

Supporting Information

Transition metal-free, base-mediated one-pot approach for the construction of benzo[*b*] [1,4,5]oxathiazepine 1-oxide core

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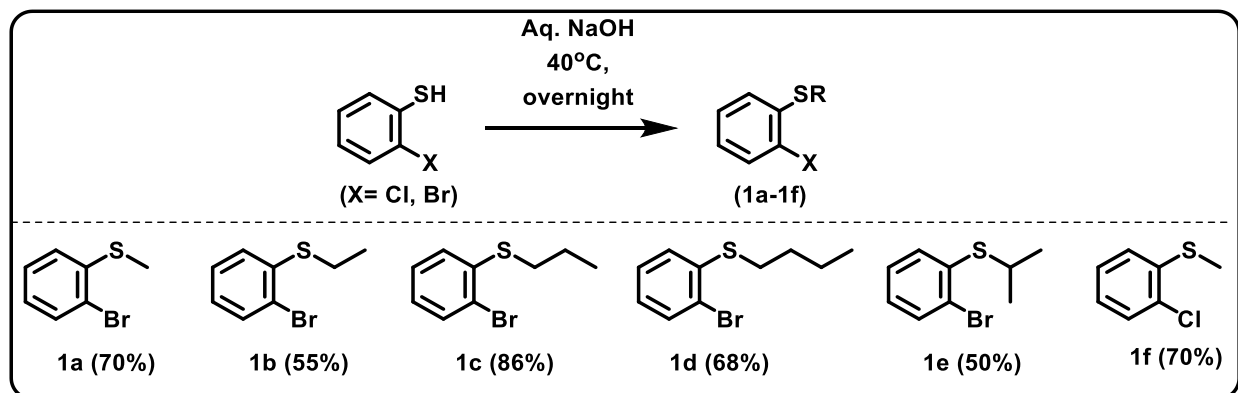
1.

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General procedure for the preparation of sulfoximines (GP1): (1a-1g)

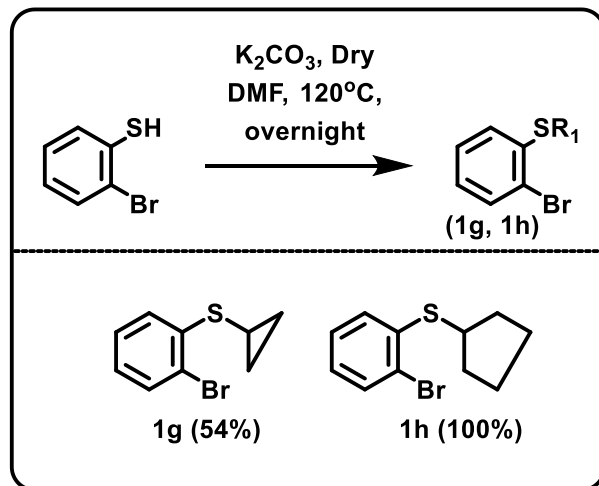
Step 1A (a-e) 2-bromo thiophenol (1 mmol, 1 equiv.) was added dropwise into a freshly prepared aqueous solution of NaOH (1.88 mmol, 1.88 equiv.) in 2.5 ml H₂O with continuously stirring at 40 °C. After that, alkyl iodide (methyl, ethyl, propyl, butyl & isopropyl iodide, 2.0 mmol, 2 equiv.) was further added slowly into the reaction mixture. Thereafter white suspension was generated & the reaction mixture was stirred at 40 °C for 12 h. In the completion of the reaction, the reaction mixture was extracted with EtOAc (3 X 50 ml), washed with brine, dried with Na₂SO₄ & organic layer was concentrated by rotary evaporator. Yellow oil was yielded with 90 % of conversion. The compound was placed for next reaction without purification.

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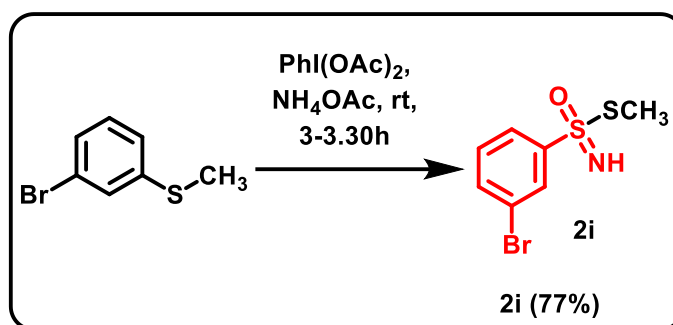
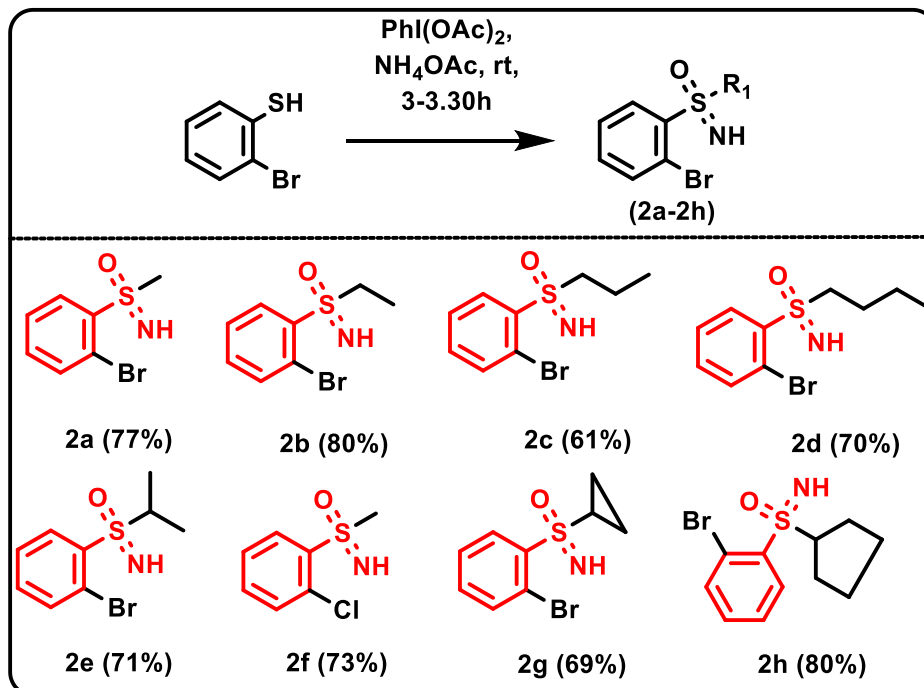


Step 1B (1f, 1g) To a stirred solution of substituted 2-Bromo thiophenol (1.0 mmol, 1.0 equiv.) and K₂CO₃ (1.5 mmol, 1.5 equiv) in dry DMF (2.5 mL) under nitrogenous atmosphere, alkyl bromide (cyclopropyl, cyclopentyl iodide, 1.3 mmol, 1.3 equiv.) was added dropwise. The reaction mixture was stirred at 120 °C for overnight. After the complete conversion (monitored by TLC), reaction mixture was cooled to room temperature & quenched with water. The crude reaction mixture was transferred into a separatory funnel, extracted with chilled Et₂O (3 × 50 mL) & water (1 × 50 mL). The organic layer was washed with brine, dried in Na₂SO₄ & solvent

was evaporated by rotary evaporator which yielded yellow oil with good yield. The compound was further placed for next reaction without purification.²

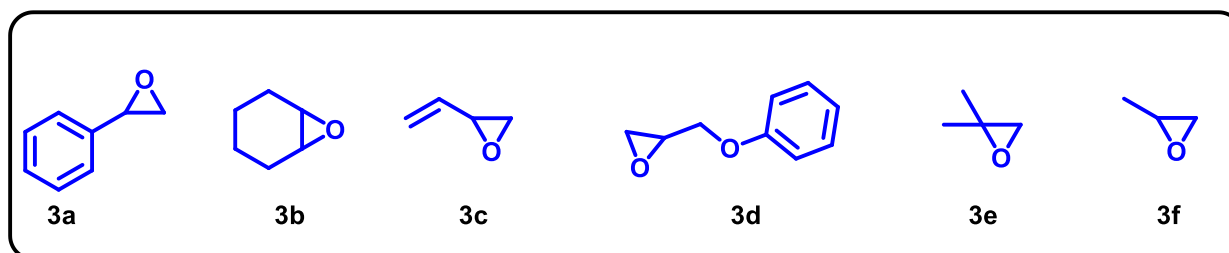


Step 2 (2a-2i) The sulfide (obtained from **Step 1A**, **Step 1B**) (1 mmol, 1 equiv.), (diacetoxyiodo)benzene (2.5 mmol, 2.5 equiv) and ammonium acetate (2.5 mmol, 2 equiv.) were taken in a 100 ml round bottom flask. MeOH (2.5 mL) was added under N_2 condition and the reaction mixture was stirred at $25^\circ C$ for 3-3.30 h.³ At the end of the reaction (monitored by TLC), the solvent was removed under reduced pressure by rotary evaporator. *Ortho*-bromo NH-sulfoximine(**2a-2i**) were purified by basified column chromatography (100-200 mesh size).

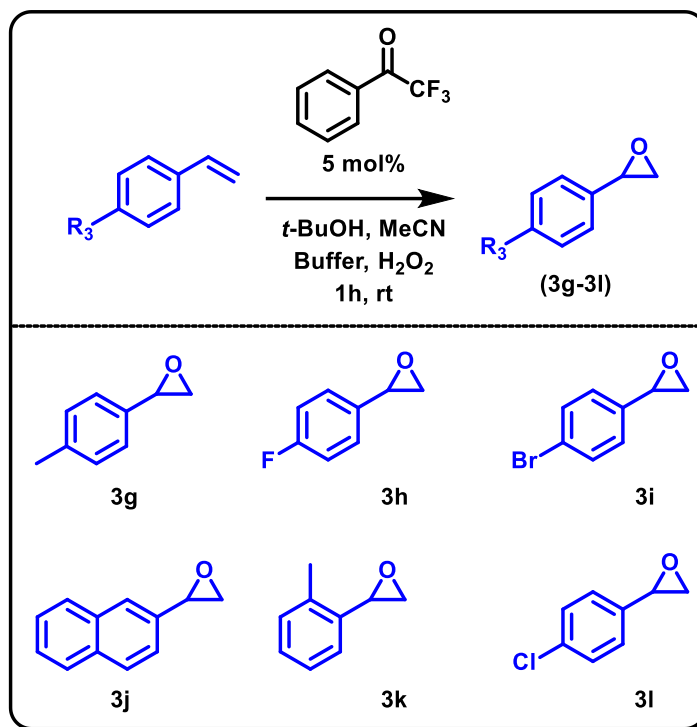


2. General procedures for the preparation of epoxides (GP2): (3a-3n)

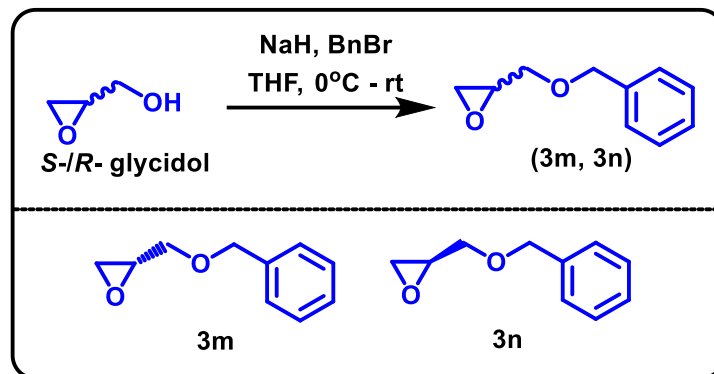
Note- Compound 3a- 3f are commercially available.



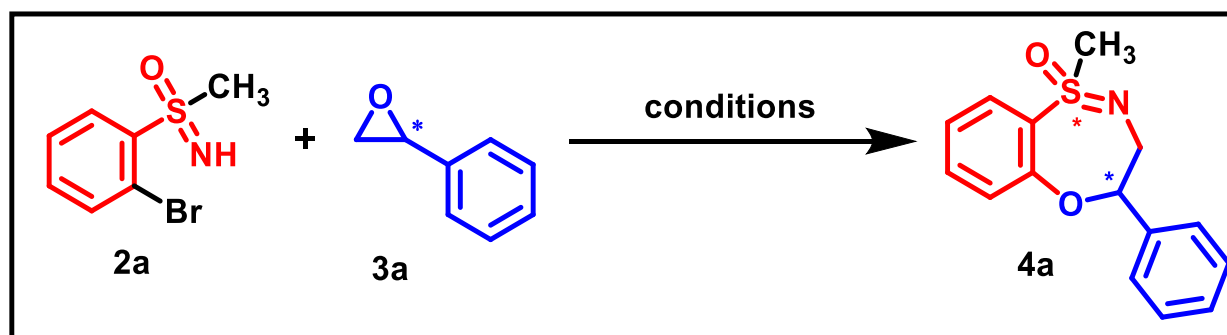
Step 1 Alkene (1.00 mmol) was taken in a 50 ml round-bottom flask, afterwards 2,2,2- trifluoro-1-phenylethanone (0.01 ml). *tert*-Butyl alcohol (1.875 mL), aqueous buffer solution (1.0 mL, 0.6 M K₂CO₃, 4 × 10⁻⁵ M EDTA tetrasodium salt), acetonitrile (0.13 ml), and 30% aqueous H₂O₂ (0.28 mL) were added consecutively. The reaction mixture was stirred for 1 h at room temperature. Thereafter the reaction mixture was extracted with EtOAc (3 x 25 ml) & concentrated under vacuo. The compounds (**3g-3l**) were further placed for next reaction without purification.⁴



Step 2 (**3m**, **3n**) OH group of *S*-/*R*- glycidol (**3m**, **3n**) was protected to OBn by the treatment with benzyl bromide (1.5 equiv), NaH (3.0 equiv) as modified version of Finch approach.⁵



2. Other Optimizations

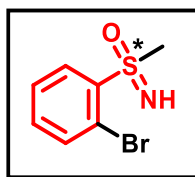


Entry	Base (equiv.)	Temperature(°C)	Solvent	Yield (%)
1.	NaOH(2.5)	rt	DMSO	NR
2.	K ₂ CO ₃ (2.5)	rt	DMF	NR
3.	Cs ₂ CO ₃ (2.5)	rt	DMF/DMSO	NR
4.	Et ₃ N(2.5)	rt	DMF	NR

5.	DIPEA(2.5)	rt	DMF	NR
6.	DBU(2.5)	rt	DMSO	NR
7.	DABCO(2.5)	rt	DMSO	NR

4. Characterization of Sulfoximines

(2-Bromophenyl)(imino)(methyl)- λ^6 -sulfanone (2a)



Compound **2a** was prepared by **GP1**.^{1,6}

Physical state: Orange colored solid

Yield: 77% (2.7 g)

Melting Point: 65 °C

R_f: 0.38 (Hexane: Ethyl Acetate = 70:30)

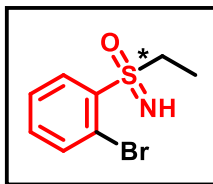
¹H NMR (400 MHz, CDCl₃): δ_{H} 8.24 (1H, dd, $J = 7.8, 1.7$ Hz), 7.77 (1H, dd, $J = 7.8, 1.2$ Hz), 7.53-7.49 (1H, m), 7.45-7.41 (1H, m), 3.33 (3H, s), 2.02 (1H, brs).

¹³C NMR (125 MHz, CDCl₃): 142.6, 135.6, 134.0, 130.9, 128.1, 120.7, 43.1.

MS (ESI): ($[M]^+$) 234.3, 236.3.

HPLC analysis: column= CHIRALPAKIA, 2-propanol/n-hexane=5/95, flow rate =1.0 mL/min, $\lambda = 230$ nm, retention time: 46.018 min, 54.957 min.

(2-Bromophenyl)(ethyl)(imino)- λ^6 -sulfanone (2b)



Compound **2b** was prepared by **GP1**.^{1,7}

Physical state: Light yellow oil

Yield: 80% (800 mg)

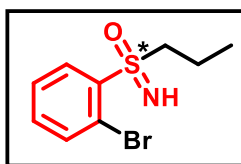
R_f : 0.6 (Hexane: Ethyl Acetate = 70:30)

¹H NMR (400 MHz, CDCl₃): δ_{H} 8.20 (1H, dd, $J = 7.9, 1.8$ Hz), 7.76 (1H, dd, $J = 7.8, 1.2$ Hz), 7.53-7.49 (1H, m), 7.45-7.41 (1H, m), 3.62-3.53 (1H, m), 3.50-3.41 (1H, m), 2.54 (1H, brs), 1.27 (3H, t, $J = 7.5$ Hz).

¹³C NMR (125 MHz, CDCl₃): 140.6, 135.7, 133.9, 132.2, 127.9, 120.9, 48.7, 7.4.

MS (ESI): ($[\text{M}]^+$) 248.3.

(2-Bromophenyl)(imino)propyl- λ^6 -sulfanone (2c)



Compound **2c** was prepared by **GP1**.

Physical state: Light yellow oil

Yield: 61% (1.2g)

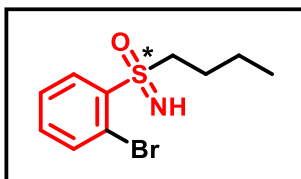
R_f: 0.4 (Hexane: Ethyl Acetate =50:50)

¹H NMR (400 MHz, CDCl₃): δ_{H} 8.20 (1H, dd, $J = 7.9, 1.8$ Hz), 7.75 (1H, dd, $J = 7.8, 1.3$ Hz), 7.52-7.48 (1H, m), 7.43-7.40 (1H, m), 3.55-3.48 (1H, m), 3.45-3.38 (1H, m), 1.86-1.63 (2H, m), 1.01 (3H, t, $J = 7.5$ Hz).

¹³C NMR (125 MHz, CDCl₃): 141.4, 135.6, 133.9, 131.9, 127.9, 120.9, 55.9, 16.6, 12.9.

HRMS(ESI) (m/z) [C₉H₁₂BrNOS+H]⁺: Calcd. 261.9896, found 261.9892.

(2-Bromophenyl)(butyl)(imino)- λ^6 -sulfanone (2d)



Compound **2d** was prepared by **GP1**.

Physical state: Light yellow oil

Yield: 70% (394 mg)

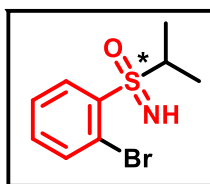
R_f: 0.5 (Hexane: Ethyl Acetate =50:50)

¹H NMR (400 MHz, CDCl₃): δ_{H} 8.21 (1H, dd, $J = 7.9, 1.8$ Hz), 7.76 (1H, dd, $J = 7.8, 1.3$ Hz), 7.52-7.48 (1H, m), 7.44-7.40 (1H, m), 3.57-3.50 (1H, m), 3.47-3.39 (1H, m), 2.44(1H, brs), 1.796-1.59 (2H, m), 1.46-1.37 (2H, m), 0.91 (3H, t, $J = 7.4$ Hz).

¹³C NMR (125 MHz, CDCl₃): 141.3, 135.6, 133.9, 131.9, 127.9, 120.8, 53.9, 24.7, 21.5, 13.5.

HRMS(ESI) (m/z) [C₁₀H₁₄BrNOS+H]⁺: Calcd. 276.0052, found 276.0053.

(2-Bromophenyl)(imino)(isopropyl)- λ^6 -sulfanone (2e)



Compound **2e** was prepared by **GP1**.

Physical state: White solid

Yield: 71% (770 mg)

Melting Point: 98°C

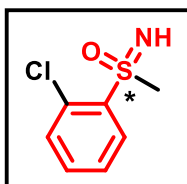
R_f: 0.5 (Hexane: Ethyl Acetate =50:50)

¹H NMR (400 MHz, CDCl₃) δ_H 8.20 (1H, dd, *J*= 7.8, 1.8 Hz), 7.75(1H, dd, *J*= 7.8, 1.3 Hz), 7.51-7.47 (1H, m), 7.43-7.39 (1H, m), 4.00-3.91 (1H, m), 3.27(1H, brs), 1.40 (3H, d, *J* = 6.7 Hz), 1.26 (3H, d, *J* = 6.7 Hz).

¹³C NMR (125 MHz, CDCl₃): 140.1, 135.7, 133.8, 132.6, 127.8, 120.9.

HRMS(ESI) (m/z) [C₉H₁₂BrNOS+H]⁺: Calcd. 261.9896, found 261.9891.

(2-chlorophenyl)(imino)(methyl)-λ⁶-sulfanone(2f)



Compound **2h** was prepared by **GP1**.

Physical state: semisolid

Yield: 73% (439 mg)

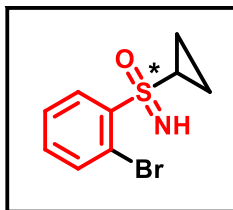
R_f: 0.38 (Hexane: Ethyl Acetate = 70:30)

¹H NMR (400 MHz, CDCl₃): δ_H 8.19-8.16 (1H, m), 7.55-7.50 (2H, m), 7.46-7.43 (1H, m), 3.31 (3H, s).

¹³C NMR (125 MHz, CDCl₃): 140.9, 134.1, 132.4, 132.1, 130.7, 127.5, 43.4.

HRMS(ESI) (m/z) [C₇H₈ClNOS+H]⁺: Calcd. 190.0088, found 190.0087.

(2-Bromophenyl)(cyclopropyl)(imino)- λ⁶-sulfanone(2g)



Compound **2f** was prepared by **GP1**.

Physical state: Brownish semi solid;

Yield: 69% (382 mg)

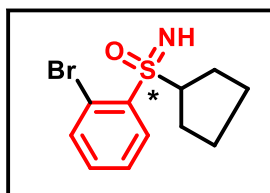
R_f: 0.5 (Hexane: Ethyl Acetate =50:50)

¹H NMR (400 MHz, CDCl₃): δ_H 8.08 (1H, dd, *J*= 7.8, 1.8 Hz), 7.73 (1H, dd, *J*= 7.8, 1.3 Hz), 7.46-7.42 (1H, m), 7.39-7.35 (1H, m), 3.21-3.15 (1H, m), 2.87 (1H, brs), 1.51-1.43 (1H, m), 1.14-1.09 (1H, m), 1.08-1.04 (1H,m), 0.97-0.89 (1H, m).

¹³C NMR (125 MHz, CDCl₃): 142.4, 135.6, 133.5, 130.6, 127.8, 120.4, 31.3, 6.1, 5.7.

HRMS(ESI) (m/z) [C₉H₁₀BrNOS+H]⁺: Calcd. 259.9739, found 259.9738.

(2-Bromophenyl)(cyclopentyl)(imino)- λ⁶-sulfanone (2h)



Compound **2g** was prepared by **GP1**.

Physical state: Light yellow oil.

Yield: 80% (1.32 g)

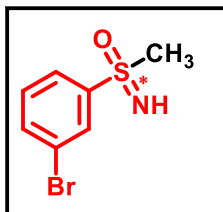
R_f: 0.3 (Hexane: Ethyl Acetate = 50:50)

¹H NMR (400 MHz, CDCl₃): δ_H 8.21 (1H, dd, *J*= 7.8, 1.7 Hz), 7.74 (1H, dd, *J*= 7.8, 1.2 Hz), 7.49-7.45 (1H, m), 7.41-7.37 (1H, m), 4.32-4.24 (1H, m), 3.75(1H, brs), 2.33-2.24 (1H, m), 2.33-2.24 (1H, m), 2.03-2.00 (1H, m), 1.88-1.56 (3H,m).

^{13}C NMR (125 MHz, CDCl_3): 141.3, 135.7, 133.7, 132.2, 127.8, 120.8, 61.7, 27.5, 26.8, 26.1, 26.0.

HRMS(ESI) (m/z) [$\text{C}_{11}\text{H}_{14}\text{BrNOS}+\text{H}$] $^+$: Calcd. 288.0052, found 288.0053.

S-methyl 3-bromobenzenesulfonimidothioate (2i)



Compound **2h** was prepared by **GP1**.^{1,6}

Physical state: semisolid

Yield: 77% (111 mg)

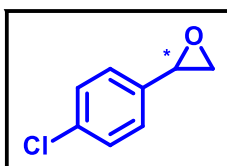
R_f: 0.38 (Hexane: Ethyl Acetate = 70:30)

^1H NMR (500 MHz, CDCl_3): δ_{H} 8.15-8.14 (1H, m), 7.93-7.92 (1H, m), 7.73-7.72(1H, m), 7.44-7.40 (1H, m), 3.09 (3H, s).

^{13}C NMR (125 MHz, CDCl_3): 145.6, 136.1, 130.8, 130.7, 126.3, 123.3, 46.1.

MS (ESI): ($[\text{M}+3]^+$) 236.1.

2-(4-chlorophenyl)-2 λ^3 -oxirane (3l)



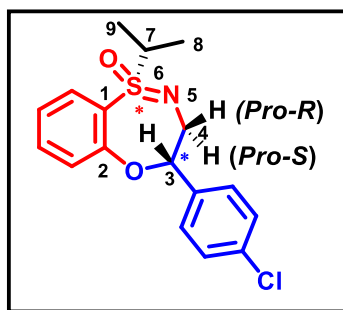
HPLC analysis: column= CHIRALPAKIG, 2-propanol/n-hexane=5/95, flow rate =1.0 mL/min, λ =230 nm, retention time: 4.786 min, 5.010 min.

5. 2D NMR analysis

5.1 Stereochemistry analysis of the exact structure of **4q**

NMR Study

NMR spectra were recorded on Bruker Advance DPX 200FT, Bruker Robotics, Bruker DRX 400 Spectrometers at 400 MHz (^1H) and 125 MHz (^{13}C) in suitable solvents (CDCl_3). Resonance assignments were carried out using various one dimensional, two-dimensional experiments. **4q** isomer was separable & that was proved by integration of ^1H NMR spectra (18H protons) along with molecular formula $\text{C}_{17}\text{H}_{18}\text{ClNO}_2\text{S}$ of both the compounds. The formation of seven-membered ring was confirmed by two dimensional NOESY, COSY, HSQC and HMBC experiments. The formation of seven membered rings was confirmed with the characteristics long range correlation obtained from HMBC. From NOESY (**R,S**)-**4q**, C_{11}H (7.80 ppm) shows weak interaction with C_8H (3.72-3.64 ppm), C_5H (*Pro-S*) (3.62 ppm) weak interaction with C_{15} , C_{16}H (7.44-7.42 ppm), C_4H (5.07 ppm) strong interaction with C_{15} , C_{16}H (7.44-7.42 ppm), C_4H (5.07 ppm) strong interaction with C_5H (*Pro-S*) (3.82 ppm) and C_4H (5.07 ppm) weak interaction with C_5H (*Pro-S*) (3.62 ppm). In HMBC correlation for compound (**R,S**)-**4q** [C_4 (87 ppm) shows moderate interaction with $\text{C}_{15,16}\text{H}$ (7.44-7.42 ppm)]. Isopropyl group has no interaction with *pro-R*, *Pro-S* H's. As a result, isopropyl group is in the below site.



Proton	^1H Chemical shift and coupling constant	^{13}C Chemical shift
C_2H	-	156.6

C₄H	5.07 (dd, <i>J</i> = 5.9, 3.08 Hz)	87.1
C₅H_(Pro-R)	3.82 (dd, <i>J</i> = 14.6, 3.8 Hz)	51.3
C₅H_(Pro-S)	3.62 (dd, <i>J</i> = 14.6, 6.0 Hz)	51.3
C₈H	3.72-3.6 4(m)	54.5
C_{9,10}H	1.36, 1.30 (d, <i>J</i> = 6.9, 6.8 Hz)	16.2, 15.1
C₁₁H	7.8 (dd, <i>J</i> = 7.8, 1.6 Hz)	131.0
C_{15, 16}H	7.44-7.42 (m)	127.7

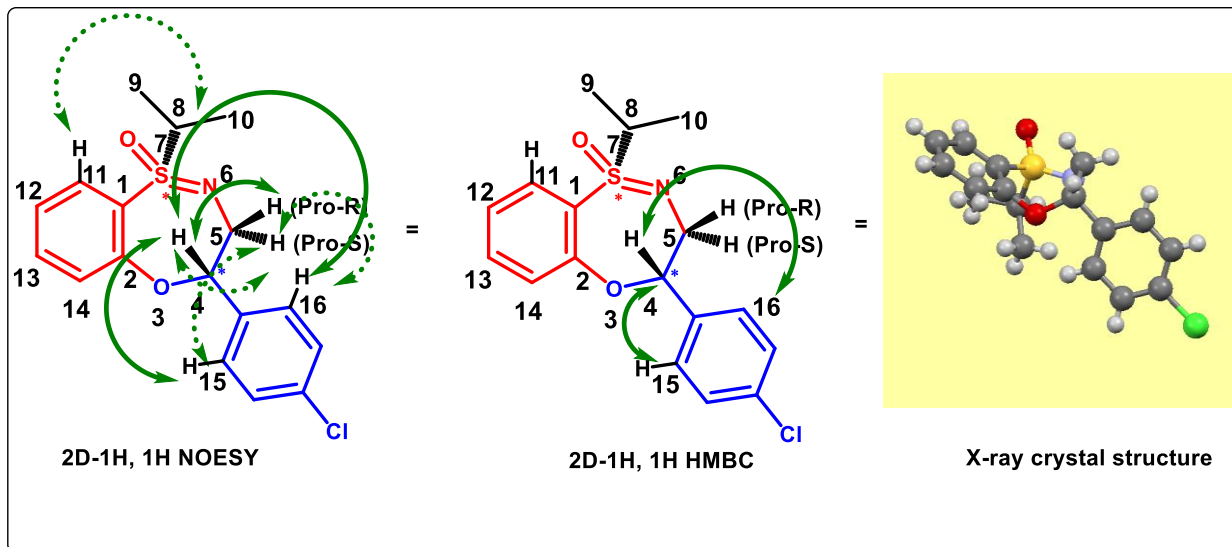
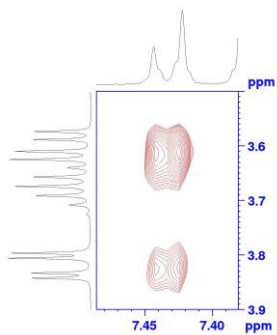
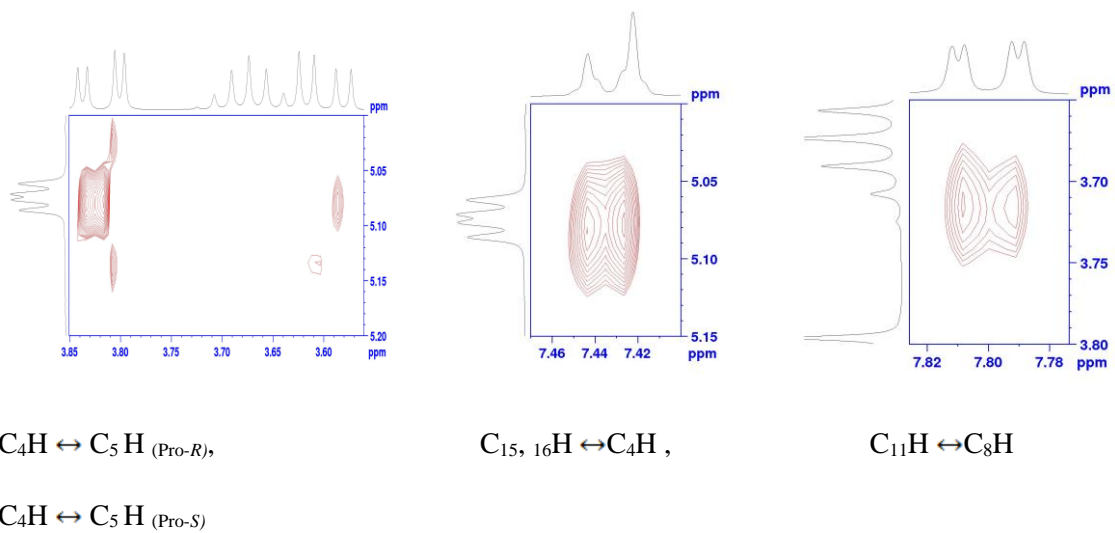


Figure S1-Characteristics NOESY and HMBC correlation for compound **(R,S)-4q**



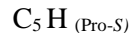
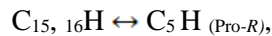


Figure S2- Characteristics NOESY correlation for compound (*R,S*)-**4q**

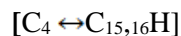
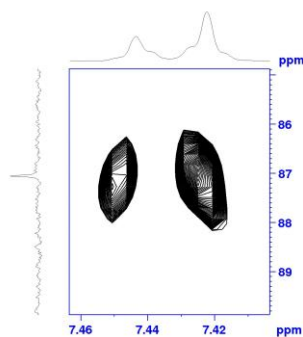
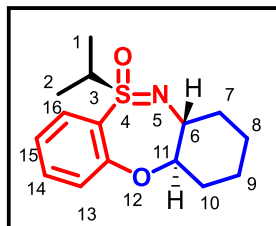


Figure S3- Characteristics HMBC correlation for compound (*R,S*)-**4q**

5.2 Stereochemistry analysis of the exact structure of **4r**

NMR Study

NMR spectra were recorded on Bruker Advance DPX 200FT, Bruker Robotics, Bruker DRX 400 Spectrometers at 400 MHz (1H) and 125 MHz (^{13}C) in suitable solvents ($CDCl_3$). Resonance assignments were carried out using various one dimensional, two-dimensional experiments. Single isomer of **4r** was separable & that was proved by integration of 1H NMR spectra (**21H** protons) along with molecular formula $C_{15}H_{21}NO_2S$ of both the compounds. The formation of seven membered ring was confirmed by two dimensional NOESY, HSQC experiments. From NOESY (*R,R,S*)-**4r**, C_6H (3.37-3.33 ppm) shows moderate interaction with $C_1 H$ (1.29 ppm), $C_{11} H$ has no such interaction with $C_1/ C_2/ C_3 H$'s So C_6 proton is in above orientation while C_{11} proton is in below orientation.



Proton	¹ H Chemical shift and coupling constant	¹³ C Chemical shift
C ₁ H	1.29 (d, <i>J</i> = 6.8 Hz)	14.2
C ₂ H	1.16 (d, <i>J</i> = 6.8 Hz)	16.2
C ₃ H	3.88-3.79(m)	51.0
C ₆ H	3.37-3.31(m)	62.0
C ₁₁ H	3.77-3.71 (m)	89.9
C _{8,9} H	1.54-1.33(m)	32.8

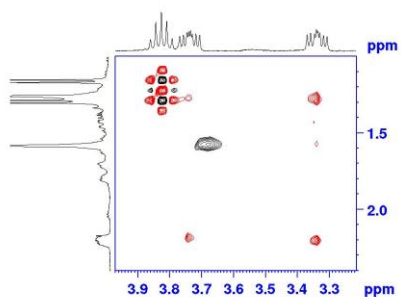
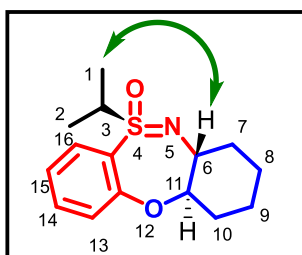
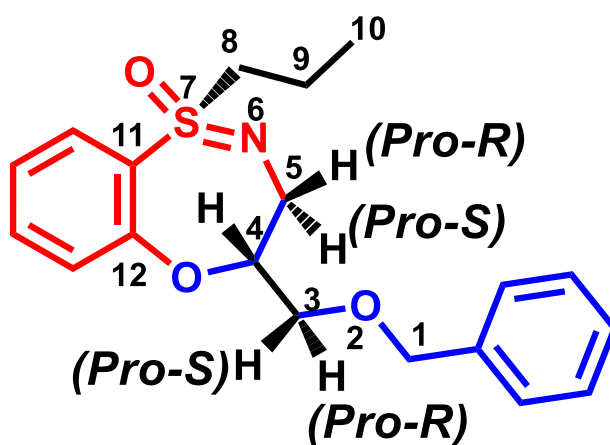


Figure S4- Characteristics NOESY correlation for compound (*R,R,S*)-4r

5.3 Stereochemistry analysis of the exact structure of 4z and 4z'

NMR Study

NMR spectra were recorded on Bruker Advance DPX 200FT, Bruker Robotics, Bruker DRX 300 and 400 Spectrometers at 200, 300, 400 MHz (¹H) and 100, 125 MHz (¹³C) in suitable solvents (CDCl₃, DMSO-D₆). Resonance assignments were carried out using various one dimensional, two-dimensional experiments.



Proton	¹ H Chemical shift and coupling constant	¹³ C Chemical shift
C ₁ H	4.63 (s)	73.5
C ₃ H _(Pro-S)	3.72 (dd, <i>J</i> = 10, 4.9 Hz)	70.6
C ₃ H _(Pro-R)	3.83 (dd, <i>J</i> = 14.6, 6.0 Hz)	70.6
C ₄ H	4.30-4.27 (m)	85.2
C ₅ H _(Pro-R)	3.57 (dd, <i>J</i> = 14.7, 6.0 Hz)	46.2

	3.4 Hz)	
C₅H_(Pro-S)	3.49 (dd, J= 14.7, 4.7 Hz)	46.2
C₈H	3.33-3.18(m)	57.8
C₉H	1.76-1.68(m)	16.9
C₁₀H	0.94(t, J=7.4 Hz)	12.8

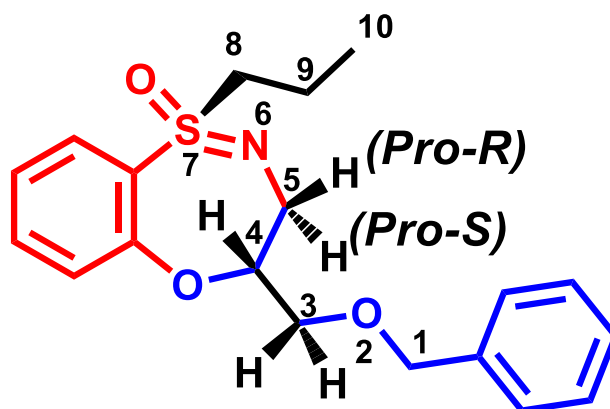


Table:- Chemical shift assignment for *(R,S)*-4z'

Proton	¹H Chemical shift and coupling constant	¹³C Chemical shift
C₁H	4.52 (q, J= 27, 12 Hz)	73.3
C₃H_(Pro-S)	3.49-3.46 (m)	68.6
C₃H_(Pro-R)	3.34 (dd, J =10.8,	68.6

	3.4 Hz)	
C₄H	4.67-4.62(m)	84.2
C₅H_(Pro-R)	3.30-3.27 (m)	44.9
C₅H_(Pro-S)	3.44-3.40(m)	44.9
C₈H	3.27-3.19(m)	59.2
C₉H	1.81-1.71(m)	17.10
C₁₀H	0.94(t, J=7.4 Hz)	12.8
C₁₂H	0.94(t, J=7.4 Hz)	152.5

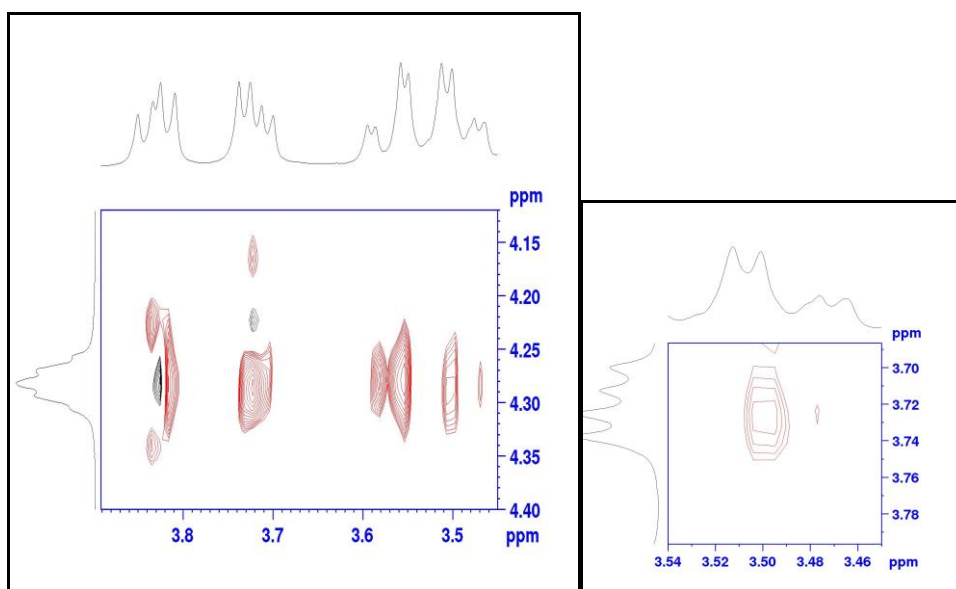


Figure S5- Characteristics NOESY correlation for compound (*R,R*)-**4z**[C₄H (3 ppm) ↔ C₃ H_(Pro-R) (3.83 ppm), C₄H (4.30-4.27 ppm) ↔ C₃ H_(Pro-S) (3.72 ppm), C₄H (4.30-4.27 ppm) ↔ C₅ H_(Pro-R) (3.57 ppm) , C₄H (4.30-4.27 ppm) ↔ C₅ H_(Pro-S) (3.49 ppm) and C₅ H_(Pro-S) (3.49 ppm)↔C₃ Pro S H C₃ H_(Pro-S) (3.72 ppm)]

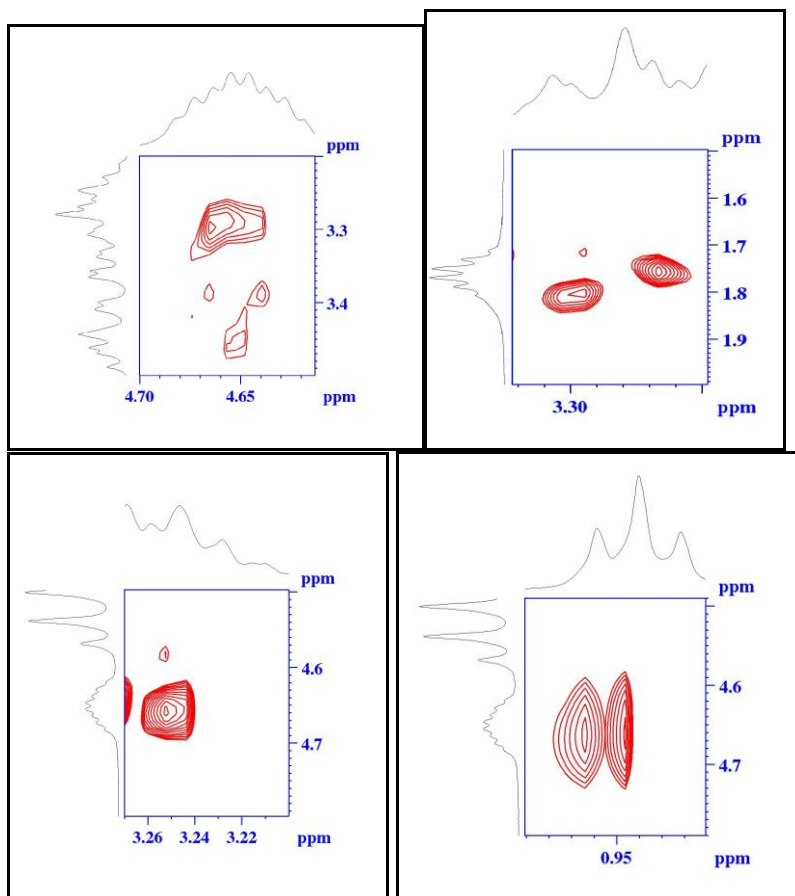


Figure S6- Characteristics NOESY correlation for compound **(R,S)-4z'** [C₄H (4.67-4.62 ppm)

↔ C₁₀ H (0.94 ppm), C₄H (4.67-4.62 ppm) ↔ C₈ H_(Pro-S) (3.27-3.19 ppm), C₄H (4.67-4.62 ppm) ↔

C₅ H_(Pro-R) (3.30-3.27 ppm), C₄H (4.67-4.62 ppm) ↔ C₅ H_(Pro-S) (3.44-3.40 ppm)]

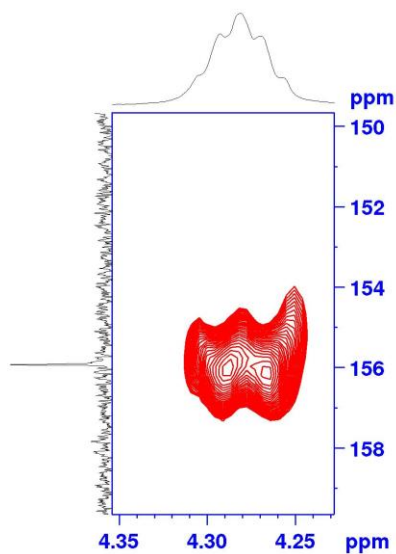


Figure S7-Characteristics HMBC correlation for compound (*R,R*)-**4z**[C_4H (δ 4.30-4.27 ppm) \leftrightarrow C_{12} (156.0 ppm)]

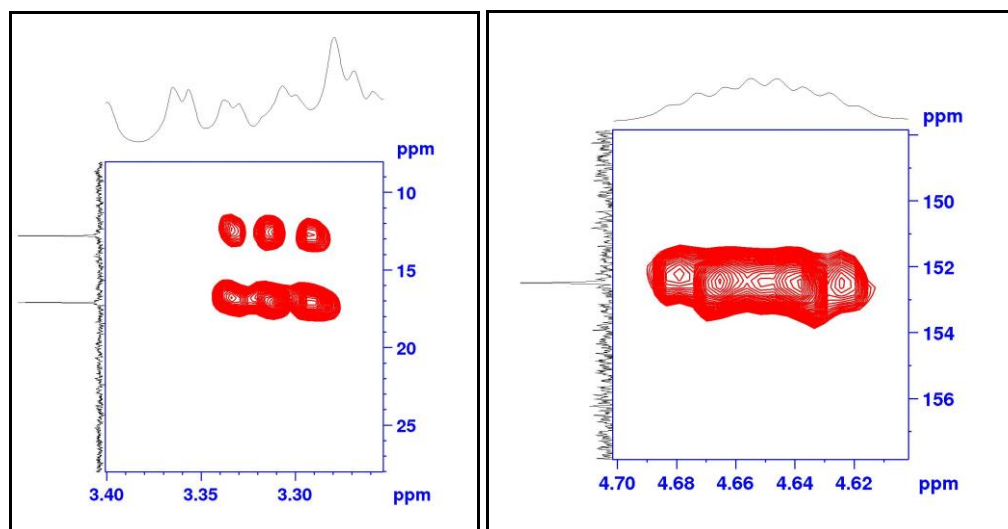


Figure S8-Characteristics HMBC correlation for compound (*R,S*)-**4z'**[C_4H (δ 4.30-4.27 ppm) \leftrightarrow C_{12} (152.5 ppm), $C_5 H_{(Pro-S)}$ (3.44-3.40 ppm) \leftrightarrow C_4 (84.2 ppm), $C_5 H_{(Pro-R)}$ (3.30-3.27 ppm) \leftrightarrow C_4 (84.2 ppm), $C_5 H_{(Pro-R)}$ (3.30-3.27 ppm) \leftrightarrow both C_9 (17.10 ppm) C_{10} (12.8 ppm)]

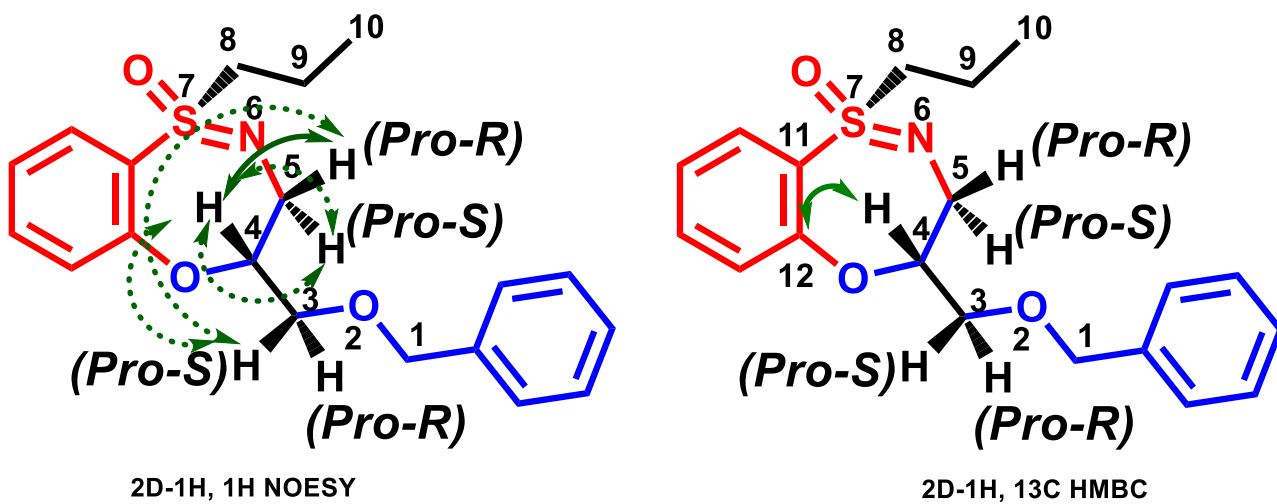


Figure S9- Characteristics NOESY and HMBC correlation for compound (*R,R*)-4z

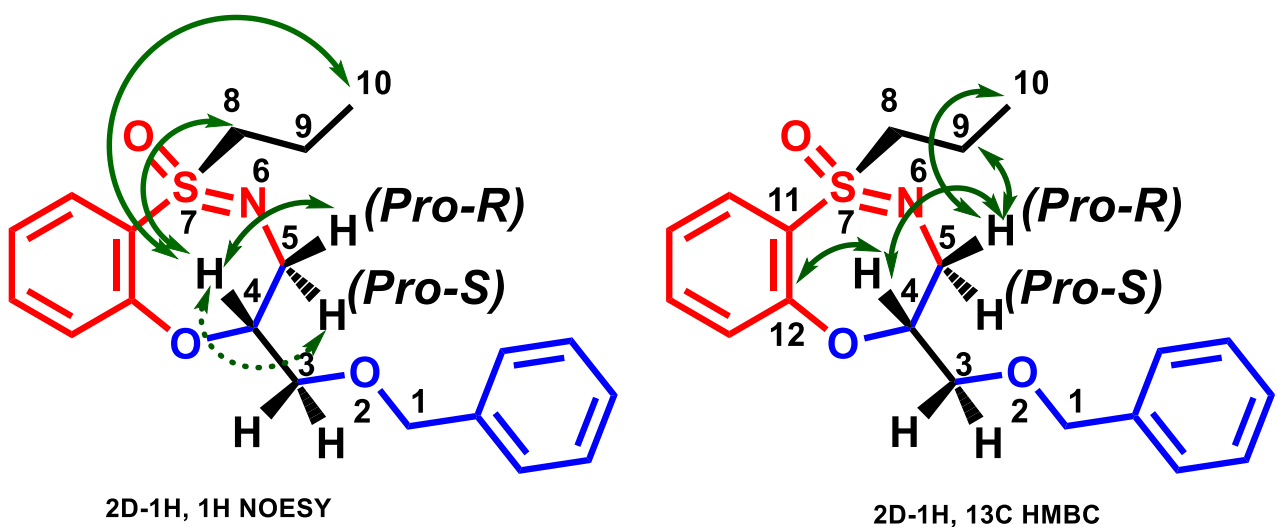


Figure S10- Chemical shift NOESY and HMBC assignment for (*R,S*)-4z'

5.4 Stereochemistry analysis of the exact structure of 4zb

NMR Study

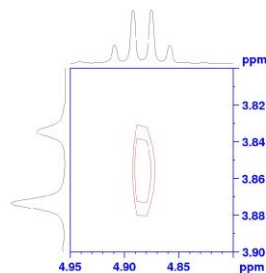


Figure S11- Characteristics NOESY correlation for compound (*S,S*)-**4zb**[C₆H (4.91-4.86 ppm)

↔ C₄ H (_{Pro-S}) (3.87-3.83 ppm)]

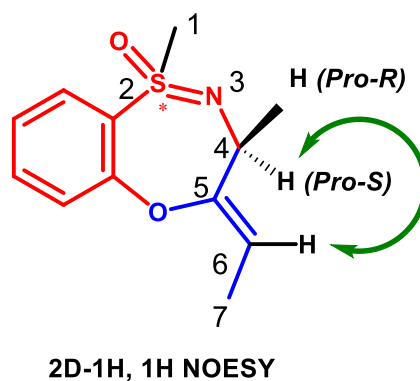


Figure S12-Chemical shift NOESY assignment for **4zb**

6. X-ray Crystallographic Information

6.1 X-Ray Crystallography data of compound 4p

Single-crystal diffraction analysis data were collected at 296K with a Bruker APEX-II CCD Duo diffractometer (operated at 1500 W power: 50 kV, 30 mA) using graphite monochromatic Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$).

Sample preparation for crystal growth- The 5 mg compound (3g) was taken in a 1 ml HPLC glass vial & compound was dissolved in 1ml acetonitrile solvent (lower solubility). This HPLC vial was placed in a 15ml glass vial which already contains 2-3ml of hexane. Then the vial was closed by wrapping with aluminium foil & one small niddle

was attached on the neck. Kept it for 4 to 5 day for growing of crystal. Slow evaporation at room temperature in a dark place till crystals formed.

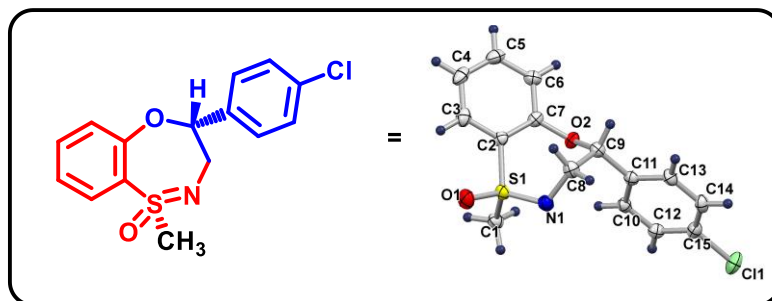


Figure S13- ORTEP diagram drawn with 50% ellipsoid for non-H atoms of the crystal structure of compound 4p determined at 296K (CCDC deposition No. 2113397)

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) AB_175

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: AB_175

Bond precision:	C-C = 0.0019 Å	Wavelength=0.71073	
Cell:	a=9.9201(3) alpha=90	b=7.5619(2) beta=99.478(1)	c=18.7671(5) gamma=90
Temperature:	296 K		
Volume	Calculated 1388.59(7)	Reported 1388.59(7)	
Space group	P 21/n	P 21/n	
Hall group	-P 2yn	-P 2yn	
Moiety formula	C15 H14 Cl N O2 S	C15 H14 Cl N O2 S	
Sum formula	C15 H14 Cl N O2 S	C15 H14 Cl N O2 S	
Mr	307.78	307.78	
Dx, g cm ⁻³	1.472	1.472	
Z	4	4	
Mu (mm ⁻¹)	0.425	0.425	
F000	640.0	640.0	
F000'	641.32		
h, k, lmax	13, 10, 25	13, 10, 25	
Nref	3478	3459	
Tmin, Tmax	0.894, 0.938	0.411, 0.590	
Tmin'	0.880		
Correction method=	# Reported T Limits: Tmin=0.411 Tmax=0.590		
AbsCorr =	MULTI-SCAN		
Data completeness=	0.995	Theta(max)= 28.386	
R(reflections)=	0.0300(3017)	wR2(reflections)=	0.1158(3459)
S =	0.925	Npar=	182

6.2 X-Ray Crystallography data of compound 4q

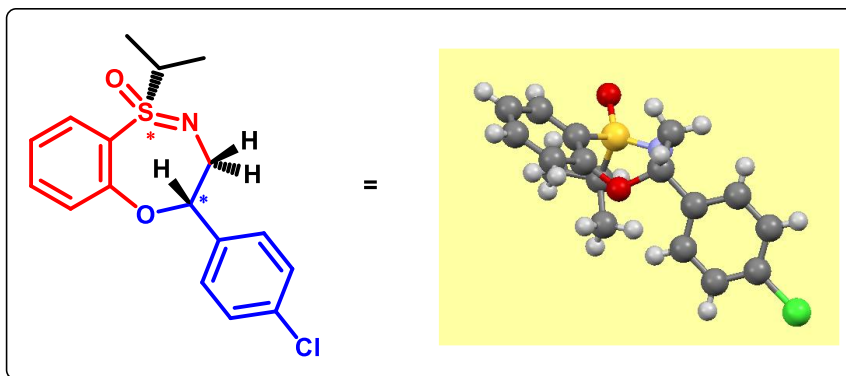


Figure S14- ORTEP diagram drawn with 50% ellipsoid for non-H atoms of the crystal structure of compound **4q** determined at 273K (CCDC deposition No. 2113503)

Single-crystal diffraction analysis data were collected at 273.15K with a Bruker APEX-II CCD Duo diffractometer (operated at 1500 W power: 50 kV, 30 mA) using graphite monochromatic Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$). Sample preparation for crystal growth- The 5 mg compound (3g) was taken in a 1 ml HPLC glass vial & compound was dissolved in 1ml acetonitrile solvent (lower solubility). This HPLC vial was placed in a 15ml glass vial which already contains 2-3ml of hexane. Then the vial was closed by wrapping it with aluminium foil & one small niddle was attached to the neck. Kept it for 4 to 5 days for growing of the crystal. Slow evaporation at room temperature in a dark place till crystals form.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) AB_176A

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: AB_176A

Bond precision: C-C = 0.0021 Å Wavelength=0.71073

Cell: a=8.4820 (5) b=9.2719 (6) c=20.5792 (12)
alpha=90 beta=95.217 (2) gamma=90

Temperature: 273 K

	Calculated	Reported
Volume	1611.73 (17)	1611.73 (17)
Space group	P 21/n	P 21/n
Hall group	-P 2yn	-P 2yn
Moiety formula	C17 H18 Cl N O2 S	C17 H18 Cl N O2 S
Sum formula	C17 H18 Cl N O2 S	C17 H18 Cl N O2 S
Mr	335.83	335.83
Dx, g cm ⁻³	1.384	1.384
Z	4	4
Mu (mm ⁻¹)	0.373	0.373
F000	704.0	704.0
F000'	705.34	
h, k, lmax	11, 12, 27	11, 12, 27
Nref	4039	4027
Tmin, Tmax	0.914, 0.949	0.914, 0.949
Tmin'	0.891	

Correction method= # Reported T Limits: Tmin=0.914 Tmax=0.949
AbsCorr = MULTI-SCAN

Data completeness= 0.997 Theta(max)= 28.365

R(reflections)= 0.0350 (3461) wR2(reflections)=
S = 0.895 Npar= 201 0.1292 (4027)

1. K. Oyaizu, T. Mikami, F. Mitsuhashi, E. Tsuchida, *Macromolecules*, 2002, **35**, 67–78.
2. S. Ponra, A. Nyadanu, N. Pan, E. Martinand-Lurin, A. Savy, M. Vitale, L. E. K. L. Grimaud, *Org. Process Res. Dev.* 2020, **24**, 5, 827–834.
3. A. Tota, M. Zenzola, S. J. Chawner, S. St. John-Campbell, C. Carlucci, G. Romanazzi, L. Degennaro, J. A. Bullb, R. Luisia, *Chem. Commun.* 2017, **53**, 348-351.
4. D. Limnios, C. G. Kokotos, *J. Org. Chem.* 2014, **79**, 4270–4276.
5. O- C. Finch, D. P. Furkert, M.A. Brimble, *Tetrahedron*, 2014, **70**, 590-596.
6. L. Chen, Z. Wang, Y. Wang, L. Hao, X. Xu, G. Wu, Y. Ji, *Org. Biomol. Chem.*, 2022, **20**, 887–894.
7. C. Wu, R. Huang, M. Zhang, Z. Chen, *J. Org. Chem.* 2020, **85**, 841–850.

Supporting Information

Transition metal-free, base mediated one-pot approach for the construction of benzo[*b*] [1,4,5]oxathiazepine 1-oxide core

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1. ¹H, ¹³C, ¹⁹F, DEPT-135, 2D NMR, diastereomeric ratio calculation, HRMS and HPLC data of Compounds

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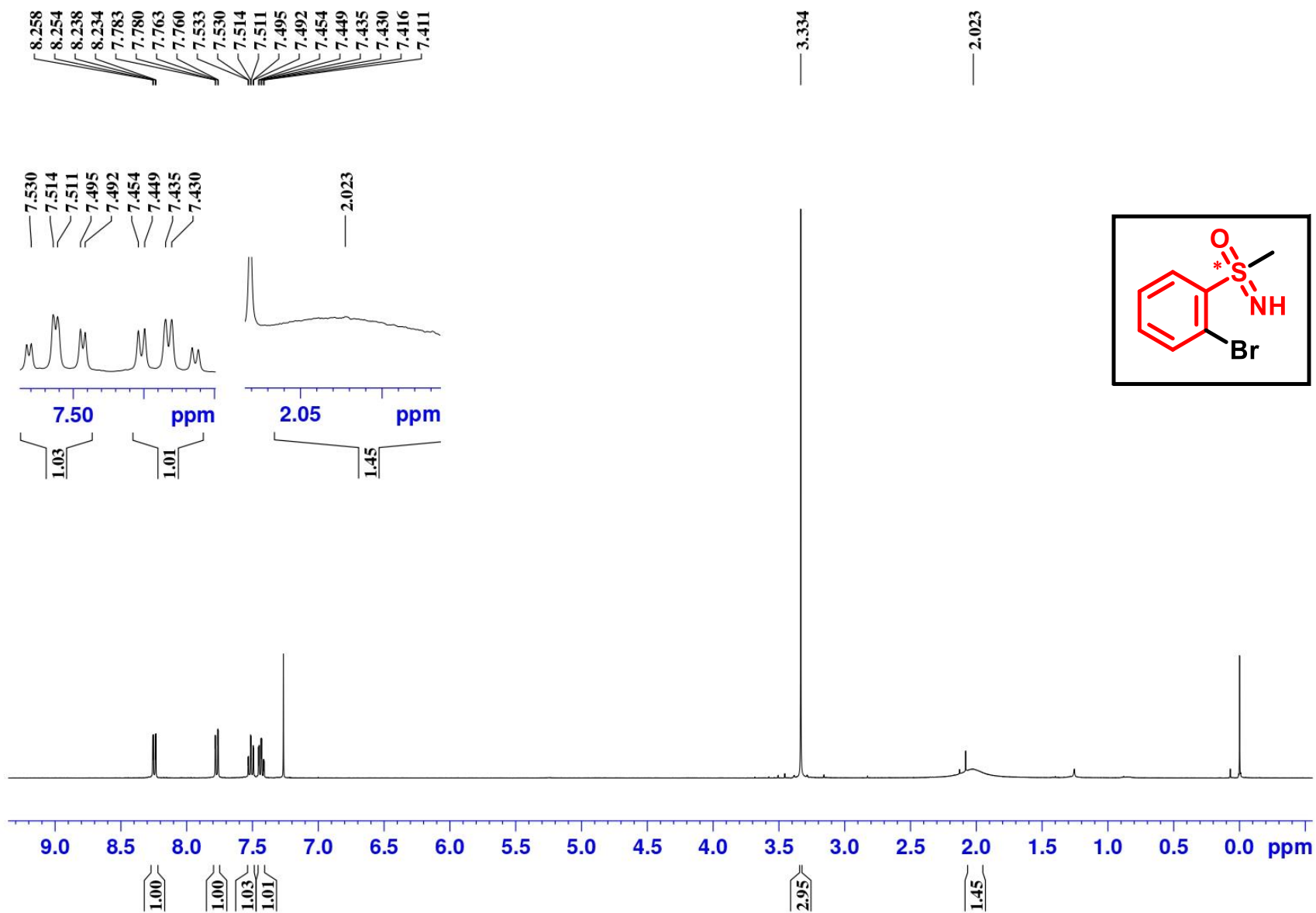


Fig S-1: ^1H NMR Spectra of Compound 2a (400 MHz, CDCl_3)

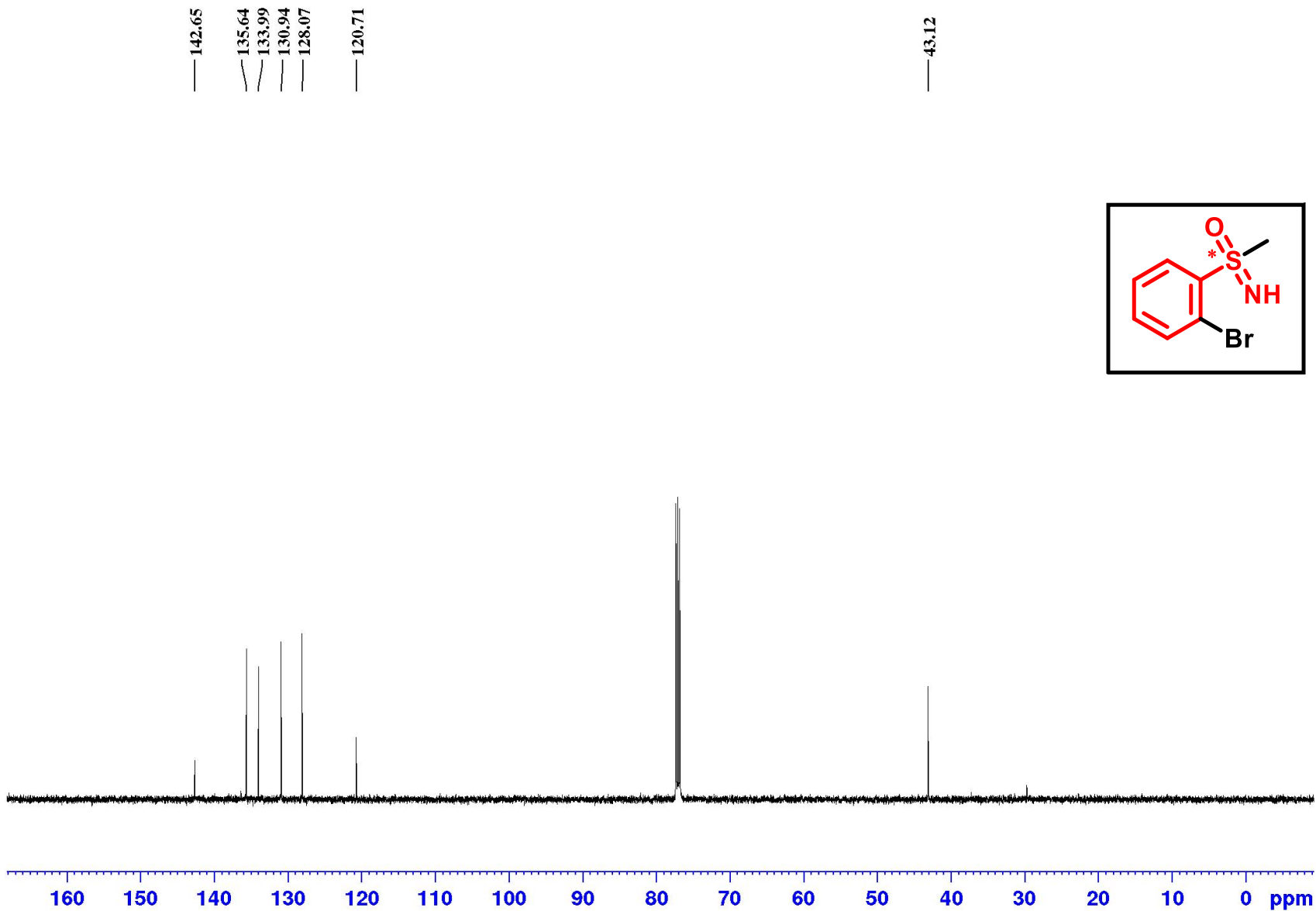


Fig S-2: ^{13}C NMR Spectra of Compound **2a** (125 MHz, CDCl_3)

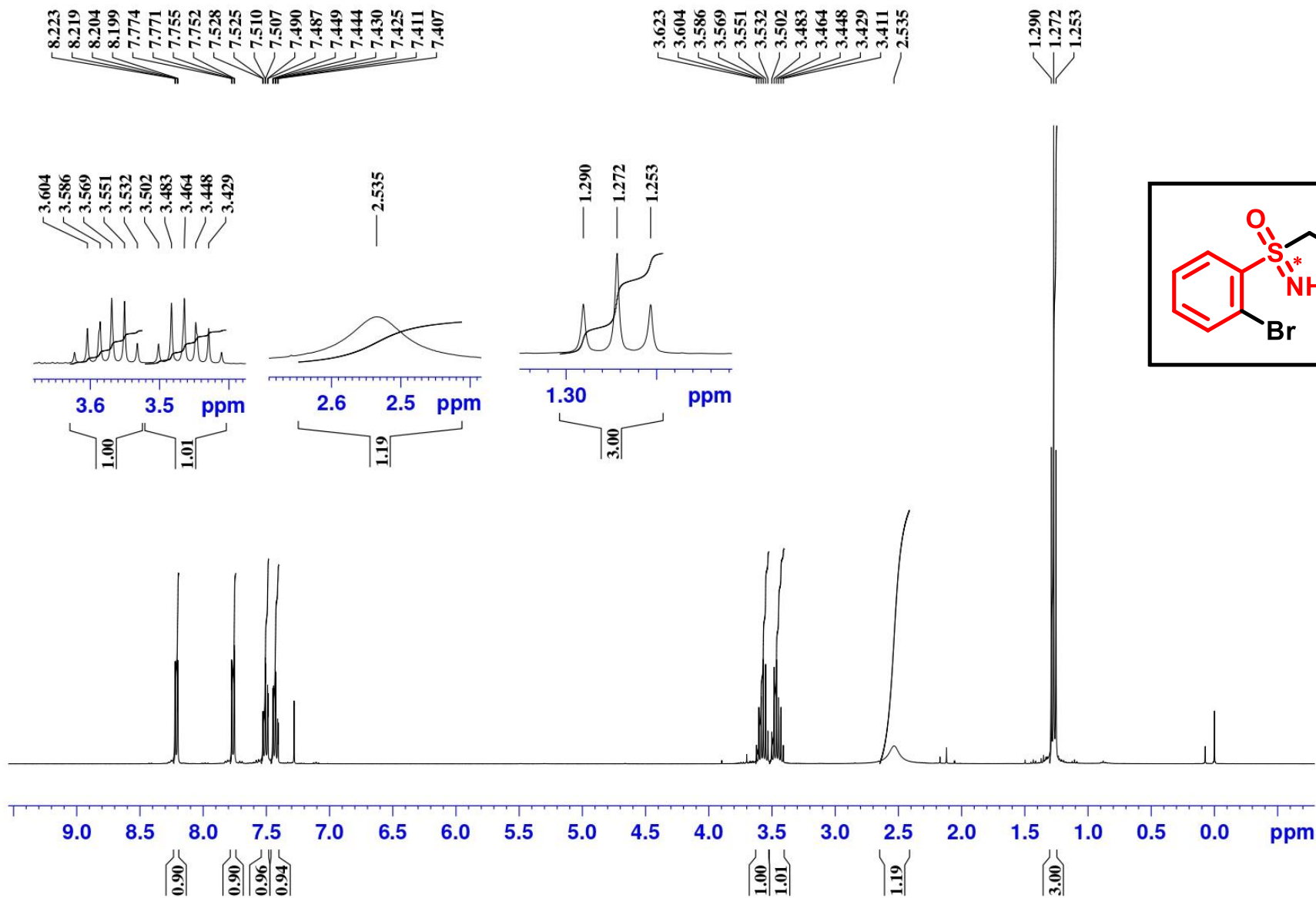


Fig S-3: ^1H NMR Spectra of Compound 2b (400 MHz, CDCl_3)

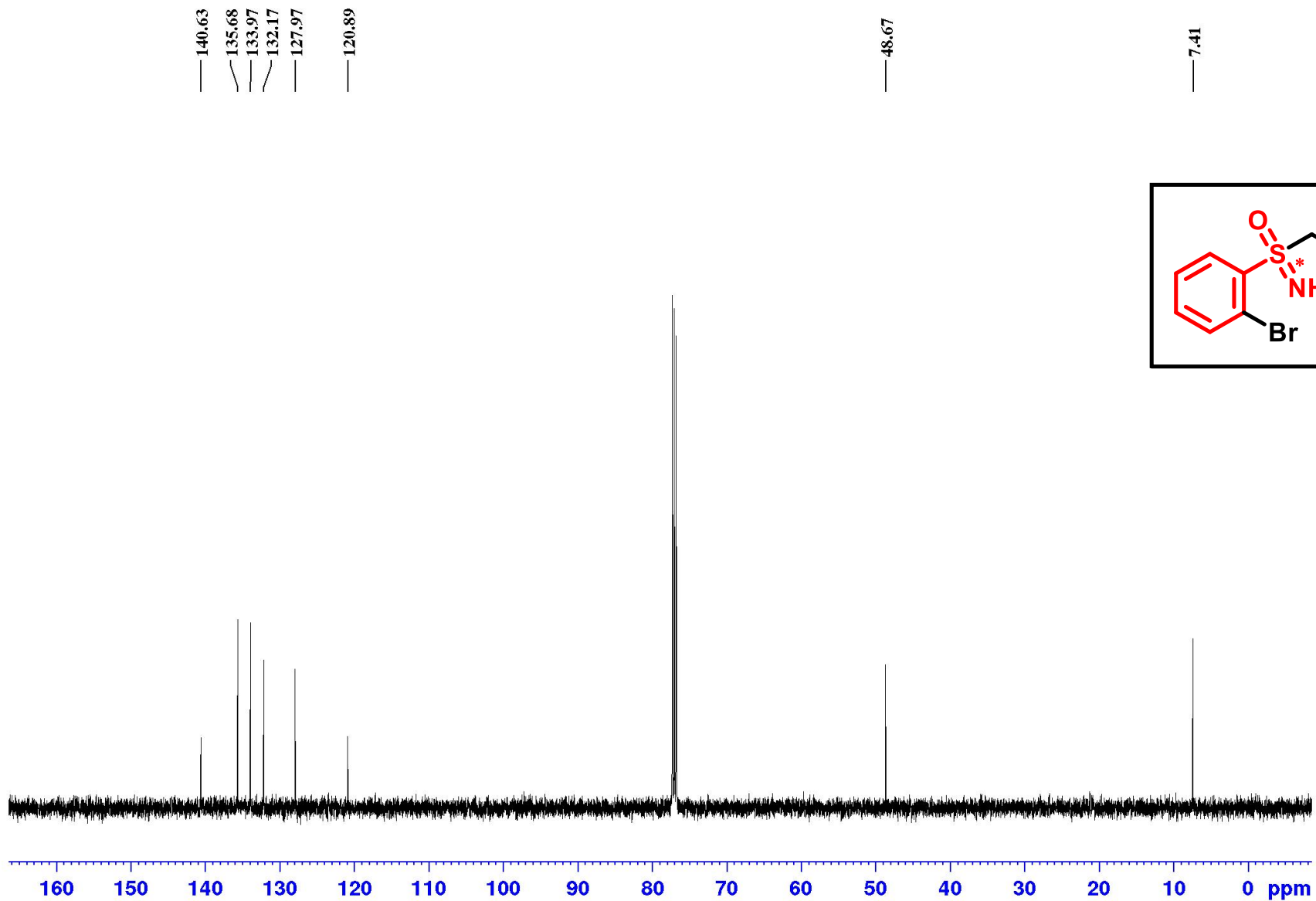


Fig S-4: ^{13}C NMR Spectra of Compound 2b (125 MHz, CDCl_3)

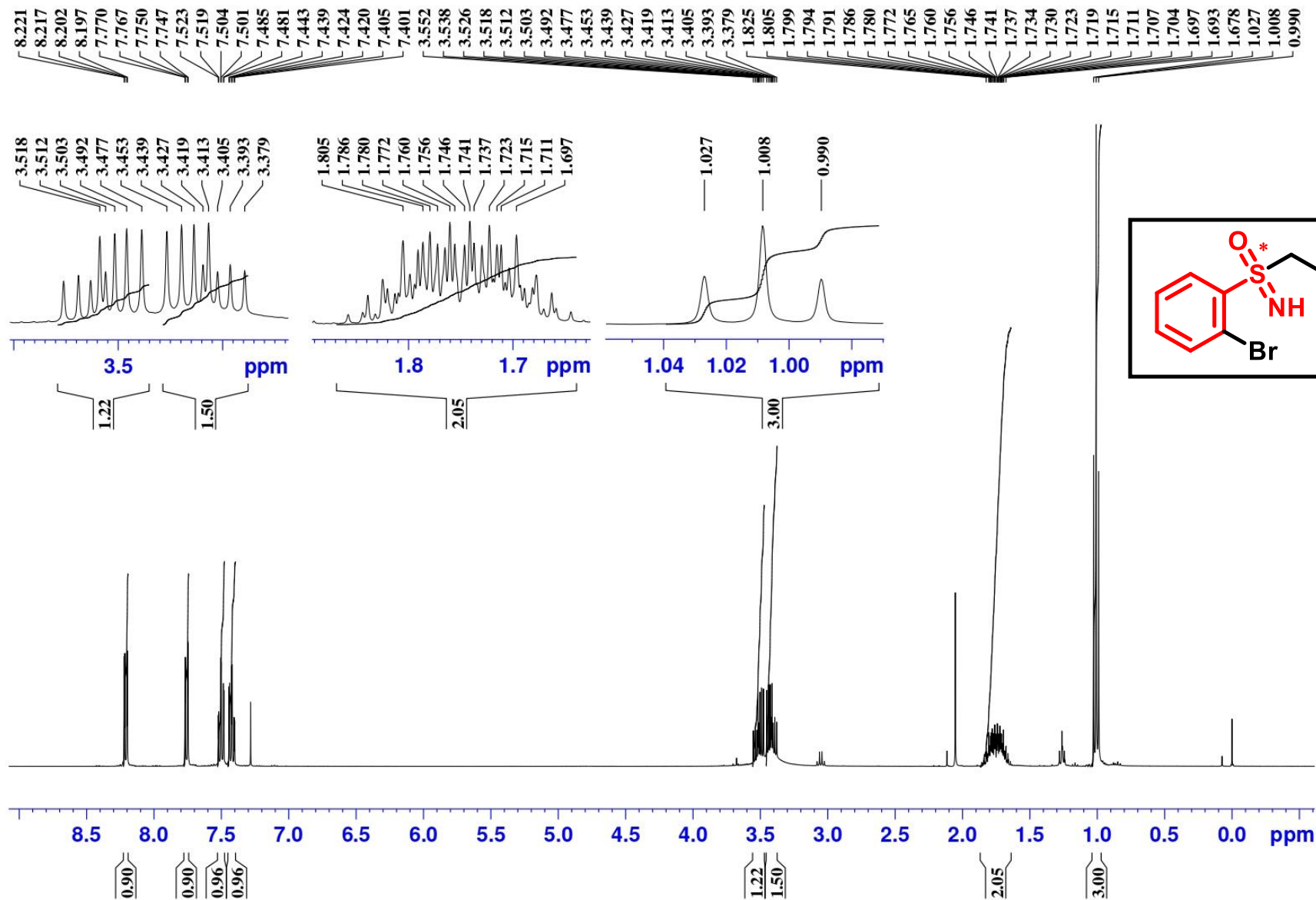


Fig S-5: ^1H NMR Spectra of Compound 2c (400 MHz, CDCl_3)

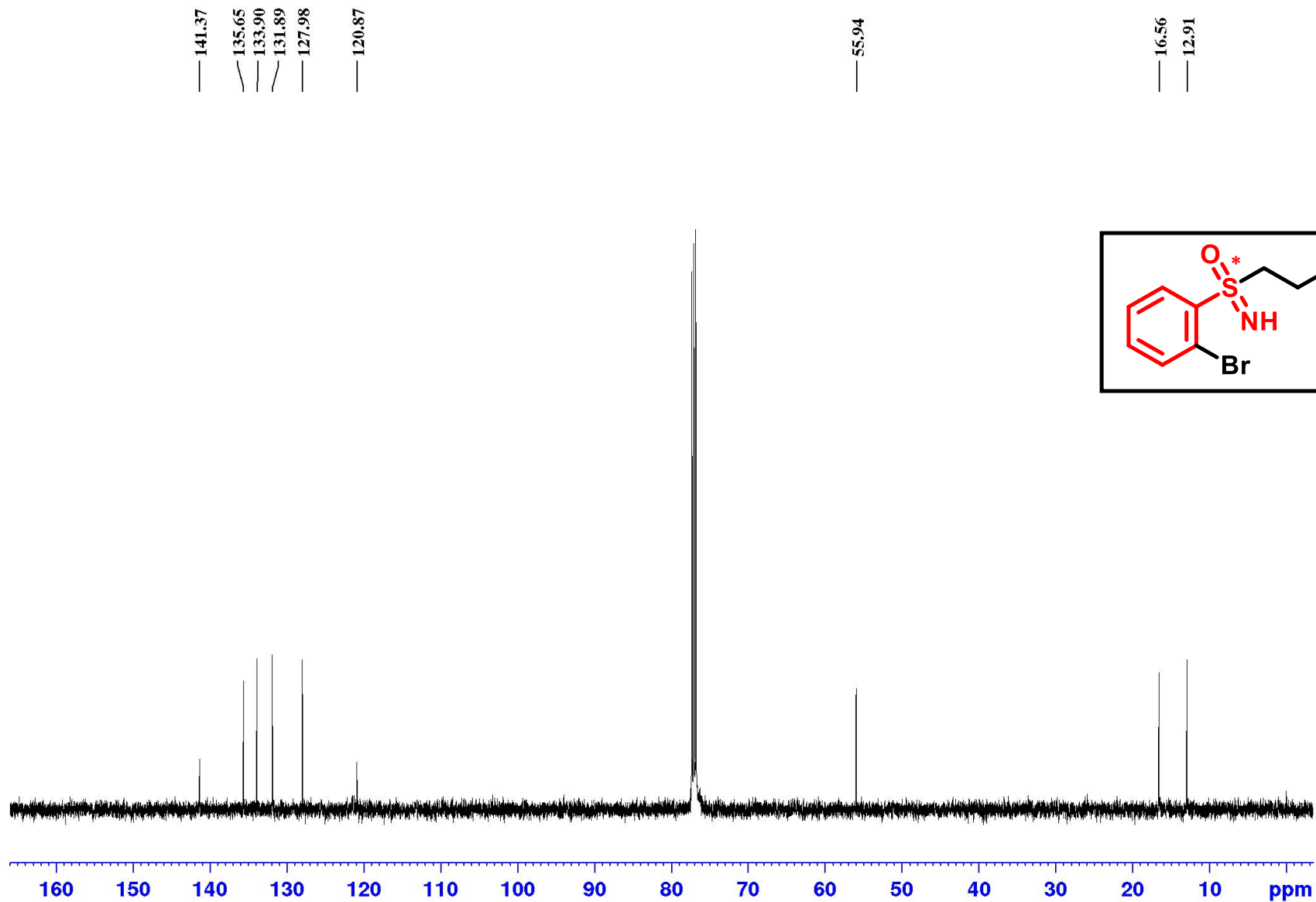


Fig S-6: ^{13}C NMR Spectra of Compound **2c** (125 MHz, CDCl_3)

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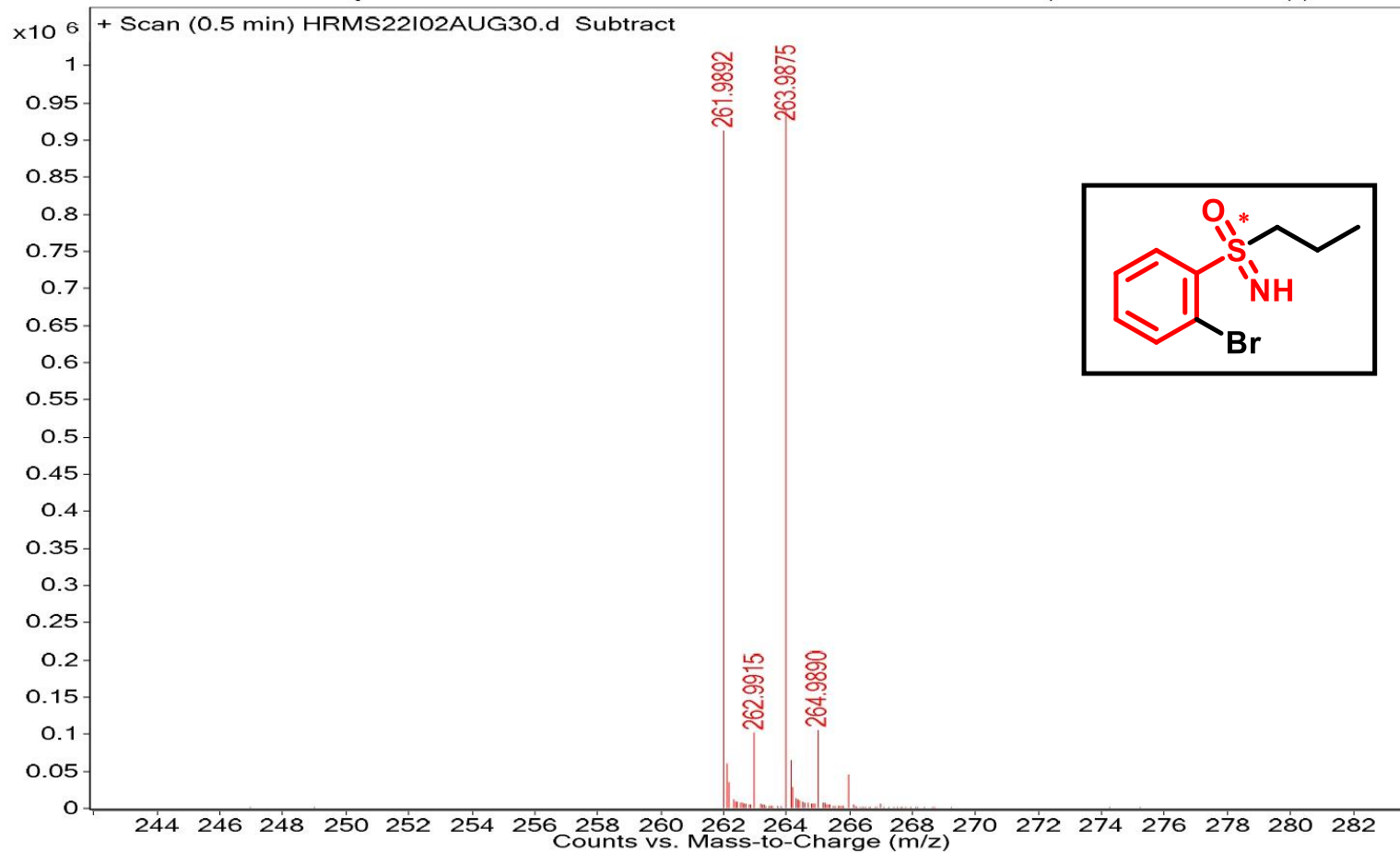


Fig S-7: HRMS report of Compound **2c**

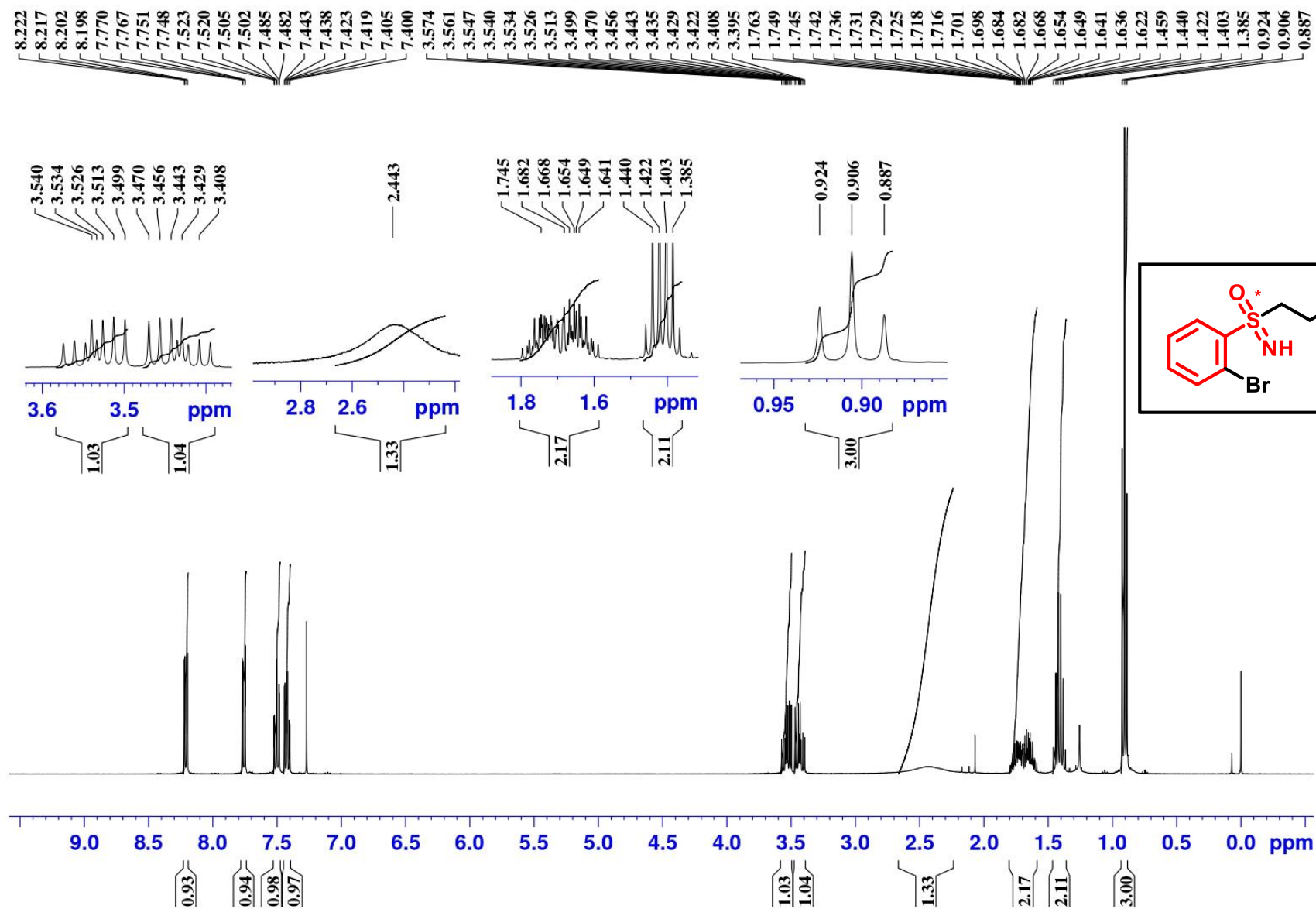


Fig S-8: ^1H NMR Spectra of Compound **2d** (400 MHz, CDCl_3)

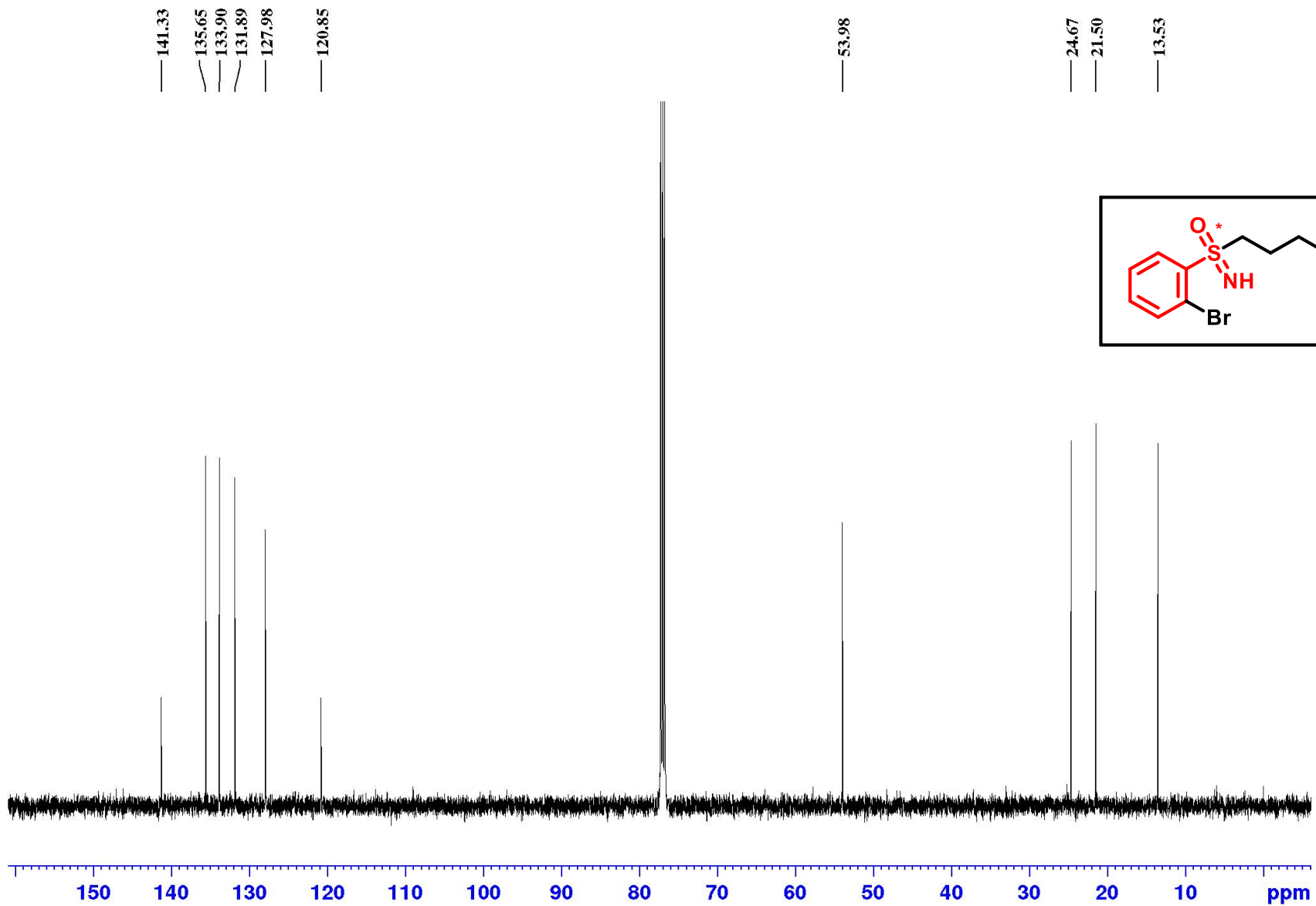


Fig S-9: ^{13}C NMR Spectra of Compound **2d** (125 MHz, CDCl_3)

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Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
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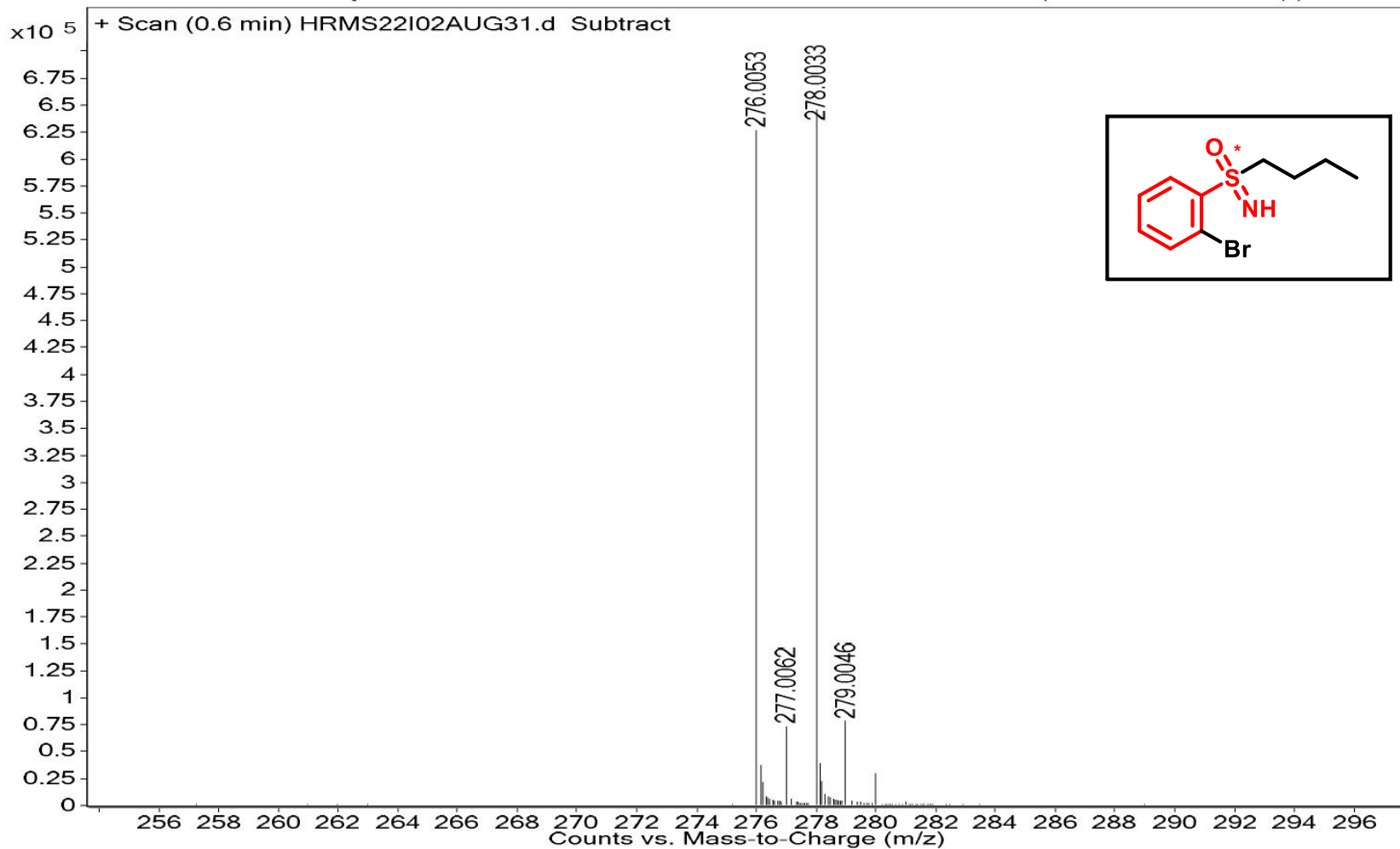


Fig S-10: HRMS report of Compound **2d**

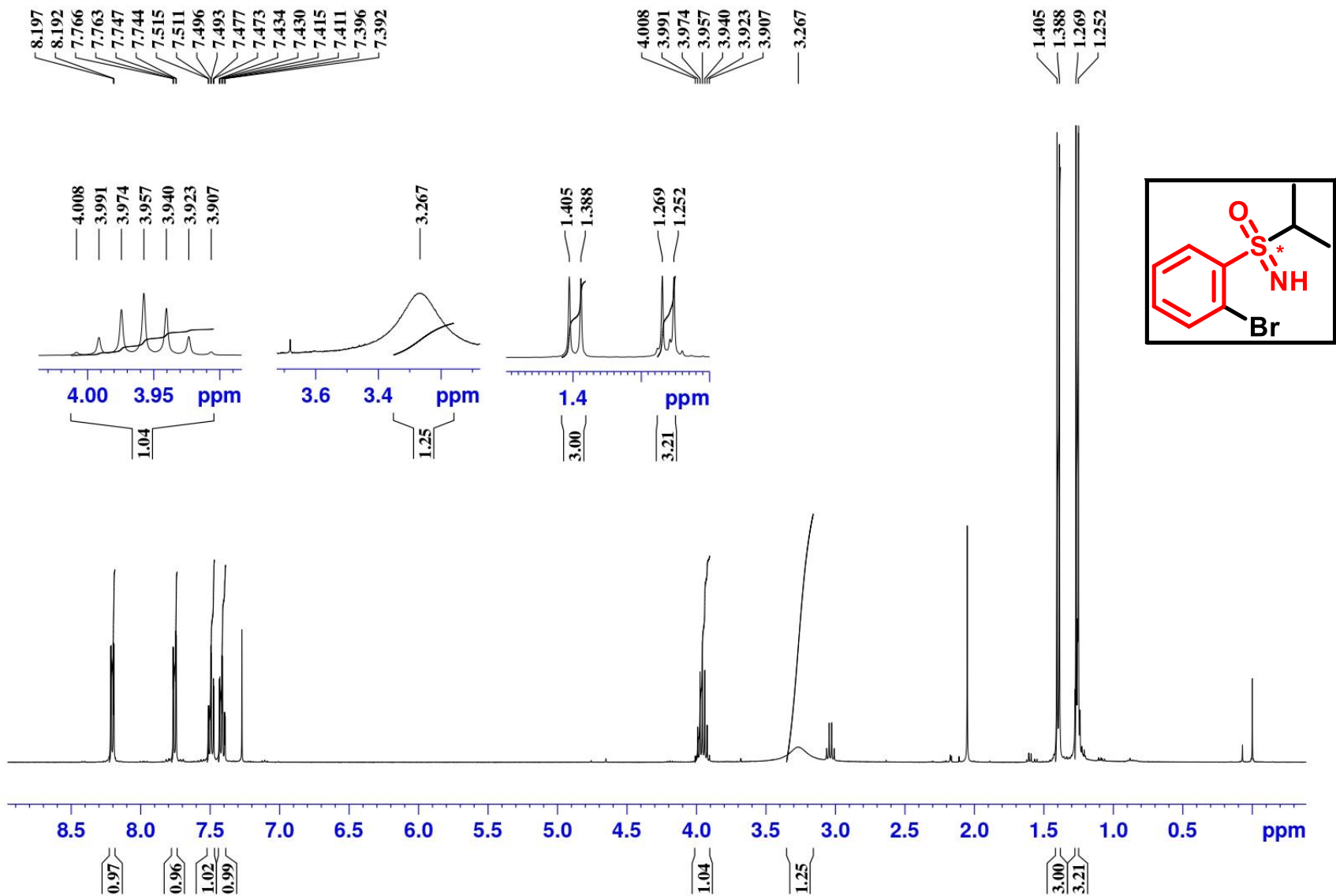


Fig S-11: ^1H NMR Spectra of Compound 2e (400 MHz, CDCl_3)

