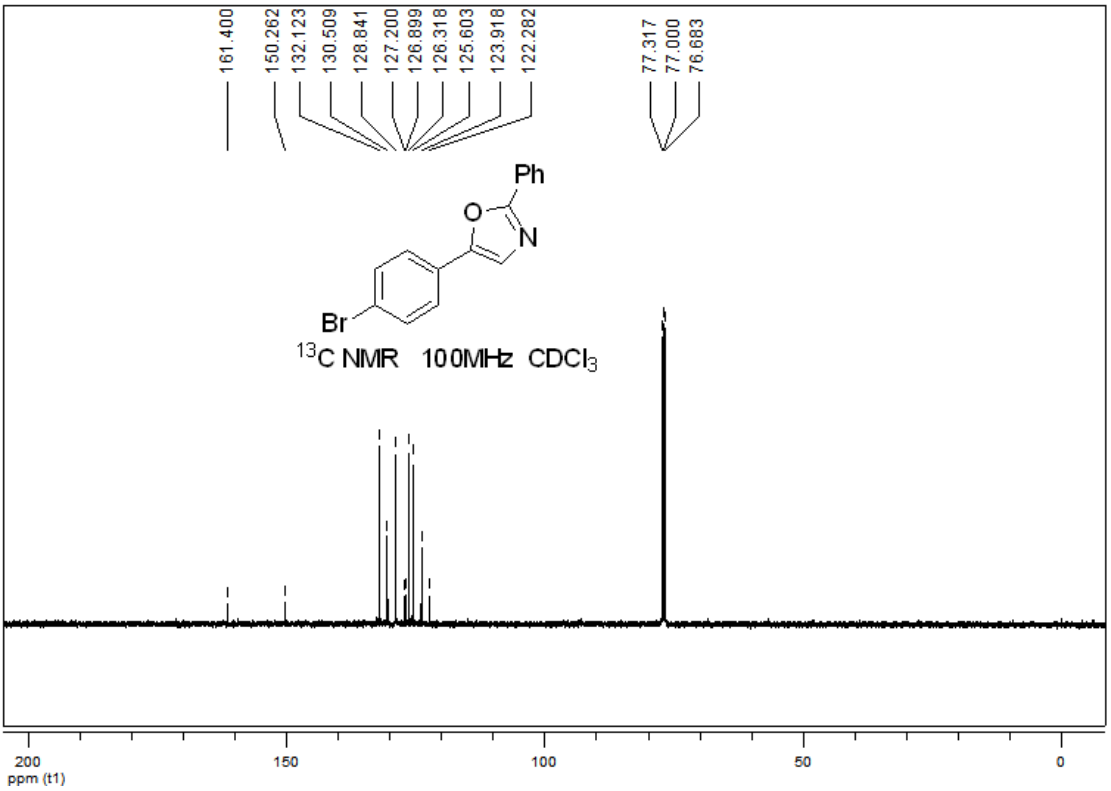








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Br
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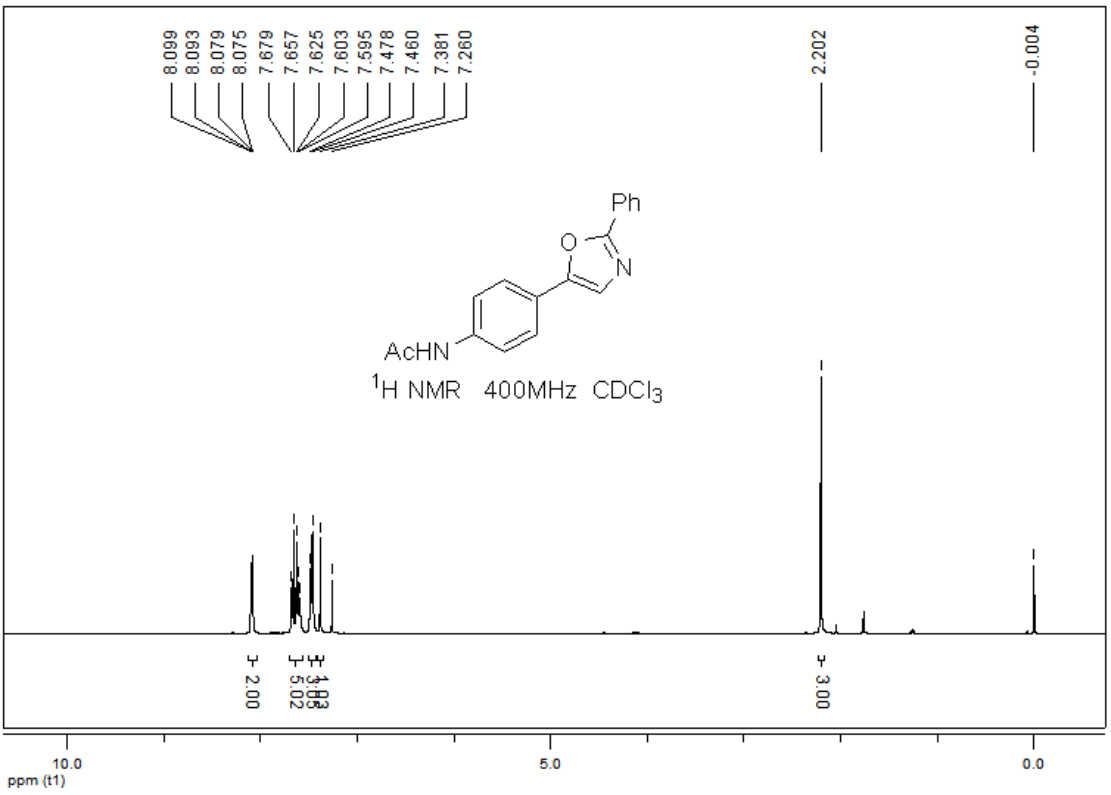
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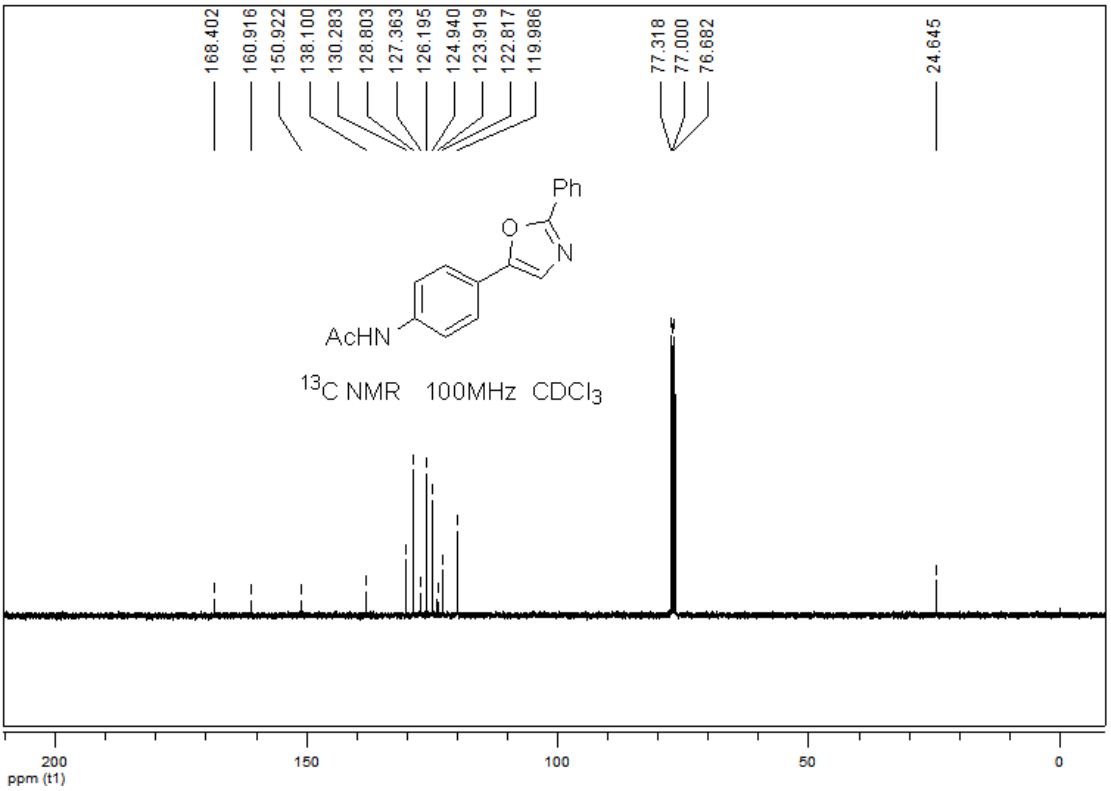

