

## Supplementary material

### **Highly Diastereoselective Synthesis of Polycyclic Amines via-Redox Neutral C-H Functionalization**

#### **New Journal of Chemistry**

Chottanahalli. S. Pavan Kumar,<sup>a#</sup> Kachigere. B. Harsha,<sup>a#</sup> Nagarakere. C. Sandhaya,<sup>a</sup> Ajjalli. B. Ramesha,<sup>a</sup> Kempegowda Mantelingu,<sup>a\*</sup> and Kanchugarakoppal. S. Rangappa,<sup>a\*</sup>

<sup>#</sup>*DOS in Chemistry, University of Mysore, Manasagangotri, Mysuru-06, India*

#### **Contents.**

1. X-ray crystallographic data (experimental and tables) **1-11**
2. The <sup>1</sup>H and <sup>13</sup>C NMR Spectra of **3 (a-p)** and ORTEP of **3c** and **3p** **12-46**
3. The <sup>1</sup>H and <sup>13</sup>C NMR Spectra of **4 (a-l)** and ORTEP of **4c** **47-70**

Date: 01-07-2015

## Crystallographic Data

### Experimental

Single crystals of suitable dimensions were chosen carefully for X-ray diffraction studies. The X-intensity data were collected at a temperature of 293(2) K on a Bruker Proteum2 CCD diffractometer equipped with an X-ray generator operating at 45 kV and 10 mA, using CuK $\alpha$  radiation of wavelength 1.54178 Å. Data were collected for 24 frames per set with different settings of  $\varphi$  (0° and 90°), keeping the scan width of 0.5°, exposure time of 2 s, the sample to detector distance of 45.10 mm and  $2\theta$  value at 46.6°. The complete data sets were processed using *SAINT PLUS*.<sup>1</sup> The structures were solved by direct methods and refined by full-matrix least squares method on  $F^2$  using *SHELXS* and *SHELXL* programs<sup>2</sup>. The geometrical calculations were carried out using the program *PLATON*.<sup>3</sup> The molecular and packing diagrams were generated using the software *MERCURY*.<sup>4</sup> The details of the crystal structure and data refinement are given in Table 1. The list of bond lengths and bond angles of the non-hydrogen atoms are given in Table 4 and Table 5 respectively. Figures **3c**, **3p** and **4c** represent the ORTEP of the molecule with thermal ellipsoids drawn at 50% probability.

### References

- (1). Bruker, (2012). *SAINT PLUS*, Bruker AXS Inc., Madison, Wisconsin, USA.
- (2). G. M. Sheldrick, *Acta. Cryst.*, 2008, *A64*, 112.
- (3). A. L. Spek, *Acta. Cryst.*, *A46*, 1990, C34.
- (4). C. F. Macrae, I. J. Bruno, J. A. Chisholm, P. R. Edgington, P. McCabe, E. Pidcock, L. Rodriguez-Monge, R. Taylor, J. van de Streek, and P.A. Wood, *J. Appl. Cryst.*, 2008, **41**, 466.

Table 1: Crystal data and structure refinement details **3c**

Empirical formula	$C_{30}H_{28}Br_2NO_5$		
Formula weight	642.35		
Temperature	296(2) K		
Wavelength	1.54178 Å		
$\theta$ range for above	3.50° to 64.52°		
Crystal system	Triclinic		
Space group	P $\bar{1}$		
Cell dimensions			
a = 9.9779(11) Å	b = 11.5105(12) Å	c = 13.4027(14) Å	
$\alpha = 91.472(6)^\circ$	$\beta = 107.929(6)^\circ$	$\gamma = 109.296(6)^\circ$	
Volume	1368.3(3) Å <sup>3</sup>		
Z	2		
Density(calculated)	1.559 Mg m <sup>-3</sup>		
Absorption coefficient	4.094 mm <sup>-1</sup>		
F <sub>000</sub>	650		
Crystal size	0.25 × 0.25 × 0.25 mm		
$\theta$ range for data collection	3.50° to 64.52°		
Index ranges	-11 ≤ h ≤ 11		
	-13 ≤ k ≤ 13		
	-15 ≤ l ≤ 14		
Reflections collected	13269		
Independent reflections	4454 [R <sub>int</sub> = 0.0522]		
Refinement method	Full matrix least-squares on F <sup>2</sup>		
Data / restraints / parameters	4454 / 0 / 346		
Goodness-of-fit on F <sup>2</sup>	1.041		
Final [I > 2σ(I)]	R1 = 0.0609, wR2 = 0.1680		
R indices (all data)	R1 = 0.0758, wR2 = 0.1850		
Largest diff. peak and hole	0.591 and -0.556 e Å <sup>-3</sup>		

Table 2: Atomic coordinates and equivalent thermal parameters of the non-hydrogen atoms.

Atom	x	y	z	U <sub>eq</sub>
Br1	0.81469(9)	0.24281(7)	0.25466(4)	0.0768(3)
C2	0.9232(6)	0.2883(5)	0.4022(4)	0.0523(12)
C3	1.0708(7)	0.2946(5)	0.4393(4)	0.0634(14)
C4	1.1500(6)	0.3276(6)	0.5467(4)	0.0648(15)
C5	1.0816(6)	0.3531(5)	0.6163(4)	0.0530(12)
O6	1.1715(4)	0.3848(4)	0.7209(3)	0.0685(11)
C7	1.1134(5)	0.4155(5)	0.7999(4)	0.0550(12)
C8	0.9463(5)	0.3471(4)	0.7668(3)	0.0411(10)
C9	0.8620(5)	0.3793(4)	0.8337(3)	0.0395(9)
C10	0.7043(5)	0.3737(4)	0.7478(3)	0.0363(9)
C11	0.5649(5)	0.2959(4)	0.7720(3)	0.0384(9)
C12	0.5438(5)	0.3310(4)	0.8646(3)	0.0436(10)
C13	0.4155(6)	0.2724(5)	0.8871(4)	0.0493(11)
C14	0.2980(6)	0.1749(5)	0.8144(4)	0.0523(12)
C15	0.3190(6)	0.1352(5)	0.7238(4)	0.0528(12)
C16	0.4533(5)	0.1946(4)	0.7024(3)	0.0432(10)
C17	0.4746(5)	0.1495(4)	0.6037(4)	0.0485(11)
C18	0.6410(5)	0.1900(4)	0.6187(3)	0.0415(10)
N19	0.7070(4)	0.3276(3)	0.6431(2)	0.0347(7)
C20	0.8702(5)	0.3800(4)	0.6594(3)	0.0373(9)
C21	0.9318(5)	0.3465(4)	0.5785(3)	0.0405(10)
C22	0.8540(5)	0.3154(4)	0.4701(3)	0.0454(10)
C23	0.6951(5)	0.5039(4)	0.7387(3)	0.0342(9)
C24	0.7783(5)	0.6081(4)	0.8140(3)	0.0444(10)
C25	0.7552(6)	0.7196(4)	0.8024(4)	0.0519(12)
C26	0.6462(5)	0.7287(4)	0.7135(3)	0.0433(10)
C27	0.5602(6)	0.6269(4)	0.6373(3)	0.0491(11)
C28	0.5855(6)	0.5161(4)	0.6496(3)	0.0455(10)
Br29	0.60863(7)	0.87909(5)	0.69793(4)	0.0650(3)
C30	0.8472(5)	0.3015(4)	0.9213(3)	0.0441(10)
O31	0.8673(4)	0.3421(3)	1.0093(2)	0.0598(9)
O32	0.8152(5)	0.1812(3)	0.8903(3)	0.0694(11)
C33	0.8062(10)	0.0976(7)	0.9713(5)	0.090(2)
C34	0.6577(12)	0.0543(8)	0.9739(6)	0.112(3)
O35	0.1704(4)	0.1259(4)	0.8383(3)	0.0742(12)
C36	0.0430(7)	0.0430(6)	0.7606(6)	0.086(2)
O37	0.3898(4)	0.3019(4)	0.9775(3)	0.0642(10)
C38	0.5202(7)	0.3633(7)	1.0677(4)	0.0798(19)

Table 3: Anisotropic thermal parameters of the non-hydrogen atoms.

Atom	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
Br1	0.1042(6)	0.0965(5)	0.0485(4)	0.0537(4)	0.0316(4)	0.0129(3)
C2	0.071(4)	0.054(3)	0.052(3)	0.034(3)	0.035(3)	0.020(2)
C3	0.075(4)	0.073(3)	0.068(3)	0.038(3)	0.045(3)	0.022(3)
C4	0.045(3)	0.093(4)	0.074(3)	0.037(3)	0.031(3)	0.022(3)
C5	0.047(3)	0.061(3)	0.059(3)	0.024(2)	0.021(2)	0.020(2)
O6	0.039(2)	0.111(3)	0.060(2)	0.034(2)	0.0135(17)	0.014(2)
C7	0.035(3)	0.072(3)	0.051(3)	0.018(2)	0.008(2)	0.002(2)
C8	0.042(3)	0.041(2)	0.041(2)	0.0184(19)	0.011(2)	0.0079(17)
C9	0.039(2)	0.037(2)	0.039(2)	0.0138(18)	0.0100(19)	0.0051(17)
C10	0.038(2)	0.038(2)	0.0328(19)	0.0143(18)	0.0104(18)	0.0040(16)
C11	0.038(2)	0.033(2)	0.044(2)	0.0133(18)	0.0138(19)	0.0105(17)
C12	0.043(3)	0.043(2)	0.048(2)	0.016(2)	0.017(2)	0.0091(18)
C13	0.049(3)	0.058(3)	0.052(3)	0.023(2)	0.025(2)	0.017(2)
C14	0.045(3)	0.048(3)	0.071(3)	0.013(2)	0.031(3)	0.016(2)
C15	0.038(3)	0.045(2)	0.066(3)	0.004(2)	0.017(2)	0.004(2)
C16	0.040(3)	0.038(2)	0.053(2)	0.0134(19)	0.018(2)	0.0077(18)
C17	0.047(3)	0.041(2)	0.050(2)	0.008(2)	0.015(2)	-0.0030(19)
C18	0.043(3)	0.035(2)	0.046(2)	0.0115(18)	0.016(2)	-0.0005(17)
N19	0.0347(19)	0.0312(16)	0.0377(17)	0.0108(14)	0.0124(15)	0.0030(13)
C20	0.032(2)	0.038(2)	0.042(2)	0.0120(17)	0.0118(19)	0.0073(16)
C21	0.036(2)	0.043(2)	0.049(2)	0.0183(19)	0.018(2)	0.0132(18)
C22	0.046(3)	0.051(2)	0.047(2)	0.024(2)	0.019(2)	0.0135(19)
C23	0.035(2)	0.034(2)	0.0357(19)	0.0113(17)	0.0150(18)	0.0051(15)
C24	0.043(3)	0.040(2)	0.043(2)	0.0184(19)	0.001(2)	0.0016(18)
C25	0.048(3)	0.038(2)	0.056(3)	0.010(2)	0.005(2)	-0.0086(19)
C26	0.046(3)	0.040(2)	0.050(2)	0.019(2)	0.020(2)	0.0073(18)
C27	0.051(3)	0.049(3)	0.044(2)	0.024(2)	0.005(2)	0.0047(19)
C28	0.047(3)	0.044(2)	0.042(2)	0.019(2)	0.007(2)	-0.0032(18)
Br29	0.0777(5)	0.0432(3)	0.0732(4)	0.0313(3)	0.0134(3)	0.0066(2)
C30	0.048(3)	0.049(2)	0.037(2)	0.020(2)	0.013(2)	0.0077(18)
O31	0.071(2)	0.064(2)	0.0369(17)	0.0229(19)	0.0096(16)	0.0032(15)
O32	0.121(3)	0.0490(19)	0.0499(18)	0.035(2)	0.039(2)	0.0185(15)
C33	0.138(7)	0.071(4)	0.075(4)	0.034(4)	0.056(4)	0.030(3)
C34	0.142(8)	0.100(6)	0.092(5)	0.034(5)	0.043(5)	0.039(5)
O35	0.050(2)	0.077(3)	0.094(3)	0.006(2)	0.041(2)	0.012(2)
C36	0.058(4)	0.062(4)	0.142(6)	0.009(3)	0.051(4)	0.006(4)
O37	0.054(2)	0.089(3)	0.058(2)	0.0251(19)	0.0317(18)	0.0117(18)
C38	0.077(4)	0.120(6)	0.052(3)	0.038(4)	0.031(3)	0.012(3)

Table 4: Bond lengths ( $\text{\AA}$ ).

Atoms	Length	Atoms	Length
Br1-C2	1.900(5)	C14-C15	1.385(7)
C2-C3	1.377(8)	C15-C16	1.409(7)
C2-C22	1.384(6)	C16-C17	1.508(6)
C3-C4	1.384(8)	C17-C18	1.514(6)
C4-C5	1.389(7)	C18-N19	1.483(5)
C5-O6	1.373(6)	N19-C20	1.480(5)
C5-C21	1.398(7)	C20-C21	1.498(6)
O6-C7	1.441(6)	C21-C22	1.392(6)
C7-C8	1.502(7)	C23-C24	1.382(6)
C8-C9	1.517(6)	C23-C28	1.396(6)
C8-C20	1.530(6)	C24-C25	1.381(7)
C9-C30	1.510(6)	C25-C26	1.377(7)
C9-C10	1.618(6)	C26-C27	1.374(6)
C10-N19	1.499(5)	C26-Br29	1.892(4)
C10-C11	1.520(6)	C27-C28	1.384(7)
C10-C23	1.538(6)	C30-O31	1.189(5)
C11-C16	1.385(6)	C30-O32	1.338(6)
C11-C12	1.393(6)	O32-C33	1.473(6)
C12-C13	1.365(6)	C33-C34	1.411(13)
C13-O37	1.369(6)	O35-C36	1.395(7)
C13-C14	1.398(7)	O37-C38	1.426(7)
C14-O35	1.352(6)		

Table 5: Bond angles (°).

Atoms	Angle	Atoms	Angle
C3-C2-C22	121.2(5)	C11-C16-C15	119.5(4)
C3-C2-Br1	118.9(4)	C11-C16-C17	120.1(4)
C22-C2-Br1	119.9(4)	C15-C16-C17	120.4(4)
C2-C3-C4	119.0(5)	C16-C17-C18	109.9(4)
C3-C4-C5	120.7(5)	N19-C18-C17	108.4(4)
O6-C5-C4	115.3(5)	C20-N19-C18	114.0(3)
O6-C5-C21	124.3(4)	C20-N19-C10	101.0(3)
C4-C5-C21	120.3(5)	C18-N19-C10	112.1(3)
C5-O6-C7	120.2(4)	N19-C20-C21	120.9(4)
O6-C7-C8	110.3(4)	N19-C20-C8	104.9(3)
C7-C8-C9	117.9(4)	C21-C20-C8	110.1(4)
C7-C8-C20	109.0(4)	C22-C21-C5	118.6(4)
C9-C8-C20	101.7(3)	C22-C21-C20	125.2(4)
C30-C9-C8	115.0(4)	C5-C21-C20	116.2(4)
C30-C9-C10	115.1(3)	C2-C22-C21	120.3(5)
C8-C9-C10	104.0(3)	C24-C23-C28	116.9(4)
N19-C10-C11	112.8(3)	C24-C23-C10	126.0(4)
N19-C10-C23	106.2(3)	C28-C23-C10	117.0(4)
C11-C10-C23	106.2(3)	C25-C24-C23	122.1(4)
N19-C10-C9	105.4(3)	C26-C25-C24	119.7(4)
C11-C10-C9	114.4(3)	C27-C26-C25	120.0(4)
C23-C10-C9	111.6(3)	C27-C26-Br29	119.5(4)
C16-C11-C12	118.3(4)	C25-C26-Br29	120.4(3)
C16-C11-C10	122.4(4)	C26-C27-C28	119.6(4)
C12-C11-C10	119.2(4)	C27-C28-C23	121.7(4)
C13-C12-C11	122.6(4)	O31-C30-O32	123.3(4)
C12-C13-O37	125.0(5)	O31-C30-C9	124.4(4)
C12-C13-C14	119.6(4)	O32-C30-C9	112.3(3)
O37-C13-C14	115.4(4)	C30-O32-C33	116.8(4)
O35-C14-C15	124.6(5)	C34-C33-O32	109.0(7)
O35-C14-C13	116.7(4)	C14-O35-C36	118.3(5)
C15-C14-C13	118.7(4)	C13-O37-C38	116.2(4)
C14-C15-C16	121.1(5)		

Table 1: Crystal data and structure refinement details **3P**.

Empirical formula	C <sub>35</sub> H <sub>36</sub> N <sub>2</sub> O <sub>6</sub> S <sub>2</sub>	
Formula weight	644.78	
Temperature	296(2) K	
Wavelength	1.54178 Å	
cell determination	4501	Reflns. for
θ range for above system	2.62° to 64.92° Crystal	
P 21/c	Monoclinic Space group	
Cell dimensions		
a = 17.5740(17) Å	b = 8.9640(9) Å	c = 20.843(2) Å
α = 90.00°	β = 106.682(4)°	γ = 90.00°
Volume	3145.3(5) Å <sup>3</sup>	
Z	4	
Density(calculated)	1.362 Mg m <sup>-3</sup>	
Absorption coefficient	1.943 mm <sup>-1</sup>	
F <sub>000</sub>	1360	
Crystal size	0.27 × 0.27 × 0.27 mm	
θ range for data collection	2.62° to 64.92°	
Index ranges	-20 ≤ h ≤ 20	
	-10 ≤ k ≤ 10	
	-24 ≤ l ≤ 20	
Reflections collected	14873	
Independent reflections	5126 [R <sub>int</sub> = 0.0399]	
Absorption correction	multi-scan	
Refinement method	Full matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	5126 / 0 / 410	
Goodness-of-fit on F <sup>2</sup>	1.078	
Final [I > 2σ(I)]	R1 = 0.0545, wR2 = 0.1671	
R indices (all data)	R1 = 0.0596, wR2 = 0.1727	
Largest diff. peak and hole	0.752 and -0.630 e Å <sup>-3</sup>	



Table 2: Bond lengths ( $\text{\AA}$ ).

Atoms	Length	Atoms	Length
S36-O38	1.427(2)	C9-C10	1.532(3)
S36-O37	1.428(3)	C4-C5	1.404(3)
S36-N14	1.670(2)	C4-C3	1.501(3)
S36-C39	1.757(3)	C7-C8	1.378(3)
S26-C25	1.690(3)	C7-C6	1.407(4)
S26-C22	1.704(2)	C21-C20	1.503(3)
O33-C31	1.337(3)	C21-C182	1.504(3)
O33-C34	1.453(3)	C20-C19	1.388(4)
O27-C7	1.374(3)	C20-C15	1.406(4)
O27-C28	1.413(4)	C15-C16	1.404(4)
O29-C6	1.374(3)	C12-C13	1.516(3)
O29-C30	1.421(4)	C3-C2	1.513(3)
O32-C31	1.199(3)	C5-C6	1.373(4)
N1-C2	1.470(3)	C19-C18	1.384(4)
N1-C21	1.481(3)	C16-C17	1.375(5)
N1-C10	1.495(3)	C39-C44	1.389(5)
N14-C15	1.441(4)	C39-C40	1.391(4)
N14-C13	1.488(3)	C18-C17	1.372(5)
C23-C24	1.468(4)	C24-C25	1.343(5)
C23-C22	1.489(4)	C40-C41	1.373(4)
C22-C10	1.520(3)	C41-C42	1.383(5)
C11-C31	1.516(3)	C42-C43	1.390(4)
C11-C12	1.525(3)	C42-C45	1.504(5)
C11-C10	1.631(3)	C44-C43	1.364(5)
C9-C4	1.378(4)	C34-C35	1.476(5)
C9-C8	1.405(3)		

Table 1: Crystal data and structure refinement details **4C**.

Empirical formula	$C_{30}H_{26}BrClN_2O_3$		
Formula weight	577.89		
Temperature	296(2) K		
Wavelength	1.54178 Å		
Reflns. for cell determination	4104		
$\theta$ range for above	6.72° to 64.51°		
Crystal system	Triclinic		
Space group	P $\bar{1}$		
Cell dimensions			
a = 9.5848(7) Å	b = 11.0031(8) Å	c = 14.0353(11) Å	
$\alpha$ = 87.764(2)°	$\beta$ = 70.878(2)°	$\gamma$ = 65.624(2)°	
Volume	1265.88(16) Å <sup>3</sup>		
Z	2		
Density(calculated)	1.516 Mg m <sup>-3</sup>		
Absorption coefficient	3.478 mm <sup>-1</sup>		
F <sub>000</sub>	592		
Crystal size	0.25 × 0.25 × 0.25 mm		
$\theta$ range for data collection	6.72° to 64.51°		
Index ranges	-11 ≤ h ≤ 11		
	-12 ≤ k ≤ 12		
	-16 ≤ l ≤ 16		
Reflections collected	14548		
Independent reflections	4174 [R <sub>int</sub> = 0.0313]		
Absorption correction	multi-scan		
Refinement method	Full matrix least-squares on F <sup>2</sup>		
Data / restraints / parameters	4174 / 0 / 335		
Goodness-of-fit on F <sup>2</sup>	1.103		
Final [I > 2σ(I)]	R1 = 0.0331, wR2 = 0.0959		
R indices (all data)	R1 = 0.0335, wR2 = 0.0965		
Largest diff. peak and hole	0.376 and -0.584 e Å <sup>-3</sup>		

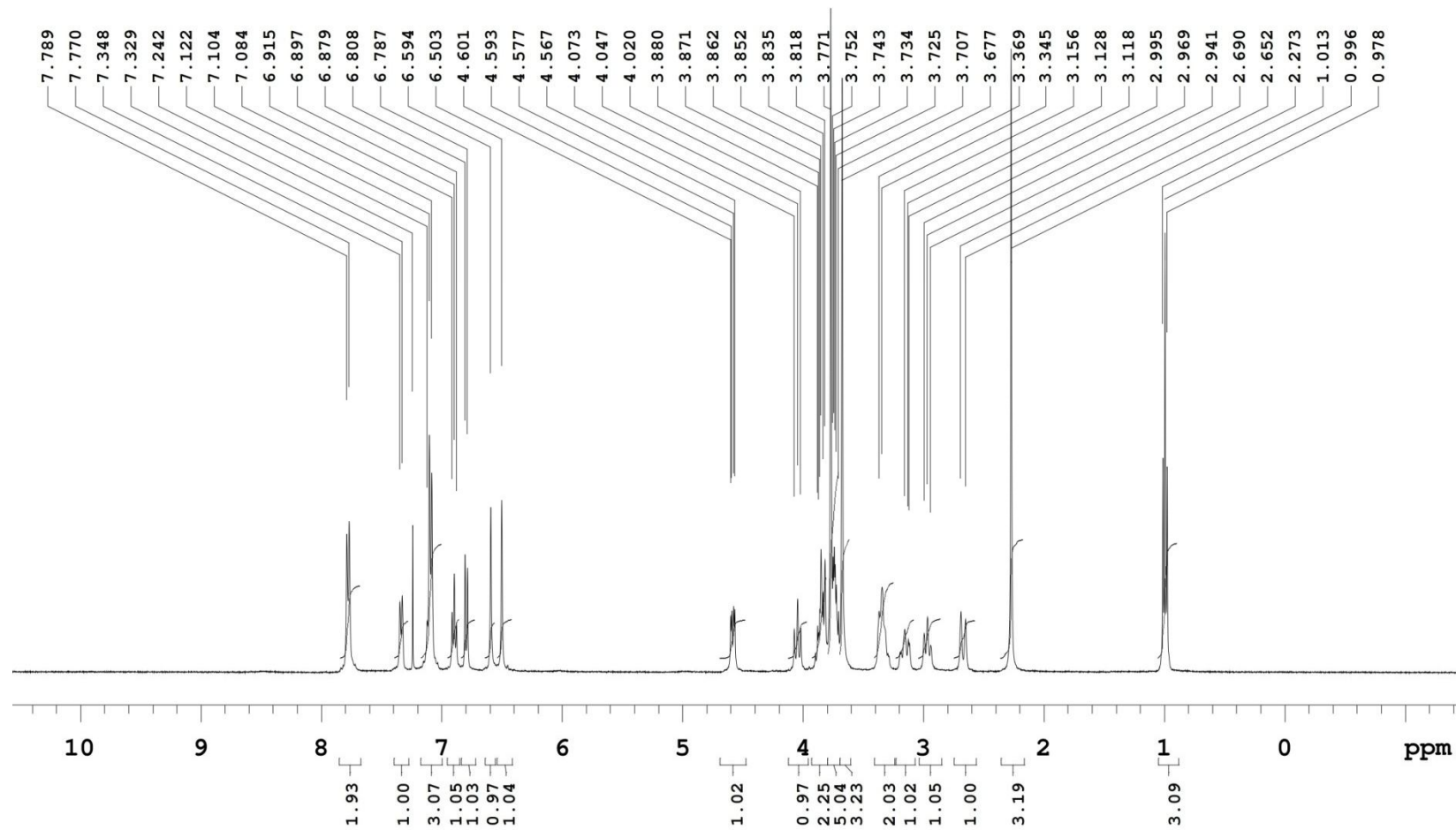
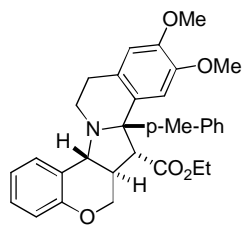
Table 2: Bond lengths ( $\text{\AA}$ ).

Atoms	Length	Atoms	Length
Br1-C2	1.904(2)	C15-C16	1.406(3)
C2-C25	1.381(3)	C16-C17	1.384(3)
C2-C3	1.387(3)	C17-C18	1.402(3)
C3-C4	1.376(3)	C18-C19	1.435(3)
C4-C5	1.390(3)	C19-C20	1.495(3)
C5-O6	1.369(3)	C20-C21	1.532(3)
C5-C24	1.403(3)	C21-N22	1.484(2)
O6-C7	1.443(3)	N22-C23	1.478(3)
C7-C8	1.516(3)	C23-C24	1.499(3)
C8-C23	1.523(3)	C24-C25	1.395(3)
C8-C9	1.532(3)	C26-C31	1.387(3)
C9-C33	1.514(3)	C26-C27	1.397(3)
C9-C10	1.614(3)	C27-C28	1.386(3)
C10-N22	1.493(3)	C28-C29	1.387(3)
C10-C11	1.504(3)	C29-C30	1.381(3)
C10-C26	1.546(3)	C29-C132	1.741(2)
C11-C19	1.360(3)	C30-C31	1.395(3)
C11-N12	1.378(3)	C33-O34	1.209(3)
N12-C13	1.378(3)	C33-O35	1.334(2)
C13-C14	1.392(3)	O35-C36	1.456(2)
C13-C18	1.420(3)	C36-C37	1.506(3)
C14-C15	1.385(3)		

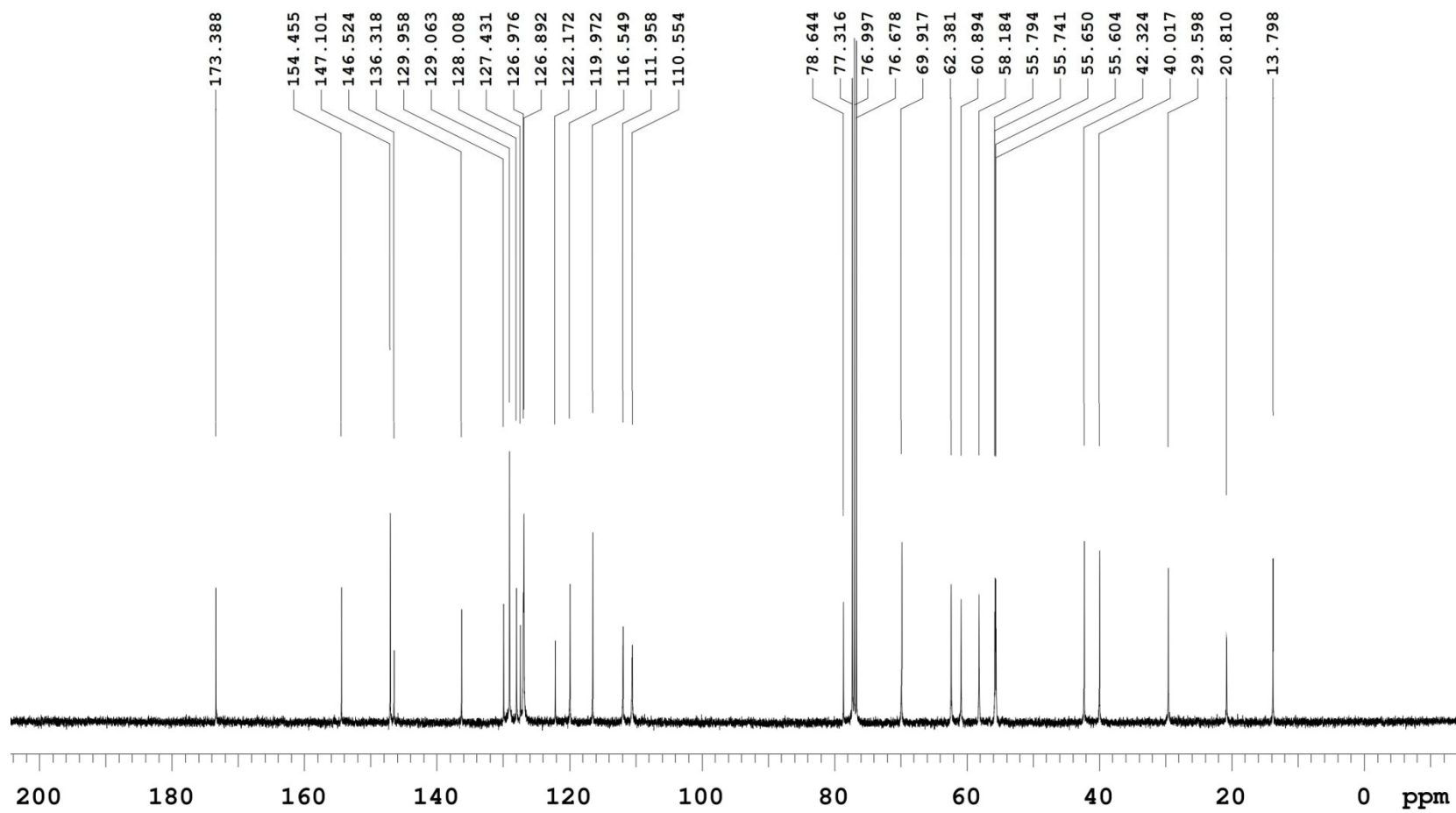
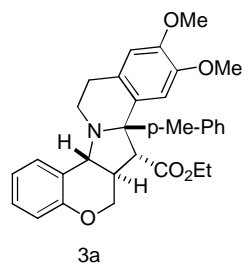
Table 3: Bond angles (°).

