

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Hypervalent Iodine(III)-Mediated Cyclopropa(e)nation of Alkenes/Alkynes Under Mild Conditions

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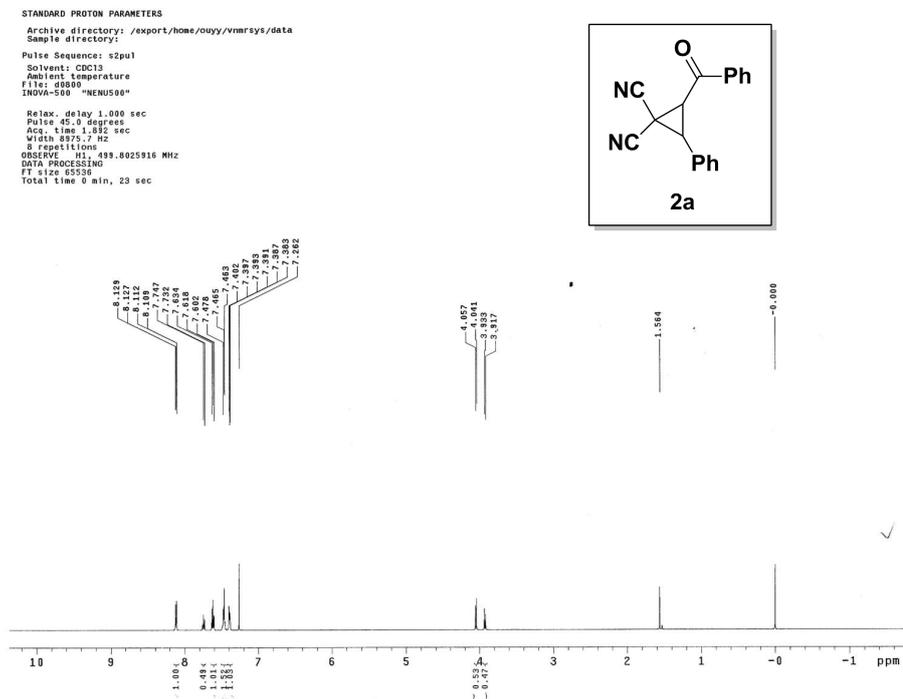
I. General information:

All reagents were purchased from commercial sources and used without further treatment, unless otherwise indicated. The products were purified by column chromatography over silica gel. ^1H NMR and ^{13}C NMR spectra were recorded at 25 °C on a Varian 500 MHz and 125 MHz, respectively, with TMS as the internal standard. Melting points were obtained with a micro melting point XT4A Beijing Keyi electrooptic apparatus and are uncorrected. High resolution mass spectra were recorded on Bruck microtof. All reactions were monitored by TLC with Taizhou GF254 silica gel coated plates. Flash column chromatography was carried out using 200-300 mesh silica gel at increased pressure.

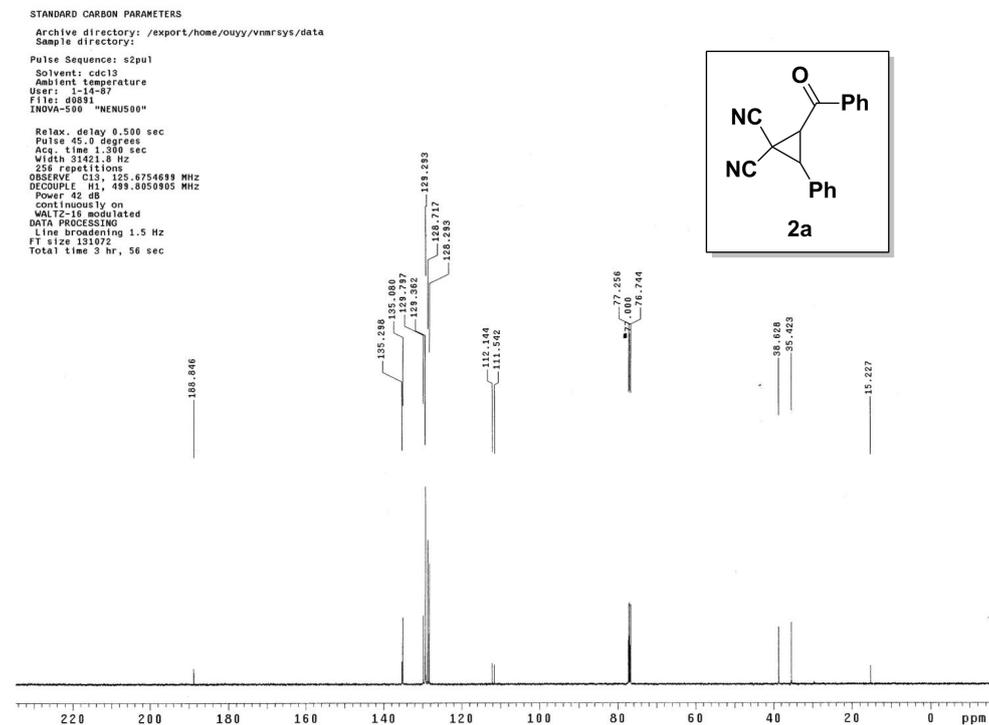
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II. ^1H and ^{13}C NMR spectra of compounds 2, 3, 5, 6 and 7

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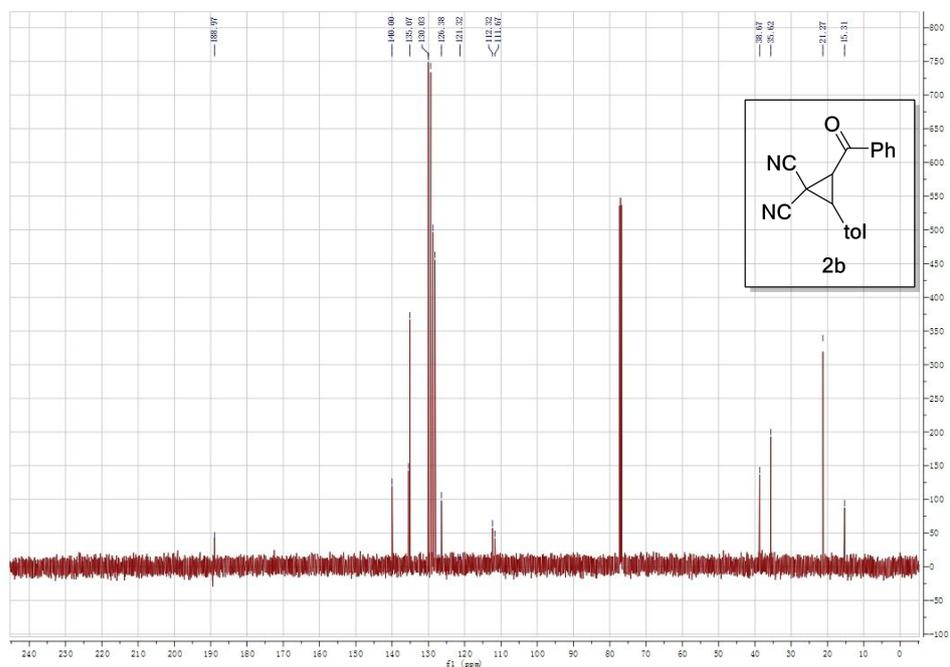
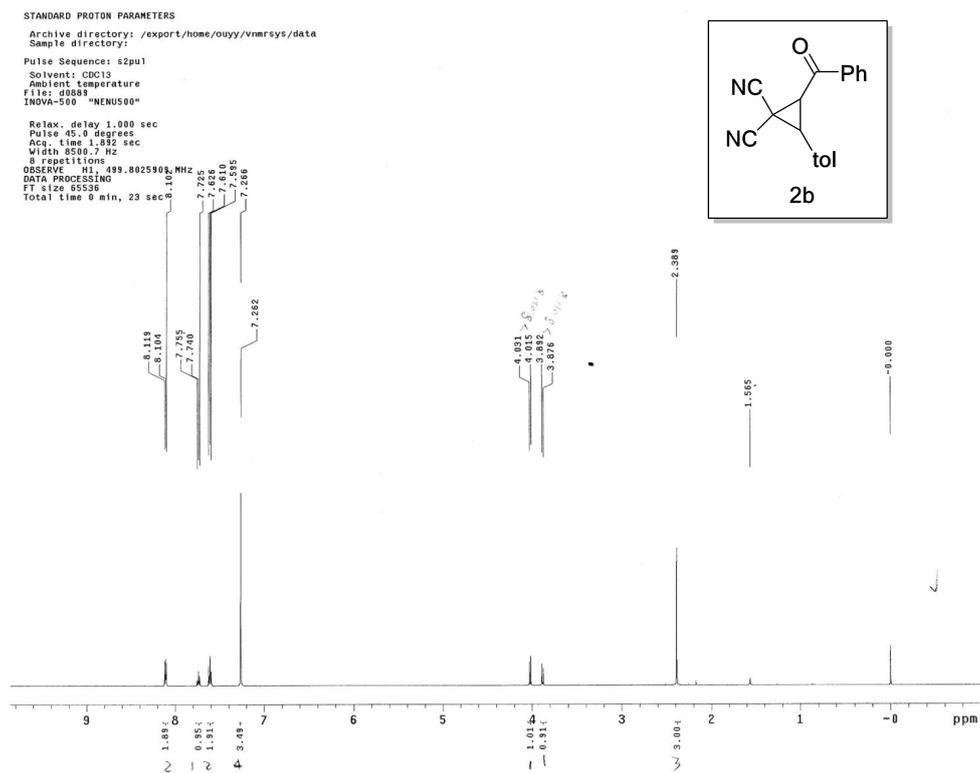


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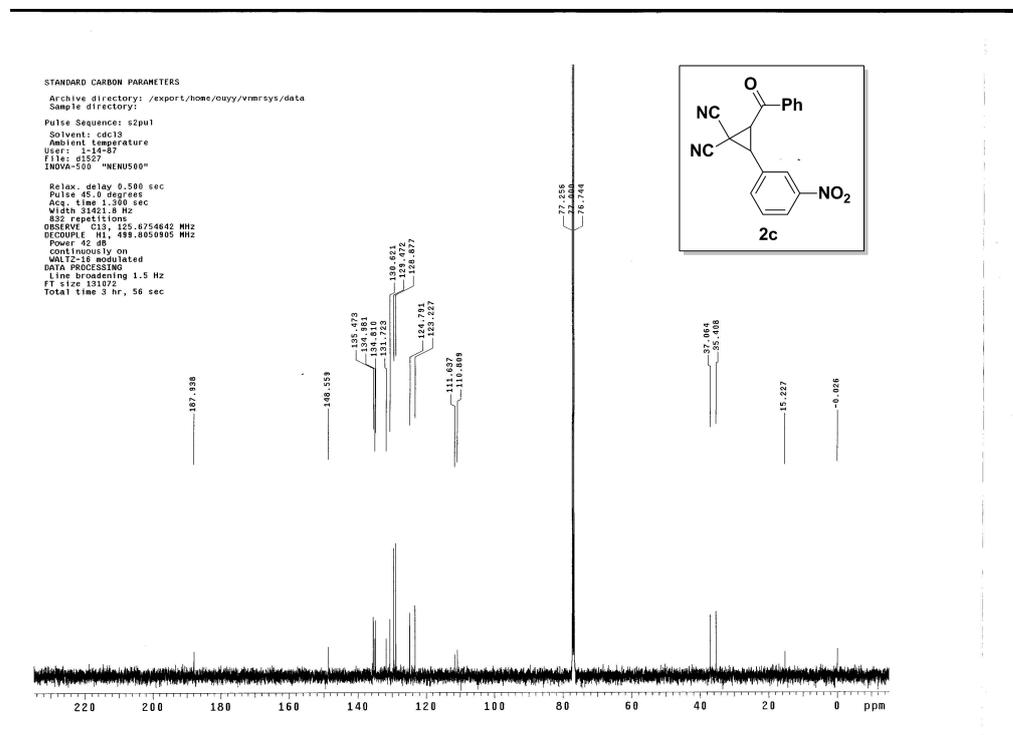
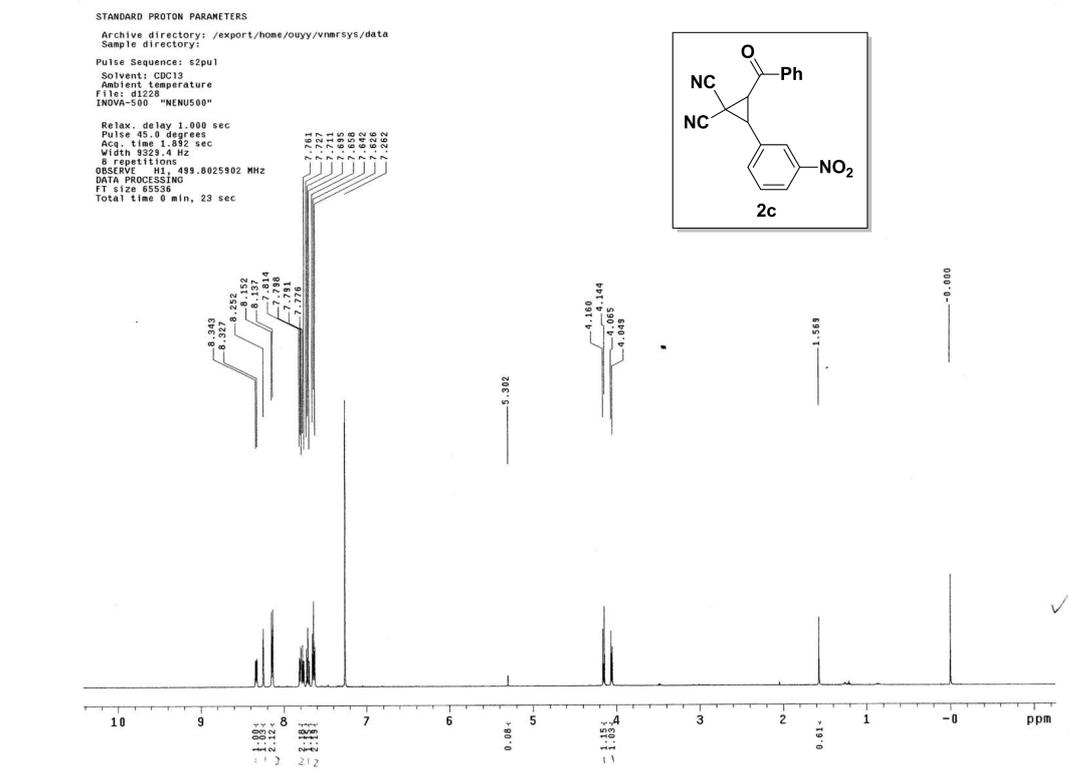