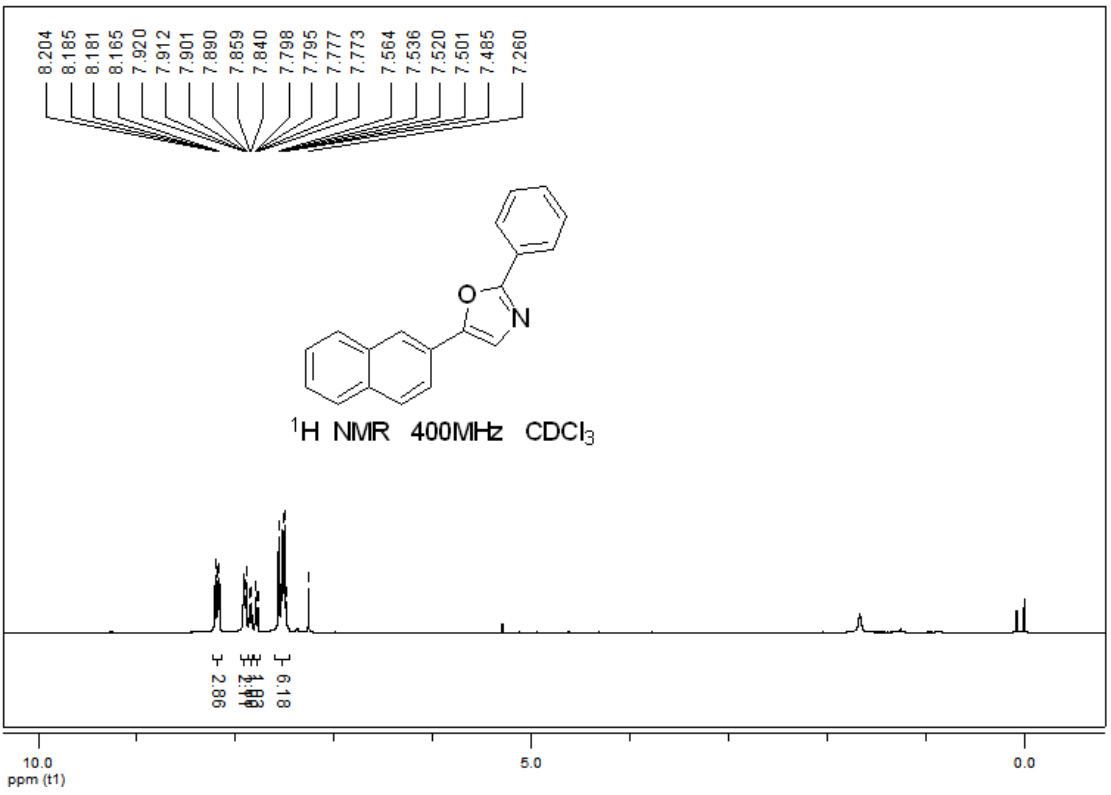

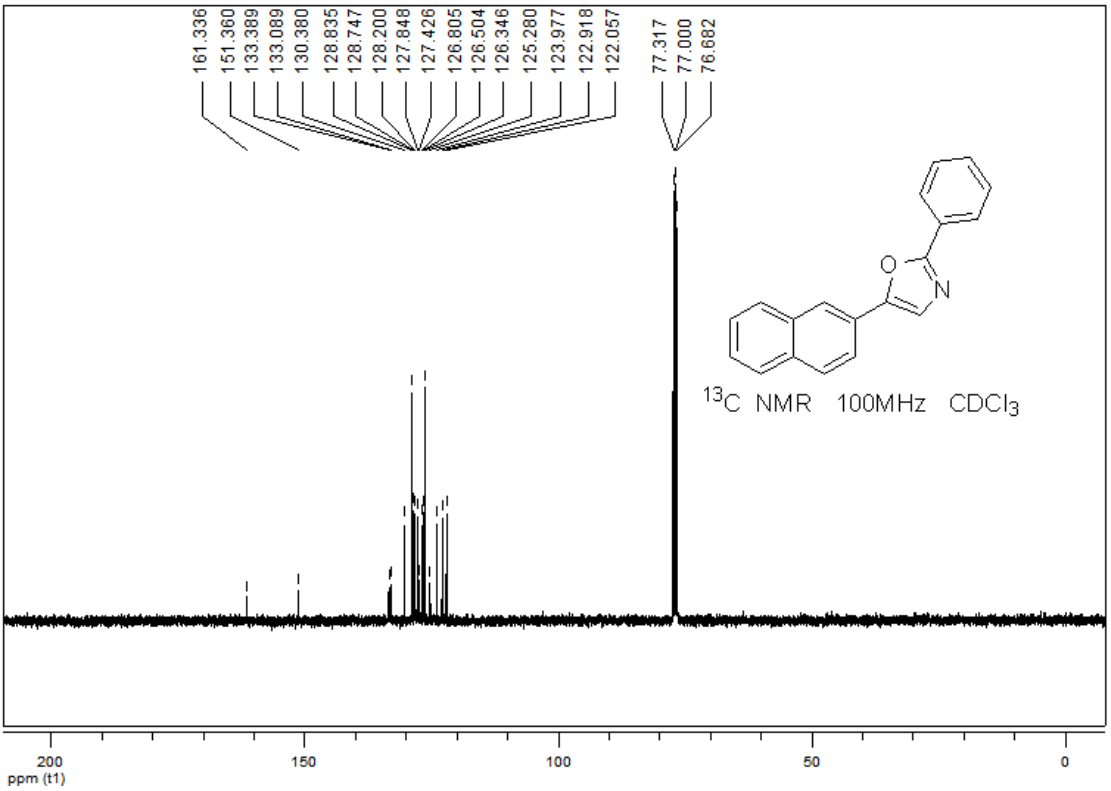

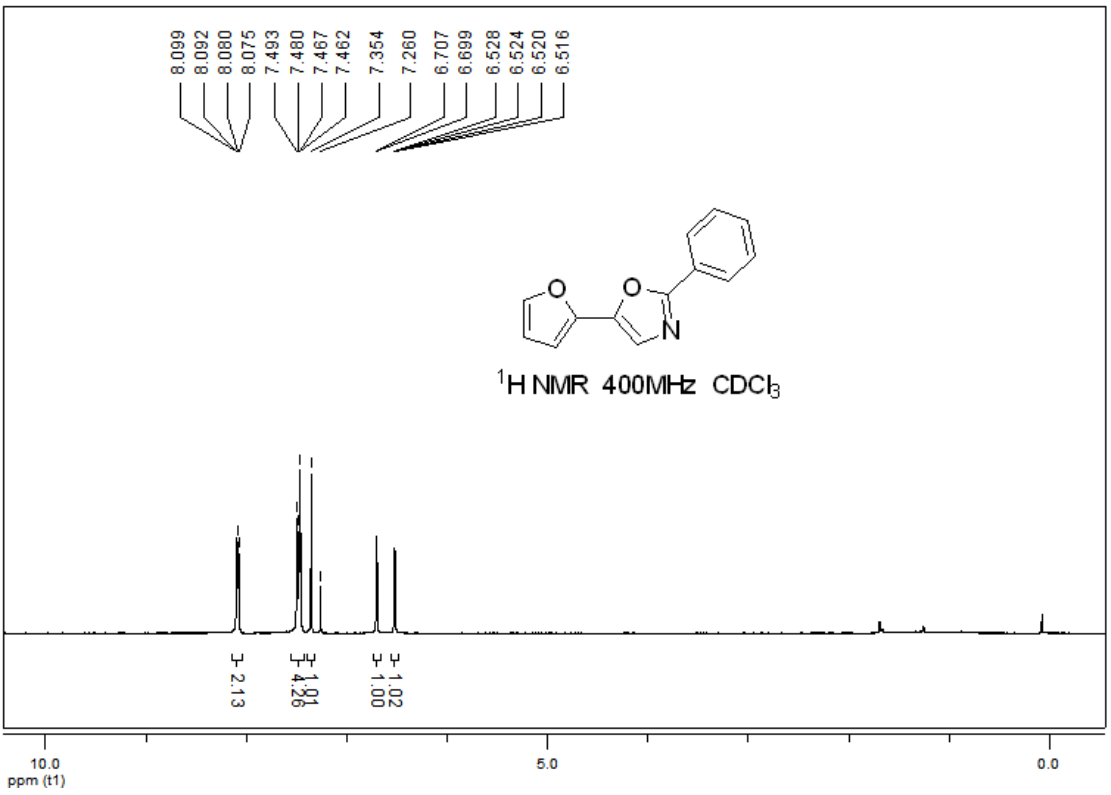

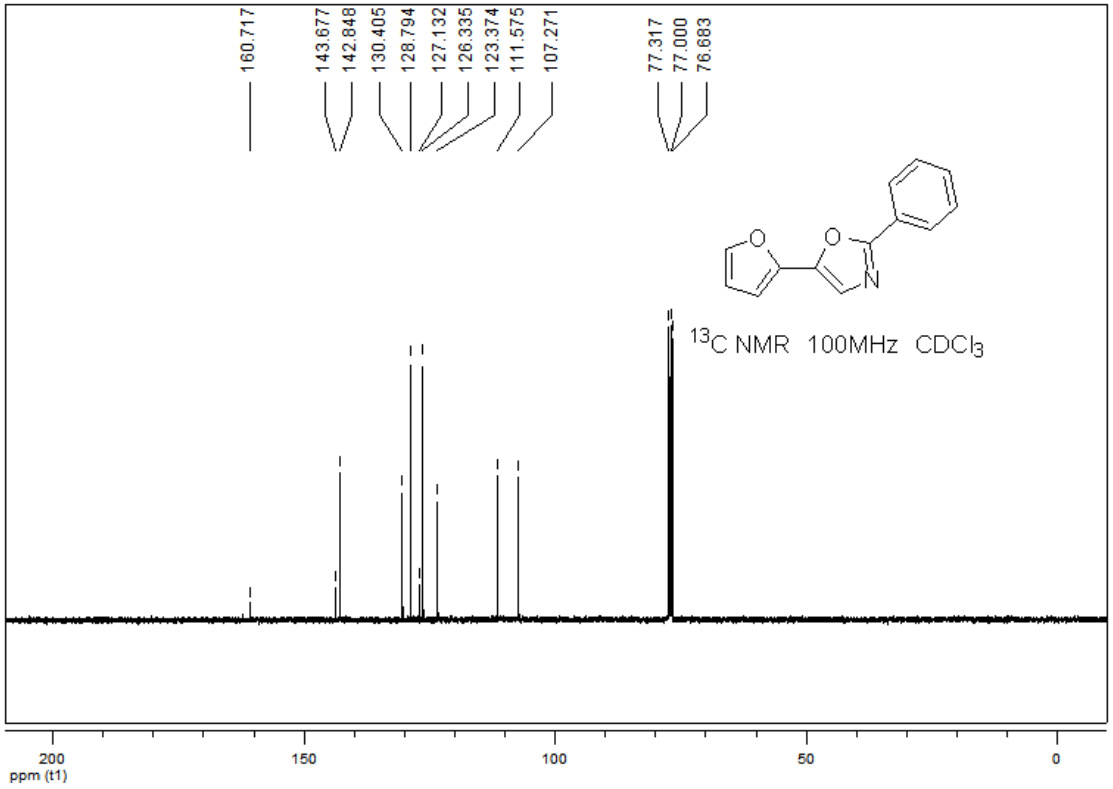
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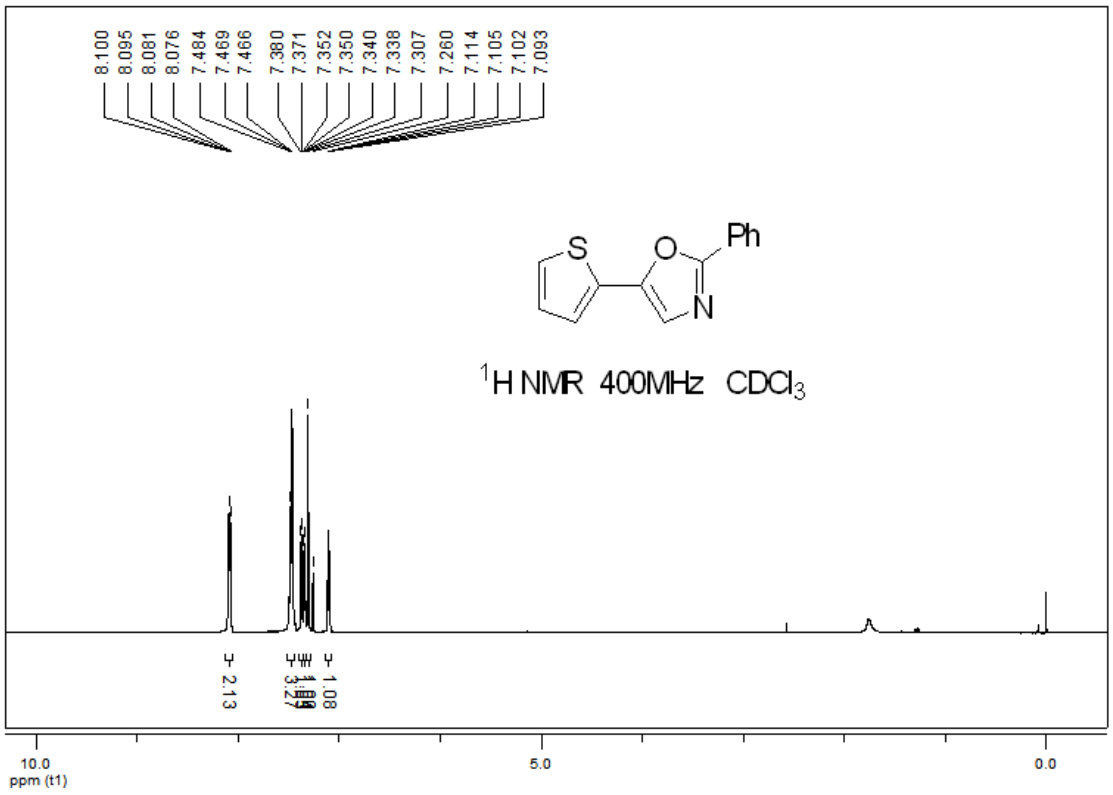