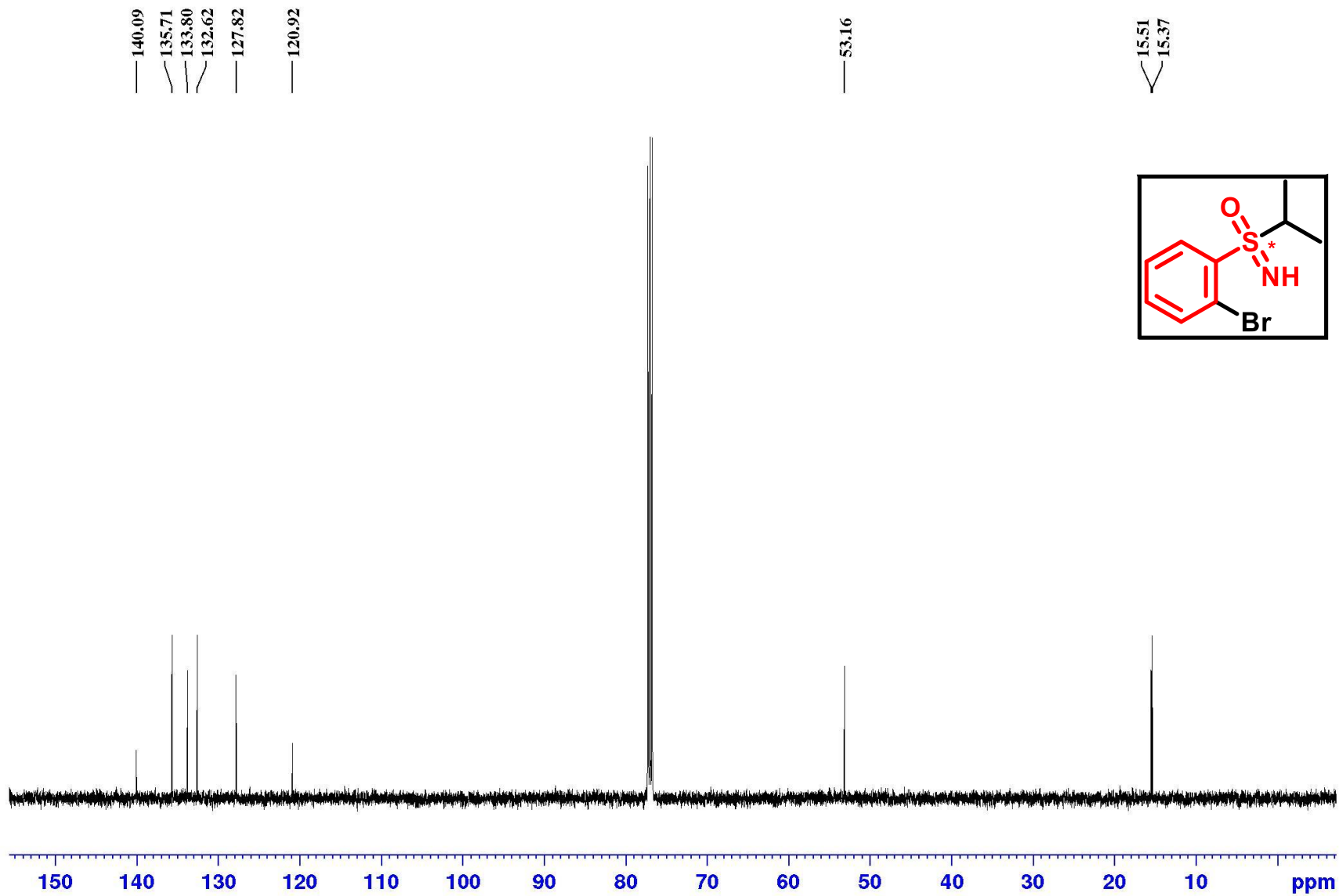


Fig S-12: ^{13}C NMR Spectra of Compound **2e** (125 MHz, CDCl_3)

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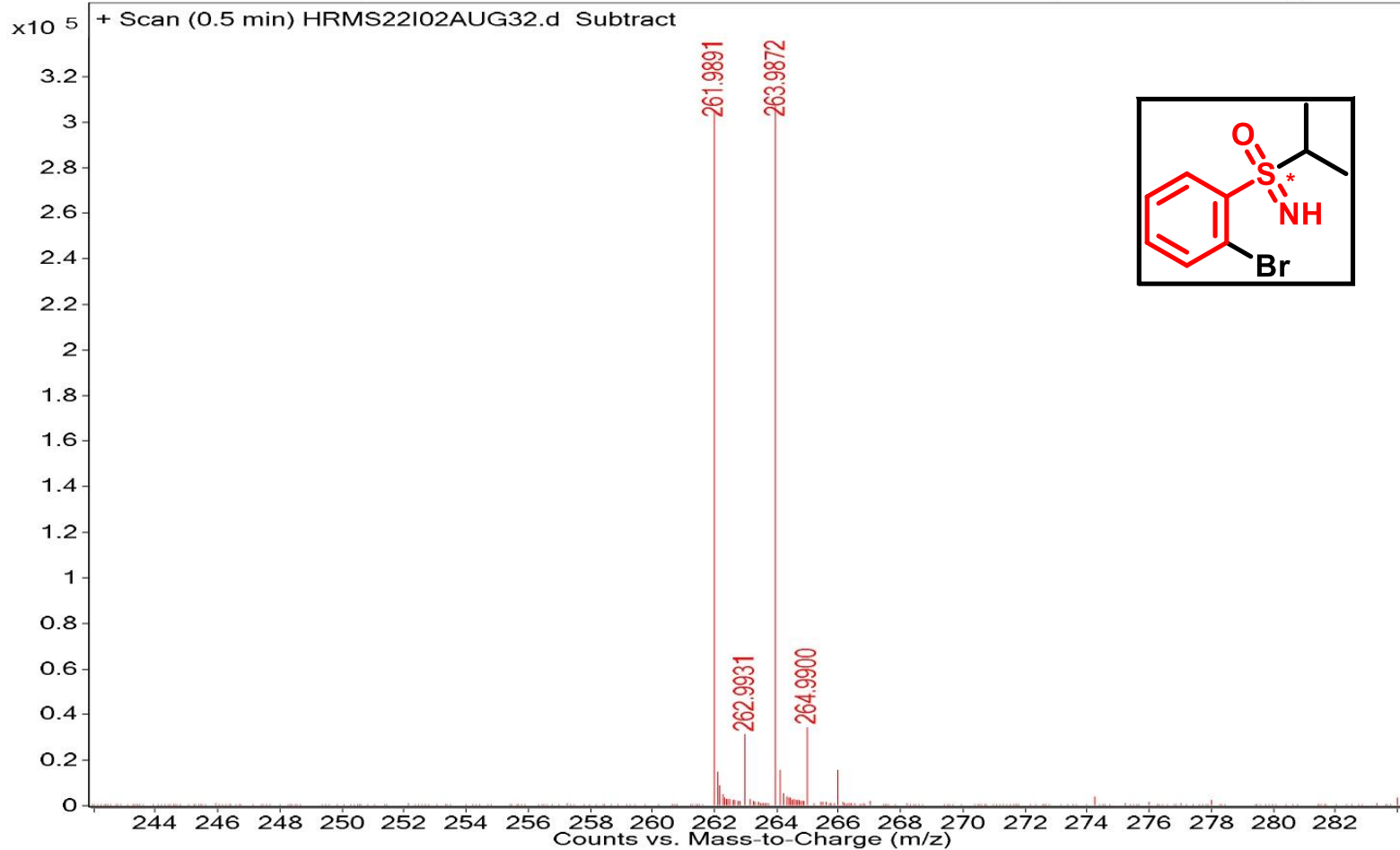


Fig S-13: HRMS report of Compound **2e**

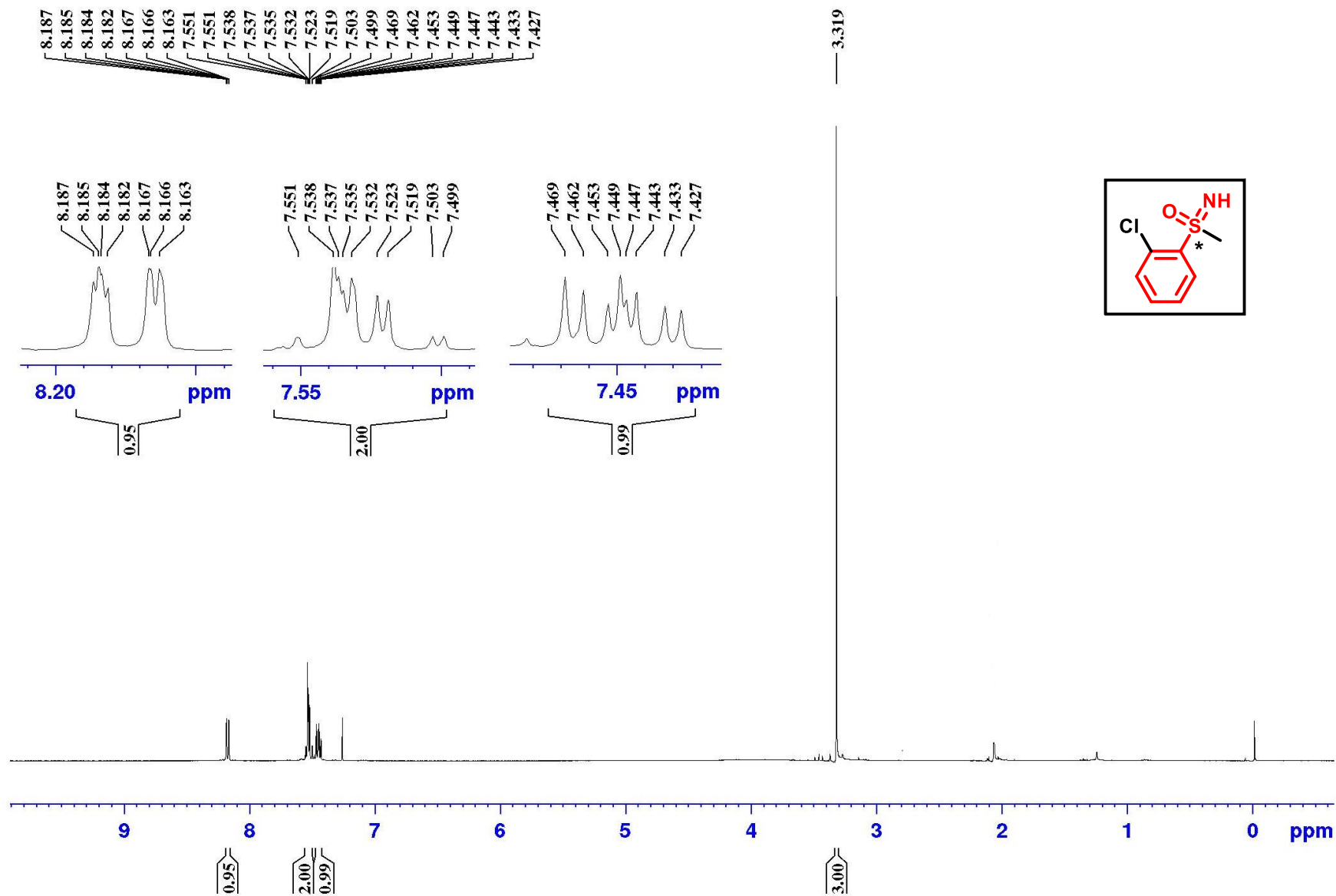


Fig S-14: ^1H NMR Spectra of Compound 2f (400 MHz, CDCl_3)

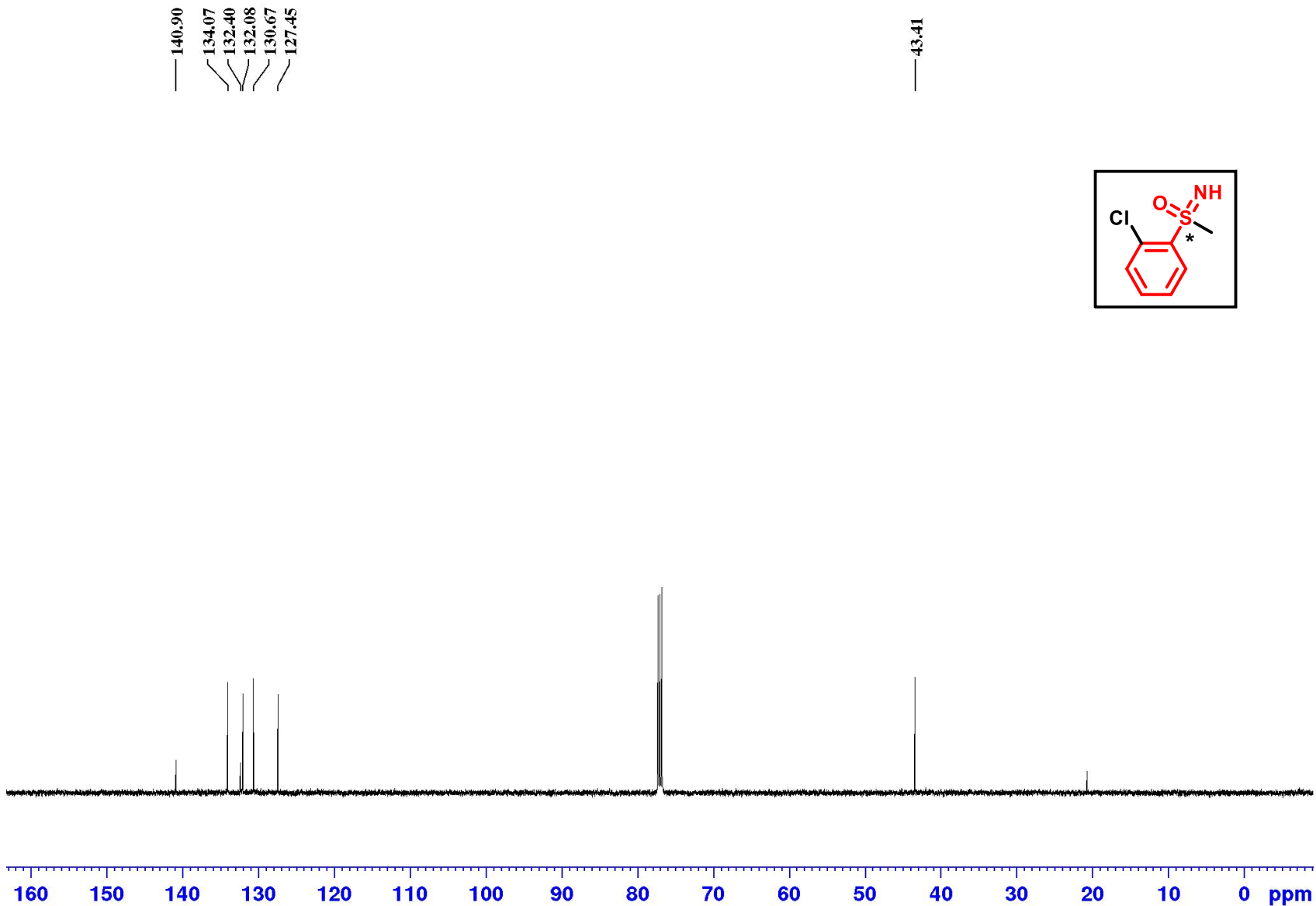


Fig S-15: ^{13}C NMR Spectra of Compound **2f** (125 MHz, CDCl_3)

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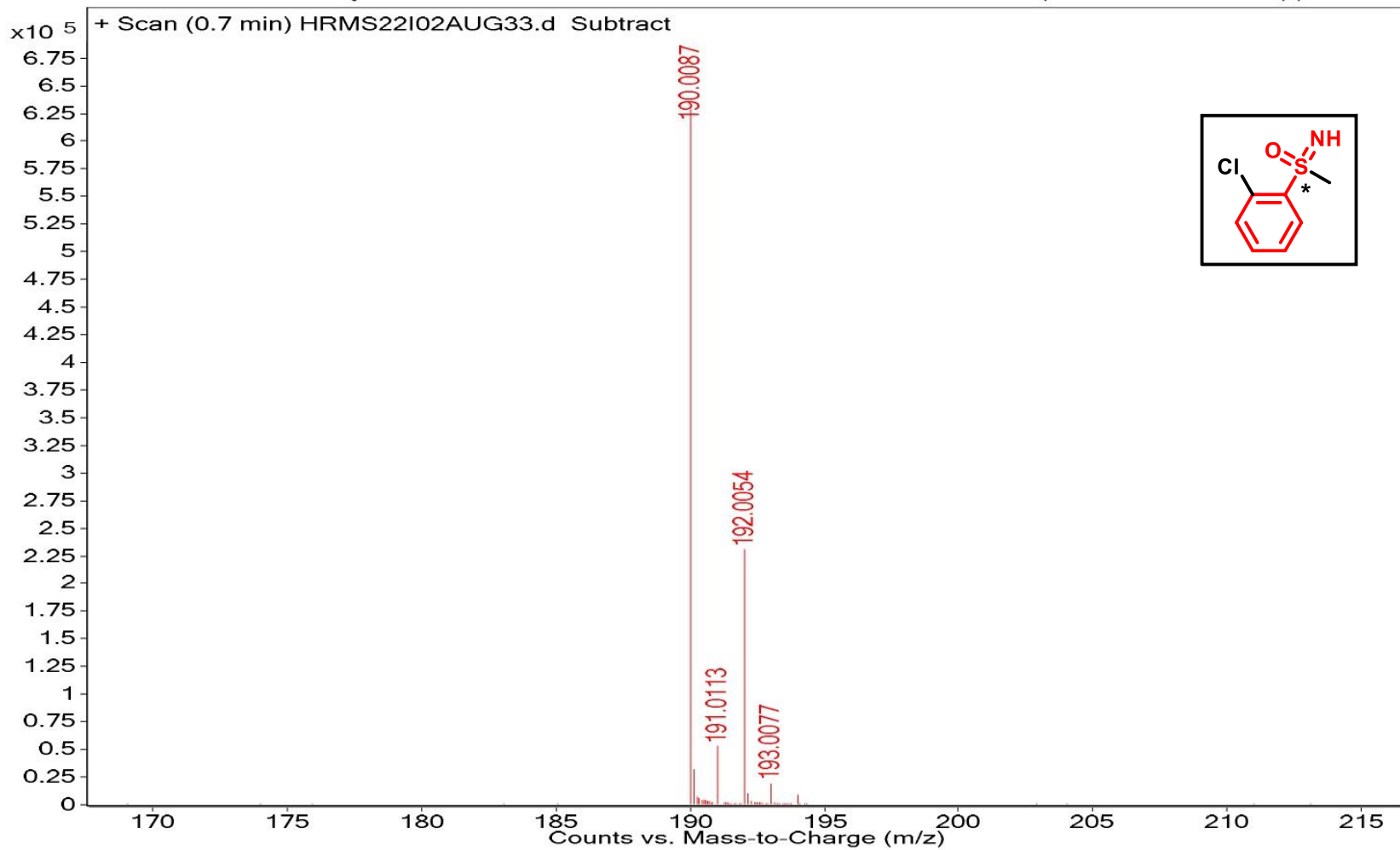


Fig S-16: HRMS report of Compound **2f**

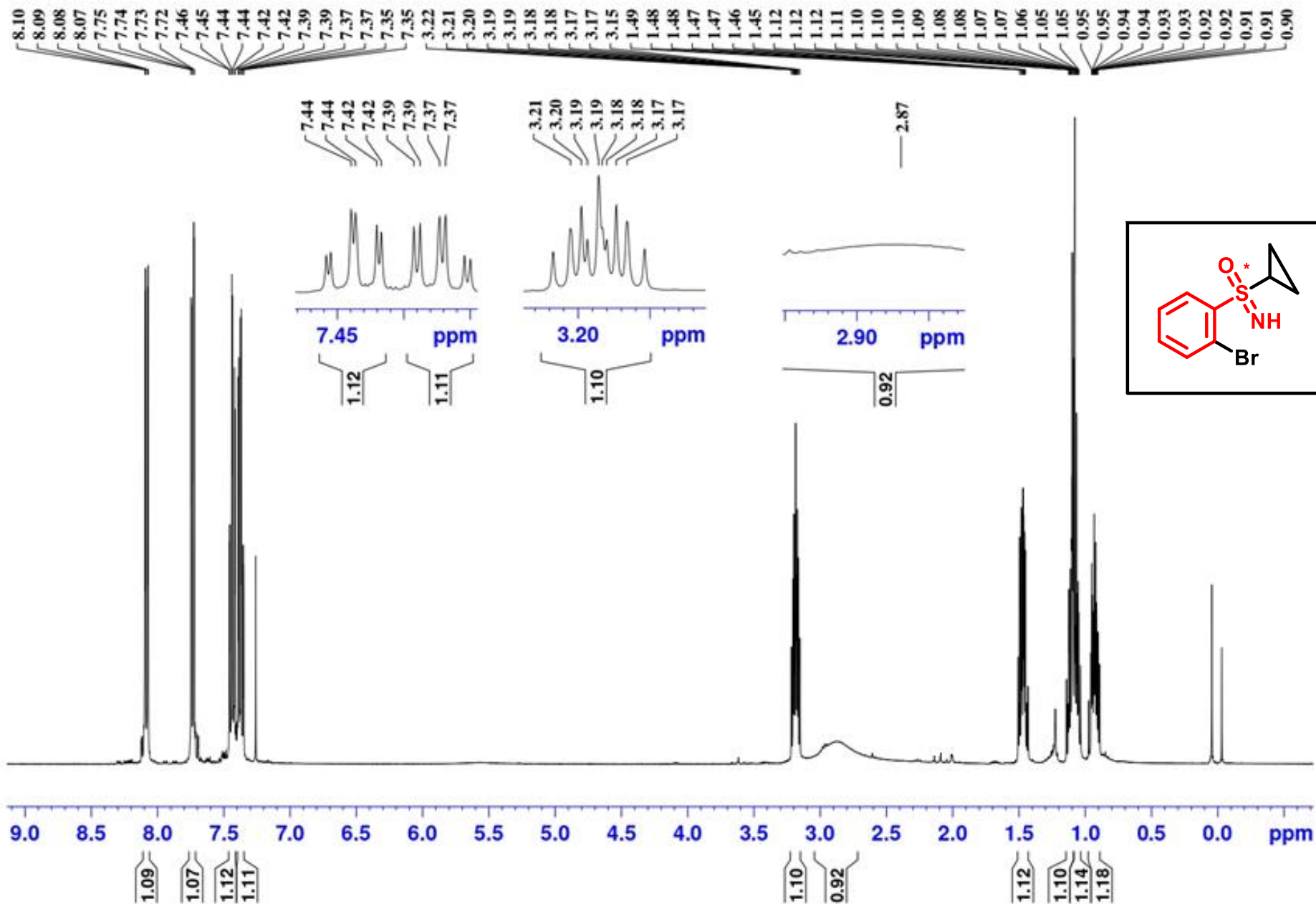


Fig S-17: ^1H NMR Spectra of Compound **2g** (400 MHz, CDCl_3)

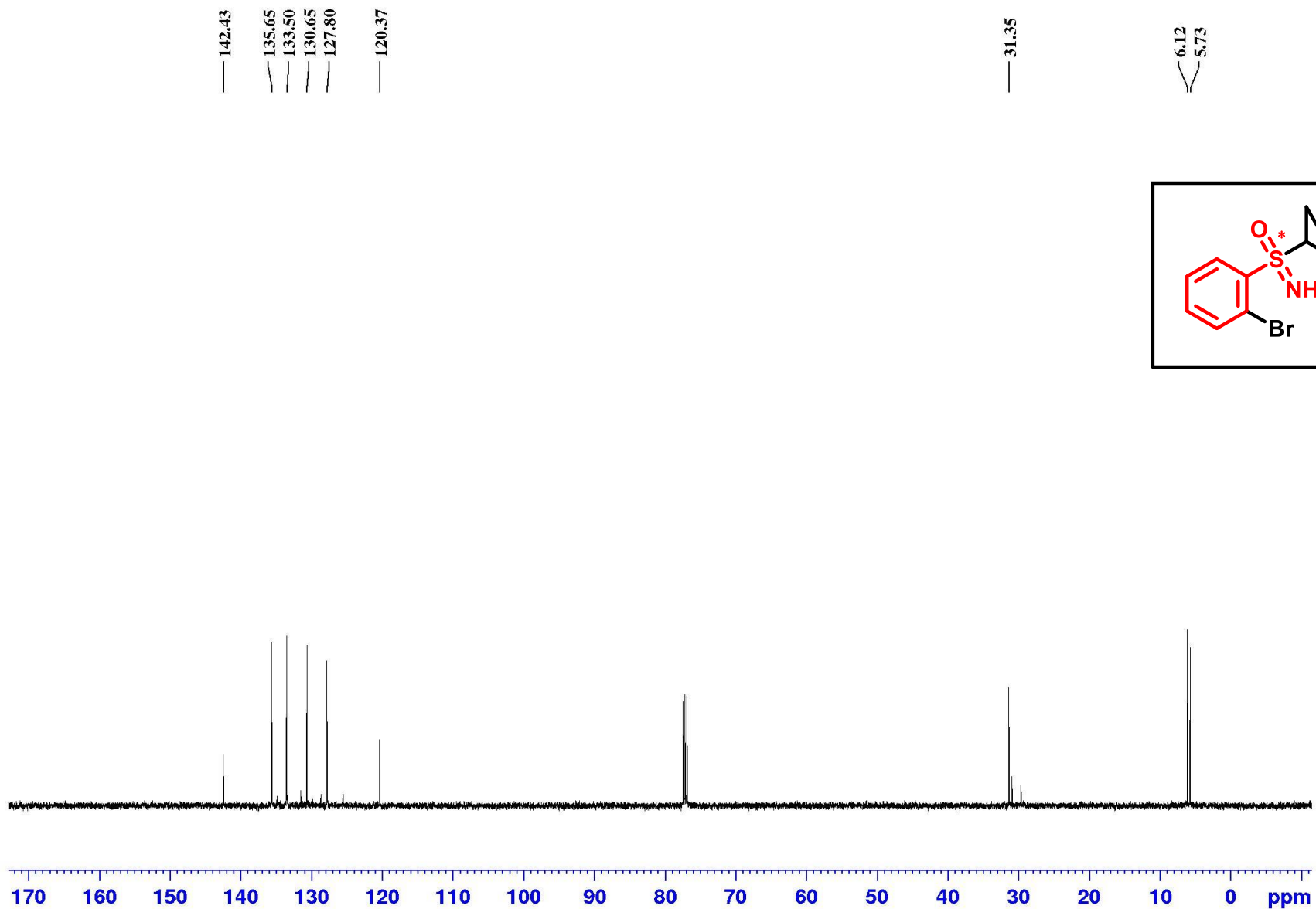


Fig S-18: ^{13}C NMR Spectra of Compound 2g (125 MHz, CDCl_3)

Sample Name	2g	Position	Vial 35	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
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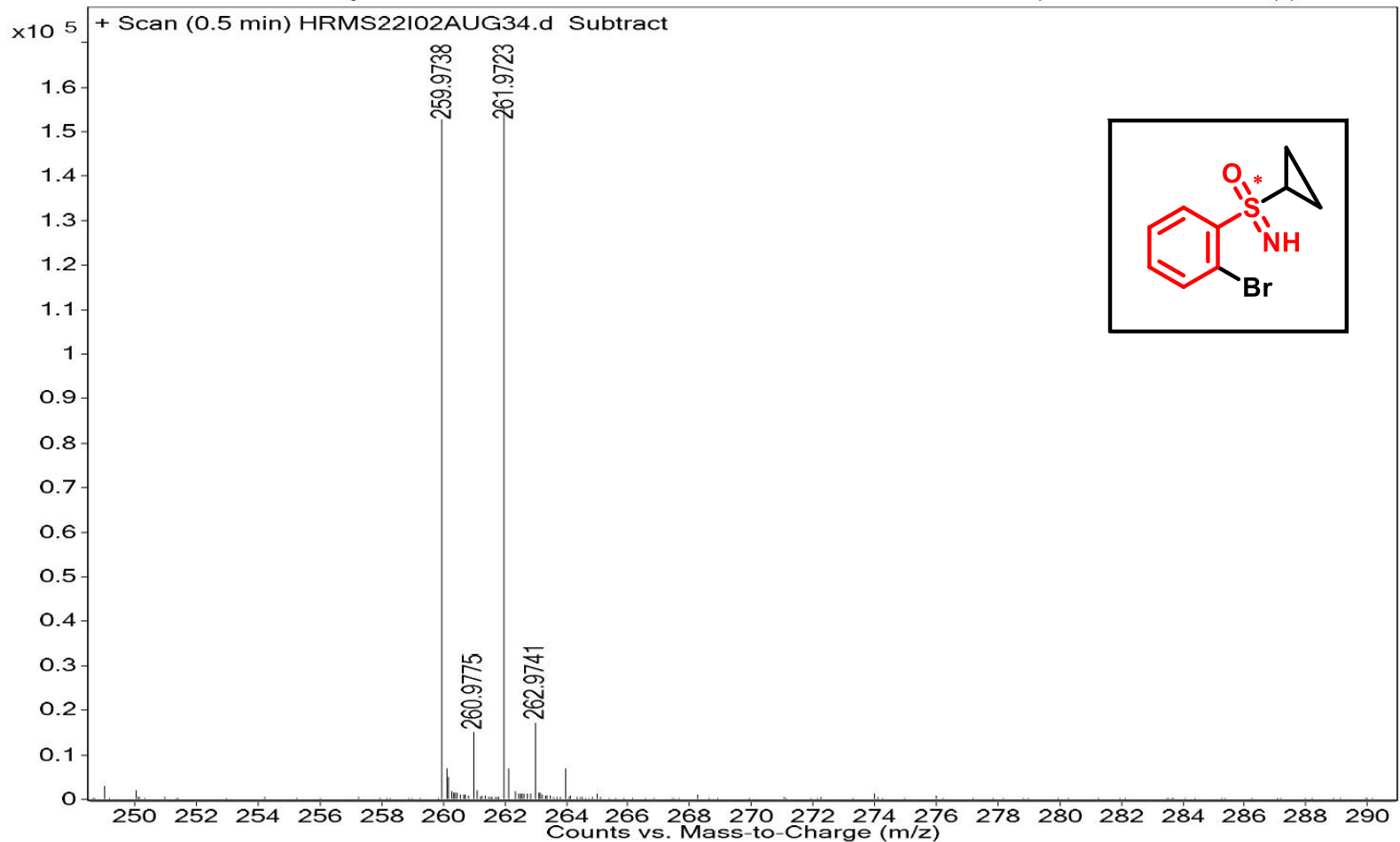


Fig S-19: HRMS report of Compound **2g**

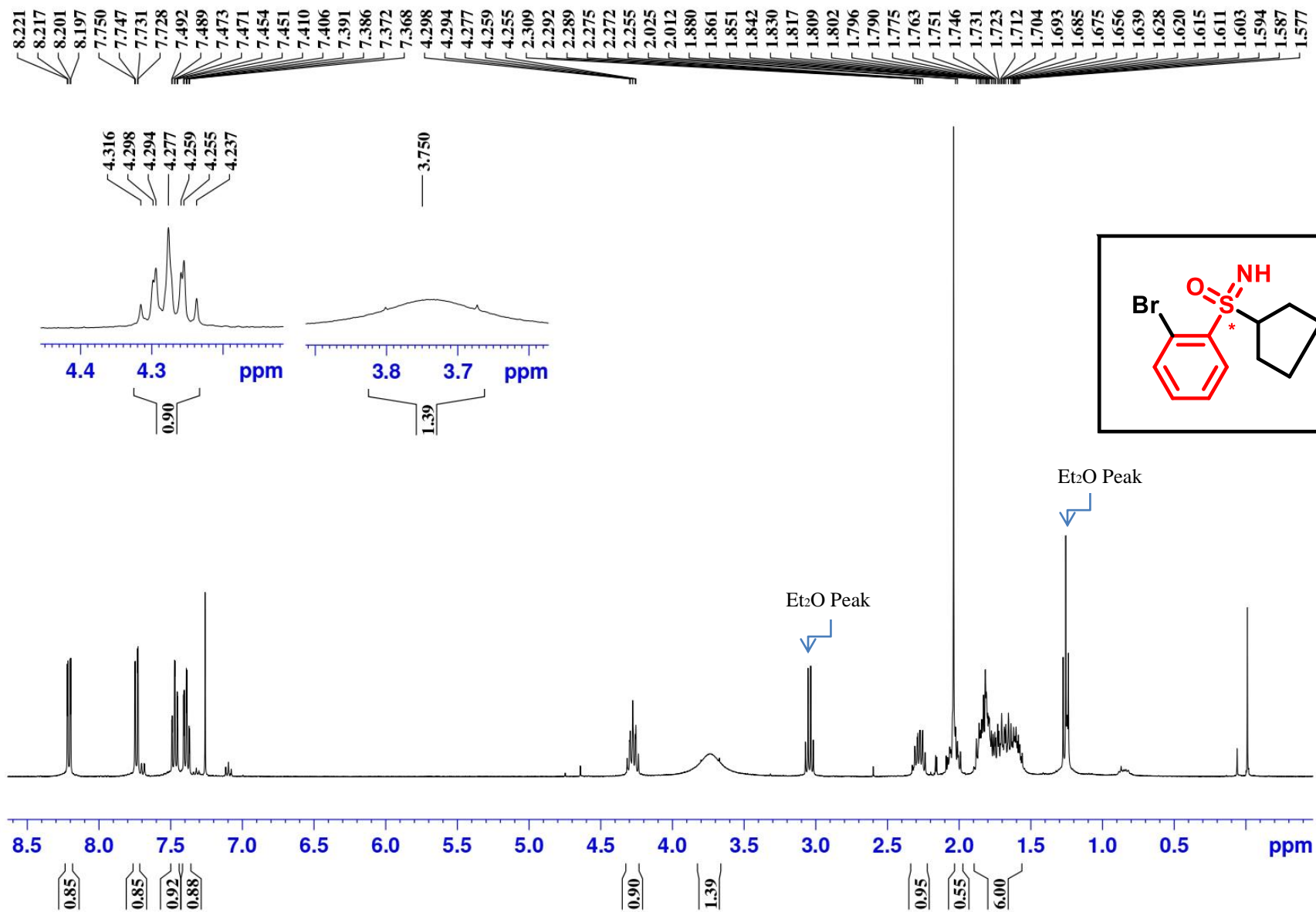


Fig S-20: ^1H NMR Spectra of Compound **2h** (400 MHz, CDCl_3)

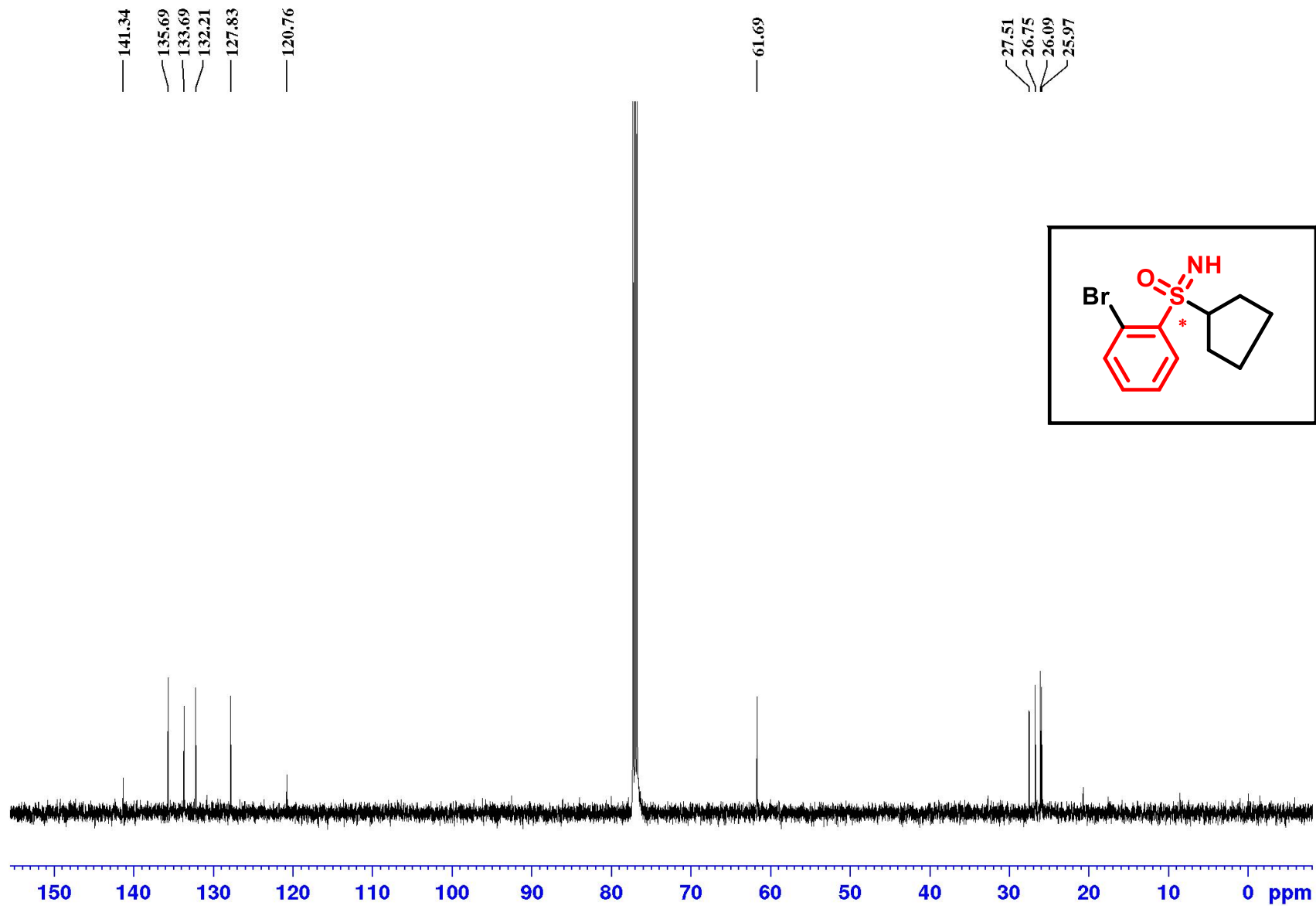


Fig S-21: ^{13}C NMR Spectra of Compound 2h (125 MHz, CDCl_3)

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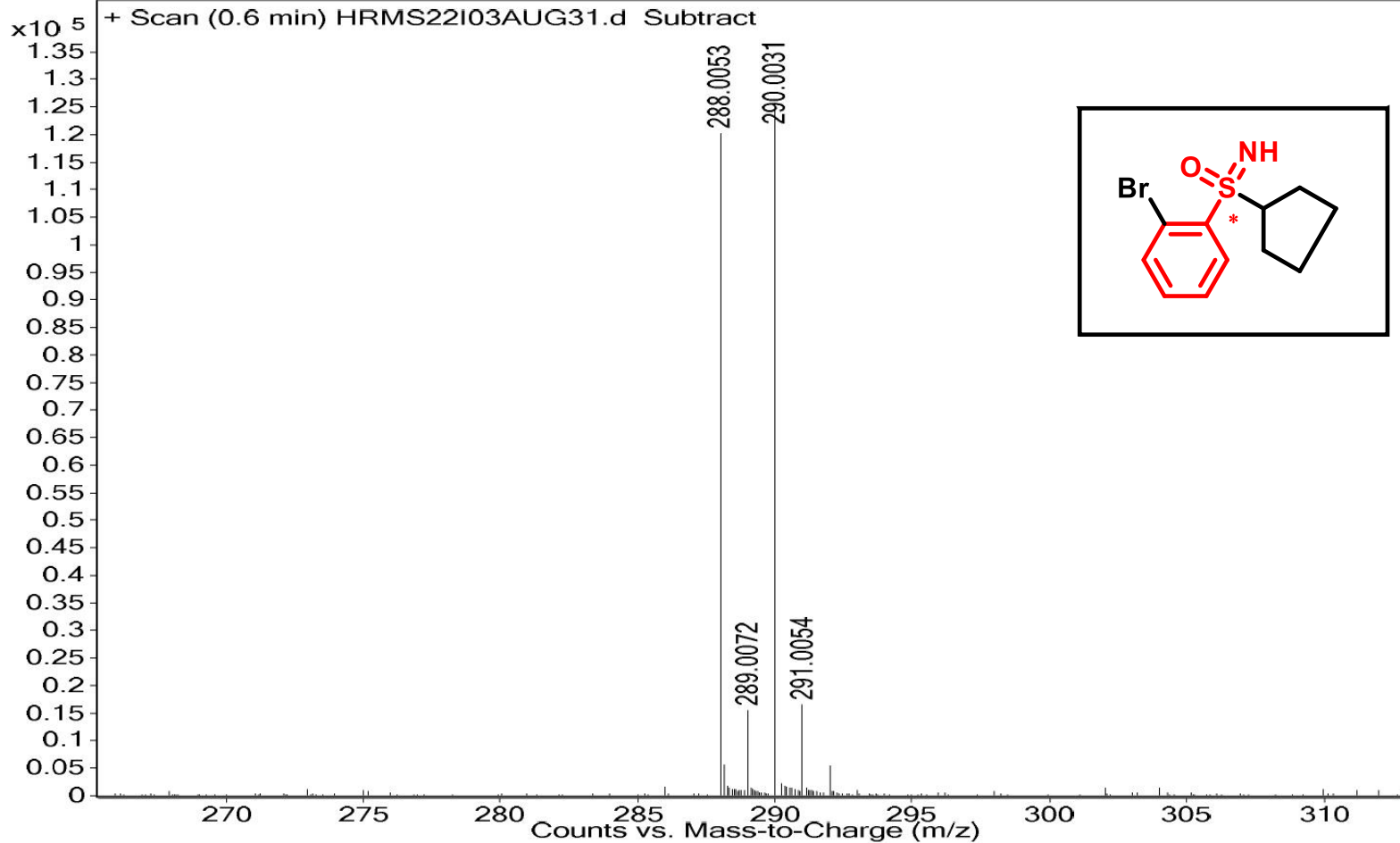


Fig S-22: HRMS report of Compound **2h**

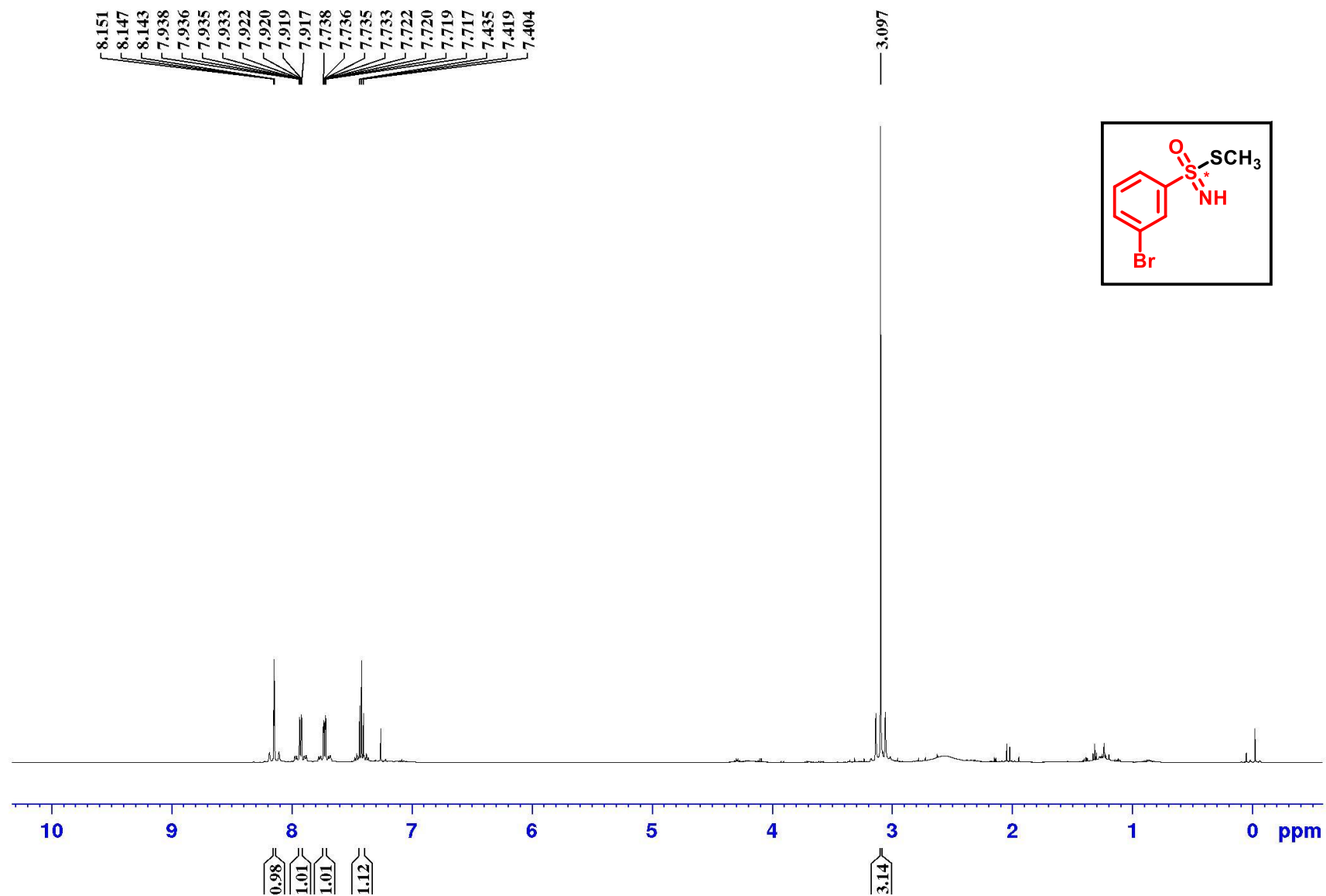


Fig S-23: ¹H NMR Spectra of Compound **2i** (500 MHz, CDCl₃)

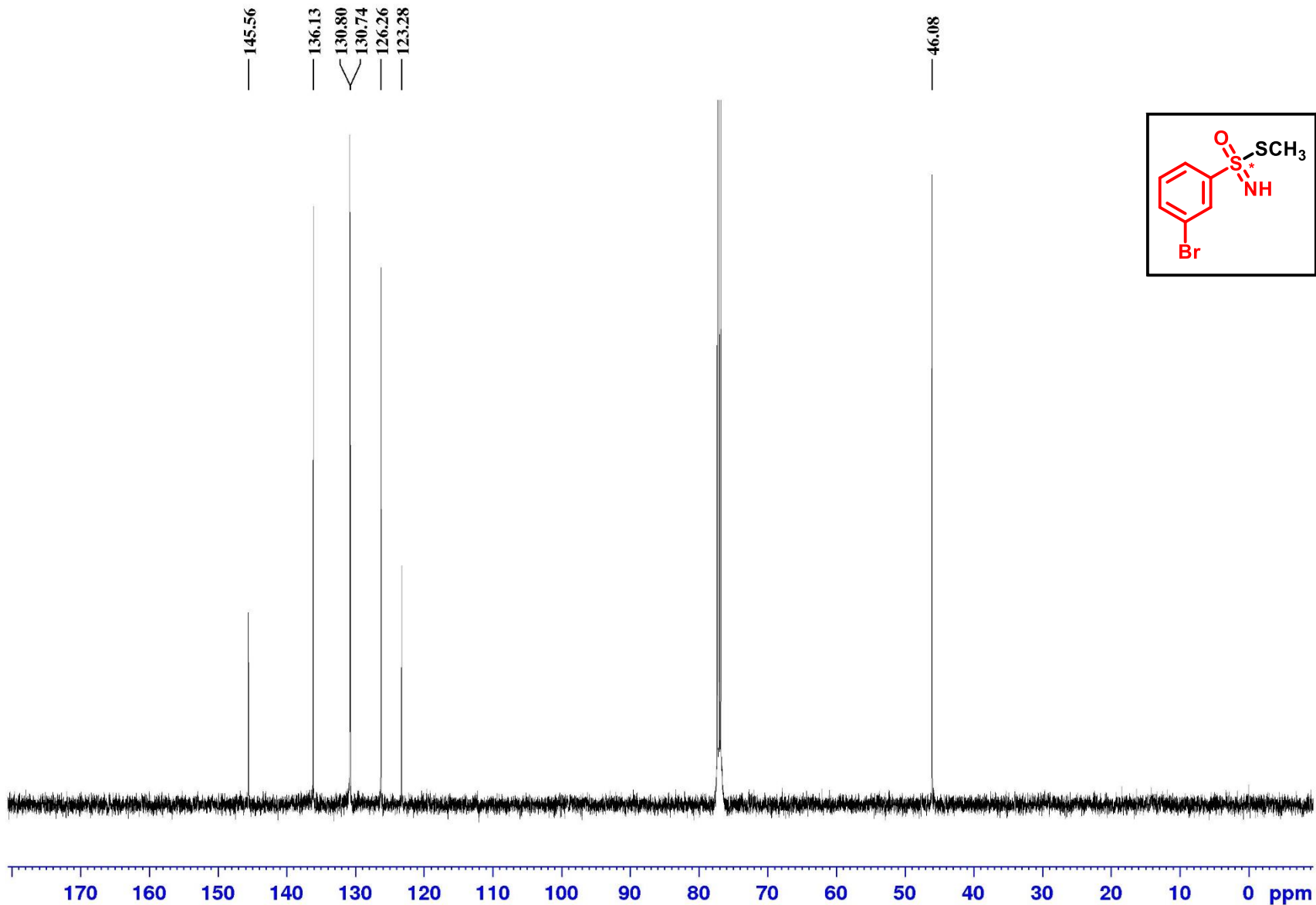


Fig S-24: ^{13}C NMR Spectra of Compound **2i** (125 MHz, CDCl_3)

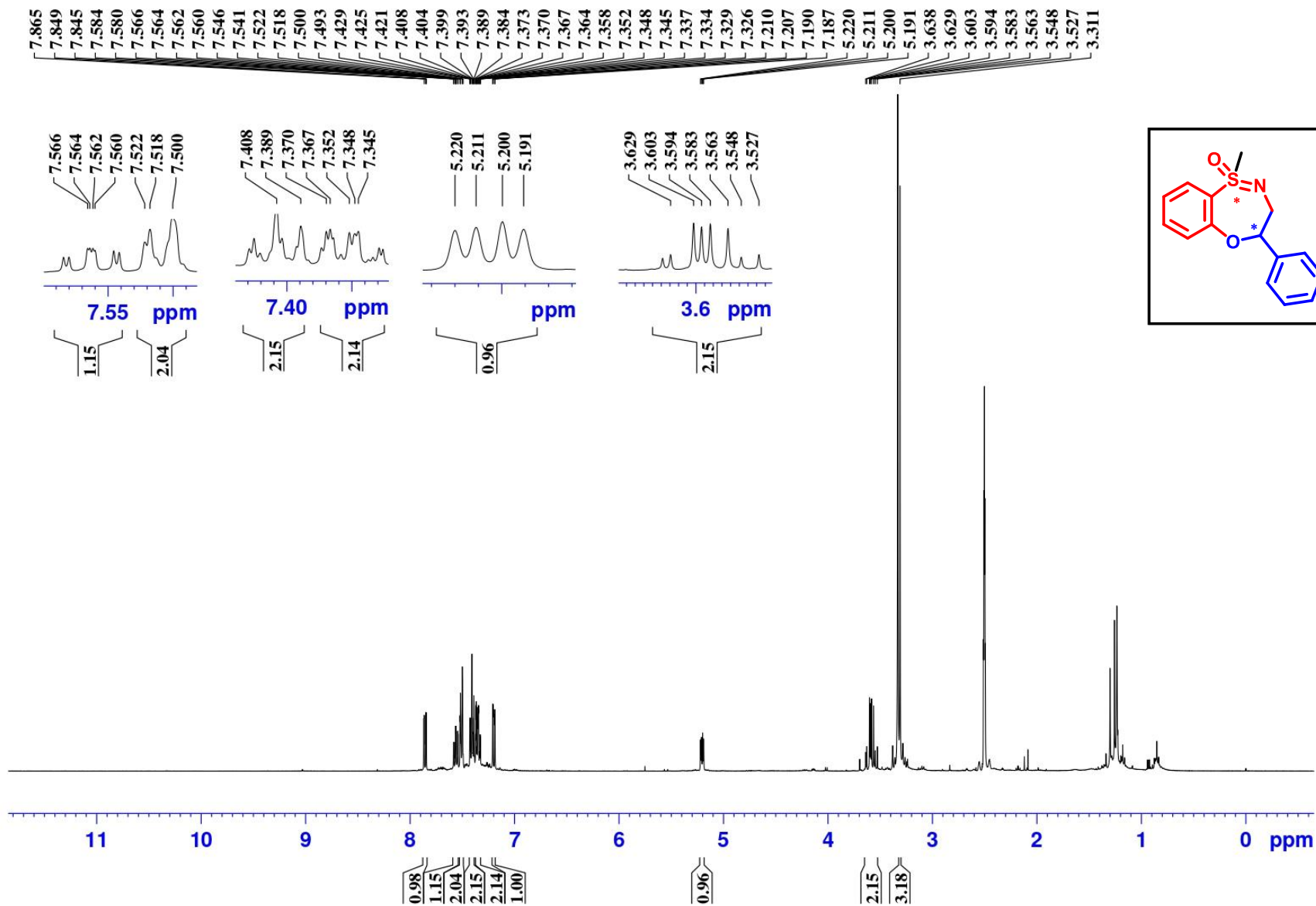


Fig S-25: ^1H NMR Spectra of Compound 4a (400 MHz, DMSO-d_6)

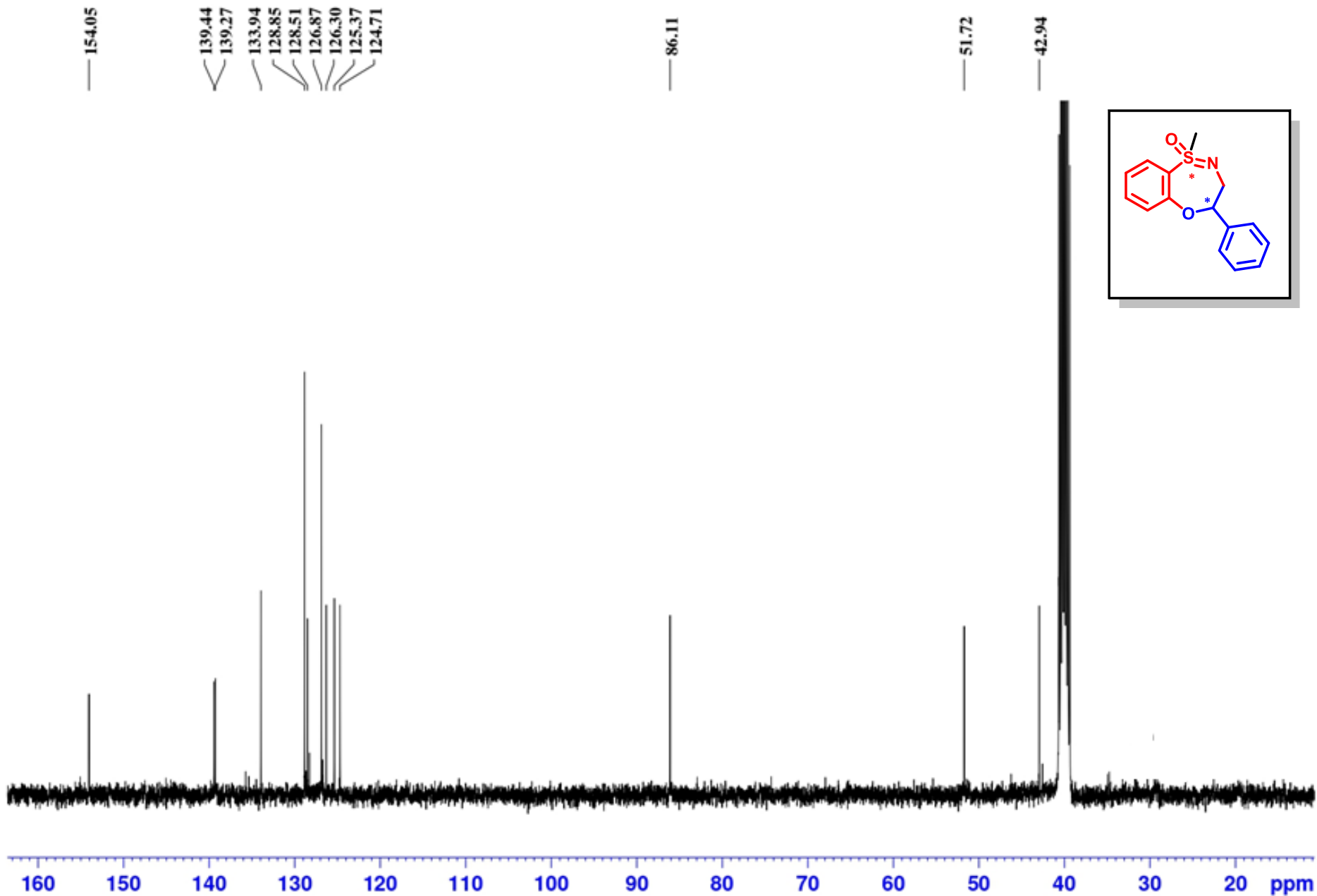


Fig S-26: ^{13}C NMR Spectra of Compound **4a** (100 MHz, DMSO-d_6)

SAIF [HRMS Report]

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Sample ID:	AB-134E	Sample Name:	
Acquisition Date:	02/11/21 11:03:53 AM	Run Time(min):	0.00
Vial:	CStk1-01:4	Injection Volume(ul):	1.00

HRMS21111FEB04 #33-65 RT: 0.25-0.50 AV: 33 SB: 1 0.01 NL: 6.56E6
T: FTMS + c ESI Full ms [100.00-750.00]

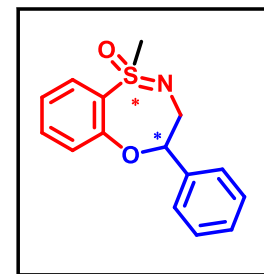
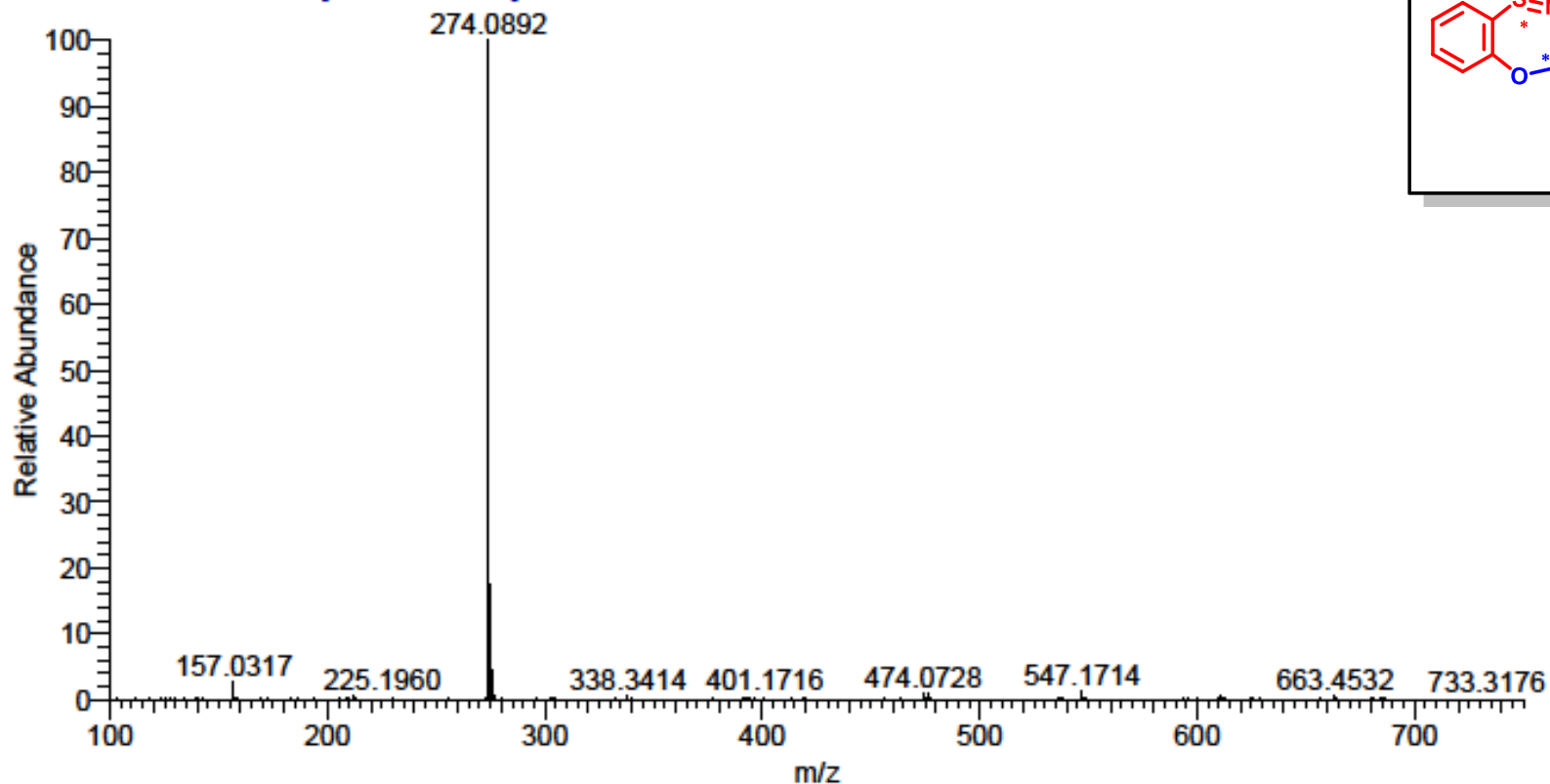


Fig S-27: HRMS report of Compound 4a

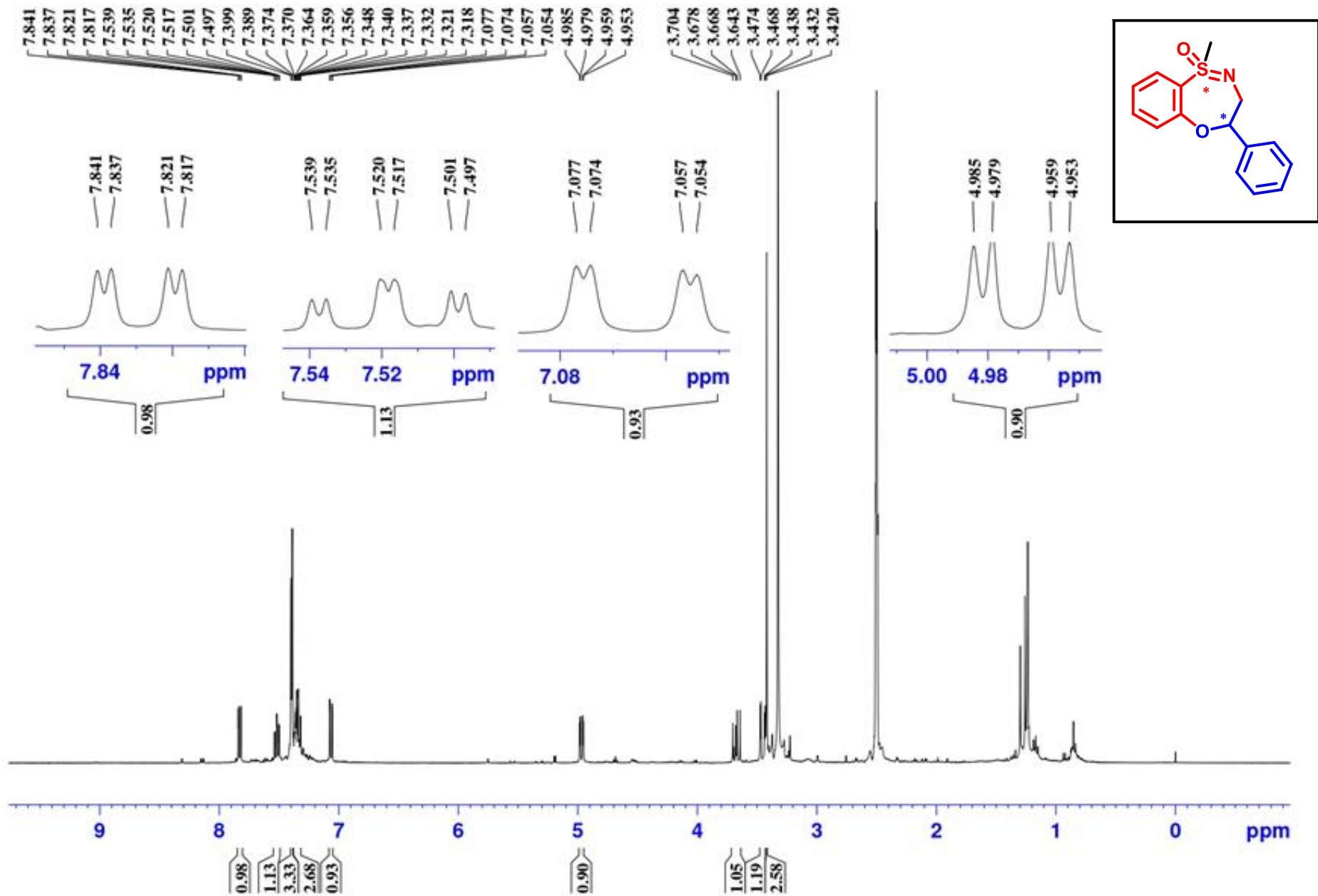


Fig S-28: ^1H NMR Spectra of Compound 4a' (400 MHz, DMSO-d_6)

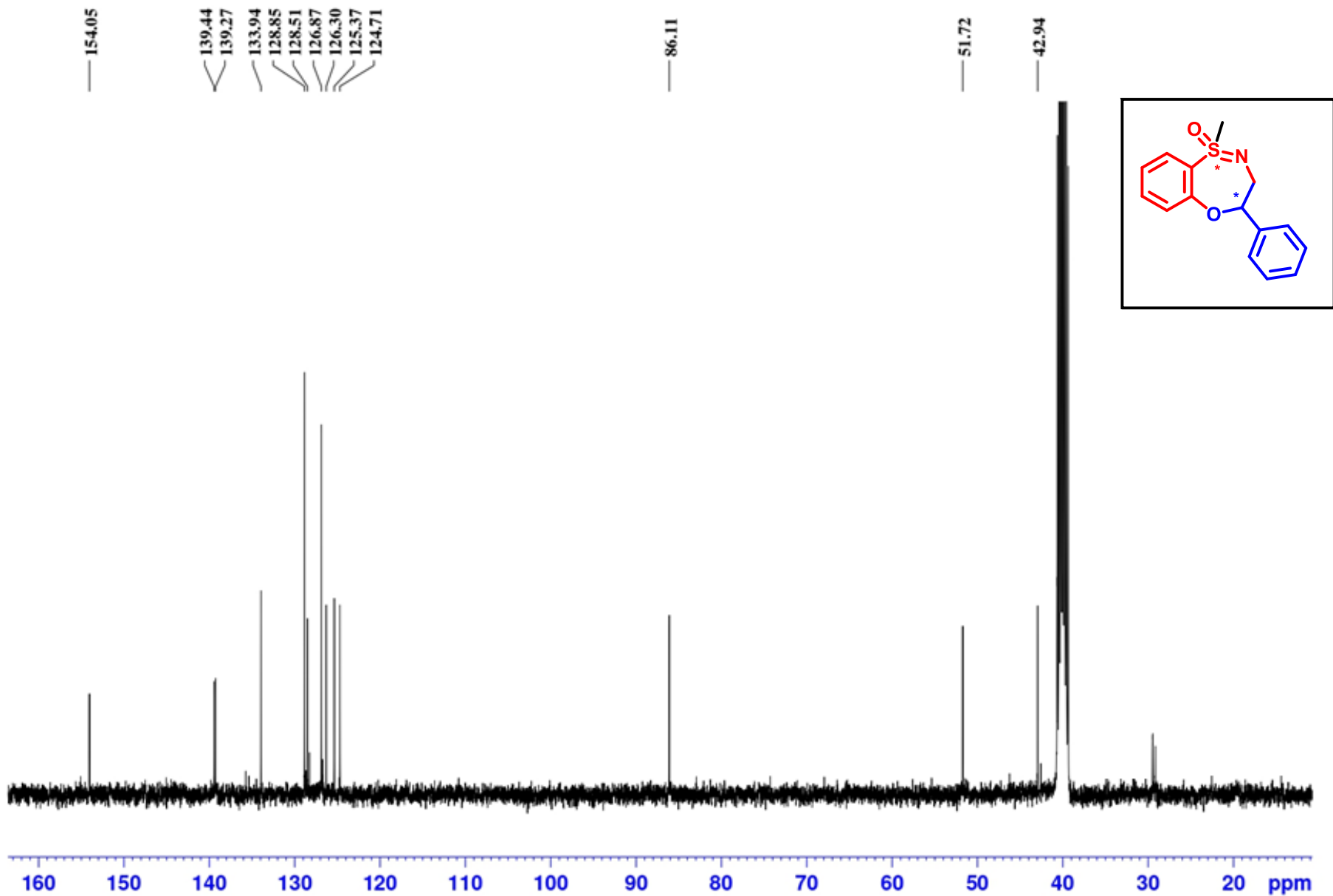


Fig S-29: ^{13}C NMR Spectra of Compound **4a'** (100 MHz, DMSO-d_6)

SAIF [HRMS Report]

Data File:	HRMS21111FEB05	Original Data Path:	D:\INTERNAL NEW\2021\Feb 2021
Sample ID:	AB-134F	Sample Name:	
Acquisition Date:	02/11/21 11:05:54 AM	Run Time(min):	0.00
Vial:	CStk1-01:5	Injection Volume(μl):	1.00

HRMS21111FEB05 #32-64 RT: 0.25-0.50 AV: 33 SB: 1 0.01 NL: 4.07E6
T: FTMS + c ESI Full ms [100.00-750.00]

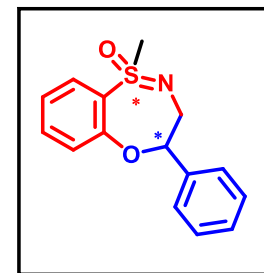
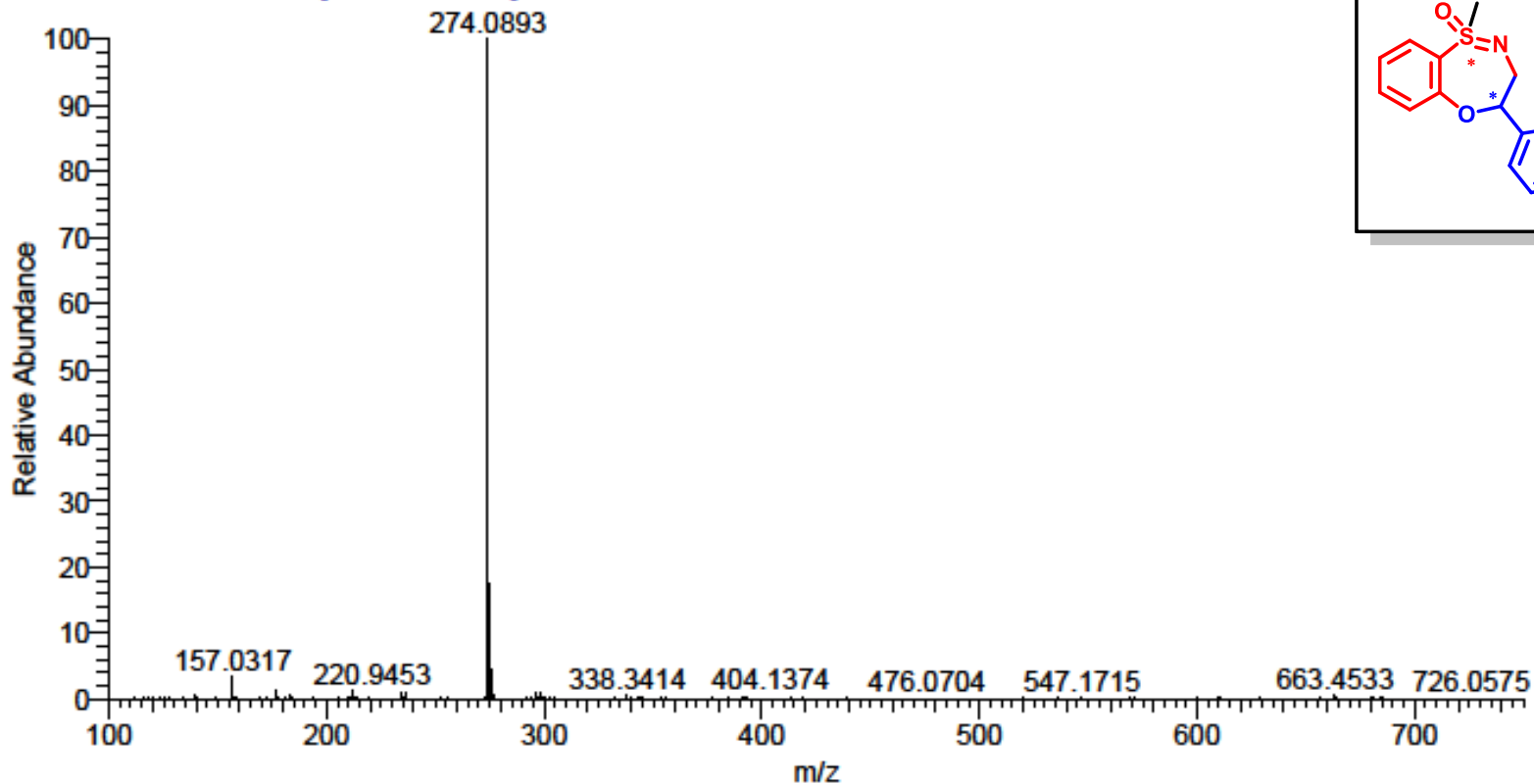


Fig S-30: HRMS report of Compound 4a'

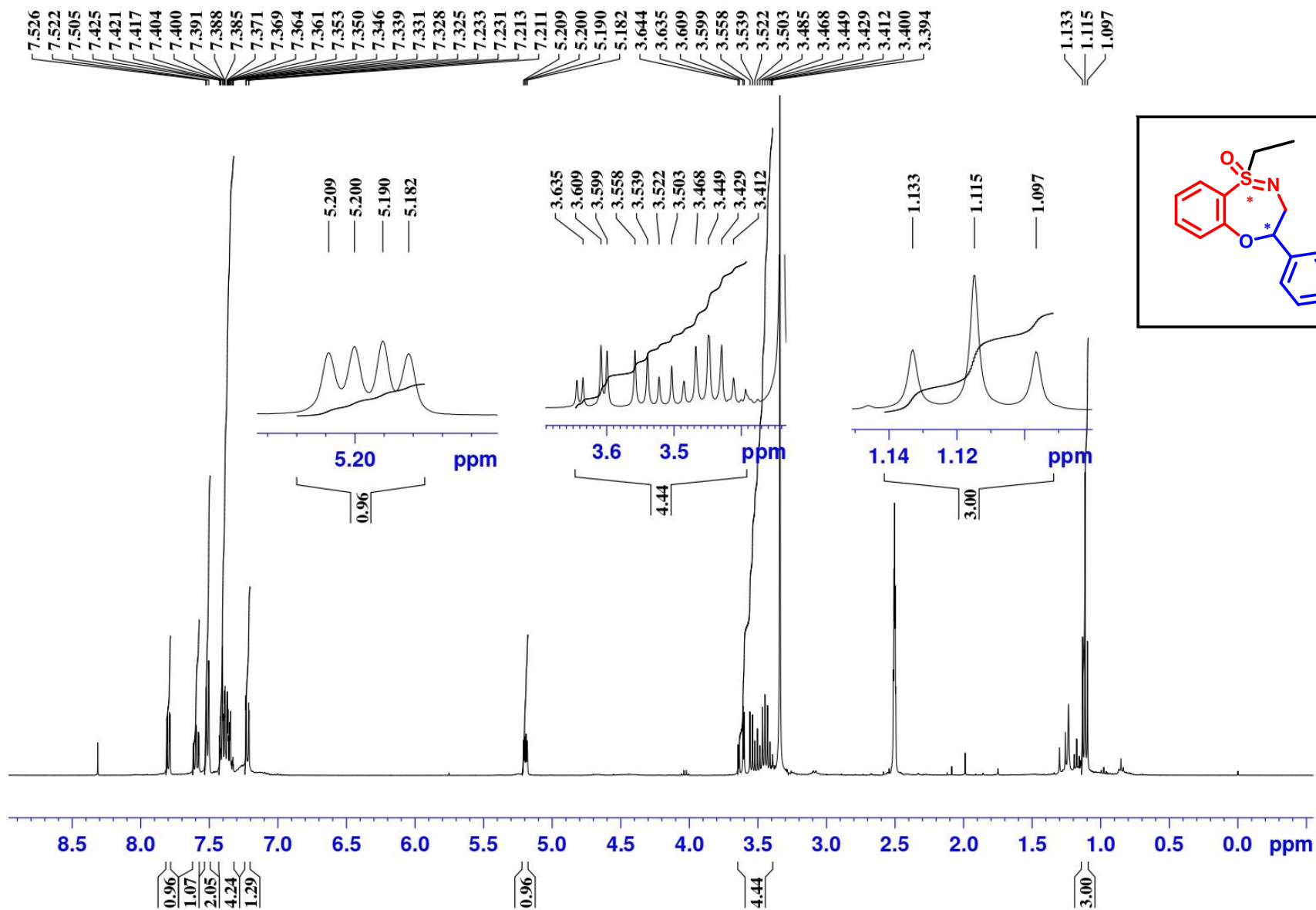


Fig S-31: ¹H NMR Spectra of Compound 4b' (400 MHz, DMSO-d₆)

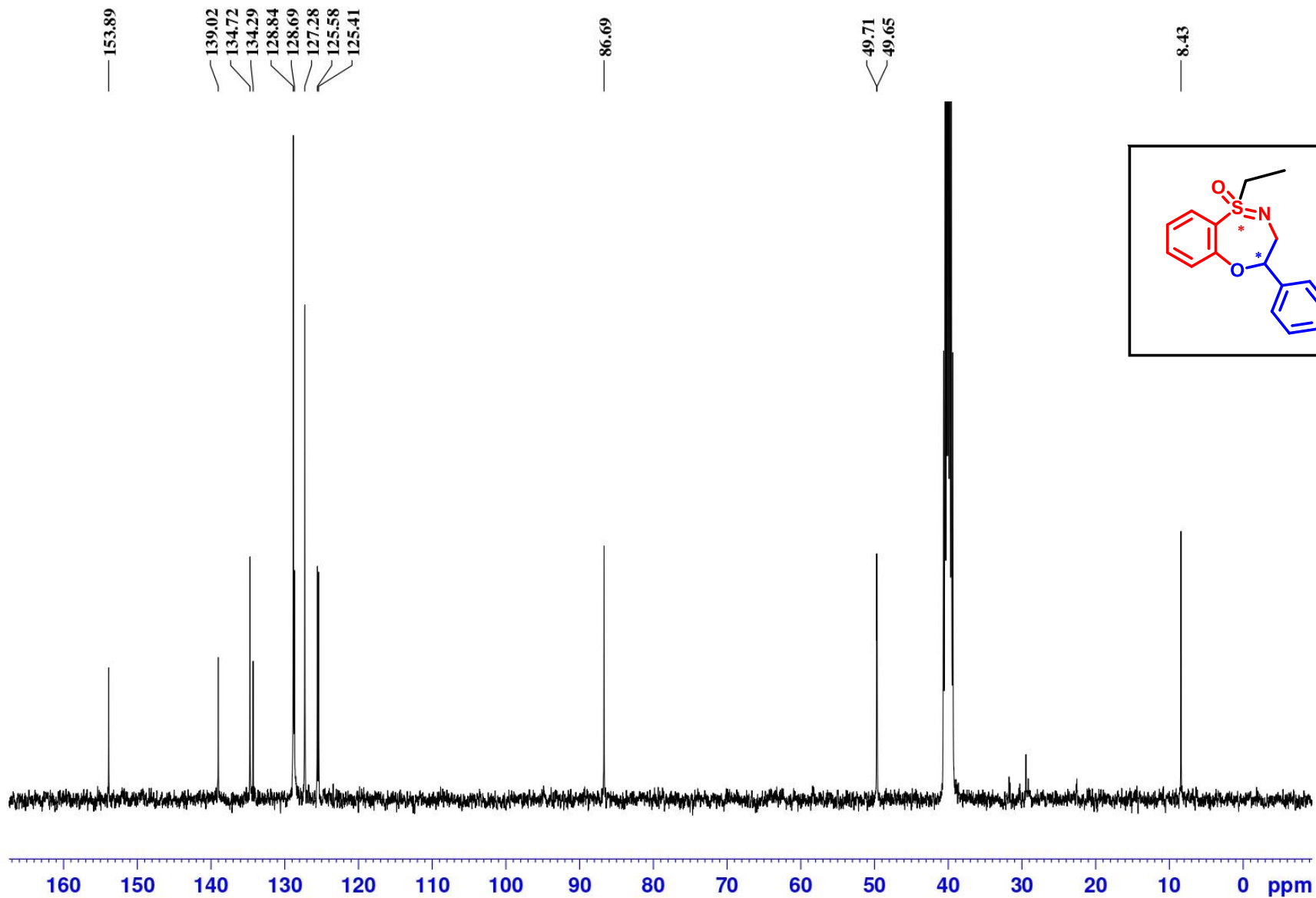


Fig S-32: ^{13}C NMR Spectra of Compound **4b**(100 MHz, DMSO-d_6)

SAIF [HRMS Report]

Data File:	HRMS21I15FEB09	Original Data Path:	D:\INTERNAL NEW\2021\Feb
Sample ID:	AB-138A	Sample Name:	2021
Acquisition Date:	02/15/21 12:06:28 PM	Run Time(min):	0.00
Vial:	CStk1-01:9	Injection Volume(ul):	1.00

HRMS21I15FEB09 #30-64 RT: 0.25-0.50 AV: 35 SB: 1 0.01 NL: 1.65E7
T: FTMS + c ESI Full ms [100.00-750.00]

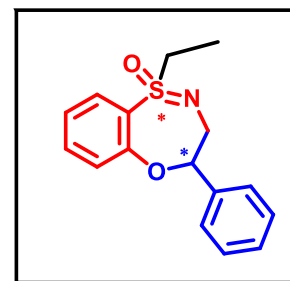
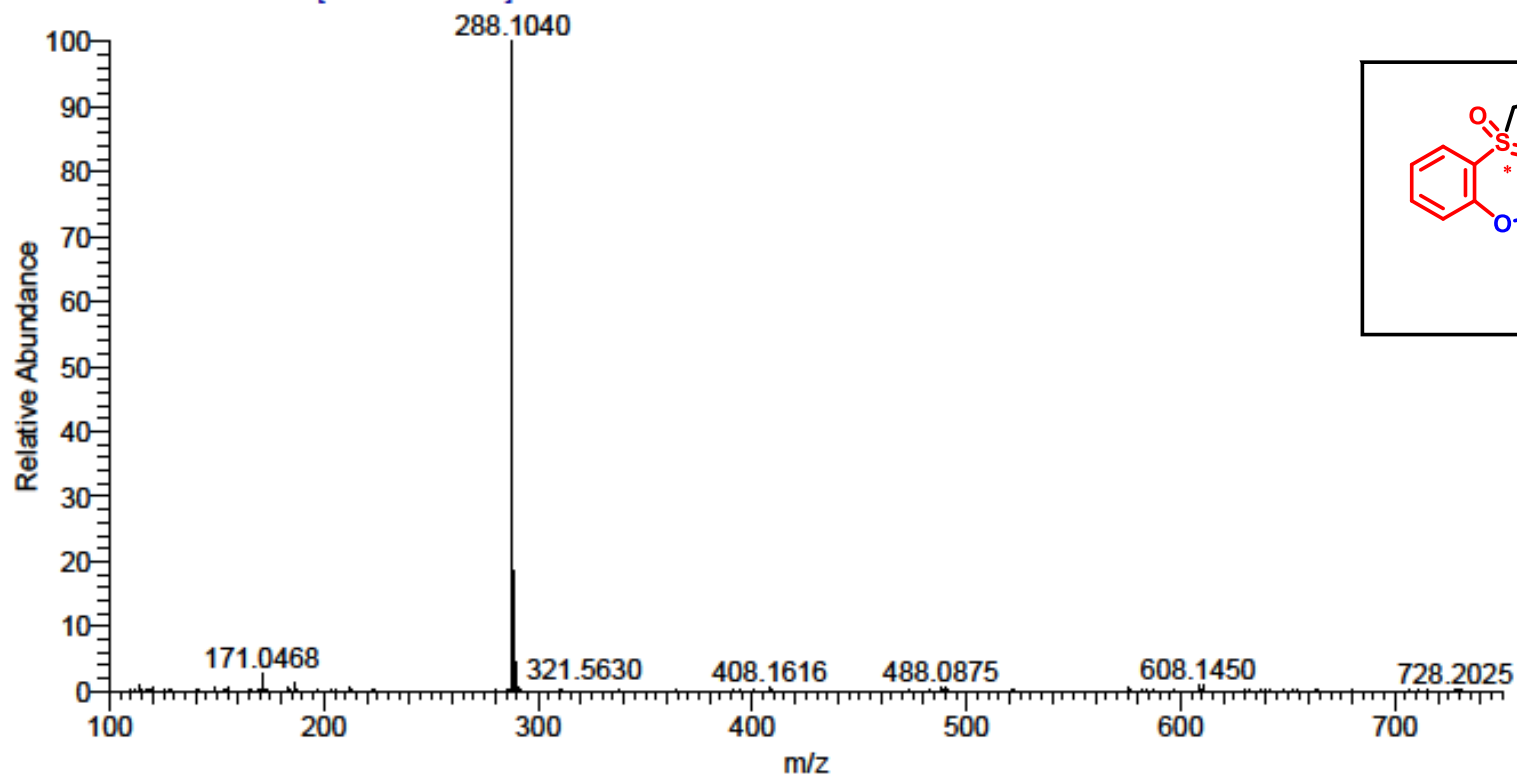


Fig S-33: HRMS report of Compound 4b.

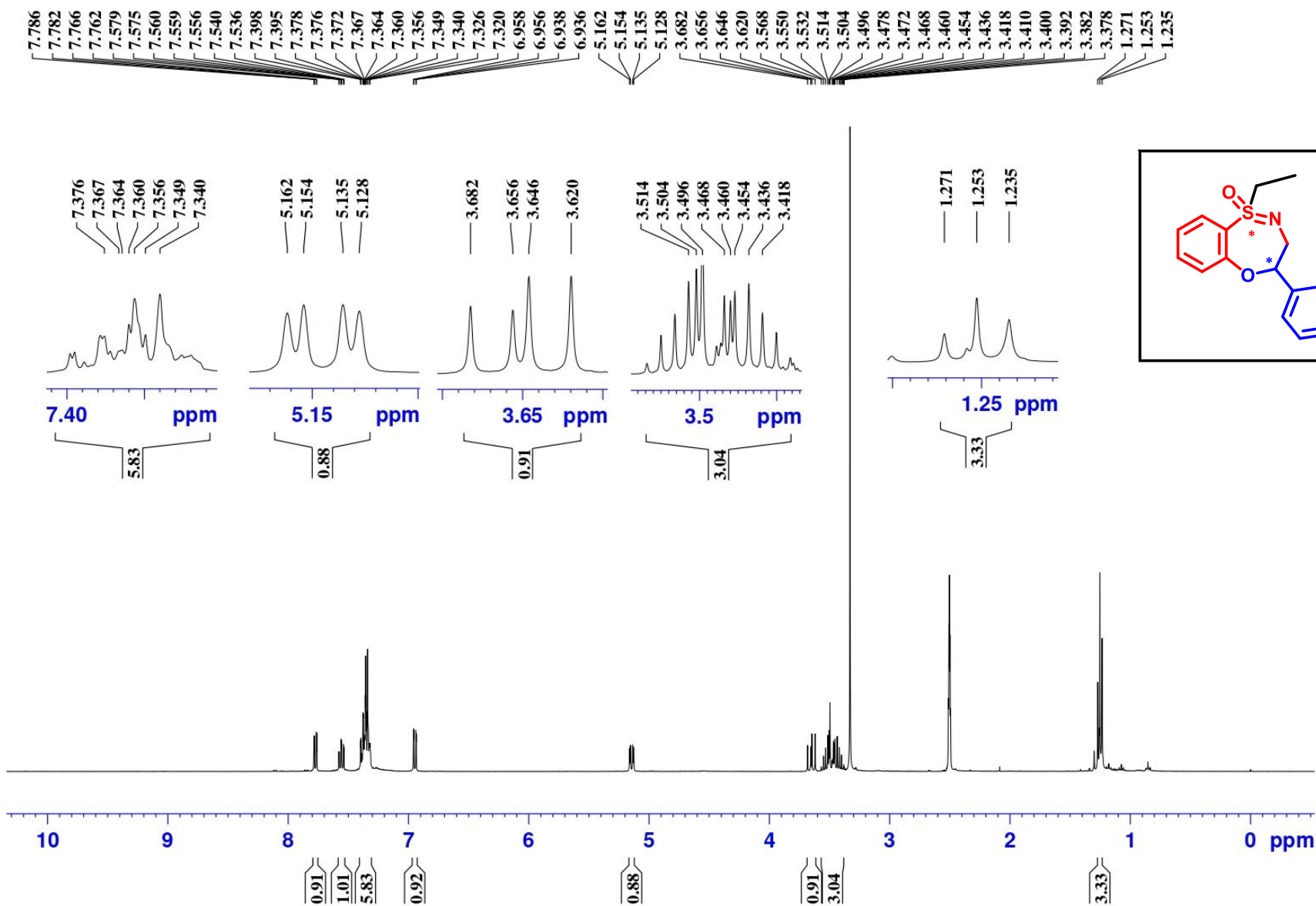


Fig S-34: ^1H NMR Spectra of Compound **4b'** (400 MHz, DMSO-d_6)

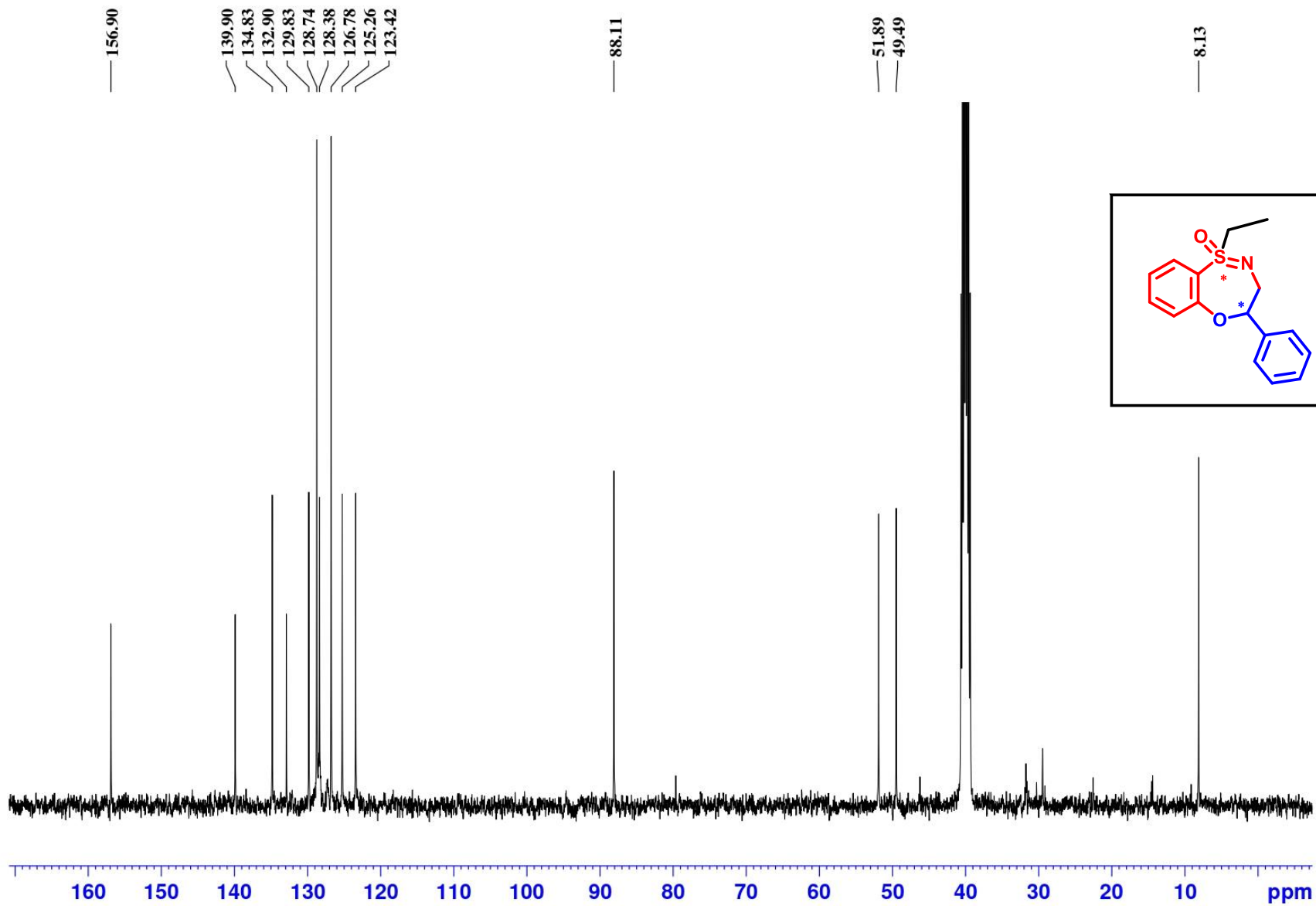


Fig S-35: ¹³C NMR Spectra of Compound **4b'** (100 MHz, DMSO-d₆)

SAIF [HRMS Report]

Data File:	HRMS21I15FEB10	Original Data Path:	D:\INTERNAL NEW\2021\Feb 2021
Sample ID:	AB-138B	Sample Name:	
Acquisition Date:	02/15/21 12:08:27 PM	Run Time(min):	0.00
Vial:	CS&k1-01:10	Injection Volume(μl):	1.00

HRMS21I15FEB10 #30-63 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 6.41E6
T: FTMS + c ESI Full ms [100.00-750.00]

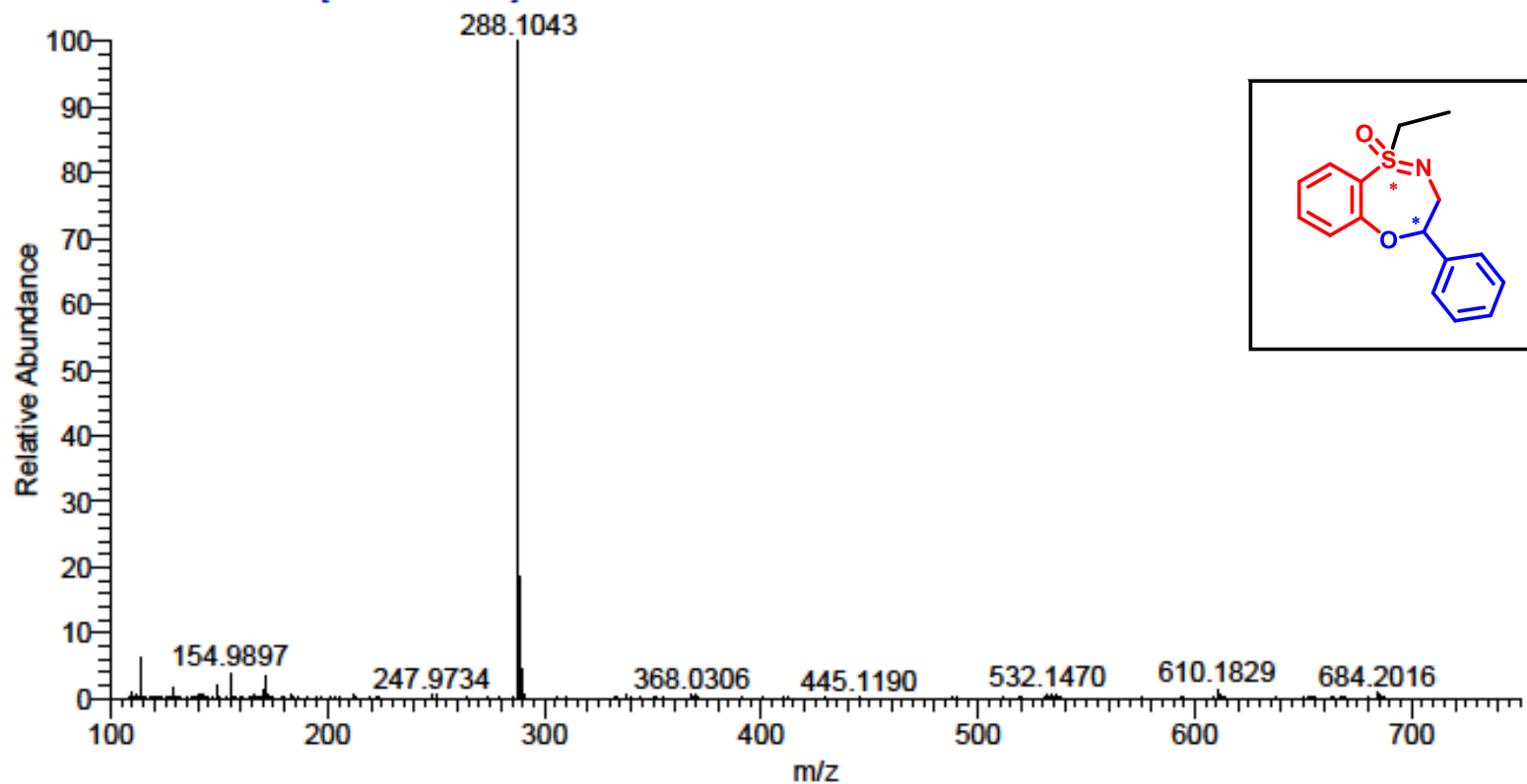


Fig S-36: HRMS report of Compound 4b'

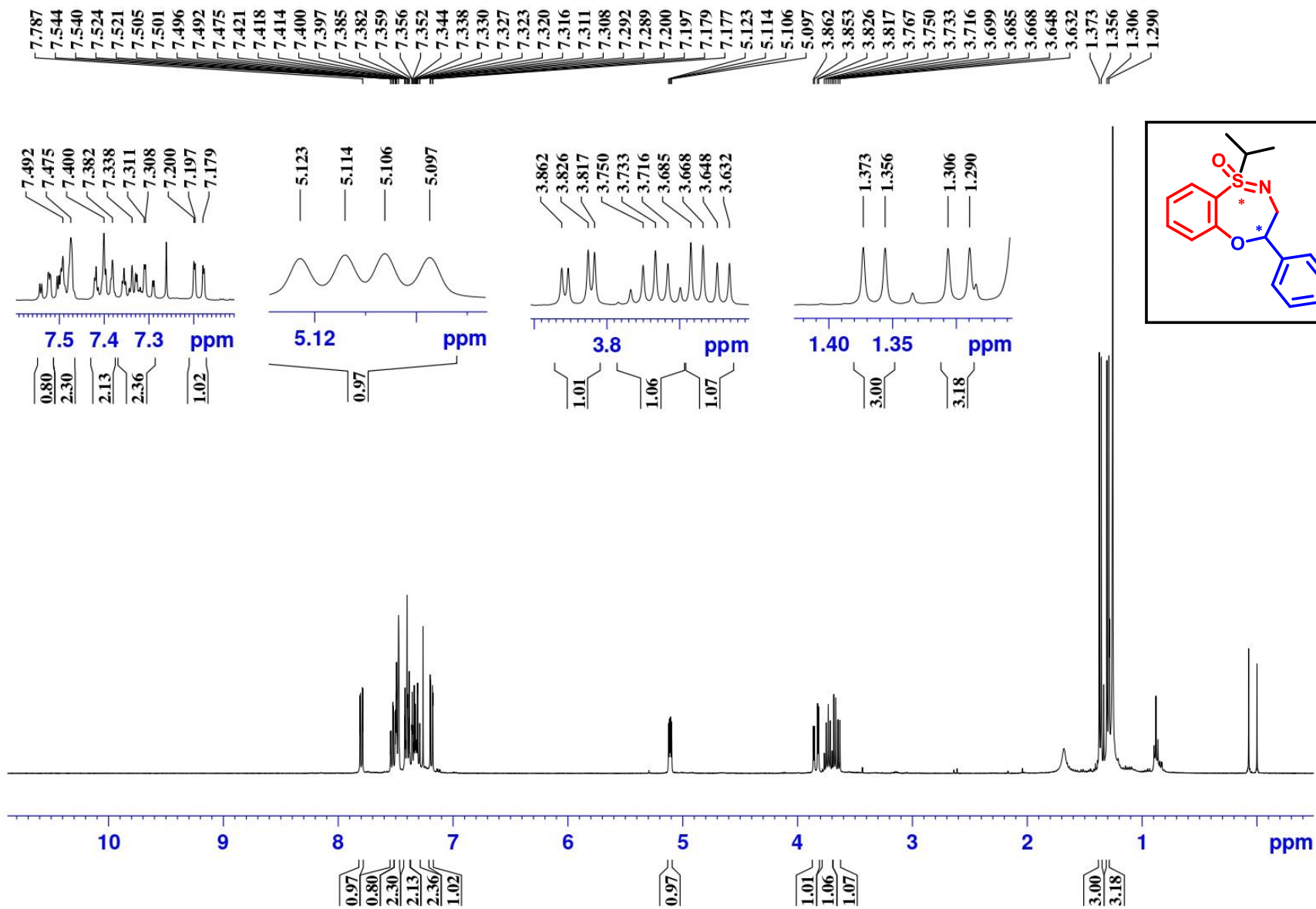


Fig S-37: ^1H NMR Spectra of Compound **4c** (400 MHz, CDCl_3)

