

Supplementary Information

Diastereoselective Synthesis of Dispiro[Imidazothiazolotriazine-Pyrrolidin- Oxindoles] and Their Isomerization Pathways in Basic Medium

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X-ray crystallographic data and refinement details for **4f**, **5b**, **6a** and **11e**

Single crystals of **4f**, **5b**, **6a** and **7e** were grown from MeCN.

Data collection for samples **4f** and **6a** X-ray were collected at 100K on a four-circle Rigaku Synergy S diffractometer equipped with a HyPix600HE area-detector (kappa geometry, shutterless ω -scan technique), using graphite monochromatized Cu K $_{\alpha}$ -radiation. The intensity data were integrated and corrected for absorption and decay by the CrysAlisPro program.¹ The structure was solved by direct methods using SHELXT² and refined on F^2 using SHELXL-2018³ in the OLEX2 program.⁴ All non-hydrogen atoms were refined with individual anisotropic displacement parameters. Locations of amino hydrogen atoms (9-NH and 1''-NH) were found from the electron density-difference map; these hydrogen atoms were refined with individual isotropic displacement parameters. All other hydrogen atoms were placed in ideal calculated positions and refined as riding atoms with relative isotropic displacement parameters. The SHELXTL program suite⁵ was used for molecular graphics. The crystal of compound **6a** contains disordered solvent molecules in the voids with volume of 182 Å³ per unit cell. Presumably, it includes an acetonitrile molecule with approximately 85% occupancy and probably water molecules with small partial occupancies, located at the same place, which does not allow correct modeling of the lattice solvent molecules in the crystal. A solvent mask was used by OLEX2⁴ to remove solvent molecules (48 electrons were found).

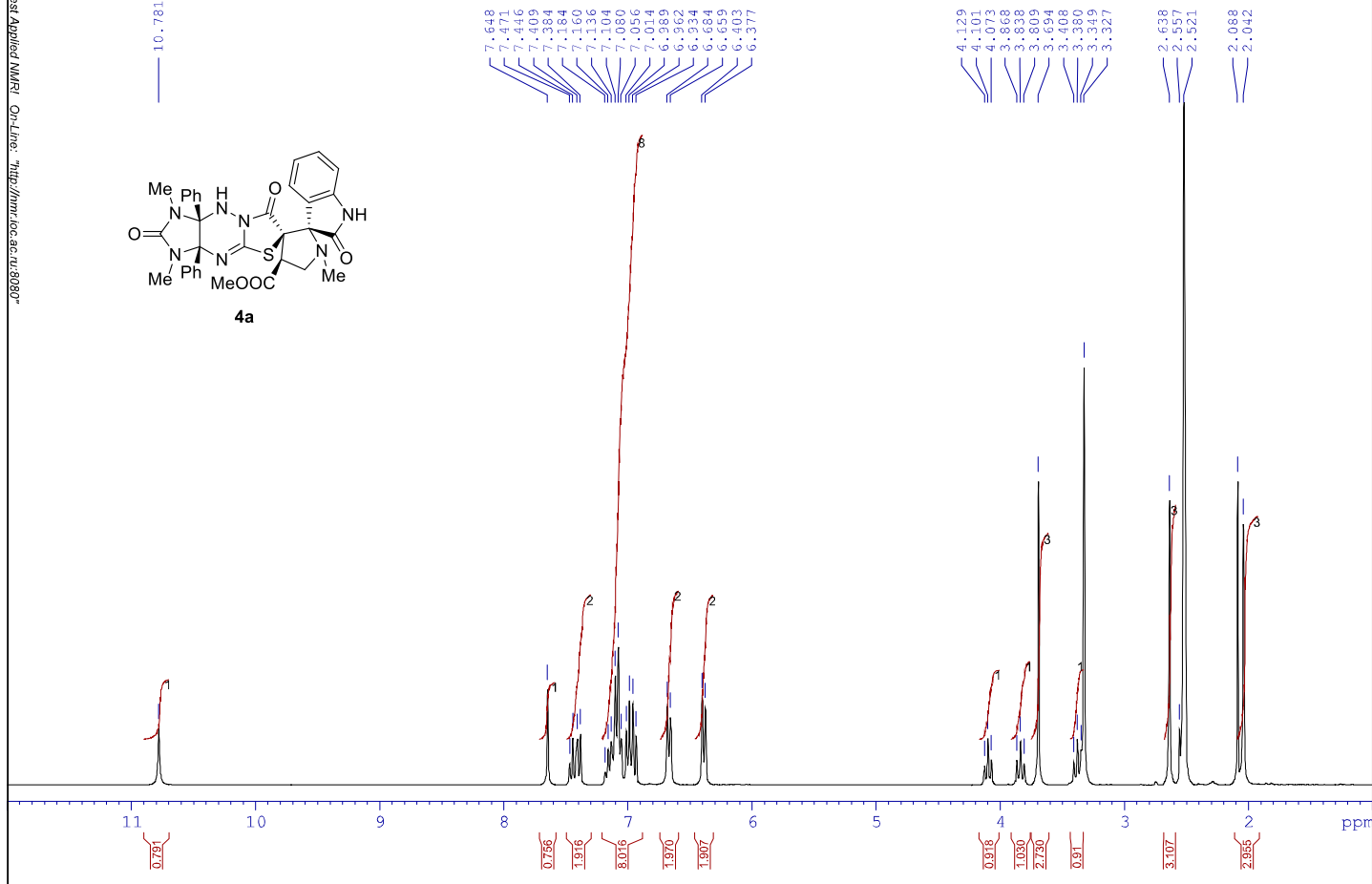
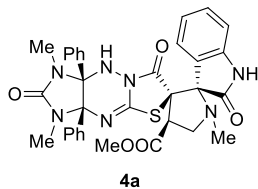
Data collection for samples **5b** and **11e** X-ray were collected at 100K on a Bruker Quest D8 diffractometer equipped with a Photon-III area-detector (graphite monochromator, shutterless ϕ - and ω -scan technique), using Mo K $_{\alpha}$ -radiation. The intensity data were integrated by the SAINT program⁵ and were corrected for absorption and decay using SADABS.⁶ The structure was solved by direct methods using SHELXT² and refined on F^2 using SHELXL-2018.³ All non-hydrogen atoms were refined with individual anisotropic displacement parameters. Locations of amino hydrogen atoms (4-NH, 9-NH and 1''-NH) were found from the electron density-difference map; these hydrogen atoms were refined with individual isotropic displacement parameters. All other hydrogen atoms were placed in ideal calculated positions and refined as riding atoms with relative isotropic displacement parameters. The SHELXTL program suite⁵ was used for molecular graphics.

Deposition Numbers 2297041–2297044 contain the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service www.ccdc.cam.ac.uk/structures.