ppm (t1)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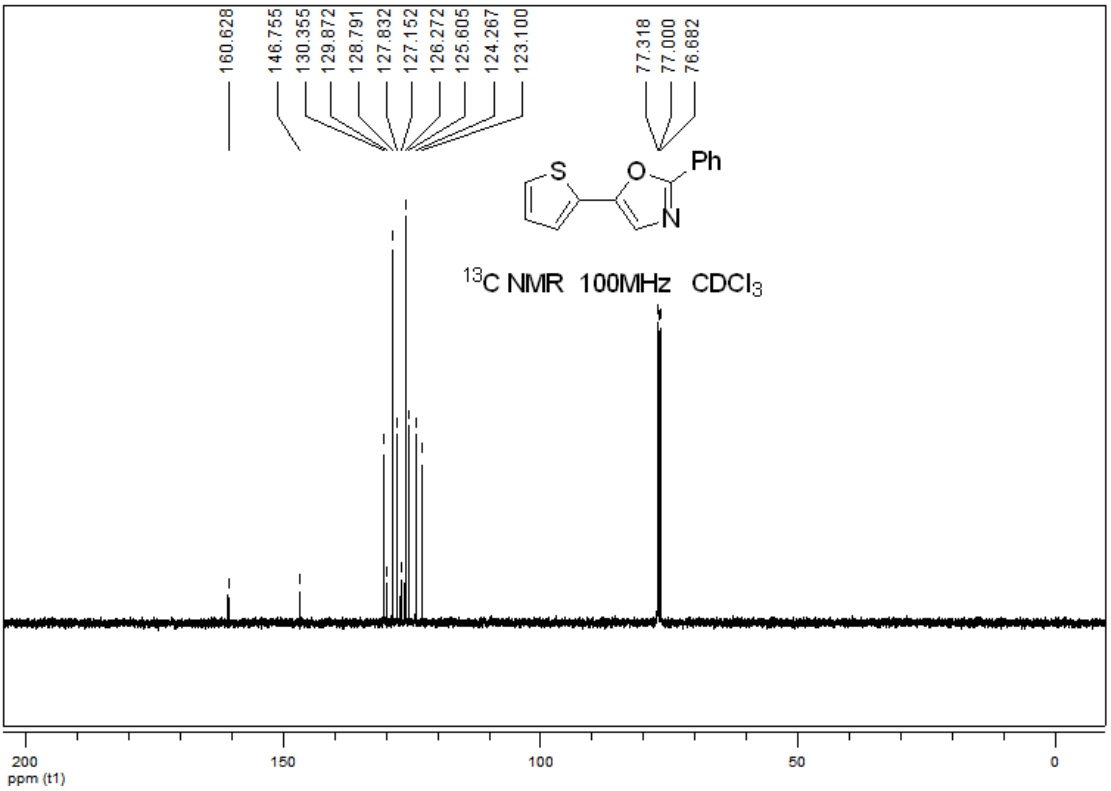
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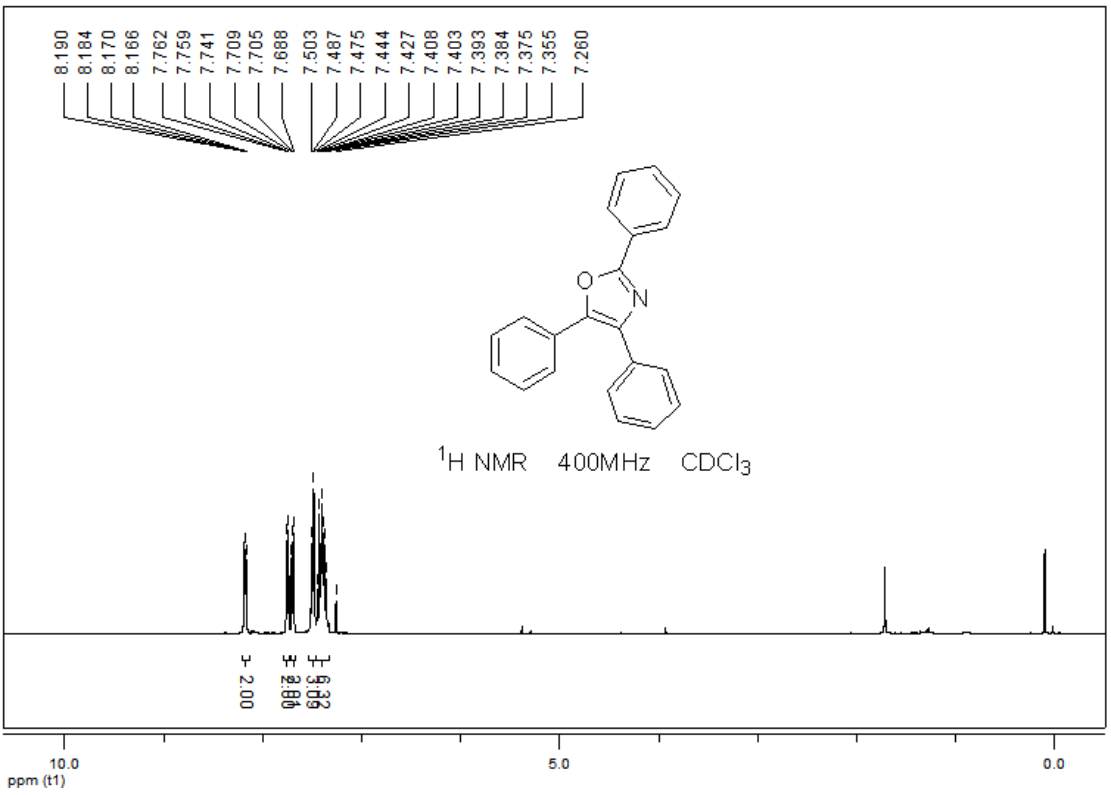
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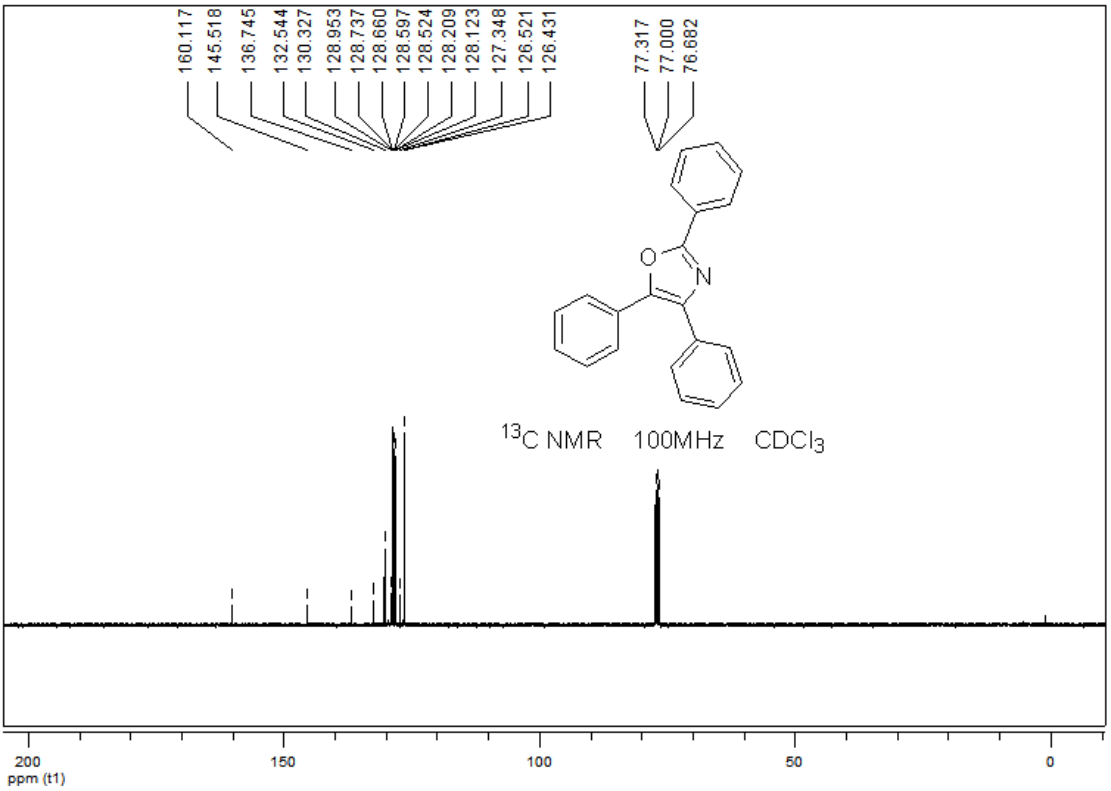