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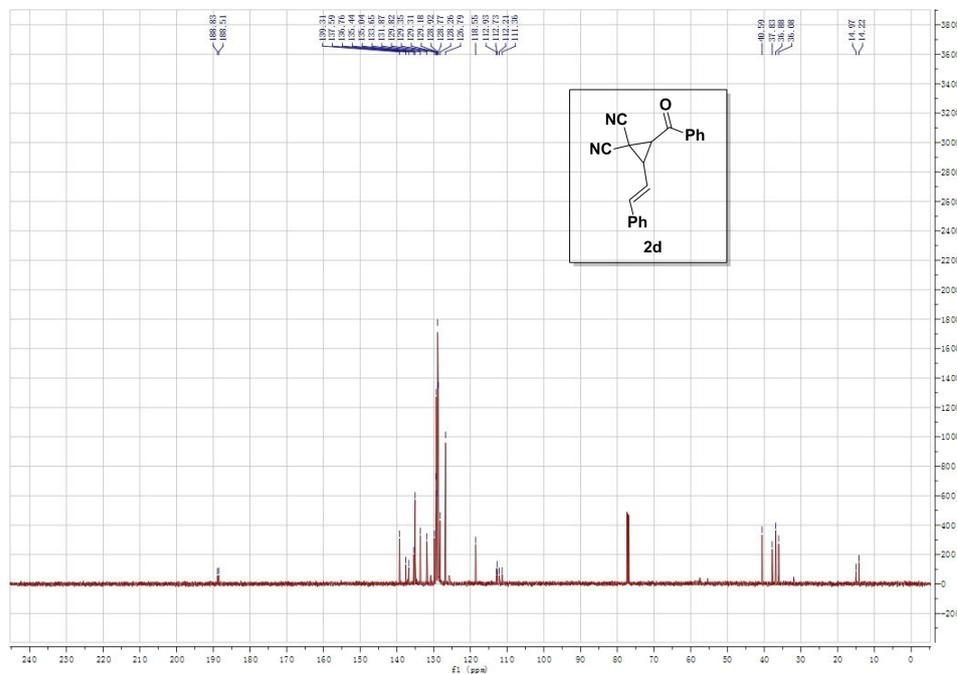
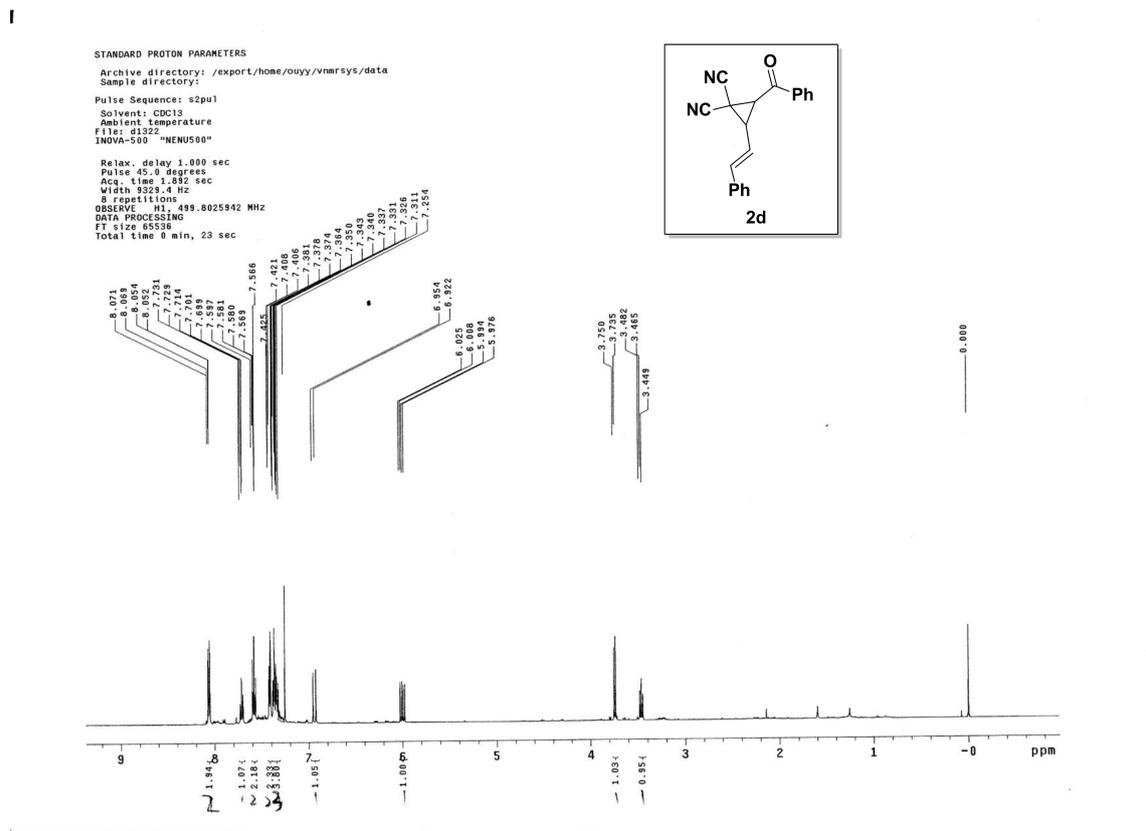
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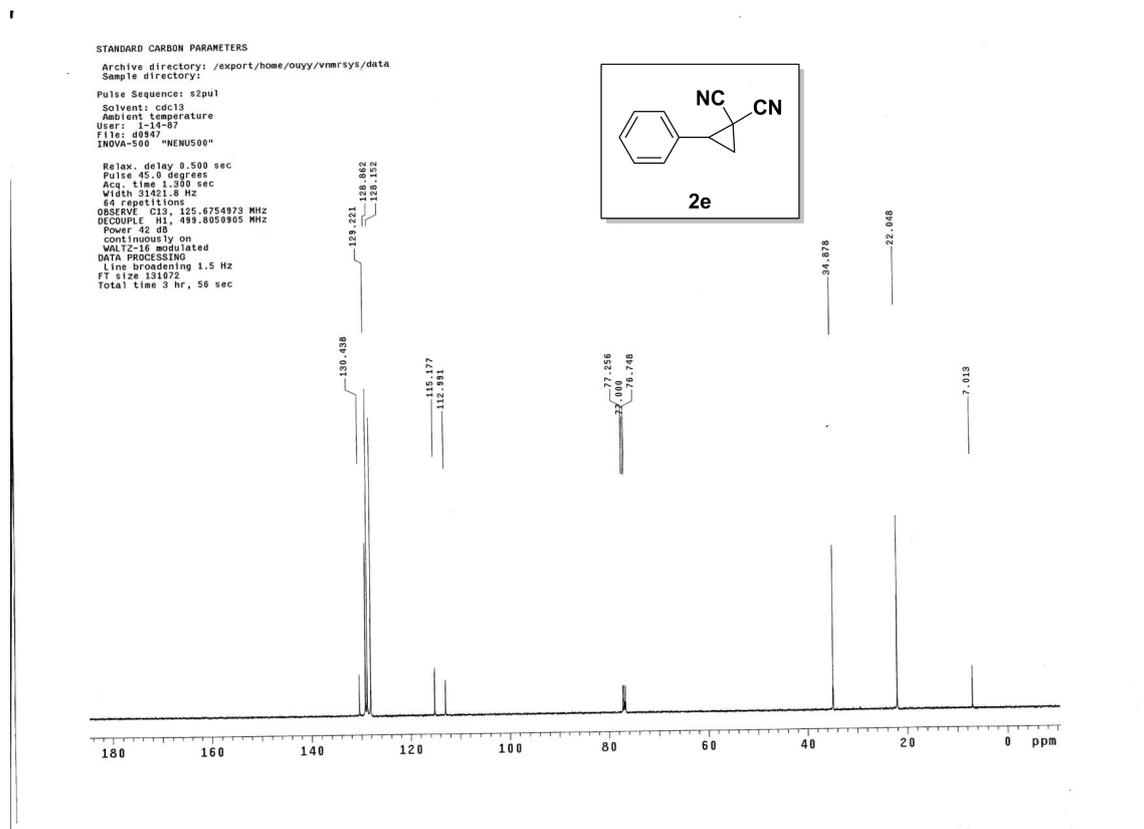
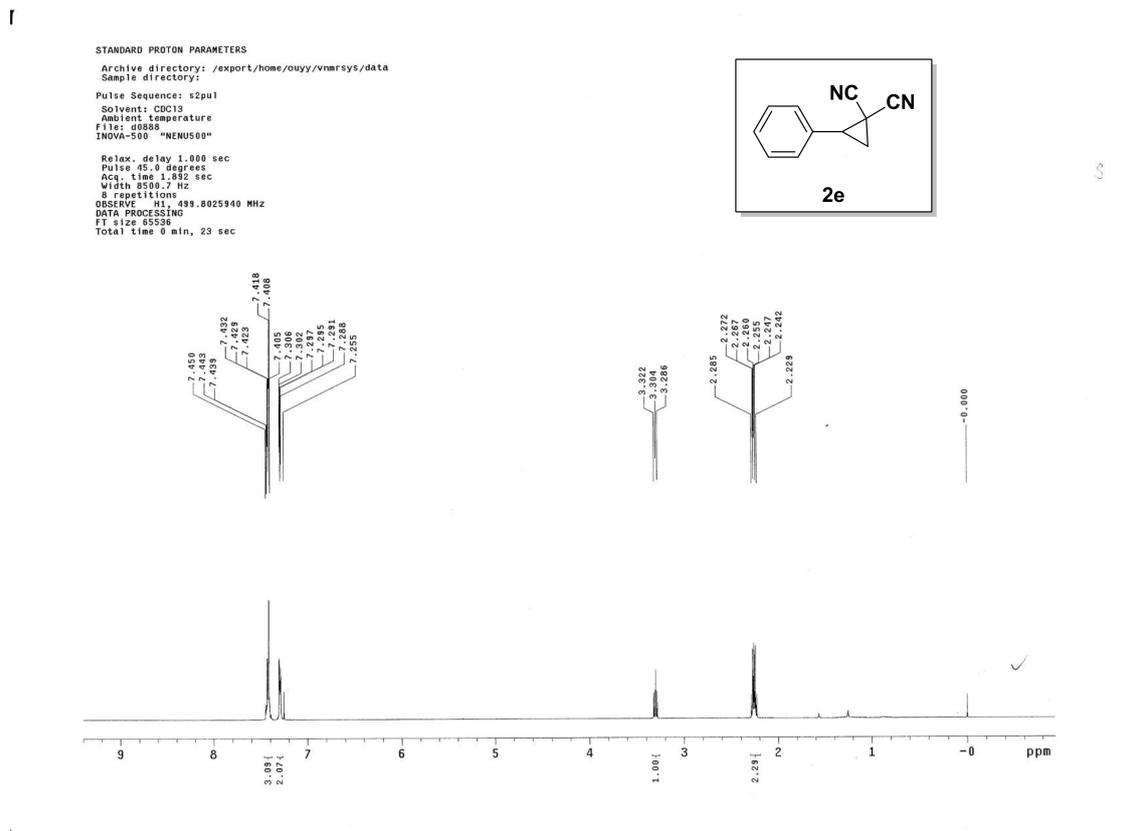
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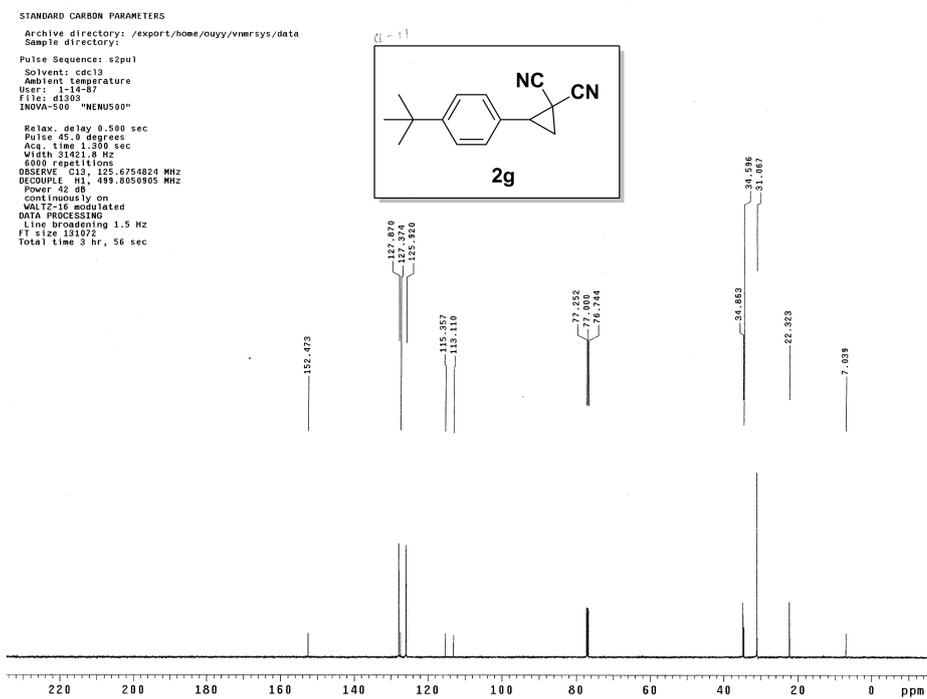
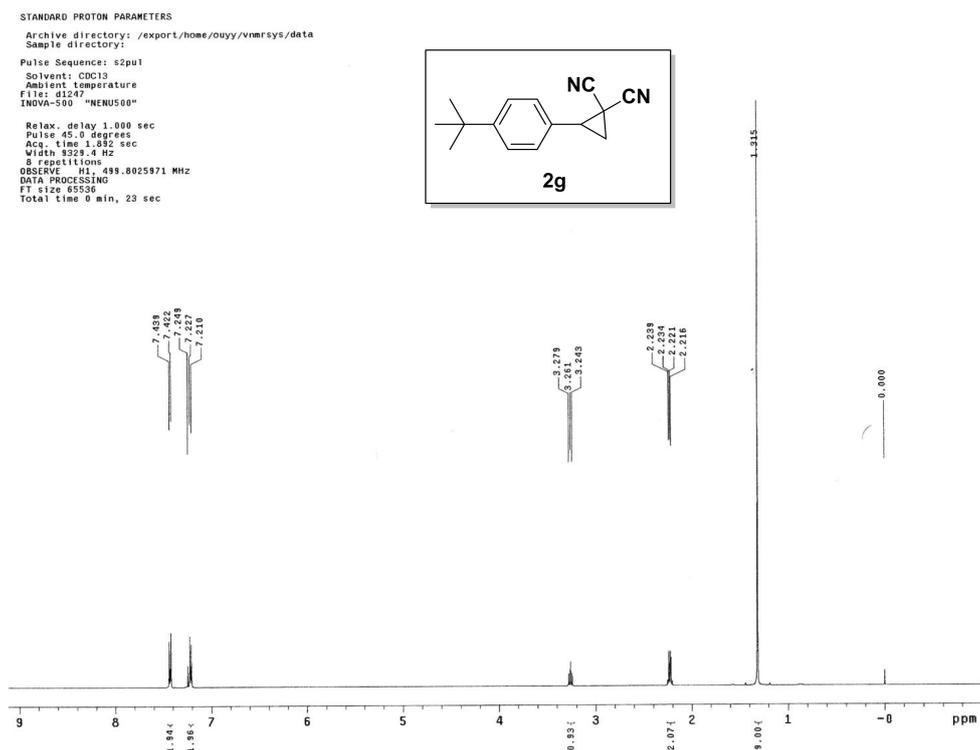
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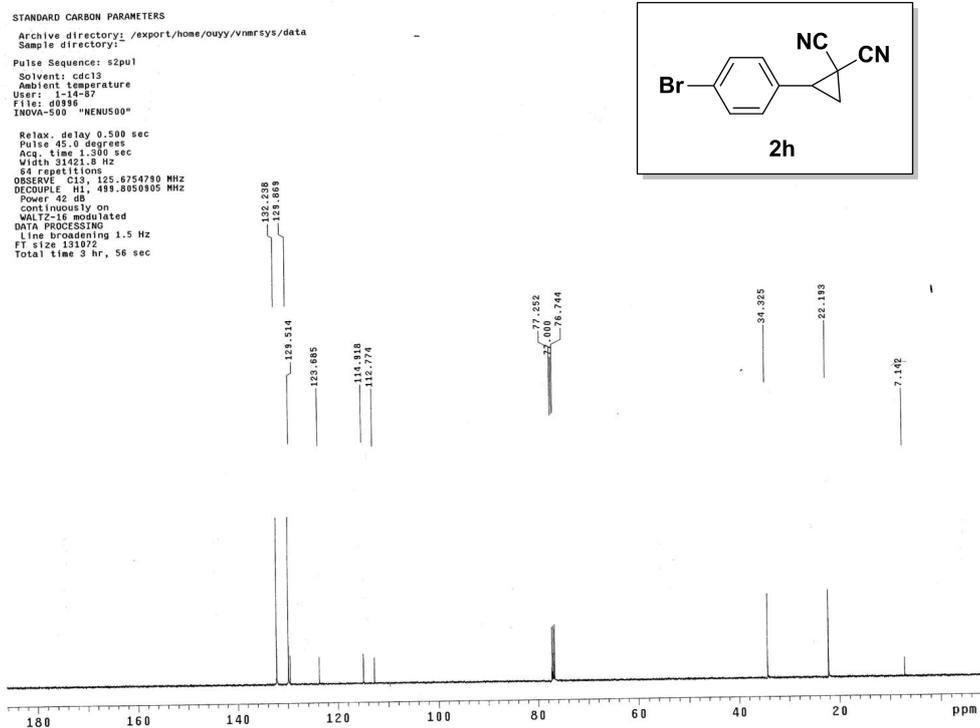
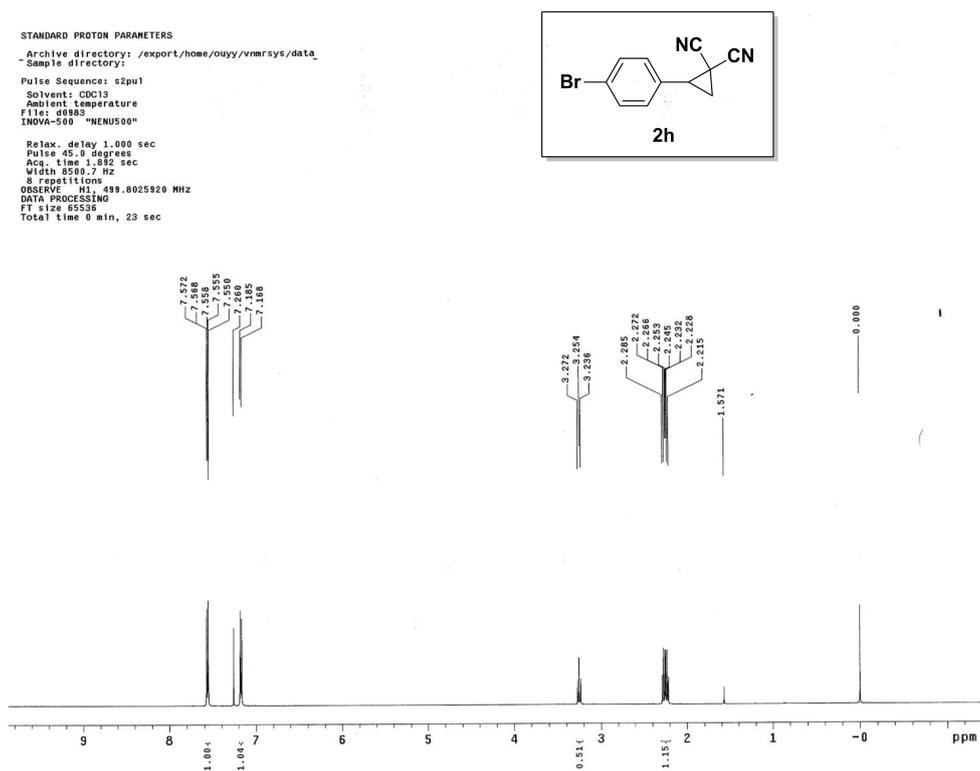
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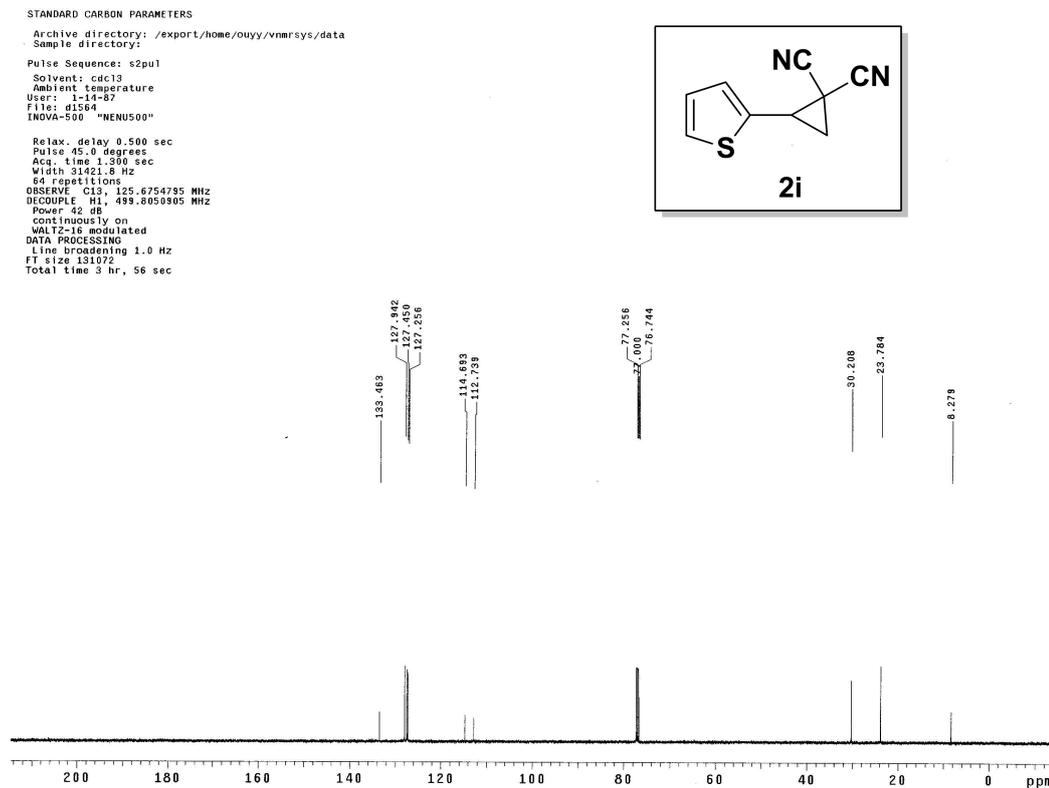
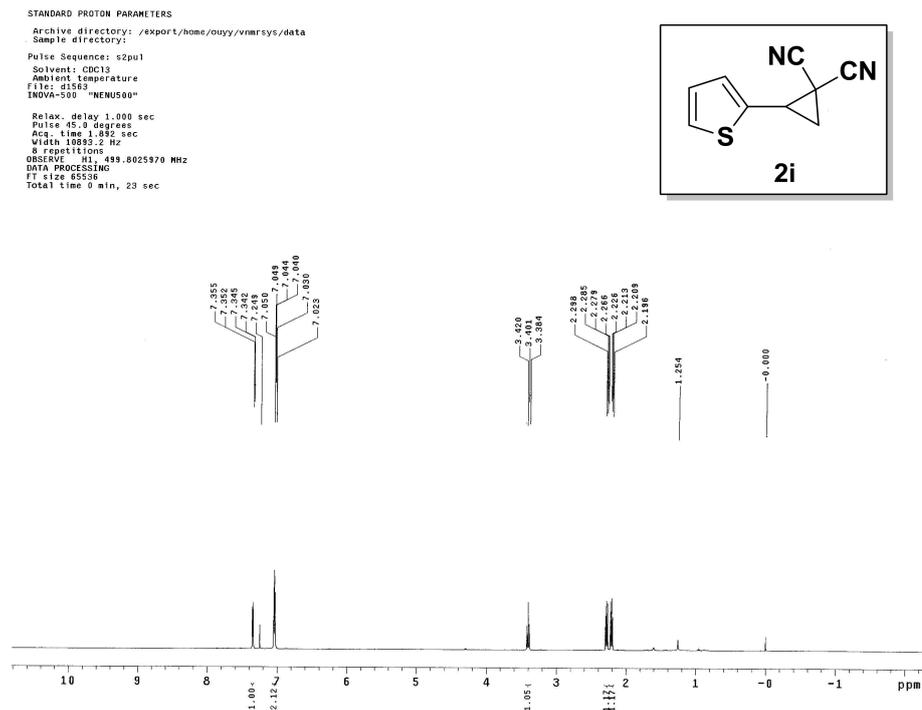
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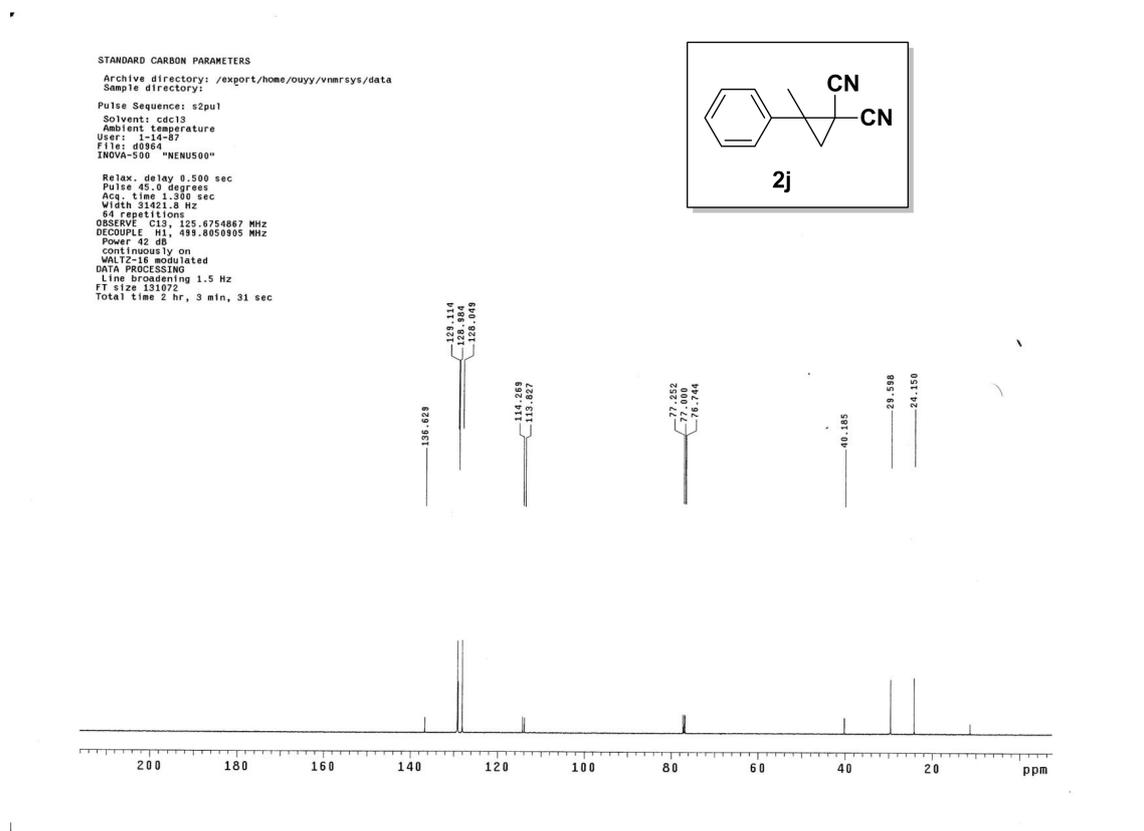
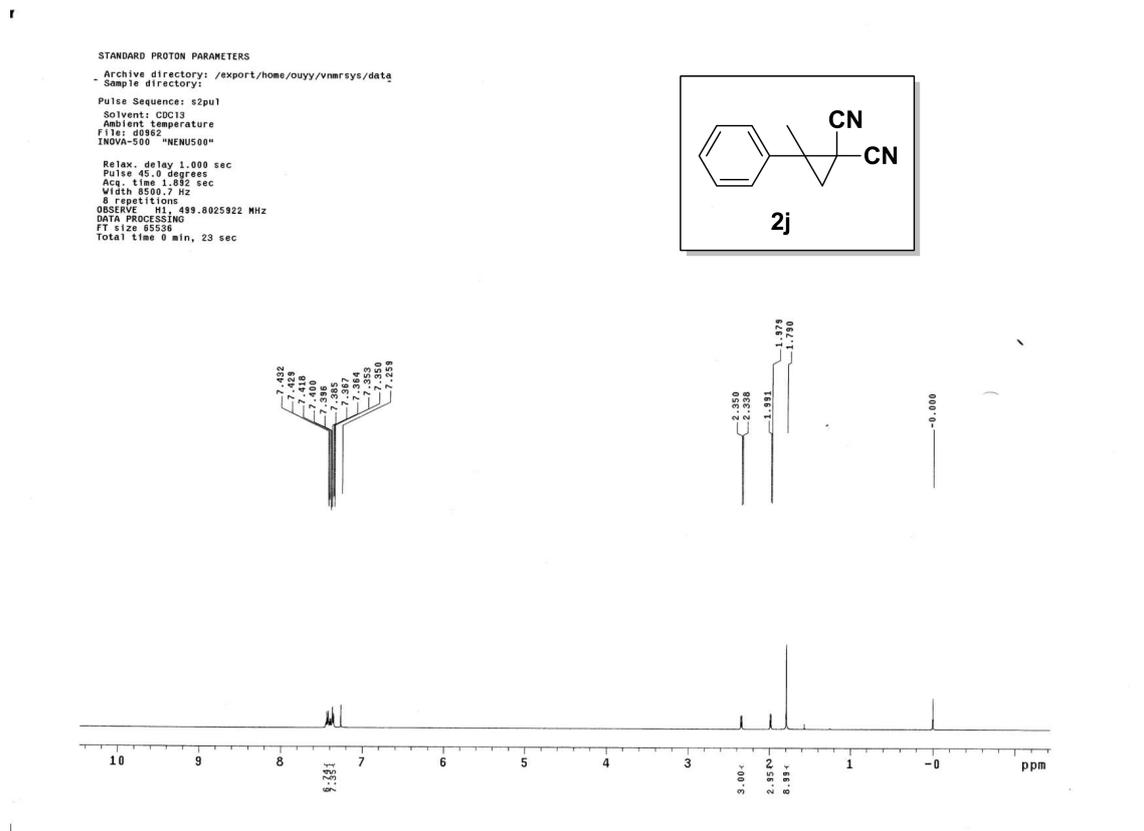
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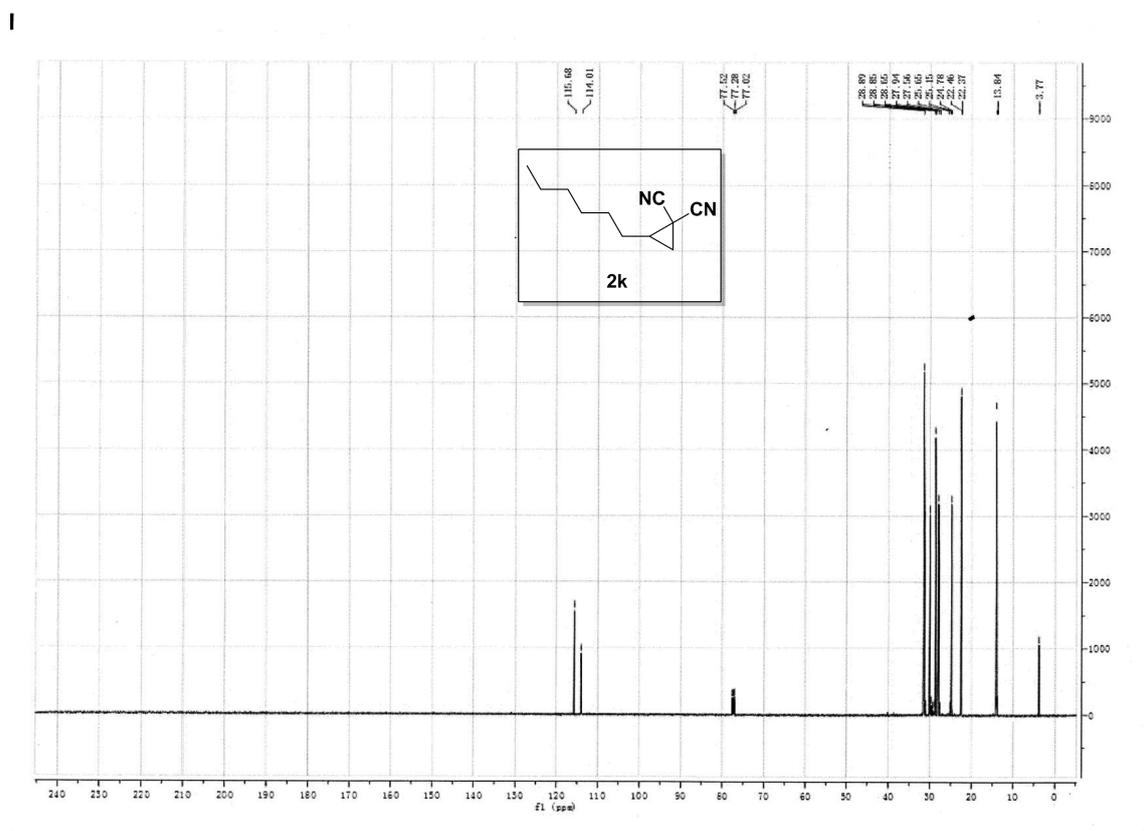
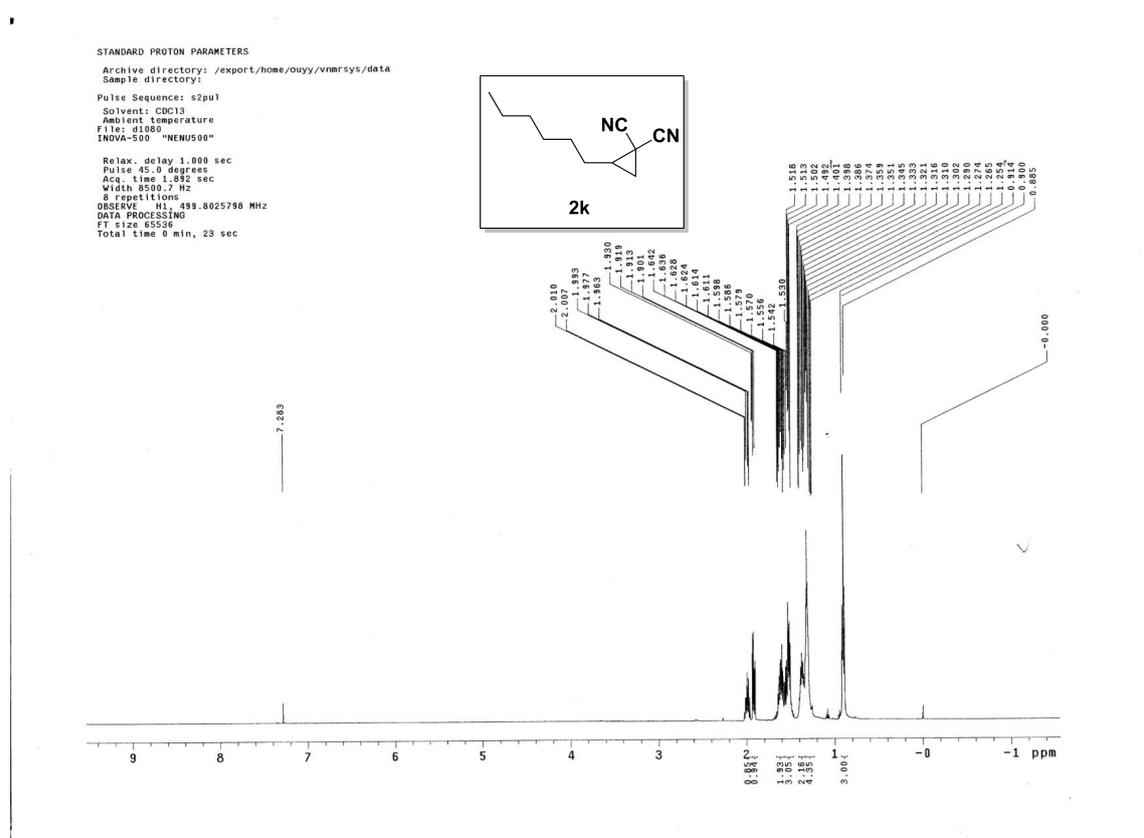
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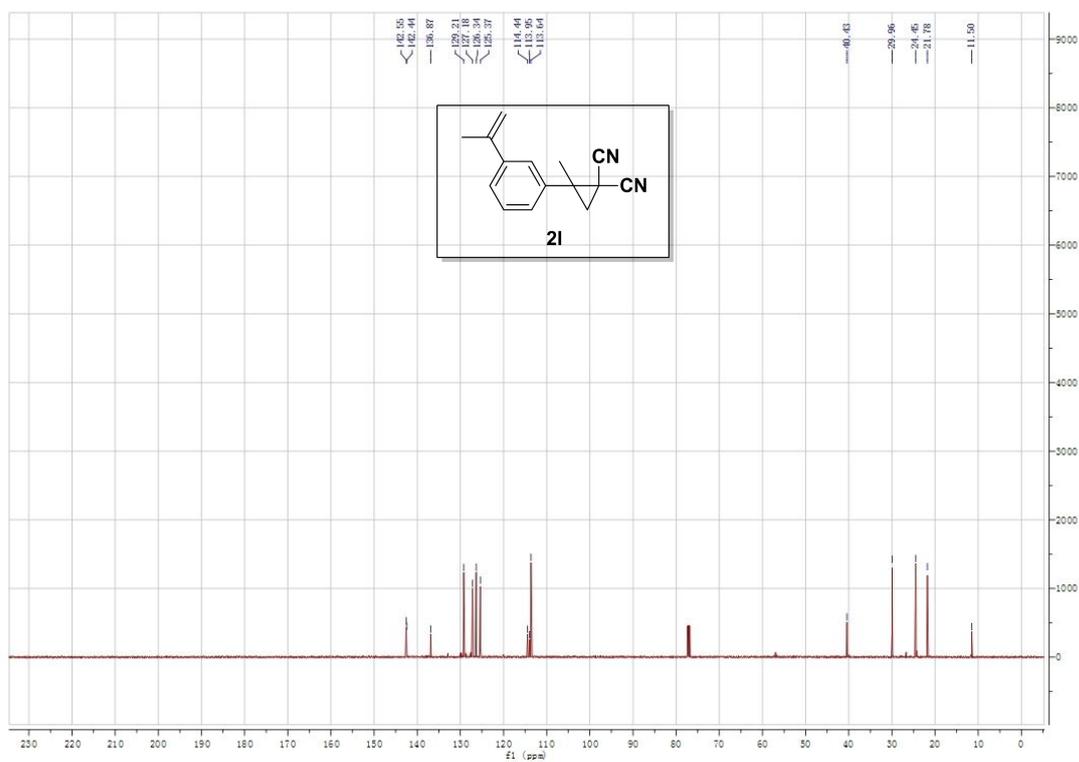
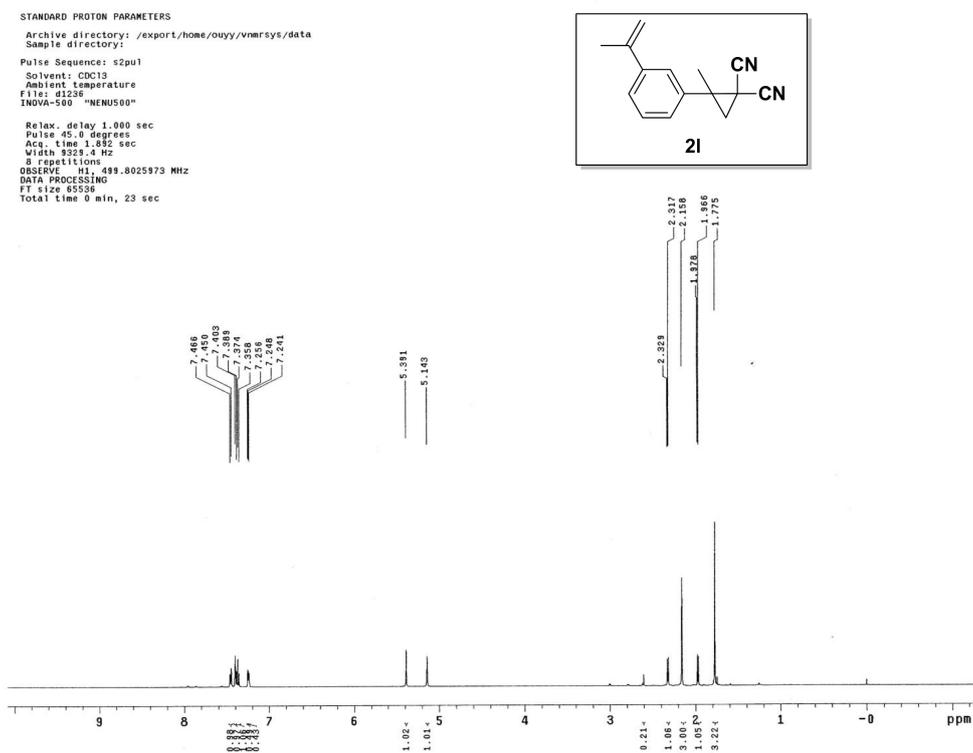