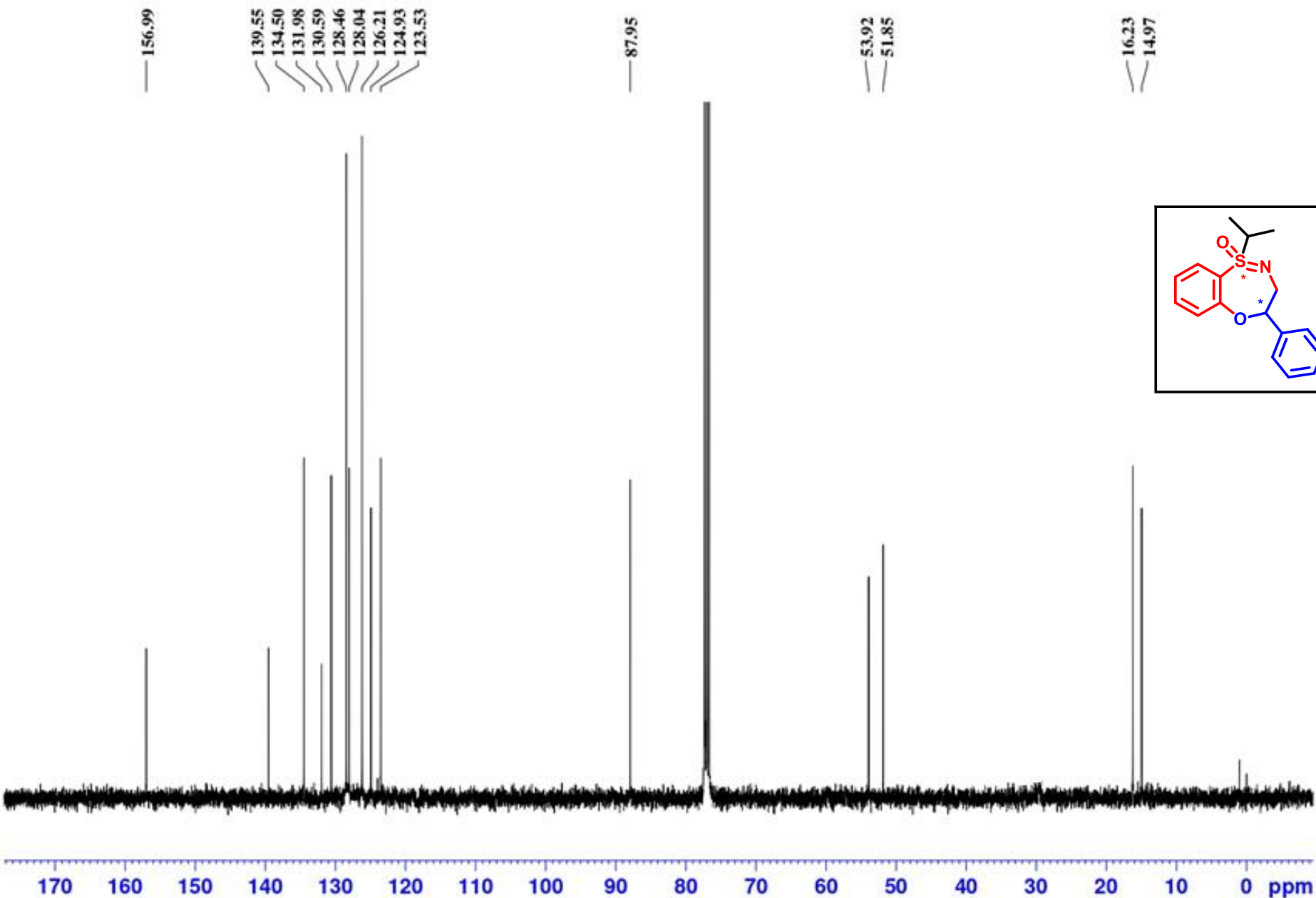


Fig S-38: ^{13}C NMR Spectra of Compound **4c** (100 MHz, DMSO-d_6)

SAIF [HRMS Report]

Data File:	HRMS21111FEB02	Original Data Path:	D:\INTERNAL NEW\2021\Feb 2021
Sample ID:	AB-149A	Sample Name:	
Acquisition Date:	02/11/21 10:59:55 AM	Run Time(min):	0.00
Vial:	CStk1-01:2	Injection Volume(ul):	1.00

HRMS21111FEB02 #33-66 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 5.55E6
T: FTMS + c ESI Full ms [100.00-750.00]

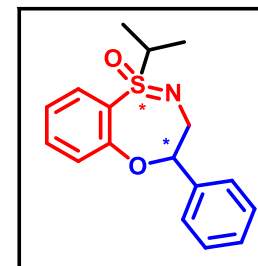
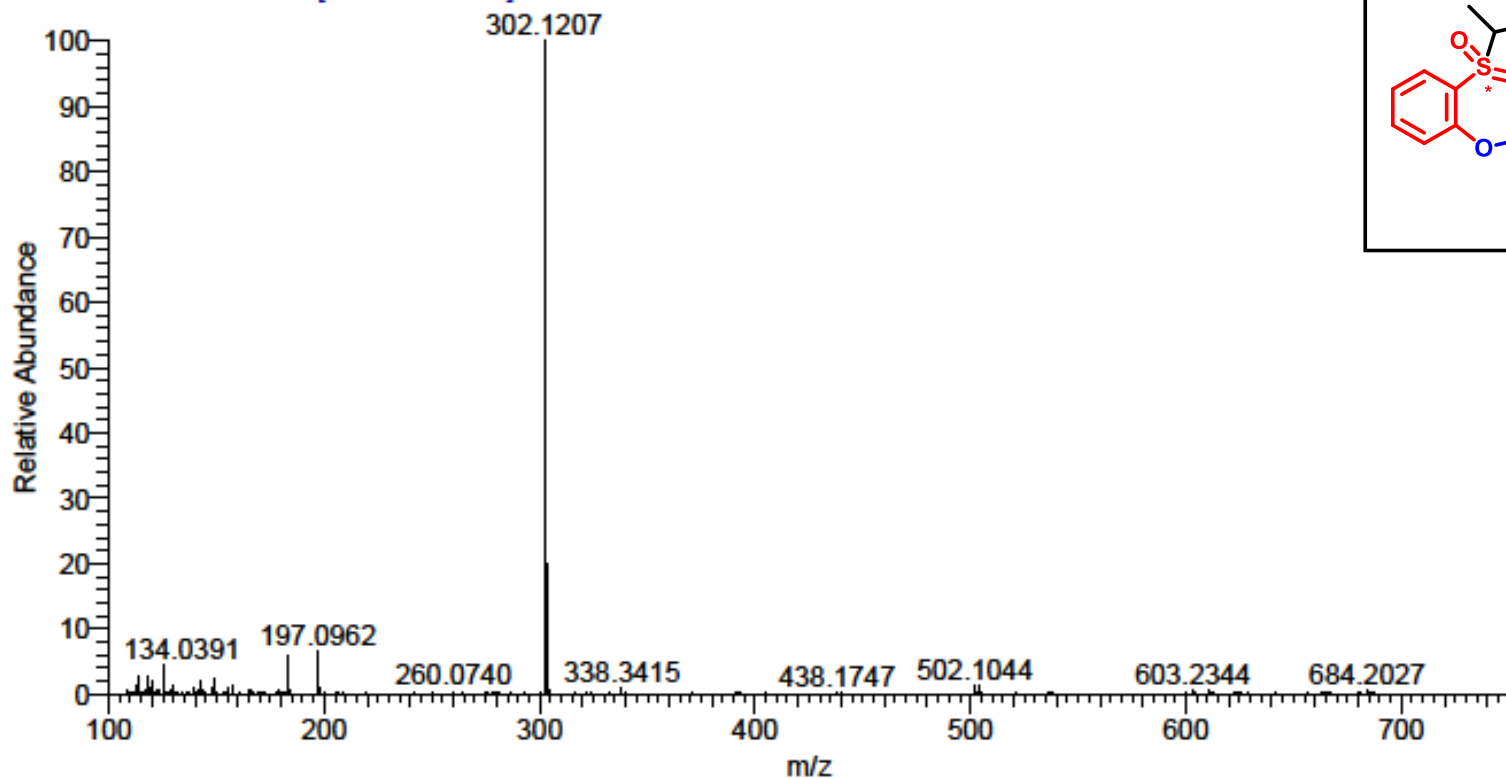


Fig S-39: HRMS report of Compound 4c

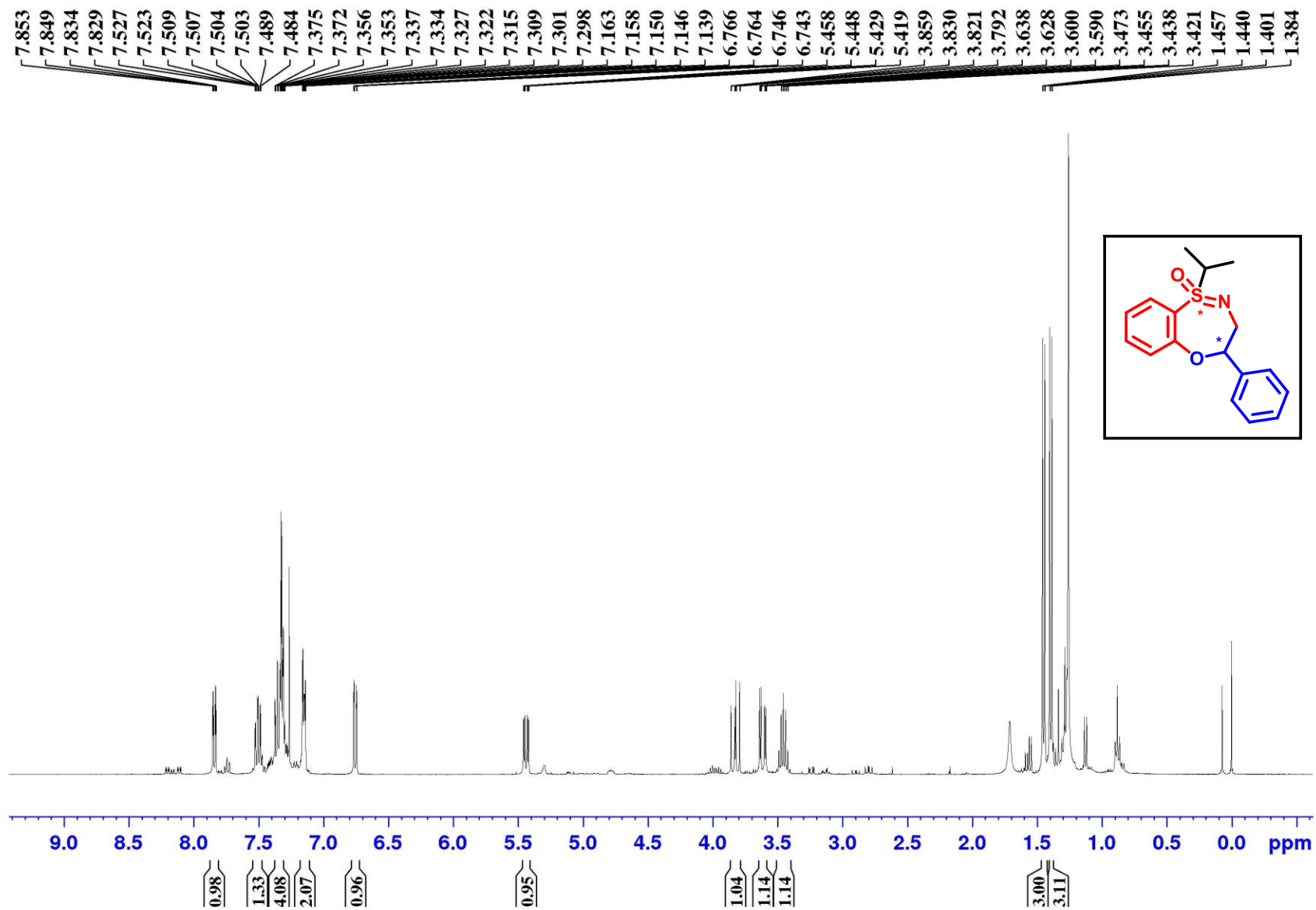


Fig S-40: ^1H NMR Spectra of Compound 4c' (400 MHz, CDCl_3)

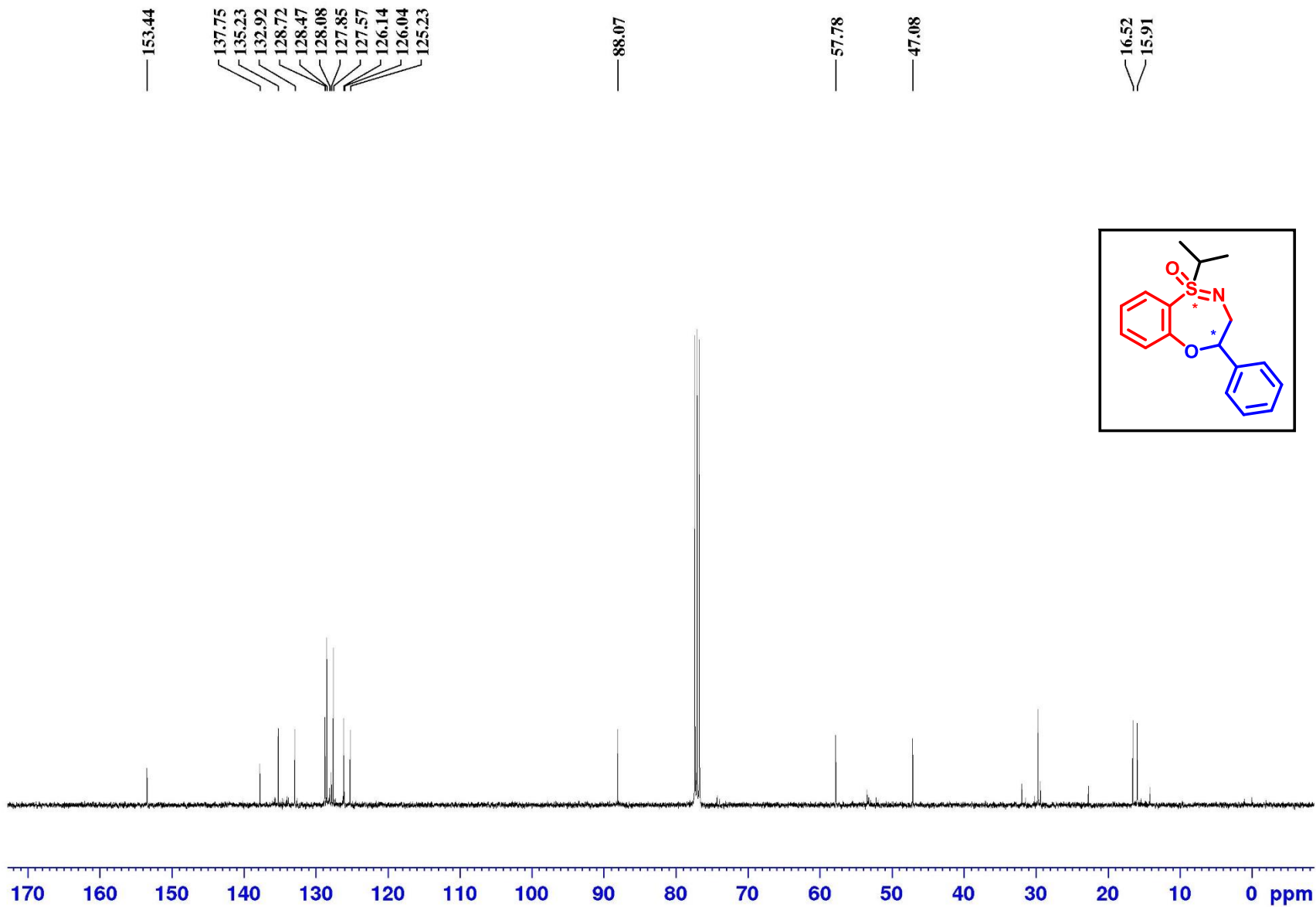


Fig S-41: ^{13}C NMR Spectra of Compound **4c'** (100 MHz, DMSO-d_6)

SAIF [HRMS Report]

Data File:	HRMS21111FEB03	Original Data Path:	D:\INTERNAL NEW\2021\Feb 2021
Sample ID:	AB-149B	Sample Name:	
Acquisition Date:	02/11/21 11:01:54 AM	Run Time(min):	0.00
Vial:	CStk1-01:3	Injection Volume(ul):	1.00

HRMS21111FEB03 #32-66 RT: 0.25-0.50 AV: 35 SB: 1 0.01 NL: 1.75E7
T: FTMS + c ESI Full ms [100.00-750.00]

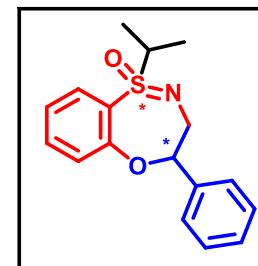
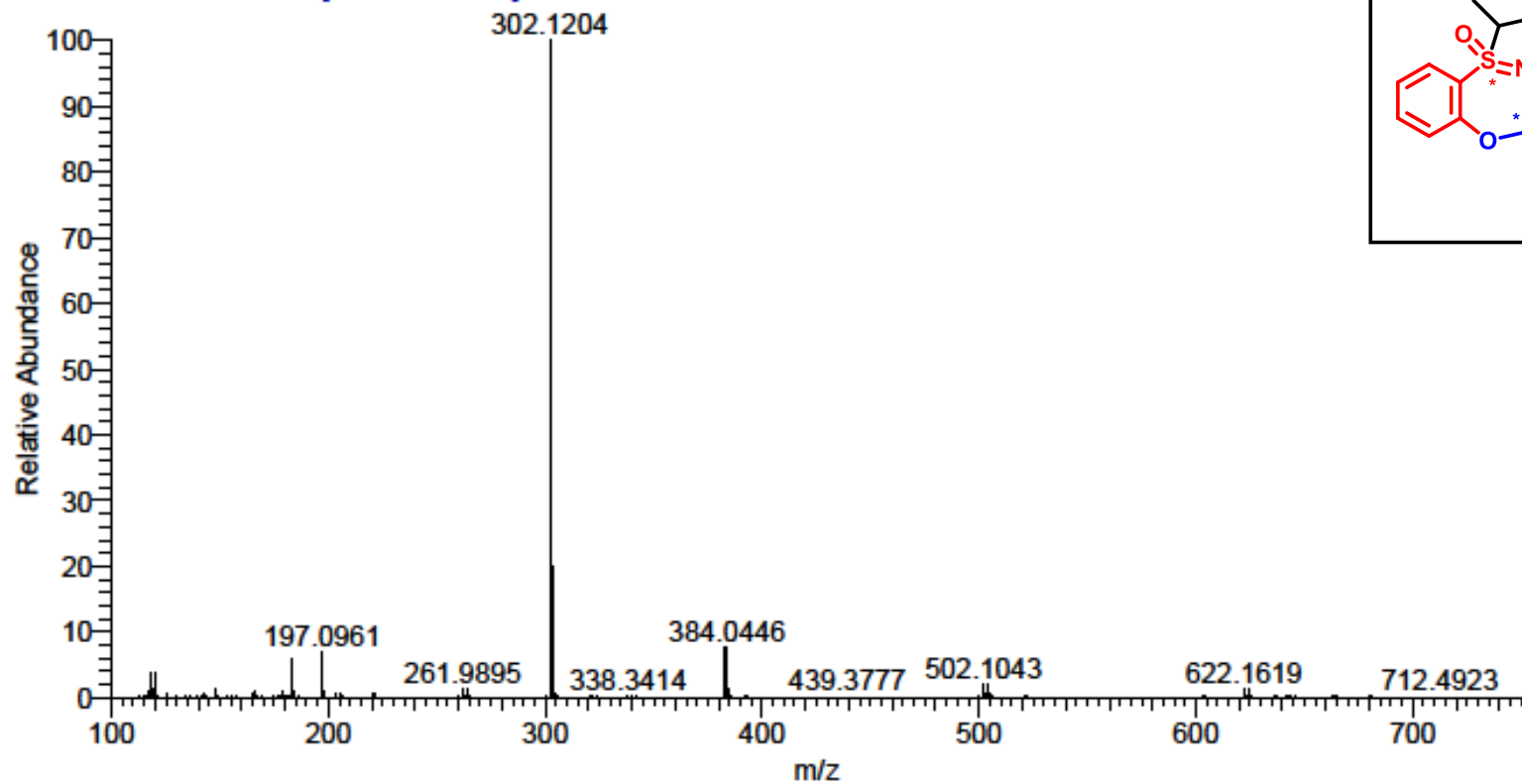


Fig S-42: HRMS report of Compound 4c'

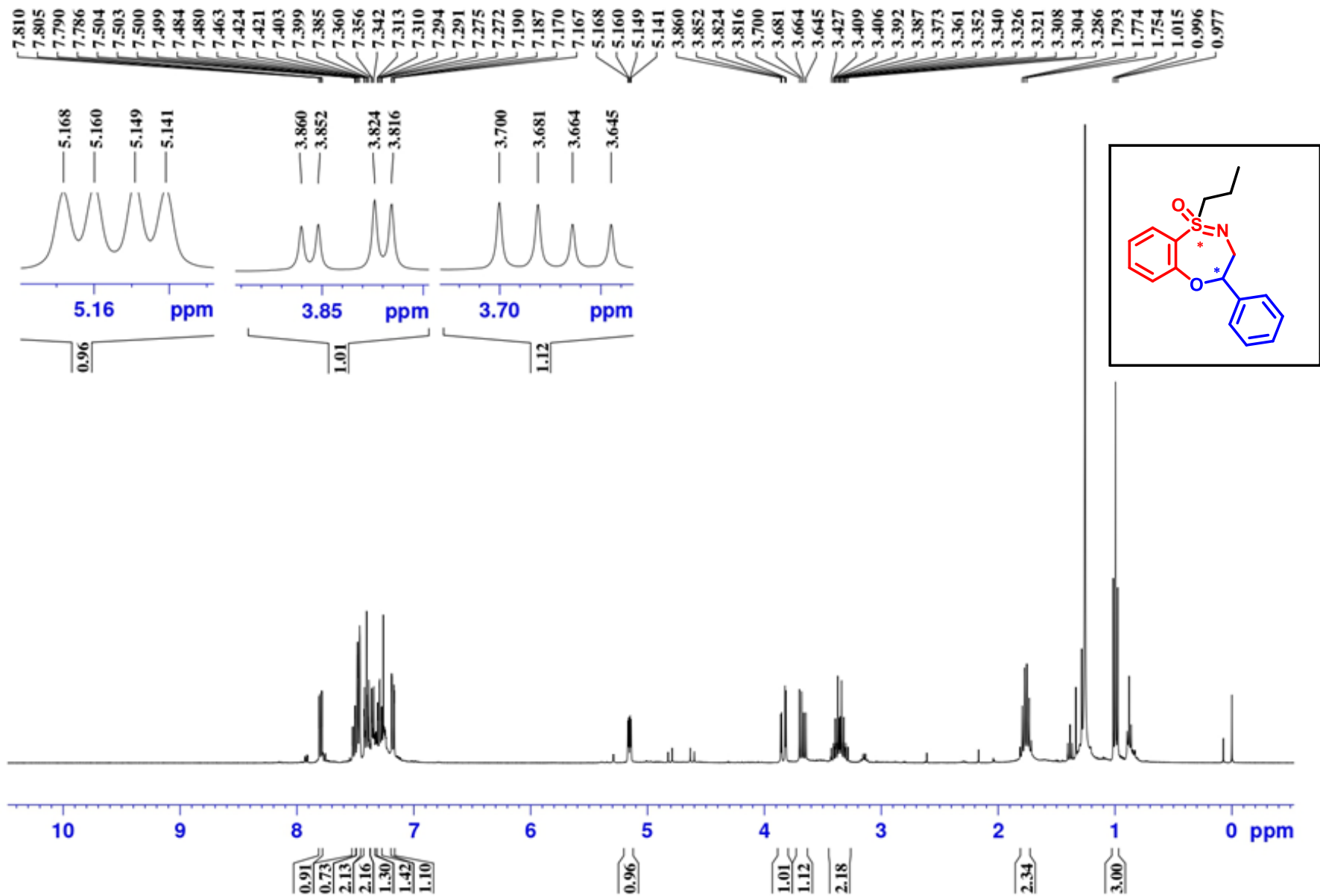


Fig S-43: ^1H NMR Spectra of Compound **4d** (400 MHz, CDCl_3)

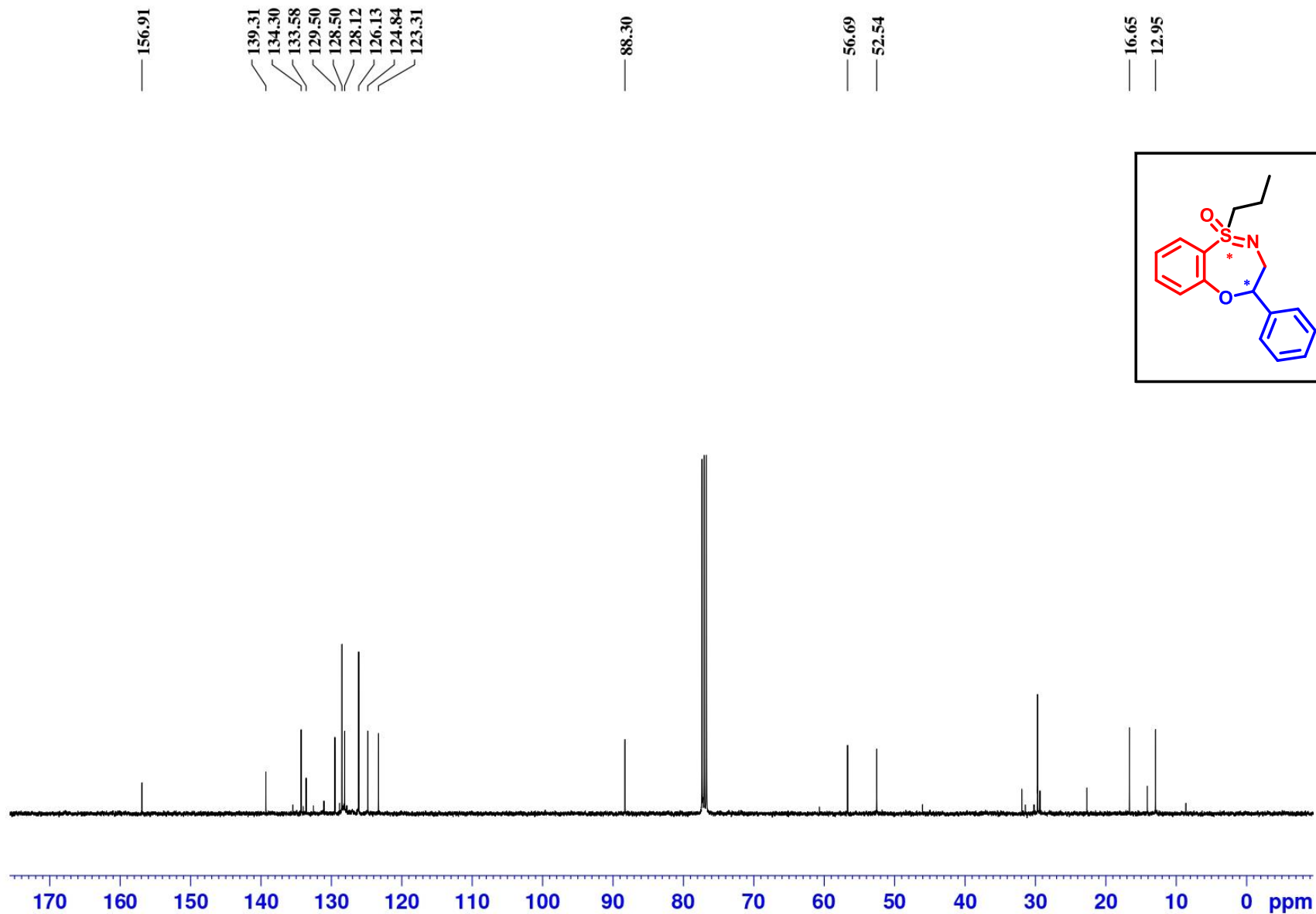


Fig S-44: ^{13}C NMR Spectra of Compound 4d (100 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I10FEB06	Original Data Path:	D:\INTERNAL NEW\2021\Feb 2021
Sample ID:	AB-151A	Sample Name:	
Acquisition Date:	02/10/21 10:58:14 AM	Run Time(min):	0.00
Vial:	CStk1-01:6	Injection Volume(μl):	1.00

HRMS21I10FEB06 #31-65 RT: 0.25-0.50 AV: 35 SB: 1 0.01 NL: 4.66E7
T: FTMS + c ESI Full ms [100.00-750.00]

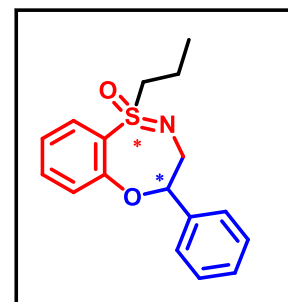
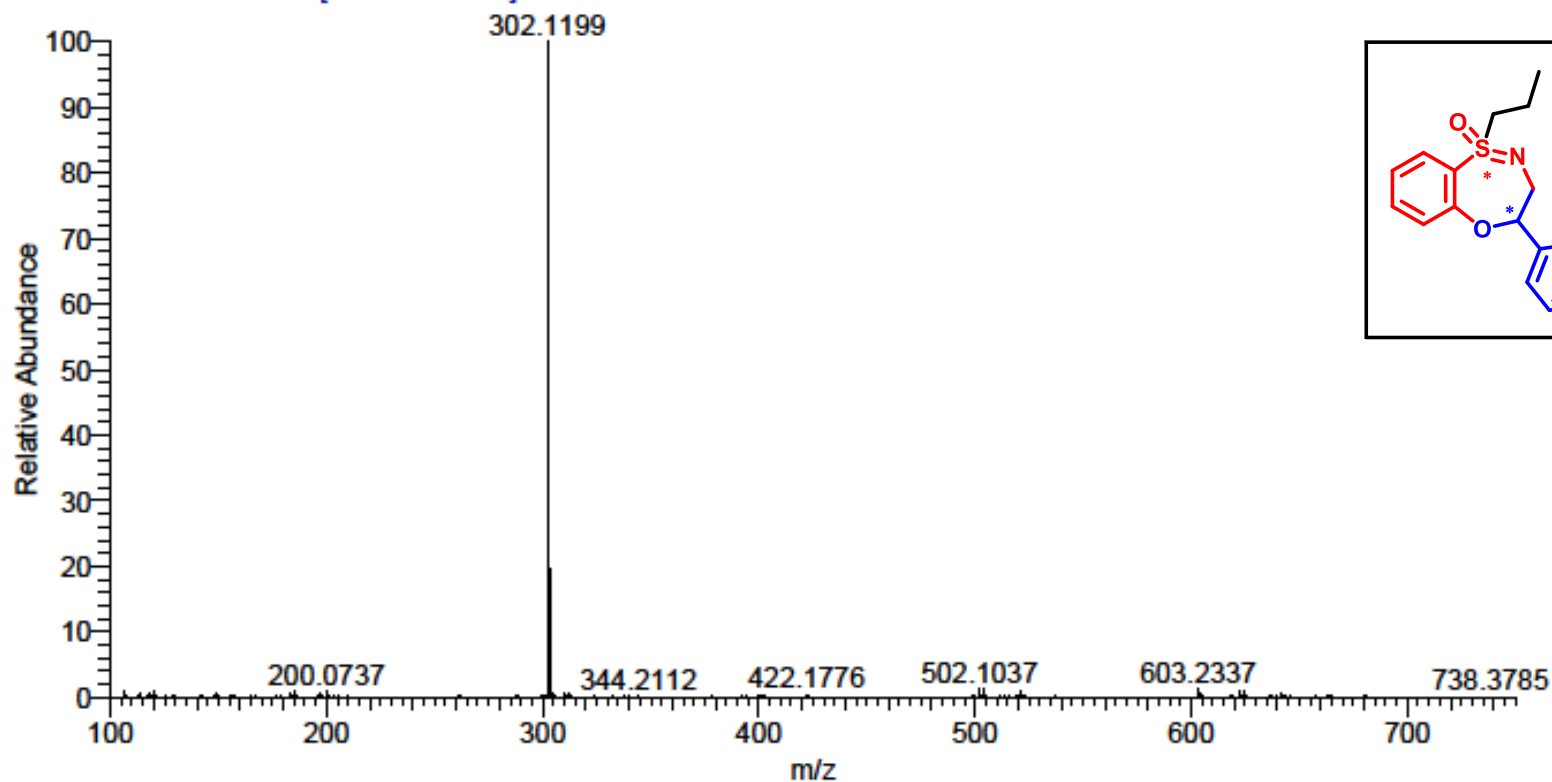


Fig S-45: HRMS report of Compound 4d

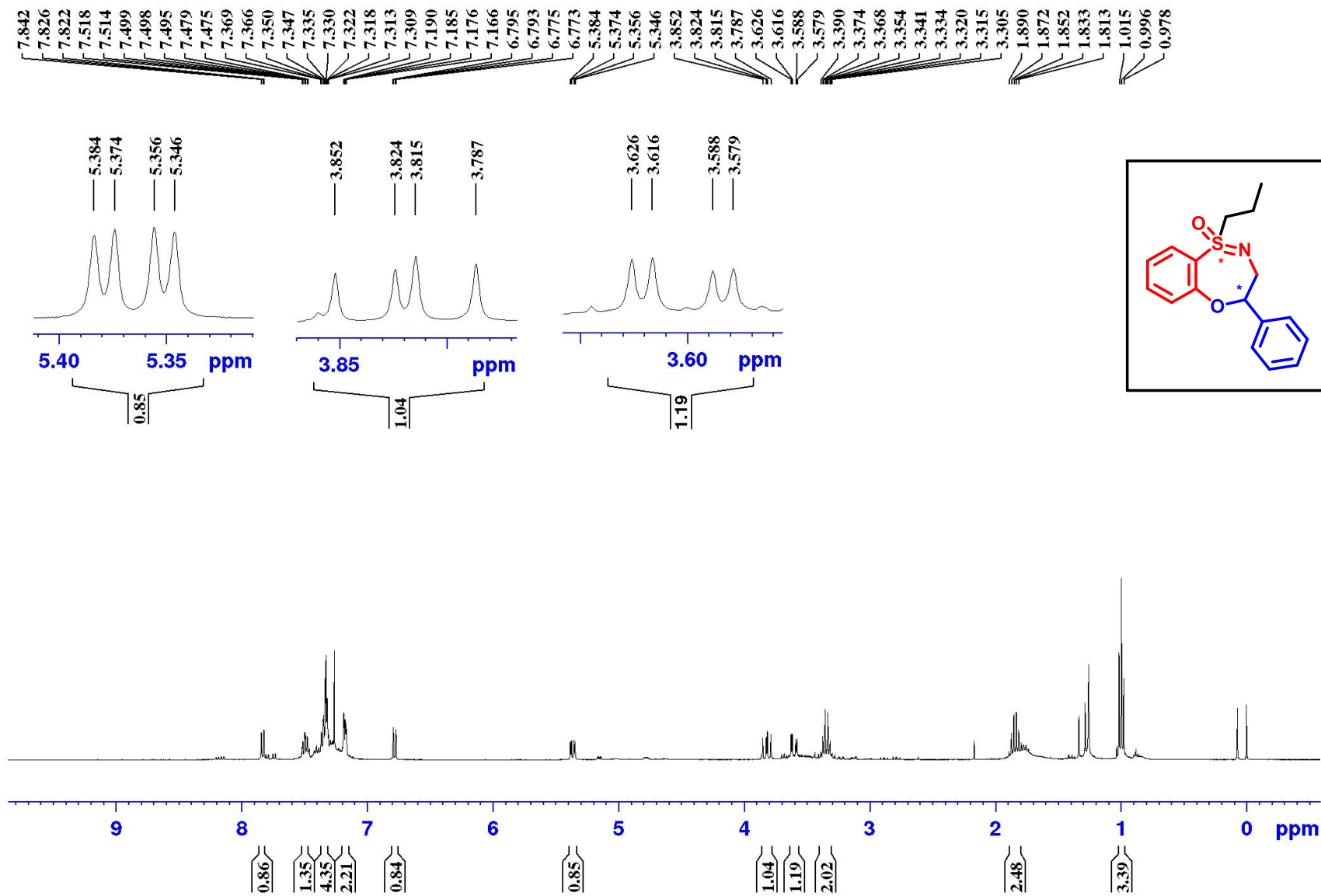


Fig S-46: ¹H NMR Spectra of Compound 4d' (400 MHz, CDCl₃)

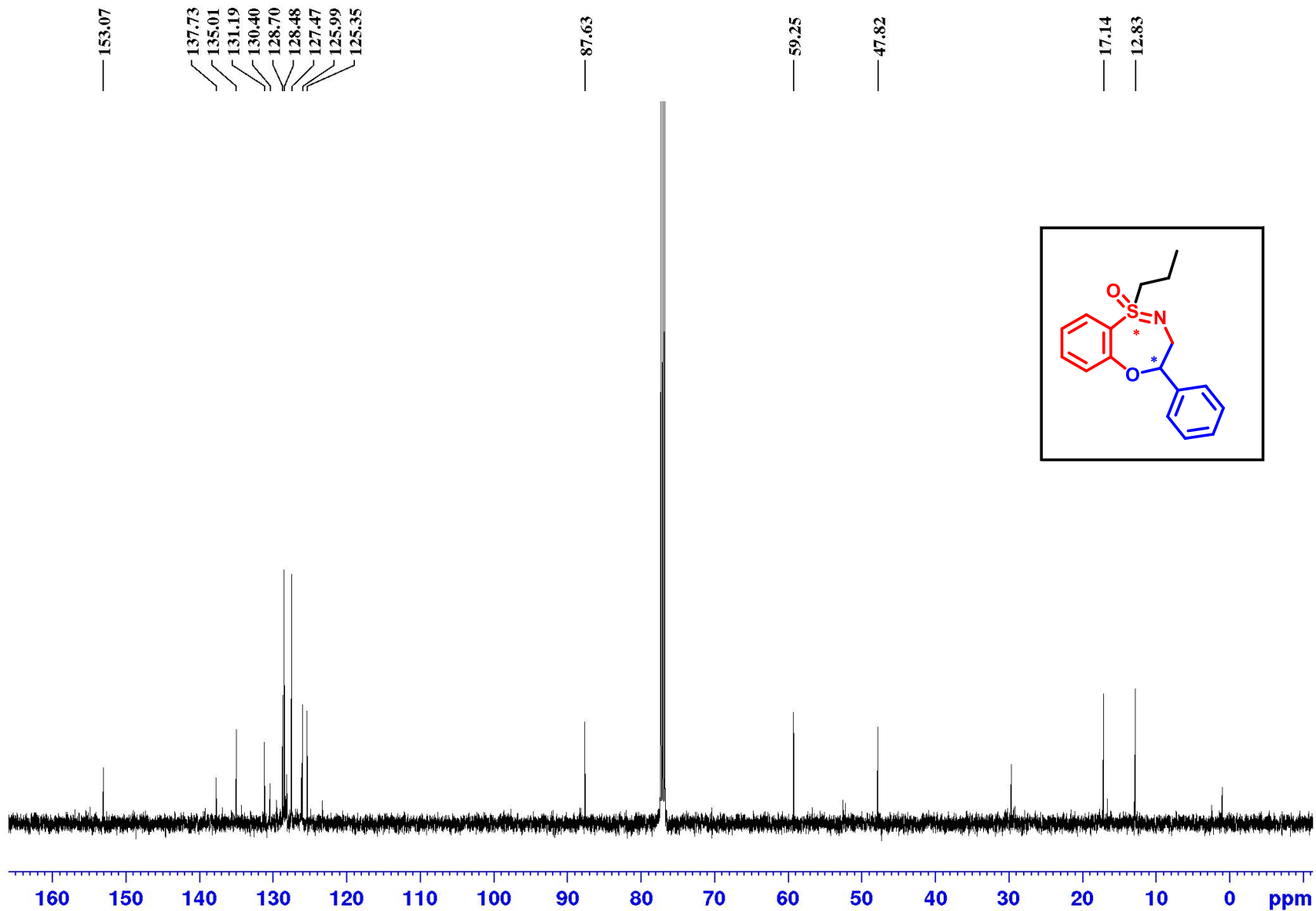


Fig S-47: ^{13}C NMR Spectra of Compound 4d' (100 MHz, CDCl₃).

SAIF [HRMS Report]

Data File:	HRMS21I10FEB07	Original Data Path:	D:\INTERNAL NEW\2021\Feb 2021
Sample ID:	AB-151B	Sample Name:	
Acquisition Date:	02/10/21 11:00:13 AM	Run Time(min):	0.00
Vial:	CStk1-01:7	Injection Volume(μl):	1.00

HRMS21I10FEB07 #31-64 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 2.87E7
T: FTMS + c ESI Full ms [100.00-750.00]

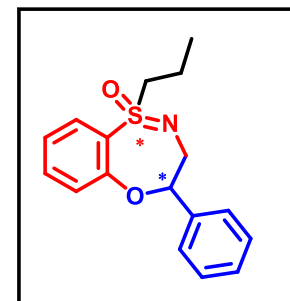
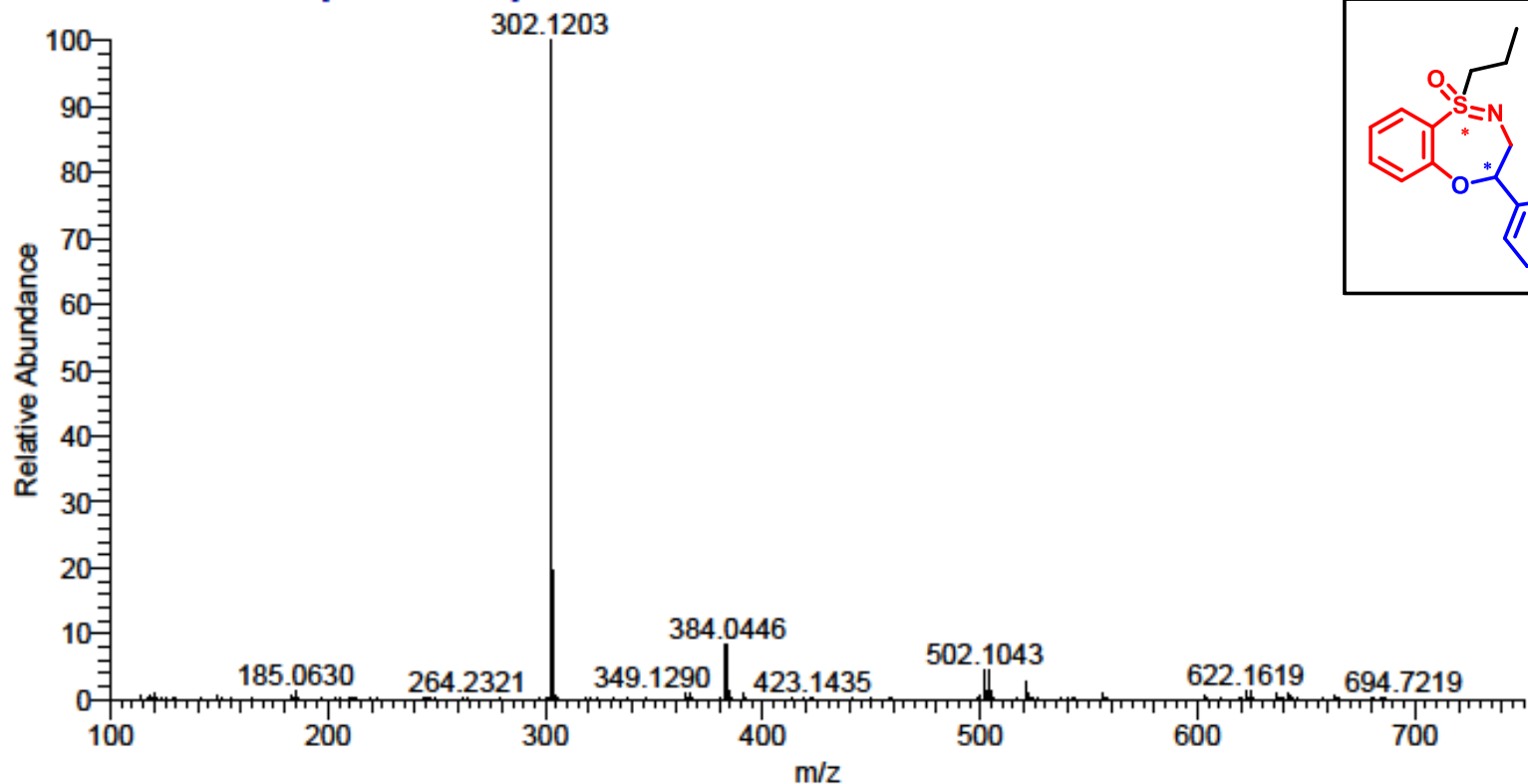


Fig S-48: HRMS report of Compound 4d' (400 MHz, CDCl₃)

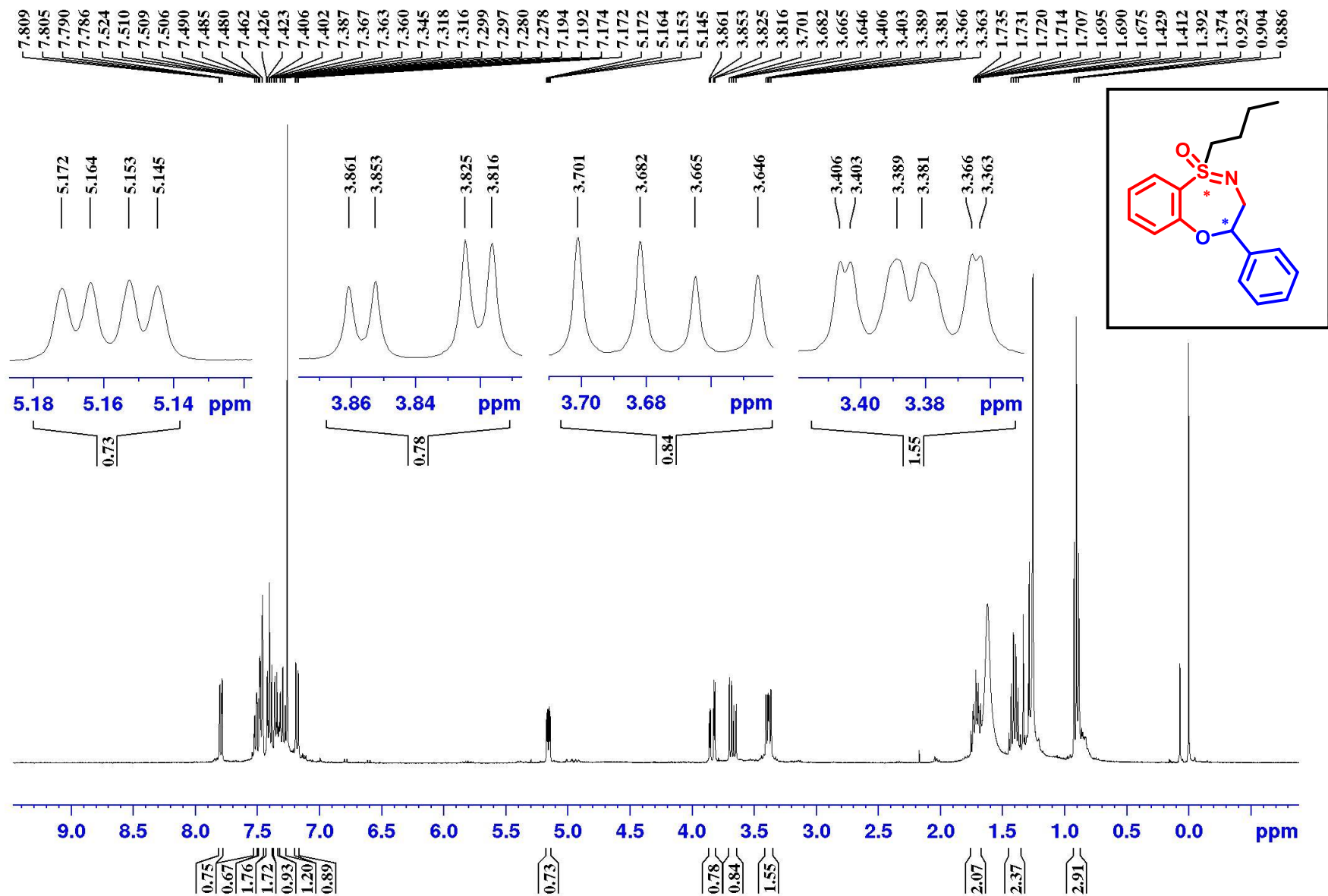


Fig S-49: ^1H NMR Spectra of Compound **4e** (400 MHz, CDCl_3)

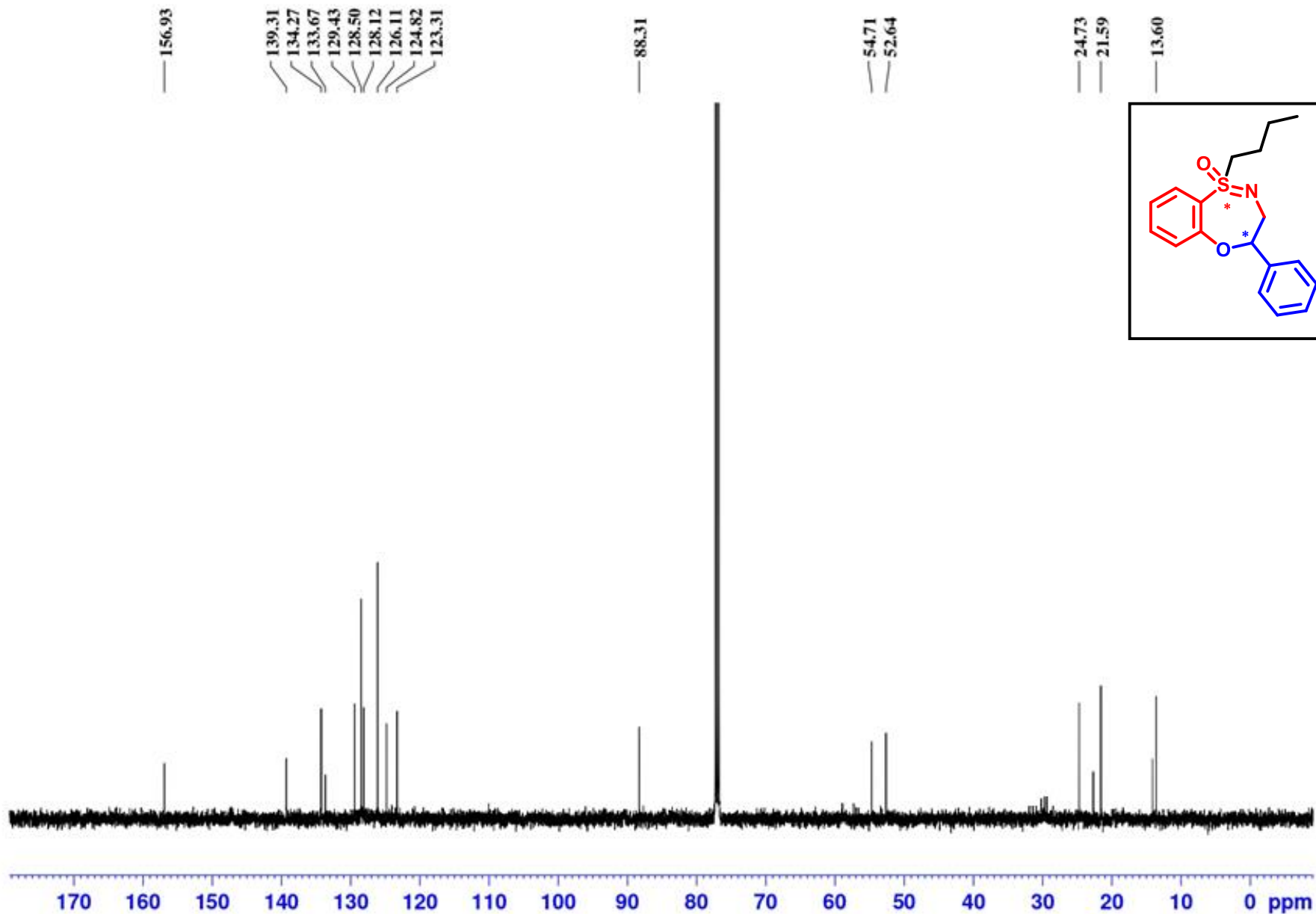


Fig S-50: ^{13}C NMR Spectra of Compound 4e (100 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21110FEB08	Original Data Path:	D:\INTERNAL NEW\2021\Feb 2021
Sample ID:	AB-152A	Sample Name:	
Acquisition Date:	02/10/21 11:02:11 AM	Run Time(min):	0.00
Vial:	CStk1-01:8	Injection Volume(μ l):	1.00

HRMS21110FEB08 #30-64 RT: 0.25-0.50 AV: 35 SB: 1 0.01 NL: 2.76E7
T: FTMS + c ESI Full ms [100.00-750.00]

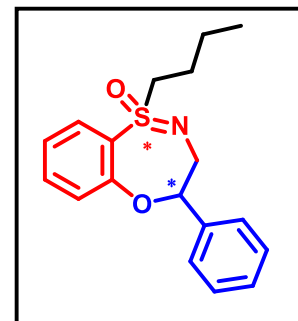
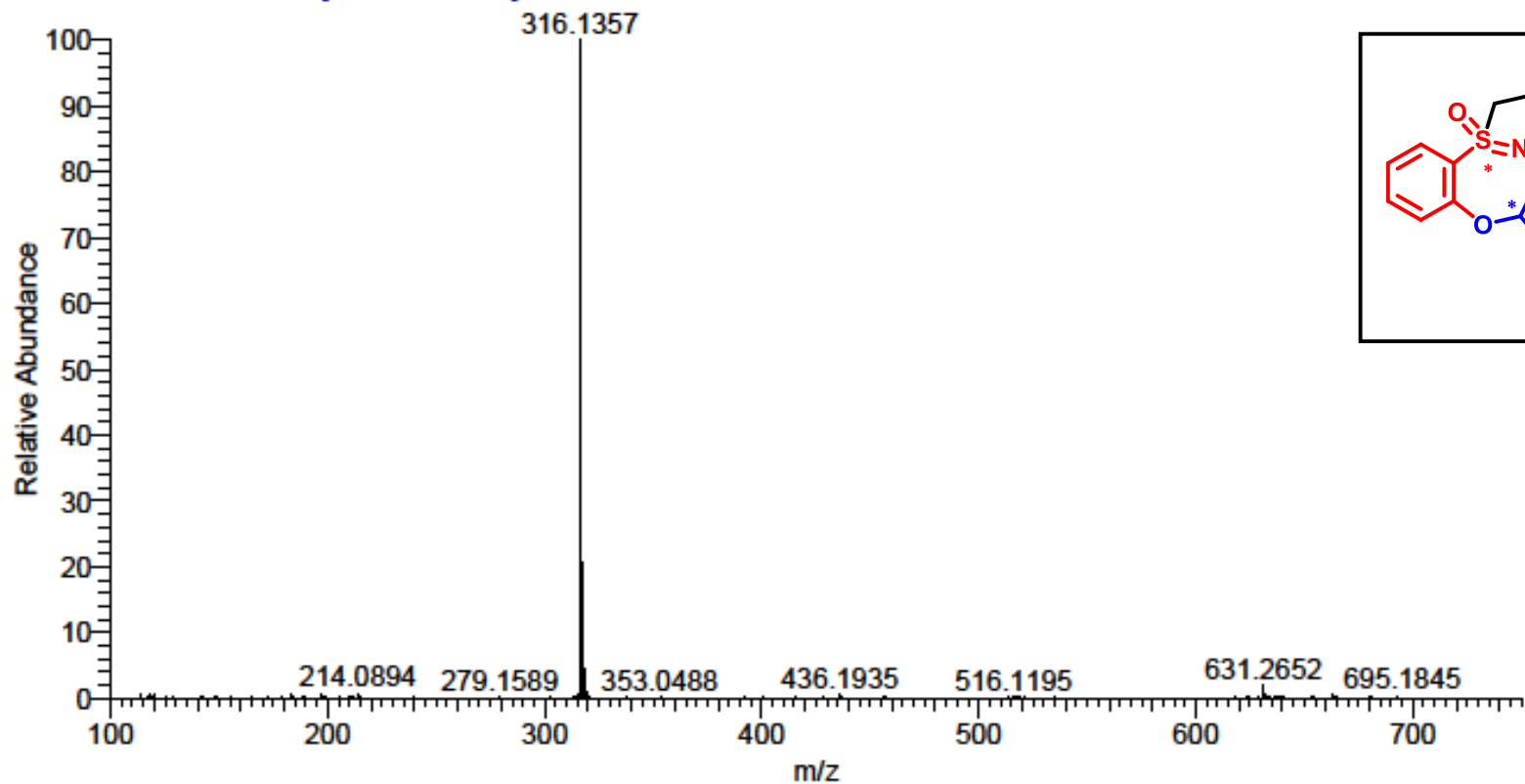


Fig S-51: HRMS report of Compound 4e

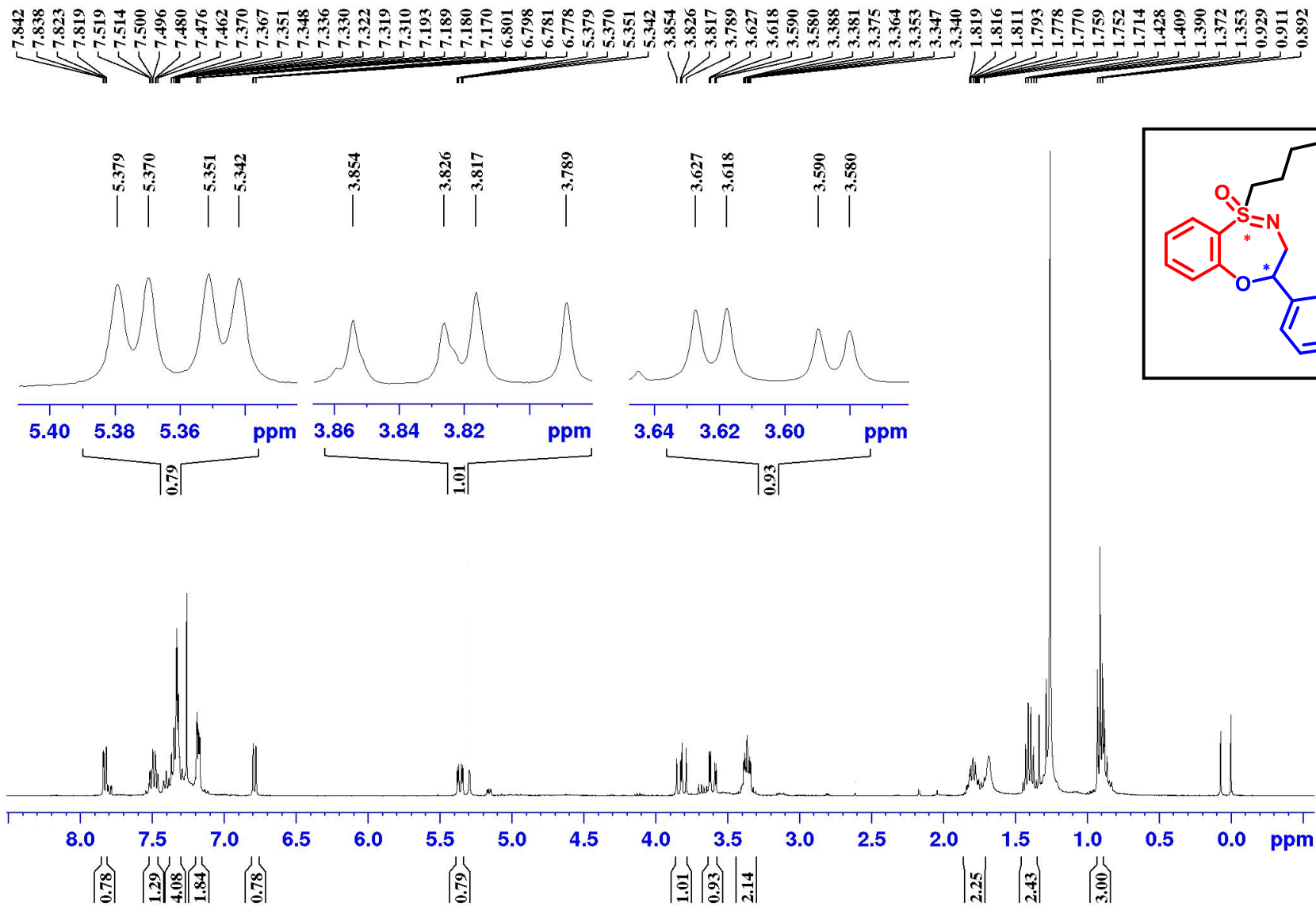


Fig S-52: ¹H NMR Spectra of Compound 4e' (400 MHz, CDCl₃)

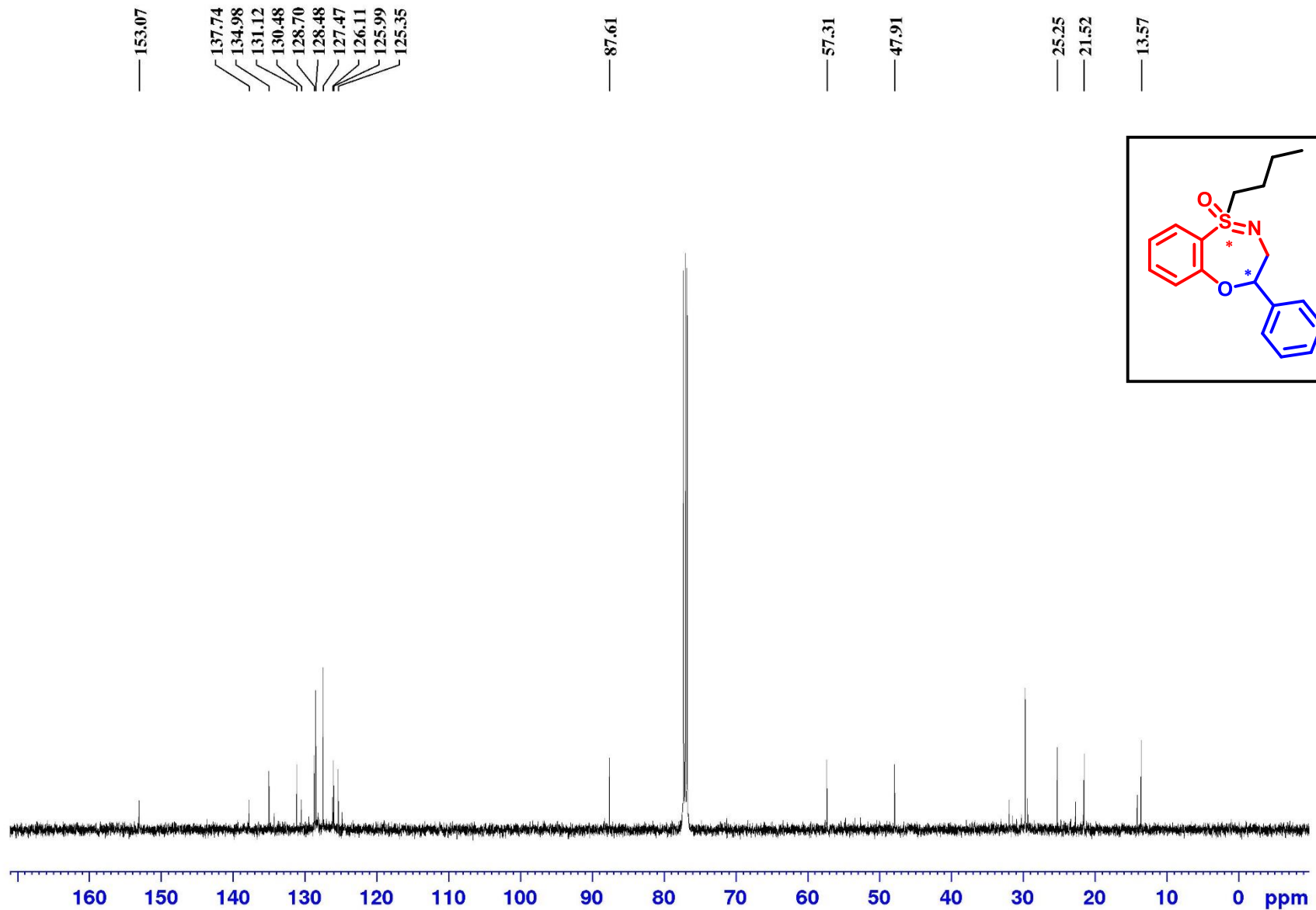


Fig S-53: ^{13}C NMR Spectra of Compound **4e'** (100 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21110FEB09	Original Data Path:	D:\INTERNAL NEW\2021\Feb 2021
Sample ID:	AB-152B	Sample Name:	
Acquisition Date:	02/10/21 11:04:10 AM	Run Time(min):	0.00
Vial:	CS&k1-01:9	Injection Volume(μl):	1.00

HRMS21110FEB09 #30-64 RT: 0.25-0.50 AV: 35 SB: 1 0.01 NL: 3.23E7
T: FTMS + c ESI Full ms [100.00-750.00]

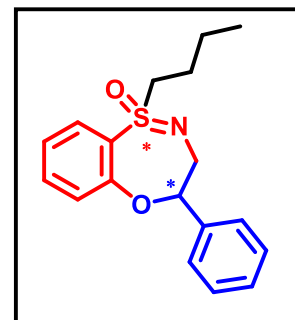
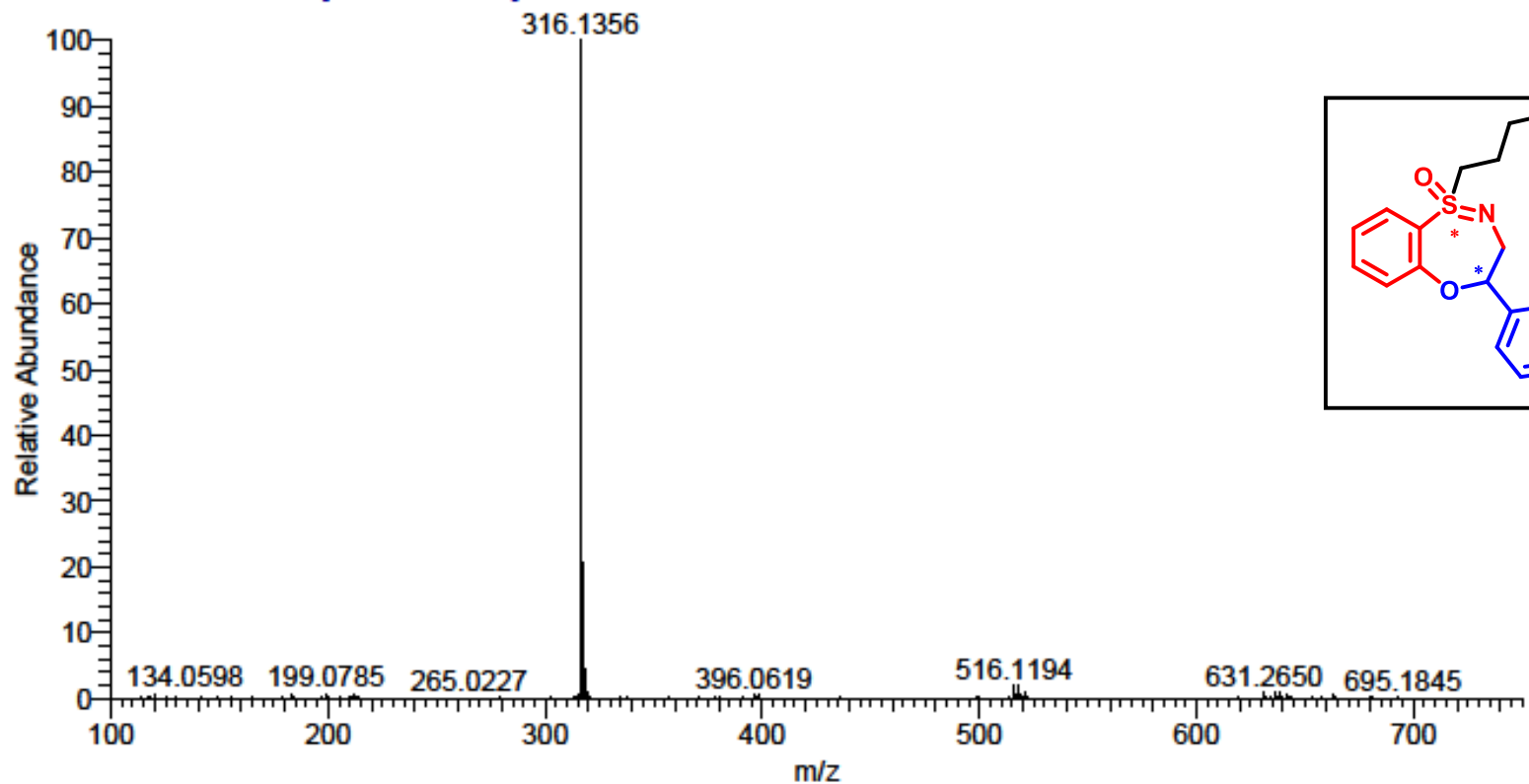


Fig S-54: HRMS report of Compound 4e'

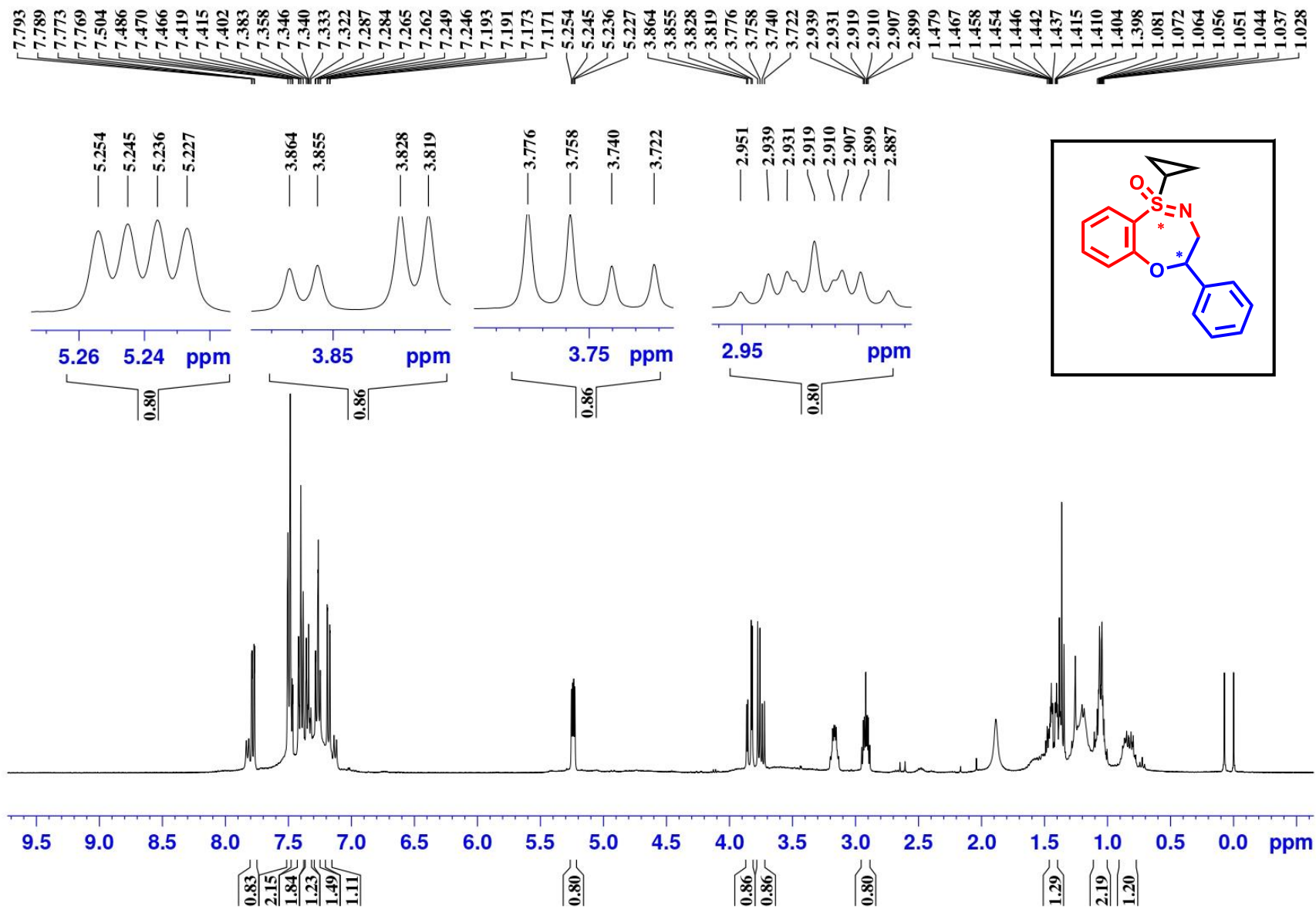


Fig S-55: ^1H NMR Spectra of Compound **4f** (400 MHz, CDCl_3)

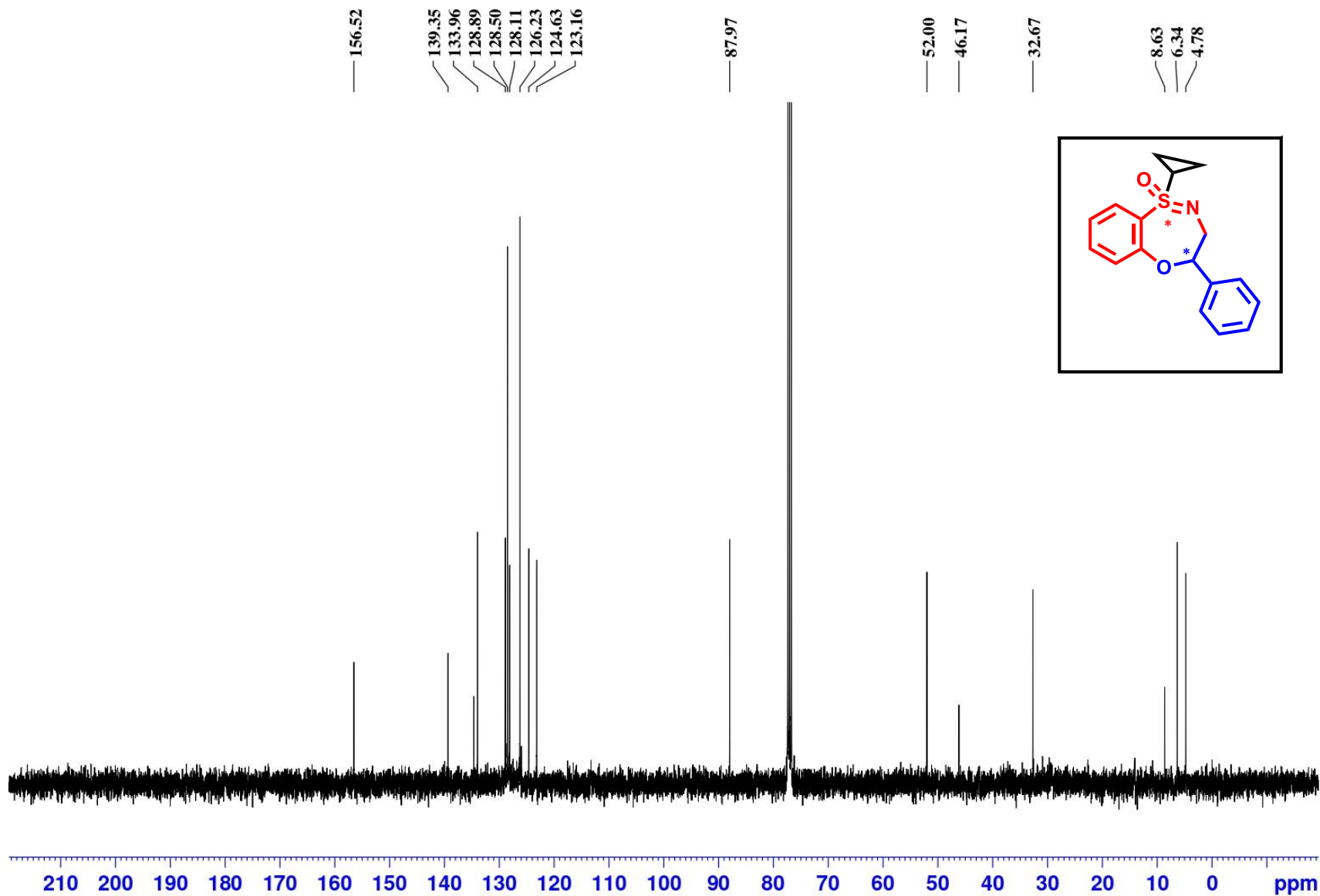


Fig S-56: ^{13}C NMR Spectra of Compound **4f** (100 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I22FEB15	Original Data Path:	D:\INTERNAL NEW\2021\Feb 2021
Sample ID:	AB-157A	Sample Name:	
Acquisition Date:	02/22/21 11:41:52 AM	Run Time(min):	0.00
Vial:	CStk1-01:15	Injection Volume(μl):	1.00

HRMS21I22FEB15 #30-62 RT: 0.25-0.50 AV: 33 SB: 1 0.01 NL: 6.63E6
T: FTMS + c ESI Full ms [100.00-750.00]

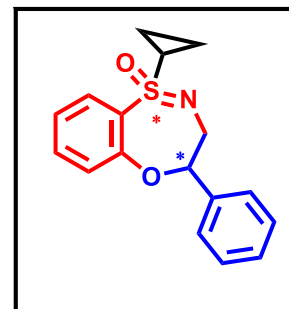
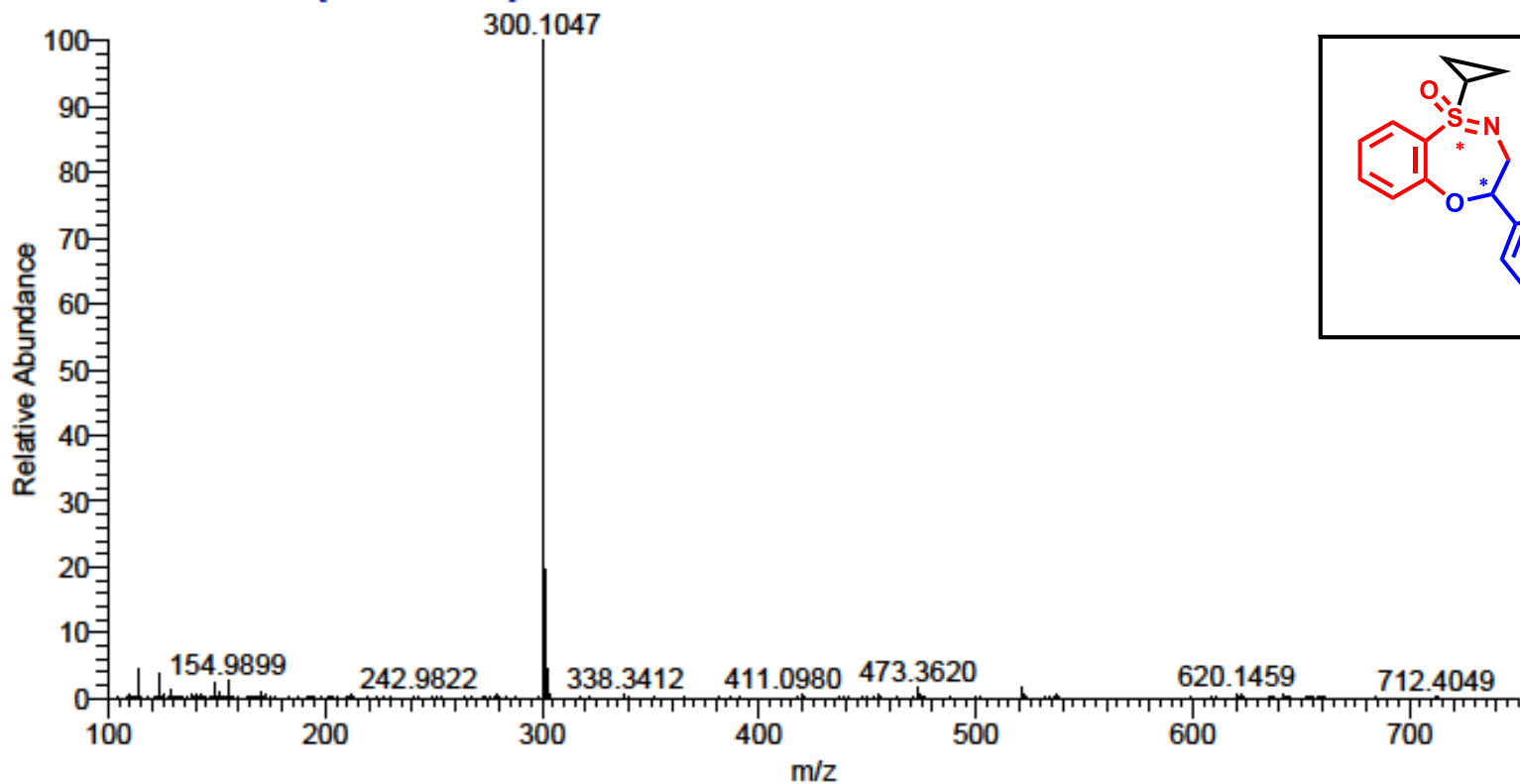


Fig S-57: HRMS report of Compound 4f

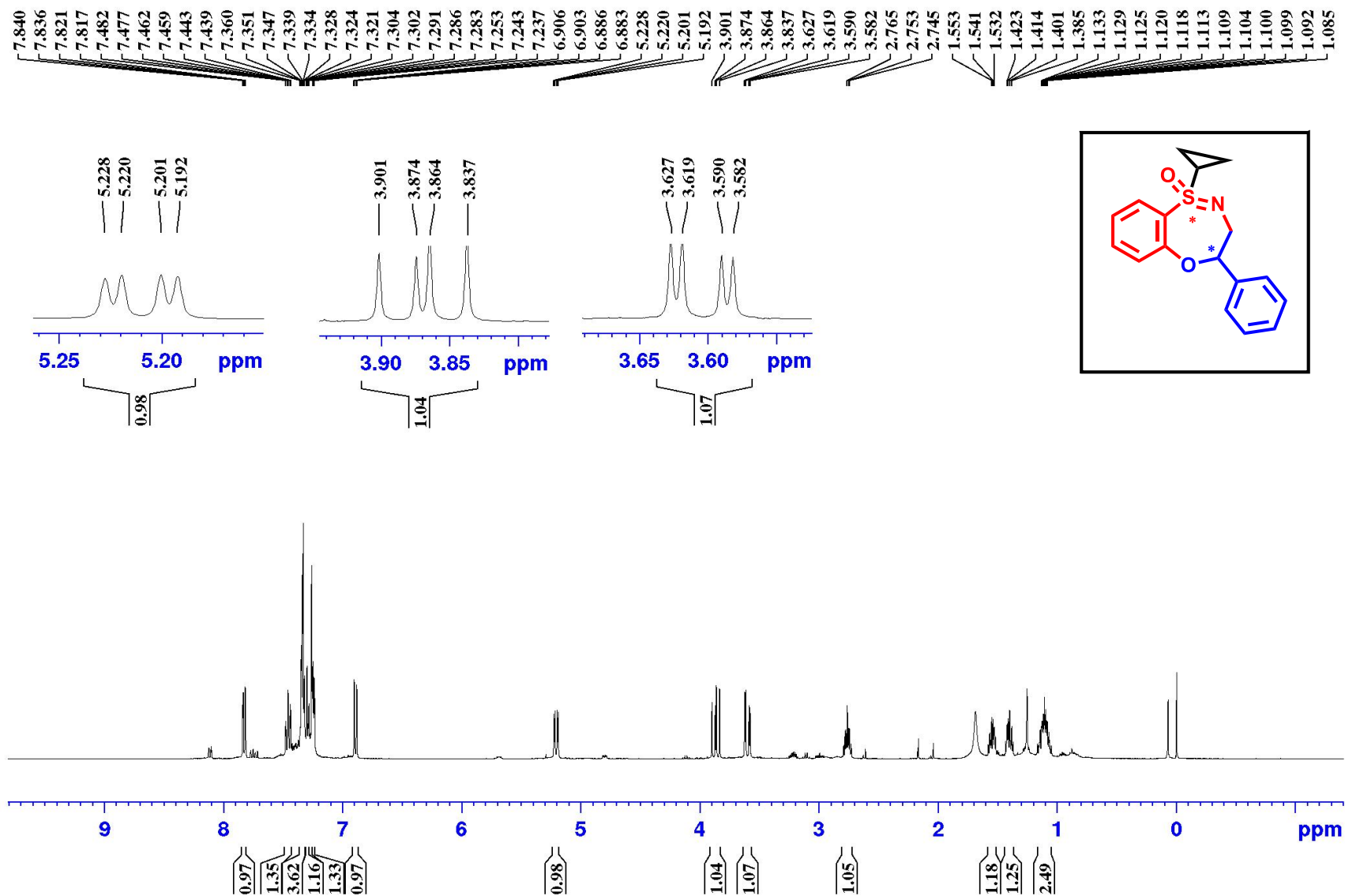


Fig S-58: ¹H NMR Spectra of Compound 4f' (400 MHz, CDCl₃)

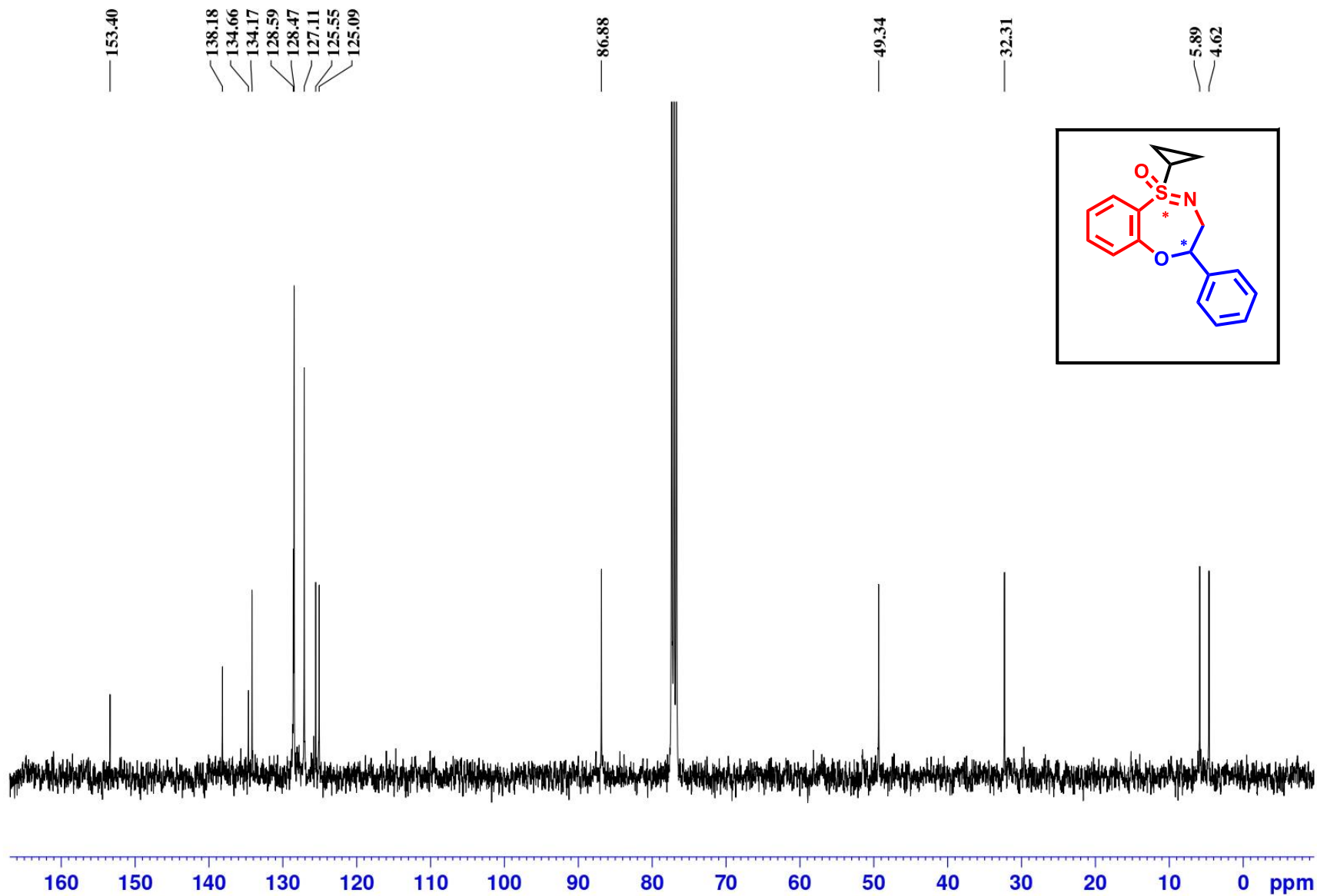


Fig S-59: ^{13}C NMR Spectra of Compound 4f' (100 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I22FEB16	Original Data Path:	D:\INTERNAL NEW\2021\Feb 2021
Sample ID:	AB-157B	Sample Name:	
Acquisition Date:	02/22/21 11:43:51 AM	Run Time(min):	0.00
Vial:	CS&k1-01:16	Injection Volume(μl):	1.00

HRMS21I22FEB16 #30-63 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 8.48E6
T: FTMS + c ESI Full ms [100.00-750.00]

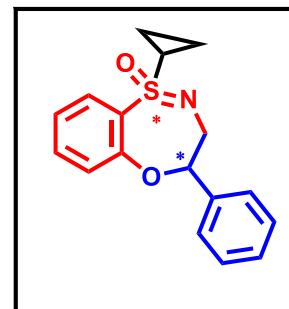
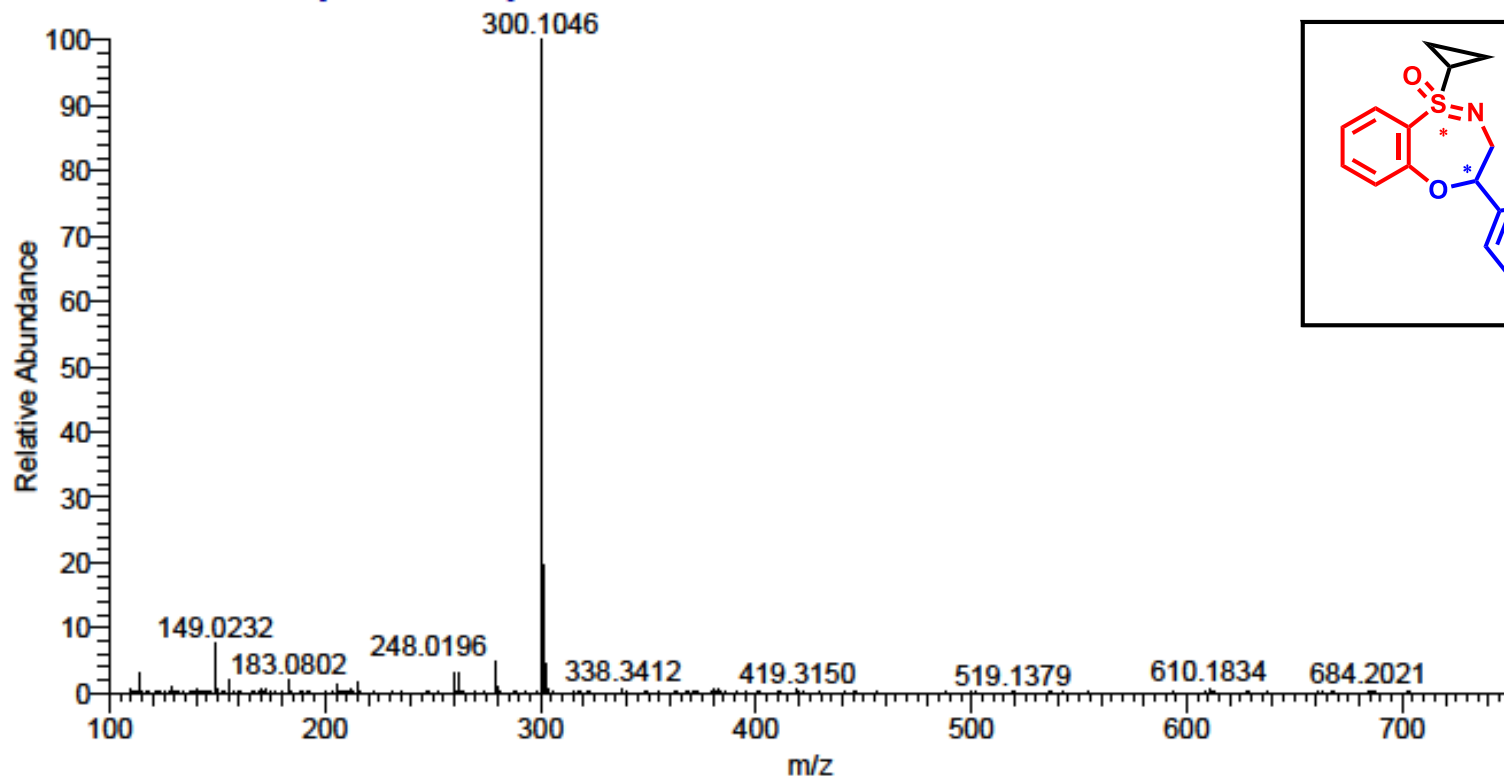


Fig S-60: HRMS report of Compound 4f'

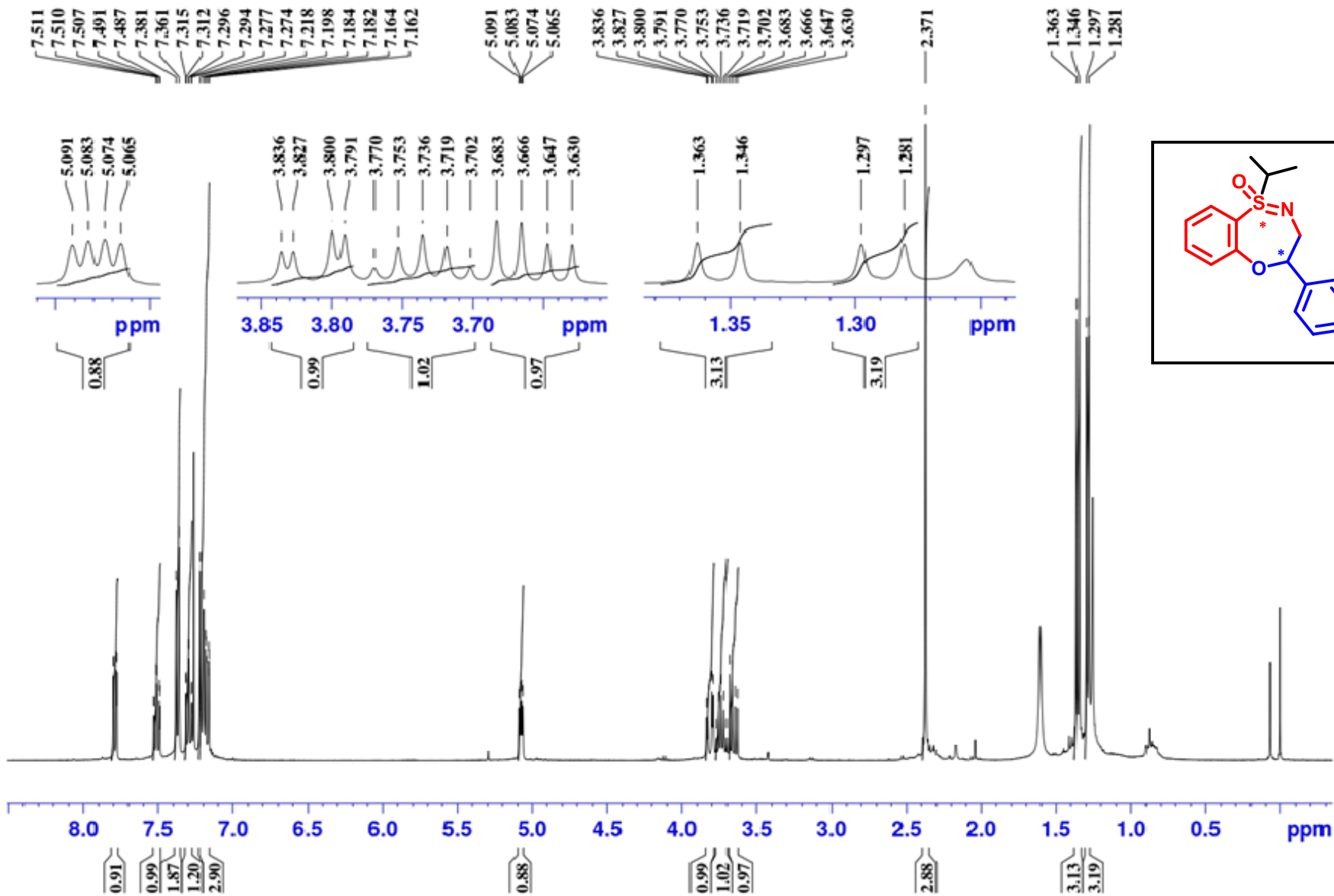


Fig S-61: ^1H NMR Spectra of Compound **4g** (400 MHz, CDCl_3)

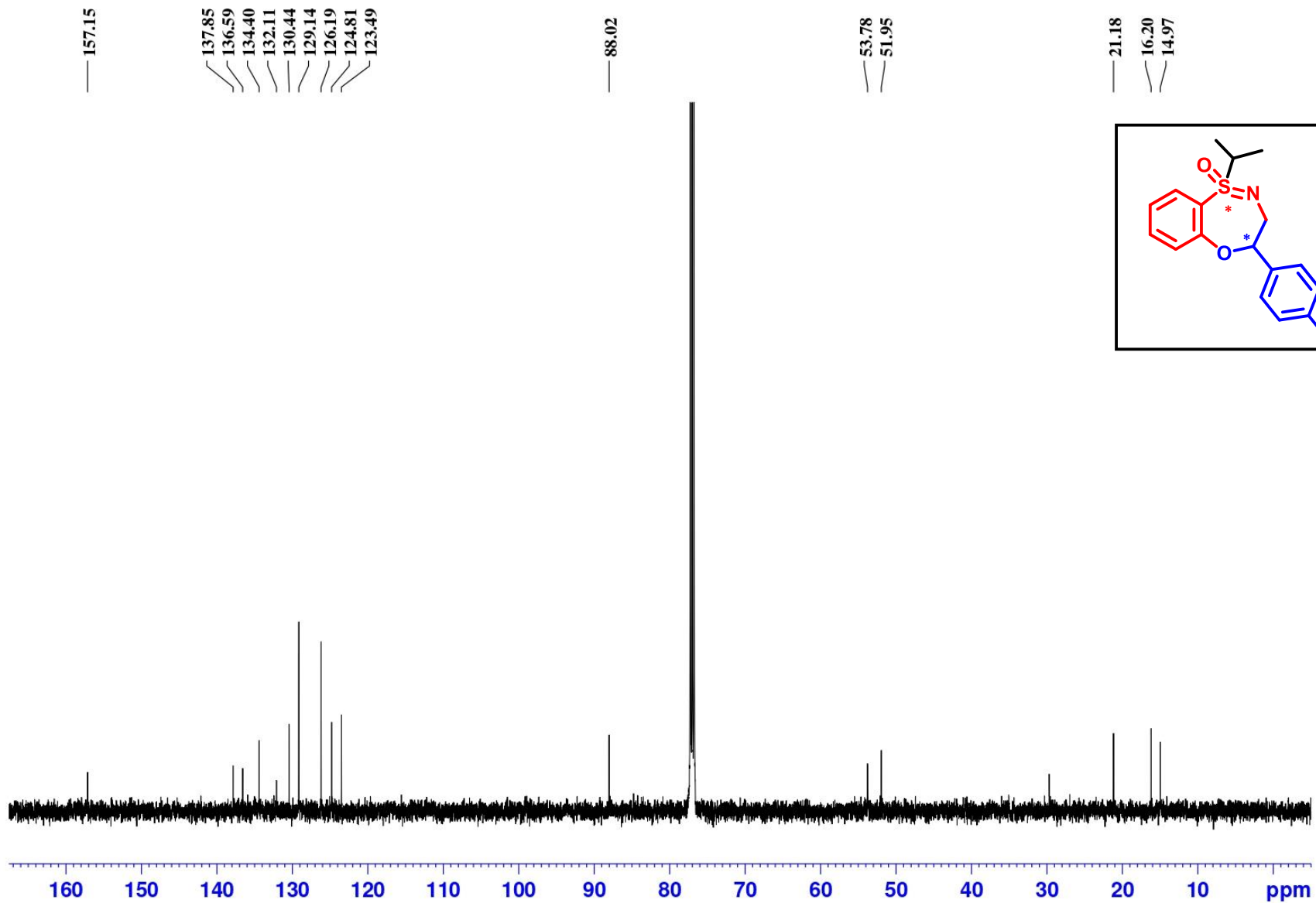


Fig S-62: ^{13}C NMR Spectra of Compound **4g** (125 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I02MAR20	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	AB-162A	Sample Name:	
Acquisition Date:	03/02/21 11:34:09 AM	Run Time(min):	0.00
Vial:	CStk1-01:20	Injection Volume(μ l):	1.00

HRMS21I02MAR20 #26-57 RT: 0.25-0.50 AV: 32 SB: 1 0.01 NL: 4.45E6
T: FTMS + c ESI Full ms [100.00-750.00]