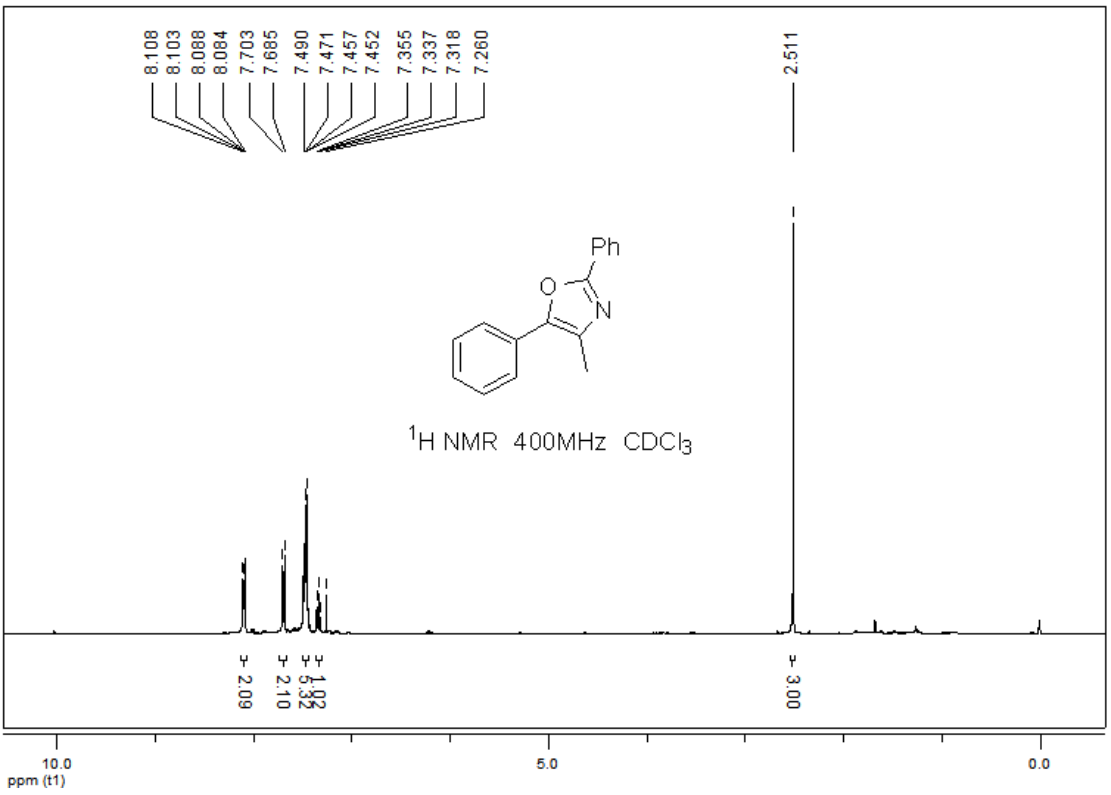

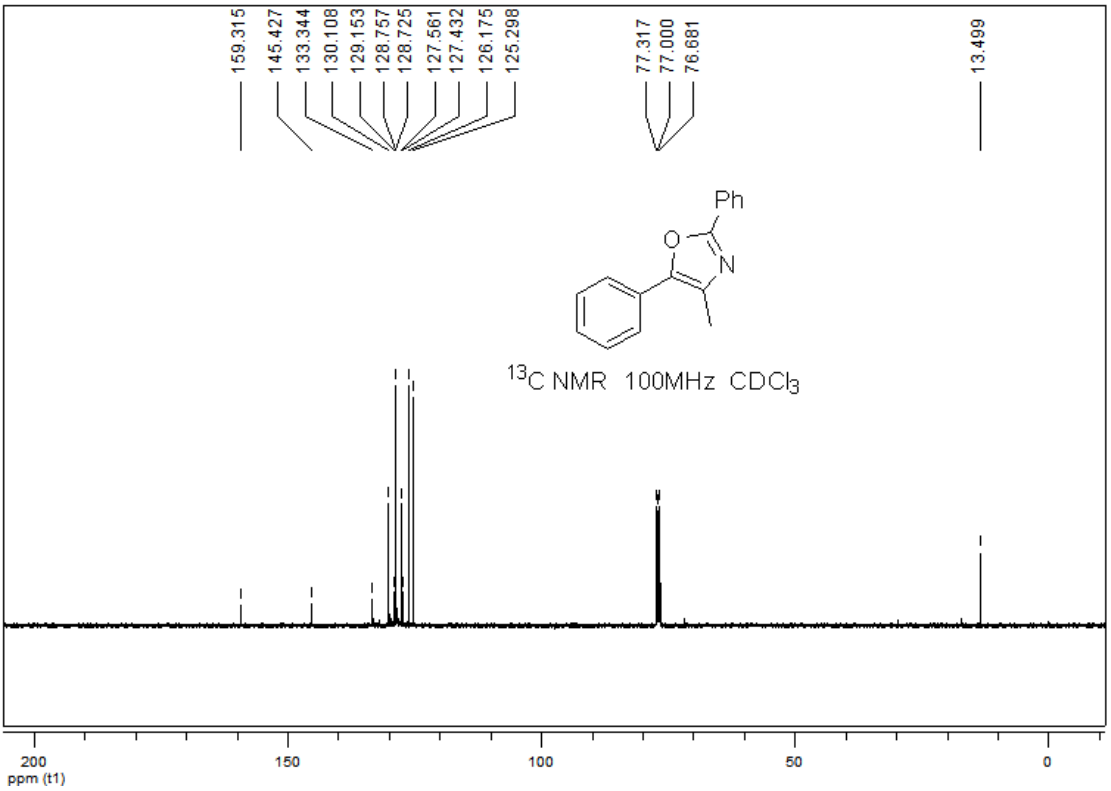

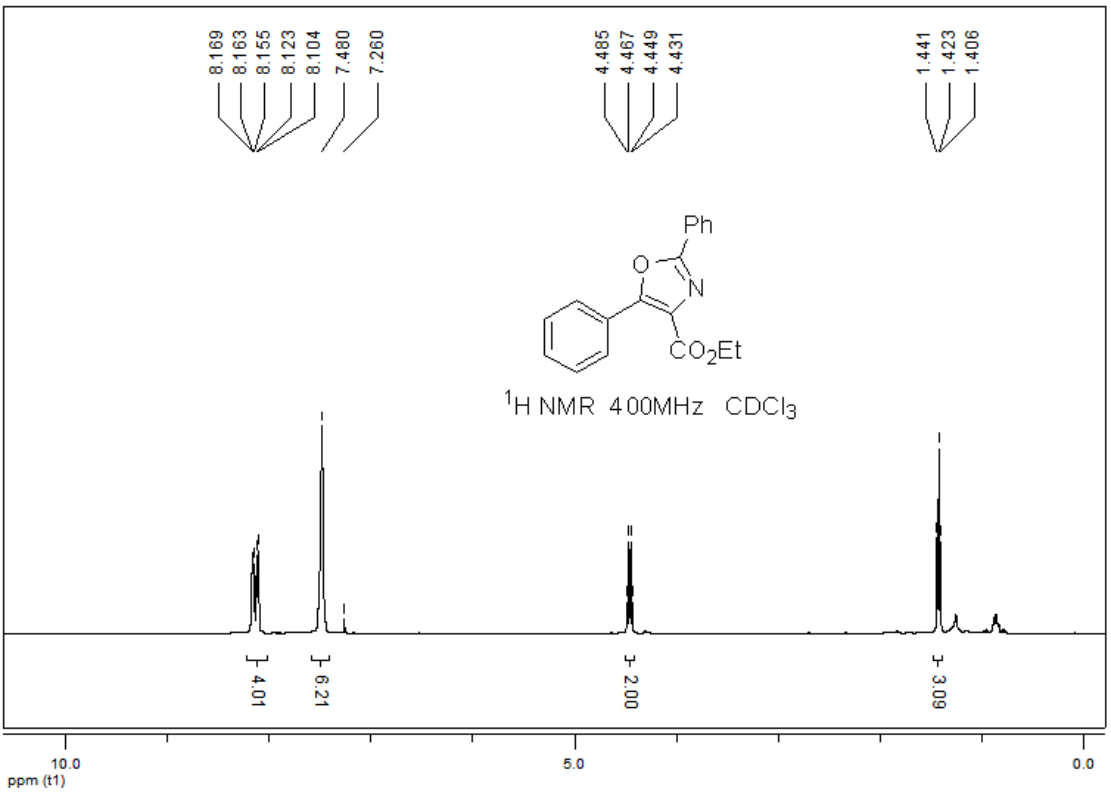

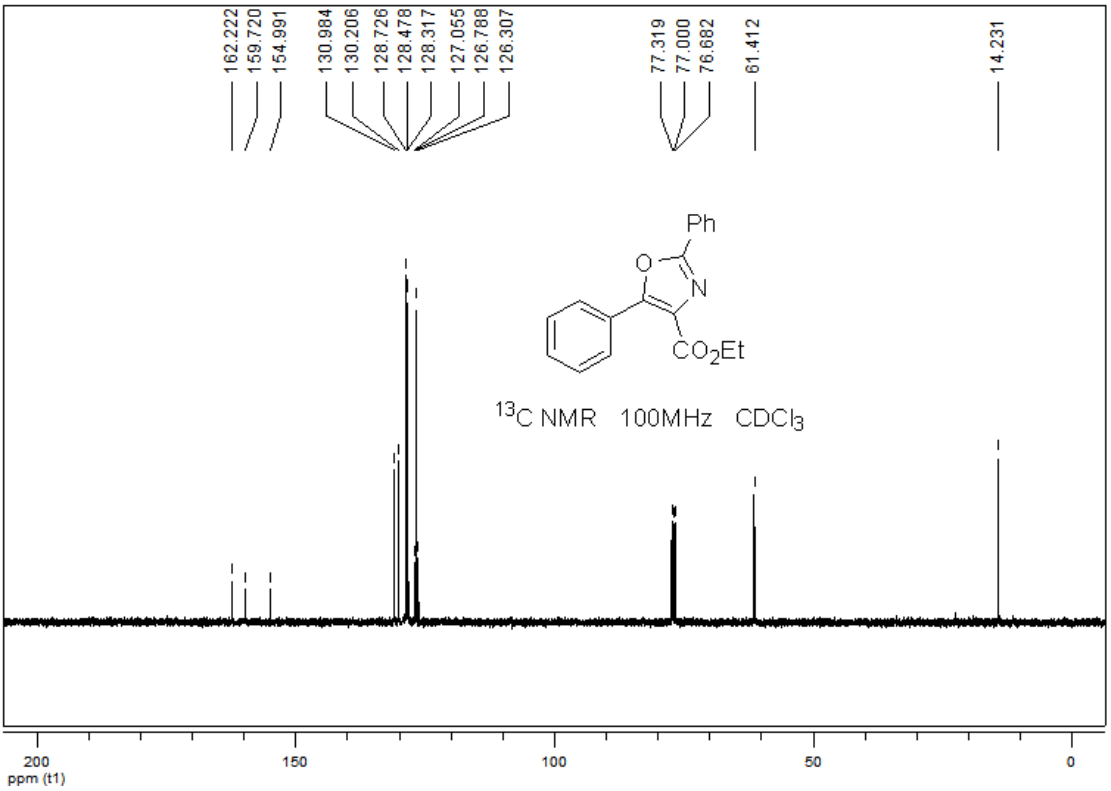

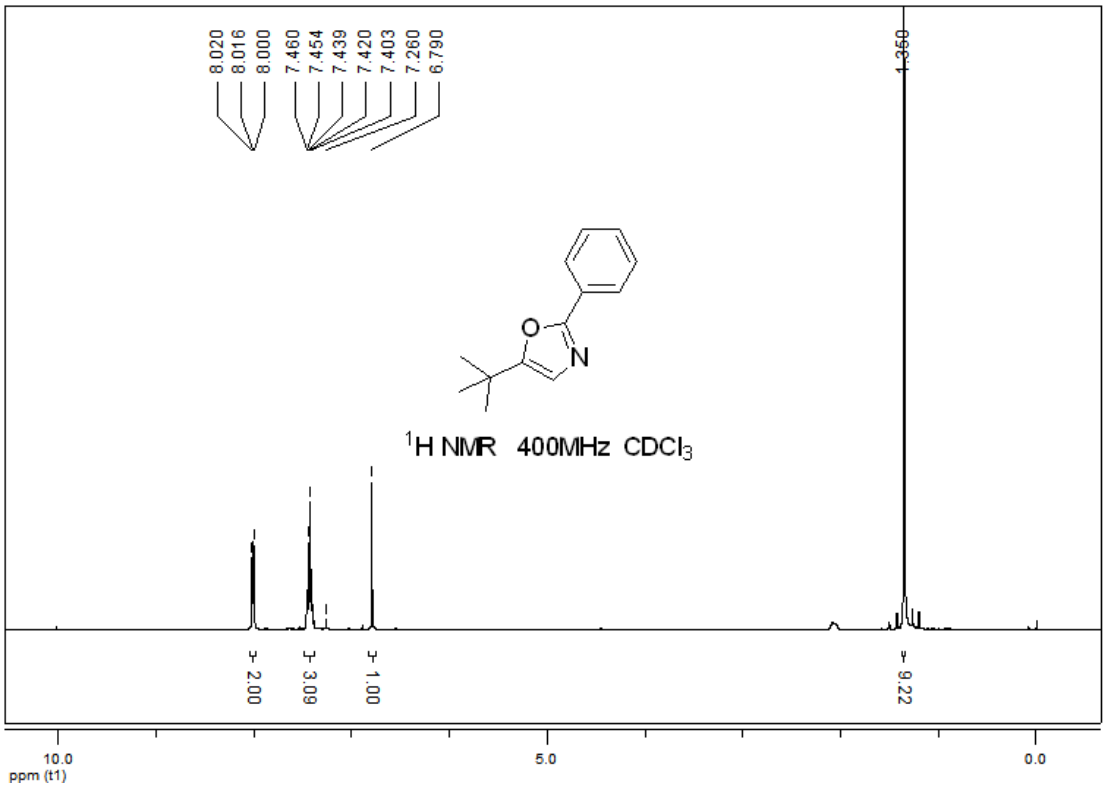