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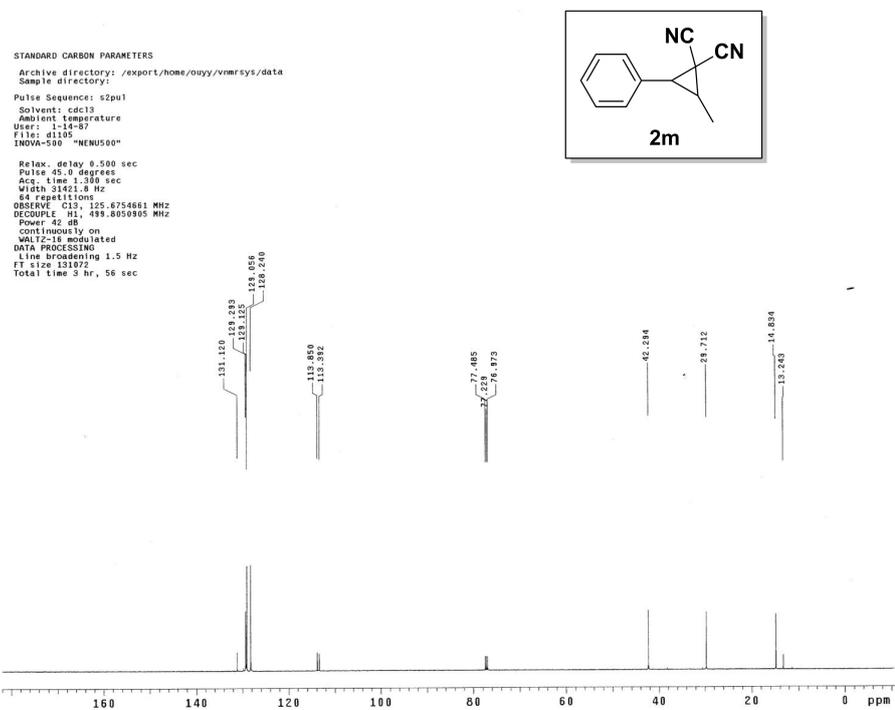
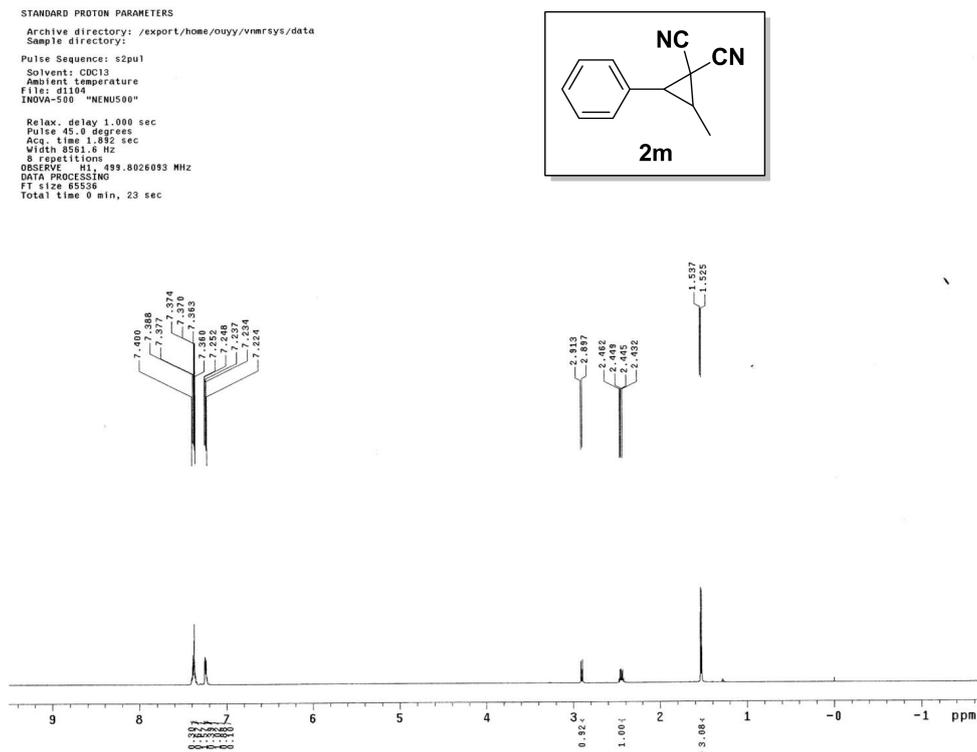