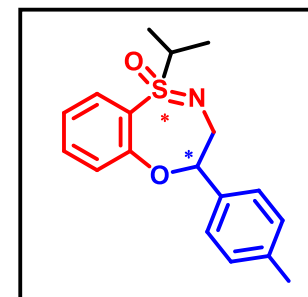
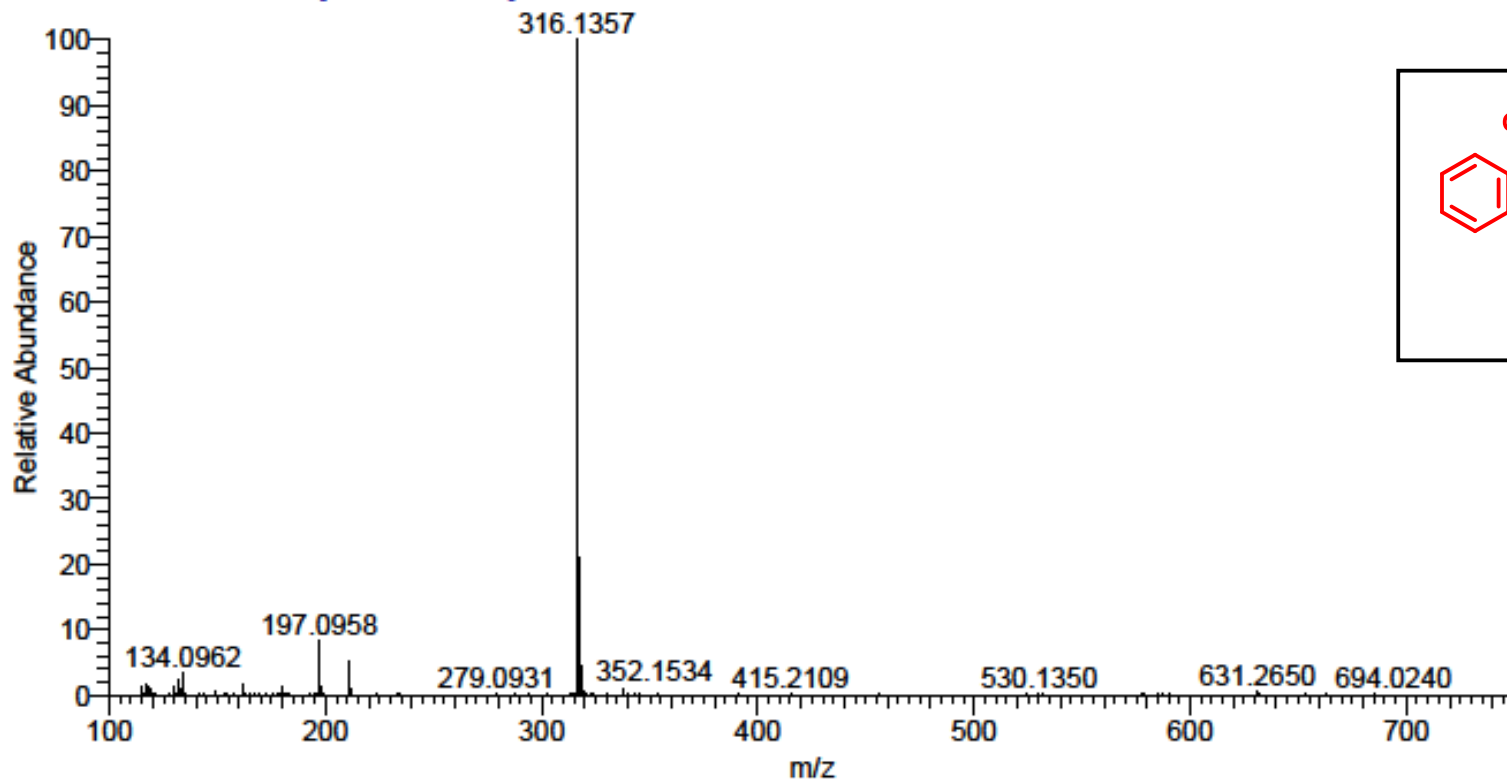


Fig S-63: HRMS report of Compound 4g

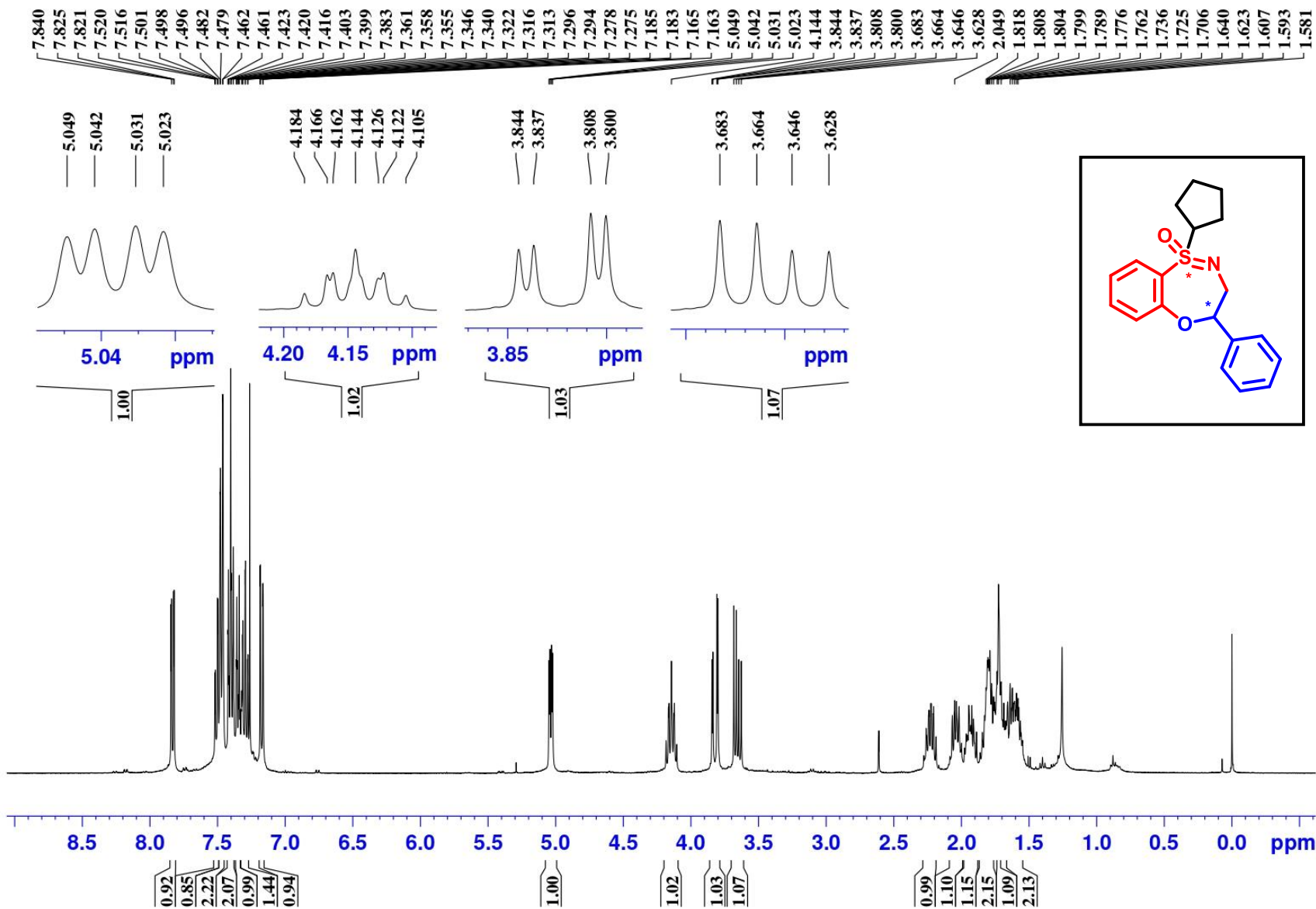


Fig S-64: ^1H NMR Spectra of Compound **4h** (400 MHz, CDCl_3)

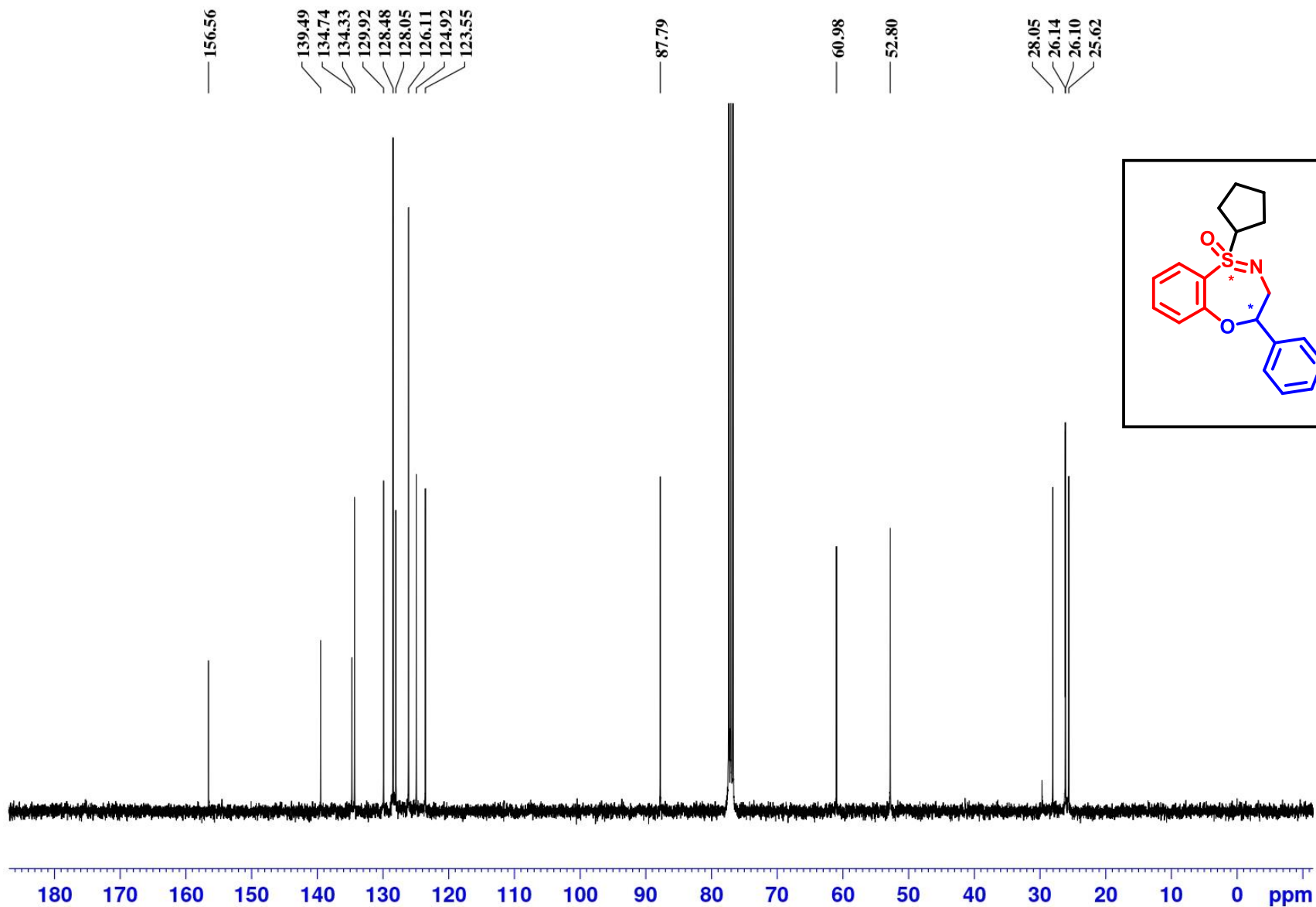


Fig S-65: ^{13}C NMR Spectra of Compound **4h** (100 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I09MAR16	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	AB-164A	Sample Name:	
Acquisition Date:	03/09/21 11:11:18 AM	Run Time(min):	0.00
Vial:	CStk1-01:16	Injection Volume(μl):	1.00

HRMS21I09MAR16 #30-63 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 1.47E7
T: FTMS + c ESI Full ms [100.00-750.00]

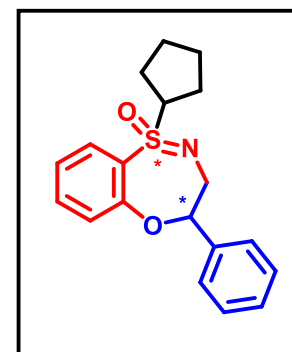
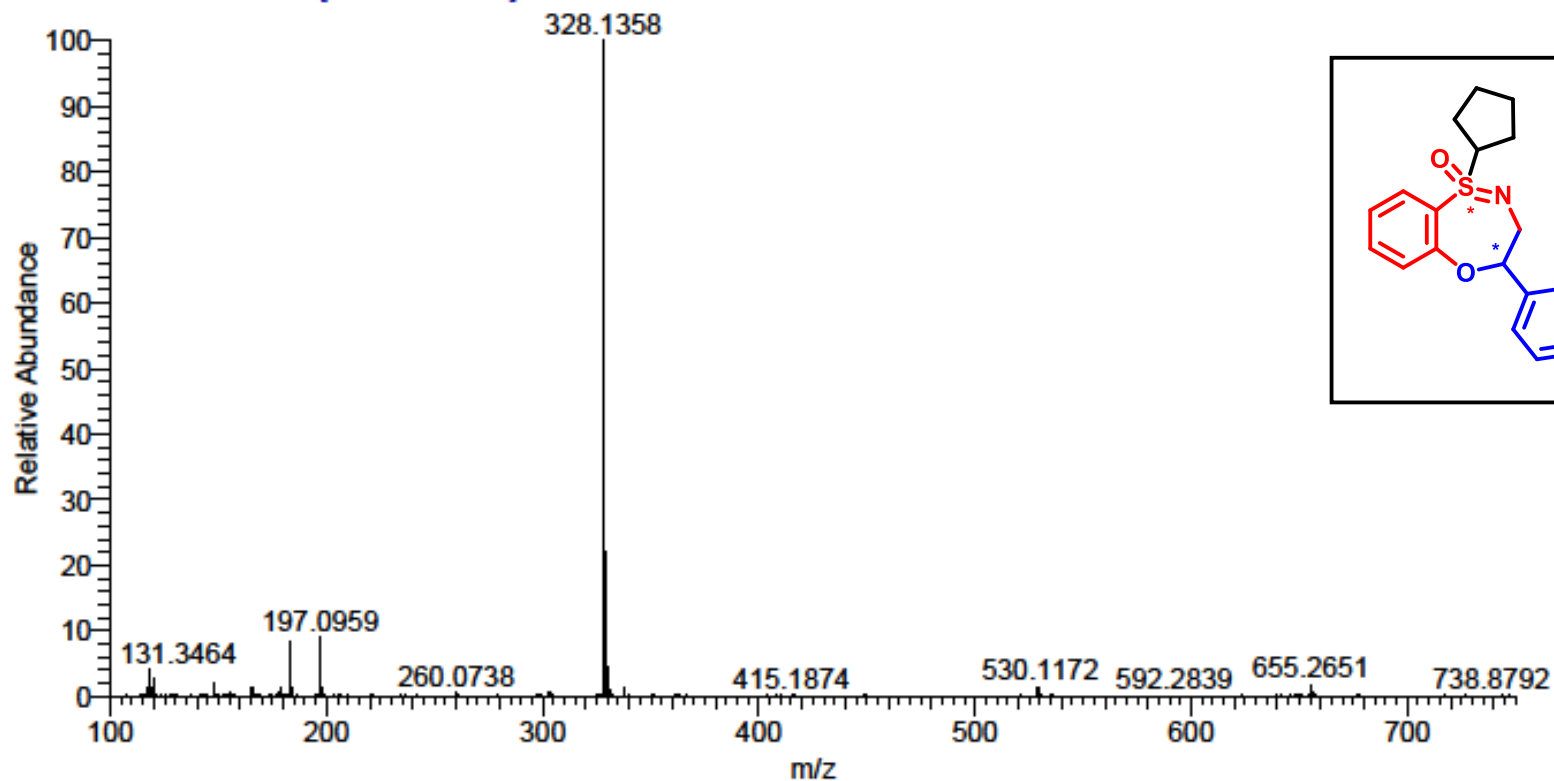


Fig S-66: HRMS report of Compound 4h

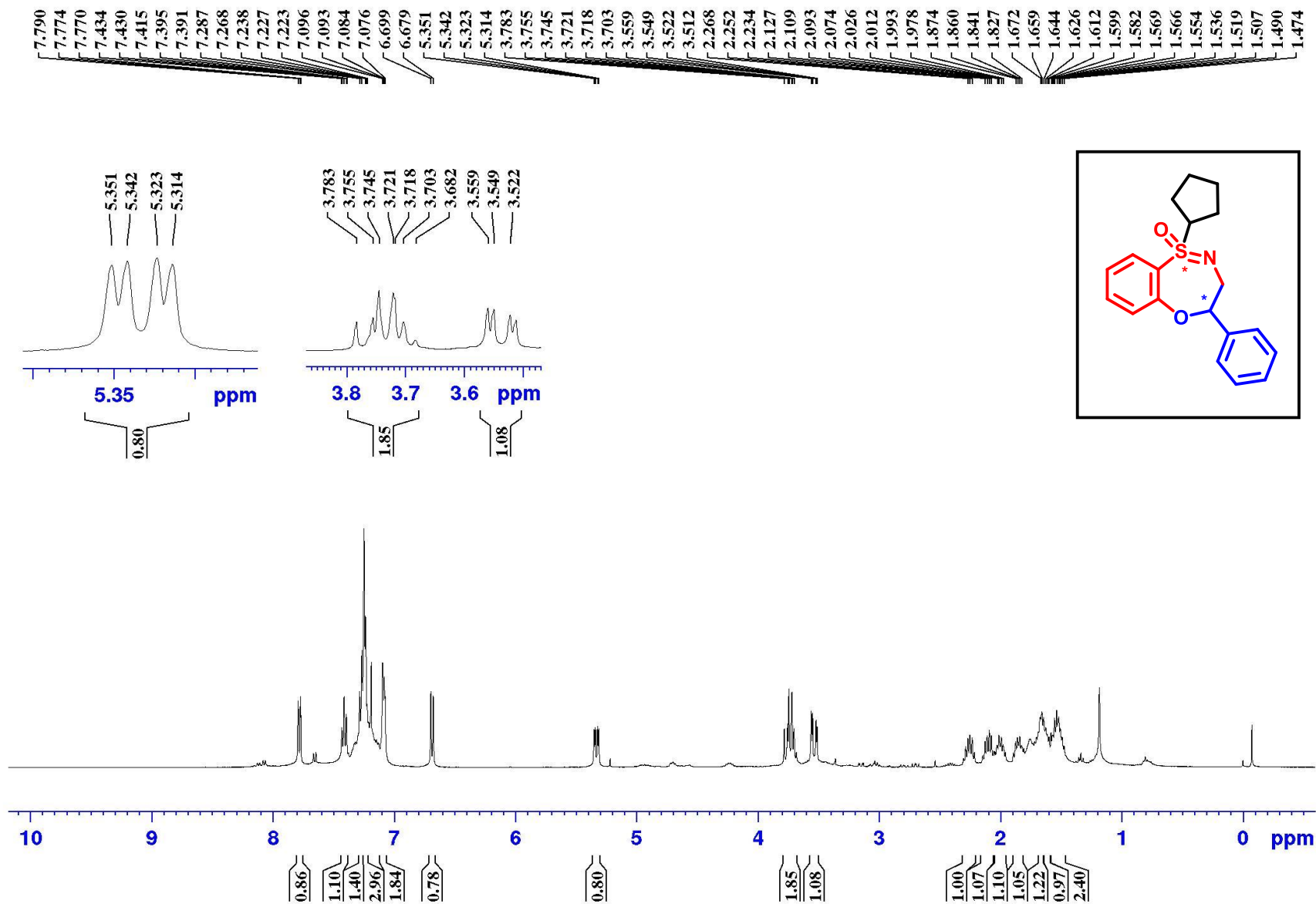


Fig S-67: ¹H NMR Spectra of Compound 4h' (400 MHz, CDCl₃)

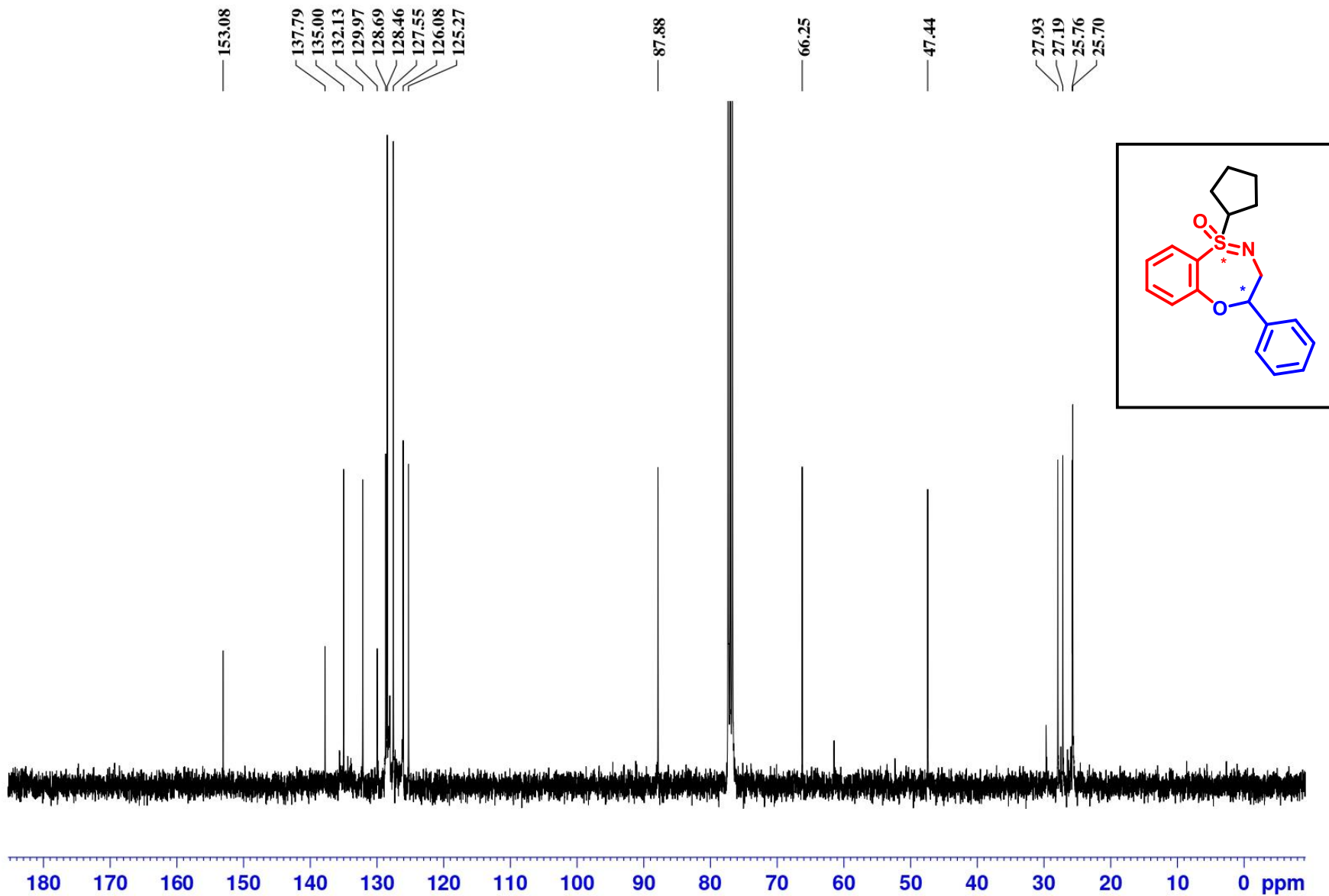


Fig S-68: ^{13}C NMR Spectra of Compound 4h' (100 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I09MAR17	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	AB-164B	Sample Name:	
Acquisition Date:	03/09/21 11:13:17 AM	Run Time(min):	0.00
Vial:	CS&k1-01:17	Injection Volume(μl):	1.00

HRMS21I09MAR17 #30-62 RT: 0.25-0.50 AV: 33 SB: 1 0.01 NL: 6.07E6
T: FTMS + c ESI Full ms [100.00-750.00]

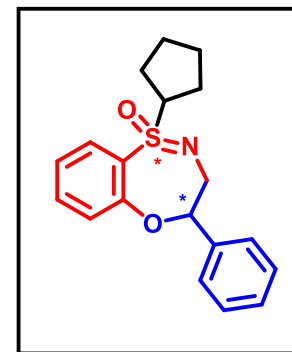
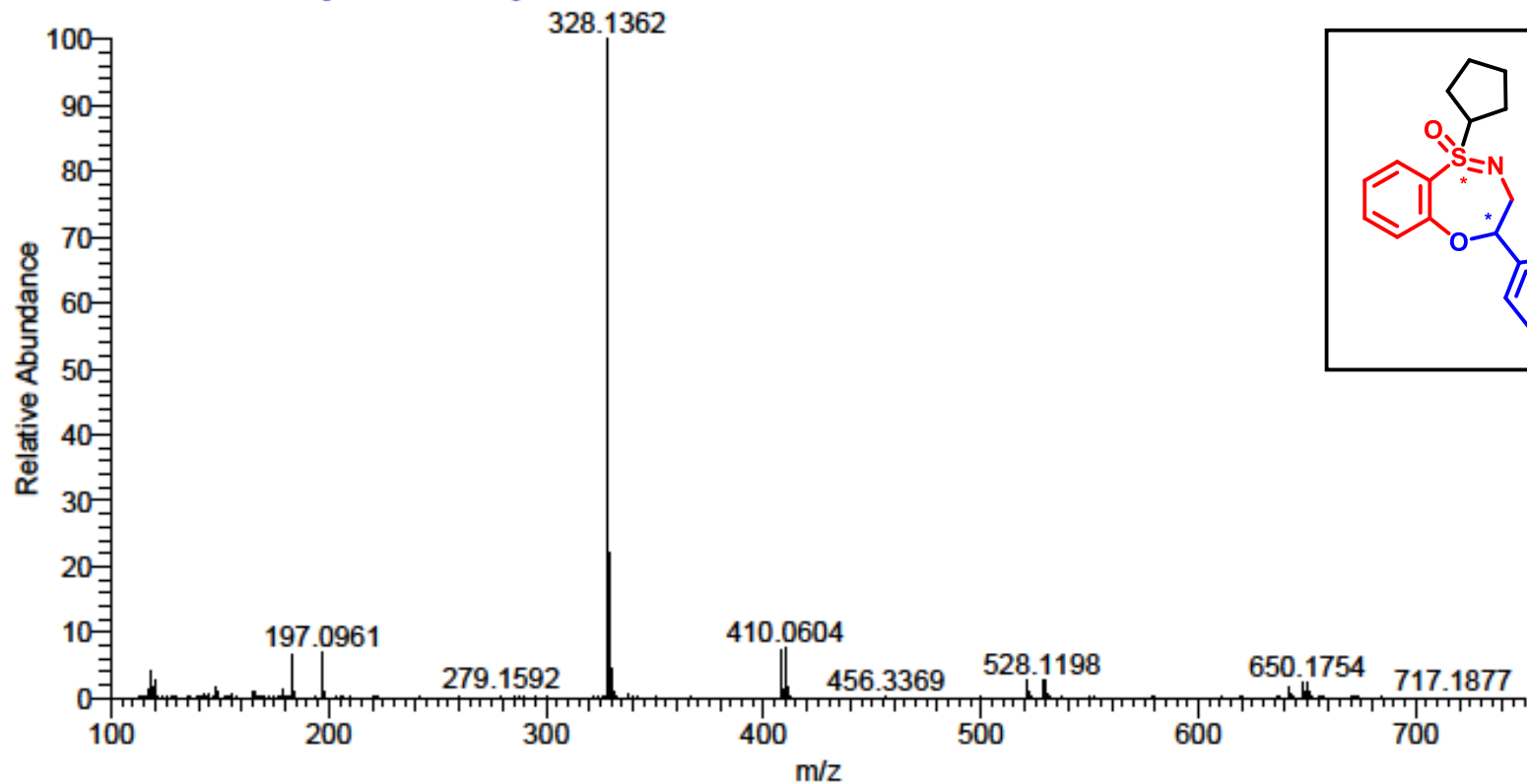


Fig S-69: HRMS report of Compound 4h'

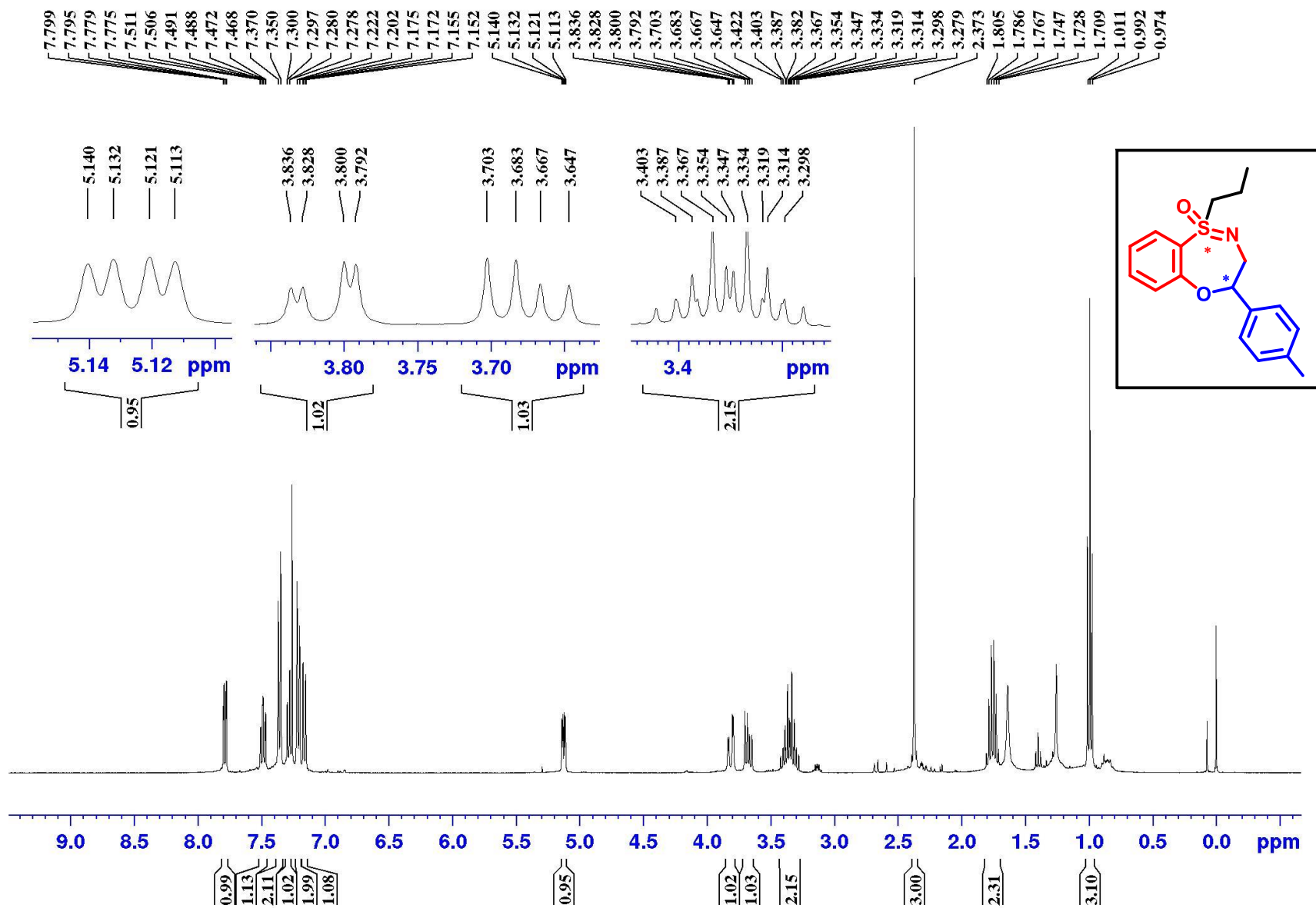


Fig S-70: ^1H NMR Spectra of Compound **4i** (400 MHz, CDCl_3)

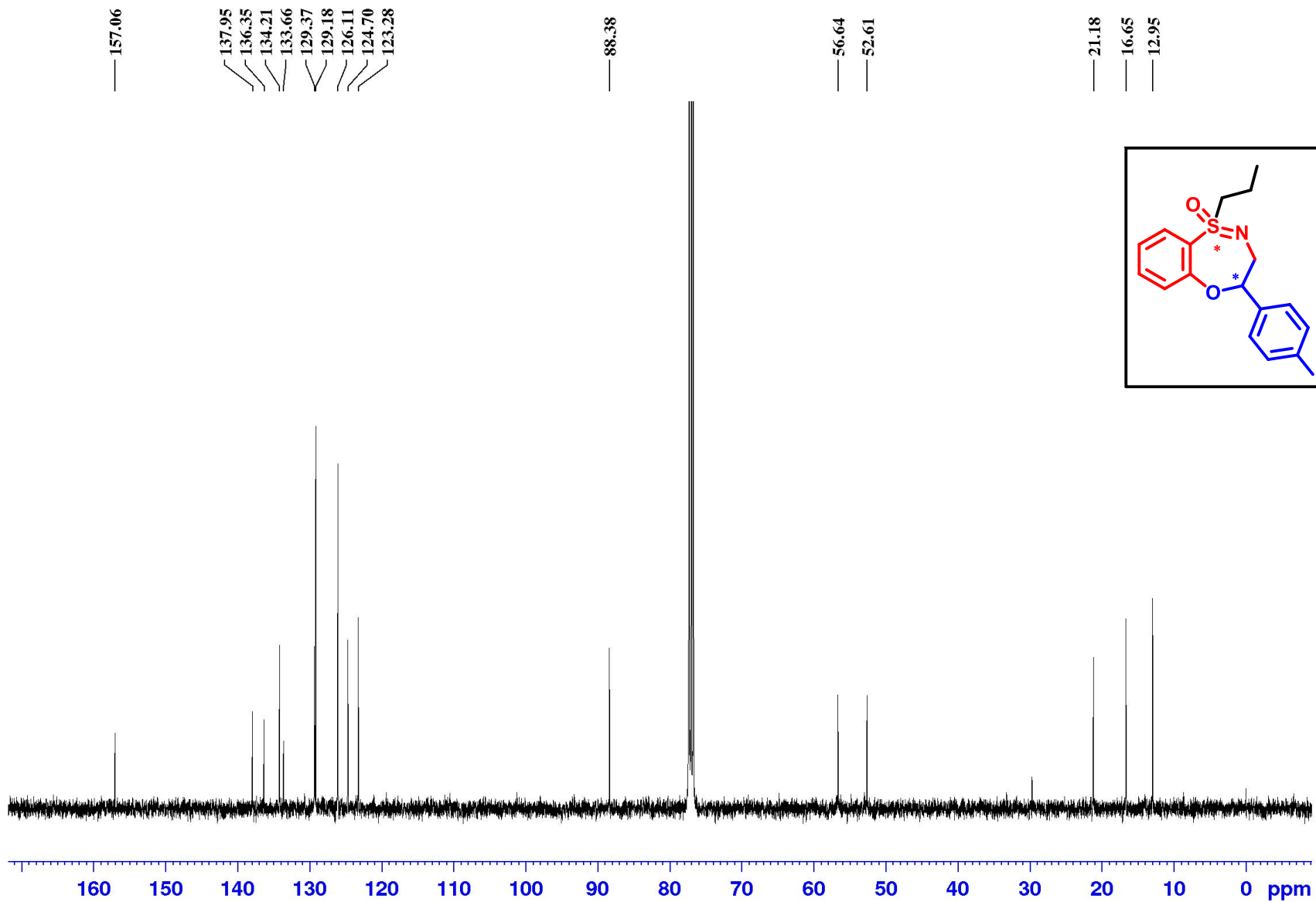


Fig S-71: ¹³C NMR Spectra of Compound **4i** (100 MHz, CDCl₃)

SAIF [HRMS Report]

Data File:	HRMS21I18MAR34	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	AB-166A	Sample Name:	
Acquisition Date:	03/18/21 11:52:52 AM	Run Time(min):	0.00
Vial:	CStk1-01:34	Injection Volume(μl):	1.00

HRMS21I18MAR34 #32-64 RT: 0.25-0.50 AV: 33 SB: 1 0.01 NL: 3.70E6
T: FTMS + c ESI Full ms [100.00-750.00]

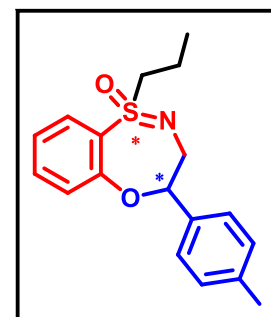
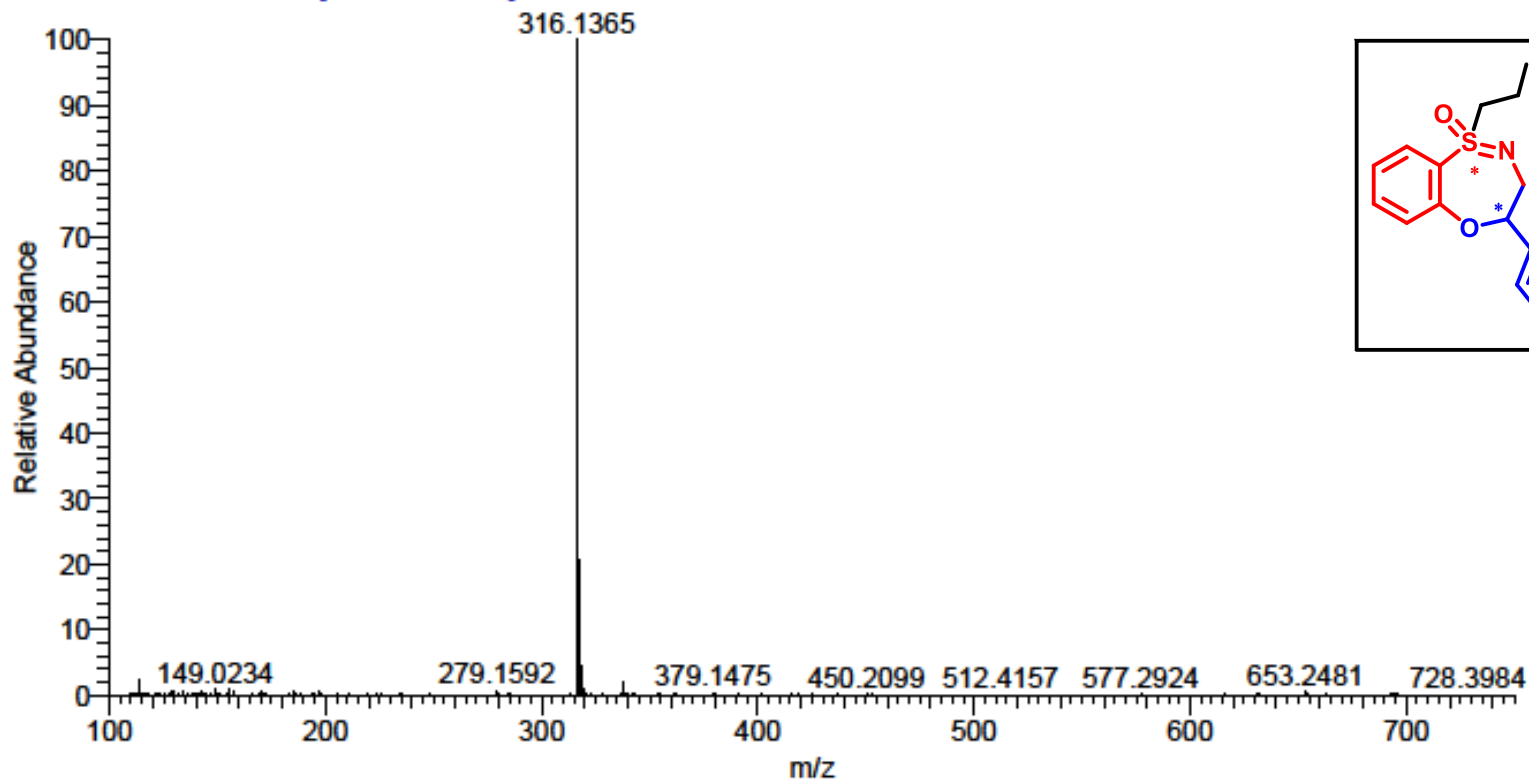


Fig S-72: HRMS report of Compound 4i

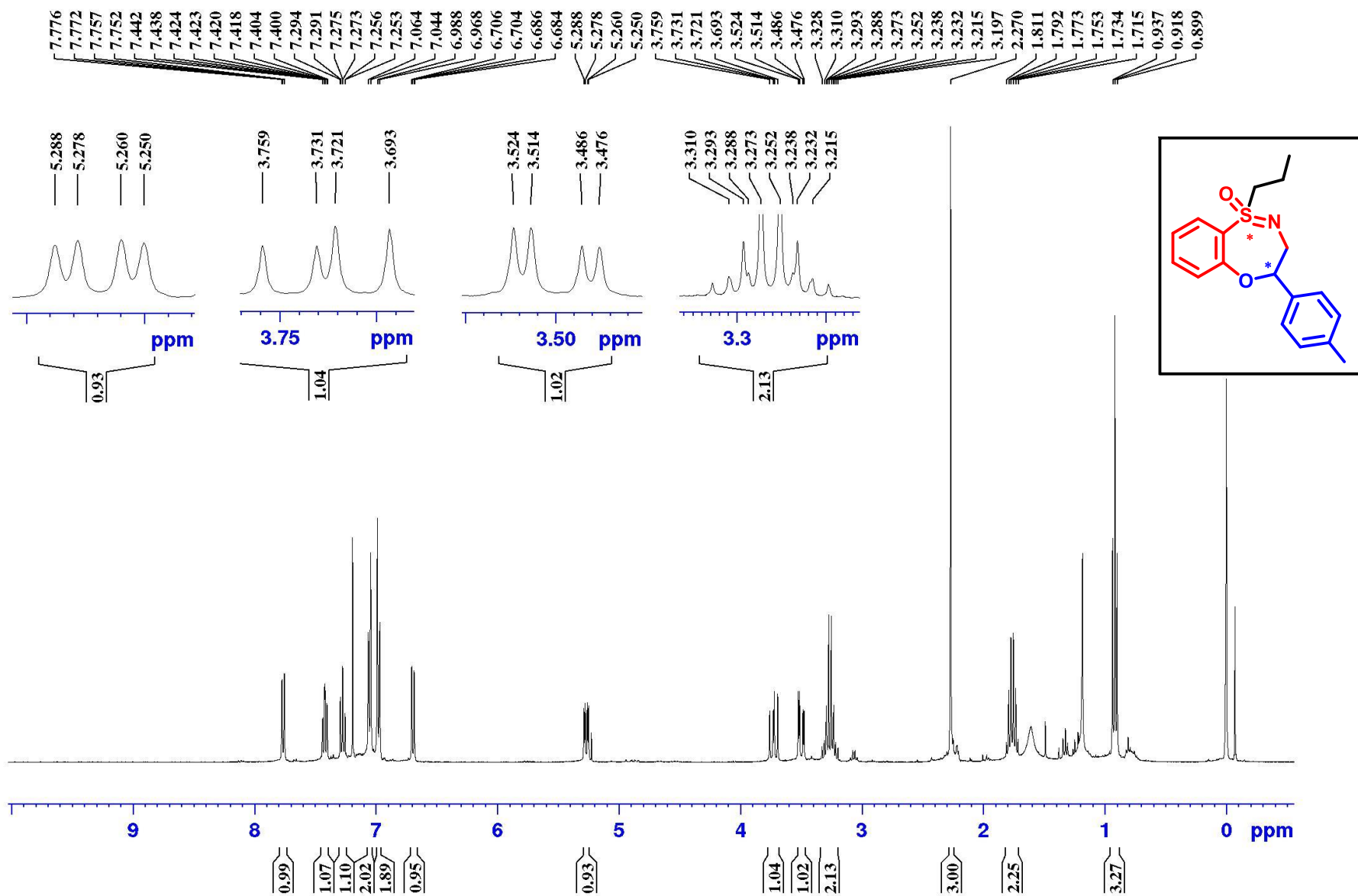


Fig S-73: ^1H NMR Spectra of Compound 4i' (400 MHz, CDCl_3)

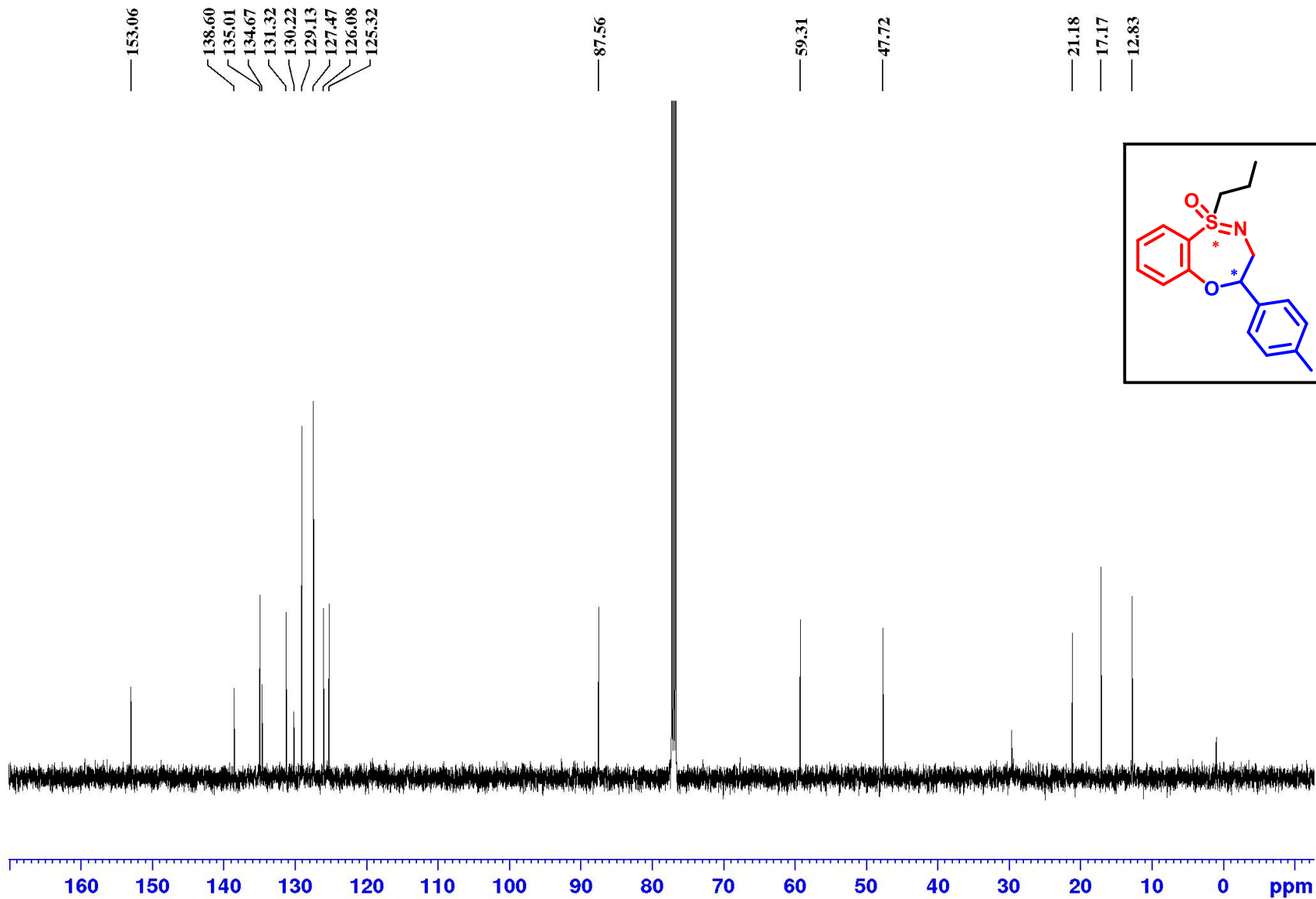


Fig S-74: ^{13}C NMR Spectra of Compound 4i' (100 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I17MAR12	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	AB-166B	Sample Name:	
Acquisition Date:	03/17/21 11:26:08 AM	Run Time(min):	0.00
Vial:	CStk1-01:12	Injection Volume(μl):	1.00

HRMS21I17MAR12 #32-65 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 7.06E6
T: FTMS + c ESI Full ms [100.00-750.00]

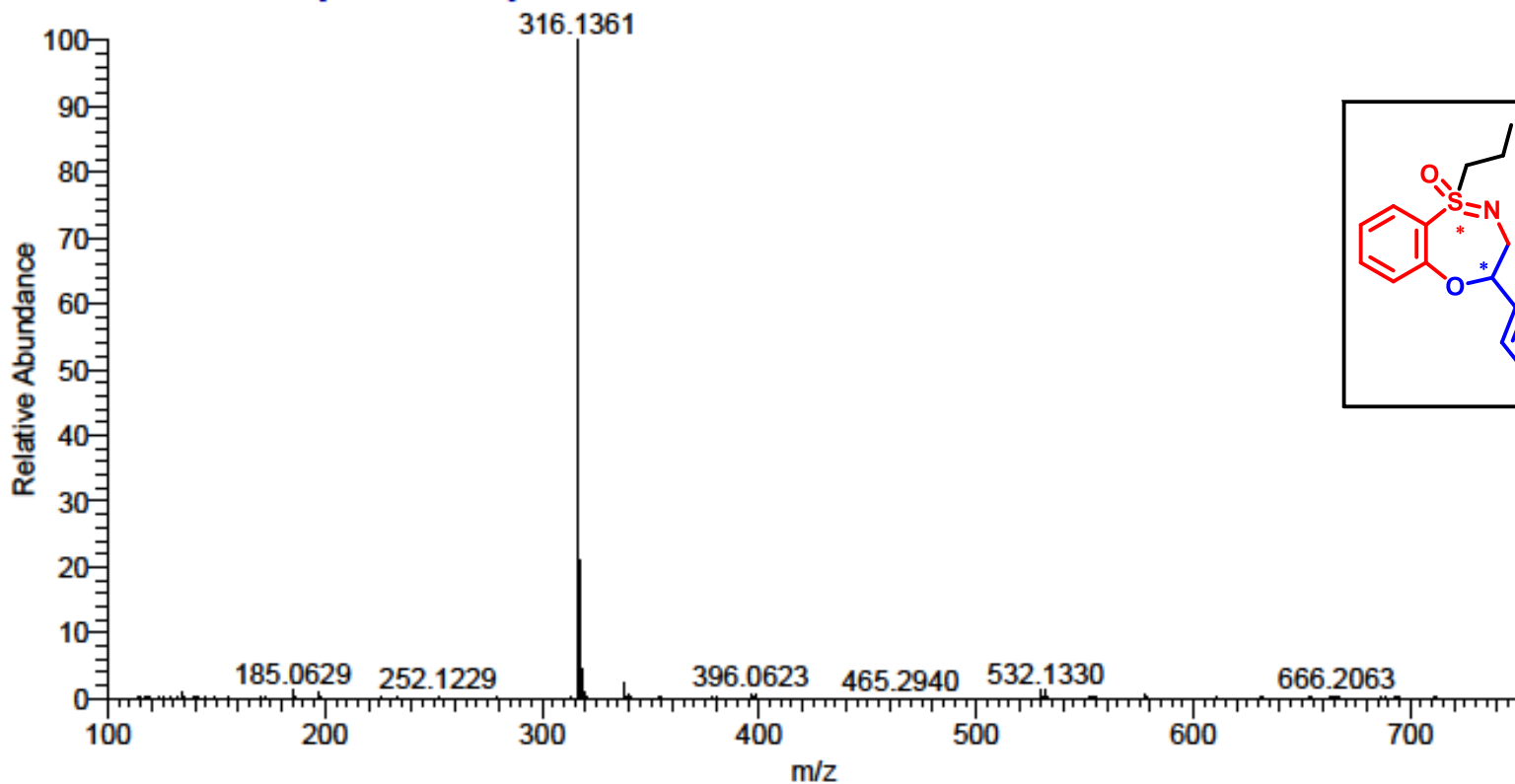


Fig S-75: HRMS report of Compound 4i'

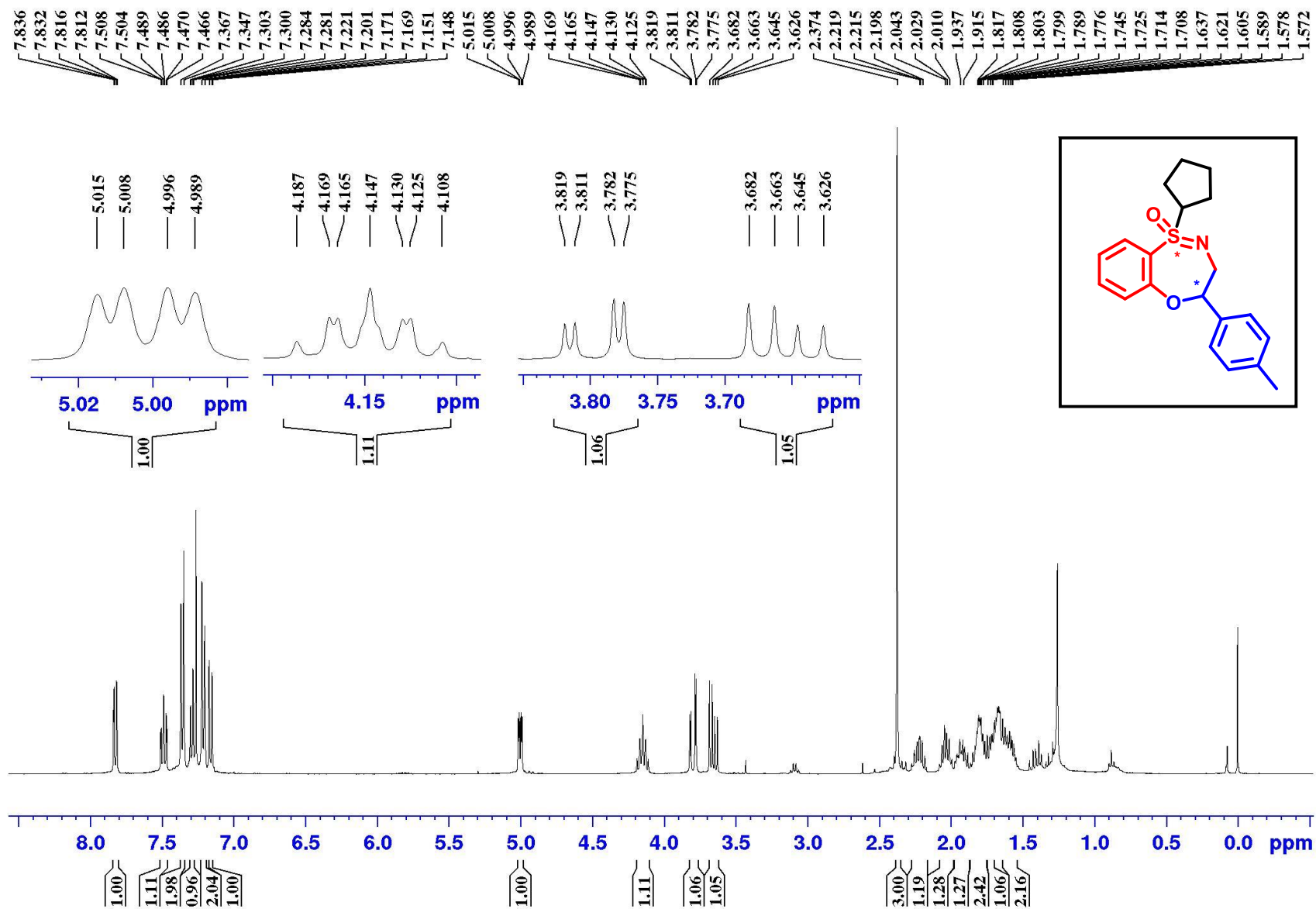


Fig S-76: ¹H NMR Spectra of Compound 4j (400 MHz, CDCl₃)

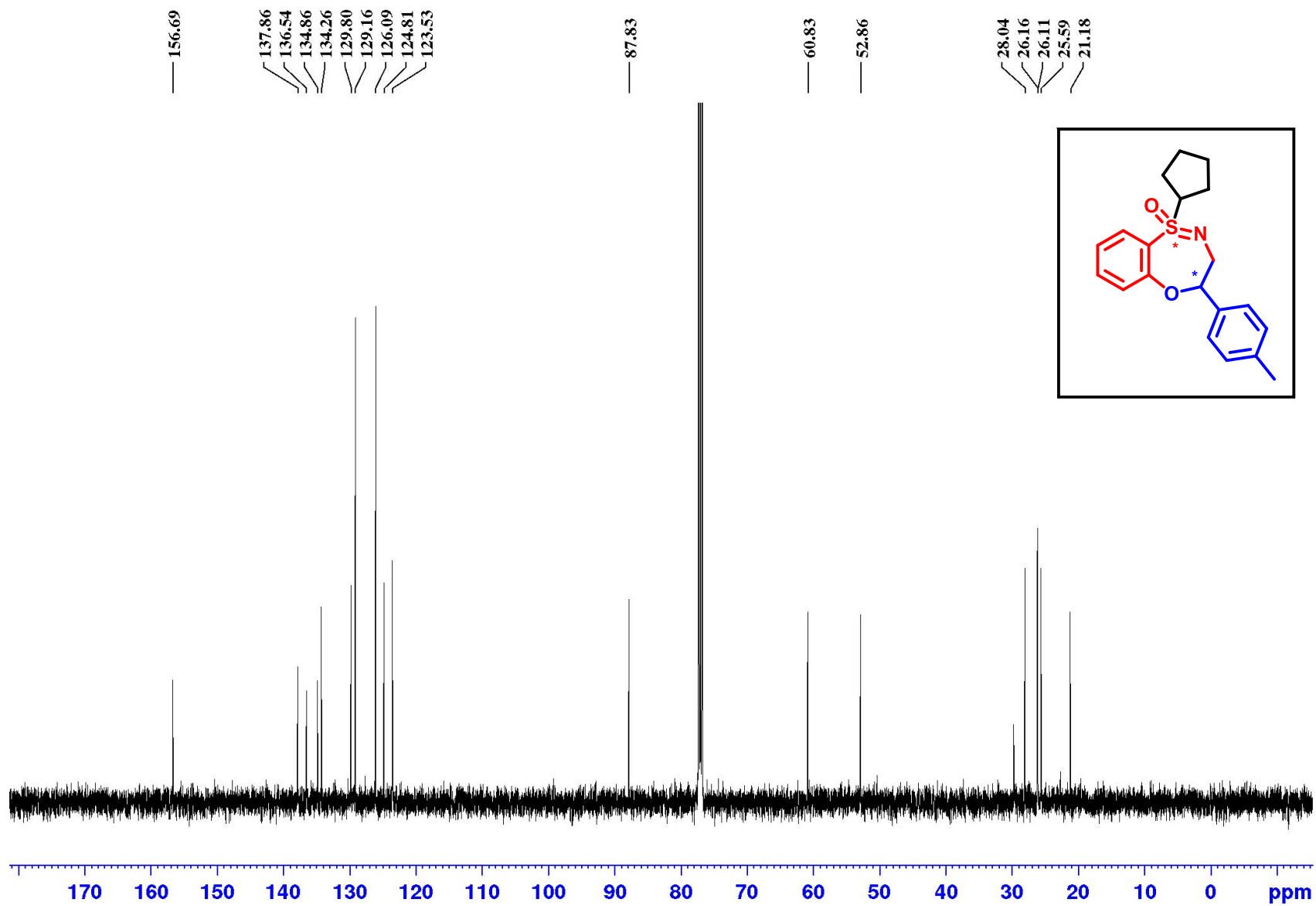


Fig S-77: ^{13}C NMR Spectra of Compound 4j (100 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I17MAR13	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	AB-167A	Sample Name:	
Acquisition Date:	03/17/21 11:28:07 AM	Run Time(min):	0.00
Vial:	CStk1-01:13	Injection Volume(μl):	1.00

HRMS21I17MAR13 #32-65 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 5.20E6
T: FTMS + c ESI Full ms [100.00-750.00]

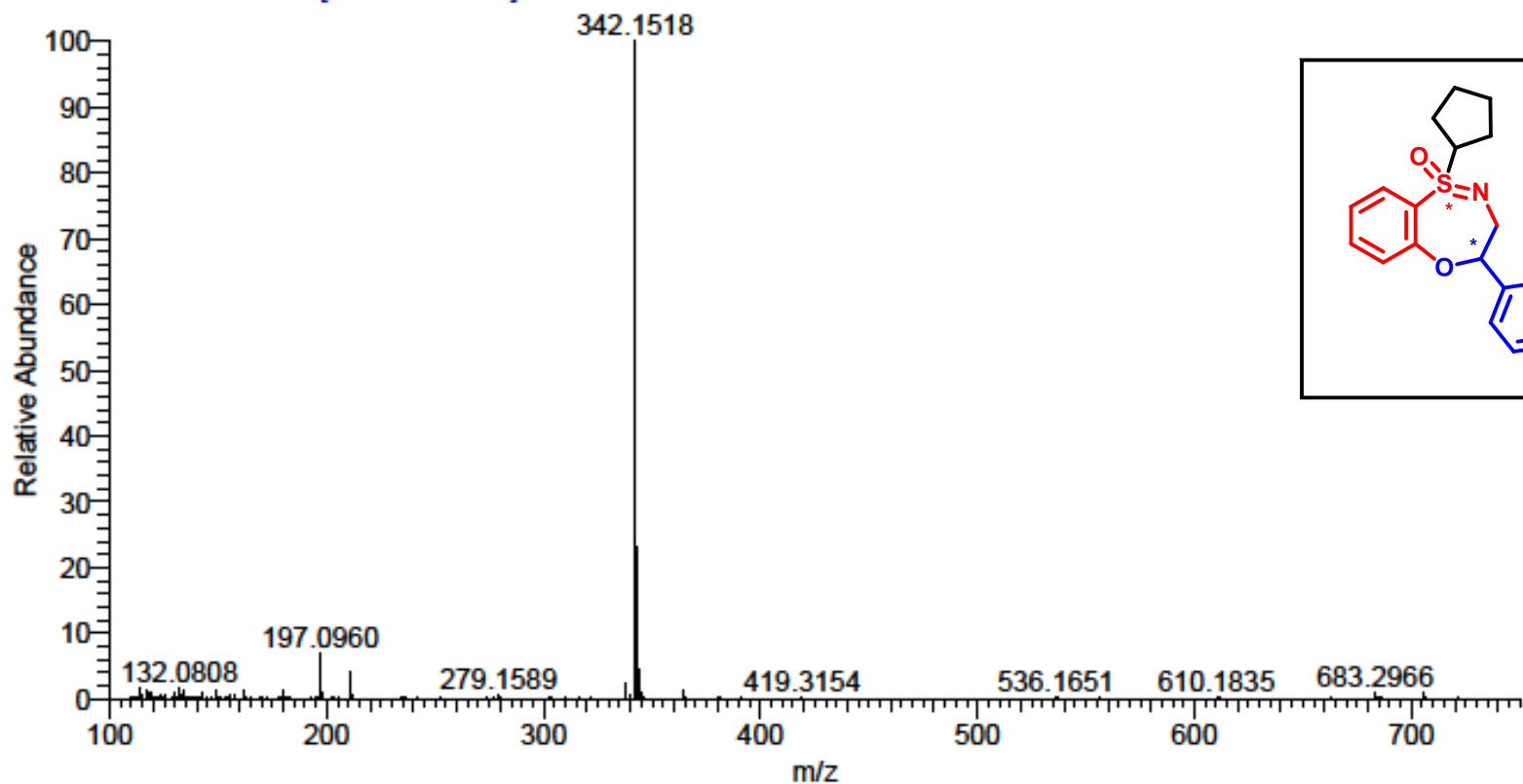


Fig S-78: HRMS report of Compound 4j

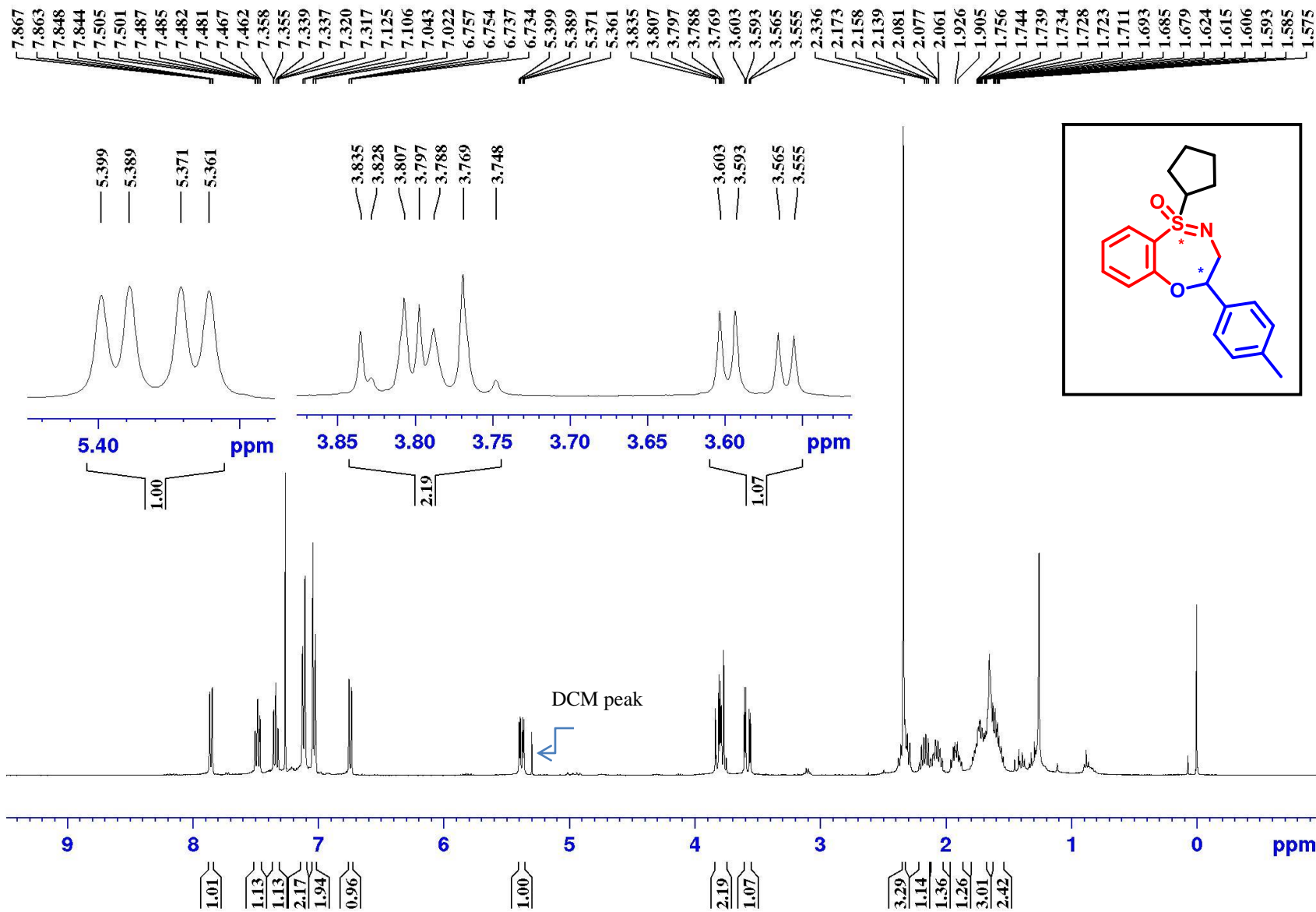


Fig S-79: ¹H NMR Spectra of Compound 4j' (400 MHz, CDCl₃)

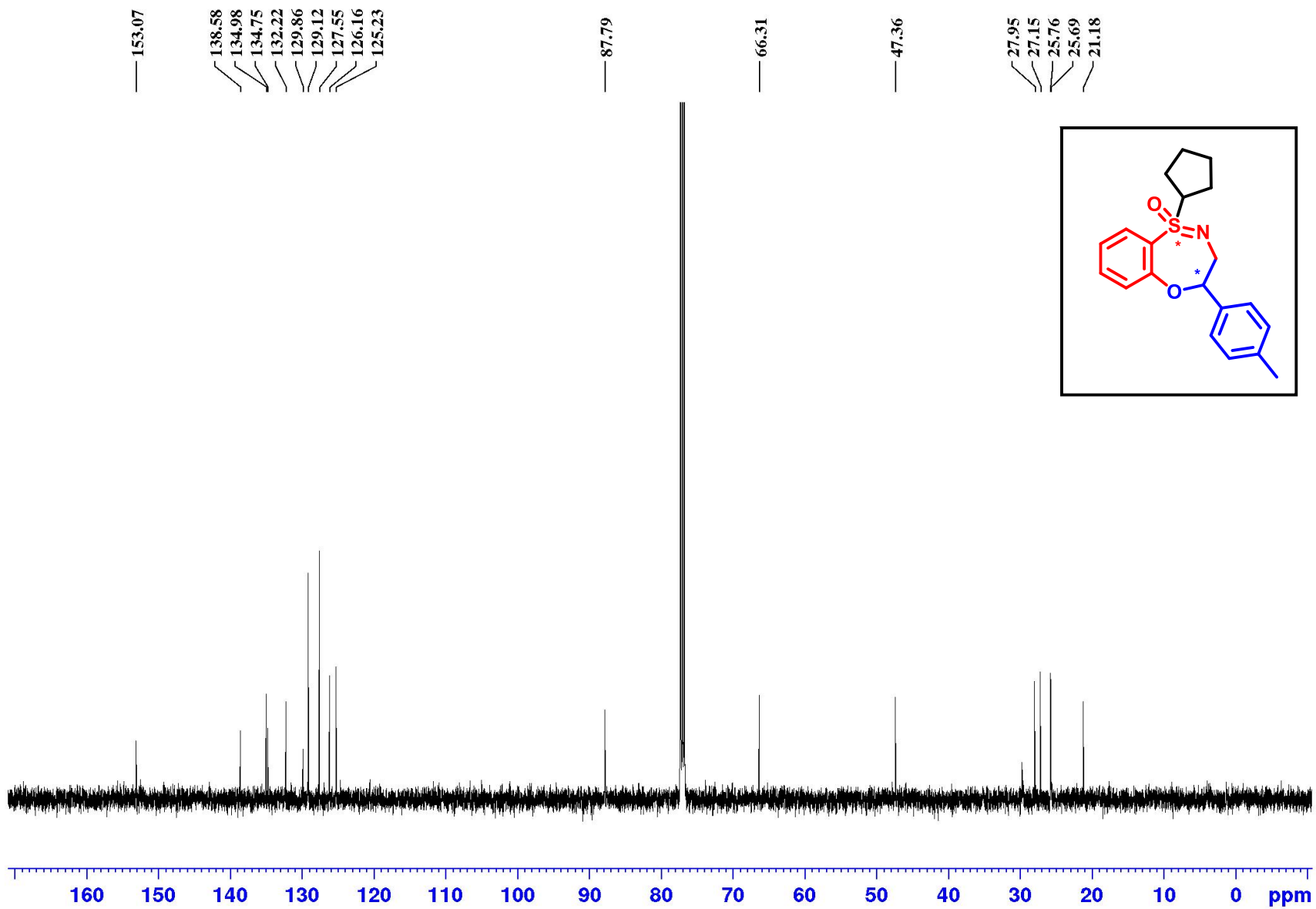


Fig S-80: ^{13}C NMR Spectra of Compound **4j'** (100 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I17MAR14	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	AB-167B	Sample Name:	
Acquisition Date:	03/17/21 11:30:06 AM	Run Time(min):	0.00
Vial:	CStk1-01:14	Injection Volume(μl):	1.00

HRMS21I17MAR14 #32-65 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 4.71E6
T: FTMS + c ESI Full ms [100.00-750.00]

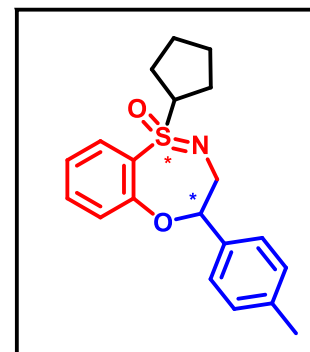
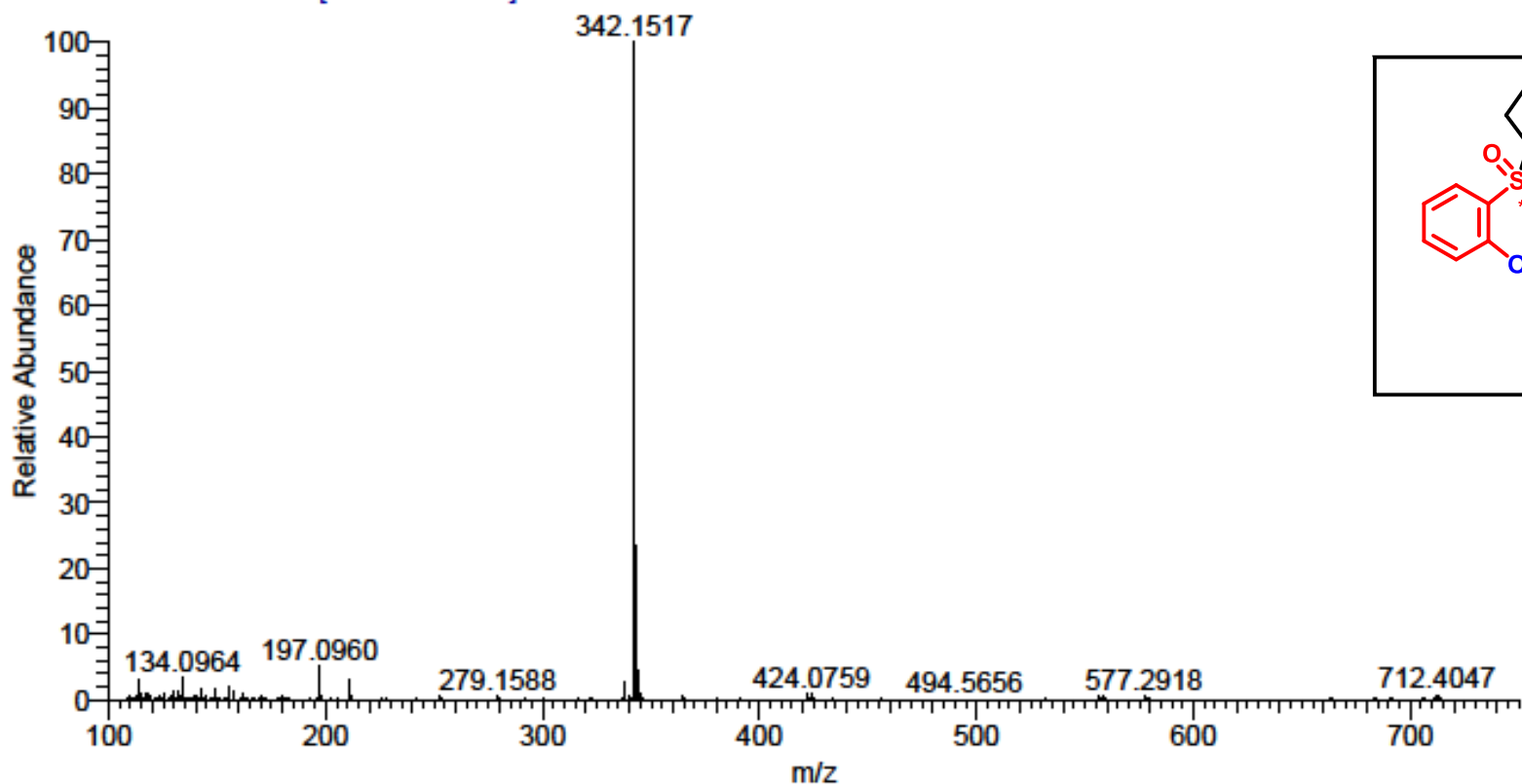


Fig S-81: HRMS report of Compound 4j'

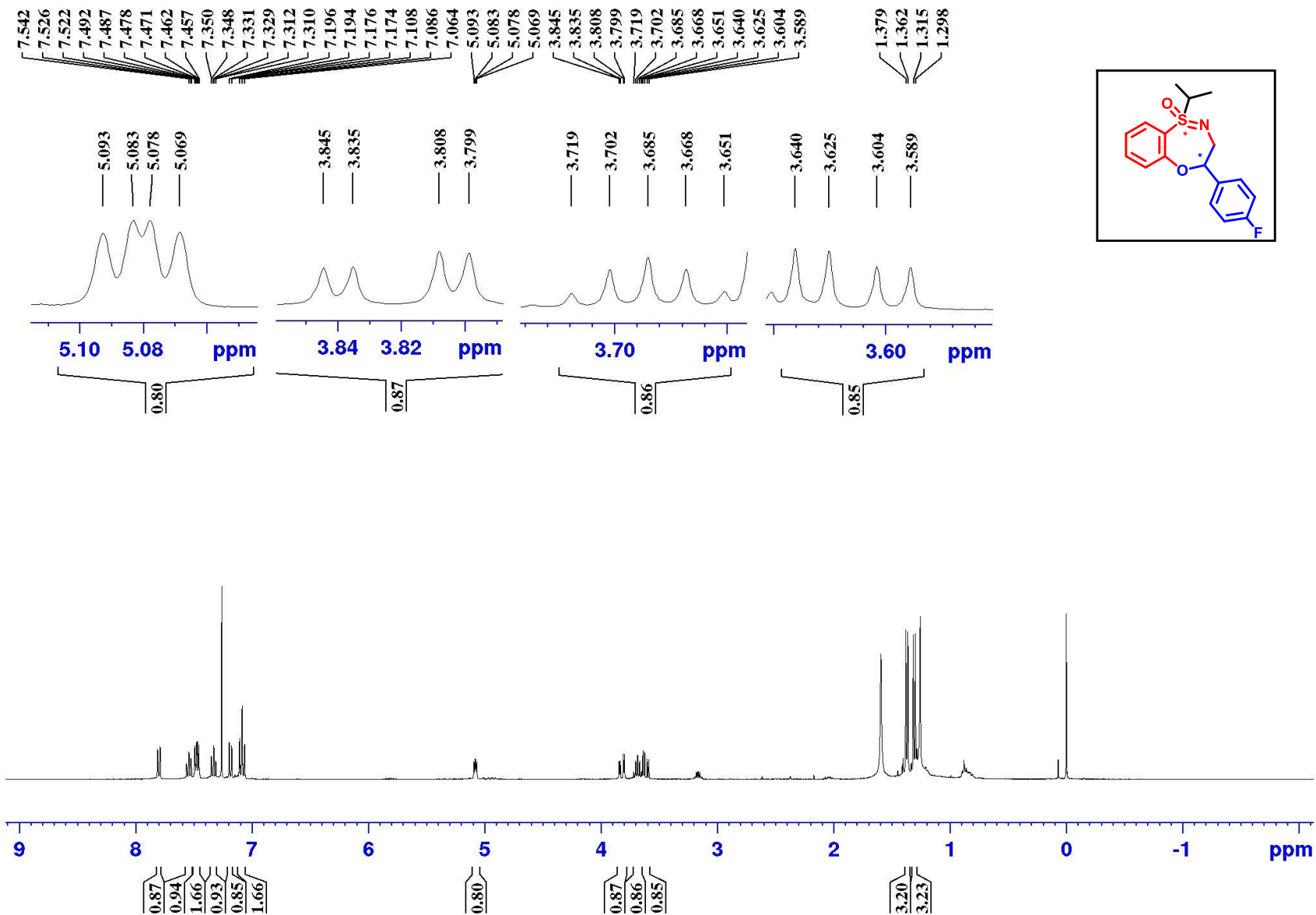


Fig S-82: ^1H NMR Spectra of Compound **4k** (400 MHz, CDCl_3)

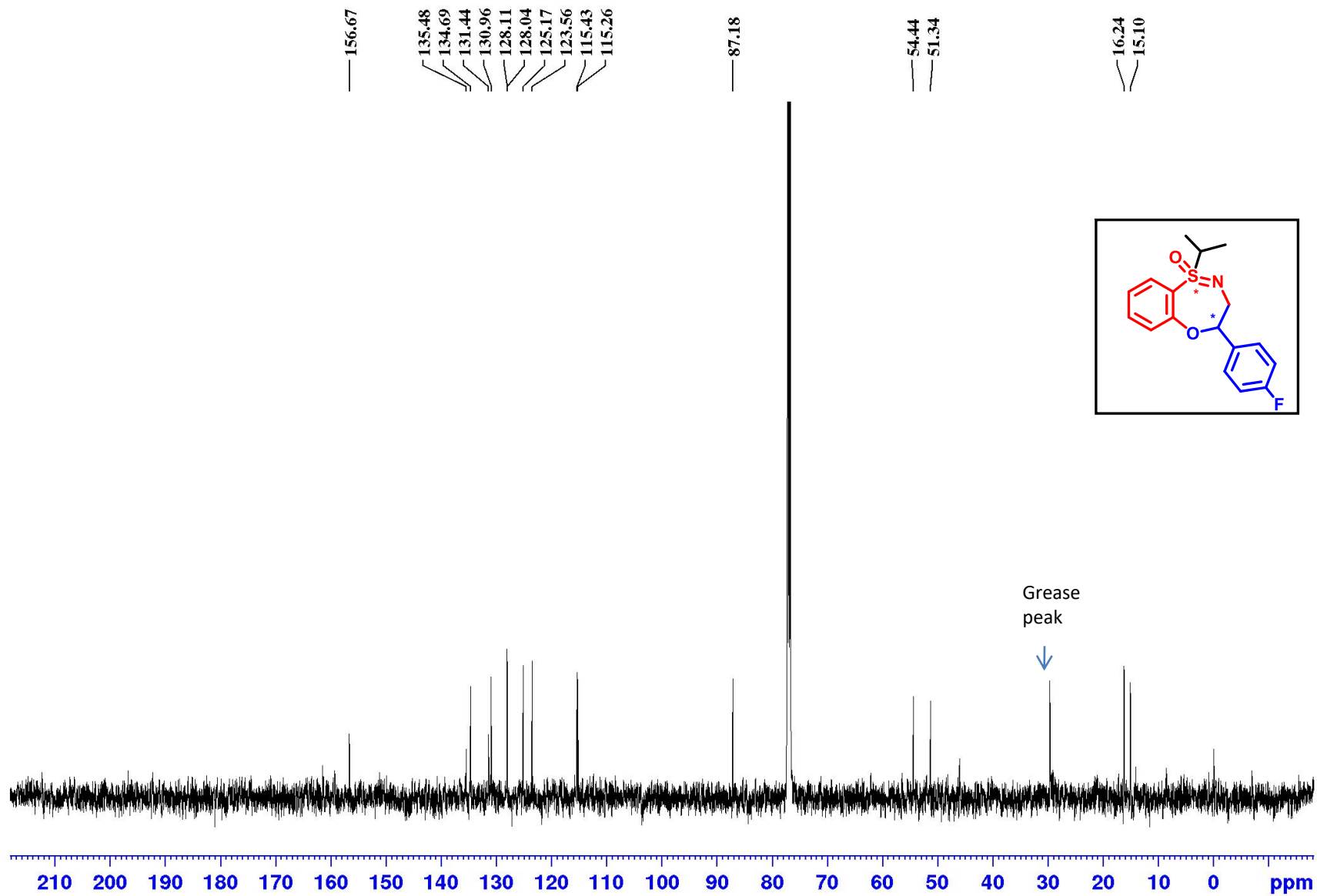


Fig S-83: ^{13}C NMR Spectra of Compound **4k** (125 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I05APR01	Original Data Path:	D:\INTERNAL NEW\2021\April 2021
Sample ID:	AB-169A	Sample Name:	
Acquisition Date:	04/05/21 11:37:20 AM	Run Time(min):	0.00
Vial:	CStk1-01:01	Injection Volume(ul):	1.00

HRMS21I05APR01 #34-68 RT: 0.25-0.50 AV: 35 SB: 1 0.01 NL: 3.69E6
T: FTMS + c ESI Full ms [100.00-750.00]

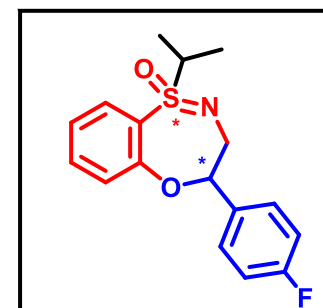
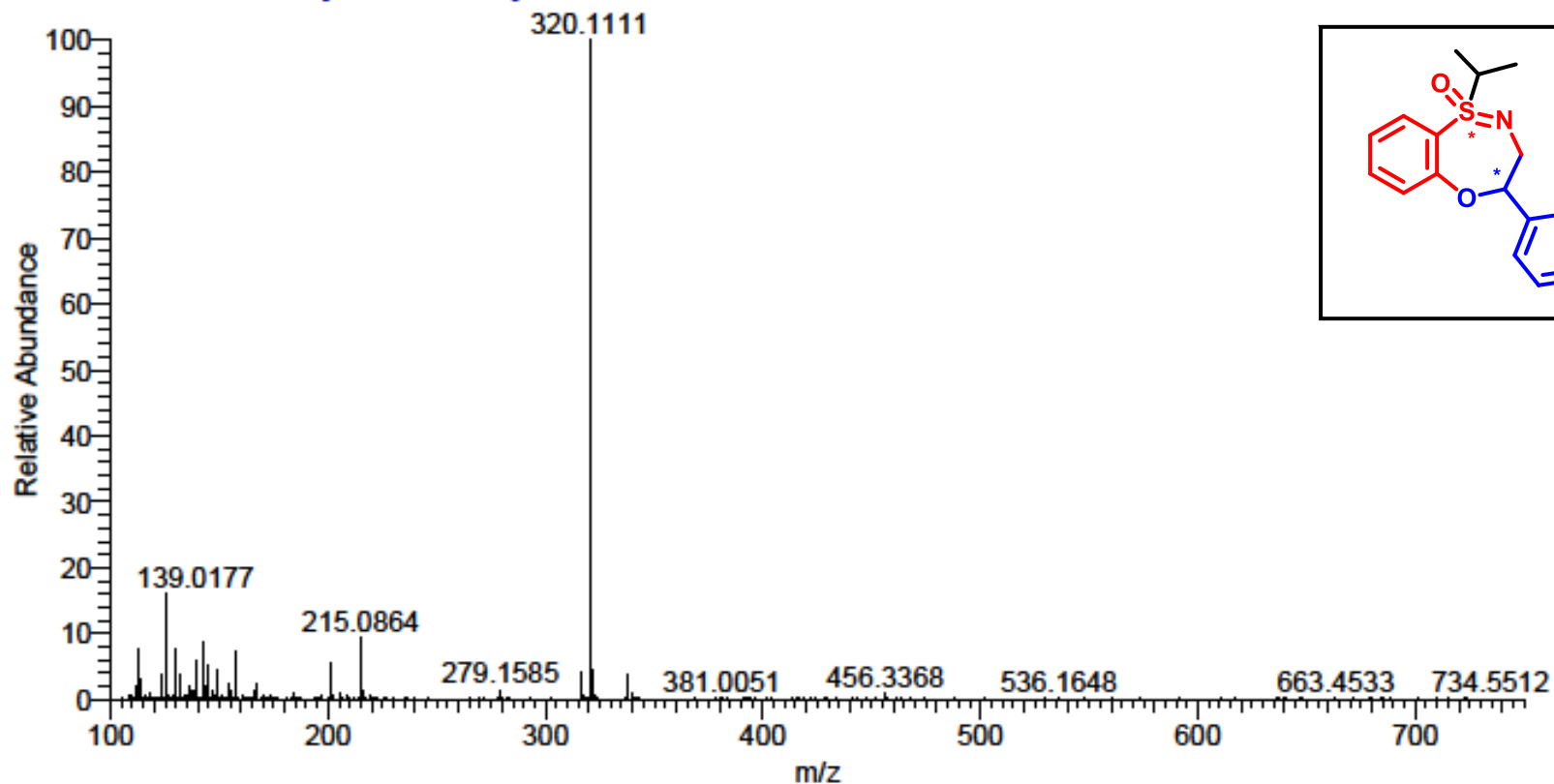


Fig S-84: HRMS report of Compound 4k

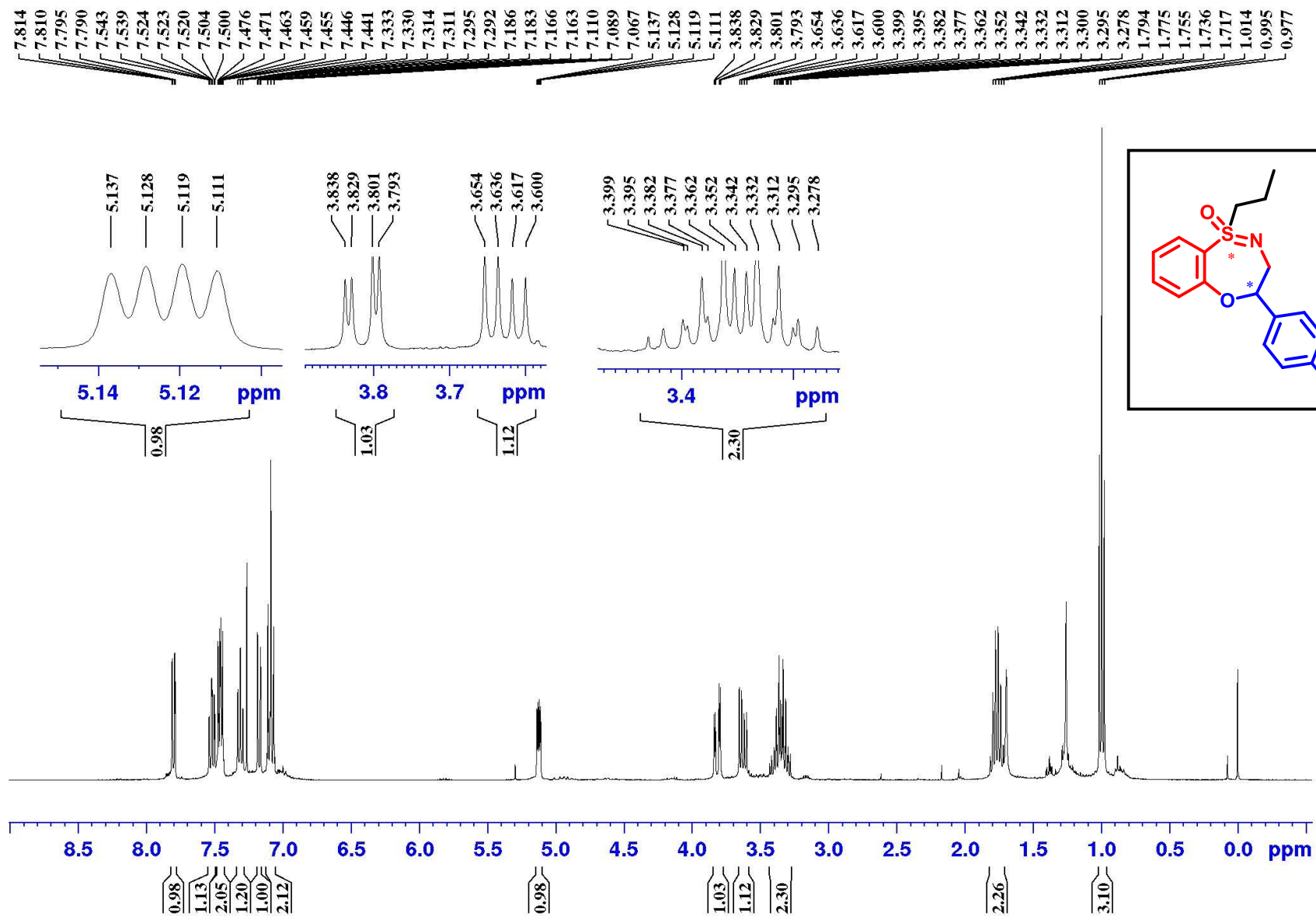


Fig S-85: ^1H NMR Spectra of Compound **41** (400 MHz, CDCl_3)

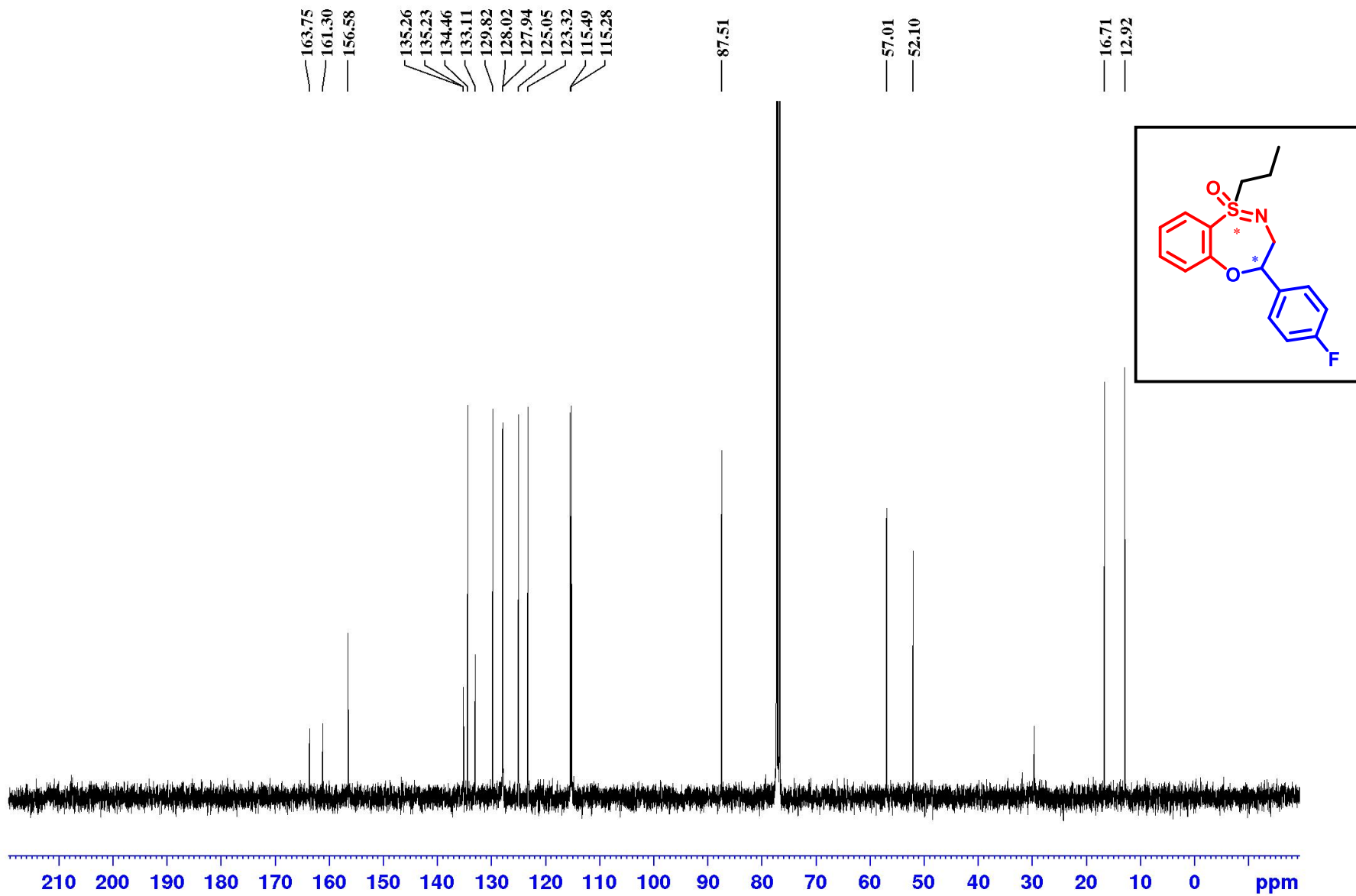


Fig S-86: ^{13}C NMR Spectra of Compound **41** (100 MHz, CDCl_3)

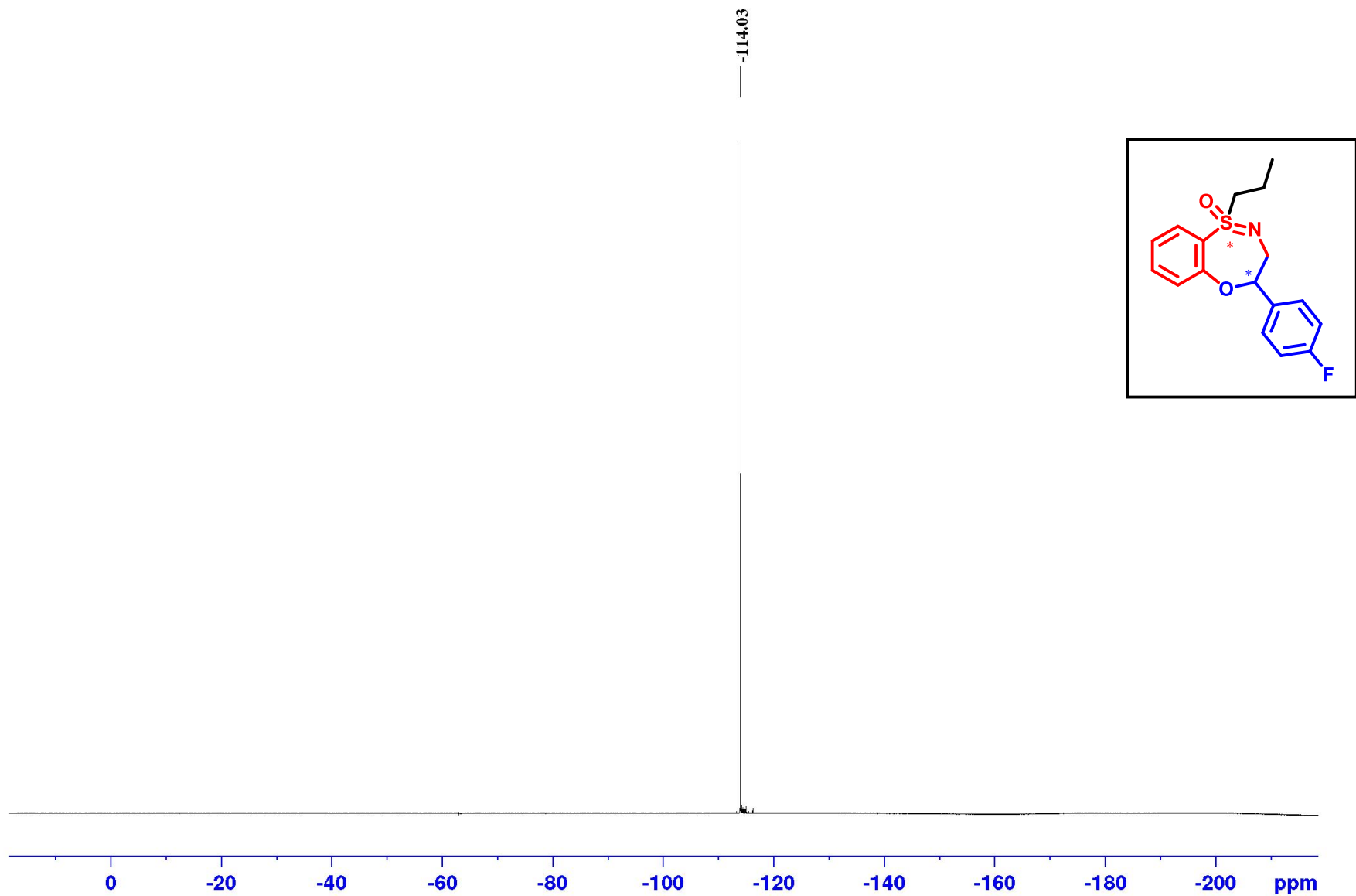


Fig S-87: ^{19}F NMR Spectra of Compound 41 (376 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I30MAR04	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	AB-171A	Sample Name:	
Acquisition Date:	03/30/21 11:58:20 AM	Run Time(min):	0.00
Vial:	CStk1-01:4	Injection Volume(μl):	1.00

HRMS21I30MAR04 #33-66 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 7.38E6
T: FTMS + c ESI Full ms [100.00-750.00]