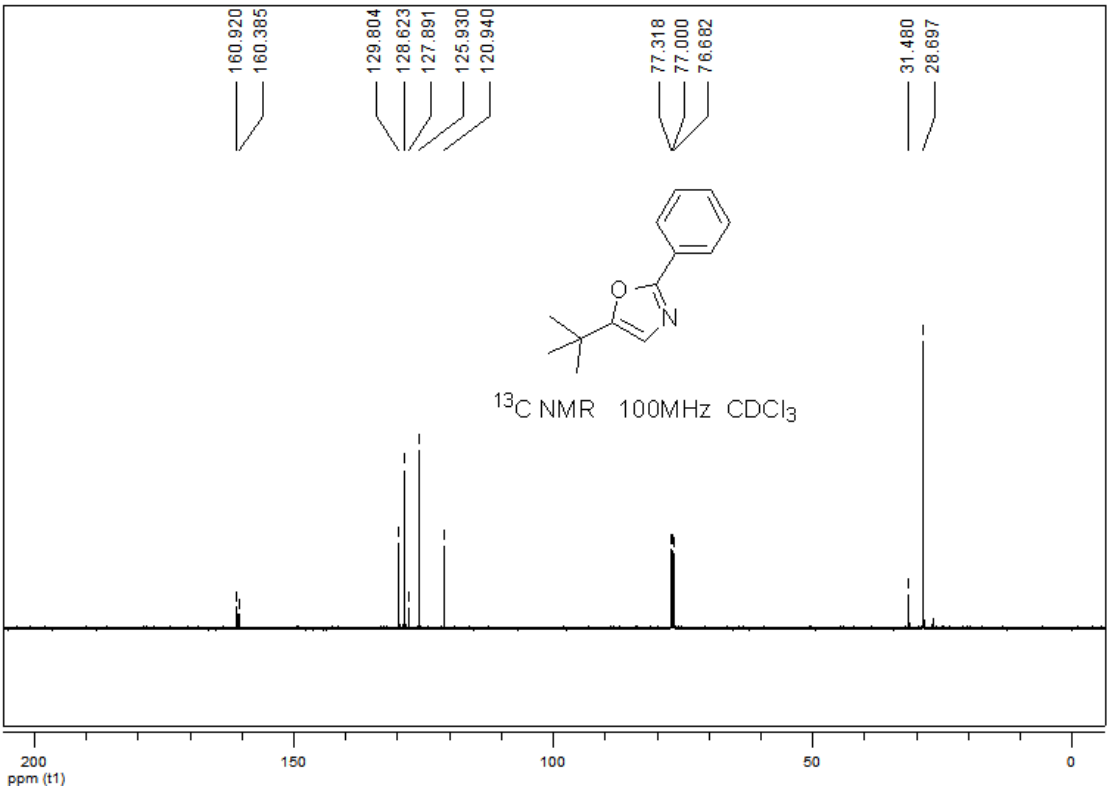

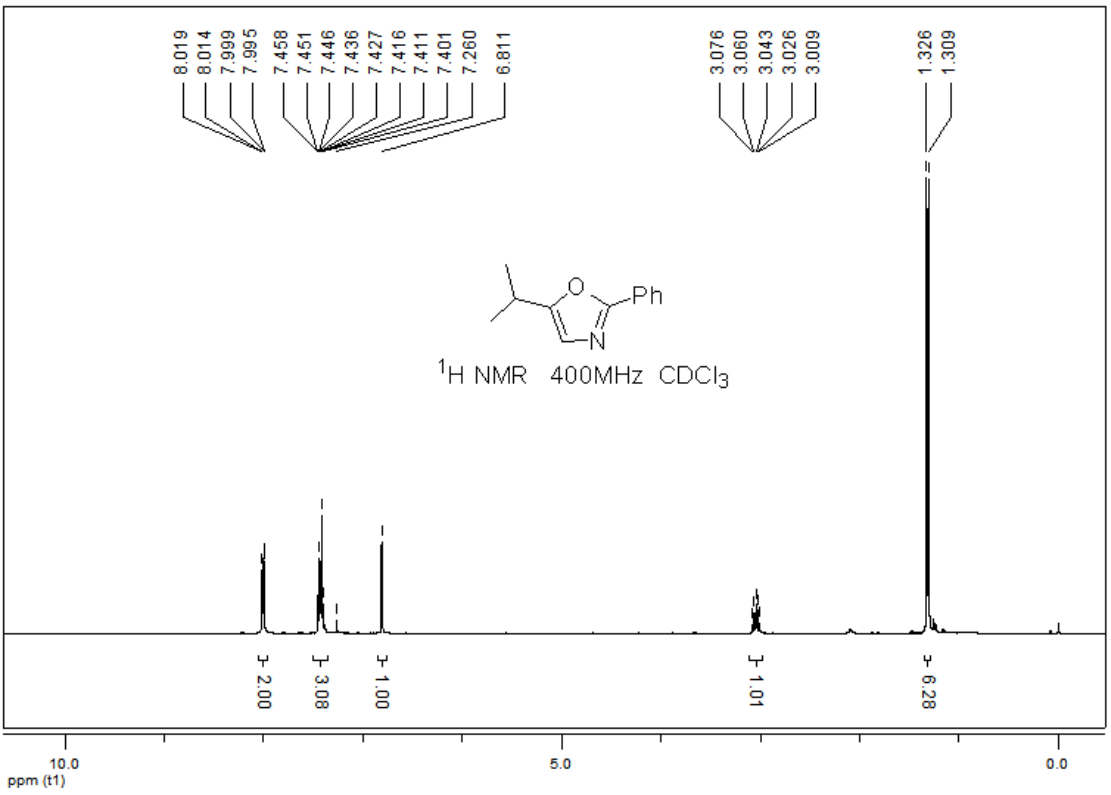

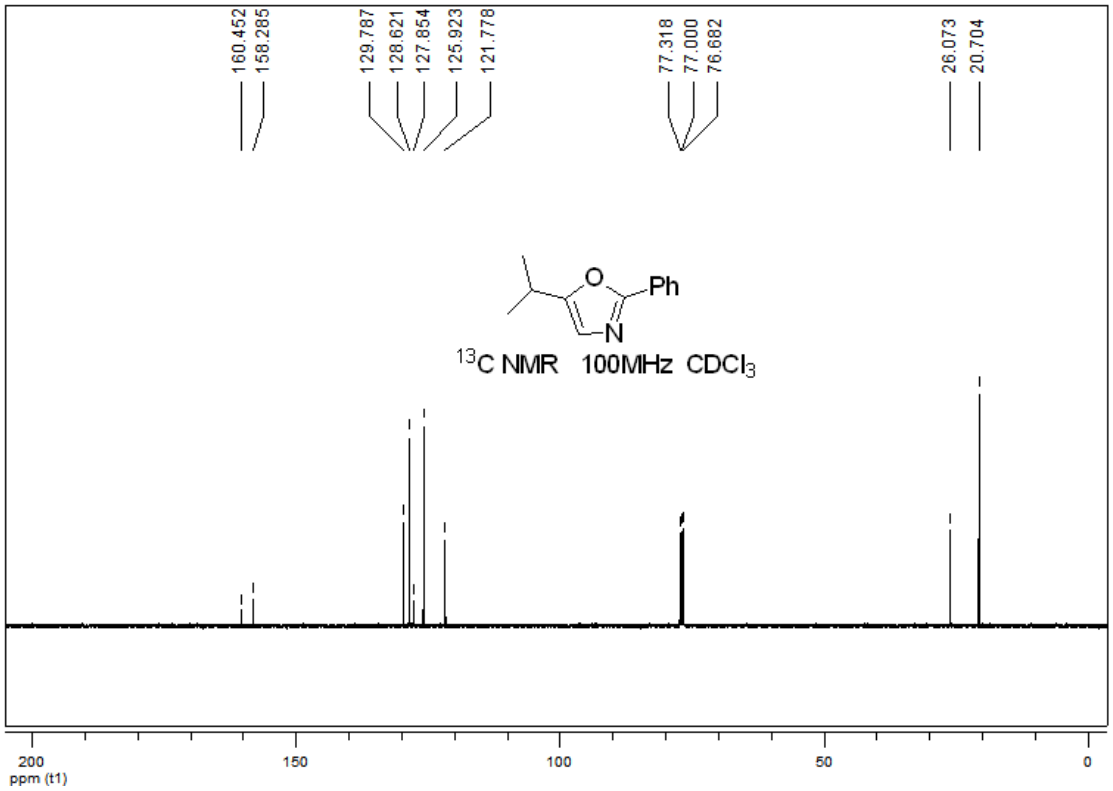

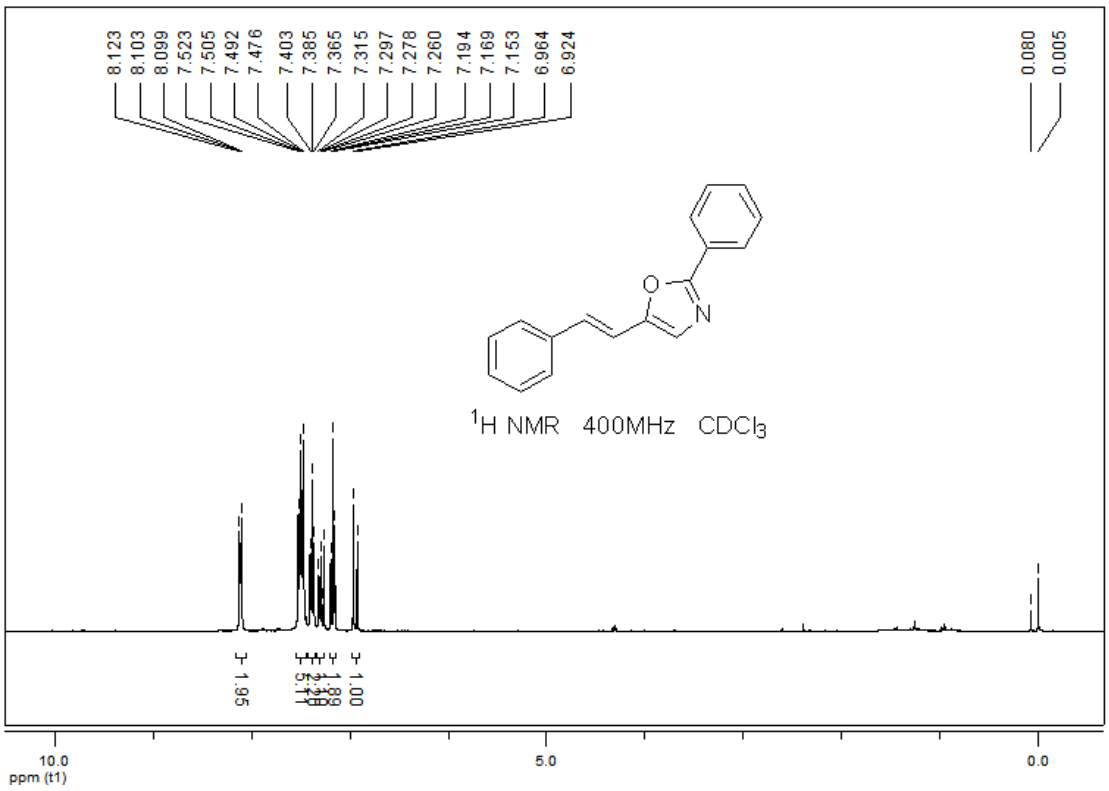

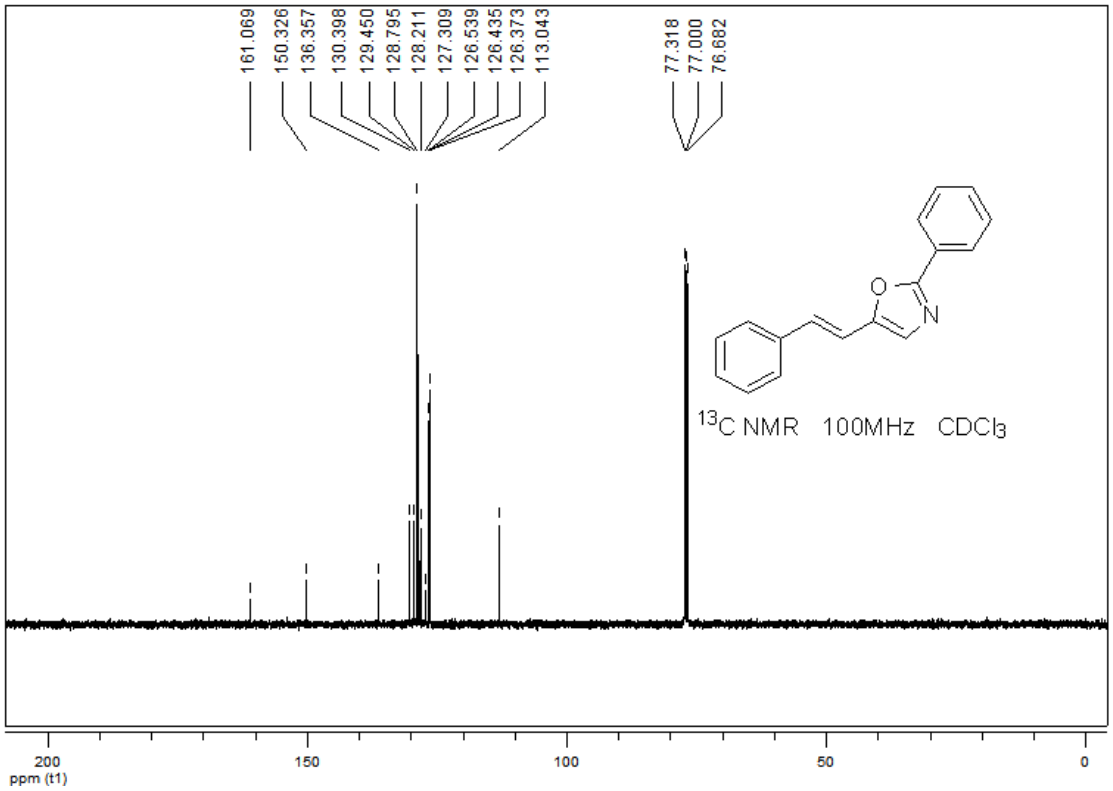