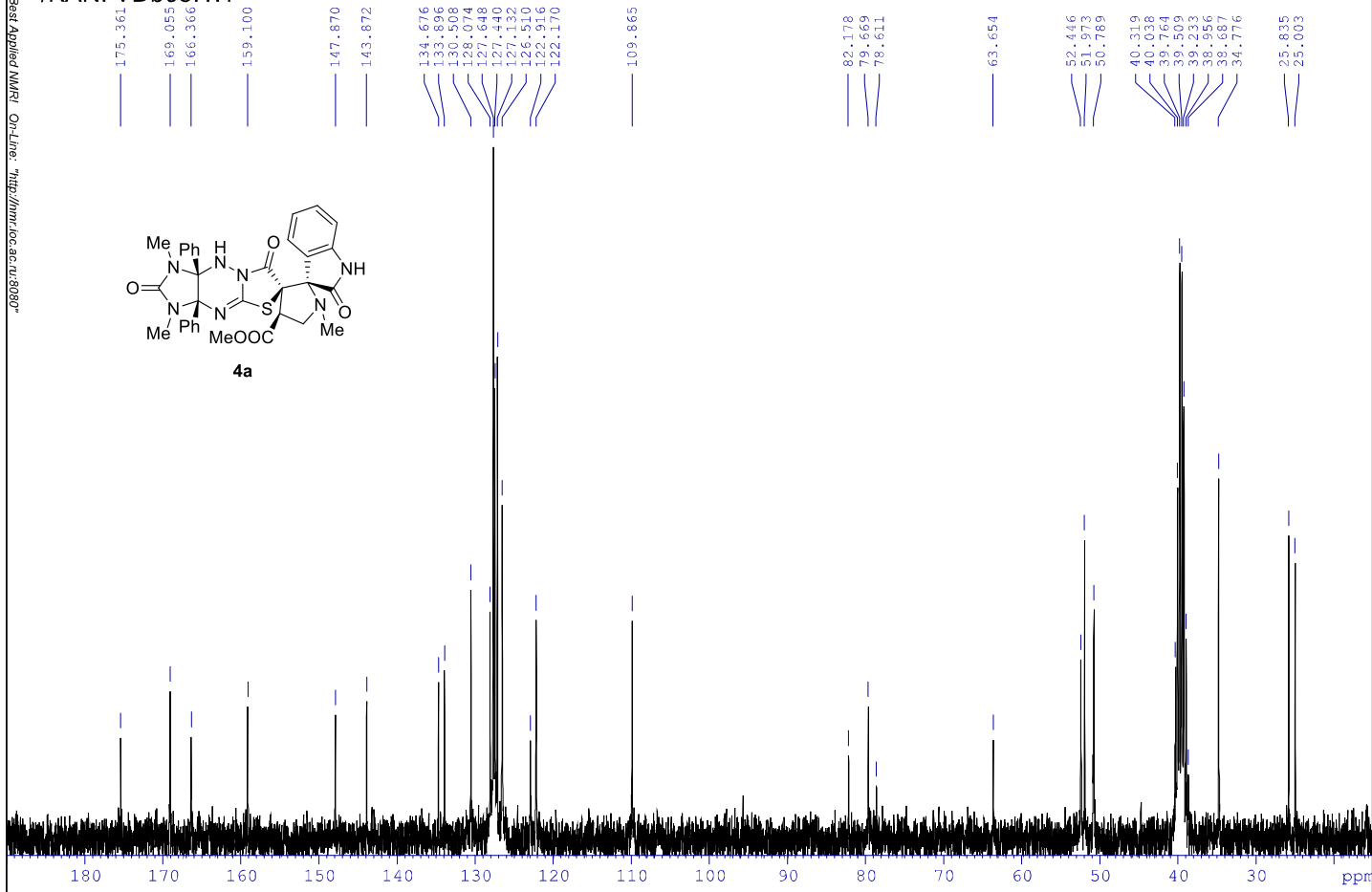
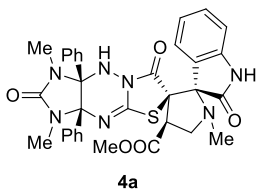
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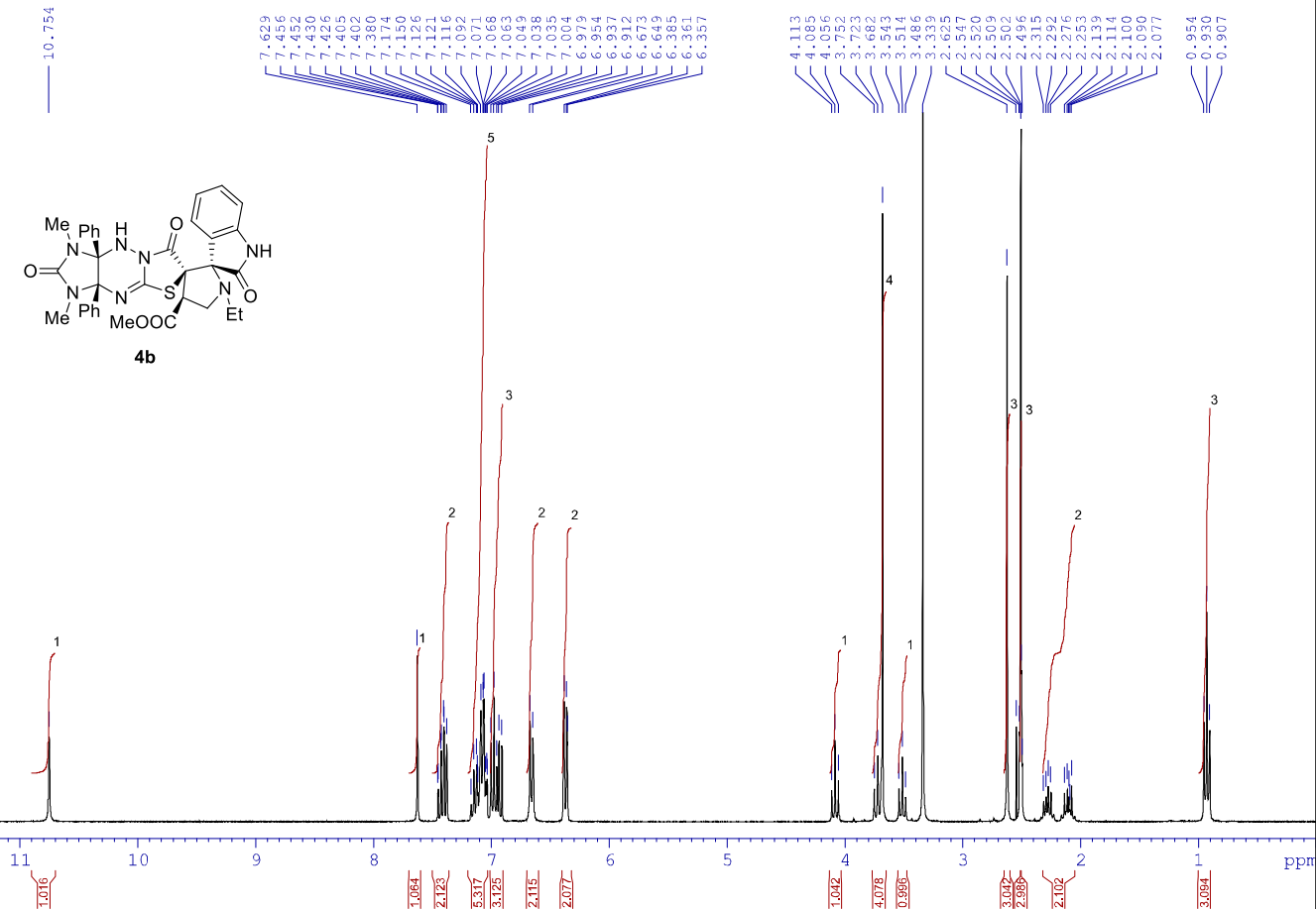
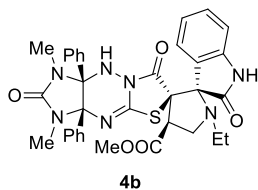
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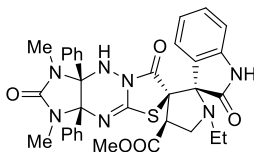
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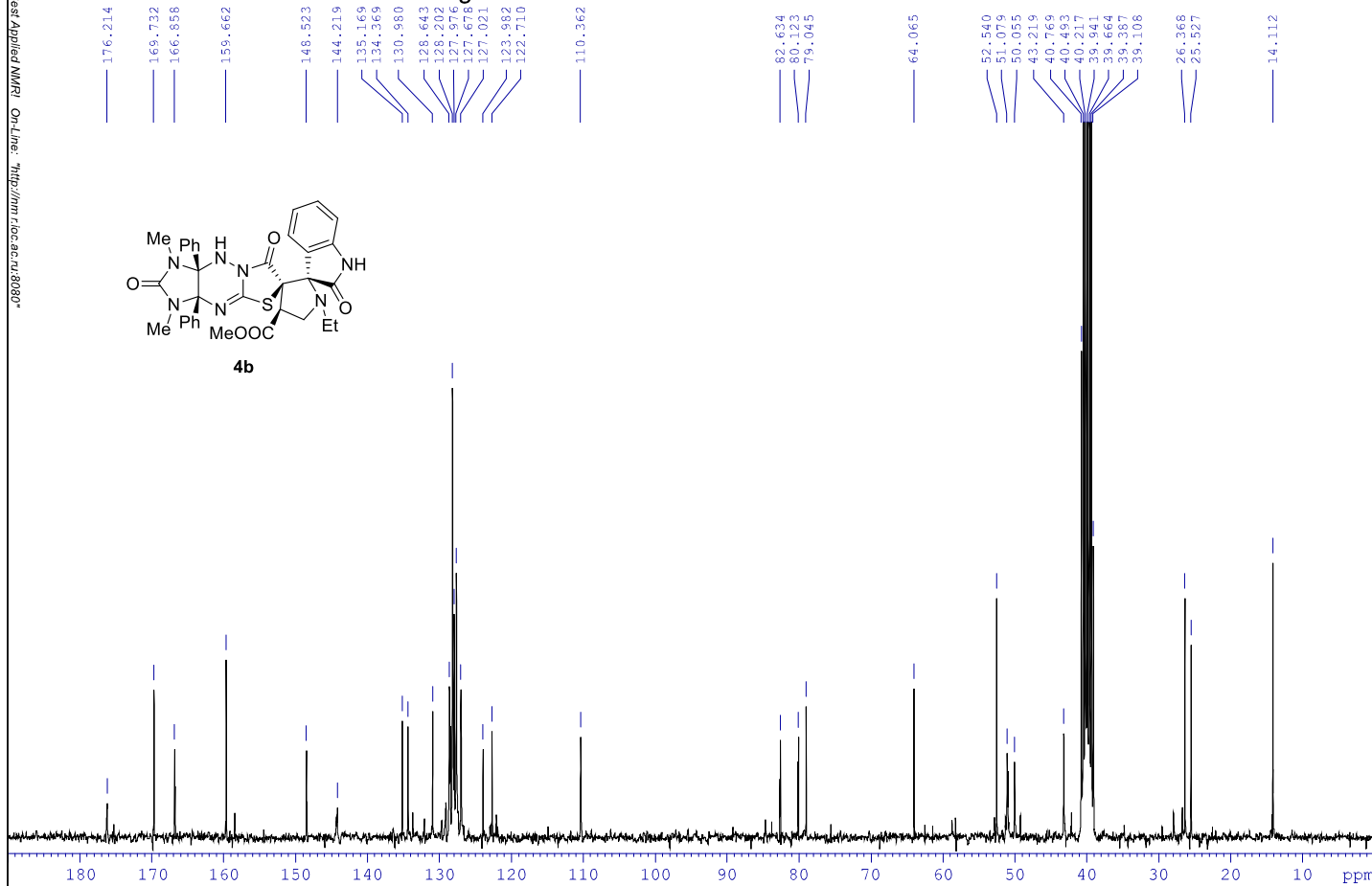
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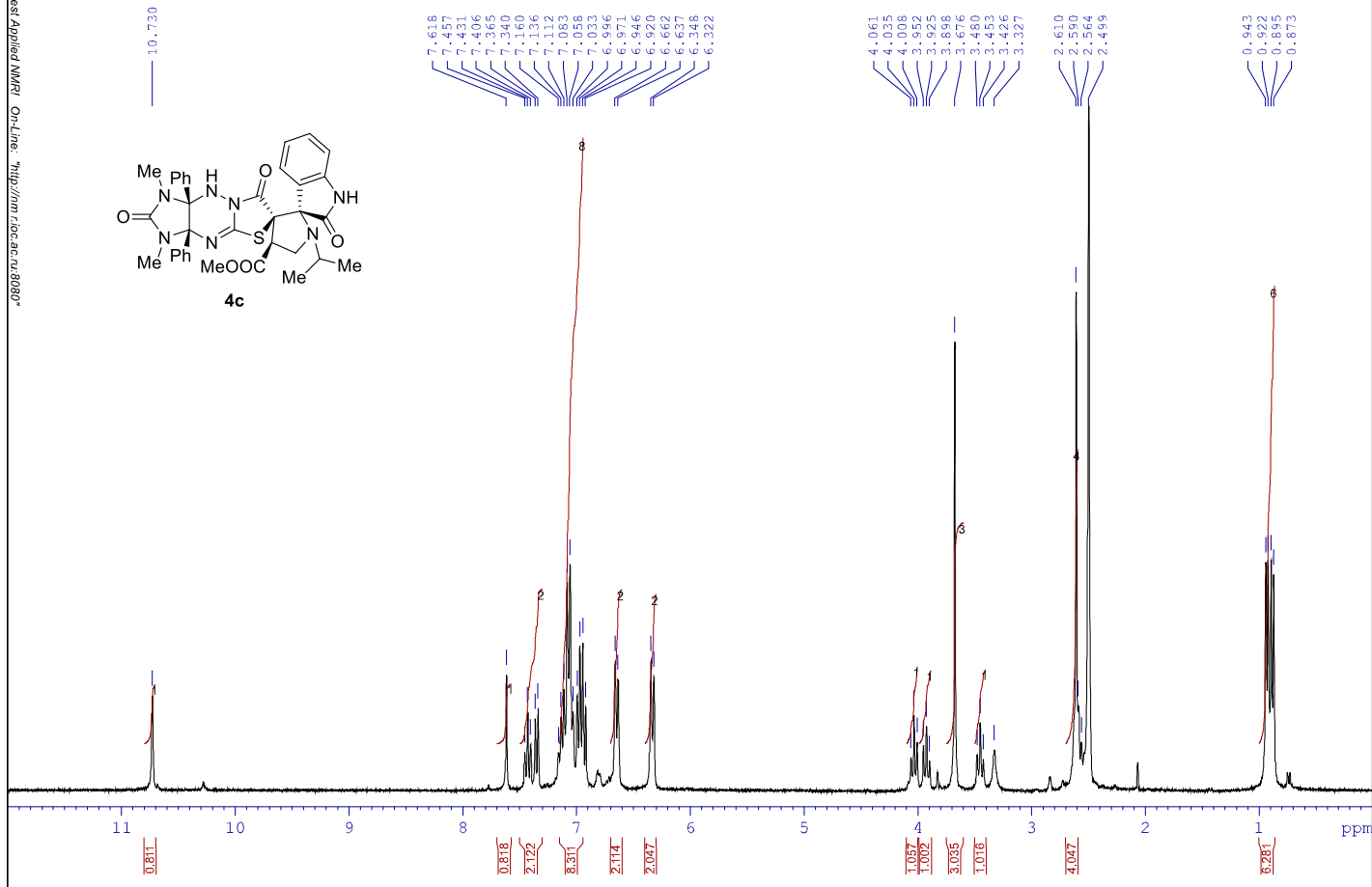
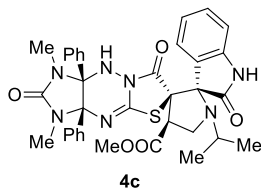
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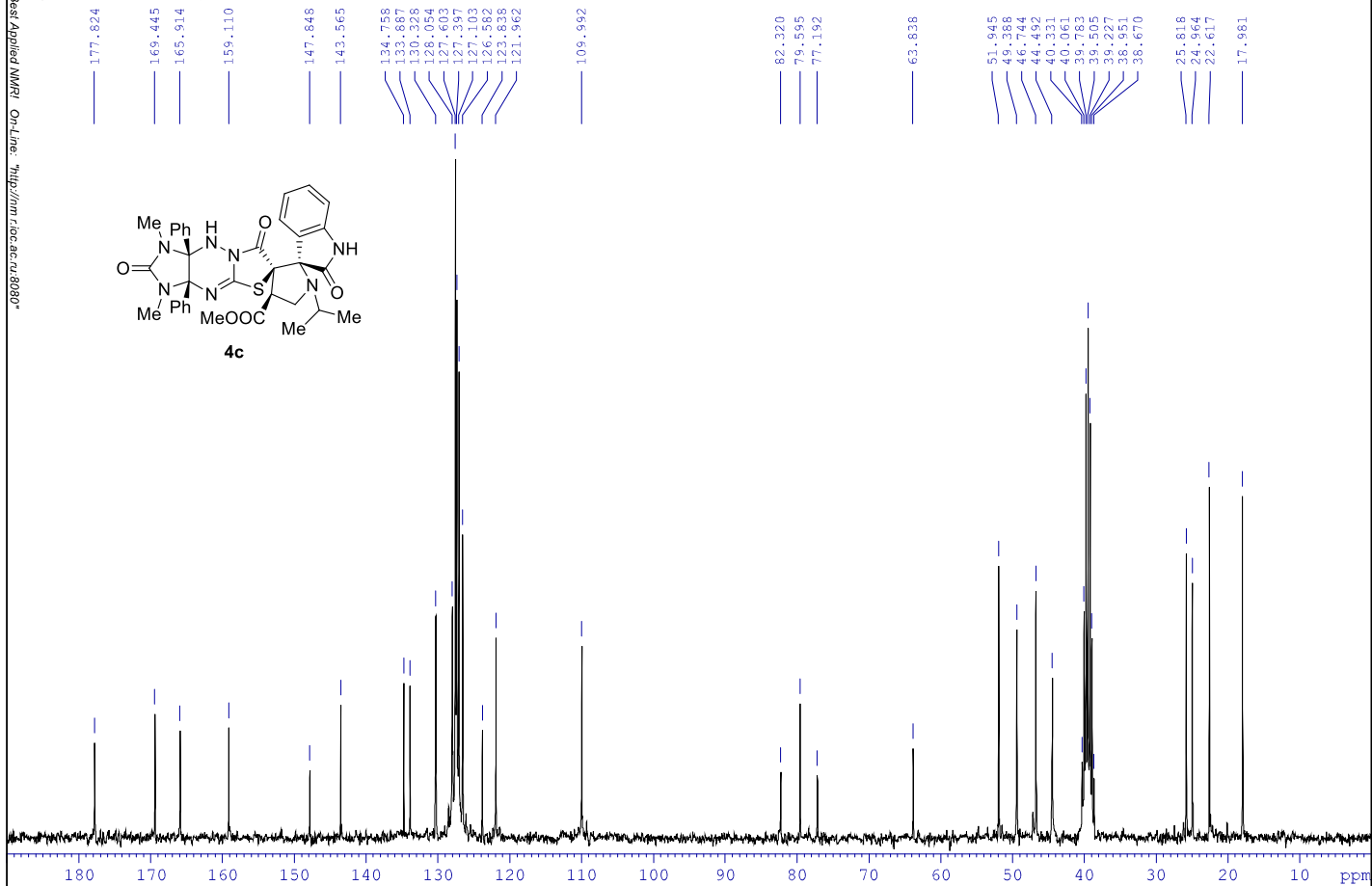
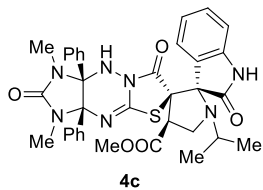
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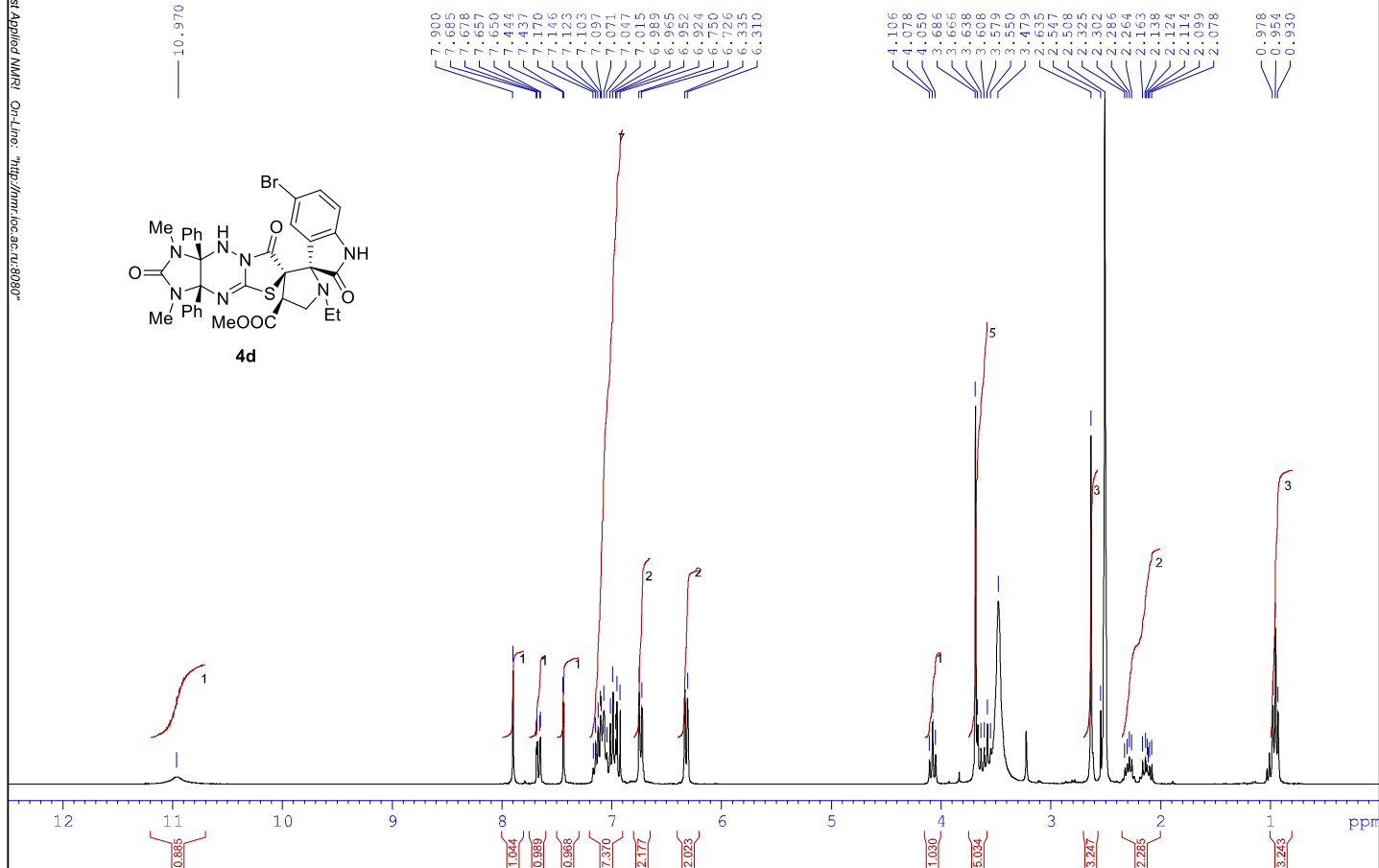
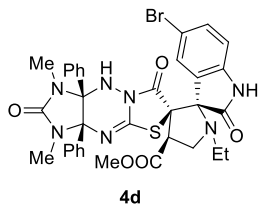
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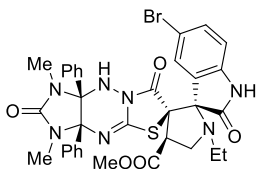
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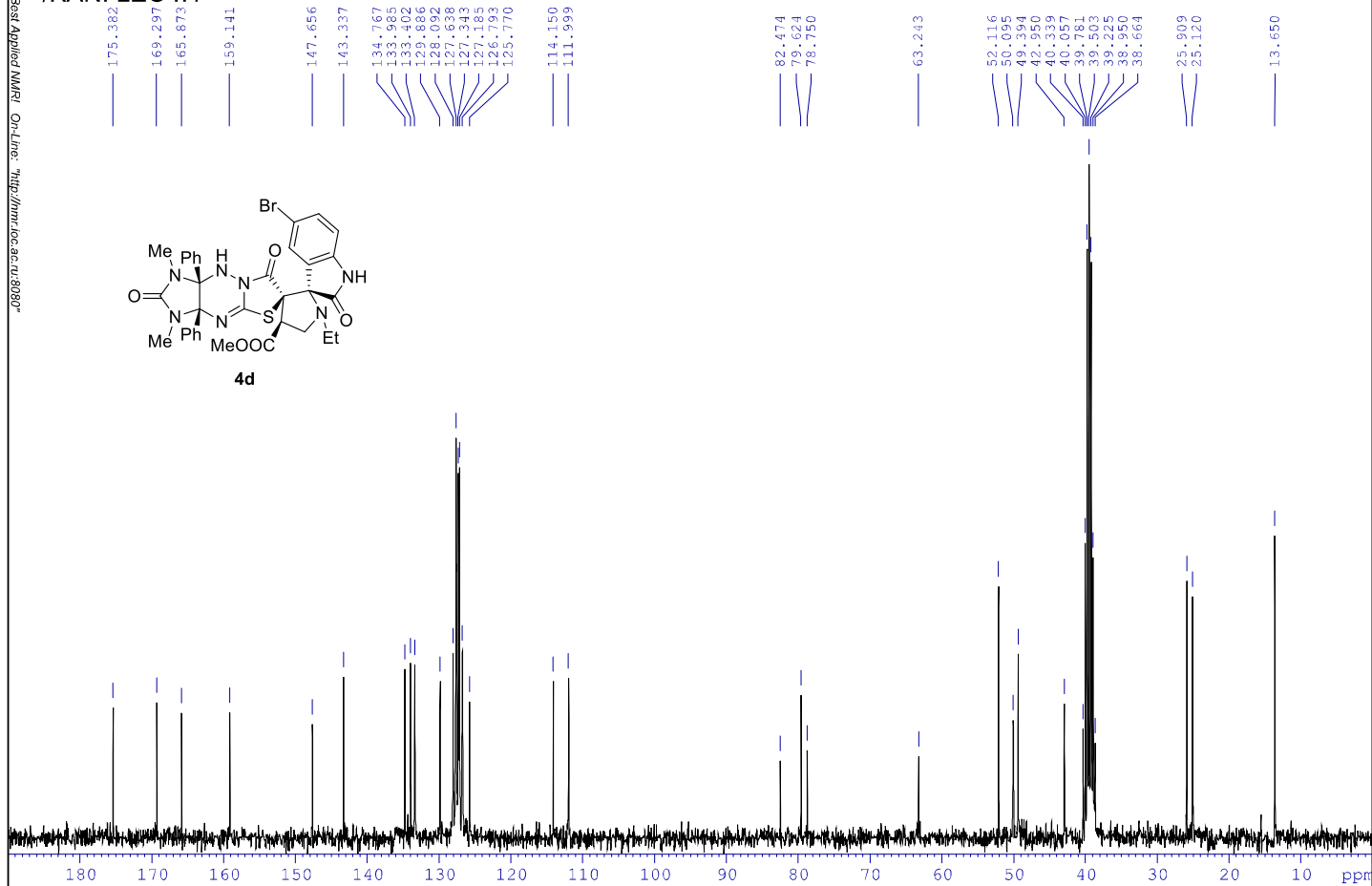
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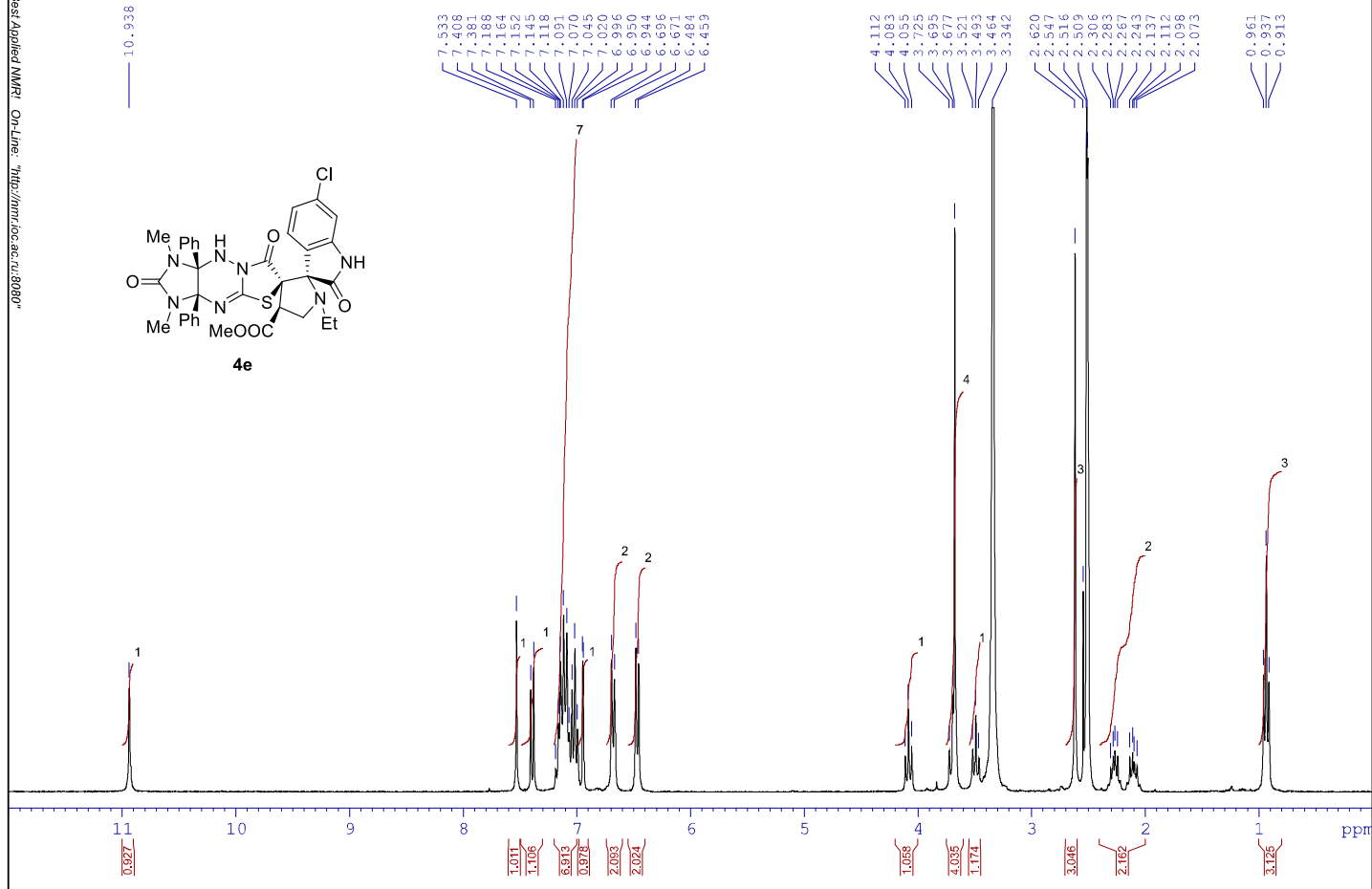
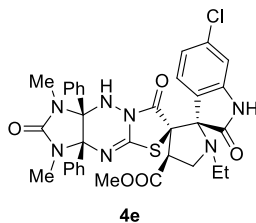
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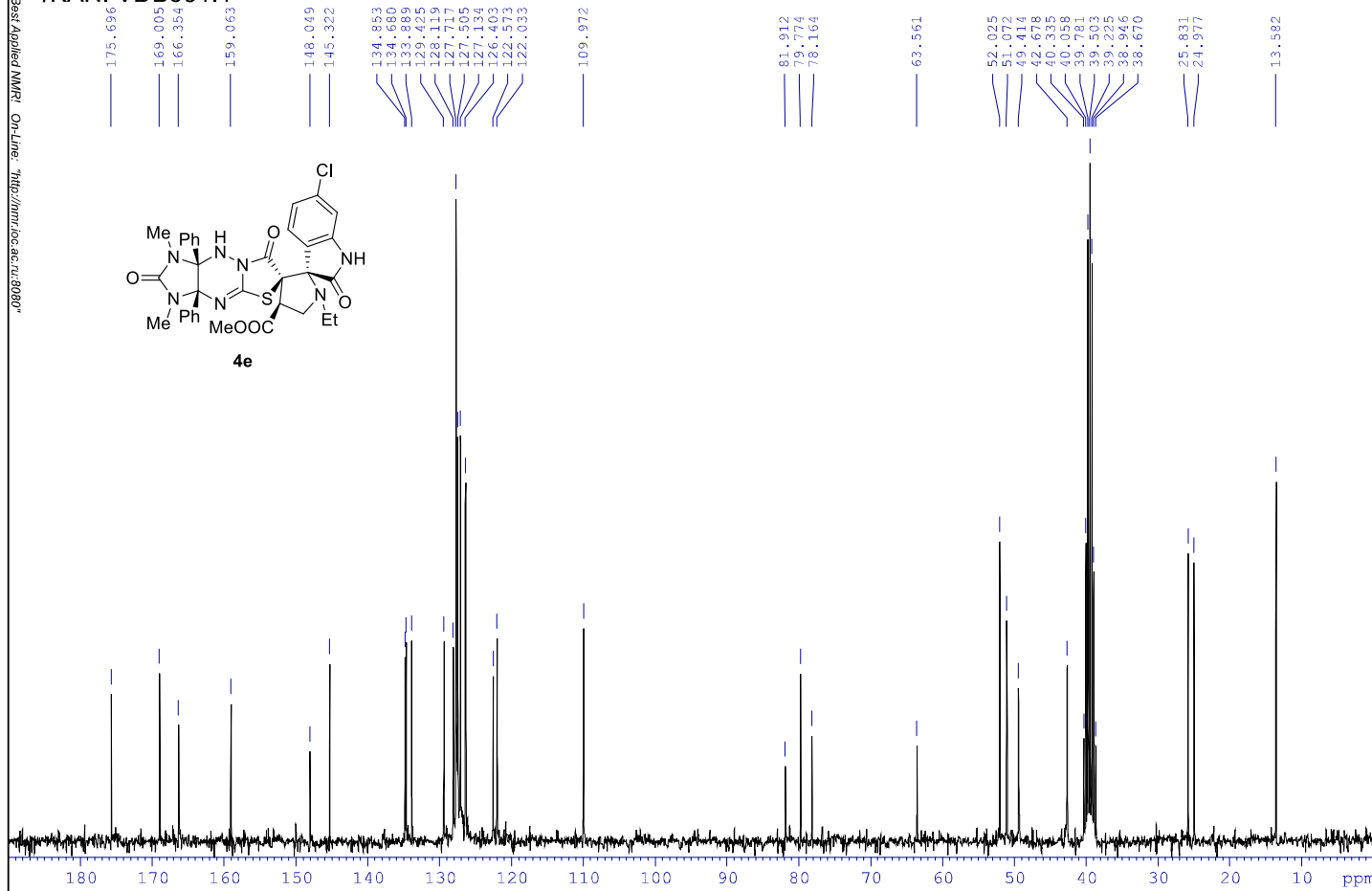
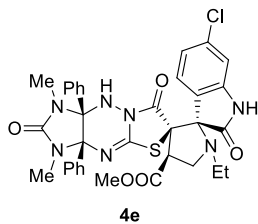
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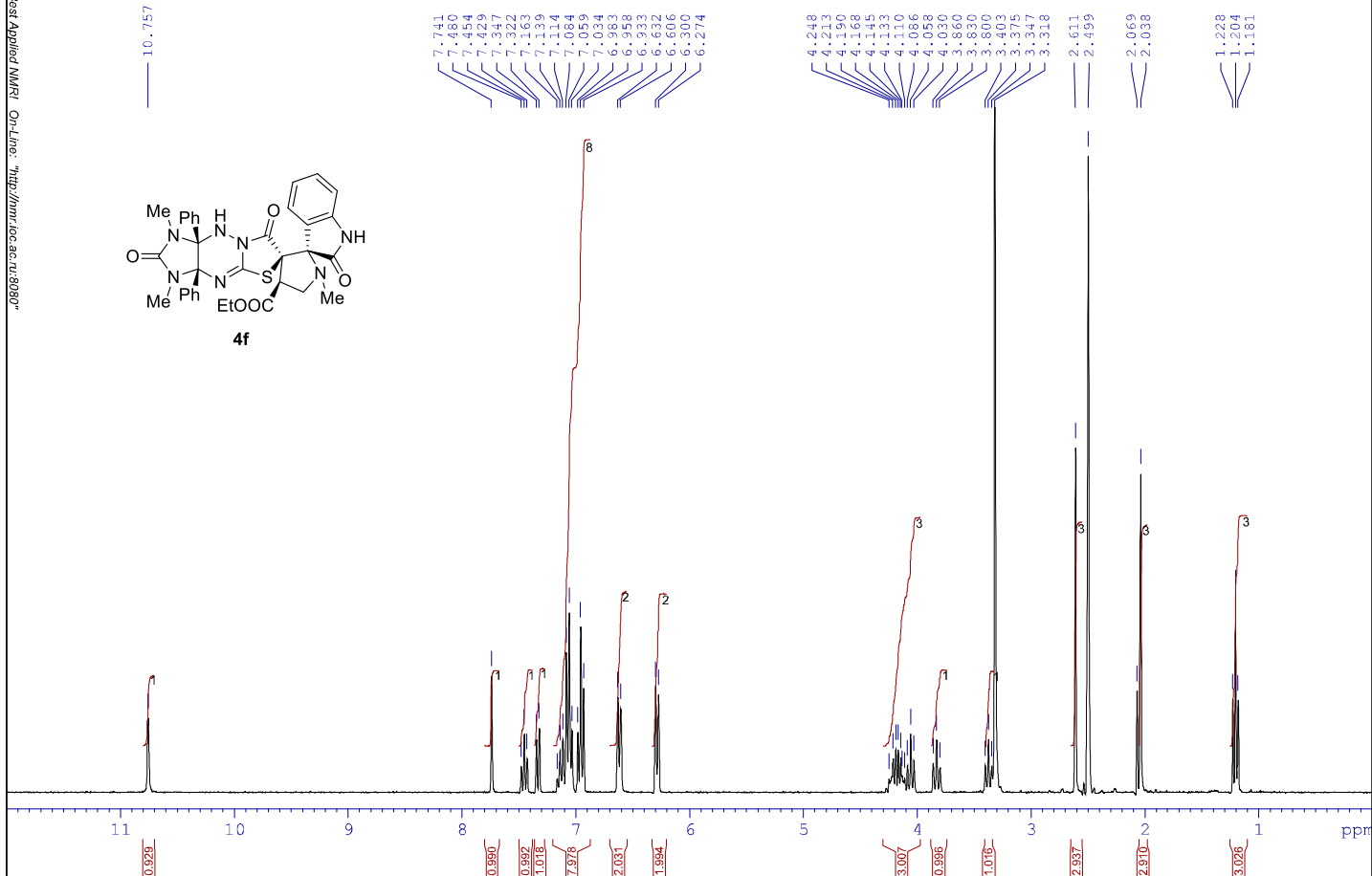
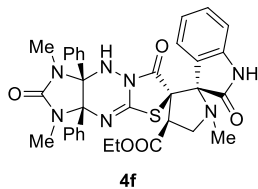
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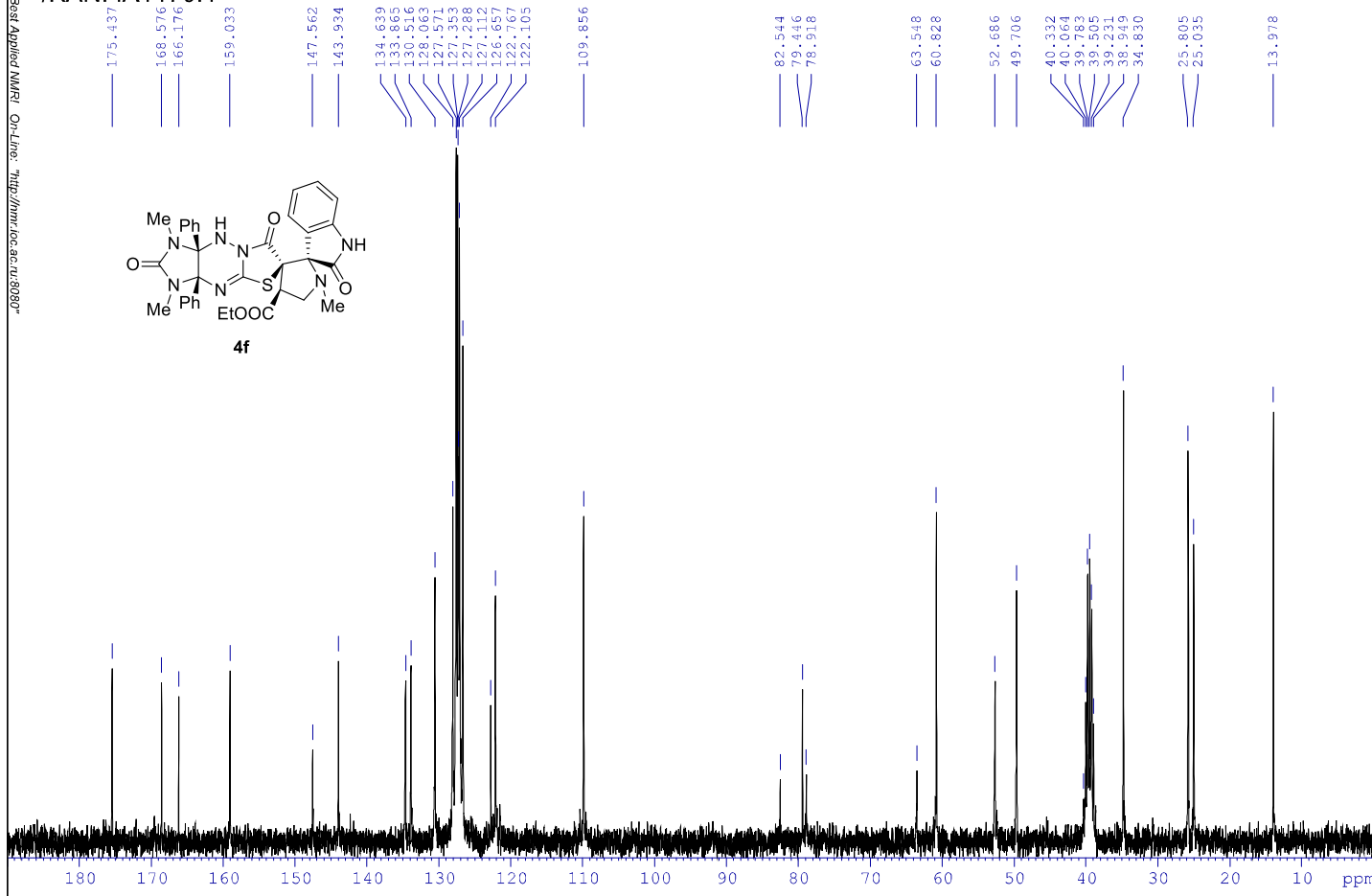
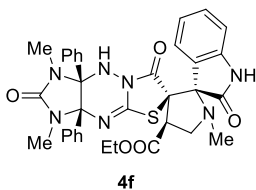
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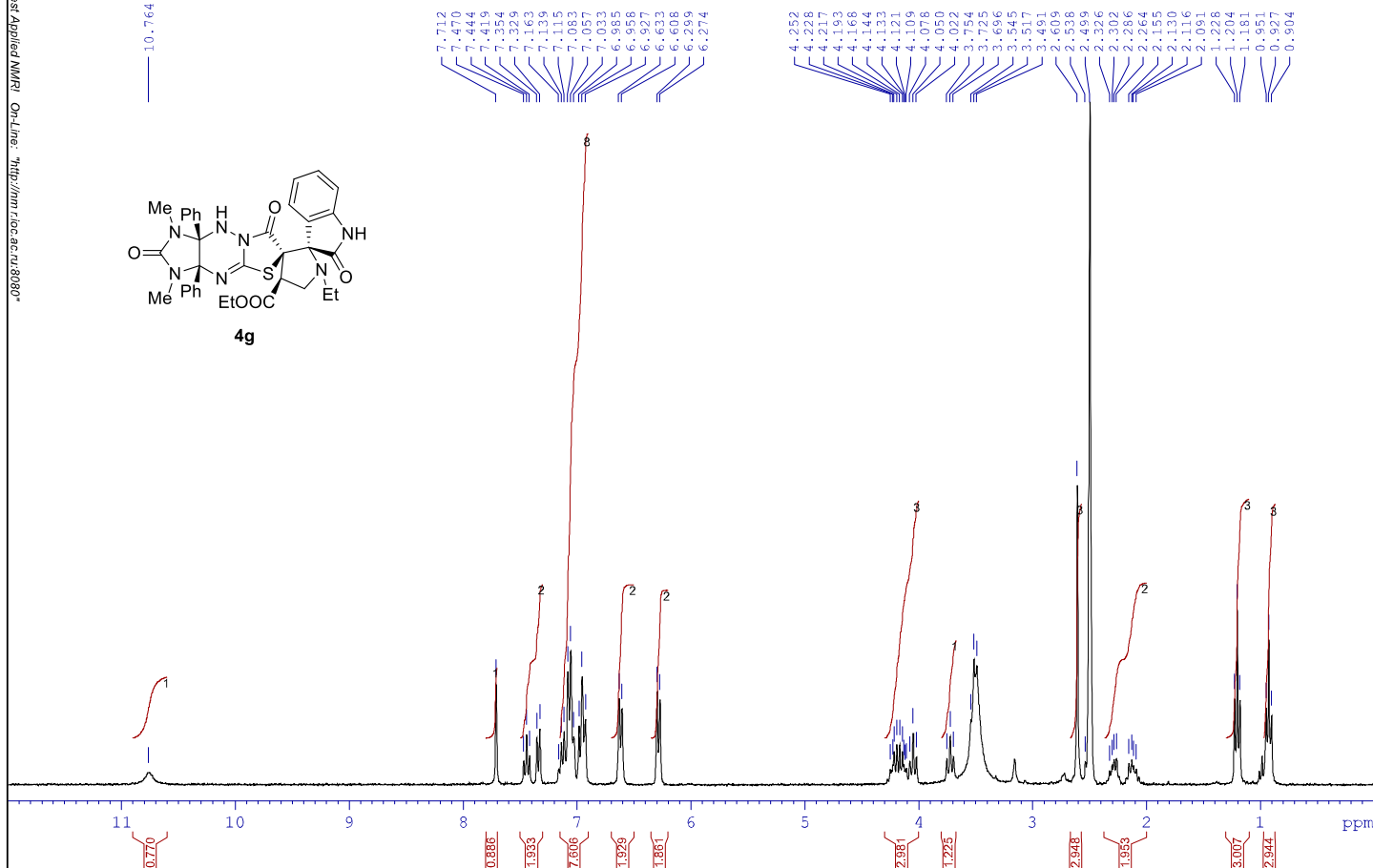
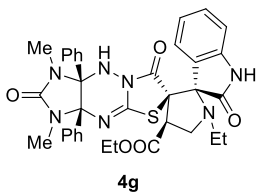
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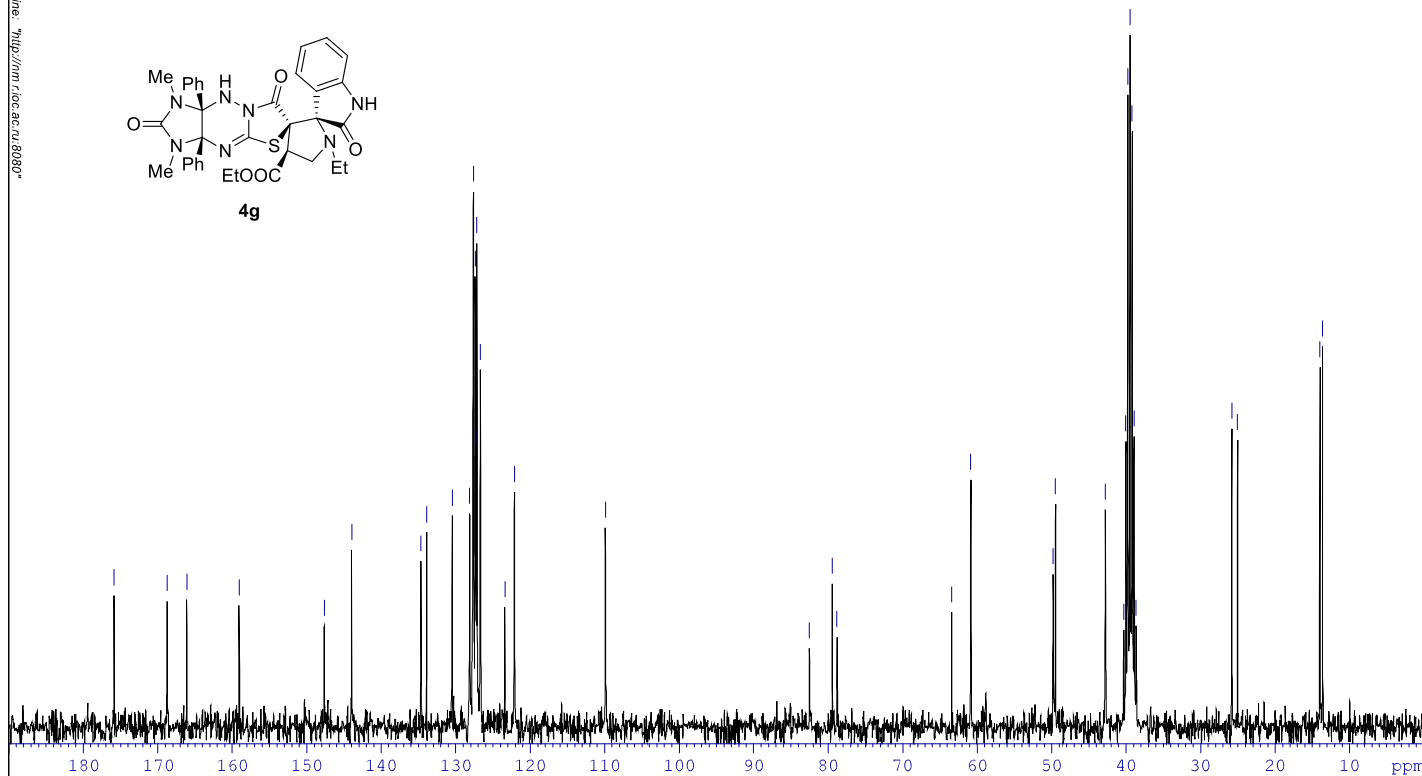
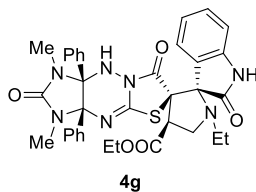
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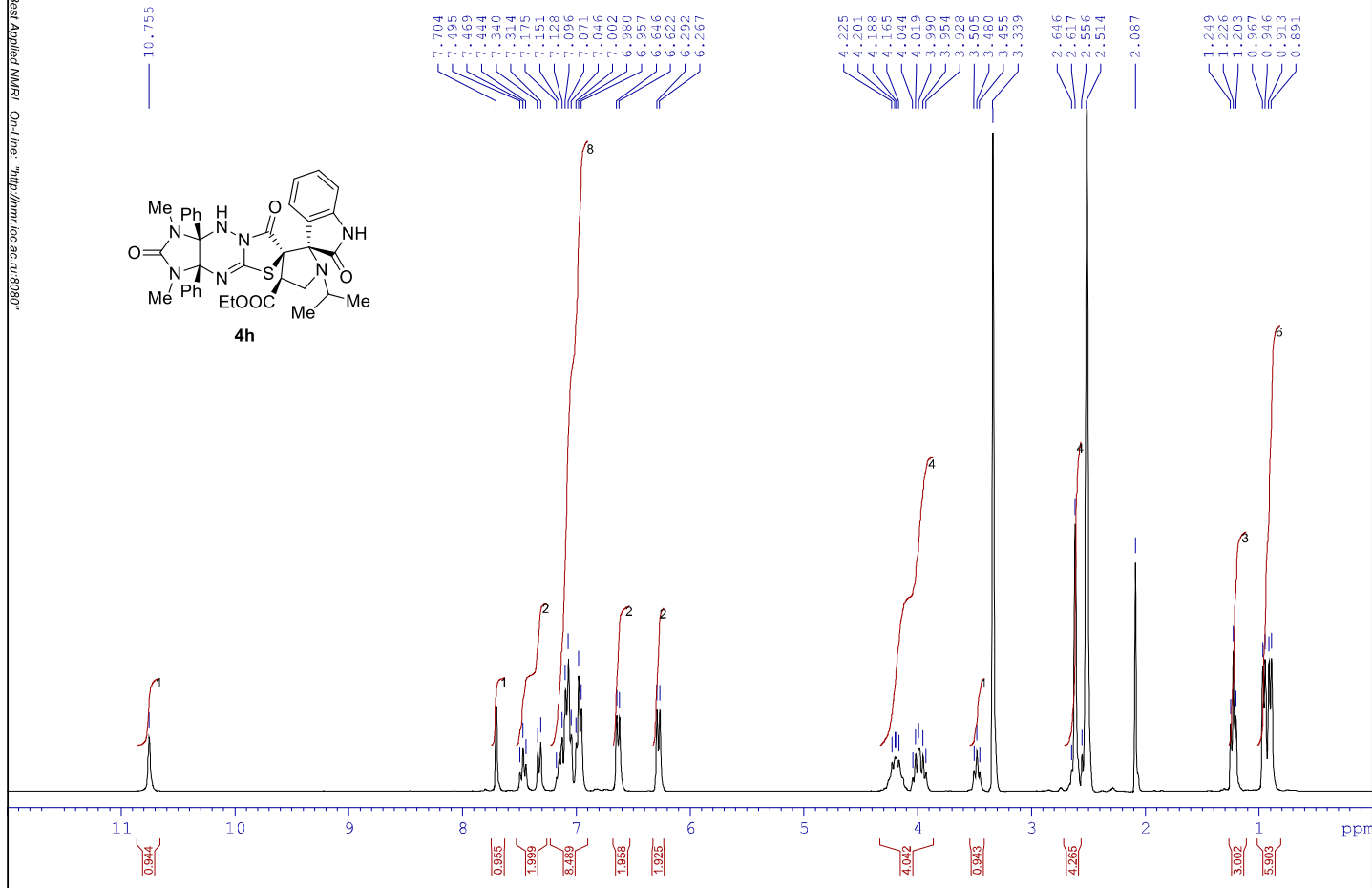
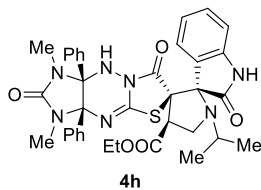
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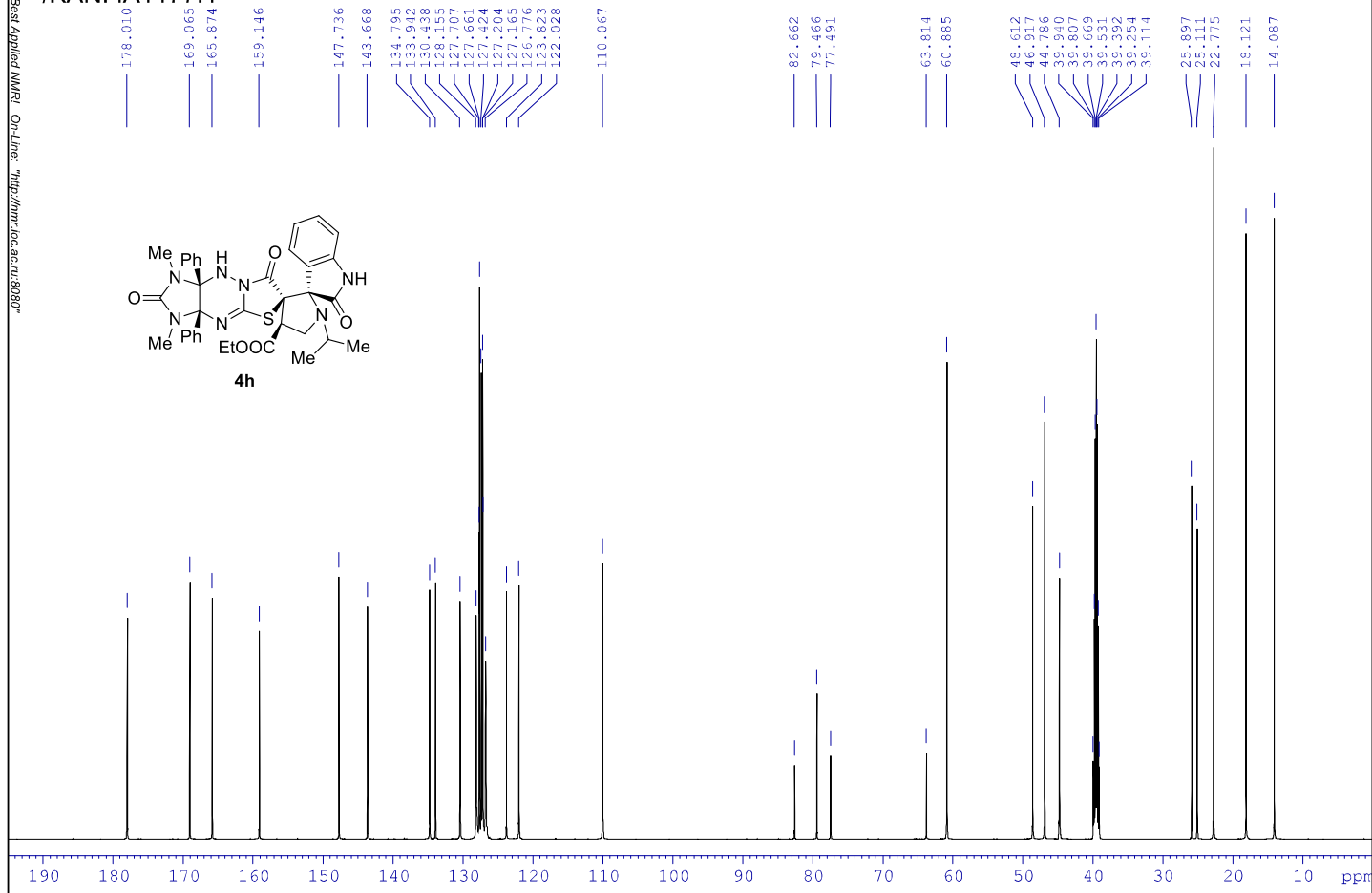
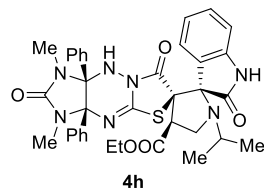
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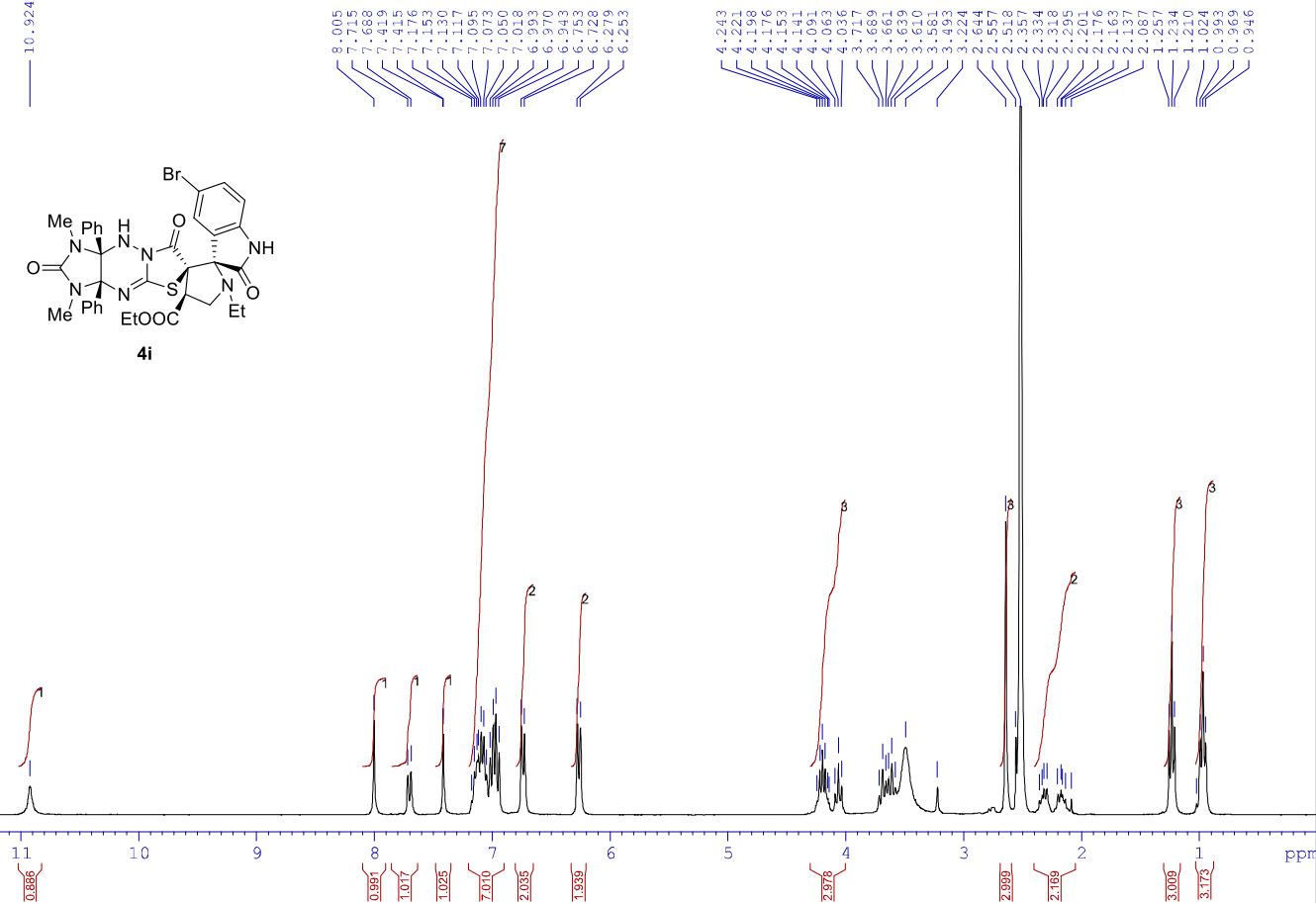
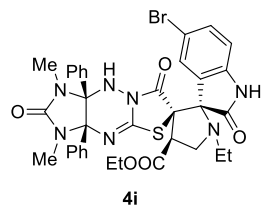
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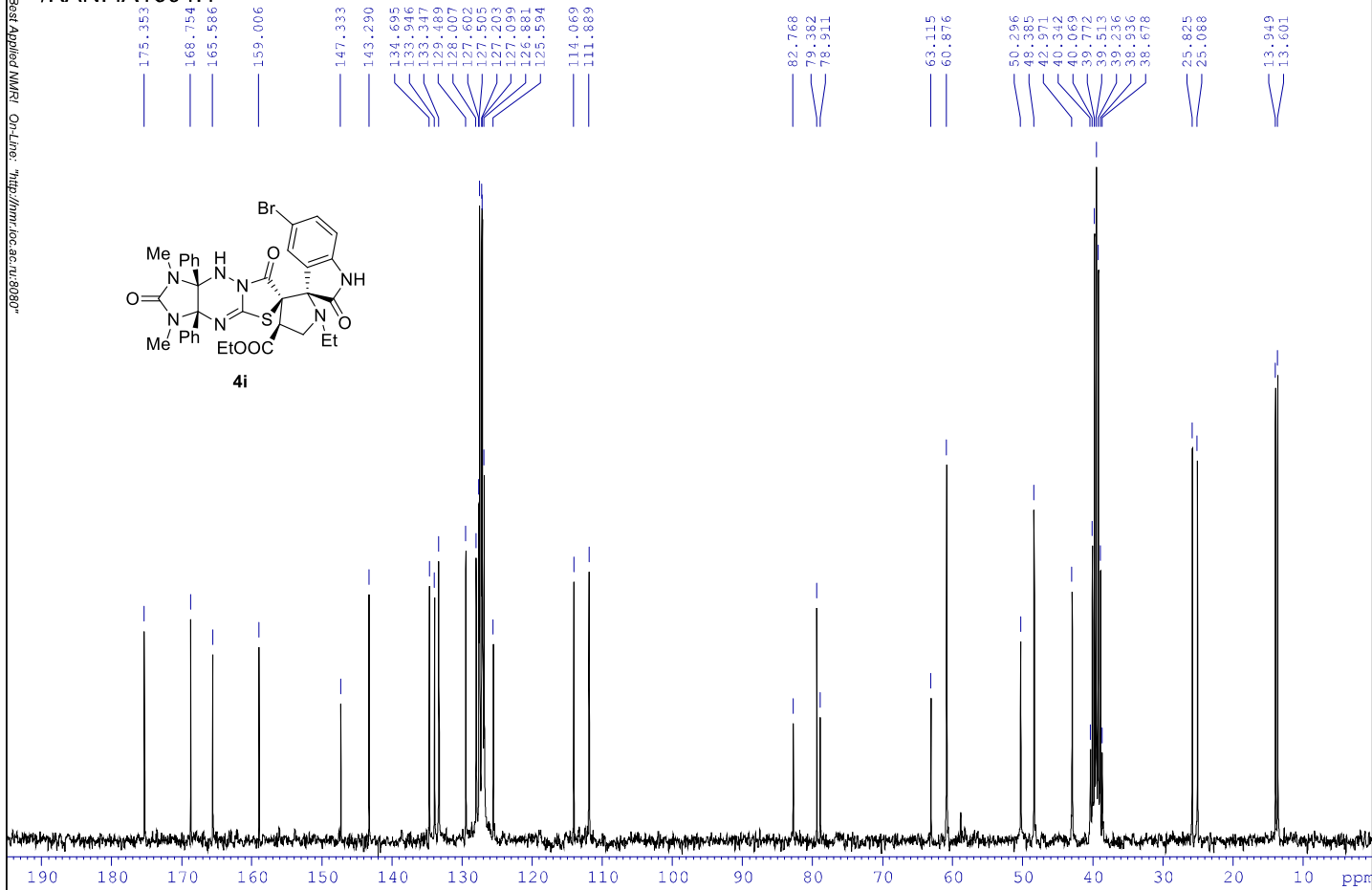
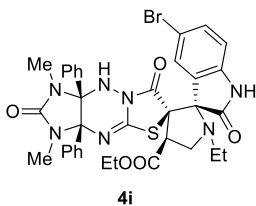
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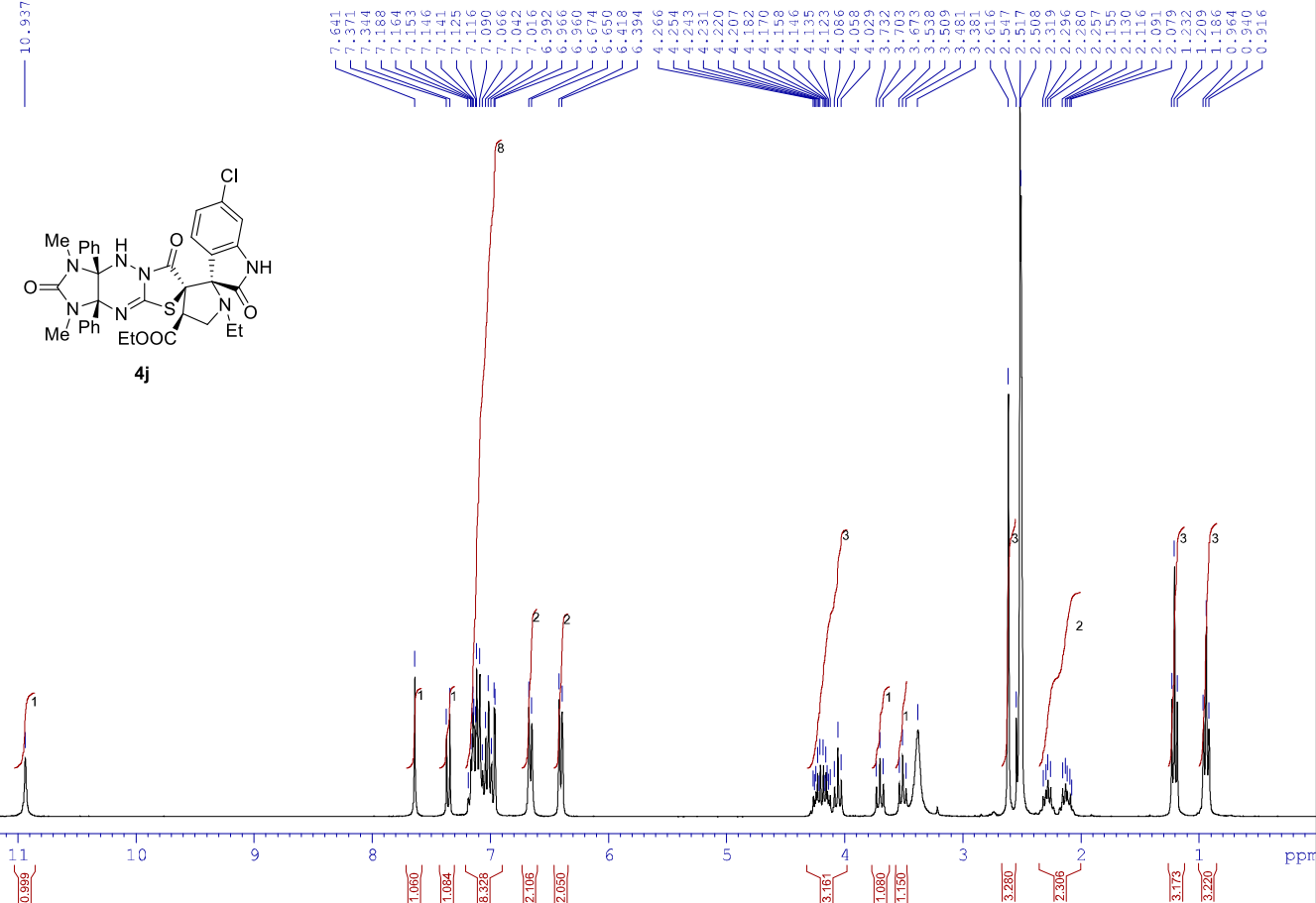
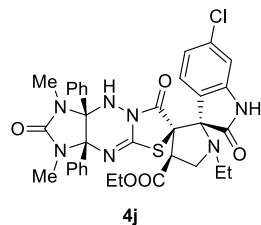