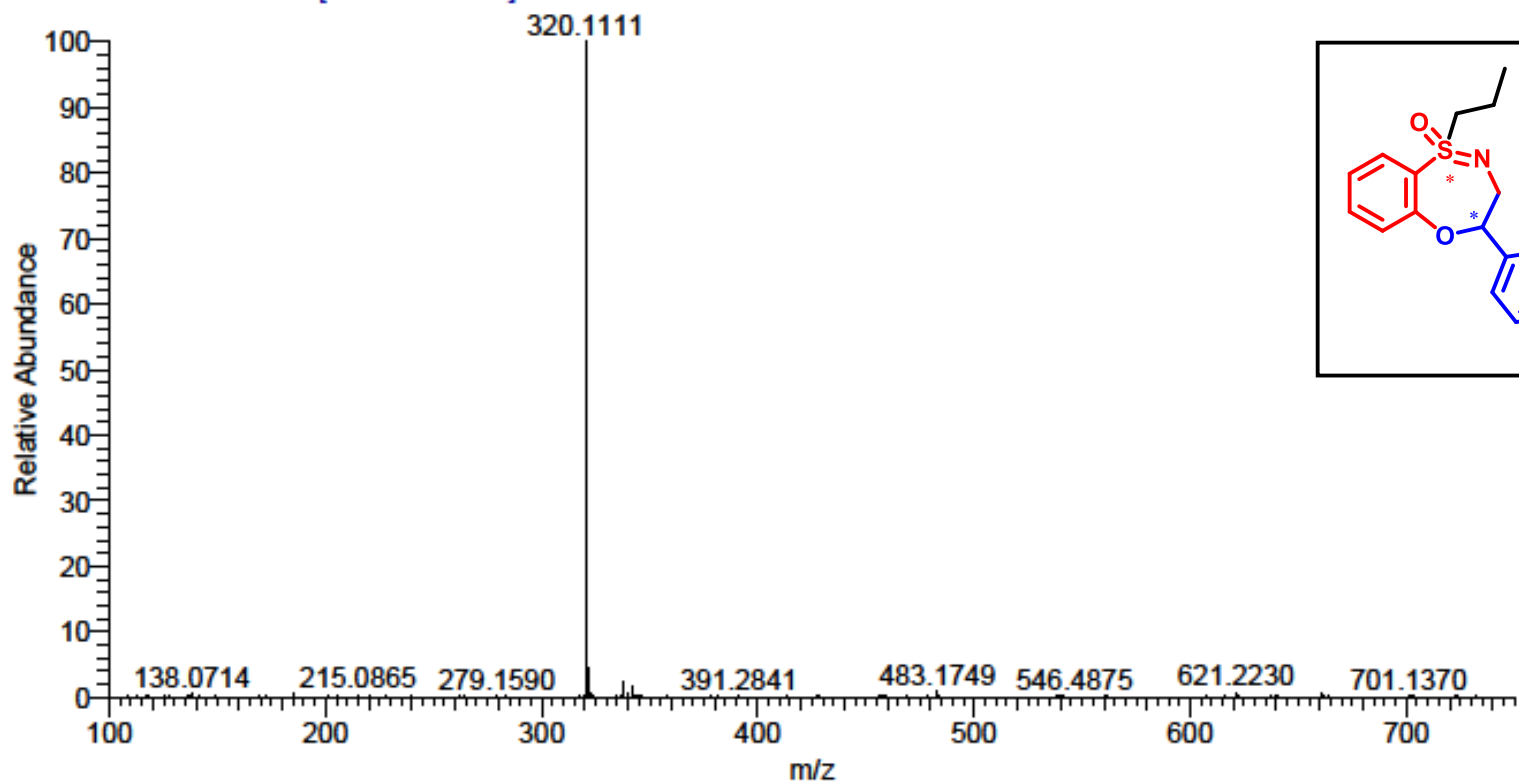


Fig S-88: HRMS report of Compound 4l

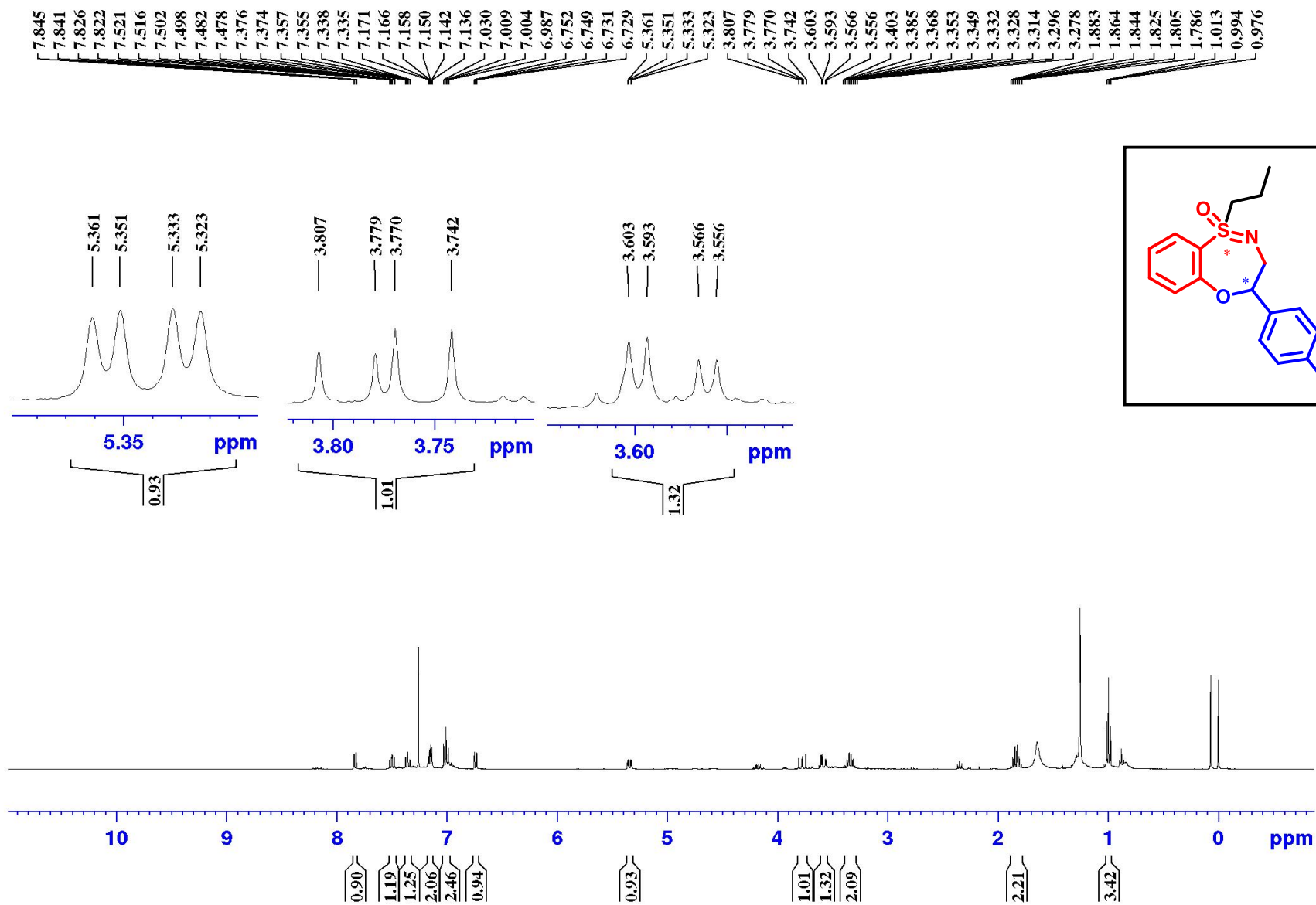


Fig S-89: ¹H NMR Spectra of Compound 4I' (400 MHz, CDCl₃)

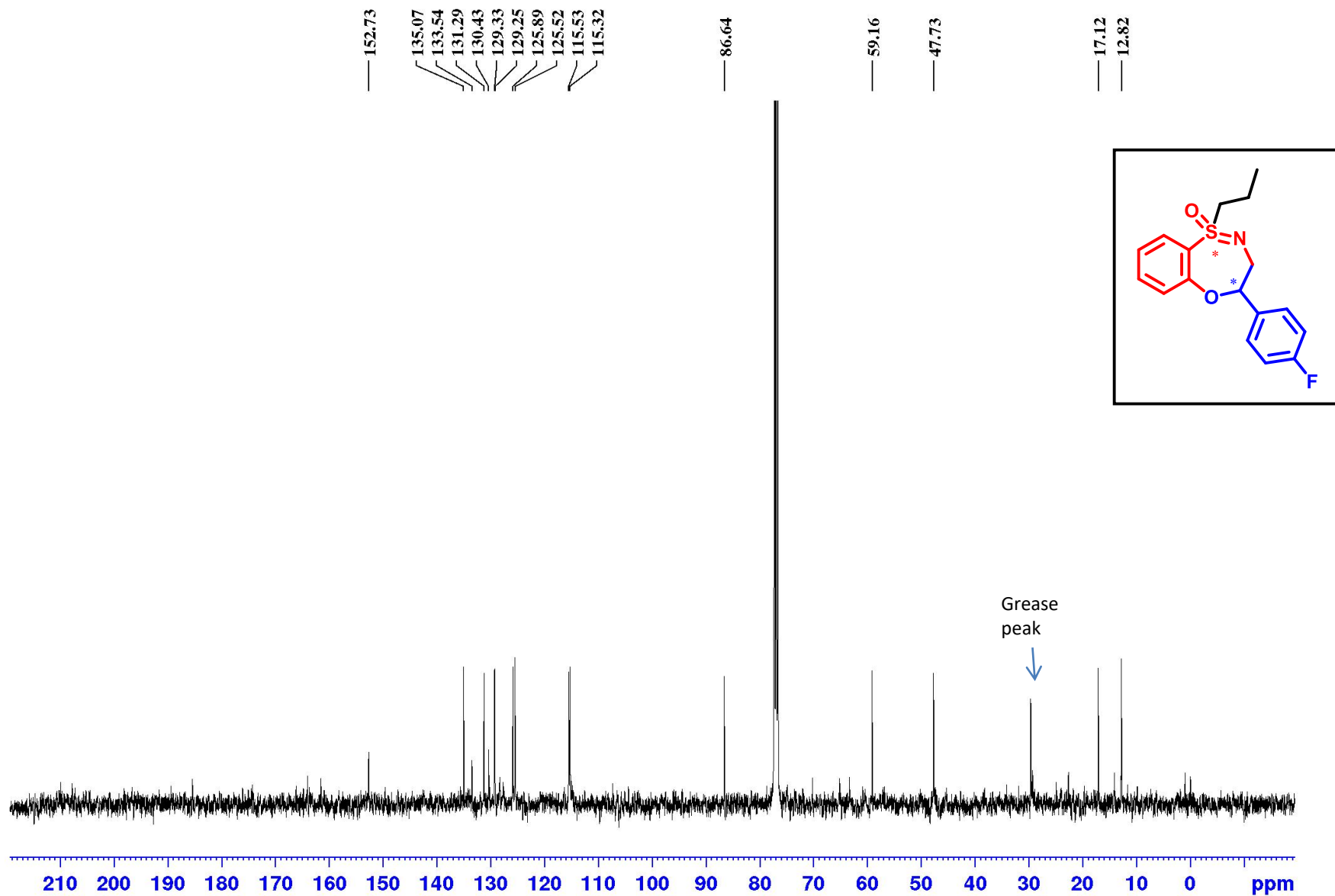


Fig S-90: ^{13}C NMR Spectra of Compound 4I' (100 MHz, CDCl_3)

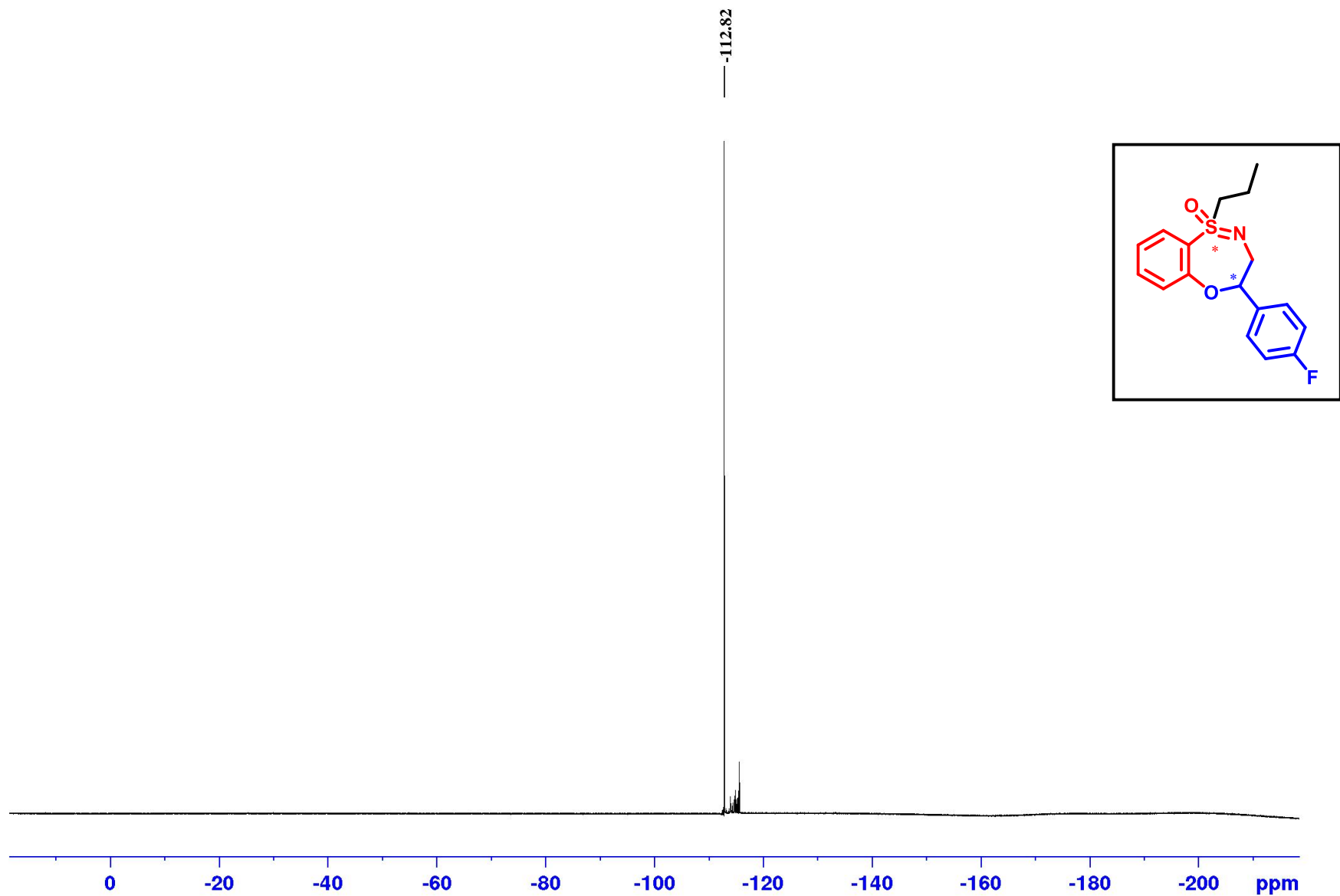


Fig S-91: ^{19}F NMR Spectra of Compound 4I' (376 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I30MAR05	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	AB-171B	Sample Name:	
Acquisition Date:	03/30/21 12:00:18 PM	Run Time(min):	0.00
Vial:	CStk1-01:5	Injection Volume(μl):	1.00

HRMS21I30MAR05 #33-66 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 4.51E6
T: FTMS + c ESI Full ms [100.00-750.00]

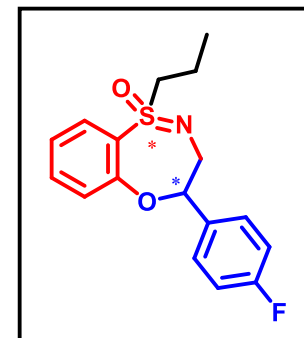
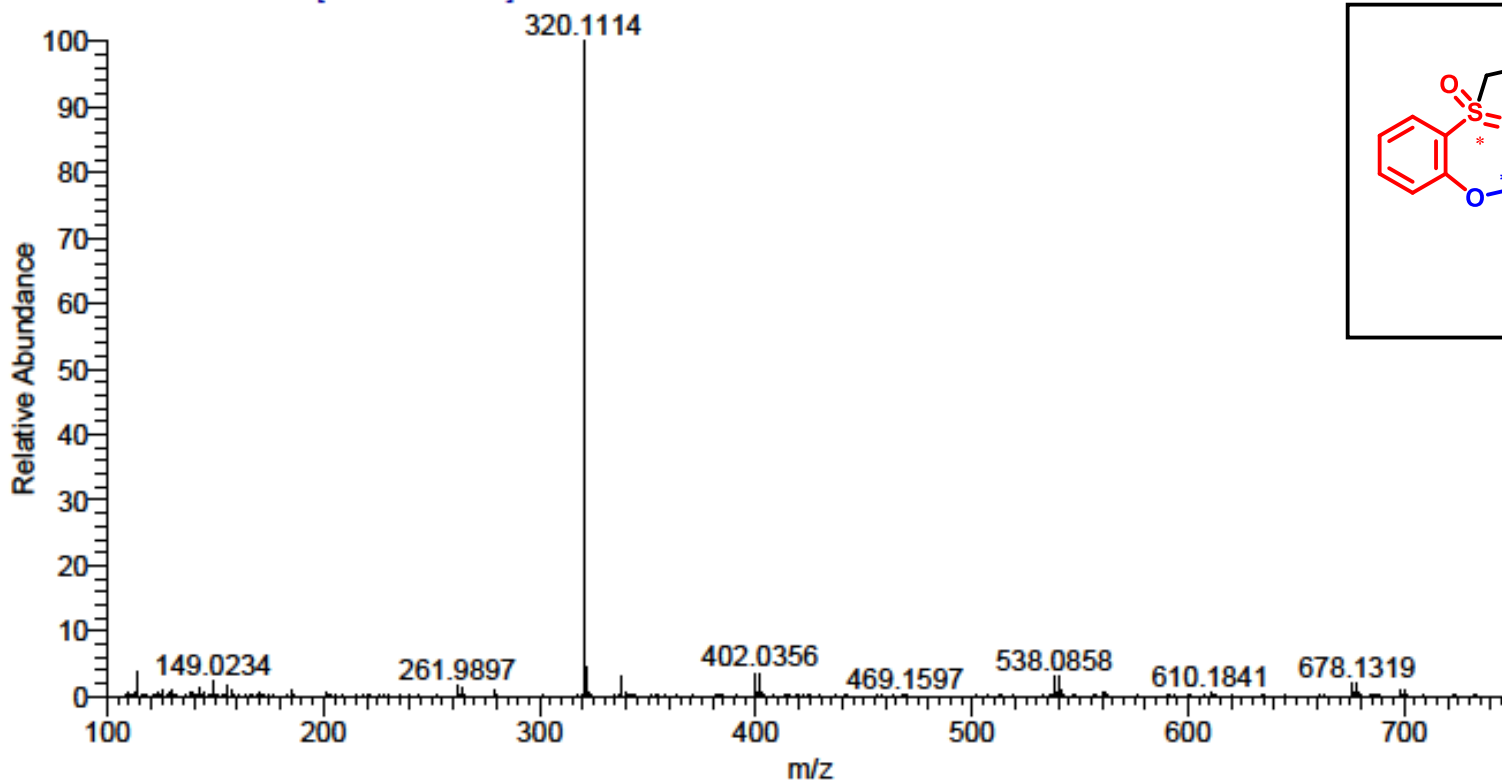


Fig S-92: HRMS report of Compound 4I'

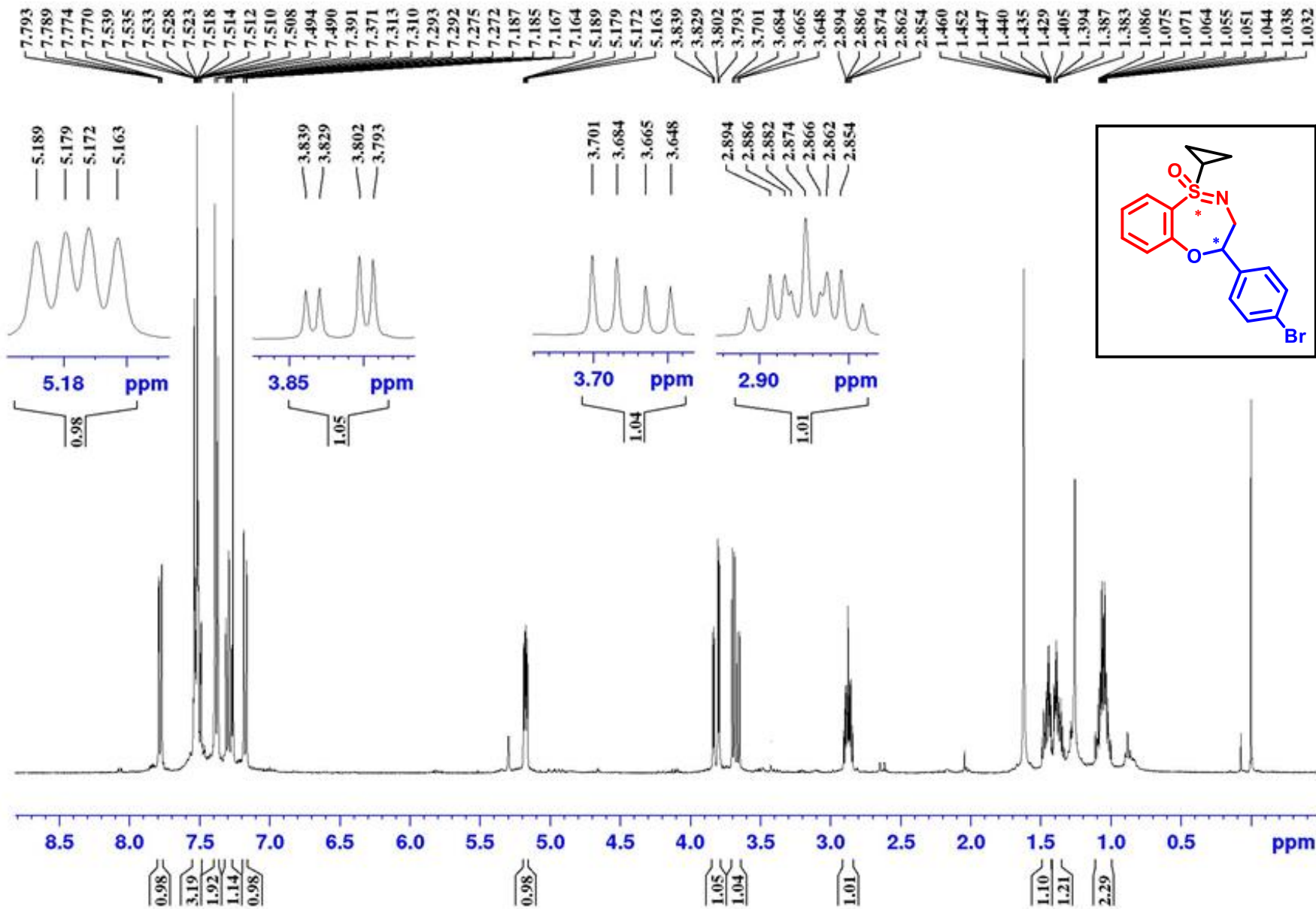


Fig S-93: ^1H NMR Spectra of Compound **4m** (400 MHz, CDCl_3)

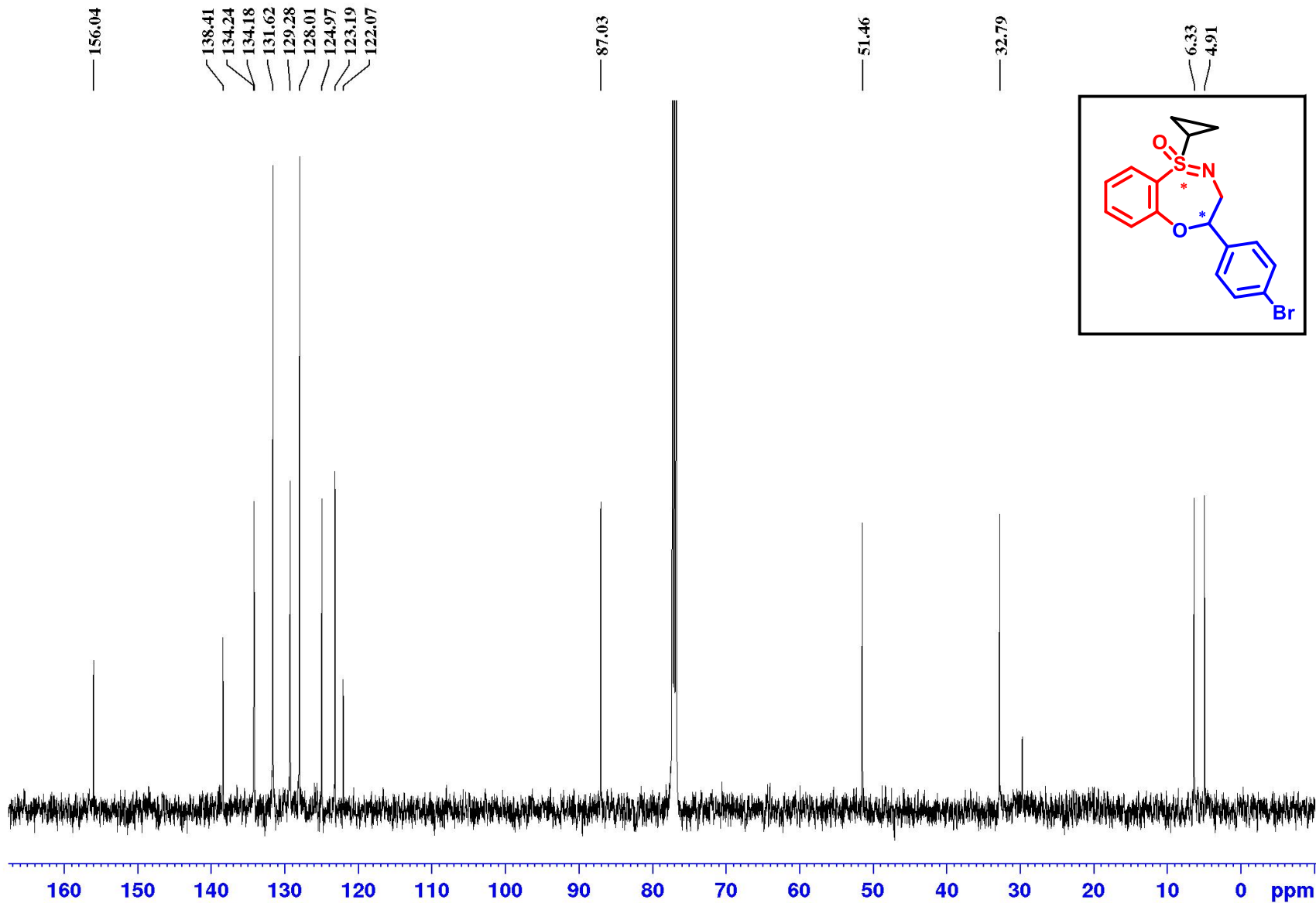


Fig S-94: ^{13}C NMR Spectra of Compound 4m (100 MHz, CDCl_3)

HRMS21105APR02 #35 RT: 0.25 AV: 1 SB: 8 0.02-0.08 NL: 3.50E7

T: FTMS + c ESI Full ms [100.00-750.00]

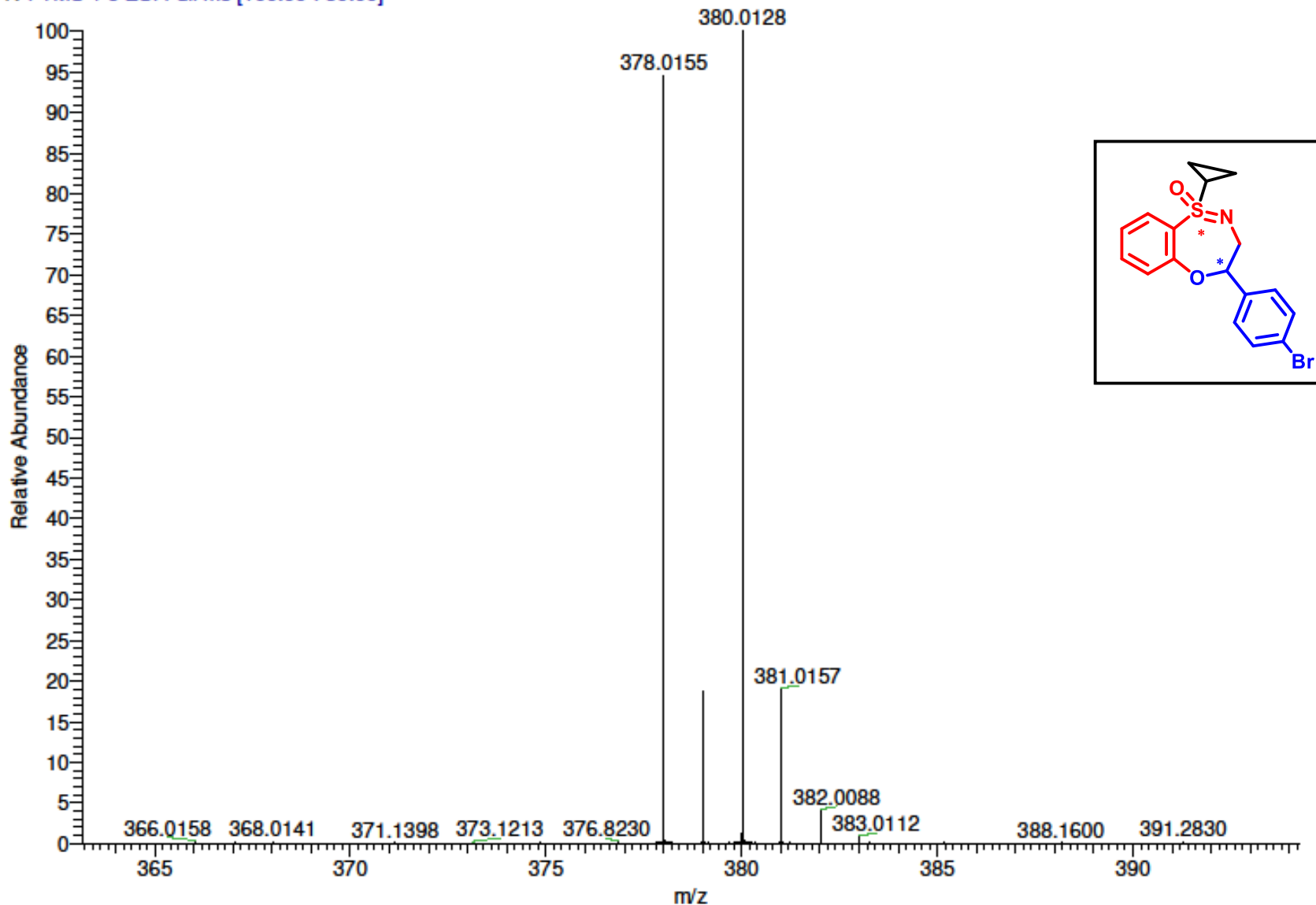


Fig S-95: HRMS report of Compound 4m

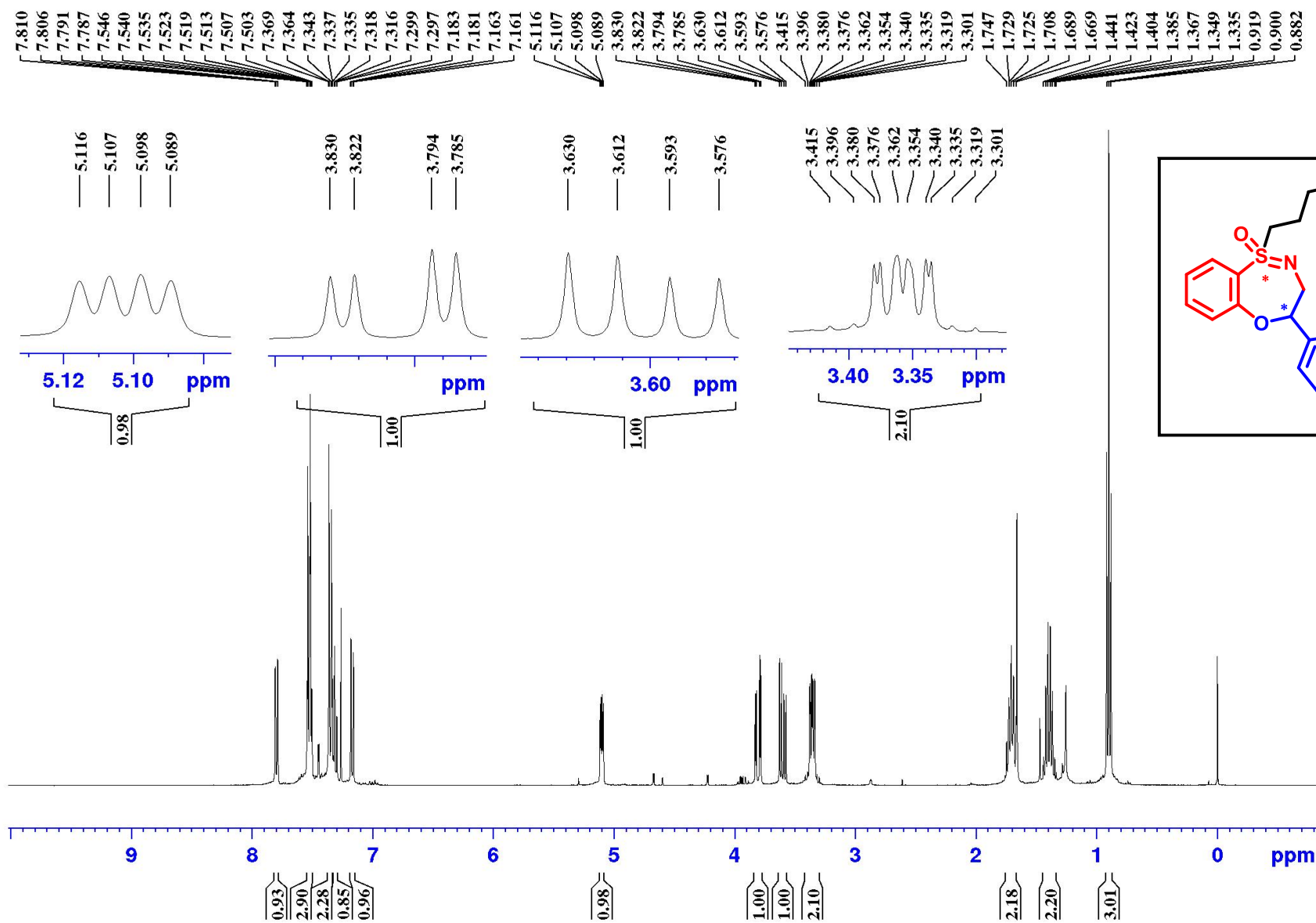


Fig S-96: ^1H NMR Spectra of Compound 4n (400 MHz, CDCl_3)

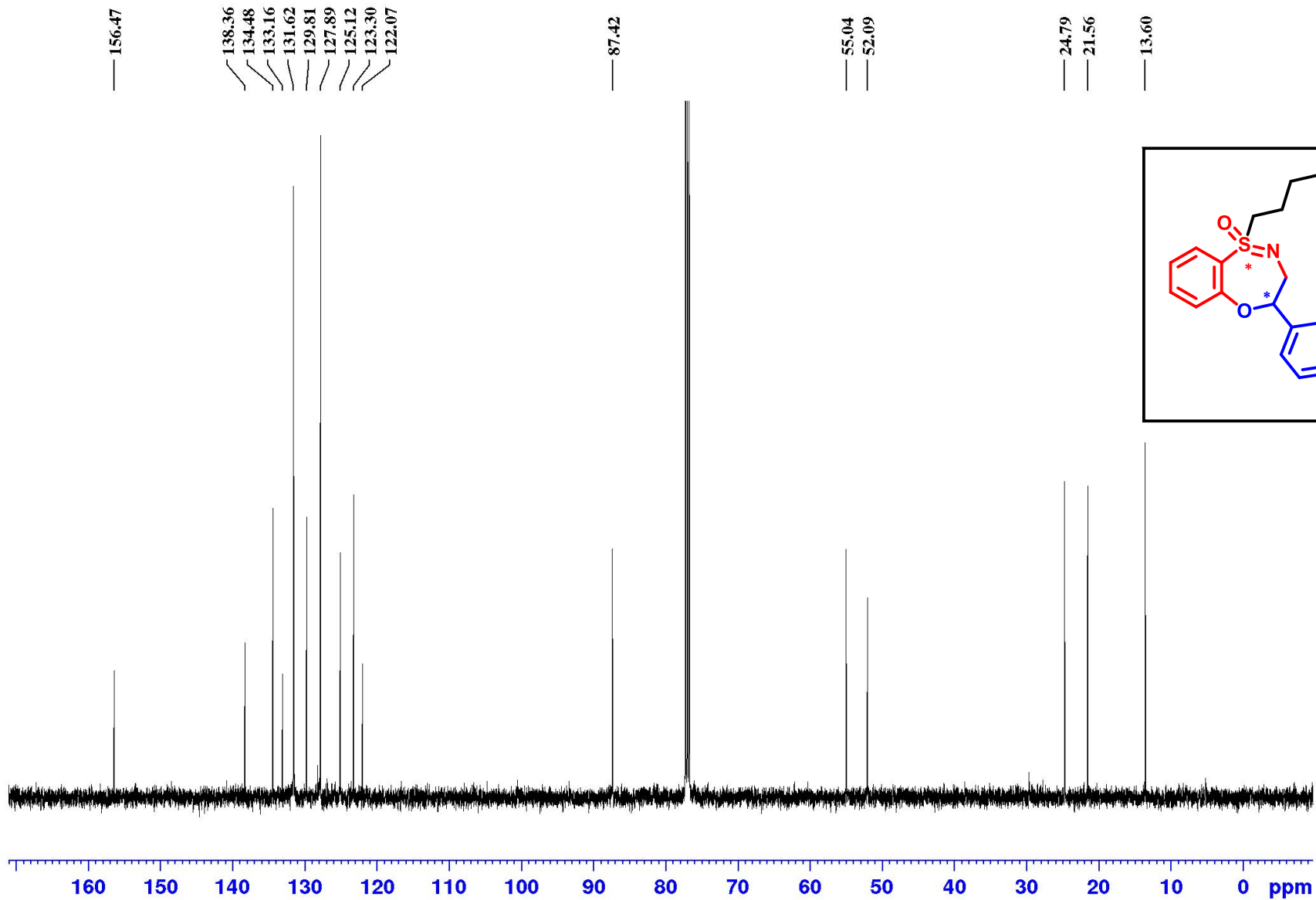


Fig S-97: ^{13}C NMR Spectra of Compound **4n** (125 MHz, CDCl_3)

HRMS21131MAR03 #38 RT: 0.29 AV: 1 SB: 8 0.02-0.08 NL: 7.59E6
T: FTMS + c ESI Full ms [100.00-750.00]

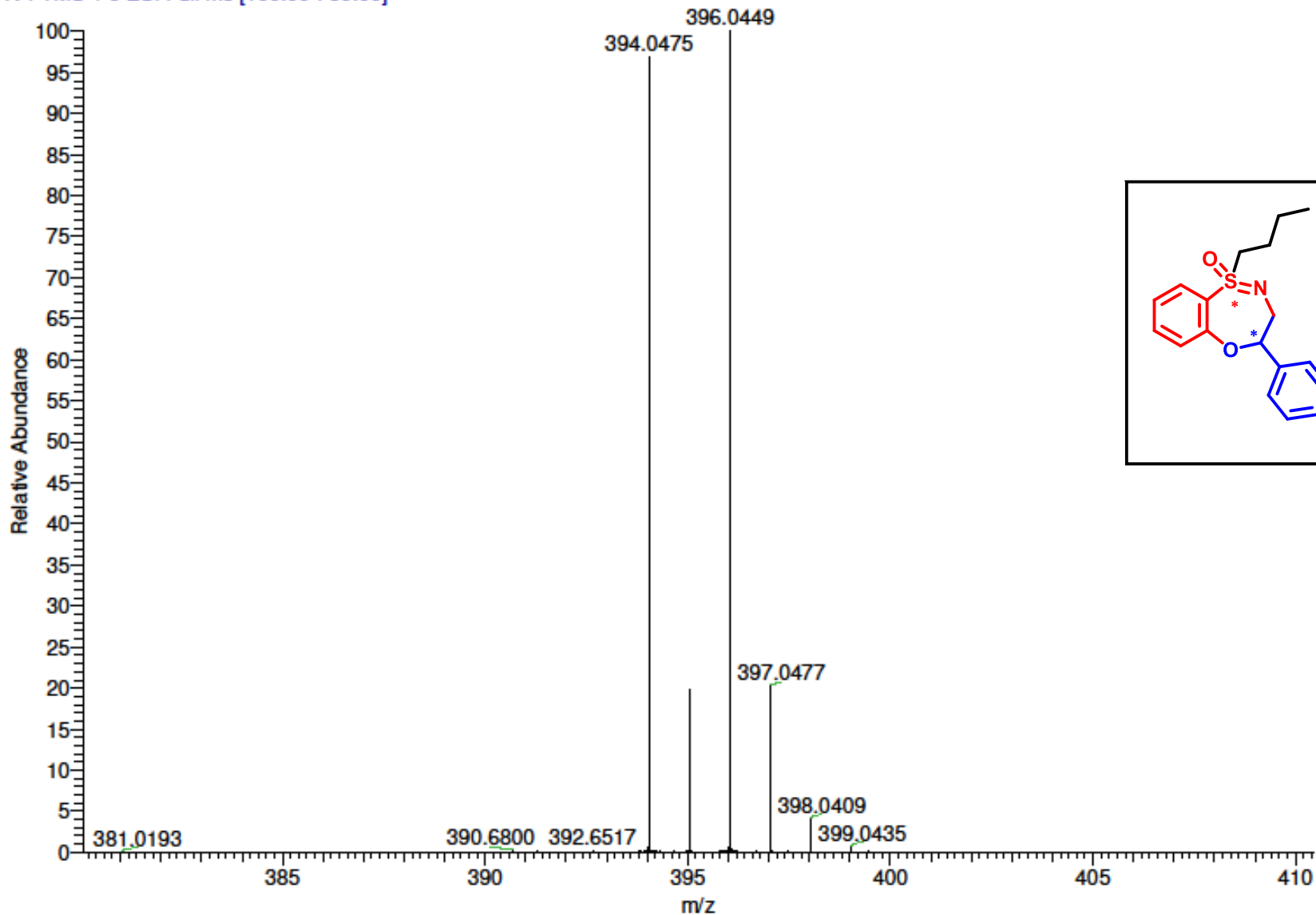


Fig S-98: HRMS report of Compound 4n

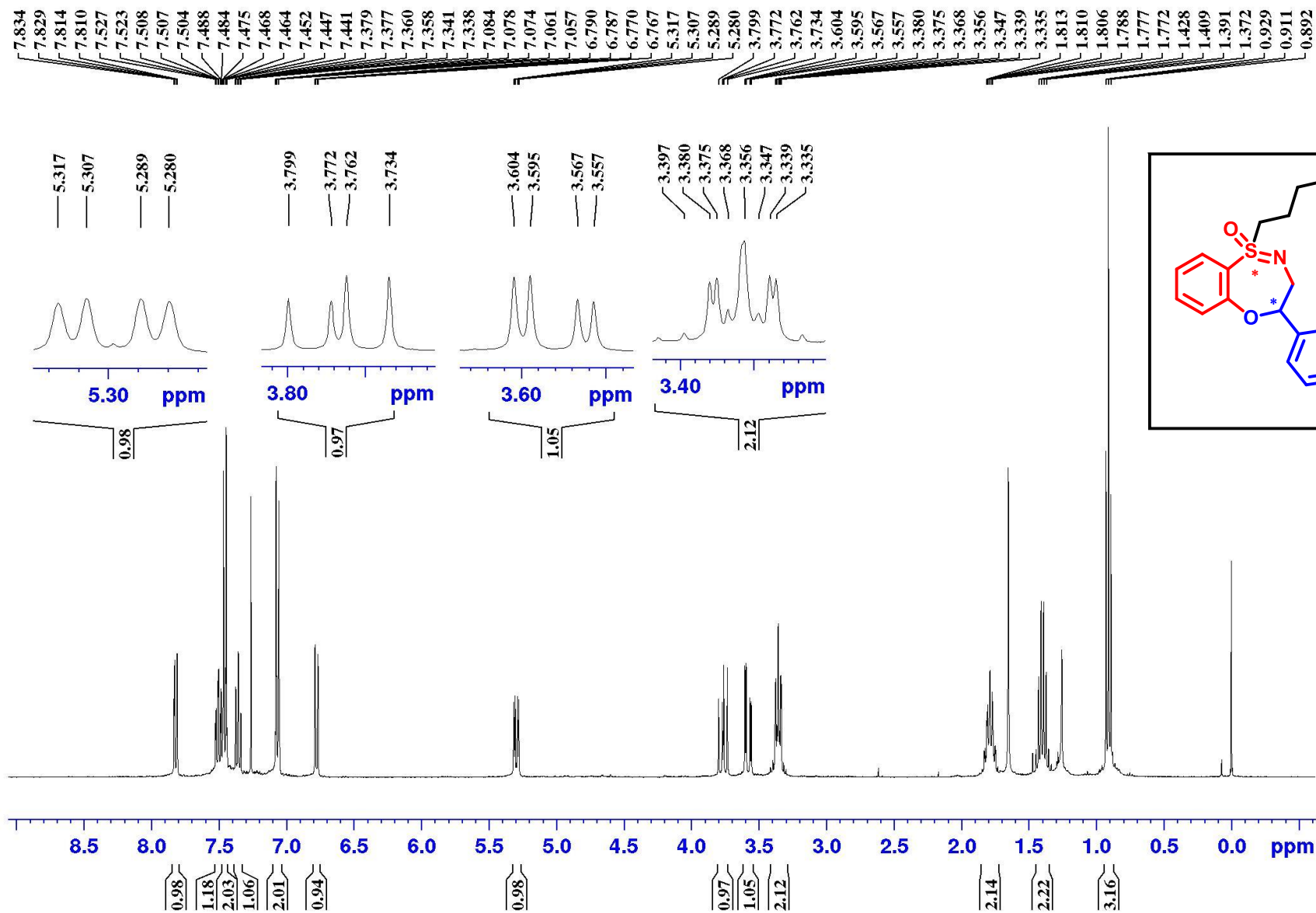


Fig S-99: ¹H NMR Spectra of Compound 4n' (400 MHz, CDCl₃)

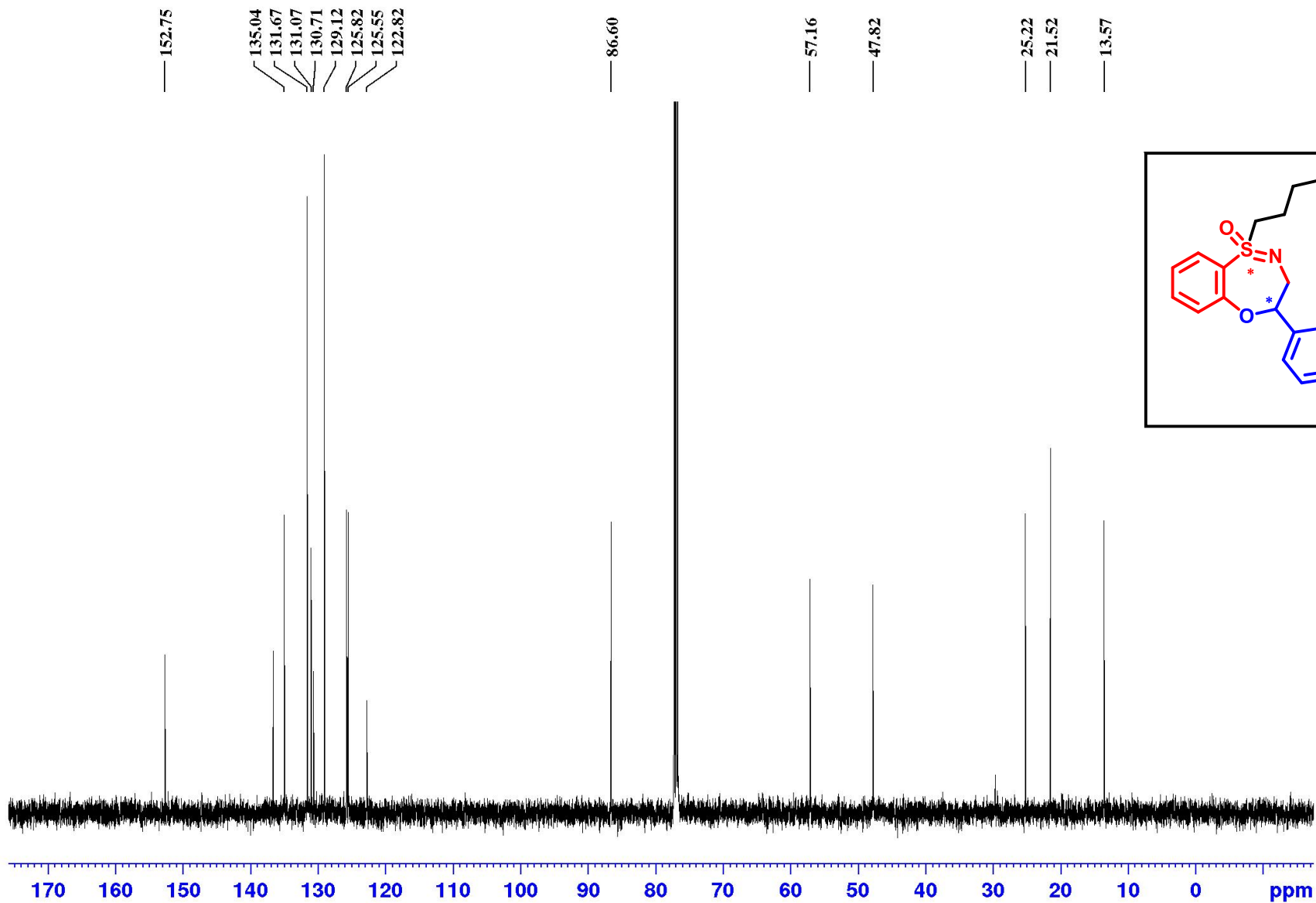


Fig S-100: ^{13}C NMR Spectra of Compound **4n'** (125 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I31MAR04	Original Data Path:	D:\INTERNAL NEW\2021\Mar 2021
Sample ID:	AB-173B	Sample Name:	
Acquisition Date:	03/31/21 11:37:53 AM	Run Time(min):	0.00
Vial:	CStk1-01:4	Injection Volume(μl):	1.00

HRMS21I31MAR04 #33-66 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 5.43E6
T: FTMS + c ESI Full ms [100.00-750.00]

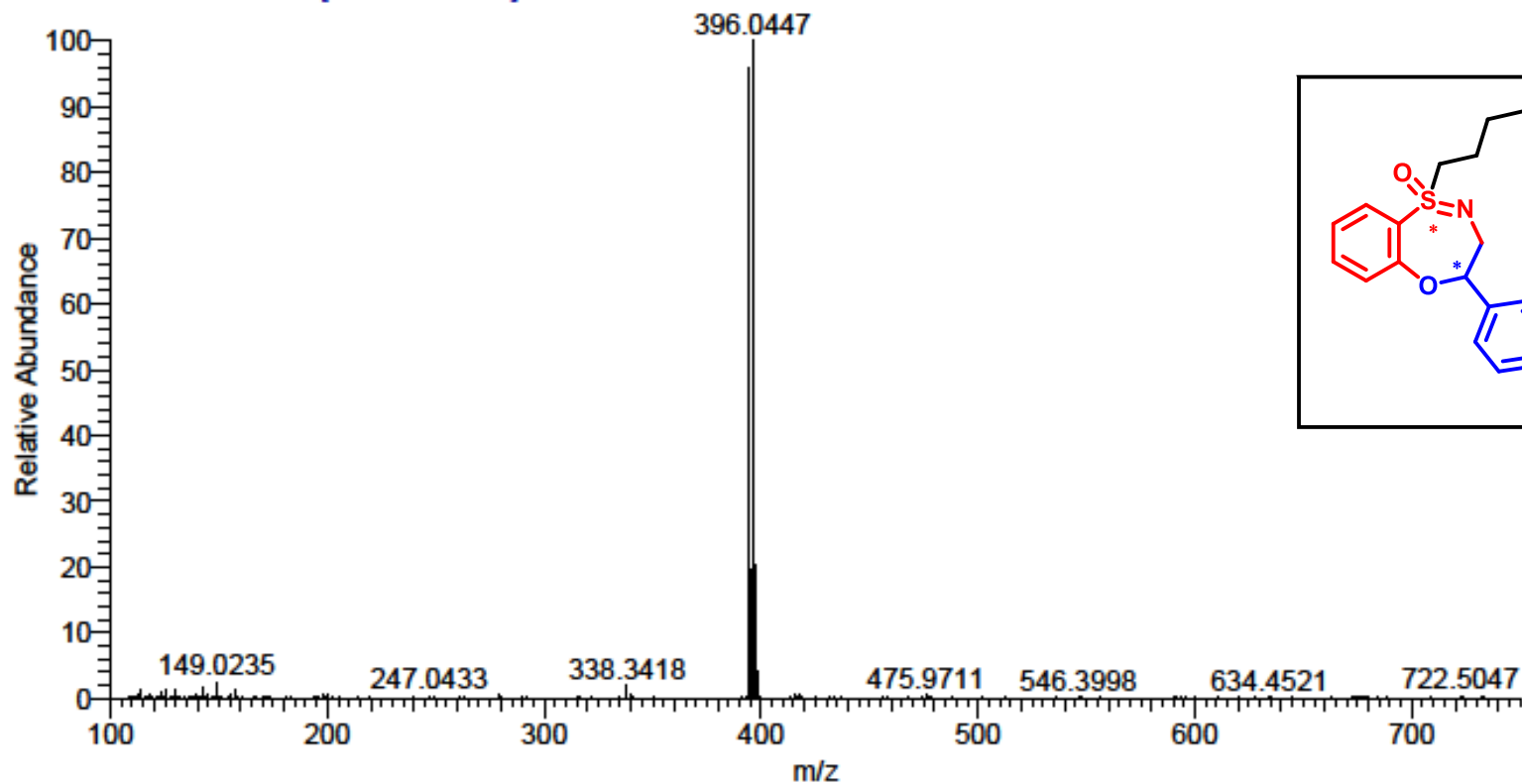


Fig S-101: HRMS report of Compound 4n'

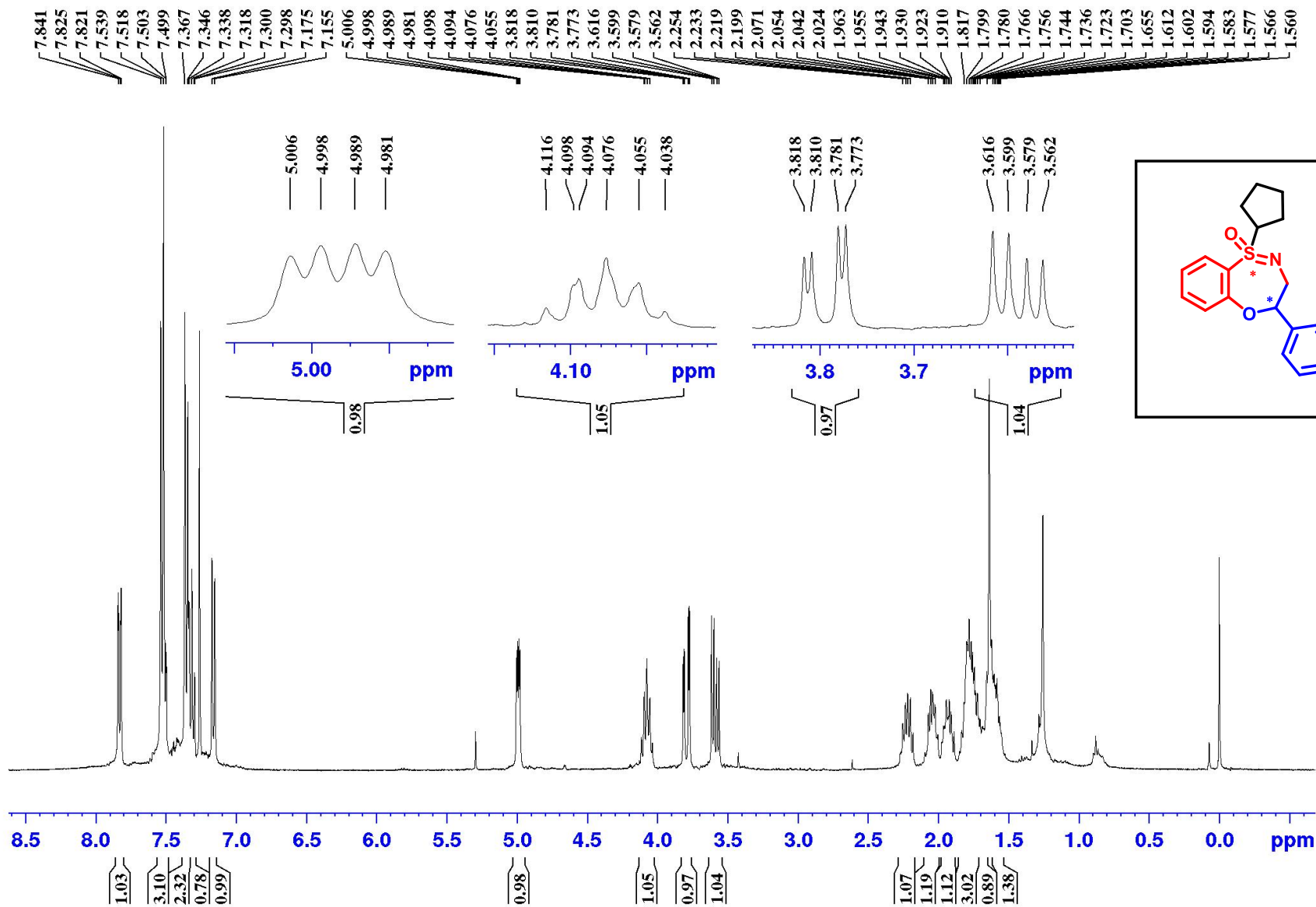


Fig S-102: ^1H NMR Spectra of Compound **4o** (400 MHz, CDCl_3)

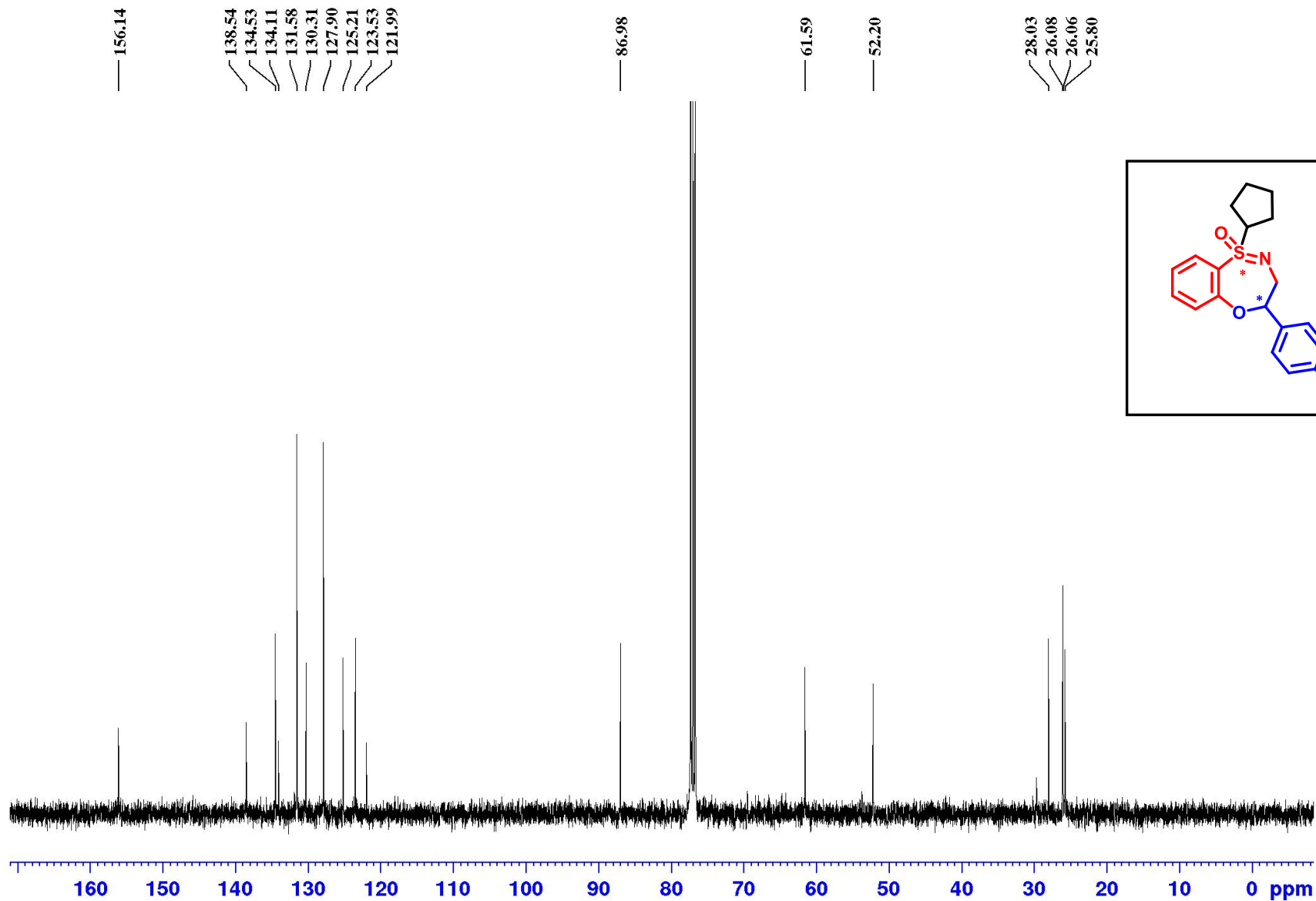


Fig S-103: ^{13}C NMR Spectra of Compound **4o** (100 MHz, CDCl_3)

HRMS21105APR03 #30-45 RT: 0.22-0.33 AV: 16 SB: 8 0.02-0.08 NL: 2.73E6
T: FTMS + c ESI Full ms [100.00-750.00]

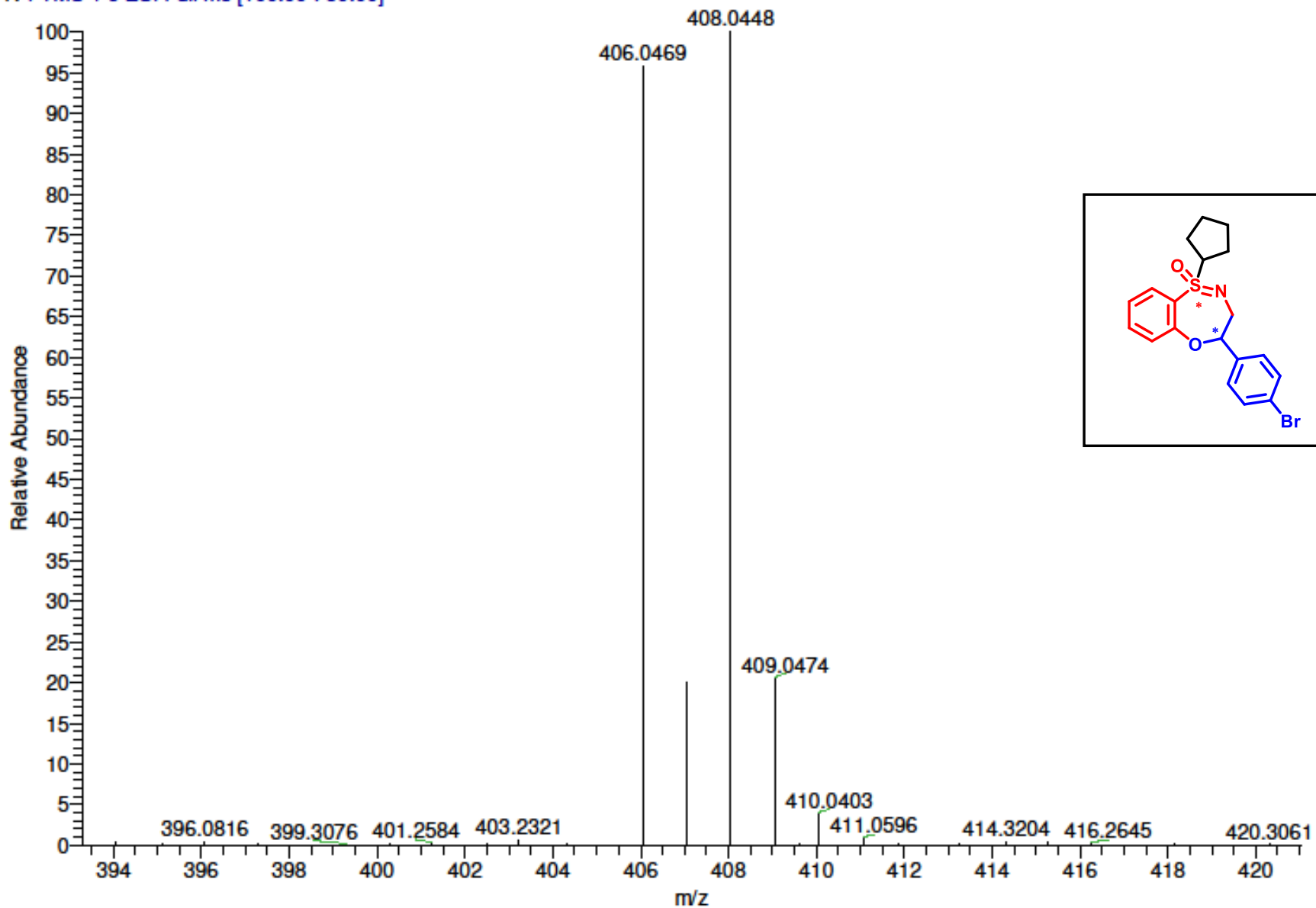


Fig S-104: HRMS report of Compound 4o

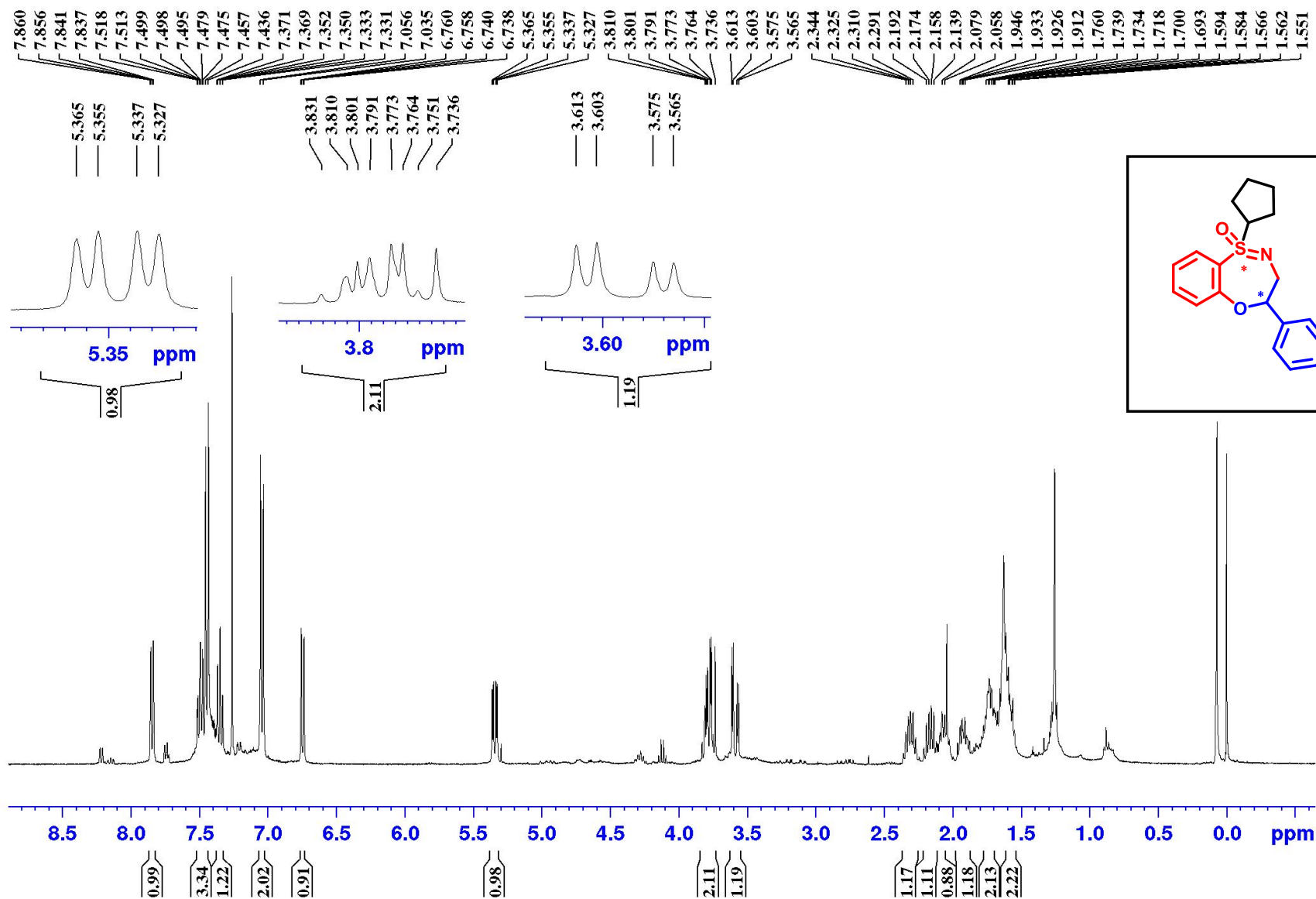


Fig S-105: ¹H NMR Spectra of Compound 4o' (400 MHz, CDCl₃)

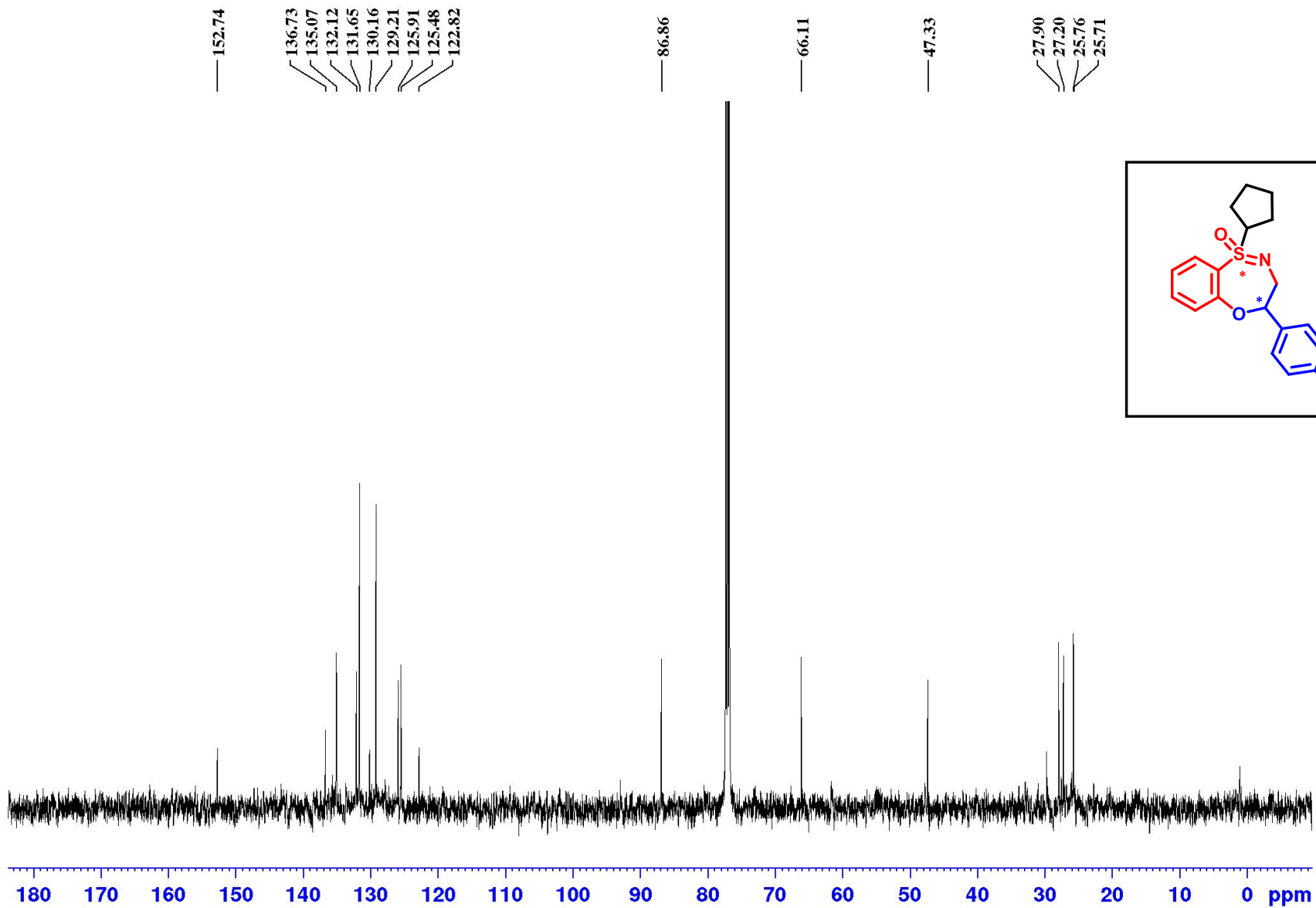


Fig S-106: ^{13}C NMR Spectra of Compound **4o'** (100 MHz, CDCl_3)

SAIF [HRMS Report]

Data File:	HRMS21I05APR04	Original Data Path:	D:\INTERNAL NEW\2021\April 2021
Sample ID:	AB-174B	Sample Name:	
Acquisition Date:	04/05/21 11:43:16 AM	Run Time(min):	0.00
Vial:	CSfk1-01:4	Injection Volume(μl):	1.00

HRMS21I05APR04 #34-67 RT: 0.25-0.50 AV: 34 SB: 1 0.01 NL: 3.37E6
T: FTMS + c ESI Full ms [100.00-750.00]

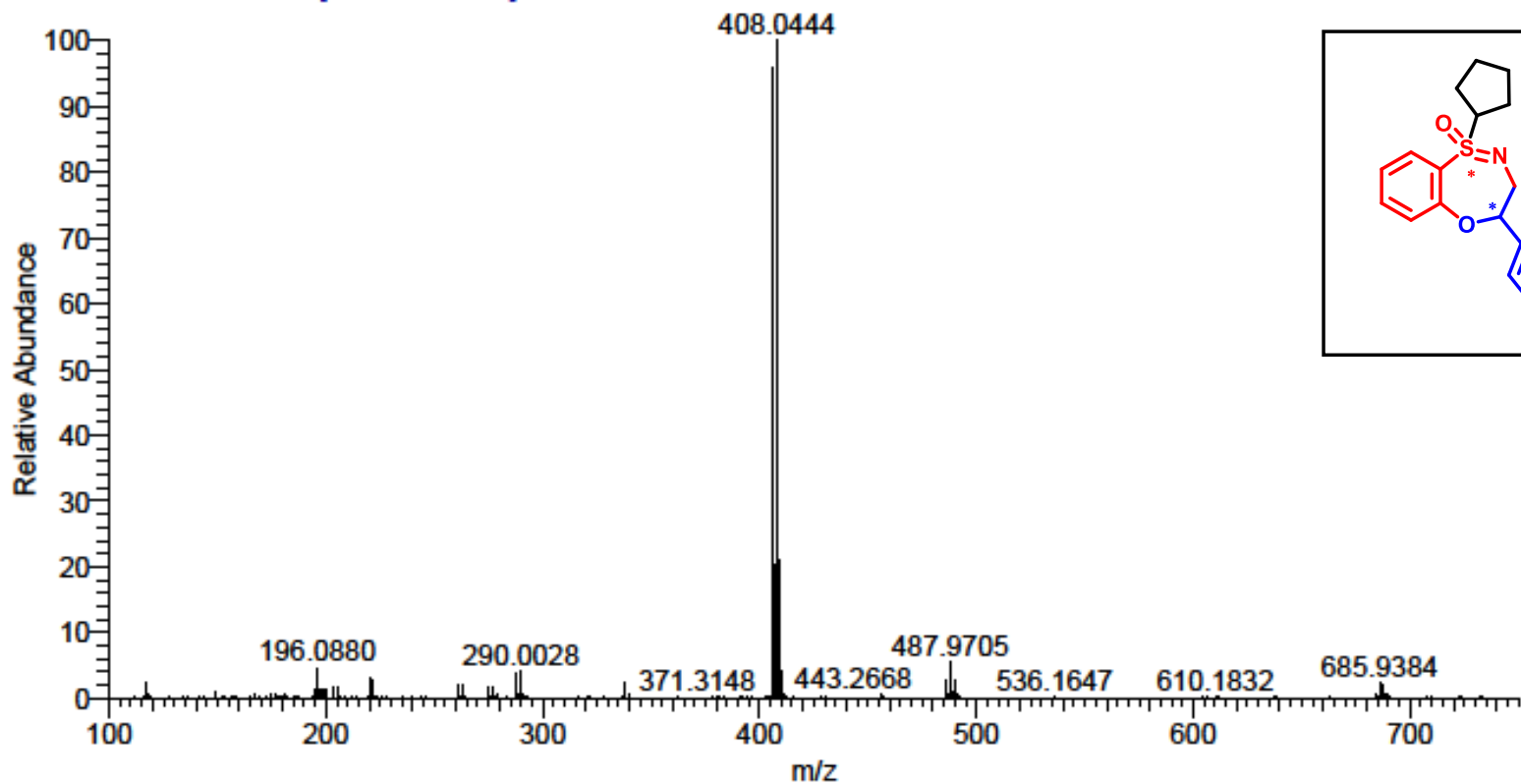


Fig S-107: HRMS report of Compound 4o'

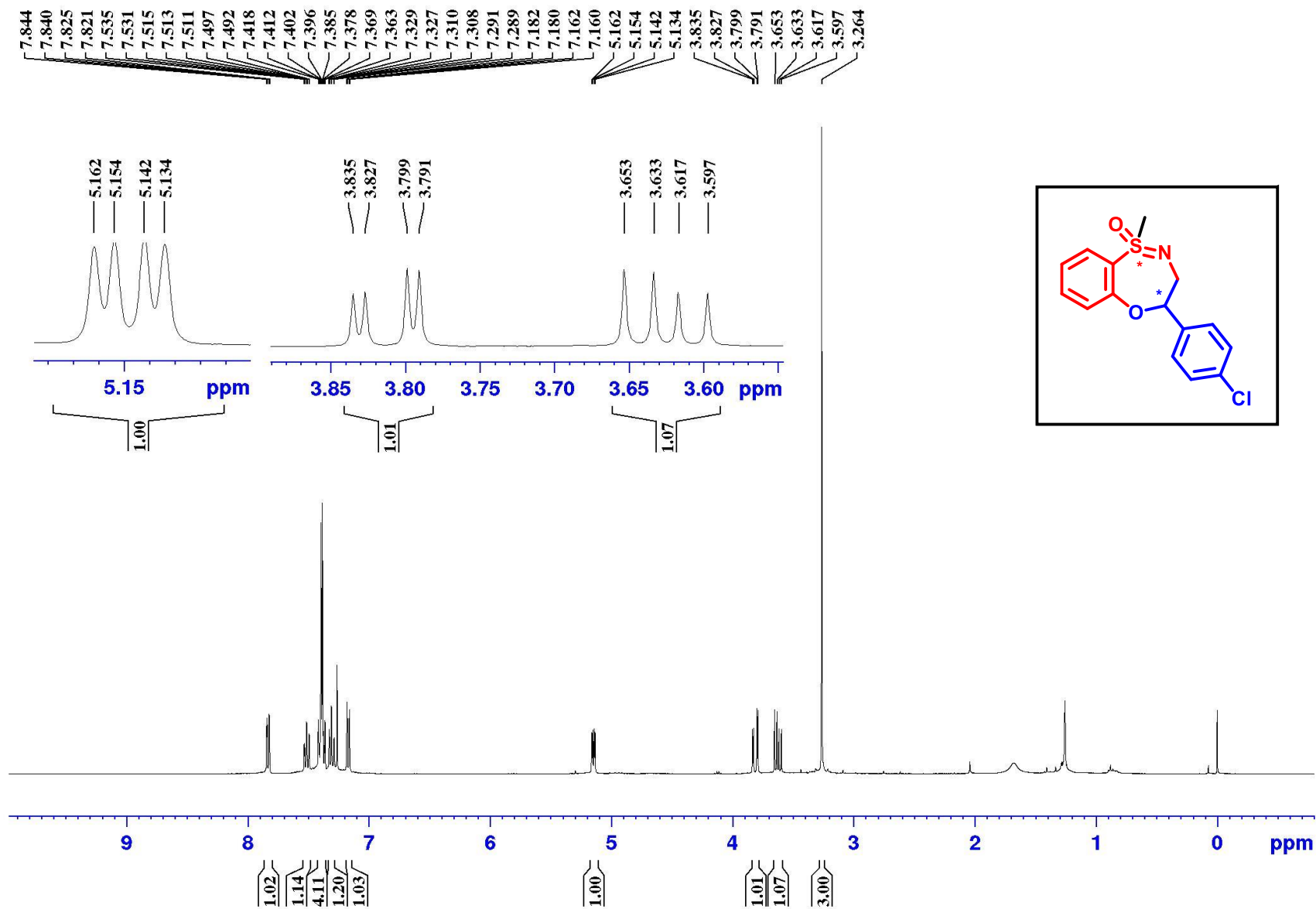


Fig S-108: ¹H NMR Spectra of Compound **4p** (400 MHz, CDCl₃)

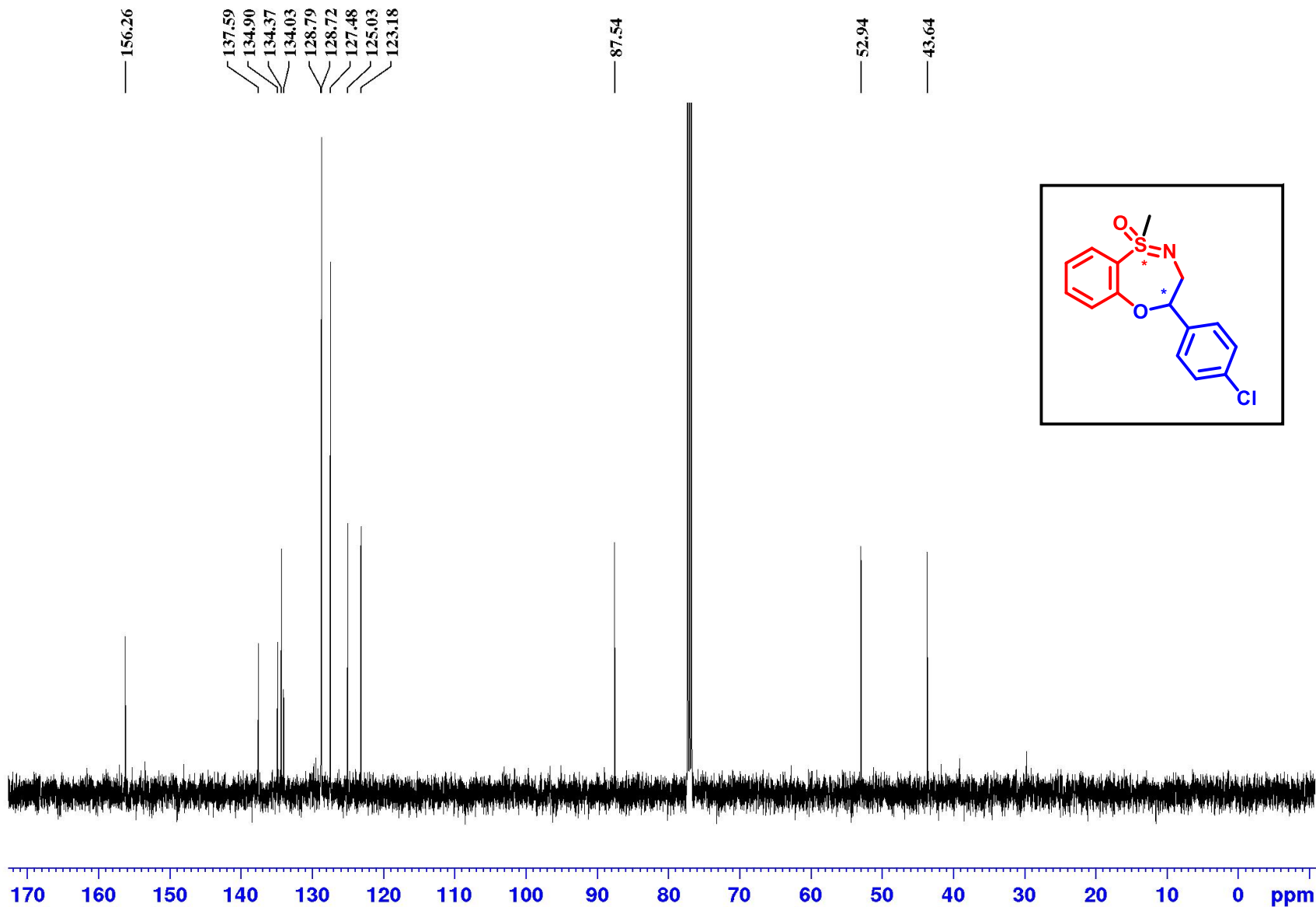


Fig S-109: ^{13}C NMR Spectra of Compound **4p** (125 MHz, CDCl_3)

Sample Name	ASA 1610	Position	Vial 24	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I29APR24.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	4/29/2022 1:39:29 PM

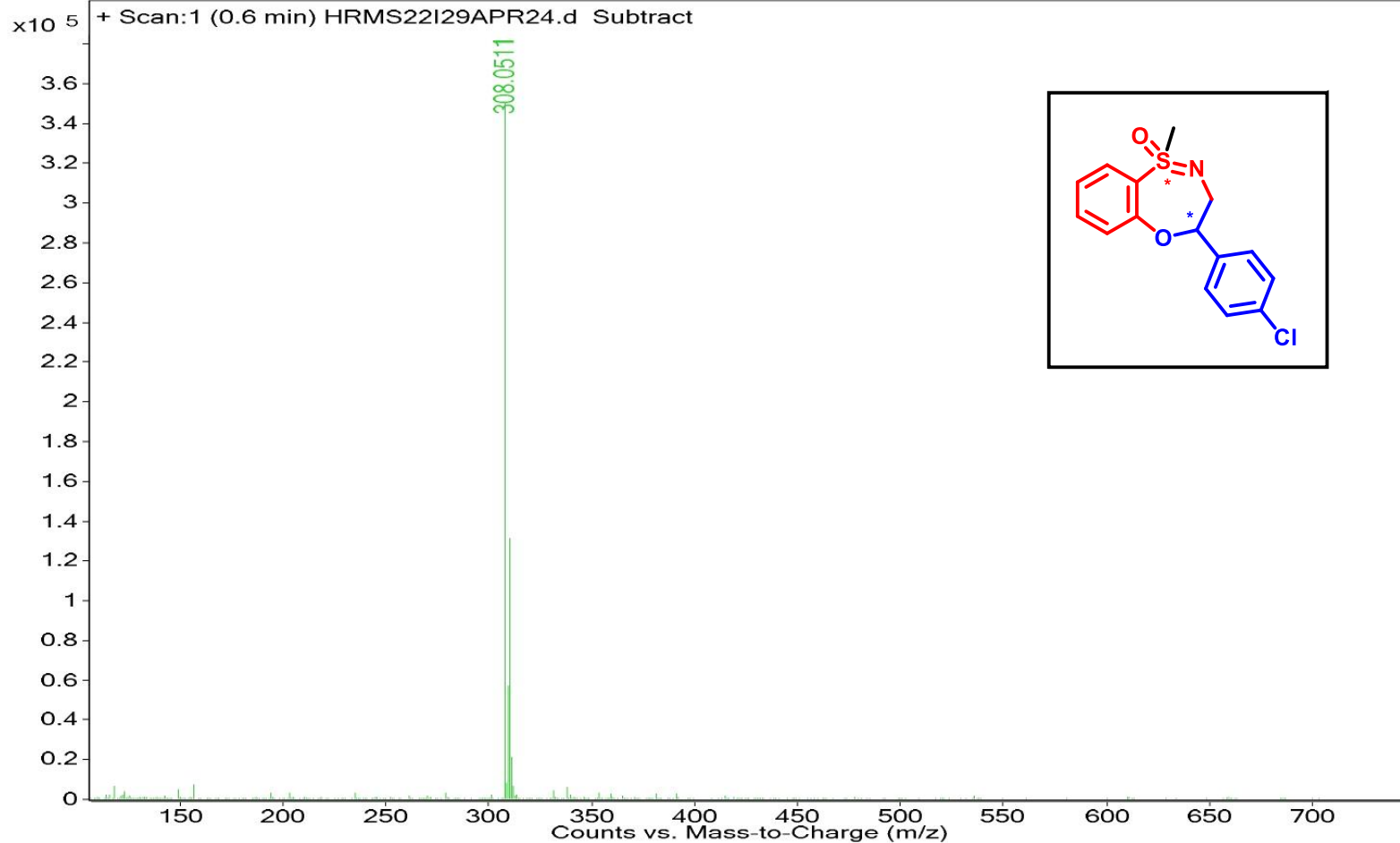


Fig S-110: HRMS report of Compound **4p**

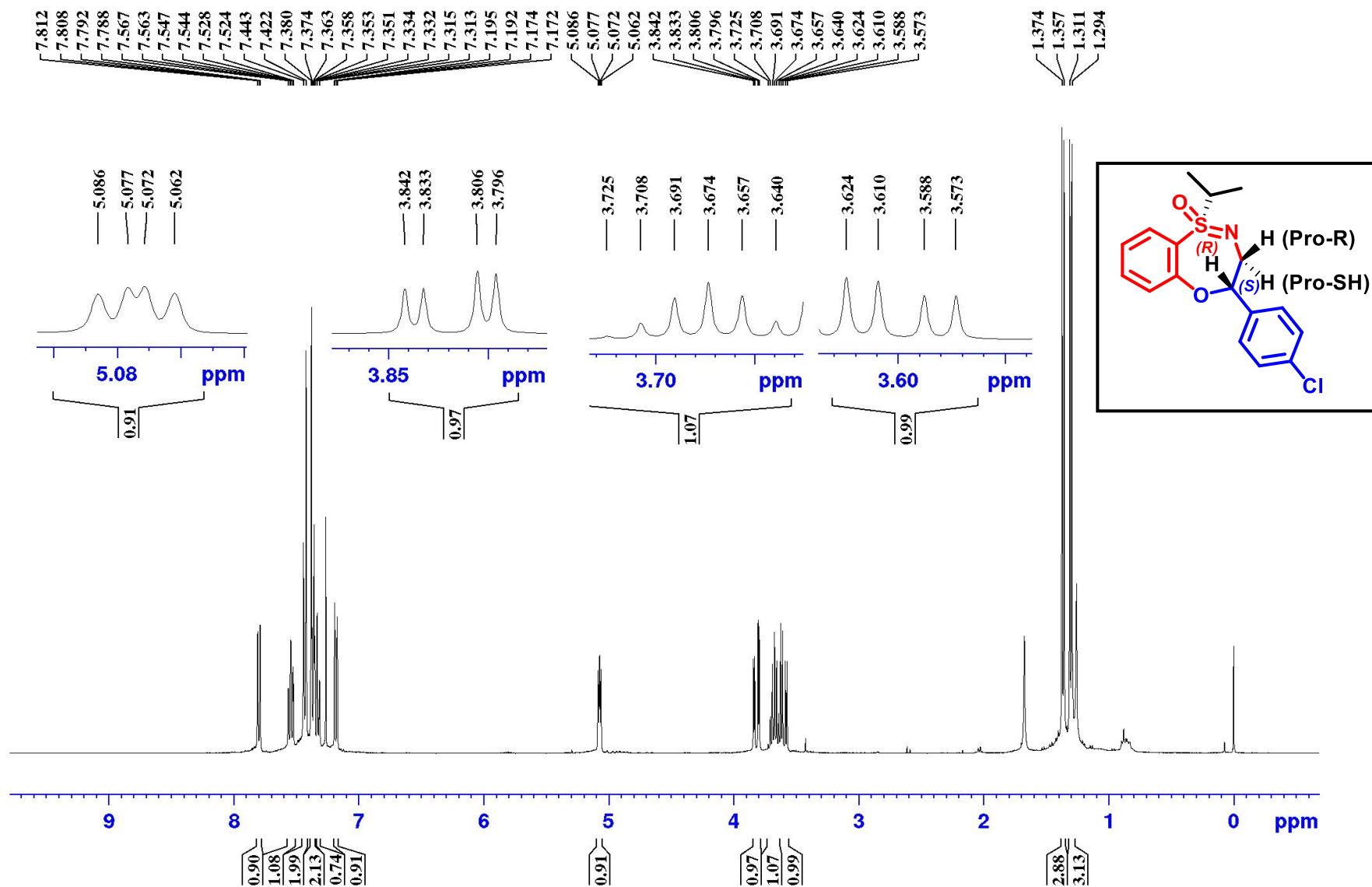


Fig S-111: ¹H NMR Spectra of Compound (S,R) 4q (400 MHz, CDCl₃)

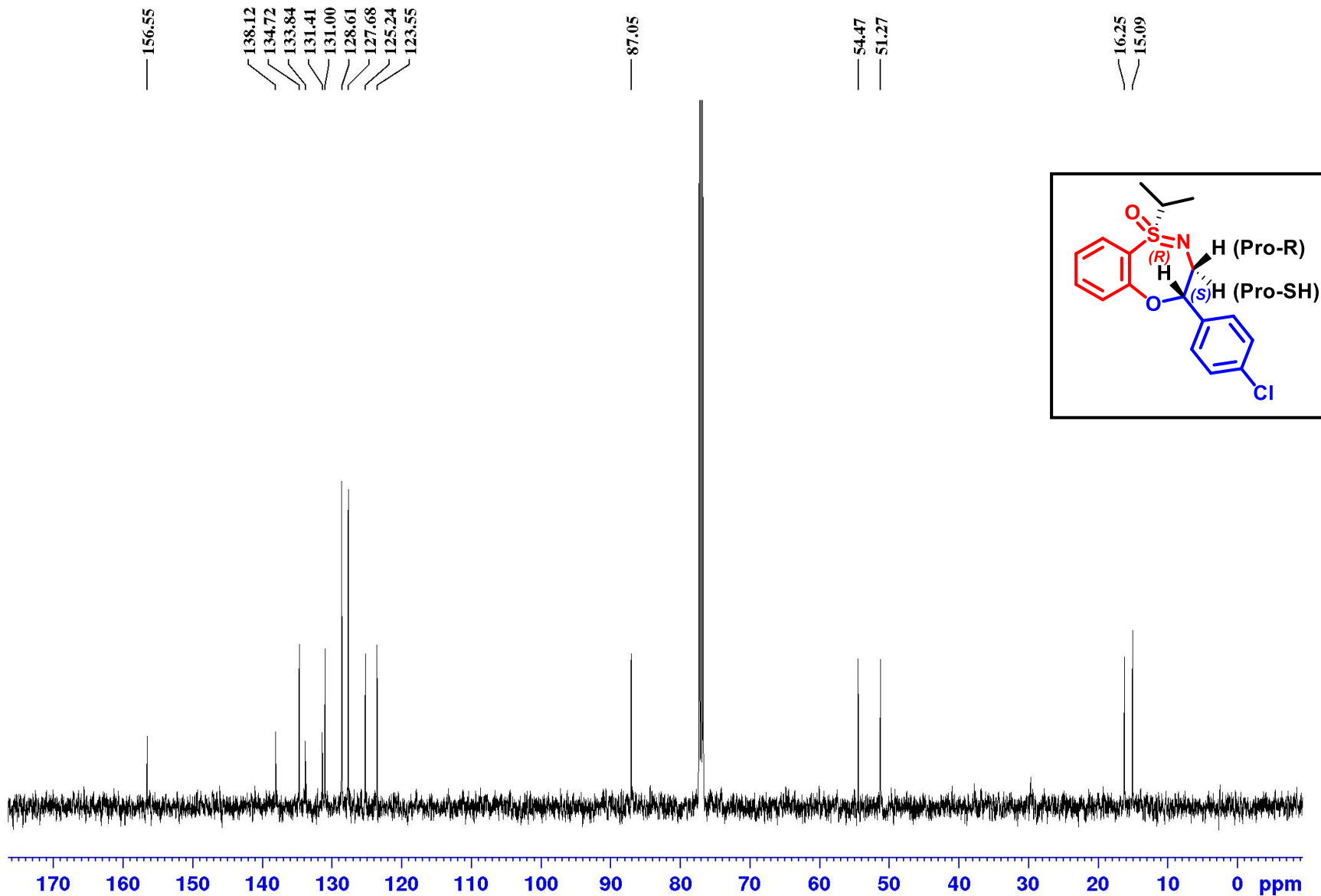


Fig S-112: ^{13}C NMR Spectra of Compound (*S,R*) 4q (125 MHz, CDCl_3)

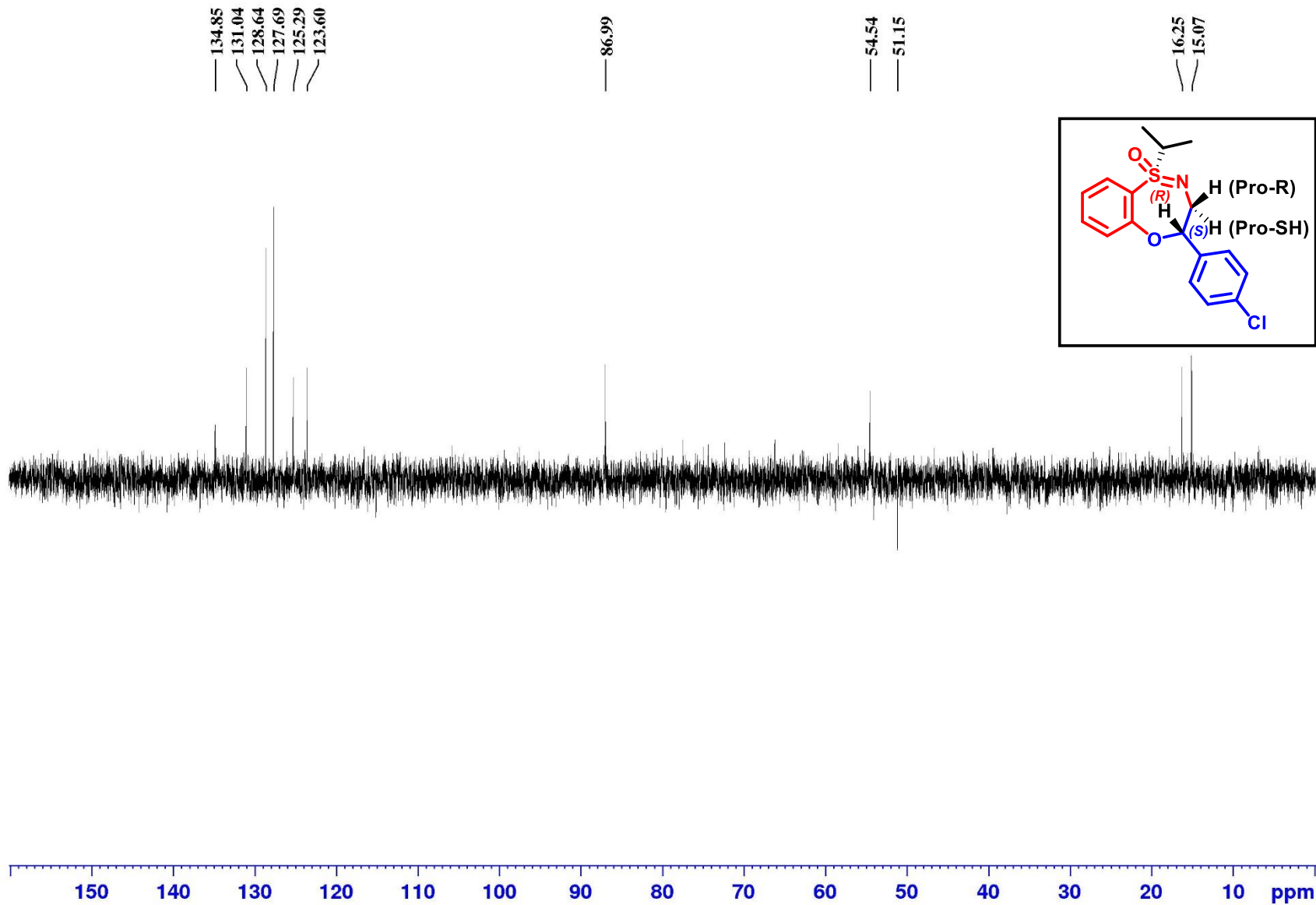


Fig S-113: DEPT 135 Spectra of Compound (*S,R*) 4q (100 MHz, CDCl₃)

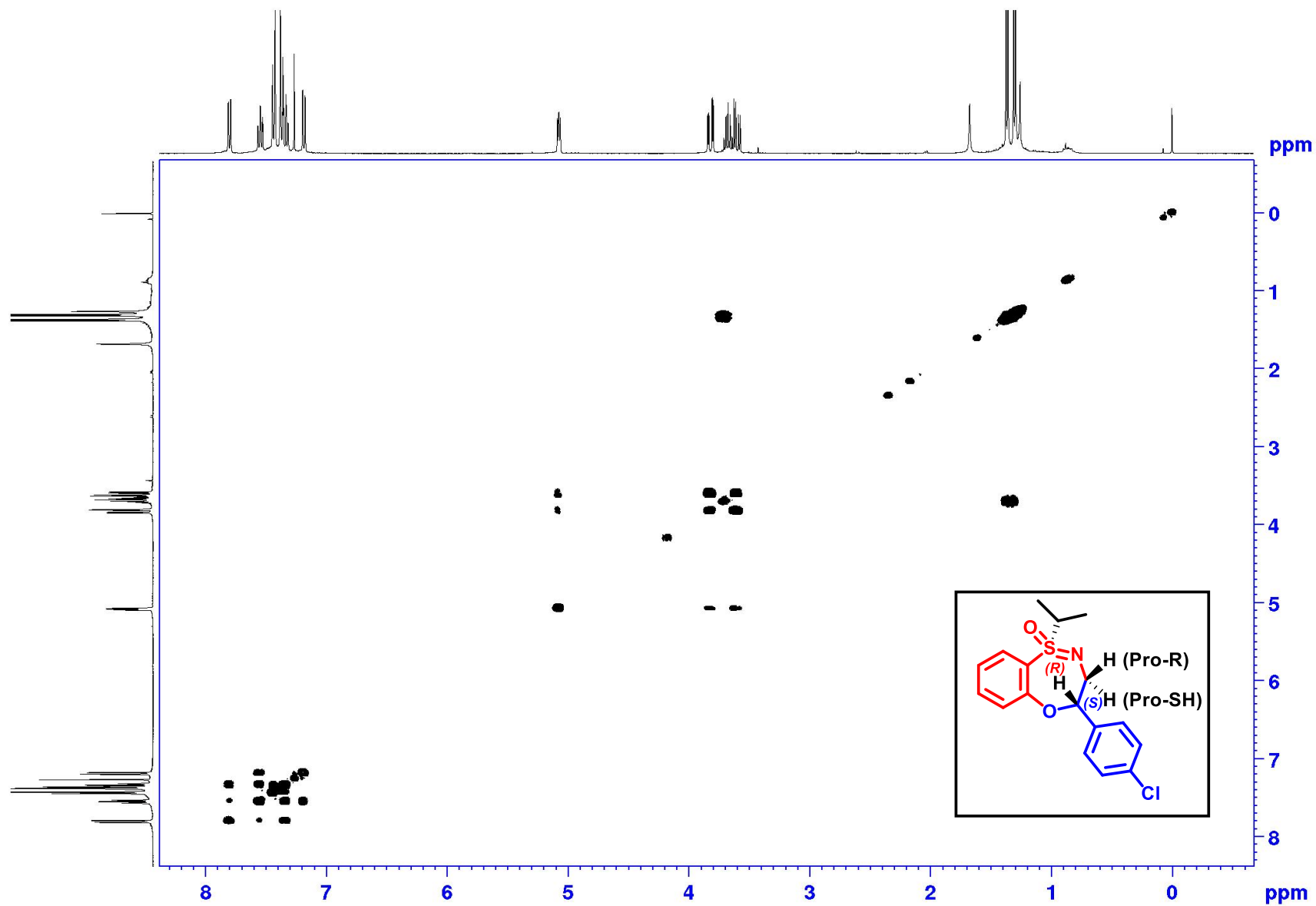


Fig S-114: COSY Spectra of Compound (*S,R*) 4q (400 MHz, CDCl₃)

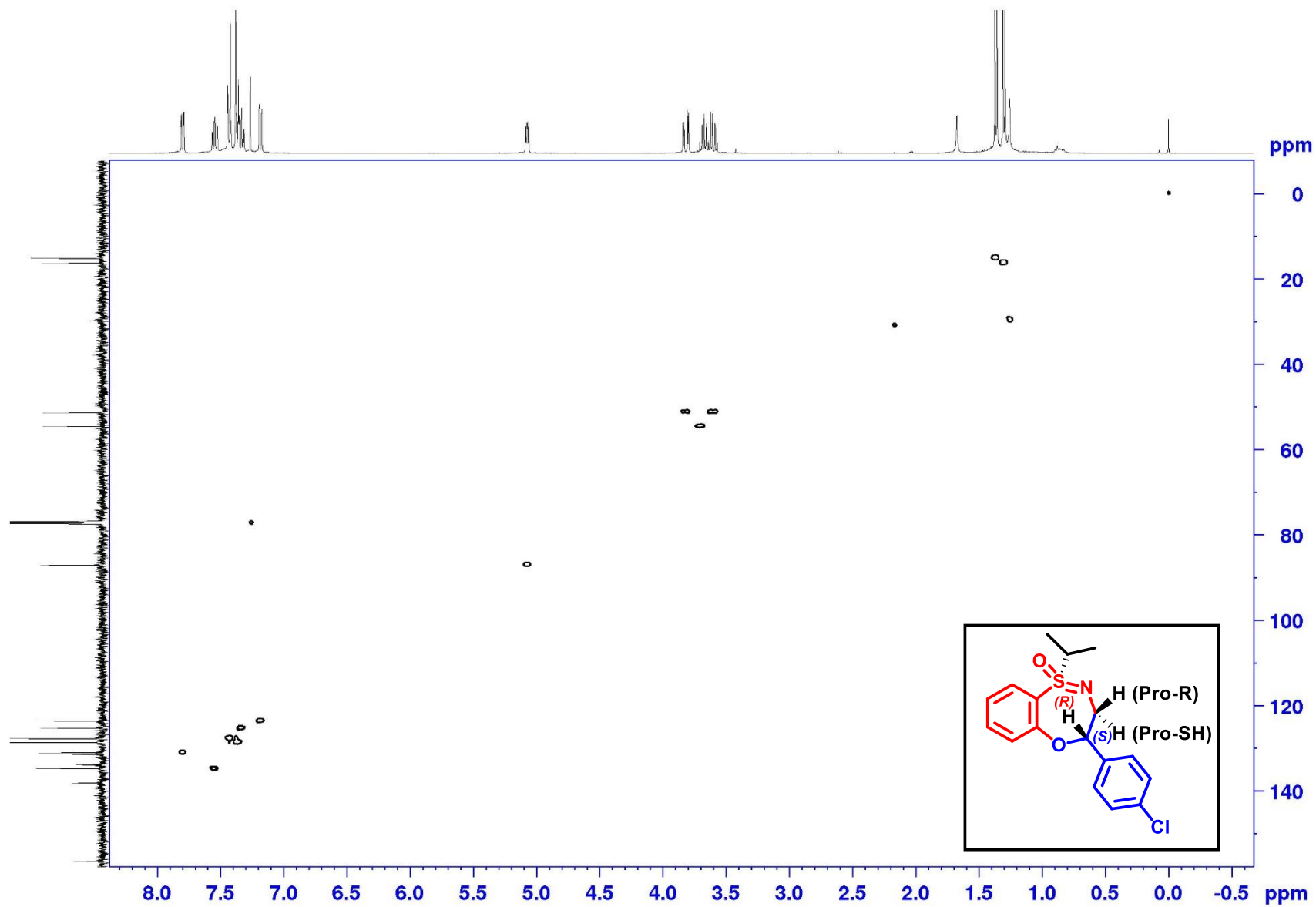


Fig S-115: HSQC Spectra of Compound (*S,R*) 4q (400 MHz, CDCl₃)

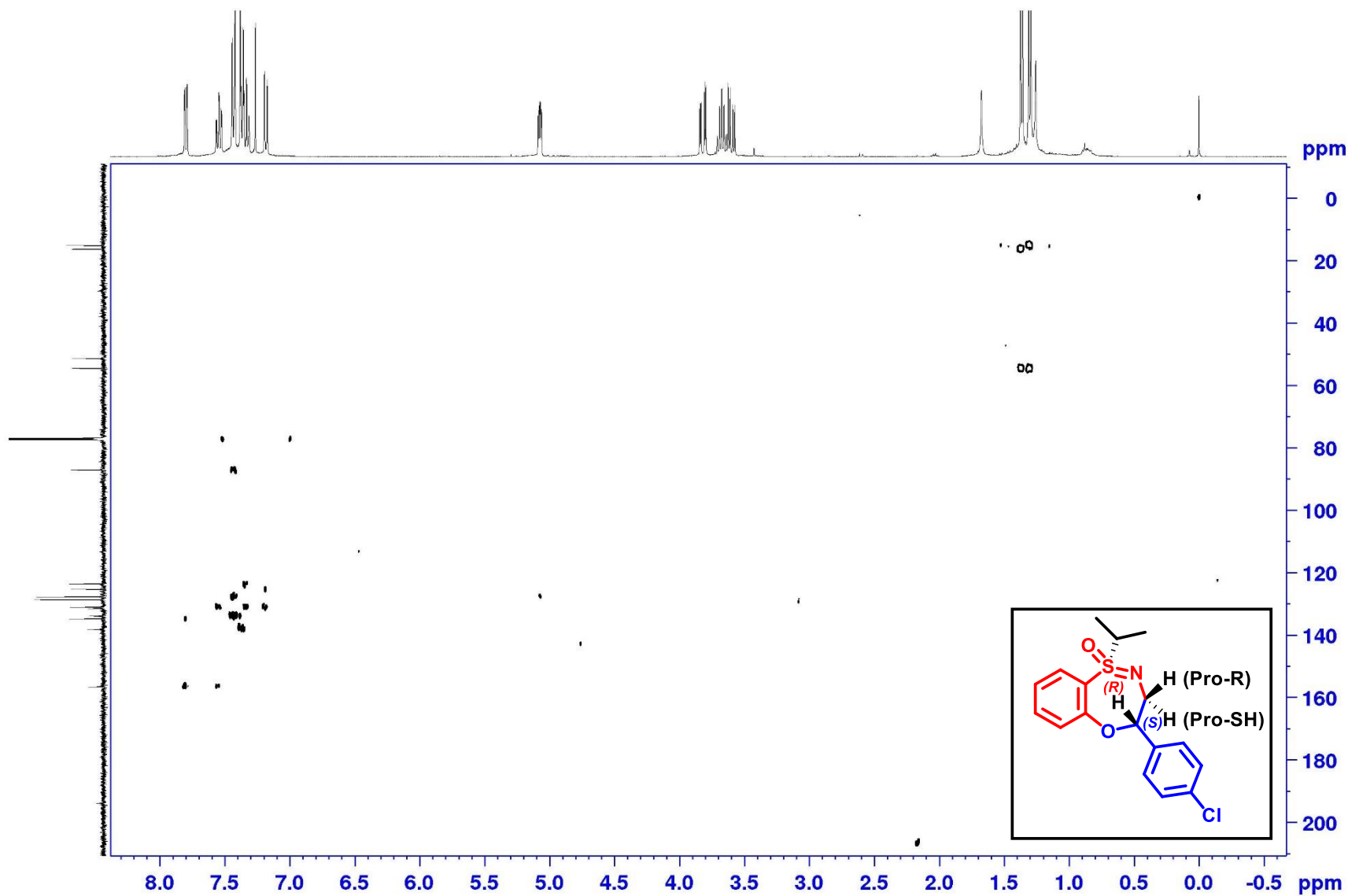


Fig S-116: HMBC Spectra of Compound (S,R) 4q (400 MHz, CDCl₃)

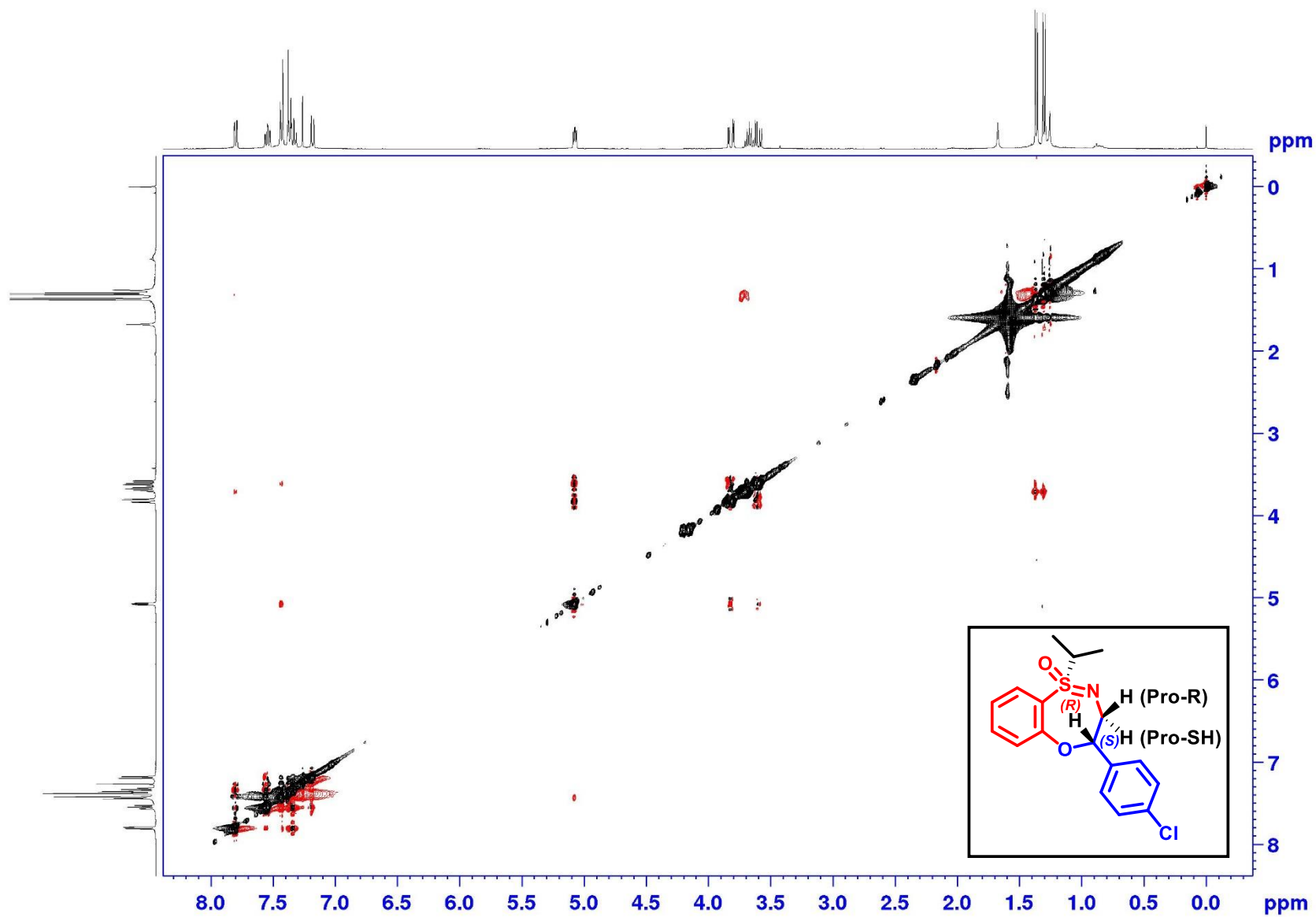


Fig S-117: NOESY Spectra of Compound (S,R) 4q (500 MHz, CDCl₃)

SAIF [HRMS Report]

Data File:	HRMS21I06APR14	Original Data Path:	D:\INTERNAL NEW\2021\April 2021
Sample ID:	AB-176A	Sample Name:	
Acquisition Date:	04/06/21 11:14:27 AM	Run Time(min):	0.00
Vial:	CSik1-01:14	Injection Volume(μl):	1.00

HRMS21I06APR14 #33-65 RT: 0.25-0.50 AV: 33 SB: 1 0.01 NL: 2.87E6
T: FTMS + c ESI Full ms [100.00-750.00]

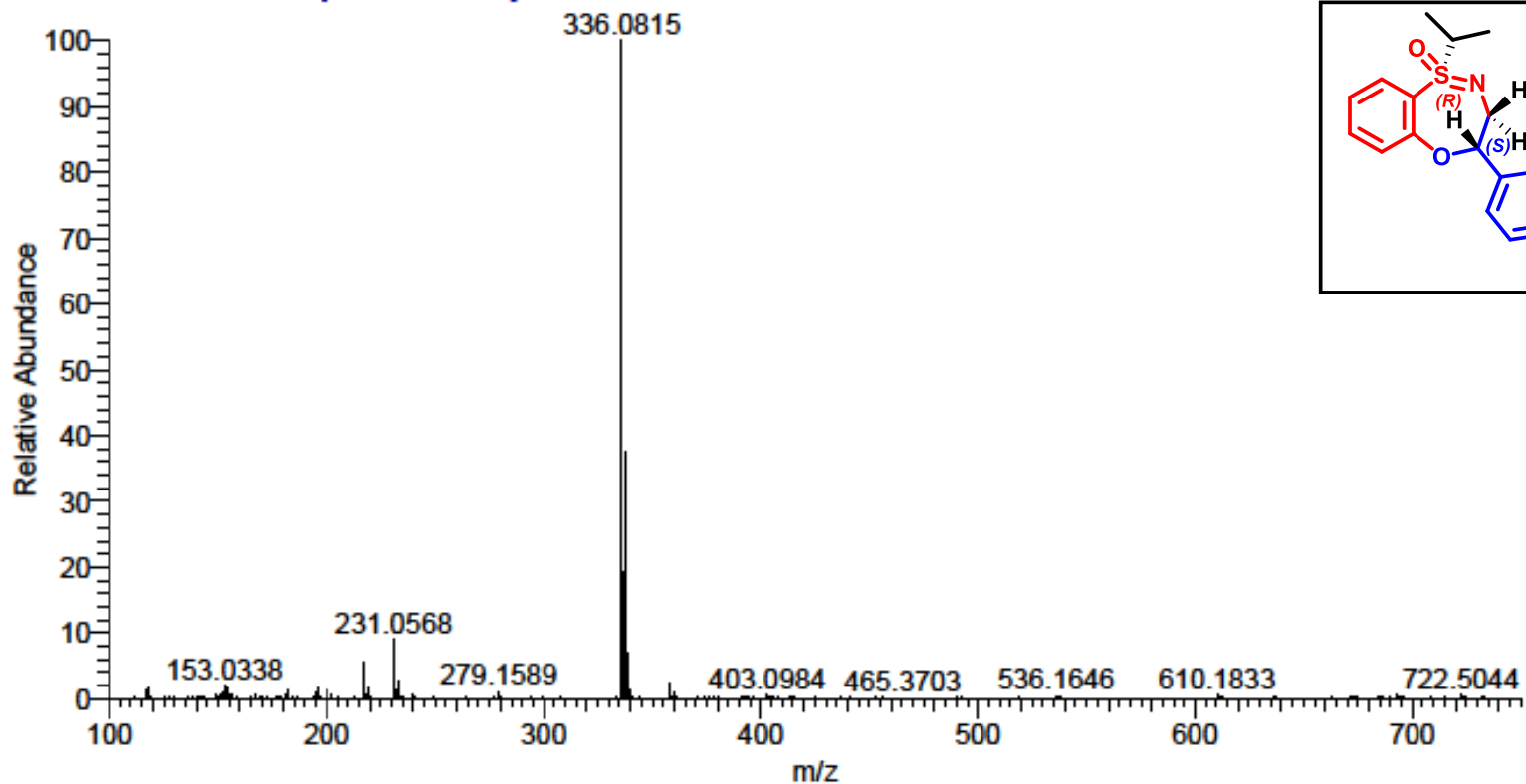


Fig S-118: HRMS report of Compound (S,R)-4q

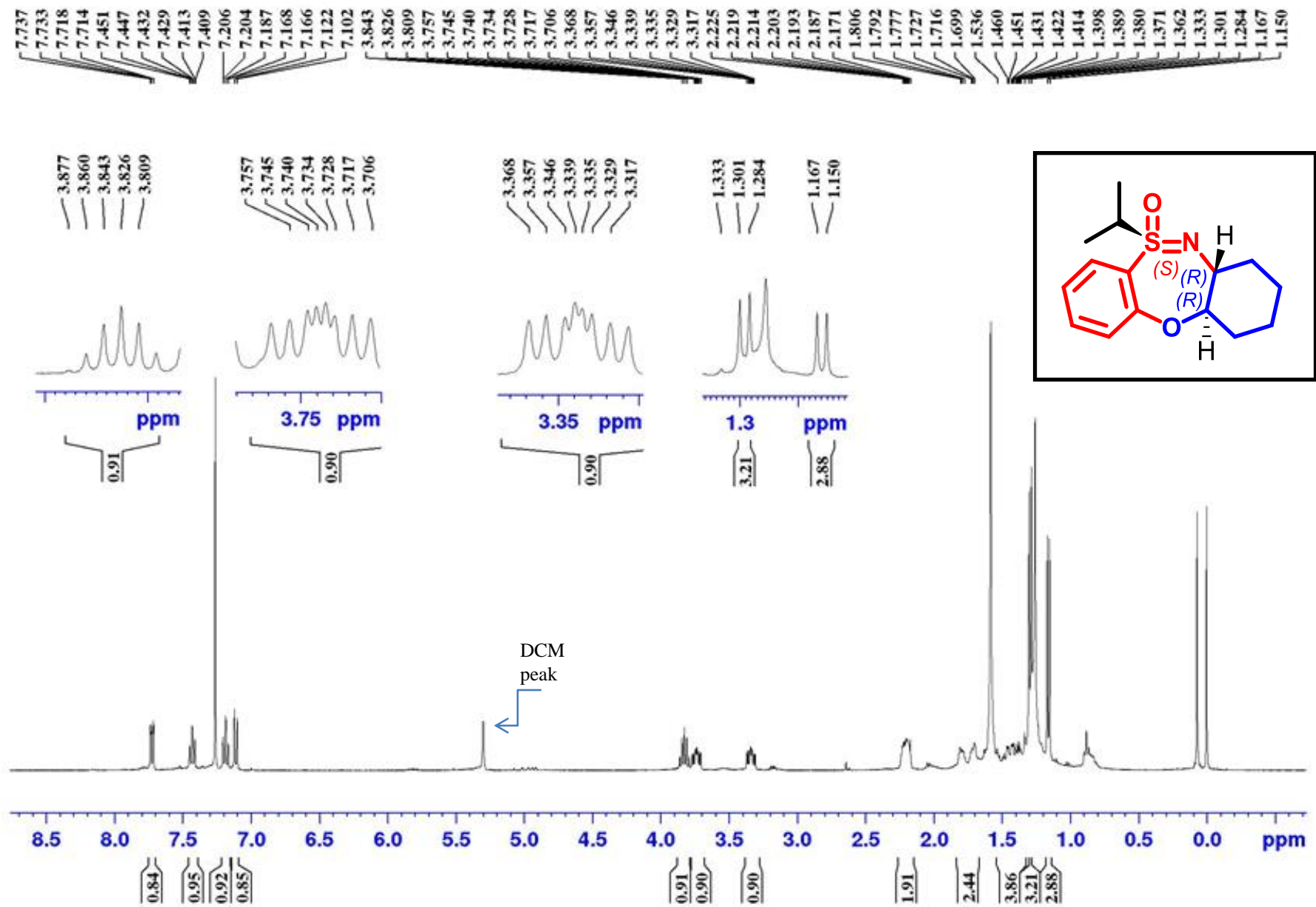


Fig S-119: ¹H NMR Spectra of Compound *(R,R,S)*-4r (400 MHz, CDCl₃)

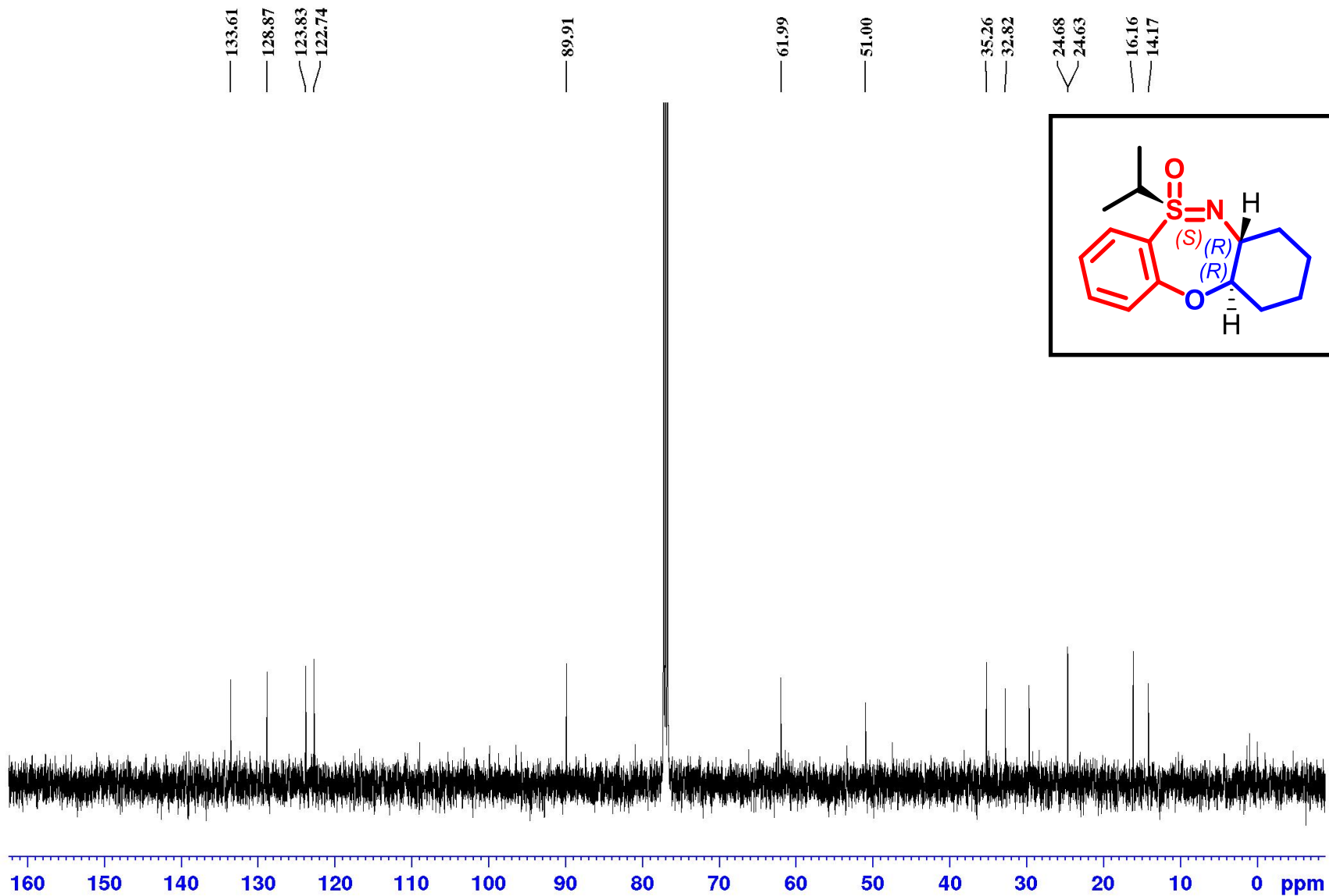


Fig S-120: ^{13}C NMR Spectra of Compound *(R,R,S)*-4r (125 MHz, CDCl_3)

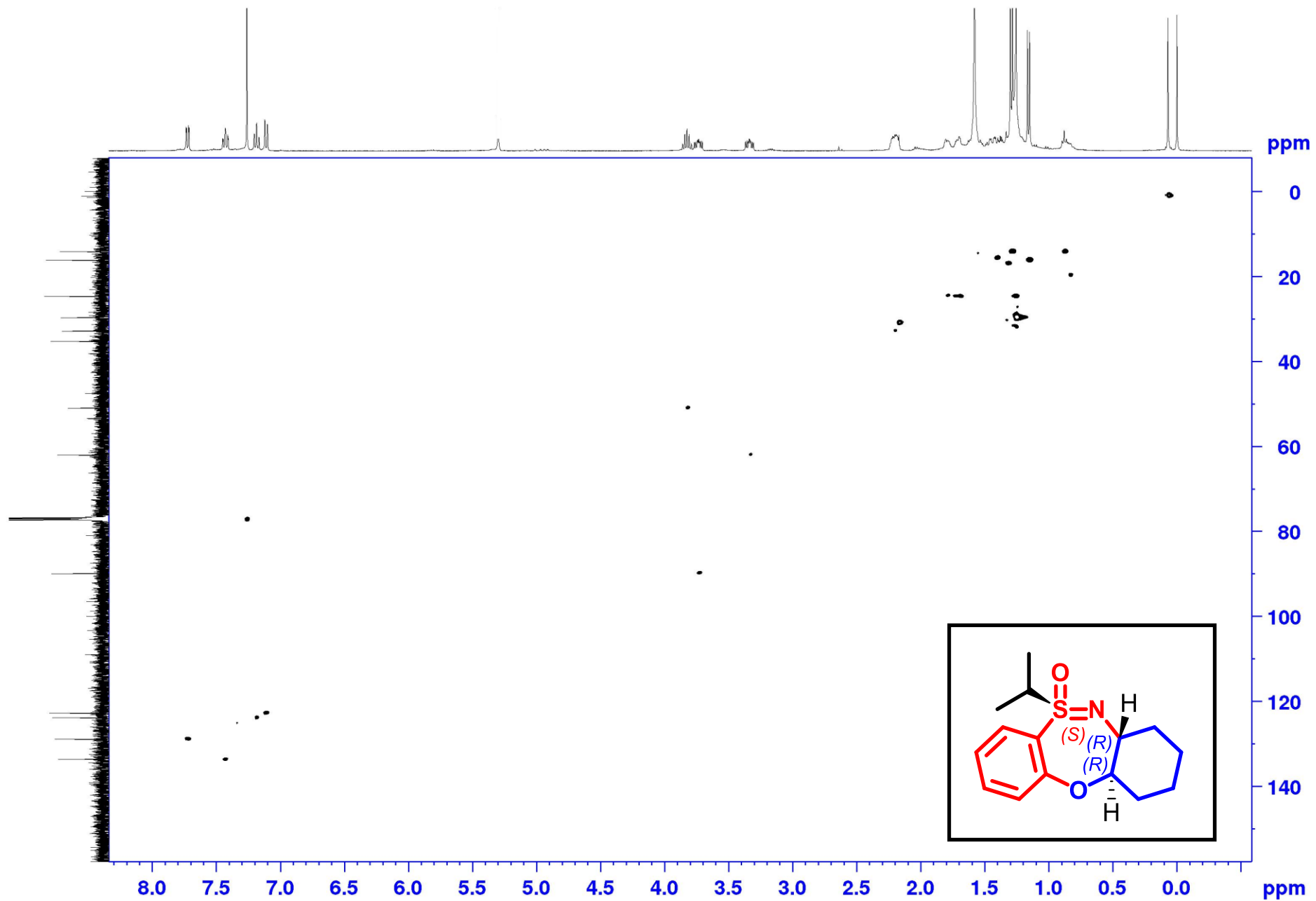


Fig S-121: HSQC Spectra of Compound **(R,R,S)-4r** (400 MHz, CDCl_3)

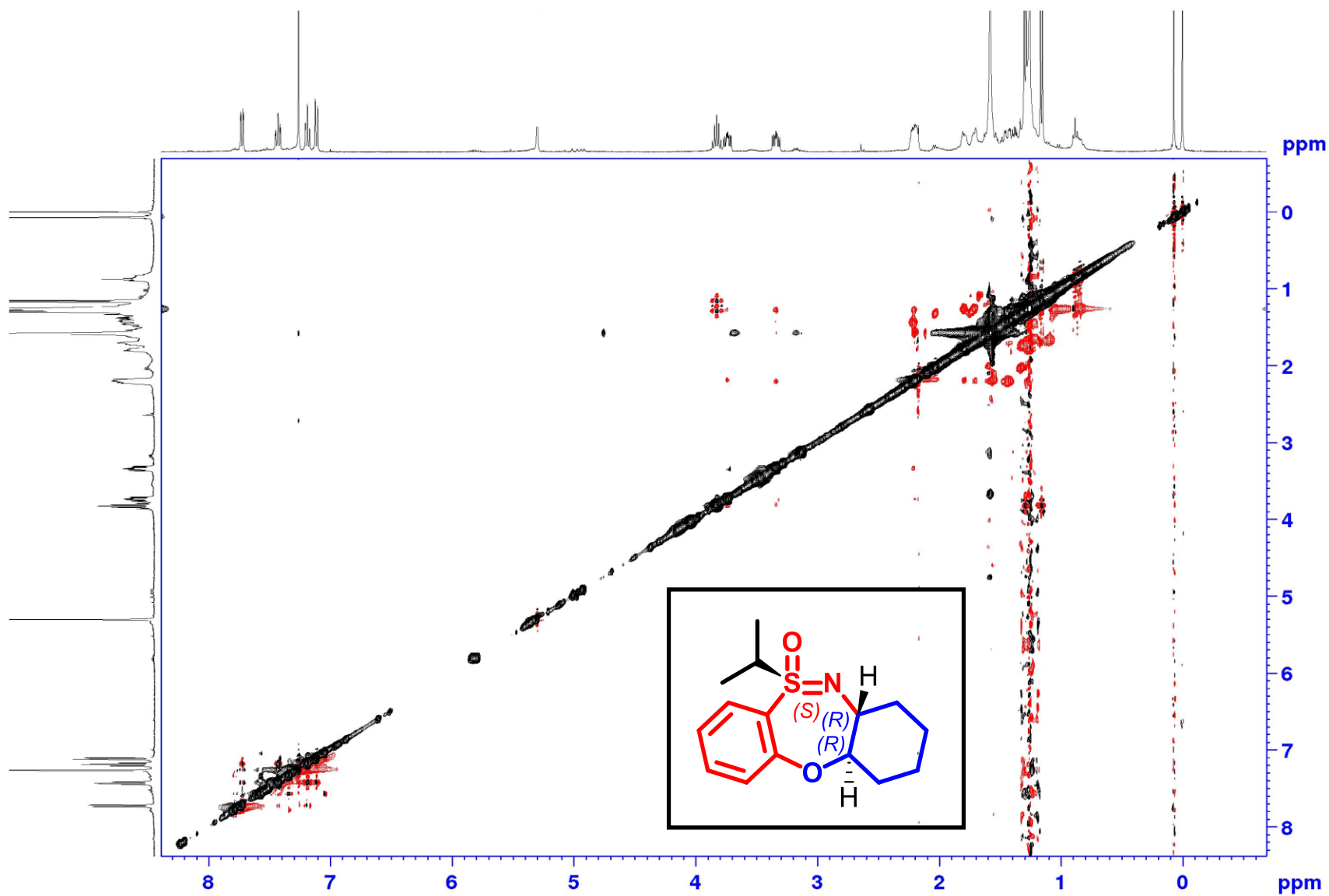


Fig S-122: NOESY Spectra of Compound (R,R,S)-4r (500 MHz, CDCl₃)

Sample Name	Ena-2	Position	Vial 26	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22102MAY26.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	5/2/2022 1:49:14 PM

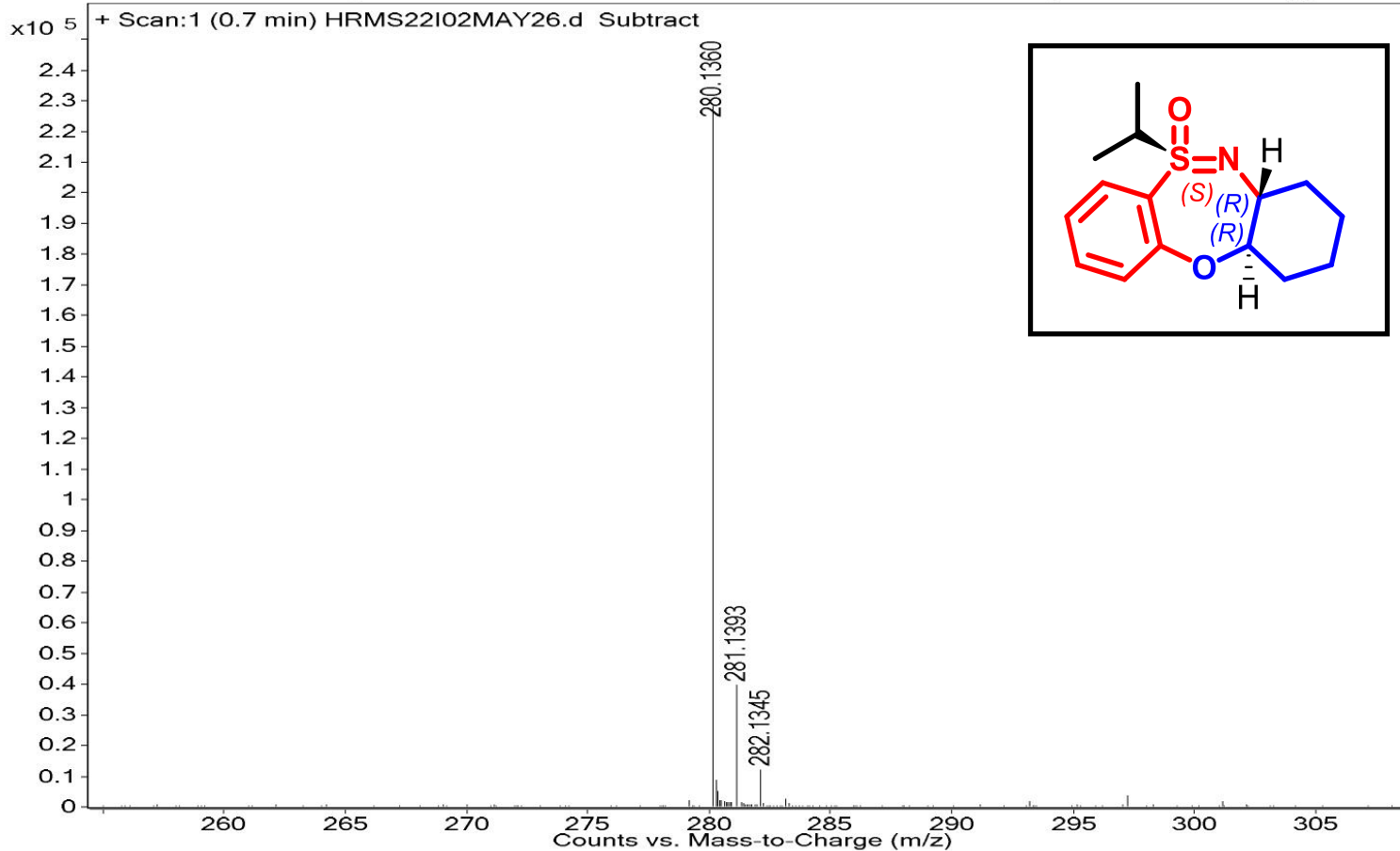


Fig S-123: HRMS report of Compound (*R,R,S*)-4r

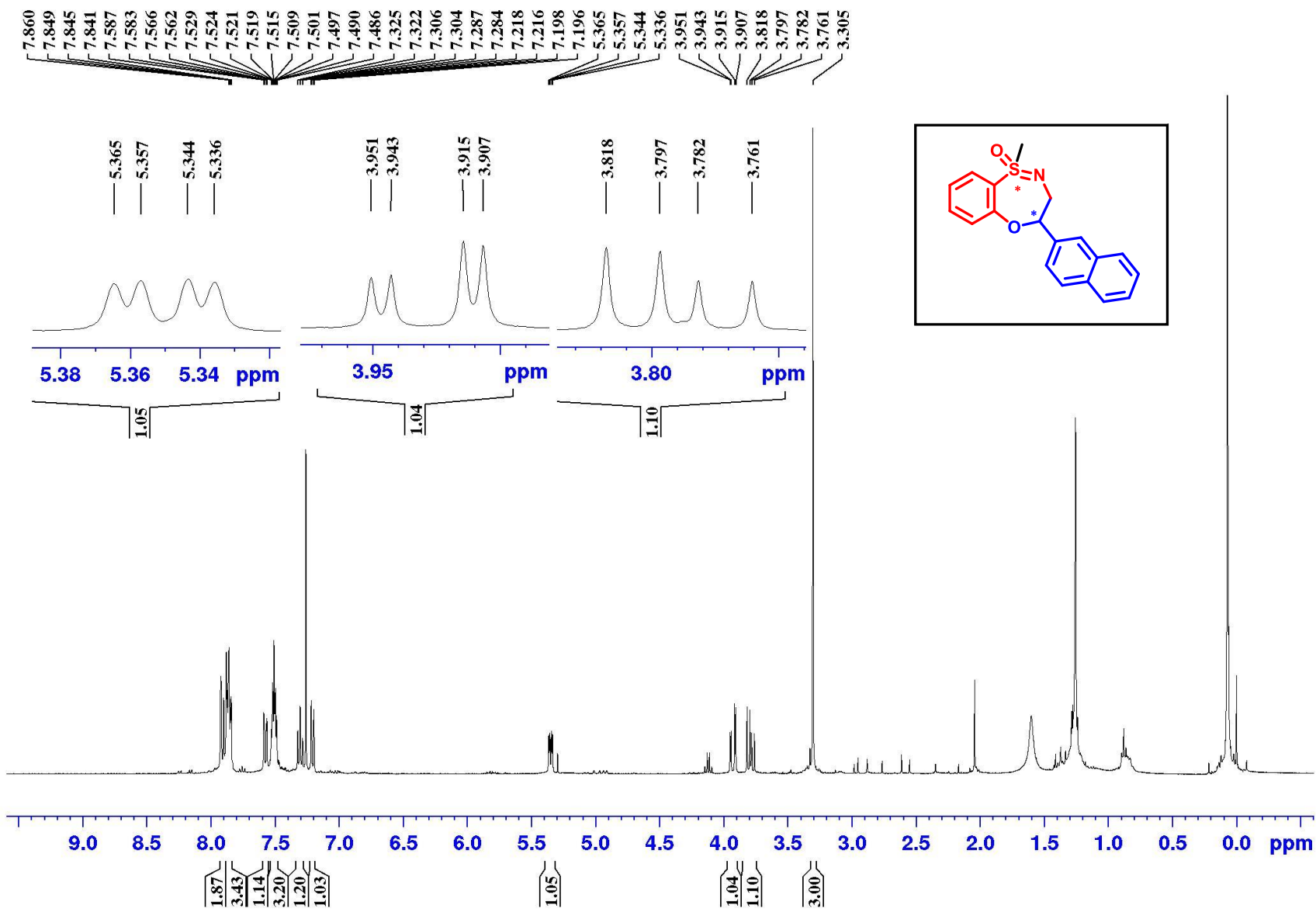


Fig S-124: ^1H NMR Spectra of Compound **4s**(400 MHz, CDCl_3)

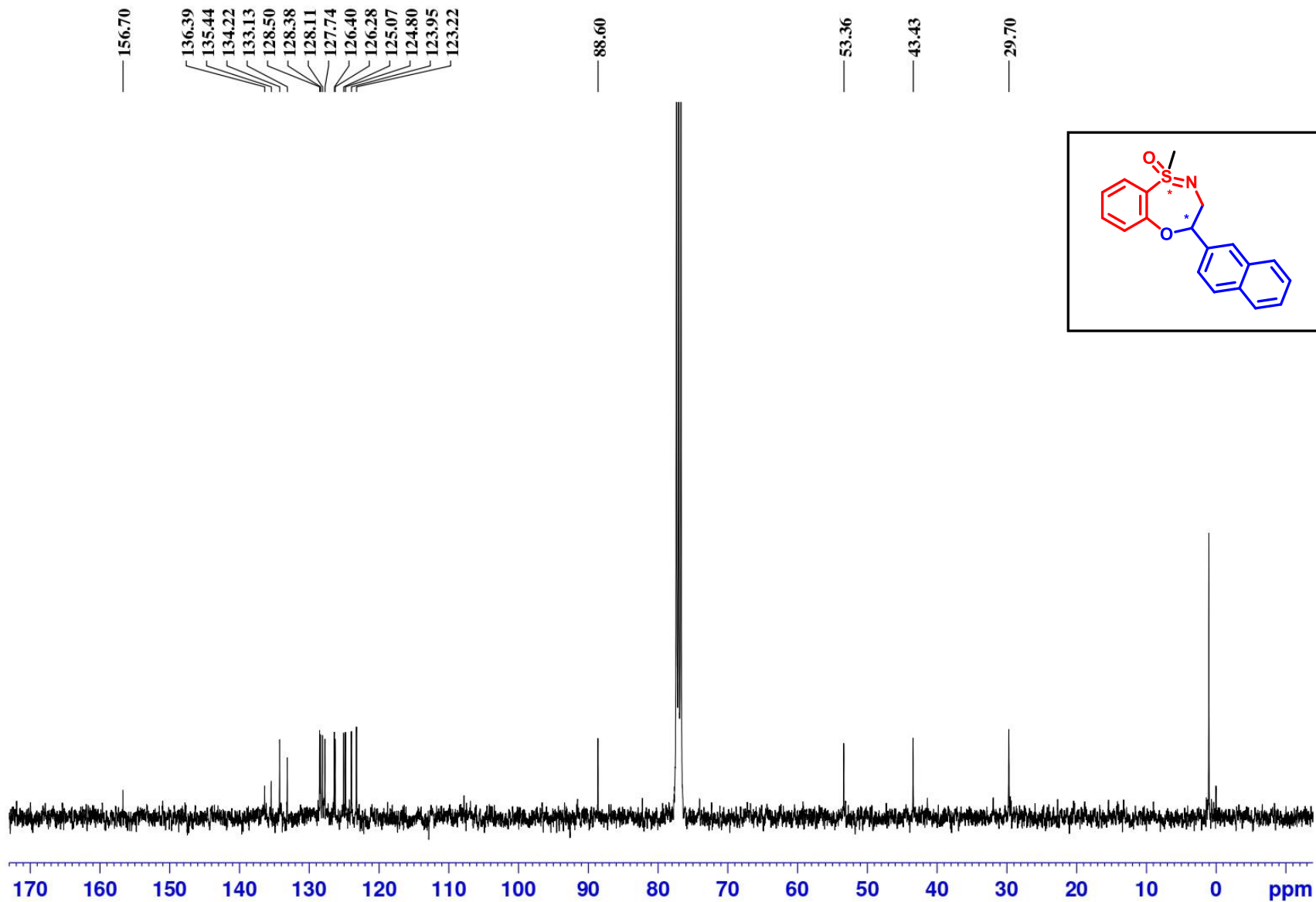


Fig S-125: ^{13}C NMR Spectra of Compound **4s** (125 MHz, CDCl_3)

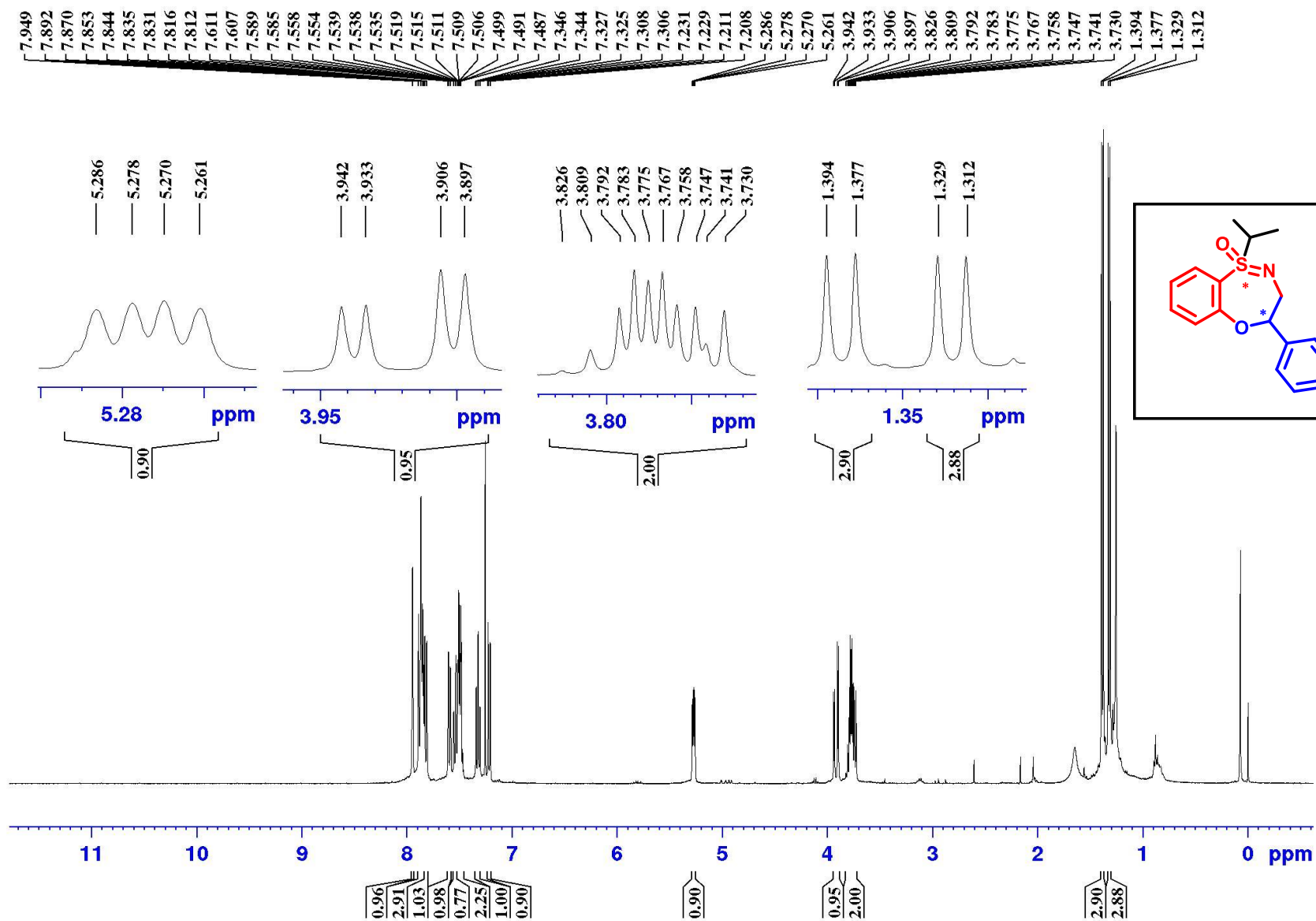


Fig S-126: ^1H NMR Spectra of Compound **4t** (400 MHz, CDCl_3)

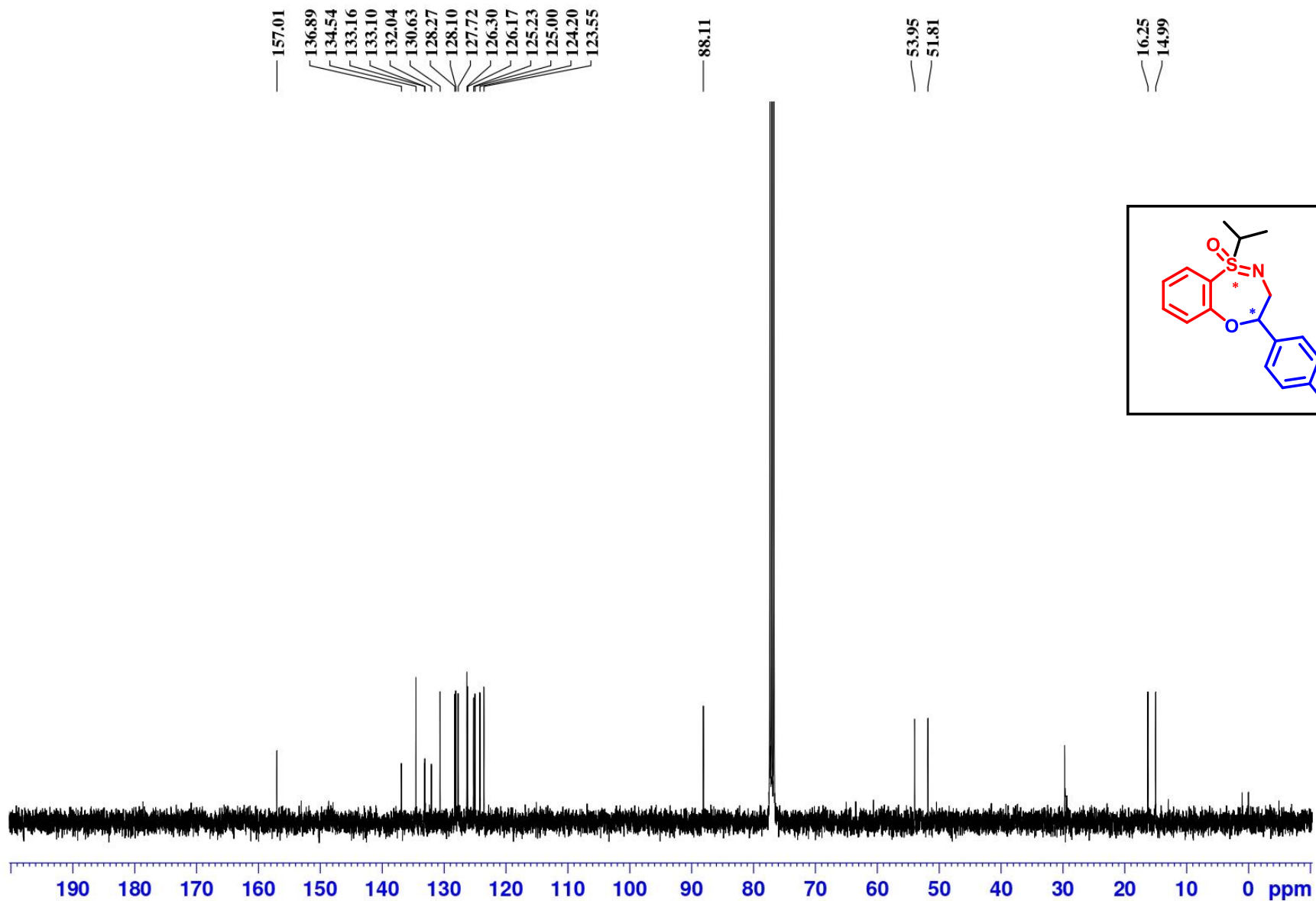


Fig S-127: ^{13}C NMR Spectra of Compound **4t** (100 MHz, CDCl_3)

Sample Name	2i	Position	Vial 27	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I02MAY27.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	5/2/2022 1:54:55 PM

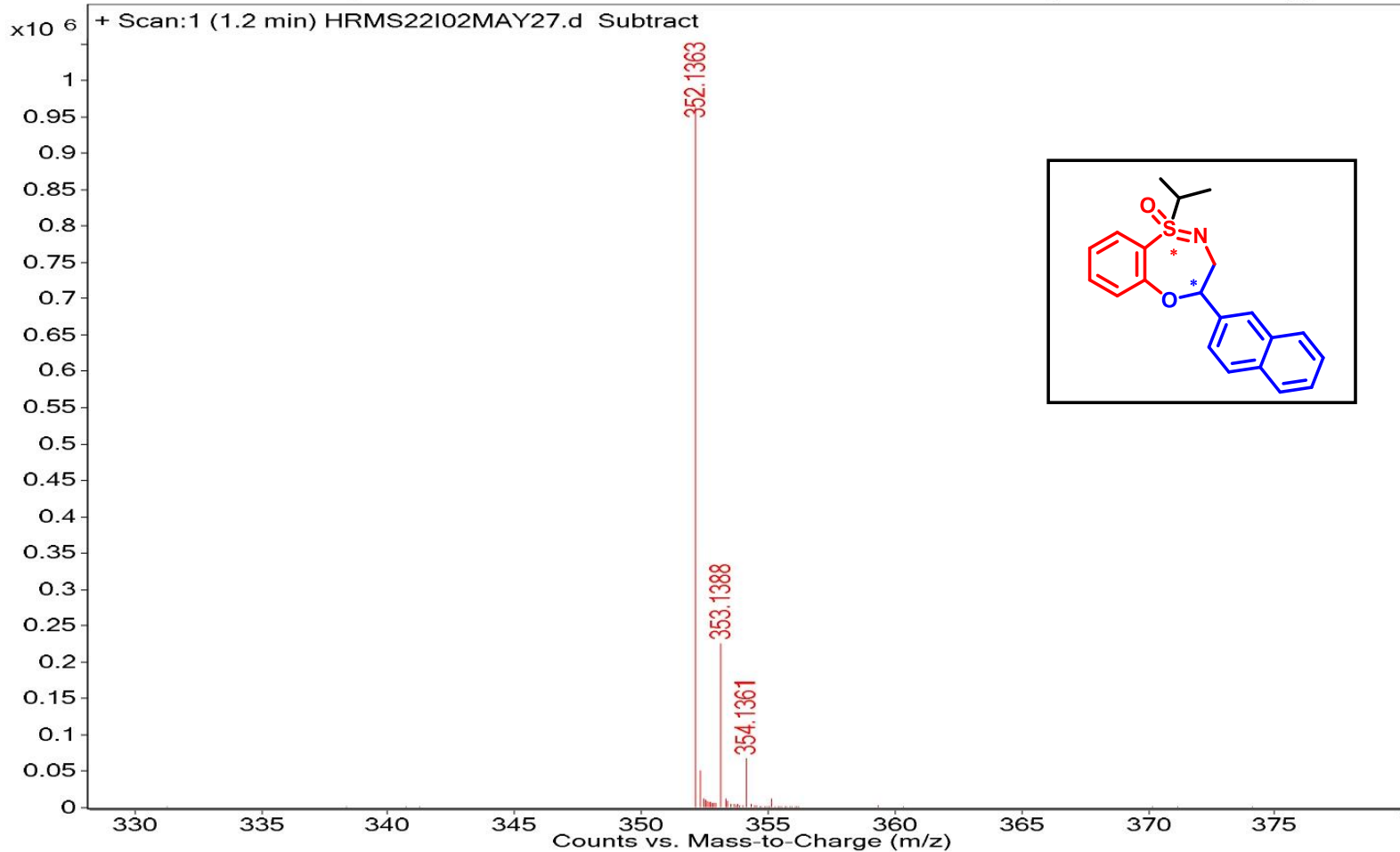


Fig S-128: HRMS report of Compound **4t**

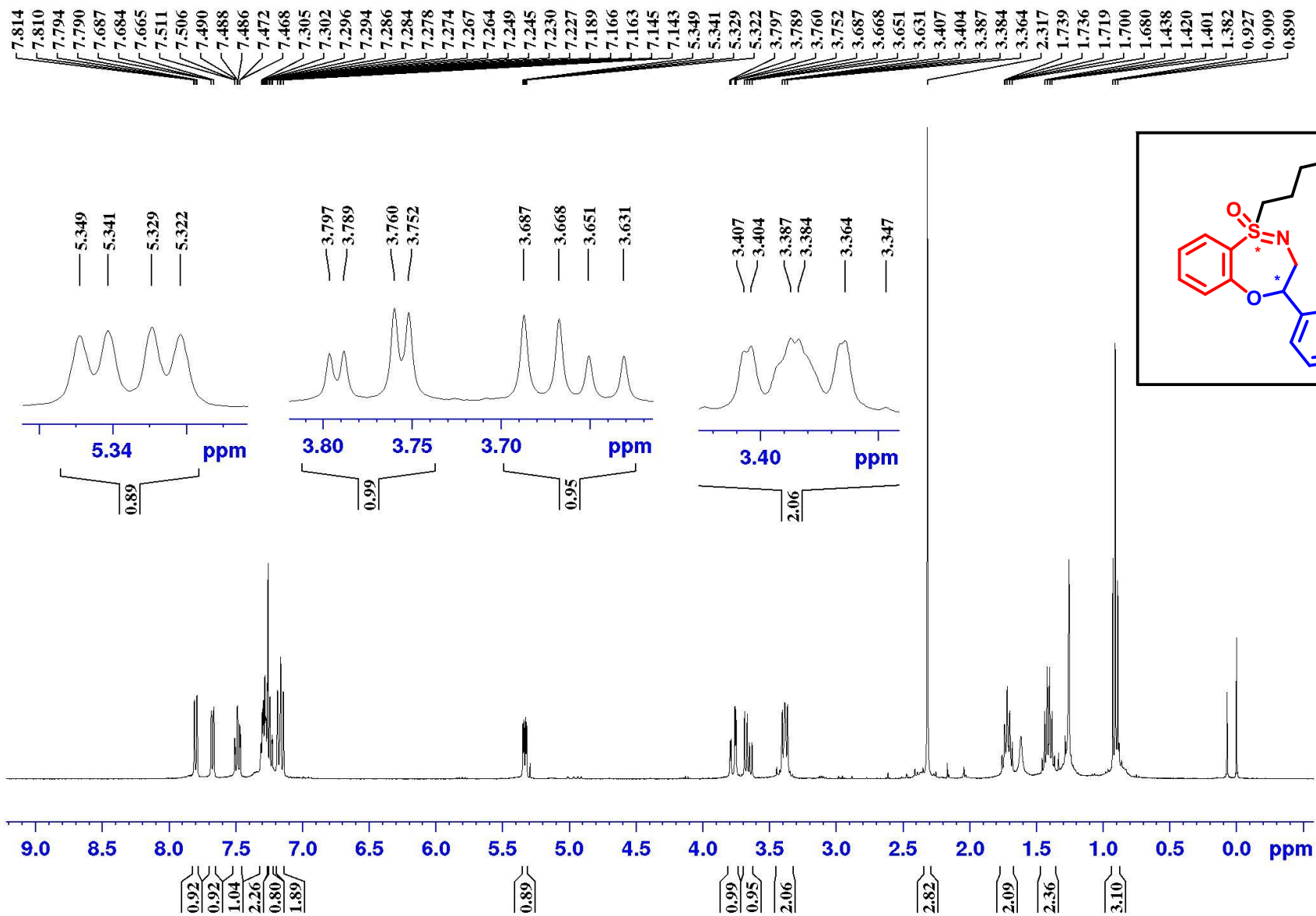


Fig S-129: ¹H NMR Spectra of Compound 4u (400 MHz, CDCl₃)

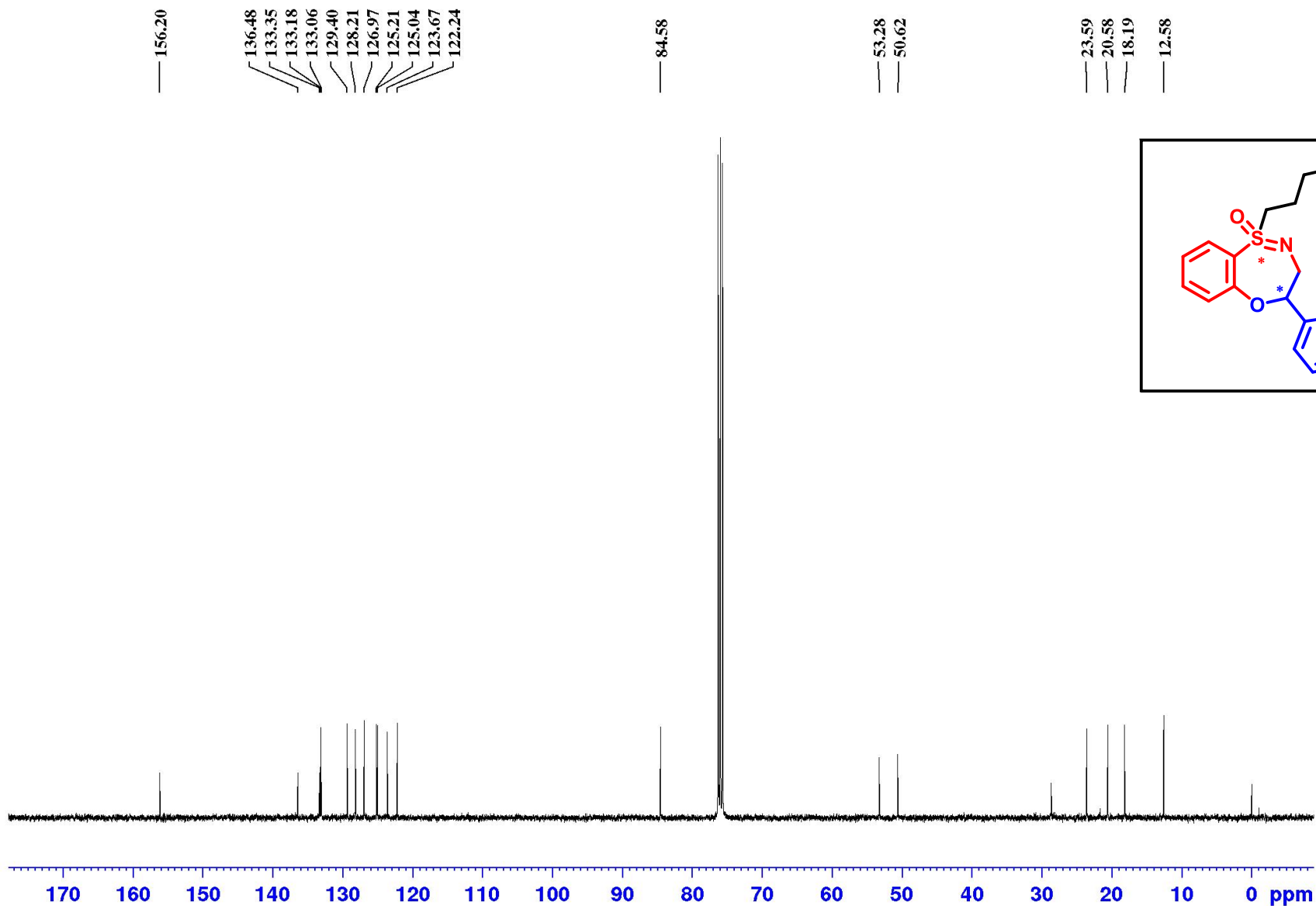


Fig S-130: ^{13}C NMR Spectra of Compound **4u** (125 MHz, CDCl_3)

Sample Name	asa1391p	Position	Vial 25	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I29APR25.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	4/29/2022 1:45:03 PM

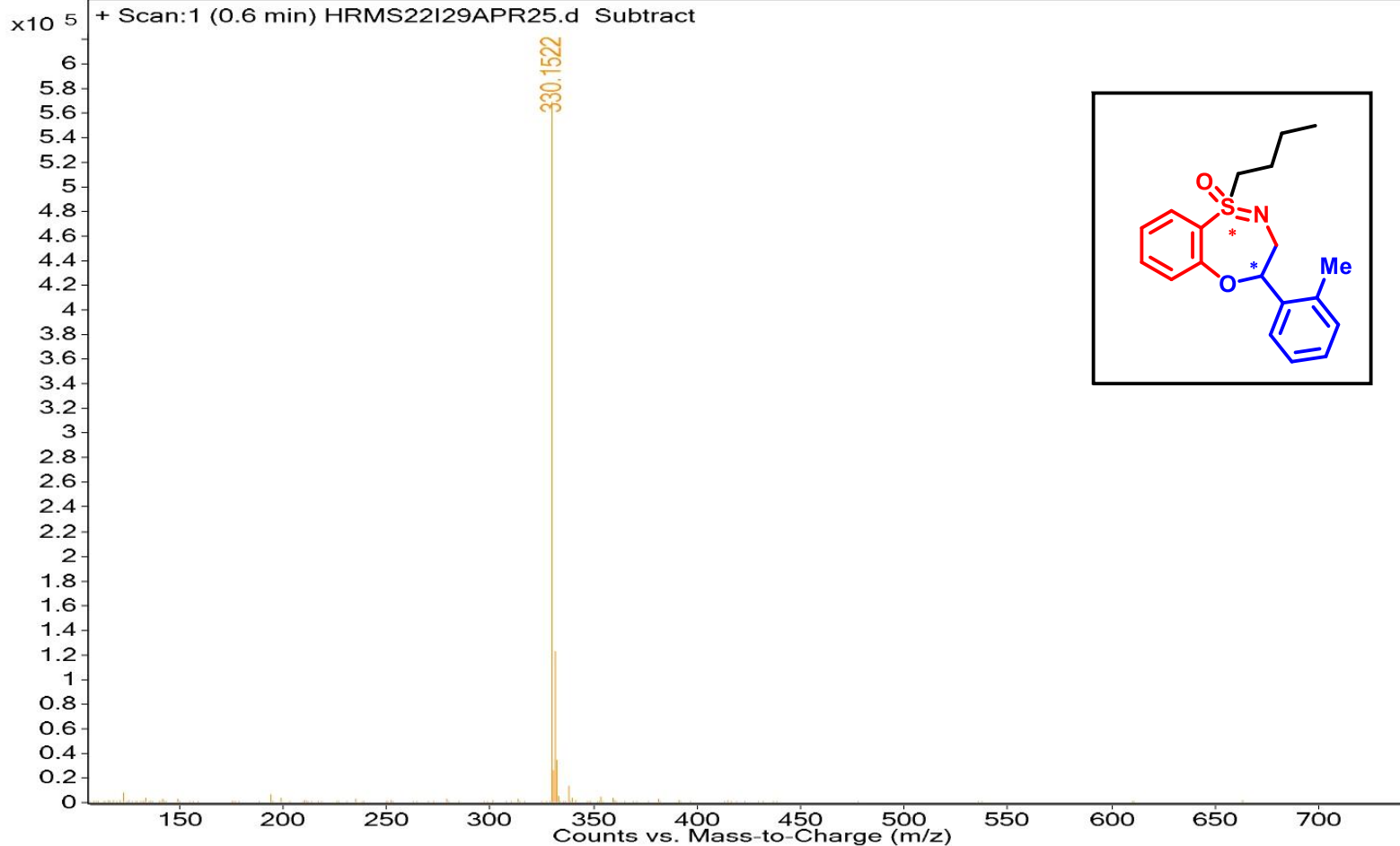


Fig S-131: HRMS report of Compound **4u**

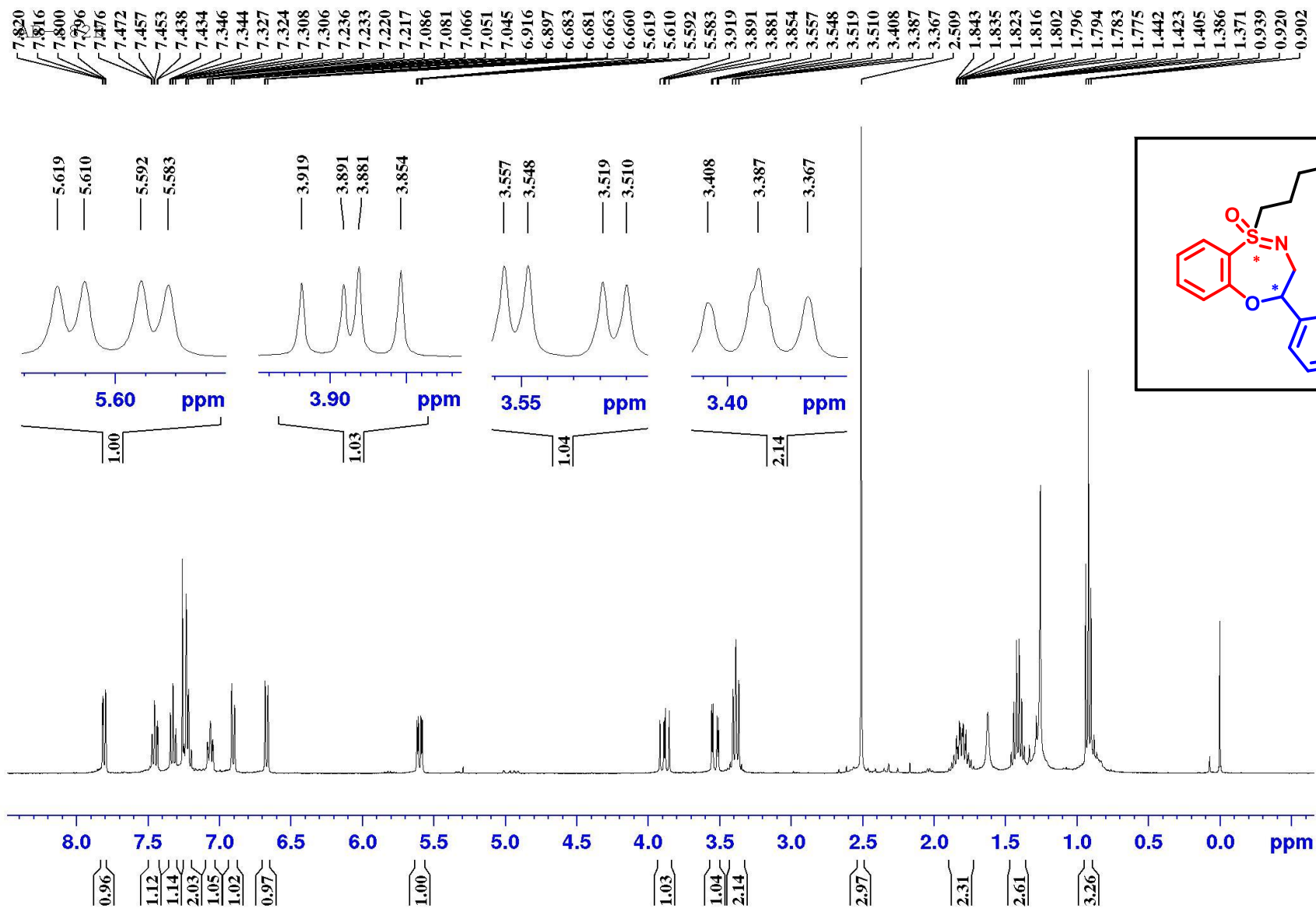


Fig S-132: ¹H NMR Spectra of Compound 4u' (400 MHz, CDCl₃)

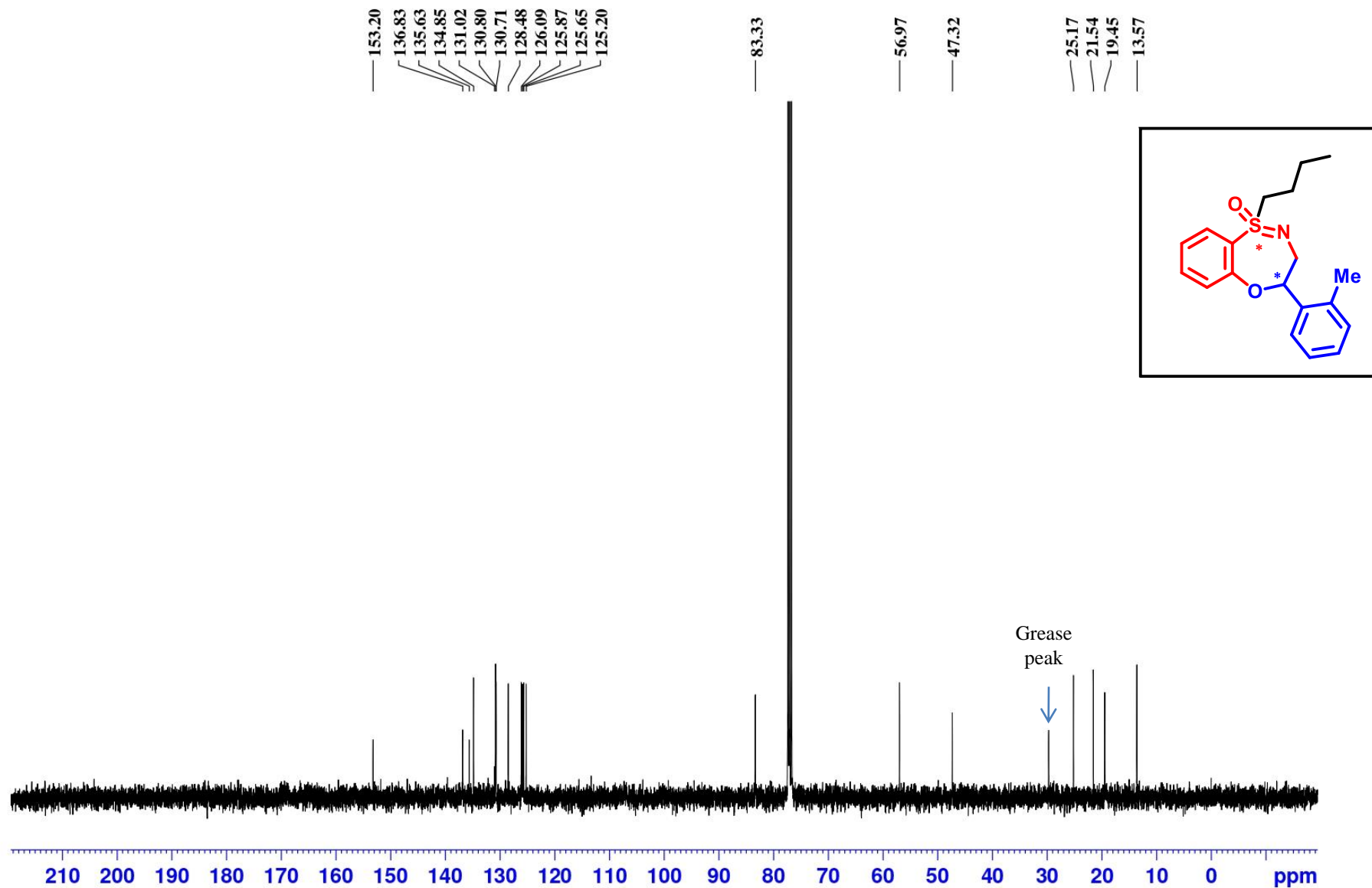


Fig S-133: ^{13}C NMR Spectra of Compound 4u' (100 MHz, CDCl_3)

Sample Name	asa1391p	Position	Vial 25	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I29APR25.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	4/29/2022 1:45:03 PM

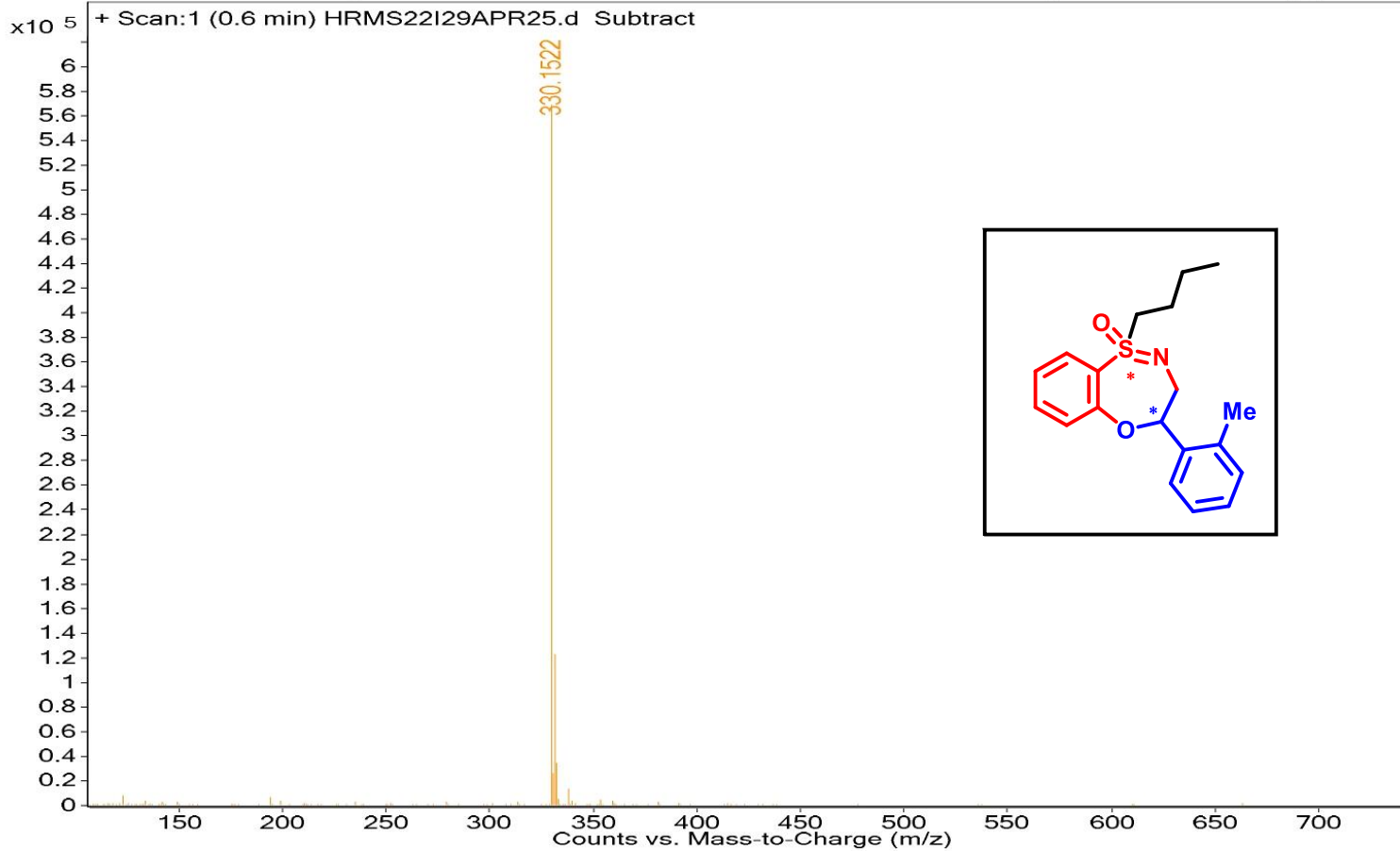


Fig S-134: HRMS report of Compound **4u'**

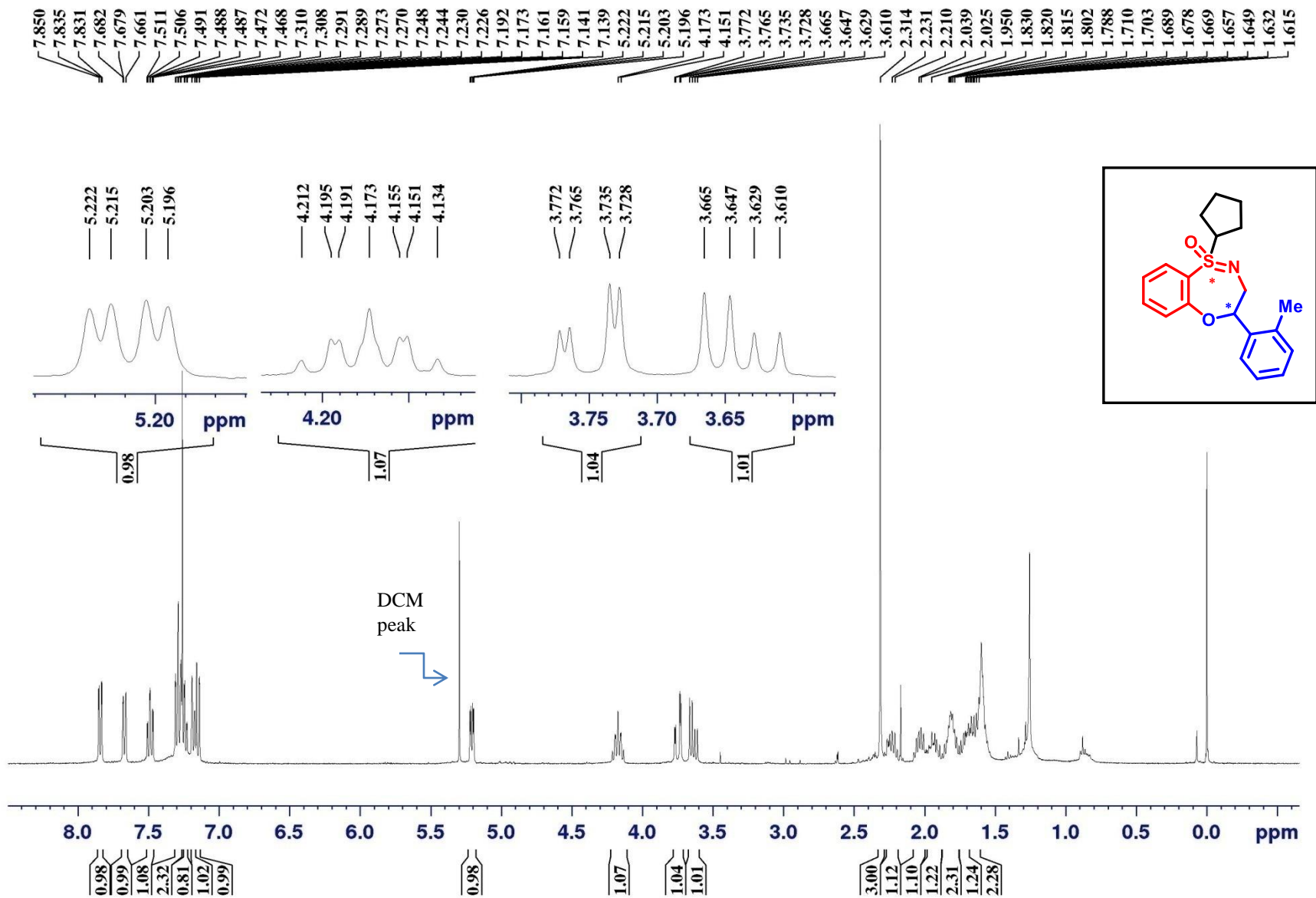


Fig S-135: ^1H NMR Spectra of Compound 4v (400 MHz, CDCl_3)

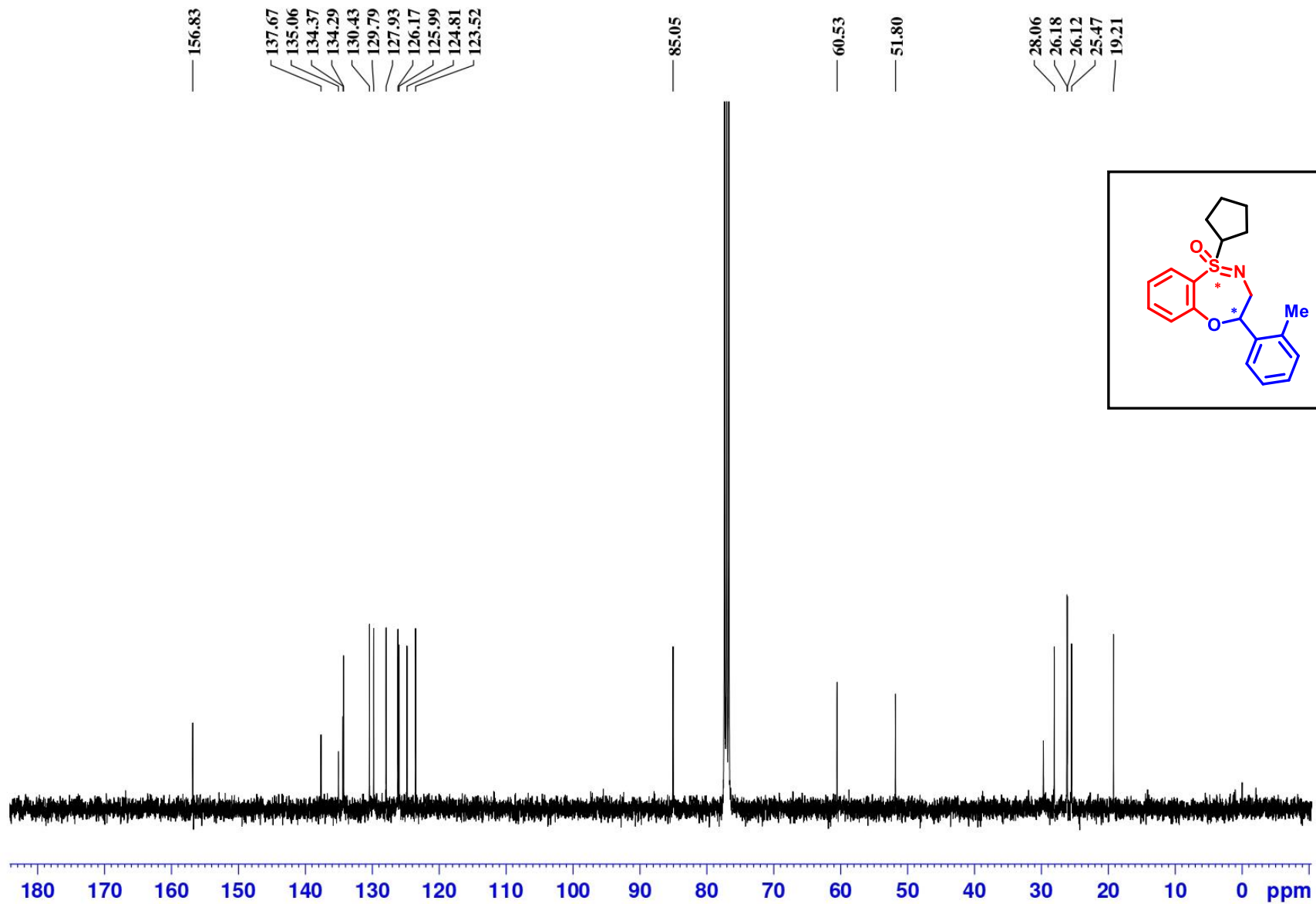


Fig S-136: ^{13}C NMR Spectra of Compound 4v (100 MHz, CDCl_3)

Qualitative Compound Report

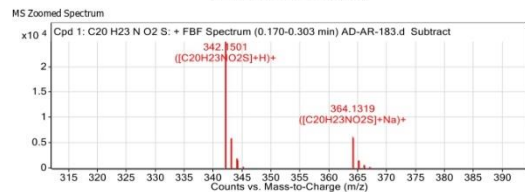
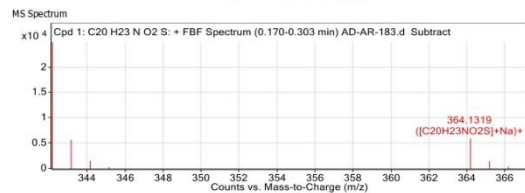
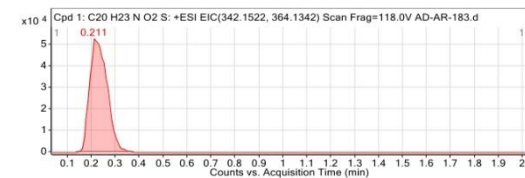
Data File	AD-AR-183.d	Sample Name	AD-AR-183
Sample Type	Sample	Position	Vial 13
Instrument Name	Instrument 1	User Name	
Acq Method	Direct Mass-2017.m	Acquired Time	08-09-2021 16:00:53
IRN Calibration Status	Some Ions Missed	DA Method	Default.m

Sample Group		Info.
Acquisition SW	6200 series TOF/6500 series	
Version	Q.TOF B.05.00 (B5042.0)	

Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C20 H23 N O2 S	0.211	341.1427	24978	C20 H23 N O2 S	341.1449	-6.61	C20 H23 N O2 S	C20 H23 N O2 S

Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C20 H23 N O2 S	342.1501	0.211	Find By Formula	341.1427



MS Spectrum Peak List

m/z	z	Abund	Formula	Ion
342.1501	1	24977.58	C20H23NO2S	(M+H)+
343.1535	1	5810.22	C20H23NO2S	(M+H)+
344.149	1	1703.95	C20H23NO2S	(M+H)+
345.1499	1	410.64	C20H23NO2S	(M+H)+
364.1319	1	6076.95	C20H23NO2S	(M+Na)+
365.1342	1	1521.42	C20H23NO2S	(M+Na)+
366.1299	1	484.23	C20H23NO2S	(M+Na)+
367.1281	1	67.92	C20H23NO2S	(M+Na)+

--- End Of Report ---

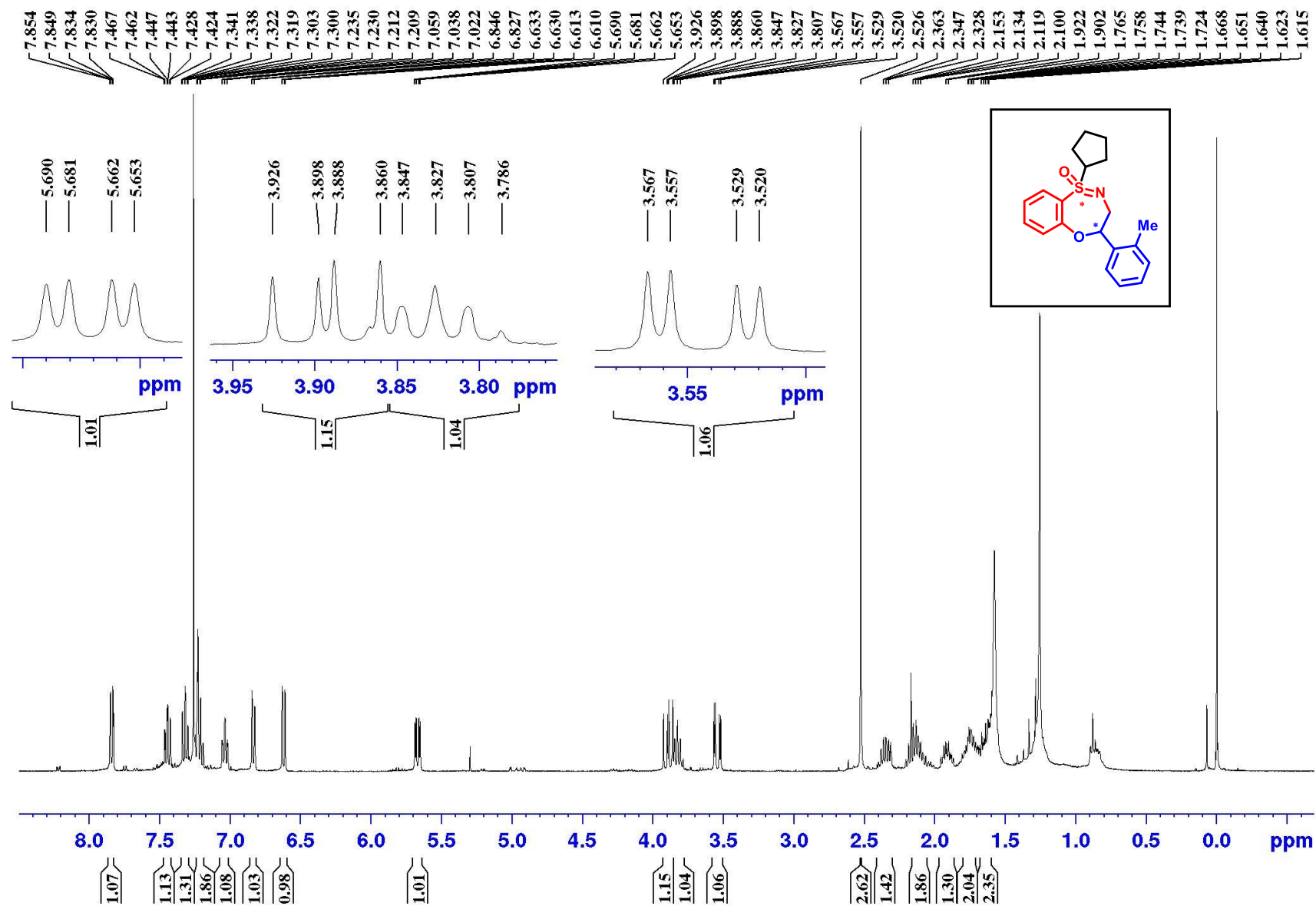


Fig S-138: ^1H NMR Spectra of Compound **4v' (400 MHz, CDCl_3)**

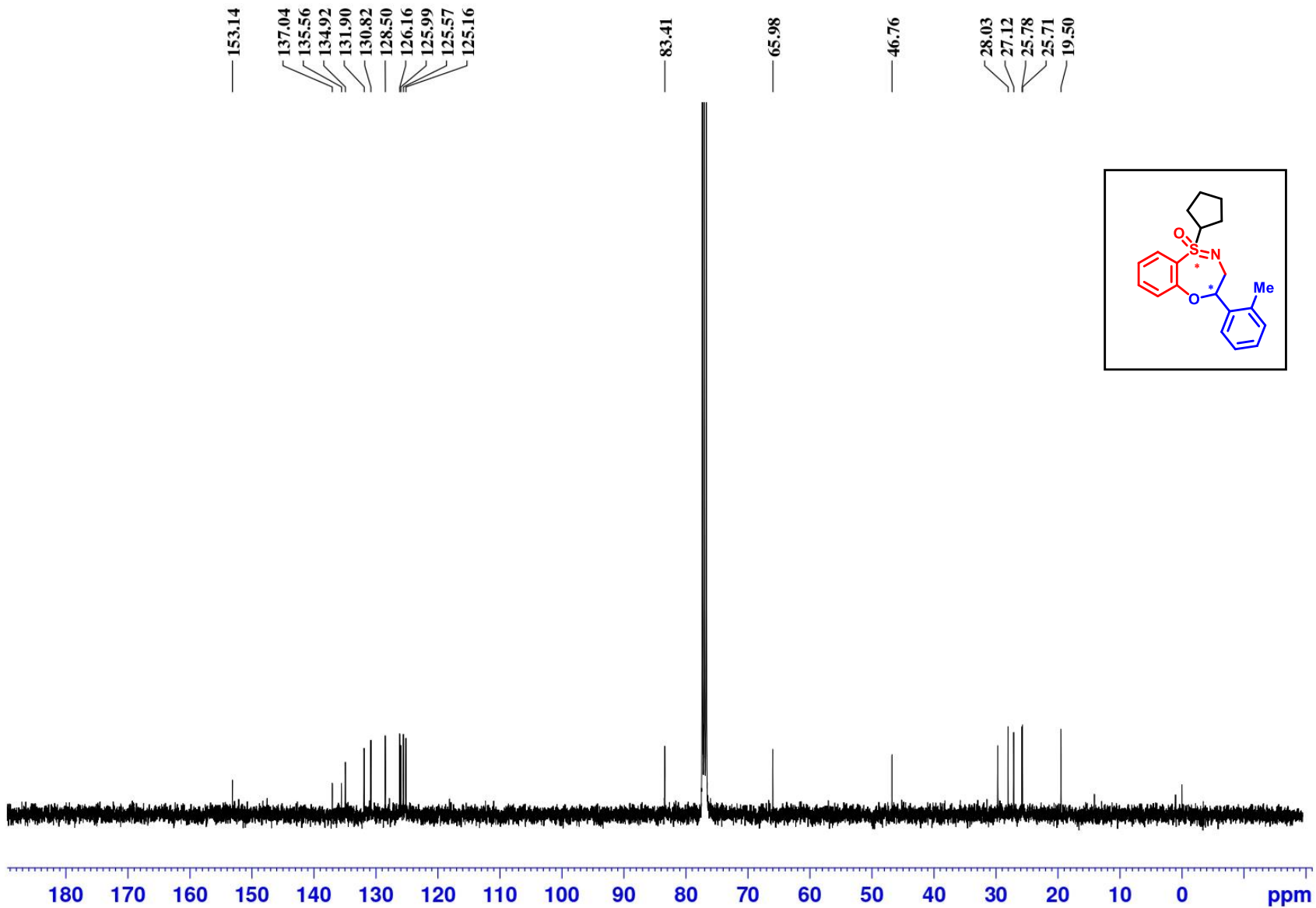


Fig S-139: ^{13}C NMR Spectra of Compound **4v'** (100 MHz, CDCl_3)

Sample Name	4v'	Position	Vial 36	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I02AUG35.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	8/2/2022 1:34:21 PM

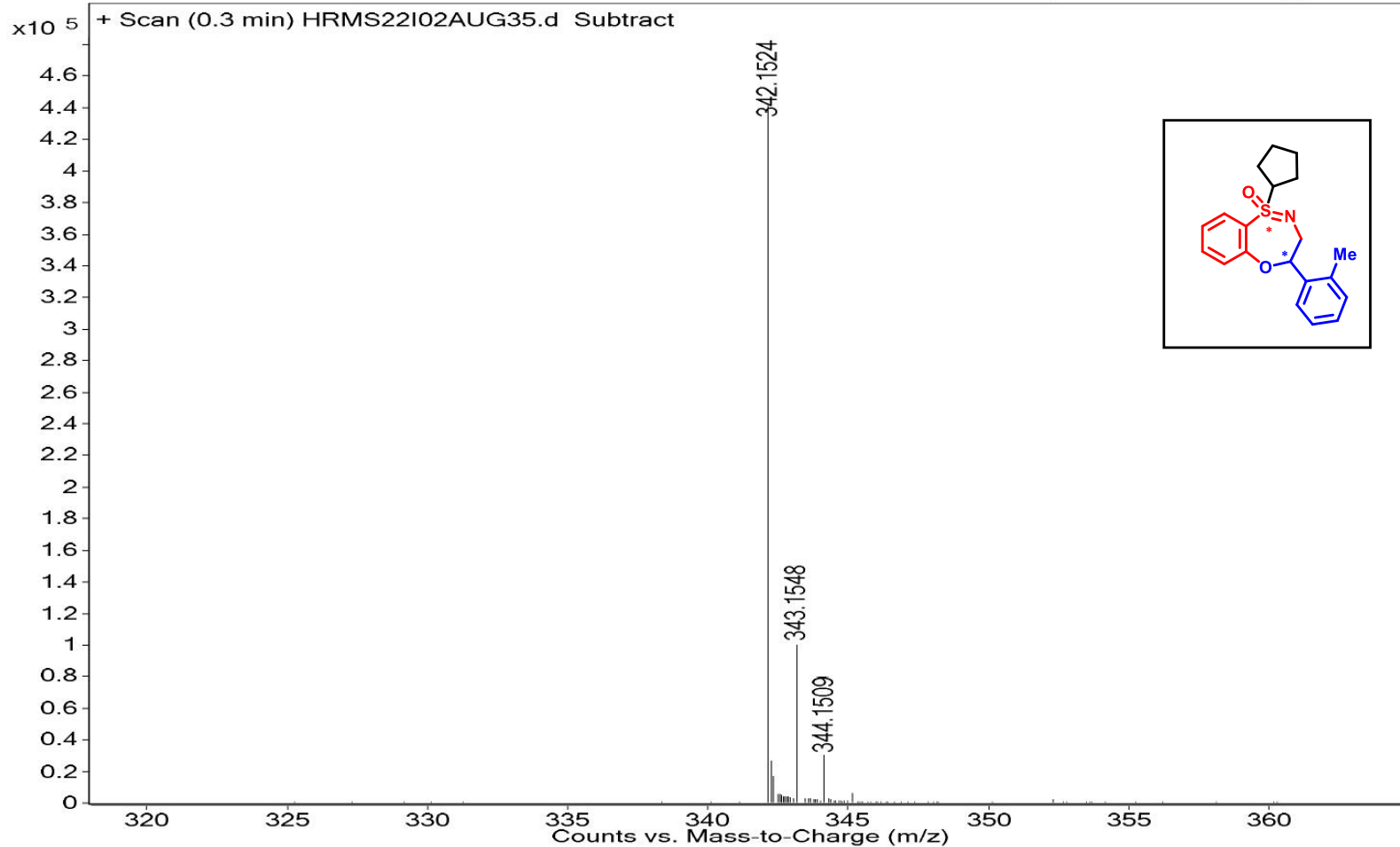


Fig S-140: HRMS report of Compound 4v'

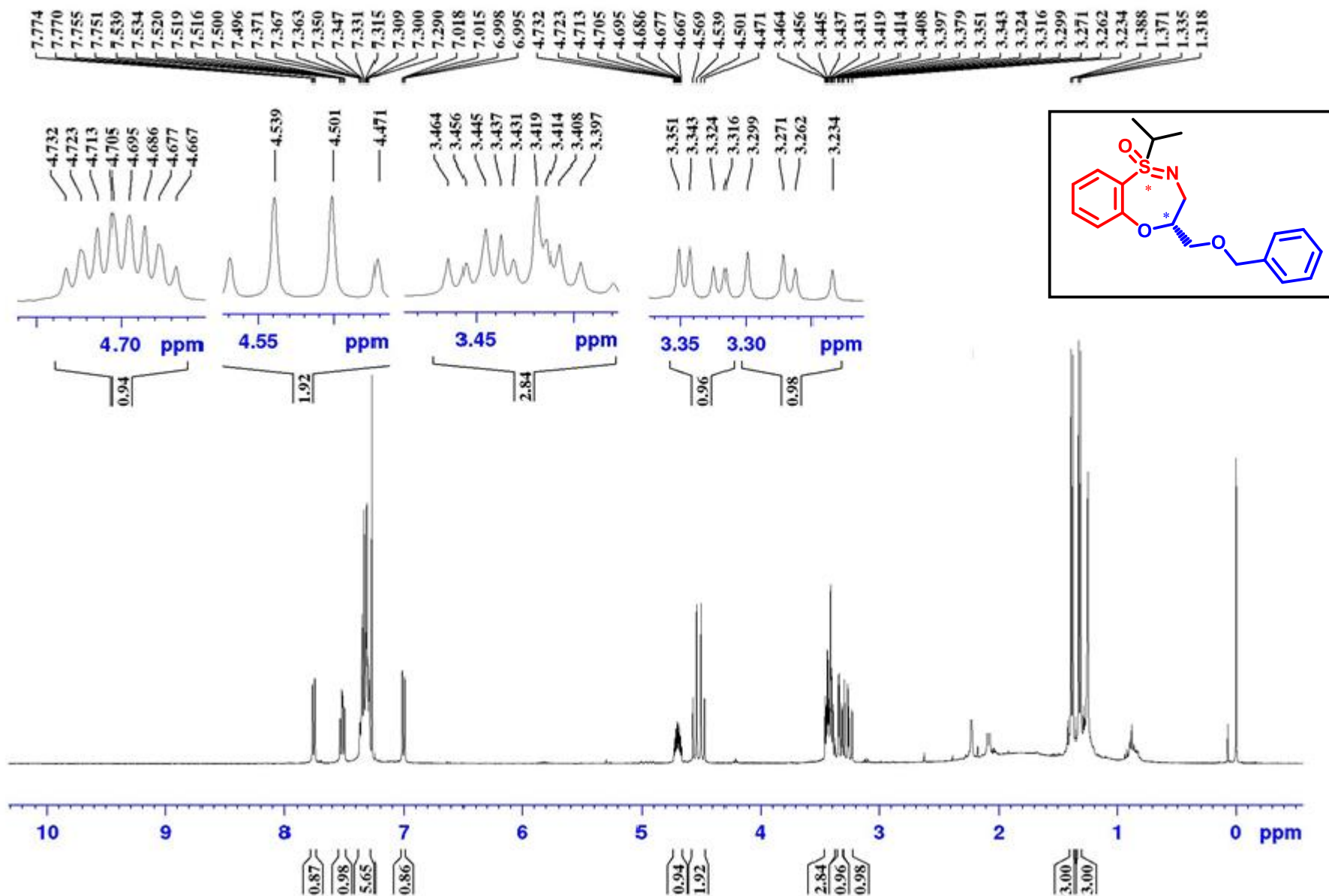


Fig S-141: ¹H NMR Spectra of Compound 4w (400 MHz, CDCl₃)