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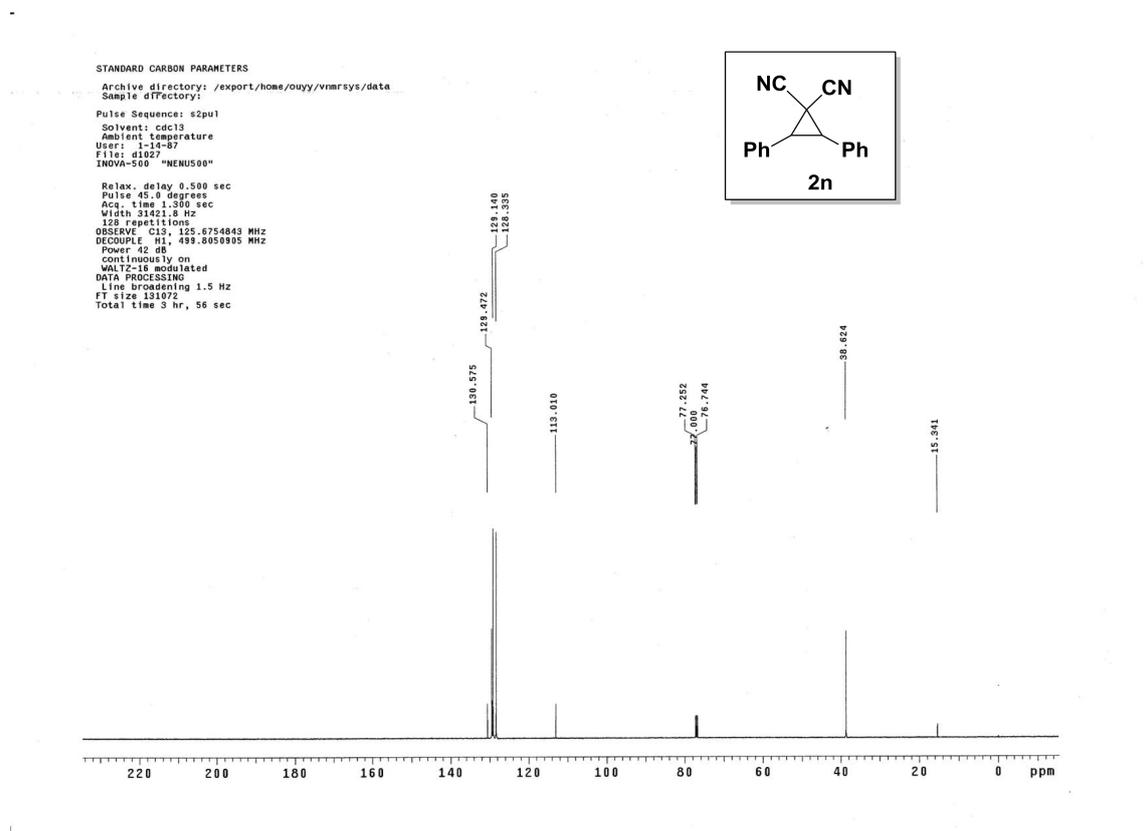
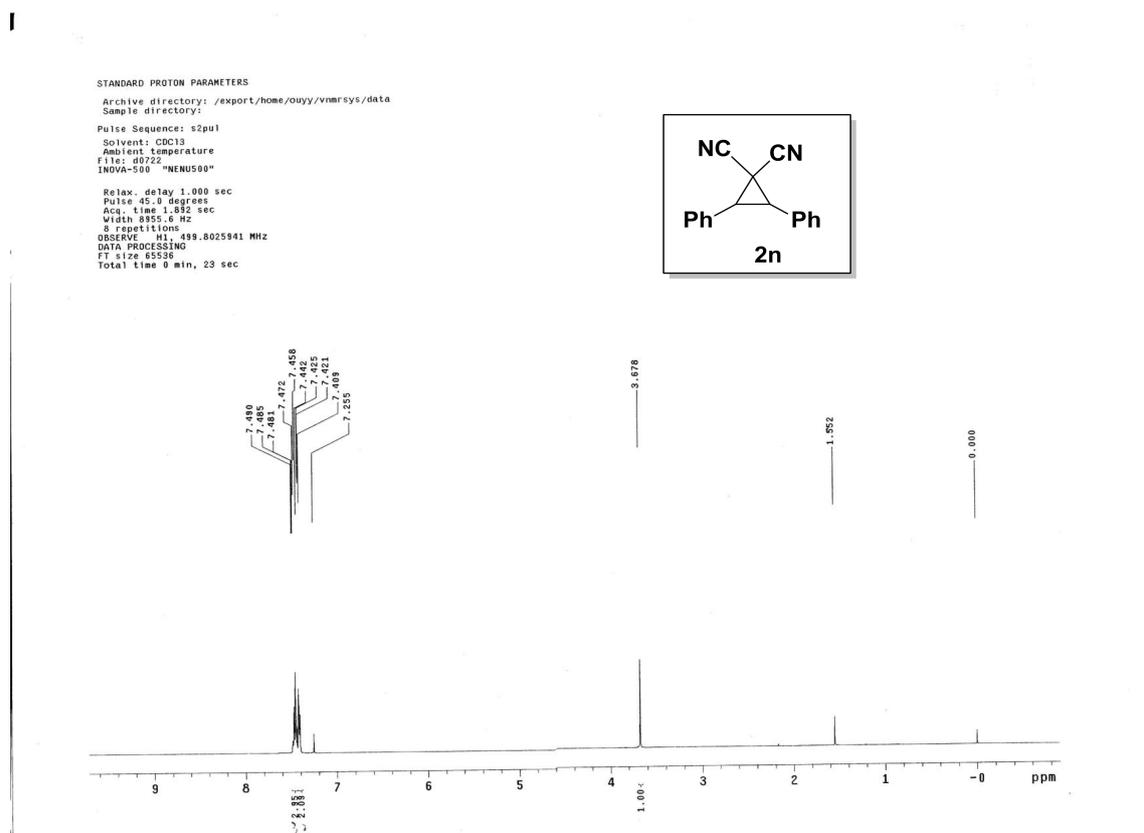
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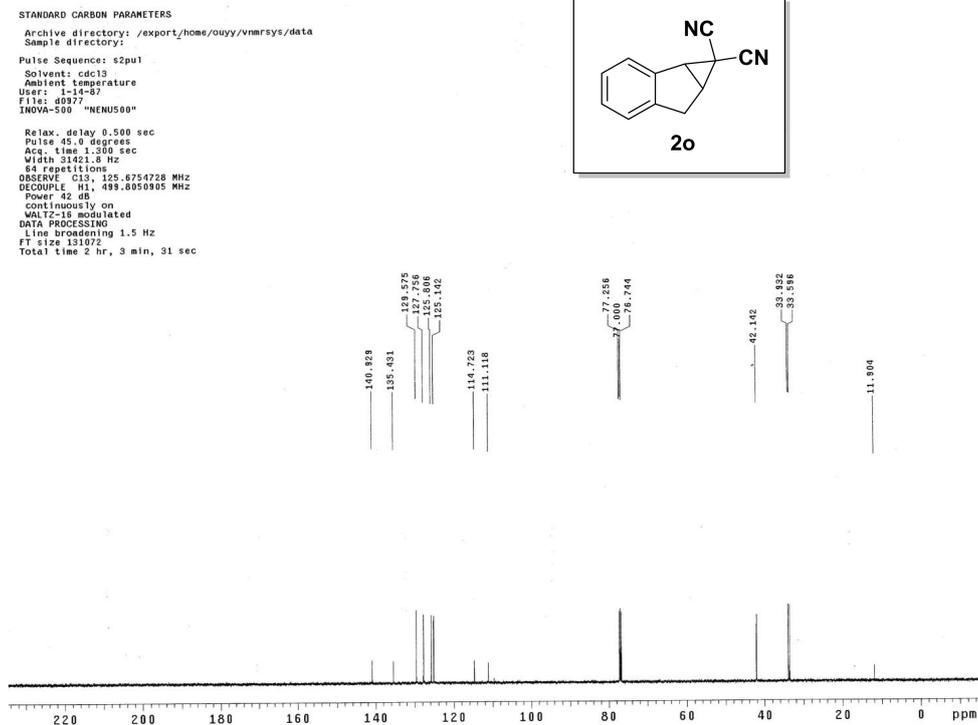
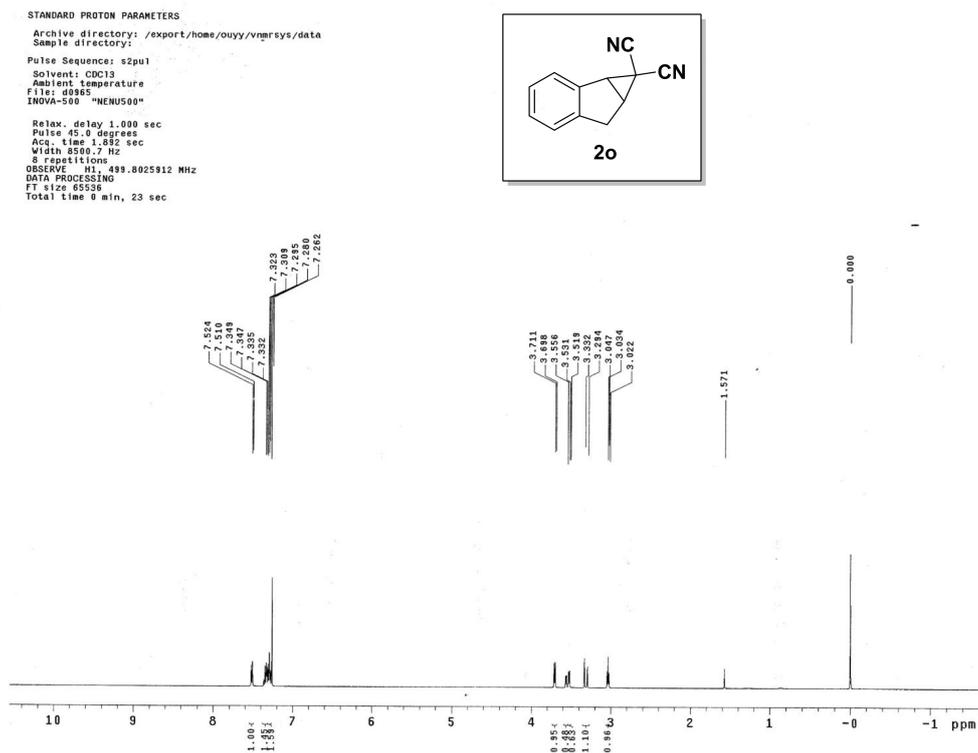
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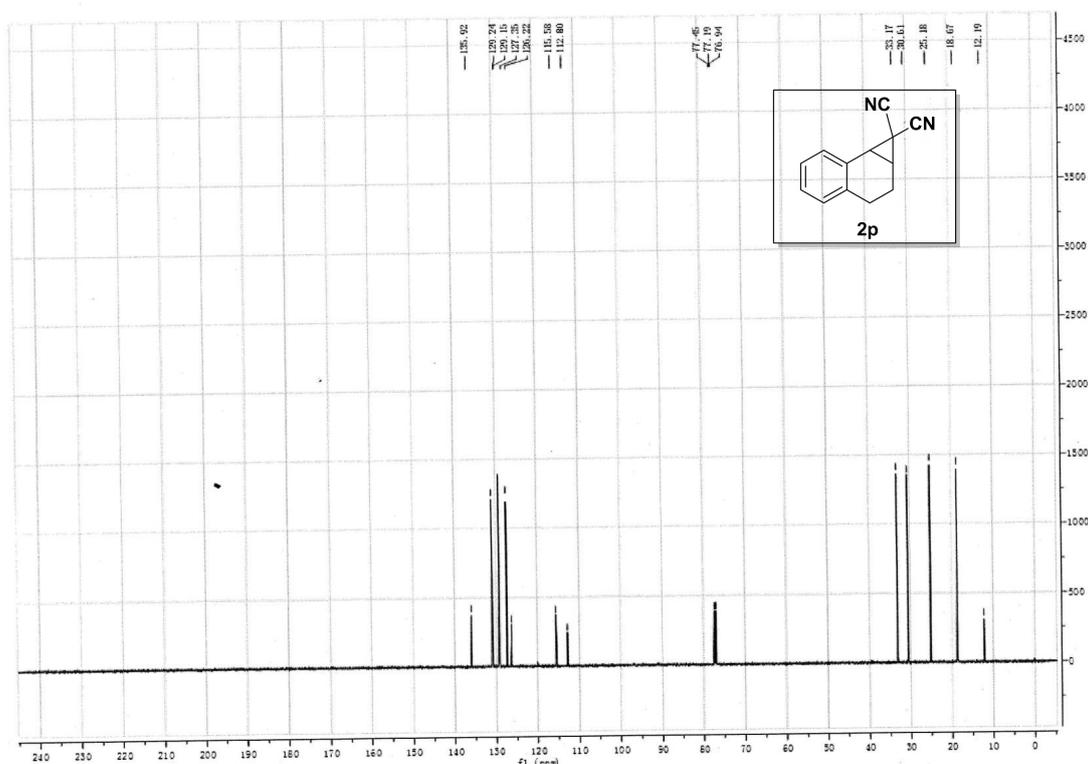
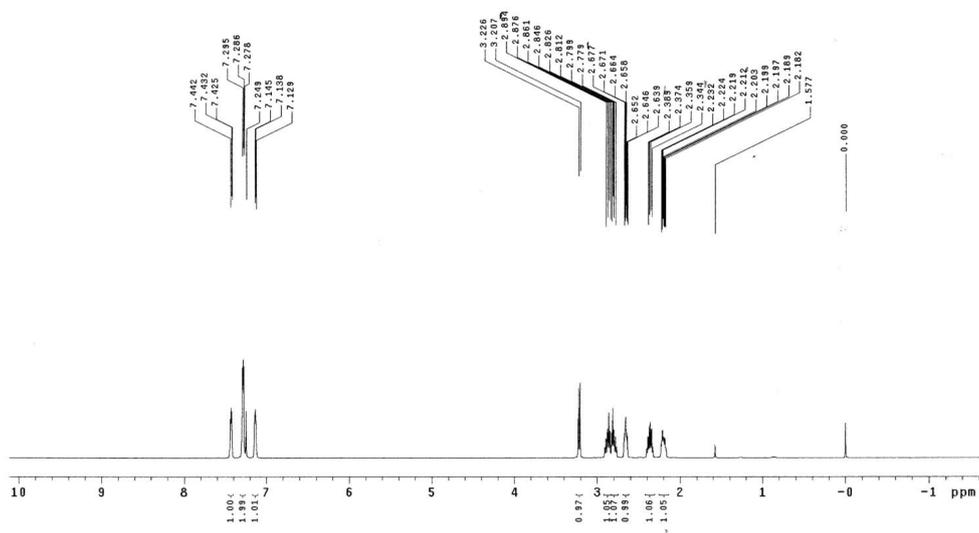
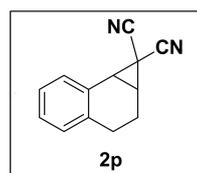
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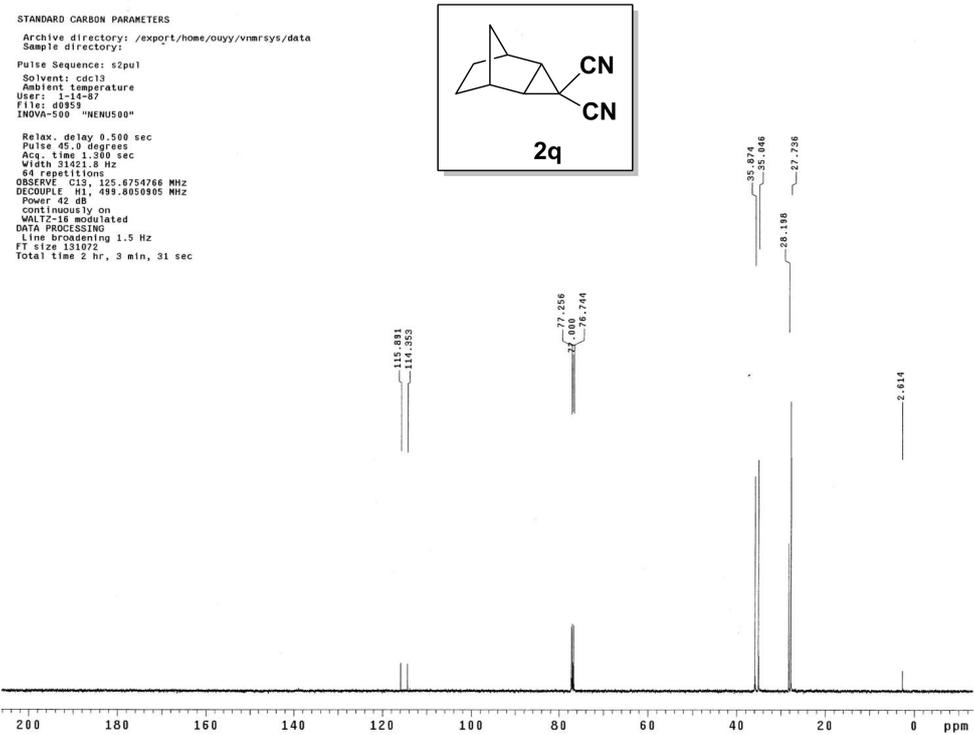
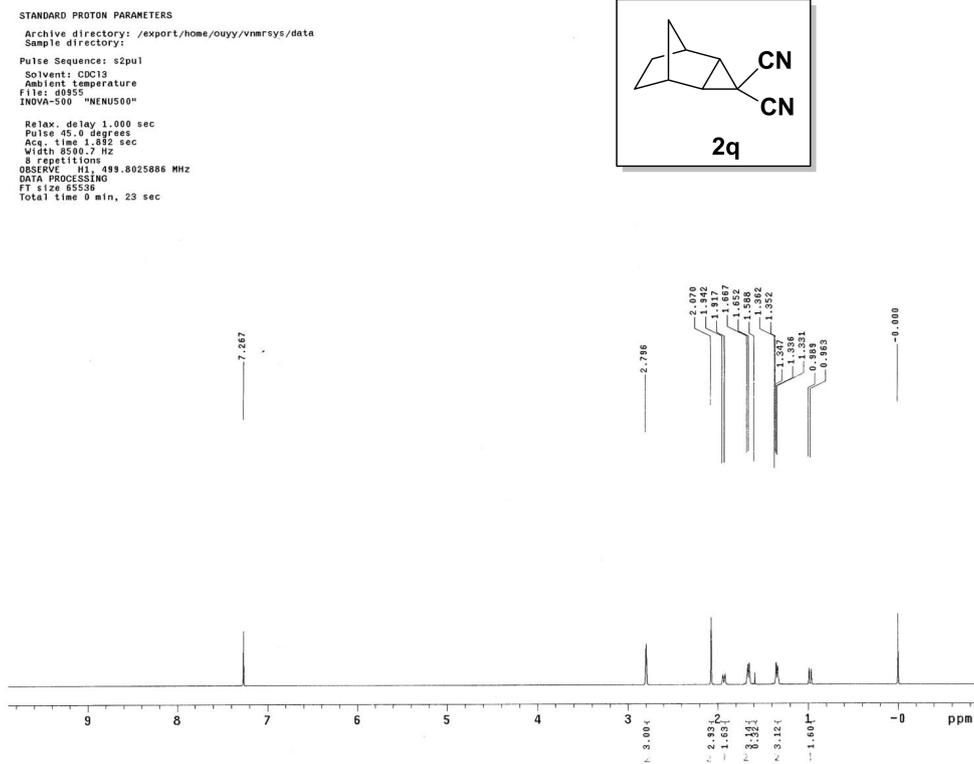
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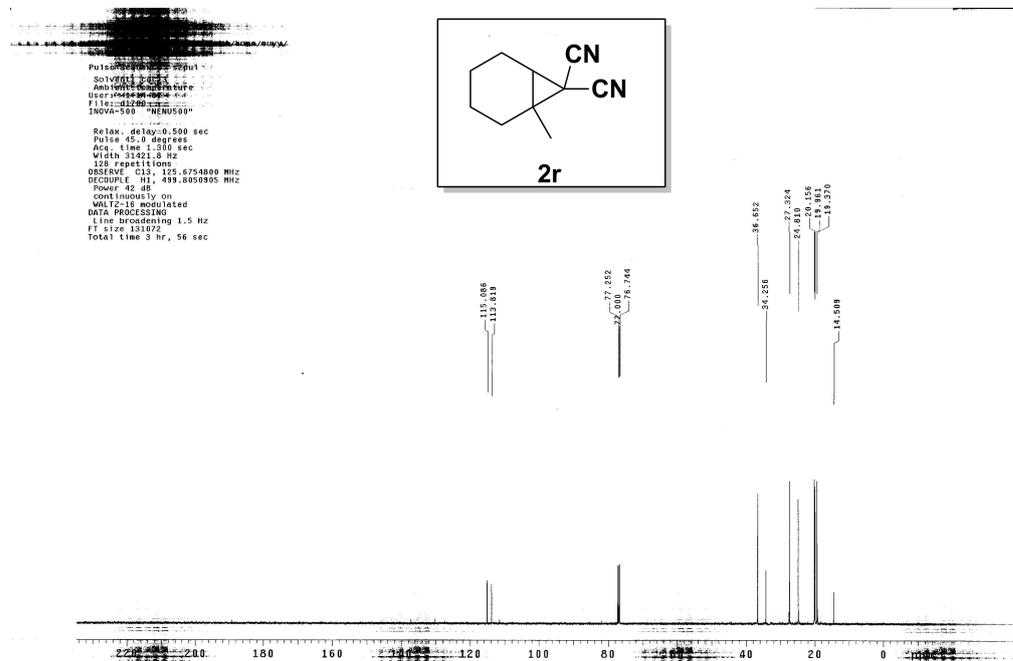
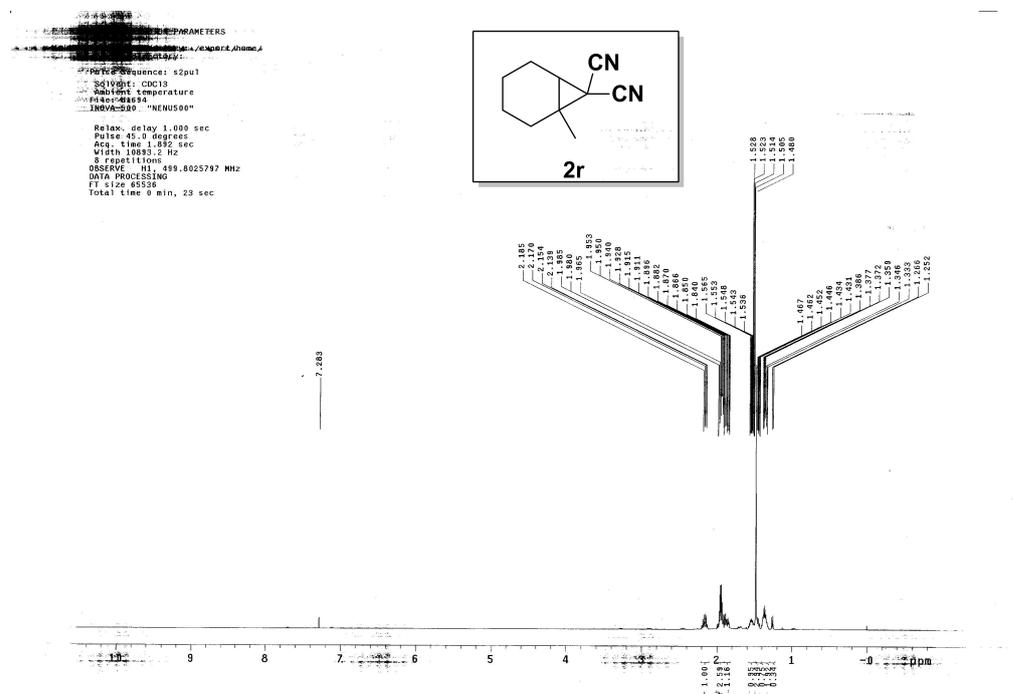
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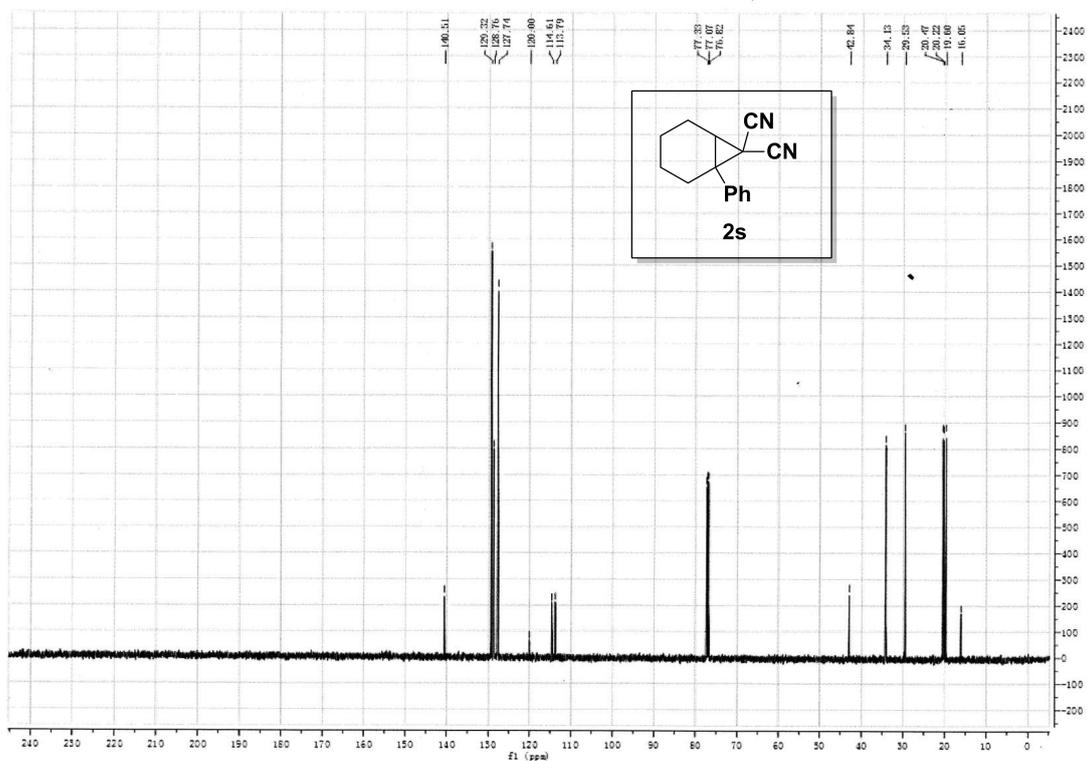
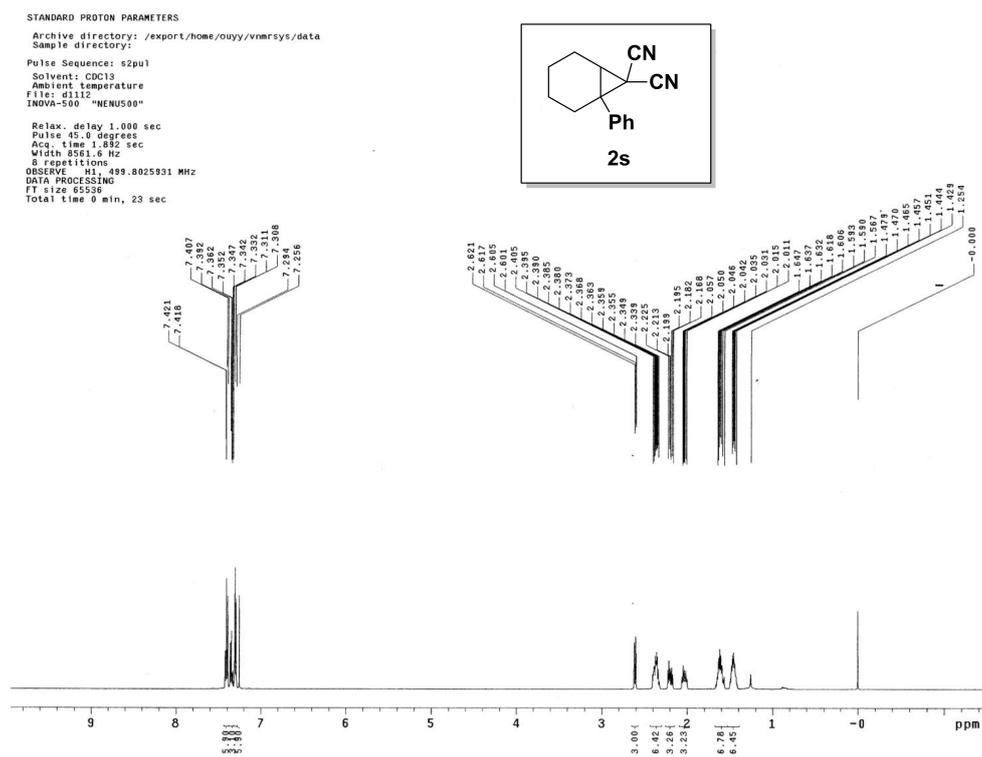
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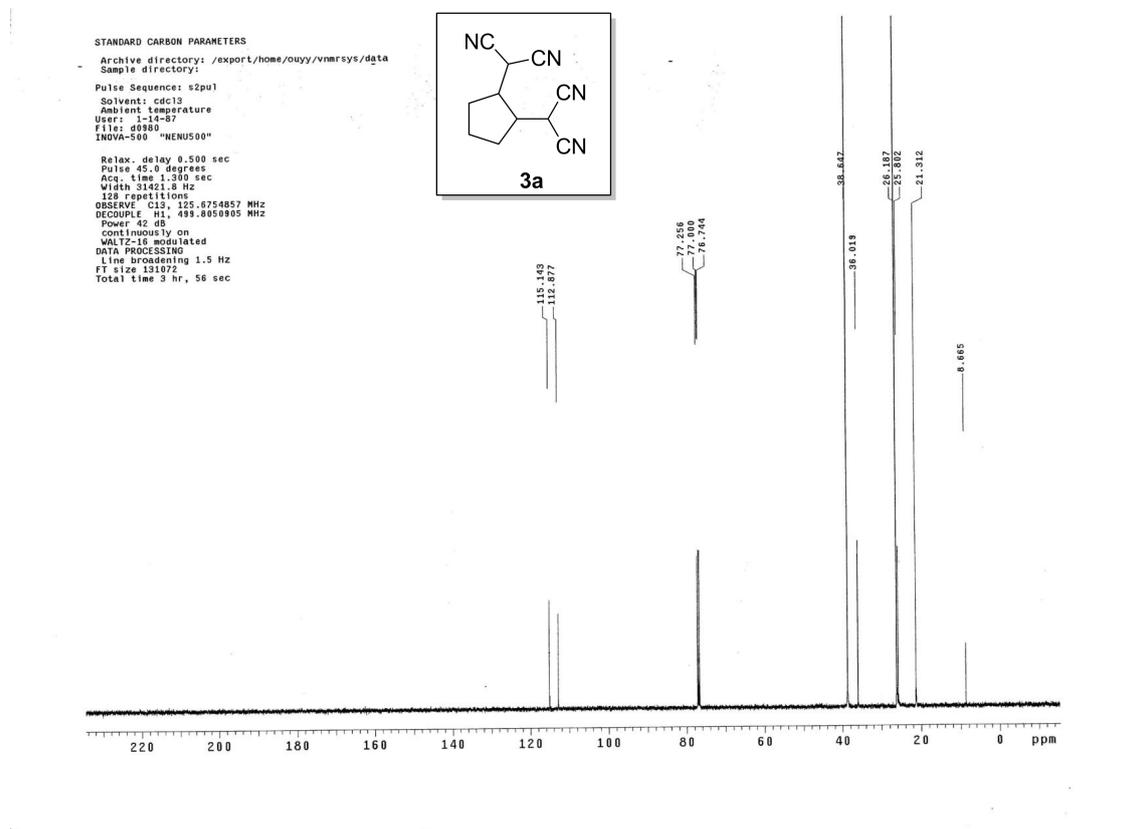
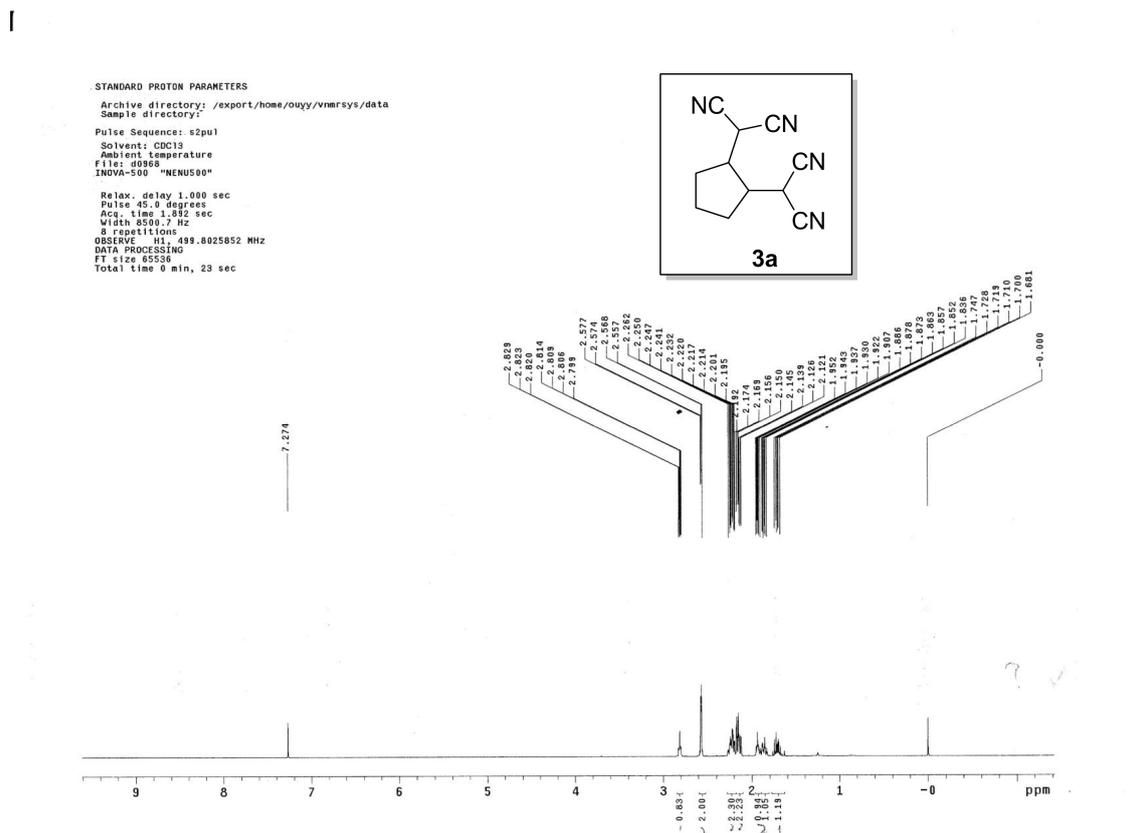
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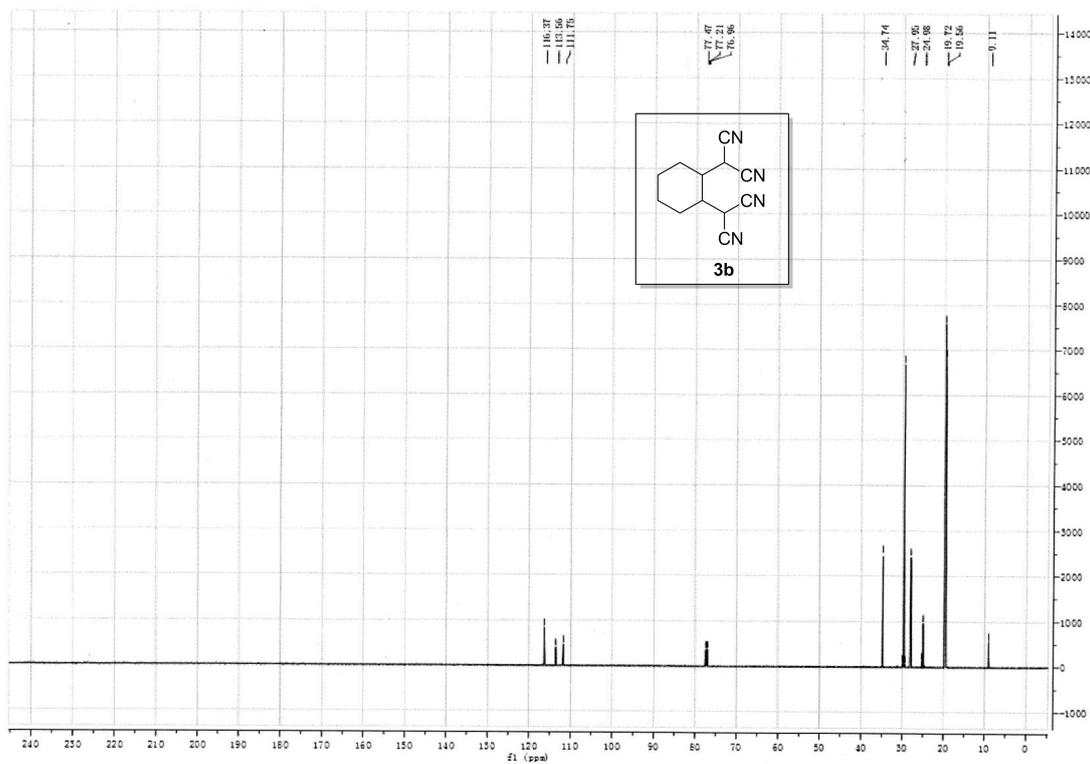
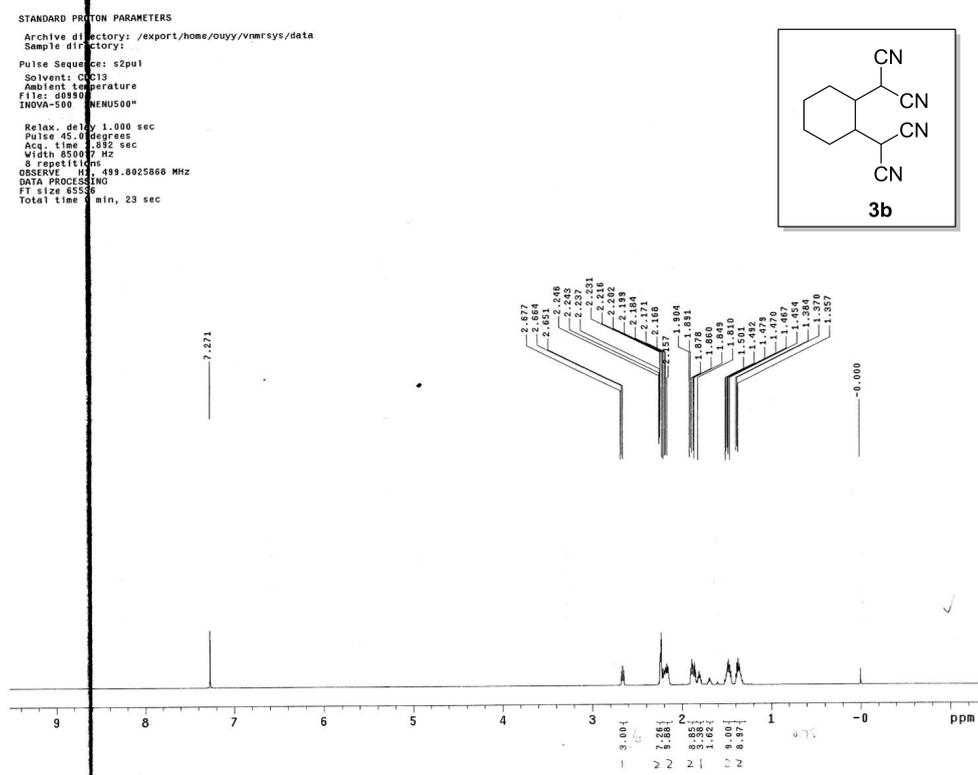
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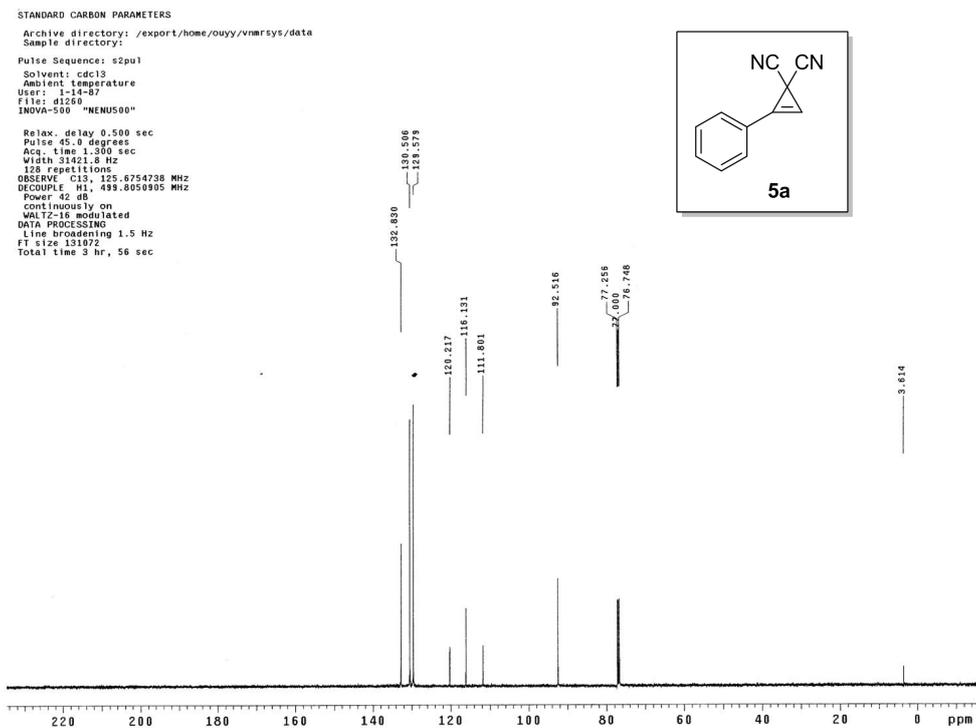
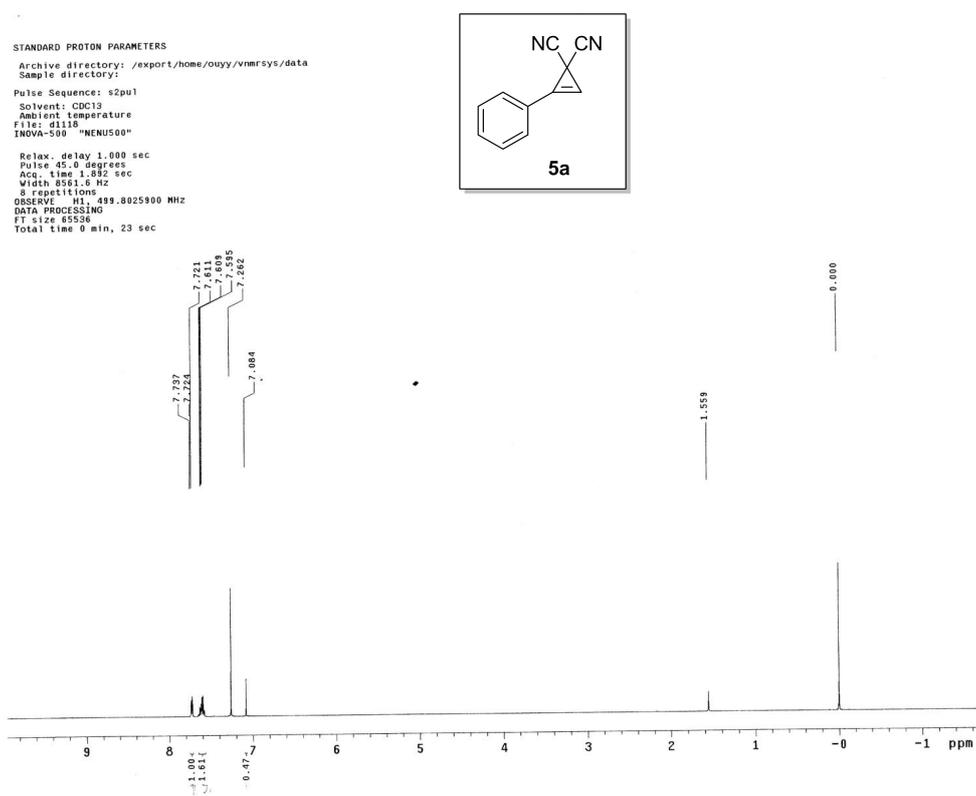
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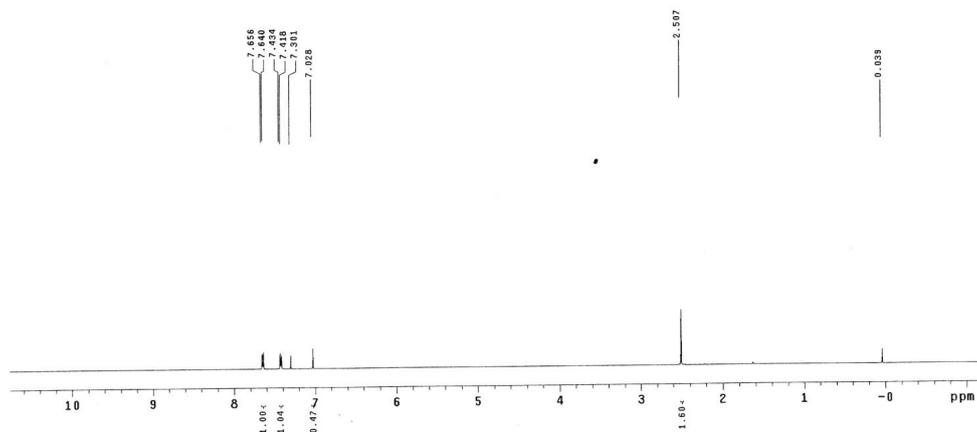
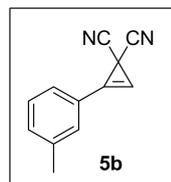