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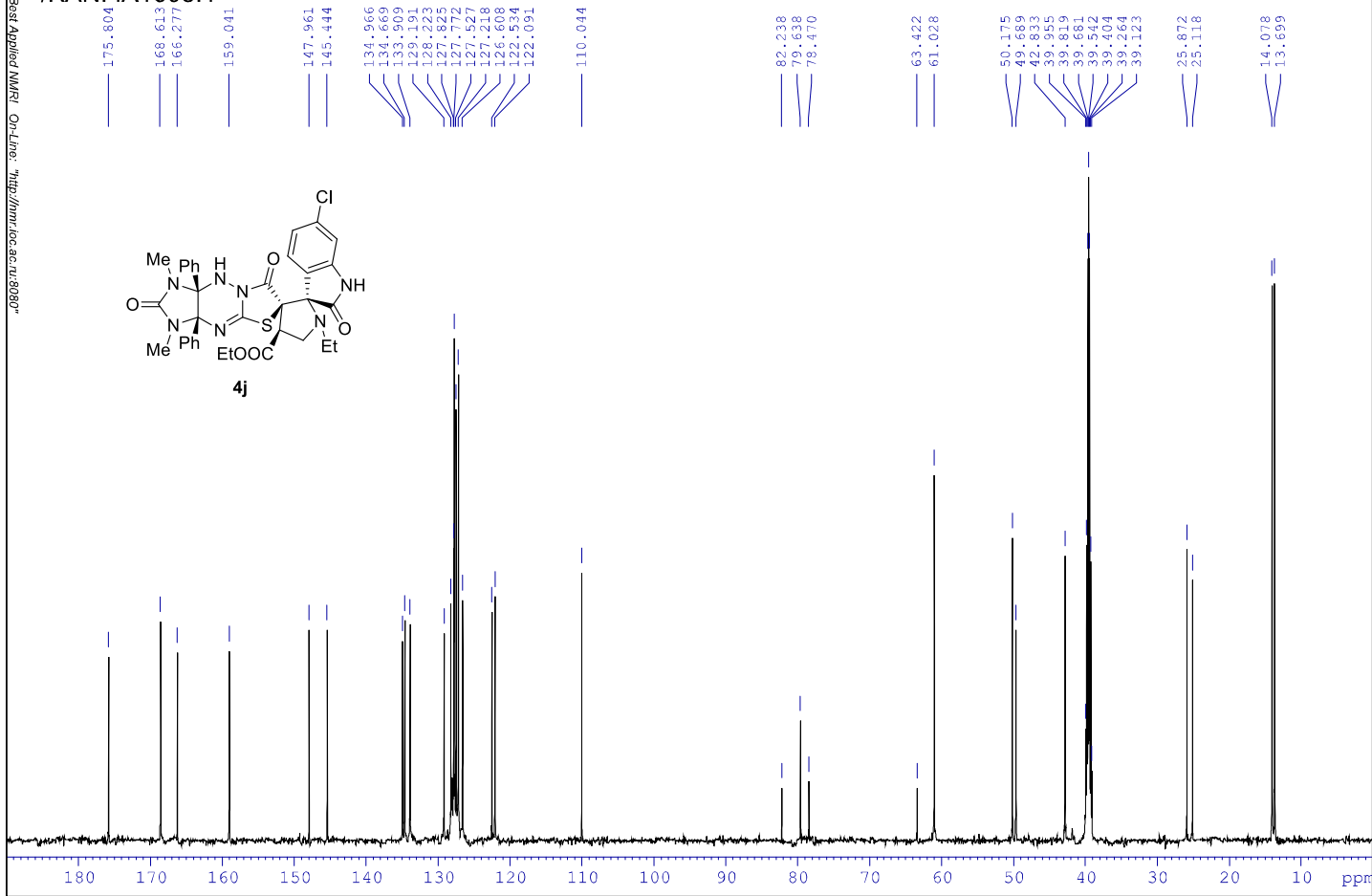
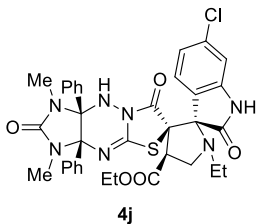


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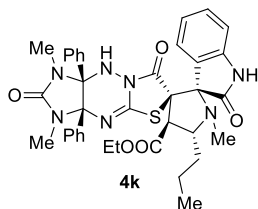


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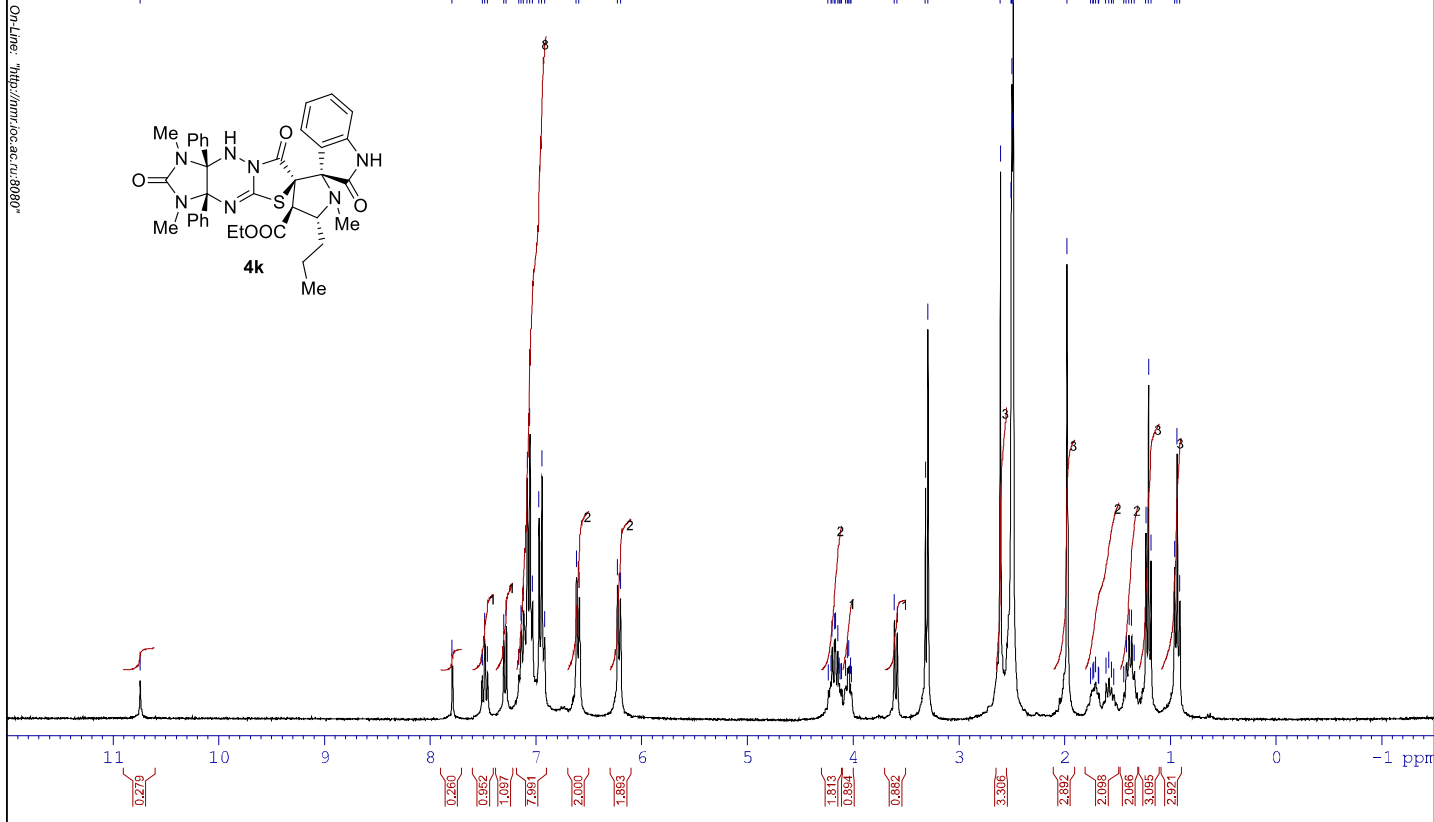