Atoms	Angle	Atoms	Angle
C25-C2-C3	121.0(2)	C17-C18-C13	118.72(19)
C25-C2-Br1	120.01(17)	C17-C18-C19	134.58(19)
C3-C2-Br1	118.95(17)	C13-C18-C19	106.68(17)
C4-C3-C2	119.3(2)	C11-C19-C18	106.57(18)
C3-C4-C5	120.7(2)	C11-C19-C20	121.23(18)
O6-C5-C4	115.67(19)	C18-C19-C20	132.19(18)
O6-C5-C24	124.4(2)	C19-C20-C21	108.48(16)
C4-C5-C24	119.9(2)	N22-C21-C20	109.64(16)
C5-O6-C7	118.03(16)	C23-N22-C21	115.14(15)
O6-C7-C8	108.07(17)	C23-N22-C10	100.67(14)
C7-C8-C23	109.03(16)	C21-N22-C10	114.62(15)
C7-C8-C9	120.24(17)	N22-C23-C24	120.64(17)
C23-C8-C9	101.80(16)	N22-C23-C8	104.31(15)
C33-C9-C8	111.98(16)	C24-C23-C8	110.13(16)
C33-C9-C10	114.35(15)	C25-C24-C5	118.84(19)
C8-C9-C10	102.51(15)	C25-C24-C23	123.36(19)
N22-C10-C11	109.88(16)	C5-C24-C23	117.67(18)
N22-C10-C26	107.57(15)	C2-C25-C24	120.1(2)
C11-C10-C26	105.35(15)	C31-C26-C27	118.56(18)
N22-C10-C9	106.39(15)	C31-C26-C10	123.30(17)
C11-C10-C9	115.43(15)	C27-C26-C10	117.75(17)
C26-C10-C9	112.01(15)	C28-C27-C26	121.25(19)
C19-C11-N12	110.83(17)	C27-C28-C29	119.01(19)
C19-C11-C10	125.37(18)	C30-C29-C28	120.90(18)
N12-C11-C10	123.40(17)	C30-C29-C132	120.10(16)
C11-N12-C13	108.13(16)	C28-C29-C132	118.99(16)
N12-C13-C14	129.87(19)	C29-C30-C31	119.46(19)
N12-C13-C18	107.78(17)	C26-C31-C30	120.78(19)
C14-C13-C18	122.35(19)	O34-C33-O35	123.99(18)
C15-C14-C13	117.6(2)	O34-C33-C9	124.55(18)
C14-C15-C16	121.1(2)	O35-C33-C9	111.45(16)
C17-C16-C15	121.2(2)	C33-O35-C36	118.01(15)
C16-C17-C18	119.00(19)	O35-C36-C37	110.88(16)

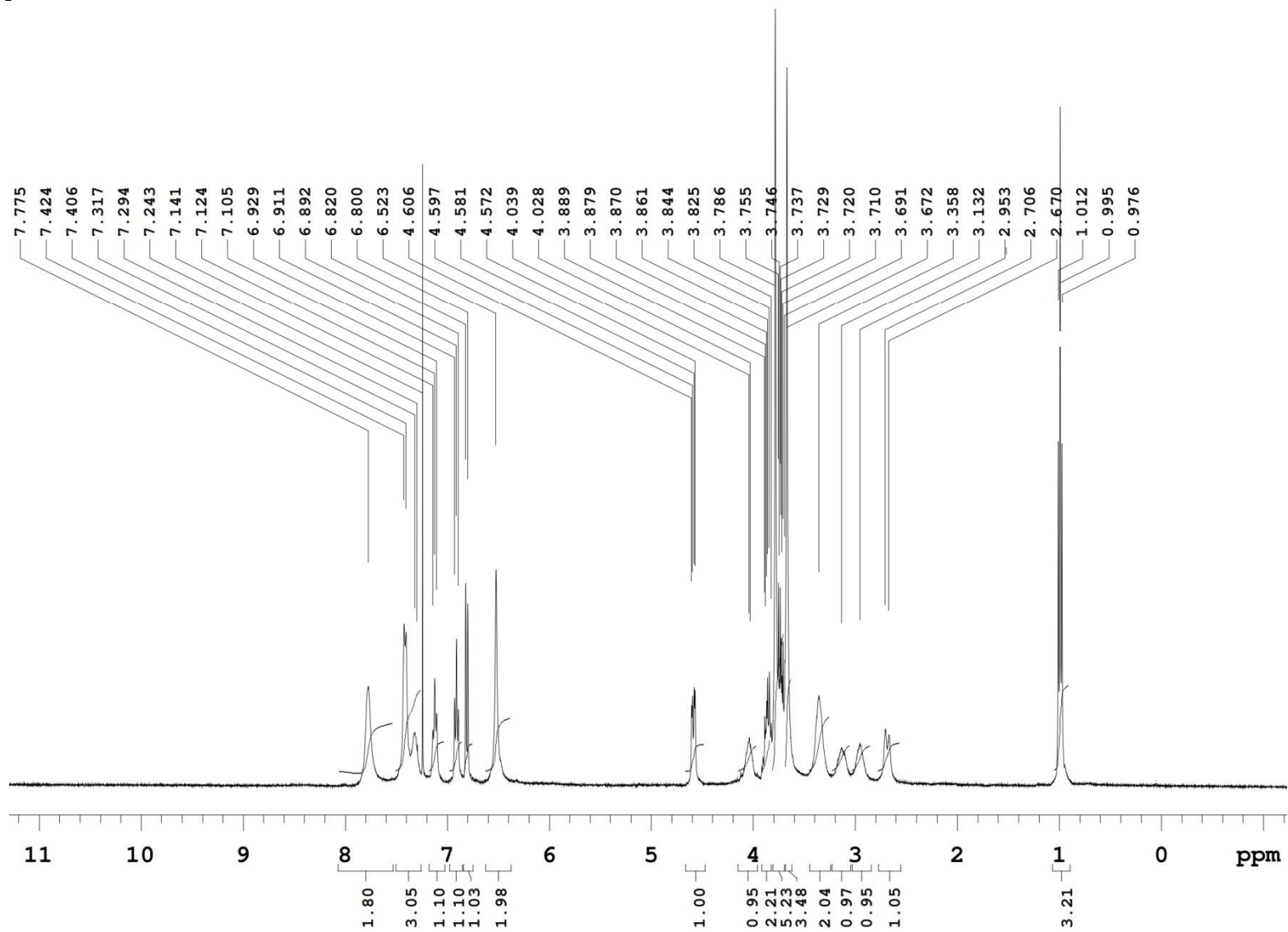
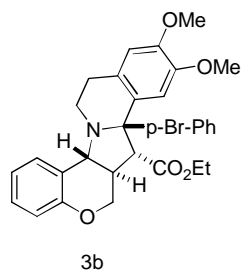
<sup>1</sup>H NMR of **3a** in CDCl<sub>3</sub>



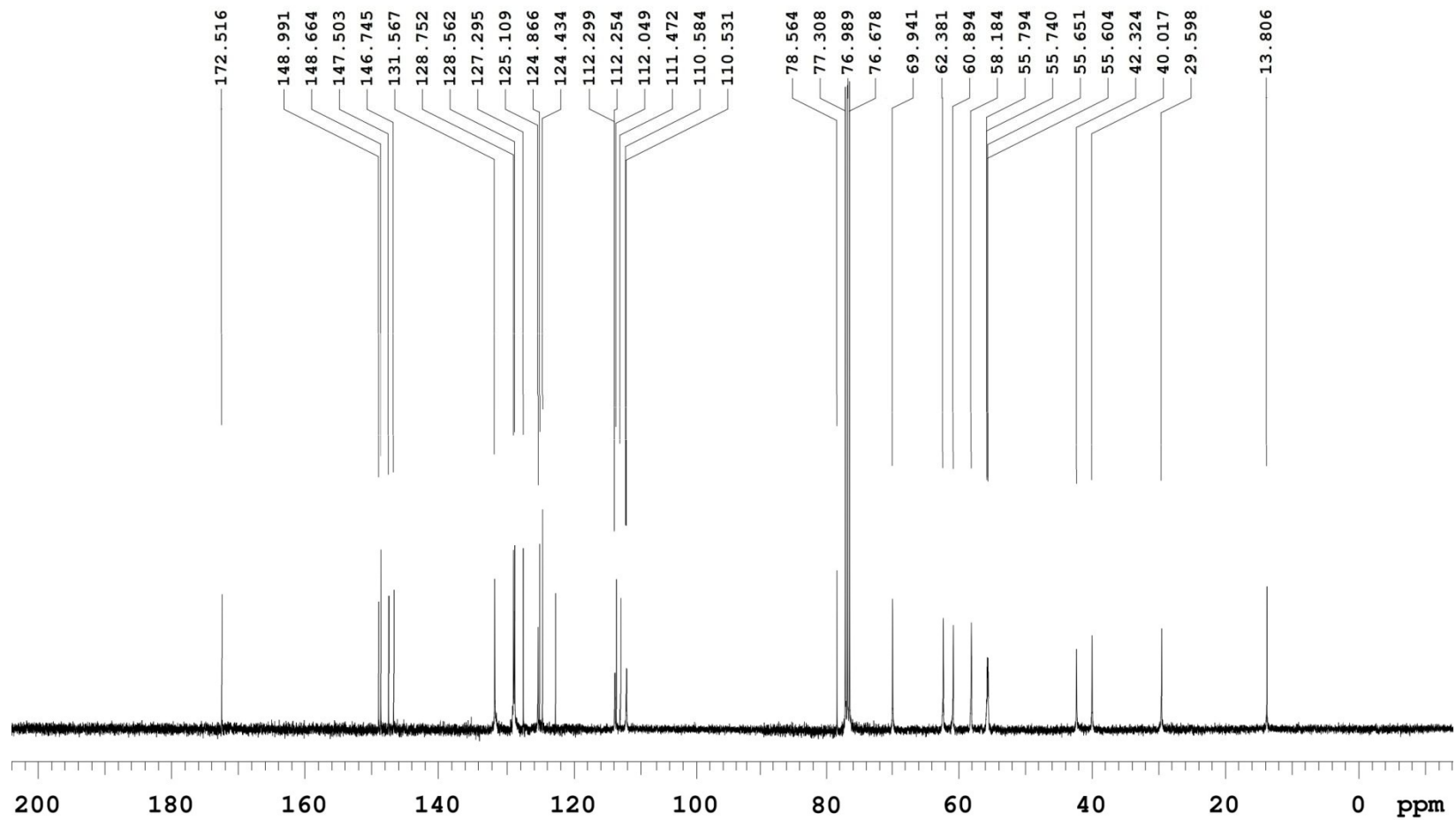
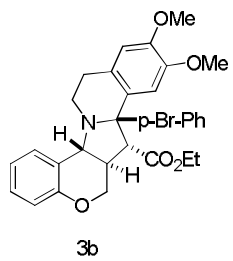
<sup>13</sup>C NMR of **3a** in CDCl<sub>3</sub>



$^1\text{H}$  NMR of **3b** in  $\text{CDCl}_3$

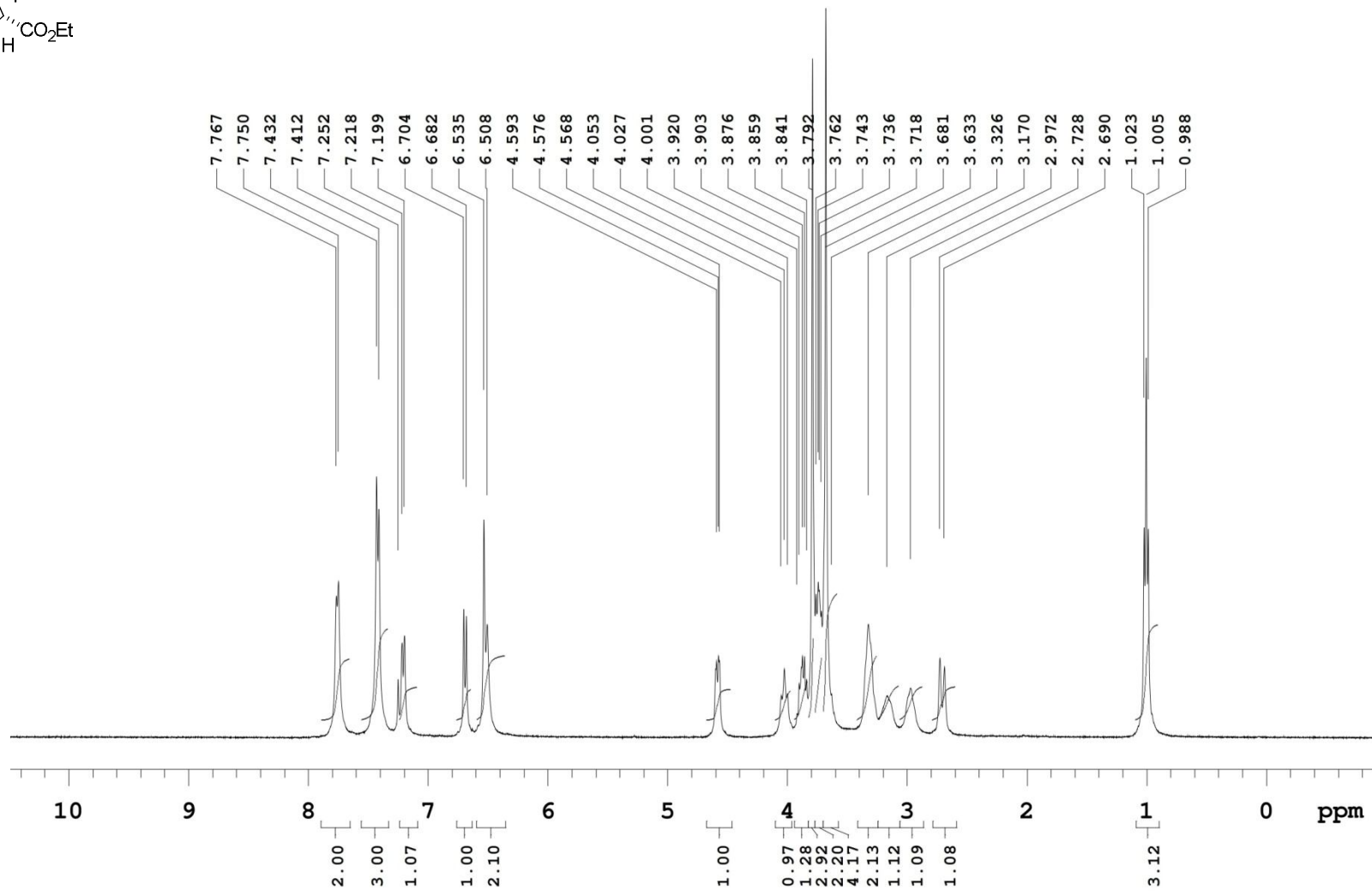
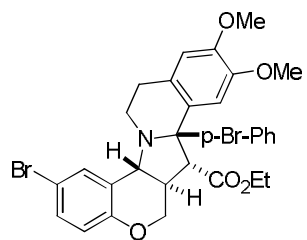


$^{13}\text{C}$  NMR of **3b** in  $\text{CDCl}_3$

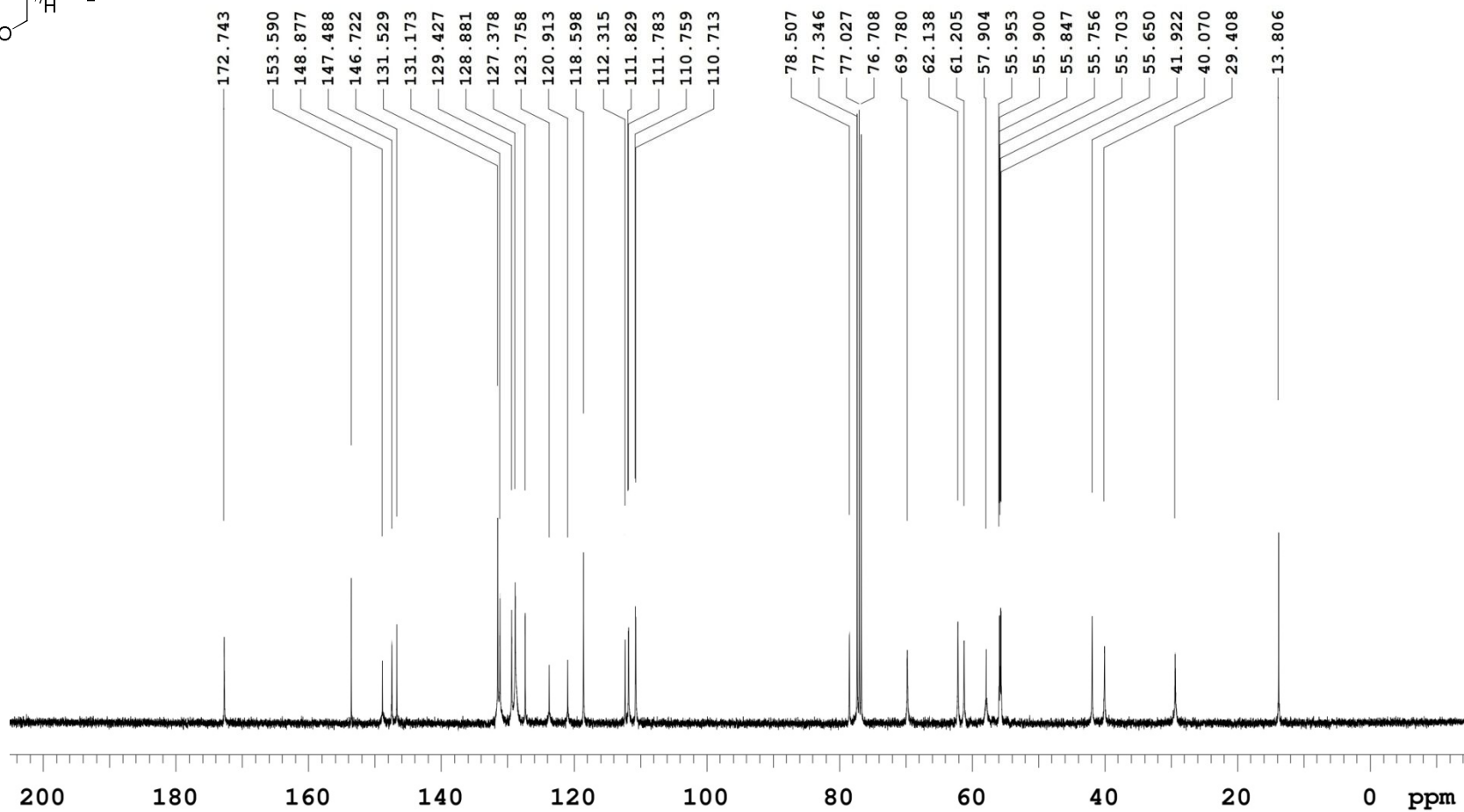
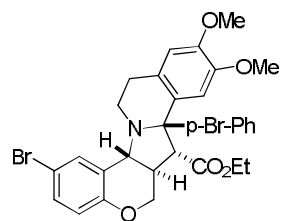




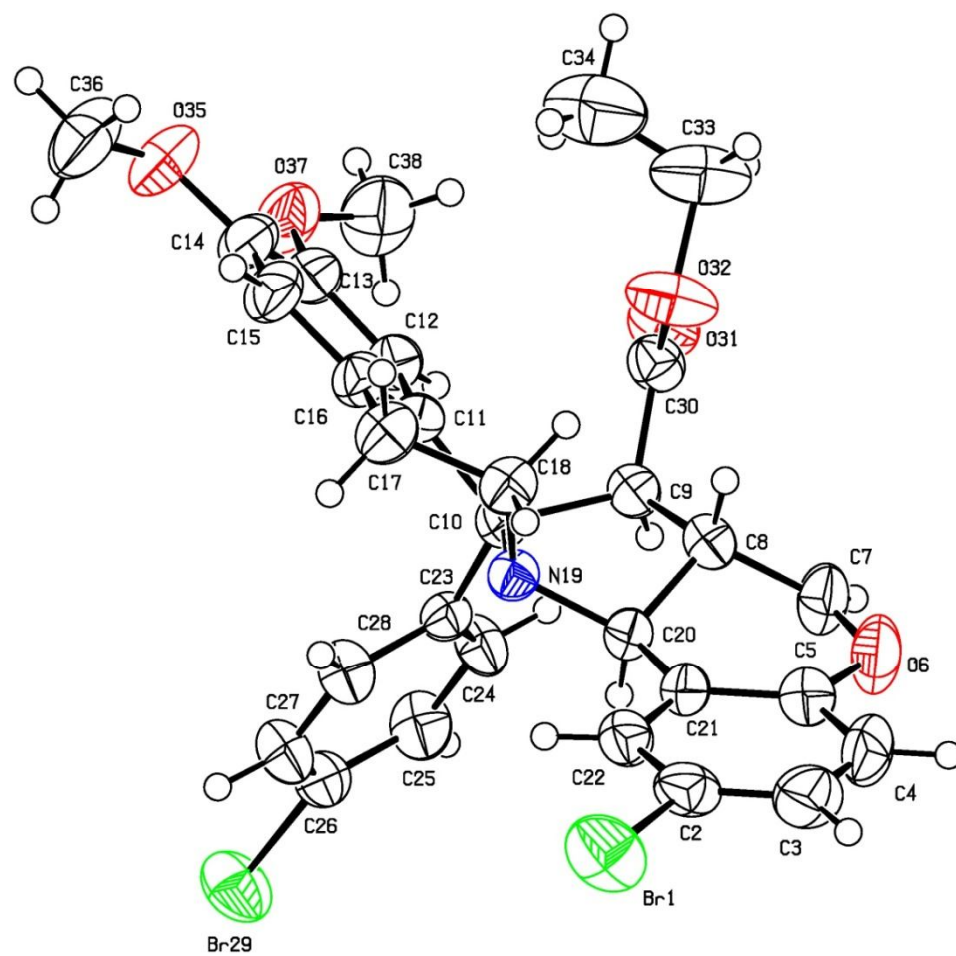
<sup>1</sup>H NMR of **3c** in CDCl<sub>3</sub>



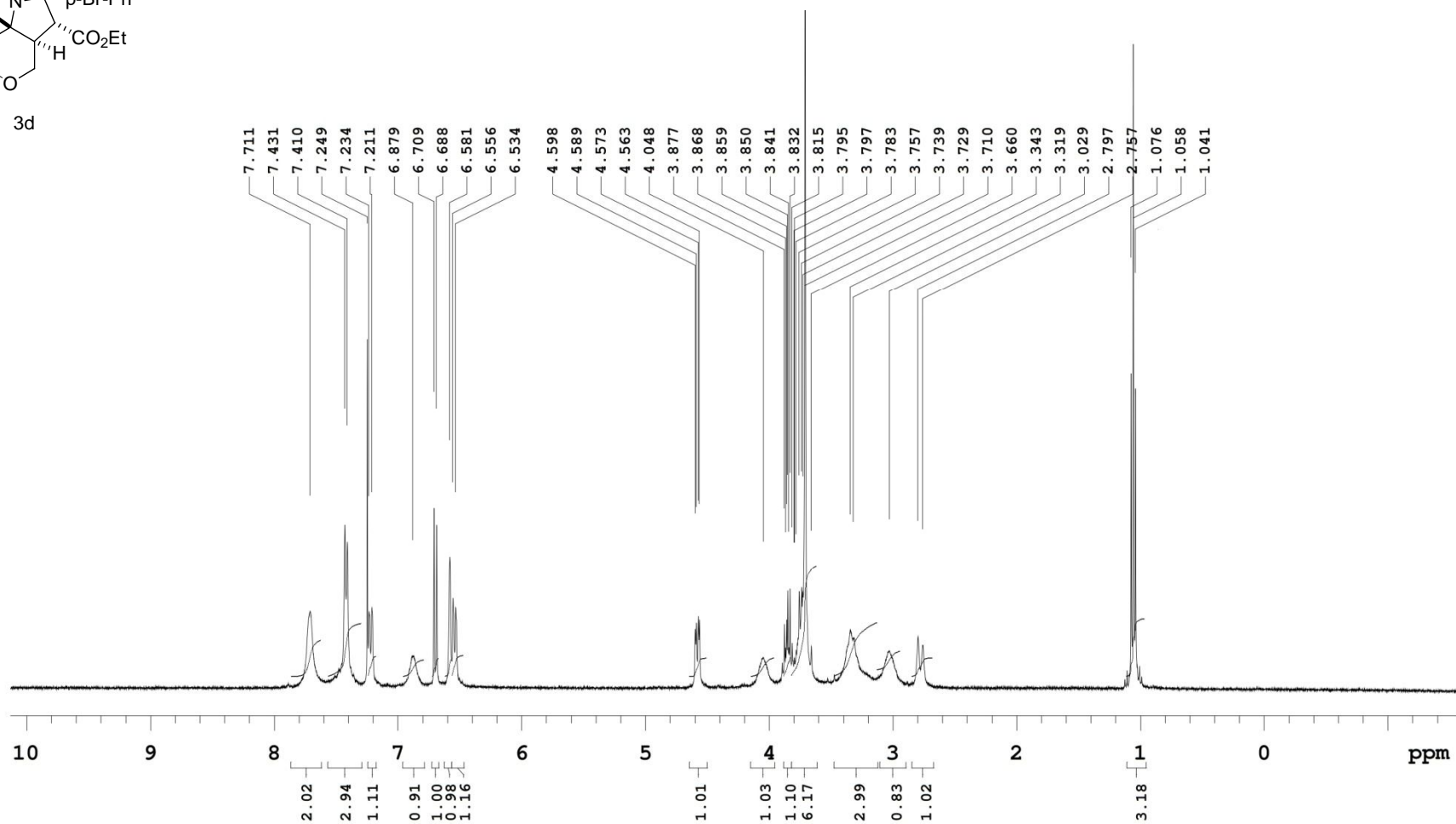
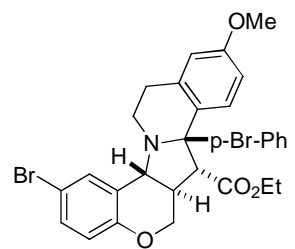
<sup>13</sup>C NMR of 3c in CDCl<sub>3</sub>



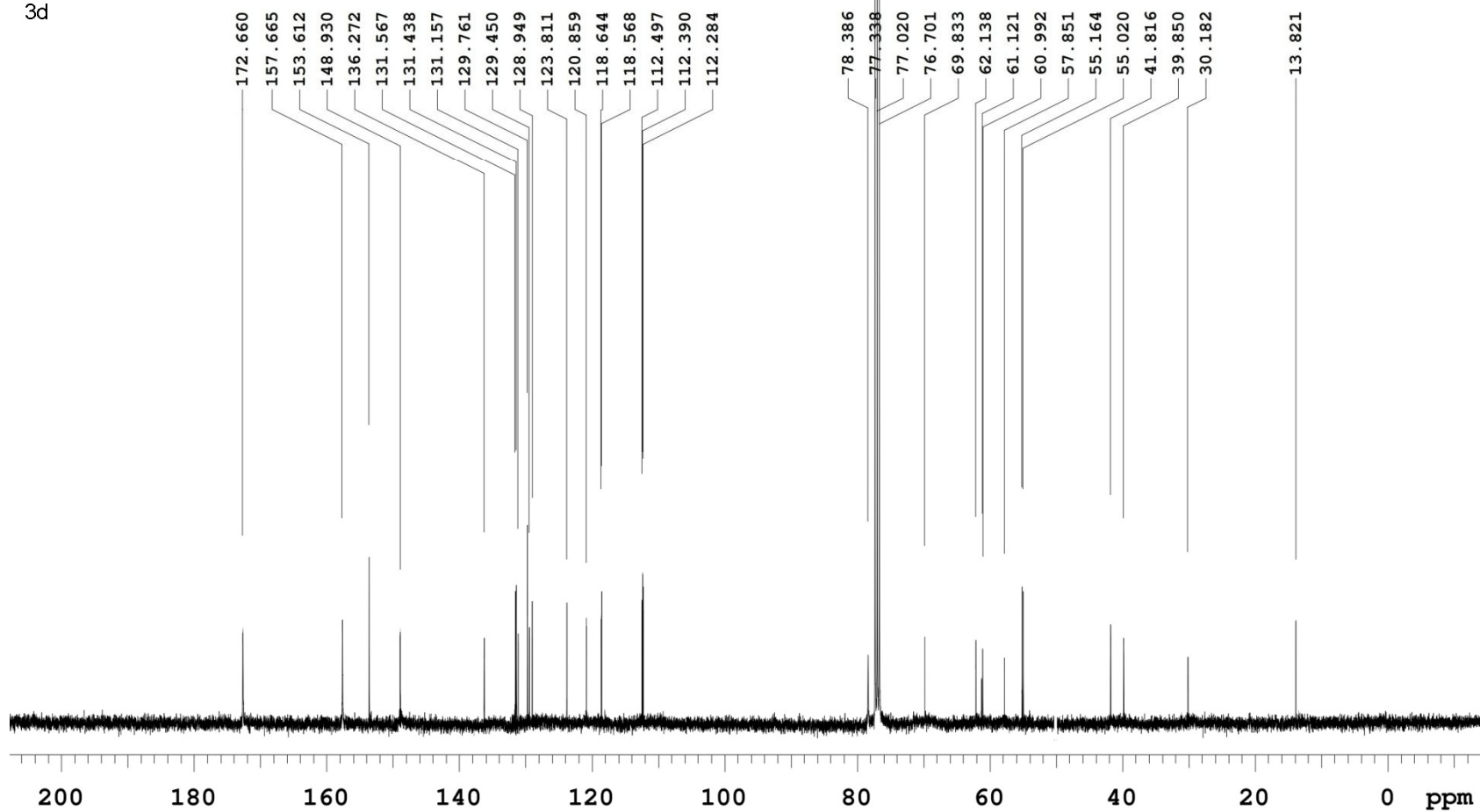
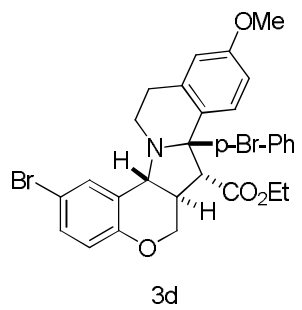
ORTEP diagram of **3c**



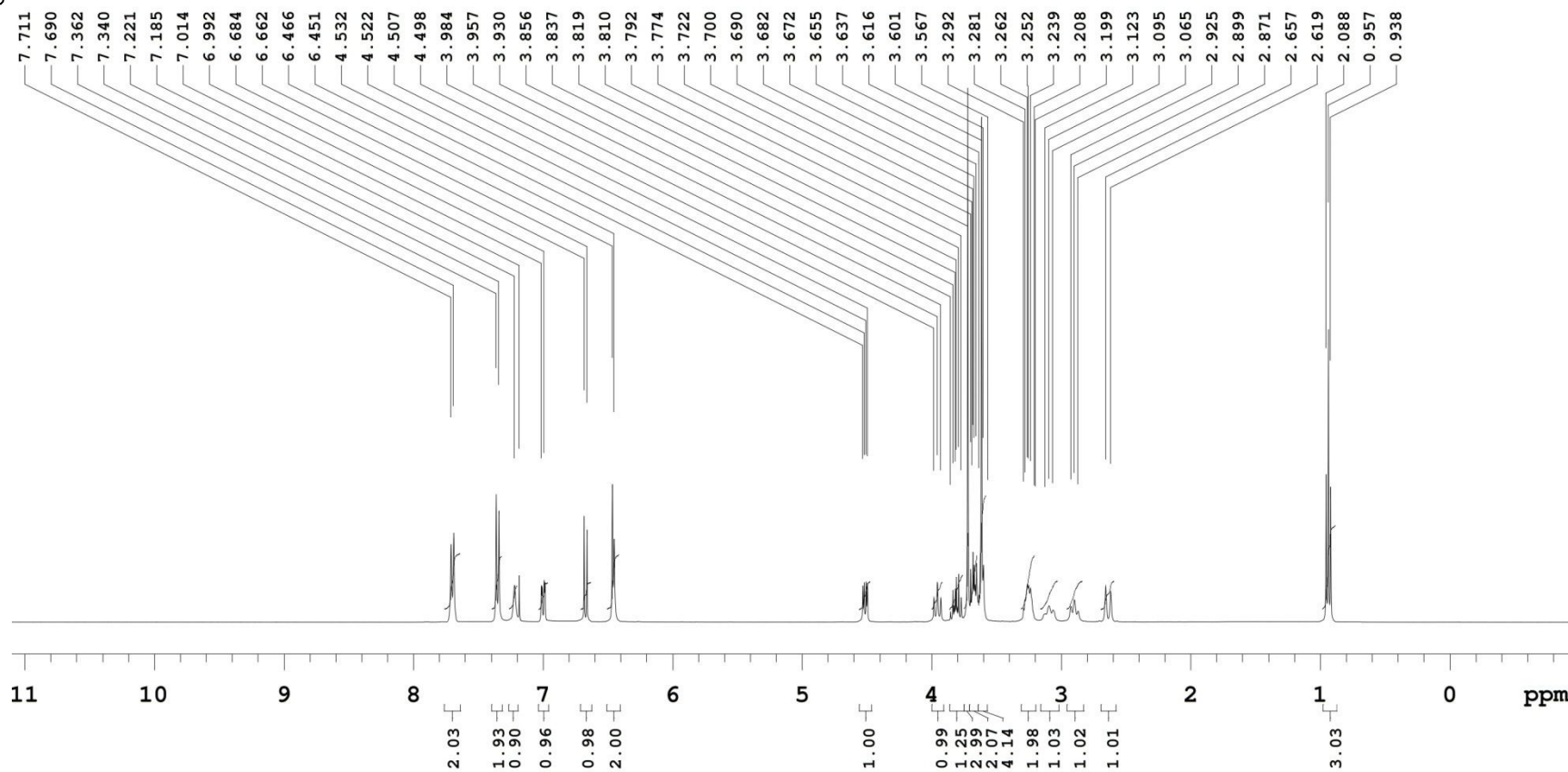
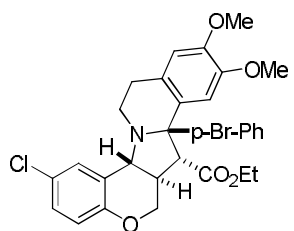
$^1\text{H}$  NMR of **3d** in  $\text{CDCl}_3$



