

Supporting information for

**Spirocyclizations involving oxonium ylides
derived from cyclic α -diazocarbonyl compounds:
an entry into 6-oxa-2-azaspiro[4.5]decane scaffold**

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Crystallographic data for compounds **2c, **2k**, **2r**, **2w**, (*S/R,S/R*)-**6v**^{maj} and (*R/S,S/R*)-**6v**^{min}**

X-ray Single Crystal analyses were performed on Agilent Technologies «Xcalibur» and «Supernova» diffractometers with monochromated MoK α or CuK α radiation, respectively. Crystal growth was performed by slow evaporation of solution in MeOH/DCM mixture (2:1) at 5 °C. Crystals were measuring at the temperature of 100 K The structures has been solved by the Superflip¹ and ShelXS² structure solution programs using Charge Flipping and Direct Methods, respectively, and refined with the ShelXL³ refinement incorporated in the OLEX2 program package⁴. CCDC # 2032237 (**2c**), CCDC # 2032235 (**2k**), CCDC # 2032236 (**2r**), CCDC # 2032234 (**2w**), CCDC # 2032238 ((*S/R,S/R*)-**6v**^{maj}) and CCDC # 2027755 ((*R/S,S/R*)-**6v**^{min}) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via <http://www.ccdc.cam.ac.uk>.

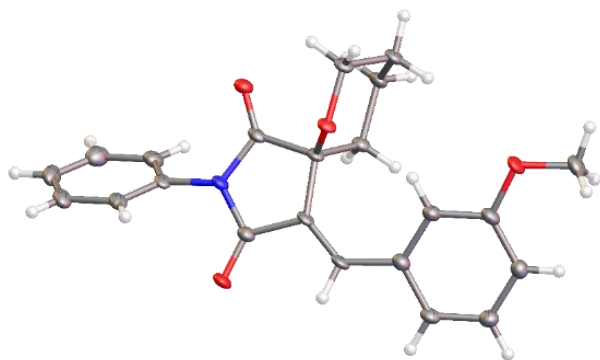


Figure S1. ORTEP representation of compound **2c** (thermal ellipsoids are shown at 50% probability).

Table S1. Crystal data and structure refinement for 2c	
Empirical formula	C ₂₂ H ₂₁ NO ₄
Formula weight	363.40
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	P2 ₁ /n
a/Å	16.2411(4)
b/Å	5.5813(2)
c/Å	20.3860(5)
α /°	90
β /°	106.766(3)
γ /°	90
Volume/Å ³	1769.36(9)
Z	4
ρ_{calc} /cm ³	1.364
μ /mm ⁻¹	0.764
F(000)	768.0
Crystal size/mm ³	0.12 × 0.09 × 0.04
Radiation	Cu K α (λ = 1.54184)
2 θ range for data collection/°	6.162 to 141.092
Index ranges	-19 ≤ h ≤ 19, -6 ≤ k ≤ 6, -24 ≤ l ≤ 24
Reflections collected	11704
Independent reflections	3366 [Rint = 0.0482, Rsigma = 0.0459]
Data/restraints/parameters	3366/0/245
Goodness-of-fit on F ²	1.056
Final R indexes [I ≥ 2 σ (I)]	R1 = 0.0596, wR2 = 0.1552
Final R indexes [all data]	R1 = 0.0715, wR2 = 0.1662
Largest diff. peak/hole / e Å ⁻³	0.65/-0.40
CCDC 2032237	

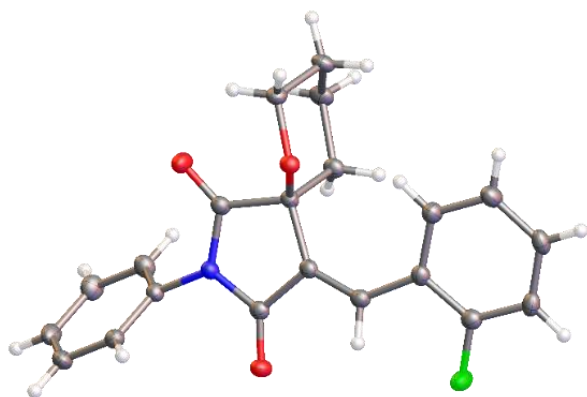


Figure S2. ORTEP representation of compound **2k** (thermal ellipsoids are shown at 50% probability).

Table S2. Crystal data and structure refinement for 2k	
Empirical formula	C ₂₁ H ₁₈ NO ₃ F
Formula weight	351.36
Temperature/K	100.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	8.86500(10)
b/Å	9.2249(2)
c/Å	11.4276(2)
α /°	109.772(2)
β /°	94.3680(10)
γ /°	104.3340(10)
Volume/Å ³	838.85(3)
Z	2
ρ_{calc} /cm ³	1.391
μ /mm ⁻¹	0.830
F(000)	368.0
Crystal size/mm ³	0.18 × 0.1 × 0.05
Radiation	Cu K α (λ = 1.54184)
2 θ range for data collection/°	8.352 to 154.506
Index ranges	-10 ≤ h ≤ 11, -11 ≤ k ≤ 11, -14 ≤ l ≤ 14
Reflections collected	28581
Independent reflections	3493 [Rint = 0.1138, Rsigma = 0.0470]
Data/restraints/parameters	3493/0/235
Goodness-of-fit on F ²	1.100
Final R indexes [I ≥ 2 σ (I)]	R1 = 0.0471, wR2 = 0.1350
Final R indexes [all data]	R1 = 0.0500, wR2 = 0.1380
Largest diff. peak/hole / e Å ⁻³	0.33/-0.29
CCDC 2032235	

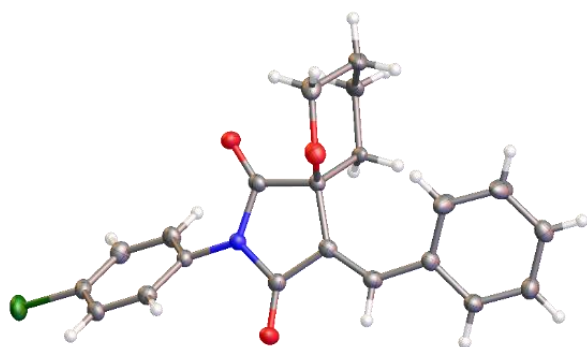


Figure S3. ORTEP representation of compound **2r** (thermal ellipsoids are shown at 50% probability).

Table S3. Crystal data and structure refinement for 2r	
Empirical formula	C ₂₁ H ₁₈ ClNO ₃
Formula weight	367.81
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	P21/n
a/Å	5.62620(10)
b/Å	12.0230(3)
c/Å	25.1409(6)
α /°	90
β /°	92.578(2)
γ /°	90
Volume/Å ³	1698.91(7)
Z	4
ρ_{calc} /cm ³	1.438
μ /mm ⁻¹	2.172
F(000)	768.0
Crystal size/mm ³	0.11 × 0.08 × 0.06
Radiation	Cu K α (λ = 1.54184)
2 θ range for data collection/°	7.04 to 154.22
Index ranges	-7 ≤ h ≤ 4, -15 ≤ k ≤ 15, -31 ≤ l ≤ 31
Reflections collected	22343
Independent reflections	3530 [Rint = 0.0487, Rsigma = 0.0256]
Data/restraints/parameters	3530/0/235
Goodness-of-fit on F ²	1.170
Final R indexes [I ≥ 2 σ (I)]	R1 = 0.0552, wR2 = 0.1234
Final R indexes [all data]	R1 = 0.0572, wR2 = 0.1243
Largest diff. peak/hole / e Å ⁻³	0.47/-0.33
CCDC 2032236	

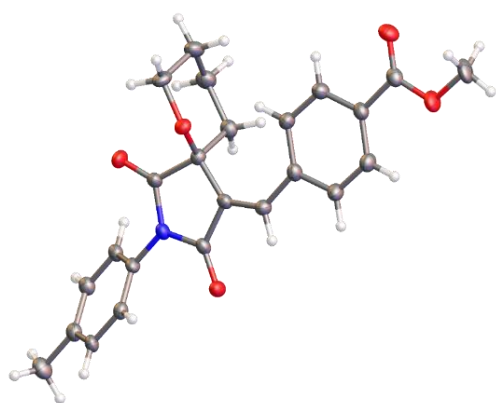


Figure S4. ORTEP representation of compound **2w** (thermal ellipsoids are shown at 50% probability).

Table S4. Crystal data and structure refinement for 2w	
Empirical formula	C ₂₄ H ₂₃ NO ₅
Formula weight	405.43
Temperature/K	100.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	12.0824(3)
b/Å	13.2365(3)
c/Å	14.8059(3)
α /°	64.555(2)
β /°	71.958(2)
γ /°	74.726(2)
Volume/Å ³	2009.49(9)
Z	4
ρ_{calc} /cm ³	1.340
μ /mm ⁻¹	0.769
F(000)	856.0
Crystal size/mm ³	0.9 × 0.6 × 0.4
Radiation	Cu K α (λ = 1.54184)
2 θ range for data collection/°	6.786 to 155.408
Index ranges	-15 ≤ h ≤ 15, -16 ≤ k ≤ 14, -18 ≤ l ≤ 18
Reflections collected	81542
Independent reflections	8372 [Rint = 0.0863, Rsigma = 0.0366]
Data/restraints/parameters	8372/0/545
Goodness-of-fit on F ²	1.021
Final R indexes [I ≥ 2 σ (I)]	R1 = 0.0471, wR2 = 0.1418
Final R indexes [all data]	R1 = 0.0871, wR2 = 0.1737
Largest diff. peak/hole / e Å ⁻³	0.23/-0.27
CCDC 2032234	

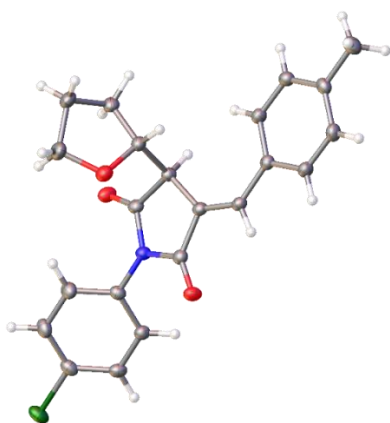


Figure S5. ORTEP representation of compound (*S/R,S/R*)-**6v**^{maj} (thermal ellipsoids are shown at 50% probability).

Table S5. Crystal data and structure refinement for (<i>S/R,S/R</i>)- 6v ^{maj}	
Empirical formula	C ₂₂ H ₂₀ ClNO ₃
Formula weight	381.84
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	P21/n
a/Å	8.7458(2)
b/Å	19.7502(4)
c/Å	11.0676(2)
α/°	90
β/°	104.068(2)
γ/°	90
Volume/Å ³	1854.38(7)
Z	4
ρ _{calc} /cm ³	1.368
μ/mm ⁻¹	2.009
F(000)	800.0
Crystal size/mm ³	0.14 × 0.12 × 0.05
Radiation	Cu Kα (λ = 1.54184)
2θ range for data collection/°	8.954 to 140.972
Index ranges	-10 ≤ h ≤ 10, -24 ≤ k ≤ 21, -13 ≤ l ≤ 13
Reflections collected	22048
Independent reflections	3556 [Rint = 0.0503, Rsigma = 0.0297]
Data/restraints/parameters	3556/0/245
Goodness-of-fit on F ²	1.050
Final R indexes [I ≥ 2σ (I)]	R1 = 0.0406, wR2 = 0.1018
Final R indexes [all data]	R1 = 0.0441, wR2 = 0.1042
Largest diff. peak/hole / e Å ⁻³	0.28/-0.46
CCDC 2032238	

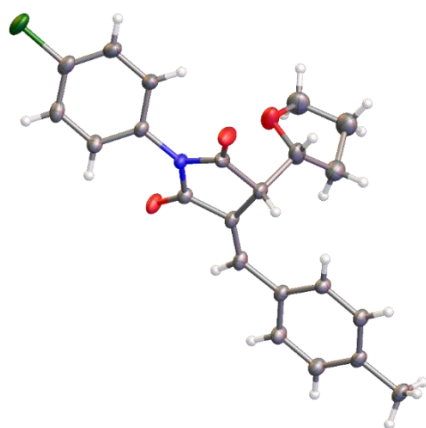


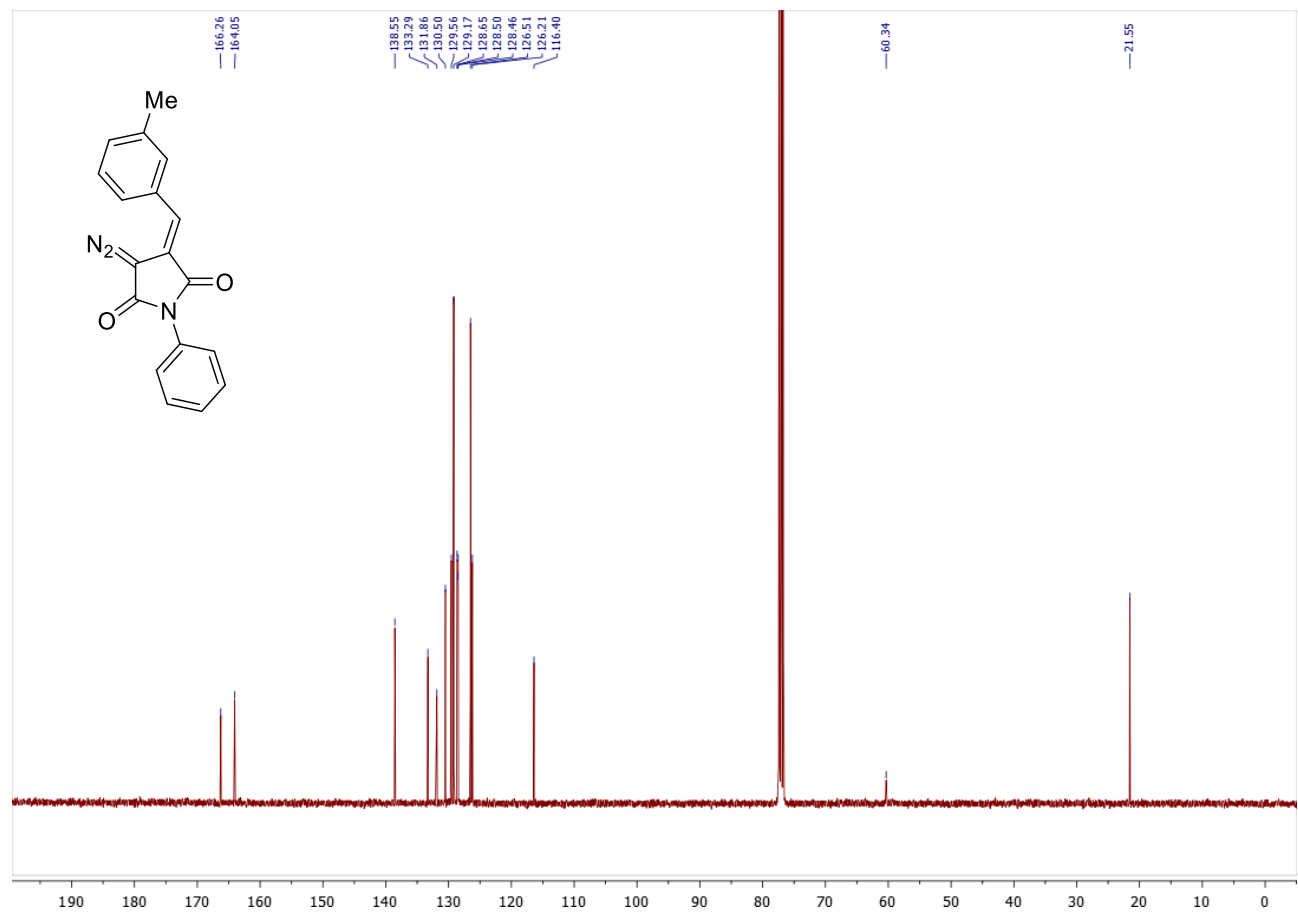
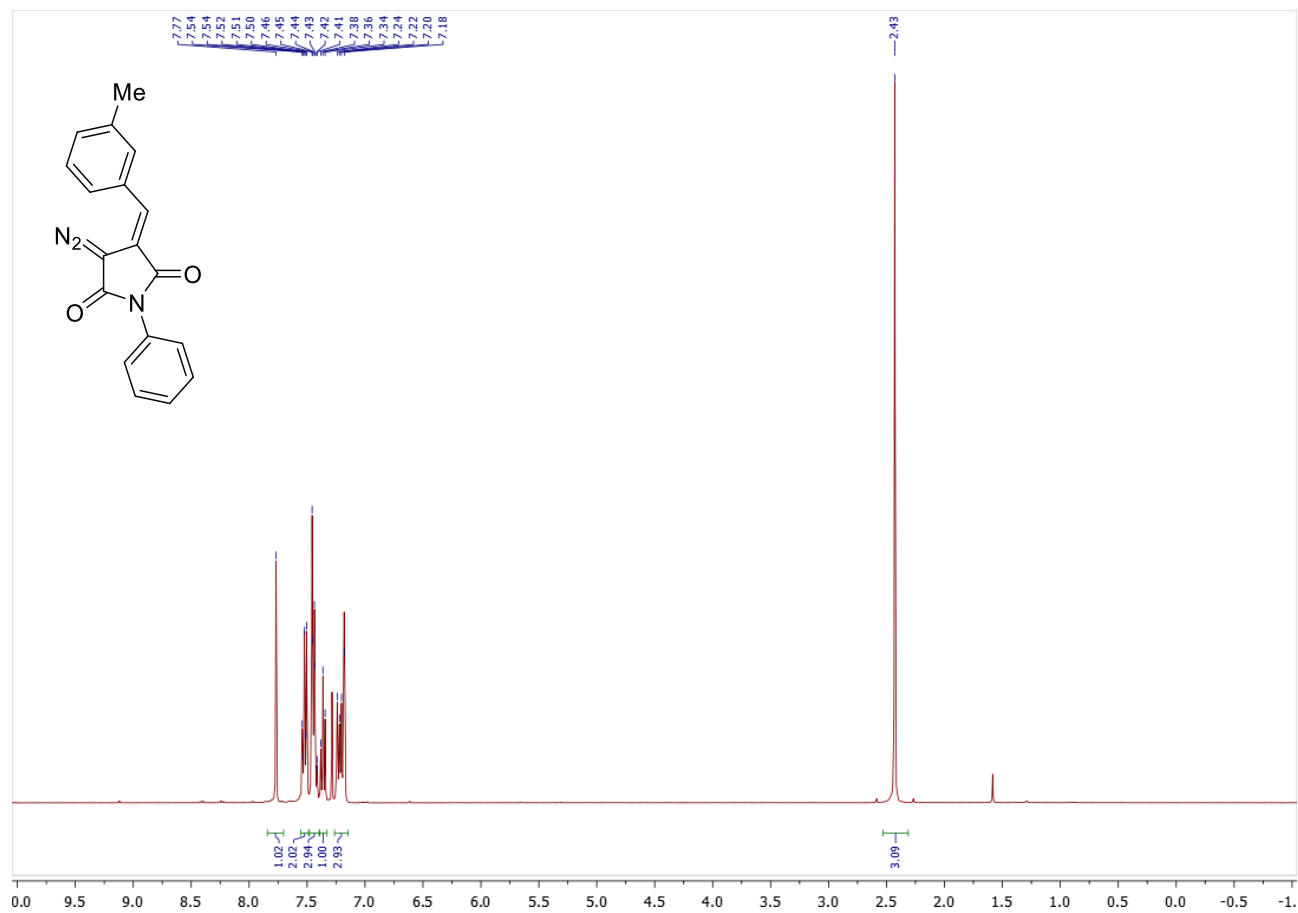
Figure S6. ORTEP representation of compound (*R/S,S/R*)-**6v**^{min} (thermal ellipsoids are shown at 50% probability).

Table S6. Crystal data and structure refinement for (<i>R/S,S/R</i>)- 6v ^{min}	
Empirical formula	C ₂₂ H ₂₀ ClNO ₃
Formula weight	381.84
Temperature/K	100.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	8.3269(3)
b/Å	10.7684(3)
c/Å	11.0047(4)
α/°	70.283(3)
β/°	82.873(3)
γ/°	81.861(3)
Volume/Å ³	916.46(5)
Z	2
ρ _{calc} /cm ³	1.384
μ/mm ⁻¹	2.033
F(000)	400.0
Crystal size/mm ³	0.22 × 0.18 × 0.08
Radiation	Cu Kα (λ = 1.54184)
2θ range for data collection/°	8.564 to 140.85
Index ranges	-8 ≤ h ≤ 10, -13 ≤ k ≤ 13, -13 ≤ l ≤ 13
Reflections collected	15962
Independent reflections	3499 [Rint = 0.0284, Rsigma = 0.0204]
Data/restraints/parameters	3499/0/245
Goodness-of-fit on F ²	1.046
Final R indexes [I ≥ 2σ (I)]	R1 = 0.0811, wR2 = 0.2022
Final R indexes [all data]	R1 = 0.0842, wR2 = 0.2046
Largest diff. peak/hole / e Å ⁻³	1.87/-0.94
CCDC 2027755	

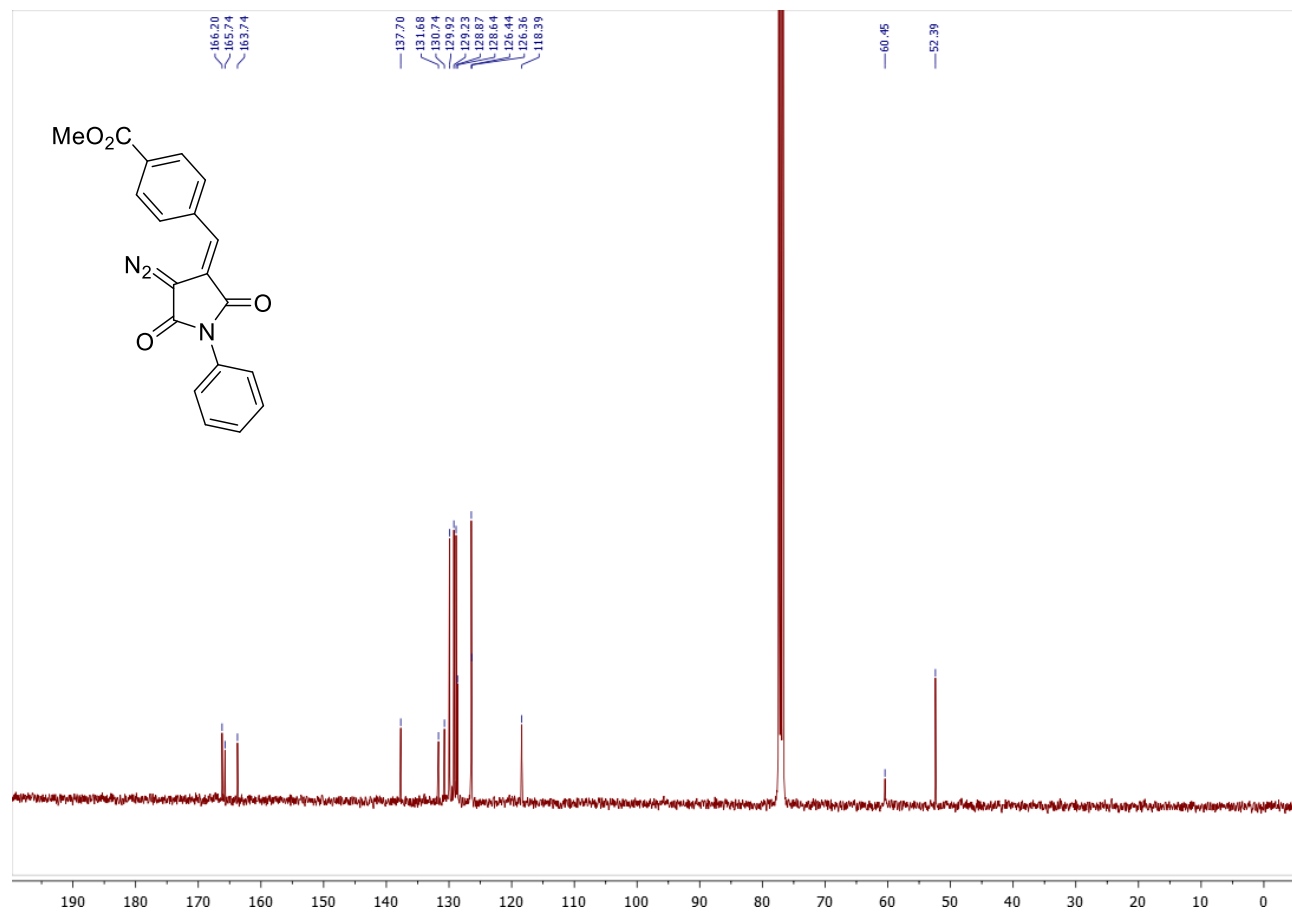
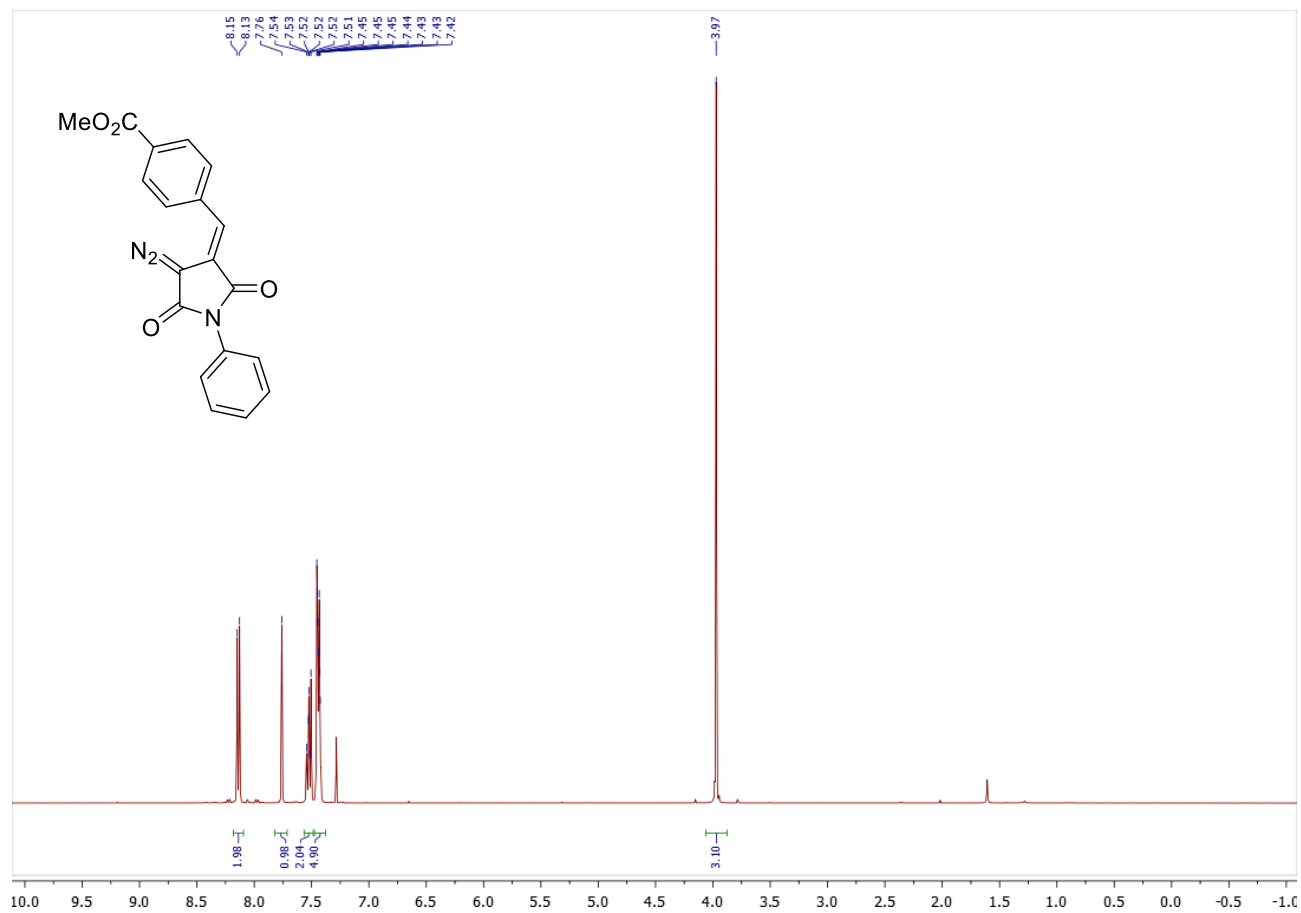
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- (1) Palatinus, L.; Chapuis, G. SUPERFLIP– a Computer Program for the Solution of Crystal Structures by Charge Flipping in Arbitrary Dimensions. *J. Appl. Crystallogr.* **2007**, *40*, 786–790.
- (2) Sheldrick, G. M. A Short History of *SHELX*. *Acta Crystallogr. Sect. A* **2008**, *64*, 112–122.
- (3) Sheldrick, G. M. Crystal Structure Refinement with *SHELXL*. *Acta Crystallogr. Sect. C* **2015**, *71*, 3–8.
- (4) Dolomanov, O. V; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. *OLEX2*: A Complete Structure Solution, Refinement and Analysis Program. *J. Appl. Crystallogr.* **2009**, *42*, 339–341.