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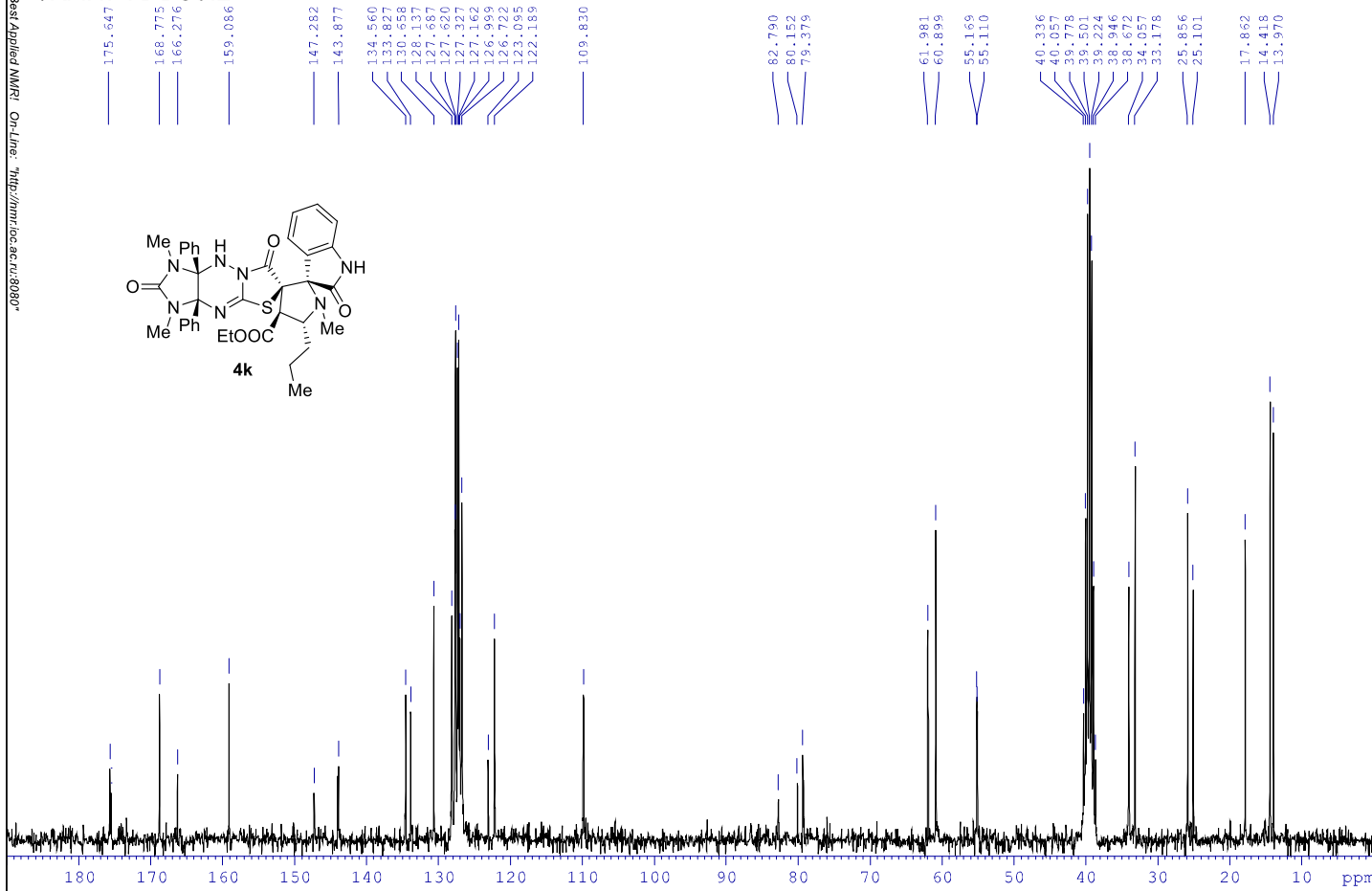
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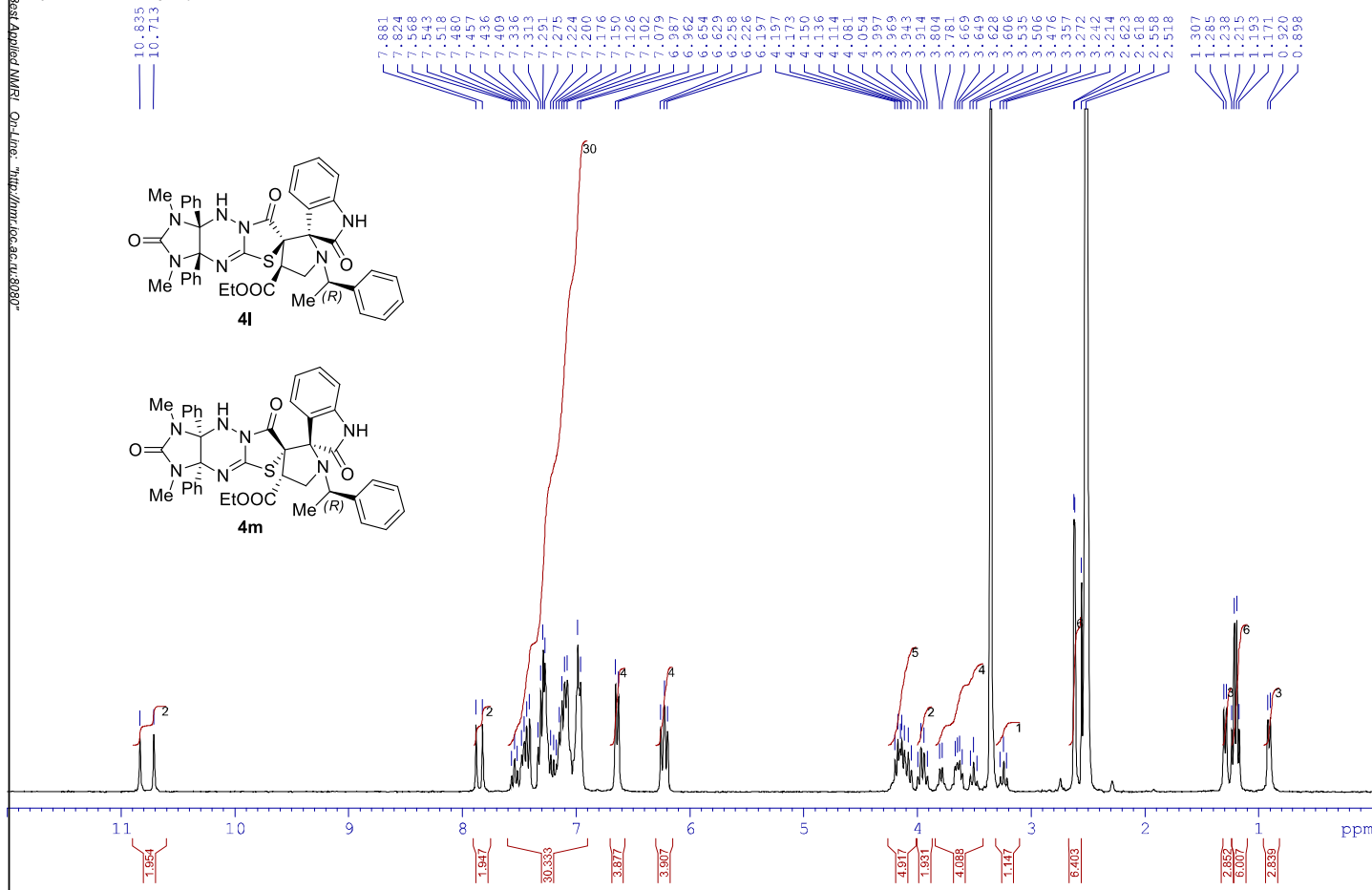
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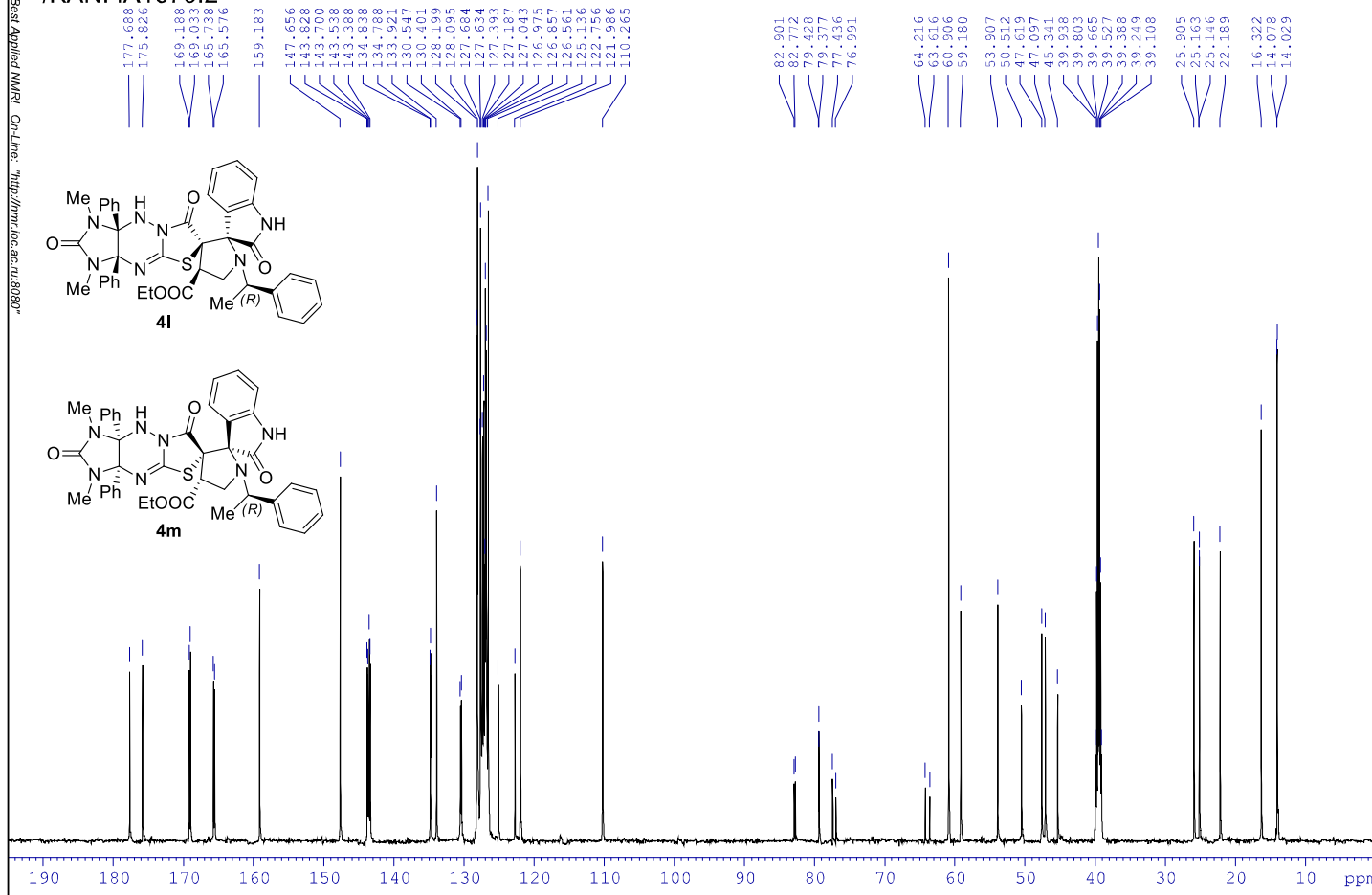
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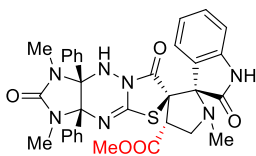
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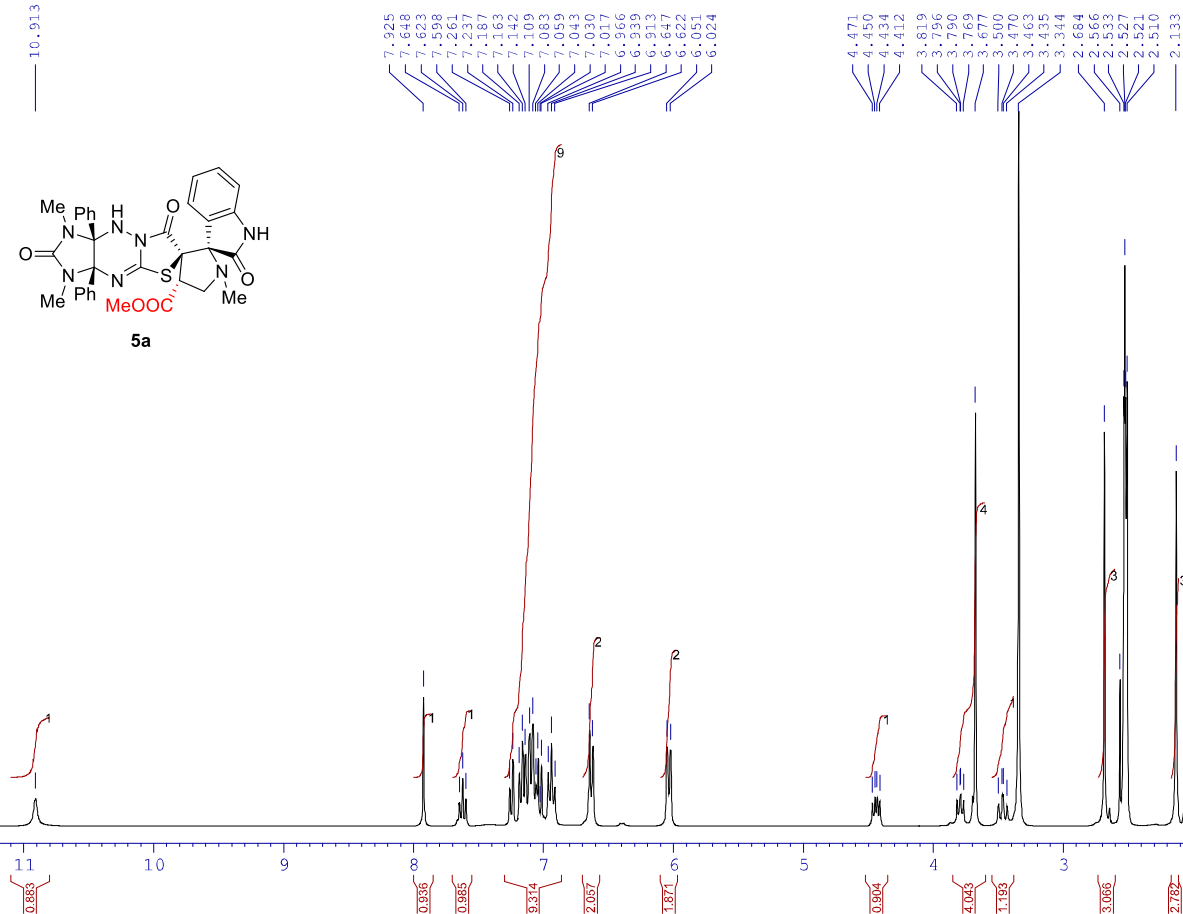
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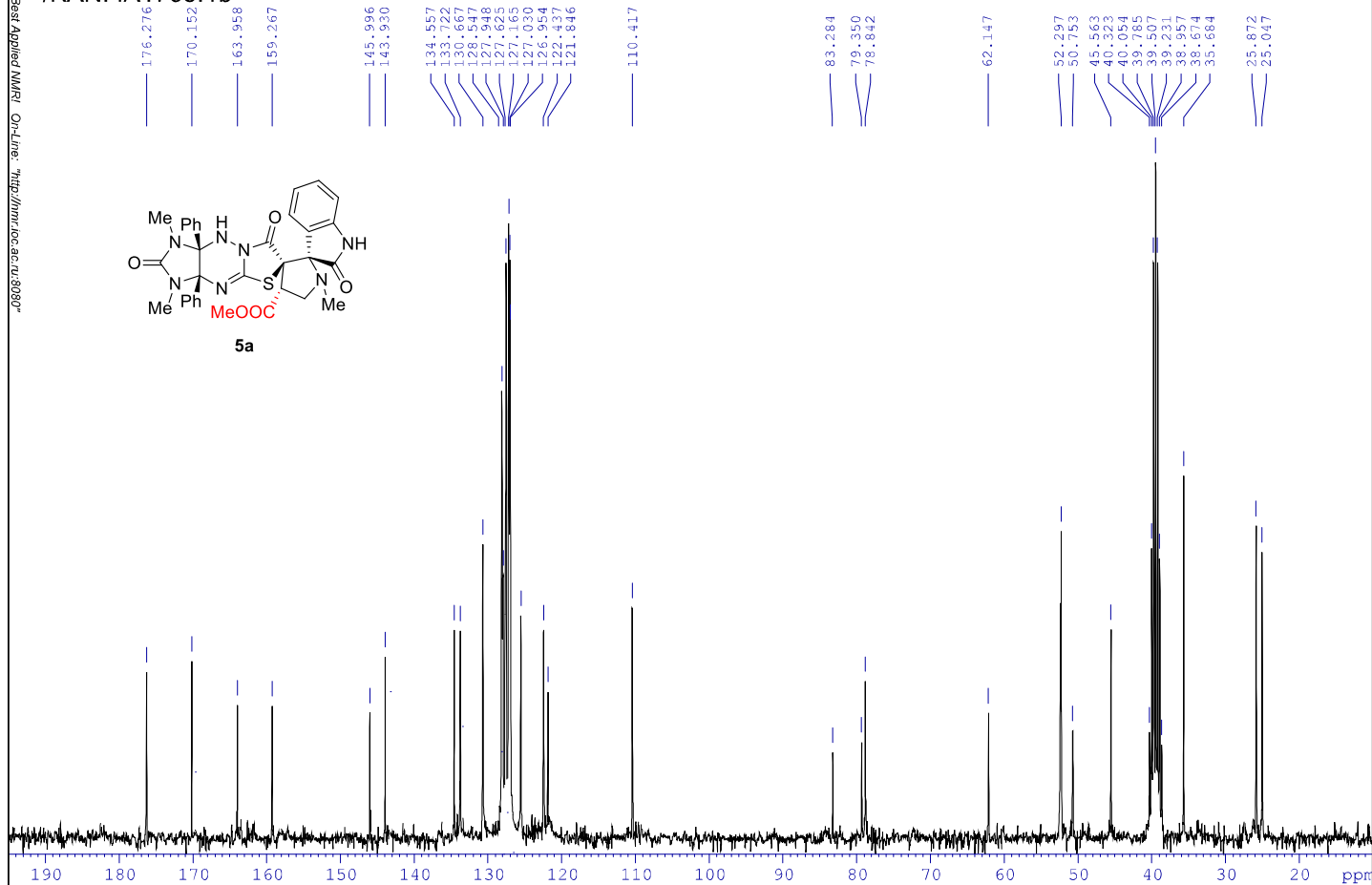
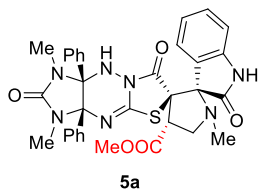
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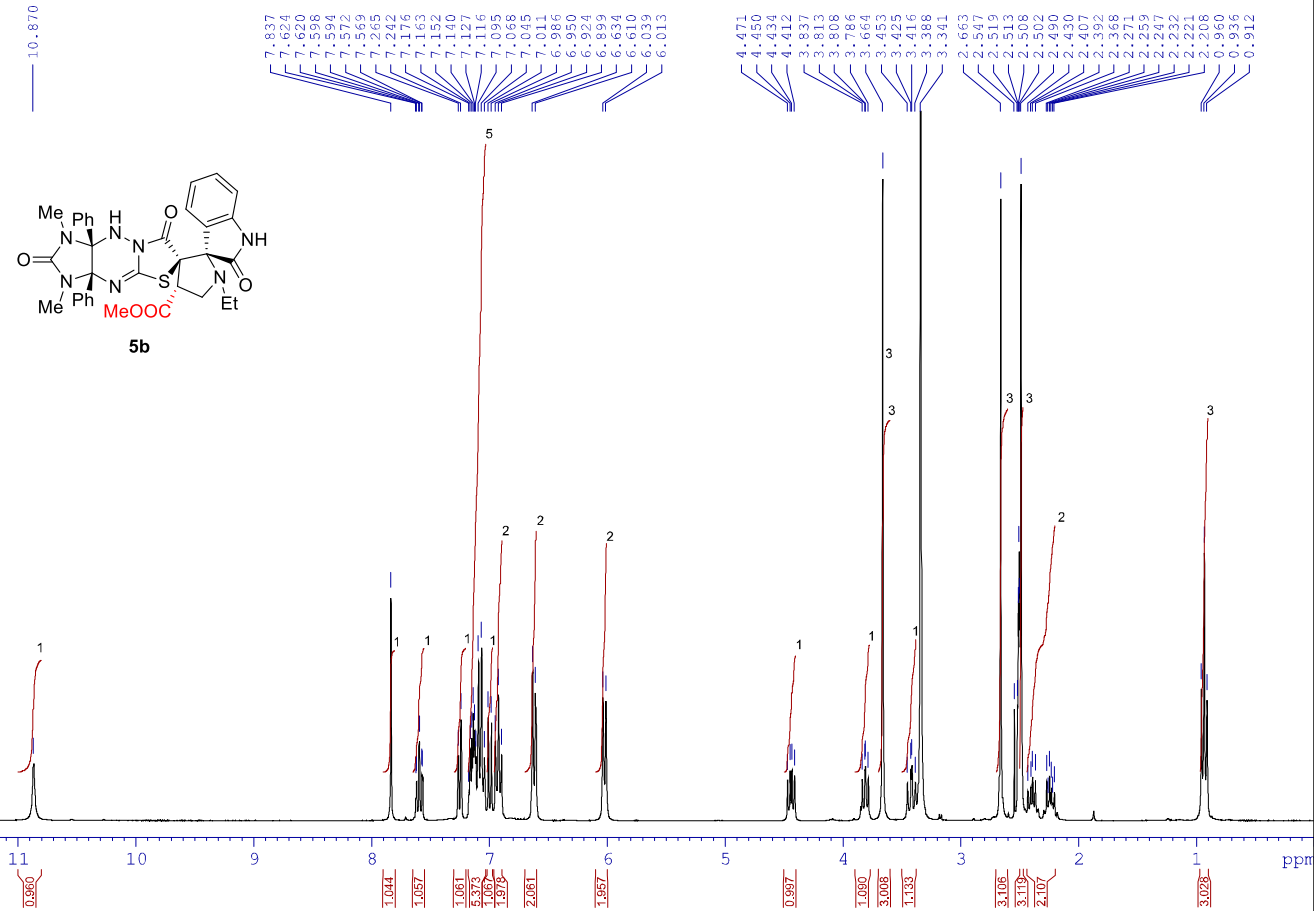
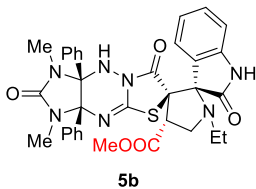
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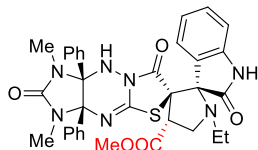
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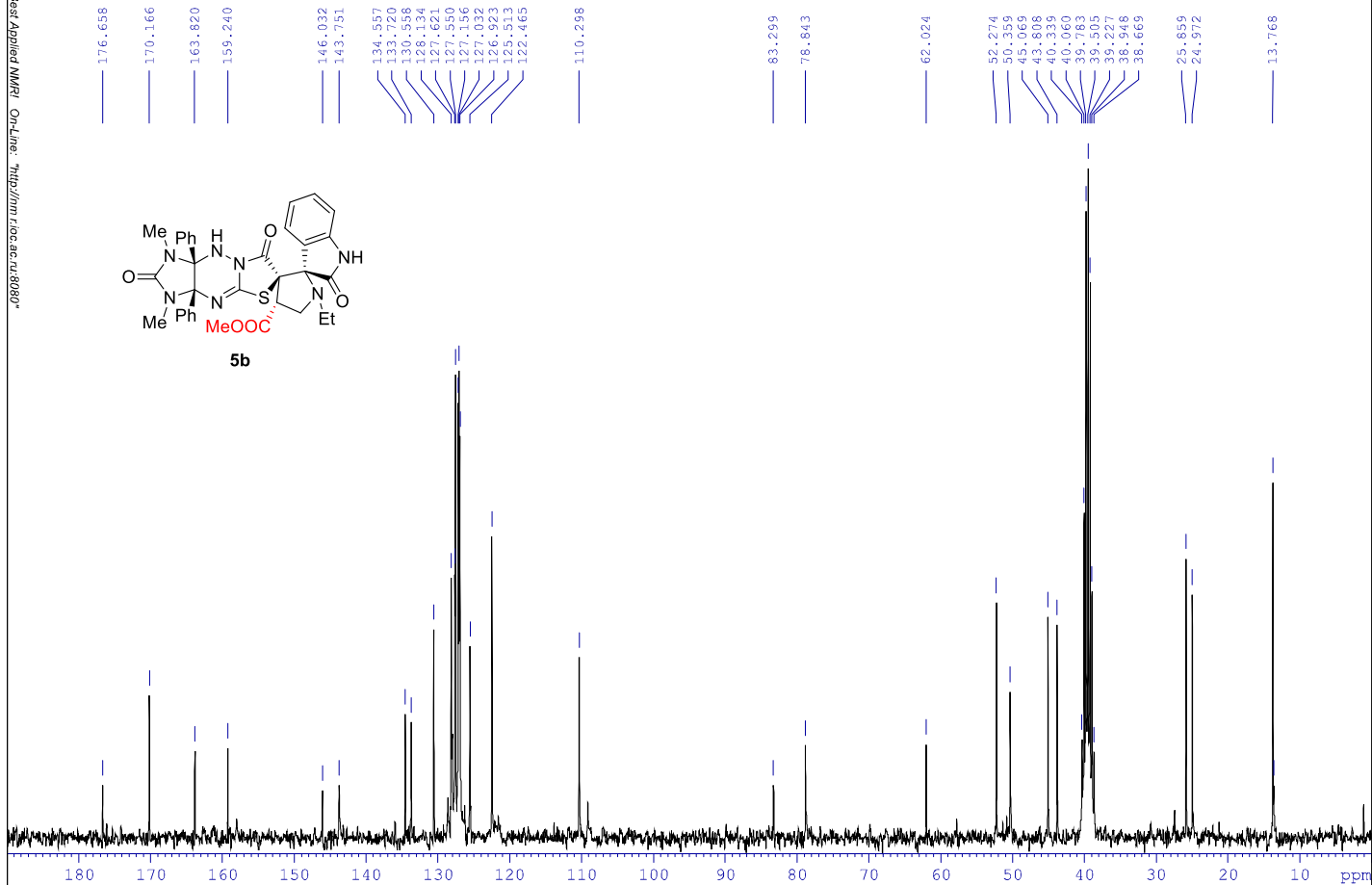
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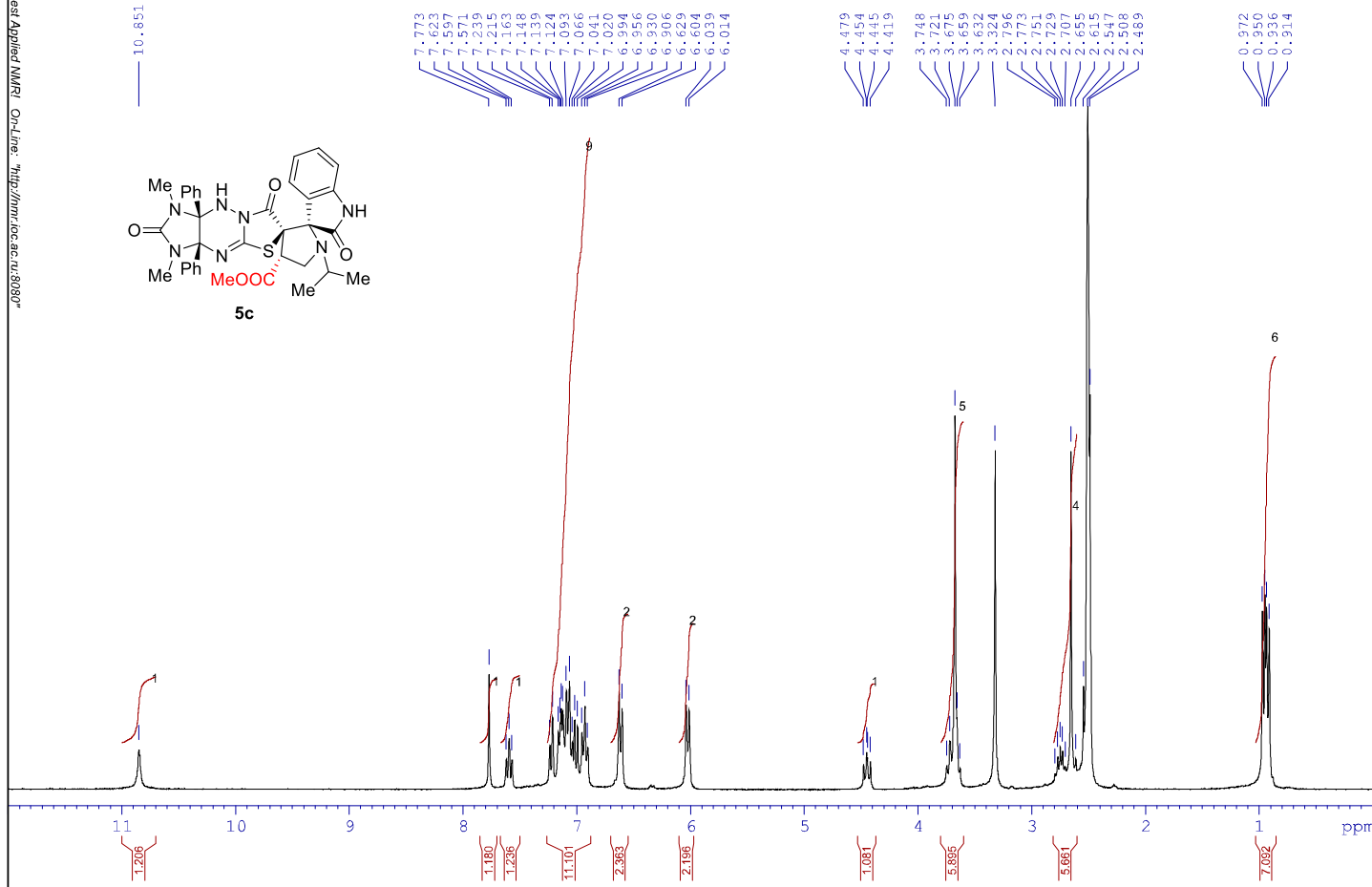
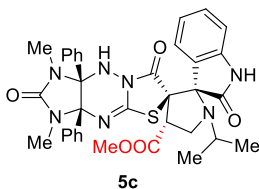
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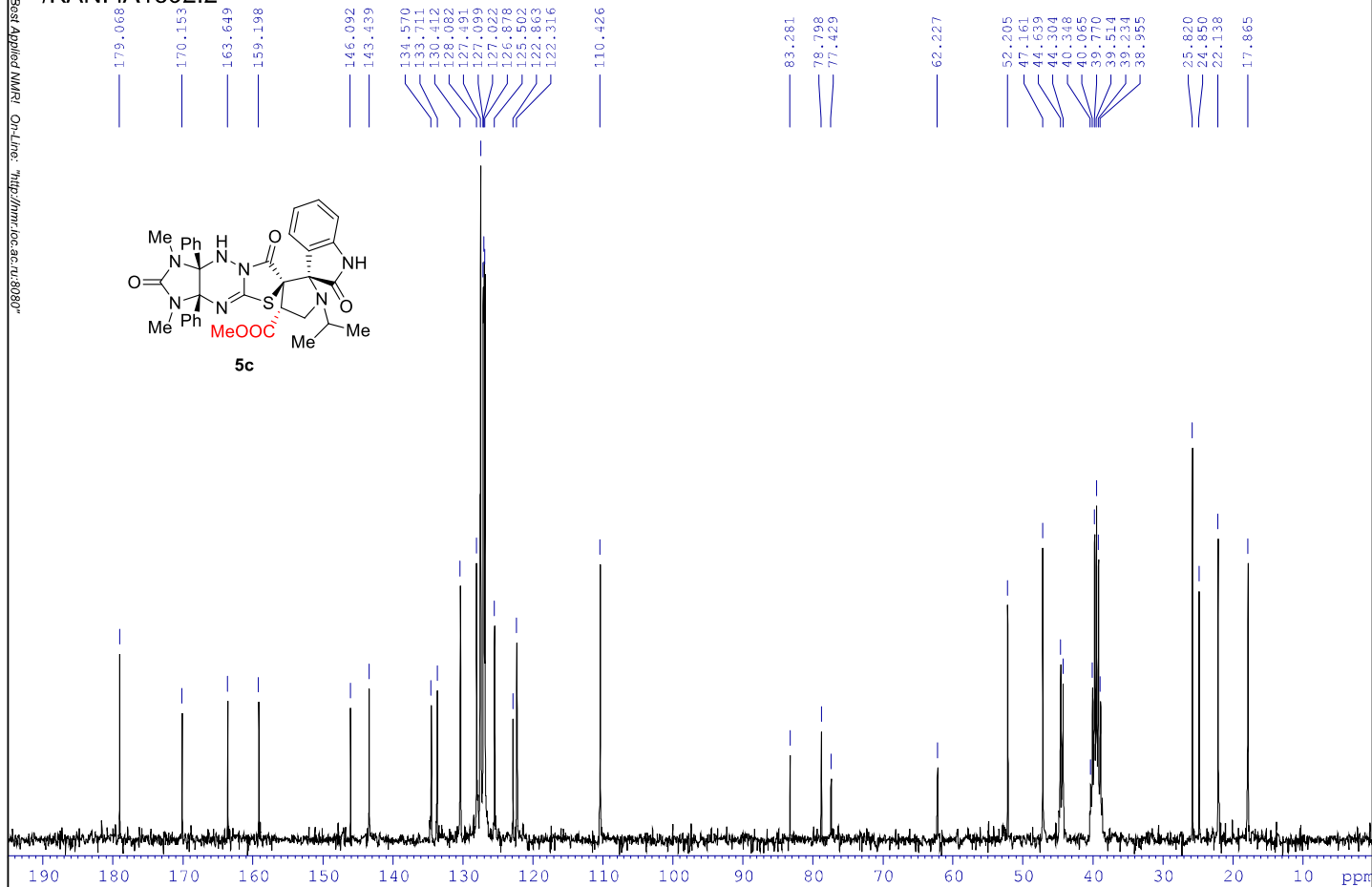
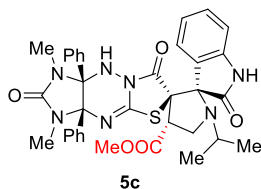
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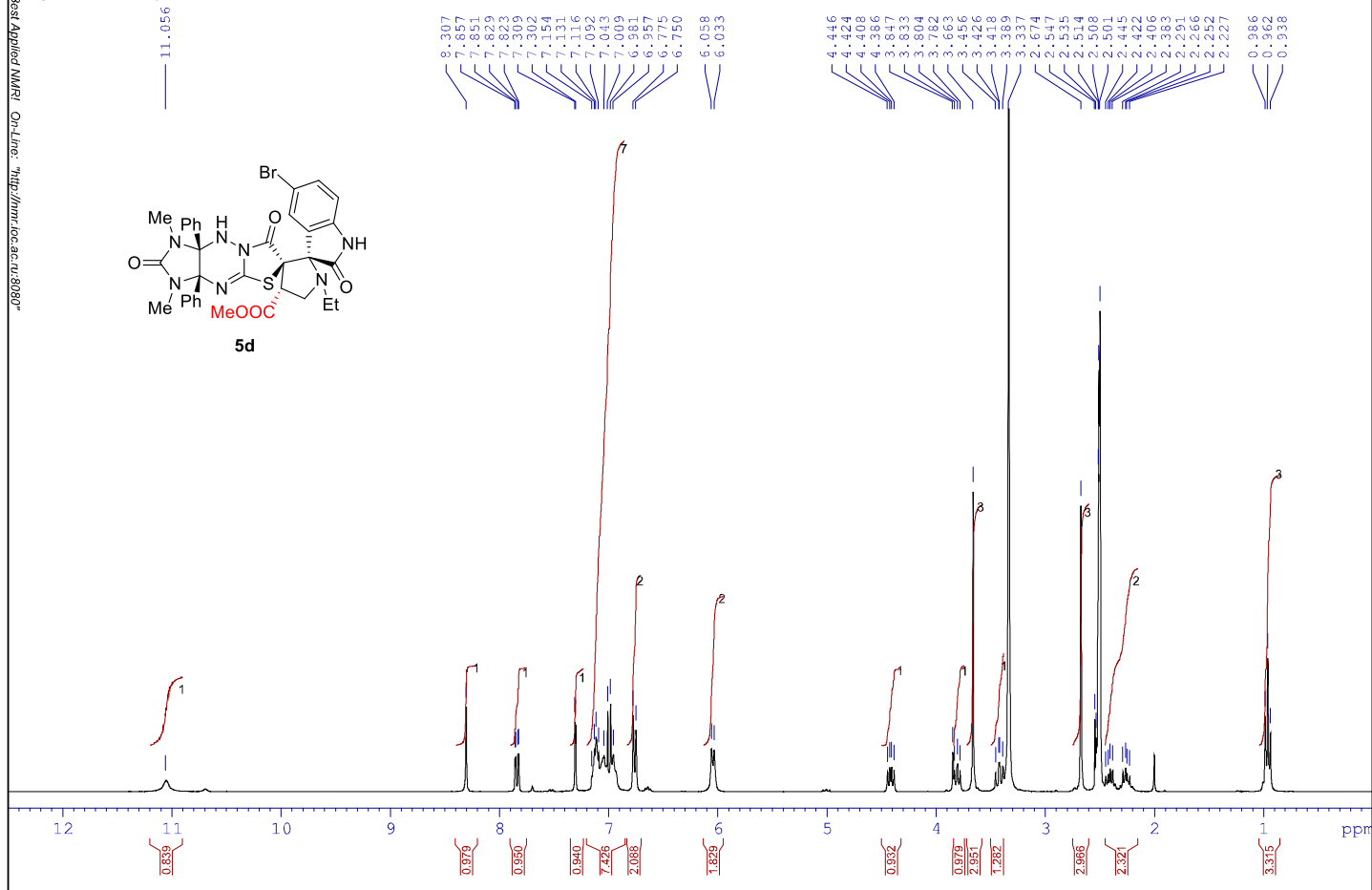
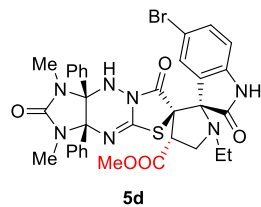
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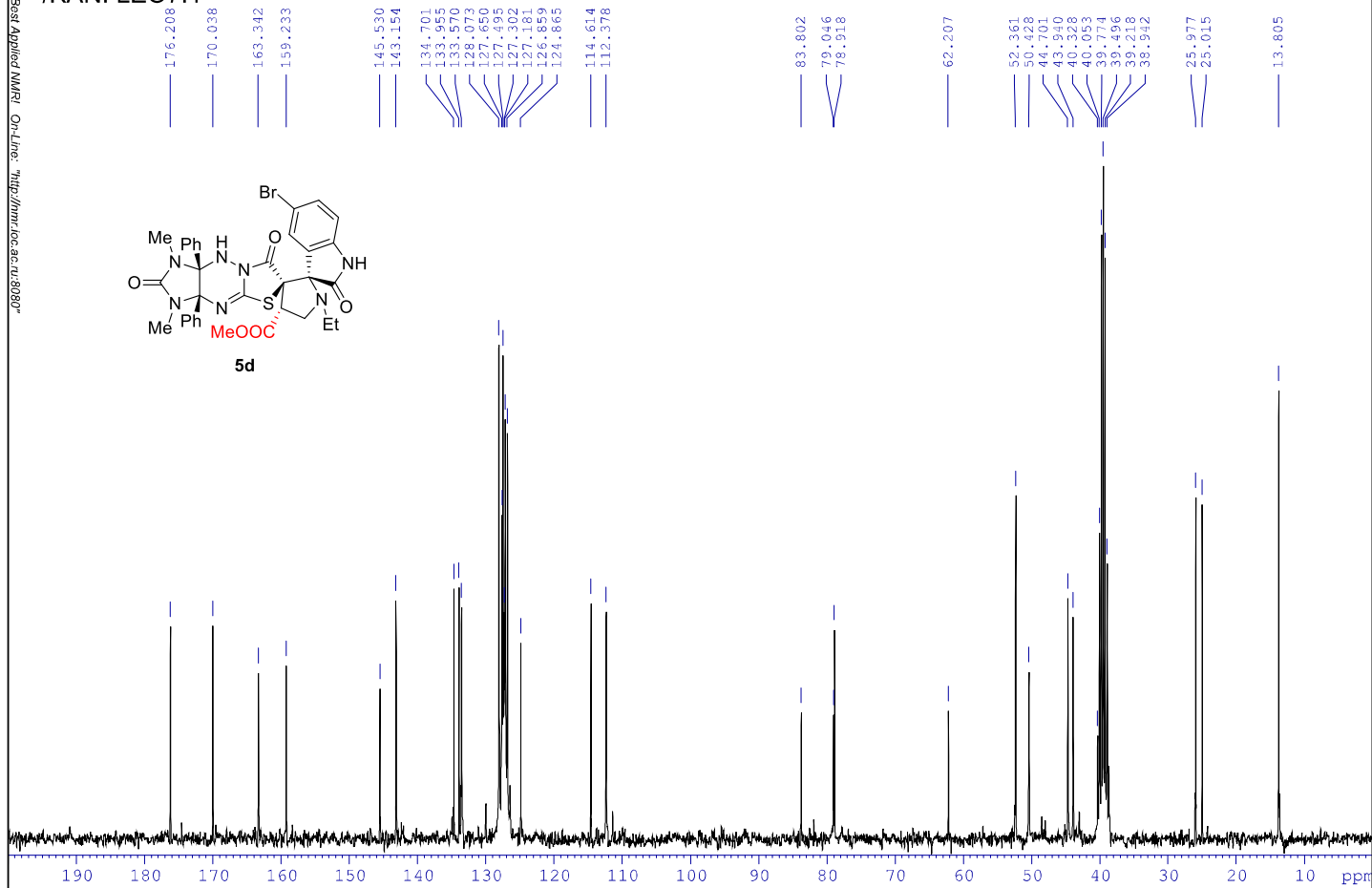
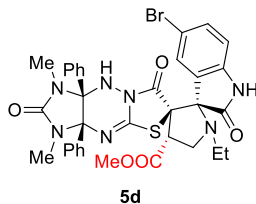
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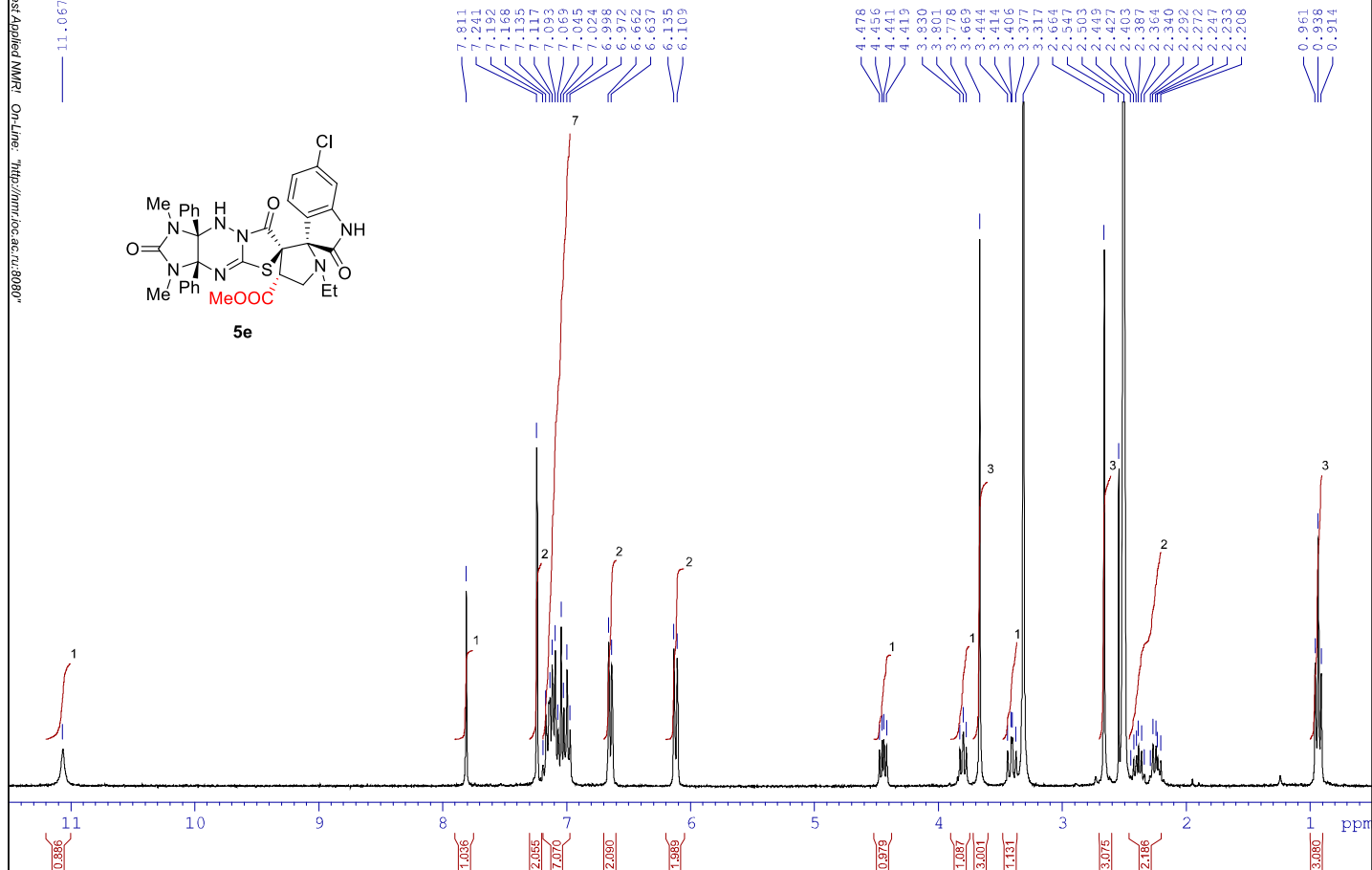
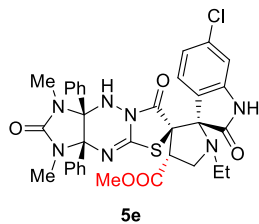
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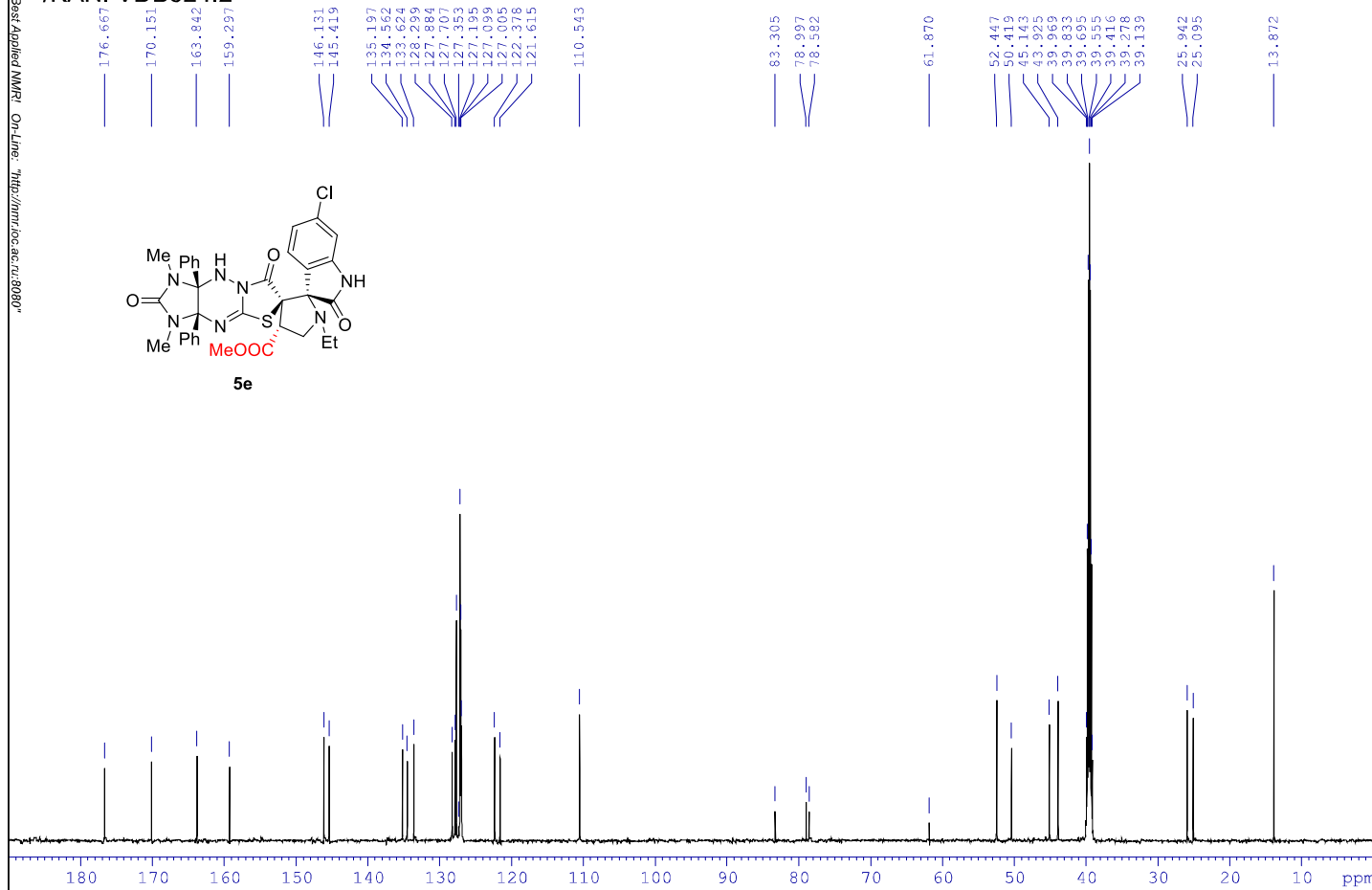
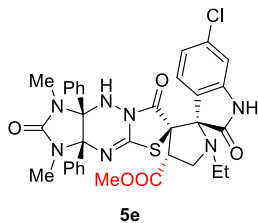
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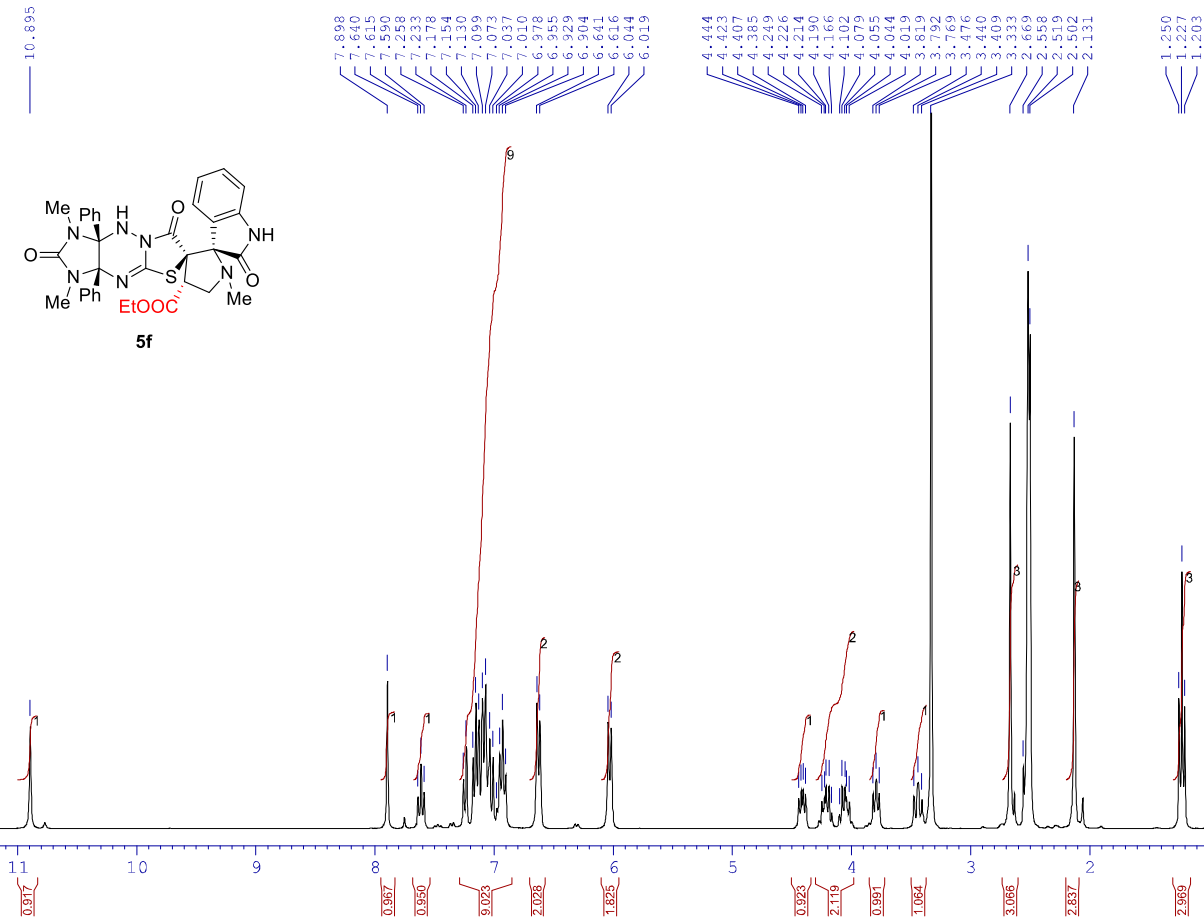
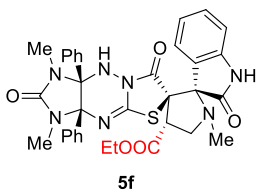
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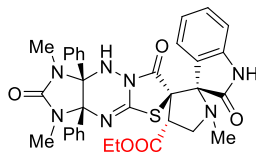
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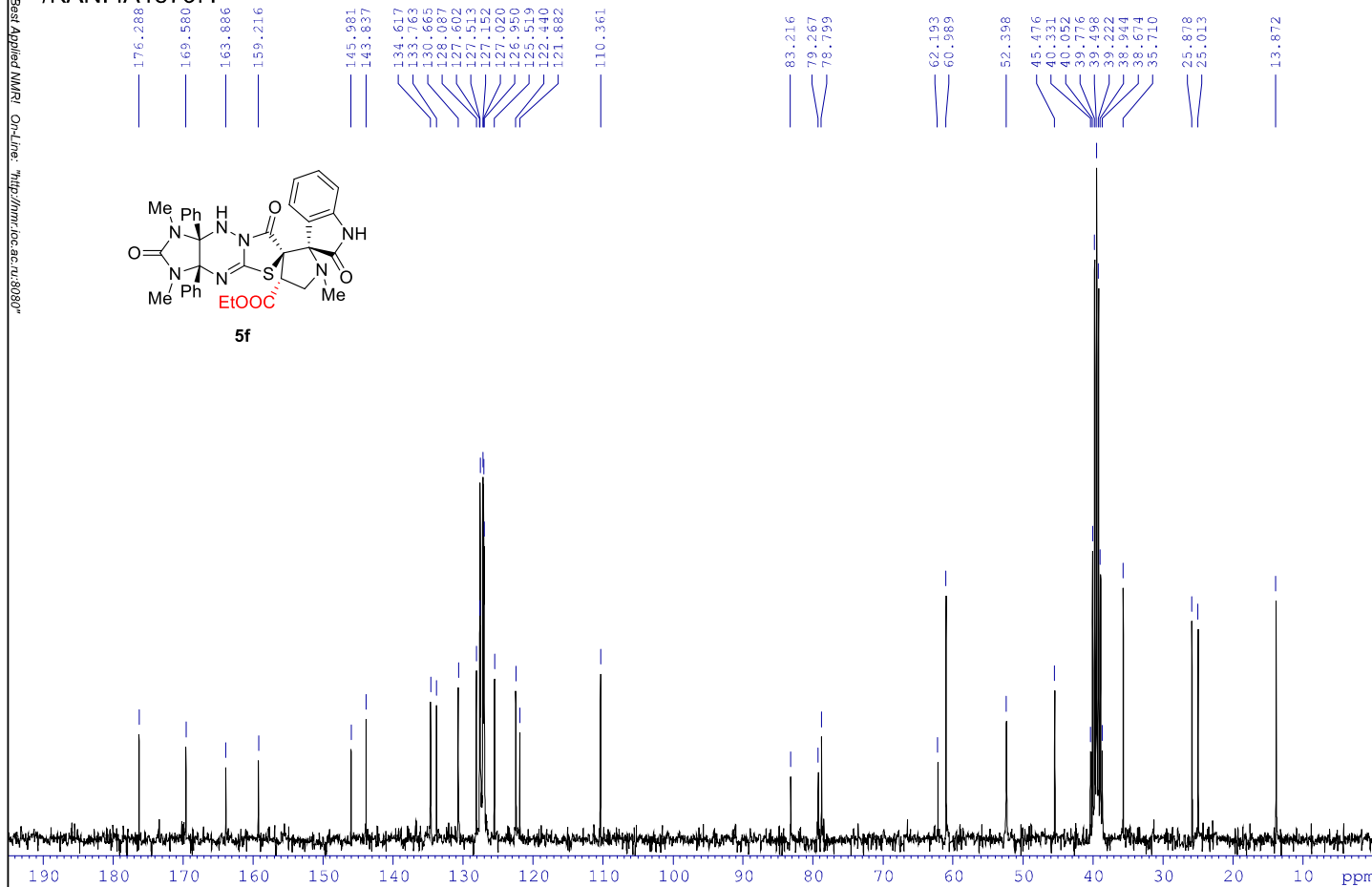
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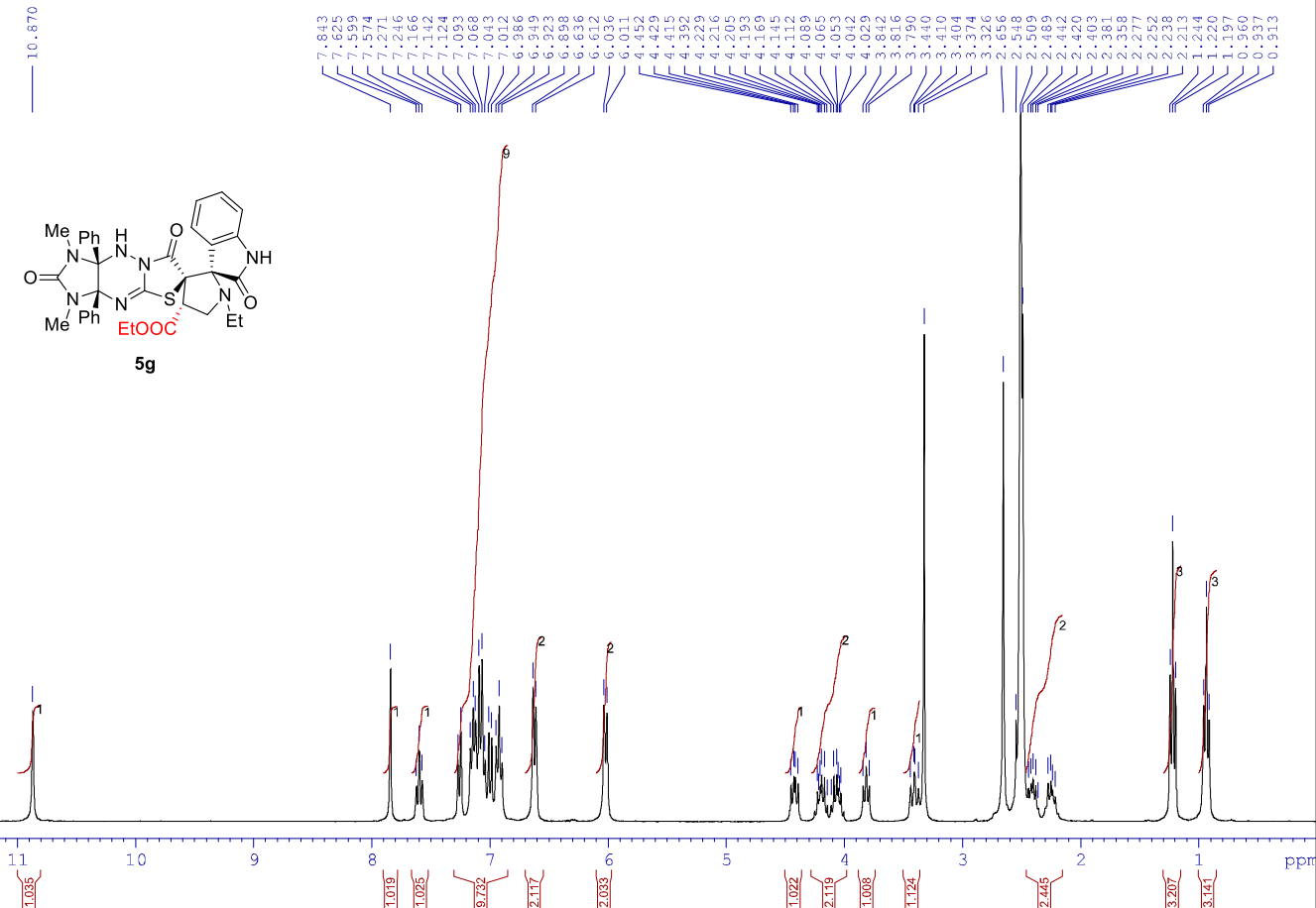
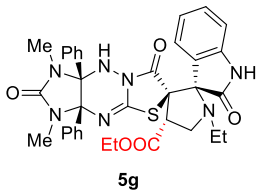
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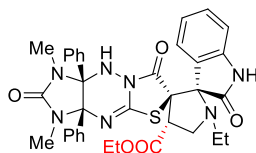
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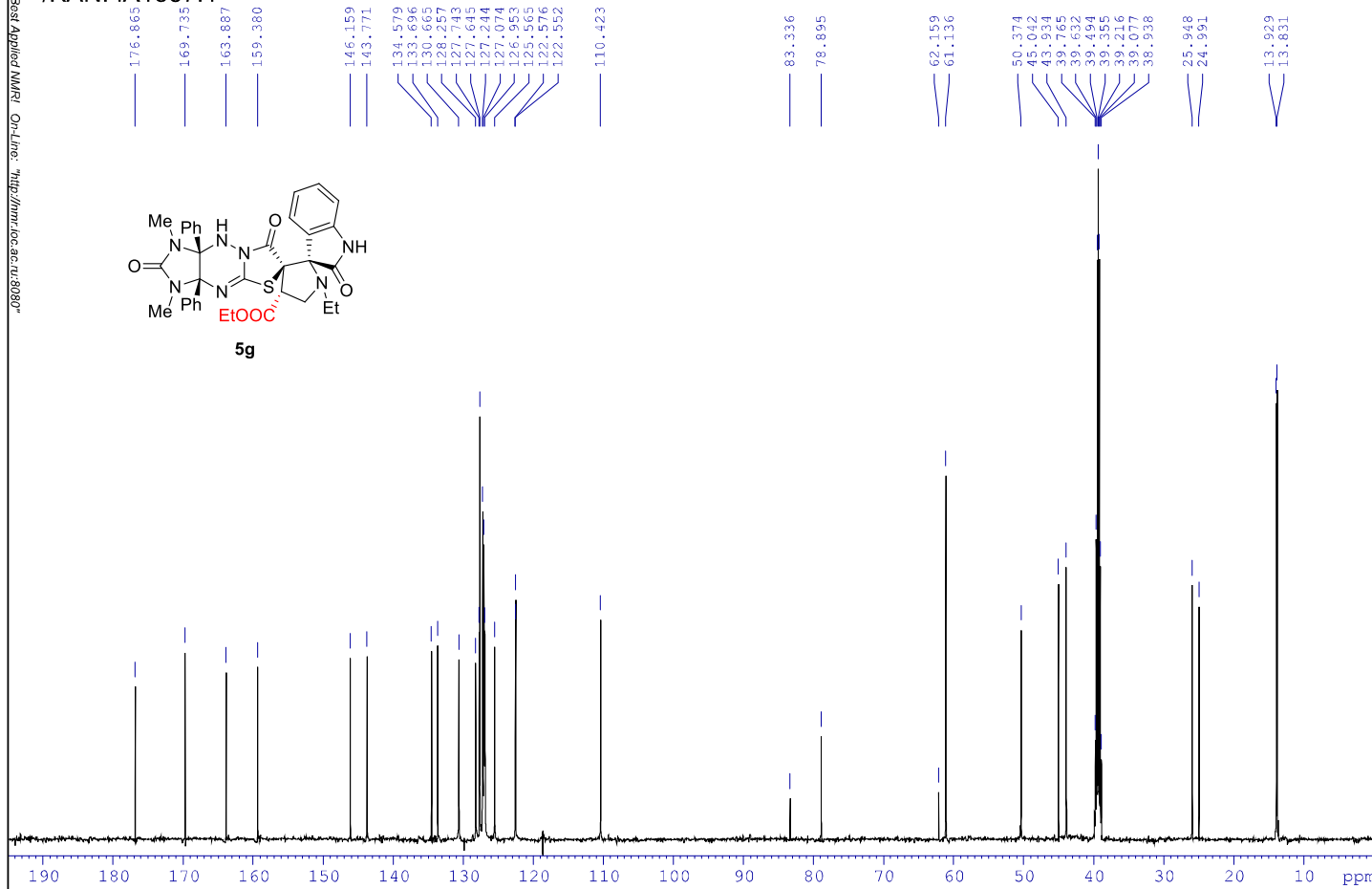
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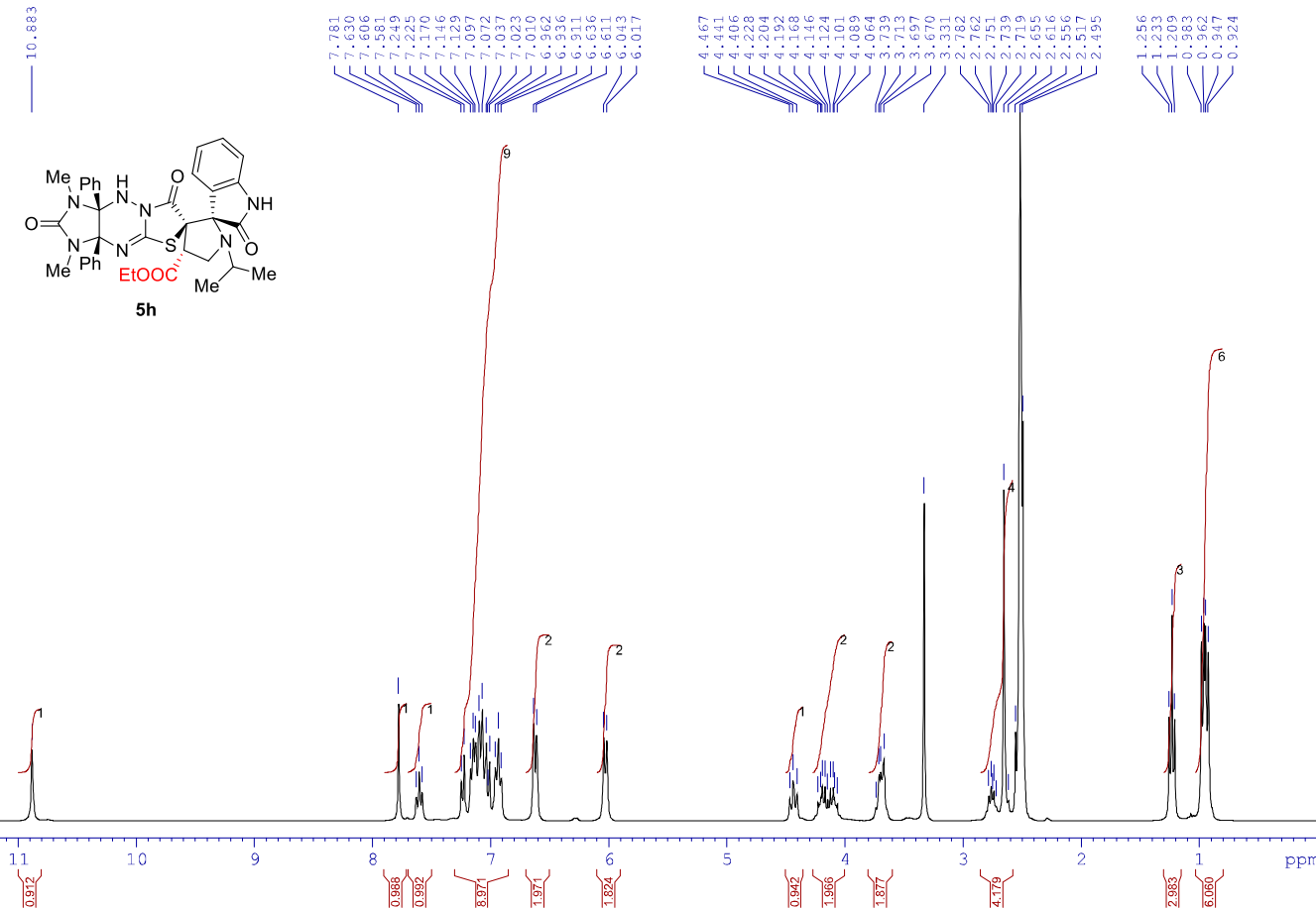
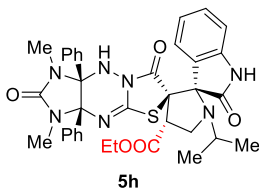
/KANI IA1587.1



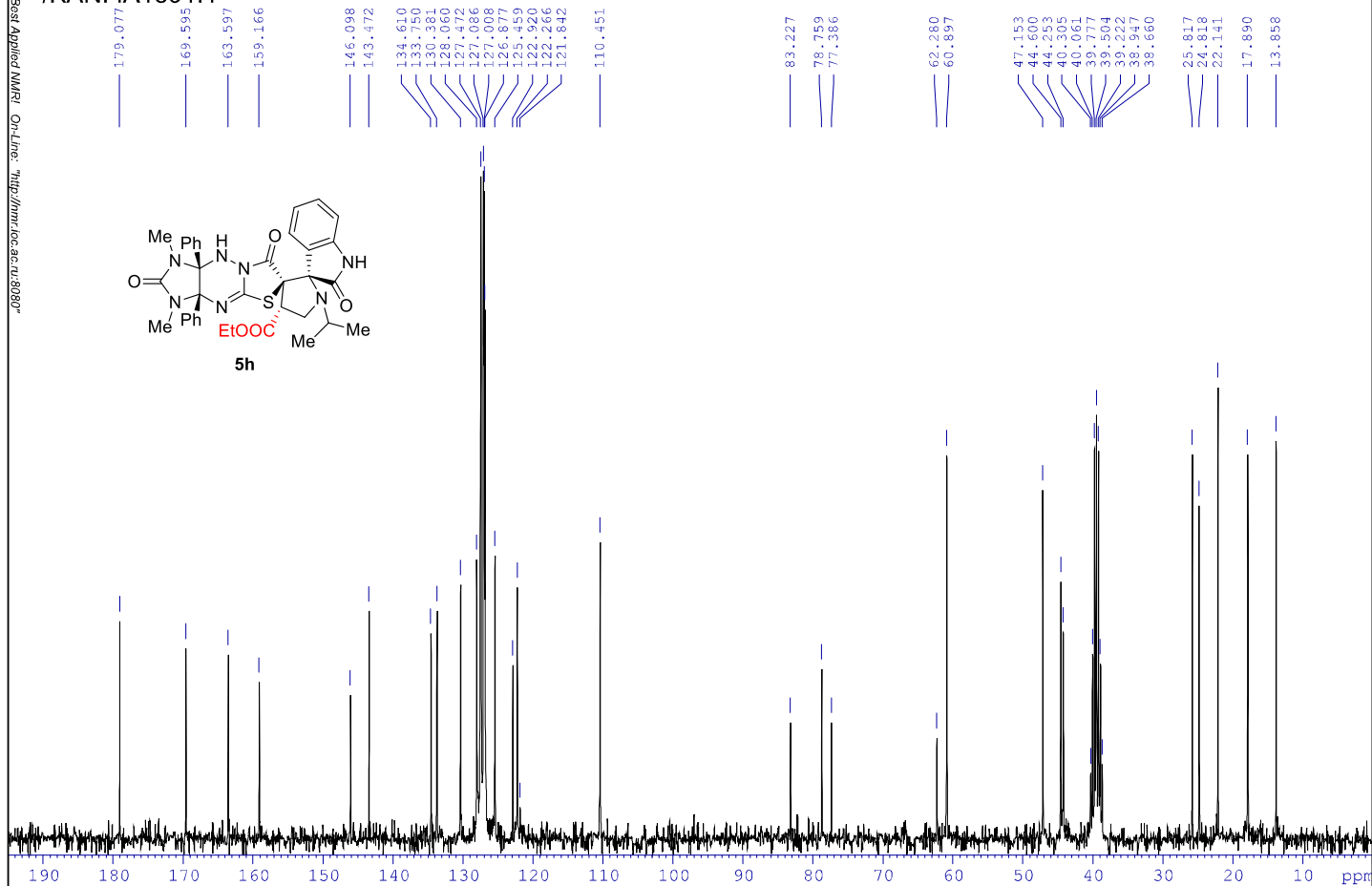
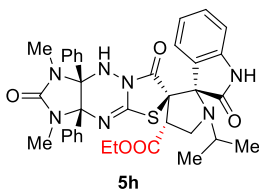
5g



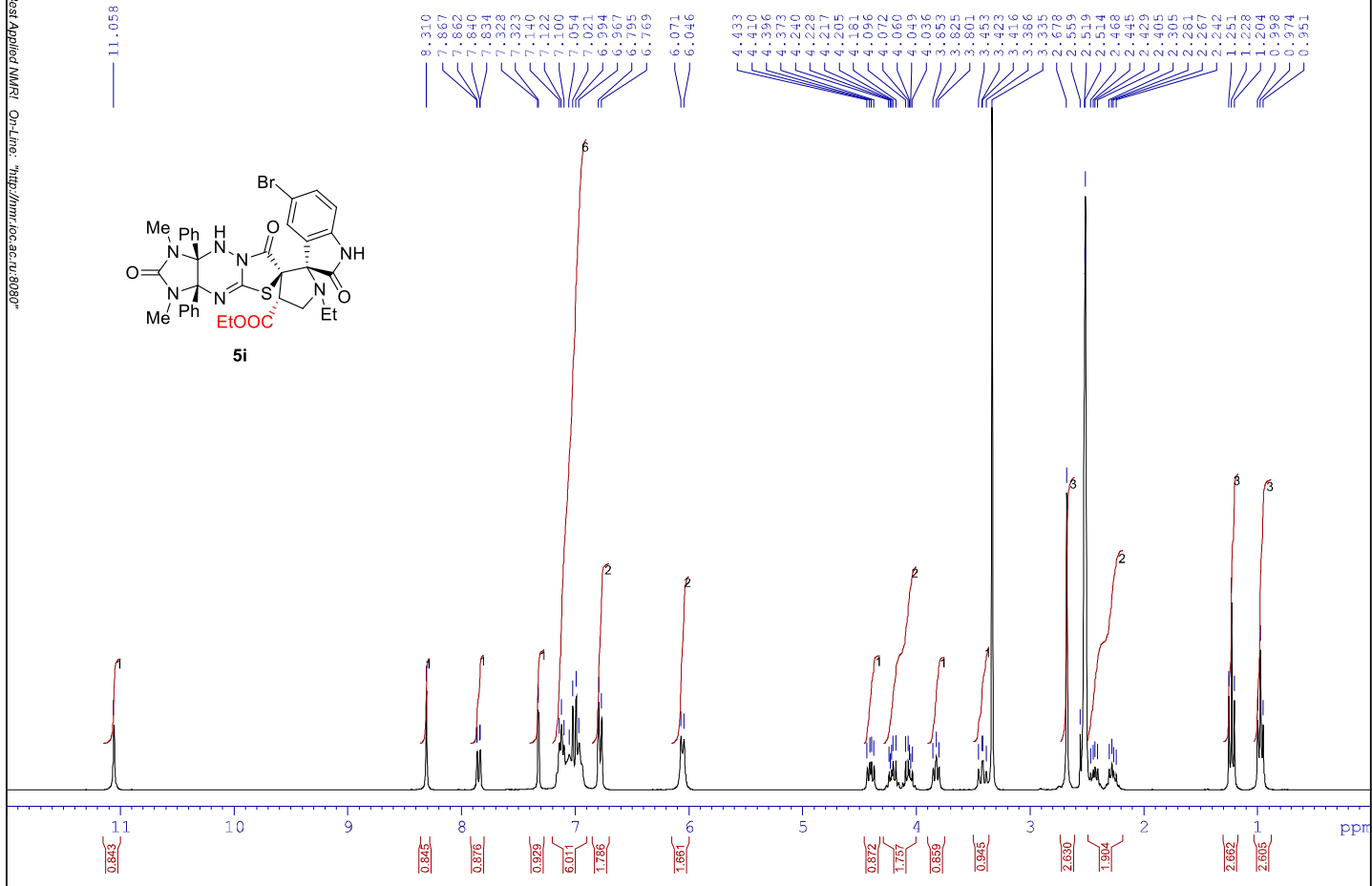
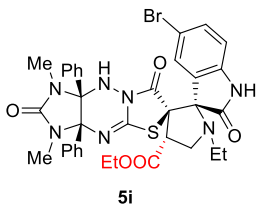
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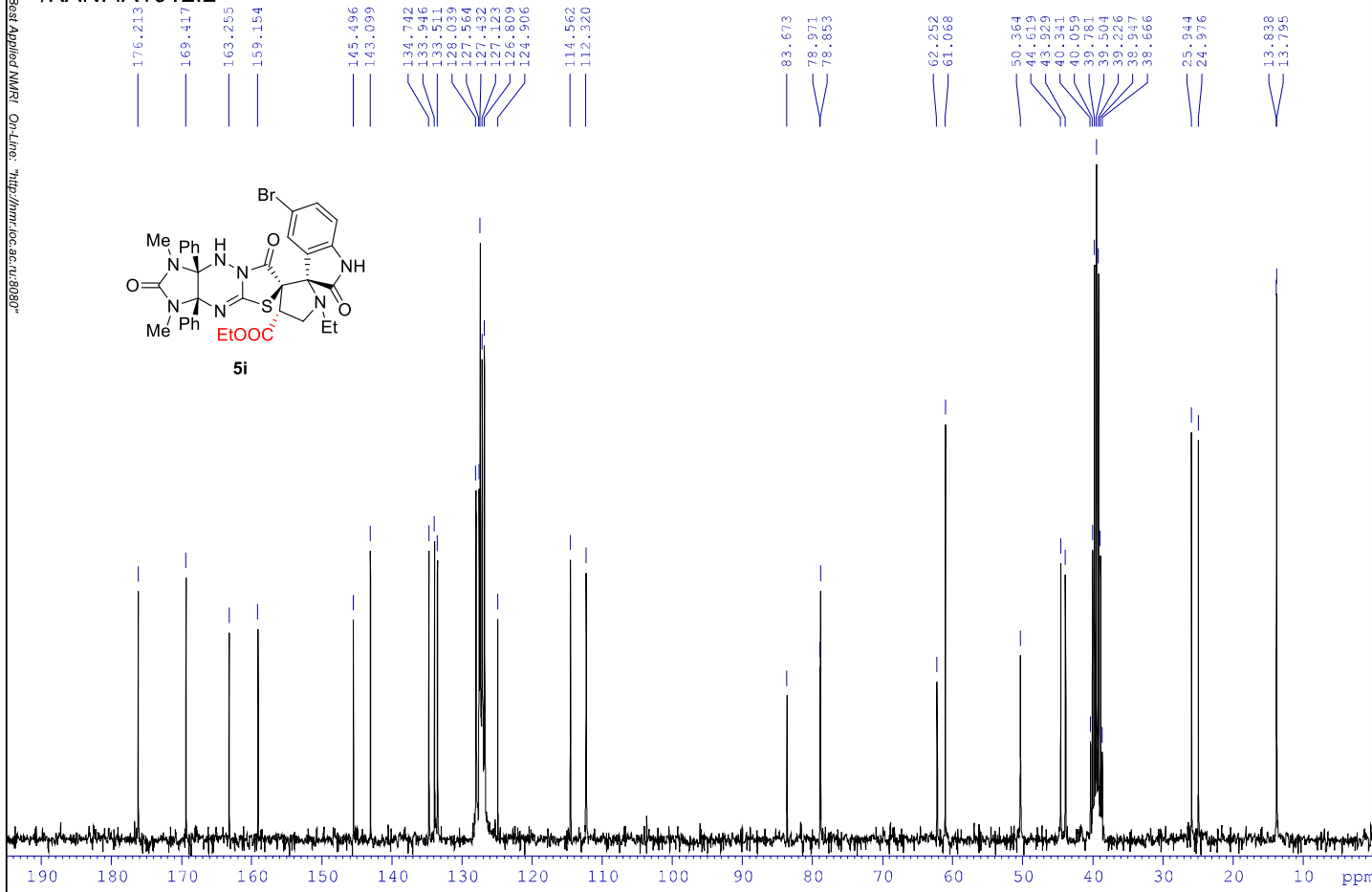
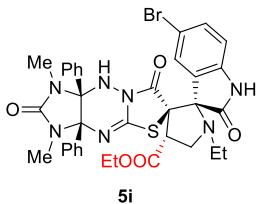
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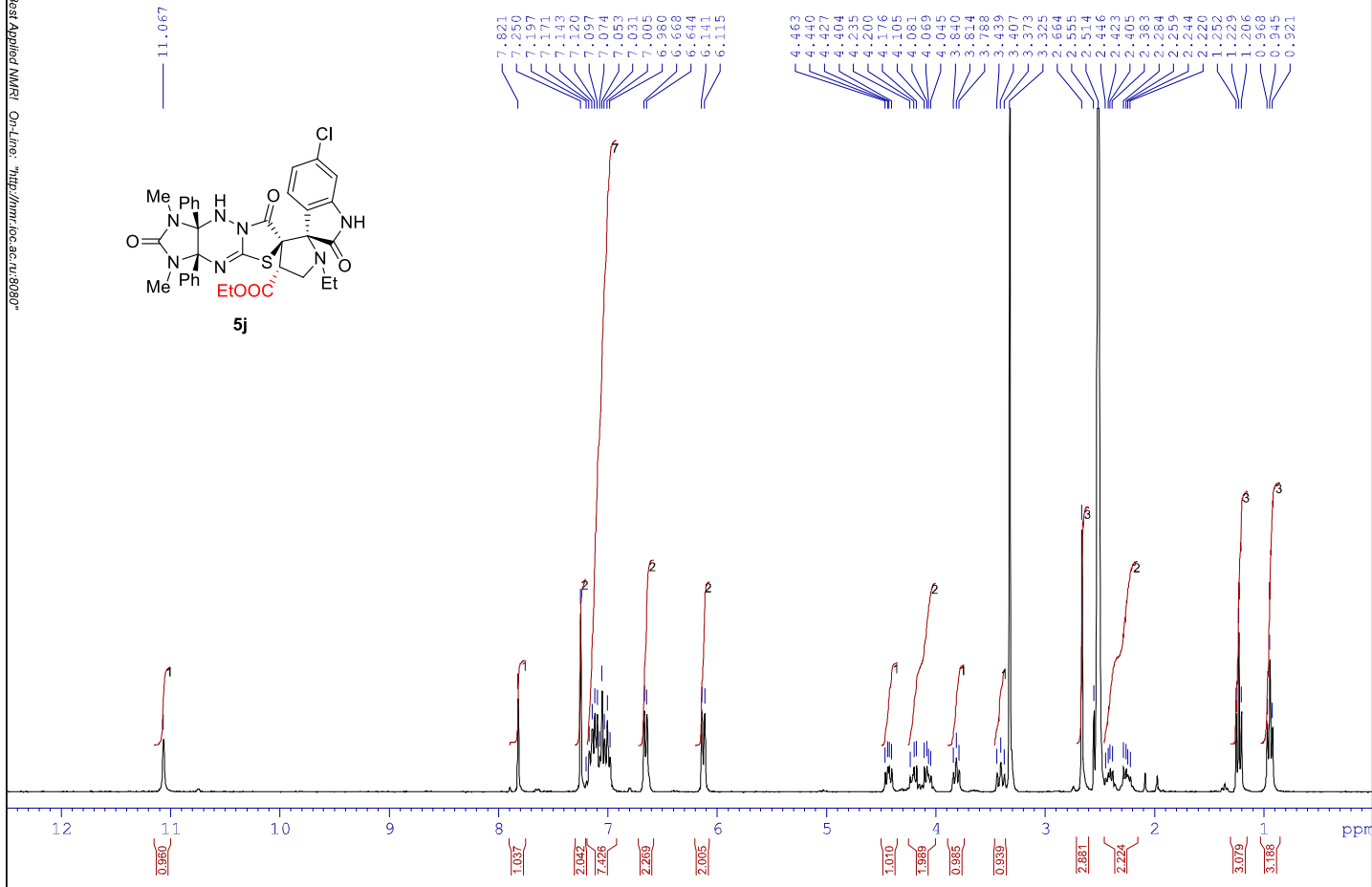
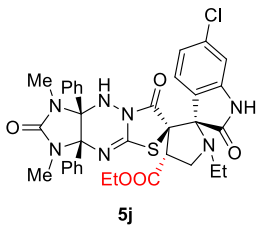
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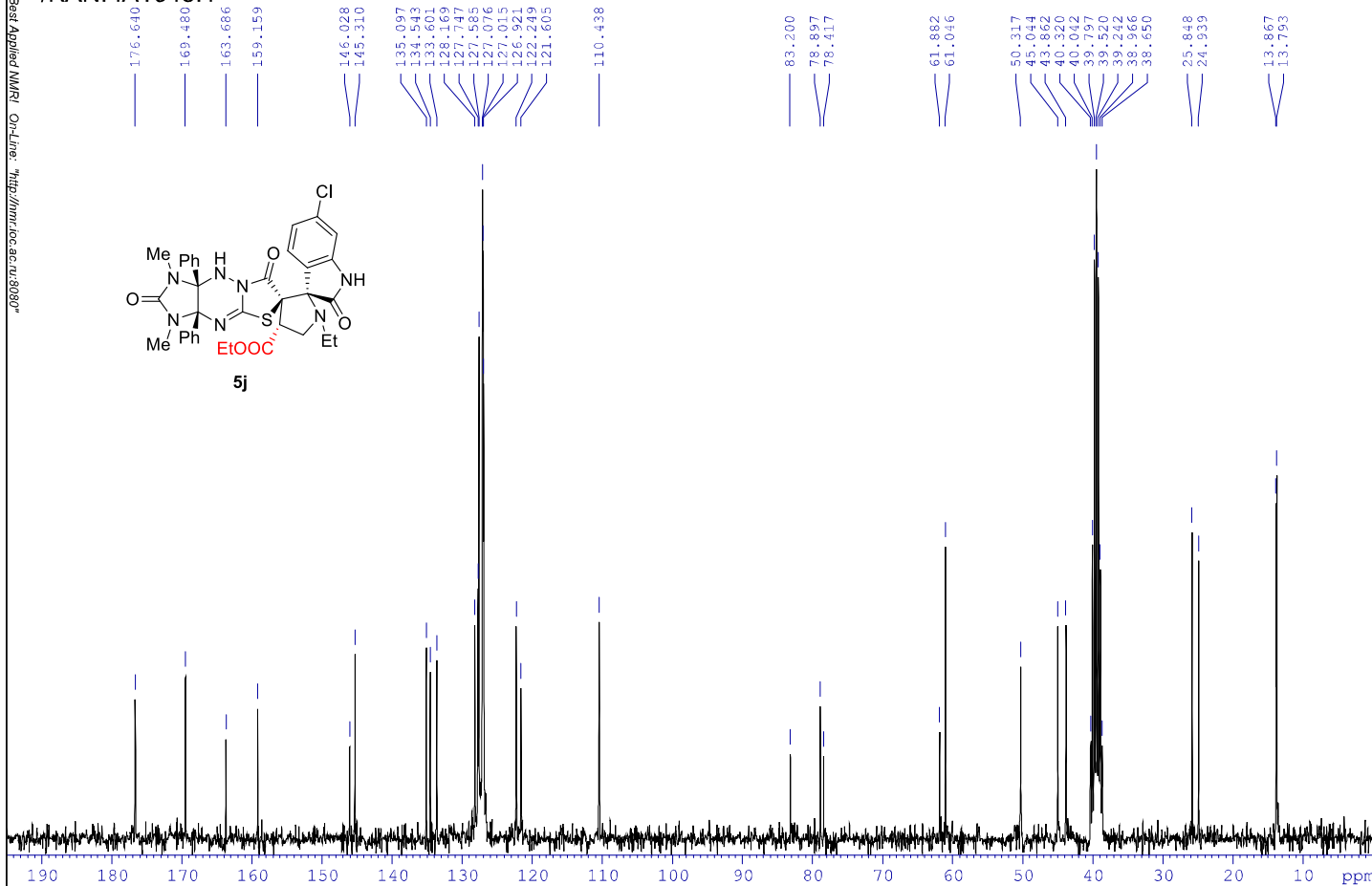
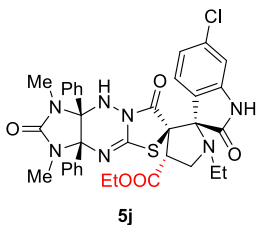
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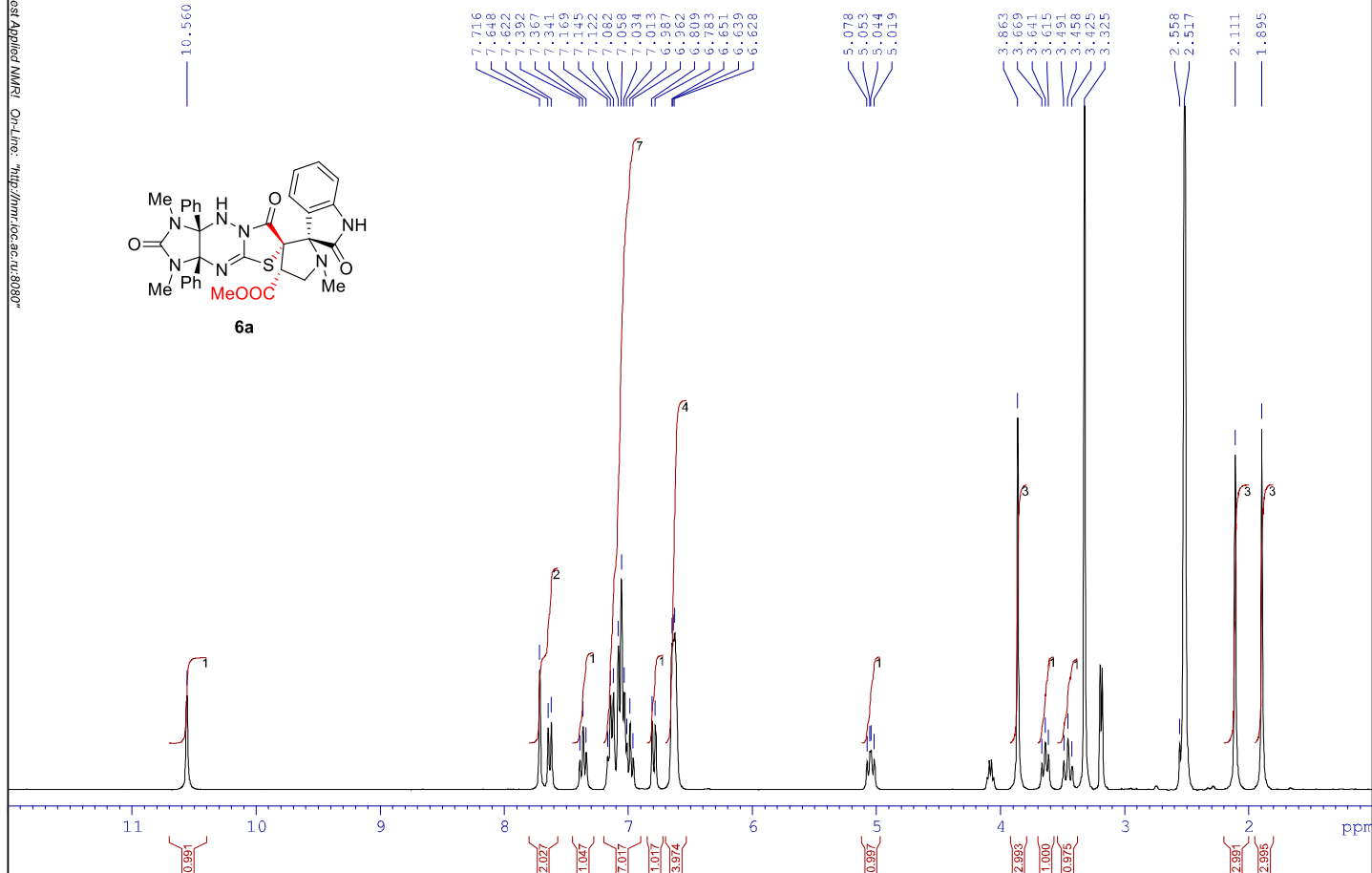
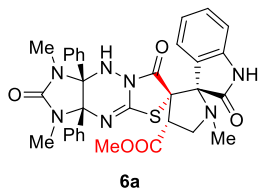
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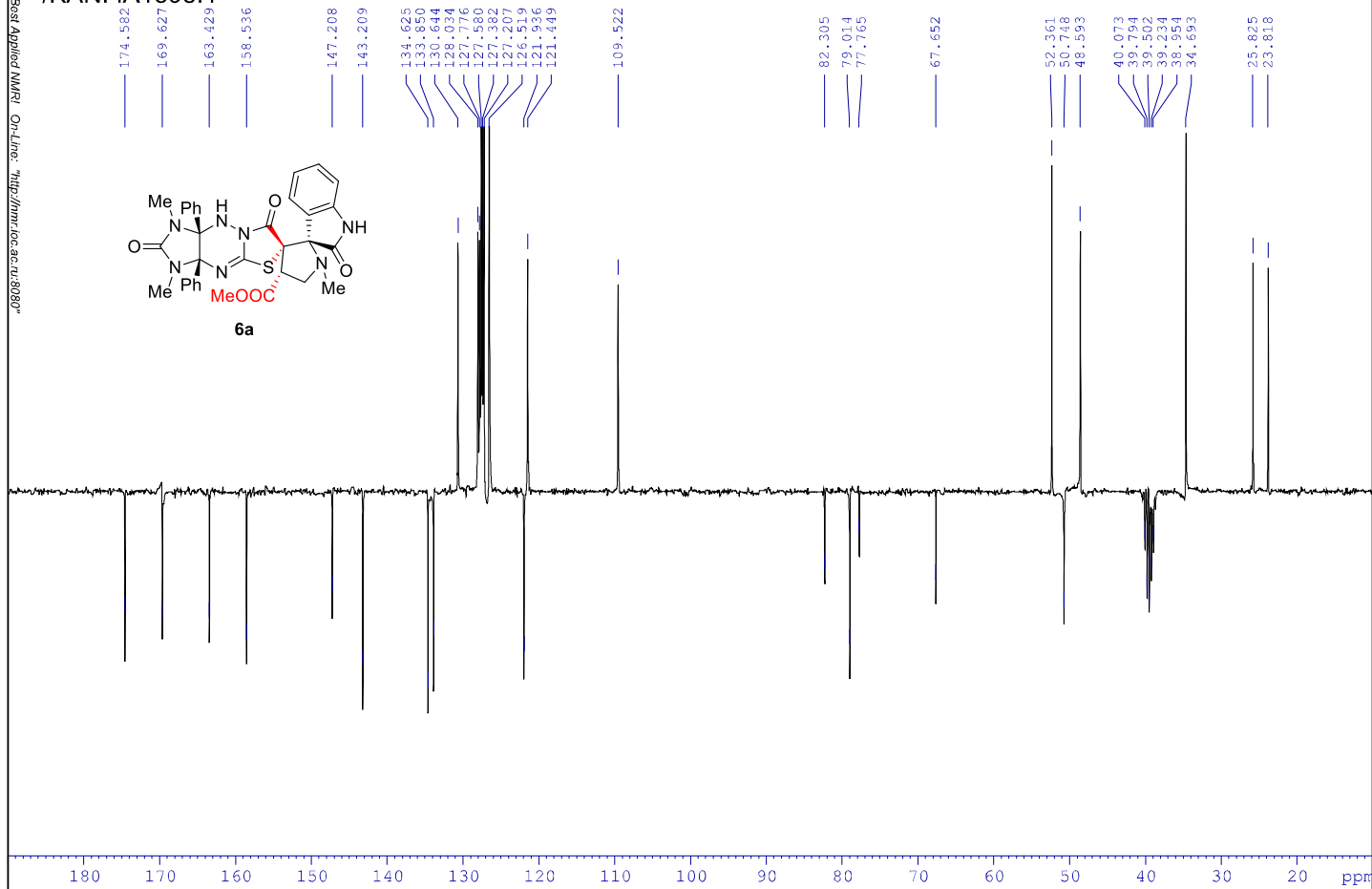
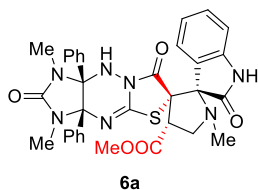
/KANI IA1645.1



