Supporting Information

The one pot asymmetric synthesis of 3,3′-pyrrolidonyl spiroxindoles via regio- and stereoselective domino reaction

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Sci-finder substructure on February 28, 2019

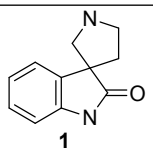
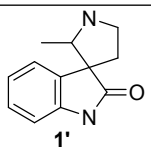
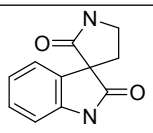
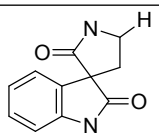
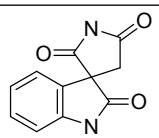
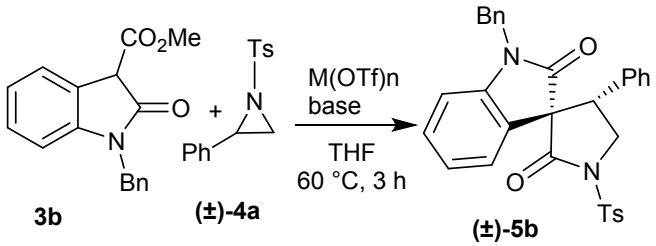
structure	 1	 1'	 2	 2'	 2''
hit	14,700	11,466	434	273	248
hit for Biological study	2972	2488	181	150	150
ref. for all (including patent)	1987	1759	88	38	45
ref. for biological study (including patent)	1188	1104	43	10	34

Table 1 Optimization of the domino reaction with different metal triflates



3b (±)-4a (±)-5b

entry	M(OTf)n	base	dr	yield of **(±)-5b** (%)
1	$In(OTf)_3$	K^{t}OBu	>99:1	42
2	$Y(OTf)_3$	K^{t}OBu	>99:1	27
3	$Fe(OTf)_3$	K^{t}OBu	>99:1	32
4	$Mg(OTf)_2$	K^{t}OBu	>99:1	35
5	$Zn(OTf)_2$	K^{t}OBu	>99:1	30

X-ray crystal structure details:

Datablock for the compound 5b

The details of crystal data collection and refinement of **5b** are summarized in Table S1. CCDC 1898778 contains the supplementary crystallographic data for this paper. This data can be obtained free of charge from The Cambridge Crystallography Data Center via www.ccdc.cam.ac.uk/data_request/cif.

Datablock: gp_67_0m

Bond precision: C-C = 0.0068 A Wavelength=0.71073

Cell: a=12.1351(6) b=8.4982(4) c=26.9828(18)
alpha=90 beta=95.024(4) gamma=90

Temperature: 293 K

	Calculated	Reported
Volume	2772.0(3)	2772.0(3)
Space group	I 2	I2
Hall group	I 2y	?
Moiety formula	C31 H26 N2 O4 S	C31 H26 N2 O4 S
Sum formula	C31 H26 N2 O4 S	C31 H26 N2 O4 S
Mr	522.60	522.60
Dx,g cm-3	1.252	1.252
Z	4	4
Mu (mm-1)	0.155	0.155
F000	1096.0	1096.0
F000'	1096.97	
h,k,lmax	14,10,31	14,10,31
Nref	4790[2572]	3782
Tmin,Tmax	0.969,0.985	
Tmin'	0.969	

Correction method= Not given

Data completeness= 1.47/0.79 Theta(max)= 24.810

R(reflections)= 0.0528(2620) wR2(reflections)= 0.1230(3782)

S = 0.944 Npar= 346

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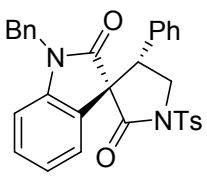
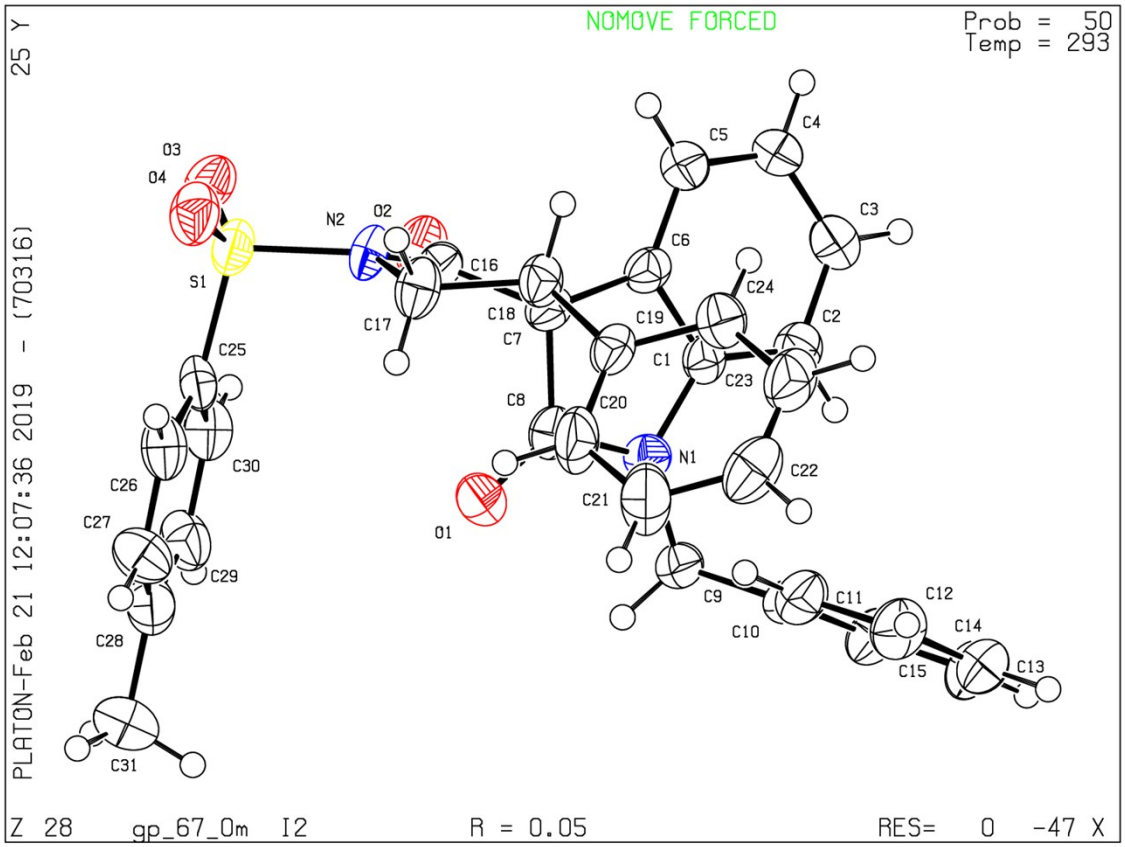
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.



(3*R*,4'*S*)-1-benzyl-4'-phenyl-1'-tosylspiro[indoline-3,3'-pyrrolidine]-2,2'-dione

ORTEP diagram of 5b:



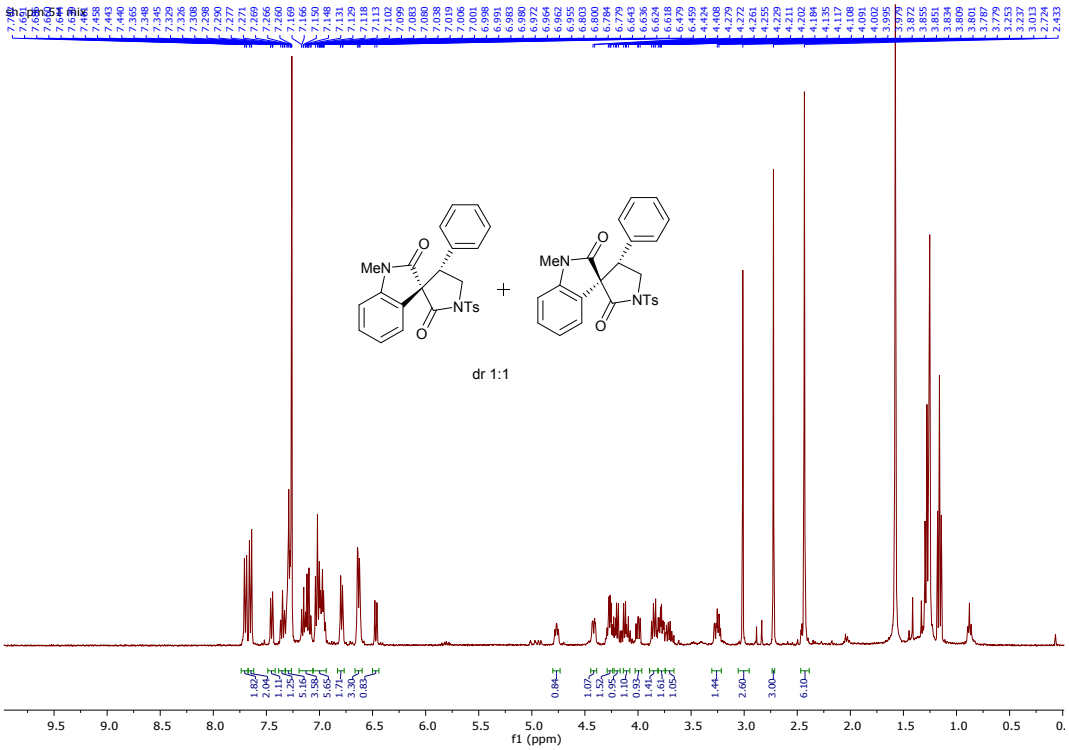
Reference

(1) L. J. Farrugia, *J. Appl. Crystallogr.* 1999, **32**, 837.

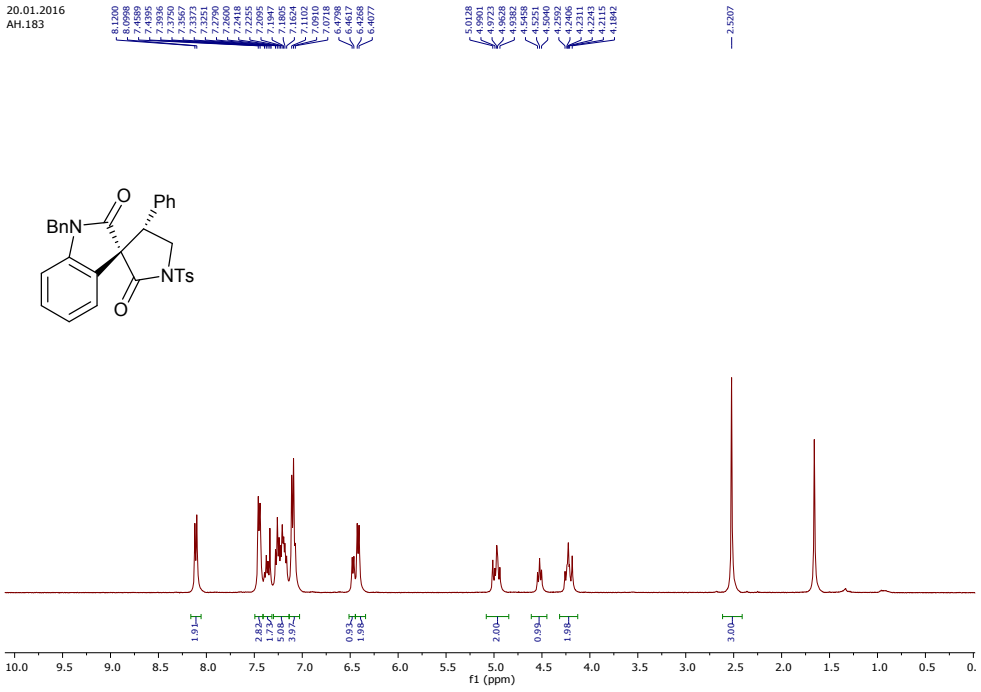
(2) G. M. Sheldrick, *Acta Crystallogr.* 2008, **C71**, 112

(3) L. J. Farrugia, *J. Appl. Crystallogr.* 1997, **30**, 565.

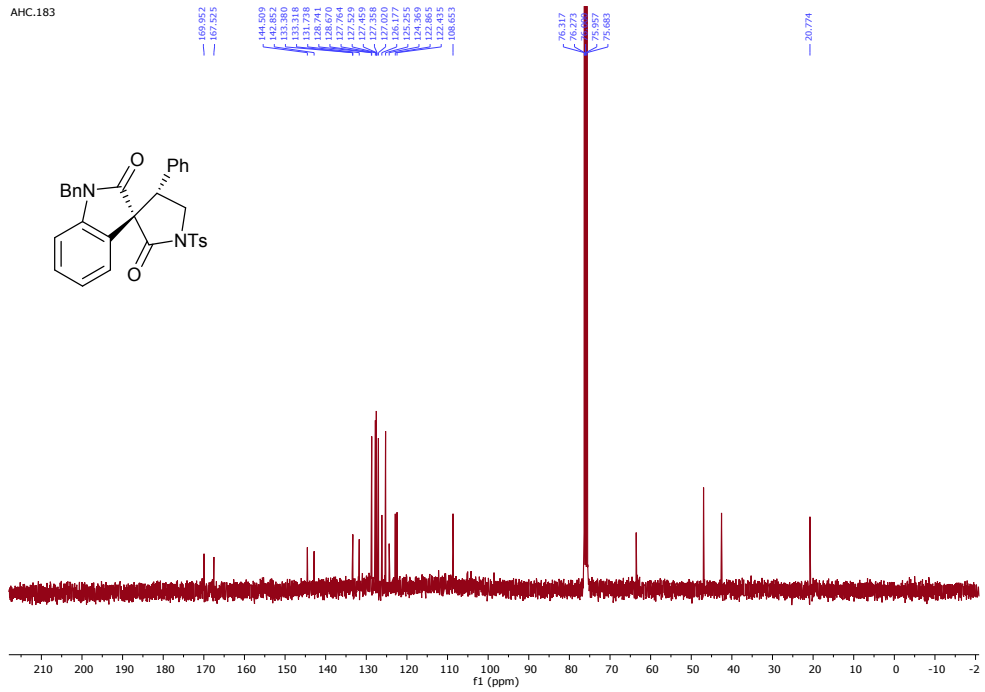
^{1}H NMR and ^{13}C NMR Spectra of Compounds



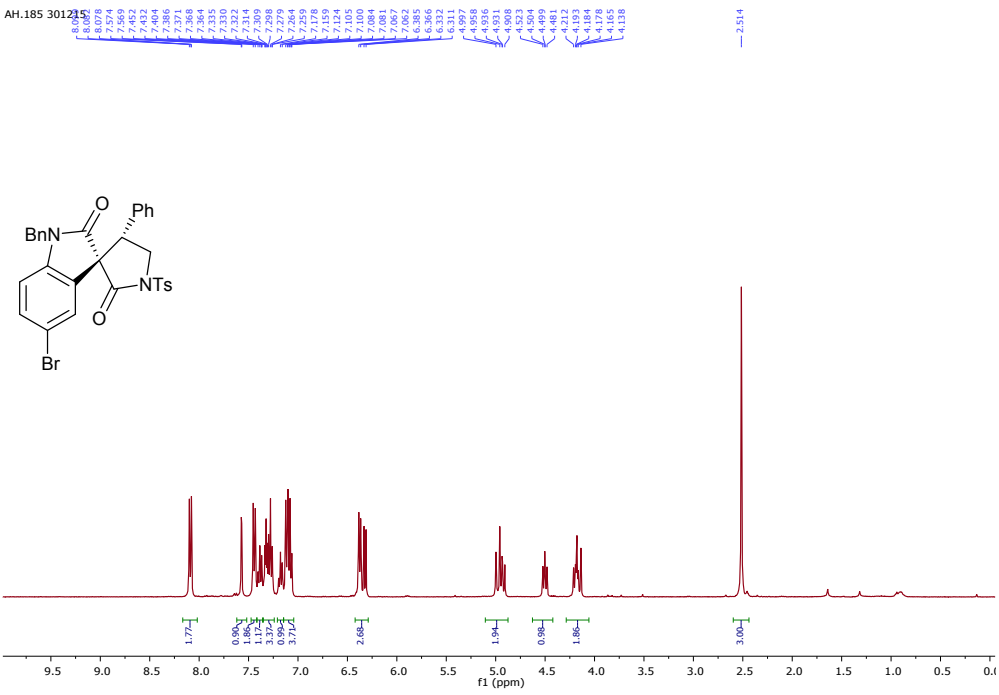
^{1}H NMR Spectrum of Compound **5a**(crude reaction) (400 MHz, $CDCl_3$)



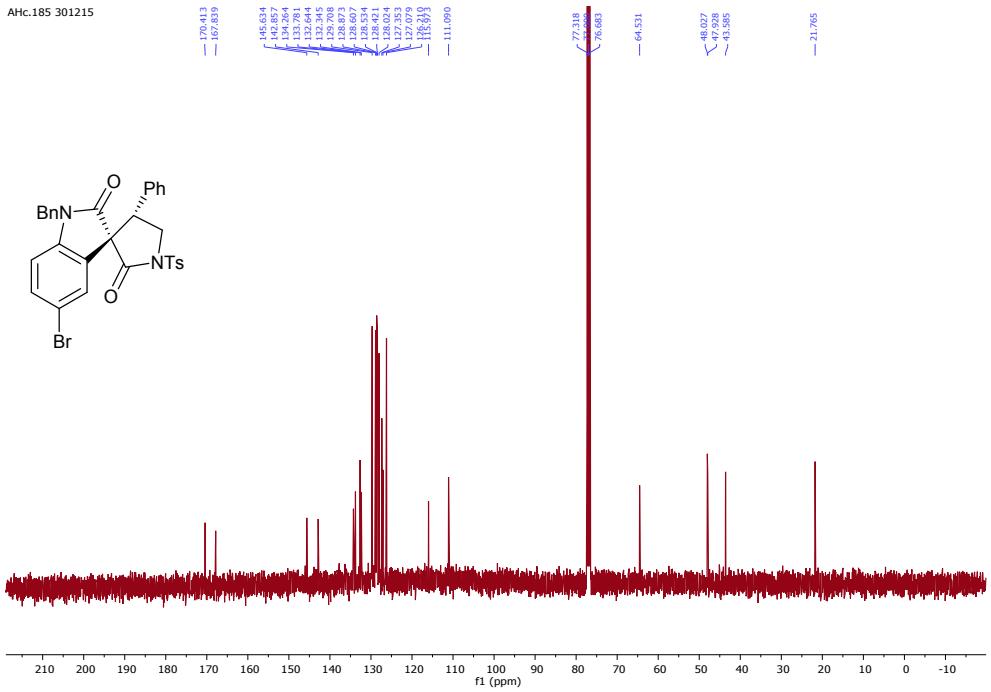
^{1}H NMR Spectrum of Compound **5b** (400 MHz, $CDCl_3$)



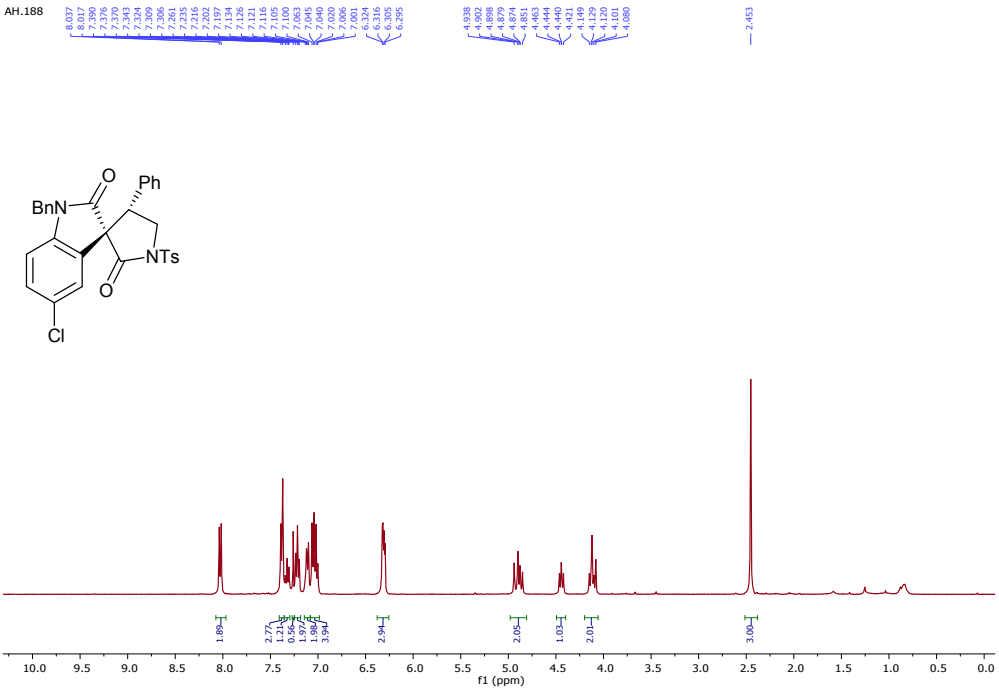
^{13}C NMR Spectrum of Compound **5b** (100 MHz, $CDCl_3$)



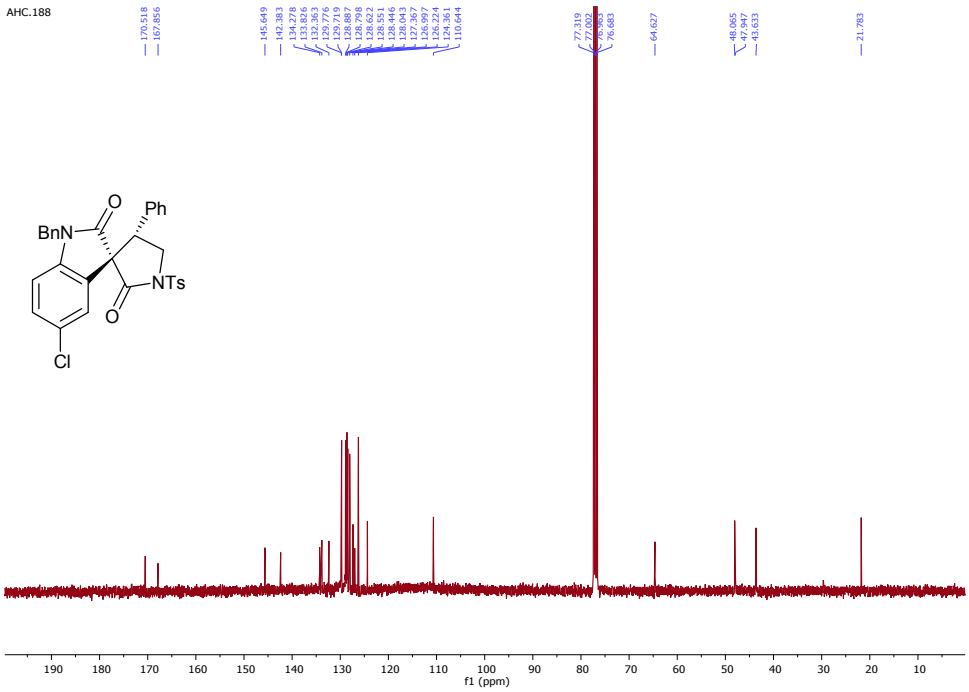
^{1}H NMR Spectrum of Compound **5d** (400 MHz, $CDCl_3$)



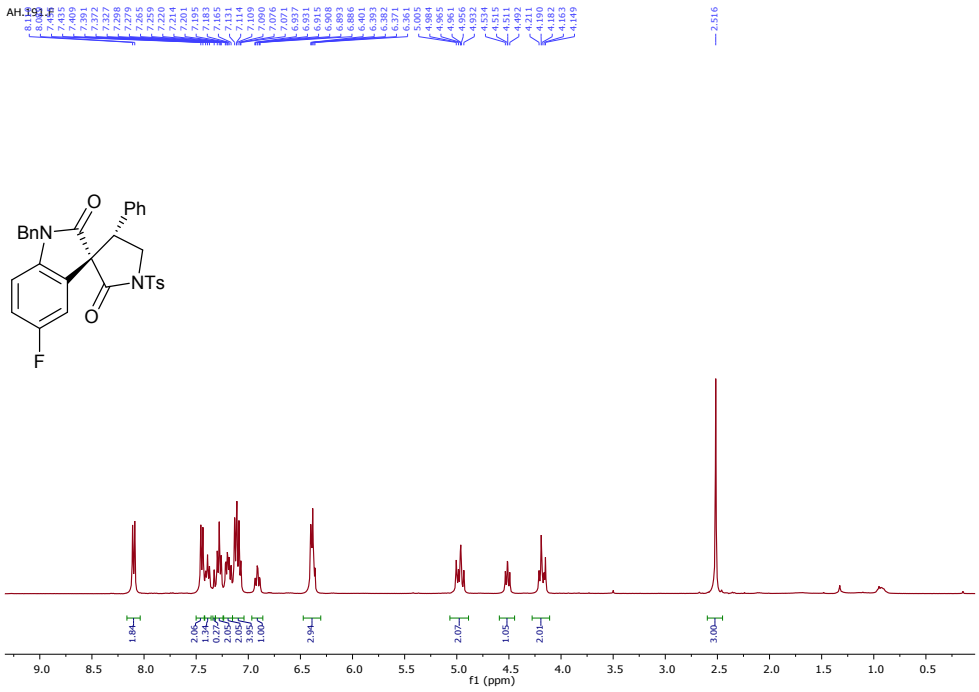
^{13}C NMR Spectrum of Compound **5d** (100 MHz, $CDCl_3$)



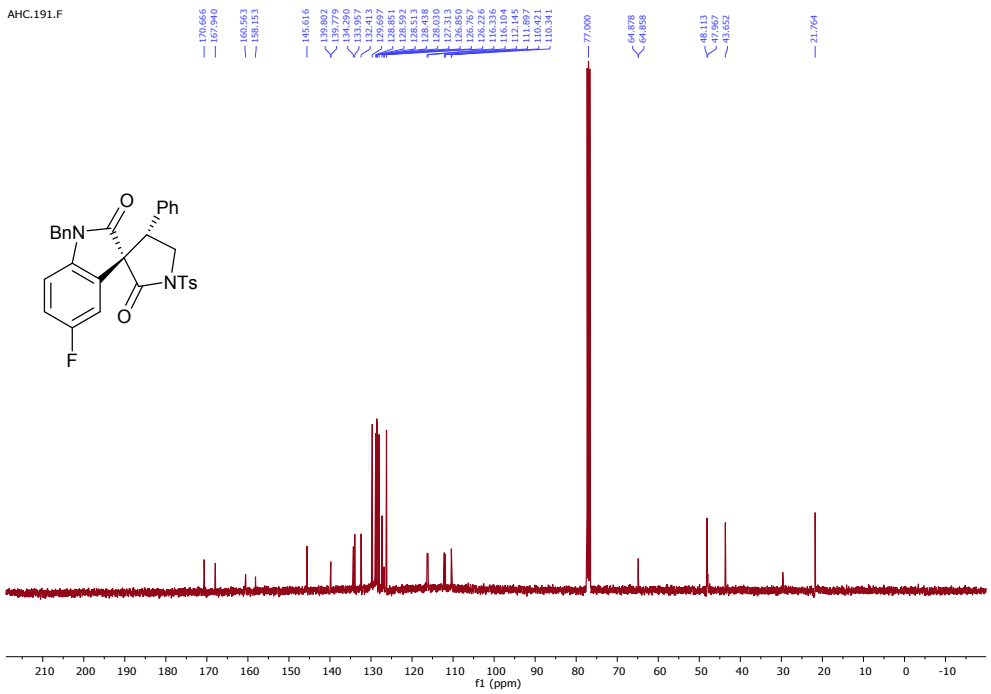
^{1}H NMR Spectrum of Compound **5e** (400 MHz, $CDCl_3$)



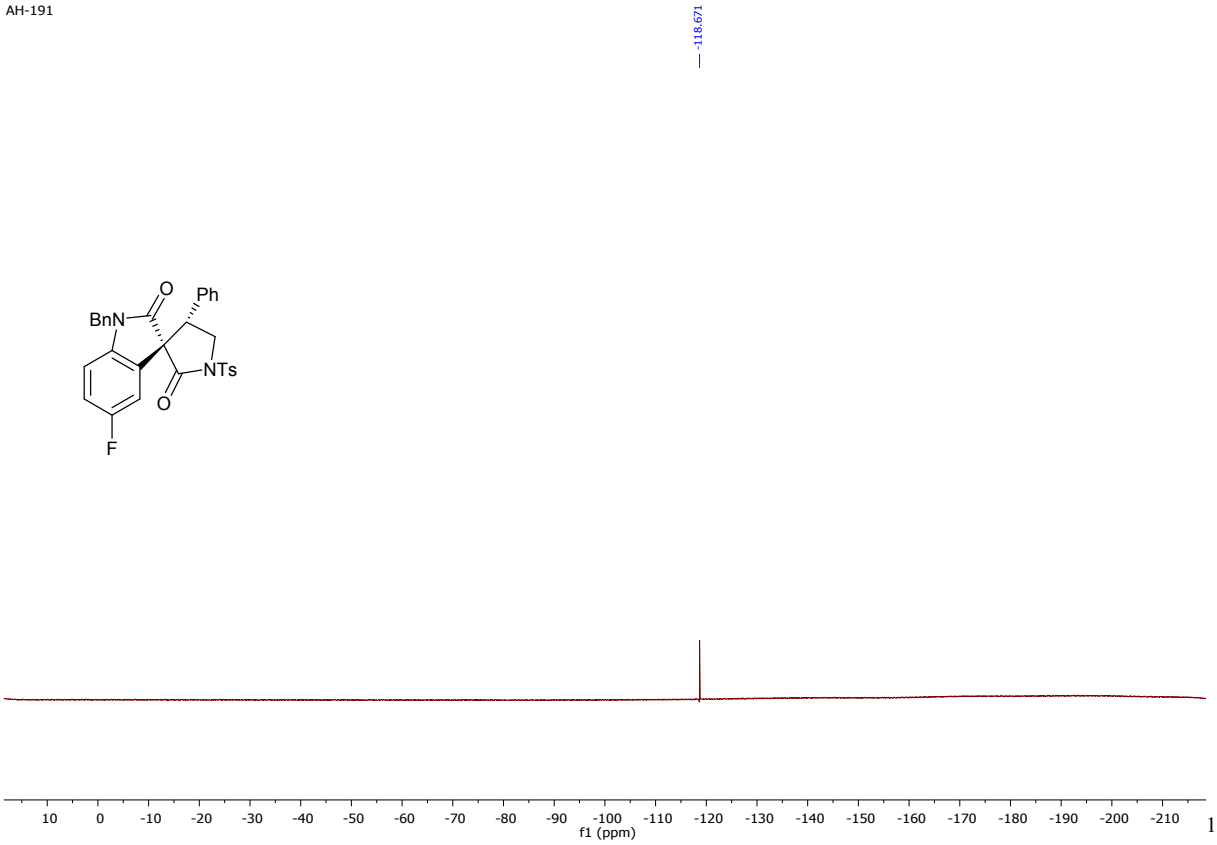
^{13}C NMR Spectrum of Compound **5e** (100 MHz, $CDCl_3$)



^{1}H NMR Spectrum of Compound **5f** (400 MHz, $CDCl_3$)



^{13}C NMR Spectrum of Compound **5f** (100 MHz, $CDCl_3$)



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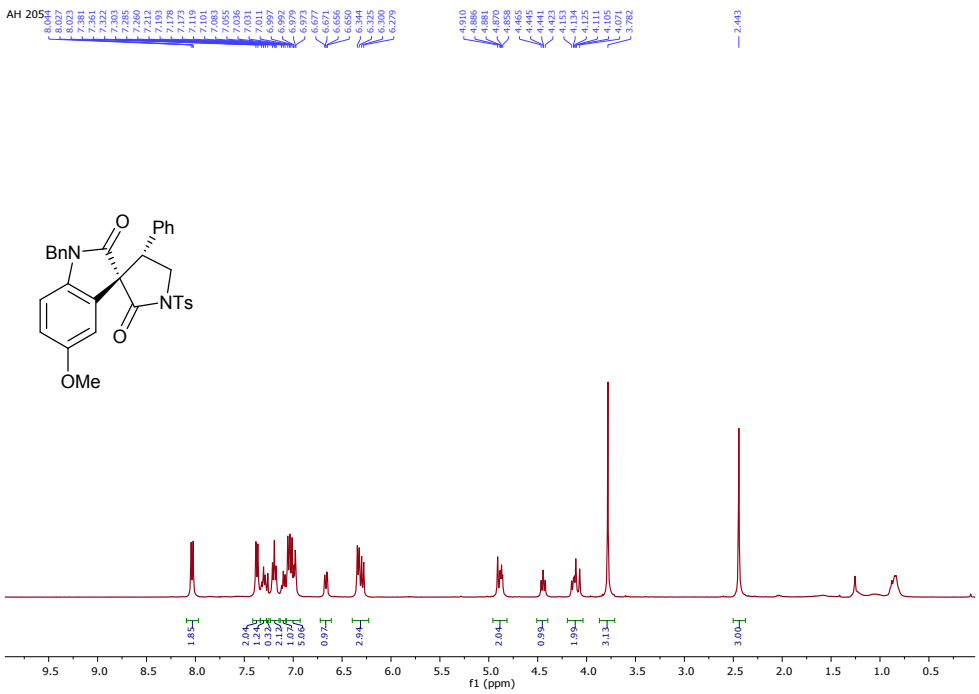
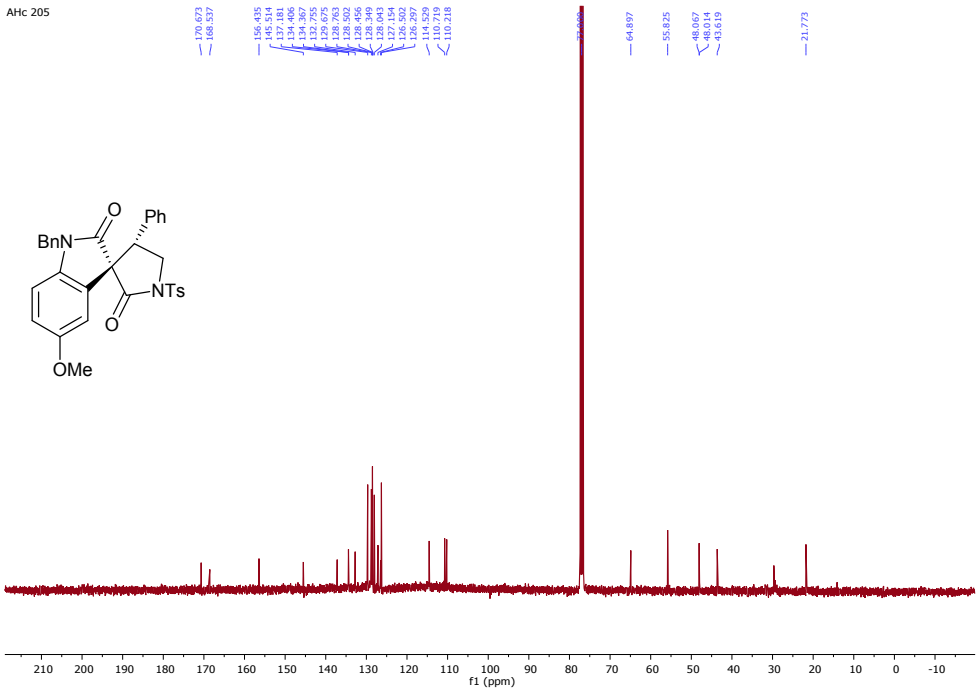
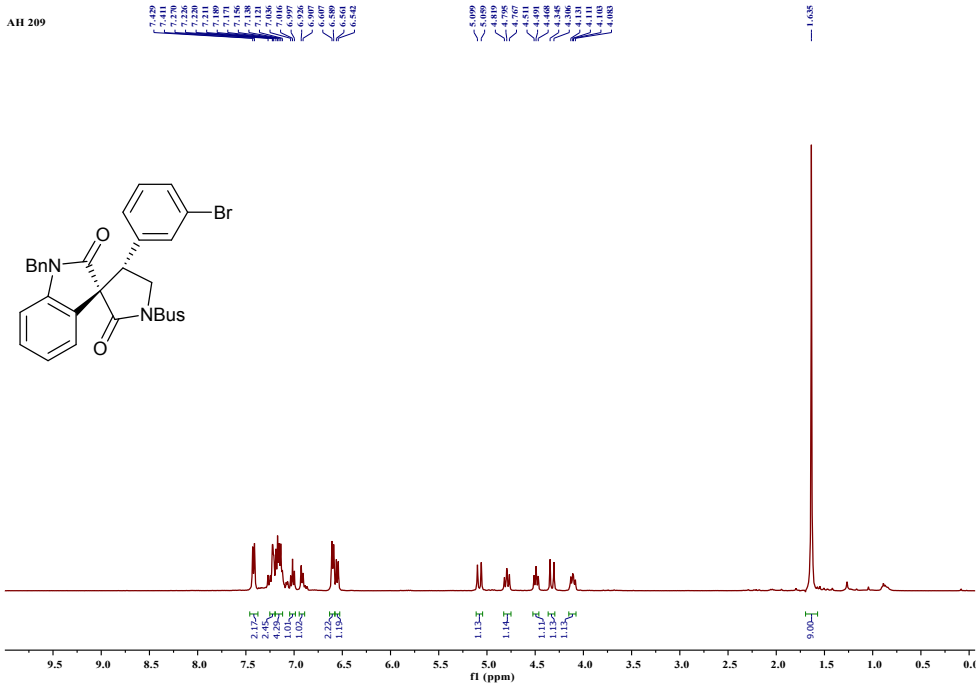
F NMR Spectrum of Compound **5f** (376 MHz, $CDCl_3$)



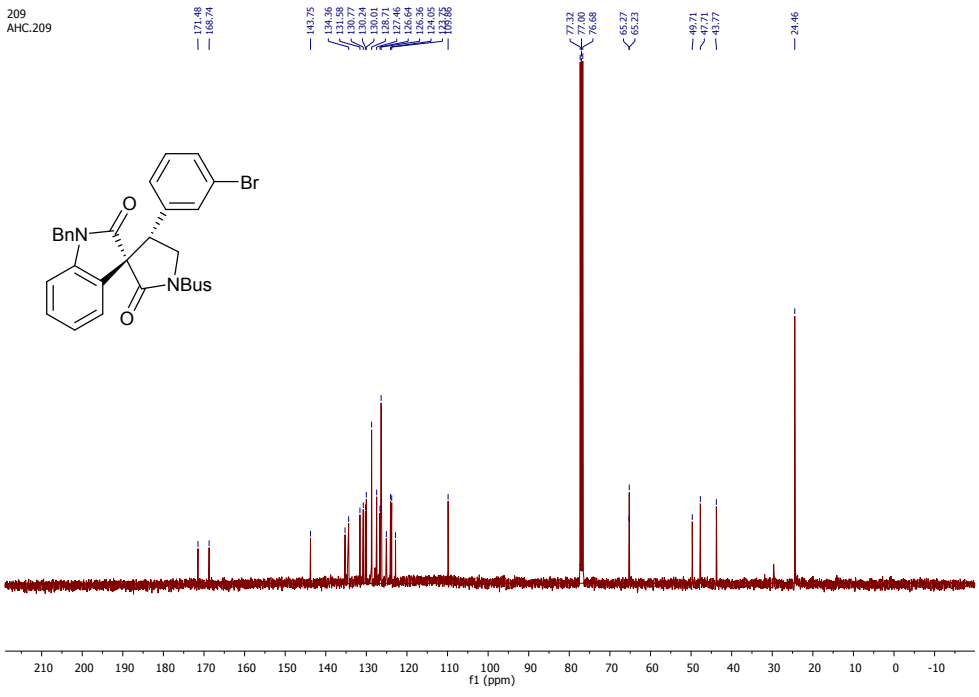
^{1}H NMR Spectrum of Compound **5g** (400 MHz, $CDCl_3$)



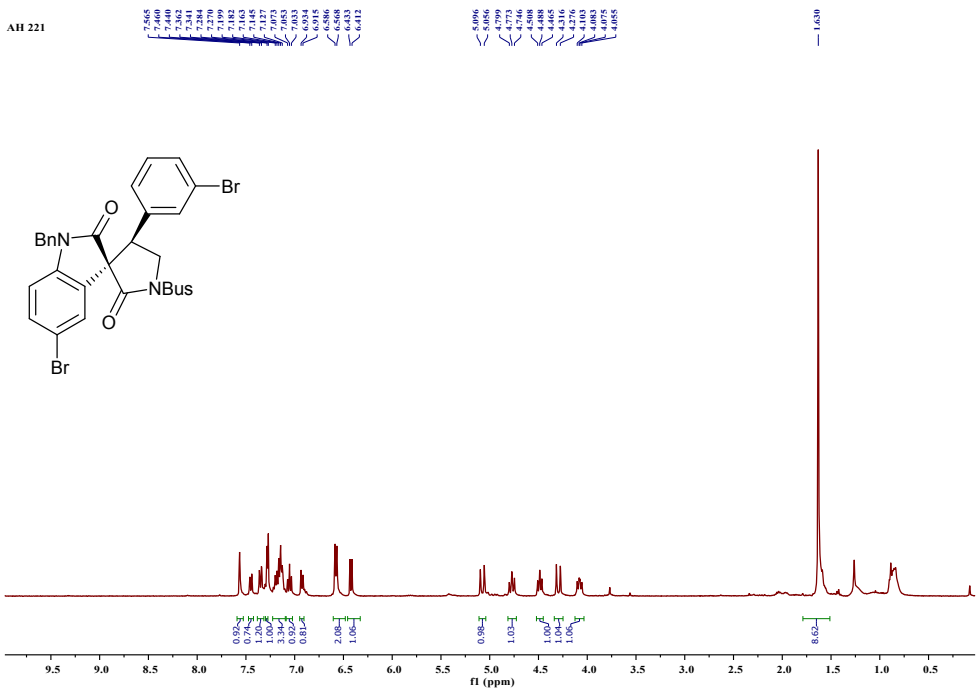
^{13}C NMR Spectrum of Compound **5g** (100 MHz, $CDCl_3$)



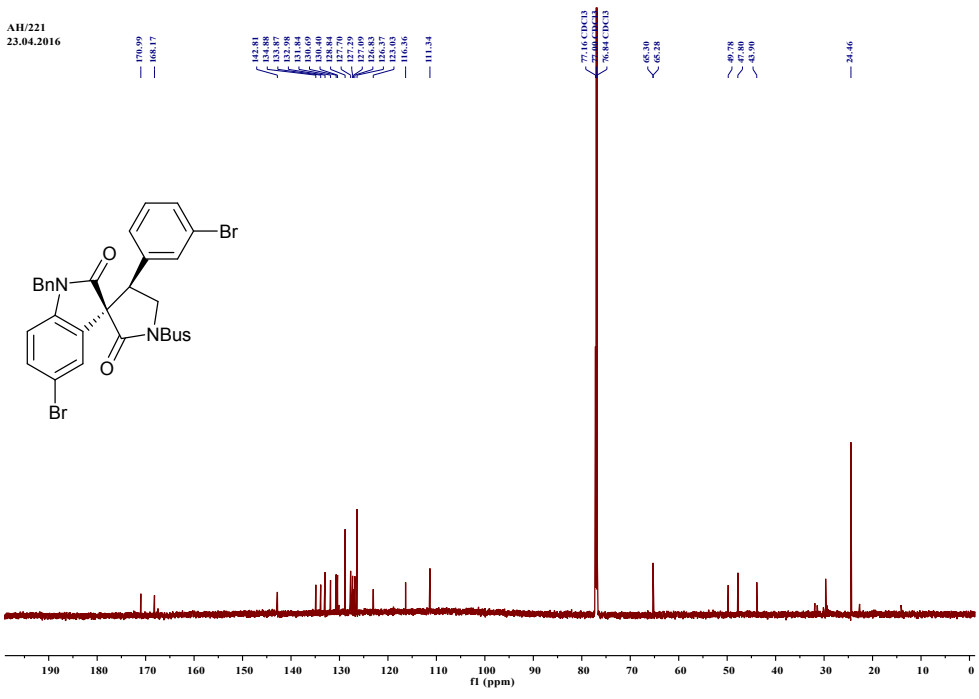
^{1}H NMR Spectrum of Compound **5u** (400 MHz, $CDCl_3$)



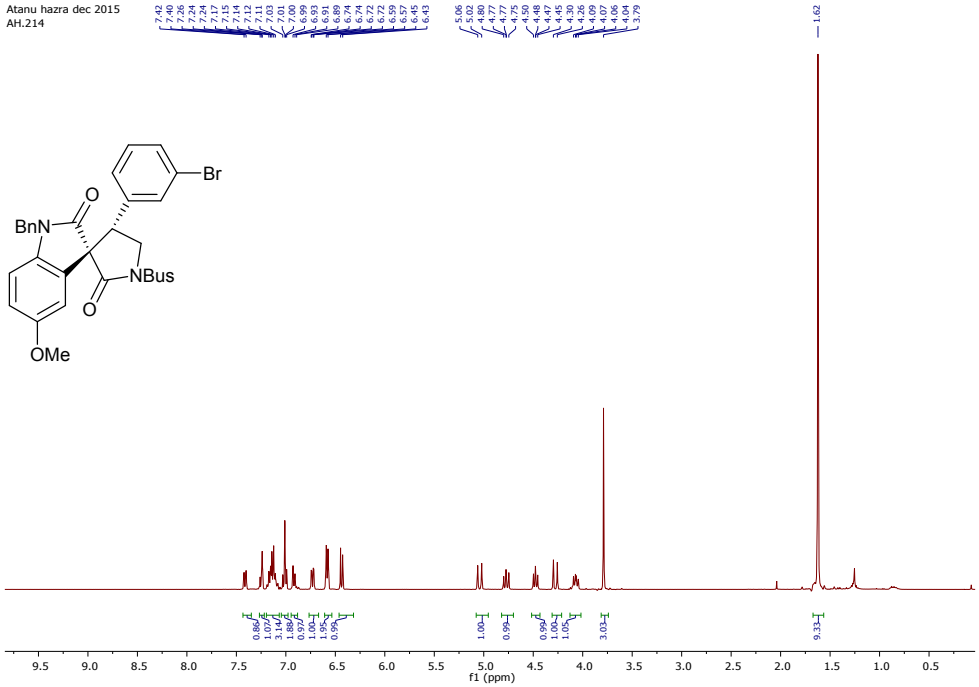
^{13}C NMR Spectrum of Compound **5u** (100 MHz, $CDCl_3$)



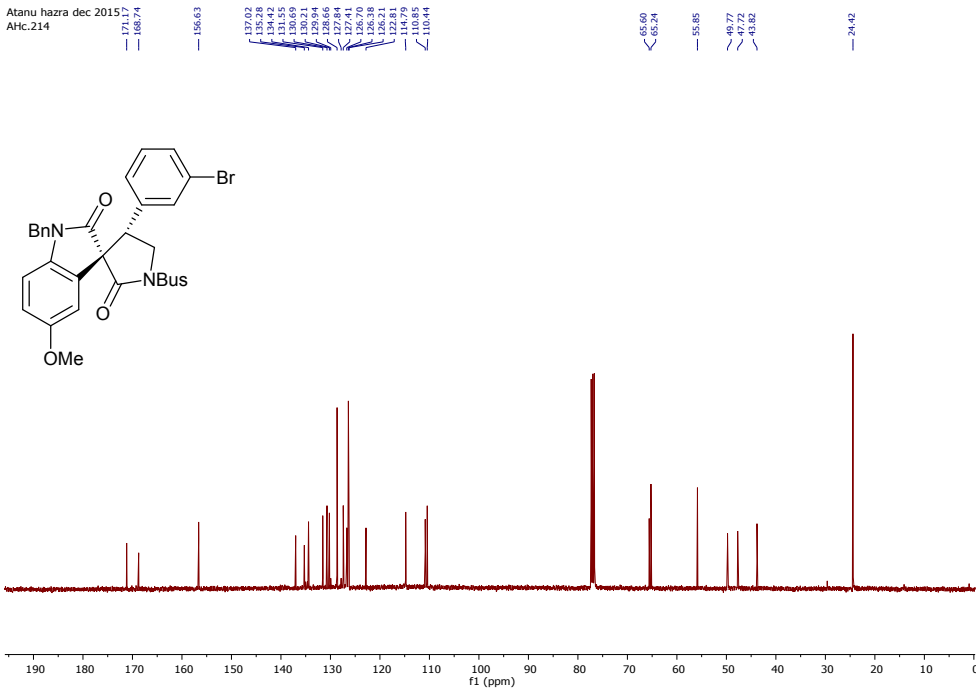
^{1}H NMR Spectrum of Compound ***ent*-5v** (400 MHz, $CDCl_3$)



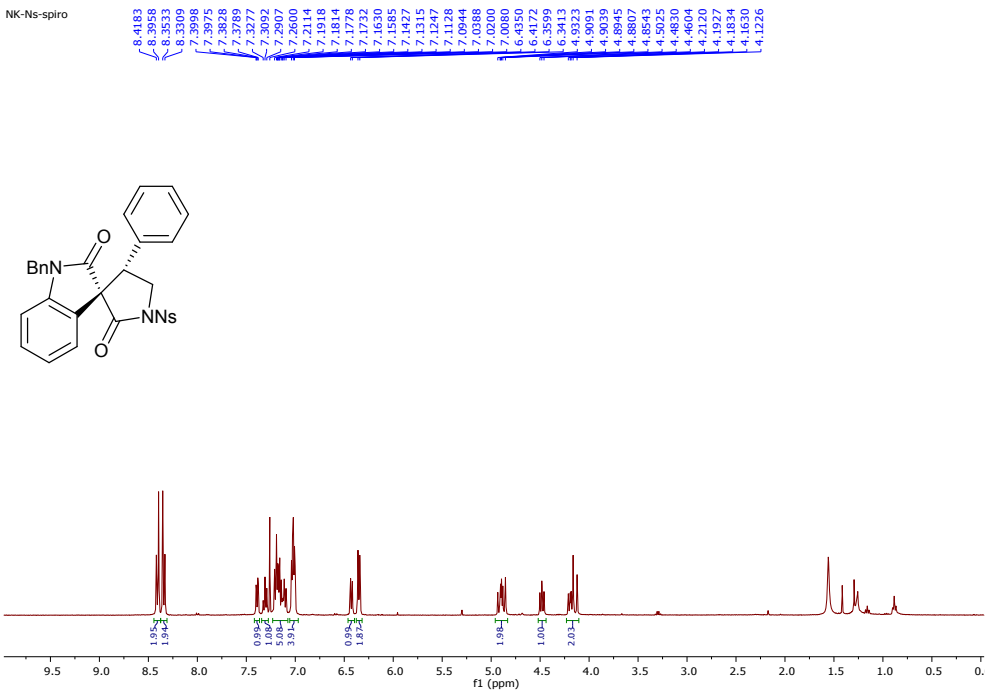
^{13}C NMR Spectrum of Compound ***ent*-5v** (100 MHz, $CDCl_3$)



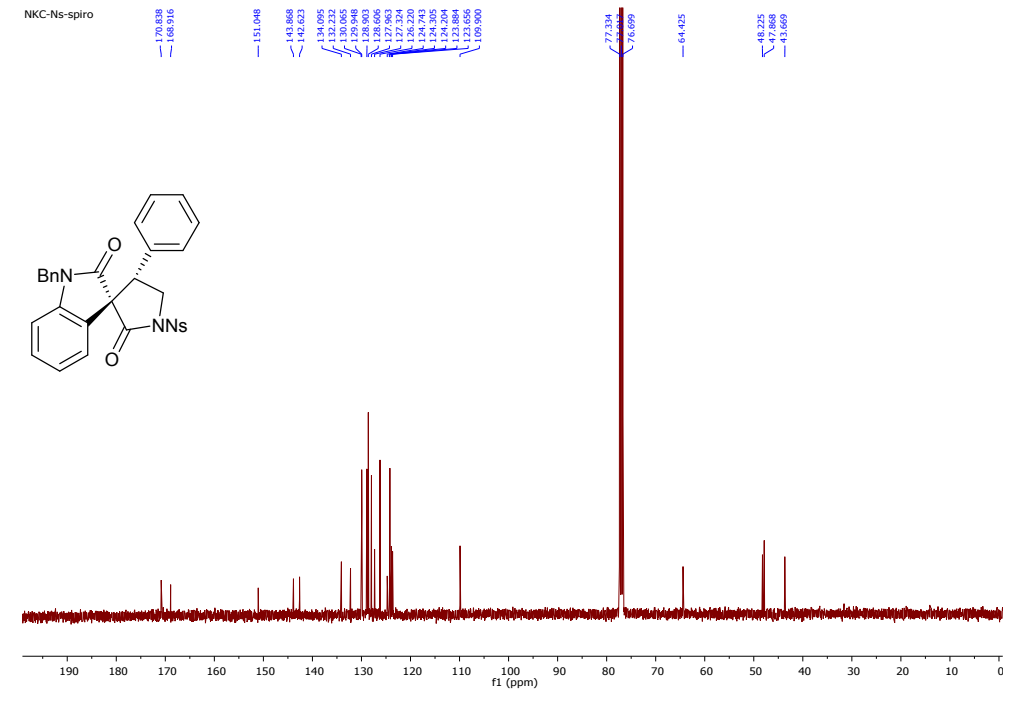
^{1}H NMR Spectrum of Compound **5w (**400 MHz, $CDCl_3$)



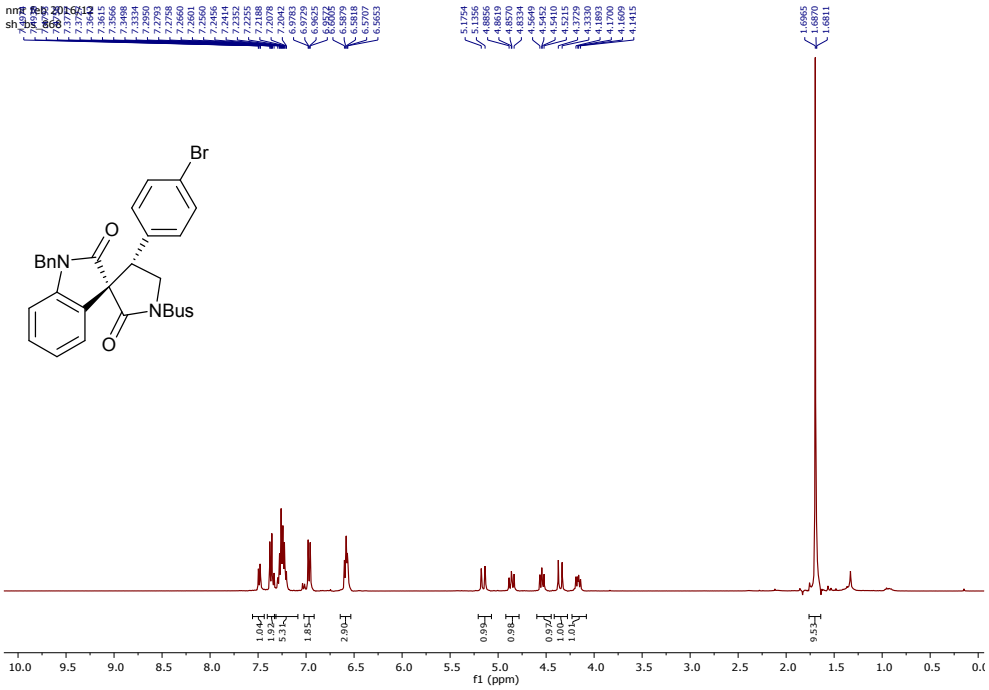
^{13}C NMR Spectrum of Compound **5w** (100 MHz, $CDCl_3$)



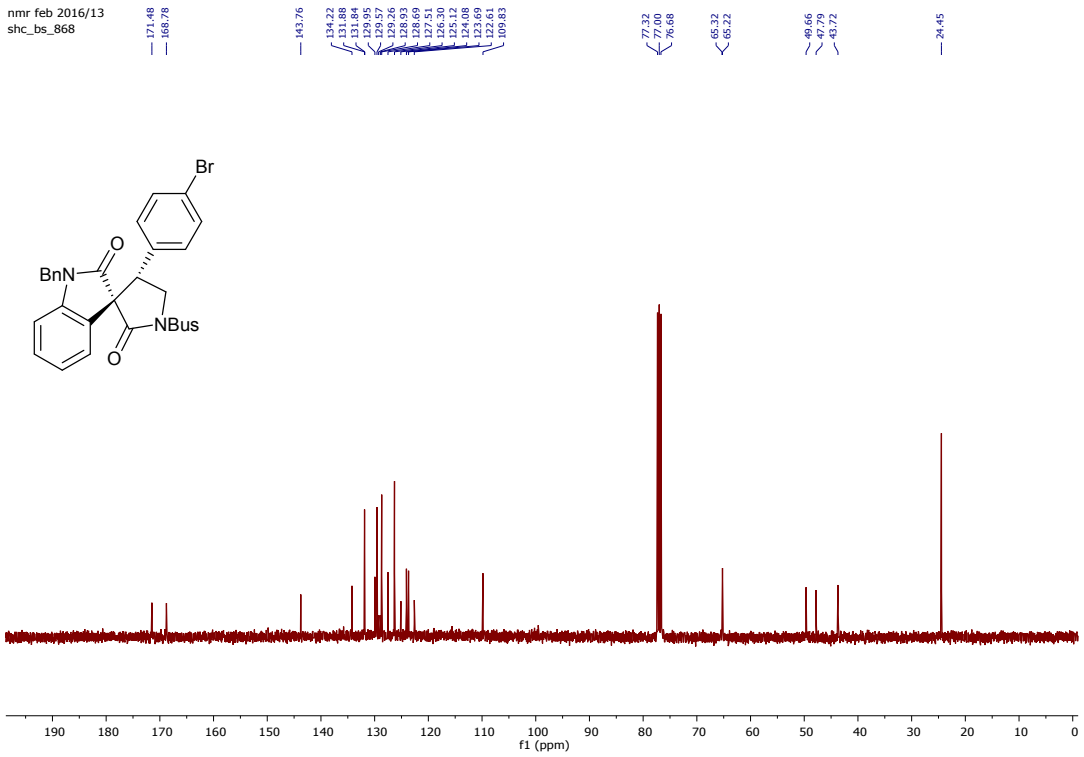
^{1}H NMR Spectrum of Compound **5c** (400 MHz, $CDCl_3$)



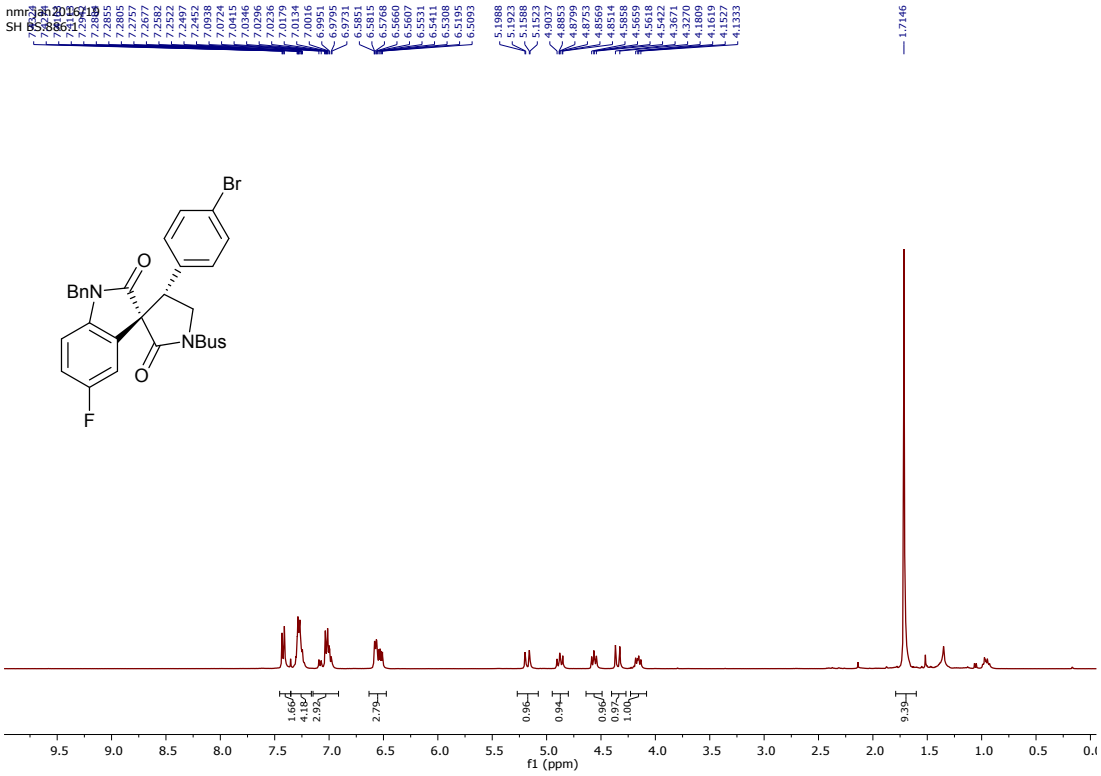
^{13}C NMR Spectrum of Compound **5c** (100 MHz, $CDCl_3$)



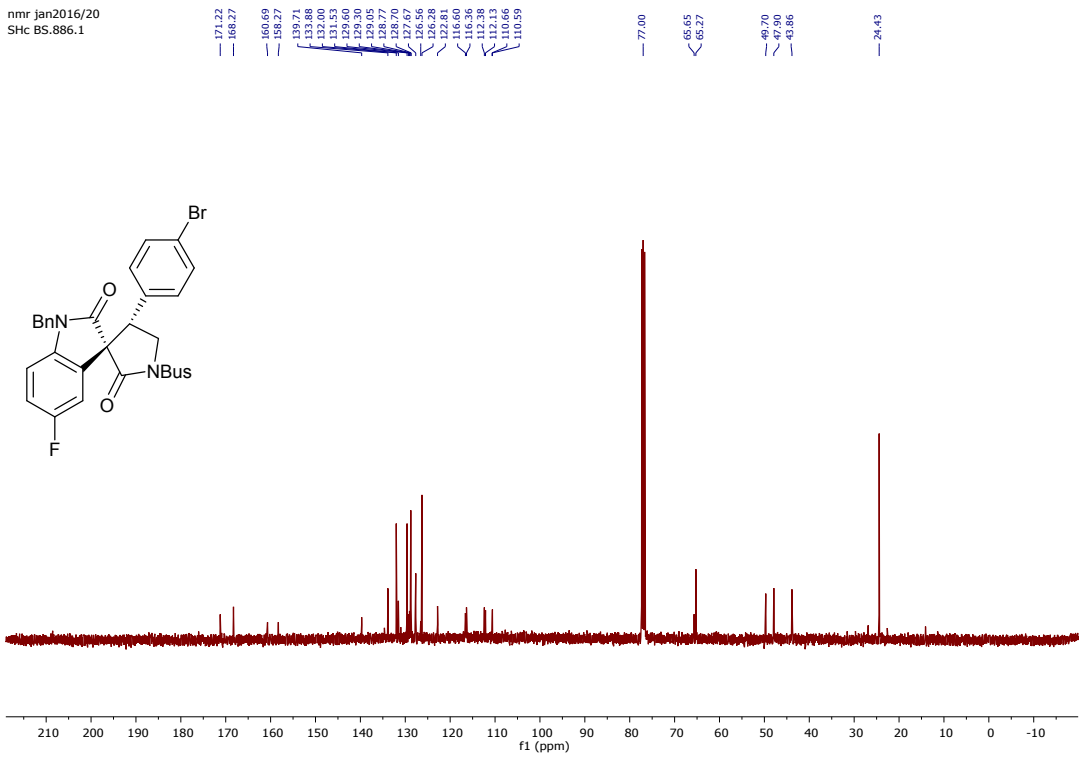
^{1}H NMR Spectrum of Compound **(±)-5h** (400 MHz, $CDCl_3$)



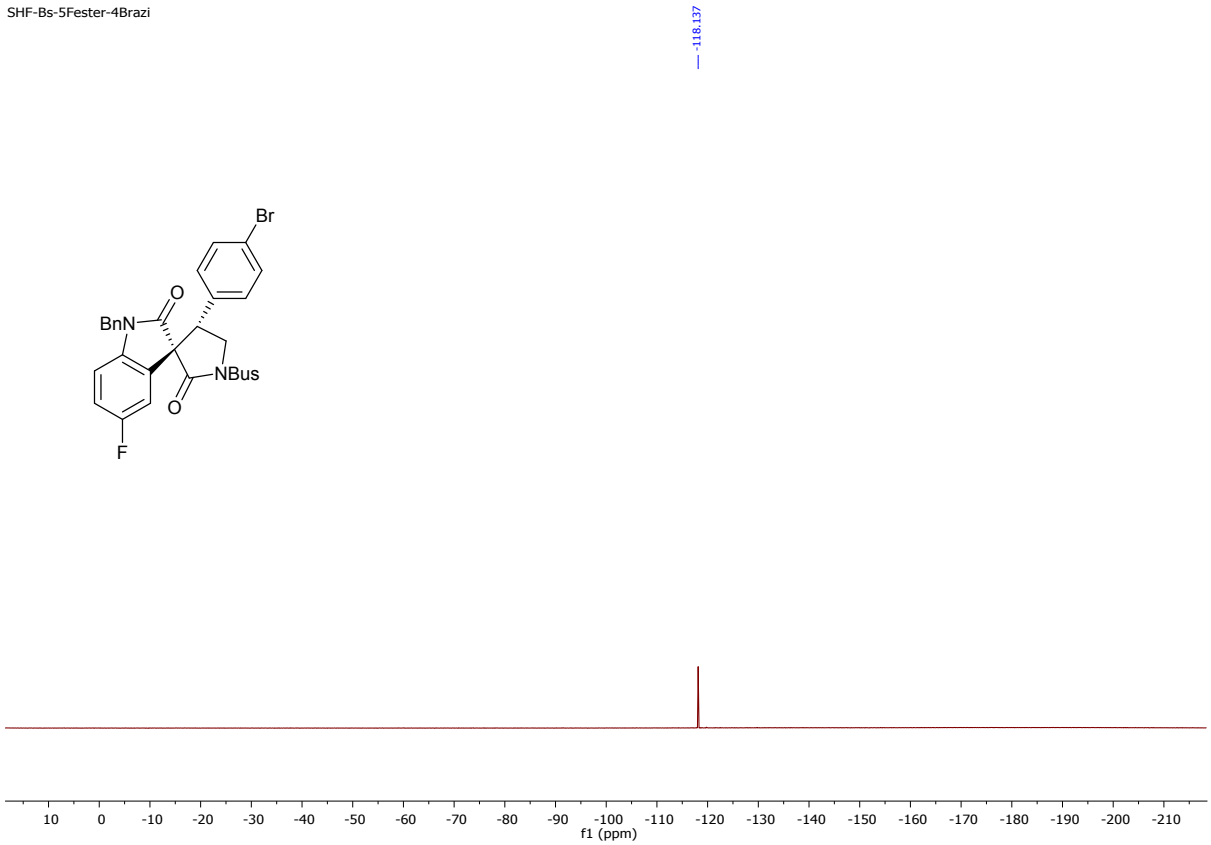
^{13}C NMR Spectrum of Compound **(±)-5h** (100 MHz, $CDCl_3$)



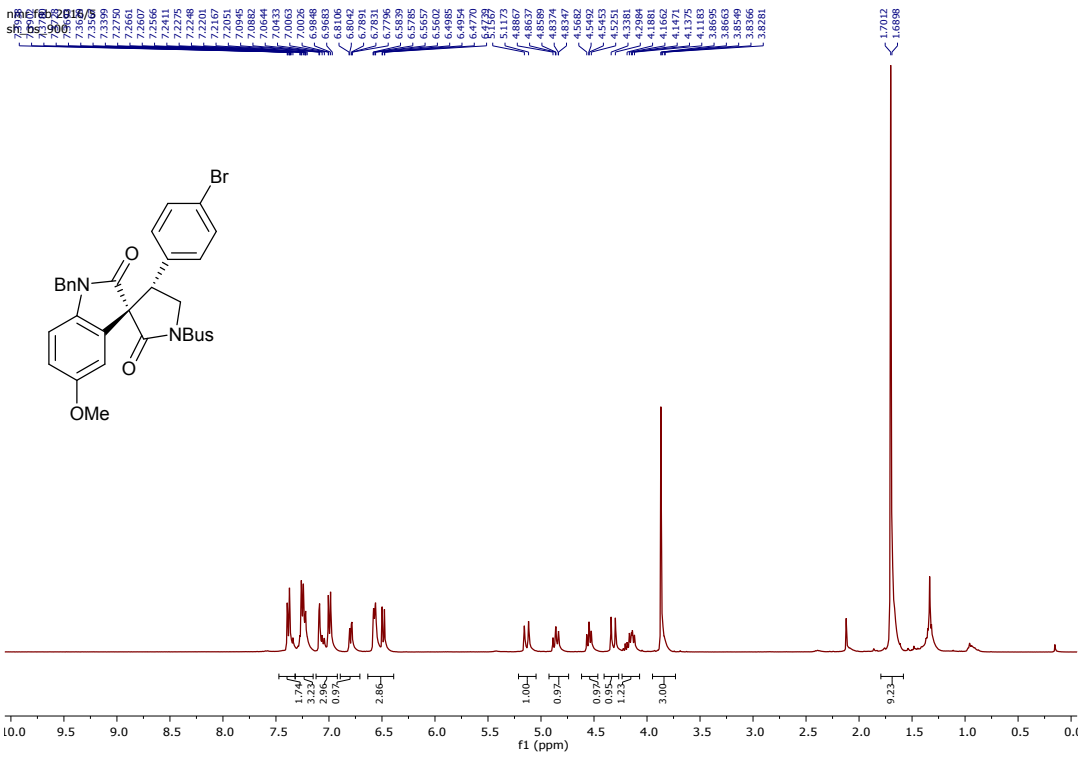
^{1}H NMR Spectrum of Compound **(±)-5i (**400 MHz, $CDCl_3$)



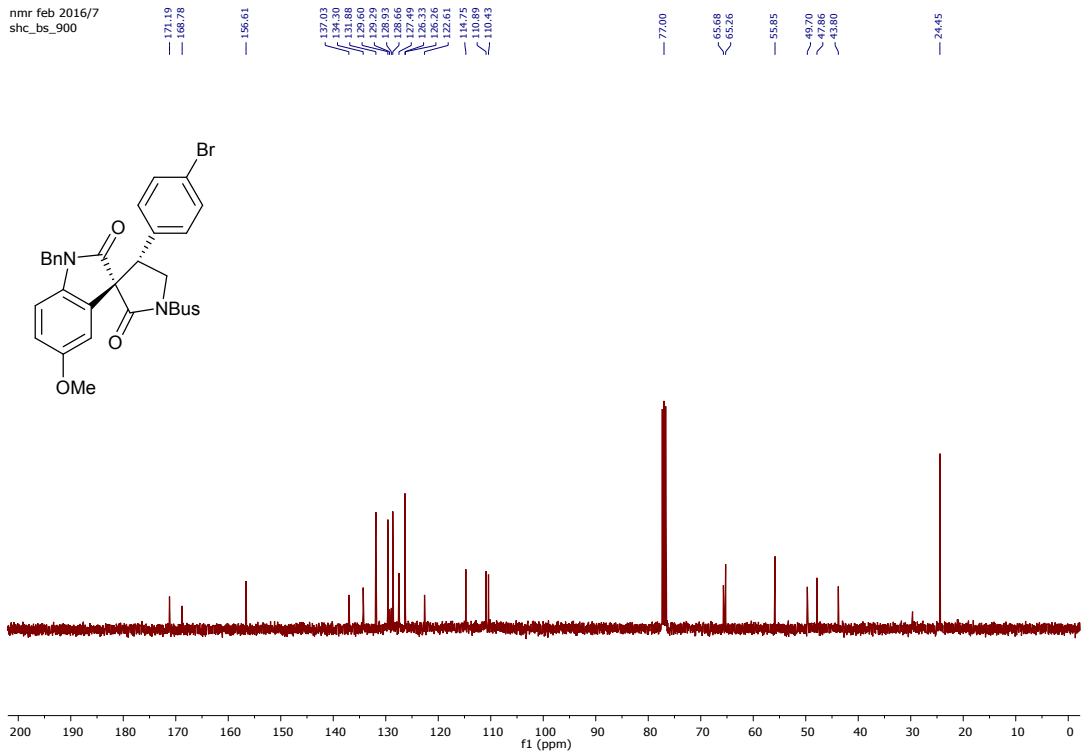
^{13}C NMR Spectrum of Compound **(±)-5i** (100 MHz, $CDCl_3$)



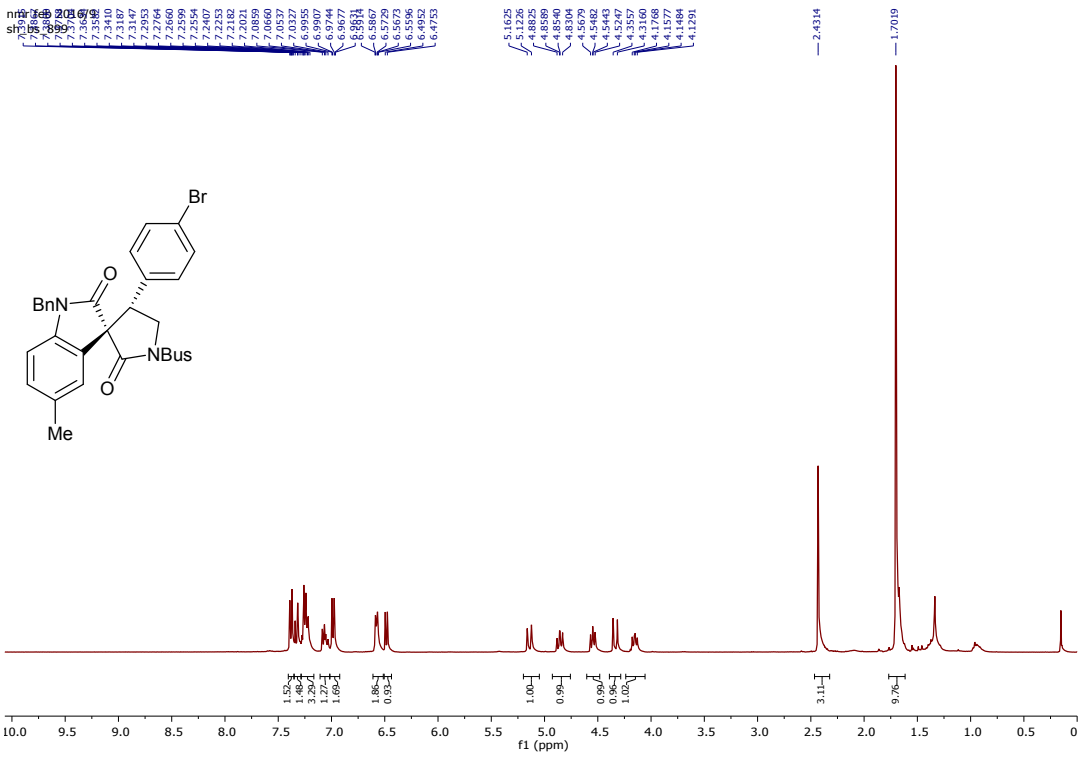
^{19}F NMR Spectrum of Compound **(±)-5i** (376 MHz, $CDCl_3$)



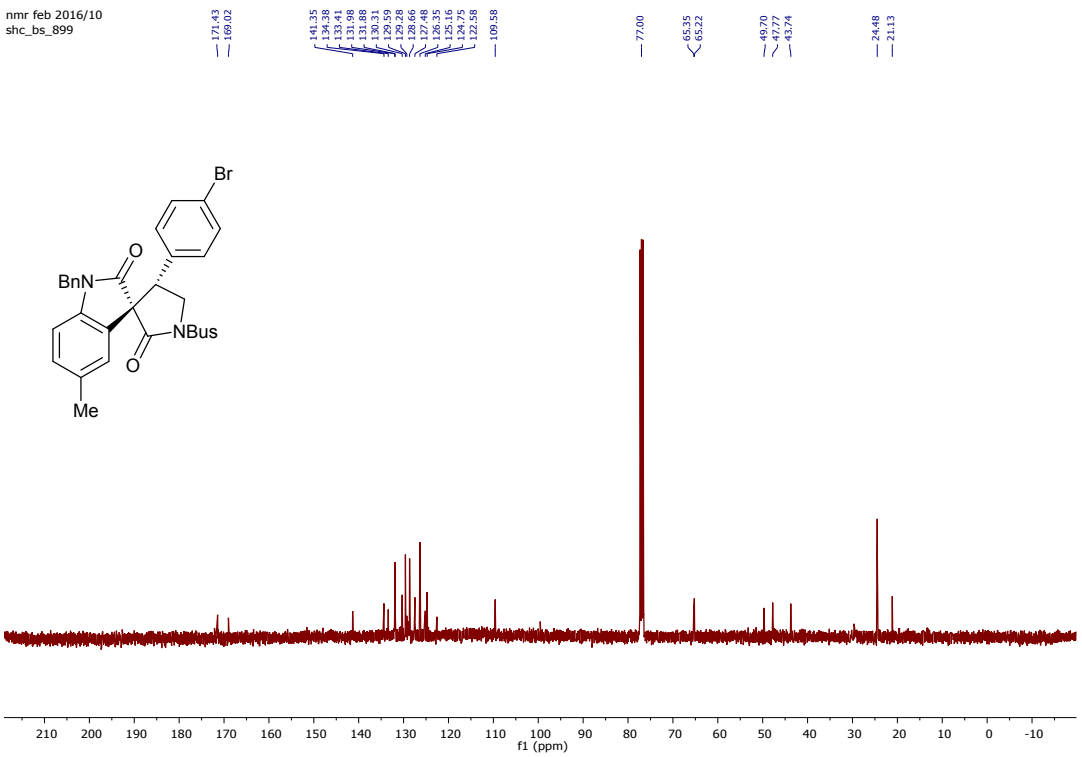
^{1}H NMR Spectrum of Compound **(±)-5j** (400 MHz, $CDCl_3$)



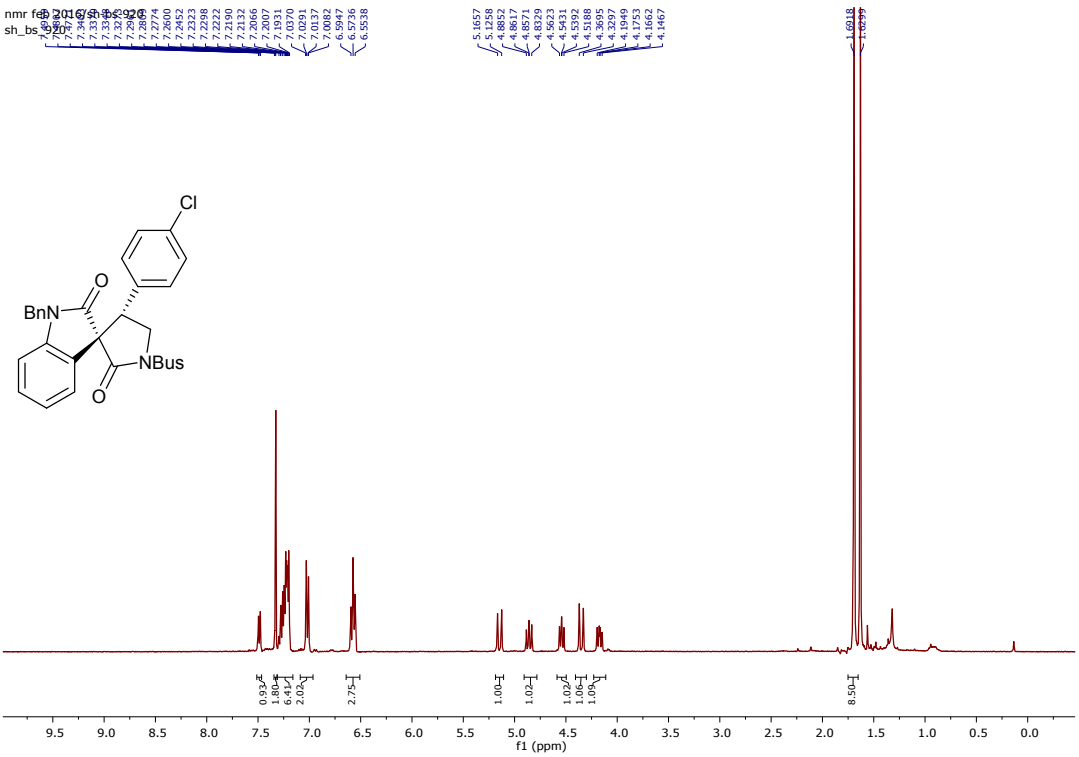
^{13}C NMR Spectrum of Compound **(±)-5j** (100 MHz, $CDCl_3$)



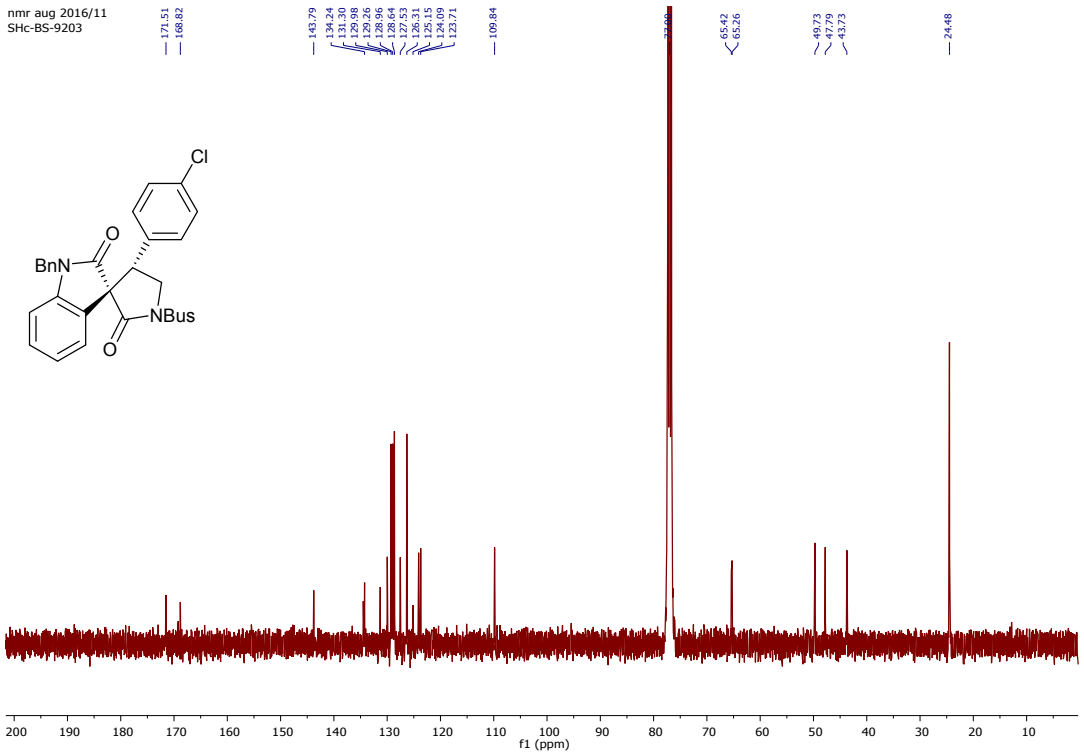
^{1}H NMR Spectrum of Compound **(±)-5k (**400 MHz, $CDCl_3$)



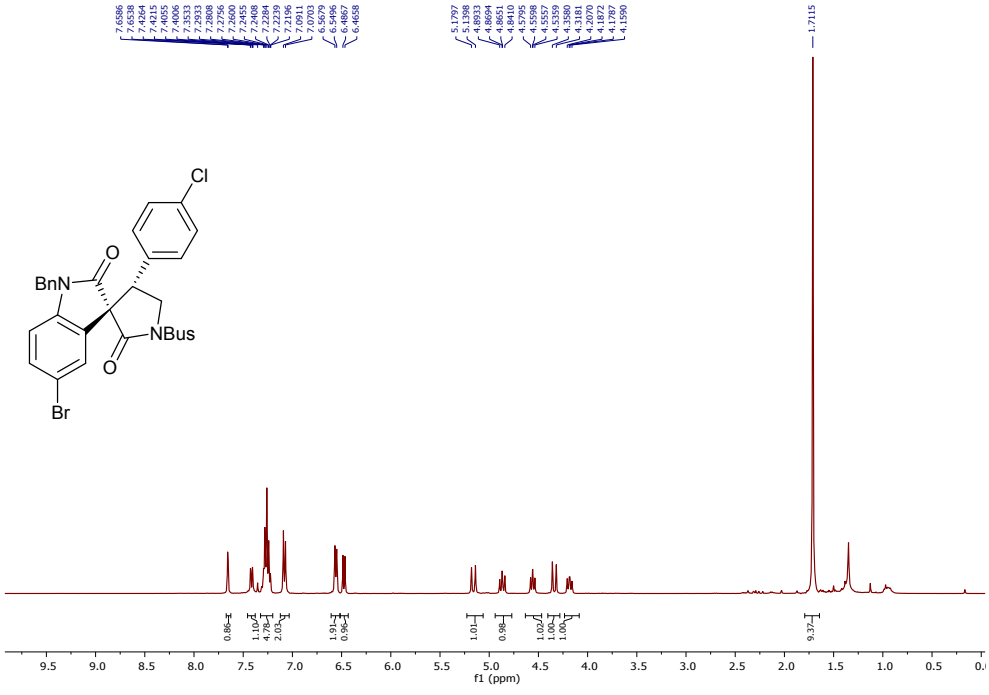
^{13}C NMR Spectrum of Compound **(±)-5k** (400 MHz, $CDCl_3$)



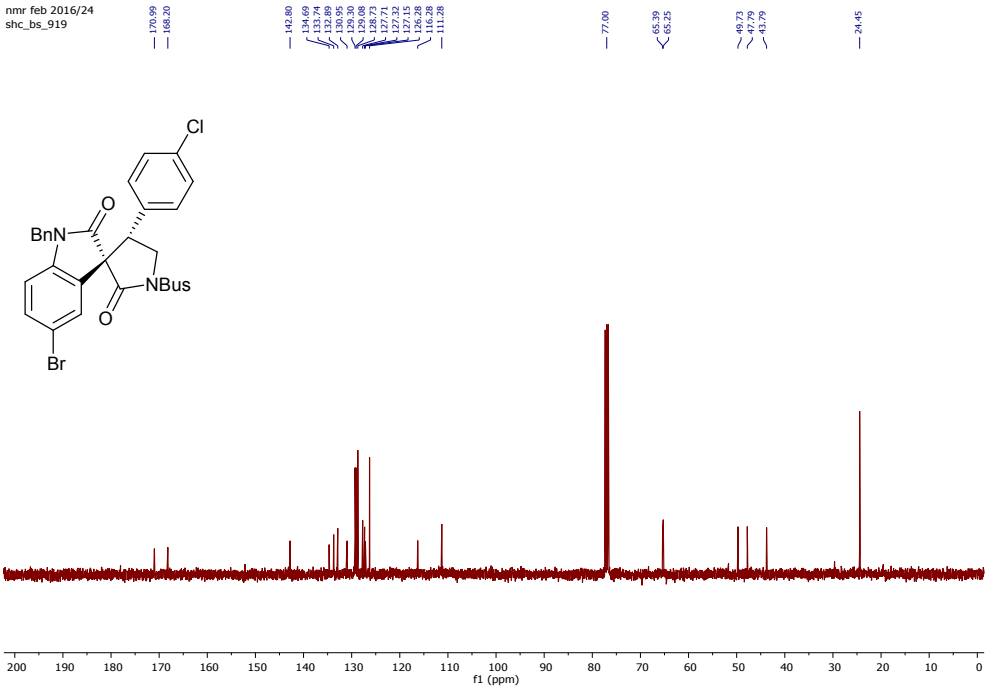
^{1}H NMR Spectrum of Compound **(±)-5l** (400 MHz, $CDCl_3$)



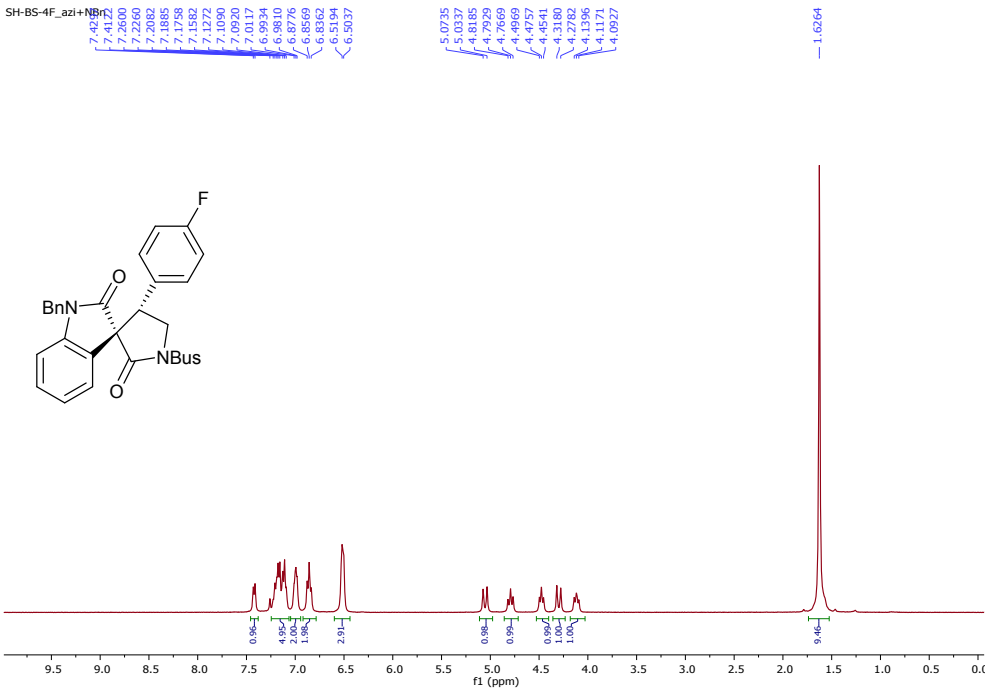
^{13}C NMR Spectrum of Compound **(±)-5l (**400 MHz, $CDCl_3$)



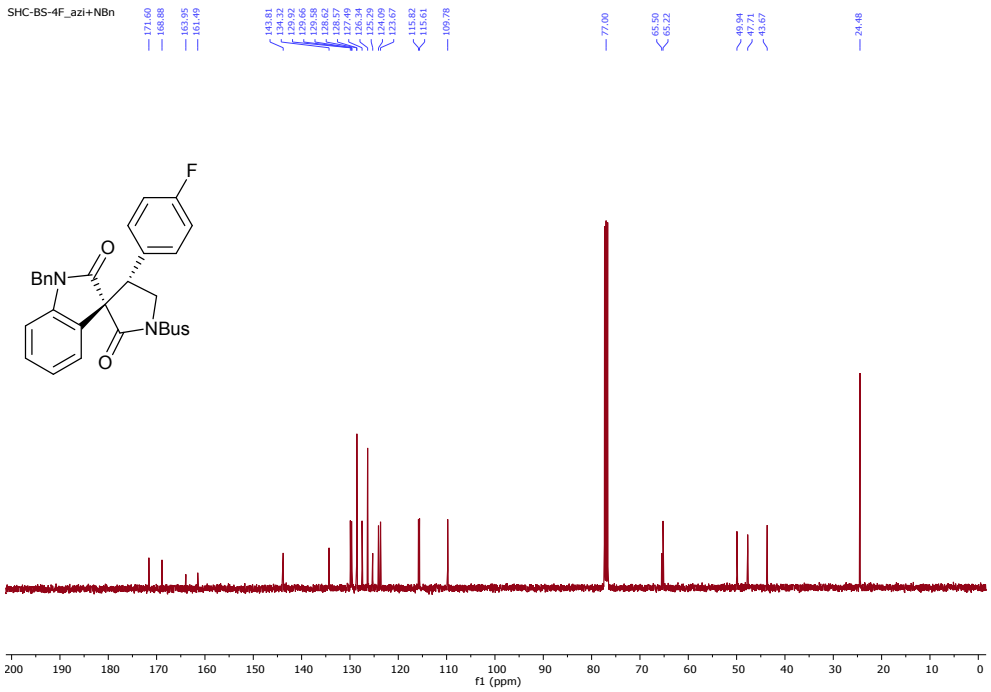
^{1}H NMR Spectrum of Compound **(±)-5m (**400 MHz, $CDCl_3$)



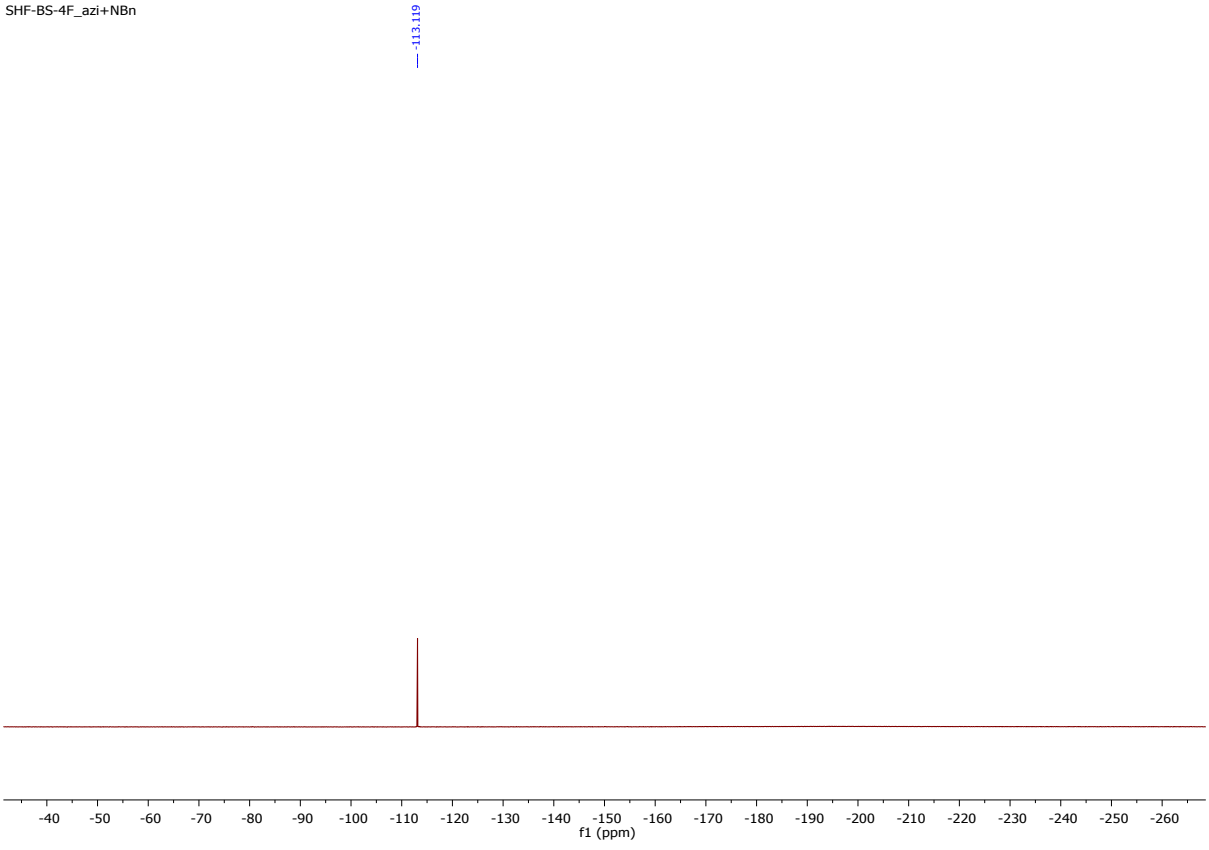
^{13}C NMR Spectrum of Compound **(±)-5m** (400 MHz, $CDCl_3$)



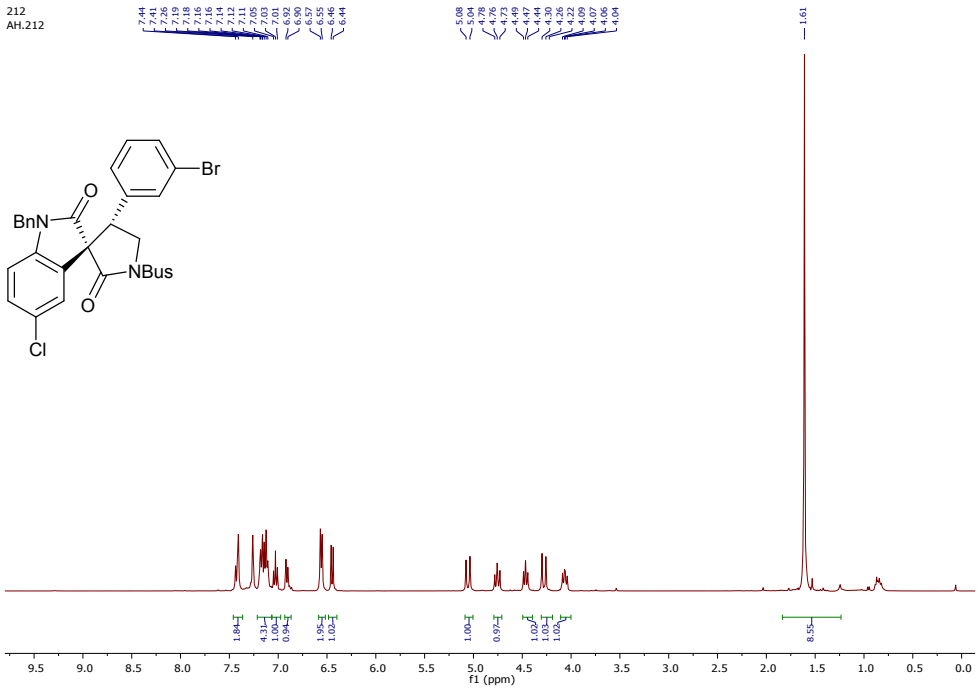
^{1}H NMR Spectrum of Compound **(±)-5n** (400 MHz, $CDCl_3$)



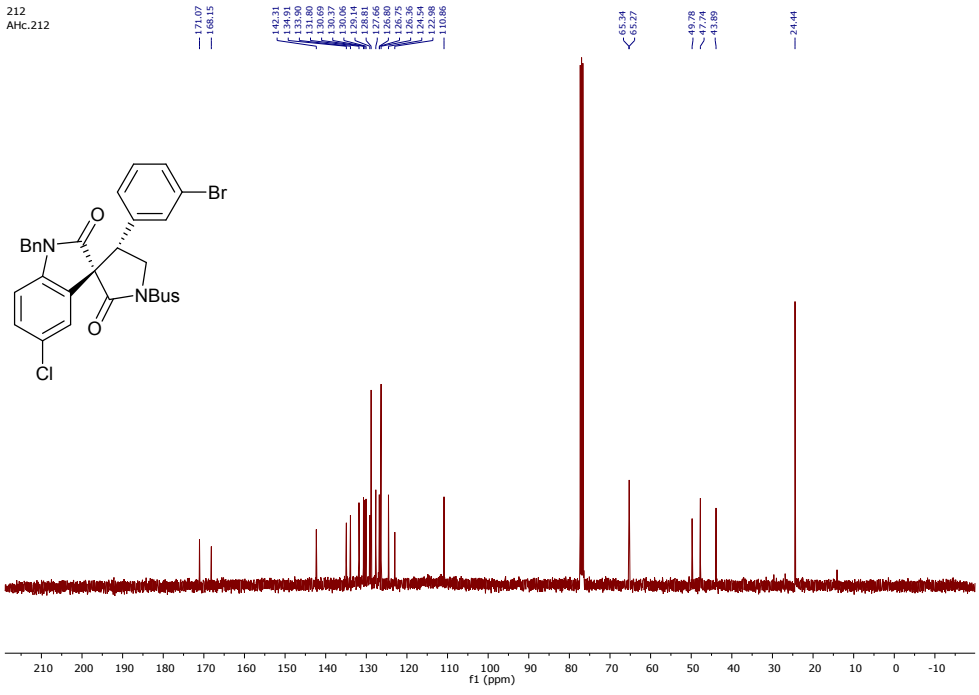
^{13}C NMR Spectrum of Compound **(±)-5n** (100 MHz, $CDCl_3$)



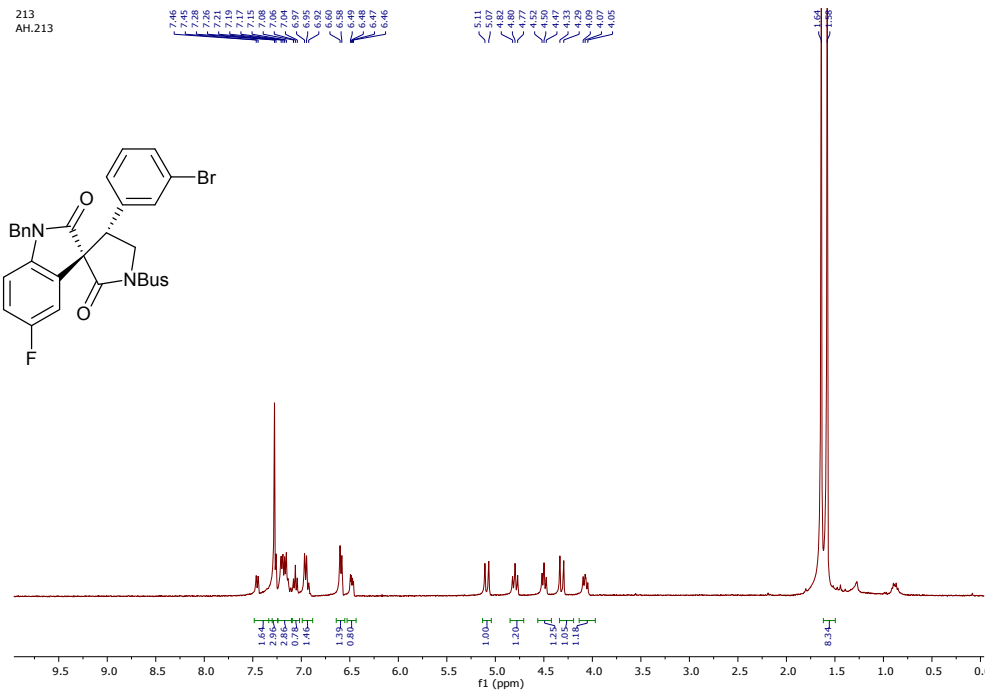
^{19}F NMR Spectrum of Compound **(±)-5n** (376 MHz, $CDCl_3$)



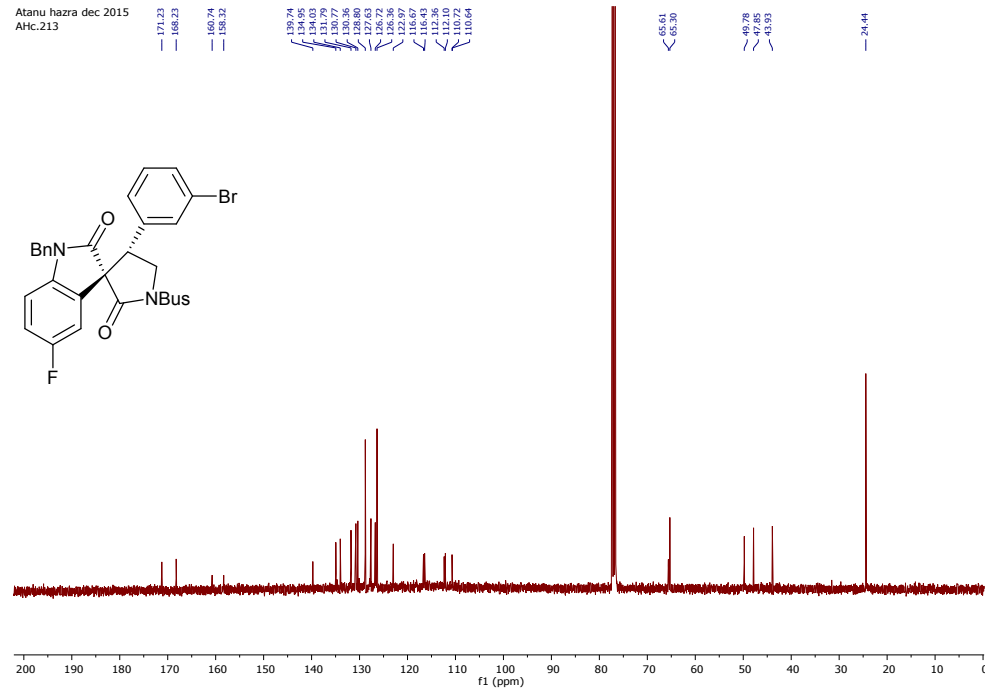
^{1}H NMR Spectrum of Compound **(±)-5o** (400 MHz, $CDCl_3$)



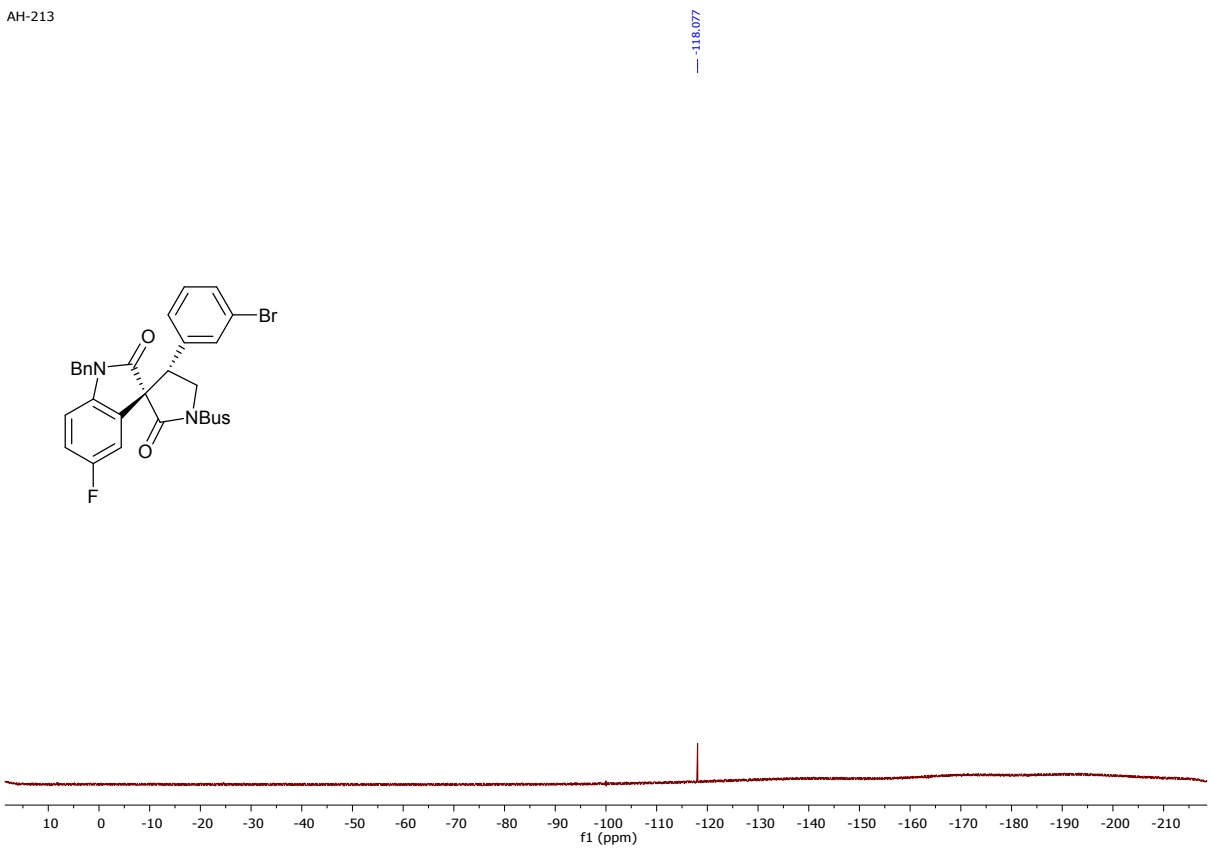
^{13}C NMR Spectrum of Compound **(±)-5o** (100 MHz, $CDCl_3$)



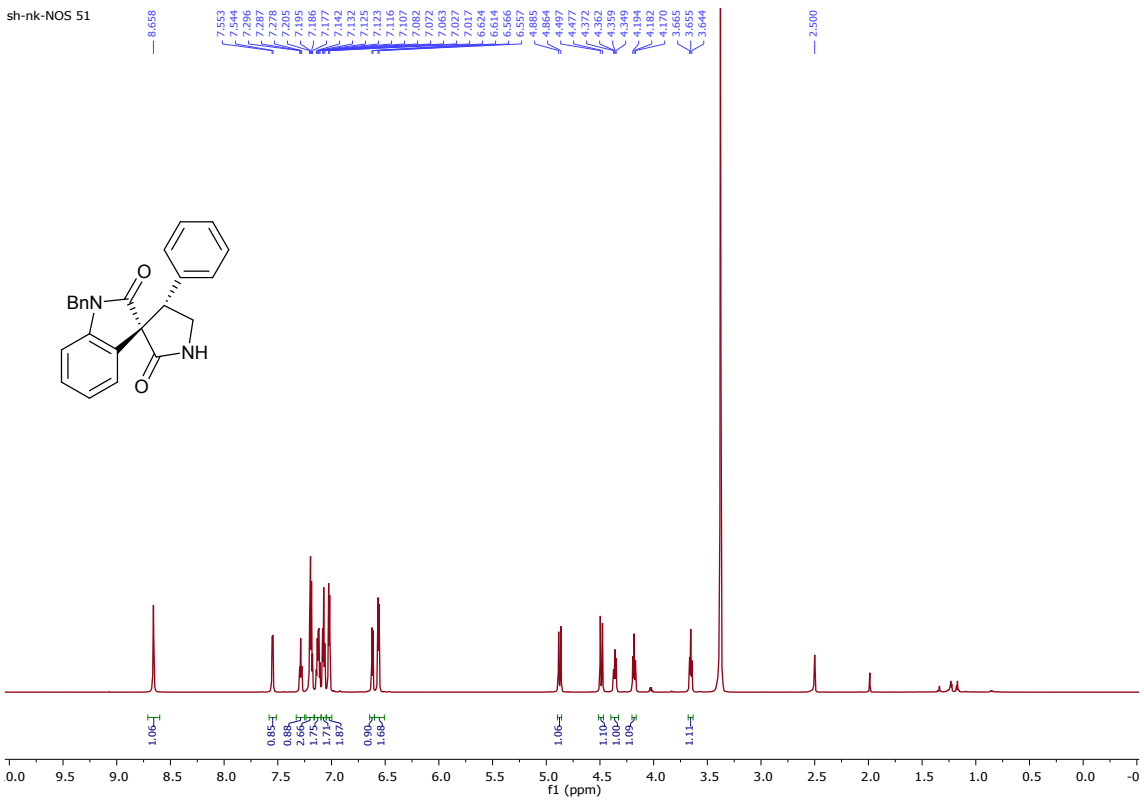
^{1}H NMR Spectrum of Compound **(±)-5p (**400 MHz, $CDCl_3$)



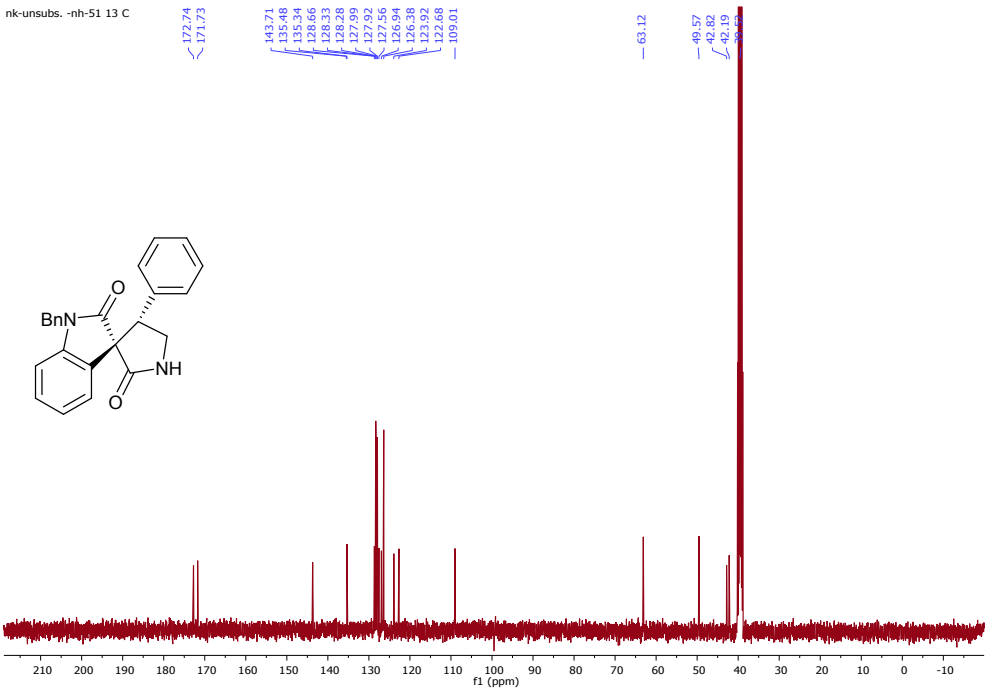
^{13}C NMR Spectrum of Compound **(±)-5p** (100 MHz, $CDCl_3$)



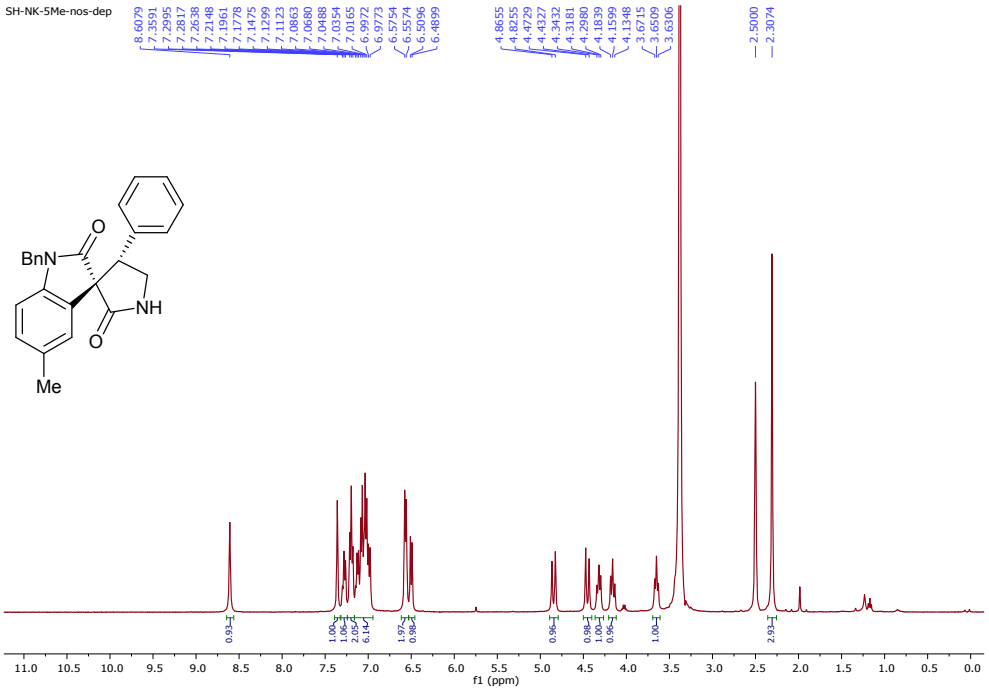
^{19}F NMR Spectrum of Compound **(±)-5p** (376 MHz, $CDCl_3$)



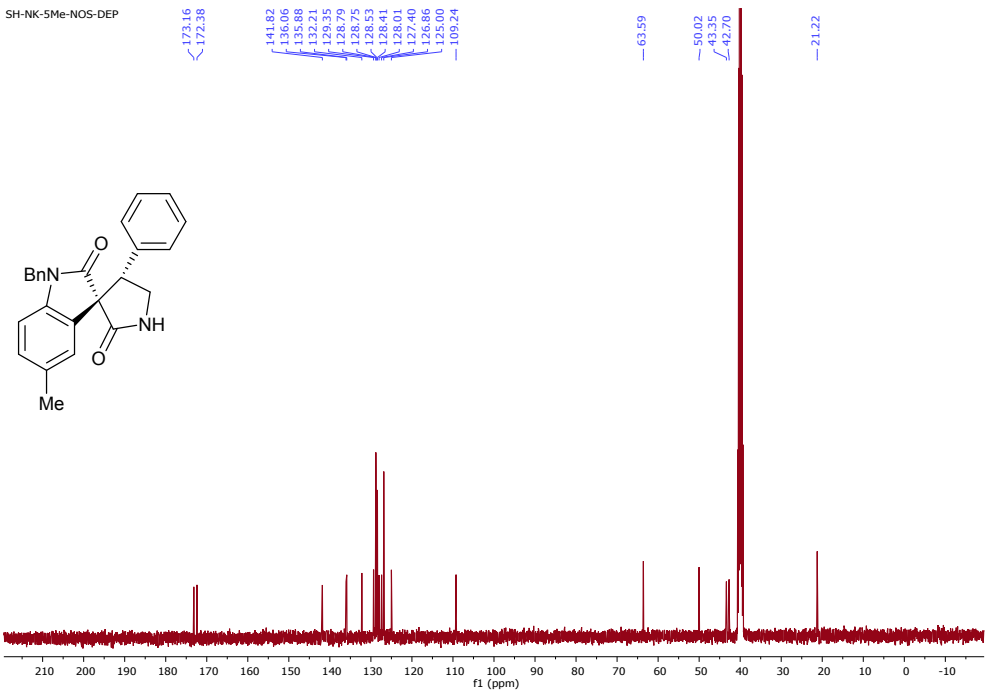
^{1}H NMR Spectrum of Compound **7b** (400 MHz, DMSO-d_6)



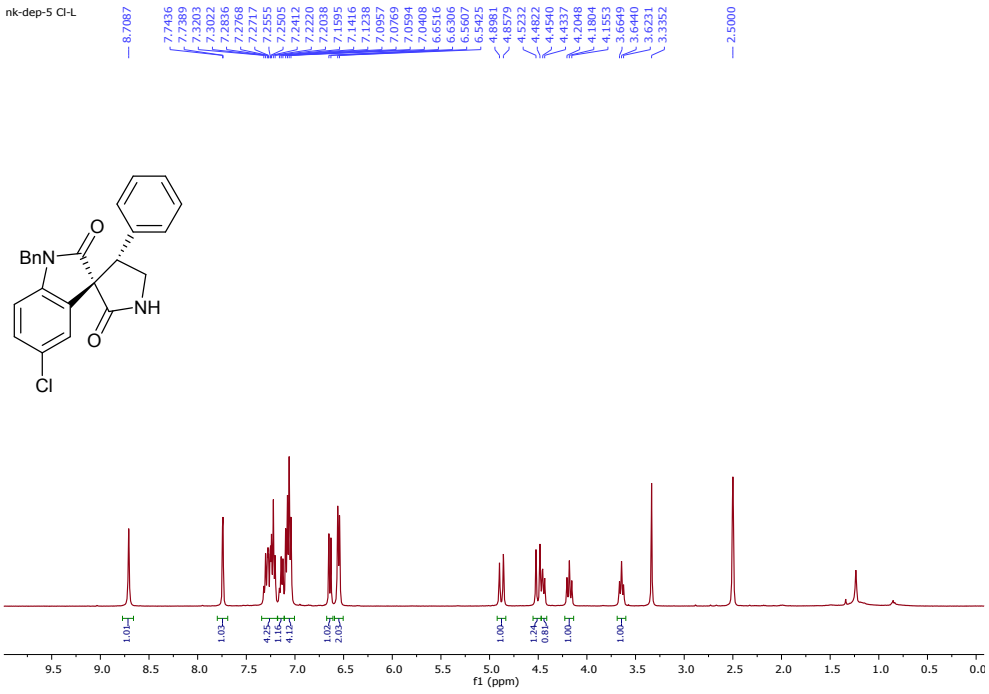
^{13}C NMR Spectrum of Compound **7b** (400 MHz, DMSO-d_6)



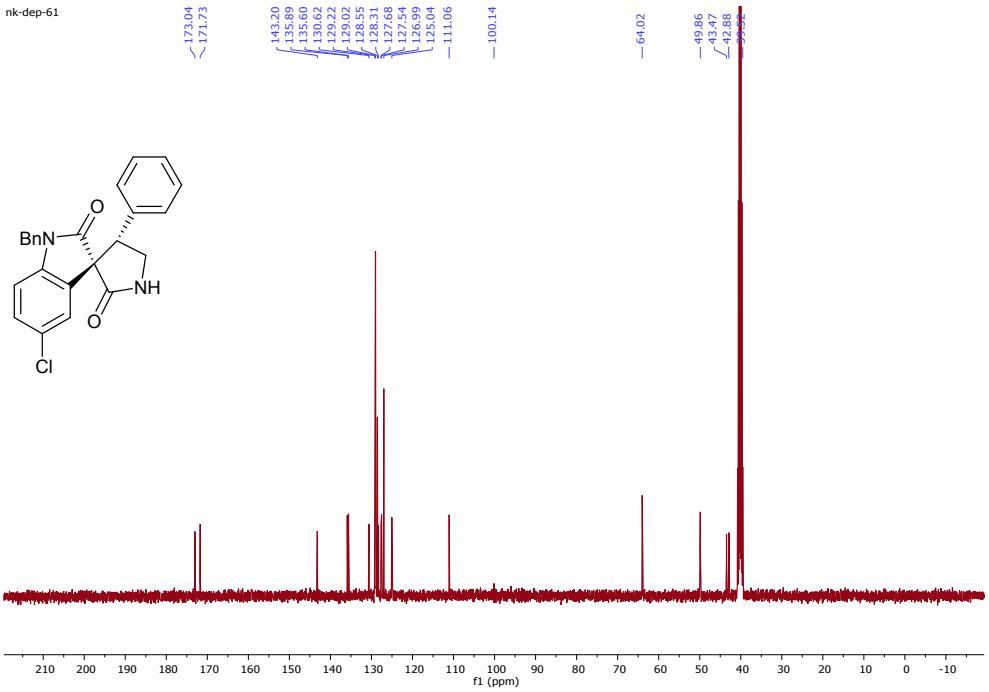
^{1}H NMR Spectrum of Compound **7c** (400 MHz, DMSO-d_6)



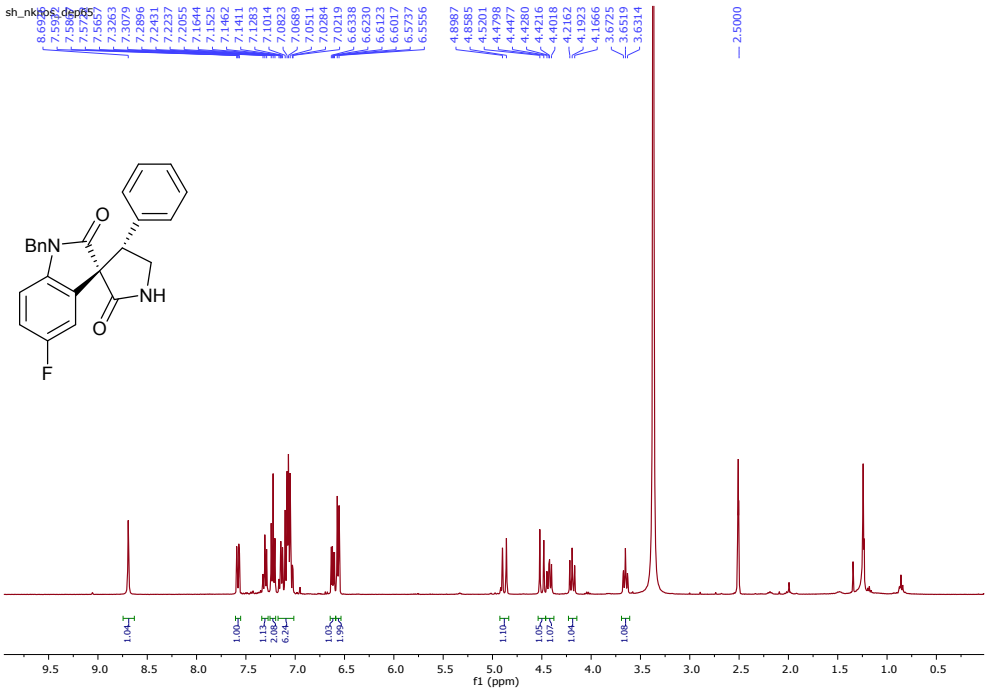
^{13}C NMR Spectrum of Compound **7c** (100 MHz, DMSO-d_6)



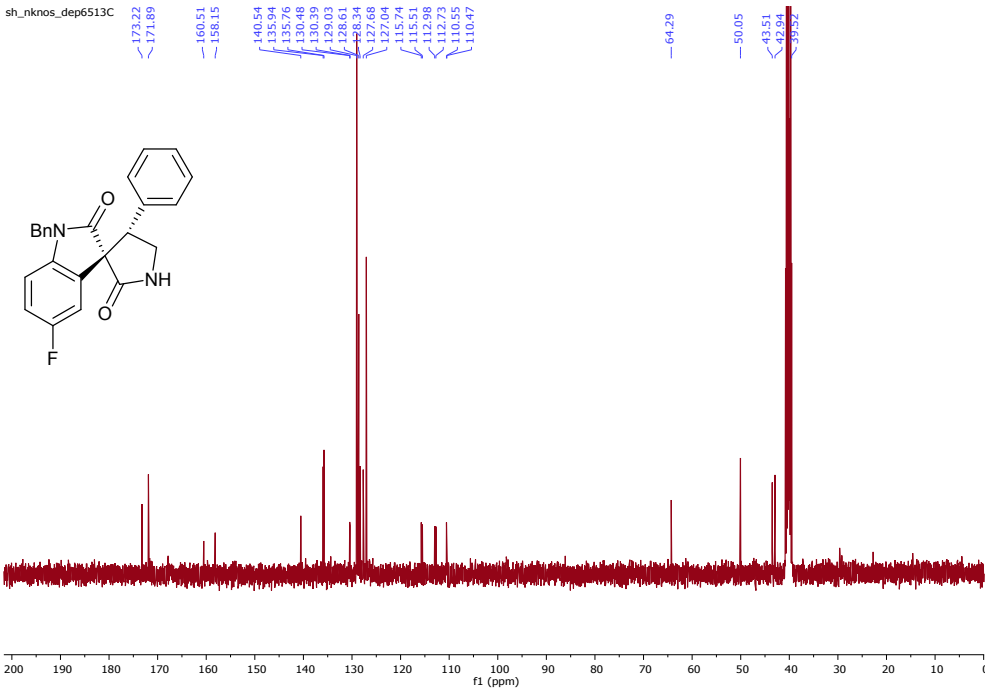
^{1}H NMR Spectrum of Compound **7d** (400 MHz, DMSO-d_6)



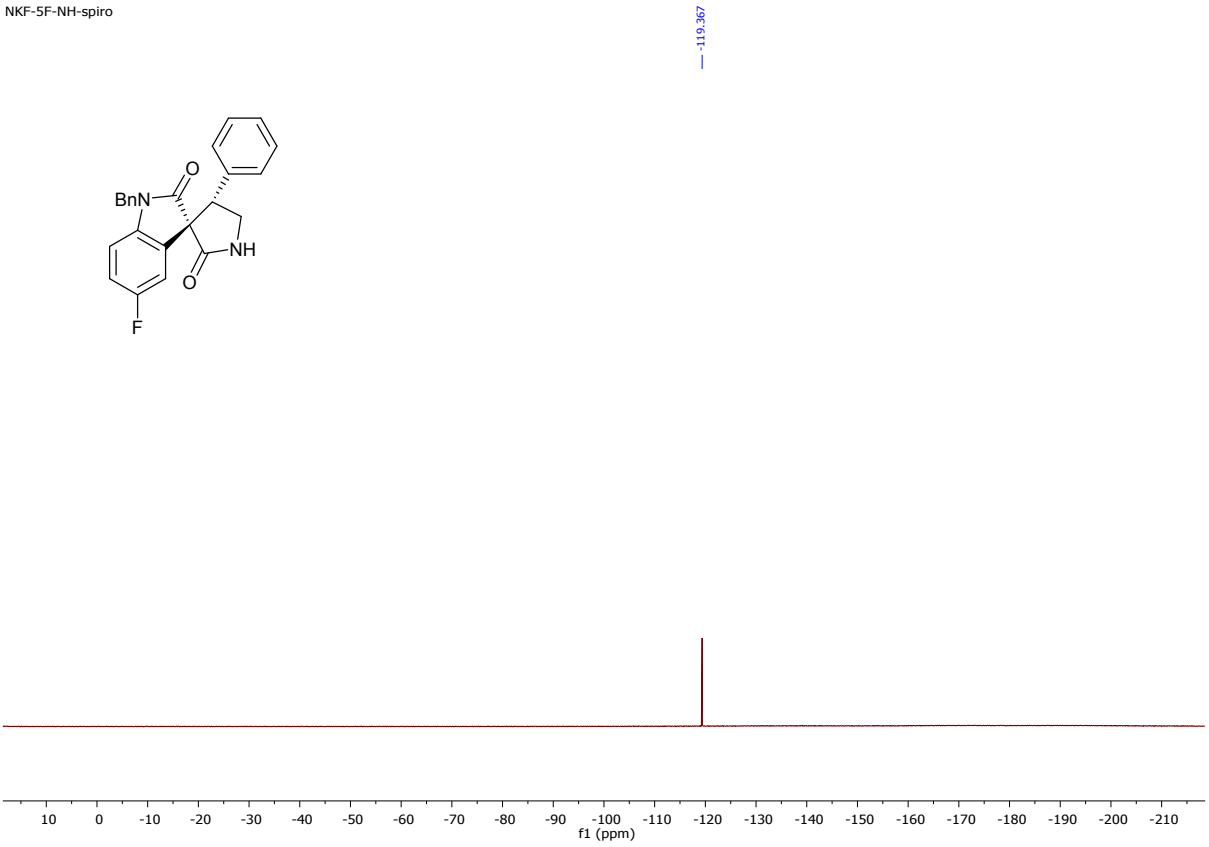
^{13}C NMR Spectrum of Compound **7d** (100 MHz, DMSO-d_6)



^{1}HNMR Spectrum of Compound **7e** (400 MHz, DMSO-d_6)



^{13}C NMR Spectrum of Compound **7e** (100 MHz, DMSO-d_6)

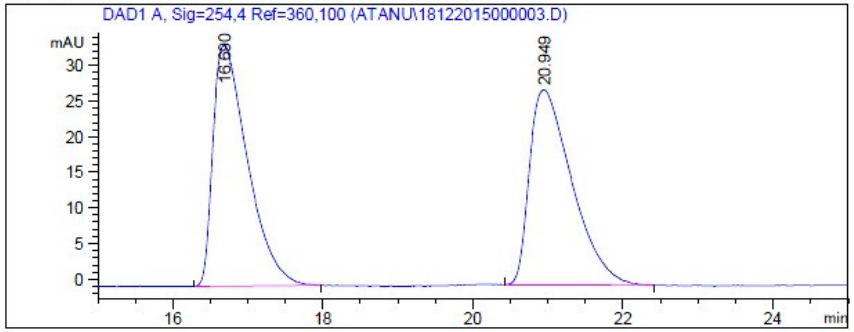


^{19}F NMR Spectrum of Compound **7e** (376 MHz, $CDCl_3$)

HPLC Chromatograms

Sample Info : RAC-PH_AZI_18_12_0C
,OJ-H Column,30 % iPr/Hexane 1.0 ml/min flow rate, 254 nm

Additional Info : Peak(s) manually integrated

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=====================================================================
                      Area Percent Report
=====================================================================

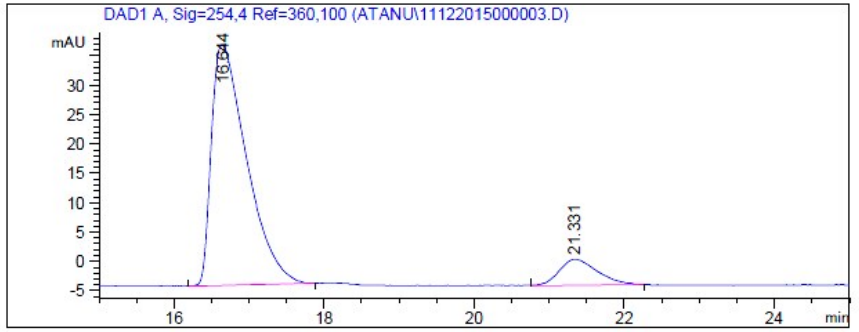
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.690 BB    0.4795 1083.28918   33.83873  50.0799
   2  20.949 BB    0.5864 1079.83362   27.36277  49.9201
```

HPLC Chromatogram of recovered aziridine **(±)-4a**

Sample Info : L_PH_GLY_AZI_11_12
,OJ-H Column, % iPr/Hexane 1.0 ml/min flow rate, 254 nm

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                      Area Percent Report
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Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

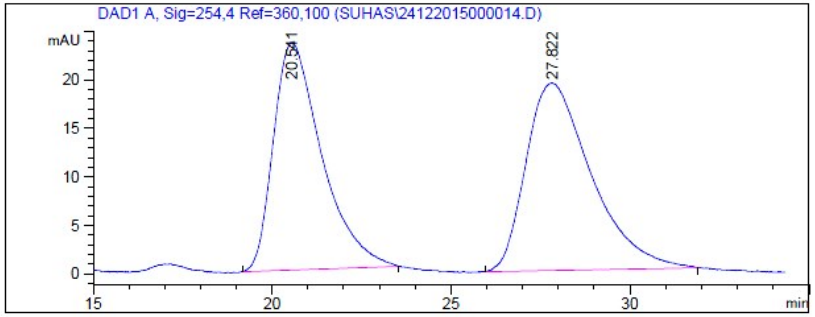

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.644 BB    0.4889 1337.37000   40.96301  89.4888
   2  21.331 BB    0.4301  157.08560    4.42545  10.5112
```

HPLC Chromatogram of recovered aziridine **4a**

HPLC condition **4a**: CHIRALCEL OJ-H, 30% iPrOH/n-Hexane, 1ml/min, 254 nm, t_R = 16.644 min (major) t_R = 21.331 min (minor)

Sample Info : 87050503
,OD-H Column,30 % iPr/Hexane 1.0 ml/min flow rate, 254 nm

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                      Area Percent Report
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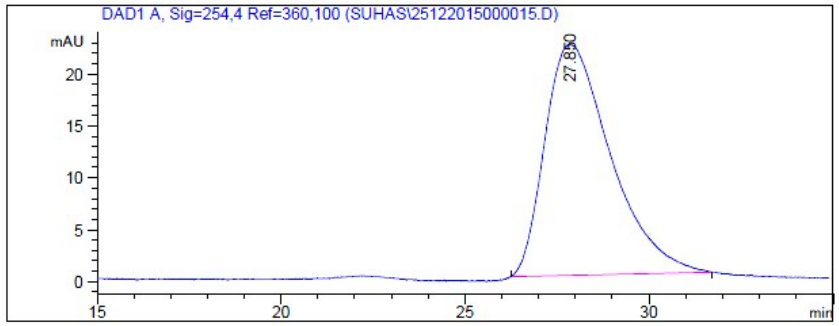
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  20.541 BB    1.1132 2195.75366   23.43390  47.2603
   2  27.822 BB    1.4927 2450.32886   19.30606  52.7397
```

HPLC Chromatogram of Compound **(±)-5b**

Sample Info : 874_cu_odh
,OD-H Column,30 % iPr/Hexane 1.0 ml/min flow rate, 254 nm

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                      Area Percent Report
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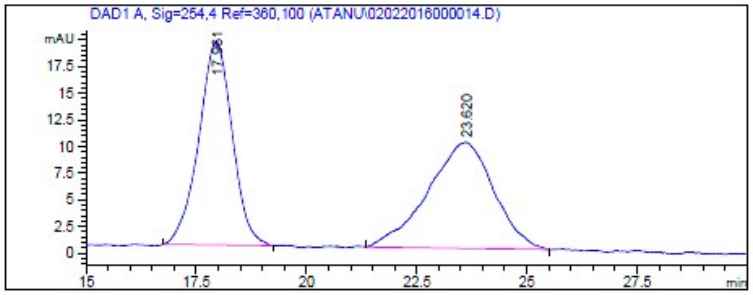
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  27.850 BB    1.4642 2769.36743   22.24870 100.0000
```

HPLC Chromatogram of Compound **5b**

Sample Info : AH_186 RAC
CHIRALCEL AD-H Column,25 % iPr/Hexane 1.0 ml/min flow rate, 254nm



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

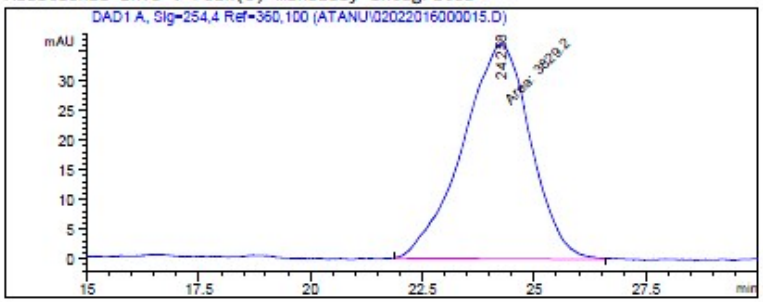
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.961	BB	0.6446	1008.68311	19.08668	49.3327
2	23.620	BB	1.2240	1035.97302	9.98836	50.6673

HPLC Chromatogram of Compound **(±)-5d**

Sample Info : AH_185 CHIRAL
CHIRALCEL AD-H Column,25 % iPr/Hexane 1.0 ml/min flow rate, 254nm

Additional Info : Peak(s) manually integrated



Area Percent Report

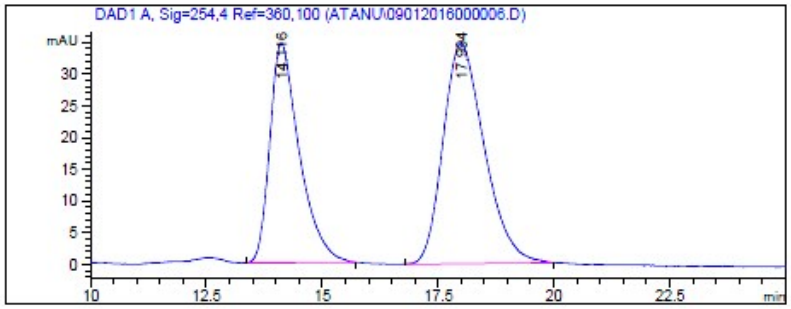
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.258	MM	1.7619	3829.20386	36.22127	100.0000

HPLC Chromatogram of Compound **5d**

Sample Info : AH_188_REC
,AD-H Column,25 % iPr/Hexane 1.0 ml/min flow rate, 230 nm

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                          Area Percent Report
================================================================================

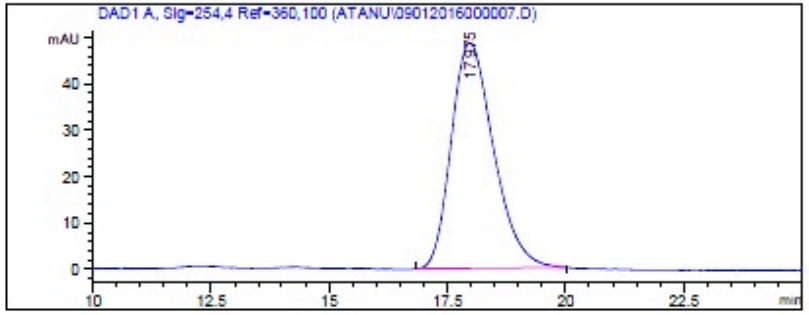
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.116 BB    0.6450 1537.12256   34.55408  41.7017
   2  17.994 BB    0.8183 2148.86841   34.70823  58.2983
```

HPLC Chromatogram of Compound **(±)-5e**

Sample Info : AH_187
,AD-H Column,25 % iPr/Hexane 1.0 ml/min flow rate, 230 nm

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================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

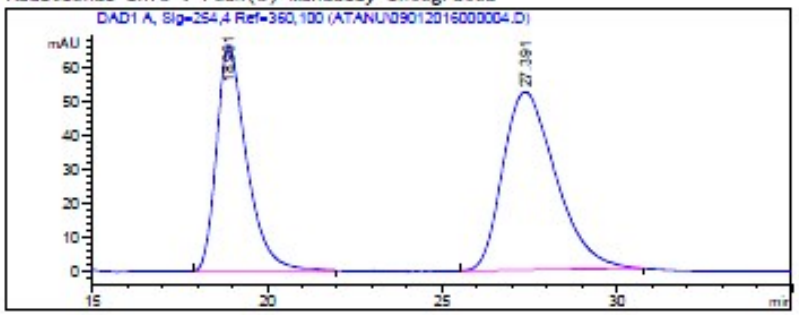

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  17.975 BB    0.8970 3027.78125   48.58718 100.0000
```

HPLC Chromatogram of Compound **5e**

Sample Info : AH_191_REC
,AD-H Column,20 % iPr/Hexane 1.0 ml/min flow rate, 230 nm

Additional Info : Peak(s) manually integrated



Area Percent Report

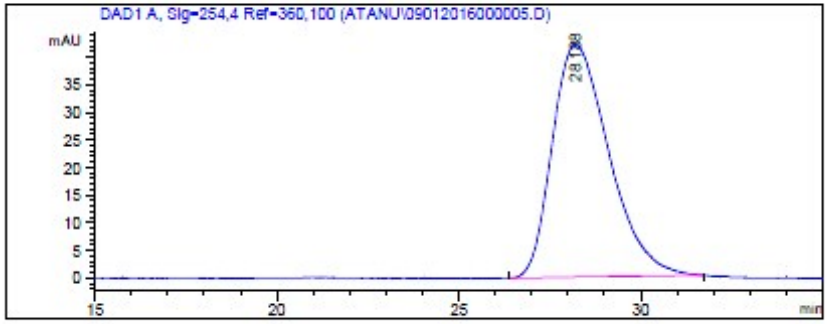
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.901	BB	0.8796	3942.10107	65.74316	41.7192
2	27.391	BB	1.2555	5507.02686	52.34144	58.2808

HPLC Chromatogram of Compound **(±)-5f**

Sample Info : AH_190
,AD-H Column,20 % iPr/Hexane 1.0 ml/min flow rate, 230 nm



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

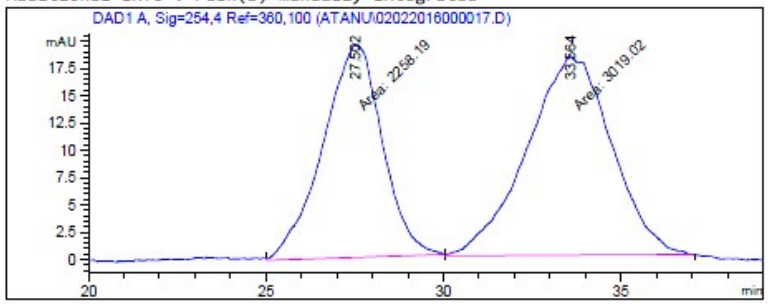
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.188	BB	1.2765	4560.57715	42.14491	100.0000

HPLC Chromatogram of Compound **5f**

Sample Info : AH_205 RAC1
CHIRALCEL AD-H Column,30 % iPr/Hexane 1.0 ml/min flow rate, 254nm

Additional Info : Peak(s) manually integrated



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Area Percent Report

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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

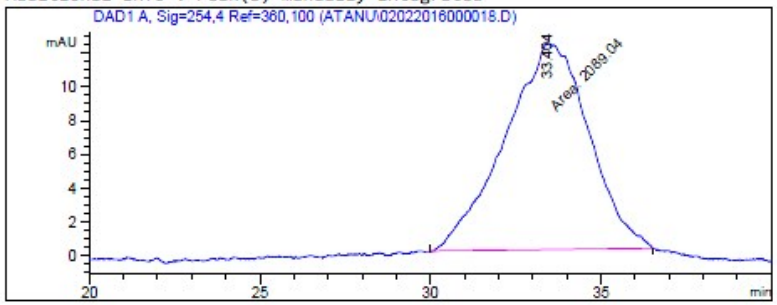
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.502	MM T	1.9411	2258.19263	19.38975	42.7914
2	33.564	MM T	2.9956	3019.02051	18.18397	57.2086

HPLC Chromatogram of Compound **(±)-5g**

Sample Info : AH_207 CHI
CHIRALCEL AD-H Column,30 % iPr/Hexane 1.0 ml/min flow rate, 254nm

Additional Info : Peak(s) manually integrated



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Area Percent Report

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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

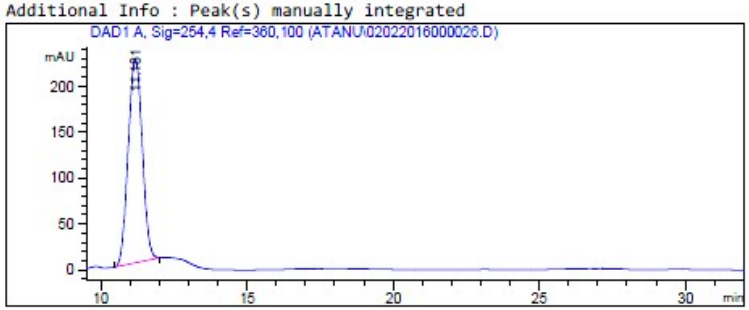
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	33.404	MM	2.8591	2089.04199	12.17757	100.0000

HPLC Chromatogram of Compound **5g**

Sample Info : AH_220S1.1
CHIRALCEL AD-H Column,30 % iPr/Hexane 1.0 ml/min flow rate, 254nm

Additional Info : Peak(s) manually integrated



Area Percent Report

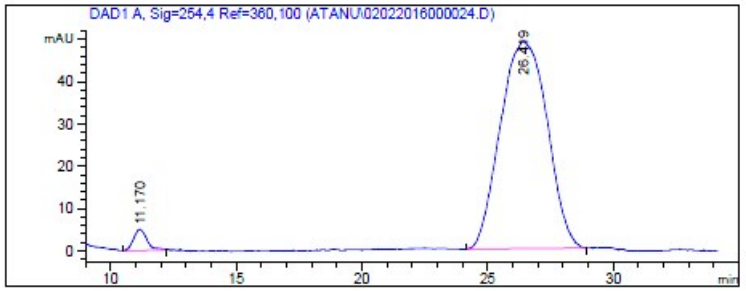
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.181	BB	0.5316	7431.44971	222.54340	100.0000

HPLC Chromatogram of Compound (*S*)-**5u** (reported enantiomer)

Sample Info : AH_209R
CHIRALCEL AD-H Column,30 % iPr/Hexane 1.0 ml/min flow rate, 254nm



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

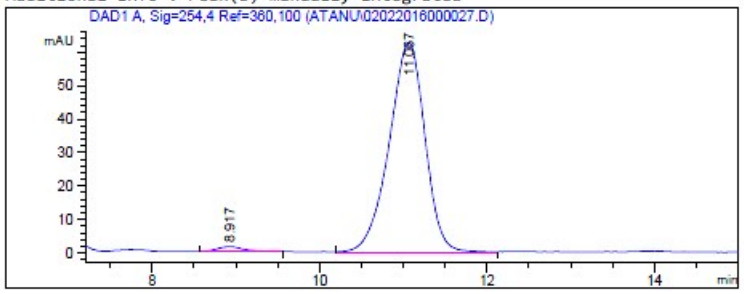
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.170	BB	0.4330	178.62865	4.95025	2.7392
2	26.419	BB	1.5261	6342.62354	49.00185	97.2608

HPLC Chromatogram of Compound (*R*)-**5u** (other enantiomer)

Sample Info : AH_221R
CHIRALCEL AD-H Column,30 % iPr/Hexane 1.0 ml/min flow rate, 254nm

Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

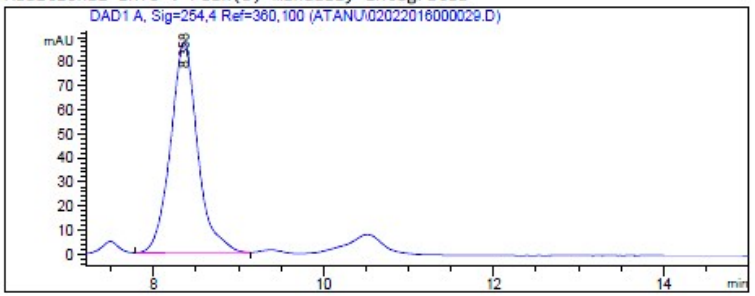
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.917	BB	0.2990	34.84306	1.45722	1.8440
2	11.067	BB	0.4557	1854.67712	62.58494	98.1560

HPLC Chromatogram of Compound (*R*)-**5v** (reported enantiomer)

Sample Info : AH_210S
CHIRALCEL AD-H Column,30 % iPr/Hexane 1.0 ml/min flow rate, 254nm

Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

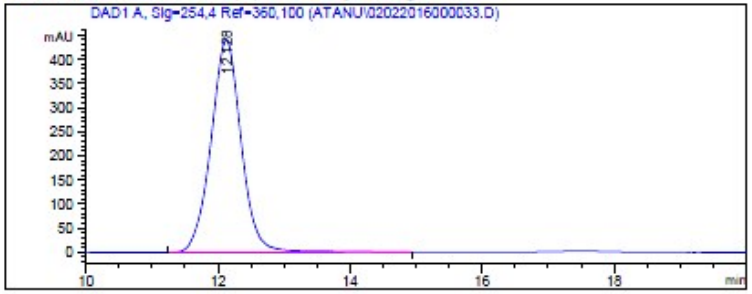
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.358	BB	0.3284	1928.62915	87.08982	100.0000

HPLC Chromatogram of Compound (*S*)-**5v** (other enantiomer)

Sample Info : AH_214S
CHIRALCEL AD-H Column,30 % iPr/Hexane 1.0 ml/min flow rate, 254nm

Additional Info : Peak(s) manually integrated



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Area Percent Report
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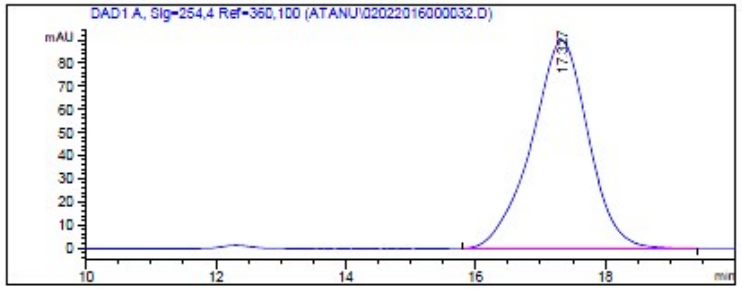
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.128	BB	0.4858	1.40268e4	440.15732	100.0000

HPLC Chromatogram of Compound (*S*)-**5w** (reported enantiomer)

Sample Info : AH_216R
CHIRALCEL AD-H Column,30 % iPr/Hexane 1.0 ml/min flow rate, 254nm



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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

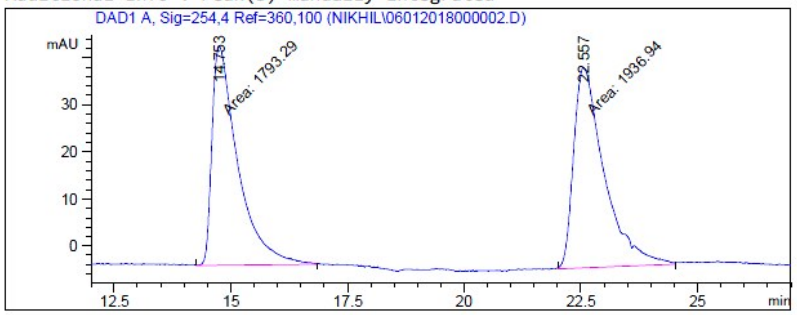
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.327	BB	0.8952	5466.40674	89.92130	100.0000

HPLC Chromatogram of Compound (*R*)-**5w** (other enantiomer)

Sample Info : DEPROT-UNSBS-AZI-RAC
CHIRALPAK-IA-3, 20% IPA/HEXANE 1.0 ml/min flow rate, 254 nm

Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

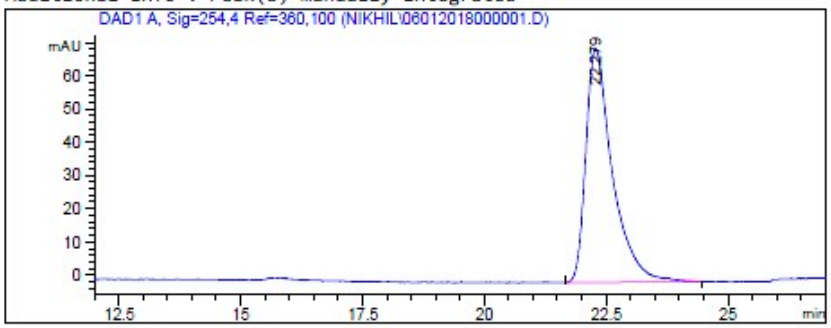
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.753	MM	0.6429	1793.29297	46.49168	48.0745
2	22.557	MM	0.7602	1936.94287	42.46735	51.9255

HPLC Chromatogram of Compound **(±)-7b**

Sample Info : DEPROT-UNSBS-AZI-L
CHIRALPAK-IA-3, 20% IPA/HEXANE 1.0 ml/min flow rate, 254 nm

Additional Info : Peak(s) manually integrated



Area Percent Report

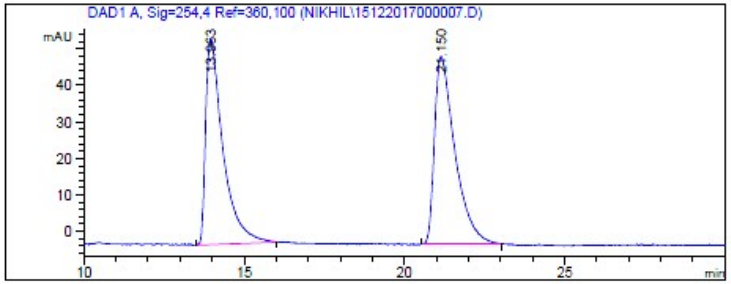
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.279	BB	0.5529	2675.72021	70.48747	100.0000

HPLC Chromatogram of Compound **7b**

Sample Info : DEPROT-5-METHYL-RAC
CHIRALPAK_IA3_250MM, 20% IPA/HEXANE 1.0 ml/min flow rate, 254 nm



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

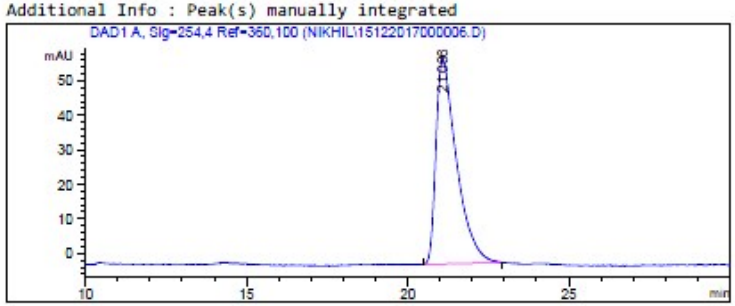
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.963	BB	0.5175	2108.31909	56.21965	48.0367
2	21.150	BB	0.6311	2280.65967	51.45952	51.9633

HPLC Chromatogram of Compound **(±)-7c**

Sample Info : DEPROT-5-METHYL-L
CHIRALPAK_IA3_250MM, 20% IPA/HEXANE 1.0 ml/min flow rate, 254 nm

Additional Info : Peak(s) manually integrated



Area Percent Report

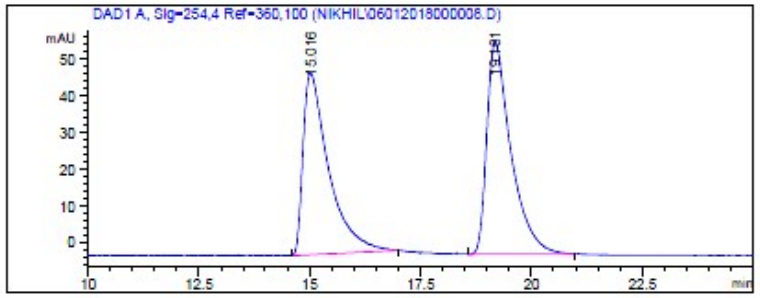
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.086	BB	0.6201	2638.87427	59.42680	100.0000

HPLC Chromatogram of Compound **7c**

Sample Info : 5-Cl-DEPROT-UNSBS-AZI-RAC
CHIRALPAK-IA-3, 20% IPA/HEXANE 1.0 ml/min flow rate, 254 nm

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                      Area Percent Report
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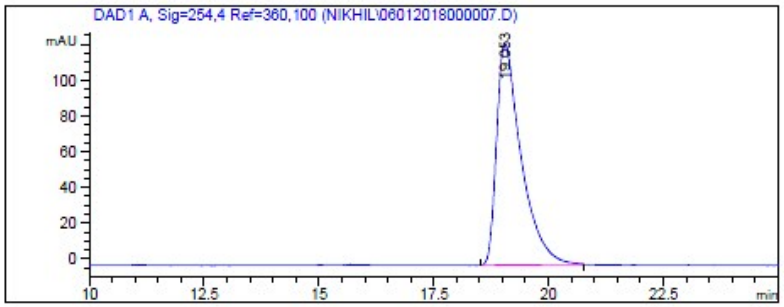
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.016 BB    0.5730 1991.44397   49.53590  47.5660
   2  19.181 BB    0.5443 2195.25635   57.90427  52.4340
```

HPLC Chromatogram of Compound **(±)-7d**

Sample Info : 5-Cl-DEPROT-UNSBS-AZI-L
CHIRALPAK-IA-3, 20% IPA/HEXANE 1.0 ml/min flow rate, 254 nm

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                      Area Percent Report
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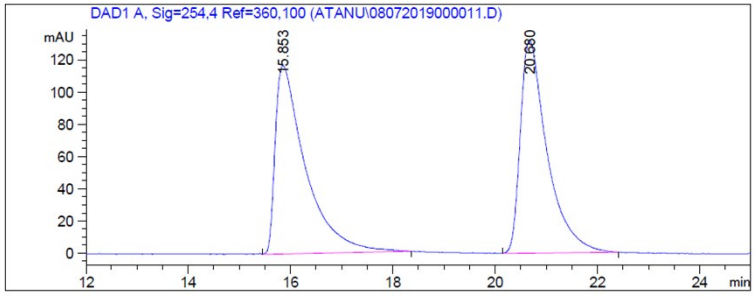
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  19.053 BB    0.5384 4604.67236  123.67815 100.0000
```

HPLC Chromatogram of Compound **7d**

CHIRALPAK IA-3; 250 MM
IPA/HEXANE : 20/80
FLOW RATE: 1.0 mL/ min
254 nm

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                      Area Percent Report
=====================================================================

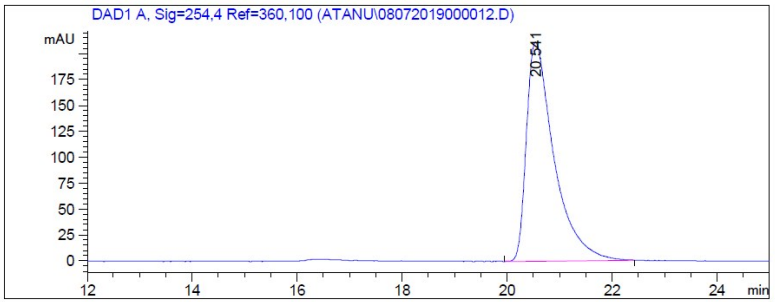
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.853 BB    0.5756 4803.17676  116.84882  49.9378
   2  20.680 BB    0.5227 4815.14551  132.28288  50.0622
```

HPLC Chromatogram of Compound **(±)-7e**

CHIRALPAK IA-3; 250 MM
IPA/HEXANE : 20/80
FLOW RATE: 1.0 mL/ min
254 nm




```
=====================================================================
                      Area Percent Report
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Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  20.541 BB    0.5359 7716.73877  211.37752 100.0000
```

HPLC Chromatogram of Compound **7e**