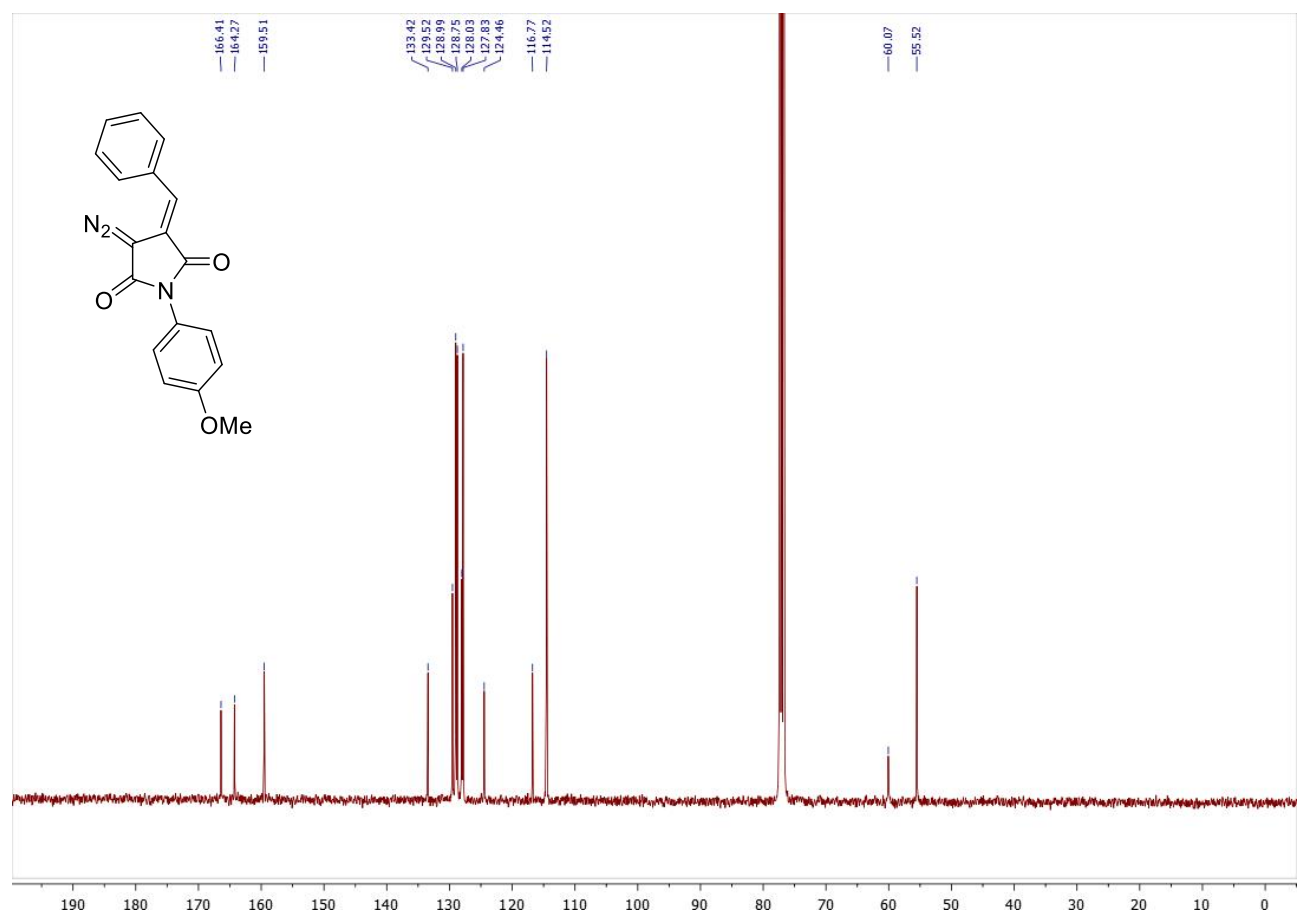
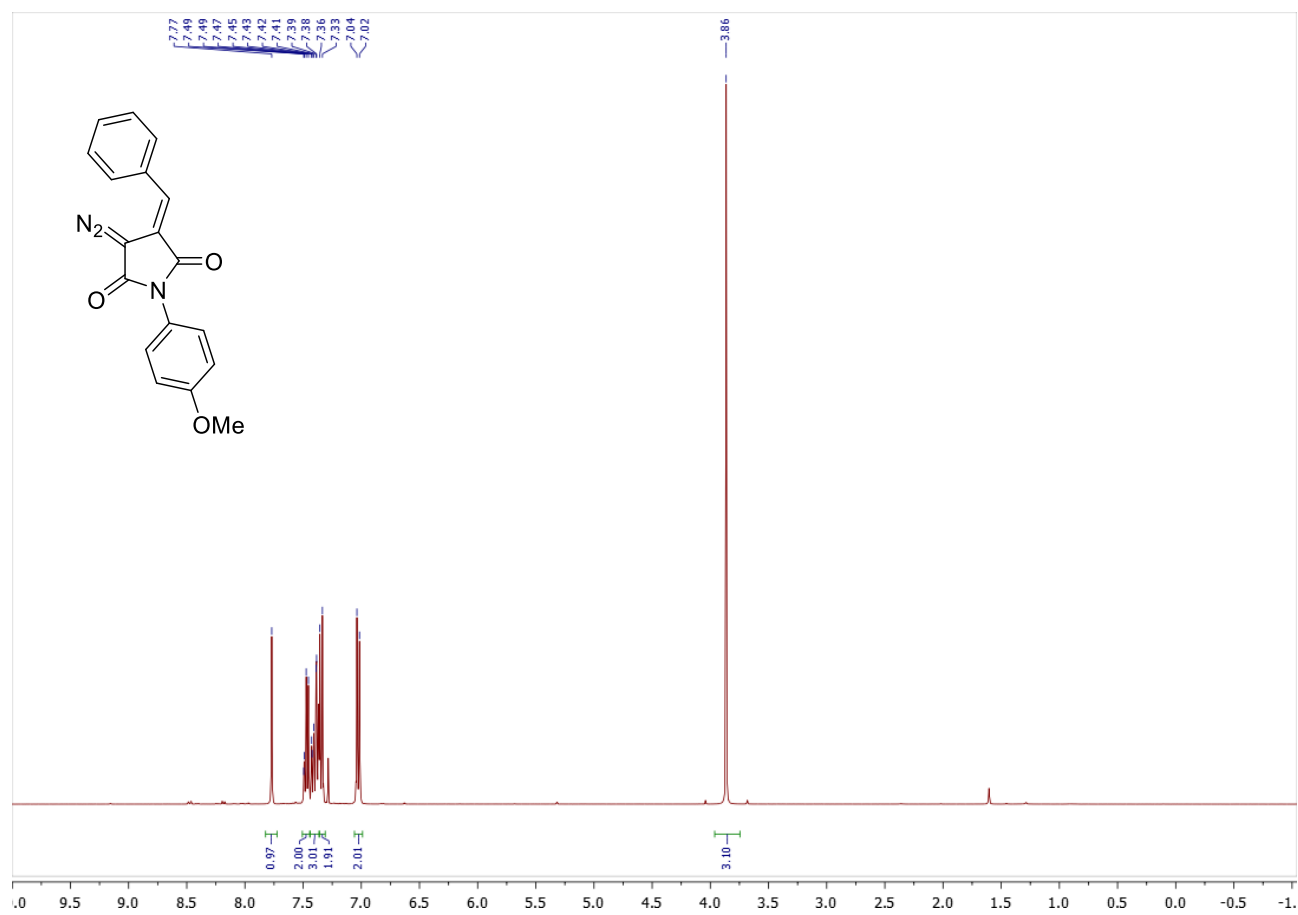
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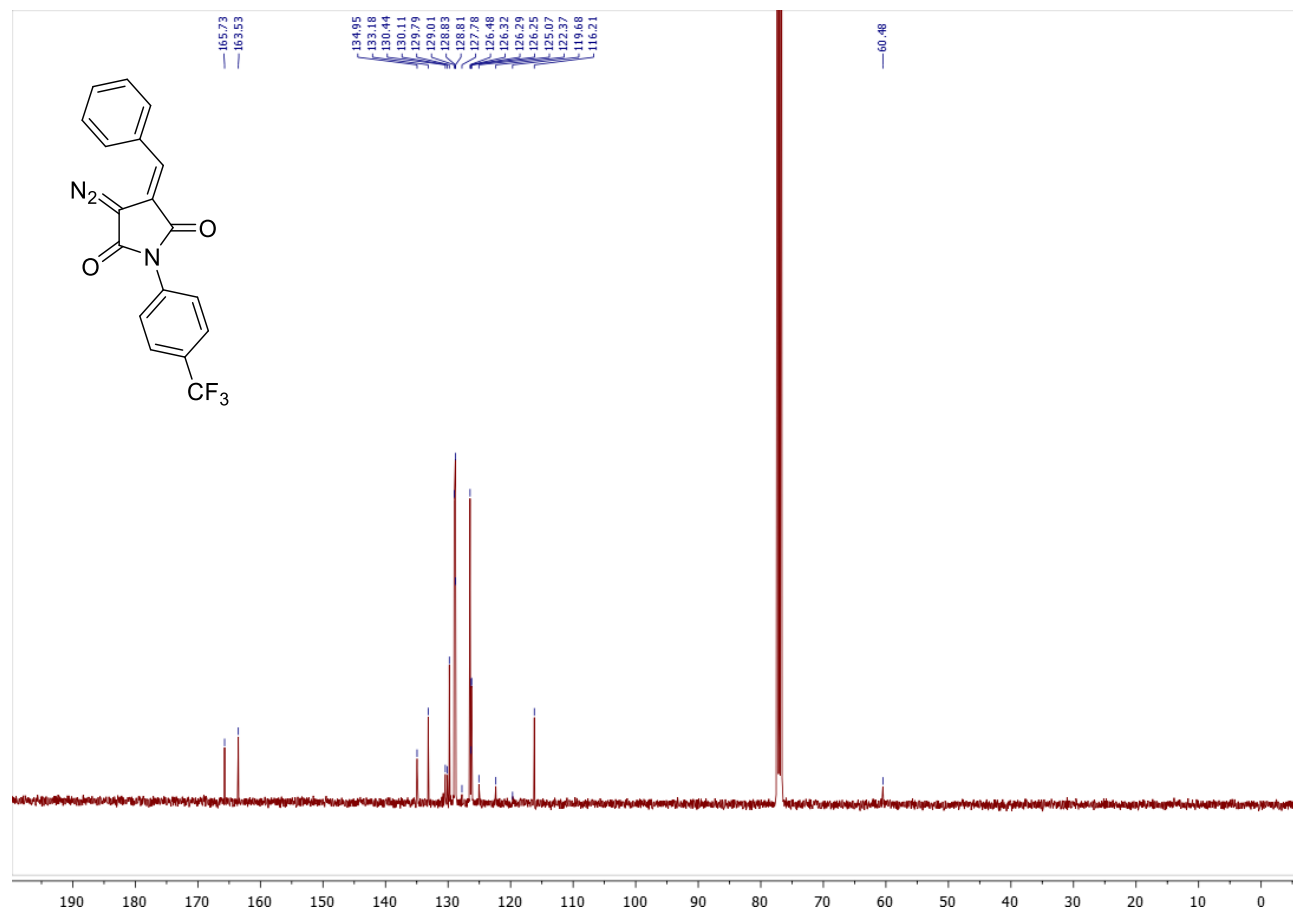
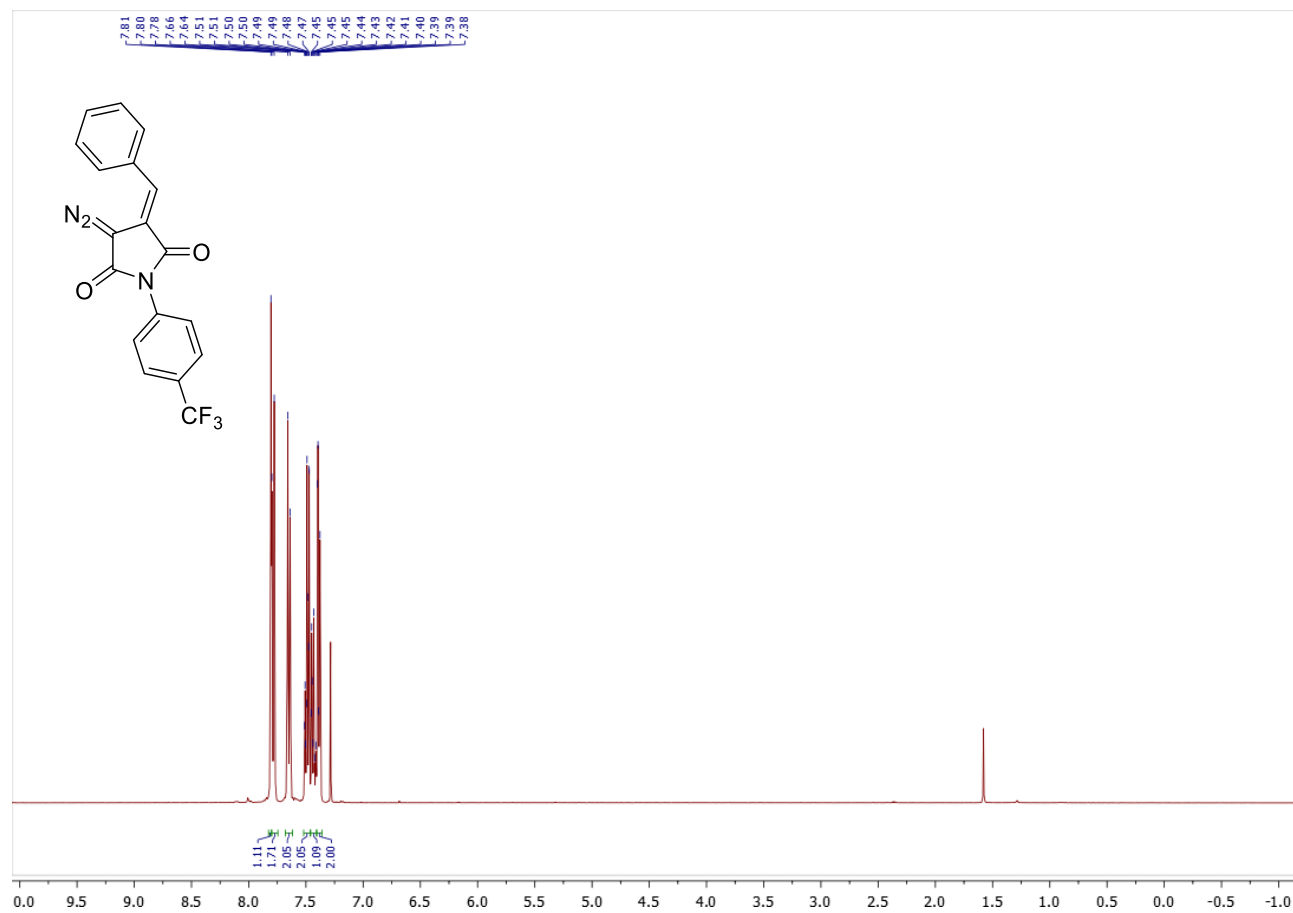
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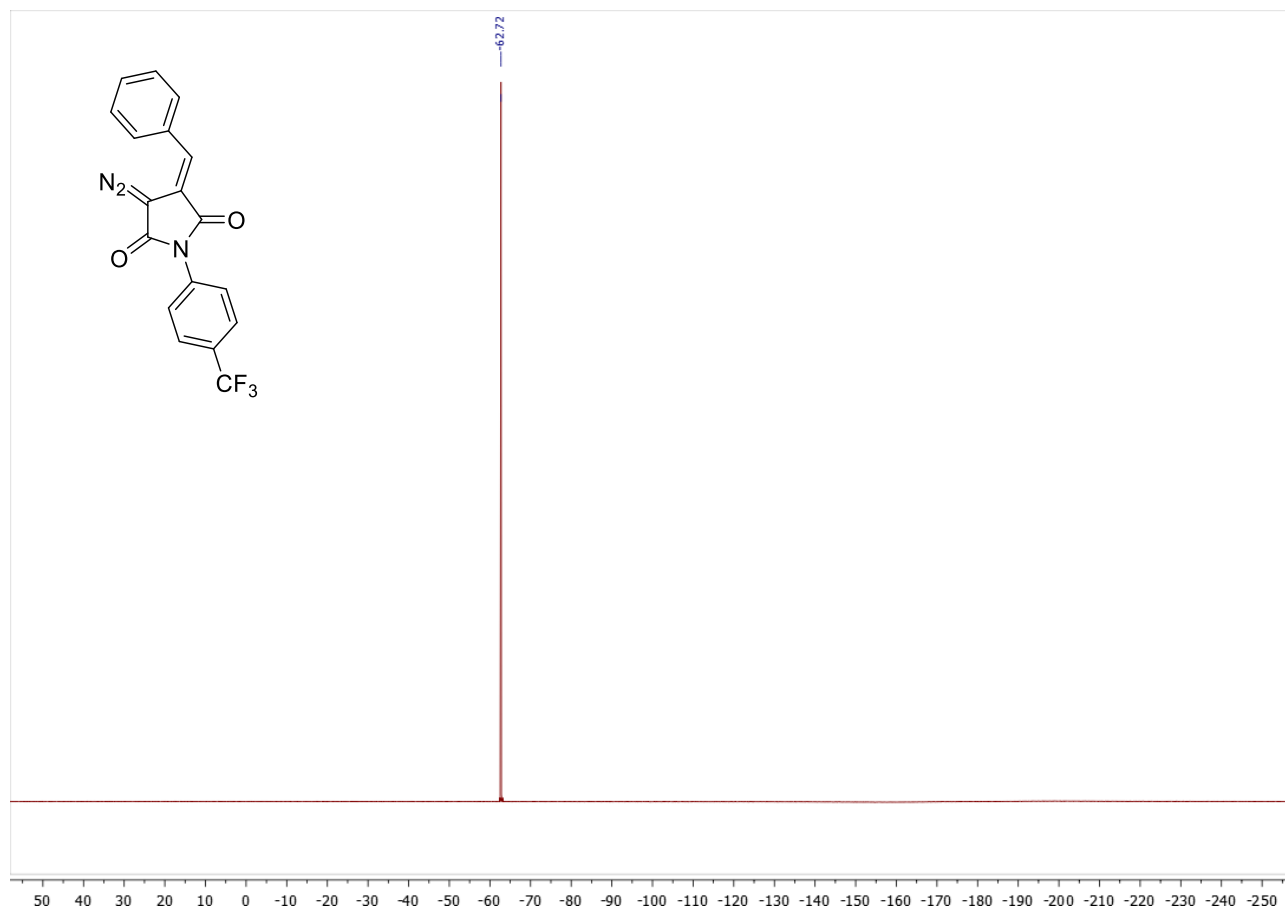
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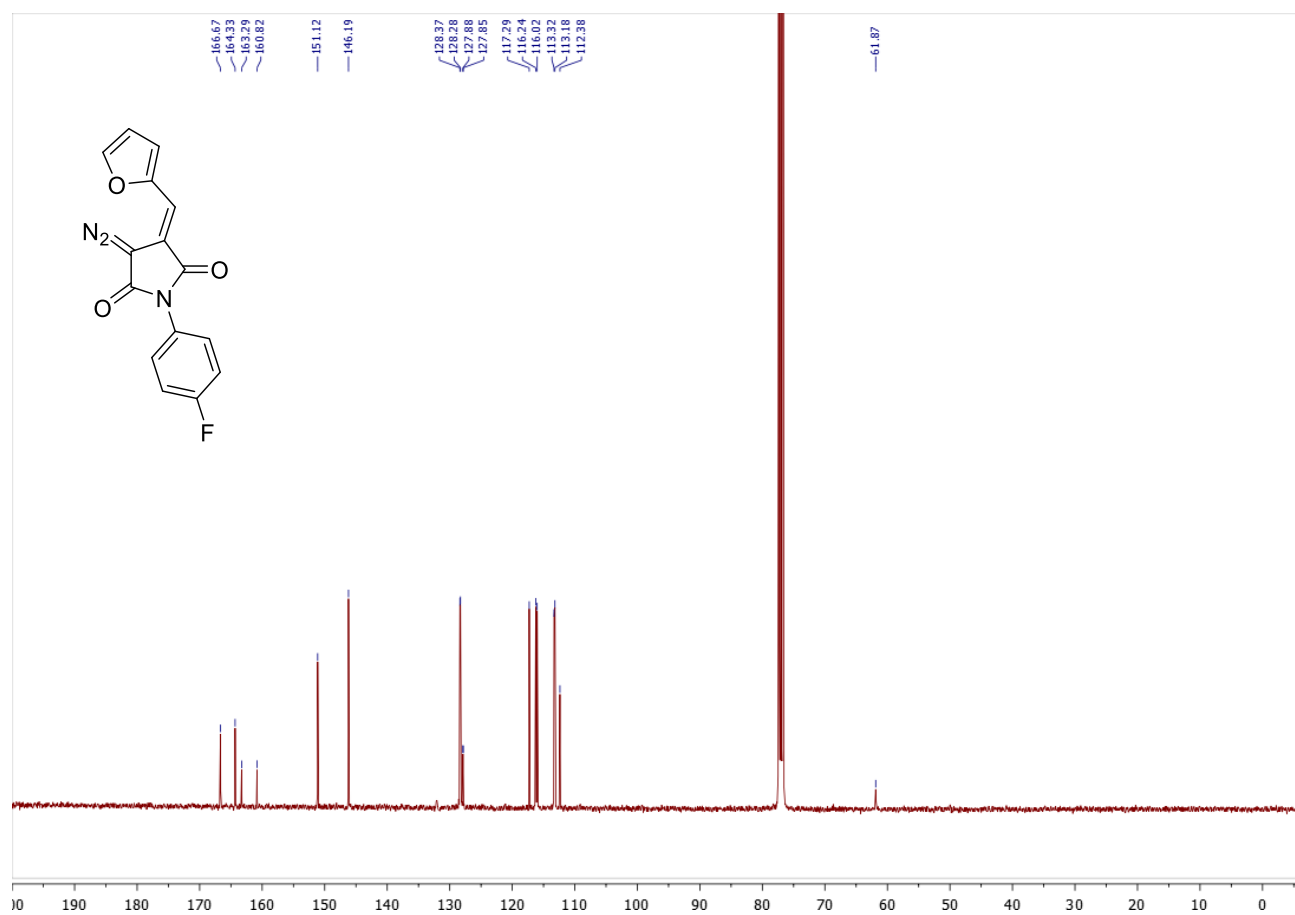
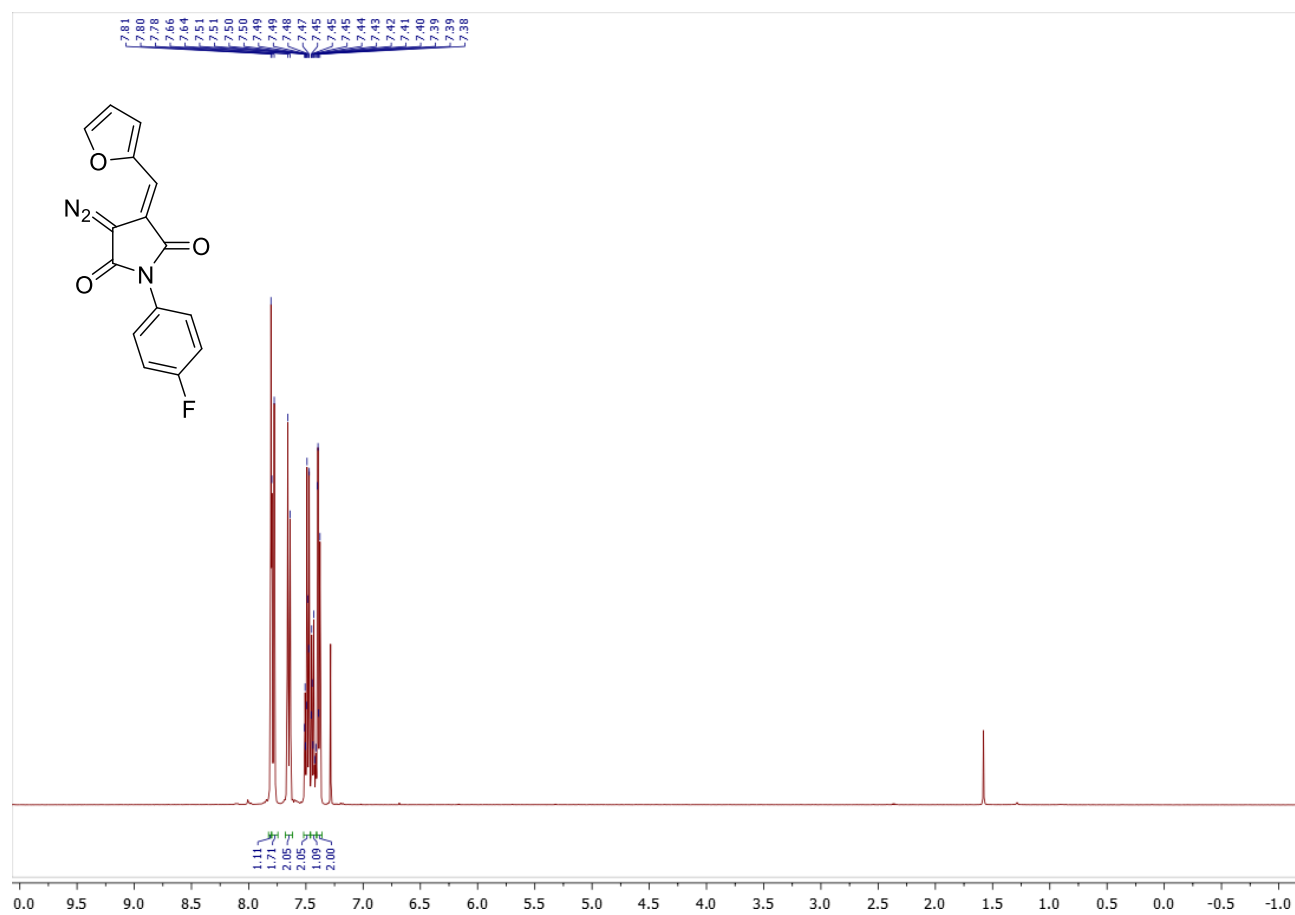
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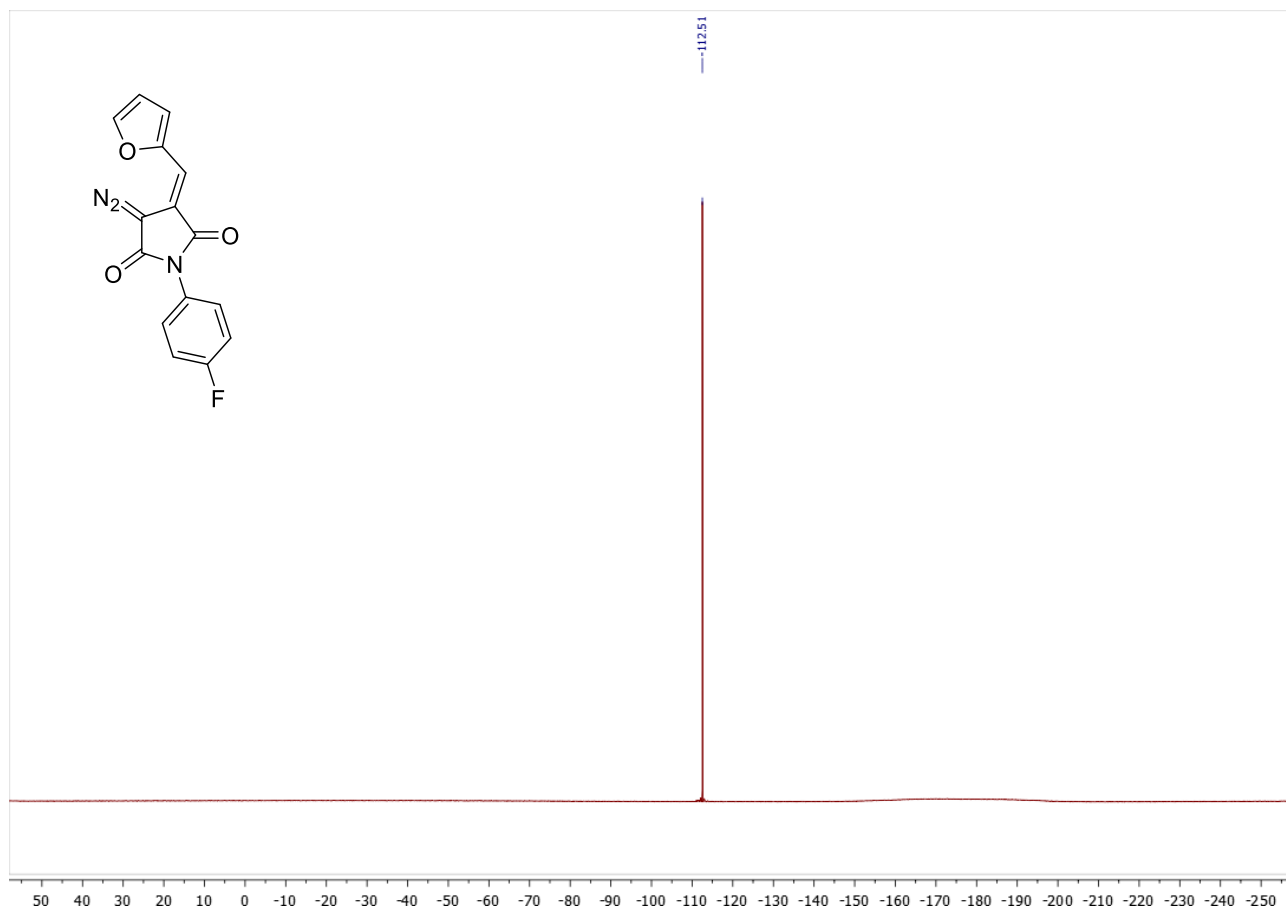
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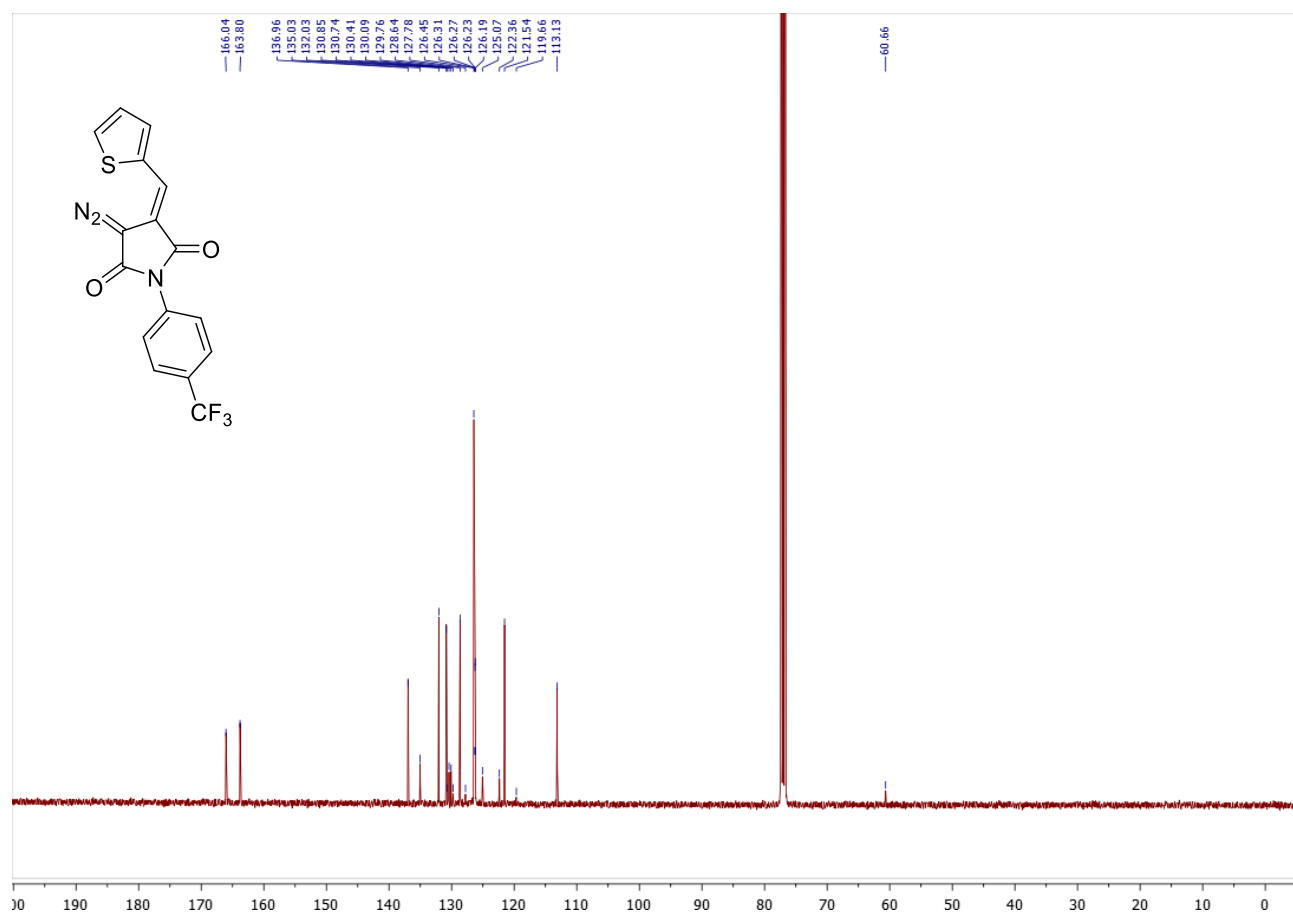
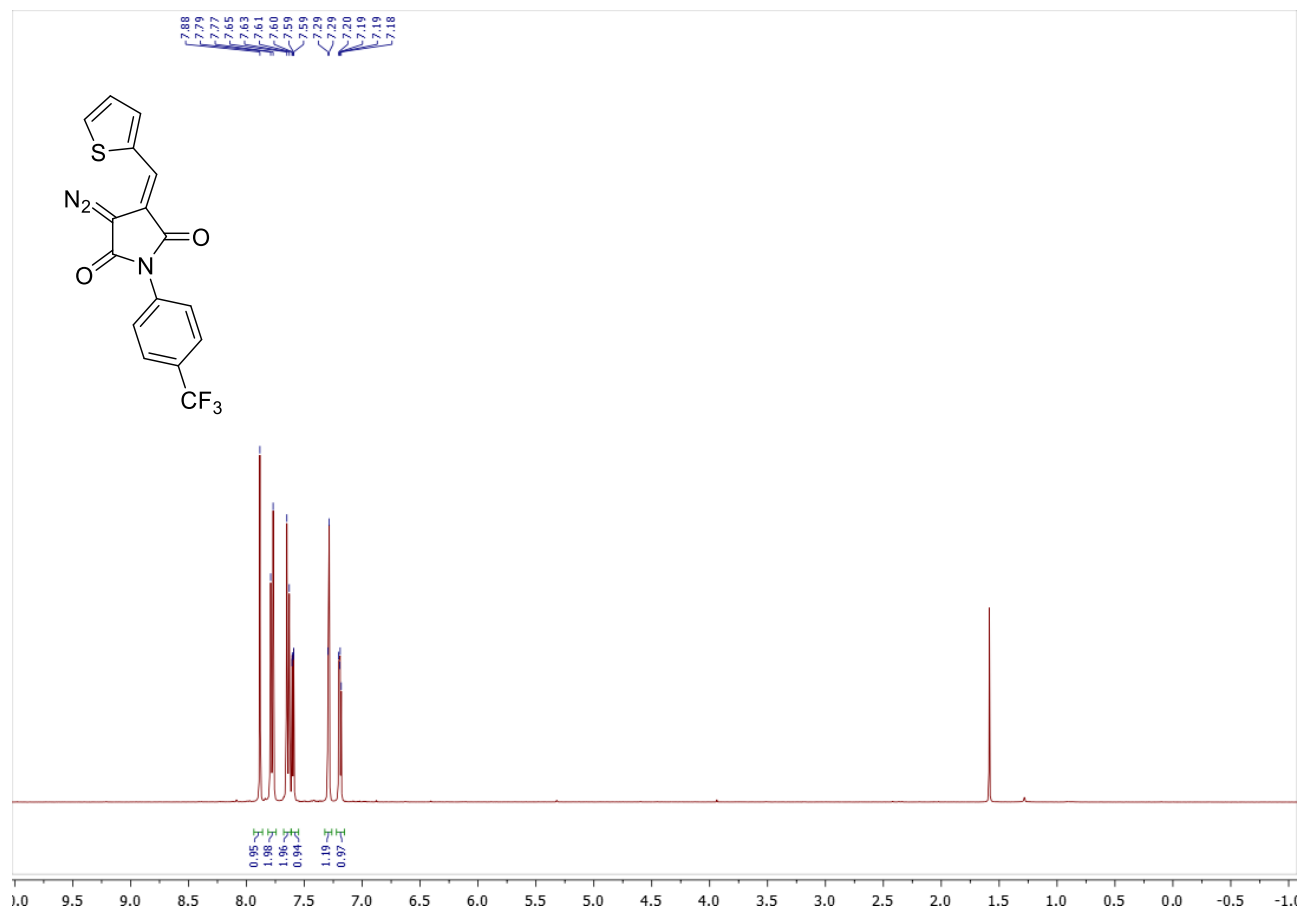
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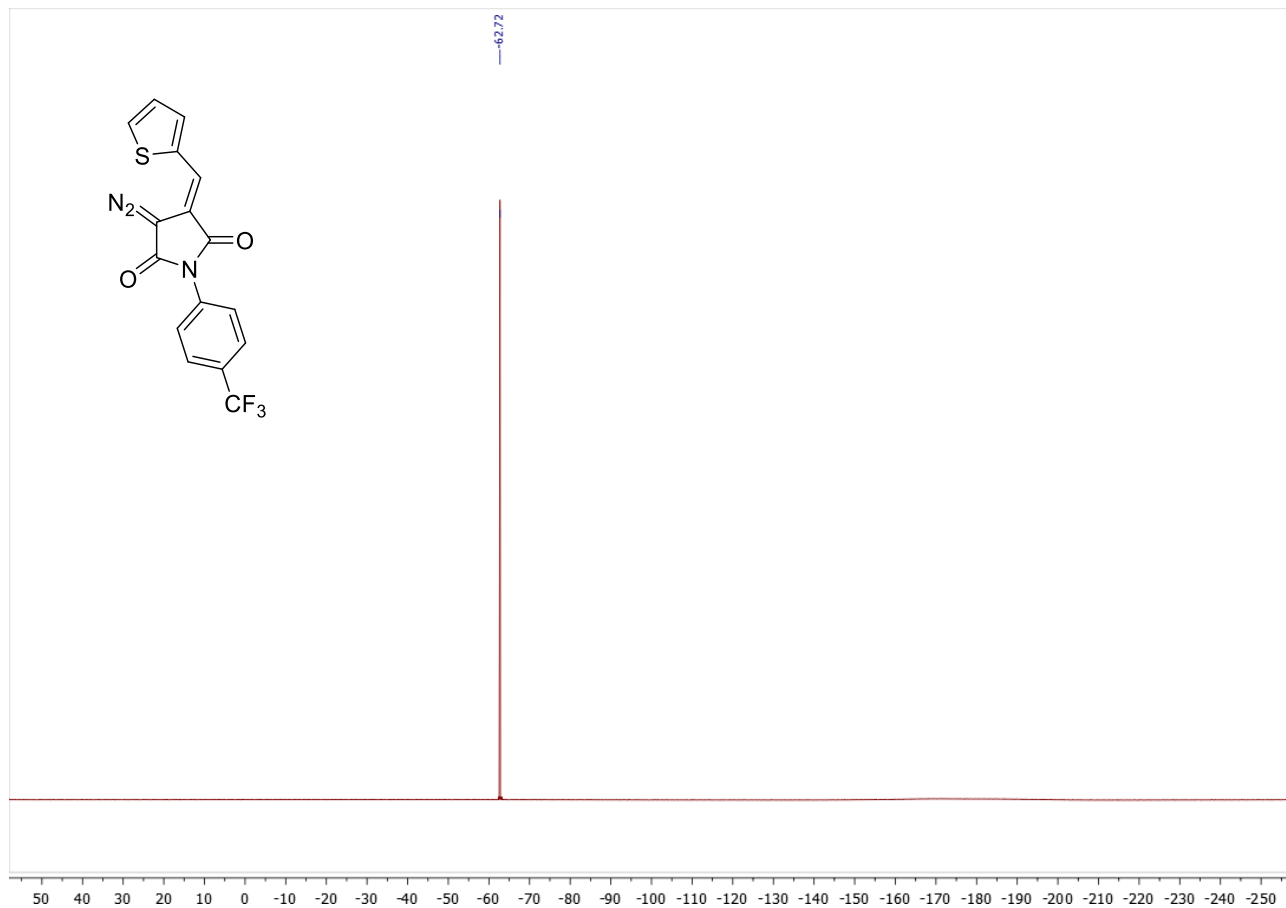
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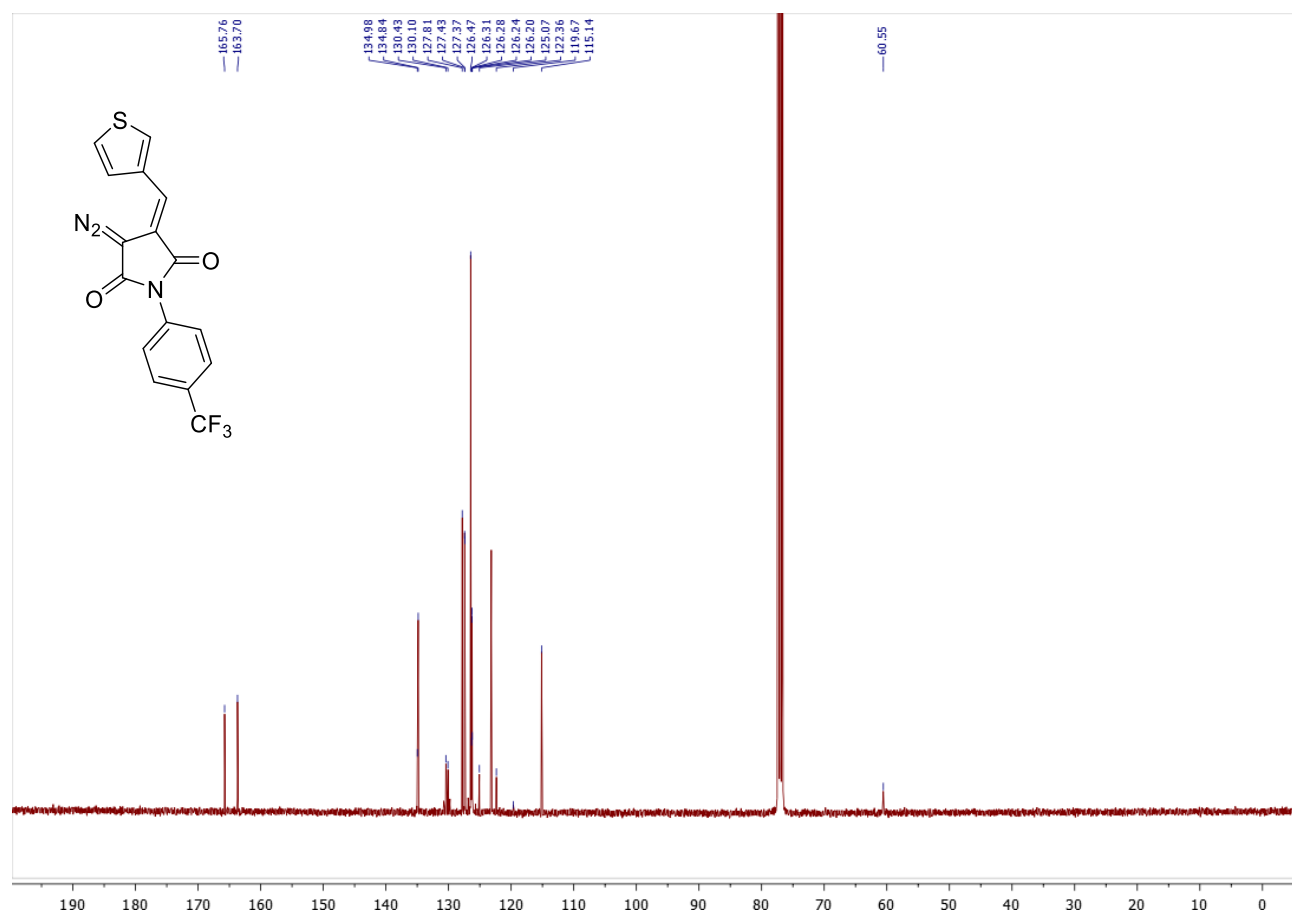
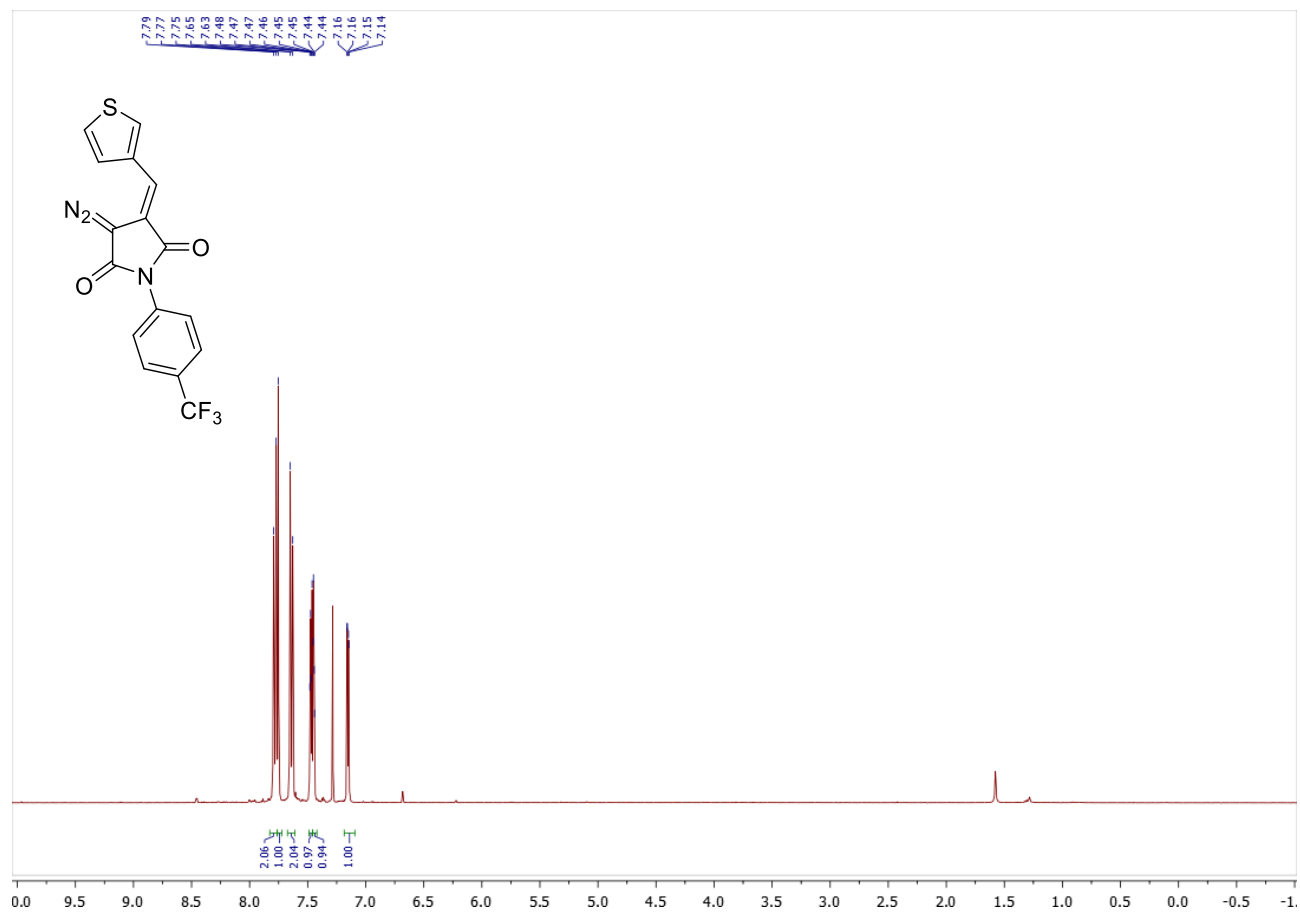
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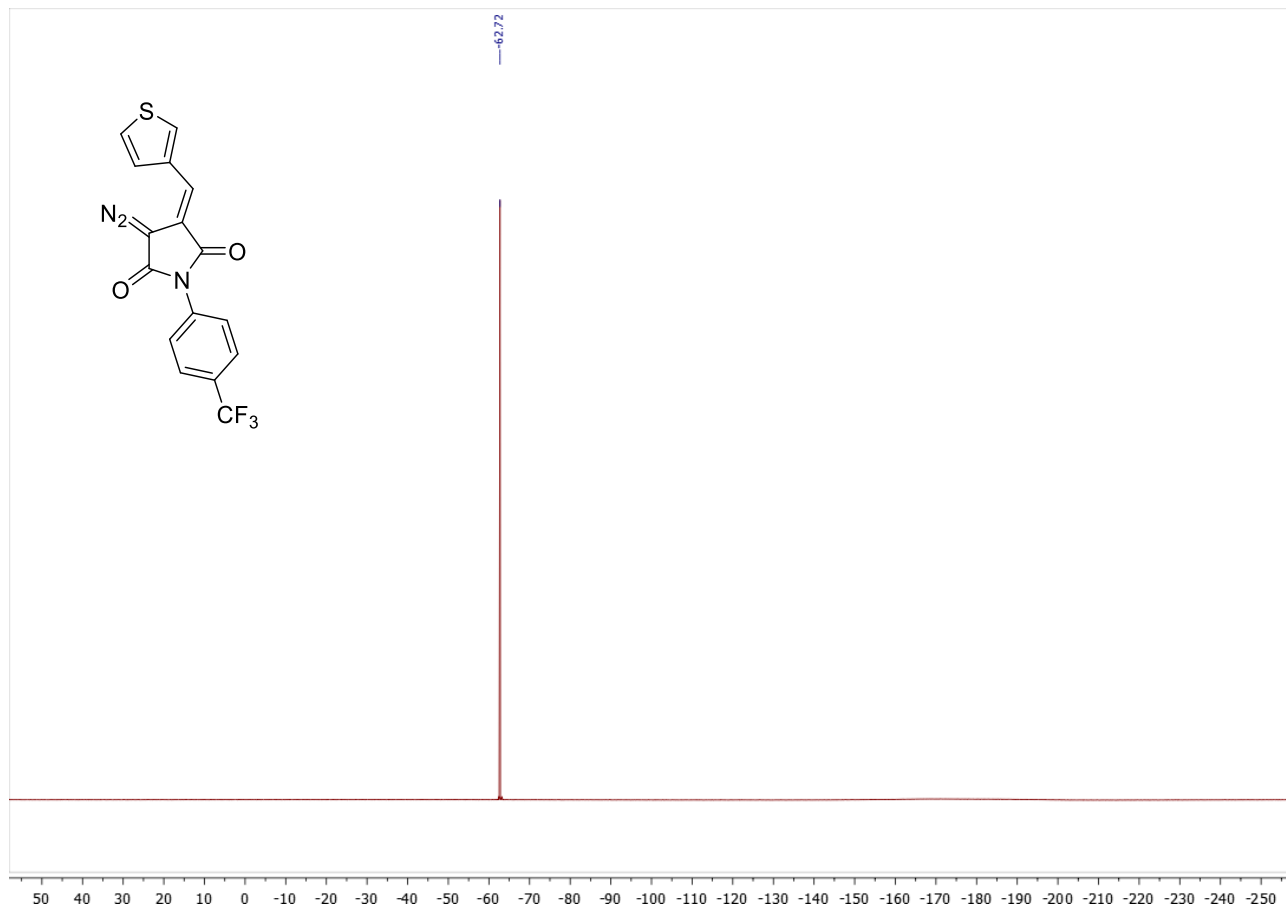
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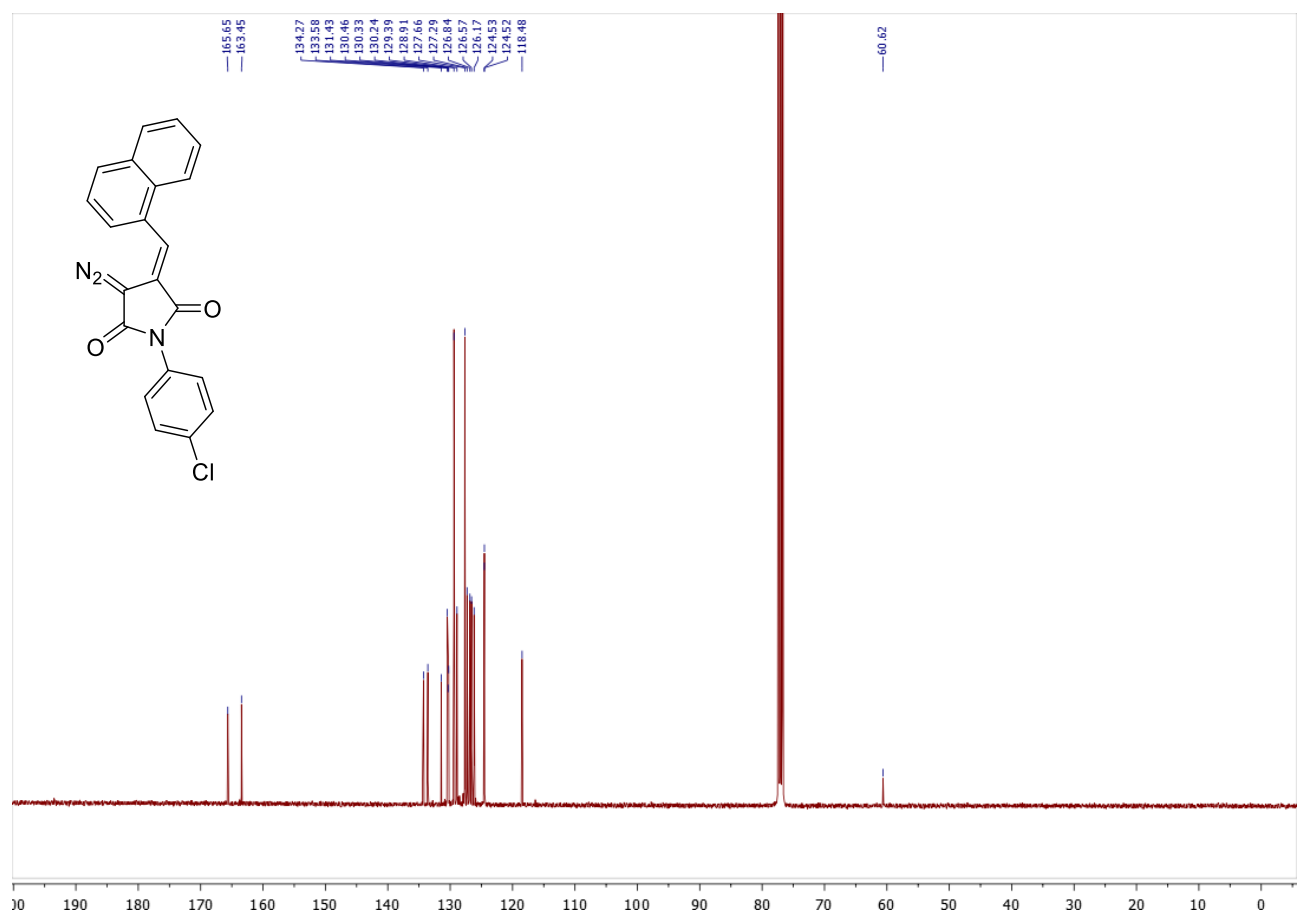
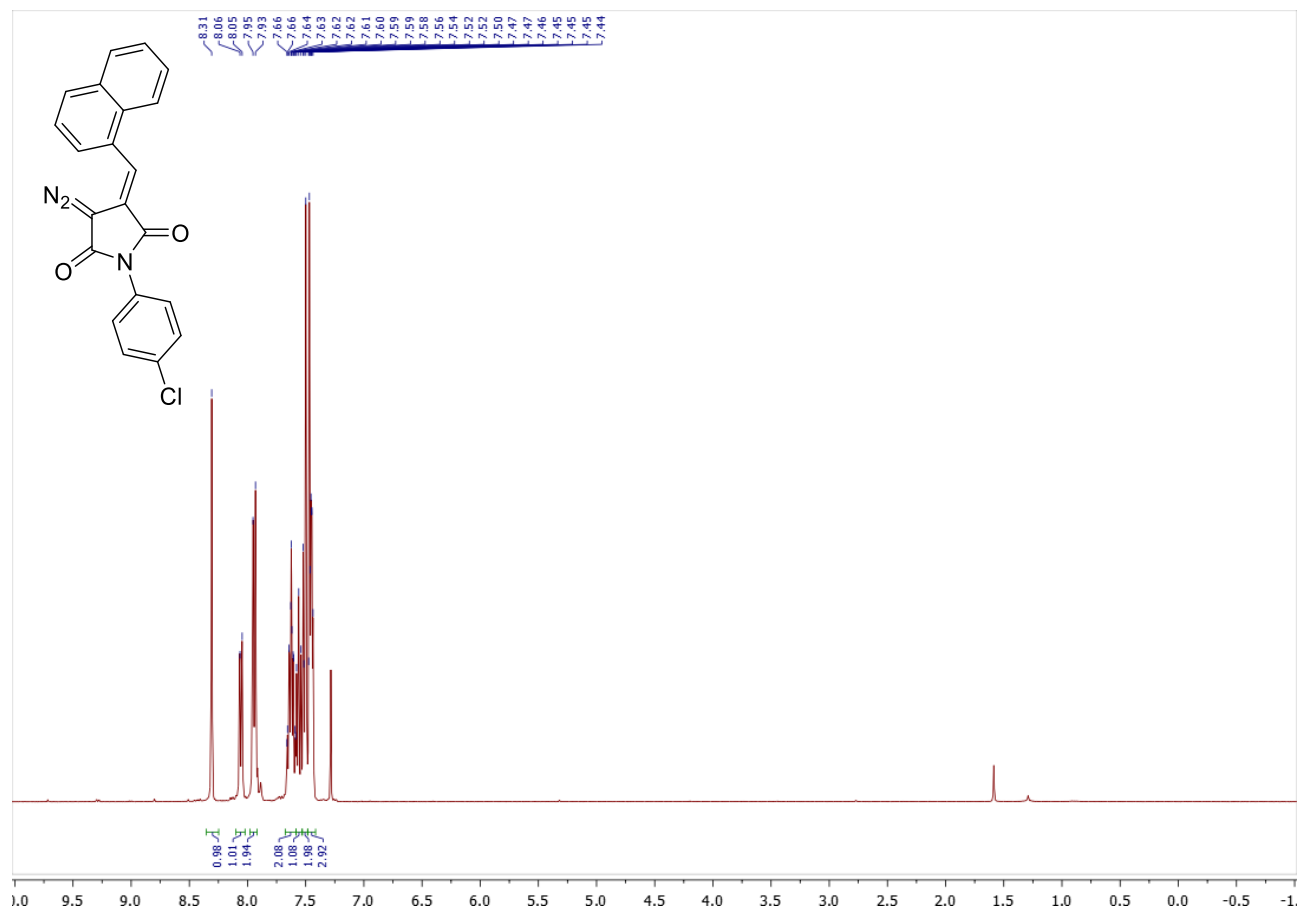
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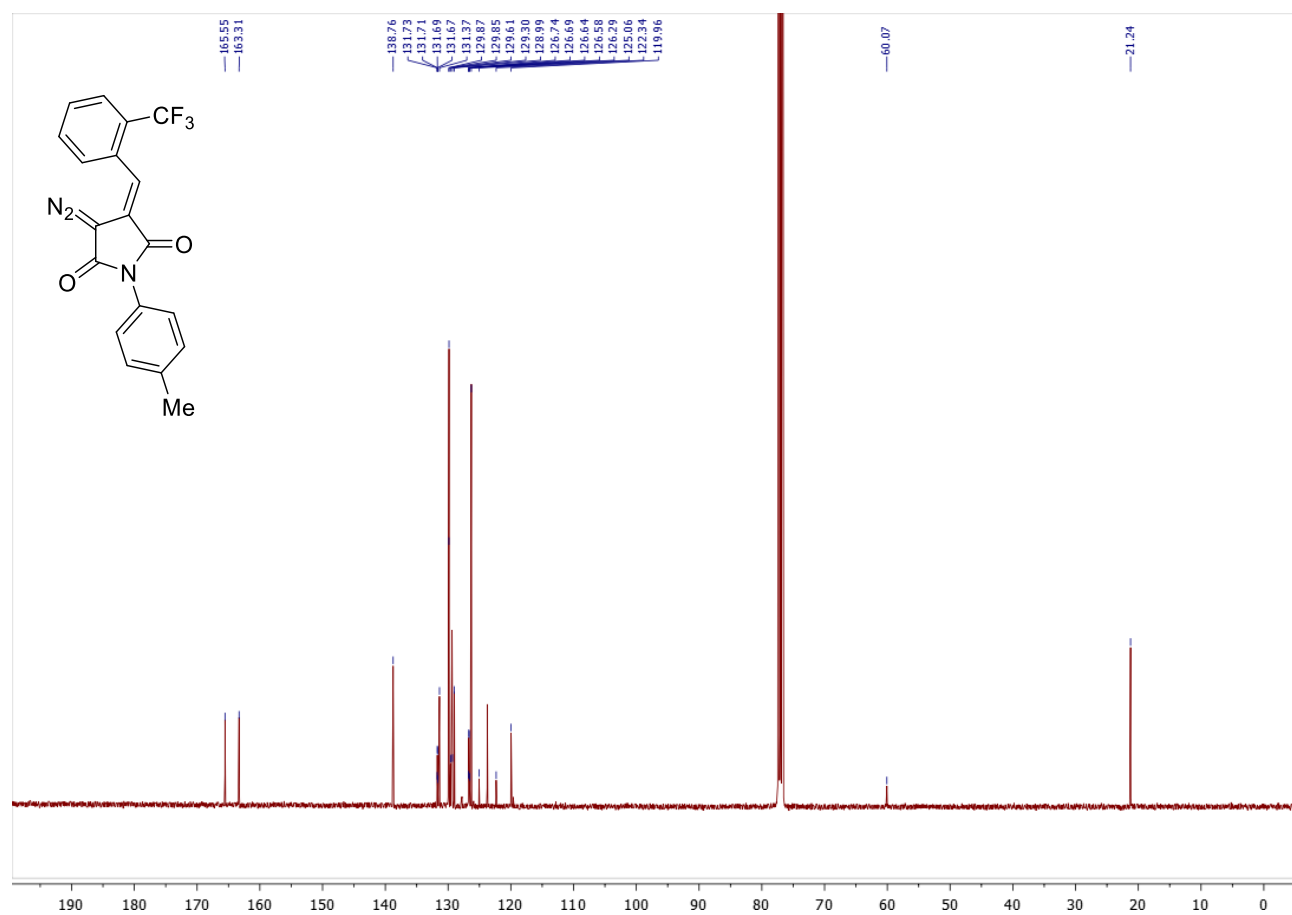
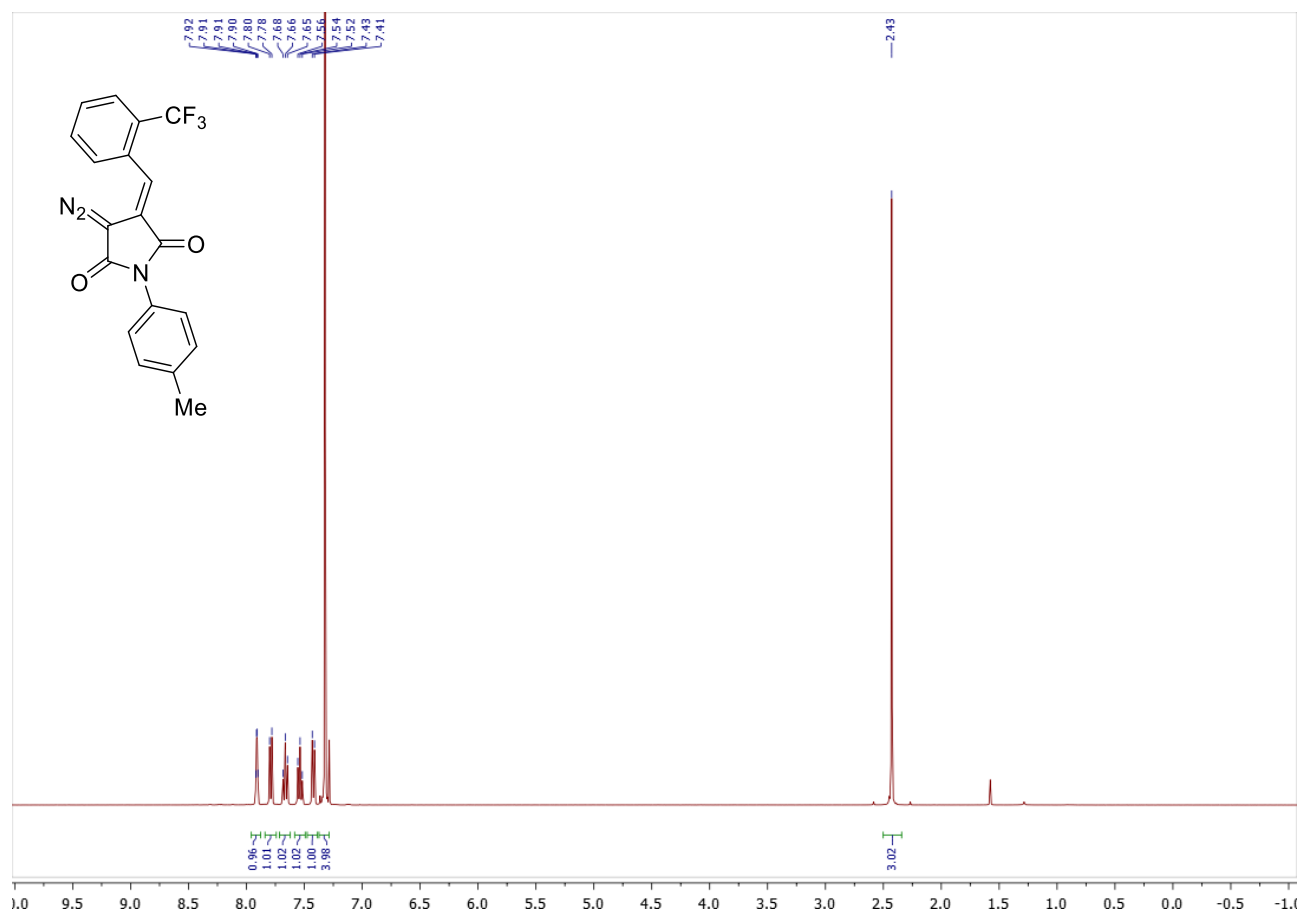
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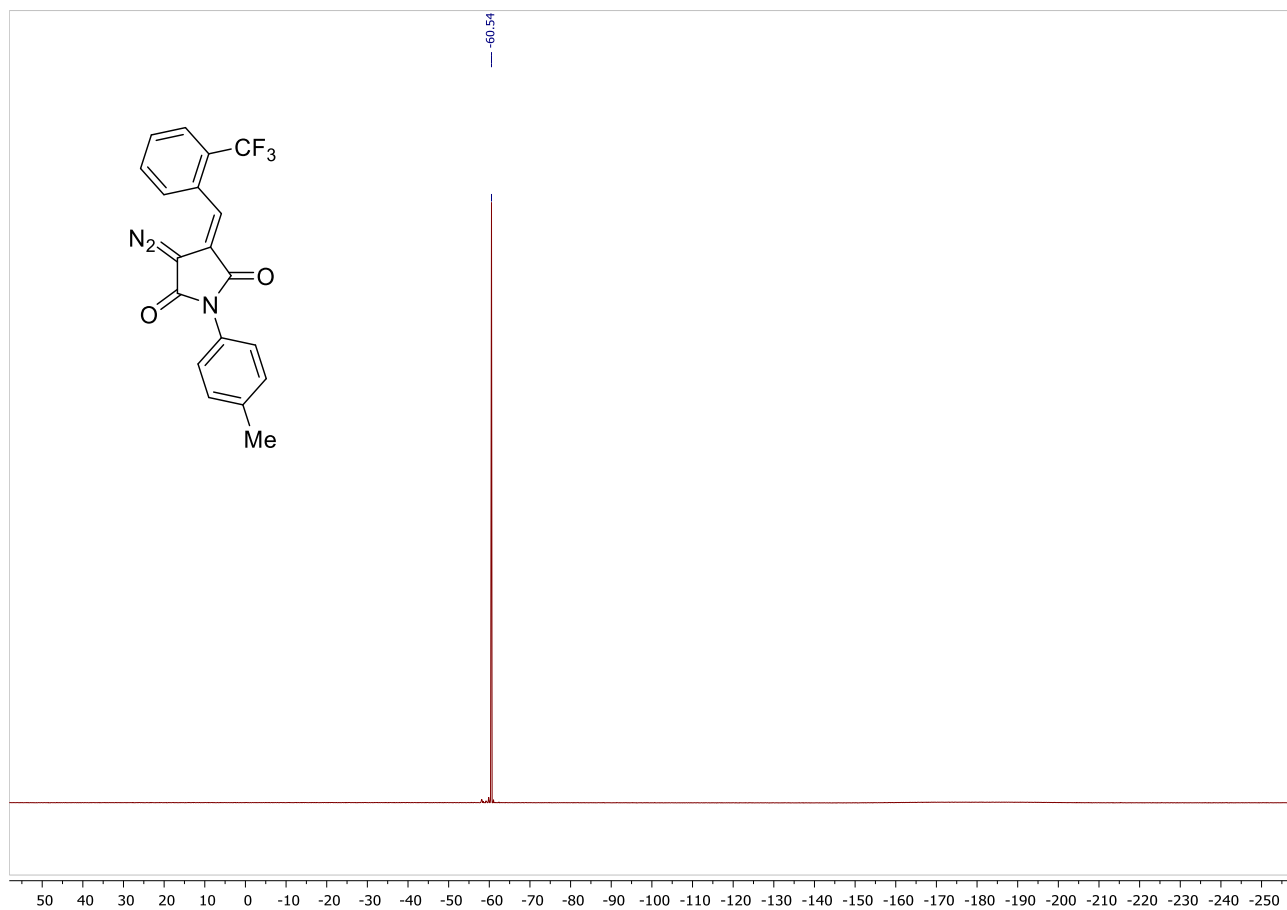
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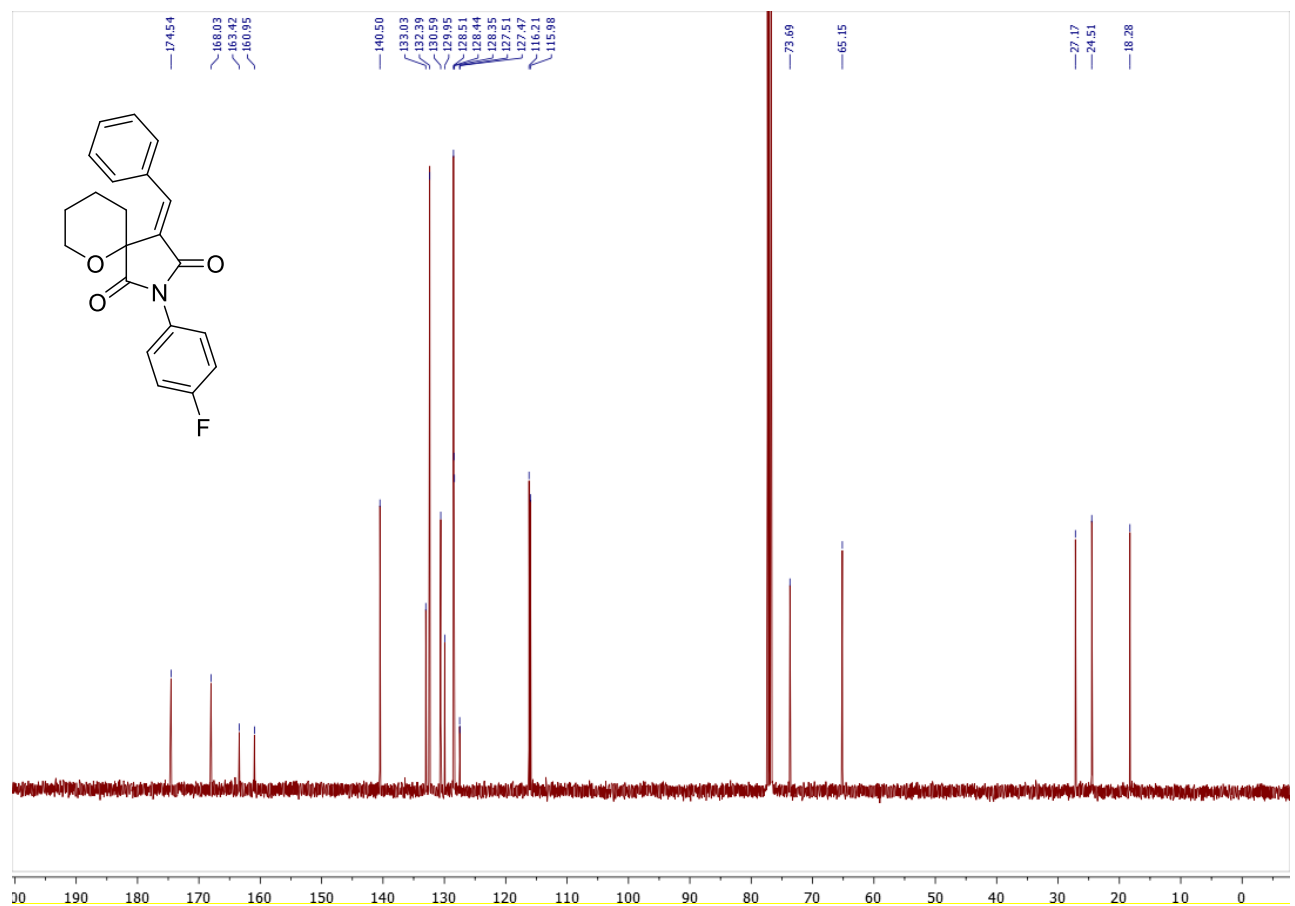
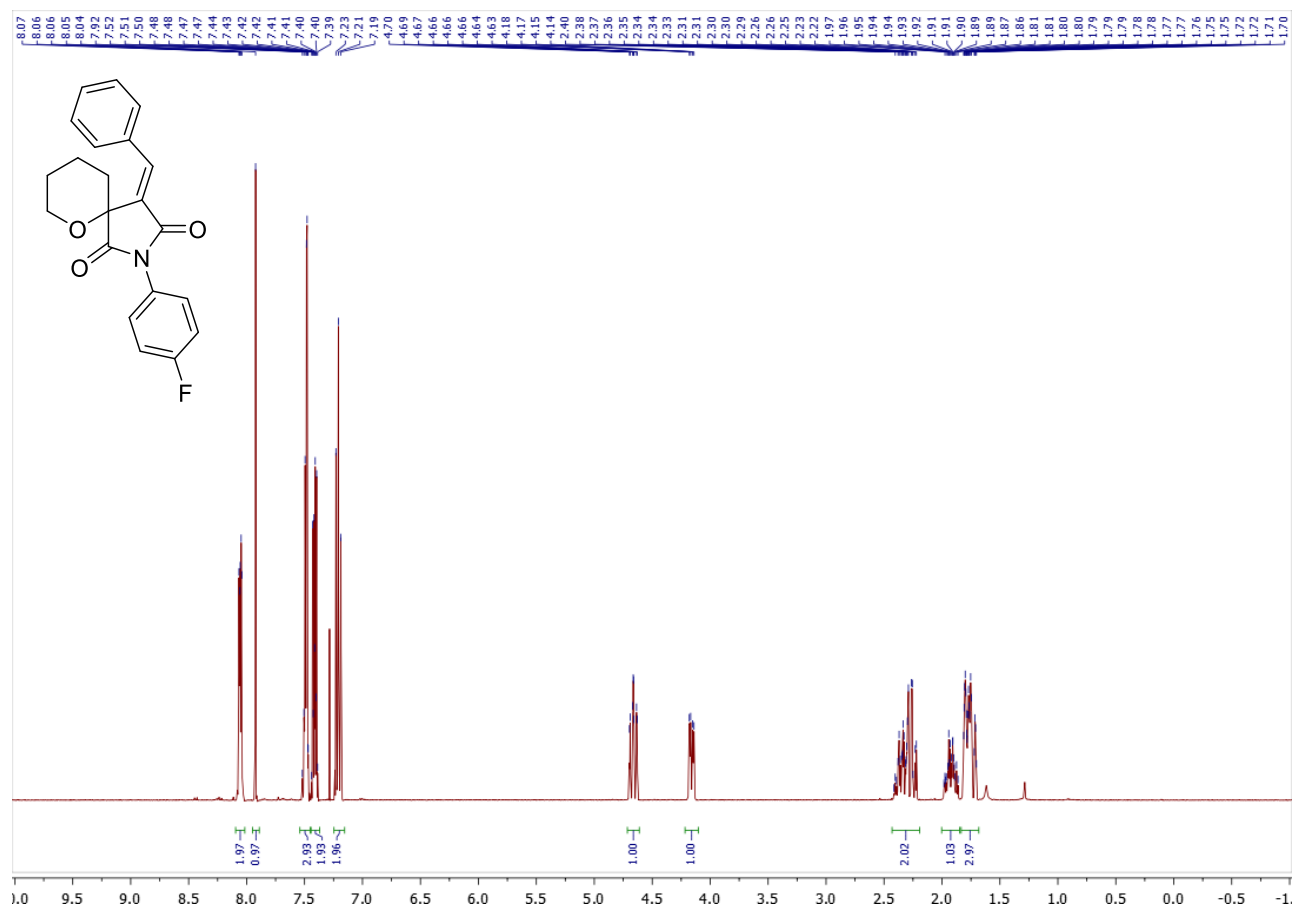
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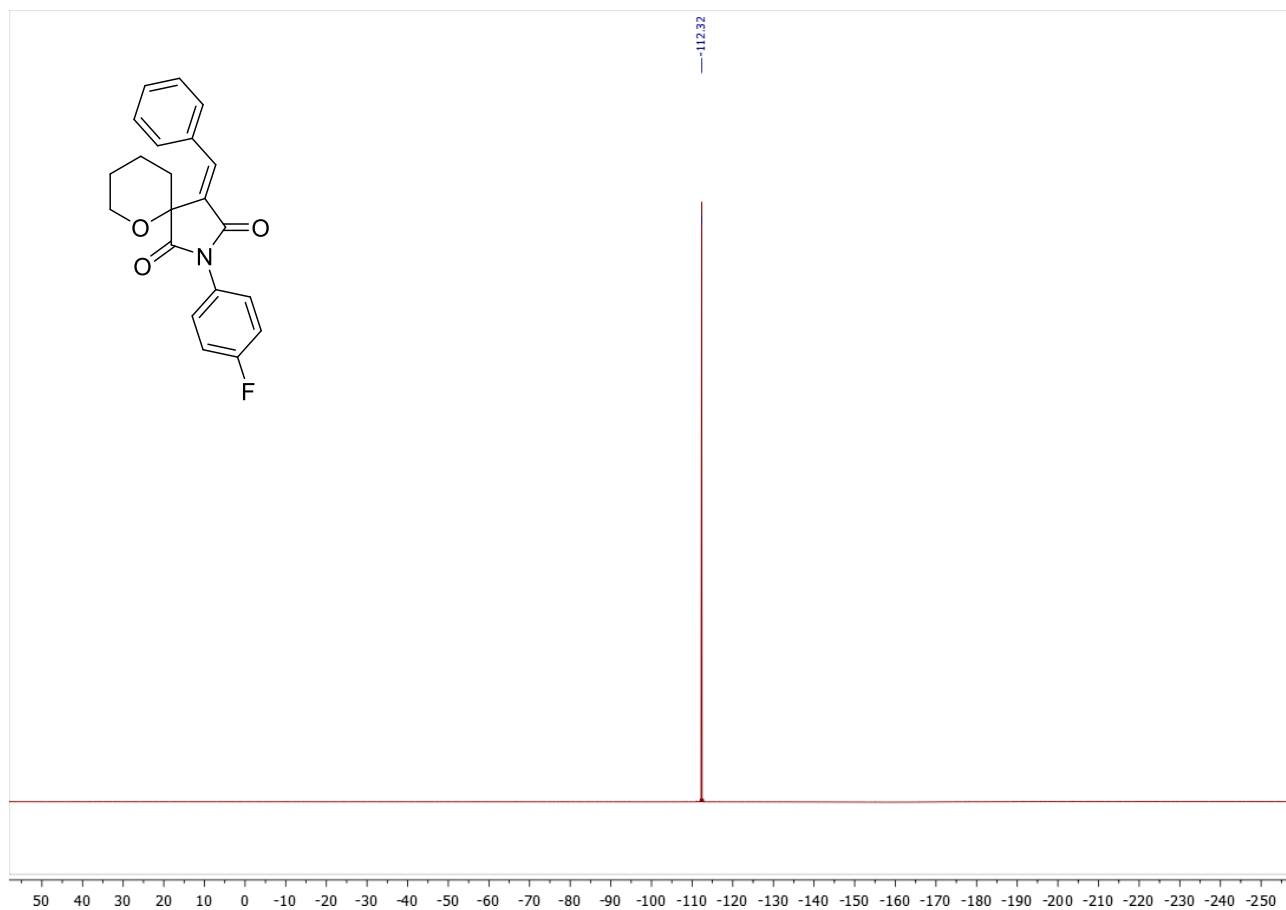
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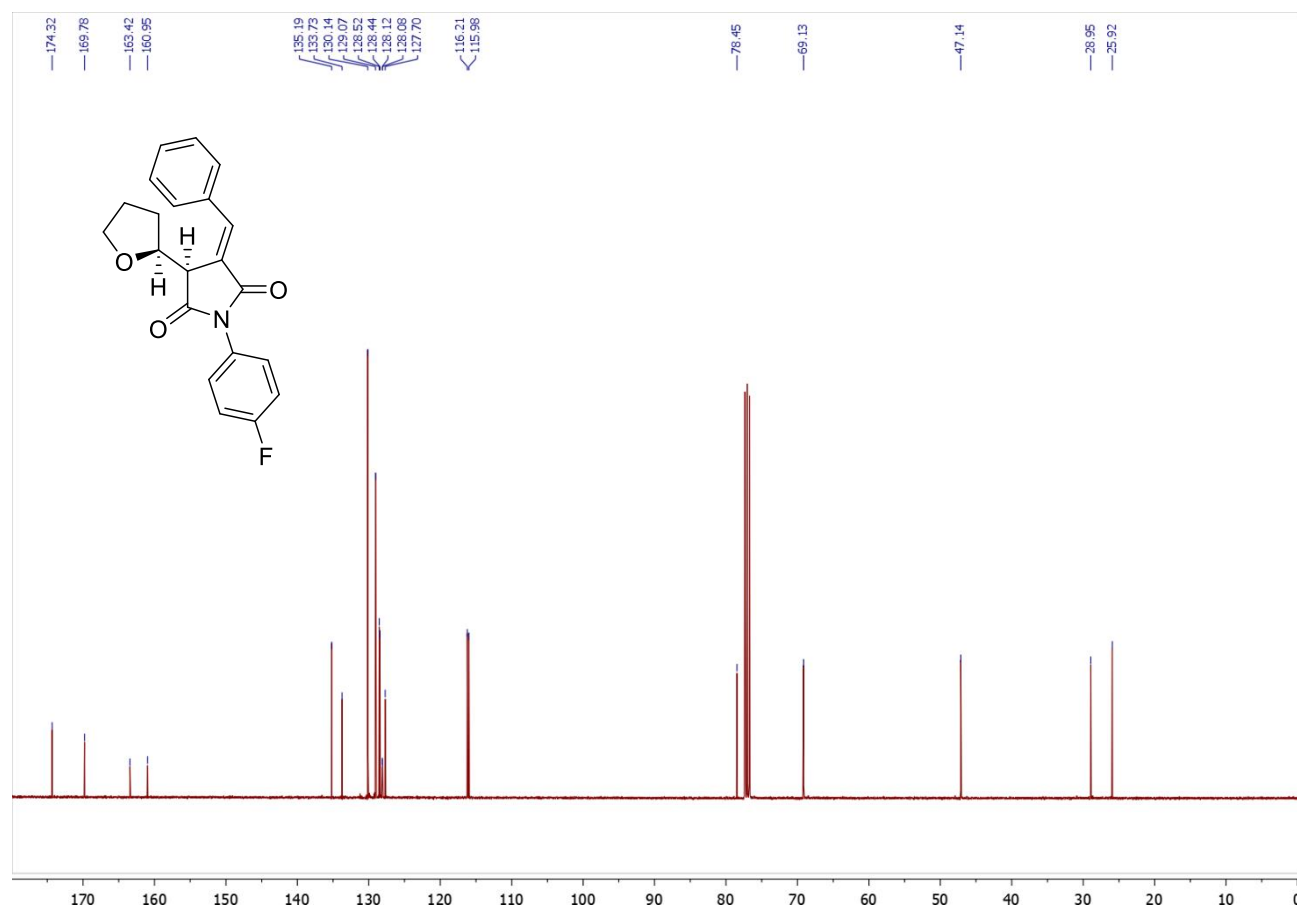
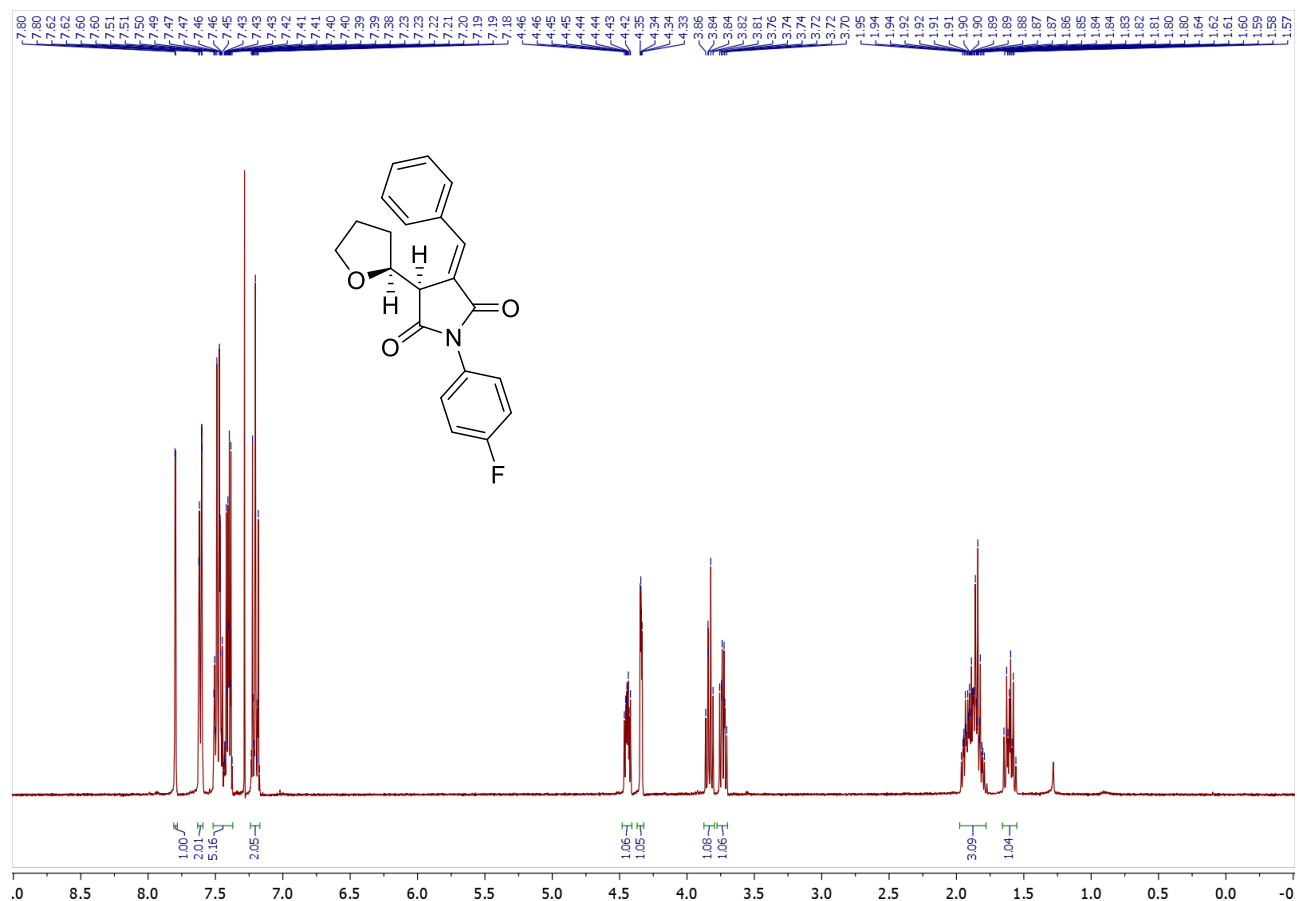
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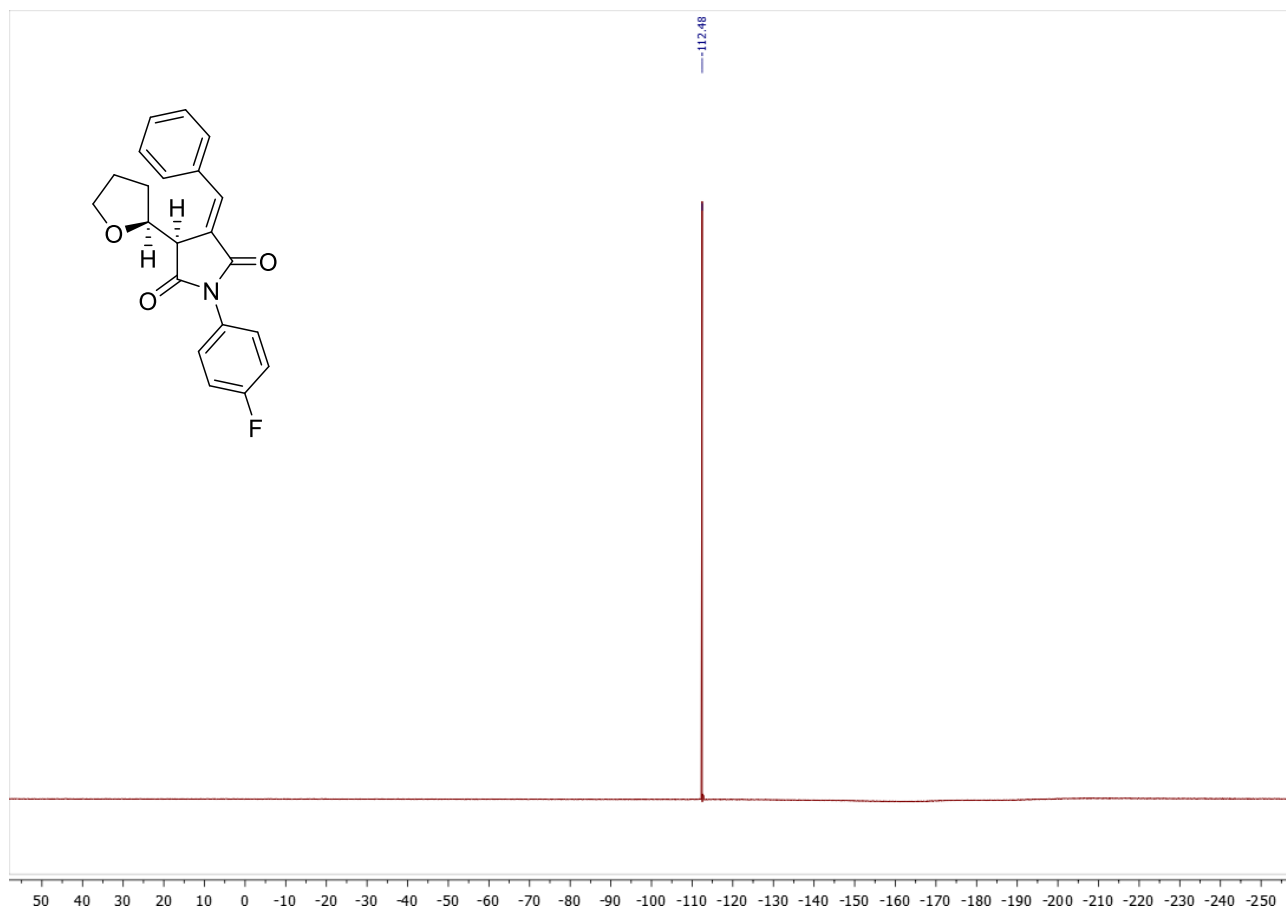
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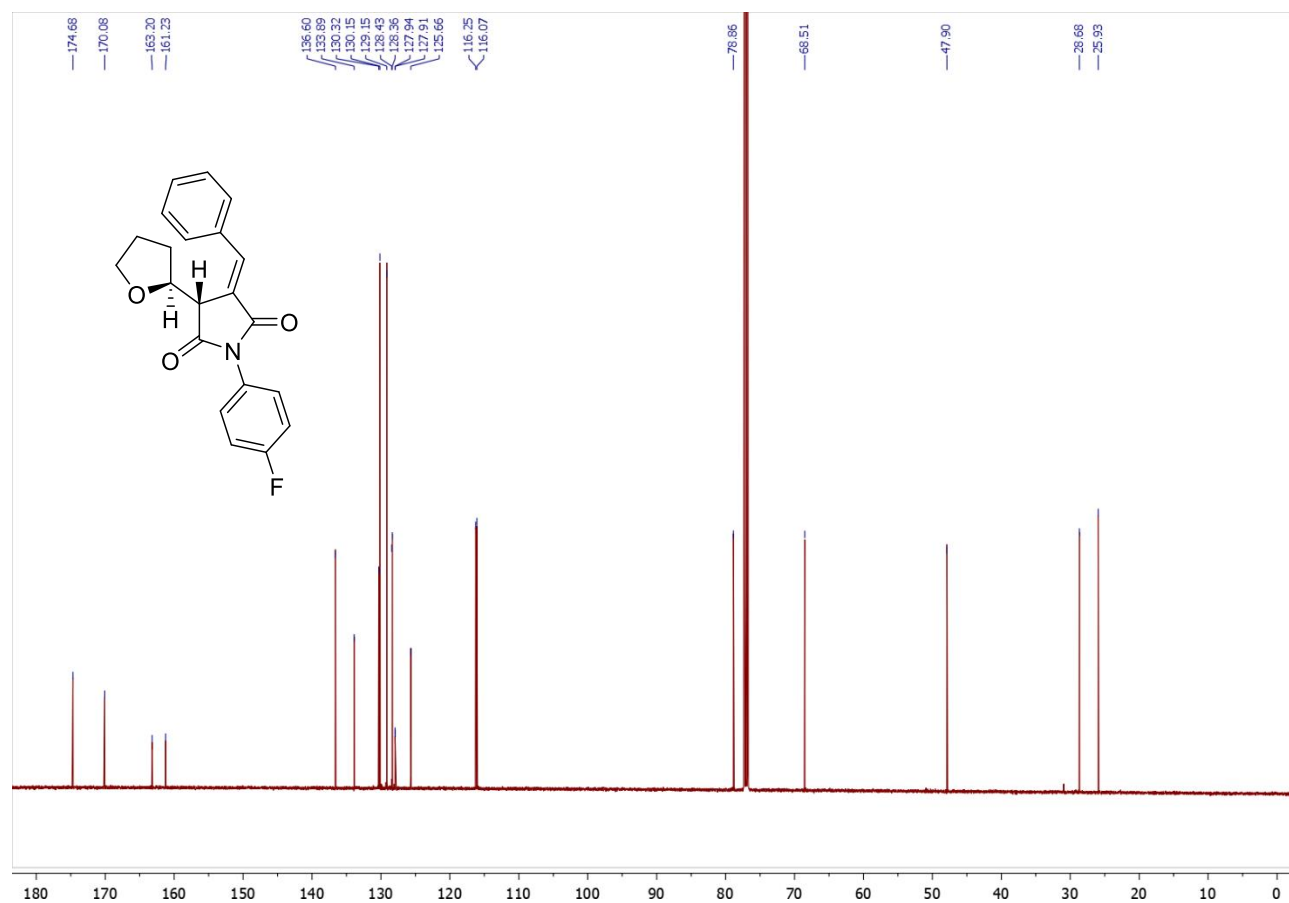
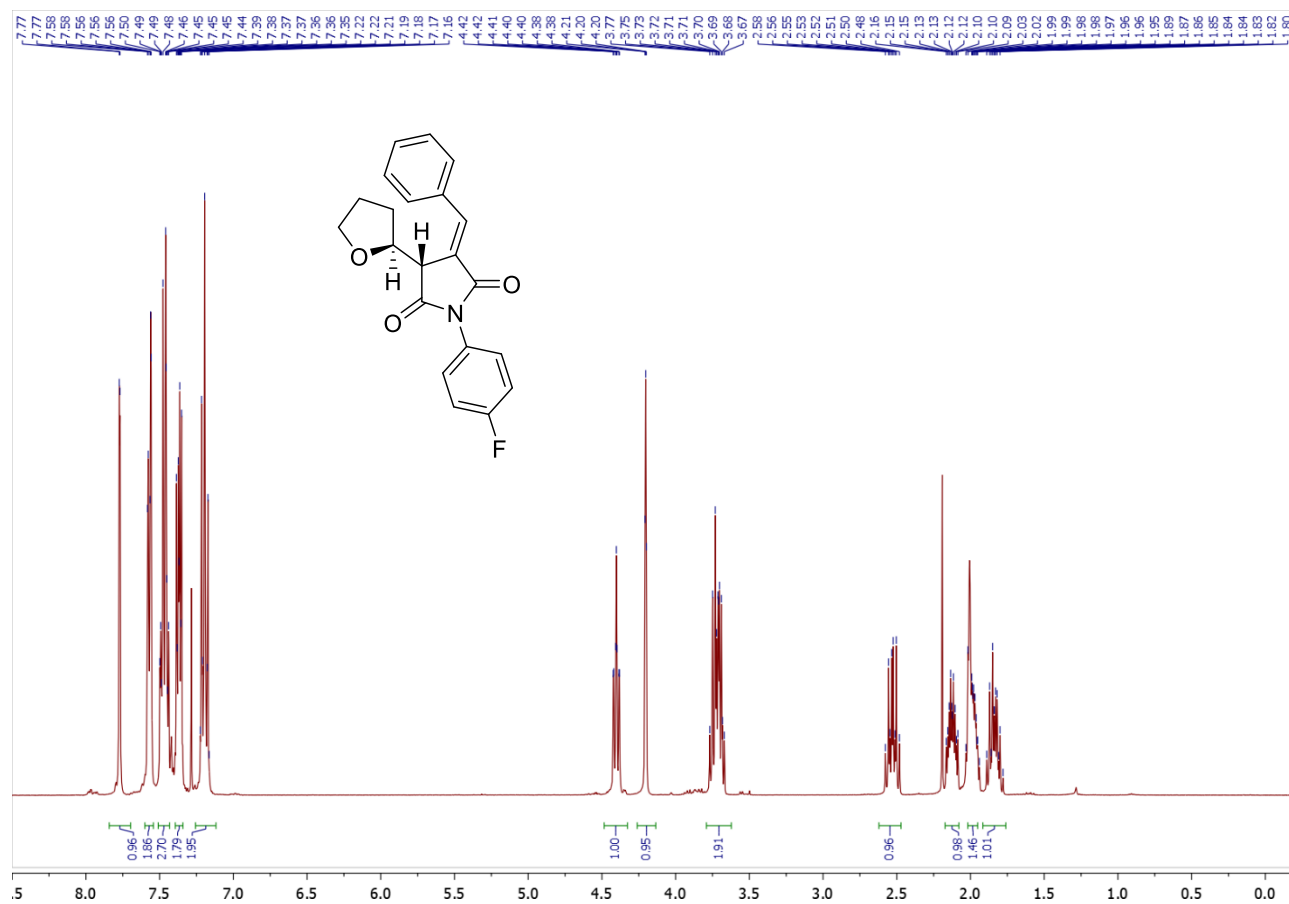
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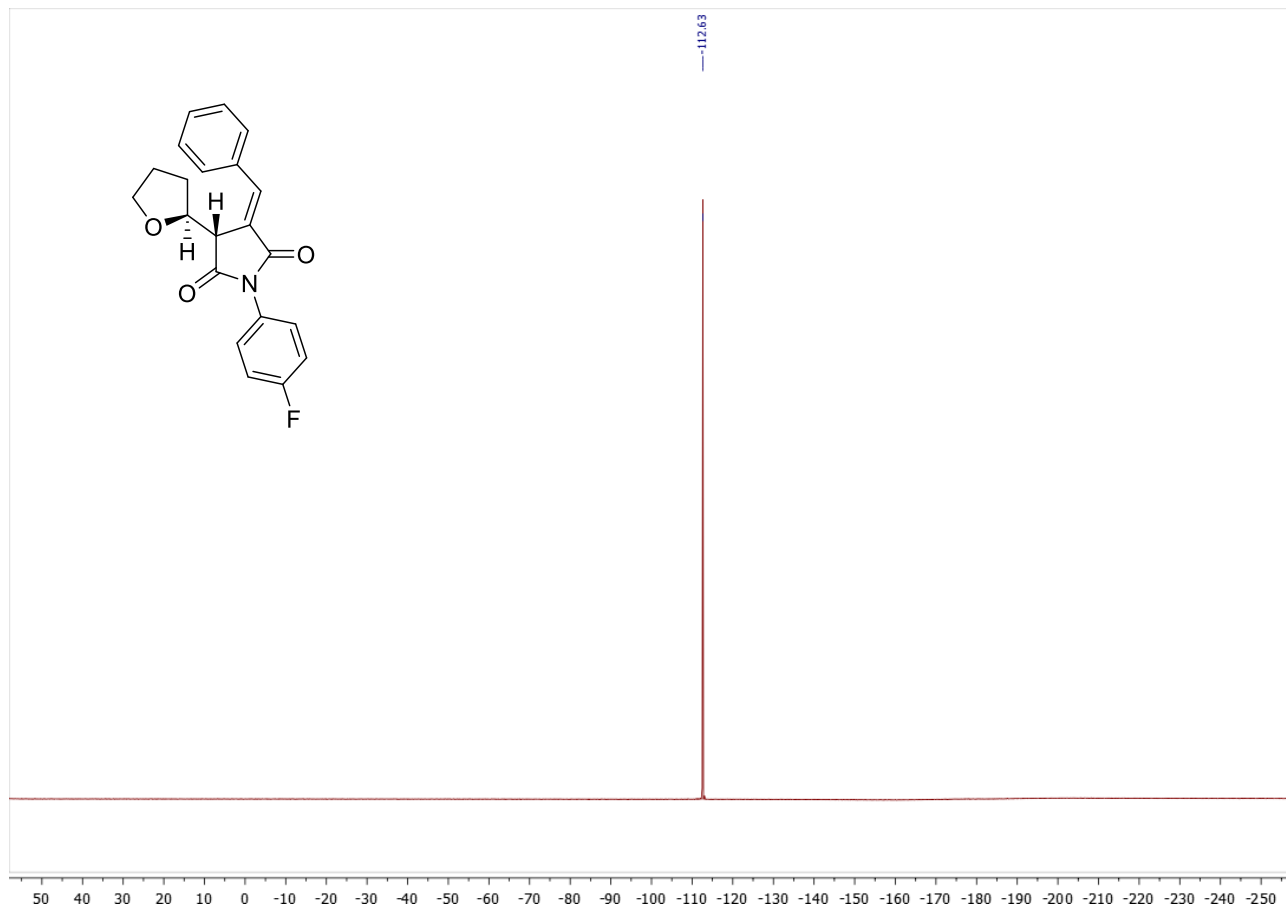
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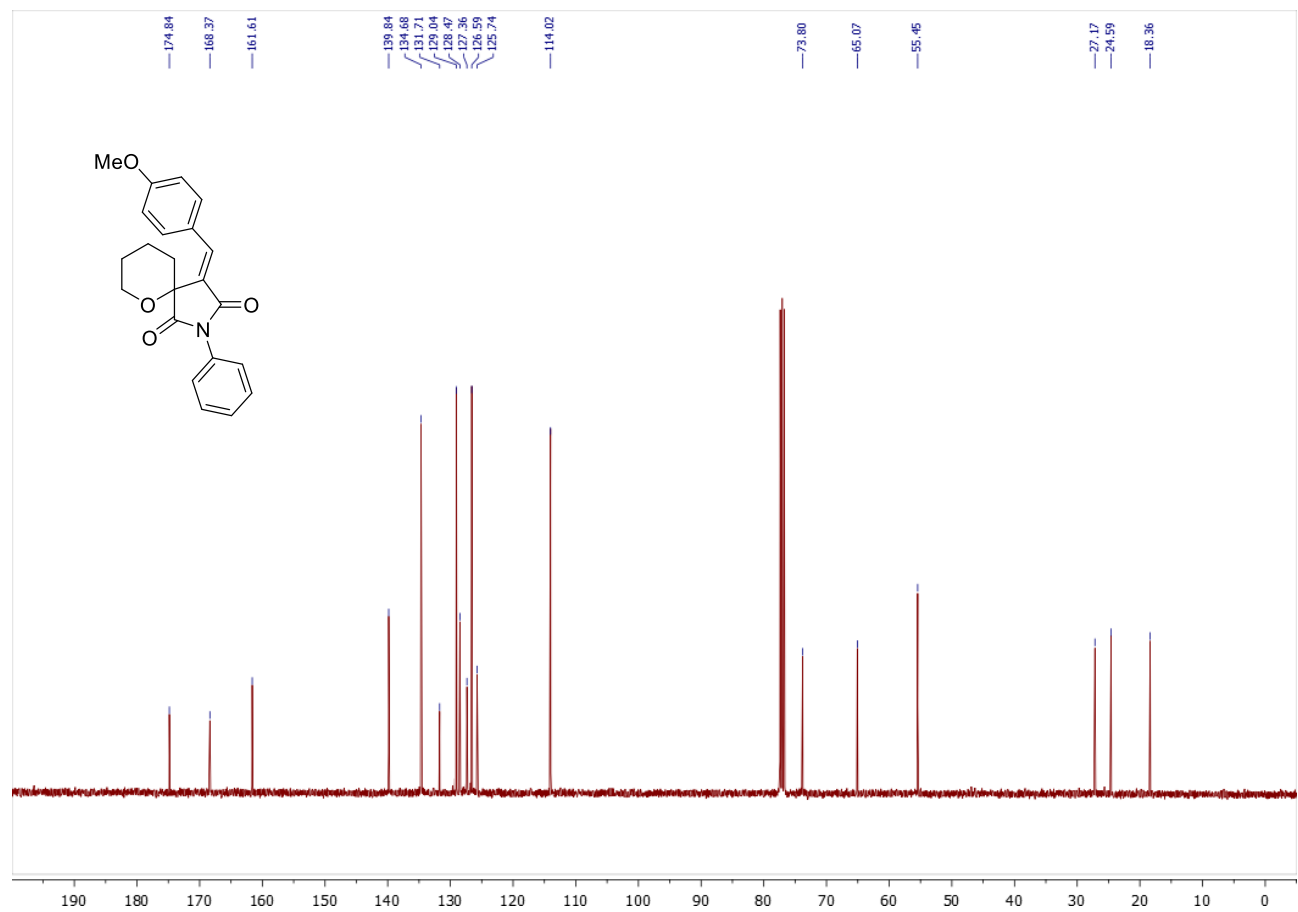
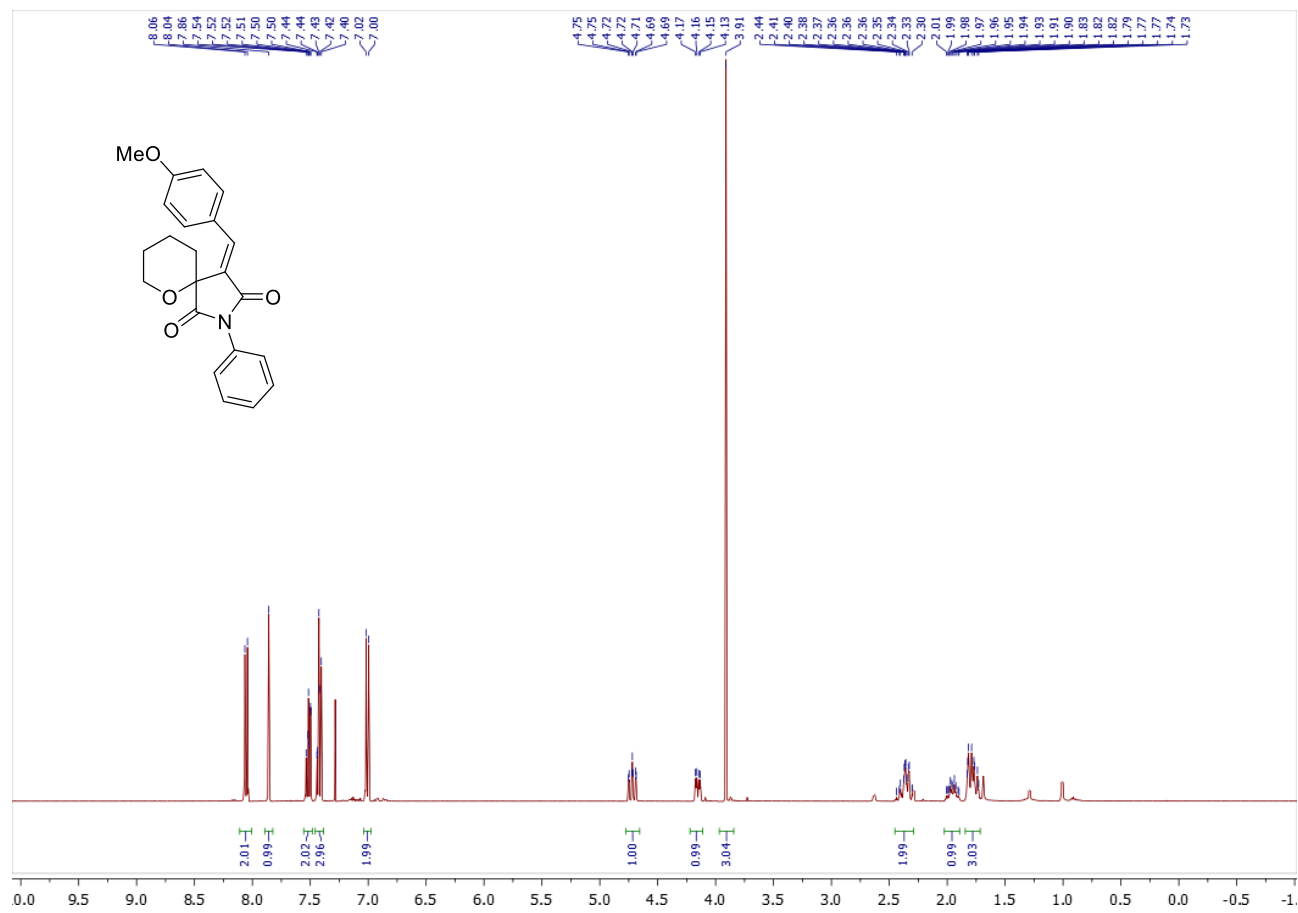
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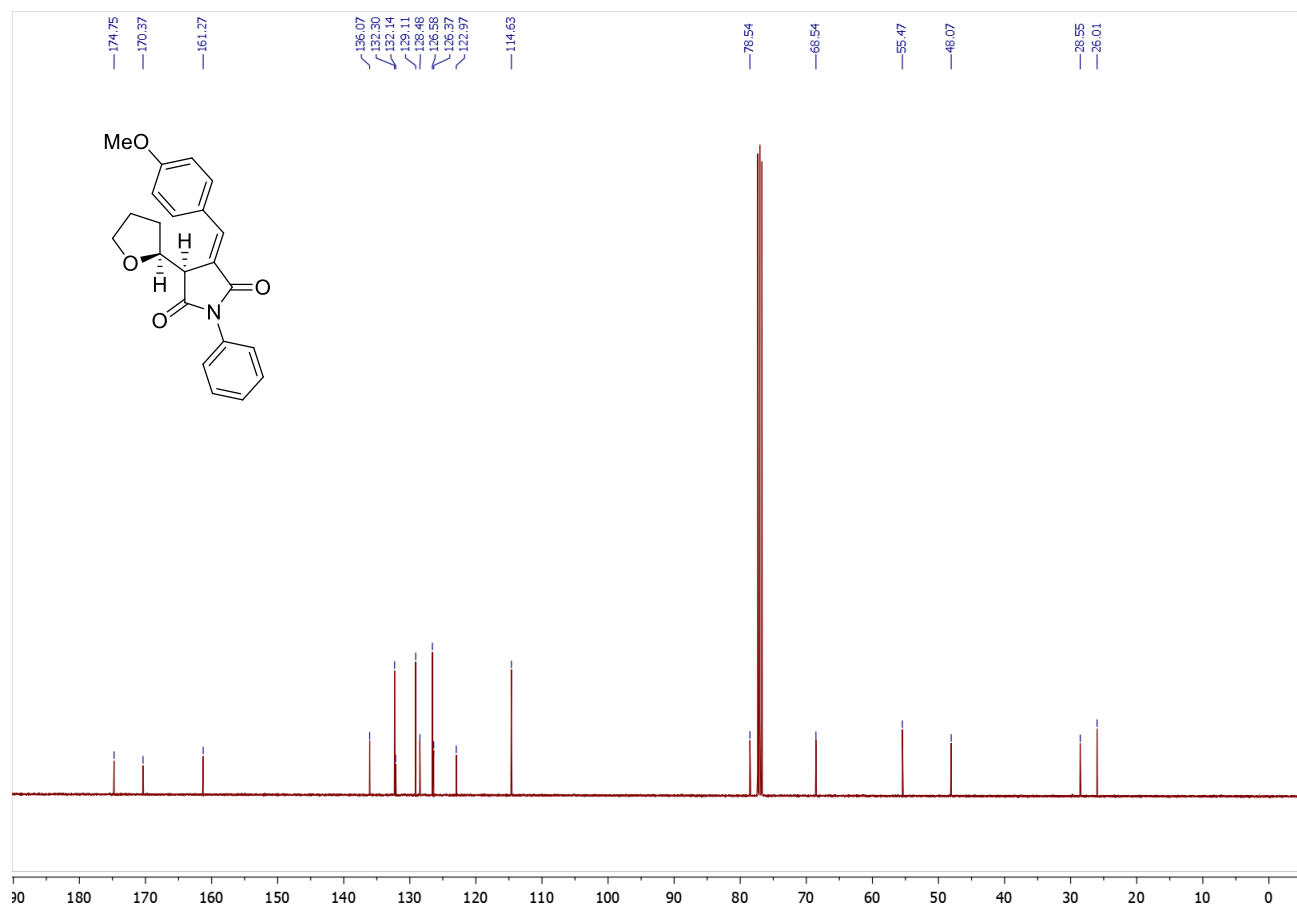
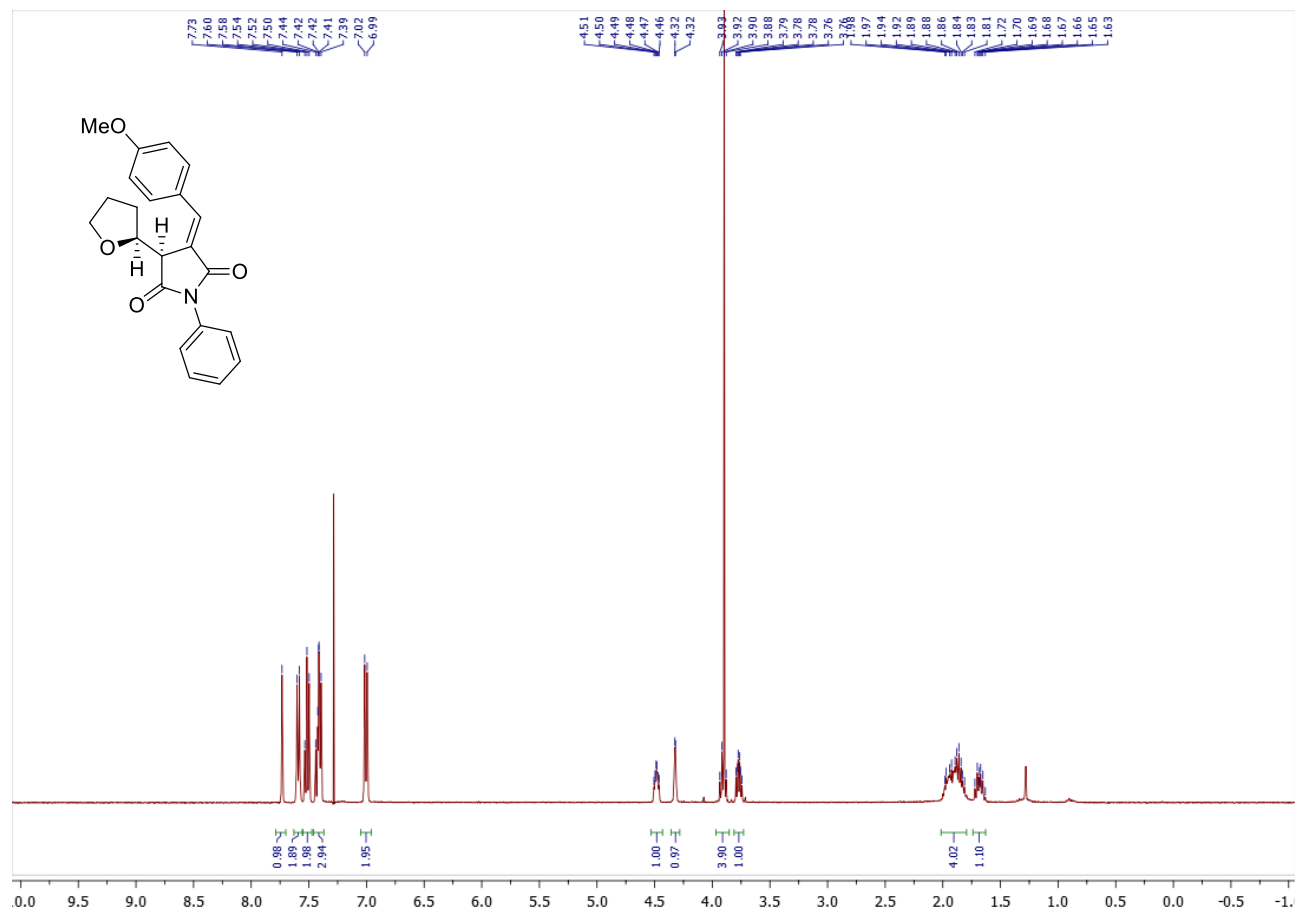
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of $(S/R,S/R)$ -6a^{maj}



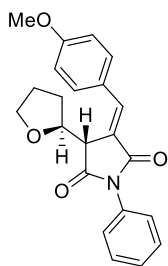
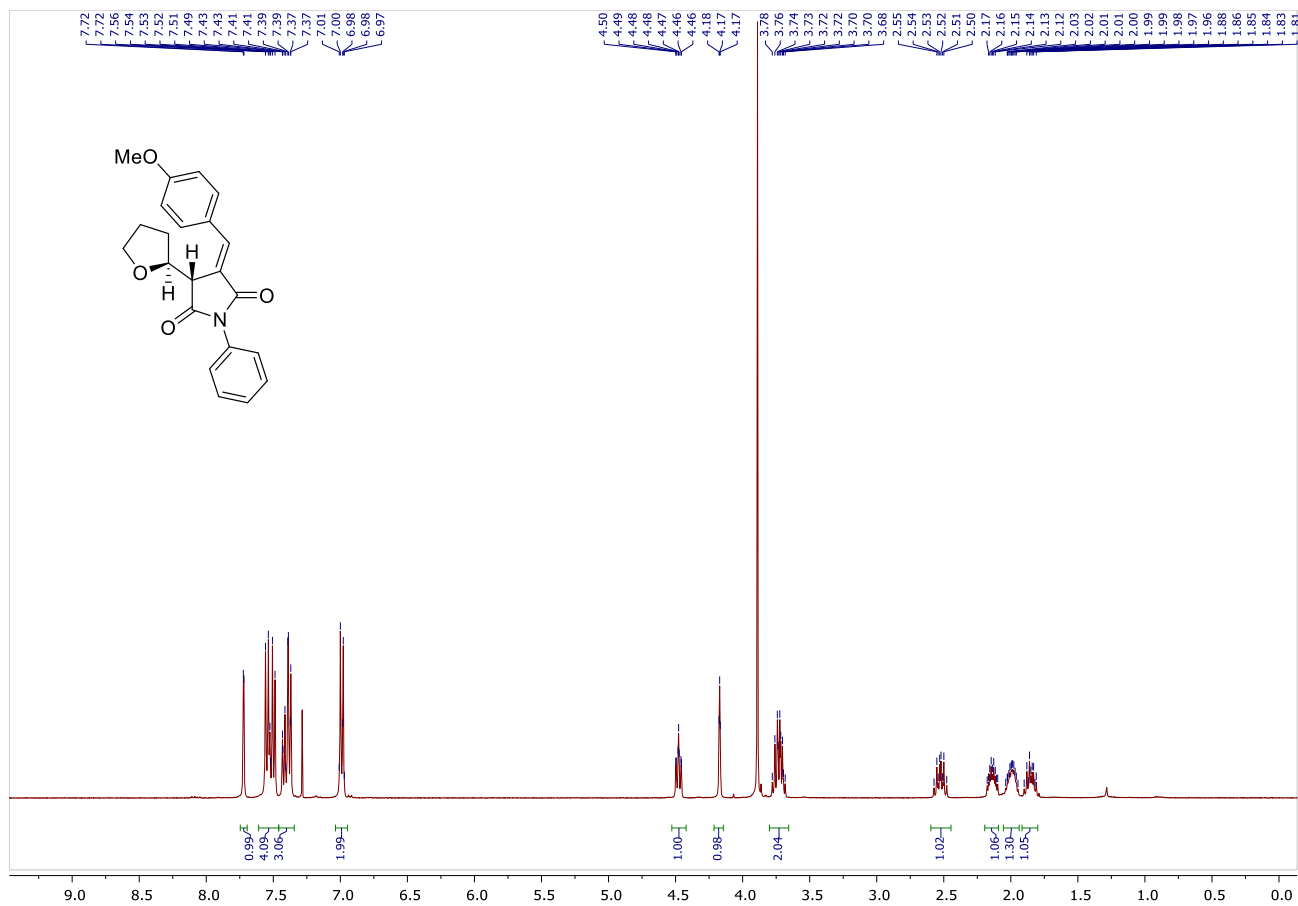
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2b**

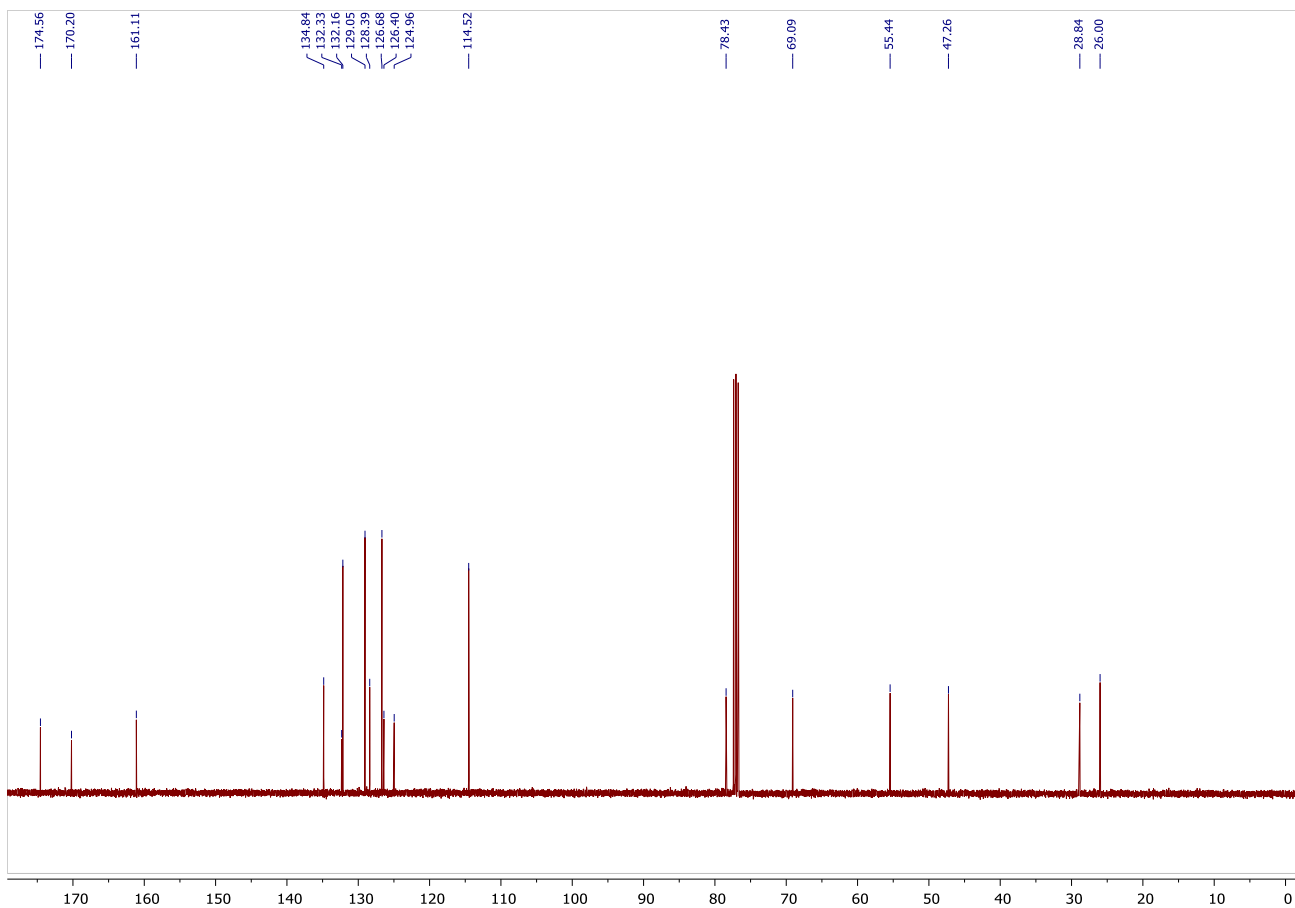


Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*R/S,S/R*)-6b^{min}

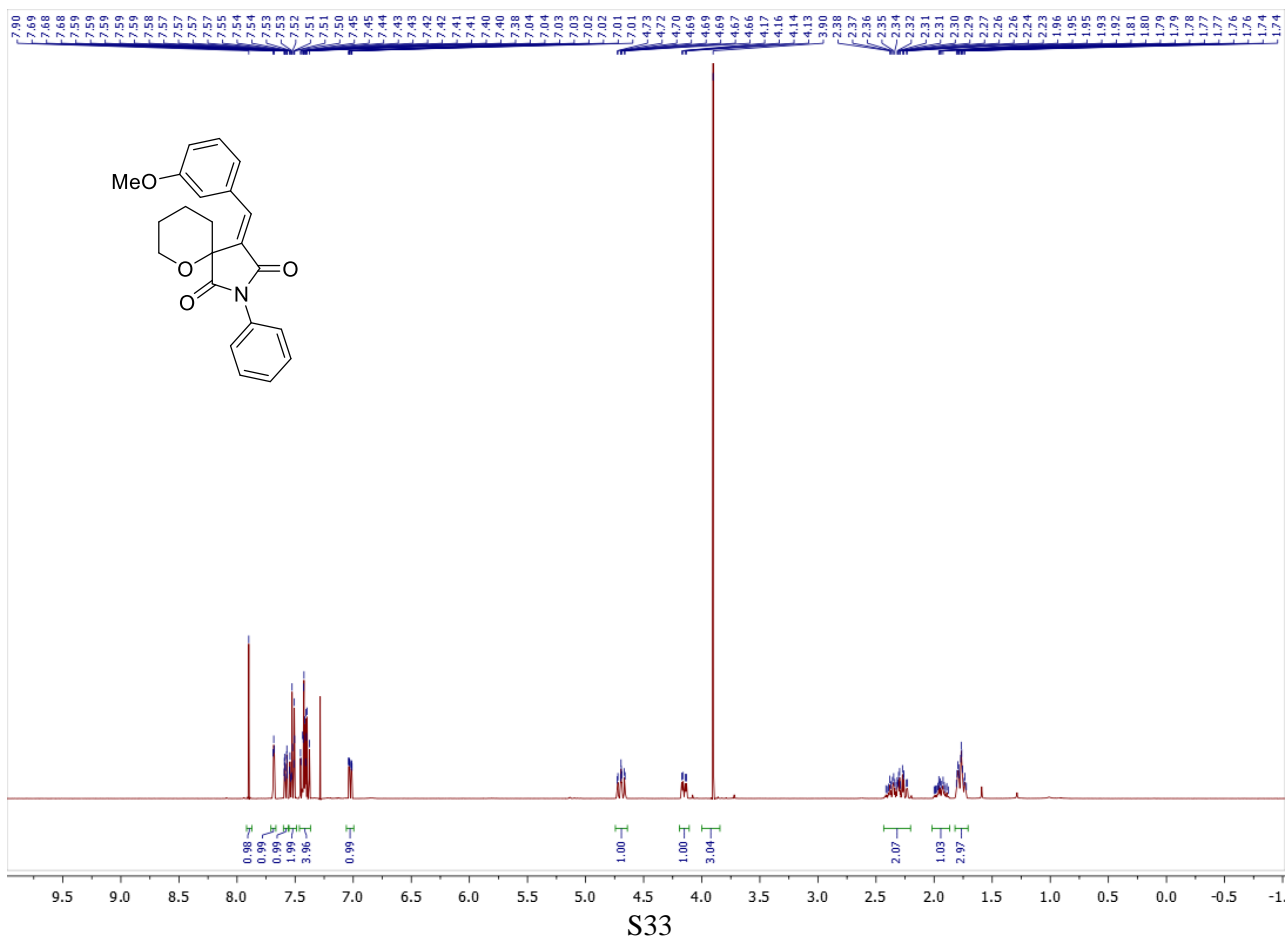


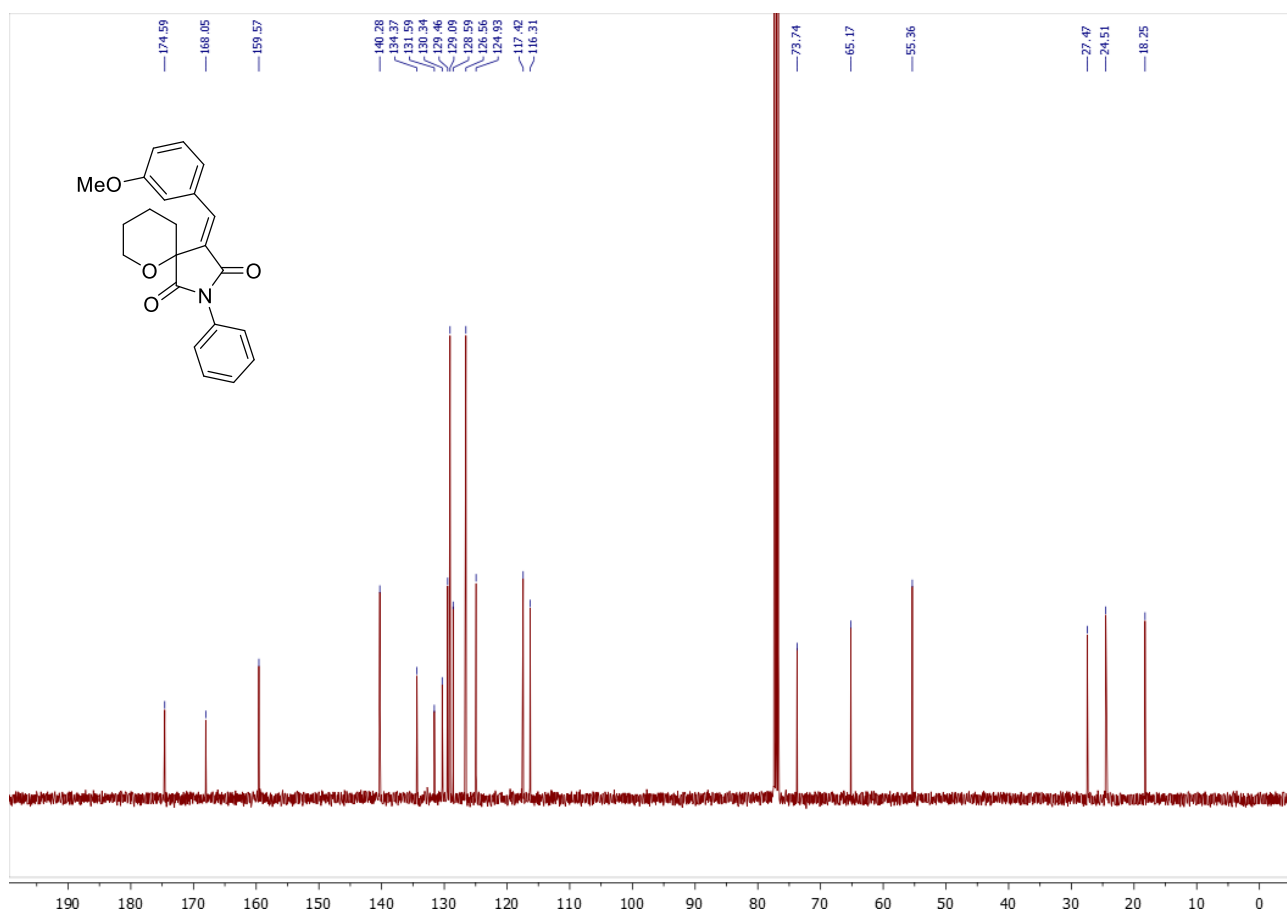
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*S/R,S/R*)-**6b**^{maj}



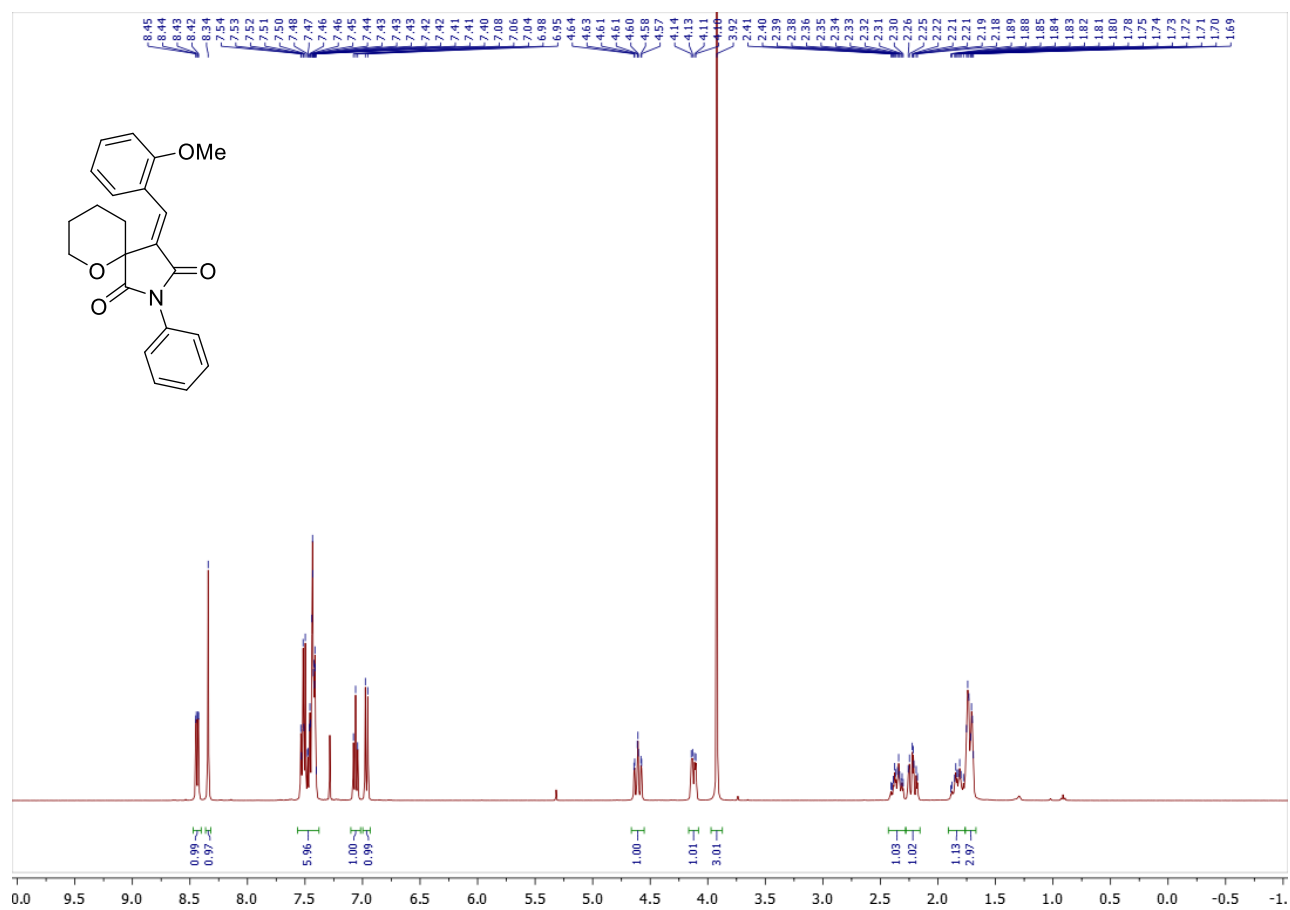


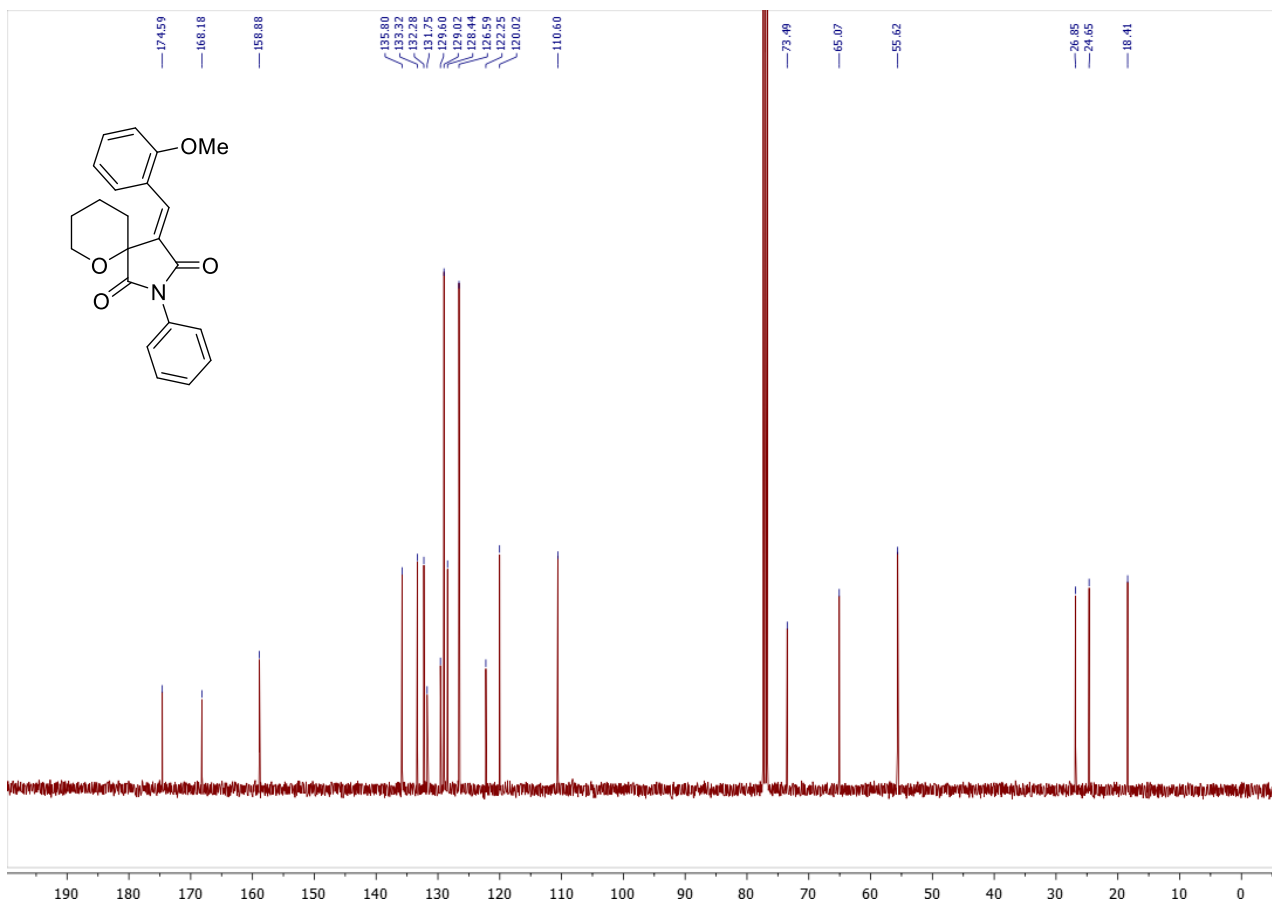
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2c**



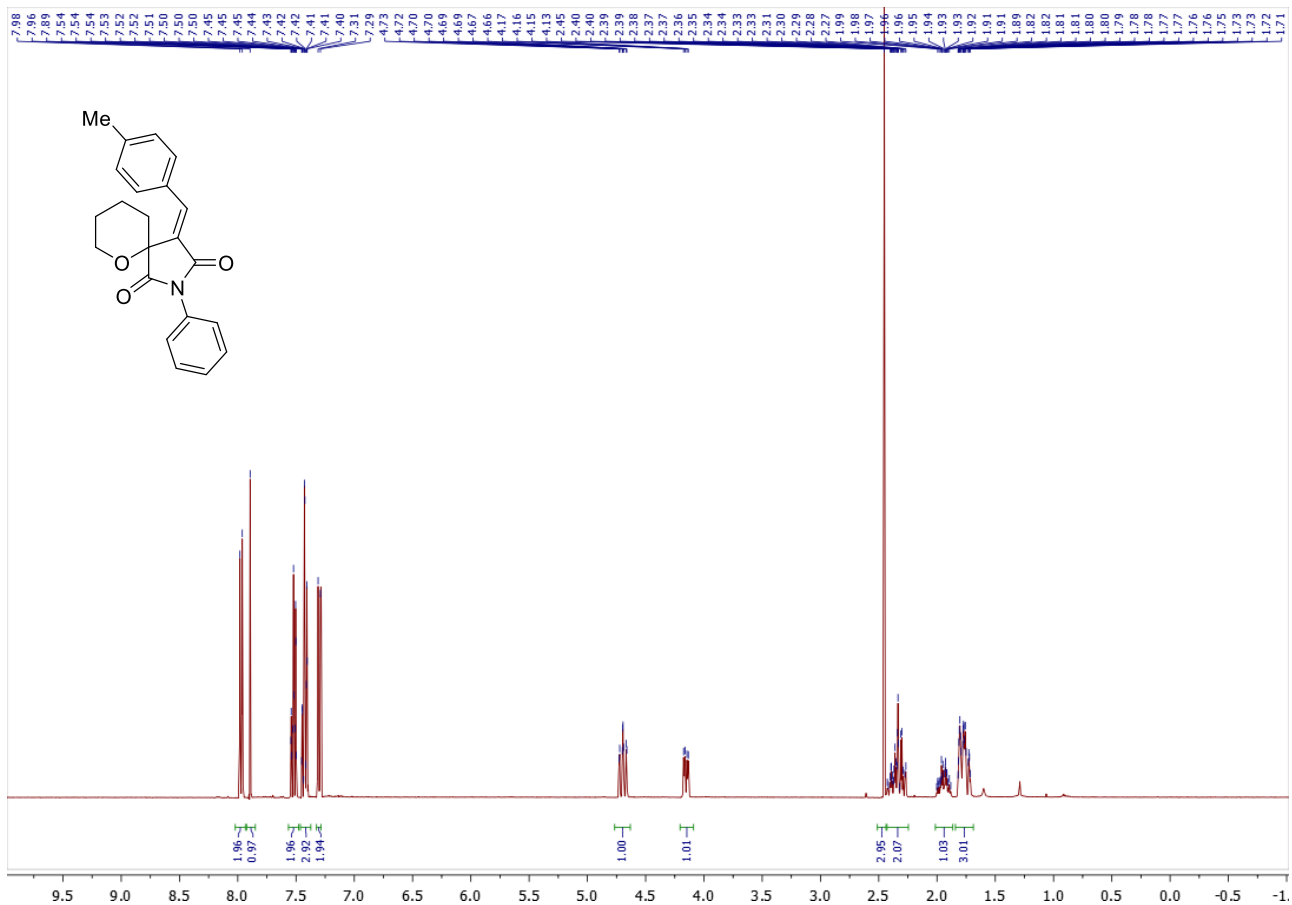


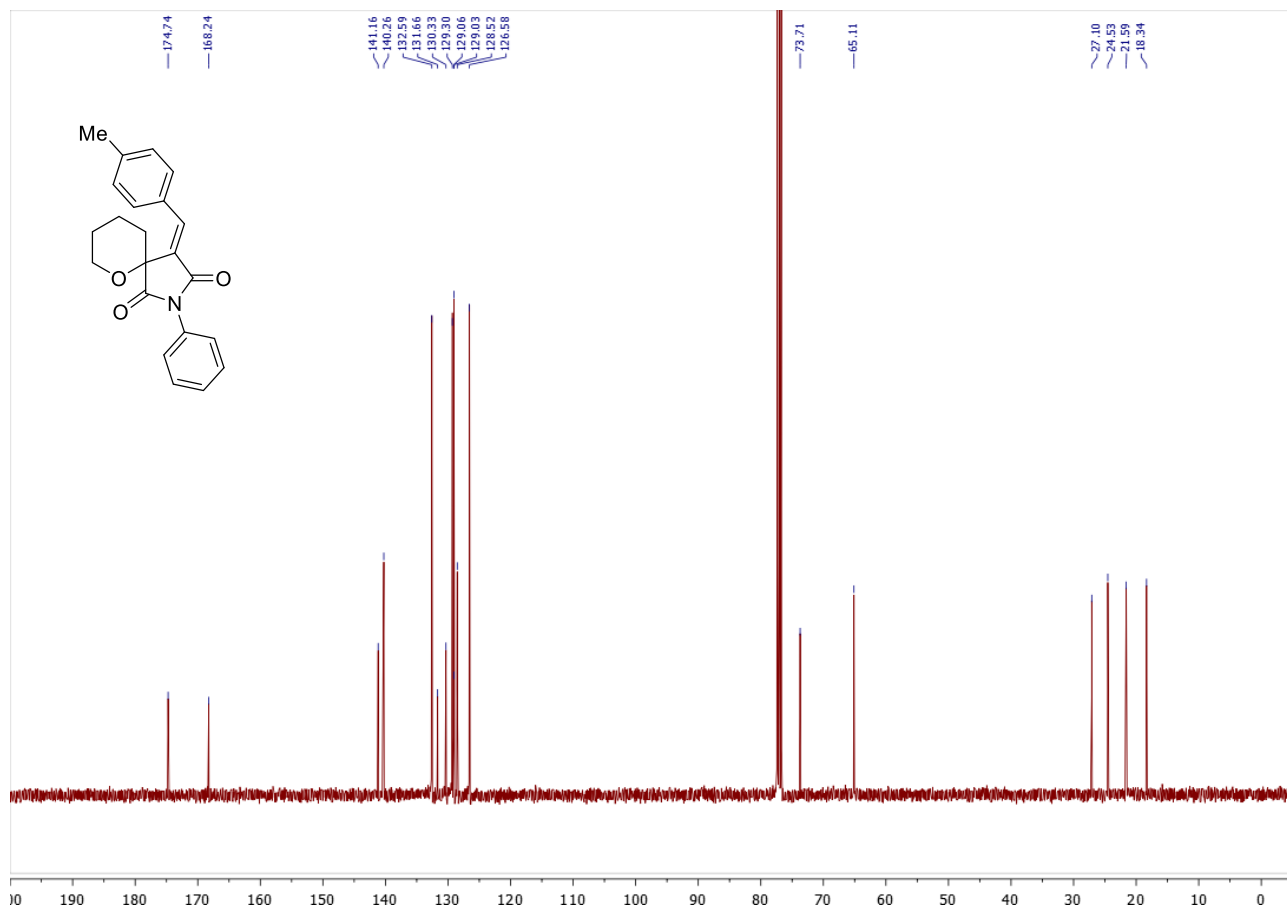
Copies of ¹H (400.13 MHz, CDCl₃) and ¹³C (100.61 MHz, CDCl₃) spectra of **2d**



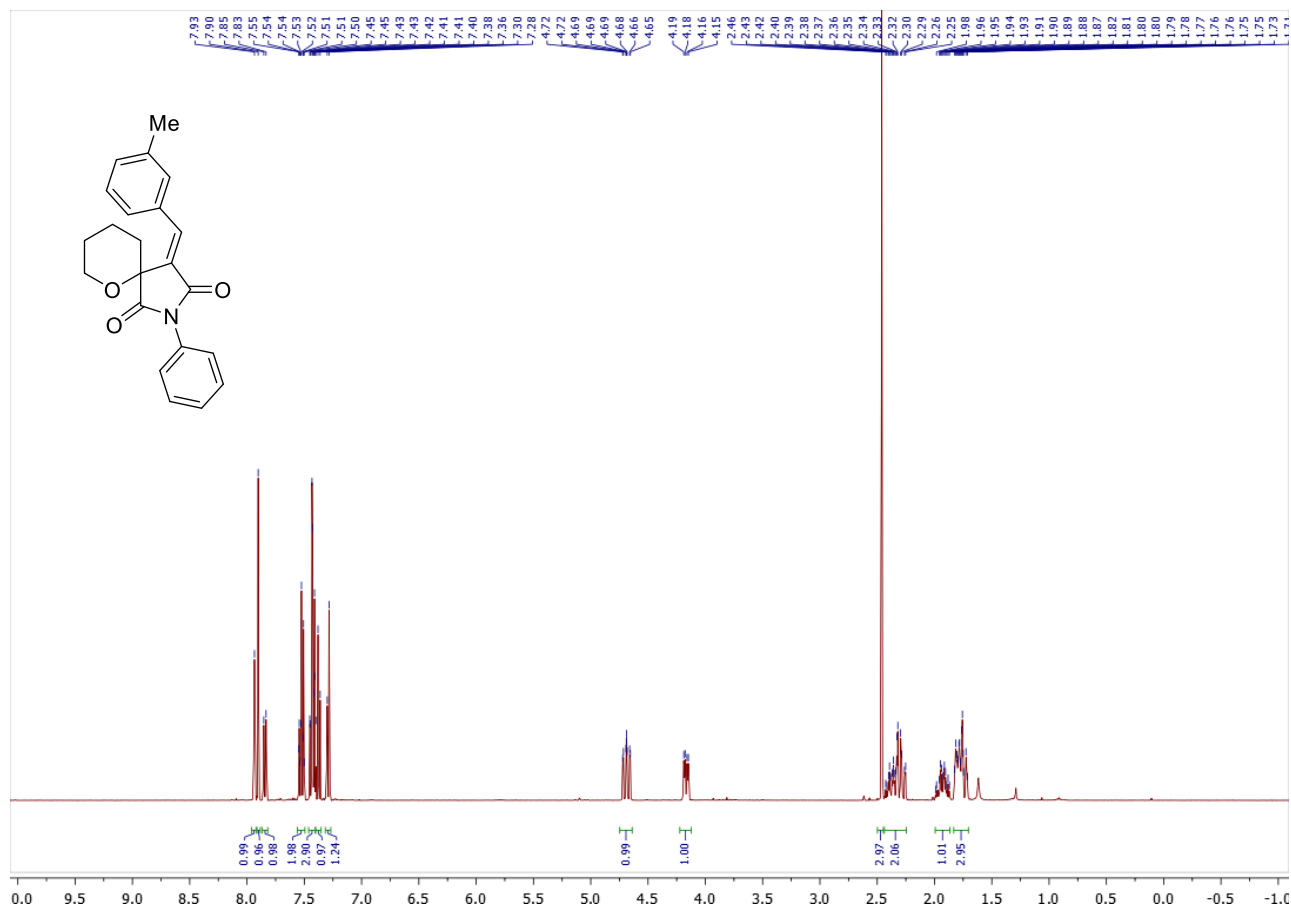


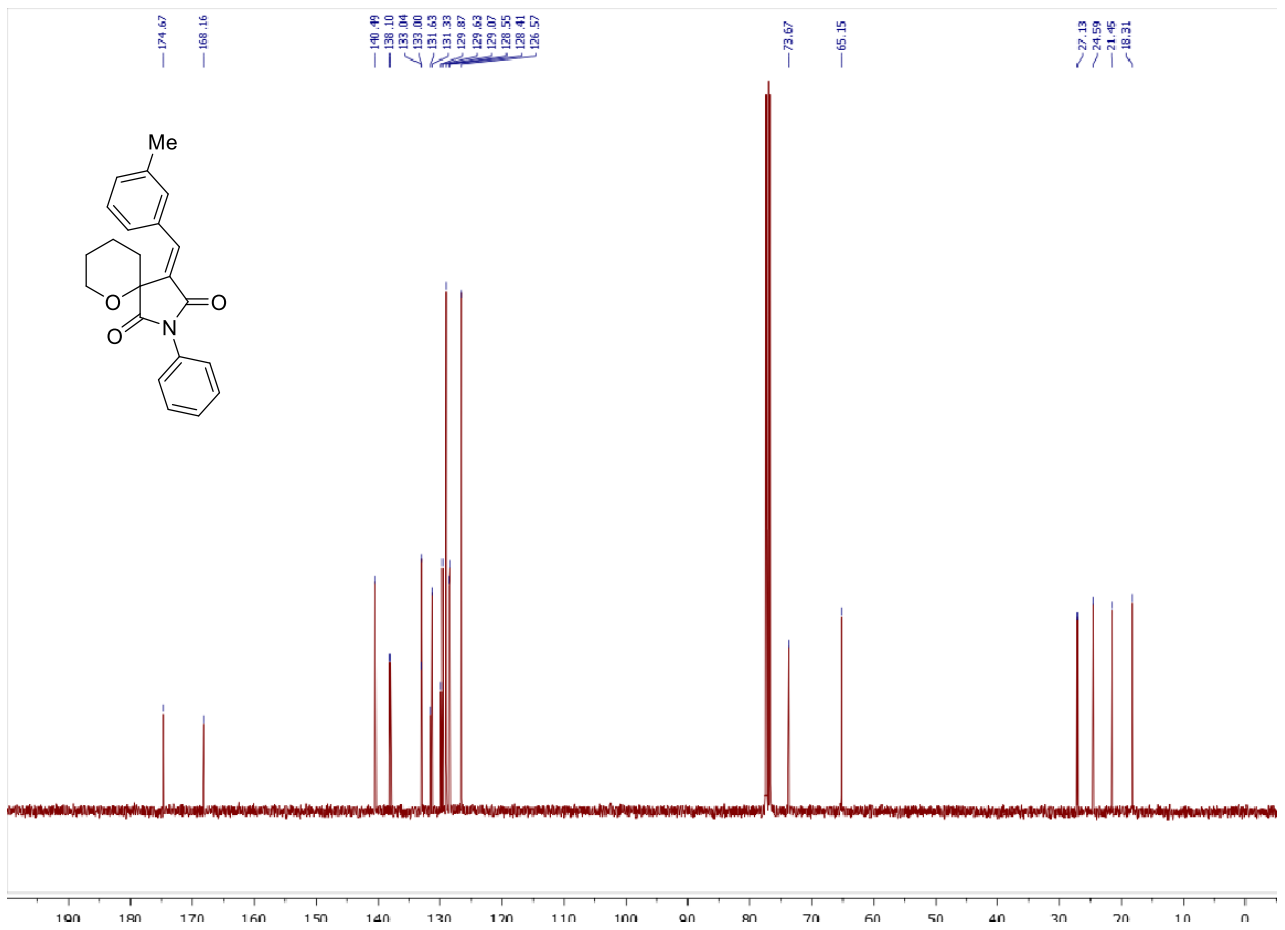
Copies of ¹H (400.13 MHz, CDCl₃) and ¹³C (100.61 MHz, CDCl₃) spectra of 2e



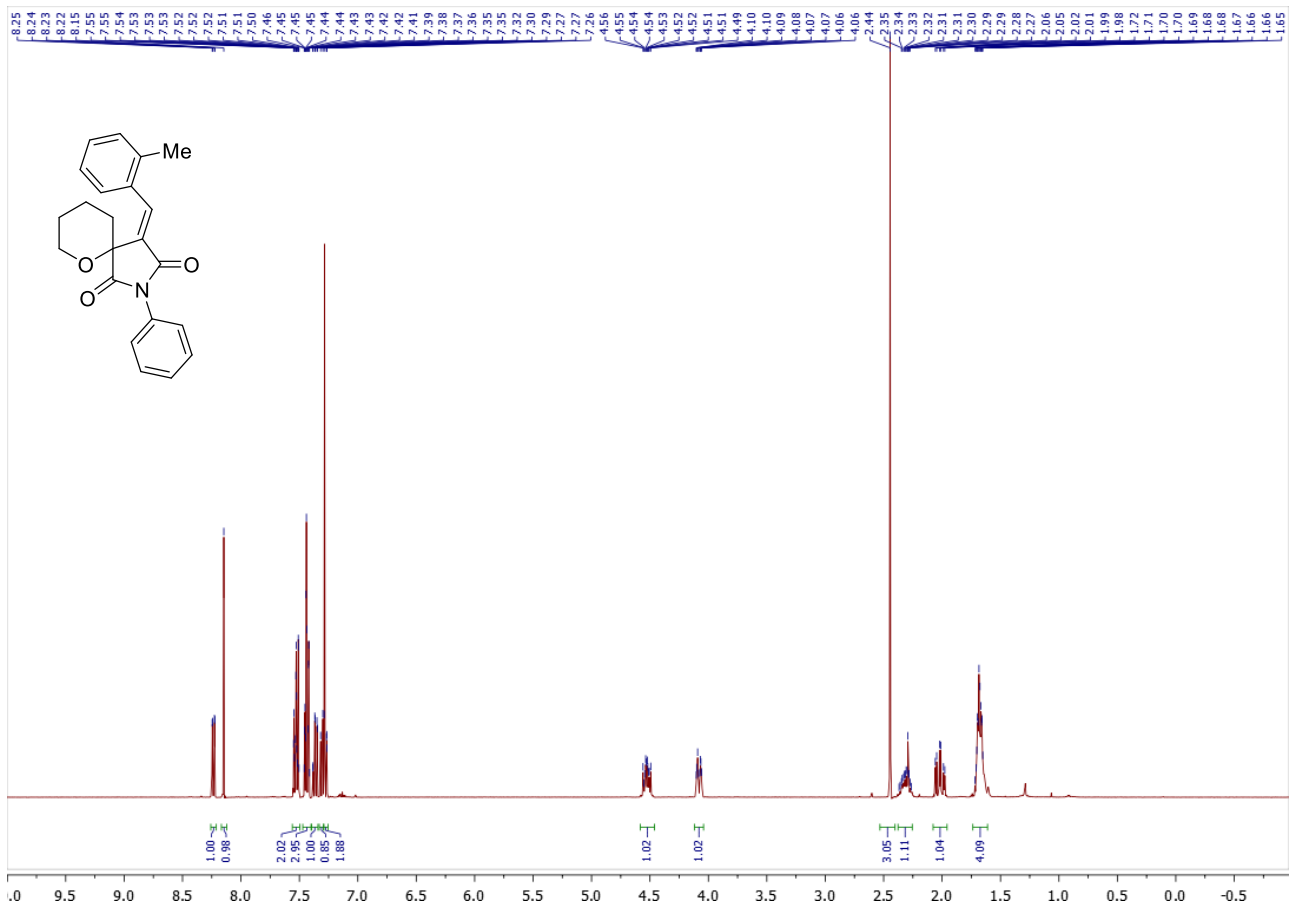


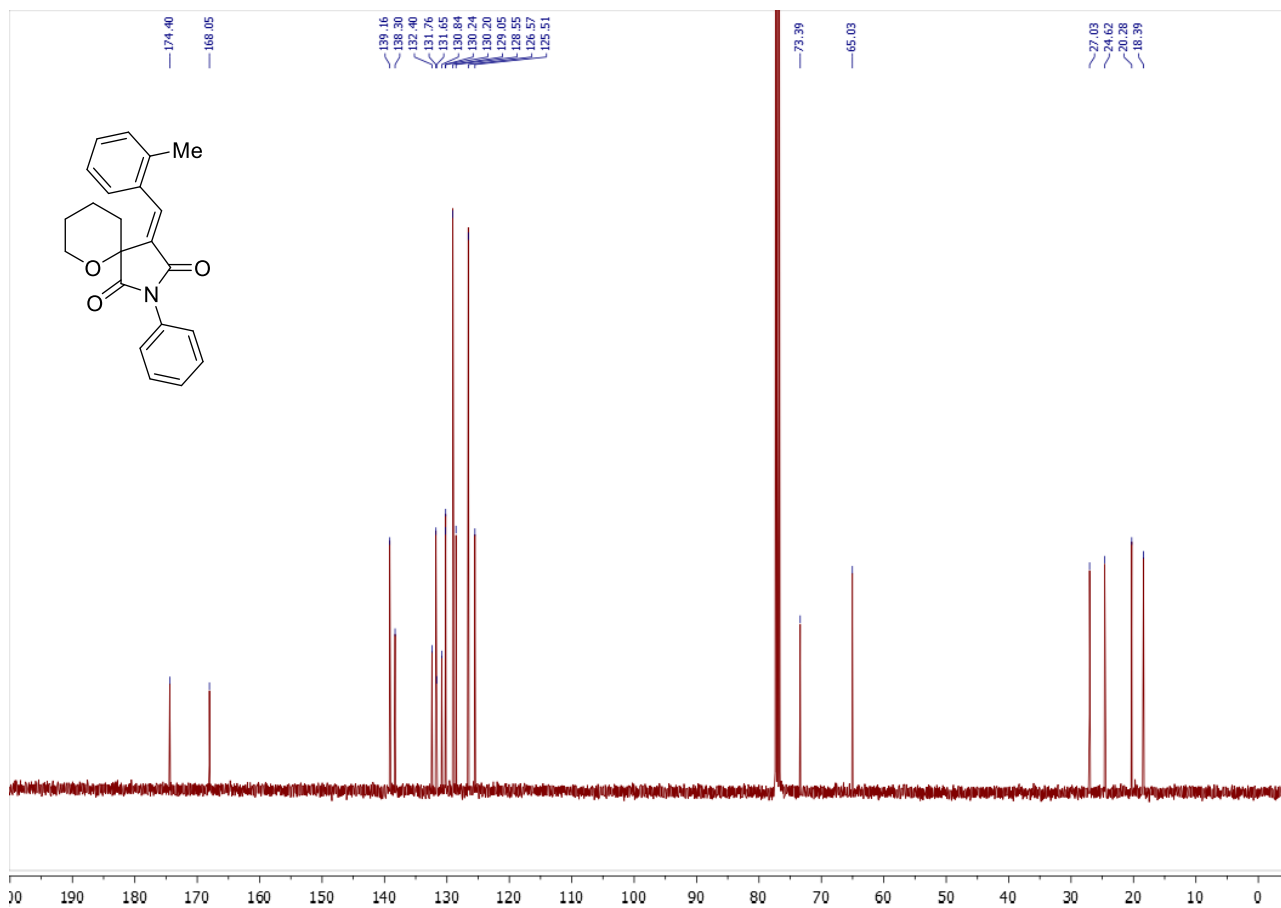
Copies of ¹H (400.13 MHz, CDCl₃) and ¹³C (100.61 MHz, CDCl₃) spectra of **2f**



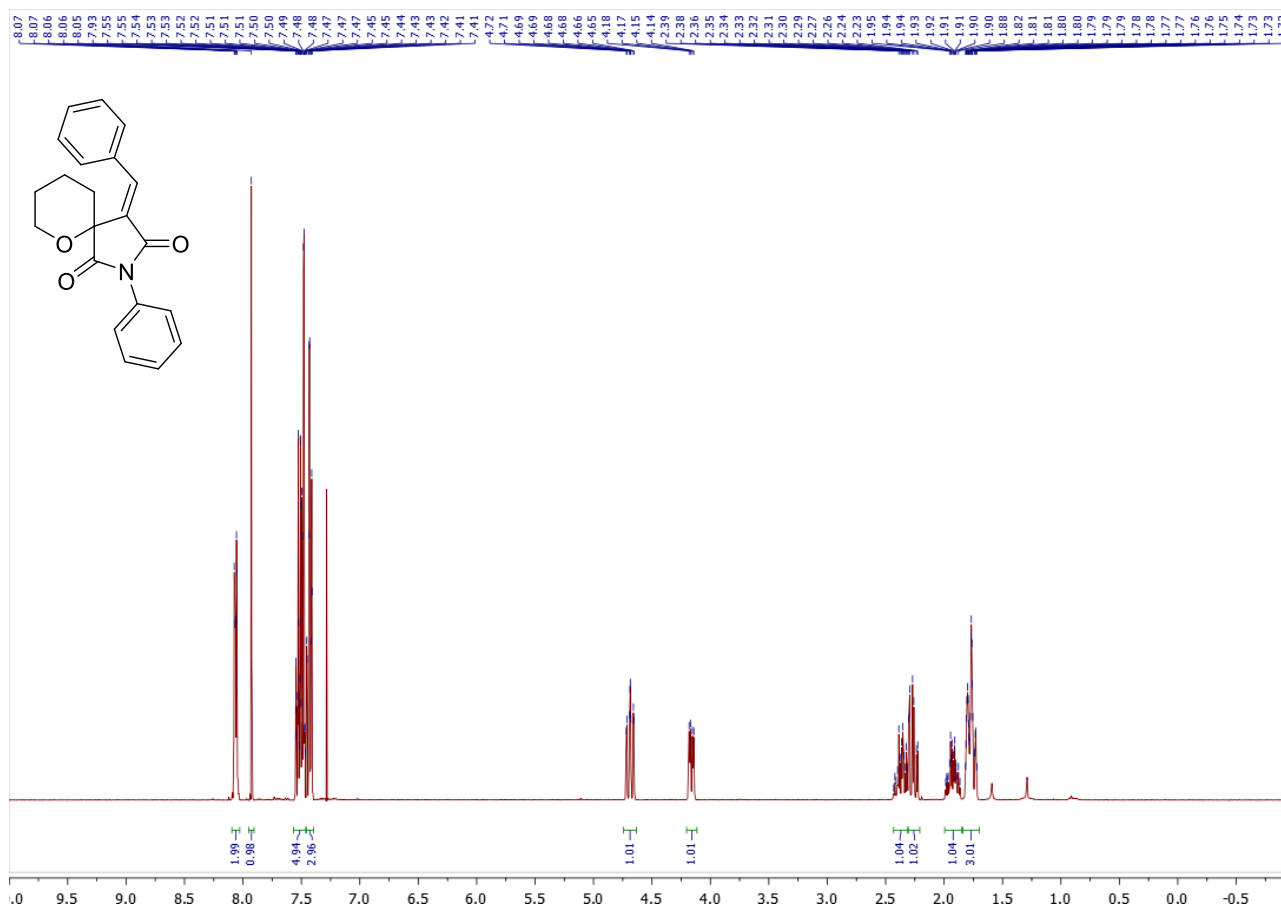


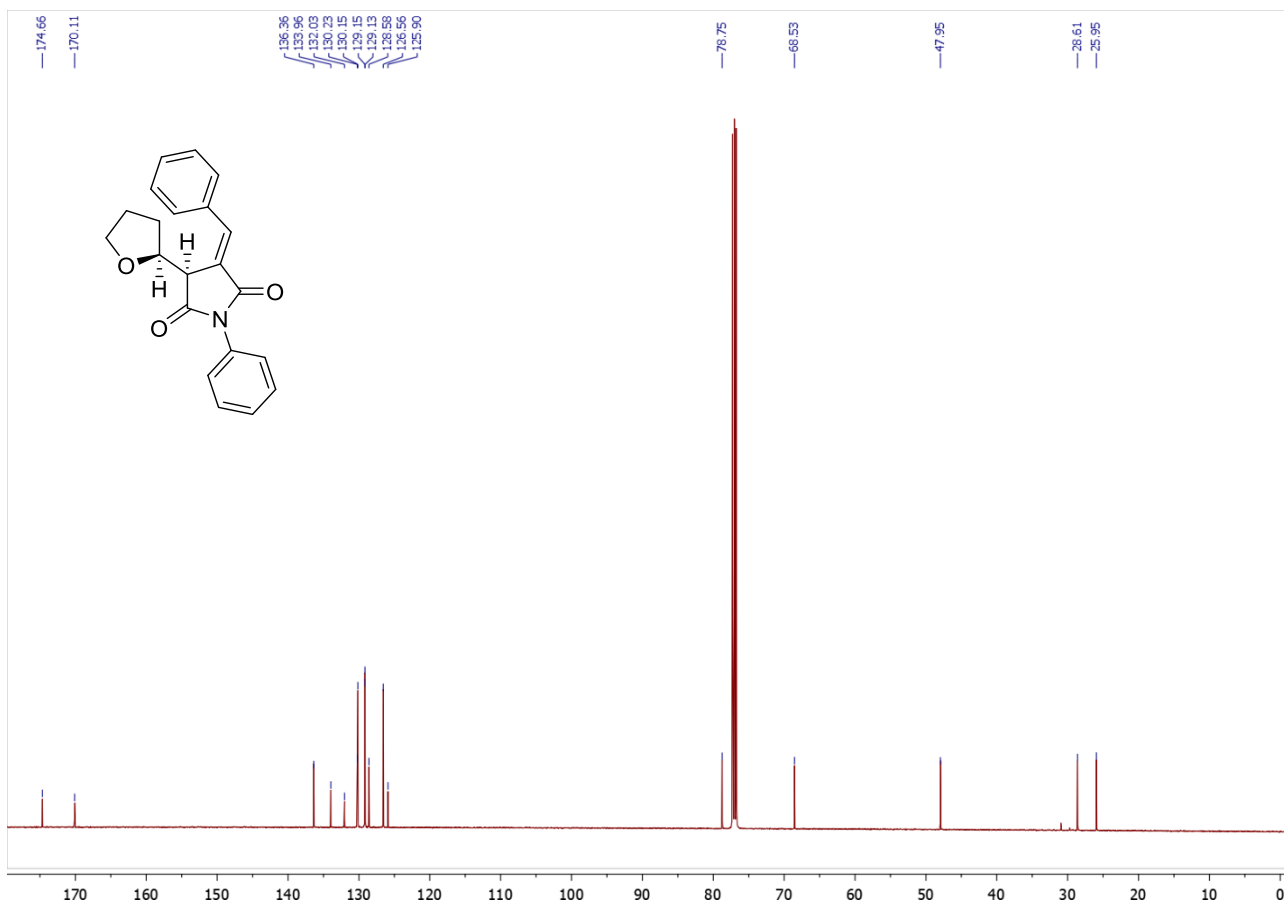
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2g**



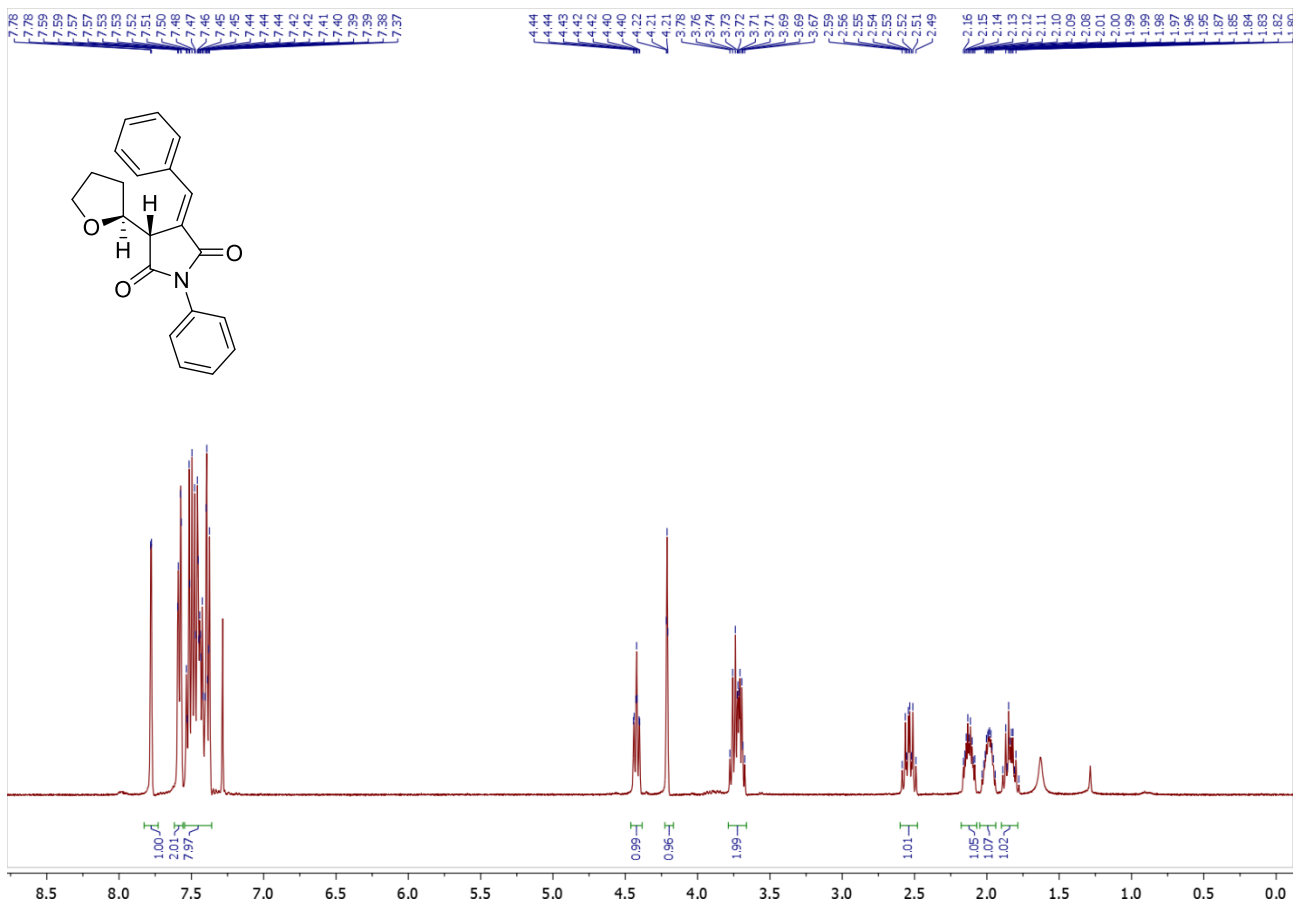


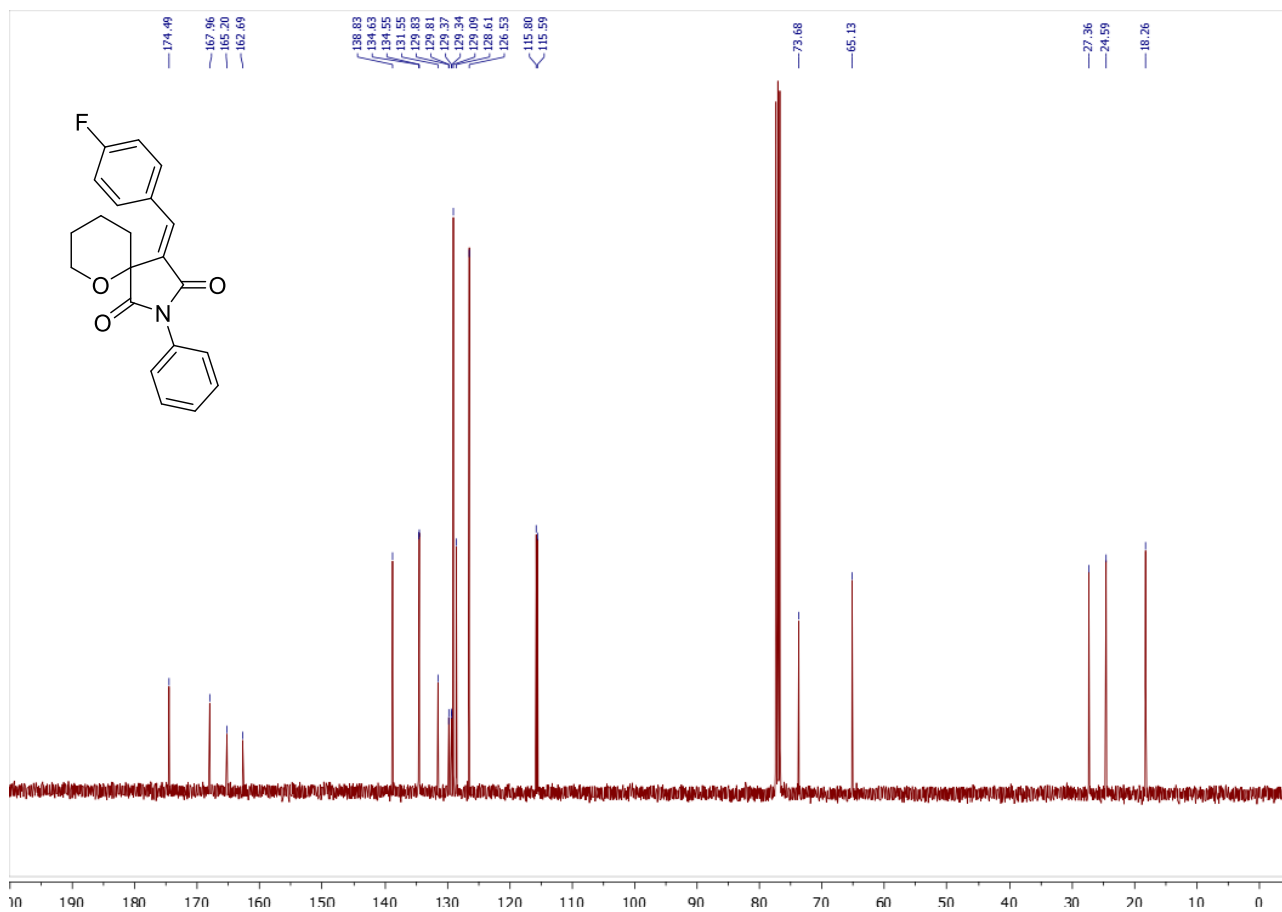
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2h**



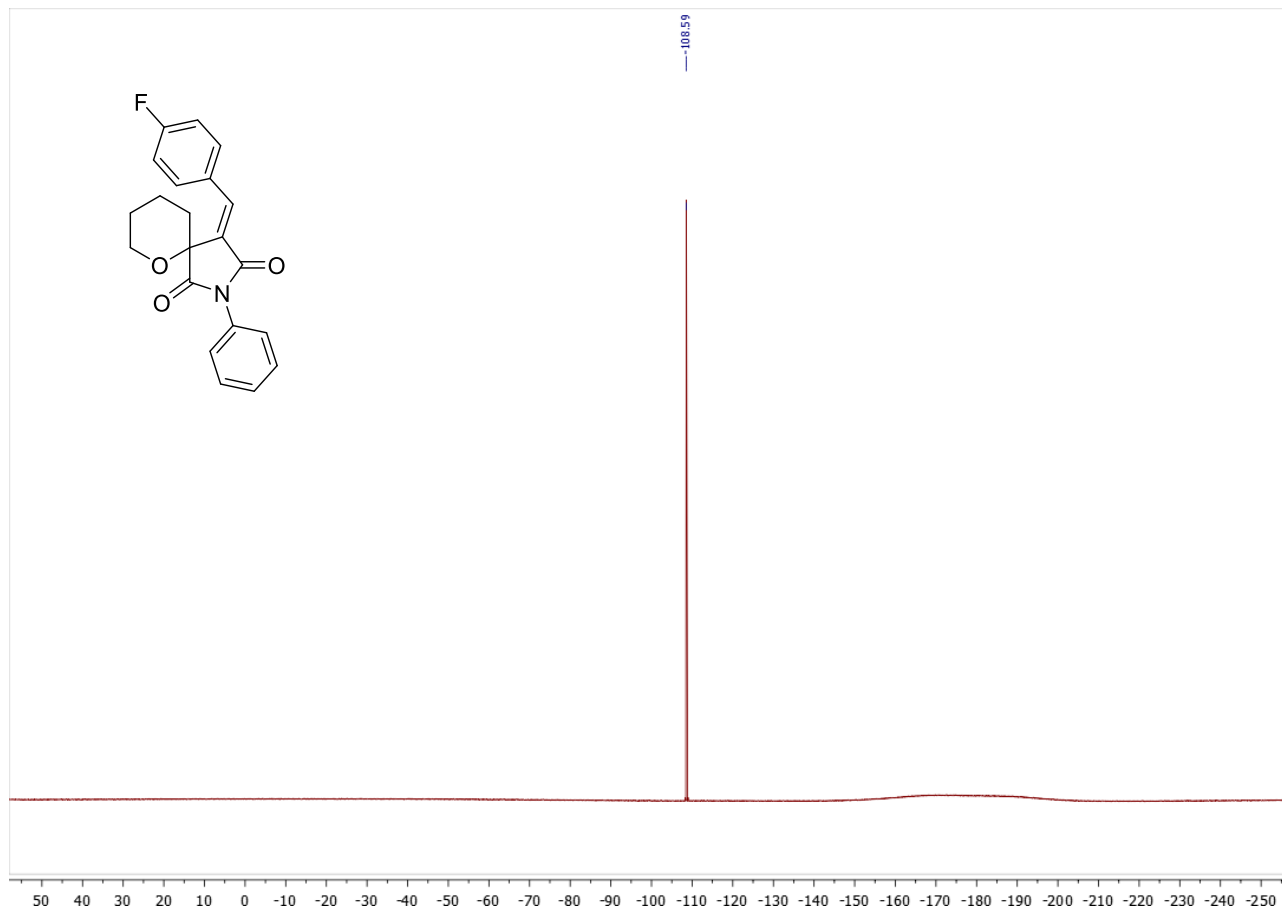


Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of $(S/R,S/R)$ -6h^{maj}

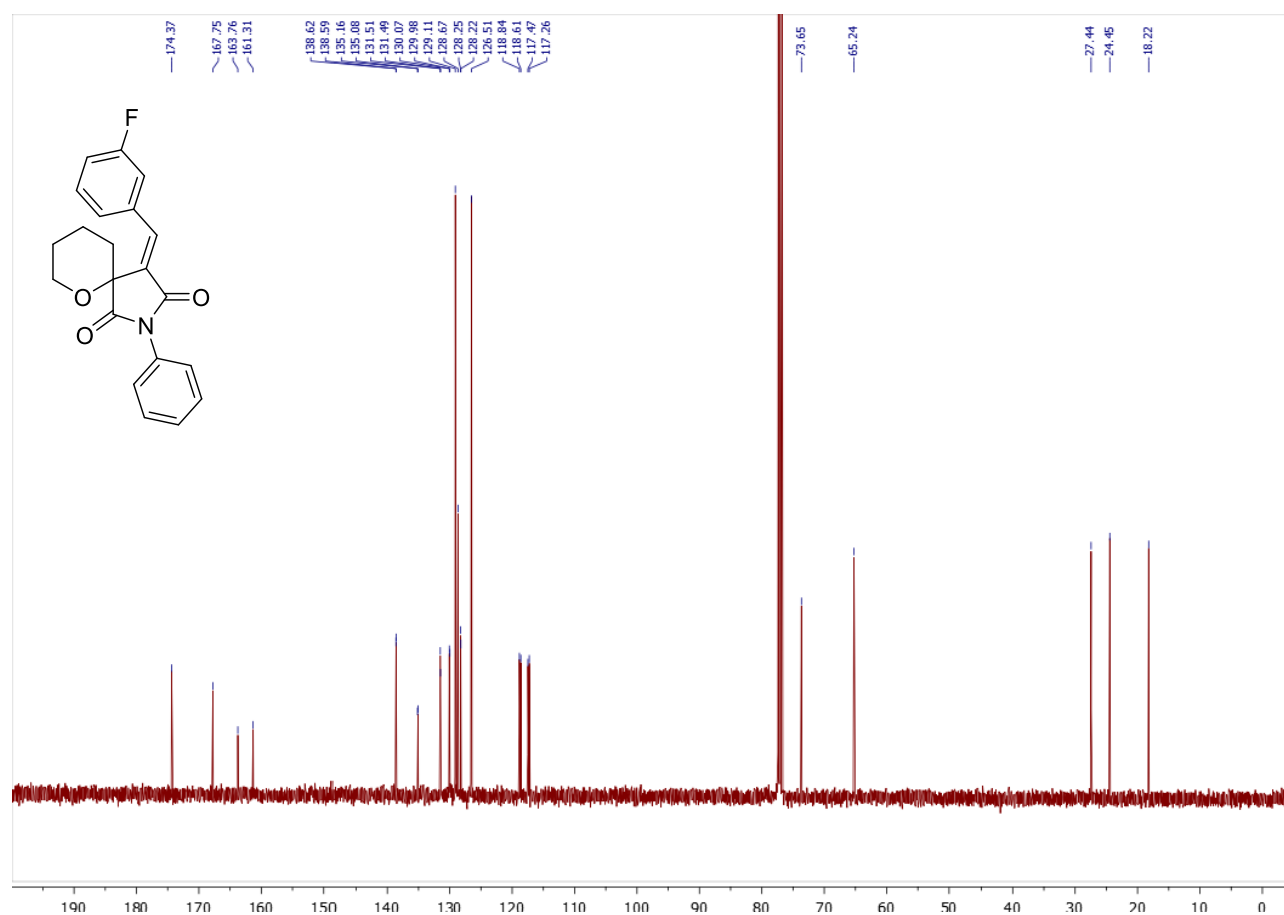
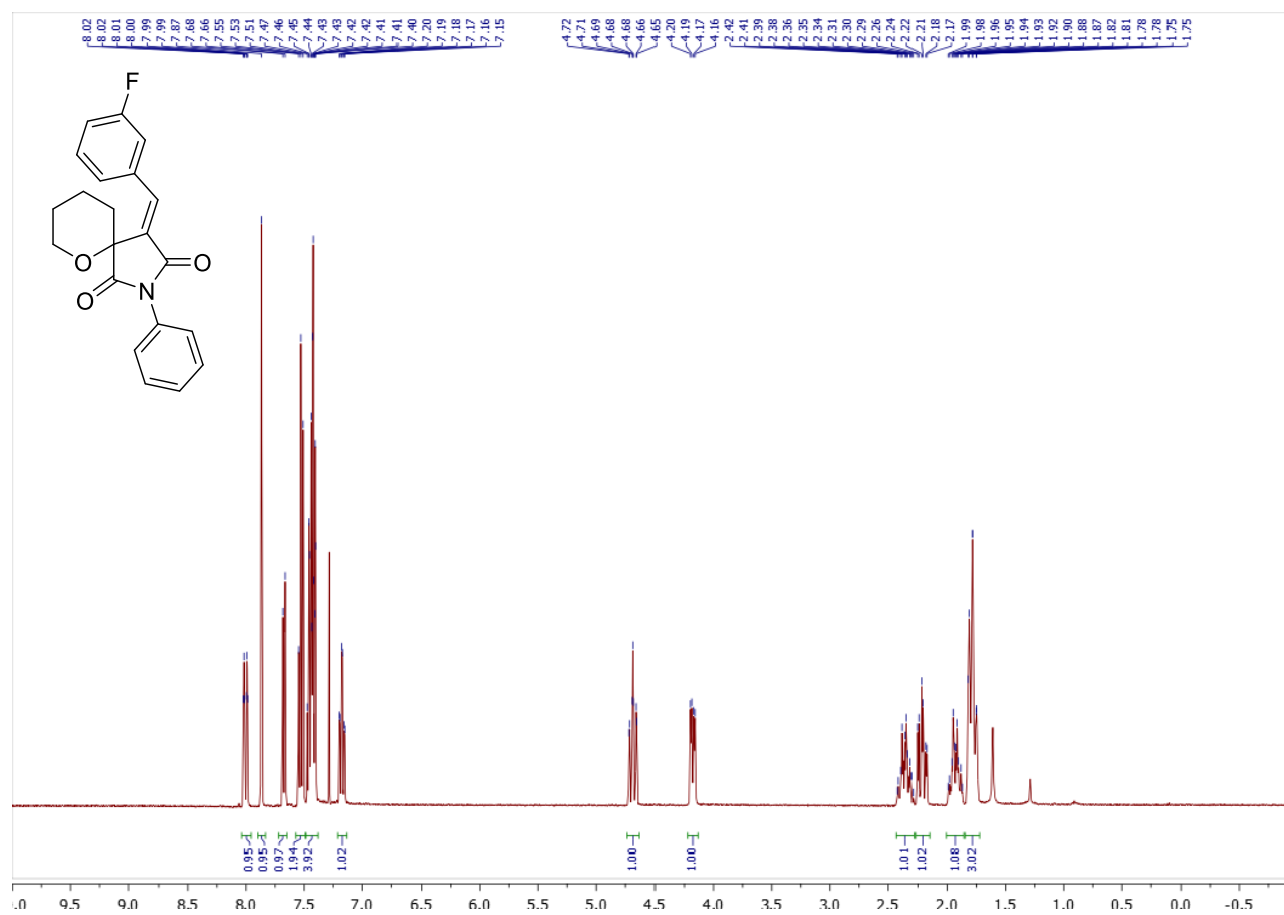




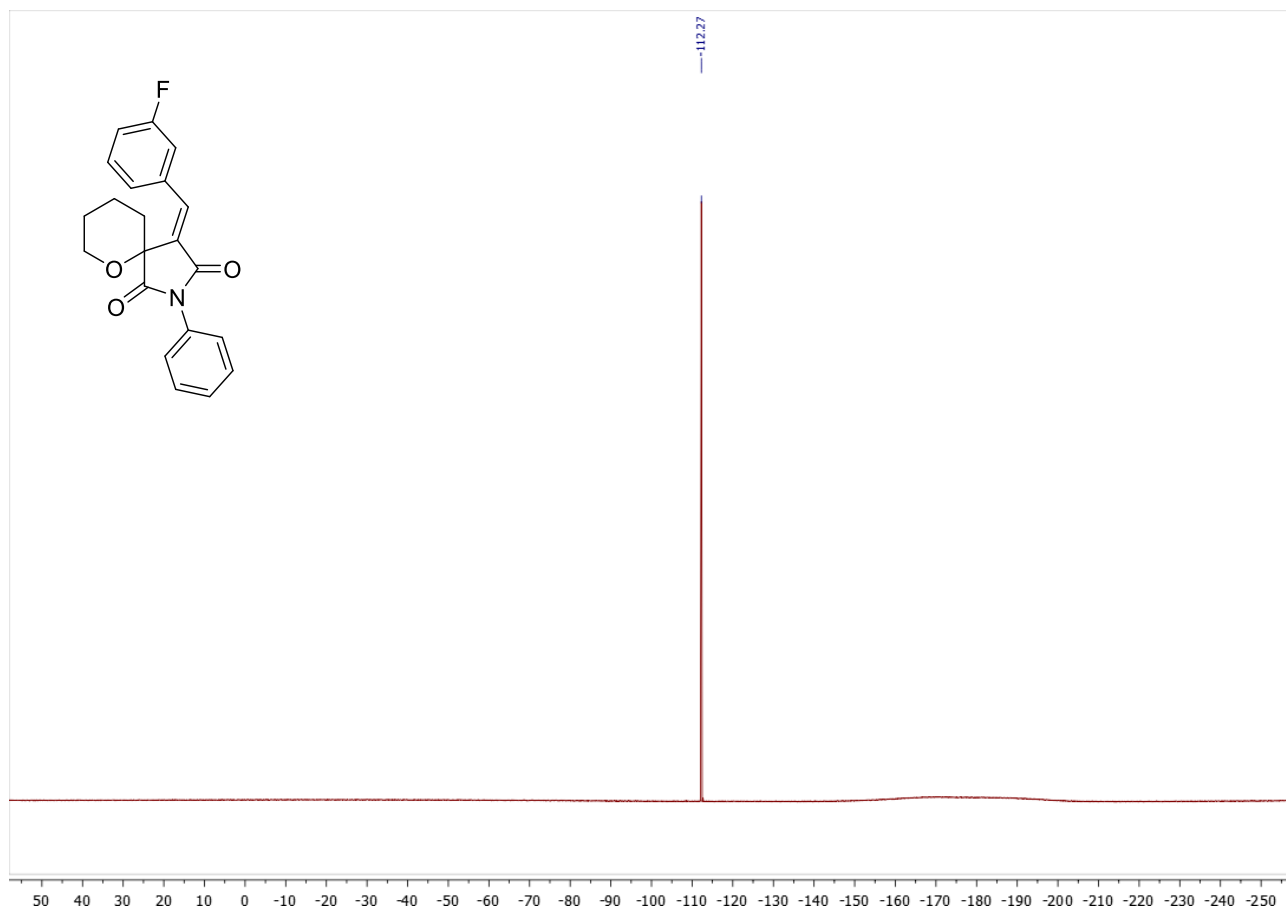
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of **2i**



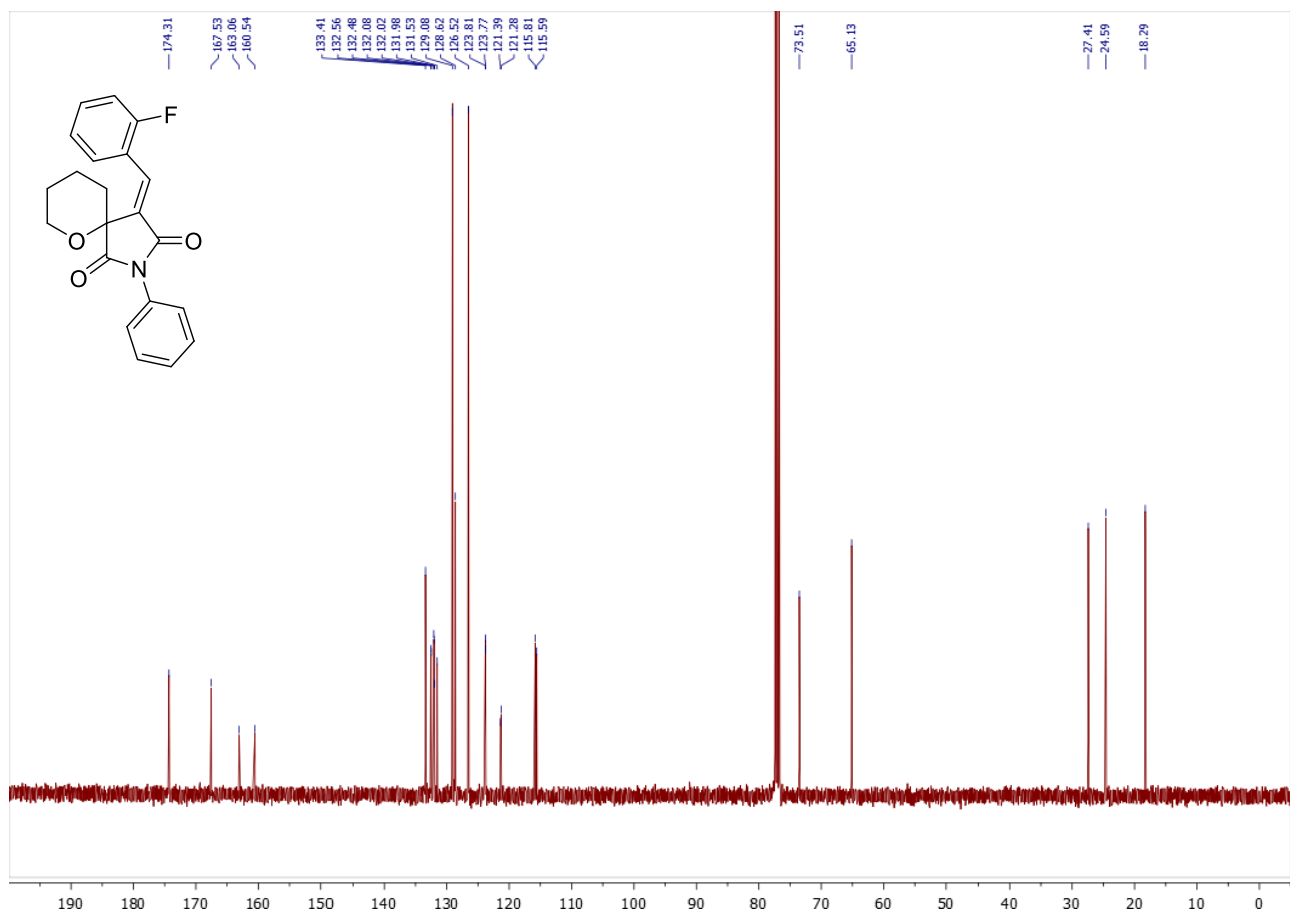
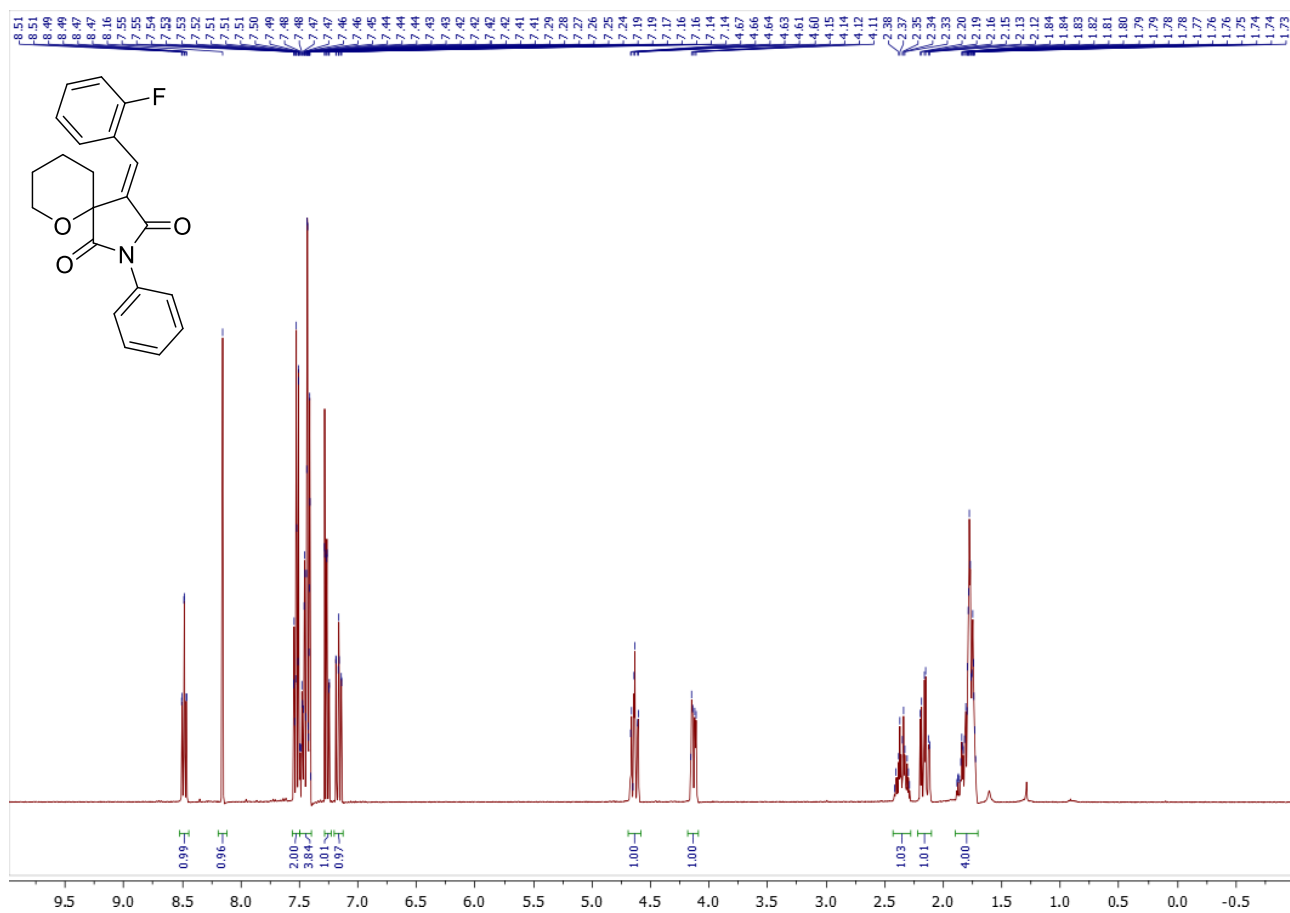
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2j**



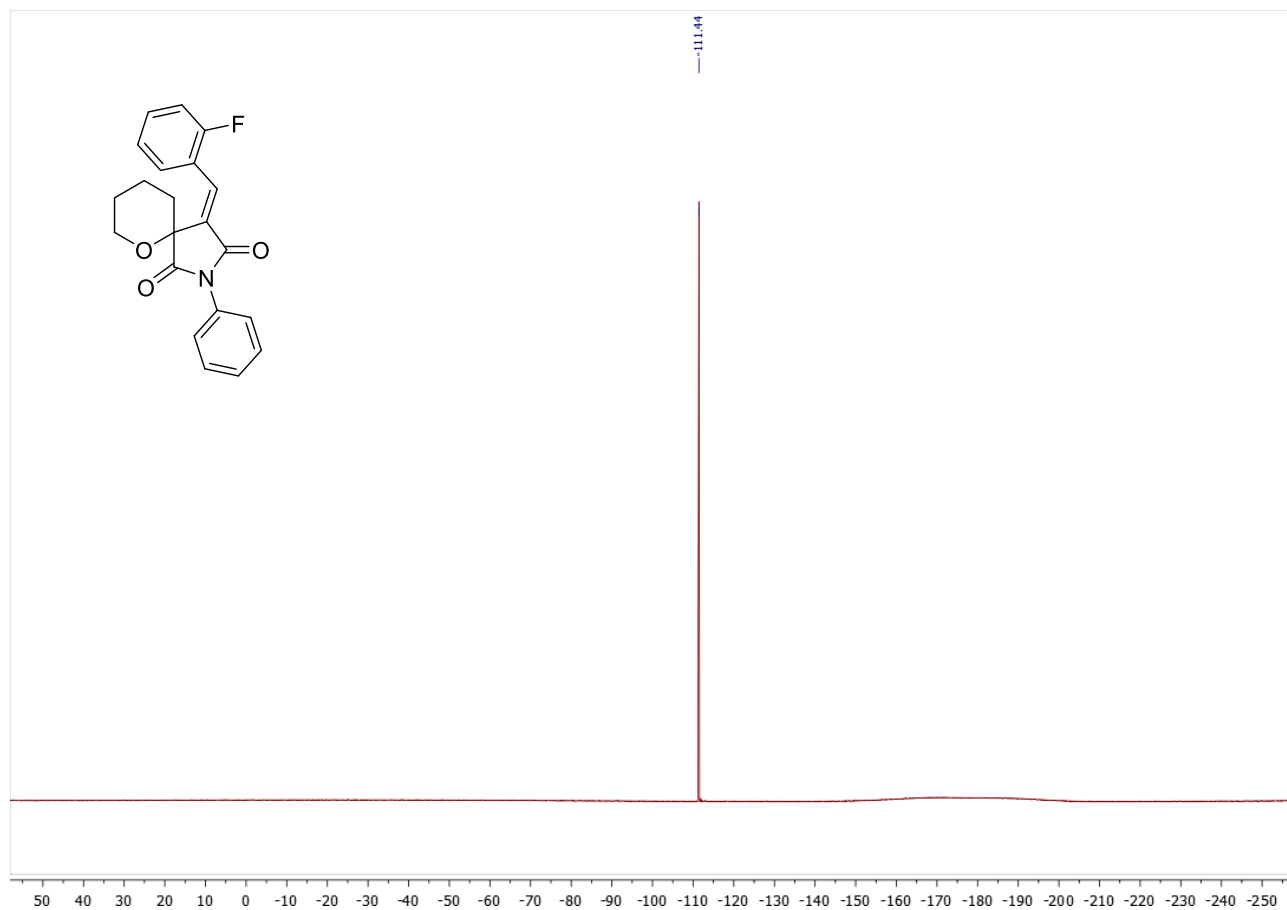
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of **2j**



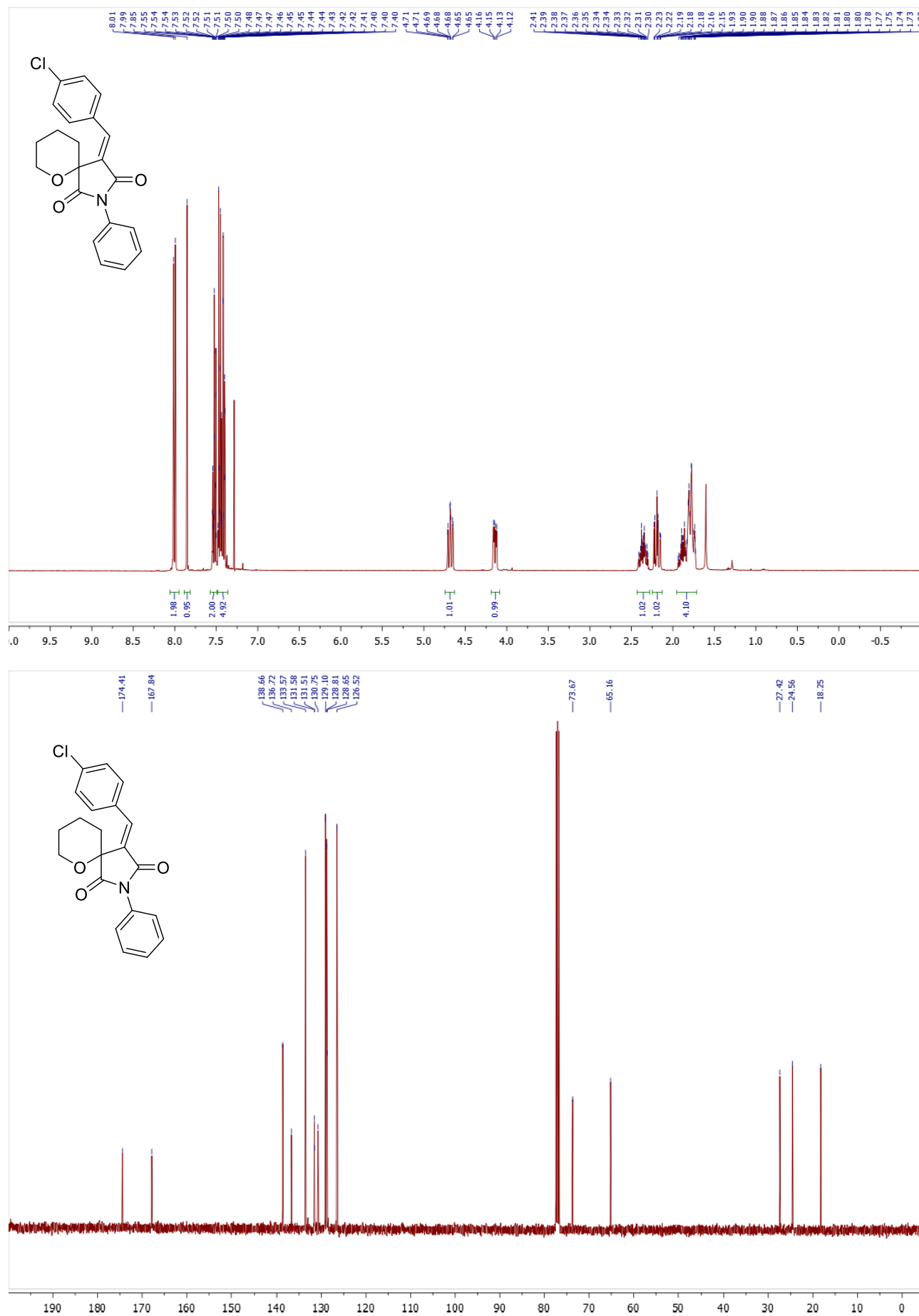
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2k**



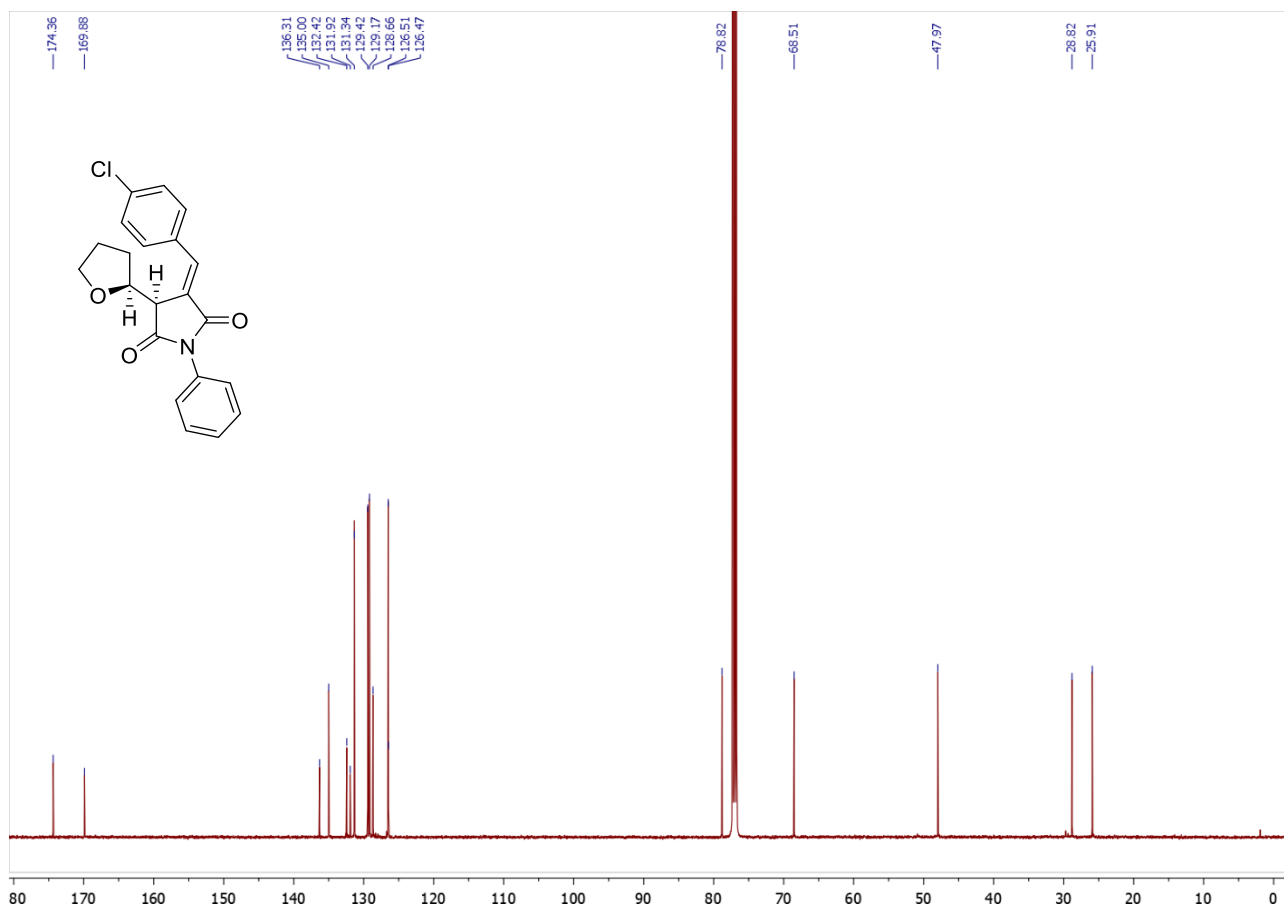
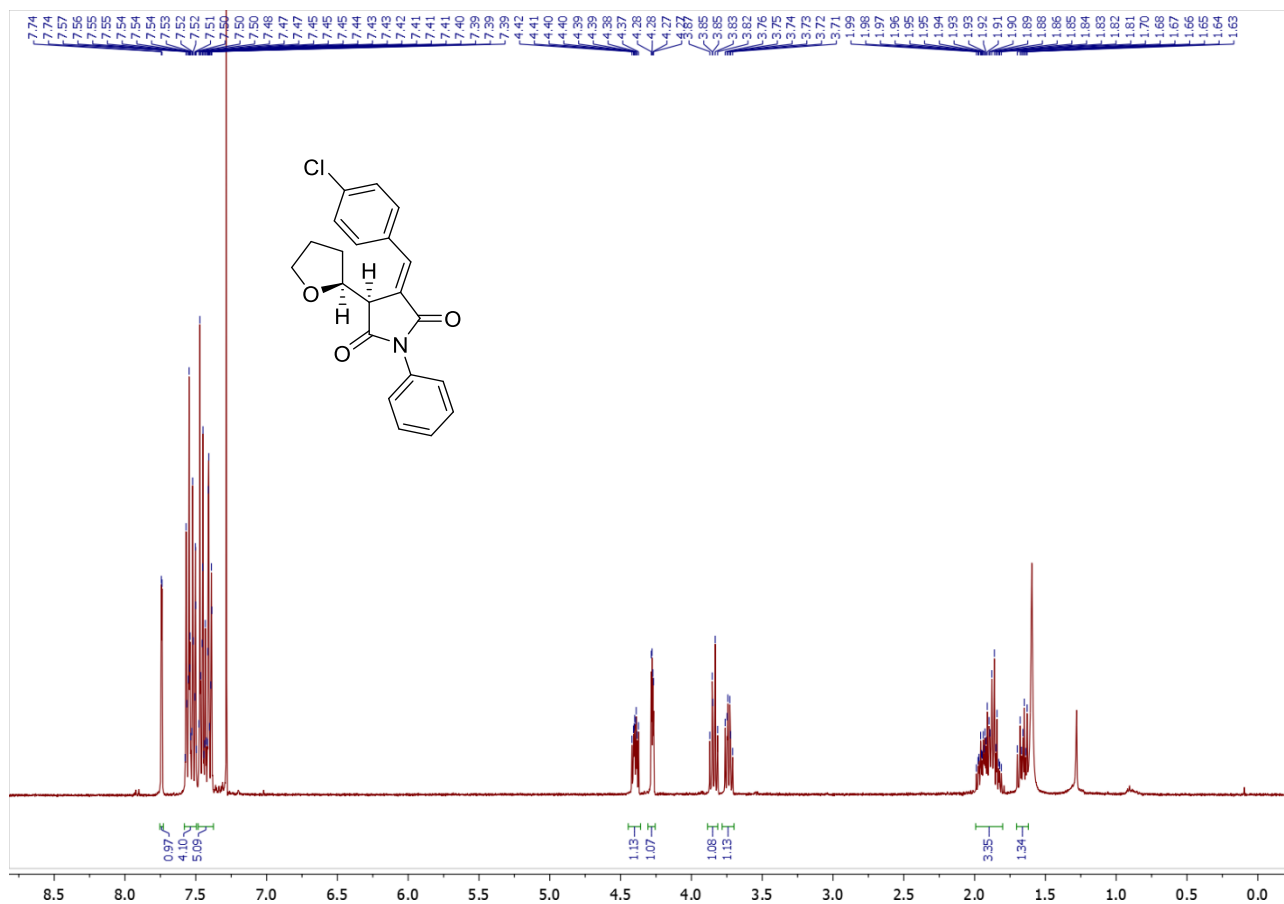
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of **2k**



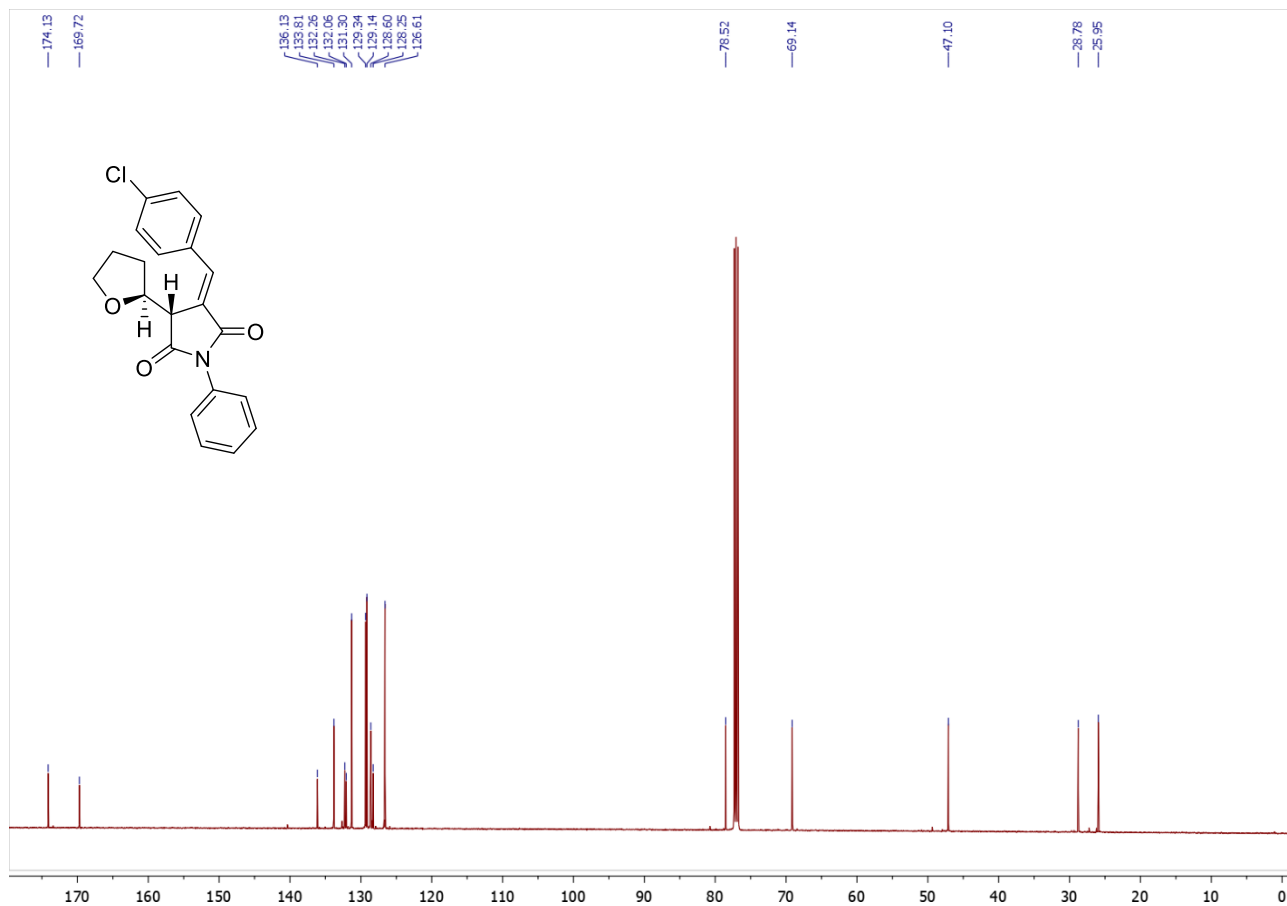
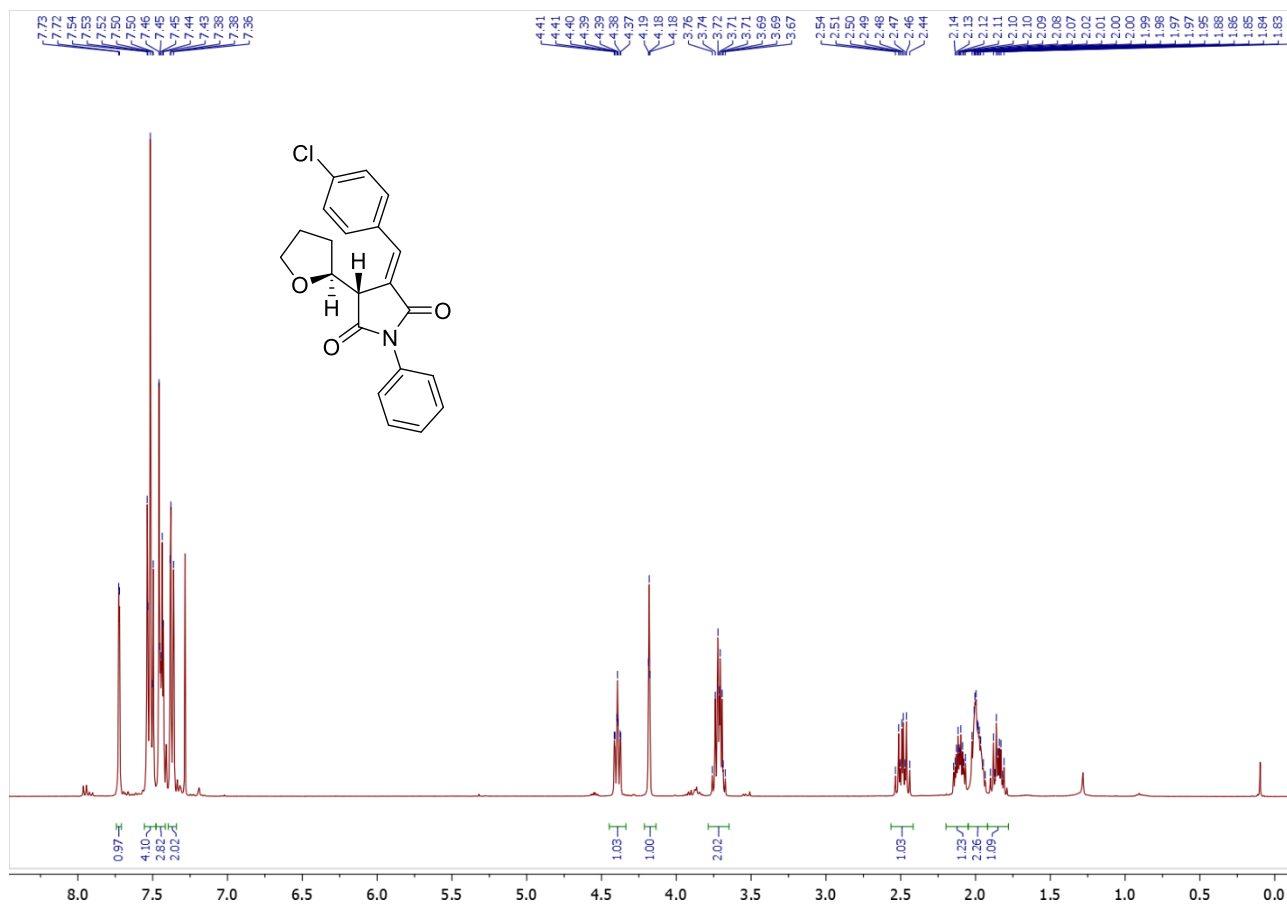
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **21**



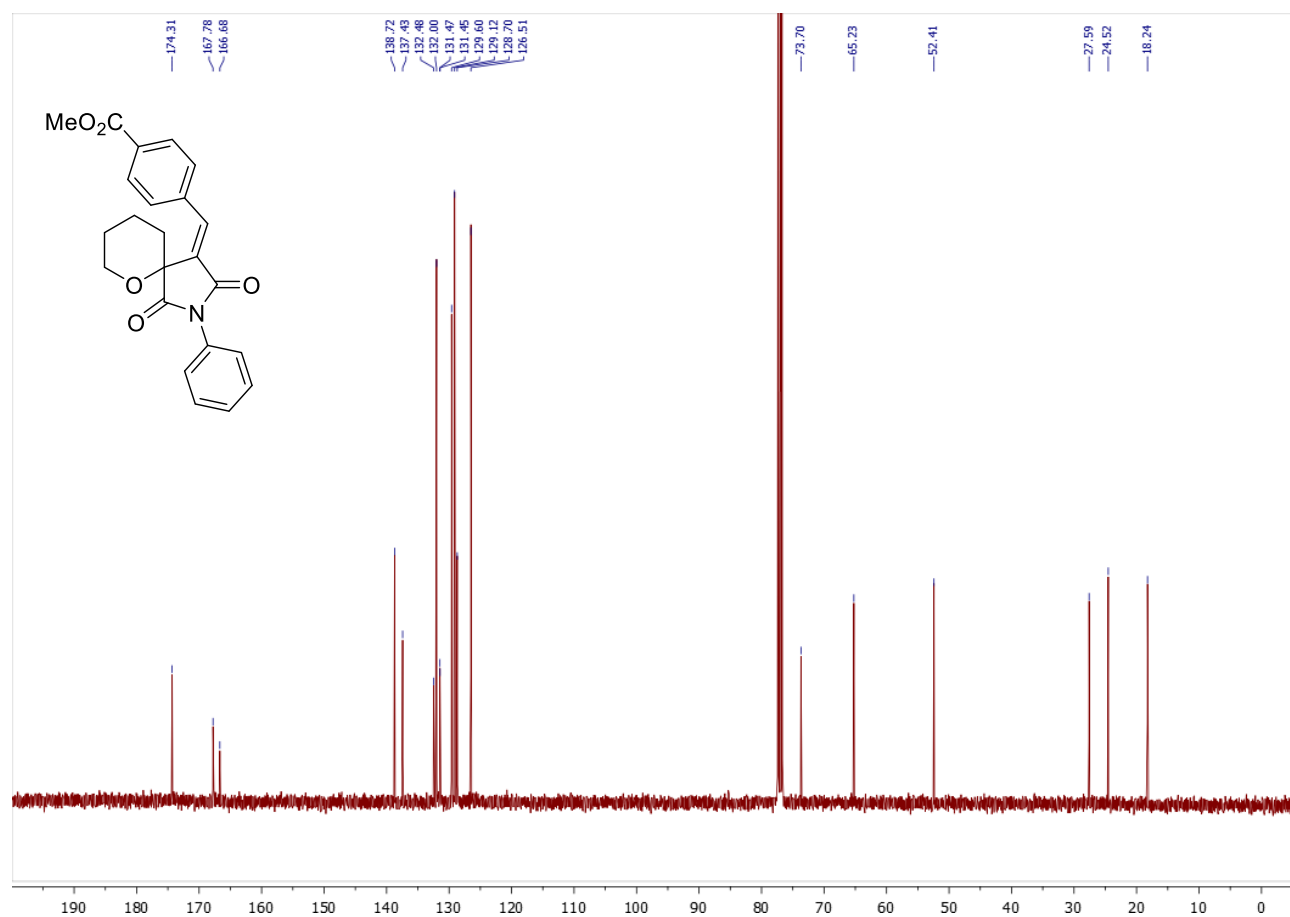
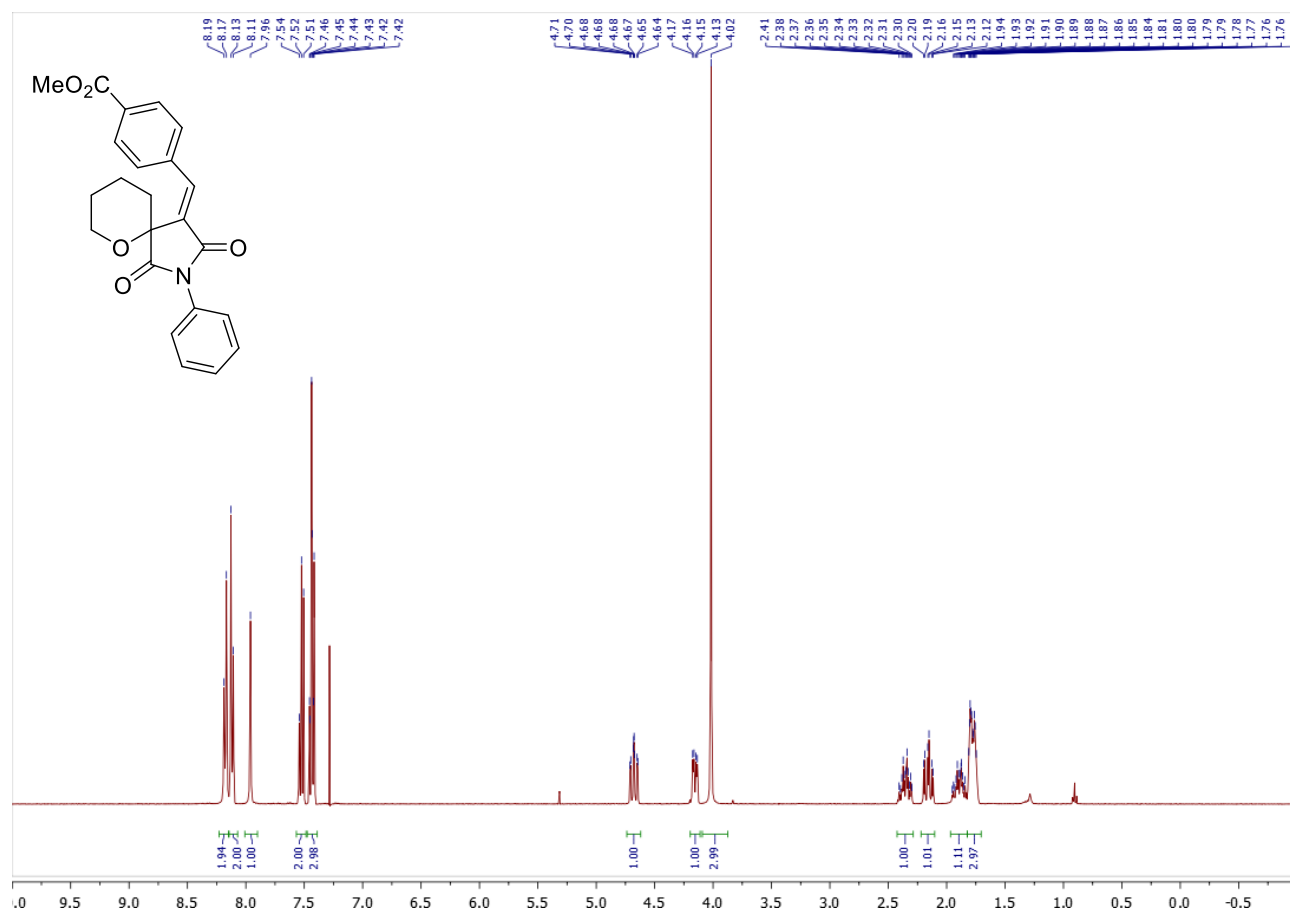
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*R/S,S/R*)-61^{min}



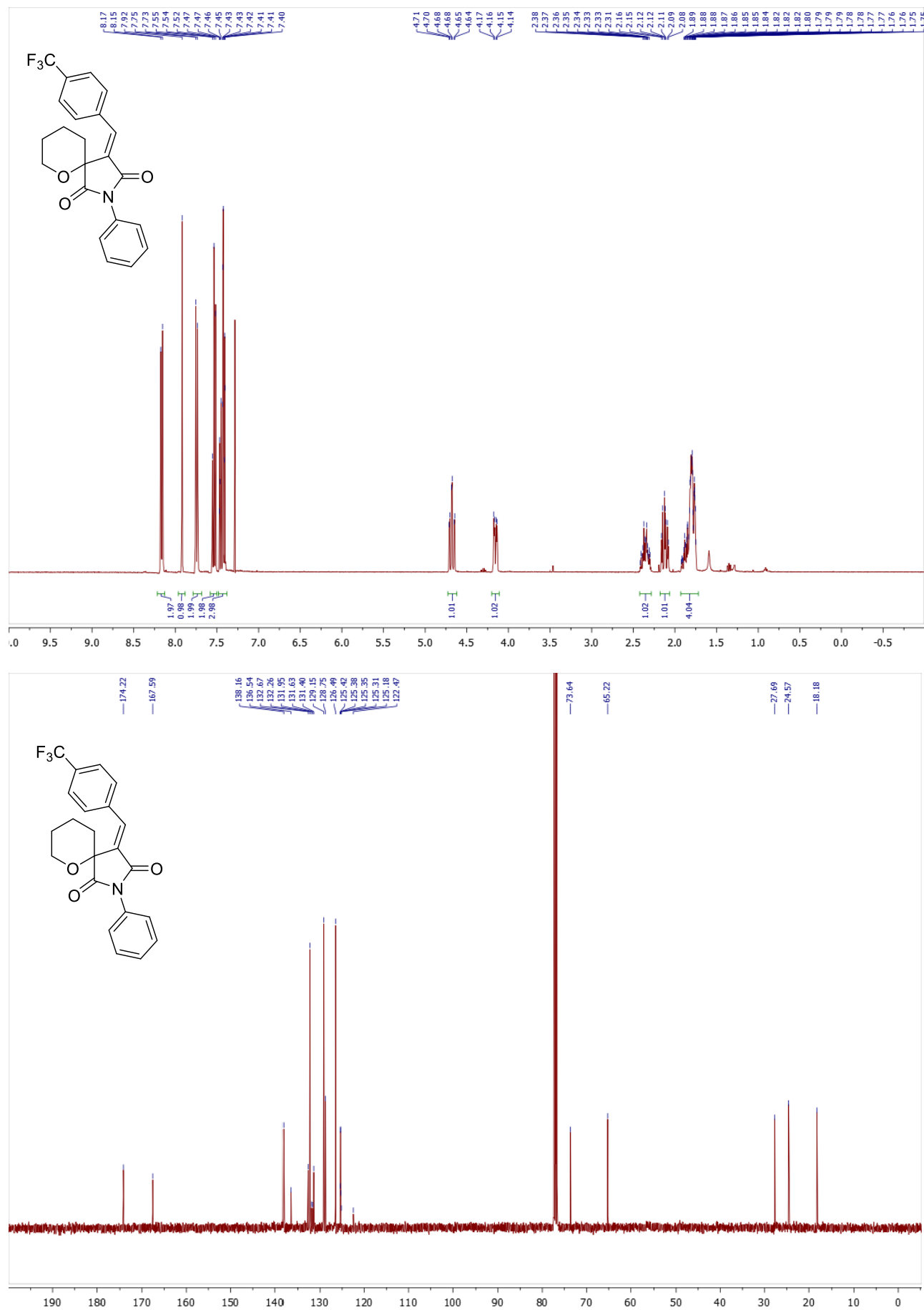
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*S/R,S/R*)-61^{maj}



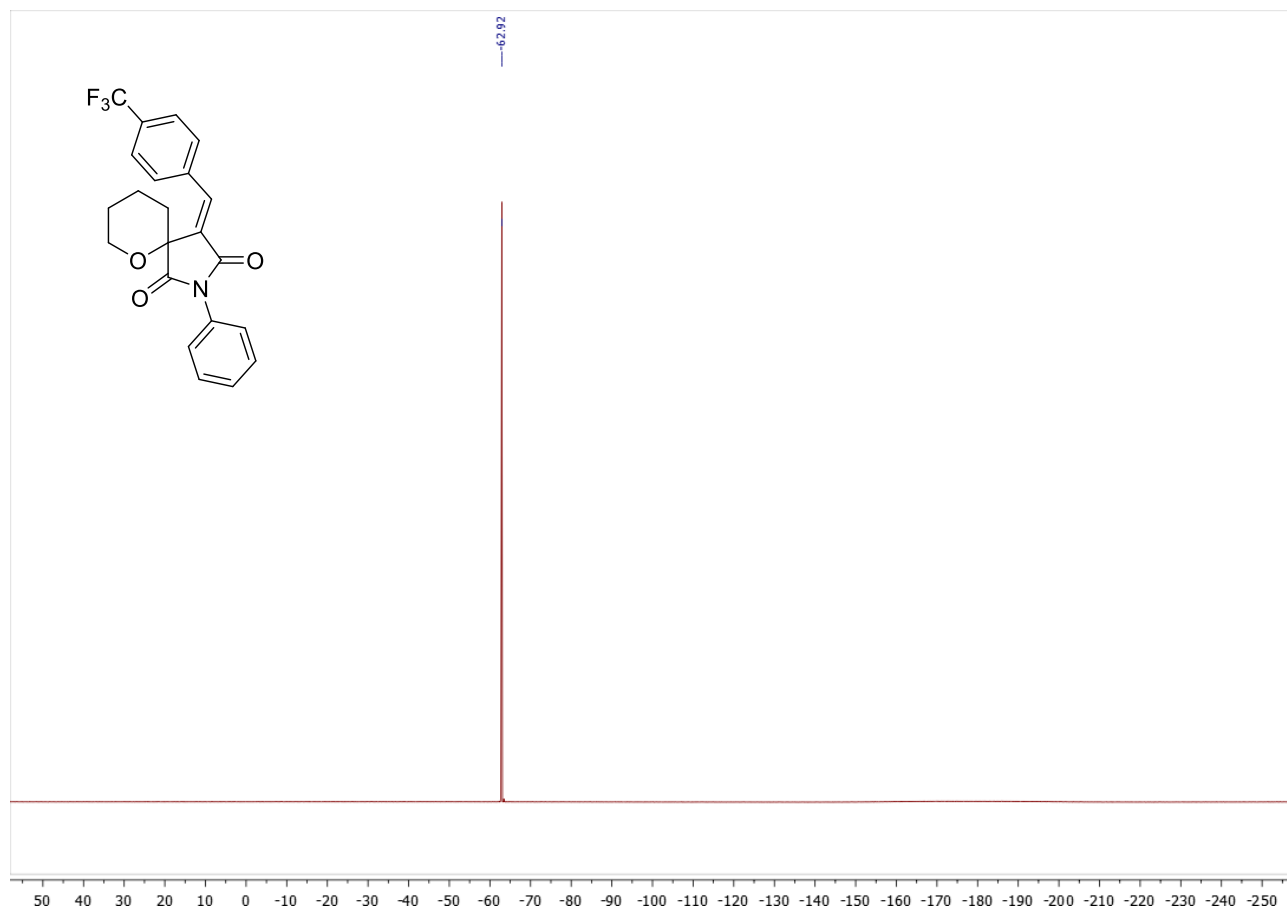
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2m**



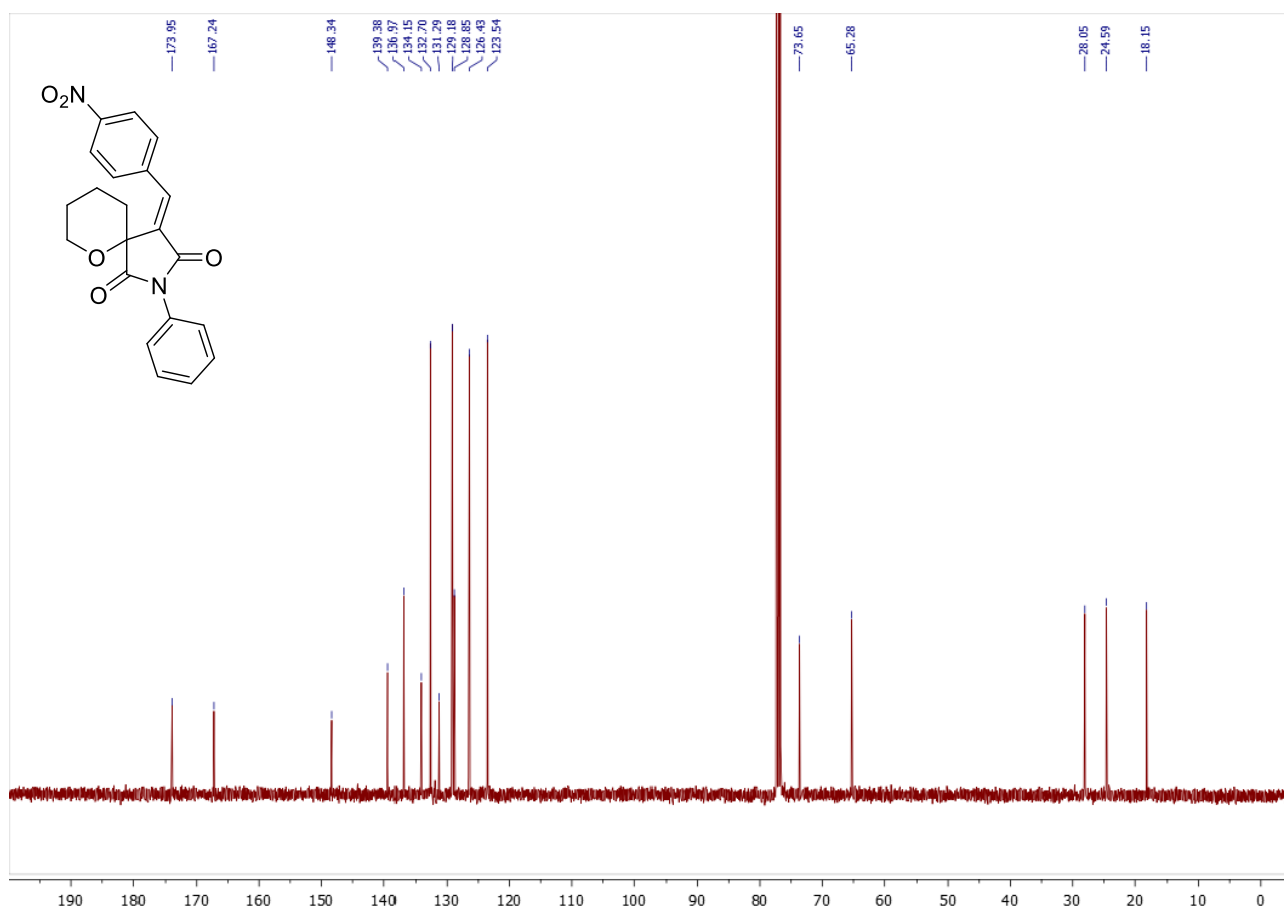
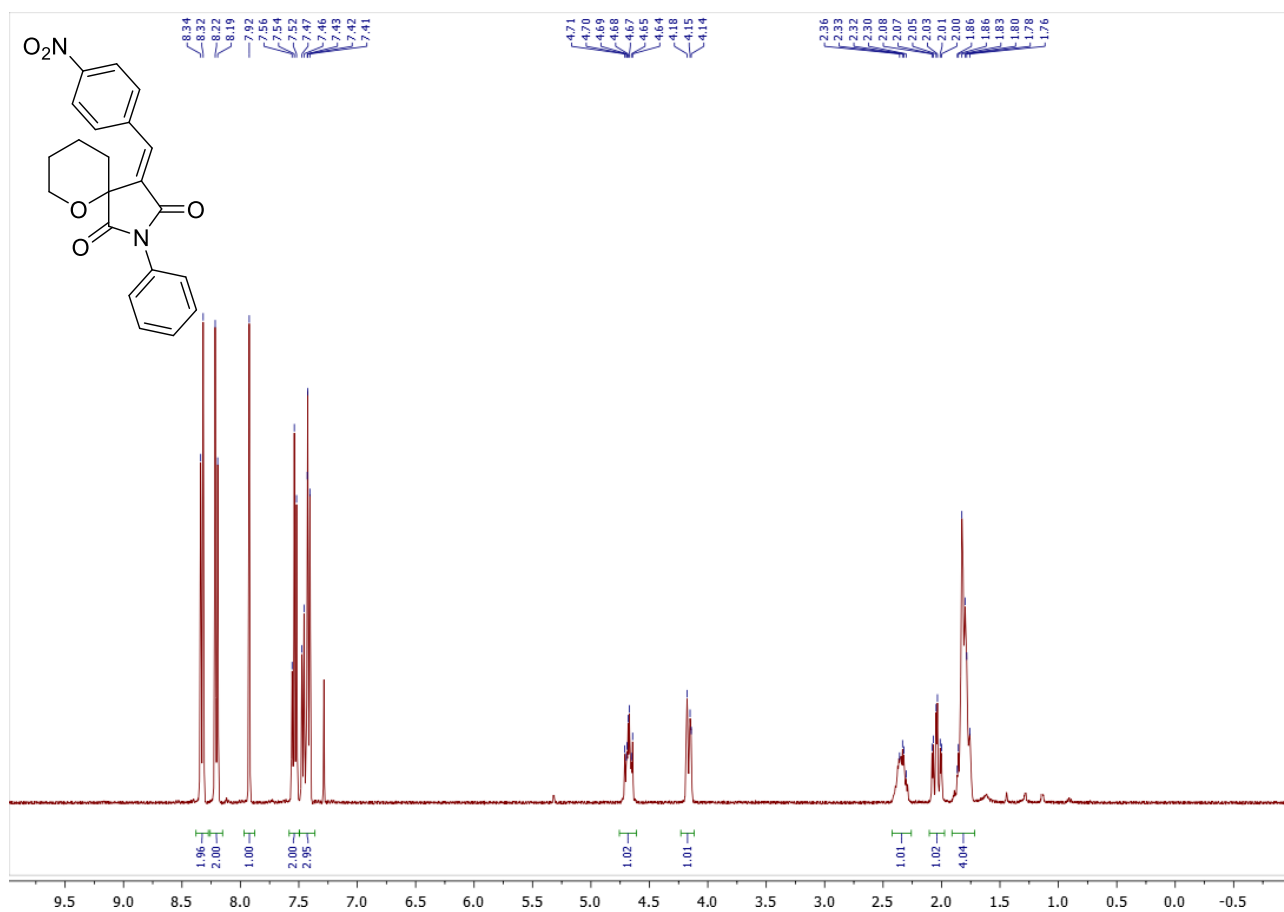
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2n**



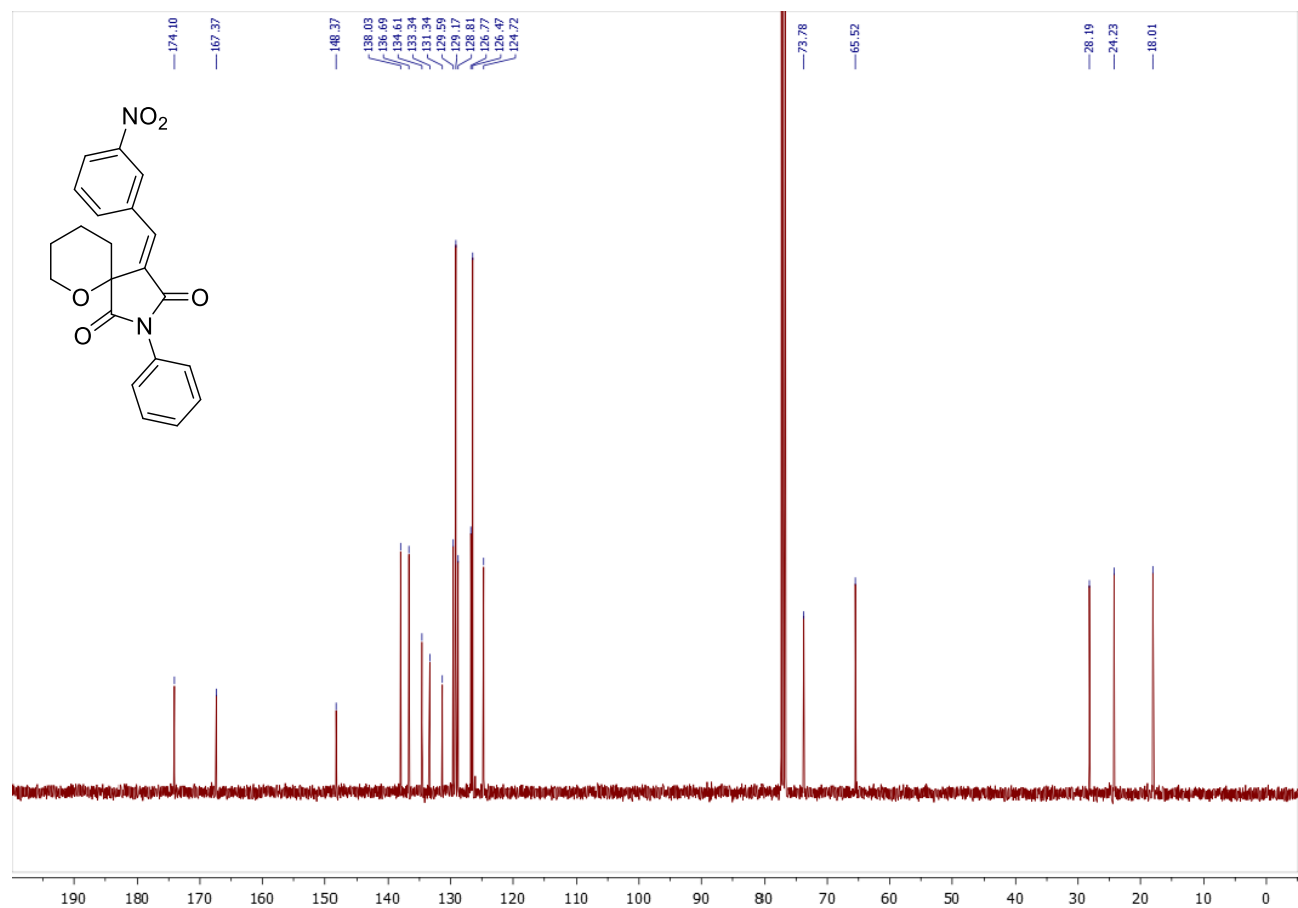
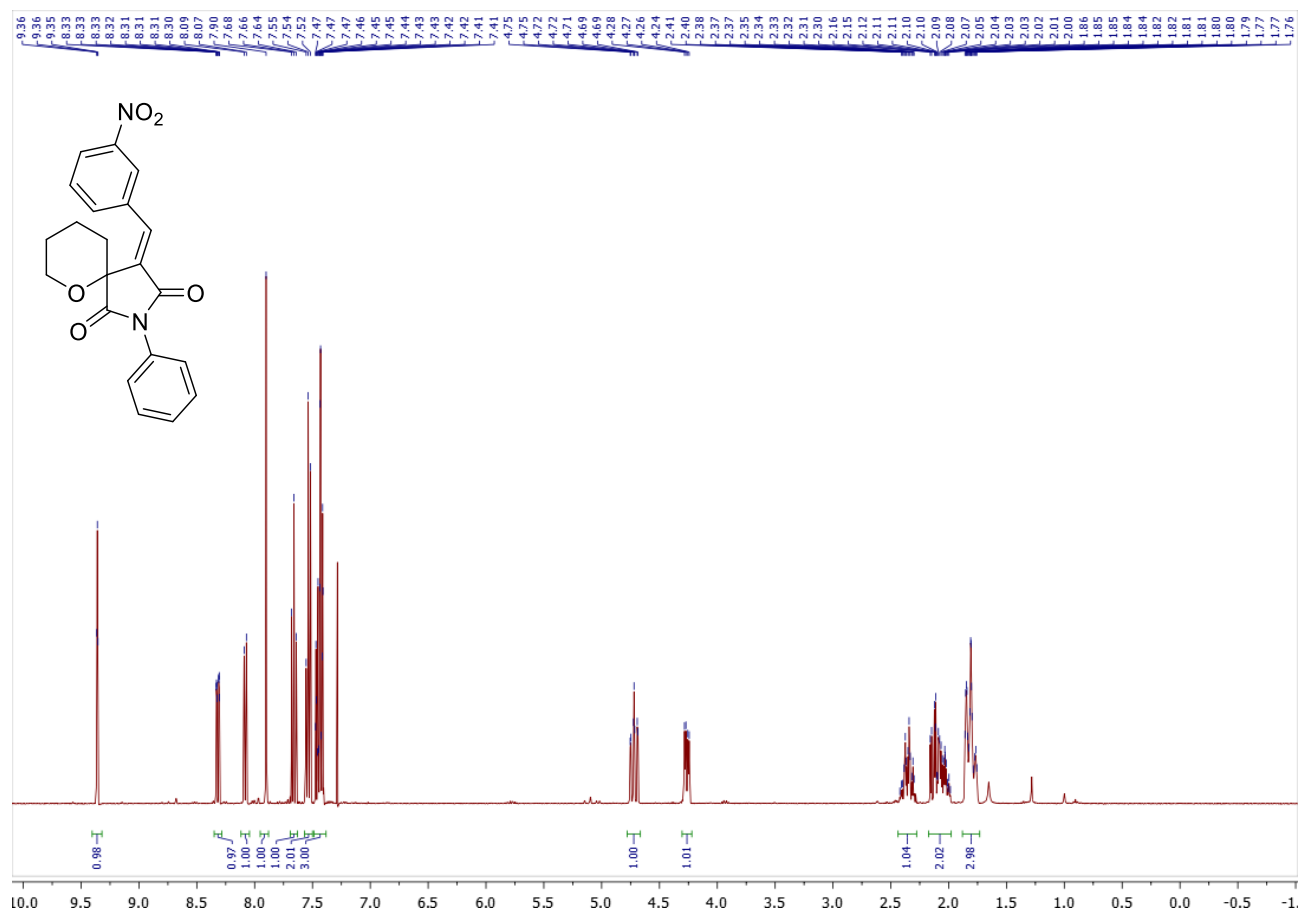
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of **2n**



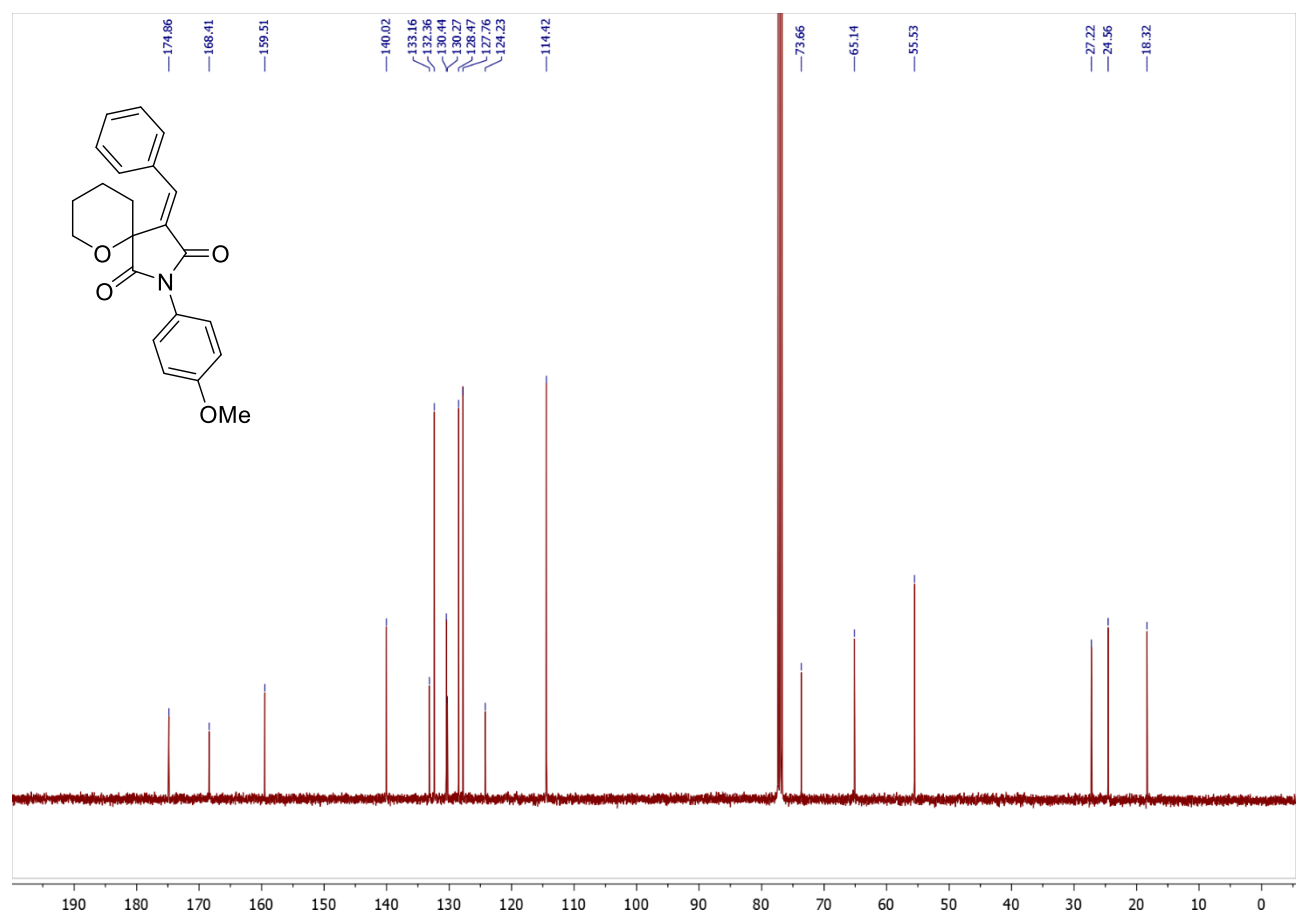
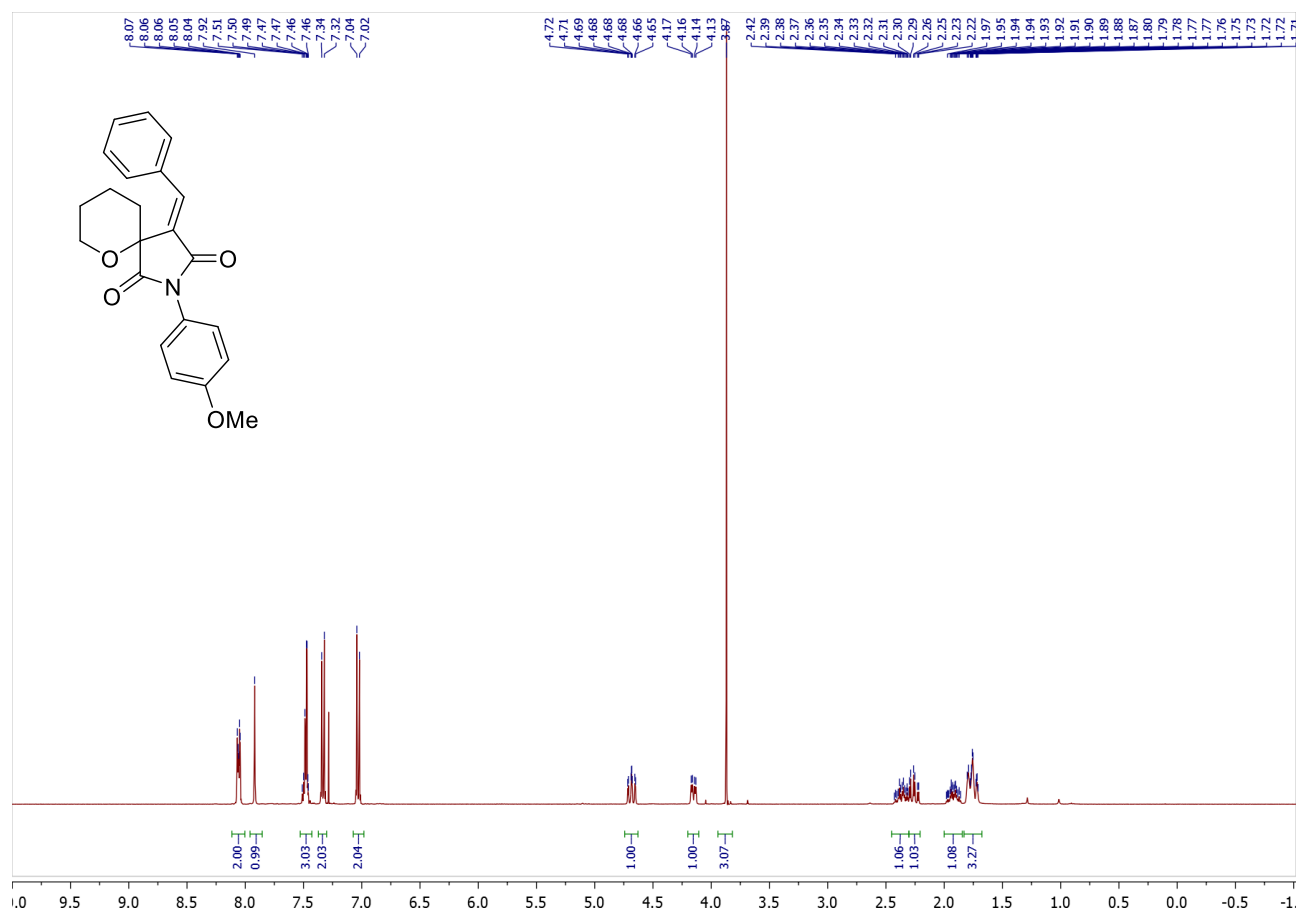
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2o**



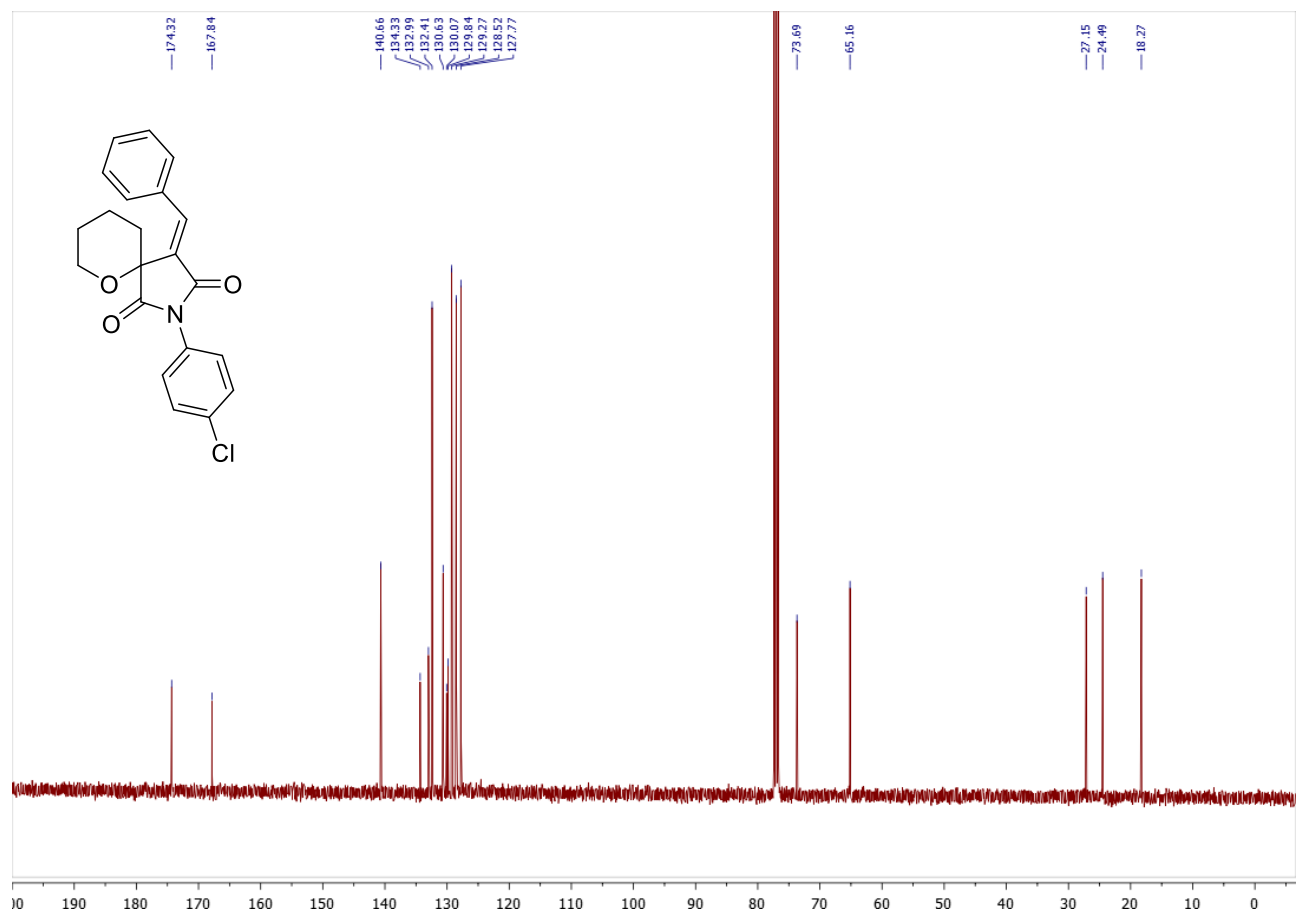
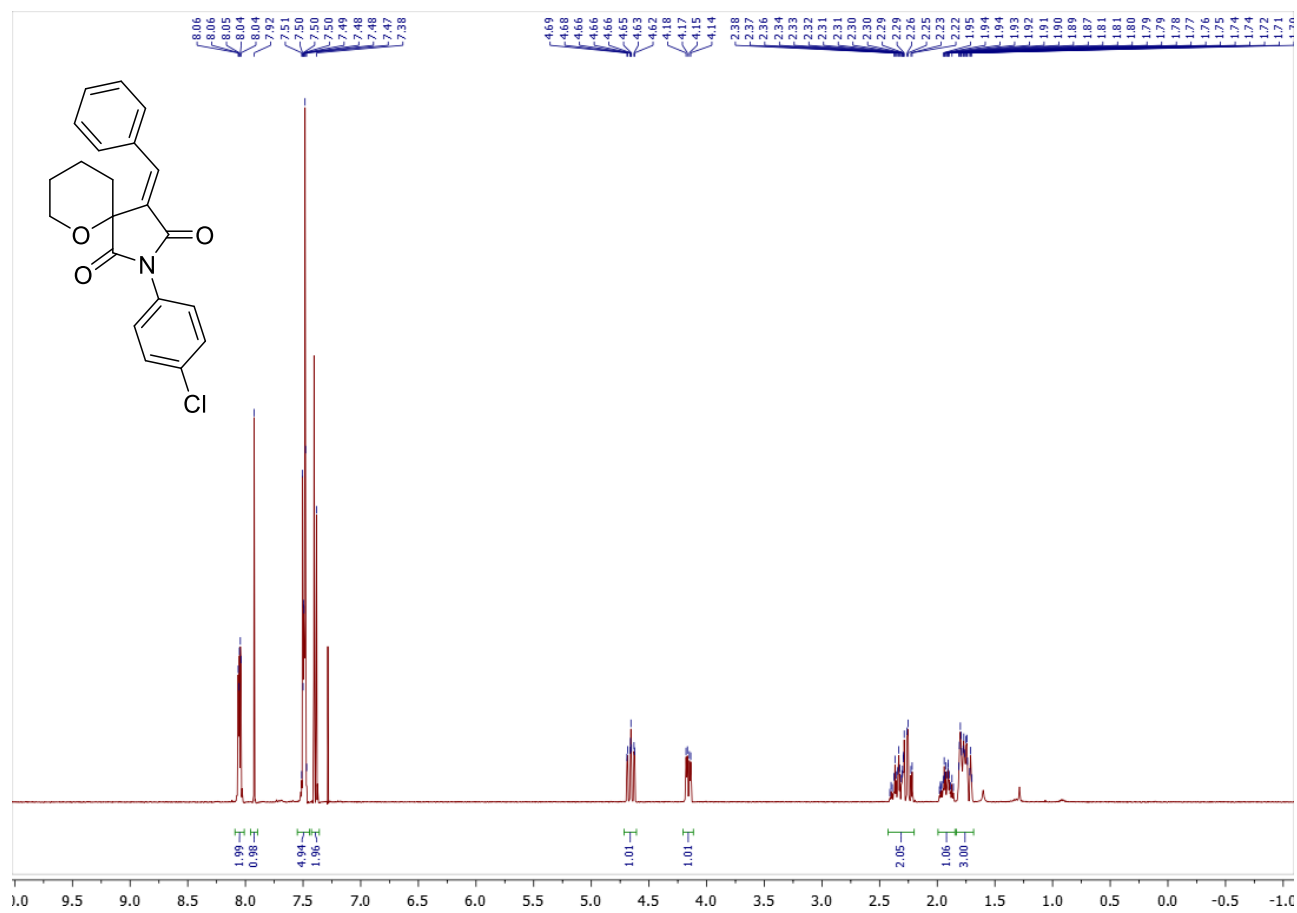
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2p**



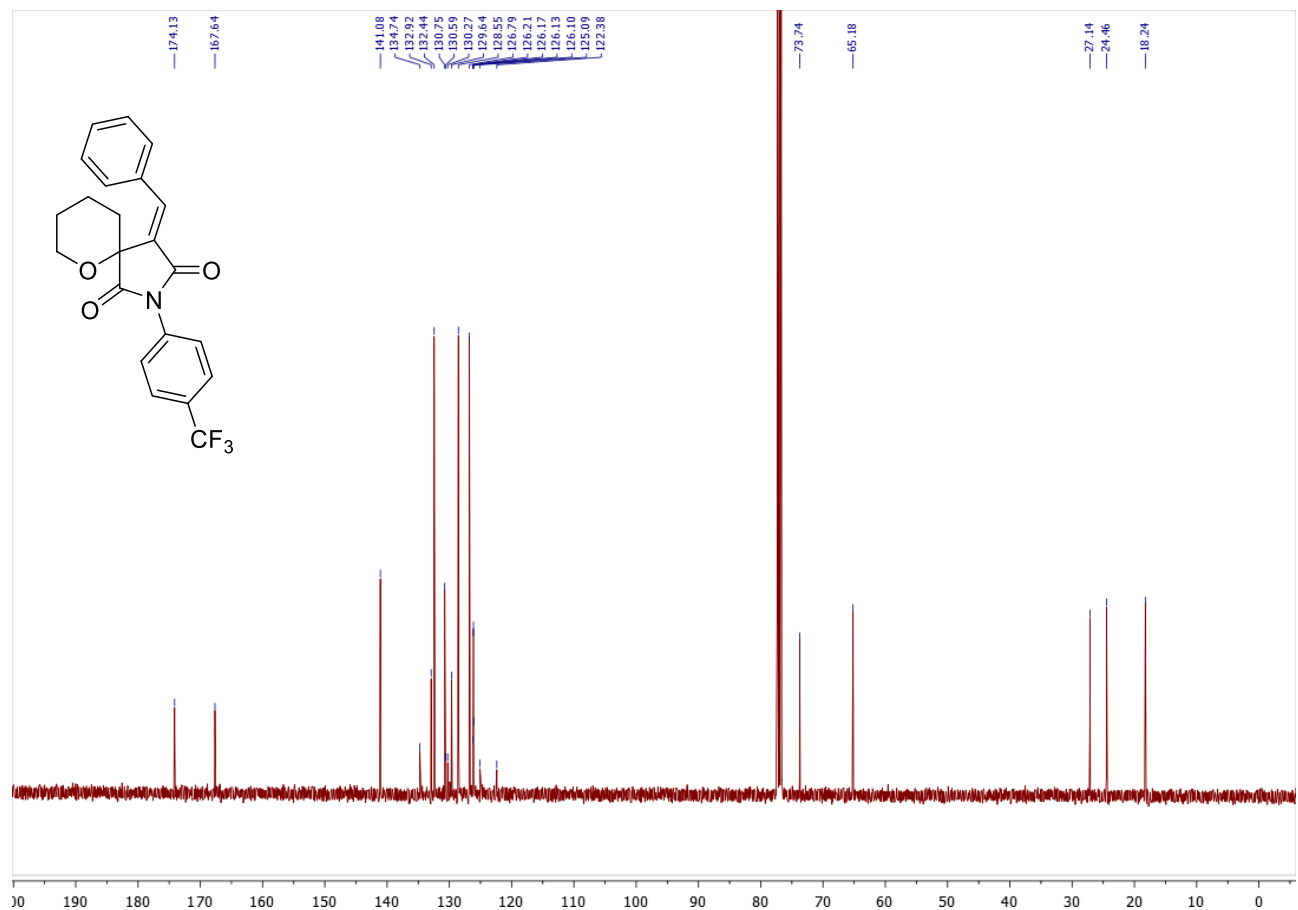
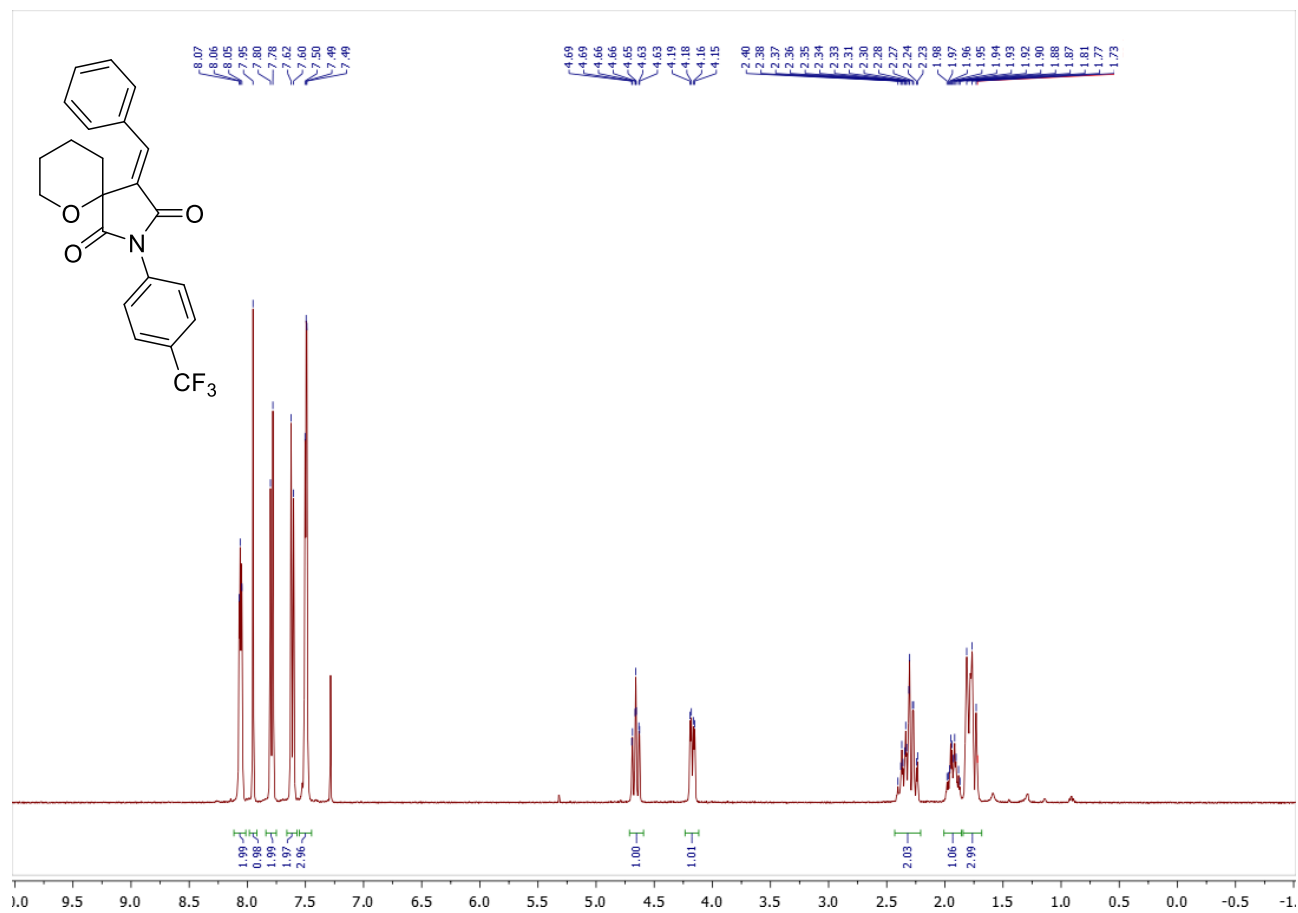
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2q**



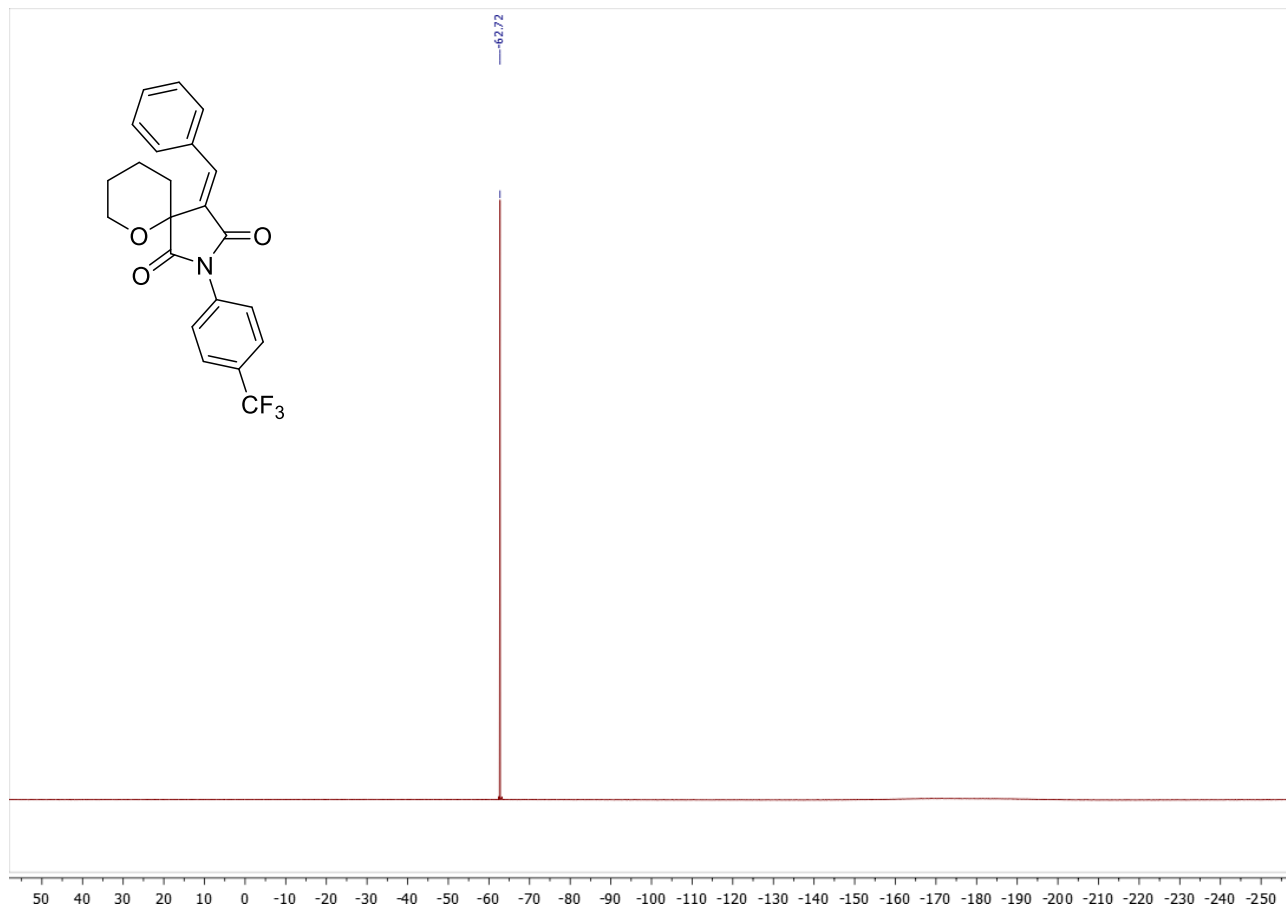
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2r**



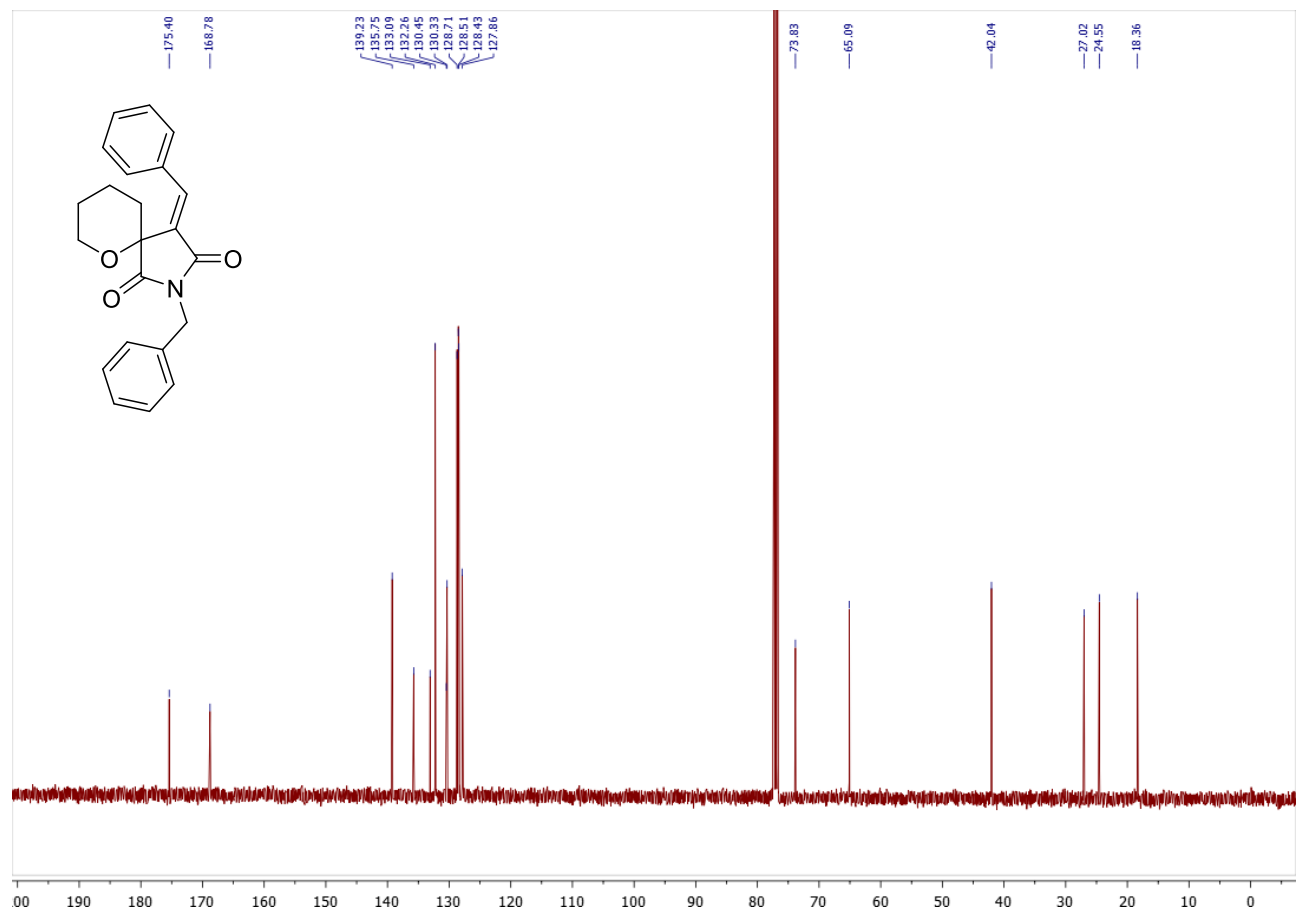
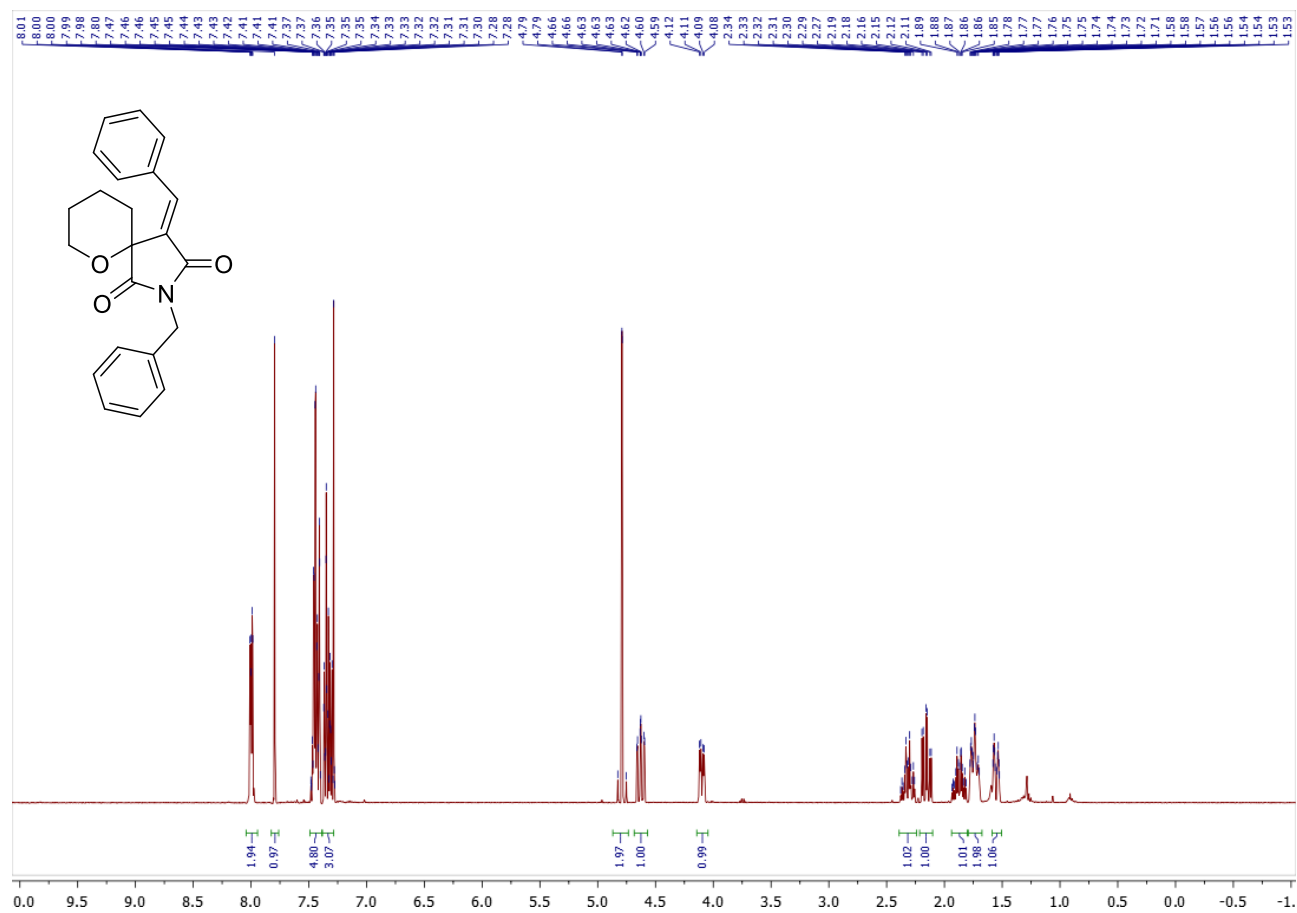
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2s**



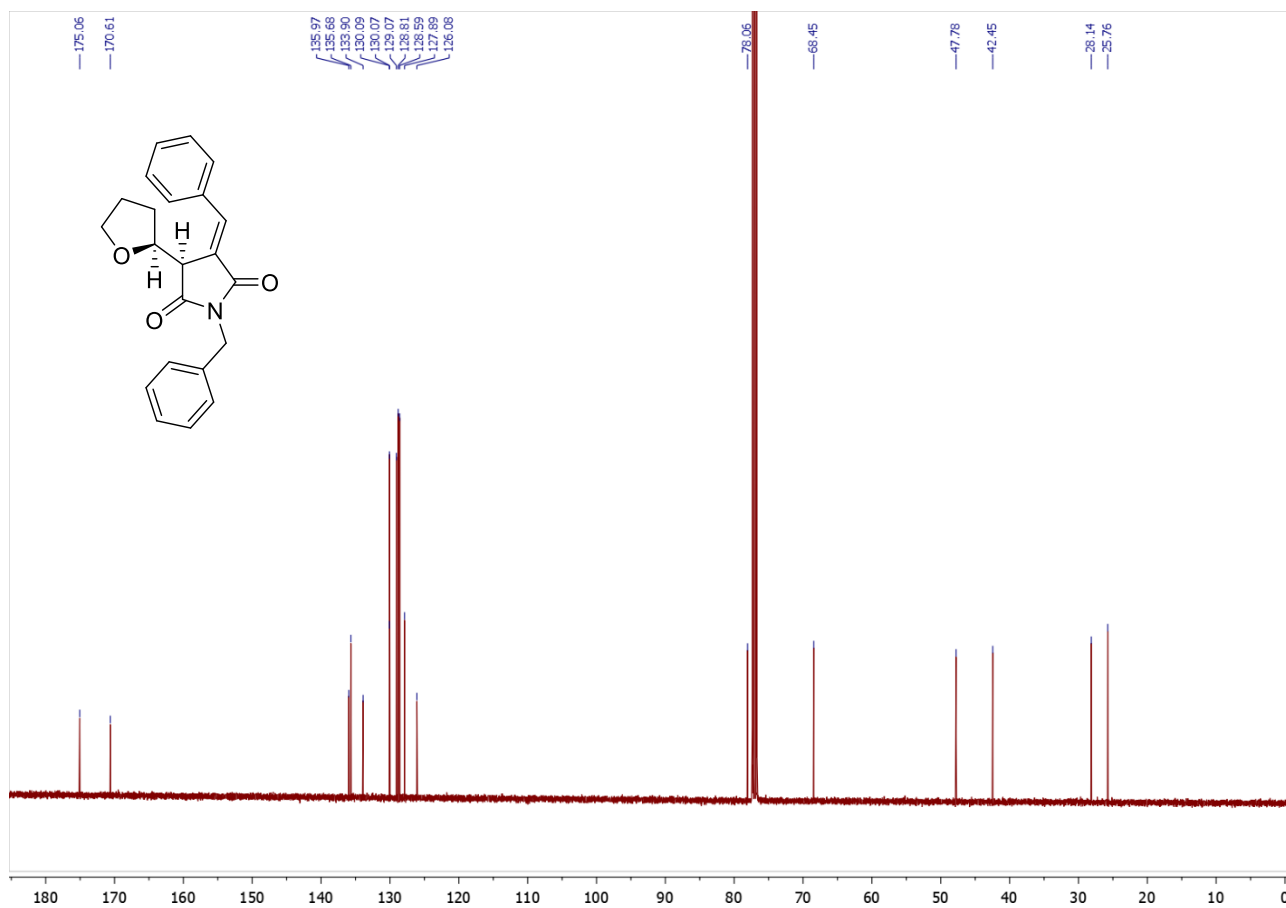
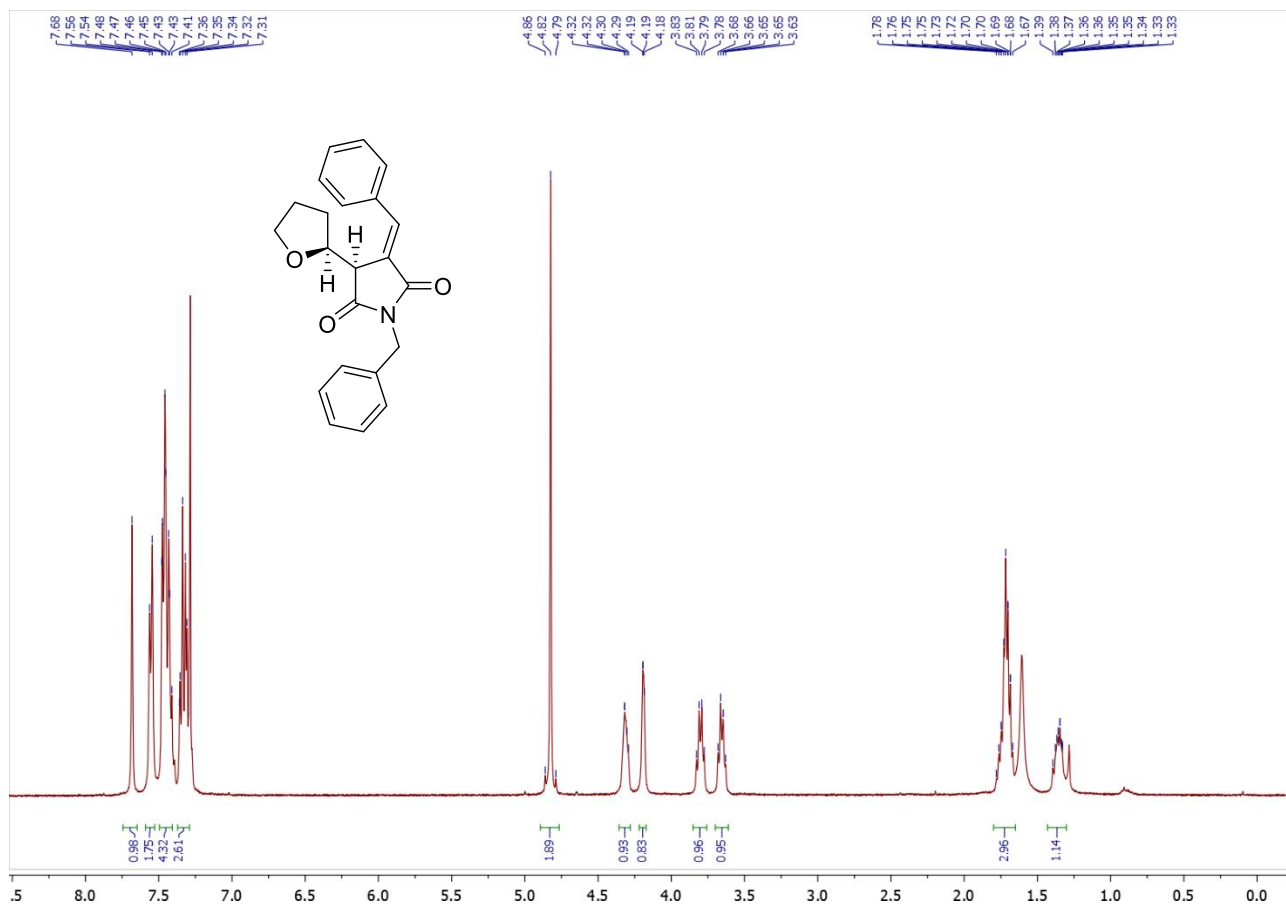
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of **2s**



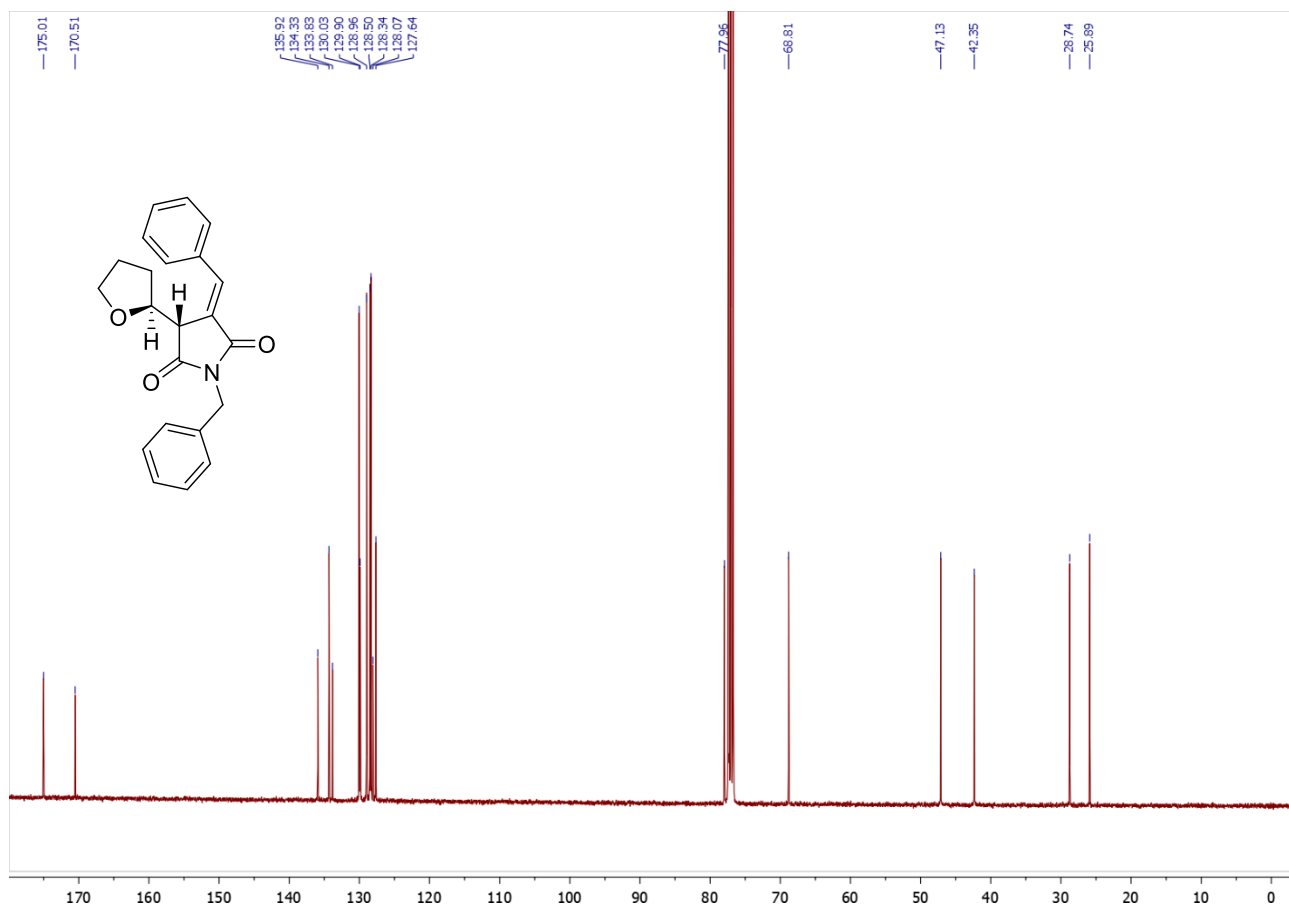
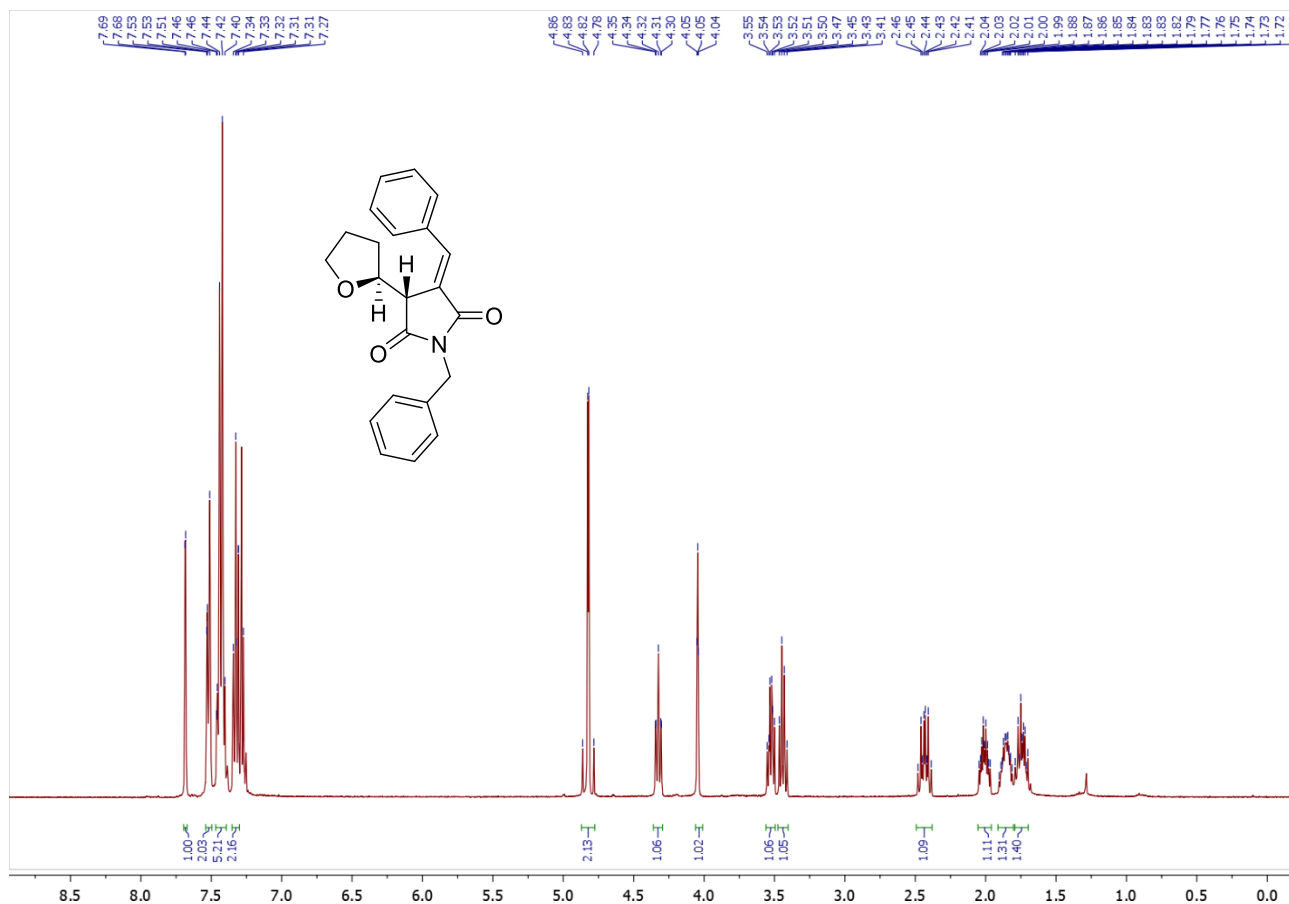
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2t**



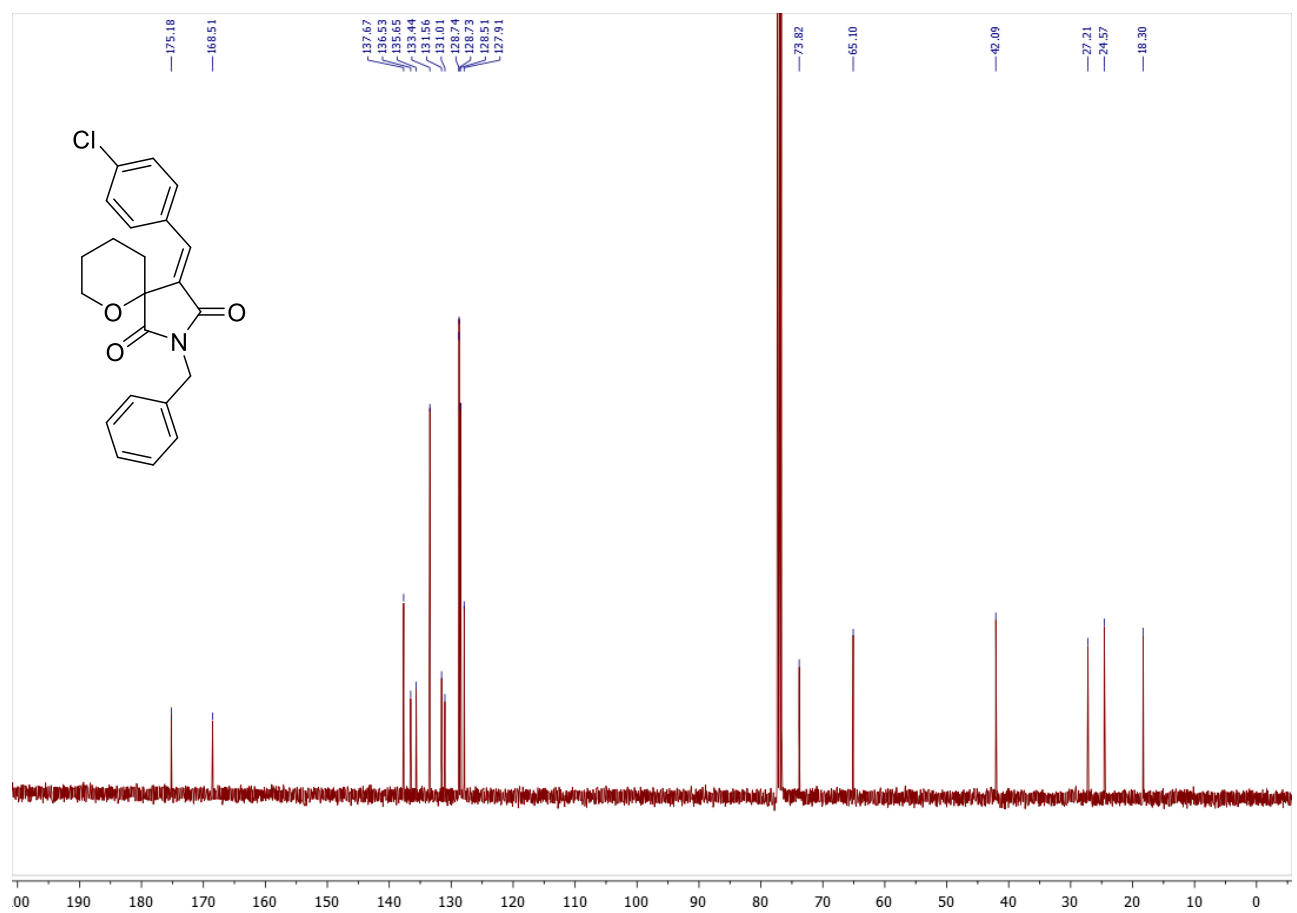
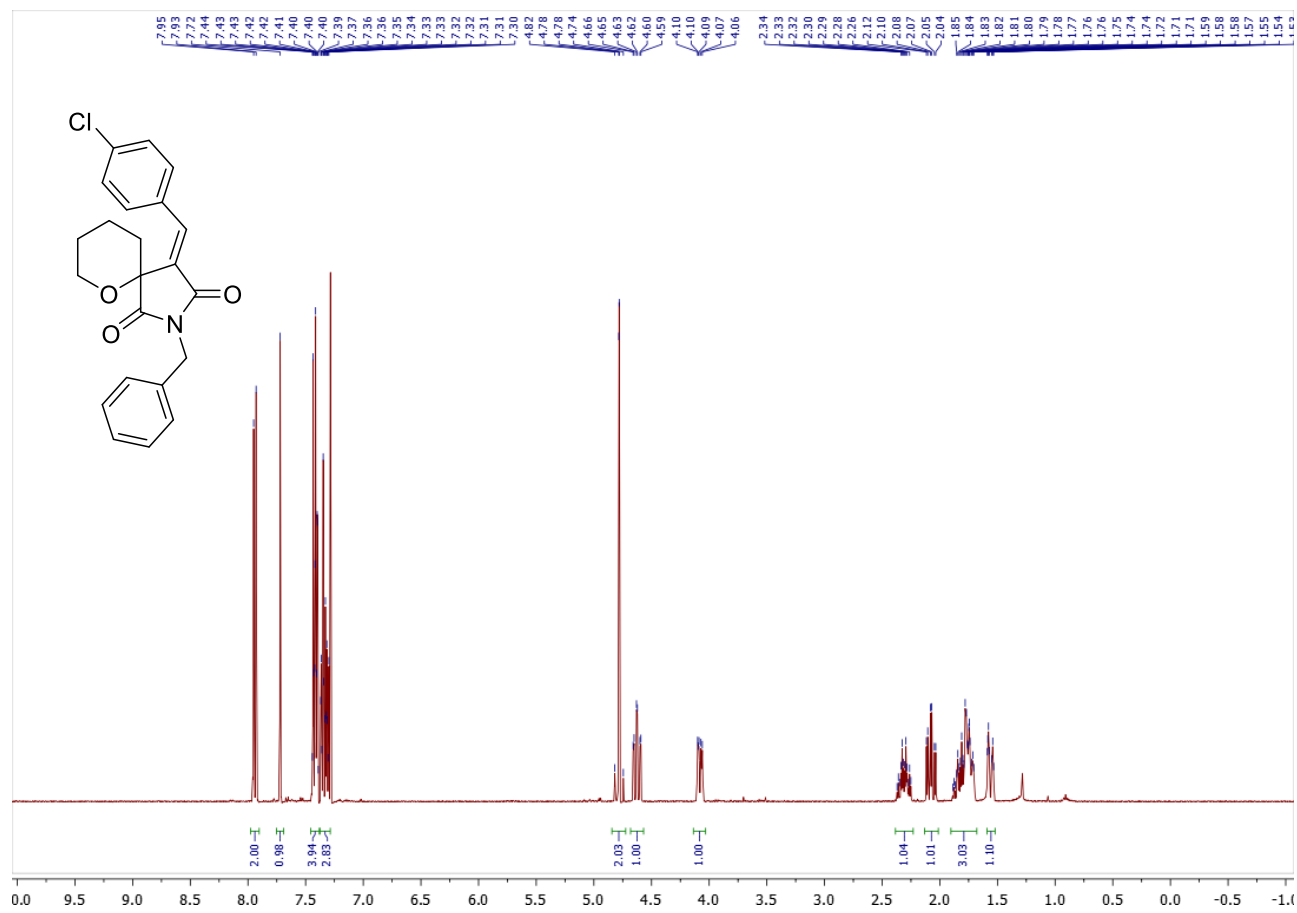
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*R/S,S/R*)-6t^{min}



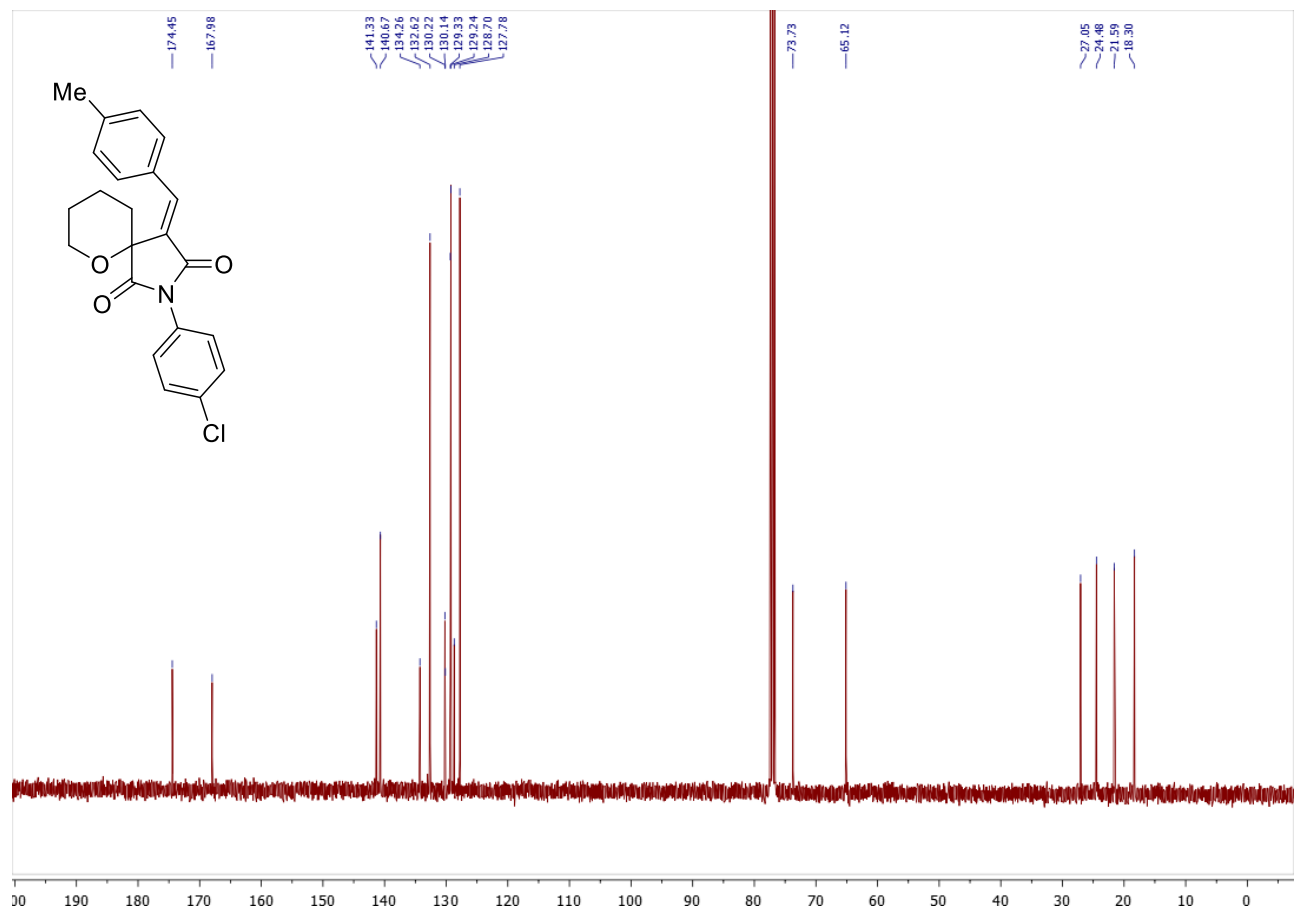
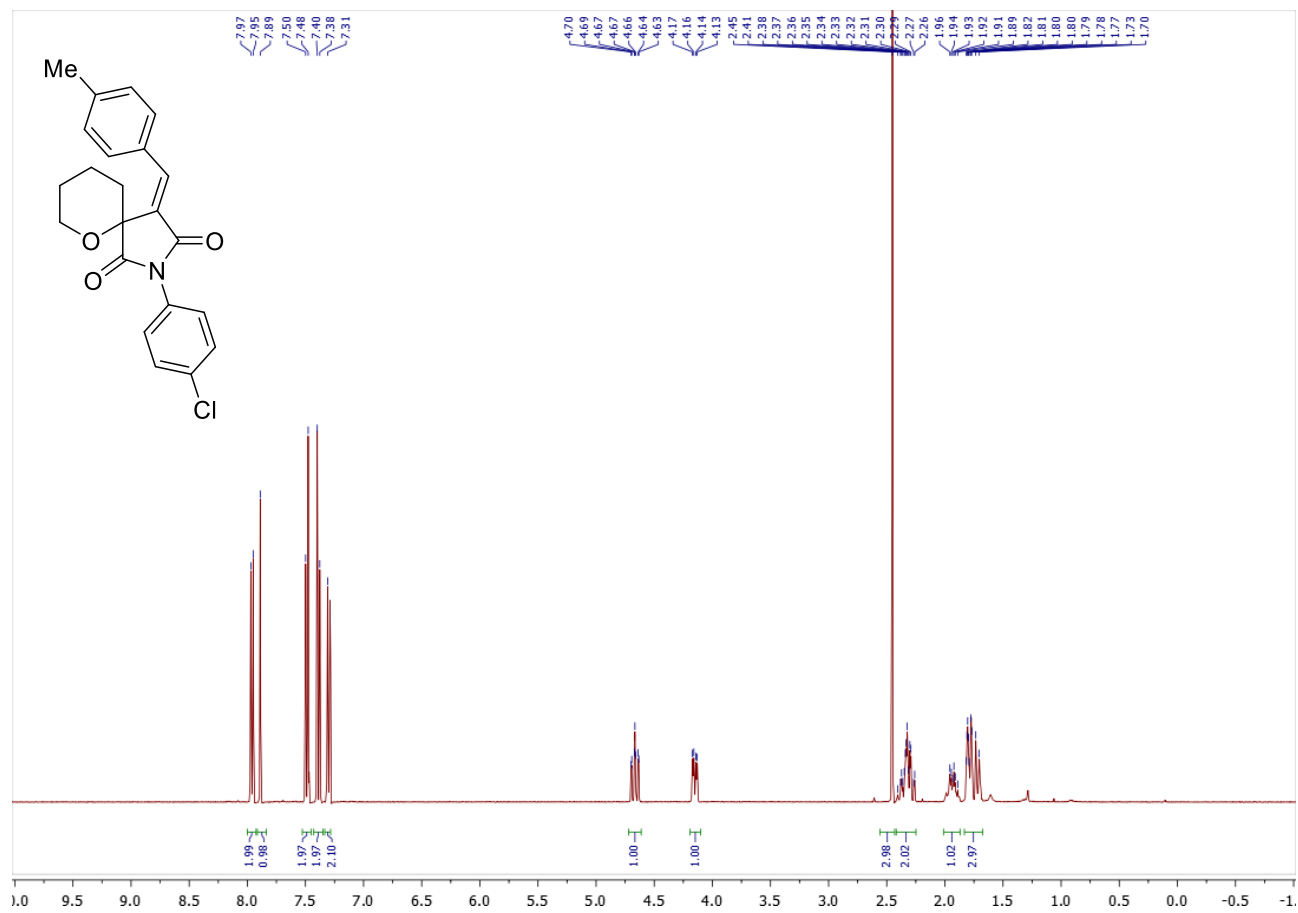
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*S/R,S/R*)-6t^{maj}



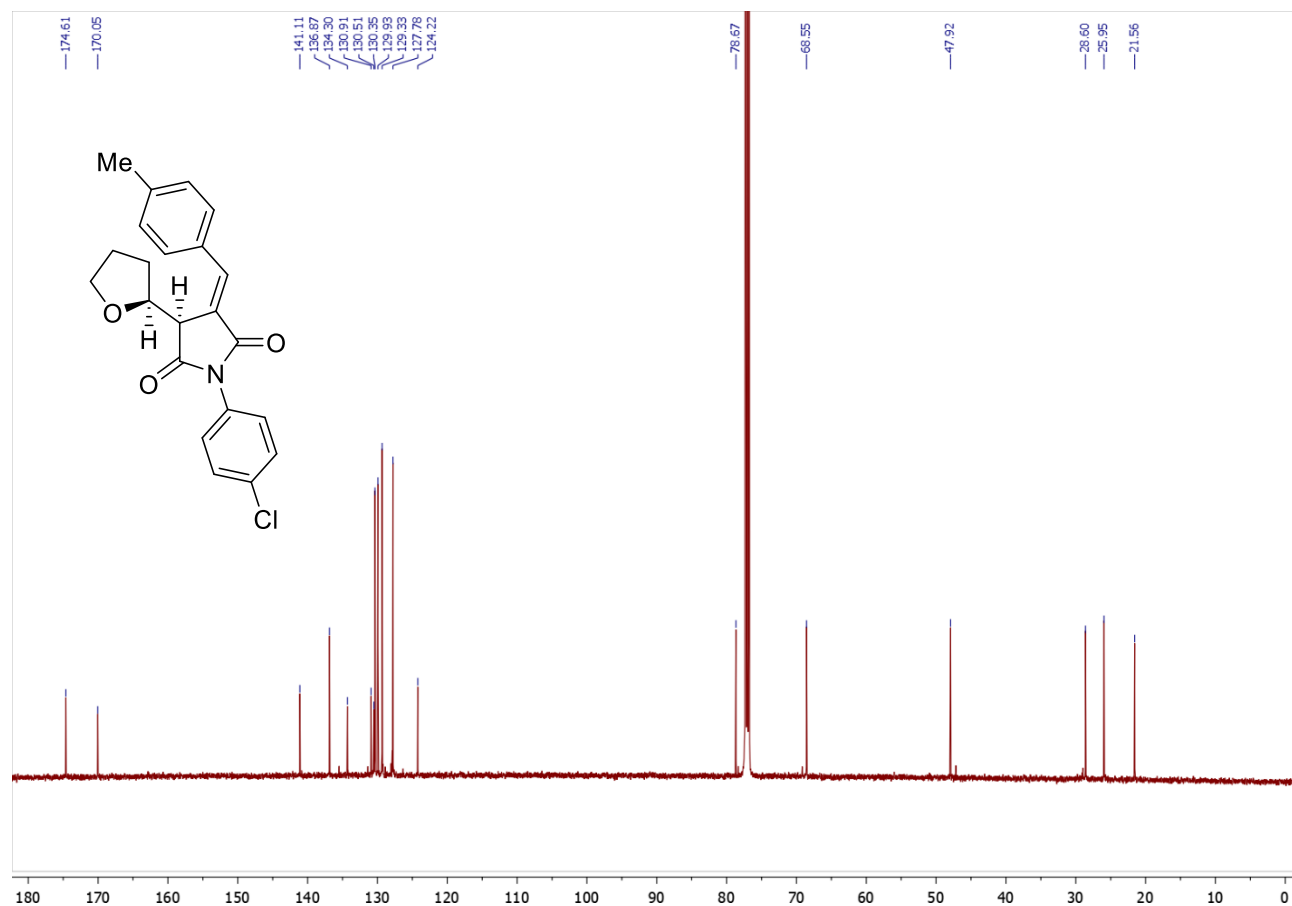
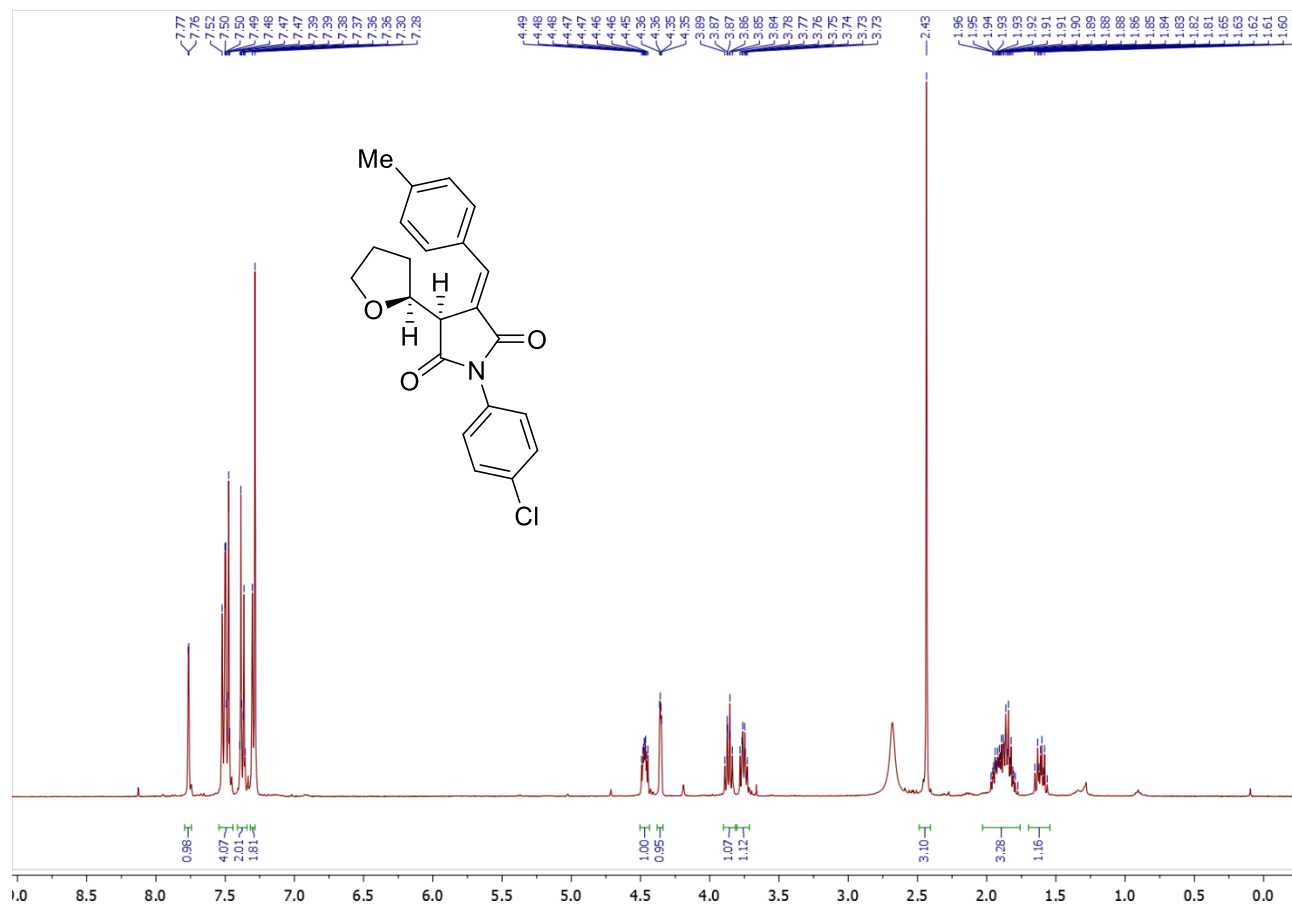
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2u**



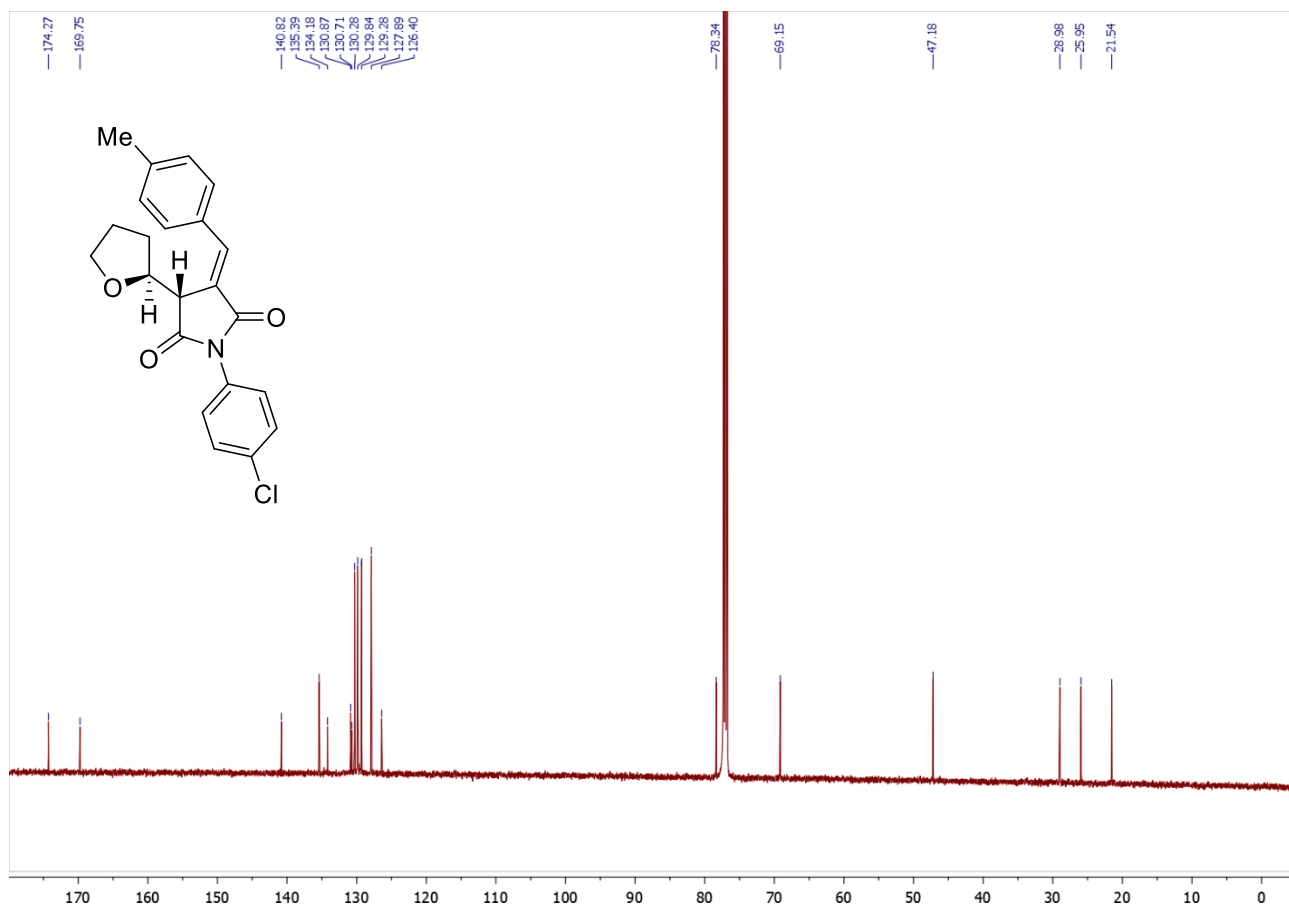
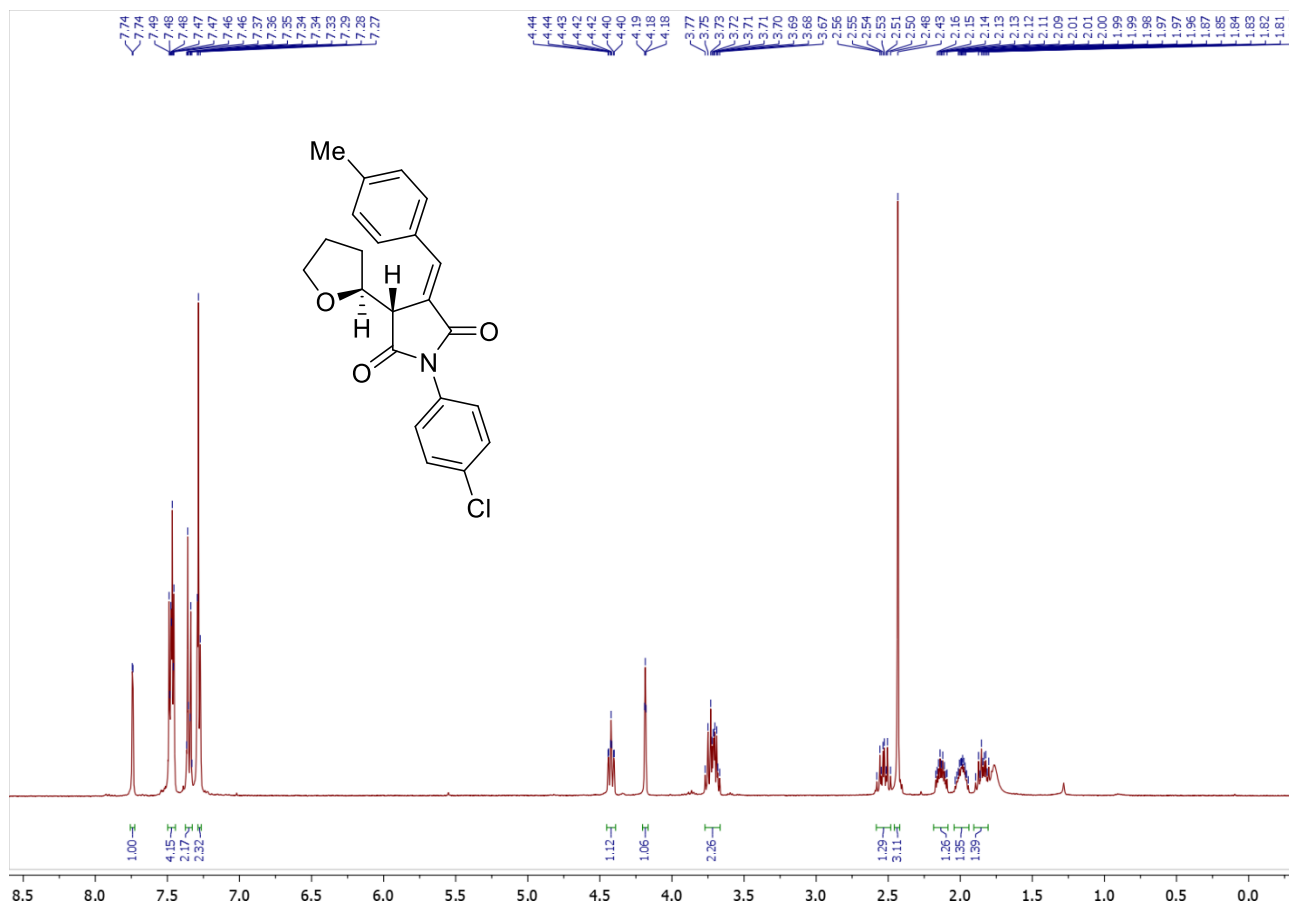
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2v**



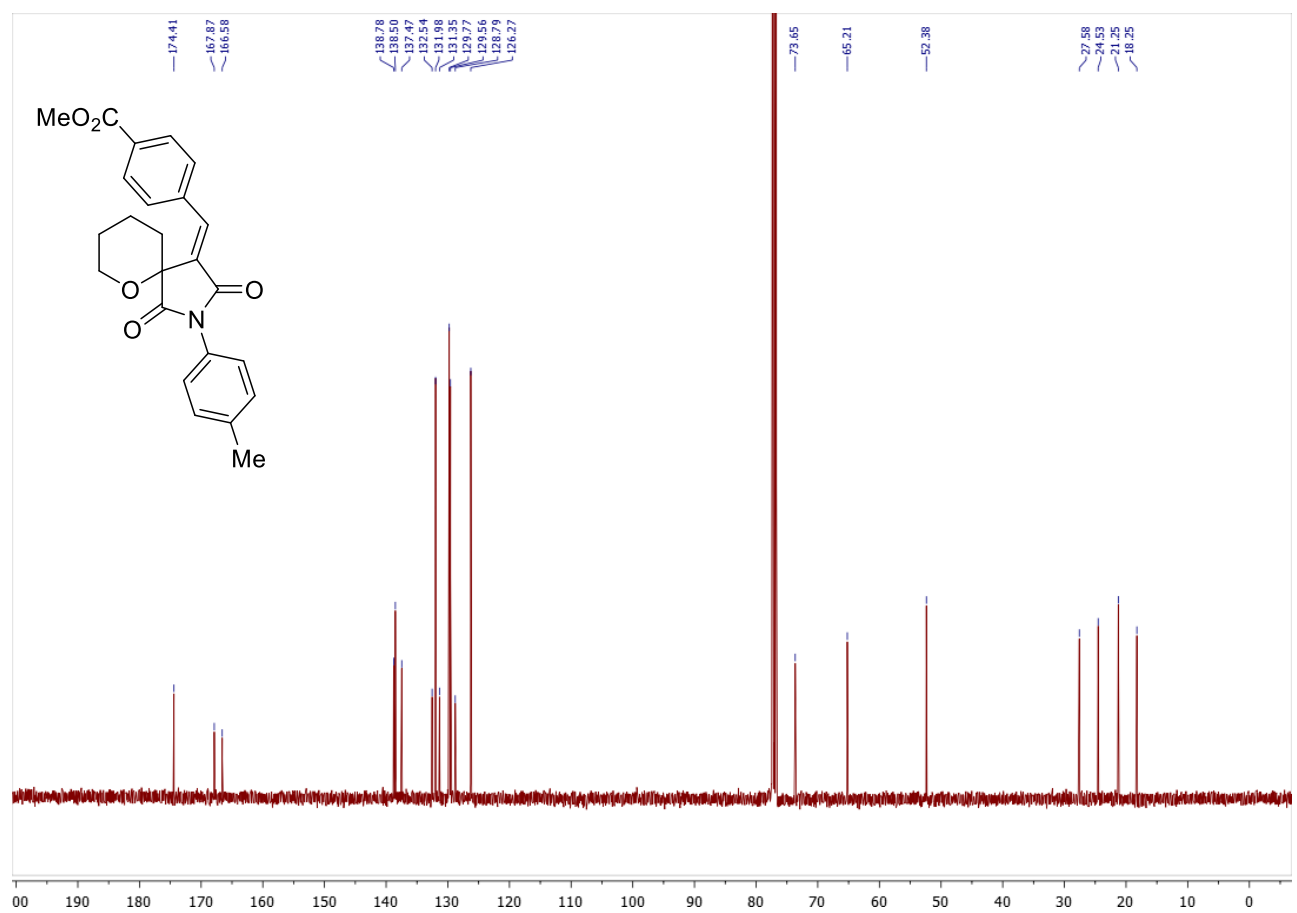
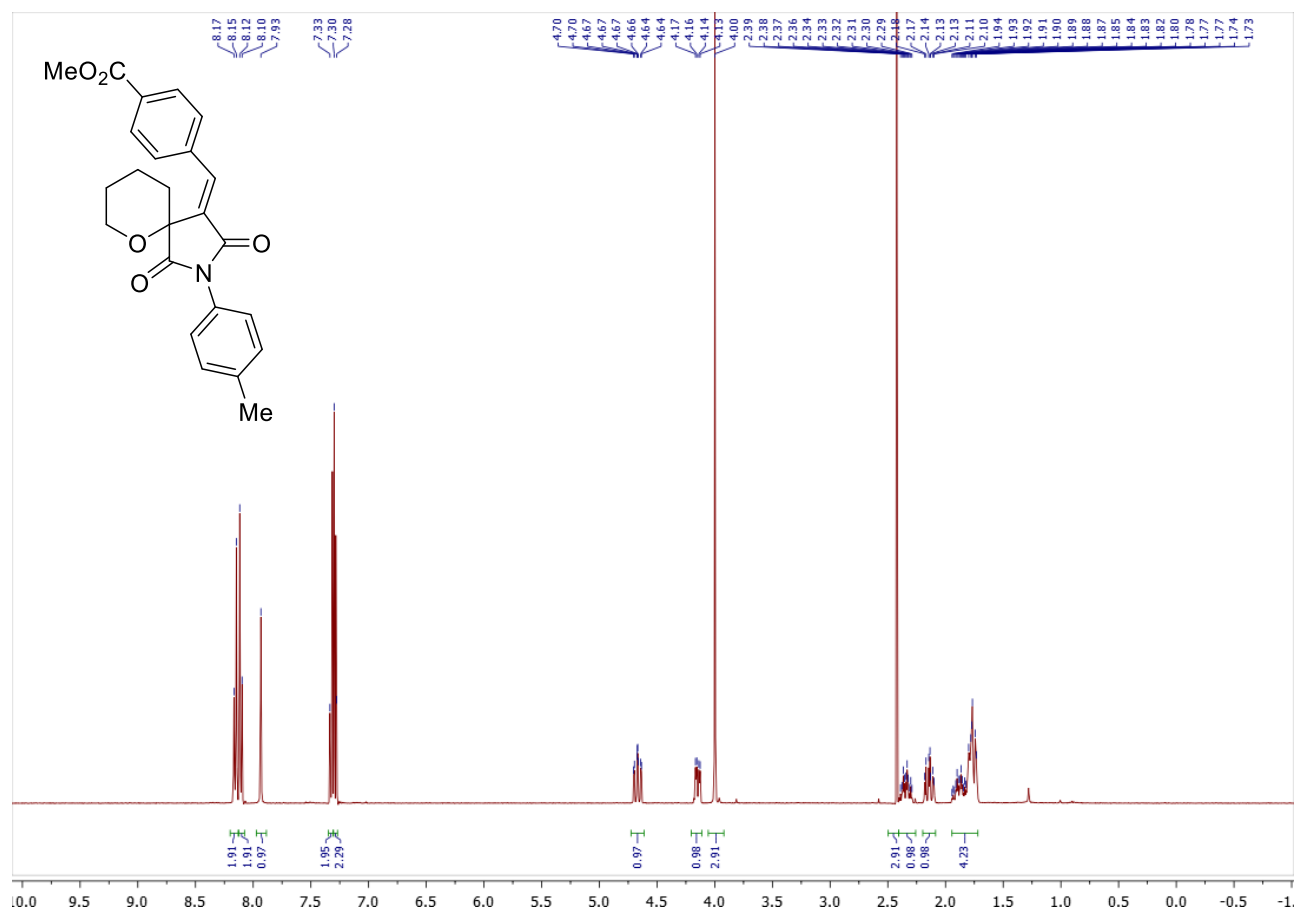
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*R/S,S/R*)-6 v^{min}



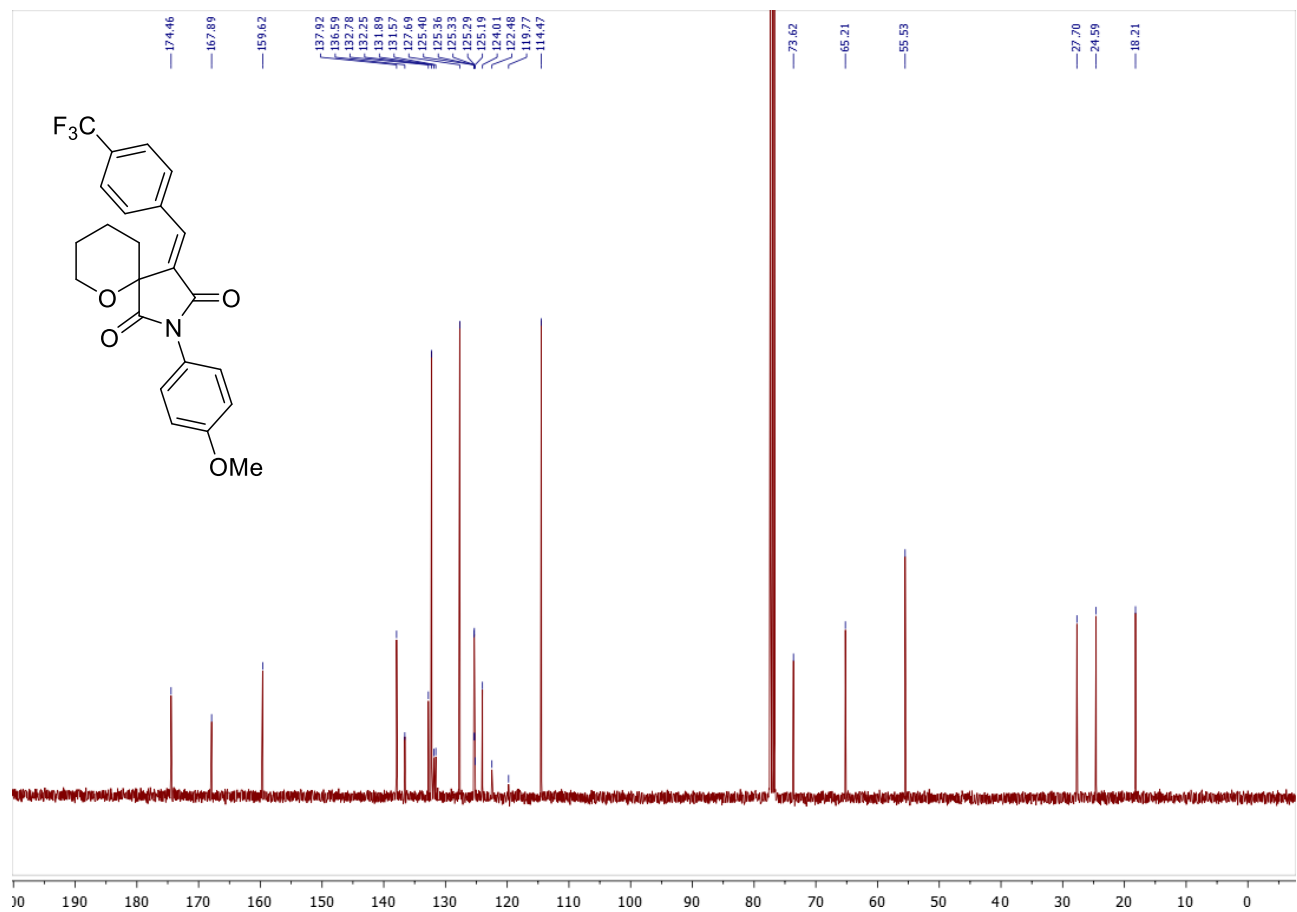
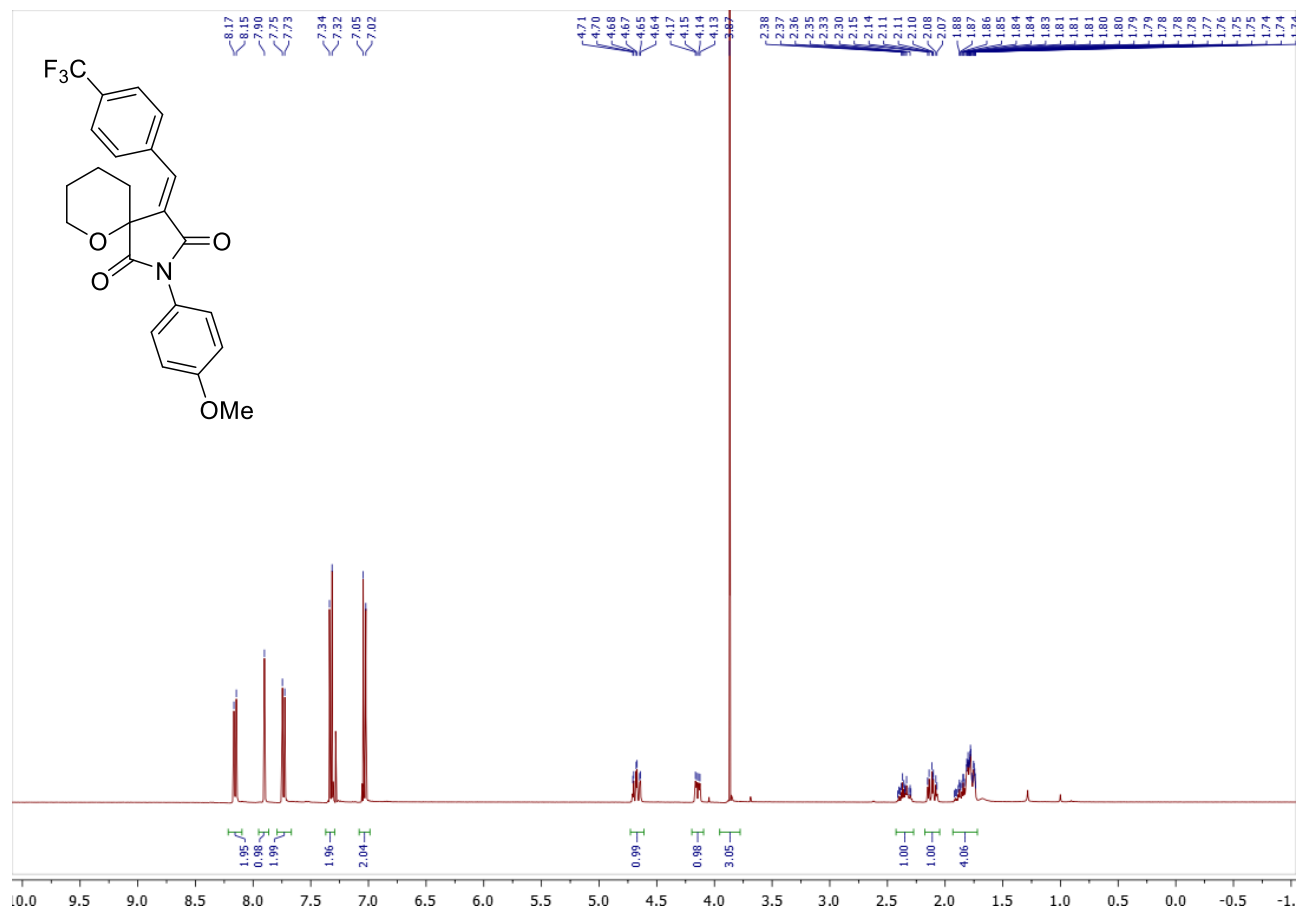
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*S/R,S/R*)-6v^{maj}



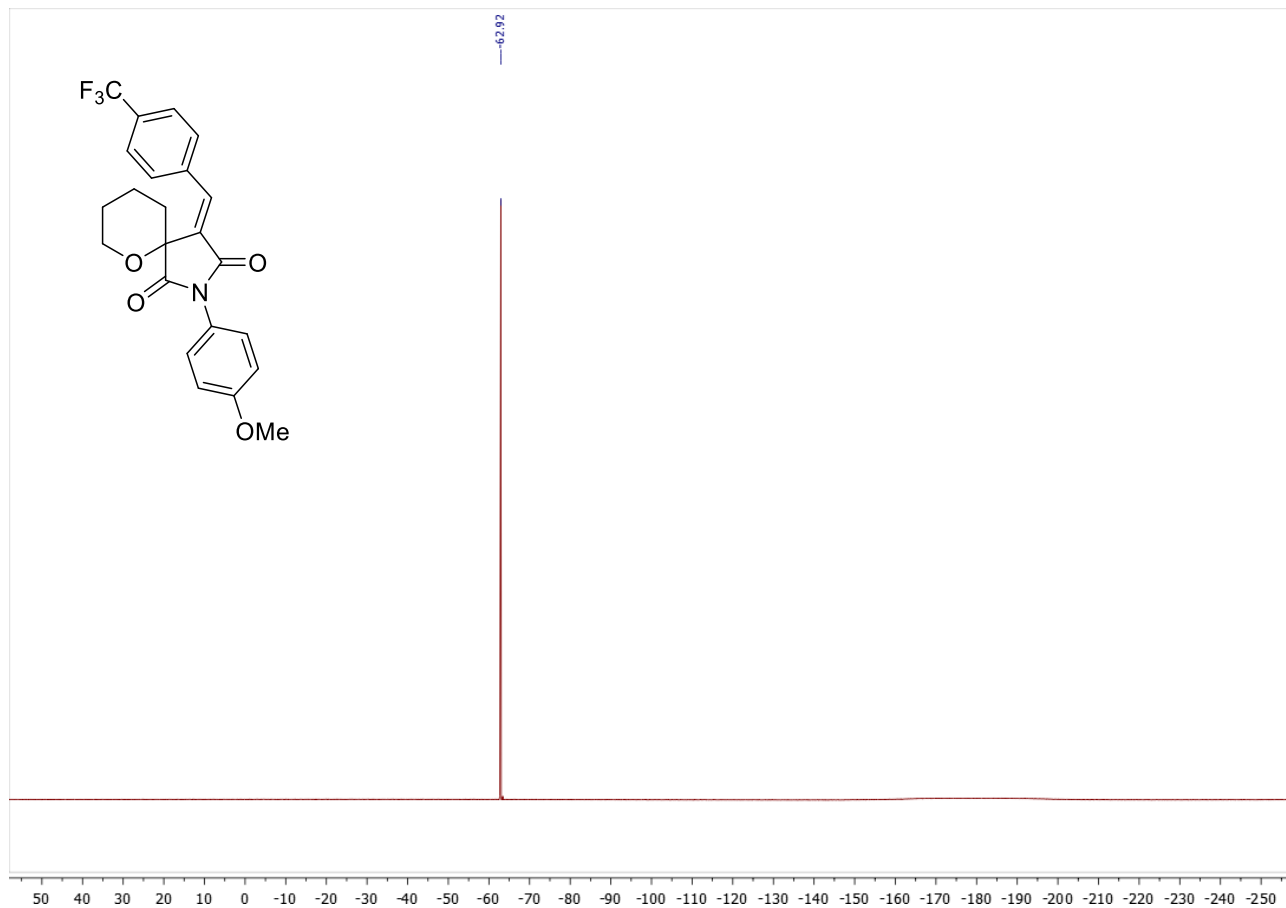
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2w**



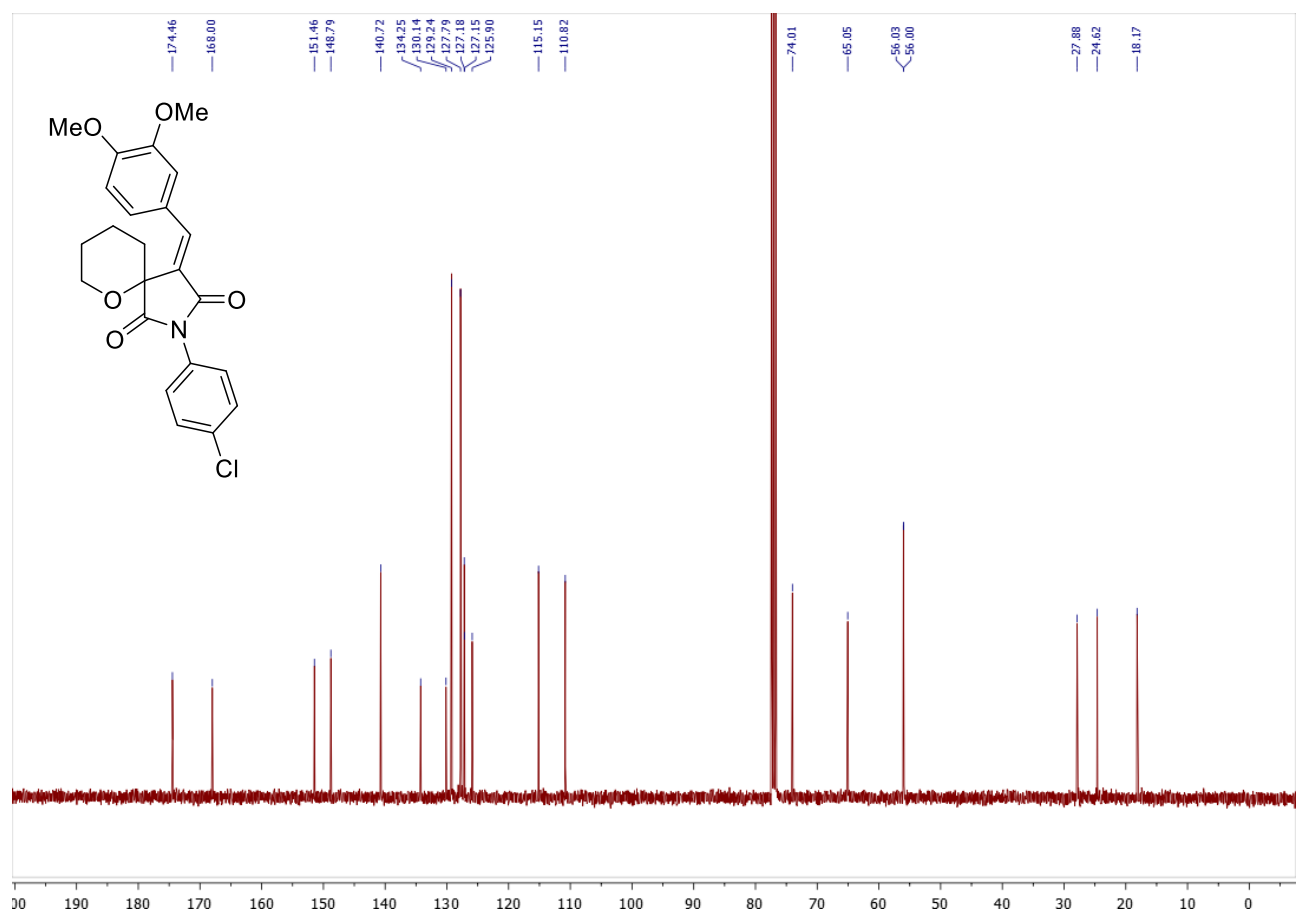
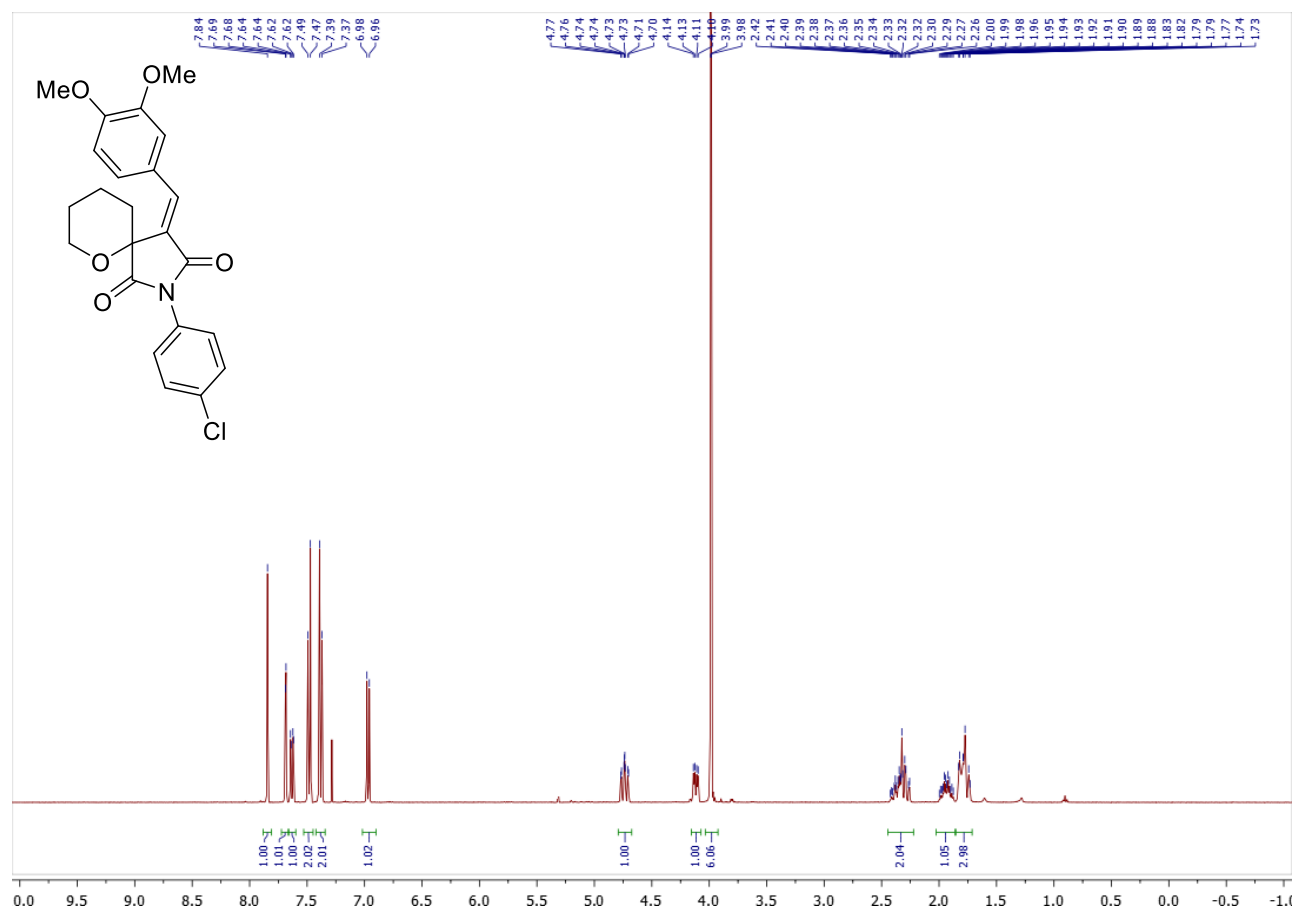
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2x**



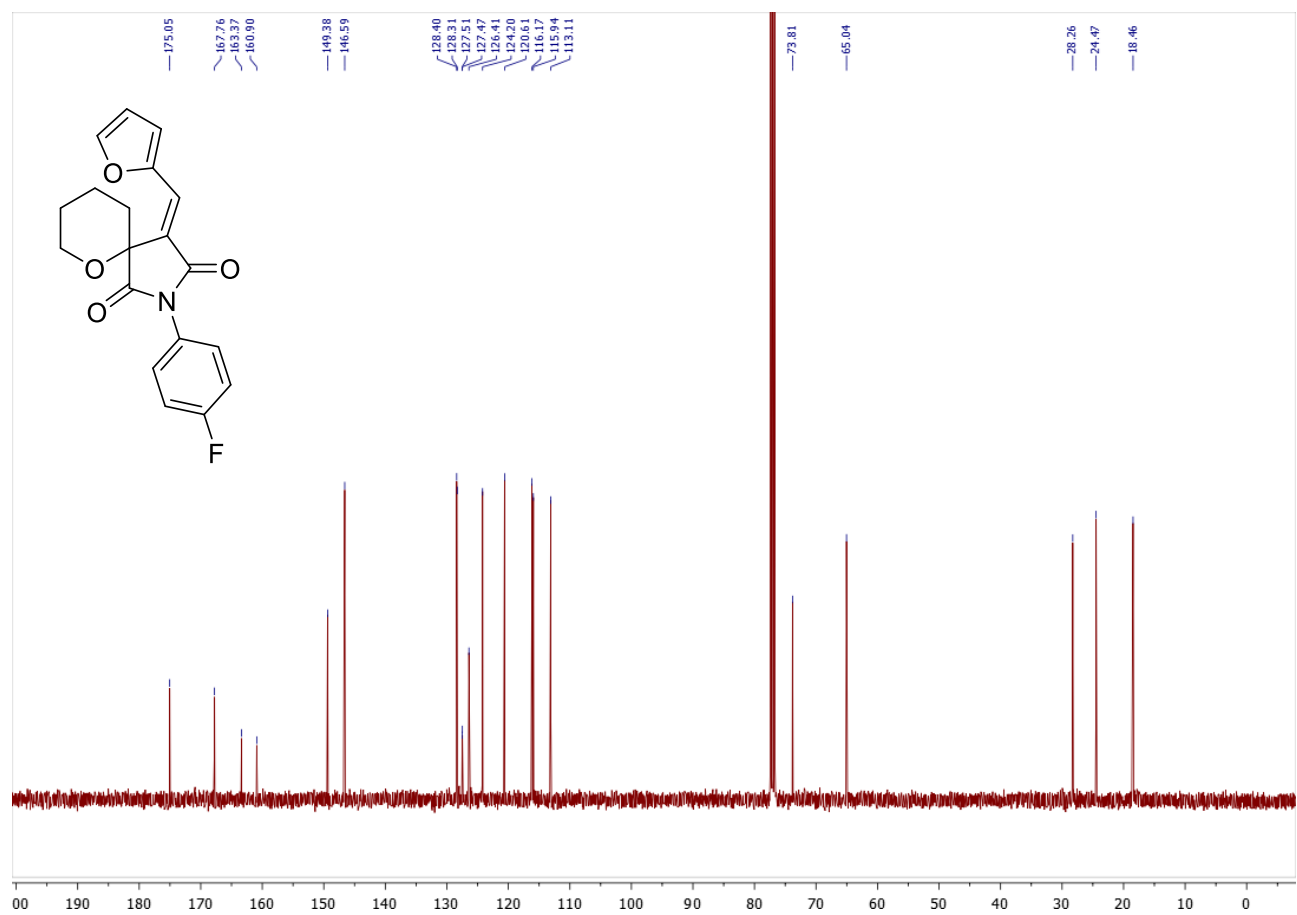
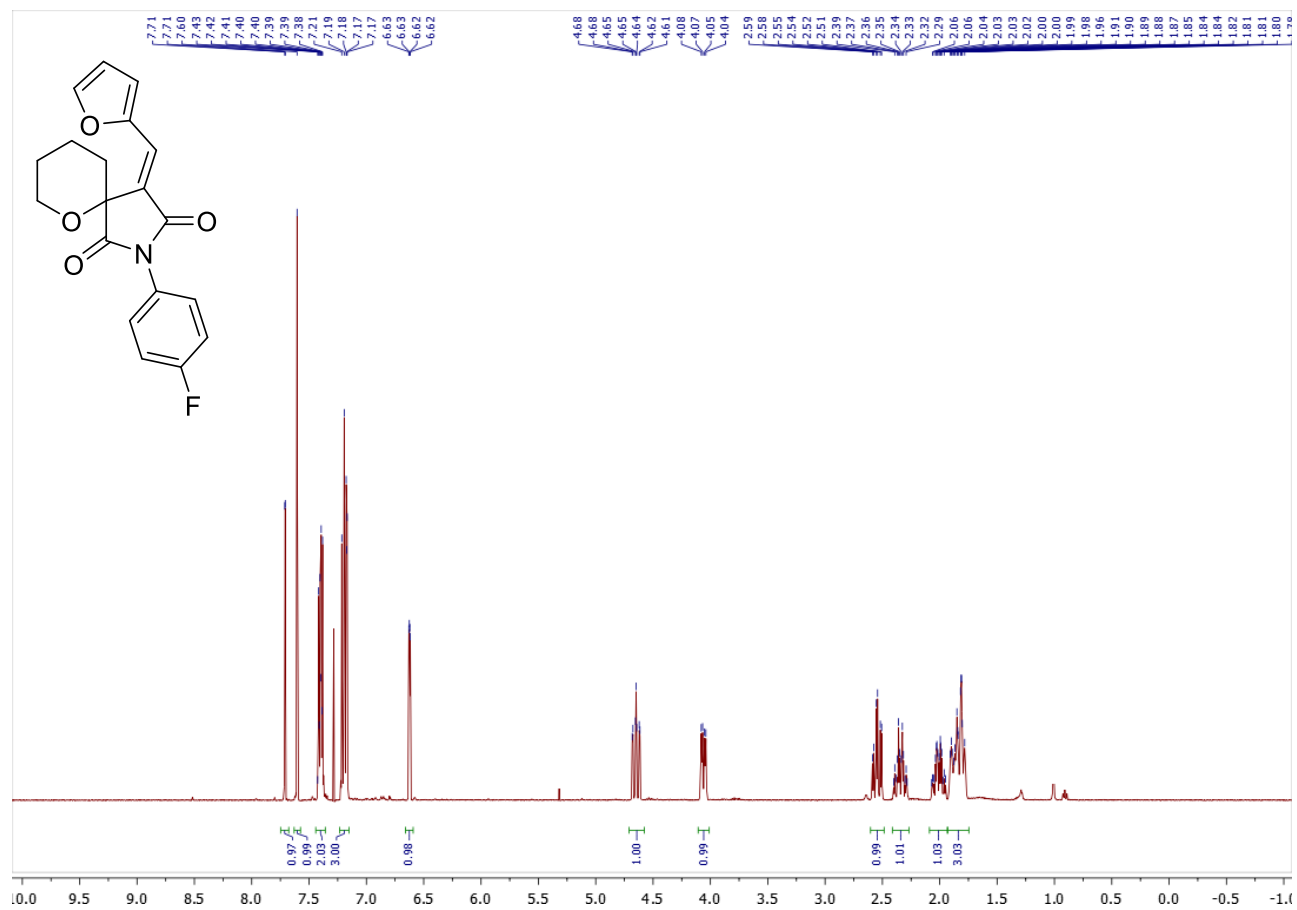
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of **2x**



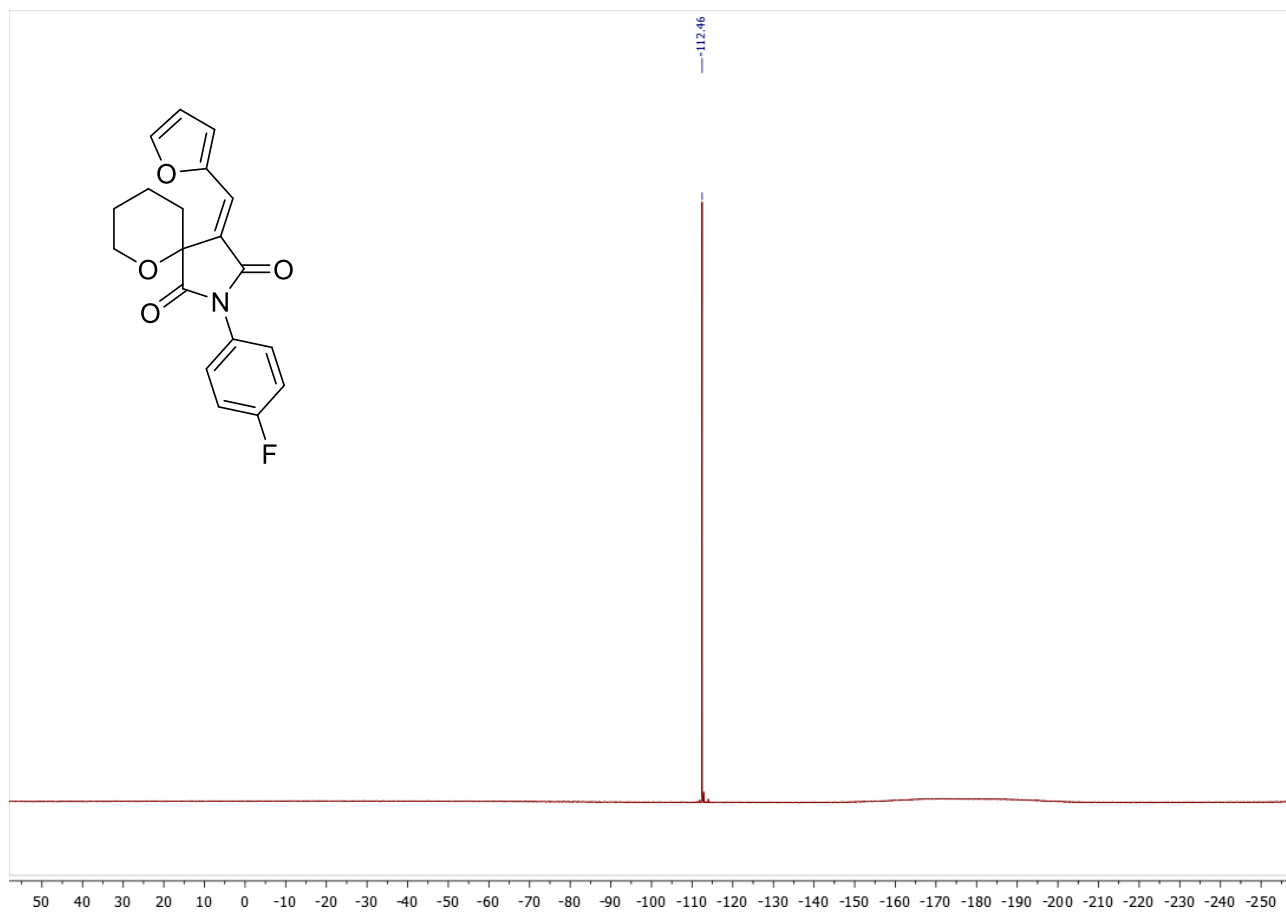
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2y**



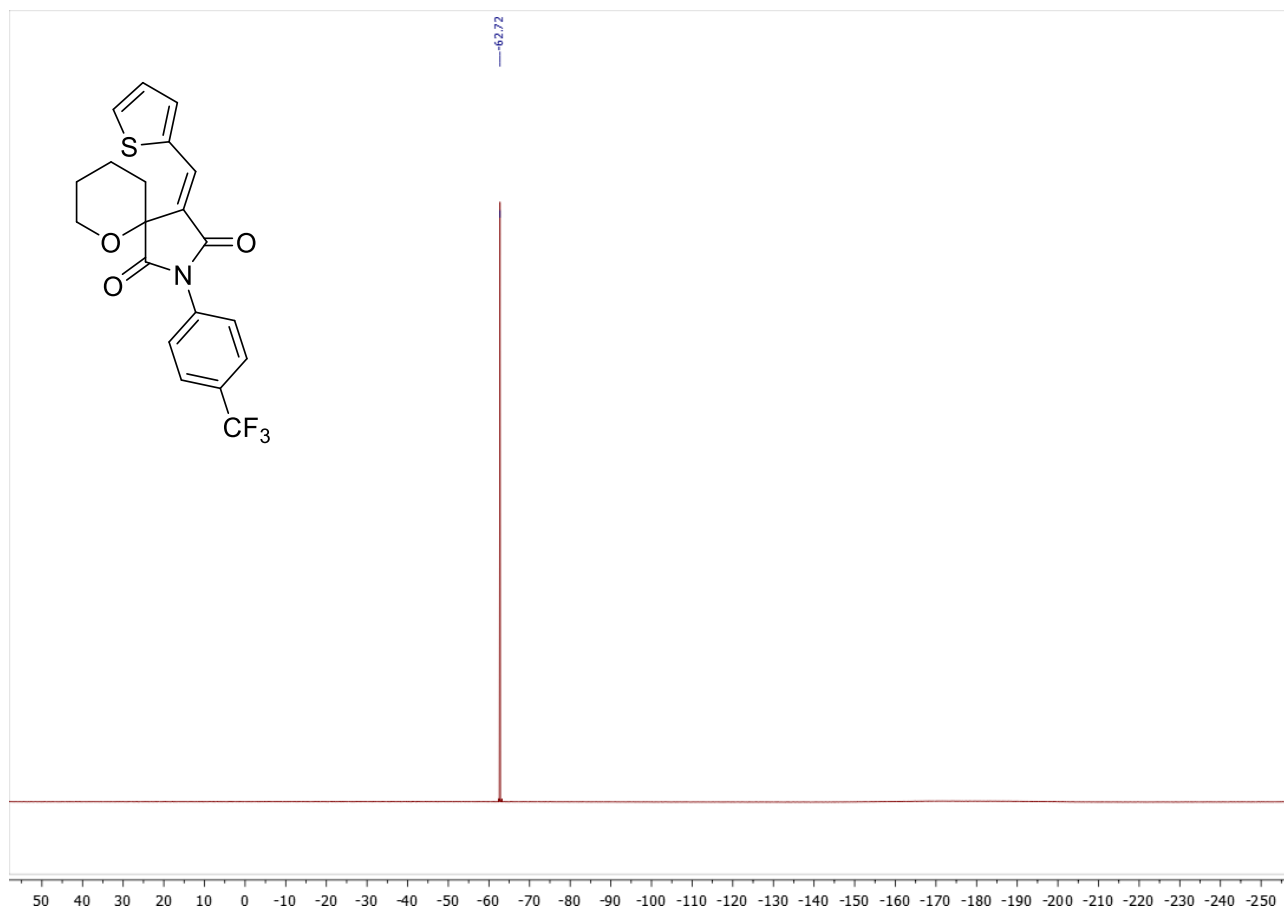
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2z**



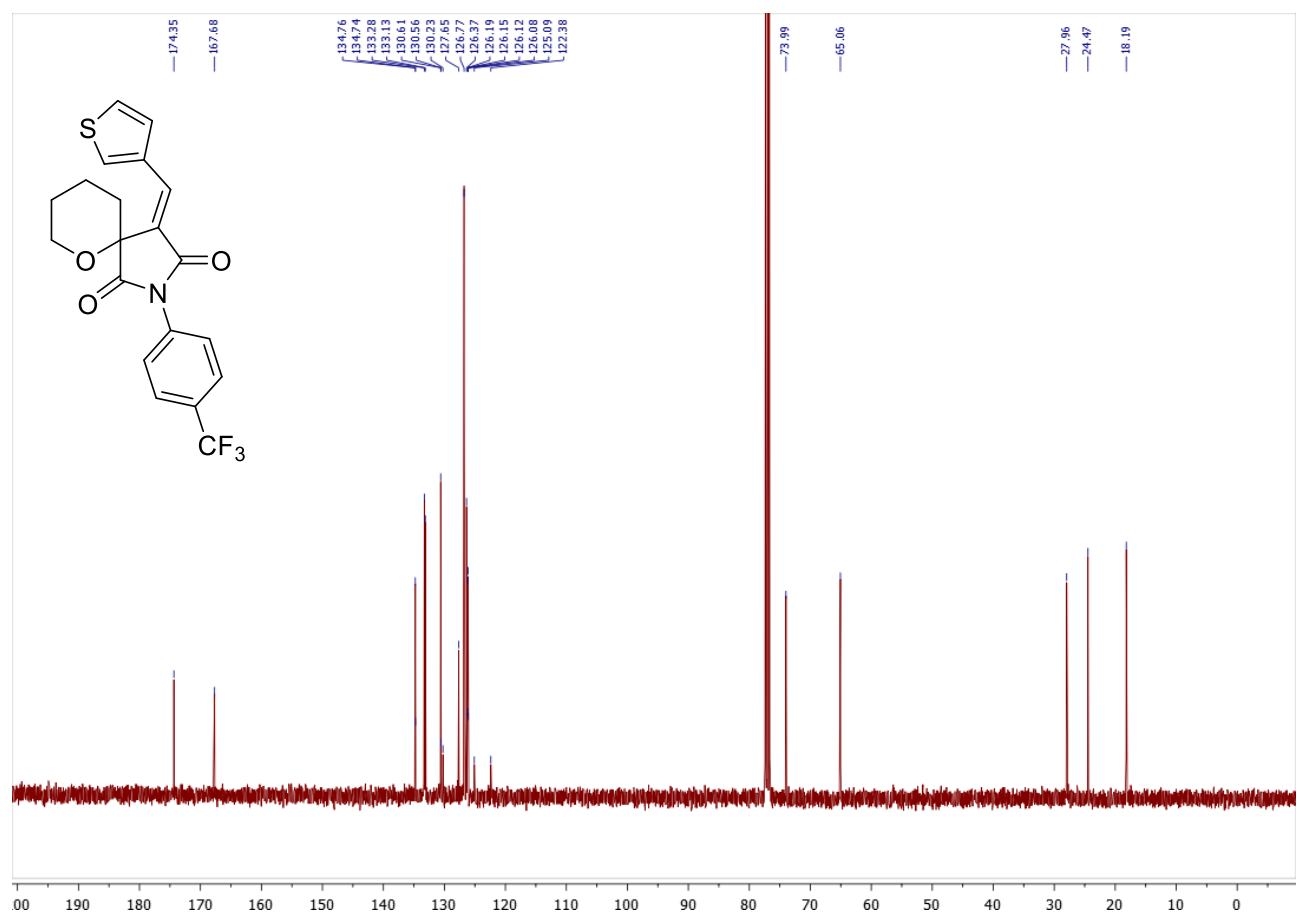
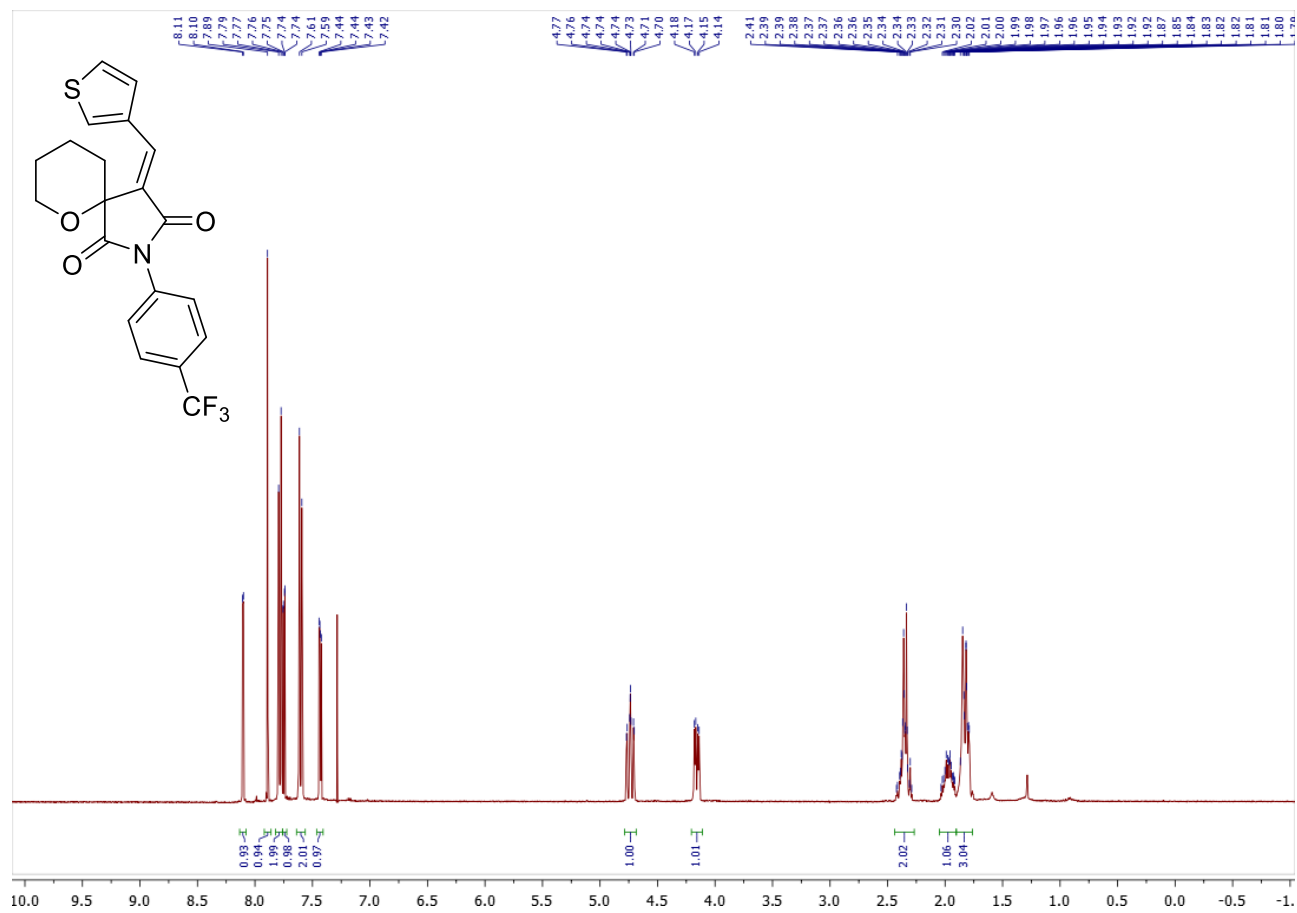
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of **2z**



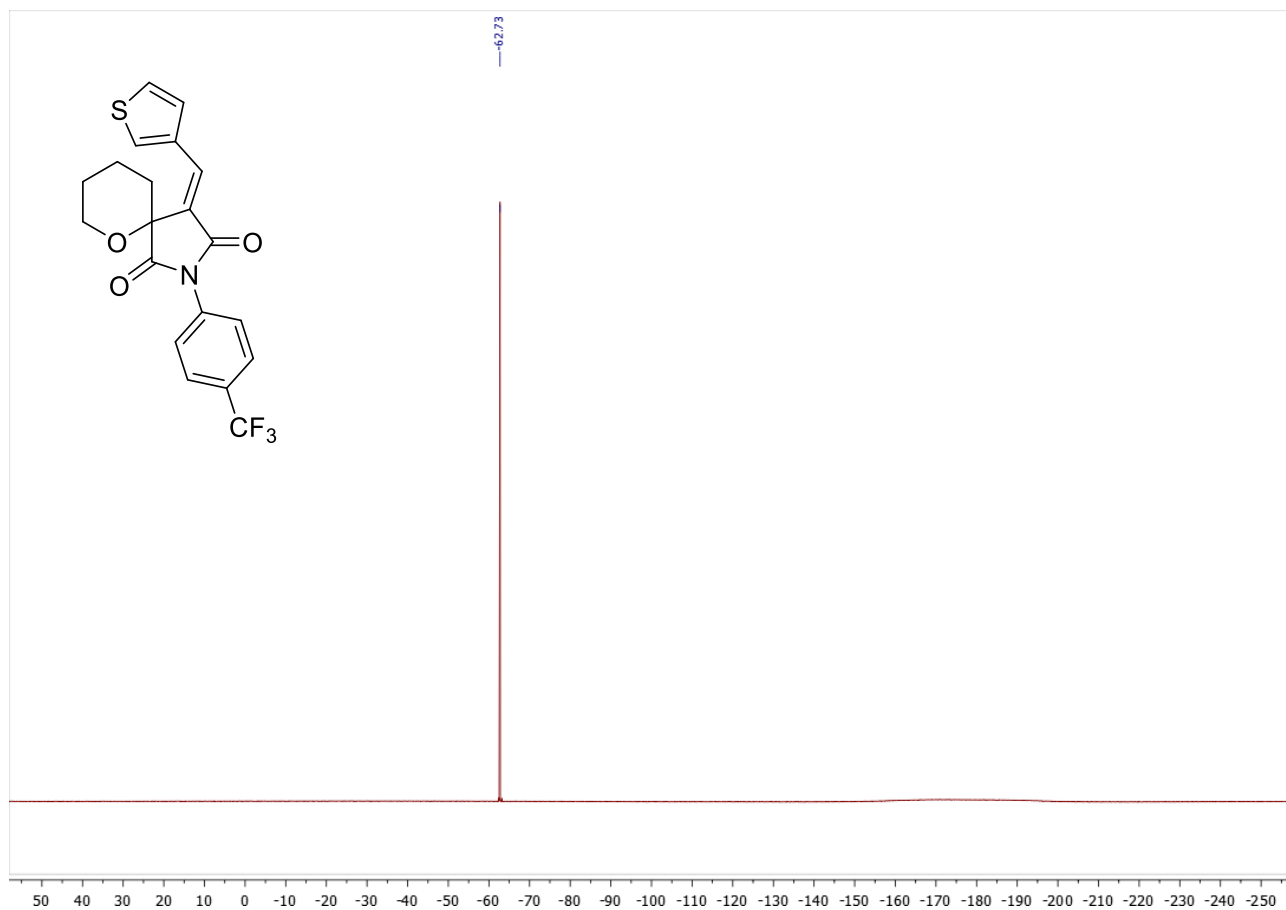
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of **2aa**



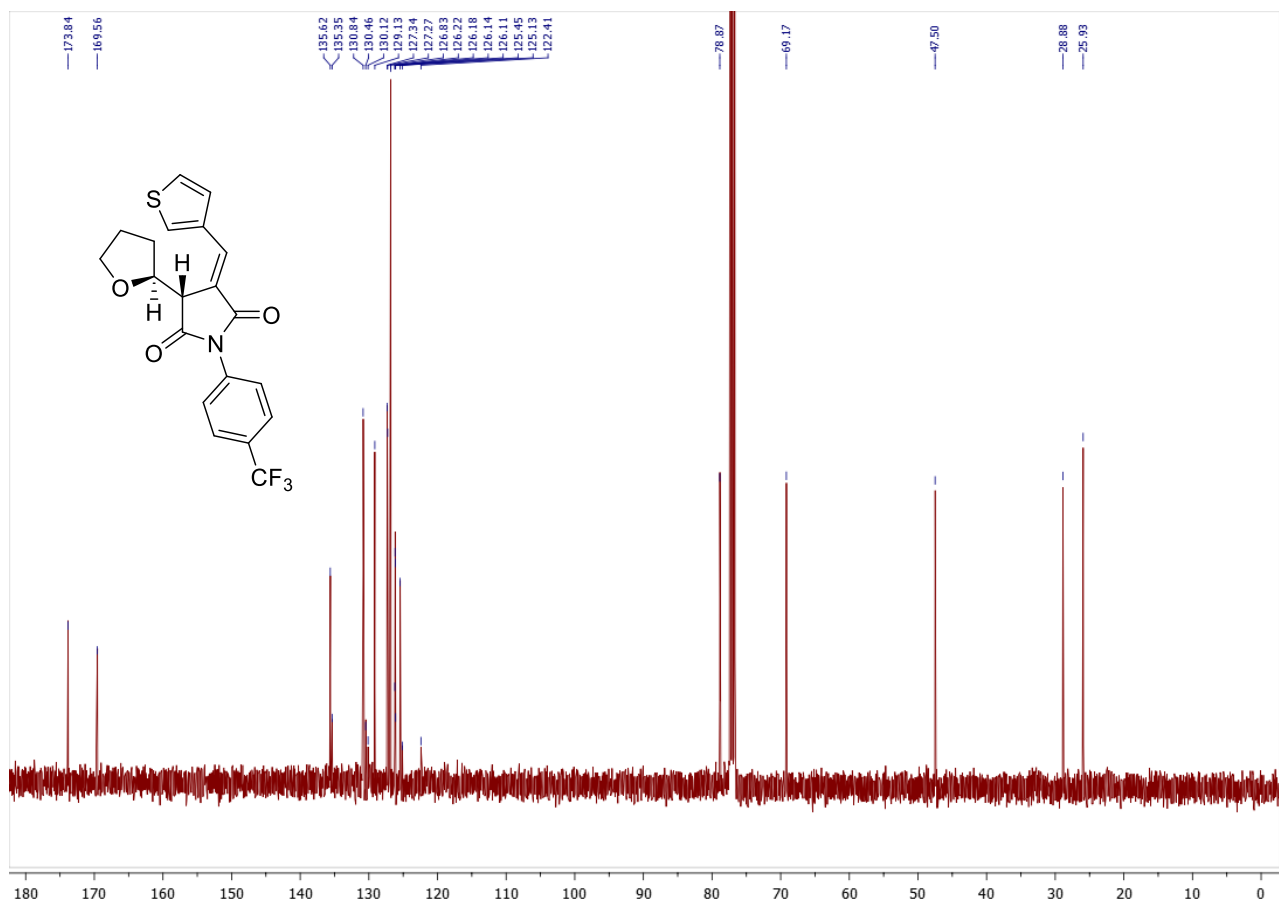
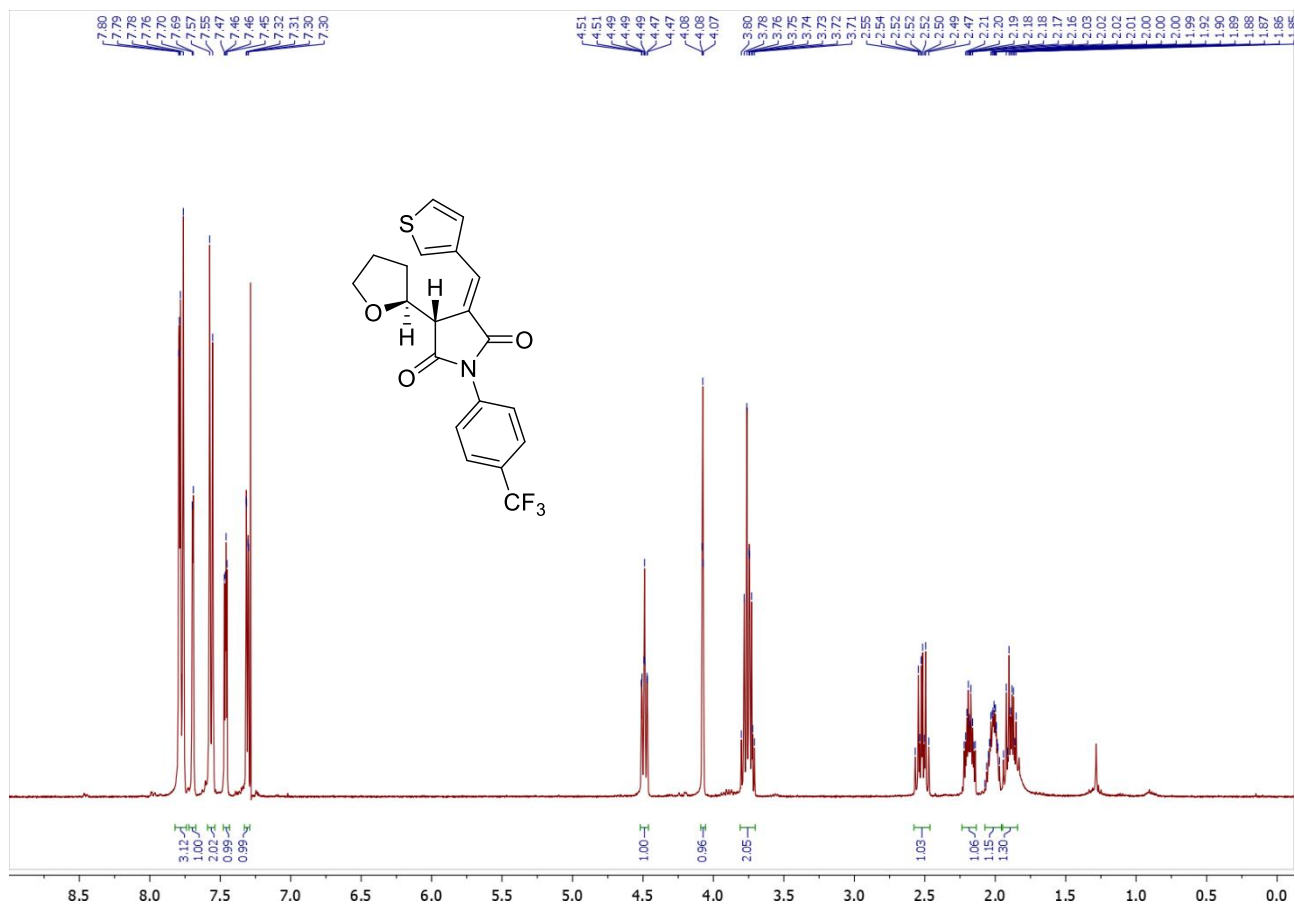
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2ab**



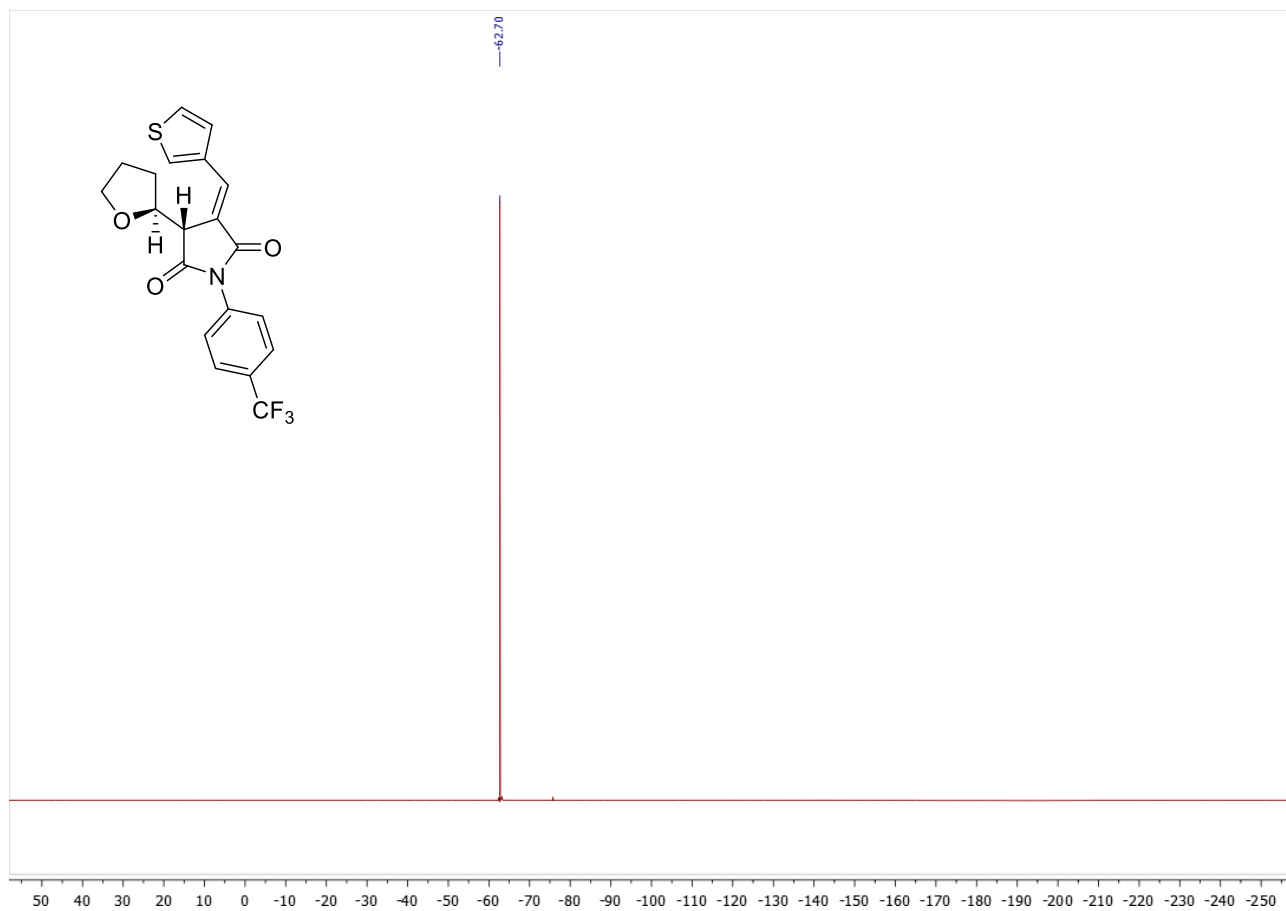
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of **2ab**



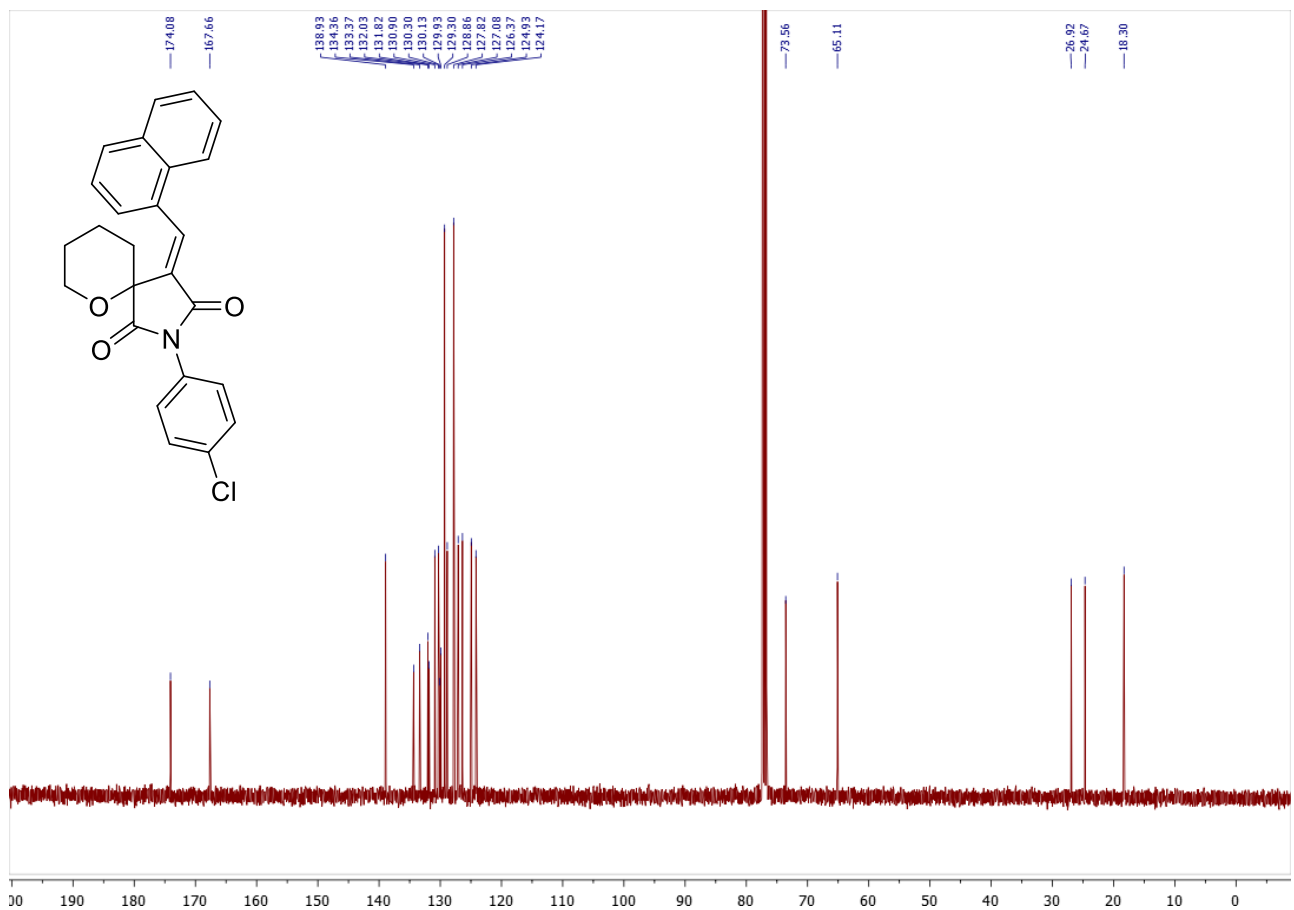
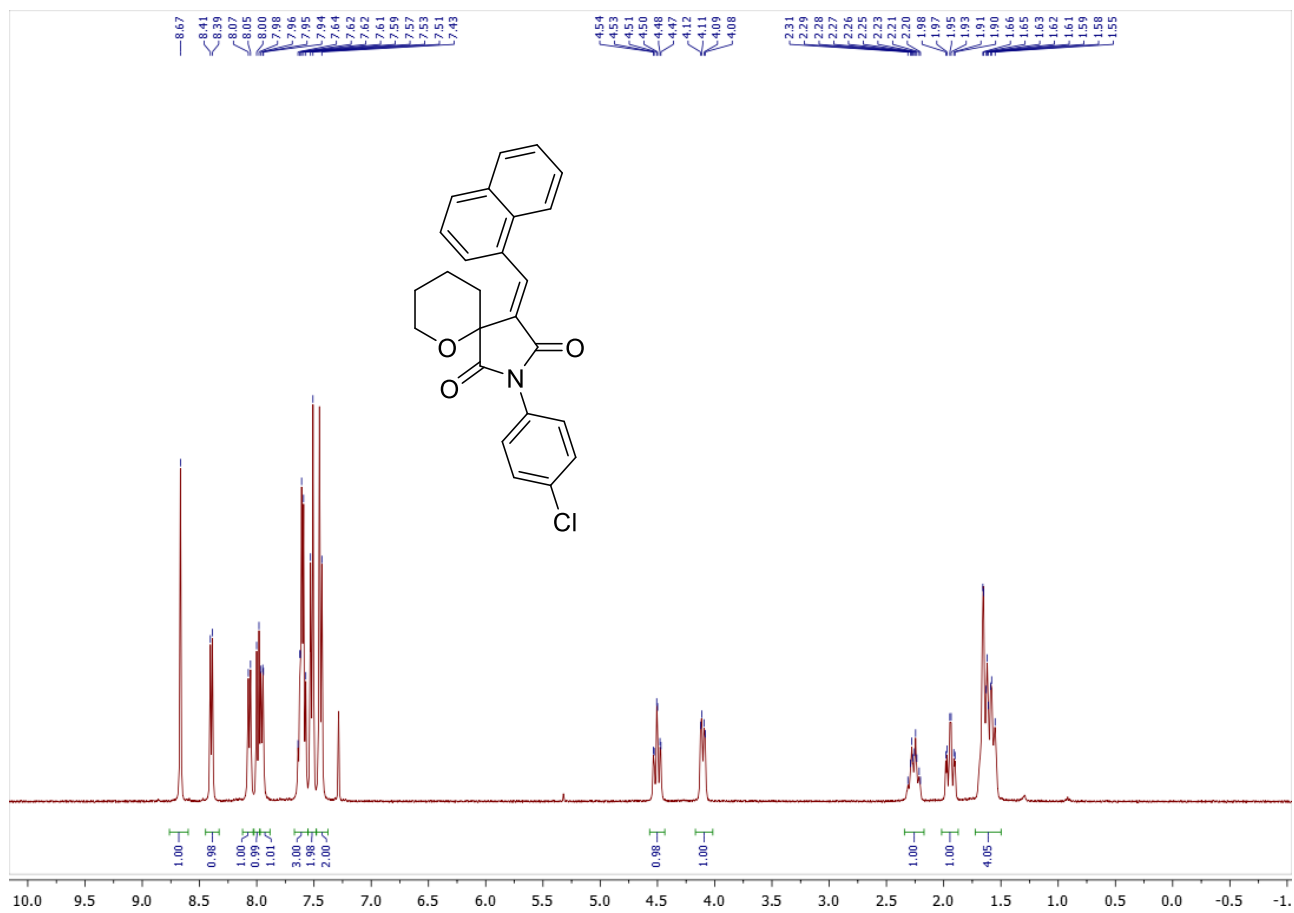
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*S/R,S/R*)-6ab^{maj}



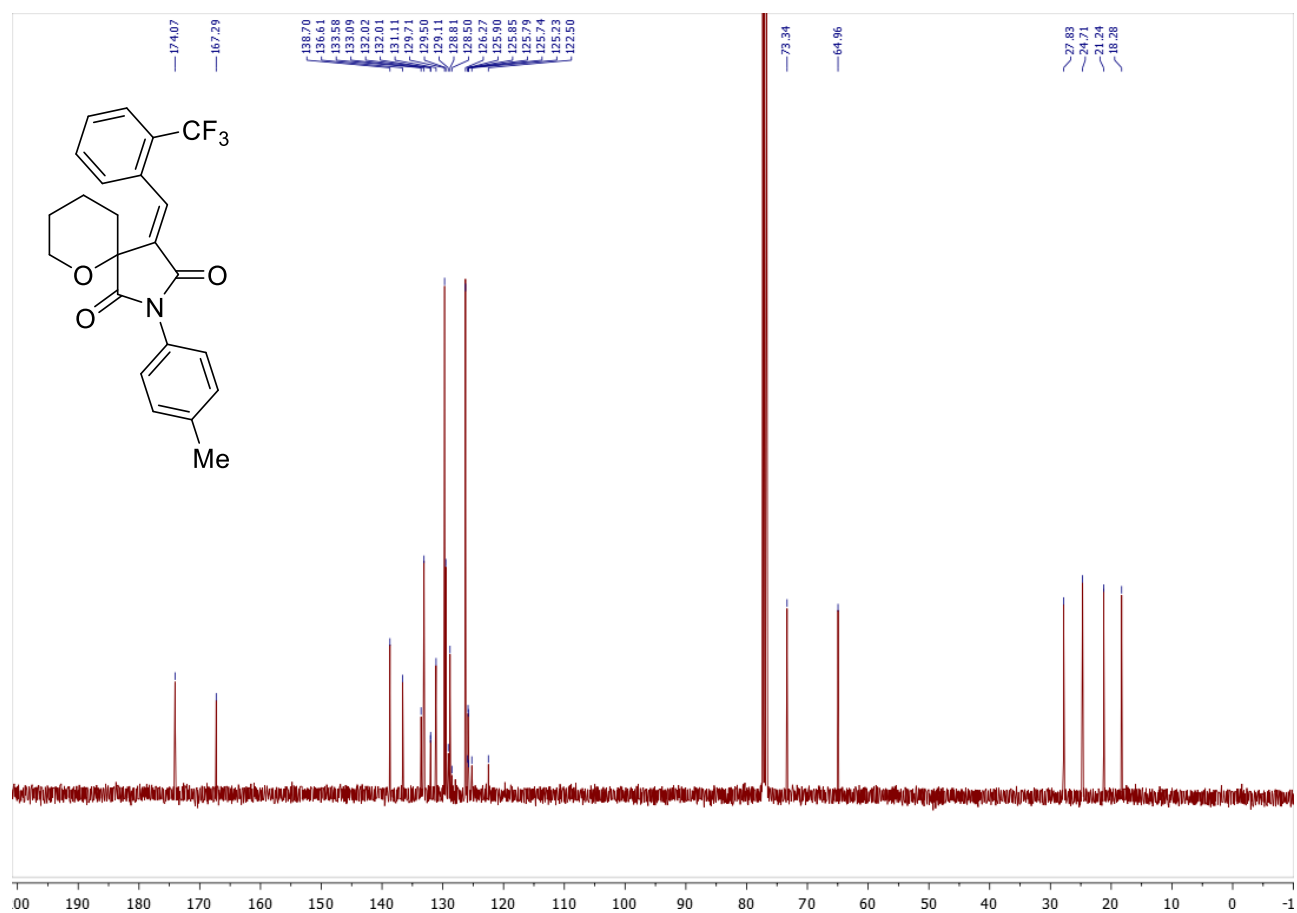
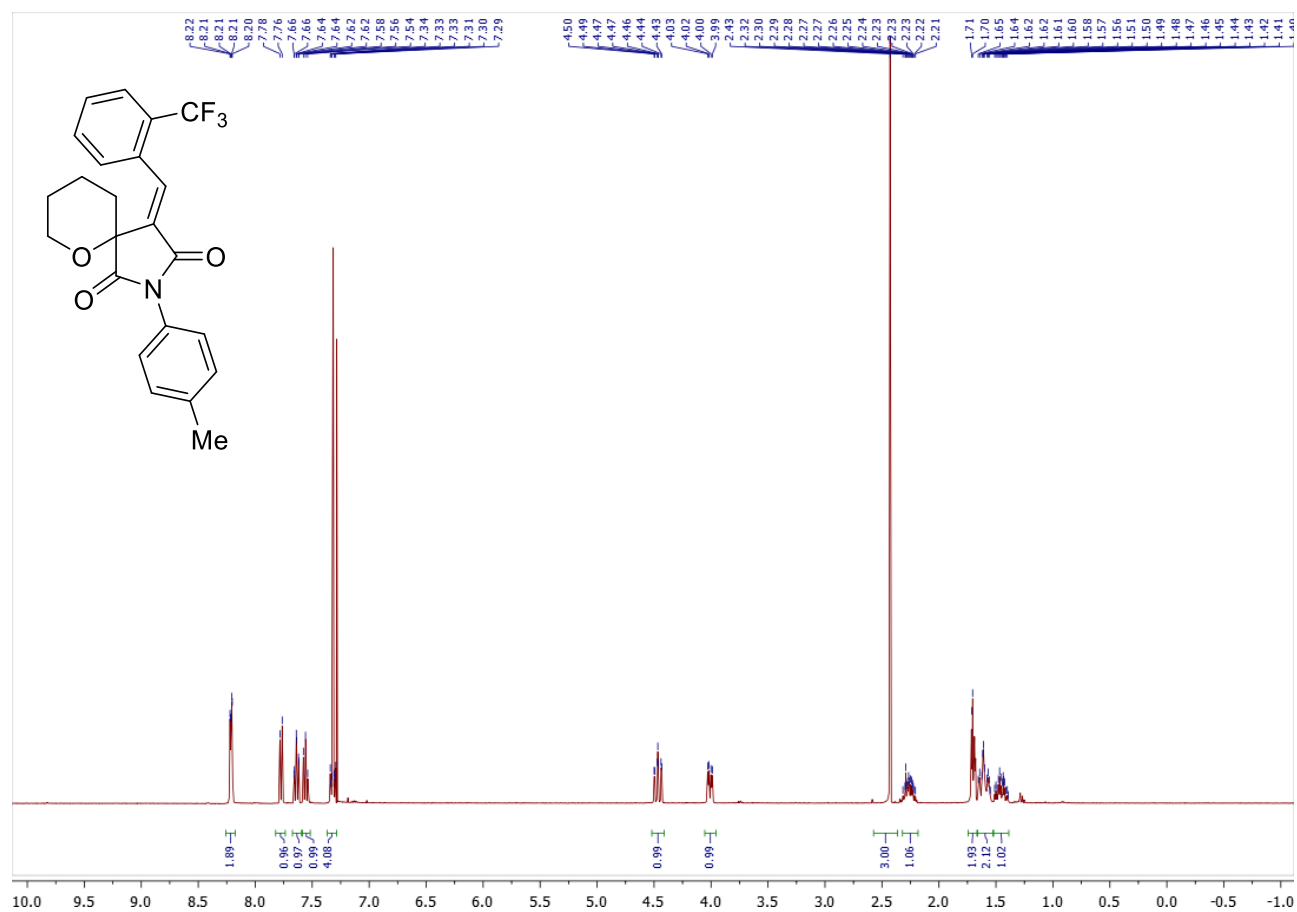
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of (*S/R,S/R*)-**6ab**^{maj}



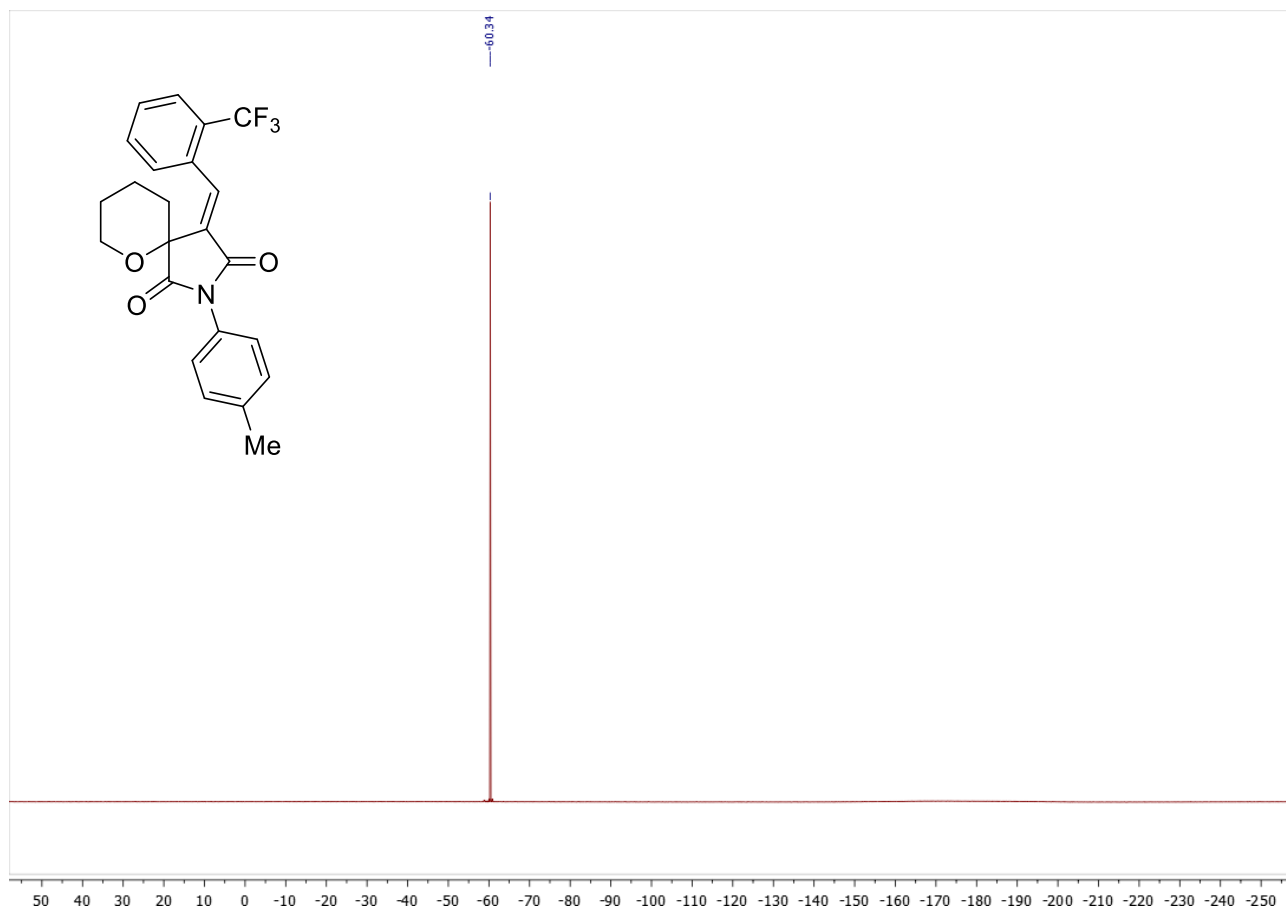
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2ac**



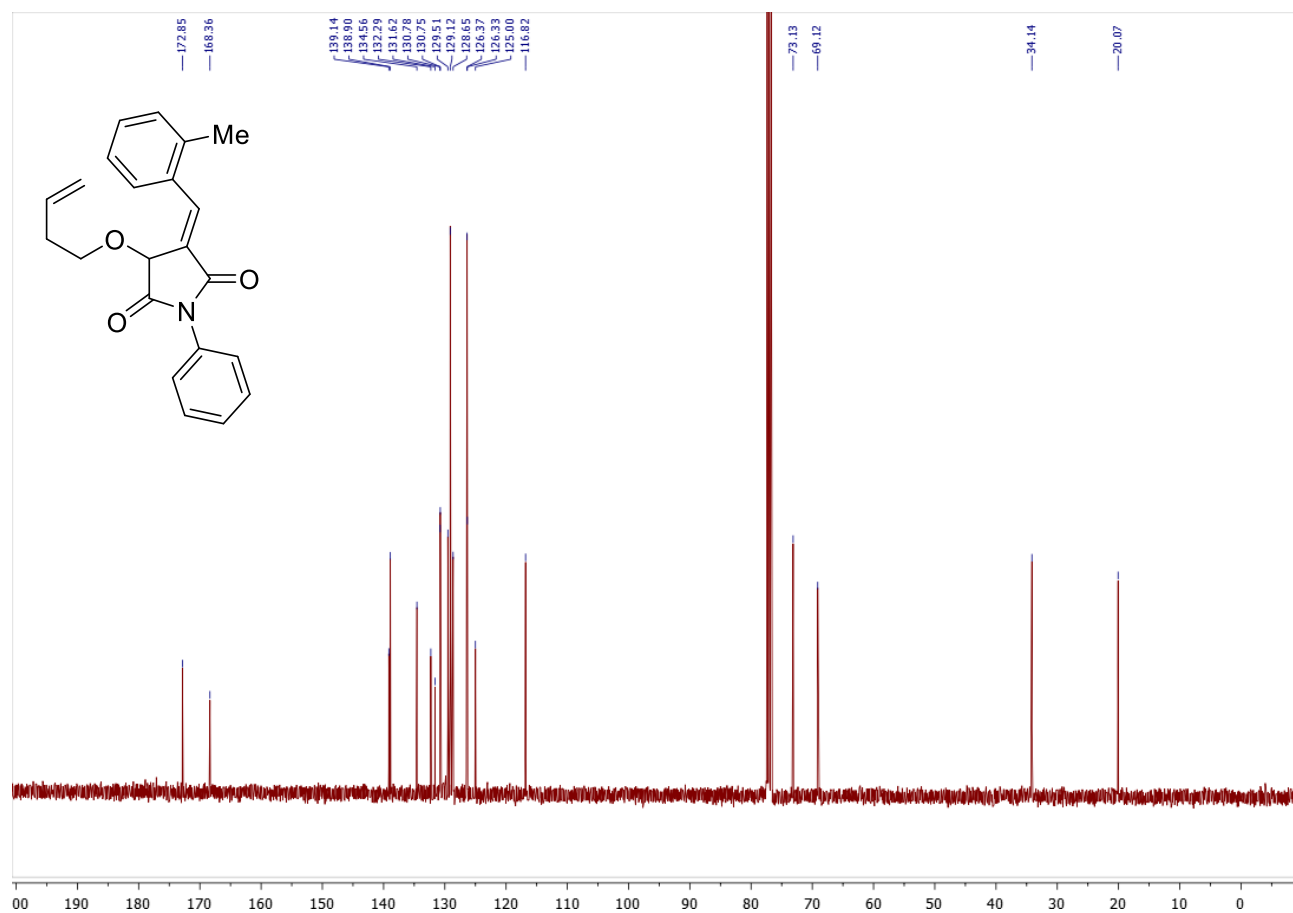
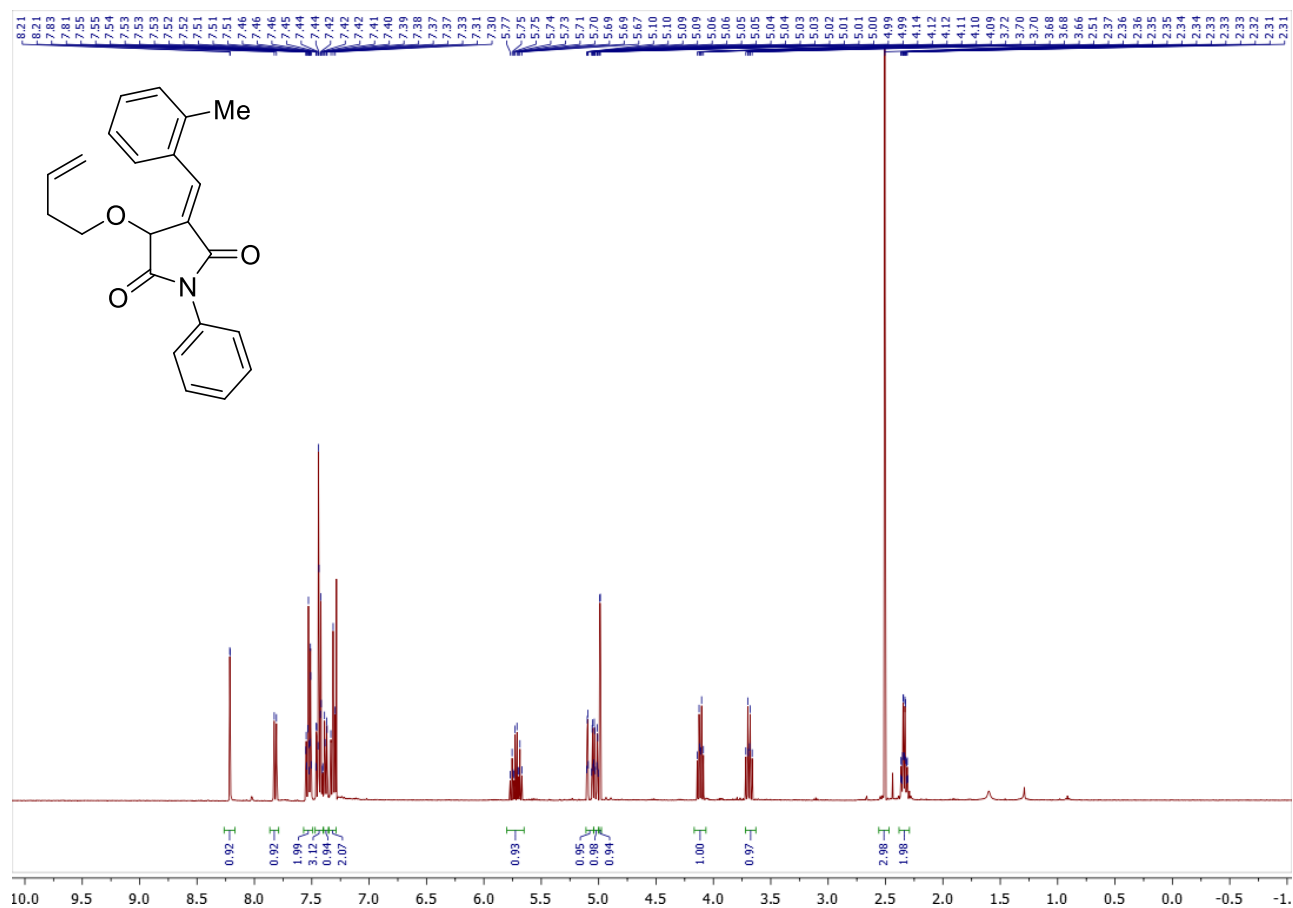
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **2ad**



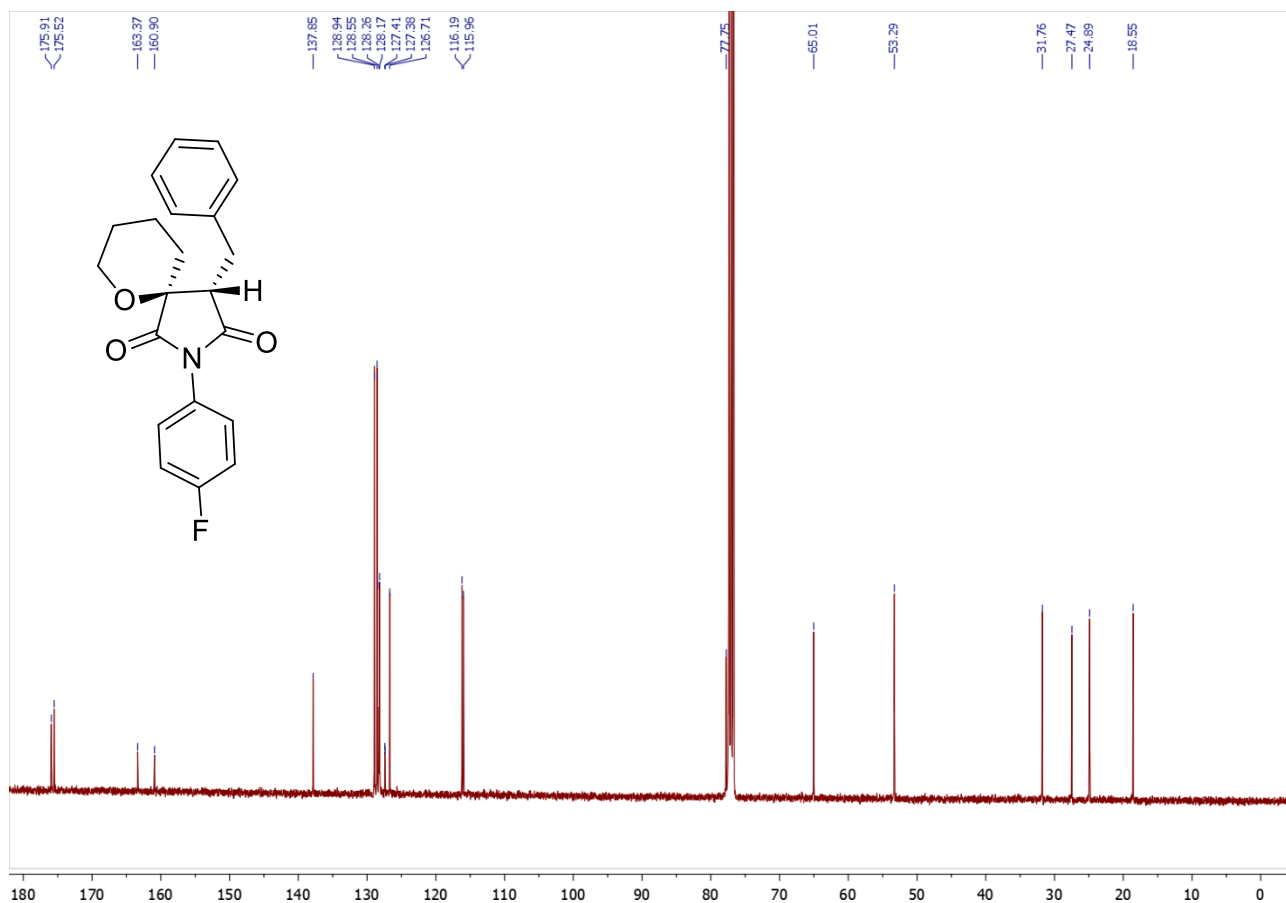
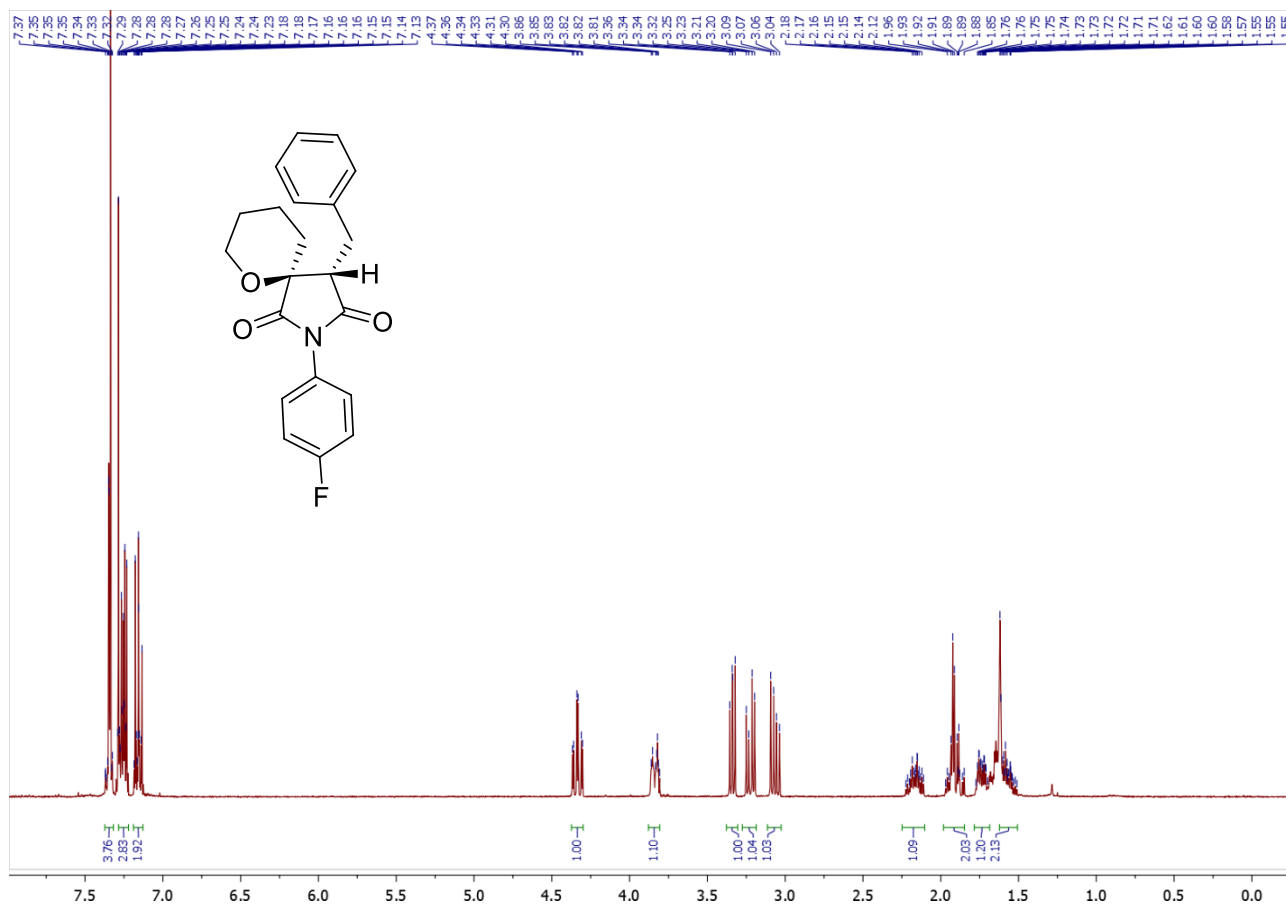
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of **2ad**



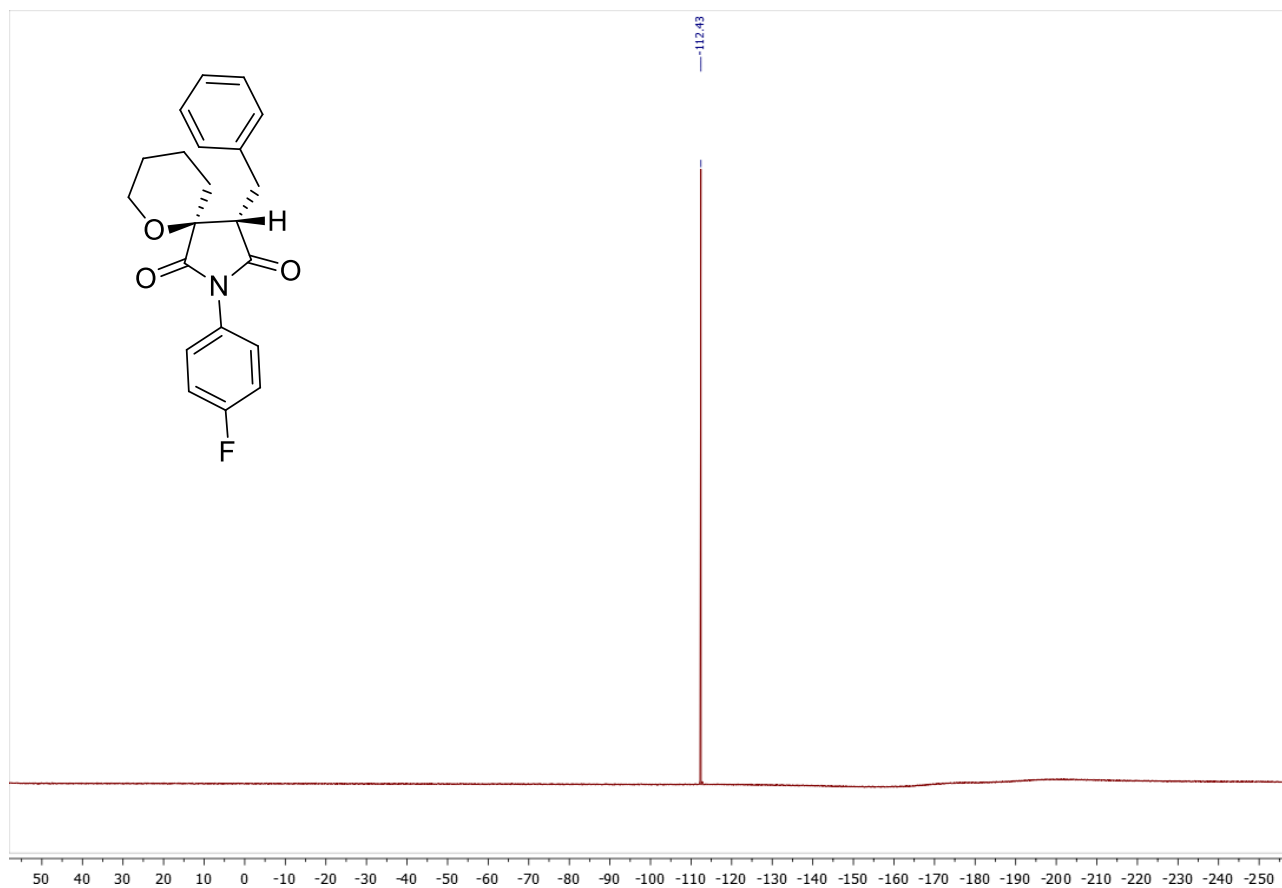
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of **10g**



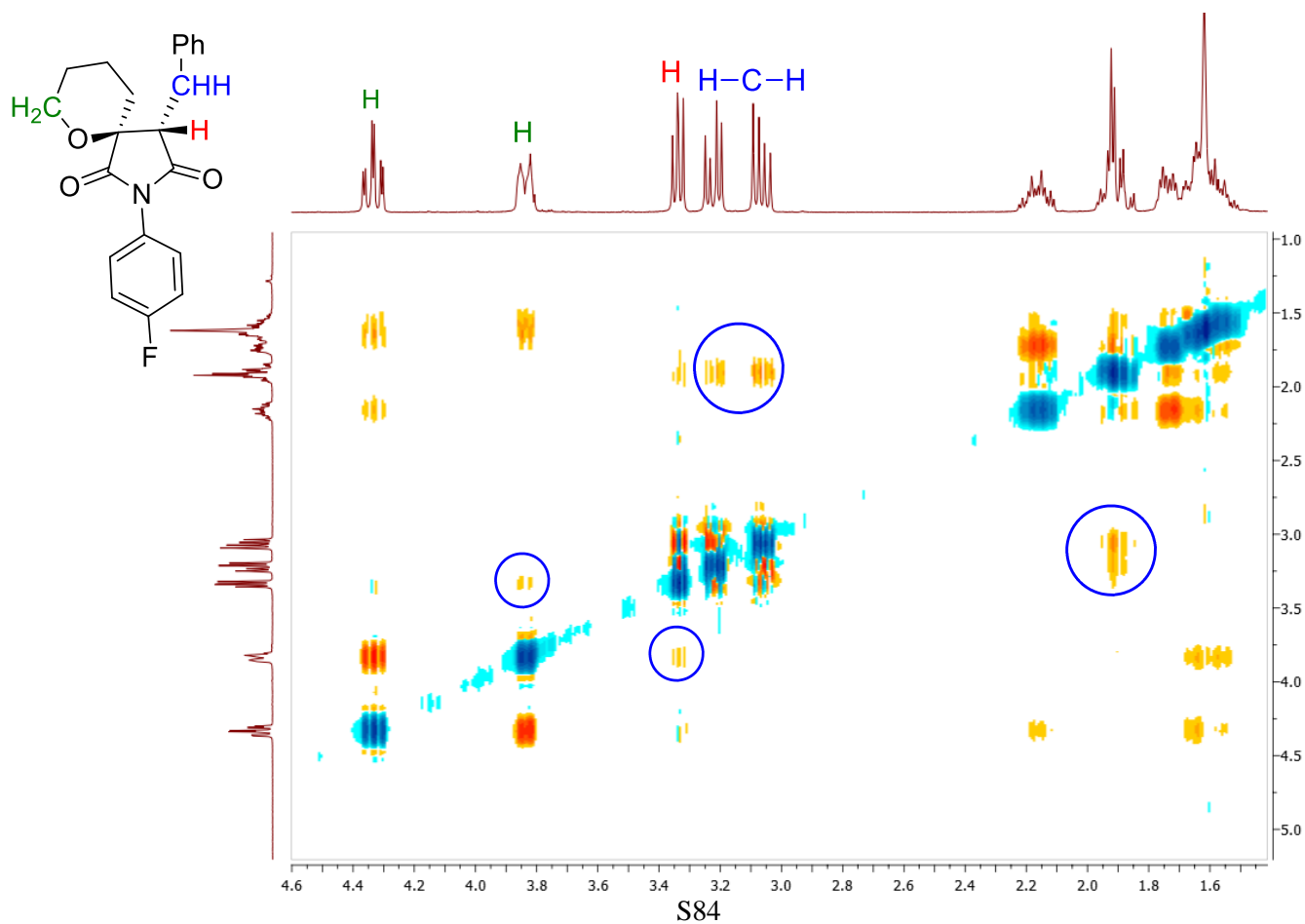
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*R/S,R/S*)-17a



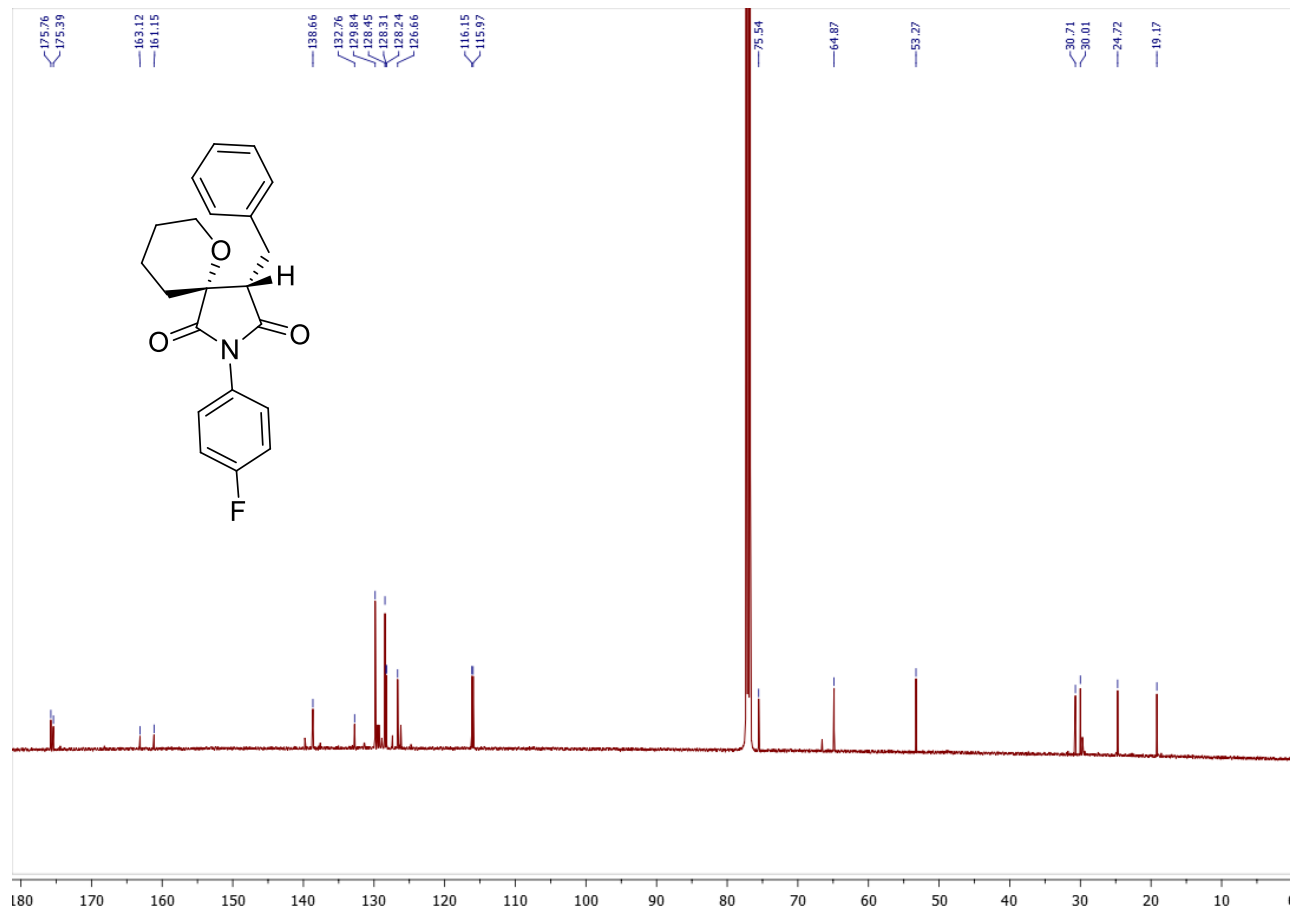
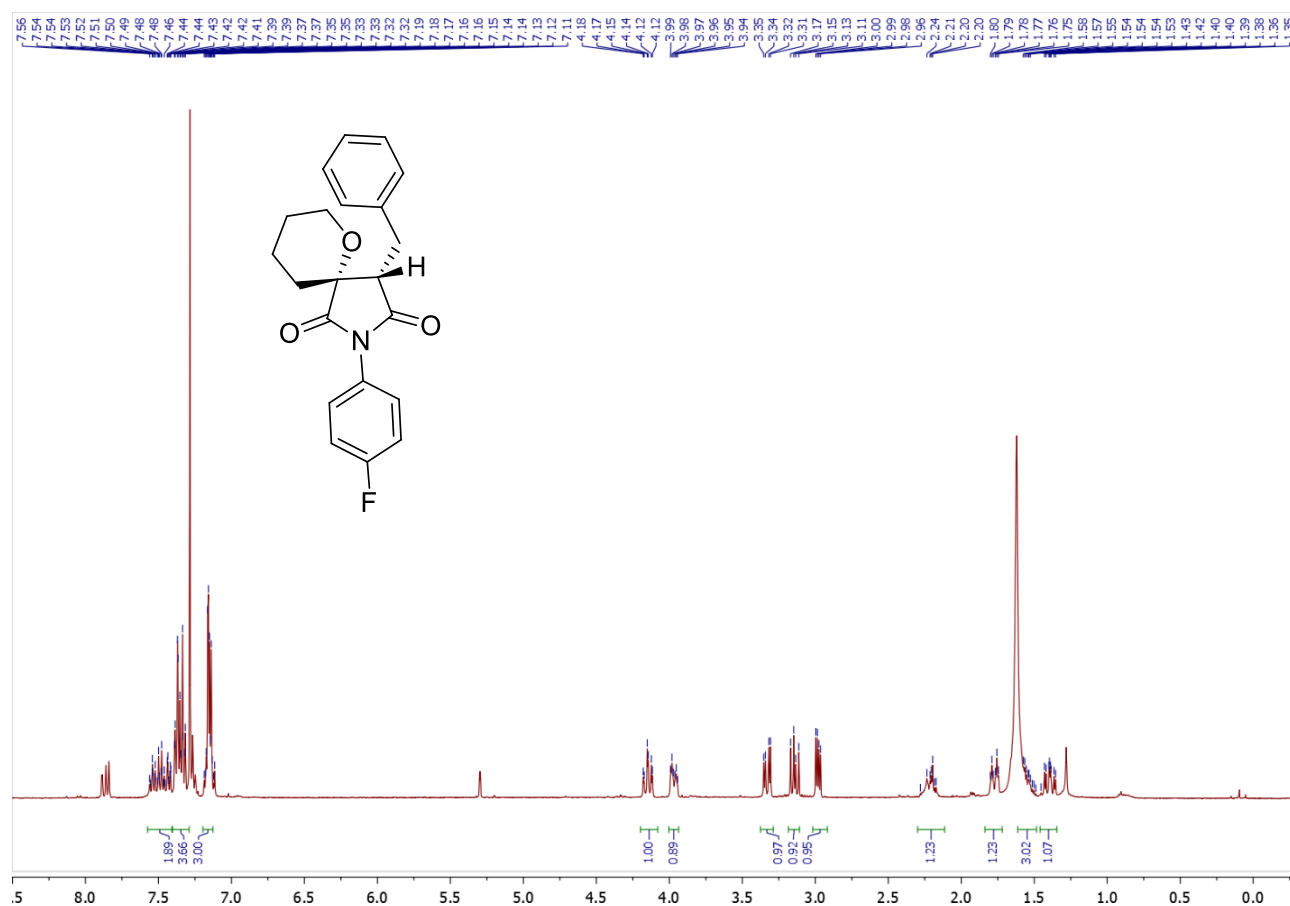
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of (*R/S,R/S*)-17a



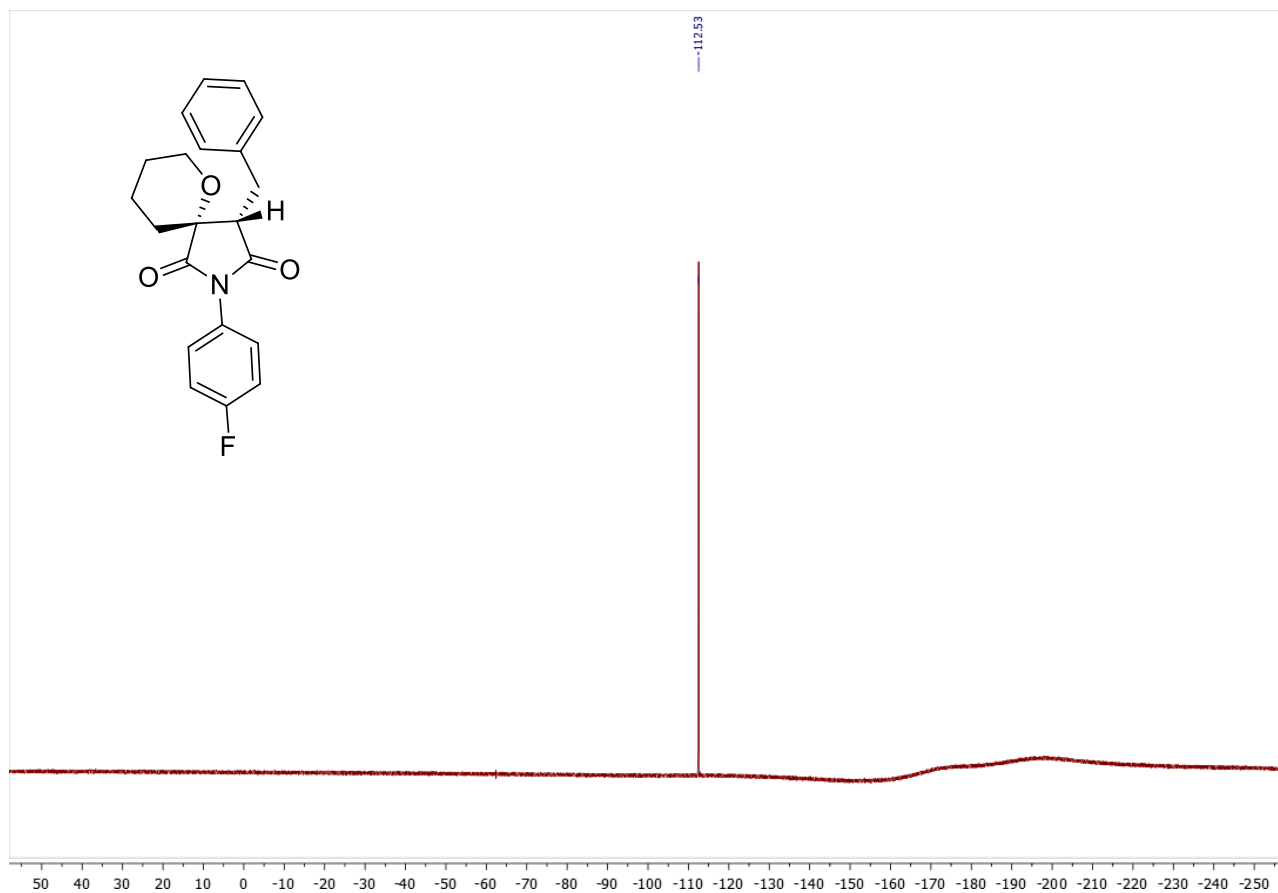
NOESY (400.13 MHz, CDCl_3) spectrum of compound (*R/S,R/S*)-17a



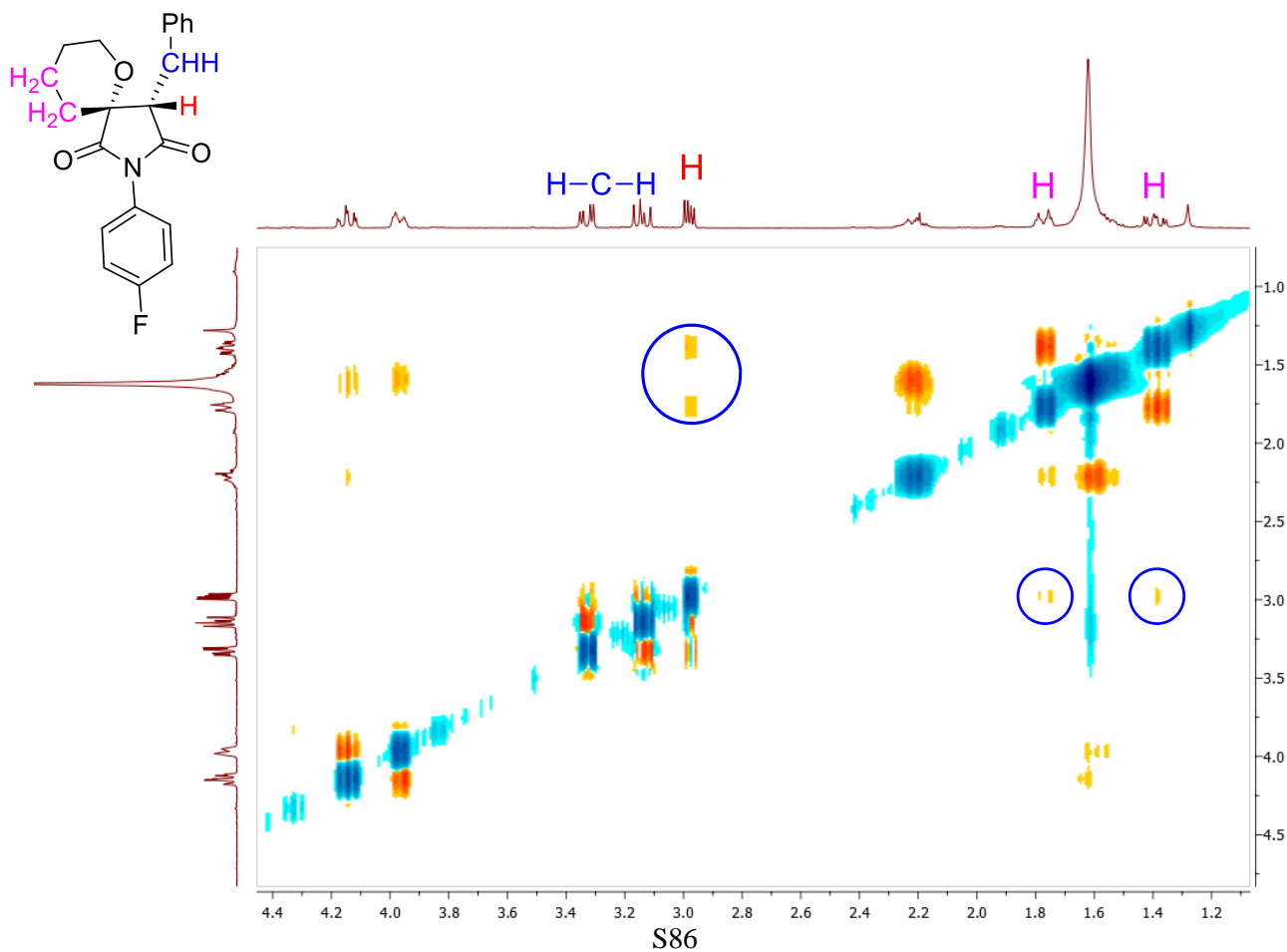
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*R/S,S/R*)-17a



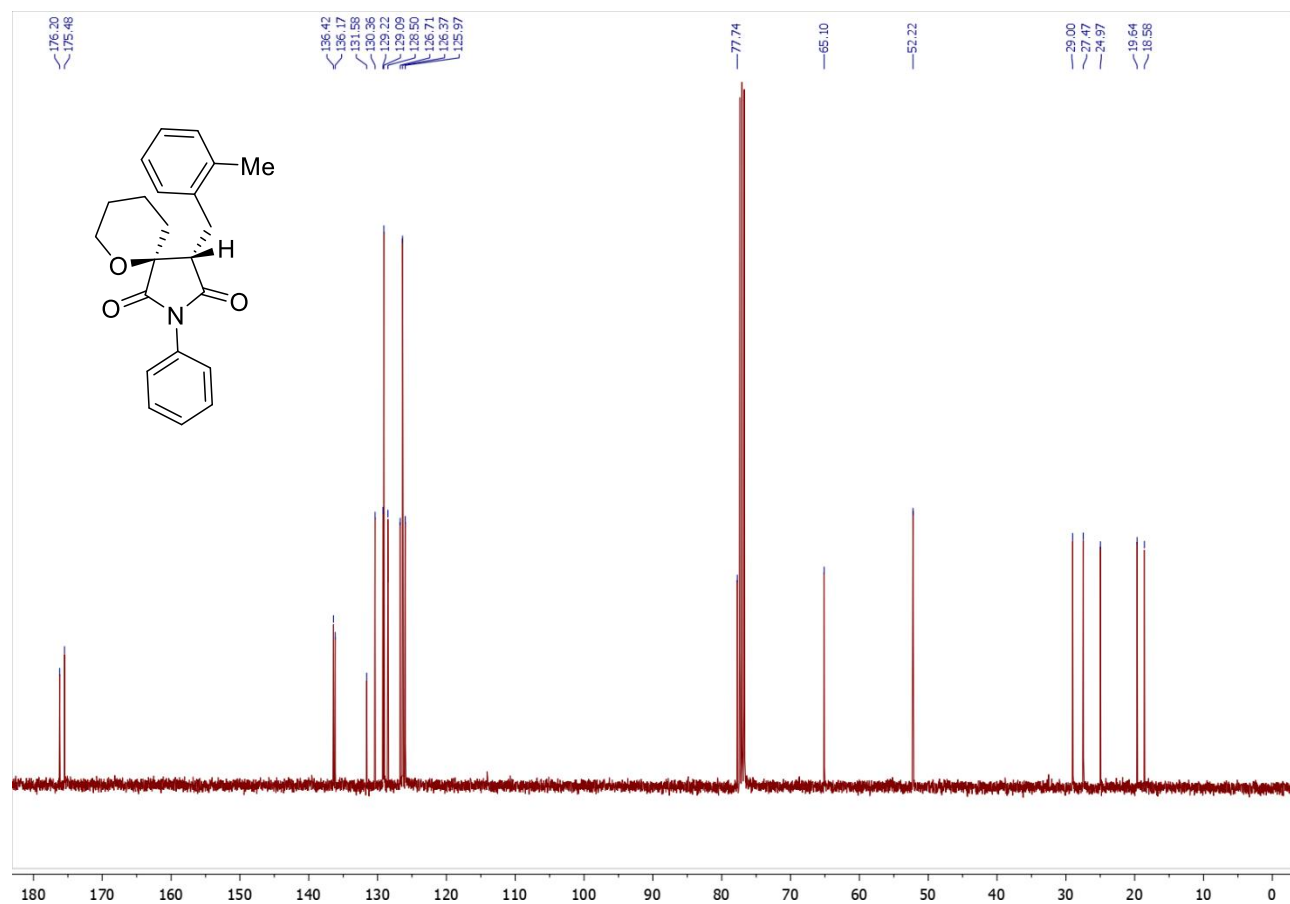
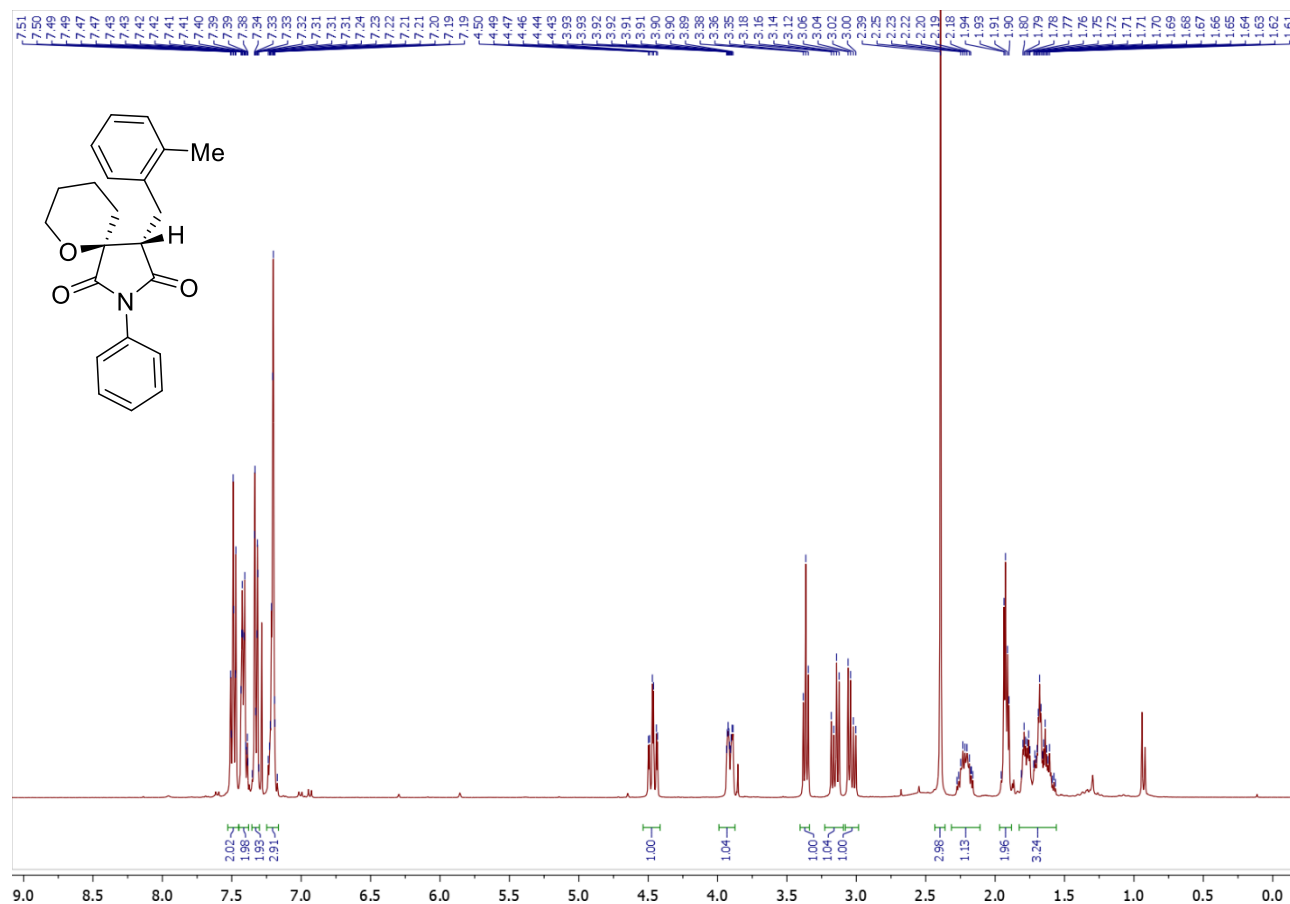
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of (*R/S,S/R*)-17a



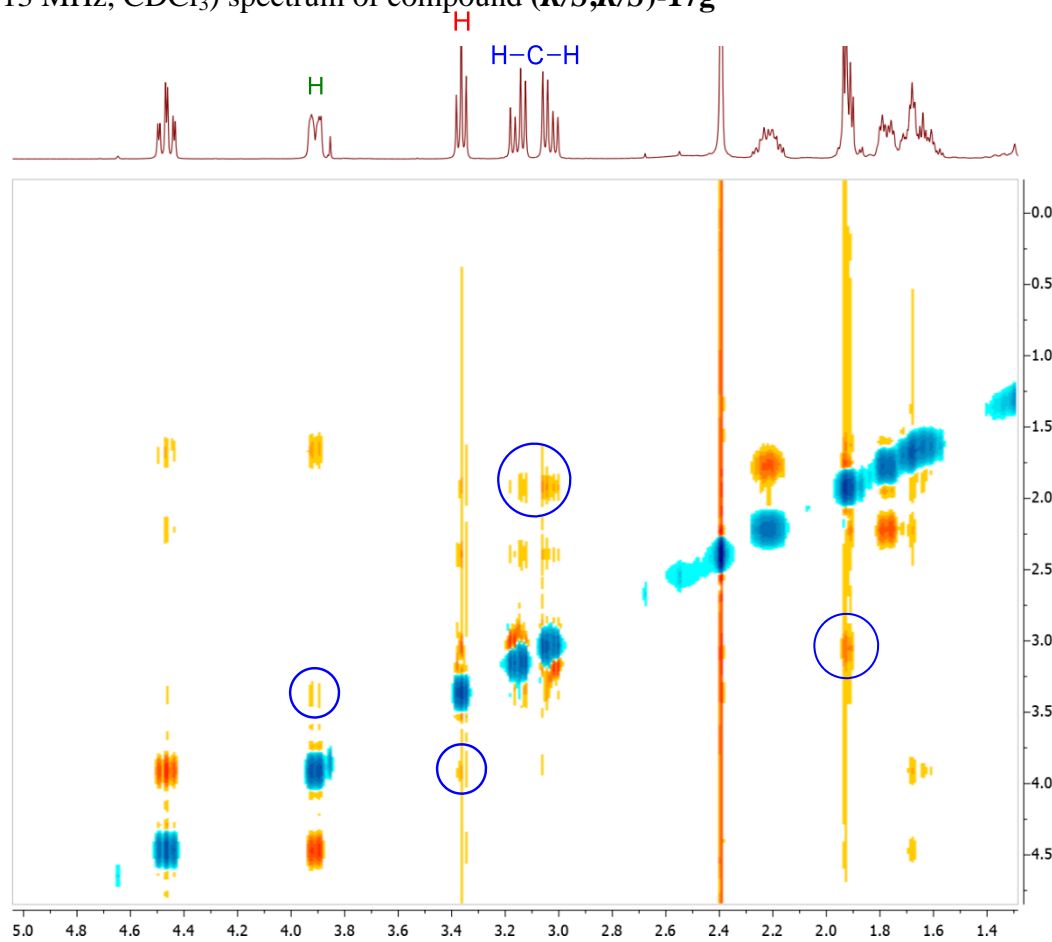
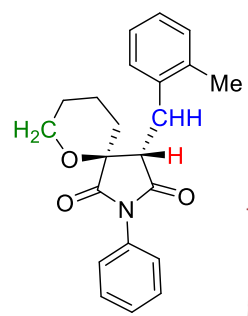
NOESY (400.13 MHz, CDCl_3) spectrum of compound (*R/S,S/R*)-17a



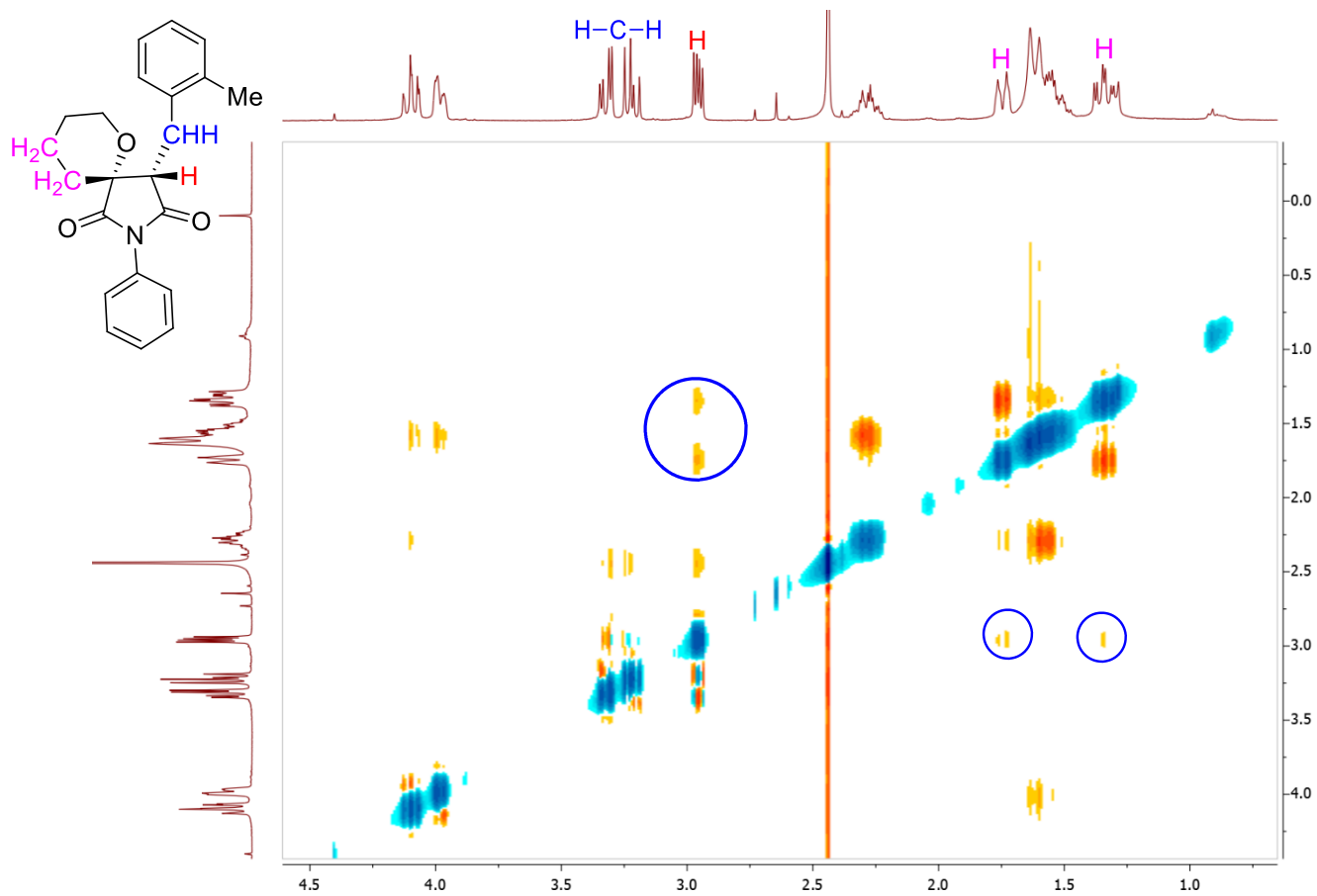
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*R/S,R/S*)-17g



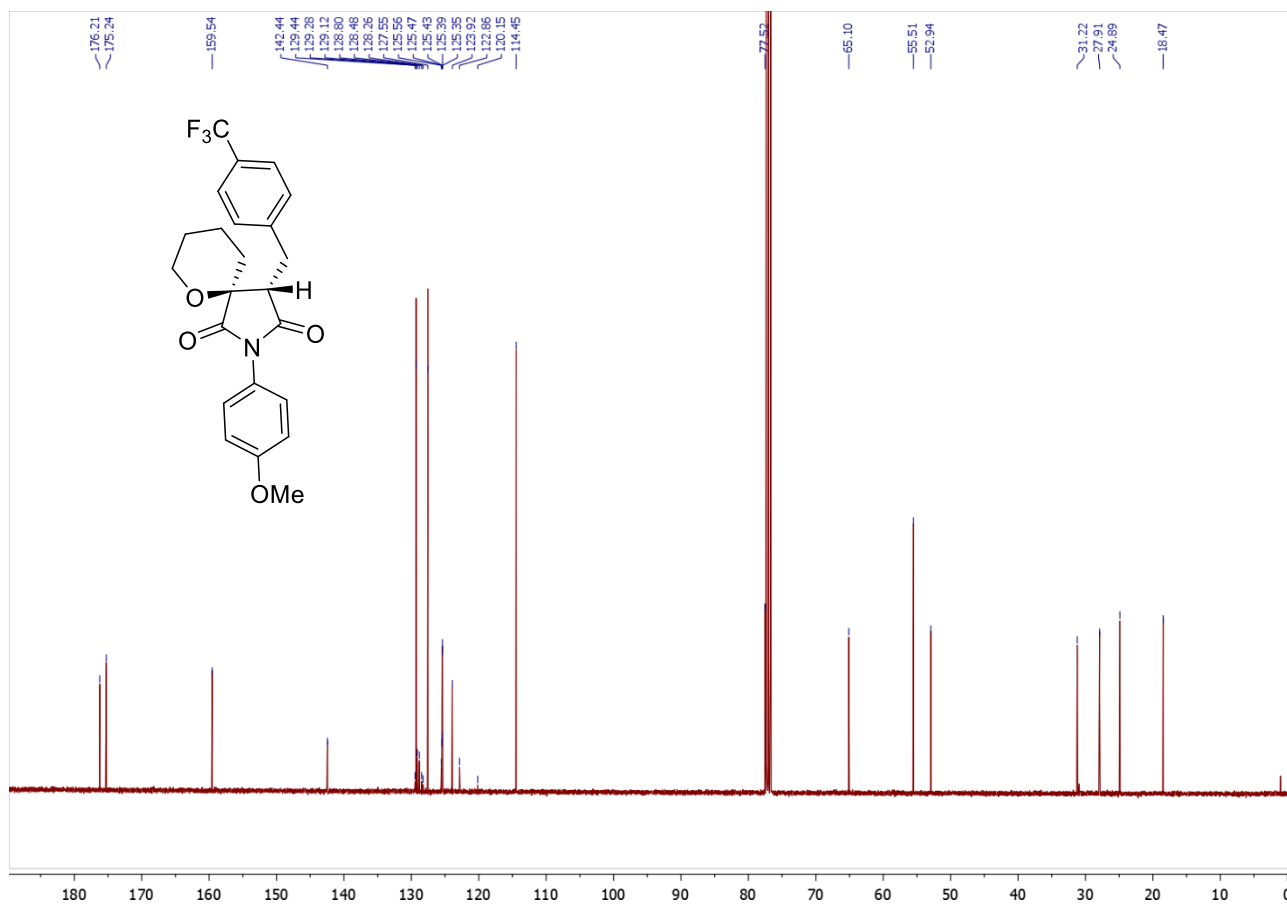
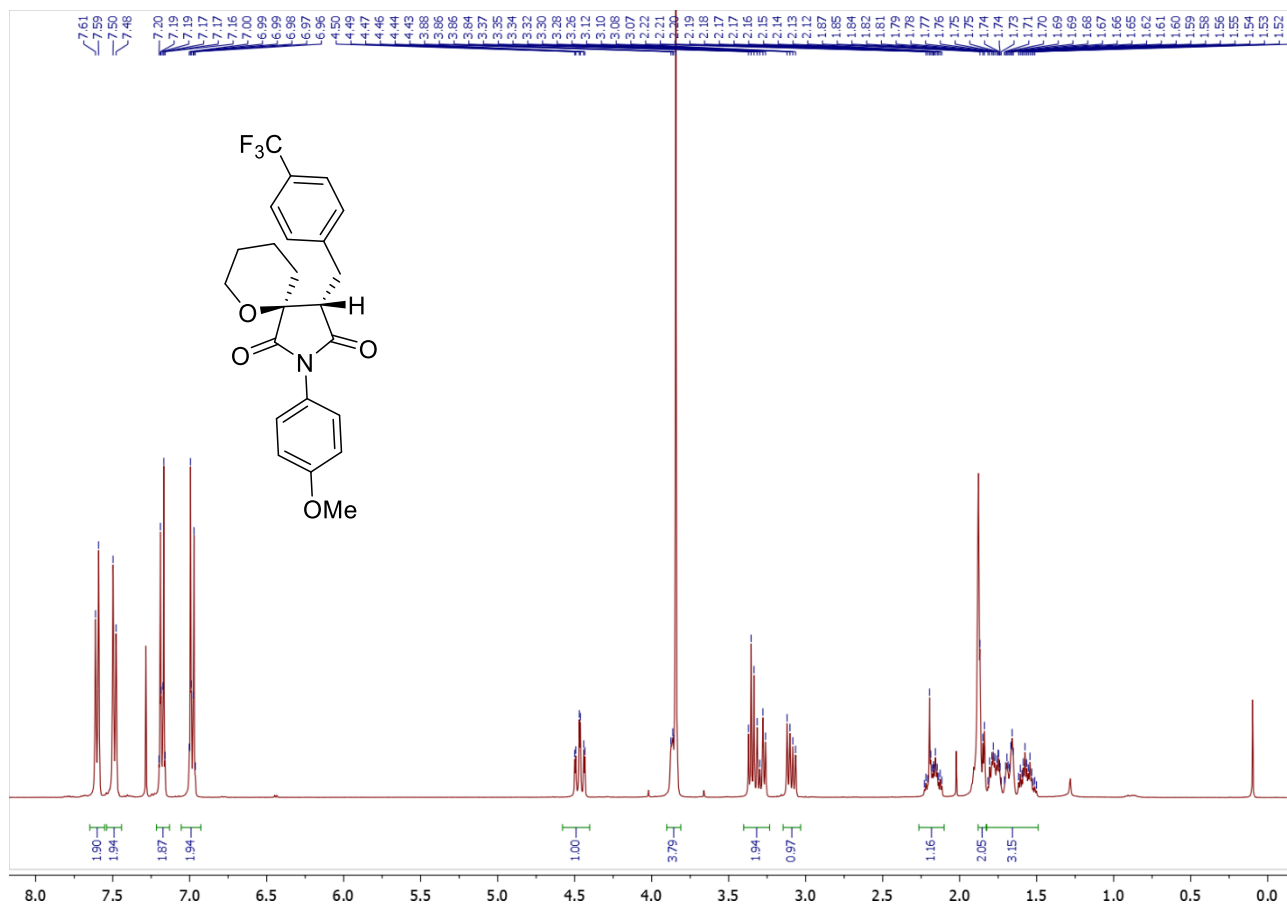
NOESY (400.13 MHz, CDCl₃) spectrum of compound (*R/S,R/S*)-17g



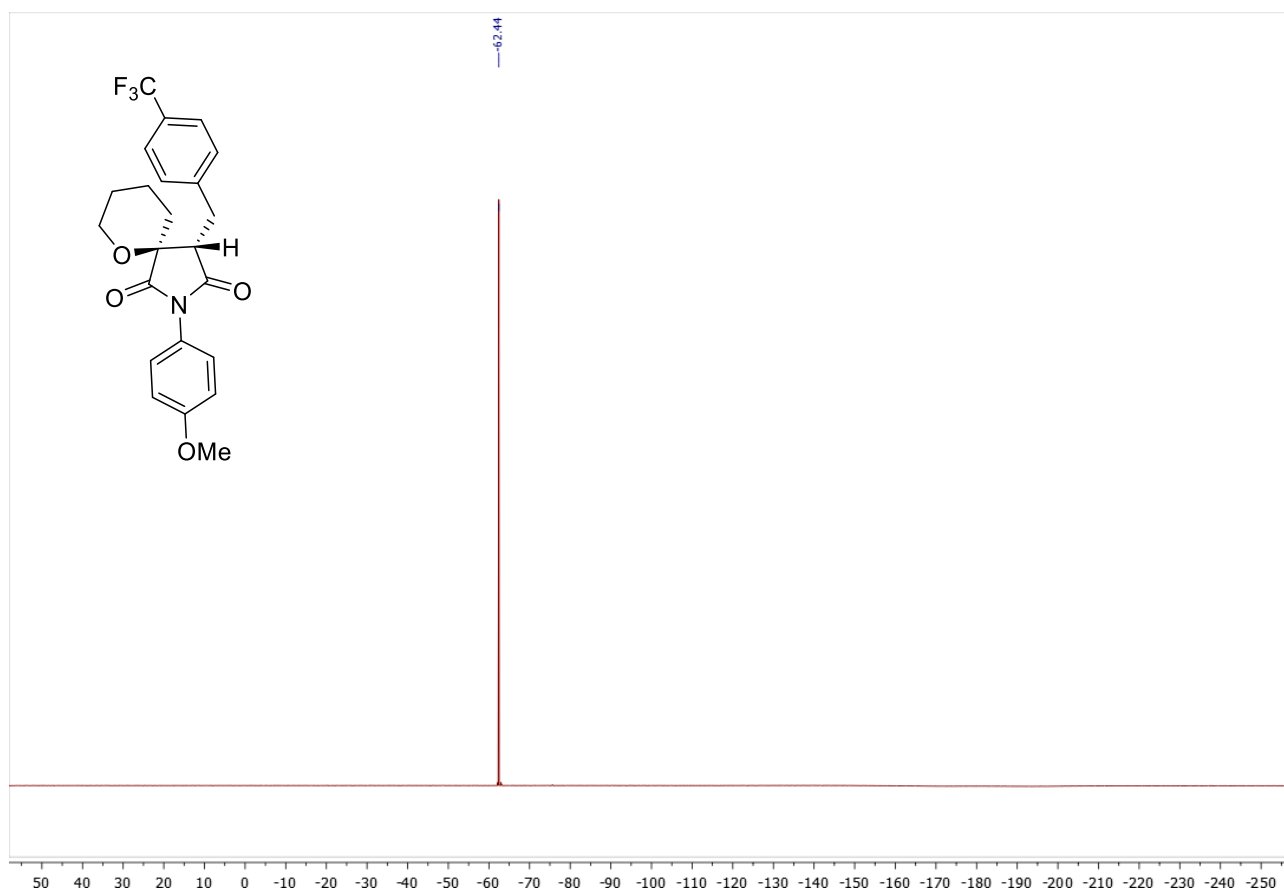
NOESY (400.13 MHz, CDCl₃) spectrum of compound (*R/S,S/R*)-17g



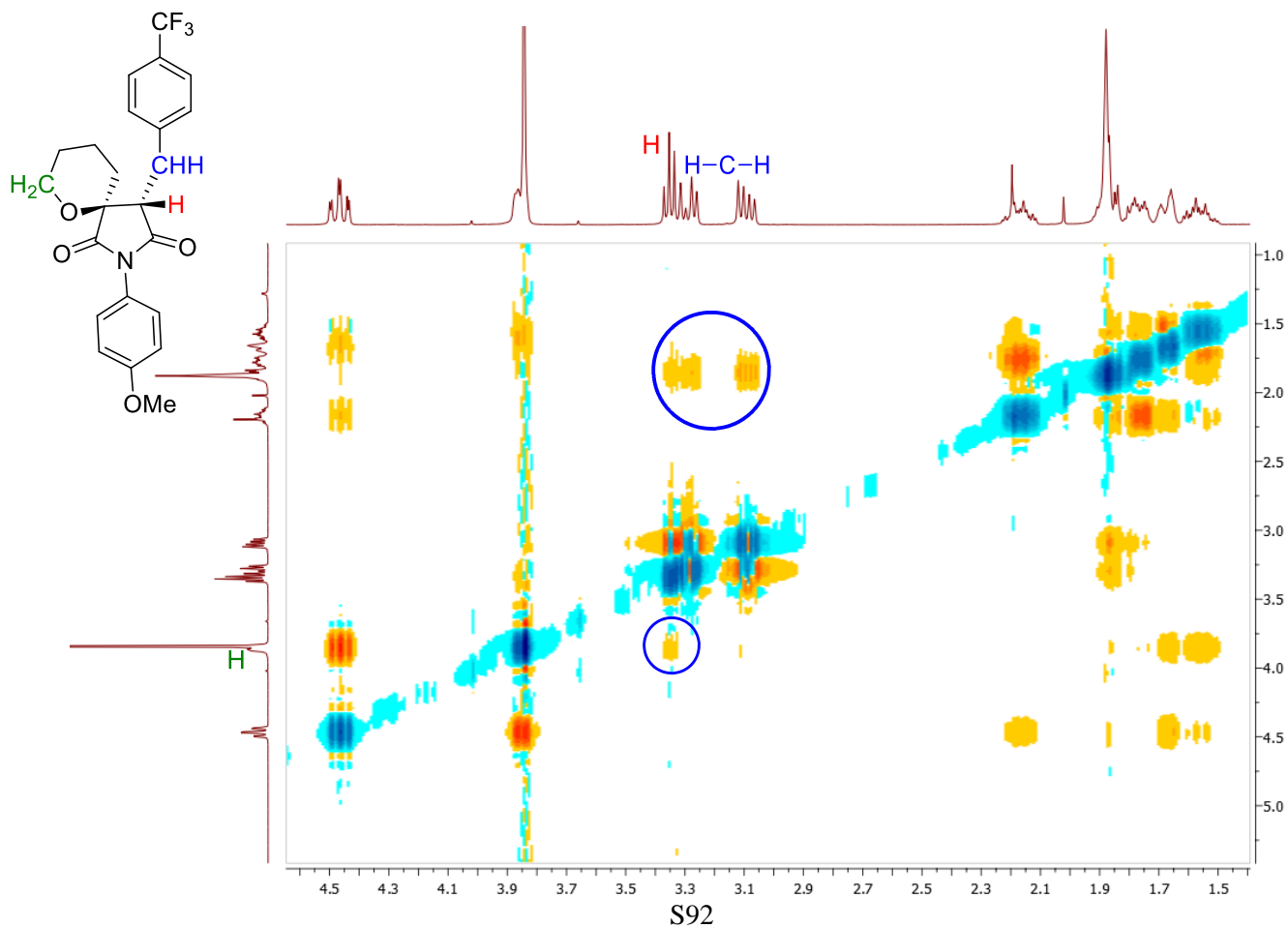
Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*R/S,R/S*)-17x



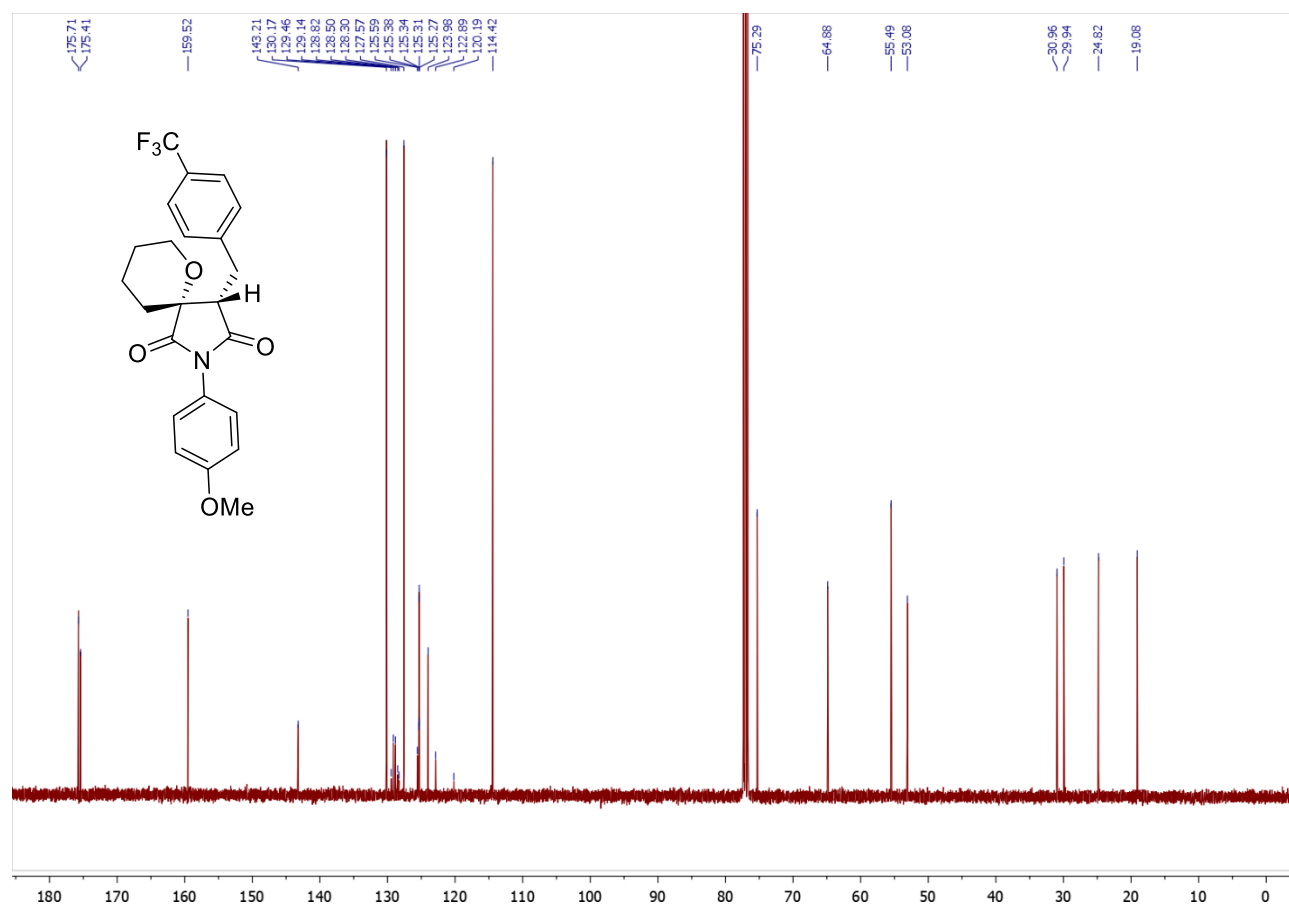
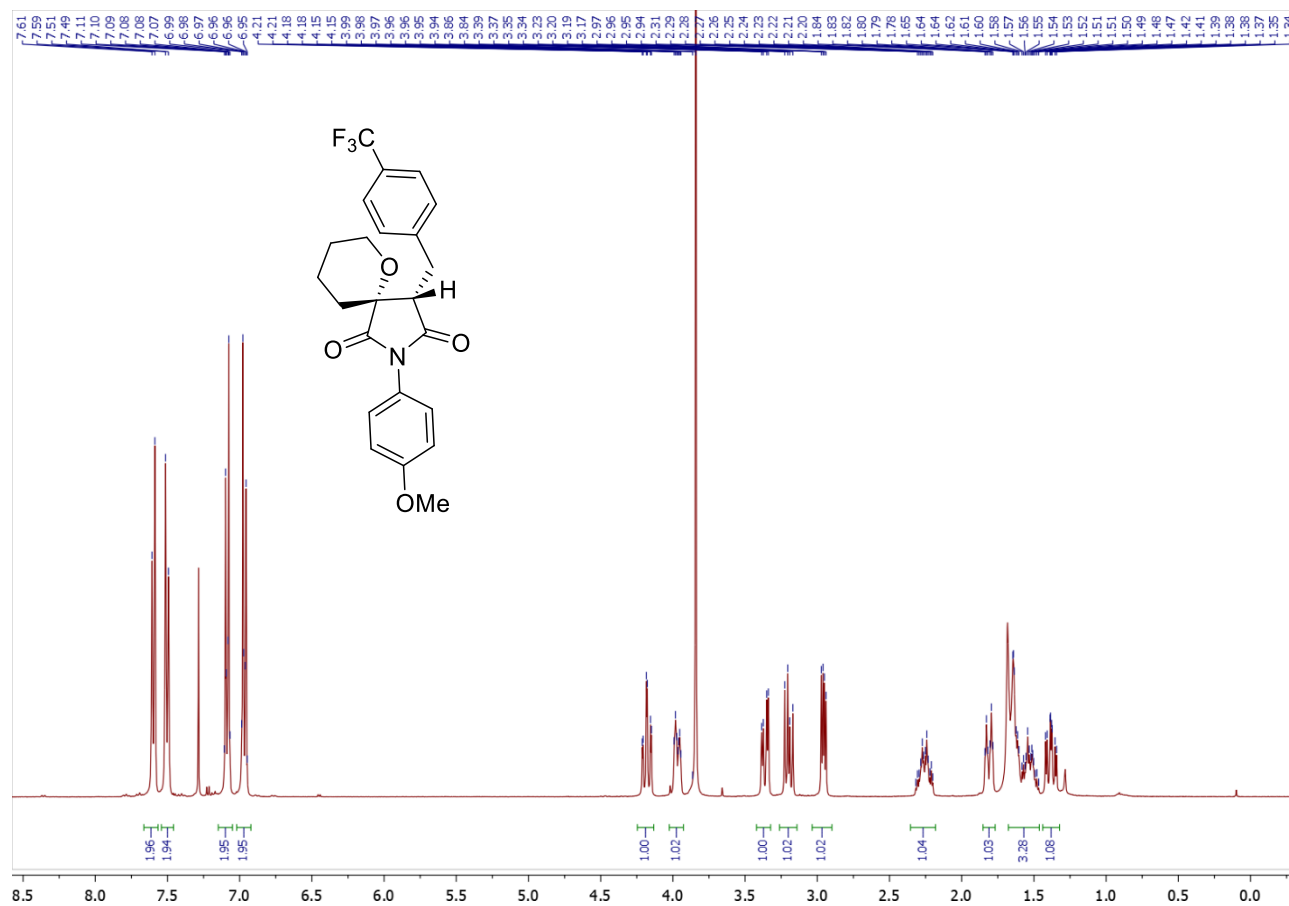
Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of (*R/S,R/S*)-17x



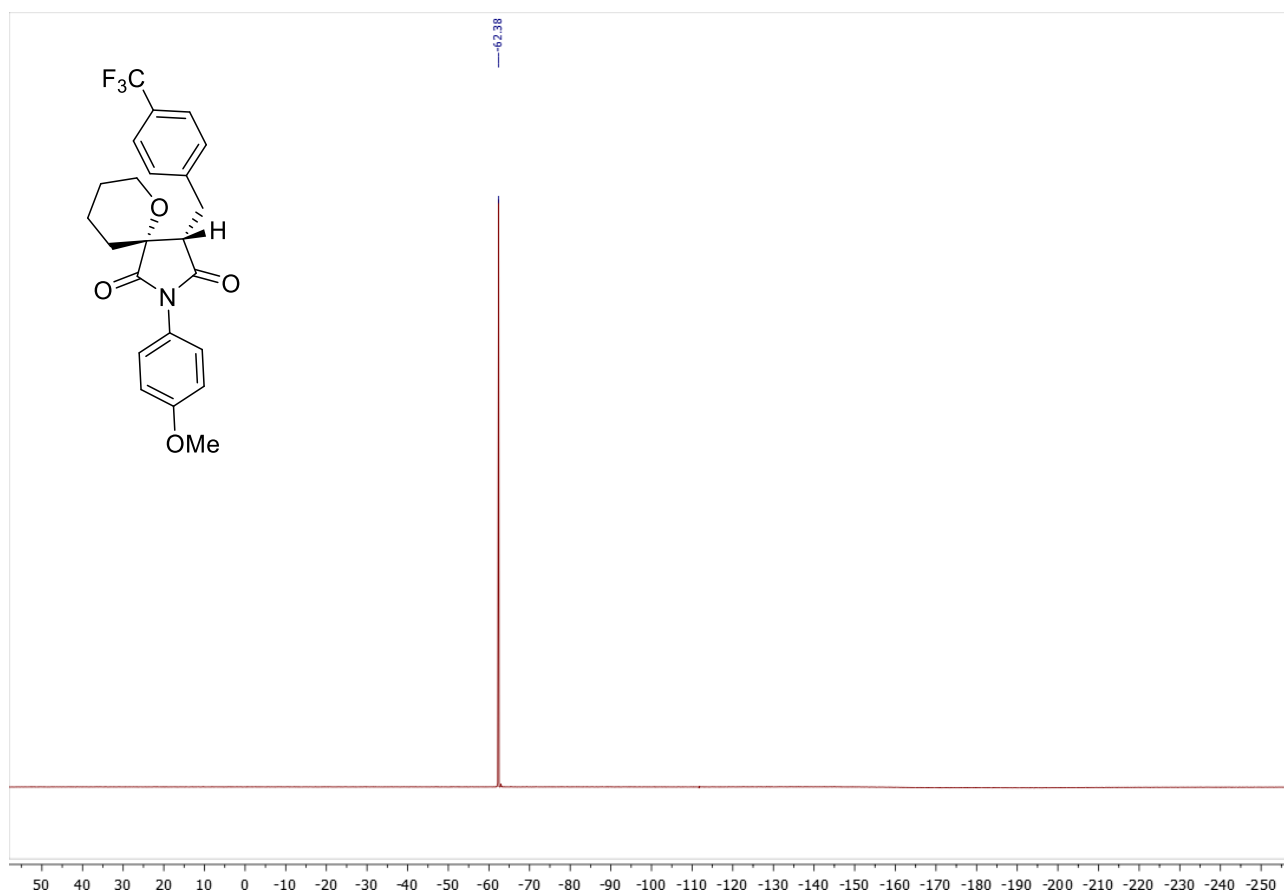
NOESY (400.13 MHz, CDCl_3) spectrum of compound (*R/S,R/S*)-17x



Copies of ^1H (400.13 MHz, CDCl_3) and ^{13}C (100.61 MHz, CDCl_3) spectra of (*R/S,S/R*)-17x



Copy of ^{19}F (376.50 MHz, CDCl_3) spectrum of (*R/S,S/R*)-17x



NOESY (400.13 MHz, CDCl_3) spectrum of compound (*R/S,S/R*)-17x