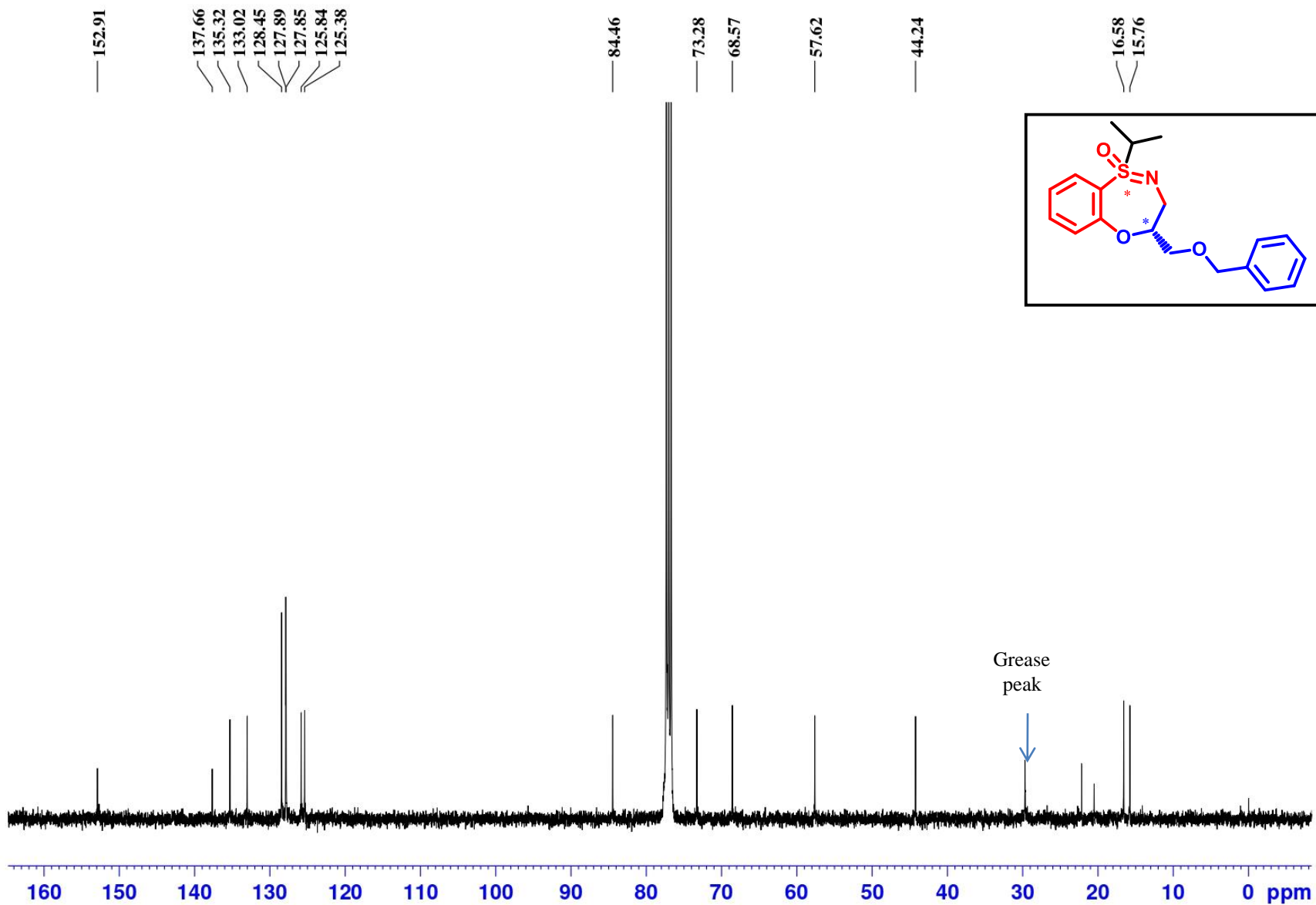


Fig S-142: ^{13}C NMR Spectra of Compound **4w** (100 MHz, CDCl_3)

Sample Name	4w	Position	Vial 37	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I02AUG36.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	8/2/2022 1:37:30 PM

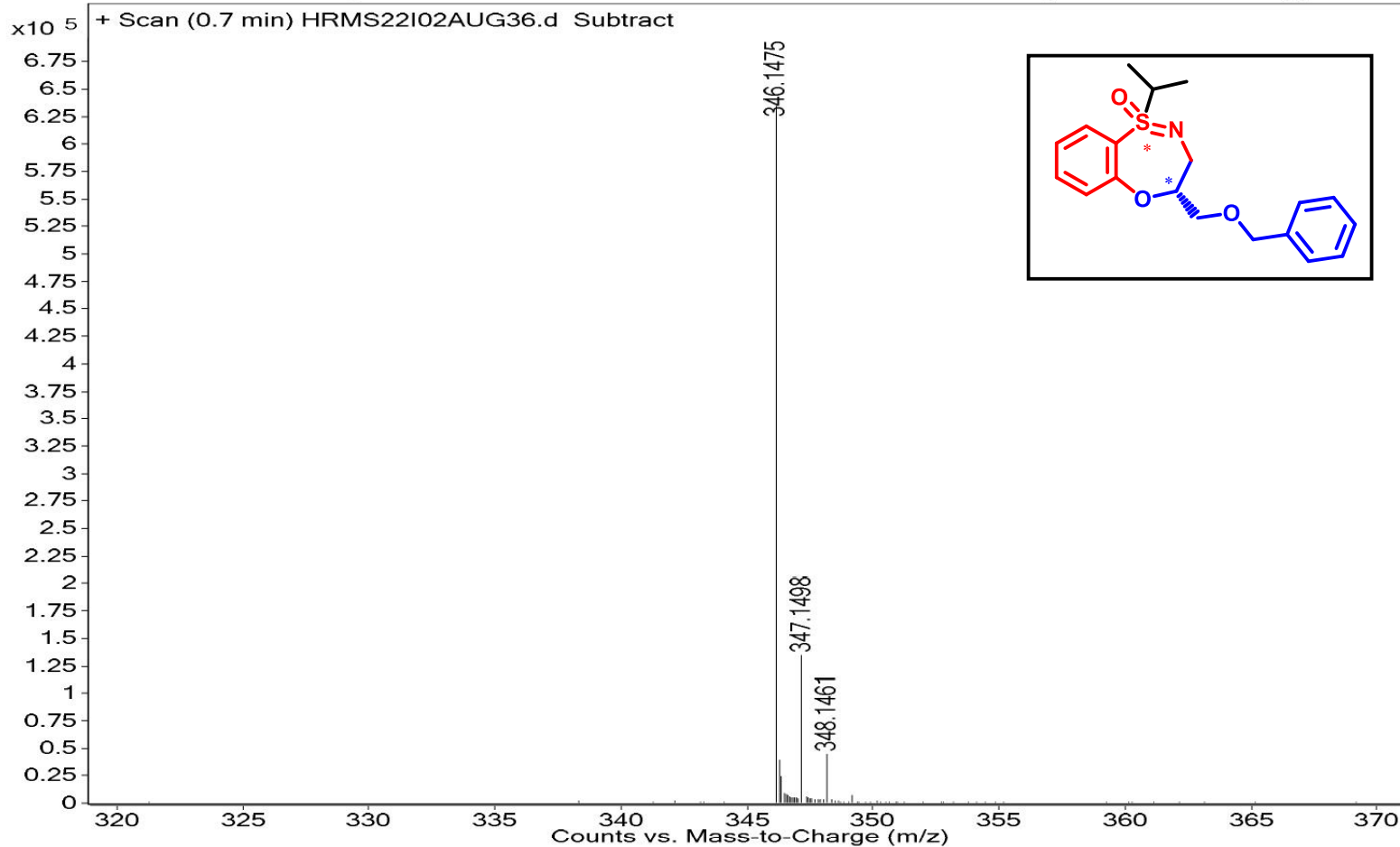


Fig S-143: HRMS report of Compound **4w**

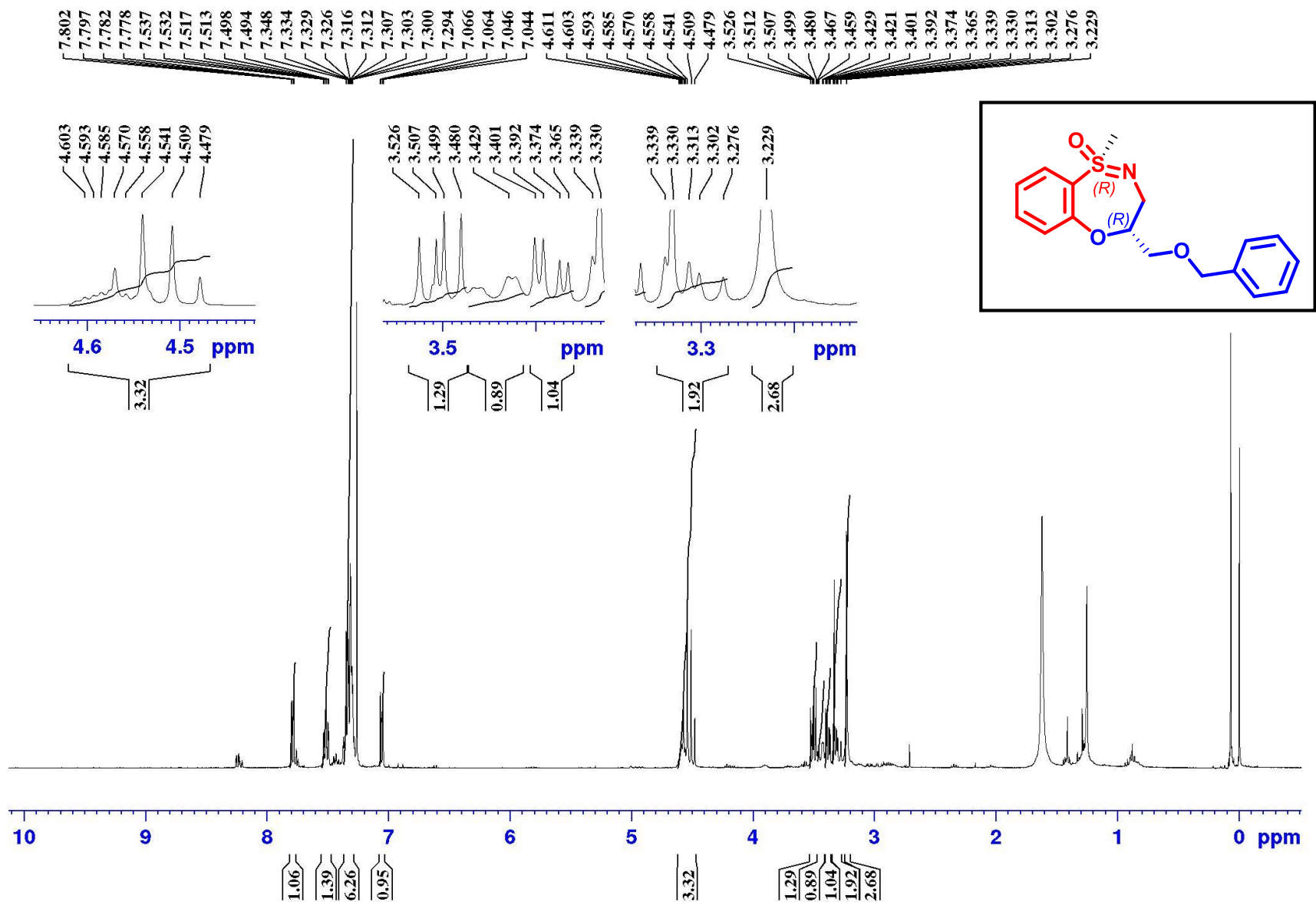


Fig S-144: ^1H NMR Spectra of Compound (*R,R*)-4x (400 MHz, CDCl_3)

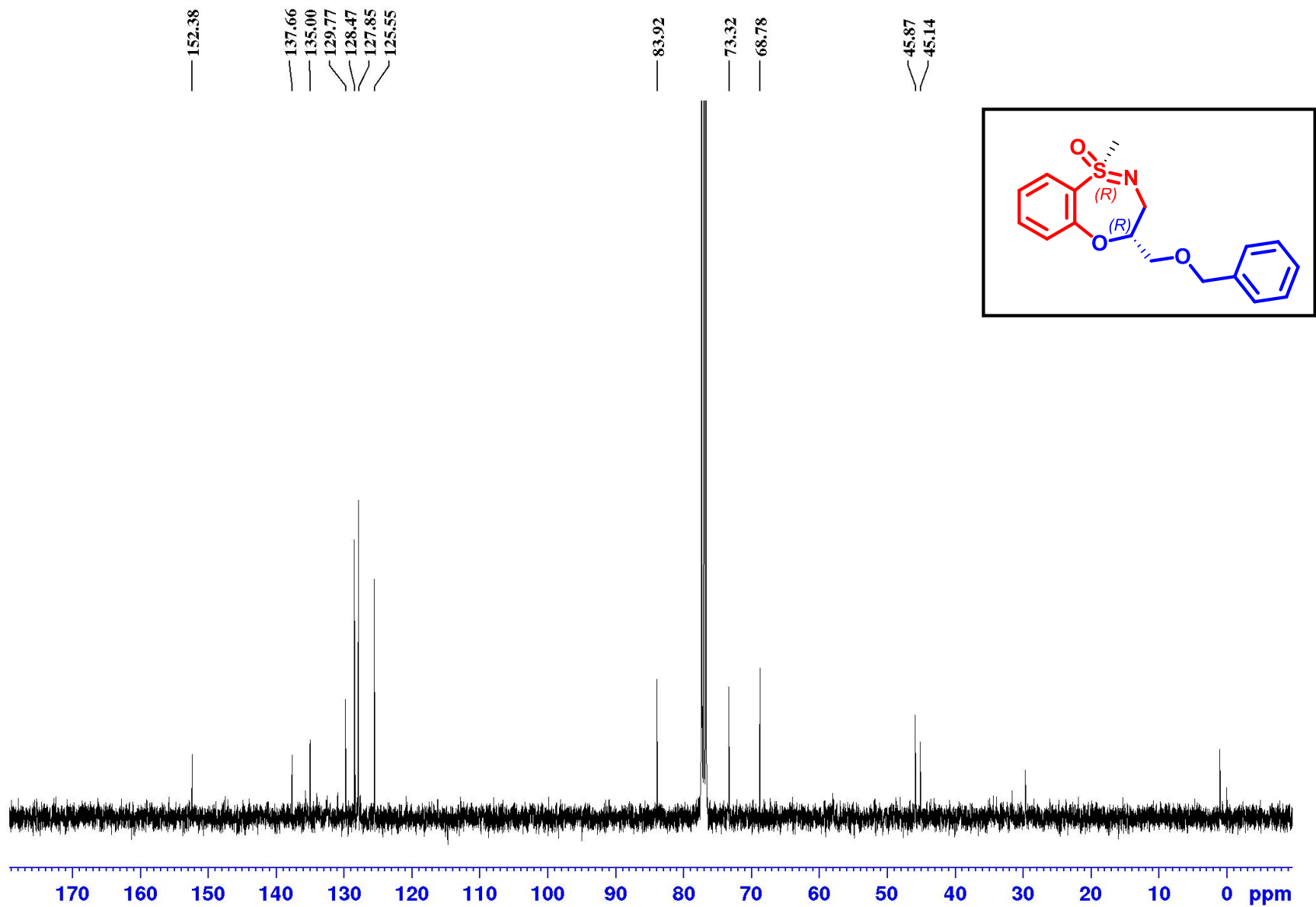


Fig S-145: ^{13}C NMR Spectra of Compound *(R,R)*-4x (100 MHz, CDCl_3)

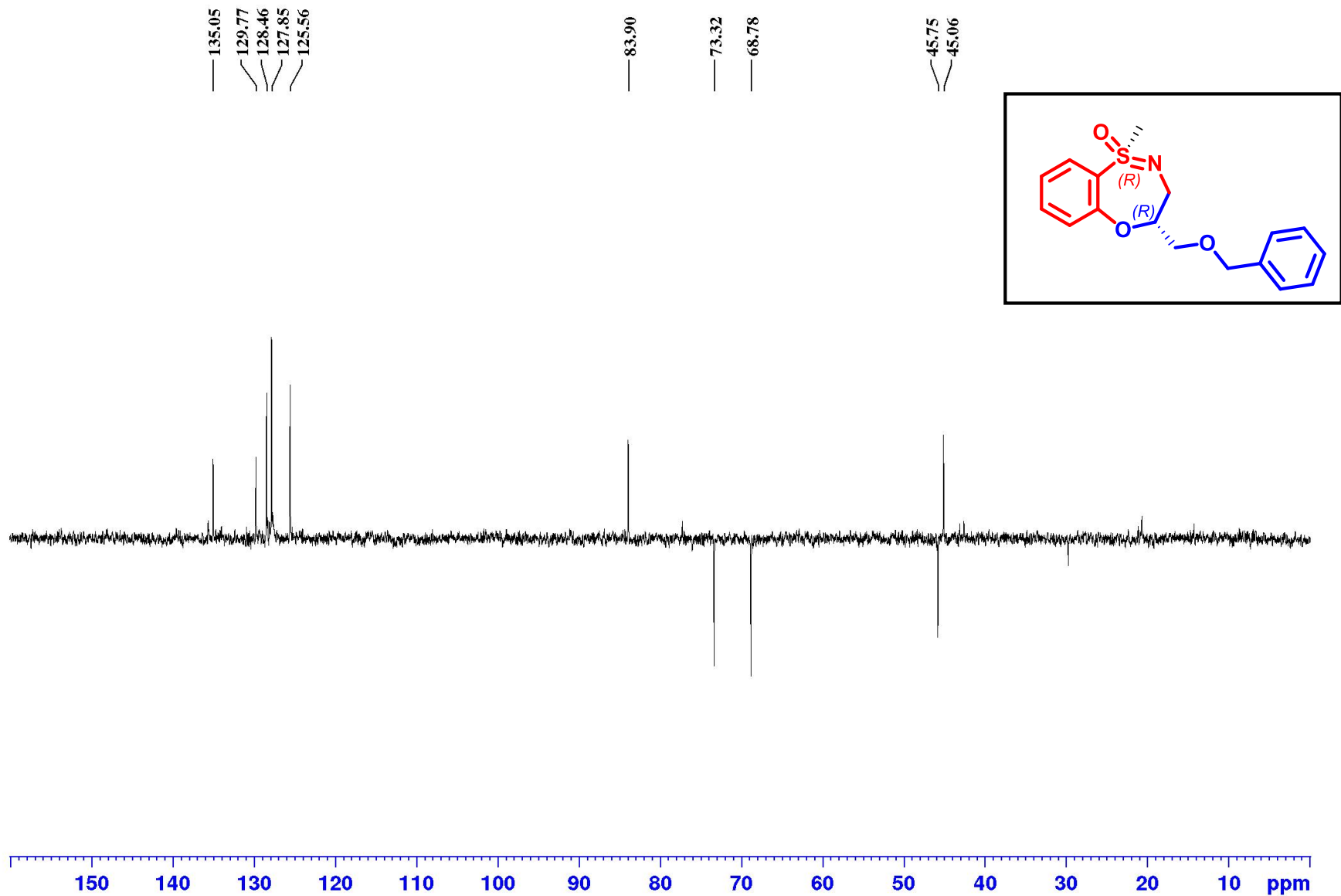


Fig S-146: DEPT-135 Spectra of Compound *(R,R)*-4x (100 MHz, CDCl₃)

Sample Name	AB-195A	Position	Vial 12	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I04MAY12.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	5/4/2022 12:02:33 PM

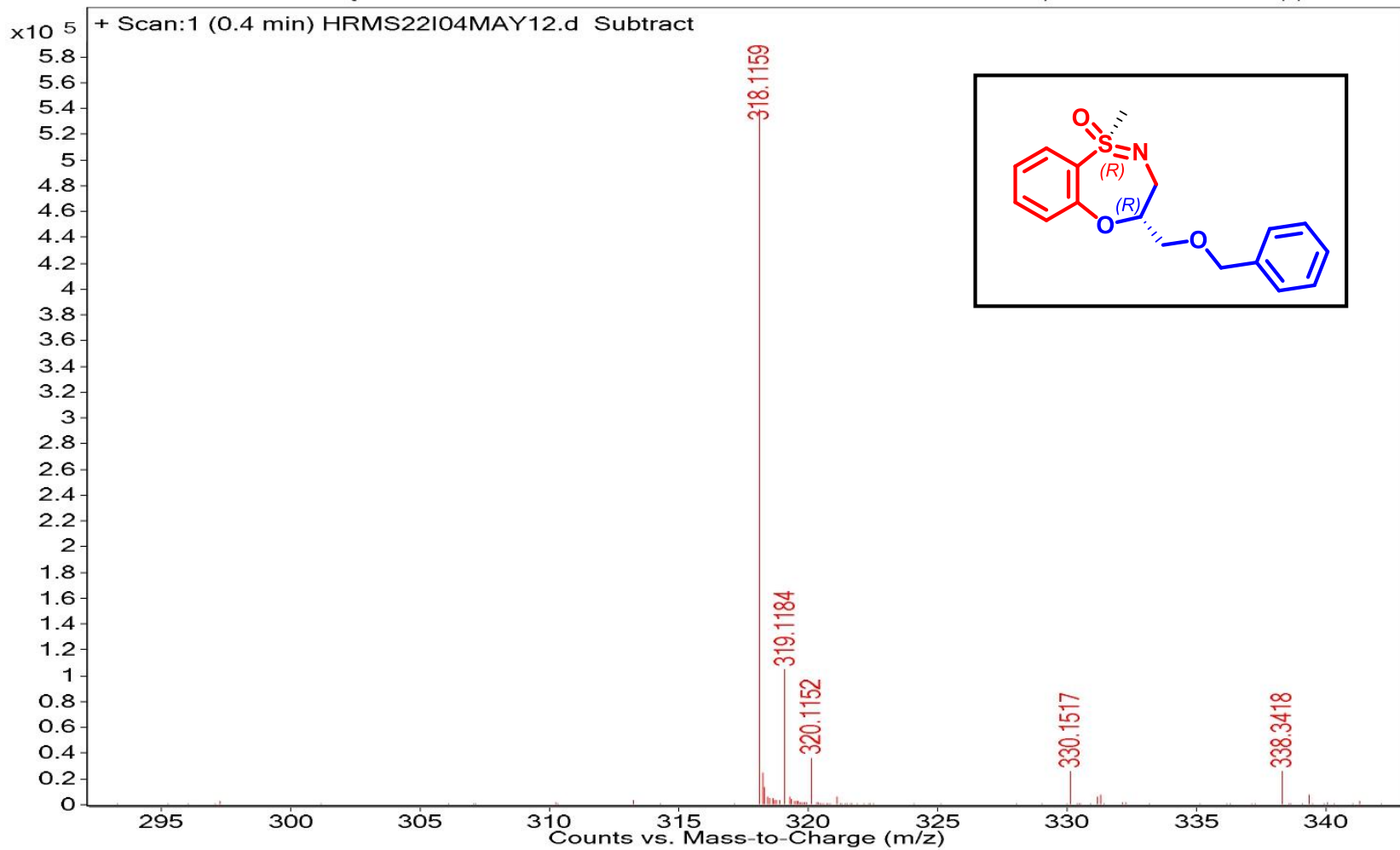


Fig S-147: HRMS report of Compound (*R,R*)-4x

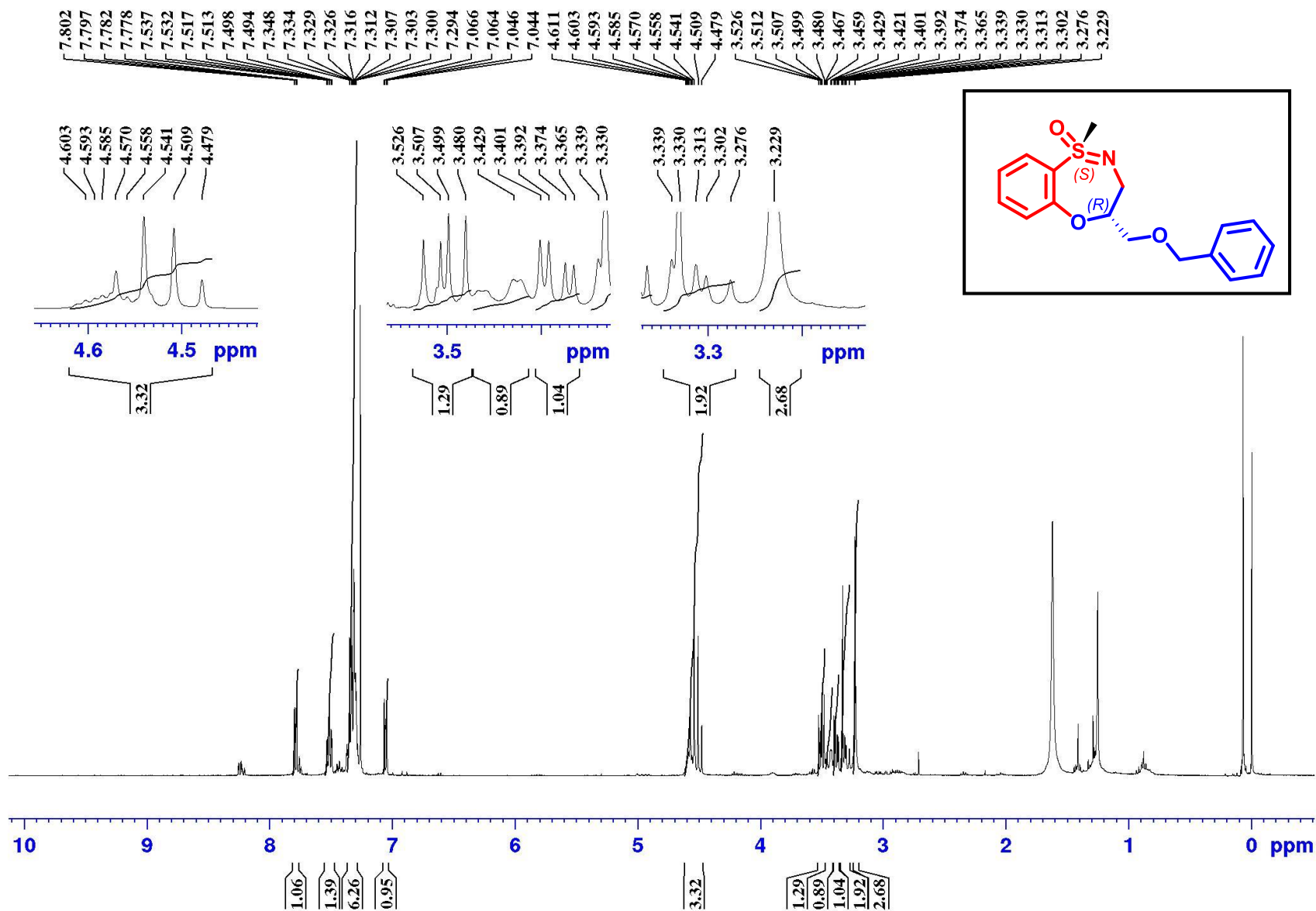


Fig S-148: ^1H NMR Spectra of Compound (*R,S*)- 4x'(400 MHz, CDCl_3)

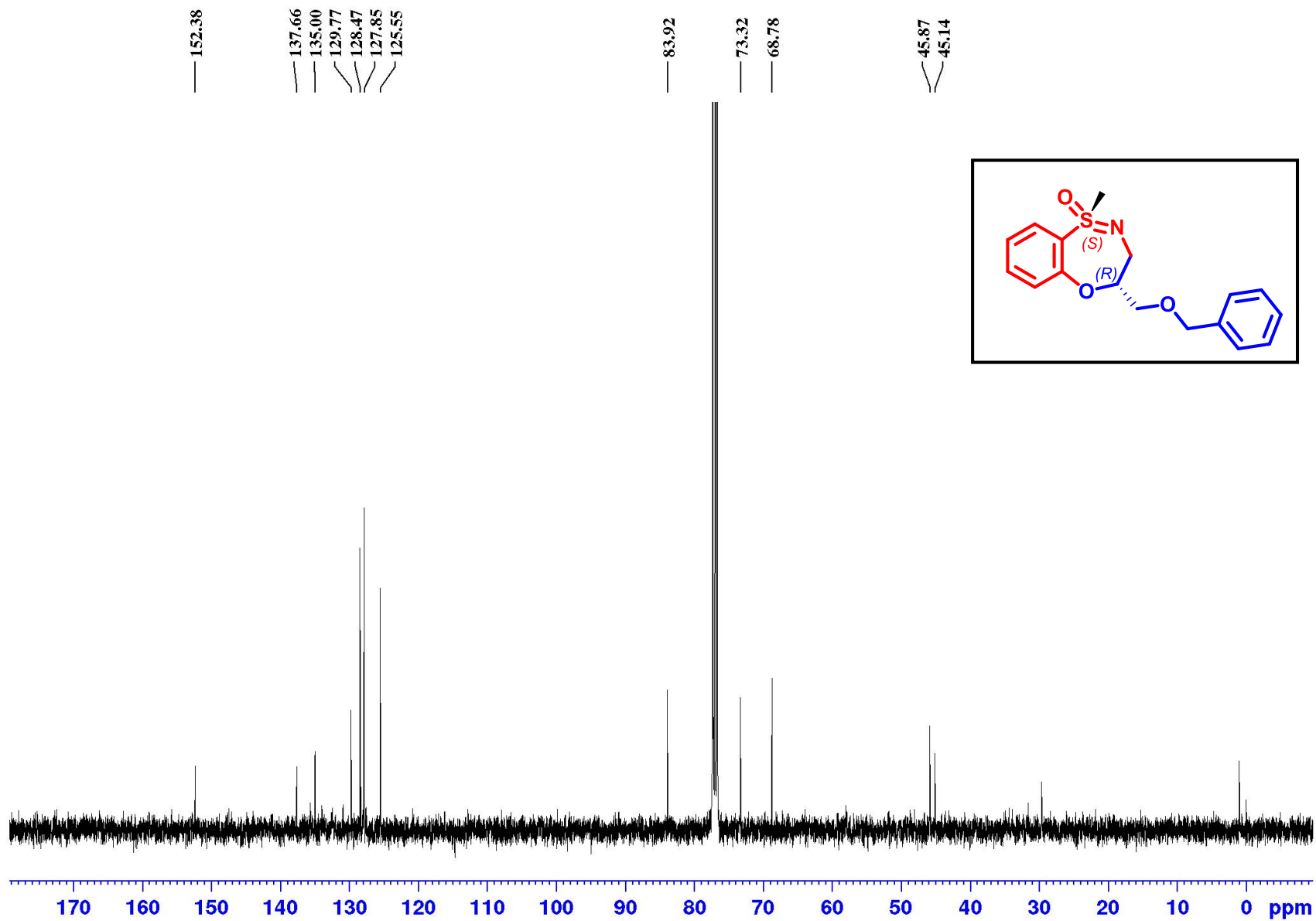


Fig S-149: ^{13}C NMR Spectra of Compound (R,S) -4x' (100 MHz, CDCl_3)

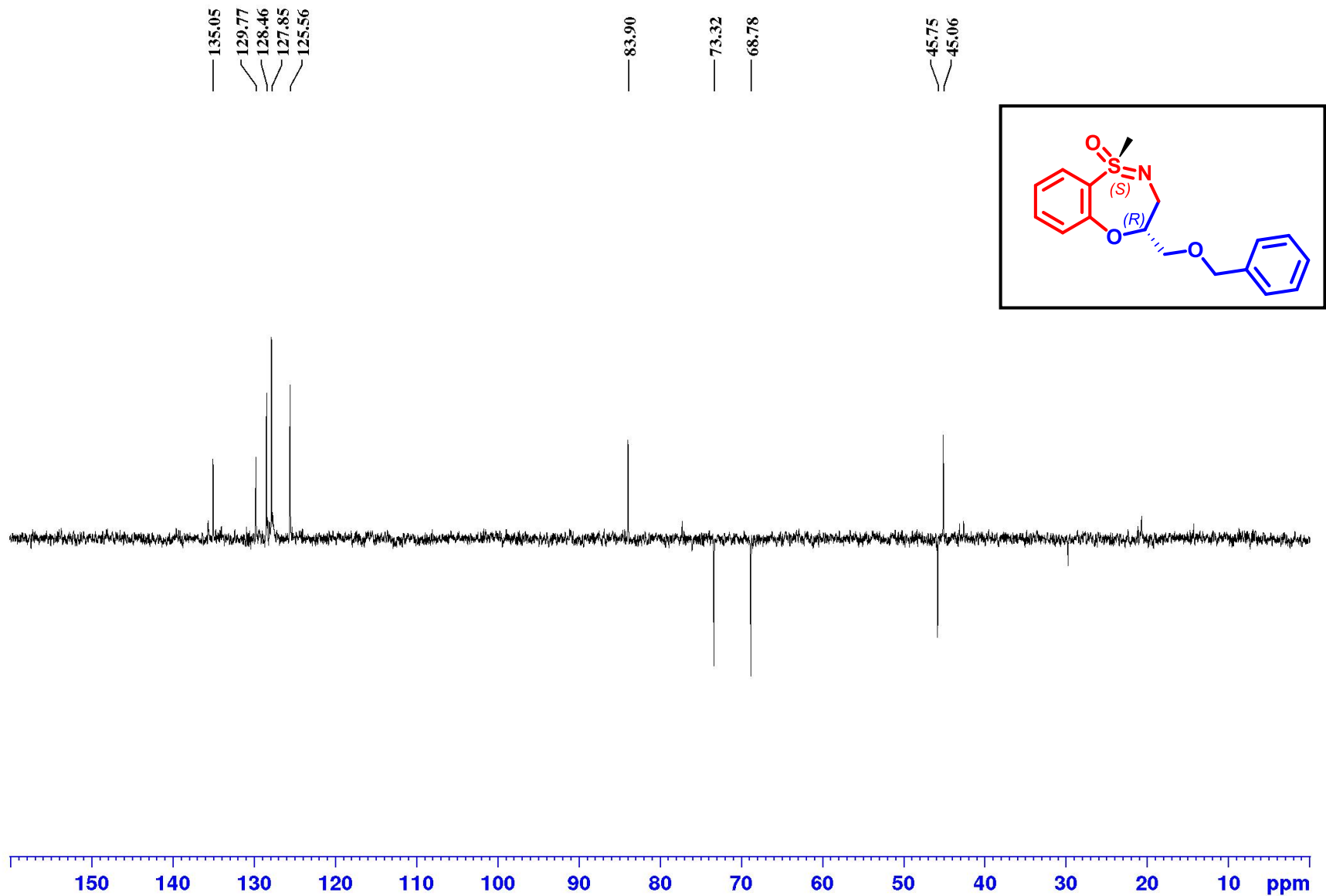


Fig S-150: DEPT-135 Spectra of Compound (*R,S*)- 4x' (100 MHz, CDCl₃)

Sample Name	4x'	Position	Vial 38	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I02AUG37.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	8/2/2022 1:40:39 PM

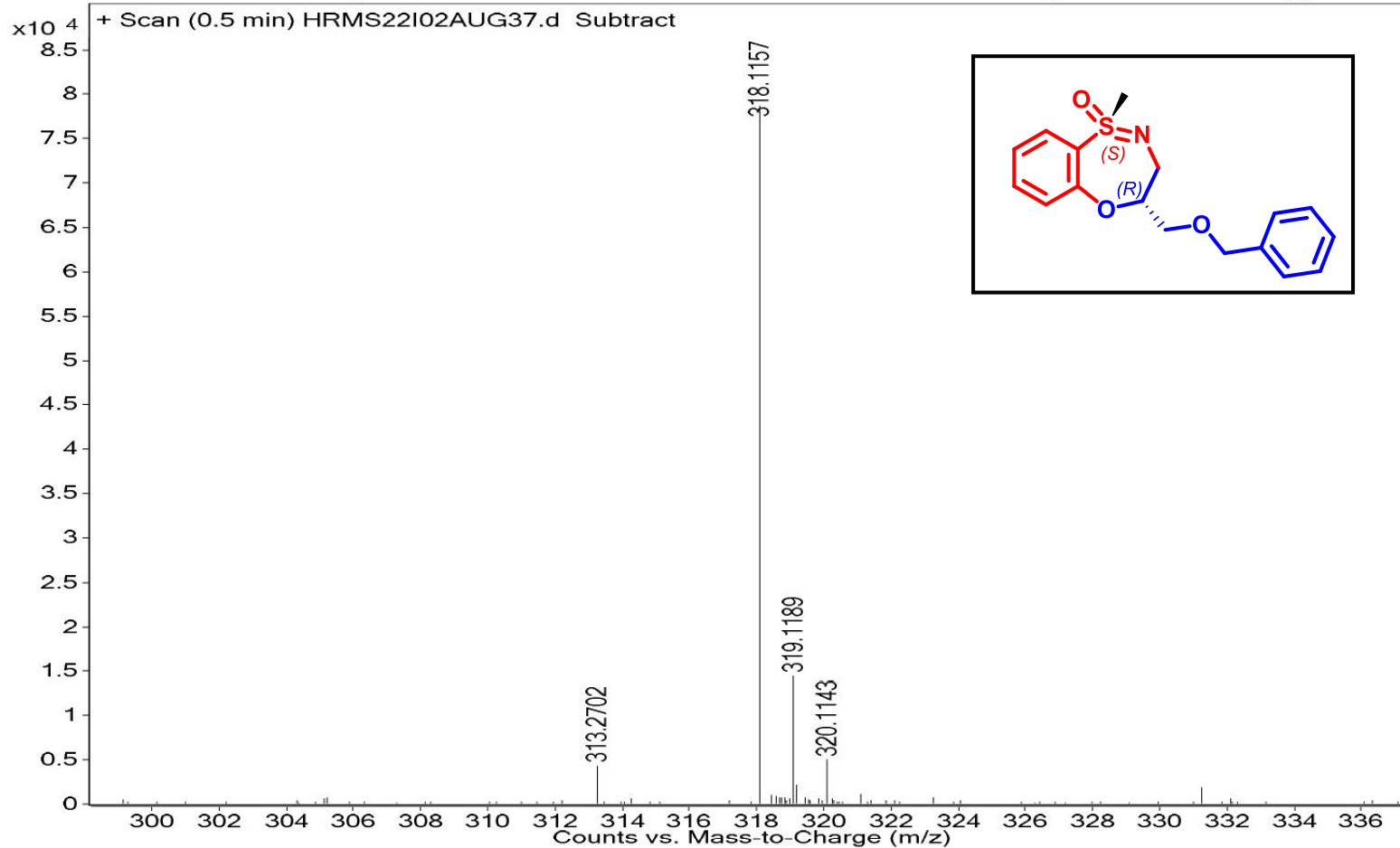


Fig S-151: HRMS report of Compound 4x'

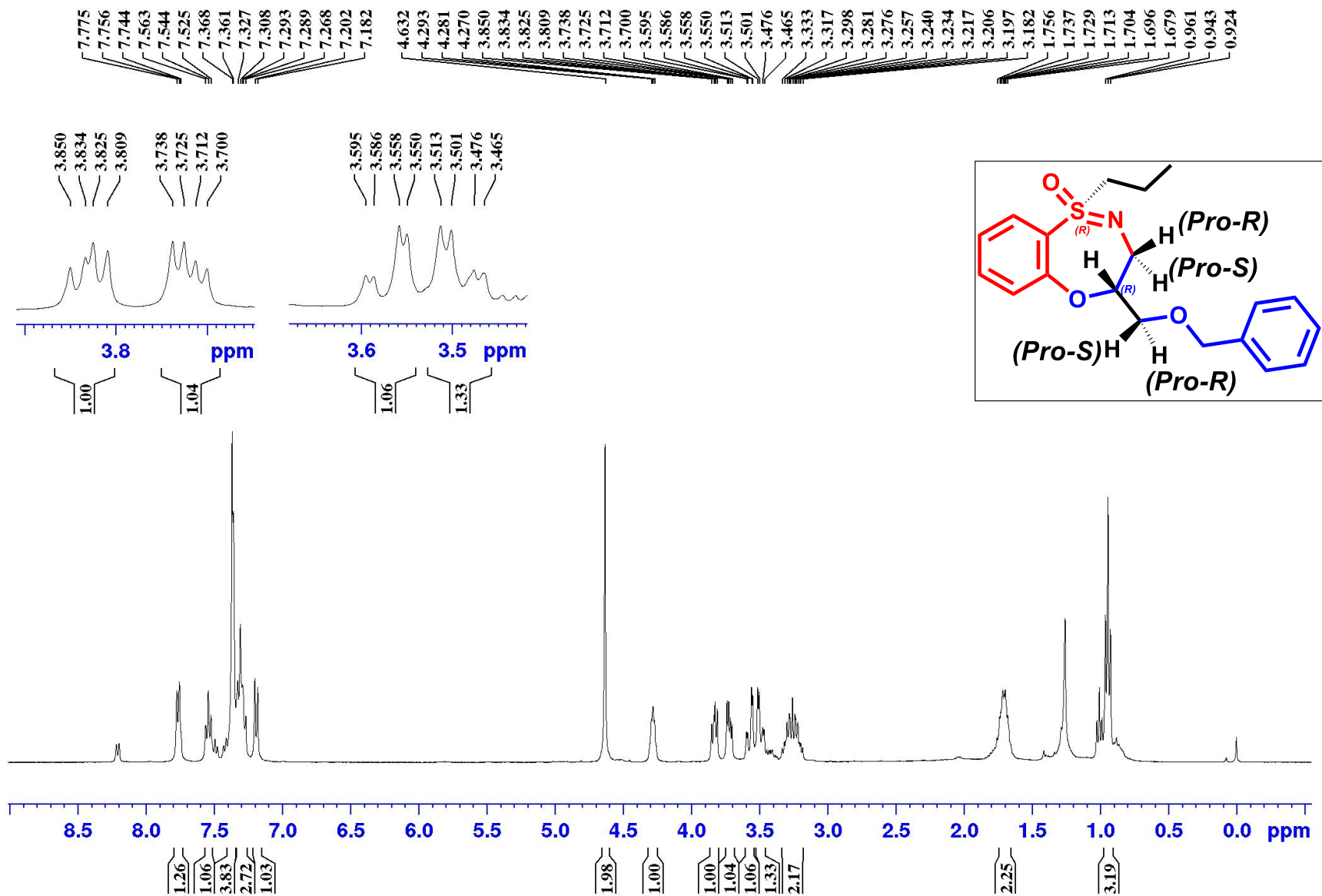


Fig S-152: ¹H NMR Spectra of Compound *(R,R)*-4z (400 MHz, CDCl₃)

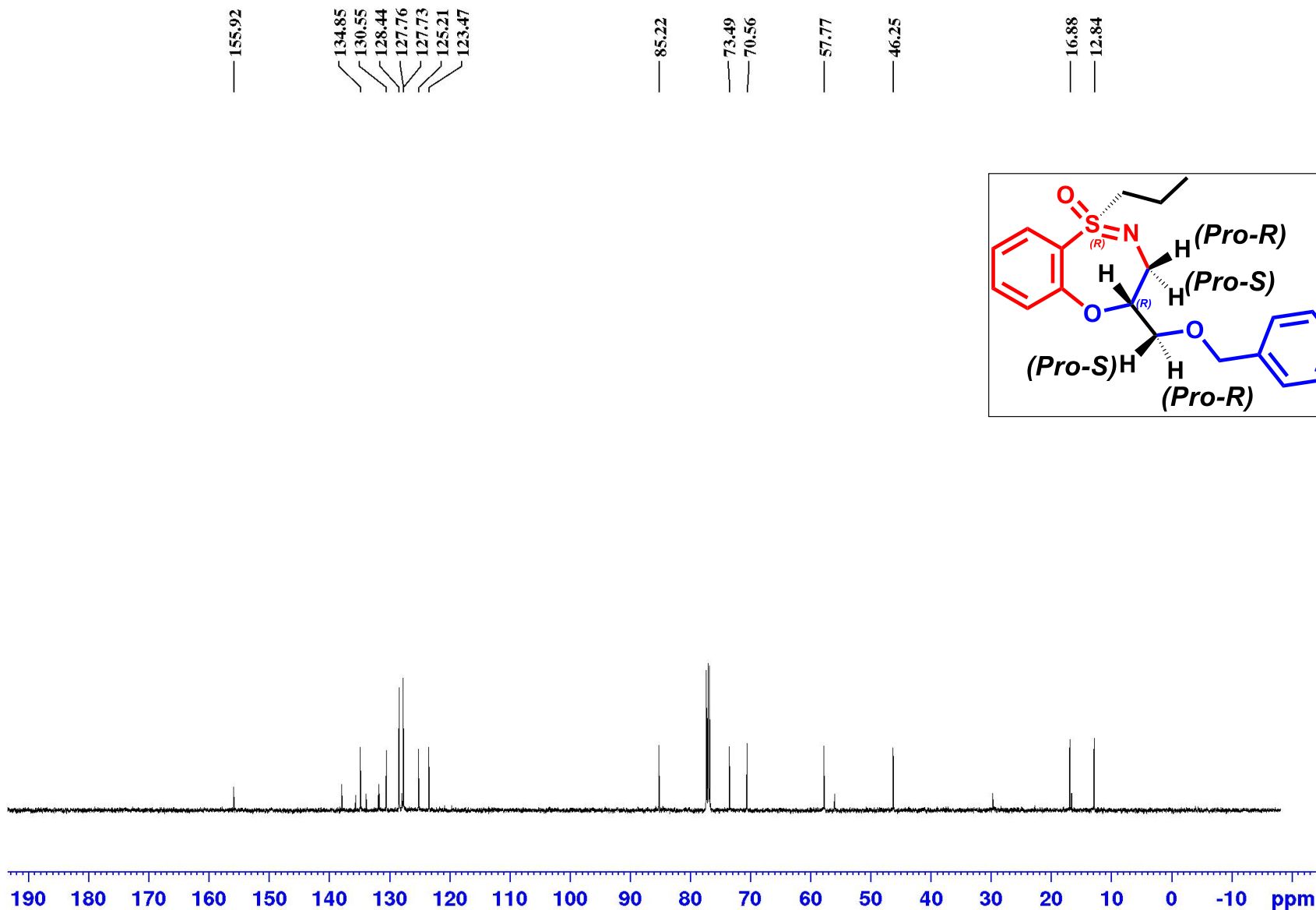


Fig S-153: ^{13}C NMR Spectra of Compound **(R,R)-4z** (125 MHz, CDCl_3)

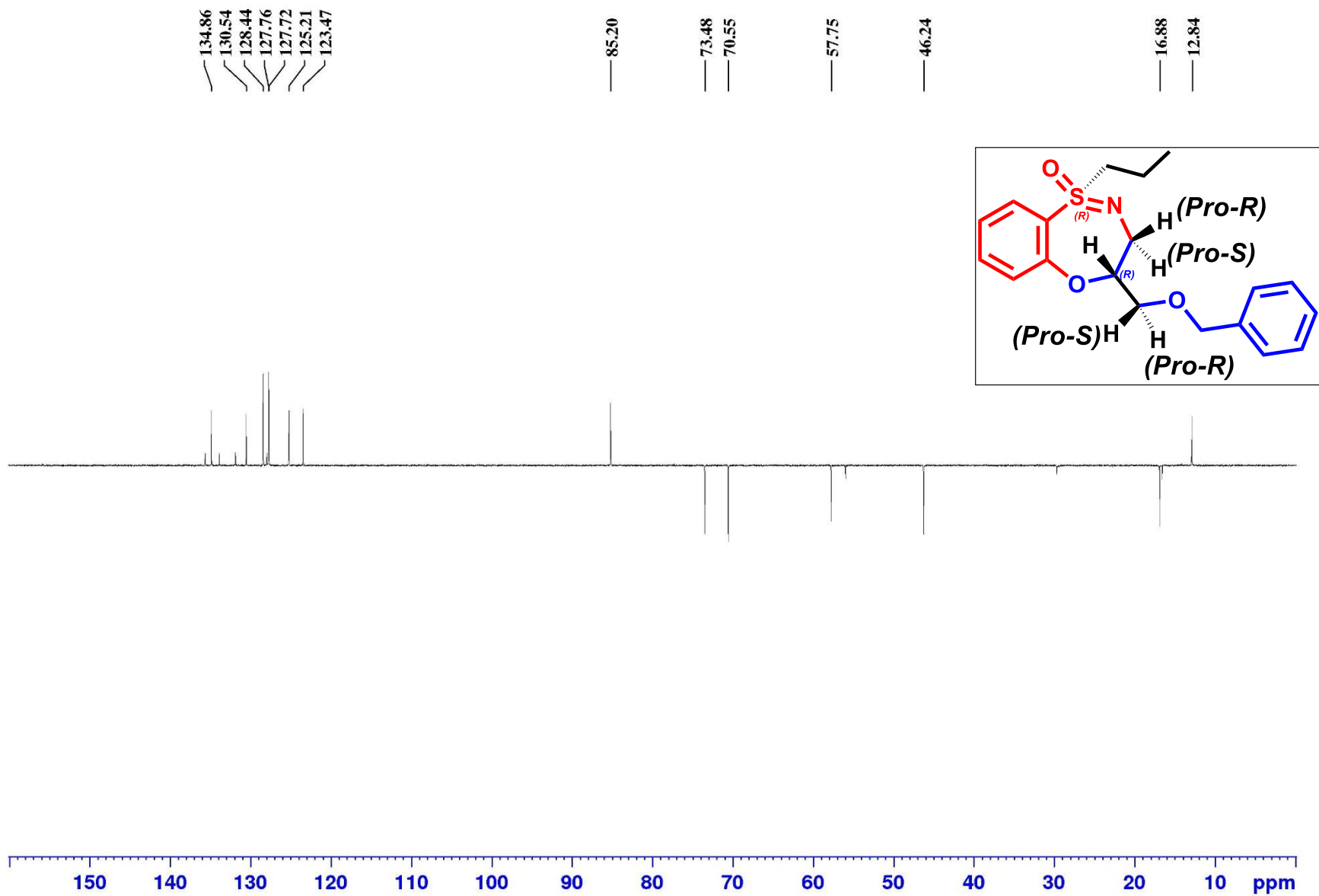


Fig S-154: DEPT-135 Spectra of Compound **(R,R)-4z** (100 MHz, CDCl_3)

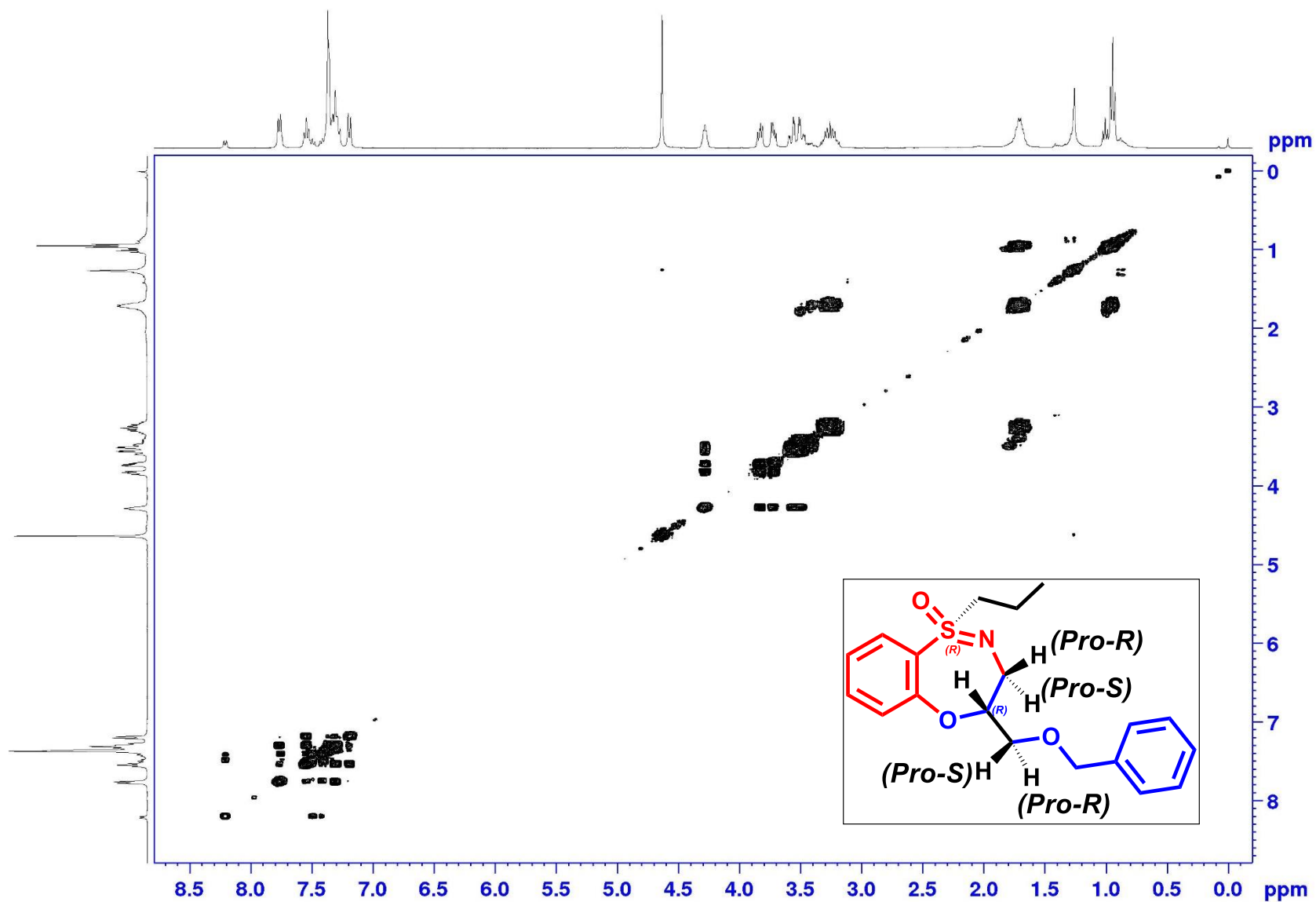


Fig S-155: COSY Spectra of Compound (*R,R*)-4z (400 MHz, CDCl₃)

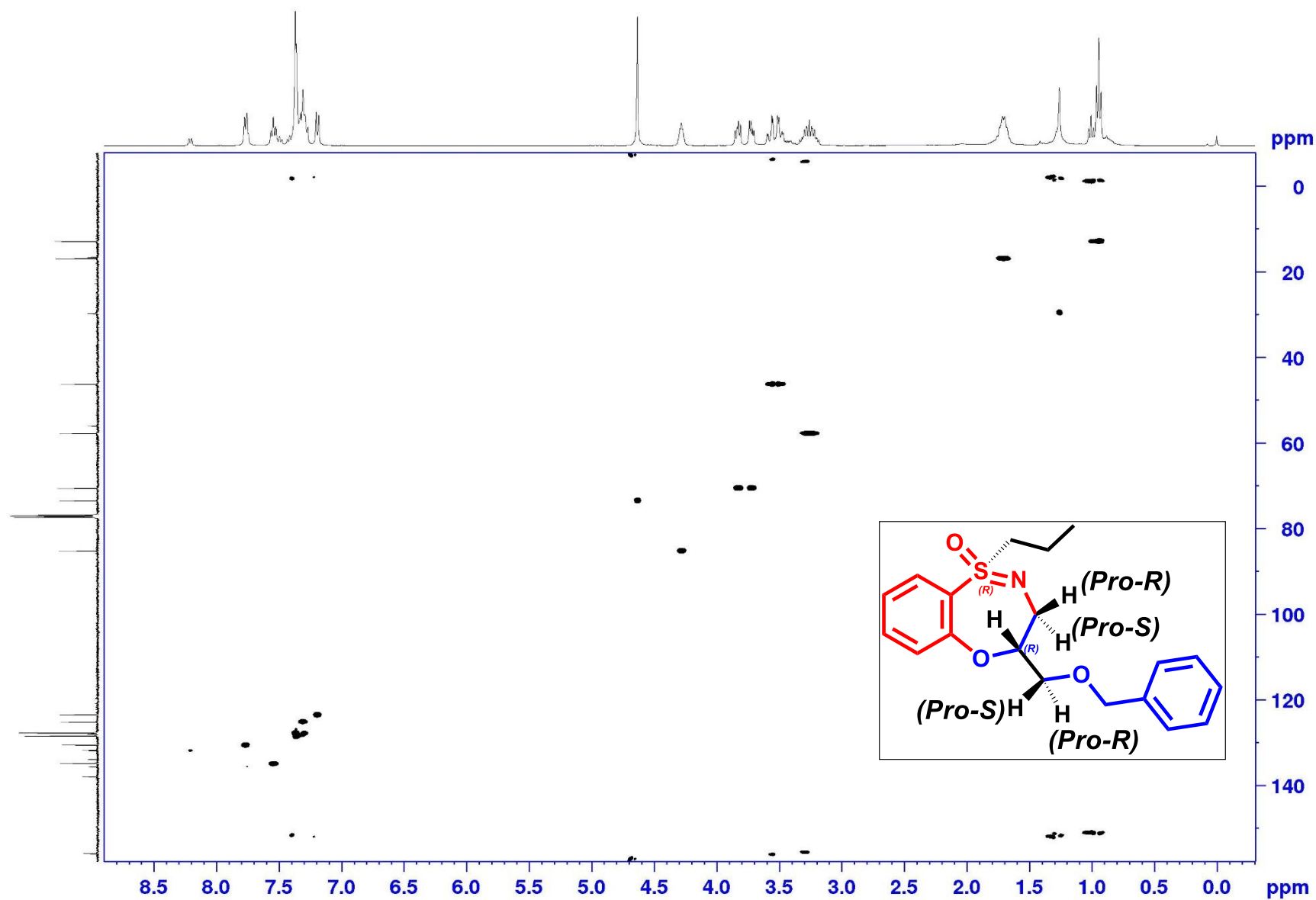


Fig S-156: HSQC Spectra of Compound (*R,R*)-4z (400 MHz, CDCl₃)

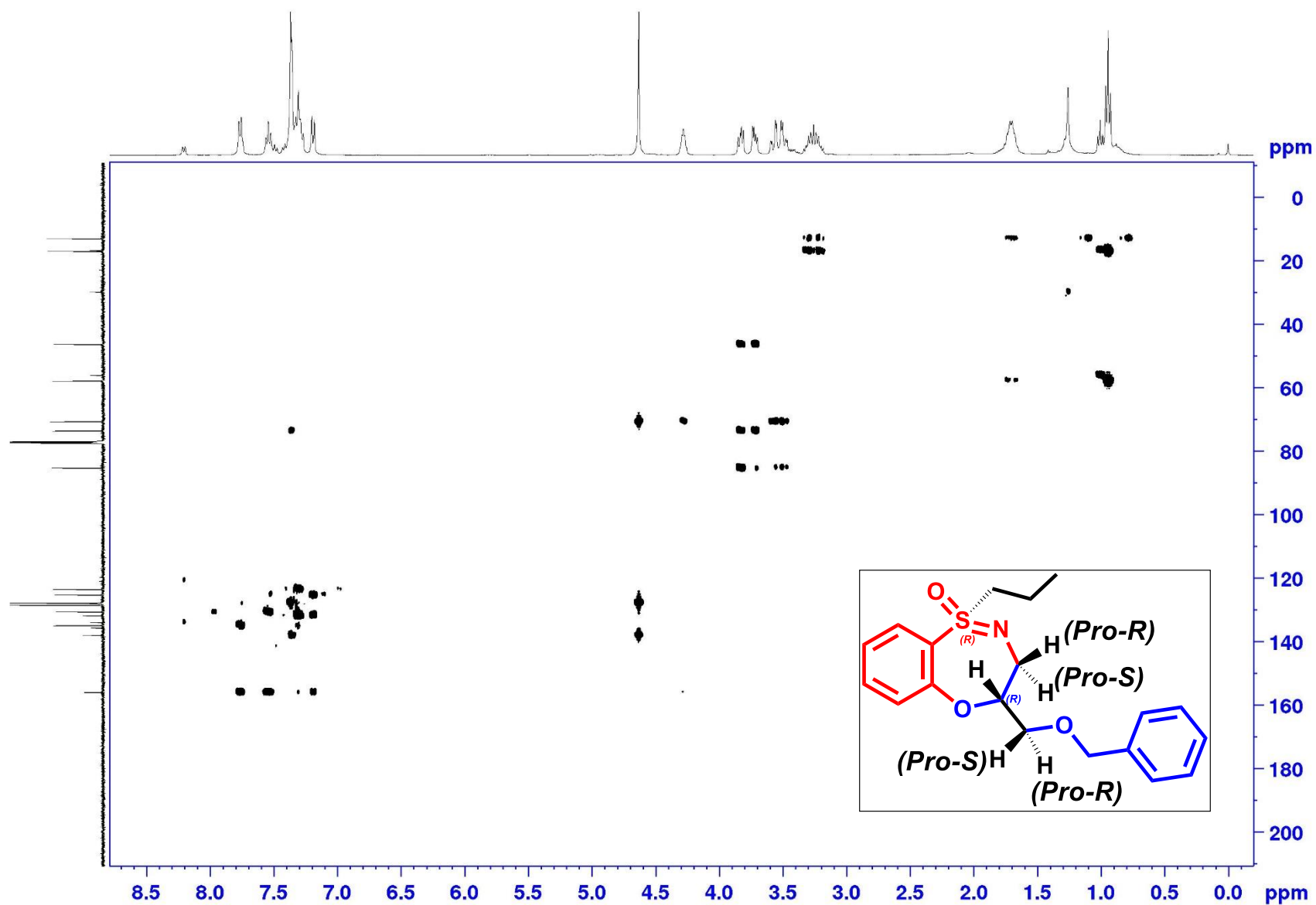


Fig S-157: HMBC Spectra of Compound (R,R)-4z (400 MHz, CDCl₃)

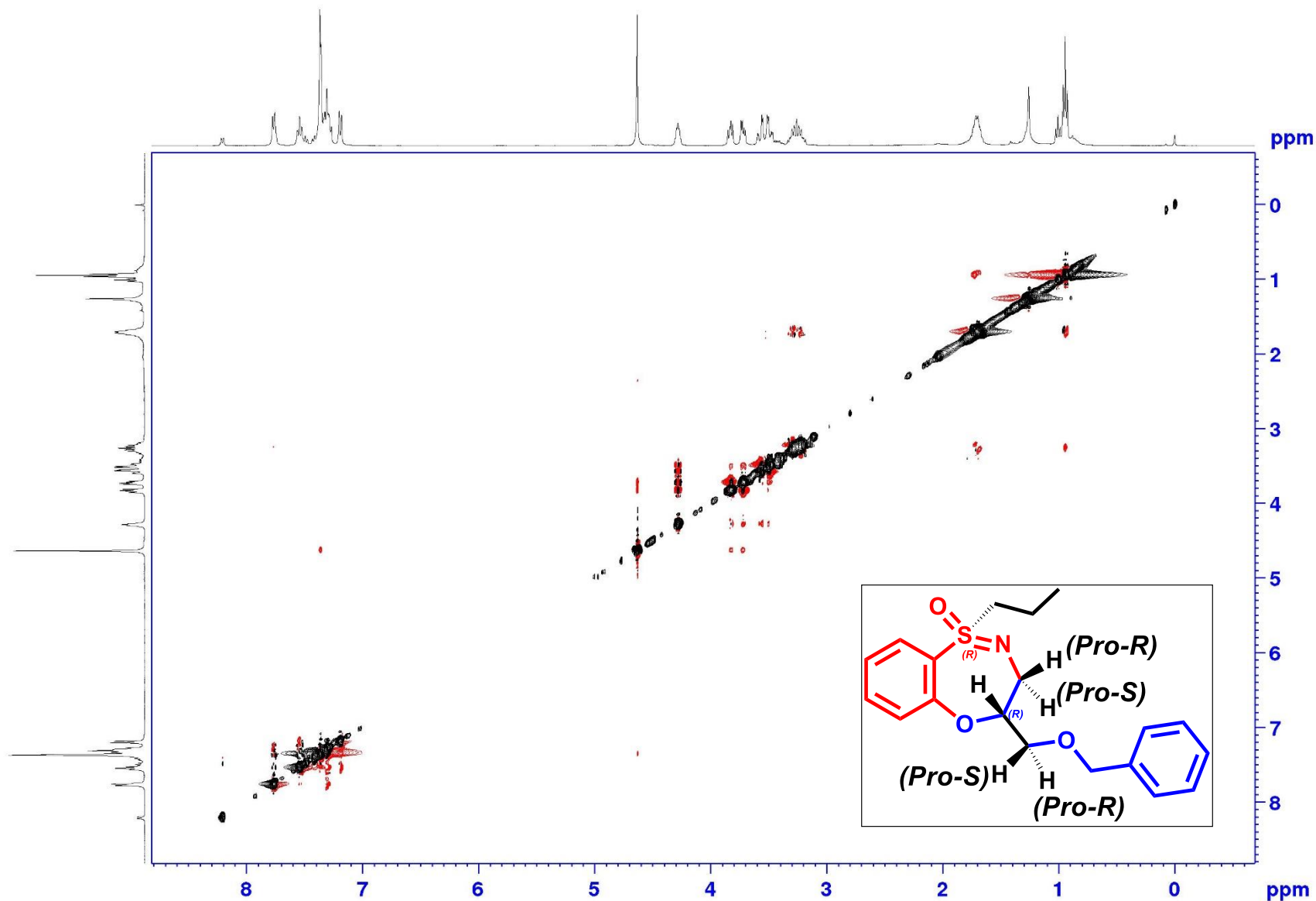


Fig S-158: NOESY Spectra of Compound (R,R)-4z (500 MHz, CDCl₃)

Sample Name	Ref-3-1a	Position	Vial 12	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I20APR12.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	4/20/2022 12:01:29 PM

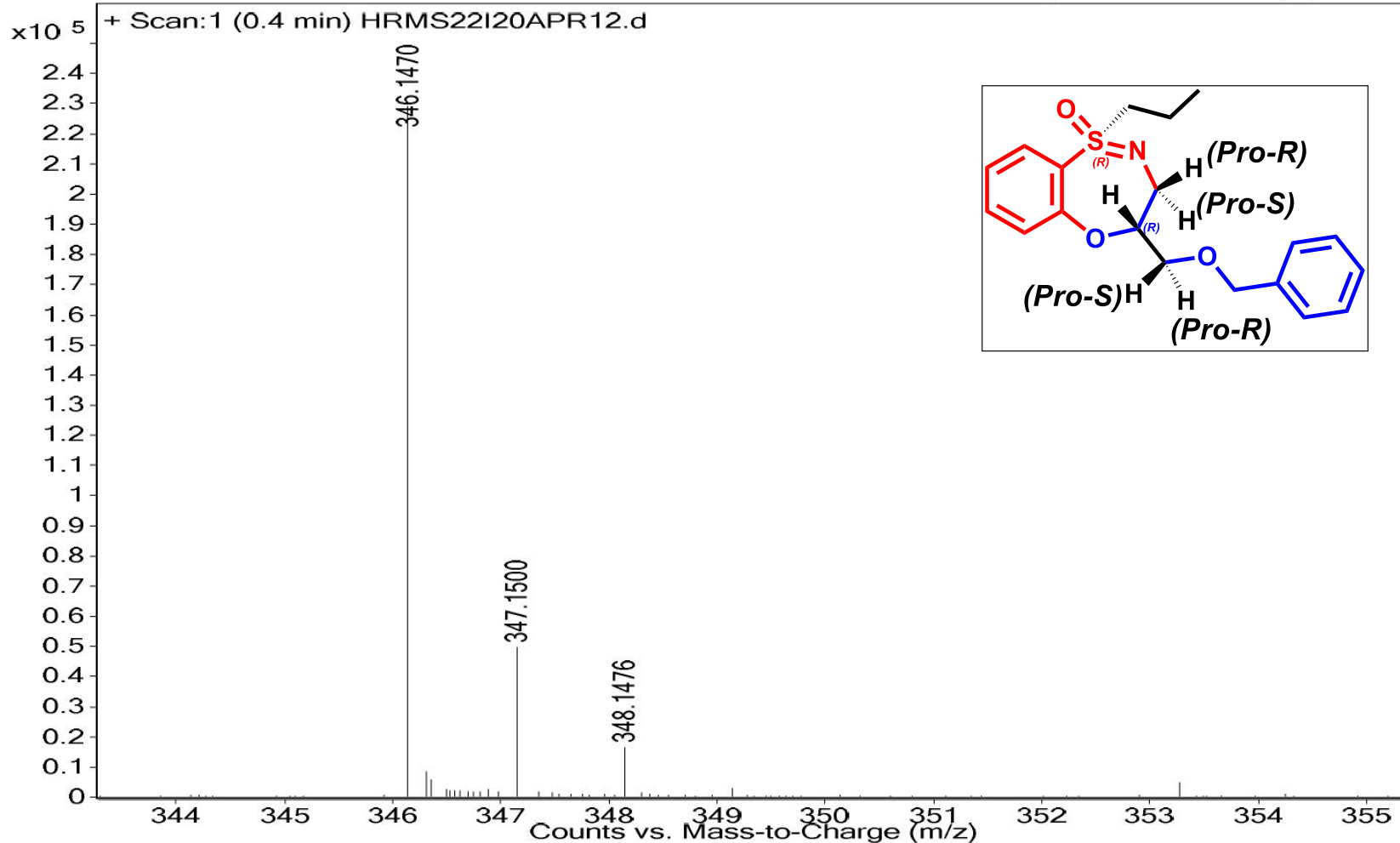


Fig S-159: HRMS report of Compound (*R,R*)- **4z**

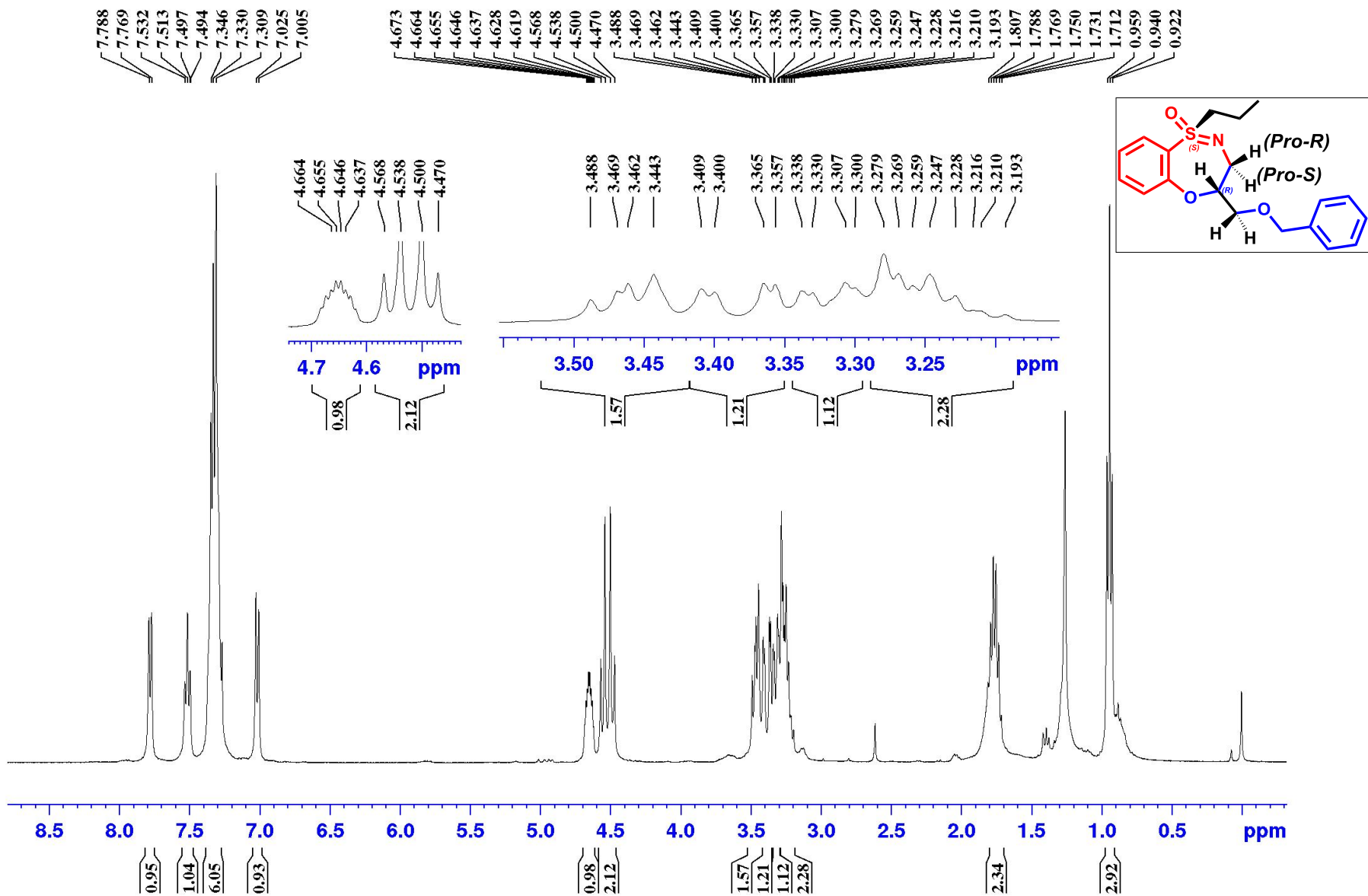


Fig S-160: ^1H NMR Spectra of Compound (R,S) -4z' (400 MHz, CDCl_3)

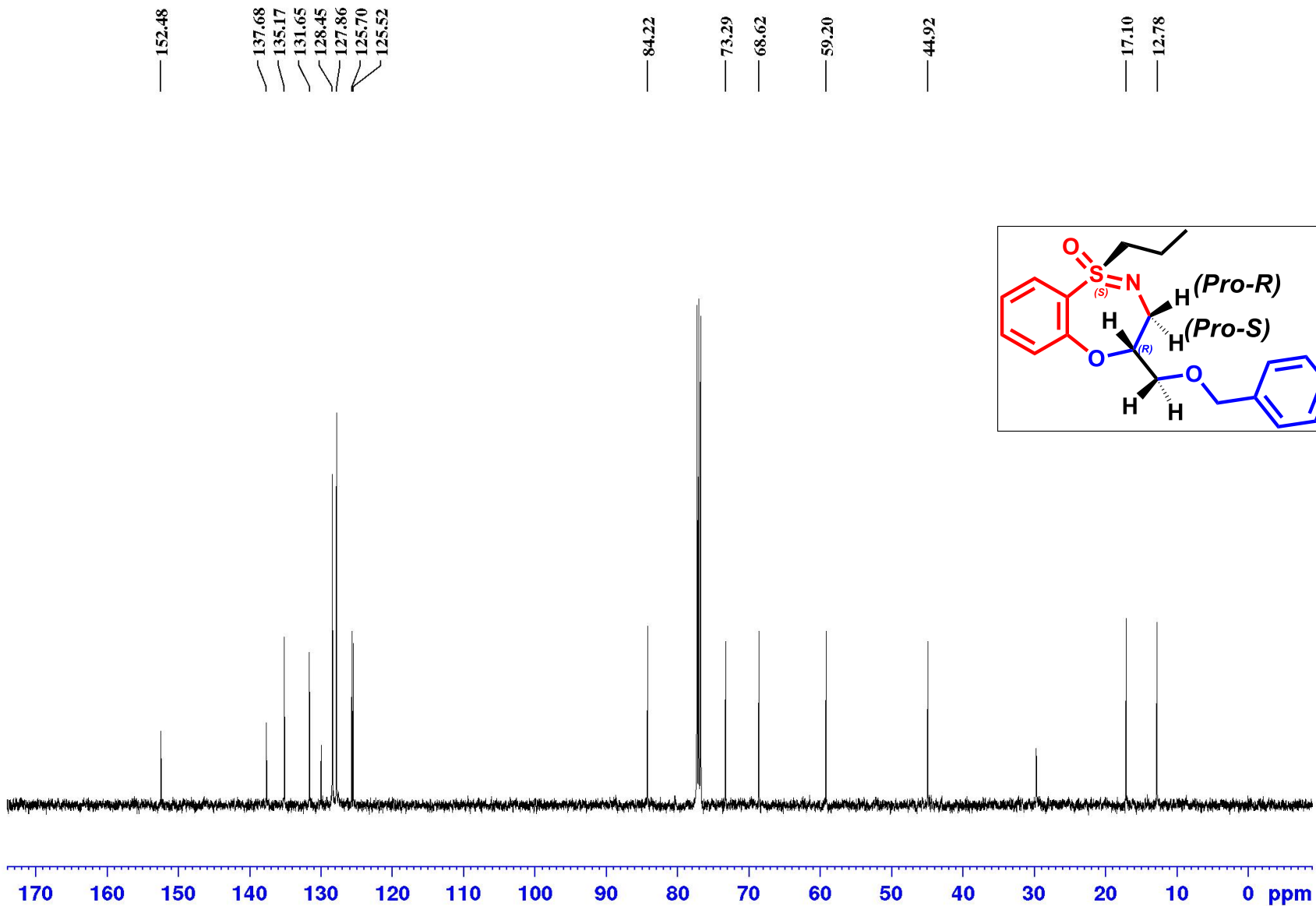


Fig S-161: ¹³C NMR Spectra of Compound (*R,S*)-4z' (125MHz, CDCl₃)

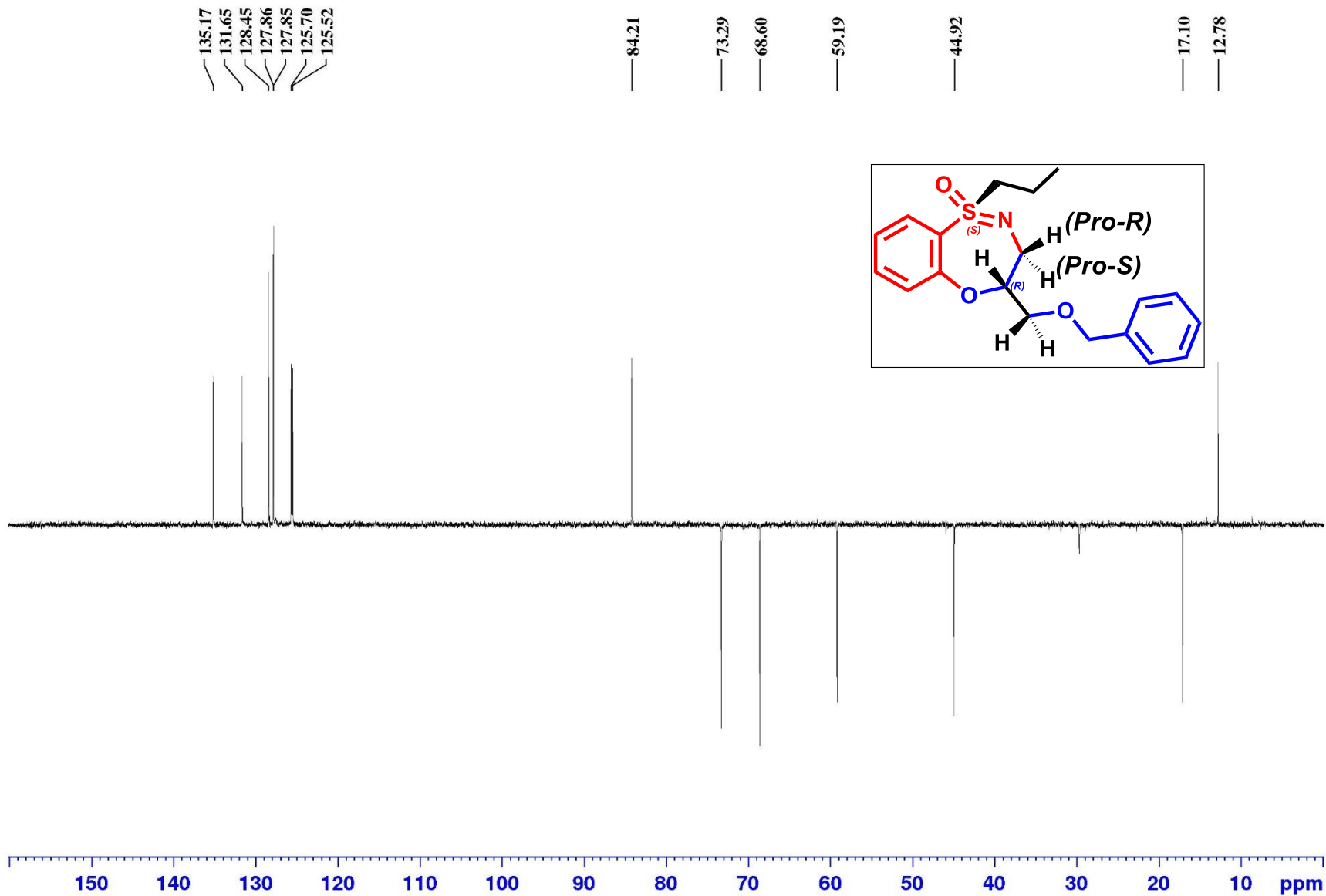


Fig S-162: DEPT-135 Spectra of Compound **(R,S)-4z'** (100 MHz, CDCl_3)

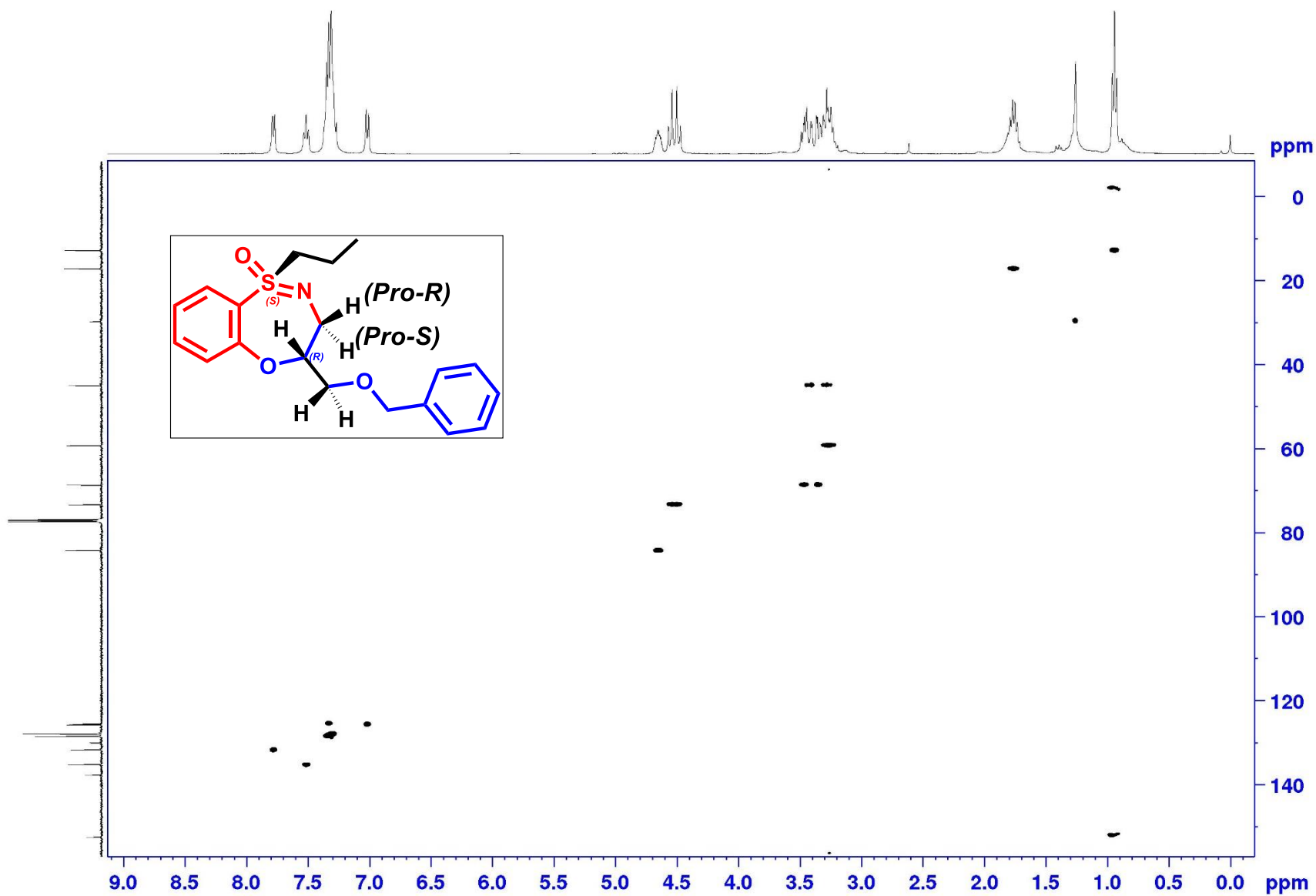


Fig S-163: COSY Spectra of Compound (*R,S*)-4z' (400 MHz, CDCl₃)

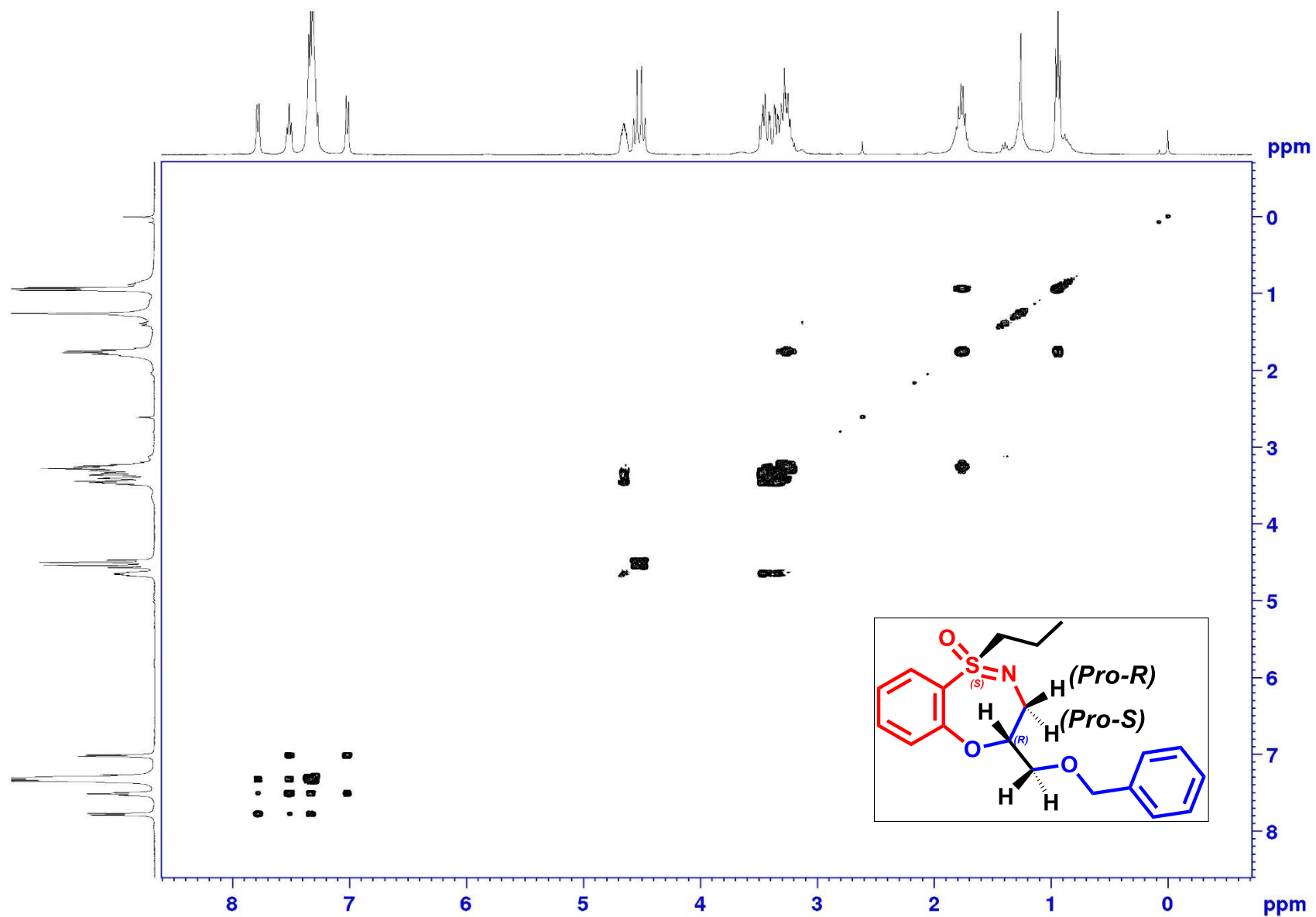


Fig S-164: HSQC Spectra of Compound (*R,S*)-4z' (400 MHz, CDCl₃)

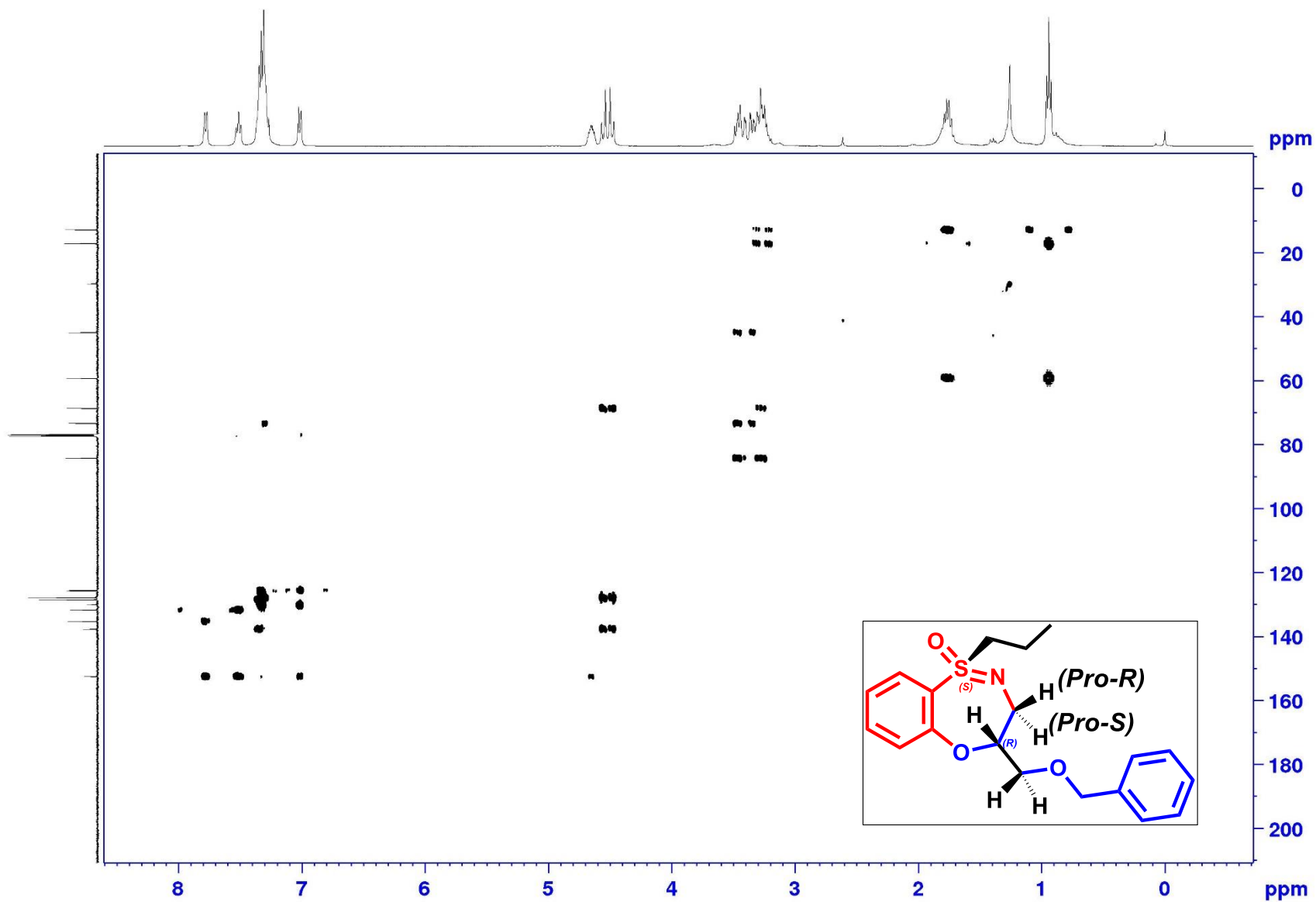


Fig S-165: HMBC Spectra of Compound (*R,S*)-4z' (400 MHz, CDCl₃)

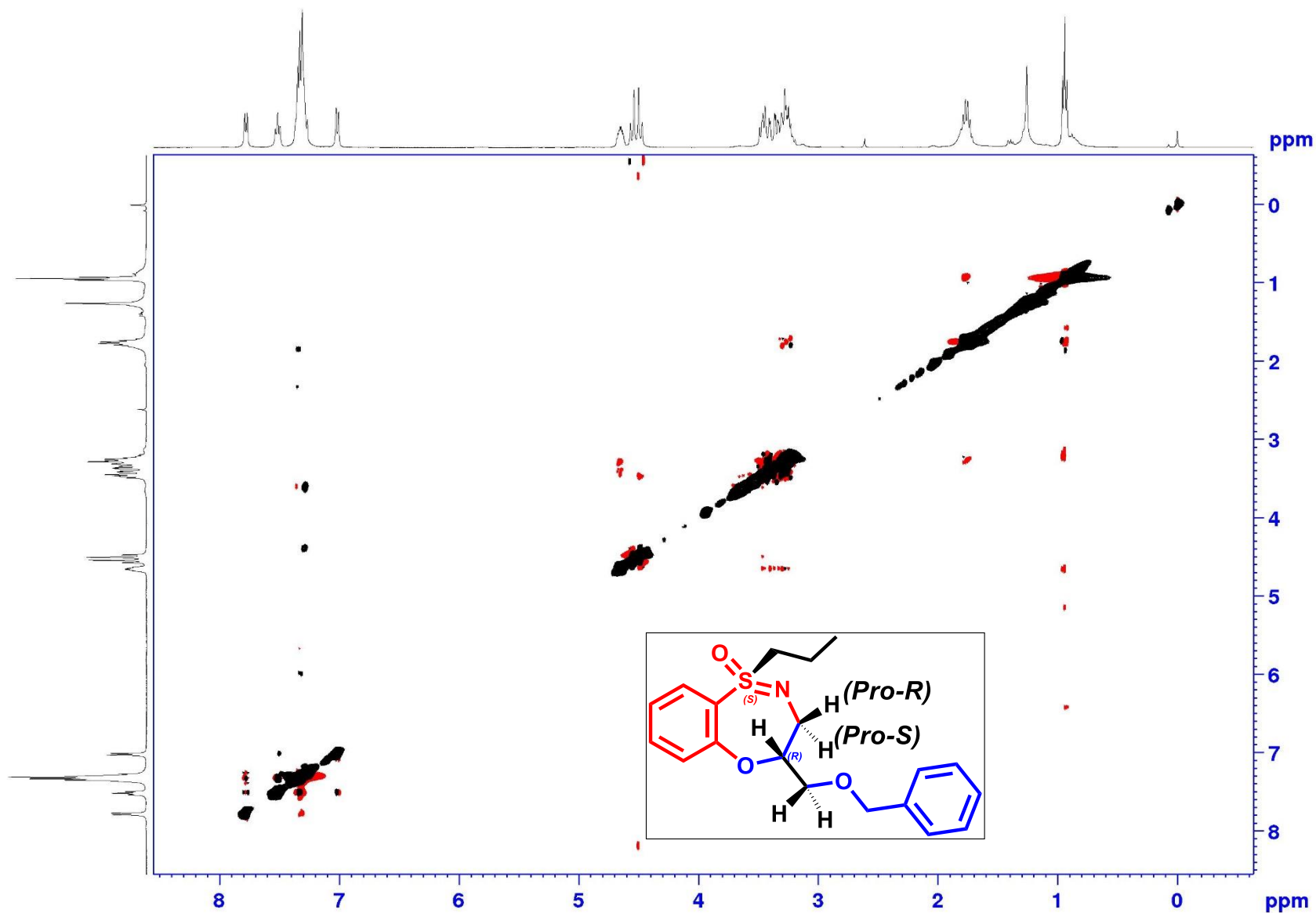


Fig S-166: NOESY Spectra of Compound (R,S)-4z' (500 MHz, CDCl₃)

Sample Name	Ref-3-1b	Position	Vial 13	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I20APR13.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	4/20/2022 12:04:04 PM

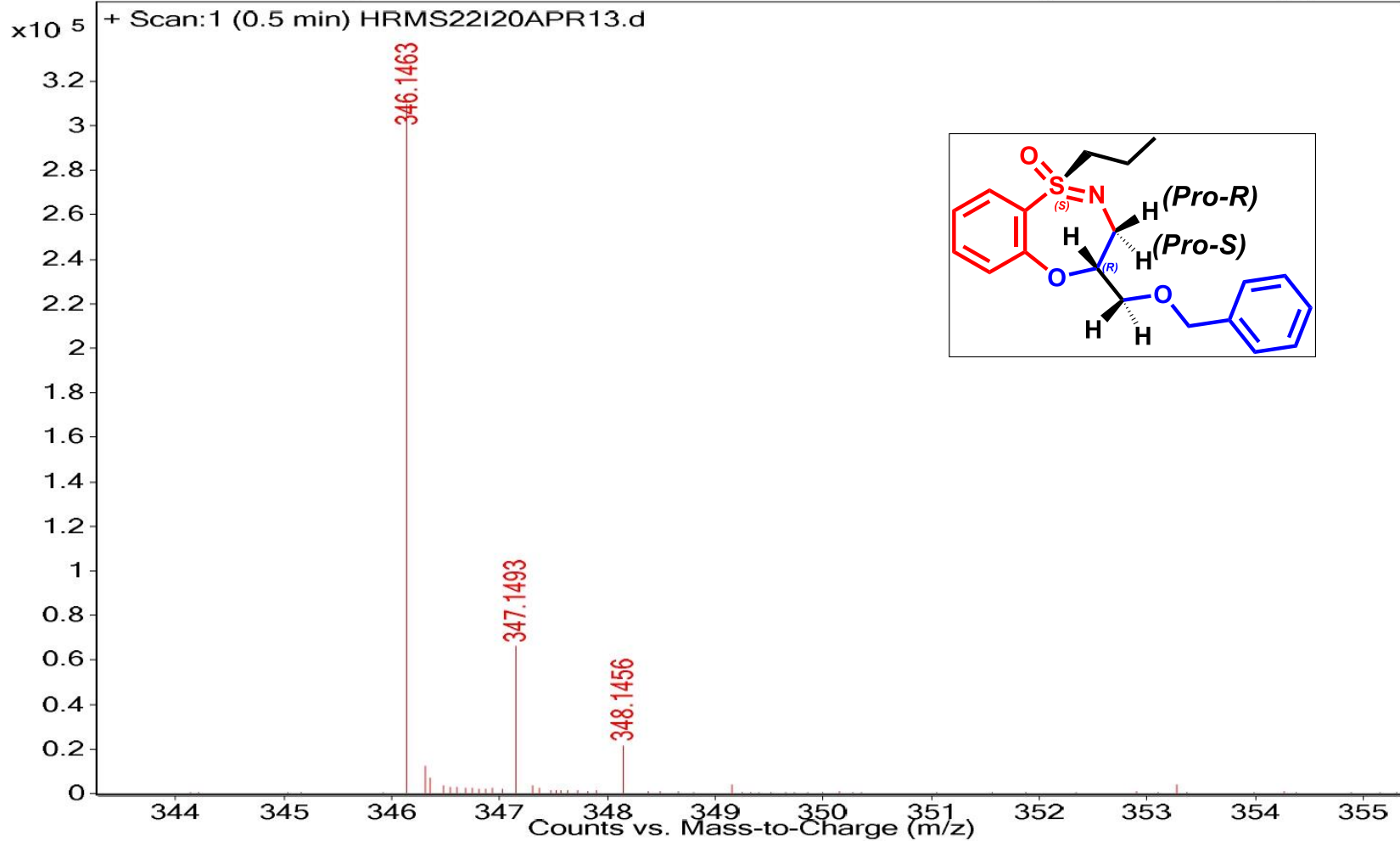


Fig S-167: HRMS report of Compound (R,S)-4z'