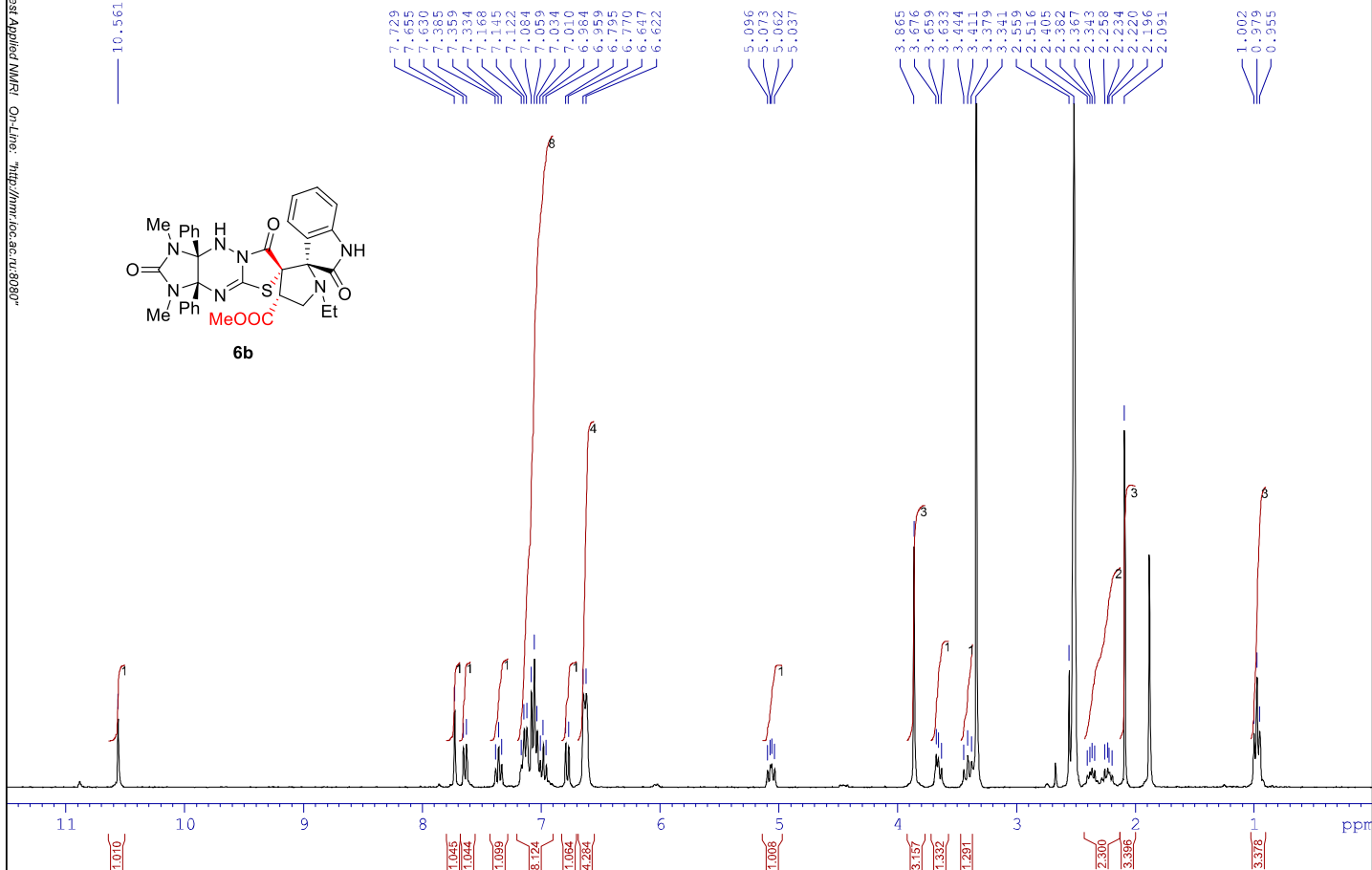
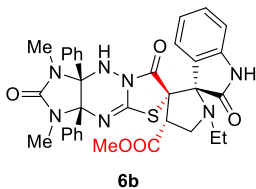
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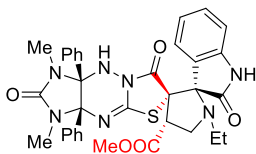
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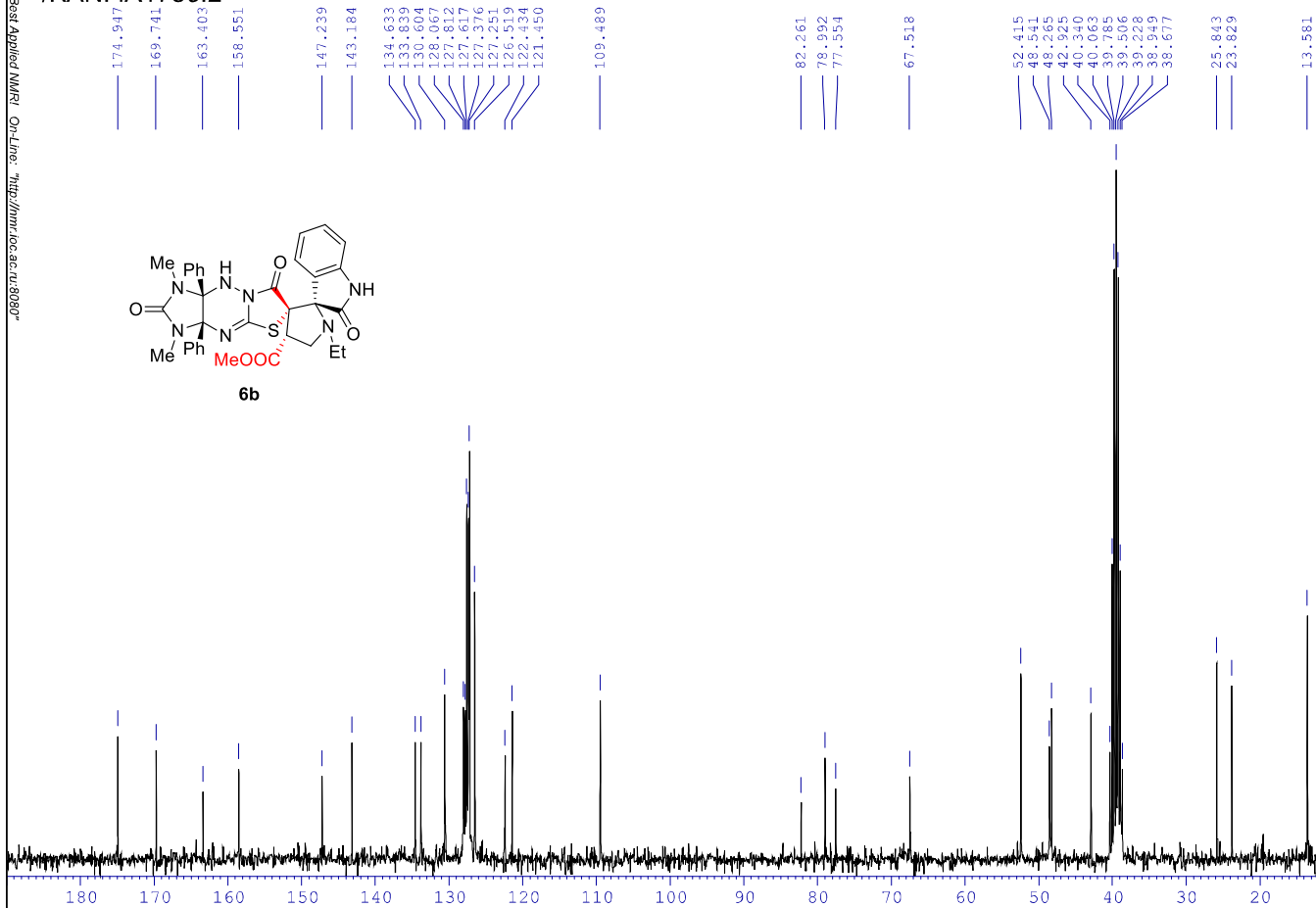
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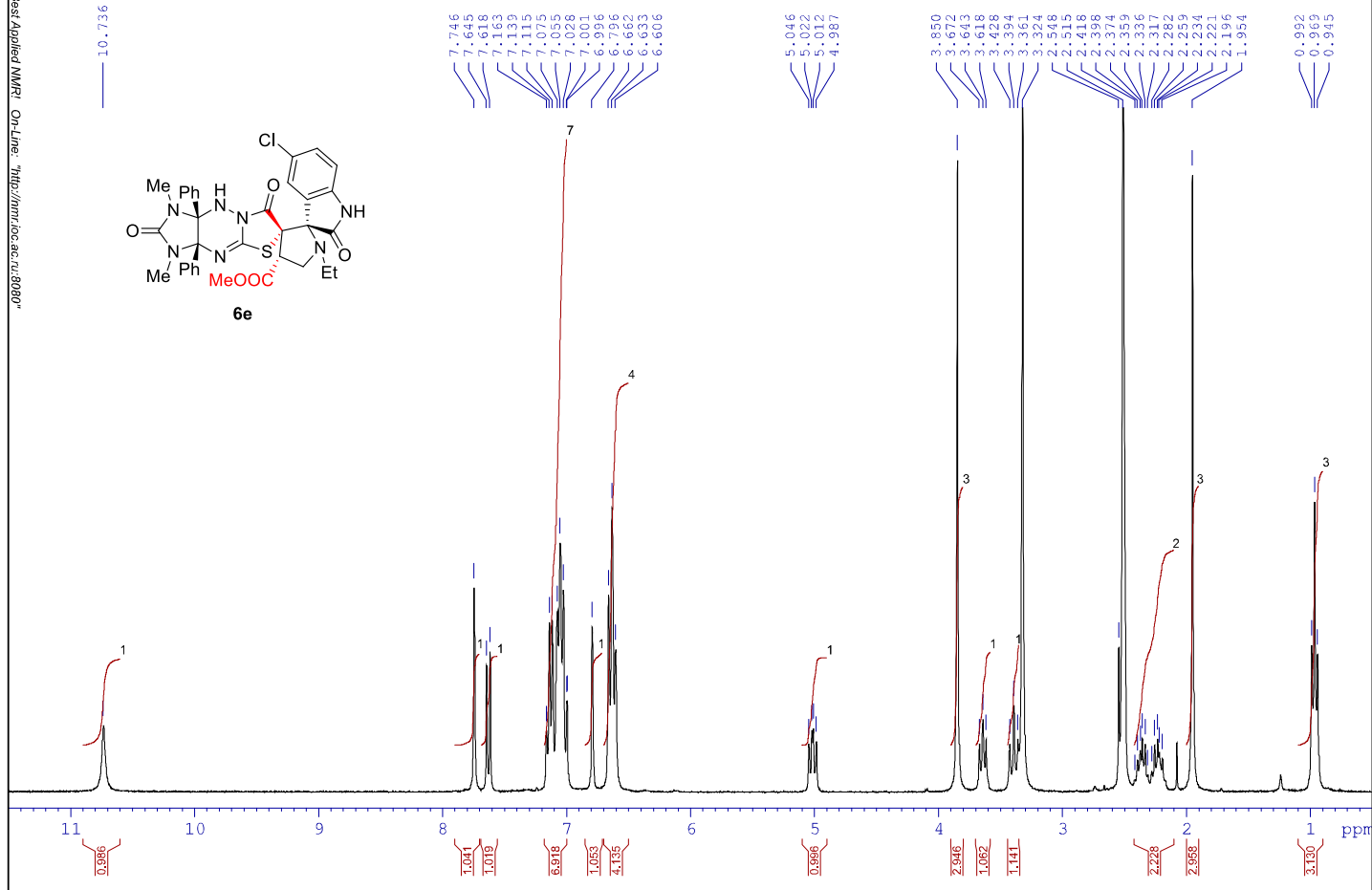
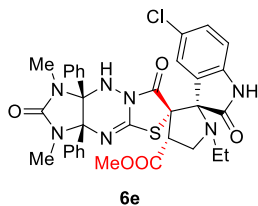
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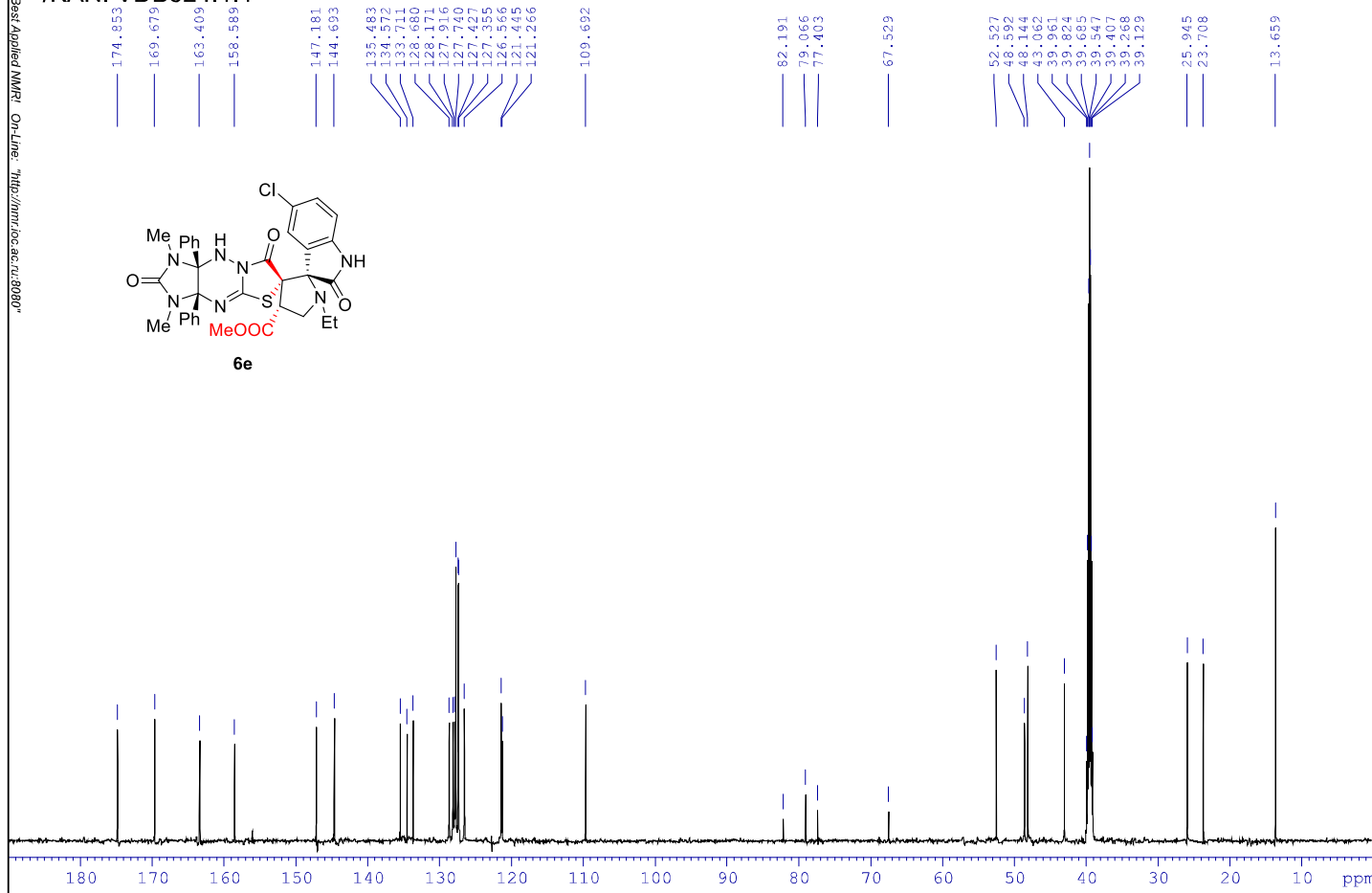
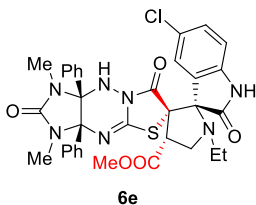
6b



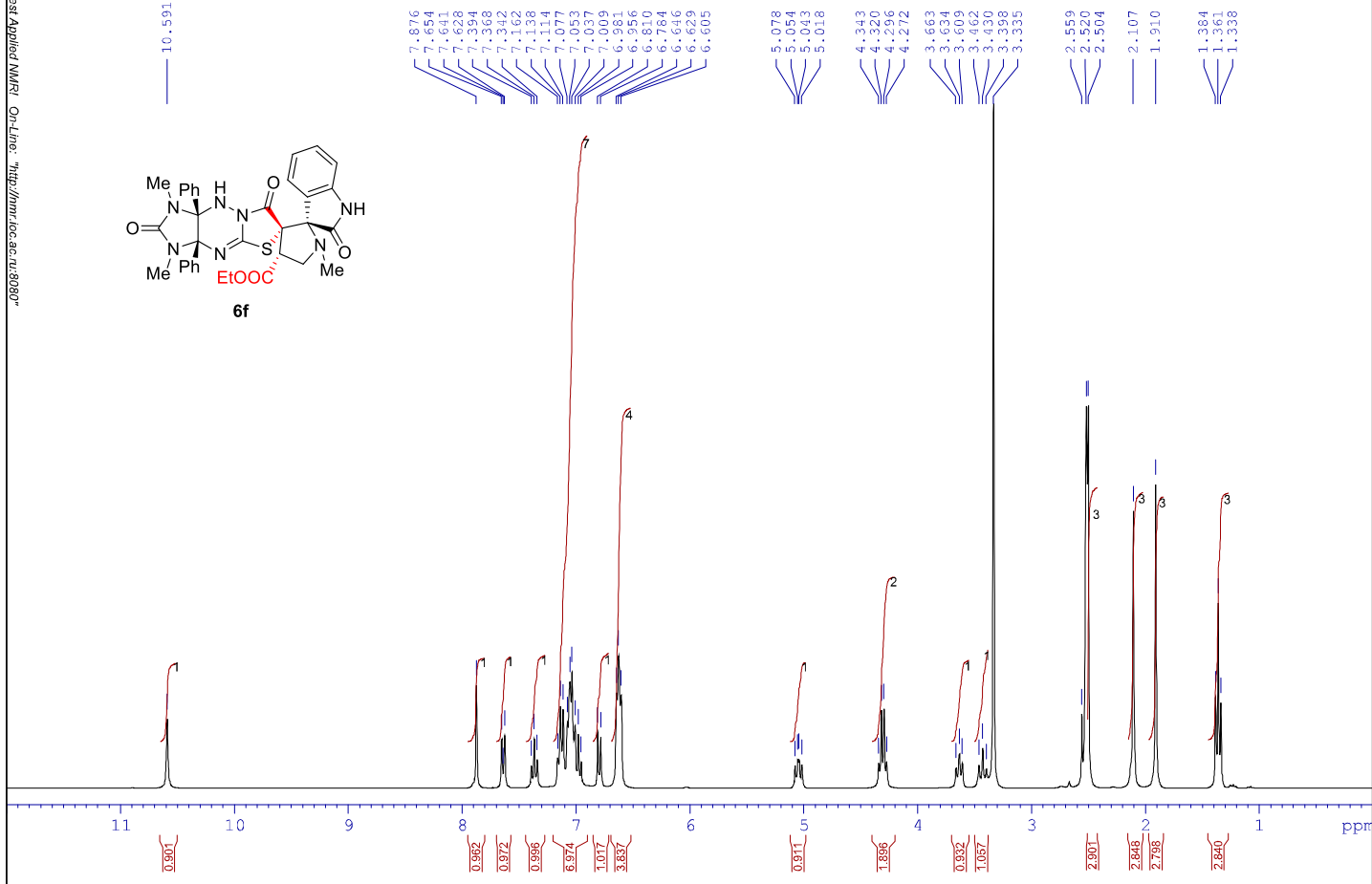
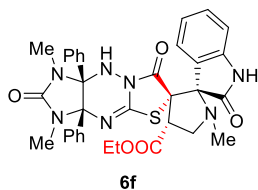
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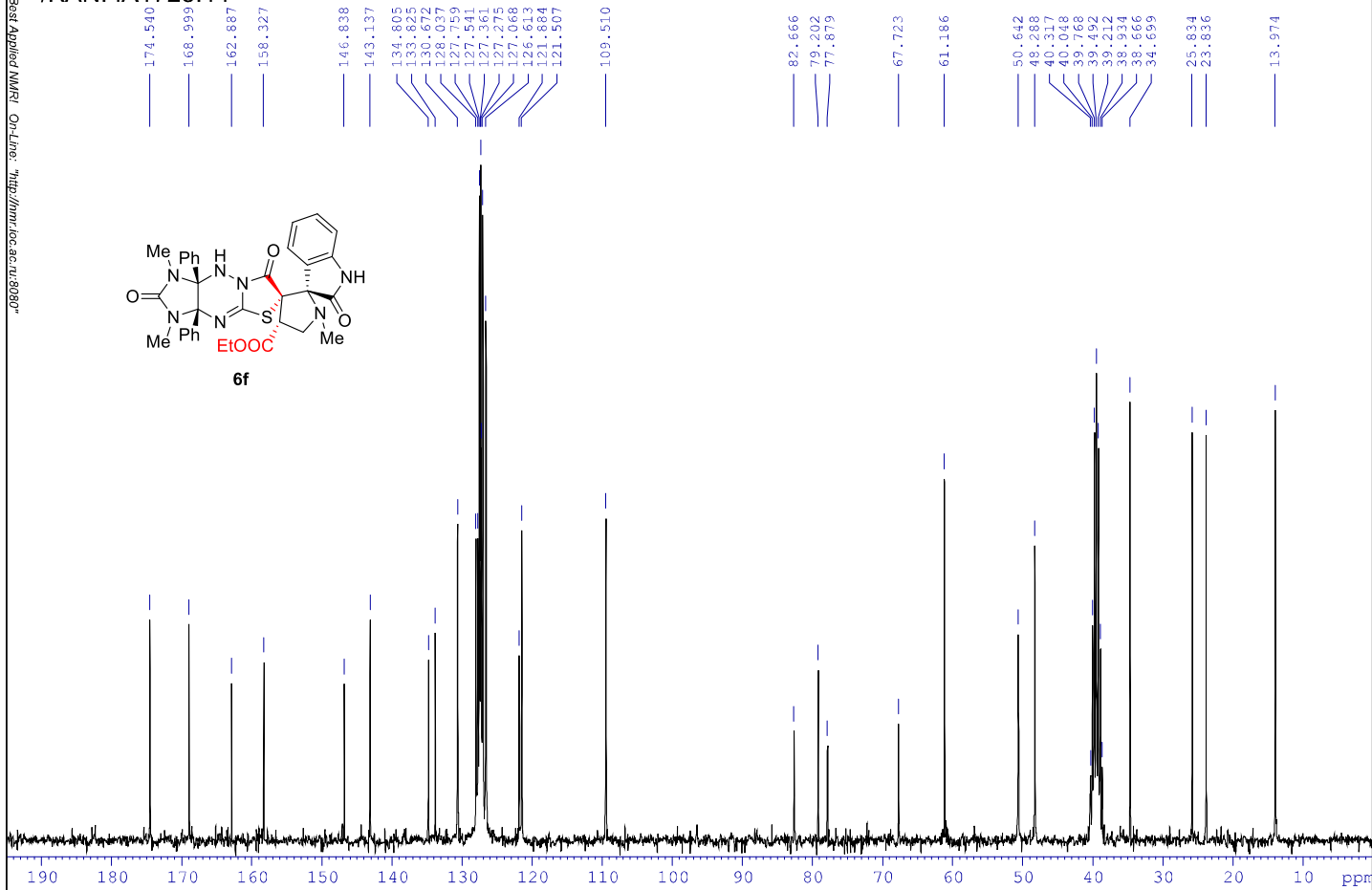
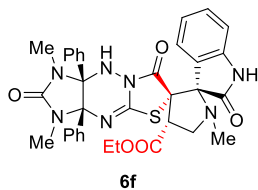
/KANI VDB524.1.1



