Supplementary Information (SI) for Green Chemistry.
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Supporting Information

For

Diastereoselective Construction of Complex Spiroheterocycles via γ-C(sp³)–H Functionalization of 1,1-Disubstituted Cyclohexanes with Diverse Bifunctional Reagents

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1. General Information

^{1}H and ^{13}C NMR spectra were recorded on a Varian Unity Plus or Mercury Plus 400 MHz spectrometer. Chemical shifts (δ) are reported in ppm, and coupling constants (J) in Hz. $CDCl_3$ was used as the solvent. ^1H NMR signals are referenced to TMS (0.00 ppm), and ^{13}C NMR signals to $CDCl_3$ (77.0 ppm). The following abbreviations are used: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, ddd = doublet of doublet of doublets, dt = doublet of triplets, td = triplet of doublets, m = multiplet. Mass spectra and high-resolution mass spectra (HRMS) were obtained using an LTQ Orbitrap XL (Thermo Fisher Scientific) at National Taiwan Normal University. Melting points were measured with an EZ-Melt automated melting point apparatus. All synthesized compounds showed ^{1}H NMR spectra consistent with the proposed structures. Reaction progress and product mixtures were routinely monitored by TLC using Merck silica gel 60 F254 aluminum plates. Column chromatography was performed using 230–400 mesh silica gel (Merck) with hexane/ethyl acetate mixtures as eluents. Solvents and Some starting materials were obtained from commercial suppliers (e.g., Sigma-Aldrich, TCI, or Alfa Aesar) with appropriate purities. Reagents were used as received without further purification, unless otherwise stated.

1.1. Light Sources

Reactions were carried out using 40 W blue LED lamp (Kessil A160WE Controllable LED Aquarium Light) purchased from Kessil via Amazon (Taiwan). See the following links for more details. https://www.kessil.com/aquarium/saltwater_A160.php
https://www.kessil.com/support/downloadfiles/aquarium/A160WE_UserManual.pdf

40 W blue LED lamp (PR160L-456 nm) purchased from Fiberoptics & DiCon Lighting, Kaohsiung, Taiwan. See the following f links for more details.

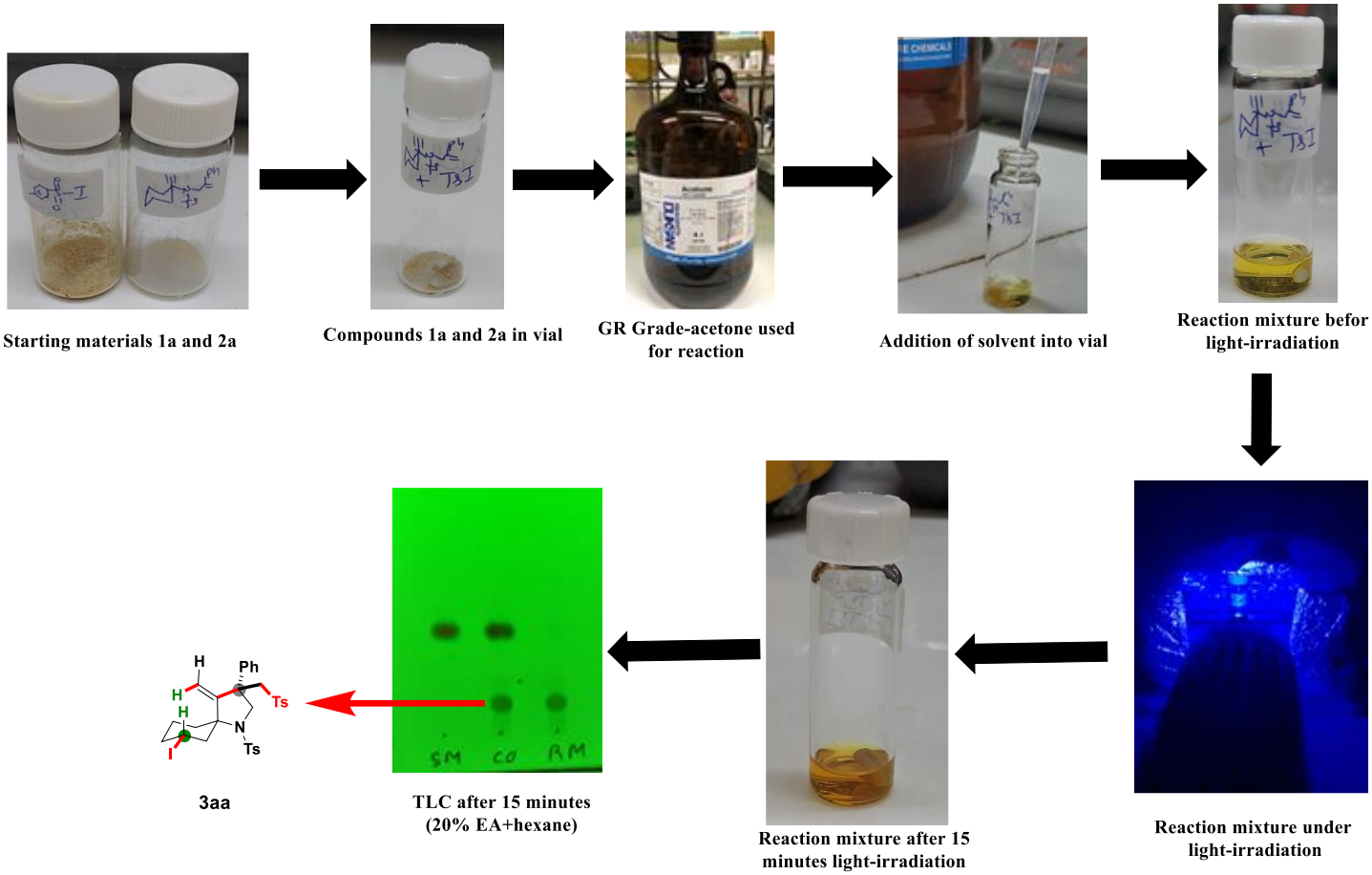
https://kessil.com/science/PR160L.php

1.2. Blue LED Emission Spectra

Emission spectra were measured using Ocean Optics USB 2000+ Spectrometer. Spectra were normalized to 1.0 at the emission maximum. These emission spectra were provided by Miss Angela Liou, Sales Specialist, DiCon Fiberoptics & DiCon Lighting, aliou@diconfiberoptics.com, Kaohsiung, Taiwan, +886 7 815-8055 Ext 485, DiCon Brands - Kessil | Fiilex | Cielux.

2. Handling of reaction and blue LED technical details

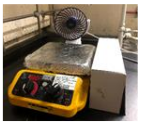
2.1 Reaction setup under blue LED irradiation



Supplementary Figure 1. Reaction setup for visible-light-induced transformation under blue LED irradiation. The reaction employs substrates **1a** and **2a**, and the progress is monitored by thin-layer chromatography (TLC).

2.2. Complete reaction setup and technical details of the blue LED light source

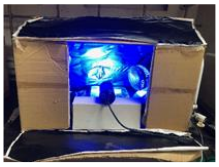
Front side view of stirrer with fan (For clarity we took picture without cardboard box)



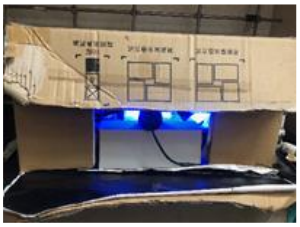
side view of stirrer with fan (For clarity we took picture without cardboard box)

Upper view of stirrer with fan (For clarity we took picture without cardboard box)

The reaction setup under cardboard box



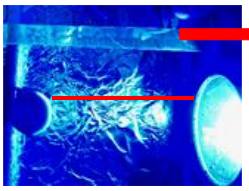
Outside view of the setup(door open)



Outside view of the setup(door closed)

Cooling of the reaction under light irradiation

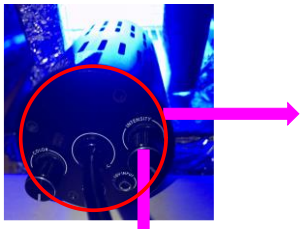
1 cm hight (light source)



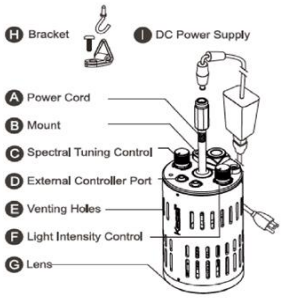
8.5 cm distance from light source to reaction vial

A160WE Tuna Blue

Manufacturing and other technical details on light source



100% intensity of blue LED was used in our reaction



A160WE Tuna Blue power adapter

This A160WE Tuna Blue LED purched from amazon Taiwan website

Technical Specifications

Specifications	
Dimensions	4" x 2.48" (H x D)
Unit Weight	0.69 lb / 0.32kg
Spectrum	Deep Ocean Blue to Sky
Coverage	Up to 24" surface diameter
Power Adapter	100-240V AC (input) 19-24V DC (output)
Power Consumption	40W

https://www.kessil.com/aquarium/saltwater_A160.php

https://www.kessil.com/support/downloadfiles/aquarium/A160WE_UserManual.pdf

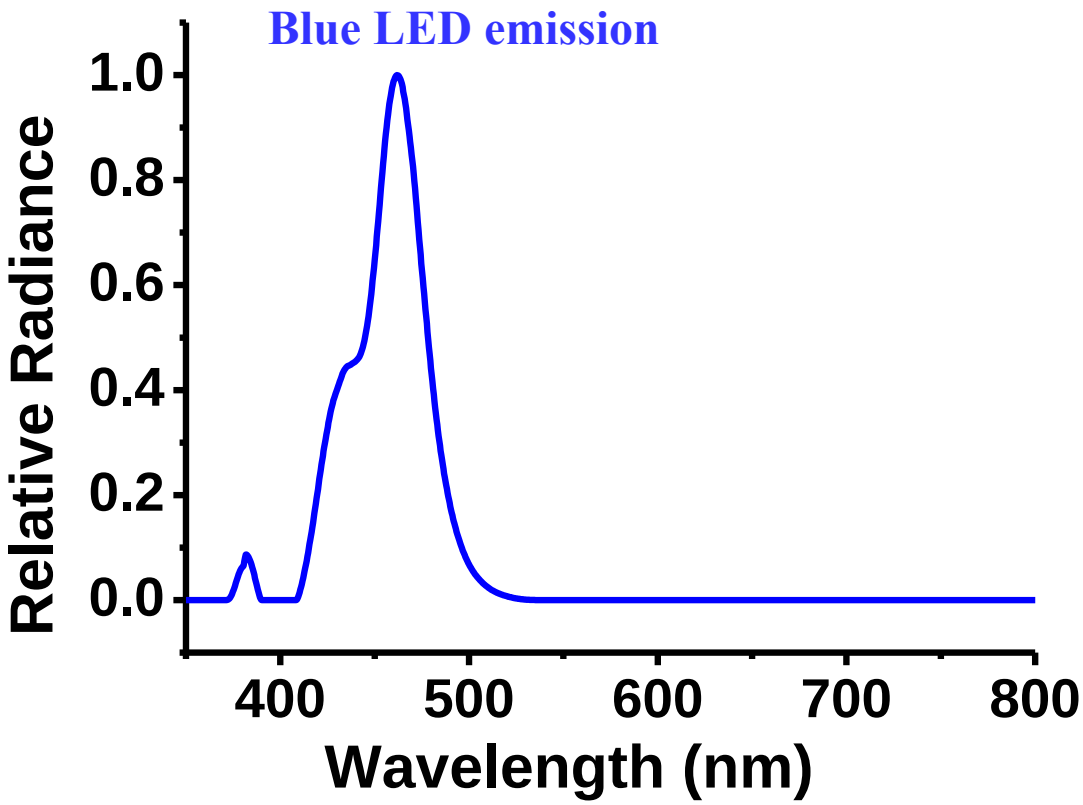
Supplementary Figure 2. Complete reaction setup and technical details of the 40 W Blue LED (Kessil A160WE Controllable LED Aquarium Light).

3. Mechanistic investigations

3.1. Instrument used to measure Blue LED emission spectra

The emission spectra of the blue LED were measured using an Ocean Optics USB 2000+ spectrometer. The spectra were normalized to an intensity of 1.0 at the emission maximum. The emission data were kindly provided by Miss Angela Liou (Sales Specialist, DiCon Fiberoptics & DiCon Lighting; aliou@diconfiberoptics.com; Kaohsiung, Taiwan; +886 7 815-8055 Ext. 485), representing DiCon Brands – Kessil | Fiilex | Cielux.

3.1.1. 40 W Kessil A160WE Tuna Blue LED emission spectra

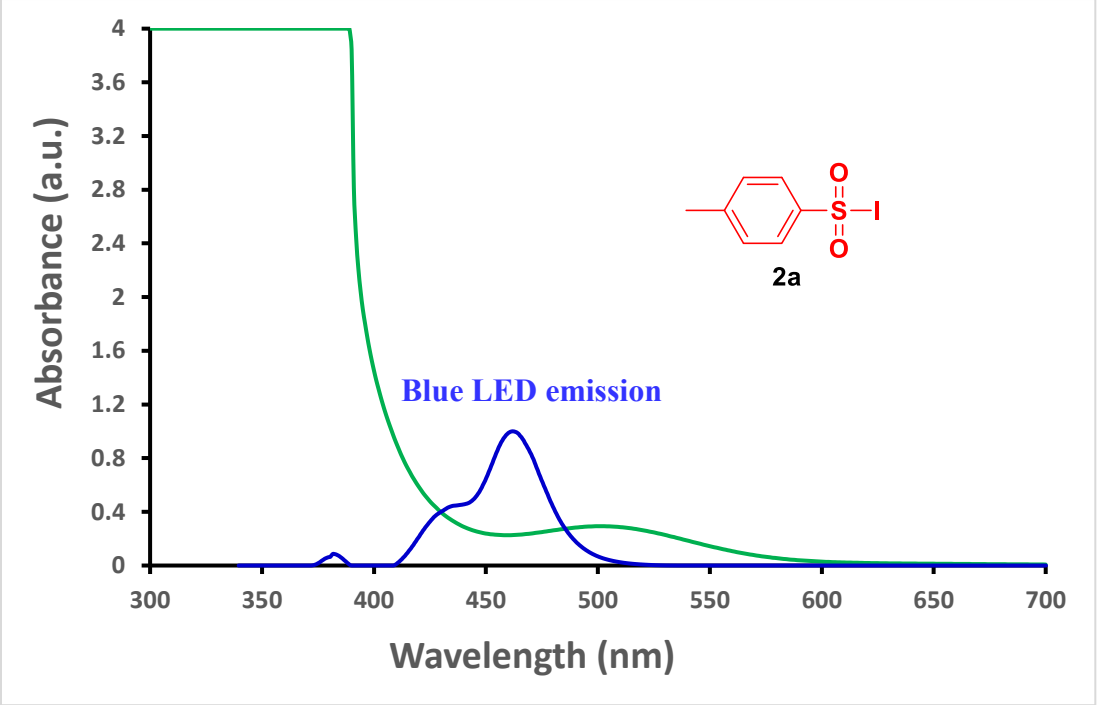


Supplementary Figure 3. Emission spectrum from a 40 W Kessil A160WE Tuna Blue LED shown as blue color line with emission maximum at λ max = 462 nm flanked by a second peak at λ = 382 nm.

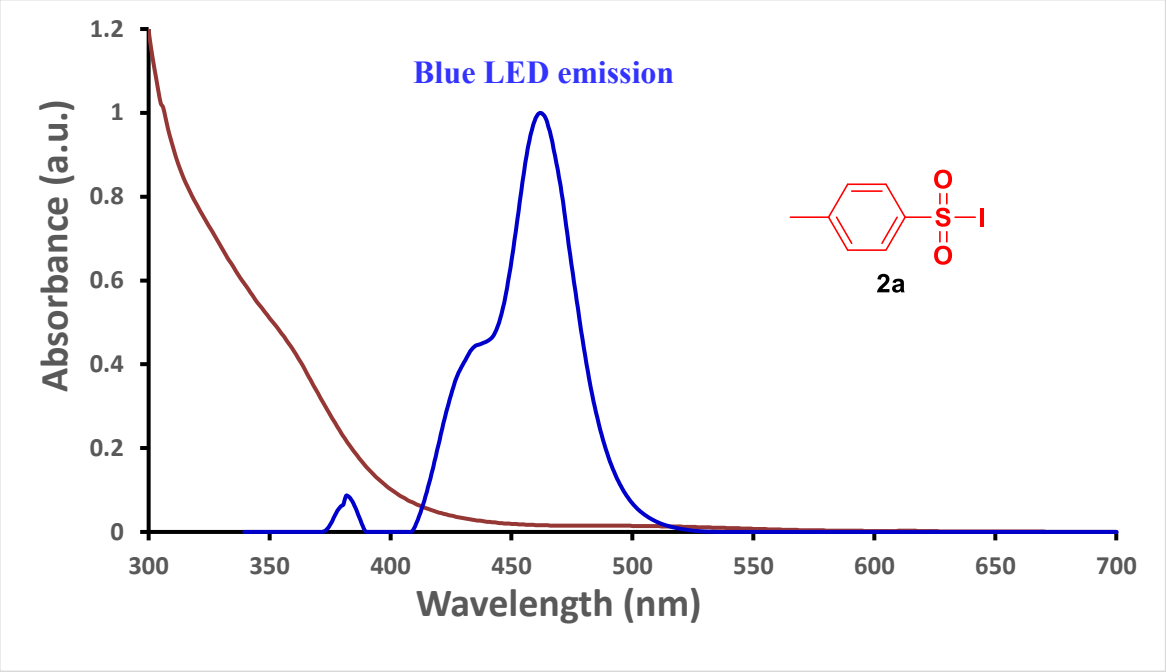
3.2. Absorption spectra of compounds 2a

UV-vis absorption spectra were recorded using a Shimadzu UV-1900 UV-Vis spectrophotometer. Measurements were carried out in 1 cm path length quartz cuvettes (dimensions: 1 cm × 4.4 cm), each filled with 4.0 mL of the sample solution. The sample solutions were prepared using HPLC-grade solvents under ambient conditions (open air).

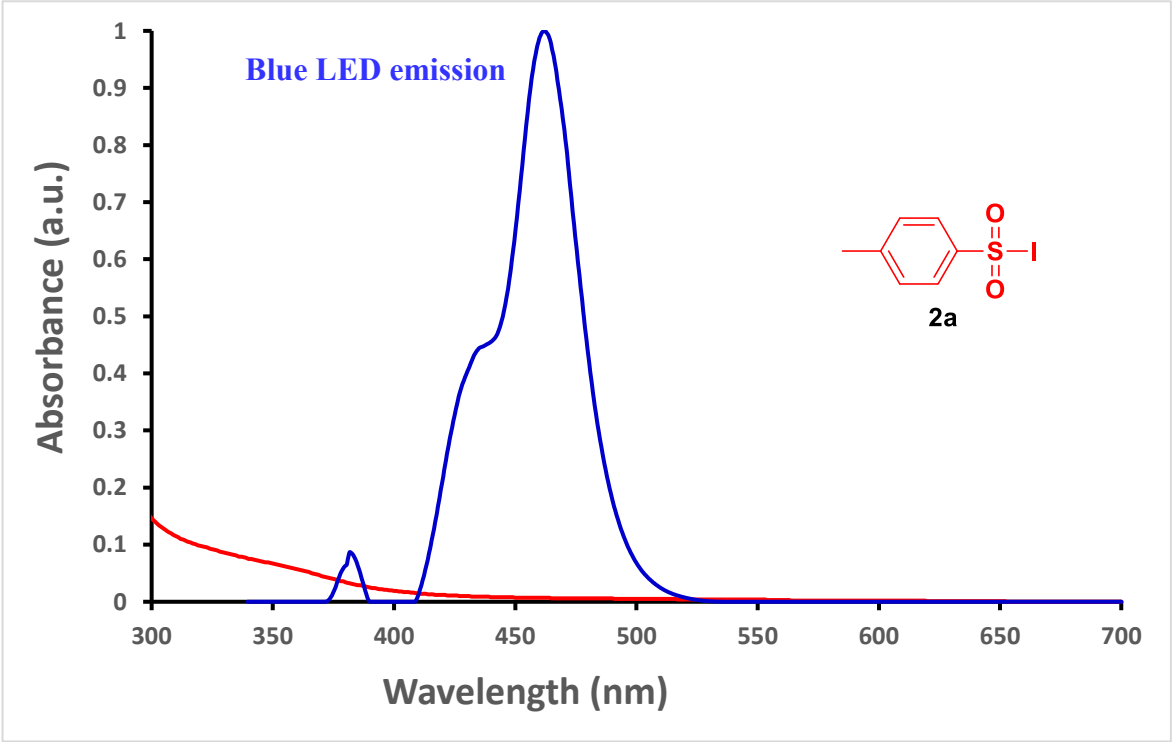
3.2.1. Absorption spectra of 4-methylbenzenesulfonyl iodide (2a) in DCM solvent



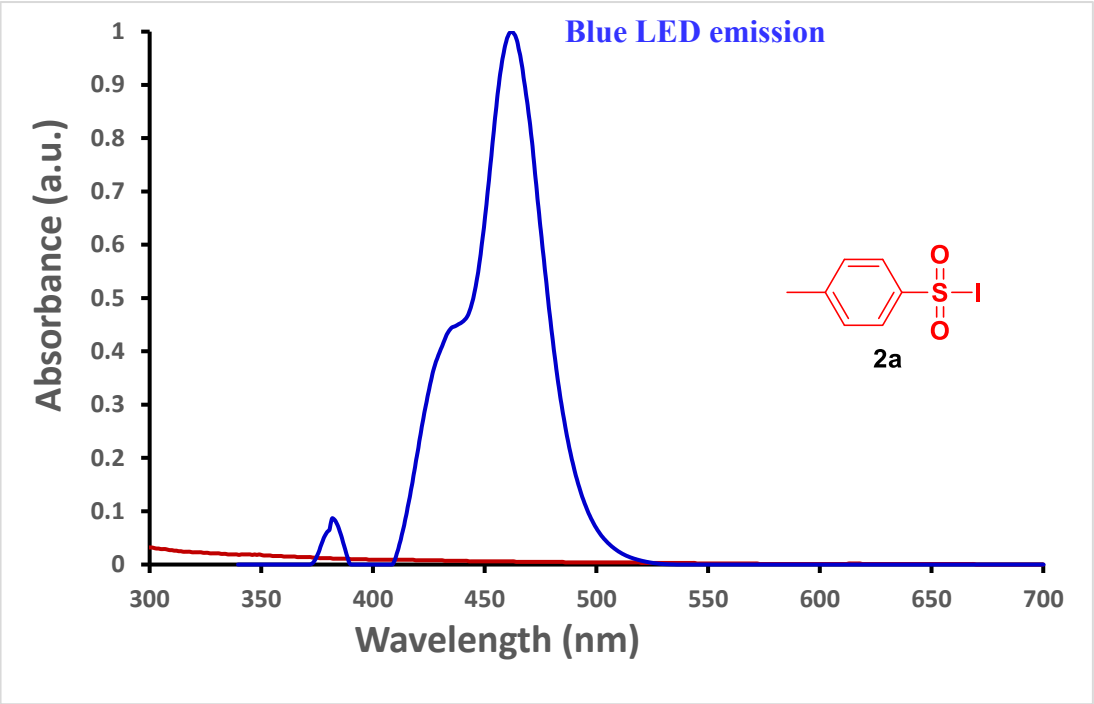
Supplementary Figure 4. Absorption spectra of 4-methylbenzenesulfonyl iodide (**2a**) (10^{-2} M) in DCM, and blue LED emission.



Supplementary Figure 5. Absorption spectra of 4-methylbenzenesulfonyl iodide (**2a**) (10^{-3} M) in DCM, and blue LED emission.

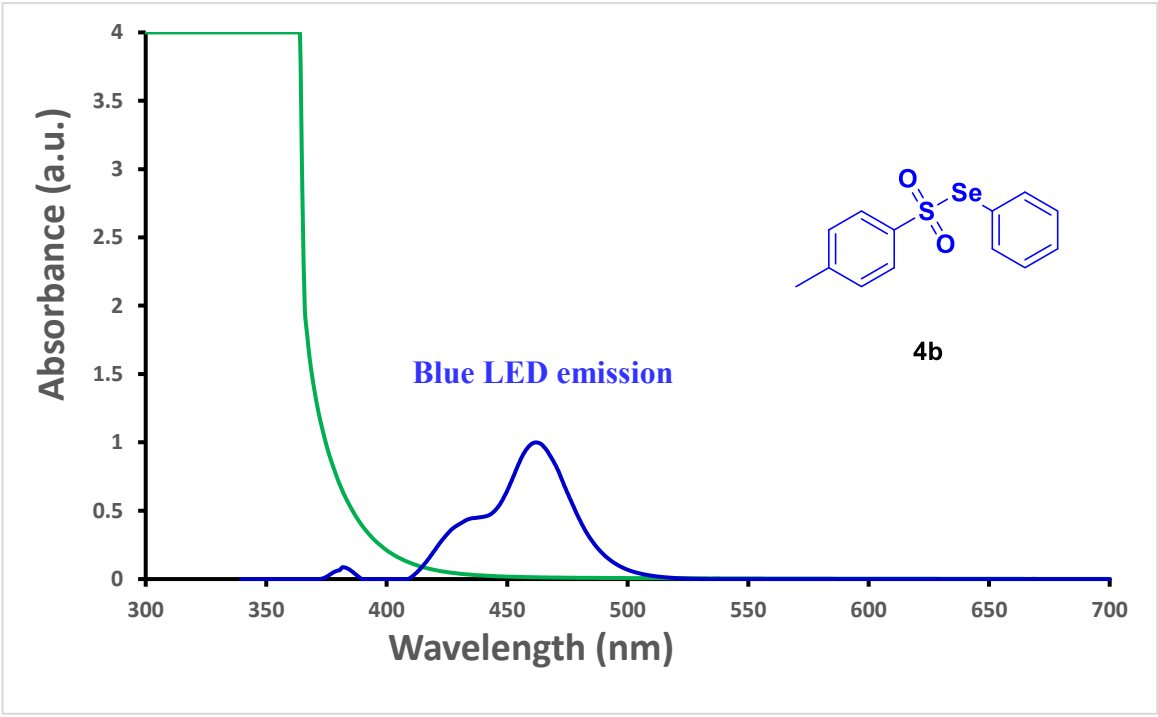


Supplementary Figure 6. Absorption spectra of 4-methylbenzenesulfonyl iodide (**2a**) (10^{-4} M) in DCM, and blue LED emission.

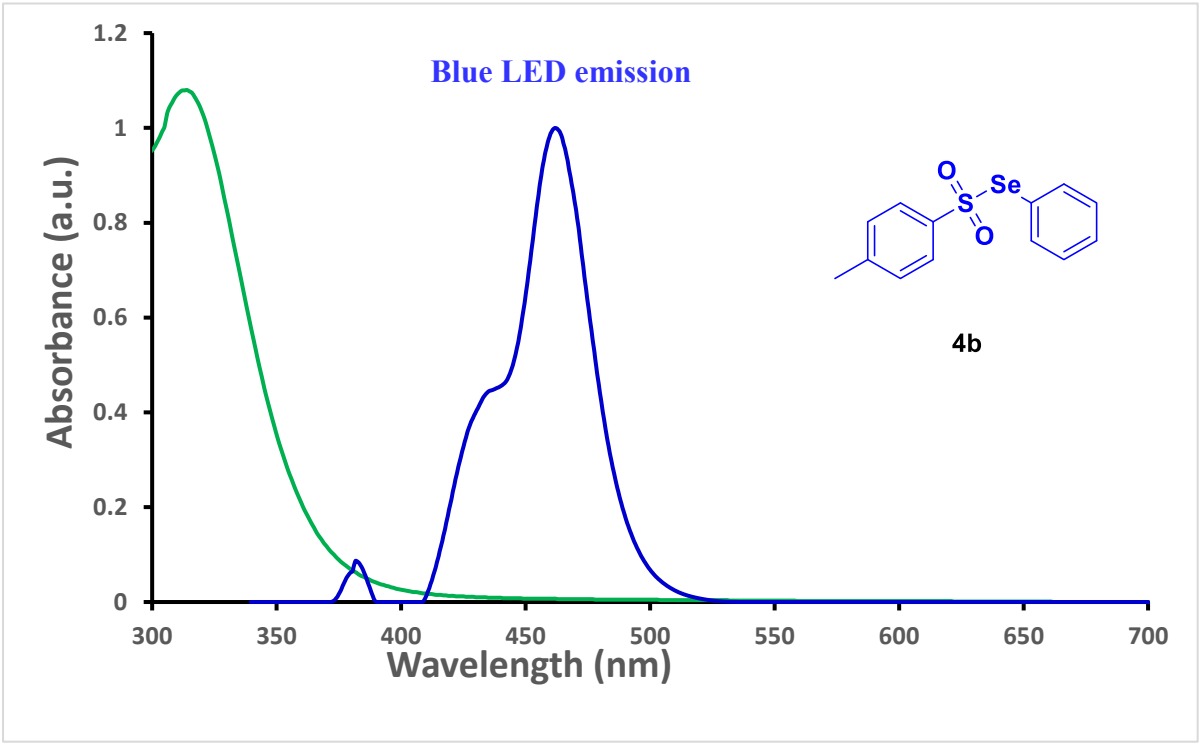


Supplementary Figure 7. Absorption spectra of 4-methylbenzenesulfonyl iodide (**2a**) (10^{-5} M) in DCM, and blue LED emission.

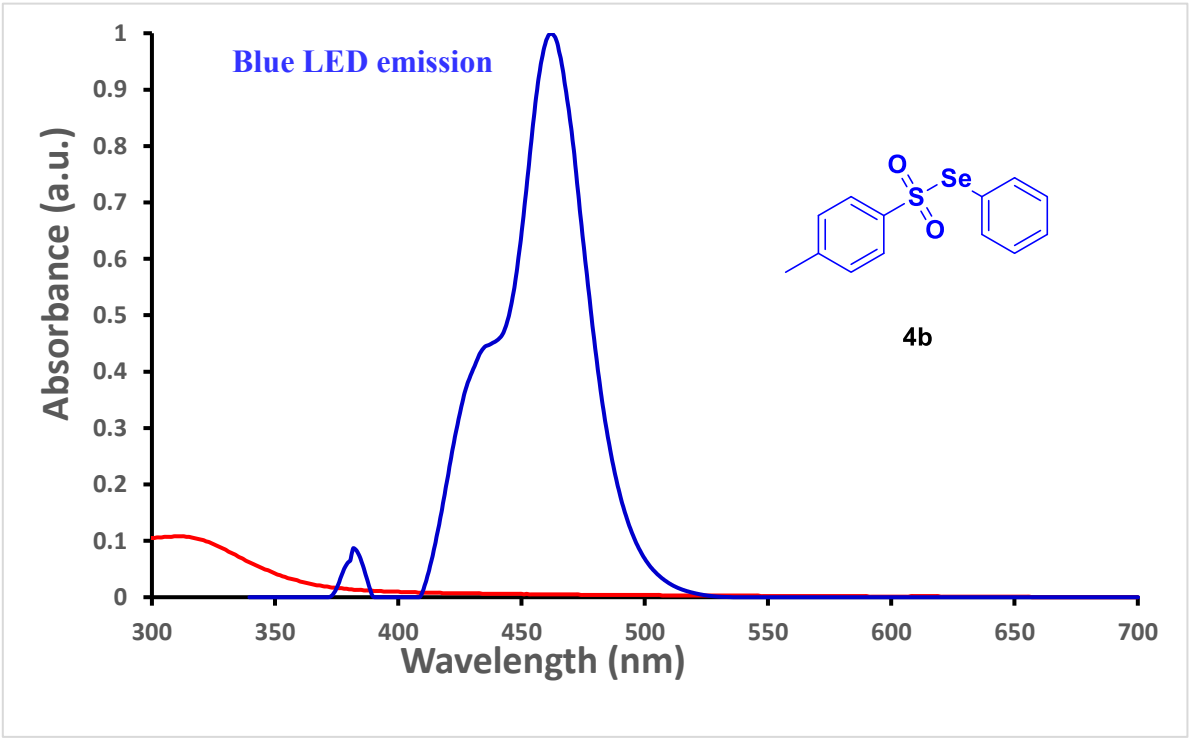
3.3. Absorption spectra of Se-phenyl 4-methylbenzenesulfonoselenoate (4b) in DCM



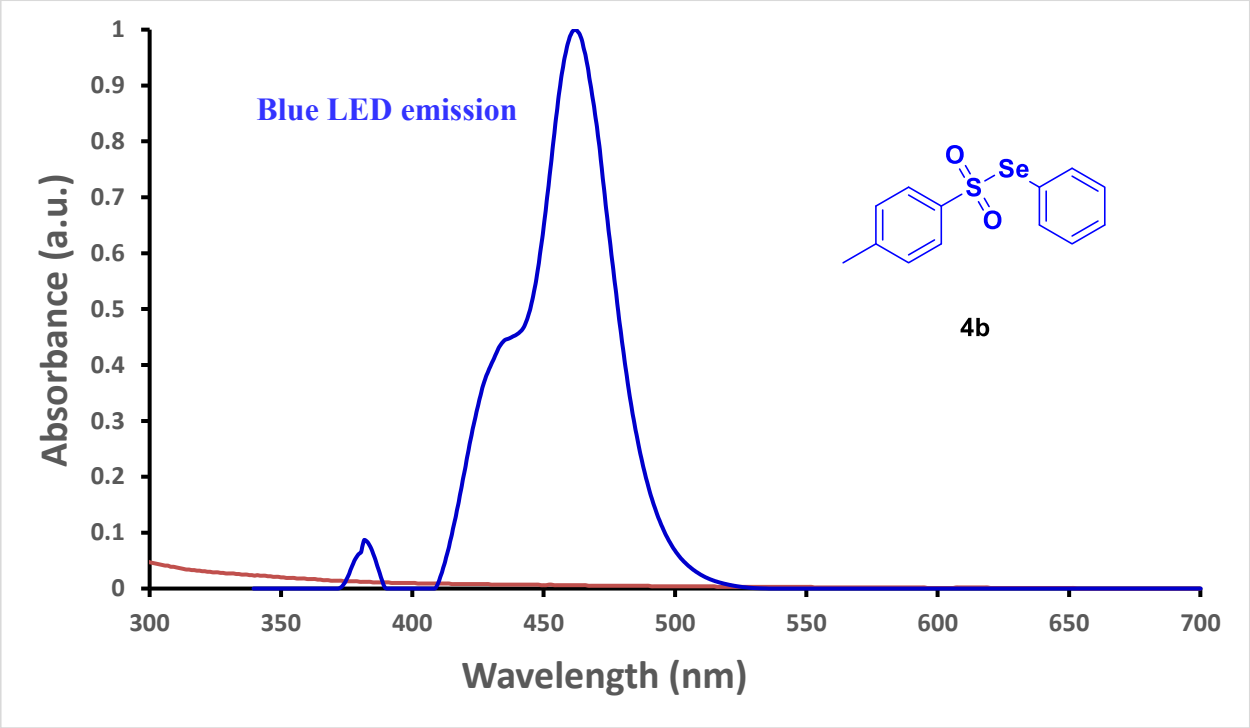
Supplementary Figure 8. Absorption spectra of Se-phenyl 4-methylbenzenesulfonoselenoate (**4b**) (10^{-2} M) in DCM, and blue LED emission.



Supplementary Figure 9. Absorption spectra of Se-phenyl 4-methylbenzenesulfonoselenoate (**4b**) (10^{-3} M) in DCM, and blue LED emission.



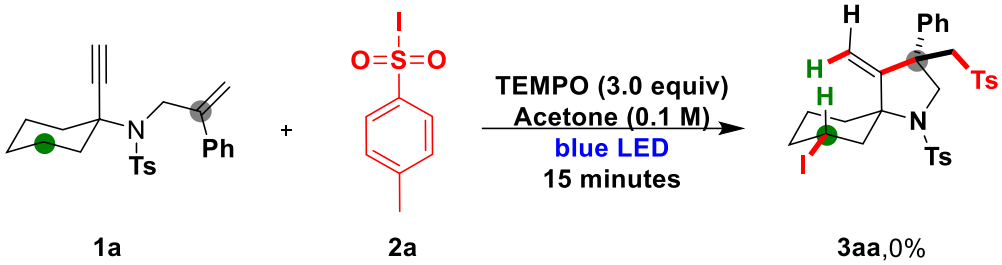
Supplementary Figure 10. Absorption spectra of Se-phenyl 4-methylbenzenesulfonoselenoate (**4b**) (10^{-4} M) in DCM, and blue LED emission.



Supplementary Figure 11. Absorption spectra of Se-phenyl 4-methylbenzenesulfonoselenoate (**4b**) (10^{-5} M) in DCM, and blue LED emission.

3.3. Radical inhibitors studies in the synthesis of (3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3aa)

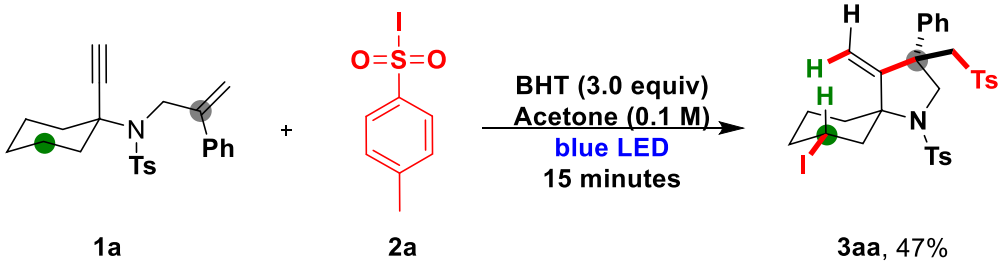
3.3.1 Radical experiment with 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO)



1a **2a** **3aa**,0%

An oven-dried 8 mL screw-capped vial equipped with a magnetic stir bar was charged with N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (**1a**, 0.10 mmol, 1.0 equiv), 4-methylbenzenesulfonyl iodide (**2a**, 0.11 mmol, 1.1 equiv), TEMPO (0.30 mmol, 3.0 equiv), and acetone (0.1 M) was added under ambient atmosphere. The reaction mixture was stirred and irradiated with a blue LED at room temperature for 15 min. After 15 min, TLC analysis indicated that the starting material **1a** remained unchanged, and no product formation was observed. The complete inhibition of the reaction in the presence of TEMPO is consistent with a radical-mediated mechanism, as the radical scavenger effectively suppresses the transformation.

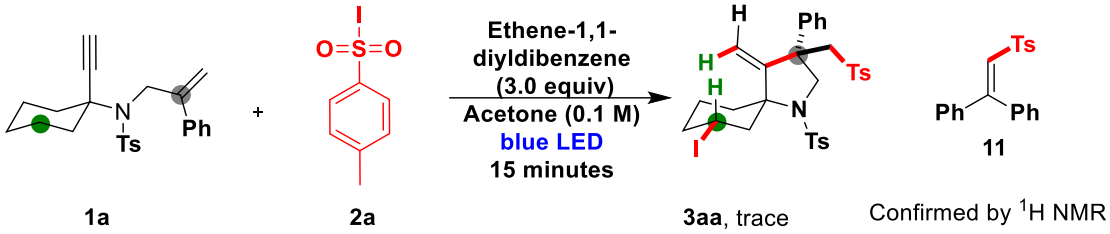
3.3.2 Radical experiment with 22,6-Di-tert-butyl-4-methylphenol (BHT)



1a **2a** **3aa**, 47%

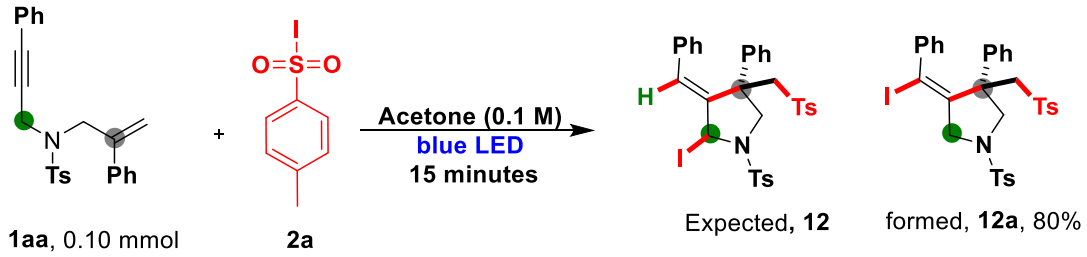
An oven-dried 8 mL screw-capped vial equipped with a magnetic stir bar was charged with N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (**1a**, 0.10 mmol, 1.0 equiv), 4-methylbenzenesulfonyl iodide (**2a**, 0.11 mmol, 1.1 equiv), BHT (0.30 mmol, 3.0 equiv), and acetone (0.1 M) was added under ambient atmosphere. The reaction mixture was stirred and irradiated with a blue LED at room temperature for 15 min. After 15 min, TLC analysis indicated the formation of the desired spirocyclic product **3aa** in 47% yield. These results are consistent with a radical-mediated mechanism, as the radical scavenger BHT partially inhibits the transformation.

3.3.3 Radical experiment with ethene-1,1-diyldibenzene



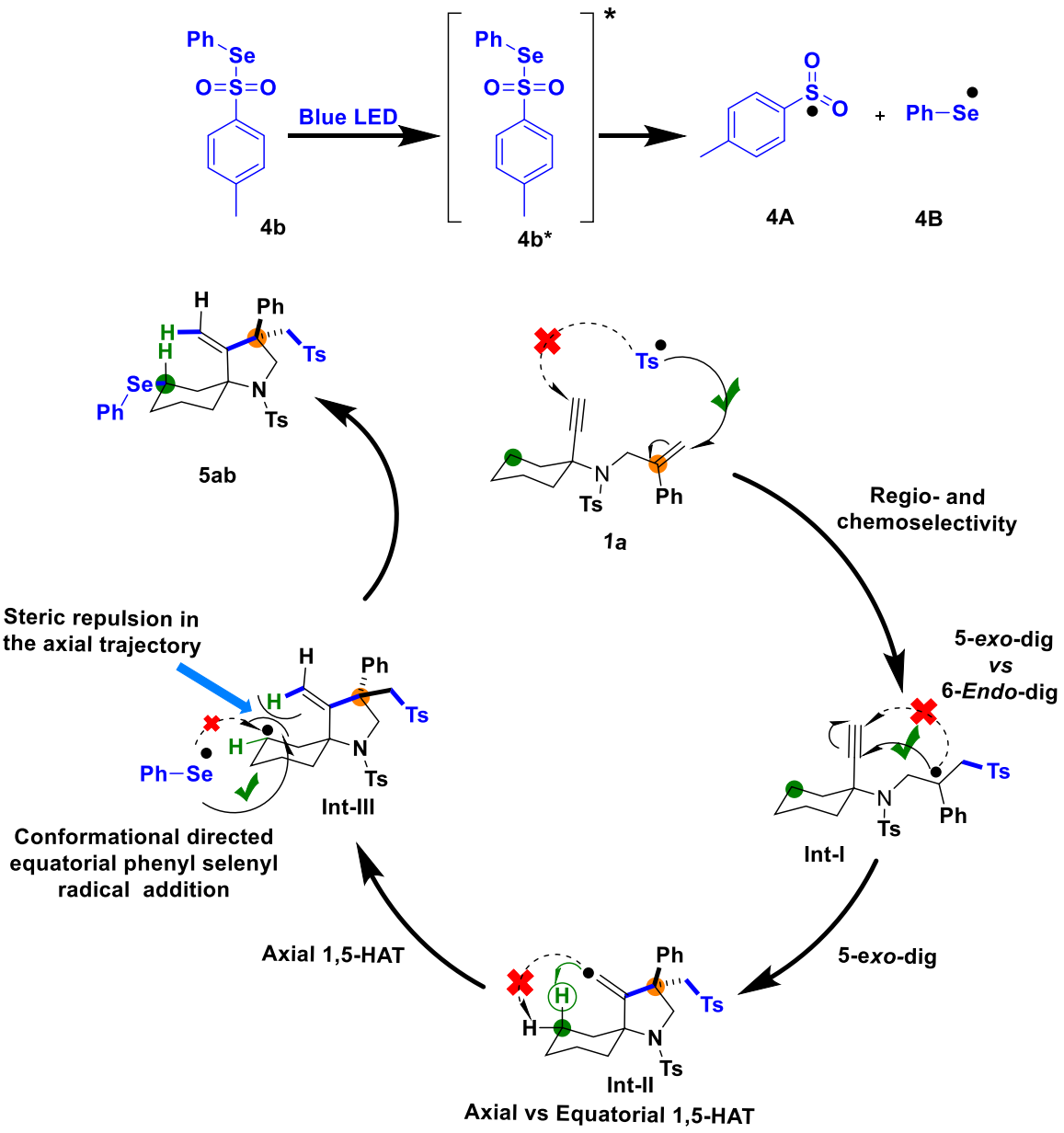
An oven-dried 8 mL screw-capped vial equipped with a magnetic stir bar was charged with N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (**1a**, 0.10 mmol, 1.0 equiv.), 4-methylbenzenesulfonyl iodide (**2a**, 0.11 mmol, 1.1 equiv.), ethene-1,1-diyldibenzene (0.30 mmol, 3.0 equiv), and acetone (0.1 M) was added under ambient atmosphere. The reaction mixture was stirred and irradiated with a blue LED at room temperature for 15 min. After 15 min, TLC analysis showed only trace formation of the spirocyclic product **3aa**. In addition, formation of the tosyl radical adduct with ethene-1,1-diyldibenzene was confirmed by ^{1}H NMR spectroscopy. These results are consistent with a radical-mediated mechanism, as the radical inhibitor effectively suppresses product formation and traps the tosyl radical.

3.3.4 Synthesis of (*Z*)-4-(iodo(phenyl)methylene)-3-phenyl-1-tosyl-3-(tosylmethyl)pyrrolidine (12a) from 4-methyl-N-(2-phenylallyl)-N-(3-phenylprop-2-yn-1-yl)benzenesulfonamide



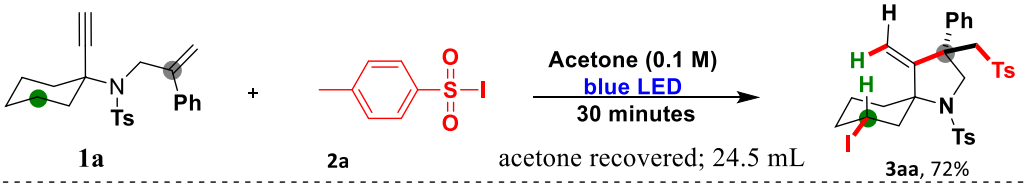
An oven-dried 8 mL screw-capped vial equipped with a magnetic stir bar was charged with 4-methyl-N-(2-phenylallyl)-N-(3-phenylprop-2-yn-1-yl)benzenesulfonamide (**1aa**, 0.10 mmol, 1.0 equiv.) and 4-methylbenzenesulfonyl iodide (**2a**, 0.11 mmol, 1.1 equiv.), and acetone (1.0 mL, 0.1 M) was added.. The reaction mixture was stirred and irradiated with a blue LED at room temperature for 15 min. After 15 min, TLC and ^{1}H NMR analysis indicated no formation of the 1,3-HAT product **12**. Instead, the major product was identified as (*Z*)-4-(iodo(phenyl)methylene)-3-phenyl-1-tosyl-3-(tosylmethyl)pyrrolidinevinyl iodide (**12a**; 80%), resulting from direct trapping of the vinyl radical by an iodine radical. This result demonstrates that the rigid cyclohexyl scaffold is essential for enforcing the spatial proximity required for intramolecular 1,5-HAT, and its absence diverts the reaction toward radical quenching pathways.

4. Mechanism for formation of 5ab



5. Gram-scale synthesis of 3aa from 1a and evaluation of green metrics

Table S1. Gram-scale synthesis and green chemistry assessment of (3aa)



1.0 g, 2.54 mmol	0.748 g, 2.79 mmol	1.24 g
$C_{24}H_{27}NO_2S$; 393.54	$C_7H_7IO_2S$; 282.09	$C_{31}H_{34}INO_4S_2$; 675.64

$$\text{Atom Economy (\%)} = \frac{\text{Molecular weight of desired product}}{\text{Molecular weight of all reactants}} \times 100 = \frac{555.69}{447.55+108.14+150.08} \times 100 = 100$$

$$\text{Atom Efficiency (\%)} = \frac{(\text{\% yield of product} \times \text{\% atom economy})}{100} = \frac{(72 \times 100)}{100} = 72$$

$$\text{Carbon Efficiency (\%)} = \frac{\text{Total amount of carbons in product}}{\text{Total amount of carbons in all reactants}} \times 100 = \frac{31}{24+7} \times 100 = 100$$

$$\text{Reaction Mass Efficiency (\%)} = \frac{\text{Mass of isolated product}}{\text{Total mass of all reactants}} \times 100 = \frac{1.24}{1.0+0.748} \times 100 = 70.93$$

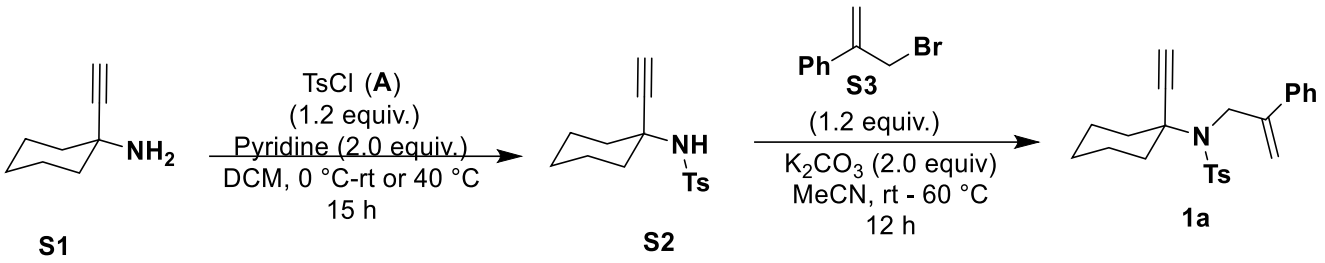
$$\text{E-factor} = \frac{\text{Total waste (g)}}{\text{Total product (g)}} \quad \frac{(1.0+0.748+25.4)-(1.24+24.5)}{1.0} = 1.4 \text{ g waste/1 g product}$$

Table S2. EcoScale penalty points in synthesis of 3aa

Parameter	Penalty points	
1. Yield (100 – 72% yield)/2 = 14	=	14
2. Price of reaction components (to obtain 10 mmol of end product)	=	5
Very expensive (> $50)		
3. Safety		
Acetone; environmentally friendly	=	0
4. Technical setup		
(Simple setup)	=	0
5. Temperature/time		
Room temperature, < 1 h	=	0
6. Workup and purification		
Classical chromatography	=	10
Total of individual penalties		29
EcoScale calculation = 100 - sum of individual penalties: 100-29 = 71		
Following scores: > 75, excellent; > 50, acceptable; and < 50, inadequate.		

6. Preparation of starting materials

6.1. Synthesis of 4-methyl-*N*-(2-phenylallyl)-*N*-(3-phenylprop-2-yn-1-yl)benzenesulfonamide (General procedure 1)[1]



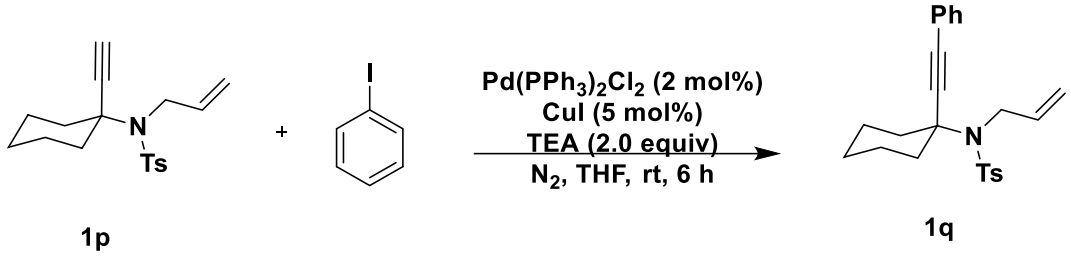
Synthesis of N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (S2):

To a stirred solution of 1-ethynylcyclohexan-1-amine (1.0 equiv) in dichloromethane (DCM) at 0 °C was added pyridine/triethylamine (2.0 equiv), followed by *p*-toluenesulfonyl chloride (TsCl, 1.2 equiv). The reaction mixture was then room temperature/heated to 40 °C (depends on sulfonyl chloride reactivity) and stirred for 15 h. Upon completion, the solvent was removed under reduced pressure, and the residue was dissolved in DCM. The organic layer was washed sequentially with water, saturated aqueous copper(II) sulfate solution, and brine. The combined organic phases were dried over anhydrous sodium sulfate (Na_2SO_4), filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography using hexane/ethyl acetate to afford the N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (**S2**) as an off-white solid.

Synthesis of N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (1a):

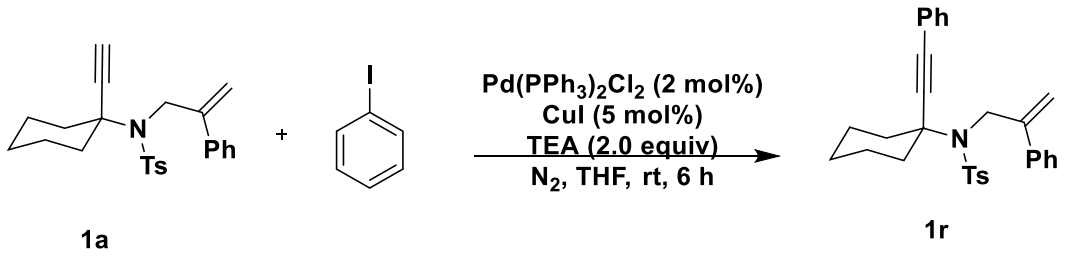
To a stirred solution of compound N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (**S2**) (1.0 equiv) in acetonitrile (MeCN) were added potassium carbonate (K_2CO_3, 2.0 equiv) and 2-phenylallyl bromide **S3** (40.0 mmol). The reaction mixture was heated at 60 °C for 12 h. After completion, the mixture was cooled to room temperature, and the solvent was removed under reduced pressure. The resulting residue was dissolved in ethyl acetate, washed sequentially with water and brine, and dried over anhydrous magnesium sulfate (Na_2SO_4). Filtration and concentration under reduced pressure afforded the crude product, which was purified by column chromatography to yield compound **1a**.

6.2. N-allyl-4-methyl-N-(1-(phenylethynyl)cyclohexyl)benzenesulfonamide (1q) (General procedure A1)



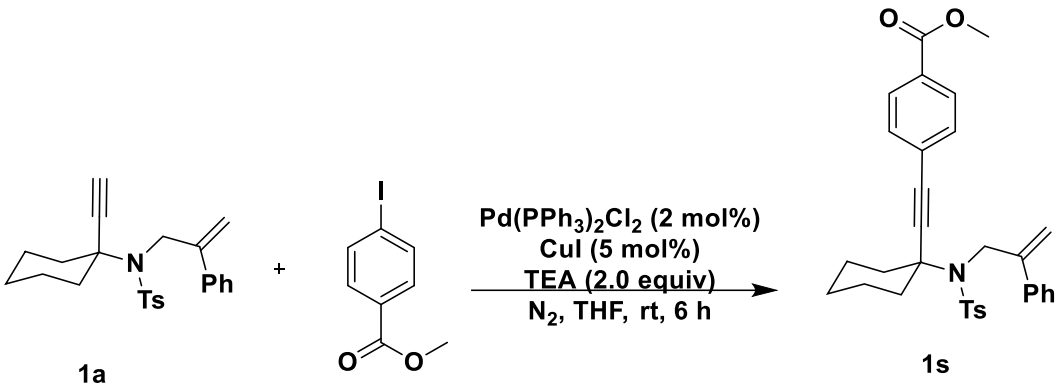
A dried Schlenk flask was charged with N-allyl-N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (150 mg, 0.47 mmol, 1.0 equiv) and iodobenzene (106 mg, 0.52 mmol, 1.1 equiv) in dry THF (5 mL), and the system was purged with nitrogen. $Pd(PPh_3)_2Cl_2$ (6.6 mg, 0.0094 mmol, 2 mol%), CuI (4.5 mg, 0.023 mmol, 5 mol%), and freshly distilled triethylamine (95 mg, 0.94 mmol, 2.0 equiv) were added under a nitrogen atmosphere, and the resulting reaction mixture was stirred at room temperature for 12 h. After completion of the reaction as indicated by TLC, the mixture was diluted with water and extracted with ethyl acetate (3 × 15 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure, and the crude material was purified by column chromatography on silica gel using hexane/ethyl acetate (96:4) as the eluent to afford N-allyl-4-methyl-N-(1-(phenylethynyl)cyclohexyl)benzenesulfonamide (**1q**) as a colorless gummy solid (114 mg, 59% yield).

6.3. 4-methyl-N-(2-phenylallyl)-N-(1-(phenylethynyl)cyclohexyl)benzenesulfonamide (1r) (GP-A1)



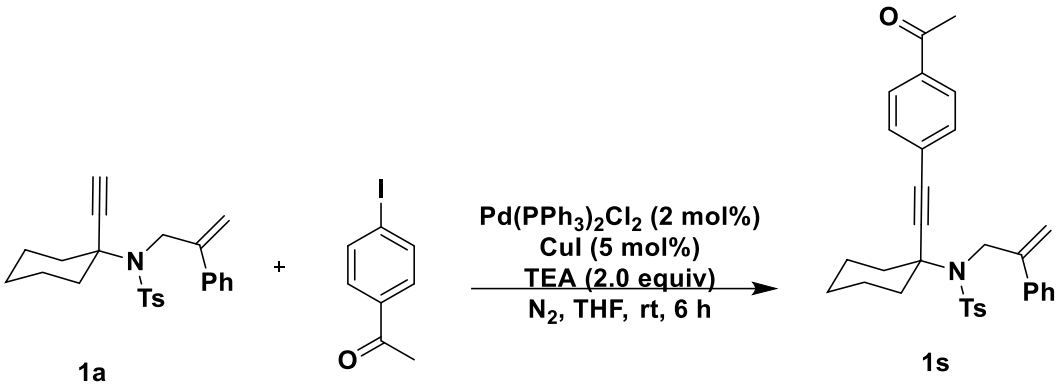
A dried Schlenk flask was charged with N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (150 mg, 0.38 mmol, 1.0 equiv) and iodobenzene (86 mg, 0.42 mmol, 1.1 equiv) in dry THF (5 mL), and the system was purged with nitrogen. $Pd(PPh_3)_2Cl_2$ (5.3 mg, 0.0076 mmol, 2 mol%), CuI (3.6 mg, 0.019 mmol, 5 mol%), and freshly distilled triethylamine (77 mg, 0.76 mmol, 2.0 equiv) were added under a nitrogen atmosphere, and the resulting reaction mixture was stirred at room temperature for 12 h. After completion of the reaction as indicated by TLC, the mixture was diluted with water and extracted with ethyl acetate (3 × 15 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure, and the crude material was purified by column chromatography on silica gel using hexane/ethyl acetate (96:4) as the eluent to afford 4-methyl-N-(2-phenylallyl)-N-(1-(phenylethynyl)cyclohexyl)benzenesulfonamide (**1r**) as a colorless gummy solid (95 mg, 61% yield).

6.4. methyl 4-((1-((4-methyl-N-(2-phenylallyl)phenyl)sulfonamido)cyclohexyl)ethynyl)benzoate (1s) (GP-A1)



A dried Schlenk flask was charged with N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (150 mg, 0.38 mmol, 1.0 equiv) and methyl 4-iodobenzoate (123 mg, 0.42 mmol, 1.1 equiv) in dry THF (5 mL), and the system was purged with nitrogen. $Pd(PPh_3)_2Cl_2$ (5.3 mg, 0.0076 mmol, 2 mol%), CuI (3.6 mg, 0.019 mmol, 5 mol%), and freshly distilled triethylamine (77 mg, 0.76 mmol, 2.0 equiv) were added under a nitrogen atmosphere, and the resulting reaction mixture was stirred at room temperature for 12 h. After completion of the reaction as indicated by TLC, the mixture was diluted with water and extracted with ethyl acetate (3 × 15 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure, and the crude material was purified by column chromatography on silica gel using hexane/ethyl acetate (90:10) as the eluent to afford methyl 4-((1-((4-methyl-N-(2-phenylallyl)phenyl)sulfonamido)cyclohexyl)ethynyl)benzoate (**1s**) as a colorless gummy solid (108 mg, 55% yield).

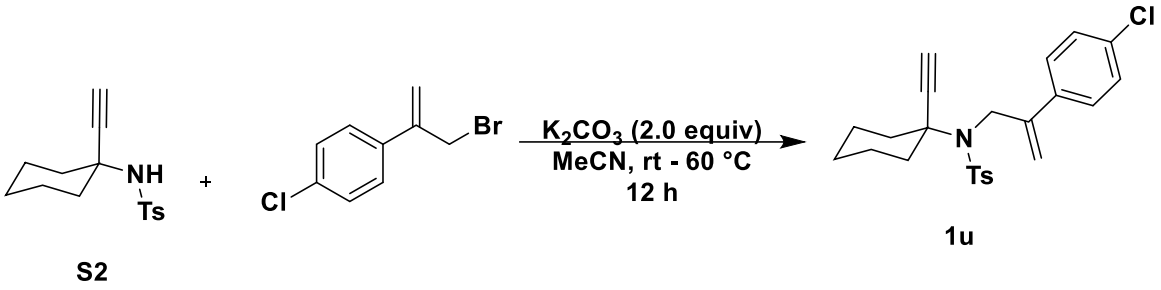
6.5. N-(1-((4-acetylphenyl)ethynyl)cyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (1t) (GP-A1)



A dried Schlenk flask was charged with N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (150 mg, 0.38 mmol, 1.0 equiv) and 1-(4-iodophenyl)ethan-1-one (101 mg, 0.42 mmol, 1.1 equiv) in dry THF (5 mL), and the system was purged with nitrogen. $Pd(PPh_3)_2Cl_2$ (5.3 mg, 0.0076 mmol, 2 mol%), CuI (3.6 mg, 0.019 mmol, 5 mol%), and freshly distilled triethylamine (77 mg, 0.76 mmol, 2.0 equiv) were added under a nitrogen atmosphere, and the resulting reaction mixture was stirred at room

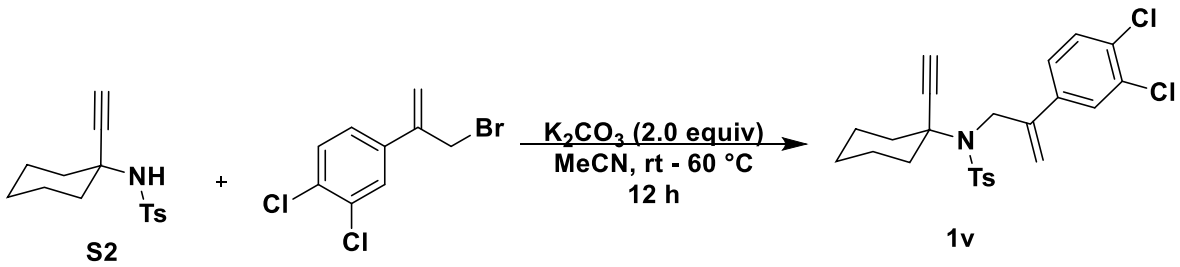
temperature for 12 h. After completion of the reaction as indicated by TLC, the mixture was diluted with water and extracted with ethyl acetate (3 × 15 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure, and the crude material was purified by column chromatography on silica gel using hexane/ethyl acetate (88:12) as the eluent to afford N-(1-((4-acetylphenyl)ethynyl)cyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (**1t**) as a colorless gummy solid (97 mg, 50% yield).

6.6. N-(2-(4-chlorophenyl)allyl)-N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (1u)



To a stirred solution of N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (**S2**) (150 mg, 0.54 mmol, 1.0 equiv) in dry acetonitrile (5 mL) were added potassium carbonate (149 mg, 1.08 mmol, 2.0 equiv) and 1-(3-bromoprop-1-en-2-yl)-4-chlorobenzene (150 mg, 0.65 mmol, 1.2 equiv). The reaction mixture was heated at 60 °C for 12 h. After completion of the reaction as indicated by TLC, the mixture was cooled to room temperature and the solvent was removed under reduced pressure. The resulting residue was dissolved in ethyl acetate, washed successively with water and brine, and dried over anhydrous Na_2SO_4. Filtration and concentration under reduced pressure afforded the crude product, which was purified by column chromatography on silica gel using an appropriate eluent to yield N-(2-(4-chlorophenyl)allyl)-N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (**1u**) as an off-white solid (130 mg, 60% yield).

6.7. N-(2-(3,4-dichlorophenyl)allyl)-N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (1v):

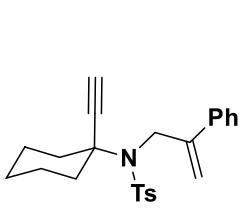


To a stirred solution of N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (**S2**) (150 mg, 0.54 mmol, 1.0 equiv) in dry acetonitrile (5 mL) were added potassium carbonate (149 mg, 1.08 mmol, 2.0 equiv) and 4-(3-bromoprop-1-en-2-yl)-1,2-dichlorobenzene (173 mg, 0.65 mmol, 1.2 equiv). The reaction mixture was heated at 60 °C for 12 h. After completion of the reaction as indicated by TLC, the mixture was cooled to room temperature

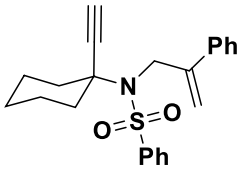
and the solvent was removed under reduced pressure. The resulting residue was dissolved in ethyl acetate, washed successively with water and brine, and dried over anhydrous Na_2SO_4. Filtration and concentration under reduced pressure afforded the crude product, which was purified by column chromatography on silica gel using an appropriate eluent to yield N-(2-(3,4-dichlorophenyl)allyl)-N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (**1v**) as an off-white solid (150 mg, 63% yield).

Note: Additional 1,1-disubstituted cyclohexanes (**1b**–**1p**) were synthesized analogously using the corresponding 1-ethynylcyclohexan-1-amines as starting materials, in combination with the appropriate sulfonamides and allylic bromides. Reactions involving the allylic bromides and sulfonamide derivatives were carried out at 60 °C for 12 h. In all cases, a trace amount of starting material remained after 12 h; however, the reactions were stopped and the products were isolated at this point, as prolonged reaction times led to the formation of additional byproducts. For the synthesis of **1q**, the starting material **1p** was used. Compounds **1r**, **1s**, and **1t** were prepared from **1a**, while **S2** served as the starting material for **1u** and **1v**.

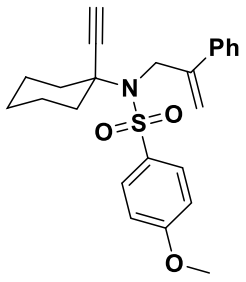
6.8. 1,1-Disubstituted cyclohexane substrates employed in our transformation



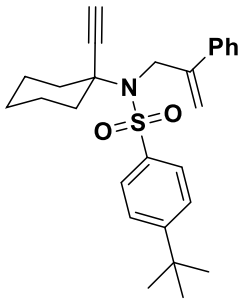
1a



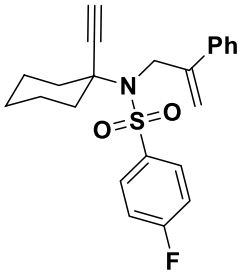
1b



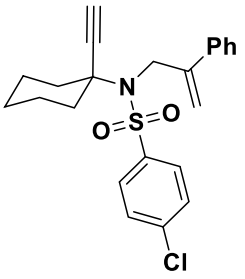
1c



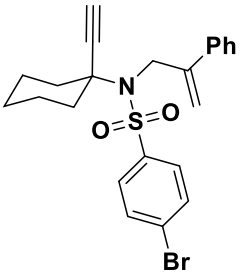
1d



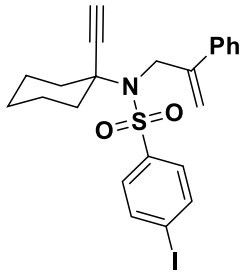
1e



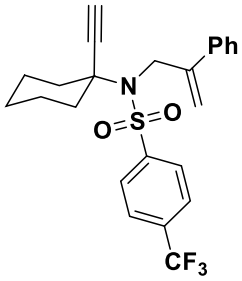
1f



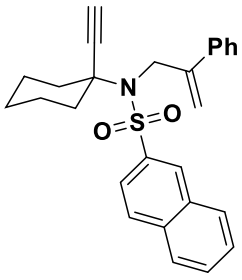
1g



1h



1i



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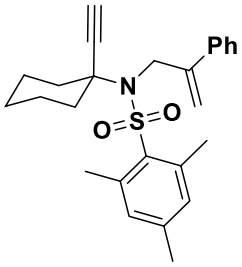
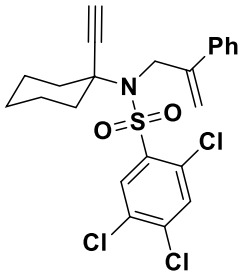
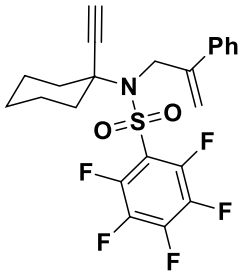


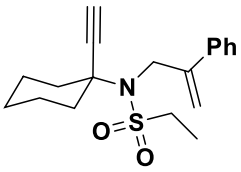
1k



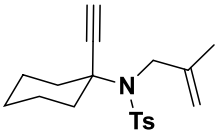
1l



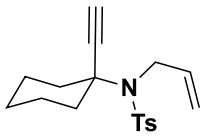
1m



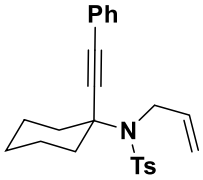
1n



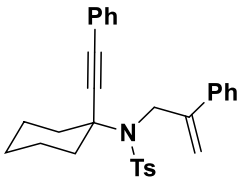
1o



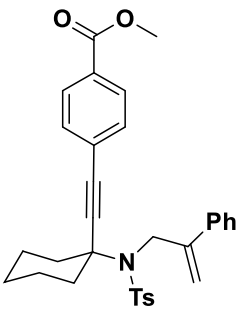
1p



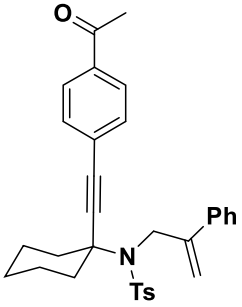
1q



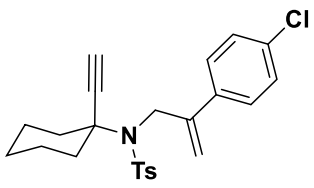
1r



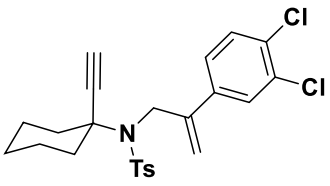
1s



1t

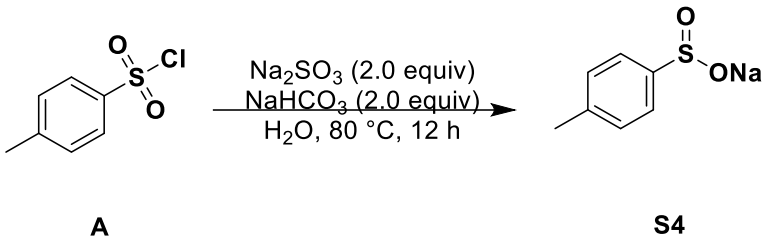


1u



1v

6.9. Synthesis of sodium *p*-toluenesulfinate (S4)

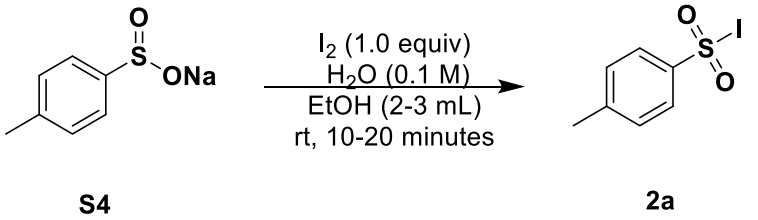


A **S4**

To a 100 mL round-bottom flask equipped with a magnetic stir bar was added sodium sulfite (2.64 g, 20.98 mmol, 2.0 equiv) and sodium bicarbonate (1.76 g, 20.98 mmol, 2.0 equiv) in distilled water (30 mL). The mixture was stirred until a clear solution formed. *p*-Toluenesulfonyl chloride (2.0 g, 10.49 mmol, 1.0 equiv) was then added portion-wise over 5 minutes at room temperature. The resulting mixture was heated to 80 °C and stirred for 4–12 h (monitored by TLC or LC-MS). After completion, the reaction was cooled to room temperature, and the aqueous layer was concentrated to dryness under reduced pressure using a rotary evaporator. The resulting crude solid was triturated with cold ethanol (10 mL) to remove impurities and then filtered. The residue was recrystallized from hot ethanol (10 mL) to afford the sodium *p*-toluenesulfinate as an off-white crystalline solid (1.2 g, 64%).

Note: Other aryl sulfinates (e.g., sodium 4-chlorobenzenesulfinate, sodium 4-methoxybenzenesulfinate, etc.) were synthesized analogously using the corresponding aryl sulfonyl chlorides.

6.9.1. Synthesis of 4-methylbenzenesulfonyl iodide (2a)[2]



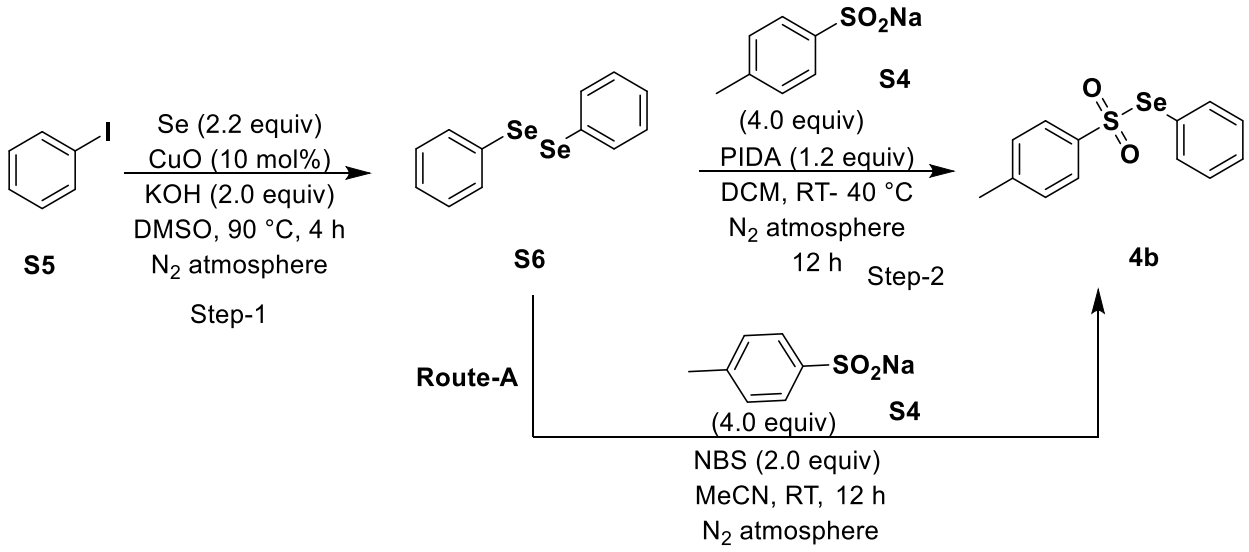
S4 **2a**

To a 50 mL round-bottom flask was added sodium *p*-toluenesulfinate (200 mg, 1.12 mmol, 1.0 equiv) dissolved in distilled water at room temperature. A saturated solution of iodine (284 mg, 1.12 mmol, 1.0 equiv) in ethanol (2–3 mL) was prepared separately and added dropwise to the sulfinate solution. During the addition, a yellow precipitate formed gradually. Upon completion, the precipitate was collected by filtration, washed with cold water, and carefully dried at room temperature to afford the *p*-toluenesulfonyl iodide as a yellow solid.

Note: The freshly prepared 4-methylbenzenesulfonyl iodide was used immediately in the next step. The product may contain trace amounts of water due to insufficient drying under high vacuum.

Note: Other sulfonyl iodides were synthesized using a similar procedure.

6.10. Synthesis of Se-phenyl 4-methylbenzenesulfonoselenoate (4b)[3]



6.10.1. Synthesis of 1,2-Diphenyldiselane (S6) – Step 1

A stirred solution of selenium powder (426 mg, 5.39 mmol, 2.2 equiv) and iodobenzene (500 mg, 2.45 mmol, 1.0 equiv) in DMSO (10 mL) was treated with CuO nanoparticles (20 mg, 0.245 mmol, 0.1 equiv). The reaction vessel (Schlenk tube) was then evacuated and backfilled with nitrogen three times. Potassium hydroxide (4.90 mmol, 2.0 equiv) was added under a nitrogen atmosphere. The resulting mixture was stirred at 90 °C for 4 h. Upon completion, the reaction mixture was cooled to room temperature and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (hexane/ethyl acetate = 99:1) to afford the 1,2-diphenyldiselane (**S6**) in 46% yield.

Caution: All selenium compounds possess a strong and unpleasant odor; proper ventilation and handling precautions are advised**.**

6.10.2. Synthesis of Se-phenyl 4-methylbenzenesulfonoselenoate (4b) (Step-2)

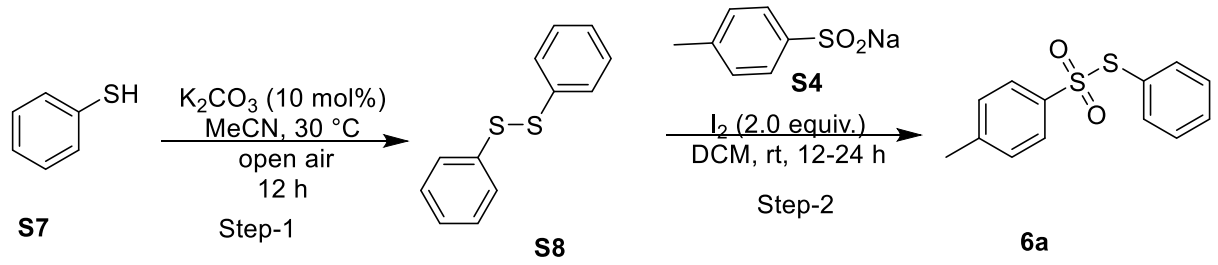
A suspension of sodium p-toluenesulfinate (799 mg, 4.48 mmol, 4.0 equiv) in CH_2Cl_2 (30 mL) containing 1,2-diphenyldiselane (350 mg, 1.12 mmol, 1.0 equiv) was prepared in a Schlenk tube. The tube was evacuated and backfilled with nitrogen three times. The reaction mixture was cooled to 0 °C, and a solution of (diacetoxyiodo)benzene (433 mg, 1.34 mmol, 1.2 equiv) in CH_2Cl_2 was added dropwise under a nitrogen atmosphere. The mixture was then stirred at room temperature to 40 °C for 12 h. After completion, the reaction mixture was washed with water, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude residue was purified by column chromatography on silica gel (hexane/ethyl acetate = 95:5) to afford the Se-phenyl 4-methylbenzenesulfonoselenoate (**4b**) as a yellow solid in 53% yield.

Alternative procedure for the synthesis of Se-phenyl 4-methylbenzenesulfonoselenoate (4b) (route-A)

To a suspension of 1,2-diphenyldiselane (300 mg, 0.96 mmol, 1.0 equiv) in MeCN (30 mL) was added N-bromosuccinimide (NBS, 342 mg, 1.92 mmol, 2.0 equiv). The reaction flask was evacuated and backfilled with nitrogen three times. Under a nitrogen atmosphere, sodium p-toluenesulfinate (685 mg, 3.84 mmol, 4.0 equiv) was added. The reaction mixture was stirred at room temperature for 12 h. Upon completion (monitored by TLC), the mixture was diluted with water and extracted with ethyl acetate. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude residue was purified by column chromatography (hexane/ethyl acetate = 95:5) to afford the Se-phenyl 4-methylbenzenesulfonoselenoate (**4b**) as a yellow solid in 48% yield.

Note: Other sulfonoselenoates were synthesized using a similar protocol by varying the sulfinate salt.

6.11. Procedure for the synthesis of S-phenyl 4-methylbenzenesulfonothioate (6a)[4]



6.11.1. Procedure for the synthesis of 1,2-diphenyldisulfane (S8) (step-1)

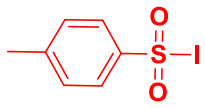
To an oven-dried 25 mL round-bottom flask equipped with a magnetic stir bar were added potassium carbonate (K_2CO_3, 28 mg, 0.20 mmol, 0.1 equiv) and benzenethiol (220 mg, 2.00 mmol, 1.0 equiv) in acetonitrile (MeCN, 10 mL). The reaction mixture was stirred at 30 °C under open air for 12 h and monitored by TLC. Upon completion, the mixture was diluted with ethyl acetate (10 mL) and concentrated under reduced pressure. The crude residue was purified by column chromatography on silica gel (EtOAc/hexane = 1:99) to afford the 1,2-diphenyldisulfane as an off-white solid in 80% yield.

6.11.2.Procedure for the synthesis of S-phenyl 4-methylbenzenesulfonothioate (6a)

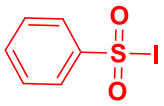
To an oven-dried 50 mL round-bottom flask were added 1,2-diphenyldisulfane (0.49 g, 2.00 mmol), 4-methylbenzenesulfinate (1.06 g, 6.00 mmol), and iodine (I_2, 1.01 g, 4.00 mmol) in dichloromethane (DCM, 10 mL). The reaction mixture was stirred at room temperature for 12 h and monitored by TLC. Upon completion, the mixture was diluted with DCM (25 mL) and sequentially washed with water (10 mL) and 5% aqueous sodium thiosulfate solution (10 mL). The organic layer was dried over anhydrous sodium sulfate (Na_2SO_4), filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel

using 5% ethyl acetate in hexane as eluent afford the S-phenyl 4-methylbenzenesulfonothioate (**6a**) as a pale white solid (0.48 g, 91% yield).