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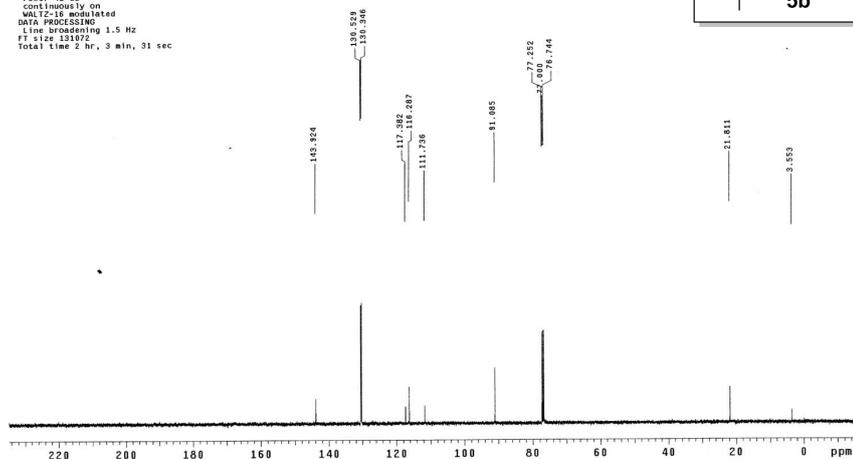
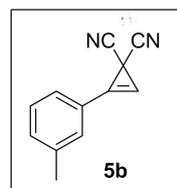