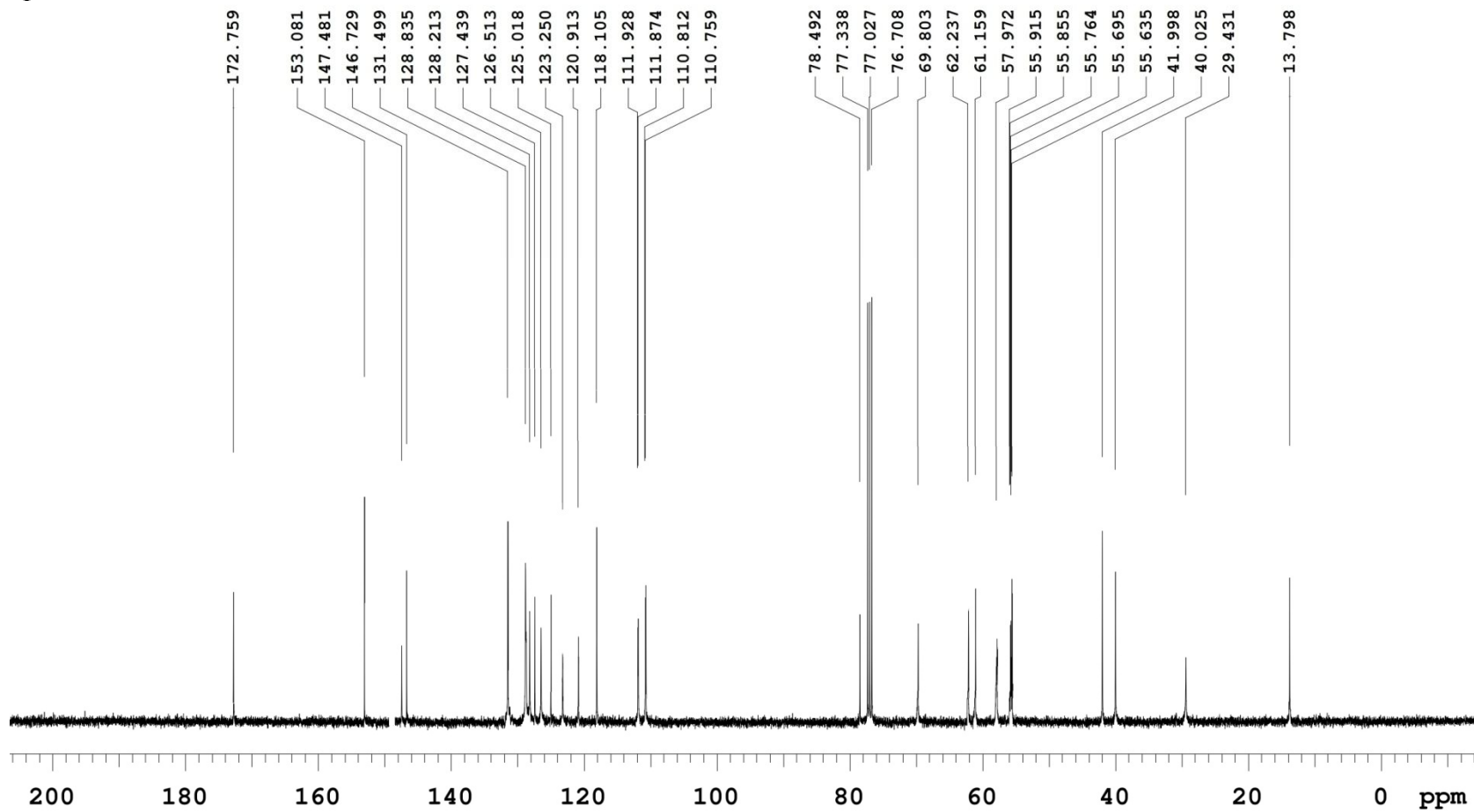
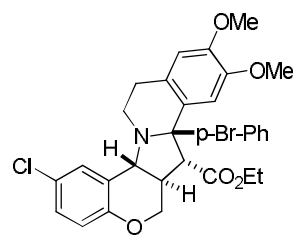
<sup>13</sup>C NMR of **3d** in CDCl<sub>3</sub>



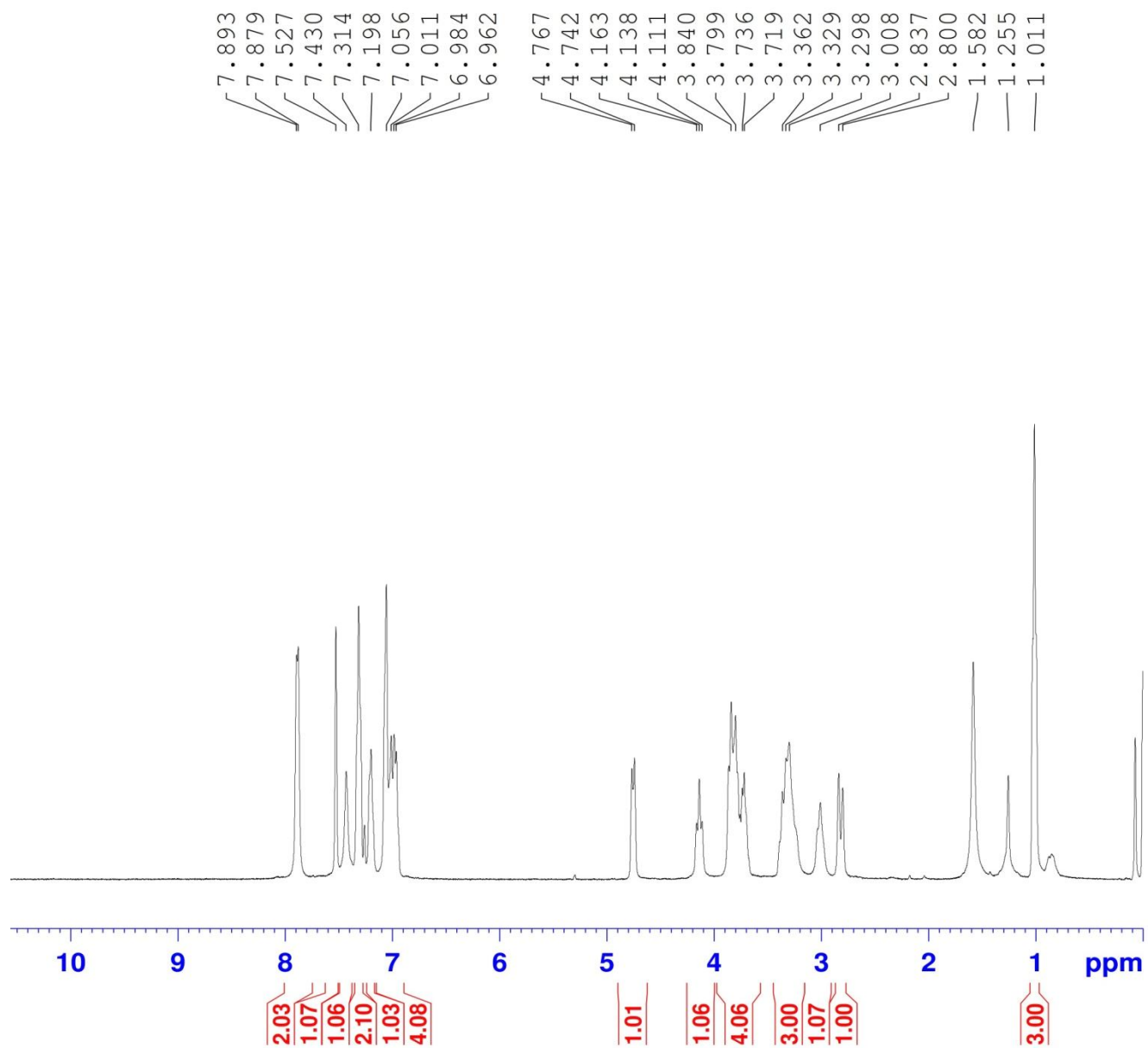
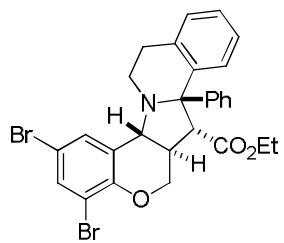
<sup>1</sup>H NMR of **3e** in CDCl<sub>3</sub>



<sup>13</sup>C NMR of **3e** in CDCl<sub>3</sub>

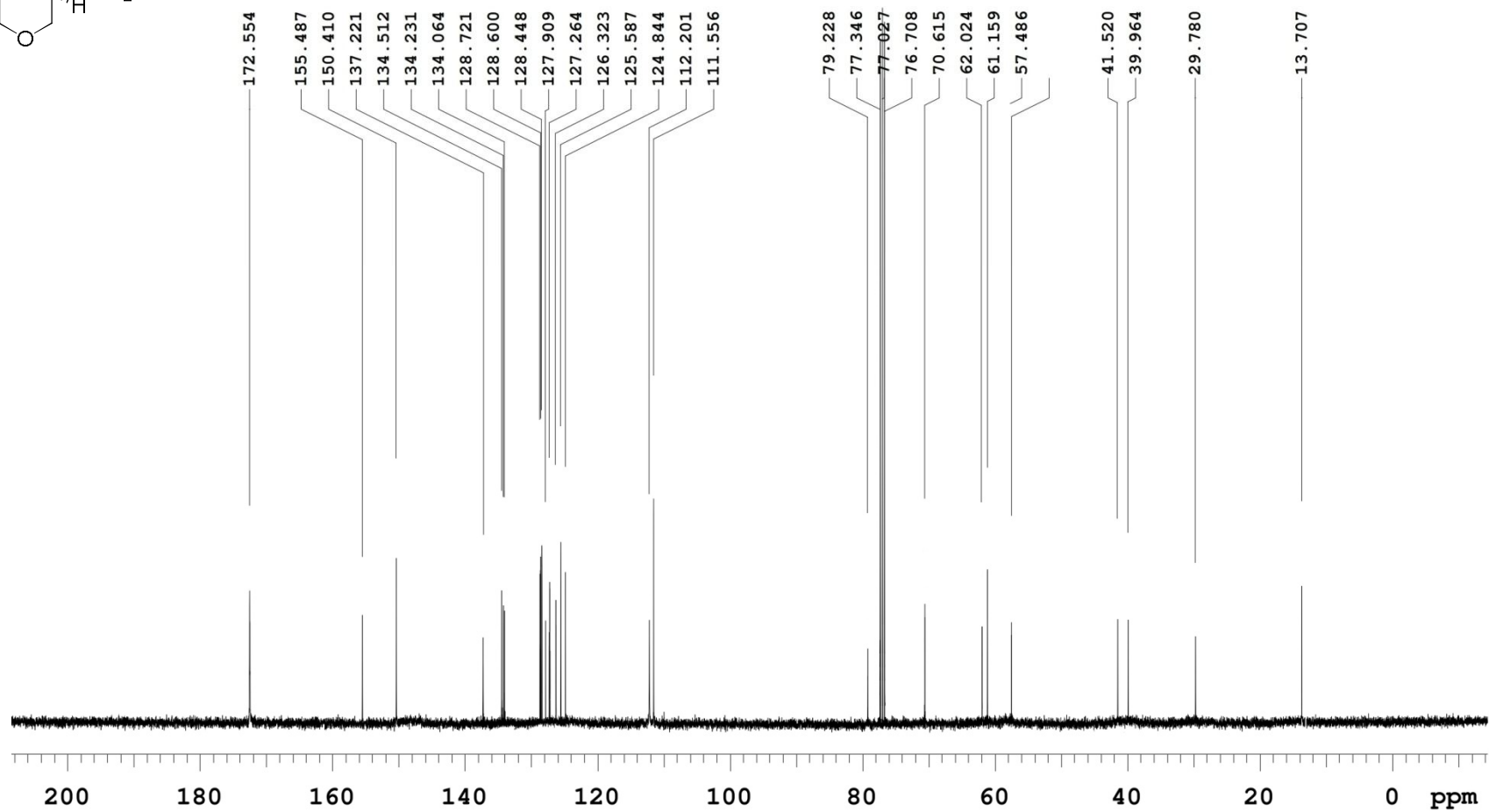
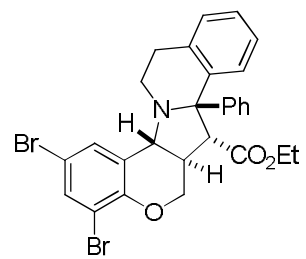


$^1\text{H}$  NMR of **3f** in  $\text{CDCl}_3$

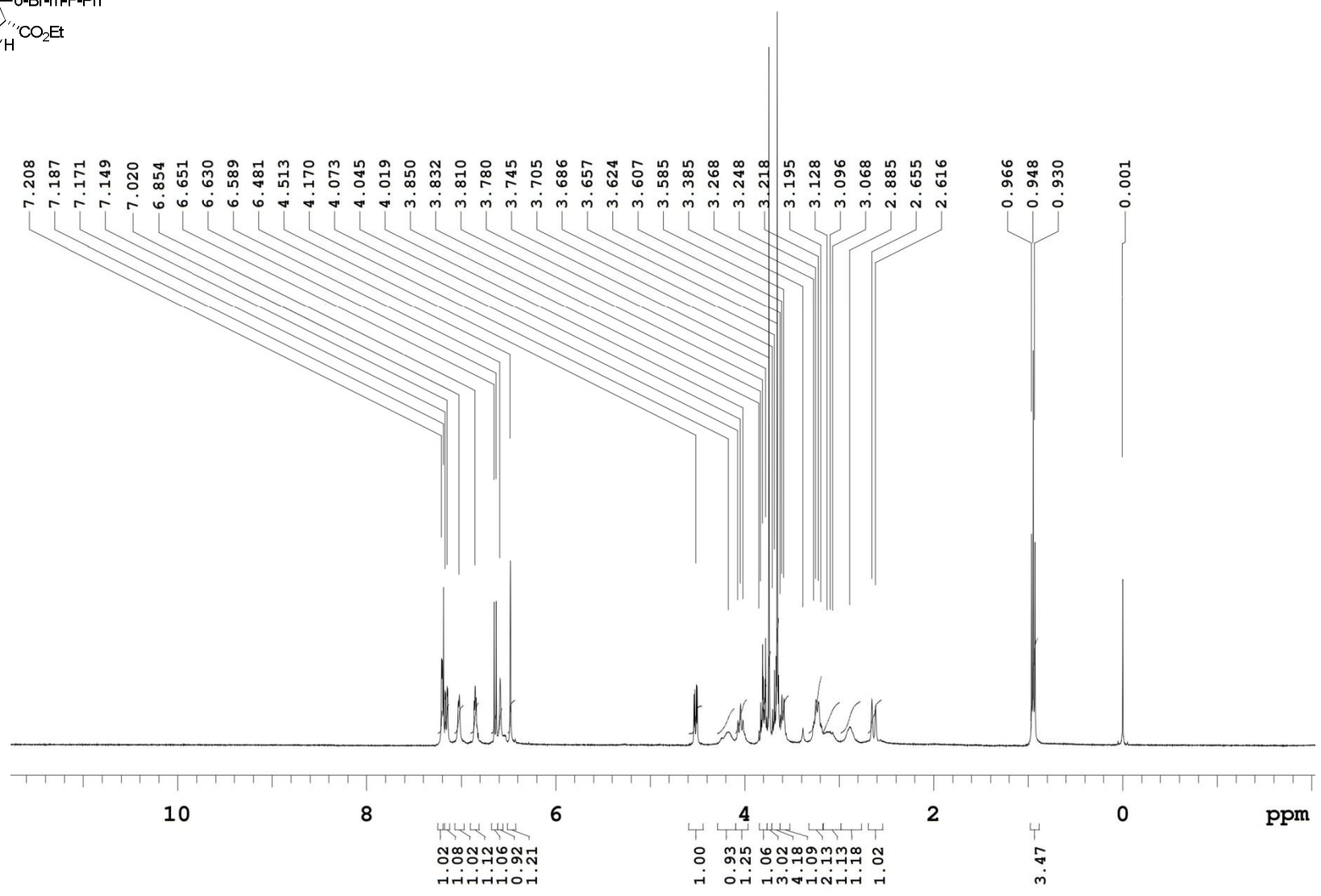
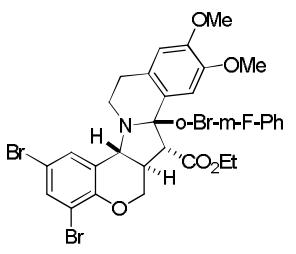




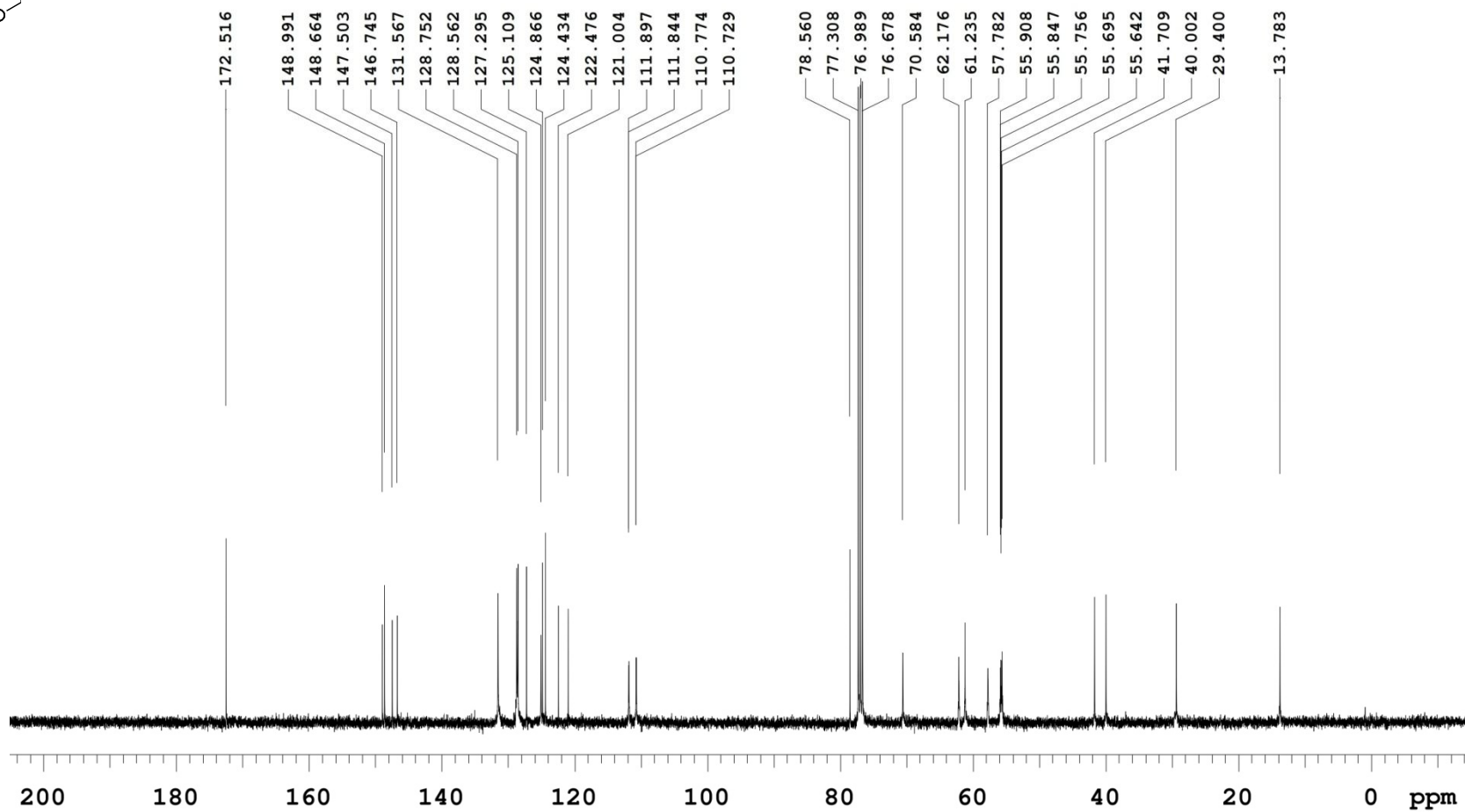
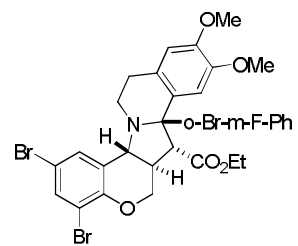
$^{13}\text{C}$  NMR of **3f** in  $\text{CDCl}_3$



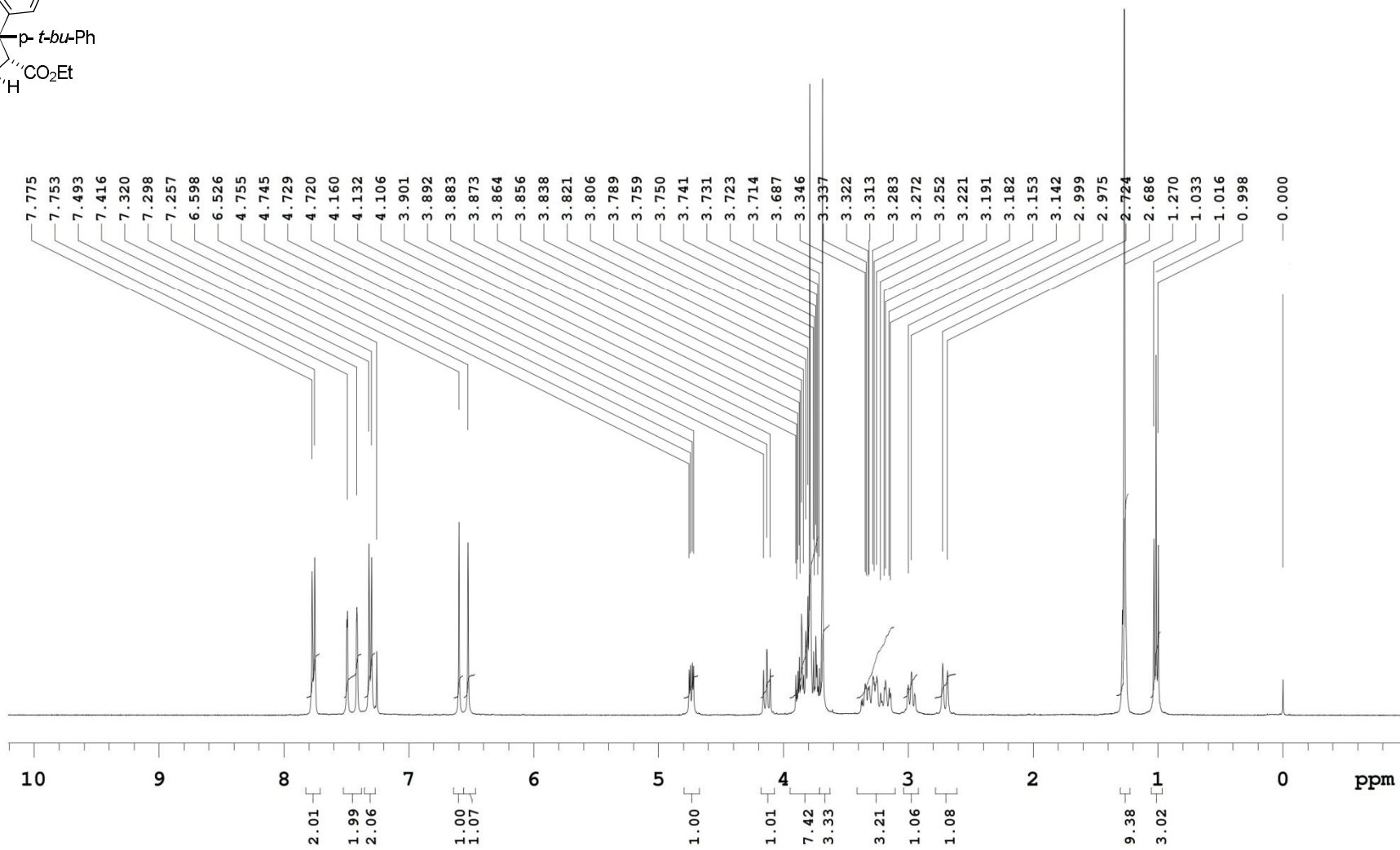
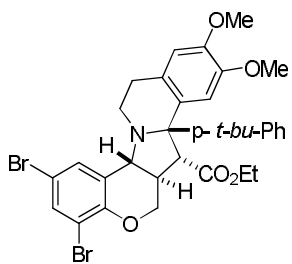
<sup>1</sup>H NMR of 3g in CDCl<sub>3</sub>



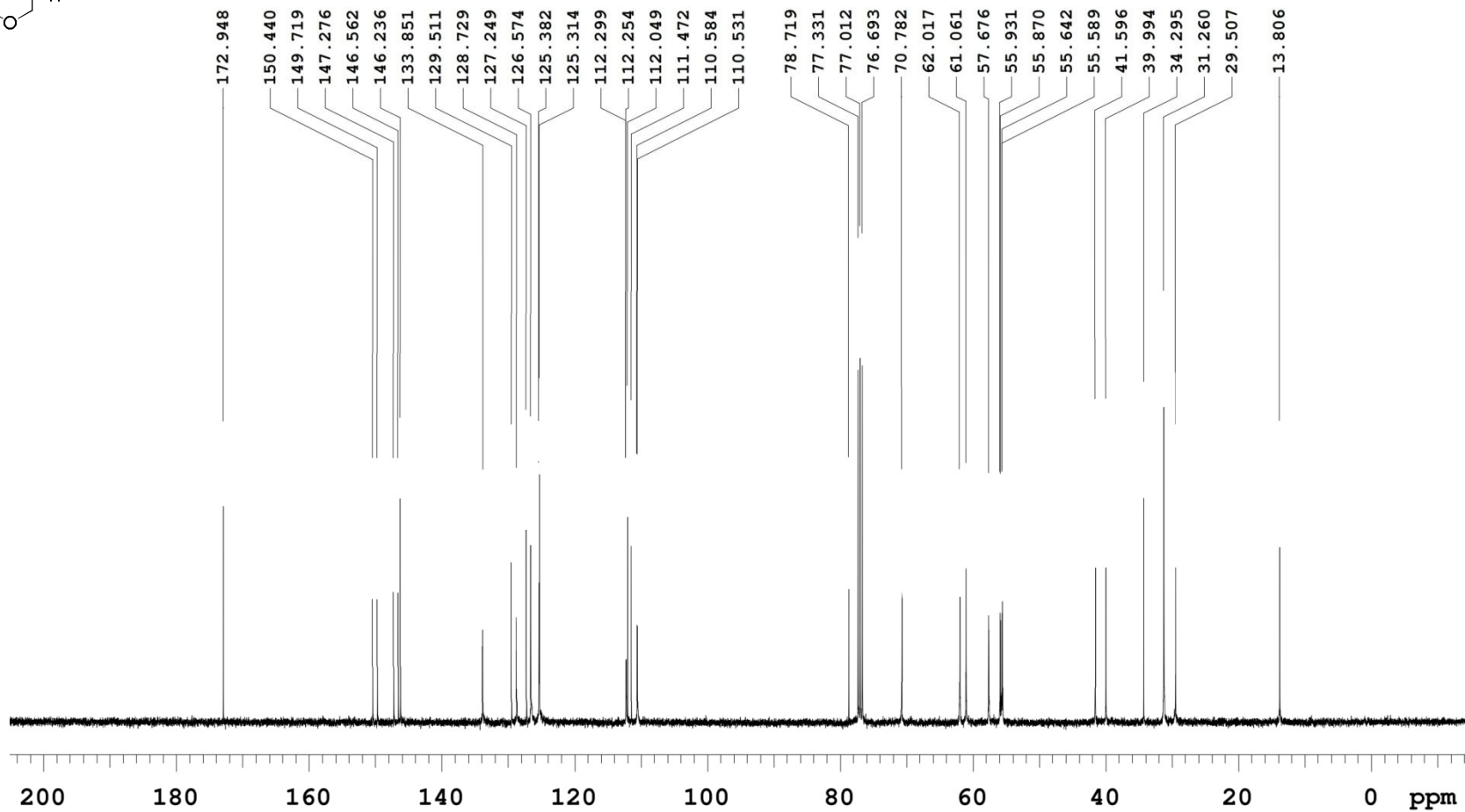
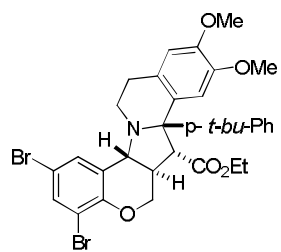
$^{13}\text{C}$  NMR of **3g** in  $\text{CDCl}_3$



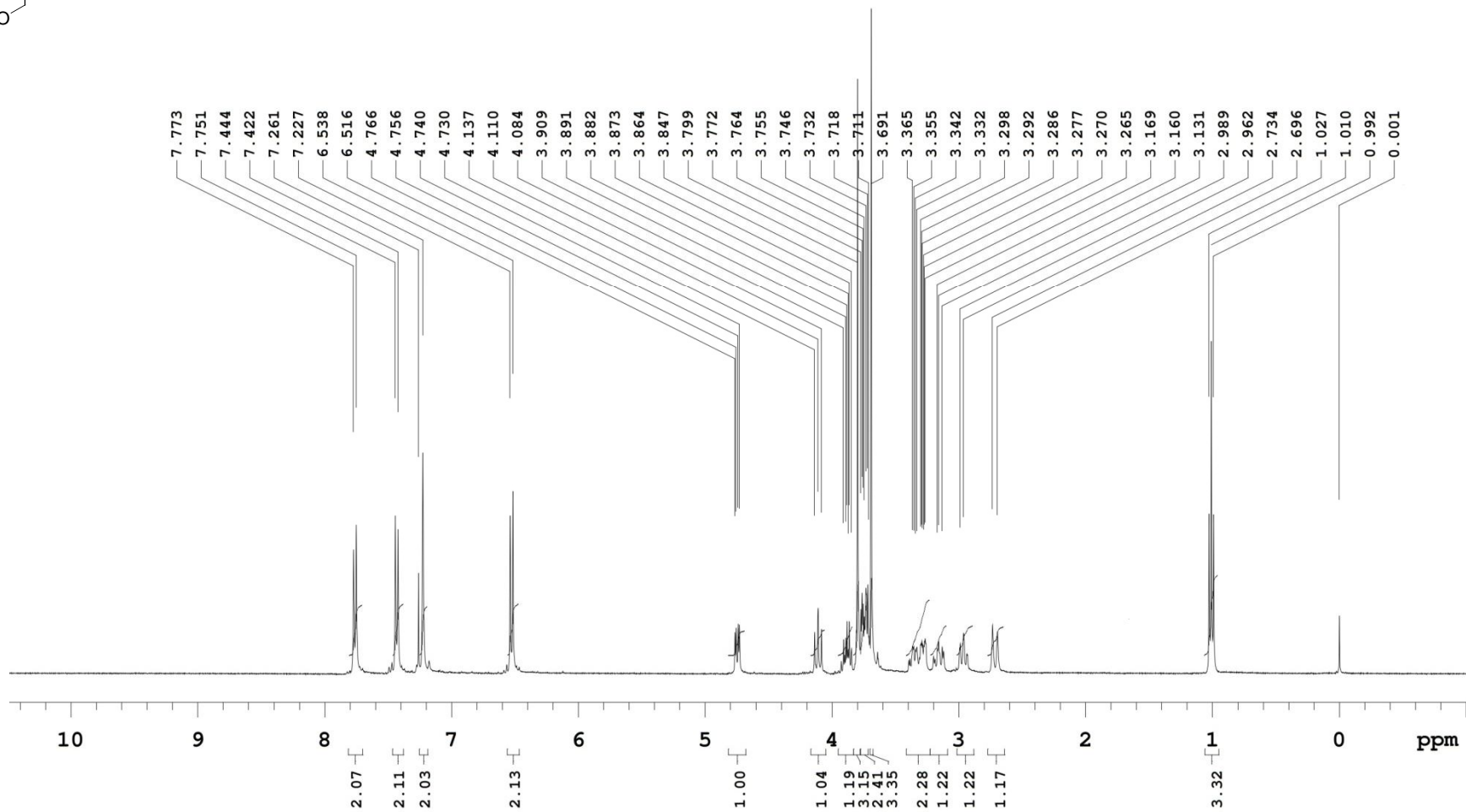
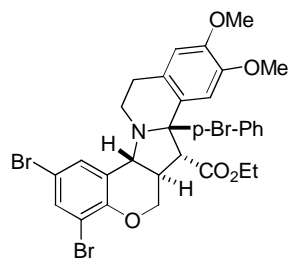
<sup>1</sup>H NMR of **3h** in CDCl<sub>3</sub>