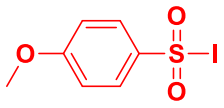
6.12. List of bifunctional reagents utilized in our transformation[2-4]



2a



2b



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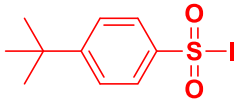

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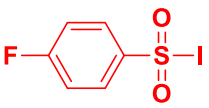

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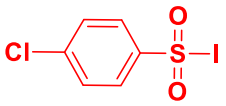

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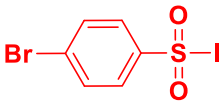

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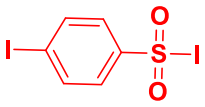

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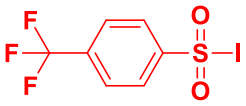

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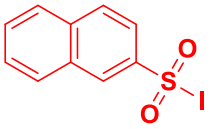

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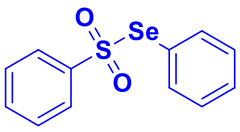
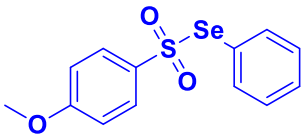


4a

4b



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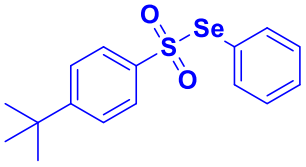

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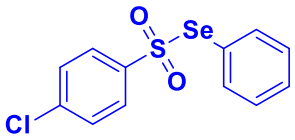

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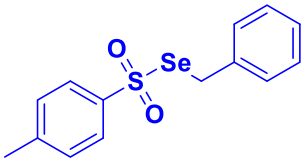
4f

4g



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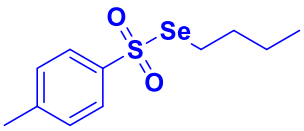

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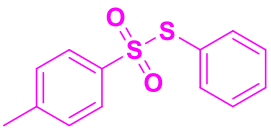

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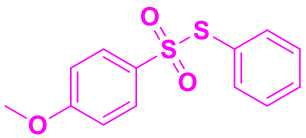

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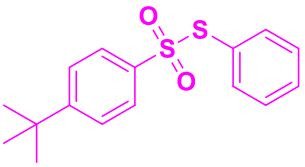

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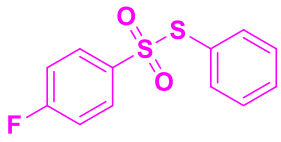

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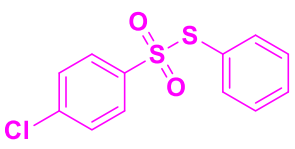

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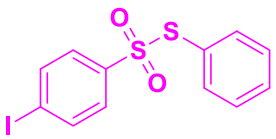

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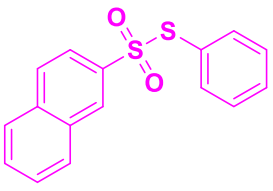

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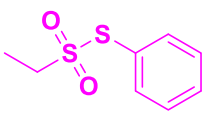

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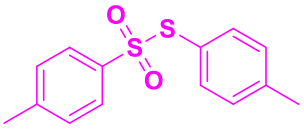

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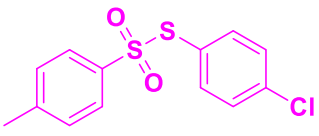

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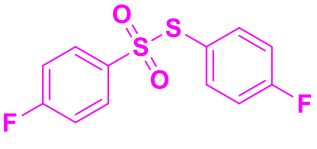
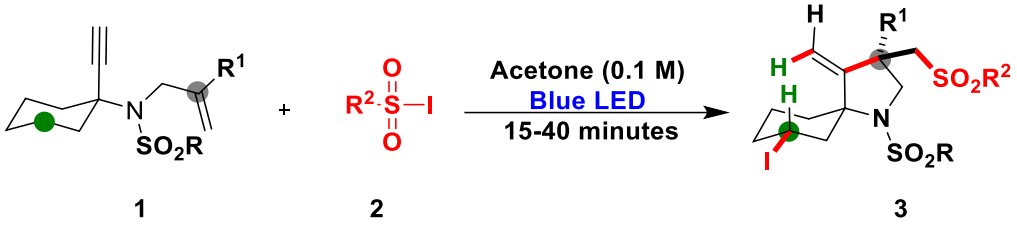


6h

7. Experimental procedures

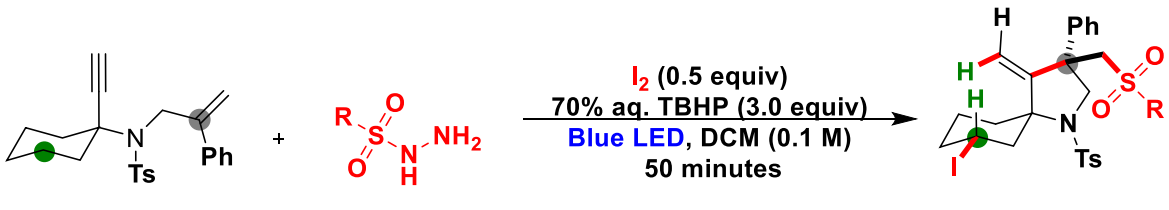
7.1. General Procedure 2 (GP-2) for the synthesis of 3aa-3ai and 3am-3ra from 1,1-disubstituted cyclohexane with sulfonyl iodides



1 2 3

An oven-dried 8 mL screw-capped vial equipped with a magnetic stir bar was charged the appropriate 1,1-disubstituted cyclohexane (0.10 mmol, 1.0 equiv) and the corresponding sulfonyl iodide (0.11 mmol, 1.1 equiv), followed by the addition of acetone (0.1 M). The reaction mixture was stirred at room temperature under blue LED irradiation (with the vial placed approximately 8.5 cm from the LED light source and a clip fan for cooling) until complete consumption of the starting material was observed by TLC (typically within 15–40 minutes). After completion, the reaction mixture was diluted with water and extracted with CH_2Cl_2. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude residue was purified by flash column chromatography (hexanes/ethyl acetate) to afford the corresponding products **3aa-3ai** and **3am-3ra** as off-white solids.

7.2. General Procedure 3 (GP-3) for the synthesis of 3aj, 3ak, and 3al from N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide with sulfonyl hydrazines

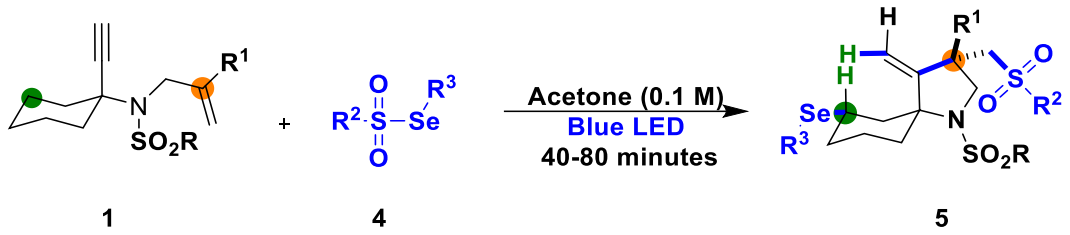


1a 2k 3aj, 3ak and 3al

An oven-dried 8 mL screw-capped vial equipped with a magnetic stir bar was charged with N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (0.10 mmol, 1.0 equiv), the appropriate sulfonyl hydrazine (0.15 mmol, 1.5 equiv), and CH_2Cl_2 (0.1 M). Iodine (I_2, 0.05 mmol, 0.5 equiv) and aqueous 70% tert-butyl hydroperoxide (TBHP, 0.30 mmol, 3.0 equiv) were then added sequentially. The reaction mixture was stirred at room temperature under blue LED irradiation (approximately 8.5 cm from the light source, with a clip fan used for cooling) for 50 minutes or until complete consumption of the starting material, as monitored by TLC. After completion, the reaction mixture was diluted with water and extracted with CH_2Cl_2. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude

residue was purified by flash column chromatography (hexanes/ethyl acetate) to afford the corresponding **3aj**, **3ak**, and **3al** as off-white solids.

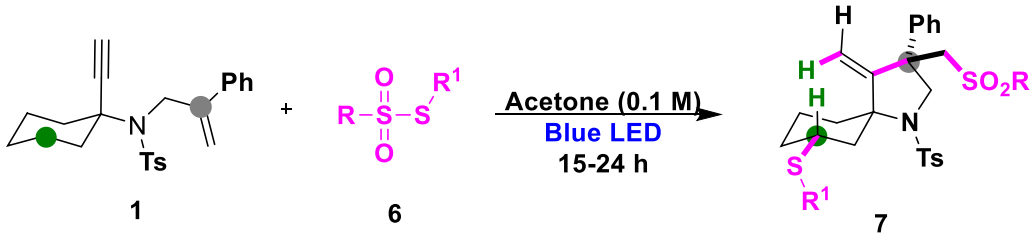
7.3. General Procedure 4 (GP-4) for the synthesis of 5aa-5ai, 5fb, 5gh, 5hb, 5jb, 5lb, 5ob and 5pb from 1,1-disubstituted cyclohexane with selanyl sulfonates



1 4 5

An oven-dried 8 mL screw-capped vial equipped with a magnetic stir bar was charged with the appropriate 1,1-disubstituted cyclohexane (0.10 mmol, 1.0 equiv), the corresponding selanyl sulfonate reagent (0.11 mmol, 1.1 equiv) bearing either aryl or aliphatic substituents, and acetone (0.1 M). The reaction mixture was stirred at room temperature under blue LED irradiation (with the vial placed approximately 8.5 cm from the LED light source and cooled by a clip fan) for 40–80 minutes, or until the starting material was fully consumed as monitored by TLC. Upon completion, the reaction mixture was diluted with water and extracted with CH_2Cl_2. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude residue was purified by flash column chromatography (hexanes/ethyl acetate) to afford the desired products **5aa-5ai, 5fb, 5gh, 5hb, 5jb, 5lb, 5ob and 5pb** as off-white solids.

7.4. General Procedure 5 (GP-5) for the synthesis of 7aa-al from N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide cyclohexane with selanyl sulfonates

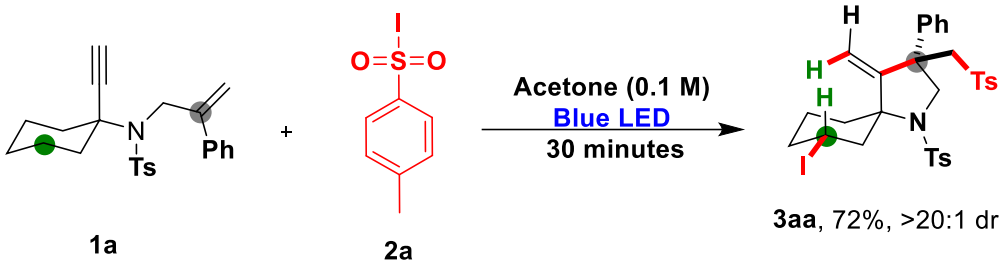


1 6 7

An oven-dried 8 mL screw-capped vial equipped with a magnetic stir bar was charged with N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (0.10 mmol, 1.0 equiv), the corresponding sulfonothioate reagent (1.1 equiv), and acetone (0.1 M). The reaction mixture was stirred at room temperature under blue LED irradiation (the vial was placed approximately 8.5 cm from the LED light source with a clip fan used for cooling) for 15–24 h, or until complete consumption of the starting material, as monitored by TLC. After completion, the reaction mixture was diluted with water and extracted with CH_2Cl_2. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude product was

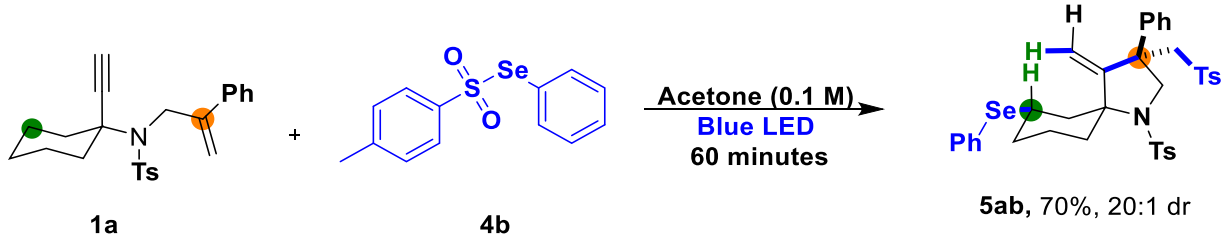
purified by flash column chromatography (hexane/ethyl acetate) to afford the desired 4-products **7aa-al** as off-white solids.

7.5. Gram-scale synthesis of (3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3aa)



In an oven-dried 50 mL screw-capped vial equipped with a magnetic stir bar, N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (1.0 g, 2.54 mmol, 1.0 equiv) and 4-methylbenzenesulfonyl iodide (0.79 g, 2.79 mmol, 1.1 equiv) were dissolved in acetone (25.4 mL, 0.1 M). The reaction mixture was stirred at room temperature under blue LED irradiation (vial placed ~8.5 cm from the LED light source with fan cooling) until full consumption of the starting material (monitored by TLC, 30 min). Upon completion, the mixture was diluted with water and extracted with CH_2Cl_2 (3 × 30 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude residue was purified by flash column chromatography (hexane/ethyl acetate = 83:17) to afford the product **3aa** (72%, 1.24 g) as off-white solid.

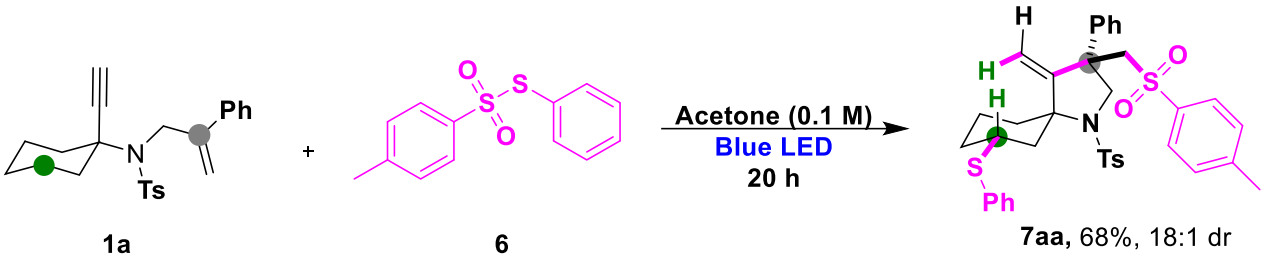
7.6. Gram-scale synthesis of (3S*,5R*,7S*)-4-methylene-3-phenyl-7-(phenylselanyl)-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (5ab)



In an oven-dried 50 mL screw-capped vial equipped with a magnetic stir bar, N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (1.0 g, 2.54 mmol, 1.0 equiv), Se-phenyl 4-methylbenzenesulfonoselenoate (0.87 g, 2.79 mmol, 1.1 equiv), and acetone (25.4 mL, 0.1 M) were combined. The mixture was stirred at room temperature under blue LED irradiation (vial placed approximately 8.5 cm from the LED light source with a clip fan for cooling) for 60 minutes or until complete consumption of starting material was confirmed by TLC. Upon completion, the reaction mixture was diluted with water and extracted with CH_2Cl_2 (3 × 30 mL). The combined organic layers were dried over anhydrous sodium sulfate (Na_2SO_4), filtered, and

concentrated under reduced pressure. The crude residue was purified by flash column chromatography (hexane/ethyl acetate = 82:18) to afford the desired product **5ab** (70%, 1.25 g) as off-white solid.

7.7. Gram-scale synthesis of (3R*,5S*,7R*)-4-methylene-3-phenyl-7-(phenylthio)-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (7aa)

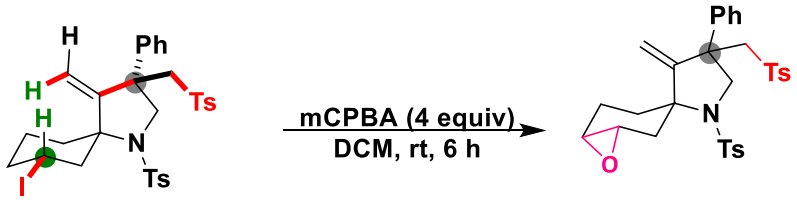


1a **6** **7aa**, 68%, 18:1 dr

In an oven-dried 50 mL screw-capped vial equipped with a magnetic stir bar, N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (1.0 g, 2.54 mmol, 1.0 equiv), S-phenyl 4-methylbenzenesulfonothioate (0.71 g, 2.79 mmol, 1.1 equiv), and acetone (25.4 mL, 0.1 M) were combined. The resulting solution was stirred at room temperature under blue LED irradiation (vial placed approximately 8.5 cm from the LED light source with a clip fan for cooling) for 20 h or until complete consumption of starting material was confirmed by TLC. After completion, the reaction mixture was diluted with water and extracted with CH_2Cl_2 (3 × 30 mL). The combined organic layers were dried over anhydrous sodium sulfate (Na_2SO_4), filtered, and concentrated under reduced pressure. The crude residue was purified by flash column chromatography (hexane/ethyl acetate = 83:17) to afford the product **7aa** (68%, 1.13 g) as off-white solid.

7.8 Synthetic transformations of (3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3aa)

7.8.1. Procedure (B) for synthesis of (1S*,3S*,6R*)-3'-methylene-4'-phenyl-1'-tosyl-4'-(tosylmethyl)-7-oxaspiro[bicyclo[4.1.0]heptane-3,2'-pyrrolidine] (8a)

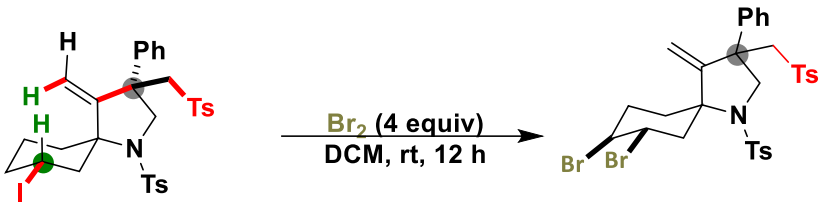


3aa, 70 mg **8a**, 52%

An oven-dried sealed tube (15 mL) was charged with (3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (**3aa**, 70 mg, 0.1 mmol, 1.0 equiv) and meta-chloroperoxybenzoic acid (mCPBA; 73 mg, 0.4 mmol, 4 equiv)in dichloromethane (3 mL) at room temperature. The reaction mixture was stirred at room temperature for 6 h. Upon completion of the reaction, as monitored by TLC, the mixture was

diluted with water and extracted with dichloromethane. The combined organic layers were then dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure to afford the crude product. Purification by column chromatography on silica gel using hexane–ethyl acetate (55:45) as the eluent furnished 3'-methylene-4'-phenyl-1'-tosyl-4'-(tosylmethyl)-7-oxaspiro[bicyclo[4.1.0]heptane-3,2'-pyrrolidine] (**8a**) as an off-white solid (30 mg, 52% yield).

7.8.2. Procedure (C) for synthesis of (5S*,7S*,8S*)-7,8-dibromo-4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (9a)

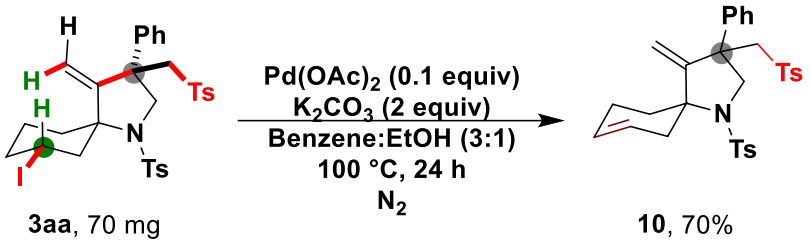


3aa, 70 mg

9a; 73%

An oven-dried sealed tube (15 mL) was charged with (3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (**3aa**, 70 mg, 0.1 mmol, 1.0 equiv) in dichloromethane (3 mL). Bromine (Br_2; 66 mg, 0.4 mmol, 4 equiv) was added dropwise at 0 °C, and the resulting mixture was then stirred at room temperature for 12 h. Upon completion of the reaction, as monitored by TLC, the mixture was diluted with water and extracted with dichloromethane. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure to afford the crude product. Purification by column chromatography on silica gel using hexane–ethyl acetate (78:22) as the eluent furnished 7,8-dibromo-4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (**9a**) as off-white solid (53 mg, 73% yield).

7.8.3. Procedure (D) for synthesis of 4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]dec-7-ene (10)



3aa, 70 mg

10, 70%

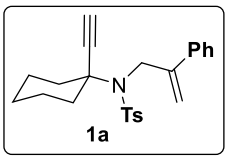
An oven-dried sealed tube (15 mL) was charged with (3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (**3aa**; 70 mg, 0.1 mmol, 1.0 equiv), $Pd(OAc)_2$ (11 mg, 0.01 mmol, 10 mol%), and K_2CO_3 (27 mg, 0.2 mmol, 2.0 equiv) in a mixture of benzene:ethanol (3:1, 3 mL) under a nitrogen atmosphere. The reaction mixture was stirred at 100 °C for 24 h. Progress of the reaction could not be reliably monitored by

TLC, as the starting material and the product exhibit the same Rf. After stirring, the mixture was cooled to room temperature, diluted with water, and extracted with ethyl acetate. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The desired 4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]dec-7-ene (**10**) was obtained as a solid (40 mg, 70% yield) after purification by column chromatography using ethyl acetate/hexane (17:83).

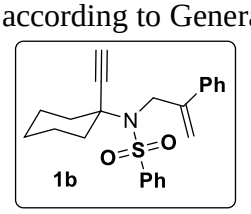
Note: Complete separation of the product from the remaining starting material was challenging due to their identical Rf values. This is also reflected in the NMR spectrum of the column-purified material.

8. Characterization data

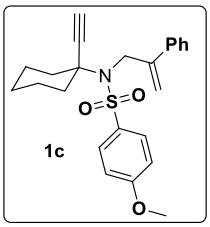
N-(1-ethynylcyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (1a): The title compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 5:95) to afford the product as an off-white solid (yield = 62%); Mp. 113-114 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.3 Hz, 2H), 7.41 (dd, *J* = 8.2, 1.4 Hz, 2H), 7.37 – 7.28 (m, 3H), 7.26 (d, *J* = 7.9 Hz, 2H), 5.59 (s, 1H), 5.50 (s, 1H), 4.52 (s, 2H), 2.40 (s, 3H), 2.22 (s, 1H), 2.13 (d, *J* = 11.4 Hz, 2H), 1.93 (td, *J* = 12.7, 3.5 Hz, 2H), 1.73 – 1.56 (m, 4H), 1.54 – 1.47 (m, 1H), 1.17 – 1.04 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.5, 143.0, 139.7, 138.9, 129.2, 128.3, 127.7, 126.1, 114.0, 83.0, 74.8, 62.5, 51.5, 37.8, 24.7, 23.5, 21.5. **HRMS** (ESI) calcd for $C_{24}H_{27}O_2NNaS$ $[M+Na]^+$ 416.1654; found: 416.1652.



N-(1-ethynylcyclohexyl)-N-(2-phenylallyl)benzenesulfonamide (1b): The title compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 5:95) to afford the product as an off-white solid (yield = 60%); Mp. 73-74 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 (ddd, *J* = 7.0, 3.2, 1.8 Hz, 2H), 7.57 – 7.53 (m, 1H), 7.50 – 7.46 (m, 2H), 7.43 – 7.39 (m, 2H), 7.38 – 7.30 (m, 3H), 5.59 (d, *J* = 0.9 Hz, 1H), 5.51 (d, *J* = 0.9 Hz, 1H), 4.54 (s, 2H), 2.19 (s, 1H), 2.17 – 2.11 (m, 2H), 1.93 (td, *J* = 12.7, 3.5 Hz, 2H), 1.71 – 1.65 (m, 3H), 1.61 – 1.57 (m, 1H), 1.53 – 1.47 (m, 1H), 1.17 – 1.06 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.4, 141.7, 139.7, 132.4, 128.7, 128.4, 127.8, 127.8, 126.1, 114.1, 82.9, 74.8, 62.6, 51.6, 37.8, 24.7, 23.5. **HRMS** (ESI) calcd for $C_{23}H_{25}O_2NNaS$ $[M+Na]^+$ 402.1498; found: 402.1498.

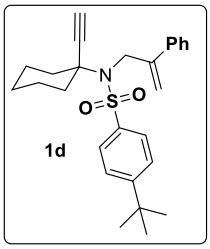


N-(1-ethynylcyclohexyl)-4-methoxy-N-(2-phenylallyl)benzenesulfonamide (1c): The title compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 8:92) to afford the product as a colorlessgummy solid (yield = 55%); **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.81 (d, *J* = 9.0 Hz, 2H), 7.42 – 7.39 (m, 2H), 7.37 – 7.27 (m, 3H), 6.94 (d, *J* = 9.0 Hz, 2H), 5.58 (d, *J* = 0.9 Hz, 1H), 5.50 (d, *J* = 1.0 Hz, 1H), 4.50 (s, 2H), 3.86 (s, 3H), 2.22 (s, 1H), 2.14 (d, *J* = 11.5 Hz, 2H), 1.92 (td, *J* = 12.7, 3.4 Hz, 2H), 1.73 – 1.56 (m, 4H), 1.52 (t, *J* = 8.3 Hz, 1H), 1.19 – 1.03 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.6, 145.5, 139.8, 133.5, 129.9, 128.4,



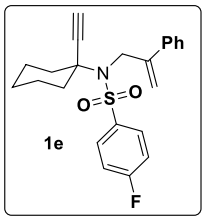
127.7, 126.1, 114.0, 113.7, 83.2, 74.7, 62.5, 55.5, 51.5, 37.8, 24.7, 23.5. **HRMS** (ESI) calcd for $C_{24}H_{27}O_3NNaS$ $[M+Na]^+$ 432.1603; found: 432.1601.

4-(*tert*-butyl)-N-(1-ethynylcyclohexyl)-N-(2-phenylallyl)benzenesulfonamide (1d): The title compound was



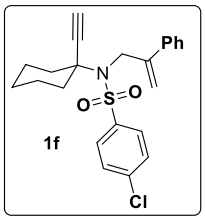
prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 6:94) to afford the product as an off-white solid (yield = 56%); Mp. 144-145 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.7 Hz, 2H), 7.48 (d, *J* = 8.7 Hz, 2H), 7.42 – 7.39 (m, 2H), 7.37 – 7.28 (m, 3H), 5.60 (d, *J* = 0.9 Hz, 1H), 5.51 (d, *J* = 0.9 Hz, 1H), 4.51 (d, *J* = 1.6 Hz, 2H), 2.20 – 2.11 (m, 3H), 1.94 (td, *J* = 12.8, 3.6 Hz, 2H), 1.68 (d, *J* = 13.7 Hz, 2H), 1.60 (s, 1H), 1.52 (dd, *J* = 13.6, 3.5 Hz, 2H), 1.34 (s, 9H), 1.18 – 1.07 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 156.1, 145.5, 139.8, 138.6, 128.4, 127.8, 127.6, 126.1, 125.6, 114.1, 83.1, 74.6, 62.6, 51.5, 37.8, 35.1, 31.1, 24.7, 23.5. **HRMS** (ESI) calcd for $C_{27}H_{33}O_2NNaS$ $[M+Na]^+$ 458.2124; found: 458.2122.

N-(1-ethynylcyclohexyl)-4-fluoro-N-(2-phenylallyl)benzenesulfonamide (1e): The title compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl



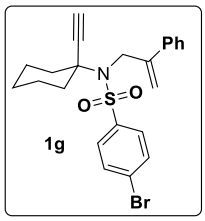
acetate/hexane = 5:95) to afford the product as an off-white solid (yield = 61%); Mp. 91-92 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 9.0, 5.1 Hz, 2H), 7.44 – 7.28 (m, 5H), 7.20 – 7.11 (m, 2H), 5.53 (dd, *J* = 17.9, 0.9 Hz, 2H), 4.51 (s, 2H), 2.23 (s, 1H), 2.13 (d, *J* = 11.1 Hz, 2H), 1.92 (td, *J* = 12.8, 3.5 Hz, 2H), 1.68 (d, *J* = 14.5 Hz, 2H), 1.59 (s, 1H), 1.57 – 1.48 (m, 2H), 1.18 – 1.05 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.2 (d, *J* = 254.5 Hz), 163.6 (d, *J* = 24.1 Hz), 145.3 (d, *J* = 9.2 Hz), 139.6, 137.7, 130.5, 130.4, 128.4, 127.9, 126.1, 115.9, 115.7, 114.1 (d, *J* = 3.2 Hz), 82.9, 74.96, 62.65, 51.52, 37.86, 24.64, 23.46. ^{19}F NMR (377 MHz, $CDCl_3$) δ -105.7. **HRMS** (ESI) calcd for $C_{23}H_{24}O_2NFNaS$ $[M+Na]^+$ 420.1404; found: 420.1402.

4-chloro-N-(1-ethynylcyclohexyl)-N-(2-phenylallyl)benzenesulfonamide (1f): The title compound was

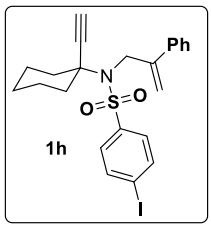


prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 5:95) to afford the product as an off-white solid (yield = 65%); Mp. 102-103 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.8 Hz, 2H), 7.45 (d, *J* = 8.8 Hz, 2H), 7.42 – 7.28 (m, 5H), 5.53 (dd, *J* = 15.4, 0.8 Hz, 2H), 4.52 (s, 2H), 2.25 (s, 1H), 2.12 (d, *J* = 11.2 Hz, 2H), 1.99 – 1.87 (m, 2H), 1.72 – 1.64 (m, 2H), 1.60 (d, *J* = 3.5 Hz, 1H), 1.60 – 1.59 (m, 1H), 1.54 – 1.46 (m, 1H), 1.19 – 1.04 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.3, 140.2, 139.5, 138.8, 129.2, 128.9, 128.4, 127.9, 126.1, 114.2, 82.8, 75.1, 62.7, 51.6, 37.9, 24.6, 23.5. **HRMS** (ESI) calcd for $C_{23}H_{24}O_2NClNaS$ $[M+Na]^+$ 436.1108; found: 436.1108.

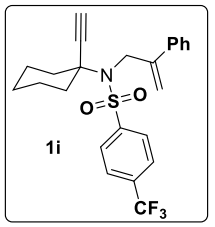
4-bromo-N-(1-ethynylcyclohexyl)-N-(2-phenylallyl)benzenesulfonamide (1g): The title compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 4:96) to afford the product as an off-white solid (yield = 66%); Mp. 110-111 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.74 (d, J = 8.8 Hz, 2H), 7.61 (d, *J* = 8.8 Hz, 2H), 7.42 – 7.28 (m, 5H), 5.55 (d, *J* = 0.8 Hz, 1H), 5.51 (d, *J* = 0.9 Hz, 1H), 4.52 (s, 2H), 2.26 (s, 1H), 2.15 – 2.09 (m, 2H), 1.92 (td, *J* = 12.7, 3.5 Hz, 2H), 1.72 – 1.64 (m, 2H), 1.63 – 1.56 (m, 2H), 1.54 – 1.48 (m, 1H), 1.18 – 1.05 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.3, 140.8, 139.6, 131.9, 129.3, 128.4, 127.9, 127.3, 126.1, 114.2, 82.9, 75.2, 62.8, 51.6, 37.9, 24.6, 23.5. **HRMS** (ESI) calcd for $C_{23}H_{24}O_2NBrNaS$ $[M+Na]^+$ 480.0603; found: 480.0602.



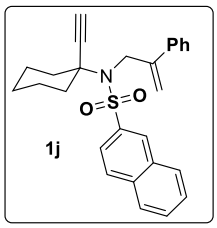
N-(1-ethynylcyclohexyl)-4-iodo-N-(2-phenylallyl)benzenesulfonamide (1h): The title compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 4:96) to afford the product as an off-white solid (yield = 62%); Mp. 98-99 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.7 Hz, 2H), 7.58 (d, *J* = 8.7 Hz, 2H), 7.42 – 7.27 (m, 5H), 5.55 (d, *J* = 0.7 Hz, 1H), 5.51 (d, *J* = 0.8 Hz, 1H), 4.51 (s, 2H), 2.26 (s, 1H), 2.11 (d, *J* = 11.3 Hz, 2H), 1.91 (td, *J* = 12.7, 3.4 Hz, 2H), 1.73 – 1.56 (m, 4H), 1.54 – 1.46 (m, 1H), 1.17 – 1.04 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.3, 141.4, 139.5, 137.8, 129.1, 128.4, 127.8, 126.0, 114.1, 99.6, 82.8, 75.2, 62.7, 51.5, 37.8, 24.6, 23.4. **HRMS** (ESI) calcd for $C_{23}H_{24}O_2NINaS$ $[M+Na]^+$ 528.0464; found: 528.0464.



N-(1-ethynylcyclohexyl)-N-(2-phenylallyl)-4-(trifluoromethyl)benzenesulfonamide (1i): The title compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 8:92) to afford the product as an off-white solid (yield = 55%); Mp. 130-131 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.2 Hz, 2H), 7.74 (d, *J* = 8.3 Hz, 2H), 7.43 – 7.29 (m, 5H), 5.54 (d, *J* = 10.6 Hz, 2H), 4.56 (s, 2H), 2.24 (s, 1H), 2.13 (d, *J* = 10.8 Hz, 2H), 1.94 (td, *J* = 12.7, 3.4 Hz, 2H), 1.68 (d, *J* = 14.5 Hz, 2H), 1.62 – 1.48 (m, 3H), 1.18 – 1.05 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.1, 145.0, 139.3, 134.2 (q, *J* = 32.6 Hz), 133.8, 128.4, 128.2, 127.9, 126.0, 125.8 (q, *J* = 3.7 Hz), 125.8 (q, *J* = 3.7 Hz), 125.7 (q, *J* = 3.7 Hz), 125.7 (q, *J* = 3.7 Hz), 124.6 (q, *J* = 272.4 Hz, CF_3), 121.9, 114.2, 82.6, 75.2, 62.8, 51.5, 37.8, 30.9, 24.5, 23.4. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -63.0. **HRMS** (ESI) calcd for $C_{24}H_{24}O_2NF_3NaS$ $[M+Na]^+$ 470.1372; found: 470.1368.

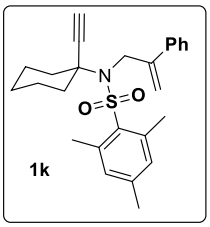


N-(1-ethynylcyclohexyl)-N-(2-phenylallyl)naphthalene-2-sulfonamide (1j): The title compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 10:90) to afford the product as an off-white solid (yield = 59%); Mp. 90-91 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.45 (d, *J* = 1.6 Hz, 1H), 7.98 – 7.86 (m, 4H), 7.66 – 7.55 (m, 2H), 7.46 – 7.41 (m, 2H), 7.40 – 7.29 (m, 3H), 5.64 (d, *J* = 0.8 Hz, 1H), 5.53 (d, *J* = 0.9 Hz, 1H), 4.64 (s, 2H), 2.20 – 2.16 (m, 3H), 1.98 (td, *J* = 12.7, 3.5 Hz, 2H), 1.70 – 1.63 (m, 2H),

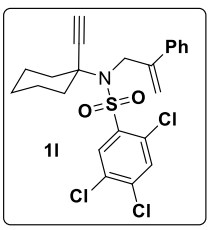


1.56 (dd, *J* = 11.0, 2.4 Hz, 2H), 1.53 – 1.46 (m, 1H), 1.18 – 1.05 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.5, 139.7, 138.8, 134.7, 132.0, 129.3, 128.9, 128.7, 128.6, 128.4, 127.8, 127.8, 127.2, 126.1, 123.3, 114.1, 83.1, 75.0, 62.7, 51.7, 37.8, 24.7, 23.5. **HRMS** (ESI) calcd for $C_{27}H_{27}O_2NNaS$ $[M+Na]^+$ 452.1654; found: 452.1653.

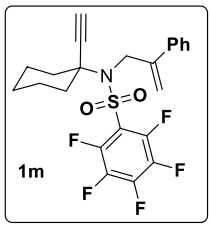
N-(1-ethynylcyclohexyl)-2,4,6-trimethyl-N-(2-phenylallyl)benzenesulfonamide (1k): The title compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 10:90) to afford the product as an off-white solid (yield = 40%); Mp. 195.6-197.2 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35 – 7.26 (m, 5H), 6.91 (s, 2H), 5.62 (s, 1H), 5.37 (s, 1H), 4.57 (s, 2H), 2.67 (s, 6H), 2.47 (s, 1H), 2.29 (s, 3H), 2.09 (td, *J* = 12.3, 4.2 Hz, 2H), 2.02 (d, *J* = 11.7 Hz, 2H), 1.62 (dd, *J* = 9.7, 6.6 Hz, 3H), 1.53 (dd, *J* = 12.6, 3.4 Hz, 2H), 1.17 – 1.05 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.7, 142.2, 140.1, 139.4, 137.3, 132.2, 128.3, 127.6, 126.1, 113.9, 83.5, 75.5, 63.4, 51.4, 36.8, 24.7, 23.8, 23.3, 20.9. **HRMS** (ESI) calcd for $C_{26}H_{31}O_2NNaS$ $[M+Na]^+$ 444.1967; found: 504.1964.



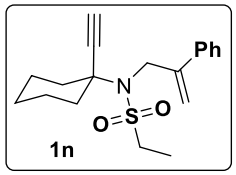
2,4,5-trichloro-N-(1-ethynylcyclohexyl)-N-(2-phenylallyl)benzenesulfonamide (1l): The title compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 12:88) to afford the product as an off-white solid (yield = 43%); Mp. 141-142 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.26 (s, 1H), 7.60 (s, 1H), 7.45 – 7.28 (m, 5H), 5.62 (s, 1H), 5.51 (s, 1H), 4.76 (s, 2H), 2.42 (s, 1H), 2.02 – 1.91 (m, 4H), 1.71 – 1.57 (m, 4H), 1.55 – 1.47 (m, 1H), 1.16 – 0.99 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.7, 140.3, 139.6, 137.3, 133.2, 132.8, 131.5, 131.1, 128.4, 127.9, 126.2, 114.1, 82.4, 76.0, 62.6, 52.4, 37.7, 24.6, 23.6. **HRMS** (ESI) calcd for $C_{23}H_{22}O_2NCl_3NaS$ $[M+Na]^+$ 504.0329; found: 504.0328.



N-(1-ethynylcyclohexyl)-2,3,4,5,6-pentafluoro-N-(2-phenylallyl)benzenesulfonamide (1m): The title compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 10:90) to afford the product as an off-white solid (yield = 45%); Mp. 146-149 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.45 – 7.30 (m, 5H), 5.55 (d, *J* = 2.0 Hz, 2H), 4.64 (s, 2H), 2.35 (s, 1H), 2.06 (d, *J* = 11.3 Hz, 2H), 1.98 (td, *J* = 12.6, 3.4 Hz, 2H), 1.71 (d, *J* = 14.9 Hz, 2H), 1.58 (s, 2H), 1.54 (d, *J* = 3.0 Hz, 1H), 1.16 – 1.08 (m, 1H), 0.84 (d, *J* = 2.5 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.2, 139.2, 128.5, 128.0, 126.0, 114.0, 81.5, 75.1, 63.0, 51.6, 37.8, 24.5, 23.5. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -133.8, -146.2, -159.1. **HRMS** (ESI) calcd for $C_{23}H_{20}O_2NF5NaS$ $[M+Na]^+$ 492.1027; found: 492.1023.

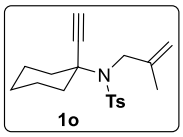


N-(1-ethynylcyclohexyl)-N-(2-phenylallyl)ethanesulfonamide (1n): The title compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 1:4) to afford the product as a colorless gummy solid (yield = 33%); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.44 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.38 – 7.32 (m, 3H), 5.49 (s, 1H), 5.44 (s, 1H), 4.90 (s, 2H), 4.08 (s, 1H), 3.46 (d, J = 1.2 Hz, 1H), 3.17 (q, J = 7.4 Hz, 2H), 2.55 (s, 1H), 2.15 – 2.11 (m, 2H), 1.66 (d, J = 3.1 Hz, 4H), 1.58 (s, 1H), 1.51 (d, J = 3.4 Hz, 1H), 1.28 (t, J = 7.4 Hz,



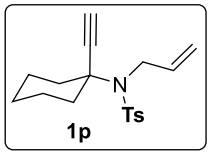
3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.5, 142.4, 128.3, 125.7, 122.1, 113.7, 73.8, 58.3, 48.7, 39.0, 24.8, 22.4, 22.3, 8.4. **HRMS** (ESI) calcd for $C_{19}H_{25}O_2NNaS$ $[M+Na]^+$ 354.1498; found: 354.1494.

N-(1-ethynylcyclohexyl)-4-methyl-N-(2-methylallyl)benzenesulfonamide (1o)[1a,1d]**:** The title compound was



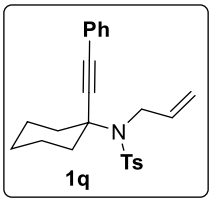
prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 4:96) to afford the product as an off-white solid (yield = 49%); Mp. 182-184 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.4 Hz, 2H), 7.26 (d, *J* = 8.7 Hz, 2H), 5.13 (d, *J* = 0.8 Hz, 1H), 4.94 (d, *J* = 1.4 Hz, 1H), 4.12 (s, 2H), 2.41 (s, 3H), 2.24 (s, 1H), 2.07 (d, *J* = 11.3 Hz, 2H), 1.88 (td, *J* = 12.6, 3.5 Hz, 2H), 1.80 (s, 3H), 1.65 (dd, *J* = 10.0, 3.7 Hz, 2H), 1.52 (tdd, *J* = 13.7, 10.0, 3.4 Hz, 3H), 1.16 – 1.04 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.2, 142.9, 139.3, 129.2, 127.5, 111.8, 83.1, 74.9, 62.3, 53.5, 37.6, 24.7, 23.4, 21.5, 20.3.

N-allyl-N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (1p)[1a]**:** The title compound was prepared



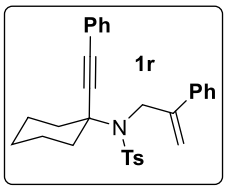
according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 4:96) to afford the product as an off-white solid (yield = 57%); Mp. 182-184 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.3 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 6.04 (ddt, *J* = 17.2, 10.3, 5.8 Hz, 1H), 5.27 (dd, *J* = 17.2, 1.5 Hz, 1H), 5.16 (dd, *J* = 10.3, 1.4 Hz, 1H), 4.18 (d, *J* = 5.8 Hz, 2H), 2.41 (s, 3H), 2.36 (s, 1H), 2.08 (d, *J* = 12.8 Hz, 2H), 1.91 (td, *J* = 12.5, 3.9 Hz, 2H), 1.70 – 1.54 (m, 5H), 1.17 – 1.04 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.8, 139.7, 137.1, 129.2, 127.3, 116.7, 83.5, 75.0, 62.1, 50.0, 37.5, 24.7, 23.4, 21.4.

N-allyl-4-methyl-N-(1-(phenylethynyl)cyclohexyl)benzenesulfonamide (1q): The title compound was



prepared according to General Procedure A and A1 and purified by column chromatography on silica gel (ethyl acetate/hexane = 4:96) to afford the product as a colorless gummy solid (114 mg, yield = 59%); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.3 Hz, 2H), 7.27 (dd, *J* = 7.2, 0.7 Hz, 3H), 7.23 – 7.16 (m, 4H), 6.12 – 6.03 (m, 1H), .31 (d, *J* = 18.7 Hz, 1H), 5.17 (d, *J* = 11.7 Hz, 1H), 4.22 (d, *J* = 5.7 Hz, 2H), 2.33 (s, 3H), 2.17 (d, *J* = 11.4 Hz, 2H), 2.02 (td, *J* = 12.4, 3.9 Hz, 2H), 1.75 – 1.67 (m, 2H), 1.66 – 1.61 (m, 2H), 1.58 (d, *J* = 3.0 Hz, 1H), 1.23 – 1.11 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.7, 139.4, 137.3, 131.4, 129.3, 128.2, 128.1, 127.4, 122.6, 116.5, 89.0, 87.0, 62.8, 50.4, 37.9, 24.8, 23.7, 21.4. HRMS (ESI) calcd for C24H27O2NNaS [M+Na]+ 416.1654; found: 416.1651.

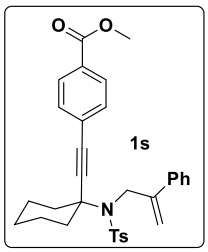
4-methyl-N-(2-phenylallyl)-N-(1-(phenylethynyl)cyclohexyl)benzenesulfonamide (1r): The title compound was prepared according to General Procedure A and A1 and purified by column chromatography on silica gel



(ethyl acetate/hexane = 4:96) to afford the product as a colorless gummy solid (95 mg, yield = 61%); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.3 Hz, 2H), 7.42 (dd, *J* = 8.2, 1.5 Hz, 2H), 7.38 – 7.22 (m, 6H), 7.20 (d, *J* = 7.9 Hz, 2H), 7.08 (dd, *J* = 8.1, 1.6 Hz, 2H), 5.67 (s, 1H), 5.55 (s, 1H), 4.55 (d, *J* = 1.6 Hz, 2H), 2.32 (s, 3H), 2.22 (d, *J* = 11.8 Hz, 2H), 2.03 (td, *J* = 12.8, 3.6 Hz, 2H), 1.73 – (m, 2H), 1.64 (d, *J* = 3.2 Hz, 2H), 1.20 – 1.10 (m, 1H), 0.91 – 0.81 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.6, 143.0, 139.8, 138.7, 131.5, 129.3, 128.4, 128.2, 128.0, 127.8, 126.1, 122.5, 114.2,

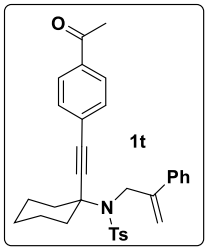
88.5, 86.8, 63.2, 51.8, 38.2, 29.7, 24.8, 23.8, 21.4. HRMS (ESI) calcd for C30H31O2NNaS [M+Na]+ 492.1967; found: 492.1968.

methyl 4-((1-((4-methyl-N-(2-phenylallyl)phenyl)sulfonamido)cyclohexyl)ethynyl)benzoate (**1s**): The title



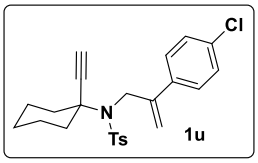
compound was prepared according to General Procedure A and A1 and purified by column chromatography on silica gel (ethyl acetate/hexane = 10:90) to afford the product as a colorless gummy solid (108 mg, yield = 55%); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.6 Hz, 2H), 7.78 (d, *J* = 8.4 Hz, 2H), 7.41 (dd, *J* = 8.2, 1.6 Hz, 2H), 7.38 – 7.28 (m, 3H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 8.6 Hz, 2H), 5.65 (s, 1H), 5.55 (s, 1H), 4.54 (s, 2H), 3.92 (s, 3H), 2.32 (s, 3H), 2.24 (d, *J* = 11.7 Hz, 2H), 2.04 (td, *J* = 12.8, 3.3 Hz, 2H), 1.73 (d, *J* = 14.4 Hz, 2H), 1.56 (d, *J* = 3.0 Hz, 2H), 1.18 (dd, *J* = 13.2, 1.5 Hz, 1H), 0.94 – 0.85 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.4, 145.4, 143.1, 139.7, 138.6, 131.4, 129.4, 129.2, 128.4, 127.8, 126.1, 114.3, 91.8, 86.0, 63.1, 52.3, 51.7, 38.1, 24.7, 23.8, 21.4.
HRMS (ESI) calcd for C32H33O4NNaS [M+Na]+ 550.2022; found: 550.2018.

N-(1-((4-acetylphenyl)ethynyl)cyclohexyl)-4-methyl-N-(2-phenylallyl)benzenesulfonamide (**1t**): The title



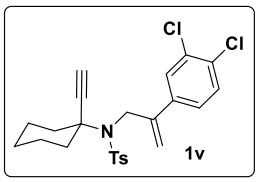
compound was prepared according to General Procedure A and A1 and purified by column chromatography on silica gel (ethyl acetate/hexane = 12:88) to afford the product as an as a colorless gummy solid (97 mg, yield = 50%); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.6 Hz, 2H), 7.78 (d, *J* = 8.3 Hz, 2H), 7.41 (dd, *J* = 8.2, 1.6 Hz, 2H), 7.38 – 7.28 (m, 3H), 7.21 (d, *J* = 7.9 Hz, 2H), 7.16 (d, *J* = 8.6 Hz, 2H), 5.65 (s, 1H), 5.54 (s, 1H), 4.54 (s, 2H), 2.59 (s, 3H), 2.32 (s, 3H), 2.25 (d, *J* = 11.4 Hz, 2H), 2.04 (td, *J* = 12.7, 3.3 Hz, 2H), 1.73 (d, *J* = 14.3 Hz, 2H), 1.65 (d, *J* = 19.9 Hz, 2H), 1.58 (d, *J* = 15.5 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.2, 145.4, 143.1, 139.7, 138.7, 136.2, 131.6, 129.4, 128.4, 127.9, 127.8, 126.1, 114.2, 92.2, 86.0, 63.1, 51.7, 38.0, 26.6, 24.7, 23.8, 21.4. HRMS (ESI) calcd for C32H33O3NNaS [M+Na]+ 534.2073; found: 534.2074.

N-(2-(4-chlorophenyl)allyl)-N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (1u): The title compound



was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 5:95) to afford the product as an off-white solid (yield = 57%); Mp. 106-109 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.4 Hz, 2H), 7.37 – 7.29 (m, 4H), 7.27 (d, *J* = 8.0 Hz, 2H), 5.61 (s, 1H), 5.50 (s, 1H), 4.49 (s, 2H), 2.42 (s, 3H), 2.22 (s, 1H), 2.10 (d, *J* = 11.3 Hz, 2H), 1.92 (td, *J* = 12.7, 3.5 Hz, 2H), 1.71 – 1.62 (m, 2H), 1.61 – 1.46 (m, 3H), 1.18 – 1.03 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.5, 143.2, 138.8, 138.1, 133.5, 129.3, 128.5, 127.7, 127.4, 114.6, 83.0, 74.9, 62.6, 51.4, 37.7, 24.6, 23.5, 21.5. HRMS (ESI) calcd for C24H26O2NClNaS [M+Na]+ 450.1373; found: 450.1376.

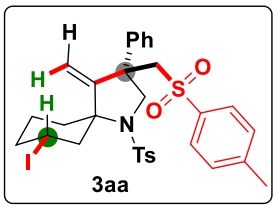
N-(2-(3,4-dichlorophenyl)allyl)-N-(1-ethynylcyclohexyl)-4-methylbenzenesulfonamide (1v): The title



compound was prepared according to General Procedure A and purified by column chromatography on silica gel (ethyl acetate/hexane = 5:95) to afford the product as an off-white solid (yield = 57%); Mp. 120-124 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 2.1 Hz, 1H), 7.41 (d, *J* = 8.4 Hz, 1H), 7.28 (s, 1H), 7.25 (dd, *J* =

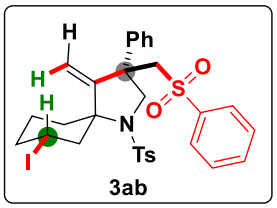
8.7, 2.4 Hz, 2H), 5.66 (s, 1H), 5.53 (s, 1H), 4.46 (s, 2H), 2.42 (s, 3H), 2.24 (s, 1H), 2.08 (d, *J* = 11.2 Hz, 2H), 1.92 (td, *J* = 12.6, 3.3 Hz, 2H), 1.67 (d, *J* = 14.2 Hz, 2H), 1.61 – 1.47 (m, 3H), 1.17 – 1.06 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.6, 143.3, 139.7, 138.7, 132.4, 131.6, 130.3, 129.3, 128.0, 127.7, 125.5, 115.7, 82.9, 75.07, 62.69, 51.2, 37.7, 24.6, 23.5, 21.5. HRMS (ESI) calcd for C24H25O2NCl2NaS [M+Na]+ 484.0983; found: 484.0980.

(3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3aa): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (53 mg, yield = 77%; >20:1 dr); Mp. 194-195 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.3 Hz, 2H), 7.09 – 6.98 (m, 7H), 5.35 (d, *J* = 1.8 Hz, 1H), 5.09 (d, *J* = 1.9 Hz, 1H), 4.71 (d, *J* = 11.0 Hz, 1H), 4.53 – 4.45 (m, 1H), 3.93 (d, *J* = 14.6 Hz, 1H), 3.41 (d, *J* = 11.0 Hz, 1H), 3.35 (d, *J* = 14.6 Hz, 1H), 3.19 (t, *J* = 12.9 Hz, 1H), 2.49 (s, 3H), 2.43 – 2.29 (m, 6H), 2.02 – 1.91 (m, 1H), 1.35 – 1.26 (m, 2H), 0.89 (d, *J* = 12.9 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.9, 144.2, 143.5, 137.3, 137.1, 136.9, 129.6, 129.5, 128.3, 127.7, 127.6, 127.1, 126.5, 108.5, 70.7, 65.1, 53.2, 49.5, 47.4, 38.0, 34.4, 23.9, 23.8, 21.6, 21.5. **HRMS** (ESI) calcd for $C_{31}H_{34}O_4NINaS_2$ $[M+Na]^+$ 698.0866; found: 698.0860.



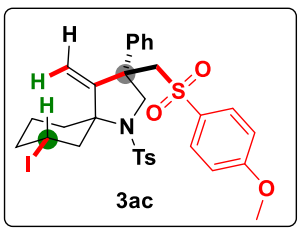
3aa

(3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-3-((phenylsulfonyl)methyl)-1-tosyl-1-azaspiro[4.5]decane (3ab): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 16:84) to afford the product as an off-white solid (51 mg, yield = 76%; 20:1 dr); Mp. 132-135 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.49 – 7.43 (m, 3H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.32 – 7.26 (m, 2H), 7.08 – 6.97 (m, 5H), 5.36 (d, *J* = 2.0 Hz, 1H), 5.10 (d, *J* = 2.1 Hz, 1H), 4.75 (d, *J* = 11.0 Hz, 1H), 4.49 (tt, *J* = 12.7, 4.5 Hz, 1H), 3.97 (d, *J* = 14.7 Hz, 1H), 3.41 (d, *J* = 11.0 Hz, 1H), 3.37 (d, *J* = 14.6 Hz, 1H), 3.19 (t, *J* = 12.9 Hz, 1H), 2.49 (s, 3H), 2.44 – 2.29 (m, 3H), 2.03 – 1.91 (m, 1H), 1.33 – 1.26 (m, 2H), 0.88 (d, *J* = 14.1 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.9, 143.5, 140.1, 137.1, 136.9, 133.2, 129.7, 128.9, 128.3, 127.7, 127.5, 127.3, 126.5, 108.5, 70.7, 65.1, 53.3, 49.5, 47.4, 38.0, 34.4, 23.8, 23.8, 21.6. **HRMS** (ESI) calcd for $C_{30}H_{32}O_4NINaS_2$ $[M+Na]^+$ 684.0709; found: 684.0707.



3ab

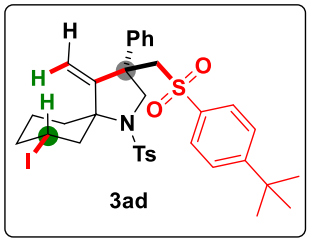
(3R*,5S*,7R*)-7-iodo-3-(((4-methoxyphenyl)sulfonyl)methyl)-4-methylene-3-phenyl-1-tosyl-1-azaspiro[4.5]decane (3ac): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 18:82) to afford the product as an off-white solid (48 mg, yield = 68%; 20:1 dr); Mp. 191-192 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.37 (d, *J* = 8.7 Hz, 4H), 7.10 – 6.99 (m, 5H), 6.73 (d, *J* = 8.9 Hz, 2H), 5.35 (d, *J* = 1.7 Hz, 1H), 5.10 (d, *J* = 1.9 Hz, 1H), 4.70 (d, *J* = 11.0 Hz, 1H), 4.51 – 4.45 (m, 1H), 3.94 (d, *J* = 14.6 Hz, 1H), 3.82 (s, 3H), 3.44 – 3.32 (m,



3ac

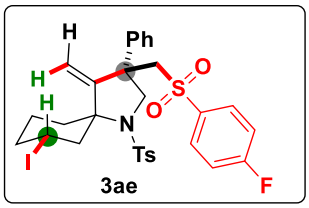
2H), 3.18 (t, *J* = 12.9 Hz, 1H), 2.48 (s, 3H), 2.43 – 2.27 (m, 3H), 2.02 – 1.91 (m, 1H), 1.35 – 1.25 (m, 2H), 0.88 (d, *J* = 12.5 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 163.2, 159.0, 143.5, 137.3, 136.9, 131.5, 129.7, 129.6, 128.3, 127.7, 127.1, 126.5, 114.1, 108.5, 70.7, 65.2, 55.6, 53.2, 49.5, 47.4, 38.0, 34.4, 23.9, 23.7, 21.5. **HRMS** (ESI) calcd for $C_{31}H_{34}O_5NINaS_2$ $[M+Na]^+$ 714.0815; found: 714.0810.

(3R*,5S*,7R*)-3-(((4-(tert-butyl)phenyl)sulfonyl)methyl)-7-iodo-4-methylene-3-phenyl-1-tosyl-1-



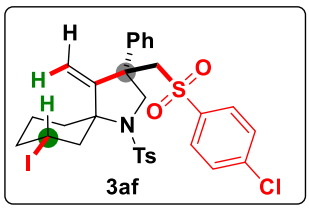
azaspiro[4.5]decane (3ad): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (54 mg, yield = 74%; 7.7:1 dr); Mp. 117-118 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.3 Hz, 2H), 7.38 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.26 – 7.22 (m, 2H), 7.05 – 6.99 (m, 3H), 6.95 (dd, *J* = 11.4, 4.5 Hz, 2H), 5.34 (d, *J* = 1.9 Hz, 1H), 5.10 (d, *J* = 2.0 Hz, 1H), 4.77 (d, *J* = 11.0 Hz, 1H), 4.50 (tt, *J* = 12.7, 4.5 Hz, 1H), 3.97 (d, *J* = 14.7 Hz, 1H), 3.42 (d, *J* = 11.1 Hz, 1H), 3.35 (d, *J* = 14.6 Hz, 1H), 3.19 (t, *J* = 12.9 Hz, 1H), 2.49 (s, 3H), 2.36 (ddd, *J* = 17.5, 13.1, 6.3 Hz, 3H), 2.08 – 1.89 (m, 1H), 1.28 (s, 9H), 1.25 (s, 2H), 0.88 (t, J = 7.0 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.3, 156.9, 143.5, 137.1, 136.9, 136.8, 129.7, 128.2, 127.7, 127.6, 127.3, 127.2, 126.5, 125.9, 108.3, 70.7, 65.1, 53.2, 49.4, 47.4, 38.0, 35.1, 34.5, 31.0, 23.9, 23.8, 21.6. **HRMS** (ESI) calcd for $C_{34}H_{40}O_4NINaS_2$ $[M+Na]^+$ 740.1335; found: 740.1330.

(3R*,5S*,7R*)-3-(((4-fluorophenyl)sulfonyl)methyl)-7-iodo-4-methylene-3-phenyl-1-tosyl-1-



azaspiro[4.5]decane (3ae): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (54 mg, yield = 78%; 4.7:1 dr); Mp. 173-174 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.3 Hz, 2H), 7.44 – 7.34 (m, 4H), 7.13 – 6.97 (m, 5H), 6.95 – 6.87 (m, 2H), 5.36 (d, *J* = 2.0 Hz, 1H), 5.10 (d, *J* = 2.1 Hz, 1H), 4.78 (d, *J* = 10.9 Hz, 1H), 4.53 – 4.45 (m, 1H), 3.99 (d, *J* = 14.8 Hz, 1H), 3.45 – 3.36 (m, 2H), 3.19 (t, *J* = 12.8 Hz, 1H), 2.49 (s, 3H), 2.45 – 2.22 (m, 3H), 2.07 – 1.86 (m, 1H), 1.34 – 1.23 (m, 2H), 0.85 (d, *J* = 11.9 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.5, 164.0 (d, *J* = 247.5 Hz), 159.0 (d, *J* = 22.8 Hz), 157.8 (d, *J* = 9.0 Hz), 143.6, 137.0, 136.9, 136.0, 135.9, 130.5, 130.4, 130.4, 130.3, 129.7, 129.6, 128.4, 128.3, 127.7, 127.6, 127.4, 126.7, 126.6, 126.5, 125.8, 116.2, 116.1, 116.0 (d, *J* = 3.5 Hz), 115.9, 108.4, 70.7, 65.4, 53.3, 49.4, 47.4, 38.0, 34.5, 24.3, 23.8, 21.6. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -103.8. **HRMS** (ESI) calcd for $C_{30}H_{31}O_4NFINaS_2$ $[M+Na]^+$ 702.0615; found: 702.0614.

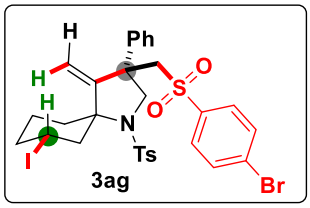
(3R*,5S*,7R*)-3-(((4-chlorophenyl)sulfonyl)methyl)-7-iodo-4-methylene-3-phenyl-1-tosyl-1-



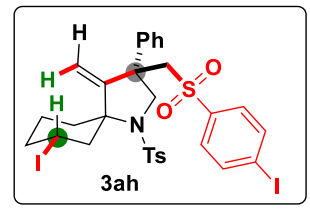
azaspiro[4.5]decane (3af): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (57 mg, yield = 80%; >20:1 dr); Mp. 179-180 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.3 Hz, 2H), 7.39 (s, 2H), 7.32 (d, *J* =

8.7 Hz, 2H), 7.20 (d, *J* = 8.7 Hz, 2H), 7.13 – 7.07 (m, 1H), 7.05 – 6.97 (m, 4H), 5.35 (d, *J* = 2.1 Hz, 1H), 5.10 (d, *J* = 2.1 Hz, 1H), 4.75 (d, *J* = 11.0 Hz, 1H), 4.49 (tt, *J* = 12.7, 4.5 Hz, 1H), 3.98 (d, *J* = 14.8 Hz, 1H), 3.41 – 3.34 (m, 2H), 3.19 (t, *J* = 12.8 Hz, 1H), 2.49 (s, 3H), 2.43 – 2.28 (m, 3H), 2.04 – 1.91 (m, 1H), 1.32 – 1.25 (m, 2H), 0.85 (d, *J* = 13.5 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.0, 143.6, 139.9, 138.3, 137.0, 136.9, 129.7, 129.1, 129.0, 128.4, 127.7, 127.3, 126.6, 108.4, 70.7, 65.4, 53.2, 49.3, 47.4, 38.0, 34.5, 23.8, 23.70, 21.6. **HRMS** (ESI) calcd for $C_{30}H_{31}O_4NClINaS_2$ $[M+Na]^+$ 718.0319; found: 718.0319.

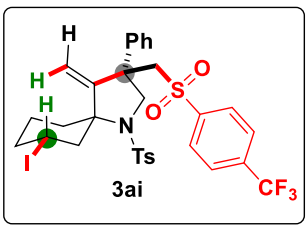
(3R*,5S*,7R*)-3-(((4-bromophenyl)sulfonyl)methyl)-7-iodo-4-methylene-3-phenyl-1-tosyl-1-azaspiro[4.5]decane (3ag): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (59 mg, yield = 79%; >20:1 dr); Mp. 200-201 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 – 7.68 (m, 4H), 7.35 (d, *J* = 8.3 Hz, 2H), 7.13 – 6.99 (m, 7H), 5.39 (d, *J* = 1.9 Hz, 1H), 5.11 (d, *J* = 2.1 Hz, 1H), 4.72 (d, *J* = 11.0 Hz, 1H), 4.53 – 4.44 (m, 1H), 3.91 (d, *J* = 14.6 Hz, 1H), 3.47 (d, *J* = 11.0 Hz, 1H), 3.36 (d, *J* = 14.6 Hz, 1H), 3.13 (t, *J* = 12.8 Hz, 1H), 2.40 – 2.30 (m, 6H), 2.01 – 1.90 (m, 1H), 1.39 – 1.30 (m, 2H), 0.90 (d, *J* = 13.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.3, 144.3, 138.9, 137.2, 137.1, 132.3, 129.6, 129.2, 128.4, 127.7, 127.5, 127.3, 126.4, 109.0, 70.9, 65.1, 53.3, 49.7, 47.6, 38.0, 34.3, 23.7, 23.4, 21.5. **HRMS** (ESI) calcd for $C_{30}H_{31}O_4NBrINaS_2$ $[M+Na]^+$ 761.9814; found: 761.9808.



(3R*,5S*,7R*)-7-iodo-3-(((4-iodophenyl)sulfonyl)methyl)-4-methylene-3-phenyl-1-tosyl-1-azaspiro[4.5]decane (3ah): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 16:84) to afford the product as an off-white solid (65 mg, yield = 81%; >20:1 dr); Mp. 198-199 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.6 Hz, 2H), 7.57 (d, *J* = 8.6 Hz, 2H), 7.35 (d, *J* = 8.3 Hz, 2H), 7.10 (d, *J* = 7.9 Hz, 3H), 7.07 – 6.98 (m, 4H), 5.38 (d, *J* = 1.9 Hz, 1H), 5.10 (d, *J* = 2.1 Hz, 1H), 4.71 (d, *J* = 11.0 Hz, 1H), 4.49 (tt, *J* = 12.6, 4.5 Hz, 1H), 3.90 (d, *J* = 14.6 Hz, 1H), 3.47 (d, *J* = 11.0 Hz, 1H), 3.35 (d, *J* = 14.5 Hz, 1H), 3.13 (t, *J* = 12.8 Hz, 1H), 2.47 – 2.29 (m, 6H), 2.01 – 1.90 (m, 1H), 1.32 – 1.26 (m, 2H), 0.90 (d, *J* = 14.6 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.4, 144.3, 139.6, 138.3, 137.2, 137.2, 129.6, 129.0, 128.4, 127.6, 127.3, 126.4, 108.9, 100.1, 70.9, 65.1, 53.3, 49.8, 47.7, 38.0, 34.3, 23.7, 23.4, 21.5. **HRMS** (ESI) calcd for $C_{30}H_{31}O_4NI_2NaS_2$ $[M+Na]^+$ 809.9676; found: 809.9677.

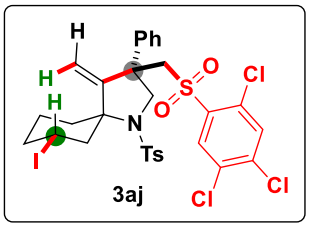


(3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-1-tosyl-3-(((4-(trifluoromethyl)phenyl)sulfonyl)methyl)-1-azaspiro[4.5]decane (3ai): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 81:19) to afford the product as an off-white solid (54 mg, yield = 73%; 20:1 dr); Mp. 203-204 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.4 Hz, 2H), 7.51 – 7.45 (m, 4H), 7.39 (d, *J* = 8.5 Hz, 2H), 7.05 – 6.97 (m, 3H), 6.96 – 6.90 (m, 2H), 5.35 (d, *J* = 2.1 Hz, 1H), 5.11 (d, *J* = 2.2 Hz, 1H), 4.81 (d, *J* = 11.0 Hz, 1H), 4.48 (tt, *J* = 12.7, 4.6 Hz, 1H), 4.05 (d, *J* = 14.9 Hz, 1H), 3.41 (d, *J* = 10.8 Hz, 1H),

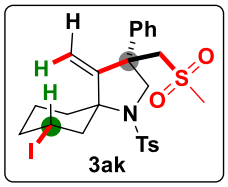


3.38 (d, *J* = 7.0 Hz, 1H), 3.19 (t, *J* = 12.8 Hz, 1H), 2.50 (s, 3H), 2.42 – 2.27 (m, 3H), 2.03 – 1.89 (m, 1H), 1.27 (dt, *J* = 13.7, 5.1 Hz, 2H), 0.82 (d, *J* = 13.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.1, 143.7, 143.1, 136.8, 136.6, 134.8 (q, *J* = 32.7 Hz), 134.4, 129.7, 128.4, 128.1, 127.7, 127.6, 126.5, 125.9 (q, *J* = 3.9 Hz), 125.9, 124.3 (q, *J* = 272.2 Hz), 108.3, 70.8, 65.3, 53.2, 49.2, 47.3, 38.0, 34.6, 23.8, 23.6, 21.6. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -63.5. **HRMS** (ESI) calcd for $C_{31}H_{31}O_4NF_3INaS_2$ $[M+Na]^+$ 752.0583; found: 752.0582.

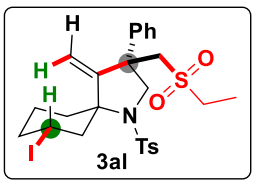
(3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-1-tosyl-3-(((2,4,5-trichlorophenyl)sulfonyl)methyl)-1-azaspiro[4.5]decane (3aj): The title compound was prepared according to GP-3 and purified by column chromatography on silica gel (ethyl acetate/hexane = 25:75) to afford the product as an off-white solid (56 mg, yield = 72%; 13:1 dr); Mp. 190-191 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.3 Hz, 2H), 7.40 (d, *J* = 8.1 Hz, 2H), 7.34 (s, 1H), 7.31 (s, 1H), 7.07 – 7.02 (m, 2H), 6.99 – 6.91 (m, 3H), 5.38 (d, *J* = 2.1 Hz, 1H), 5.20 (d, *J* = 2.2 Hz, 1H), 4.81 (d, *J* = 11.0 Hz, 1H), 4.60 (m, 2H), 3.35 (d, *J* = 15.3 Hz, 1H), 3.28 (d, *J* = 11.0 Hz, 1H), 3.19 (t, *J* = 12.8 Hz, 1H), 2.50 (s, 3H), 2.38 – 2.27 (m, 3H), 2.06 – 1.90 (m, 1H), 1.31 – 1.23 (m, 2H), 0.78 (d, *J* = 13.5 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.6, 143.7, 138.5, 136.8, 136.2, 136.0, 132.8, 132.2, 132.1, 130.1, 129.7, 128.2, 128.1, 127.7, 127.6, 126.3, 108.3, 70.9, 62.5, 53.4, 49.0, 47.2, 38.0, 34.7, 23.7, 21.6, 6.7. **HRMS** (ESI) calcd for $C_{30}H_{29}O_4NCl_3INaS_2$ $[M+Na]^+$ 785.9540; found: 785.9539.



(3R*,5S*,7R*)-7-iodo-4-methylene-3-((methylsulfonyl)methyl)-3-phenyl-1-tosyl-1-azaspiro[4.5]decane (3ak): The title compound was prepared according to GP-3 and purified by column chromatography on silica gel (ethyl acetate/hexane = 15:85) to afford the product as an off-white solid (40 mg, yield = 66%; 17.2:1 dr); Mp. 182-183 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.0 Hz, 2H), 7.36 (d, J = 8.0 Hz, 2H), 7.34 – 7.25 (m, 5H), 5.46 (d, *J* = 2.1 Hz, 1H), 5.20 (d, *J* = 2.2 Hz, 1H), 4.80 (d, *J* = 11.1 Hz, 1H), 4.54 (tt, *J* = 12.8, 4.5 Hz, 1H), 3.80 (d, *J* = 14.9 Hz, 1H), 3.26 (d, *J* = 11.2 Hz, 1H), 3.20 (d, *J* = 12.9 Hz, 1H), 3.13 (dd, *J* = 14.9, 0.9 Hz, 1H), 2.48 (s, 3H), 2.45 – 2.31 (m, 3H), 2.04 – 1.91 (m, 4H), 1.40 – 1.28 (m, 2H), 0.92 (d, *J* = 13.4 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.6, 143.5, 137.3, 136.9, 129.7, 129.1, 128.1, 127.7, 126.6, 108.6, 70.8, 64.6, 52.8, 49.3, 47.4, 42.3, 38.0, 34.5, 23.8, 21.5. **HRMS** (ESI) calcd for $C_{25}H_{30}O_4NINaS_2$ $[M+Na]^+$ 622.0553; found: 622.0548.

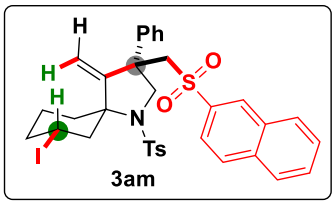


(3R*,5S*,7R*)-3-((ethylsulfonyl)methyl)-7-iodo-4-methylene-3-phenyl-1-tosyl-1-azaspiro[4.5]decane (3al): The title compound was prepared according to GP-3 and purified by column chromatography on silica gel (ethyl acetate/hexane = 15:85) to afford the product as an off-white solid (40 mg, yield = 64%; 14.6:1 dr); Mp. 98-99 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.3 Hz, 2H), 7.30 (dd, *J* = 7.7, 1.9 Hz, 2H), 7.18 – 7.12 (m, 5H), 5.50 (d, *J* = 1.9 Hz, 1H), 5.14 (d, *J* = 2.0 Hz, 1H), 4.68 (d, *J* = 11.1 Hz, 1H), 4.55 (tt, *J* = 12.7, 4.4 Hz, 1H), 3.91 (d, *J* = 14.6 Hz, 1H), 3.85 (d, *J* = 11.1 Hz, 1H), 3.48 (d, *J* = 14.6 Hz, 1H), 3.17 – 3.06 (m, 2H), 2.99 (dq, *J* = 14.5, 7.3 Hz, 1H), 2.67 – 2.60 (m, 1H), 2.37 (s, 3H), 2.30 – 2.21 (m, 1H), 1.98 – 1.87 (m, 1H), 1.42 (d, *J* = 6.2 Hz, 1H), 1.32

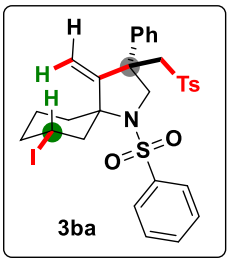


(t, *J* = 7.4 Hz, 3H), 1.29 – 1.23 (m, 2H), 1.08 (d, *J* = 14.1 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.2, 144.4, 138.0, 137.3, 129.6, 128.5, 127.6, 127.4, 126.7, 109.8, 70.0, 64.9, 53.5, 50.2, 48.0, 47.9, 37.9, 33.8, 23.7, 23.4, 21.5, 8.0. **HRMS** (ESI) calcd for $C_{26}H_{32}O_4NINaS_2$ $[M+Na]^+$ 636.0817; found: 636.0815.

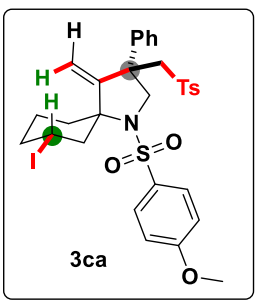
(3R*,5S*,7R*)-7-iodo-4-methylene-3-((naphthalen-2-ylsulfonyl)methyl)-3-phenyl-1-tosyl-1-azaspiro[4.5]decane (3am): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 26:74) to afford the product as an off-white solid (54 mg, yield = 75%; 16.7:1 dr); Mp. 193-194 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 1.2 Hz, 1H), 7.83 (d, *J* = 8.1 Hz, 1H), 7.78 – 7.74 (m, 4H), 7.66 – 7.61 (m, 1H), 7.58 (dd, *J* = 11.0, 4.0 Hz, 1H), 7.48 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.34 (d, *J* = 8.2 Hz, 2H), 7.02 (d, *J* = 7.5 Hz, 2H), 6.84 – 6.74 (m, 3H), 5.34 (d, *J* = 1.8 Hz, 1H), 5.11 (d, *J* = 2.0 Hz, 1H), 4.80 (d, *J* = 11.0 Hz, 1H), 4.53 – 4.44 (m, 1H), 4.05 (d, *J* = 14.7 Hz, 1H), 3.44 (d, *J* = 5.7 Hz, 1H), 3.41 (d, *J* = 1.9 Hz, 1H), 3.19 (t, *J* = 12.9 Hz, 1H), 2.48 (s, 3H), 2.40 – 2.29 (m, 3H), 2.01 – 1.90 (m, 1H), 1.30 – 1.24 (m, 2H), 0.84 (d, *J* = 13.3 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.0, 143.5, 136.9, 136.6, 134.8, 131.8, 129.8, 129.6, 129.4, 129.2, 129.2, 128.1, 127.7, 127.4, 127.2, 126.4, 121.9, 108.4, 70.7, 65.0, 53.3, 49.5, 47.4, 38.0, 34.5, 23.9, 23.7, 21.6. **HRMS** (ESI) calcd for $C_{34}H_{34}O_4NINaS_2$ $[M+Na]^+$ 734.0866; found: 734.0865.



(3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-1-(phenylsulfonyl)-3-(tosylmethyl)-1-azaspiro[4.5]decane (3ba): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (53 mg, yield = 80%; >20:1 dr); Mp. 197-198 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.1 Hz, 2H), 7.67 – 7.61 (m, 1H), 7.60 – 7.54 (m, 2H), 7.33 (d, *J* = 8.3 Hz, 2H), 7.11 – 6.97 (m, 7H), 5.36 (d, *J* = 2.0 Hz, 1H), 5.10 (d, *J* = 2.1 Hz, 1H), 4.72 (d, *J* = 11.0 Hz, 1H), 4.49 (tt, *J* = 12.7, 4.5 Hz, 1H), 3.93 (d, *J* = 14.6 Hz, 1H), 3.46 (d, *J* = 11.1 Hz, 1H), 3.36 (d, *J* = 14.6 Hz, 1H), 3.18 (t, *J* = 12.8 Hz, 1H), 2.44 – 2.30 (m, 6H), 2.04 – 1.91 (m, 1H), 1.40 – 1.25 (m, 2H), 0.90 (d, *J* = 13.5 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.7, 144.2, 139.9, 137.3, 137.1, 132.6, 129.5, 129.0, 128.3, 127.6, 127.5, 127.1, 126.5, 108.7, 70.7, 65.1, 53.3, 49.6, 47.5, 38.0, 34.3, 23.7, 21.5. **HRMS** (ESI) calcd for $C_{30}H_{32}O_4NINaS_2$ $[M+Na]^+$ 684.0709; found: 684. 0707.



(3R*,5S*,7R*)-7-iodo-1-((4-methoxyphenyl)sulfonyl)-4-methylene-3-phenyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3ca): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 19:81) to afford the product as an off-white solid (48 mg, yield = 69%; >20:1 dr); Mp. 180-181 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.9 Hz, 2H), 7.34 (d, *J* = 8.3 Hz, 2H), 7.11 – 6.99 (m, 9H), 5.36 (d, J = 1.9 Hz, 1H), 5.09 (d, *J* = 2.0 Hz, 1H), 4.68 (d, *J* = 11.0 Hz, 1H), 4.69 – 4.46 (m, 1H), 3.95 (s, 1H), 3.92 (s, 3H), 3.42 (d, *J* = 11.0 Hz, 1H), 3.36 (d, *J* = 14.6 Hz, 1H), 3.20 (t, *J* = 12.9 Hz, 1H), 2.43 – 2.38 (m, 2H), 2.35 (s, 3H), 2.34 – 2.31 (m, 1H), 2.02 – 1.92 (m, 1H), 1.37 – 1.25 (m, 2H), 0.90 (d, *J* = 13.7 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.9, 159.0, 144.2, 137.4, 137.2,



131.6, 129.8, 129.5, 128.3, 127.6, 127.1, 126.5, 114.1, 108.6, 70.6, 65.1, 55.7, 53.2, 49.6, 47.4, 38.0, 34.3, 23.9, 23.8, 21.5. **HRMS** (ESI) calcd for $C_{31}H_{34}O_5NINaS_2$ $[M+Na]^+$ 714.0815; found: 714.0813.

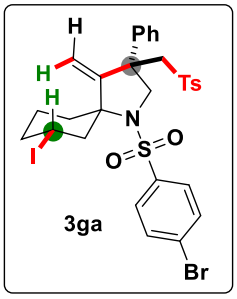
(3R*,5S*,7R*)-1-((4-(tert-butyl)phenyl)sulfonyl)-7-iodo-4-methylene-3-phenyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3da): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (51 mg, yield = 70%; 20:1 dr); Mp. 116-117 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.7 Hz, 2H), 7.57 (d, *J* = 8.7 Hz, 2H), 7.34 (d, *J* = 8.3 Hz, 2H), 7.08 (d, *J* = 8.0 Hz, 2H), 7.05 – 6.92 (m, 5H), 5.36 (d, J = 1.9 Hz, 1H), 5.09 (d, J = 2.0 Hz, 1H), 4.71 (d, *J* = 11.1 Hz, 1H), 4.54 – 4.45 (m, 1H), 3.93 (d, *J* = 14.6 Hz, 1H), 3.46 (d, *J* = 11.1 Hz, 1H), 3.37 (d, *J* = 14.6 Hz, 1H), 3.20 (t, J = 12.9 Hz, 1H), 2.50 – 2.37 (m, 2H), 2.35 (s, 3H), 2.03 – 1.92 (m, 1H), 1.40 (s, 9H), 1.37 – 1.30 (m, 2H), 1.25 (d, *J* = 6.0 Hz, 1H), 0.91 (d, *J* = 6.3 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.1, 156.5, 144.2, 137.4, 136.9, 129.5, 128.3, 127.6, 127.6, 127.1, 126.5, 126.0, 108.6, 70.7, 65.2, 53.2, 49.6, 47.6, 38.0, 35.2, 34.3, 31.2, 24.7, 23.8, 21.5. **HRMS** (ESI) calcd for $C_{34}H_{40}O_4NINaS_2$ $[M+Na]^+$ 740.1335; found: 740.1335.

(3R*,5S*,7R*)-1-((4-fluorophenyl)sulfonyl)-7-iodo-4-methylene-3-phenyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3ea): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 16:84) to afford the product as an off-white solid (50 mg, yield = 74%; >20:1 dr); Mp. 200-201 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 – 7.86 (m, 2H), 7.35 (d, *J* = 8.3 Hz, 2H), 7.26 – 7.20 (m, 2H), 7.12 – 7.01 (m, 7H), 5.39 (d, *J* = 2.1 Hz, 1H), 5.11 (d, *J* = 2.1 Hz, 1H), 4.73 (d, *J* = 11.0 Hz, 1H), 4.75 – 4.45 (m, 1H), 3.92 (d, *J* = 14.6 Hz, 1H), 3.48 (d, *J* = 11.0 Hz, 1H), 3.36 (d, *J* = 14.6 Hz, 1H), 3.14 (t, *J* = 12.9 Hz, 1H), 2.42 – 2.37 (m, 2H), 2.36 (s, 3H), 2.35 – 2.33 (m, 1H), 2.02 – 1.91 (m, 1H), 1.39 – 1.25 (m, 2H), 0.92 (d, *J* = 13.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.3, 163.7 (d, *J* = 248.7 Hz), 158.5 (d, *J* = 24.3 Hz), 144.3, 137.2, 137.1, 136.1, 136.0, 130.4, 130.3, 129.6, 128.4, 127.5, 127.2, 126.8, 126.5, 116.4 (d, *J* = 7.7 Hz), 116.3 (d, *J* = 7.7 Hz), 116.1 (d, *J* = 7.7 Hz), 114.3 (d, *J* = 21.1 Hz), 108.9, 70.8, 65.1, 53.3, 49.7, 47.6, 38.0, 34.3, 23.7, 23.5, 21.5. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -105.1. **HRMS** (ESI) calcd for $C_{30}H_{31}O_4NFINaS_2$ $[M+Na]^+$ 702.0615; found: 702.0613.

