Synthesis of Spirocyclic Thiazolidinediones Using Ring-Closing Metathesis and One-Pot Sequential Ring-Closing/Cross Metathesis

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Supporting Information

Contents

X-Ray Crystallography of 4: Intensity data were collected on a Bruker's Kappa Apex II CCD Duo diffractometer with graphite monochromated $Mo_{K\alpha}$ radiation (0.71073 Å) at the temperature of 296 K. Scaling and multi-scan absorption correction were employed using SADABS.[1] The structure was solved by direct methods and all the non-hydrogen atoms were refined anisotropically while the hydrogen atoms fixed in the predetermined positions by Shelxs-97 and Shelxl-97 packages respectively.[2]

[1] Bruker. SADABS V2008-1, Bruker AXS: Madison, WI, USA (**2008**).
[2] G. M. Sheldrick, SHELX93, Program for Crystal Structure Determination, University of Göttingen (**1997**).

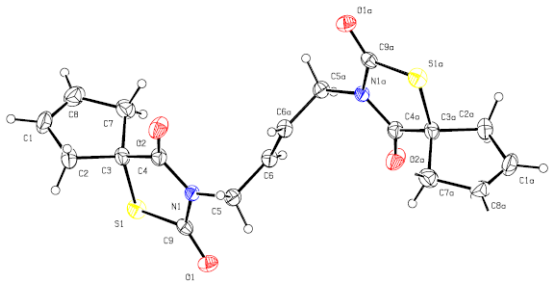


Figure 1: The ORTEP diagram of dimer **4** showing the atomic numbering scheme. Ellipsoids are drawn at the 30% probability level.

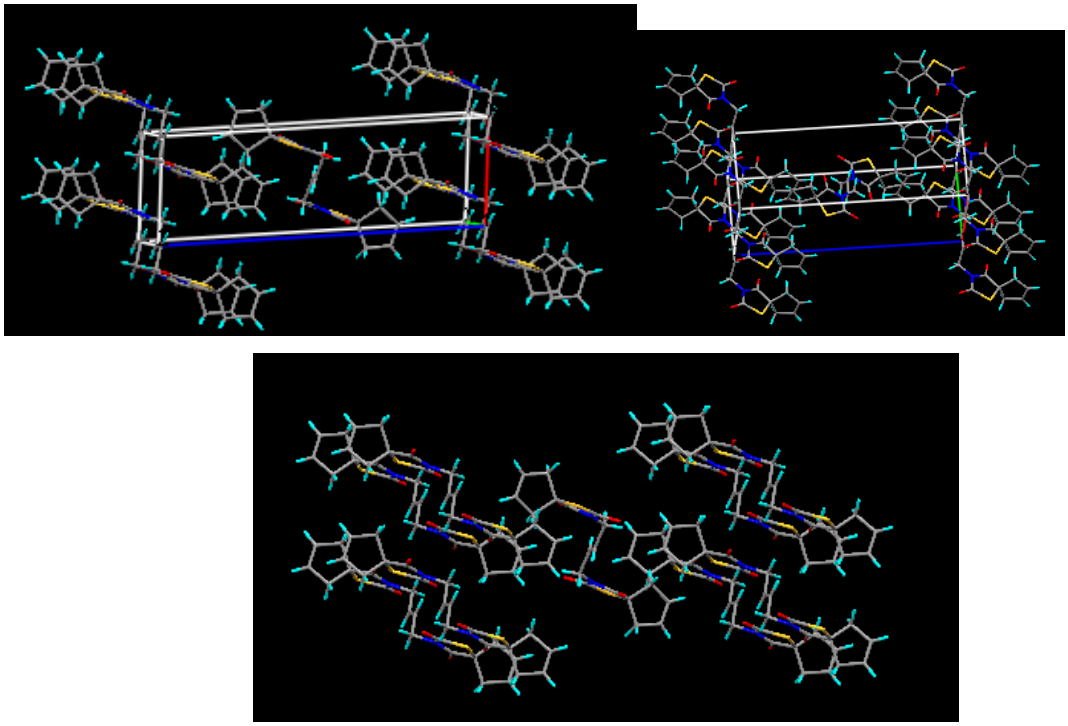


Figure 2: The packing diagram of dimer **4** shows body centered cubic arrangement in the unit cell.

Crystal Data and Details of the Structure Determination for dimer 4:

Formula	$C_{18}\ H_{18}\ N_2\ O_4\ S_2$
Formula weight	390.48
Crystal system	Monoclinic
Crystal size	0.11 x 0.11 x 0.11 mm
Space group	P21/n
a, b, c [Å]	6.1705(7), 7.7879(8),18.703(3)
α, β, γ [º]	90, 94.971, 90
U [$Å^3$]	895.4
Z	2
dm [Mgm^{-3}]	1.448

Data Collection

Temperature (K)	296
MoKα (Å)	0.71073
θ Min, Max [º]	2.2, 22.6
Tot., Uniq. Data	6657, 1192
Observed data [I > 2σ(I)]	927

Refinement

R, wR2	0.0390, 0.1138
Min., Max. resd. dens. [e/Å-^{3}]	-0.23, 0.19

Table 1: Bond Distances (Å)

S1-C3	1.821	C5-C6	1.490
S1-C9	1.755	C6-C6a	1.301
O1-C9	1.203	C7-C8	1.480
O2-C4	1.212	C1-H1	0.9300
N1-C4	1.363	C2-H2A	0.9700
N1-C5	1.478	C2-H2B	0.9700
O2-C4	1.212	C1-H1	0.9300
N1-C4	1.363	C2-H2A	0.9700
N1-C5	1.478	C2-H2B	0.9700
N1-C9	1.389	C5-H5A	0.9700
C1-C2	1.482	C5-H5B	0.9700
C1-C8	1.309	C6-H6	0.9300
C2-C3	1.553	C7-H7A	0.9700
C3-C4	1.514	C7-H7B	0.9700
C3-C7	1.544	C8-H8	0.9300

Table 2: Bond Angles (°)

C3-S1-C9	93.29	C2-C1-H1	124.00
C4-N1-C5	121.4	C8-C1-H1	123.00
C4-N1-C9	116.7	C1-C2-H2A	111.00
C5-N1-C9	121.8	C1-C2-H2B	111.00
C2-C1-C8	113.0	C3-C2-H2A	111.00
C1-C2-C3	103.0	C3-C2-H2B	111.00
S1-C3-C2	111.3	H2A-C2-H2B	109.00
S1-C3-C2	111.3	H2A-C2-H2B	109.00
S1-C3-C4	105.5	N1-C5-H5A	109.00
S1-C3-C7	111.3	N1-C5 -H5B	109.00
C2-C3-C4	112.5	C6-C5-H5A	109.00
C2-C3-C7	104.9	C6 -C5-H5B	109.00
C4-C3-C7	111.5	H5A-C5-H5B	108.00
O2-C4-N1	123.2	C5-C6-H6	118.00
O2-C4-C3	123.3	C6a-C6-H6	118.00
N1-C4-C3	113.5	C3-C7-H7A	111.00
N1-C5-C6	111.5	C3-C7-H7B	111.00
C5-C6-C6a	124.8	C8-C7-H7A	111.00
C3-C7-C8	103.5	C8-C7-H7B	111.00
C1-C8-C7	112.5	H7A-C7-H7B	109.00
S1-C9-O1	124.8	C1-C8-H8	124.00
S1-C9-N1	110.6	C7-C8-H8	124.00
O1-C9-N1	124.6		

Table 3 - Torsion Angles (º)

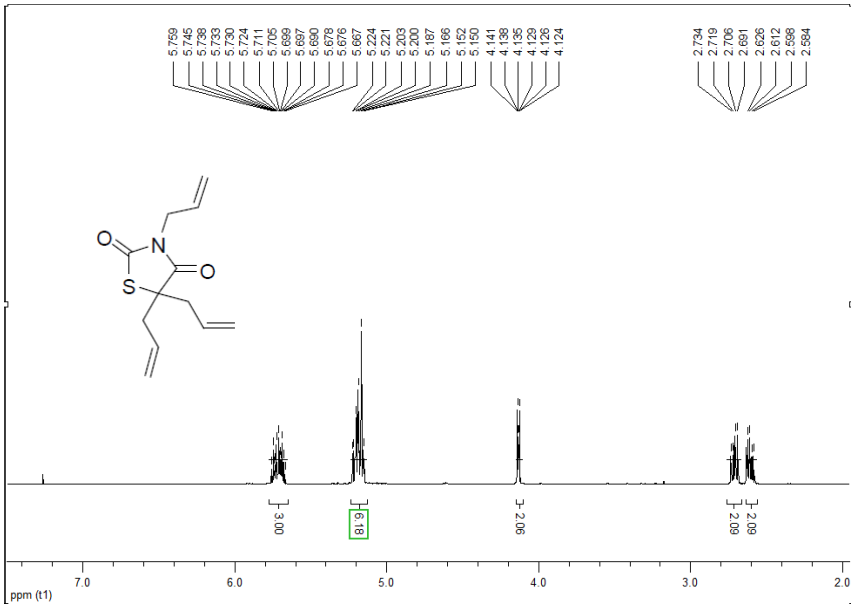
C9-S1-C3-C2	-122.1	C9-S1-C3-C4	0.21
C9-S1-C3-C7	121.3	C3-S1-C9-O1	175.2
C3-S1-C9-N1	-3.94	C5-N1-C4-O2	-4.2
C9-N1-C4-O2	173.3	C5-N1-C4-C3	175.3
C9-N1-C4-C3	-7.3	C4-N1-C5-C6	-71.7
C9-N1-C5-C6	110.9	C4-N1-C9-S1	7.2
C4-N1-C9-O1	-171.9	C5-N1-C9-S1	-175.28
C5-N1-C9-O1	5.6	C8-C1-C2-C3	-10.6
C2-C1-C8-C7	-0.4	C1-C2-C3-C7	16.5
C1-C2-C3-S1	-104.0	C1-C2-C3-C4	137.9
S1-C3-C4-N1	3.7	C2-C3-C4-O2	-55.3
C2-C3-C4-N1	125.2	C7-C3-C4-O2	62.2
C4-C3-C7-C8	-138.8	C7-C3-C4-N1	-117.2
S1-C3-C7-C8	103.7	C2-C3-C7-C8	-16.8
S1-C3-C4-O2	-176.8	N1-C5-C6-C6a	125.3
C5-C6-C6a-C5a	-180.0	C3-C7-C8-C1	11.2

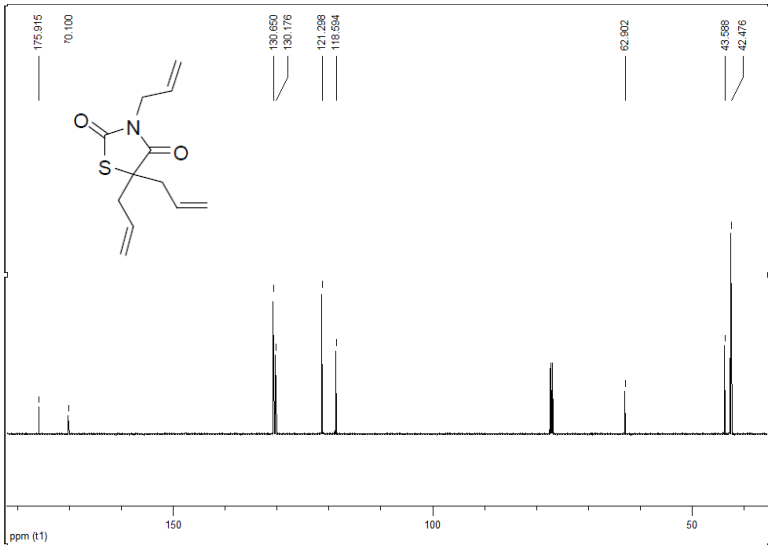
Table 4: Intermolecular Hydrogen Bonding Parameters

D-H-A	H---A (Å)	D---A (Å)	D-H
C1 -- H1..O2	2.5800	3.480(4)	162.00
C5 -- H5A..O1	2.4700	2.866(4)	104.00
C6 -- H6...O1	2.4900	3.248(3)	139.00

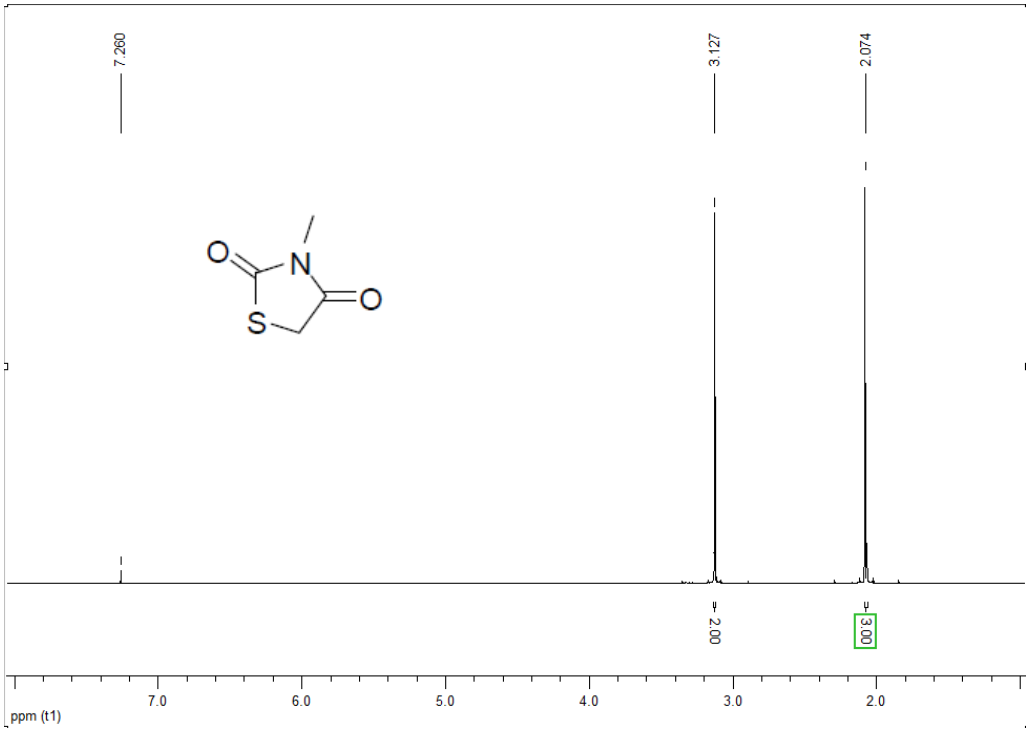
2.0 NMR spectra of all compounds

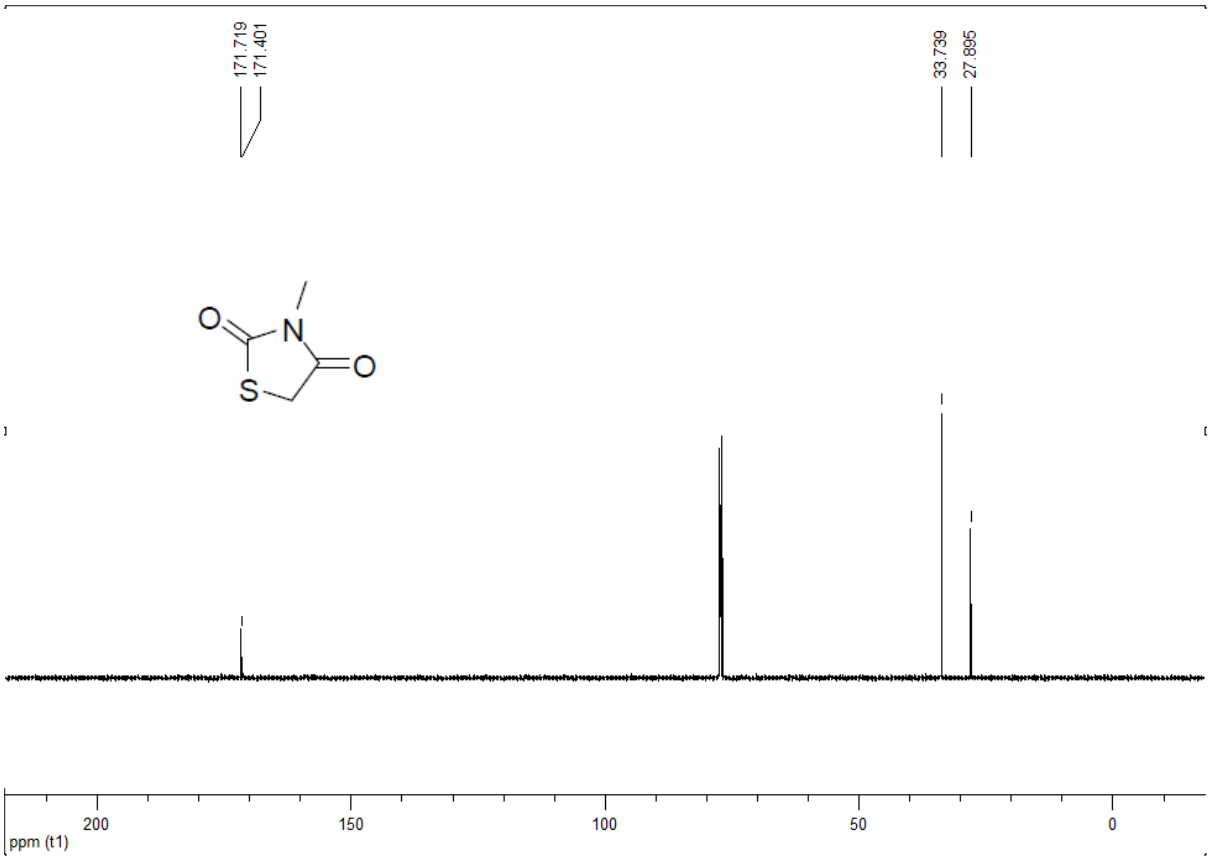
^{1}H and ^{13}C NMR for 2:



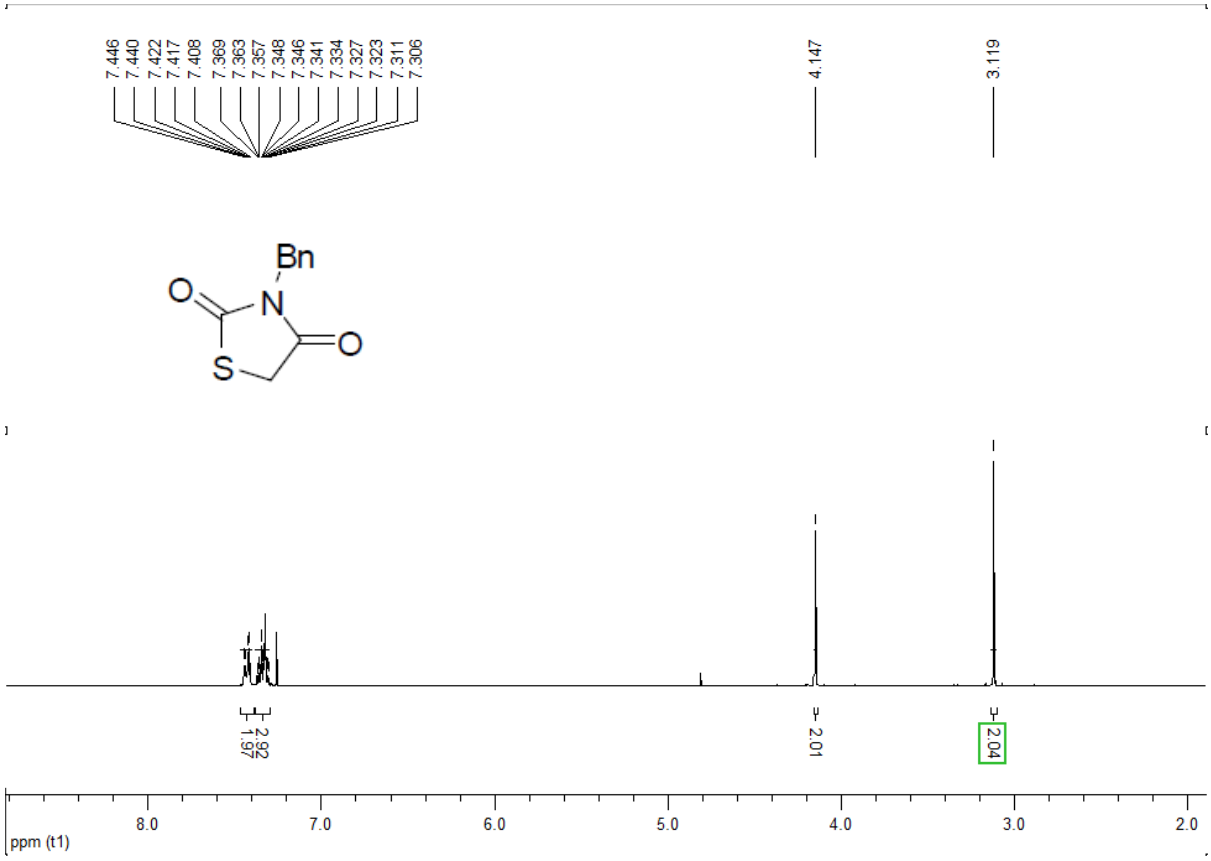


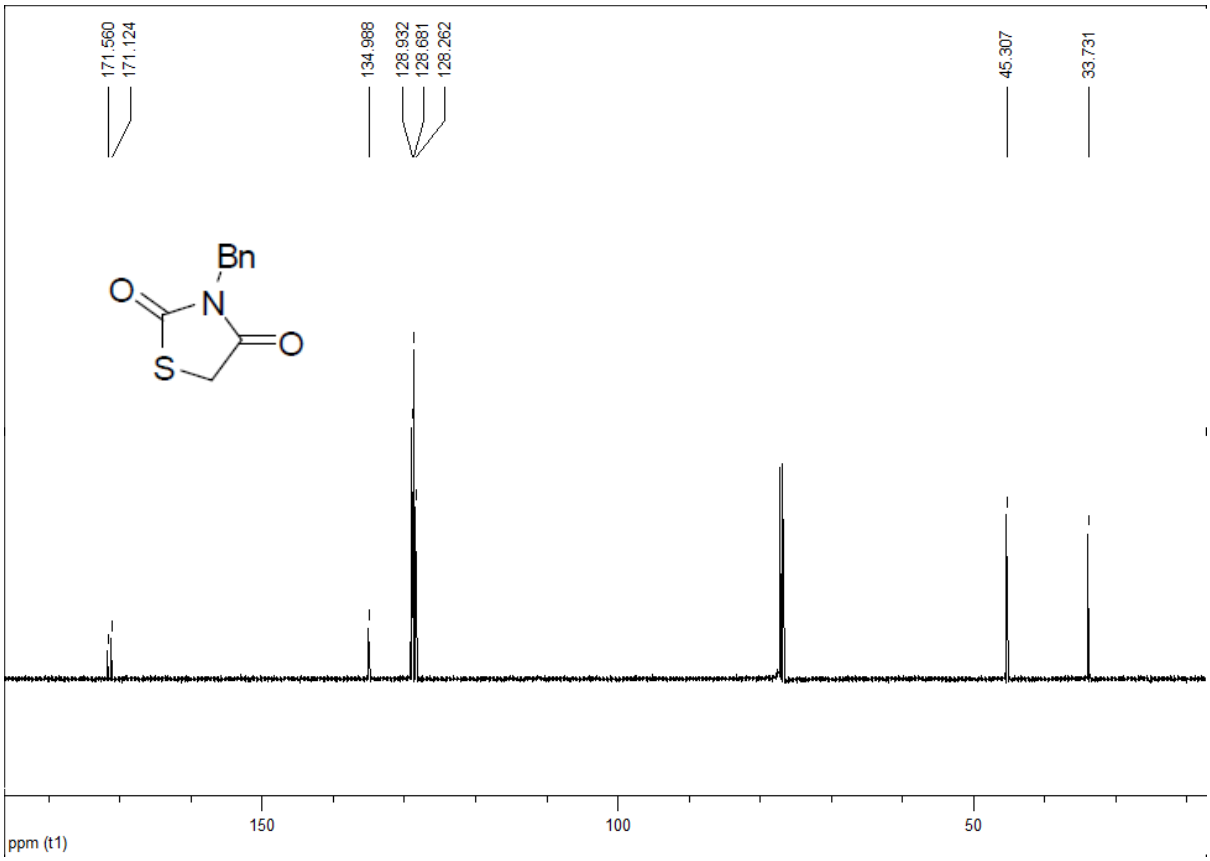
^{1}H and ^{13}C NMR for 5a:



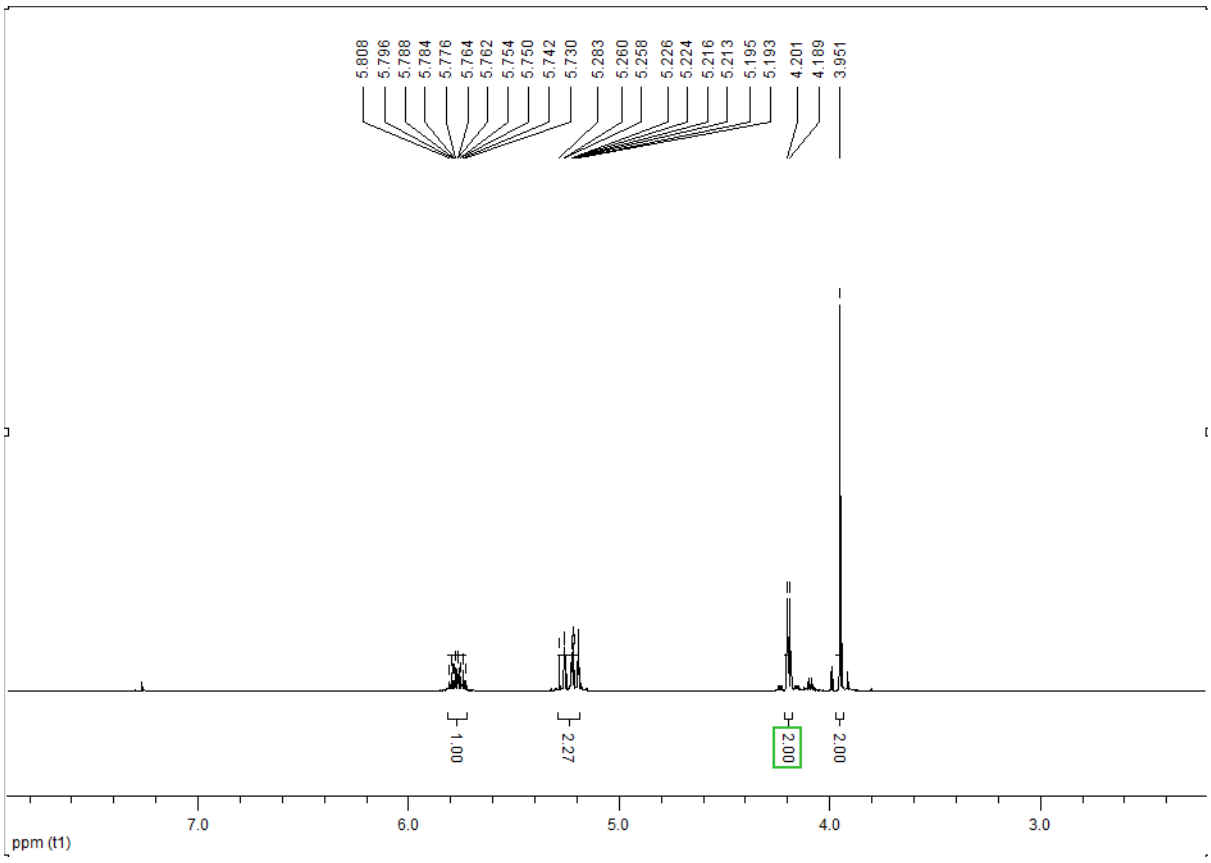


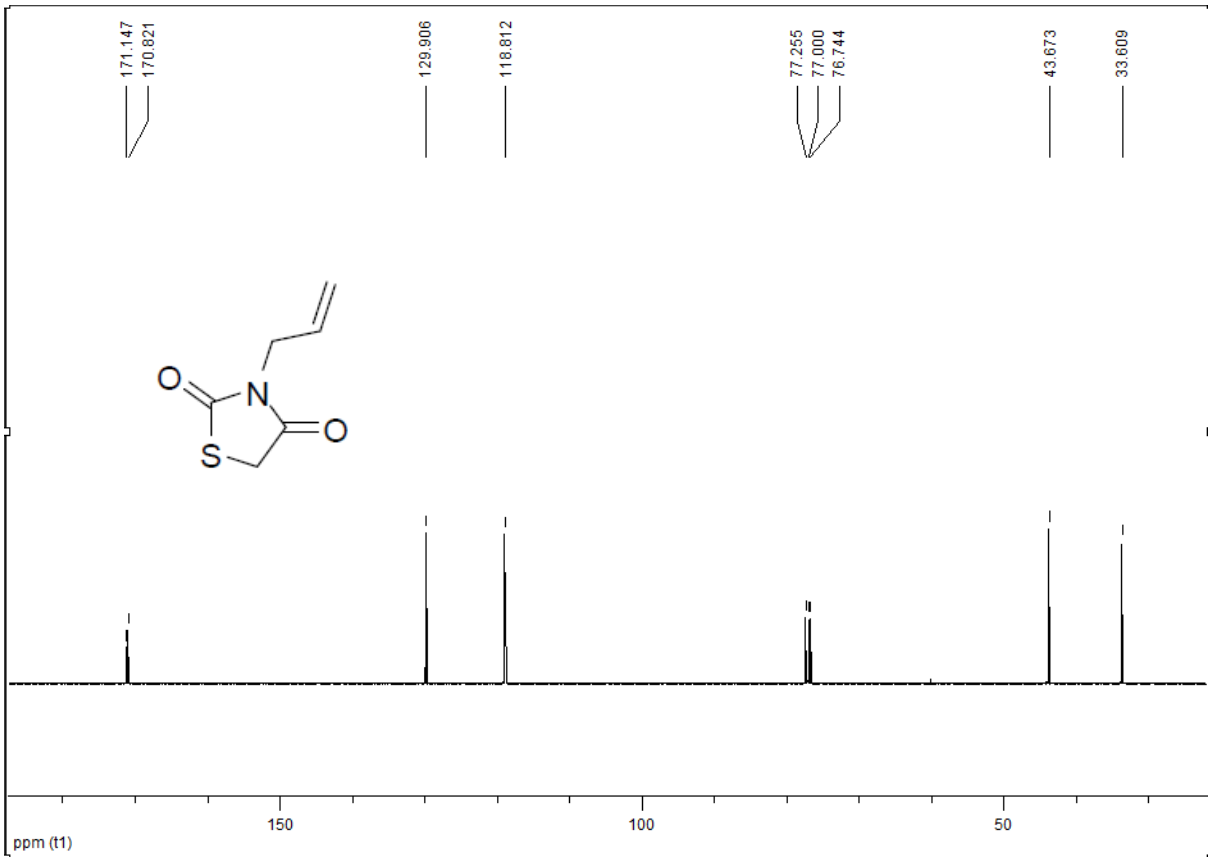
^{1}H and ^{13}C NMR for 5b:



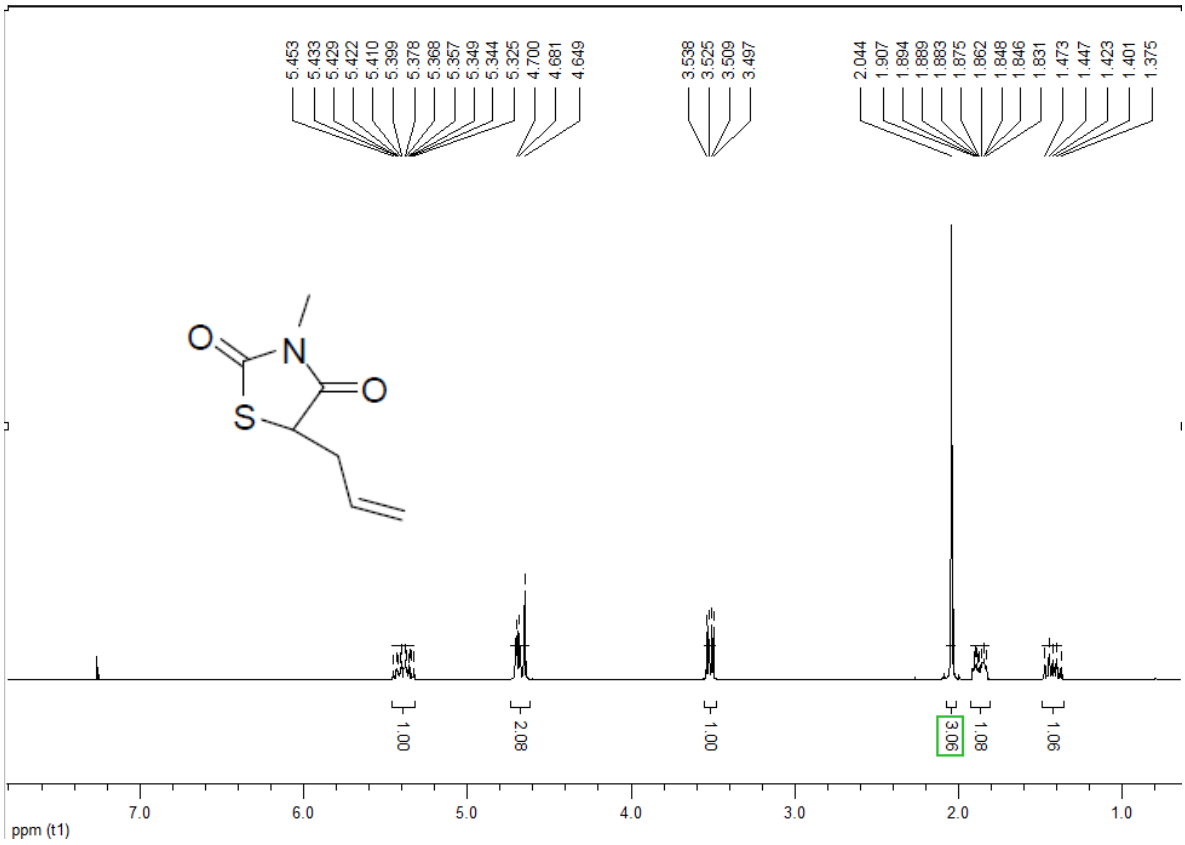


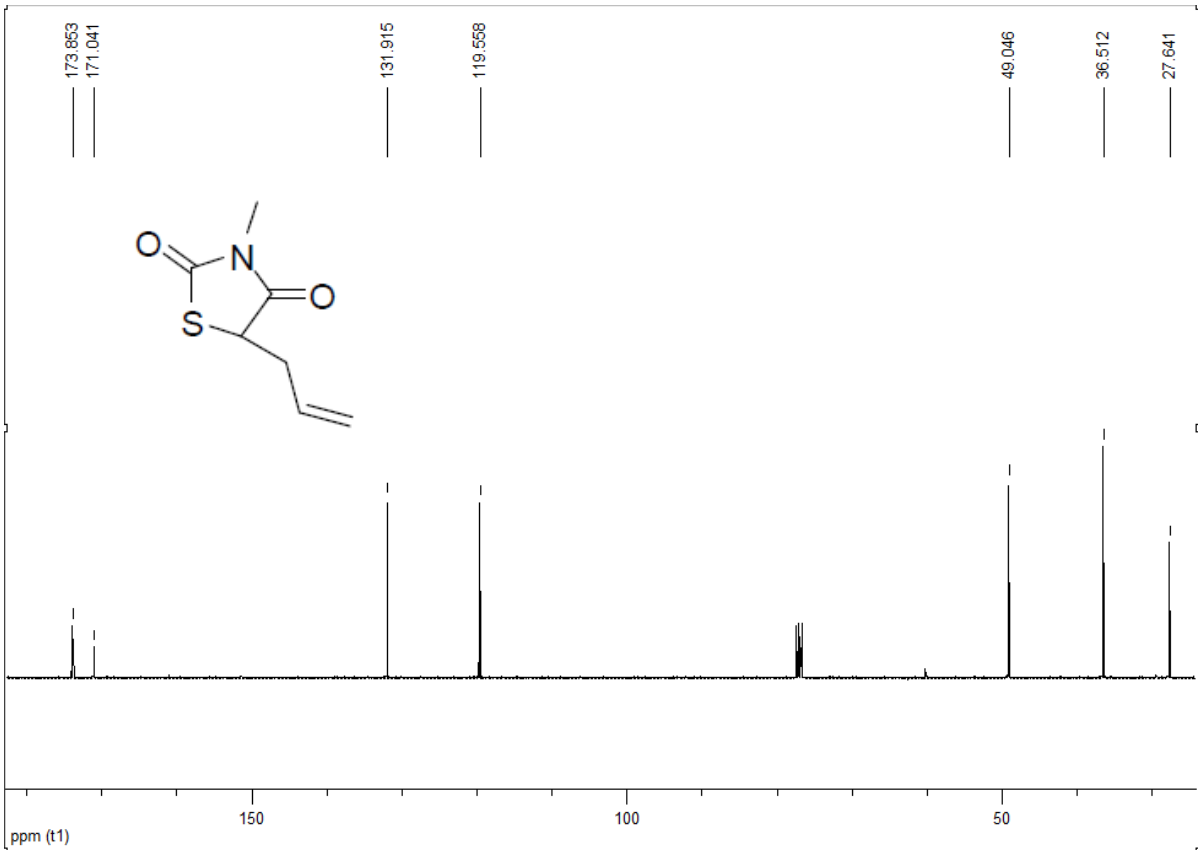
^{1}H and ^{13}C NMR for 5c:



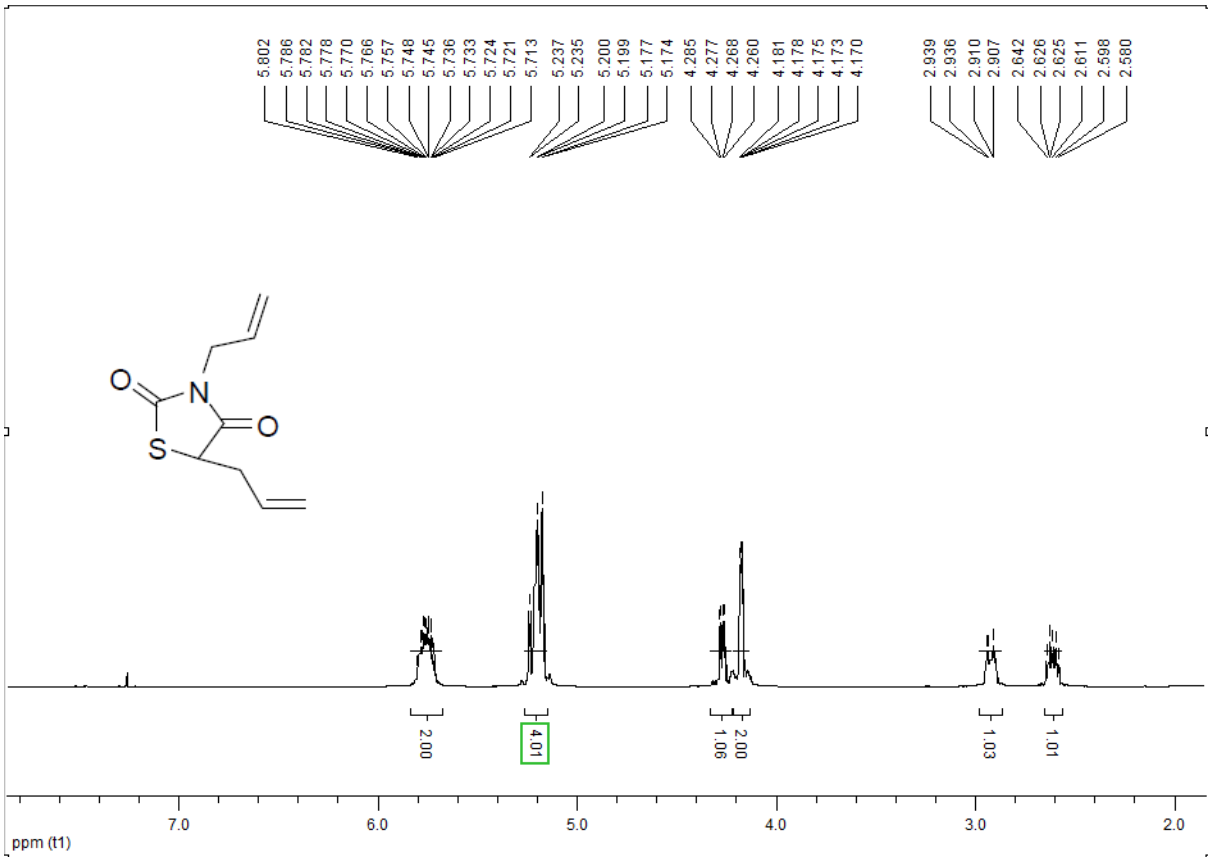


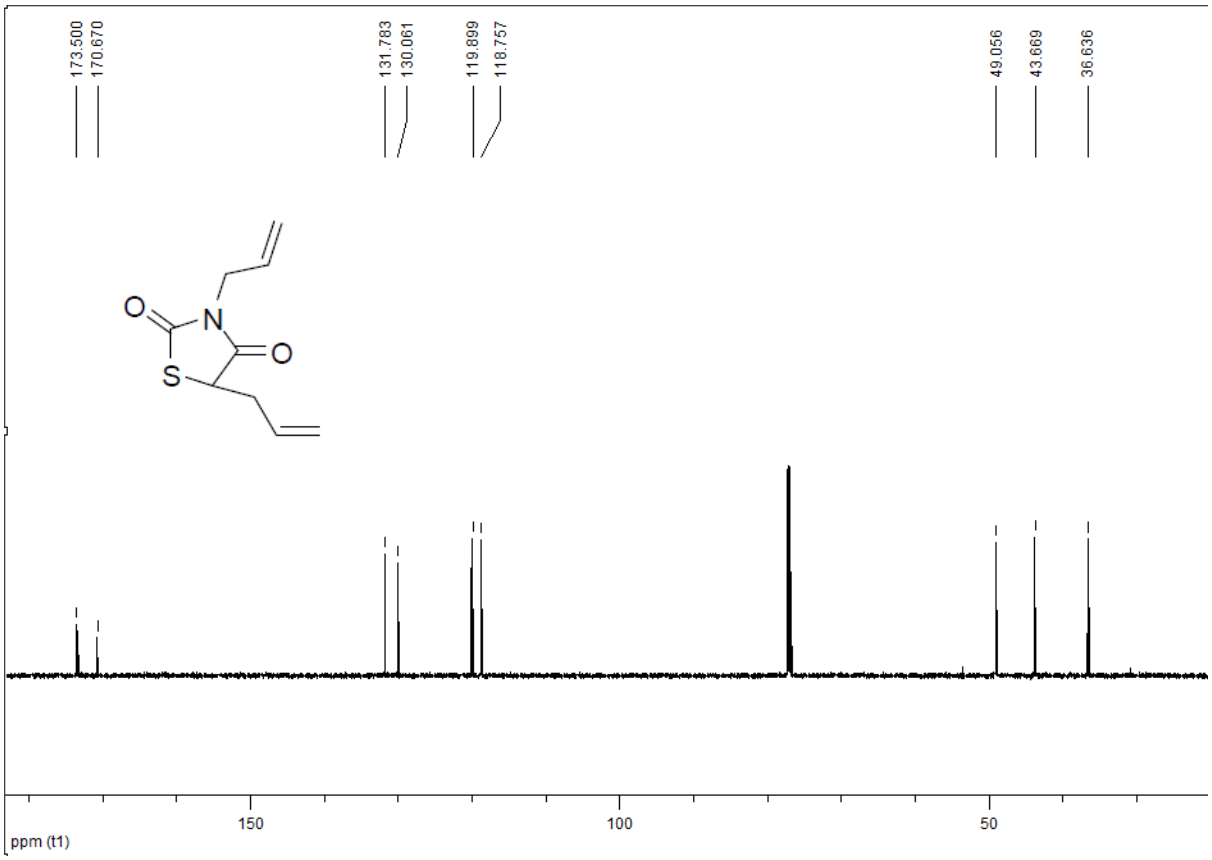
^{1}H and ^{13}C NMR for 6a:



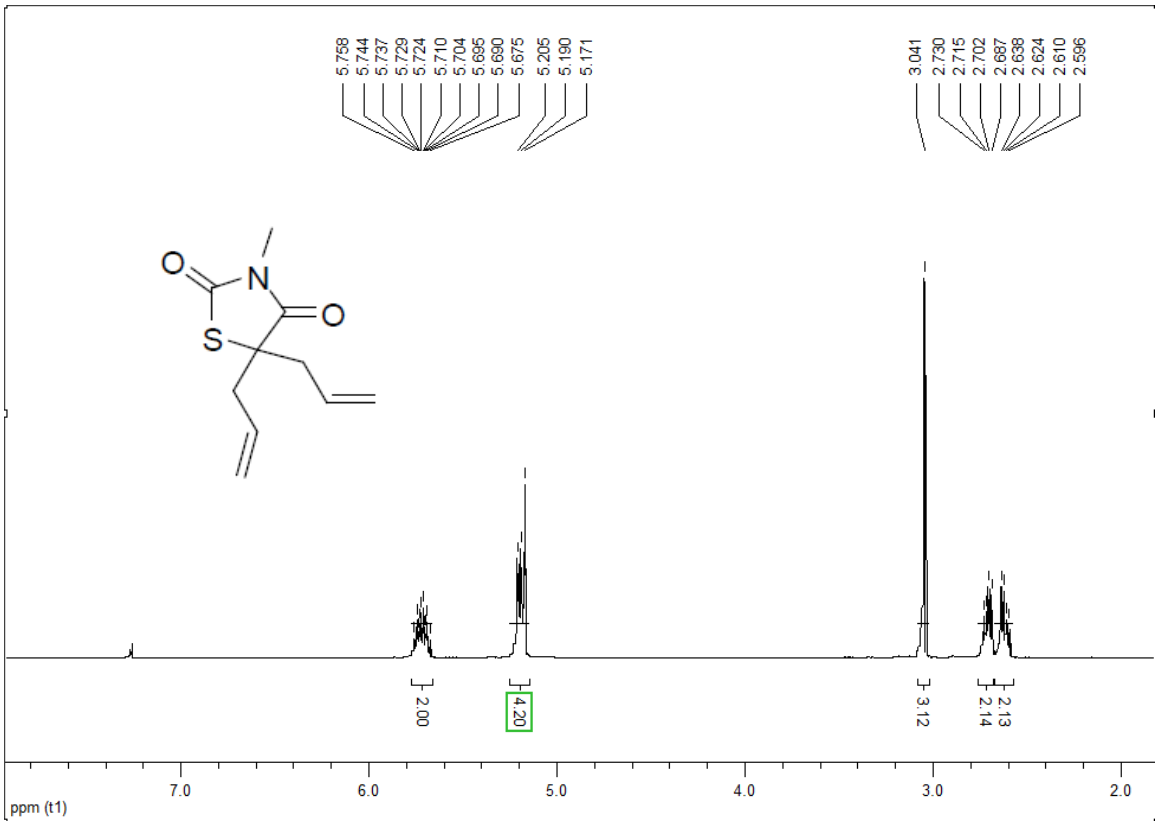


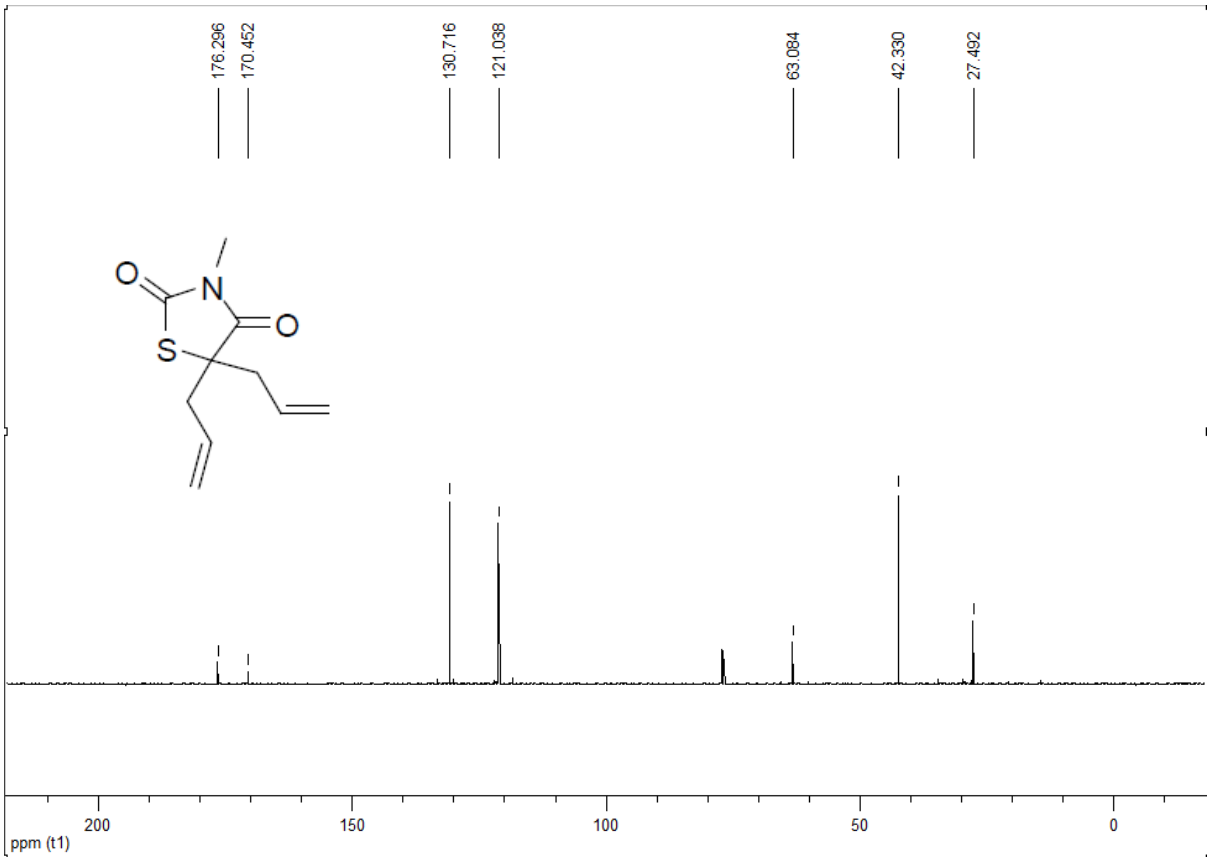
^{1}H and ^{13}C NMR for 6c:



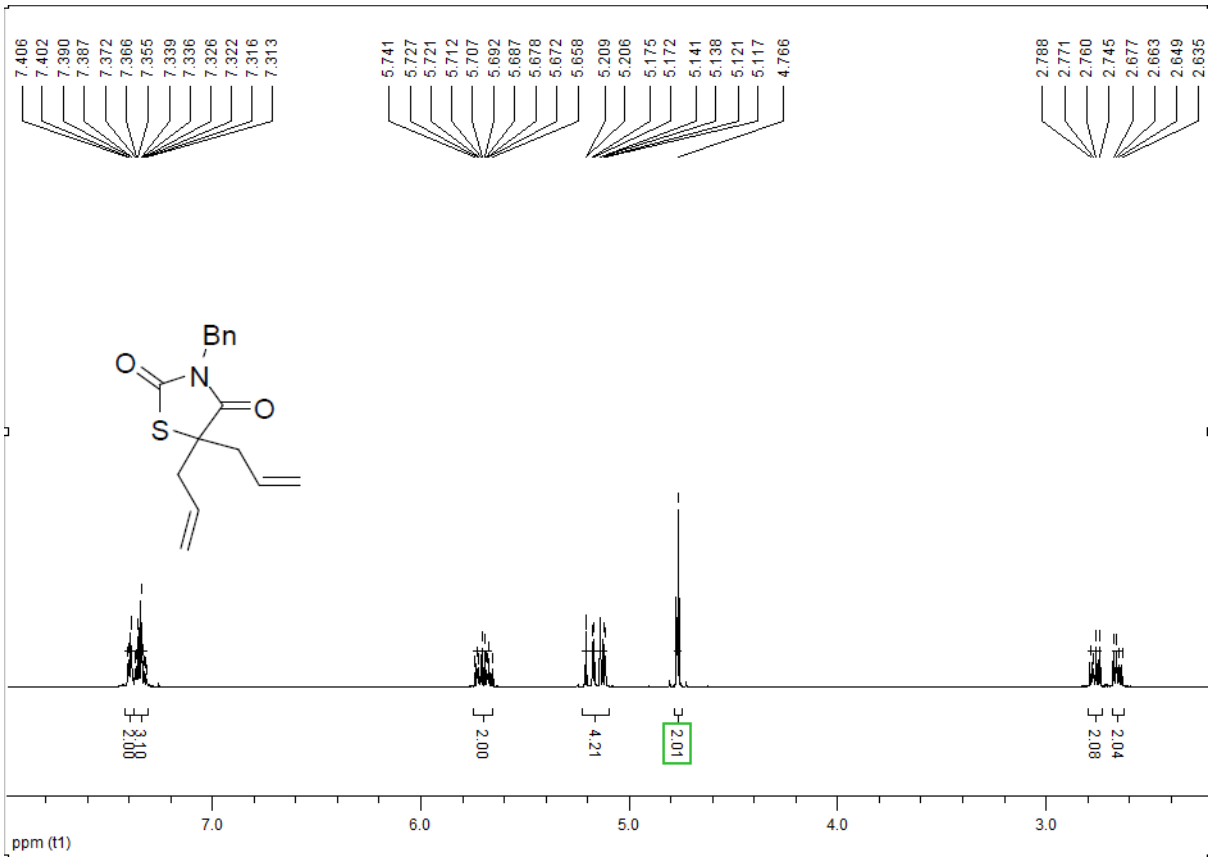


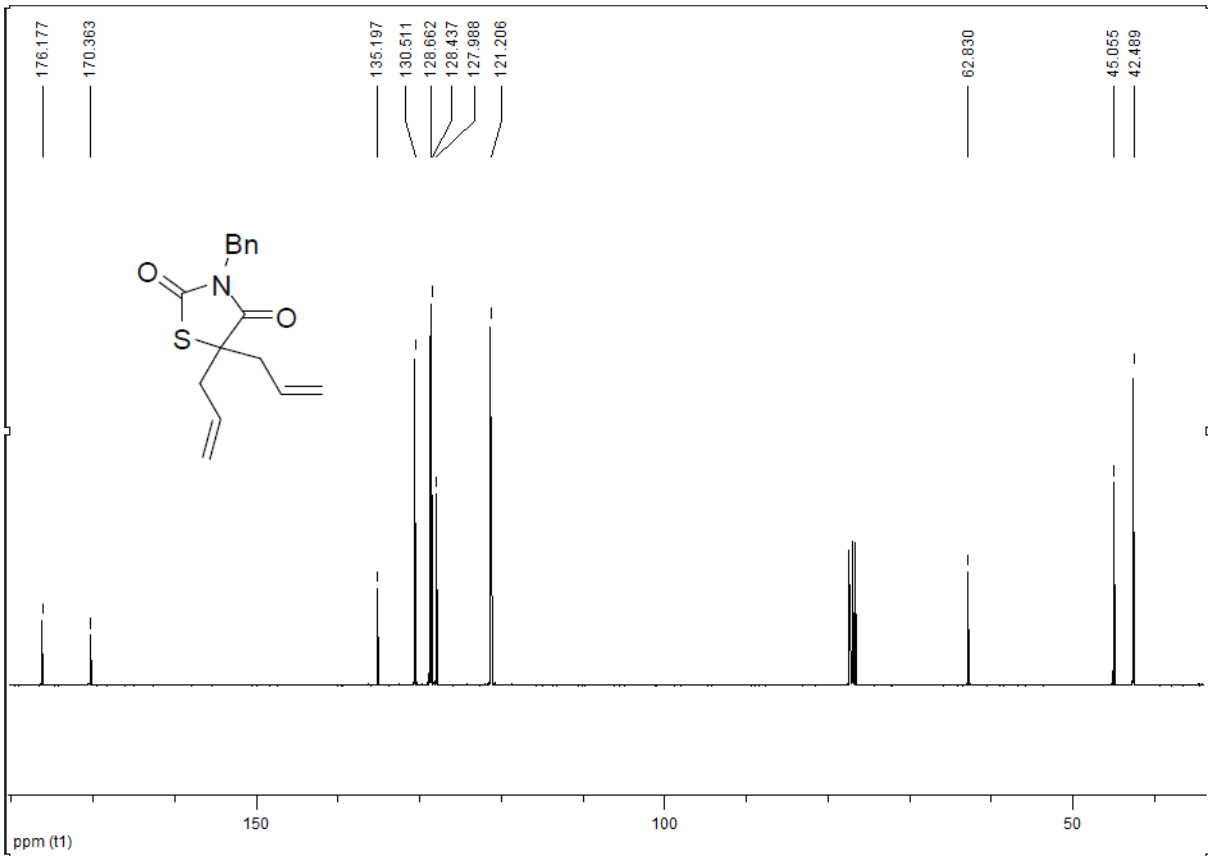
^{1}H and ^{13}C NMR for 7a:



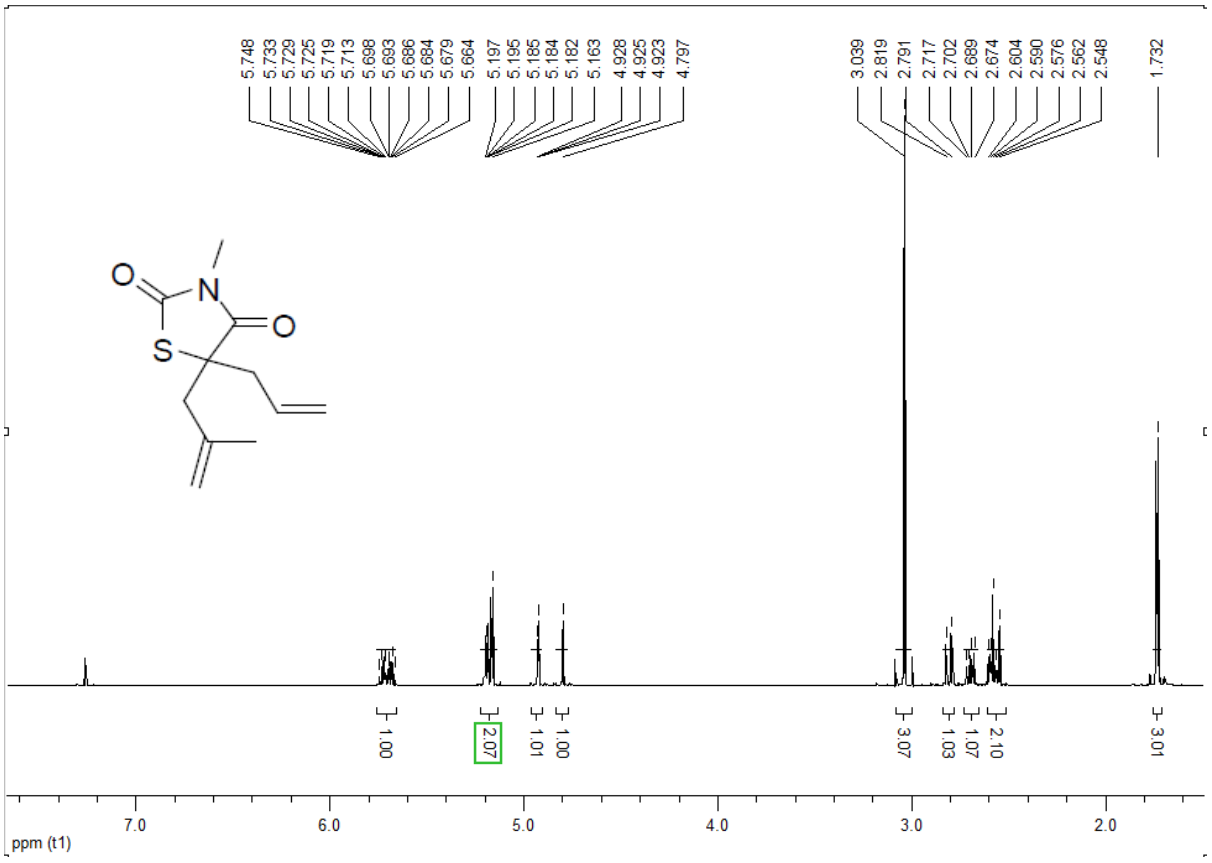


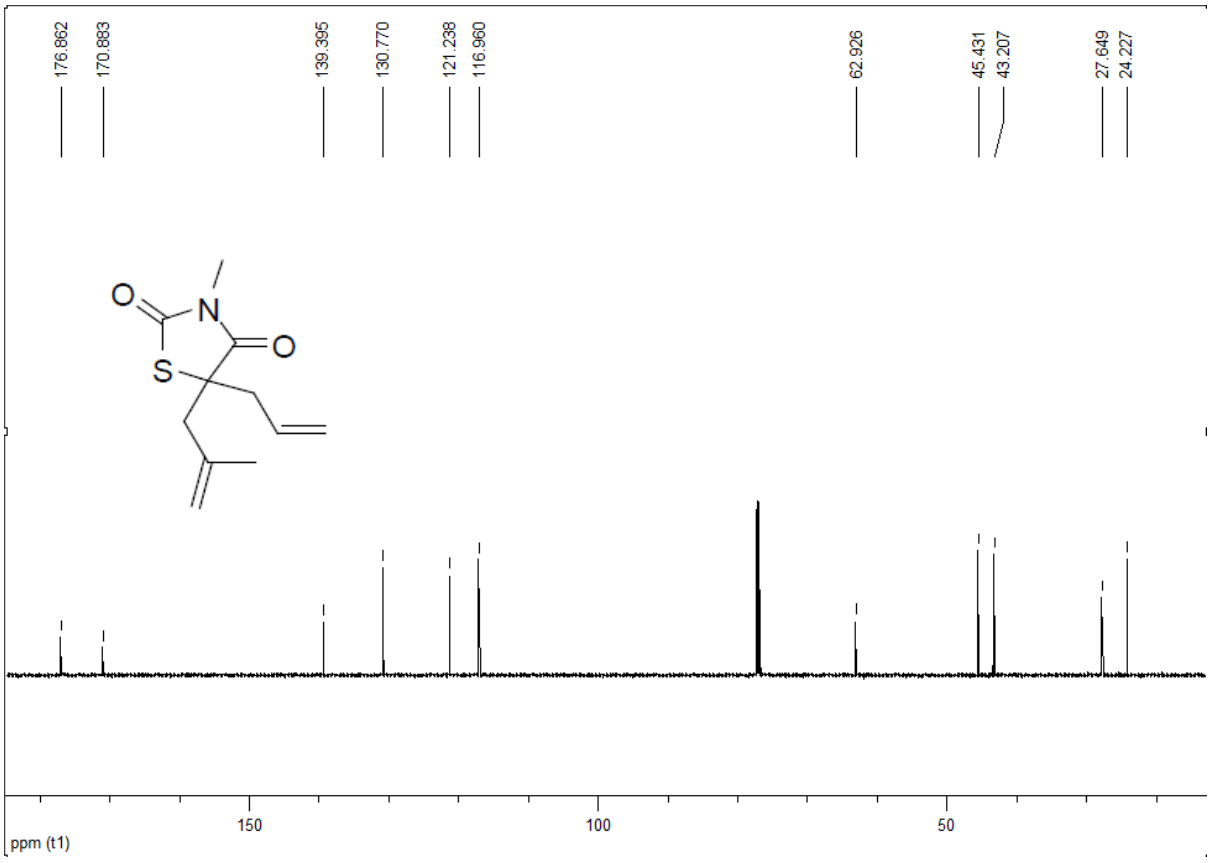
^{1}H and ^{13}C NMR for 7b:



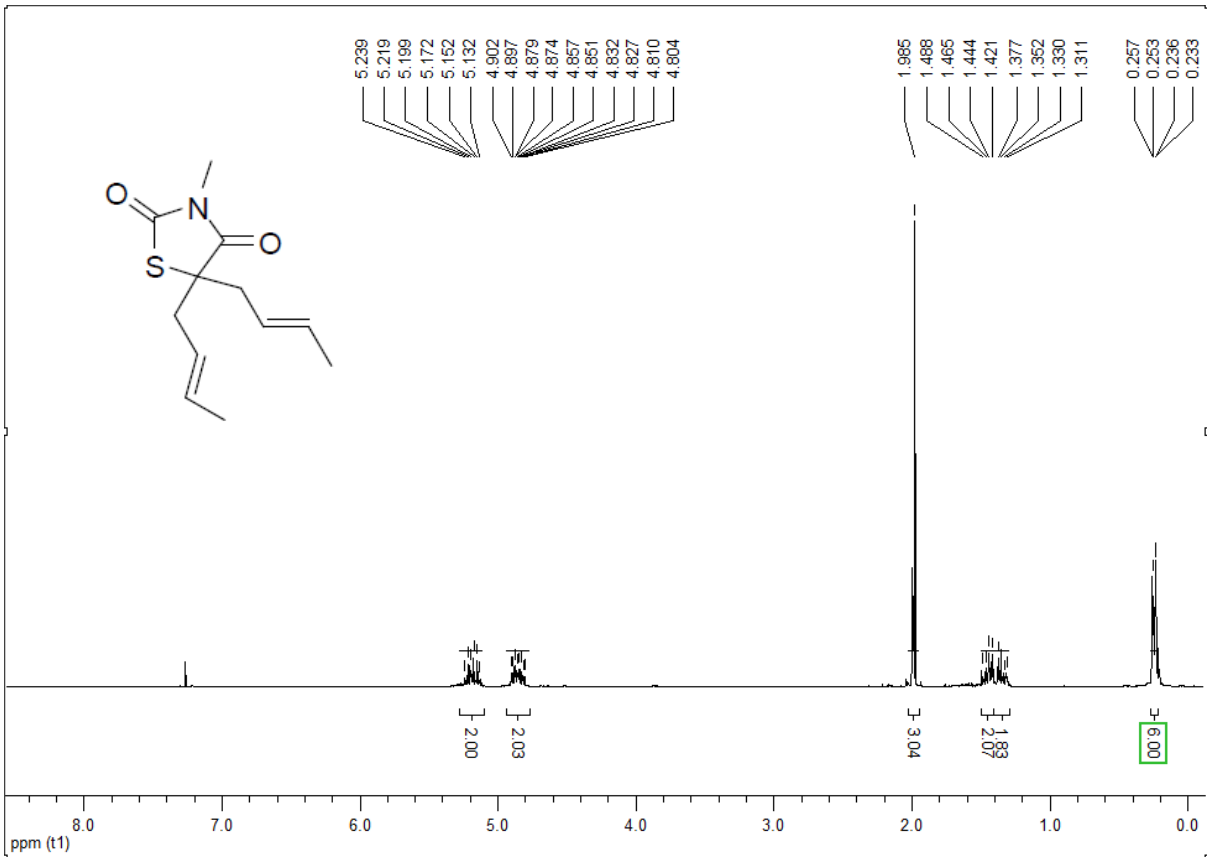


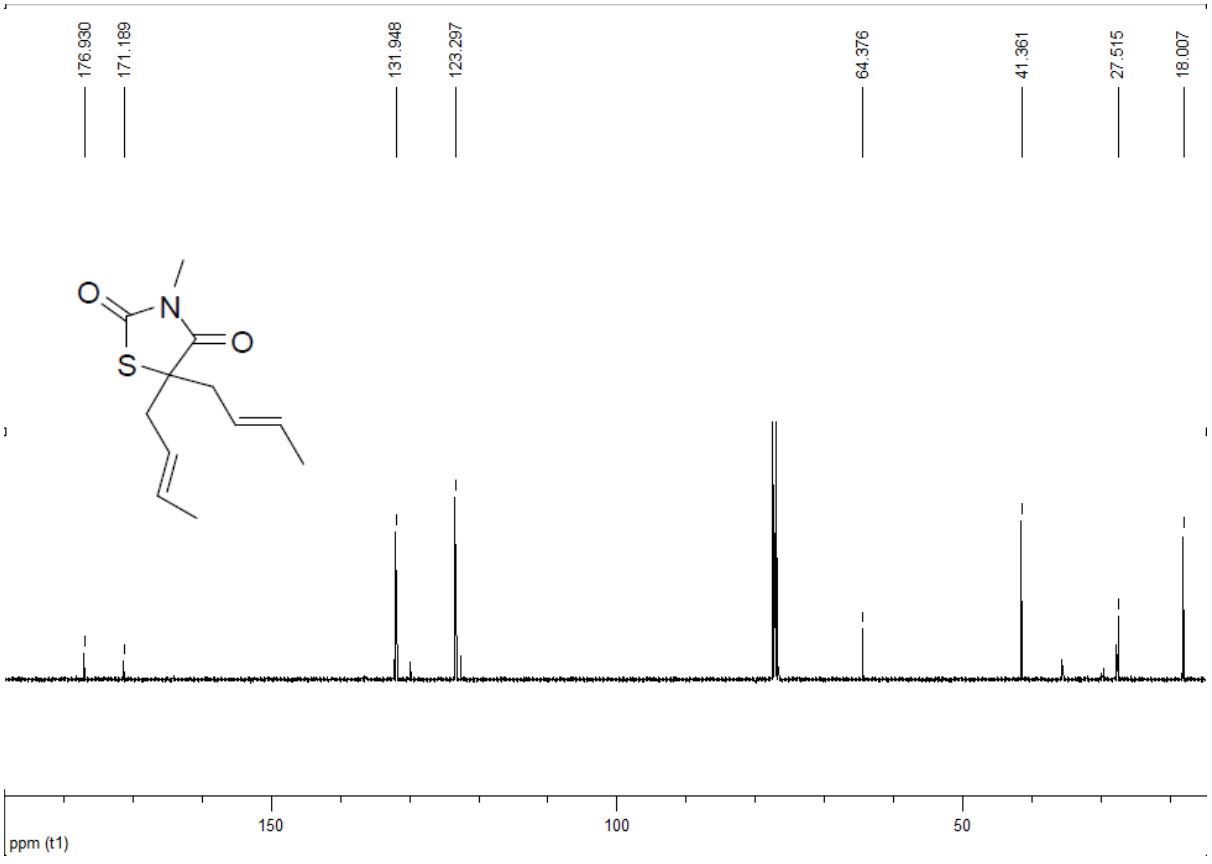
^{1}H and ^{13}C NMR for 8a:





^{1}H and ^{13}C NMR for 7c:





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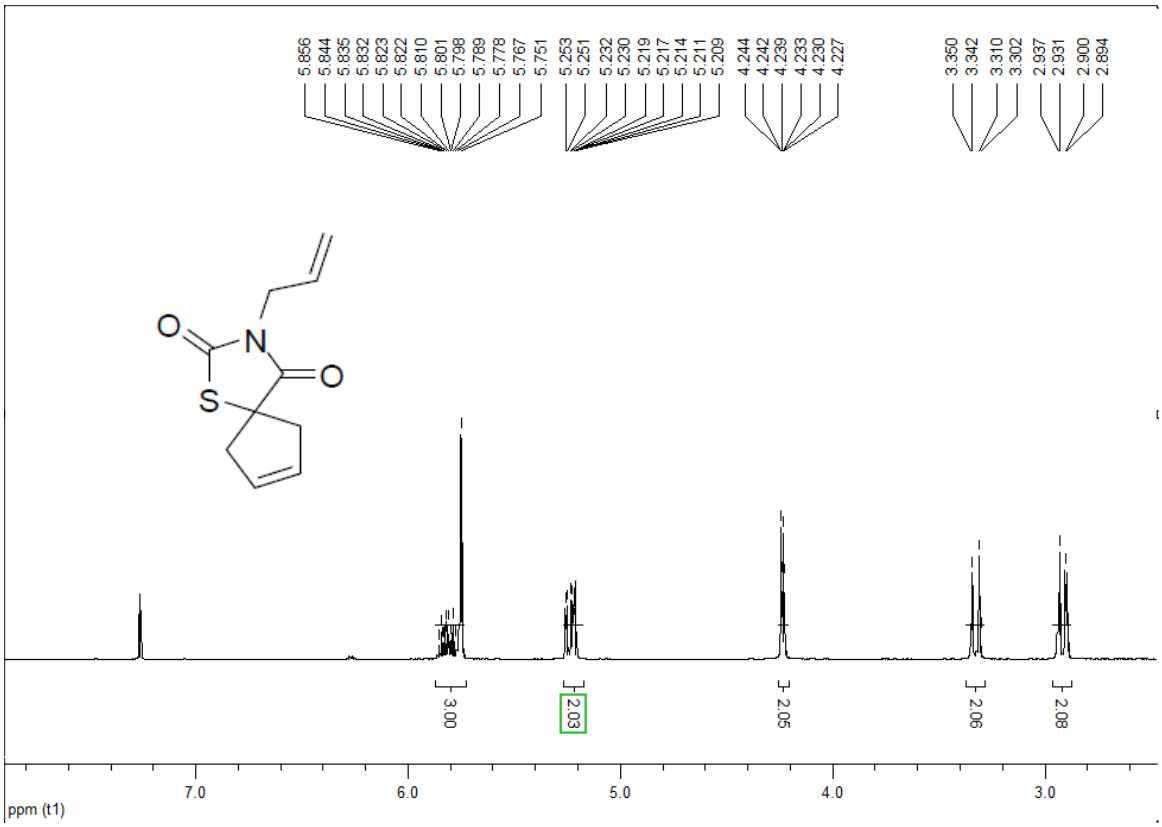
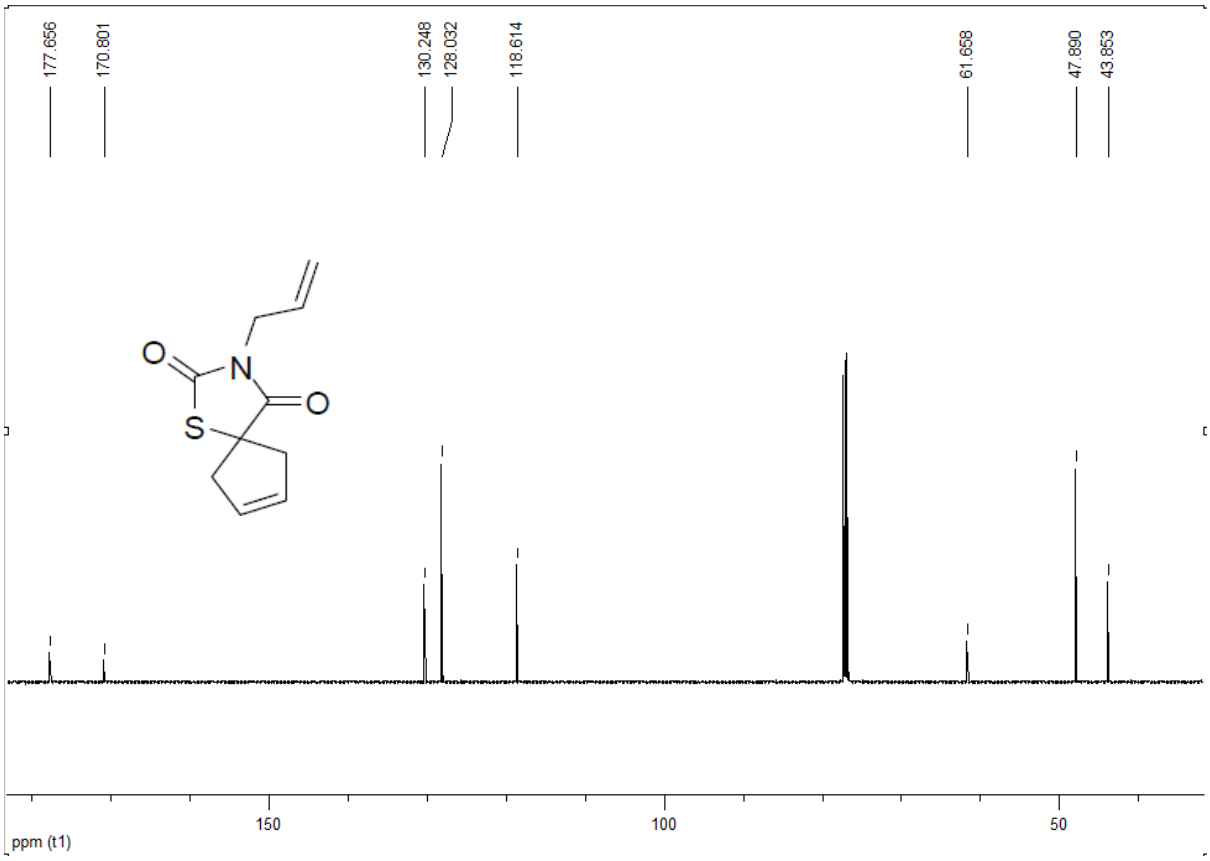
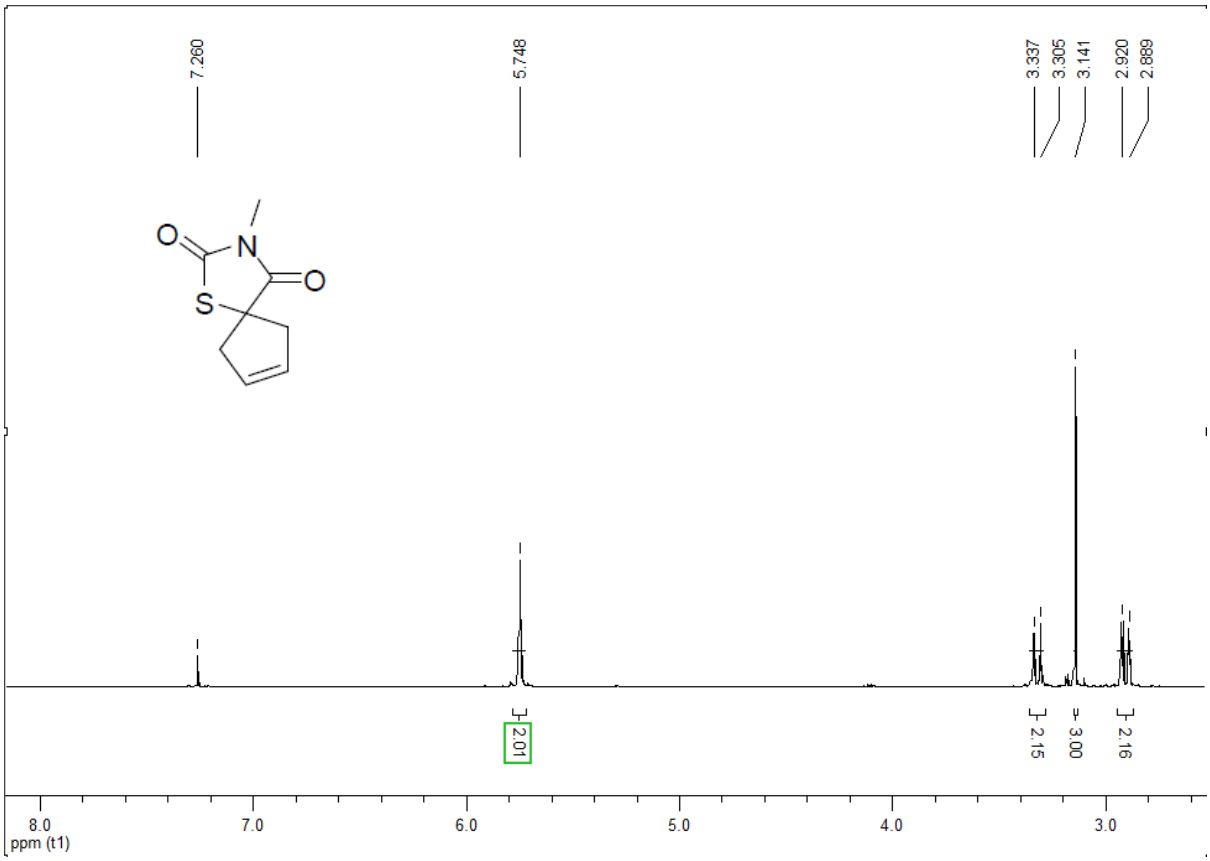
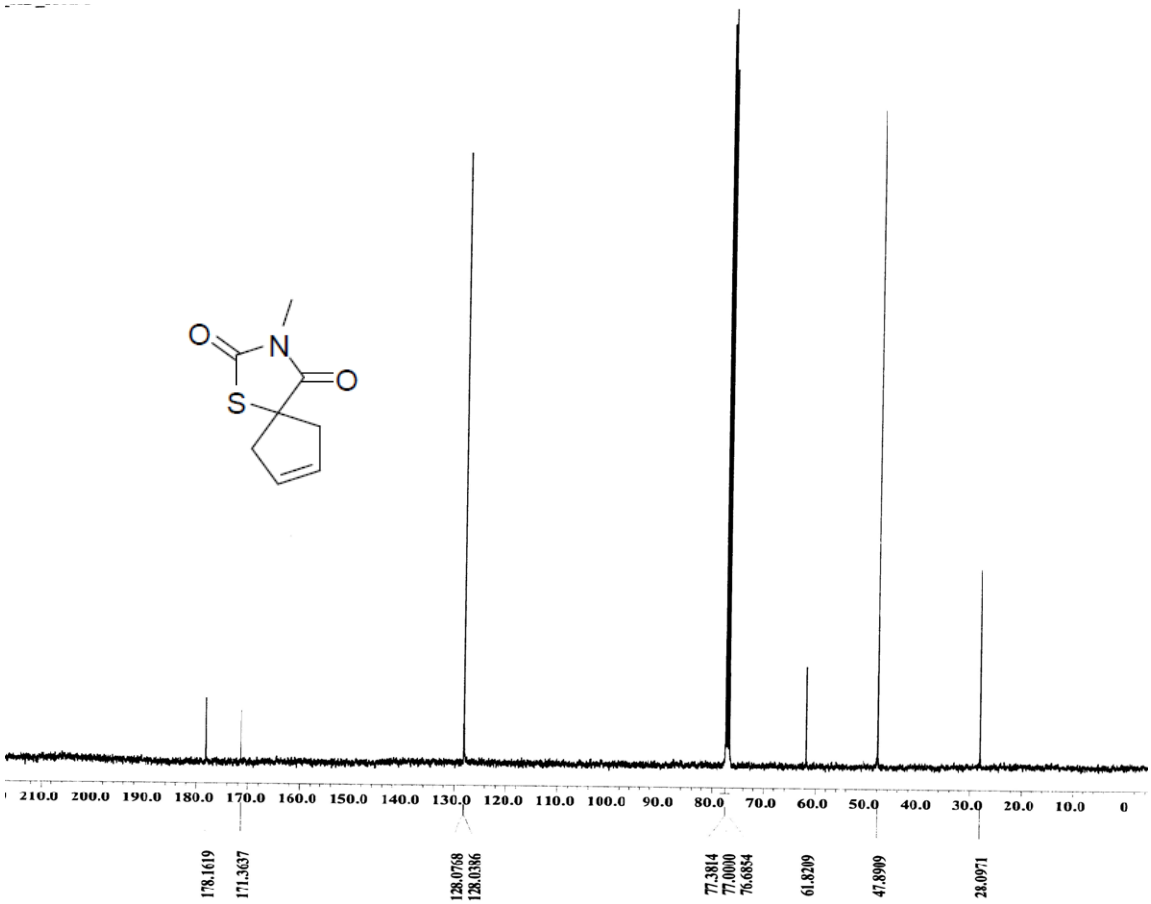




^{1}H and ^{13}C NMR for 9a:





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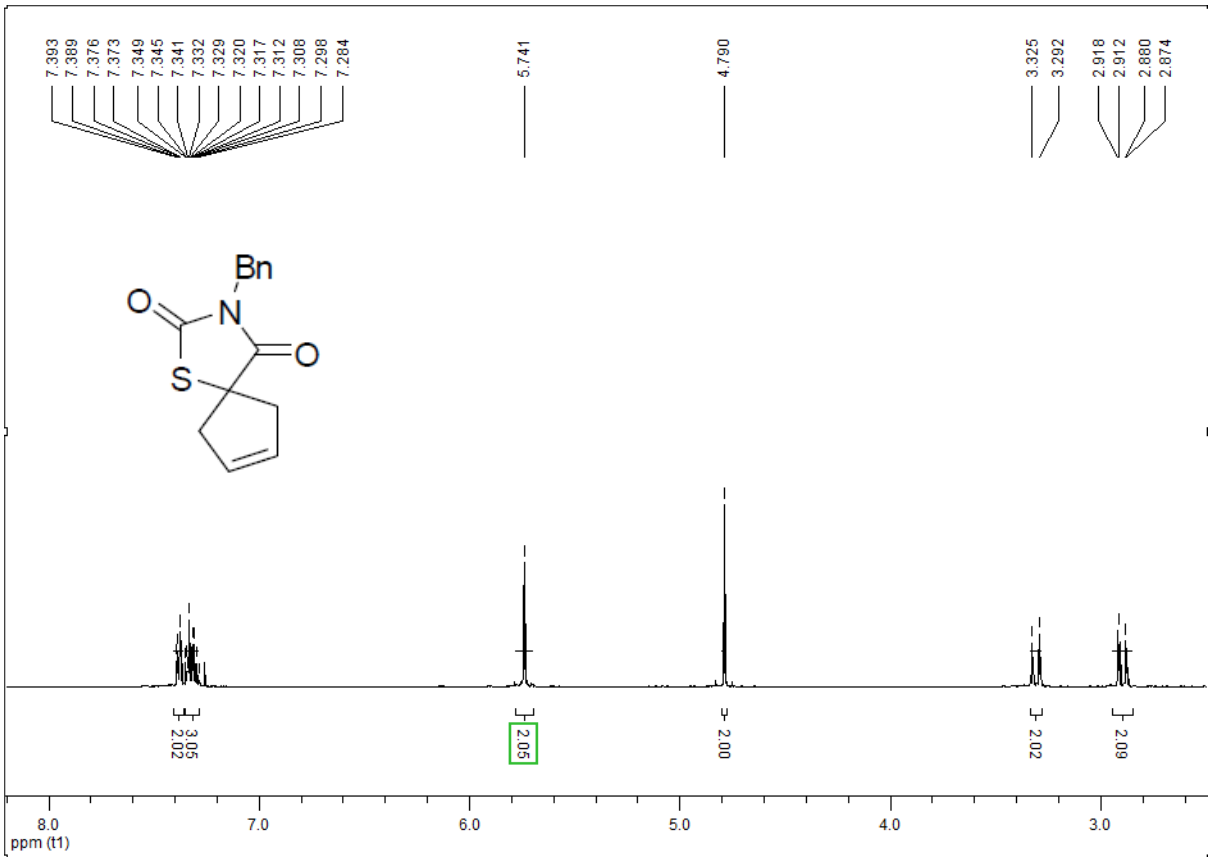
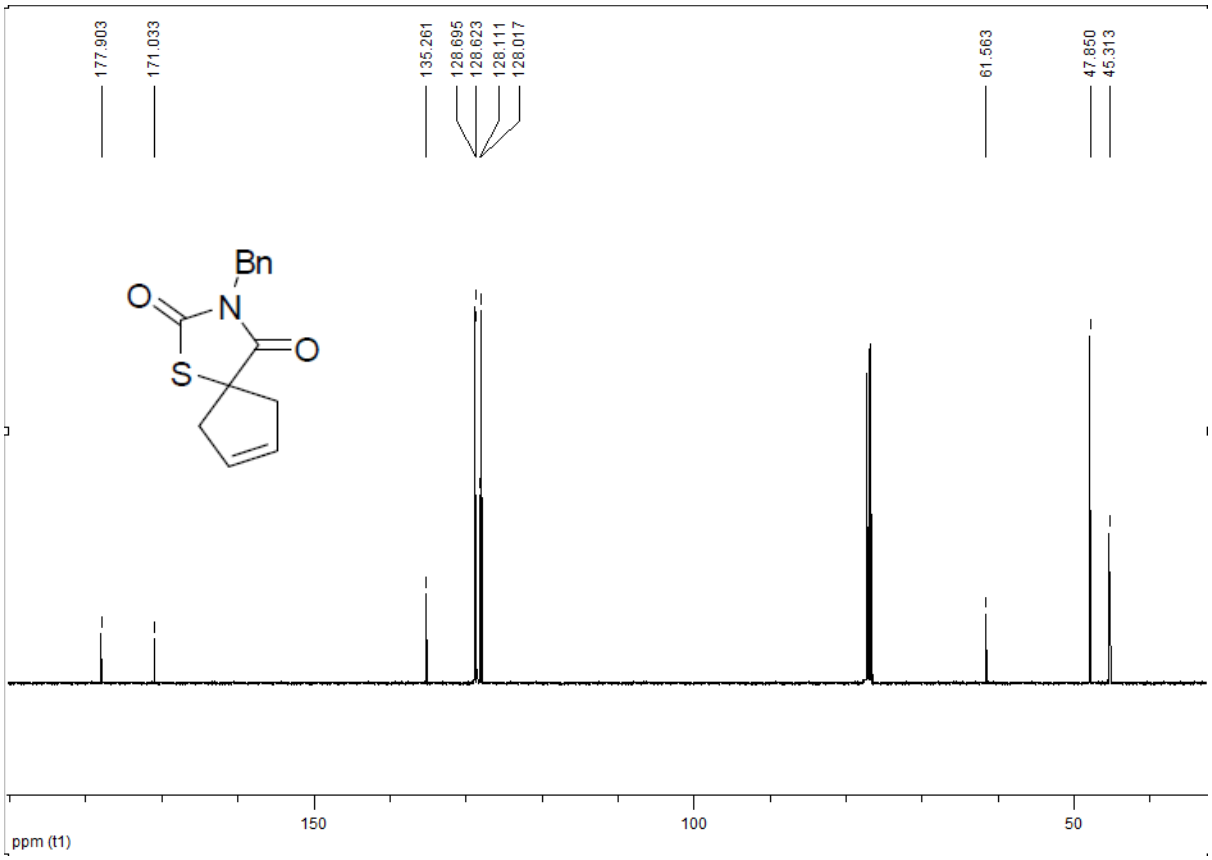
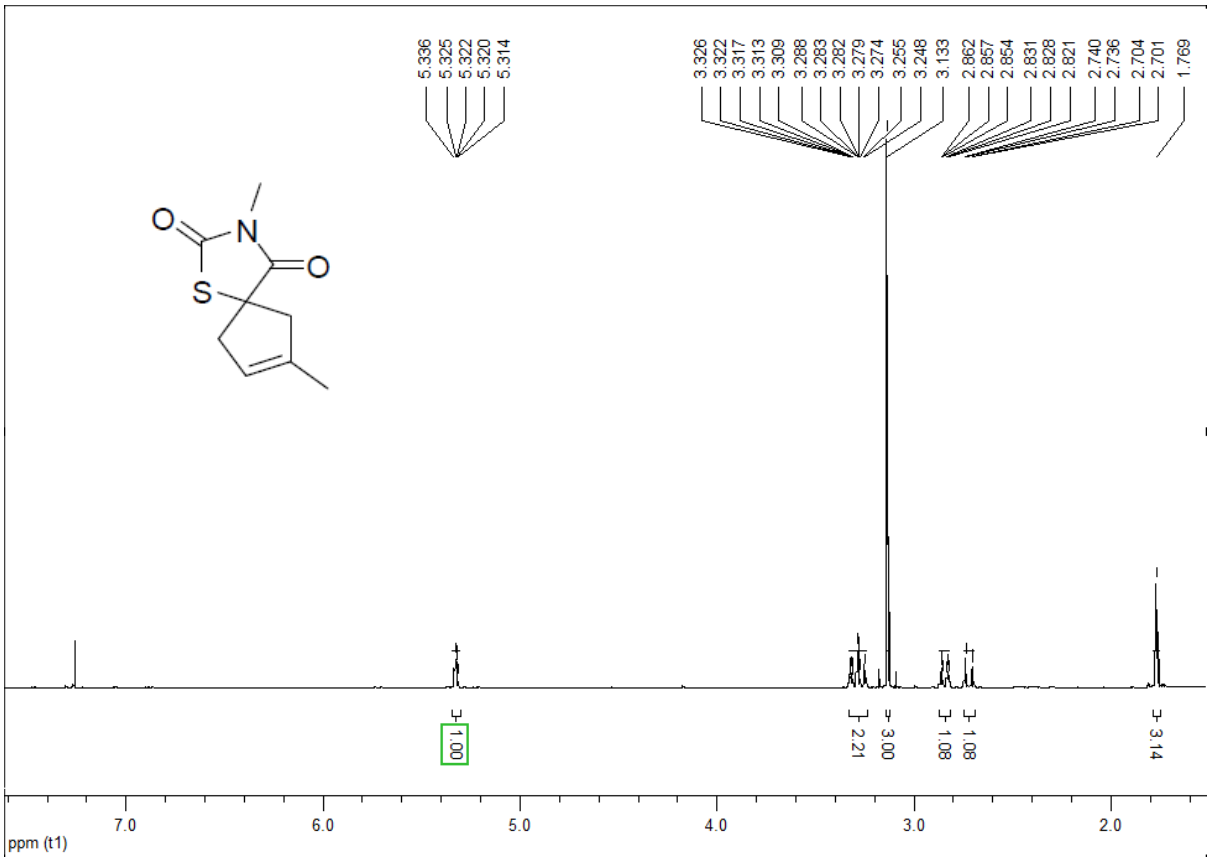
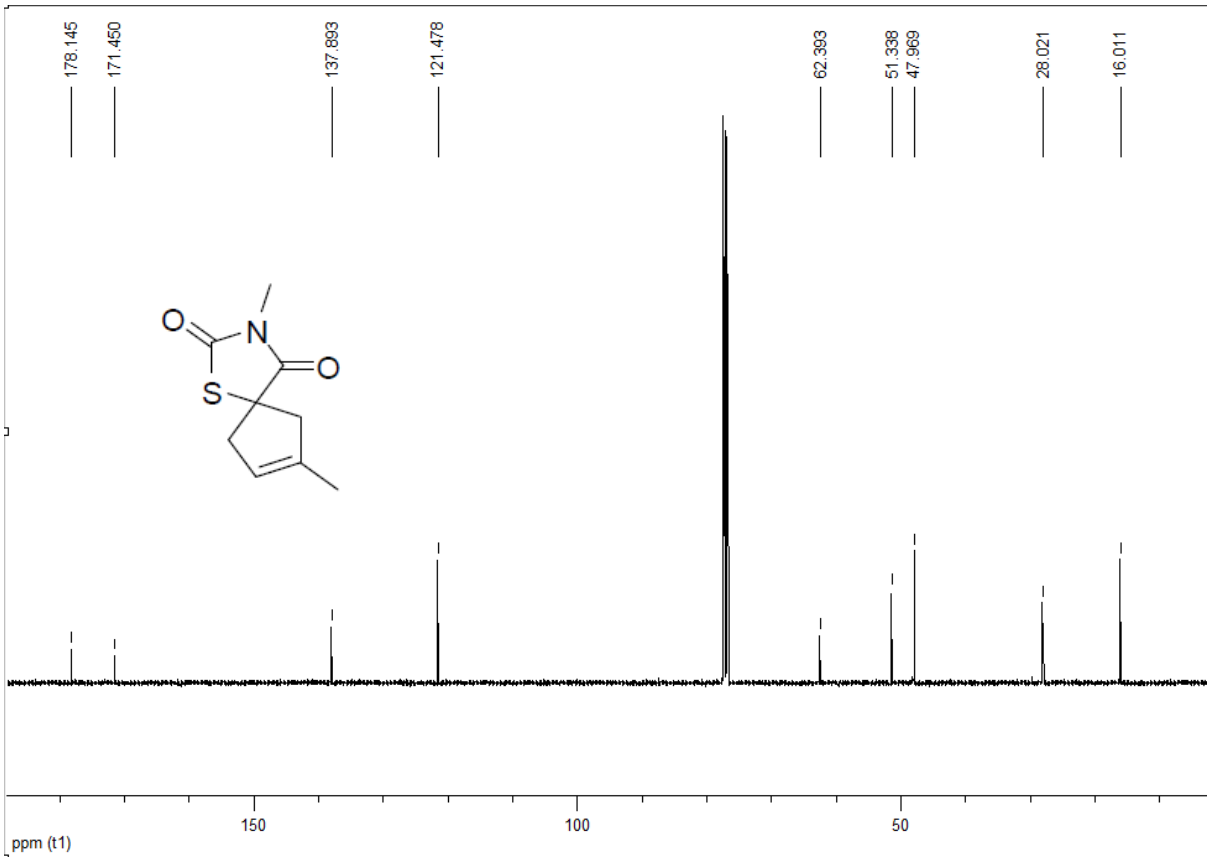