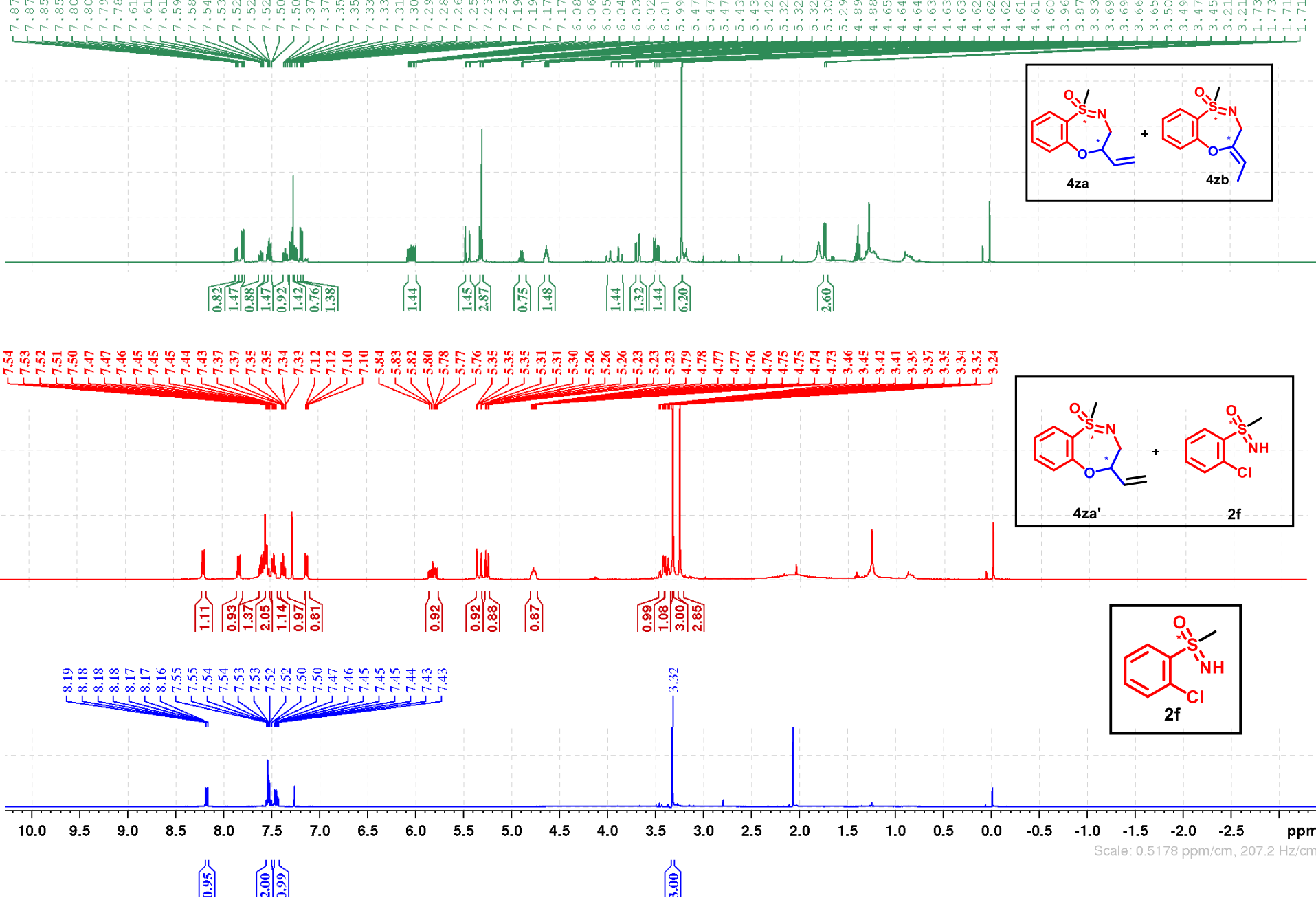


Fig S-168: ^1H NMR Spectra interpretation of Compound **4za**, **4za'**, **4zb** by correlated with **2f** (400 MHz, CDCl_3)

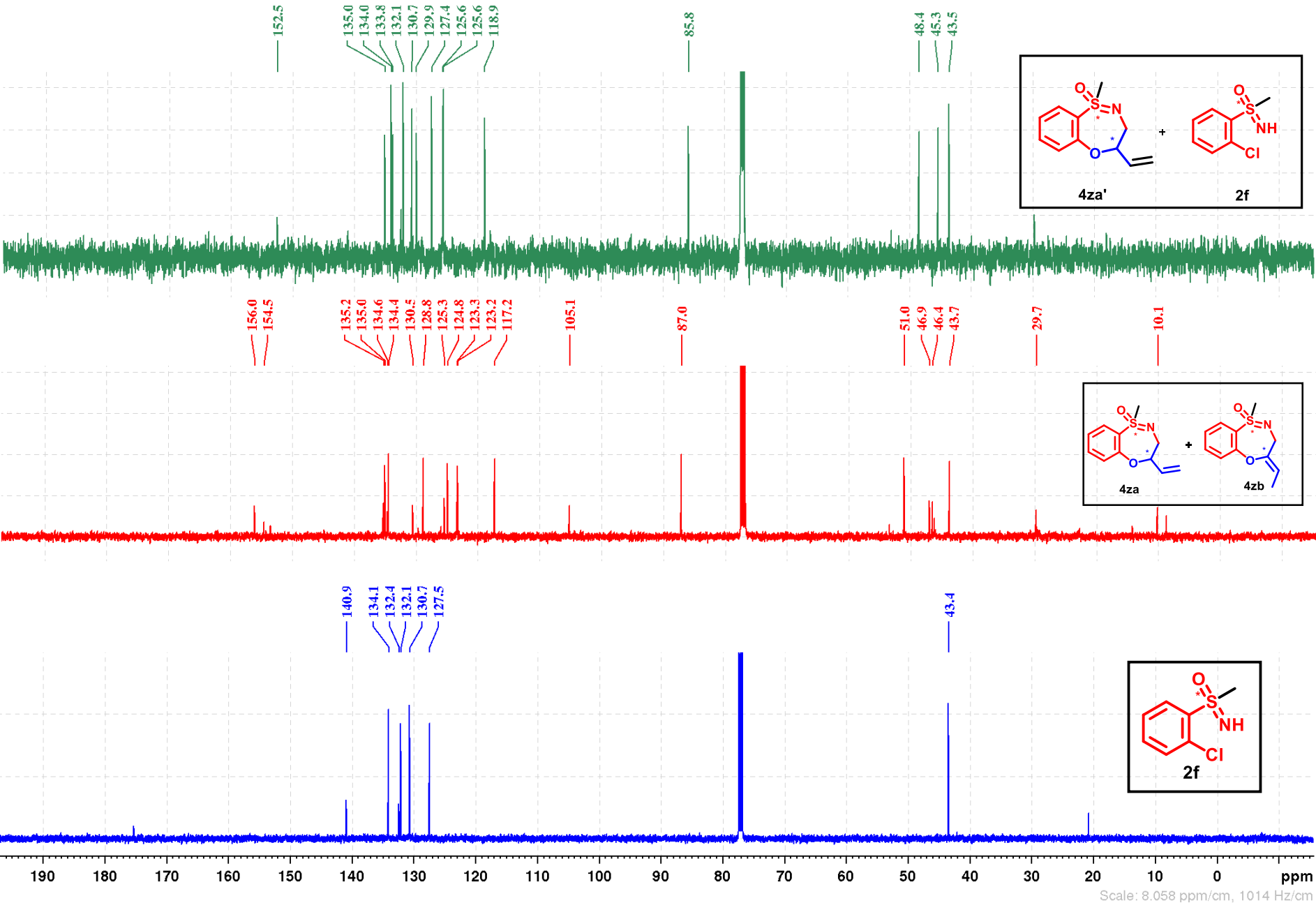


Fig S-169: ^{13}C NMR Spectra interpretation of Compound **4za**, **4za'**, **4zb** by correlated with **2f** (400 MHz, CDCl_3)

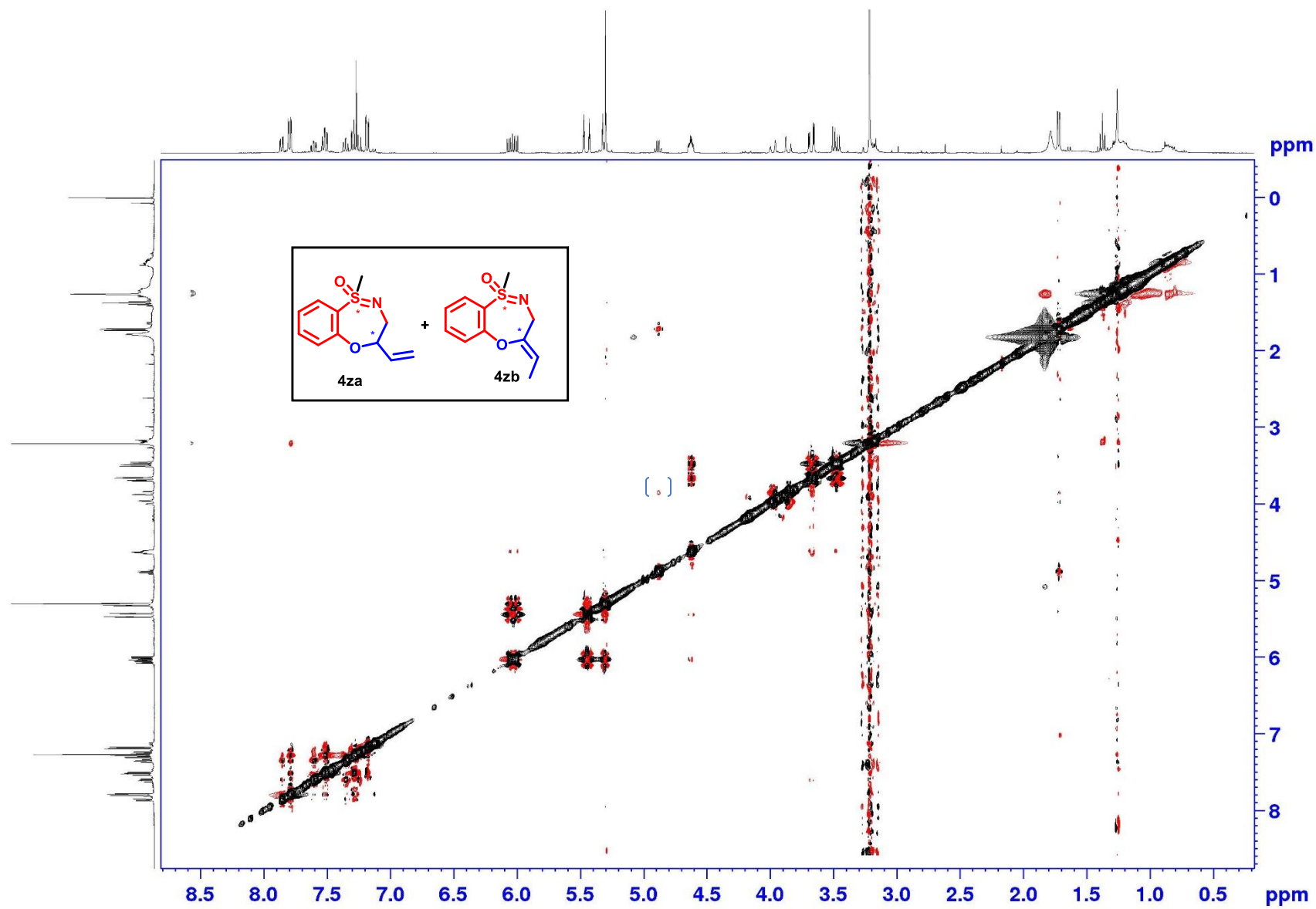


Fig S-170: NOESY Spectra of Compound 4za+4zb (500 MHz, CDCl₃)

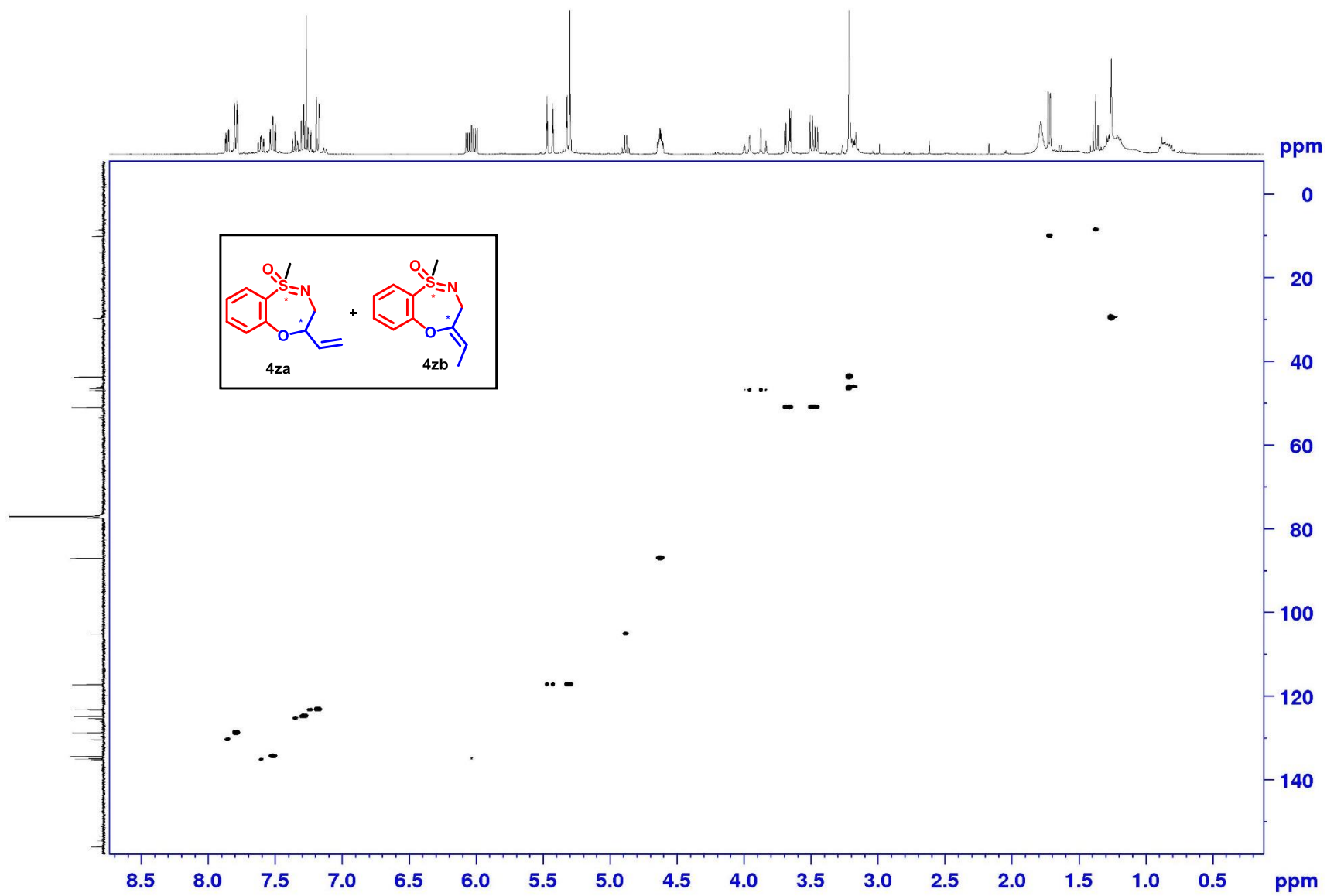


Fig S-171: HSQC Spectra of Compound 4za+4zb (400 MHz, CDCl_3)

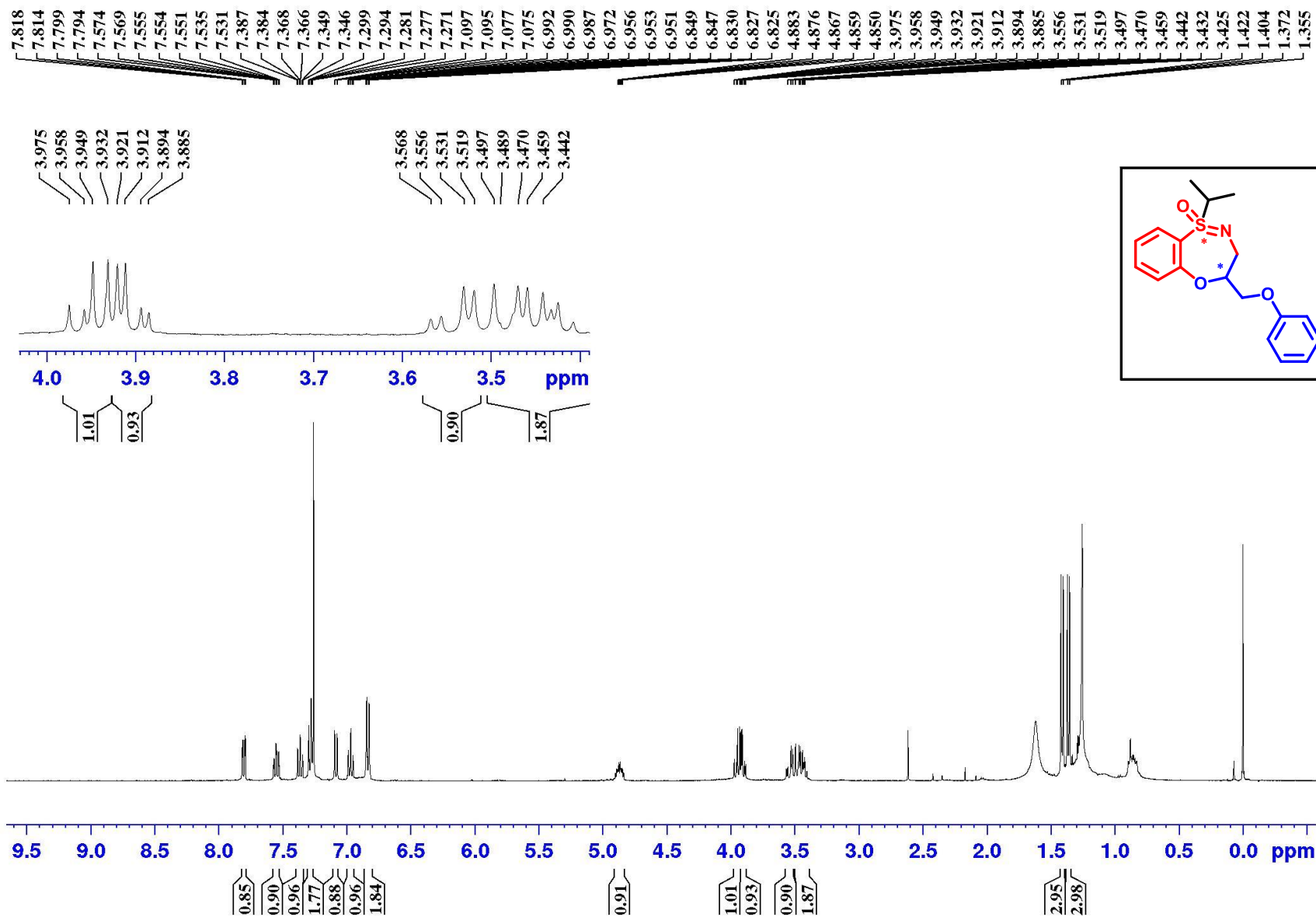


Fig S-172: ^1H NMR Spectra of Compound 4zc (400 MHz, CDCl_3)

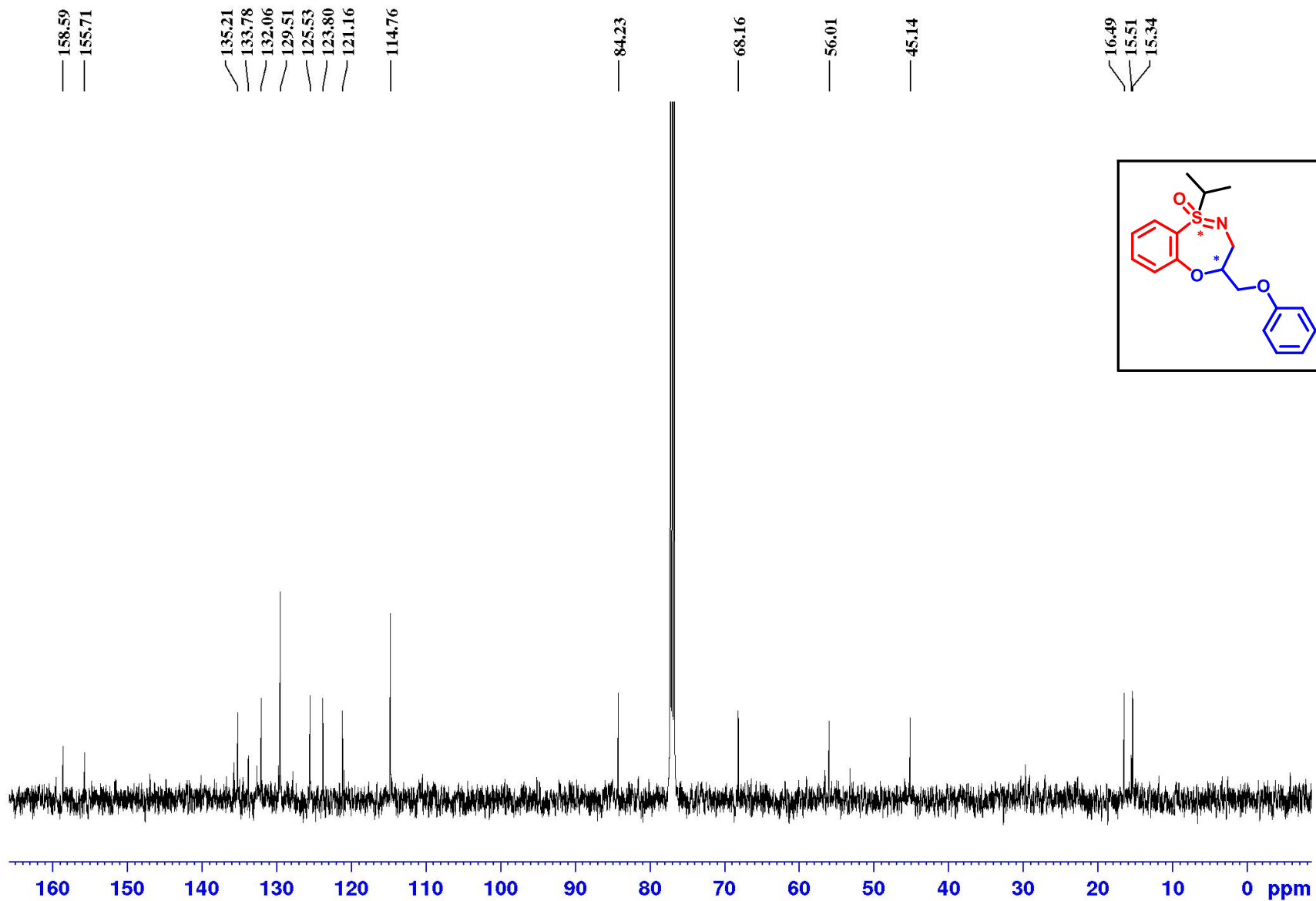


Fig S-173: ^{13}C NMR Spectra of Compound **4zc** (125 MHz, CDCl_3)

Sample Name	SP 1854	Position	Vial 1	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I12MAY01.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	5/12/2022 11:24:12 AM

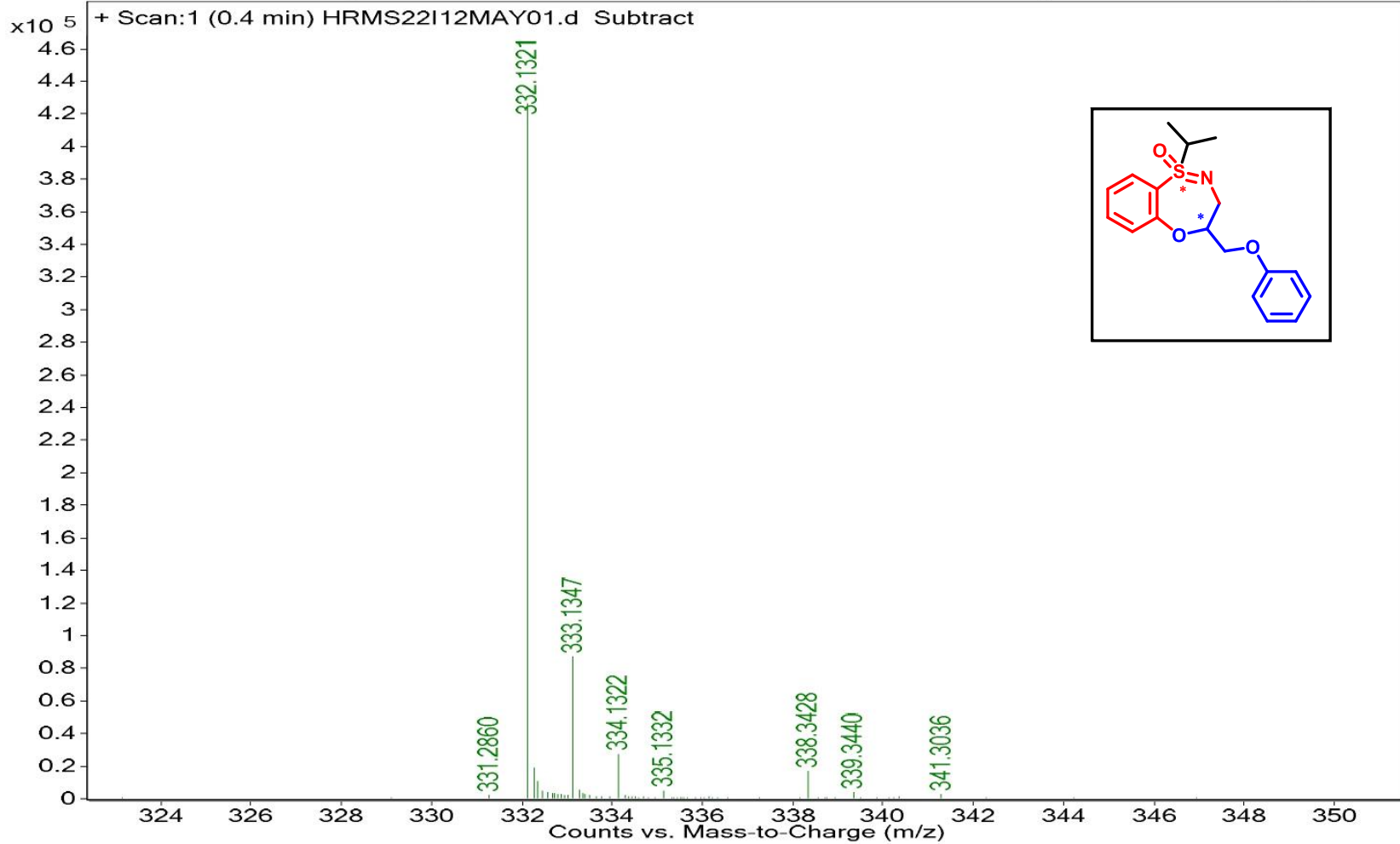


Fig S-174: HRMS report of Compound 4zc

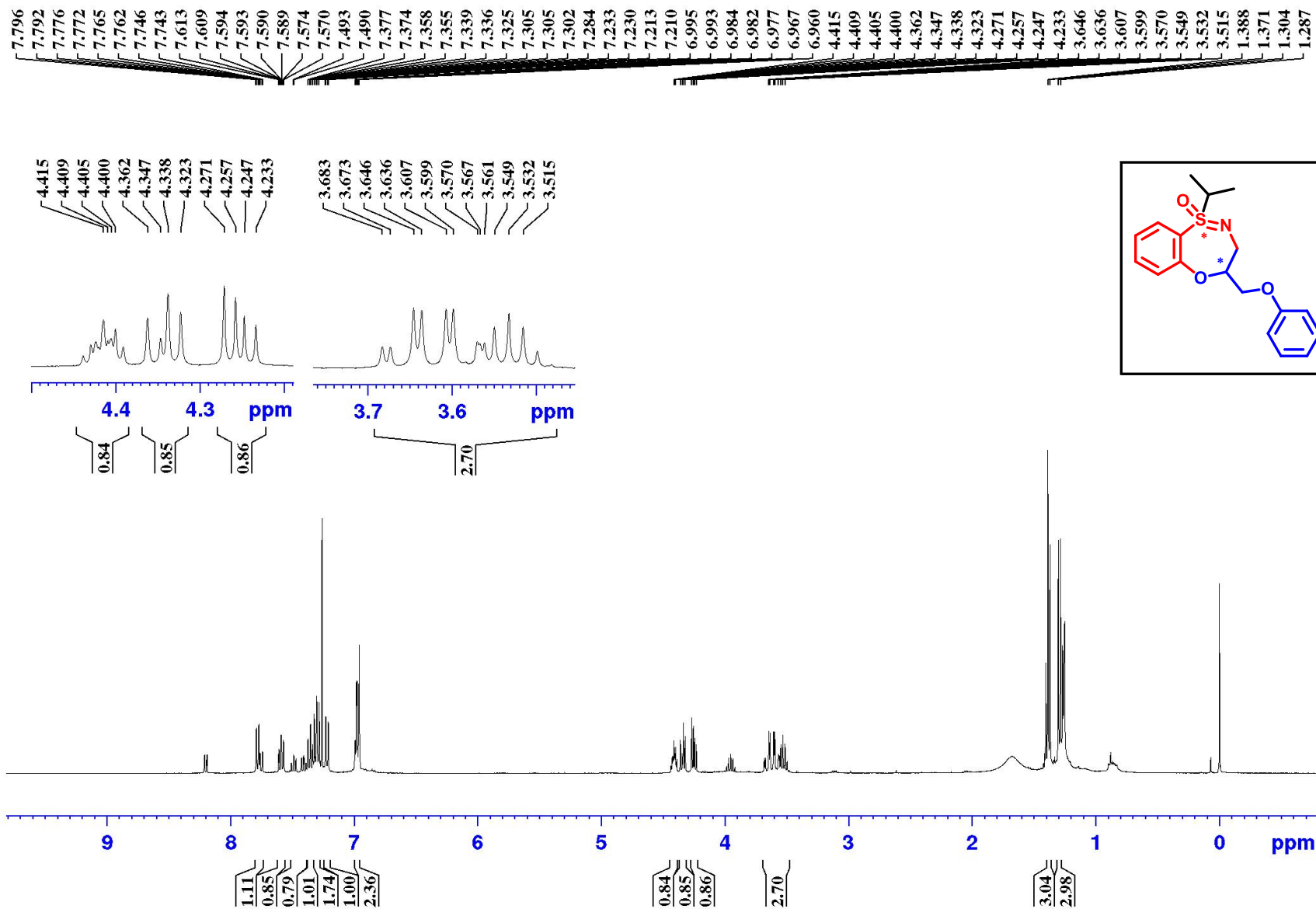


Fig S-175: ^1H NMR Spectra of Compound **4zc'** (400 MHz, CDCl_3)

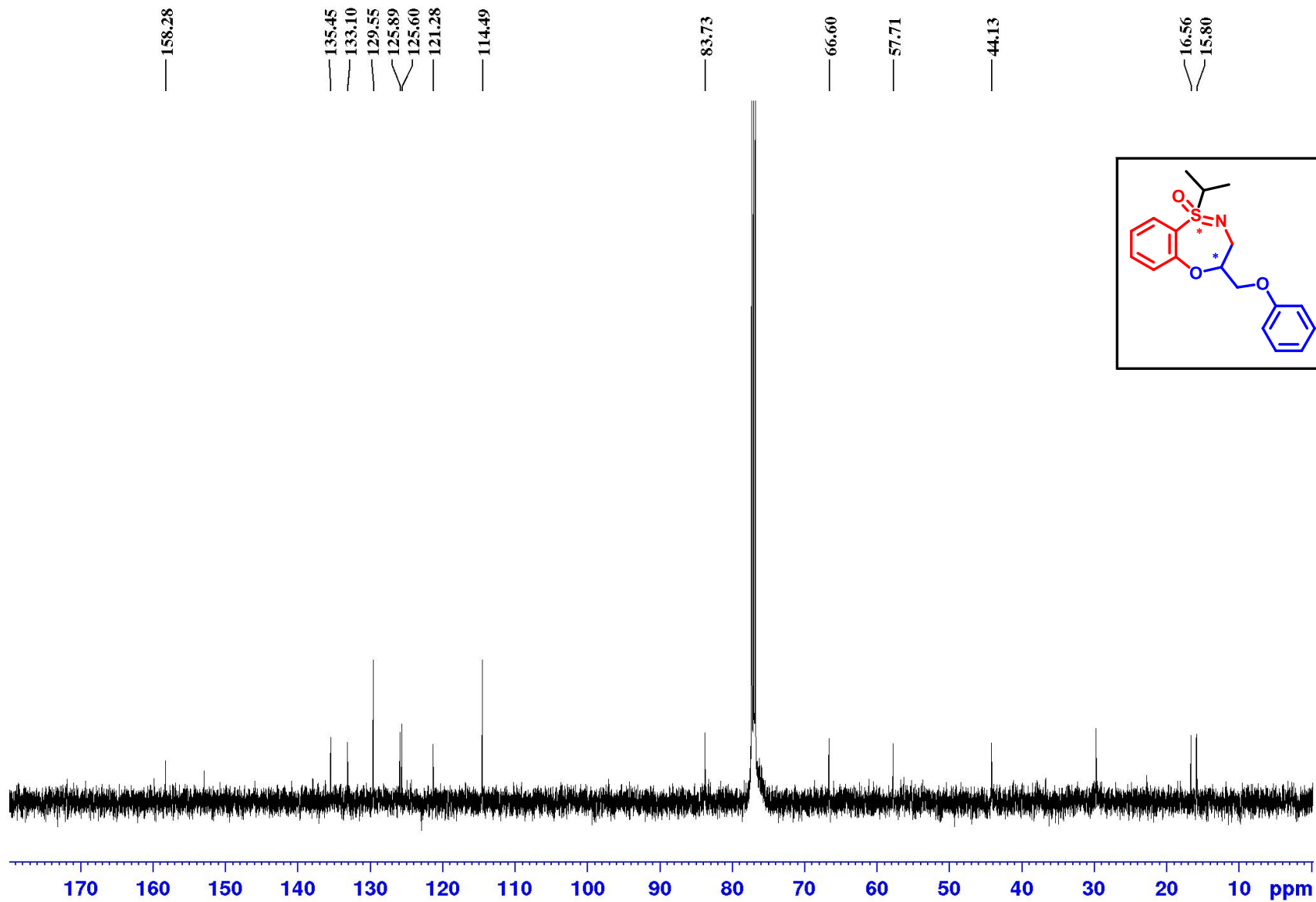


Fig S-176: ^{13}C NMR Spectra of Compound 4zc' (100 MHz, CDCl_3)

Sample Name	SP 1853	Position	Vial 2	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I12MAY02.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	5/12/2022 11:27:49 AM

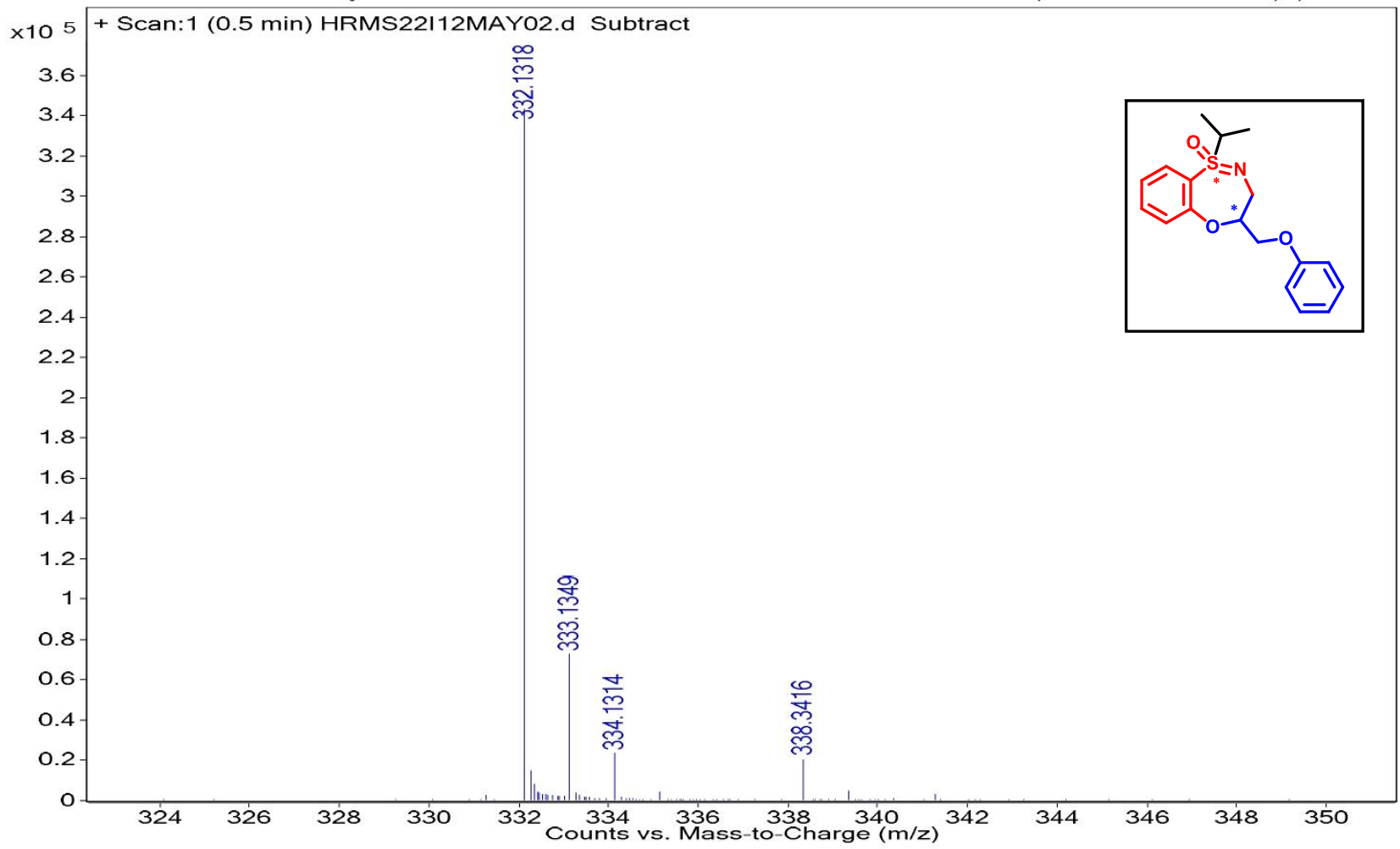


Fig S-177: HRMS report of Compound 4zc'

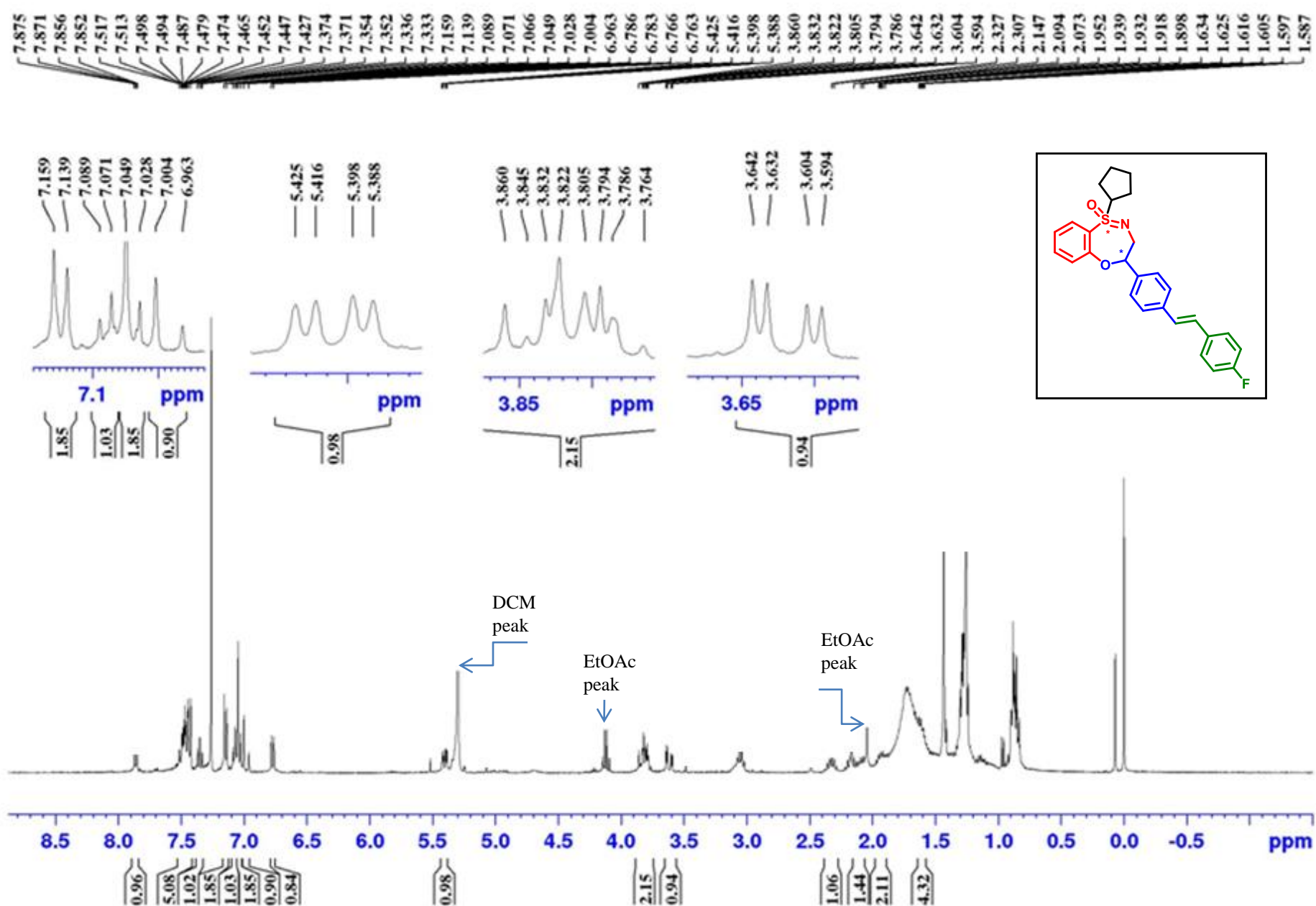


Fig S-178: ^1H NMR Spectra of Compound **5a** (400 MHz, CDCl_3)

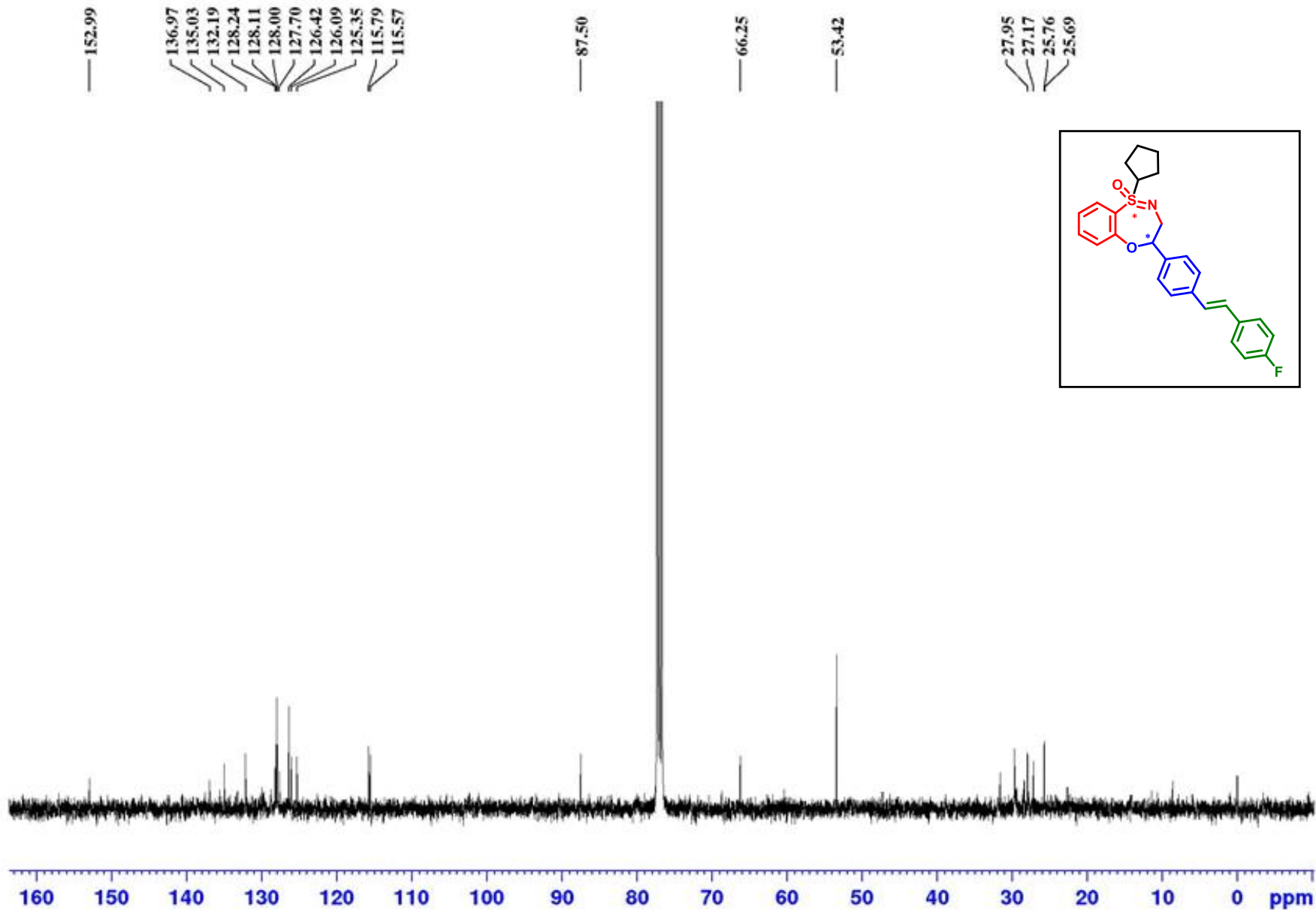


Fig S-179: ^{13}C NMR Spectra of Compound **5a** (100 MHz, CDCl_3)

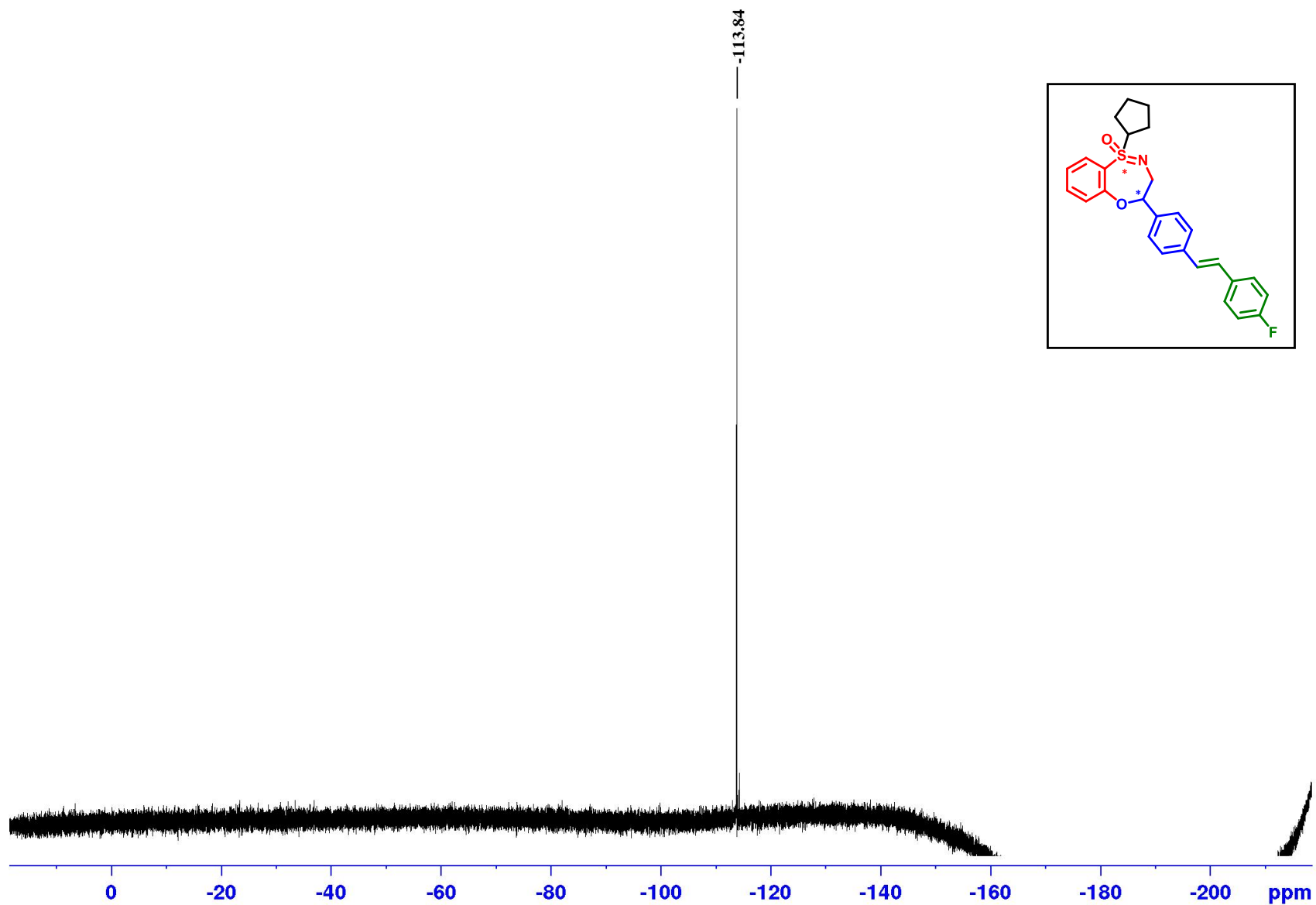


Fig S-180: ^{19}F NMR Spectra of Compound **5a** (376 MHz, CDCl_3)

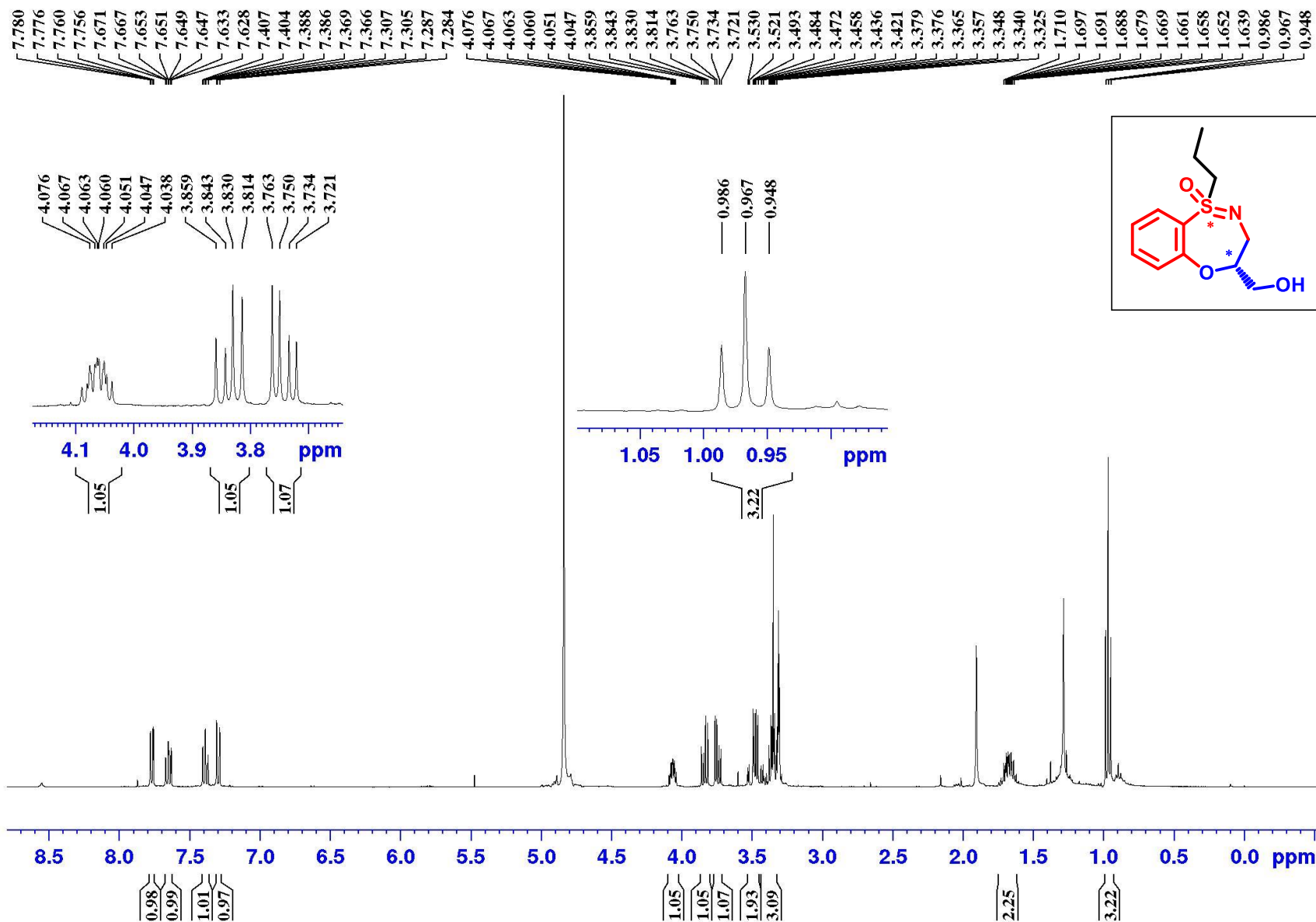


Fig S-181: ^1H NMR Spectra of Compound **5b** (400 MHz, CD_3OD)

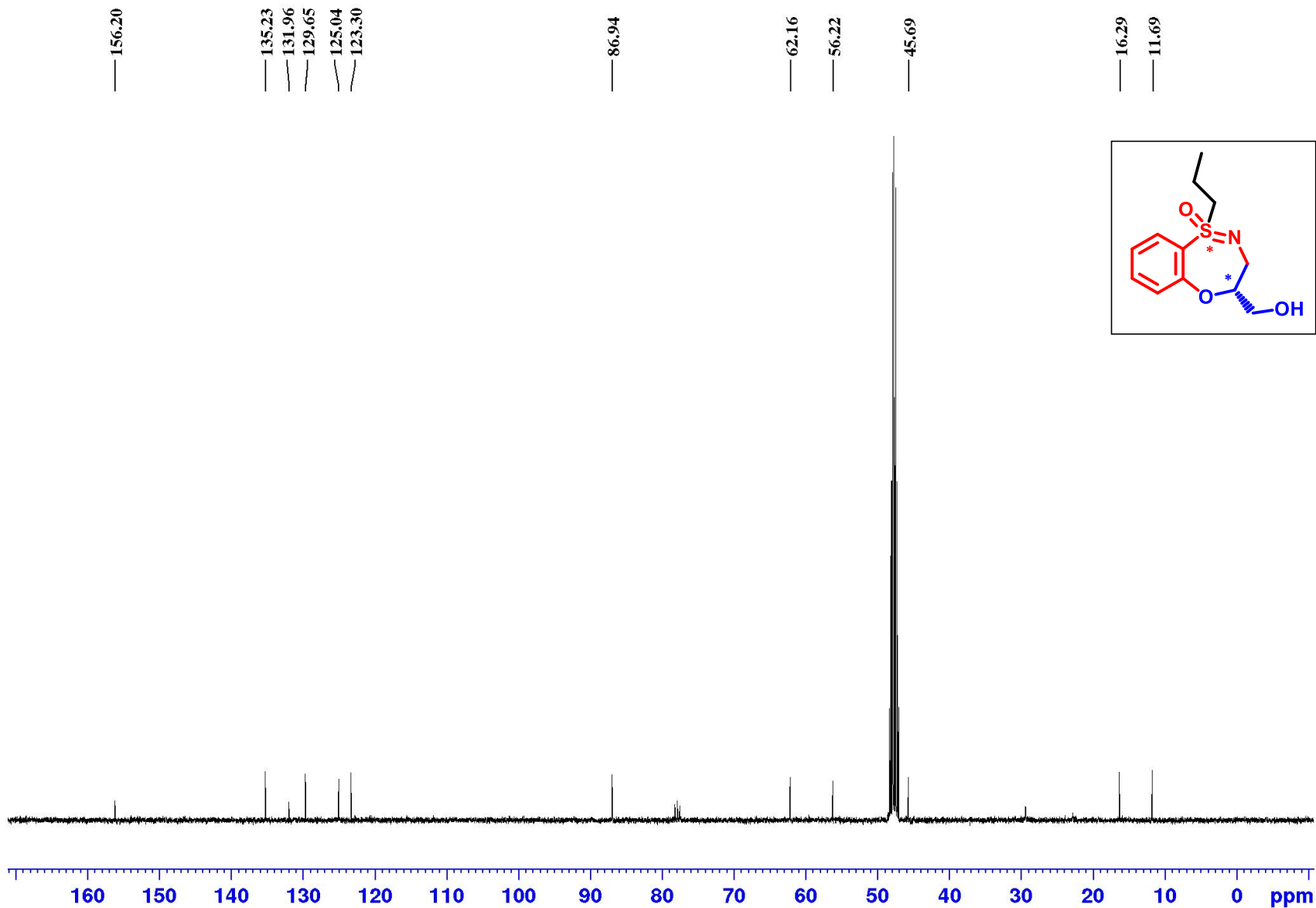


Fig S-182: ^{13}C NMR Spectra of Compound **5b** (100 MHz, CD_3OD)

Sample Name	5b	Position	Vial 25	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I10MAY25.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	5/10/2022 12:32:08 PM

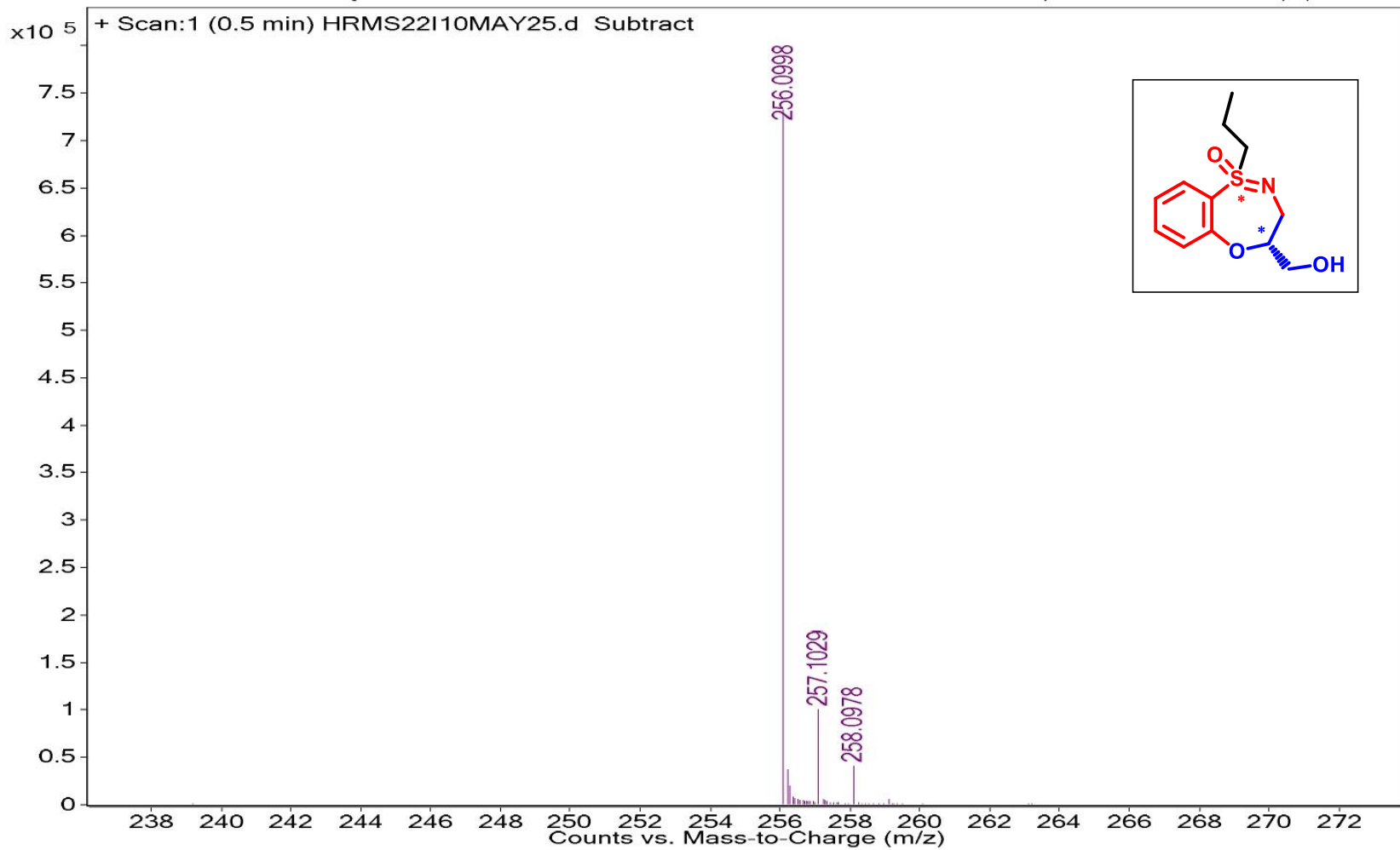


Fig S-183: HRMS report of Compound **5b**

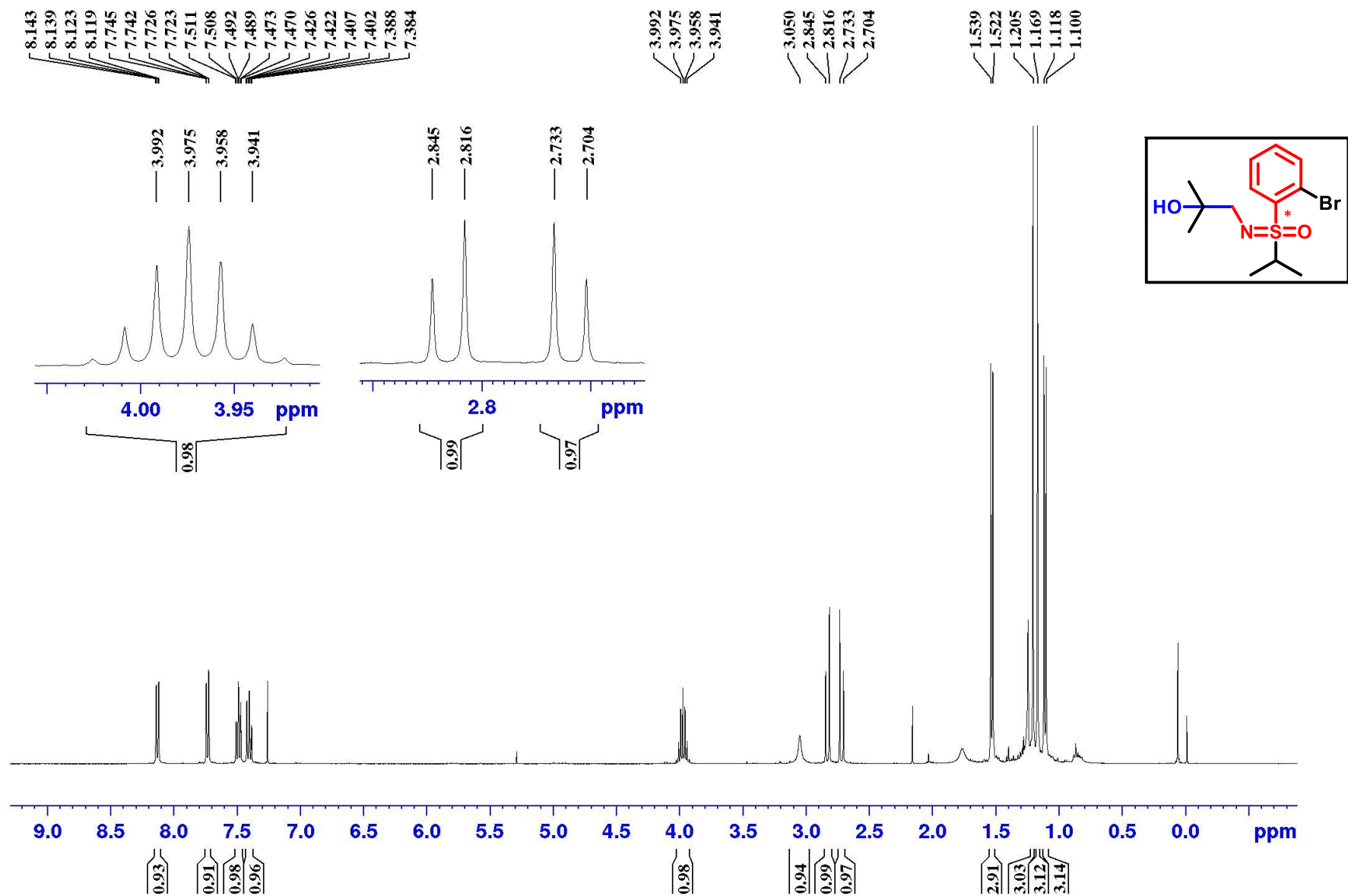


Fig S-184: ^1H NMR Spectra of Compound **5c** (400 MHz, CDCl_3)

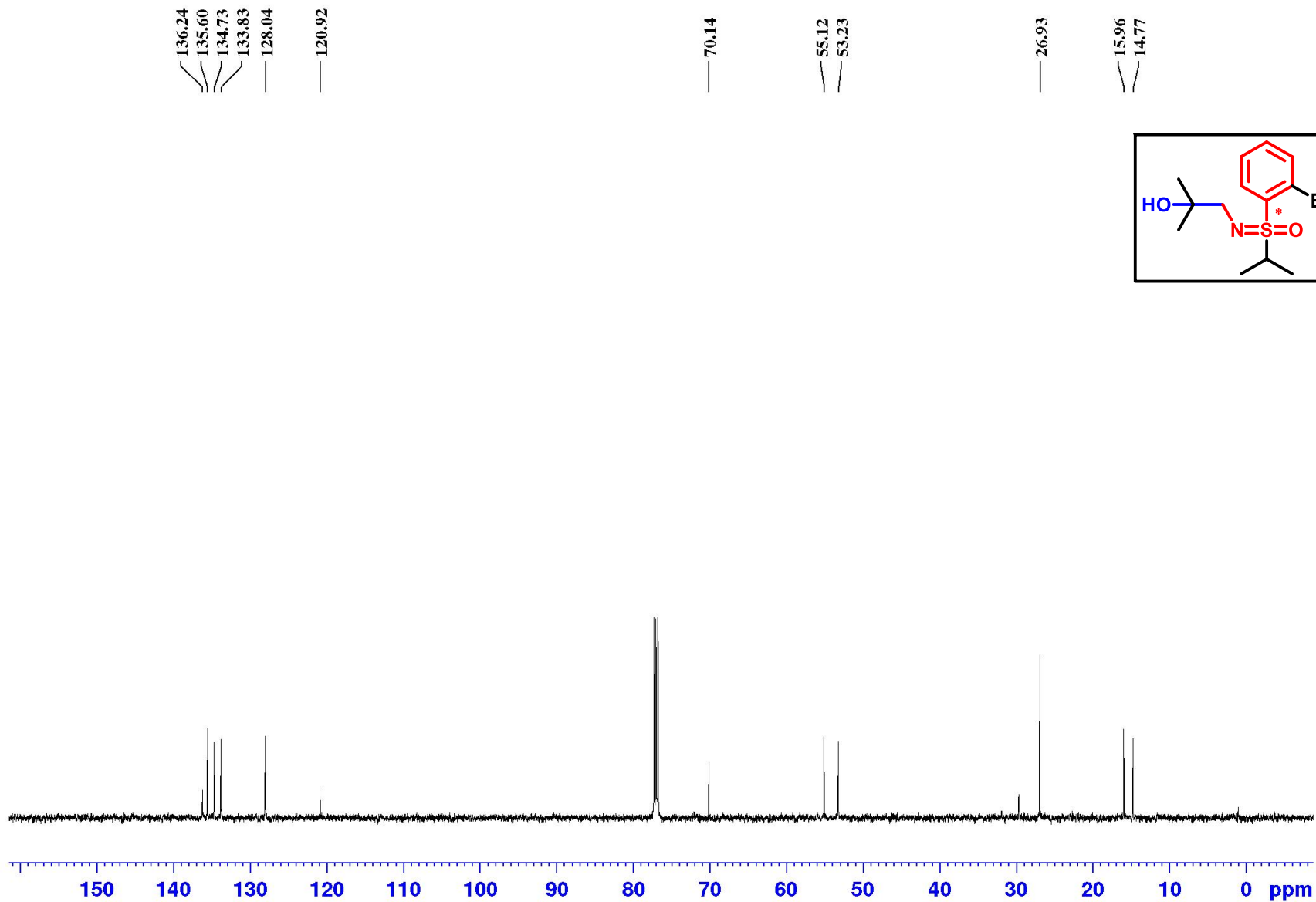


Fig S-185: ^{13}C NMR Spectra of Compound 5c (125 MHz, CDCl_3)

Sample Name	4ze	Position	Vial 29	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	HRMS22I11MAY29.d	ACQ Method	ISOCRATIC.m	Comment		Acquired Time	5/11/2022 12:39:56 PM

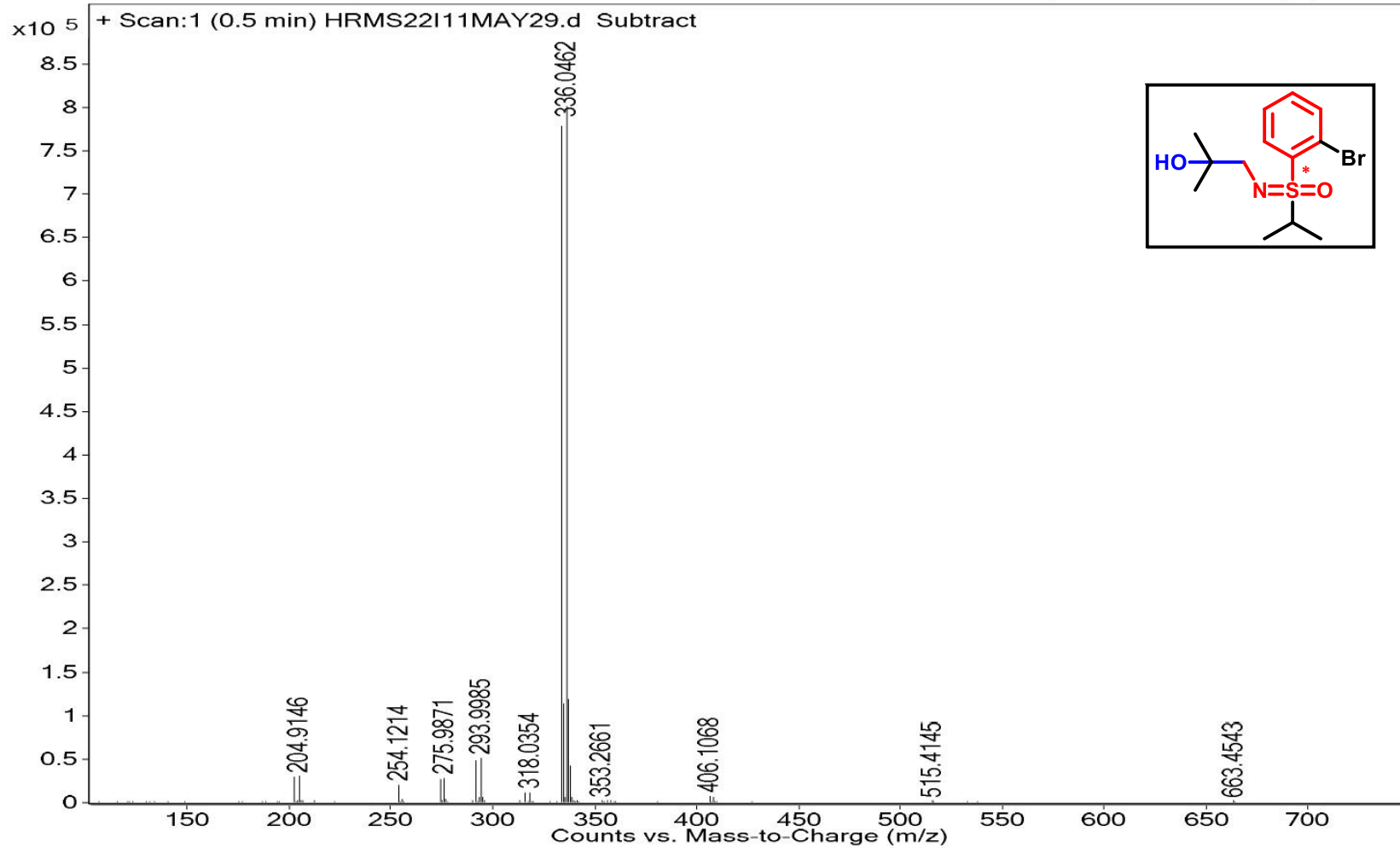


Fig S-186: HRMS report of Compound 5c

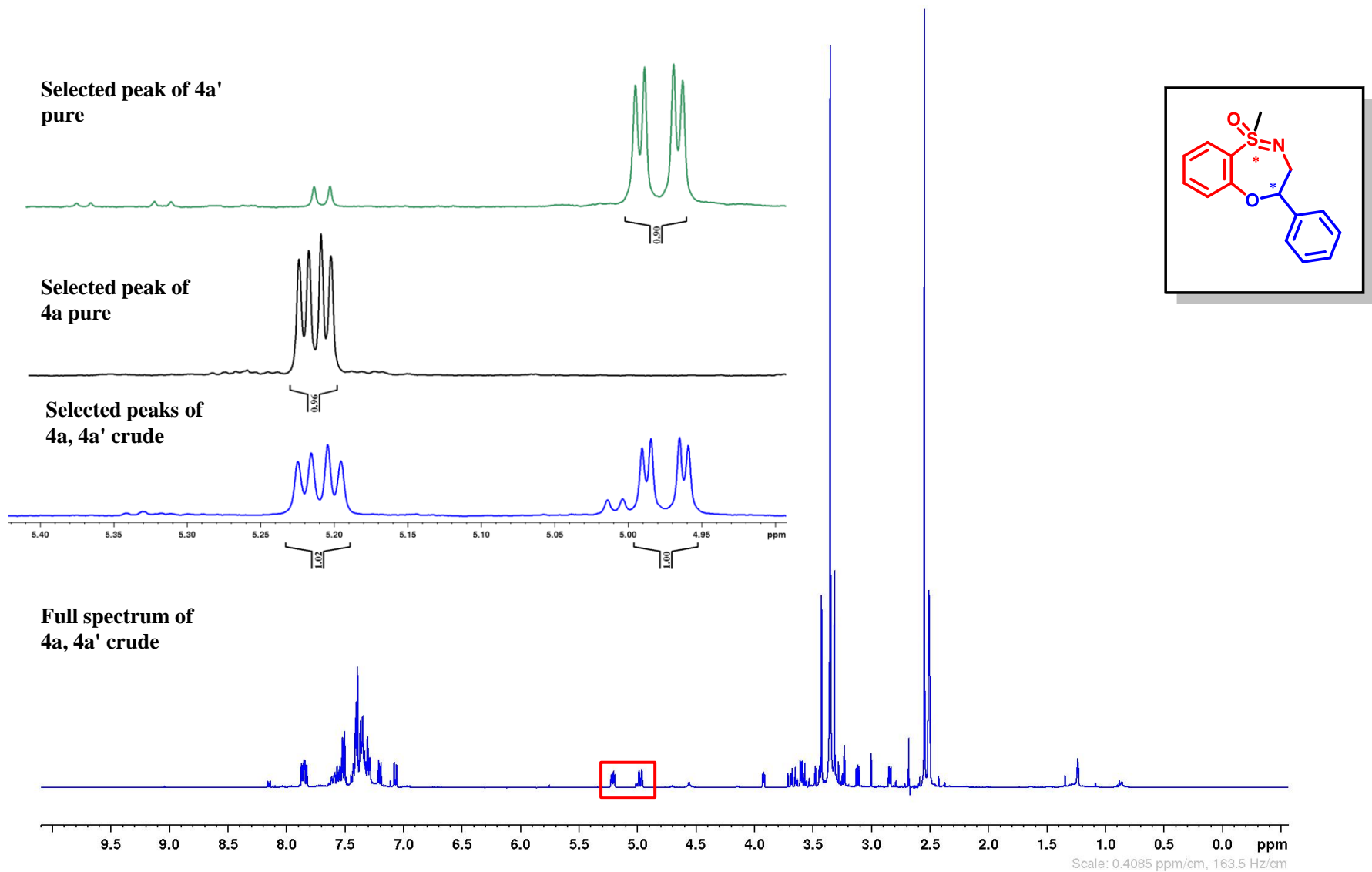


Fig S-187: Diastereomeric ratio calculation of Compound **4a** and **4a'** by ¹H NMR Spectra (400 MHz, CDCl₃)

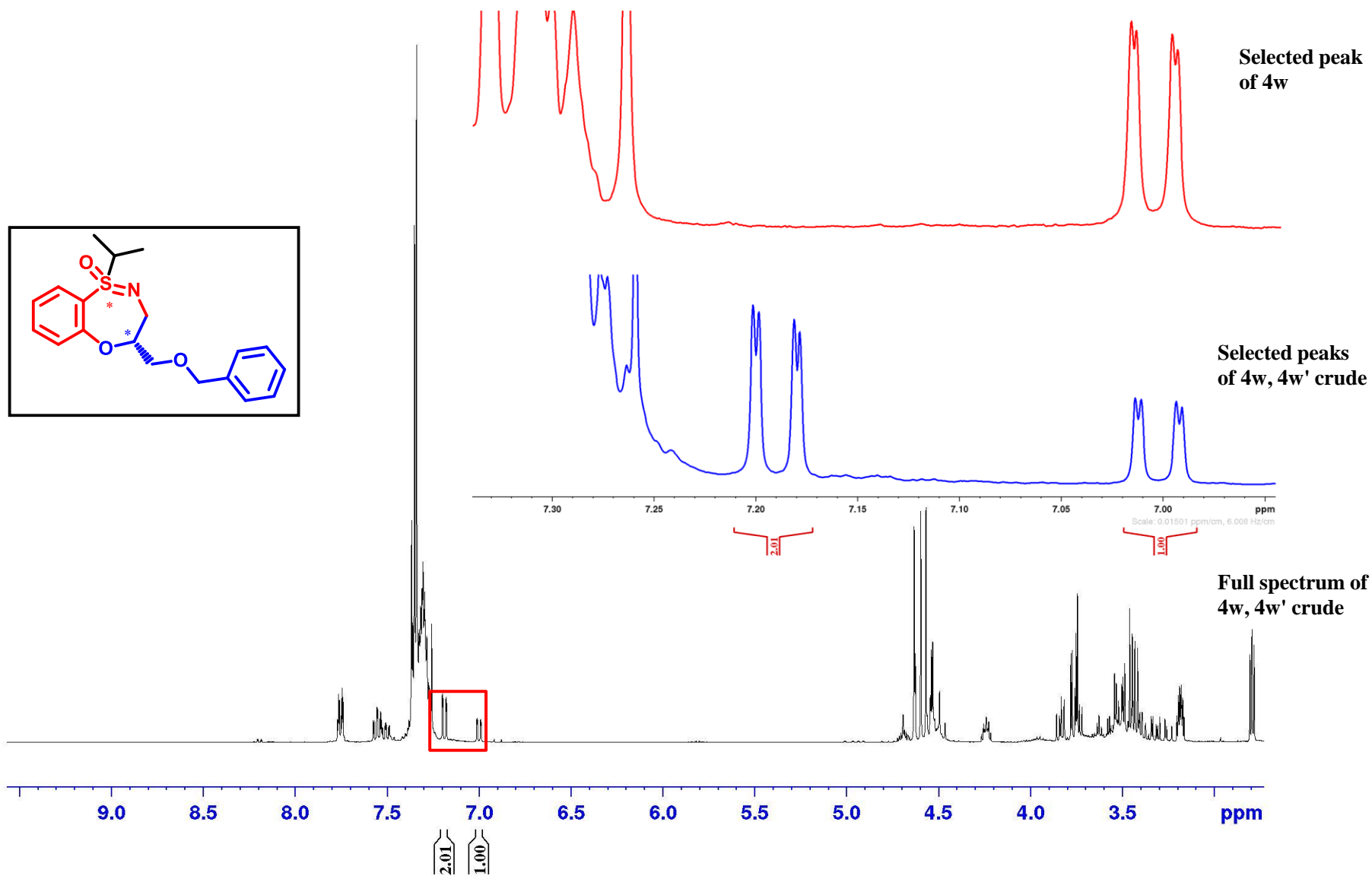
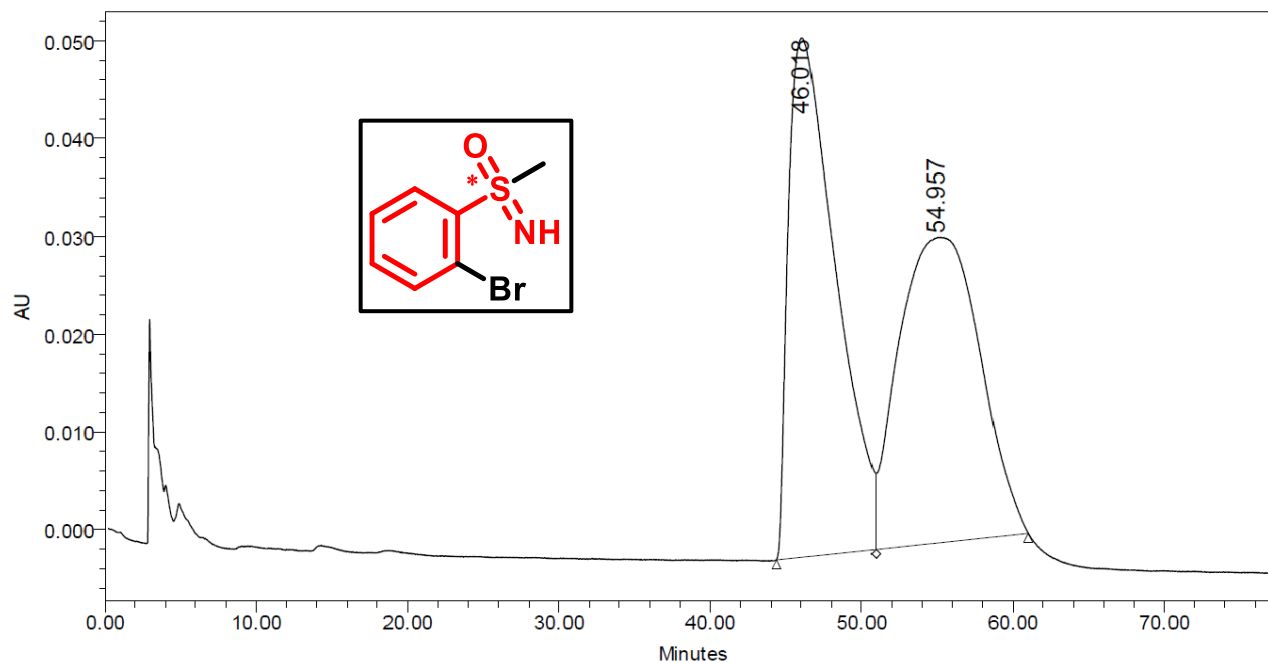


Fig S-188: Diastereomeric ratio calculation of Compound 4w and 4w' by ¹H NMR Spectra (400 MHz, CDCl₃)

SAMPLE INFORMATION

Sample Name:	AB6-5%-IPA-1-CHI PAKIA	Acquired By:	System
Sample Type:	Unknown	Sample Set Name:	
Vial:	1	Acq. Method Set:	ana_5_95
Injection #:	1	Processing Method:	AB6 230 NM
Injection Volume:	10.00 ul	Channel Name:	230.0nm
Run Time:	120.0 Minutes	Proc. Chnl. Descr.:	PDA 230.0 nm
Date Acquired:	12/18/2021 9:08:56 PM IST		
Date Processed:	12/21/2021 6:10:06 PM IST		

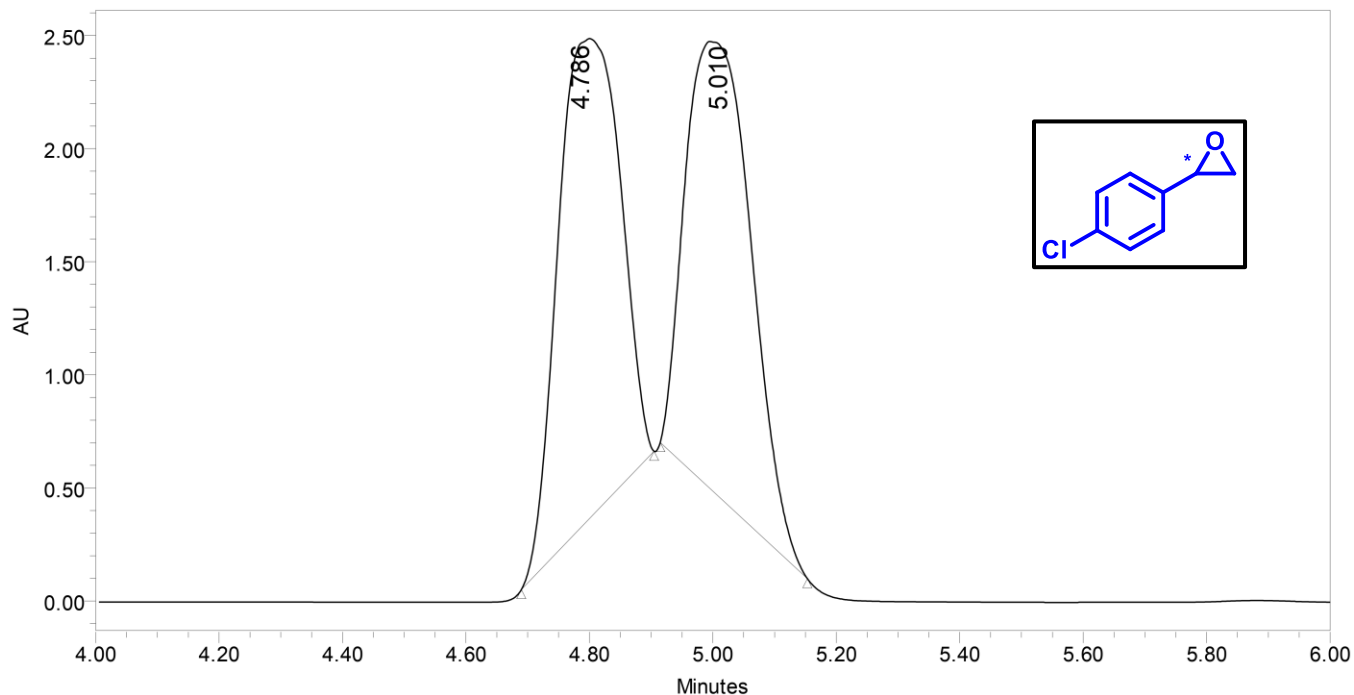


	RT	Area	% Area	Height
1	46.018	11522945	49.63	53121
2	54.957	11694455	50.37	31312

Fig S-189: Purity data (HPLC)of Compound **1a**

SAMPLE INFORMATION

Sample Name:	sp21-2-5%-IPA-1-CHIPAK IG	Acquired By:	System
Sample Type:	Unknown	Sample Set Name:	
Vial:	1	Acq. Method Set:	ana_5_95
Injection #:	1	Processing Method:	AB 21
Injection Volume:	10.00 ul	Channel Name:	230.0nm
Run Time:	120.0 Minutes	Proc. Chnl. Descr.:	PDA 230.0 nm
Date Acquired:	3/27/2022 5:07:52 PM IST		
Date Processed:	3/27/2022 5:43:40 PM IST		

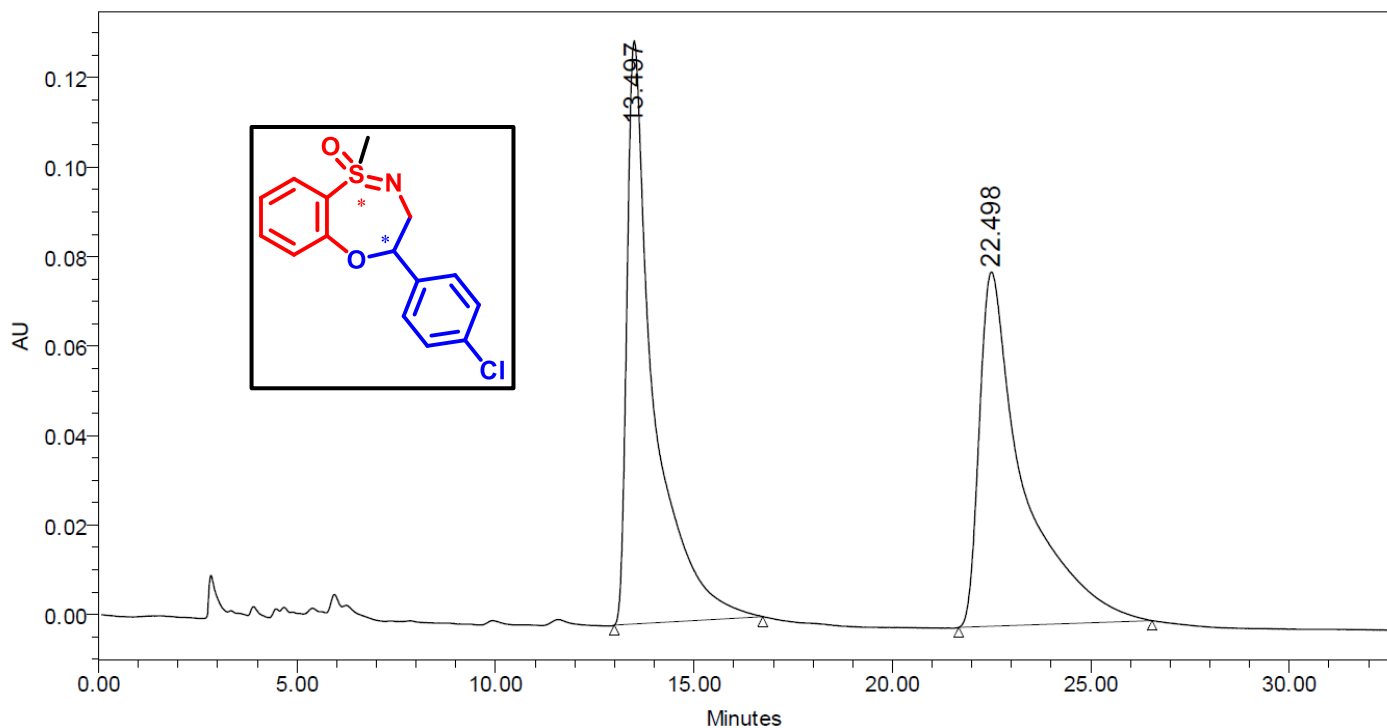


	RT	Area	% Area	Height
1	4.786	14994838	50.07	2140744
2	5.010	14954396	49.93	2000738

Fig S-190: Purity data (HPLC)of Compound 31

SAMPLE INFORMATION

Sample Name:	AB 4P-1-20%IPA-1-CHIPAK IA	Acquired By:	System
Sample Type:	Unknown	Sample Set Name:	
Vial:	1	Acq. Method Set:	ana_20_80
Injection #:	1	Processing Method:	AB 4p 230 nm
Injection Volume:	10.00 ul	Channel Name:	230.0nm
Run Time:	500.0 Minutes	Proc. Chnl. Descr.:	PDA 230.0 nm
Date Acquired:	1/6/2022 9:22:37 PM IST		
Date Processed:	1/6/2022 9:56:06 PM IST		

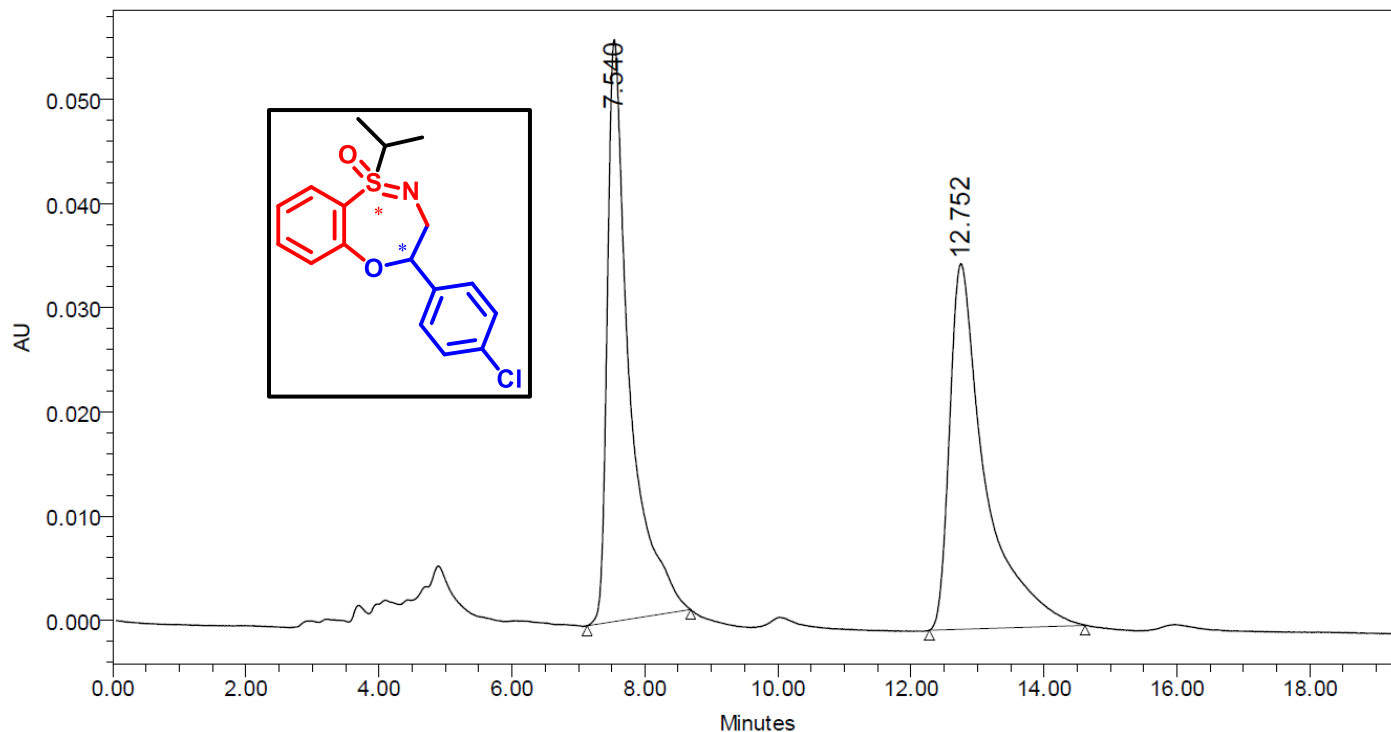


	RT	Area	% Area	Height
1	13.497	6104783	50.00	130277
2	22.498	6105167	50.00	79120

Fig S-191: Purity data (HPLC)of Compound 4p

SAMPLE INFORMATION

Sample Name:	AB 4q-1-30%IPA-1-CHIPAK IA	Acquired By:	System
Sample Type:	Unknown	Sample Set Name:	
Vial:	1	Acq. Method Set:	ana_30_70
Injection #:	1	Processing Method:	AB 4q 230 nm
Injection Volume:	10.00 ul	Channel Name:	230.0nm
Run Time:	500.0 Minutes	Proc. Chnl. Descr.:	PDA 230.0 nm
Date Acquired:	1/6/2022 10:13:08 PM IST		
Date Processed:	1/6/2022 10:32:01 PM IST		

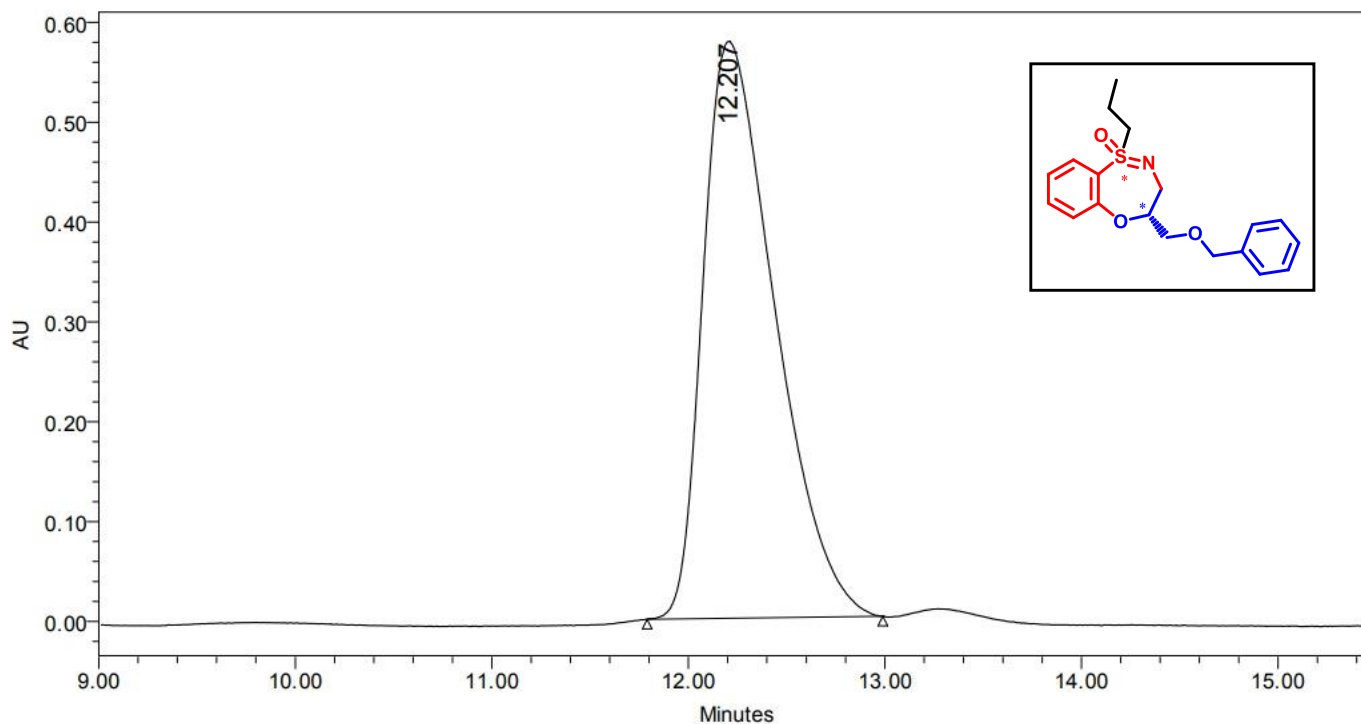


	RT	Area	% Area	Height
1	7.540	1297109	50.00	55857
2	12.752	1297201	50.00	35090

Fig S-192: Purity data (HPLC)of Compound **4q**

SAMPLE INFORMATION

Sample Name:	AB-3-1a-1-20%-1-CHICELOJH	Acquired By:	System
Sample Type:	Unknown	Sample Set Name:	
Vial:	1	Acq. Method Set:	ana_20_80
Injection #:	1	Processing Method:	AB 3 1a
Injection Volume:	10.00 ul	Channel Name:	210.0nm
Run Time:	120.0 Minutes	Proc. Chnl. Descr.:	PDA 210.0 nm
Date Acquired:	5/11/2022 10:35:59 PM IST		
Date Processed:	5/11/2022 11:03:34 PM IST		

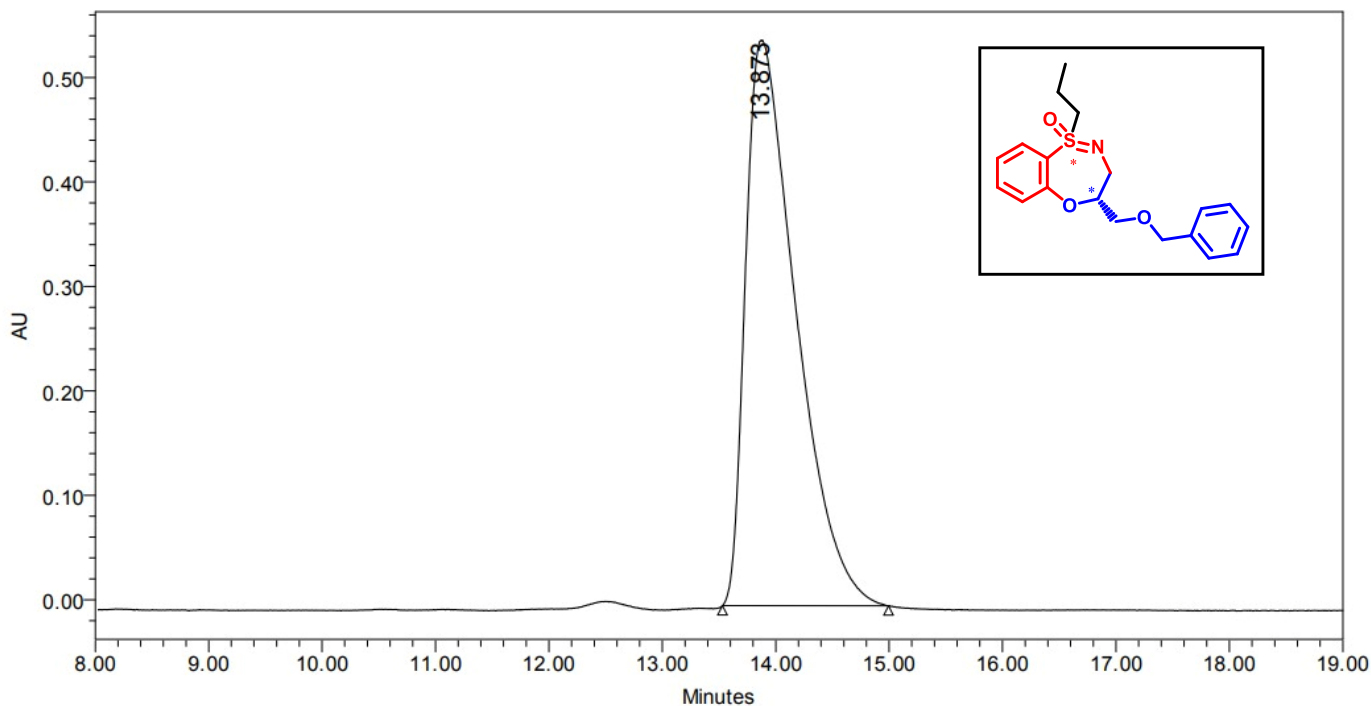


	RT	Area	% Area	Height
1	12.207	14662005	100.00	577617

Fig S-193: Purity data (HPLC)of Compound **4z**

SAMPLE INFORMATION

Sample Name:	AB-3-1b-1-20%-1-CHICELOJH	Acquired By:	System
Sample Type:	Unknown	Sample Set Name:	
Vial:	1	Acq. Method Set:	ana_20_80
Injection #:	1	Processing Method:	AB 3 1b
Injection Volume:	10.00 ul	Channel Name:	210.0nm
Run Time:	120.0 Minutes	Proc. Chnl. Descr.:	PDA 210.0 nm
Date Acquired:	5/11/2022 11:07:38 PM IST		
Date Processed:	5/11/2022 11:27:48 PM IST		



	RT	Area	% Area	Height
1	13.873	16736013	100.00	541357

Fig S-194: Purity data (HPLC)of Compound (R,S)-4z'