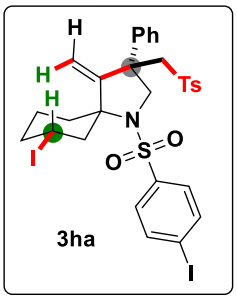
(3R*,5S*,7R*)-1-((4-chlorophenyl)sulfonyl)-7-iodo-4-methylene-3-phenyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3fa): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 16:84) to afford the product as an off-white solid (56 mg, yield = 78%; >20:1 dr); Mp. 199-200 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.7 Hz, 2H), 7.53 (d, *J* = 8.7 Hz, 2H), 7.35 (d, *J* = 8.3 Hz, 2H), 7.14 – 7.00 (m, 7H), 5.38 (d, *J* = 1.9 Hz, 1H), 5.11 (d, *J* = 2.1 Hz, 1H), 4.73 (d, *J* = 11.0

Hz, 1H), 4.53 – 4.44 (m, 1H), 3.91 (d, *J* = 14.6 Hz, 1H), 3.47 (d, *J* = 11.0 Hz, 1H), 3.36 (d, *J* = 14.6 Hz, 1H), 3.13 (t, *J* = 12.8 Hz, 1H), 2.40 – 2.31 (m, 6H), 2.01 – 1.90 (m, 1H), 1.37 – 1.28 (m, 2H), 0.91 (d, *J* = 13.6 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.4, 144.3, 139.2, 138.4, 137.2, 137.1, 129.6, 129.3, 129.1, 128.4, 127.5, 127.3, 126.4, 108.9, 70.9, 65.1, 53.3, 49.7, 47.6, 38.0, 34.3, 23.7, 23.4, 21.5. **HRMS** (ESI) calcd for $C_{30}H_{31}O_4NClINaS_2$ $[M+Na]^+$ 718.0319; found: 702.0314.

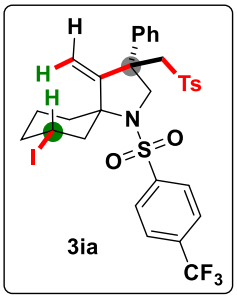
(3R*,5S*,7R*)-1-((4-bromophenyl)sulfonyl)-7-iodo-4-methylene-3-phenyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3ga): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 15:85) to afford the product as an off-white solid (59 mg, yield = 80%; >20:1 dr); Mp. 206-207 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 – 7.68 (m, 4H), 7.35 (d, *J* = 8.3 Hz, 2H), 7.13 – 6.99 (m, 7H), 5.39 (d, *J* = 1.9 Hz, 1H), 5.11 (d, *J* = 2.1 Hz, 1H), 4.72 (d, *J* = 11.0 Hz, 1H), 4.53 – 4.44 (m, 1H), 3.91 (d, *J* = 14.6 Hz, 1H), 3.47 (d, *J* = 11.0 Hz, 1H), 3.36 (d, *J* = 14.6 Hz, 1H), 3.13 (t, *J* = 12.8 Hz, 1H), 2.40 – 2.30 (m, 6H), 2.01 – 1.90 (m, 1H), 1.39 – 1.30 (m, 2H), 0.90 (d, *J* = 13.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.3, 144.3, 138.9, 137.2, 137.1, 132.3, 129.6, 129.2, 128.4, 127.7, 127.5, 127.3, 126.4, 109.0, 70.9, 65.1, 53.3, 49.7, 47.6, 38.0, 34.3, 23.7, 23.4, 21.5. **HRMS** (ESI) calcd for $C_{30}H_{31}O_4NBrINaS_2$ $[M+Na]^+$ 761.9814; found: 761.9808.



(3R*,5S*,7R*)-7-iodo-1-((4-iodophenyl)sulfonyl)-4-methylene-3-phenyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3ha): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 15:85) to afford the product as an off-white solid (65 mg, yield = 81%; >20:1 dr); Mp. 203-204 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.6 Hz, 2H), 7.57 (d, *J* = 8.6 Hz, 2H), 7.35 (d, *J* = 8.3 Hz, 2H), 7.10 (d, *J* = 7.9 Hz, 3H), 7.07 – 6.98 (m, 4H), 5.38 (d, *J* = 1.9 Hz, 1H), 5.10 (d, *J* = 2.1 Hz, 1H), 4.71 (d, *J* = 11.0 Hz, 1H), 4.49 (tt, *J* = 12.6, 4.5 Hz, 1H), 3.90 (d, *J* = 14.6 Hz, 1H), 3.47 (d, *J* = 11.0 Hz, 1H), 3.35 (d, *J* = 14.5 Hz, 1H), 3.13 (t, *J* = 12.8 Hz, 1H), 2.47 – 2.29 (m, 6H), 2.01 – 1.90 (m, 1H), 1.32 – 1.26 (m, 2H), 0.90 (d, *J* = 14.6 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.4, 144.3, 139.6, 138.3, 137.2, 137.2, 129.6, 129.0, 128.4, 127.6, 127.3, 126.4, 108.9, 100.1, 70.9, 65.1, 53.3, 49.8, 47.7, 38.0, 34.3, 23.7, 23.4, 21.5. **HRMS** (ESI) calcd for $C_{30}H_{31}O_4NI_2NaS_2$ $[M+Na]^+$ 809.9676; found: 809.9677.

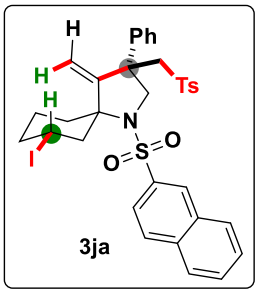


(3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-3-(tosylmethyl)-1-((4-(trifluoromethyl)phenyl)sulfonyl)-1-azaspiro[4.5]decane (3ia): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 19:81) to afford the product as an off-white solid (49 mg, yield = 67%; >20:1 dr); Mp. 199-200 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.2 Hz, 2H), 7.80 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.3 Hz, 2H), 7.13 – 7.06 (m, 3H), 7.06 – 6.97 (m, 4H), 5.41 (d, *J* = 2.0 Hz, 1H), 5.13 (d, *J* = 2.2 Hz, 1H), 4.75 (d, *J* = 11.1 Hz, 1H), 4.49 (tt, *J* = 12.7, 4.5 Hz, 1H), 3.90 (d, *J* = 14.6 Hz, 1H), 3.55 (d, *J* = 11.1 Hz, 1H), 3.37 (d, *J* = 14.6 Hz, 1H), 3.13 (t, *J* = 12.8 Hz, 1H), 2.44 – 2.32 (m, 6H), 2.02 – 1.91 (m,

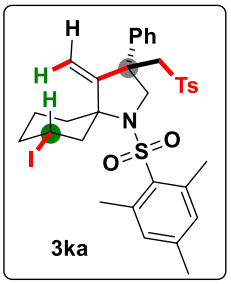


1H), 1.43 – 1.25 (m, 2H), 0.94 (d, *J* = 13.2 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.9, 144.3, 143.4, 137.2, 137.1, 134.4, 134.1 (q, *J* = 32.7 Hz), 129.6, 128.3, 128.1, 127.5, 127.3, 126.4, 126.2 (q, *J* = 3.8 Hz), 126.1, 126.1 (q, *J* = 3.8 Hz), 124.6 (q, *J* = 272.0 Hz), 121.8 (q, *J* = 32.0 Hz), 109.3, 71.0, 64.9, 53.3, 49.9, 47.8, 37.9, 34.1, 23.7, 23.2, 21.5. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -63.0. **HRMS** (ESI) calcd for $C_{31}H_{31}O_4NF_3INaS_2$ $[M+Na]^+$ 752.0583; found: 752.0582

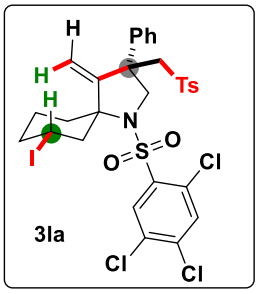
(3R*,5S*,7R*)-7-iodo-4-methylene-1-(naphthalen-2-ylsulfonyl)-3-phenyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3ja): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 25:75) to afford the product as an off-white solid (55 mg, yield = 77%; >20:1 dr); Mp. 191-192 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 1.6 Hz, 1H), 8.05 (d, *J* = 7.9 Hz, 1H), 7.97 (dd, *J* = 11.1, 8.5 Hz, 2H), 7.82 (dd, *J* = 8.7, 1.9 Hz, 1H), 7.72 – 7.61 (m, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.08 – 6.98 (m, 5H), 6.91 (dd, *J* = 10.9, 4.5 Hz, 2H), 5.36 (d, *J* = 2.0 Hz, 1H), 5.08 (d, *J* = 2.1 Hz, 1H), 4.77 (d, *J* = 11.0 Hz, 1H), 4.53 – 4.44 (m, 1H), 3.90 (d, *J* = 14.6 Hz, 1H), 3.51 (d, *J* = 11.1 Hz, 1H), 3.38 – 3.25 (m, 2H), 2.50 – 2.34 (m, 3H), 2.33 (s, 3H), 2.07 – 1.95 (m, 1H), 1.38 – 1.29 (m, 2H), 0.95 (d, *J* = 13.4 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.7, 144.2, 137.3, 137.1, 136.7, 134.8, 132.1, 129.6, 129.5, 129.2, 129.0, 128.9, 128.2, 127.8, 127.5, 127.1, 126.4, 123.0, 108.7, 70.8, 65.1, 53.3, 49.6, 47.7, 38.0, 34.3, 23.8, 23.7, 21.5. **HRMS** (ESI) calcd for $C_{34}H_{34}O_4NINaS_2$ $[M+Na]^+$ 734.0866; found: 734.0863.



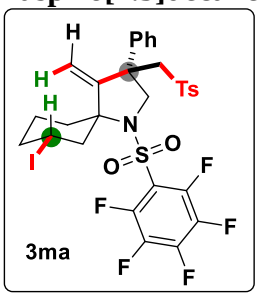
(3R*,5S*,7R*)-7-iodo-1-(mesitylsulfonyl)-4-methylene-3-phenyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3ka): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 20:80) to afford the product as an off-white solid (34 mg, yield = 47%; 9.1:1 dr); Mp. 195-196 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.3 Hz, 2H), 7.12 (d, *J* = 8.2 Hz, 2H), 7.07 (d, *J* = 9.5 Hz, 1H), 7.04 (s, 2H), 6.98 (t, *J* = 7.7 Hz, 2H), 6.69 (d, *J* = 7.6 Hz, 2H), 5.48 (d, *J* = 1.8 Hz, 1H), 5.12 (d, *J* = 2.0 Hz, 1H), 4.58 – 4.50 (m, 1H), 4.00 (d, *J* = 10.6 Hz, 1H), 3.79 (d, *J* = 14.4 Hz, 1H), 3.63 (d, *J* = 10.7 Hz, 1H), 3.40 (d, *J* = 14.4 Hz, 1H), 3.25 (t, *J* = 12.9 Hz, 1H), 2.83 (d, *J* = 9.0 Hz, 1H), 2.69 (dd, *J* = 13.4, 4.9 Hz, 1H), 2.55 (s, 6H), 2.41 – 2.37 (m, 7H), 2.03 (qd, *J* = 13.1, 4.3 Hz, 1H), 1.56 – 1.46 (m, 2H), 1.15 (d, *J* = 13.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.4, 144.4, 143.5, 140.9, 137.9, 137.4, 132.1, 129.7, 128.0, 127.5, 127.1, 126.3, 109.4, 71.2, 65.0, 52.3, 49.8, 47.6, 38.1, 33.5, 23.9, 23.0, 21.6, 21.2. **HRMS** (ESI) calcd for $C_{33}H_{39}O_4NIS_2$ $[M+H]^+$ 704.1359; found: 704.1355.



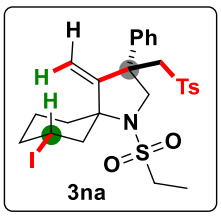
(3R*,5S*,7R*)-7-iodo-4-methylene-3-phenyl-3-(tosylmethyl)-1-((2,4,5-trichlorophenyl)sulfonyl)-1-azaspiro[4.5]decane (3la): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 26:74) to afford the product as an off-white solid (53 mg, yield = 68%; 20:1 dr); Mp. 126-127 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.23 (s, 1H), 7.63 (s, 1H), 7.41 (d, *J* = 8.3 Hz, 2H), 7.18 – 7.01 (m, 7H), 5.48 (d, *J* = 1.9 Hz, 1H), 5.12 (d, *J* = 2.1 Hz, 1H), 4.53 – 4.45 (m, 1H), 4.42 (d, *J* = 11.1 Hz, 1H), 3.79 (dd, *J* = 12.9, 7.6 Hz, 2H), 3.46 (d, *J* = 14.6 Hz, 1H), 3.12 (t, *J* = 12.8 Hz, 1H), 2.66 – 2.58 (m, 1H), 2.49 (td, J = 13.3, 5.2 Hz, 1H), 2.41 – 2.38 (m, 4H), 2.08 – 1.87 (m, 2H), 1.51 – 1.43 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 157.6, 144.5, 138.3, 137.5, 137.4, 136.7, 133.4, 133.2, 131.8, 131.5, 129.7, 128.4, 127.5, 127.5, 126.4, 110.3, 72.0, 64.4, 53.7, 50.4, 48.0, 38.0, 33.6, 31.6, 23.7, 22.6, 21.6. **HRMS** (ESI) calcd for $C_{30}H_{29}O_4NCl_3INaS_2$ $[M+Na]^+$ 785.9540; found: 785.9537.



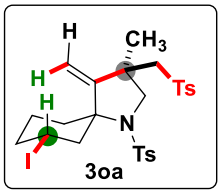
(3R*,5S*,7R*)-7-iodo-4-methylene-1-((perfluorophenyl)sulfonyl)-3-phenyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3ma): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 27:73) to afford the product as an off-white solid (48 mg, yield = 64%; >20:1 dr); Mp. 207-208 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.42 (d, *J* = 8.3 Hz, 2H), 7.19 – 7.09 (m, 7H), 5.48 (d, *J* = 2.1 Hz, 1H), 5.15 (d, *J* = 2.2 Hz, 1H), 4.75 (d, *J* = 11.3 Hz, 1H), 4.49 (tt, *J* = 12.7, 4.4 Hz, 1H), 3.85 (dd, *J* = 12.9, 10.9 Hz, 2H), 3.44 (d, *J* = 14.7 Hz, 1H), 3.04 (t, *J* = 12.7 Hz, 1H), 2.59 – 2.50 (m, 1H), 2.43 – 2.33 (m, 5H), 2.01 – 1.90 (m, 1H), 1.49 – 1.33 (m, 2H), 1.13 (d, *J* = 13.3 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.0 (d, *J* = 250 Hz), 144.6 (d, *J* = 20 Hz), 137.3 (d, *J* = 15 Hz), 137.2 (d, J = 15 Hz), 129.7, 128.5, 127.6, 127.5, 126.5, 110.2, 72.4, 64.4, 53.5, 50.2, 47.1, 37.9, 33.6, 23.6, 22.2, 21.5. **HRMS** (ESI) calcd for $C_{30}H_{27}O_4NF_3INaS_2$ $[M+Na]^+$ 774.0238; found: 774.0236. ^{19}F NMR (377 MHz, $CDCl_3$) δ -133.4, -145.4, -158.2.



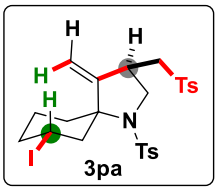
(3R*,5S*,7R*)-1-(ethylsulfonyl)-7-iodo-4-methylene-3-phenyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3na): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 16:84) to afford the product as an off-white solid (45 mg, yield = 71%; 14.6:1 dr); Mp. 90-91 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.3 Hz, 2H), 7.30 (dd, *J* = 7.7, 1.9 Hz, 2H), 7.18 – 7.12 (m, 5H), 5.50 (d, *J* = 1.9 Hz, 1H), 5.14 (d, *J* = 2.0 Hz, 1H), 4.68 (d, *J* = 11.1 Hz, 1H), 4.55 (tt, *J* = 12.7, 4.4 Hz, 1H), 3.91 (d, *J* = 14.6 Hz, 1H), 3.85 (d, *J* = 11.1 Hz, 1H), 3.48 (d, *J* = 14.6 Hz, 1H), 3.17 – 3.06 (m, 2H), 2.99 (dq, *J* = 14.5, 7.3 Hz, 1H), 2.67 – 2.60 (m, 1H), 2.37 (s, 3H), 2.30 – 2.21 (m, 1H), 1.98 – 1.87 (m, 1H), 1.42 (d, J = 6.2 Hz, 1H), 1.32 (t, *J* = 7.4 Hz, 3H), 1.29 – 1.23 (m, 2H), 1.08 (d, *J* = 14.1 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.2, 144.4, 138.0, 137.3, 129.6, 128.5, 127.6, 127.4, 126.7, 109.8, 70.0, 64.9, 53.5, 50.2, 48.0, 47.9, 37.9, 33.8, 23.7, 23.4, 21.5, 8.0. **HRMS** (ESI) calcd for $C_{26}H_{32}O_4NINaS_2$ $[M+Na]^+$ 636.0817; found: 636.0815.



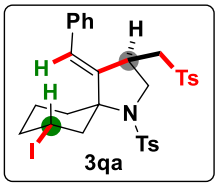
(3R*,5S*,7R*)-7-iodo-3-methyl-4-methylene-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3oa): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 15:85) to afford the product as an off-white solid (35 mg, yield = 56%; 6.7:1 dr); Mp. 80-81 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.59 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.6 Hz, 2H), 7.31 (d, *J* = 8.5 Hz, 2H), 5.31 (d, *J* = 1.4 Hz, 1H), 4.99 (d, *J* = 1.6 Hz, 1H), 4.97 (d, *J* = 1.6 Hz, 1H), 3.87 (d, *J* = 10.5 Hz, 1H), 3.28 – 3.18 (m, 1H), 3.14 (d, *J* = 10.5 Hz, 1H), 3.00 (s, 2H), 2.81 – 2.69 (m, 1H), 2.47 (s, 3H), 2.45 (s, 3H), 2.44 – 2.37 (m, 2H), 2.12 – 2.01 (m, 1H), 1.69 (d, *J* = 11.6 Hz, 2H), 1.42 (s, 3H), 1.33 – 1.24 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 160.9, 144.8, 143.4, 138.2, 137.7, 129.9, 129.6, 127.6, 127.5, 109.2, 70.4, 63.3, 55.7, 47.8, 44.3, 38.0, 33.5, 23.7, 23.6, 22.8, 21.6, 21.6. **HRMS** (ESI) calcd for $C_{26}H_{32}O_4NINaS_2$ $[M+Na]^+$ 636.0709; found: 639.0707.



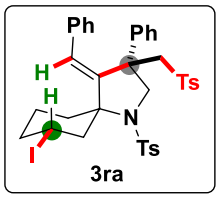
(3R*,5S*,7R*)-7-iodo-4-methylene-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3pa): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 15:85) to afford the product as an off-white solid (31 mg, yield = 50%; 5.5:1 dr); Mp. 82-83 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.4 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.36 (d, *J* = 7.9 Hz, 2H), 7.32 (d, *J* = 7.9 Hz, 2H), 5.24 (s, 1H), 4.90 (s, 1H), 4.43 – 4.31 (m, 1H), 3.74 – 365 (m, 1H), 3.37 (t, *J* = 12.9 Hz, 1H), 3.22 – 3.07 (m, 3H), 2.98 – 2.87 (m, 1H), 2.63 – 2.52 (m, 1H), 2.47 (s, 3H), 2.46 (s, 3H), 2.41 (d, *J* = 13.9 Hz, 1H), 2.28 – 2.21 (m, 1H), 2.10 – 1.98 (m, 1H), 1.80 (d, *J* = 13.2 Hz, 1H), 1.60 – 1.50 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 154.5, 145.2, 143.4, 137.8, 136.1, 130.1, 130.1, 129.6, 127.9, 127.2, 108.9, 70.1, 58.6, 50.8, 47.5, 38.1, 36.9, 33.9, 24.1, 23.3, 21.7, 21.5. **HRMS** (ESI) calcd for $C_{25}H_{30}O_4NINaS_2$ $[M+Na]^+$ 622.0553; found: 622.0551.



(3R*,5S*,7R*)-4-((E)-benzylidene)-7-iodo-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3qa): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 14:86) to afford the product as an off-white solid (34 mg, yield = 49%; 20:1 dr); Mp. 188-189 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.75 (d, *J* = 8.3 Hz, 2H), 7.44 – 7.20 (m, 8H), 7.11 (brs, 1H), 6.92 (brs, 1H), 4.23 (dd, *J* = 15.0, 8.8 Hz, 1H), 3.88 (dd, *J* = 14.2, 2.1 Hz, 1H), 3.74 (dd, *J* = 15.0, 7.4 Hz, 1H), 3.06 (dd, *J* = 14.2, 12.0 Hz, 1H), 2.49 (s, 3H), 2.44 (s, 3H), 2.37 (ddd, *J* = 12.1, 9.2, 4.7 Hz, 1H), 2.08 – 2.01 (m, 1H), 1.96 (dd, *J* = 13.7, 2.8 Hz, 1H), 1.68 (ddd, *J* = 13.3, 8.2, 3.3 Hz, 1H), 1.56 (d, *J* = 12.6 Hz, 1H), 1.44 (d, *J* = 10.0 Hz, 1H), 1.28 (dd, *J* = 8.9, 5.5 Hz, 1H), 1.25 – 1.17 (m, 1H), 0.97 – 0.78 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.7, 145.0, 144.1, 143.7, 136.2, 135.2, 129.9, 129.8, 128.7, 128.6, 127.8, 127.3, 97.3, 74.2, 59.6, 50.1, 45.6, 37.6, 33.1, 25.4, 23.3, 21.7, 21.6, 21.4. **HRMS** (ESI) calcd for $C_{31}H_{34}O_4NINaS_2$ $[M+Na]^+$ 698.0866; found: 698.0865.

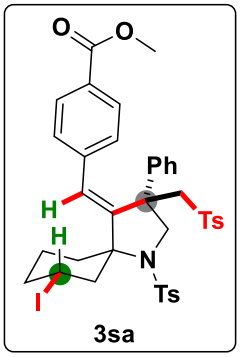


(3R*,5S*,7R*)-4-((E)-benzylidene)-7-iodo-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (**3ra**):

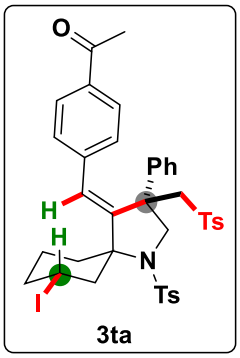


The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 15:85) to afford the product as an off-white solid (46 mg, yield = 60%; 4.3:1 dr); Mp. 189-190 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 1.7 Hz, 1H), 7.25 (d, *J* = 1.5 Hz, 1H), 7.23 – 7.12 (m, 11H), 7.09 – 7.05 (m, 4H), 4.76 (tt, *J* = 12.6, 4.2 Hz, 1H), 4.26 (d, *J* = 10.9 Hz, 1H), 4.21 (d, *J* = 10.9 Hz, 1H), 3.57 (d, *J* = 14.7 Hz, 1H), 3.46 (d, *J* = 14.7 Hz, 1H), 3.21 (t, *J* = 12.9 Hz, 1H), 2.97 – 2.87 (m, 1H), 2.71 (td, *J* = 13.6, 5.2 Hz, 1H), 2.51 (d, *J* = 13.0 Hz, 1H), 2.41 (s, 3H), 2.35 (s, 3H), 2.20 – 2.09 (m, 1H), 1.85 – 1.74 (m, 1H), 1.67 (d, *J* = 14.0 Hz, 1H), 1.60 – 1.54 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.8, 144.1, 143.0, 141.7, 138.2, 137.8, 135.6, 129.4, 129.3, 129.1, 128.4, 128.2, 127.5, 127.3, 127.3, 127.1, 126.5, 71.6, 61.6, 56.7, 51.4, 47.6, 38.2, 33.1, 24.2, 23.9, 21.5, 21.5. **HRMS** (ESI) calcd for $C_{37}H_{38}O_4NINaS_2$ $[M+Na]^+$ 774.1179; found: 774.1175.

methyl 4-((*E*)-((3R*,5S*,7R*)-7-iodo-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decan-4-ylidene)methyl)benzoate (**3sa**): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 25:75) to afford the product as an off-white solid (44 mg, yield = 54%; 4.7:1 dr); Mp. 207-209 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.3 Hz, 2H), 7.21 (ddd, *J* = 8.1, 4.2, 1.7 Hz, 6H), 7.16 (d, *J* = 7.3 Hz, 4H), 7.13 – 7.11 (m, 1H), 7.09 (d, *J* = 3.4 Hz, 2H), 7.07 (s, 1H), 4.78 – 4.65 (m, 1H), 4.34 (d, *J* = 10.9 Hz, 1H), 4.14 (d, *J* = 10.9 Hz, 1H), 3.91 (s, 3H), 3.90 (d, *J* = 3.6 Hz, 1H), 3.50 – 3.40 (m, 2H), 3.19 (t, *J* = 12.9 Hz, 1H), 3.00 (dd, *J* = 13.1, 4.1 Hz, 1H), 2.78 (td, *J* = 13.4, 5.1 Hz, 1H), 2.53 (d, *J* = 11.2 Hz, 1H), 2.40 (s, 3H), 2.35 (s, 3H), 2.16 (dd, *J* = 13.3, 4.0 Hz, 1H), 1.78 (d, *J* = 13.6 Hz, 1H), 1.62 (d, *J* = 14.0 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.4, 151.3, 144.4, 143.0, 142.2, 140.4, 138.2, 137.8, 129.6, 129.3, 129.3, 129.1, 128.5, 127.4, 127.1, 127.0, 125.7, 71.6, 61.1, 56.8, 52.2, 51.4, 47.4, 38.2, 32.7, 24.3, 23.3, 21.5, 21.5. **HRMS** (ESI) calcd for $C_{39}H_{40}O_6NINaS_2$ $[M+Na]^+$ 832.1233; found: 832.1225.

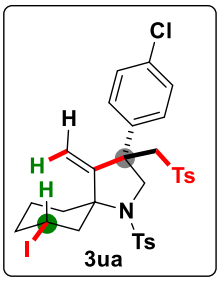


1-(4-((*E*)-((3R*,5S*,7R*)-7-iodo-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decan-4-ylidene)methyl)phenyl)ethan-1-one (**3ta**): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 27:73) to afford the product as an off-white solid (41 mg, yield = 51%; 5.4:1 dr); Mp. 205-207 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.3 Hz, 2H), 7.44 (d, *J* = 8.3 Hz, 2H), 7.25 – 7.19 (m, 5H), 7.18 (s, 1H), 7.14 (d, *J* = 8.6 Hz, 6H), 7.08 (d, *J* = 8.0 Hz, 2H), 4.79 – 4.71 (m, 1H), 4.36 (d, *J* = 10.9 Hz, 1H), 4.12 (d, *J* = 10.9 Hz, 1H), 3.46 (q, *J* = 14.7 Hz, 2H), 3.19 (t, *J* = 12.9 Hz, 1H), 3.07 – 2.98 (m, 1H), 2.78 (td, *J* = 13.3, 5.0 Hz, 1H), 2.55 (s, 3H), 2.53 (d, *J* = 3.0 Hz, 1H), 2.40 (s, 3H), 2.35 (s, 3H), 2.16 (qd, *J* = 13.1, 4.2 Hz, 1H), 1.82 (dd, *J* = 20.8, 13.8 Hz, 2H), 1.64 (d, *J* = 12.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.4, 151.3, 144.4, 143.0, 142.2, 140.4, 138.2, 137.8, 129.6,



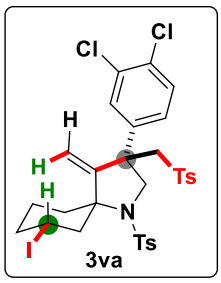
129.3, 129.3, 129.1, 128.5, 127.4, 127.1, 127.0, 125.7, 71.6, 61.1, 56.8, 52.2, 51.4, 47.4, 38.2, 32.7, 24.3, 23.3, 21.5, 21.5. **HRMS** (ESI) calcd for $C_{39}H_{40}O_5NINaS_2$ $[M+Na]^+$ 816.1284; found: 816.1279.

(3R*,5S*,7R*)-3-(4-chlorophenyl)-7-iodo-4-methylene-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (3ua): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 20:80) to afford the product as an off-white solid (52 mg, yield = 73%; > 20:1 dr); 178-181 °C;



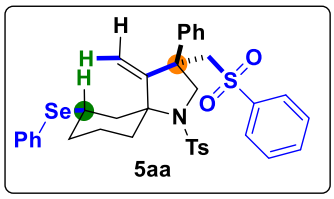
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.34 – 7.29 (m, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 6.99 – 6.89 (m, 4H), 5.35 (s, 1H), 5.09 (s, 1H), 4.70 (d, *J* = 11.1 Hz, 1H), 4.51 – 4.43 (m, 1H), 3.92 (d, *J* = 14.7 Hz, 1H), 3.34 (dd, *J* = 14.0, 13.1 Hz, 2H), 3.15 (t, *J* = 12.8 Hz, 1H), 2.49 (s, 3H), 2.44 (d, J = 9.3 Hz, 1H), 2.40 (s, 3H), 2.38 – 2.27 (m, 2H), 2.02 – 1.90 (m, 1H), 1.35 – 1.28 (m, 2H), 0.88 (d, *J* = 14.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.8, 144.6, 143.7, 136.7, 136.7, 135.9, 133.3, 129.7, 129.6, 128.3, 128.0, 127.7, 127.5, 108.6, 64.9, 53.3, 49.0, 47.3, 37.9, 34.6, 23.7, 23.6, 21.6. **HRMS** (ESI) calcd for $C_{33}H_{33}O_4NClINaS_2$ $[M+Na]^+$ 732.0584; found: 732.0585.

(3R*,5S*,7R*)-3-(3,4-dichlorophenyl)-7-iodo-4-methylene-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane



(3ua): The title compound was prepared according to GP-2 and purified by column chromatography on silica gel (ethyl acetate/hexane = 21:79) to afford the product as an off-white solid (55 mg, yield = 72%; >20:1 dr); Mp. 215-218 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.3 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.14 – 7.09 (m, 2H), 7.07 (d, *J* = 8.5 Hz, 1H), 7.00 (dd, *J* = 8.5, 2.3 Hz, 1H), 6.85 (s, 1H), 5.39 (s, 1H), 5.13 (s, 1H), 4.64 (d, *J* = 11.3 Hz, 1H), 4.51 – 4.42 (m, 1H), 3.94 (d, *J* = 14.8 Hz, 1H), 3.36 (d, *J* = 11.3 Hz, 1H), 3.32 (d, *J* = 14.8 Hz, 1H), 3.15 (t, *J* = 12.8 Hz, 1H), 2.49 (s, 3H), 2.47 – 2.41 (m, 1H), 2.39 (s, 3H), 2.34 – 2.29 (m, 2H), 2.04 – 1.90 (m, 1H), 1.36 – 1.33 (m, 2H), 0.92 (d, *J* = 13.6 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.5, 144.8, 143.8, 137.7, 136.4, 136.3, 132.3, 131.7, 130.0, 129.8, 129.5, 128.8, 127.6, 127.3, 126.1, 109.1, 70.6, 64.5, 53.2, 48.8, 47.2, 37.8, 34.6, 23.7, 23.3, 21.6, 21.6. **HRMS** (ESI) calcd for $C_{31}H_{32}O_4NCl2INaS_2$ $[M+Na]^+$ 766.0194; found: 766.0192.

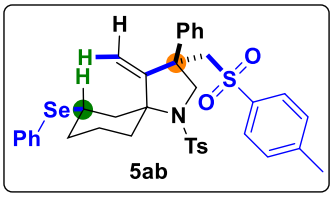
(3S*,5R*,7S*)-4-methylene-3-phenyl-7-(phenylselanyl)-3-((phenylsulfonyl)methyl)-1-tosyl-1-azaspiro[4.5]decane (5aa): The title compound was prepared according to GP-4 and purified by column



chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (49 mg, yield = 70%; >20:1 dr); Mp. 160-161 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.3 Hz, 2H), 7.50 (dd, *J* = 7.8, 1.7 Hz, 2H), 7.43 (d, *J* = 8.5 Hz, 3H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.30 – 7.23 (m, 5H), 7.06 – 7.02 (m, 3H), 7.00 – 6.95 (m, 2H), 5.42 (s, 1H), 5.36 (d, J = 1.6 Hz, 1H), 5.05 (d, J = 1.8 Hz, 1H), 4.73 (d, J = 11.0 Hz, 1H), 3.95 (d, J = 14.7 Hz, 1H), 3.53 – 3.46 (m, 1H), 3.39 (d, *J* = 11.0 Hz, 1H), 3.34 (d, *J* = 14.6 Hz, 1H), 2.74 (t, *J* = 13.0 Hz, 1H), 2.49 (s, 3H), 2.29 (td, *J* = 13.4, 4.3 Hz, 1H), 2.01 (d, *J* = 11.3 Hz, 2H), 1.53 – 1.39 (m, 2H), 1.33 – 1.22 (m, 1H), 0.77 (d, *J* = 13.3 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.3, 143.2, 140.1, 137.3, 137.2, 135.2, 133.1,

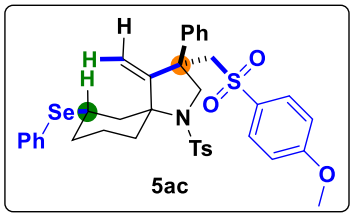
129.6, 128.9, 128.9, 128.3, 127.7, 127.7, 127.5, 127.2, 126.5, 108.5, 70.5, 65.3, 53.2, 49.5, 42.0, 38.6, 34.9, 32.2, 22.7, 21.6.

(3S*,5R*,7S*)-4-methylene-3-phenyl-7-(phenylselanyl)-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (5ab): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel

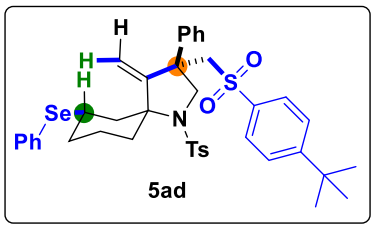


(ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (52 mg, yield = 72%; 20:1 dr); Mp. 145-146 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.3 Hz, 2H), 7.50 (dd, *J* = 7.8, 1.7 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.29 – 7.23 (m, 3H), 7.08 – 6.96 (m, 7H), 5.36 (d, *J* = 1.4 Hz, 1H), 5.04 (d, *J* = 1.6 Hz, 1H), 4.69 (d, *J* = 10.9 Hz, 1H), 3.95 – 3.88 (m, 1H), 3.53 – 3.46 (m, 1H), 3.39 (d, *J* = 11.0 Hz, 1H), 3.32 (d, *J* = 14.6 Hz, 1H), 2.73 (t, *J* = 13.0 Hz, 1H), 2.49 (s, 3H), 2.34 (s, 3H), 2.27 (dd, *J* = 13.4, 4.2 Hz, 1H), 2.01 (d, *J* = 12.7 Hz, 2H), 1.52 – 1.41 (m, 2H), 1.33 – 1.23 (m, 1H), 0.77 (d, *J* = 13.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.4, 144.1, 143.2, 137.4, 137.3, 137.1, 135.1, 129.6, 129.5, 128.9, 128.2, 127.7, 127.6, 127.0, 126.5, 108.5, 70.5, 65.3, 53.2, 49.4, 42.0, 38.6, 34.9, 32.2, 22.7, 21.6, 21.5. **HRMS** (ESI) calcd for $C_{37}H_{39}O_4NNaS_2Se$ $[M+Na]^+$ 728.1377; found: 728.1373.

(3S*,5R*,7S*)-3-(((4-methoxyphenyl)sulfonyl)methyl)-4-methylene-3-phenyl-7-(phenylselanyl)-1-tosyl-1-azaspiro[4.5]decane (5ac): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 20:80) to afford the product as an off-white solid (50 mg, yield = 68%; 20:1 dr); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.3 Hz, 2H), 7.50 (dd, *J* = 7.8, 1.7 Hz, 2H), 7.35 (d, *J* = 3.1 Hz, 2H), 7.33 (d, *J* = 2.3 Hz, 2H), 7.30 – 7.26 (m, 2H), 7.26 – 7.23 (m, 1H), 7.08 – 6.98 (m, 5H), 6.71 (d, *J* = 8.9 Hz, 2H), 5.35 (d, *J* = 1.4 Hz, 1H), 5.04 (d, *J* = 1.6 Hz, 1H), 4.68 (d, *J* = 11.0 Hz, 1H), 3.92 (d, *J* = 14.6 Hz, 1H), 3.81 (s, 3H), 3.50 (tt, *J* = 12.6, 4.1 Hz, 1H), 3.37 (d, *J* = 11.0 Hz, 1H), 3.32 (d, *J* = 14.6 Hz, 1H), 2.73 (t, *J* = 13.0 Hz, 1H), 2.48 (s, 3H), 2.29 (td, *J* = 13.3, 4.2 Hz, 1H), 2.04 – 1.97 (m, 2H), 1.49 – 1.39 (m, 2H), 1.29 – 1.25 (m, 1H), 0.76 (d, J = 13.3 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 163.2, 159.5, 143.2, 137.5, 137.2, 135.1, 131.6, 129.7, 129.6, 128.9, 128.2, 127.7, 127.7, 127.1, 126.5, 114.1, 108.5, 70.5, 65.4, 55.6, 53.2, 49.4, 41.9, 38.6, 34.9, 32.2, 22.7, 21.6. **HRMS** (ESI) calcd for $C_{37}H_{39}O_5NNaS_2Se$ $[M+Na]^+$ 744.1327; found: 744.1322.

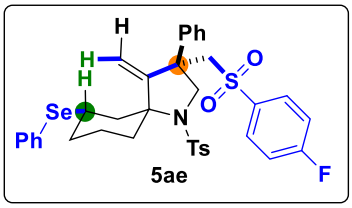


(3S*,5R*,7S*)-3-(((4-(tert-butyl)phenyl)sulfonyl)methyl)-4-methylene-3-phenyl-7-(phenylselanyl)-1-tosyl-1-azaspiro[4.5]decane (5ad): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 16:84) to afford the product as an off-white solid (50 mg, yield = 66%; >20:1 dr); Mp. 100-101 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.3 Hz, 2H), 7.49 (dd, *J* = 7.8, 1.7 Hz, 2H), 7.38 – 7.29 (m, 4H), 7.29 – 7.19 (m, 5H), 7.04 – 6.96 (m, 3H), 6.96 – 6.88 (m, 2H), 5.34 (d, *J* = 1.7 Hz, 1H), 5.05 (d, *J* = 1.8 Hz, 1H), 4.75 (d, *J* = 11.0 Hz, 1H), 3.96 (d, *J* = 14.7 Hz, 1H), 3.51 (tt, *J* = 12.8, 4.2 Hz, 1H), 3.40 (d, *J* = 11.0 Hz, 1H), 3.32 (d, *J* = 14.6 Hz, 1H), 2.73 (t, *J* = 13.0 Hz, 1H), 2.48 (s, 3H), 2.29 (td, *J* = 13.3, 4.2 Hz, 1H), 2.04 – 1.95 (m, 2H), 1.52 – 1.39 (m, 3H), 1.28 (s, 9H), 0.74 (d, *J* = 13.4 Hz, 1H).

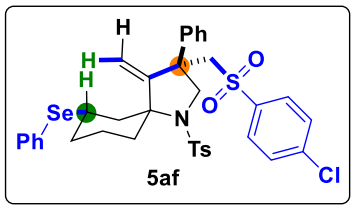


^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6, 156.8, 143.2, 137.3, 137.2, 136.8, 135.1, 129.6, 128.9, 128.2, 128.1, 127.7, 127.3, 127.1, 126.5, 125.8, 108.4, 70.5, 65.2, 53.2, 49.3, 42.0, 38.6, 35.0, 34.9, 32.2, 30.9, 22.7, 21.5. **HRMS** (ESI) calcd for $C_{40}H_{45}O_4NNaS_2Se$ $[M+Na]^+$ 770.1847; found: 684.1845.

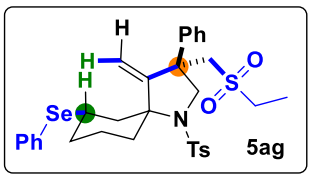
(3S*,5R*,7S*)-3-(((4-fluorophenyl)sulfonyl)methyl)-4-methylene-3-phenyl-7-(phenylselanyl)-1-tosyl-1-azaspiro[4.5]decane (5ae): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (51 mg, yield = 71%; 15.8:1 dr); Mp. 148-149 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.3 Hz, 2H), 7.54 – 7.46 (m, 2H), 7.42 – 7.33 (m, 4H), 7.32 – 7.22 (m, 3H), 7.10 – 6.96 (m, 5H), 6.89 (t, *J* = 8.5 Hz, 2H), 5.36 (d, *J* = 1.3 Hz, 1H), 5.05 (d, *J* = 1.4 Hz, 1H), 4.75 (d, *J* = 10.9 Hz, 1H), 3.96 (d, *J* = 14.8 Hz, 1H), 3.50 (tt, *J* = 13.0, 4.2 Hz, 1H), 3.37 (d, *J* = 4.4 Hz, 1H), 3.34 (d, *J* = 8.1 Hz, 1H), 2.73 (t, *J* = 13.0 Hz, 1H), 2.49 (s, 3H), 2.29 (td, *J* = 13.4, 4.1 Hz, 1H), 1.99 (d, *J* = 8.3 Hz, 2H), 1.53 – 1.39 (m, 2H), 1.32 – 1.20 (m, 1H), 0.74 (d, *J* = 13.0 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.48, 163.93 (d, *J* = 250 Hz), 159.38 (d, *J* = 20 Hz), 143.28, 137.20, 137.11, 135.17, 130.44, 130.34, 129.60, 128.93, 128.34, 127.75, 127.69, 127.28, 126.55, 116.17 (d, *J* = 22 Hz), 115.94 (d, *J* = 22 Hz), 108.45, 70.6, 65.5, 53.2, 49.3, 42.0, 38.6, 35.0, 32.2, 22.7, 21.6. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -104.0. **HRMS** (ESI) calcd for $C_{36}H_{36}O_4NNaFS_2Se$ $[M+Na]^+$ 732.1240; found: 732.1238.



(3S*,5R*,7S*)-3-(((4-chlorophenyl)sulfonyl)methyl)-4-methylene-3-phenyl-7-(phenylselanyl)-1-tosyl-1-azaspiro[4.5]decane (5af): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 1:4) to afford the product as an off-white solid (58 mg, yield = 78%; >20:1 dr); Mp. 150-151 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.1 Hz, 2H), 7.32 – 7.27 (m, 3H), 7.26 – 7.21 (m, 2H), 7.19 (d, *J* = 8.6 Hz, 2H), 7.11 – 7.06 (m, 1H), 7.05 – 6.97 (m, 4H), 5.34 (d, *J* = 1.4 Hz, 1H), 5.06 (d, *J* = 1.6 Hz, 1H), 4.74 (d, *J* = 10.9 Hz, 1H), 3.97 (d, *J* = 14.8 Hz, 1H), 3.44 – 3.36 (m, 2H), 3.34 (d, *J* = 5.2 Hz, 1H), 2.57 (t, *J* = 12.8 Hz, 1H), 2.48 (s, 3H), 2.28 (td, *J* = 13.3, 4.2 Hz, 1H), 1.98 – 1.90 (m, 2H), 1.49 – 1.41 (m, 1H), 1.36 – 1.22 (m, 2H), 0.73 (d, *J* = 13.4 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.5, 143.3, 139.8, 138.3, 137.2, 137.1, 133.8, 132.7, 129.6, 129.1, 129.0, 128.8, 128.4, 127.7, 127.3, 127.3, 126.6, 108.5, 70.4, 65.4, 53.2, 49.2, 43.6, 41.1, 35.1, 31.4, 21.9, 21.6. **HRMS** (ESI) calcd for $C_{36}H_{36}O_4NClNaS_2Se$ $[M+Na]^+$ 478.0831; found: 748.0825.

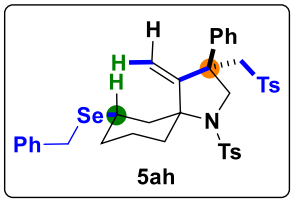


(3S*,5R*,7S*)-3-((ethylsulfonyl)methyl)-4-methylene-3-phenyl-7-(phenylselanyl)-1-tosyl-1-azaspiro[4.5]decane (5ag): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 15:85) to afford the product as an off-white solid (39 mg, yield = 60%; >20:1 dr); Mp. 160-161 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.3 Hz, 2H), 7.51 (dd, *J* = 7.8, 1.7 Hz, 2H), 7.35 – 7.22 (m, 10H), 5.46 (d, *J* = 1.7 Hz, 1H), 5.14 (d, *J* = 1.8 Hz, 1H), 4.76 (d, *J* = 11.1 Hz, 1H), 3.74 (d, *J* = 14.9 Hz, 1H), 3.55 (tt, *J* = 12.7, 4.3 Hz, 1H), 3.28 (d, *J* = 11.2 Hz, 1H), 3.03 (d, *J* = 14.8 Hz, 1H), 2.76 (t, *J* =

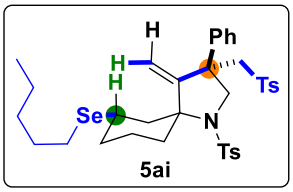


13.0 Hz, 1H), 2.47 (s, 3H), 2.31 (td, *J* = 13.4, 4.1 Hz, 1H), 2.05 (d, *J* = 7.6 Hz, 1H), 2.02 – 1.99 (m, 1H), 1.95 – 1.86 (m, 1H), 1.53 – 1.42 (m, 2H), 1.38 – 1.31 (m, 1H), 1.27 – 1.14 (m, 1H), 1.00 (t, *J* = 7.4 Hz, 3H), 0.81 (d, *J* = 13.5 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.2, 143.2, 137.6, 137.2, 135.2, 129.6, 128.9, 128.8, 128.2, 127.9, 127.8, 127.7, 126.6, 108.7, 70.6, 61.7, 52.8, 49.3, 48.7, 42.1, 38.7, 34.9, 32.2, 22.7, 21.5, 6.3. **HRMS** (ESI) calcd for $C_{32}H_{37}O_4NNaS_2Se$ $[M+Na]^+$ 666.1221; found: 666.1215.

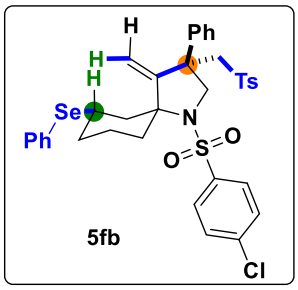
(3S*,5R*,7S*)-7-(benzylselanyl)-4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (5ah): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 15:85) to afford the product as an off-white solid (45 mg, yield = 62%; 16.4:1 dr); Mp. 100-101 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.3 Hz, 2H), 7.37 – 7.30 (m, 4H), 7.25 – 7.15 (m, 5H), 7.09 – 6.94 (m, 7H), 5.16 (d, *J* = 1.6 Hz, 1H), 4.99 (d, *J* = 1.8 Hz, 1H), 4.68 (d, *J* = 11.0 Hz, 1H), 3.92 (d, *J* = 14.6 Hz, 1H), 3.83 – 3.71 (m, 2H), 3.40 (d, *J* = 11.0 Hz, 1H), 3.32 (d, *J* = 14.6 Hz, 1H), 3.04 (tt, *J* = 12.7, 4.1 Hz, 1H), 2.69 (t, *J* = 12.9 Hz, 1H), 2.48 (s, 3H), 2.34 (s, 3H), 2.28 (dd, *J* = 13.4, 4.1 Hz, 1H), 1.93 (t, *J* = 12.7 Hz, 2H), 1.51 – 1.38 (m, 2H), 1.24 (d, *J* = 14.7 Hz, 1H), 0.76 (d, *J* = 13.3 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.2, 144.1, 143.2, 139.2, 137.4, 137.3, 137.2, 129.5, 129.5, 128.9, 128.4, 128.2, 127.7, 127.5, 126.9, 126.6, 126.5, 108.5, 70.4, 65.3, 53.2, 49.4, 42.1, 34.8, 33.7, 32.2, 25.9, 22.8, 21.5, 21.5. **HRMS** (ESI) calcd for $C_{38}H_{41}O_4NNaS_2Se$ $[M+Na]^+$ 742.1534; found: 742.1530.



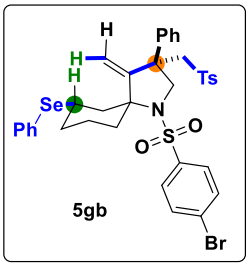
(3S*,5R*,7S*)-4-methylene-7-(pentylselanyl)-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (5ai): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 14:86) to afford the product as an off-white solid (36 mg, yield = 50%; 20:1 dr); Mp. 65-66 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.3 Hz, 2H), 7.37 – 7.29 (m, 4H), 7.10 – 7.02 (m, 5H), 7.02 – 6.96 (m, 2H), 5.40 (d, *J* = 1.6 Hz, 1H), 5.08 (d, *J* = 1.7 Hz, 1H), 4.71 (d, *J* = 11.0 Hz, 1H), 3.95 (d, *J* = 14.6 Hz, 1H), 3.45 (d, *J* = 11.0 Hz, 1H), 3.36 (d, *J* = 14.6 Hz, 1H), 3.19 (tt, *J* = 12.8, 4.3 Hz, 1H), 2.70 (t, *J* = 13.0 Hz, 1H), 2.58 – 2.50 (m, 2H), 2.47 (s, 3H), 2.35 (s, 3H), 2.30 (dd, *J* = 13.3, 4.2 Hz, 1H), 2.05 – 1.96 (m, 2H), 1.70 – 1.57 (m, 3H), 1.51 – 1.42 (m, 2H), 1.37 – 1.30 (m, 4H), 0.89 (t, *J* = 7.2 Hz, 3H), 0.79 (d, *J* = 13.1 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.5, 144.1, 143.2, 137.5, 137.3, 137.2, 129.5, 129.5, 128.2, 127.7, 127.5, 126.9, 126.5, 108.5, 70.4, 65.3, 53.2, 49.5, 42.4, 34.8, 33.4, 32.5, 32.2, 30.5, 22.8, 22.2, 22.1, 21.5, 21.5, 13.9. **HRMS** (ESI) calcd for $C_{36}H_{45}O_4NNaS_2Se$ $[M+Na]^+$ 722.1847; found: 722.1842.



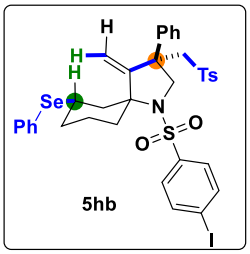
(3S*,5R*,7S*)-1-((4-chlorophenyl)sulfonyl)-4-methylene-3-phenyl-7-(phenylselanyl)-3-(tosylmethyl)-1-azaspiro[4.5]decane (5fb): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 18:82) to afford the product as an off-white solid (55 mg, yield = 75%; 15.9:1 dr); Mp. 87-88 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.7 Hz, 2H), 7.49 (dd, *J* = 9.0, 1.8 Hz, 4H), 7.32 (d, *J* = 8.3 Hz, 2H), 7.30 – 7.24 (m, 3H), 7.11 – 6.97 (m, 7H), 5.38 (d, *J* = 1.5 Hz, 1H), 5.06 (d, *J* = 1.7 Hz, 1H), 4.72 (d, *J* = 11.0 Hz, 1H), 3.90 (d, *J* = 14.6 Hz, 1H), 3.55 – 3.48 (m, 1H), 3.44 (d, *J* = 11.0 Hz, 1H), 3.32 (d, *J* = 14.6 Hz, 1H), 2.66 (t, *J* = 12.9 Hz, 1H), 2.34 (s, 3H), 2.25 (td, *J* = 13.4, 4.2 Hz, 1H), 2.01 (d, *J* = 7.7 Hz, 2H), 1.48 – 1.43 (m, 1H), 1.36 – 1.23 (m, 2H), 0.79 (d, *J* = 12.9 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.7, 144.2, 138.9, 138.7, 137.3, 137.1, 135.1, 129.5, 129.2, 129.0, 129.0, 128.3, 128.0, 127.8, 127.5, 127.1, 126.4, 108.9, 70.8, 65.2, 53.2, 49.6, 42.2, 38.3, 34.8, 32.1, 22.6, 21.5. **HRMS** (ESI) calcd for $C_{36}H_{36}O_4NClNaS_2Se$ $[M+Na]^+$ 748.0831; found: 748.0825.



(3S*,5R*,7S*)-1-((4-bromophenyl)sulfonyl)-4-methylene-3-phenyl-7-(phenylselanyl)-3-(tosylmethyl)-1-azaspiro[4.5]decane (5gb): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 18:82) to afford the product as an off-white solid (59 mg, yield = 73%; 20:1 dr); Mp. 103-104 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.71 – 7.63 (m, 4H), 7.49 (dd, *J* = 7.4, 2.0 Hz, 2H), 7.32 (d, J = 8.0 Hz, 2H), 7.30 – 7.24 (m, 3H), 7.12 – 6.97 (m, 7H), 5.38 (d, *J* = 1.6 Hz, 1H), 5.05 (d, *J* = 1.8 Hz, 1H), 4.71 (d, *J* = 11.0 Hz, 1H), 3.90 (d, *J* = 14.6 Hz, 1H), 3.55 – 3.48 (m, 1H), 3.44 (d, *J* = 11.0 Hz, 1H), 3.32 (d, *J* = 14.6 Hz, 1H), 2.66 (t, *J* = 12.9 Hz, 1H), 2.34 (s, 3H), 2.25 (td, *J* = 13.3, 4.1 Hz, 1H), 2.01 (t, *J* = 8.9 Hz, 2H), 1.48 – 1.43 (m, 1H), 1.36 – 1.24 (m, 2H), 0.78 (d, *J* = 12.9 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.7, 144.2, 139.2, 137.3, 137.1, 135.1, 132.2, 129.5, 129.1, 129.0, 128.3, 128.0, 127.8, 127.5, 127.4, 127.1, 126.4, 110.0, 108.9, 70.8, 65.2, 53.2, 49.6, 42.3, 38.3, 34.8, 32.1, 22.6, 21.5. **HRMS** (ESI) calcd for $C_{36}H_{37}O_4NBrS_2Se$ $[M+H]^+$ 770.0507; found: 770.0503.

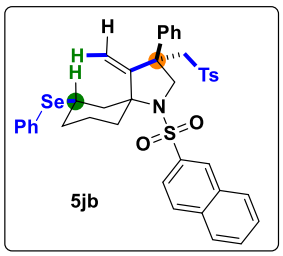


(3S*,5R*,7S*)-1-((4-iodophenyl)sulfonyl)-4-methylene-3-phenyl-7-(phenylselanyl)-3-(tosylmethyl)-1-azaspiro[4.5]decane (5hb): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (63 mg, yield = 76%; >20:1 dr); Mp. 92-93 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.6 Hz, 2H), 7.53 (d, *J* = 7.9 Hz, 2H), 7.51 – 7.46 (m, 2H), 7.34 – 7.25 (m, 5H), 7.13 – 6.96 (m, 7H), 5.37 (d, *J* = 1.6 Hz, 1H), 5.05 (d, *J* = 1.8 Hz, 1H), 4.71 (d, *J* = 11.0 Hz, 1H), 3.90 (d, *J* = 14.6 Hz, 1H), 3.55 – 3.48 (m, 1H), 3.43 (d, *J* = 11.0 Hz, 1H), 3.32 (d, *J* = 14.6 Hz, 1H), 2.66 (t, *J* = 12.9 Hz, 1H), 2.34 (s, 3H), 2.24 (td, *J* = 13.3, 4.2 Hz, 1H), 2.04 – 1.98 (m, 2H), 1.48 – 1.40 (m, 2H), 1.36 – 1.29 (m, 1H), 1.28 – 1.21 (m, 1H), 0.77 (d, *J* = 13.4 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.8, 144.2, 139.9, 138.2, 137.3, 137.1, 135.1, 129.5, 129.0, 128.9, 128.3, 128.0, 127.8, 127.5, 127.1, 126.4,

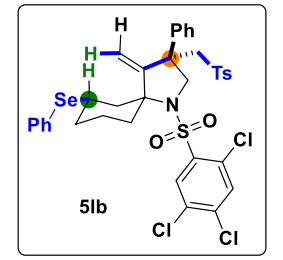


108.9, 99.8, 70.8, 65.2, 53.2, 49.6, 42.3, 38.4, 34.8, 32.0, 22.6, 21.5. **HRMS** (ESI) calcd for $C_{36}H_{36}O_4NINaS_2Se$ $[M+Na]^+$ 840.0187; found: 840.0186.

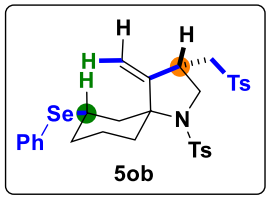
(3S*,5R*,7S*)-4-methylene-1-(naphthalen-2-ylsulfonyl)-3-phenyl-7-(phenylselanyl)-3-(tosylmethyl)-1-azaspiro[4.5]decane (5jb): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 25:75) to afford the product as an off-white solid (52 mg, yield = 69%; 20:1 dr); Mp. 98-99 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 8.49 (d, *J* = 1.6 Hz, 1H), 8.05 (d, *J* = 7.8 Hz, 1H), 7.96 (d, *J* = 8.7 Hz, 2H), 7.79 (dd, *J* = 8.7, 1.9 Hz, 1H), 7.72 – 7.63 (m, 2H), 7.40 (dd, *J* = 8.2, 1.3 Hz, 2H), 7.28 (d, *J* = 12.0 Hz, 2H), 7.24 – 7.19 (m, 1H), 7.17 – 7.11 (m, 2H), 7.04 – 6.95 (m, 5H), 6.91 – 6.87 (m, 2H), 5.35 (d, *J* = 1.6 Hz, 1H), 5.03 (d, *J* = 1.8 Hz, 1H), 4.76 (d, *J* = 11.0 Hz, 1H), 3.88 (d, *J* = 14.6 Hz, 1H), 3.56 – 3.46 (m, 2H), 3.31 (d, *J* = 14.6 Hz, 1H), 2.82 (t, *J* = 13.0 Hz, 1H), 2.37 (td, *J* = 13.4, 4.2 Hz, 1H), 2.31 (s, 3H), 2.03 (d, *J* = 12.5 Hz, 2H), 1.52 – 1.43 (m, 2H), 1.36 – 1.32 (m, 1H), 0.83 (d, *J* = 13.2 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.1, 144.1, 137.4, 137.1, 137.1, 135.1, 134.7, 132.1, 129.5, 129.4, 129.1, 128.9, 128.8, 128.2, 128.1, 127.8, 127.7, 127.5, 127.0, 126.5, 123.0, 108.7, 70.7, 65.2, 53.2, 49.5, 42.3, 38.6, 34.8, 32.1, 22.7, 21.5. **HRMS** (ESI) calcd for $C_{40}H_{39}O_4NNaS_2Se$ $[M+Na]^+$ 764.1377; found: 764.1375.



(3S*,5R*,7S*)-4-methylene-3-phenyl-7-(phenylselanyl)-3-(tosylmethyl)-1-((2,4,5-trichlorophenyl)sulfonyl)-1-azaspiro[4.5]decane (5lb): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 26:74) to afford the product as an off-white solid (45 mg, yield = 56%; 14.3:1 dr); Mp. 114-115 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 8.21 (s, 1H), 7.61 (s, 1H), 7.51 (dd, *J* = 7.5, 1.9 Hz, 2H), 7.38 (d, *J* = 8.3 Hz, 2H), 7.30 – 7.24 (m, 3H), 7.14 – 7.01 (m, 7H), 5.48 (d, *J* = 1.6 Hz, 1H), 5.07 (d, *J* = 1.7 Hz, 1H), 4.41 (d, *J* = 11.0 Hz, 1H), 3.80 (d, *J* = 2.9 Hz, 1H), 3.77 (s, 1H), 3.59 – 3.47 (m, 1H), 3.43 (dd, *J* = 14.6, 4.7 Hz, 1H), 2.71 – 2.60 (m, 1H), 2.41 – 2.32 (m, 4H), 2.30 – 2.23 (m, 1H), 2.04 (s, 1H), 1.56 – 1.50 (m, 1H), 1.48 – 1.39 (m, 2H), 1.16 (d, *J* = 13.3 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) 158.1, 144.4, 138.1, 137.5, 137.4, 137.1, 135.3, 133.3, 133.1, 131.7, 131.5, 129.6, 129.0, 128.3, 127.8, 127.5, 127.3, 126.4, 110.2, 72.0, 64.5, 53.5, 50.3, 42.6, 37.8, 34.1, 32.1, 22.6, 21.5. **HRMS** (ESI) calcd for $C_{36}H_{34}O_4NCl_3NaS_2Se$ $[M+Na]^+$ 816.0052; found: 816.0045.

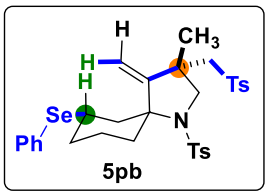


(3S*,5R*,7S*)-4-methylene-7-(phenylselanyl)-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (5ob): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 15:85) to afford the product as an off-white solid (30 mg, yield = 47%; 3.3:1 dr); Mp. 69-70 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.72 – 7.62 (m, 4H), 7.50 – 7.47 (m, 2H), 7.39 – 7.33 (m, 2H), 7.30 – 7.25 (m, 3H), 7.23 – 7.18 (m, 2H), 5.22 (s, 1H), 4.86 (s, 1H), 3.70 – 3.63 (m, 1H), 3.43 – 3.36 (m, 1H), 3.20 – 3.08 (m, 2H), 3.07 – 3.01 (m, 1H), 2.98 – 2.78 (m, 2H), 2.75 – 2.58 (m, 1H), 2.48 (s, 3H), 2.45 (s, 3H), 2.05 (t, *J* = 9.4 Hz, 1H), 1.89 – 1.82 (m, 1H), 1.68 (d, *J* = 14.0 Hz, 1H), 1.62 – 1.57 (m, 1H), 1.53 (d, *J* = 8.4 Hz, 1H), 0.93 – 0.86 (s, 1H). **^{13}C NMR** (100 MHz,



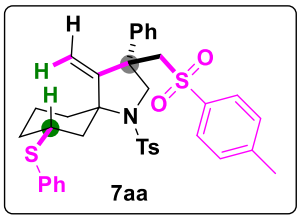
$CDCl_3$) δ 154.8, 145.1, 143.1, 138.1, 136.2, 135.1, 134.9, 130.1, 130.0, 129.5, 128.9, 128.9, 128.2, 127.9, 127.6, 127.6, 127.2, 109.0, 58.8, 50.7, 41.9, 38.3, 36.9, 34.4, 32.2, 23.2, 21.7, 21.5. **HRMS** (ESI) calcd for $C_{31}H_{35}O_4NNaS_2Se$ $[M+Na]^+$ 652.1064; found: 652.1060.

(3S*,5R*,7S*)-3-methyl-4-methylene-7-(phenylselanyl)-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane



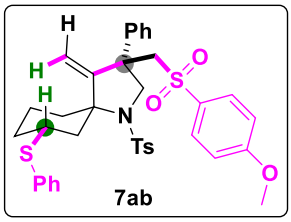
(5pb): The title compound was prepared according to GP-4 and purified by column chromatography on silica gel (ethyl acetate/hexane = 14:86) to afford the product as an off-white solid (33 mg, yield = 50%; 1.5:1 dr); Mp. 70-71 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.74 (dd, *J* = 8.3, 1.8 Hz, 3H), 7.58 (d, *J* = 8.3 Hz, 2H), 7.55 – 7.46 (m, 4H), 7.34 – 7.29 (m, 6H), 7.28 – 7.24 (m, 3H), 7.21 – 7.17 (m, 2H), 5.33 (s, 1H), 4.95 (d, *J* = 1.5 Hz, 1H), 4.94 (d, *J* = 1.5 Hz, 1H), 3.88 (d, *J* = 7.7 Hz, 1H), 3.86 (d, *J* = 7.7 Hz, 1H), 3.44 (qdd, *J* = 12.5, 8.9, 5.4 Hz, 2H), 3.21 (d, *J* = 10.6 Hz, 1H), 3.16 (d, *J* = 10.6 Hz, 1H), 3.00 (s, 2H), 2.89 (d, *J* = 4.2 Hz, 1H), 2.87 – 2.52 (m, 4H), 2.47 (s, 3H), 2.46 (s, 3H), 2.44 (s, 3H), 2.14 – 2.04 (m, 3H), 1.70 – 1.52 (m, 7H), 1.43 (s, 2H), 1.37 (s, 3H), 1.26 (s, 1H), 0.95 – 0.81 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 161.8, 161.4, 144.7, 144.7, 143.2, 143.1, 138.2, 138.1, 138.0, 135.0, 134.9, 129.8, 129.5, 128.9, 128.8, 128.3, 127.6, 127.6, 127.4, 109.1, 109.0, 70.4, 70.3, 63.4, 63.2, 55.6, 55.3, 44.3, 44.2, 42.1, 37.5, 37.4, 37.0, 33.8, 32.3, 32.3, 23.7, 23.6, 22.6, 22.6, 21.6, 21.6, 21.6. **HRMS** (ESI) calcd for $C_{32}H_{37}O_4NNaS_2Se$ $[M+Na]^+$ 666.1221; found: 666.1217.

(3R*,5S*,7R*)-4-methylene-3-phenyl-7-(phenylthio)-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (7aa):



The title compound was prepared according to GP-5 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (48 mg, yield = 71%; 18.2:1 dr); Mp. 103-104 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.3 Hz, 2H), 7.37 – 7.21 (m, 9H), 7.10 – 7.03 (m, 5H), 7.02 – 6.97 (m, 2H), 5.34 (d, *J* = 1.6 Hz, 1H), 5.06 (d, *J* = 1.8 Hz, 1H), 4.71 (d, *J* = 10.9 Hz, 1H), 3.93 (d, *J* = 14.6 Hz, 1H), 3.46 – 3.28 (m, 3H), 2.59 (d, *J* = 12.9 Hz, 1H), 2.48 (s, 3H), 2.34 (s, 3H), 2.28 (dt, *J* = 13.2, 6.6 Hz, 1H), 2.02 – 1.89 (m, 2H), 1.46 (d, *J* = 6.3 Hz, 1H), 1.39 – 1.21 (m, 2H), 0.76 (d, *J* = 13.1 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.5, 144.1, 143.2, 137.4, 137.2, 137.1, 133.9, 133.4, 132.6, 129.5, 129.5, 128.8, 128.2, 127.7, 127.5, 127.2, 127.0, 126.5, 108.6, 70.3, 65.3, 53.2, 49.4, 43.5, 41.2, 35.0, 31.4, 21.9, 21.5, 21.5. **HRMS** (ESI) calcd for $C_{37}H_{39}O_4NNaS_3$ $[M+Na]^+$ 680.1933; found: 680.1929.

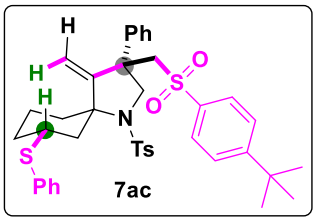
(3R*,5S*,7R*)-3-(((4-methoxyphenyl)sulfonyl)methyl)-4-methylene-3-phenyl-7-(phenylthio)-1-tosyl-1-azaspiro[4.5]decane (7ab):



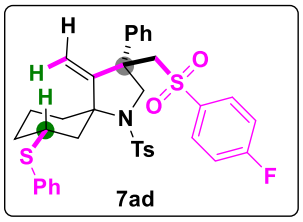
The title compound was prepared according to GP-5 and purified by column chromatography on silica gel (ethyl acetate/hexane = 20:80) to afford the product as an off-white solid (43 mg, yield = 63%; >20:1 dr); Mp. 102-103 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.3 Hz, 2H), 7.38 – 7.31 (m, 6H), 7.29 – 7.26 (m, 1H), 7.26 – 7.21 (m, 2H), 7.09 – 6.99 (m, 5H), 6.71 (d, *J* = 8.9 Hz, 2H), 5.33 (d, *J* = 1.5 Hz, 1H), 5.06 (d, *J* = 1.7 Hz, 1H), 4.70 (d, *J* = 10.9 Hz, 1H), 3.92 (d, *J* = 14.6 Hz, 1H), 3.81 (s, 3H), 3.45 – 3.30 (m, 3H), 2.56 (t, *J* = 12.8 Hz, 1H), 2.48 (s, 3H), 2.28 (td, *J* = 13.2, 4.2 Hz, 1H), 2.00 – 1.89 (m, 2H), 1.49 –

1.41 (m, 1H), 1.39 – 1.25 (m, 2H), 0.75 (d, *J* = 13.6 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 163.2, 159.6, 143.2, 137.5, 137.2, 133.9, 132.6, 131.6, 129.7, 129.6, 128.8, 128.3, 127.7, 127.2, 127.1, 126.5, 114.1, 108.5, 70.3, 65.4, 55.6, 53.2, 49.4, 43.5, 41.2, 35.0, 31.5, 22.0, 21.5. **HRMS** (ESI) calcd for $C_{37}H_{39}O_5NNaS_3$ $[M+Na]^+$ 696.1882; found: 696.1878.

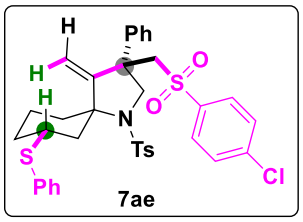
(3R*,5S*,7R*)-3-(((4-(tert-butyl)phenyl)sulfonyl)methyl)-4-methylene-3-phenyl-7-(phenylthio)-1-tosyl-1-azaspiro[4.5]decane (7ac): The title compound was prepared according to GP-5 and purified by column chromatography on silica gel (ethyl acetate/hexane = 18:82) to afford the product as an off-white solid (48 mg, yield = 68%; >20:1 dr); Mp. 116-117 °C; **^{1}H NMR** (400 MHz, $CDCl_3$)) δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.38 – 7.30 (m, 6H), 7.29 – 7.21 (m, 5H), 7.04 – 6.91 (m, 5H), 5.33 (d, *J* = 1.7 Hz, 1H), 5.07 (d, *J* = 1.8 Hz, 1H), 4.77 (d, *J* = 11.0 Hz, 1H), 3.97 (d, *J* = 14.7 Hz, 1H), 3.45 – 3.36 (m, 2H), 3.33 (d, *J* = 14.6 Hz, 1H), 2.57 (t, *J* = 12.8 Hz, 1H), 2.48 (s, 3H), 2.28 (td, *J* = 13.3, 4.3 Hz, 1H), 1.99 – 1.89 (m, 2H), 1.46 – 1.39 (m, 1H), 1.37 – 1.29 (m, 1H), 1.28 (s, 9H), 1.27 – 1.17 (m, 1H), 0.73 (d, *J* = 13.4 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.7, 156.8, 143.2, 137.2, 136.8, 133.9, 132.6, 129.6, 128.8, 128.2, 127.7, 127.3, 127.2, 127.1, 126.5, 125.8, 108.4, 70.3, 65.2, 53.2, 49.3, 43.6, 41.2, 35.0, 31.4, 30.9, 22.0, 21.6. **HRMS** (ESI) calcd for $C_{40}H_{45}O_4NNaS_3$ $[M+Na]^+$ 722.2402 found: 722.2398.



(3R*,5S*,7R*)-3-(((4-fluorophenyl)sulfonyl)methyl)-4-methylene-3-phenyl-7-(phenylthio)-1-tosyl-1-azaspiro[4.5]decane (7ad): The title compound was prepared according to GP-5 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (43 mg, yield = 64%; 20:1 dr); Mp. 172-173 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.2 Hz, 2H), 7.43 – 7.32 (m, 6H), 7.30 – 7.21 (m, 2H), 7.10 – 6.95 (m, 6H), 6.90 (td, *J* = 8.6, 1.7 Hz, 2H), 5.32 (dd, *J* = 16.2, 1.9 Hz, 1H), 5.06 (dd, *J* = 3.4, 1.9 Hz, 1H), 4.76 (dd, *J* = 11.0, 6.0 Hz, 1H), 3.96 (dd, *J* = 14.8, 5.5 Hz, 1H), 3.48 – 3.23 (m, 3H), 2.56 (td, *J* = 12.9, 7.2 Hz, 1H), 2.49 (d, *J* = 4.9 Hz, 3H), 2.37 – 2.22 (m, 1H), 2.01 – 1.86 (m, 2H), 1.45 (d, *J* = 12.8 Hz, 1H), 1.41 – 1.16 (m, 2H), 0.73 (d, *J* = 13.3 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.5 (d, *J* = 255.2 Hz), 163.9 (d, *J* = 25.3 Hz), 163.7 (d, *J* = 25.1 Hz), 161.3 (d, *J* = 8.2 Hz), 159.5 (d, *J* = 8.1 Hz), 159.4 (d, *J* = 2.6 Hz), 143.3, 137.2, 135. 7, 133.8, 132. 67, 129.6, 128.8, 128.4, 127.7, 127.7, 126.5, 116.2, 115.9, 108.5, 70.3, 65.5, 53.2, 49.3, 44.3, 43.6, 41.1, 35.1, 31.4, 21.9, 21.6. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -104.0. **HRMS** (ESI) calcd for $C_{36}H_{36}O_4NFNaS_3$ $[M+Na]^+$ 684.1682; found: 684.1678.

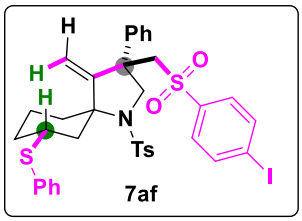


(3R*,5S*,7R*)-3-(((4-chlorophenyl)sulfonyl)methyl)-4-methylene-3-phenyl-7-(phenylthio)-1-tosyl-1-azaspiro[4.5]decane (7ae): The title compound was prepared according to GP-5 and purified by column chromatography on silica gel (ethyl acetate/hexane = 16:84) to afford the product as an off-white solid (46 mg, yield = 67%; 5.7:1 dr); Mp. 173-174 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.3 Hz, 2H), 7.37 – 7.33 (m, 3H), 7.33 – 7.22 (m, 6H), 7.19 (d, *J* = 8.7 Hz, 2H), 7.11 – 7.07 (m, 1H), 7.06 – 6.96 (m, 4H), 5.34 (d, *J* = 1.7 Hz, 1H), 5.06 (d, *J* = 1.8 Hz, 1H), 4.74 (d, *J* = 10.9 Hz, 1H), 3.97 (d, *J* = 14.8 Hz, 1H), 3.45 – 3.26

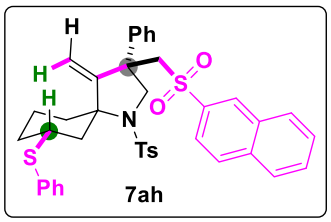


(m, 3H), 2.57 (t, *J* = 12.8 Hz, 1H), 2.49 (s, 3H), 2.28 (td, *J* = 13.2, 4.2 Hz, 1H), 2.01 – 1.87 (m, 2H), 1.50 – 1.41 (m, 1H), 1.40 – 1.20 (m, 2H), 0.73 (d, *J* = 13.6 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.5, 143.3, 139.9, 138.3, 137.2, 137.1, 133.8, 132.7, 129.6, 129.1, 129.0, 128.8, 128.5, 128.4, 127.7, 127.3, 127.3, 126.6, 108.5, 70.4, 65.5, 53.2, 49.3, 43.6, 41.1, 35.1, 31.4, 22.0, 21.6. **HRMS** (ESI) calcd for $C_{36}H_{36}O_4NClNaS_3$ $[M+Na]^+$ 700.1387; found: 700.1381.

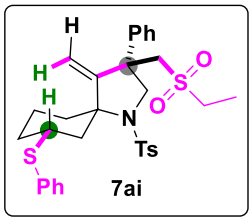
(3R*,5S*,7R*)-3-(((4-iodophenyl)sulfonyl)methyl)-4-methylene-3-phenyl-7-(phenylthio)-1-tosyl-1-azaspiro[4.5]decane (7af): The title compound was prepared according to GP-5 and purified by column chromatography on silica gel (ethyl acetate/hexane = 16:84) to afford the product as an off-white solid (49 mg, yield = 63%; >20:1 dr); Mp. 114-115 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.3 Hz, 2H), 7.57 (d, *J* = 8.6 Hz, 2H), 7.38 – 7.31 (m, 4H), 7.30 – 7.21 (m, 3H), 7.13 – 7.05 (m, 3H), 7.04 – 6.94 (m, 4H), 5.33 (d, *J* = 1.7 Hz, 1H), 5.06 (d, *J* = 1.8 Hz, 1H), 4.72 (d, *J* = 10.9 Hz, 1H), 3.97 (d, *J* = 14.8 Hz, 1H), 3.44 – 3.31 (m, 3H), 2.57 (t, *J* = 12.8 Hz, 1H), 2.48 (s, 3H), 2.28 (td, *J* = 13.3, 4.3 Hz, 1H), 1.98 – 1.89 (m, 2H), 1.48 – 1.41 (m, 1H), 1.37 – 1.29 (m, 1H), 1.27 – 1.23 (m, 1H), 0.73 (d, *J* = 13.7 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.5, 143.3, 139.4, 138.0, 137.2, 137.1, 133.8, 132.7, 129.6, 128.8, 128.4, 127.7, 127.3, 127.2, 126.5, 108.5, 101.2, 70.34, 65.4, 53.1, 49.2, 43.5, 41.1, 35.1, 31.4, 21.9, 21.6. **HRMS** (ESI) calcd for $C_{36}H_{36}O_4NINaS_3$ $[M+Na]^+$ 792.0743; found: 792.0741.



(3R*,5S*,7R*)-4-methylene-3-((naphthalen-2-ylsulfonyl)methyl)-3-phenyl-7-(phenylthio)-1-tosyl-1-azaspiro[4.5]decane (7ah): The title compound was prepared according to GP-5 and purified by column chromatography on silica gel (ethyl acetate/hexane = 26:74) to afford the product as an off-white solid (49 mg, yield = 70%; 14:1 dr); Mp. 99-100 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.91 (d, J = 1.7 Hz, 1H), 7.82 (d, *J* = 8.2 Hz, 1H), 7.75 (dt, *J* = 8.7, 6.7 Hz, 4H), 7.65 – 7.55 (m, 2H), 7.46 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.38 – 7.29 (m, 4H), 7.29 – 7.26 (m, 1H), 7.26 – 7.20 (m, 2H), 7.01 (d, *J* = 7.2 Hz, 2H), 6.85 – 6.78 (m, 2H), 6.77 – 6.72 (m, 1H), 5.33 (d, *J* = 1.7 Hz, 1H), 5.07 (d, *J* = 1.8 Hz, 1H), 4.79 (d, *J* = 10.9 Hz, 1H), 4.04 (d, *J* = 14.7 Hz, 1H), 3.44 – 3.32 (m, 3H), 2.57 (d, *J* = 11.9 Hz, 1H), 2.48 (s, 3H), 2.31 – 2.23 (m, 1H), 1.98 – 1.91 (m, 2H), 1.46 – 1.40 (m, 1H), 1.36 – 1.32 (m, 1H), 1.26 (s, 1H), 0.72 (d, *J* = 15.0 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.6, 143.2, 137.2, 137.1, 136.7, 134.9, 133.9, 132.6, 131.8, 129.8, 129.6, 129.4, 129.2, 129.2, 128.8, 128.0, 127.7, 127.4, 127.3, 127.1, 126.4, 121.9, 108.4, 70.4, 65.2, 53.2, 49.4, 43.6, 41.2, 35.0, 31.5, 22.0, 21.6. **HRMS** (ESI) calcd for $C_{40}H_{39}O_4NNaS_3$ $[M+Na]^+$ 716.1933; found: 716.1928.

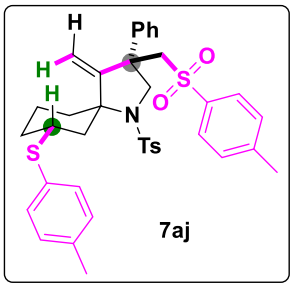


(3R*,5S*,7R*)-3-((ethylsulfonyl)methyl)-4-methylene-3-phenyl-7-(phenylthio)-1-tosyl-1-azaspiro[4.5]decane (7ai): The title compound was prepared according to GP-5 and purified by column chromatography on silica gel (ethyl acetate/hexane = 15:85) to afford the product as an off-white solid (36 mg, yield = 59%; 7.6:1 dr); Mp. 166-167 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.3 Hz, 2H), 7.42 – 7.22 (m, 12H), 5.44 (s, 1H), 5.16 (d, *J* = 1.4 Hz, 1H), 4.78 (d, *J* = 11.1 Hz, 1H), 3.75 (d, *J* = 14.9 Hz, 1H), 3.50 – 3.39 (m, 1H), 3.28 (d, *J* = 11.1

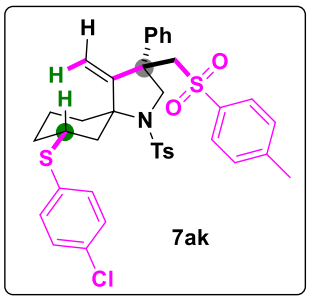


Hz, 1H), 3.04 (d, *J* = 14.7 Hz, 1H), 2.60 (t, *J* = 12.8 Hz, 1H), 2.47 (s, 3H), 2.35 – 2.26 (m, 1H), 2.07 – 1.89 (m, 4H), 1.54 – 1.46 (m, 1H), 1.42 – 1.31 (m, 2H), 1.00 (t, *J* = 7.4 Hz, 3H), 0.80 (d, *J* = 13.8 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.3, 143.2, 137.6, 137.2, 133.8, 132.7, 129.6, 128.9, 128.8, 128.0, 127.7, 127.3, 126.6, 108.7, 76.7, 70.3, 61.7, 52.8, 49.3, 48.7, 43.7, 41.2, 35.0, 31.5, 22.0, 21.5, 6.3. **HRMS** (ESI) calcd for $C_{32}H_{37}O_4NNaS_3$ $[M+Na]^+$ 618.1776; found: 618.1774.