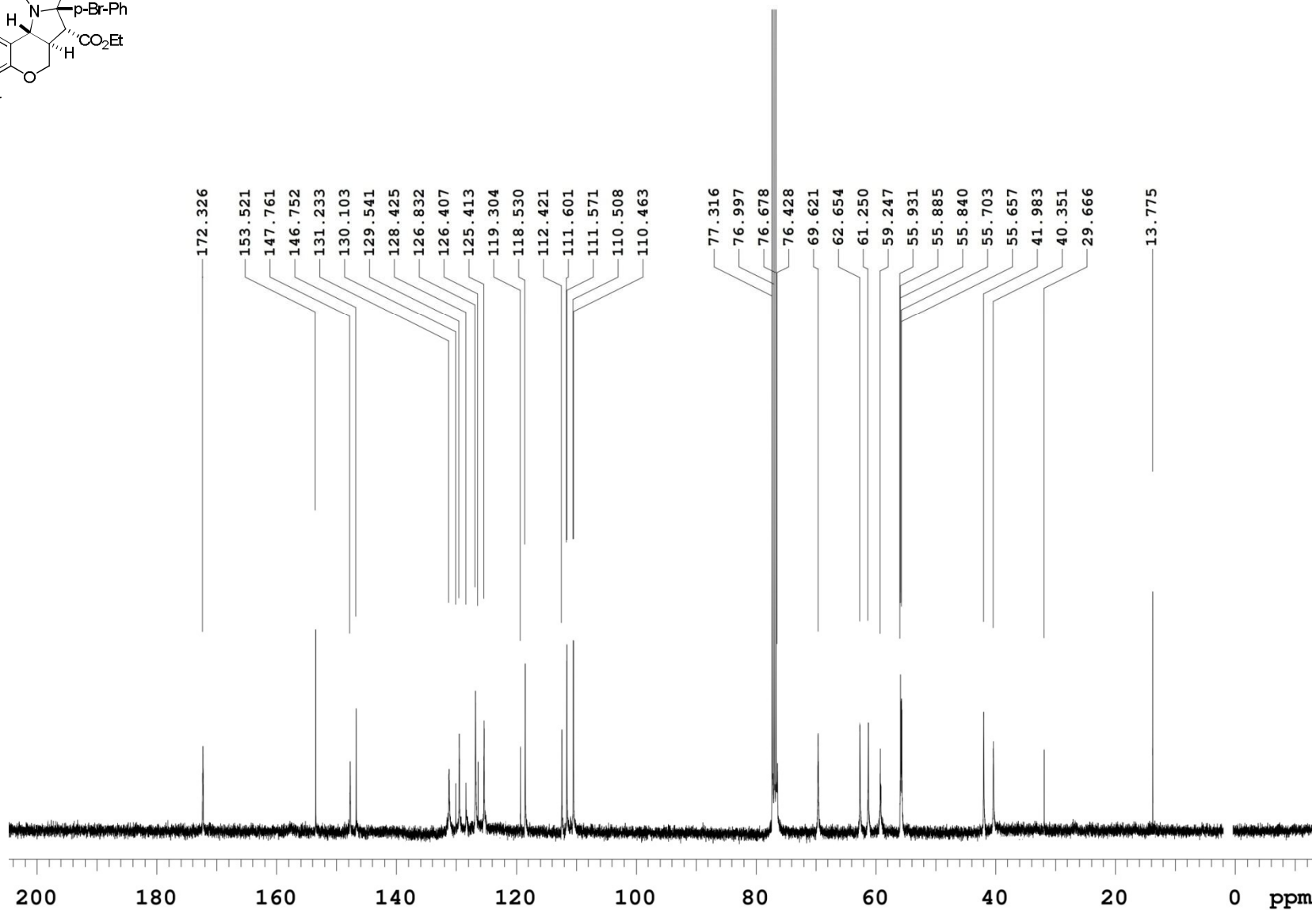
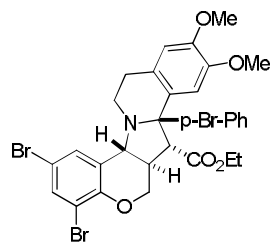
<sup>13</sup>C NMR of 3hin CDCl<sub>3</sub>



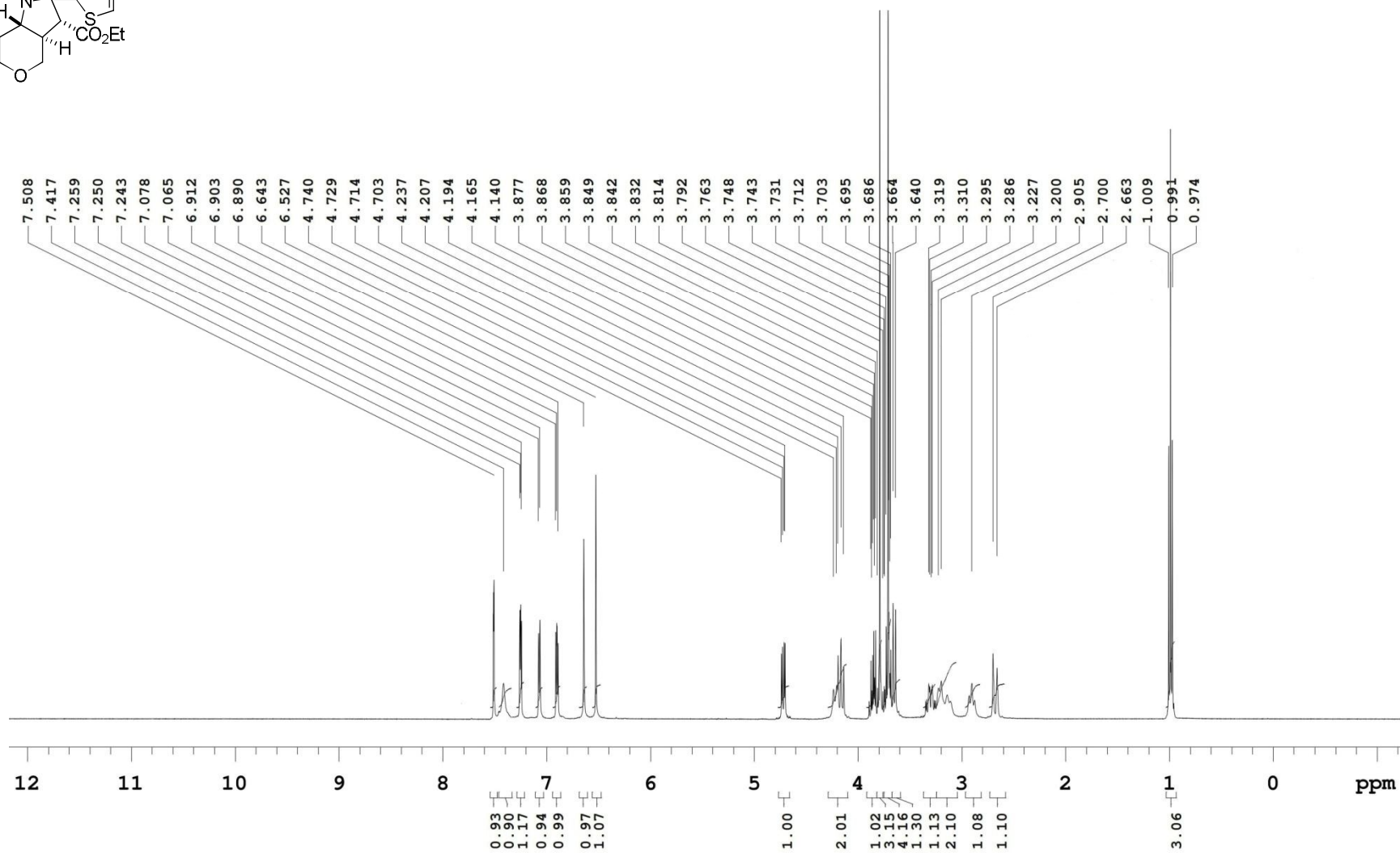
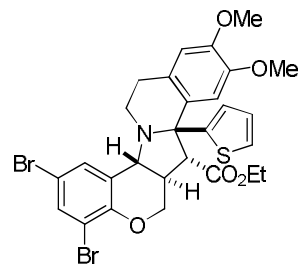
<sup>1</sup>H NMR of **3i** in CDCl<sub>3</sub>



$^{13}\text{C}$  NMR of **3i** in  $\text{CDCl}_3$

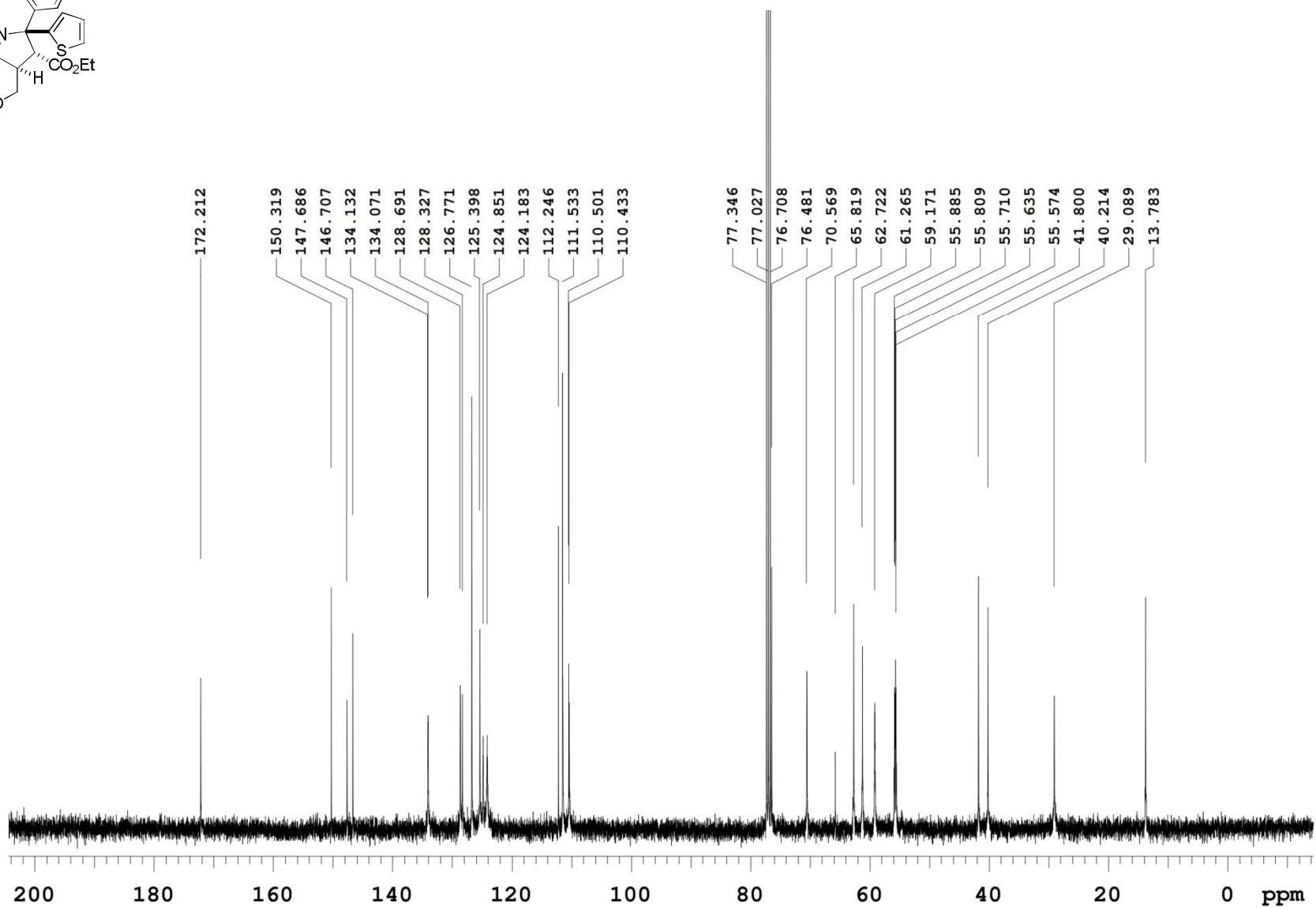
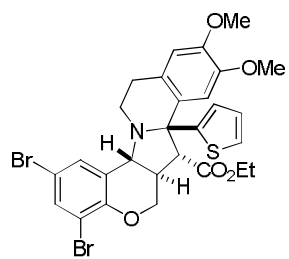


<sup>1</sup>H NMR of **3j** in CDCl<sub>3</sub>

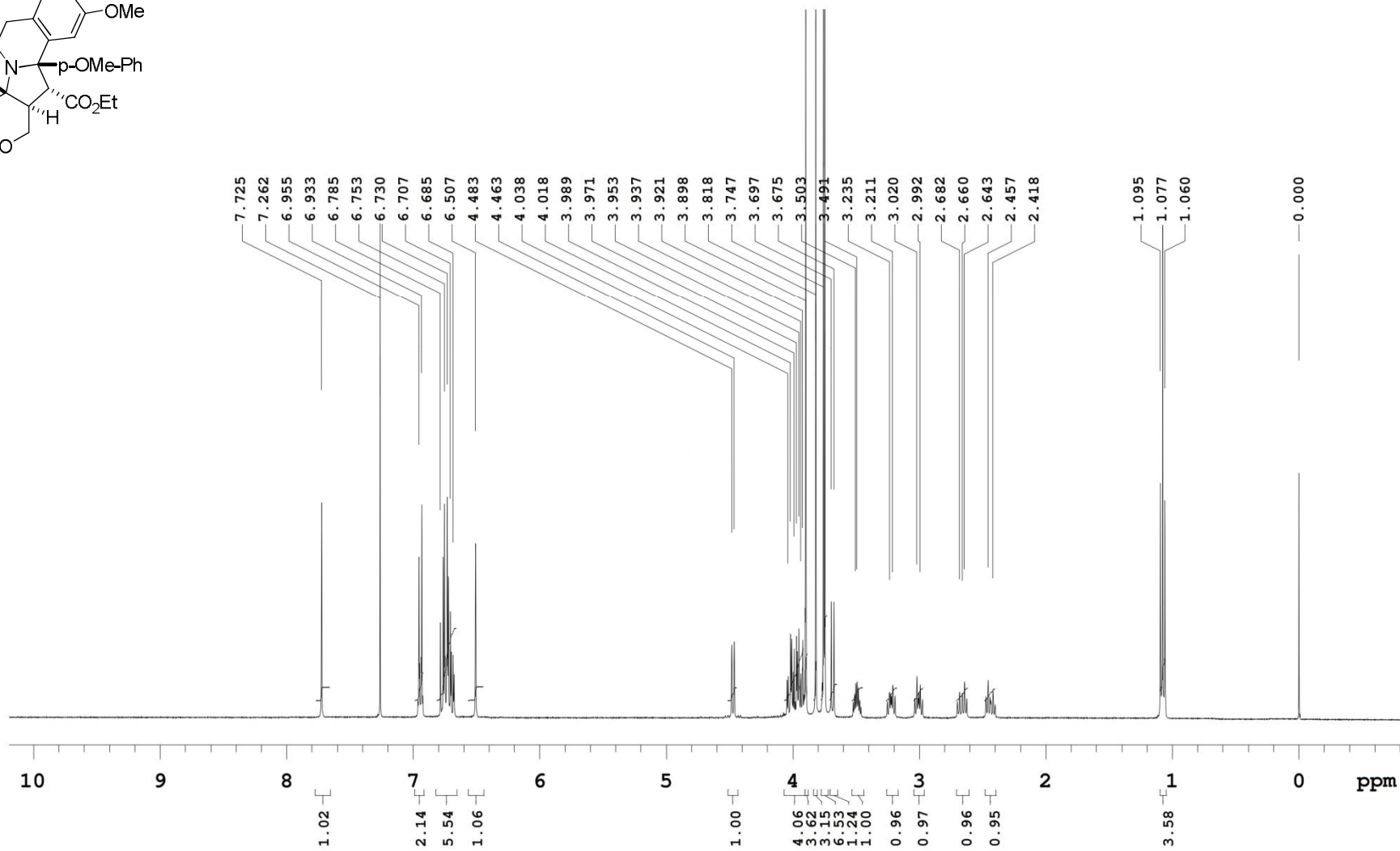
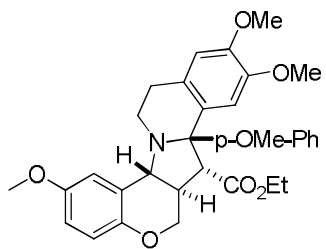




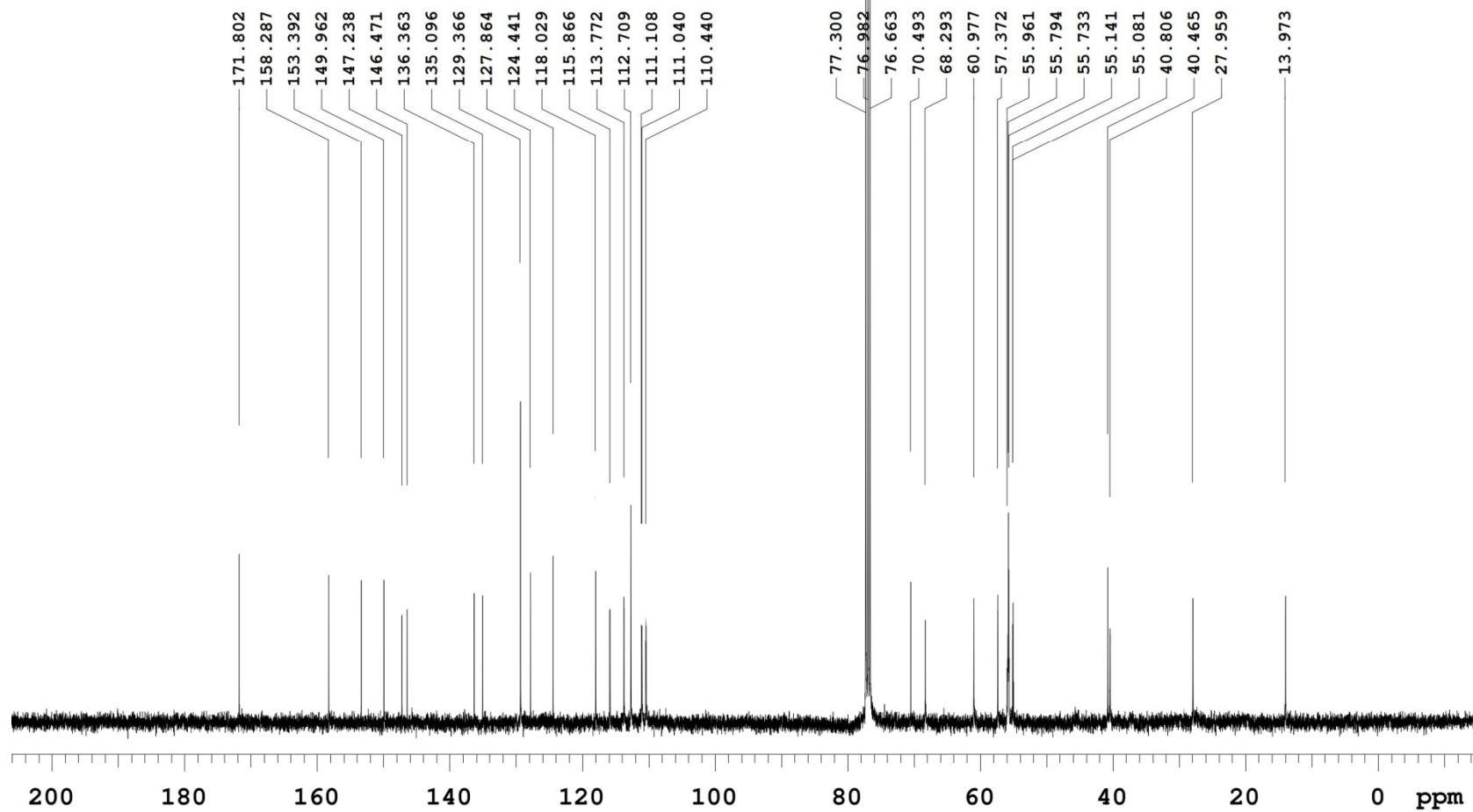
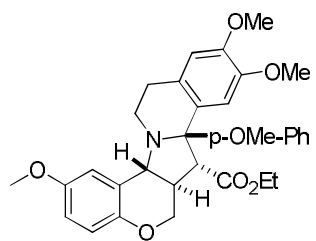
<sup>13</sup>C NMR of **3j** in CDCl<sub>3</sub>



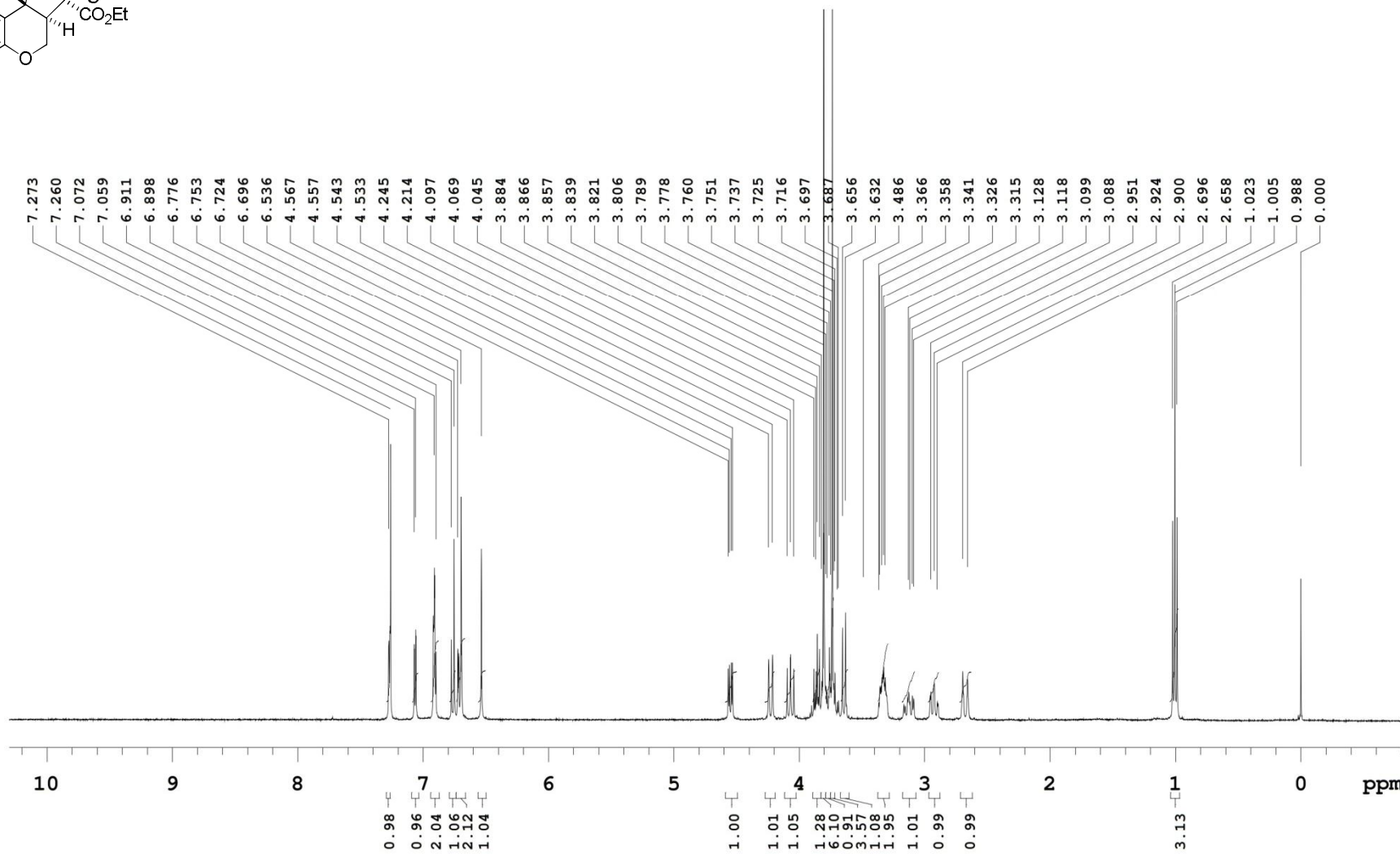
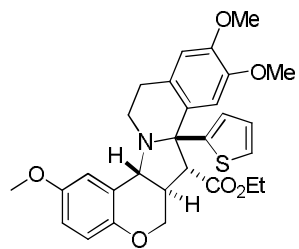
<sup>1</sup>H NMR of **3k** in CDCl<sub>3</sub>



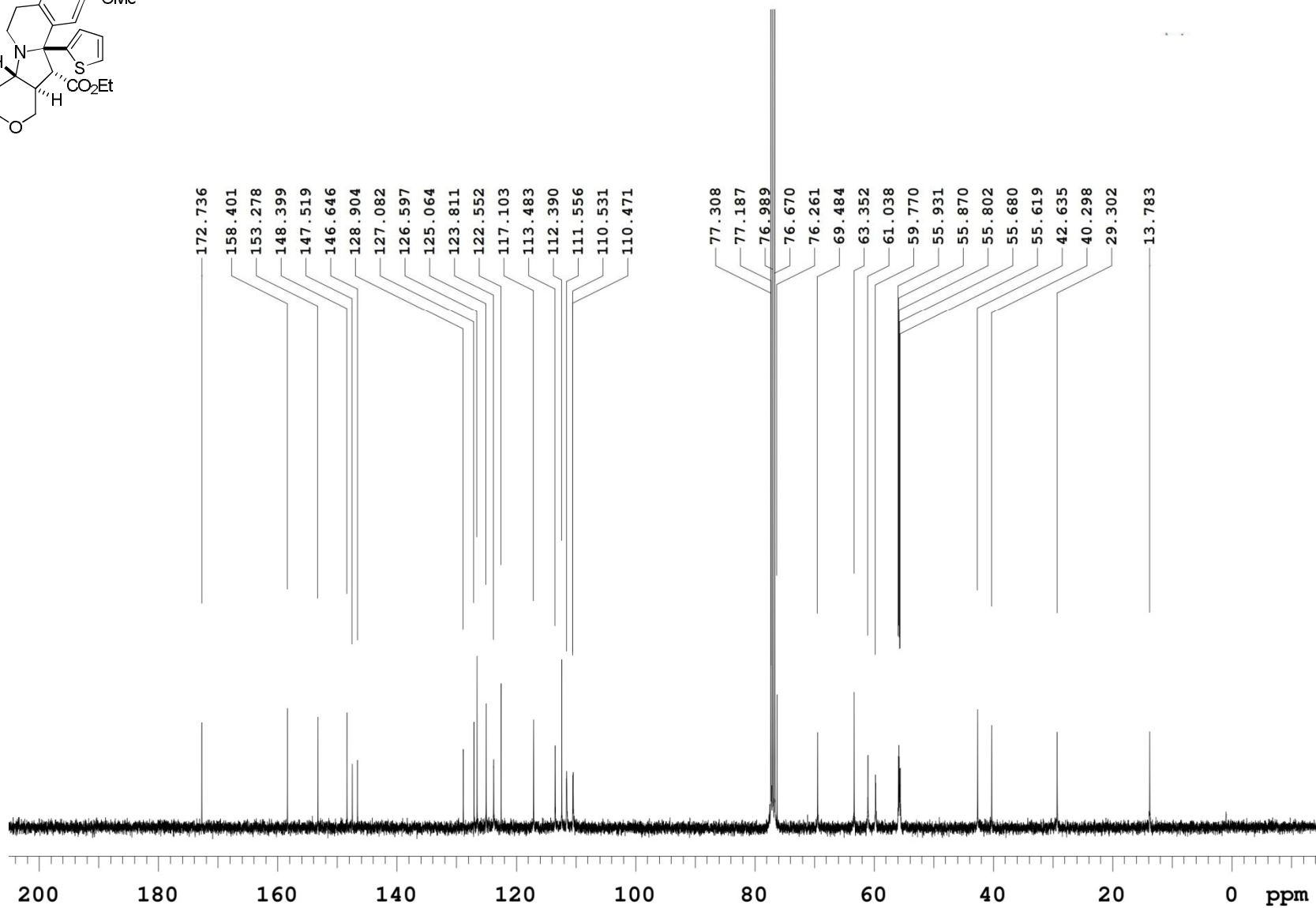
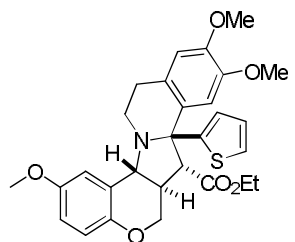
$^{13}\text{C}$  NMR of **3k** in  $\text{CDCl}_3$



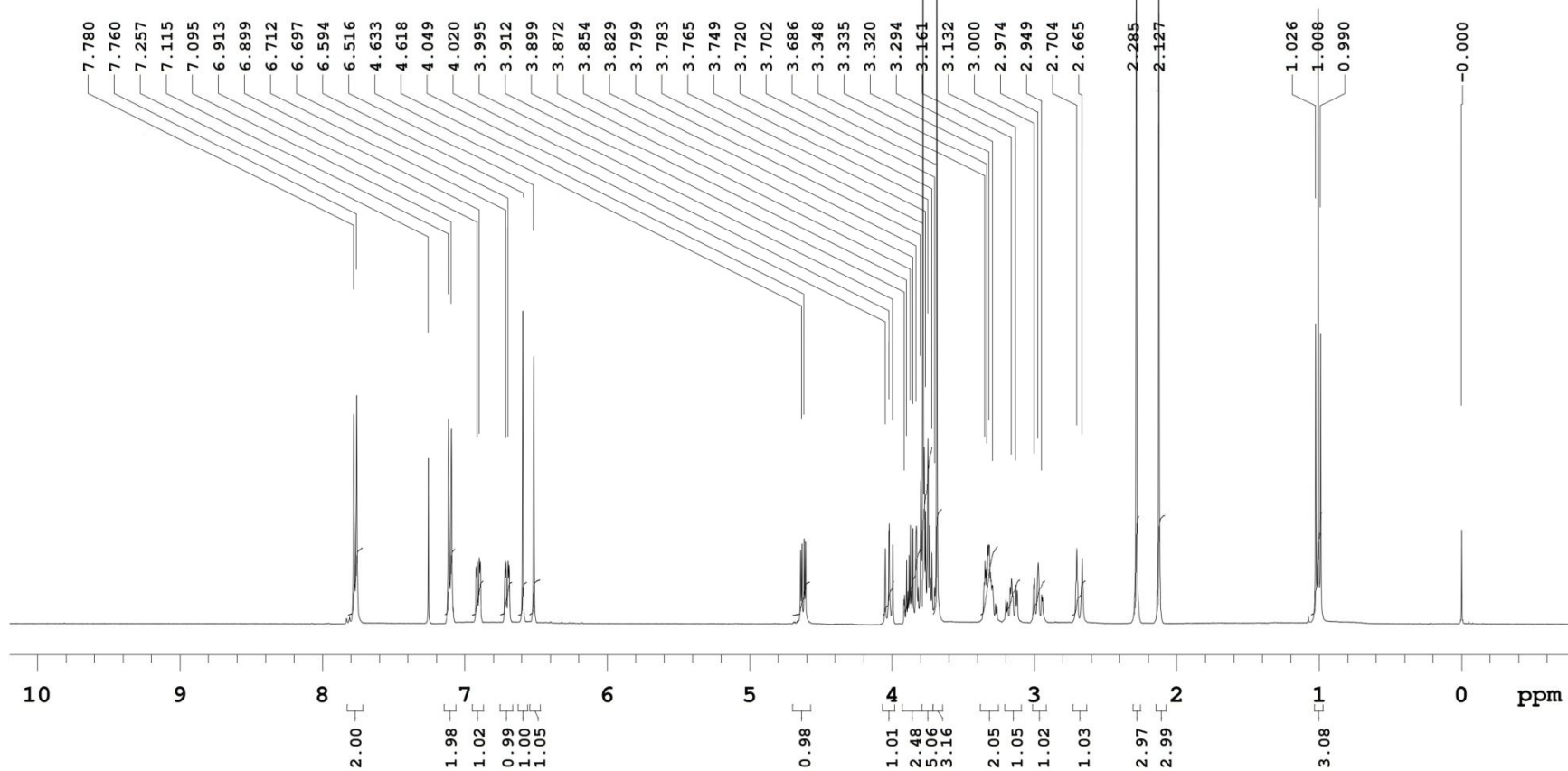
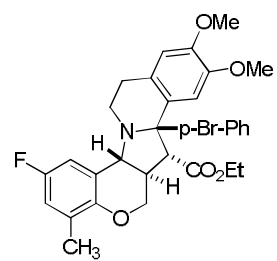
<sup>1</sup>H NMR of **31** in CDCl<sub>3</sub>



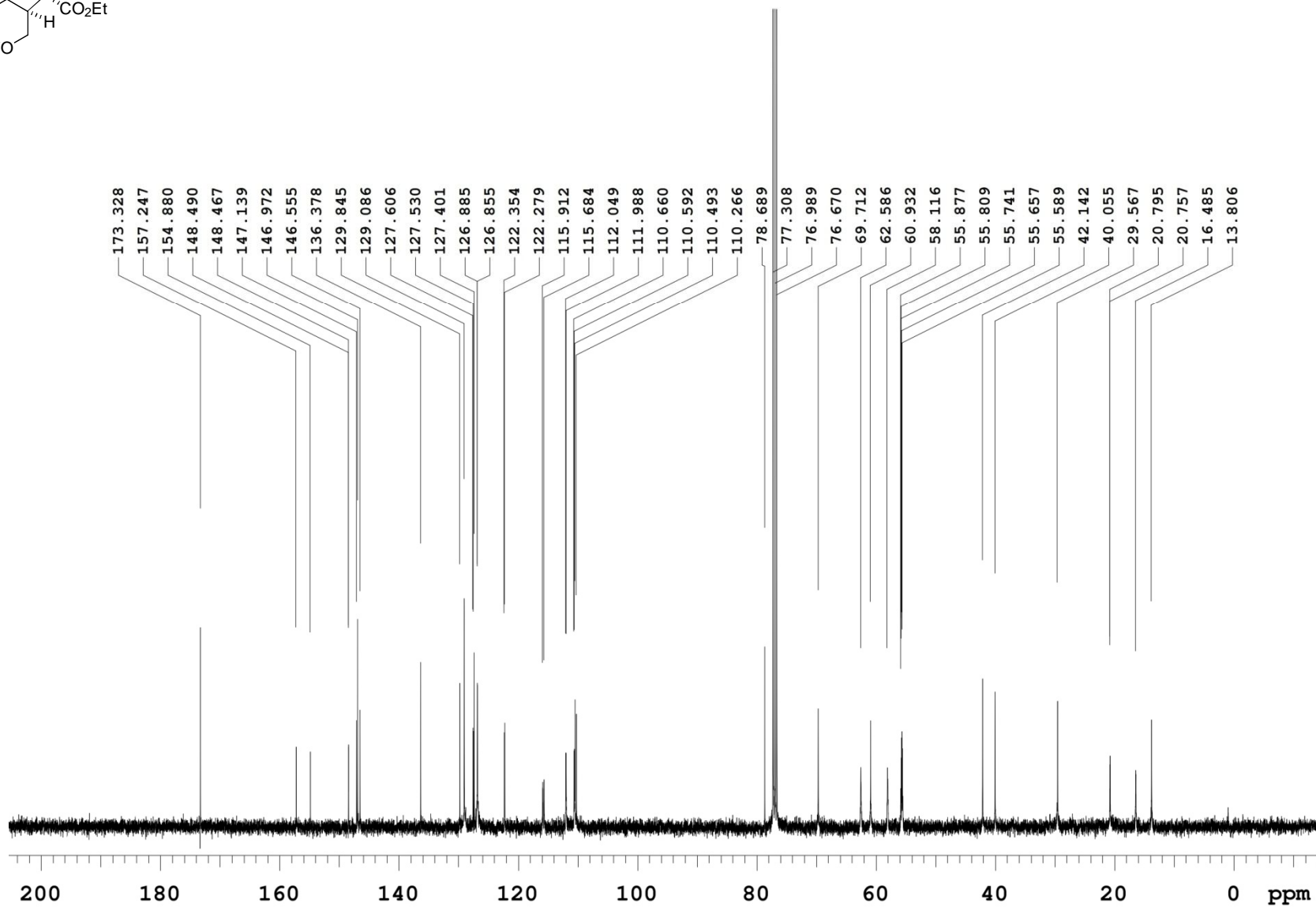
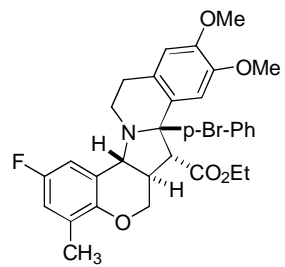
<sup>13</sup>C NMR of **31** in CDCl<sub>3</sub>



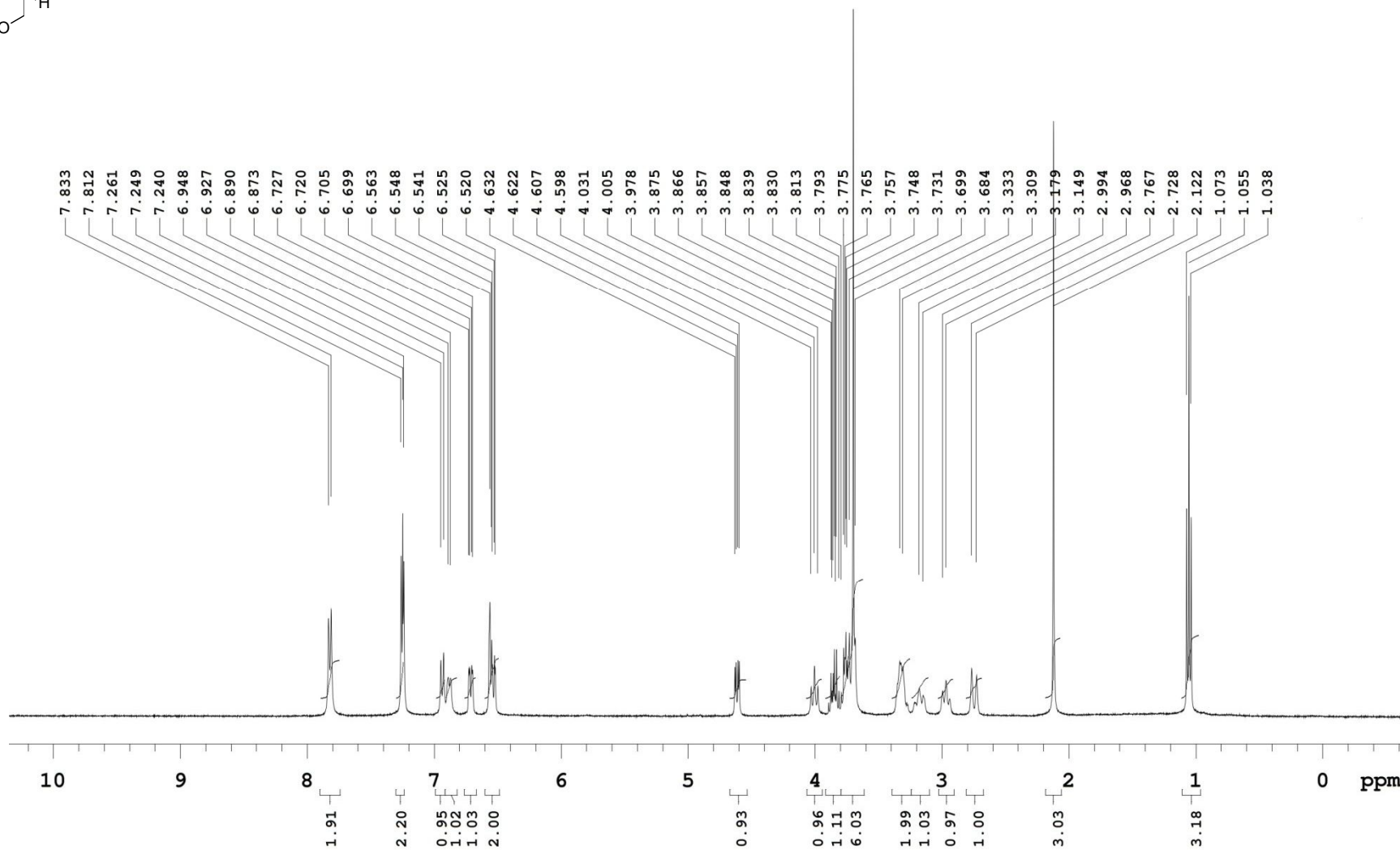
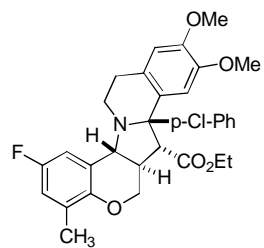
$^{13}\text{C}$  NMR of **3m** in  $\text{CDCl}_3$



<sup>13</sup>C NMR of 3m in CDCl<sub>3</sub>

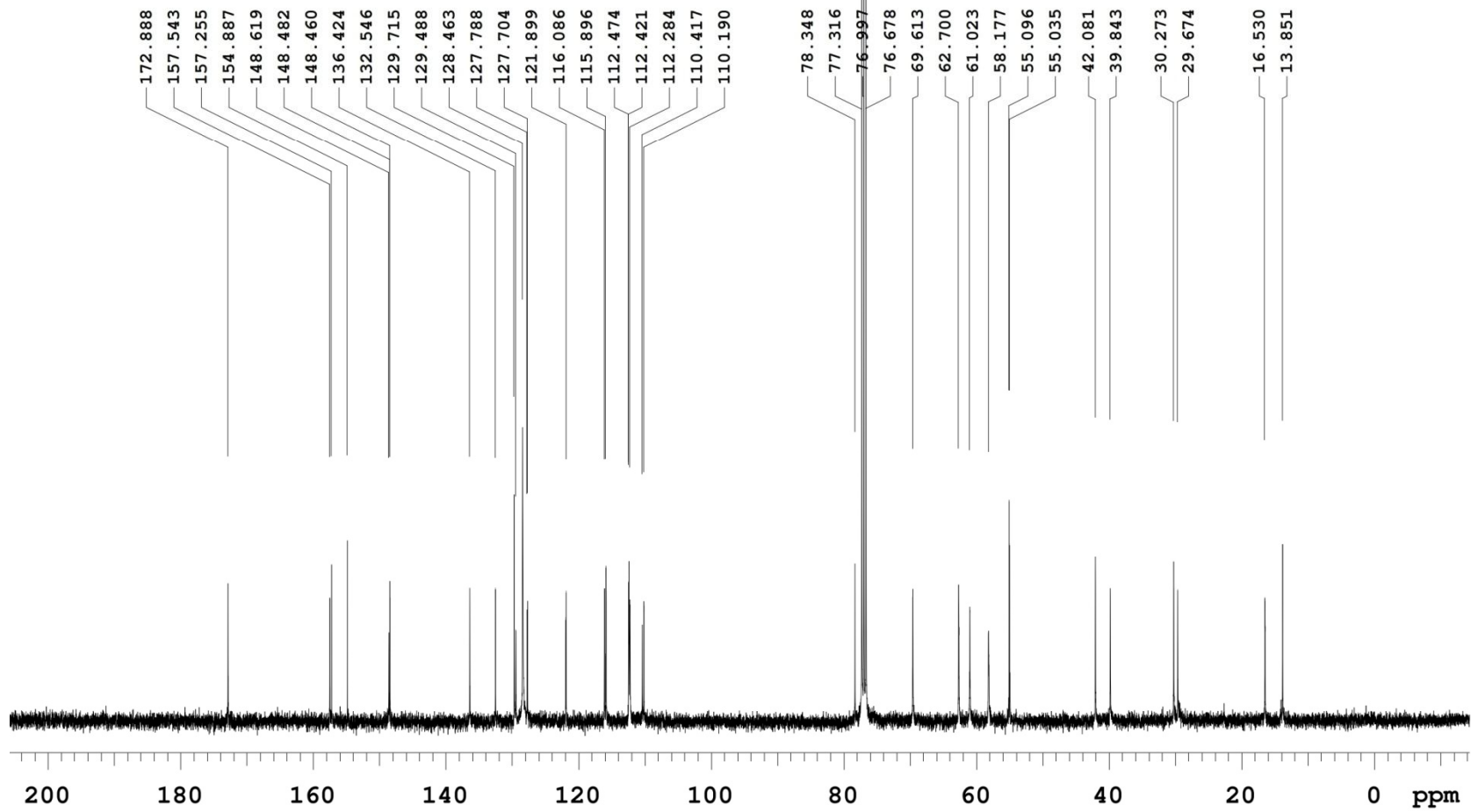
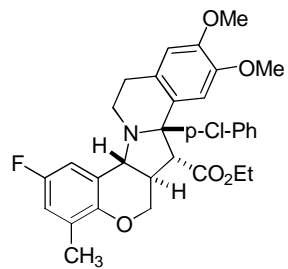


<sup>1</sup>H NMR of **3n** in CDCl<sub>3</sub>

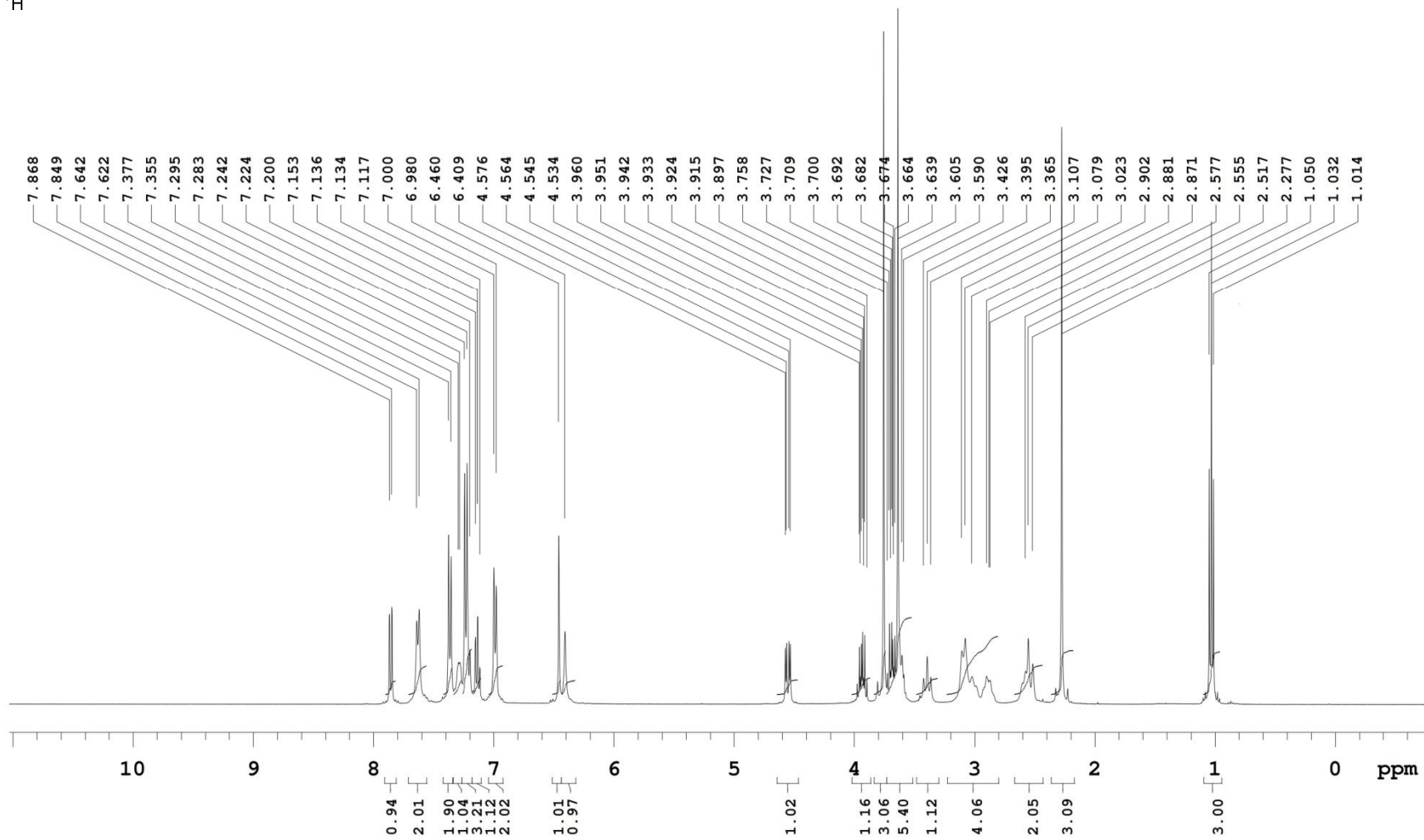
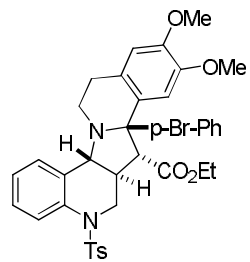




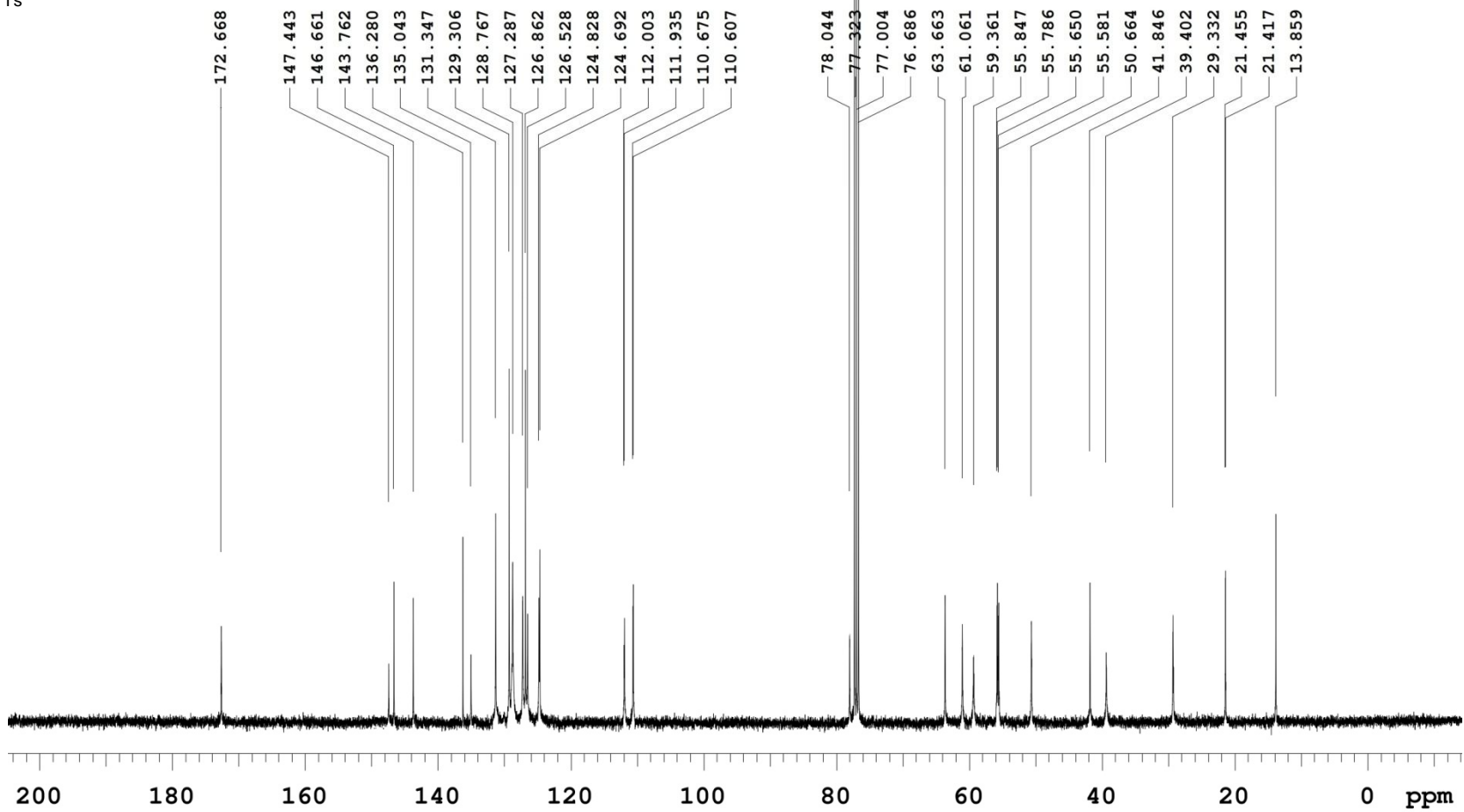
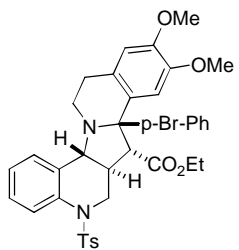
<sup>13</sup>C NMR of **3n** in CDCl<sub>3</sub>



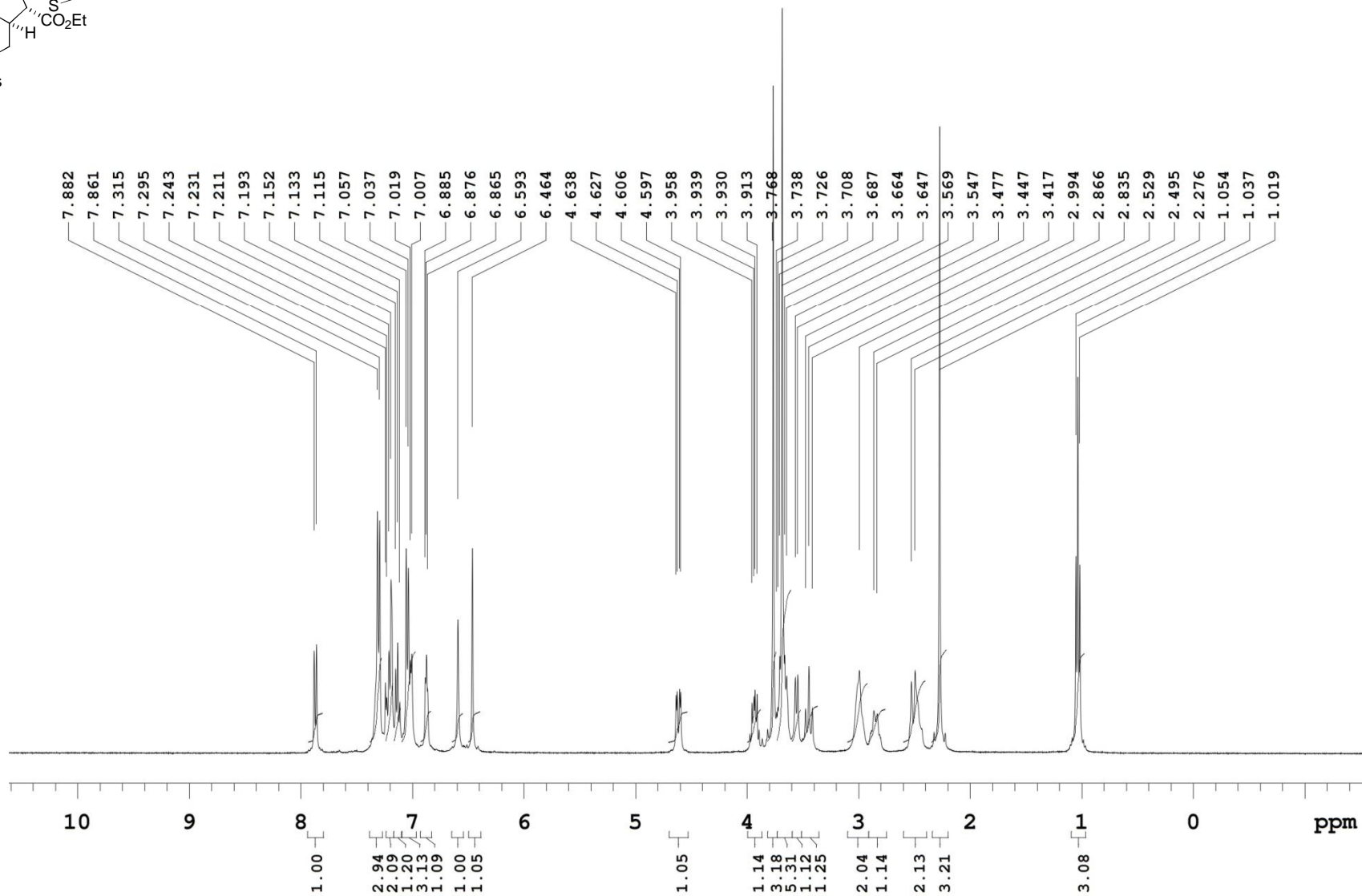
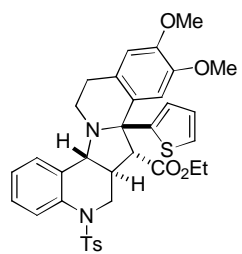
<sup>1</sup>H NMR of **3o** in CDCl<sub>3</sub>



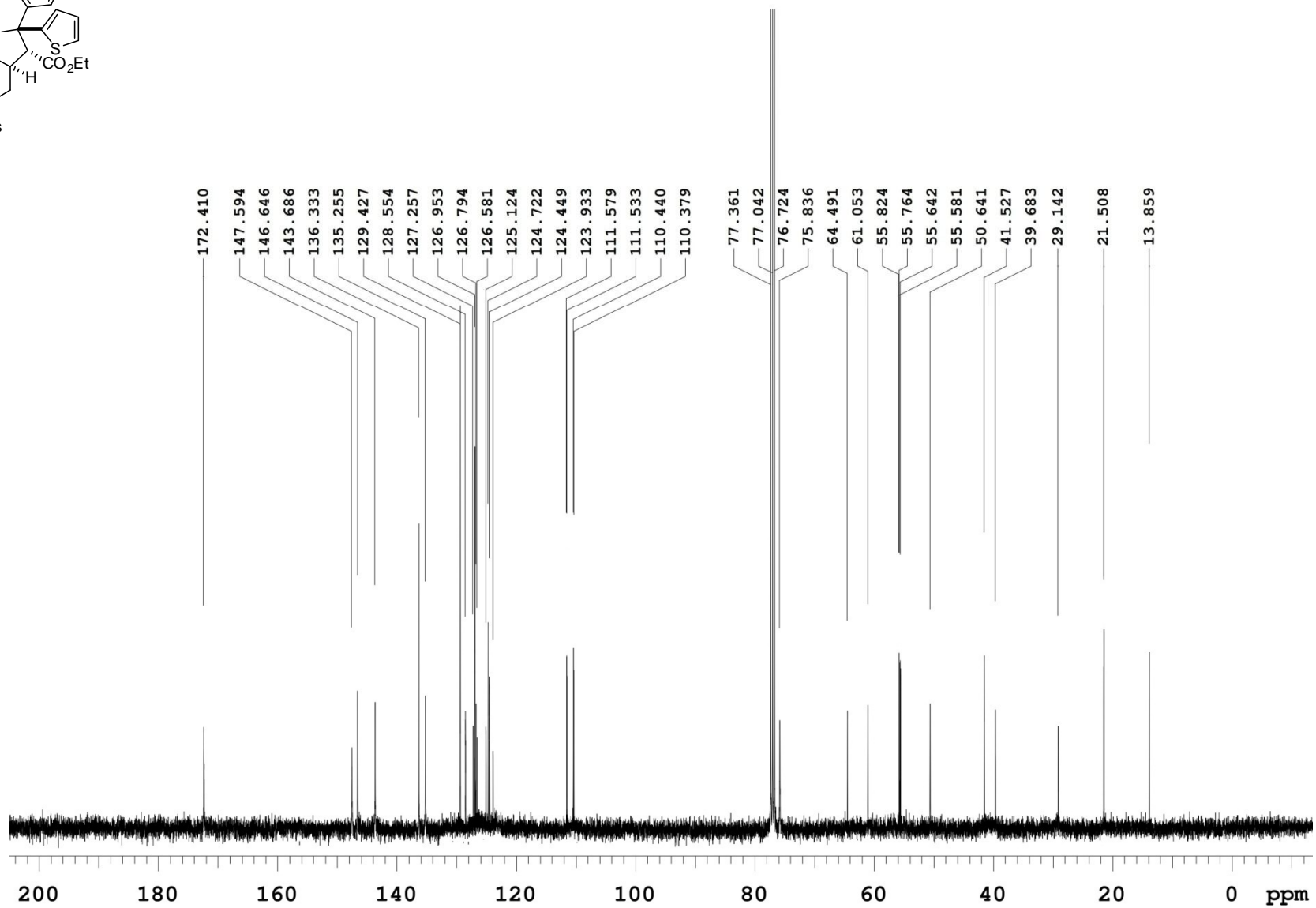
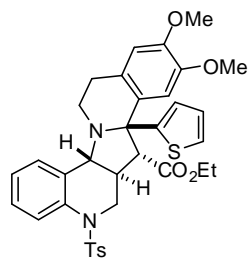
<sup>13</sup>C NMR of **3o** in CDCl<sub>3</sub>



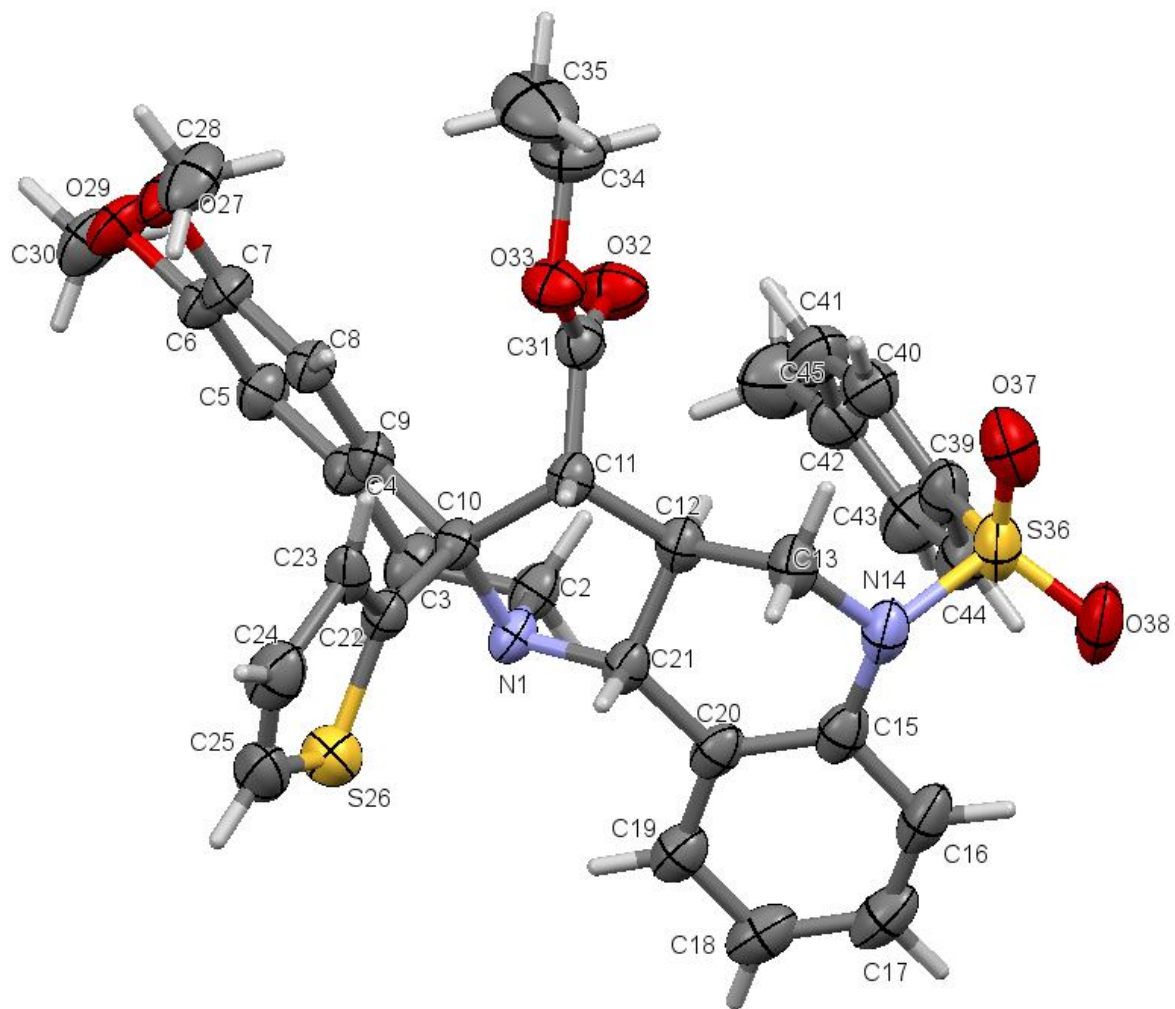
$^1\text{H}$  NMR of 3p in  $\text{CDCl}_3$