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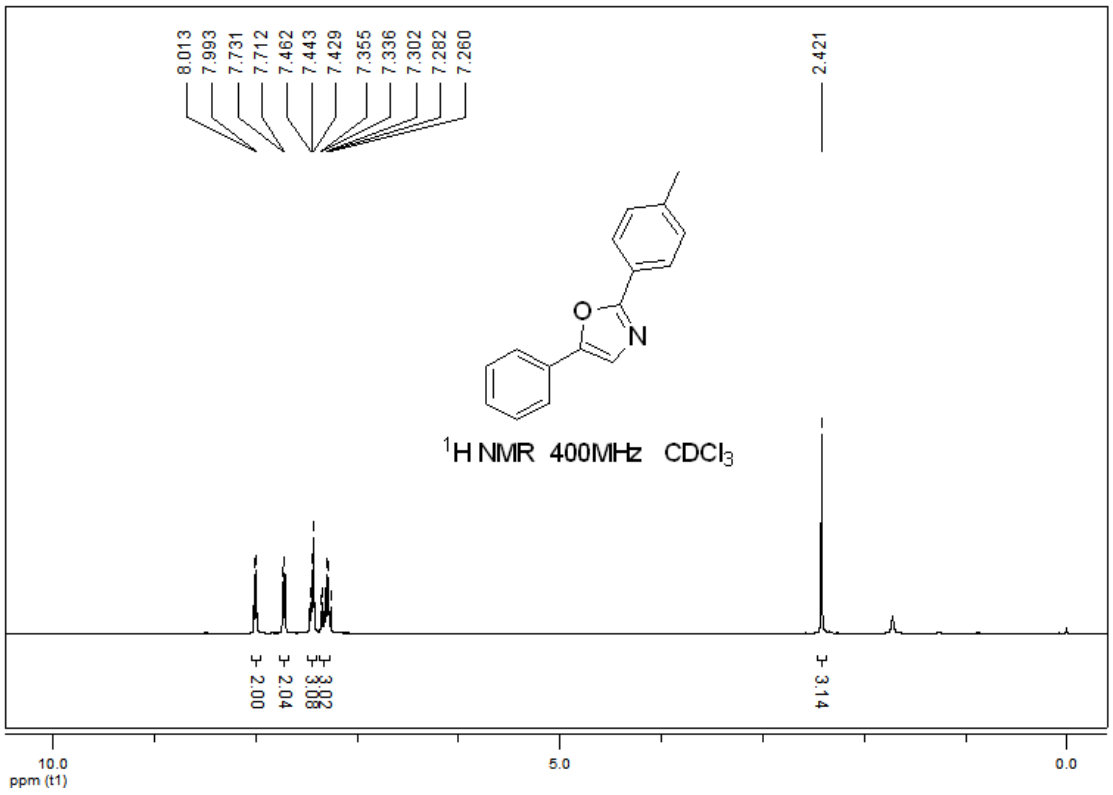
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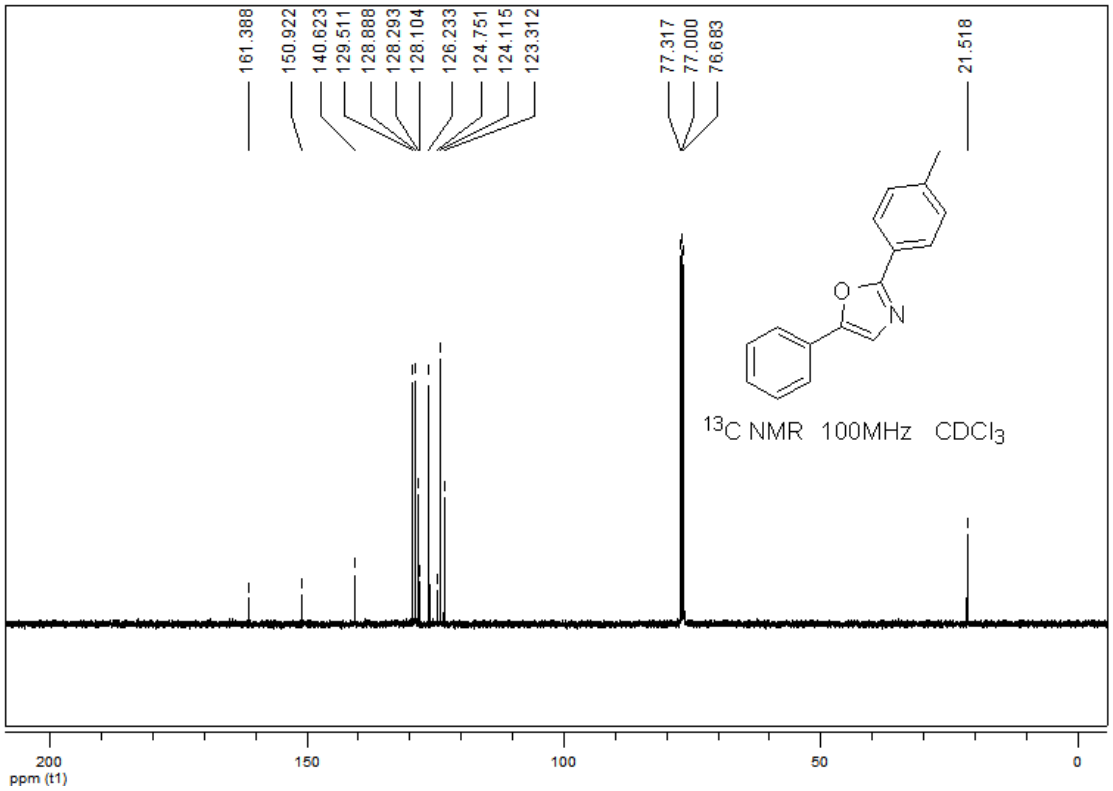