(3R*,5S*,7R*)-4-methylene-3-phenyl-7-(p-tolylthio)-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (7aj): The title compound was prepared according to GP-5 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (48 mg, yield = 70%; 11.2:1 dr); Mp. 92-93 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.26 (d, *J* = 8.1 Hz, 3H), 7.08 (d, *J* = 7.9 Hz, 2H), 7.05 (d, *J* = 8.5 Hz, 4H), 7.00 (d, *J* = 6.6 Hz, 2H), 5.31 (d, *J* = 1.6 Hz, 1H), 5.04 (d, *J* = 1.8 Hz, 1H), 4.72 (d, *J* = 10.9 Hz, 1H), 3.93 (d, *J* = 14.6 Hz, 1H), 3.38 (d, *J* = 11.0 Hz, 1H), 3.33 (d, *J* = 14.6 Hz, 1H), 2.54 (t, *J* = 11.1 Hz, 1H), 2.49 (s, 3H), 2.34 (s, 3H), 2.33 (s, 3H), 2.30 – 2.23 (m, 1H), 1.91 (t, *J* = 14.0 Hz, 2H), 1.45 – 1.42 (m, 1H), 1.36 – 1.33 (m, 1H), 1.26 – 1.23 (m, 2H), 0.74 (d, *J* = 12.7 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.6, 144.1, 143.2, 137.5, 137.5, 137.3, 137.1, 133.4, 129.9, 129.6, 129.6, 129.5, 128.2, 127.7, 127.6, 127.0, 126.5, 108.5, 70.4, 65.4, 53.2, 49.4, 43.9, 41.2, 35.0, 31.4, 21.9, 21.6, 21.5, 21.1. **HRMS** (ESI) calcd for $C_{38}H_{41}O_4NNaS_3$ $[M+Na]^+$ 694.2089; found: 694.2085.



(3R*,5S*,7R*)-7-((4-chlorophenyl)thio)-4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (7ak): The title compound was prepared according to GP-5 and purified by column chromatography on silica gel (ethyl acetate/hexane = 18:82) to afford the product as an off-white solid (47 mg, yield = 67%; >20:1 dr); Mp. 102-103 °C; **^{1}H NMR** (399 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 1.7 Hz, 2H), 7.32 (d, *J* = 2.0 Hz, 2H), 7.30 – 7.26 (m, 2H), 7.26 – 7.21 (m, 2H), 7.10 – 6.97 (m, 7H), 5.33 (d, *J* = 1.7 Hz, 1H), 5.07 (d, *J* = 1.9 Hz, 1H), 4.68 (d, *J* = 11.0 Hz, 1H), 3.91 (d, *J* = 14.6 Hz, 1H), 3.45 – 3.31 (m, 3H), 2.58 (t, *J* = 12.9 Hz, 1H), 2.48 (s, 3H), 2.35 (s, 3H), 2.33 – 2.24 (m, 1H), 2.00 – 1.88 (m, 2H), 1.50 – 1.43 (m, 1H), 1.36 – 1.26 (m, 2H), 0.77 (d, *J* = 13.6 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.5, 144.2, 143.3, 137.5, 137.3, 137.2, 133.9, 133.4, 132.4, 129.6, 129.5, 129.0, 128.3, 127.7, 127.6, 127.0, 126.5, 108.6, 70.2, 65.2, 53.2, 49.6, 43.8, 41.2, 34.8, 31.3, 21.9, 21.6, 21.5. **HRMS** (ESI) calcd for $C_{37}H_{38}O_4NClNaS_3$ $[M+Na]^+$ 714.1543; found: 714.1541.



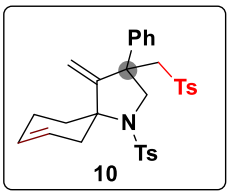
(3R*,5S*,7R*)-3-(((4-fluorophenyl)sulfonyl)methyl)-7-((4-fluorophenyl)thio)-4-methylene-3-phenyl-1-tosyl-1-azaspiro[4.5]decane (7al): The title compound was prepared according to GP-5 and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (43 mg, yield = 62%; 12.2:1 dr); Mp. 182-183 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.43 – 7.33 (m, 6H), 7.10 – 6.95 (m, 7H), 6.90 (t, J = 8.6 Hz, 2H), 5.30 (d, *J* = 1.7 Hz, 1H), 5.06 (d, *J* = 1.8 Hz, 1H), 4.75 (d, *J* = 11.0 Hz, 1H), 3.96 (d, *J* = 14.8 Hz, 1H), 3.37 (t, *J* = 13.2 Hz, 2H), 3.28 (ddd, *J* = 16.3, 8.2, 4.3 Hz, 1H), 2.60 – 2.51 (m, 1H), 2.50 (s, 3H), 2.27 (td, *J* = 13.2, 4.3 Hz, 1H), 1.97 – 1.84 (m, 2H), 1.49 – 1.39 (m, 1H), 1.36 – 1.19 (m, 2H), 0.72 (d, *J* = 13.5 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.5 (d, *J* = 256.4 Hz), 163.9 (d, *J* = 251.1 Hz), 161.3 (d, *J* = 246.9 Hz), 159.4 (d, *J* = 245.2 Hz), 143.3, 137.3, 137.1, 136.0, 135.7, 135.6, 130.4, 130.3, 129.6, 128.5, 128.3, 127.7, 127.3, 126.5, 116.2 (d, *J* = 22.5 Hz), 116.0 (d, *J* = 22.0 Hz), 115.9 (d, *J* = 21.3 Hz), 115.8 (d, *J* = 21.0 Hz), 108.5, 70.3, 65.4, 53.2, 49.3, 44.3, 41.1, 35.0, 31.3, 21.9, 21.6. ^{19}F NMR (377 MHz, $CDCl_3$) δ -104.0, -113.6. **HRMS** (ESI) calcd for $C_{36}H_{35}O_4NF_2NaS_3$ $[M+Na]^+$ 702.1588; found: 702.1583.

(1S*,3S*,6R*)-3'-methylene-4'-phenyl-1'-tosyl-4'-(tosylmethyl)-7-oxaspiro[bicyclo[4.1.0]heptane-3,2'-pyrrolidine] (8a): The title compound was prepared according to procedure-B and purified by column chromatography on silica gel (ethyl acetate/hexane = 45:55) to afford the product as an off-white solid (30 mg, yield = 52%); Mp. 213-215 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.06 (d, *J* = 8.3 Hz, 3H), 6.98 (d, *J* = 4.3 Hz, 4H), 5.15 (d, *J* = 5.9 Hz, 2H), 4.77 (d, *J* = 11.1 Hz, 1H), 3.93 (d, *J* = 14.5 Hz, 1H), 3.39 (d, *J* = 11.1 Hz, 1H), 3.34 (d, *J* = 14.5 Hz, 1H), 3.22 – 3.18 (m, 1H), 3.06 (d, *J* = 11.0 Hz, 1H), 3.03 (s, 1H), 2.48 (s, 3H), 2.34 (s, 3H), 2.27 (td, *J* = 13.1, 4.6 Hz, 1H), 1.93 (ddd, *J* = 15.0, 5.5, 2.2 Hz, 1H), 1.79 (d, *J* = 15.2 Hz, 1H), 1.48 (dd, *J* = 14.1, 4.5 Hz, 1H), 0.22 (dd, *J* = 10.8, 2.5 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 157.7, 144.2, 143.2, 137.5, 137.0, 136.3, 129.6, 129.5, 128.3, 127.5, 127.5, 127.3, 126.4, 107.9, 67.2, 65.4, 53.2, 51.8, 51.2, 49.0, 34.9, 27.7, 22.6, 21.5, 21.5. **HRMS** (ESI) calcd for $C_{31}H_{33}O_5NNaS_2$ $[M+Na]^+$ 586.1692; found: 586.1693.

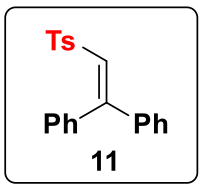
(5S*,7S*,8S*)-7,8-dibromo-4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]decane (9a): The title compound was prepared according to procedure-C and purified by column chromatography on silica gel (ethyl acetate/hexane = 22:78) to afford the product as an off-white solid (53 mg, yield = 73%); Mp. 110-113 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.2 Hz, 2H), 7.40 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.1 Hz, 2H), 7.13 (d, *J* = 8.1 Hz, 3H), 7.10 – 7.05 (m, 4H), 5.23 (d, *J* = 10.4 Hz, 2H), 5.07 (ddd, *J* = 12.5, 10.2, 4.8 Hz, 1H), 4.55 (d, *J* = 11.5 Hz, 1H), 4.15 – 4.04 (m, 1H), 3.82 (d, *J* = 14.6 Hz, 1H), 3.71 (d, *J* = 11.5 Hz, 1H), 3.42 (d, *J* = 14.6 Hz, 1H), 2.73 (dd, *J* = 15.4, 3.0 Hz, 1H), 2.60 – 2.51 (m, 1H), 2.48 (s, 3H), 2.37 (s, 3H), 2.19 (dd, *J* = 15.4, 12.6 Hz, 1H), 2.13 – 2.06 (m, 1H), 1.84 (d, J = 15.1 Hz, 1H), 1.24 – 1.14 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 160.3, 144.4, 144.0, 137.4, 137.3,

136.7, 129.9, 129.6, 128.4, 127.5, 127.5, 127.4, 126.8, 109.3, 70.5, 64.5, 55.3, 54.9, 53.8, 49.1, 46.7, 37.6, 32.8, 21.6, 21.5. **HRMS** (ESI) calcd for $C_{31}H_{33}O_4NBr_2NaS_2$ $[M+Na]^+$ 730.0089; found: 730.0084.

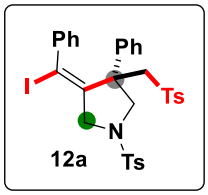
4-methylene-3-phenyl-1-tosyl-3-(tosylmethyl)-1-azaspiro[4.5]dec-7-ene (10): The title compound was prepared according to procedure-D and purified by column chromatography on silica gel (ethyl acetate/hexane = 17:83) to afford the product as an off-white solid (40 mg, yield = 70%); Mp. 90-92 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.10 – 6.95 (m, 7H), 5.66 (d, *J* = 12.1 Hz, 1H), 5.58 (s, 1H), 5.41 (s, 1H), 5.00 (s, 1H), 4.76 (d, *J* = 11.0 Hz, 1H), 3.94 (d, *J* = 14.5 Hz, 1H), 3.44 (d, *J* = 11.1 Hz, 1H), 3.36 (d, *J* = 14.5 Hz, 1H), 3.33 – 3.28 (m, 1H), 2.48 (s, 3H), 2.34 (s, 3H), 2.06 (d, *J* = 17.5 Hz, 1H), 1.88 (d, *J* = 18.0 Hz, 1H), 1.71 – 1.64 (m, 1H), 1.43 – 1.22 (m, 1H), 0.56 (dd, *J* = 12.6, 5.0 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 157.8, 144.0, 143.1, 137.5, 137.1, 137.0, 129.6, 129.4, 128.1, 127.5, 127.5, 127.0, 126.7, 126.5, 125.8, 106.7, 68.6, 65.5, 53.2, 49.0, 36.7, 31.3, 24.2, 21.5, 21.5. **HRMS** (ESI) calcd for $C_{31}H_{33}O_4NNaS_2$ $[M+Na]^+$ 570.1851; found: 570.1855.



(2-tosylethene-1,1-diyl)dibenzene (11)[5]: The title compound was obtained following procedure 3.3.3 andpurified by column chromatography on silica gel using (ethyl acetate/hexane = 15:85) as eluent to afford the product as an white solid; Mp. 141-142 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.47 (d, J = 8.3 Hz, 2H), 7.35 (ddd, J = 4.5, 3.7, 2.7 Hz, 2H), 7.33 – 7.27 (m, 4H), 7.22 – 7.18 (m, 2H), 7.15 (dd, J = 8.5, 0.6 Hz, 2H), 7.12 – 7.08 (m, 2H), 6.99 (s, 1H), 2.38 (s, 3H). 13C NMR (101 MHz, $CDCl_3$) δ 154.7, 143.7, 139.2, 138.6, 135.5, 130.2, 129.7, 129.3, 128.9, 128.8, 128.5, 128.2, 127.8, 127.7, 77.31, 21.53.



(*Z*)-4-(iodo(phenyl)methylene)-3-phenyl-1-tosyl-3-(tosylmethyl)pyrrolidine (12a): The title compound was obtained following procedure 3.3.4 and purified by column chromatography on silica gel using (ethyl acetate/hexane = 19:20) as eluent to afford as an white solid (56 mg, yield = 80%); Mp. 182-184 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.51 (d, *J* = 8.3 Hz, 2H), 7.41 (d, *J* = 7.9 Hz, 2H), 7.24 (d, *J* = 7.9 Hz, 2H), 7.20 – 7.11 (m, 3H), 7.10 – 7.05 (m, 3H), 7.01 (t, *J* = 7.4 Hz, 2H), 6.75 (d, *J* = 6.5 Hz, 2H), 4.26 (d, *J* = 14.8 Hz, 1H), 4.15 (d, *J* = 10.1 Hz, 1H), 4.03 (d, *J* = 4.5 Hz, 1H), 4.00 (d, *J* = 9.2 Hz, 1H), 3.49 (d, *J* = 14.6 Hz, 1H), 3.34 (d, *J* = 14.6 Hz, 1H), 2.49 (s, 3H), 2.42 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.8, 144.6, 144.1, 142.3, 142.0, 138.3, 131.8, 129.9, 129.8, 128.4, 128.2, 128.1, 127.9, 127.9, 127.4, 127.3, 126.2, 95.6, 61.8, 60.9, 60.4, 52.6, 21.7, 21.6. HRMS (ESI) calculated $[M+H]^+$ for $C_{32}H_{31}NO_4S_2I$ = 684.0739; found: 684.0743.



9. References

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3. a) T. Fan, X. Ma, Y. Liu, C. Jiang, Y. Xu and Y. Chen, *J. Org. Chem.*, **2022**, *87*, 5846–5855; b) W. Li and L. Zhou, *Org. Lett.*, **2022**, *24*, 3976–3981.
4. a) K. Kanemoto, S. Yoshida and T. Hosoya, *Chem. Lett.*, **2018**, *47*, 85–88; b) F. Marr, R. Fröhlich and D. Hoppe, *Tetrahedron: Asymmetry*, **2002**, *13*, 2587–2592.
5. Y. Yao, Z. Yin, F.-S. He, X. Qin, W. Xie and J. Wu, *Chem. Commun.*, **2021**, *57*, 2883–2886.

10. Solvent system and method for crystal growth of 1a, 3aa, 3ra, 5af, 7ak and 8a compounds for X-ray crystallographic analysis

Single crystals of compounds **1a**, **3aa**, **3ra**, **5af**, **7ak** and **8a** suitable for X-ray diffraction analysis were obtained by slow evaporation of acetone solutions of the pure compounds. To facilitate complete dissolution, each sample was refluxed in acetone, and the hot, clear solutions were then allowed to cool slowly to room temperature and left undisturbed under ambient conditions for several days. Crystals of suitable quality for X-ray analysis gradually formed during this process.

The crystallographic measurements were performed on a Rigaku Oxford XtaLab Pro II single-crystal X-ray diffractometer equipped with a microfocus sealed X-ray source and a HyPix detector. Data collection, structure solution, and refinement were carried out using standard crystallographic software packages. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed in idealized positions and refined using a riding model. The thermal ellipsoid plots are displayed at the 50% probability level to illustrate atomic displacement parameters.

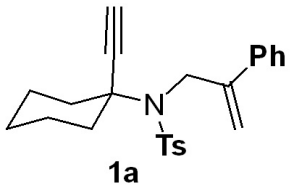
The crystal structures unambiguously confirm the molecular connectivity and relative stereochemistry of the compounds. Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre (CCDC) and are accessible free of charge via www.ccdc.cam.ac.uk/data_request/cif under the following accession numbers:

- **1a**: CCDC 2454650
- **3aa**: CCDC 2454665
- **3ra**: CCDC 2454663
- **5af**: CCDC 2454670
- **7ak**: CCDC 2454664
- **8a**: CCDC 2528349

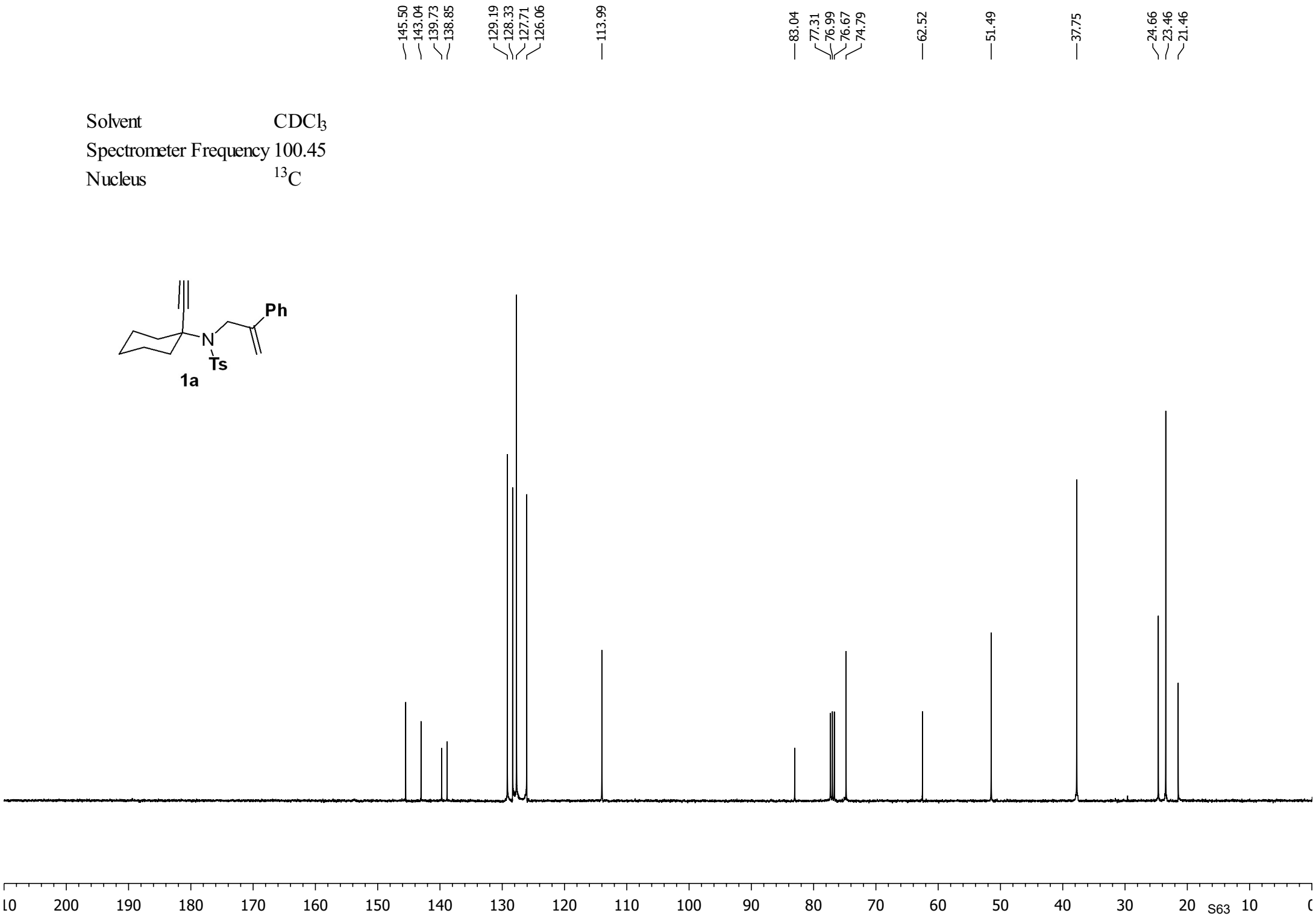
Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



1a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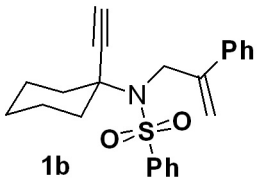
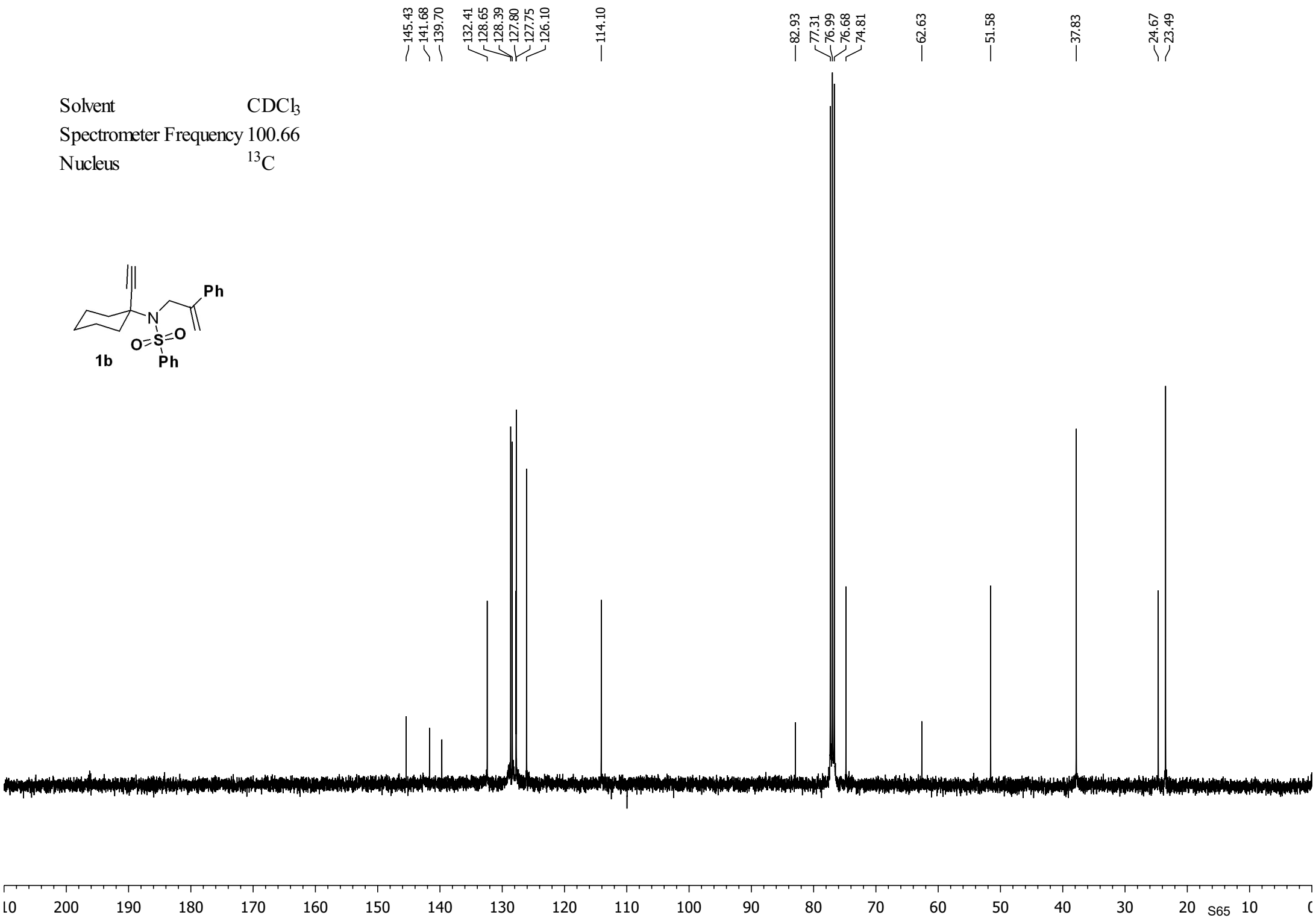
138.85
129.19
128.33
127.71
126.06
113.99
83.04
77.31
76.99
76.67
74.79
62.52
51.49
37.75
24.66
23.46
21.46
Solvent CDCl3
Spectrometer Frequency 100.45
Nucleus 13C
Ph
N
Ts
1a
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H

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139.70
132.41
128.65
128.39
127.80
127.75
126.10
114.10
82.93
77.31
76.99
76.68
74.81
62.63
51.58
37.83
24.67
23.49
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
Ph
N
O=S=O
Ph
1b
200
190
180
170
160
150
140
130
120
110
100
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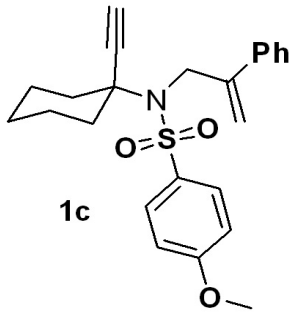
70
60
50
40
30
20
10

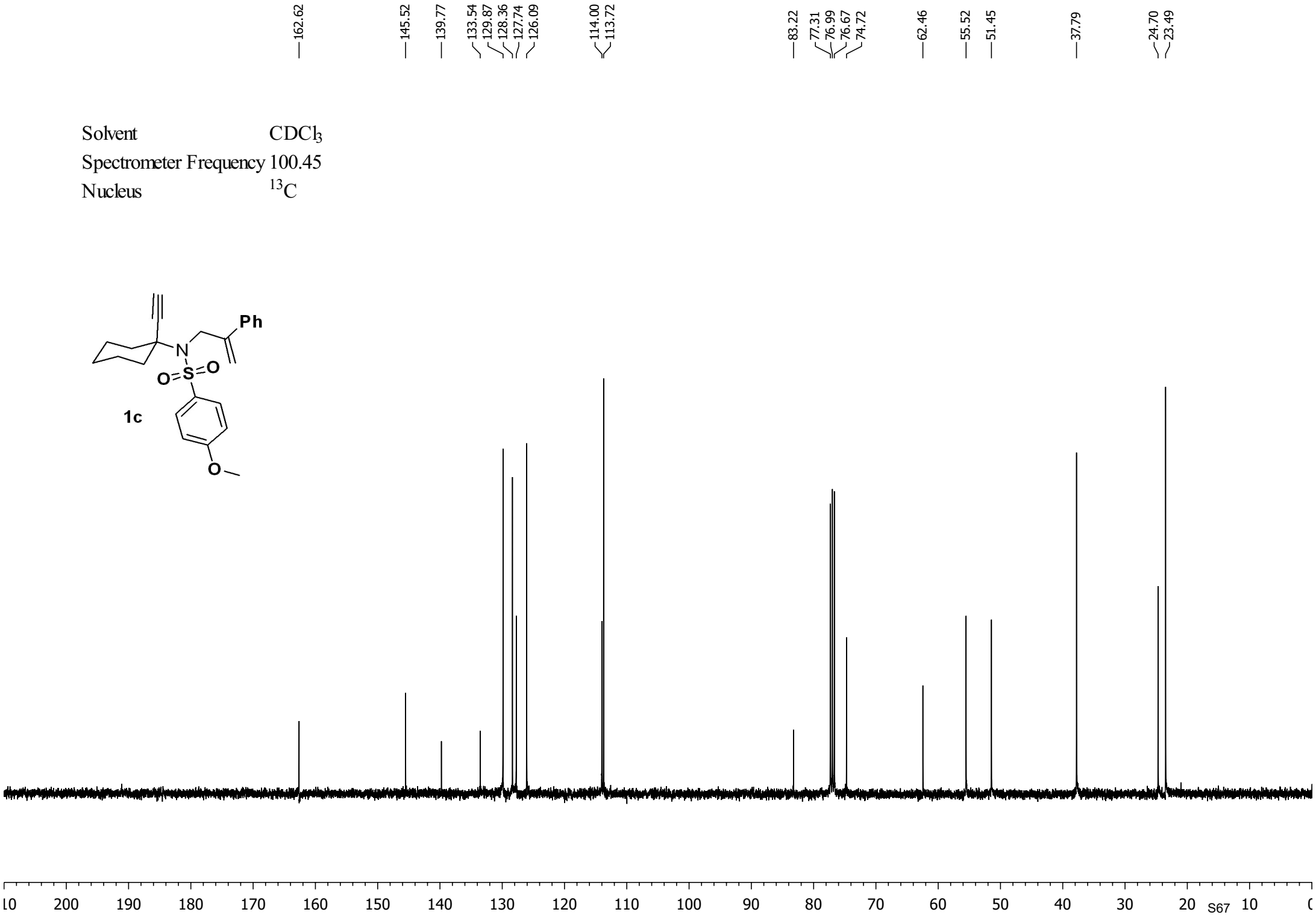
Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



1c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127.74
126.09
114.00
113.72
83.22
77.31
76.99
76.67
74.72
62.46
55.52
51.45
37.79
24.70
23.49
Solvent CDCl3
Spectrometer Frequency 100.45
Nucleus 13C
Ph
N
O=S=O
O
1c
200
190
180
170
160
150
140
130
120
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100
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80
70
60
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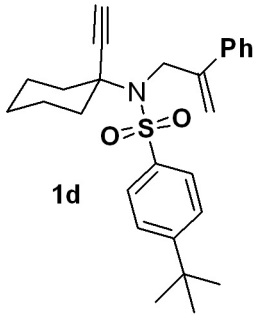
40
30
20
10

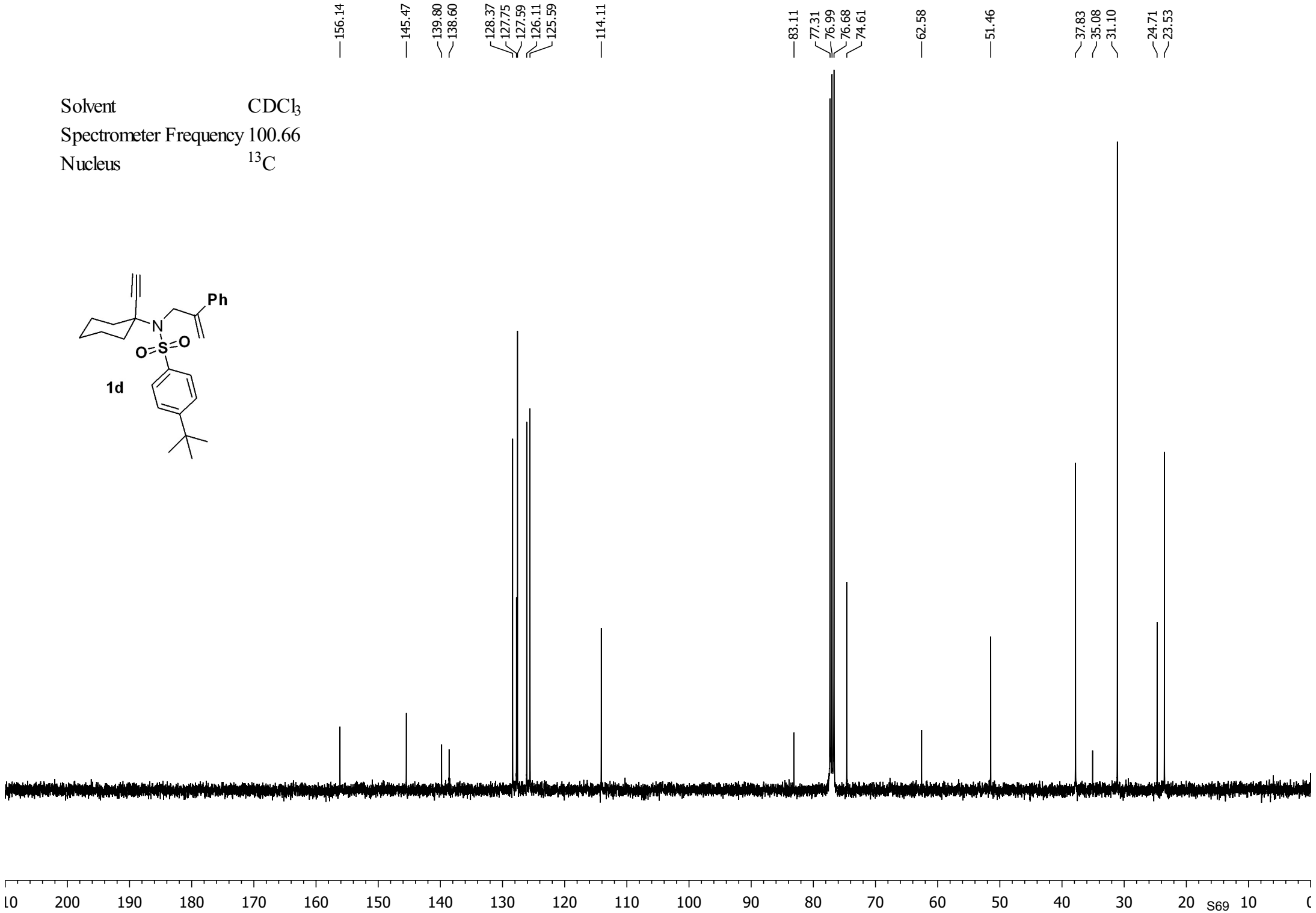
Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



1d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138.60
128.37
127.75
127.59
126.11
125.59
114.11
83.11
77.31
76.99
76.68
74.61
62.58
51.46
37.83
35.08
31.10
24.71
23.53
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
Ph
N
O=S=O
1d
200
190
180
170
160
150
140
130
120
110
100
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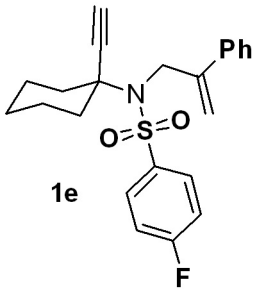
80
70
60
50
40
30
20
10

Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



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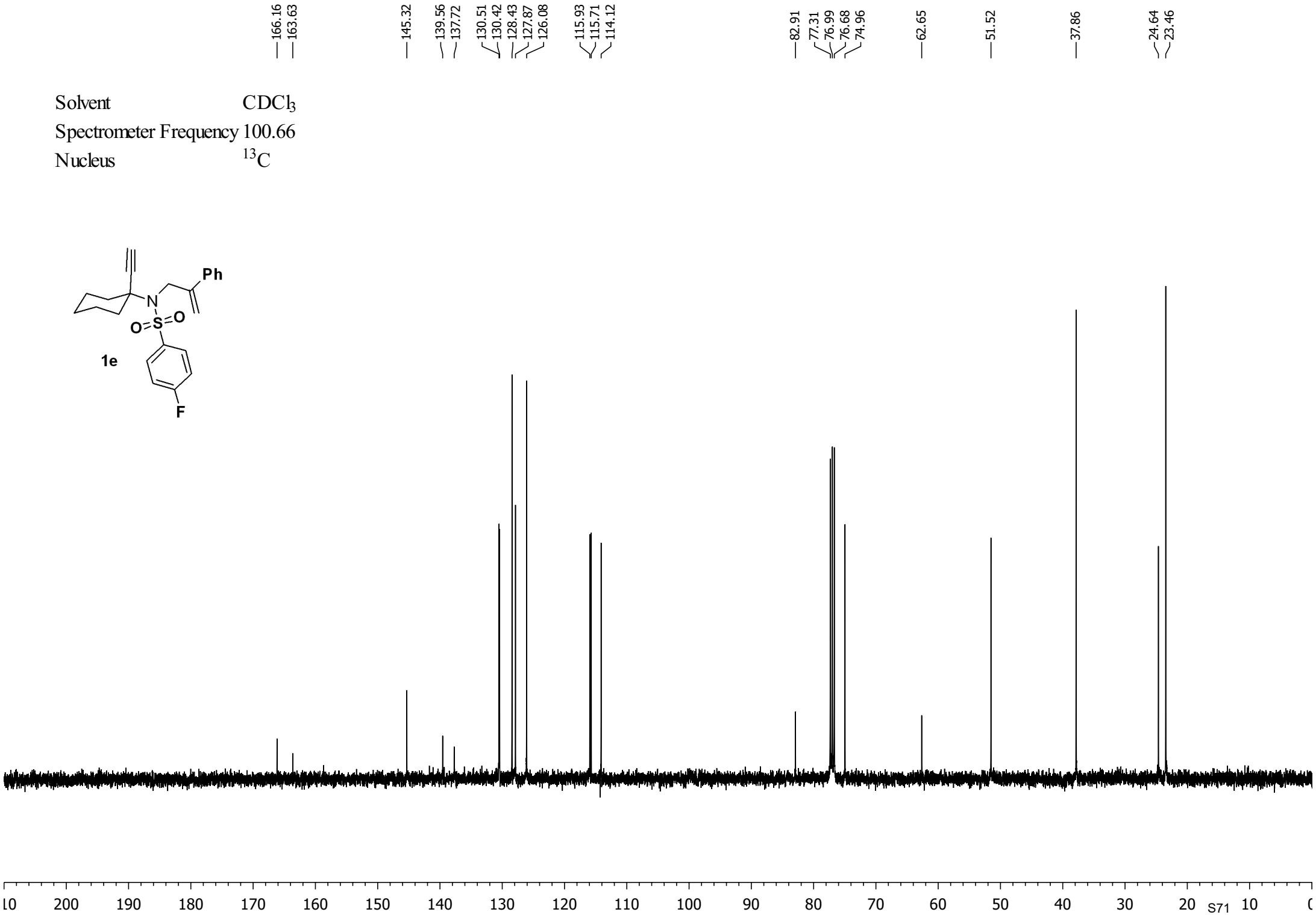
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126.08
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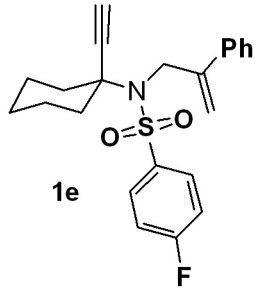
114.12
82.91
77.31
76.99
76.68
74.96
62.65
51.52
37.86
24.64
23.46
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
Ph
N
O=S=O
1e
F
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

-105.72

Solvent $CDCl_3$
Spectrometer Frequency 376.61
Nucleus ^{19}F



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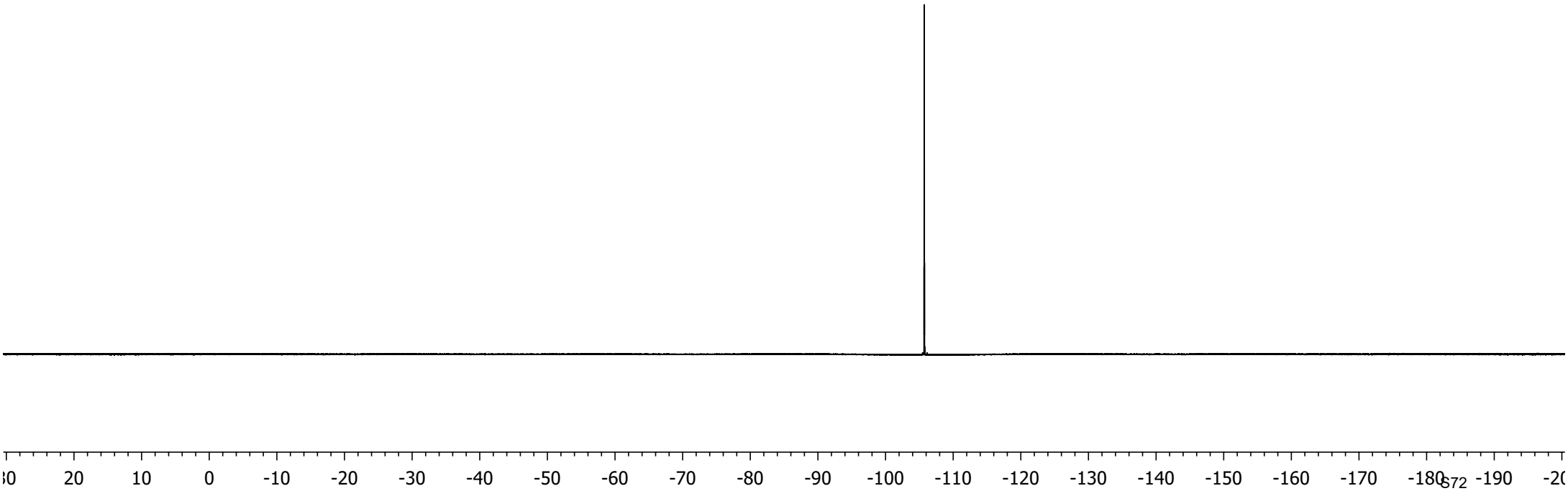

Solvent $CDCl_3$

Spectrometer Frequency 400.28

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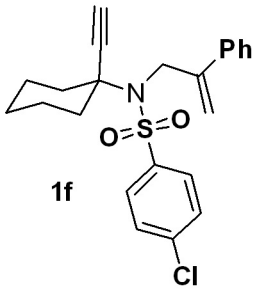

1f

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138.80
129.23
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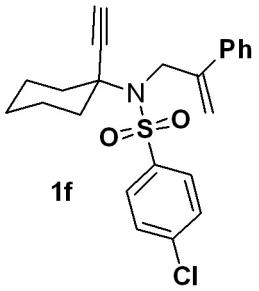
127.89
126.09
114.16
82.82
77.31
76.99
76.68
75.12
62.72
51.55
37.85
24.62
23.47

Solvent $CDCl_3$

Spectrometer Frequency 100.66

Nucleus ^{13}C



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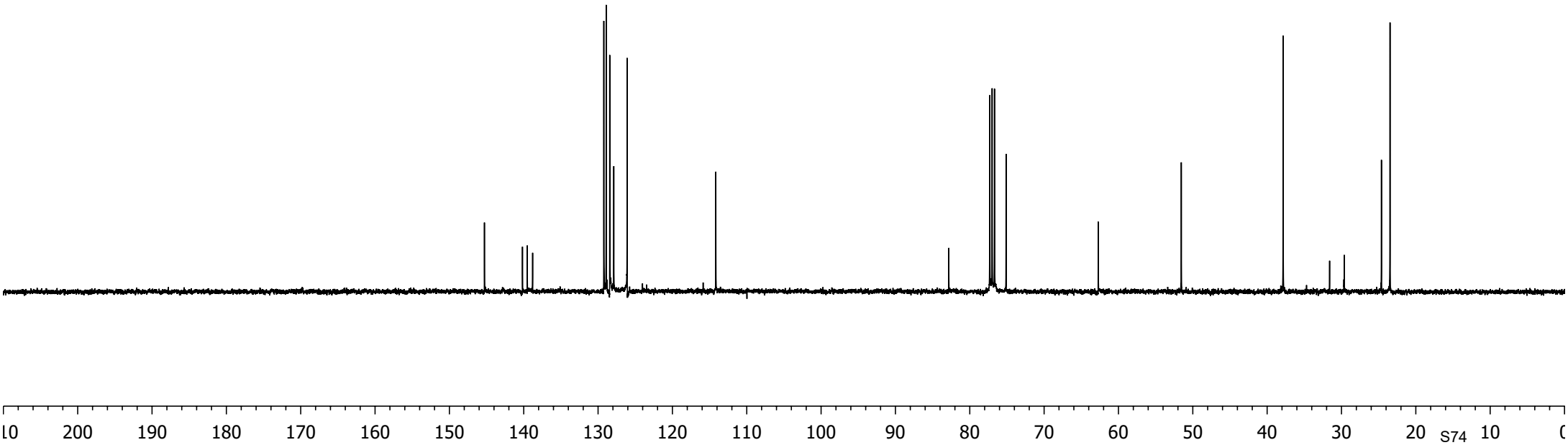
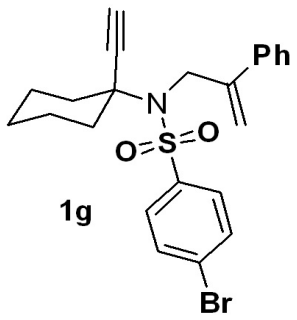


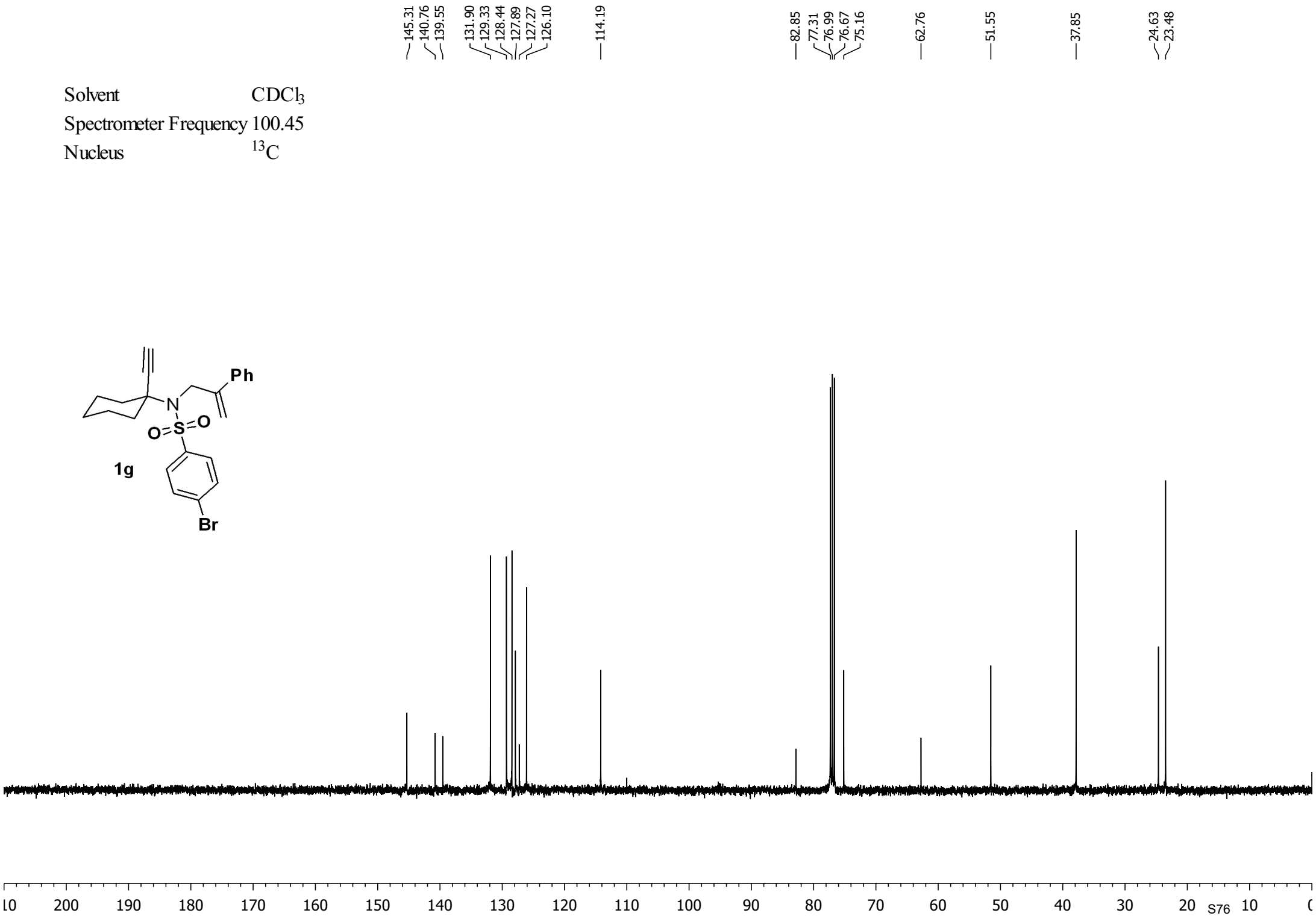
Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



1g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131.90
129.33
128.44
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127.27
126.10
114.19
82.85
77.31
76.99
76.67
75.16
62.76
51.55
37.85
24.63
23.48
Solvent CDCl3
Spectrometer Frequency 100.45
Nucleus 13C
Ph
N
O=S=O
1g
Br
200
190
180
170
160
150
140
130
120
110
100
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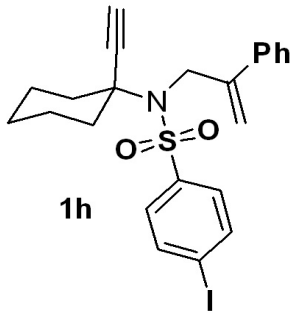
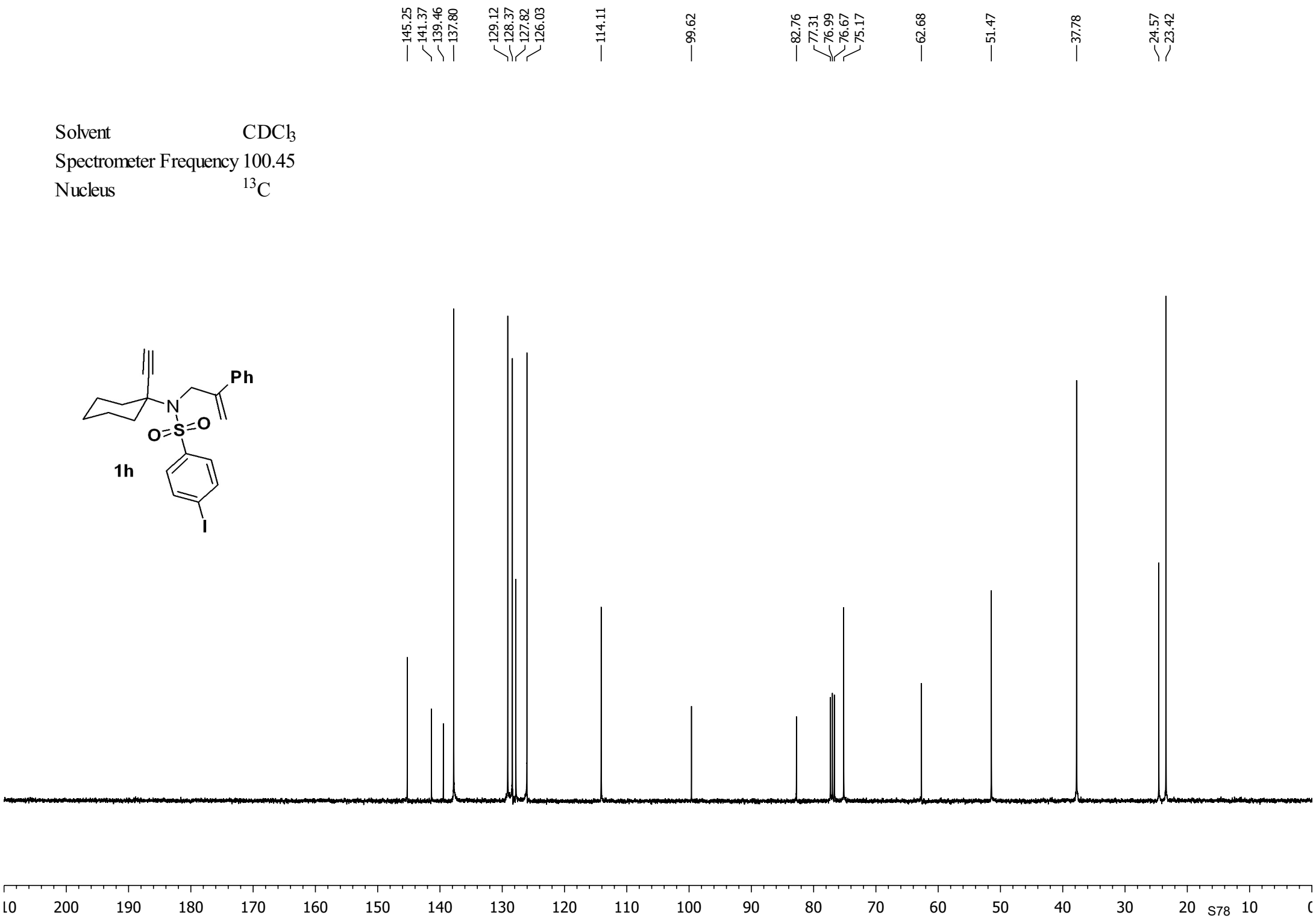
80
70
60
50
40
30
20
10

Solvent $CDCl_3$
Spectrometer Frequency 399.44
Nucleus ^1H

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76.67
75.17
62.68
51.47
37.78
24.57
23.42
Solvent CDCl3
Spectrometer Frequency 100.45
Nucleus 13C
Ph
N
O=S=O
1h
I
200
190
180
170
160
150
140
130
120
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100
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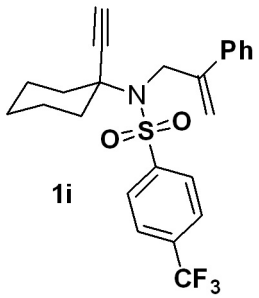
80
70
60
50
40
30
20
10

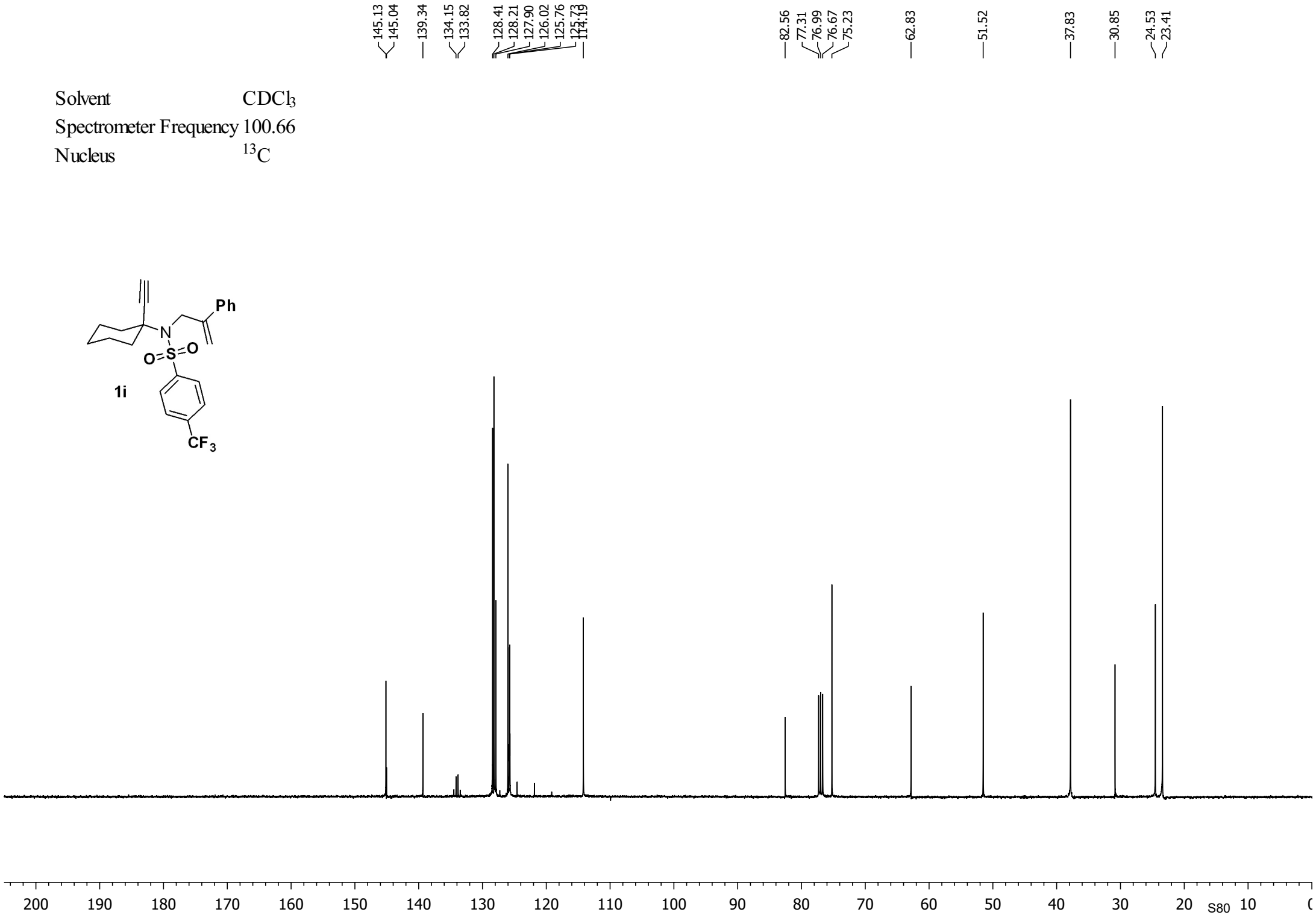
Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



1i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134.15
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127.90
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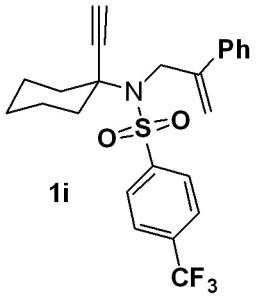
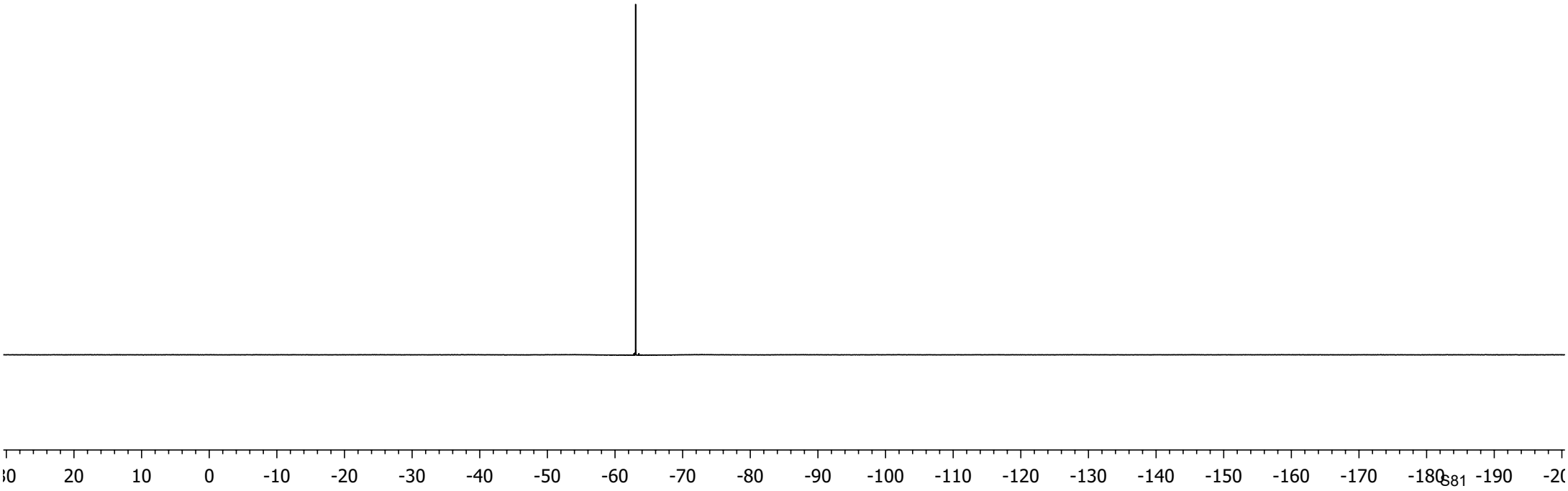
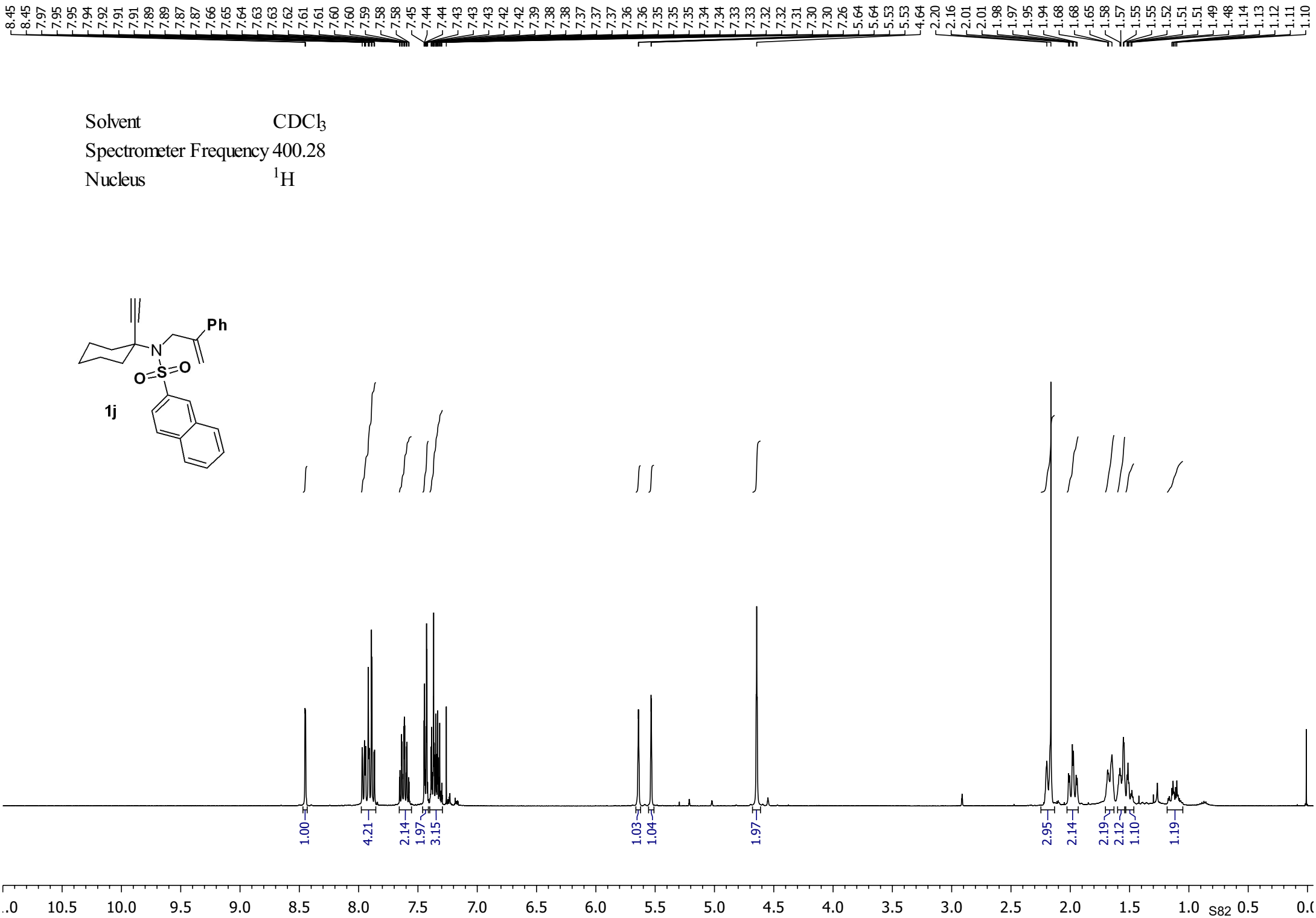
125.76
125.73
114.19
82.56
77.31
76.99
76.67
75.23
62.83
51.52
37.83
30.85
24.53
23.41
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
Ph
N
O=S=O
CF3
1i
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

Solvent $CDCl_3$
Spectrometer Frequency 376.61
Nucleus ^{19}F



1i

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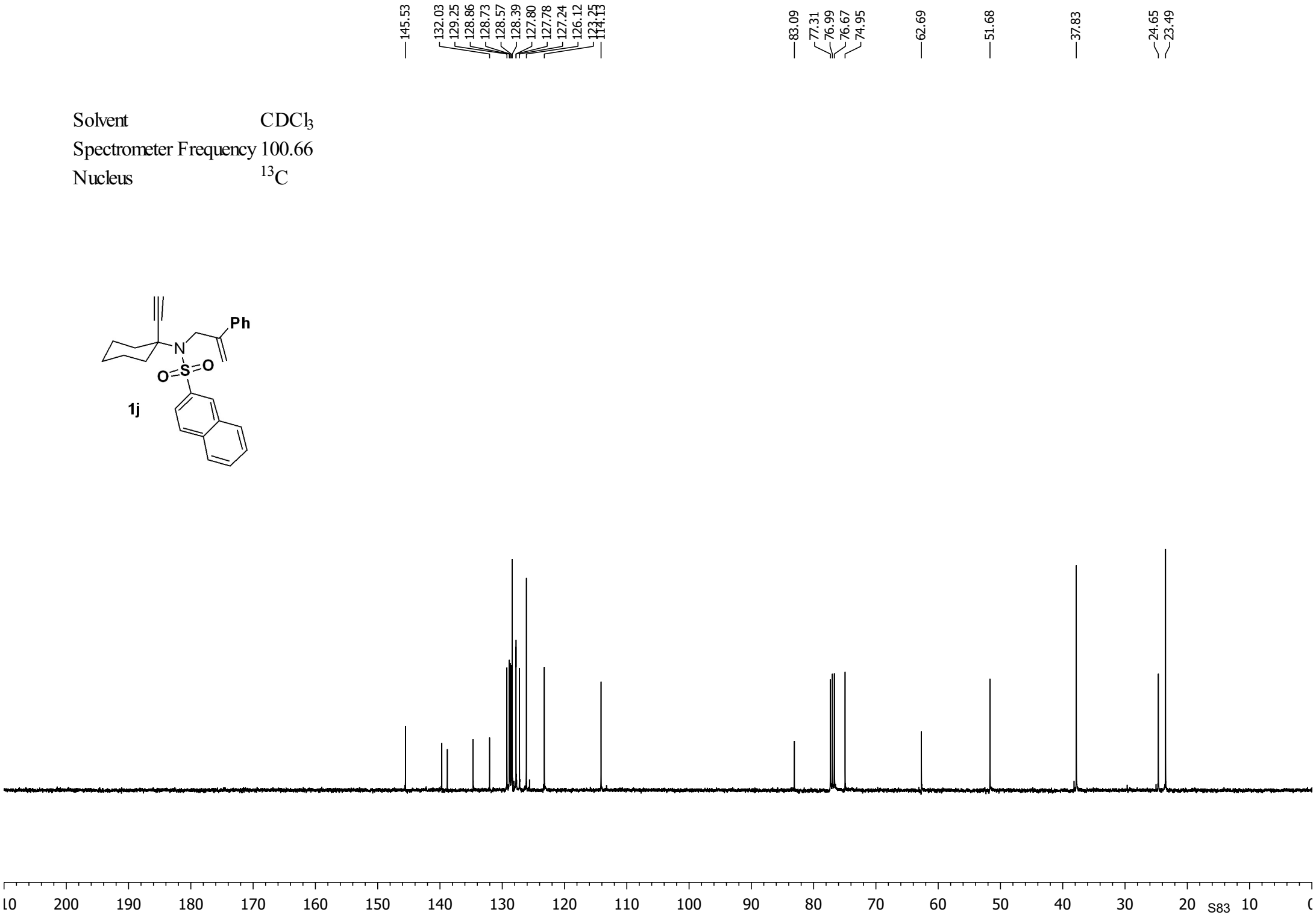
Solvent CDCl3
Spectrometer Frequency 400.28
Nucleus 1H
Ph
N
O=S=O
1j
1.00 4.21 2.14 1.97 3.15 1.03 1.04 1.97 2.95 2.14 2.19 2.12 1.10 1.19
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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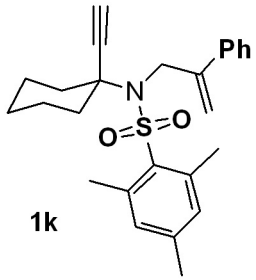
126.12
123.25
114.13
83.09
77.31
76.99
76.67
74.95
62.69
51.68
37.83
24.65
23.49
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
Ph
N
O=S=O
1j
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

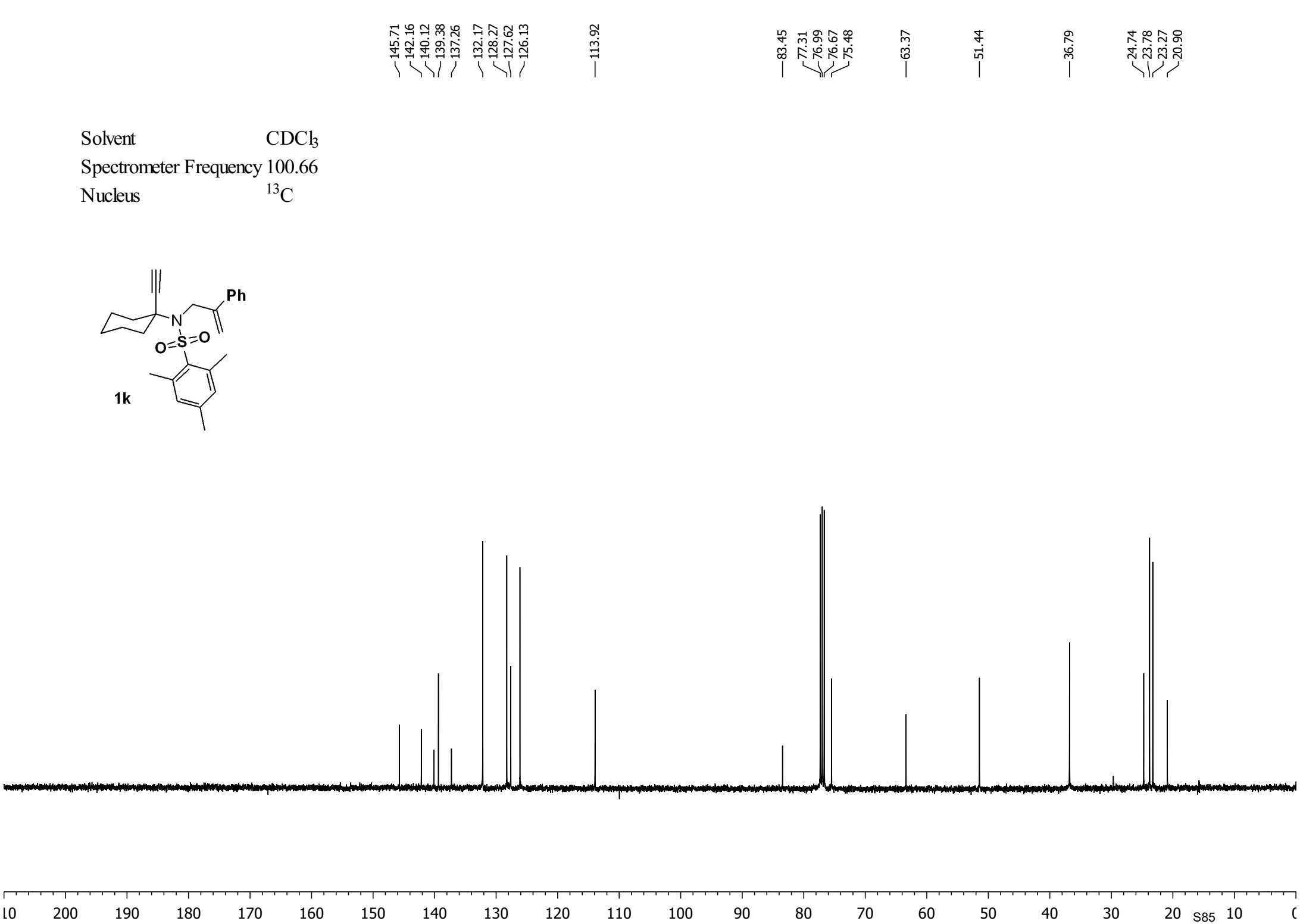
Solvent $CDCl_3$

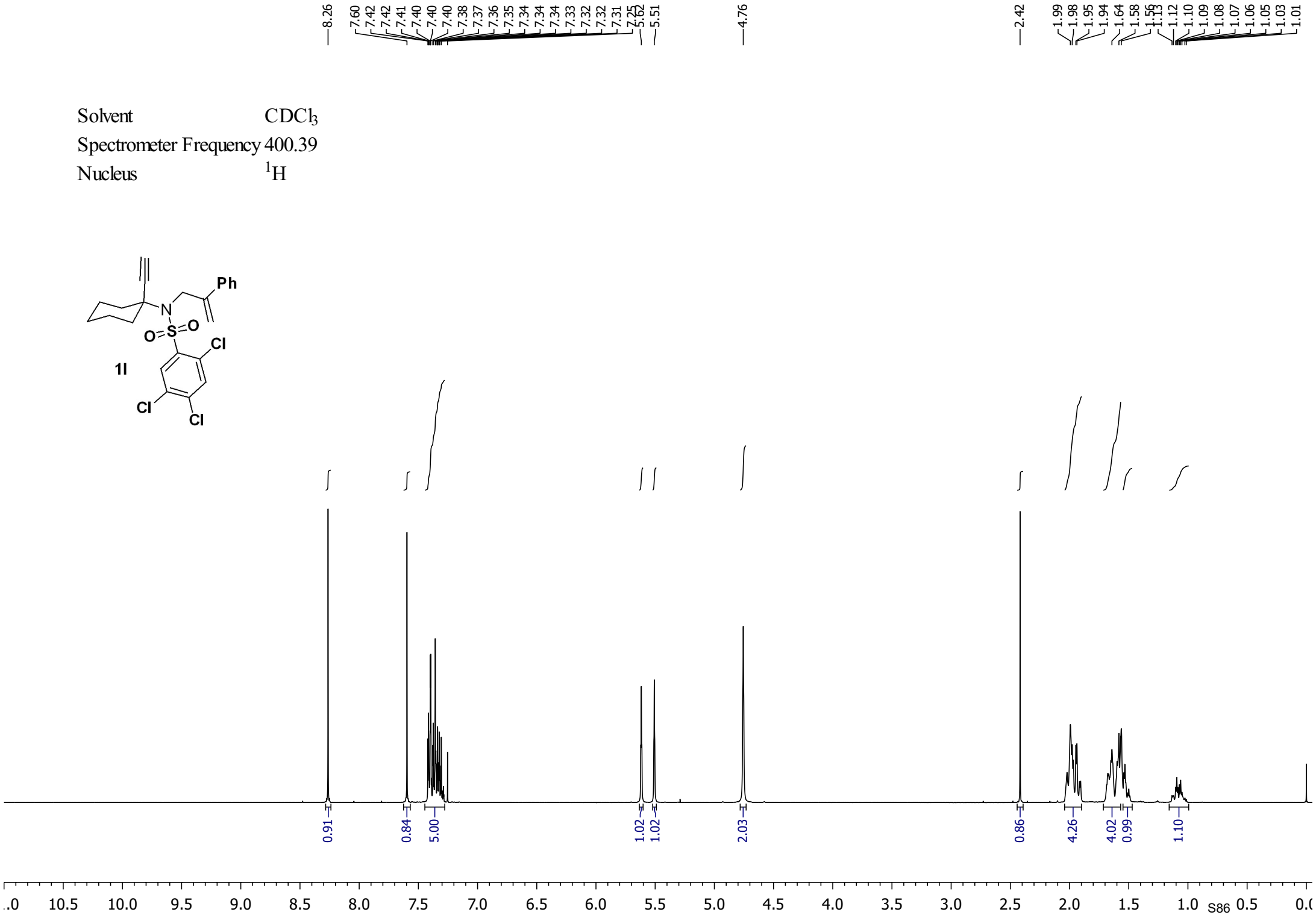
Spectrometer Frequency 400.28

Nucleus ^1H



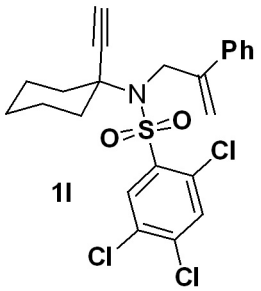
1k


145.71
142.16
140.12
139.38
137.26
132.17
128.27
127.62
126.13
113.92
83.45
77.31
76.99
76.67
75.48
63.37
51.44
36.79
24.74
23.78
23.27
20.90
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
Ph
N
O=S=O
1k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

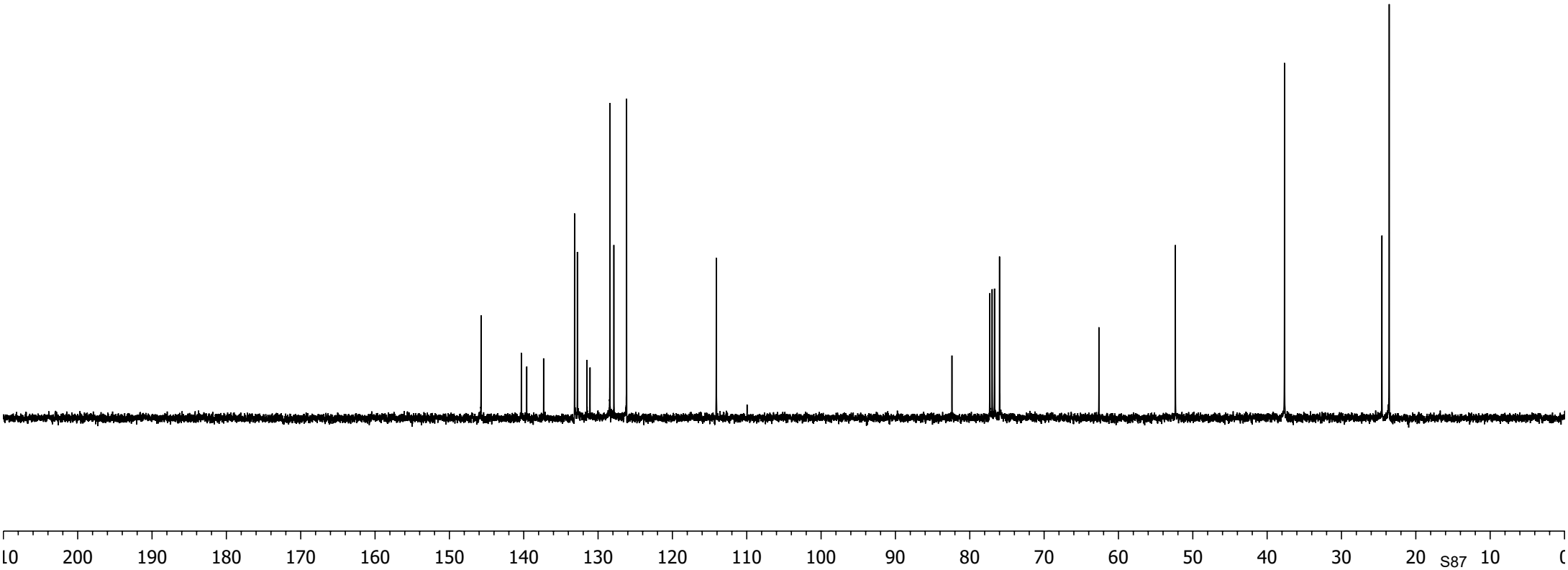

8.26
7.60
7.42
7.42
7.41
7.40
7.40
7.40
7.38
7.37
7.36
7.35
7.34
7.34
7.34
7.33
7.32
7.32
7.31
5.62
5.51
4.76
2.42
1.99
1.98
1.95
1.94
1.64
1.58
1.12
1.10
1.09
1.08
1.07
1.06
1.05
1.03
1.01
Solvent CDCl3
Spectrometer Frequency 400.39
Nucleus 1H
Ph
N
O=S=O
Cl
Cl
Cl
1l
0.91
0.84
5.00
1.02
1.02
2.03
0.86
4.26
4.02
0.99
1.10
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

145.74
140.33
139.59
137.33
133.17
132.79
131.50
131.08
128.41
127.87
126.16
114.10
82.40
77.31
76.99
76.67
76.00
62.63
52.35
37.66
24.56
23.58

Solvent $CDCl_3$
Spectrometer Frequency 100.69
Nucleus ^{13}C



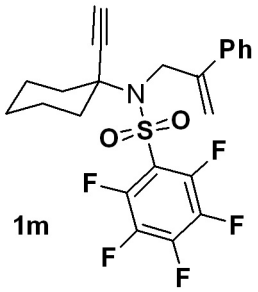
1l



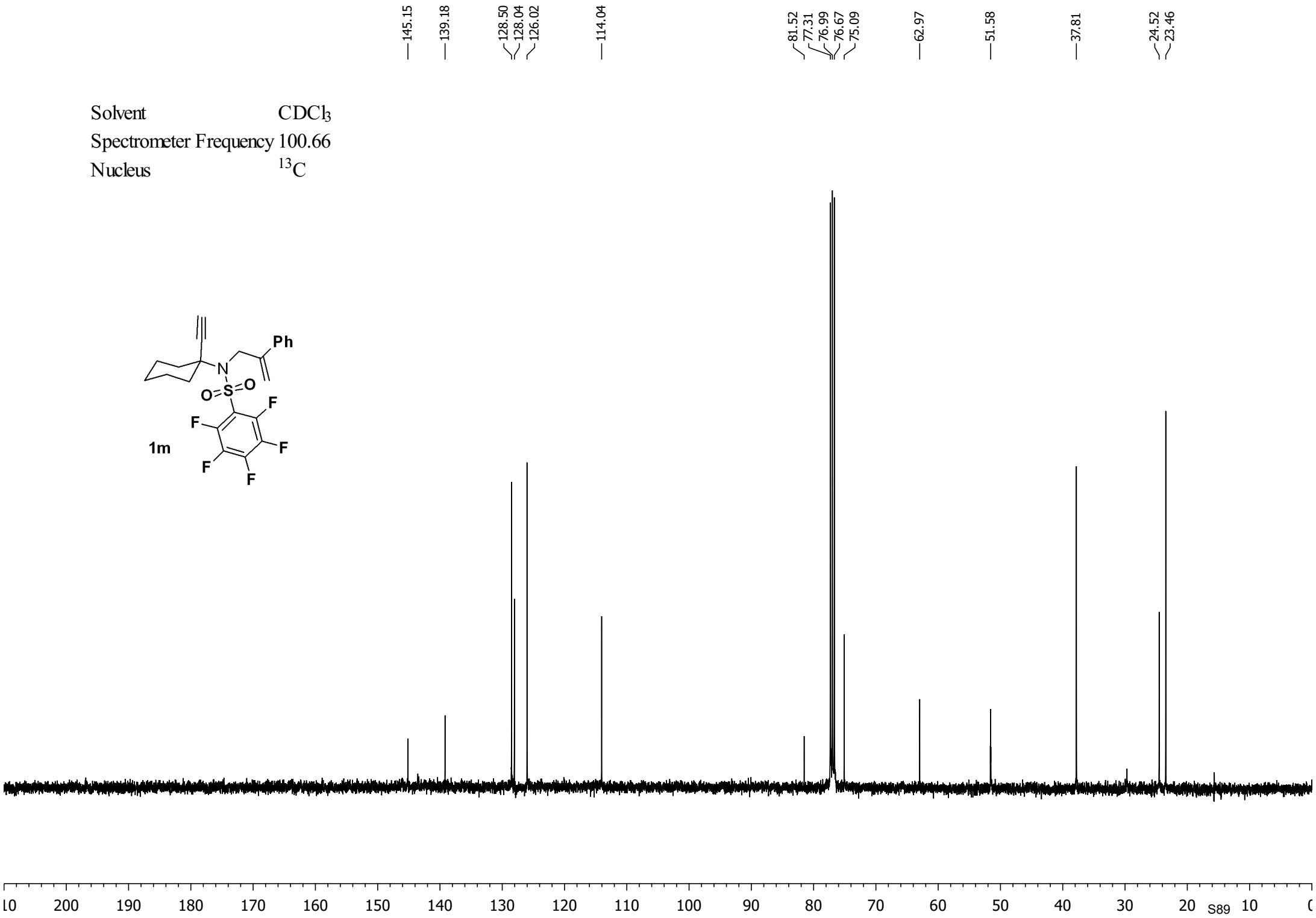
Solvent $CDCl_3$

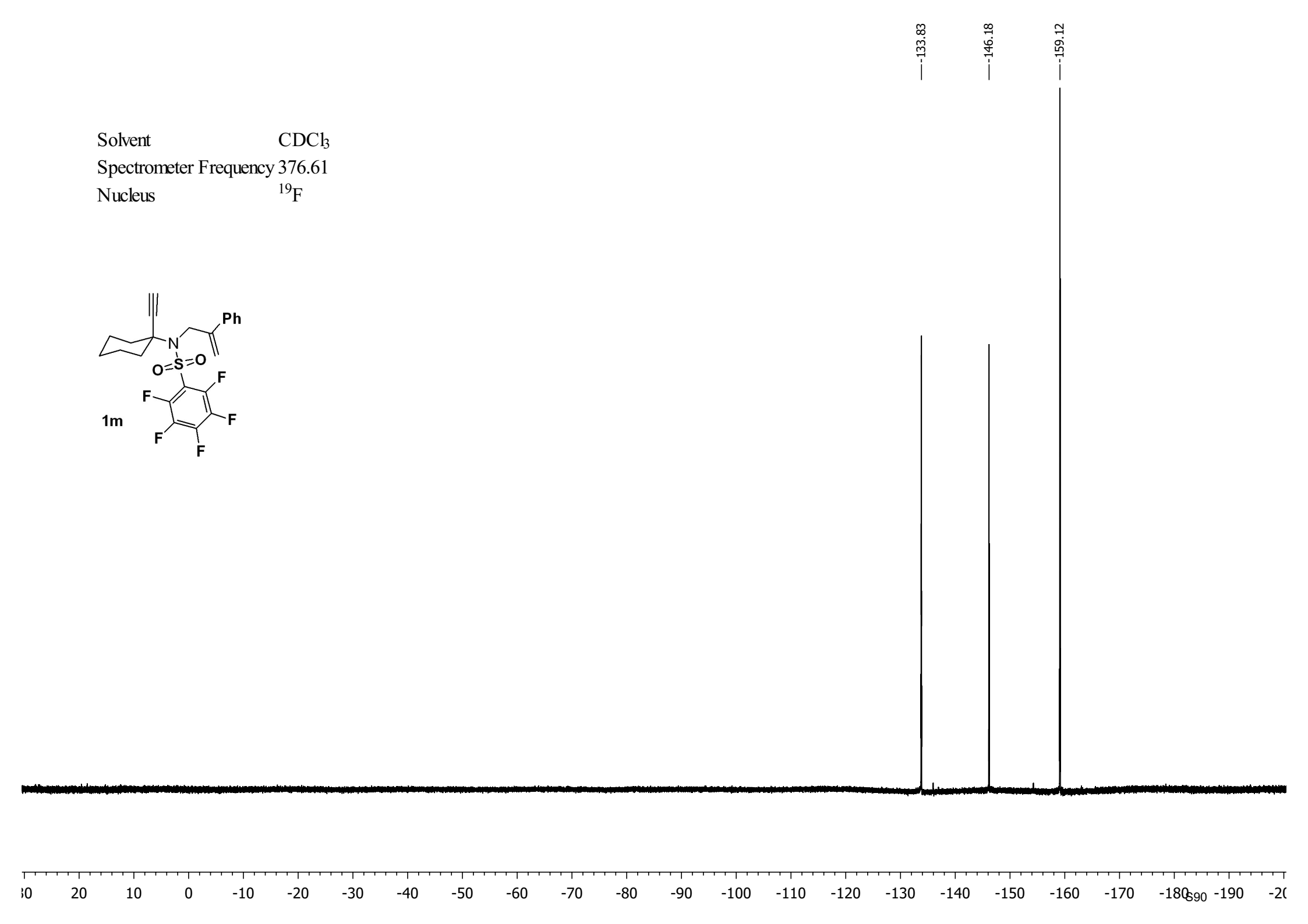
Spectrometer Frequency 400.28

Nucleus ^1H



1m

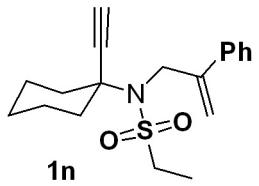

145.15
139.18
128.50
128.04
126.02
114.04
81.52
77.31
76.99
76.67
75.09
62.97
51.58
37.81
24.52
23.46
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
Ph
N
O=S=O
F
F
F
F
F
1m
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