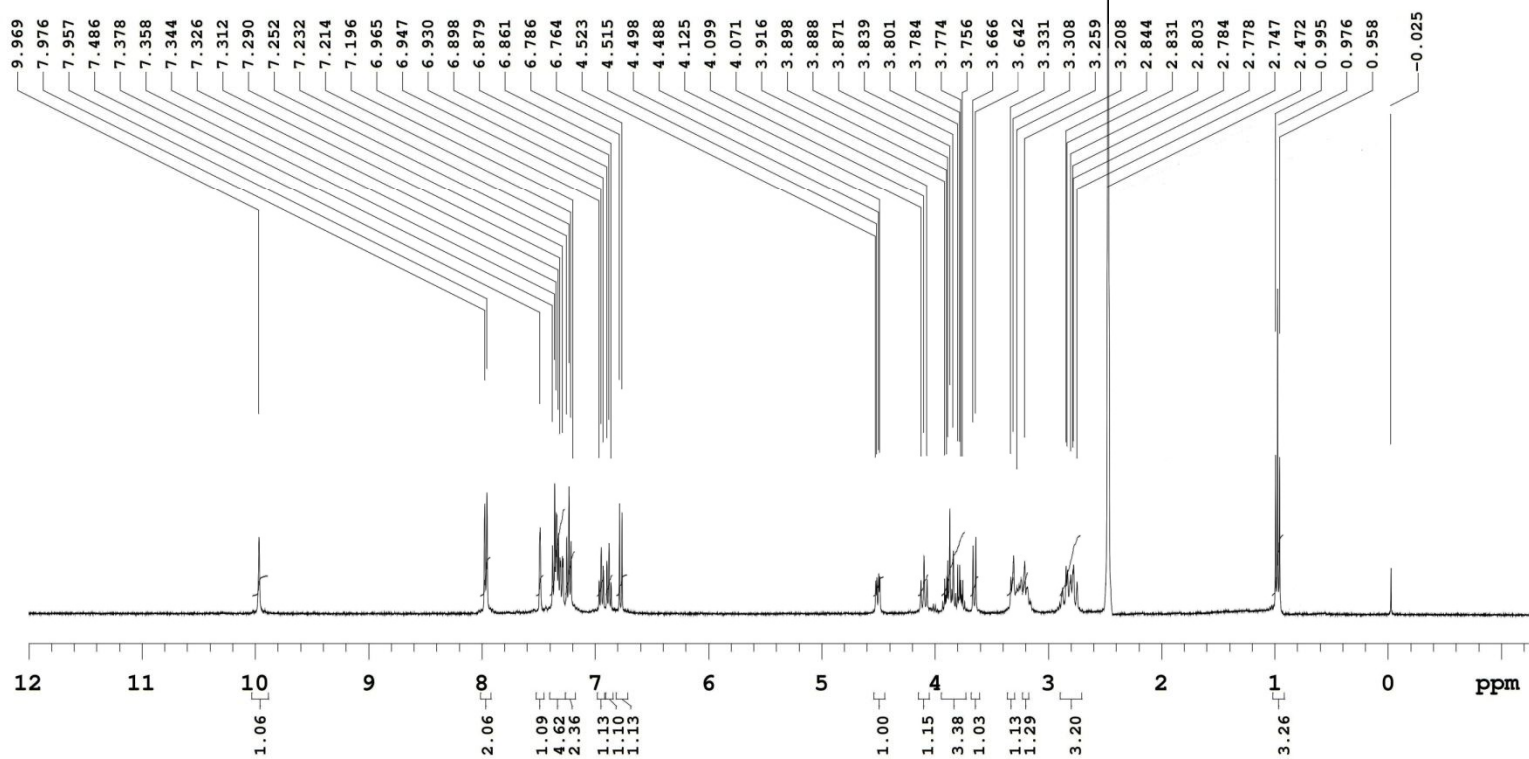
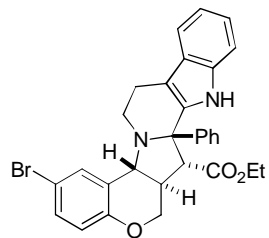
$^{13}\text{C}$  NMR of **3p** in  $\text{CDCl}_3$



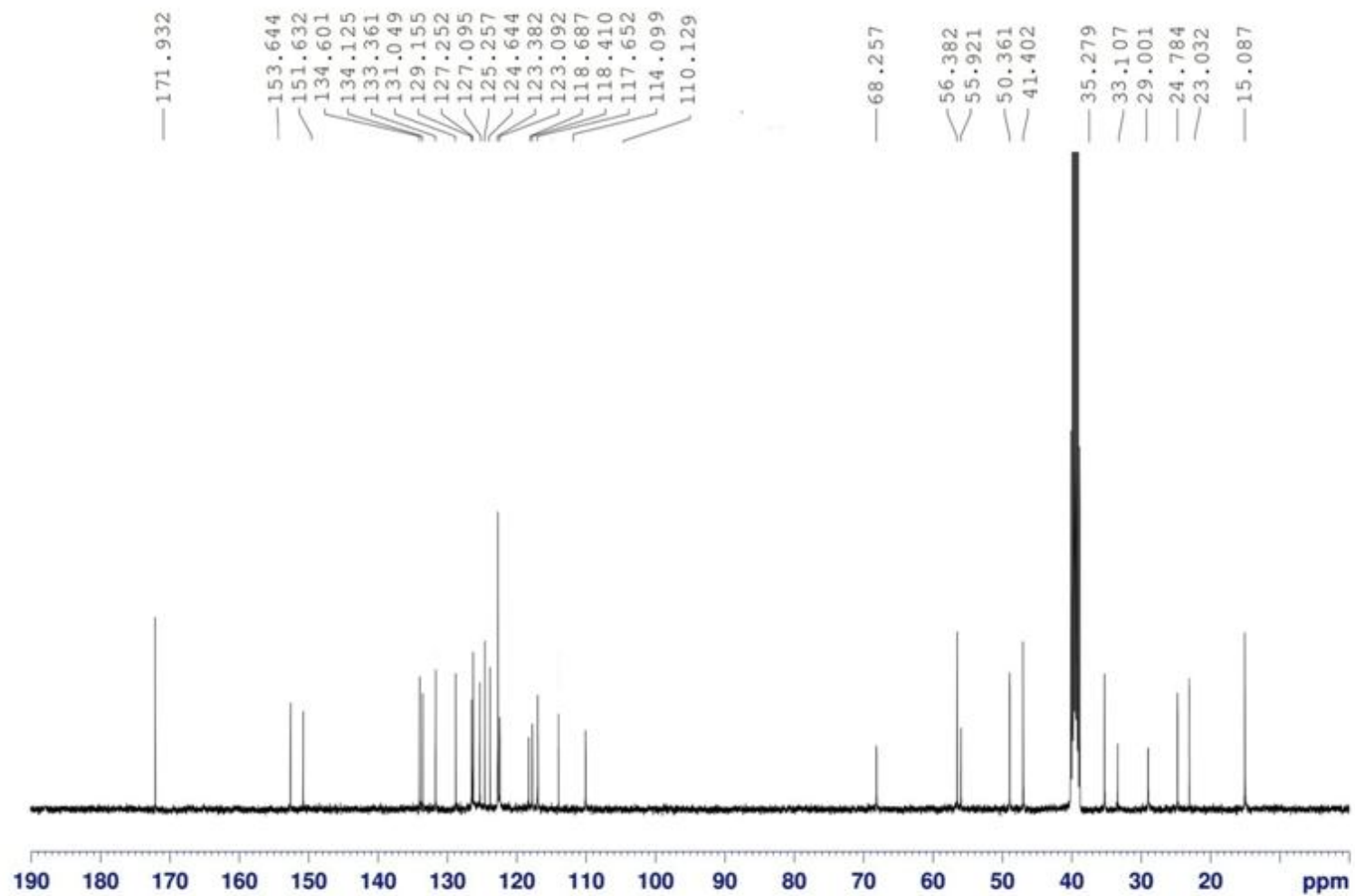
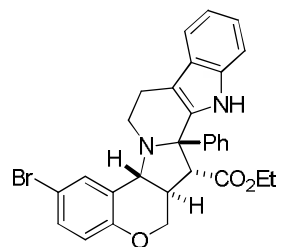
ORTEP Diagram Of 3p



<sup>1</sup>H NMR of **4a** in DMSO-d<sub>6</sub>

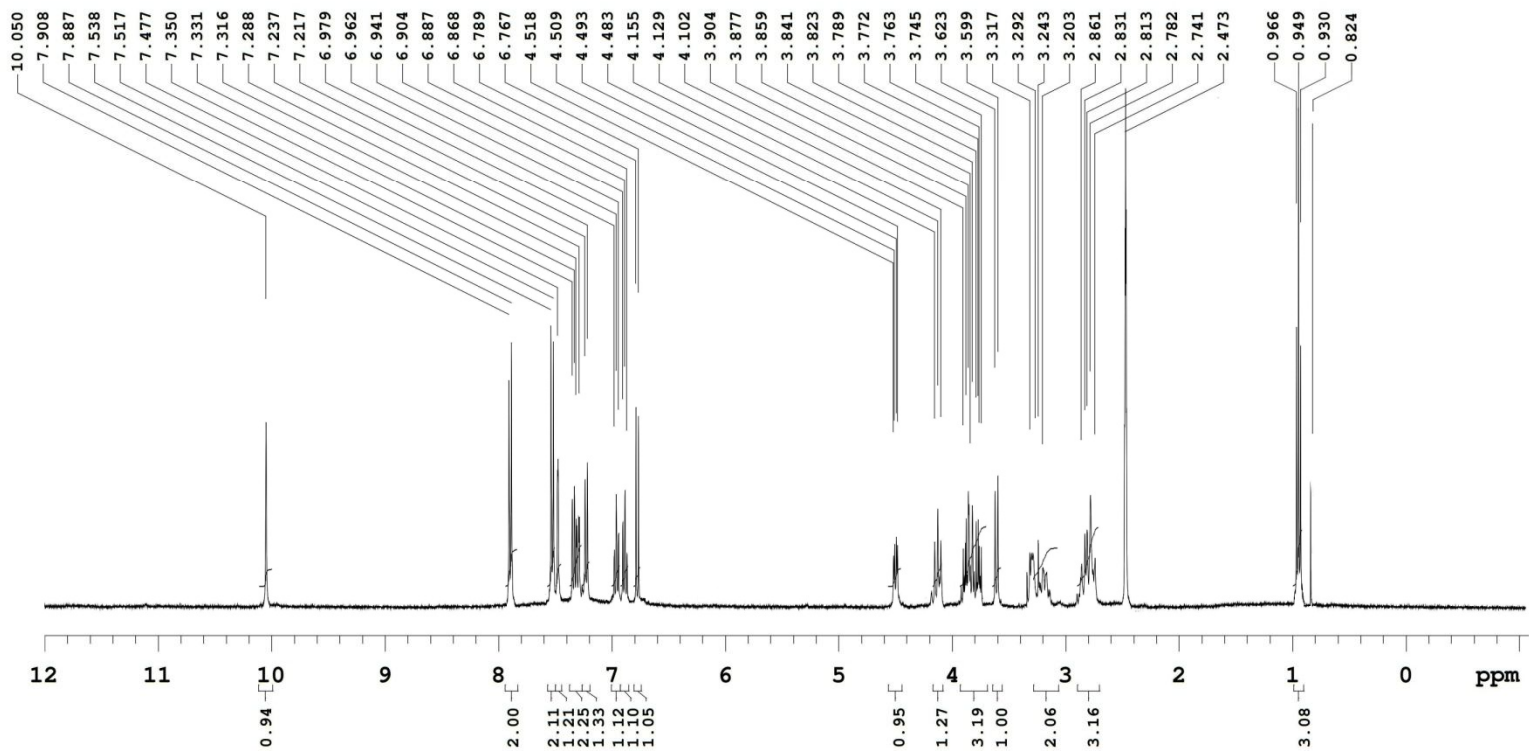
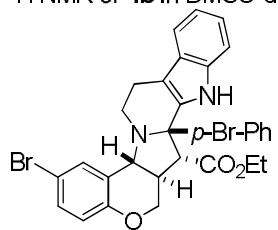


<sup>13</sup>C NMR of **4a** in DMSO-d<sub>6</sub>

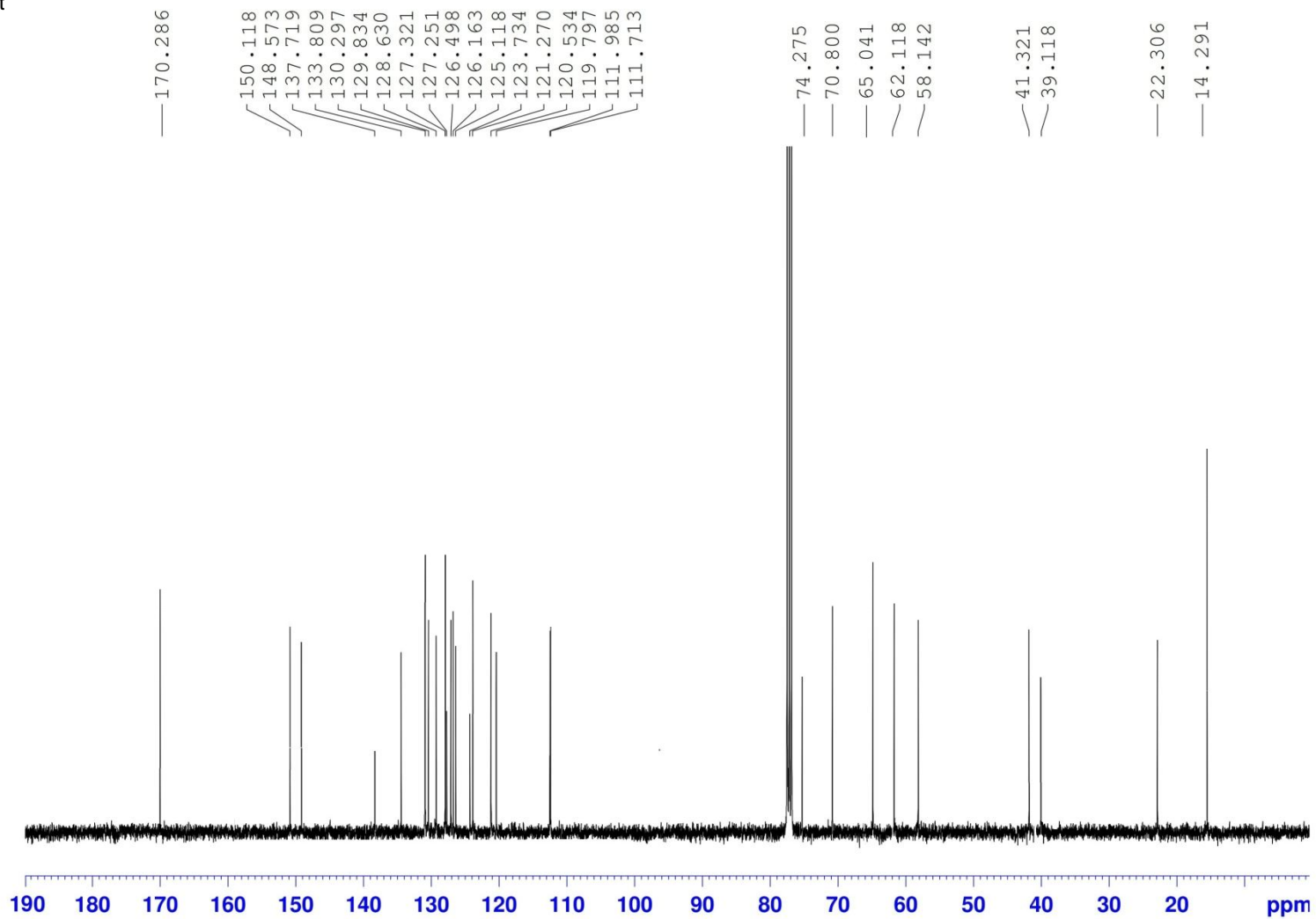
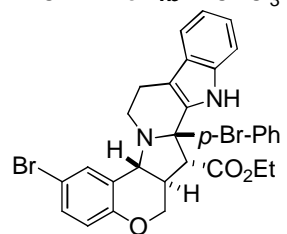




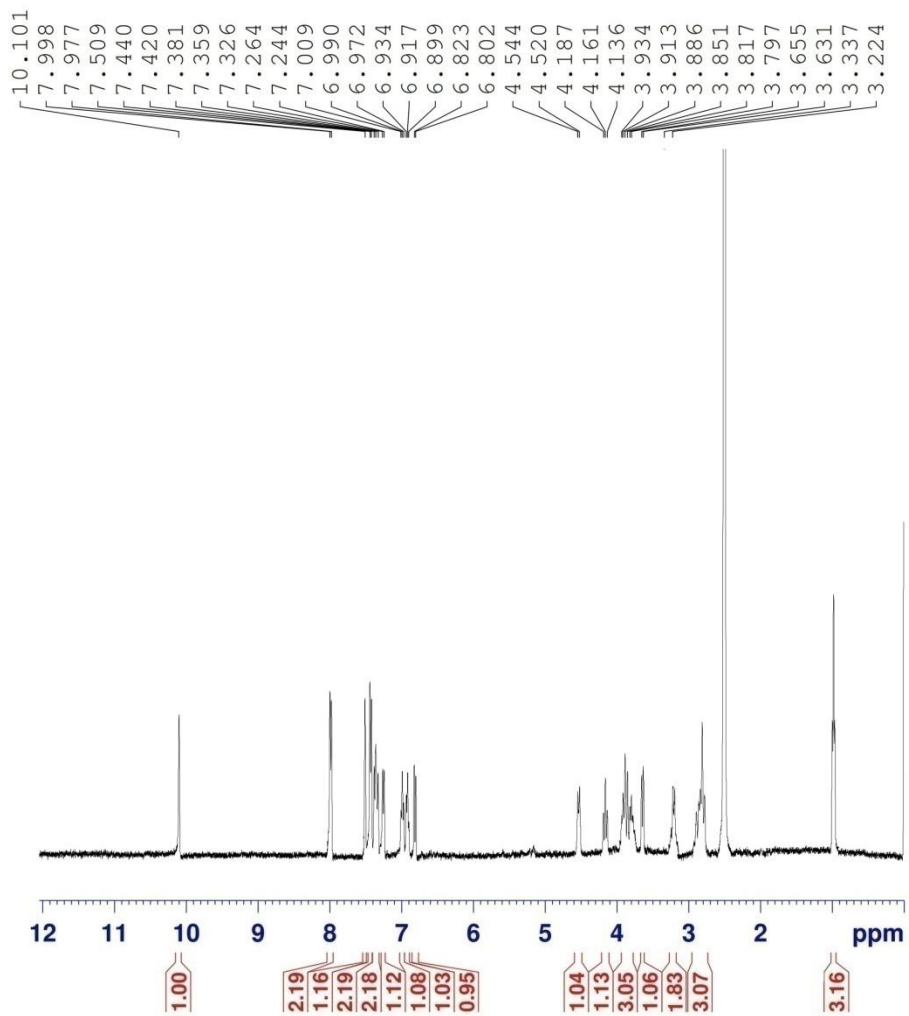
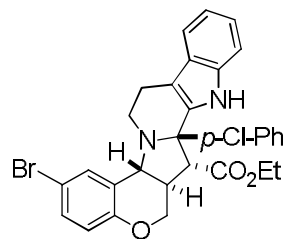
<sup>1</sup>H NMR of **4b** in DMSO-d<sub>6</sub>



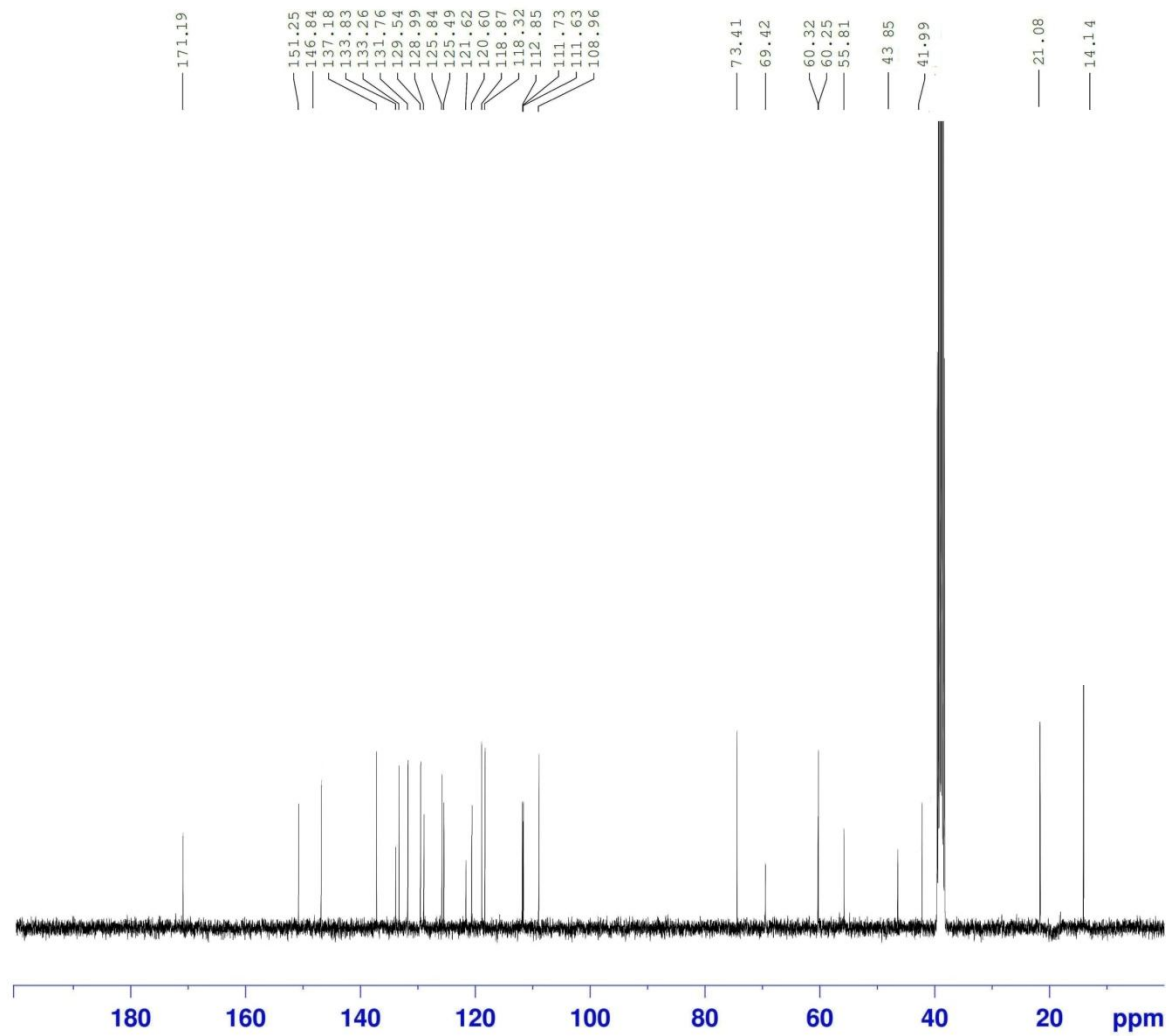
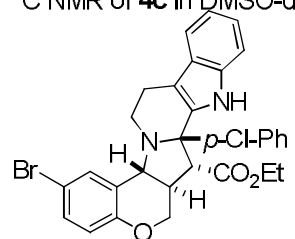
$^{13}\text{C}$  NMR of **4b** in  $\text{CDCl}_3$



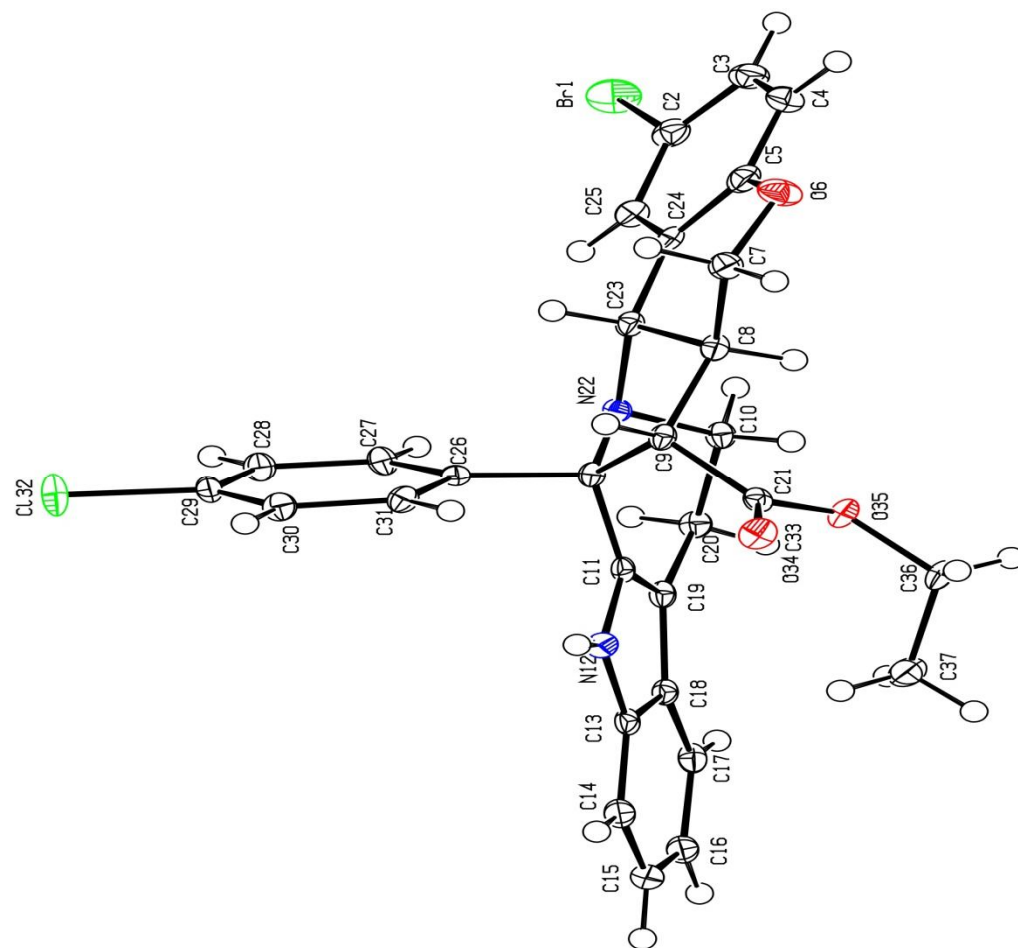
$^1\text{H}$  NMR of **4c** in  $\text{DMSO-d}_6$



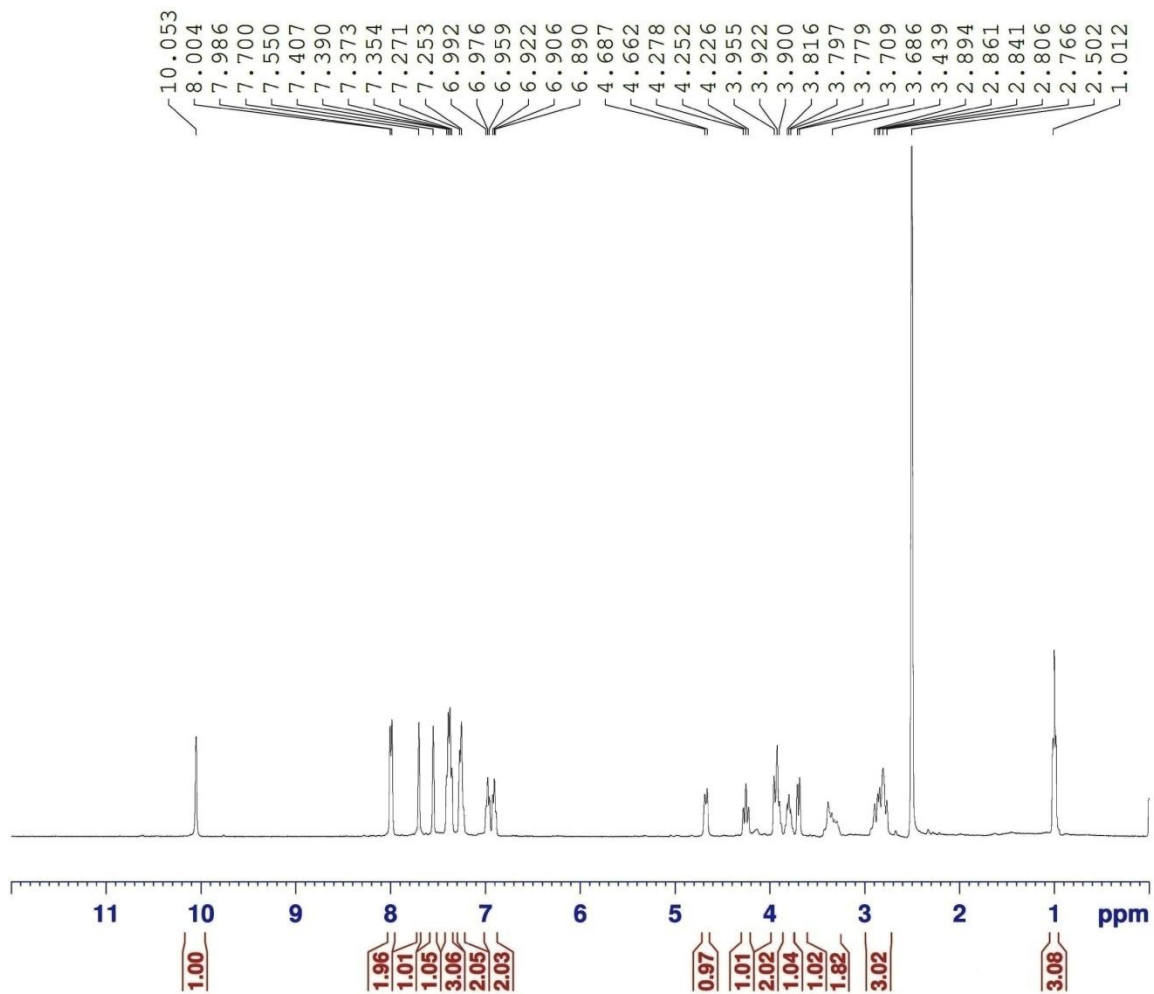
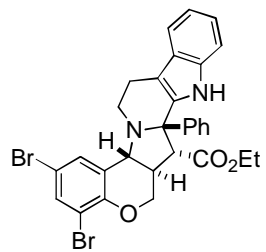
$^{13}\text{C}$  NMR of **4c** in  $\text{DMSO-d}_6$



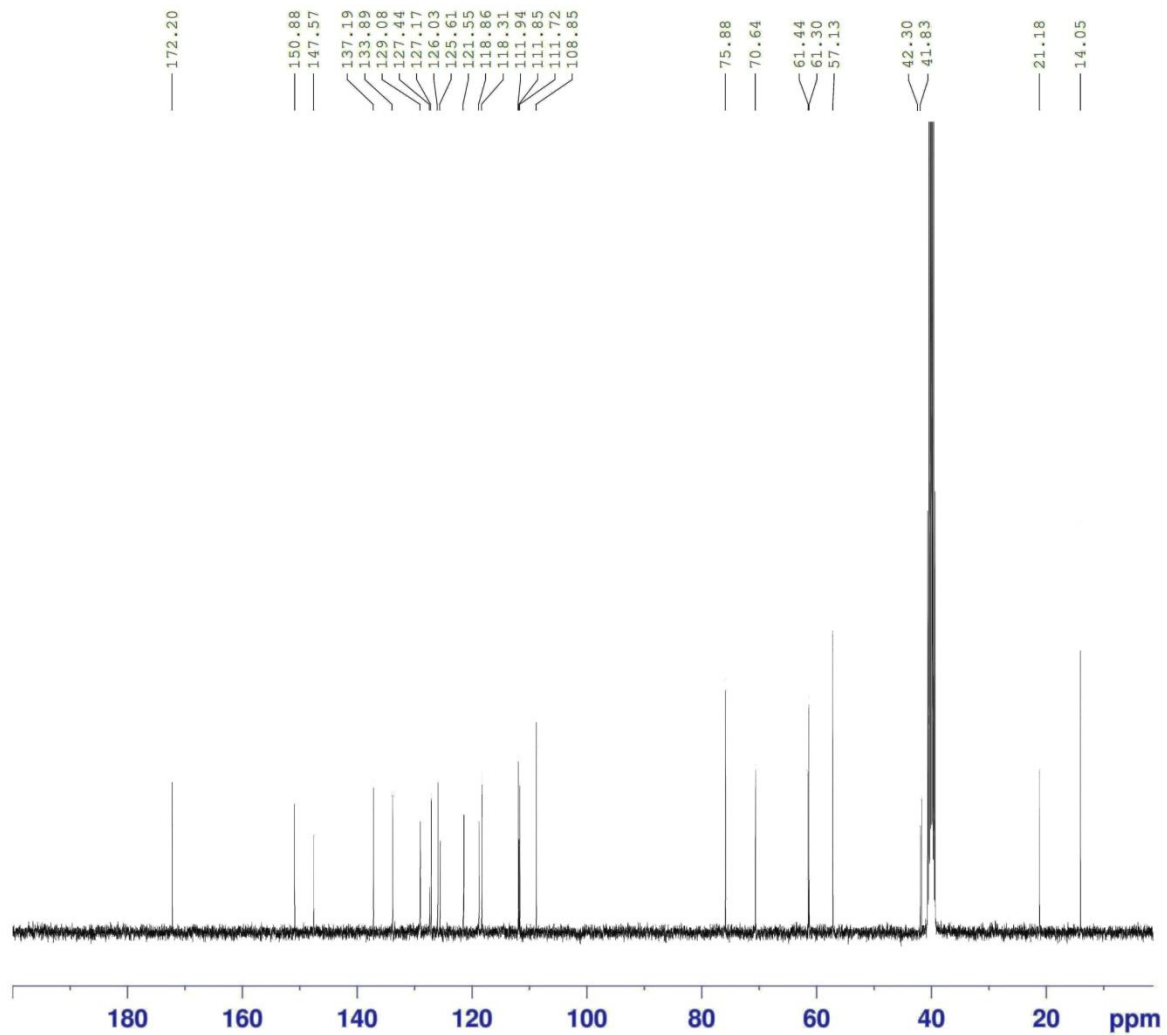
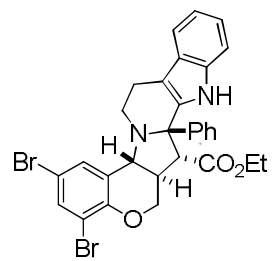
ORTEP diagram of **4c**



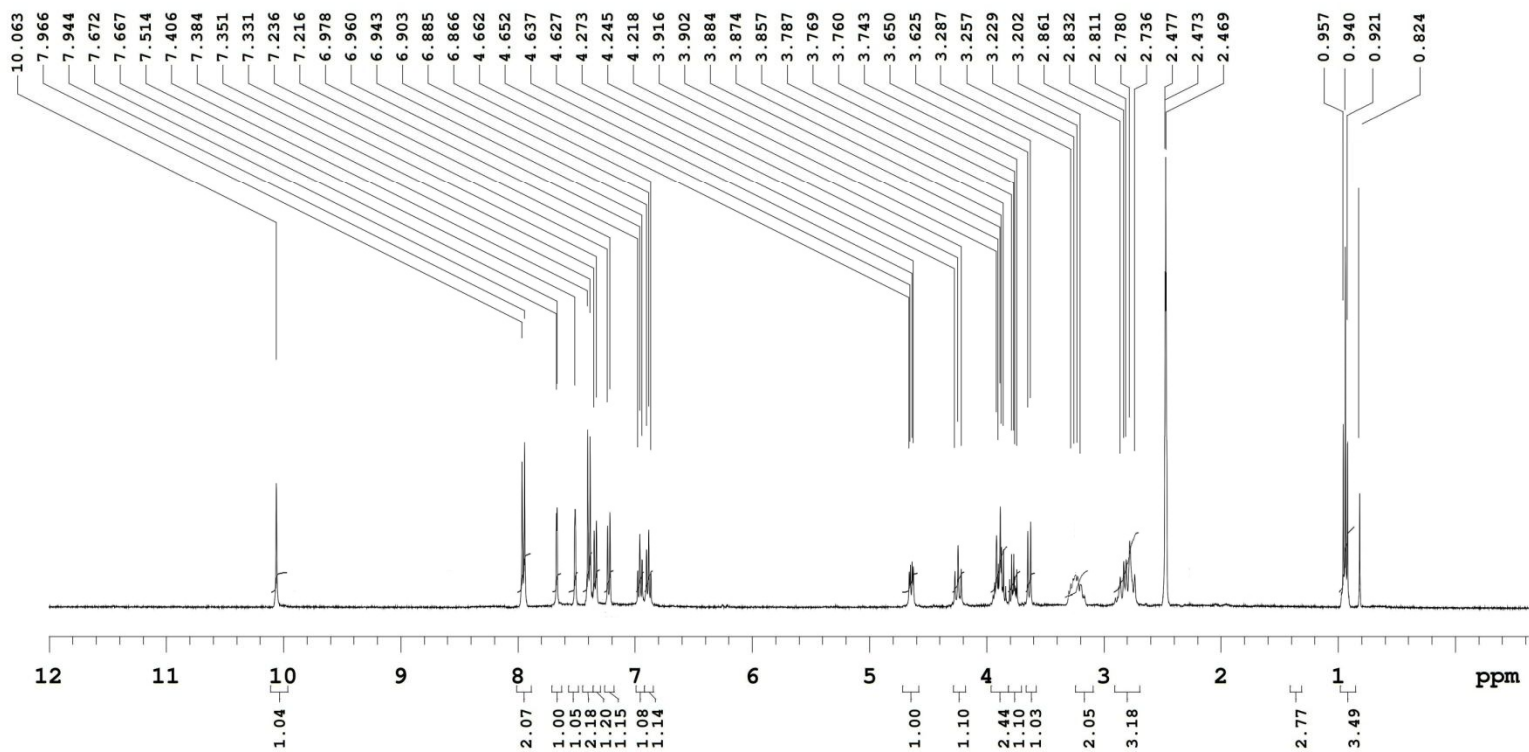
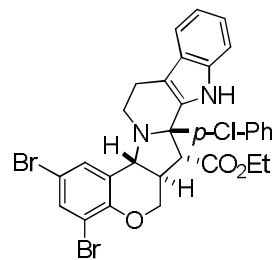
<sup>1</sup>H NMR of **4d** in DMSO-d<sub>6</sub>



$^{13}\text{C}$  NMR of **4d** in  $\text{DMSO-d}_6$

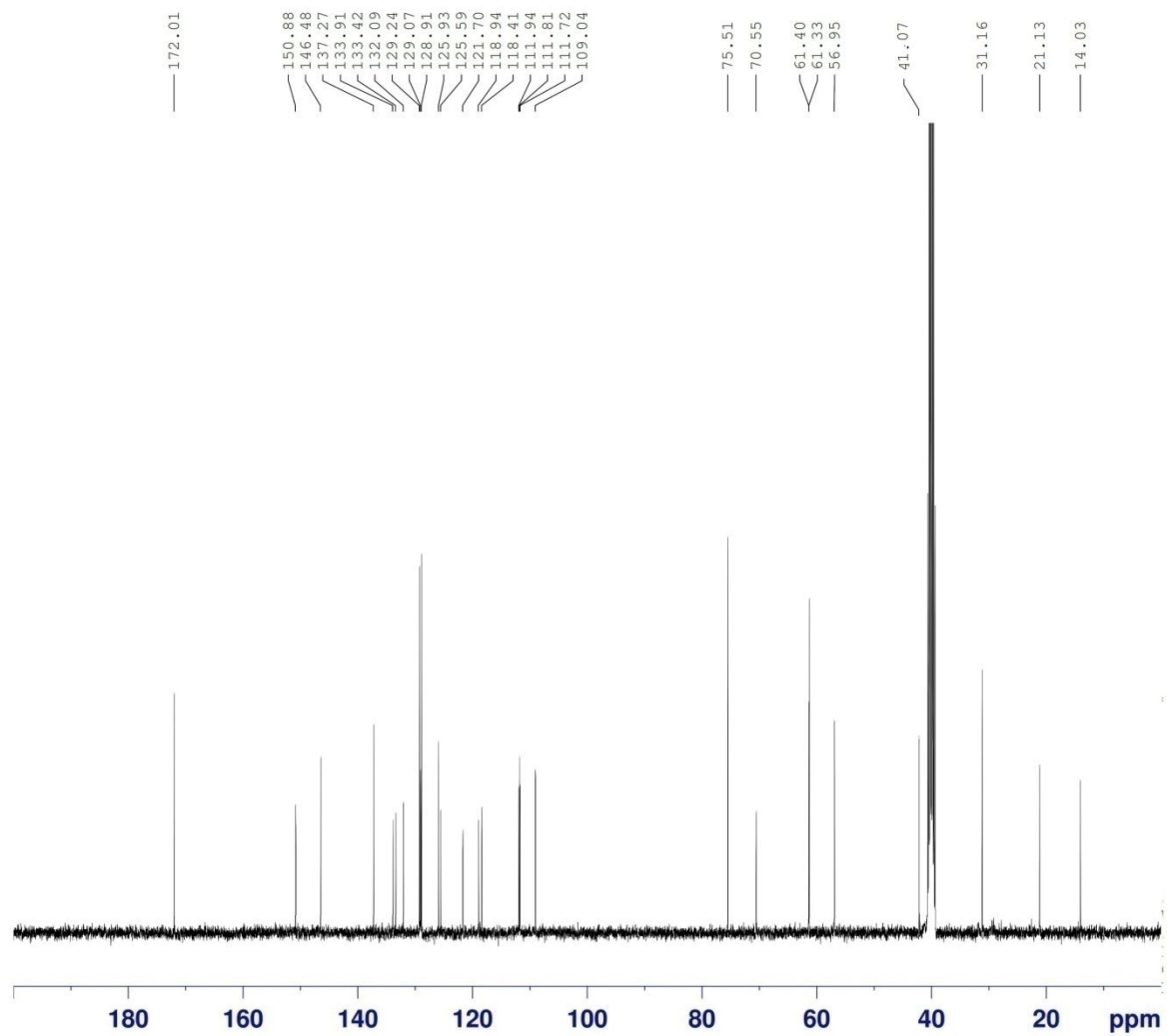
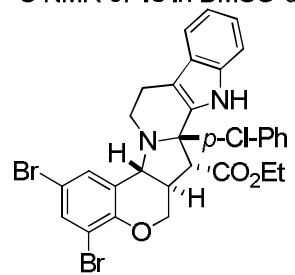


$^1\text{H}$  NMR of **4e** in  $\text{DMSO-d}_6$

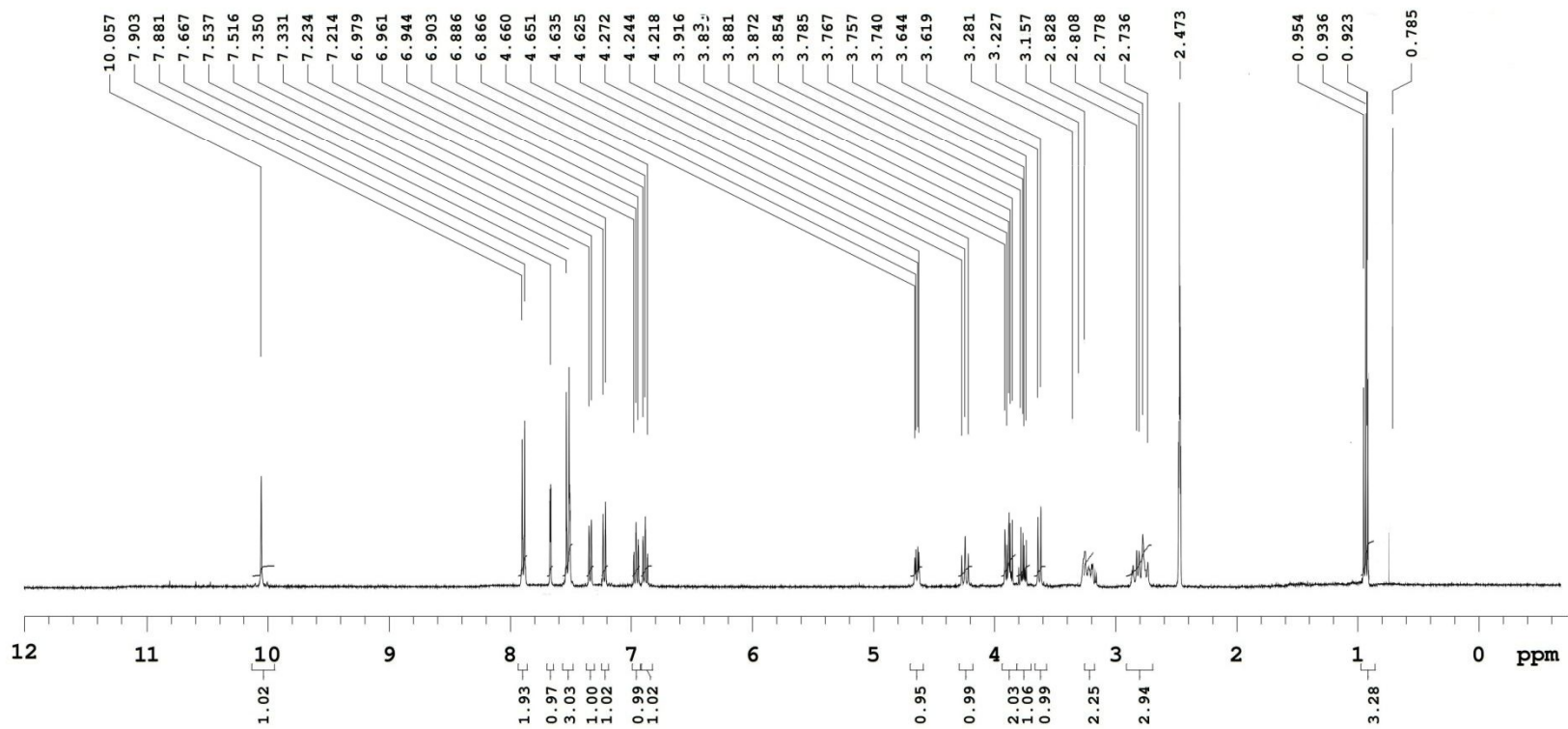
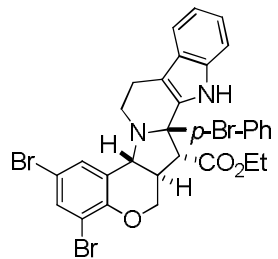




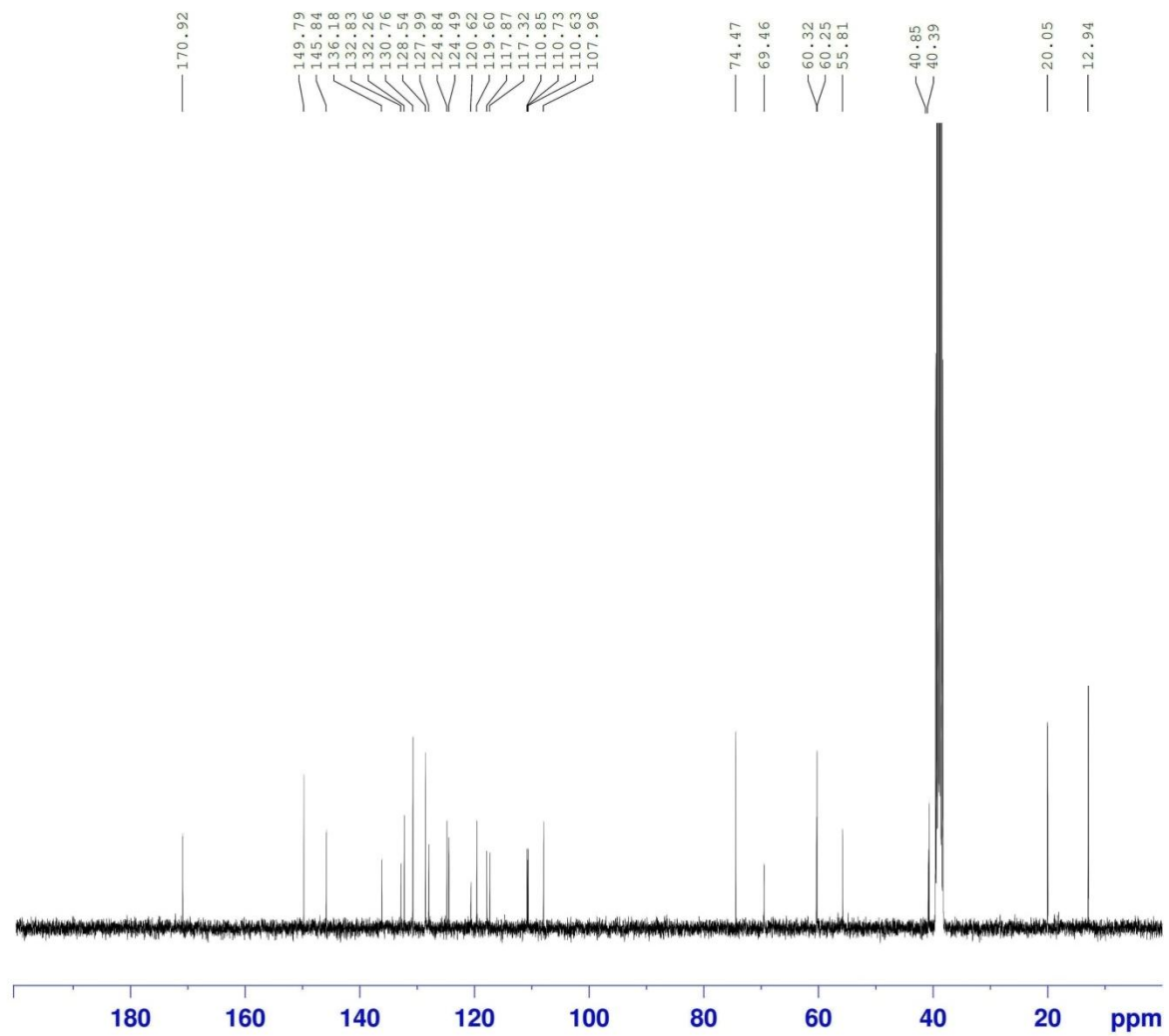
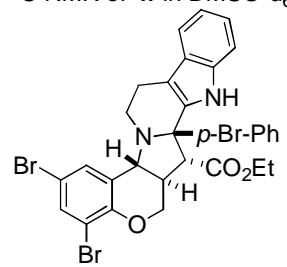
<sup>13</sup>C NMR of **4e** in DMSO-d<sub>6</sub>



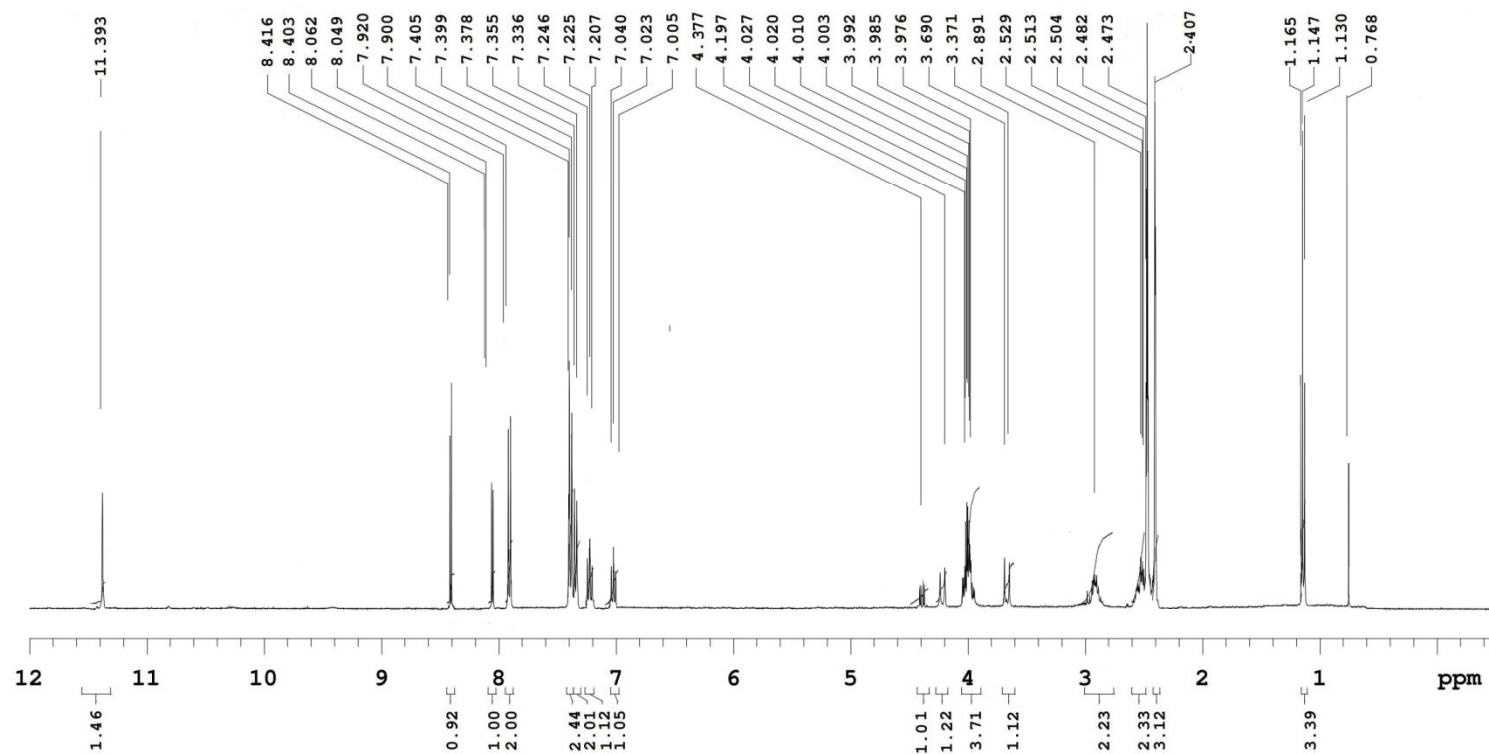
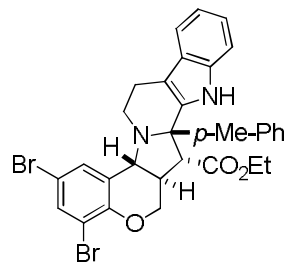
$^1\text{H}$  NMR of **4f** in  $\text{DMSO-d}_6$



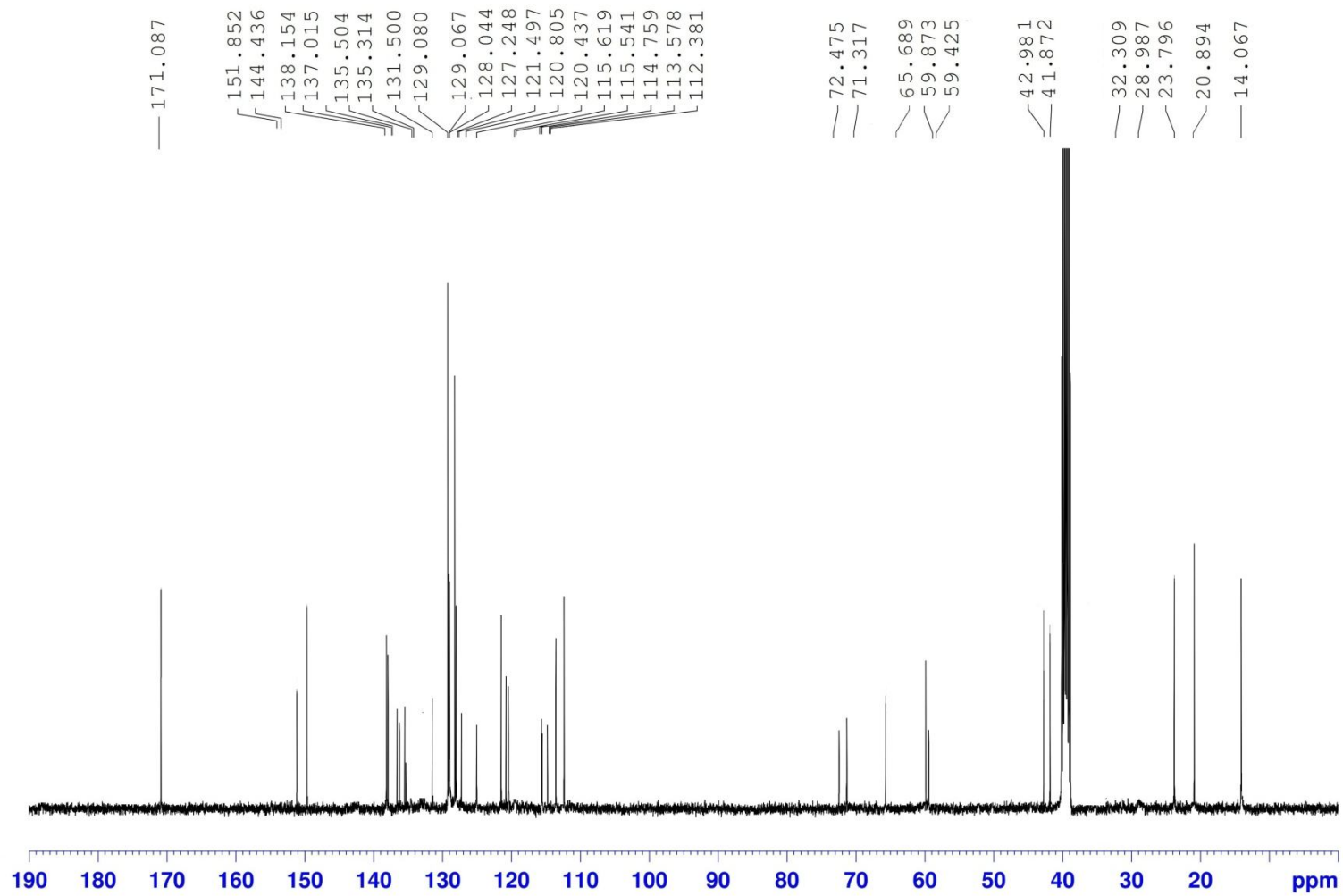
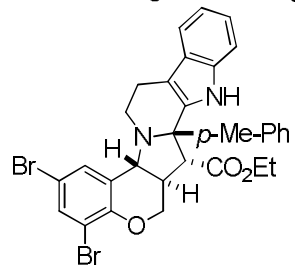
<sup>13</sup>C NMR of **4f** in DMSO-d<sub>6</sub>



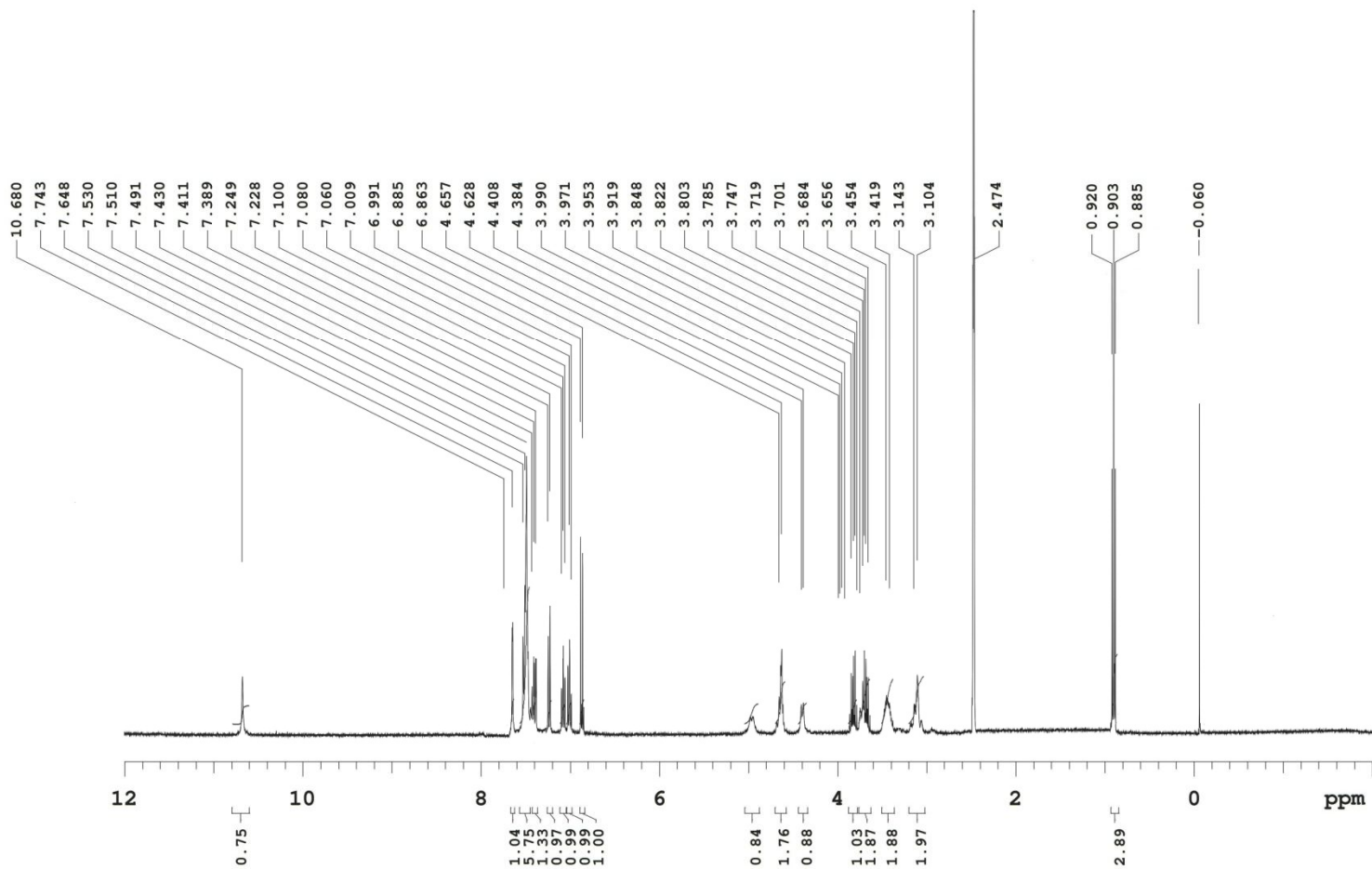
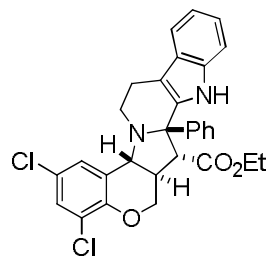
<sup>1</sup>H NMR of **4g** in DMSO-d<sub>6</sub>



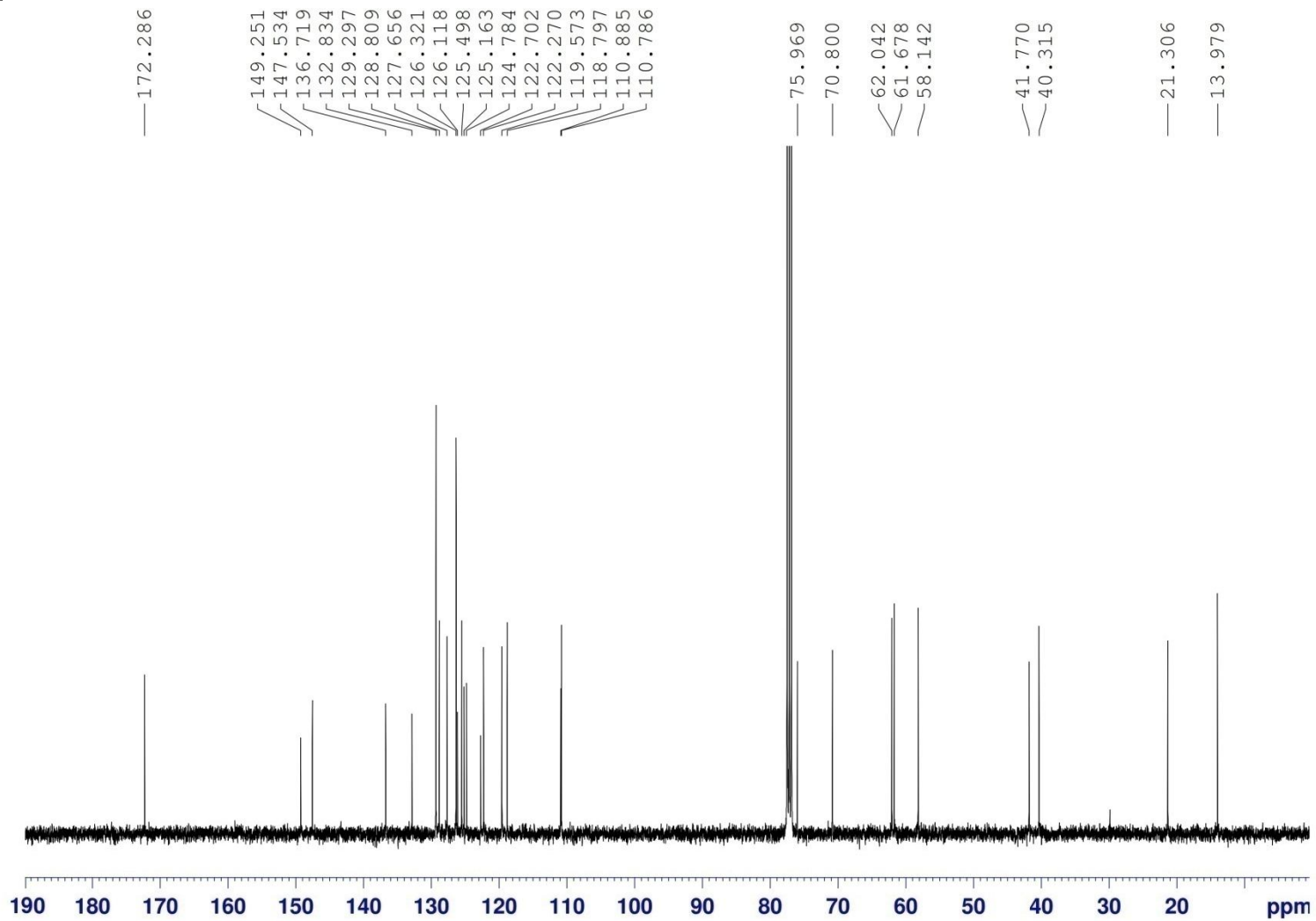
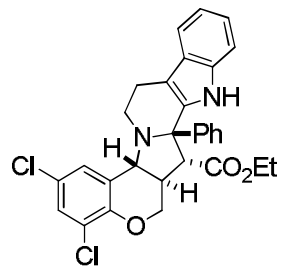
<sup>13</sup>C NMR of **4g** in DMSO-d<sub>6</sub>



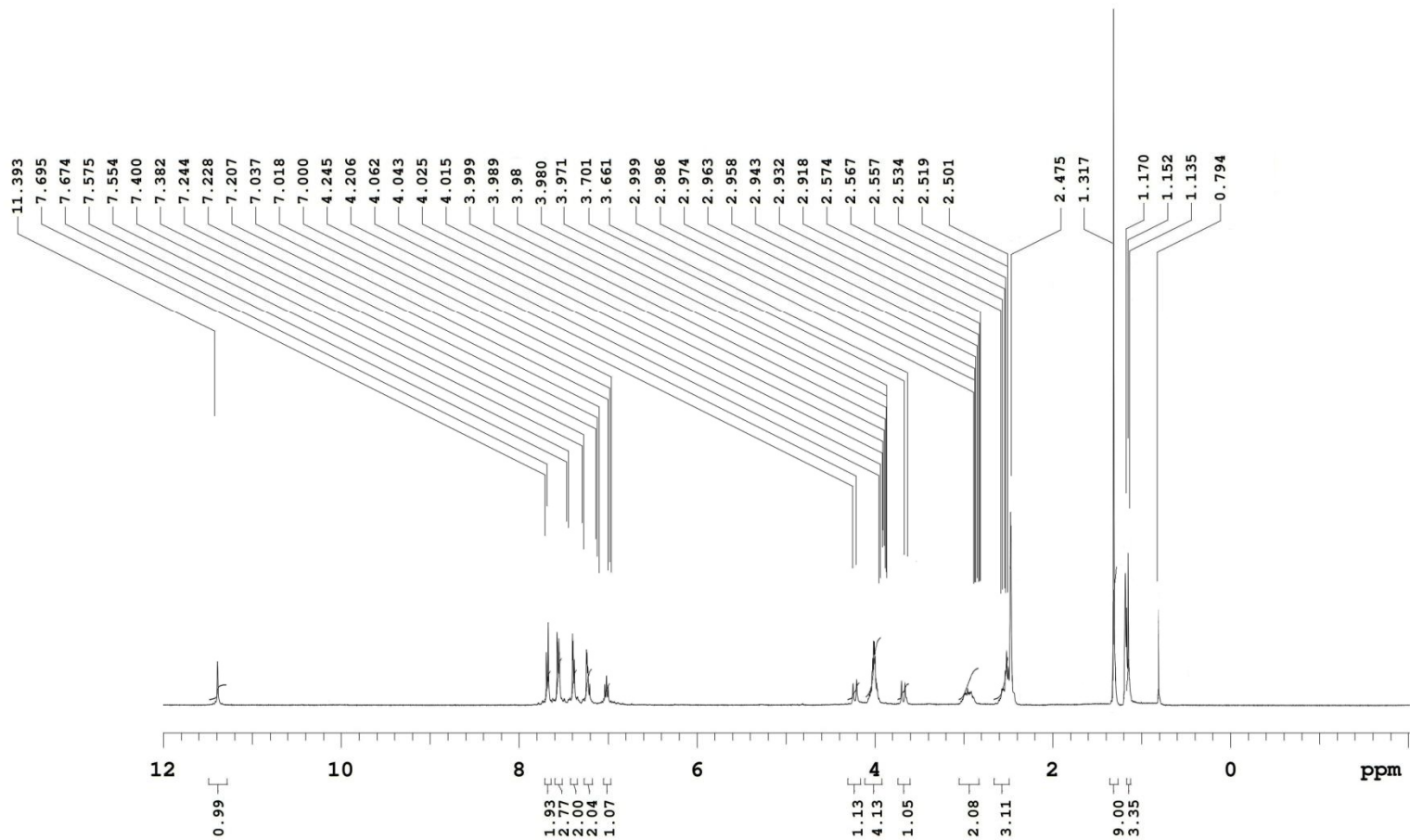
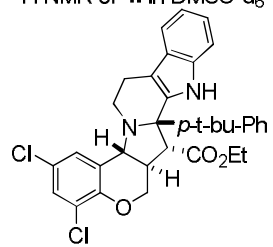
<sup>1</sup>H NMR of **4h** in DMSO-d<sub>6</sub>



<sup>13</sup>C NMR of 4h in CDCl<sub>3</sub>

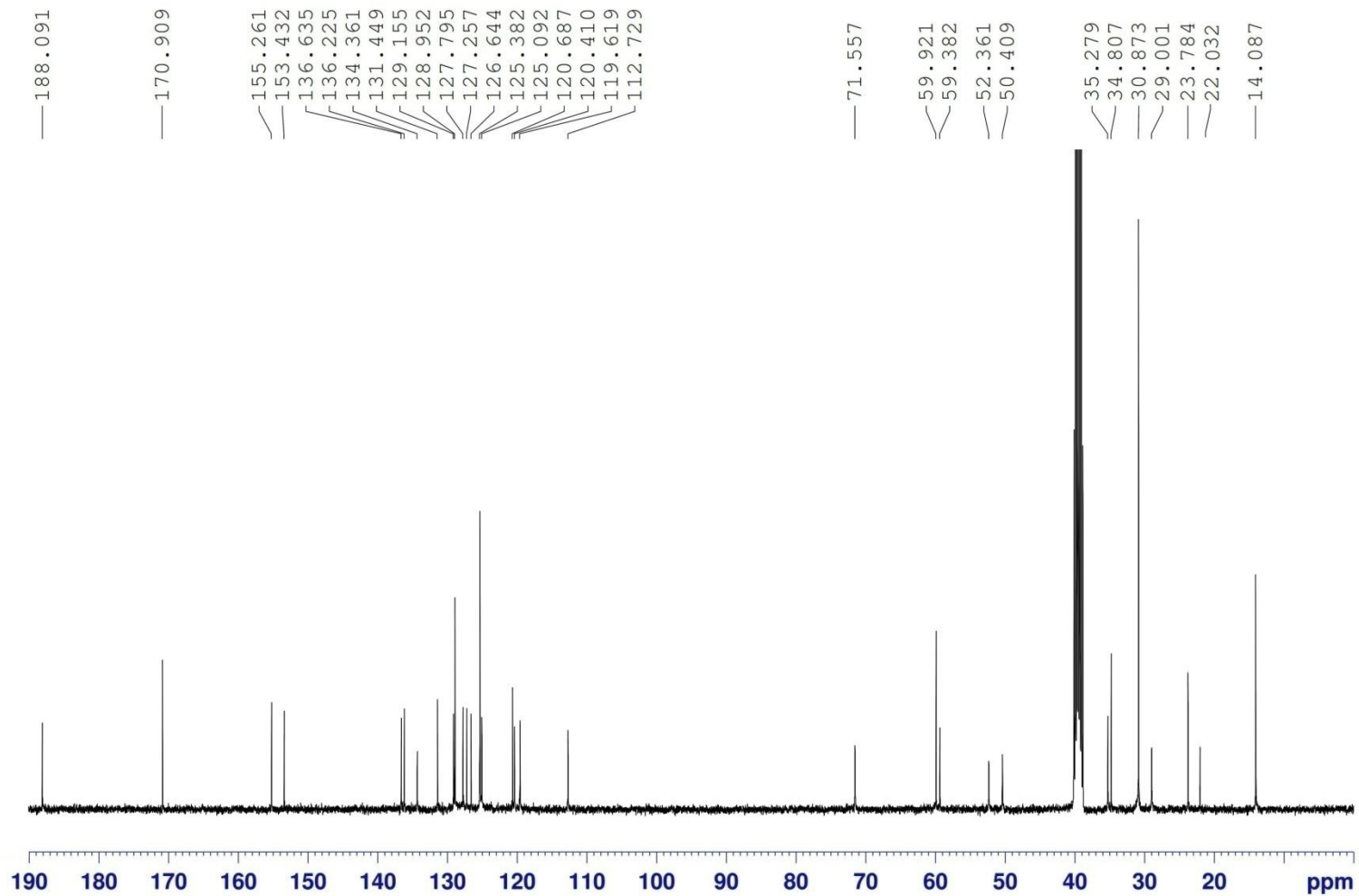
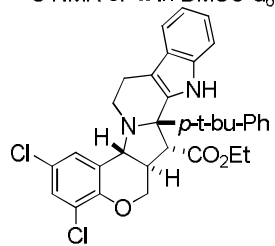


<sup>1</sup>H NMR of **4i** in DMSO-d<sub>6</sub>

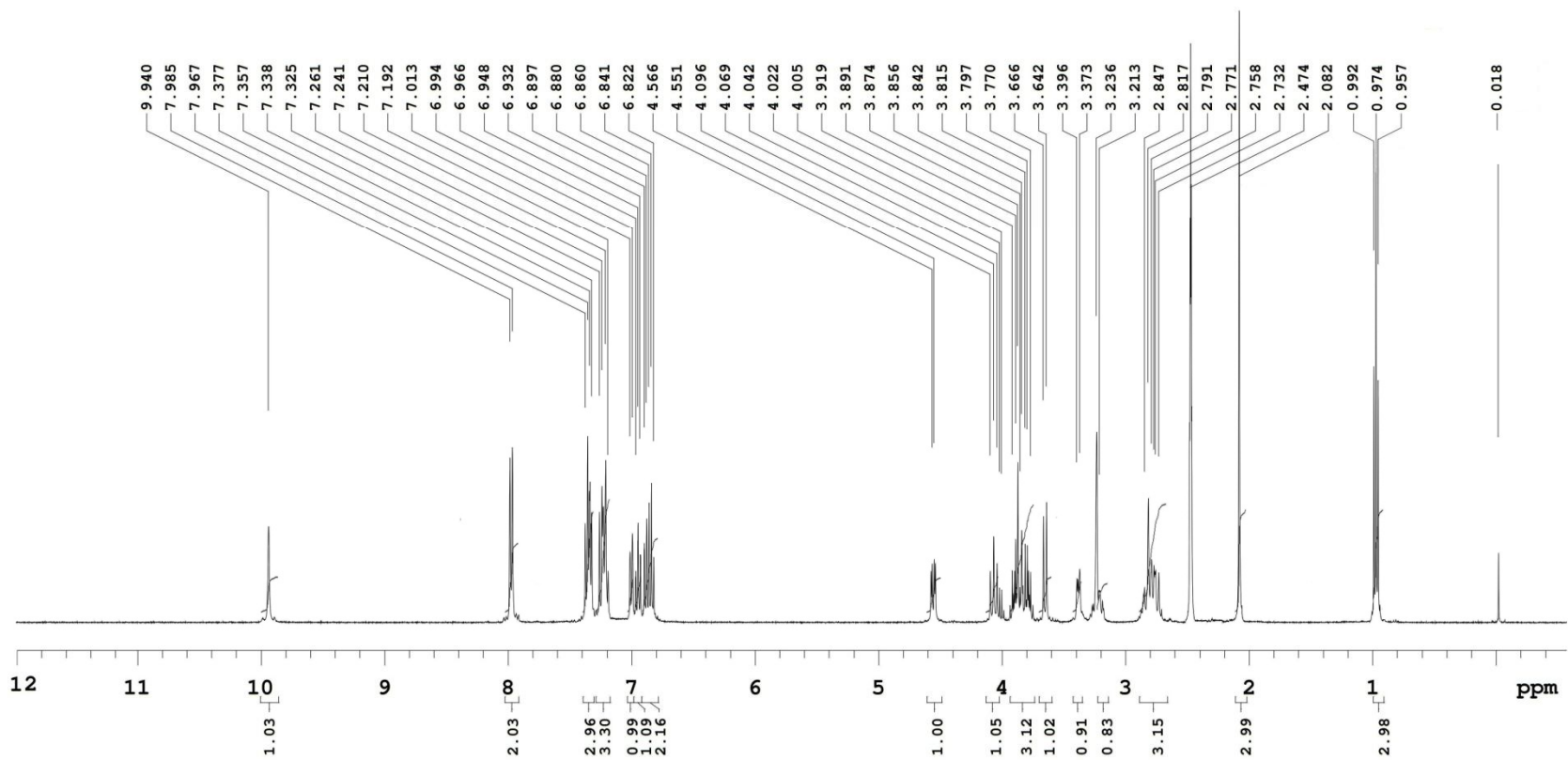
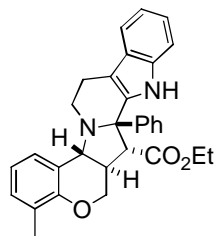




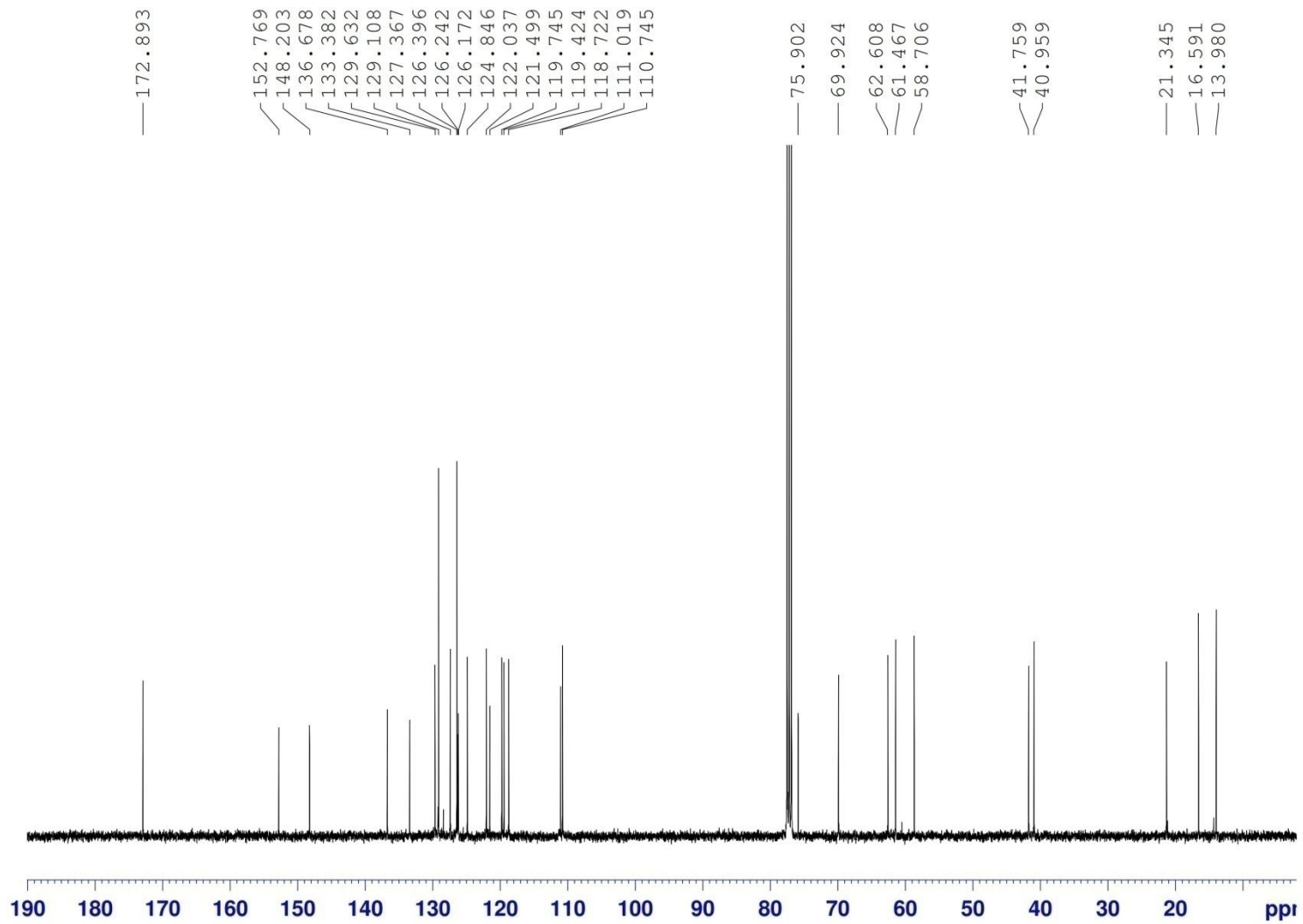
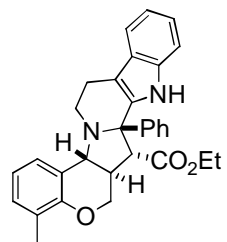
$^{13}\text{C}$  NMR of **4i** in  $\text{DMSO-d}_6$



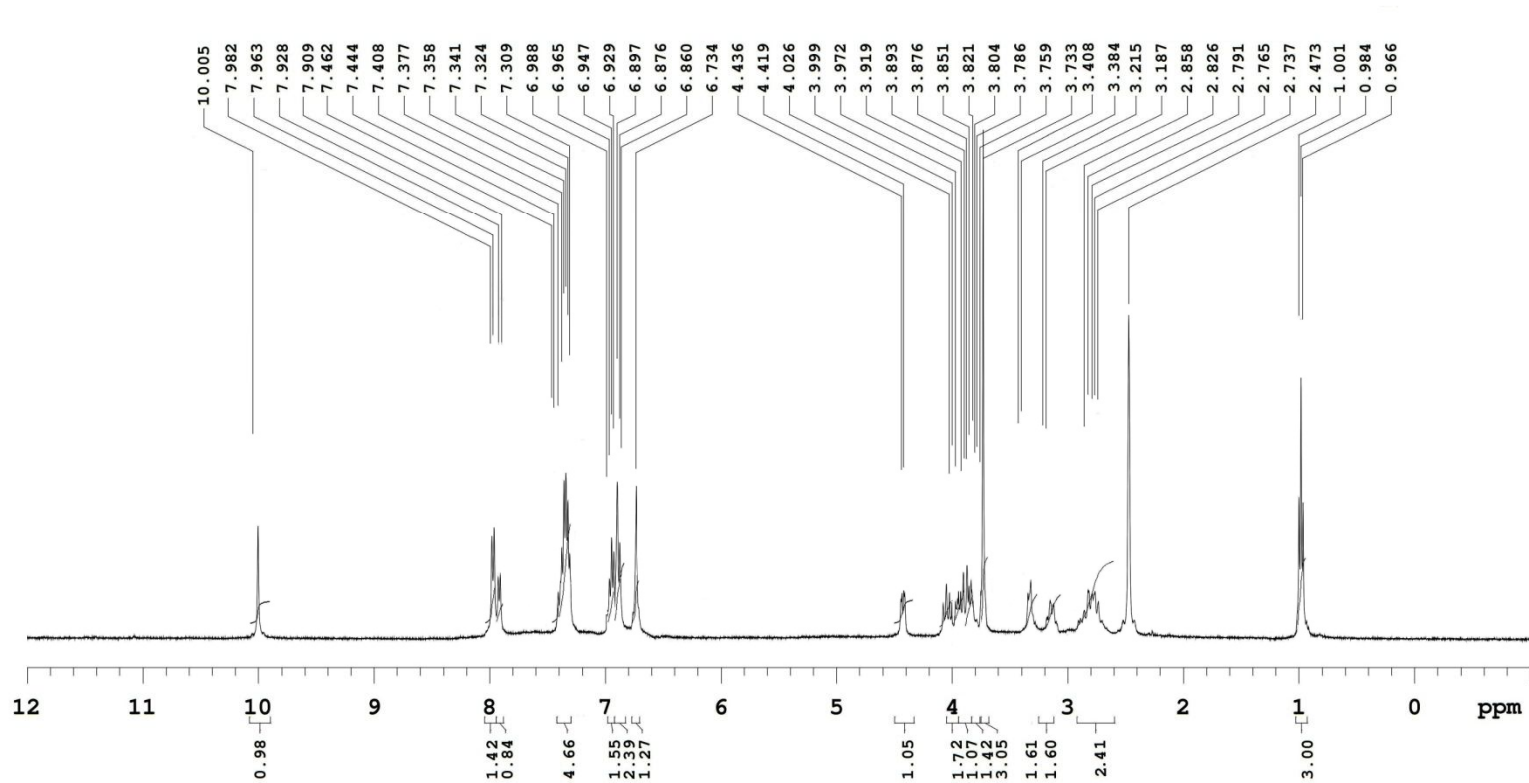
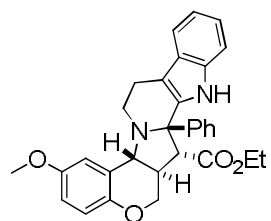
<sup>1</sup>H NMR of **4j** in DMSO-d<sub>6</sub>



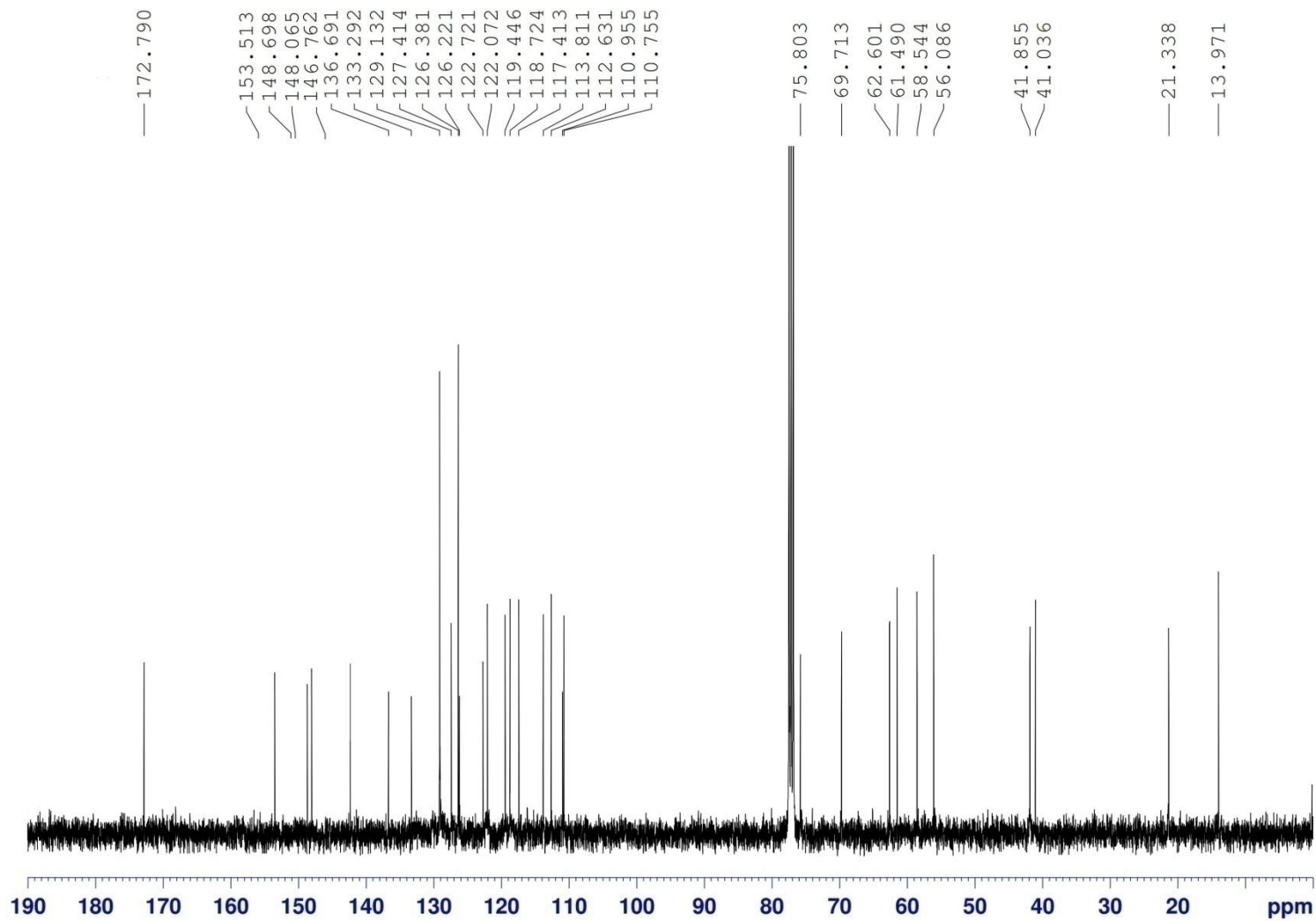
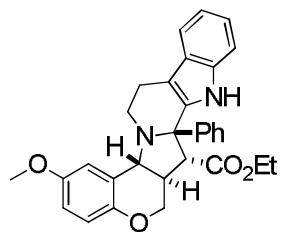
<sup>13</sup>C NMR of **4j** in CDCl<sub>3</sub>



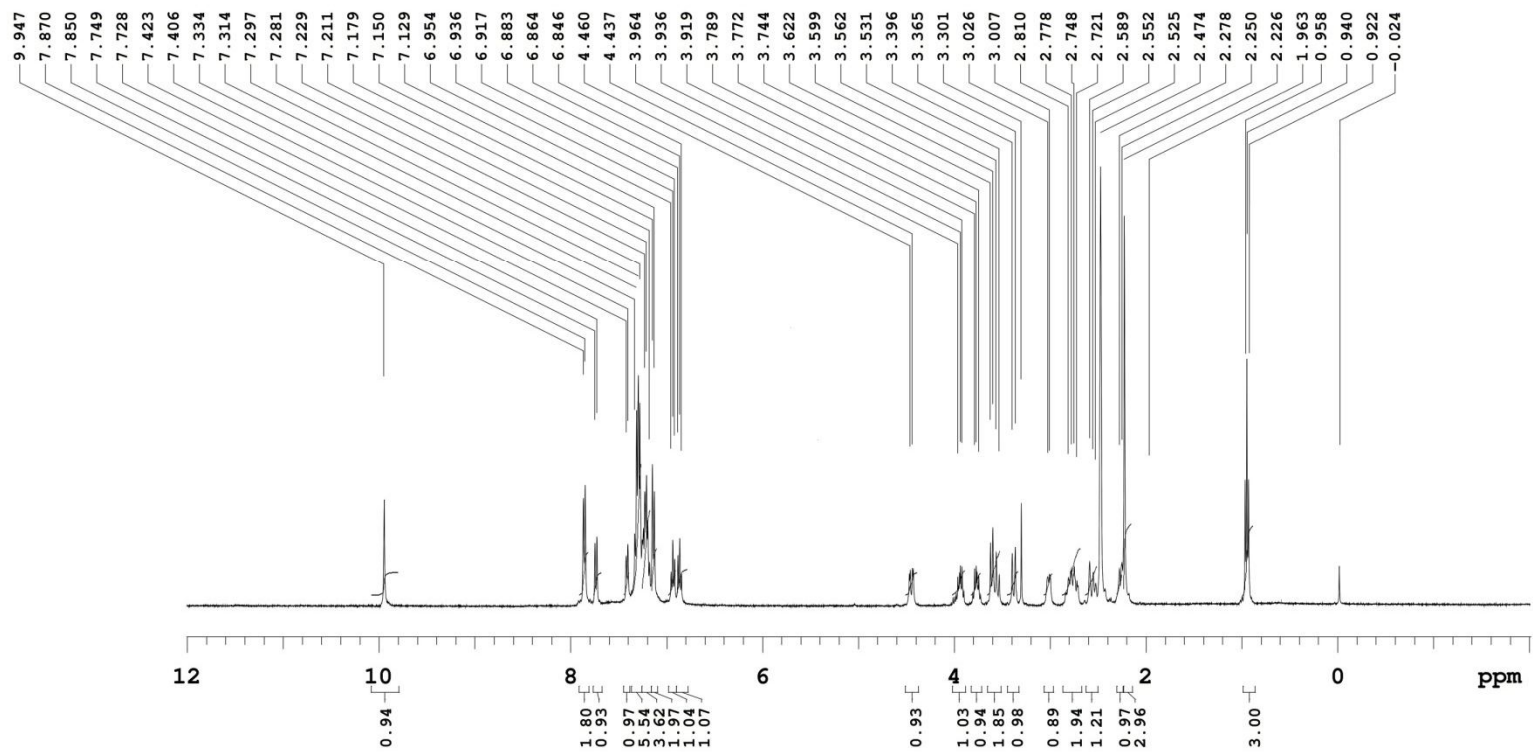
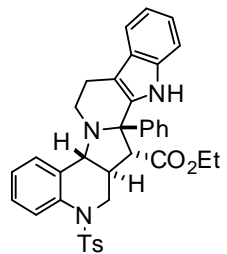
$^1\text{H}$  NMR of **4k** in  $\text{DMSO-d}_6$



$^{13}\text{C}$  NMR of **4k** in  $\text{CDCl}_3$



<sup>1</sup>H NMR of **4I** in DMSO-d<sub>6</sub>



$^{13}\text{C}$  NMR of **4I** in  $\text{CDCl}_3$