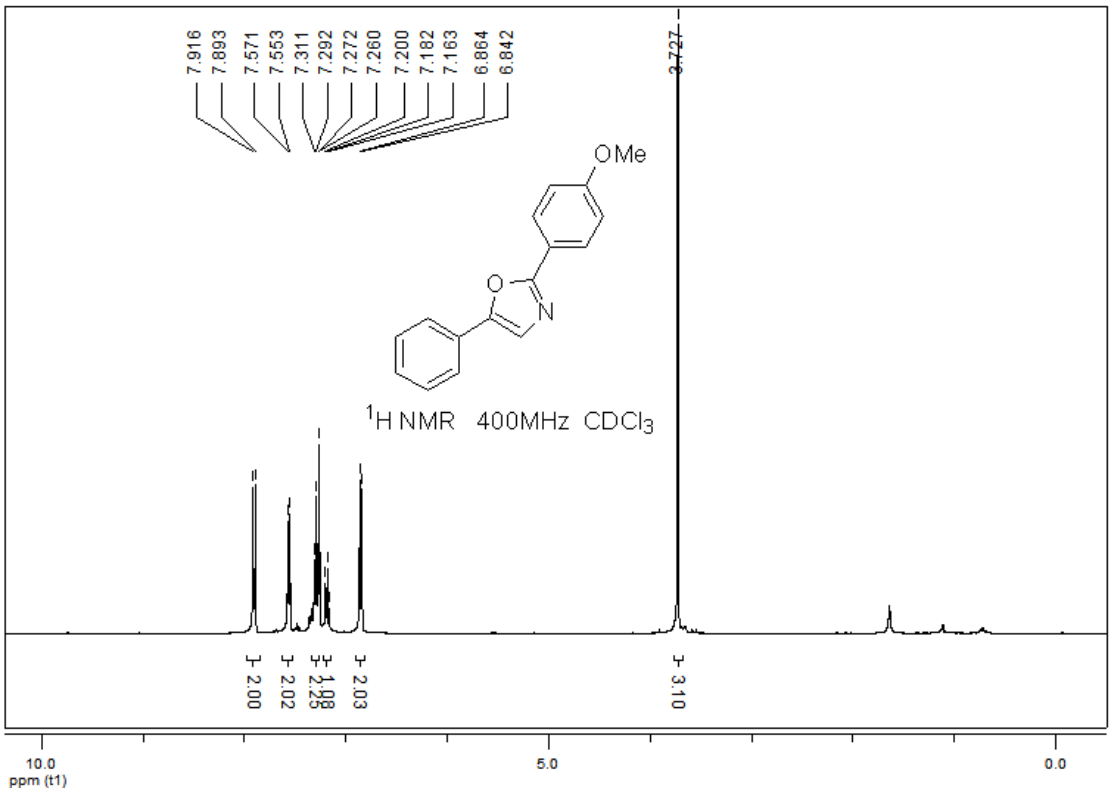

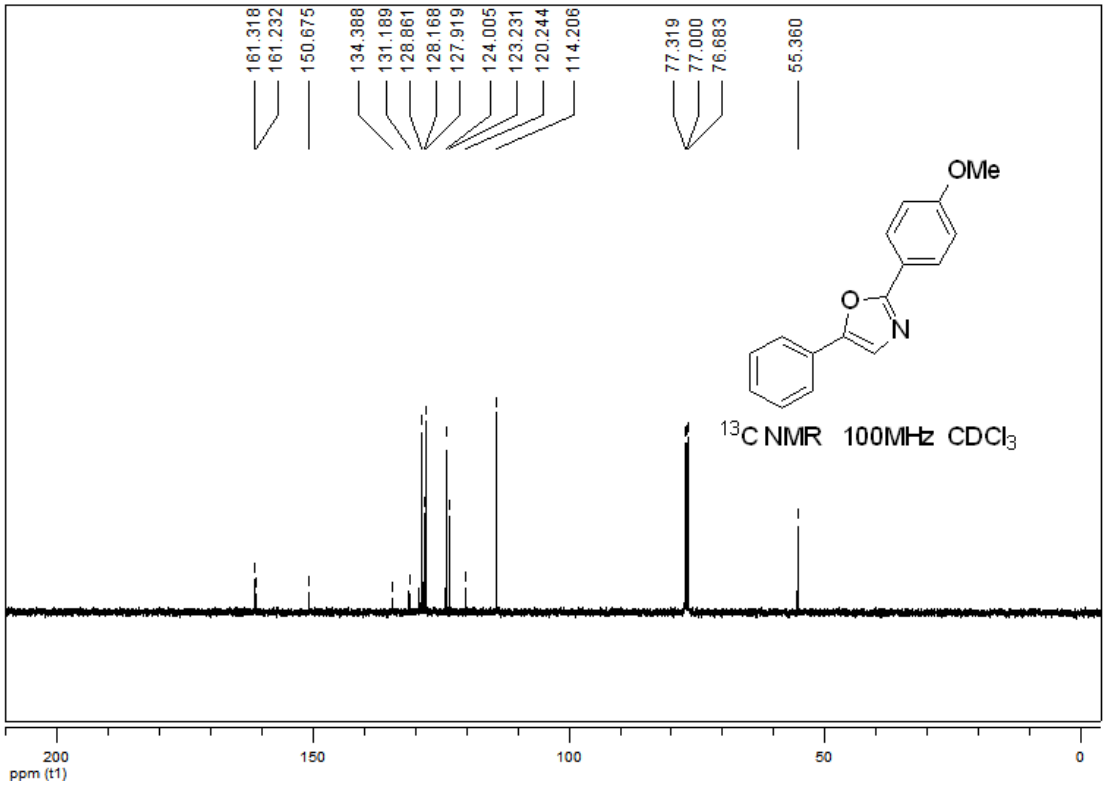

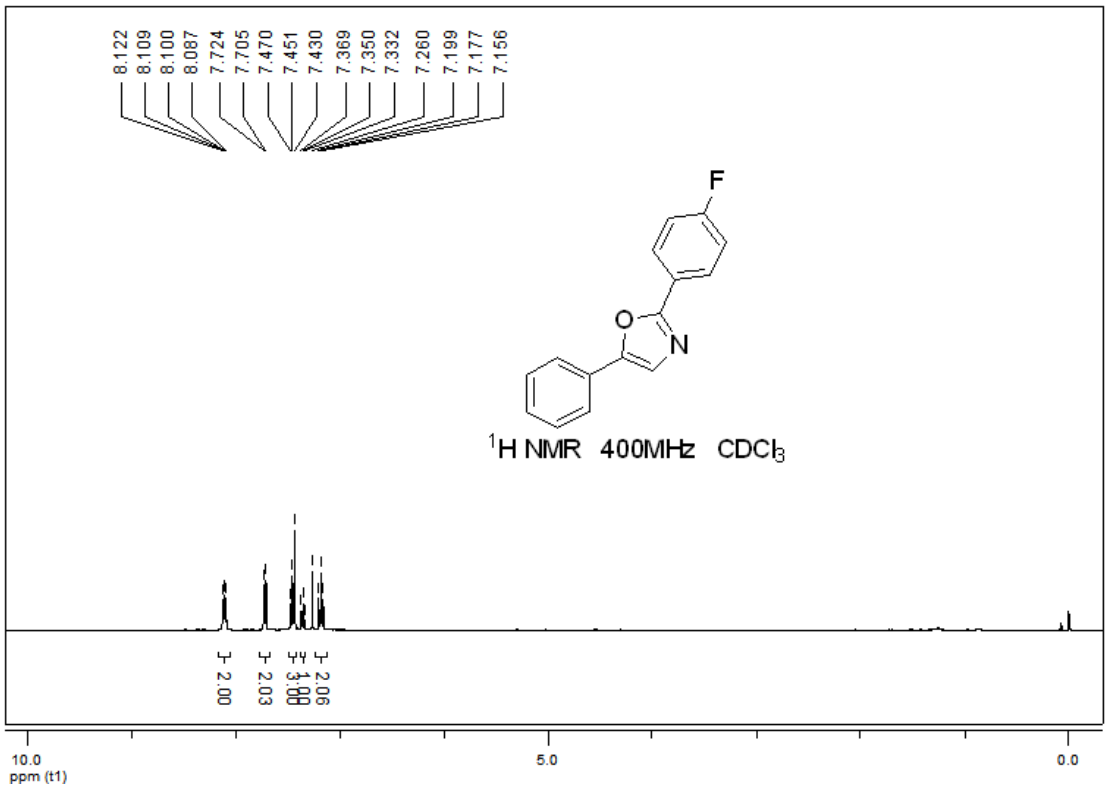

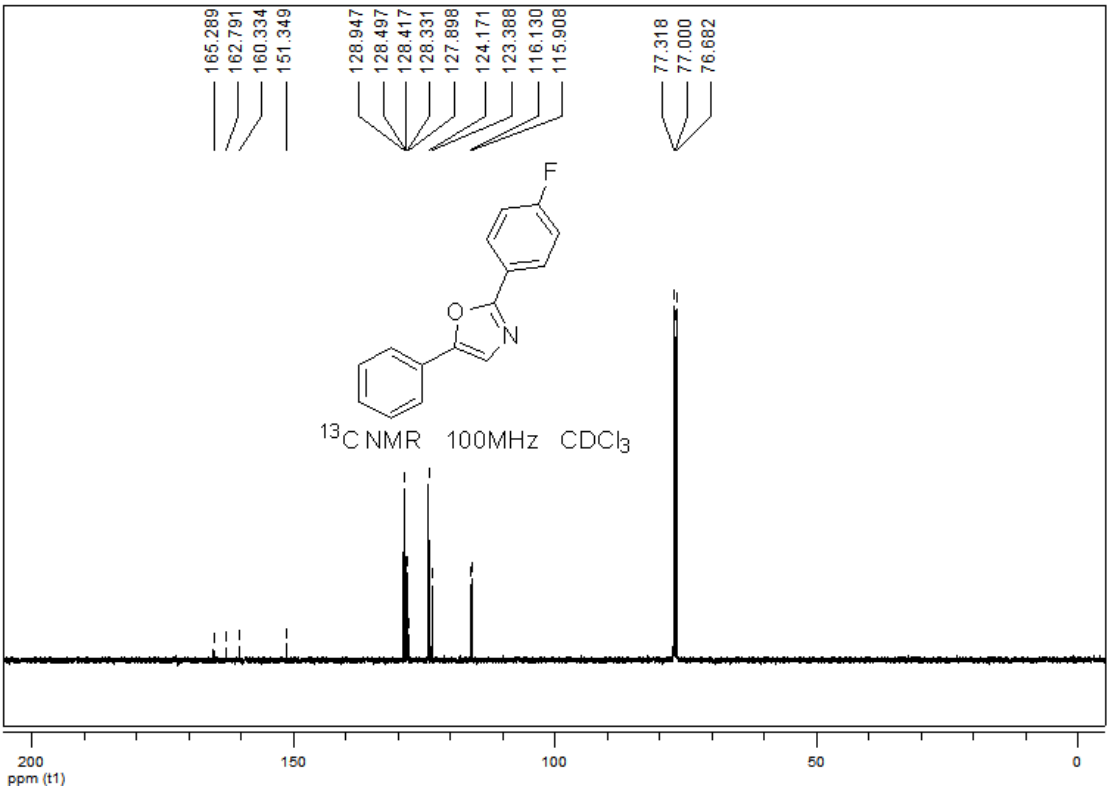
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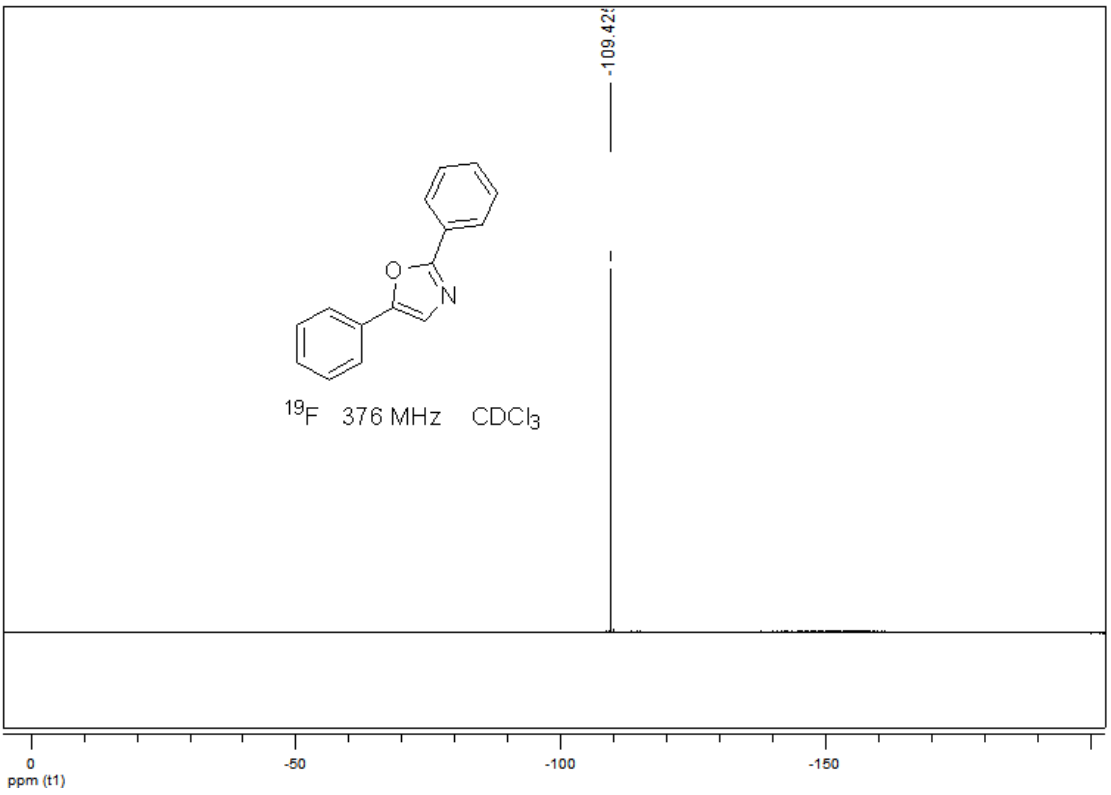
ppm (t1)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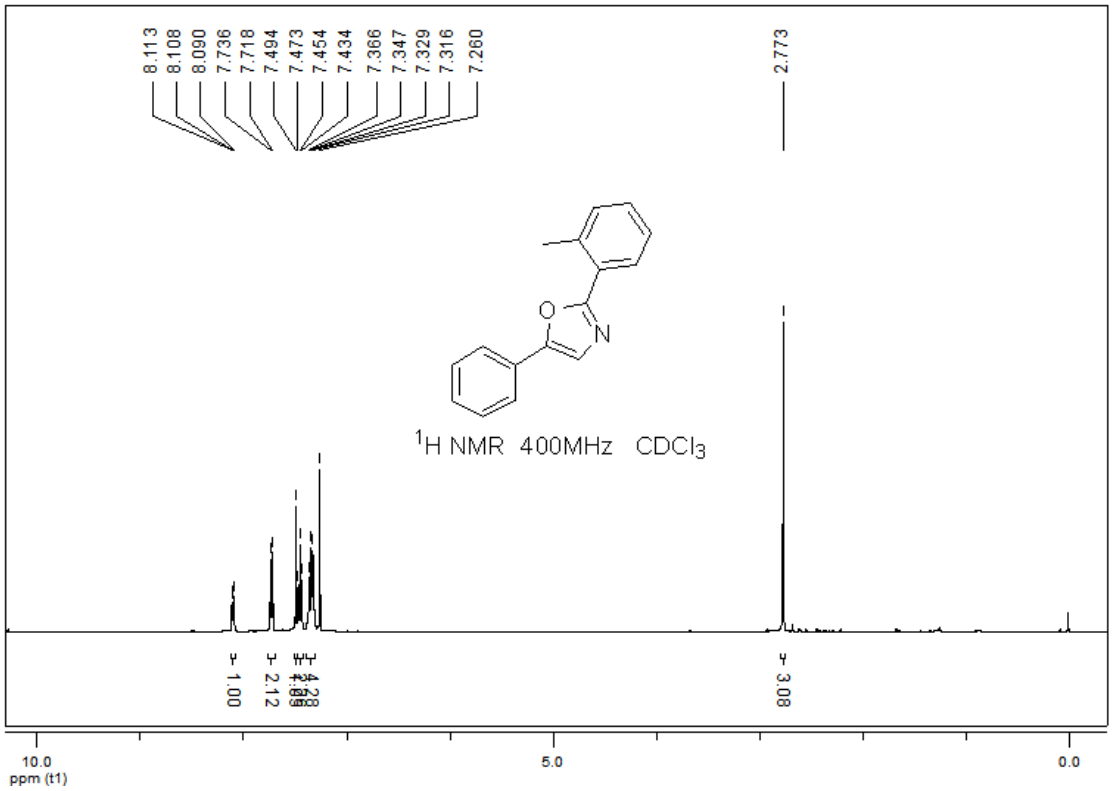
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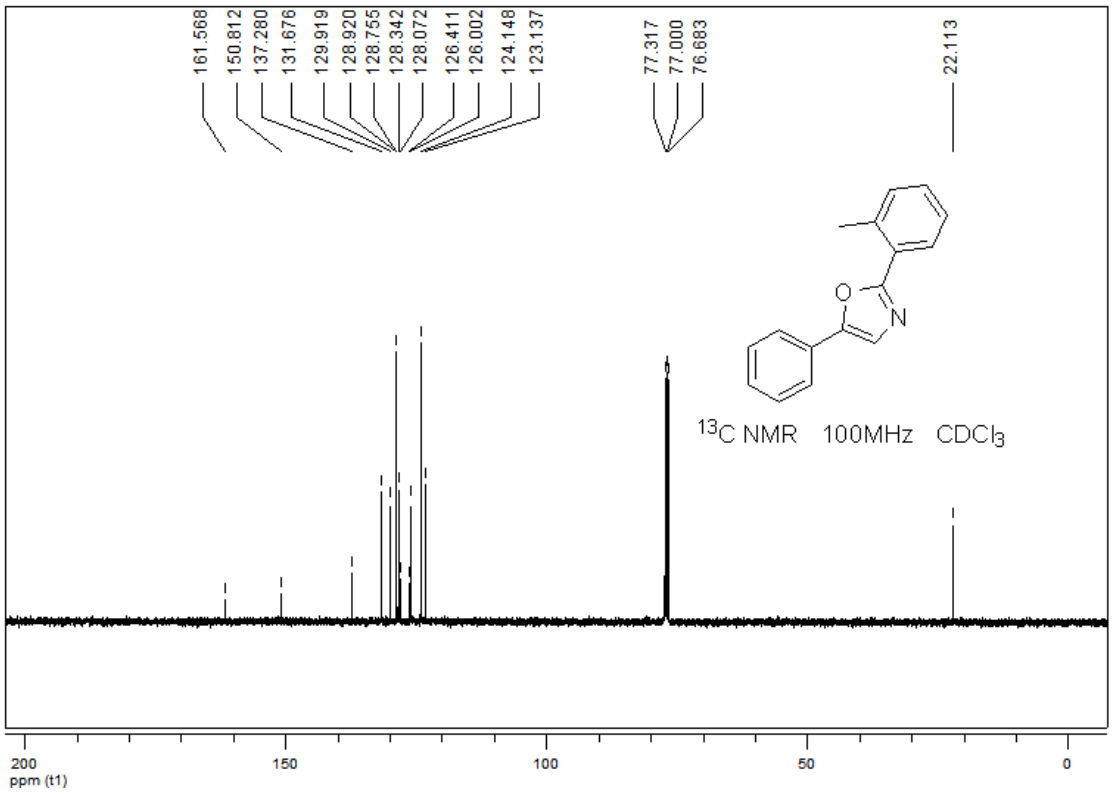
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50
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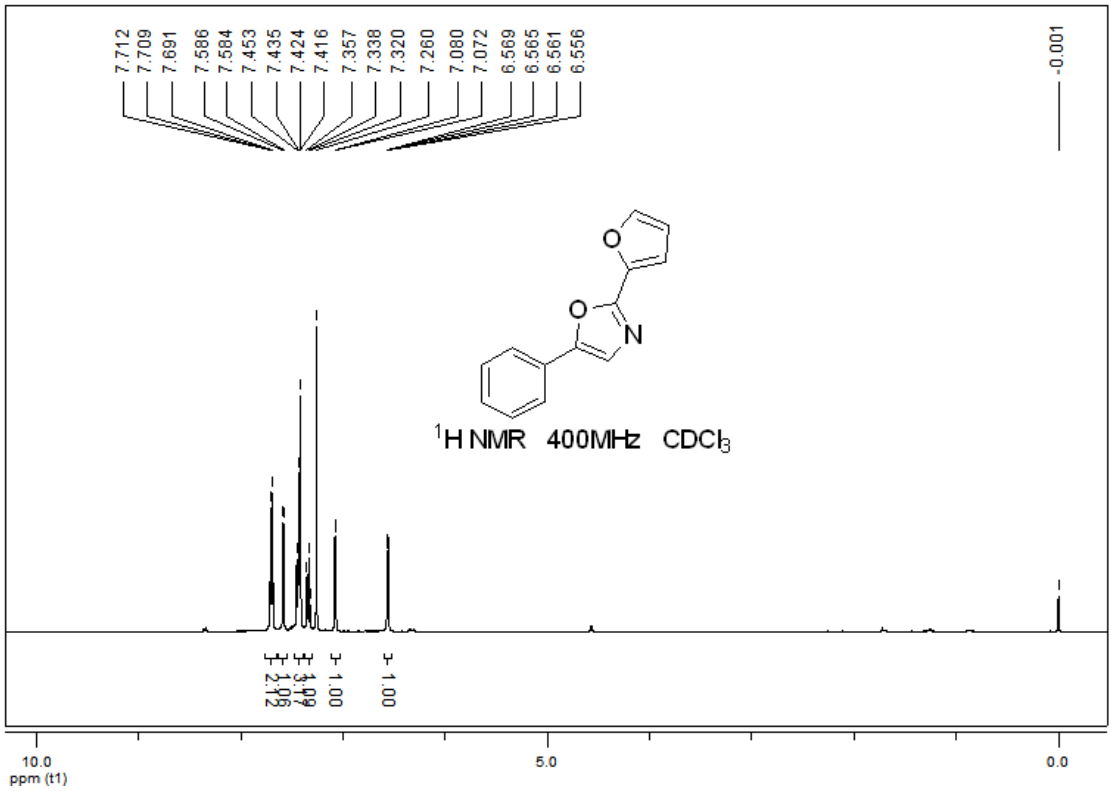
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7.080
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O