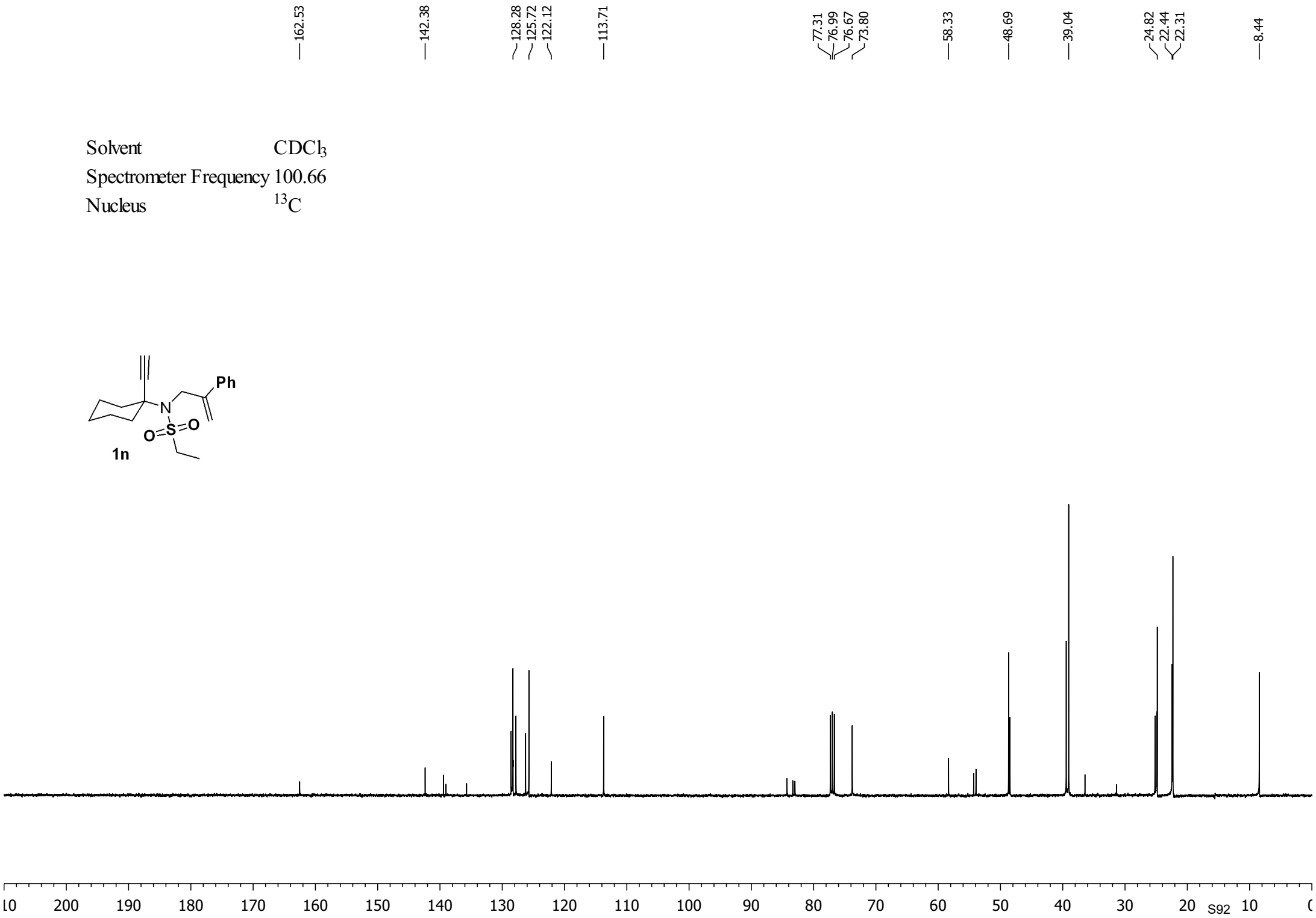

-133.83
-146.18
-159.12
Solvent CDCl3
Spectrometer Frequency 376.61
Nucleus 19F
Ph
N
O=S=O
F
F
F
F
F
1m
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -20

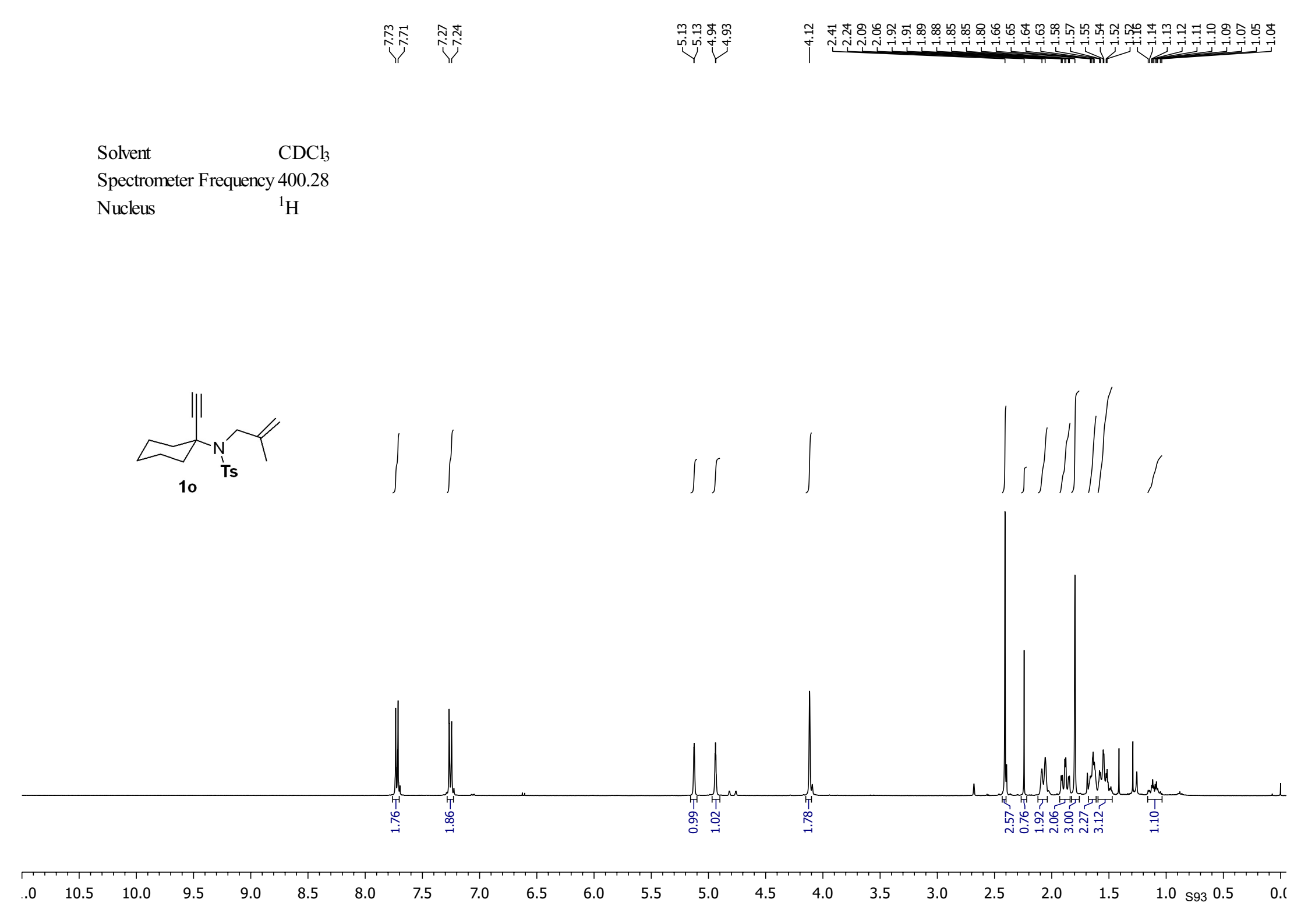
Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H

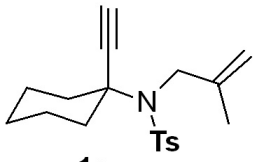



162.53
142.38
128.28
125.72
122.12
113.71
77.31
76.99
76.67
73.80
58.33
48.69
39.04
24.82
22.44
22.31
8.44
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
Ph
N
O=S=O
1n
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

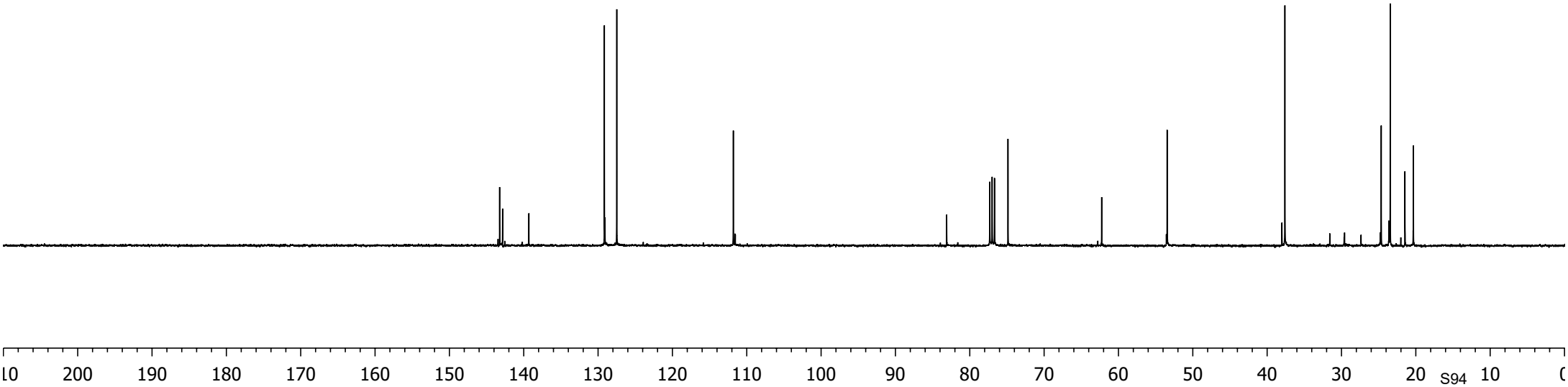

7.73
7.71
7.27
7.24
5.13
5.13
4.94
4.93
4.12
2.41
2.24
2.09
2.06
1.92
1.91
1.89
1.88
1.85
1.85
1.80
1.66
1.65
1.64
1.63
1.58
1.57
1.55
1.54
1.52
1.14
1.13
1.12
1.11
1.10
1.09
1.07
1.05
1.04
Solvent CDCl3
Spectrometer Frequency 400.28
Nucleus 1H
N
Ts
1o
1.76
1.86
0.99
1.02
1.78
2.57
0.76
1.92
2.06
3.00
2.27
3.12
1.10
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

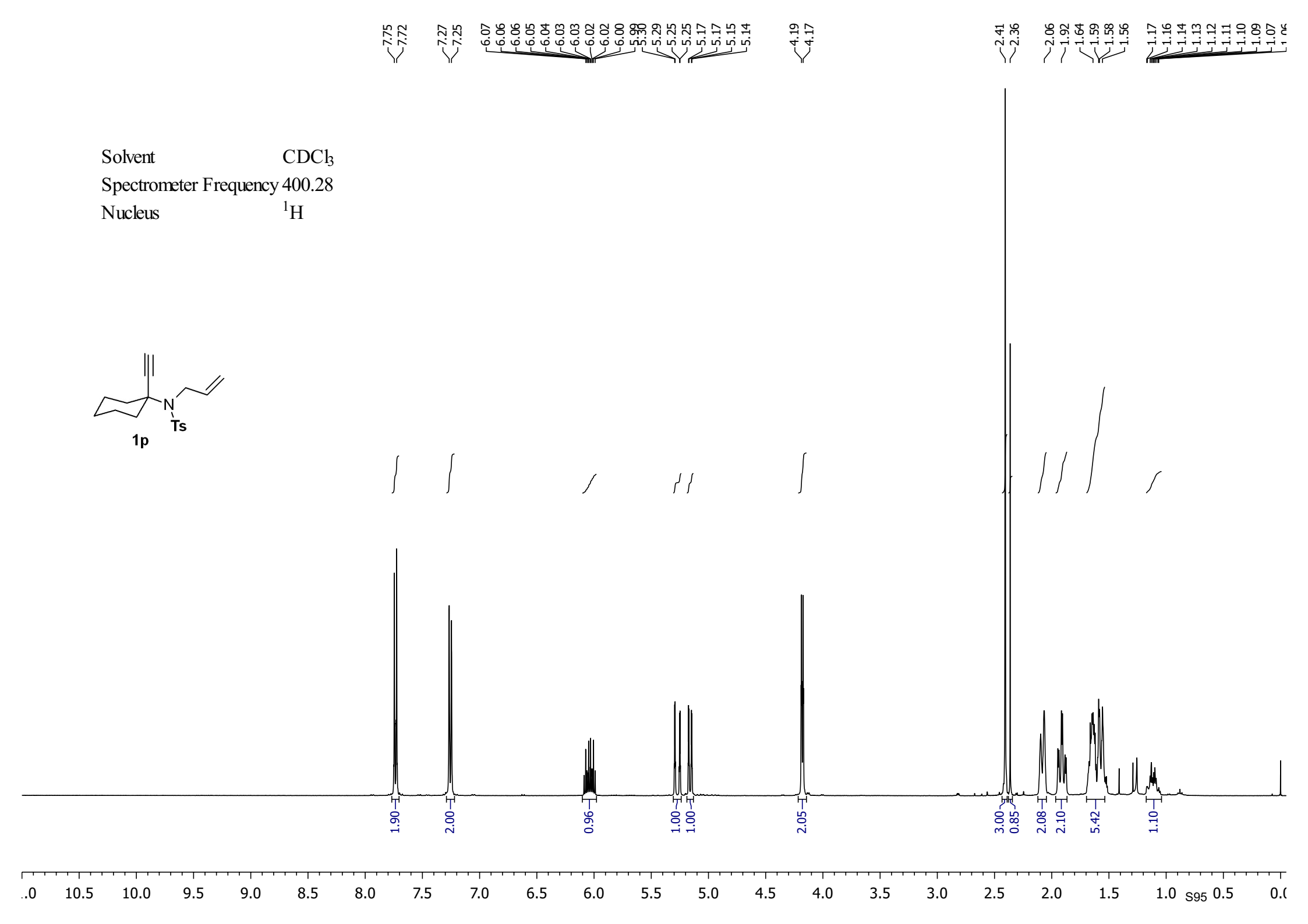
143.24
142.85
139.31
129.15
127.48
111.80
83.10
77.31
76.99
76.67
74.86
62.26
53.45
37.61
24.65
23.44
21.46
20.32

Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C



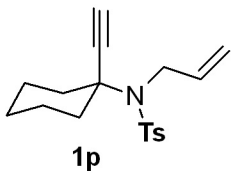
1o




Solvent CDCl3
Spectrometer Frequency 400.28
Nucleus 1H
Ts
1p
7.75
7.72
7.27
7.25
6.07
6.06
6.06
6.05
6.04
6.03
6.03
6.02
6.02
6.00
5.99
5.30
5.29
5.25
5.25
5.17
5.17
5.15
5.14
4.19
4.17
2.41
2.36
2.06
1.92
1.64
1.59
1.58
1.56
1.17
1.16
1.14
1.13
1.12
1.11
1.10
1.09
1.07
1.90
2.00
0.96
1.00
1.00
2.05
3.00
0.85
2.08
2.10
5.42
1.10
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

142.78
139.74
137.09
129.22
127.25
116.68
83.54
77.31
76.99
76.67
75.02
62.09
50.04
37.46
24.67
23.43
21.44

Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C

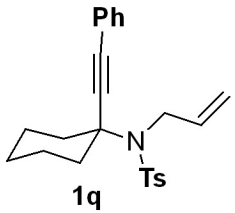


1p

Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



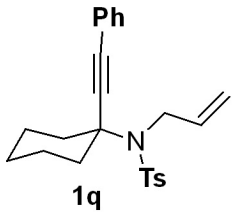
1q

142.74
139.44
137.25
131.44
129.28
128.20
128.09
127.36
122.59
116.52
89.04
86.96
77.31
76.99
76.67
62.82
50.41
37.94
24.82
23.71
21.38

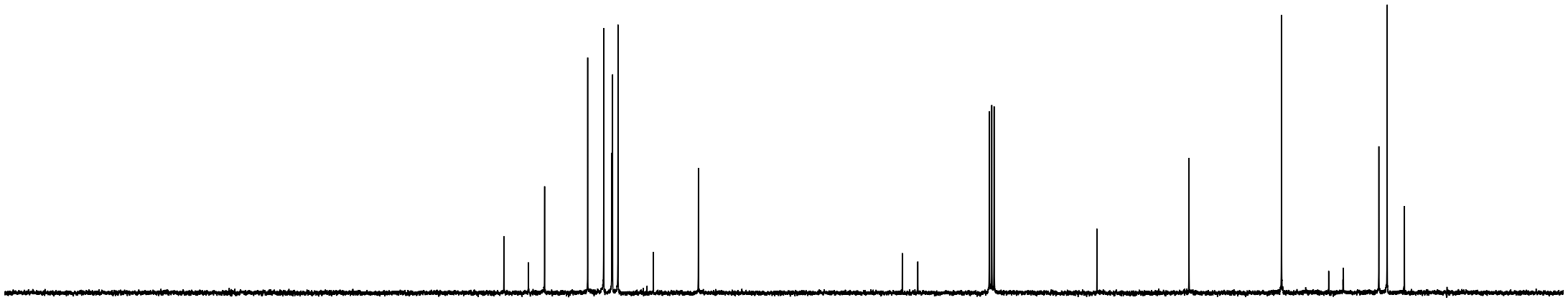
Solvent $CDCl_3$

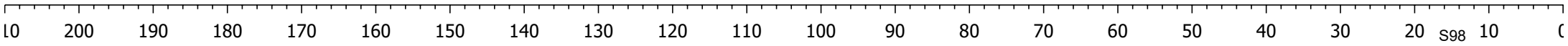
Spectrometer Frequency 100.66

Nucleus ^{13}C



1q

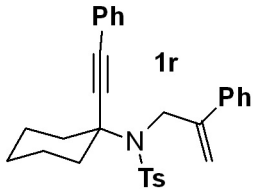


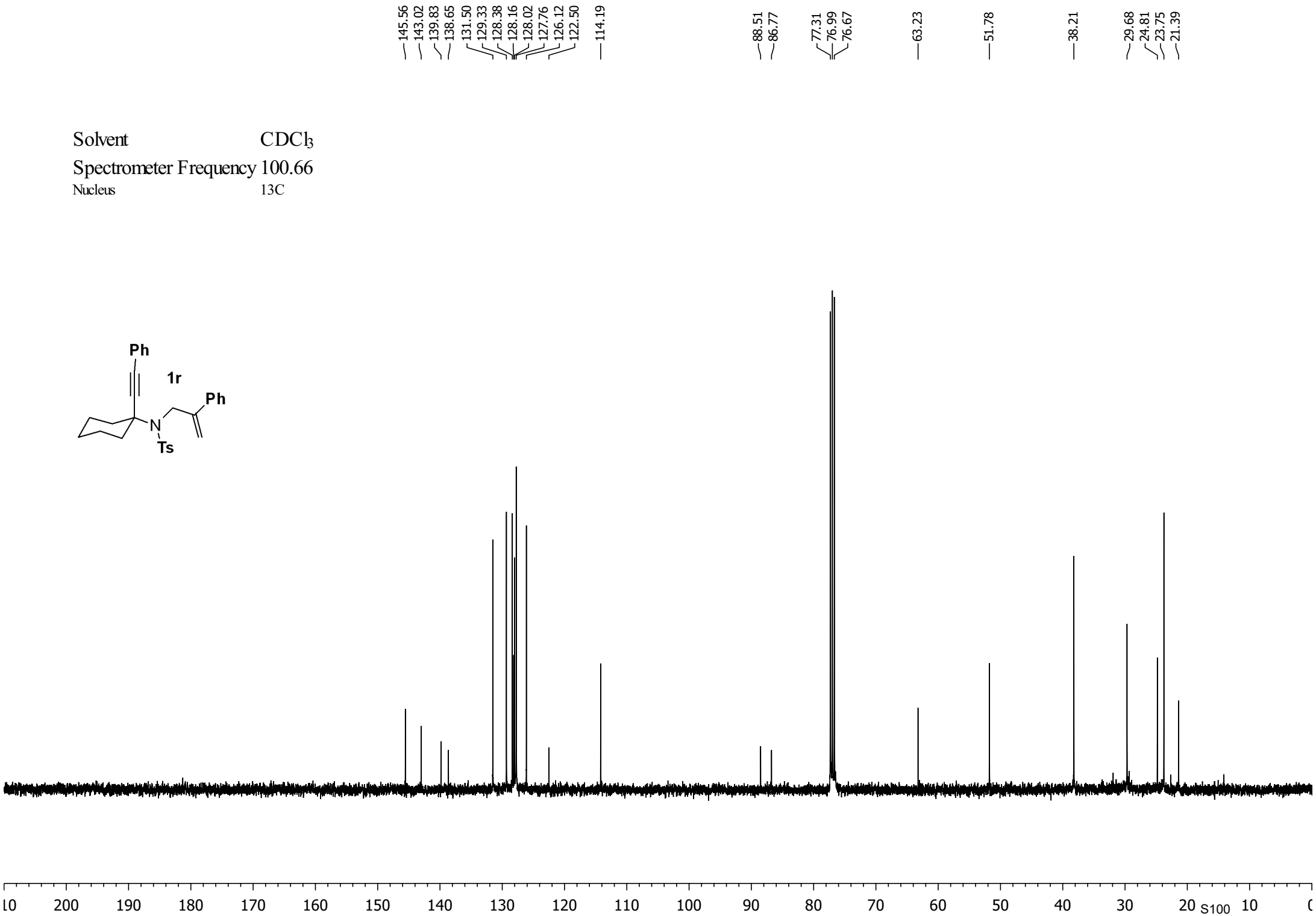


Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H

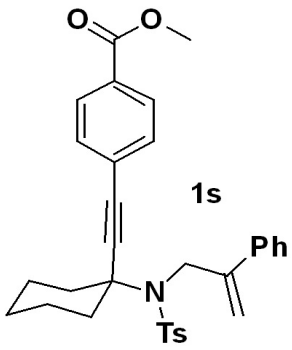



145.56
143.02
139.83
138.65
131.50
129.33
128.38
128.16
128.02
127.76
126.12
122.50
114.19
88.51
86.77
77.31
76.99
76.67
63.23
51.78
38.21
29.68
24.81
23.75
21.39
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
Ph
1r
Ph
N
Ts
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

Solvent $CDCl_3$

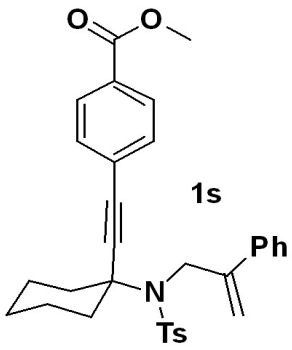
Spectrometer Frequency 400.28

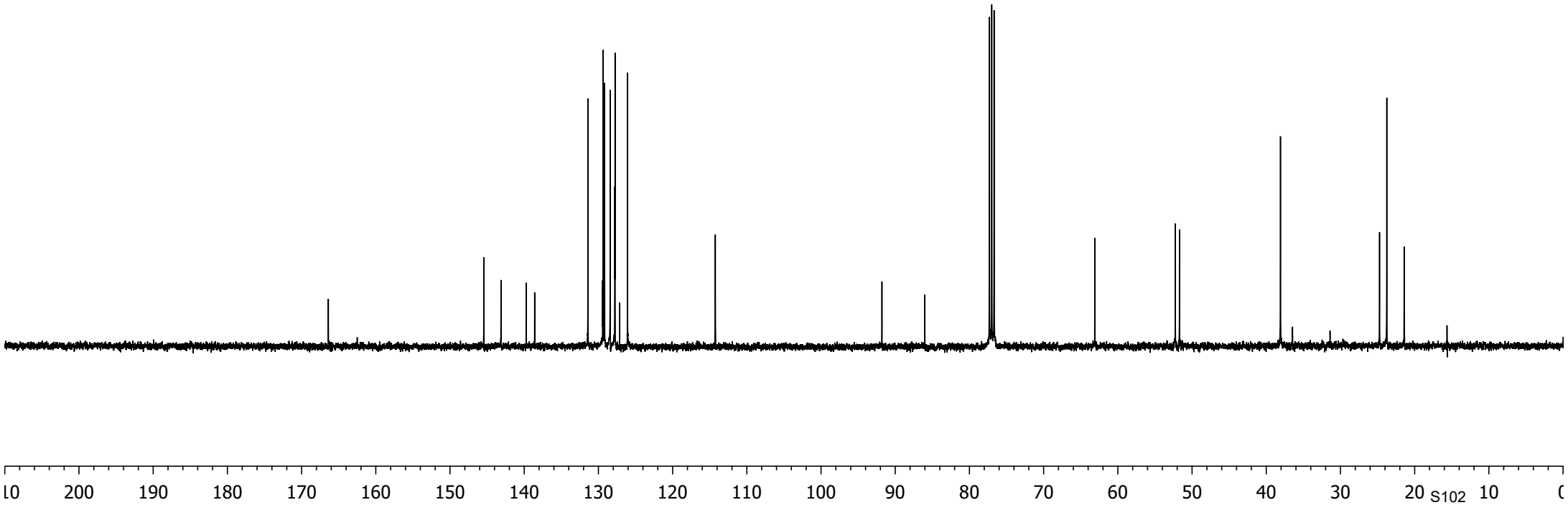
Nucleus ^1H

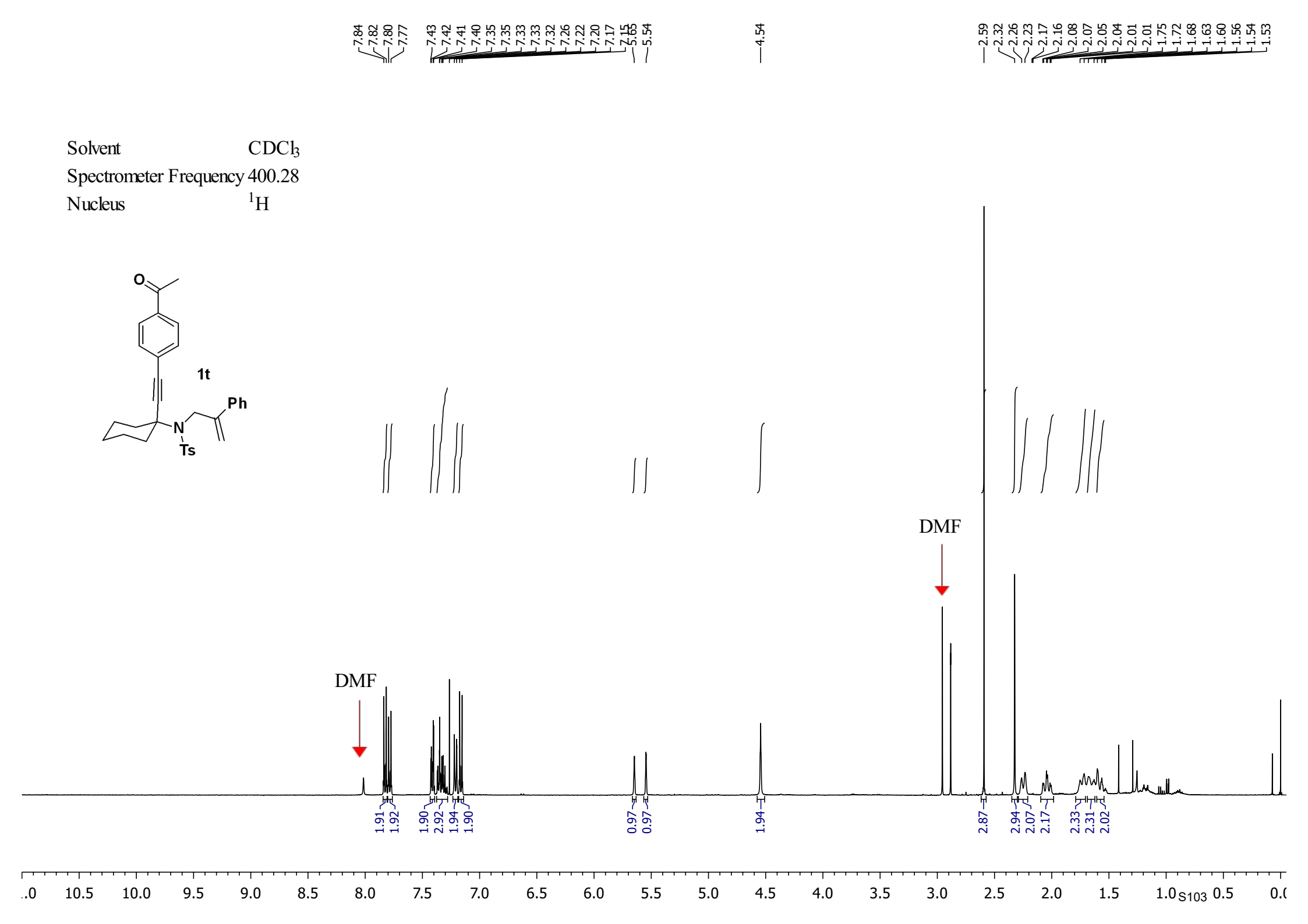


166.40
145.44
143.13
139.74
138.59
131.41
129.36
129.17
128.41
127.75
126.08
114.25
91.79
86.02
77.31
76.99
76.67
63.10
52.26
51.69
38.08
24.73
23.75
21.42

Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C



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7.82
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7.77
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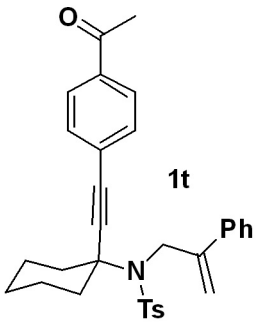
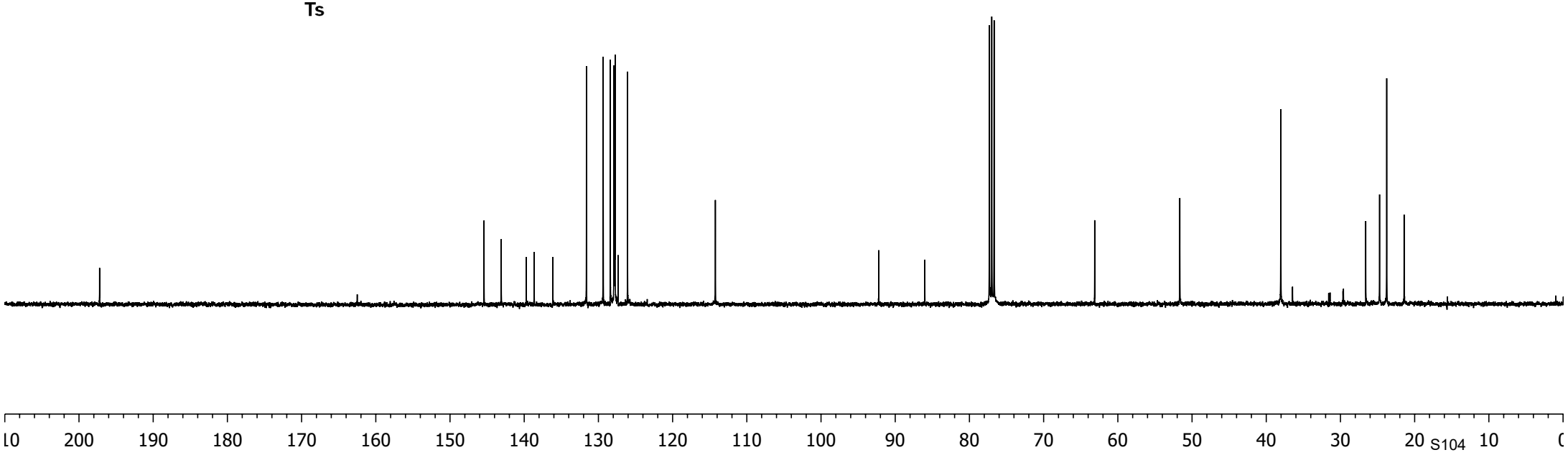
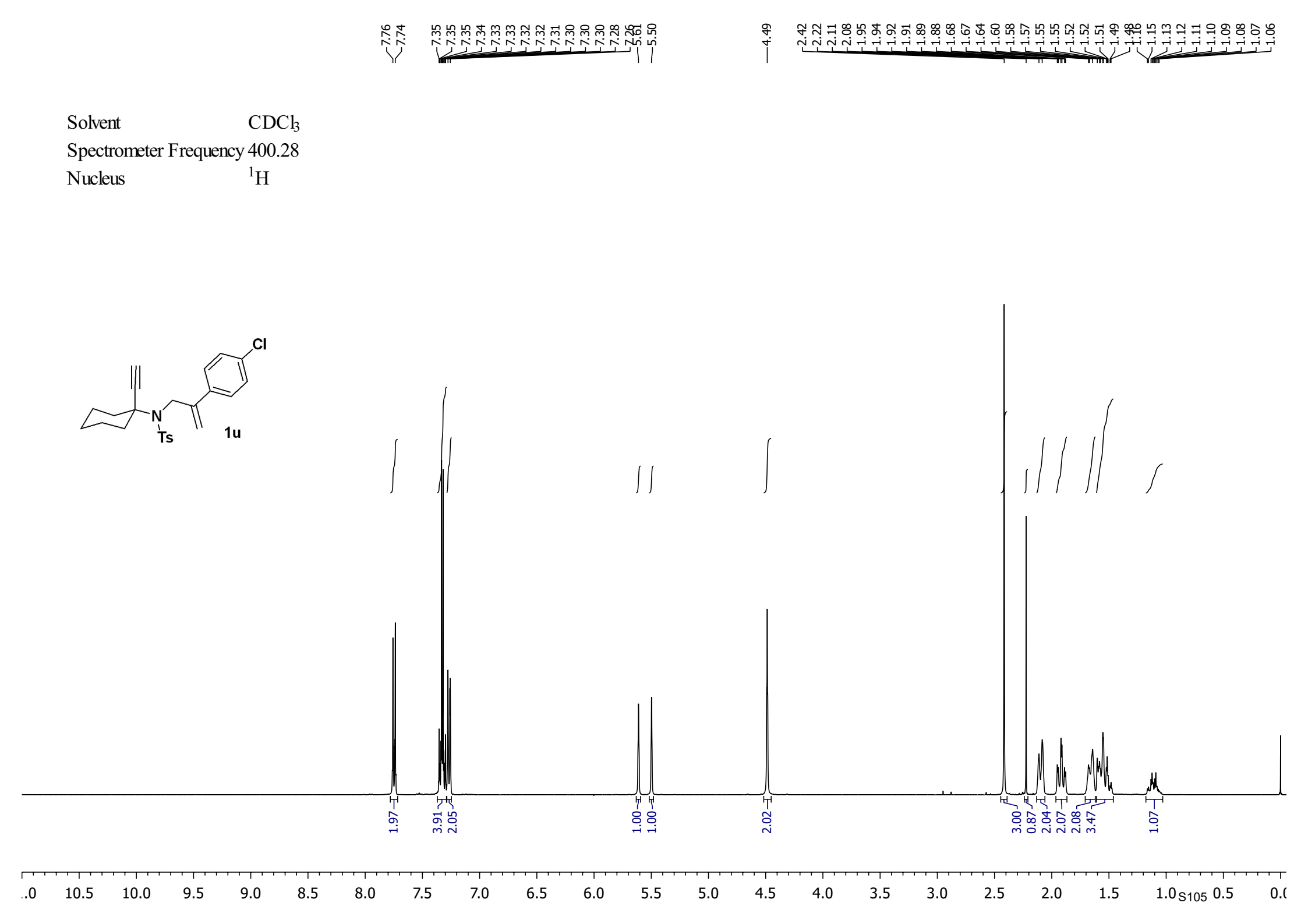
7.41
7.40
7.35
7.35
7.33
7.33
7.32
7.26
7.22
7.20
7.17
7.15
5.55
5.54
4.54
2.59
2.32
2.26
2.23
2.17
2.16
2.08
2.07
2.05
2.04
2.01
2.01
1.75
1.72
1.68
1.63
1.60
1.56
1.54
1.53
Solvent CDCl3
Spectrometer Frequency 400.28
Nucleus 1H
O
1t
Ph
N
Ts
DMF
DMF
1.91
1.92
1.90
2.92
1.94
1.90
0.97
0.97
1.94
2.87
2.94
2.07
2.17
2.33
2.31
2.02
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

197.22
145.41
143.10
139.73
138.65
136.15
131.61
129.35
128.40
127.92
127.75
126.08
114.24
92.22
86.02
77.31
76.99
76.67
63.12
51.65
38.04
26.59
24.73
23.76
21.42

Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C






7.76
7.74
7.35
7.35
7.35
7.34
7.33
7.33
7.32
7.32
7.31
7.30
7.30
7.30
7.28
7.26
5.61
5.50
4.49
2.42
2.22
2.11
2.08
1.95
1.94
1.92
1.91
1.89
1.88
1.68
1.67
1.64
1.60
1.58
1.57
1.55
1.55
1.52
1.52
1.51
1.49
1.15
1.13
1.12
1.11
1.10
1.09
1.08
1.07
1.06
Solvent CDCl3
Spectrometer Frequency 400.28
Nucleus 1H
Cl
N
Ts
1u
1.97
3.91
2.05
1.00
1.00
2.02
3.00
0.87
2.04
2.07
2.08
3.47
1.07
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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2.5
2.0
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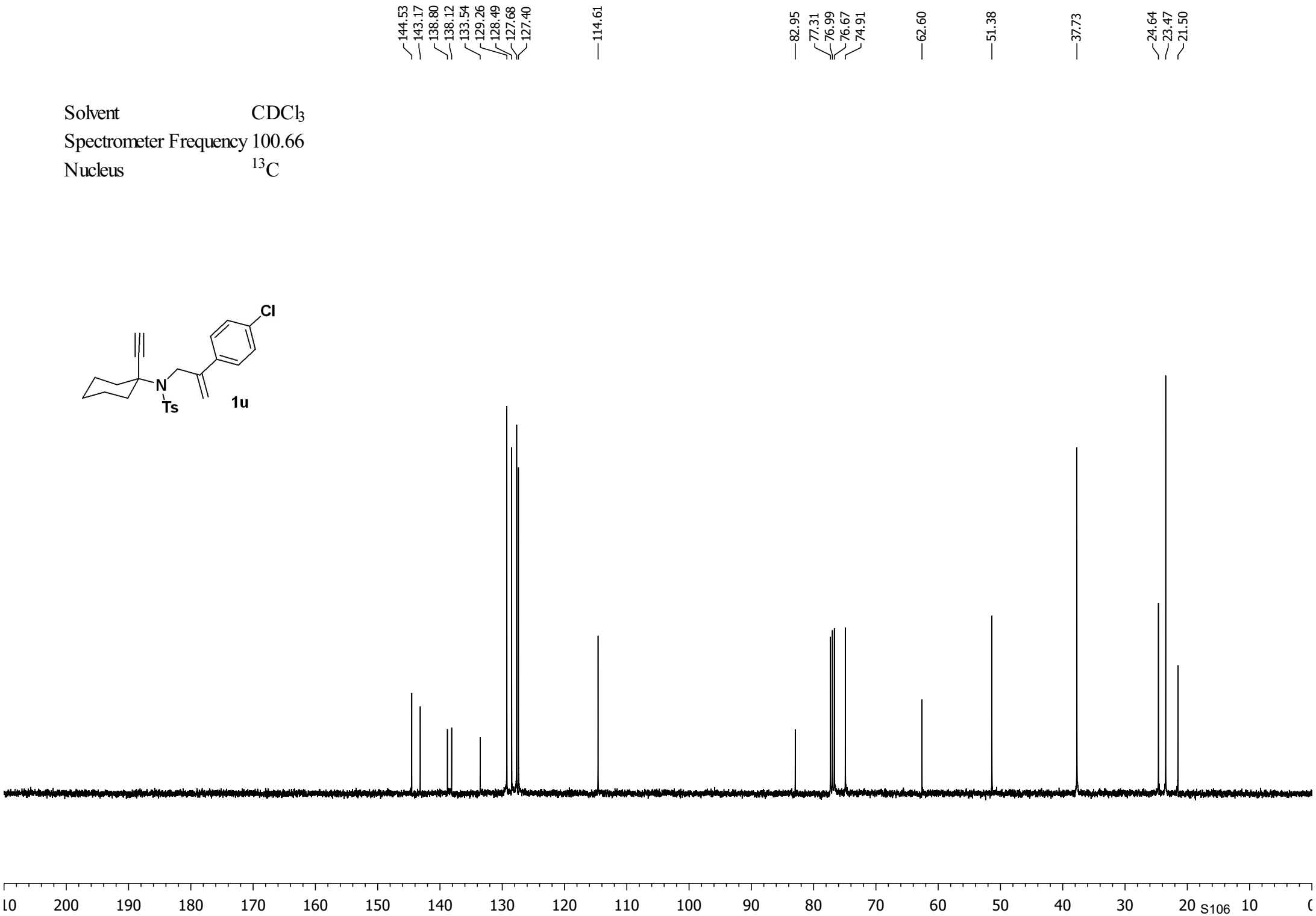
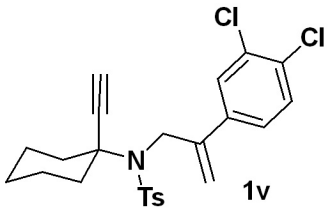
0.5


144.53
143.17
138.80
138.12
133.54
129.26
128.49
127.68
127.40
114.61
82.95
77.31
76.99
76.67
74.91
62.60
51.38
37.73
24.64
23.47
21.50
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
Cl
N
Ts
1u
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H

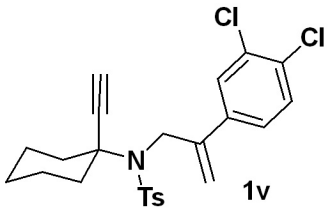


1v

Solvent $CDCl_3$

Spectrometer Frequency 100.66

Nucleus ^{13}C



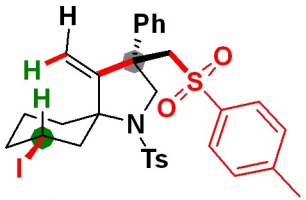
1v

143.61
143.26
139.67
138.73
132.43
131.63
130.26
129.30
127.95
127.66
125.48
115.65
82.86
77.31
76.99
76.68
75.07
62.69
51.21
37.69
24.61
23.47
21.51

Solvent $CDCl_3$

Spectrometer Frequency 400.28

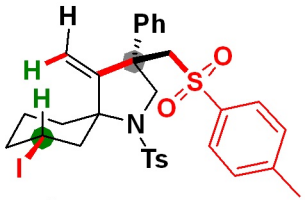
Nucleus ^1H



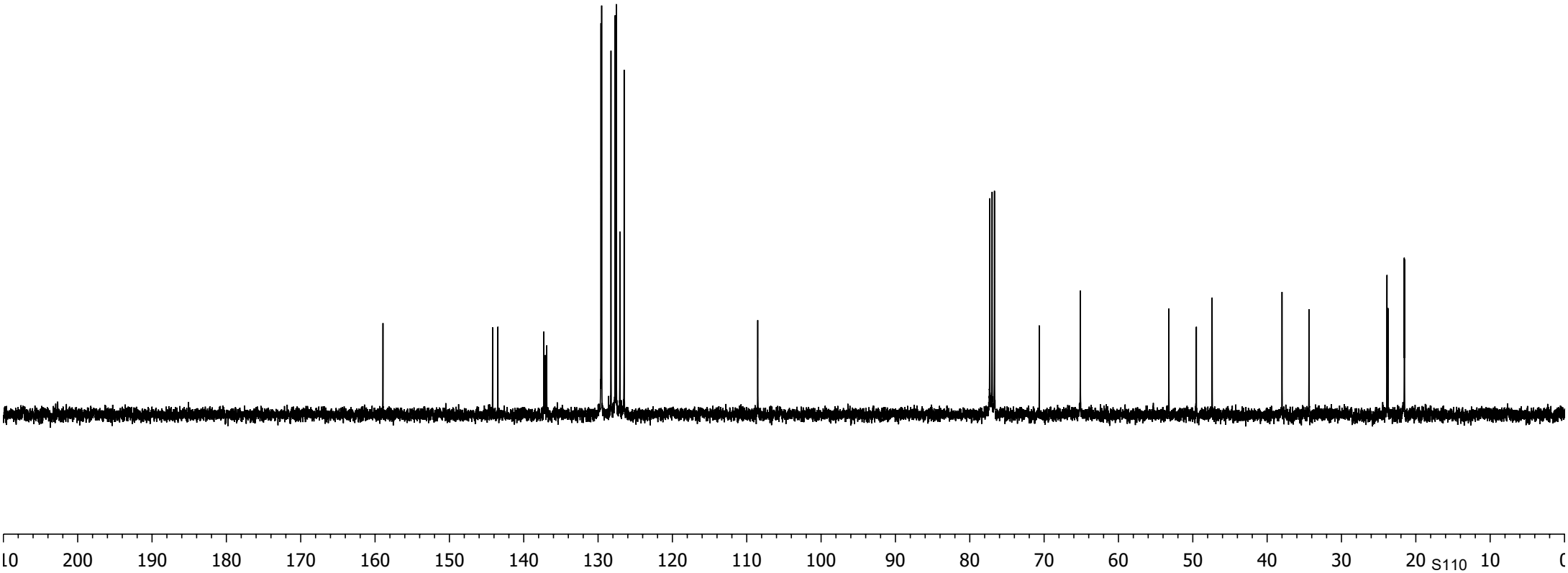
3aa, >20:1 dr

158.93
144.19
143.48
137.30
137.08
136.91
129.61
129.52
128.26
127.71
127.55
127.07
126.50
108.52
77.31
76.99
76.68
70.66
65.13
53.22
49.52
47.40
38.00
34.37
23.88
23.75
21.55
21.52

Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C



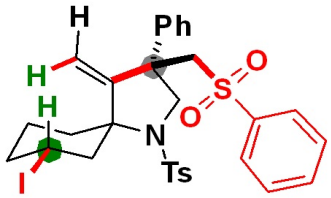
3aa, >20:1 dr



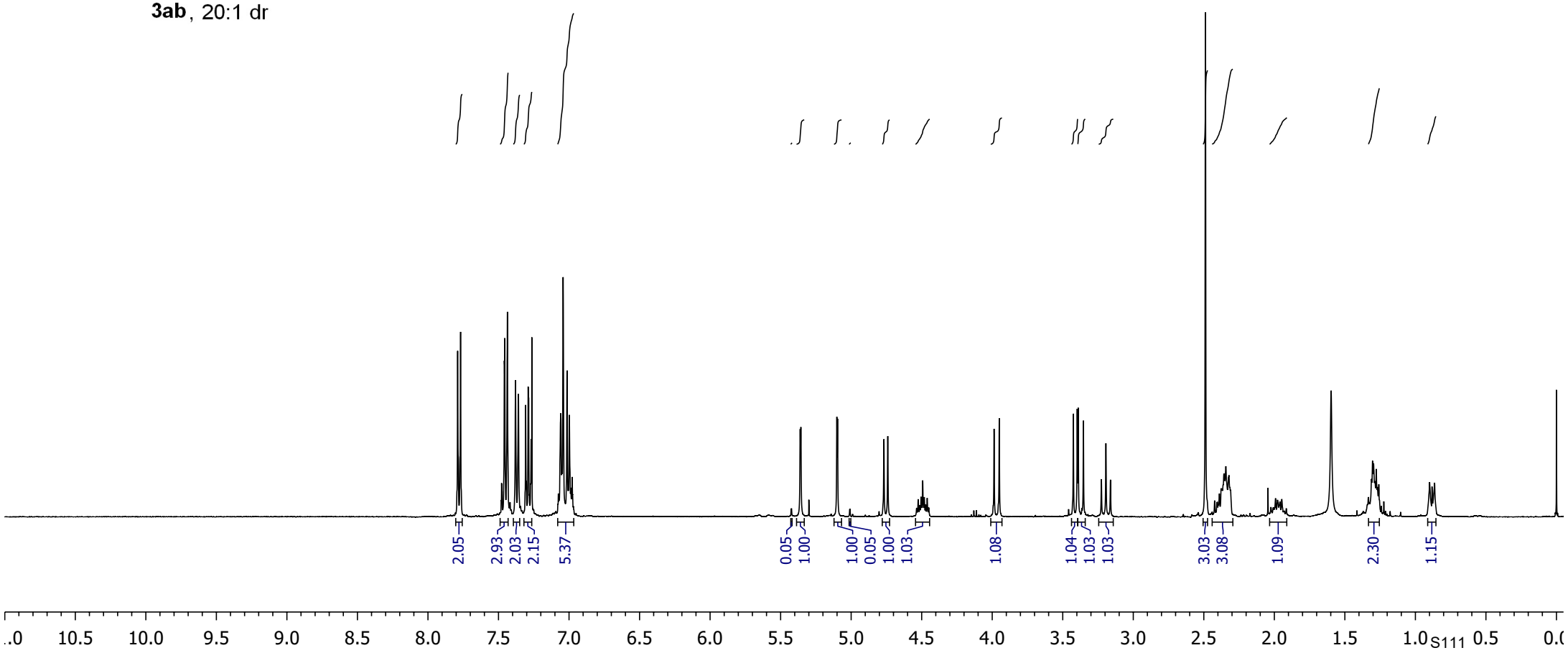
Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



3ab, 20:1 dr

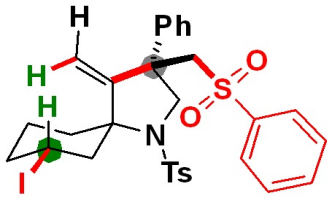


158.94
143.51
140.10
137.09
136.94
133.18
129.65
128.91
128.33
127.73
127.50
127.34
126.51
108.52
77.31
77.00
76.68
70.69
65.13
53.29
49.54
47.41
38.01
34.41
23.83
23.77
21.56

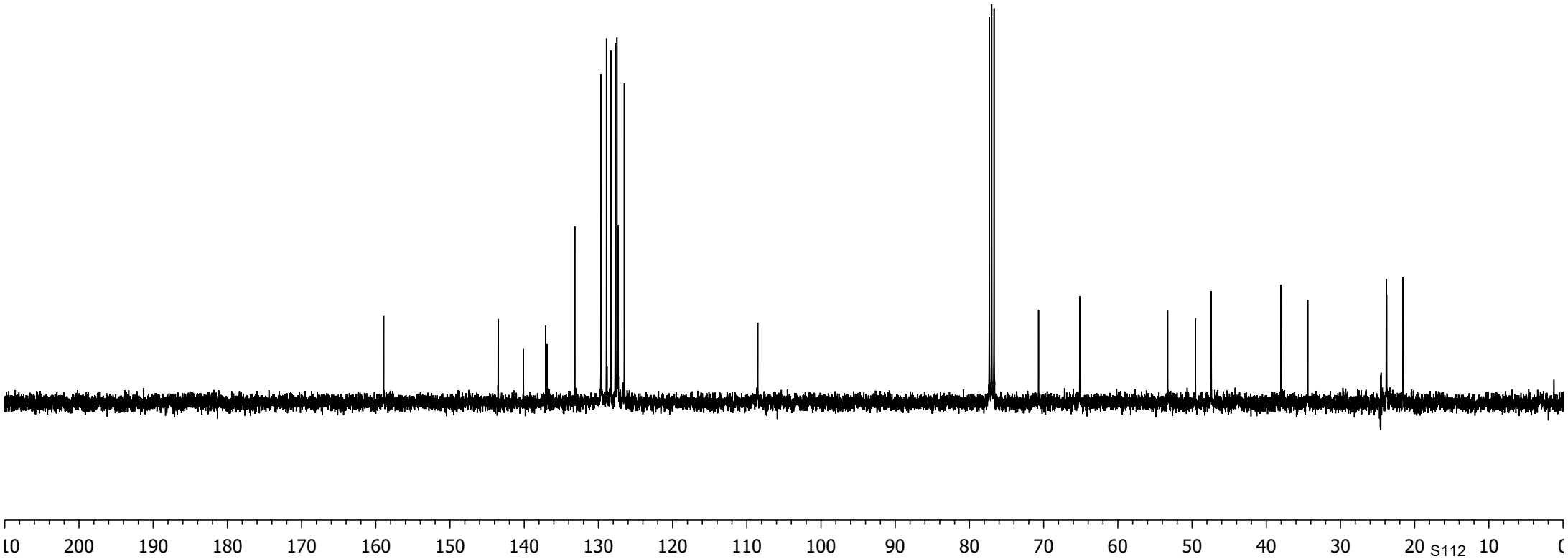
Solvent $CDCl_3$

Spectrometer Frequency 100.66

Nucleus ^{13}C



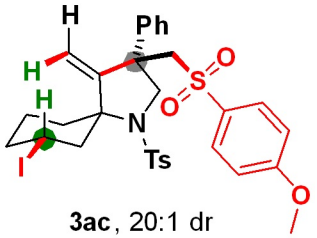
3ab, 20:1 dr



Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



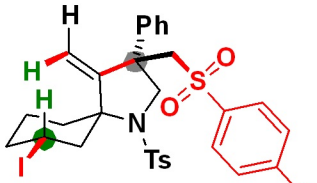
3ac, 20:1 dr

163.22 158.95 143.46 137.30 136.86 131.52 129.69 129.59 128.27 127.67 127.13 126.48 114.09 108.48 77.31 76.99 76.68 70.65 65.22 55.61 53.21 49.46 47.37 37.98 34.39 23.93 23.72 21.53

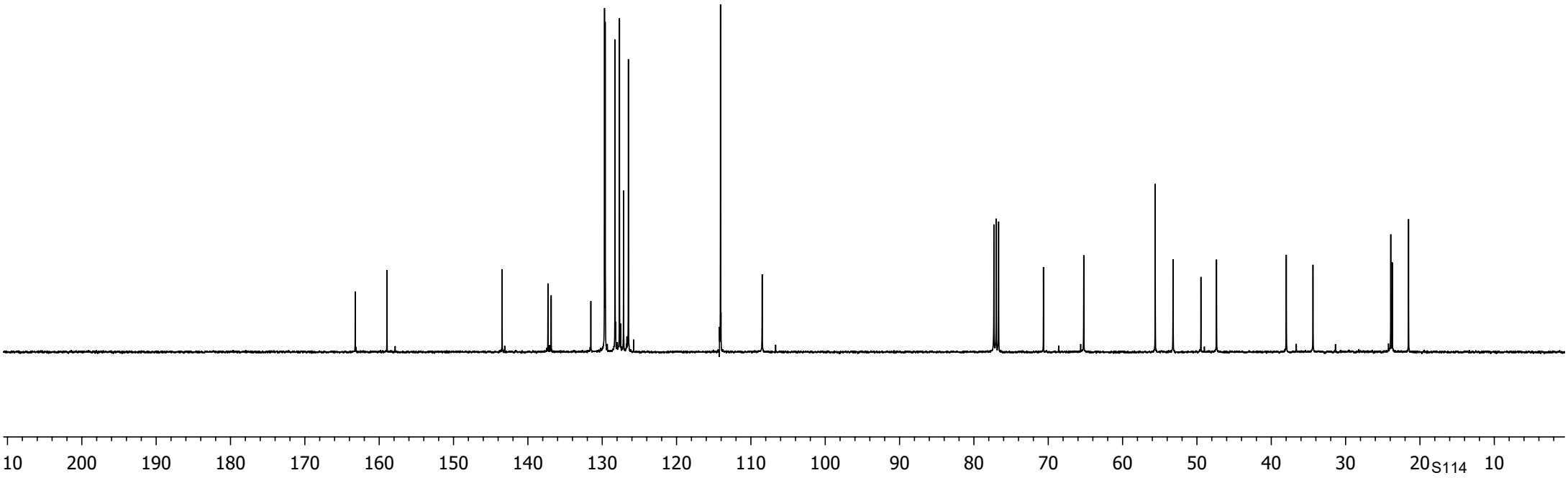
Solvent $CDCl_3$

Spectrometer Frequency 100.66

Nucleus ^{13}C



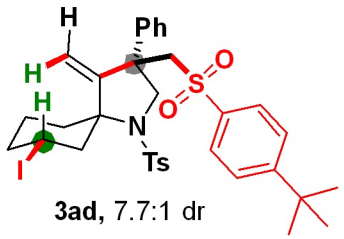
3ac, 20:1 dr



Solvent $CDCl_3$

Spectrometer Frequency 400.39

Nucleus ^1H



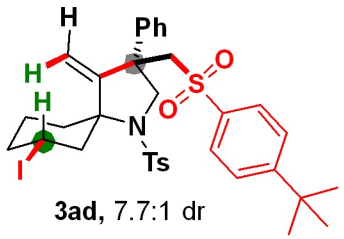
3ad, 7.7:1 dr

159.25
156.90
143.49
137.10
136.94
136.82
129.65
128.23
127.74
127.58
127.34
127.21
126.51
125.88
108.30
77.31
76.99
76.67
70.71
65.10
53.24
49.40
47.40
38.02
35.07
34.49
30.95
23.87
23.78
21.56

Solvent $CDCl_3$

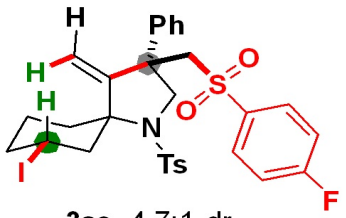
Spectrometer Frequency 100.69

Nucleus ^{13}C

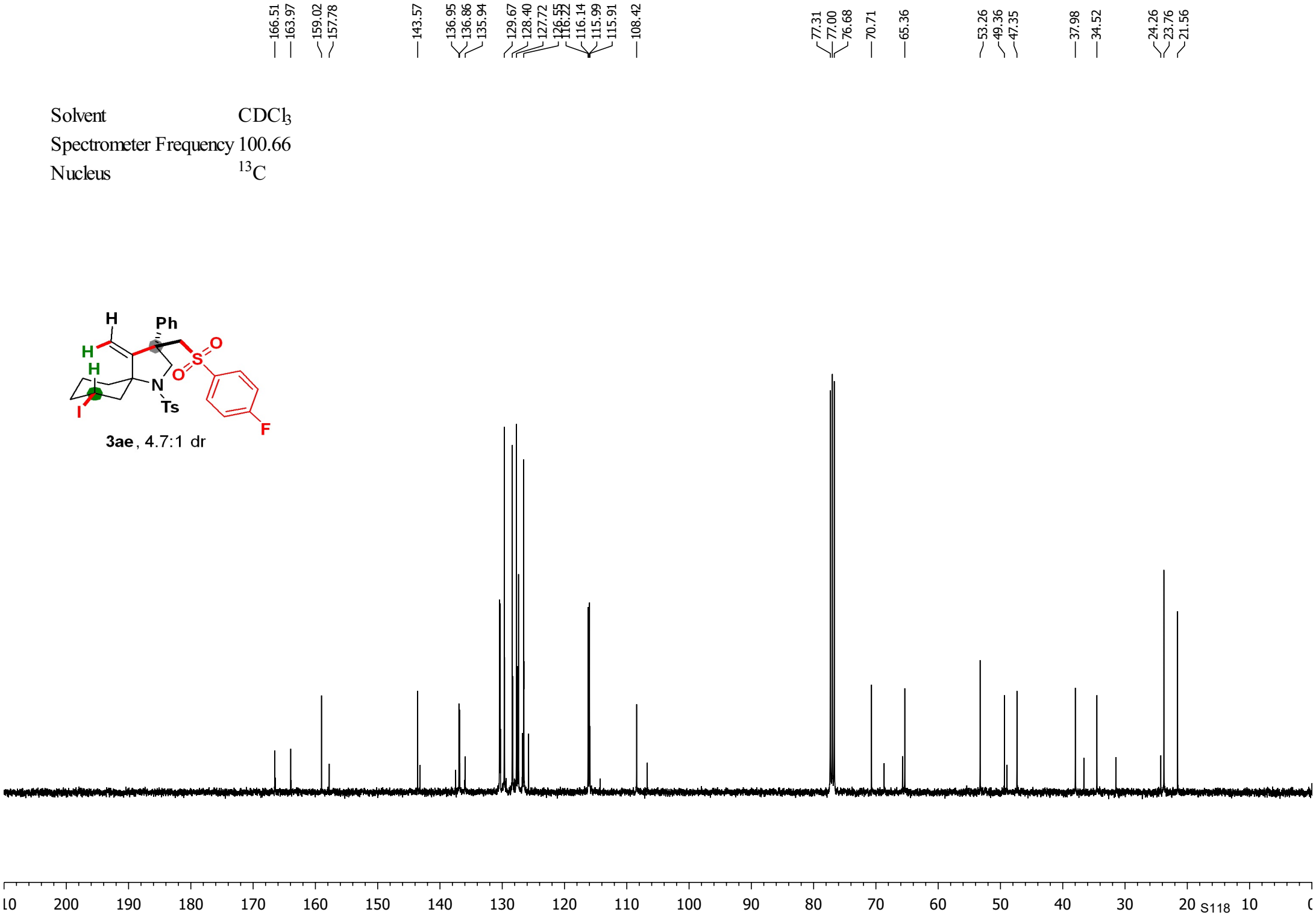


3ad, 7.7:1 dr

Solvent	$CDCl_3$
Spectrometer Frequency	400.28
Nucleus	^1H

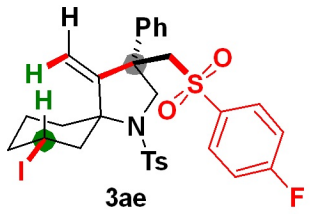


3ae, 4.7:1 dr

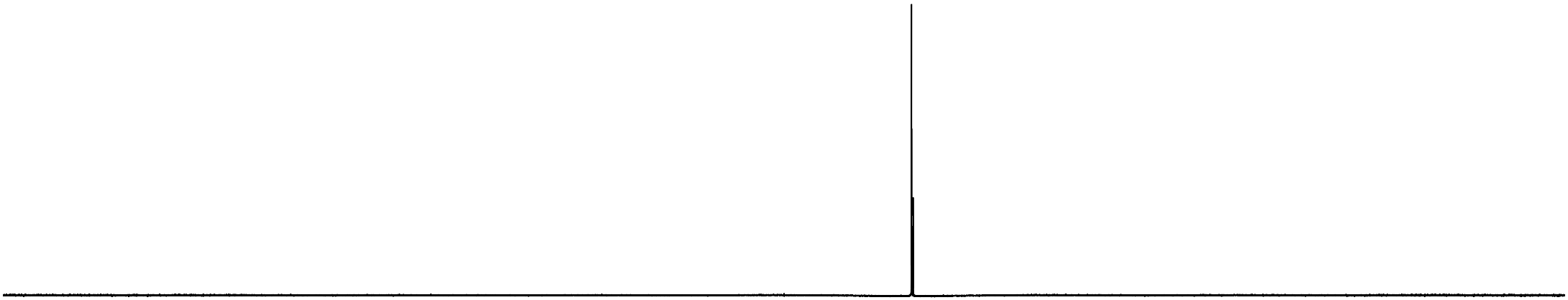

166.51
163.97
159.02
157.78
143.57
136.95
136.86
135.94
129.67
128.40
127.72
116.14
115.99
115.91
108.42
77.31
77.00
76.68
70.71
65.36
53.26
49.36
47.35
37.98
34.52
24.26
23.76
21.56
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
H
Ph
H
H
O
S
O
N
Ts
I
F
3ae, 4.7:1 dr
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

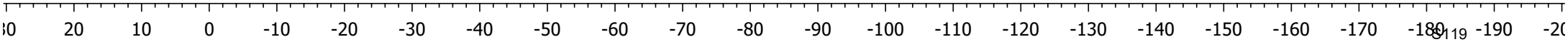
-103.84

Solvent	$CDCl_3$
Spectrometer Frequency	376.61
Nucleus	^{19}F



3ae

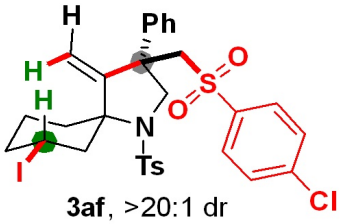




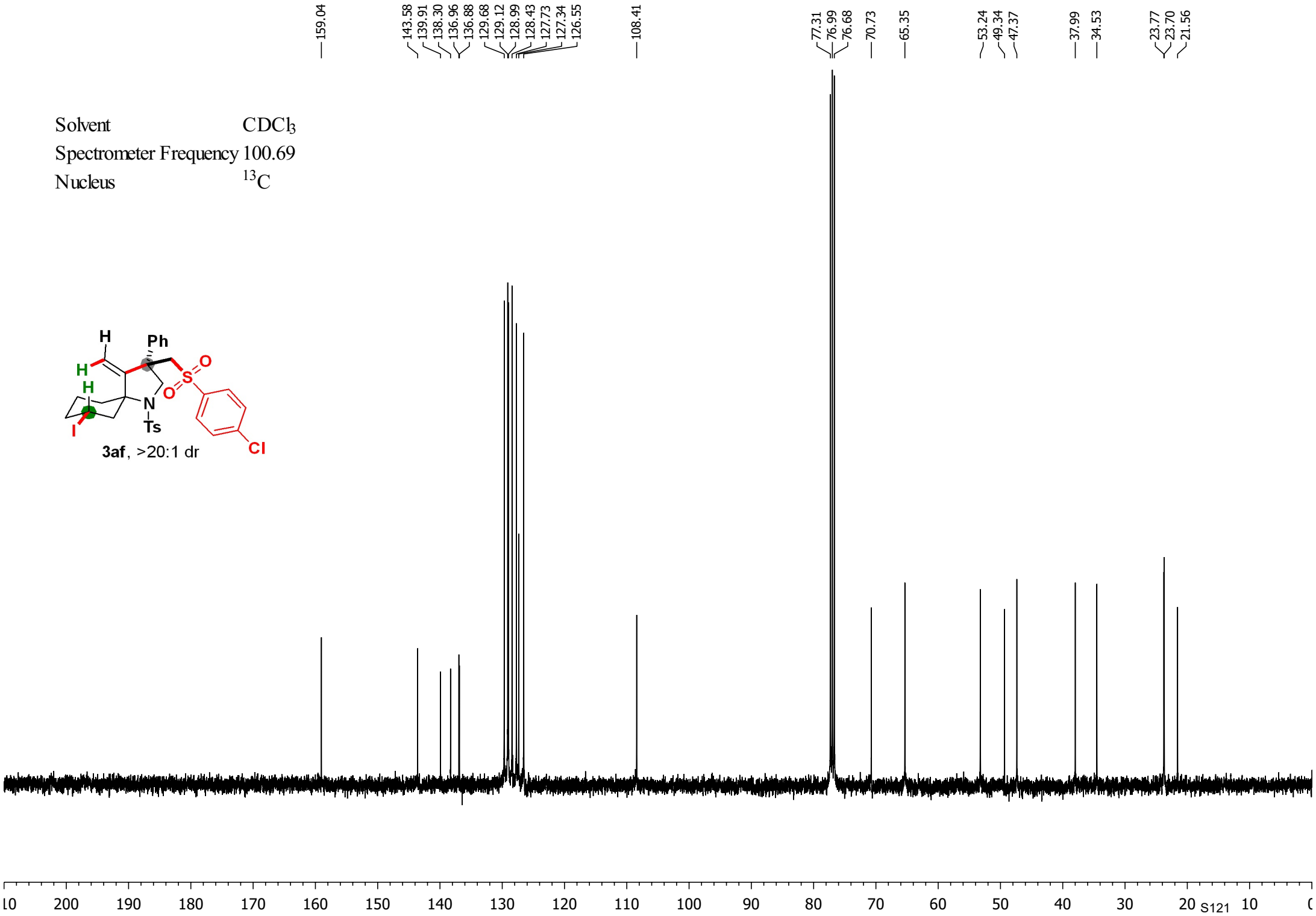
Solvent $CDCl_3$

Spectrometer Frequency 400.39

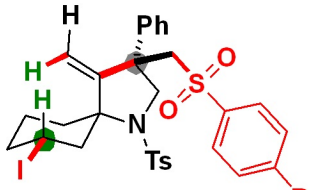
Nucleus ^1H



3af, >20:1 dr


159.04
143.58
139.91
138.30
136.96
136.88
129.68
129.12
128.99
128.43
127.73
127.34
126.55
108.41
77.31
76.99
76.68
70.73
65.35
53.24
49.34
47.37
37.99
34.53
23.77
23.70
21.56
Solvent CDCl3
Spectrometer Frequency 100.69
Nucleus 13C
H
Ph
H
H
O
S
O
N
Ts
I
Cl
3af, >20:1 dr
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

Solvent	$CDCl_3$
Spectrometer Frequency	400.28
Nucleus	^1H



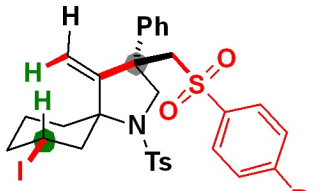
3ag,>20:1 dr

158.34
144.32
138.90
137.18
137.09
132.31
129.58
129.15
128.38
127.68
127.53
127.27
126.43
108.95
77.32
77.00
76.68
70.90
65.05
53.29
49.73
47.62
37.95
34.27
23.71
23.40
21.53

Solvent $CDCl_3$

Spectrometer Frequency 100.66

Nucleus ^{13}C

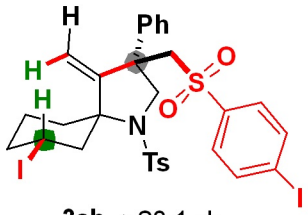


3ag,>20:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 400.28

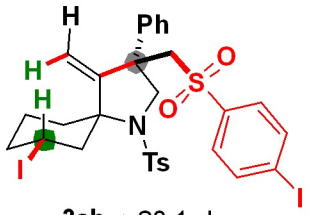
Nucleus ^1H



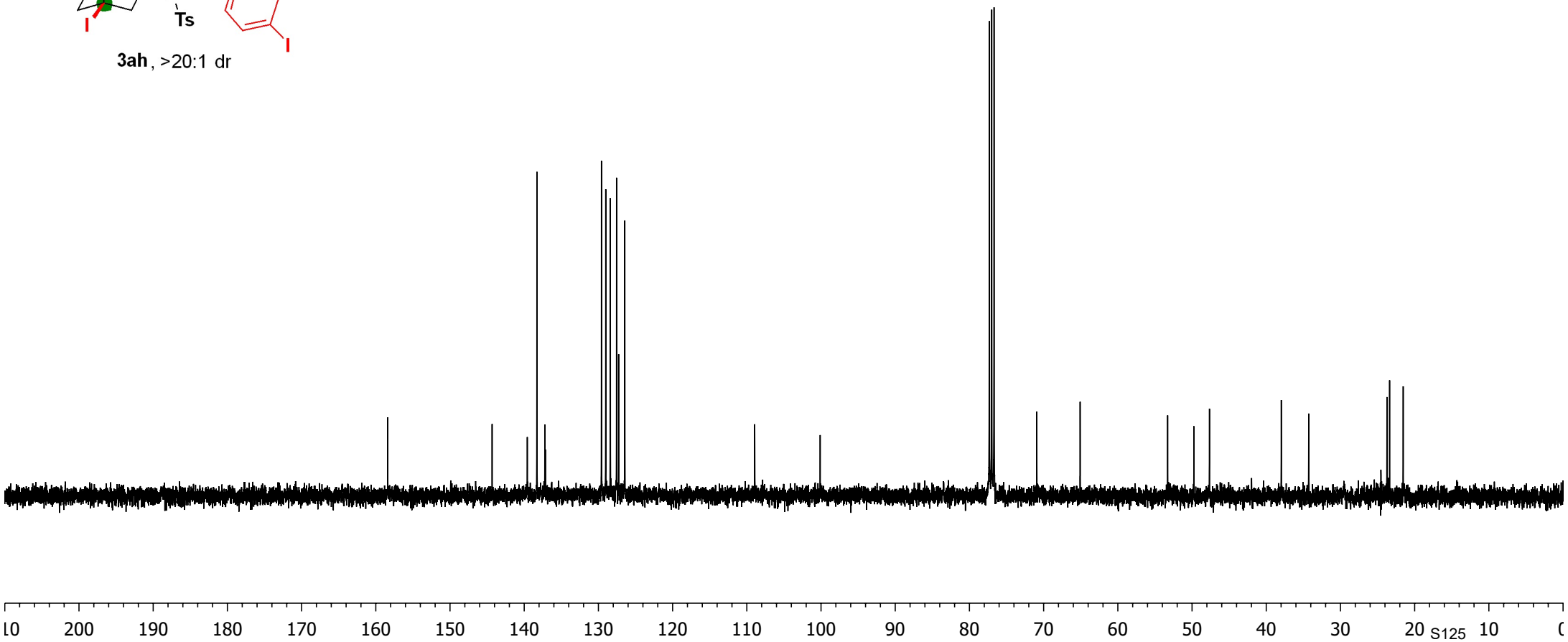
3ah, >20:1 dr

158.40
144.32
139.58
138.29
137.22
137.15
129.59
129.01
128.39
127.55
127.28
126.44
108.94
100.11
77.31
77.00
76.68
70.92
65.08
53.29
49.75
47.65
37.97
34.28
23.73
23.38
21.54

Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C



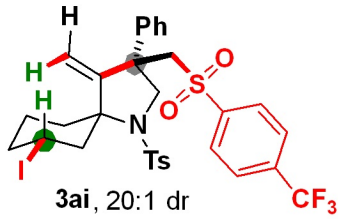
3ah, >20:1 dr



Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



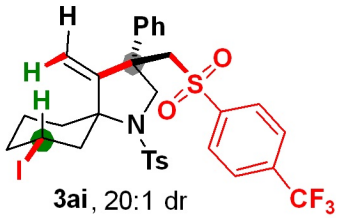
3ai, 20:1 dr

159.07
143.65
143.10
136.83
136.64
134.75
134.42
129.72
128.43
128.12
127.74
127.57
126.53
125.89
125.85
108.32
77.31
77.00
76.68
70.76
65.25
53.19
49.17
47.32
37.97
34.61
23.76
23.64
21.57

Solvent $CDCl_3$

Spectrometer Frequency 100.66

Nucleus ^{13}C



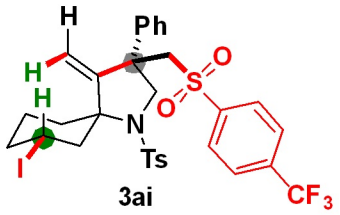
3ai, 20:1 dr

-63.46

Solvent $CDCl_3$

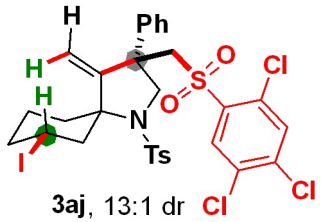
Spectrometer Frequency 376.61

Nucleus ^{19}F



3ai

Solvent	$CDCl_3$
Spectrometer Frequency	400.28
Nucleus	^1H



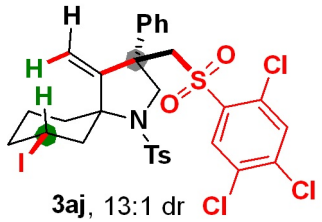
3aj, 13:1 dr

158.55
143.66
138.48
136.79
136.16
135.95
132.79
132.18
132.08
130.09
129.74
128.23
127.70
127.62
126.31
108.25
77.32
77.00
76.68
70.85
62.48
53.37
49.04
47.23
37.97
34.74
23.73
21.57
6.65

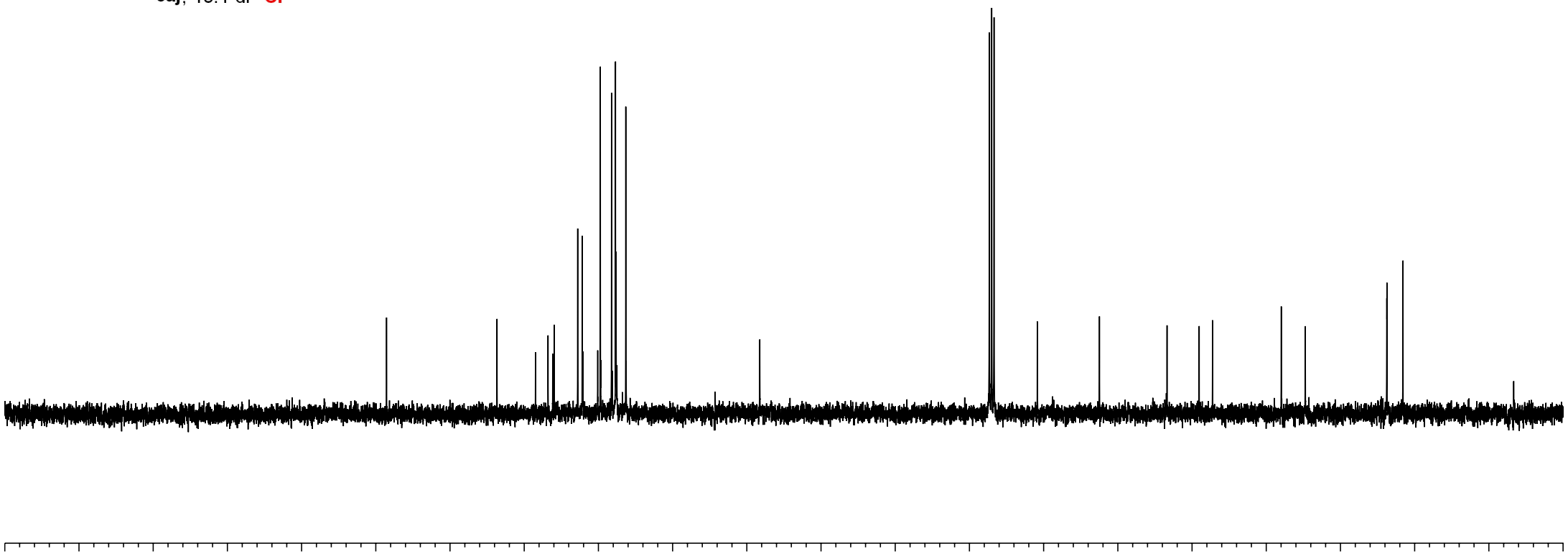
Solvent $CDCl_3$

Spectrometer Frequency 100.66

Nucleus ^{13}C



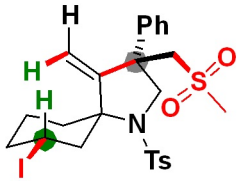
3aj, 13:1 dr



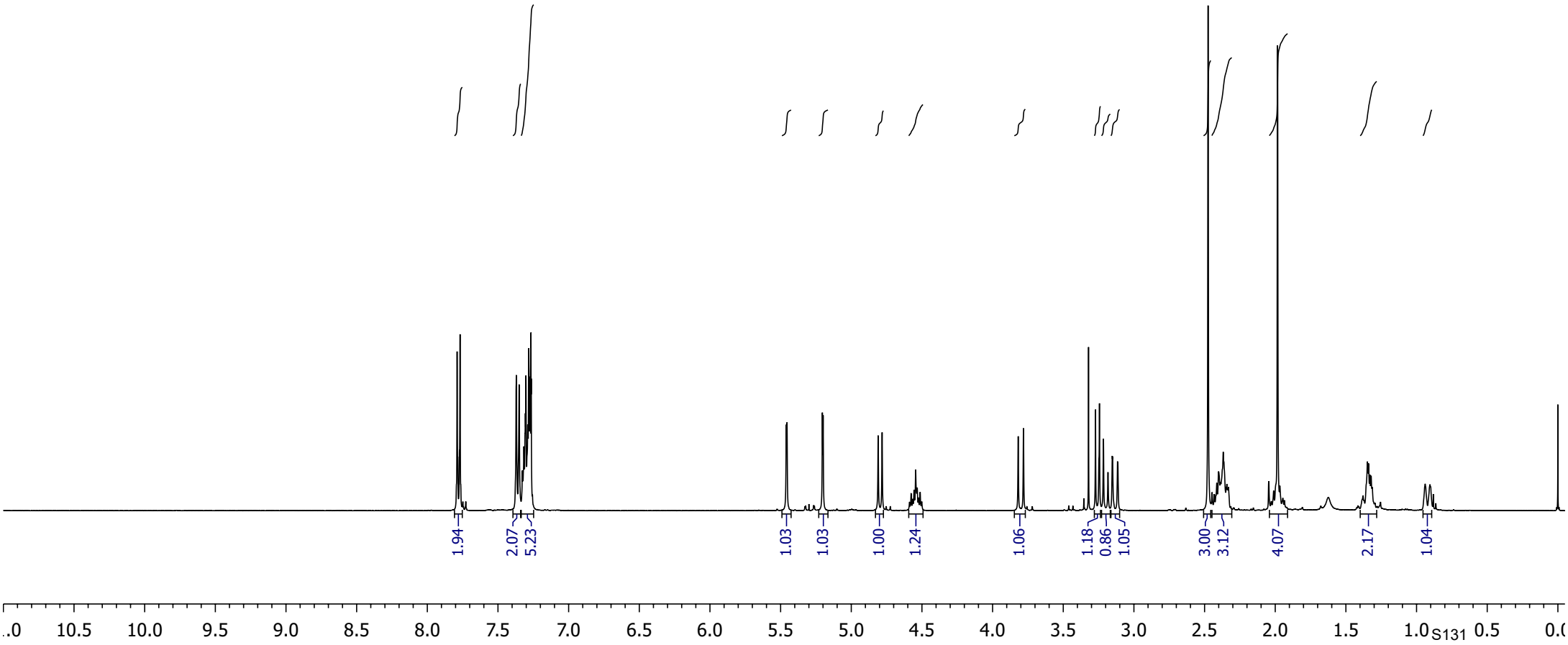
Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



3ak, 17.2:1 dr

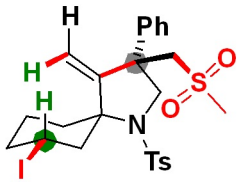


158.60
143.53
137.27
136.86
129.65
129.06
128.10
127.70
126.63
108.63
77.31
76.99
76.67
70.76
64.62
52.80
49.30
47.42
42.34
38.03
34.51
23.79
21.52

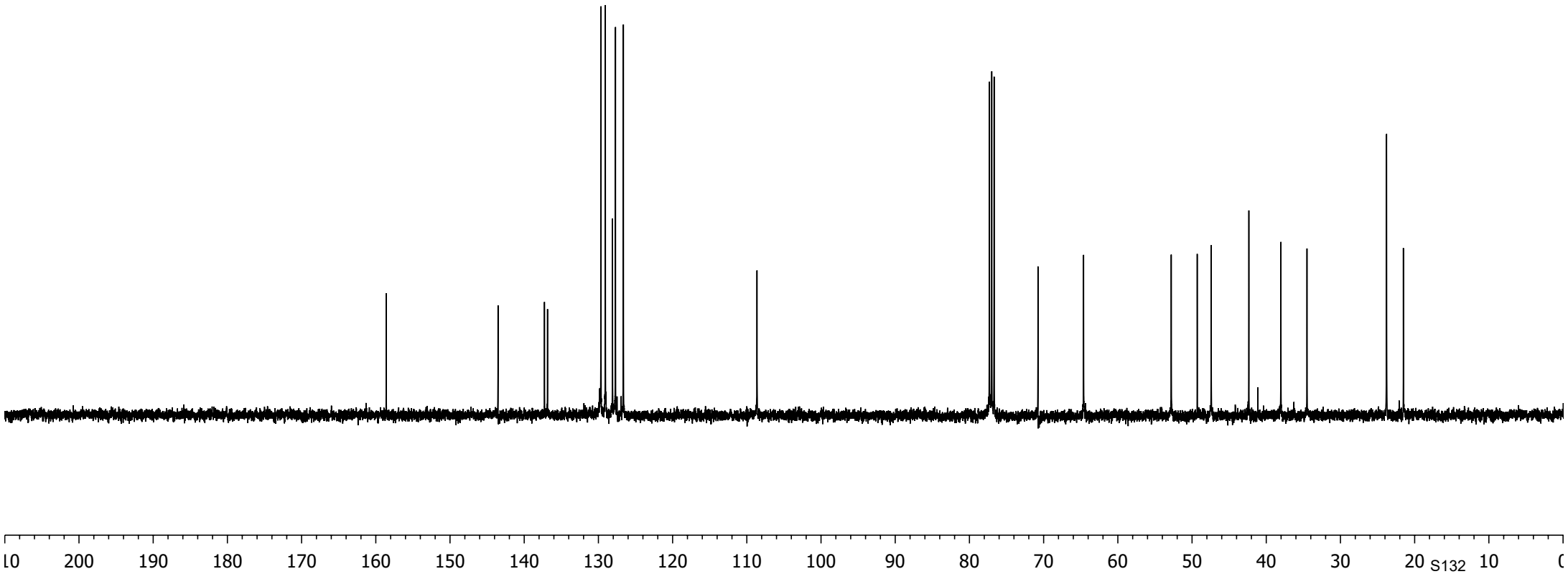
Solvent $CDCl_3$

Spectrometer Frequency 100.45

Nucleus ^{13}C



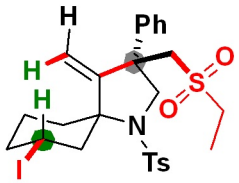
3ak, 17.2:1 dr



Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



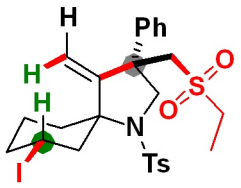
3al, 14.6:1 dr

158.18
144.39
138.01
137.30
129.64
128.49
127.56
127.38
126.65
109.75
77.31
76.99
76.67
70.00
64.89
53.53
50.19
47.98
47.87
37.93
33.82
23.72
23.44
21.52
7.98

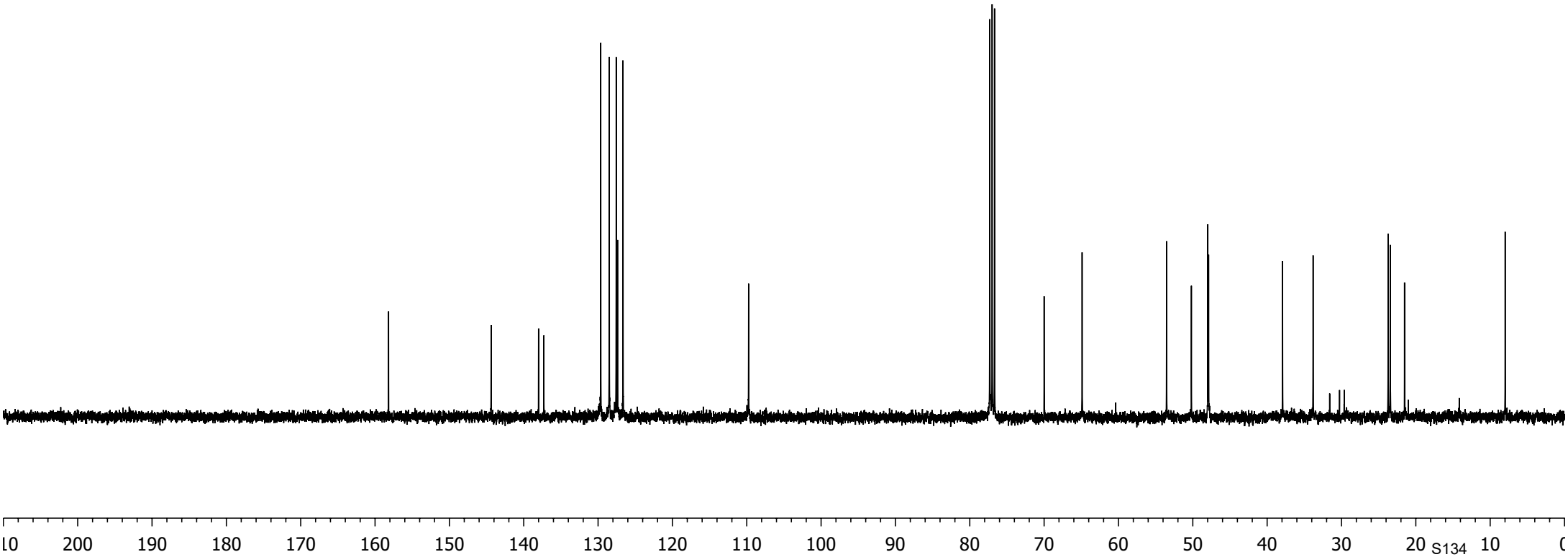
Solvent $CDCl_3$

Spectrometer Frequency 100.45

Nucleus ^{13}C



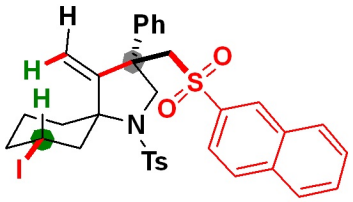
3al, 14.6:1 dr



Solvent $CDCl_3$

Spectrometer Frequency 400.39

Nucleus ^1H



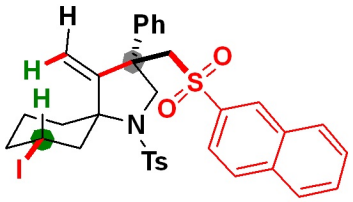
3am, 16.7:1 dr

159.00
143.47
136.88
131.75
129.80
129.62
129.42
129.24
129.19
128.07
127.70
127.41
127.18
126.38
121.88
108.41
77.31
76.99
76.68
70.70
64.99
53.25
49.46
47.38
37.99
34.45
23.87
23.74
21.55

Solvent $CDCl_3$

Spectrometer Frequency 100.69

Nucleus ^{13}C

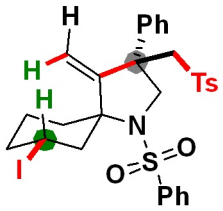


3am, 16.7:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



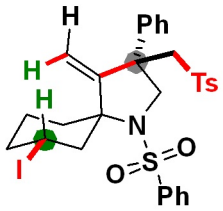
3ba, >20:1 dr

158.68
144.19
139.85
137.26
137.11
132.64
129.51
129.03
128.29
127.62
127.52
127.09
126.45
108.71
77.31
76.99
76.68
70.73
65.08
53.25
49.58
47.51
37.99
34.27
23.72
21.50

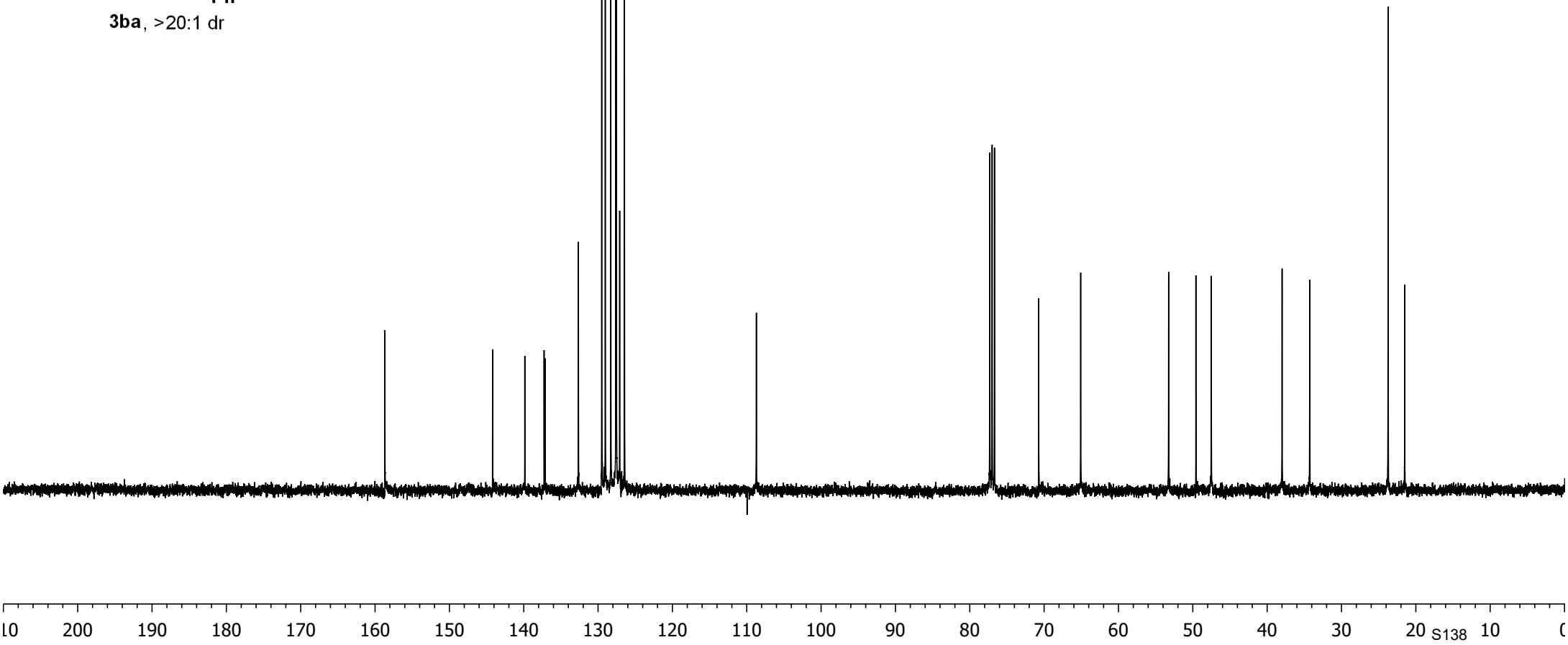
Solvent $CDCl_3$

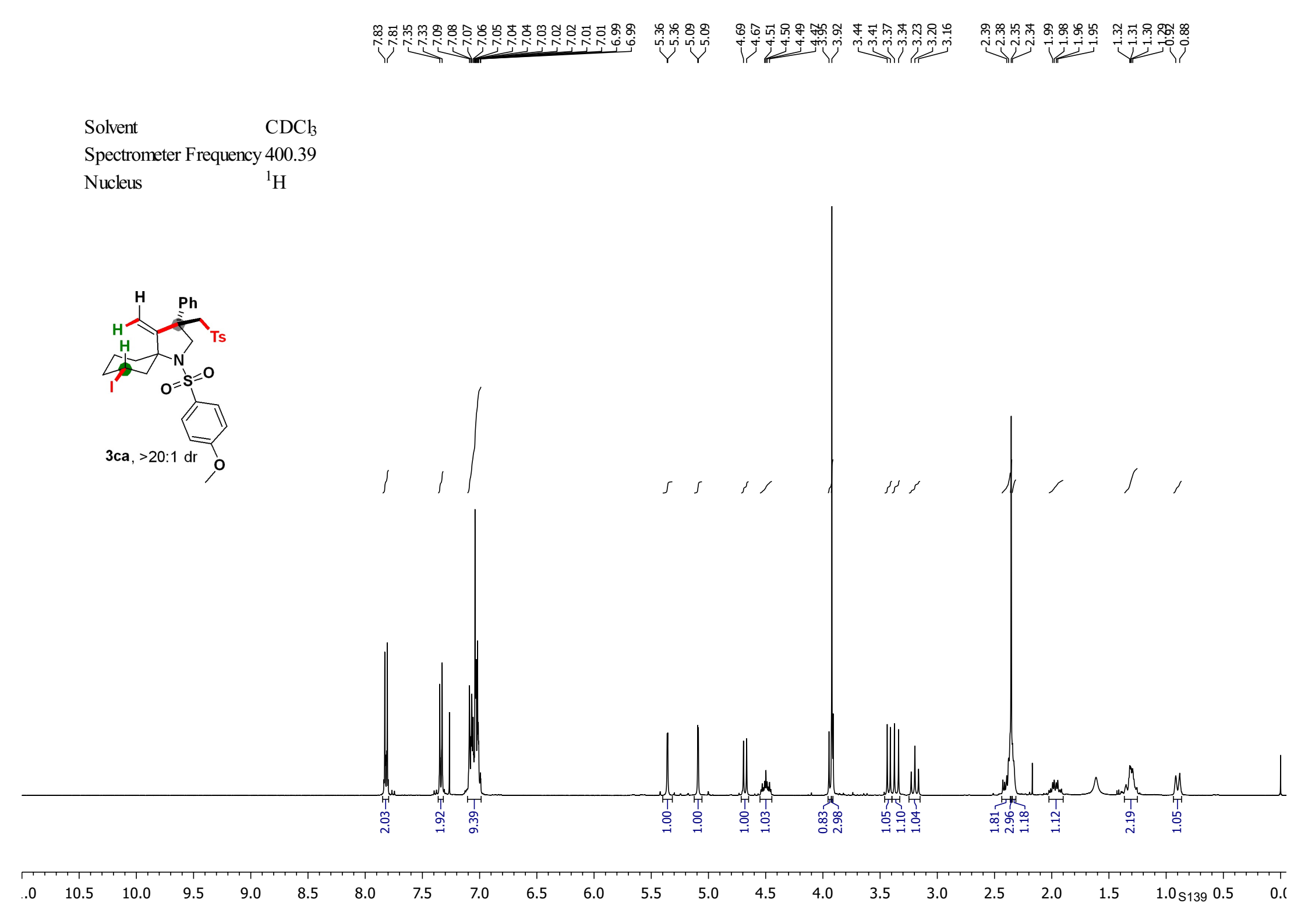
Spectrometer Frequency 100.45

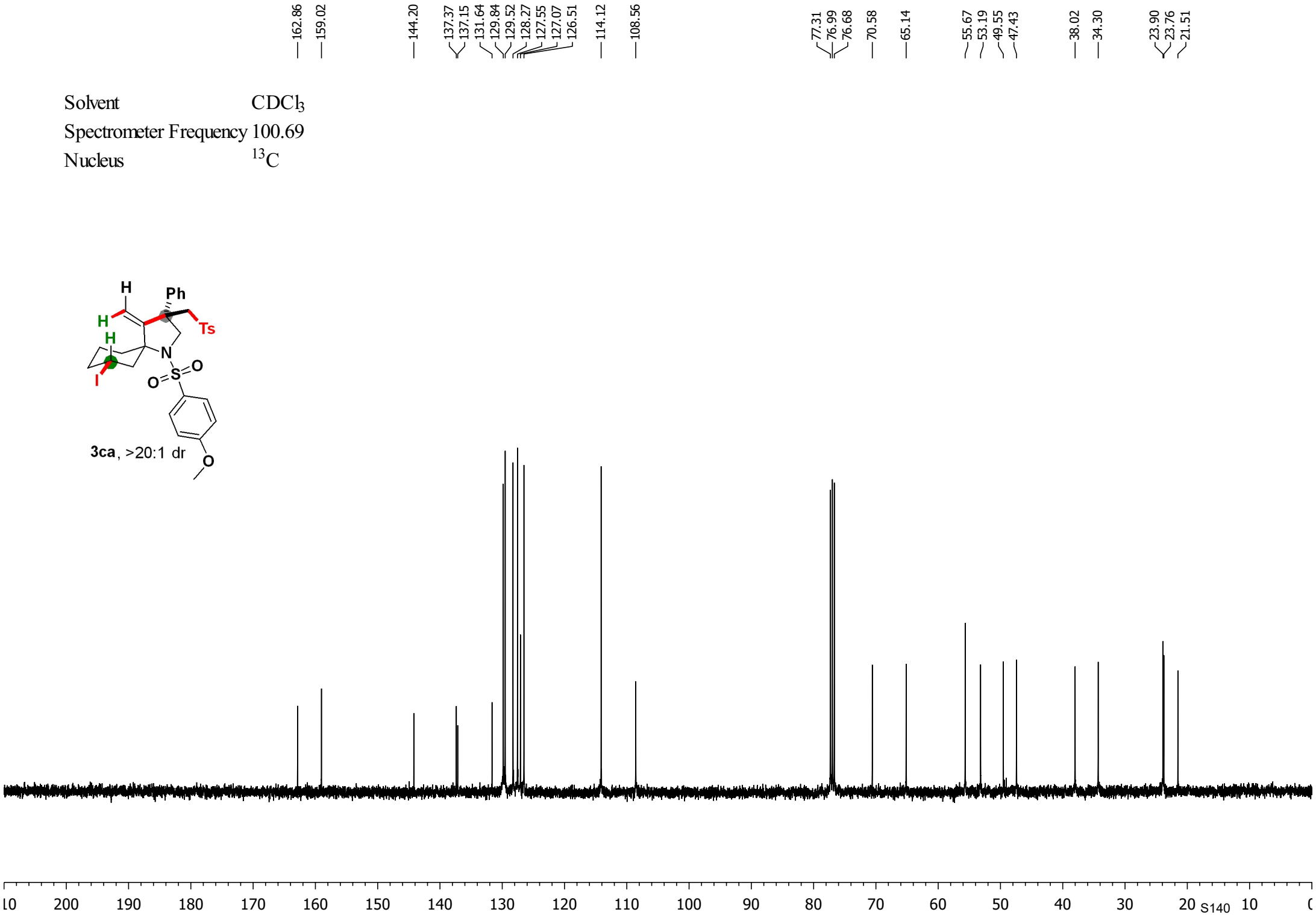
Nucleus ^{13}C



3ba, >20:1 dr



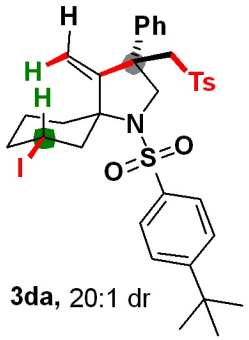

7.83 7.81 7.35 7.33 7.09 7.08 7.07 7.06 7.05 7.04 7.04 7.03 7.02 7.02 7.01 7.01 6.99 6.99
5.36 5.36 5.09 5.09
4.69 4.67 4.51 4.50 4.49 3.92
3.44 3.41 3.37 3.34 3.23 3.20 3.16
2.39 2.38 2.35 2.34
1.99 1.98 1.96 1.95
1.32 1.31 1.30
0.92 0.88
Solvent CDCl3
Spectrometer Frequency 400.39
Nucleus 1H
H
Ph
H
H
Ts
N
O
O
S
O
I
3ca, >20:1 dr
O
2.03 1.92 9.39 1.00 1.00 1.00 1.03 0.83 2.98 1.05 1.10 1.04 1.81 2.96 1.18 1.12 2.19 1.05
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
S139


162.86
159.02
144.20
137.37
137.15
131.64
129.84
129.52
128.27
127.55
127.07
126.51
114.12
108.56
77.31
76.99
76.68
70.58
65.14
55.67
53.19
49.55
47.43
38.02
34.30
23.90
23.76
21.51
Solvent CDCl3
Spectrometer Frequency 100.69
Nucleus 13C
H
Ph
H
H
Ts
N
I
O
S
O
O
3ca, >20:1 dr
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

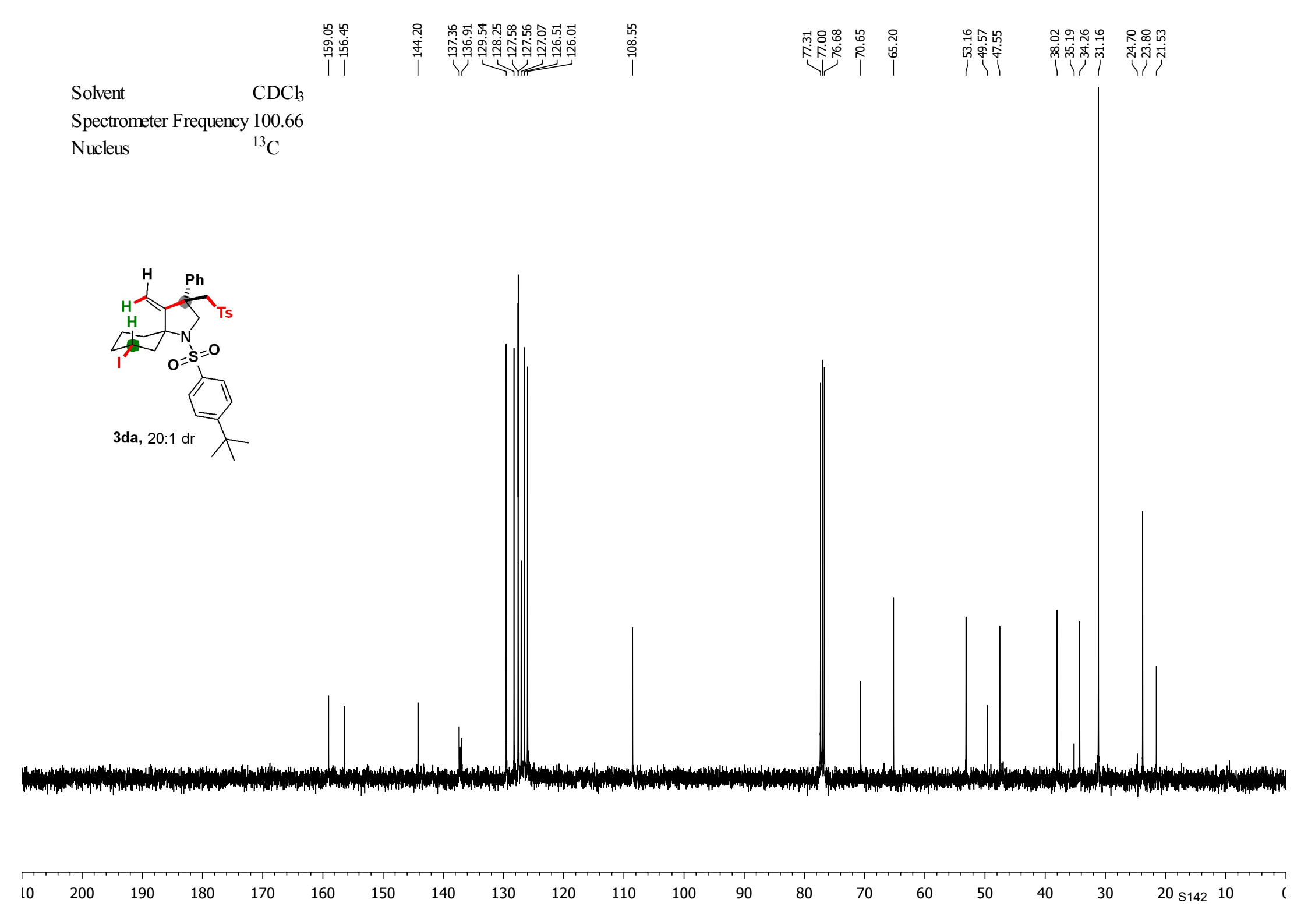
Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



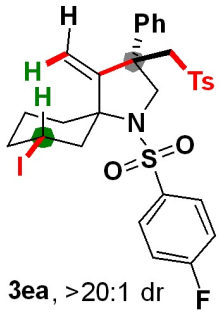
3da, 20:1 dr


159.05
156.45
144.20
137.36
136.91
129.54
128.25
127.58
127.56
127.07
126.51
126.01
108.55
77.31
77.00
76.68
70.65
65.20
53.16
49.57
47.55
38.02
35.19
34.26
31.16
24.70
23.80
21.53
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
H
Ph
H
H
Ts
N
S
O
O
I
3da, 20:1 dr
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

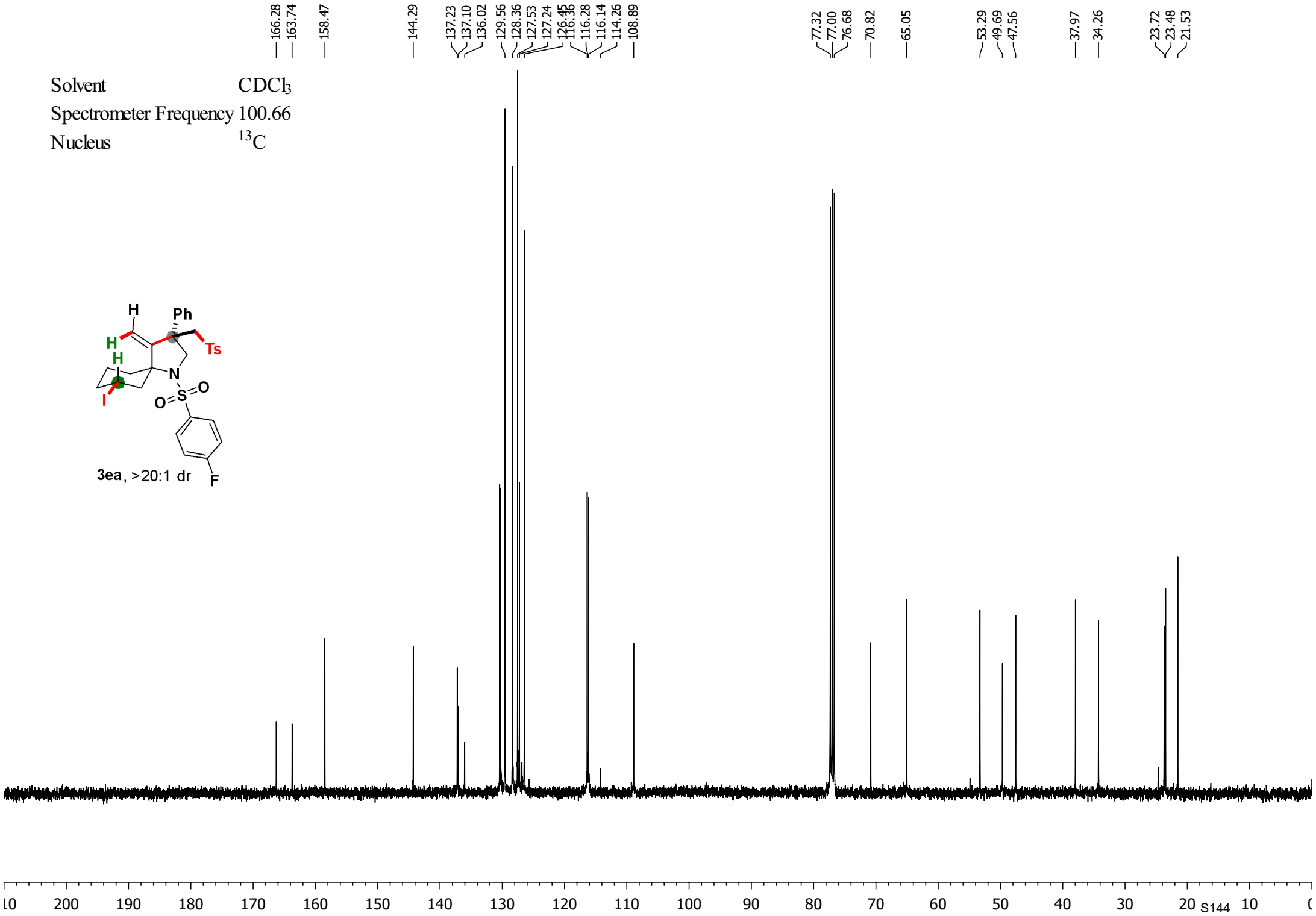
Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H

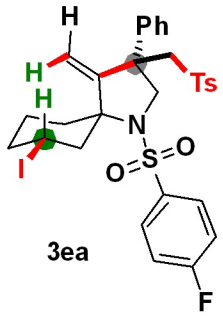


3ea, >20:1 dr

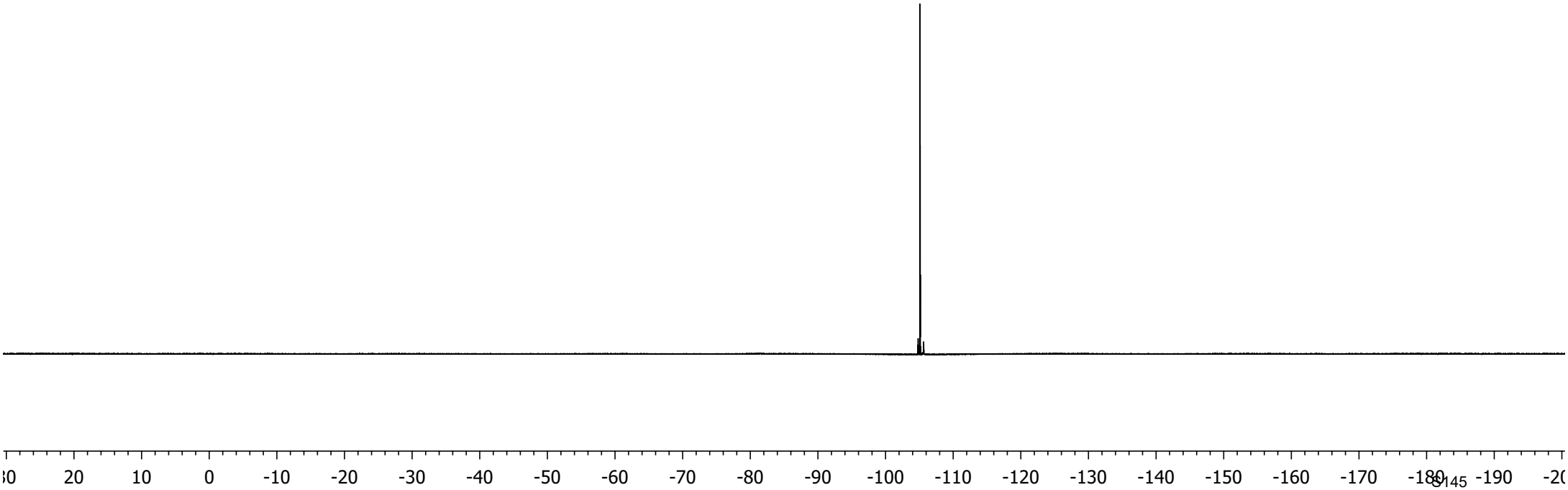

166.28
163.74
158.47
144.29
137.23
137.10
136.02
129.56
128.36
127.53
127.24
126.45
116.36
116.28
116.14
114.26
108.89
77.32
77.00
76.68
70.82
65.05
53.29
49.69
47.56
37.97
34.26
23.72
23.48
21.53
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
H
Ph
H
H
Ts
N
I
O
S
O
F
3ea, >20:1 dr
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

-105.10

Solvent	$CDCl_3$
Spectrometer Frequency	376.61
Nucleus	^{19}F



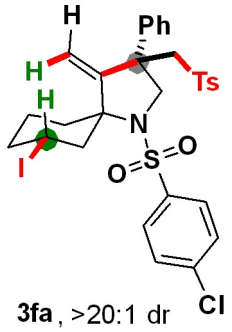
3ea



Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



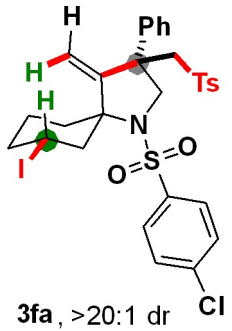
3fa, >20:1 dr

158.37
144.31
139.19
137.18
129.33
129.07
128.38
127.53
127.27
126.44
108.92
77.32
77.00
76.68
70.89
65.05
53.30
49.71
47.59
37.95
34.29
23.72
23.42
21.54

Solvent $CDCl_3$

Spectrometer Frequency 100.66

Nucleus ^{13}C

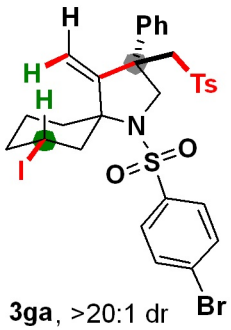


3fa, >20:1 dr

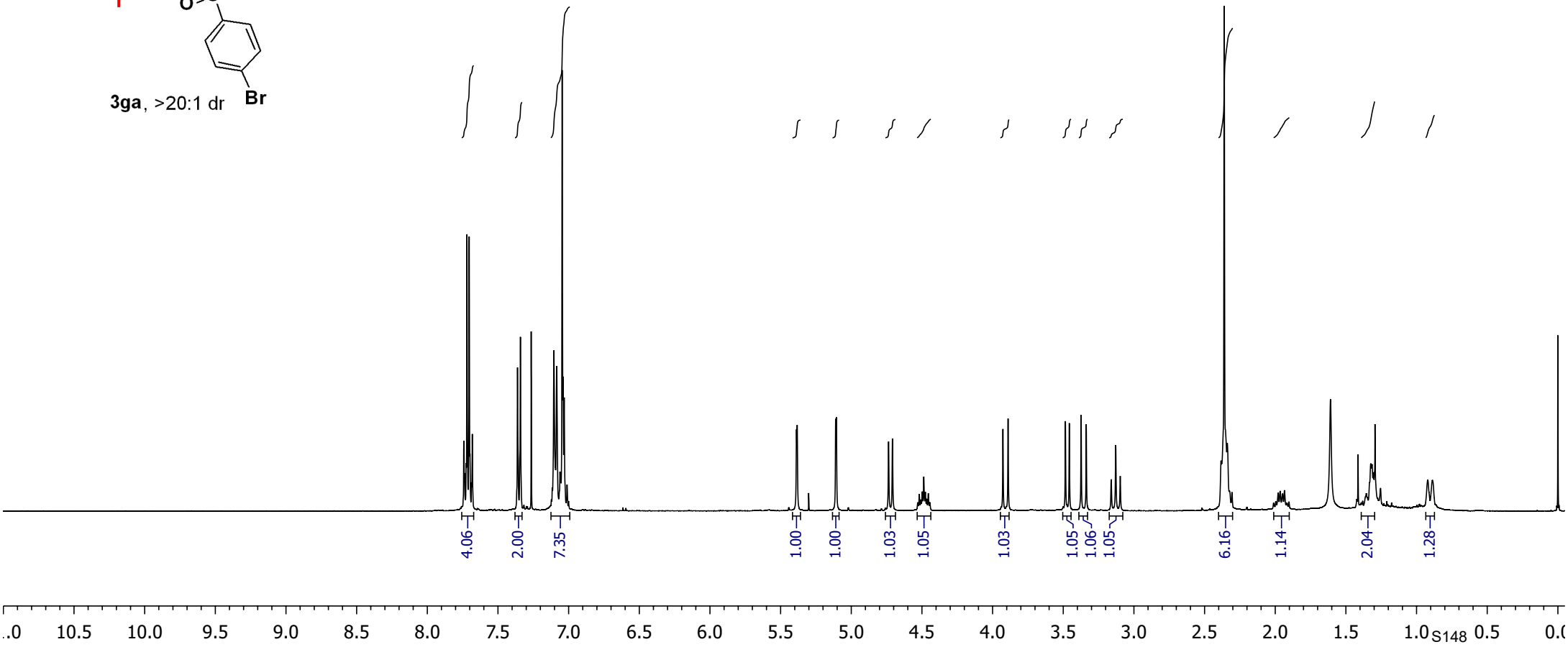
Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



3ga, >20:1 dr

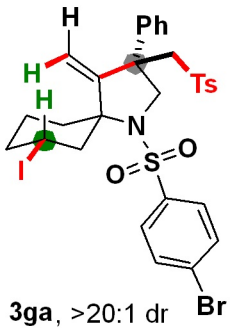


158.34
144.32
138.90
137.18
137.09
132.31
129.58
129.15
128.38
127.68
127.53
127.27
126.43
108.95
77.32
77.00
76.68
70.90
65.05
53.29
49.73
47.62
37.95
34.27
23.71
23.40
21.53

Solvent $CDCl_3$

Spectrometer Frequency 100.66

Nucleus ^{13}C

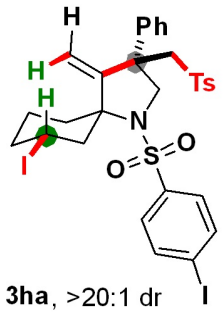


3ga, >20:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 400.28

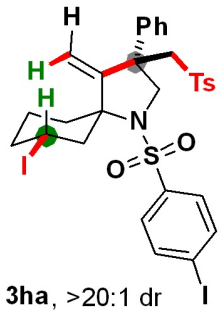
Nucleus ^1H



3ha, >20:1 dr

158.40
144.32
139.58
138.29
137.22
137.15
129.59
129.01
128.39
127.55
127.28
126.44
108.94
100.11
77.31
77.00
76.68
70.92
65.08
53.29
49.75
47.65
37.97
34.28
23.73
23.38
21.54

Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C

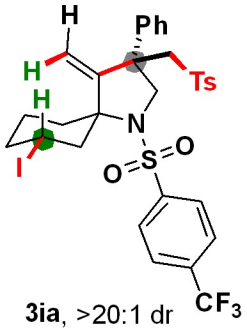


3ha, >20:1 dr

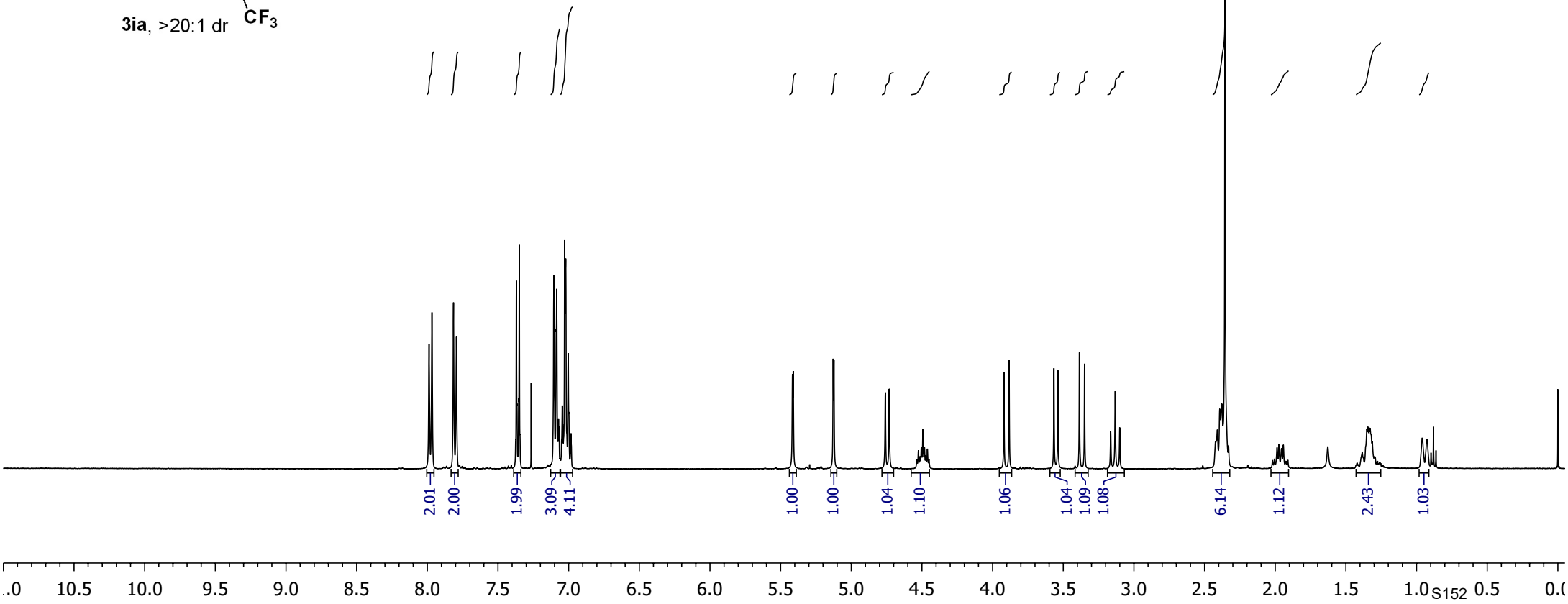
Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H

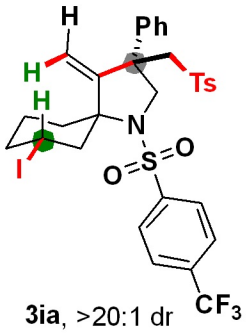


3ia, >20:1 dr

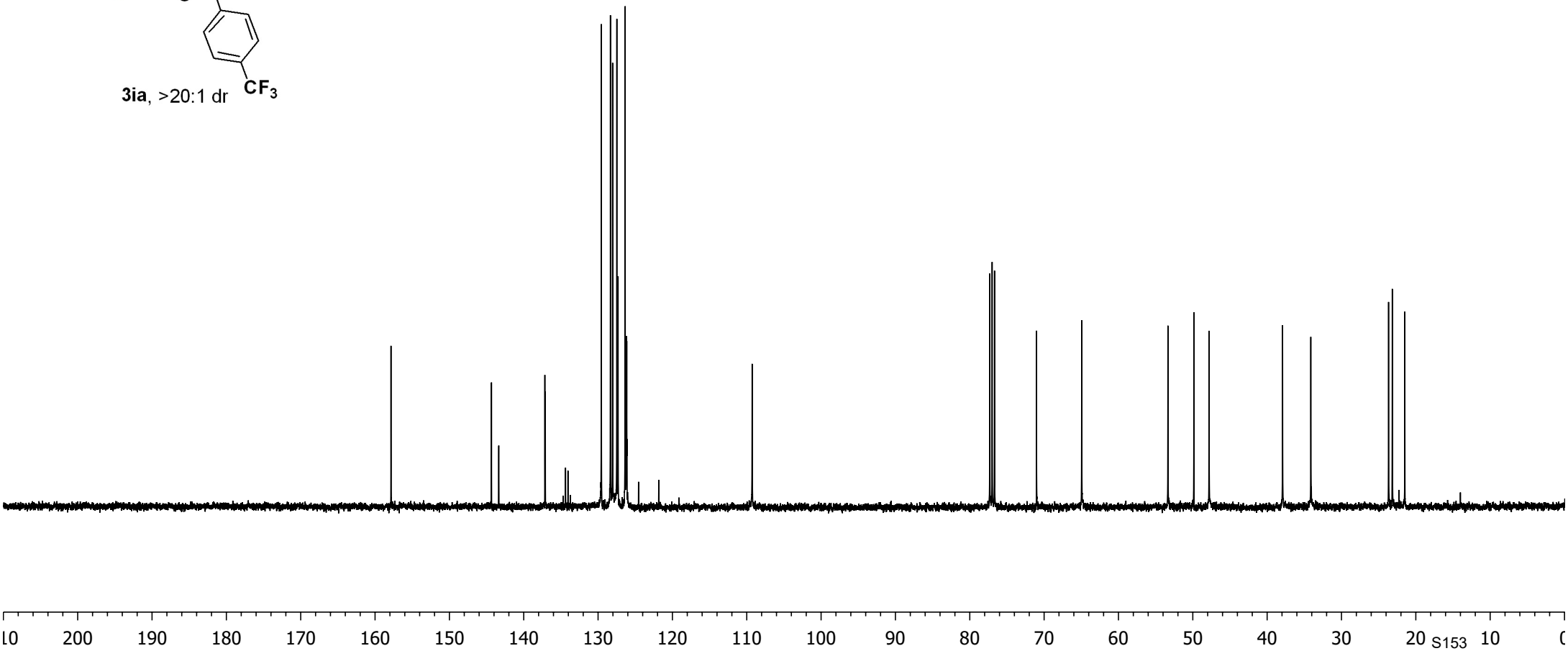


157.86
144.34
143.36
137.16
137.10
134.38
129.56
128.34
128.05
127.48
127.31
126.36
126.18
126.14
77.31
76.99
76.67
71.03
64.94
53.34
49.86
47.81
37.92
34.12
23.66
23.16
21.48

Solvent $CDCl_3$
Spectrometer Frequency 100.45
Nucleus ^{13}C

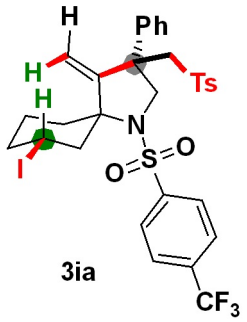


3ia, >20:1 dr

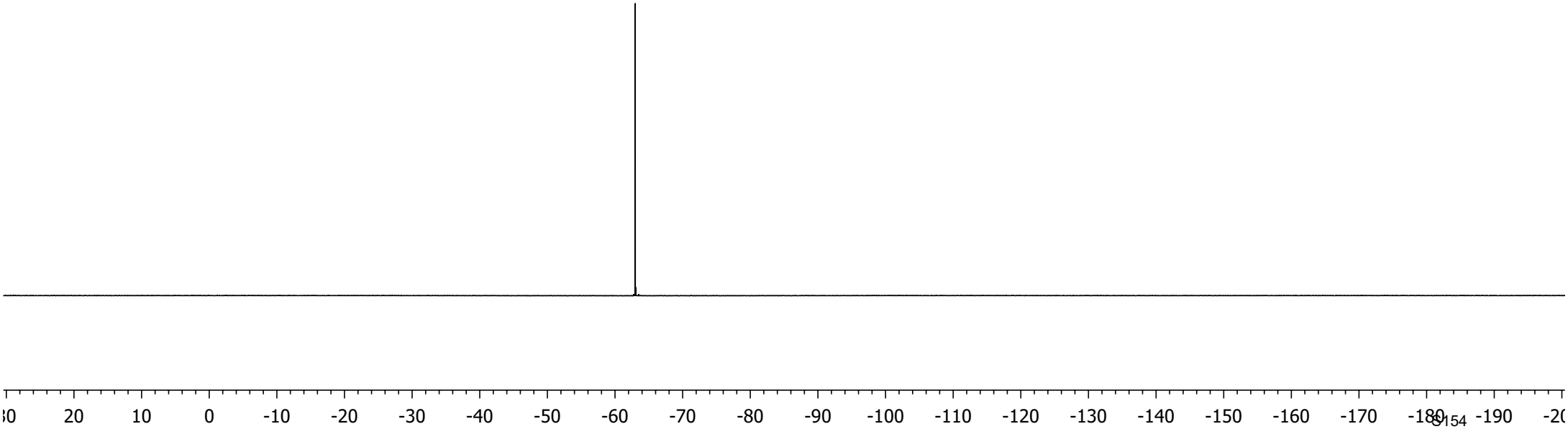


-62.99

Solvent $CDCl_3$
Spectrometer Frequency 376.61
Nucleus ^{19}F



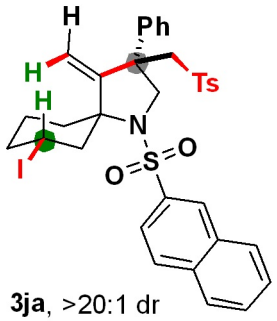
3ia



Solvent $CDCl_3$

Spectrometer Frequency 400.39

Nucleus ^1H



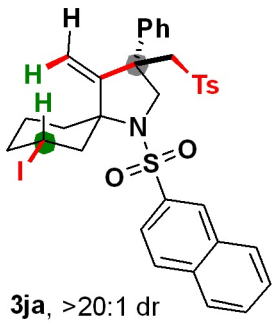
3ja, >20:1 dr

158.72
144.19
136.71
132.11
129.55
129.50
129.19
129.01
128.92
128.24
127.79
127.51
127.07
126.44
122.99
108.72
77.31
76.99
76.67
70.80
65.06
53.28
49.63
47.67
38.01
34.34
23.76
23.71
21.49

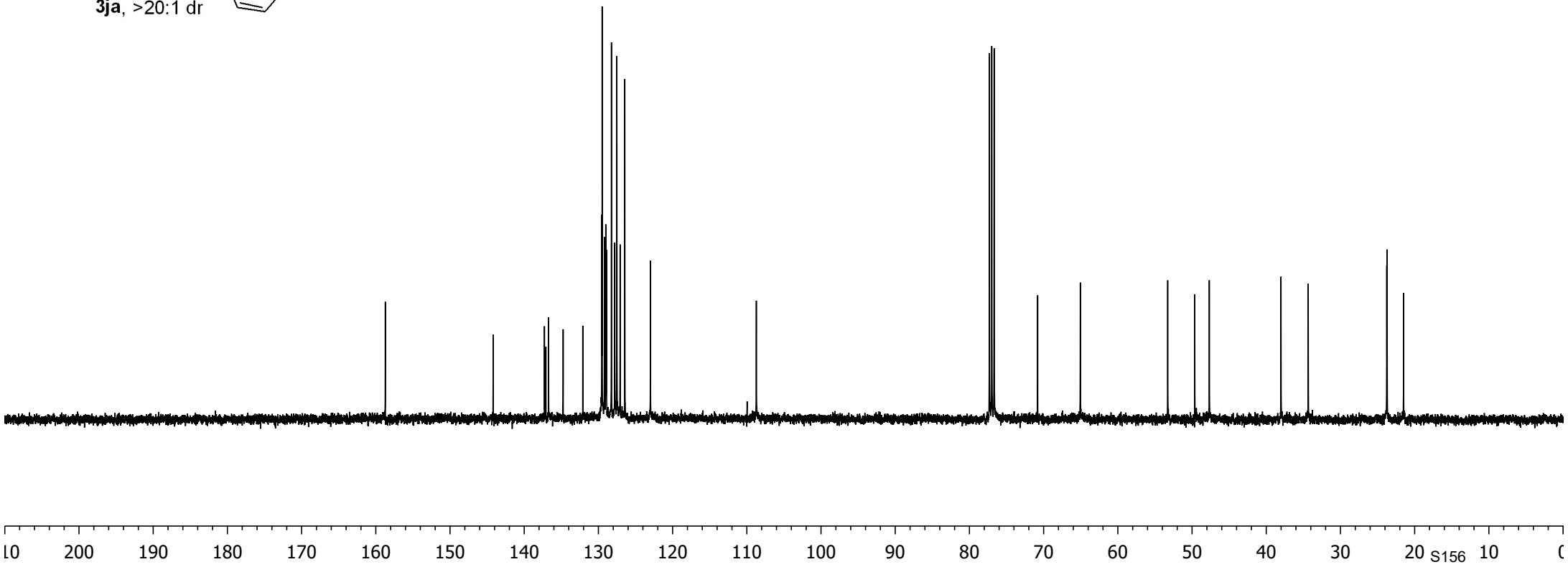
Solvent $CDCl_3$

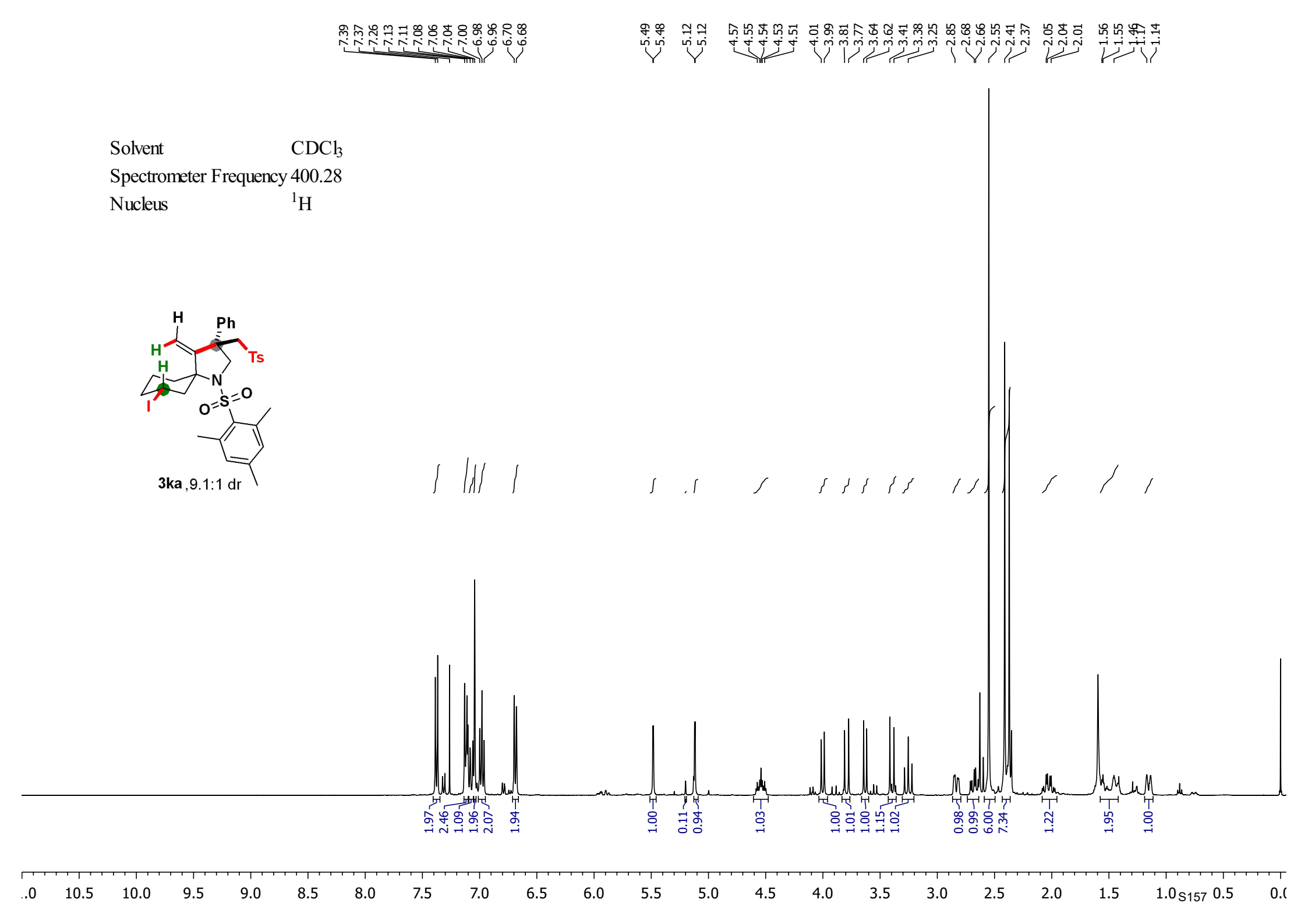
Spectrometer Frequency 100.69

Nucleus ^{13}C



3ja, >20:1 dr



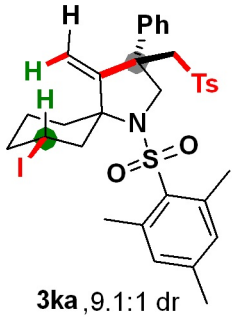

7.39
7.37
7.26
7.13
7.11
7.08
7.06
7.04
7.00
6.98
6.96
6.70
6.68
5.49
5.48
5.12
5.12
4.57
4.55
4.54
4.53
4.51
4.01
3.99
3.81
3.77
3.64
3.62
3.41
3.38
3.25
2.85
2.68
2.66
2.55
2.41
2.37
2.05
2.04
2.01
1.56
1.55
1.46
1.17
1.14
Solvent CDCl3
Spectrometer Frequency 400.28
Nucleus 1H
H
Ph
H
Ts
H
N
I
O=S=O
3ka, 9.1:1 dr
1.97
2.46
1.09
1.96
2.07
1.94
1.00
0.11
0.94
1.03
1.00
1.01
1.00
1.15
1.02
0.98
0.99
6.00
7.34
1.22
1.95
1.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

159.36
144.42
143.46
140.92
137.93
137.36
132.11
129.67
127.95
127.49
127.07
126.26
109.42
77.32
77.00
76.68
71.21
65.02
52.30
49.82
47.59
38.07
33.50
23.92
23.00
21.57
21.15

Solvent $CDCl_3$

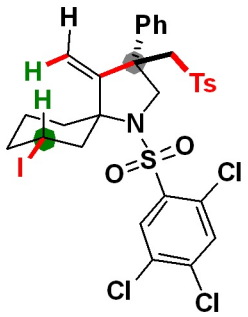
Spectrometer Frequency 100.66

Nucleus ^{13}C



3ka, 9.1:1 dr

Solvent	$CDCl_3$
Spectrometer Frequency	400.39
Nucleus	^1H



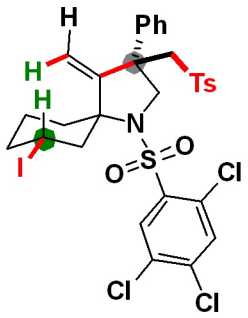
3la, >20:1

157.60
144.51
137.48
137.39
136.73
133.42
133.24
131.83
131.54
129.68
128.39
127.54
127.47
126.41
110.28
77.31
76.99
76.67
71.96
64.40
53.67
50.38
47.96
38.00
33.55
31.59
23.72
22.58
21.57

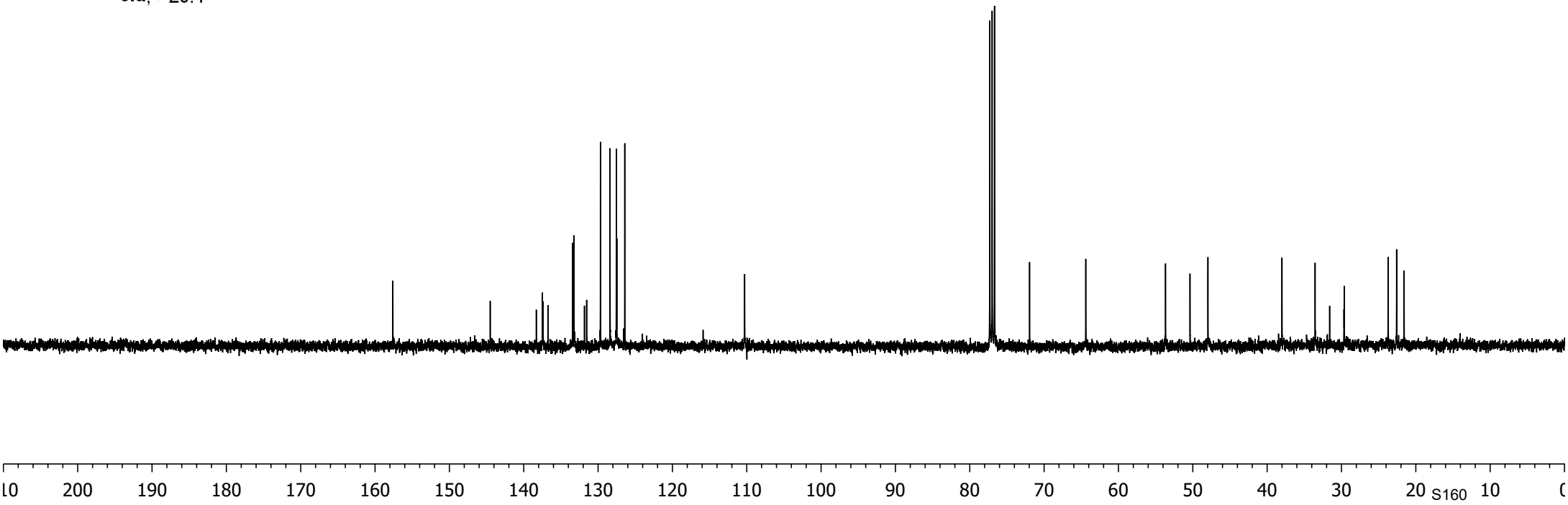
Solvent $CDCl_3$

Spectrometer Frequency 100.69

Nucleus ^{13}C



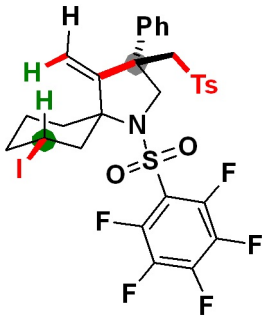
3la, >20:1



Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



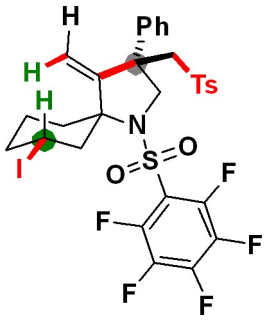
3ma, >20:1 dr

157.00
144.56
137.34
137.23
129.70
128.49
127.60
127.53
126.45
110.23
77.31
76.99
76.67
72.40
64.42
53.45
50.19
47.14
37.90
33.61
23.62
22.23
21.53

Solvent $CDCl_3$

Spectrometer Frequency 100.45

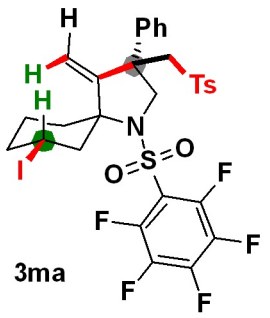
Nucleus ^{13}C



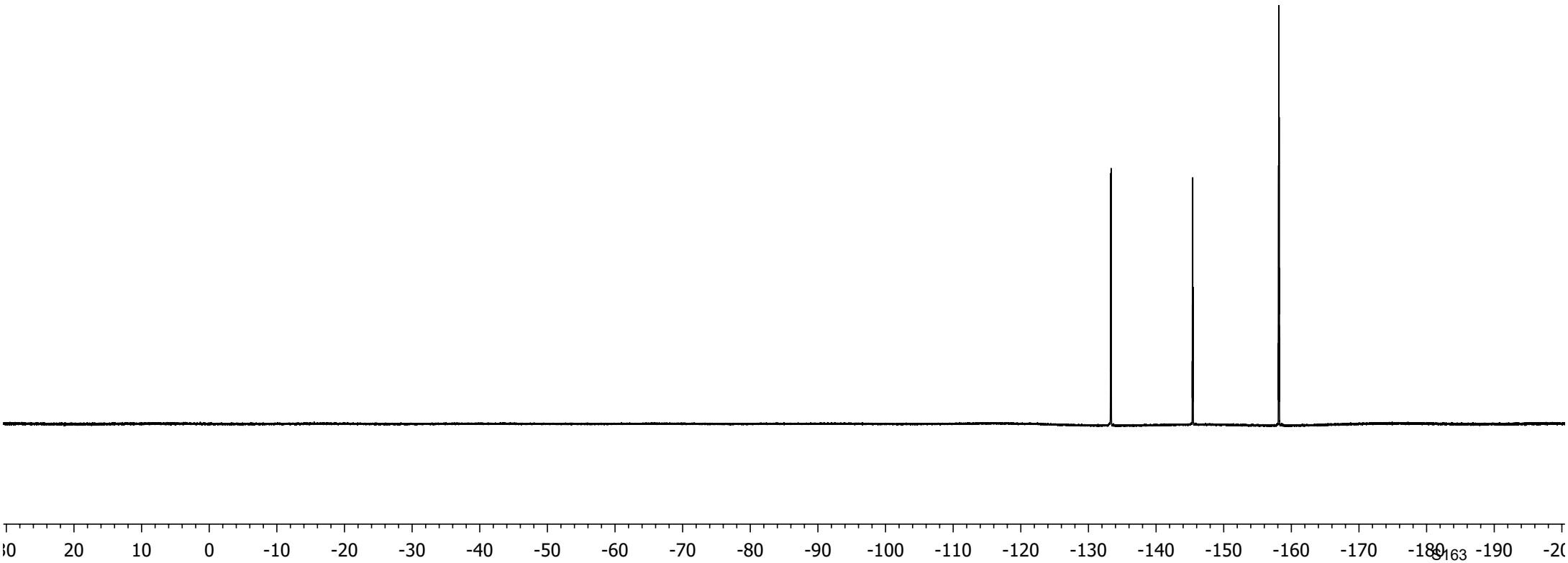
3ma, >20:1 dr

-133.35
-145.42
-158.15

Solvent $CDCl_3$
Spectrometer Frequency 376.61
Nucleus ^{19}F



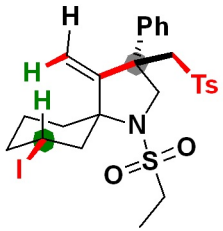
3ma



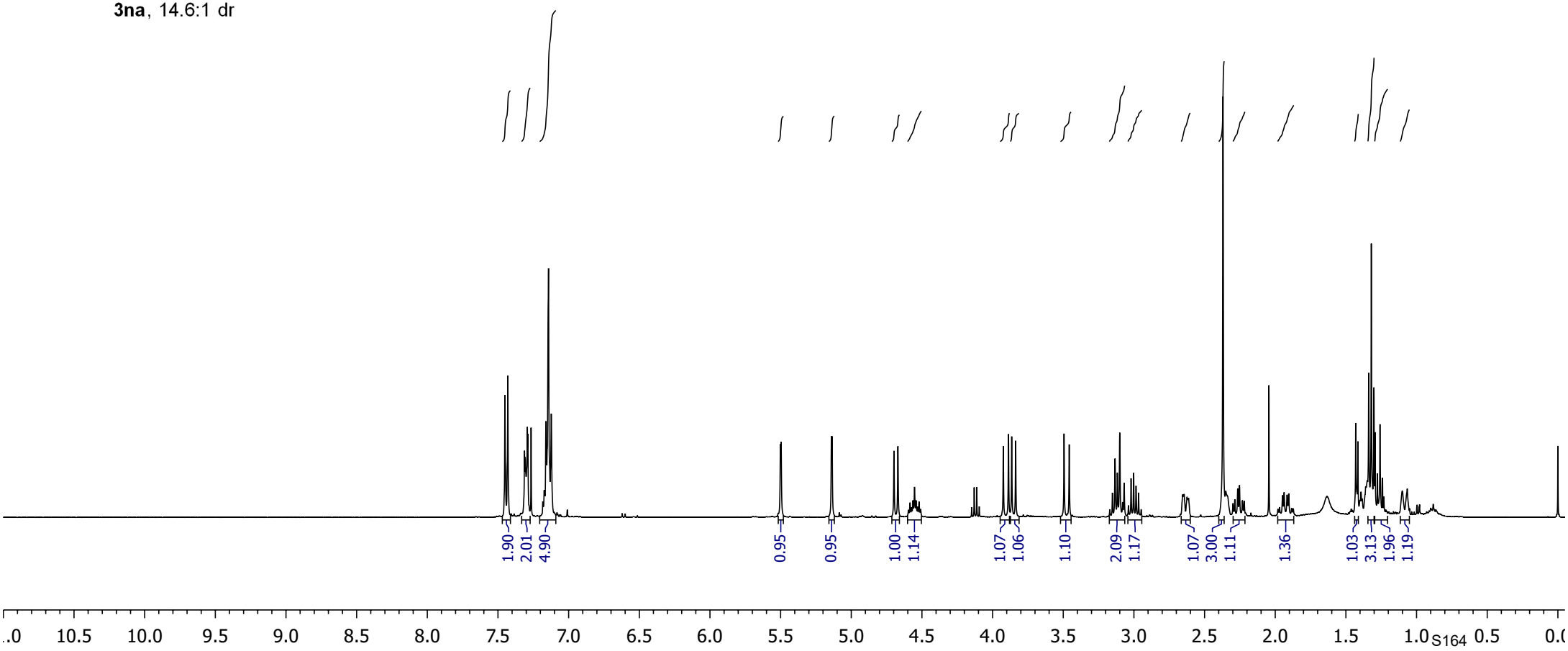
Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H

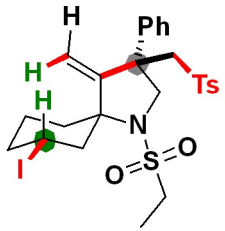


3na, 14.6:1 dr

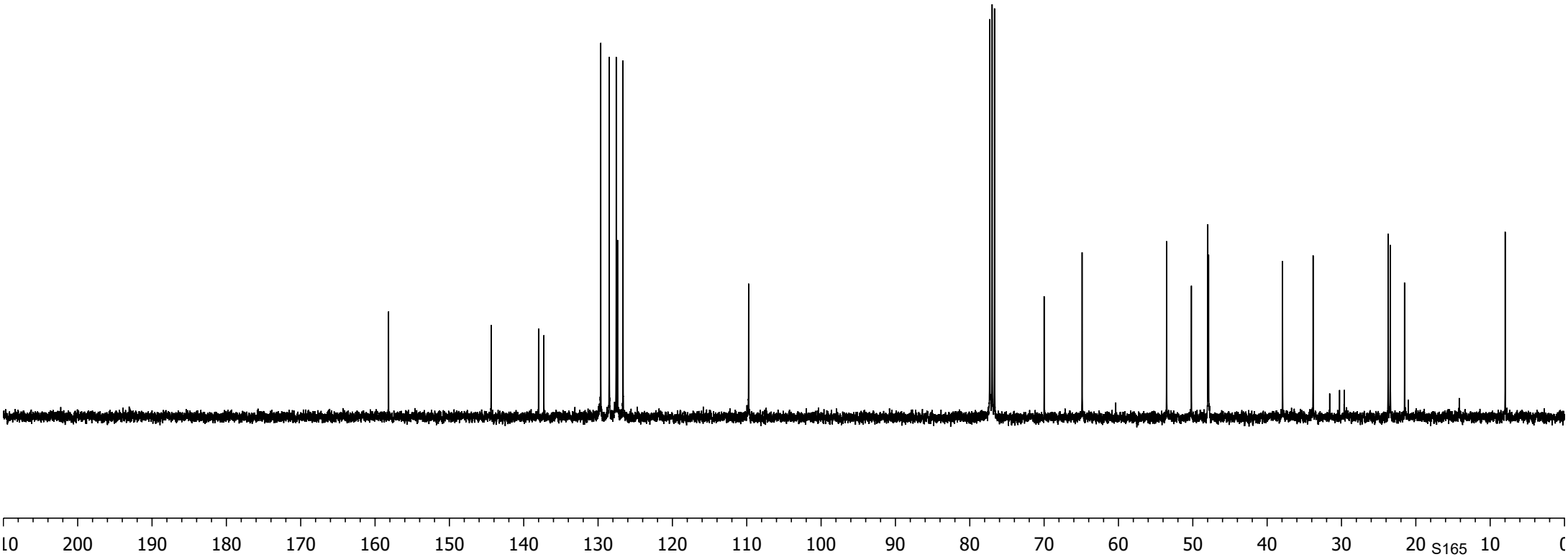


158.18
144.39
138.01
137.30
129.64
128.49
127.56
127.38
126.65
109.75
77.31
76.99
76.67
70.00
64.89
53.53
50.19
47.98
47.87
37.93
33.82
23.72
23.44
21.52
7.98

Solvent $CDCl_3$
Spectrometer Frequency 100.45
Nucleus ^{13}C



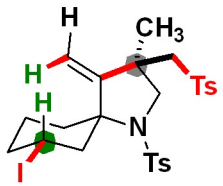
3na, 14.6:1 dr



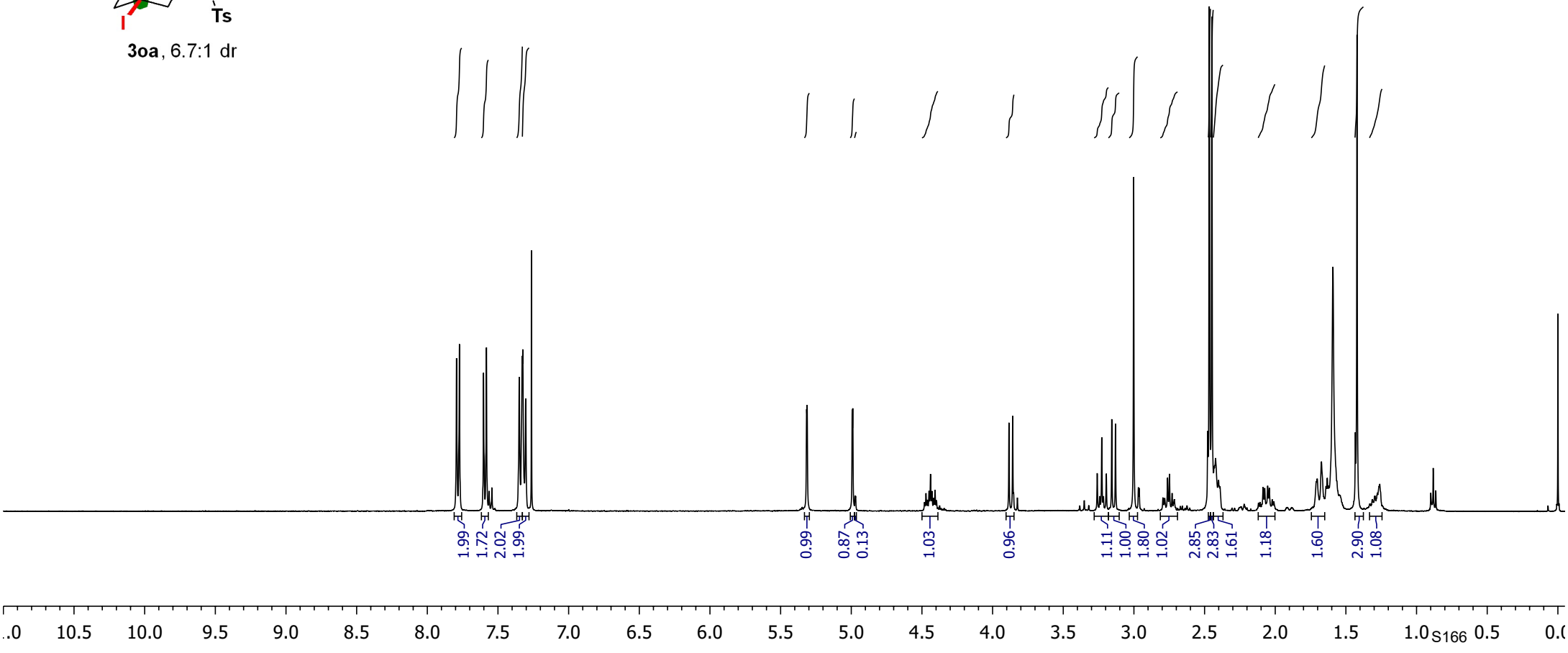
Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H

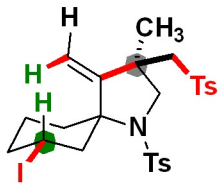


3oa, 6.7:1 dr



160.87
144.77
143.43
138.19
137.70
129.87
129.62
127.59
127.48
109.24
77.30
76.99
76.67
70.44
63.28
55.72
47.83
44.34
37.97
33.48
23.68
23.63
22.84
21.62
21.58

Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C

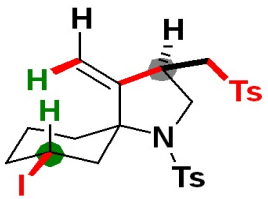


3oa, 6.7:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 400.39

Nucleus ^1H



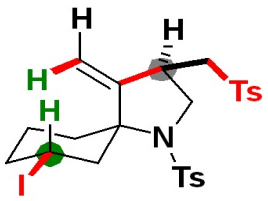
3pa, 5.5:1 dr

154.46
145.23
143.36
137.77
136.11
130.13
130.11
129.62
127.87
127.23
108.90
77.31
76.99
76.68
70.12
58.55
50.75
47.52
38.05
36.88
33.94
24.10
23.30
21.68
21.53

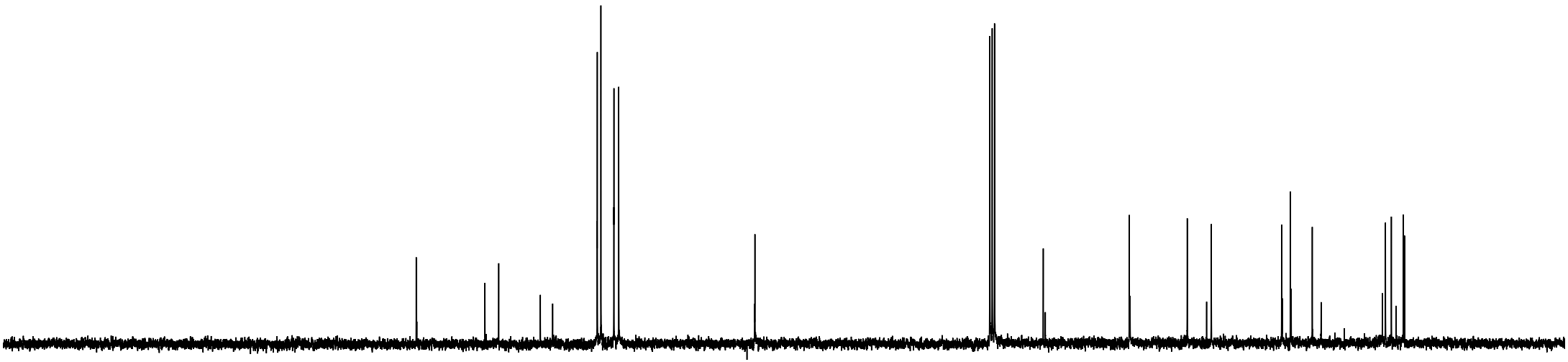
Solvent $CDCl_3$

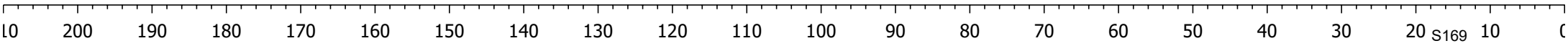
Spectrometer Frequency 100.69

Nucleus ^{13}C



3pa, 5.5:1 dr

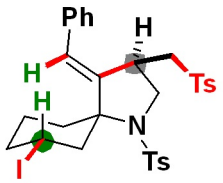




Solvent $CDCl_3$

Spectrometer Frequency 400.40

Nucleus ^1H



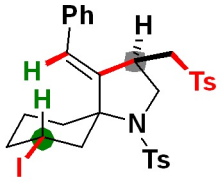
3qa, 20:1 dr

149.65
145.03
144.08
143.72
136.24
135.22
129.88
129.81
128.71
128.55
127.75
127.27
97.26
77.31
76.99
76.67
74.15
59.60
50.11
45.64
37.58
33.14
25.37
23.29
21.69
21.64
21.44