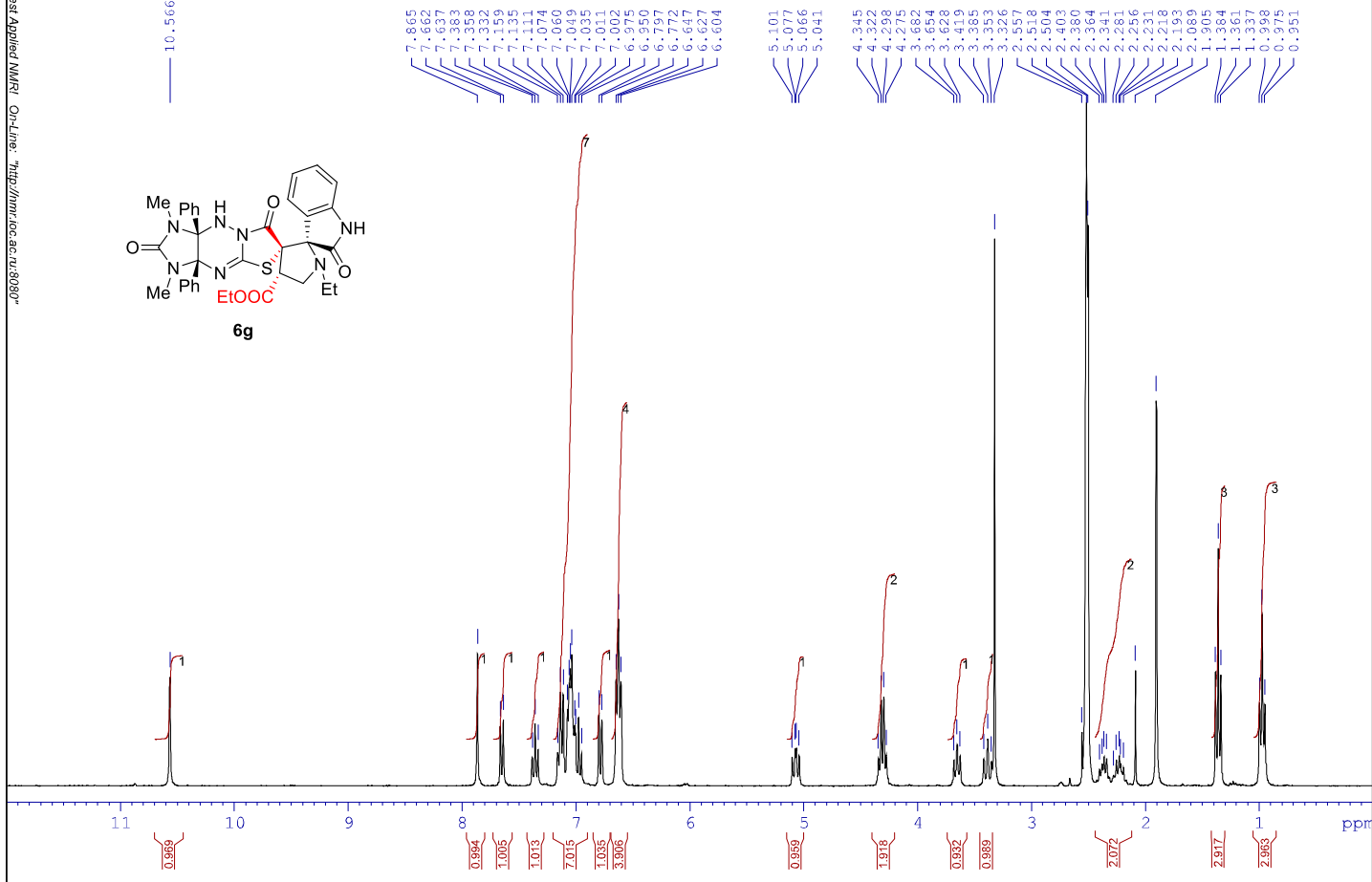
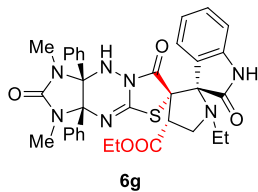
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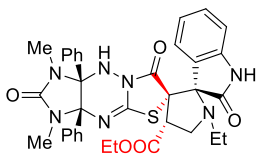
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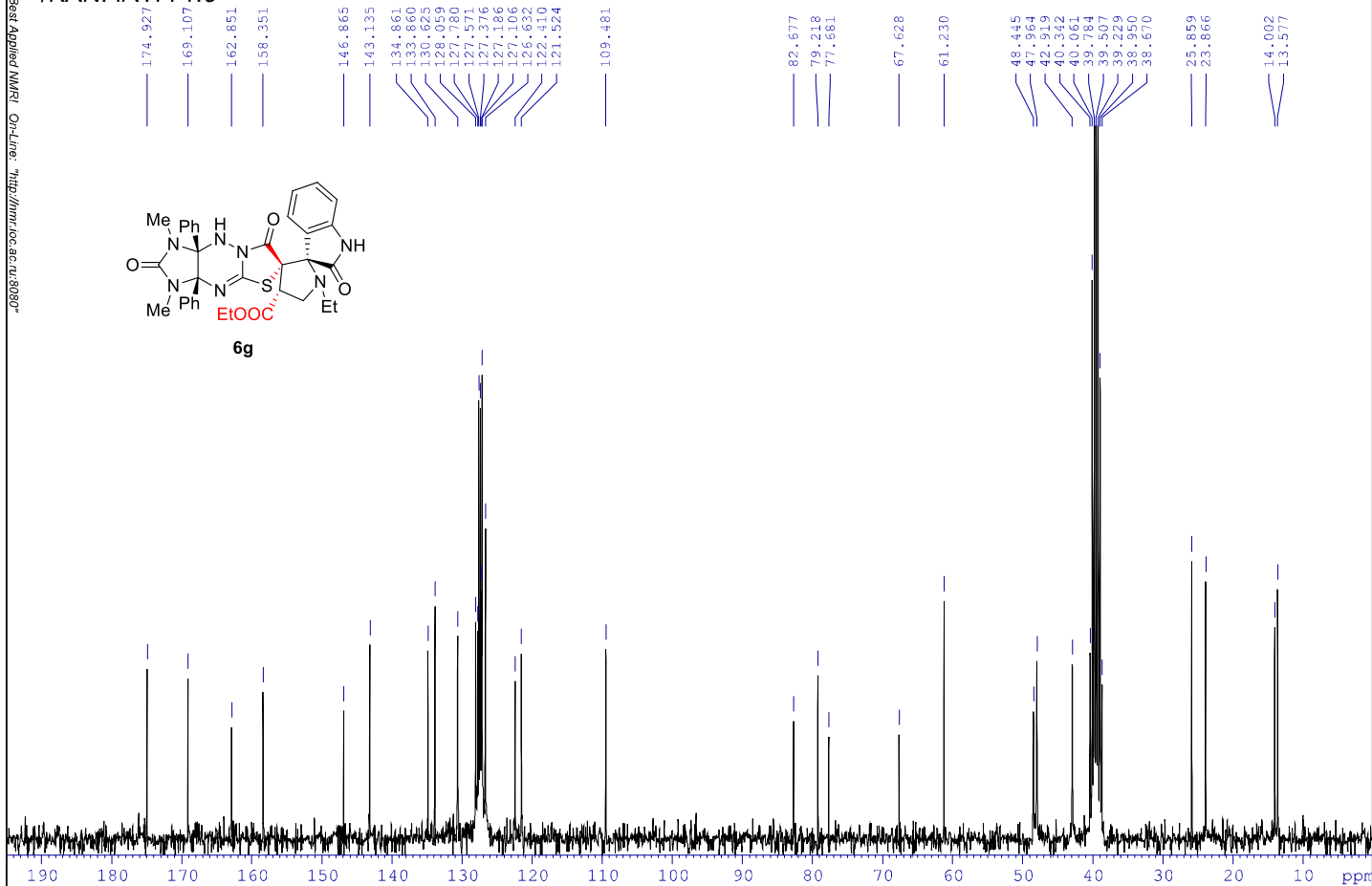
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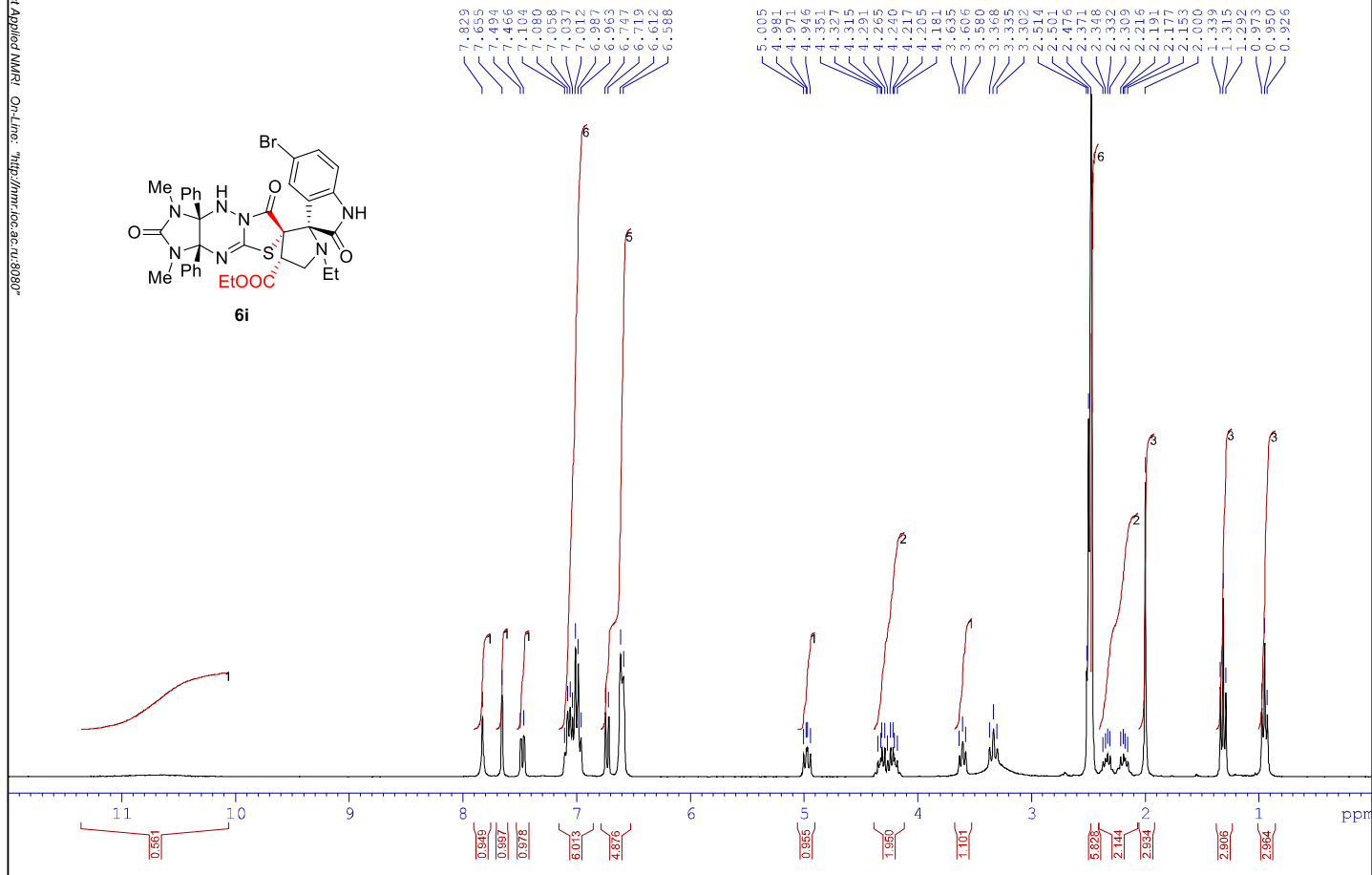
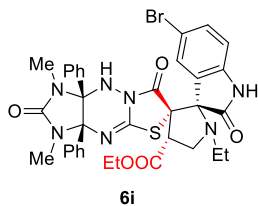
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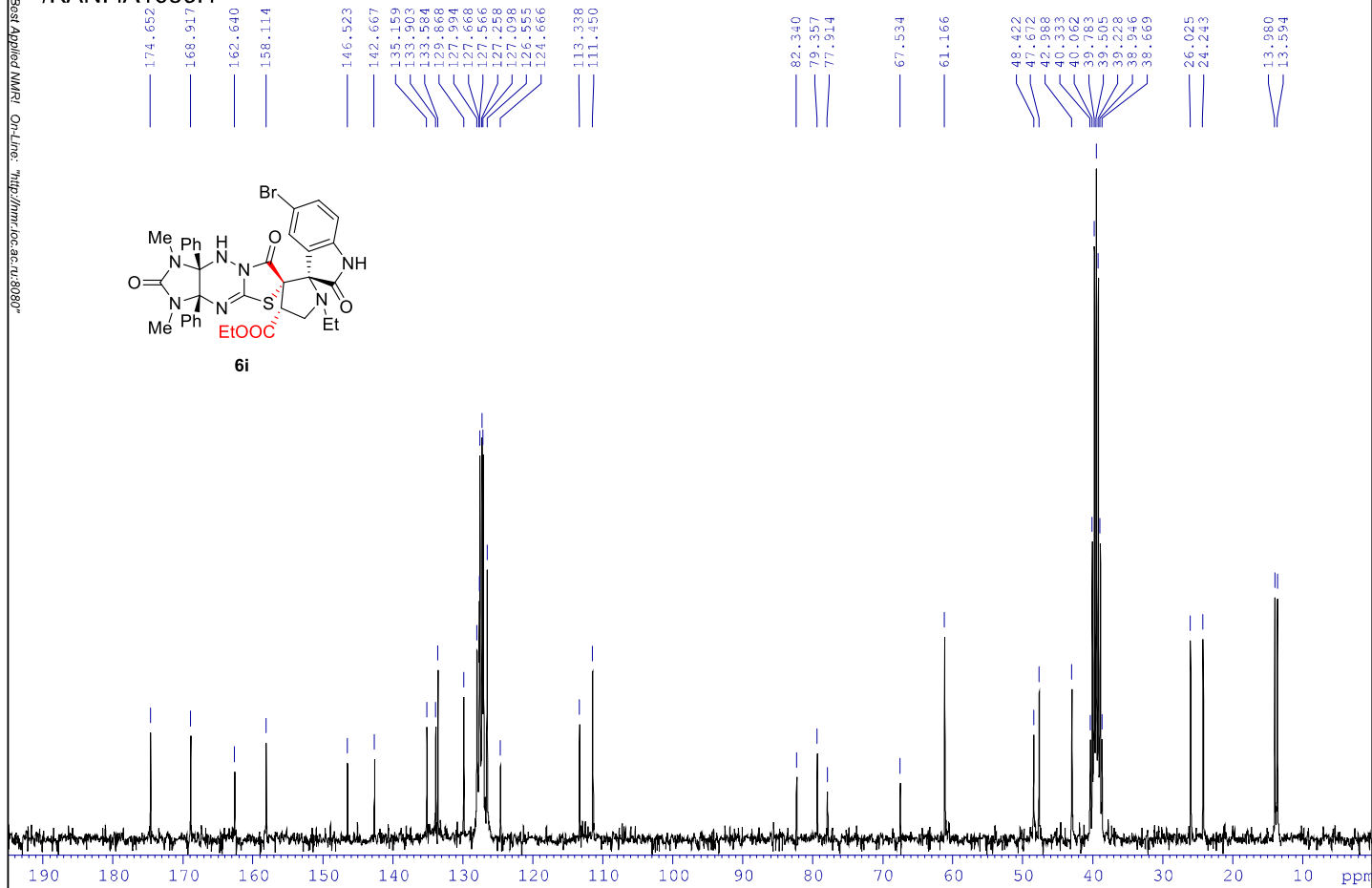
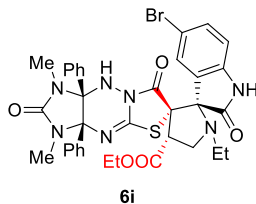
6g



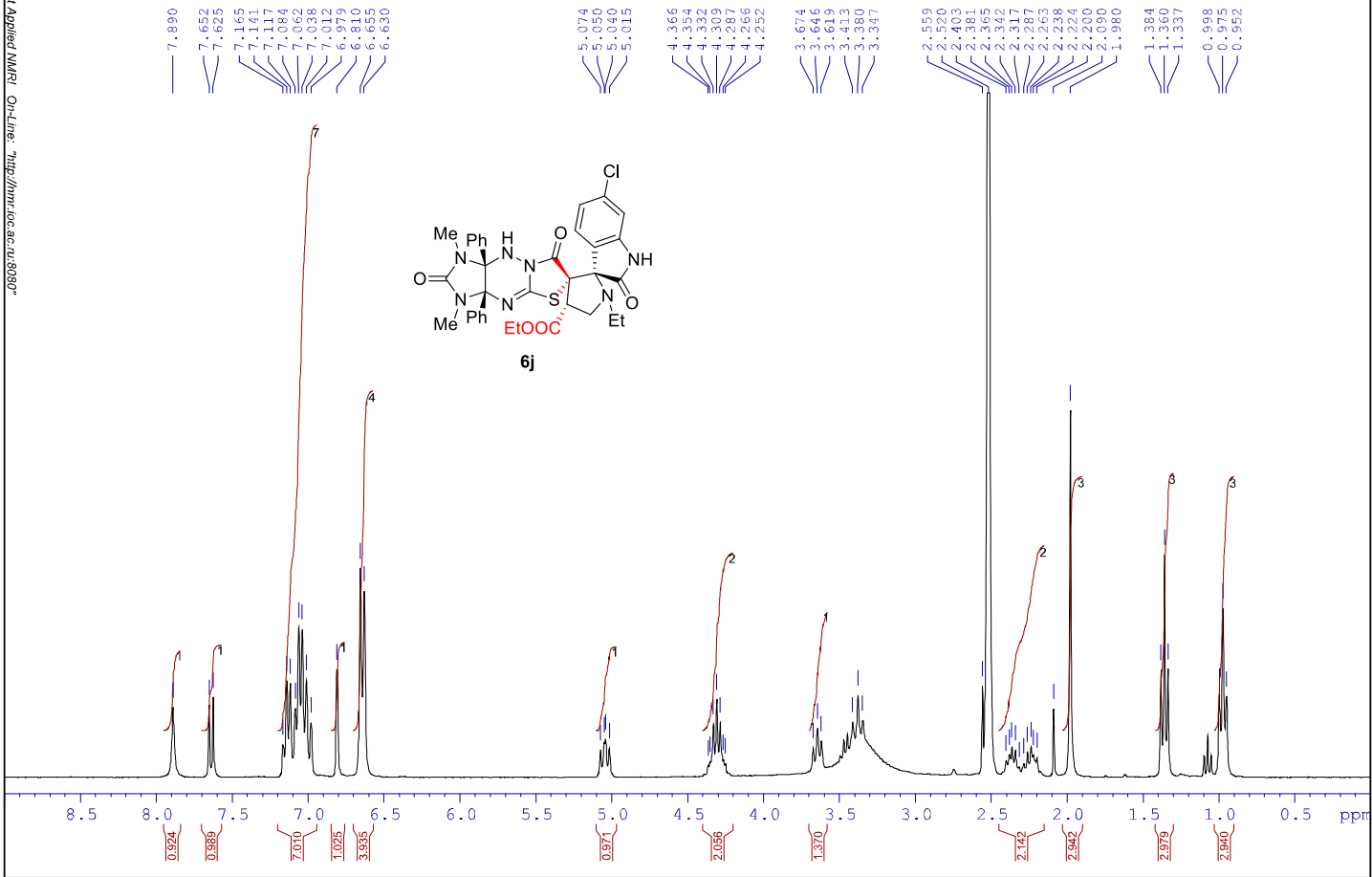
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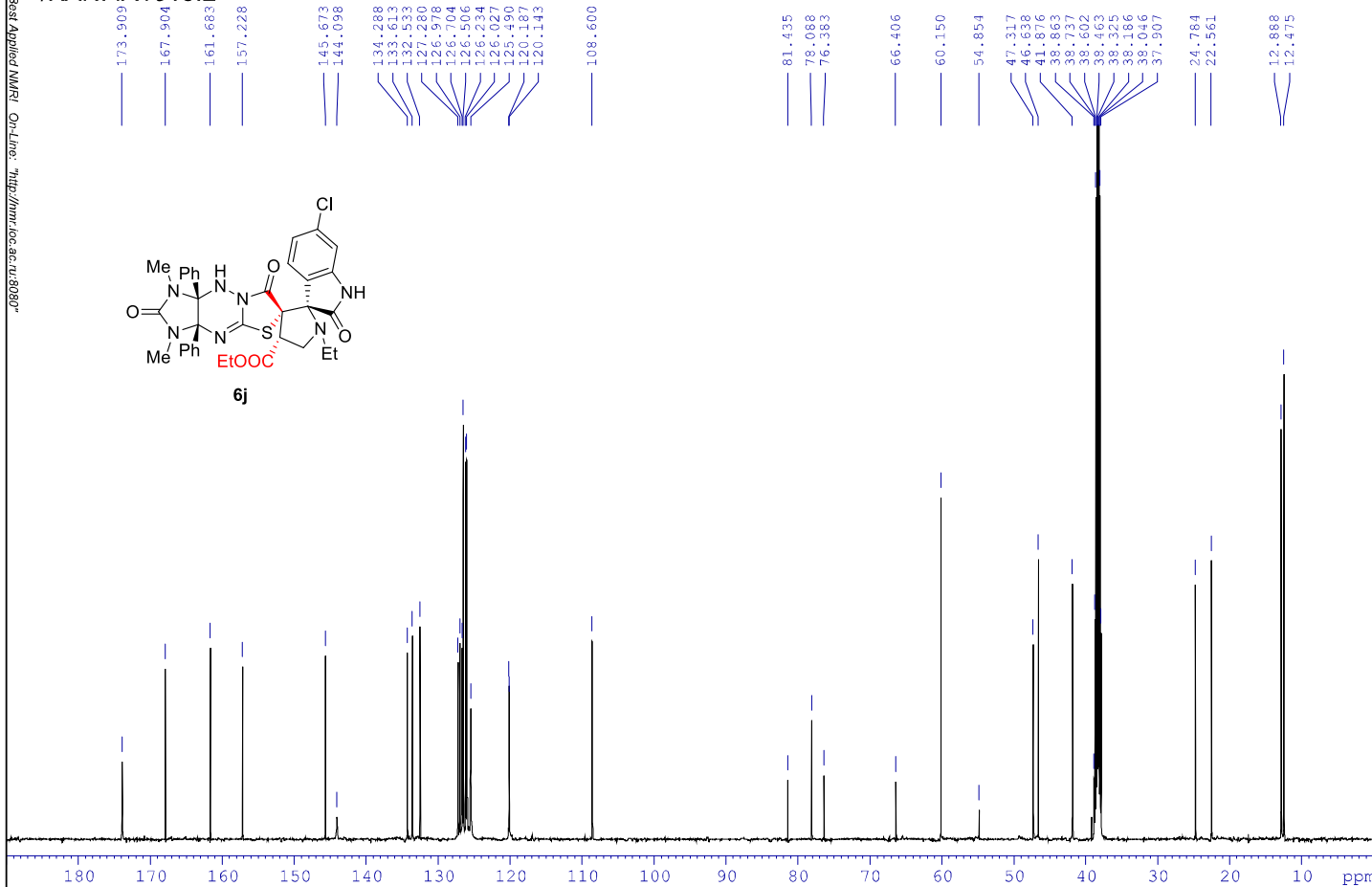
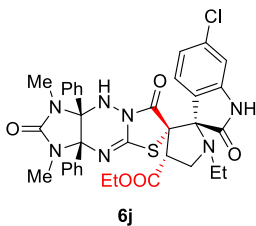
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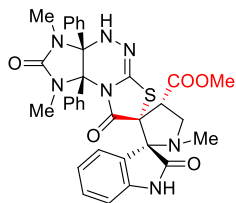
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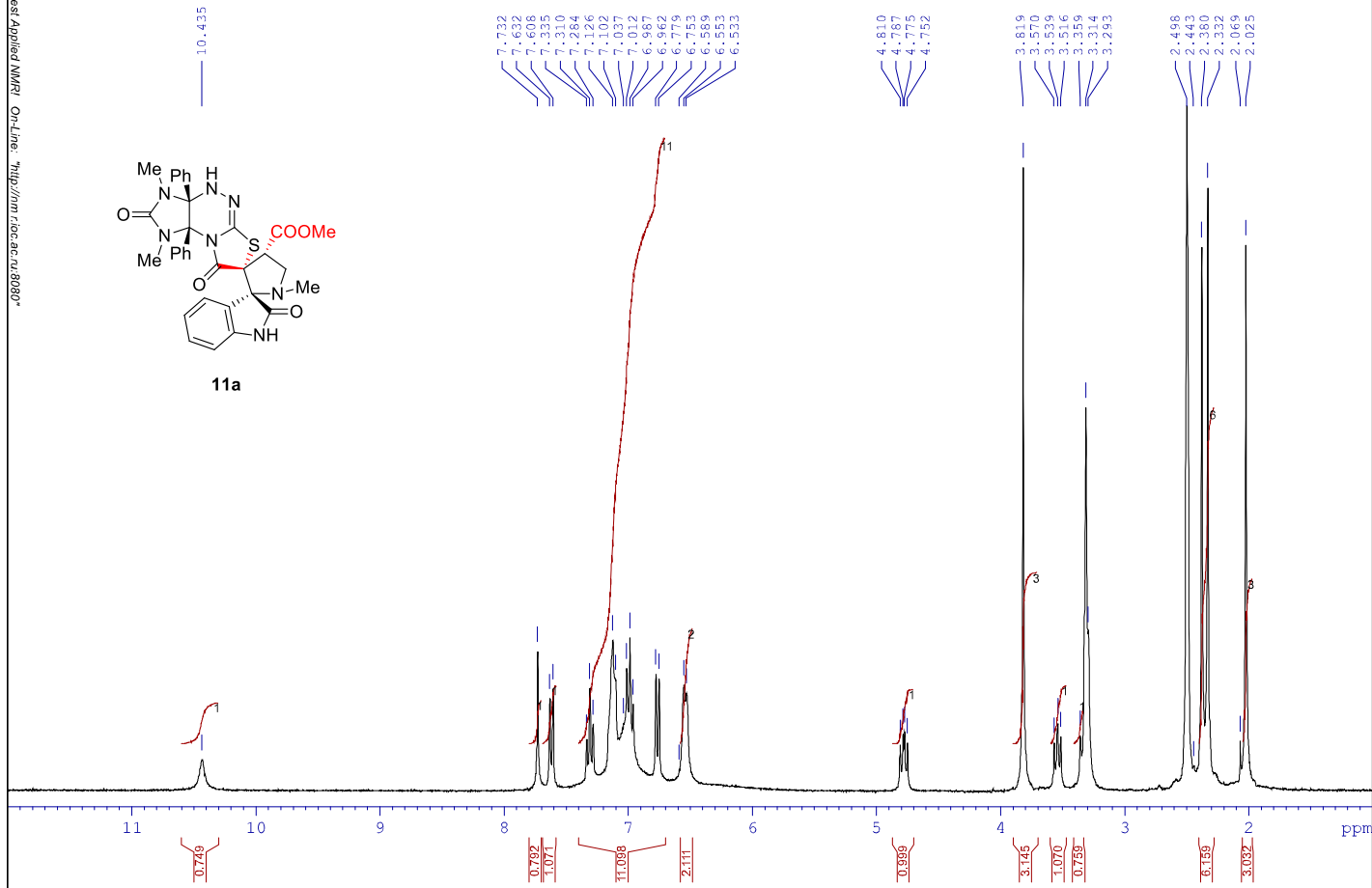
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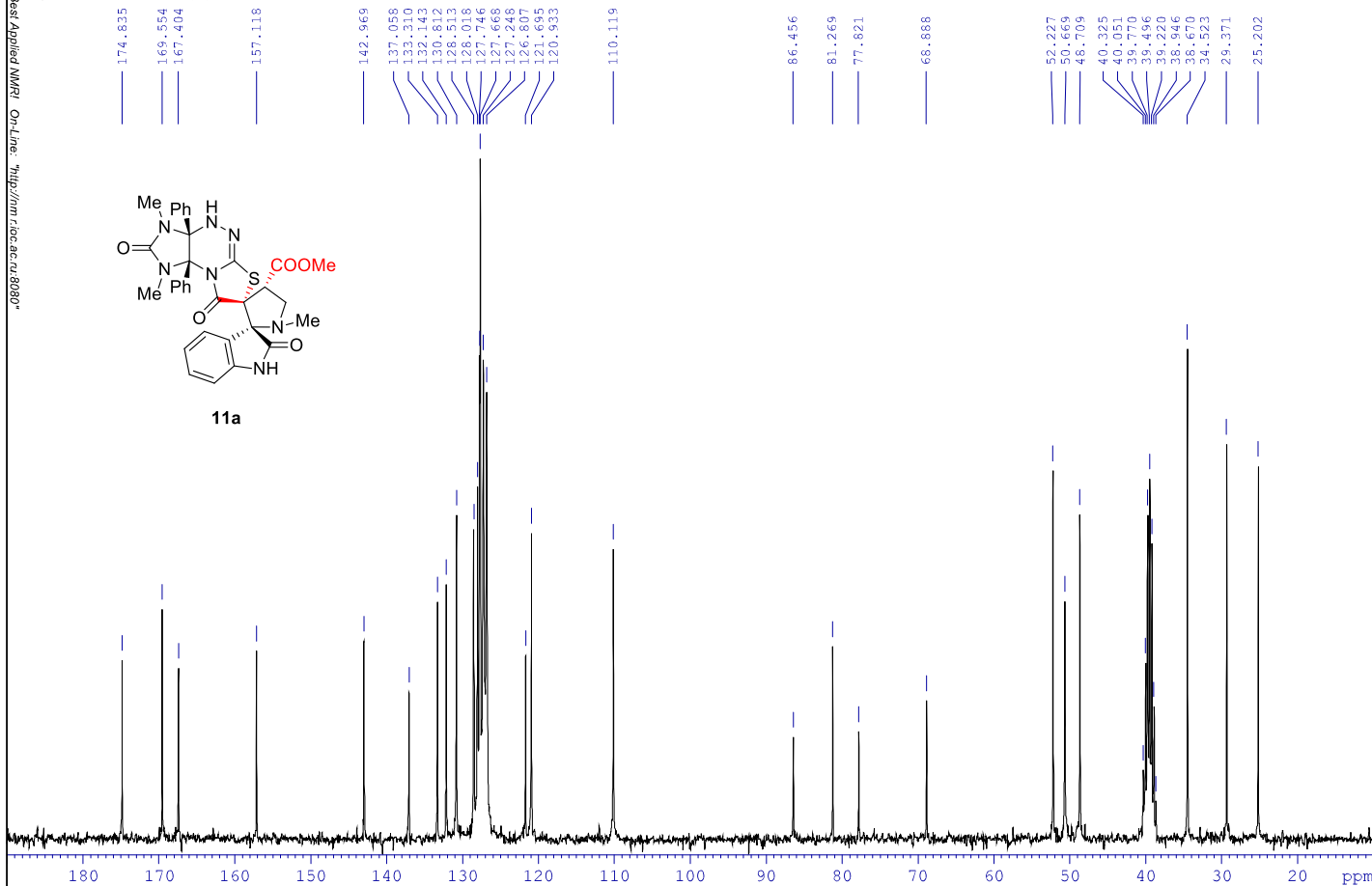
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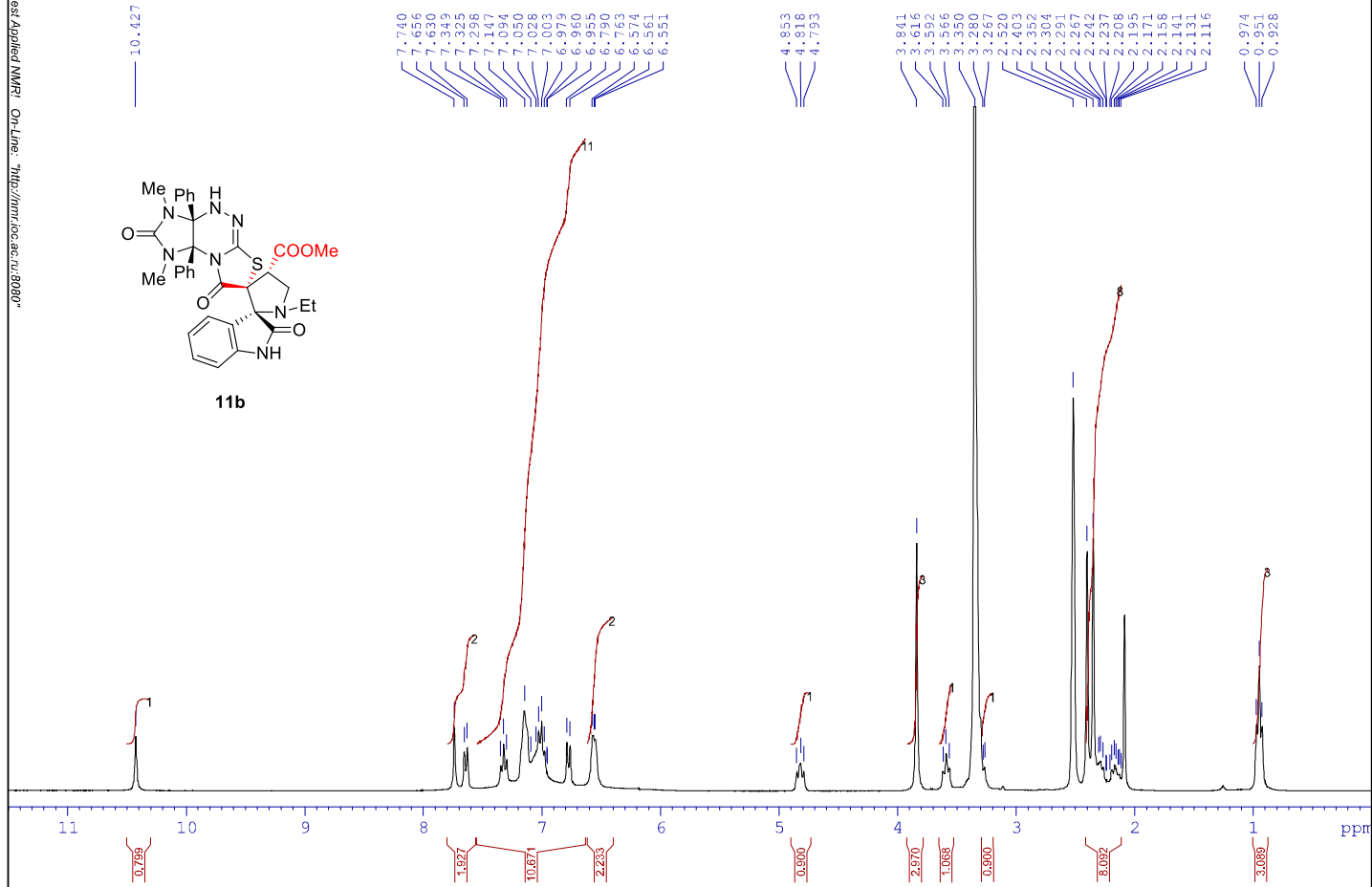
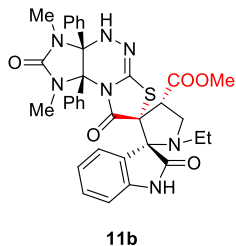
11a



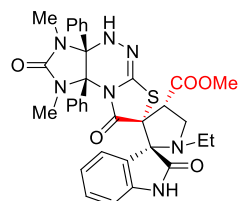
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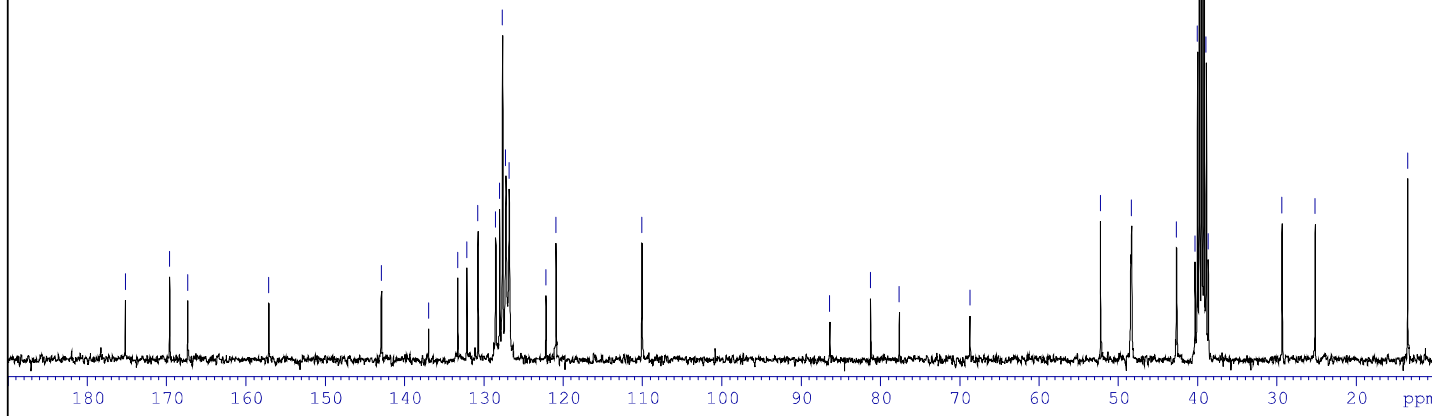
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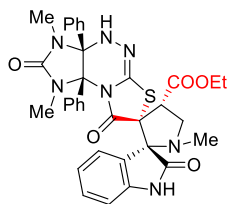
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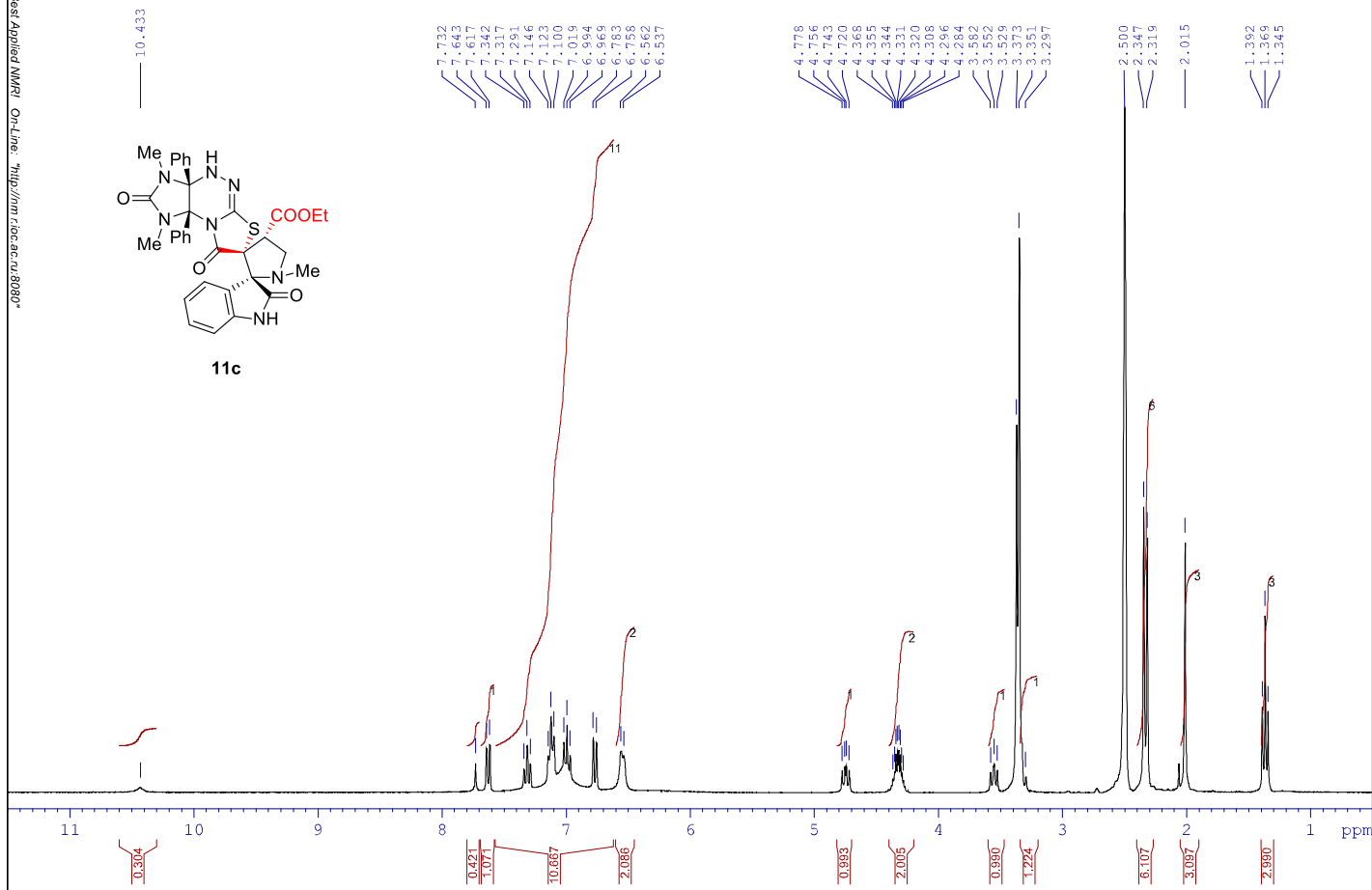
11b



/KANI VDB75.2

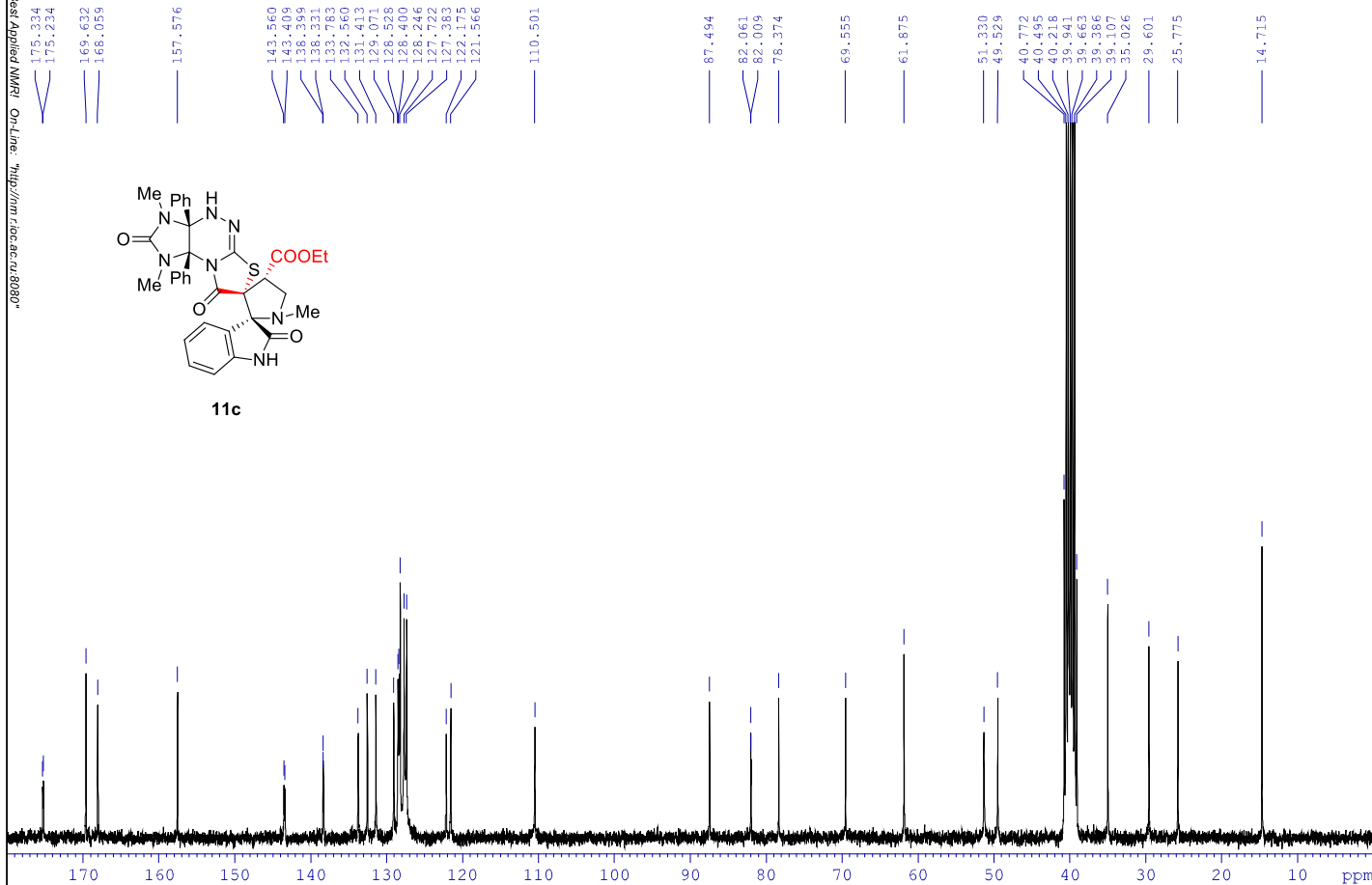


11c

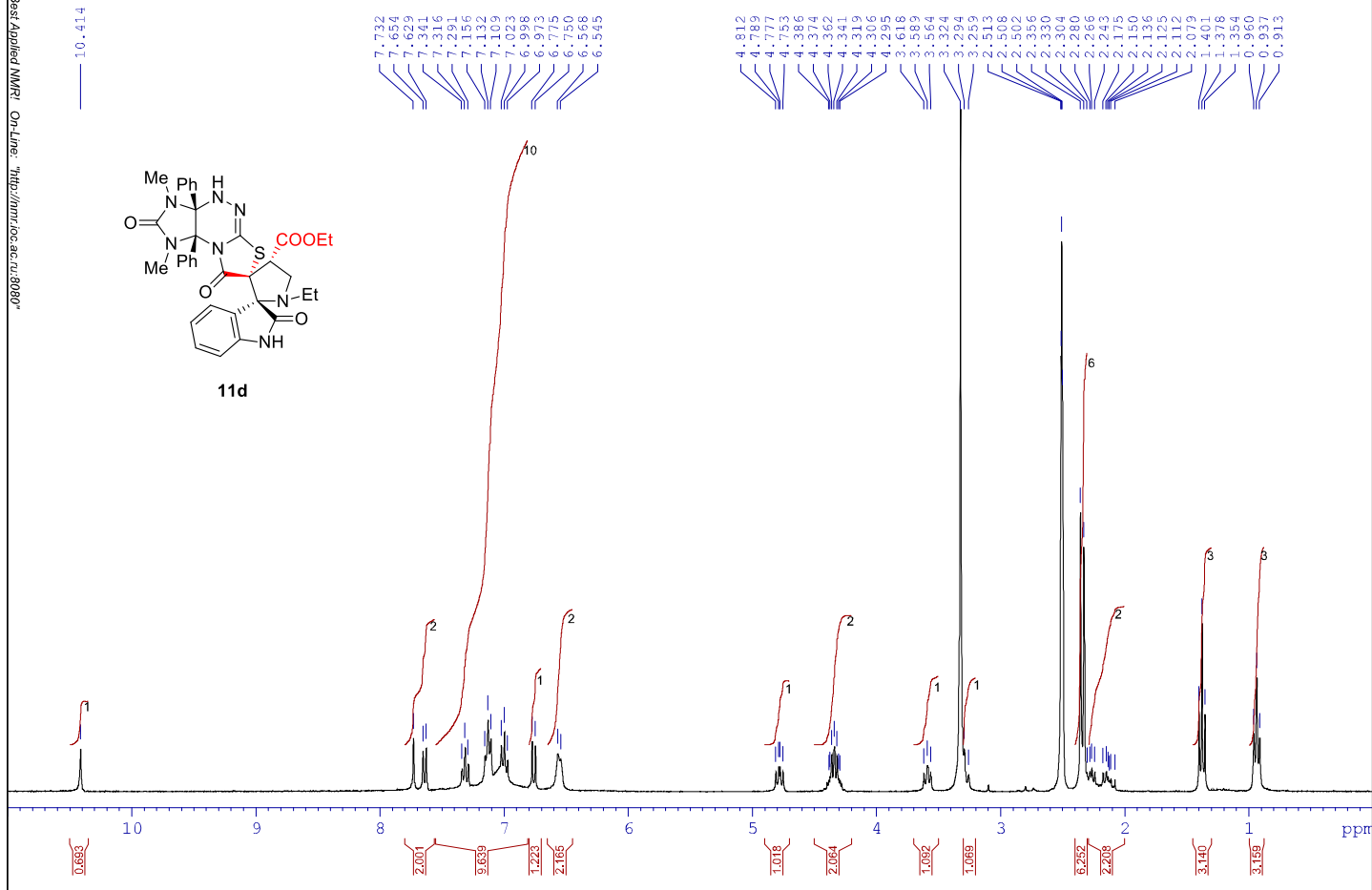


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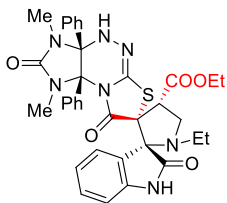
DMSO



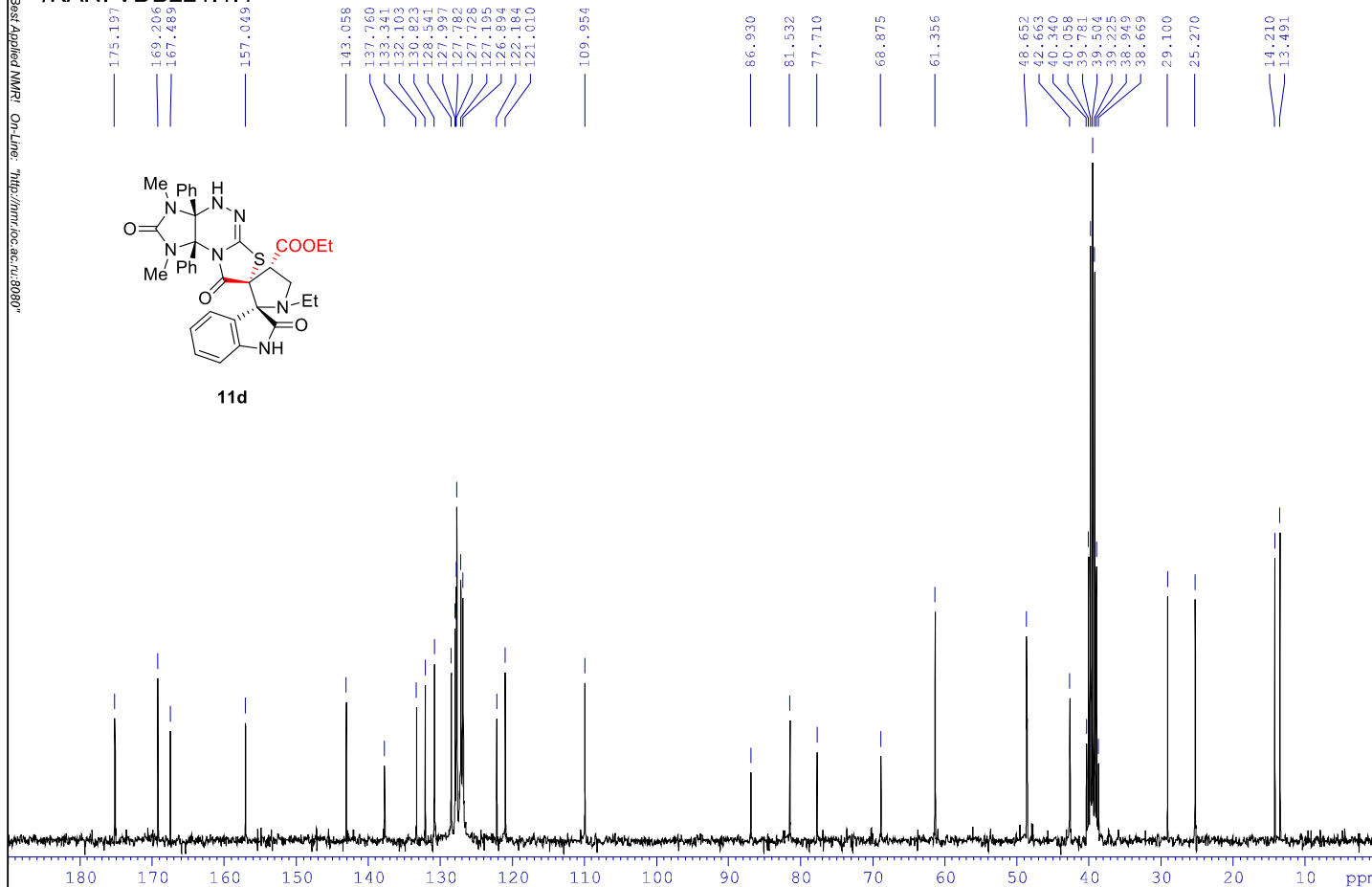
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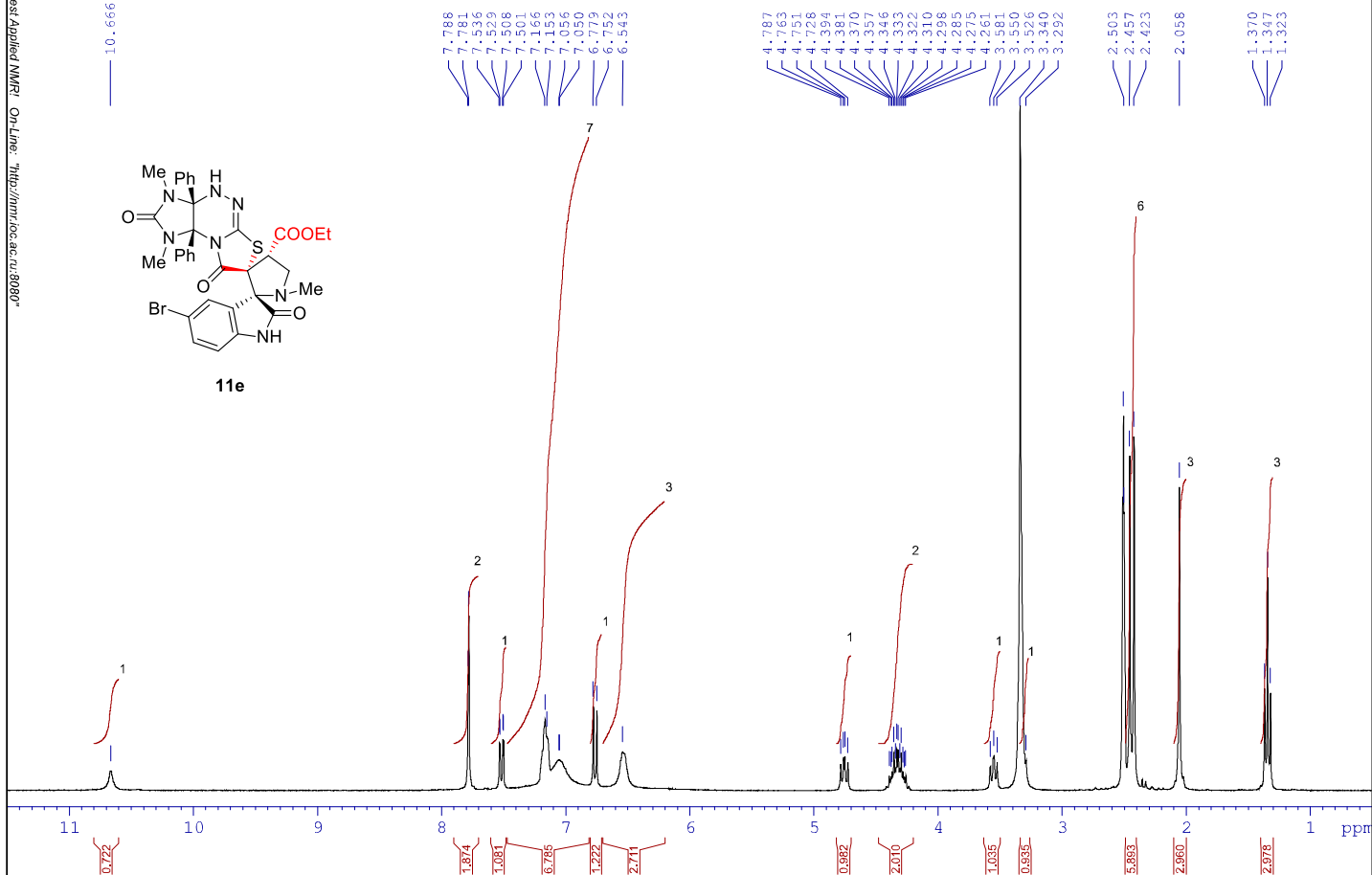
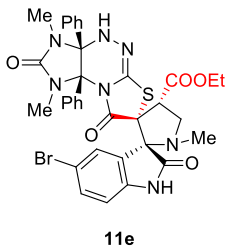
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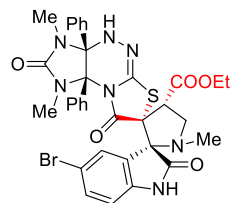
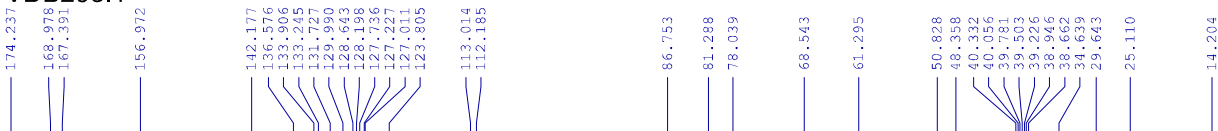
11d



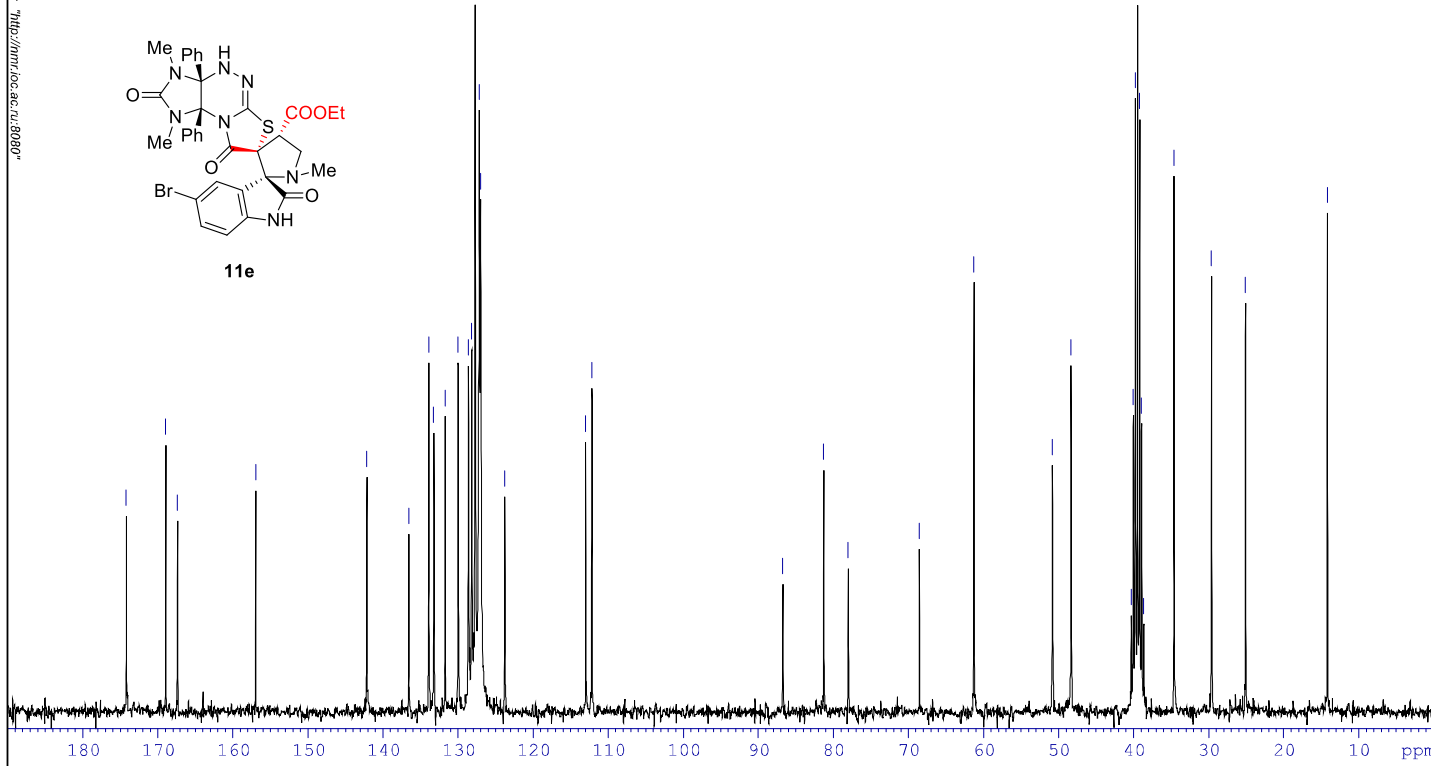
/KANI VDB268.1 Gazieva-00765



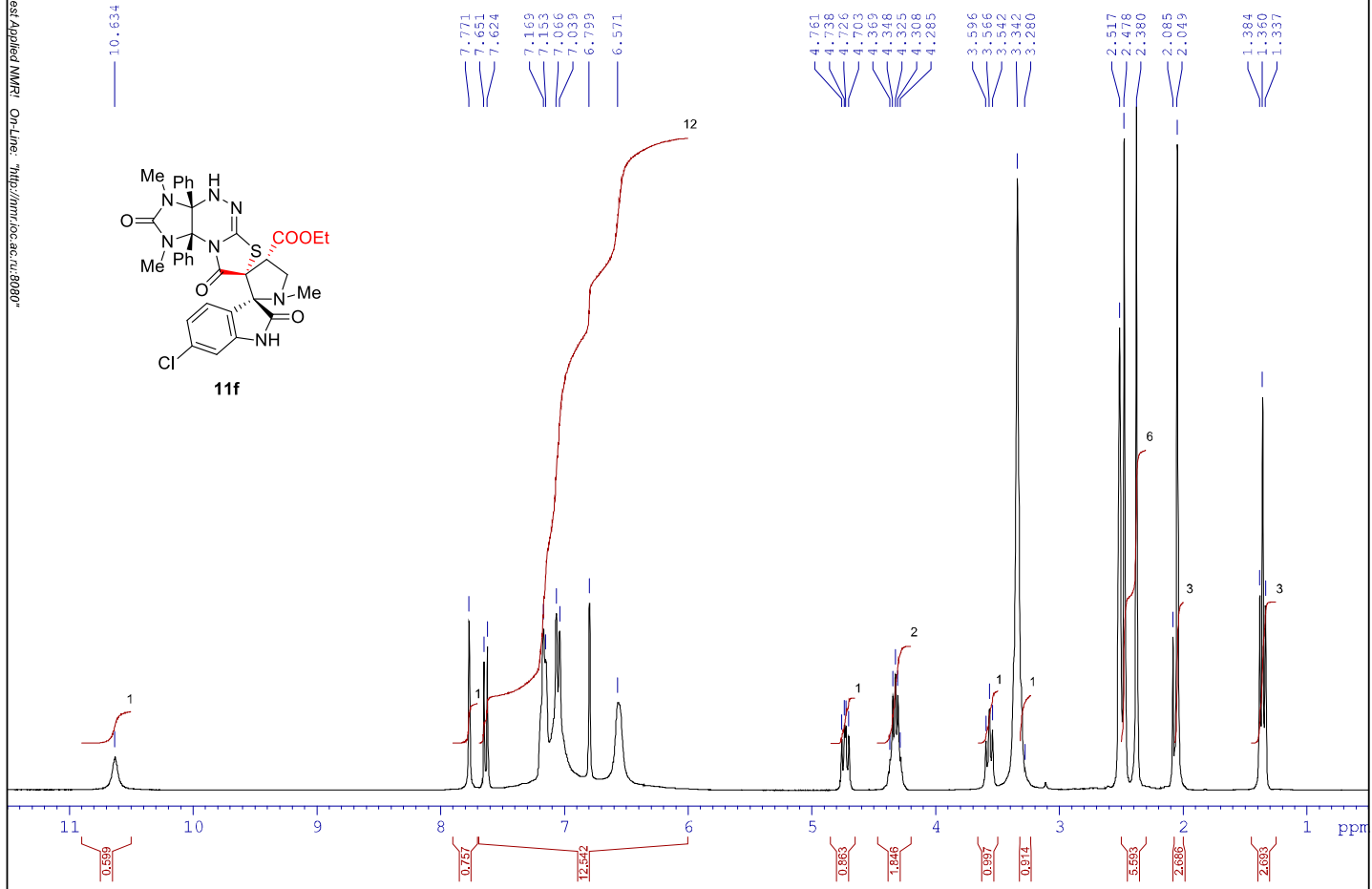
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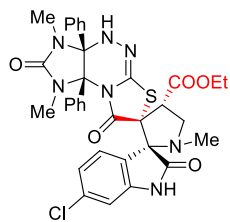
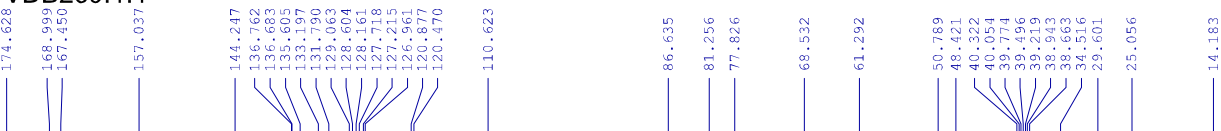
11e



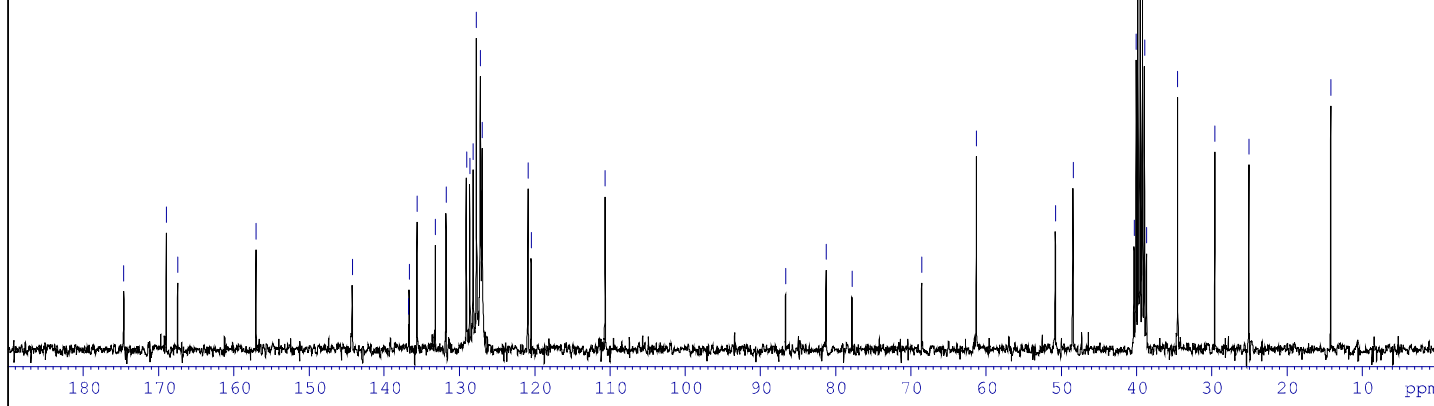
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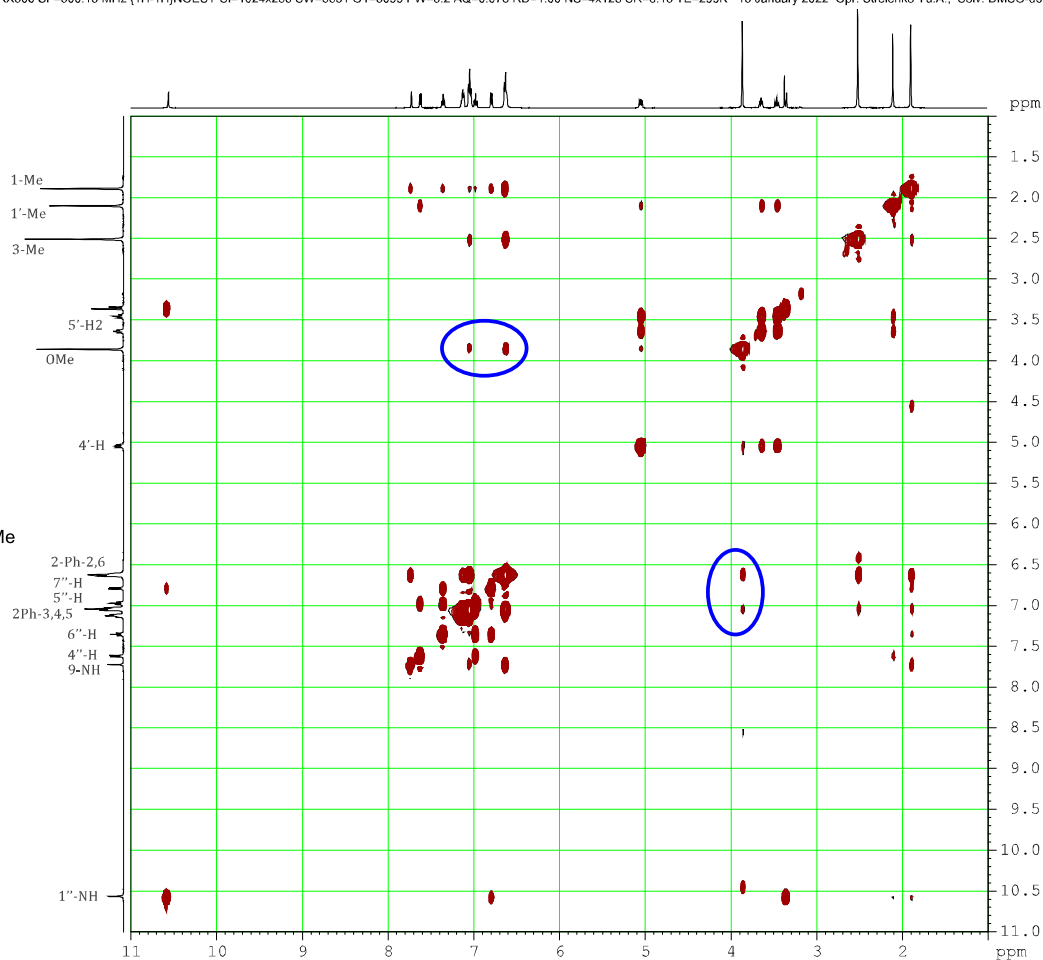
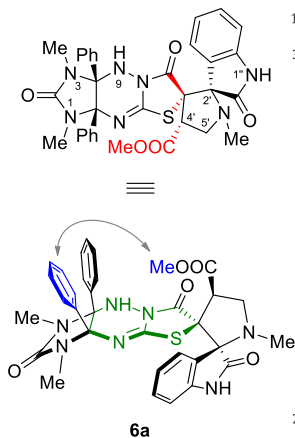
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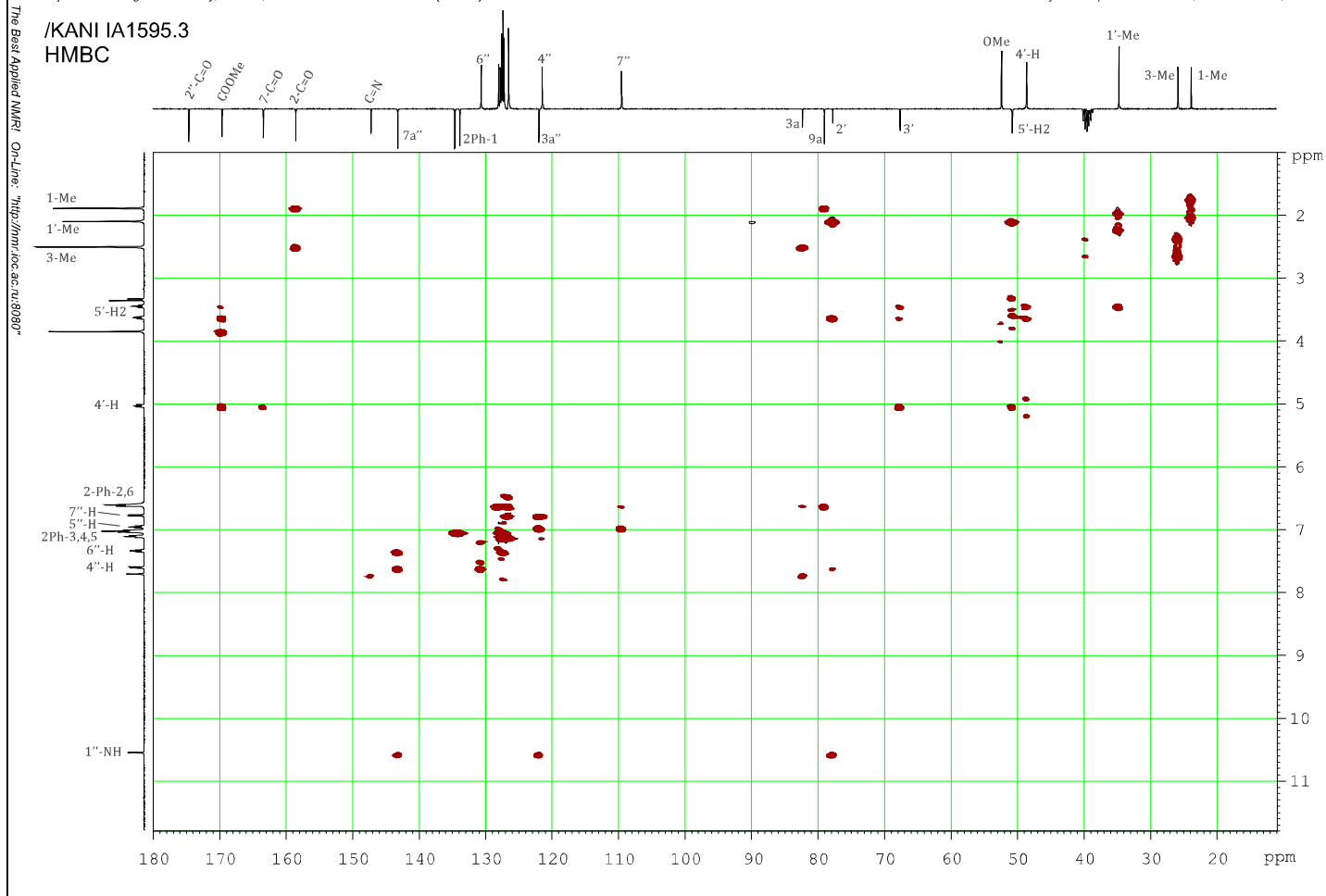
11f



/KANI IA1595.3
NOESY

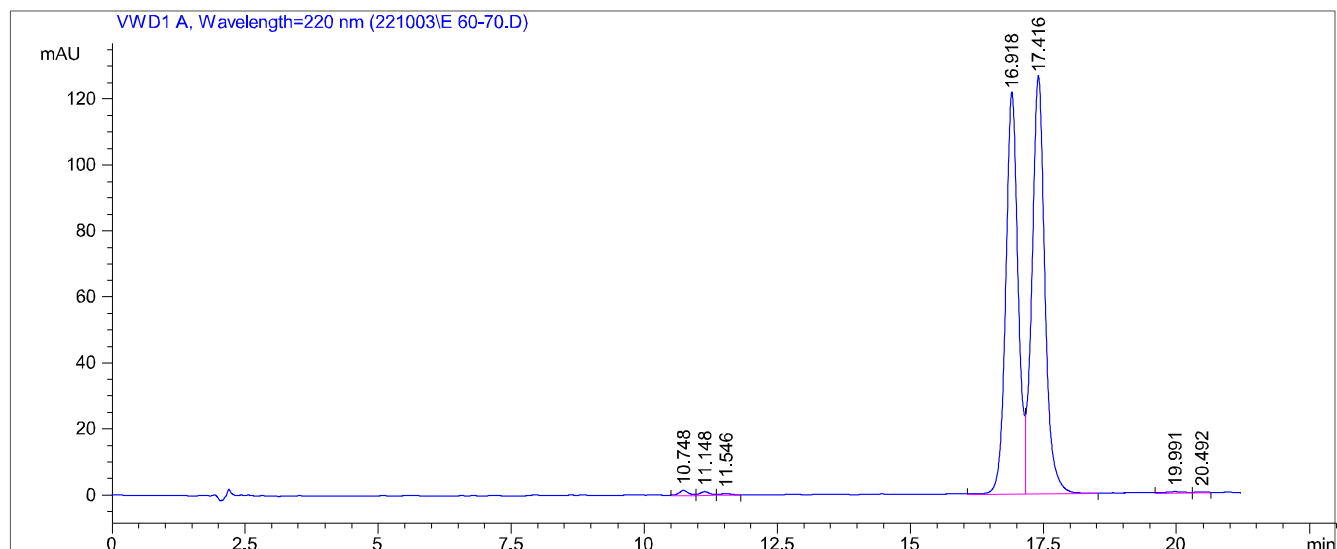


/KANI IA1595.3 HMBP



```
=====
Acq. Operator   :                               Seq. Line :    1
Acq. Instrument : Instrument 1                  Location  : IA-1670
Injection Date  : 10/3/2022 5:08:13 PM          Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : D:\METHODS\CHROM 60 70.M
Last changed    : 10/3/2022 5:07:17 PM
Analysis Method : D:\METHODS\CHROM_ADC_PN.m
Last changed    : 10/4/2022 12:01:46 PM
Sample Info     : Column: Reprosil-Pur Basic C18 250*4.6 mm 5µm
                  A: H2O 0,01% tfa  B: MeCN 0,01% tfa
                  5-100 %B at 20min
=====
```



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

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

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.748	MF	0.1911	16.90440	1.47459	0.3978
2	11.148	MF	0.2049	13.03335	1.06039	0.3067
3	11.546	FM	0.2292	6.50609	4.73162e-1	0.1531
4	16.918	MF	0.2655	1991.02295	124.98219	46.8574
5	17.416	FM	0.2833	2210.45190	130.05444	52.0215
6	19.991	MM	0.3865	8.29231	3.57625e-1	0.1952
7	20.492	MM	0.2156	2.90310	2.24429e-1	0.0683

Totals : 4249.11410 258.62682

=====
*** End of Report ***

Analysis Name: IA-1670
Method: CHROM_ADC_PN.M
Sample Name: Default
Analysis Info: Column: ReprosilPur Basic C18 250*4.6 mm 5um
A: H2O 0,01% tfa B: MeCN 0,01% tfa
5-100 %B at 20min
Instrument: LC-MSD-Trap-SL
Operator: LCMS

Print Date: 9/30/2022 11:49:09 AM
Acq. Date: 9/29/2022 5:35:20 PM