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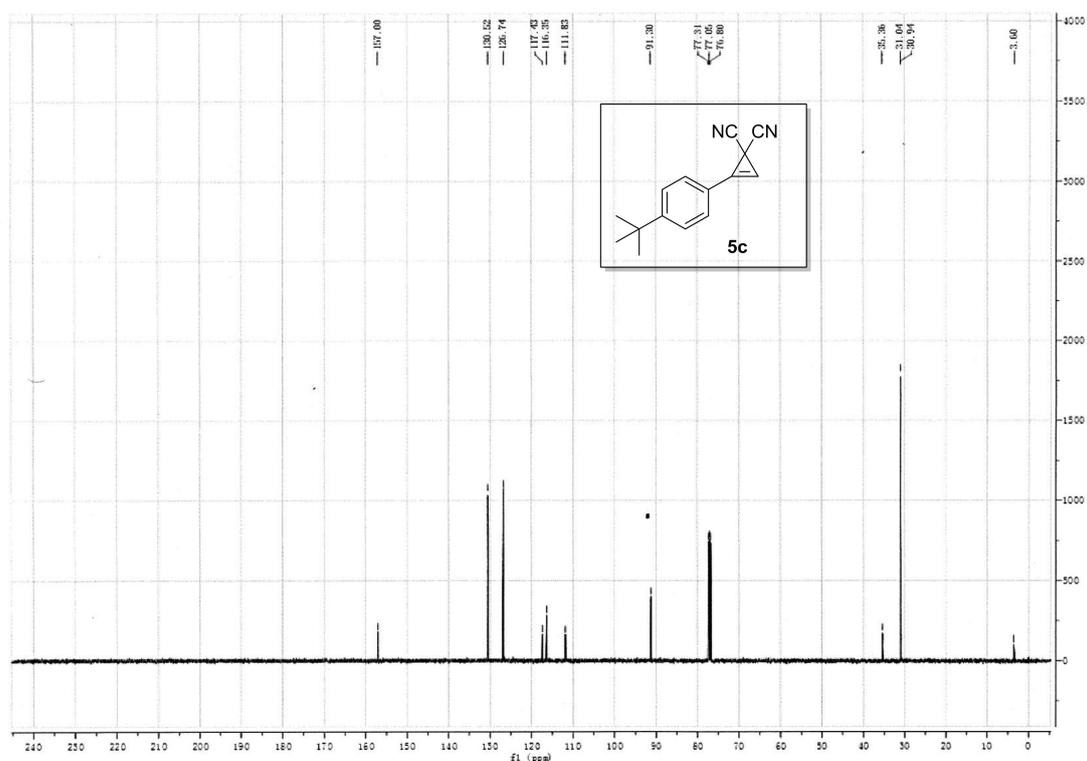
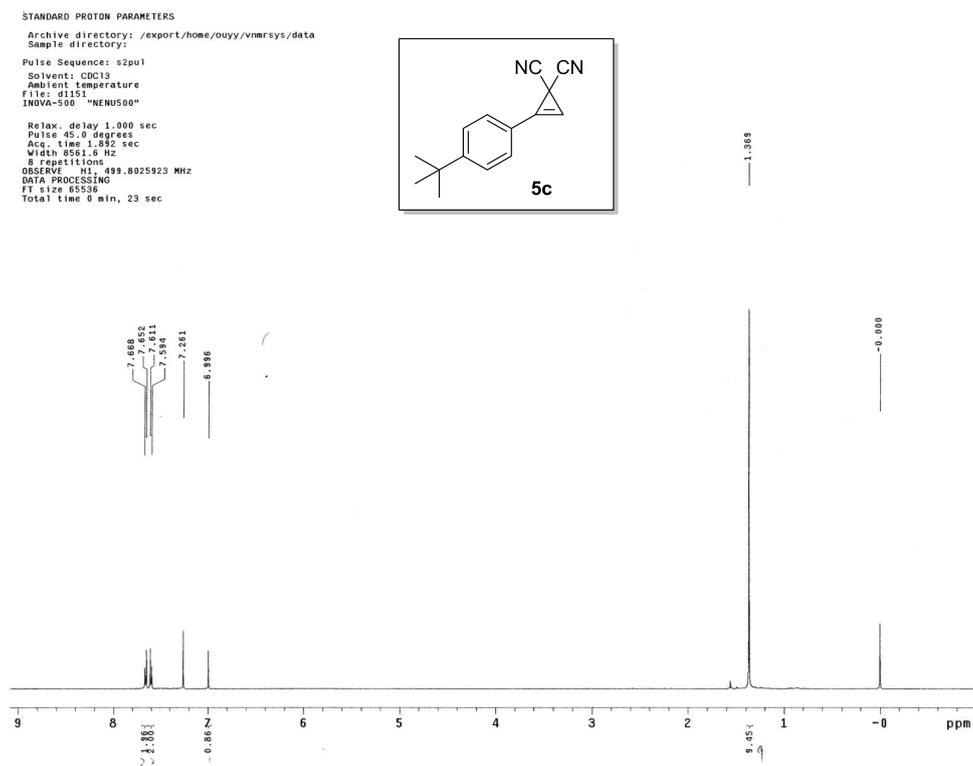
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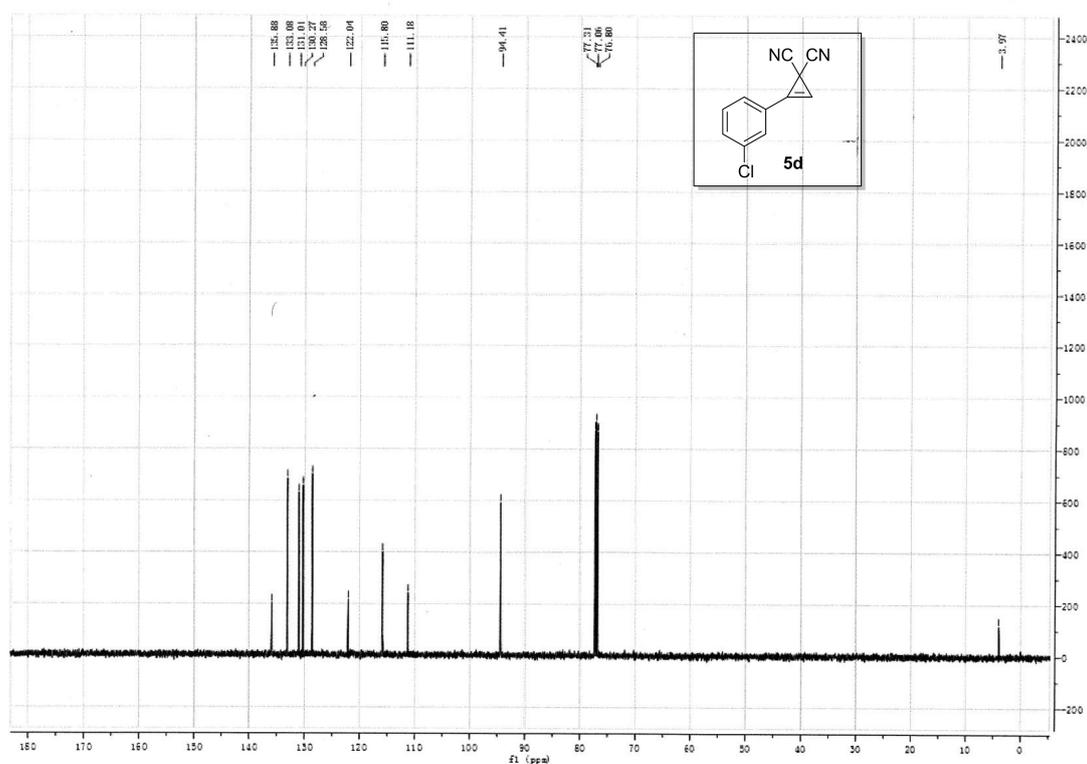
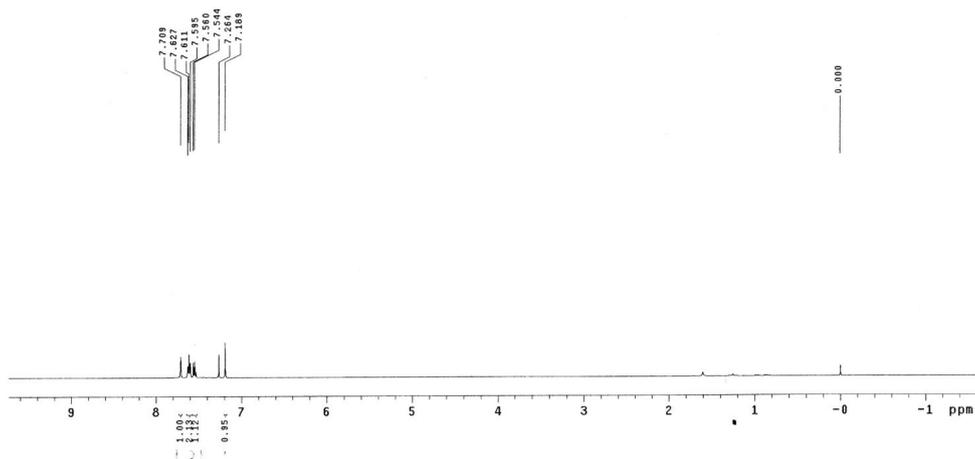
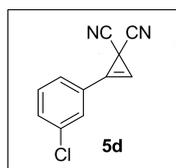


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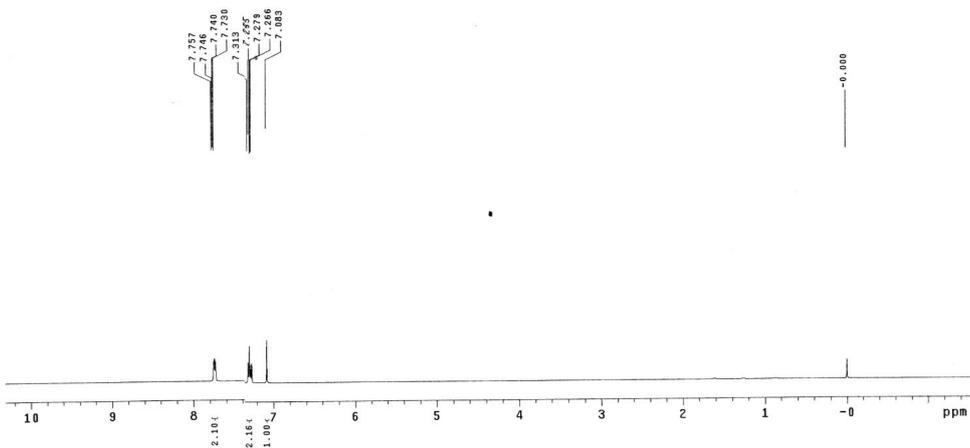
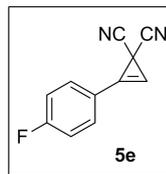
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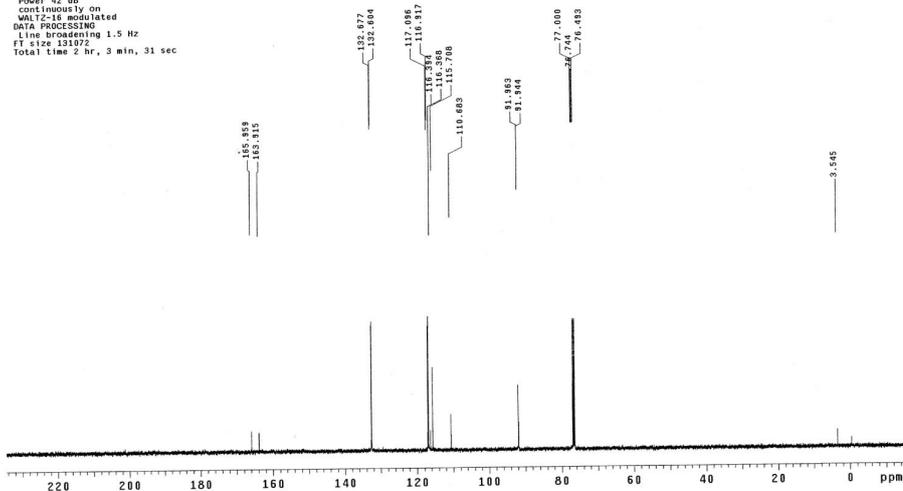
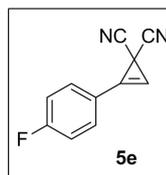


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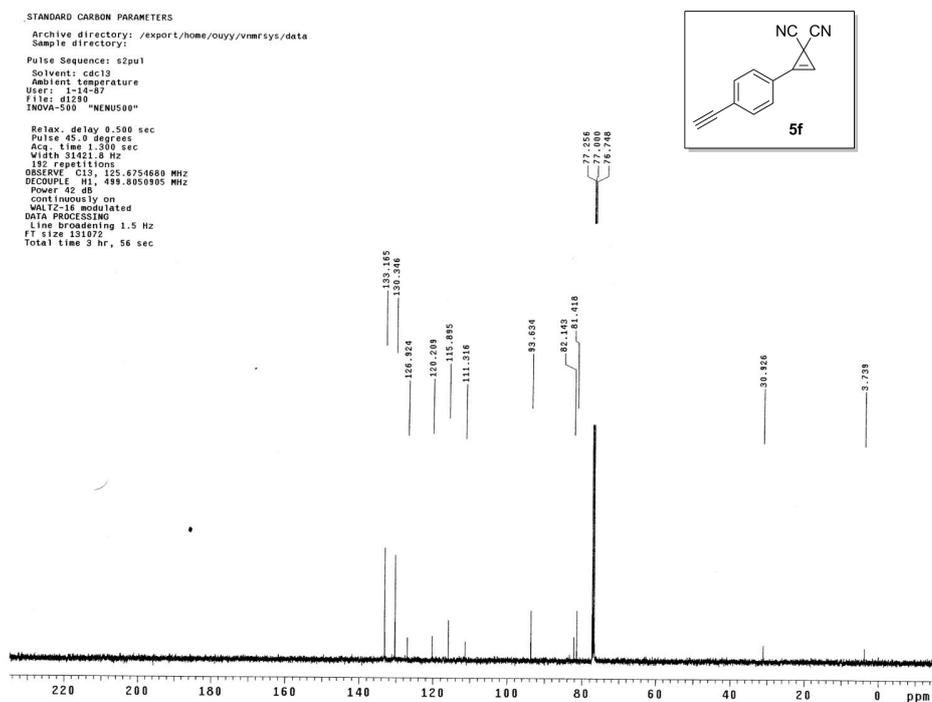
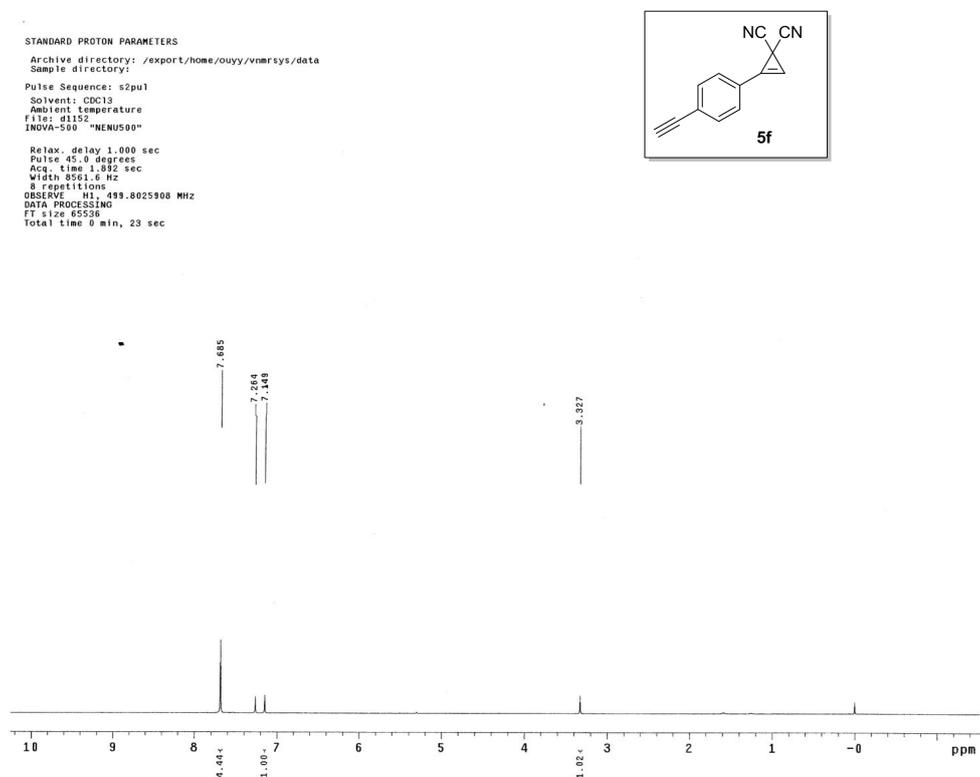
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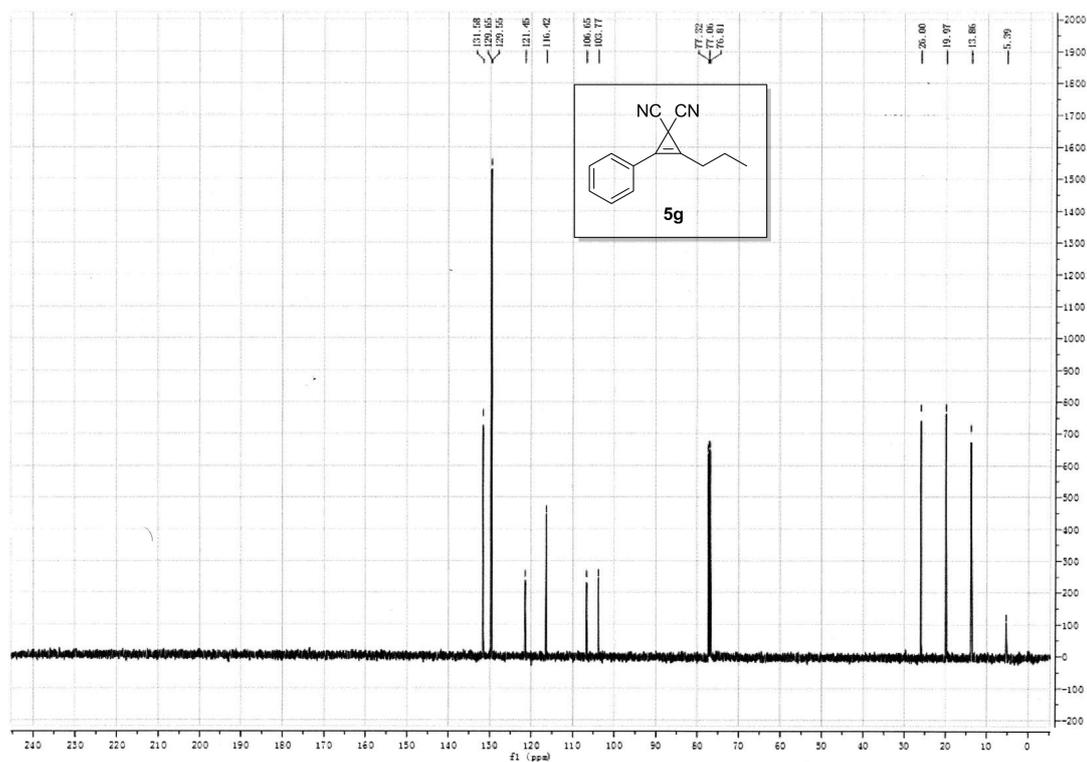
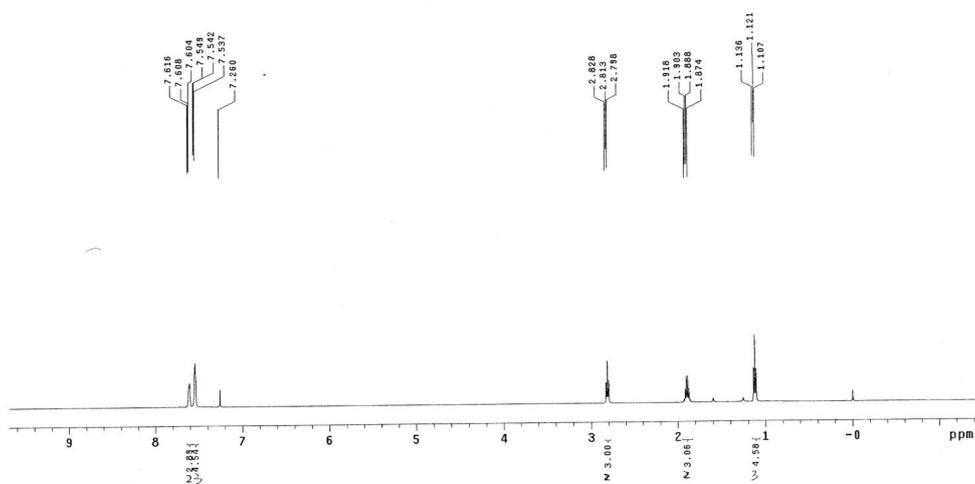
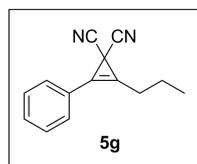


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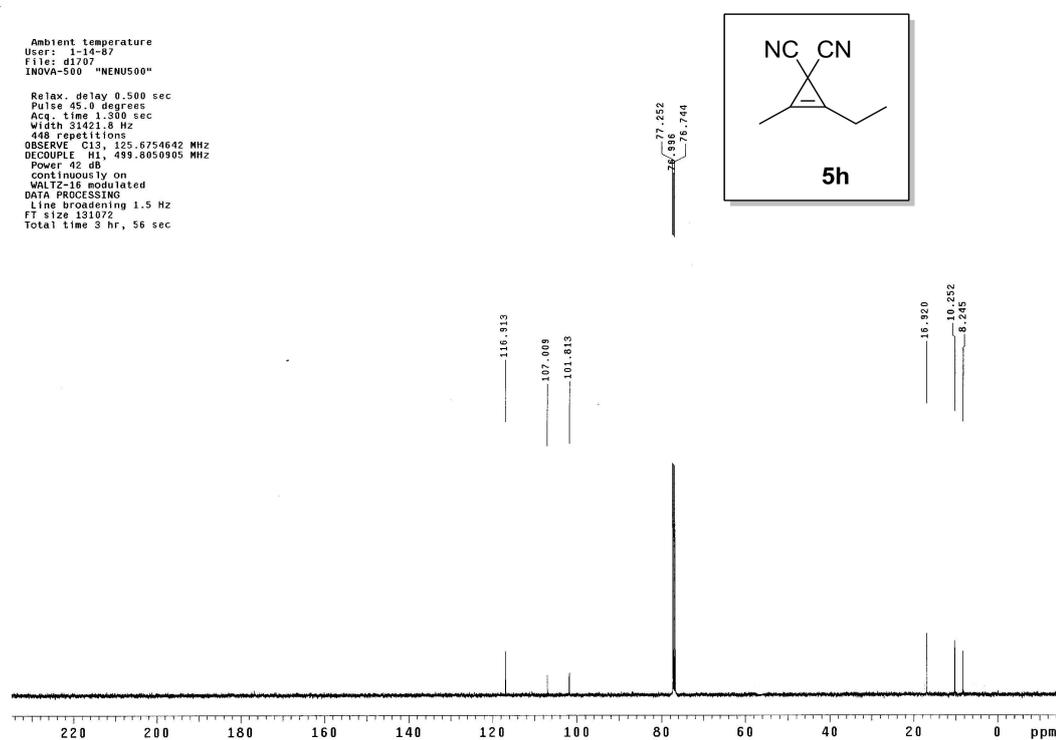
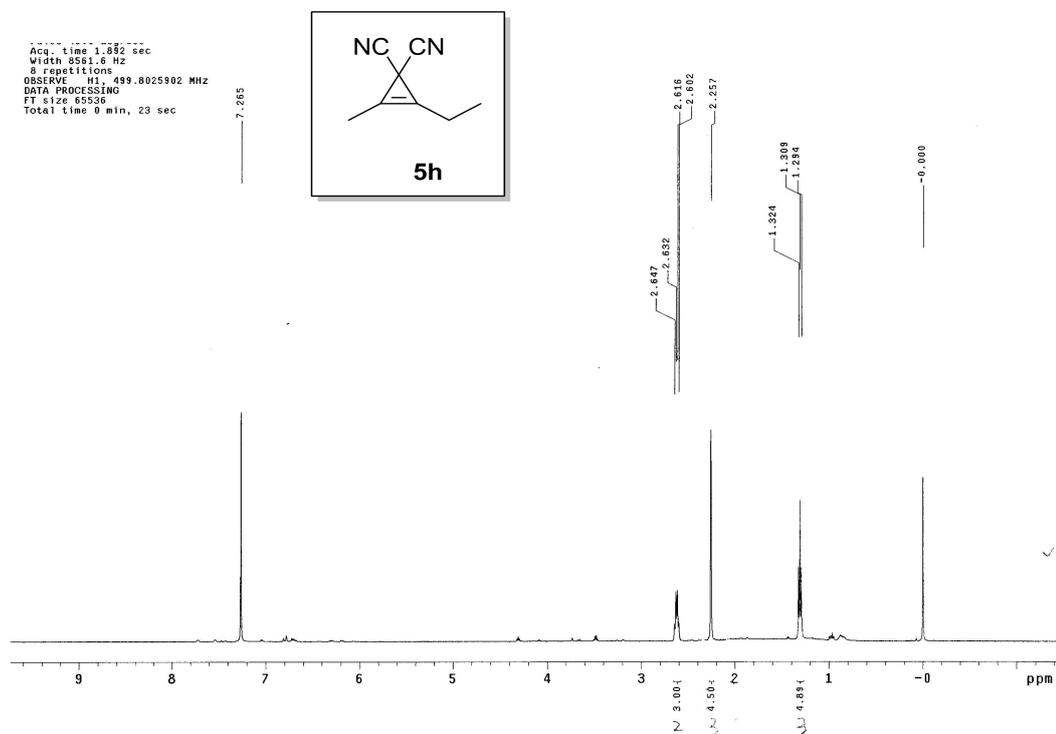


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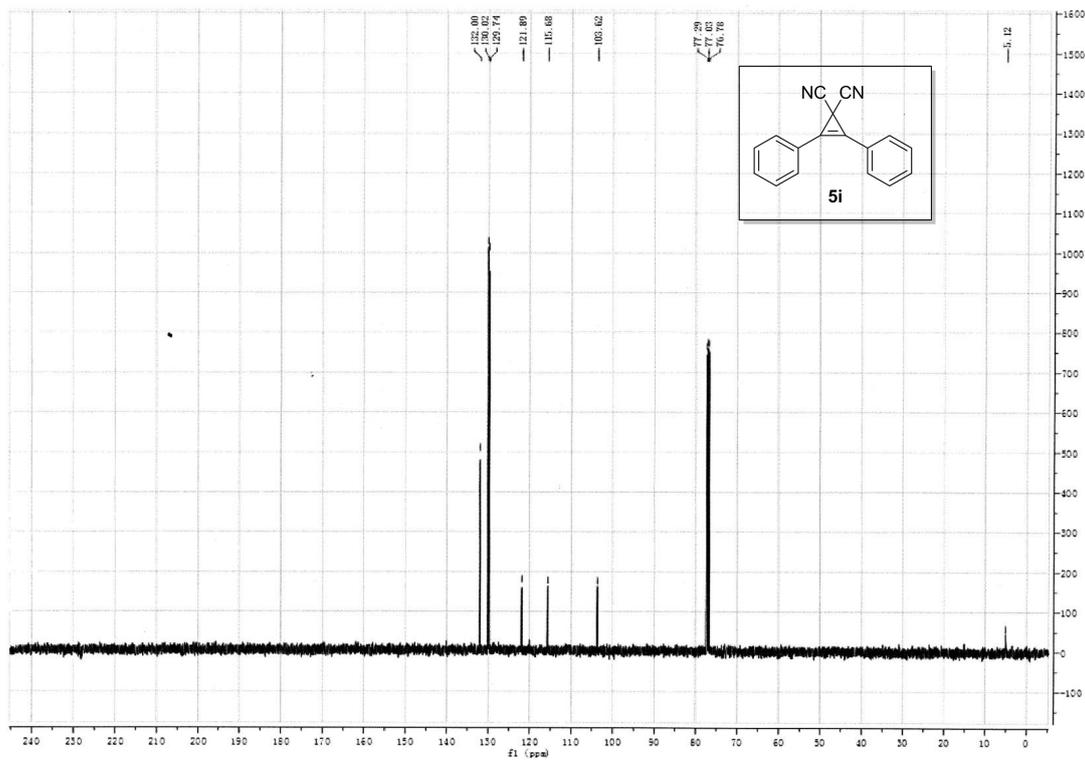
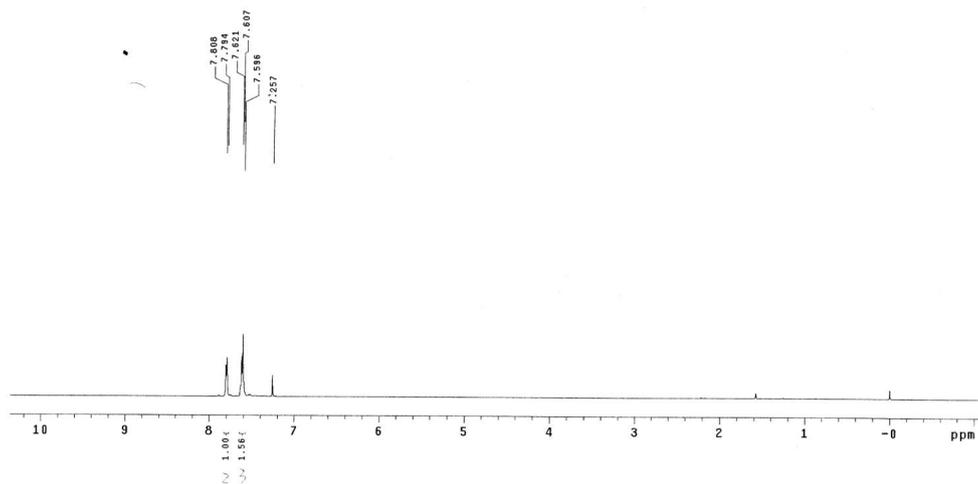
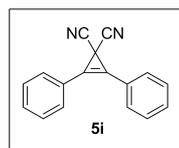


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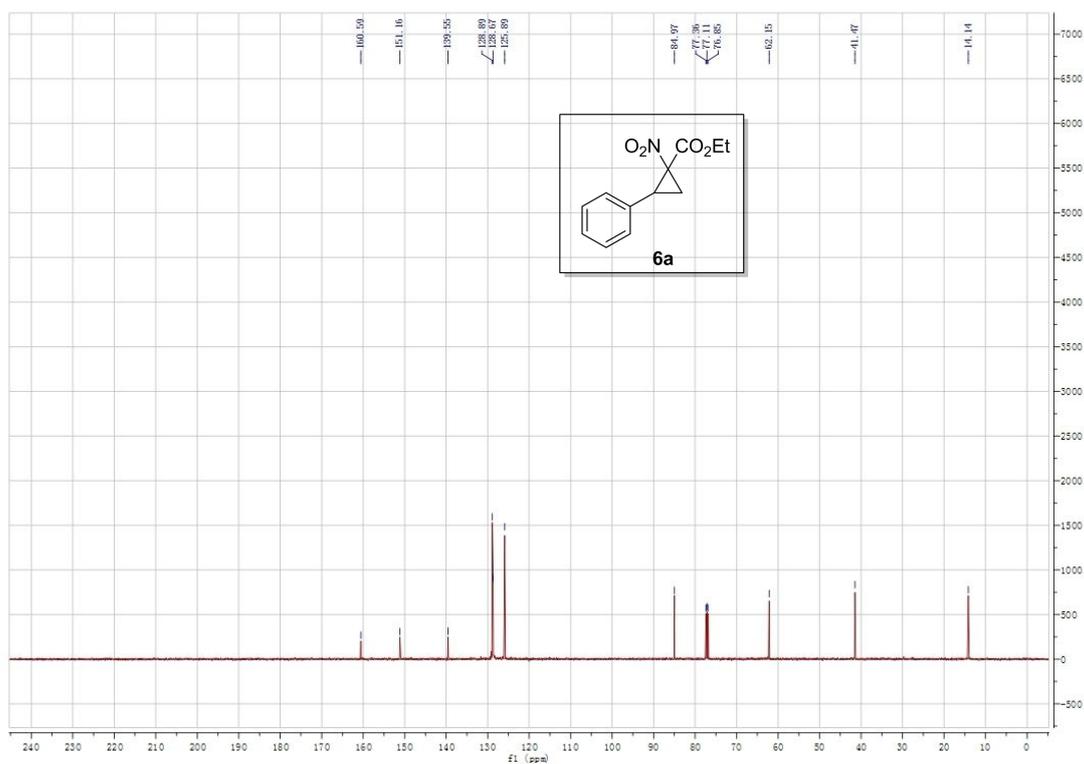
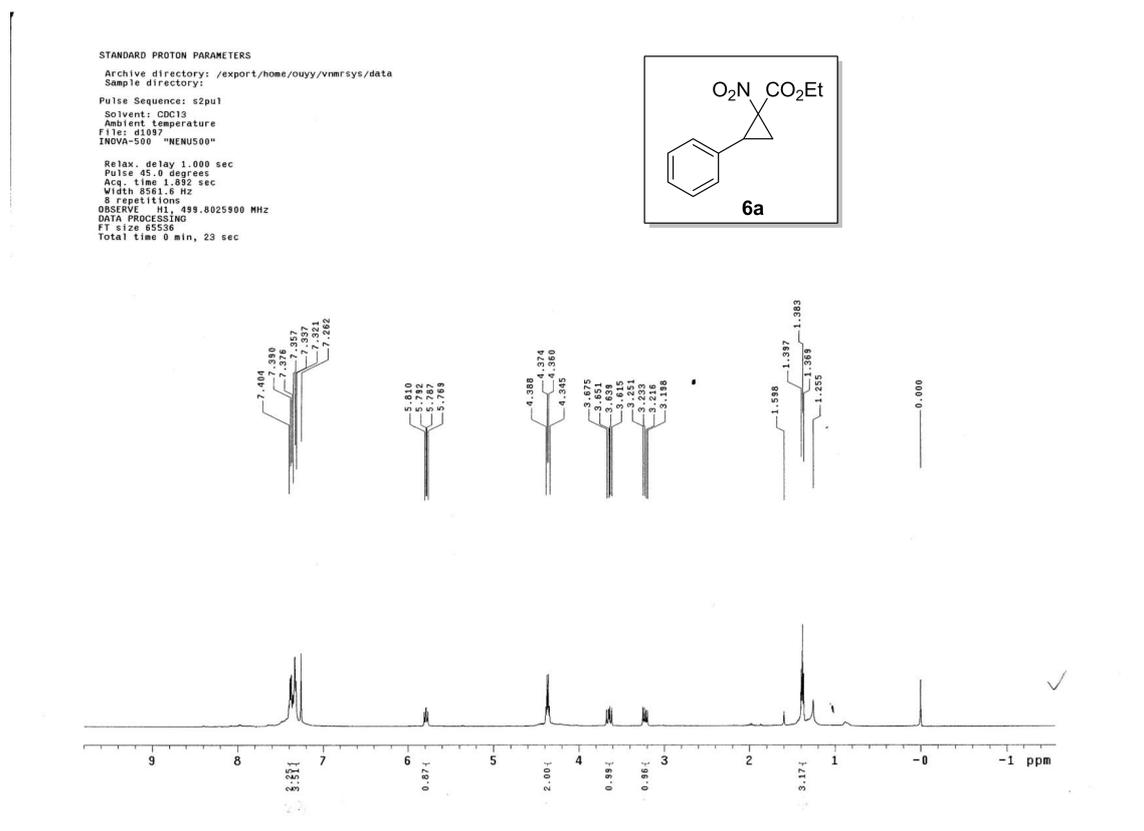


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STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient Temperature
File: d1158
INOVA-500 "HENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9329.4 Hz
S repetitions
OBSERVE H1, 499.8025845 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec



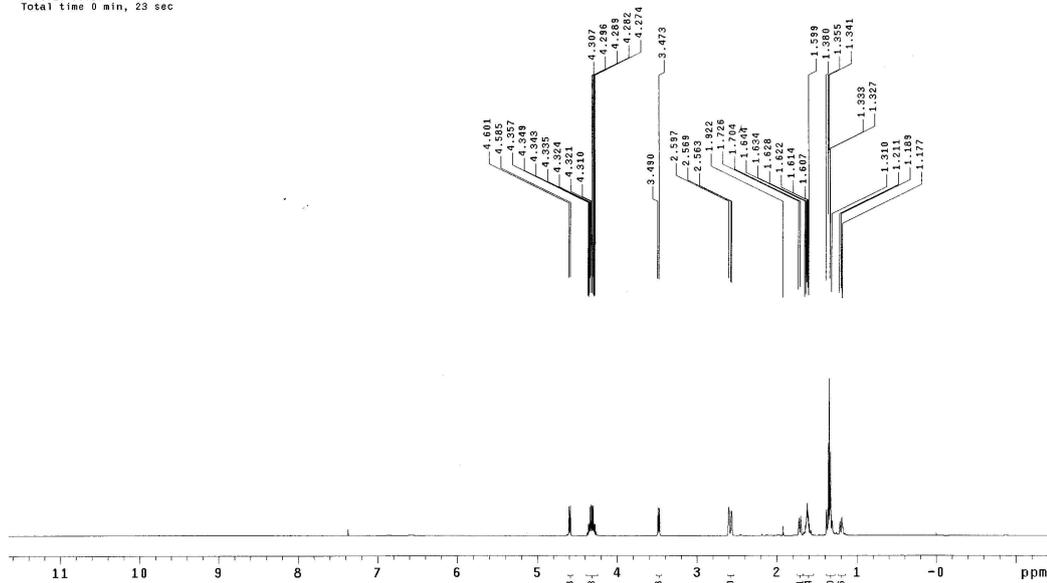
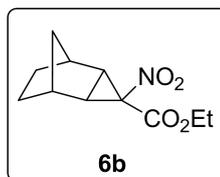
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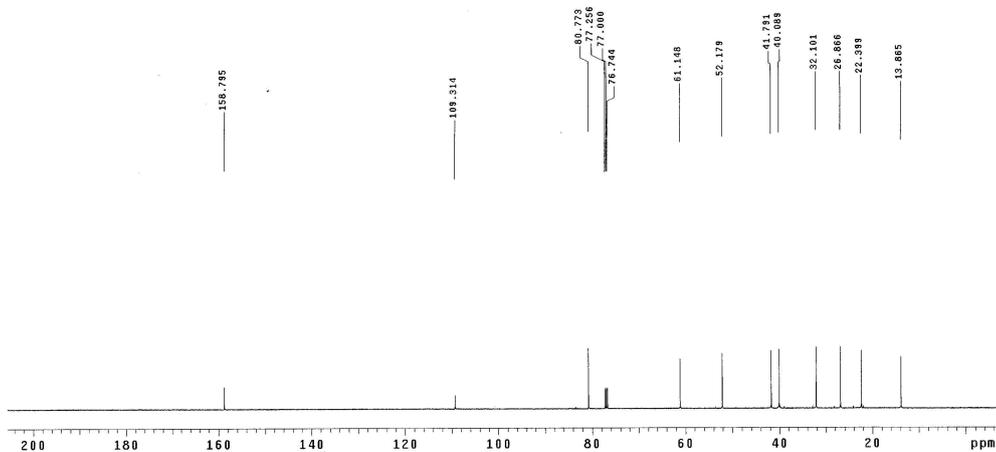
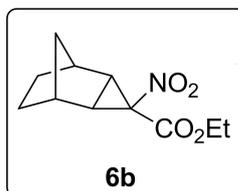
STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d1661
INOVA-500 "MNU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10895.2 Hz
8 repetitions
OBSERVE H1, 499.8025362 MHz
DATA PROCESSING
FT size 8538
Total time 0 min, 23 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d1662
INOVA-500 "MNU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754953 MHz
DECOUPLE H1, 499.8039905 MHz
Power 42 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec

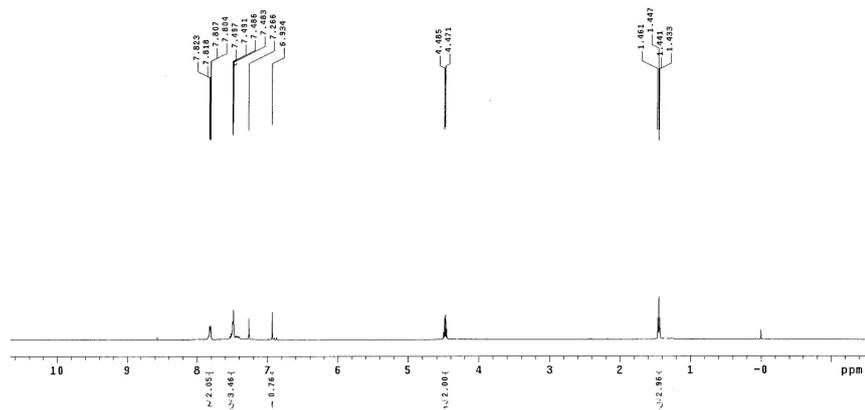
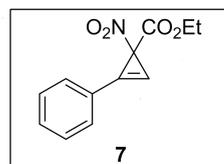


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STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: d1500
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.812 sec
Width 3223.4 Hz
8 repetitions
OBSRV: 499.8025879 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec

4-11



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III. Control experiment.

After stirring the mixture of malononitrile, $\text{PhI}(\text{OAc})_2$ and K_2CO_3 under the reaction conditions about 5-10 min, sample was taken and diluted with CH_3CN prior to the injection into the mass spectrometer. However, no peaks corresponding to PhIO ($m/z = 219.9385$) or ligand exchange between acetate anion and malononitrile ($m/z = 327.9709$ or $m/z = 333.9715$) were detected.