Solvent $CDCl_3$

Spectrometer Frequency 100.69

Nucleus ^{13}C

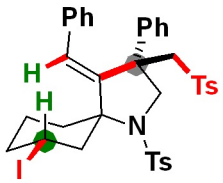


3qa, 20:1 dr

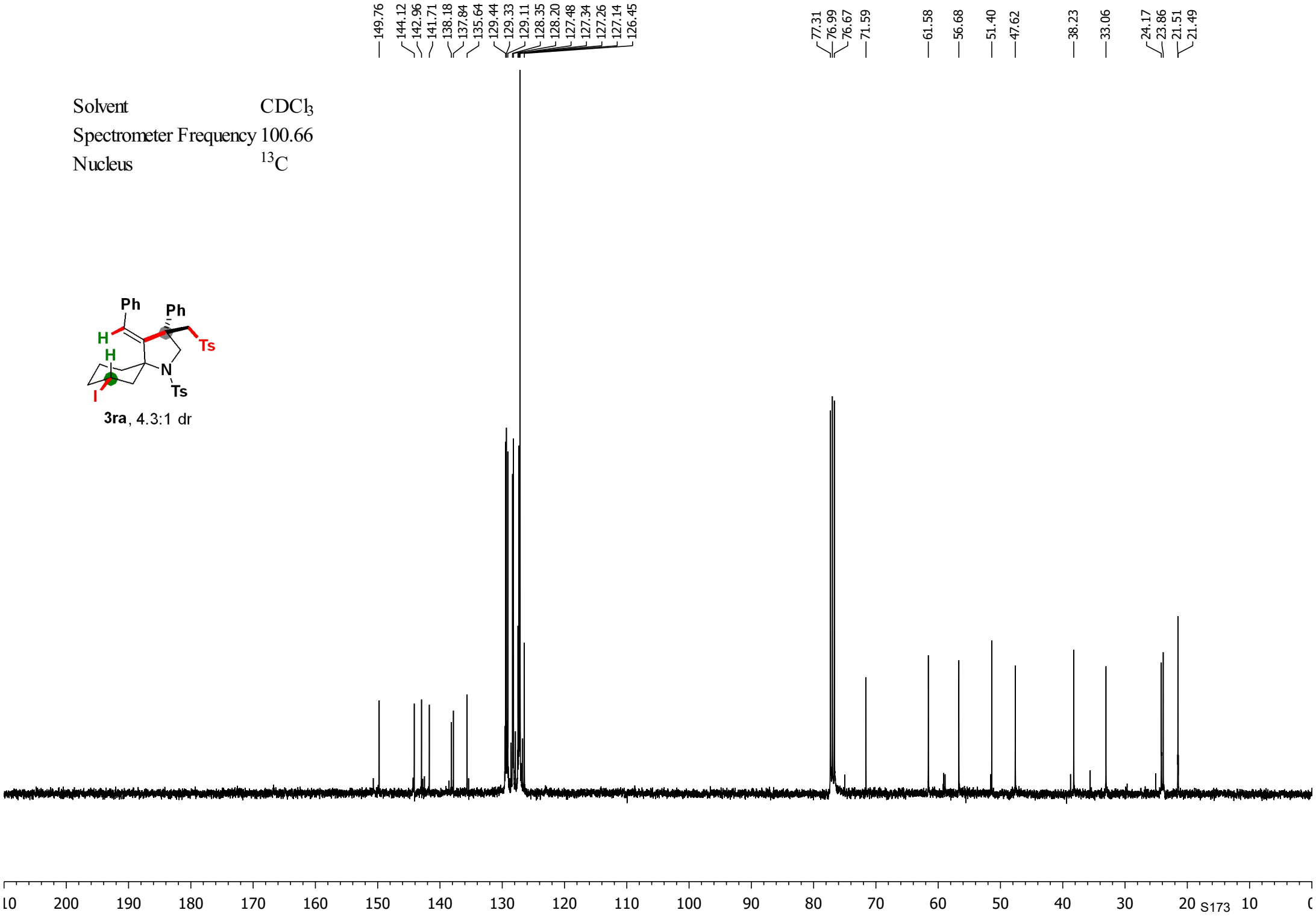
Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



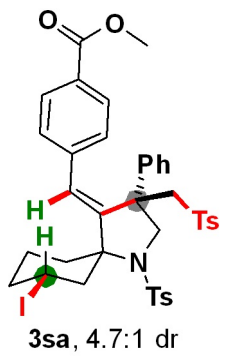
3ra, 4.3:1 dr


149.76
144.12
142.96
141.71
138.18
137.84
135.64
129.44
129.33
129.11
128.35
128.20
127.48
127.34
127.26
127.14
126.45
77.31
76.99
76.67
71.59
61.58
56.68
51.40
47.62
38.23
33.06
24.17
23.86
21.51
21.49
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
Ph
Ph
H
H
Ts
N
Ts
I
3ra, 4.3:1 dr
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H

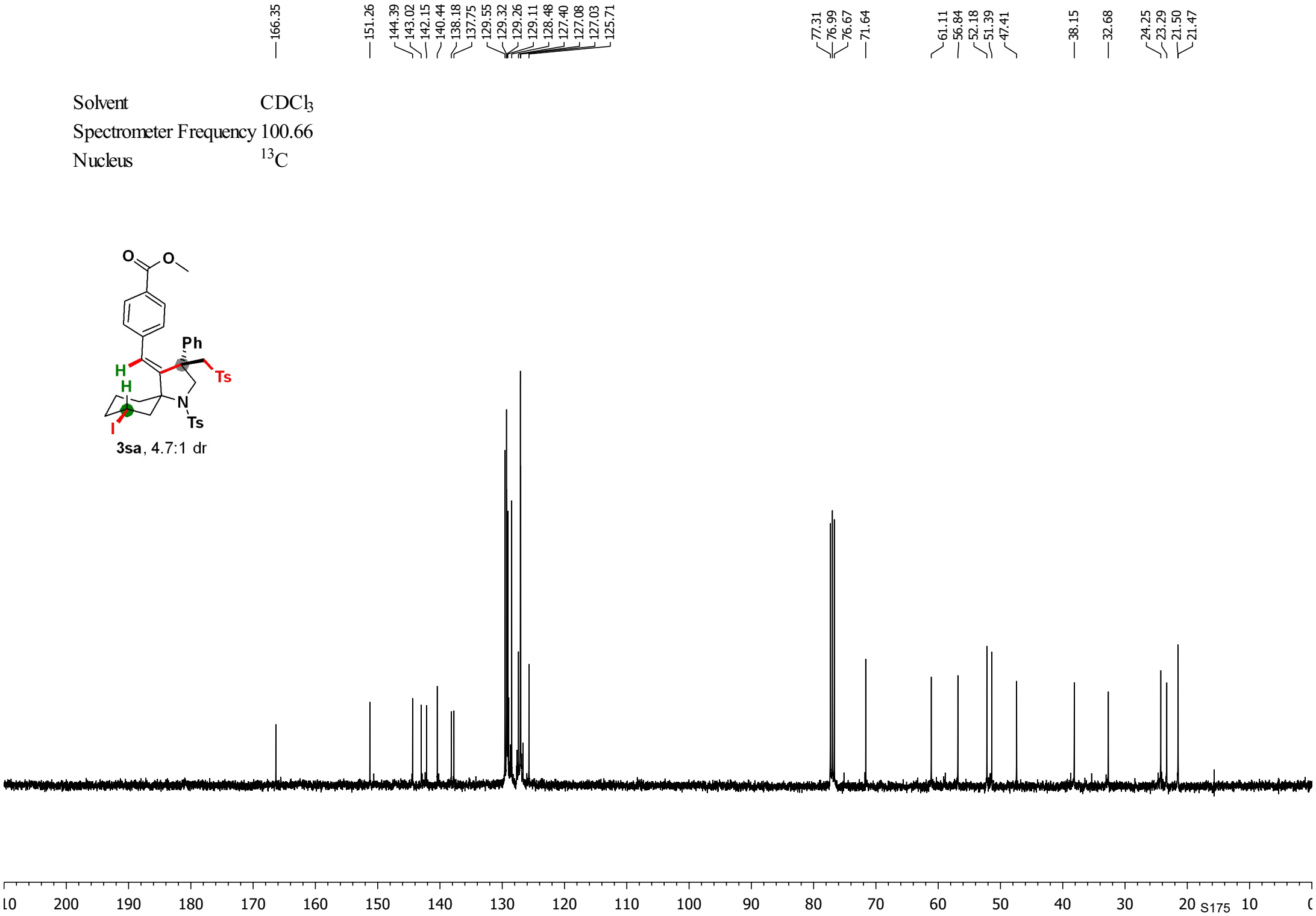


3sa, 4.7:1 dr

DMF

DMF

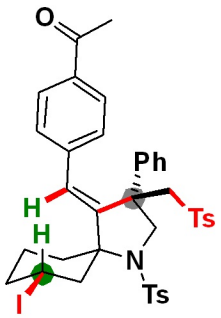
S174


166.35
151.26
144.39
143.02
142.15
140.44
138.18
137.75
129.55
129.32
129.26
129.11
128.48
127.40
127.08
127.03
125.71
77.31
76.99
76.67
71.64
61.11
56.84
52.18
51.39
47.41
38.15
32.68
24.25
23.29
21.50
21.47
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
O
O
Ph
H
H
Ts
N
Ts
I
3sa, 4.7:1 dr
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

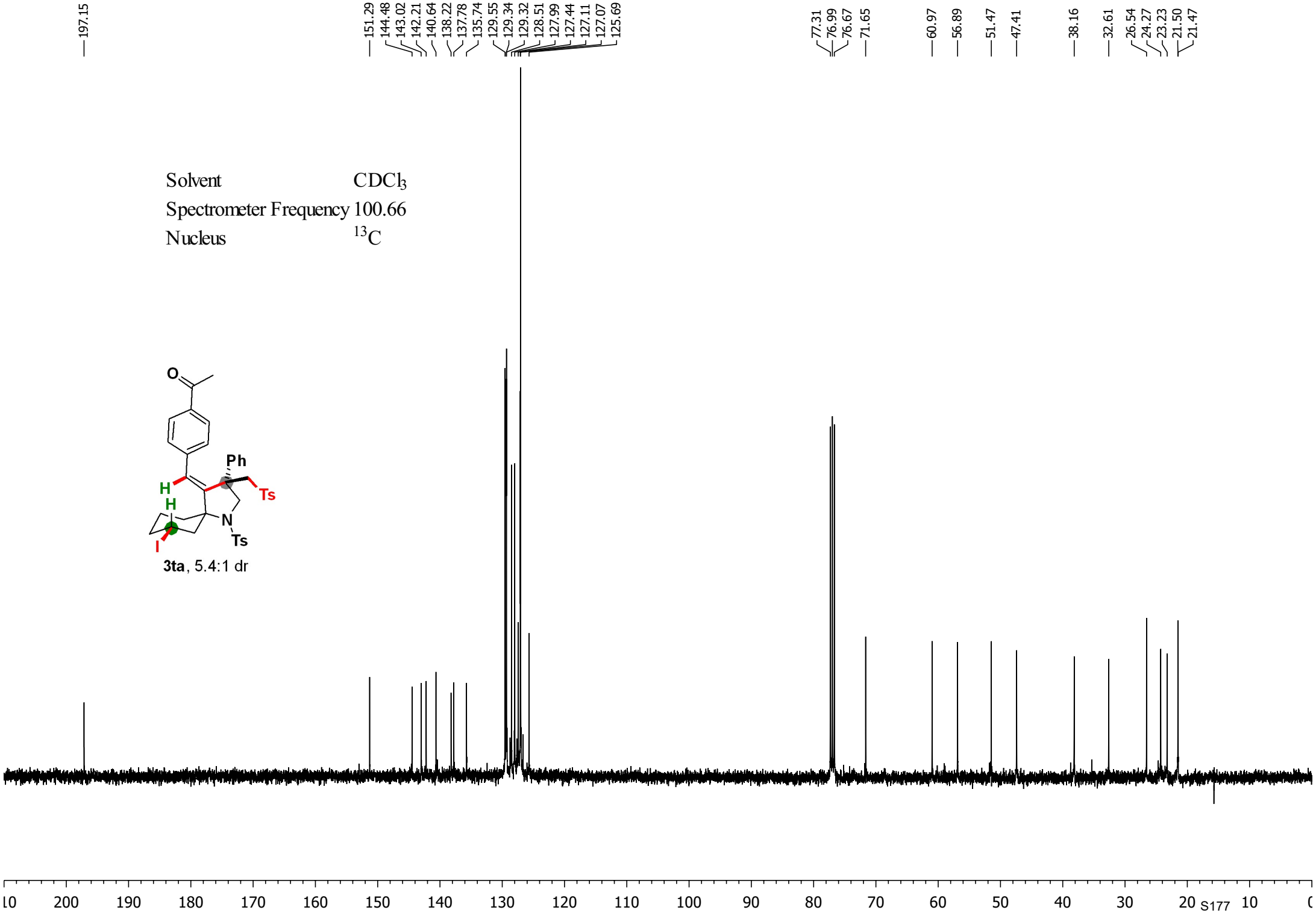
Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



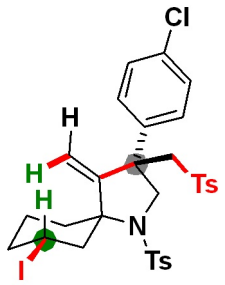
3ta, 5.4:1 dr


197.15
151.29
144.48
143.02
142.21
140.64
138.22
137.78
135.74
129.55
129.34
129.32
128.51
127.99
127.44
127.11
127.07
125.69
77.31
76.99
76.67
71.65
60.97
56.89
51.47
47.41
38.16
32.61
26.54
24.27
23.23
21.50
21.47
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
O
Ph
H
H
Ts
N
Ts
I
3ta, 5.4:1 dr
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Solvent $CDCl_3$

Spectrometer Frequency 400.28

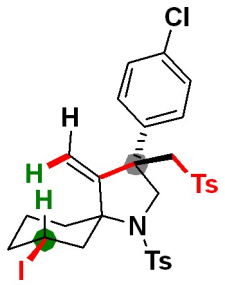
Nucleus ^1H



3ua, >20:1 dr

158.80
144.56
143.66
136.74
136.69
135.86
133.30
129.67
129.56
128.29
128.02
127.68
127.50
108.60
77.31
76.99
76.67
70.62
64.91
53.26
49.00
47.26
37.91
34.58
23.71
23.55
21.55

Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C

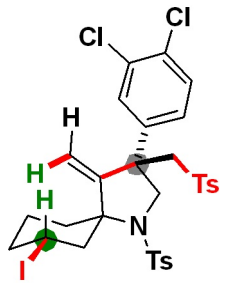


3ua, >20:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H

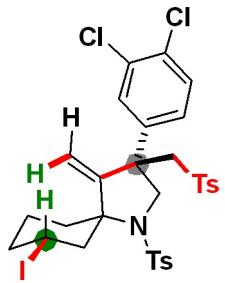


3va, >20:1 dr

DMF

158.46
144.84
143.83
137.72
136.43
136.34
132.25
131.66
130.03
129.79
129.51
128.76
127.60
127.29
126.12
109.10
77.31
76.99
76.67
70.56
64.54
53.15
48.78
47.16
37.84
34.64
23.69
23.29
21.56
21.55

Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C

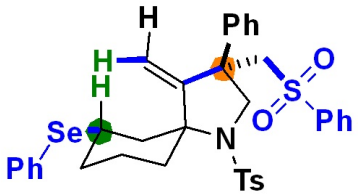


3va, >20:1 dr

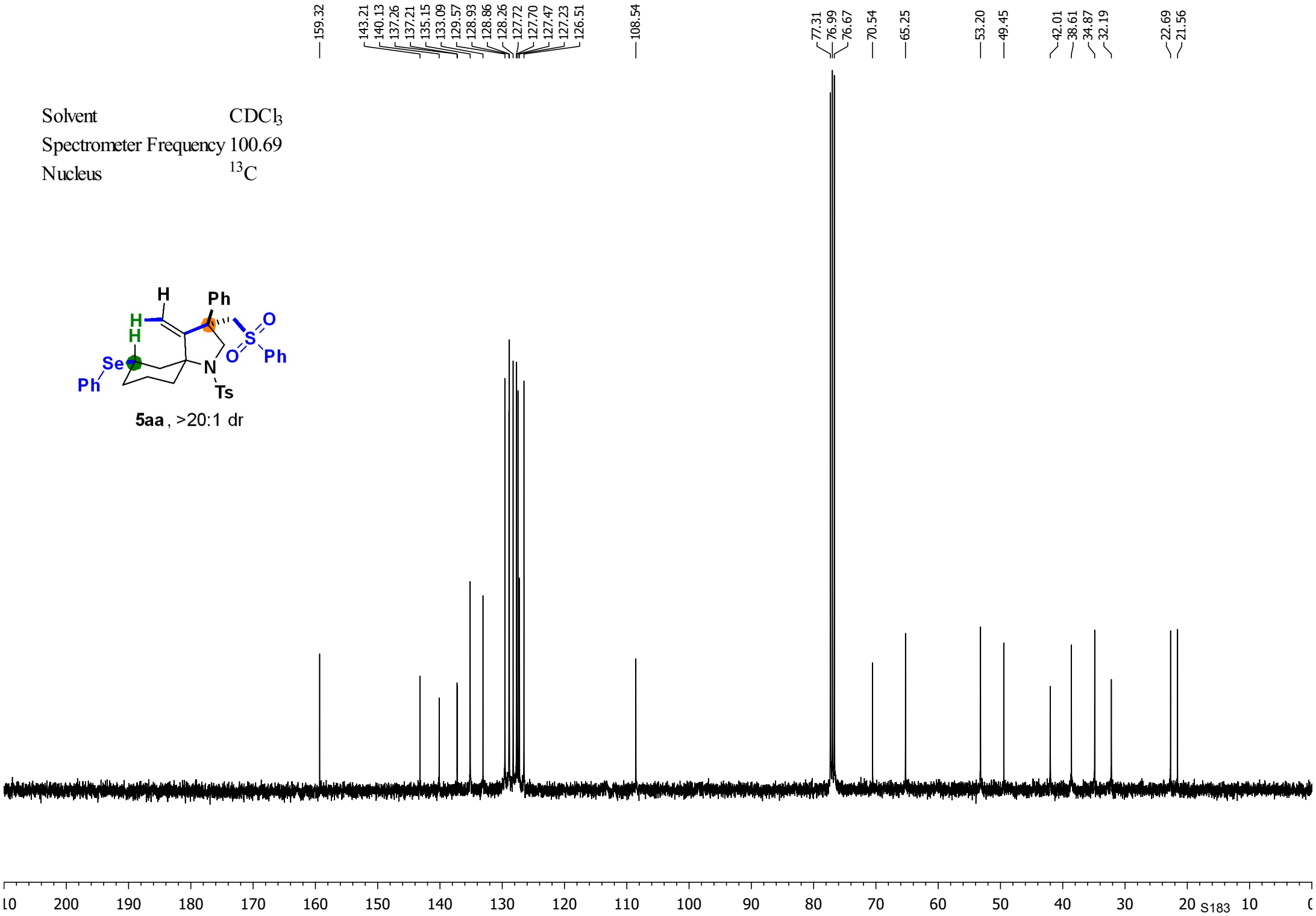
Solvent $CDCl_3$

Spectrometer Frequency 400.40

Nucleus ^1H



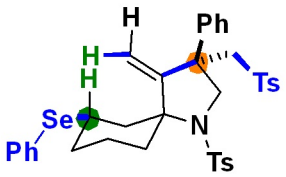
5aa, >20:1 dr


159.32
143.21
140.13
137.26
137.21
135.15
133.09
129.57
128.93
128.86
128.26
127.72
127.70
127.47
127.23
126.51
108.54
77.31
76.99
76.67
70.54
65.25
53.20
49.45
42.01
38.61
34.87
32.19
22.69
21.56
Solvent CDCl3
Spectrometer Frequency 100.69
Nucleus 13C
H
Ph
H
H
O
S
O
Ph
Se
Ph
N
Ts
5aa, >20:1 dr
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

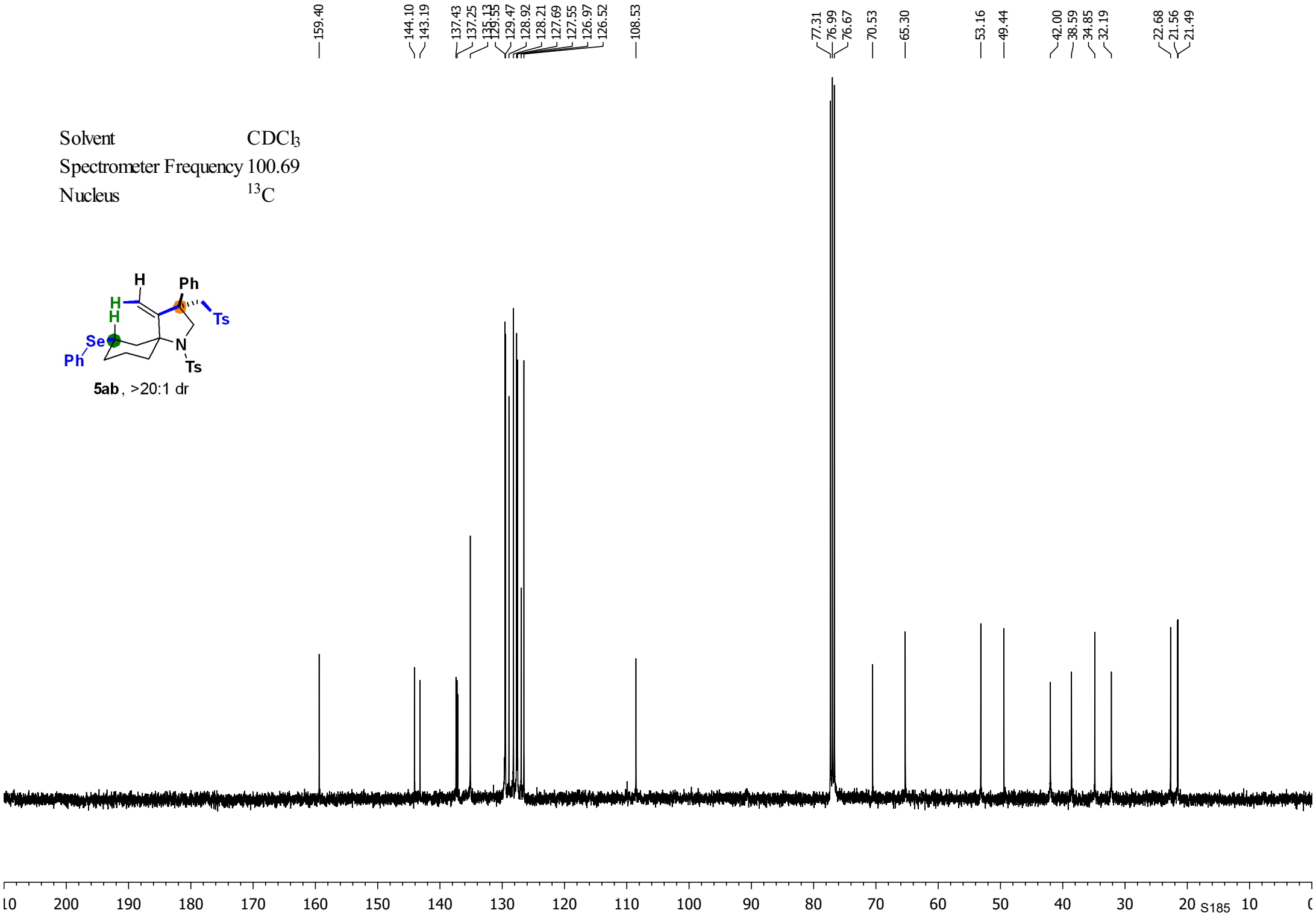
Solvent $CDCl_3$

Spectrometer Frequency 400.40

Nucleus ^1H



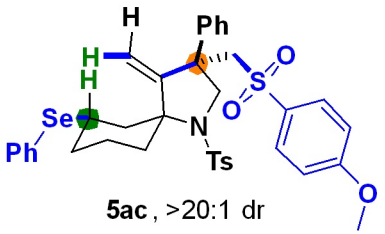
5ab, >20:1 dr


159.40
144.10
143.19
137.43
137.25
135.13
129.55
129.47
128.92
128.21
127.69
127.55
126.97
126.52
108.53
77.31
76.99
76.67
70.53
65.30
53.16
49.44
42.00
38.59
34.85
32.19
22.68
21.56
21.49
Solvent CDCl3
Spectrometer Frequency 100.69
Nucleus 13C
H
Ph
H
H
Ts
Se
Ph
N
Ts
5ab, >20:1 dr
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

Solvent $CDCl_3$

Spectrometer Frequency 400.40

Nucleus ^1H



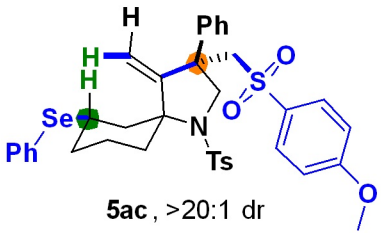
5ac, >20:1 dr

163.21
159.45
143.20
137.46
135.11
129.71
129.55
128.93
128.24
127.73
127.68
127.06
126.52
114.08
108.47
77.31
76.99
76.67
70.53
65.41
55.61
53.18
49.40
41.94
38.64
34.90
32.16
22.67
21.55

Solvent $CDCl_3$

Spectrometer Frequency 100.69

Nucleus ^{13}C

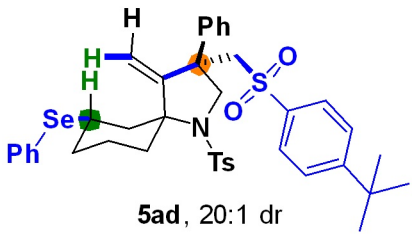


5ac, >20:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



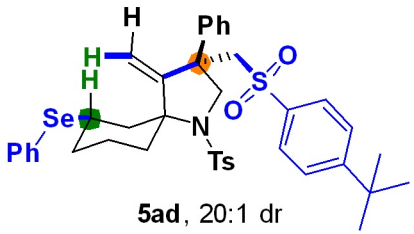
5ad, 20:1 dr

159.56
156.76
143.16
137.25
137.22
135.10
129.55
128.90
128.19
128.13
127.68
127.30
127.07
126.49
125.81
108.36
77.31
76.99
76.67
70.52
65.18
53.15
49.28
41.98
38.61
35.01
34.93
32.15
30.92
22.67
21.53

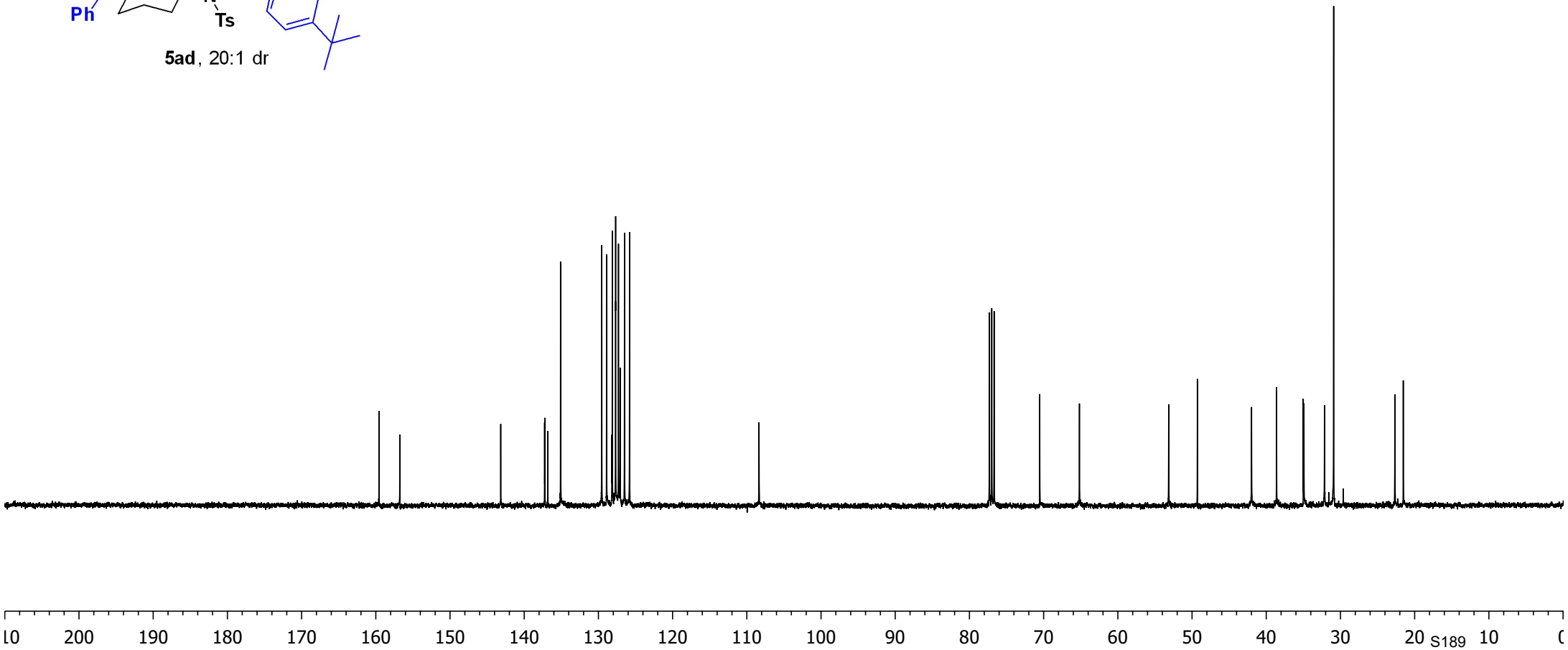
Solvent $CDCl_3$

Spectrometer Frequency 100.45

Nucleus ^{13}C



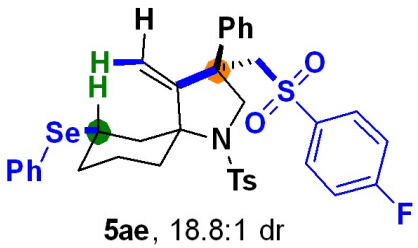
5ad, 20:1 dr



Solvent $CDCl_3$

Spectrometer Frequency 400.40

Nucleus ^1H



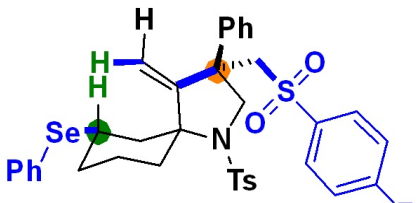
5ae, 18.8:1 dr

166.48
163.93
159.38
143.28
135.17
130.34
129.60
128.93
128.34
127.75
127.69
127.28
116.17
115.94
108.45
77.31
76.99
76.68
70.57
65.48
53.18
49.28
41.95
38.58
34.99
32.15
22.68
21.57

Solvent $CDCl_3$

Spectrometer Frequency 100.69

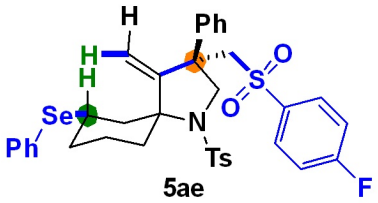
Nucleus ^{13}C



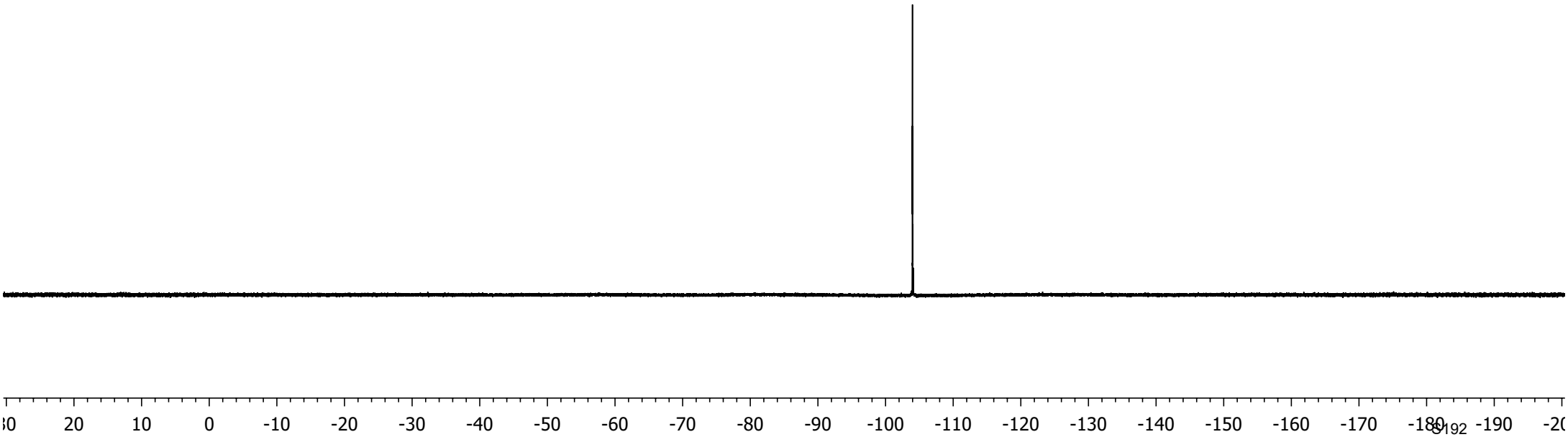
5ae, 18.8:1 dr

-103.98

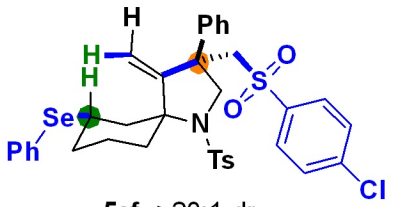
Solvent	$CDCl_3$
Spectrometer Frequency	376.61
Nucleus	^{19}F



5ae



Solvent	$CDCl_3$
Spectrometer Frequency	400.40
Nucleus	^1H



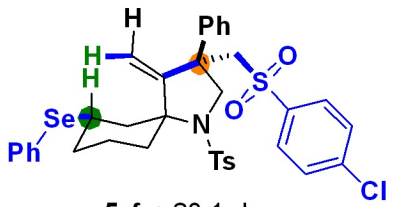
5af, >20:1 dr

159.50
143.28
139.84
138.30
137.18
137.11
133.82
132.69
129.61
129.08
128.99
128.83
128.38
127.67
127.29
127.26
126.55
108.47
77.31
76.99
76.67
70.35
65.44
53.16
49.24
43.56
41.13
35.09
31.42
21.94
21.55

Solvent $CDCl_3$

Spectrometer Frequency 100.69

Nucleus ^{13}C

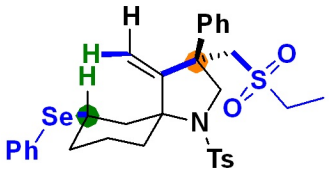


5af, >20:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 399.44

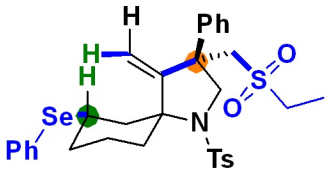
Nucleus ^1H



5ag, >20:1 dr

159.16 143.20 137.64 137.23 135.19 129.57 128.94 128.84 128.15 127.92 127.77 127.67 126.57 108.71 77.31 76.99 76.67 70.57 61.65 52.83 49.29 48.65 42.07 38.71 34.90 32.19 22.73 21.53 6.32

Solvent $CDCl_3$
Spectrometer Frequency 100.45
Nucleus ^{13}C

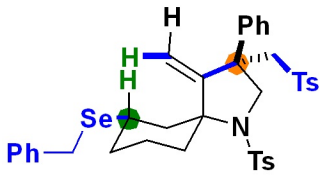


5ag, >20:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 399.44

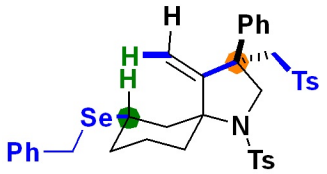
Nucleus ^1H



5ah, 16.4:1 dr

159.19
144.09
143.21
139.20
137.44
137.25
137.15
129.52
129.47
128.86
128.39
128.16
127.72
127.51
126.55
126.51
108.51
77.31
76.99
76.67
70.37
65.33
53.15
49.43
42.14
34.78
33.68
32.19
25.88
22.75
21.53
21.47

Solvent $CDCl_3$
Spectrometer Frequency 100.45
Nucleus ^{13}C

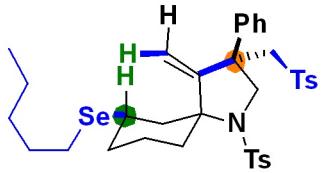


5ah, 16.4:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



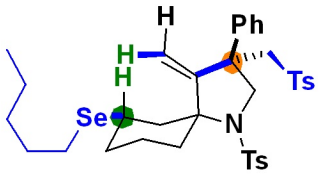
5ai, 20:1 dr

159.54
144.08
143.19
137.46
137.28
137.15
129.51
129.46
128.17
127.68
127.51
126.94
126.52
108.47
77.31
76.99
76.67
70.42
65.31
53.18
49.48
42.35
34.83
33.41
32.50
32.15
30.48
22.19
22.14
21.50
21.46
13.94

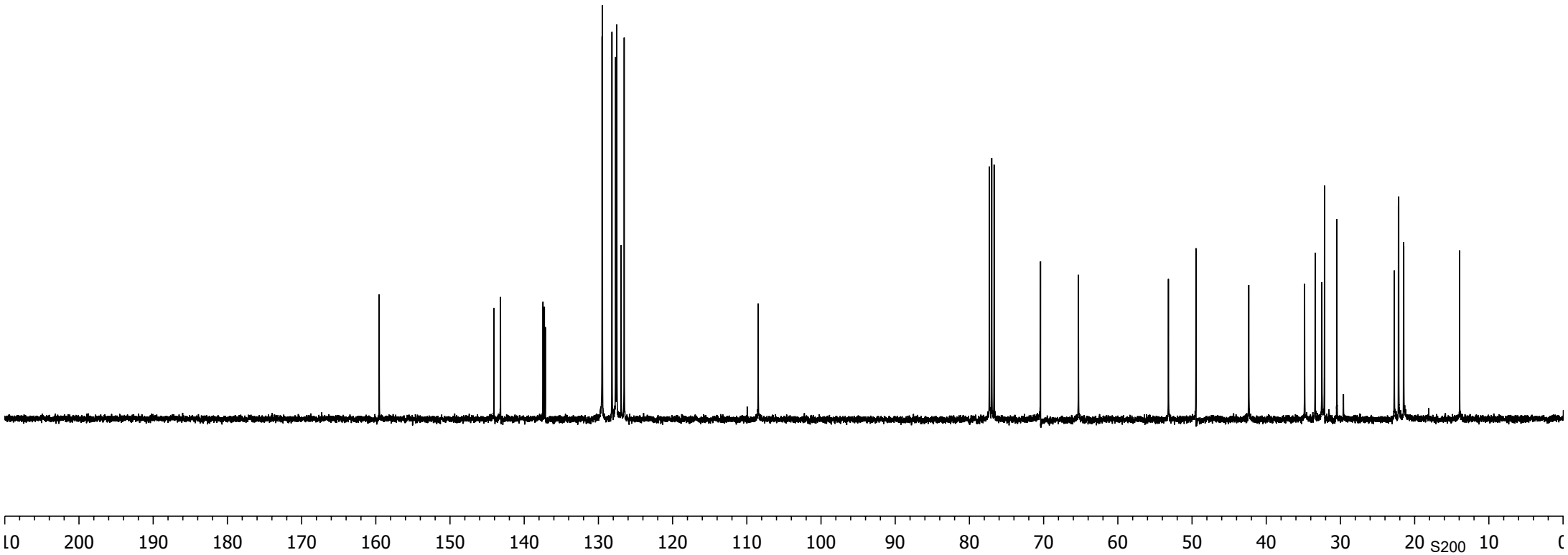
Solvent $CDCl_3$

Spectrometer Frequency 100.45

Nucleus ^{13}C



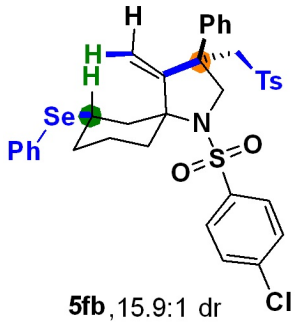
5ai, 20:1 dr



Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



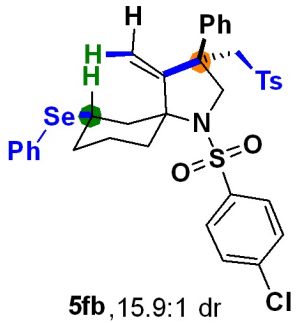
5fb, 15.9:1 dr

158.73
144.17
138.86
138.72
137.28
135.07
129.49
129.21
128.98
128.97
128.27
127.77
127.48
77.31
76.99
76.67
70.77
65.16
53.23
49.59
42.22
38.33
34.77
32.08
22.61
21.47

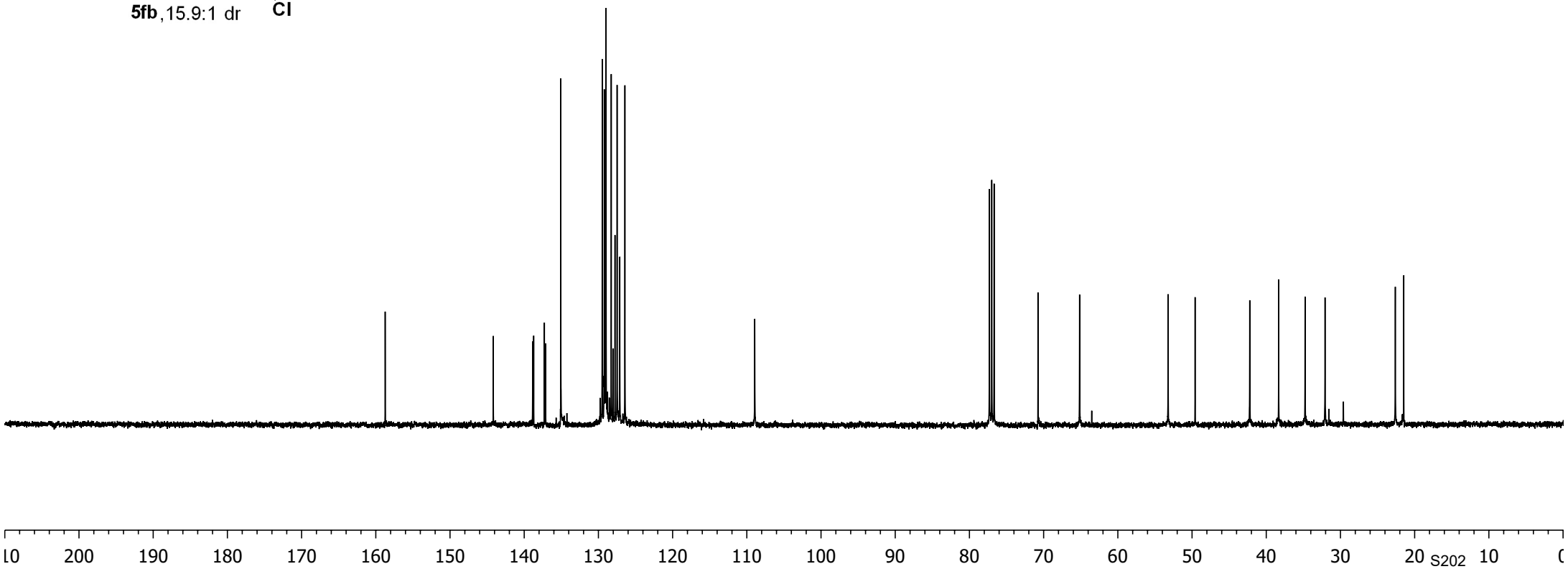
Solvent $CDCl_3$

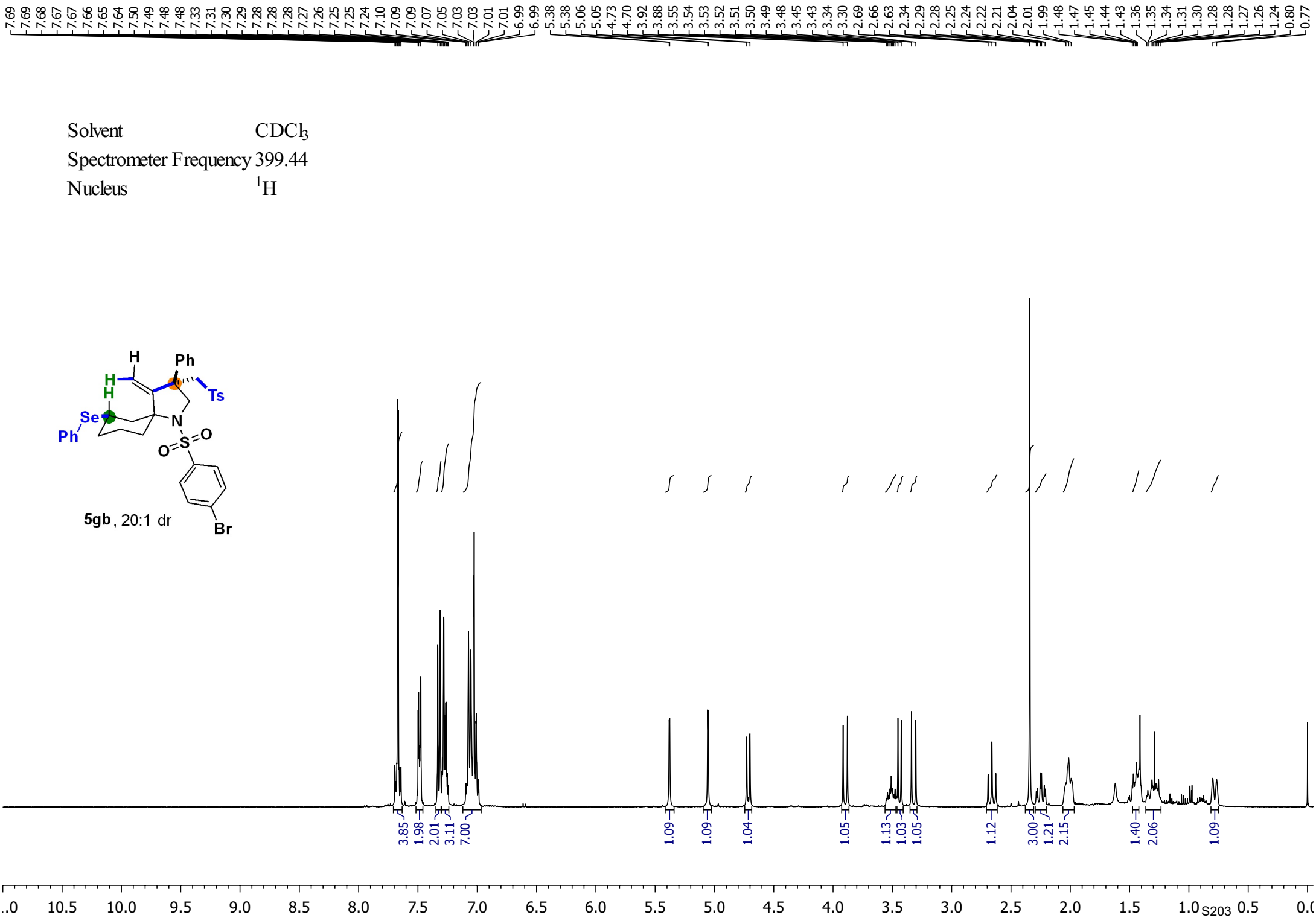
Spectrometer Frequency 100.45

Nucleus ^{13}C



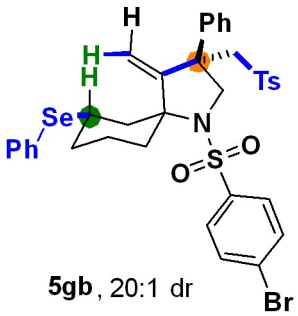
5fb, 15.9:1 dr



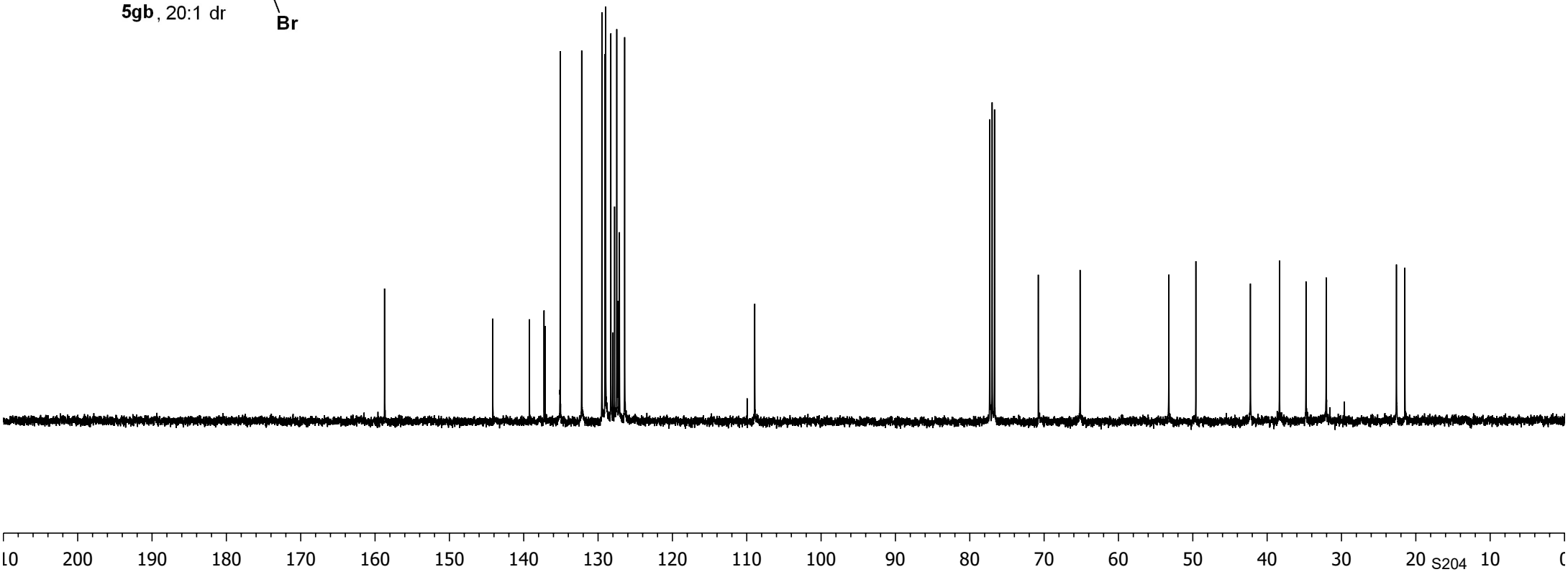

7.69 7.69 7.68 7.67 7.67 7.66 7.65 7.64 7.50 7.49 7.48 7.48 7.33 7.31 7.30 7.29 7.28 7.28 7.27 7.26 7.25 7.25 7.24 7.10 7.09 7.09 7.07 7.05 7.03 7.03 7.01 7.01 6.99 6.99
5.38 5.38 5.06 5.05 4.73 4.70 3.92 3.88 3.55 3.54 3.53 3.52 3.51 3.50 3.49 3.48 3.45 3.43 3.34 3.30 2.69 2.66 2.63 2.34 2.29 2.28 2.25 2.24 2.22 2.21 2.04 2.01 1.99 1.48 1.47 1.45 1.44 1.43 1.36 1.35 1.34 1.31 1.30 1.28 1.28 1.27 1.26 1.24 0.80 0.77
Solvent CDCl3
Spectrometer Frequency 399.44
Nucleus 1H
H
Ph
H
H
Ts
Se
Ph
N
O
O=S=O
Br
5gb, 20:1 dr
3.85 1.98 2.01 3.11 7.00 1.09 1.09 1.04 1.05 1.13 1.03 1.05 1.12 3.00 1.21 2.15 1.40 2.06 1.09
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

158.72 144.17 139.24 137.27 137.11 135.09 132.20 129.49 129.08 128.98 128.28 128.02 127.78 127.49 127.36 127.13 126.43 109.95 108.93 77.31 76.99 76.67 70.78 65.16 53.22 49.59 42.25 38.34 34.77 32.06 22.61 21.47

Solvent $CDCl_3$
Spectrometer Frequency 100.45
Nucleus ^{13}C



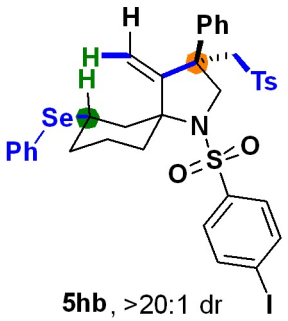
5gb, 20:1 dr



Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



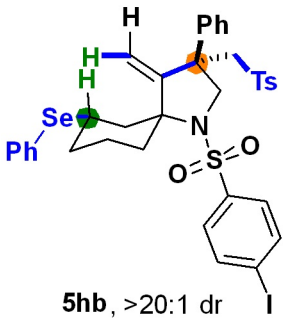
5hb, >20:1 dr

158.76
144.18
139.87
138.19
137.26
137.11
135.11
129.50
129.01
128.94
128.29
127.79
127.50
127.12
126.43
108.89
99.78
77.31
76.99
76.67
70.79
65.18
53.20
49.57
42.27
38.37
34.79
32.03
22.61
21.49

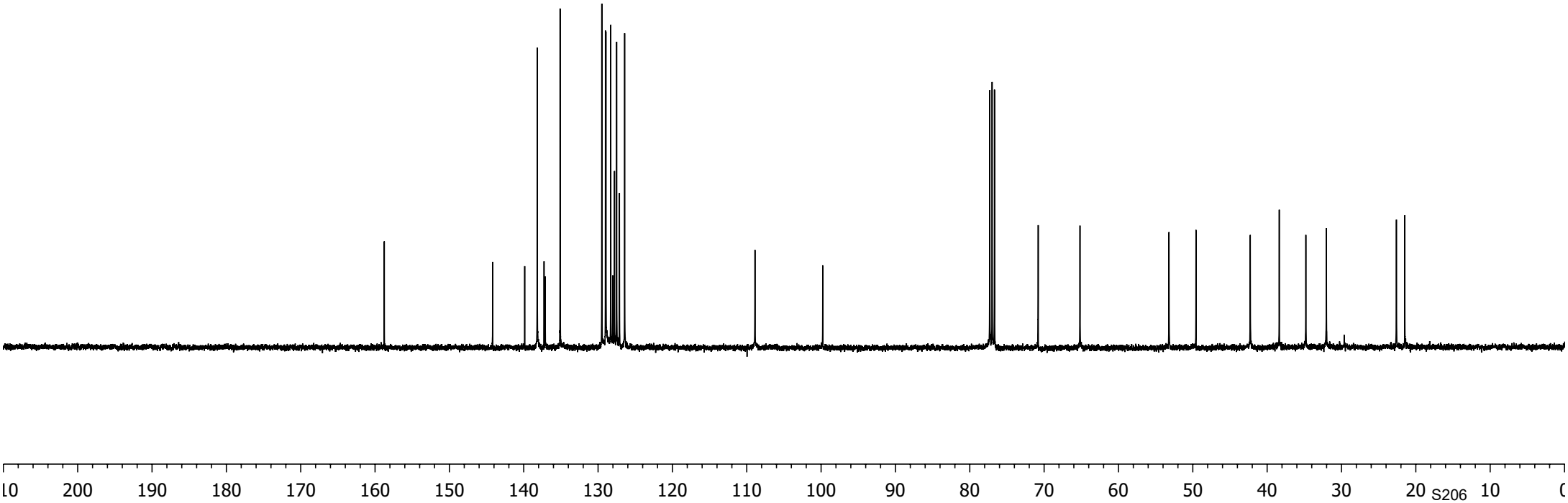
Solvent $CDCl_3$

Spectrometer Frequency 100.45

Nucleus ^{13}C



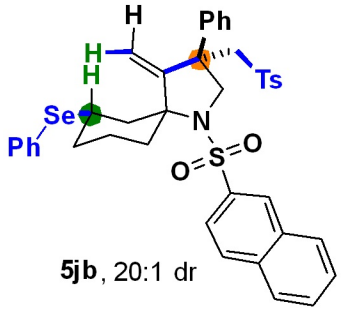
5hb, >20:1 dr



Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



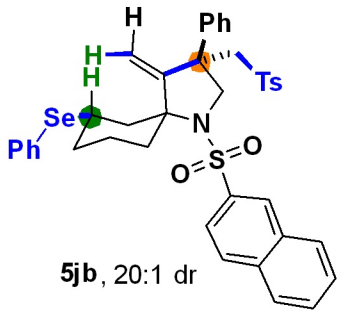
5jb, 20:1 dr

159.14
144.08
135.08
129.50
129.43
129.12
128.86
128.82
128.17
127.78
127.65
127.48
126.95
126.45
123.90
108.71
77.31
76.99
76.67
70.65
65.20
53.21
49.52
42.33
38.58
34.79
32.06
22.66
21.45

Solvent $CDCl_3$

Spectrometer Frequency 100.45

Nucleus ^{13}C

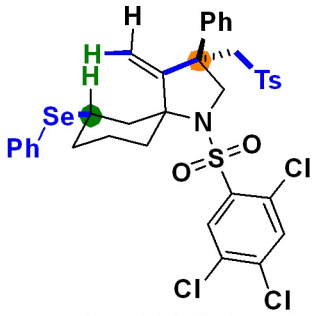


5jb, 20:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



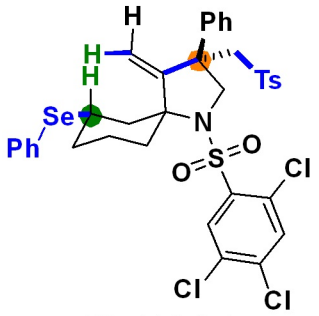
5lb, 14.3:1 dr

158.05
144.37
137.51
137.43
135.26
133.33
133.13
129.60
128.96
128.30
127.81
127.49
127.34
126.42
110.23
77.31
76.99
76.67
72.01
64.50
53.51
50.26
42.59
37.82
34.05
32.14
22.61
21.52

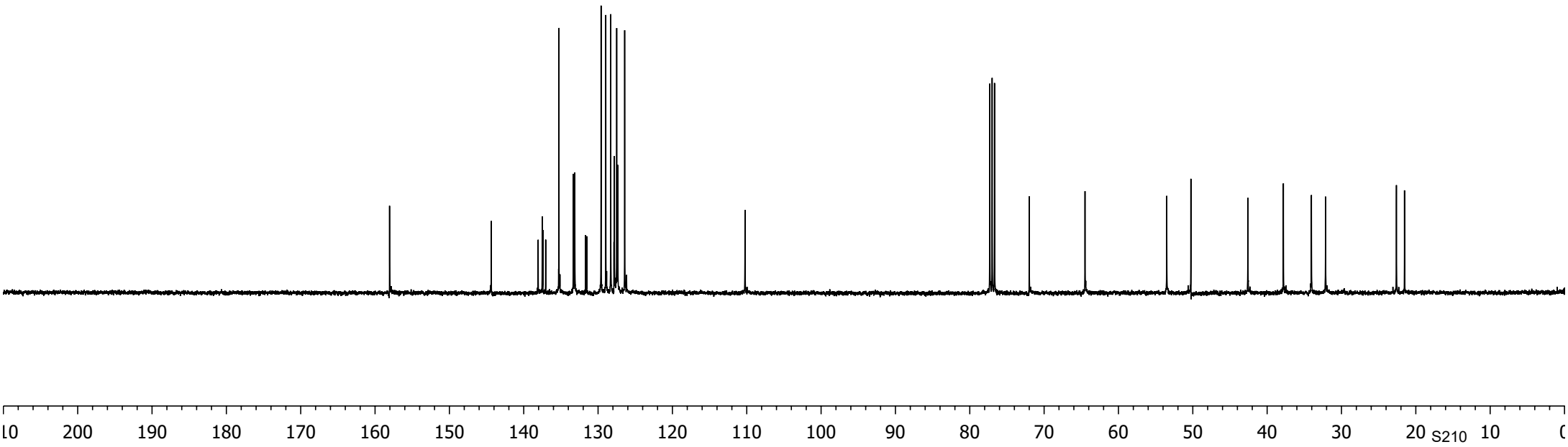
Solvent $CDCl_3$

Spectrometer Frequency 100.45

Nucleus ^{13}C



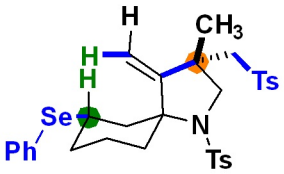
5lb, 14.3:1 dr



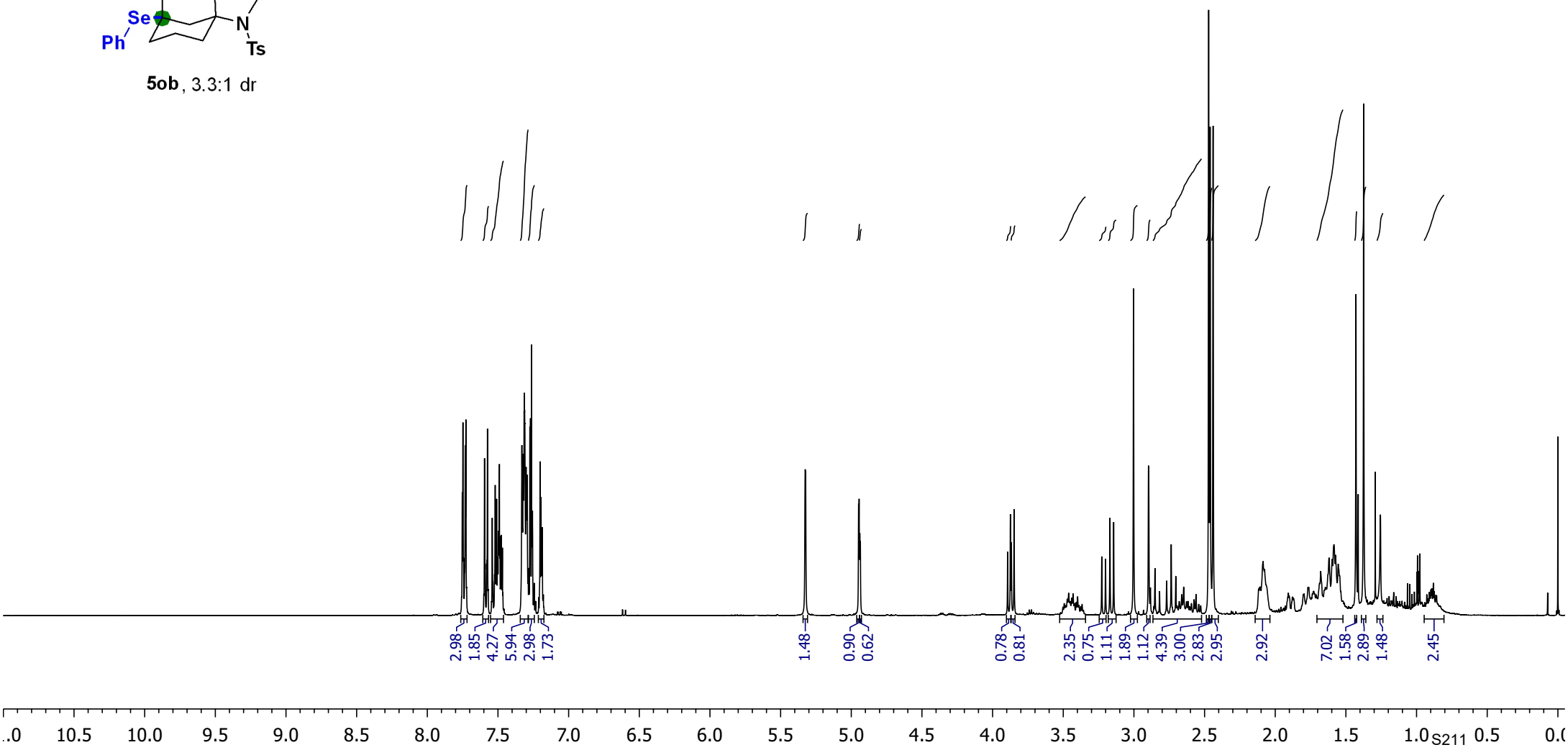
Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



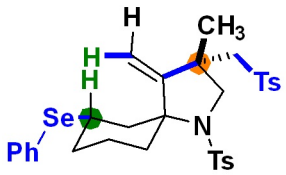
5ob, 3.3:1 dr



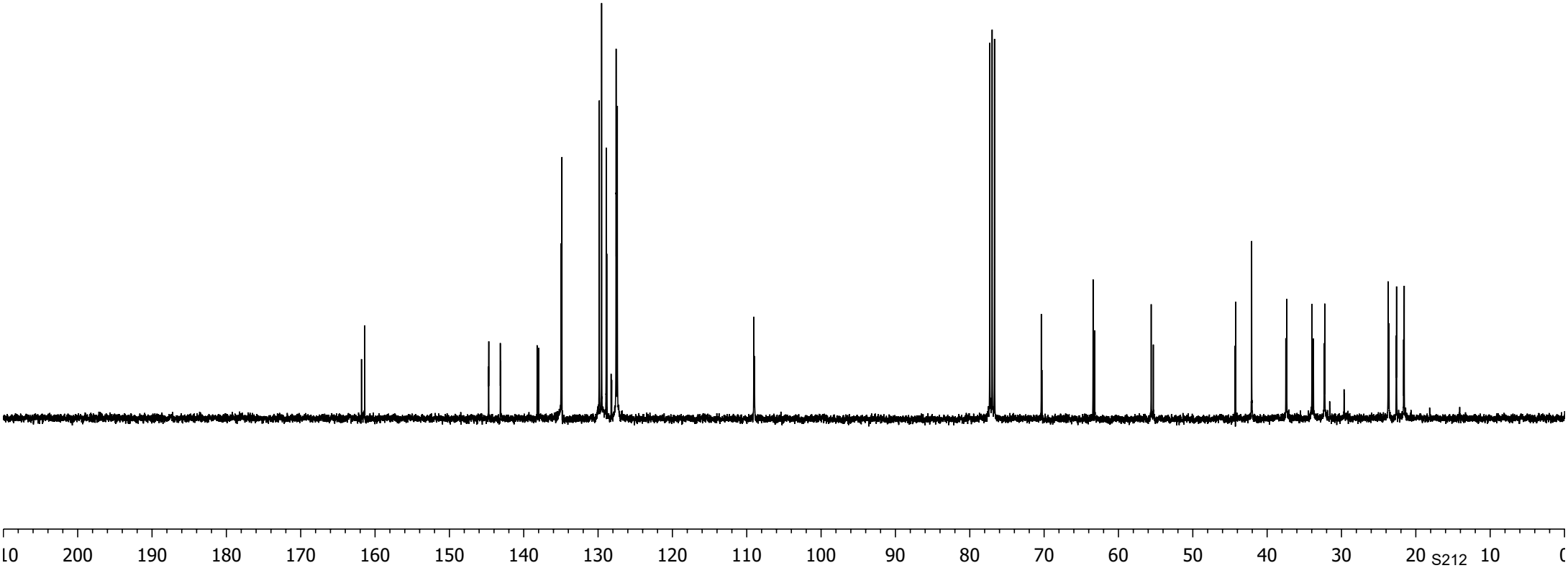
Solvent $CDCl_3$

Spectrometer Frequency 100.45

Nucleus ^{13}C



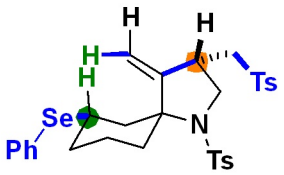
5ob, 3.3:1 dr



Solvent $CDCl_3$

Spectrometer Frequency 399.44

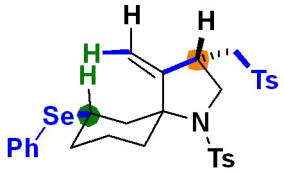
Nucleus ^1H



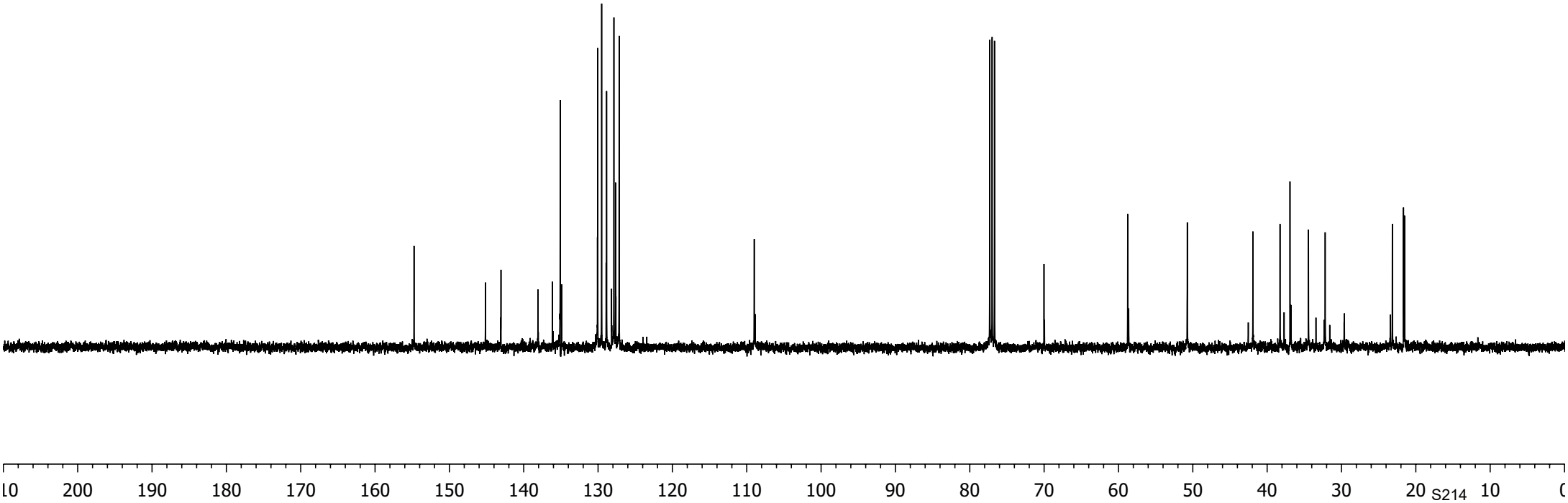
5pb, 1.5:1 dr

154.76
145.14
143.06
136.15
135.09
134.89
130.08
130.04
129.52
128.92
128.85
128.19
127.87
127.64
127.17
109.00
77.31
76.99
76.67
70.01
58.76
50.74
41.91
38.27
36.93
34.44
32.19
23.16
21.66
21.51

Solvent $CDCl_3$
Spectrometer Frequency 100.45
Nucleus ^{13}C



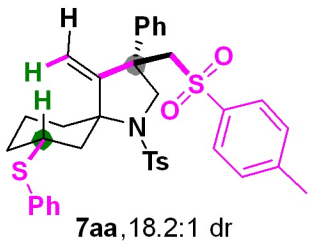
5pb, 1.5:1 dr



Solvent $CDCl_3$

Spectrometer Frequency 400.40

Nucleus ^1H



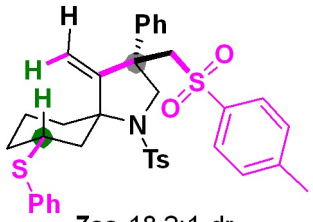
7aa,18.2:1 dr

159.51
144.11
143.18
137.44
137.23
132.58
129.54
129.47
128.81
128.21
127.67
127.54
127.21
126.98
126.52
108.55
77.31
76.99
76.68
70.28
65.28
53.17
49.43
43.50
41.16
34.95
31.44
21.93
21.54
21.48

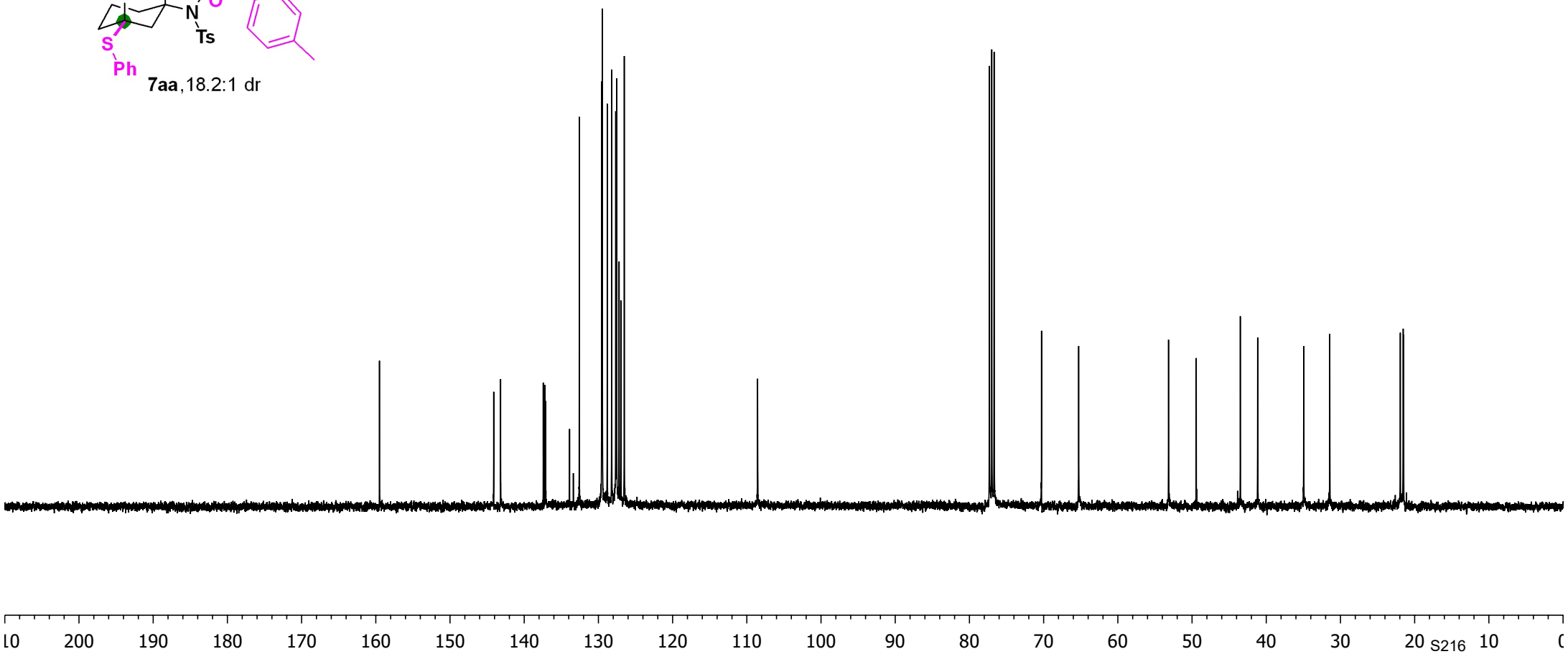
Solvent $CDCl_3$

Spectrometer Frequency 100.69

Nucleus ^{13}C



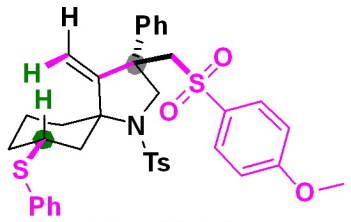
7aa, 18.2:1 dr



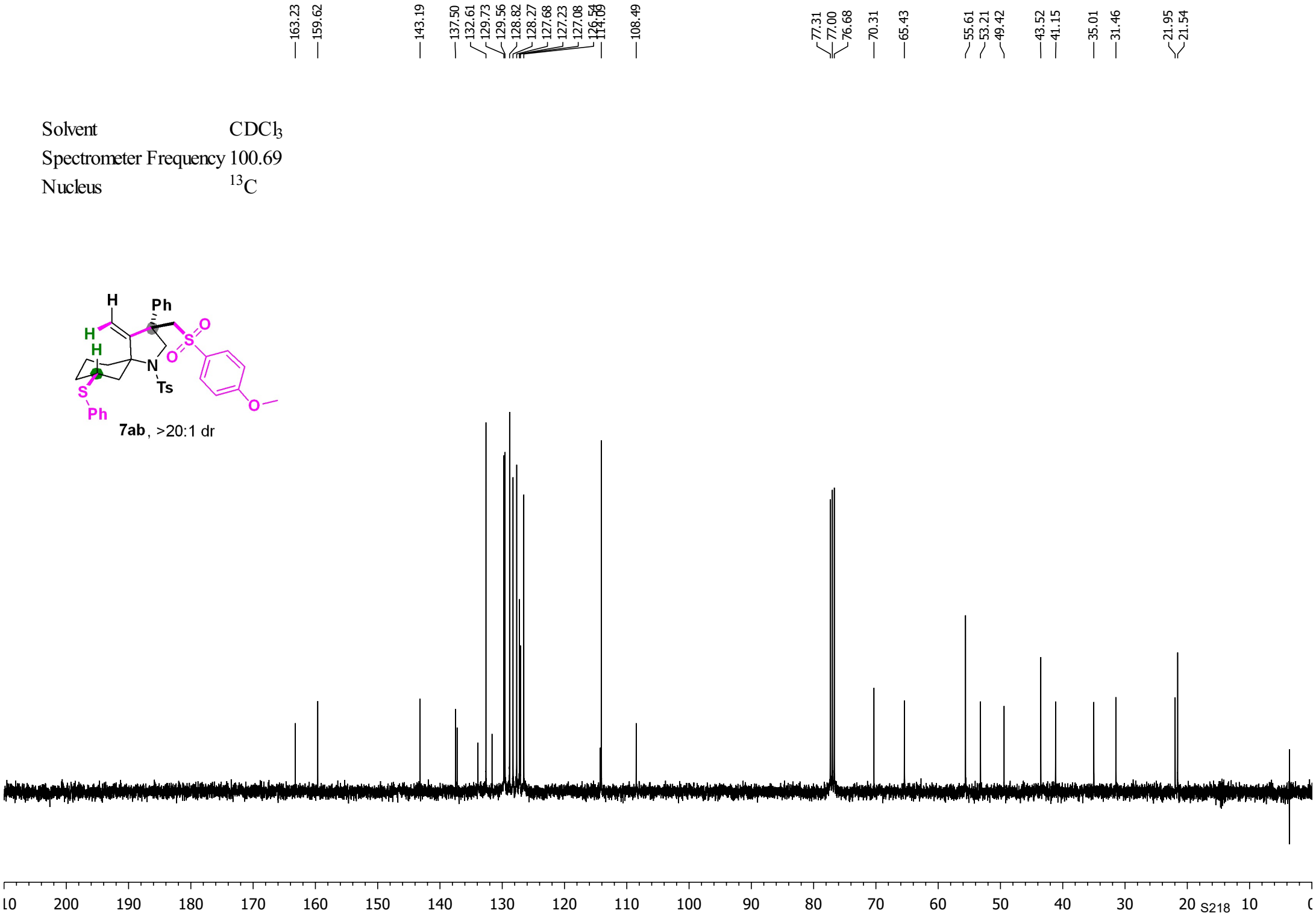
Solvent $CDCl_3$

Spectrometer Frequency 400.40

Nucleus ^1H



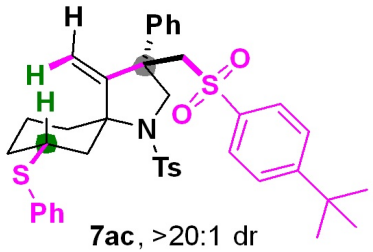
7ab, >20:1 dr


163.23
159.62
143.19
137.50
132.61
129.73
129.56
128.82
128.27
127.68
127.23
127.08
126.54
114.00
108.49
77.31
77.00
76.68
70.31
65.43
55.61
53.21
49.42
43.52
41.15
35.01
31.46
21.95
21.54
Solvent CDCl3
Spectrometer Frequency 100.69
Nucleus 13C
H
Ph
H
H
S
O
O
N
Ts
S
Ph
O
7ab, >20:1 dr
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

Solvent $CDCl_3$

Spectrometer Frequency 400.40

Nucleus ^1H



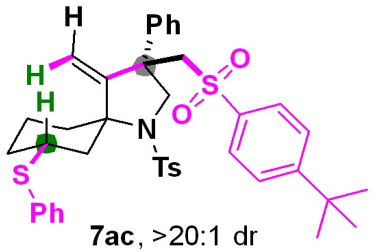
7ac, >20:1 dr

159.73
156.79
143.18
137.24
136.83
133.88
132.63
129.57
128.82
128.17
127.69
127.33
127.24
127.11
126.51
125.84
108.36
77.31
76.99
76.68
70.30
65.20
53.18
49.29
43.56
41.15
35.04
31.43
30.94
21.95
21.55

Solvent $CDCl_3$

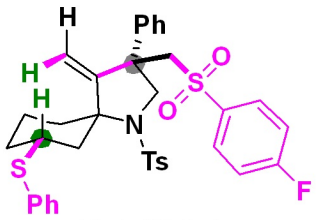
Spectrometer Frequency 100.69

Nucleus ^{13}C



7ac, >20:1 dr

Solvent	$CDCl_3$
Spectrometer Frequency	400.40
Nucleus	^1H



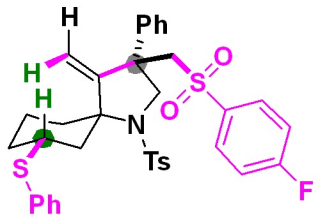
7ad, 20:1 dr

166.48
163.93
159.49
143.27
137.12
135.73
132.67
129.60
128.82
128.35
127.67
126.55
126.53
116.17
115.94
108.47
77.31
76.99
76.67
70.33
70.30
65.46
65.43
53.18
49.28
44.32
43.55
41.12
35.08
31.42
21.93
21.55

Solvent $CDCl_3$

Spectrometer Frequency 100.69

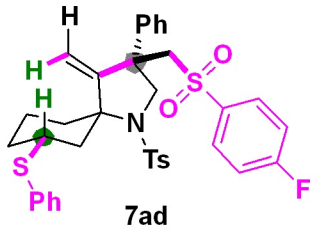
Nucleus ^{13}C



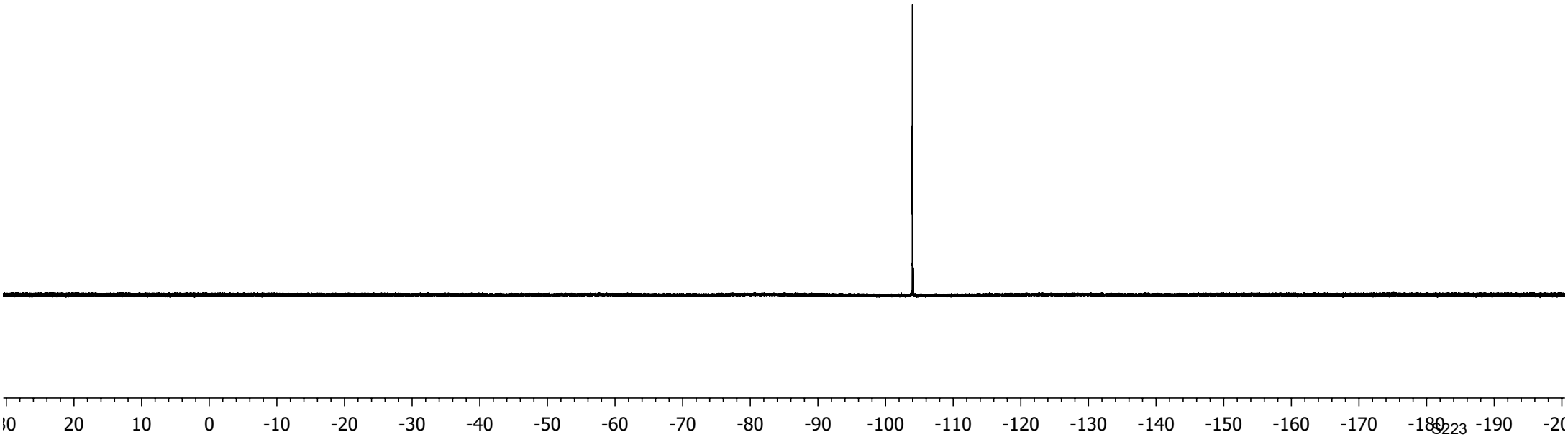
7ad, 20:1 dr

-103.98

Solvent	$CDCl_3$
Spectrometer Frequency	376.61
Nucleus	^{19}F



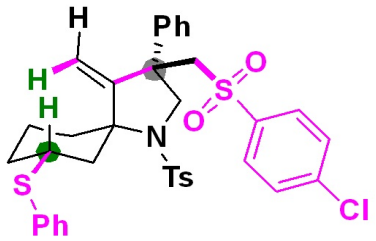
7ad



Solvent $CDCl_3$

Spectrometer Frequency 400.40

Nucleus ^1H



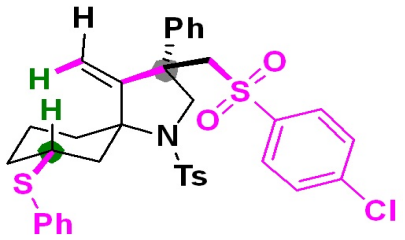
7ae, 5.7:1 dr

159.54 143.29 139.86 138.31 137.19 137.11 133.83 132.71 129.62 129.09 129.00 128.84 128.45 128.40 127.69 127.30 127.27 126.56 108.45 77.31 77.19 76.99 76.67 70.36 65.47 53.17 49.25 43.58 41.13 35.11 31.43 21.96 21.56

Solvent $CDCl_3$

Spectrometer Frequency 100.69

Nucleus ^{13}C

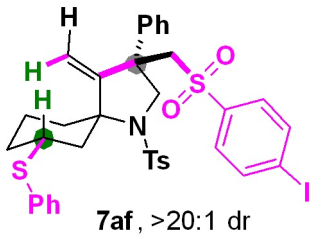


7ae, 5.7:1 dr

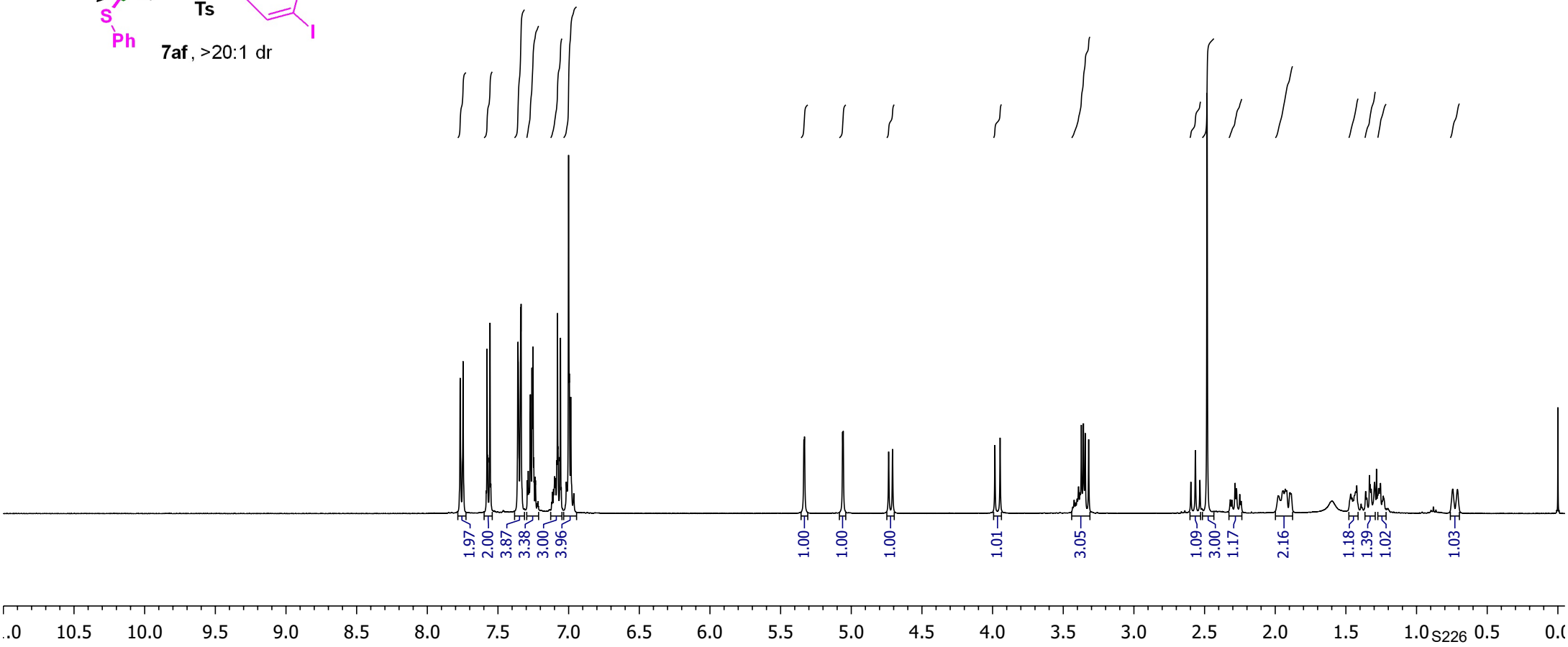
Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H

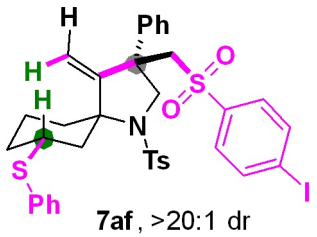


7af, >20:1 dr



159.51
143.27
139.40
138.04
137.17
137.08
133.81
132.66
129.60
128.82
128.41
127.66
127.28
127.18
126.52
108.45
101.15
77.31
76.99
76.67
70.34
65.35
53.11
49.19
43.54
41.12
35.08
31.41
21.93
21.56

Solvent $CDCl_3$
Spectrometer Frequency 100.45
Nucleus ^{13}C

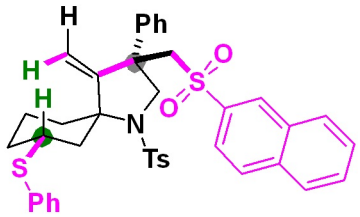


7af, >20:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H



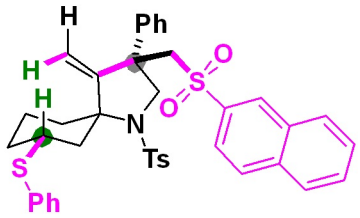
7ah, 14:1 dr

159.59
143.20
132.64
129.82
129.57
129.43
129.21
129.17
128.82
128.04
127.69
127.38
127.25
126.41
121.93
108.44
77.31
76.99
76.67
70.35
65.16
53.22
49.40
43.56
41.16
35.03
31.45
21.95
21.55

Solvent $CDCl_3$

Spectrometer Frequency 100.45

Nucleus ^{13}C

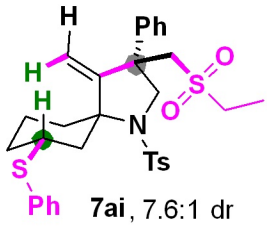


7ah, 14:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



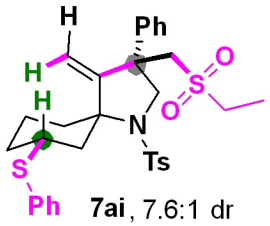
7ai, 7.6:1 dr

159.28
143.21
137.64
137.18
133.79
132.73
129.59
128.88
128.84
127.96
127.66
127.33
126.57
108.71
77.31
77.19
76.99
76.68
70.34
61.66
52.83
49.26
48.65
43.71
41.21
35.02
31.45
21.98
21.54
6.34

Solvent $CDCl_3$

Spectrometer Frequency 100.66

Nucleus ^{13}C

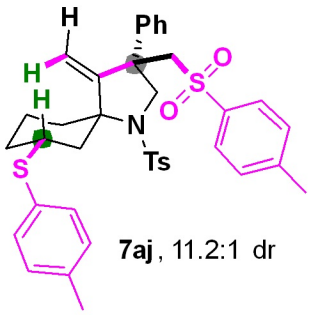


7ai, 7.6:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 399.44

Nucleus ^1H

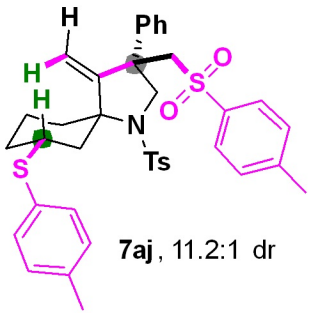


7aj, 11.2:1 dr

Solvent $CDCl_3$

Spectrometer Frequency 100.45

Nucleus ^{13}C



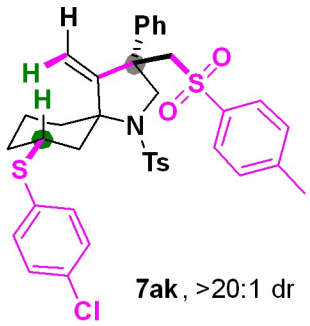
7aj, 11.2:1 dr

159.57
144.10
143.16
137.52
137.45
137.29
133.40
129.60
129.56
129.47
128.22
127.70
127.56
126.96
77.31
76.99
76.67
70.36
65.35
53.18
49.38
43.89
41.17
35.03
31.44
21.94
21.55
21.48
21.12

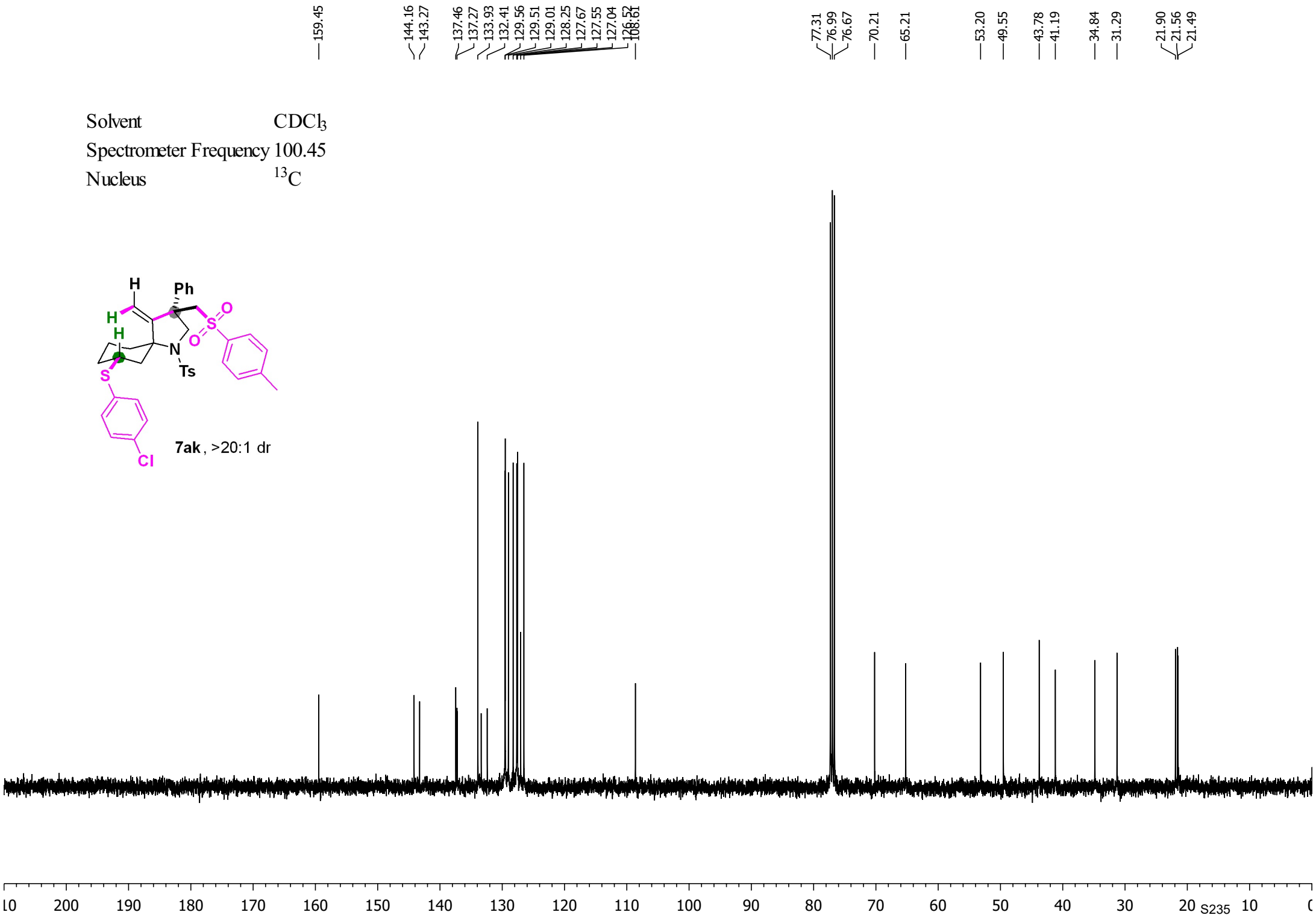
Solvent $CDCl_3$

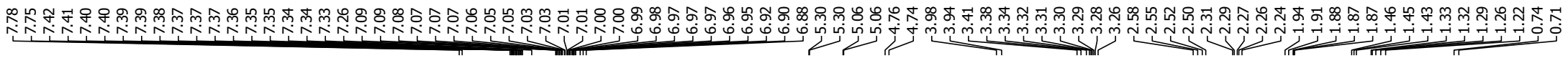
Spectrometer Frequency 399.44

Nucleus ^1H



7ak, >20:1 dr

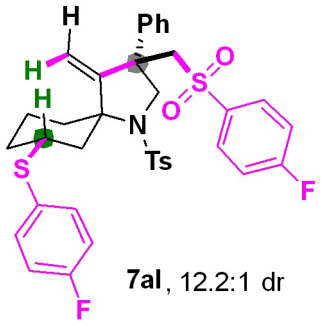

159.45
144.16
143.27
137.46
137.27
133.93
132.41
129.56
129.51
129.01
128.25
127.67
127.55
127.04
126.52
108.61
77.31
76.99
76.67
70.21
65.21
53.20
49.55
43.78
41.19
34.84
31.29
21.90
21.56
21.49
Solvent CDCl3
Spectrometer Frequency 100.45
Nucleus 13C
H
Ph
H
H
O
S
O
N
Ts
S
Cl
7ak, >20:1 dr
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10



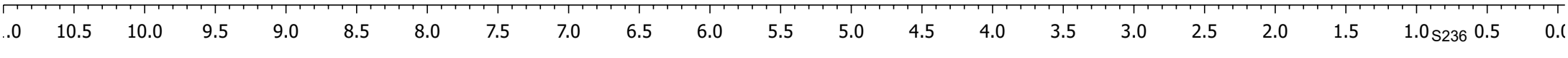
Solvent $CDCl_3$

Spectrometer Frequency 400.40

Nucleus ^1H



7al, 12.2:1 dr

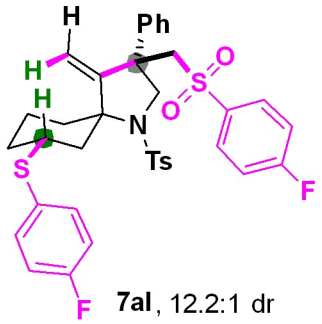


166.48 163.94 163.73 161.27 159.42 143.31 137.10 135.72 135.64 129.59 128.34 127.67 116.18 116.04 115.95 115.83 108.50 77.31 76.99 76.67 70.30 65.42 53.18 49.32 44.32 41.12 34.96 31.31 21.86 21.55

Solvent $CDCl_3$

Spectrometer Frequency 100.69

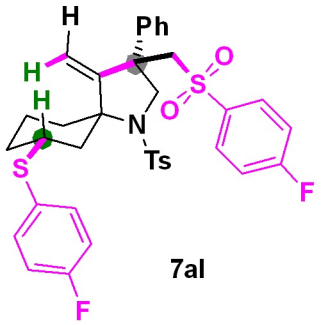
Nucleus ^{13}C



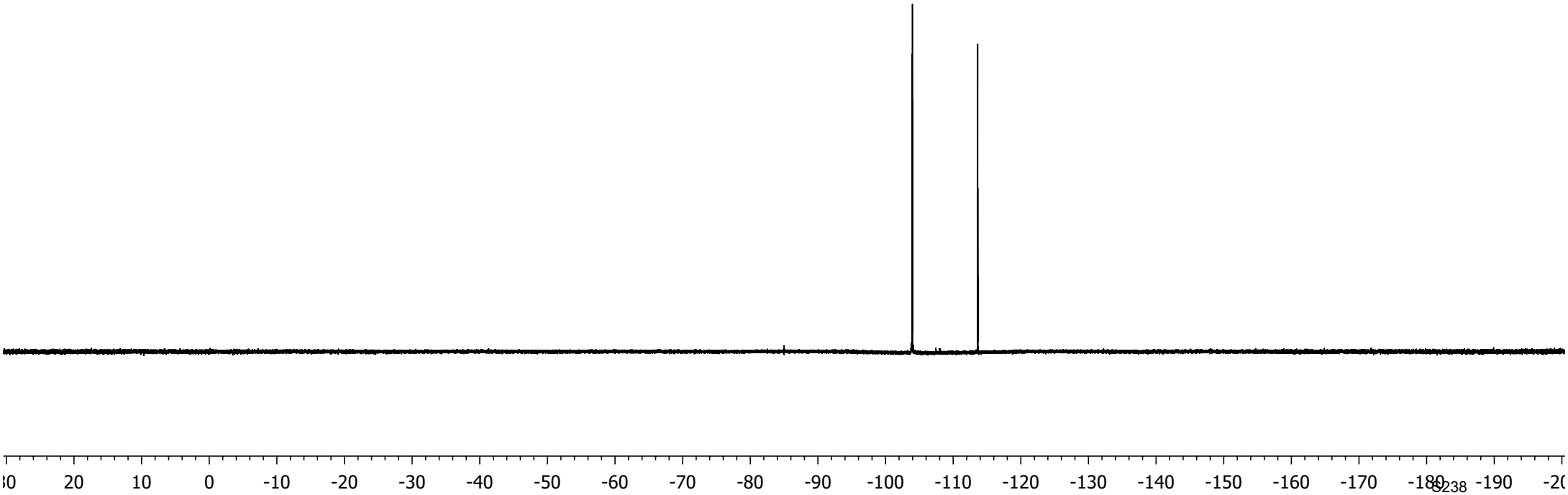
7al, 12.2:1 dr

-103.97
-113.63

Solvent $CDCl_3$
Spectrometer Frequency 376.61
Nucleus ^{19}F



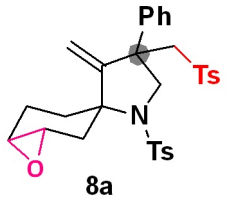
7al



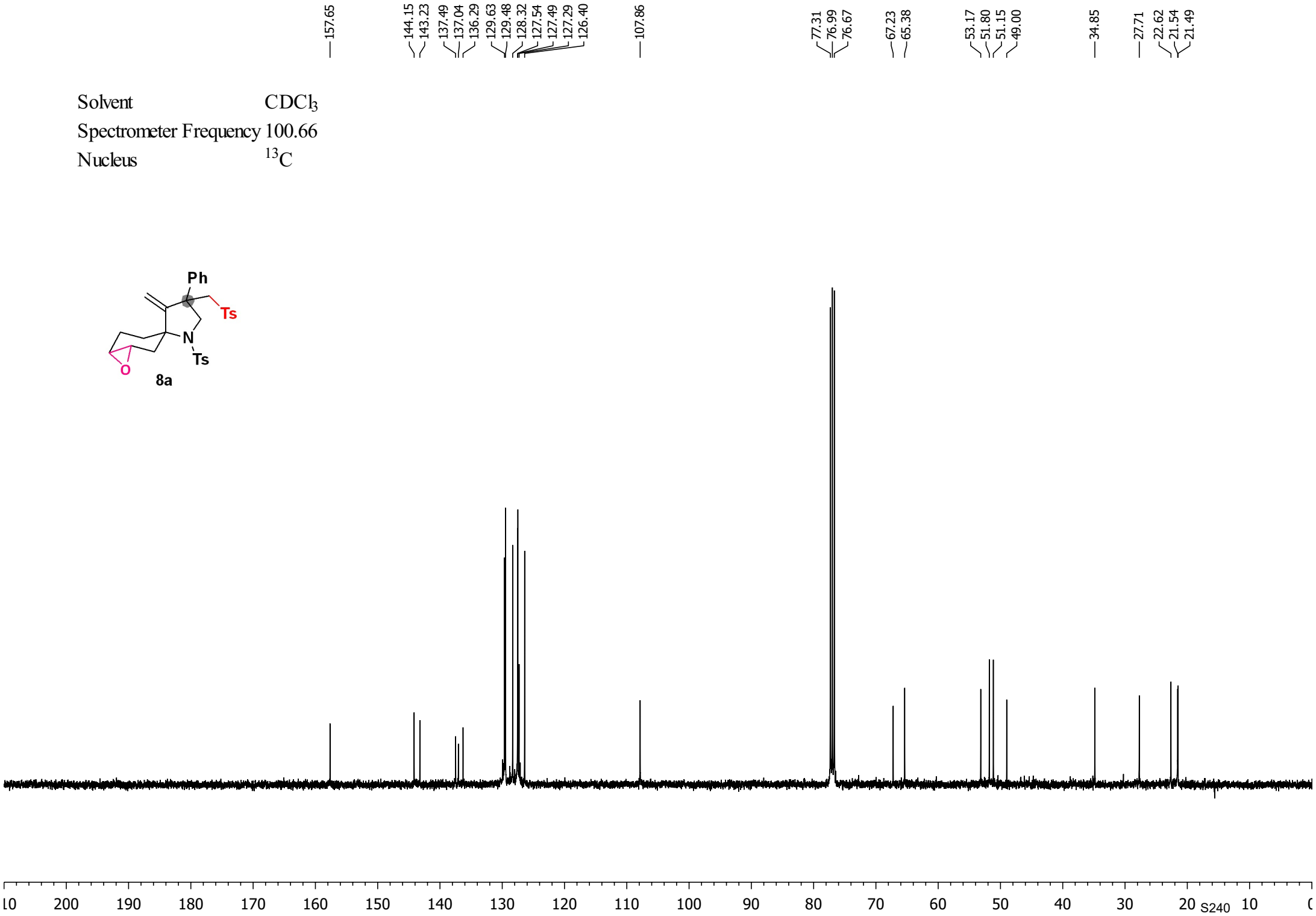
Solvent $CDCl_3$

Spectrometer Frequency 400.28

Nucleus ^1H



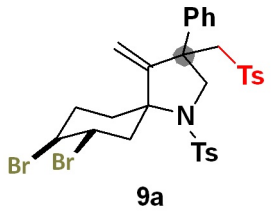
8a


157.65
144.15
143.23
137.49
137.04
136.29
129.63
129.48
128.32
127.54
127.49
127.29
126.40
107.86
77.31
76.99
76.67
67.23
65.38
53.17
51.80
51.15
49.00
34.85
27.71
22.62
21.54
21.49
Solvent CDCl3
Spectrometer Frequency 100.66
Nucleus 13C
Ph
Ts
N
Ts
O
8a
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

Solvent $CDCl_3$

Spectrometer Frequency 400.28

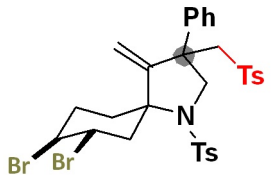
Nucleus ^1H



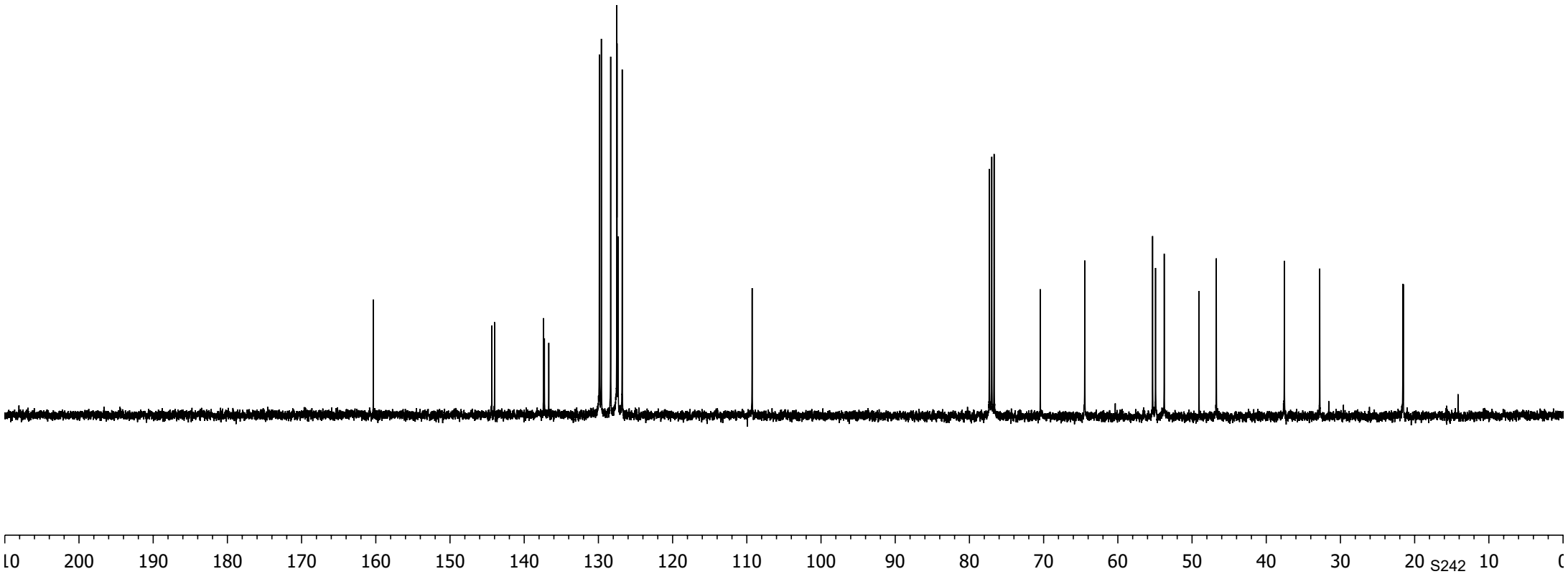
9a

160.33
144.36
143.99
137.40
137.28
136.69
129.87
129.60
128.35
127.52
127.49
127.35
126.79
109.28
77.31
76.99
76.67
70.45
64.46
55.32
54.92
53.75
49.07
46.73
37.56
32.79
21.59
21.53

Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C



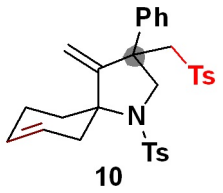
9a



Solvent $CDCl_3$

Spectrometer Frequency 400.28

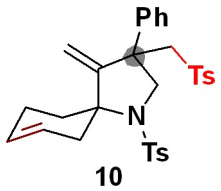
Nucleus ^1H



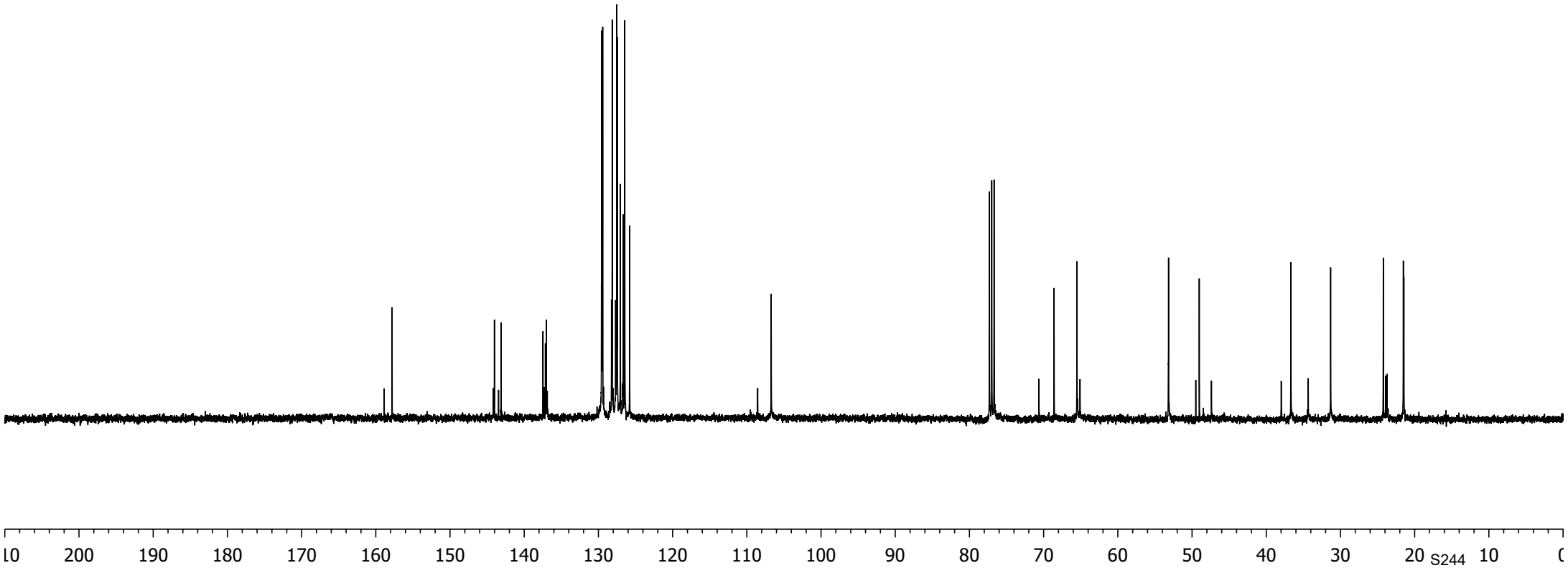
10

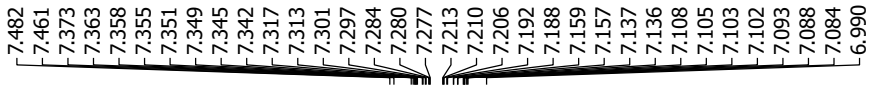
157.80
144.01
143.11
137.47
137.14
137.03
129.55
129.42
128.14
127.53
127.46
127.04
126.66
126.45
125.79
106.71
77.31
76.99
76.68
68.59
65.51
53.17
49.03
36.68
31.32
24.22
21.53
21.46

Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C



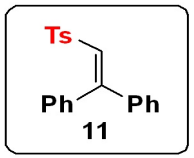
10

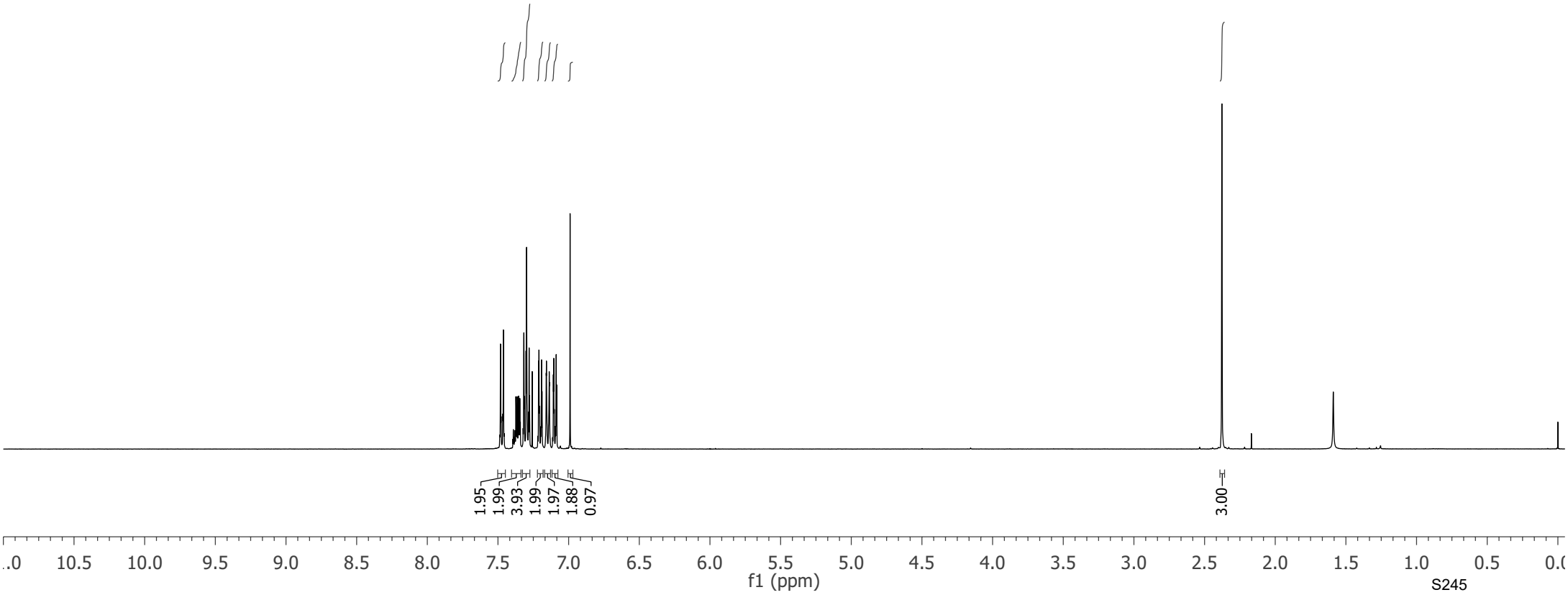




Solvent $CDCl_3$

Spectrometer Frequency 400.40

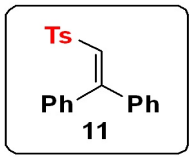




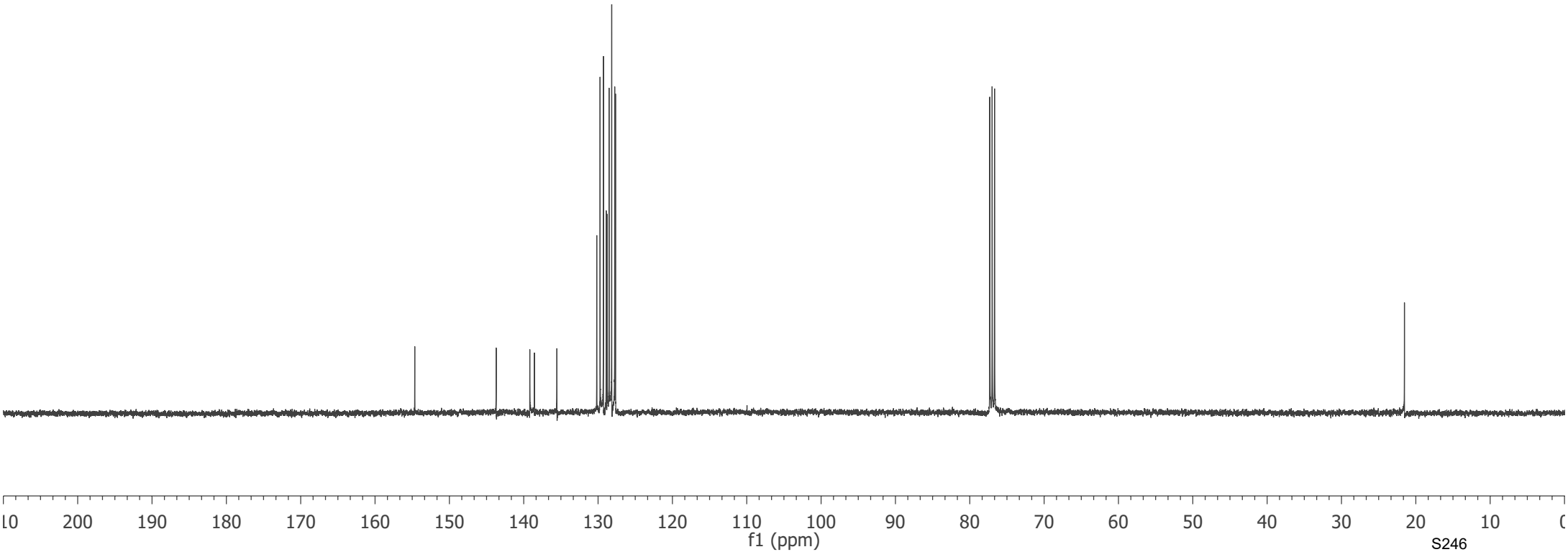
154.7
143.7
139.2
138.6
135.5
130.2
129.7
129.3
128.9
128.8
128.5
128.2
127.8
127.7
77.3
77.0
76.7
21.5

Solvent $CDCl_3$

Spectrometer Frequency 100.69



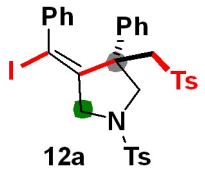
11

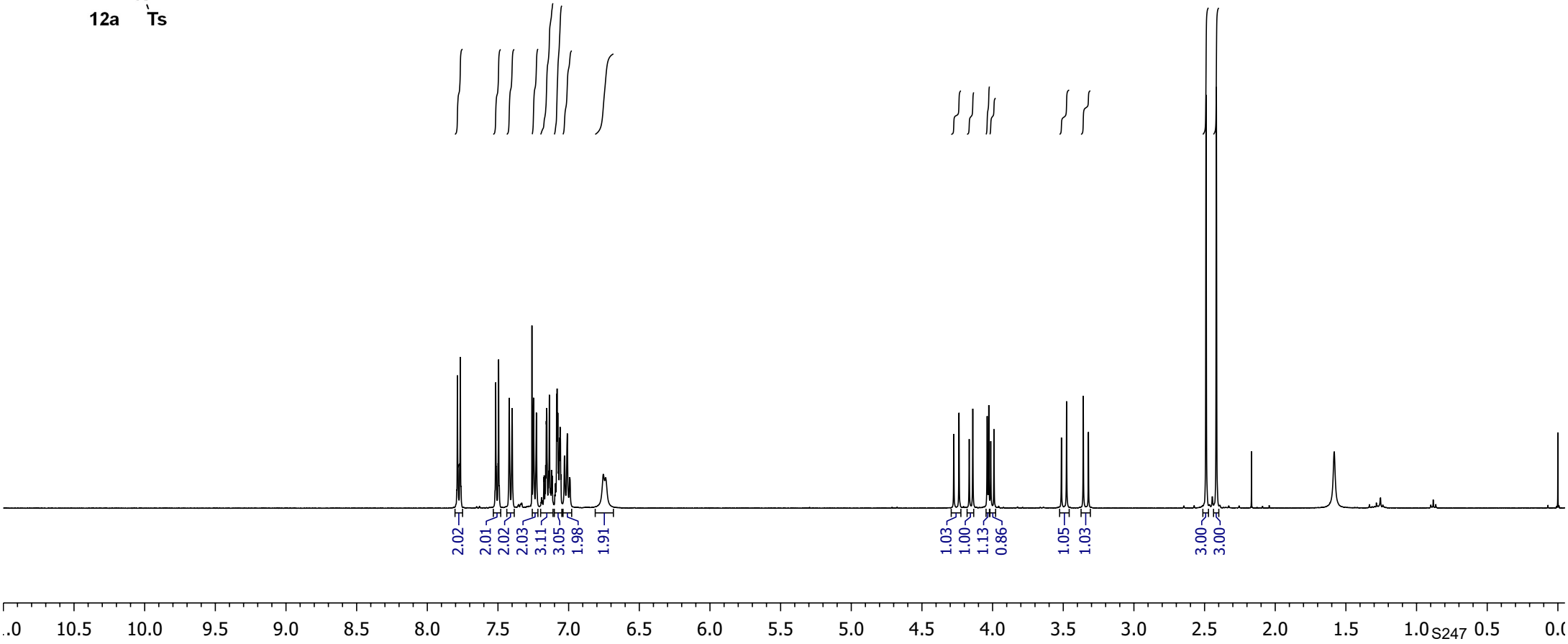


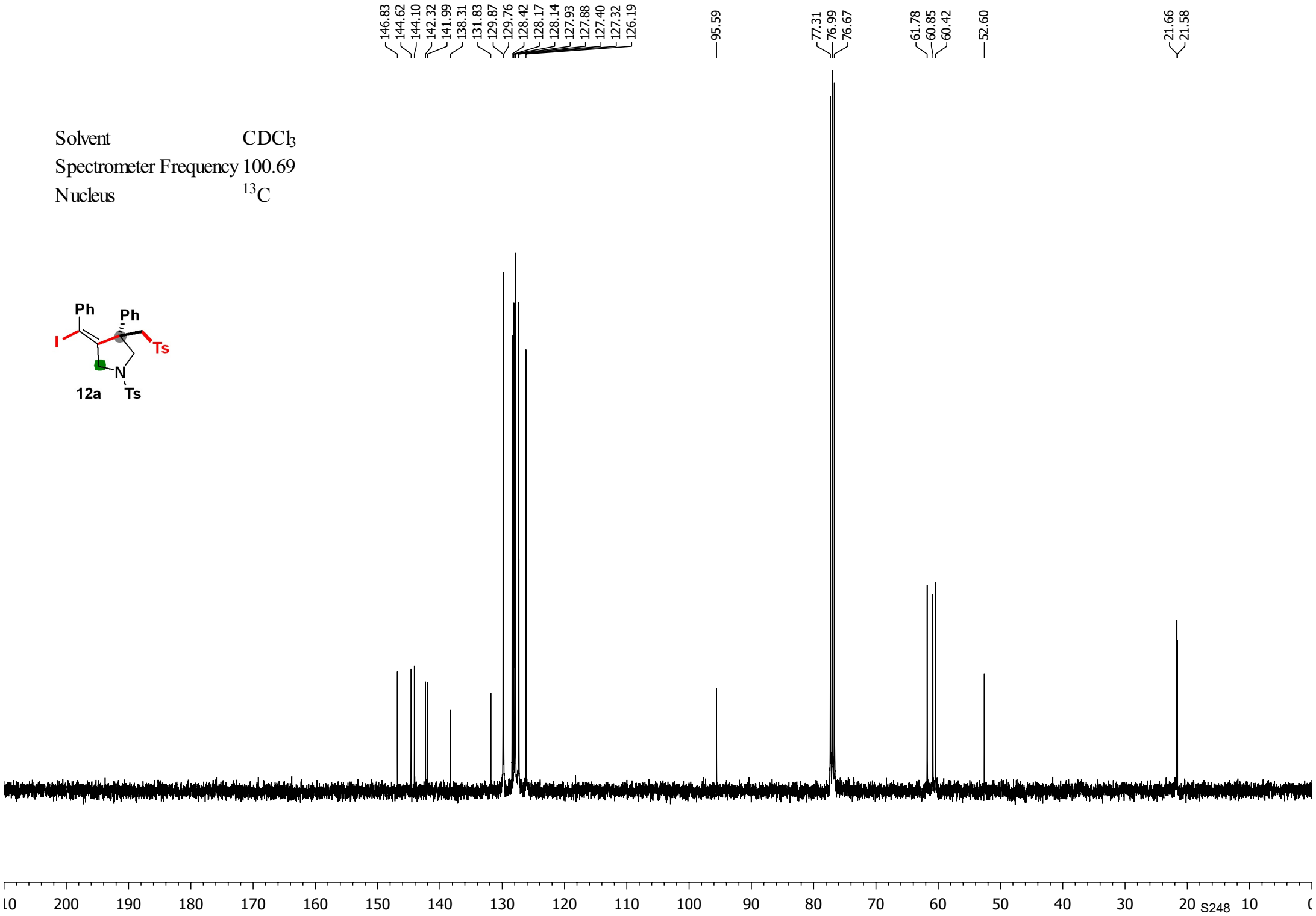
Solvent $CDCl_3$

Spectrometer Frequency 400.40

Nucleus ^1H






146.83
144.62
144.10
142.32
141.99
138.31
131.83
129.87
129.76
128.42
128.17
128.14
127.93
127.88
127.40
127.32
126.19
95.59
77.31
76.99
76.67
61.78
60.85
60.42
52.60
21.66
21.58
Solvent CDCl3
Spectrometer Frequency 100.69
Nucleus 13C
Ph
Ph
I
Ts
N
12a
Ts
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) shelx

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: shelx

```
Bond precision:   C-C = 0.0061 A          Wavelength=0.71073

Cell:             a=12.2241(14)     b=15.1783(19)     c=23.332(3)
                  alpha=90          beta=90           gamma=90
Temperature:      298 K

                 Calculated                Reported
Volume           4329.0(9)                 4329.1(9)
Space group      P b c a                   P b c a
Hall group       -P 2ac 2ab                -P 2ac 2ab
Moiety formula   C24 H27 N O2 S            C24 H27 N O2 S
Sum formula      C24 H27 N O2 S            C24 H27 N O2 S
Mr               393.53                    393.52
Dx,g cm-3        1.208                     1.208
Z                8                         8
Mu (mm-1)        0.168                     0.168
F000             1680.0                    1680.0
F000'            1681.63
h,k,lmax         14,18,27                  14,18,27
Nref             3829                      3820
Tmin,Tmax        0.959,0.993               0.936,0.993
Tmin'            0.935

Correction method= # Reported T Limits: Tmin=0.936 Tmax=0.993
AbsCorr = MULTI-SCAN
```

Data completeness= 0.998 Theta(max)= 25.054

R(reflections)= 0.0620(2393) wR2(reflections)= 0.2211(3820)

S = 1.010 Npar= 253

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT919_ALERT_3_B Reflection # Likely Affected by the Beamstop ... 1 Check
1 1 2,

Alert level C

ABSTY02_ALERT_1_C An _exptl_absorpt_correction_type has been given without
a literature citation. This should be contained in the
_exptl_absorpt_process_details field.
Absorption correction given as multi-scan
PLAT242_ALERT_2_C Low MainResAtom Ueq as Compared to Neighbours C1 Check
PLAT242_ALERT_2_C Low MainResAtom Ueq as Compared to Neighbours C4 Check
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00608 Ang.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 3.343 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.596 8 Report
0 4 0, 10 13 3, 9 7 19, 8 2 23, 5 8 23, 1 9 24,
4 4 26, 3 2 27,
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 1 Check
1 1 2,
PLAT934_ALERT_3_C Number of (Iobs-Icalc)/Sigma(W) > 10 Outliers .. 1 Check
1 1 2,
PLAT939_ALERT_3_C Large Value of Not (SHELXL) Weight Optimized S . 73.25 Check

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Alert level G

PLAT066_ALERT_1_G Predicted and Reported Tmin&Tmax Range Identical ? Check
PLAT380_ALERT_4_G Incorrectly? Oriented X(sp2)-Methyl Moiety ..... C5 Check
PLAT883_ALERT_1_G Absent Datum for _atom_sites_solution_primary .. Please Do !
PLAT910_ALERT_3_G Missing FCF Reflection(s) Below Theta(Min)[Deg]= 2.31 Note
0 0 2,
PLAT913_ALERT_3_G Missing # of Very Strong Reflections in FCF .... 1 Note
0 4 0,
PLAT965_ALERT_2_G The SHELXL WEIGHT Optimisation has not Converged Please Check
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ..... 3.732 Note
Predicted wR2: Based on SigI**2 5.93 or SHELX Weight 21.89
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 0 Info

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **ALERT level B** = A potentially serious problem, consider carefully
9 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
8 **ALERT level G** = General information/check it is not something unexpected

```
  3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
  4 ALERT type 2 Indicator that the structure model may be wrong or deficient
  9 ALERT type 3 Indicator that the structure quality may be low
  1 ALERT type 4 Improvement, methodology, query or suggestion
  1 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

PLATON version of 15/01/2026; check.def file version of 02/01/2026

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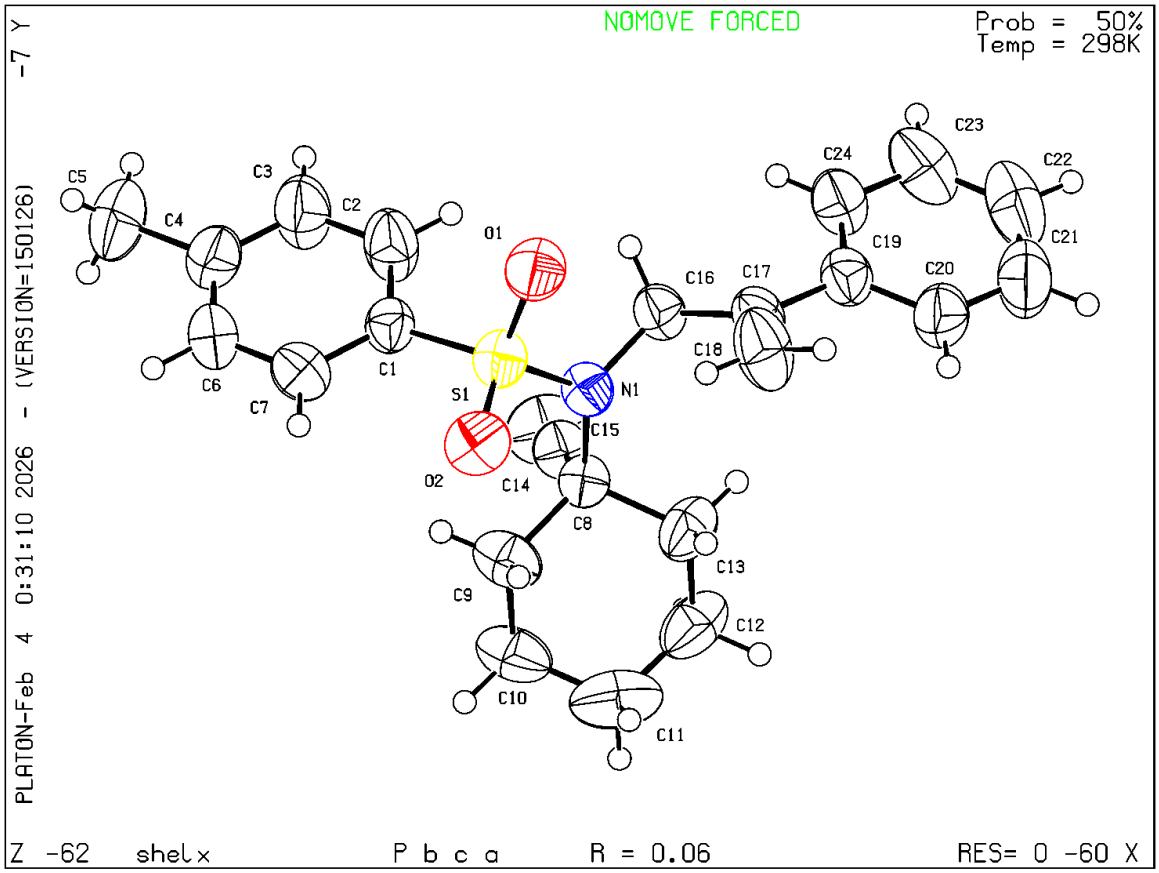
duplicate check

No duplication found

Datablock shelx - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) shelx

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: shelx

```
Bond precision:   C-C = 0.0055 A          Wavelength=0.71073

Cell:           a=8.8033(5)       b=10.9603(5)      c=16.1910(9)
                alpha=71.565(2)   beta=84.701(2)    gamma=85.036(2)
Temperature:    298 K

                Calculated                Reported
Volume          1473.00(14)               1473.00(14)
Space group     P -1                      P -1
Hall group      -P 1                      -P 1
Moiety formula  C31 H34 I N O4 S2         C31 H34 I N O4 S2
Sum formula     C31 H34 I N O4 S2         C31 H34 I N O4 S2
Mr              675.61                    675.61
Dx,g cm-3       1.523                     1.523
Z               2                         2
Mu (mm-1)       1.266                     1.266
F000            688.0                     688.0
F000'           687.80
h,k,lmax        10,13,19                  10,13,19
Nref            5210                      5199
Tmin,Tmax       0.892,0.963               0.895,0.963
Tmin'           0.892

Correction method= # Reported T Limits: Tmin=0.895 Tmax=0.963
AbsCorr = MULTI-SCAN
```

Data completeness= 0.998 Theta(max)= 25.042

R(reflections)= 0.0378(4153) wR2(reflections)= 0.1149(5199)

S = 0.987 Npar= 354

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
ABSTY02_ALERT_1_C  An _exptl_absorpt_correction_type has been given without
           a literature citation. This should be contained in the
           _exptl_absorpt_process_details field.
           Absorption correction given as multi-scan
PLAT410_ALERT_2_C Short Intra H...H Contact  H1       ..H31B     .       1.95 Ang.
                                                     x,y,z  =      1_555 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.596         10 Report
               0  1  1,   1  1  2,   0  2  3,  -6 10  3,   0 13  6,   8  9  8,
               5 -6 11,   7  6 15,  -3  7 17,   1  2 19,
```

Alert level G

```
PLAT066_ALERT_1_G Predicted and Reported Tmin&Tmax Range Identical          ? Check
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)       0.002 Degree
PLAT793_ALERT_4_G Model has Chirality at C1          (Centro SpGr)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C3          (Centro SpGr)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C15         (Centro SpGr)          R Verify
PLAT883_ALERT_1_G Absent Datum for _atom_sites_solution_primary ..     Please Do !
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still        52% Note
PLAT910_ALERT_3_G Missing FCF Reflection(s) Below Theta(Min)[Deg]=       1.96 Note
               0  0  1,
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File          2 Note
               1  1  2,   0  1  1,
PLAT965_ALERT_2_G The SHELXL WEIGHT Optimisation has not Converged     Please Check
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........      2.117 Note
             Predicted wR2: Based on SigI**2  5.43 or SHELX Weight 11.64
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          4 Info
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   3 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  12 ALERT level G = General information/check it is not something unexpected

   4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   4 ALERT type 2 Indicator that the structure model may be wrong or deficient
   3 ALERT type 3 Indicator that the structure quality may be low
   3 ALERT type 4 Improvement, methodology, query or suggestion
   1 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

PLATON version of 15/01/2026; check.def file version of 02/01/2026

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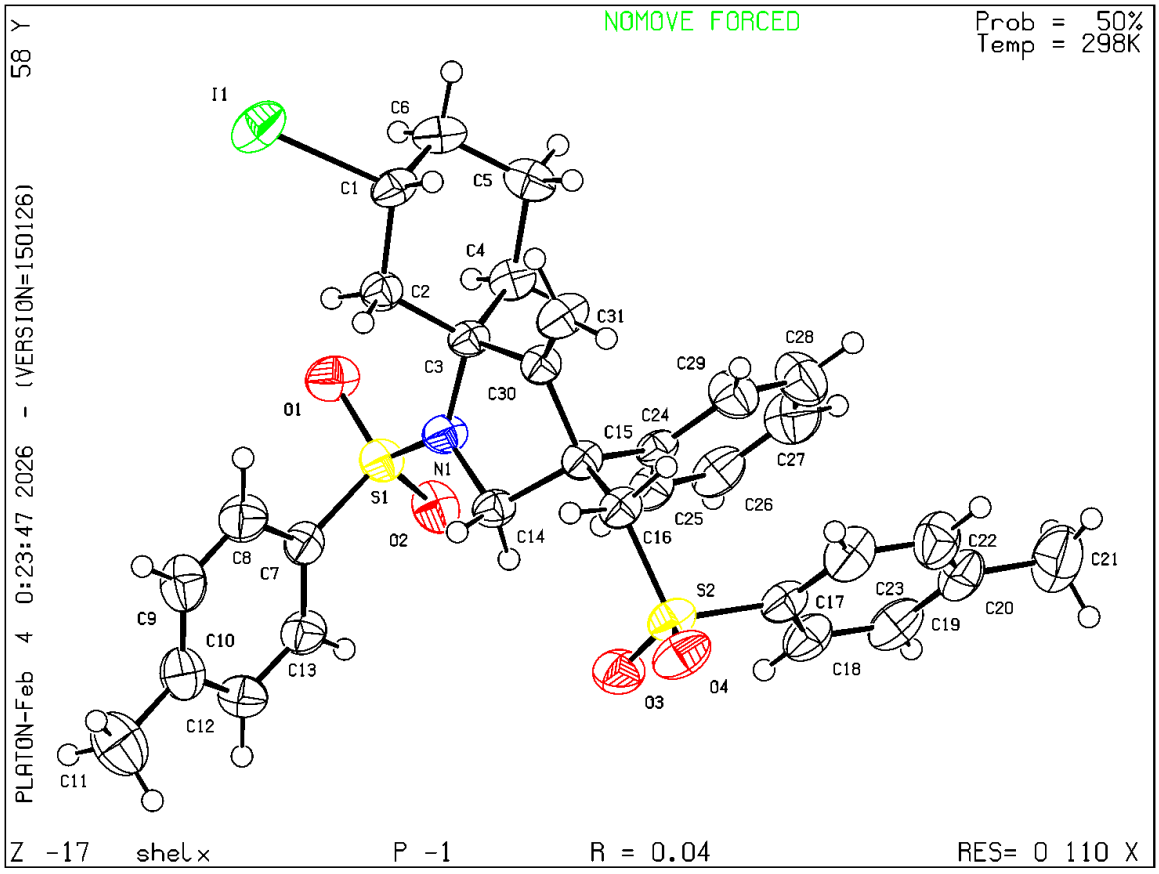
duplicate check

No duplication found

Datablock shelx - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) k11001-jjw-b

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: k11001-jjw-b

```
Bond precision:   C-C = 0.0047 A          Wavelength=0.71073

Cell:             a=8.5625(1)     b=20.6098(2)        c=19.3435(2)
                  alpha=90        beta=98.873(1)      gamma=90
Temperature:      113 K
```

	Calculated	Reported
Volume	3372.72(6)	3372.72(6)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C37 H38 I N O4 S2	C37 H38 I N O4 S2
Sum formula	C37 H38 I N O4 S2	C37 H38 I N O4 S2
Mr	751.70	751.70
Dx,g cm-3	1.480	1.480
Z	4	4
Mu (mm-1)	1.114	1.114
F000	1536.0	1536.0
F000'	1535.65	
h,k,lmax	10,26,24	10,26,24
Nref	7404	7155
Tmin,Tmax	0.765,0.800	0.625,1.000
Tmin'	0.757	

```
Correction method= # Reported T Limits: Tmin=0.625 Tmax=1.000
AbsCorr = MULTI-SCAN
```

Data completeness= 0.966 Theta(max)= 27.054

R(reflections)= 0.0436(6158) wR2(reflections)= 0.1145(7155)

S = 1.045 Npar= 408

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT220_ALERT_2_C NonSolvent   Resd 1  C   Ueq(max)/Ueq(min) Range        3.1 Ratio
PLAT410_ALERT_2_C Short Intra H...H Contact  H3A      ..H8       .       1.99 Ang.
                                                    x,y,z  =      1_555 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      2.759 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600          2 Report
                9  0  6,   9  1  7,
PLAT971_ALERT_2_C Check Calcd Resid. Dens.  1.02Ang From I1              2.13 eA-3
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  0.84Ang From O4      .       0.45 eA-3
```

Alert level G

```
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT  Unusually Large       8.44 Why ?
PLAT142_ALERT_4_G s.u. on b - Axis Small or Missing ..............    0.00020 Ang.
PLAT793_ALERT_4_G Model has Chirality at C1          (Centro SpGr)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C5          (Centro SpGr)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C15         (Centro SpGr)          R Verify
PLAT910_ALERT_3_G Missing FCF Reflection(s) Below Theta(Min)[Deg]=       1.98 Note
                0  1  1,
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        246 Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File          1 Note
                0  1  1,
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........      5.254 Note
             Predicted wR2: Based on SigI**2  2.18 or SHELX Weight 10.96
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.         10 Info
PLAT994_ALERT_1_G SHELXL .ins Contains no or MERG 0 Instruction ..          ! Note
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   6 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  11 ALERT level G = General information/check it is not something unexpected

   1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   7 ALERT type 2 Indicator that the structure model may be wrong or deficient
   3 ALERT type 3 Indicator that the structure quality may be low
   5 ALERT type 4 Improvement, methodology, query or suggestion
   1 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

PLATON version of 15/01/2026; check.def file version of 02/01/2026

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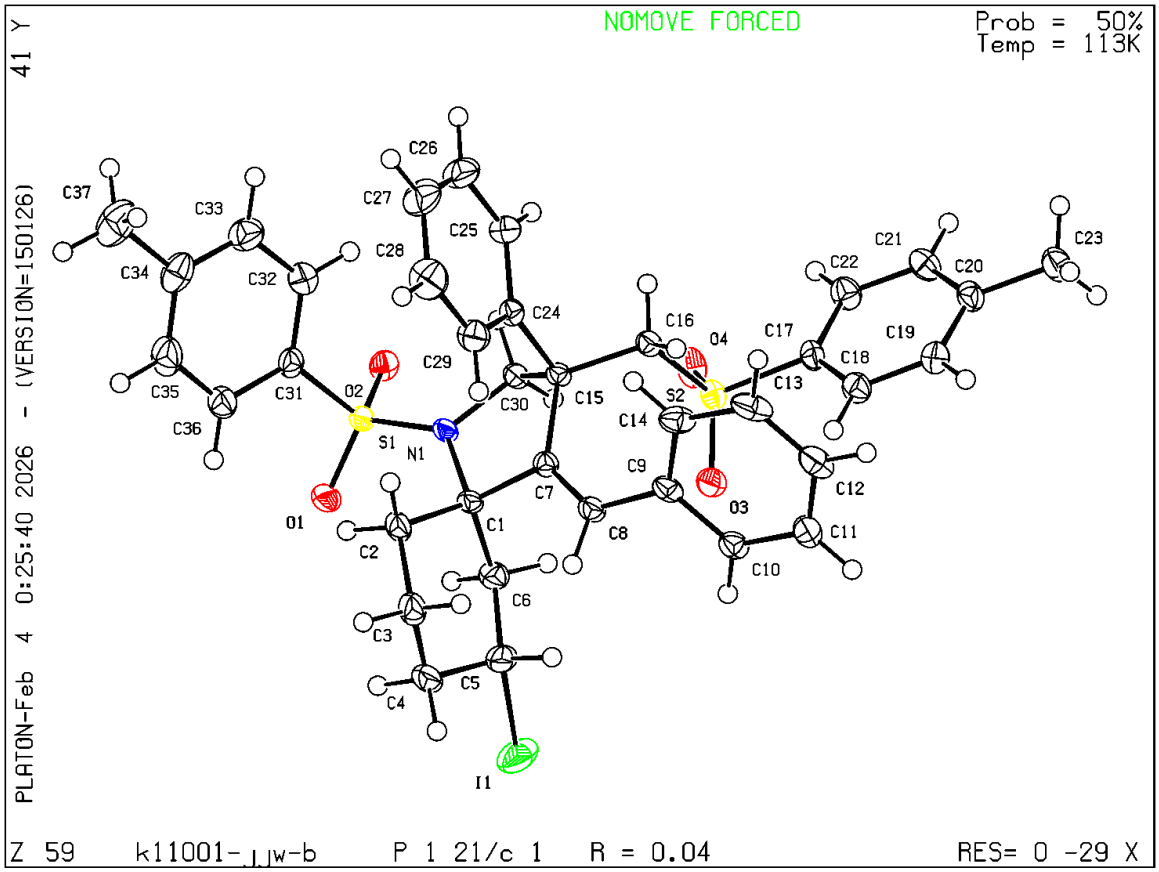
duplicate check

No duplication found

Datablock k11001-jjw-b - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) k11001-jjw-h

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: k11001-jjw-h

```
Bond precision:   C-C = 0.0038 A          Wavelength=0.71073

Cell:             a=10.2587(3)     b=14.3099(3)      c=22.3142(5)
                  alpha=90         beta=99.898(2)    gamma=90
Temperature:      113 K

                 Calculated                Reported
Volume           3226.99(14)               3226.99(14)
Space group      P 21/n                    P 1 21/n 1
Hall group       -P 2yn                    -P 2yn
Moiety formula   C36 H36 Cl N O4 S2 Se     C36 H36 Cl N O4 S2 Se
Sum formula      C36 H36 Cl N O4 S2 Se     C36 H36 Cl N O4 S2 Se
Mr               725.19                    725.19
Dx,g cm-3        1.493                     1.493
Z                4                         4
Mu (mm-1)        1.418                     1.418
F000             1496.0                    1496.0
F000'            1497.73
h,k,lmax         13,18,28                  12,18,28
Nref             7066                      6740
Tmin,Tmax        0.615,0.654               0.578,1.000
Tmin'            0.603

Correction method= # Reported T Limits: Tmin=0.578 Tmax=1.000
AbsCorr = MULTI-SCAN
```

Data completeness= 0.954 Theta(max)= 27.021

R(reflections)= 0.0390(5621) wR2(reflections)= 0.0909(6740)

S = 1.025 Npar= 419

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT410_ALERT_2_C Short Intra H...H Contact H13 ..H23A . 1.94 Ang.
x,y,z = 1_555 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 3.387 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 2 Report
-3 0 3, 1 0 11,

Alert level G

PLAT164_ALERT_4_G Nr. of Refined C-H H-Atoms in Heavy-Atom Struct. 3 Note
PLAT432_ALERT_2_G Short Inter X...Y Contact Se1 ..C9 . 3.39 Ang.
3/2-x,1/2+y,3/2-z = 2_656 Check
PLAT793_ALERT_4_G Model has Chirality at C1 (Centro SpGr) R Verify
PLAT793_ALERT_4_G Model has Chirality at C3 (Centro SpGr) S Verify
PLAT793_ALERT_4_G Model has Chirality at C13 (Centro SpGr) S Verify
PLAT910_ALERT_3_G Missing FCF Reflection(s) Below Theta(Min)[Deg]= 2.07 Note
0 1 1, 0 0 2,
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 308 Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File 4 Note
0 0 2, -3 0 3, 1 0 11, 0 1 1,
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ..... 3.110 Note
Predicted wR2: Based on SigI**2 2.92 or SHELX Weight 8.87
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 10 Info
PLAT994_ALERT_1_G SHELXL .ins Contains no or MERG 0 Instruction .. ! Note

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
3 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
11 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
4 ALERT type 2 Indicator that the structure model may be wrong or deficient
3 ALERT type 3 Indicator that the structure quality may be low
5 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

PLATON version of 15/01/2026; check.def file version of 02/01/2026

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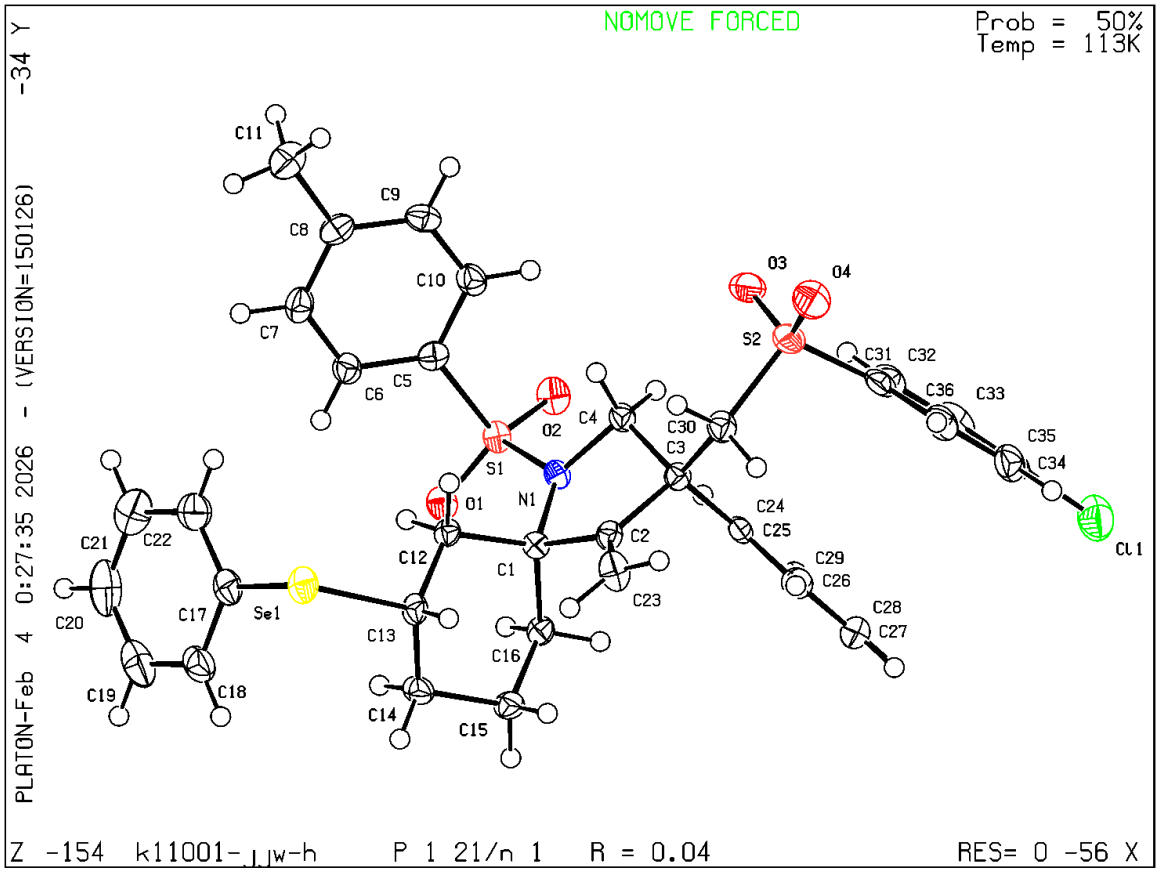
duplicate check

No duplication found

Datablock k11001-jjw-h - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) shelx

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: shelx

Bond precision: C-C = 0.0043 A Wavelength=0.71073

Cell: a=10.5710(4) b=14.5162(4) c=22.7243(9)
alpha=90 beta=101.071(1) gamma=90
Temperature: 298 K

	Calculated	Reported
Volume	3422.2(2)	3422.2(2)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C37 H38 Cl N O4 S3	C37 H38 Cl N O4 S3
Sum formula	C37 H38 Cl N O4 S3	C37 H38 Cl N O4 S3
Mr	692.31	692.31
Dx,g cm-3	1.344	1.344
Z	4	4
Mu (mm-1)	0.336	0.336
F000	1456.0	1456.0
F000'	1458.62	
h,k,lmax	12,17,27	12,17,27
Nref	6132	6085
Tmin,Tmax	0.898,0.973	0.900,0.974
Tmin'	0.898	

Correction method= # Reported T Limits: Tmin=0.900 Tmax=0.974
AbsCorr = MULTI-SCAN

Data completeness= 0.992 Theta(max)= 25.139

R(reflections)= 0.0427(4754) wR2(reflections)= 0.1369(6085)

S = 1.044 Npar= 417

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT410_ALERT_2_B Short Intra H...H Contact H7 ..H29A . 1.88 Ang.
x,y,z = 1_555 Check
PLAT919_ALERT_3_B Reflection # Likely Affected by the Beamstop ... 1 Check
0 1 2,
PLAT939_ALERT_3_B Large Value of Not (SHELXL) Weight Optimized S . 184.94 Check

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Alert level C

ABSTY02_ALERT_1_C An _exptl_absorpt_correction_type has been given without
a literature citation. This should be contained in the
_exptl_absorpt_process_details field.
Absorption correction given as multi-scan
PLAT220_ALERT_2_C NonSolvent Resd 1 C Ueq(max)/Ueq(min) Range 3.4 Ratio
PLAT222_ALERT_3_C NonSolvent Resd 1 H Uiso(max)/Uiso(min) Range 4.1 Ratio
PLAT242_ALERT_2_C Low MainResAtom Ueq as Compared to Neighbours C34 Check
PLAT334_ALERT_2_C Small <C-C> Benzene Dist. C31 -C37 . 1.37 Ang.
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00426 Ang.
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.598 45 Report
0 2 0, 0 4 0, 11 8 0, 1 0 1, -2 0 2, -2 2 2,
-1 2 2, 0 3 2, 1 3 2, 12 3 2, 2 17 2, 1 0 3,
0 1 3, 1 2 3, 1 3 3, -2 1 4, 0 1 4, 4 16 4,
-2 17 4, -1 0 5, 0 2 5, -1 17 5, 0 17 5, -1 2 6,
-12 5 8, -11 8 9, -11 7 12, 8 9 12, 6 12 12, -4 15 12,
(15 More NOT listed: see .ckf listing file)
PLAT913_ALERT_3_C Missing # of Very Strong Reflections in FCF .... 13 Note
0 2 0, 0 4 0, -2 0 2, -2 2 2, -1 2 2, 0 3 2,
1 3 2, 1 2 3, 1 3 3, -2 1 4, -1 0 5, 0 2 5,
-1 2 6,
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 1 Check
0 1 2,
PLAT934_ALERT_3_C Number of (Iobs-Icalc)/Sigma(W) > 10 Outliers .. 1 Check
0 1 2,

Alert level G

PLAT066_ALERT_1_G Predicted and Reported Tmin&Tmax Range Identical ? Check
PLAT793_ALERT_4_G Model has Chirality at C7 (Centro SpGr) R Verify
PLAT793_ALERT_4_G Model has Chirality at C9 (Centro SpGr) S Verify
PLAT793_ALERT_4_G Model has Chirality at C21 (Centro SpGr) R Verify
PLAT883_ALERT_1_G Absent Datum for _atom_sites_solution_primary .. Please Do !
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still 55% Note
PLAT910_ALERT_3_G Missing FCF Reflection(s) Below Theta(Min)[Deg]= 2.00 Note

```
               0  1  1,   0  0  2,
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........      3.226 Note
             Predicted wR2: Based on SigI**2  4.24 or SHELX Weight 13.11
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          5 Info
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
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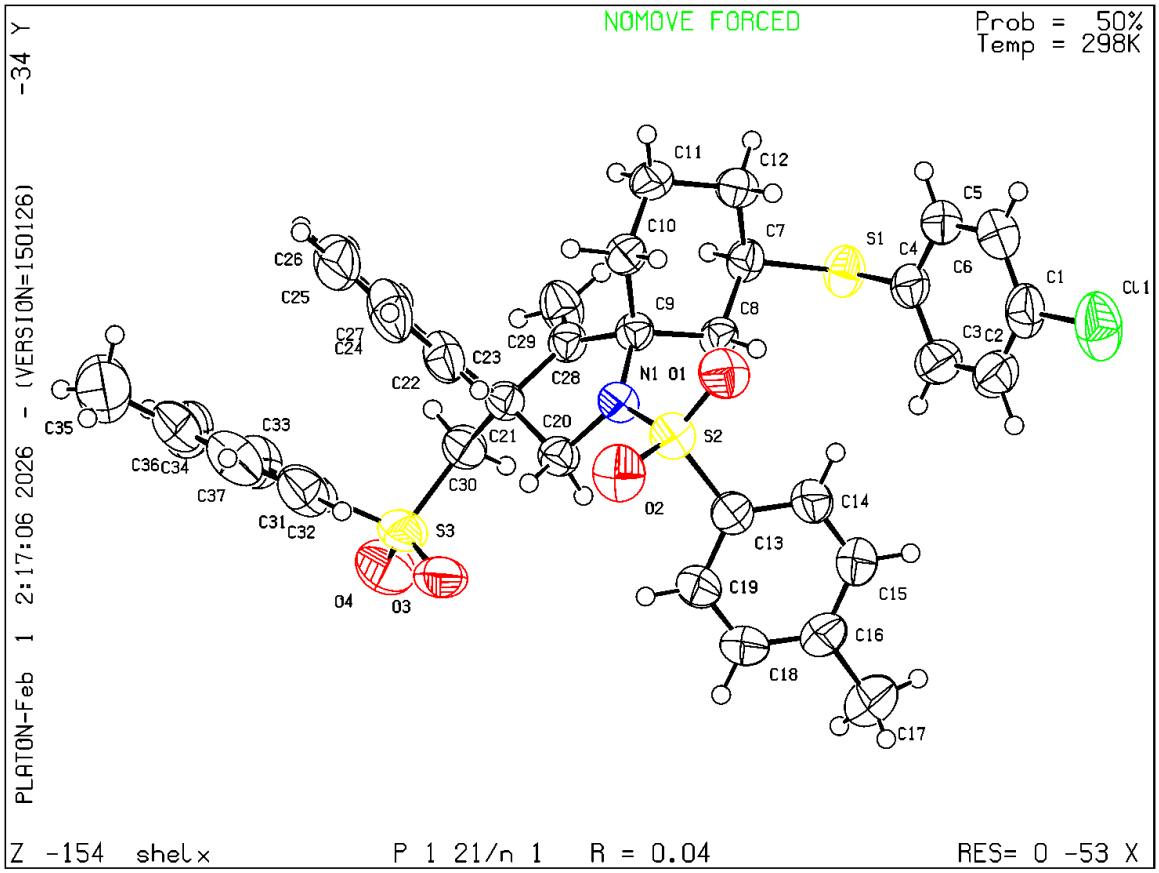
  10 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   9 ALERT level G = General information/check it is not something unexpected

   3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   5 ALERT type 2 Indicator that the structure model may be wrong or deficient
  10 ALERT type 3 Indicator that the structure quality may be low
   3 ALERT type 4 Improvement, methodology, query or suggestion
   1 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

PLATON version of 15/01/2026; check.def file version of 02/01/2026

Datablock shelx - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) shelx

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: shelx

Bond precision: C-C = 0.0048 A Wavelength=0.71073

Cell: a=17.9254(8) b=20.1083(8) c=17.3647(7)
alpha=90 beta=115.764(1) gamma=90
Temperature: 200 K

	Calculated	Reported
Volume	5636.9(4)	5636.9(4)
Space group	C 2/c	C 2/c
Hall group	-C 2yc	-C 2yc
Moiety formula	C31 H33 N O5 S2	C31 H33 N O5 S2
Sum formula	C31 H33 N O5 S2	C31 H33 N O5 S2
Mr	563.70	563.70
Dx,g cm-3	1.329	1.328
Z	8	8
Mu (mm-1)	0.230	0.230
F000	2384.0	2384.0
F000'	2387.00	
h,k,lmax	21,23,20	21,23,20
Nref	4978	4950
Tmin,Tmax	0.954,0.989	0.913,0.989
Tmin'	0.912	

Correction method= # Reported T Limits: Tmin=0.913 Tmax=0.989
AbsCorr = MULTI-SCAN

Data completeness= 0.994 Theta(max)= 25.018

R(reflections)= 0.0542(4389) wR2(reflections)= 0.1559(4950)

S = 1.035 Npar= 342

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

ABSTY02_ALERT_1_C An _exptl_absorpt_correction_type has been given without a literature citation. This should be contained in the _exptl_absorpt_process_details field. Absorption correction given as multi-scan

DIFMN02_ALERT_2_C The minimum difference density is < -0.1*ZMAX*0.75 _refine_diff_density_min given = -1.250 Test value = -1.200

DIFMN03_ALERT_1_C The minimum difference density is < -0.1*ZMAX*0.75 The relevant atom site should be identified.

PLAT098_ALERT_2_C Large Reported Min. (Negative) Residual Density -1.25 eA-3

PLAT220_ALERT_2_C NonSolvent Resd 1 C Ueq(max)/Ueq(min) Range 3.2 Ratio

PLAT230_ALERT_2_C Hirshfeld Test Diff for C3 --C4 . 6.0 s.u.

PLAT334_ALERT_2_C Small <C-C> Benzene Dist. C1 -C7 . 1.37 Ang.

PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00478 Ang.

PLAT410_ALERT_2_C Short Intra H...H Contact H22 ..H23B . 1.96 Ang.
x,y,z = 1_555 Check

PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.595 24 Report
2 0 0, 4 0 0, 3 1 0, 1 5 0, -2 4 1, 0 4 1,
-1 5 1, 0 6 1, 1 1 2, -2 2 2, -1 3 2, -4 4 2,
0 4 2, -3 1 3, -1 1 3, -4 2 3, -2 2 3, 0 2 3,
-3 3 3, -2 0 4, -1 1 4, -3 3 4, -3 1 6, 10 8 11,

PLAT913_ALERT_3_C Missing # of Very Strong Reflections in FCF .... 13 Note
2 0 0, 4 0 0, 3 1 0, -2 4 1, 0 4 1, -1 5 1,
-3 1 3, -2 2 3, 0 2 3, -2 0 4, -1 1 4, -3 3 4,
-3 1 6,

PLAT922_ALERT_1_C wR2 in the CIF and FCF Differ by ..... 0.0012 Check

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Alert level G

PLAT003_ALERT_2_G Number of Uiso or U(i,j) Restrained non-H-Atoms 9 Report

PLAT066_ALERT_1_G Predicted and Reported Tmin&Tmax Range Identical ? Check

PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large 14.02 Why ?

PLAT171_ALERT_4_G The CIF-Embedded .res File Contains EADP Records 2 Report

PLAT174_ALERT_4_G The CIF-Embedded .res File Contains FLAT Records 1 Report

PLAT177_ALERT_4_G The CIF-Embedded .res File Contains DELU Records 1 Report

PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records 1 Report

PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records 1 Report

PLAT190_ALERT_3_G A Non-default RIGU Restraint Value for First Par 0.0010 Report

PLAT192_ALERT_3_G A Non-default DELU Restraint Value for First Par 0.0400 Report

PLAT192_ALERT_3_G A Non-default DELU Restraint Value for SecondPar 0.0400 Report

PLAT398_ALERT_2_G Deviating C-O-C Angle From 120 for O3 . 60.5 Degree

PLAT793_ALERT_4_G Model has Chirality at C8 (Centro SpGr) R Verify

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PLAT793_ALERT_4_G Model has Chirality at C10         (Centro SpGr)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C11         (Centro SpGr)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C16         (Centro SpGr)          S Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............         63 Note
PLAT883_ALERT_1_G Absent Datum for _atom_sites_solution_primary ..     Please Do !
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still        76% Note
PLAT910_ALERT_3_G Missing FCF Reflection(s) Below Theta(Min)[Deg]=       2.41 Note
                1  1  0,   0  2  0,  -1  1  1,   1  1  1,
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File          2 Note
                0  2  0,  -1  5  1,
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........      7.813 Note
             Predicted wR2: Based on SigI**2  1.98 or SHELX Weight 15.06
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          6 Info
```

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duplicate check

No duplication found

Datablock shelx - ellipsoid plot