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ppm (t1)
5.0
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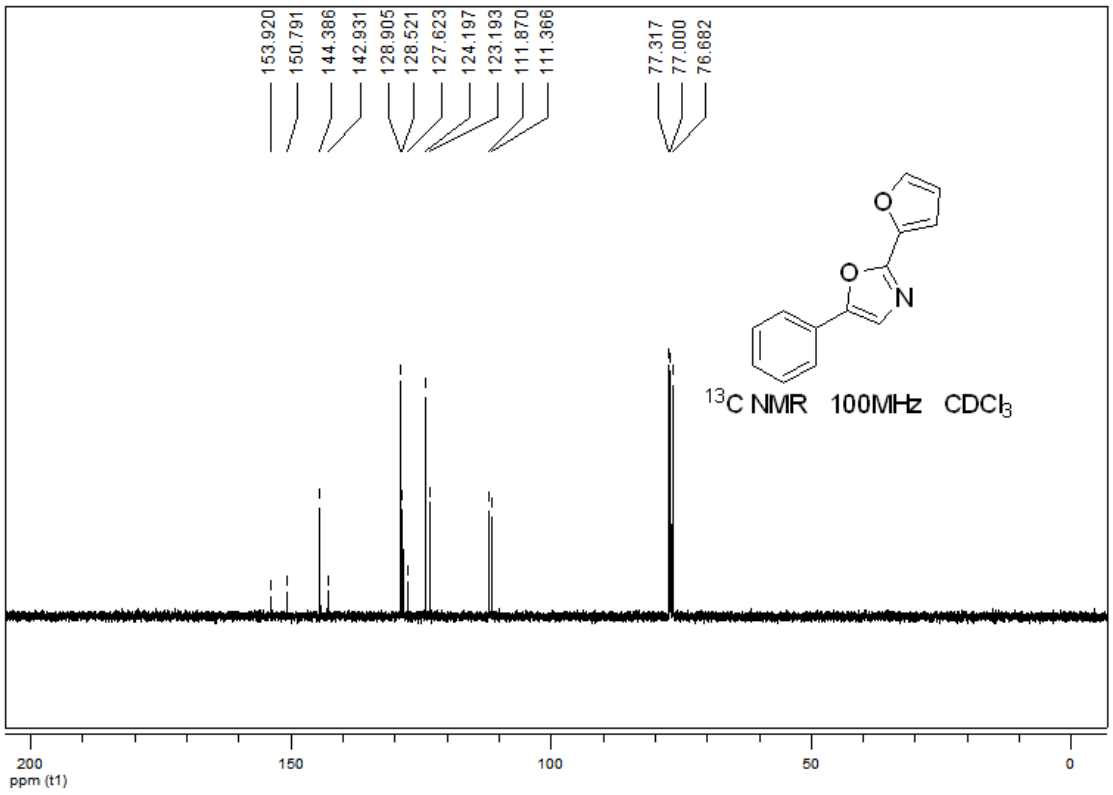
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O
N
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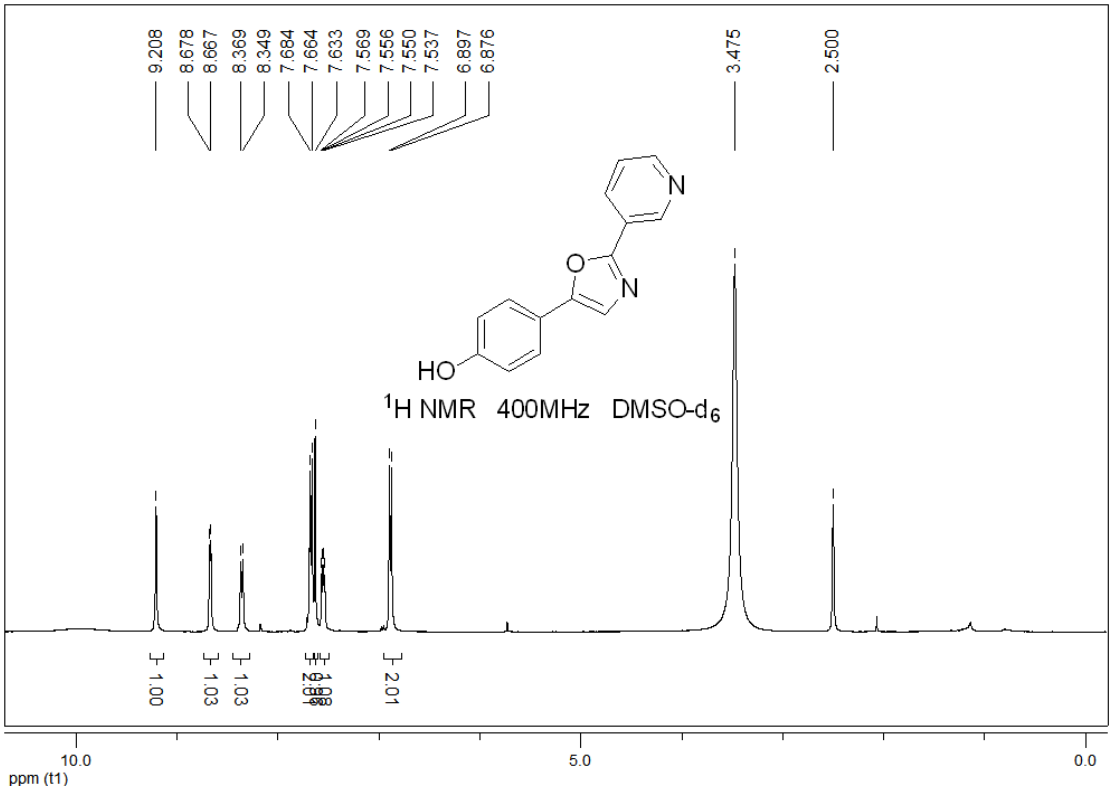
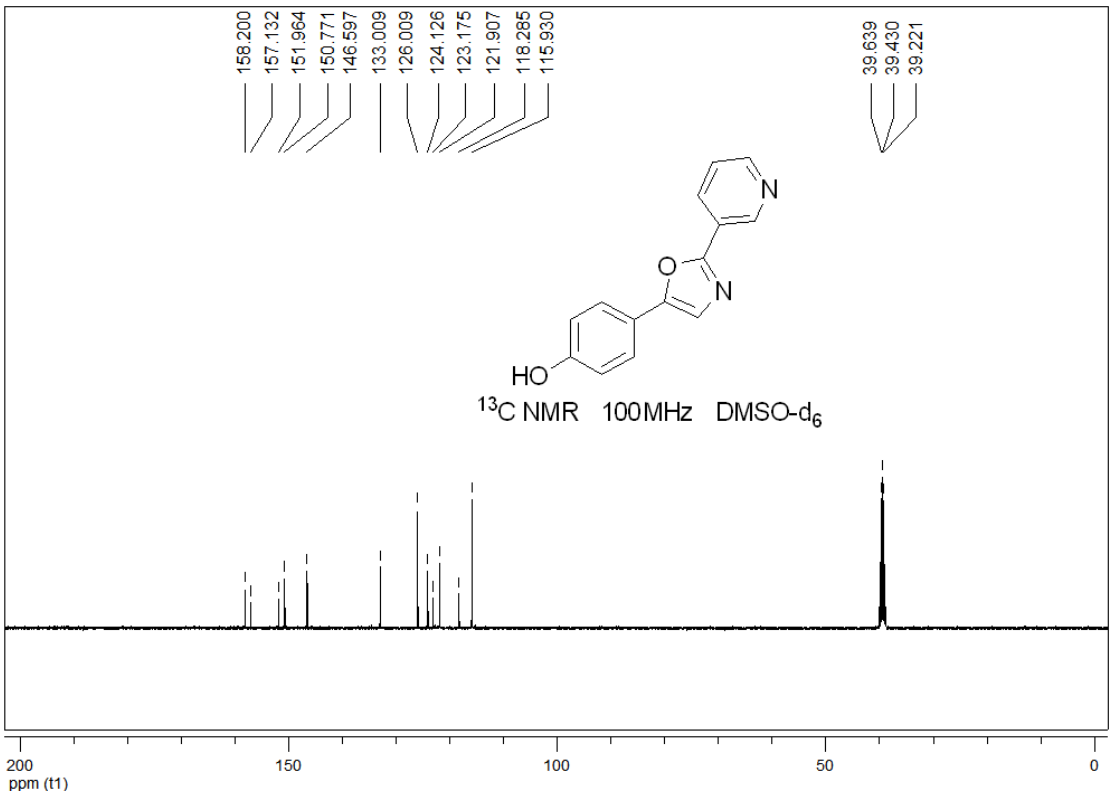
200
ppm (t1)
150
100
50
0


9.208
8.678
8.667
8.369
8.349
7.684
7.664
7.633
7.569
7.556
7.550
7.537
6.897
6.876
3.475
2.500
N
O
N
HO
1H NMR 400MHz DMSO-d6
1.00
1.03
1.03
2.01
10.0
5.0
0.0
ppm (t1)


158.200
157.132
151.964
150.771
146.597
133.009
126.009
124.126
123.175
121.907
118.285
115.930
39.639
39.430
39.221
N
O
N
HO
13C NMR 100MHz DMSO-d6
200
150
100
50
0
ppm (t1)