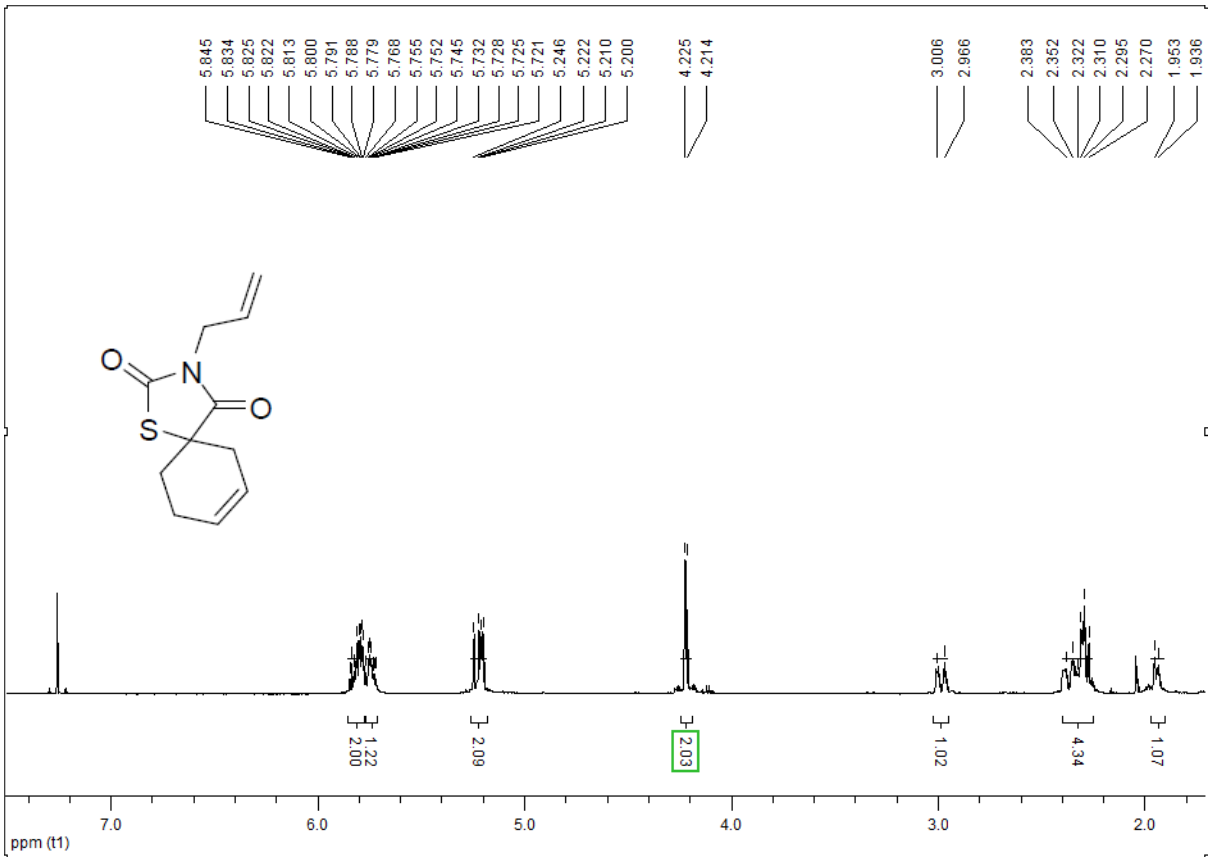


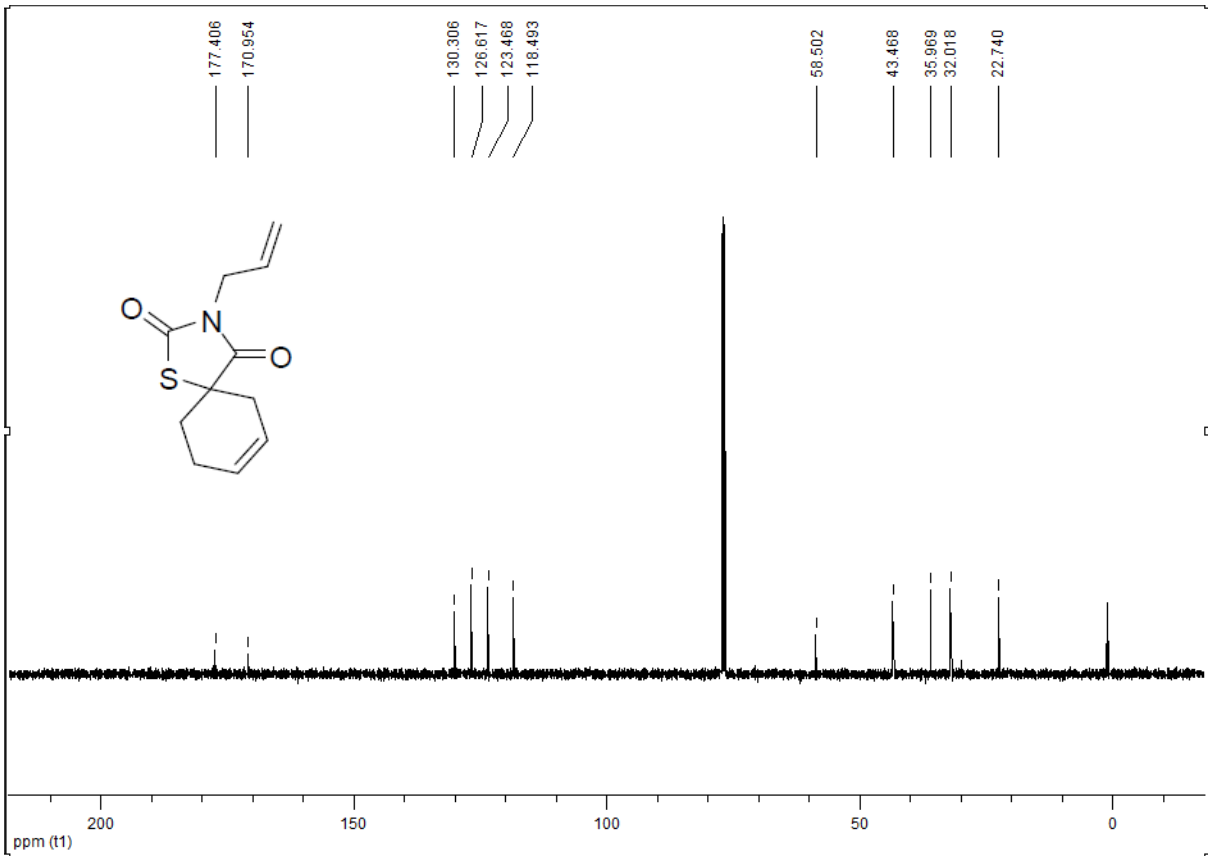
^{1}H and ^{13}C NMR for 9c:



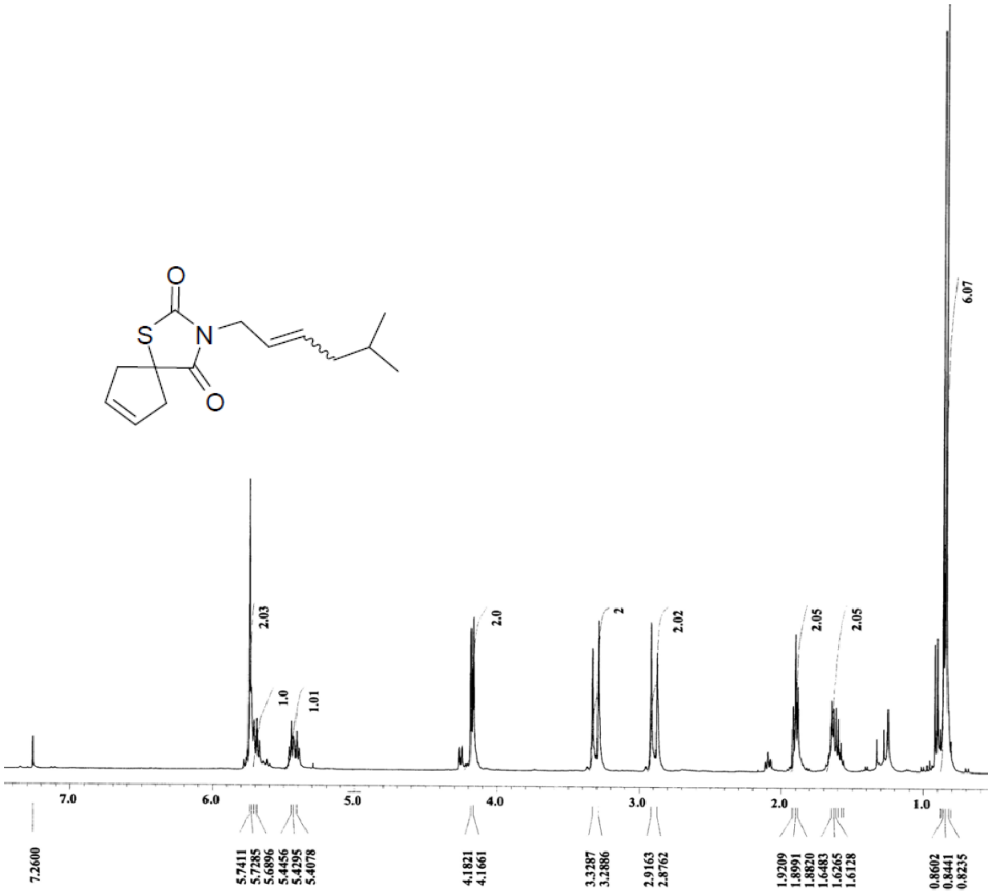


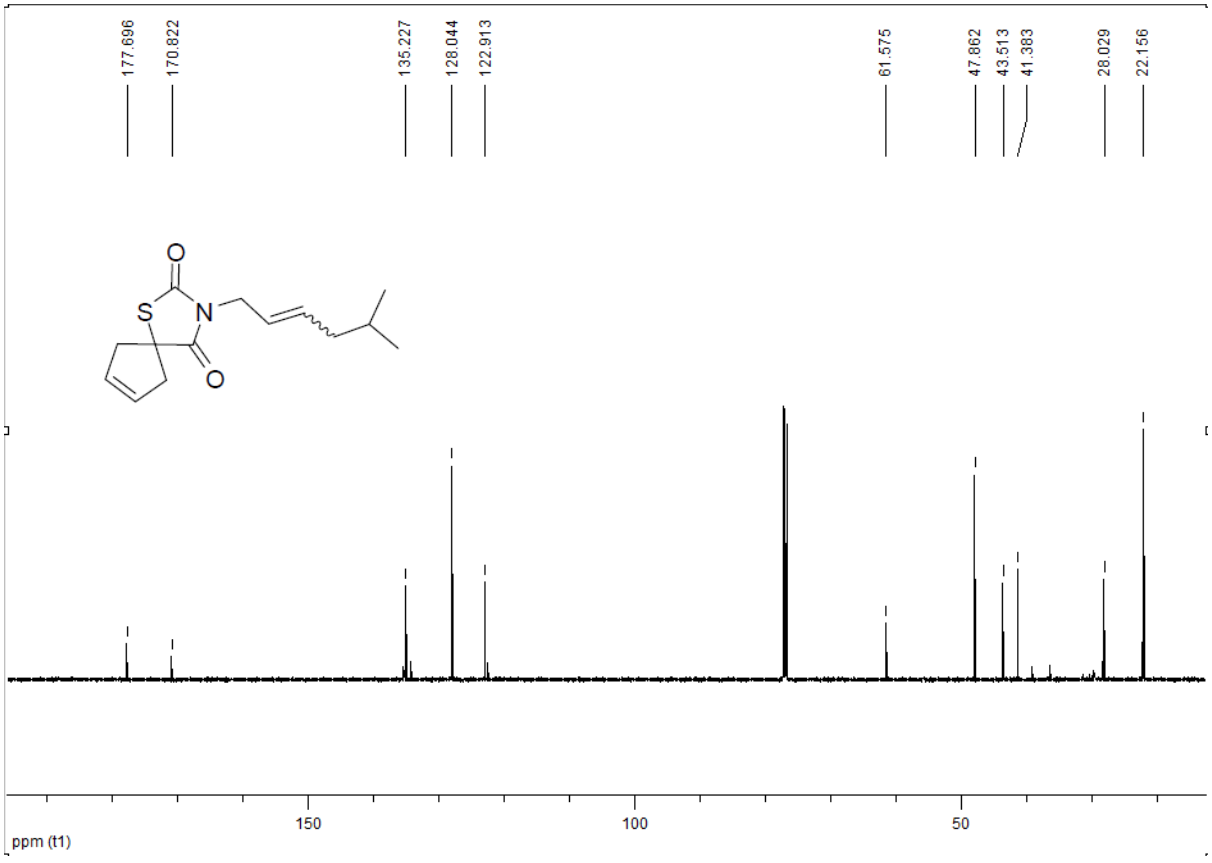
^{1}H and ^{13}C NMR for 9d:



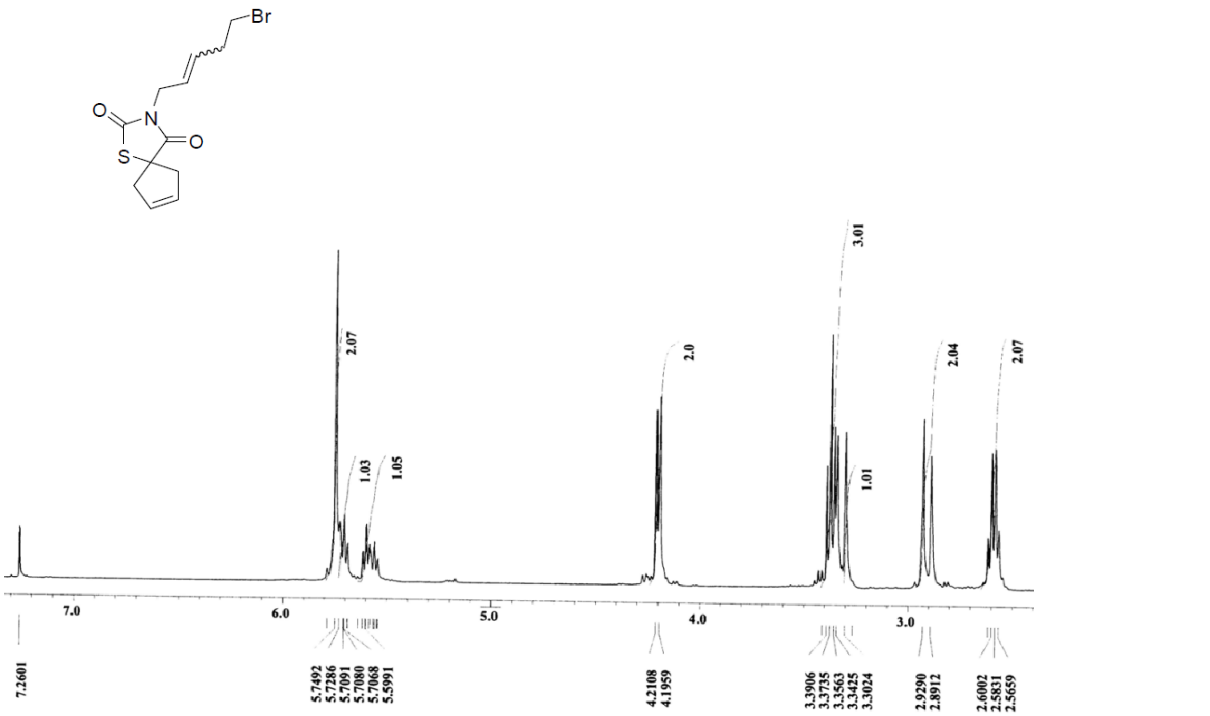


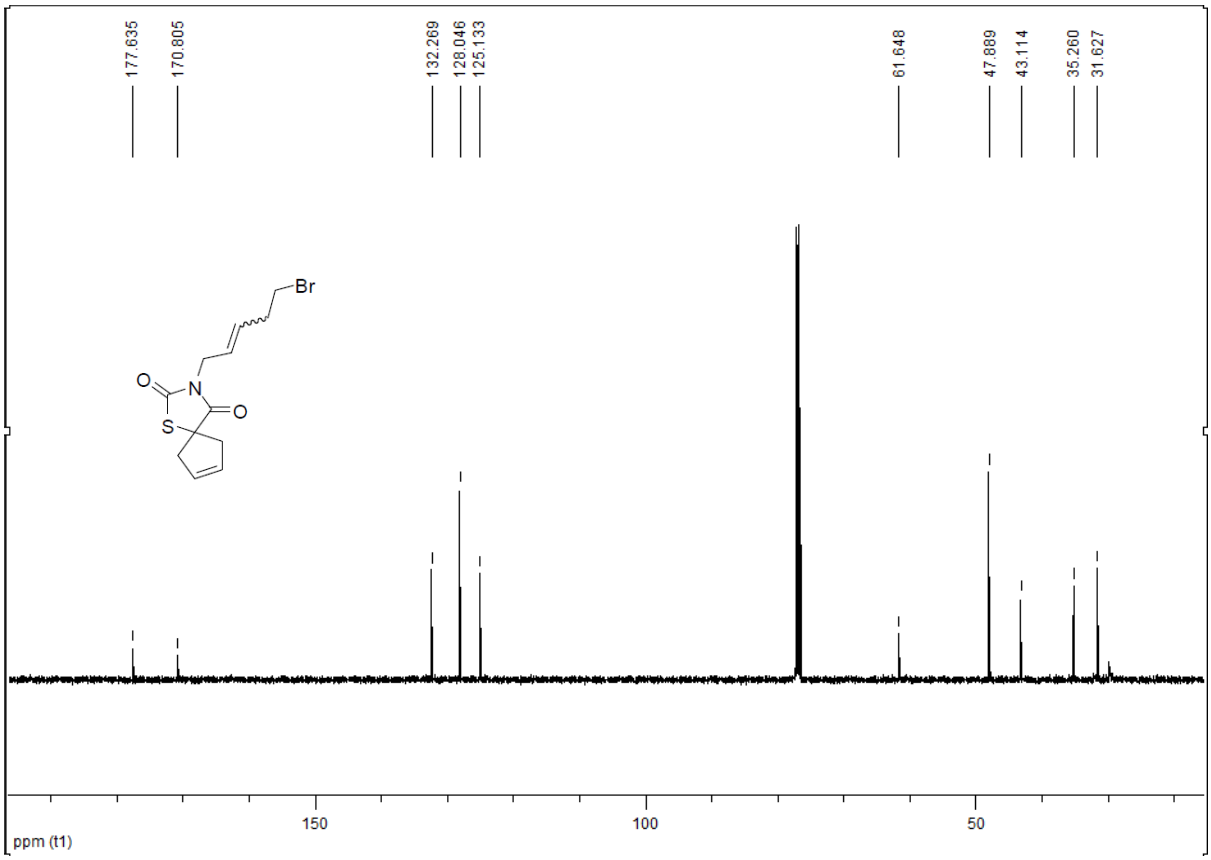
^{1}H and ^{13}C NMR for 13a:





^{1}H and ^{13}C NMR for 13b:





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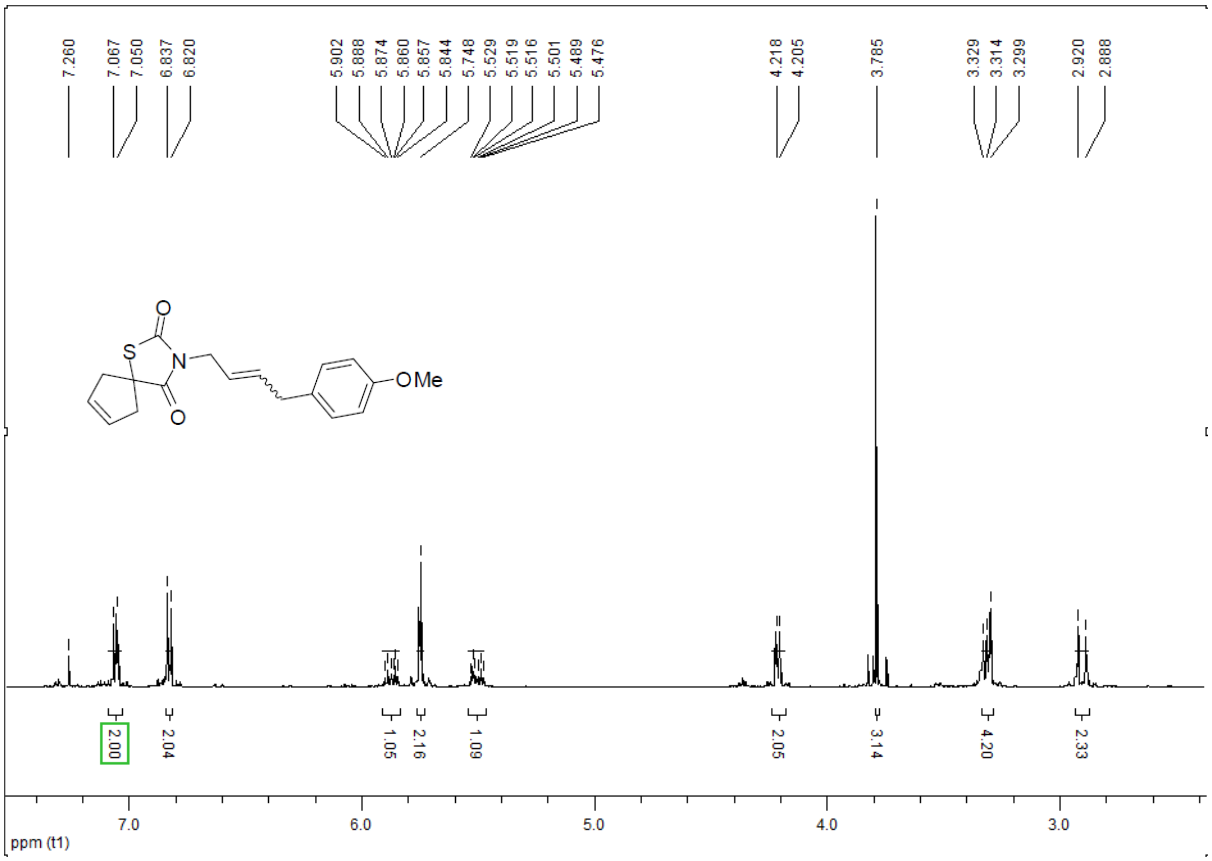
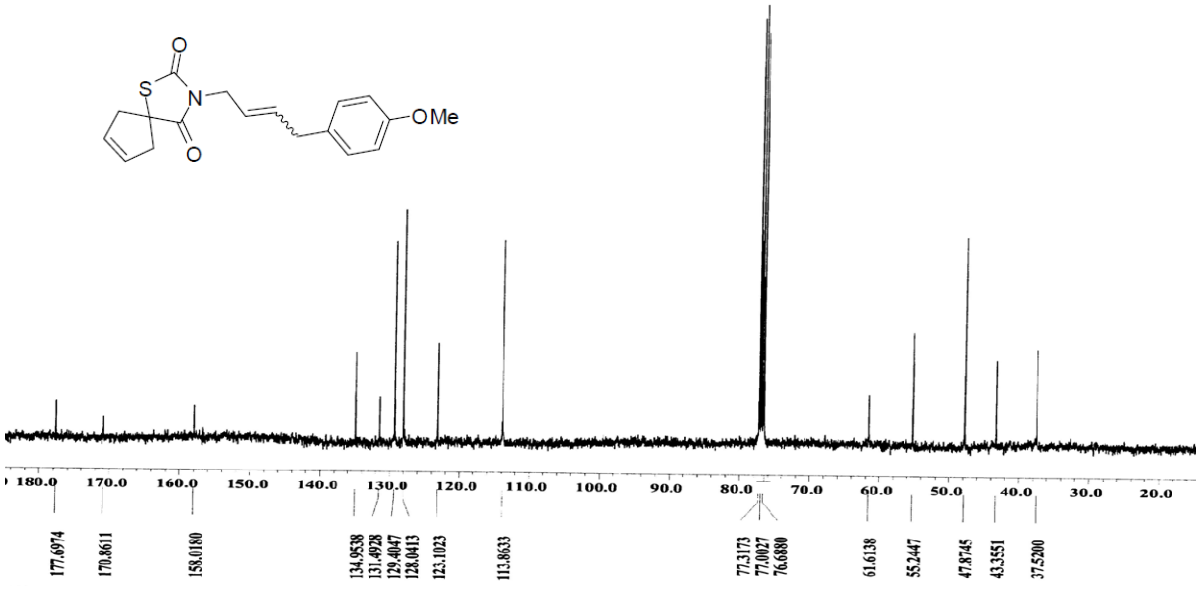
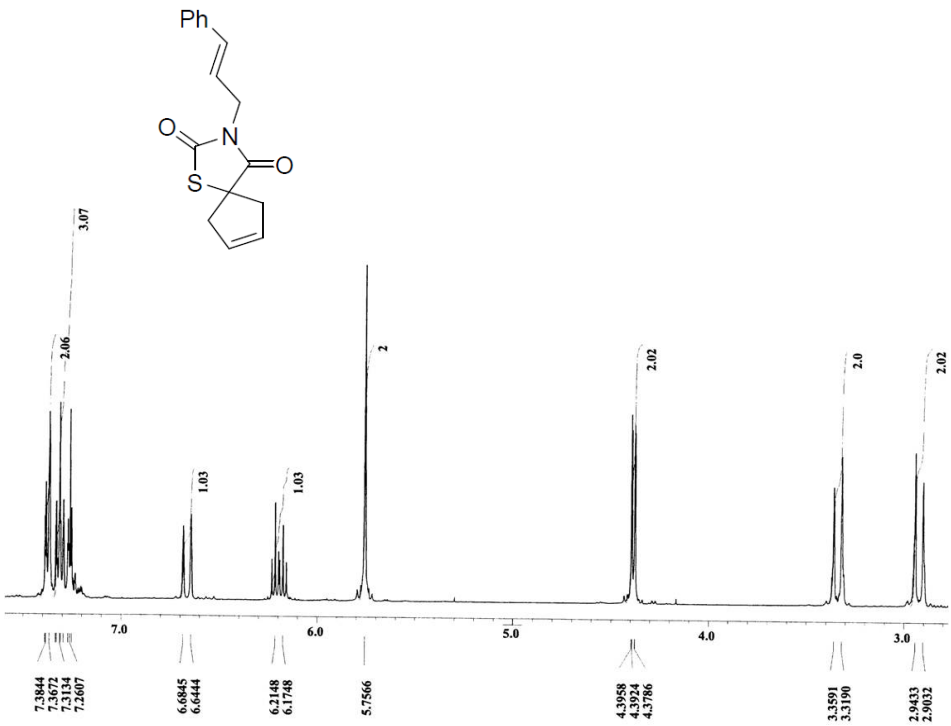
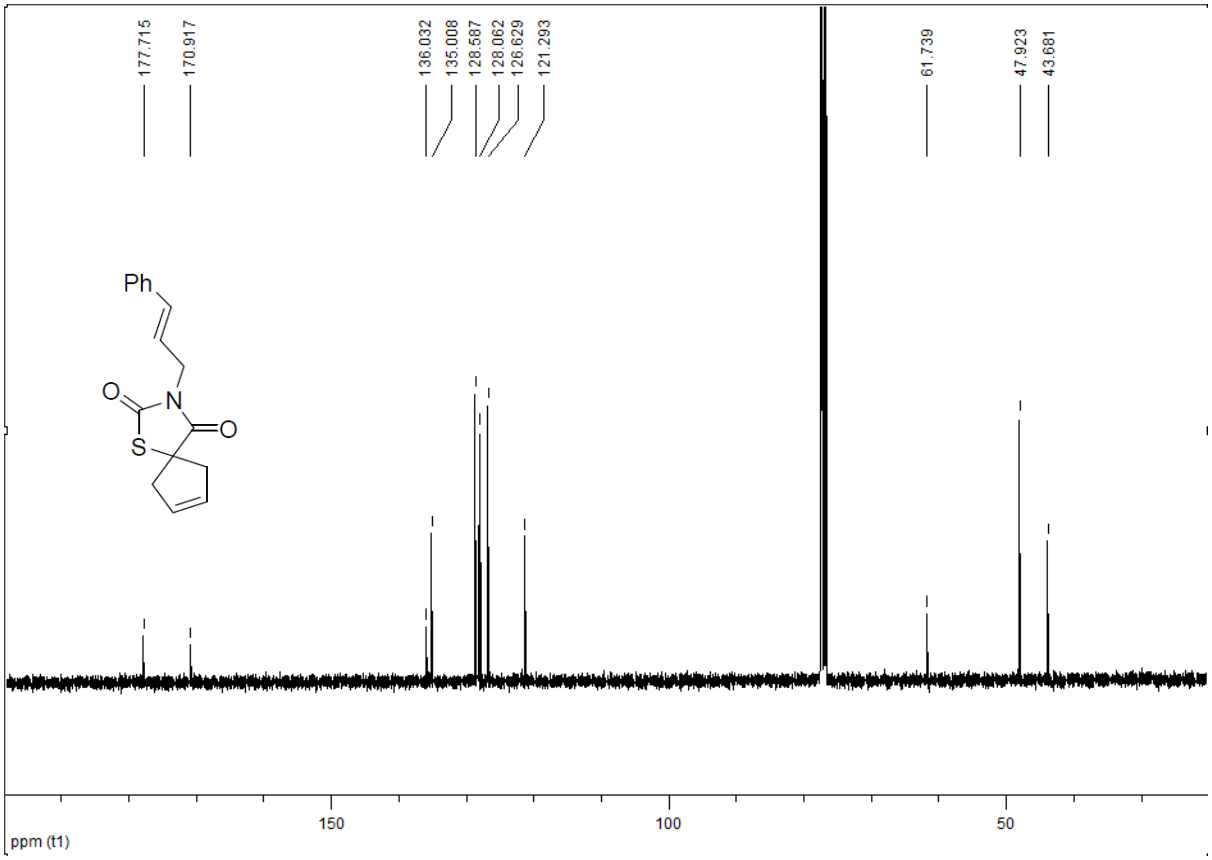




^{1}H and ^{13}C NMR for 13d:





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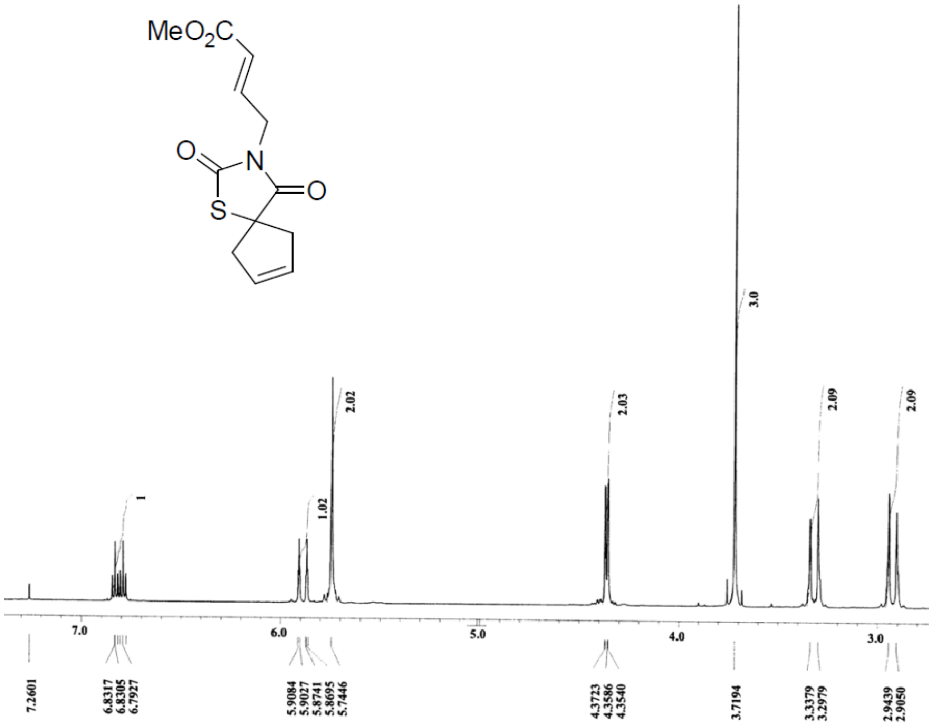
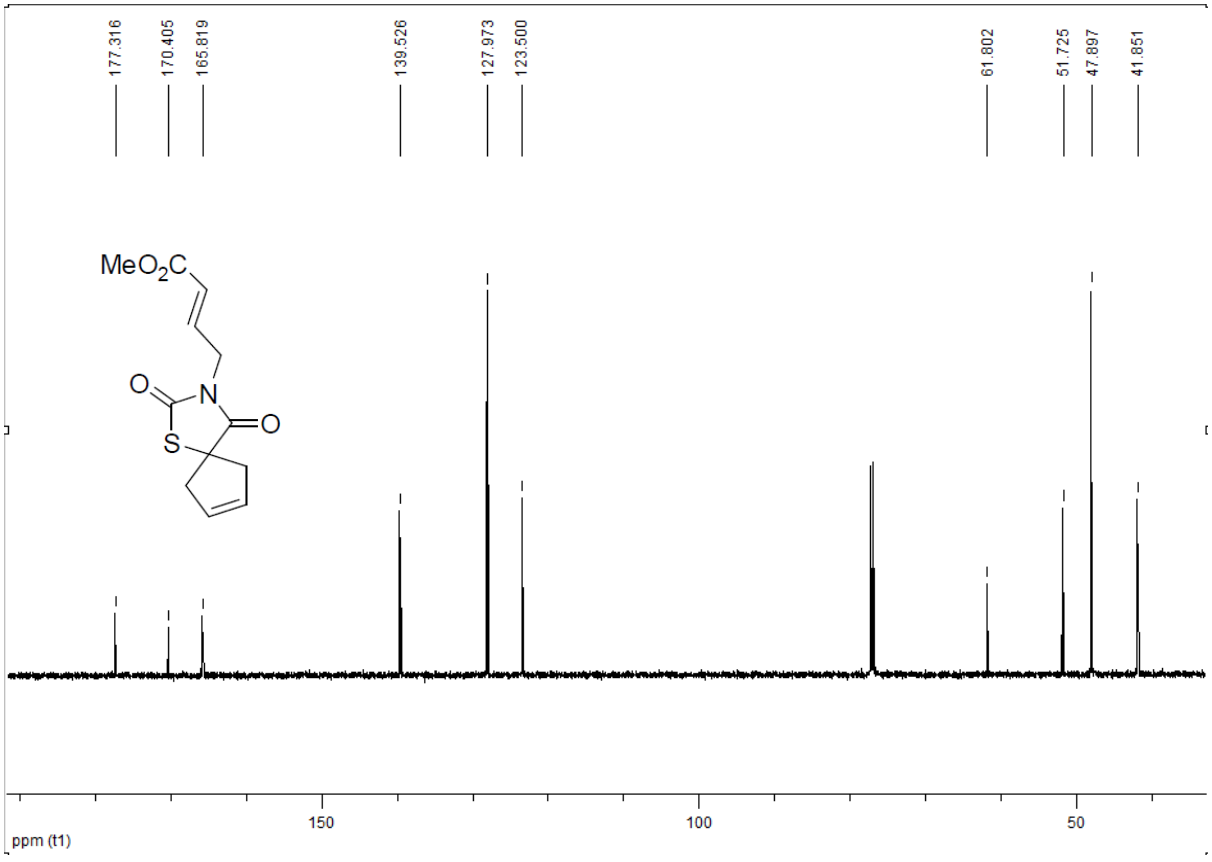
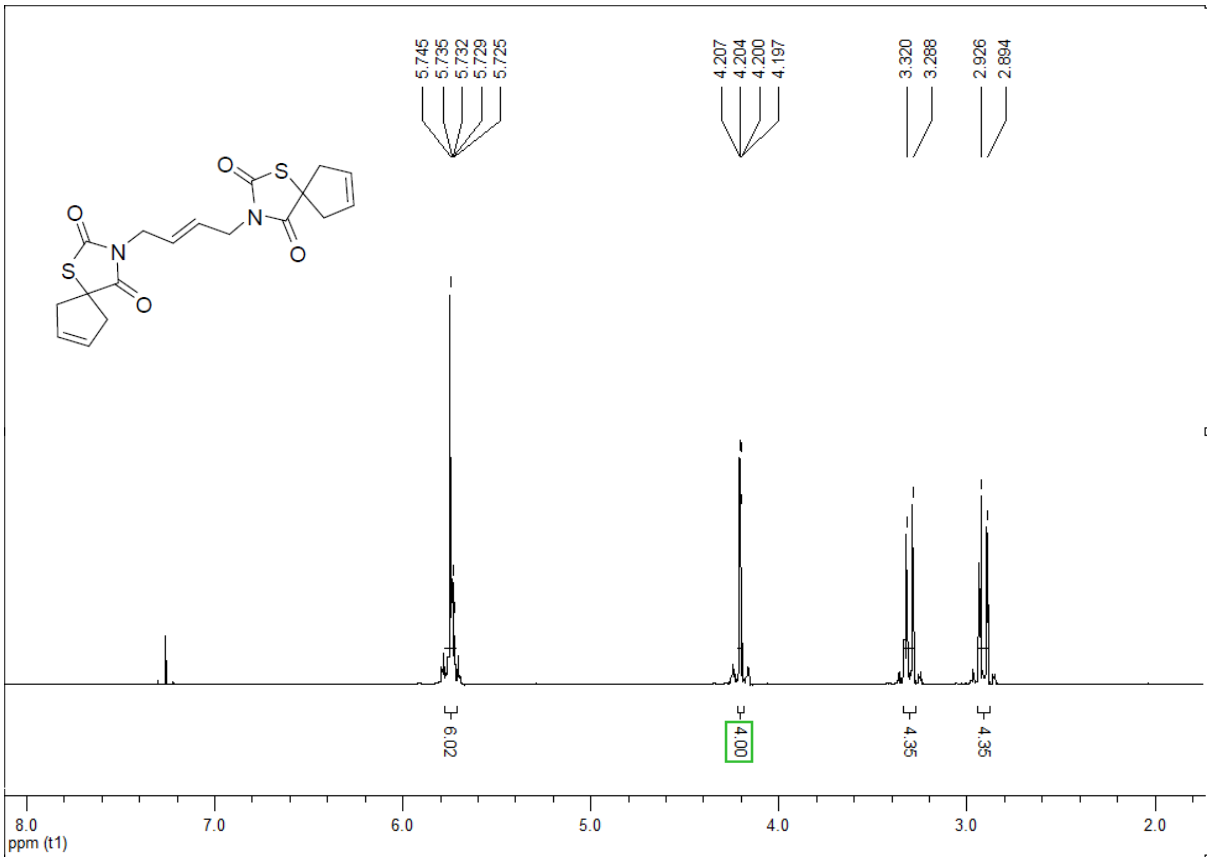
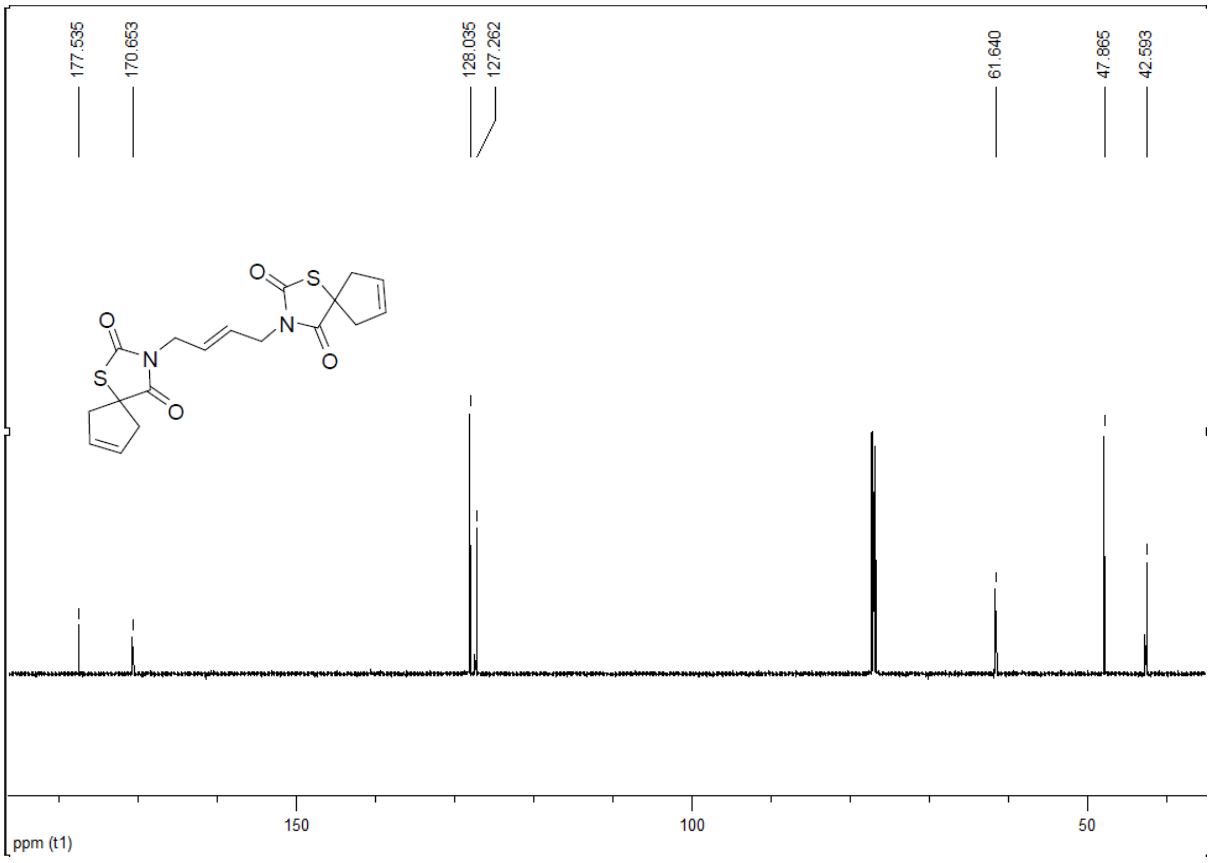




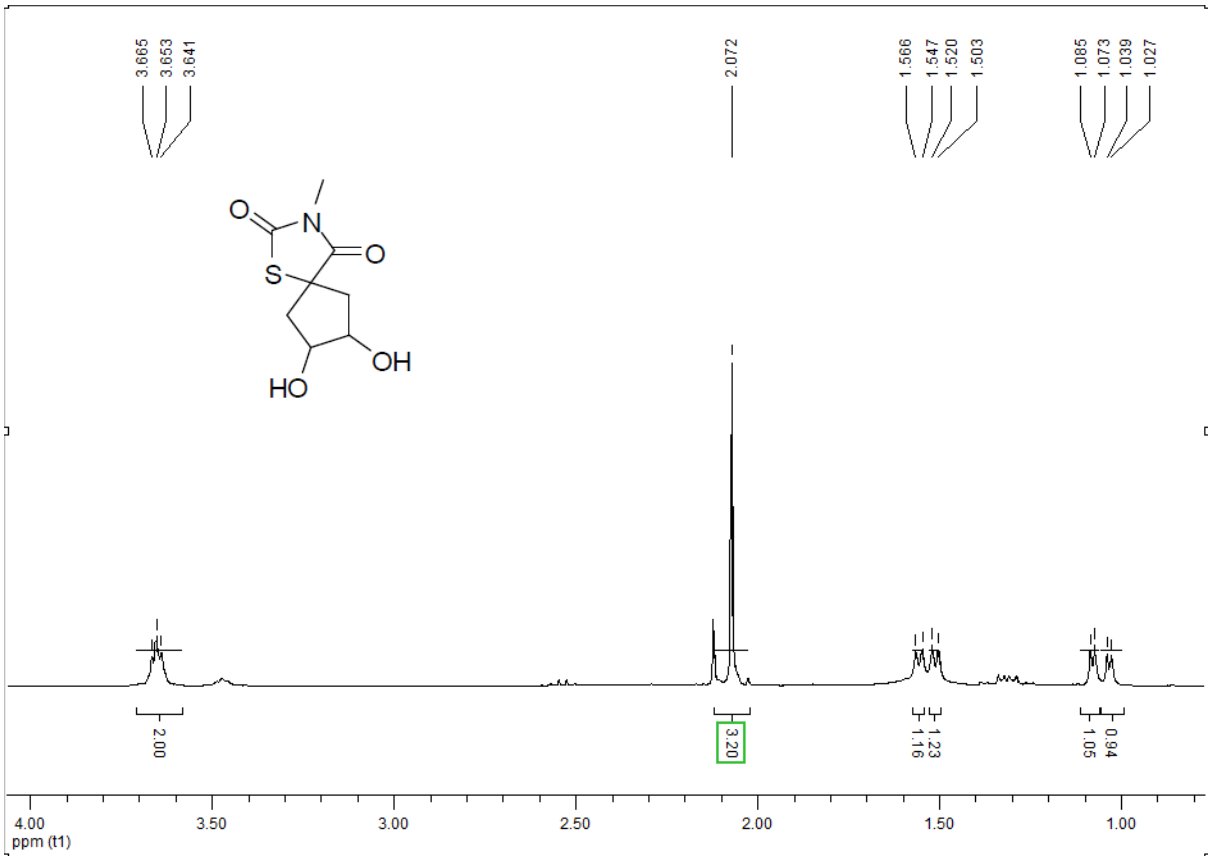
^{1}H and ^{13}C NMR for 4:

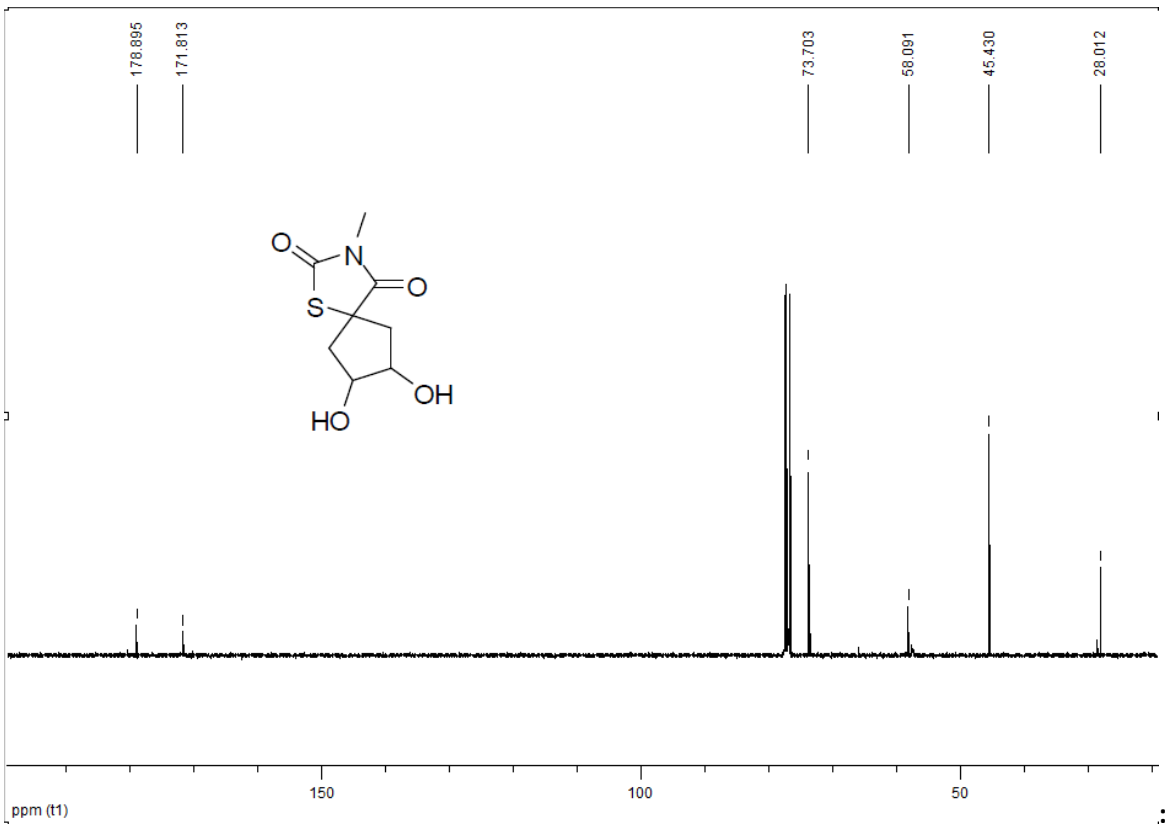




^{1}H and ^{13}C NMR for

10:





^{1}H and ^{13}C NMR for 11: