

Supplementary Materials

Diastereoselective Three-component 1,3-Dipolar Cycloaddition to Access Functionalized β -Tetrahydrocarboline and Tetrahydroisoquinoline fused Spirooxindoles

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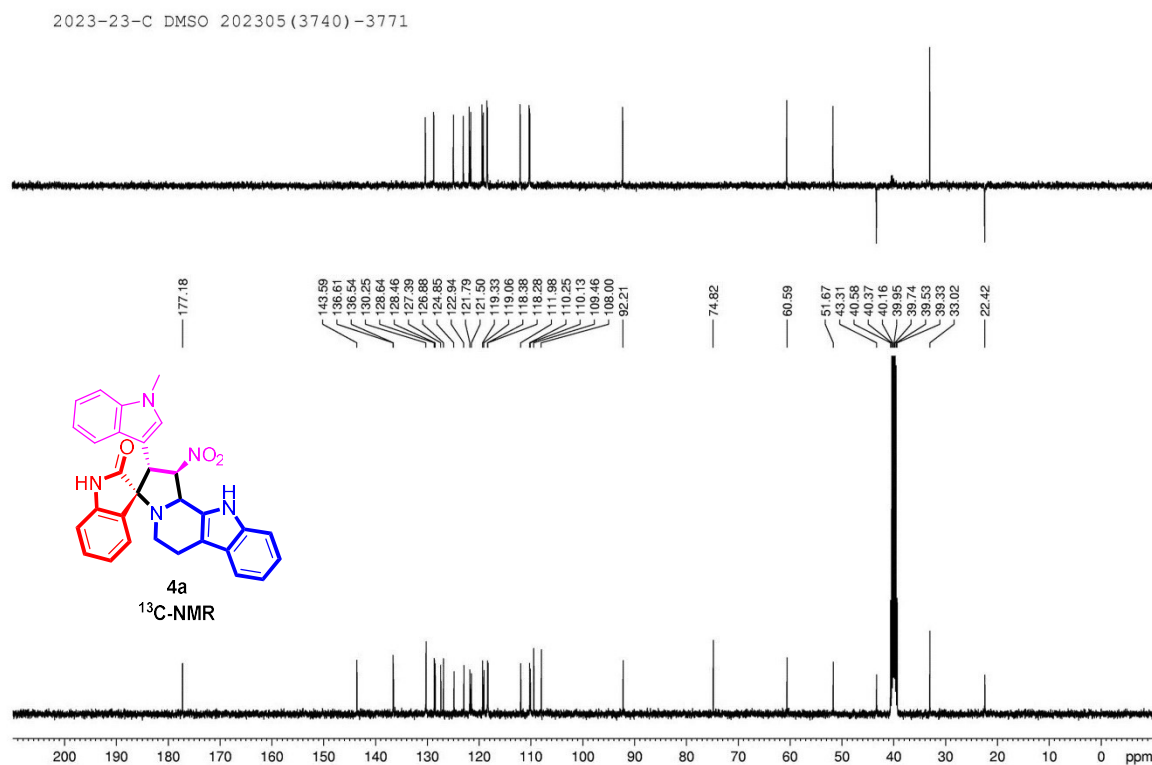
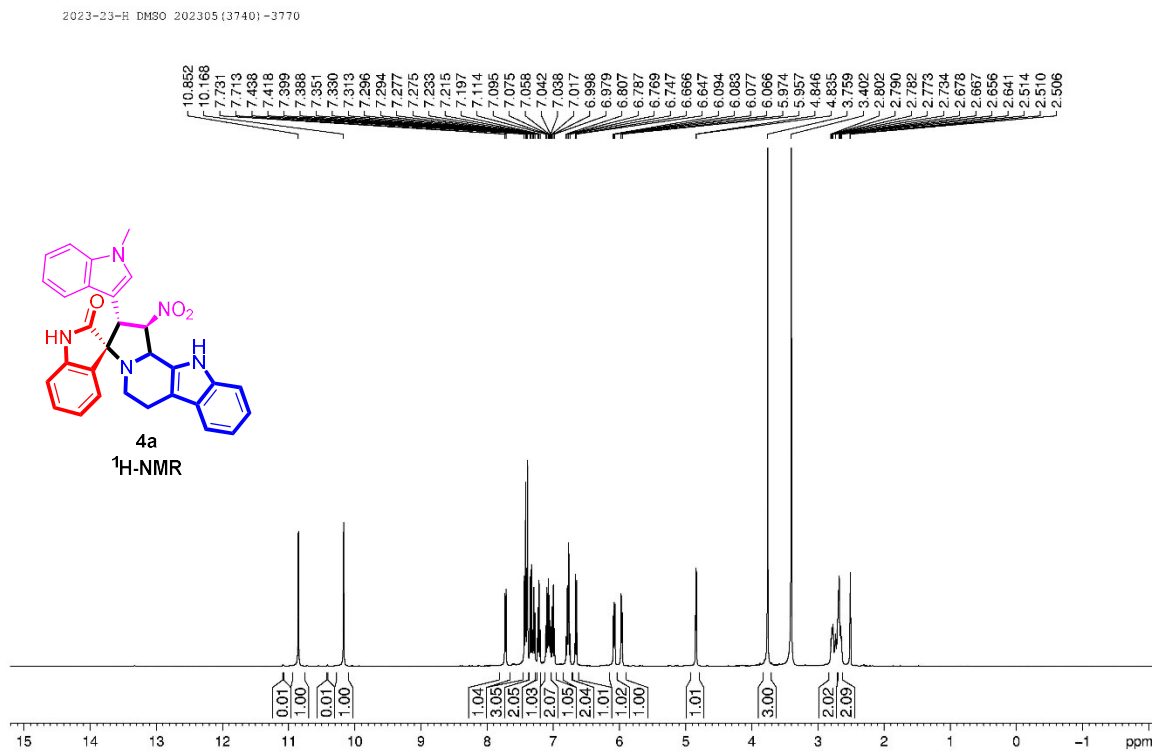
Donghua Hu (hudonghua8888@126.com)

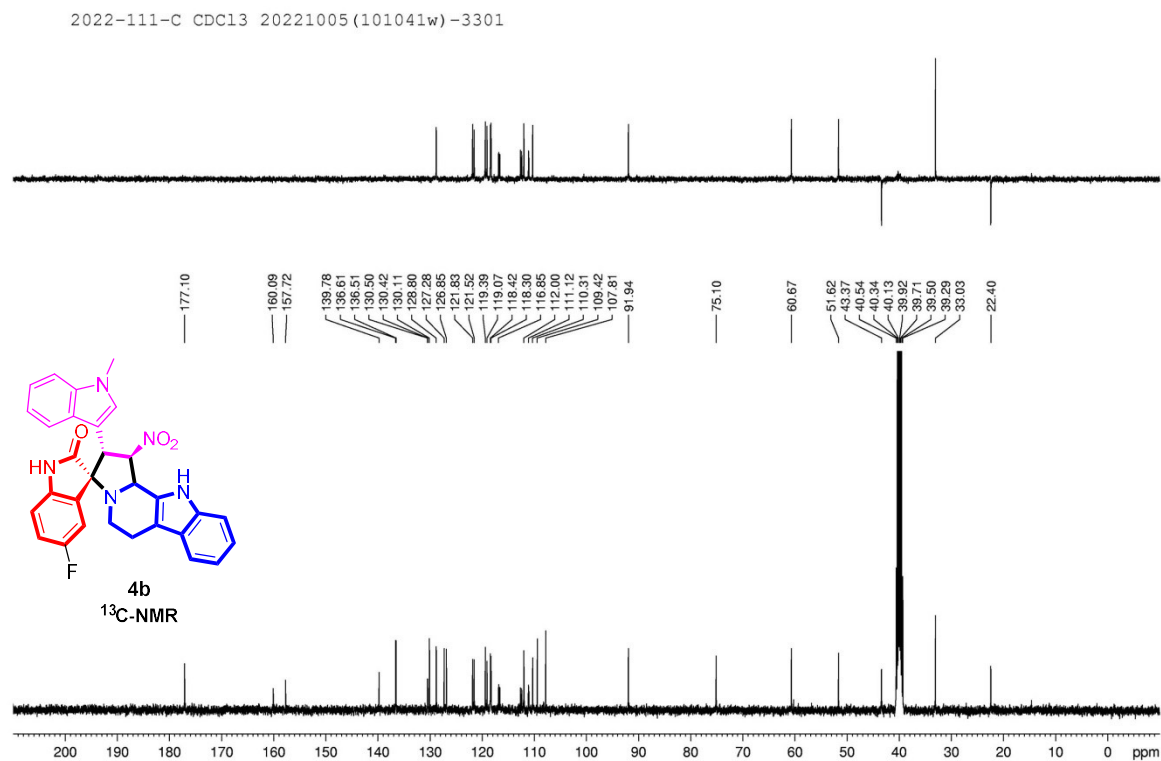
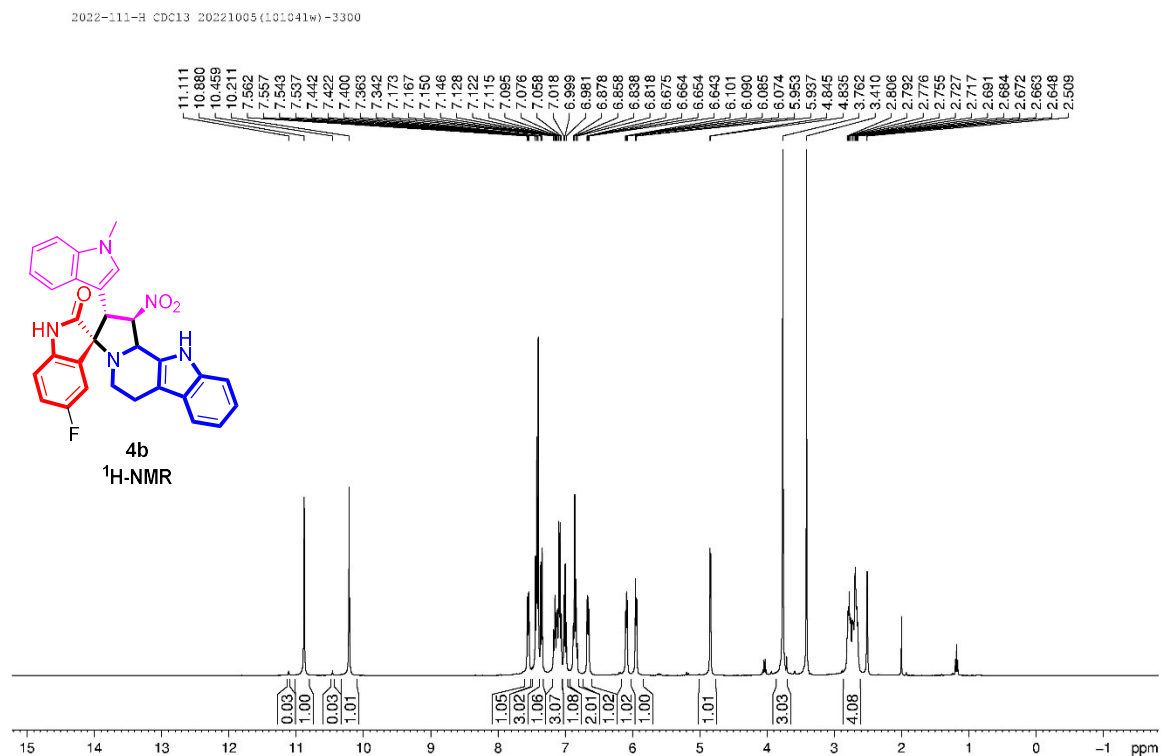
Jianwei Dong (jwdongyn@mail.qjnu.edu.cn)

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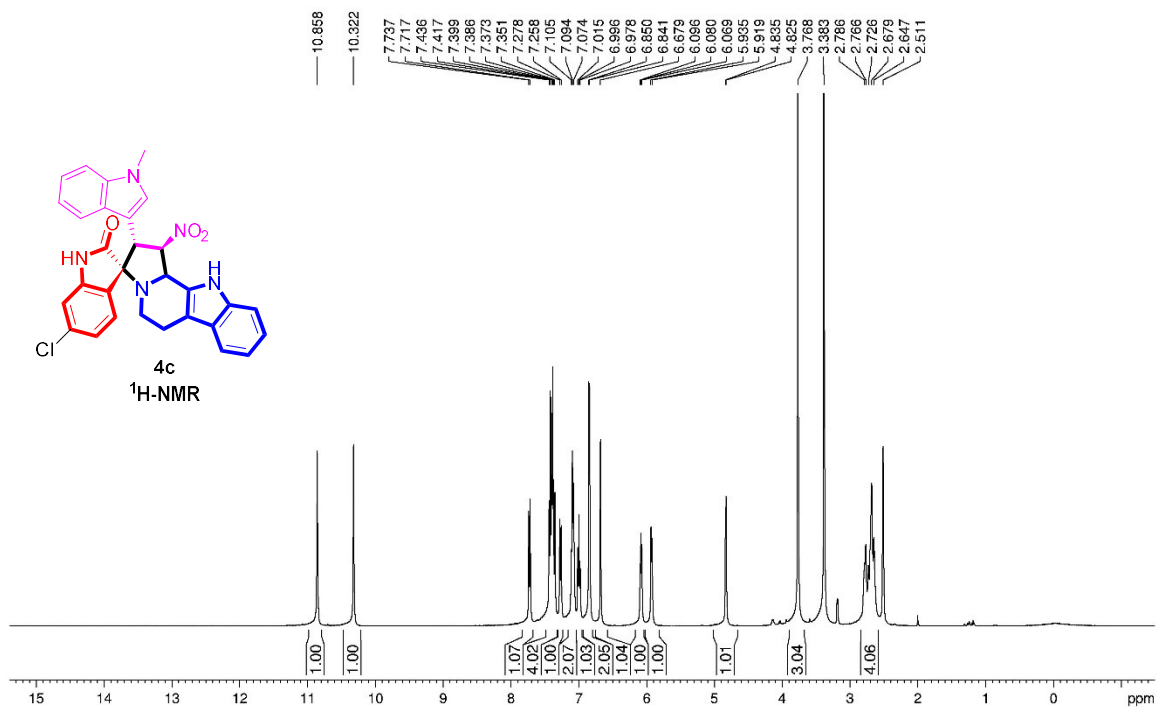
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1. ^1H and ^{13}C NMR spectras for compounds 4

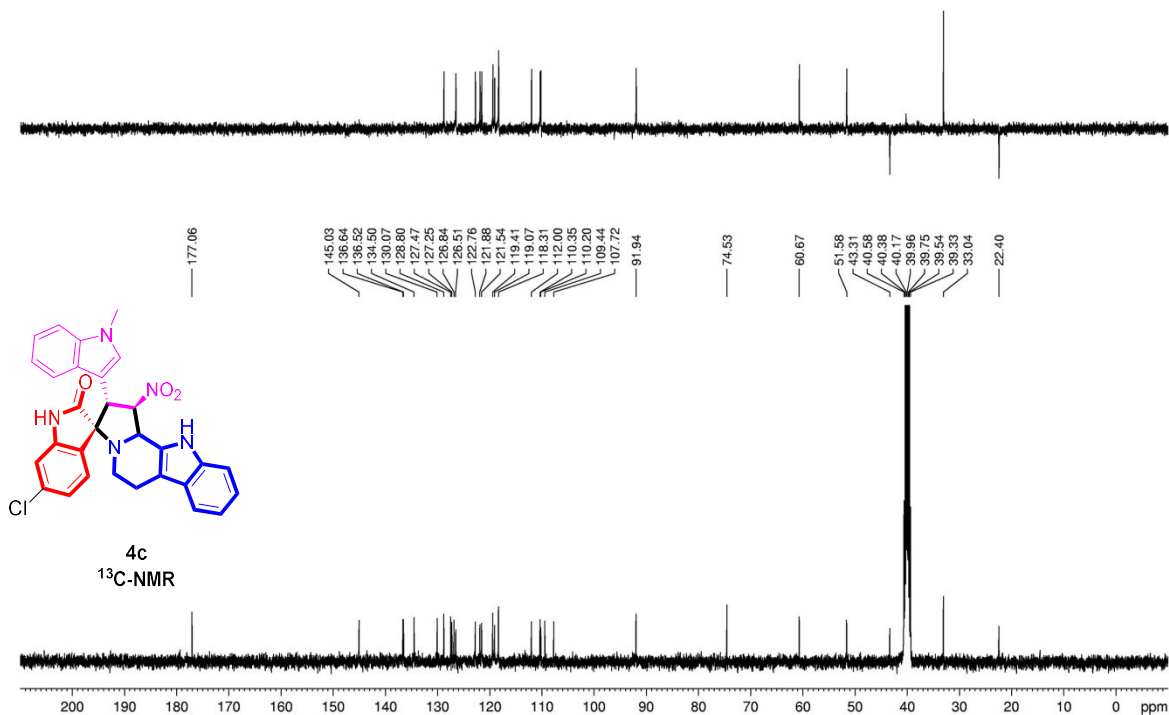




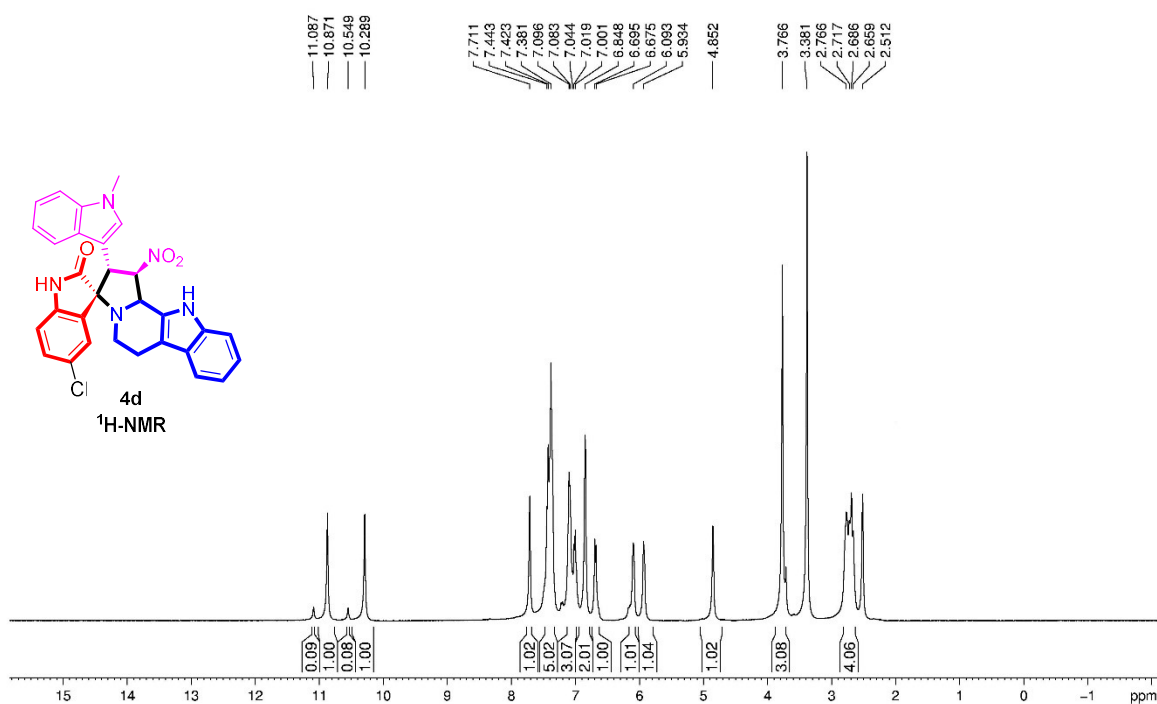
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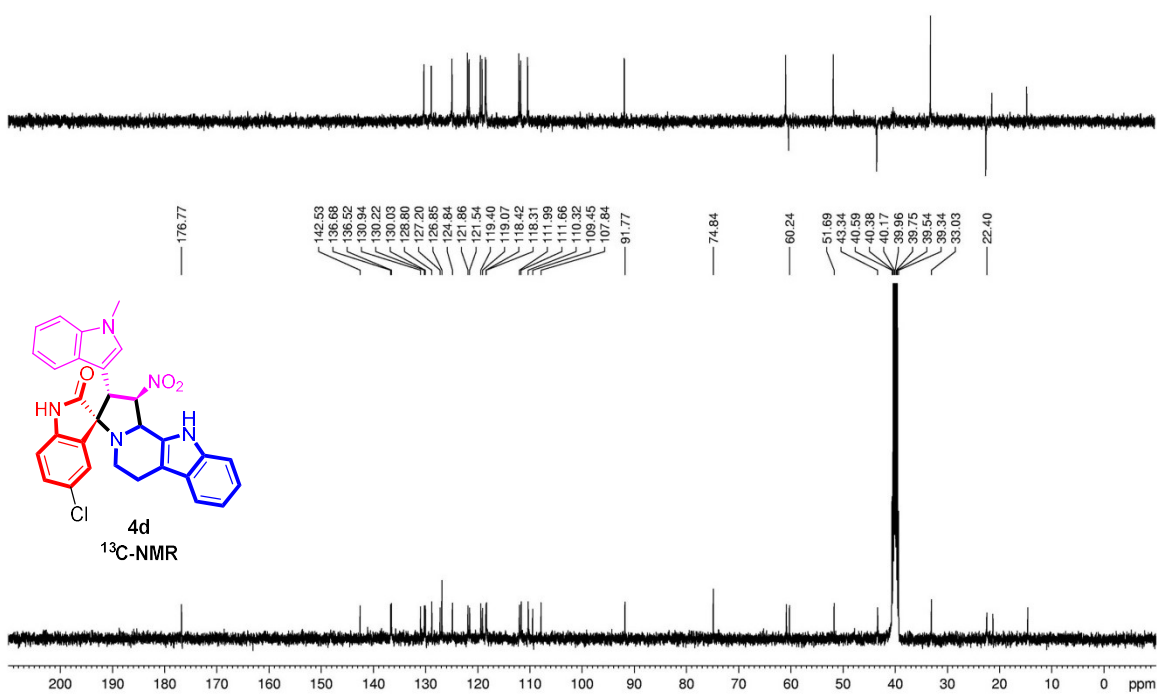
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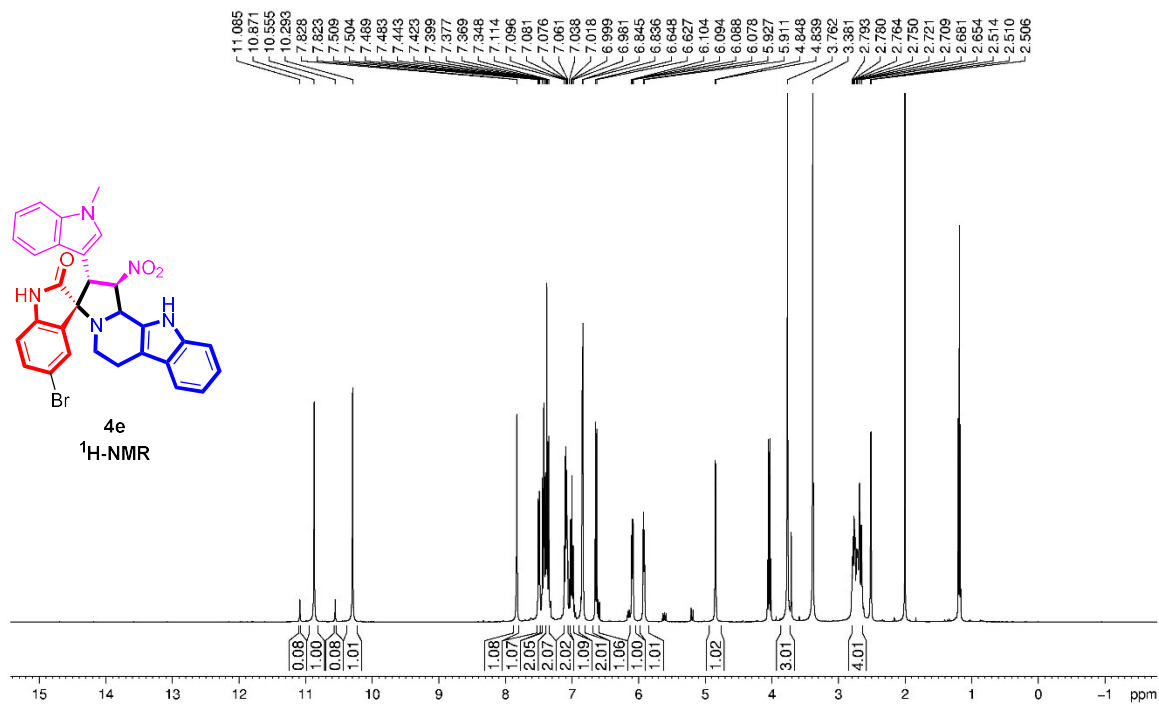
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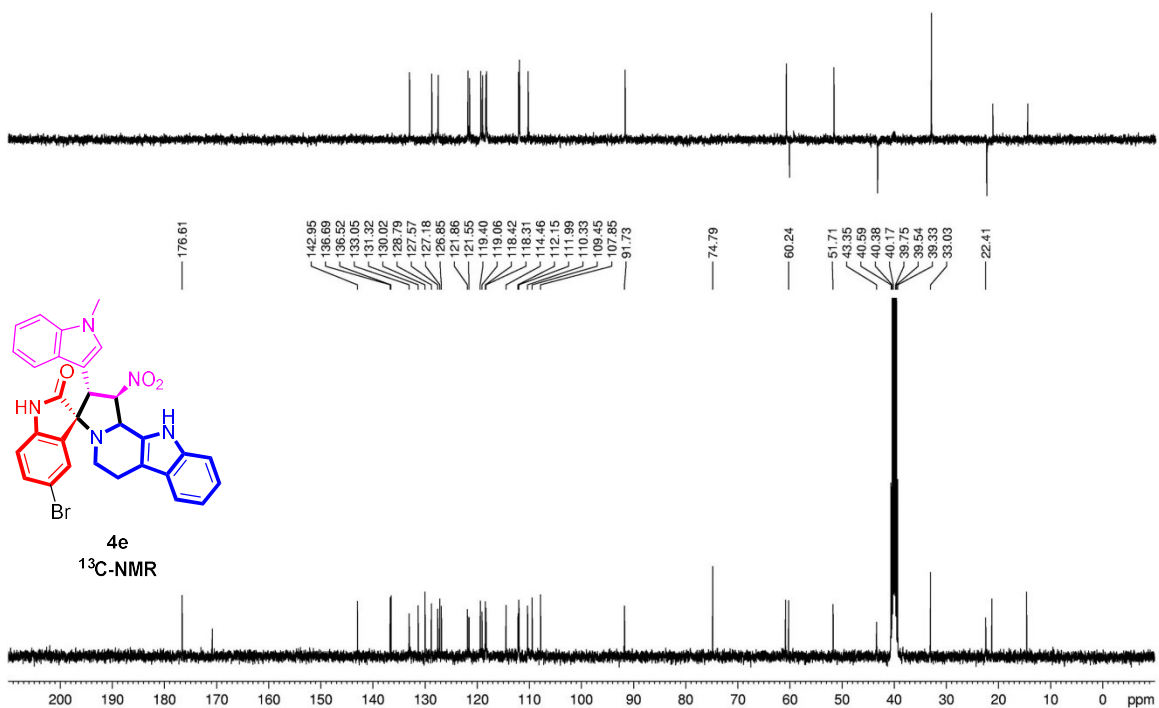
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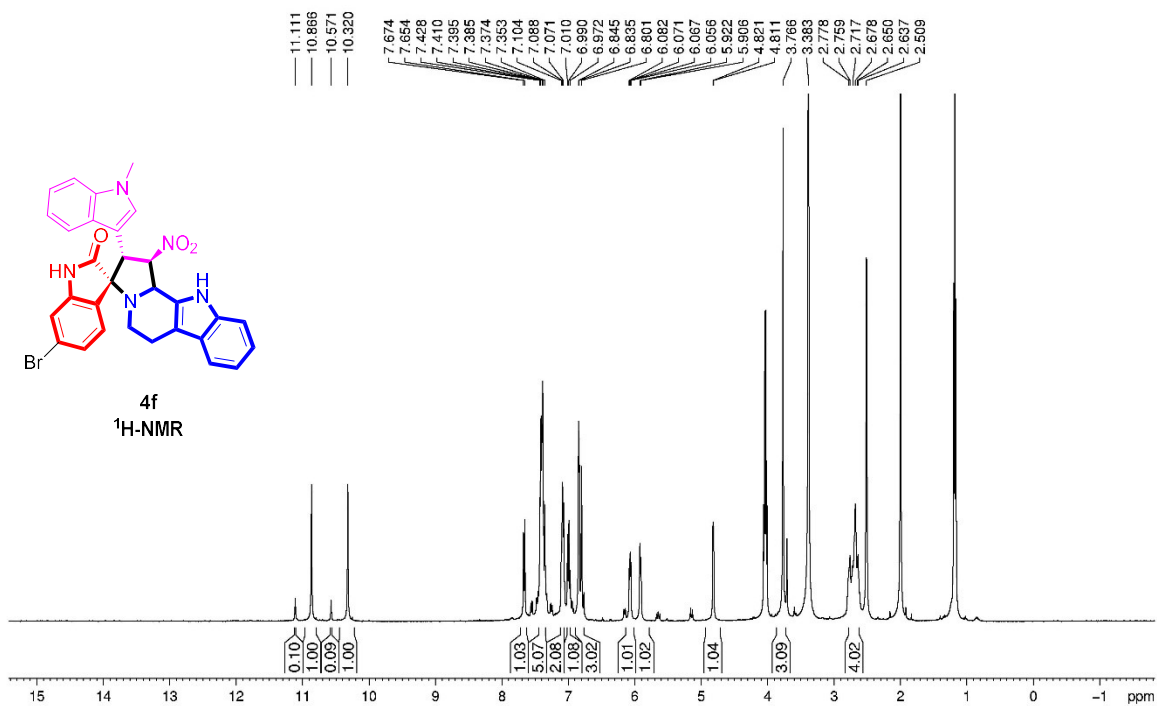
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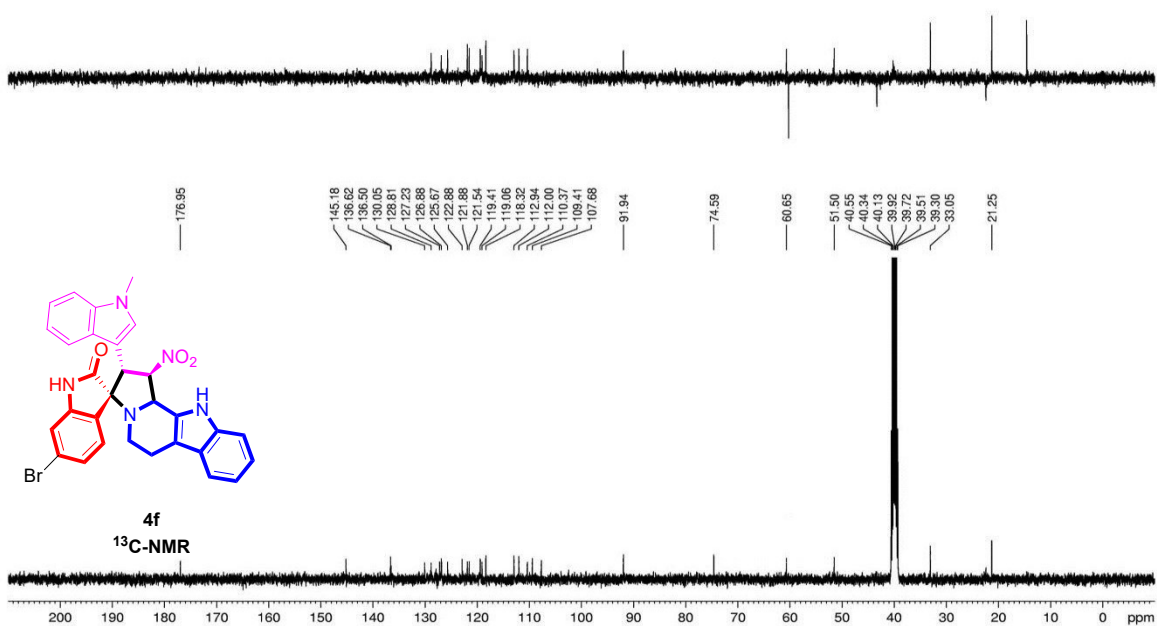
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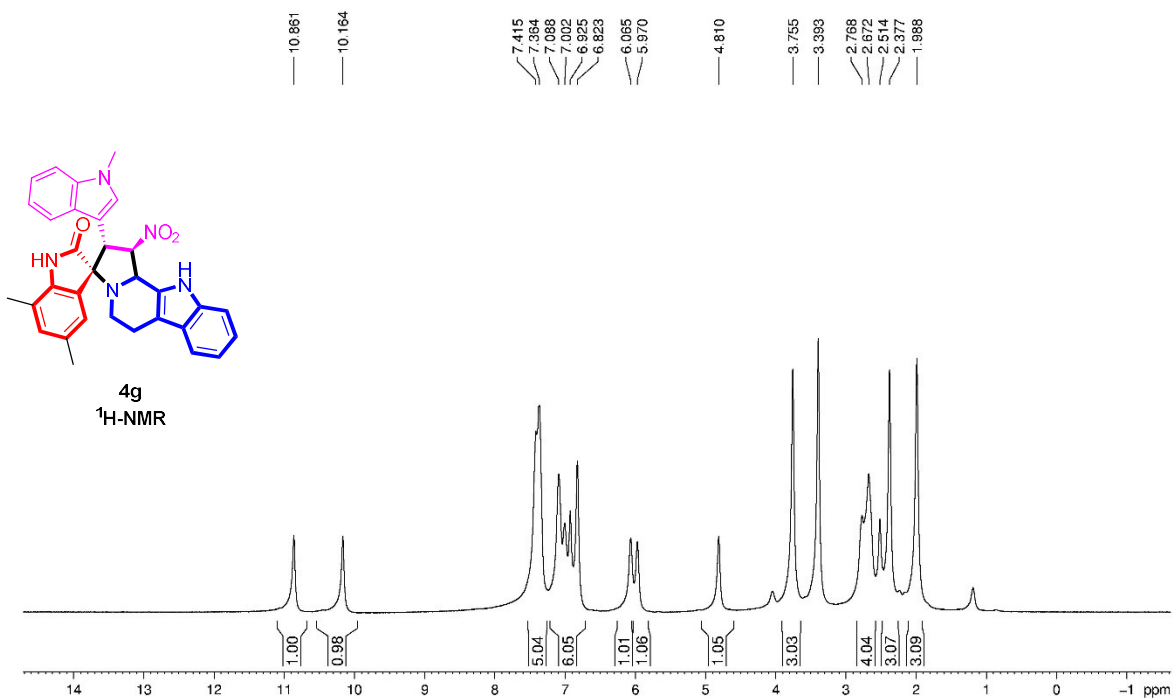
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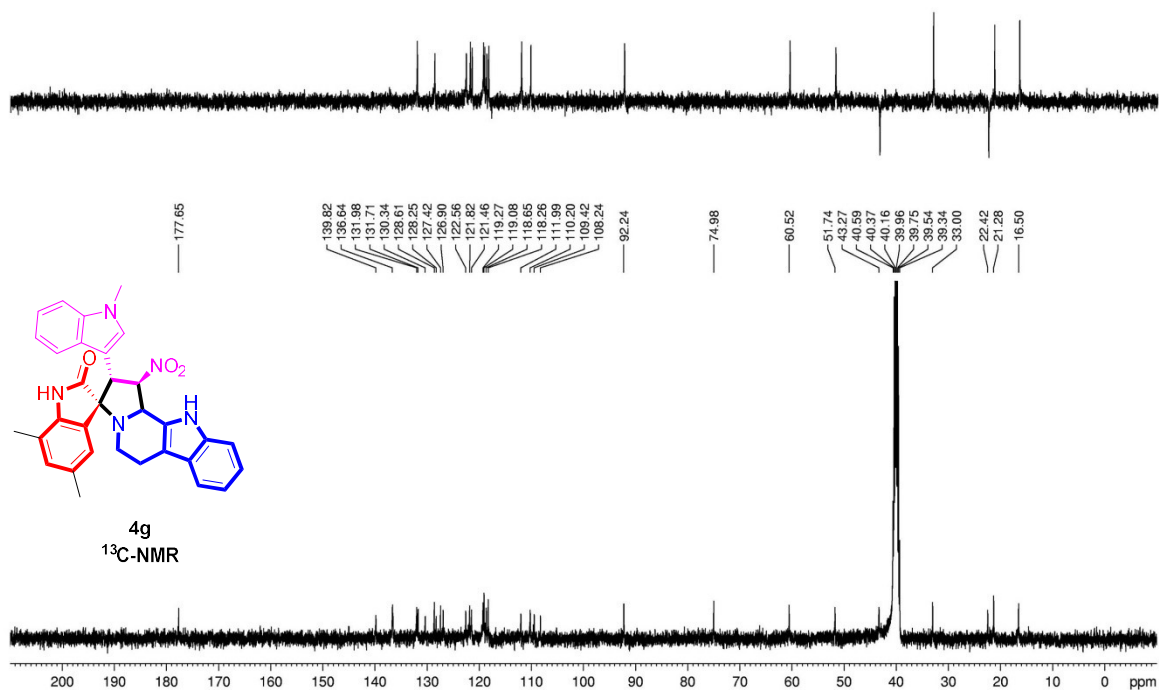
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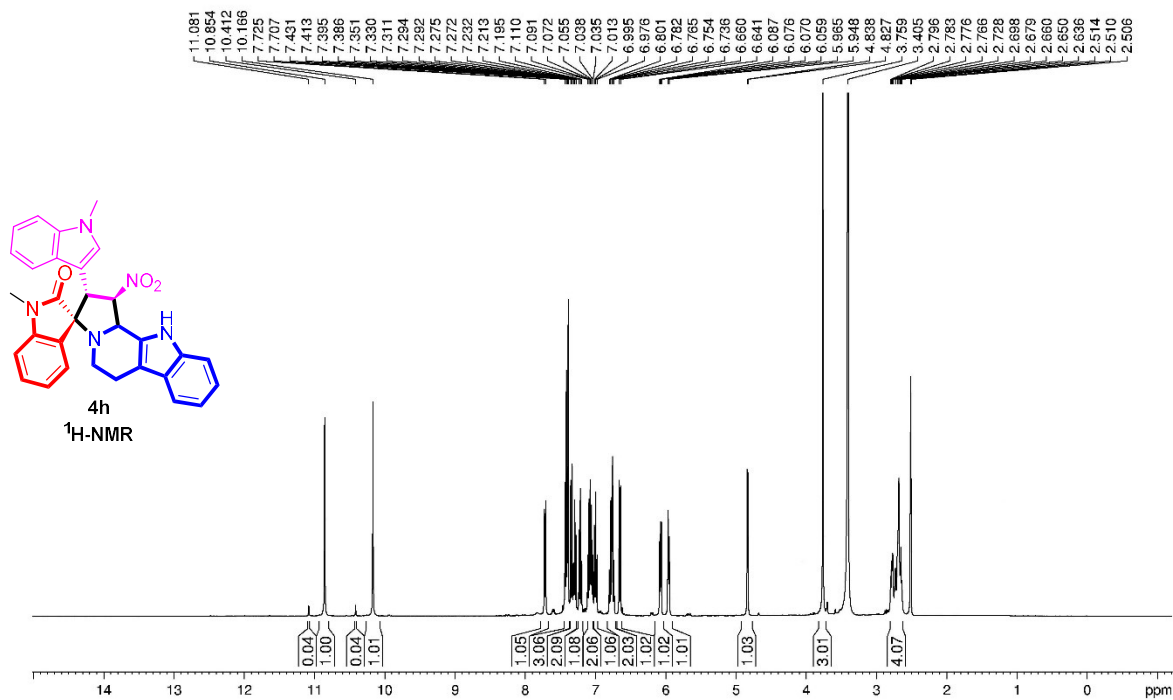
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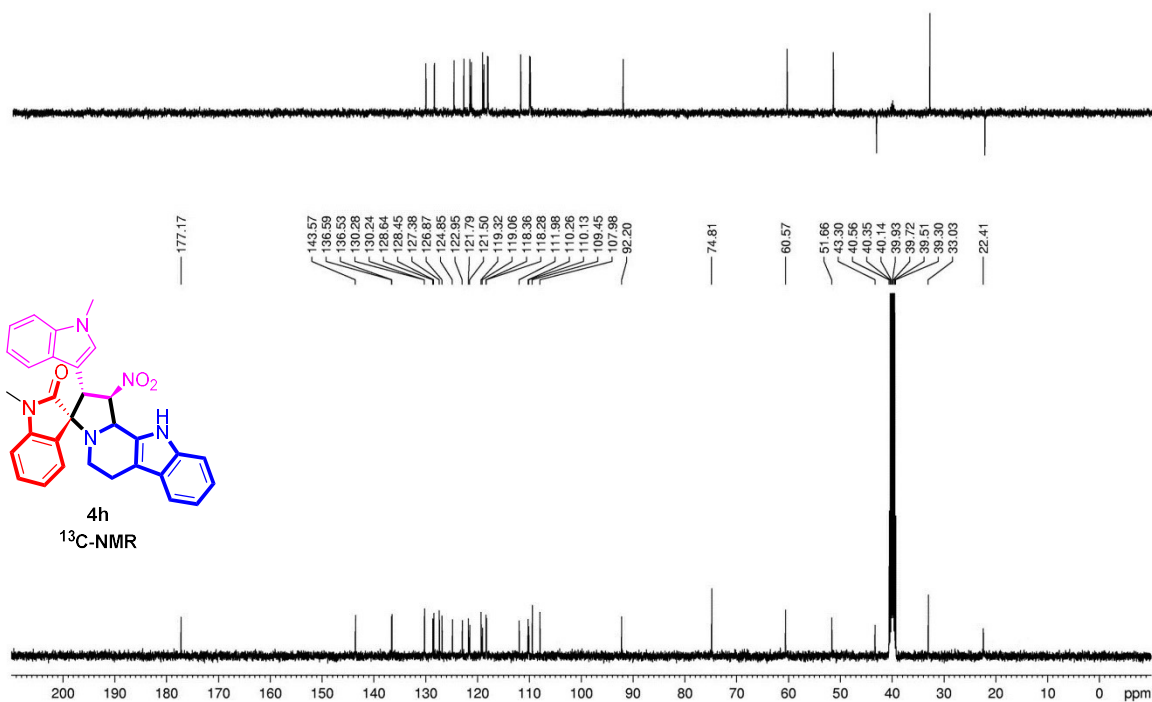
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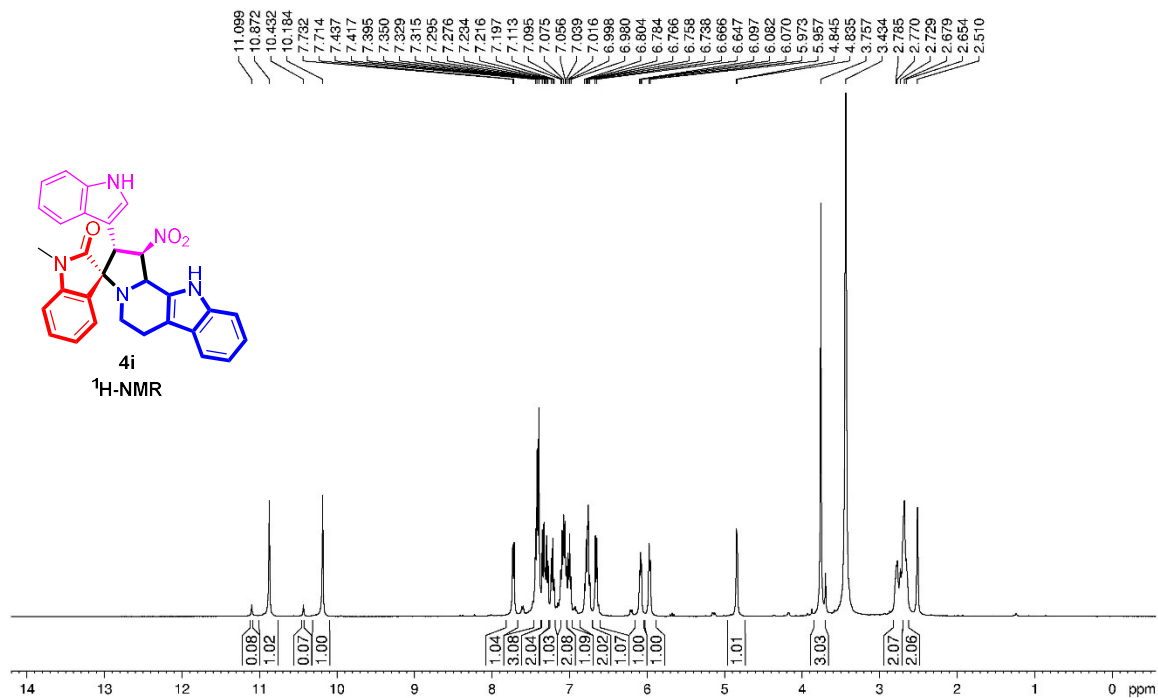
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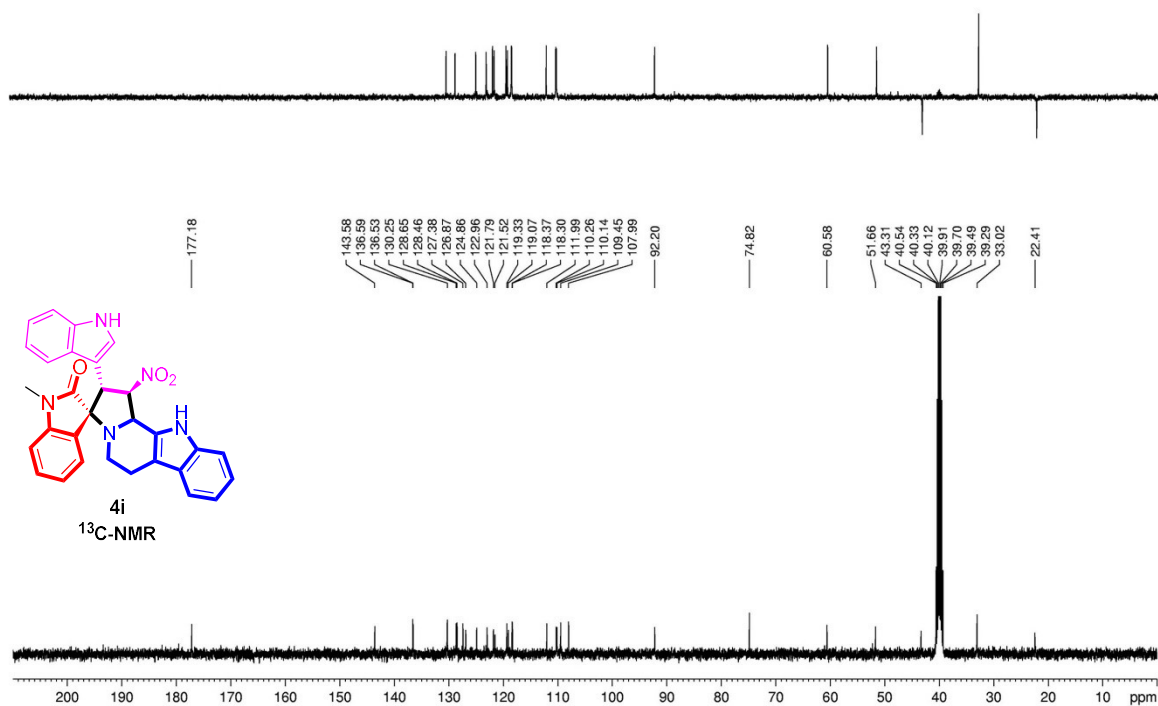
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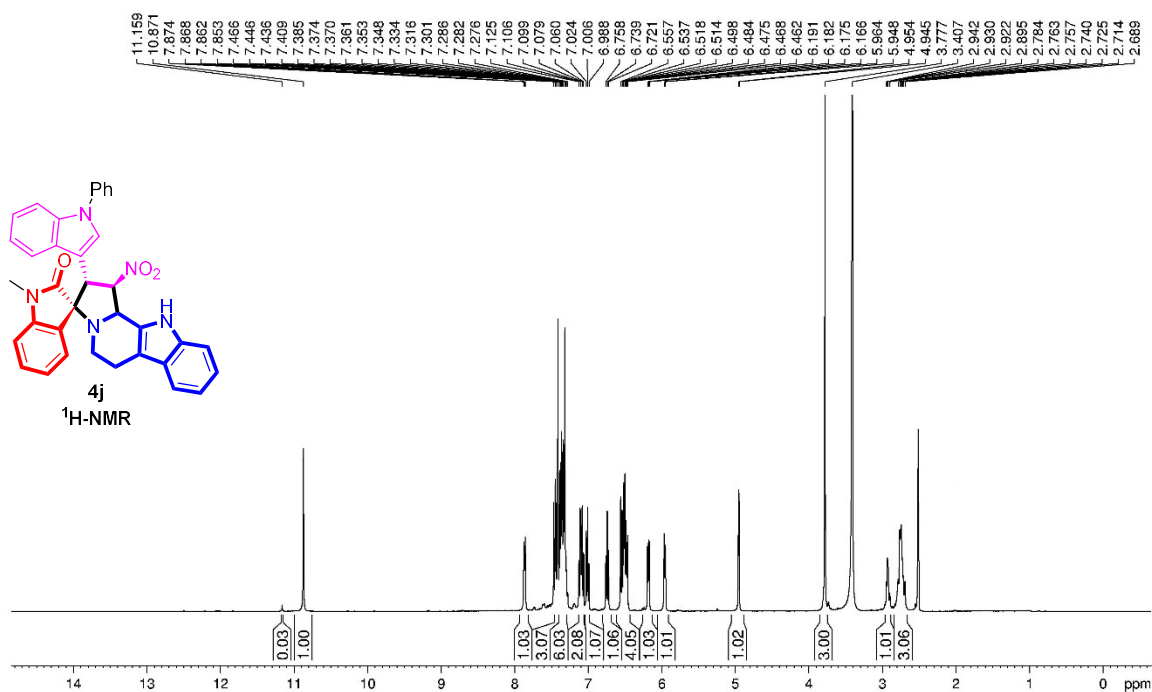
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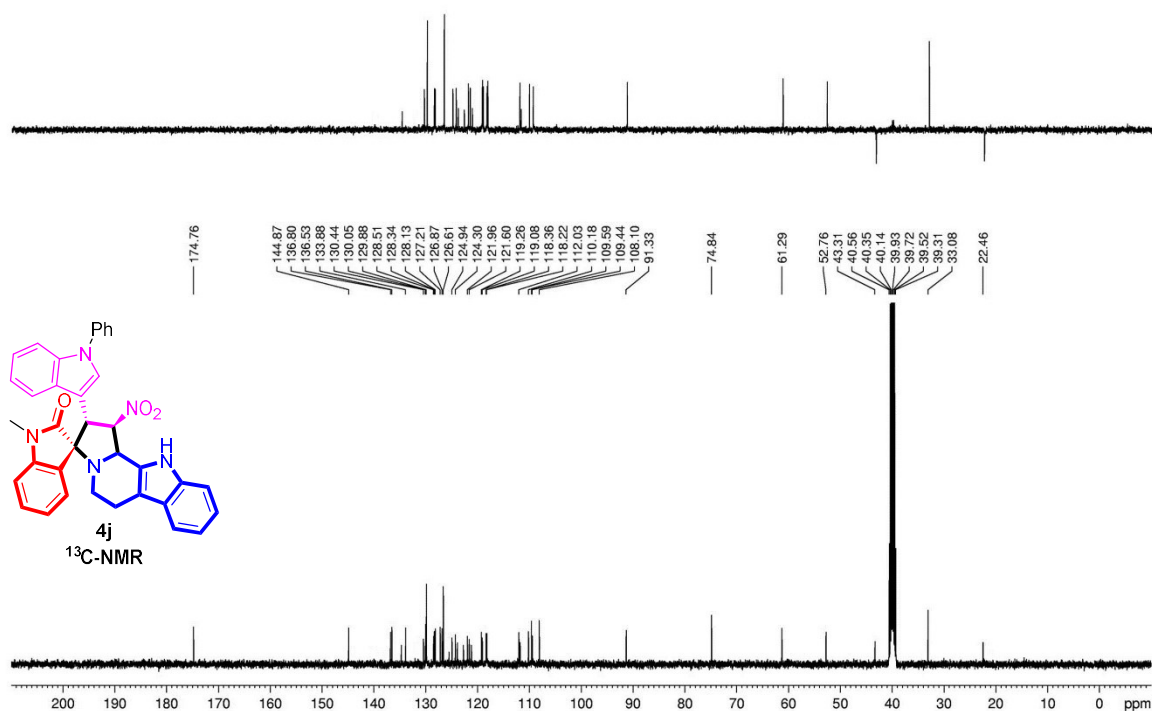
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2023-40-B DMSO 202308 (3970) -3990



2023-40-C DMSO 202308 (3970) -3991



4k
¹H-NMR

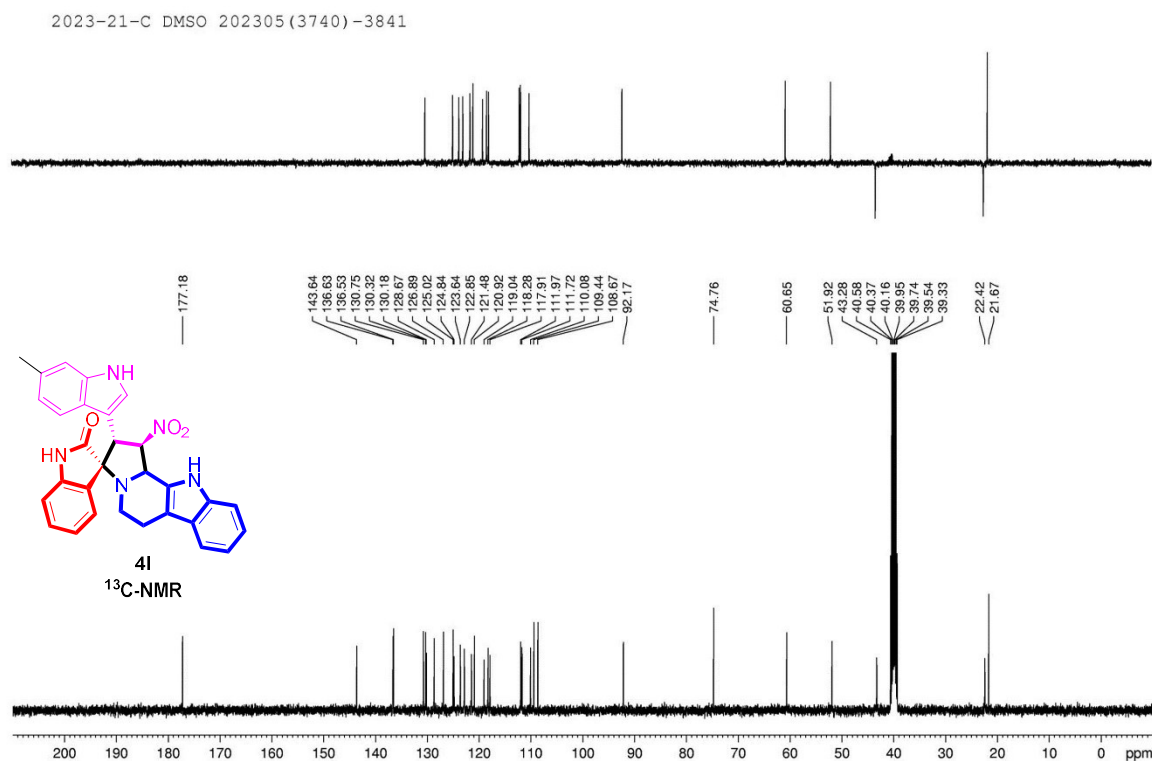
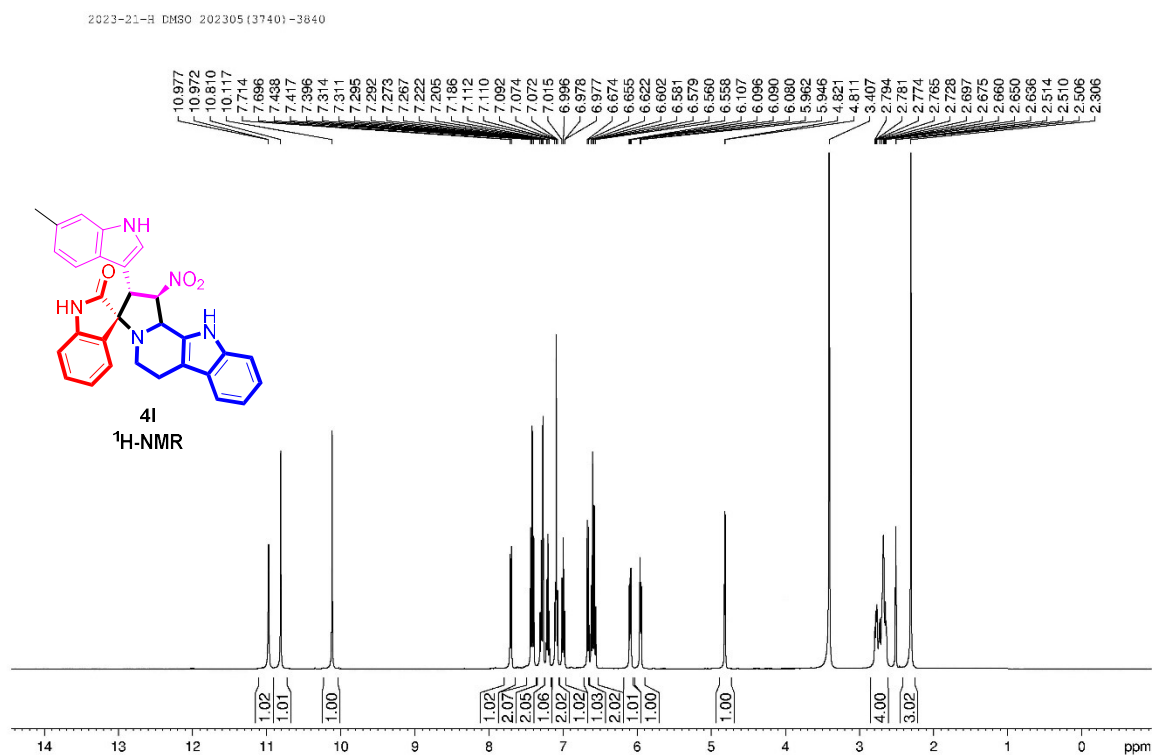
Chemical structure of **4k** is shown above the spectrum. The spectrum displays peaks corresponding to the structure, with chemical shifts (ppm) and integration values indicated.

Chemical shifts (ppm): 10.993, 10.816, 10.117, 7.738, 7.720, 7.444, 7.424, 7.418, 7.398, 7.382, 7.326, 7.319, 7.300, 7.298, 7.266, 7.237, 7.219, 7.189, 7.188, 7.113, 7.094, 7.076, 7.016, 6.997, 6.979, 6.977, 6.917, 6.876, 6.873, 6.854, 6.374, 6.125, 6.115, 6.109, 6.096, 5.955, 5.958, 4.825, 4.815, 3.407, 2.808, 2.795, 2.786, 2.777, 2.732, 2.681, 2.663, 2.653, 2.514, 2.510, 2.506, 2.124.

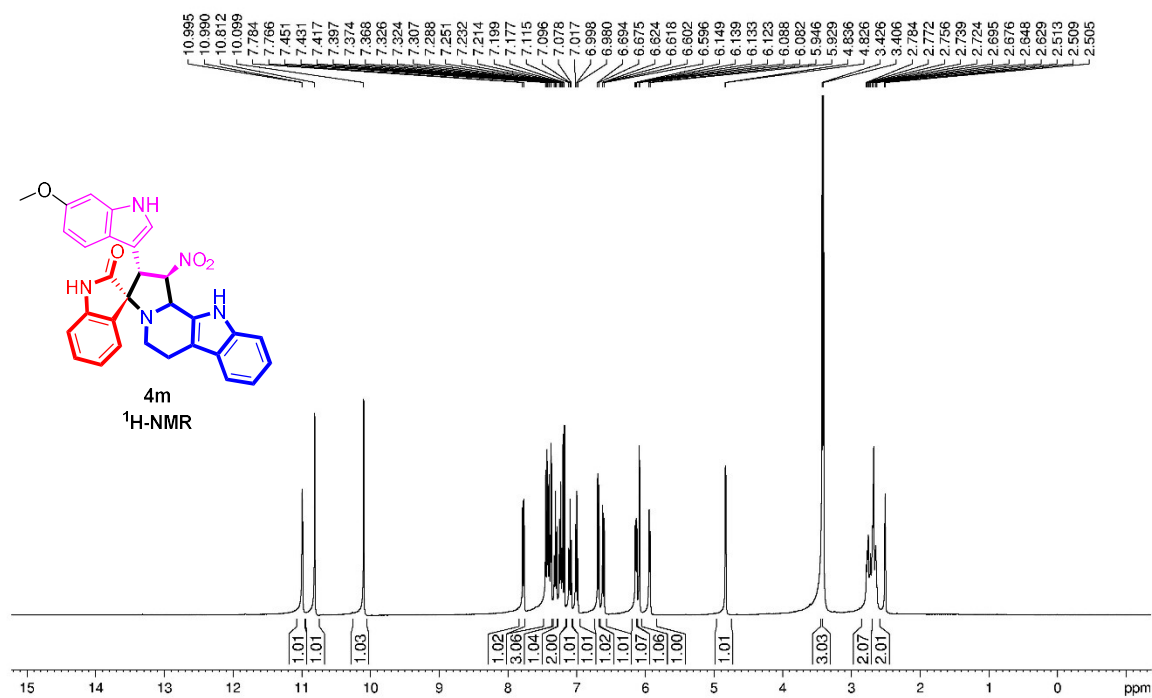
Integration values: 0.03, 1.00, 0.03, 1.00, 0.03, 1.00, 1.04, 2.05, 2.04, 1.02, 1.01, 1.07, 1.06, 1.03, 1.00, 1.00, 1.01, 1.01, 1.00, 1.01, 2.04, 2.03, 3.03.

4k
¹³C-NMR

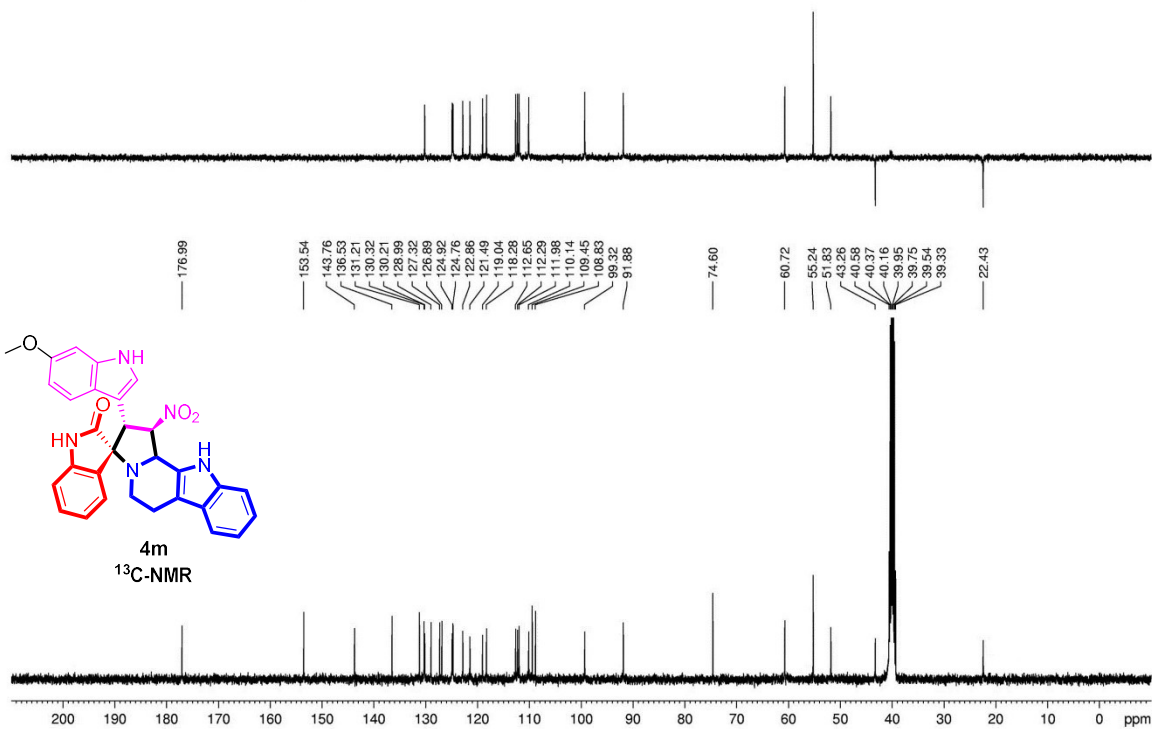
Chemical structure of **4k** is shown. The spectrum displays peaks corresponding to the structure, with labeled chemical shifts (ppm): 177.14, 143.78, 136.52, 134.61, 130.30, 128.14, 128.91, 127.47, 127.28, 126.88, 124.91, 124.23, 123.25, 122.81, 121.43, 118.28, 118.02, 111.98, 111.53, 110.06, 109.46, 108.40, 91.88, 74.70, 60.70, 52.07, 43.28, 40.57, 40.36, 40.15, 39.95, 39.77, 39.53, 39.32, 22.43, 21.56.



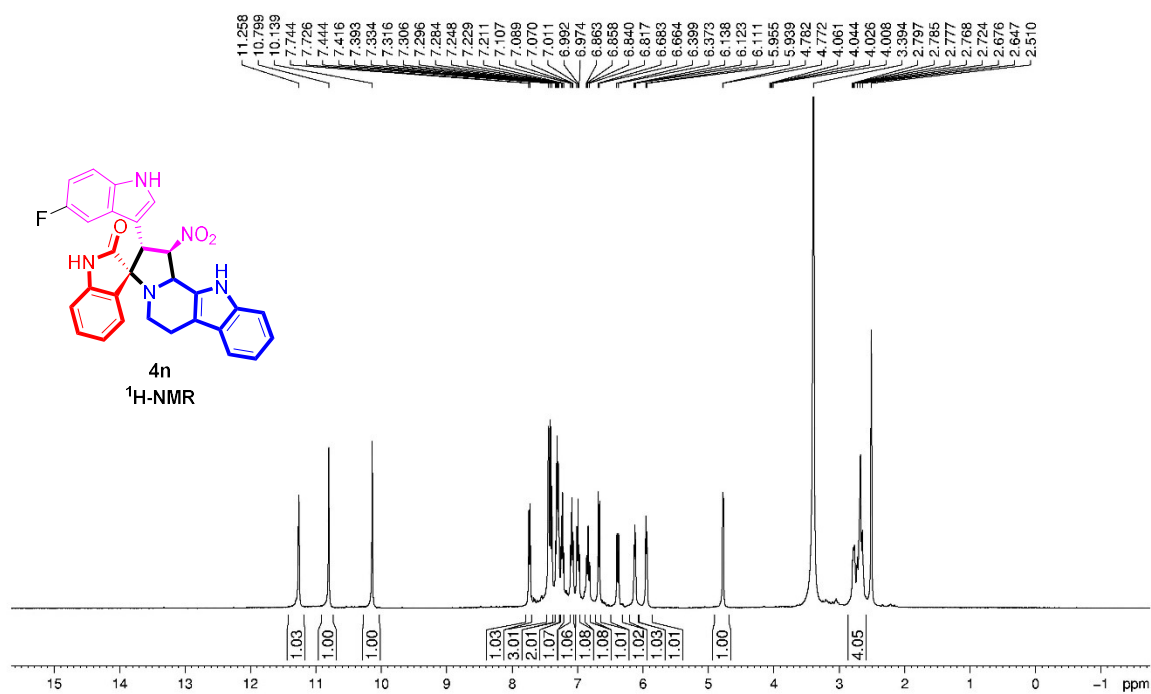
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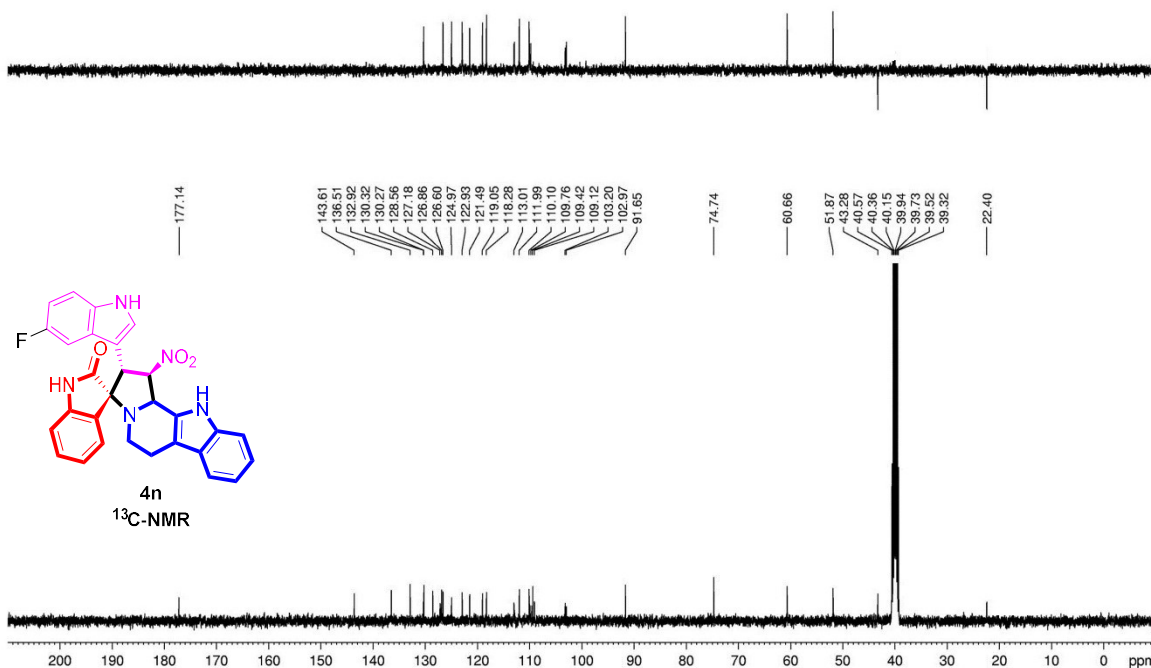
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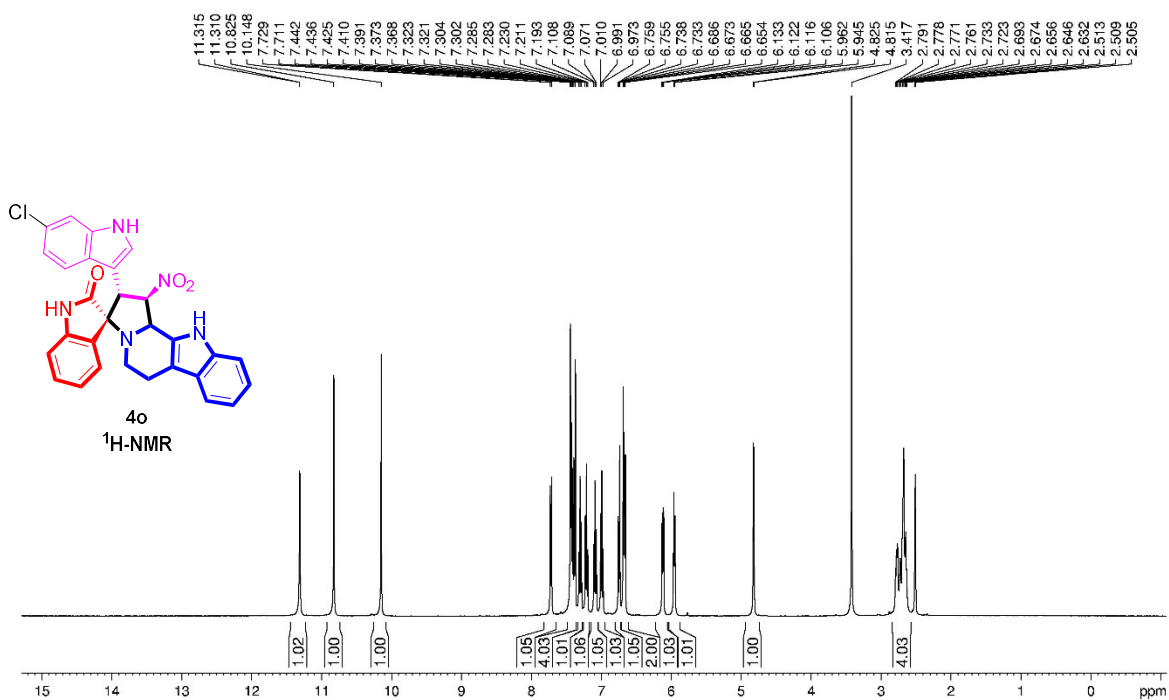
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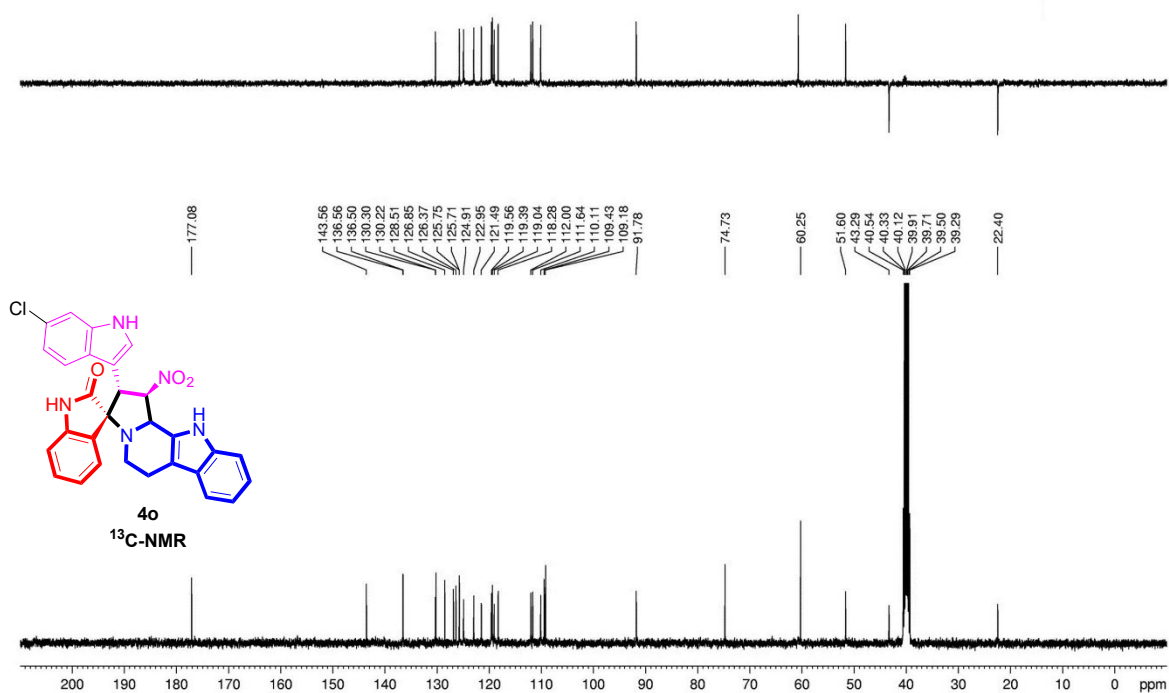
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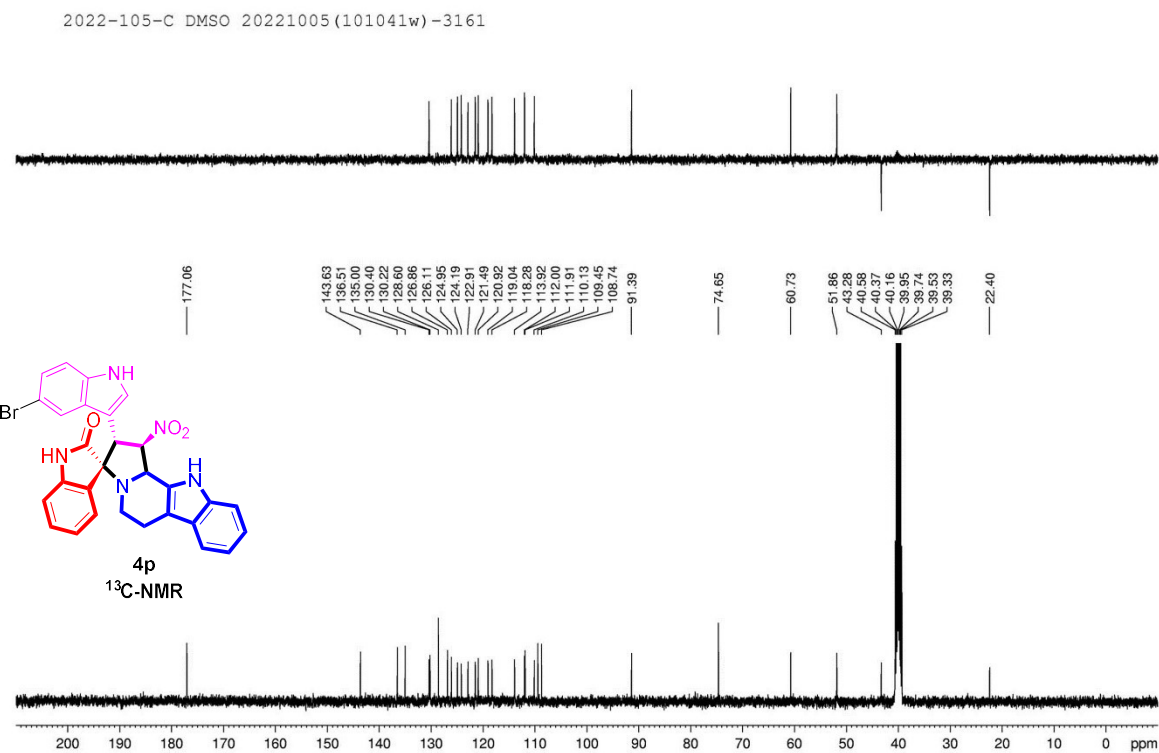
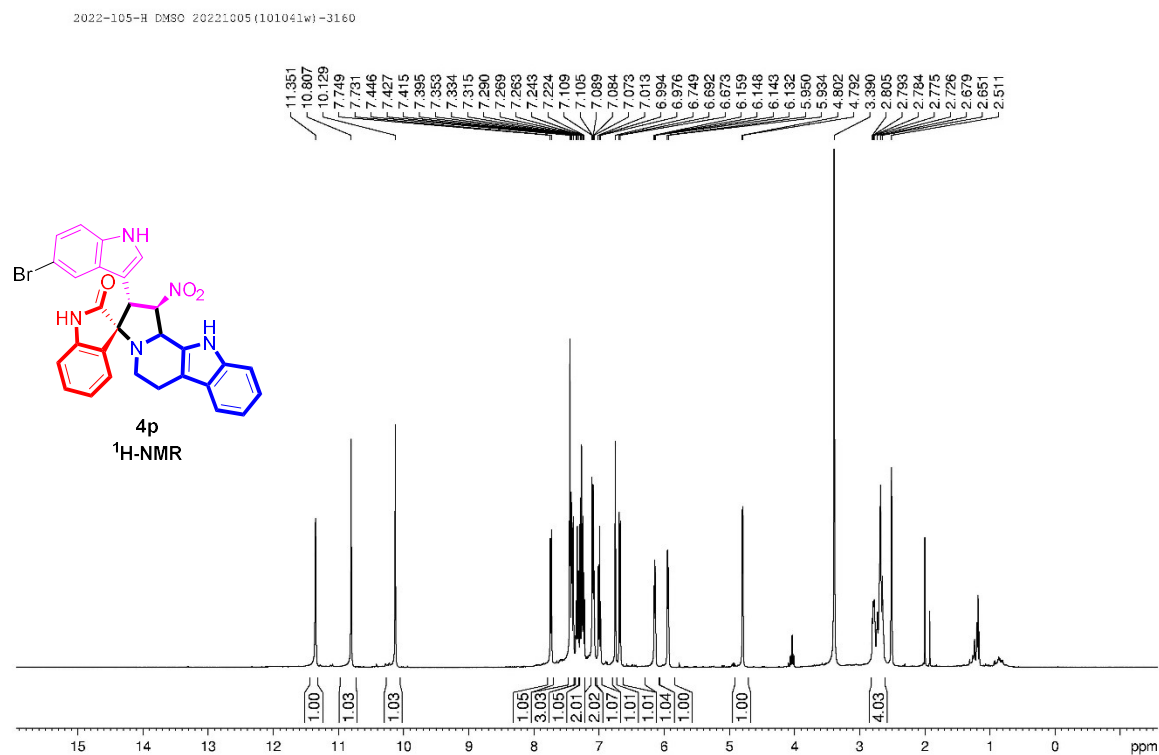


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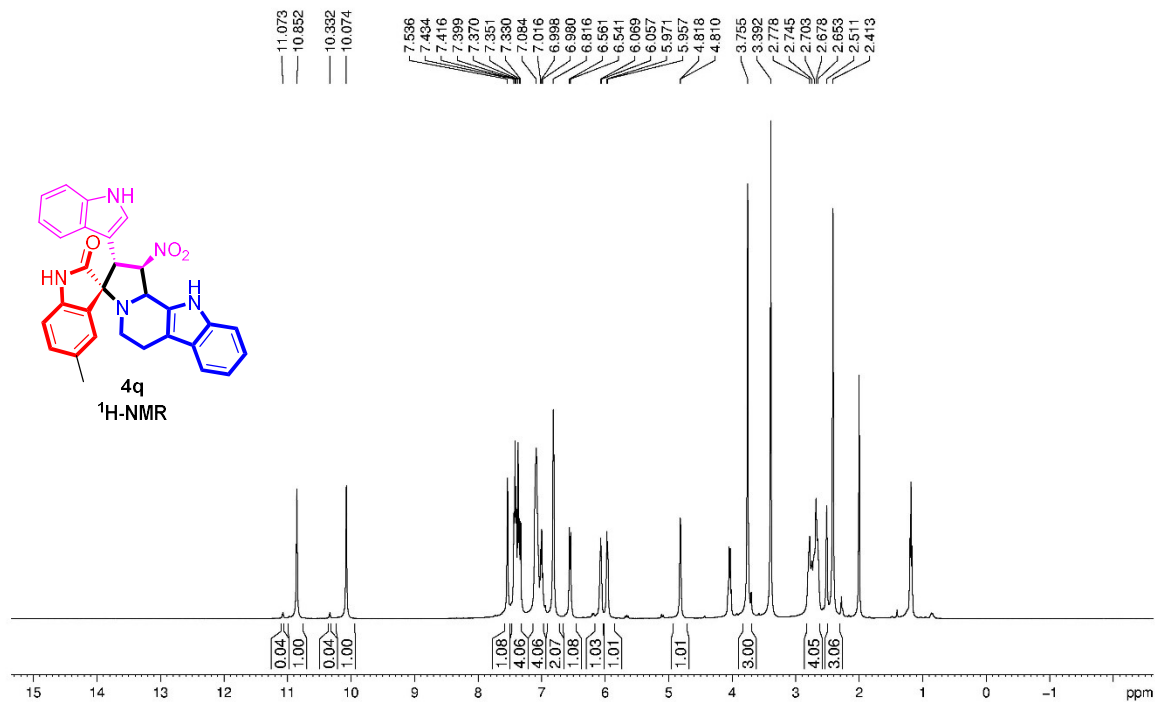


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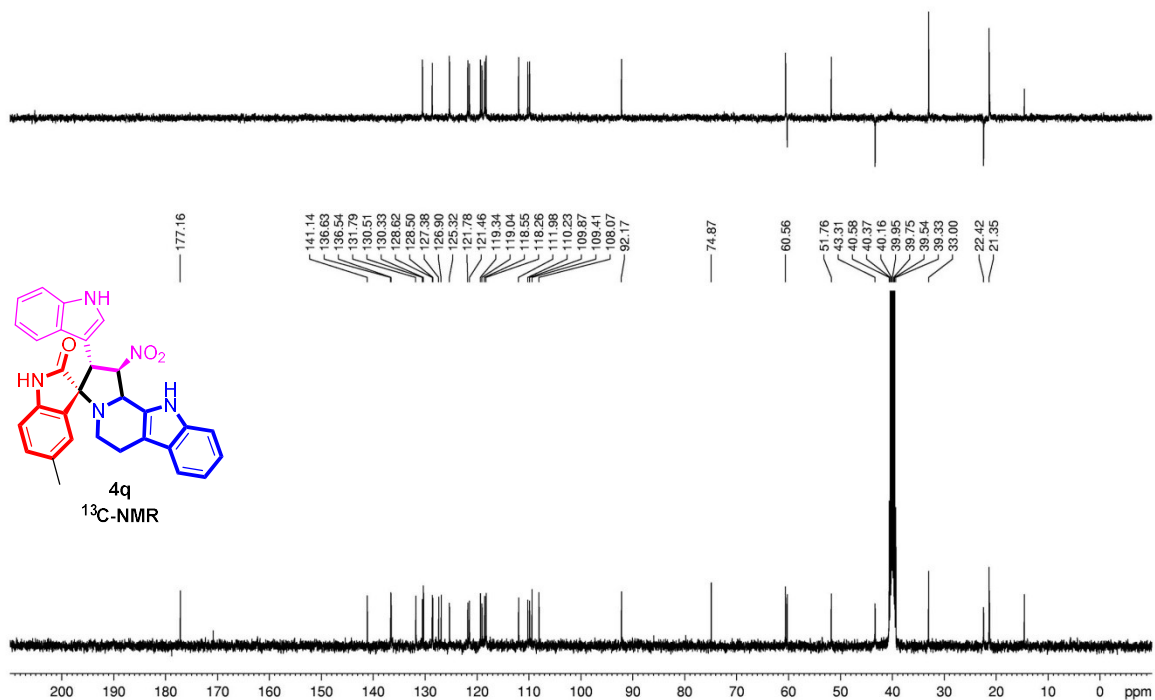




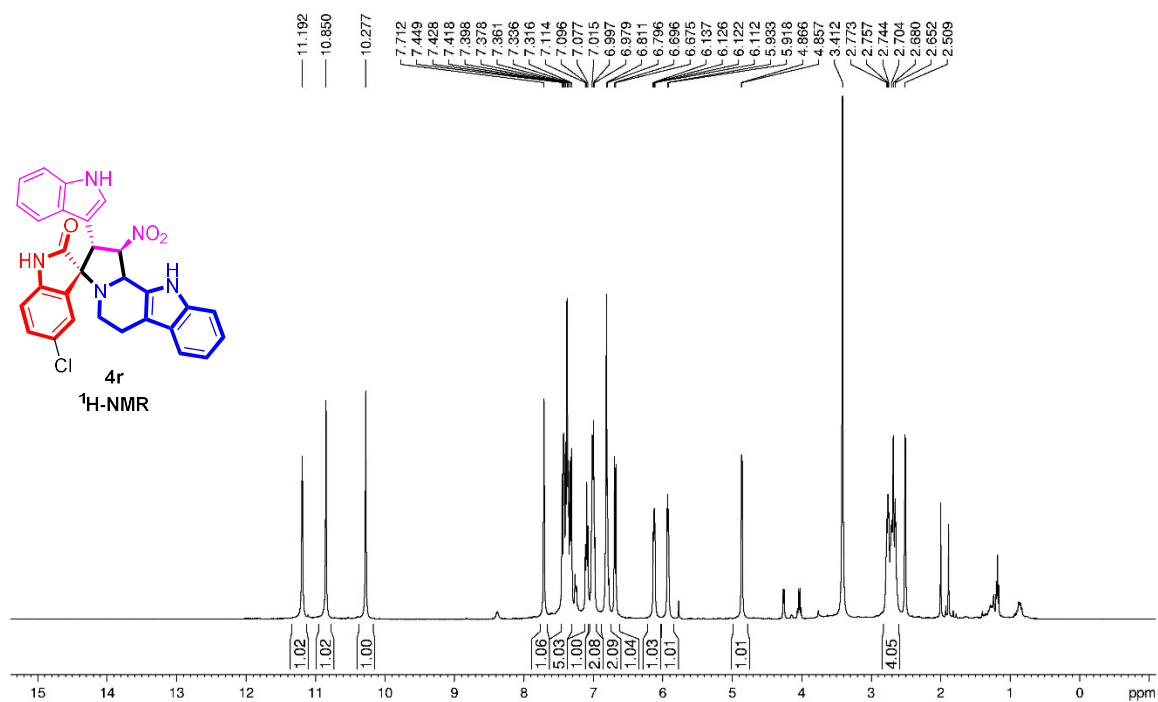
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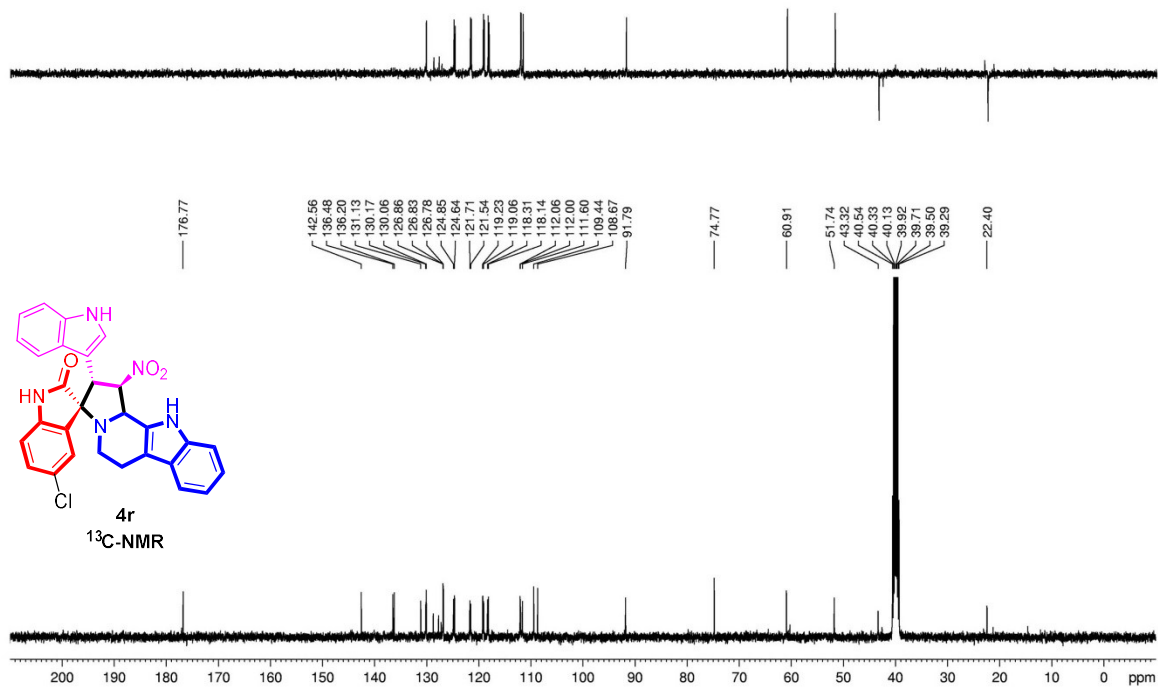
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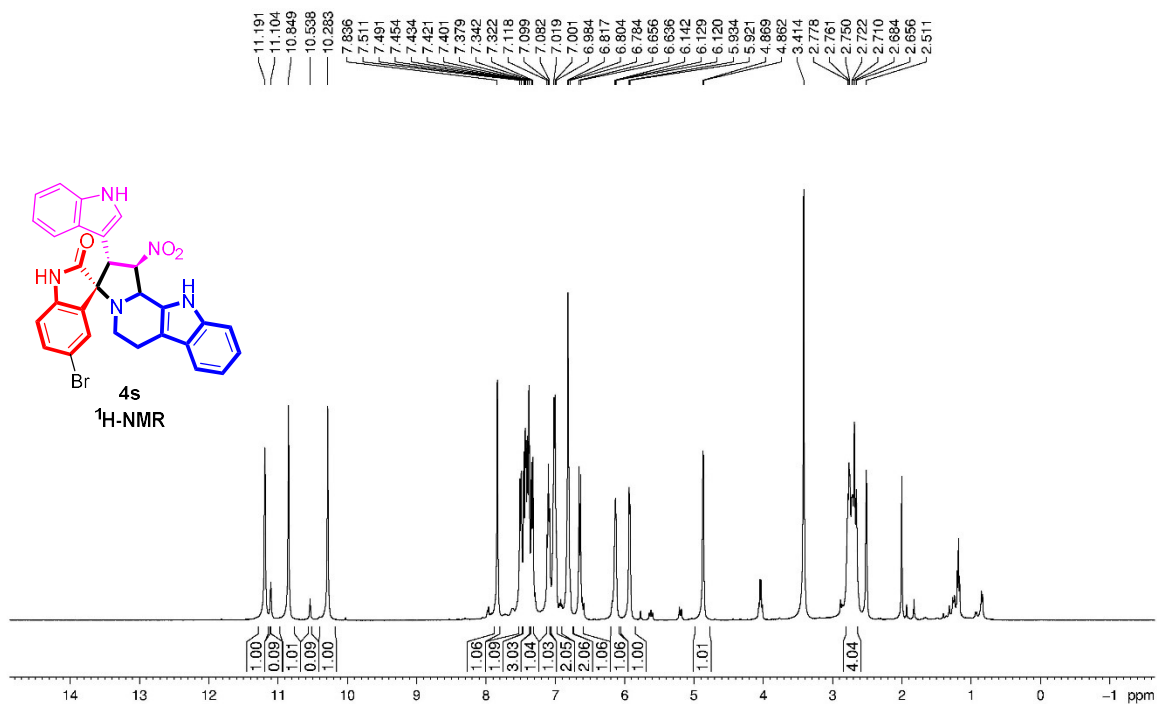
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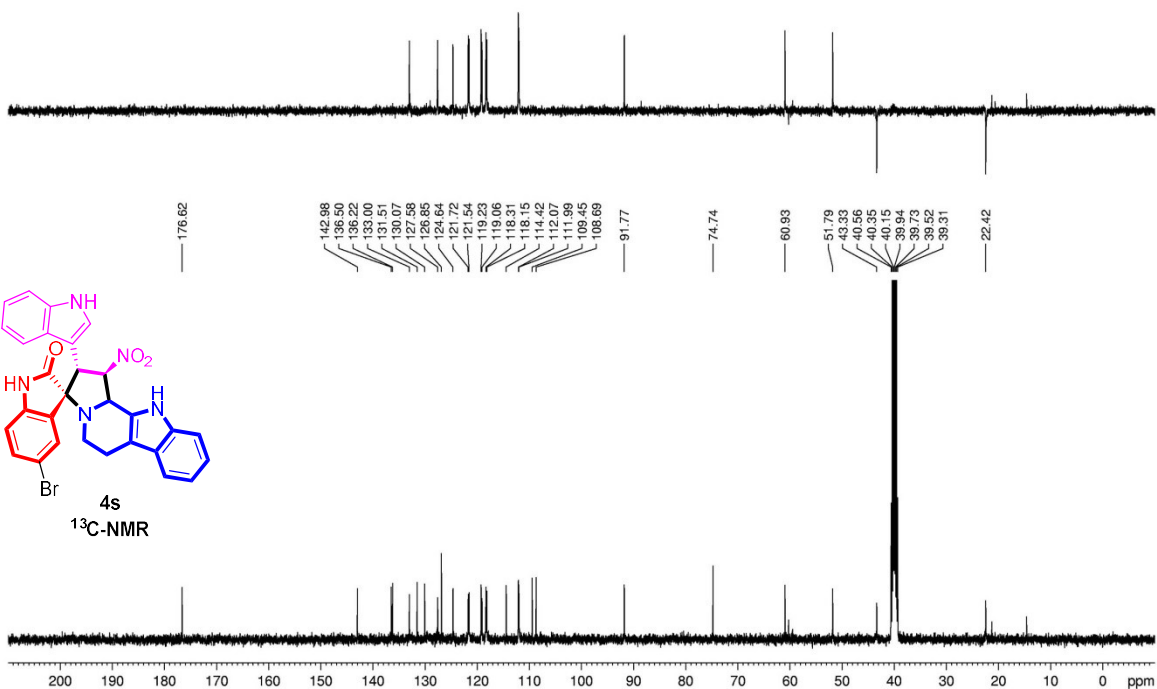
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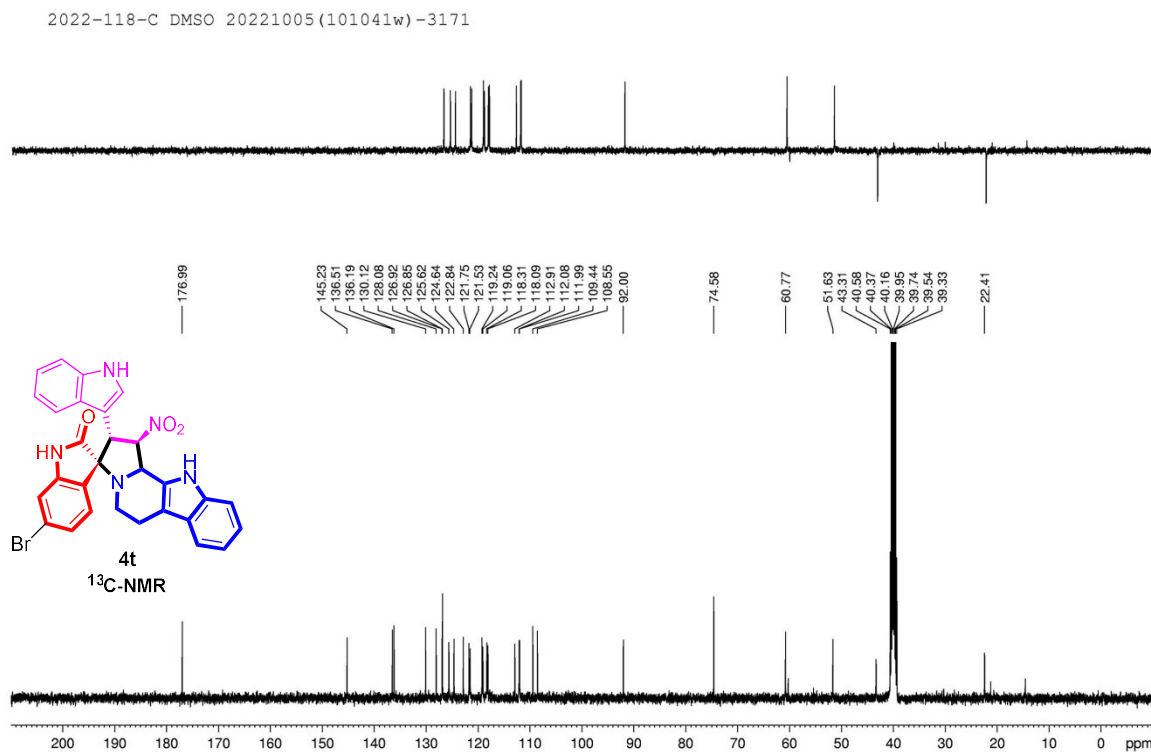
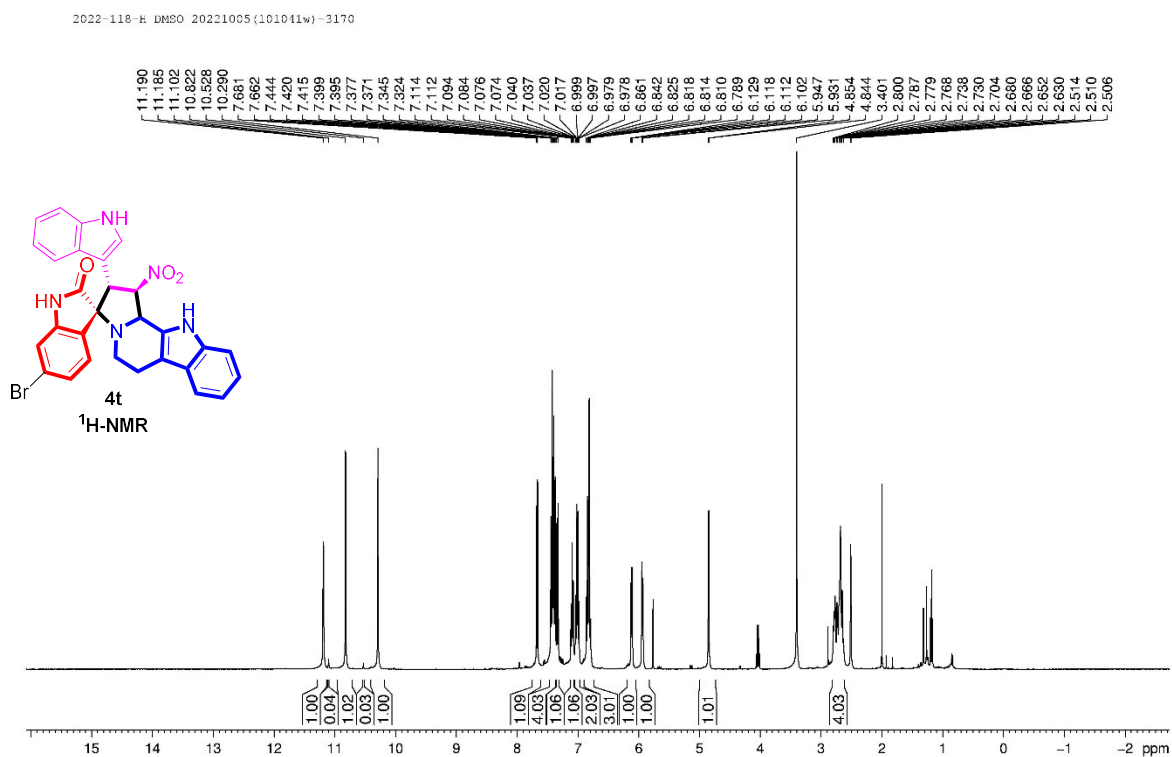


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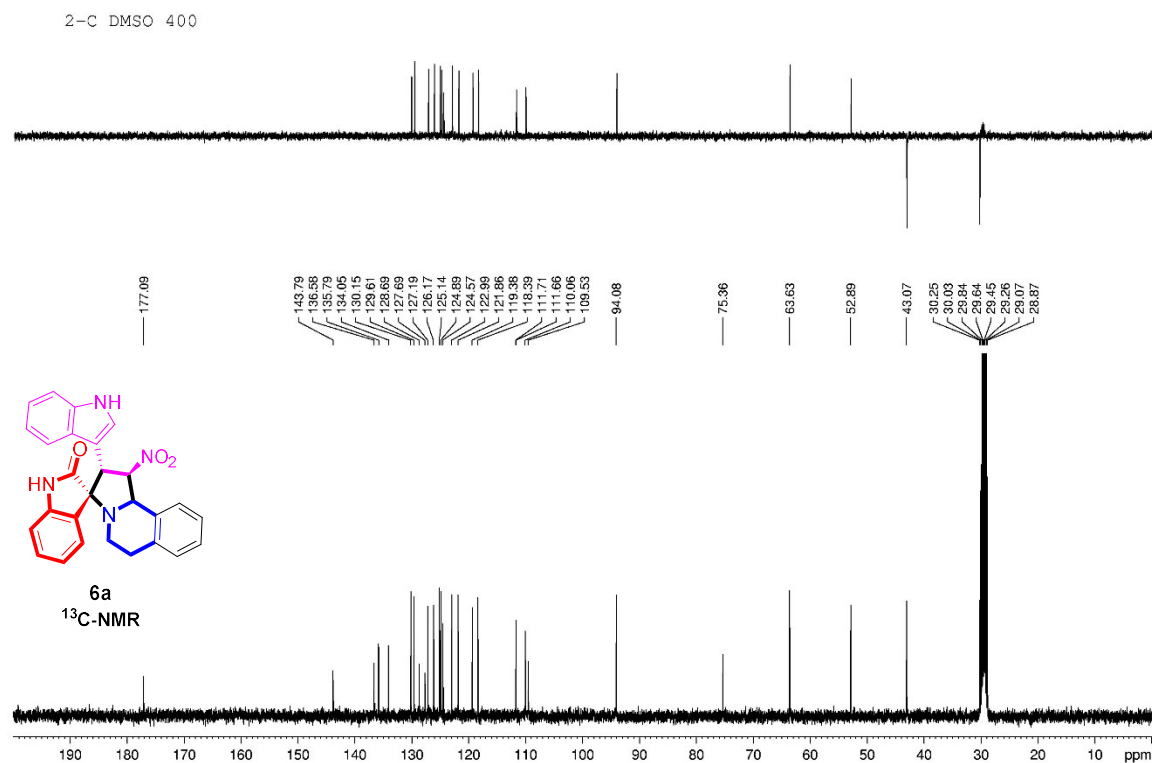
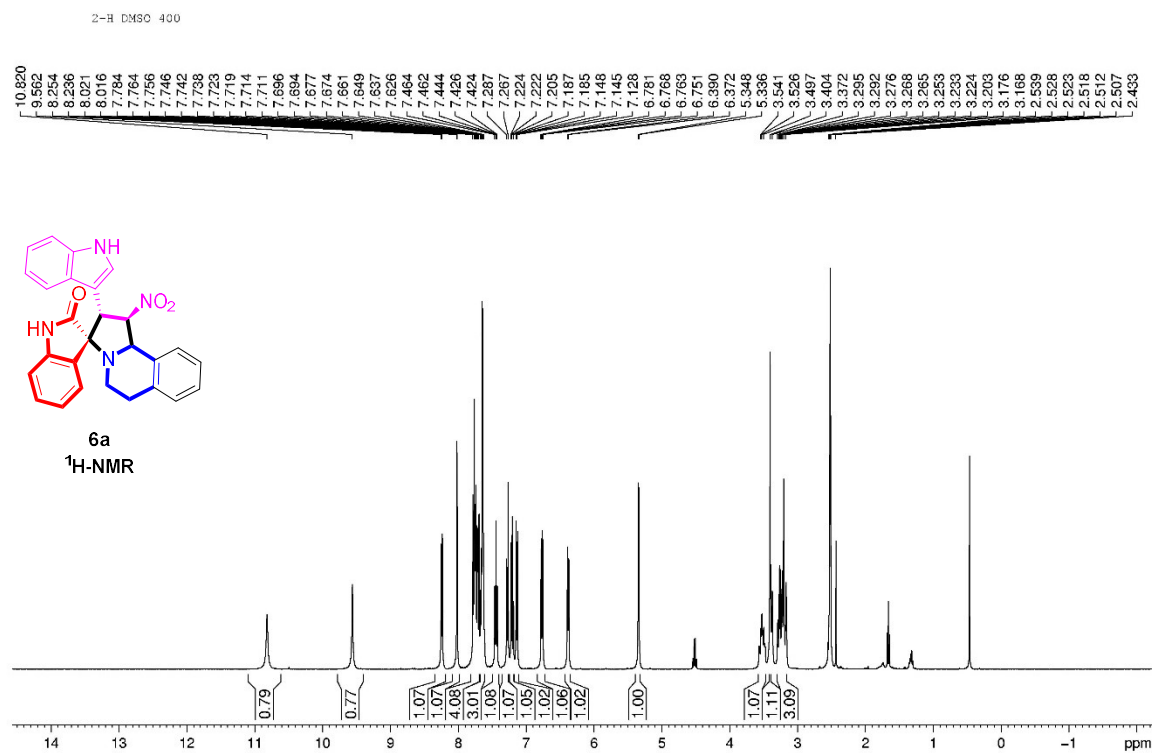


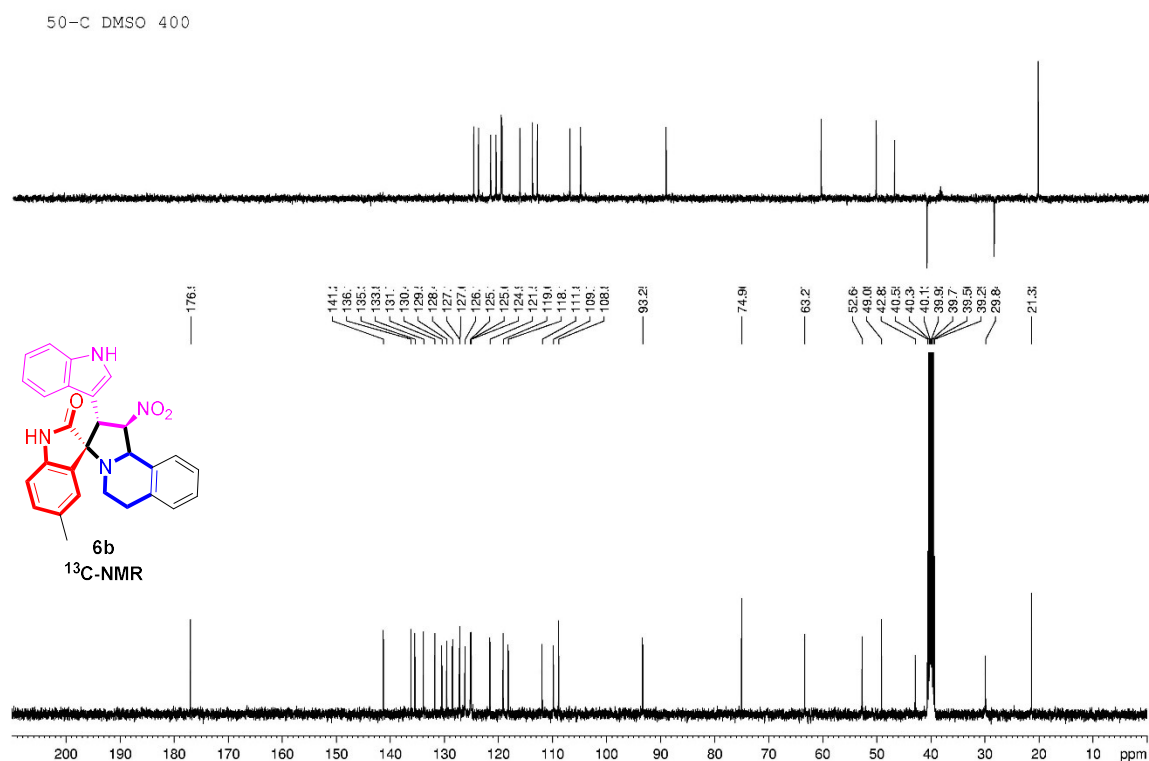
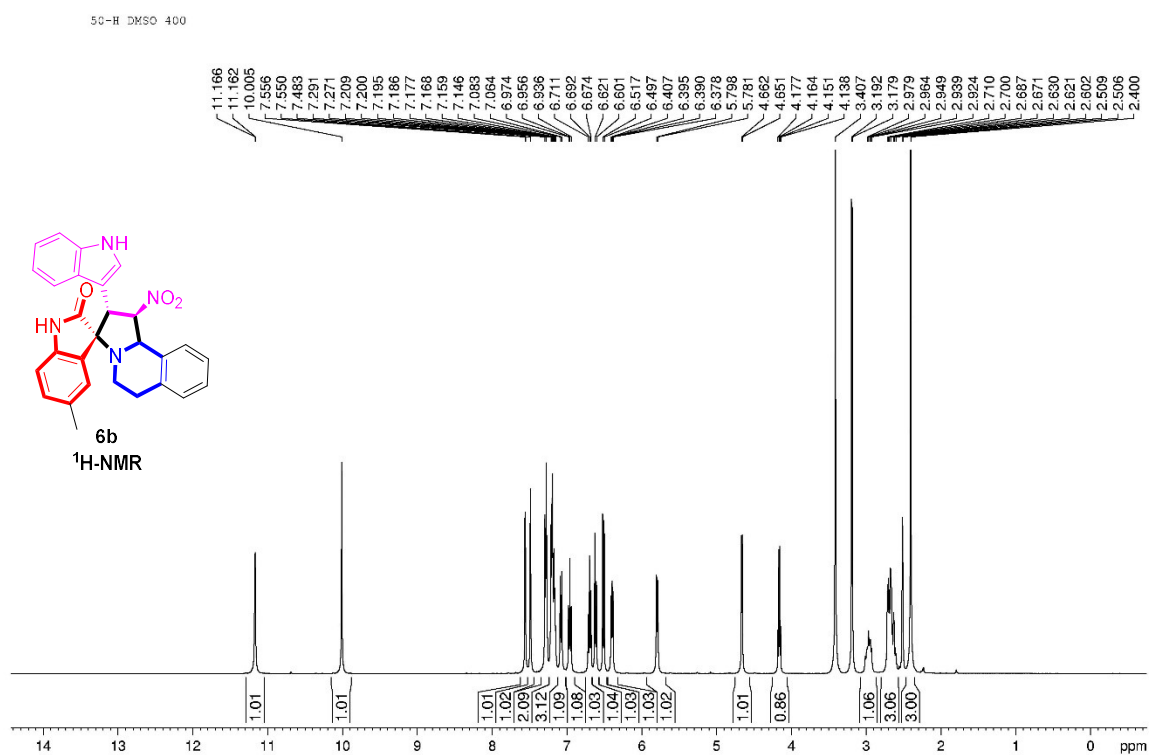
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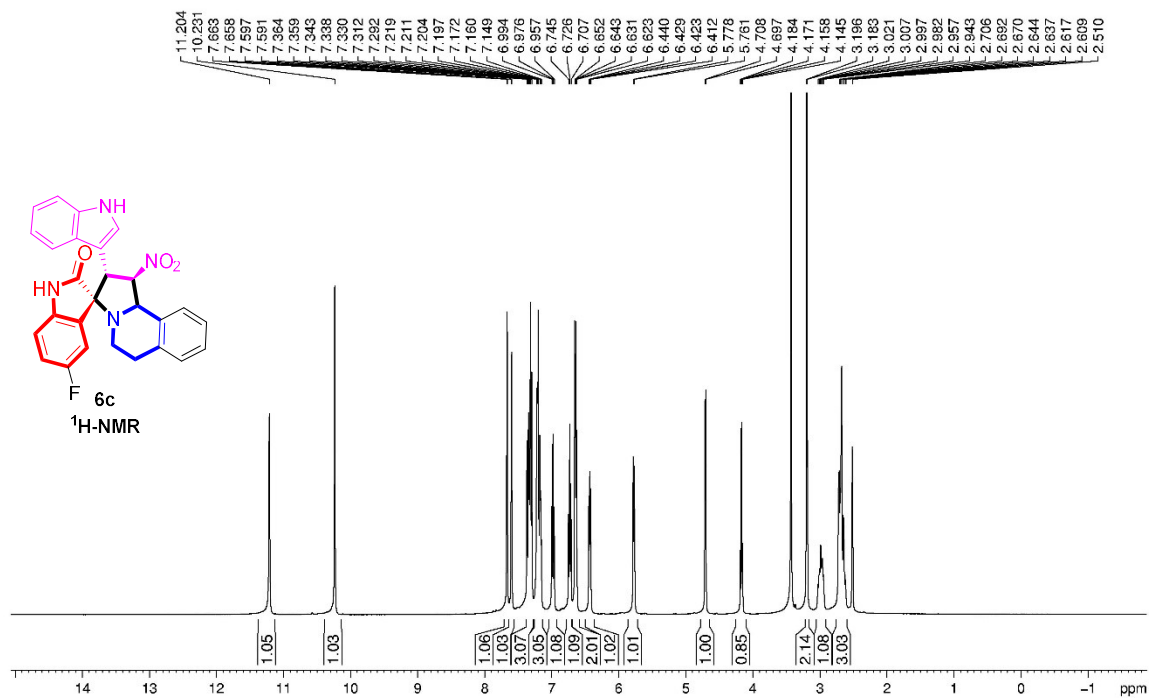


2. ^1H and ^{13}C NMR spectra for compounds **6**

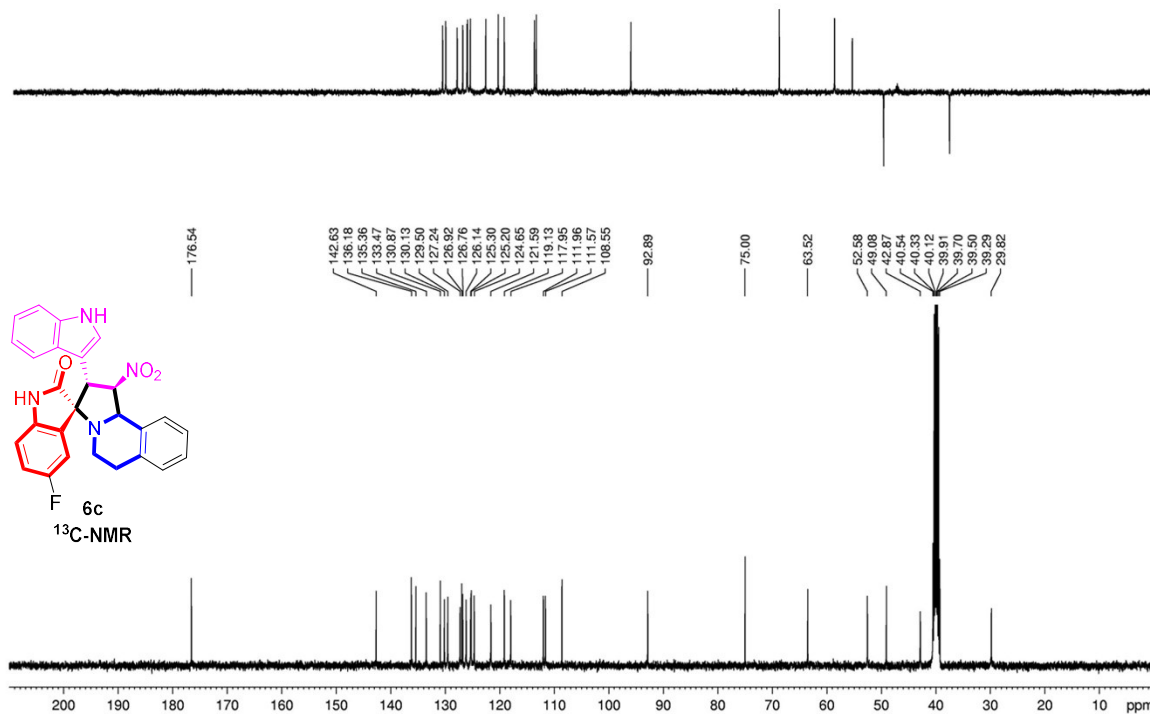


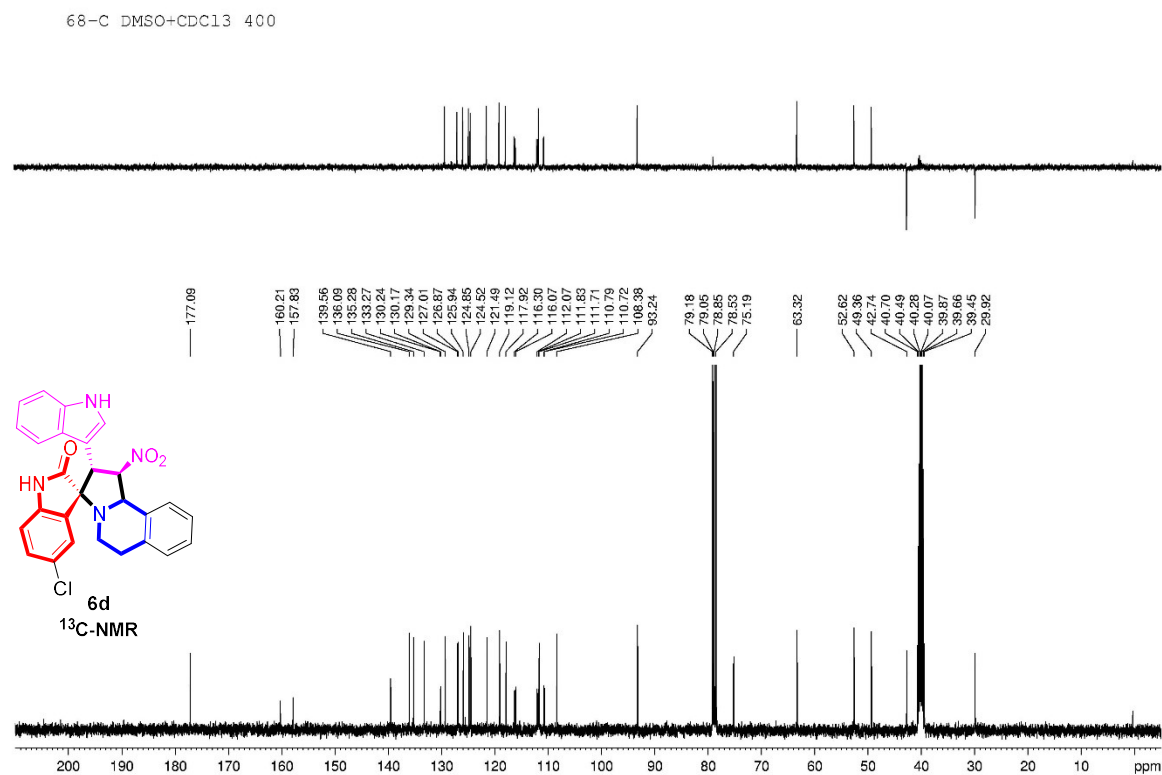
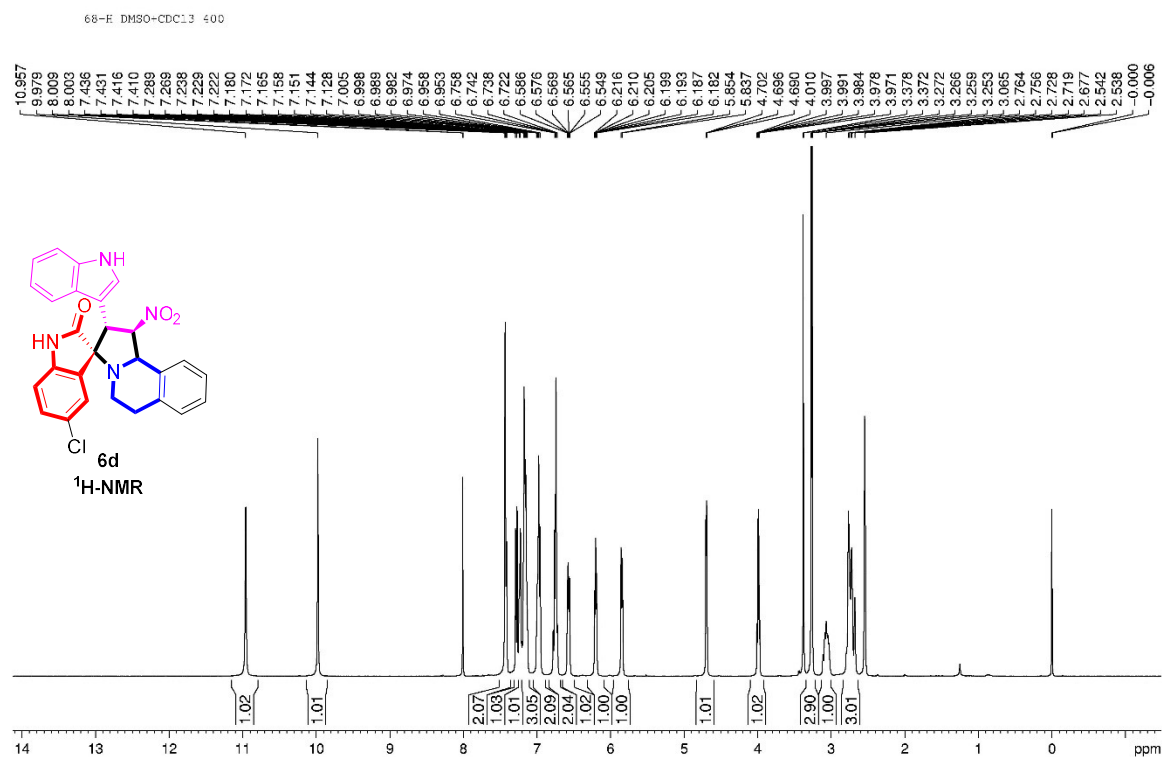


46-H DMSO 400

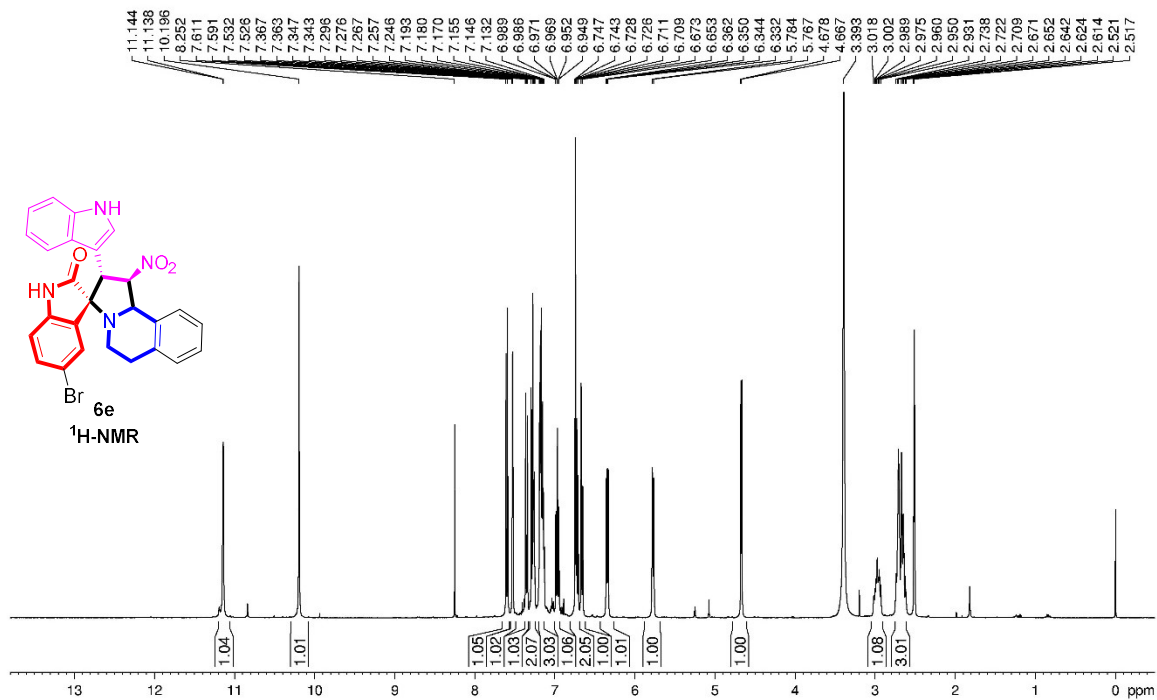


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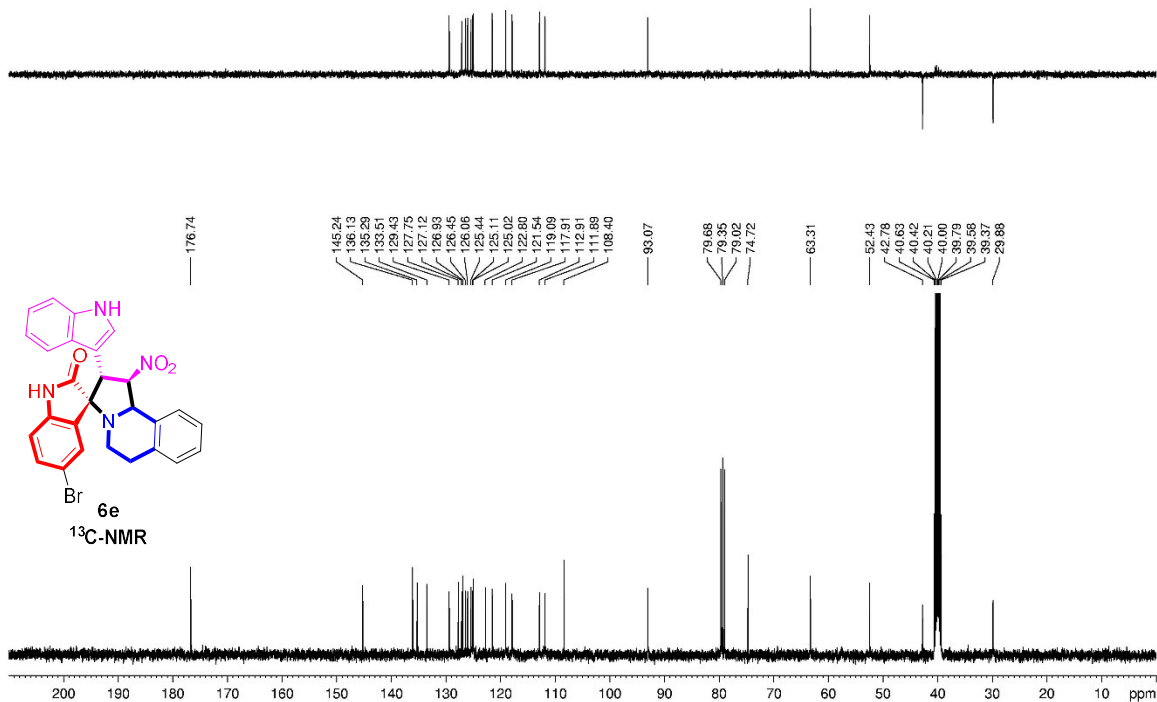


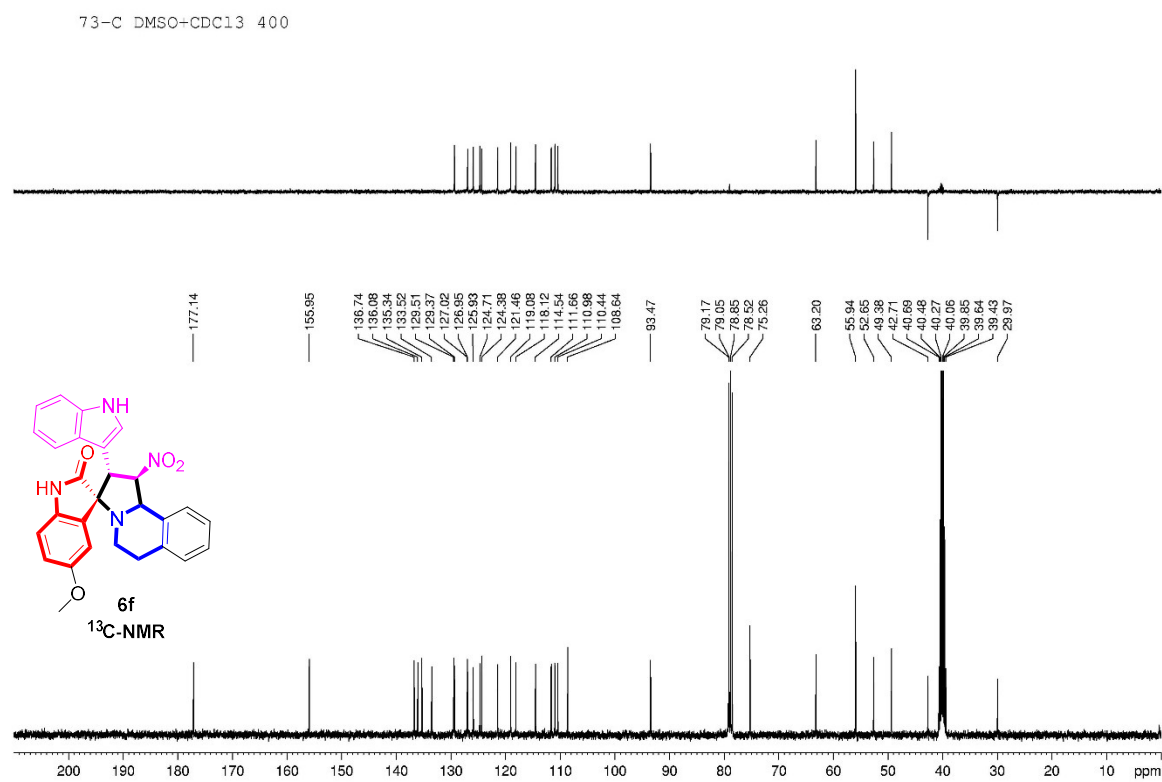
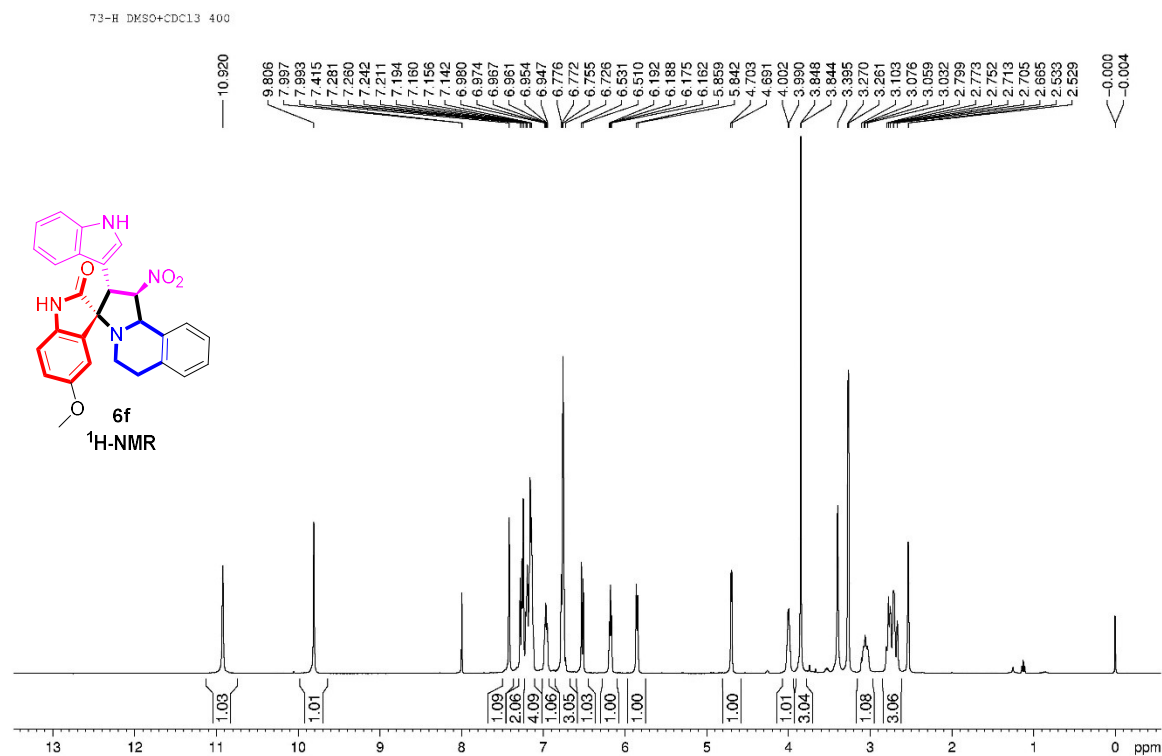


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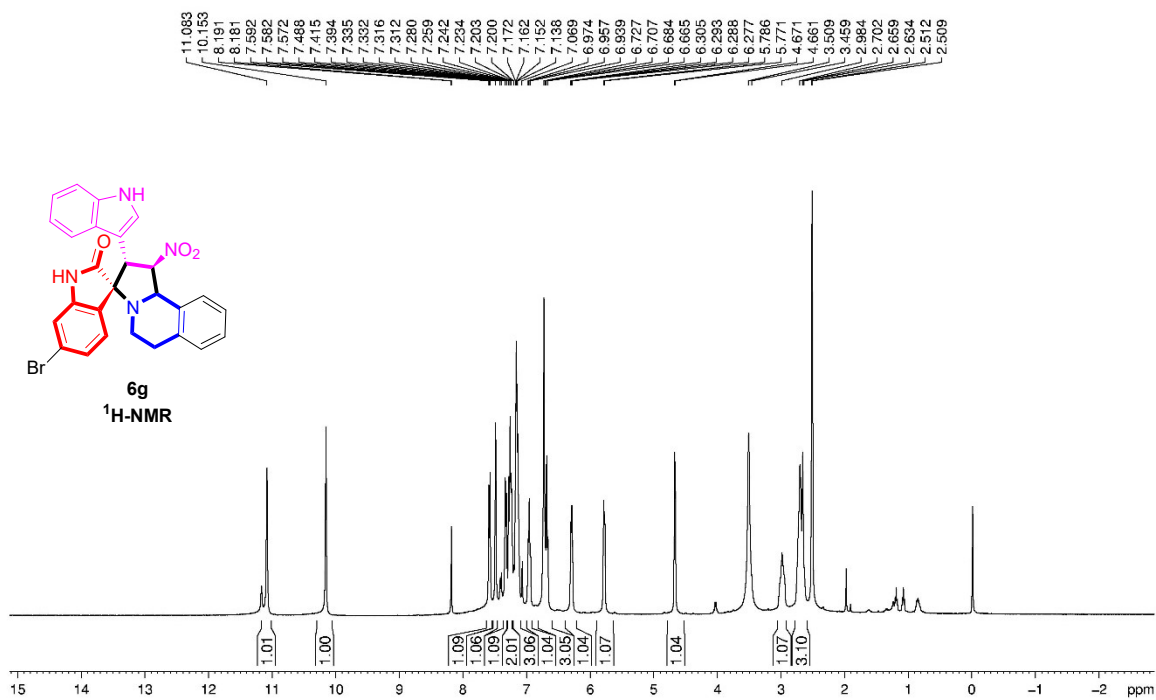


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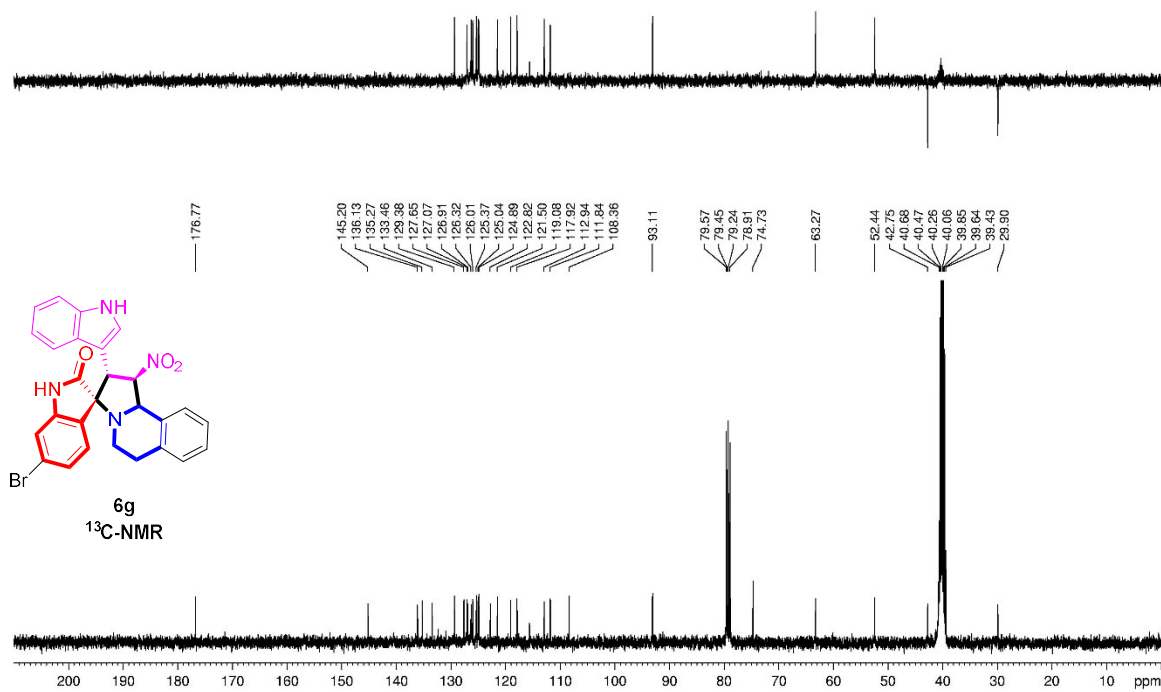




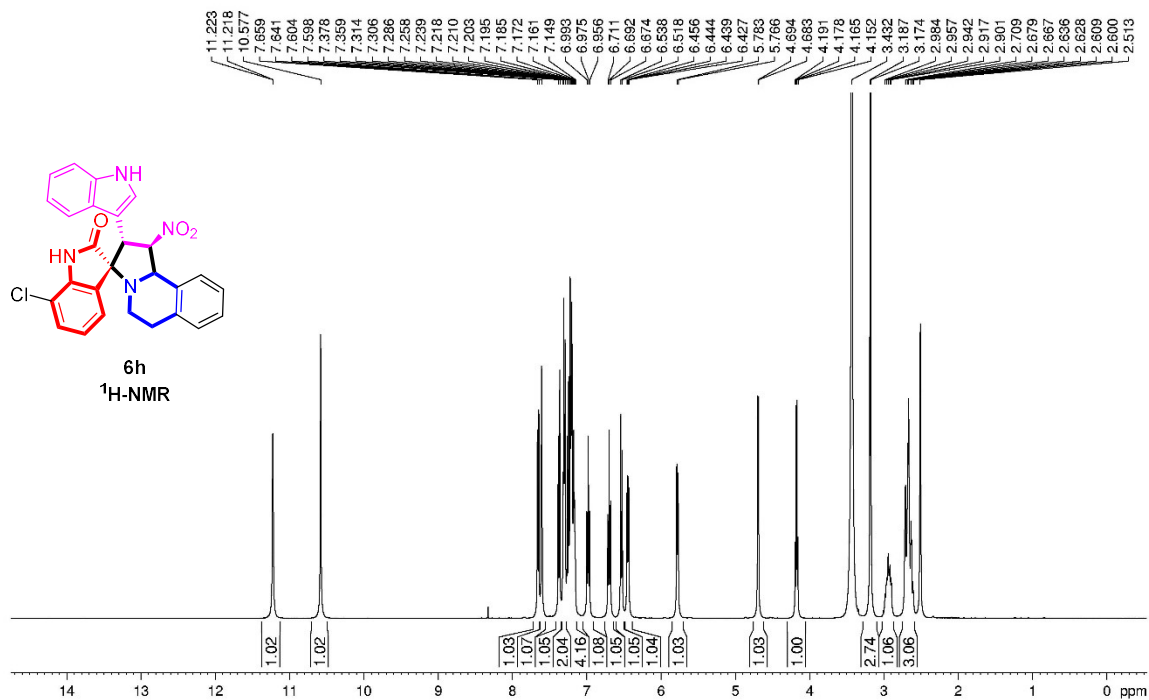
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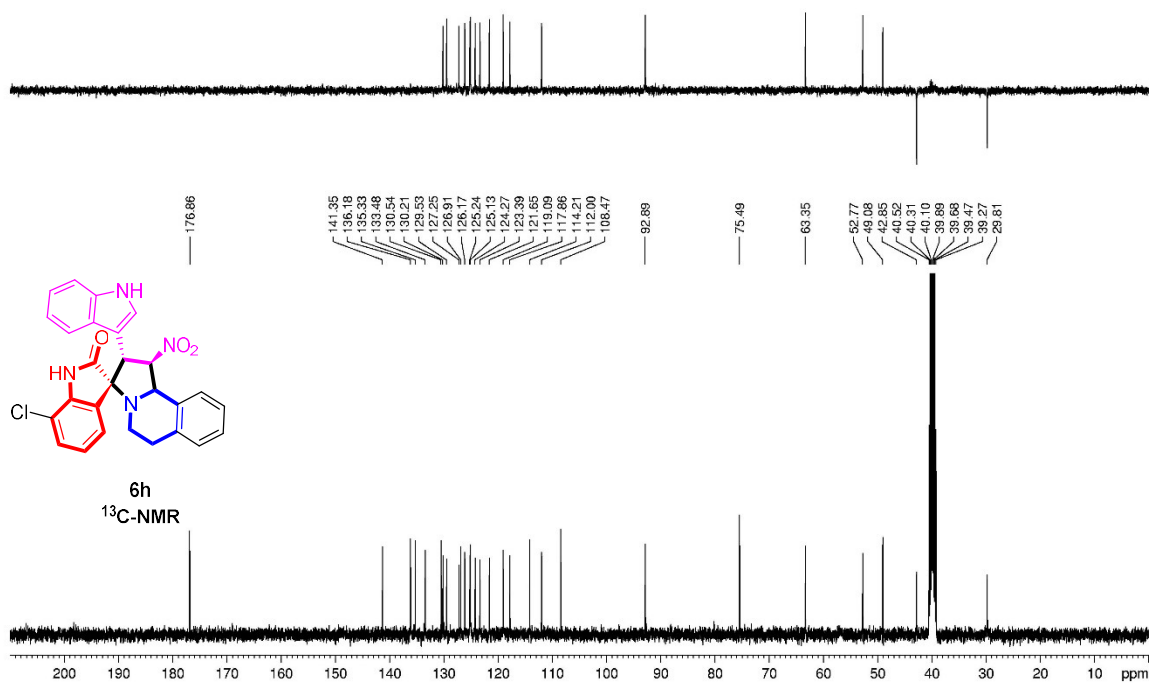
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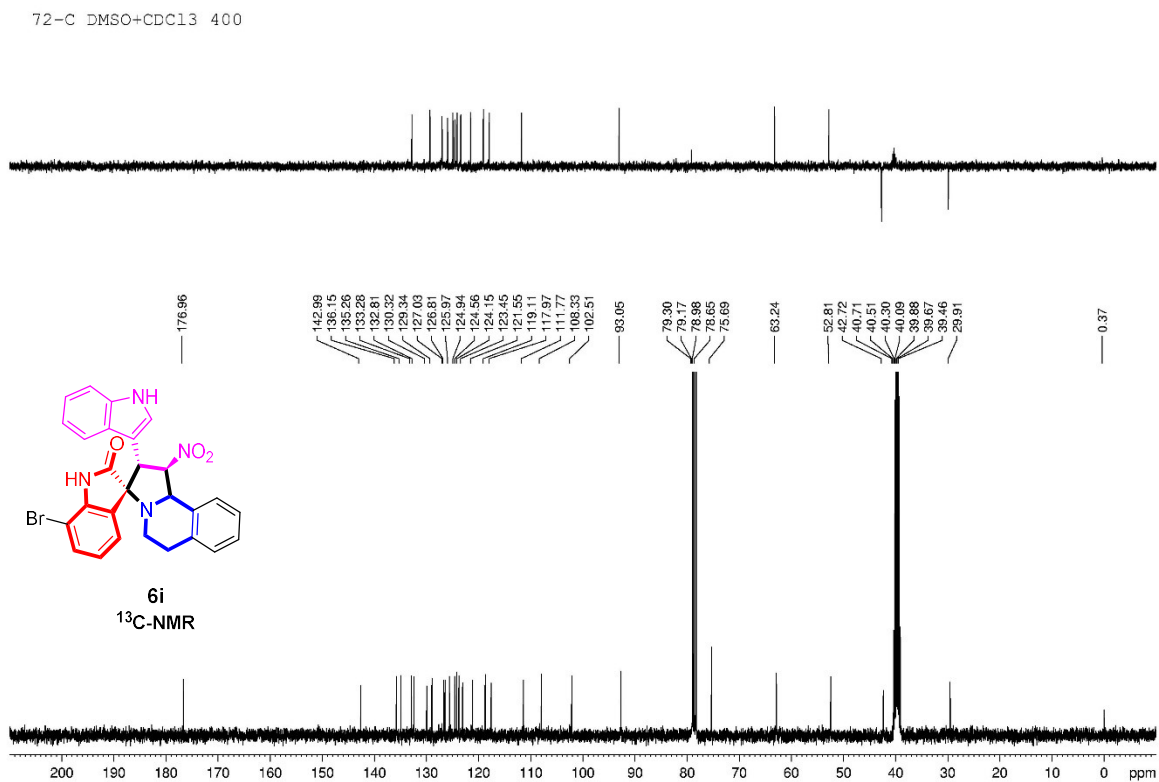
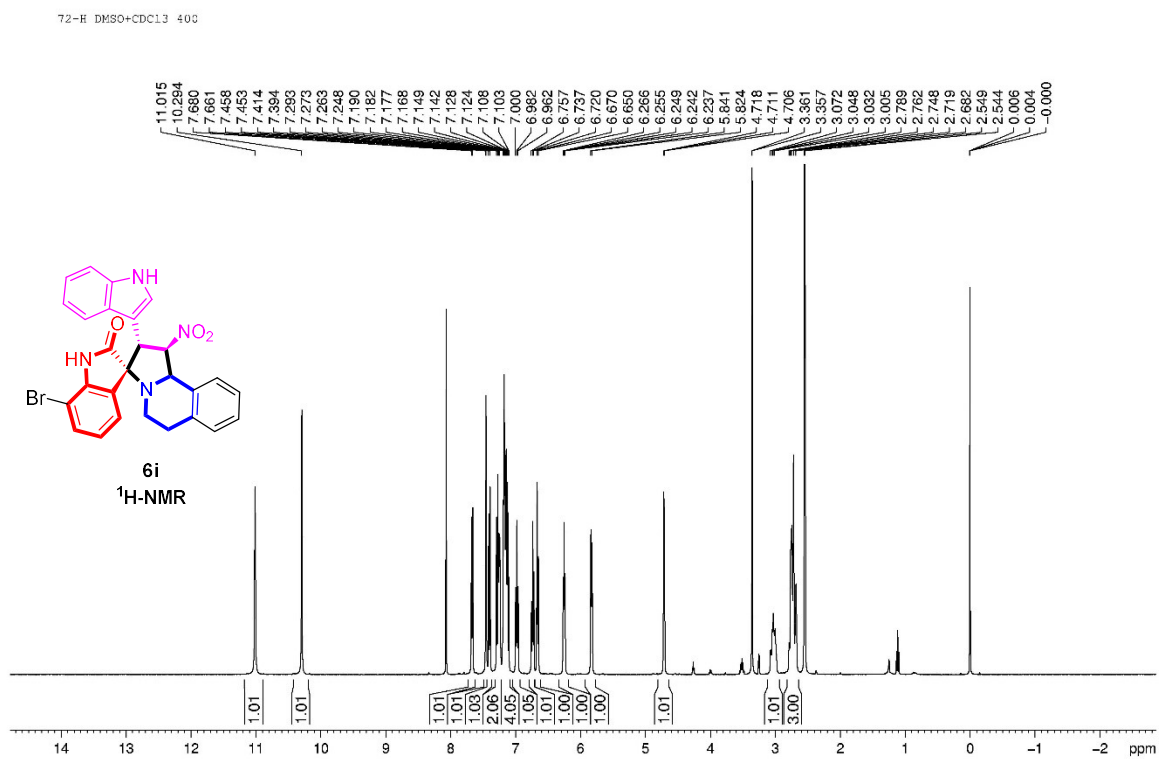


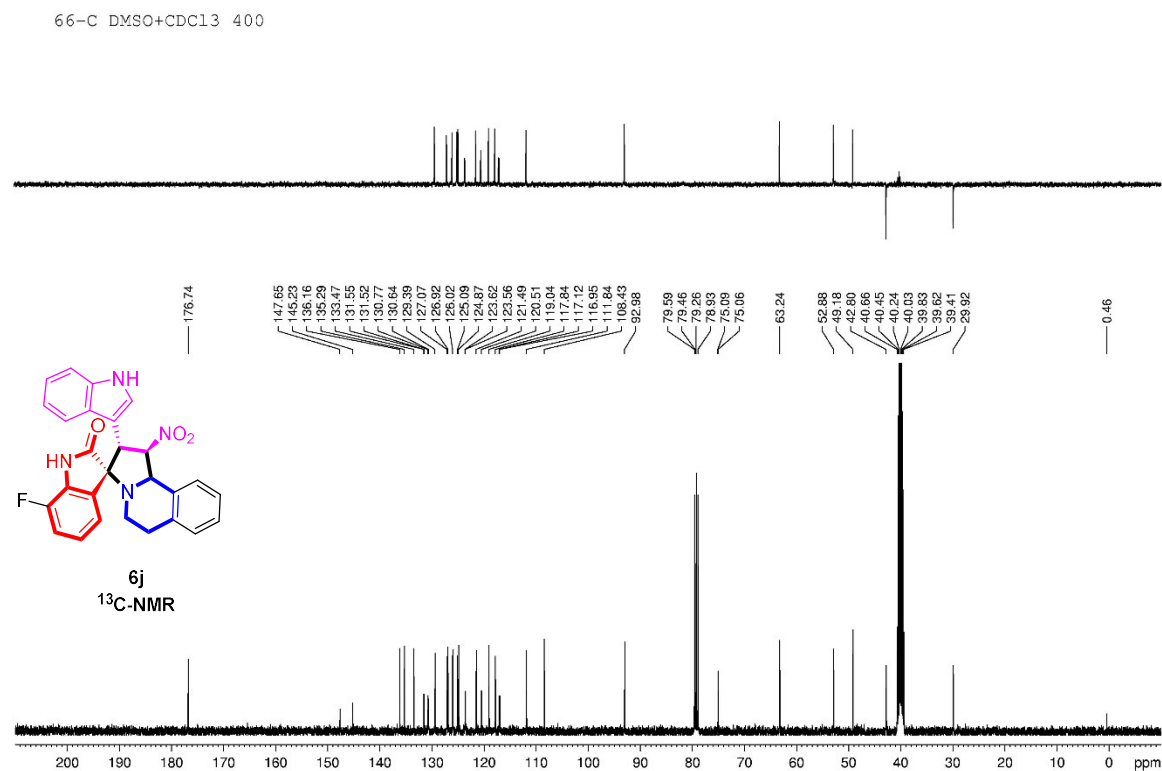
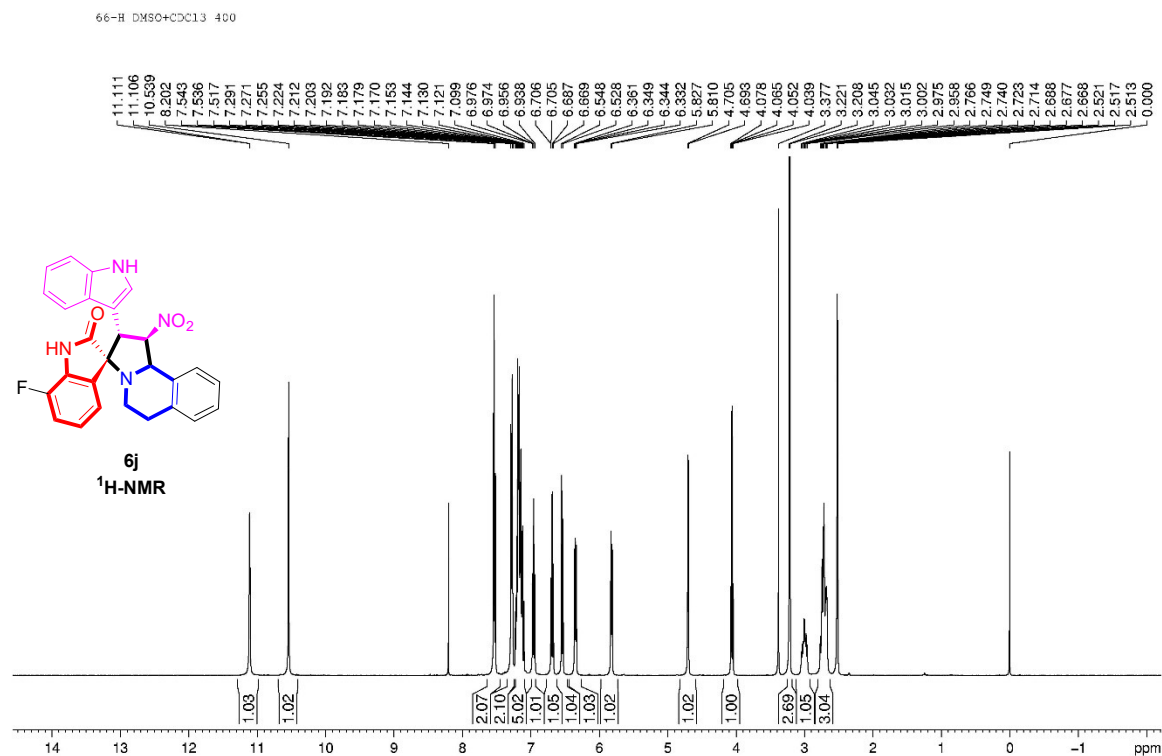
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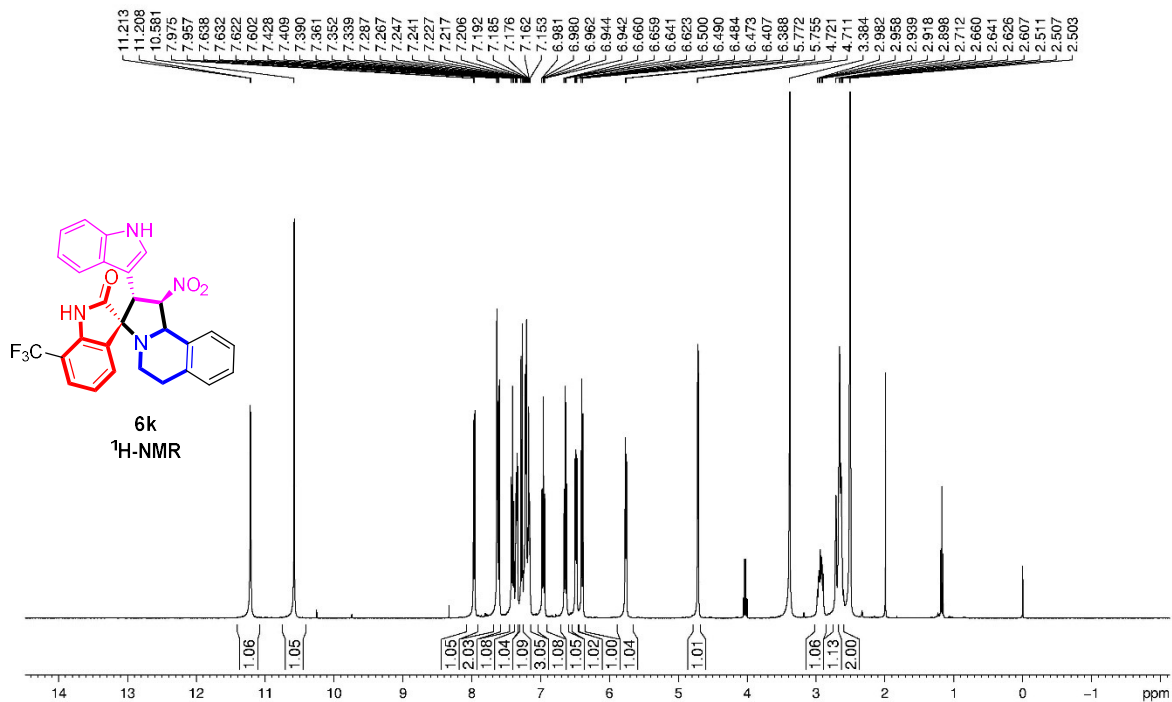
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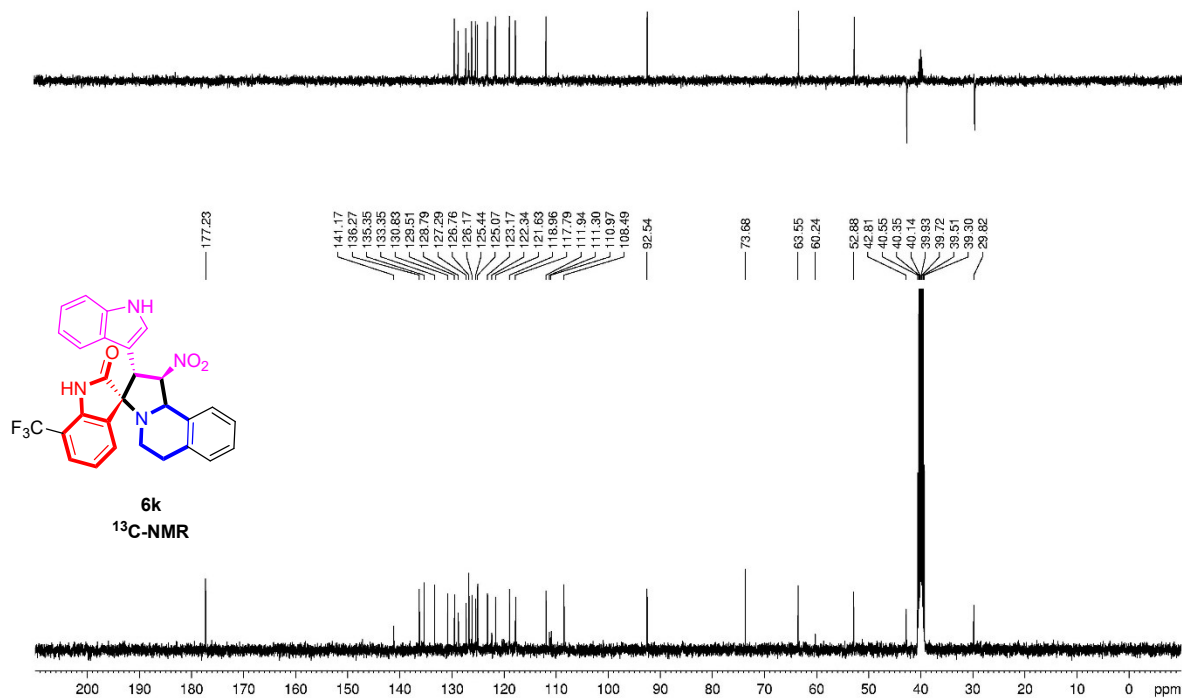


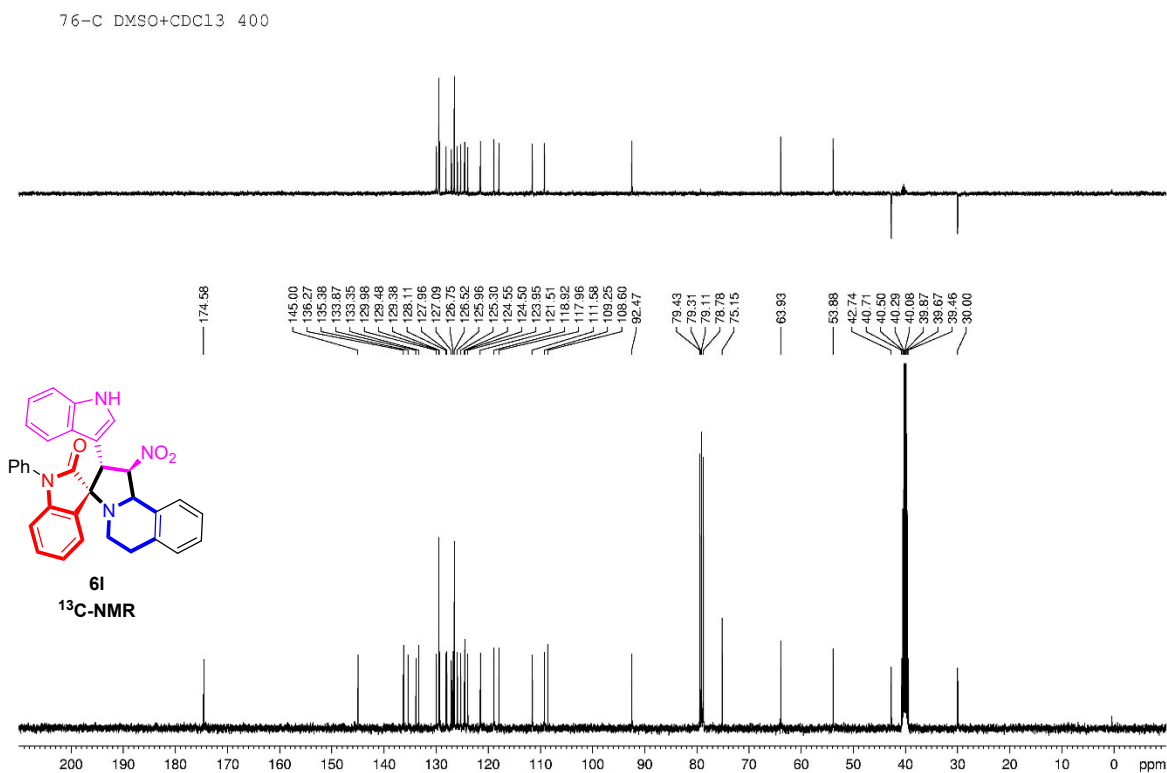
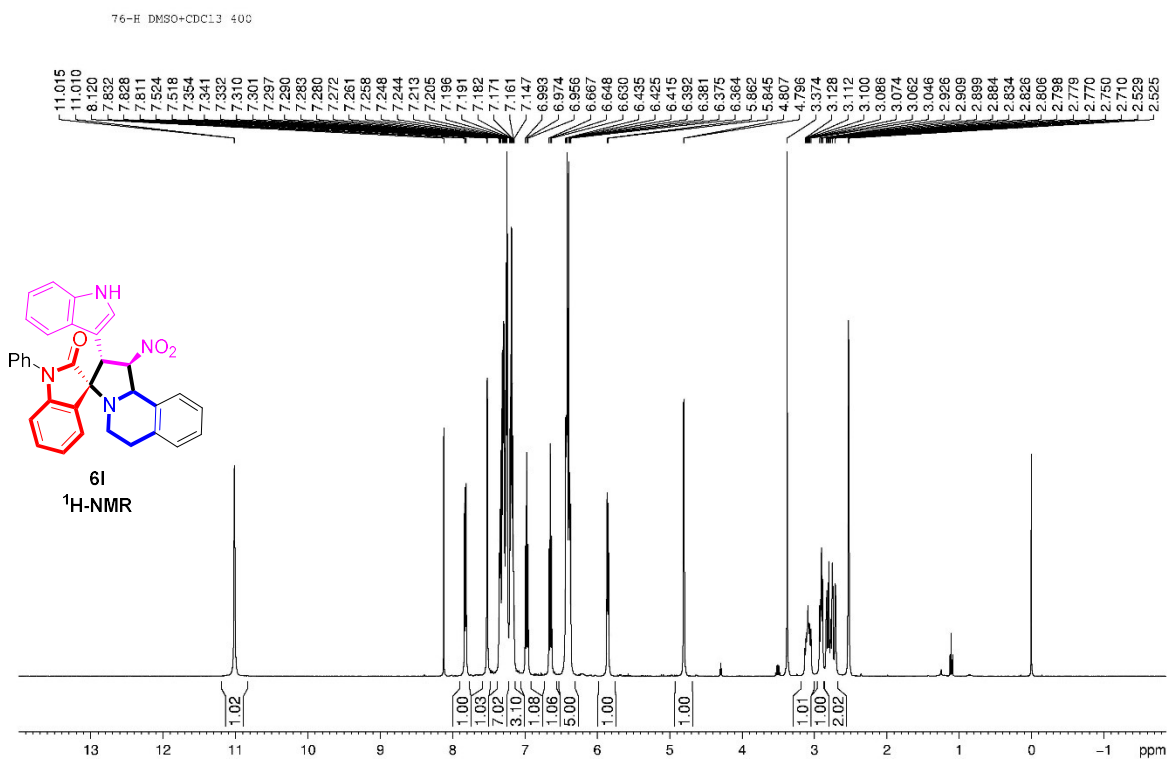


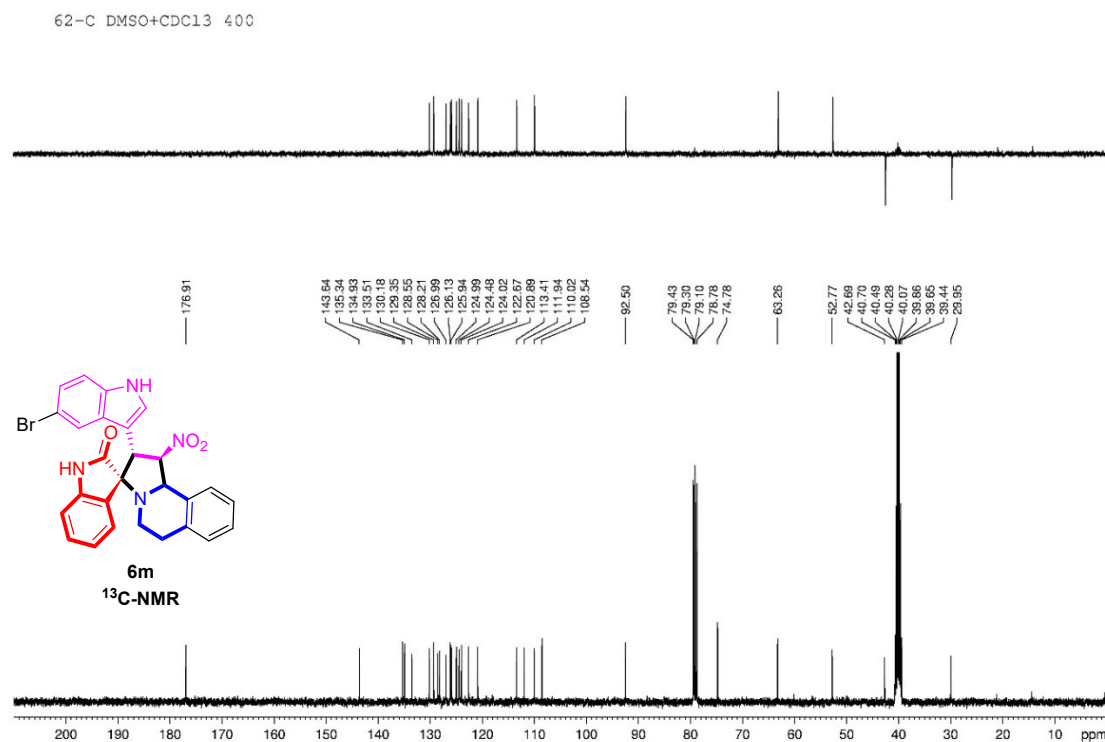
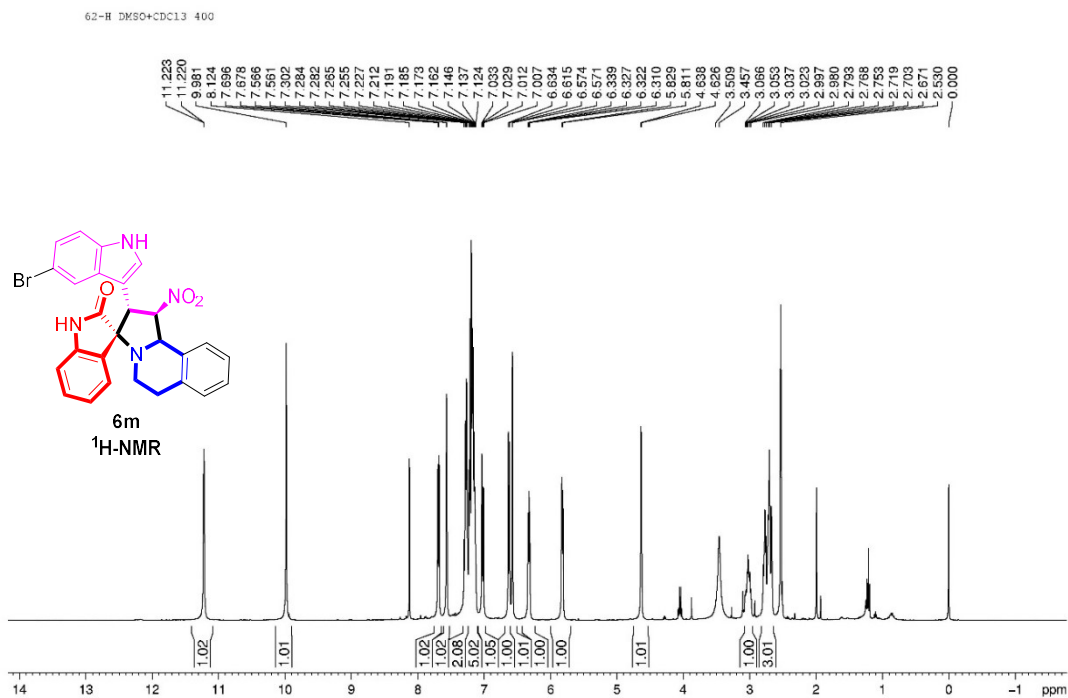
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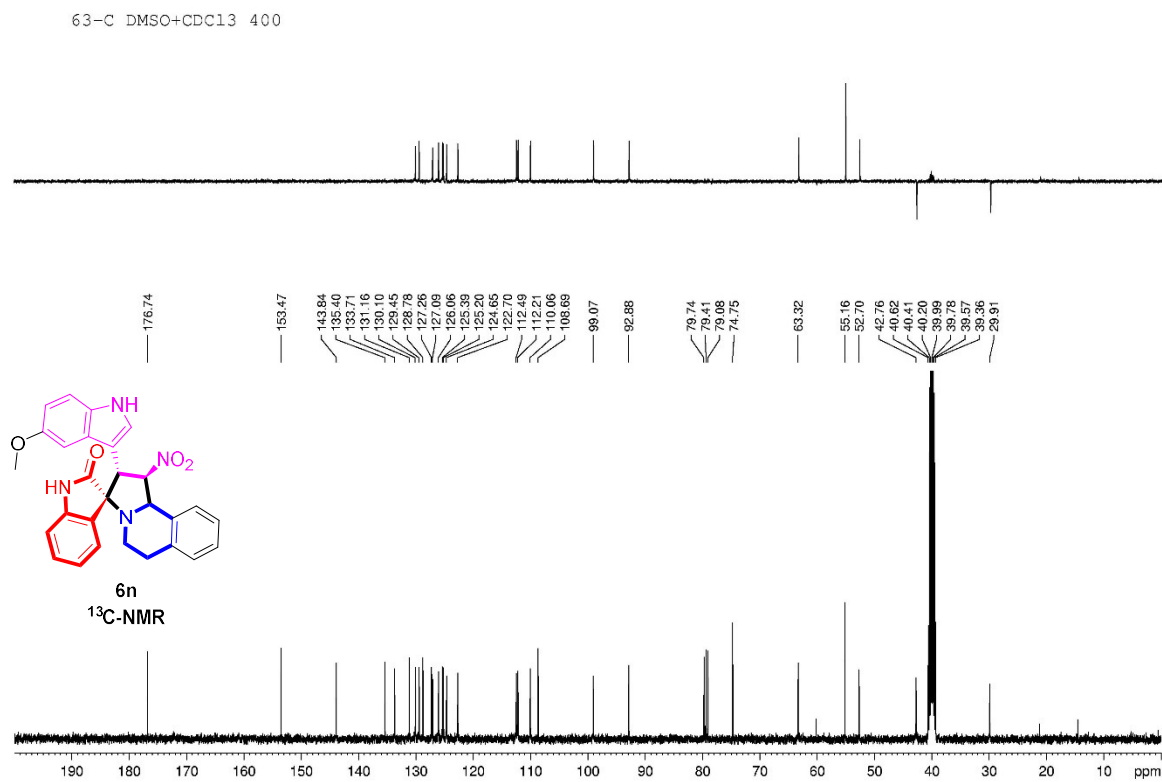
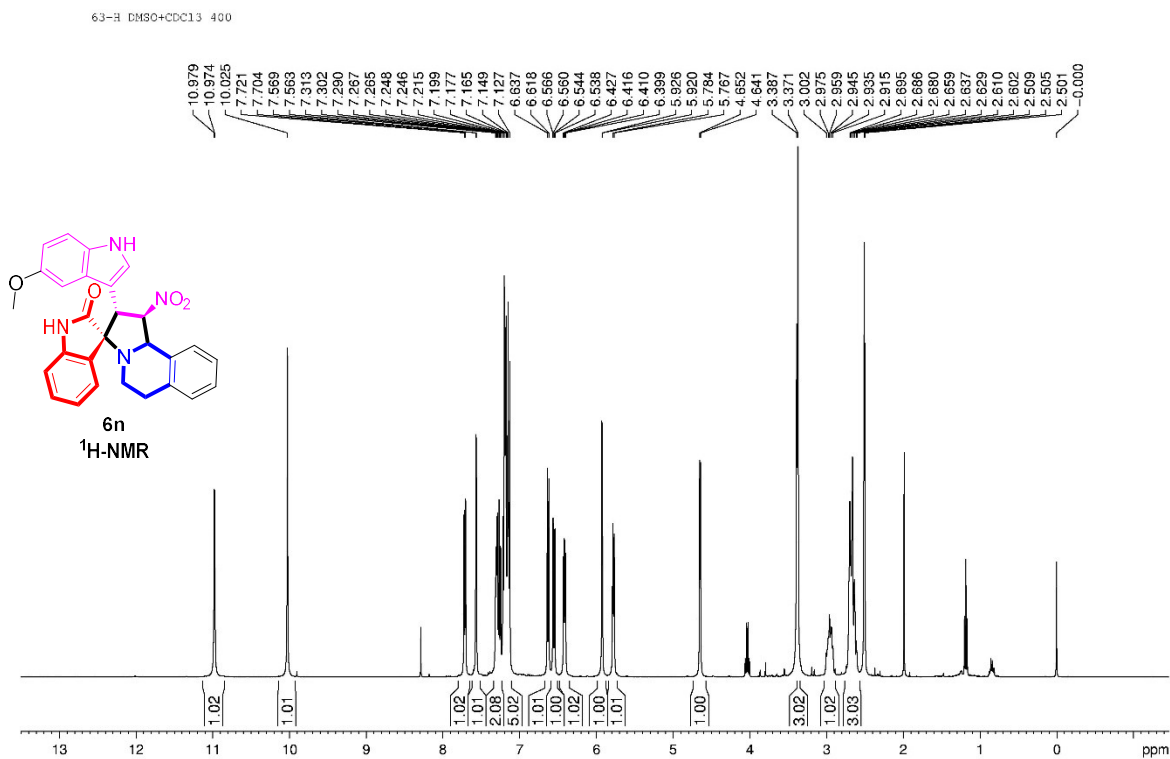


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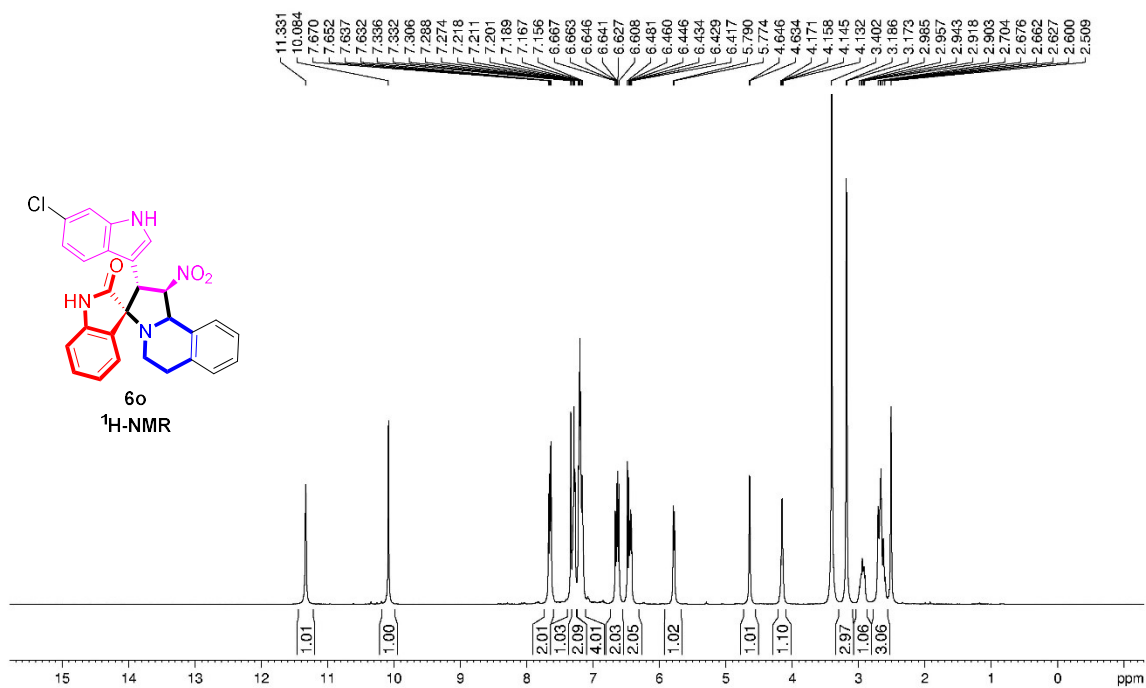




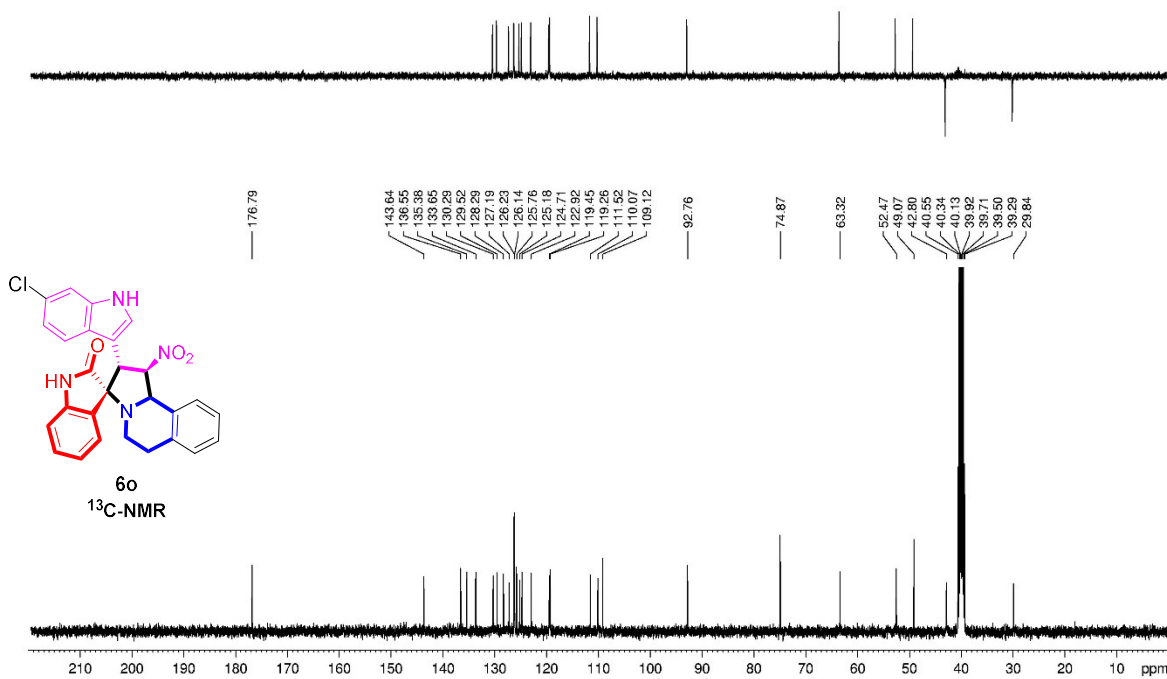


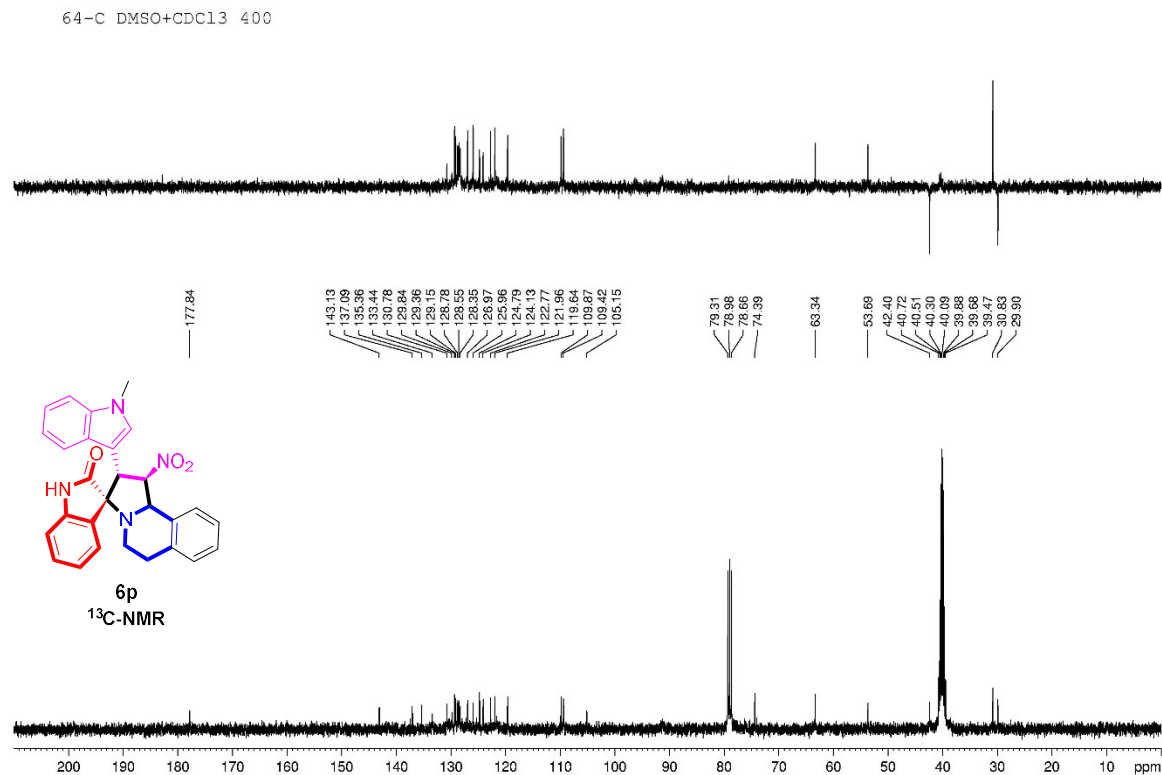
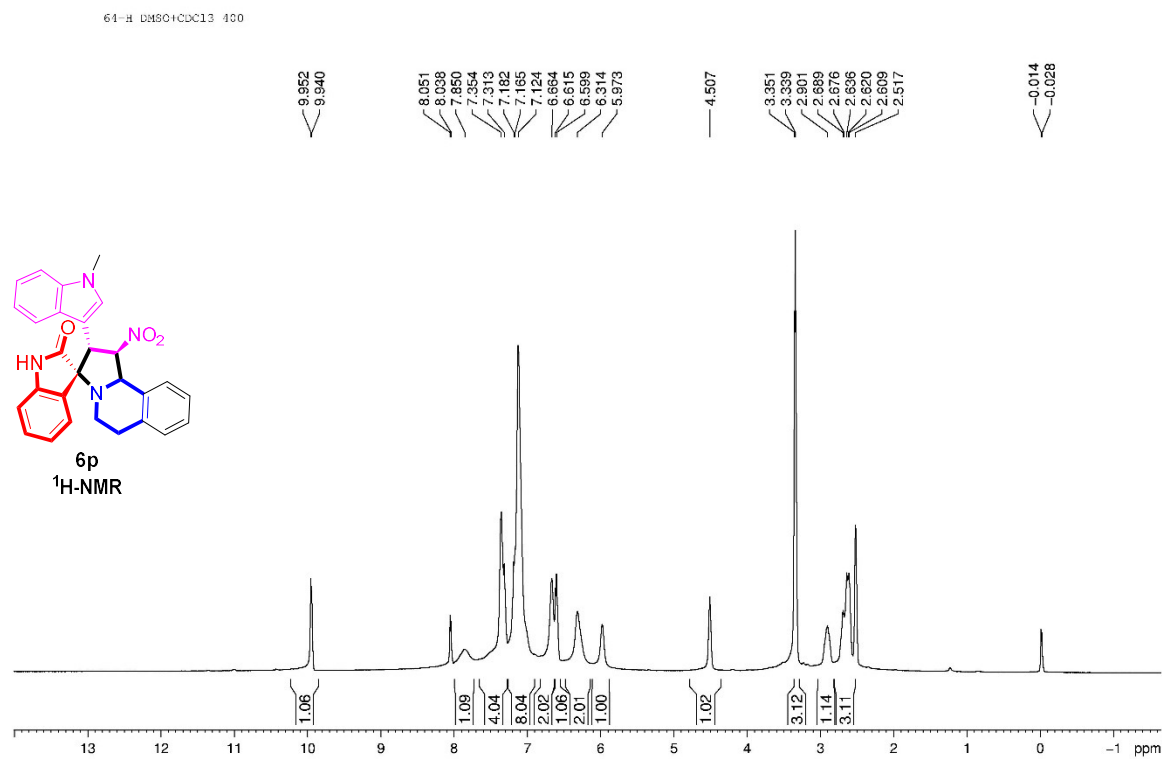


37 DMSO 400

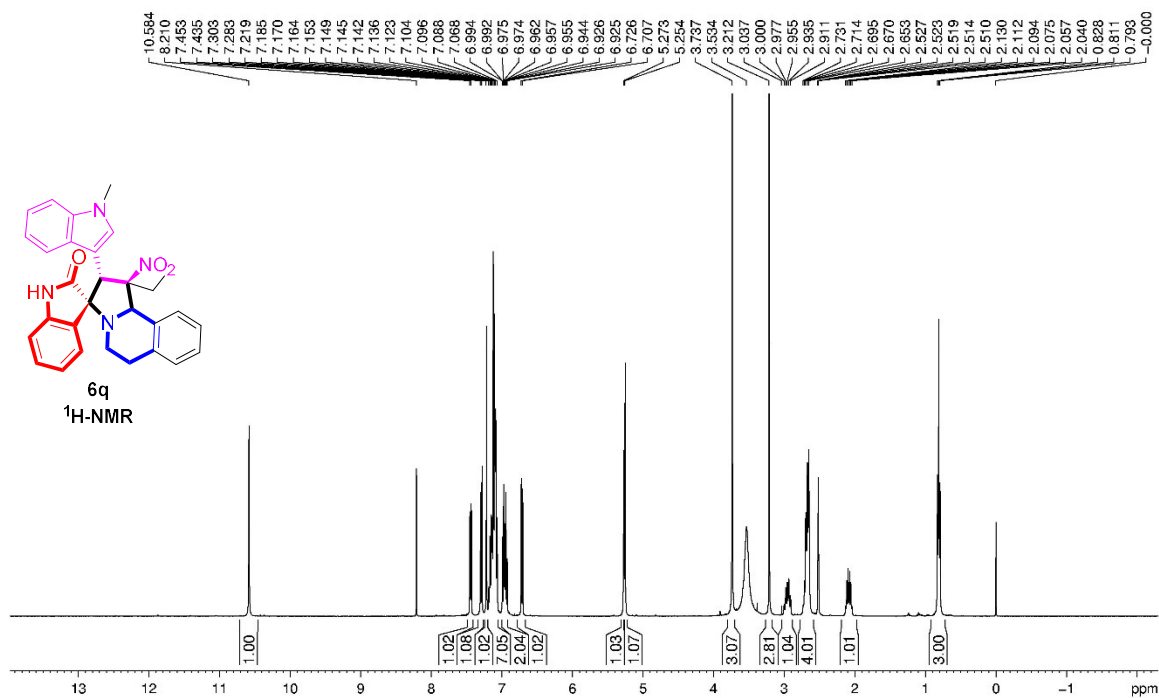


37 DMSO 400

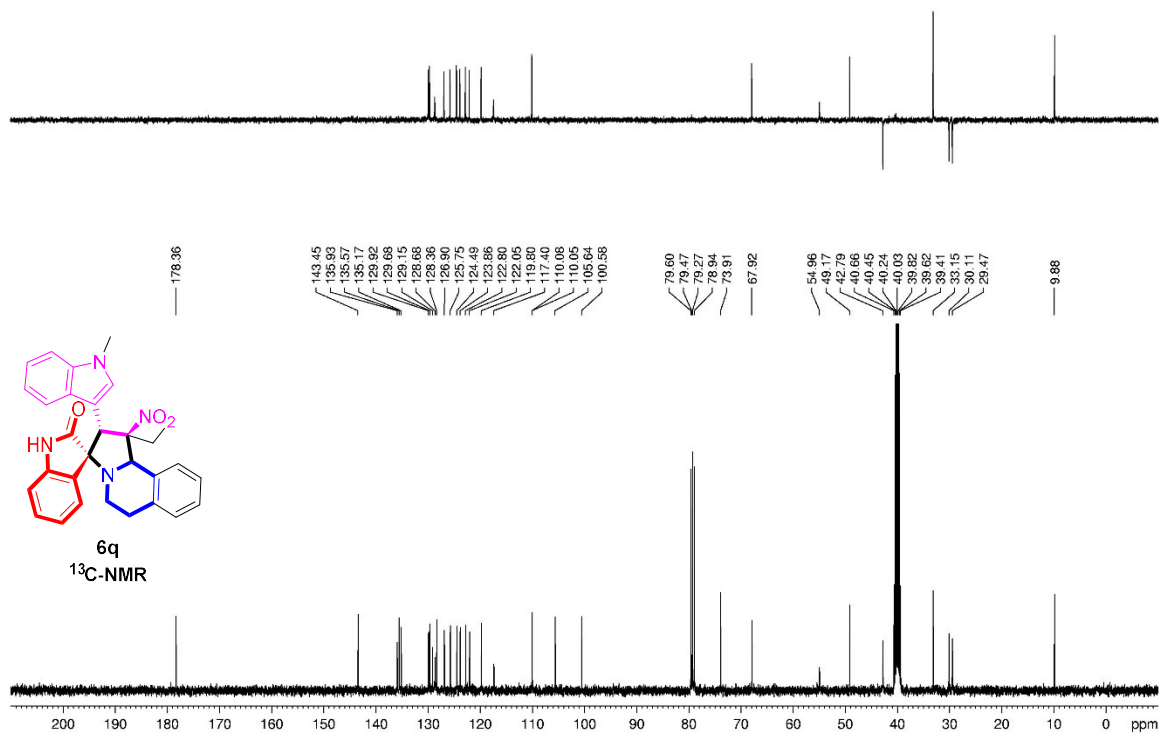


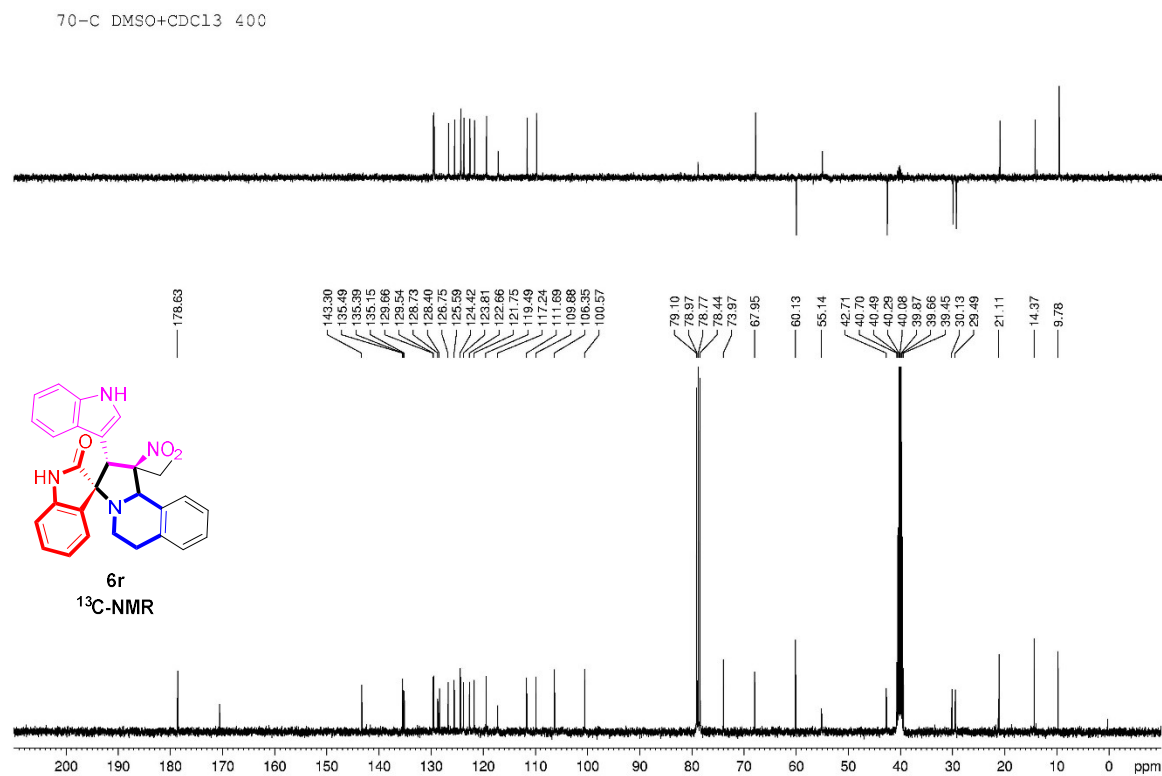
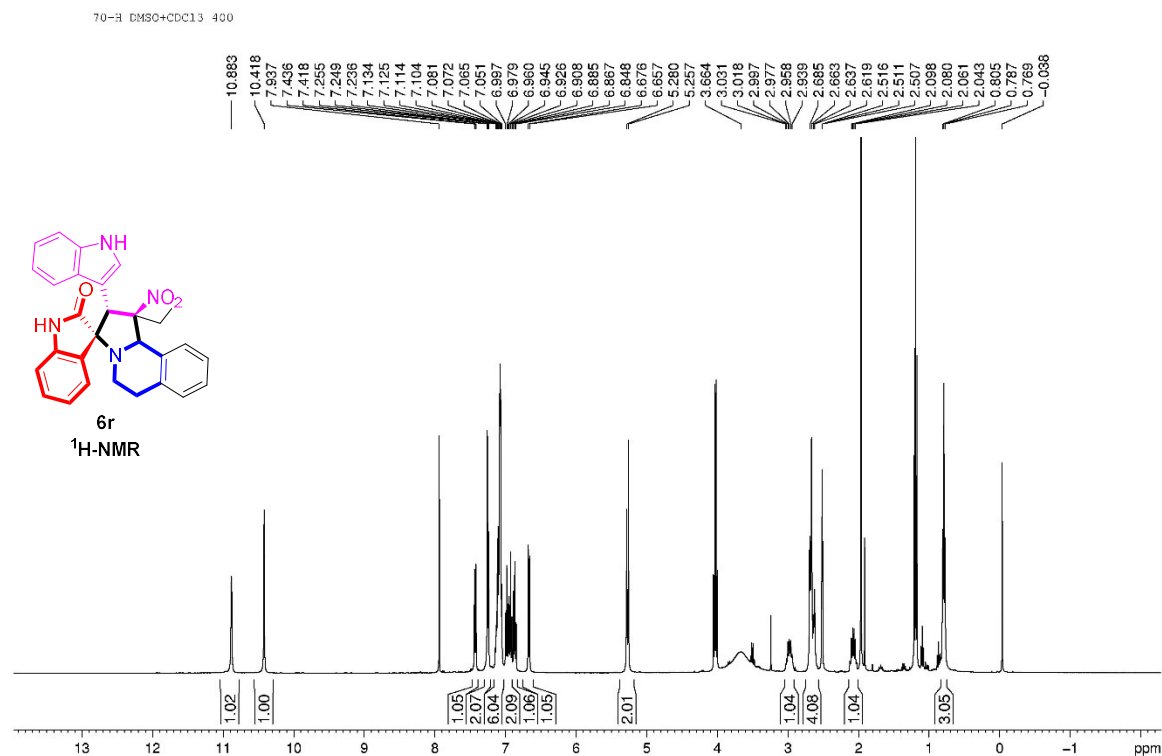


88-II DMSO- d_6 +CDCl₃ 400

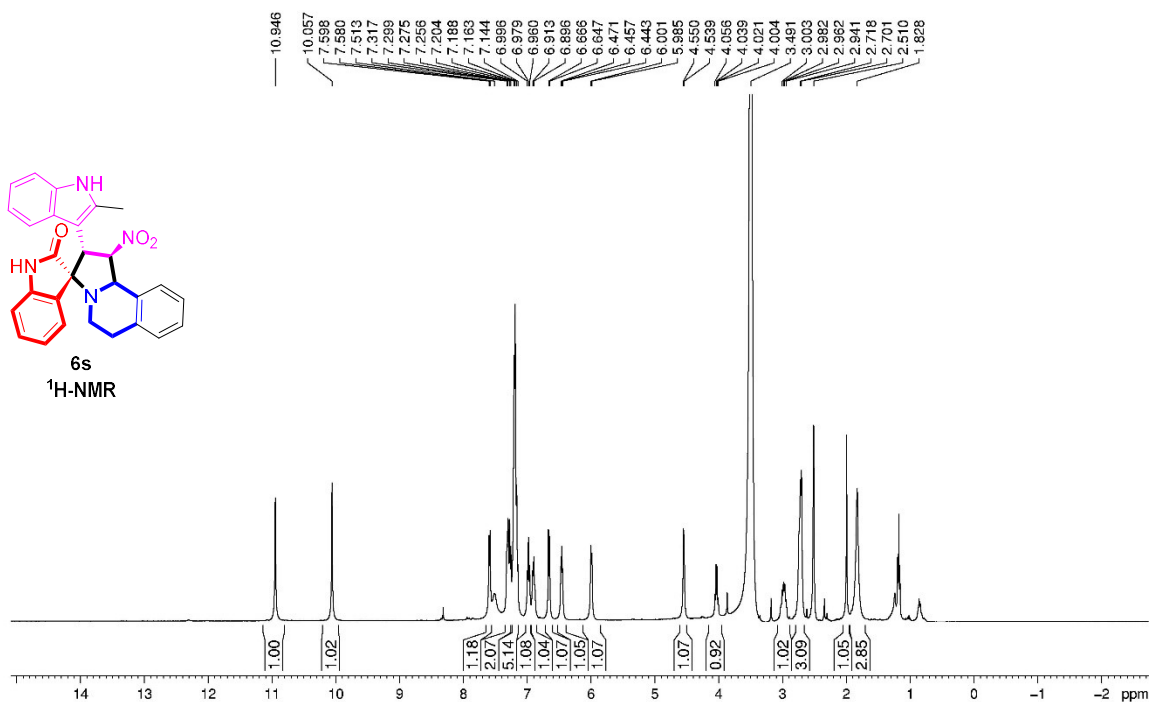


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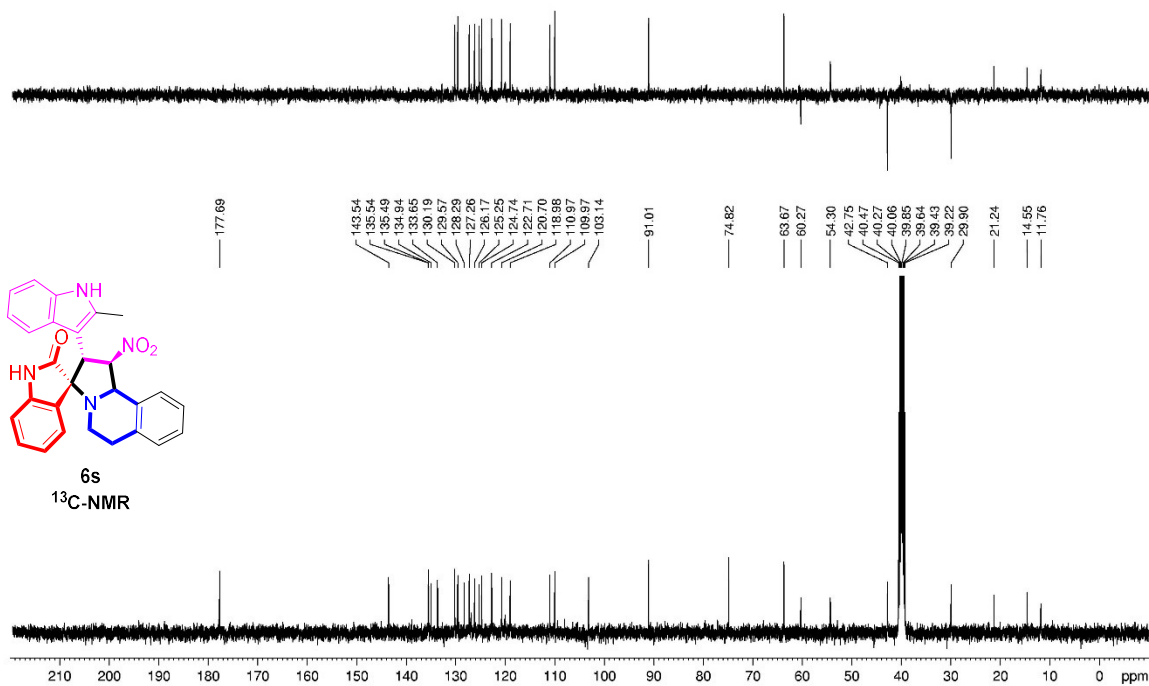


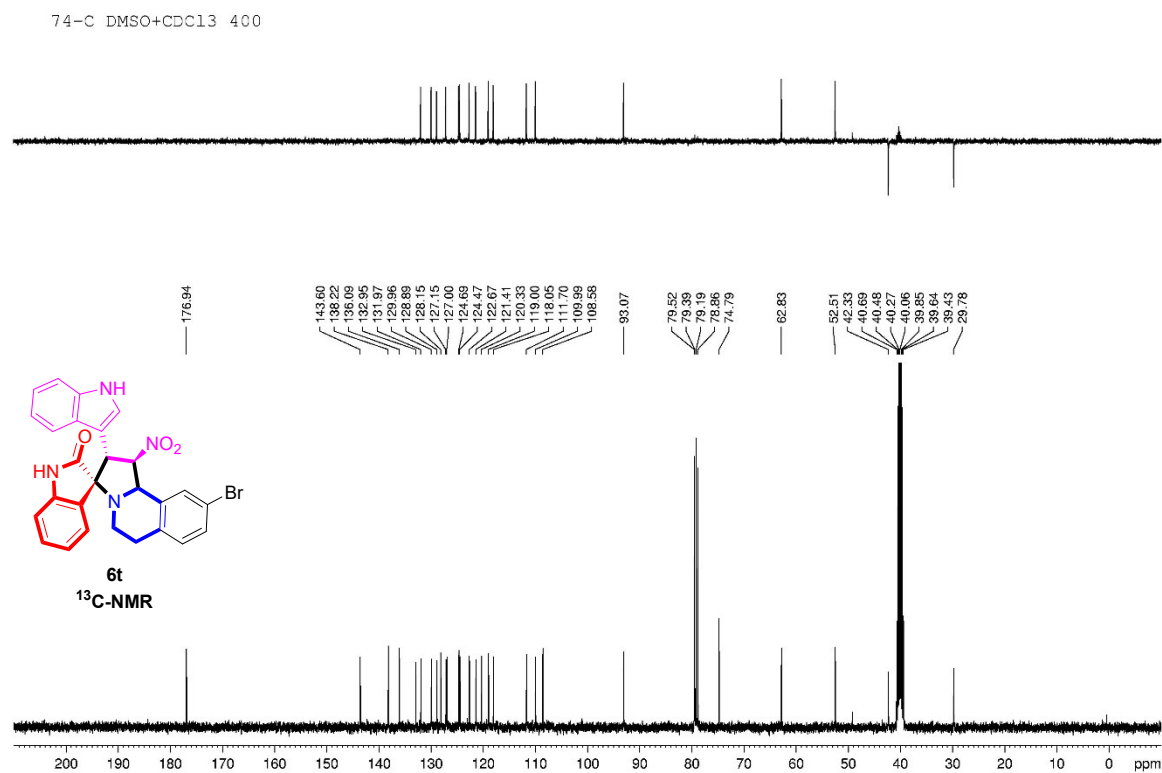
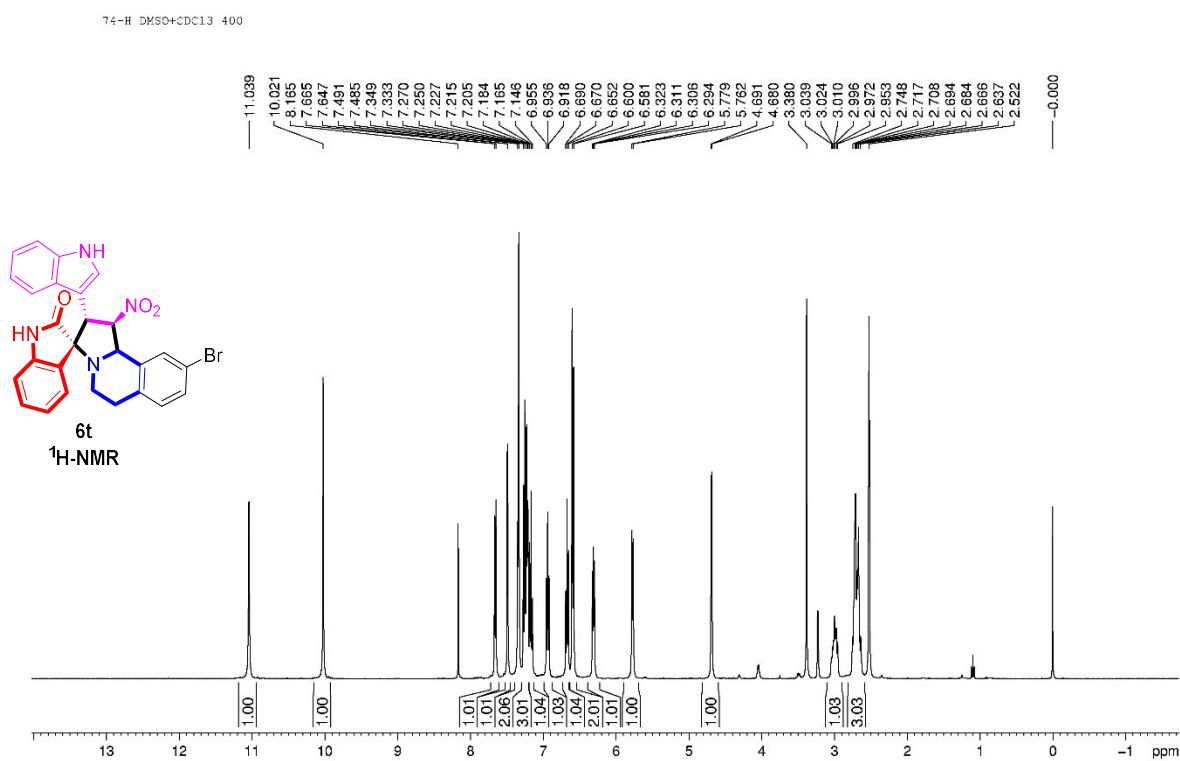


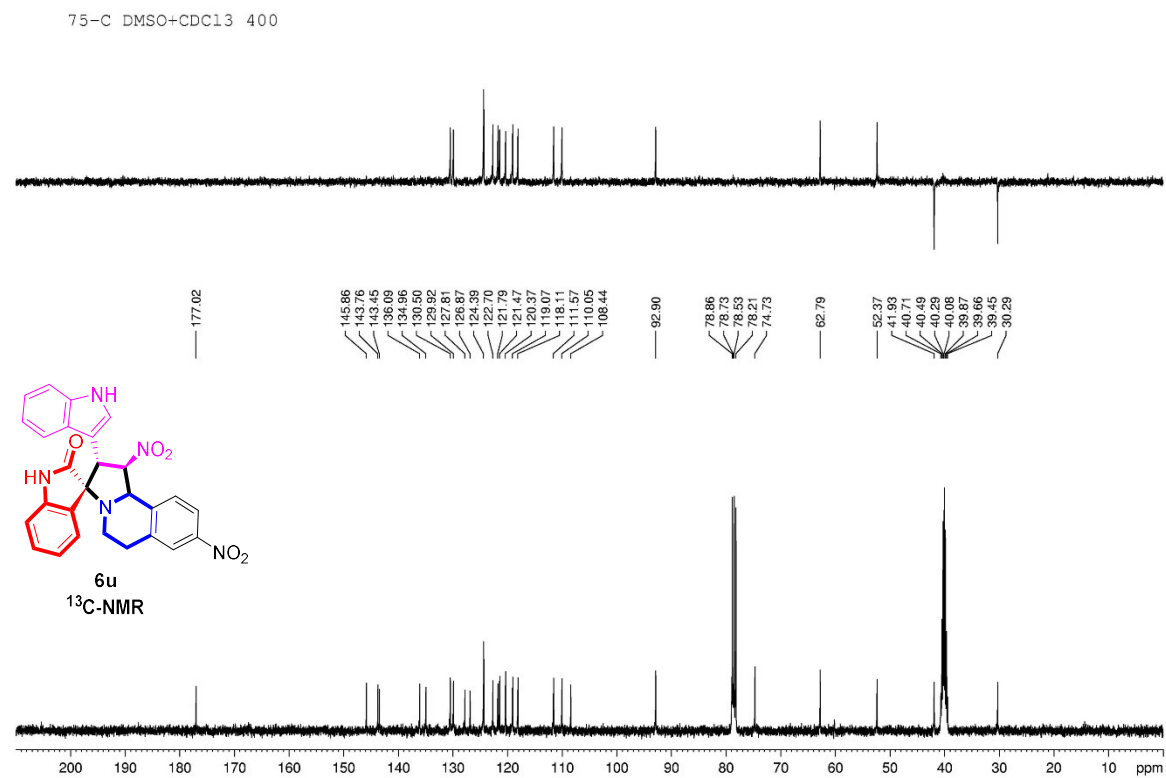
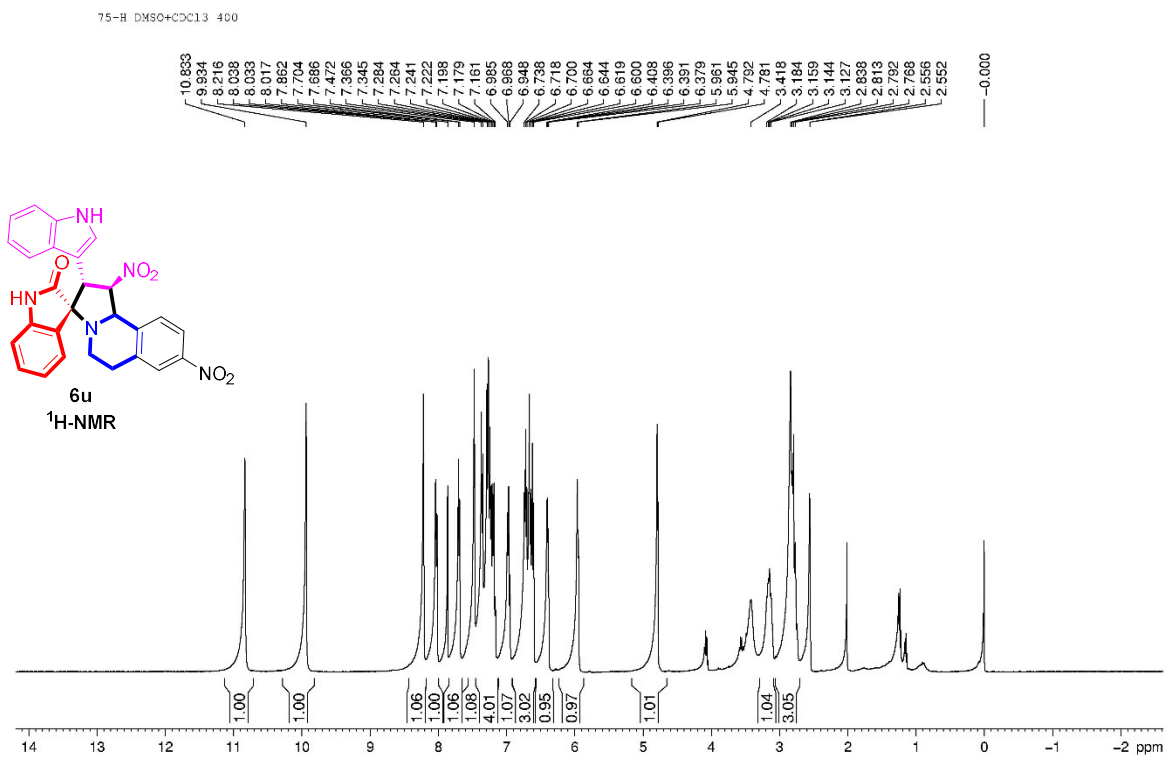
40-H DMSO 400

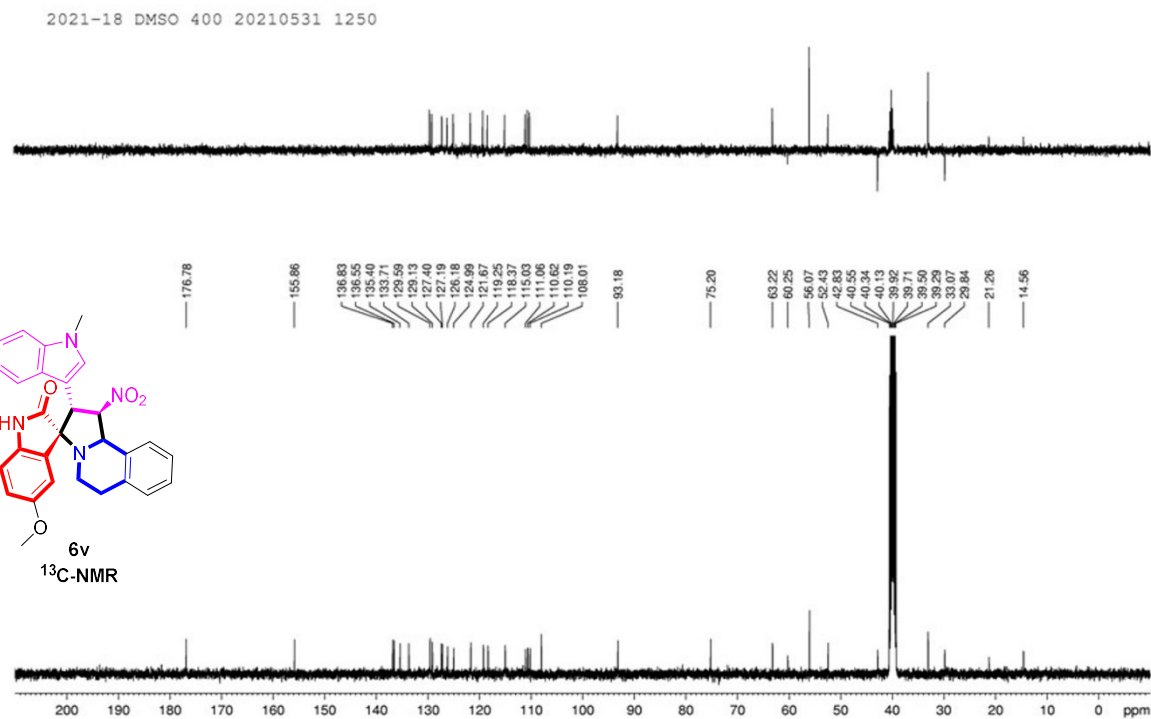
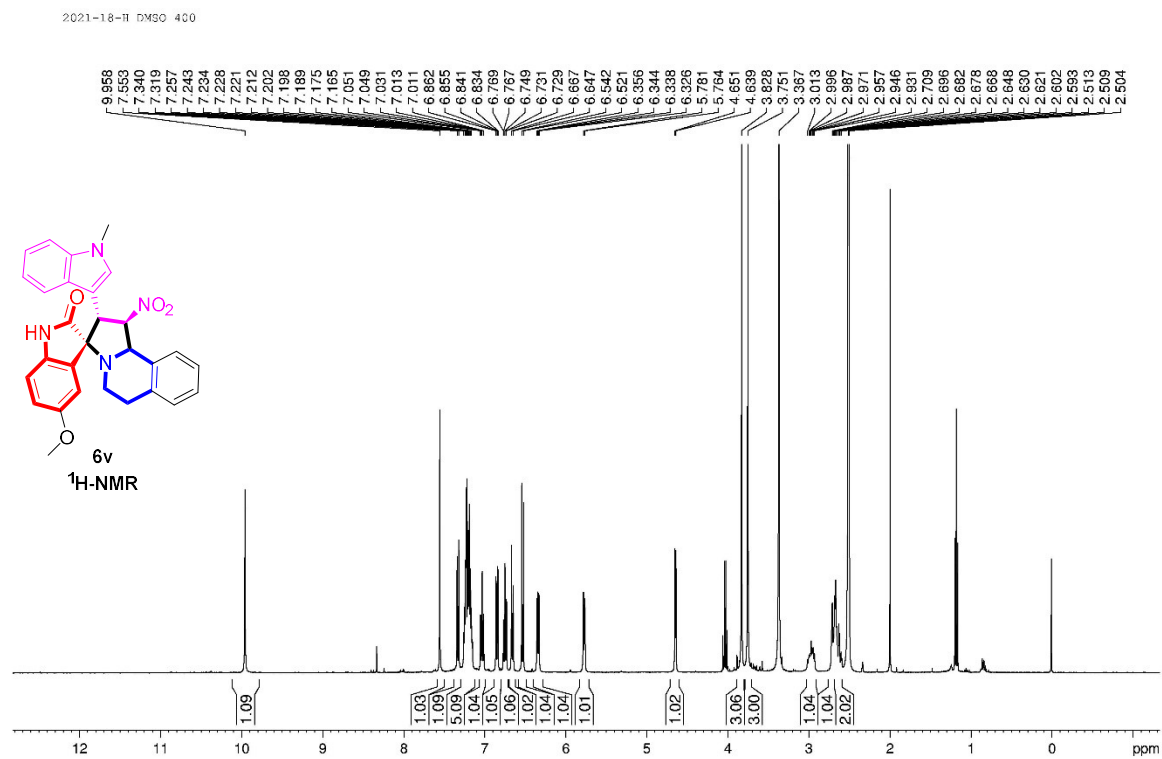


40-C DMSO 400

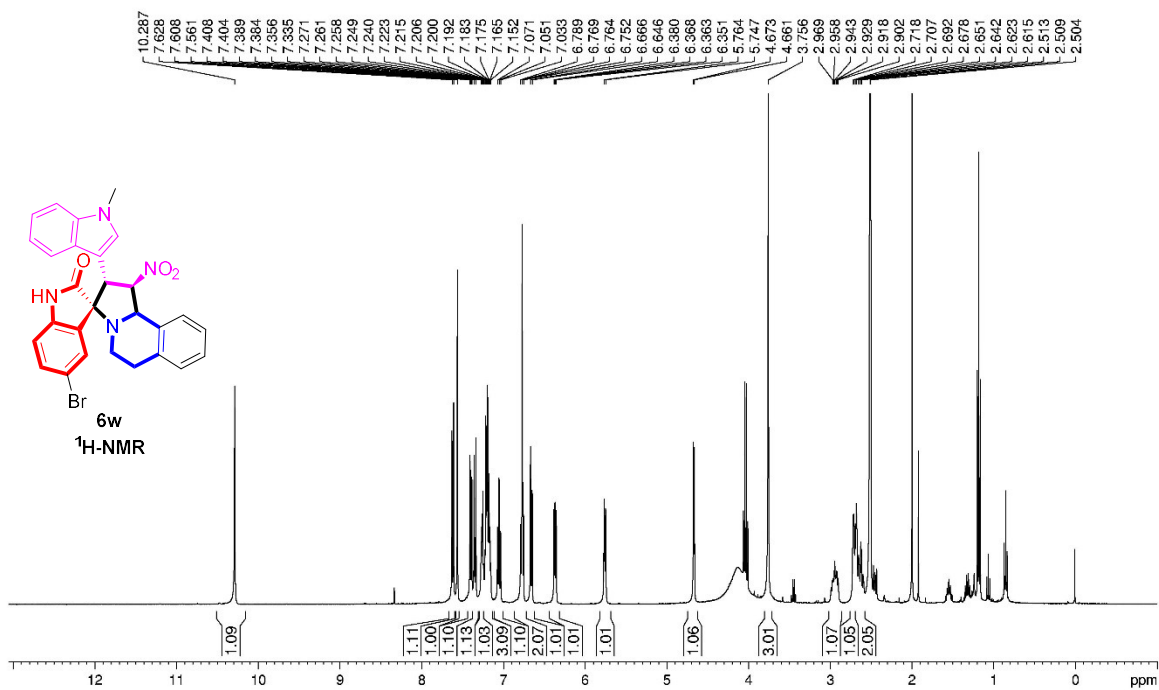




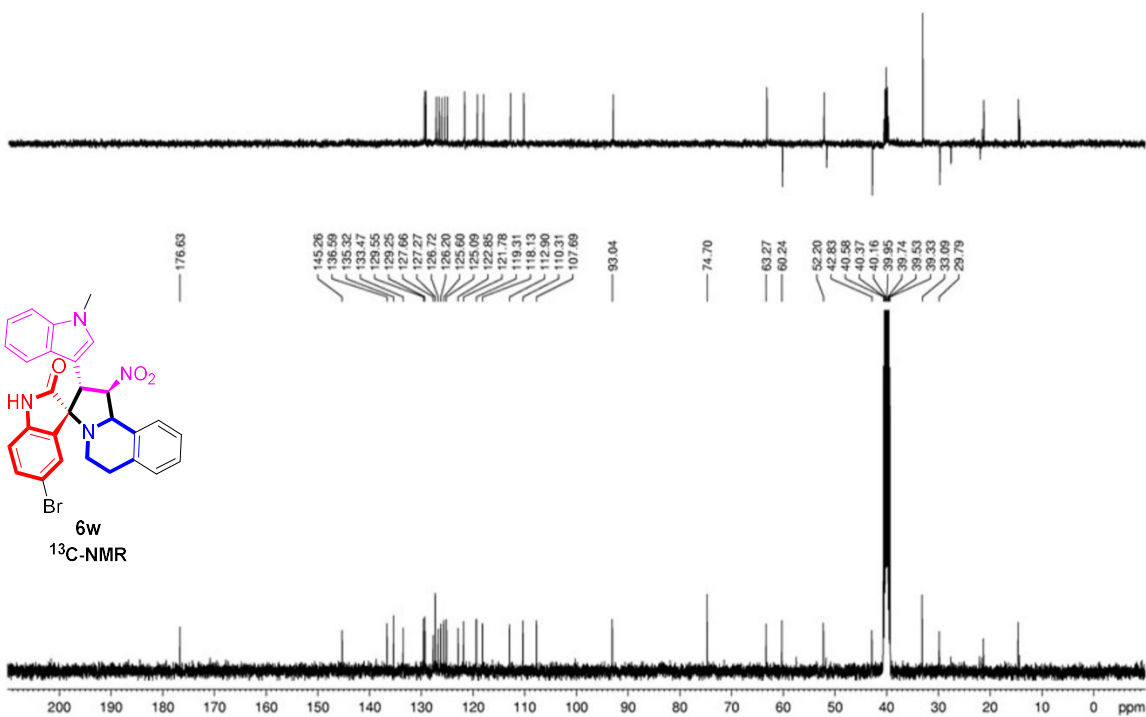


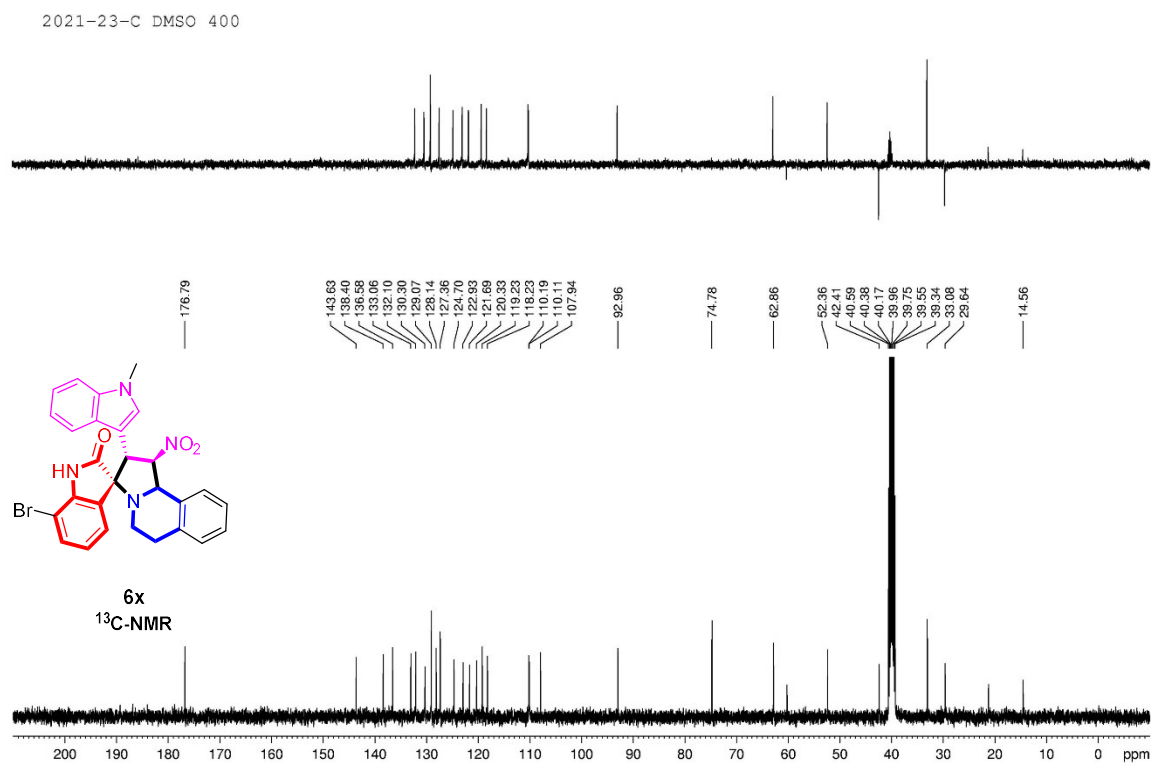
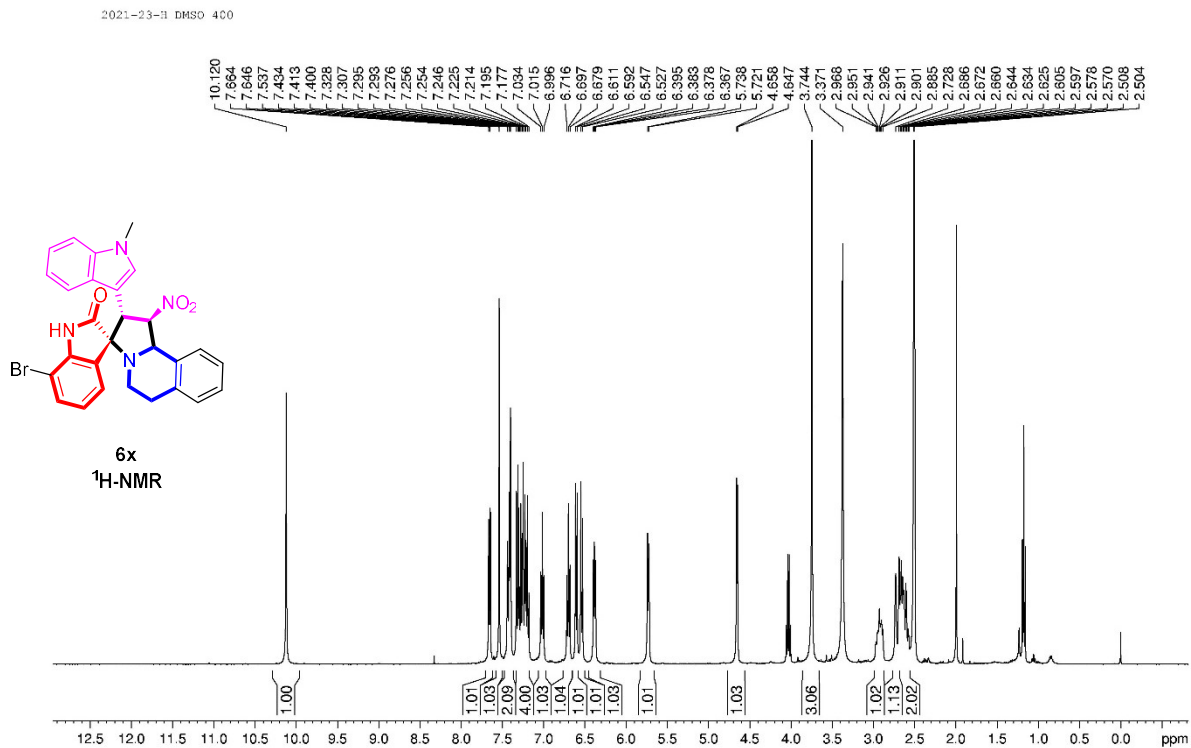


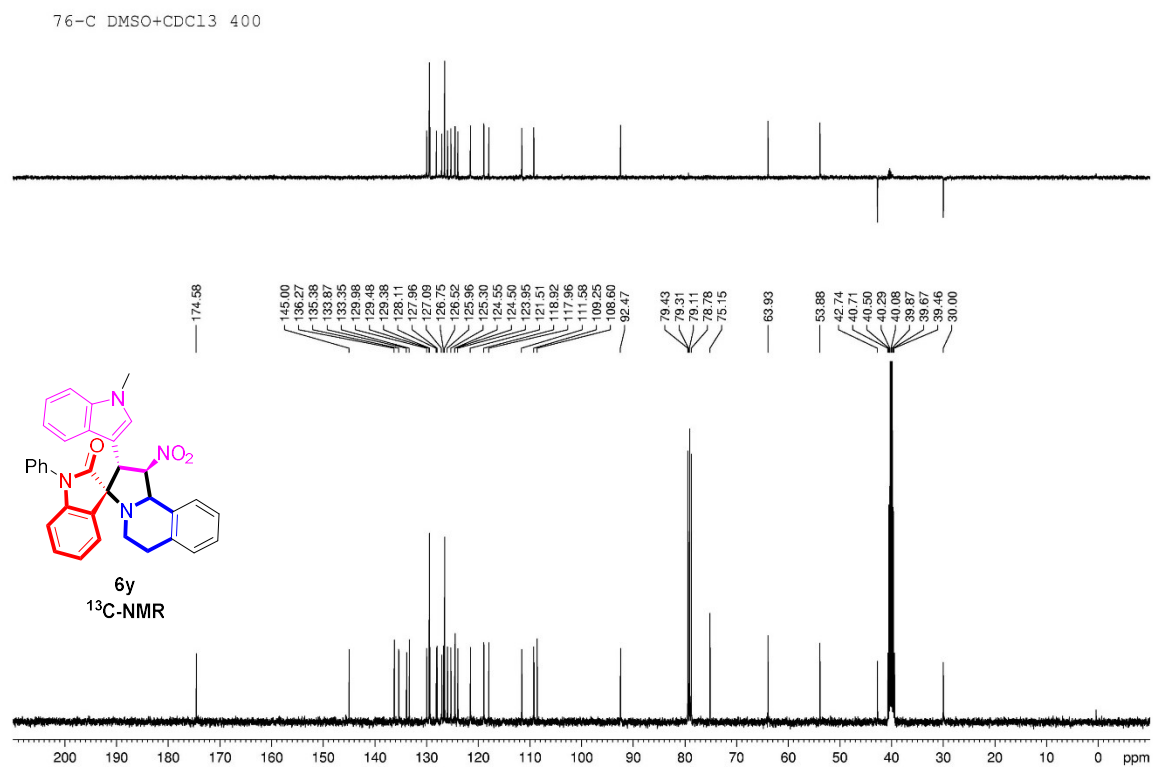
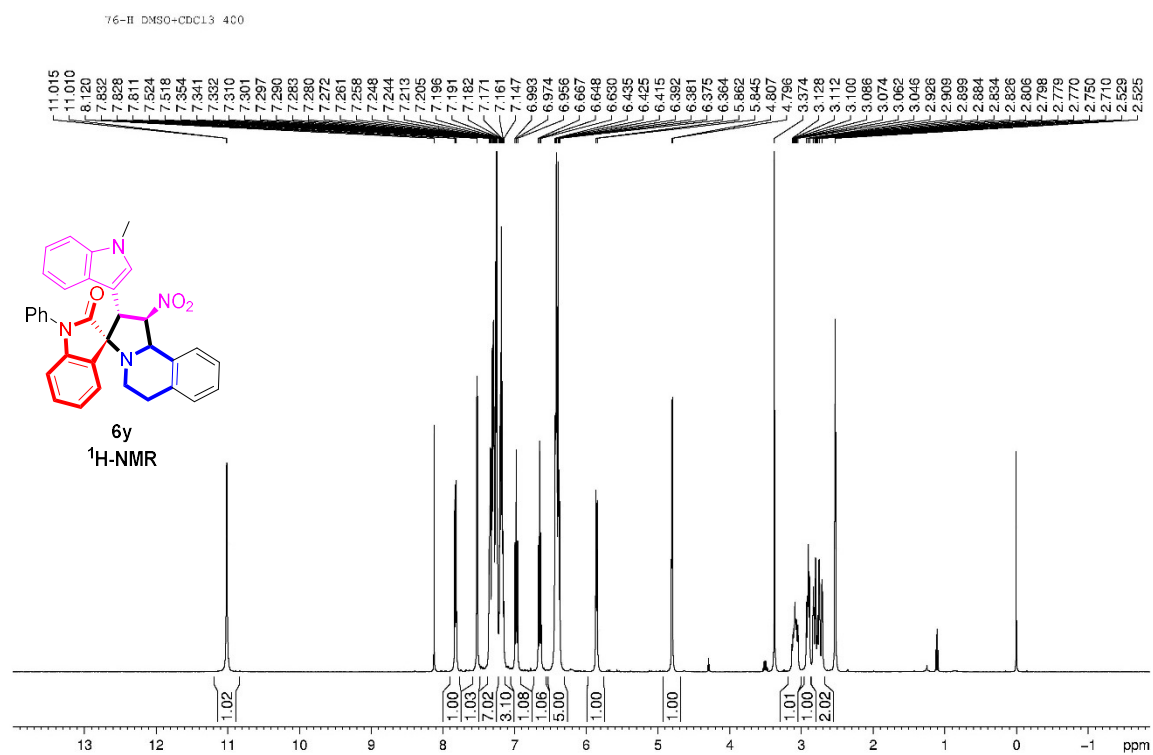
2021-19-H DMSO 400

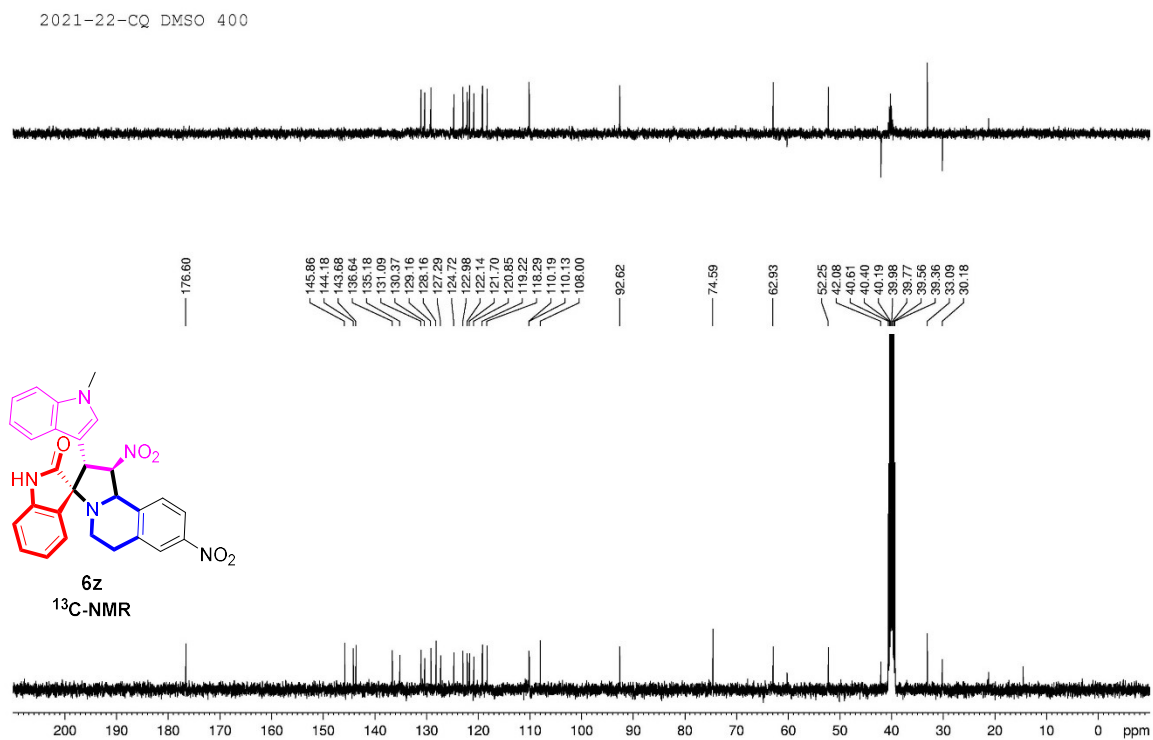
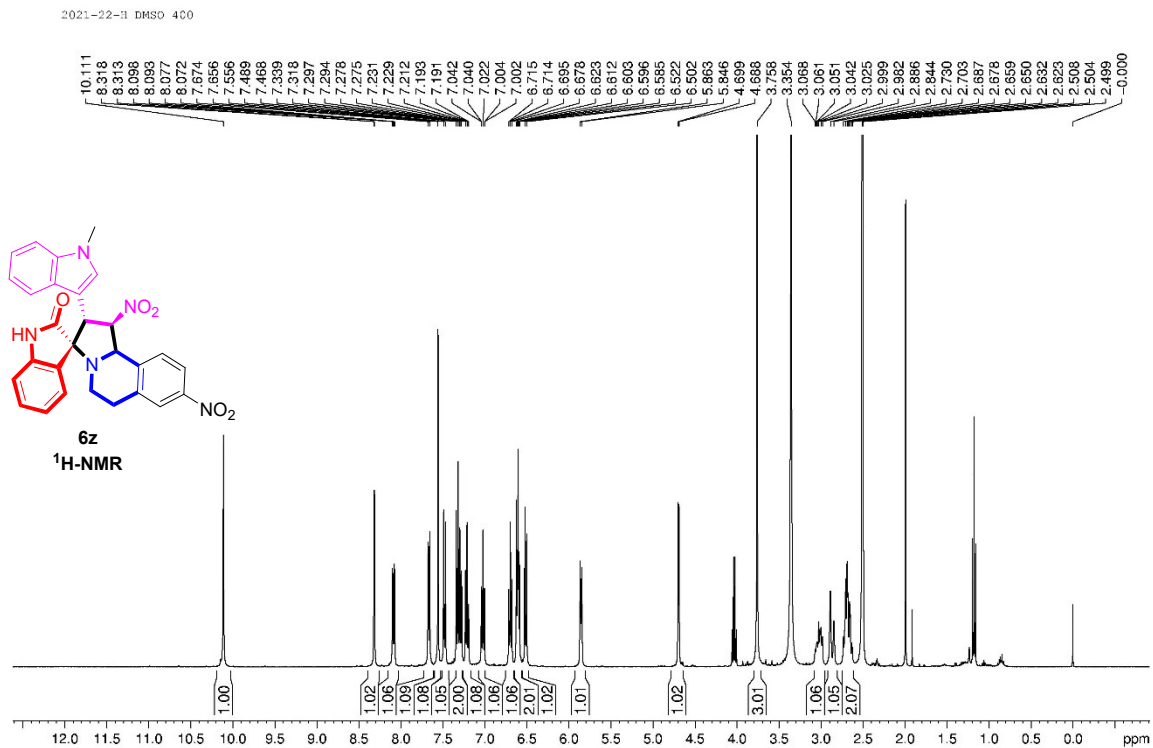


2021-19-C DMSO 400

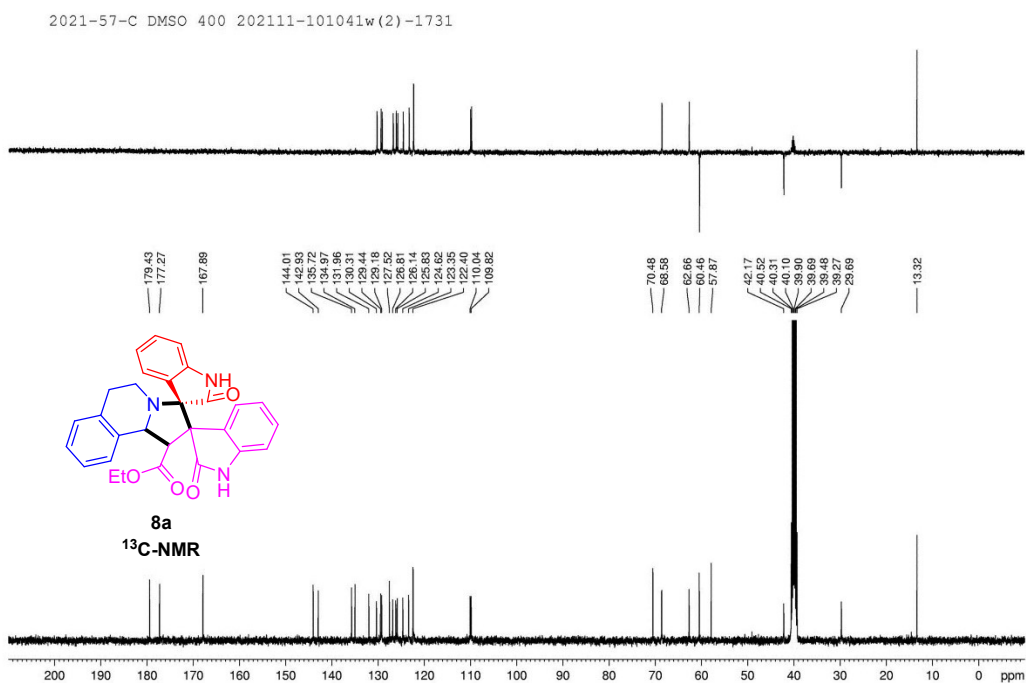
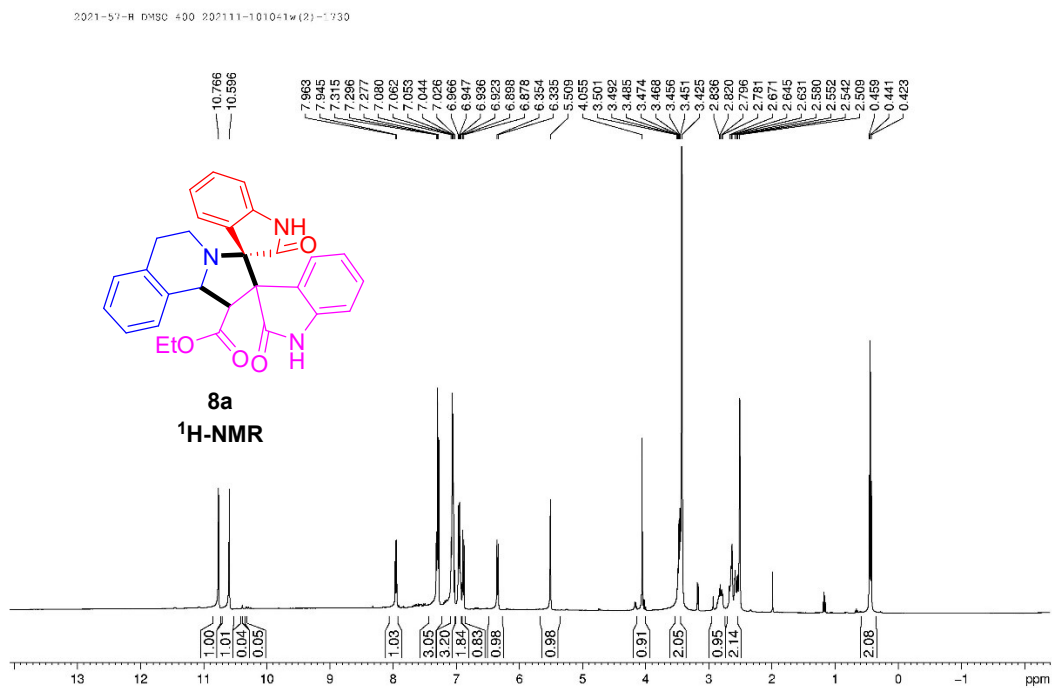


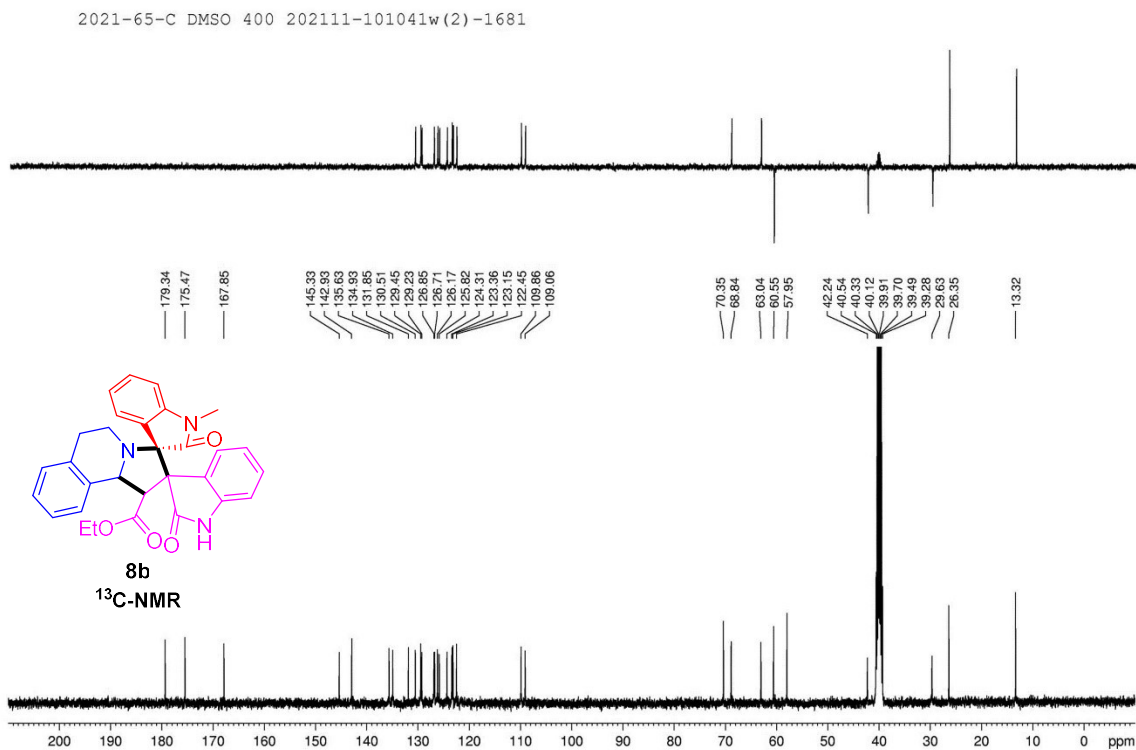
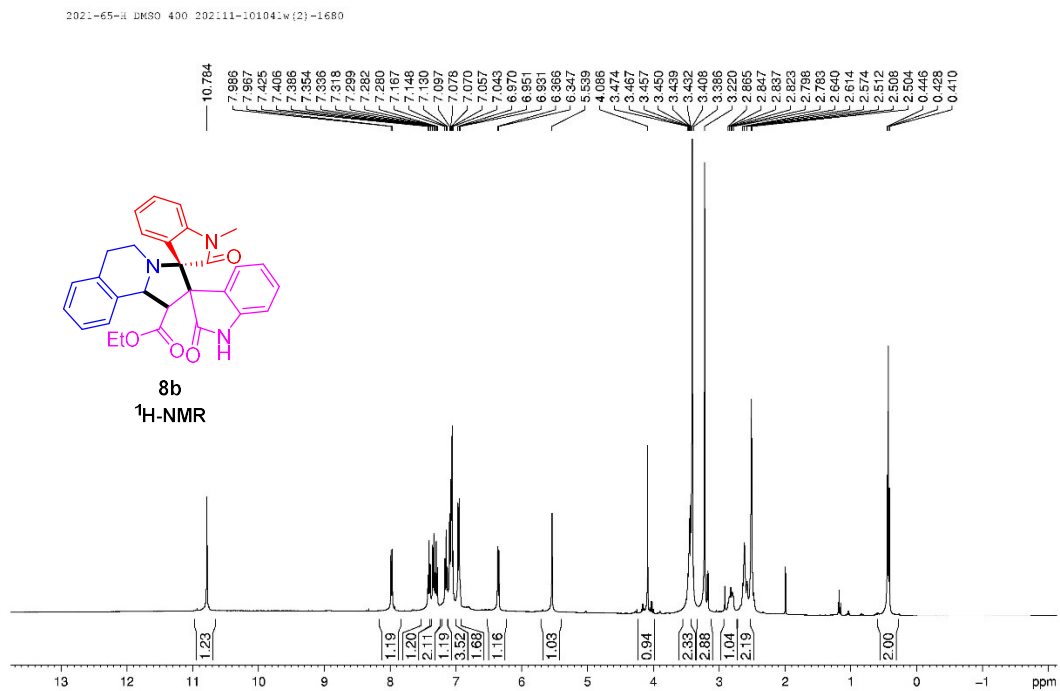




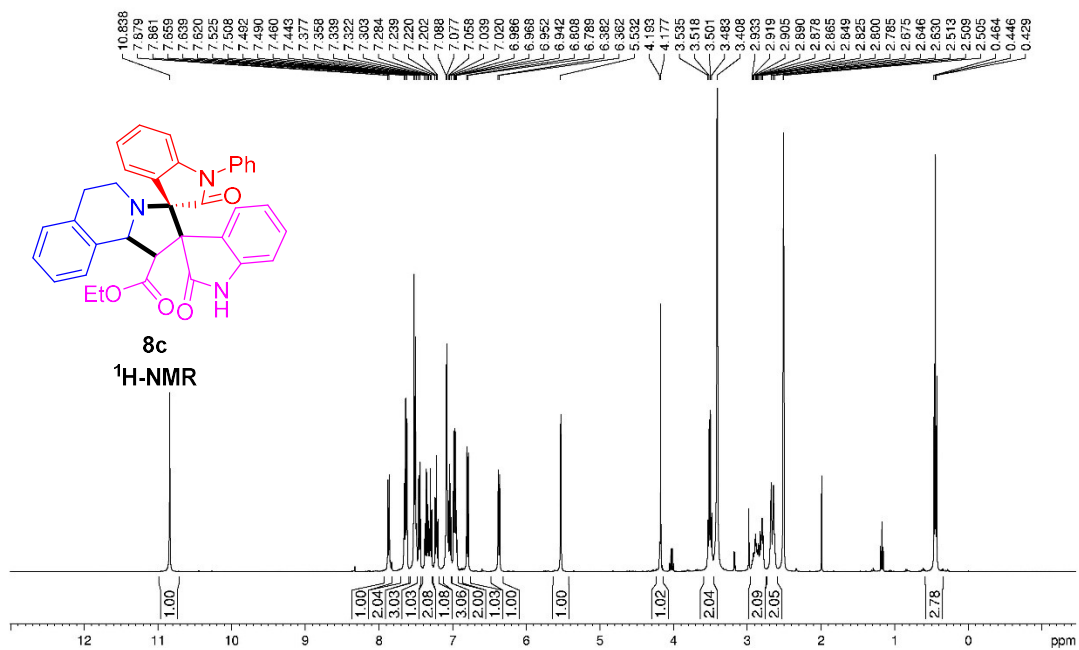


3. ^1H and ^{13}C NMR spectra for compounds **8**

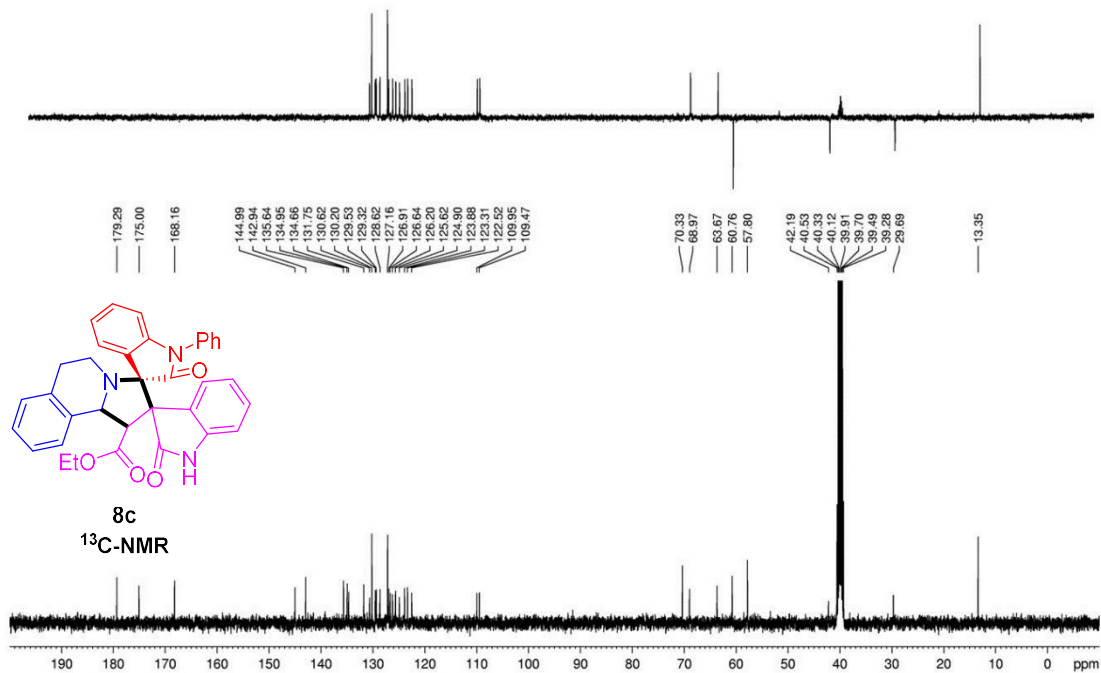


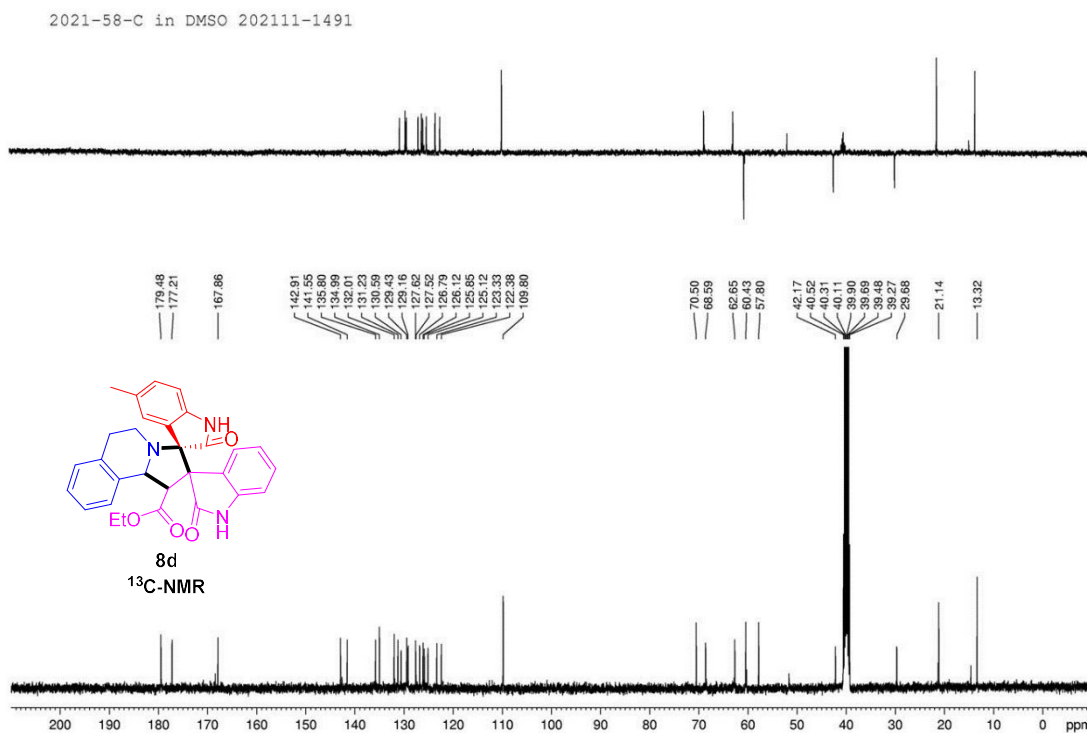
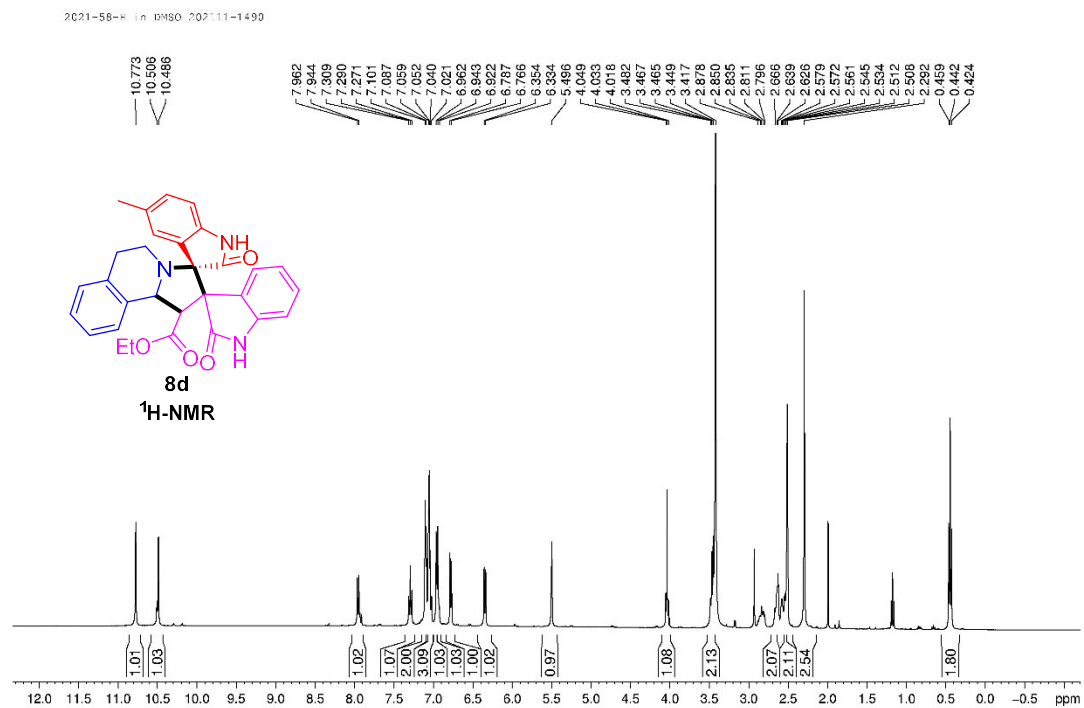


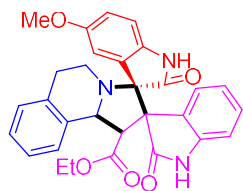
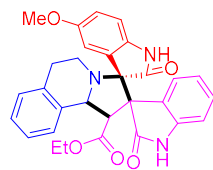
2021-64-H in DMSO 202111-1360



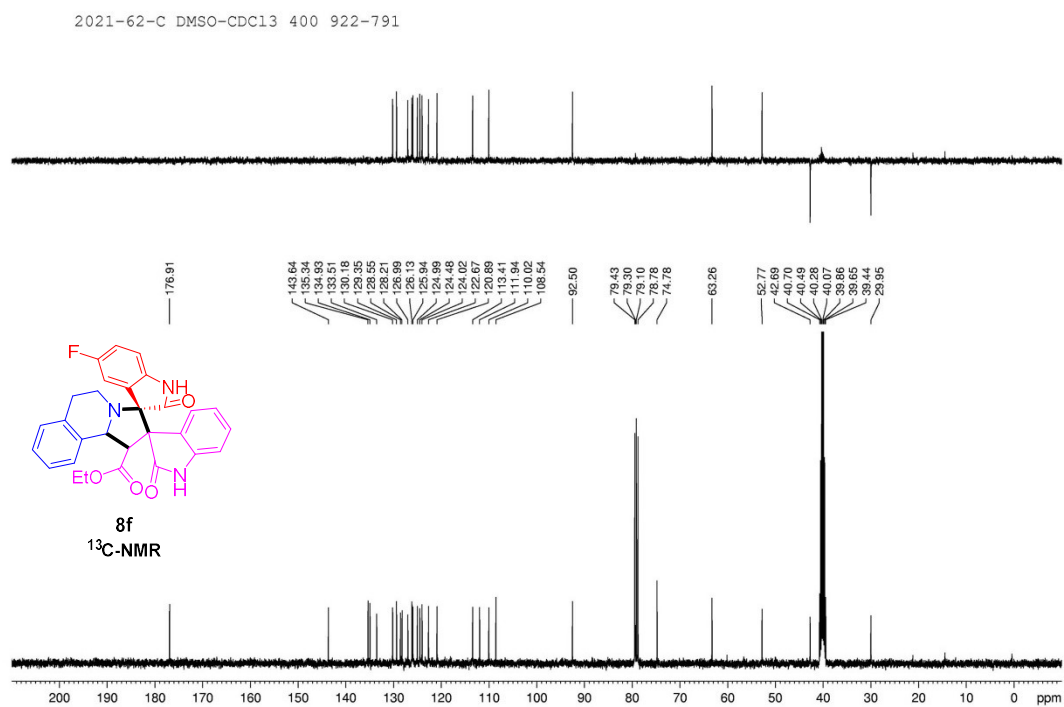
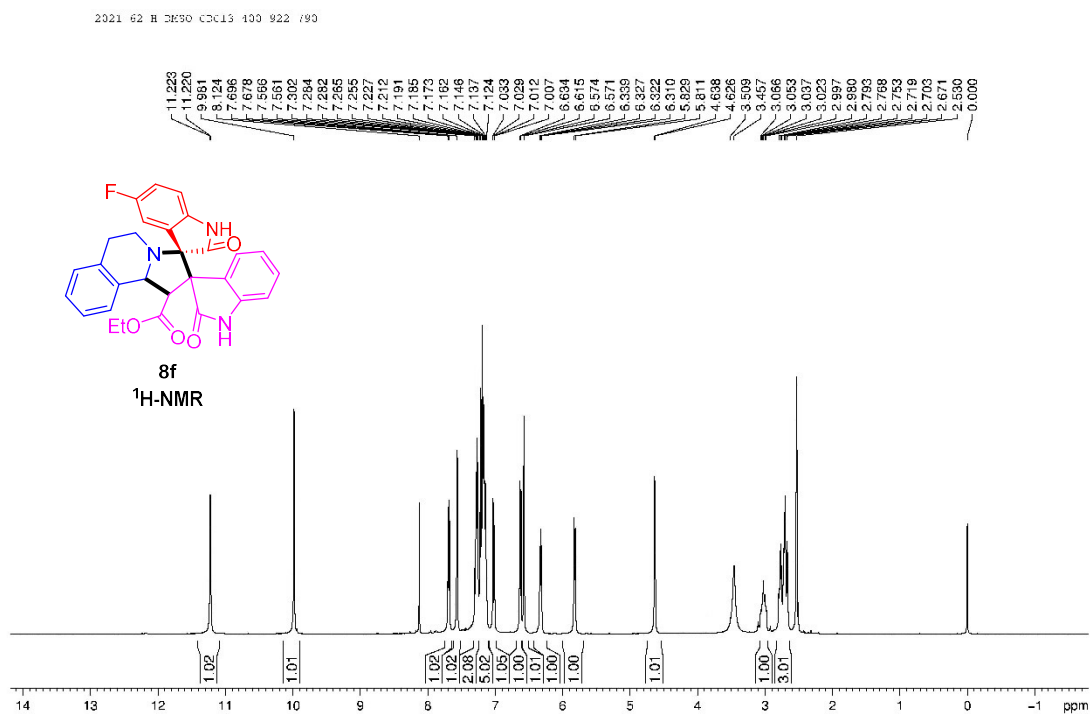
2021-64-C in DMSO 202111-1361



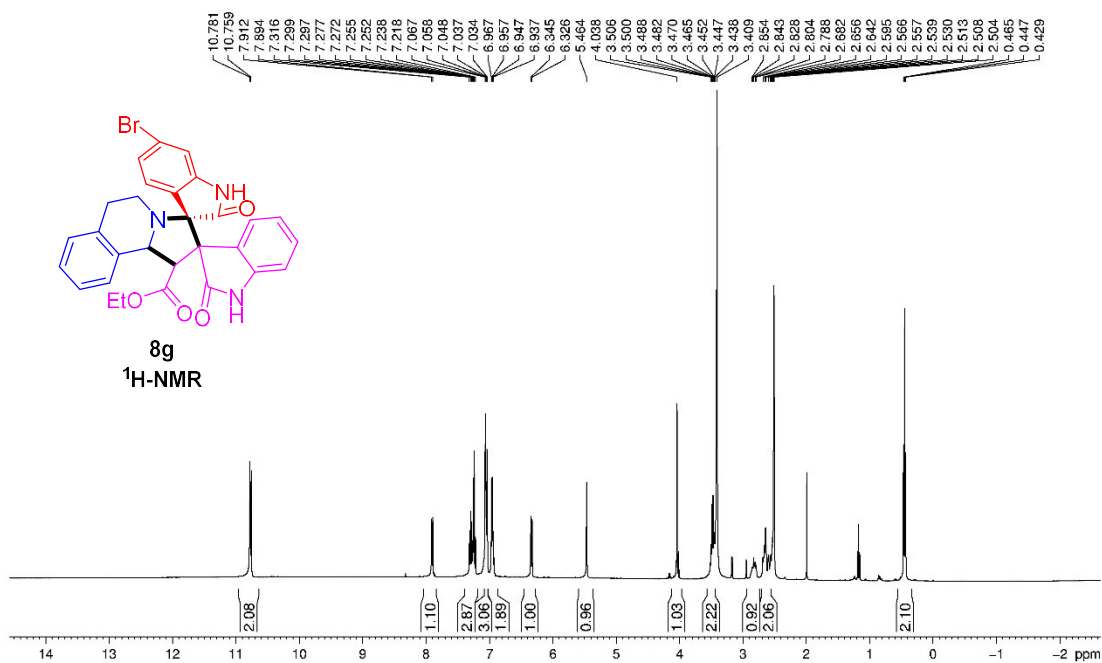


¹H-NMR

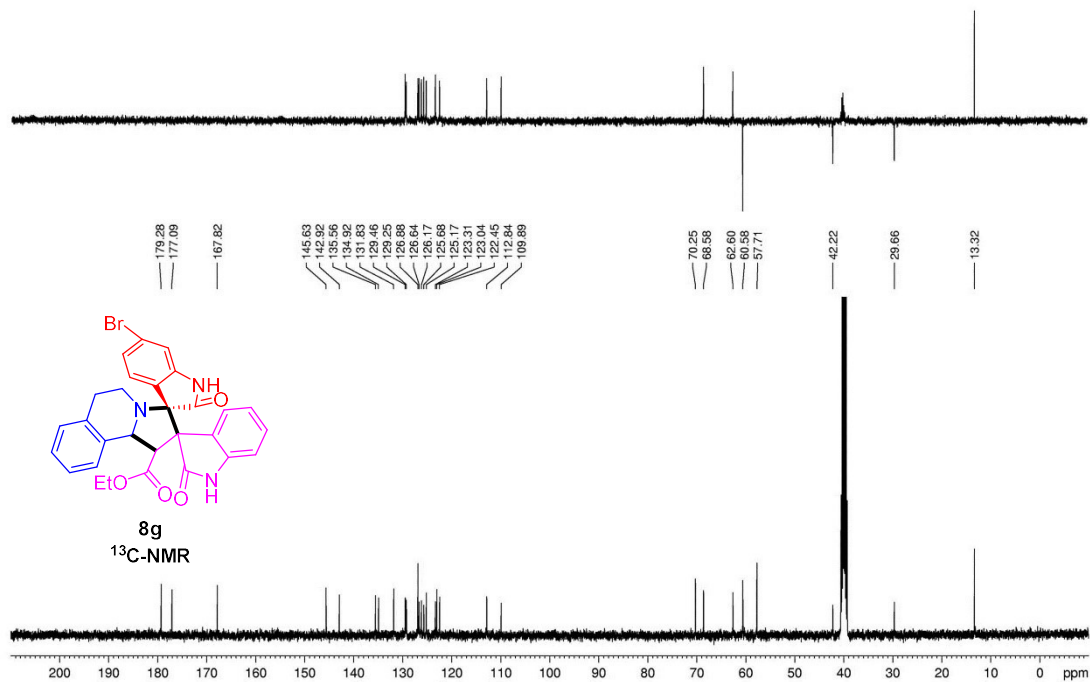
8e
¹³C-NMR

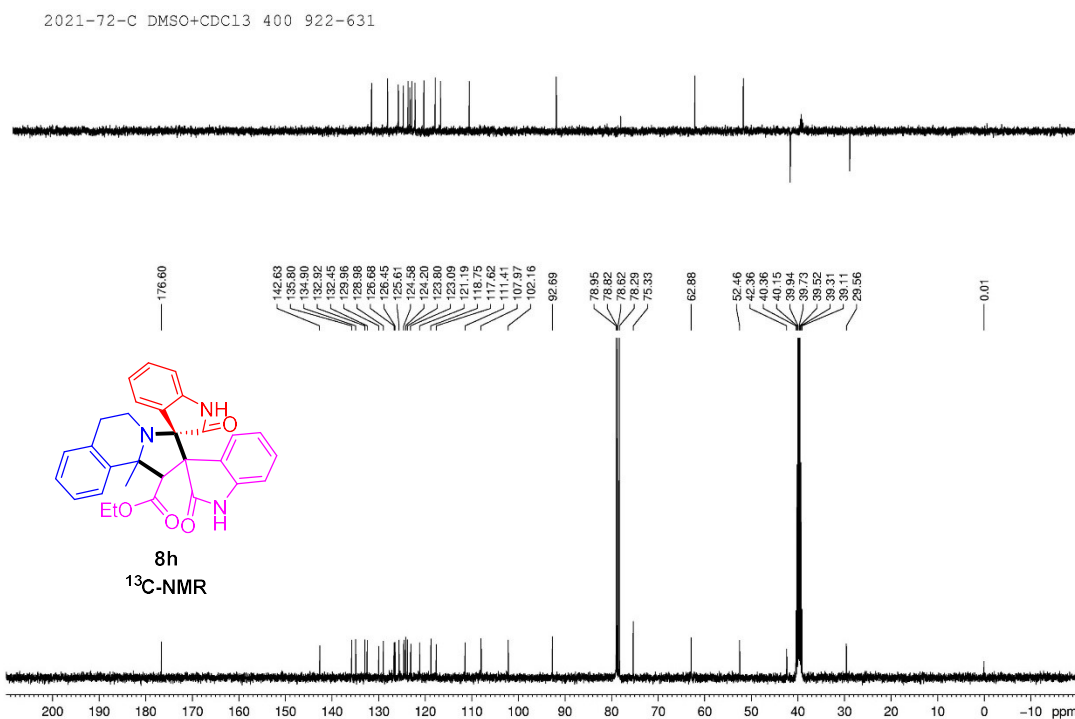


2021-60-H DMSO 400 202111-101041w(2)-1690

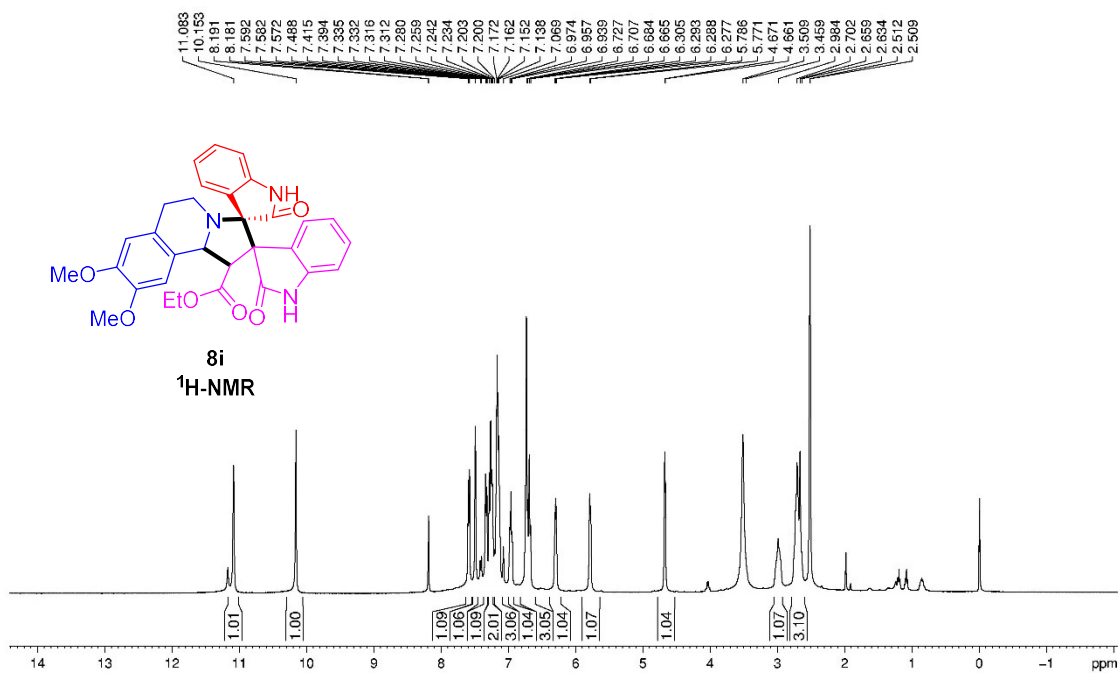


2021-60-C DMSO 400 202111-101041w(2)-1691

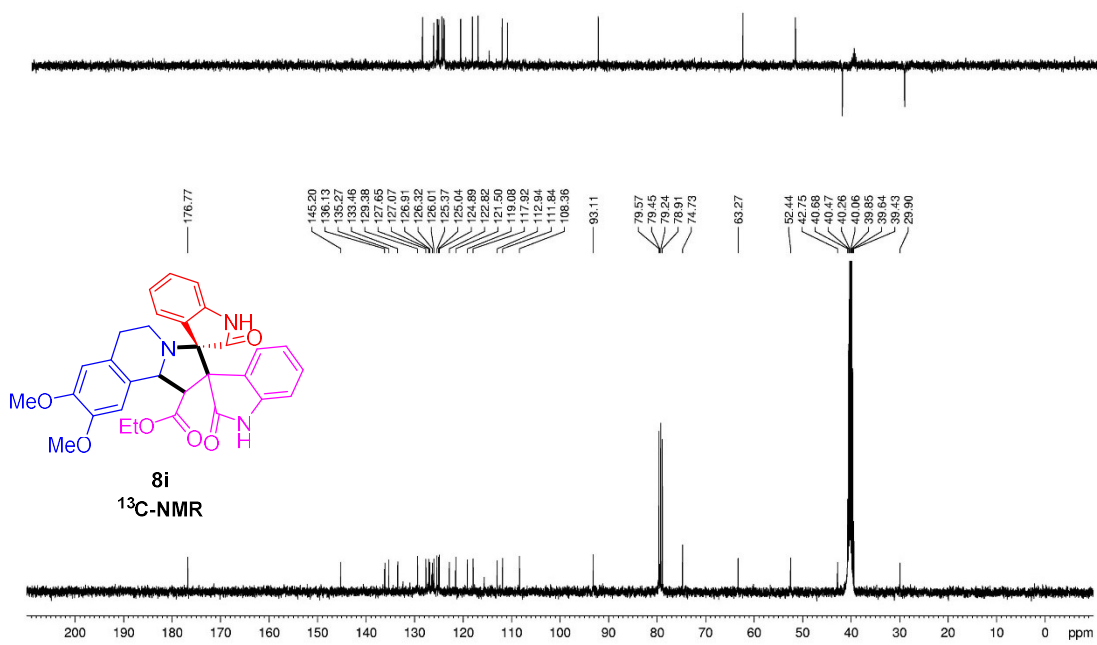


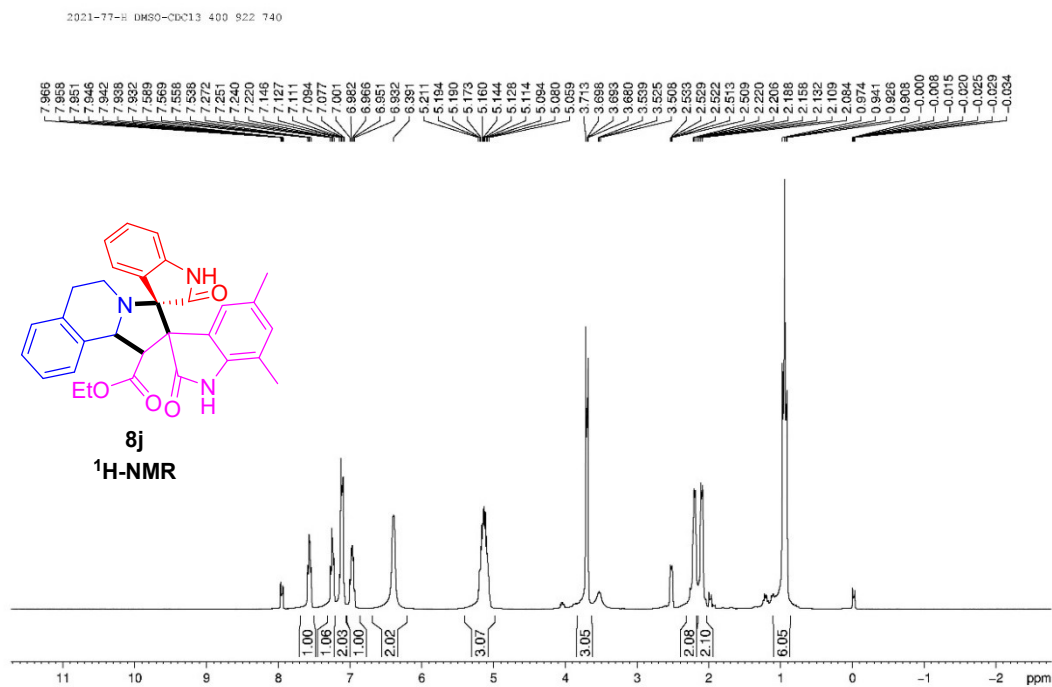


2021-71-H DMSO-CDCl₃ 400 922-870



2021-71-C DMSO-CDCl₃ 400 922-871





3. Single crystal X-ray diffraction study data of compound 4c

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) exp_2483_auto

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found.	CIF dictionary	Interpreting this report
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Datablock: exp_2483_auto

Bond precision: C-C = 0.0029 Å Wavelength=1.54184 Å

Cell: a=11.1104(2) Å b=7.3378(2) Å c=30.7872(7) Å
alpha=90° beta=94.451(2)° gamma=90°

Temperature: 100 K

	Calculated	Reported
Volume	2502.38(10)	2502.38(10)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C30 H24 Cl N5 O3	C30 H24 Cl N5 O3
Sum formula	C30 H24 Cl N5 O3	C30 H24 Cl N5 O3
Mr	537.99	537.99
Dx, g cm-3	1.428	1.428
Z	4	4
Mu (mm-1)	1.715	1.715
F000	1120.0	1120.0
F000'	1124.64	
h, k, lmax	13, 8, 36	13, 8, 36
Nref	4412	4394
Tmin, Tmax	0.872, 0.887	0.782, 1.000
Tmin'	0.872	

Correction method= # Reported T Limits: Tmin=0.782 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.996 Theta (max)= 66.600

```
R(reflections)= 0.0411( 3907)      wR2(reflections)=
S = 1.020                        0.1095( 4394)
Npar= 353
```

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

 **Alert level C**

PLAT420_ALERT_2_C D-H Bond Without Acceptor N009 --H009 .	Please Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance	2.359 Check
PLAT911_ALERT_3_C Missing PCF Refl Between Thmin & STh/L= 0.595	19 Report

 **Alert level G**

PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms	2 Report
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels	63 Note
PLAT793_ALERT_4_G Model has Chirality at C00D (Centro SPGR)	R Verify
PLAT793_ALERT_4_G Model has Chirality at C00E (Centro SPGR)	R Verify
PLAT793_ALERT_4_G Model has Chirality at C00H (Centro SPGR)	S Verify
PLAT793_ALERT_4_G Model has Chirality at C00K (Centro SPGR)	R Verify
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still	84% Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File	1 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity	3.6 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.	9 Info

- 0 **ALERT level A** - Most likely a serious problem - resolve or explain
 0 **ALERT level B** - A potentially serious problem, consider carefully
 3 **ALERT level C** - Check. Ensure it is not caused by an omission or oversight
 10 **ALERT level G** = General information/check it is not something unexpected
- 0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 3 ALERT type 2 Indicator that the structure model may be wrong or deficient
 4 ALERT type 3 Indicator that the structure quality may be low
 5 ALERT type 4 Improvement, methodology, query or suggestion
 1 ALERT type 5 Informative message, check
-

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

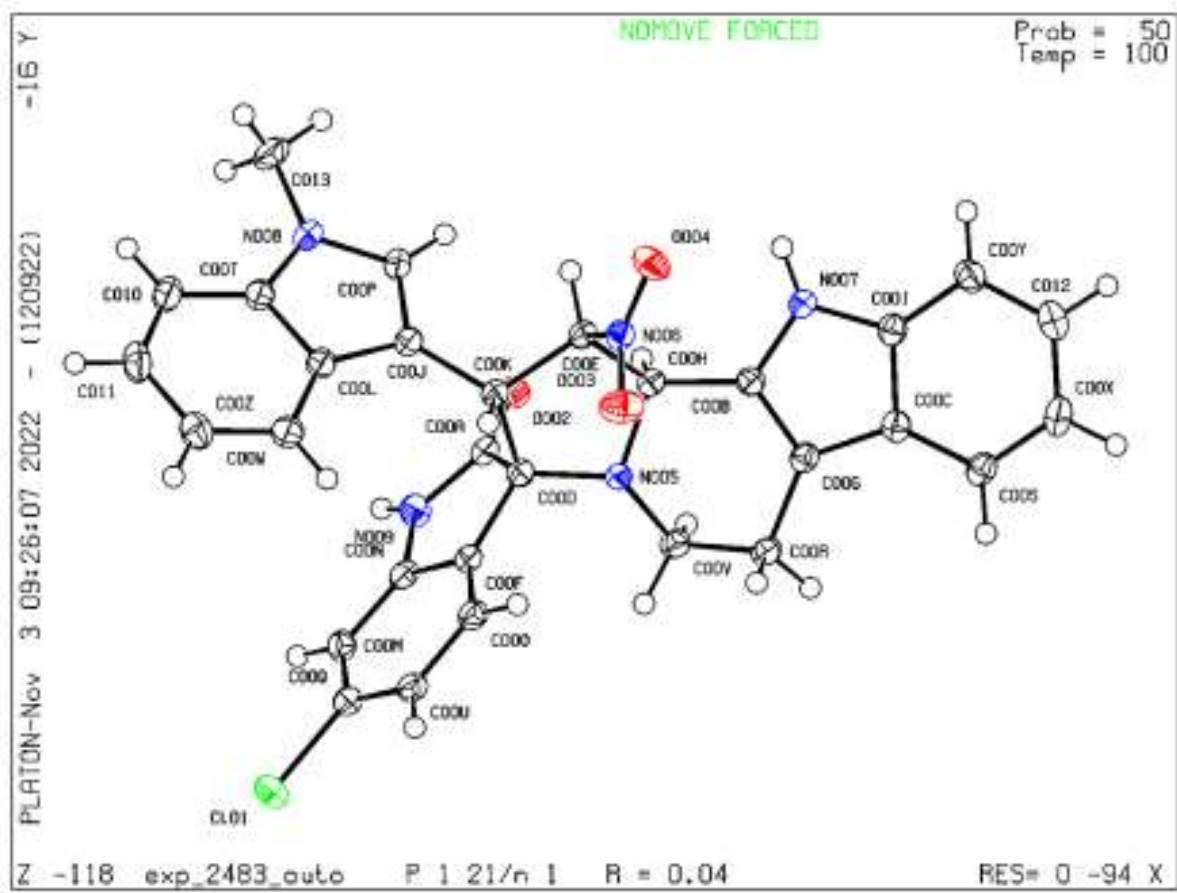
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 12/09/2022; check.def file version of 09/08/2022

Datafile exp_2483_outo - c1qnsd plot



4. Single crystal X-ray diffraction study data of compound 6b

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) exp_2413_auto

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: exp_2413_auto

Bond precision:	C-C = 0.0013 Å	Wavelength=0.71073	
Cell:	a=9.3321(2) alpha=67.455(2)	b=11.1799(2) beta=88.693(2)	c=12.8313(3) gamma=86.534(2)
Temperature:	100 K		
	Calculated	Reported	
Volume	1234.15(5)	1234.15(5)	
Space group	P -1	P -1	
Hall group	-P 1	-P 1	
Moiety formula	C28 H24 N4 O3, C H4 O	C28 H24 N4 O3, C H4 O	
Sum formula	C29 H28 N4 O4	C29 H28 N4 O4	
Mr	496.55	496.55	
Dx, g cm-3	1.336	1.336	
Z	2	2	
Mu (mm-1)	0.091	0.091	
F000	524.0	524.0	
F000'	524.23		
h, k, lmax	15, 18, 21	14, 17, 20	
Nref	11542	10382	
Tmin, Tmax	0.984, 0.991	0.901, 1.000	
Tmin'	0.973		

```
Correction method= # Reported T Limits: Tmin=0.901 Tmax=1.000
AbsCorr = MULTI-SCAN
```

Data completeness= 0.899 Theta(max)= 35.812

```
R(reflections)= 0.0440( 8251)      wR2(reflections)=
                                0.1328( 10382)
S = 1.070                        Npar= 337
```

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.



Alert level C

PLAT230_ALERT_2_C Hirshfeld Test Diff for N007 --C00R . 6.2 s.u.



Alert level G

PLAT007_ALERT_5_G	Number of Unrefined Donor-H Atoms	3	Report
PLAT154_ALERT_1_G	The s.u.'s on the Cell Angles are Equal ..(Note)	0.002	Degree
PLAT720_ALERT_4_G	Number of Unusual/Non-Standard Labels	65	Note
PLAT793_ALERT_4_G	Model has Chirality at C00G (Centro SPGR)	R	Verify
PLAT793_ALERT_4_G	Model has Chirality at C00K (Centro SPGR)	R	Verify
PLAT793_ALERT_4_G	Model has Chirality at C00P (Centro SPGR)	R	Verify
PLAT793_ALERT_4_G	Model has Chirality at C00R (Centro SPGR)	R	Verify
PLAT910_ALERT_3_G	Missing # of FCF Reflection(s) Below Theta(Min).	3	Note
PLAT912_ALERT_4_G	Missing # of FCF Reflections Above STh/L= 0.600	1052	Note
PLAT933_ALERT_2_G	Number of HKL-OMIT Records in Embedded .res File	2	Note
PLAT941_ALERT_3_G	Average HKL Measurement Multiplicity	2.9	Low
PLAT978_ALERT_2_G	Number C-C Bonds with Positive Residual Density.	24	Info

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 2 ALERT type 3 Indicator that the structure quality may be low
 6 ALERT type 4 Improvement, methodology, query or suggestion
 1 ALERT type 5 Informative message, check
-

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Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 12/09/2022; check.def file version of 09/08/2022

Datablock exp_2413_auto : cliporid plot

