Supporting Information

For

Light-Mediated Sulfonyl-Iodination of Ynamides and Internal Alkynes

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1. General Information

^{1}H and ^{13}C NMR spectra were recorded on a 400 MHz Varian Unity Plus or Varian Mercury plus spectrometer. The chemical shift (δ) values are reported in parts per million (ppm), and the coupling constants (J) are given in Hz. The spectra were recorded using $CDCl_3$ as a solvent. ^{1}H NMR chemical shifts are referenced to tetramethylsilane (TMS) (0 ppm). ^{13}CNMR was referenced to $CDCl_3$ (77.0 ppm). The abbreviations used are as follows: s, singlet; d, doublet; t, triplet; q, quartet; dd, doublet of doublet; ddd, doublet of doublet of doublet; dt, doublet of triplets; td, triplet of doublet; m, multiplet. Mass spectra and high-resolution mass spectra (HRMS) was measured using the LTQ Orbitrap XL (Thermo Fischer Scientific) Liquid chromatography–mass spectrometry at National Taiwan Normal University. Melting points were determined on an EZ-Melt (Automated melting point apparatus). All the synthesized products showed ^{1}HNMR spectra in agreement with the assigned structures. Reaction progress and products were routinely monitored by TLC using Merck TLC aluminum sheets (silica gel 60 F254). Column chromatography was carried out with 230-400 mesh silica gel 60 (Merck) using a mixture of hexane/ethyl acetate as the eluent. Importantly, the reaction temperature may differ based on place and weather condition outside environment.

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.phphttps://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.

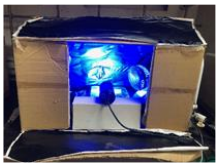
2. Reaction handling setup pictures and blue LED technical details[1]

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

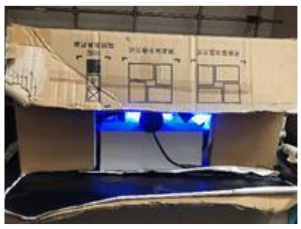
side view of stirrer with fan (For clarity we took picture without cardboard box)

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

The reaction setup under cardboard box



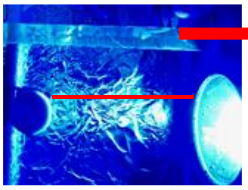
Outside view of the setup(door open



Outside view of the setup (door closed)

Cooling of the reaction under light irradiation

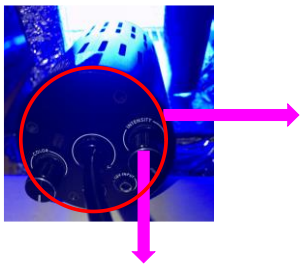
1 cm hight (light source)



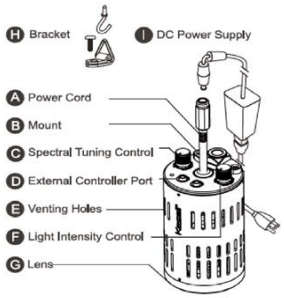
8.5 cm distance from light source to reaction vial

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

Figure S1. Pictures represent the complete reaction setup and other technical details of the 40 W Blue LED (Kessil A160WE Controllable LED Aquarium Light).

Outside box appearance of Kessil 40 W PR160L-456 nm blue LED light

Kessil PR160L-456 nm blue LED light and supporting power cables inside the box

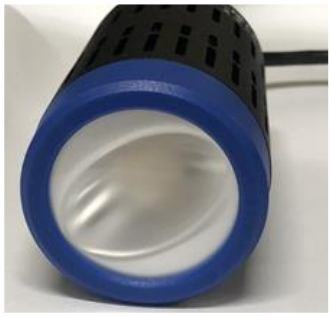
Kessil PR160L-456 nm blue LED light and supporting power cables outside the box

40 W blue LED light and adapter

40 W blue LED light

Kessil PR160L-450 nm

Blue LED intensity adjustment



Frontside view of Blue LED light

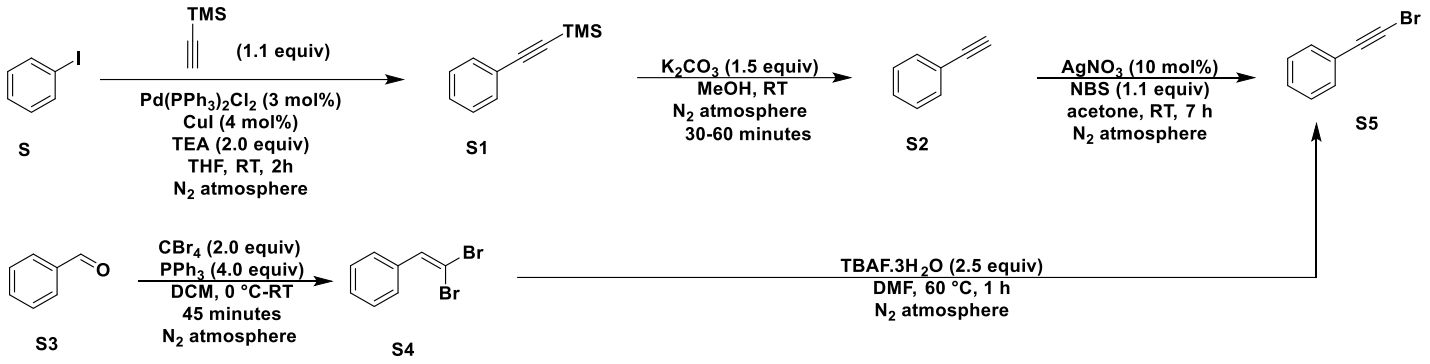
Technical Specifications

Specifications		
Power Consumption	=	456 nm (max 40W)
Input Voltage	=	100-240 VAC
Operating Temperature	=	0 - 40 °C / 32 - 104 °F
Beam Angle	=	56°
Average Intensity of PR160 series	=	352mW/cm2 (measured from 1 cm distance)
Dimensions	=	4.49" x 2.48" / 11.4cm x 6.3cm (H **x D)**

Figure S2. Pictures represents the 40 W blue LED lamp (PR160L-456 nm) and other technical details.

3. Preparation of Starting Materials

3.1. Procedure for the synthesis of (Bromoethynyl)benzene[1]



3.1.1. Procedure for the synthesis of trimethyl(phenylethynyl)silane (S1):

To a dried schlenk flask equipped with a stir bar was charged with iodobenzene (6 g, 29.41 mmol, 1.0 equiv), trimethylsilylacetylene (3.17 g, 32.35 mmol, 1.1 equiv) in THF at room temperature. Next, the schlenk tube was evacuated and filled with nitrogen (three cycles), followed by freshly distilled Et_3N (5.95 g, 58.82 mmol, 2.0 equiv), $Pd(PPh_3)_2Cl_2$ (0.61 g, 8.82 mmol, 3 mol%), CuI (0.22 g, 1.17 mmol, 4 mol%) were added under the N_2 atmosphere. The resulting mixture was stirred at room temperature for 2 h. After the completion of the reaction by TLC, the reaction mixture was diluted with water, and extracted with ethyl acetate. The combined organic layer was dried over Na_2SO_4, filtered, and concentrated to give the crude material. The crude material was purified by column chromatography using hexane (100%) as the eluent gave brown liquid trimethyl(phenylethynyl)silane in 88% (4.5 g).

3.1.2. Procedure for the synthesis of ethynylbenzene (S2)

To a dried schlenk flask equipped with a stir bar was charged with trimethyl(phenylethynyl)silane (4.5 g, 25.86 mmol, 1.0 equiv) in MeOH at room temperature. Next, the schlenk flask was evacuated and filled with nitrogen (three cycles), followed by K_2CO_3 (5.36 g, 38.79 mmol, 1.5 equiv) was added under the N_2 atmosphere. The resulting mixture was stirred at room temperature for 30-60 minutes. The solvent was removed under reduced pressure and the resulting solid/liquid was dissolved in ethyl acetate, washed with water and brine, and dried over Na_2SO_4. The crude material was purified by column chromatography using hexane-ethyl acetate (99:1%) as the eluent gave colorless liquid ethynylbenzene in 94% (2.5 g).

3.1.3. Procedure for the synthesis of (bromoethynyl)benzene (S5)

To a dried schlenk flask equipped with a stir bar was charged with ethynylbenzene (2.5 g, 24.47 mmol, 1.0 equiv) in dry acetone. Next, the schlenk flask was evacuated and filled with nitrogen (three cycles), followed by $AgNO_3$ (0.41 g, 2.44 mmol, 0.1 equiv), NBS (4.79 g, 26.92 mmol, 1.1 equiv) added in portions under the N_2 atmosphere. The mixture was stirred for 7 h at room temperature (the flask was covered with aluminium foil). After the completion of the reaction by TLC, the solvent was concentrated in vacuum. The reaction mixture was diluted with water, and extracted with ethyl acetate. The combined organic layer was dried over Na_2SO_4, filtered, and concentrated to give the crude material. The crude material was purified by column chromatography using hexane (100%) as the eluent gave yellow color liquid (bromoethynyl)benzene in 70% (3.1 g).

3.2. Alternative route for the synthesis of compound S5

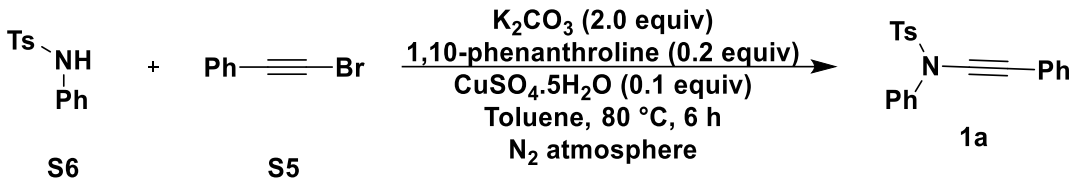
3.2.1. Procedure for Synthesis of (2,2-dibromovinyl)benzene (S4): CBr_4 (1873 mg, 5.64 mmol, 2.0 equiv) and PPh_3 (2958 mg, 11.28 mmol, 4.0 equiv) were combined in a flask which was evacuated and refilled with nitrogen (three cycles). CH_2Cl_2 (0.2 M) was added and the resulting solution was stirred for 10 minutes at room temperature. 1.0 equiv. of Benzaldehyde (0.3 g, 2.82 mmol) in CH_2Cl_2 (2 mL) was added to the above reaction mixture at 0 °C and stirred for 45 minutes, slowly the reaction mixture warming to room temperature. The solvent was removed under reduced pressure and the residue was dissolved in the minimum quantity of CH_2Cl_2. Hexane was added to the flask and quickly filtered through a pad of silica. This was repeated until all the contents had been transferred to the filter bed. The filter cake was washed with a hexane:Et_2O mixture (95:5, 200 mL), and the solvent was removed to give the as a yellow oil which was used for the next step.

3.2.2. Procedure for synthesis of (bromoethynyl)benzene (S5)

(2,2-dibromovinyl)benzene (500 mg, 1.90 mmol, 1.0 equiv) was dissolved in 5.0 mL of dry DMF. Next, the schlenk tube was evacuated and filled with nitrogen (three cycles) and TBAF·$3H_2O$ (1.2 g, 3.80 mmol, TBAF·$3H_2O$ (1505 mg, 4.77 mmol, 2.5 equiv) was added to the solution and the seal tube was evacuated and filled with nitrogen (three cycles). The reaction mixture was heated at 60 °C for 1 h (TLC). The reaction mixture was cooled to room temperature and diluted with diethyl ether (50 mL). The organic phase was washed with water and brine, dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by using silica gel column chromatography using n-hexane as eluent gave the desired yellow liquid in 72% (250 mg).

(Note: Other (bromoethynyl)benzene derivatives were obtained in a similar protocol based on availability of the starting material)

3.3. Procedure for the synthesis of 4-methyl-*N*-phenyl-*N*-(phenylethynyl)benzenesulfonamide (1a)[2]

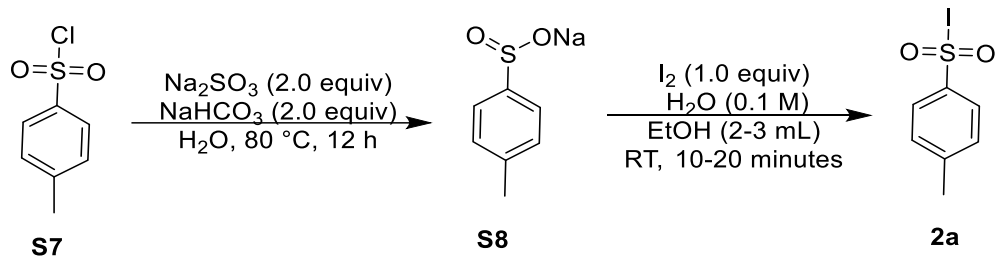


To a mixture of 4-methyl-*N*-phenylbenzenesulfonamide (250 mg, 1.0. mmol, 1.0 equiv), $CuSO_4 \cdot 5H_2O$ (25 mg, 0.10 mmol, 0.1 equiv), 1,10-phenanthroline (36 mg, 0.20 mmol, 0.2 equiv) and K_2CO_3 (279 mg, 2.02 mmol, 2.0 equiv), dry toluene (4 mL). Next, the seal tube was evacuated and filled with nitrogen (three cycles), followed by (bromoethynyl)benzene (366 mg, 2.02 mmol, 2.0 equiv) were added. The vessel was stoppered under a nitrogen atmosphere and heated in an oil bath maintained at 80 °C for 6 h. The mixture was passed through celite and concentrated in a vacuum. The crude product was purified by using silica gel column chromatography using hexane/ethyl acetate (95:5) as eluent. This gave the desired 4-methyl-N-phenyl-N-(phenylethynyl)benzenesulfonamide as yellow gummy compound in 57% (200 mg).

(Note: Other ynamide derivatives (**1b**-**1l**) were obtained through the procedure reported above. Compounds known in the literature were confirmed by comparing their ^{1}H and ^{13}C NMR spectra)[2]

(Note: The unsymmetrical alkynes derivatives (**4**[3], **5**[4], **6**[5], **7**[6], **8**[7], **9**[1], **10**[8], **11**[9], **12**[10], **13**[10], **14**[11], **15**[12], **16**[10] and **17**[10]) were obtained through the procedure reported in the literature. Compounds known in the literature were confirmed by comparing their ^{1}H and ^{13}C NMR spectra)

3.4. Synthsis of 4-methylbenzenesulfonyliodide (2a)[1,13]



3.4.1. Procedure for the preparation of sodium *p*-toluenesulfinate (S26)

To a round-bottom flask was added 4-toluenesulfonyl chloride (2.0 g, 10.49 mmol, 1.0 equiv), was added to a stirred solution of sodium sulfite (2.64 g, 20.98 mmol, 2.0 equiv), and sodium bicarbonate (1.76 g, 20.98 mmol, 2.0 equiv)in H_2O. After being stirred at 80 °C for 12 h, then water was removed by a rotary evaporator. Then the remaining solid was extracted and recrystallized from ethanol to get the desired sodium *p*-toluenesulfinate (1.2 g, 64%) (Note: Oher sodium sulfinates were obtained in a similar protocol).

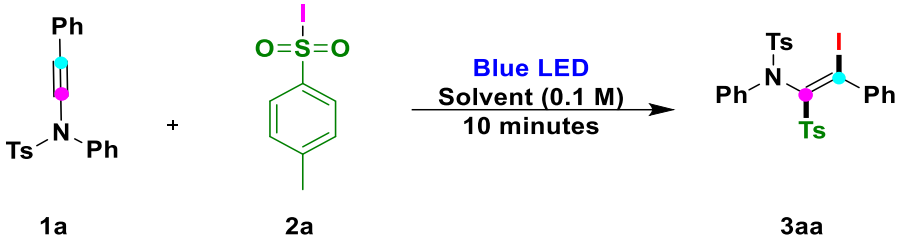
3.4.2. Procedure for the preparation of 4-methylbenzenesulfonyliodide (2a)

To a round-bottom flask (50 mL) was added sodium *p*-toluenesulfinate (100 mg, 0.56 mmol, 1.0 equiv) in distilled water at room temperature. A saturated solution of iodine (142 mg, 0.56 mmol, 1.0 equiv) in ethanol (1-2 mL) was prepared and added gradually to the sodium *p*-tolylsulfinate solution. During this addition period, yellow precipitates were formed gradually. The precipitates were filtered, washed with cold water, and dried carefully at room temperature to give p-toluenesulfonyl iodide as a yellow solid. The synthesized 4-methylbenzenesulfonyl iodide immediately used for next step because of spontaneous decomposition of sulfonyl iodides.

Note: Other sulfonyl iodides were obtained in a similar protocol.

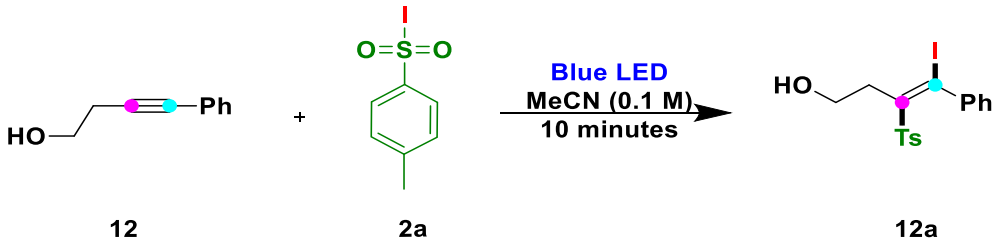
4. Experimental procedures

4.1. General procedure (A) for the Synthesis of (*E*)-*N*-(2-iodo-2-phenyl-1-tosylvinyl)-4-methyl-*N*-phenylbenzenesulfonamide derivatives



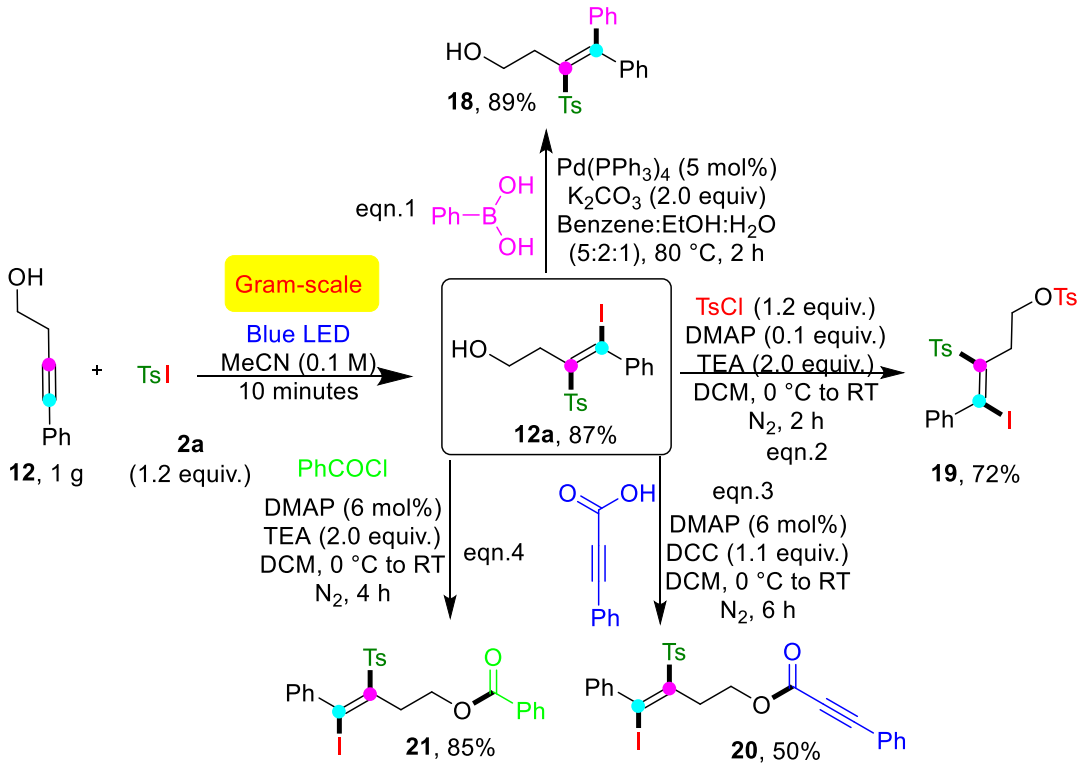
An oven-dried screw-capped, 8 mL vial equipped with a magnetic stir bar was charged with 4-methyl-*N*-phenyl-*N*-(phenylethynyl)benzenesulfonamide (55 mg, 0.15 mmol, 1.0 equiv), 4-methylbenzenesulfonyl iodide (54 mg, 0.19 mmol, 1.2 equiv), and dry acetonitrile (0.1 M) solvent was added. The resulting solution was stirred up to starting material completion (30 minutes) at 30 °C under a blue LED light (the reaction mixture vial was placed ~ 8.5 cm away from the LED light with a clip fan for cooling). After that, the crude reaction mixture was diluted with water and extracted with ethyl acetate. The organic layer was dried over Na_2SO_4, filtered, and concentrated. The crude material was purified by flash columnchromatography chromatography using hexane-ethyl acetate (85:15) as the eluent gave the desired product (*E*)-*N*-(2-iodo-2-phenyl-1-tosylvinyl)-4-methyl-*N*-phenylbenzenesulfonamide as white solid in 66% (66 mg).

4.2. General procedure (B) for the Synthesis of (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-ol derivatives (12a)



An oven-dried screw-capped, 8 mL vial equipped with a magnetic stir bar was charged with 4-phenylbut-3-yn-1-ol (45 mg, 0.30 mmol, 1.0 equiv), 4-methylbenzenesulfonyl iodide (104 mg, 0.36 mmol, 1.2 equiv), and MeCN (0.1 M) solvent was added. The resulting solution was stirred up to starting material completion (30 minutes) at 30 °C under a blue LED light (the reaction mixture vial was placed ~ 8.5 cm away from the LED light with a clip fan for cooling). After that, the crude reaction mixture was diluted with water and extracted with ethyl acetate. The organic layer was dried over Na_2SO_4, filtered, and concentrated. The crude material was purified by flash column chromatography using hexane-ethyl acetate (78:22) as the eluent gave the desired product (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-ol as white solid in 93% (60 mg).

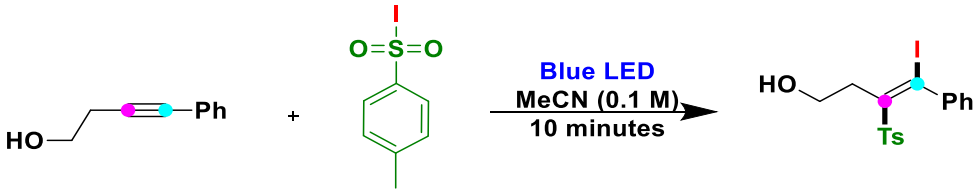
4.3. Gram-scale reaction and post-synthetic modifications (Table-S1).



To demonstrate the advantage and practicability of our transformation, a gram-scale reaction was performed by combining internal alkyne **12** with **2a** (1.2 equiv.), which afforded an sulfonyl-iodine-tetrasubstituted olefin (**12a**) product in a gram quantity (2.55 g; 87% yield) and with excellent regioselectivity and stereoselectivity (**Table-S1**). Furthermore, the synthesized **12a** product was applied

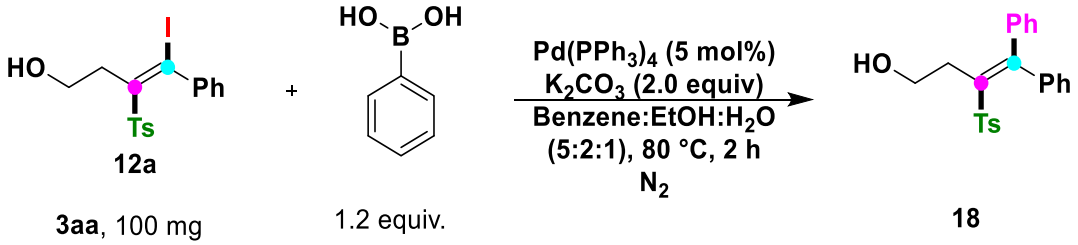
to the post-functionalization reactions. First, the active C–I bond was used in the Suzuki reaction to insert a phenyl moiety, and the desired product was produced with an excellent yield (89%) (eqn. 1). There were numerous opportunities to synthetically modify the freely available alcohols into various functional groups. In this case, we used 4-toluenesulfonyl chloride, 3-phenylpropiolic acid, and benzyl chloride under various reaction conditions (eqn. 2–4), which afforded the desired product **19**–**21** in good to excellent yields (50%–85%).

4.4. Gram scale synthesis of (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-ol (12a)



An oven-dried screw-capped, 8 mL vial equipped with a magnetic stir bar was charged with 4-phenylbut-3-yn-1-ol (1 g, 6.84 mmol, 1.0 equiv), 4-methylbenzenesulfonyl bromide (2.32 g, 8.21 mmol, 1.2 equiv), and DCM (0.1 M) solvent was added. The resulting solution was stirred up to starting material completion (30 minutes) at 30 °C under a blue LED light (the reaction mixture vial was placed ~ 8.5 cm away from the LED light with a clip fan for cooling). After that, the crude reaction mixture was diluted with water and extracted with ethyl acetate. The organic layer was dried over Na_2SO_4, filtered, and concentrated. The crude material was purified by flash column chromatography using hexane-ethyl acetate (75:25) as the eluent gave the desired product (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-ol as white solid in 87% (2550 mg).

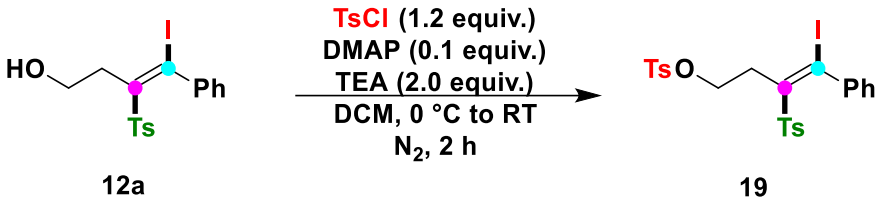
4.5. Procedure (C) for the synthesis of 4,4-diphenyl-3-tosylbut-3-en-1-ol (18)



An overnight dried seal tube (15 mL) was charged with (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-ol (100 mg, 0.23 mmol, 1.0 equiv), phenylboronic acid (34 mg, 0.28 mmol, 1.3 equiv), $Pd(PPh_3)_4$ (8 mg, 0.011 mmol, 5 mol%), K_2CO_3 (64 mg, 0.46 mmol, 2.0 equiv) in Benzene:EtOH:H_2O (5:2:1) (3 mL) under N_2 atmosphere. The resulting mixture was stirred at 80 °C for 2 h. After the completion of the reaction by TLC, the reaction mixture was cooled to room temperature, diluted with water, and extracted with ethyl acetate. The organic layer dried

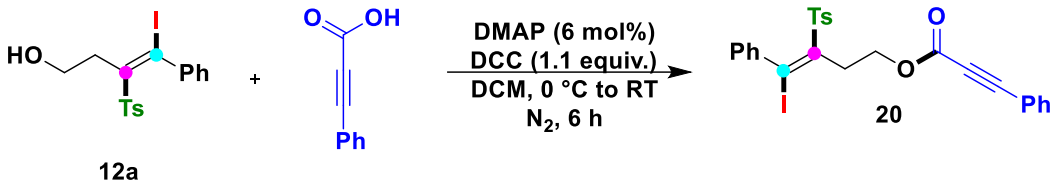
over anhydrous Na_2SO_4, filtered, and concentrated to give the crude material. The crude material was purified by column chromatography using hexane-ethyl acetate (82:18) as the eluent to afford 4,4-diphenyl-3-tosylbut-3-en-1-ol in 89% (79 mg).

4.6. Procedure (D) for the synthesis of (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-yl 4-methylbenzenesulfonate (19)



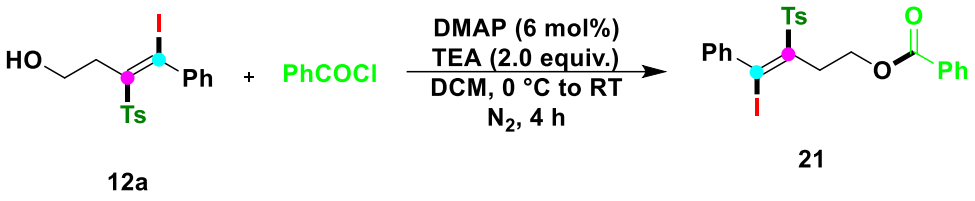
An overnight dried round bottom flask (20 mL) was charged with (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-ol (100 mg, 0.23 mmol, 1.0 equiv) in dichloromethane (10 mL) was cooled to 0 ^oC and then DMAP (3 mg, 0.02 mmol, 0.1 equiv), tosyl chloride (53 mg, 0.28 mmol, 1.2 equiv) and Et_3N (47 mg, 0.46 mmol, 2.0 equiv) were added. The reaction mixture was stirred at room temperature for 2 h and then quenched with an aqueous NH_4Cl solution, extracted with DCM, dried over anhydrous Na_2SO_4, concentrated and purified by column chromatography using hexane-ethyl acetate (80:20) to afford (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-yl 4-methylbenzenesulfonate in 72% (98 mg).

4.7. Procedure (E) for the synthesis of (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-yl 3-phenylpropiolate (20)



An overnight dried round bottom flask (20 mL) was charged with (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-ol (100 mg, 0.23 mmol, 1.0 equiv), 3-phenylpropiolic acid (34 mg, 0.23 mmol, 1.0 equiv) in dried CH_2Cl_2 (5 mL) under a nitrogen atmosphere DMAP (2 mg, 0.01 mmol, 0.06 equiv) was added and cooled to 0 °C. To the resulting solution a solution of DCC (53 mg, 0.25 mmol, 1.1 equiv) in dried CH_2Cl_2 (5 mL) was dropped in slowly under N_2 atmosphere. After this the mixture was allowed to come to room temperature and stirred for further 6 h. The resulting suspension was filtered over celite. The organic layer was washed thrice with 0.25 N aqueous HCl-, once with sat. aqueous $NaHCO_3$-solution, dried over Na_2SO_4 and concentrated under reduced pressure. The crude residue was purified by purified by column chromatography using hexane-ethyl acetate (90:90) as the eluent to afford (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-yl 3-phenylpropiolate in 50% (65 mg).

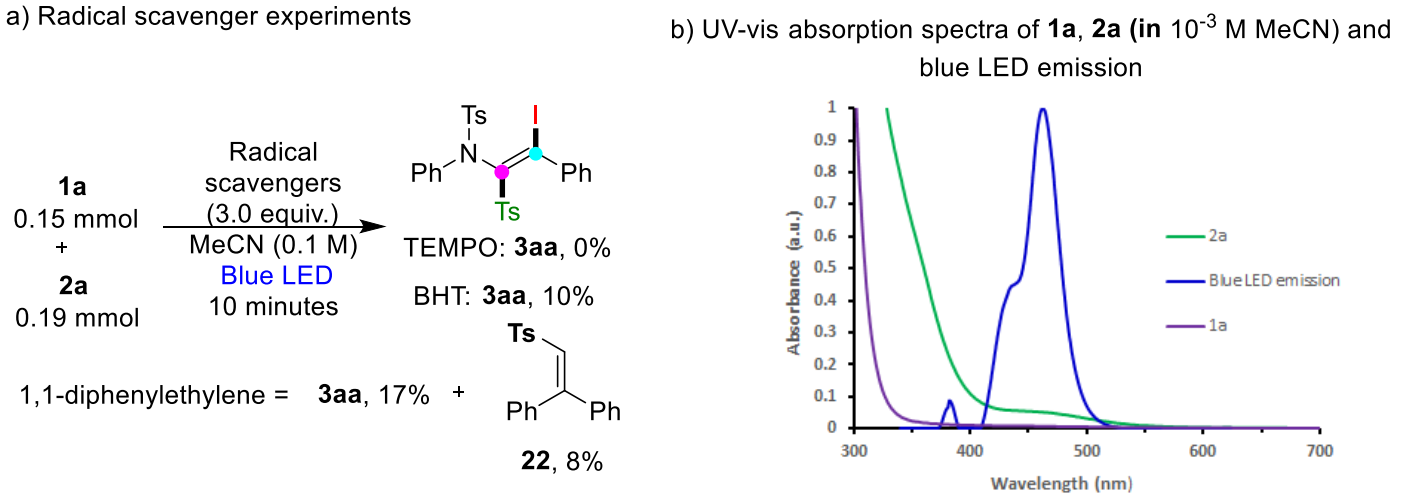
4.8. Procedure (F) for the synthesis of (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-yl benzoate (21)



In overnight dried round bottom flask (20 mL) was charged with (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-ol (100 mg, 0.23 mmol, 1.0 equiv) in DCM (5 mL) was added triethylamine (47 mg, 0.46 mmol, 2.0 equiv), benzoyl chloride (40 mg, 0.28 mmol, 1.2 equiv), and DMAP (3 mg, 0.023 mmol, 0.1 equiv) at 0 °C under N_2 atmosphere. The reaction was stirred at room temperature and monitored by TLC to the consumption of starting material **12a**. The mixture was diluted with 1 M HCl solution and the aqueous phase was extracted with DCM. Combined organic phases were washed with brine, dried, and concentrated. The crude residue was purified by purified by column chromatography using hexane-ethyl acetate (89:11) as the eluent to afford (*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-yl benzoate in 85% (106 mg).

5. Mechanistic studies

5.1. Radical scavenger experiments and UV-vis absorption spectra of 1a, 2a and blue LED emission



Scheme S1. Mechanistic studies: a) Radical scavenger experiments; b) UV-vis absorption spectra of **1a**, **2a** and blue LED emission

Radical scavenger experiments were conducted to gain insight into this transformation mechanism (Scheme S1, a). The reaction completely stopped the formation of the **3aa** product in (2,2,6,6-tetramethylpiperidin-1-yl)oxyl as a radical scavenger. However, a low yield of the **3aa** product was observed in the presence of butylated hydroxytoluene. In ethene-1,1-diyldibenzene as a radical scavenger, sulfonyl radical-trapping product 22 was observed in 8% yield along with compound **3aa** in 17% yield. The product was confirmed by ^{1}H and ^{13}C nuclear magnetic resonance (NMR). These reactions suggest that radical species participated in the reaction

pathway. To gain insight into the role of blue LED light in this experiment, we acquired absorption spectra for **1a** and **2a** (the absorption spectra were acquired in a 10⁻2–10⁻5 M MeCN solvent) and blue LED emission spectra for the blue LED light lamps in our previous studies (Scheme S1, b). The experimental results revealed that blue light influences and accelerates the radical initiation process from **2a** via the absorbance of blue LED light from the source to the radical but not ynamides. Based on mechanistic studies and previous literature support on sulfonyl iodides with other counterparts under light, we excluded the EDA or an halogen-bonding complex in the reaction mechanism.

5.1. Blue LED emission spectra and absorption spectra of compounds (2a)[1]

Emission spectra were measured using Ocean Optics USB 2000+ Spectrometer. Spectra were normalized to 1.0 at the emission maximum. This 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.

5.1.1. 40 W Kessil A160WE Tuna Blue LED emission spectra[1]

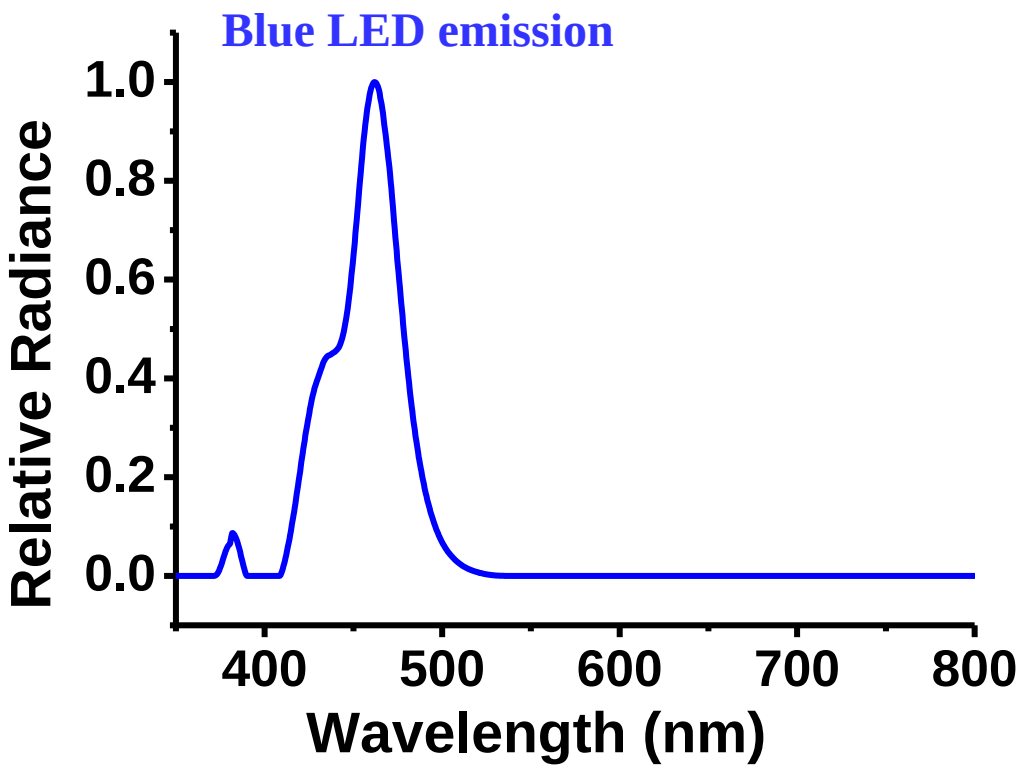


Figure S3. Emission spectrum from a 40 W Kessil A160WE Tuna Blue LED shown as blue color line with emission maximum at λ max = 462 nm flanked by a second peak at λ = 382 nm.

5.1.2. 40 W Kessil PR160L-456 nm Blue LED emission spectra[1]

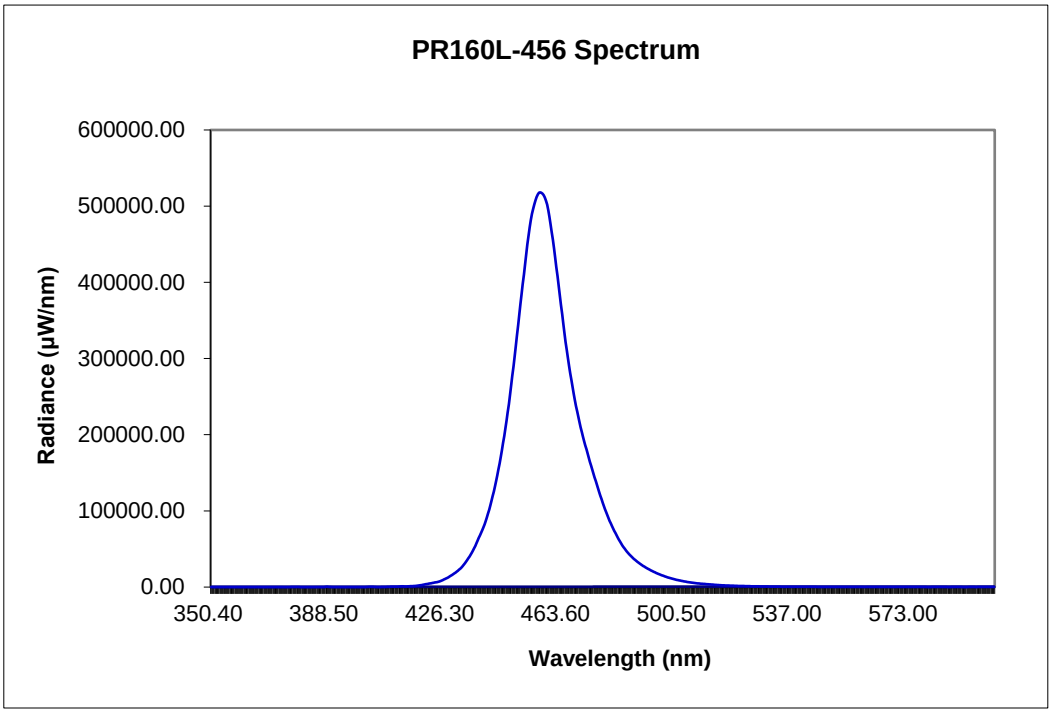


Figure S4. Emission spectrum from a 40 W Kessil PR160L-456 nm Blue LED shown as blue color line with emission maximum at λ max = 456 nm

5.2. Absorption spectra[1]

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

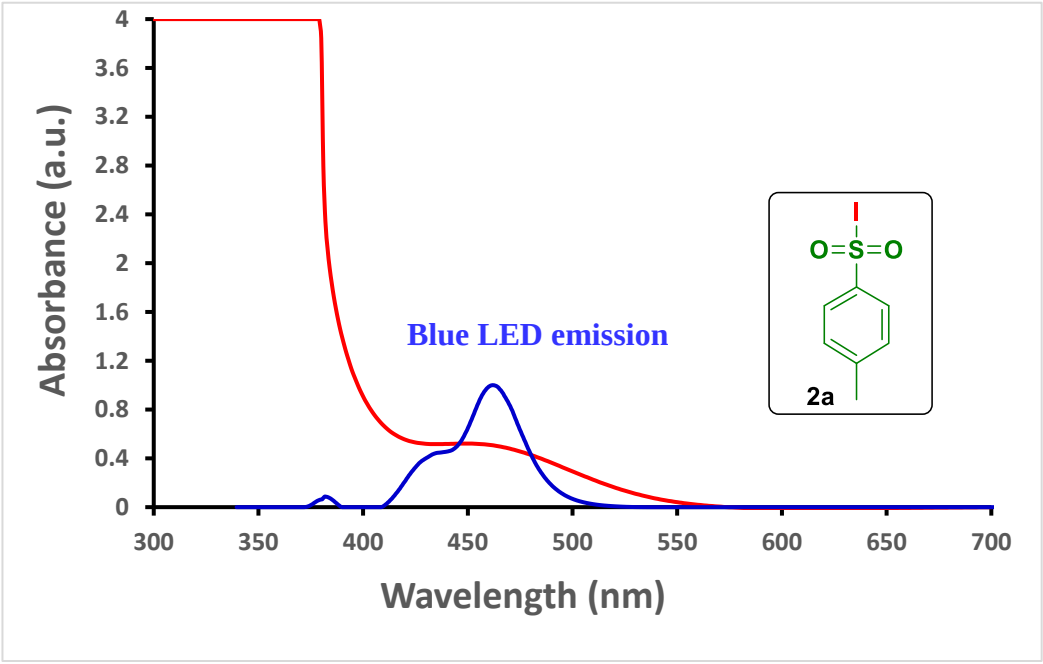


Figure S5. Absorption spectra of 4-methylbenzenesulfonyl iodide (**2a**) (10^{-2} M) in MeCN, and blue LED emission.

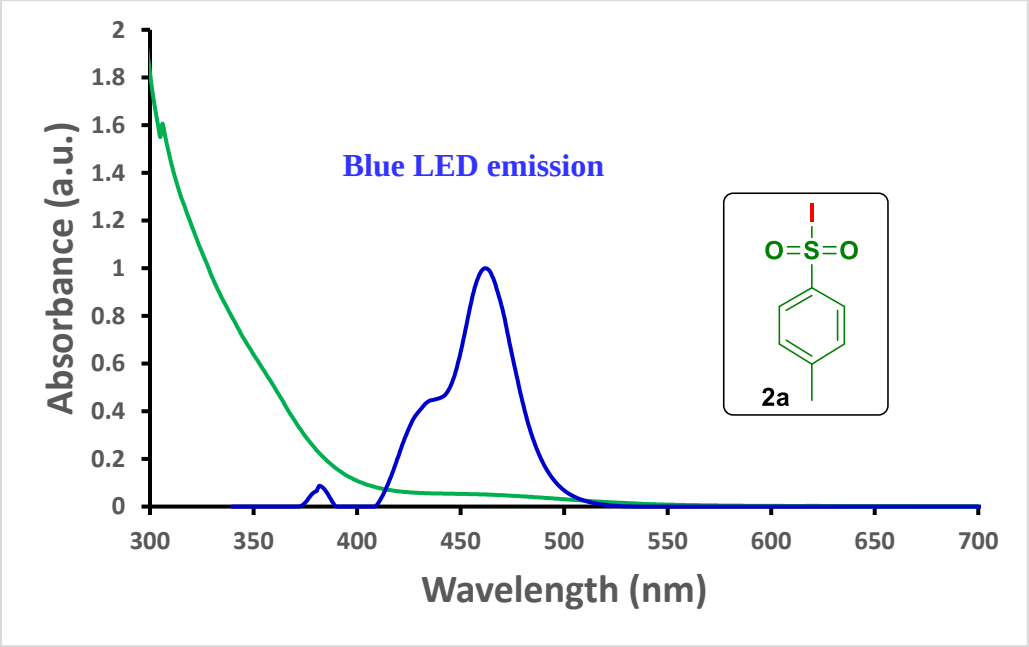


Figure S6. Absorption spectra of 4-methylbenzenesulfonyl iodide (**2a**) (10^{-3} M) in MeCN, and blue LED emission.

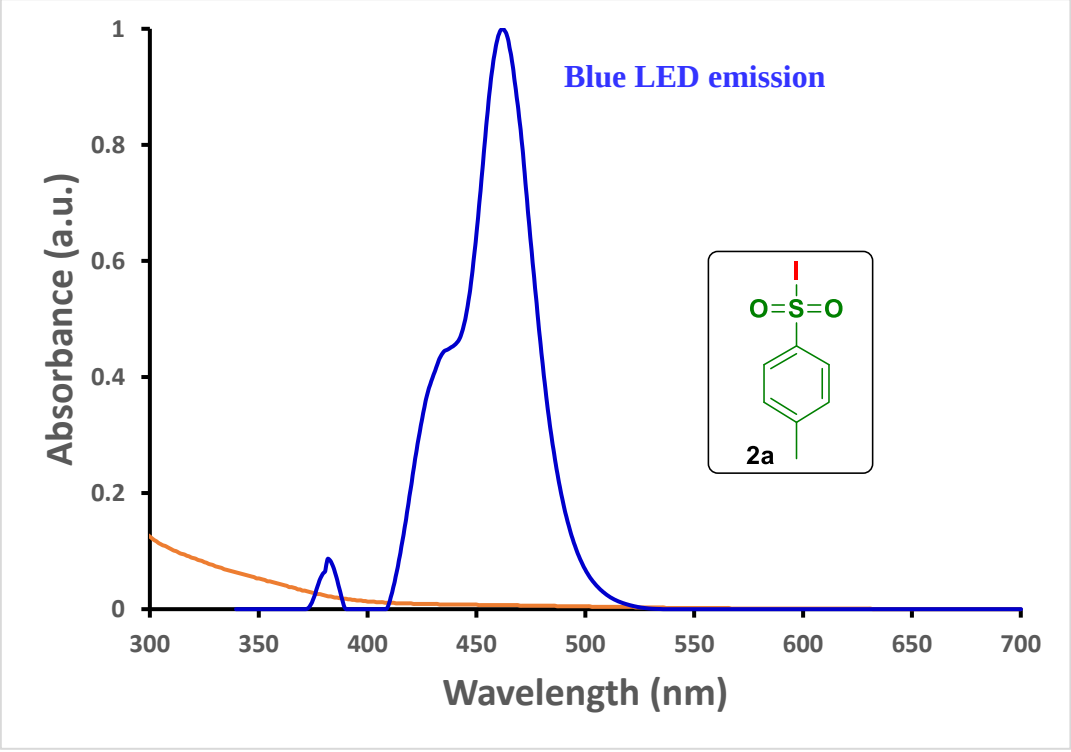


Figure S7. Absorption spectra of 4-methylbenzenesulfonyl iodide (**2a**) (10^{-4} M) in MeCN, and blue LED emission.

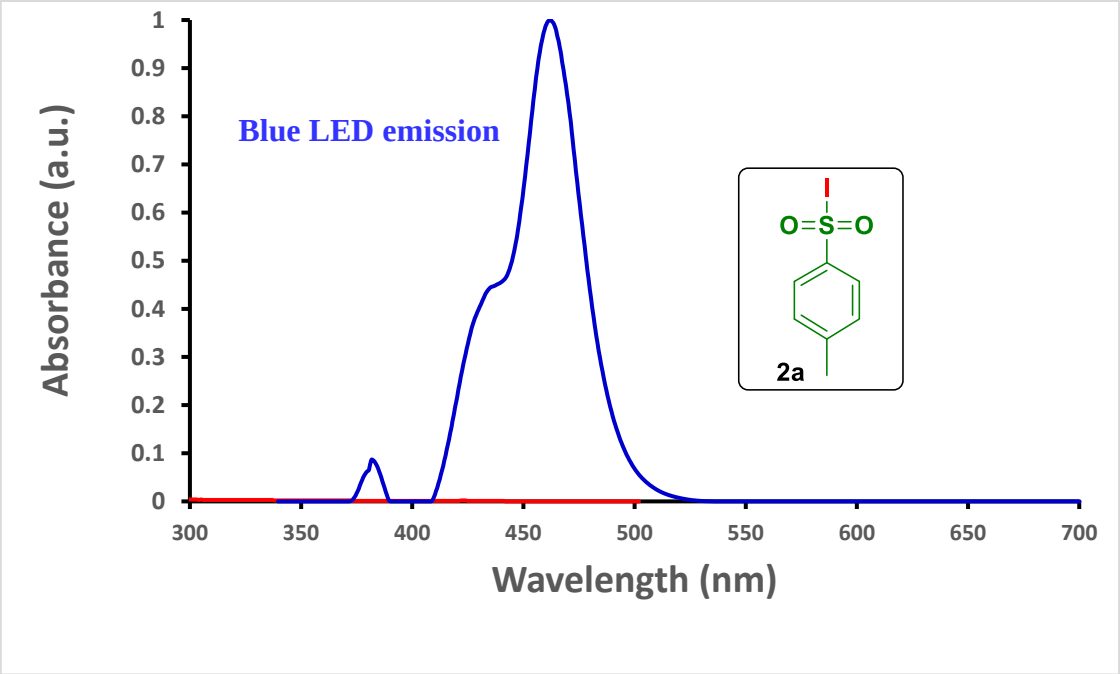


Figure S9. Absorption spectra of 4-methylbenzenesulfonyl iodide (**2a**) (10^{-5} M) in MeCN, and blue LED emission.

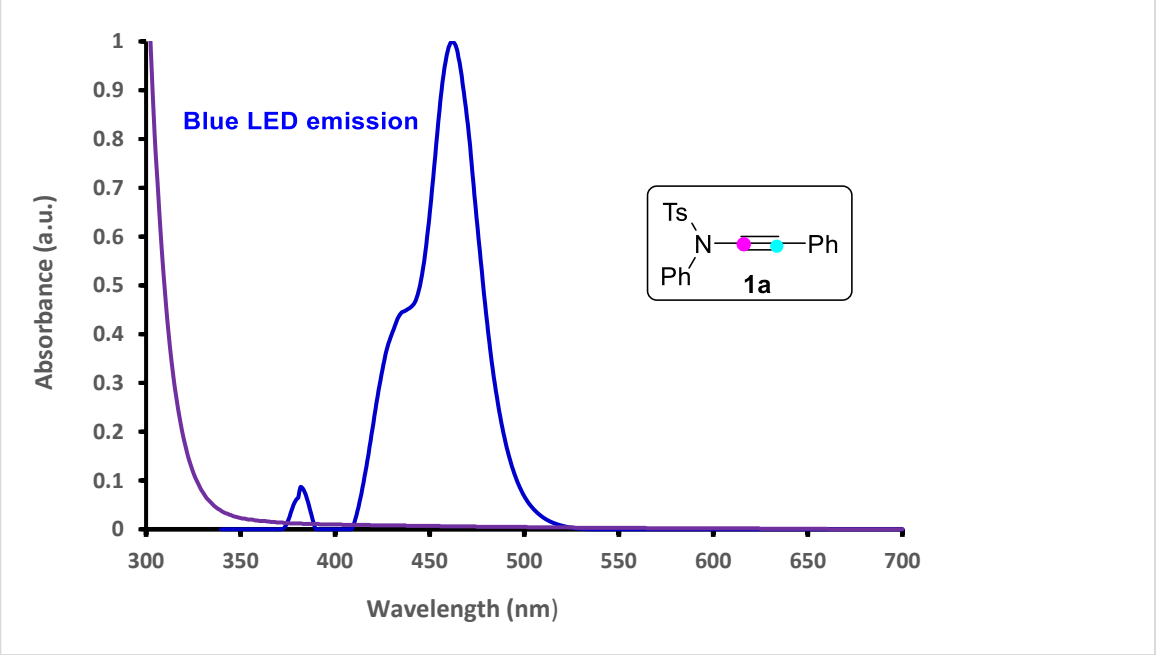
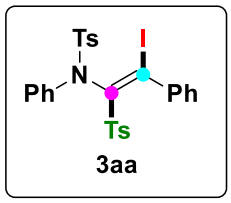


Figure S10. Absorption spectra of 4-methyl-*N*-phenyl-*N*-(phenylethynyl)benzenesulfonamide (**1a**) (10^{-3} M) in MeCN, and blue LED emission.

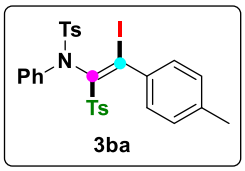
6. Characterization data

(*E*)-*N*-(2-iodo-2-phenyl-1-tosylvinyl)-4-methyl-*N*-phenylbenzenesulfonamide (3aa): The title compound



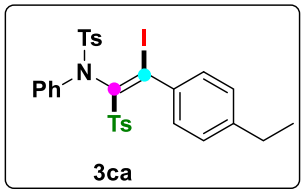
was prepared according to the general procedure (A via column chromatography of silica eluting hexane-ethyl acetate (87:13) to obtain as a lite white solid (66 mg, yield = 66%); Mp. 139.4-140 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.2 Hz, 2H), 7.70 (d, *J* = 8.3 Hz, 2H), 7.42-7.37 (m, 4H), 7.32 (d, *J* = 8.2 Hz, 2H), 7.15 (dt, *J* = 23.2, 7.1 Hz, 4H), 7.02 (d, *J* = 27.6 Hz, 1H), 7.06 (brs, 1H), 6.96 (d, *J* = 8.1 Hz, 2H), 6.75 (brs, 1H), 2.36 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.9, 144.0, 143.5, 140.0, 138.9, 137.3, 135.9, 131.4, 130.3, 129.2, 129.1, 129.0, 128.6, 128.5, 128.3, 127.8, 127.5, 123.7, 21.5, 21.5. HRMS (ESI) calculated $[M+Na]^+$ for $C_{28}H_{24}NO_4NaS_2I$ = 652.0089; found: 652.0086.

(*E*)-*N*-(2-iodo-2-(p-tolyl)-1-tosylvinyl)-4-methyl-*N*-phenylbenzenesulfonamide (3ba): The title compound



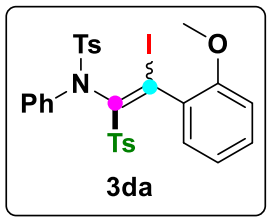
was prepared according to the general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (90:10) to obtain as a pale-yellow solid (60 mg, yield = 62%); Mp. 204-205 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 6.7 Hz, 2H), 7.69 (d, *J* = 8.2 Hz, 2H), 7.39 (d, *J* = 7.4 Hz, 3H), 7.33 (d, *J* = 8.1 Hz, 2H), 7.16 (d, *J* = 8.1 Hz, 2H), 6.96 (d, *J* = 8.0 Hz, 2H), 6.81 (brs, 4H), 2.36 (s, 3H), 2.34 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.7, 144.0, 143.5, 139.5, 139.0, 137.3, 137.2, 136.0, 131.4, 130.3, 129.1, 129.1, 128.8, 128.6, 128.6, 128.3, 127.8, 124.2, 21.6, 21.5, 21.3. HRMS (ESI) calculated $[M+Na]^+$ for $C_{29}H_{26}NO_4NaS_2I$ = 666.0246; found: 666.0244.

(*E*)-*N*-(2-(4-ethylphenyl)-2-iodo-1-tosylvinyl)-4-methyl-*N*-phenylbenzenesulfonamide (3ca): The title



compound was prepared according to the general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (90:10) to obtain as a white solid (57 mg, yield = 57%); Mp. 204-205 °C; ^{1}HNMR (400 MHz, $CDCl_3$) 7.95 (dd, *J* = 7.9, 1.6 Hz, 2H), 7.68 (d, *J* = 8.3 Hz, 2H), 7.41 (dt, *J* = 12.6, 4.2 Hz, 3H), 7.34 (d, *J* = 8.3 Hz, 2H), 7.16 (d, *J* = 8.2 Hz, 2H), 6.96 (d, *J* = 8.1 Hz, 2H), 6.83 (brs, 3H), 2.56 (q, *J* = 7.6 Hz, 2H), 2.36 (s, 3H), 2.33 (s, 3H), 1.22 (t, J = 7.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.6, 145.8, 144.0, 143.4, 139.0, 137.4, 135.1, 129.2, 129.1, 128.9, 128.6, 128.6, 128.3, 127.8, 126.9, 124.3, 28.7, 21.6, 21.5, 15.6. HRMS (ESI) calculated $[M+Na]^+$ for $C_{30}H_{28}NO_4NaS_2I$ = 680.0402; found: 680.0405.

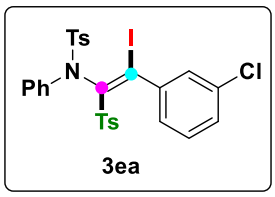
(*E*/*Z*)-*N*-(2-iodo-2-(2-methoxyphenyl)-1-tosylvinyl)-4-methyl-*N*-phenylbenzenesulfonamide (3da): The title



compound was prepared according to the general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (81:19) to obtain mixture of *E*/*Z* (63:37) isomers as a yellow solid (42 mg, yield = 41%); Mp. 142-143 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 1.6 Hz, 2H), 7.09 (d, *J* = 8.3 Hz, 2H), 7.00 (d, *J* = 8.1 Hz, 2H), 6.55 (d, *J* = 8.3 Hz, 2H), 6.42 (d, *J* = 8.3 Hz, 2H), 3.88 (s, 3H), 3.87 (s, 3H), 3.30 (s, 2H), 3.28 (s, 2H), 2.32 (s, 3H), 2.30 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.0, 166.3, 166.0, 149.1,

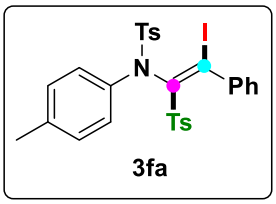
144.3, 144.0, 141.1, 140.8, 140.4, 137.6, 132.4, 130.3, 129.2, 129.2, 128.8, 128.5, 128.4, 127.5, 127.4, 127.1, 111.1, 67.0, 52.1, 52.0, 42.2, 31.4, 26.7, 21.5. HRMS (ESI) calculated $[M+Na]^+$ for $C_{29}H_{26}NO_5NaS_2I$ = 682.0195; found: 682.0196.

(*E*)-*N*-(2-(3-chlorophenyl)-2-iodo-1-tosylvinyl)-4-methyl-N-phenylbenzenesulfonamide (3ea): The title compound was prepared according to the general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (90:10) to obtain as a pale-yellow solid (44 mg, yield = 43%); Mp. 221 - 222 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.2 Hz, 2H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.43 – 7.35 (m, 5H), 7.17 (d, *J* = 8.1 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 3H), 6.99 (brs, 2H), 6.66 (brs, 1H), 2.37 (s, 3H), 2.36 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.7, 144.2, 144.0, 138.8, 138.4, 137.2, 135.8, 135.4, 129.2, 129.1, 129.1, 128.7, 128.4, 127.8, 121.7, 21.6. HRMS (ESI) calculated $[M+Na]^+$ for $C_{28}H_{23}ClINNaO_4S_2$ = 685.9693; found: 685.9696.



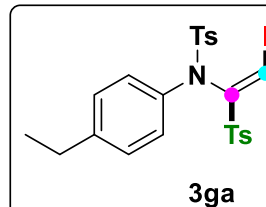
3ea

(*E*)-*N*-(2-iodo-2-phenyl-1-tosylvinyl)-4-methyl-*N*-(p-tolyl)benzenesulfonamide (3fa): The title compound was prepared according to the general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (91:9) to obtain as a white solid (59 mg, yield = 59%); Mp. 153-154 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.1 Hz, 2H), 7.68 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 7.19 (dd, *J* = 10.9, 8.5 Hz, 4H), 7.10 (brs, 2H), 6.96 (d, J = 8.0 Hz, 3H), 6.68 (brs, 1H), 2.39 (s, 3H), 2.37 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.1, 143.9, 143.5, 140.0, 138.5, 137.4, 136.3, 136.0, 131.5, 130.4, 129.8, 129.1, 129.0, 128.8, 128.6, 127.8, 123.4, 21.6, 21.5, 21.2. HRMS (ESI) calculated $[M+Na]^+$ for $C_{29}H_{26}NO_4NaS_2I$ = 666.0246; found: 666.0252.



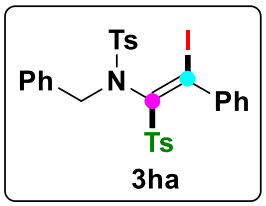
3fa

(*E*)-*N*-(4-ethylphenyl)-*N*-(2-iodo-2-phenyl-1-tosylvinyl)-4-methylbenzenesulfonamide (3ga): The title compound was prepared according to the general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (92:8) to obtain as a yellow solid (55 mg, yield = 55%); Mp. 200-201 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.5 Hz, 2H), 7.67 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.22 (d, *J* = 8.5 Hz, 2H), 7.17 (d, *J* = 8.1 Hz, 2H), 7.11-7.16 (m, 2H), 6.96 (d, *J* = 8.1 Hz, 3H), 6.70 (s, 1H), 2.70 (q, *J* = 7.6 Hz, 2H), 2.37 (s, 3H), 2.33 (s, 3H), 1.28 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.0, 144.7, 143.9, 143.5, 140.1, 137.4, 136.4, 136.1, 129.1, 129.0, 128.9, 128.6, 127.8, 123.4, 28.5, 21.6, 21.5, 15.2. HRMS (ESI) calculated $[M+Na]^+$ for $C_{30}H_{28}NO_4NaS_2I$ = 680.0402; found: 680.0401.



3ga

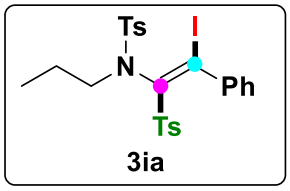
(*E*)-*N*-benzyl-*N*-(2-iodo-2-phenyl-1-tosylvinyl)-4-methylbenzenesulfonamide (3ha): The title compound was prepared according to the general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (92:8) to obtain mixture as a white solid (25 mg, yield = 25%); Mp. 197-198 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 8.3 Hz, 2H), 7.57 (dd, *J* = 6.3, 3.1 Hz, 2H), 7.41 – 7.33 (m, 5H), 7.18 (d, *J* = 8.3 Hz, 2H), 7.06 (t, *J* = 6.9 Hz, 2H), 6.93 (d, *J* = 8.1 Hz, 2H), 6.85 (brs, 2H), 6.56 (brs, 1H), 5.06 (d, *J* = 13.8 Hz, 1H), 4.67 (d, *J* = 13.7 Hz, 1H), 2.46 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.3, 144.2, 143.4, 140.3, 137.6, 136.6, 133.2, 131.5,



3ha

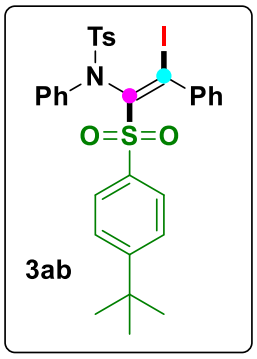
129.2, 129.2, 129.0, 128.8, 128.7, 128.2, 127.5, 125.3, 54.0, 21.7, 21.5. HRMS (ESI) calculated $[M+Na]^+$ for $C_{29}H_{26}NO_4NaS_2I$ = 666.0246; found: 666.0252.

(*E*)-*N*-(2-iodo-2-phenyl-1-tosylvinyl)-4-methyl-*N*-propylbenzenesulfonamide (3ia): The title compound was



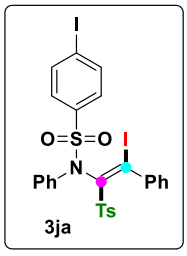
prepared according to the general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (88:12) to obtain as a white solid (19 mg, yield = 20%); Mp. 174-174 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.2 Hz, 2H), 7.84 (d, *J* = 8.2 Hz, 1H), 7.42 (d, *J* = 8.1 Hz, 1H), 7.33 (d, *J* = 8.1 Hz, 2H), 7.23 (d, *J* = 8.2 Hz, 2H), 7.13 (t, *J* = 7.0 Hz, 1H), 7.03 (brs, 2H), 6.96 (d, *J* = 8.1 Hz, 2H), 3.78 – 3.68 (m, 1H), 3.66 – 3.55 (m, 1H), 2.43 (s, 3H), 2.32 (s, 3H), 2.06 – 1.79 (m, 3H), 0.97 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.1, 146.5, 144.0, 143.5, 140.2, 137.3, 136.5, 131.4, 130.3, 129.1, 129.0, 129.0, 128.1, 127.9, 127.6, 123.8, 52.3, 23.0, 21.8, 21.6, 21.5, 11.5. HRMS (ESI) calculated $[M+Na]^+$ for $C_{25}H_{26}NO_4NaS_2I$ = 618.0246; found: 618.0253.

(*E*)-*N*-(1-((4-(tert-butyl)phenyl)sulfonyl)-2-iodo-2-phenylvinyl)-4-methyl-*N*-phenylbenzenesulfonamide



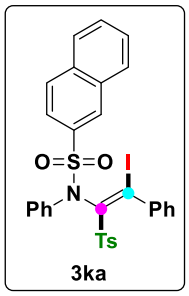
(3ab): The title compound was prepared according to the general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (90:10) to obtain as a white solid (46 mg, yield = 45%); Mp. 201-202 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 8.00 – 7.94 (m, 2H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.44 – 7.38 (m, 5H), 7.18 (dd, *J* = 11.5, 8.6 Hz, 5H), 7.09 (s, 2H), 6.83 (brs, 1H), 6.65 (brs, 1H), 2.36 (s, 3H), 1.30 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.4, 148.0, 144.0, 140.0, 138.9, 137.1, 135.9, 129.2, 129.1, 129.0, 128.7, 128.6, 128.4, 127.6, 125.5, 123.8, 35.0, 31.0, 21.6. HRMS (ESI) calculated $[M]^+$ for $C_{31}H_{30}NO_4S_2I$ = 671.0661; found: 671.0662.

(*E*)-4-iodo-*N*-(2-iodo-2-phenyl-1-tosylvinyl)-*N*-phenylbenzenesulfonamide (3ja): The title compound was



prepared according to the general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (91:9) to obtain as a pale-yellow solid (63 mg, yield = 58%); Mp. 166-167 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.1 Hz, 2H), 7.72 (d, *J* = 8.7 Hz, 2H), 7.49 – 7.41 (m, 5H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.12 (t, *J* = 7.0 Hz, 2H), 6.97 (d, *J* = 8.1 Hz, 3H), 6.94 – 6.82 (m, 1H), 6.68 (brs, 1H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.6, 143.7, 139.8, 138.6, 138.4, 137.2, 137.1, 130.3, 129.4, 129.2, 129.1, 128.8, 128.7, 128.3, 127.8, 127.5, 124.2, 101.1, 21.5. HRMS (ESI) calculated $[M+Na]^+$ for $C_{27}H_{21}I_2NNaO_4S_2$ = 763.8893; found: 763.8892.

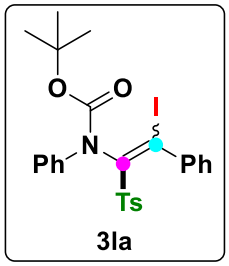
(*E*)-*N*-(2-iodo-2-phenyl-1-tosylvinyl)-*N*-phenylnaphthalene-2-sulfonamide (3ka): The title compound was



prepared according to the general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (88:12) to obtain as a pale-yellow solid (44 mg, yield = 43%); Mp. 188-189 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 8.38 (s, 1H), 8.00 – 7.94 (m, 2H), 7.89 – 7.79 (m, 5H), 7.56 (dt, *J* = 14.6, 6.9 Hz, 3H), 7.44 – 7.31 (m, 6H), 7.12 (d, *J* = 6.8 Hz, 2H), 6.98 (d, *J* = 7.9 Hz, 3H), 6.77 (brs, 1H), 2.35 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.0, 143.6, 140.1, 139.0,

137.3, 136.0, 135.1, 131.6, 130.8, 129.5, 129.3, 129.2, 129.1, 129.1, 128.8, 128.8, 128.5, 127.9, 127.9, 127.7, 127.7, 127.1, 124.3, 123.7, 21.5. HRMS (ESI) calculated $[M+Na]^+$ for $C_{31}H_{24}INNaO_4S_2$ = 688.0083; found: 688.0085.

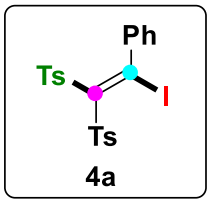
(*E*/*Z*)-*tert*-butyl (2-iodo-2-phenyl-1-tosylvinyl)(phenyl)carbamate (3la): The title compound was prepared



3la

according to the general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (92:8) to obtain mixture of *E*/*Z* (62:38) isomers as a pale-yellow solid (53 mg, yield = 60%); Mp. 115-116 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.6 Hz, 1.88H), 7.56 (d, *J* = 7.8 Hz, 1.36H), 7.44 (d, *J* = 7.5 Hz, 1.07H), 7.42 – 7.29 (m, 5.8H), 7.29 – 7.13 (m, 7.16H), 7.11 (d, *J* = 8.1 Hz, 2.25H), 7.01 (s, 1.14H), 6.98 (d, *J* = 8.1 Hz, 2.29H), 2.35 (s, 1.83H), 2.32 (s, 2.99H), 1.67 (s, 5.68H), 1.58 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.5, 145.7, 144.7, 139.6, 136.9, 136.9, 135.6, 131.3, 130.1, 129.3, 129.1, 128.9, 128.8, 128.5, 128.4, 128.2, 127.9, 127.8, 126.6, 1256.0, 125.5, 124.3, 114.3, 83.5, 82.9, 28.1, 28.1, 21.6, 21.5. HRMS (ESI) calculated $[M+Na]^+$ for $C_{26}H_{26}INNaO_4S$ = 598.05194; found: 598.05207.

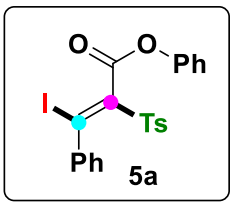
4,4'-(2-iodo-2-phenylethene-1,1-diyldisulfonyl)bis(methylbenzene) (4a): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (91:9)



4a

to obtain as a pale-yellow solid (66 mg, yield = 80%); Mp. 145-146 °C; ^{1}HNMR (600 MHz, $CDCl_3$) δ 8.20 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.5 Hz, 2H), 7.29 – 7.22 (m, 3H), 7.17 (d, *J* = 8.5 Hz, 2H), 7.09 – 6.99 (m, 2H), 2.49 (s, 3H), 2.39 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.2, 145.4, 144.7, 144.5, 137.9, 136.4, 129.7, 129.5, 129.5, 129.3, 128.4, 127.8, 127.6, 126.4, 21.8, 21.6. HRMS (ESI) calculated $[M+H]^+$ for $C_{22}H_{20}O_4S_2I$ = 538.9848; found: 538.9850.

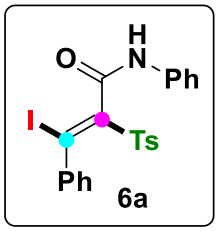
phenyl (*E*)-3-iodo-3-phenyl-2-tosylacrylate (5a): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (93:7) to obtain as a white solid



5a

(54 mg, yield = 70%); Mp. 98- 99 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.50 – 7.42 (m, 2H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.35 – 7.24 (m, 6H), 7.13 (d, *J* = 8.3 Hz, 4H), 2.39 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.2, 150.3, 146.2, 145.2, 139.4, 137.1, 129.8, 129.6, 129.4, 128.4, 127.9, 127.4, 126.7, 121.2, 115.0, 21.7. HRMS (ESI) calculated $[M]^+$ for $C_{22}H_{17}O_4SI$ = 503.9892; found: 503.9896.

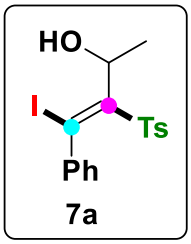
(*E*)-3-iodo-*N*,3-diphenyl-2-tosylacrylamide (6a): The title compound was prepared according to the general



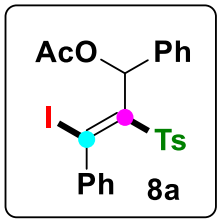
6a

procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (83:17) to obtain as a pale-yellow solid (65 mg, yield = 87%); Mp. 206-207 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.97 (s, 1H), 7.59 (d, *J* = 7.7 Hz, 2H), 7.53 (d, *J* = 8.3 Hz, 2H), 7.39 (t, *J* = 7.8 Hz, 2H), 7.33 – 7.28 (m, 3H), 7.20 (d, *J* = 7.9 Hz, 5H), 2.40 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.8, 147.2, 145.2, 140.5, 136.8, 129.6, 129.6, 129.1, 128.4, 127.9, 127.0, 125.4, 120.5, 116.0, 21.7. HRMS (ESI) calculated $[M+Na]^+$ for $C_{22}H_{18}INNaO_3S$ = 525.9944; found: 525.9941.

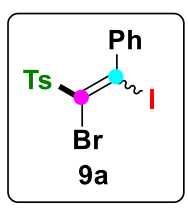
(*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-2-ol (7a): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (90:10) to obtain as a white solid (47 mg, yield = 79%); Mp. 128-129 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.15 (d, *J* = 8.3 Hz, 2H), 7.13 (d, *J* = 7.4 Hz, 1H), 7.00 (d, *J* = 8.2 Hz, 3H), 6.96 (s, 1H), 6.72 (brs, 1H), 5.18 (q, *J* = 6.8 Hz, 1H), 3.84 (brs, 1H), 2.34 (s, 3H), 1.83 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.0, 143.8, 141.6, 138.0, 129.0, 128.6, 127.4, 127.1, 115.6, 79.1, 22.2, 21.5. HRMS (ESI) calculated $[M+Na]^+$ for $C_{17}H_{17}INaO_3S$ = 450.9835; found: 450.9837.



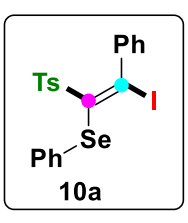
(*E*)-3-iodo-1,3-diphenyl-2-tosylallyl acetate (8a): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (87:13) to obtain as a white solid (71 mg, yield = 88%); Mp. 142-143 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.6 Hz, 2H), 7.45 – 7.34 (m, 4H), 7.21 (d, *J* = 30.9 Hz, 3H), 7.05 (s, 1H), 6.97 (d, *J* = 8.2 Hz, 2H), 6.91 (d, *J* = 8.1 Hz, 2H), 6.77 (s, 1H), 2.34 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.8, 147.1, 143.6, 142.6, 137.8, 136.2, 128.8, 128.7, 128.3, 127.9, 127.7, 127.6, 127.5, 125.5, 119.8, 79.5, 21.4, 21.0. HRMS (ESI) calculated $[M+Na]^+$ for $C_{24}H_{21}INaO_4S$ = 555.0097; found: 555.0098.



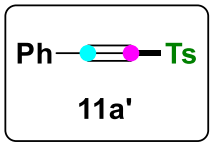
(E/Z)-1-((1-bromo-2-iodo-2-phenylvinyl)sulfonyl)-4-methylbenzene (9a): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (95:5) to obtain mixture of *E*/*Z* (92:8) isomers as a pale-yellow solid (57 mg, yield = 83%); Mp. 144-145 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.3 Hz, 0.14H), 7.56 (d, *J* = 8.3 Hz, 1.94H), 7.42 – 7.35 (m, 0.35H), 7.31 (dd, *J* = 5.0, 1.7 Hz, 3H), 7.26 – 7.19 (m, 4.04H), 7.16 – 7.13 (m, 0.19H), 2.48 (s, 0.23H), 2.42 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.0, 156.0, 145.1, 142.3, 135.9, 132.4, 129.8, 129.6, 129.3, 129.1, 128.7, 128.4, 127.8, 127.0, 126.6, 119.9, 29.6, 21.7. HRMS (ESI) calculated $[M+Na]^+$ for $C_{15}H_{12}BrINaO_2S$ = 484.8678; found: 484.8679.



(*Z*)-(2-iodo-2-phenyl-1-tosylvinyl)(phenyl)selane (10a): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (94:6) to obtain as a yellow solid (48 mg, yield = 58%); Mp. 154-155 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.52 – 7.47 (m, 2H), 7.45 (d, *J* = 8.3 Hz, 2H), 7.32 – 7.28 (m, 3H), 7.27 – 7.23 (m, 3H), 7.21 – 7.17 (m, 2H), 7.09 (d, *J* = 8.1 Hz, 2H), 2.36 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.9, 144.2, 143.8, 136.6, 131.6, 131.5, 131.3, 129.4, 129.2, 129.1, 128.6, 127.8, 127.8, 126.9, 21.6. HRMS (ESI) calculated $[M+Na]^+$ for $C_{21}H_{17}INaO_2SSe$ = 562.9051; found: 562.9052.

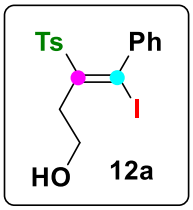


1-methyl-4-((phenylethynyl)sulfonyl)benzene (11a'): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (96:4) to obtain as a yellow solid (8 mg, yield = 20%); Mp. 78-79 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.3 Hz, 2H), 7.55 – 7.50 (m, 2H), 7.50 – 7.44 (m, 1H), 7.42 – 7.34 (m,

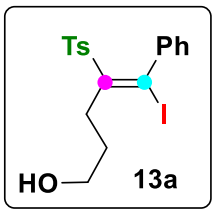


4H), 2.47 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.3, 138.9, 132.7, 131.4, 130.0, 128.6, 127.5, 118.0, 92.9, 85.6, 21.7. HRMS (ESI) calculated $[M+Na]^+$ for $C_{15}H_{12}NaO_2S$ = 279.04502; found: 279.04524.

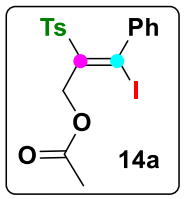
(*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-ol (12a): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (78:22) to obtain as a yellow solid (60 mg, yield = 93%); Mp. 124-125 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.27 (d, *J* = 8.3 Hz, 2H), 7.19 – 7.10 (m, 3H), 7.05 (d, *J* = 8.4 Hz, 2H), 6.97 (d, *J* = 6.8 Hz, 2H), 4.06 (t, *J* = 6.6 Hz, 2H), 3.26 (t, *J* = 6.6 Hz, 2H), 2.41 (s, 1H), 2.35 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 146.1, 143.9, 142.4, 137.2, 129.3, 128.6, 127.6, 127.5, 117.4, 60.9, 42.1, 21.5. HRMS (ESI) calculated $[M+Na]^+$ for $C_{17}H_{17}INaO_3S$ = 450.9835; found: 450.9838.



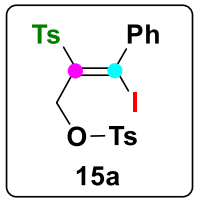
(*E*)-5-iodo-5-phenyl-4-tosylpent-4-en-1-ol (13a): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (74:26) to obtain as a yellow solid (52 mg, yield = 79%); Mp. 136-137 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.24 (d, *J* = 7.9 Hz, 2H), 7.21 – 7.11 (m, 3H), 7.07 (d, *J* = 7.8 Hz, 2H), 6.99 (d, *J* = 7.0 Hz, 2H), 3.81 (t, *J* = 5.8 Hz, 2H), 3.03 (t, *J* = 4.0 Hz, 2H), 2.35 (s, 3H), 2.16 (s, 1H), 2.11 – 2.01 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.3, 143.9, 142.6, 137.4, 129.3, 128.5, 127.7, 127.5, 127.5, 116.0, 61.8, 36.1, 31.2, 21.5. HRMS (ESI) calculated $[M+Na]^+$ for $C_{18}H_{19}INaO_3S$ = 464.9991; found: 464.9993.



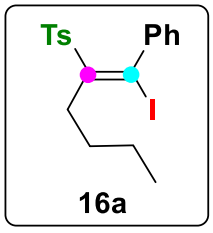
(*E*)-3-iodo-3-phenyl-2-tosylallyl acetate (14a): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (82:18) to obtain as a white solid (53 mg, yield = 74%); Mp. 163-164 °C; ^{1}HNMR (597 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.2 Hz, 2H), 7.25 – 7.18 (m, 3H), 7.11 (d, *J* = 8.1 Hz, 2H), 7.06 (d, *J* = 7.0 Hz, 2H), 5.29 (s, 2H), 2.37 (s, 3H), 2.11 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.1, 144.2, 143.5, 142.0, 137.2, 129.3, 129.1, 127.8, 127.6, 127.2, 123.5, 68.9, 21.5, 20.6. HRMS (ESI) calculated $[M+Na]^+$ for $C_{18}H_{17}INaO_4S$ = 478.9784; found: 478.9783.



(*Z*)-(2-iodo-2-phenyl-1-tosylvinyl)(phenyl)selane (15a): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (83:17) to obtain as a white solid (79 mg, yield = 91%); Mp. 95-96 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.1 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.28 – 7.12 (m, 6H), 7.06 (d, *J* = 8.0 Hz, 2H), 6.94 (d, *J* = 7.2 Hz, 2H), 5.25 (s, 2H), 2.45 (s, 3H), 2.36 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.2, 144.4, 142.0, 141.7, 136.7, 132.1, 129.8, 129.3, 128.3, 127.8, 127.5, 127.0, 126.3, 73.3, 21.6, 21.5. HRMS (ESI) calculated $[M+Na]^+$ for $C_{23}H_{21}INaO_5S_2$ = 590.9767; found: 590.9765.

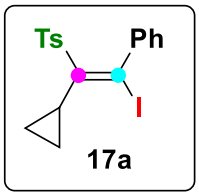


(*E*)-1-((1-iodo-1-phenylhex-1-en-2-yl)sulfonyl)-4-methylbenzene (16a): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (95:5) to obtain as a white solid (60 mg, yield = 90%); Mp. 84-85 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.23 (d, *J* = 8.3 Hz, 2H), 7.20 – 7.11 (m, 3H), 7.06 (d, *J* = 8.0 Hz, 2H),

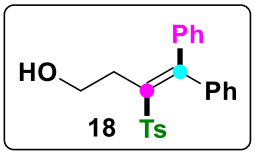


6.99 (d, *J* = 6.5 Hz, 2H), 2.93 – 2.87 (m, 2H), 2.35 (s, 3H), 1.75 (tt, *J* = 8.0, 6.5 Hz, 2H), 1.49 (dd, *J* = 14.8, 7.4 Hz, 2H), 1.00 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.0, 143.6, 142.7, 137.8, 129.2, 128.4, 127.8, 127.5, 115.0, 39.5, 30.3, 22.7, 21.5, 13.7. HRMS (ESI) calculated $[M+Na]^+$ for $C_{19}H_{21}INaO_2S$ = 463.0199; found: 463.0196.

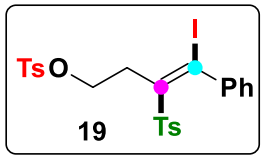
(*E*)-1-((1-cyclopropyl-2-iodo-2-phenylvinyl)sulfonyl)-4-methylbenzene (17): The title compound was prepared according to the general procedure (B) via column chromatography of silica eluting hexane-ethyl acetate (95:5) to obtain as a white solid (51 mg, yield = 77%); Mp. 159-160 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.2 Hz, 2H), 7.21-7.12 (m, 7H), 2.36 (s, 3H), 1.39 – 1.31 (m, 1H), 1.08 (d, *J* = 7.0 Hz, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.6, 143.6, 142.8, 137.6, 129.1, 128.9, 128.2, 127.8, 127.6, 119.8, 21.5, 20.6, 12.0. HRMS (ESI) calculated $[M+Na]^+$ for $C_{18}H_{17}INaO_2S$ = 446.9886; found: 446.9888.



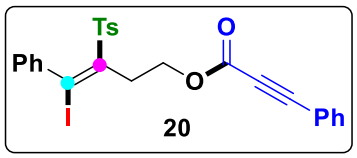
4,4-diphenyl-3-tosylbut-3-en-1-ol (18): The title compound was prepared according to the general procedure (C) via column chromatography of silica eluting hexane-ethyl acetate (75:25) to obtain as a white solid (79 mg, yield = 89%); Mp. 149-150 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.28 (ddd, *J* = 21.0, 14.3, 7.7 Hz, 5H), 7.10 (ddd, *J* = 24.9, 16.0, 7.8 Hz, 7H), 6.94 (d, *J* = 7.1 Hz, 2H), 3.87 (t, *J* = 6.4 Hz, 2H), 2.89 (t, *J* = 6.4 Hz, 2H), 2.33 (s, 3H), 2.32 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.9, 143.2, 140.8, 140.0, 138.9, 138.0, 129.0, 128.8, 128.6, 128.0, 127.7, 127.5, 127.5, 127.4, 62.2, 33.8, 21.4. HRMS (ESI) calculated $[M+H]^+$ for $C_{24}H_{24}O_5S_2I$ = 583.0110; found: 583.0103.



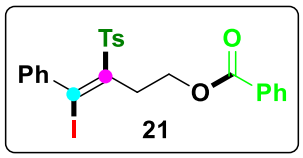
(*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-yl 4-methylbenzenesulfonate (19): The title compound was prepared according to the general procedure (D) via column chromatography of silica eluting hexane-ethyl acetate (82:18) to obtain as a white solid (98 mg, yield = 72%); Mp. 133-134 °C; ^{1}HNMR (597 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.3 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.19 (s, 2H), 7.17 (dt, *J* = 2.6, 2.0 Hz, 1H), 7.16 – 7.12 (m, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 6.94 (d, *J* = 7.0 Hz, 2H), 4.40 (t, *J* = 7.5 Hz, 2H), 3.34 – 3.29 (m, 2H), 2.45 (s, 3H), 2.35 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 144.9, 144.2, 143.7, 142.3, 136.8, 132.9, 129.9, 129.4, 128.7, 128.0, 127.6, 127.6, 127.3, 119.3, 67.0, 39.0, 21.6, 21.5. HRMS (ESI) calculated $[M+H]^+$ for $C_{23}H_{23}O_3S$ = 379.1368; found: 379.1379.



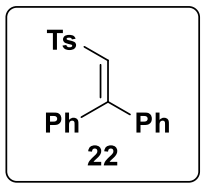
(*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-yl 3-phenylpropiolate (20): The title compound was prepared according to the general procedure (E) via column chromatography of silica eluting hexane-ethyl acetate (90:10) to obtain as a white solid (65 mg, yield = 50%); Mp. 165-166 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.0 Hz, 2H), 7.45 (ddd, *J* = 6.6, 3.9, 1.3 Hz, 1H), 7.38 (t, *J* = 7.4 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.22 – 7.12 (m, 3H), 7.07 (d, *J* = 8.1 Hz, 2H), 7.00 (d, *J* = 6.6 Hz, 2H), 4.63 (t, *J* = 7.3 Hz, 2H), 3.40 (t, *J* = 7.3 Hz, 2H), 2.35 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.8, 144.8, 144.1, 142.5, 137.2, 133.0, 130.7, 129.3, 128.7, 128.5, 127.7, 127.6, 127.5, 119.5, 118.6, 86.9, 80.5, 63.0, 38.5, 21.5. HRMS (ESI) calculated $[M+H]^+$ for $C_{26}H_{22}O_4SI$ = 557.0283; found: 557.0281.



(*E*)-4-iodo-4-phenyl-3-tosylbut-3-en-1-yl benzoate (21): The title compound was prepared according to the general procedure (F) via column chromatography of silica eluting hexane-ethyl acetate (89:11) to obtain as a white solid (106 mg, yield = 85%); Mp. 138-139 °C; ^{1}HNMR (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 7.4 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.26 – 7.20 (m, 2H), 7.14 (dt, *J* = 20.2, 7.1 Hz, 3H), 7.02 (d, *J* = 8.0 Hz, 2H), 6.97 (d, *J* = 7.0 Hz, 2H), 4.74 (t, *J* = 6.9 Hz, 2H), 3.50 (t, *J* = 6.9 Hz, 2H), 2.33 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 13C NMR (101 MHz, $CDCl_3$) δ 166.4, 145.6, 143.9, 142.4, 137.4, 132.9, 130.0, 129.7, 129.2, 128.6, 128.3, 127.6, 127.5, 127.5, 118.2, 62.3, 38.9, 21.5. HRMS (ESI) calculated $[M+H]^+$ for $C_{24}H_{22}O_4SI$ = 533.0283; found: 533.0287.



(2-tosylethene-1,1-diyl)dibenzene (22)[1]: The title compound was prepared according to the modified general procedure (A) via column chromatography of silica eluting hexane-ethyl acetate (85:15) to obtain as a white solid (12 mg, yield = 8%); Mp. 140-141 °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). ^{13}C 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.



7. References

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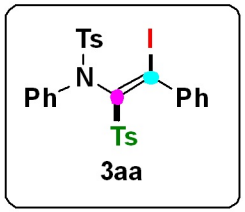
M0425-1
LJ3-3

7.97 7.94 7.71 7.69 7.42 7.41 7.39 7.37 7.33 7.31 7.19 7.18 7.16 7.13 7.12 7.10 7.06 6.97 6.95 6.75

2.36 2.33

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H



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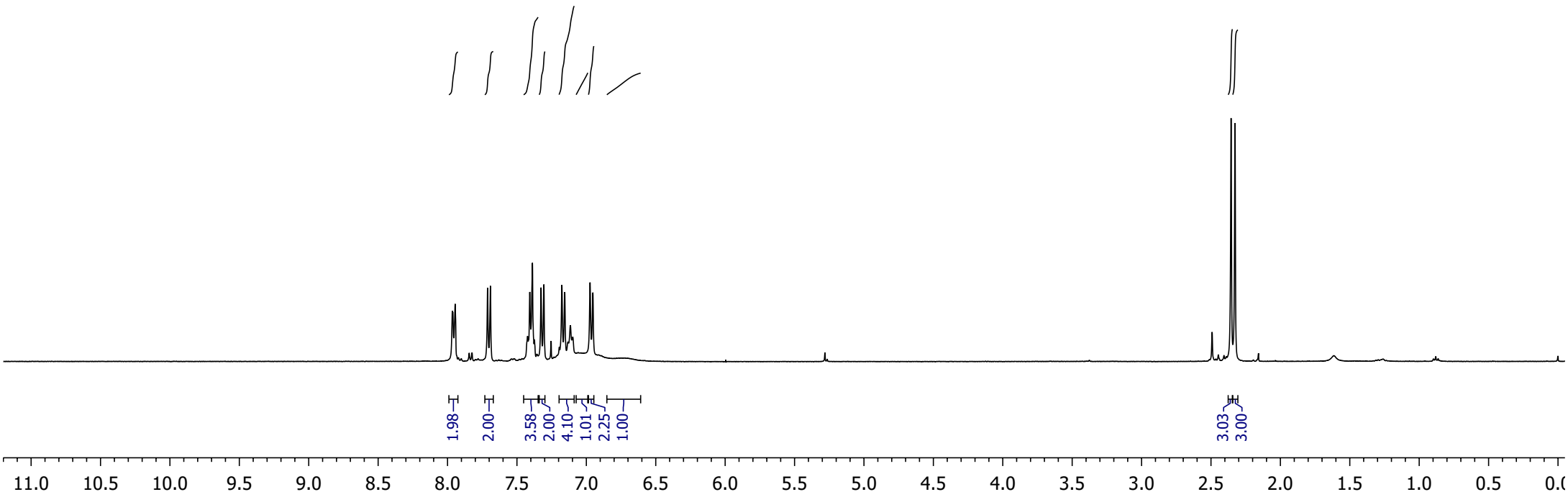


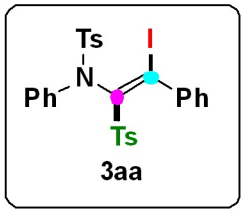
M0425-2
LJ3-3

147.9 144.0 143.5 140.0 138.9 137.3 135.9 131.4 130.3 129.2 129.1 129.0 128.6 128.5 128.3 127.8 127.5 123.7

77.3 77.0 76.7

21.6 21.5

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



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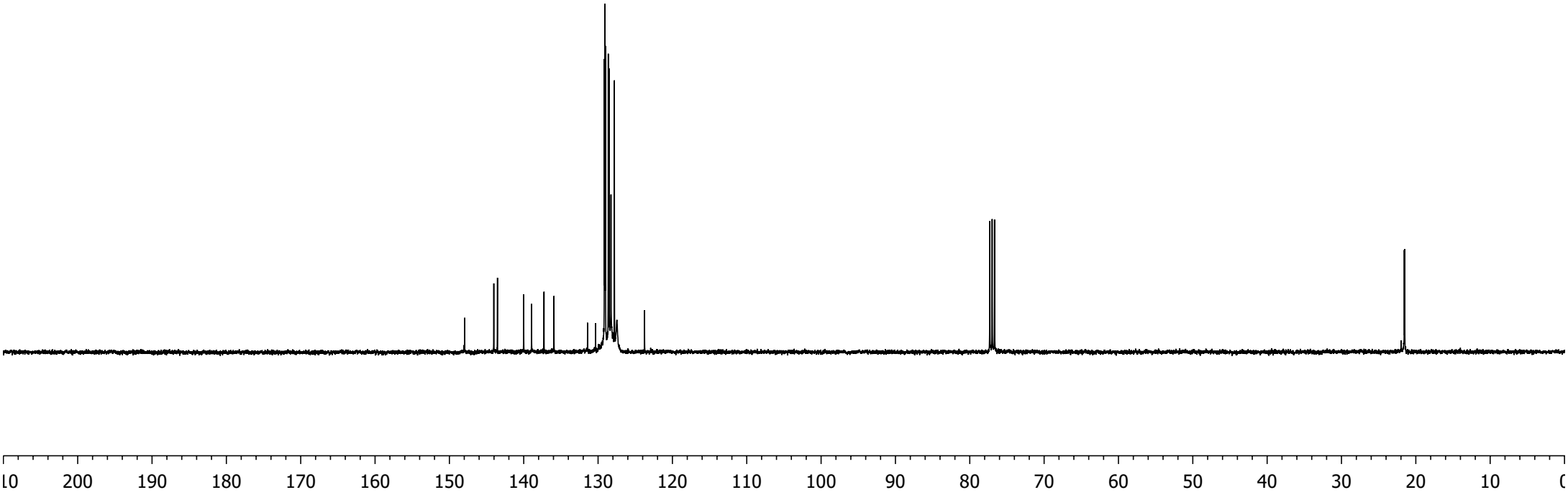

MR-CC-2D

exp15 NOESY

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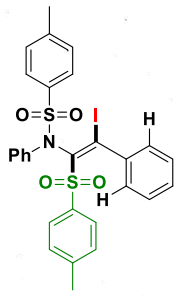
sample		PFGflg	y
ACQUISITION		hsglvl	1200
sw	6410.3	SPECIAL	
at	0.150	temp	not used
np	1920	gain	22
fb	not used	spin	0
ss	32	F2 PROCESSING	
d1	1.000	gf	0.069
nt	16	gfs	not used
2D ACQUISITION		fn	4096
sw1	6410.3	F1 PROCESSING	
ni	128	gf1	0.016
TRANSMITTER		gfs1	not used
tn	H1	proc1	lp
sfrq	400.392	fn1	4096
tof	387.0	DISPLAY	
tpwr	55	sp	-82.2
pw	12.300	wp	4081.5
NOESY		sp1	-82.2
mixN	0.600	wp1	4081.5
PRESATURATION		rf1	802.1
satmode	n	rfp	0
wet	n	rfl1	802.1
DECOUPLER		rfp1	0
dn	C13	PLOT	
dm	nnn	wc	140.0
		sc	5.0
		wc2	140.0
		sc2	5.0
		vs	1327
		th	6
		ai ph	



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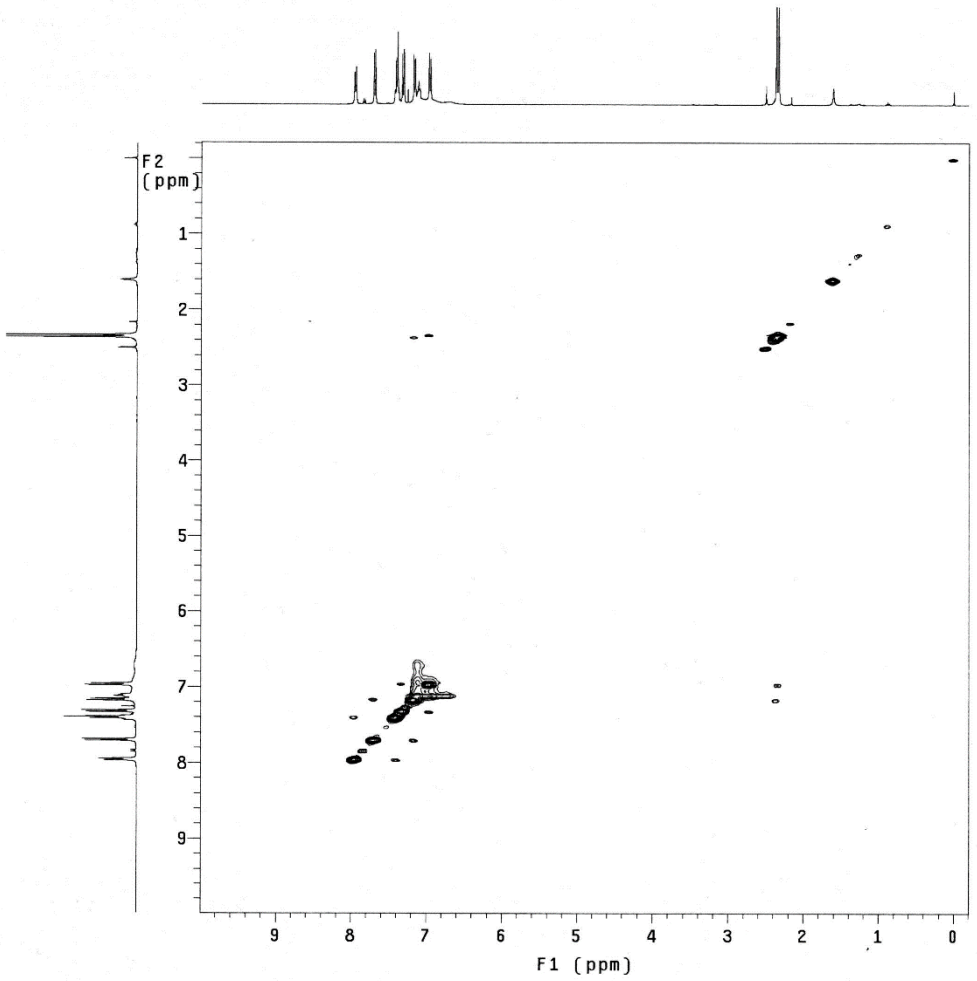

MR-CC-2D

exp14 gCOSY

SAMPLE		FLAGS	
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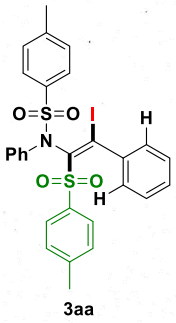
solvent	cdcl3	sspul	y
sample		hsglvl	1200
ACQUISITION		SPECIAL	
sw	6410.3	temp	not used
at	0.150	gain	22
np	1920	spin	0
fb	not used	F2 PROCESSING	
ss	32	sb	-0.075
d1	1.000	sbs	not used
nt	20	fn	4096
2D ACQUISITION		F1 PROCESSING	
sw1	6410.3	sb1	-0.020
ni	128	sbs1	not used
d2	0	proc1	lp
PRESATURATION		fn1	4096
satmode	n	DISPLAY	
wet	n	sp	-82.2
TRANSMITTER		wp	4081.5
tn	H1	sp1	-82.2
sfrq	400.392	wp1	4081.5
tof	387.0	rf1	802.1
tpwr	55	rfp	0
pw	12.300	rf11	802.1
GRADIENTS		rfp1	0
gzlvlE	1000	PLOT	
gtE	0.001000	wc	140.0
EDratio	1.000	sc	5.0
gstab	0.000500	wc2	140.0
DECOUPLER		sc2	5.0
dn	C13	vs	155
dm	nnn	th	11
		ai av	



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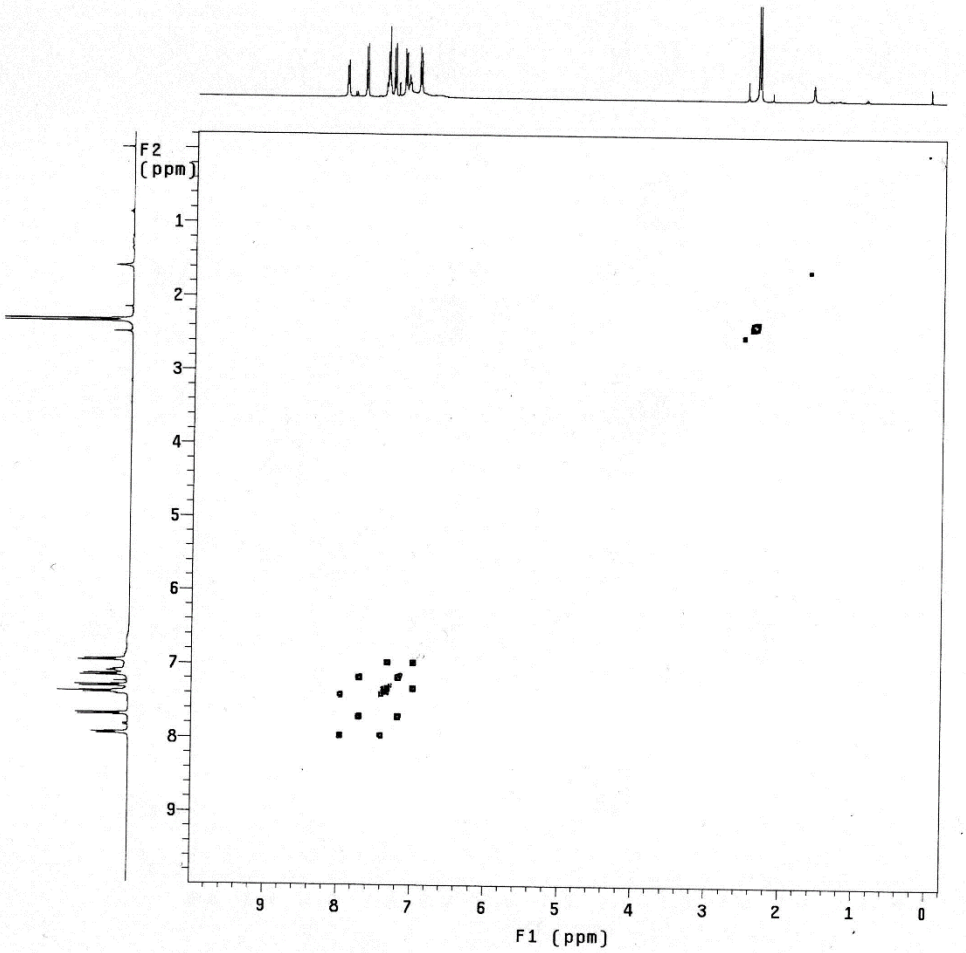

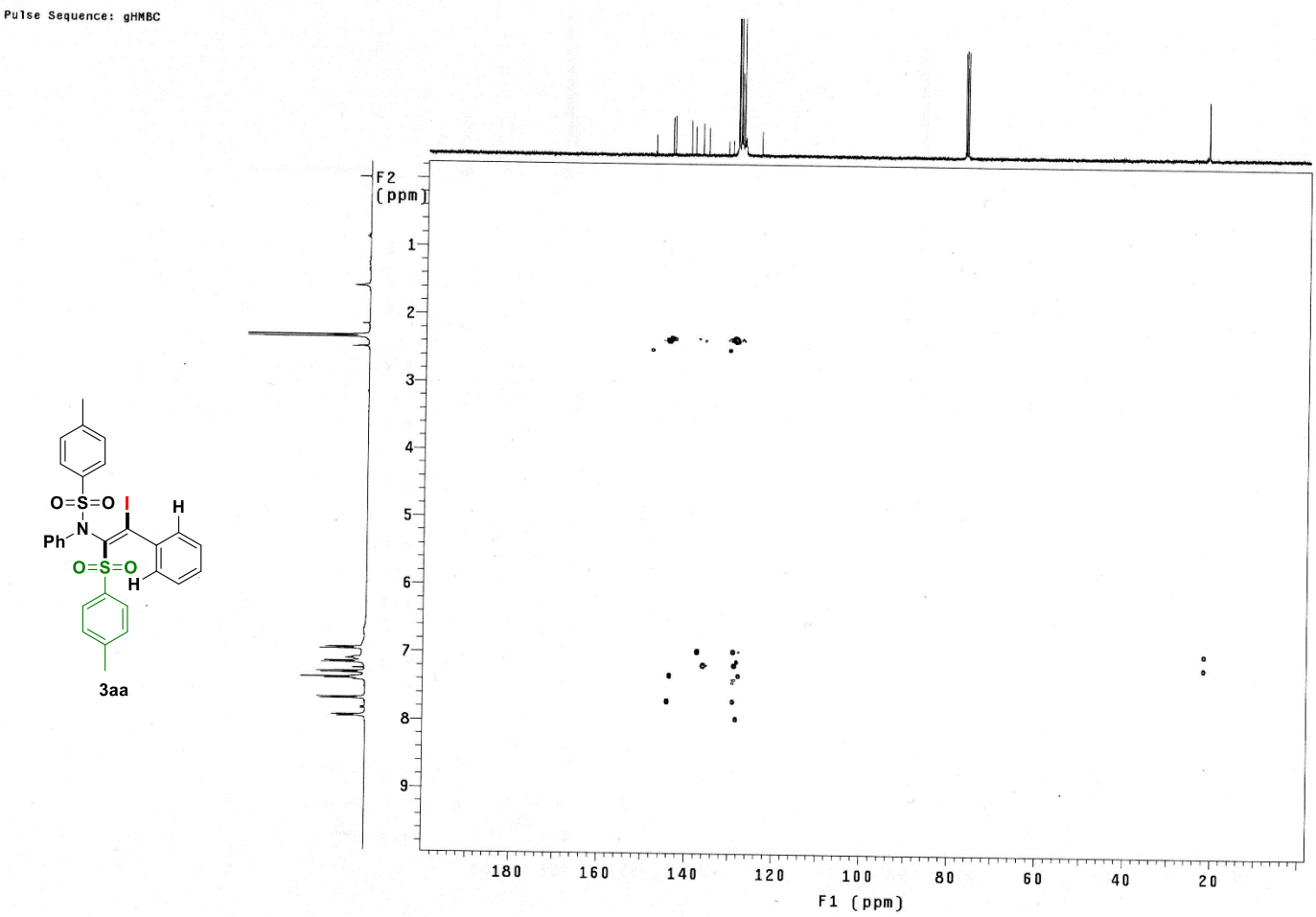
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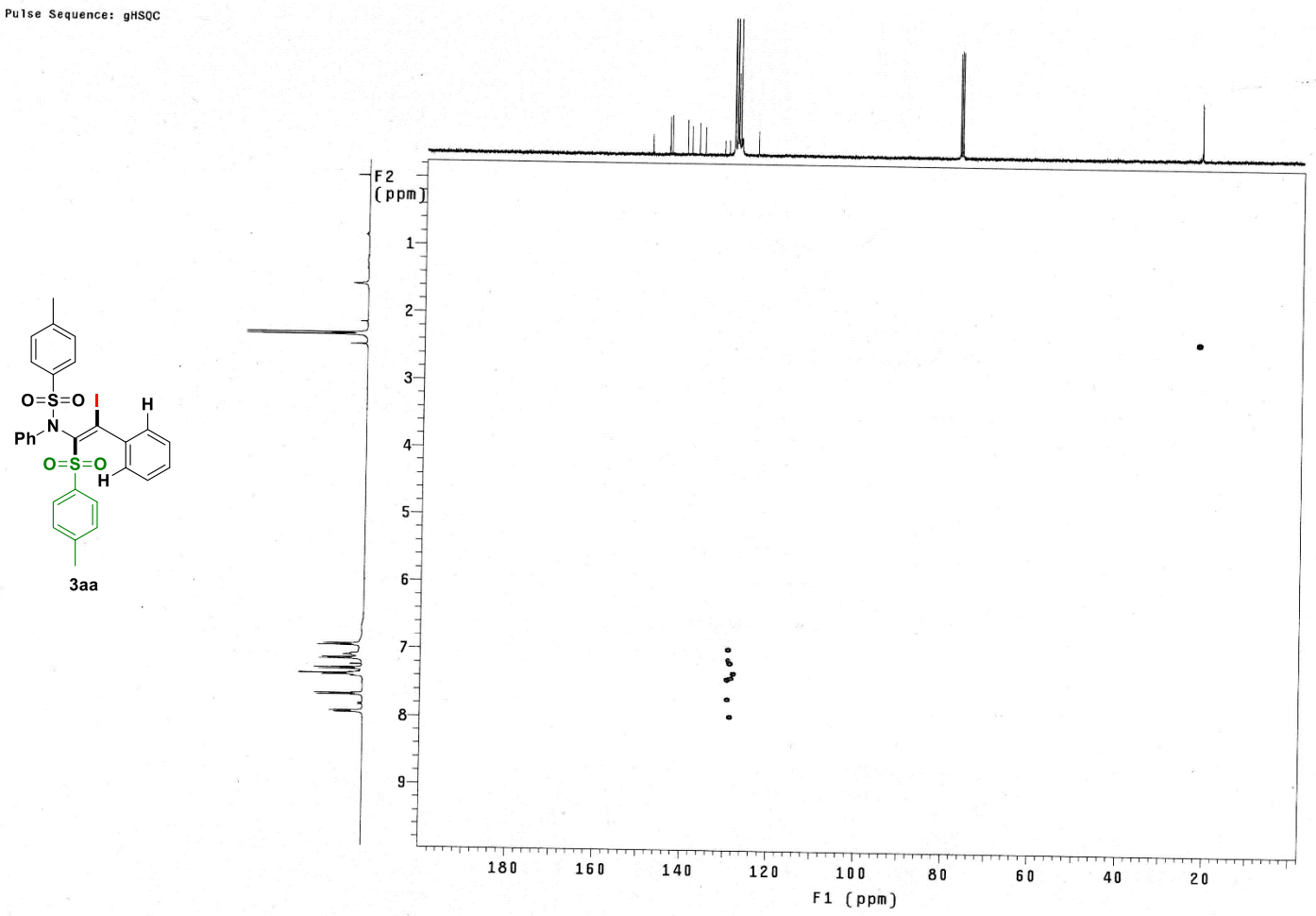
F1 (ppm)
180
160
140
120
100
80
60
40
20
1
2
3
4
5
6
7
8
9
O=S=O
I
H
N
Ph
O=S=O
H

3aa

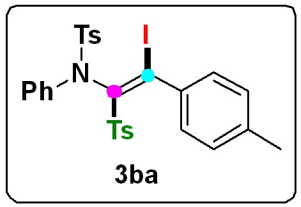

Pulse Sequence: gHSQC
F2 (ppm)
F1 (ppm)
3aa

M0027-10
LJ3-4

7.95 7.93 7.70 7.68 7.40 7.38 7.34 7.32 7.26 7.17 7.15 6.97 6.95 6.81

2.50 2.36 2.34 2.28

Solvent	$CDCl_3$
Spectrometer Frequency	400.40
Nucleus	^1H



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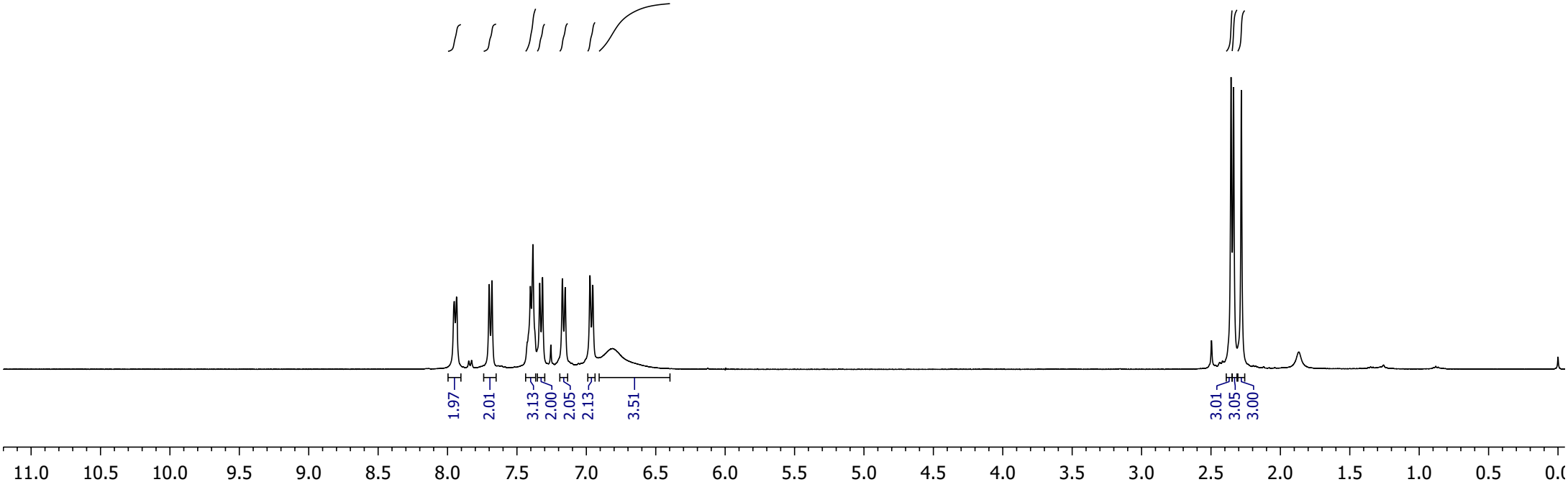

M0027-11
LJ3-4

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143.5
139.5
139.0
137.3
137.2
136.0
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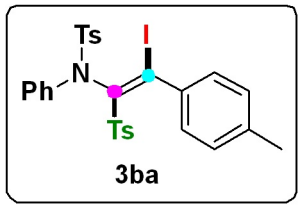
130.3
129.2
129.1
128.8
128.6
128.6
128.3
127.9
124.2

77.3
77.0
76.7

21.6
21.5
21.3

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



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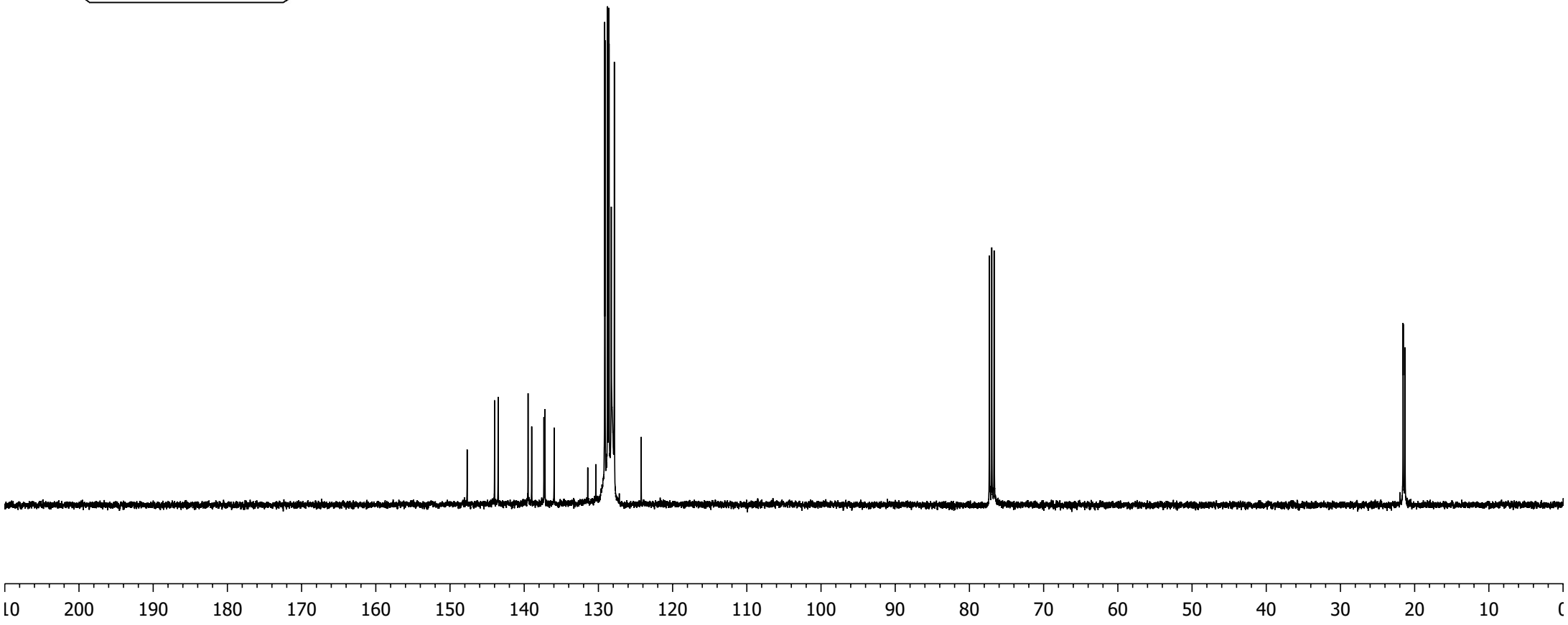

U0315-1
LJ3-10

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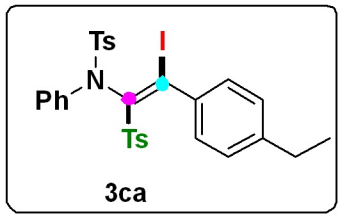
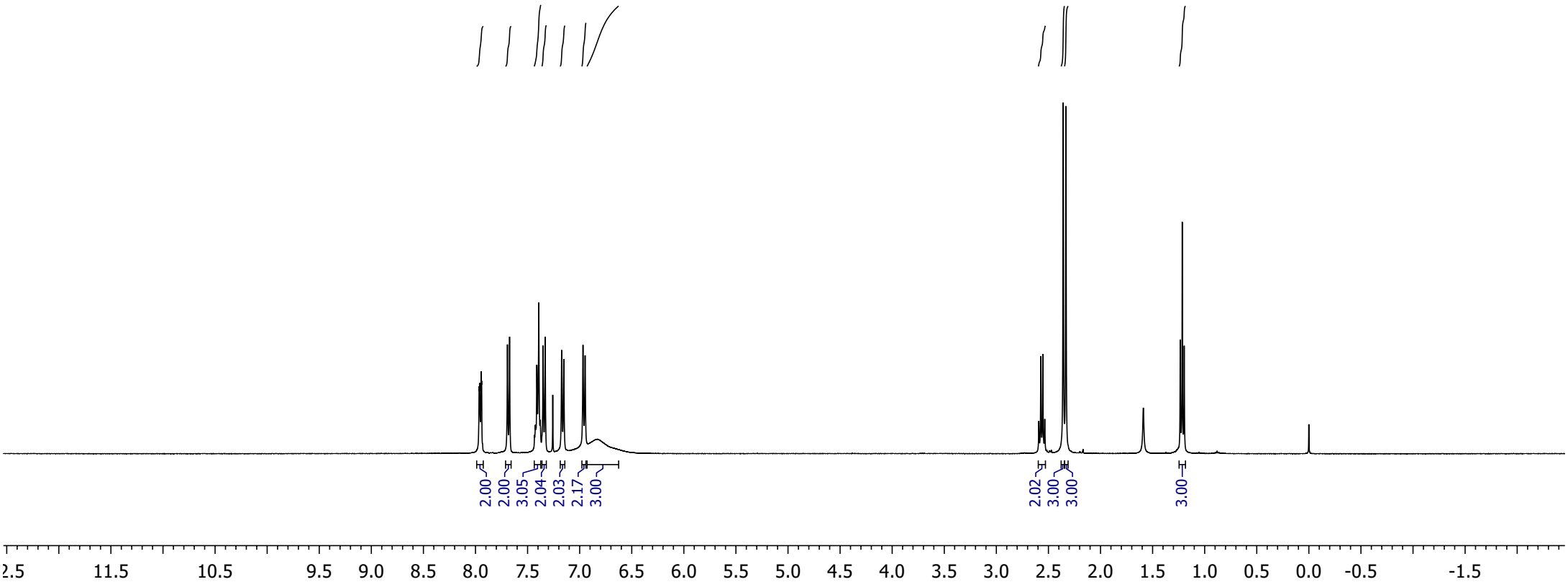
2.59 2.57 2.55 2.54 2.36 2.33 1.59 1.23 1.22 1.20 0.00

Solvent $CDCl_3$
Spectrometer Frequency 400.28
Nucleus ^1H



3ca



U0315-2
LJ3-10

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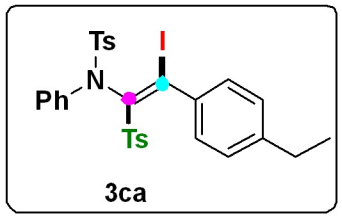
139.0
137.4
136.0
129.2
129.1
128.9
128.6
128.6
128.3
127.8
126.9
124.3
77.3
77.0
76.7
28.7
21.6
21.5
15.6

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



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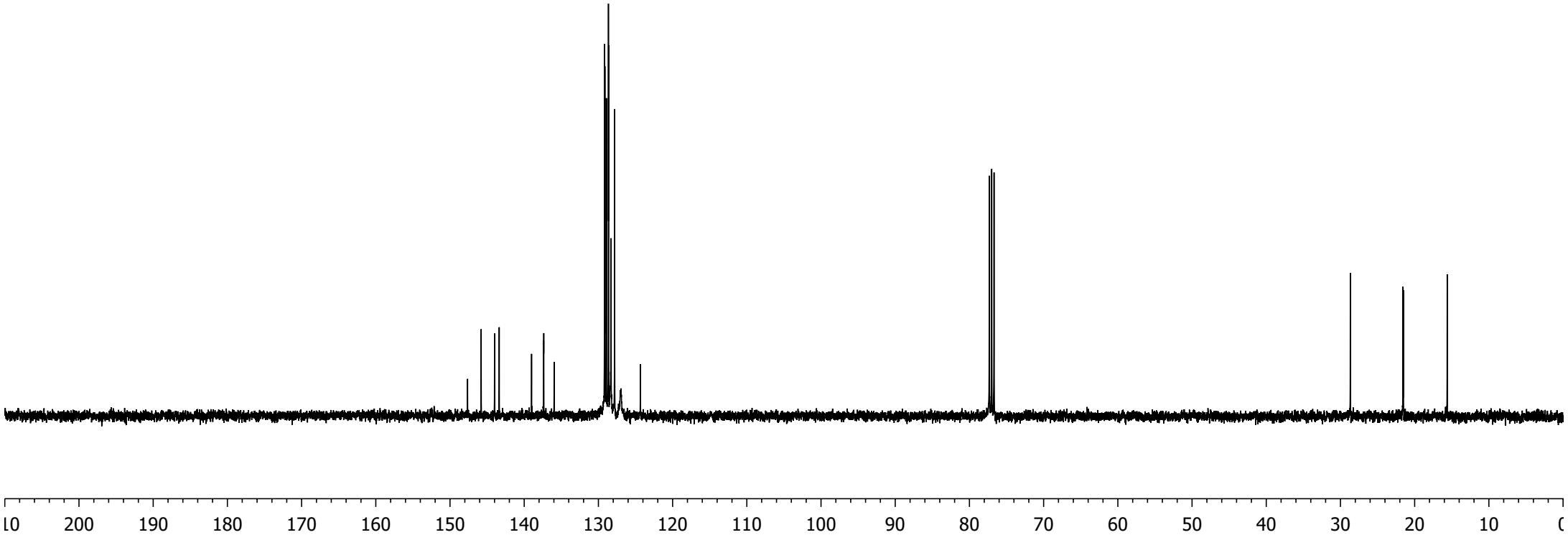
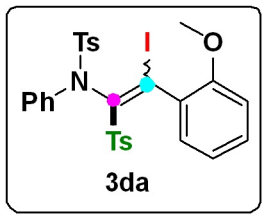


M0425-4
LJ3-15

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H

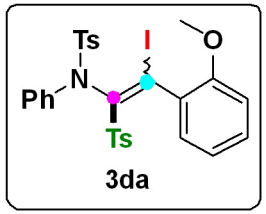


3da

M0425-5
LJ3-15

155.3
154.9
131.6
129.8
129.8
129.3
129.2
129.1
128.8
128.8
128.7
128.6
128.5
128.2
128.0
127.9
125.9
119.3
110.4
109.8
77.3
77.0
76.7
54.9
54.5
21.6
21.6
21.5

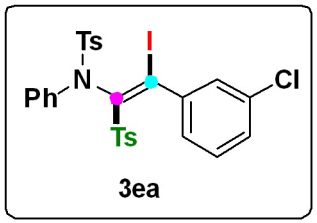
Solvent $CDCl_3$
Spectrometer Frequency 100.69
Nucleus ^{13}C



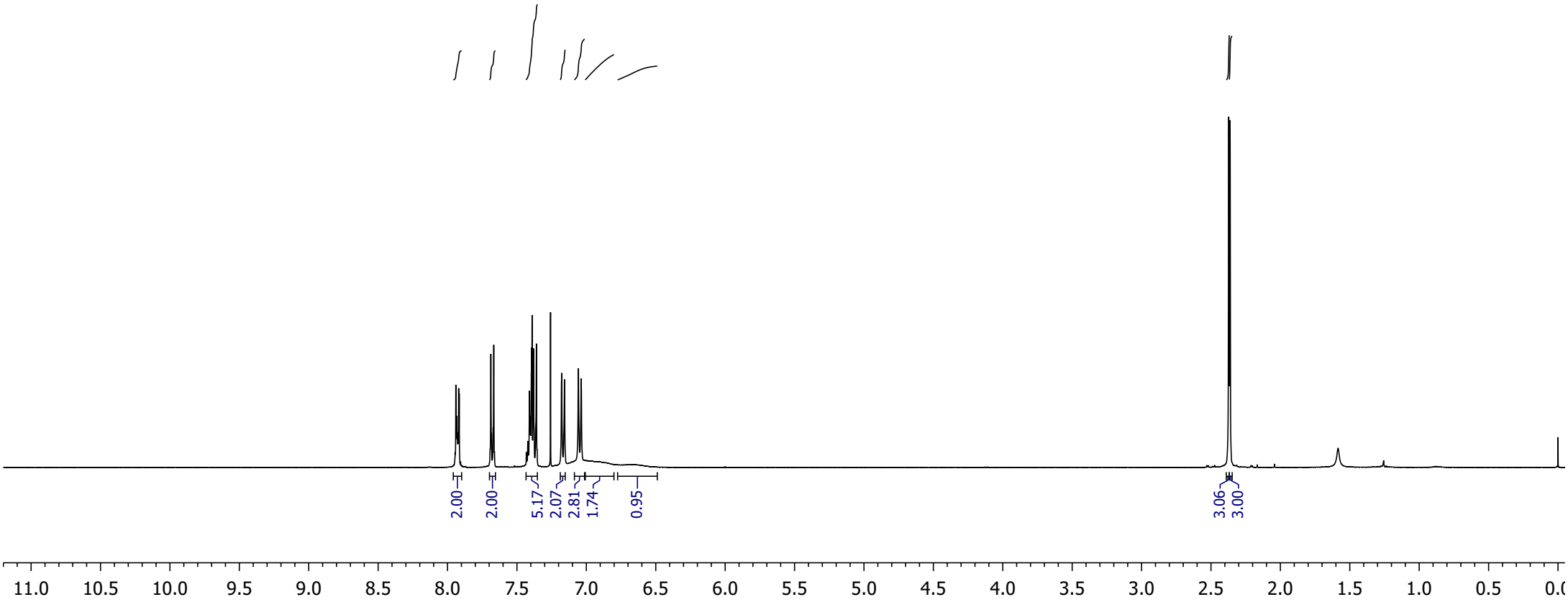
3da

M0425-7
LJ3-19(3-Cl)

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H



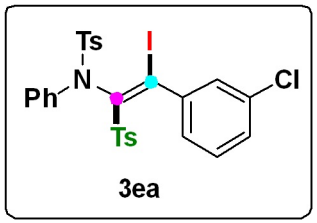
3ea



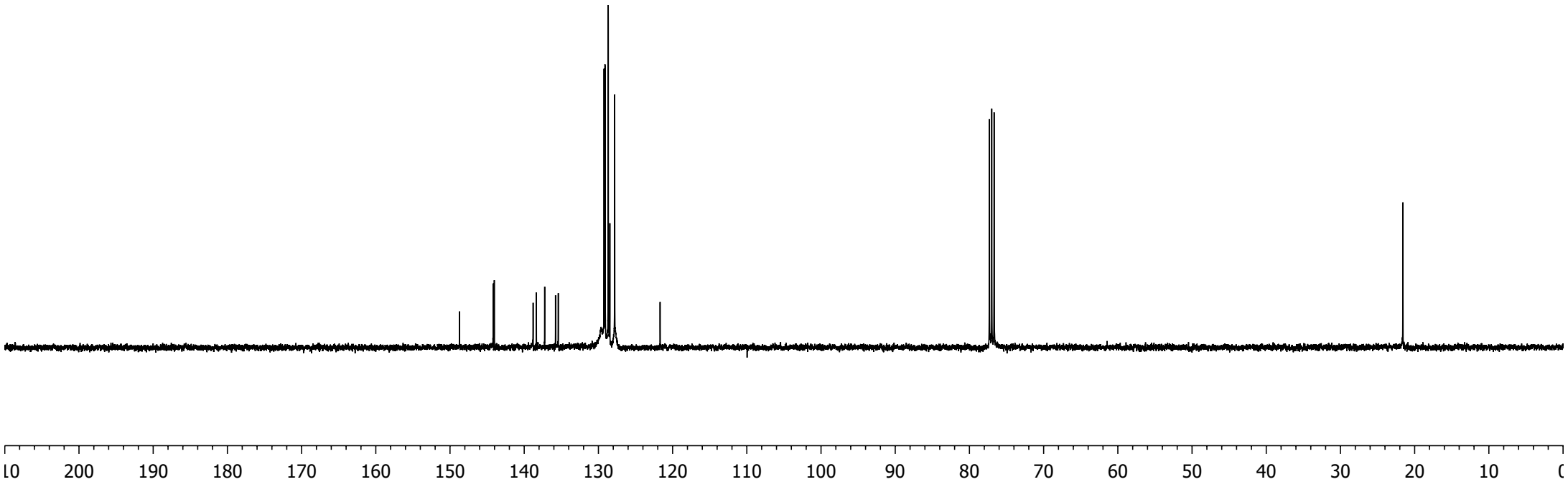
M0425-8
LJ3-19(3-Cl)

148.7
144.2
144.0
138.4
137.2
135.8
135.4
129.1
129.1
128.7
121.7
77.3
77.0
76.7
21.6

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

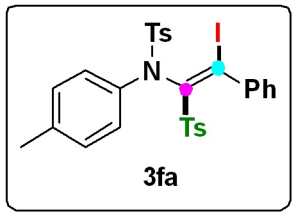


3ea

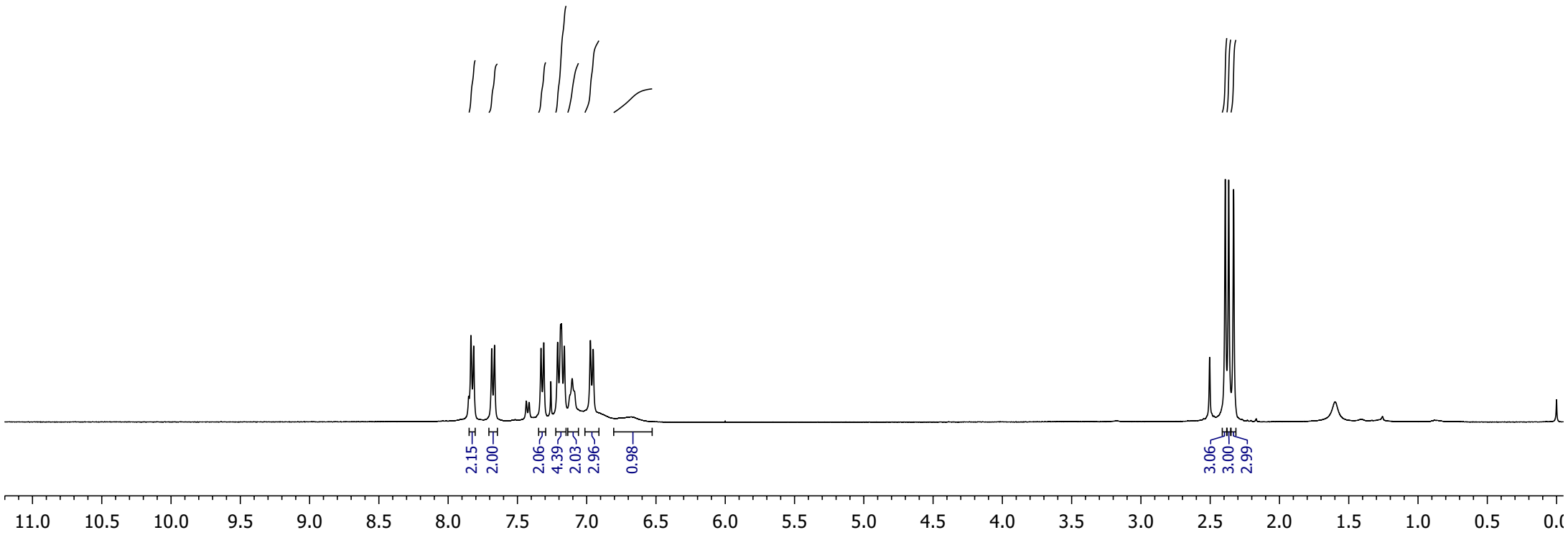


M0027-13
LJ3-5

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H



3fa



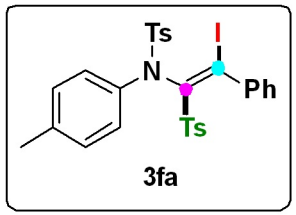
M0027-14
LJ3-5

148.1
143.9
143.5
140.0
138.5
137.4
136.3
136.0
131.5
130.4
129.8
129.1
129.0
128.8
128.6
127.8
123.4

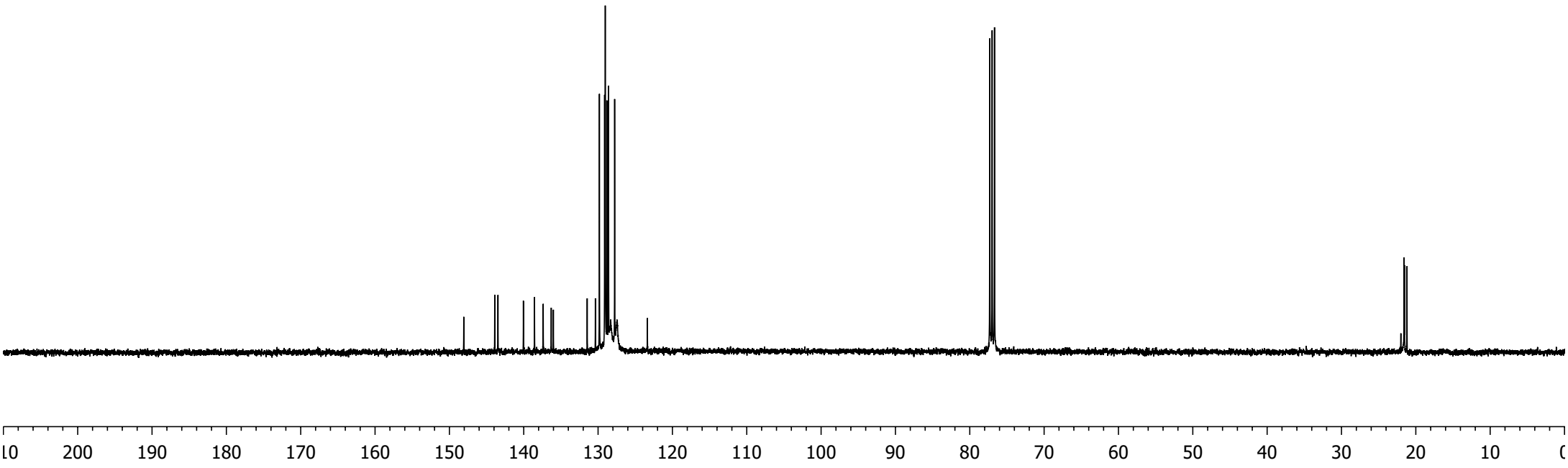
77.3
77.0
76.7

21.6
21.5
21.2

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

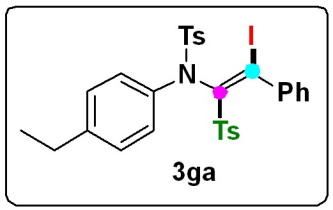


3fa



M0036-7
LJ3-6

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H

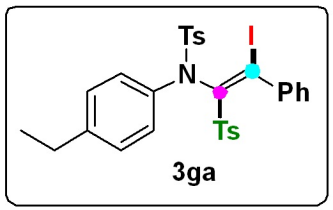


3ga

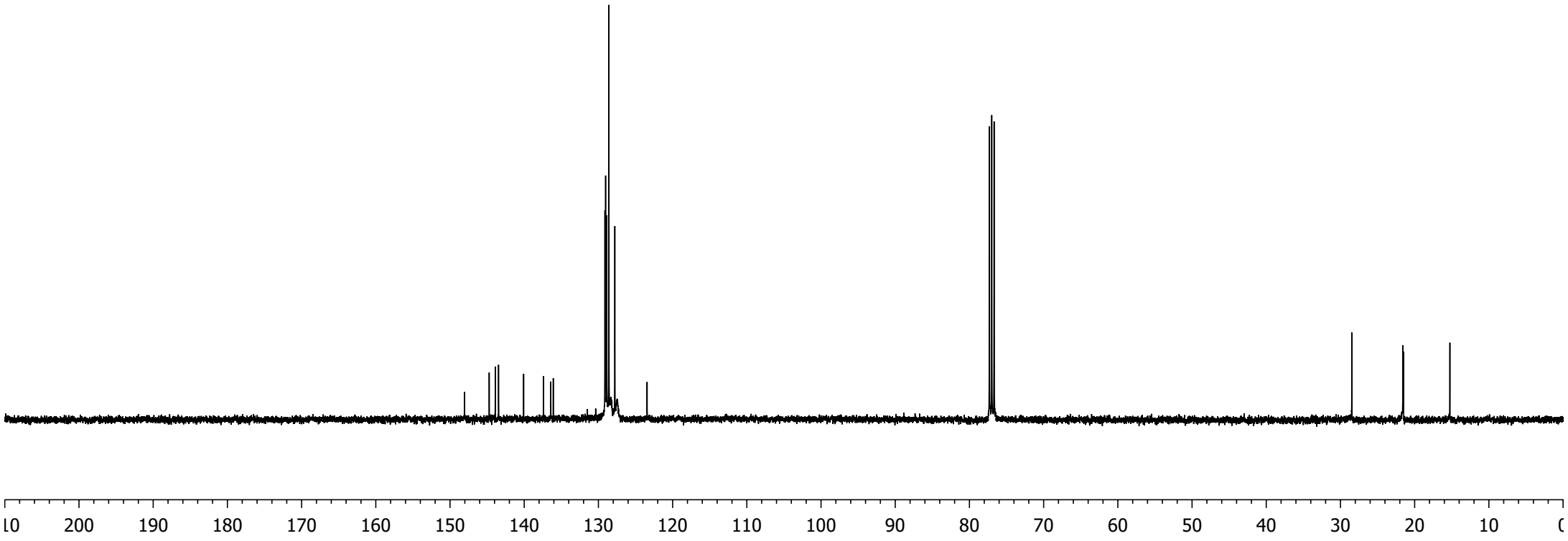
M0036-8
LJ3-6

148.0
144.7
143.9
143.5
140.1
137.4
136.4
136.1
129.1
129.0
128.9
128.6
127.8
123.4
77.3
77.0
76.7
28.5
21.6
21.5
15.3

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

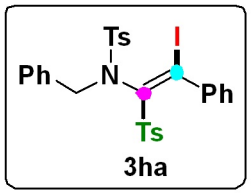


3ga



M0150-13
LJ3-1

Solvent	$CDCl_3$
Spectrometer Frequency	400.40
Nucleus	^1H

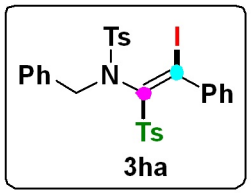


3ha

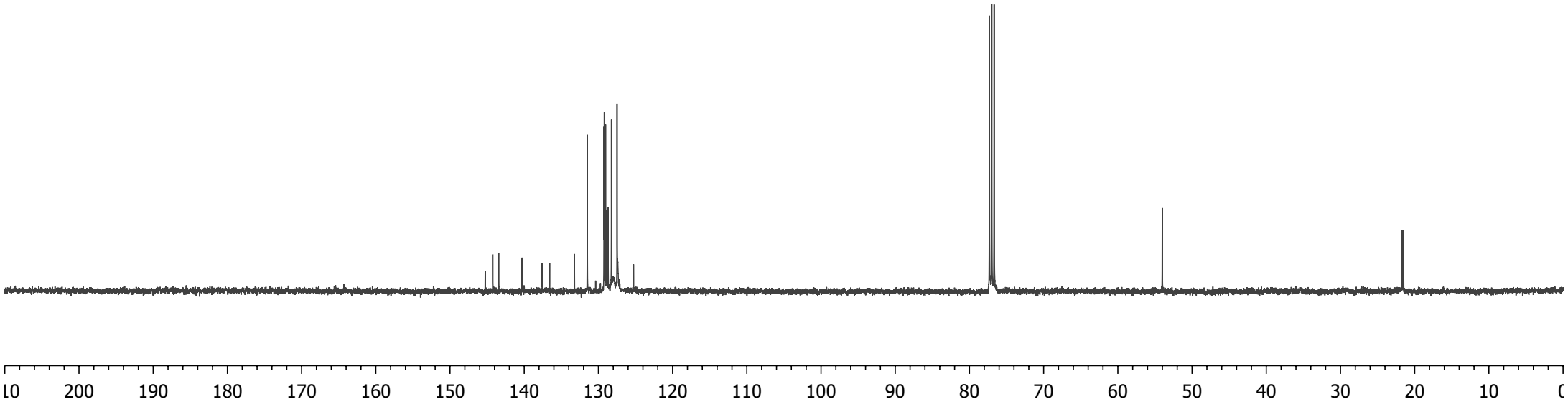
M0150-14
LJ3-1

145.3
144.2
143.4
140.3
137.6
136.6
133.2
131.5
129.2
129.2
129.0
128.9
128.7
128.2
127.5
125.3
77.3
77.0
76.7
54.0
21.7
21.5

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

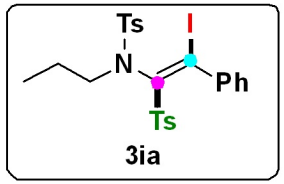


3ha



M0394-7
LJ3-14

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H

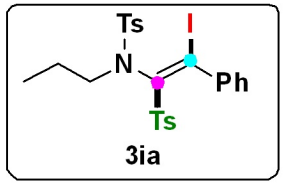


3ia

M0394-8
LJ3-14

148.1
146.5
144.0
143.5
140.2
137.3
136.5
131.4
130.3
129.1
129.0
129.0
128.2
127.9
127.6
123.8
77.3
77.0
76.7
52.3
22.0
21.7
21.6
21.5
11.5

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



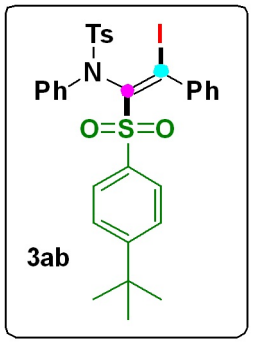
3ia

M0322-1
LJ3-11

7.98 7.97 7.96 7.96 7.95 7.69 7.67 7.43 7.43 7.42 7.41 7.41 7.40 7.38 7.26 7.20 7.18 7.17 7.15 7.09 6.83 6.65

2.36 1.30 0.00

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H



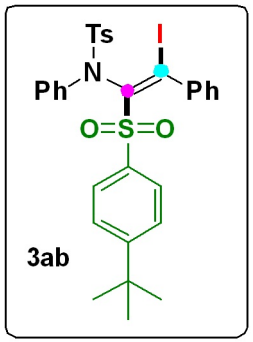
1.99 2.06 5.08 4.71 1.93 1.00 0.97 3.00 9.08

11.0 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

M0322-2
LJ3-11

156.4
148.0
144.0
140.0
138.9
137.1
135.9
129.2
129.1
129.0
128.7
128.6
128.4
127.6
125.5
123.8
77.3
77.0
76.7
35.0
31.0
21.6

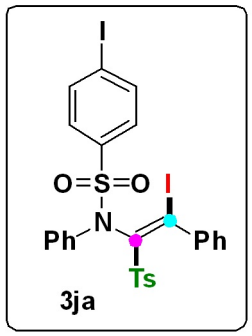
Solvent $CDCl_3$
Spectrometer Frequency 100.69
Nucleus ^{13}C



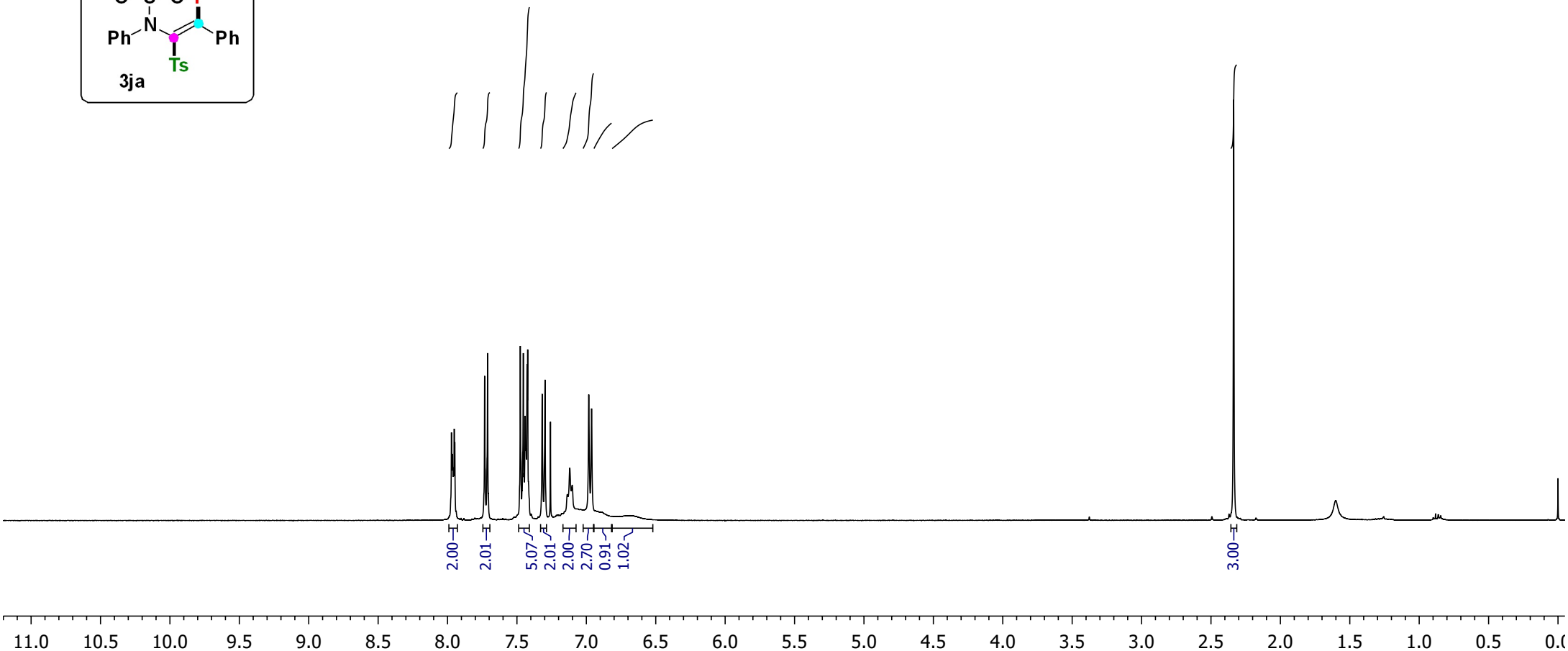
M0438-4
LJ3-24

7.97 7.95 7.73 7.71 7.48 7.45 7.45 7.44 7.44 7.43 7.43 7.42 7.32 7.30 7.26 7.14 7.12 7.10 6.98 6.96 6.68 2.34 0.00

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H



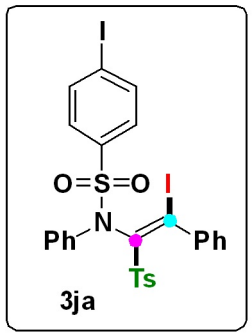
3ja



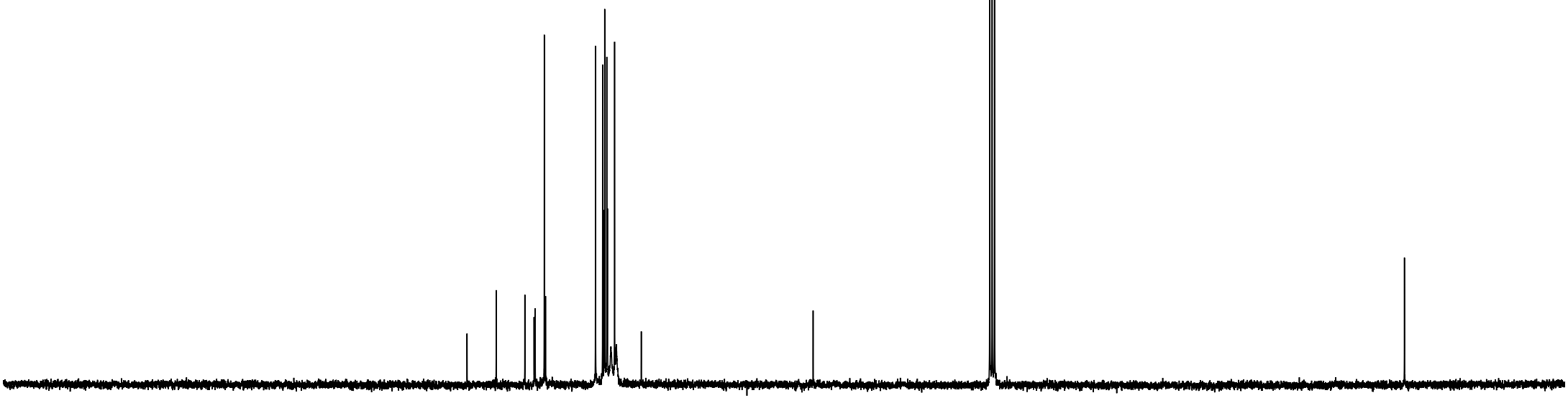
M0438-5
LJ3-24

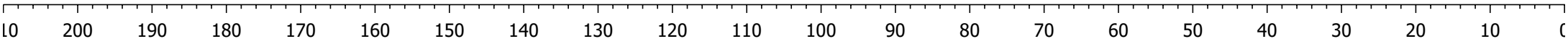
147.6 143.7 139.8 138.6 138.5 137.2 137.1 130.3 129.4 129.2 129.1 128.8 128.7 128.3 127.8 127.5 124.2 101.1 77.3 77.0 76.7 21.5

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



3ja

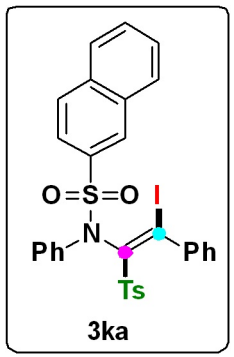




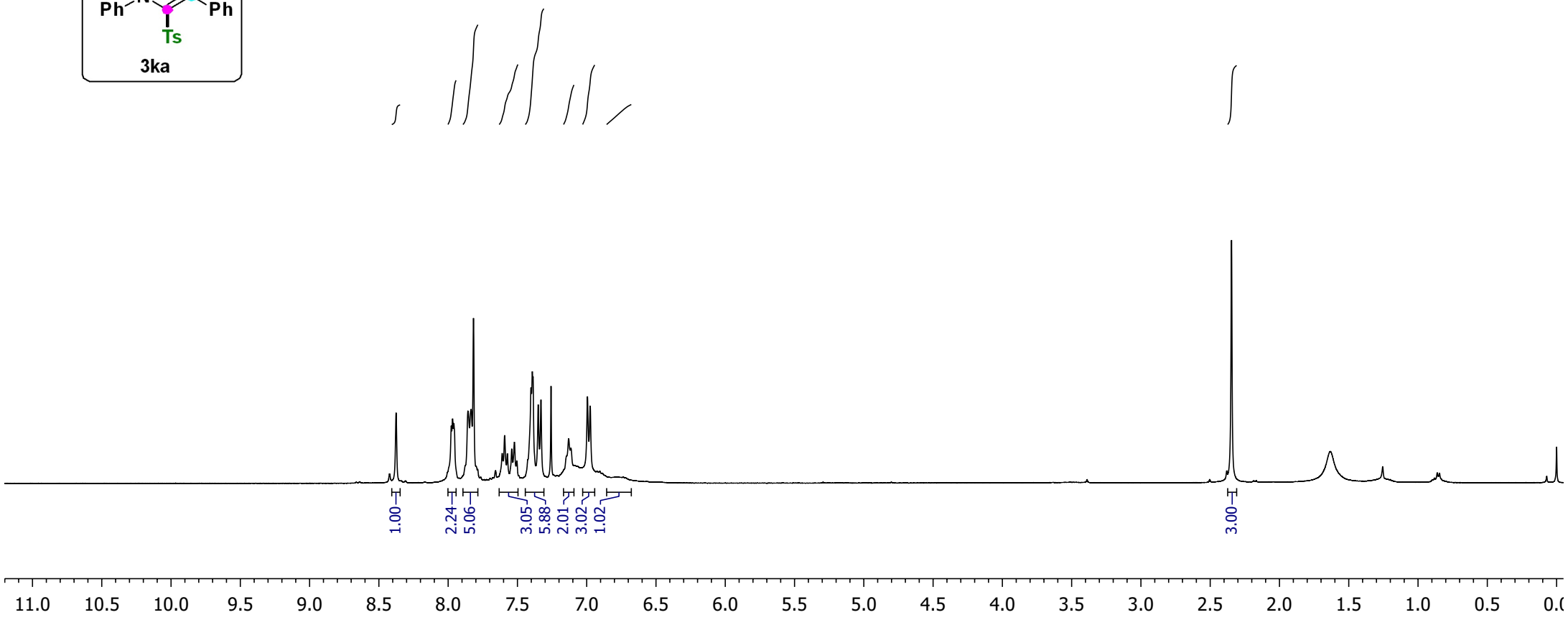
M0438-7
LJ3-25

8.38 7.98 7.97 7.96 7.86 7.83 7.82 7.61 7.59 7.57 7.54 7.52 7.50 7.40 7.39 7.39 7.35 7.33 7.26 7.13 7.11 6.99 6.97 2.35 0.00

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H



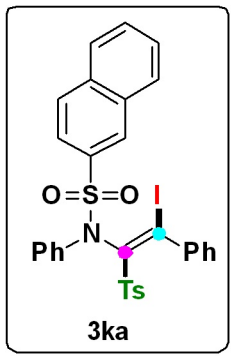
3ka



M0438-8
LJ3-25

148.0
143.6
140.1
139.0
137.3
136.0
135.1
131.6
130.8
129.5
129.3
129.3
129.1
129.1
128.8
128.8
128.5
127.9
127.9
127.8
127.7
127.1
124.3
123.7
77.3
77.0
76.7
21.5

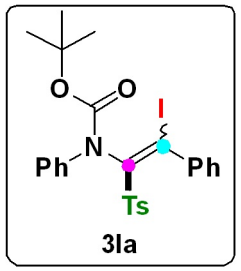
Solvent $CDCl_3$
Spectrometer Frequency 100.69
Nucleus ^{13}C



3ka

M0438-1
LJ3-23(N-Boc)

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H

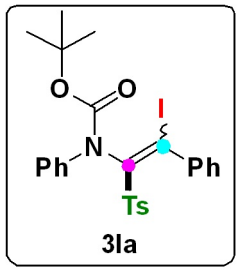


3la

M0438-2
LJ3-23(N-Boc)

150.5 145.7 144.7 130.1 129.3 129.1 128.9 128.8 128.6 128.4 128.2 127.9 127.8 126.9 114.3 83.5 82.9 77.3 77.0 76.7 28.1 28.1 21.6 21.5

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



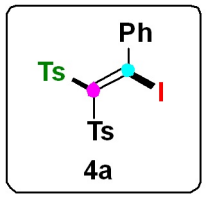
3la

M0150-16
LJ3-2

8.21 8.19 7.51 7.49 7.41 7.38 7.27 7.26 7.26 7.25 7.25 7.24 7.18 7.16 7.06 7.05 7.05 7.04 7.04 7.03

2.49 2.39

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H



4a

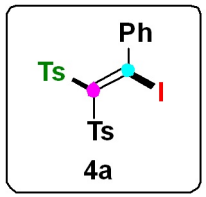
1.93 1.96 2.02 3.16 2.00 1.95 3.00 3.00

11.0 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

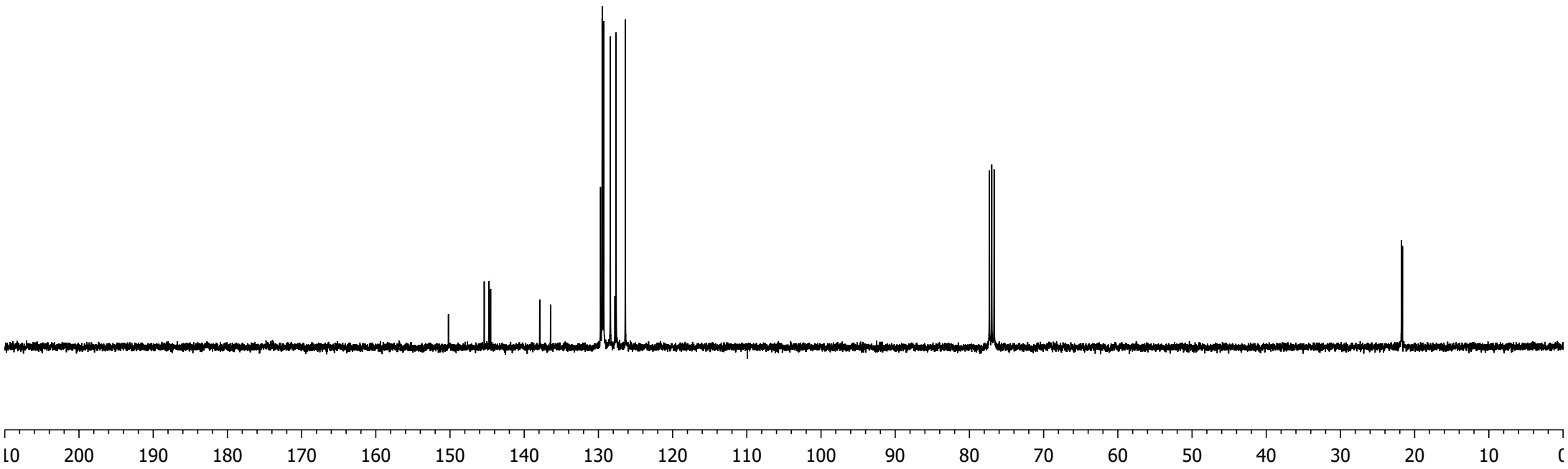
M0150-17
LJ3-2

150.2
145.4
144.7
144.5
137.9
136.5
129.7
129.5
129.5
129.3
128.4
127.8
127.6
126.4
77.3
77.0
76.7
21.8
21.6

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



4a

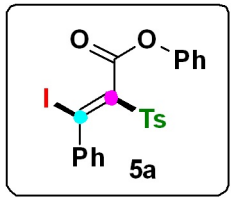


M0158-9
LJ3-7

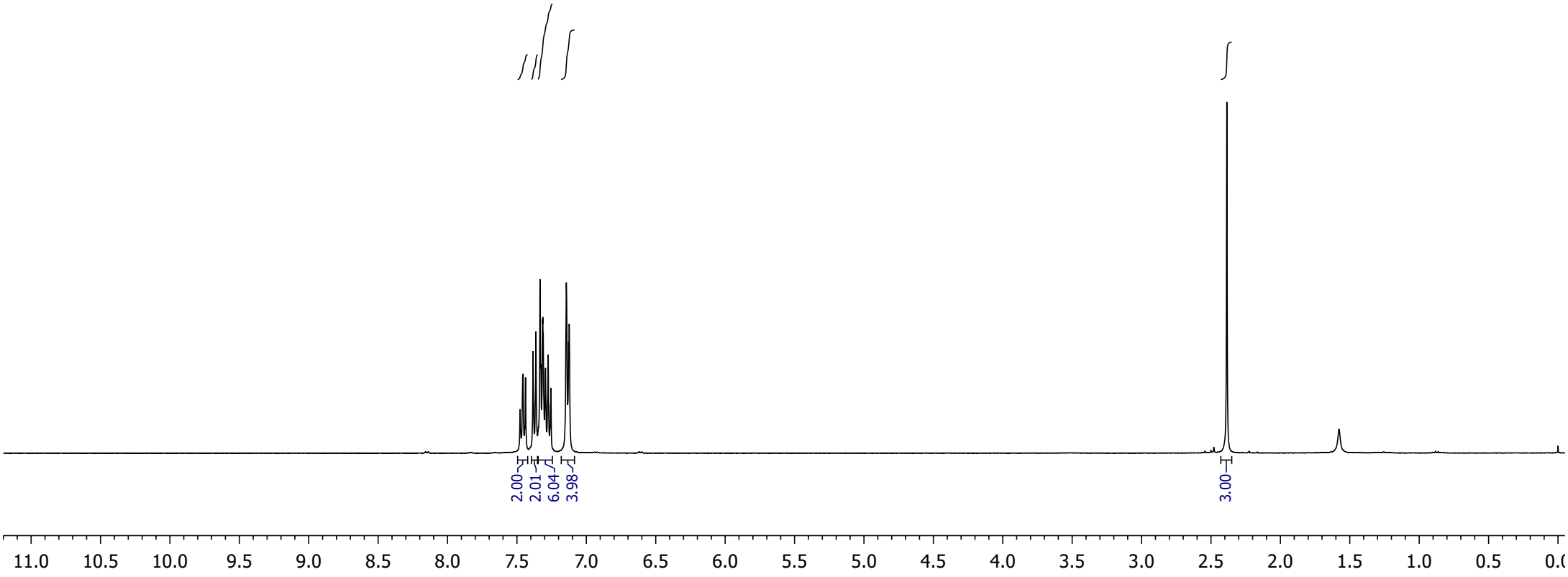
7.48 7.46 7.46 7.44 7.44 7.38 7.36 7.33 7.33 7.32 7.31 7.31 7.29 7.28 7.26 7.26 7.14 7.12

2.39

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H



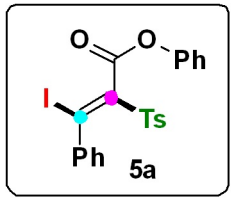
5a



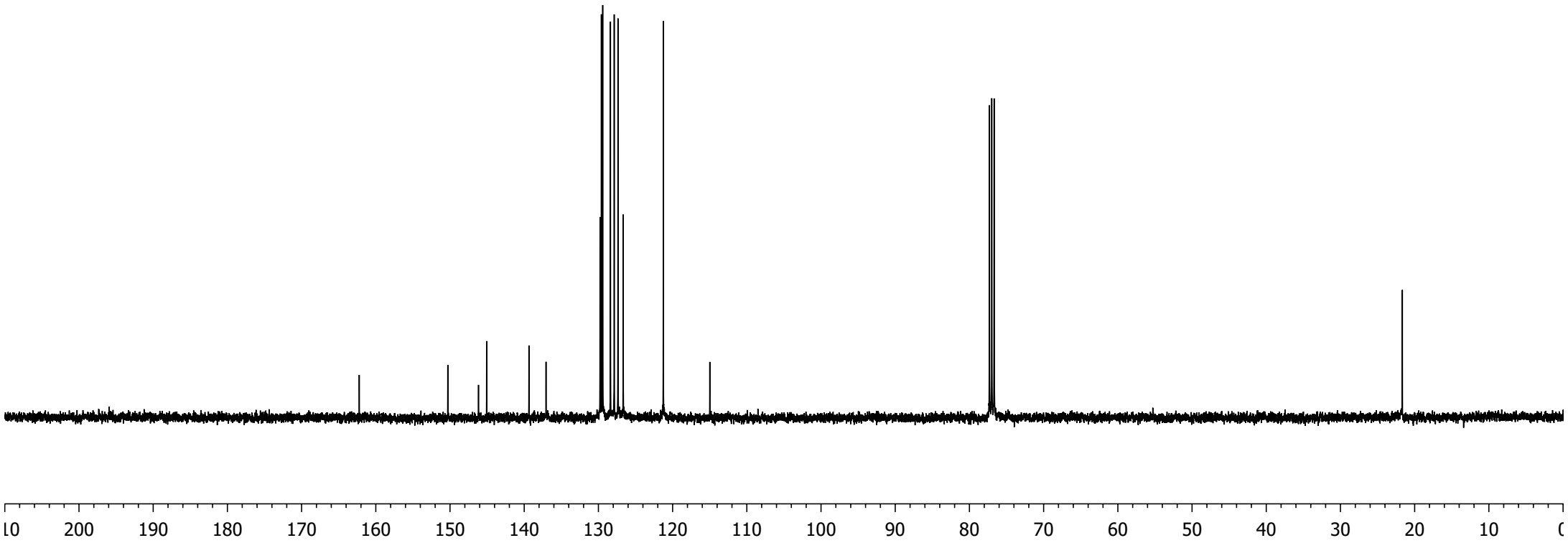
M0158-10
LJ3-7

162.2
150.3
146.2
145.1
139.4
137.1
129.6
129.4
128.4
127.9
127.4
121.3
115.0
77.3
77.0
76.7
21.7

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



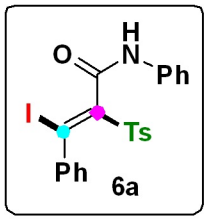
5a



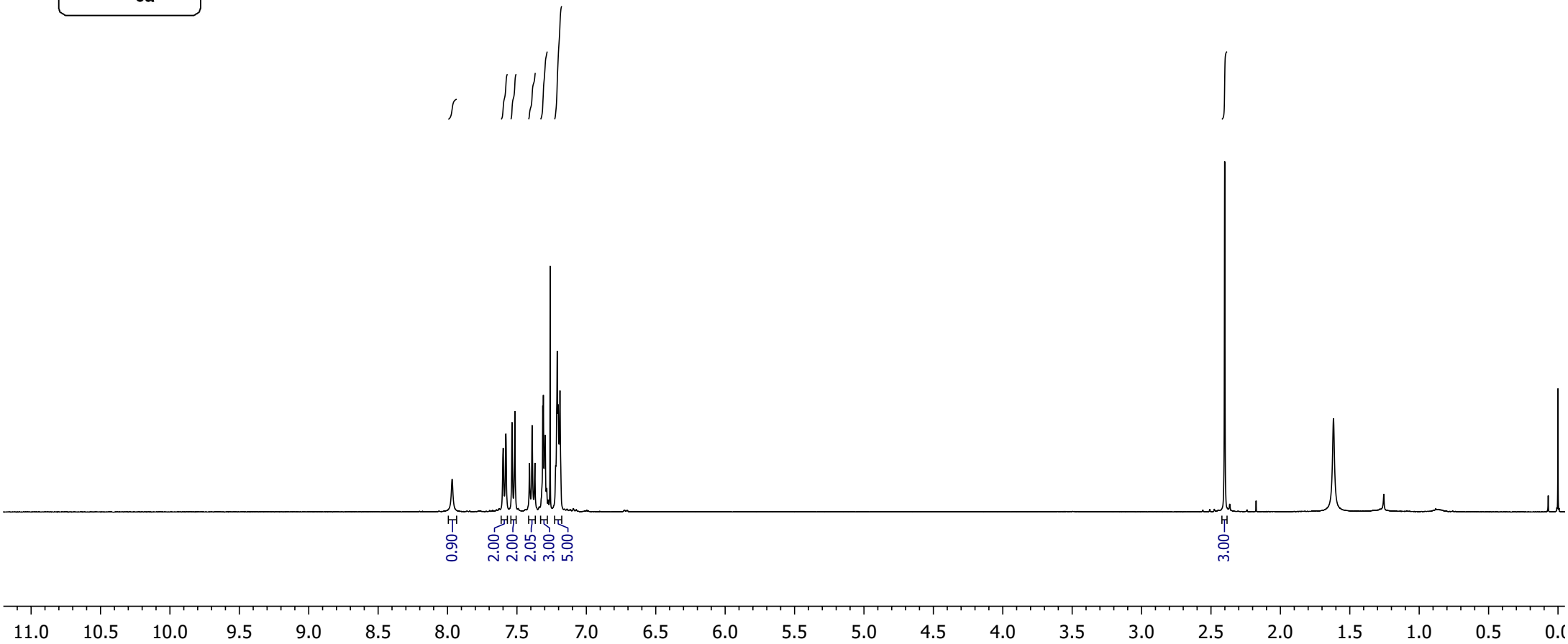
U0131-4
LJ3-32

7.97 7.60 7.58 7.54 7.52 7.41 7.39 7.37 7.31 7.31 7.30 7.26 7.21 7.19 2.40 1.62 0.00

Solvent $CDCl_3$
Spectrometer Frequency 400.28
Nucleus ^1H



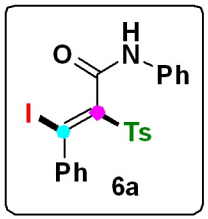
6a



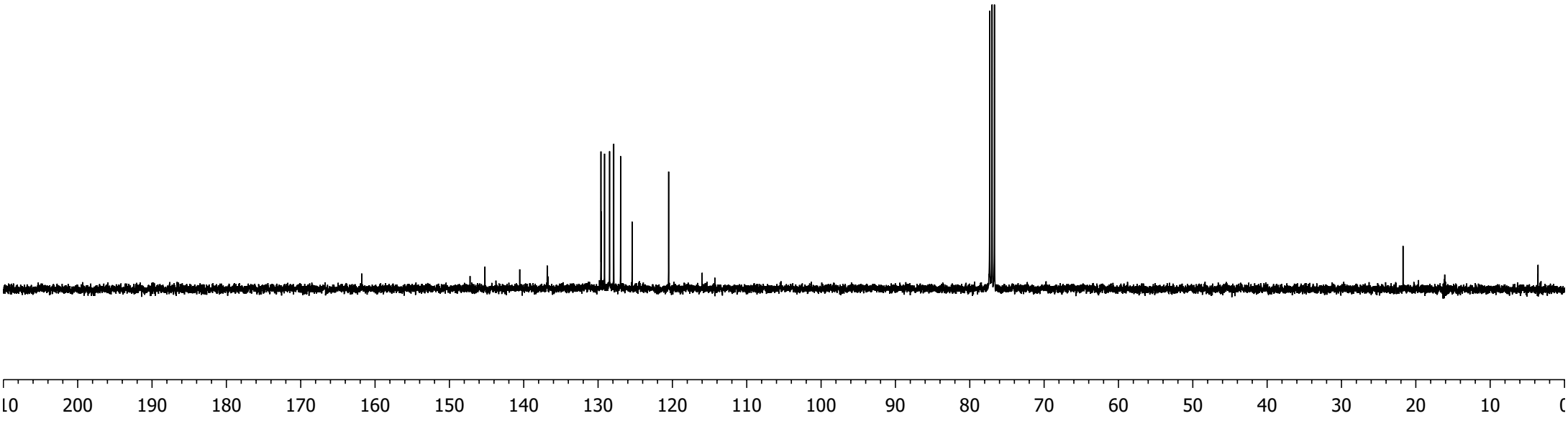
U0131-5
LJ3-32

161.8
147.2
145.2
140.5
136.8
129.6
129.6
129.1
128.5
127.9
127.0
125.4
120.5
116.0
77.3
77.0
76.7
21.7

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



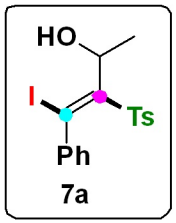
6a



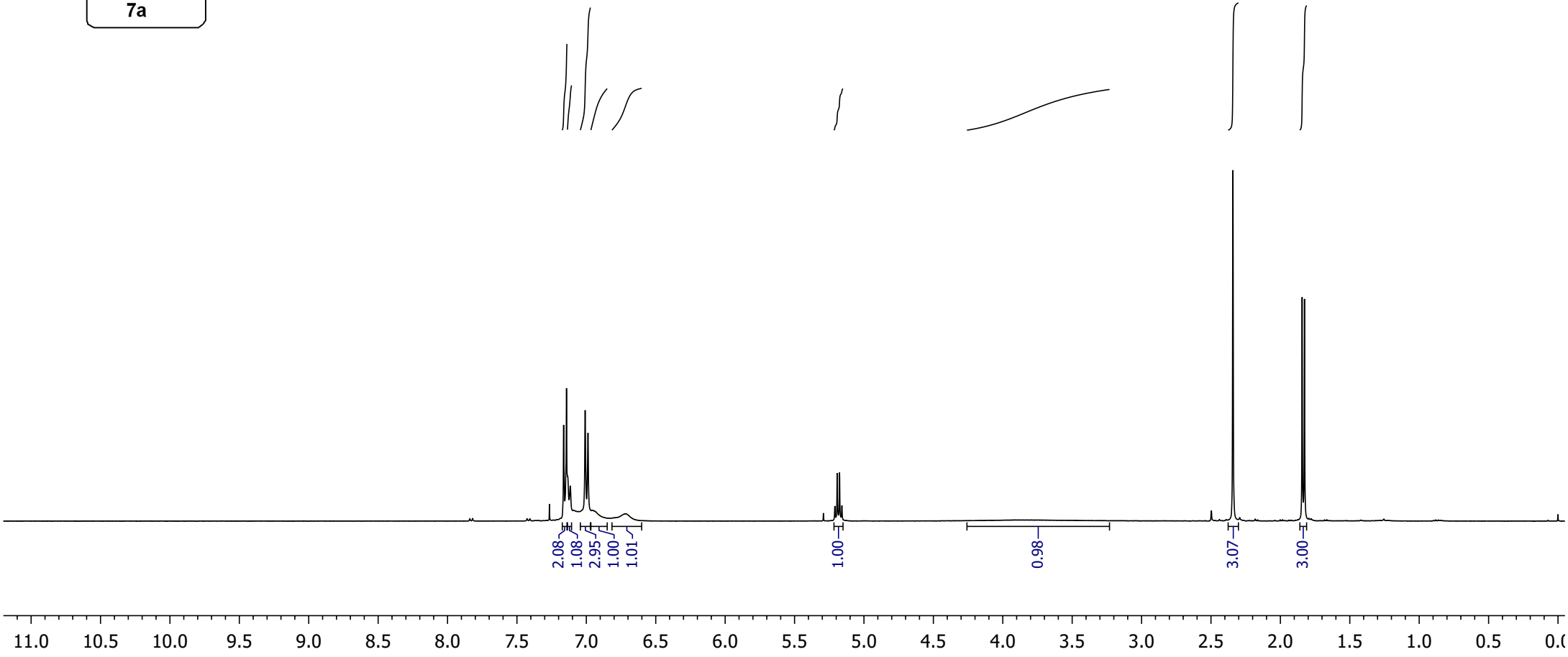
U0111-10
LJ3-30

7.27 7.16 7.14 7.13 7.12 7.01 6.99 6.72 3.84 2.34 1.84 1.83

Solvent $CDCl_3$
Spectrometer Frequency 400.28
Nucleus ^1H



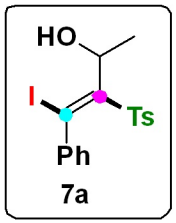
7a



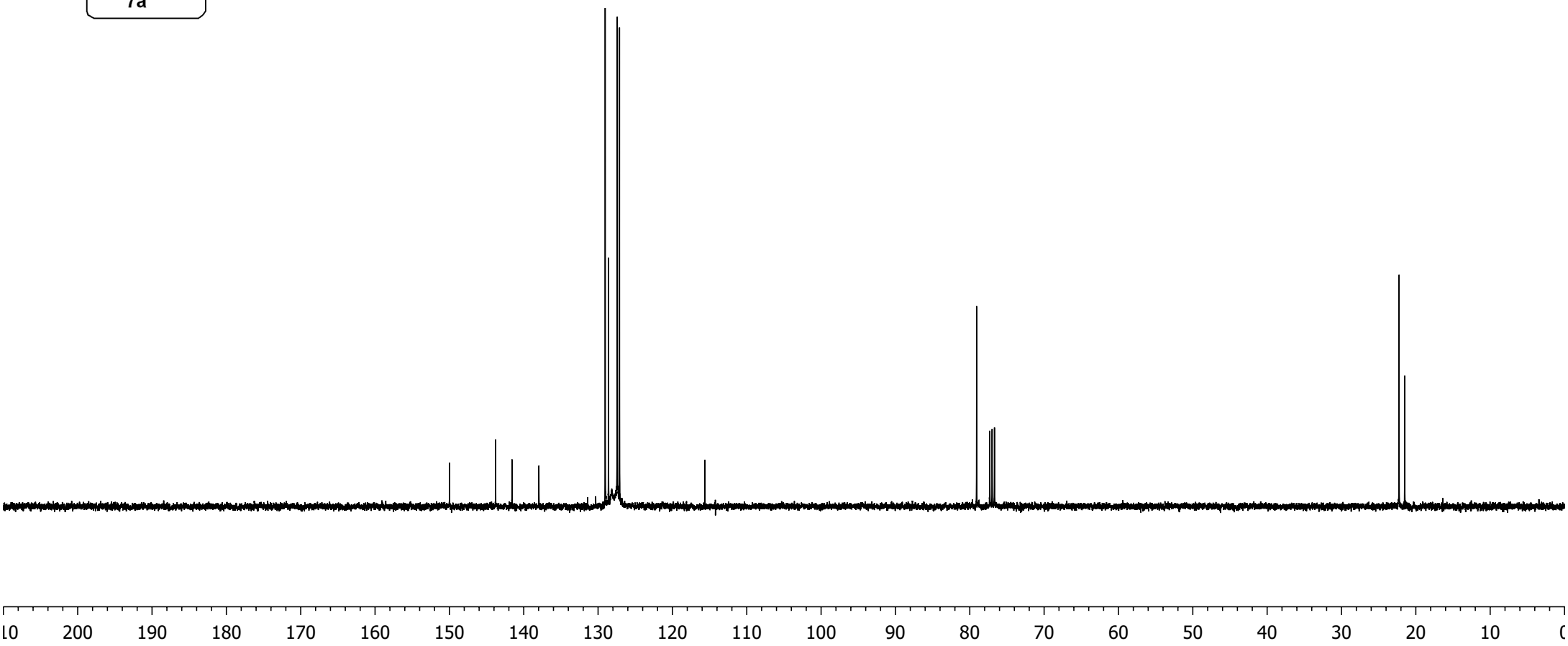
U0111-11
LJ3-30

150.0
143.8
141.6
138.0
129.0
128.6
127.4
127.1
115.6
79.1
77.3
77.0
76.7
22.3
21.5

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

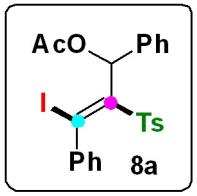


7a



U0111-4
LJ3-28

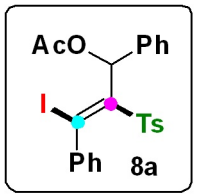
Solvent $CDCl_3$
Spectrometer Frequency 400.28
Nucleus ^1H

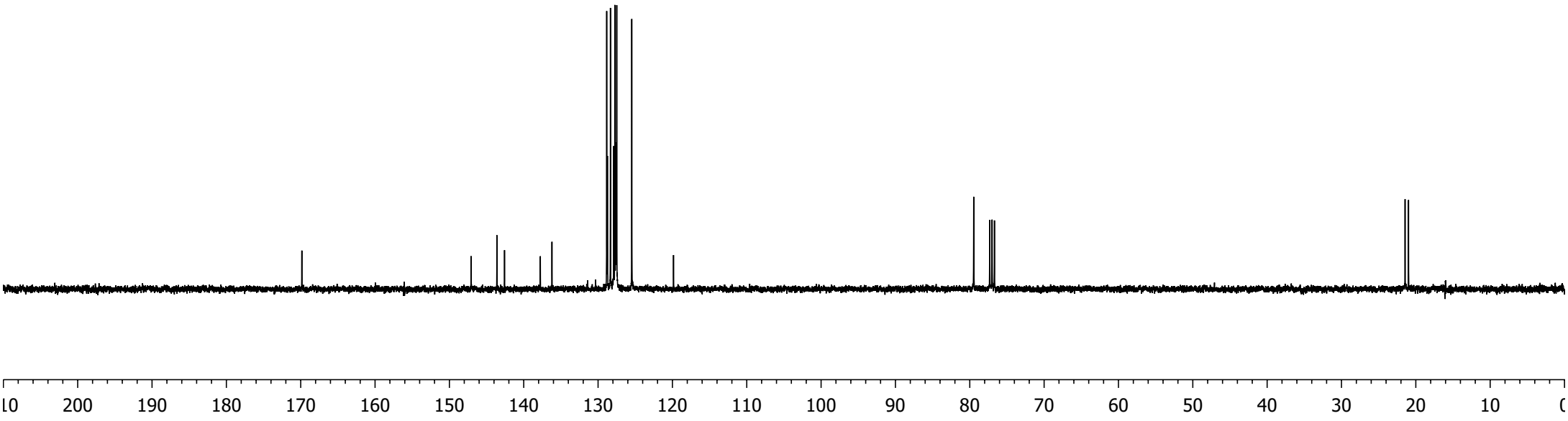


U0111-5
LJ3-28

169.8
147.1
143.6
142.6
137.8
136.2
128.8
128.3
127.7
127.5
125.5
119.9
79.5
77.3
77.0
76.7
21.4
21.0

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

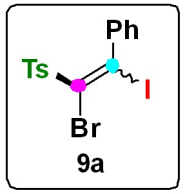




U0151-7
LJ 3-35

8.03 8.01 7.57 7.55 7.41 7.39 7.37 7.32 7.31 7.30 7.30 7.25 7.23 7.22 7.21 7.20 7.19 7.16 7.15 7.14 2.48 2.42

Solvent $CDCl_3$
Spectrometer Frequency 400.28
Nucleus ^1H

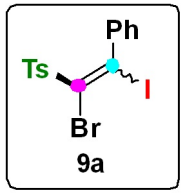


9a

U0151-8
LJ 3-35

156.0
156.0
145.1
142.3
129.8
129.6
129.3
129.1
128.7
127.8
127.0
126.6
119.9
77.3
77.0
76.7
29.6
21.7

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



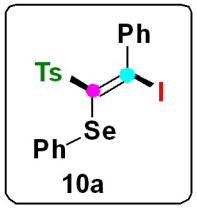
9a

U0128-1
LJ3-34

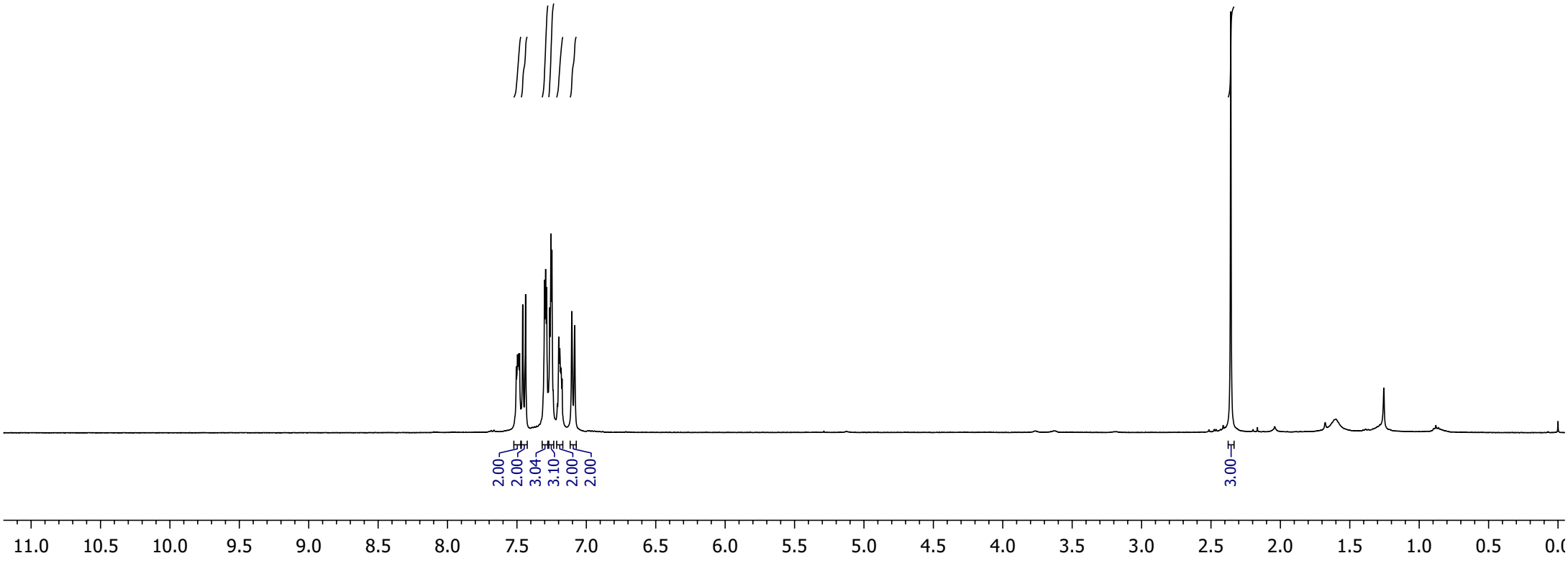
7.50 7.50 7.49 7.49 7.48 7.46 7.44 7.30 7.29 7.29 7.26 7.26 7.25 7.20 7.19 7.19 7.18 7.17 7.10 7.08

2.36

Solvent $CDCl_3$
Spectrometer Frequency 400.28
Nucleus ^1H



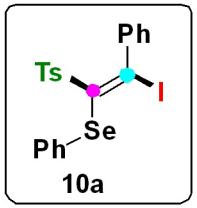
10a



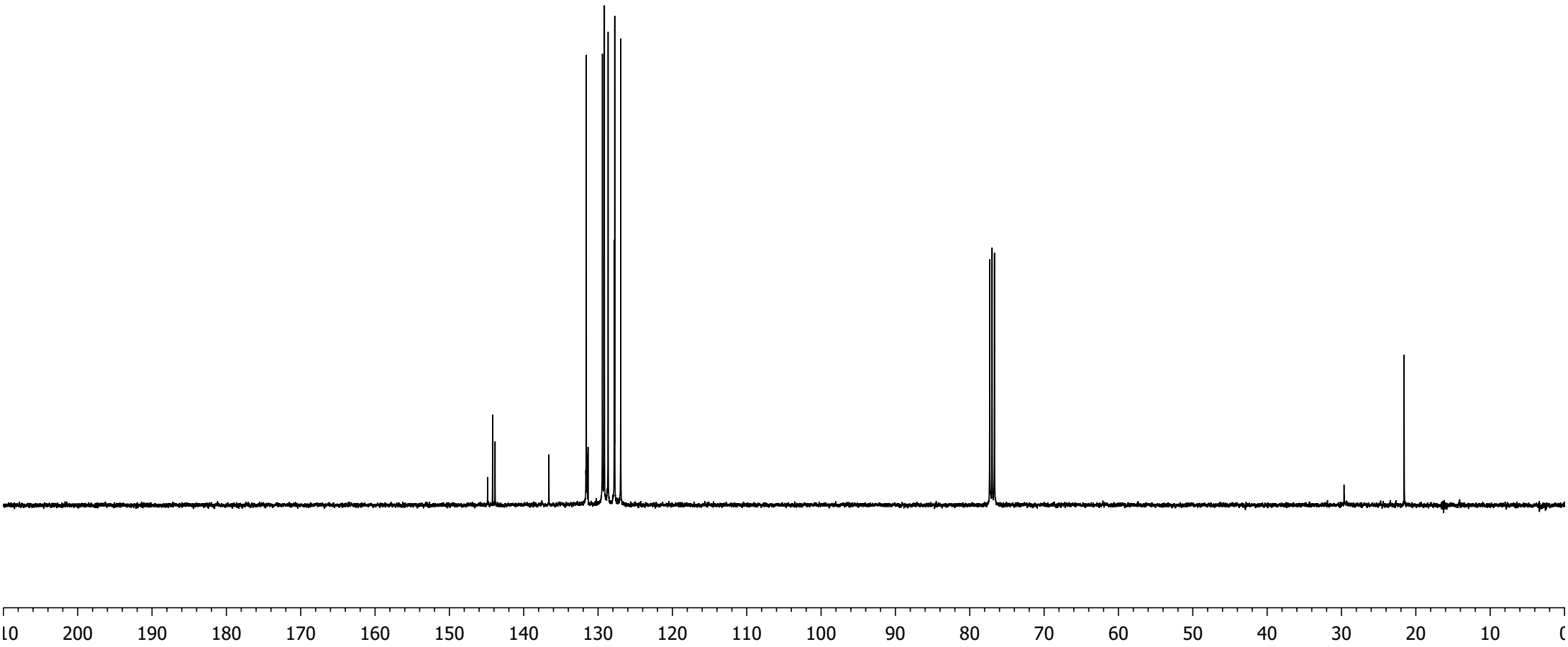
U0128-2
LJ3-34

144.9
144.2
143.9
136.6
131.6
131.5
131.3
129.4
129.2
129.1
128.6
127.8
127.8
127.0
77.3
77.0
76.7
21.6

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



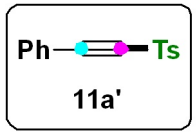
10a

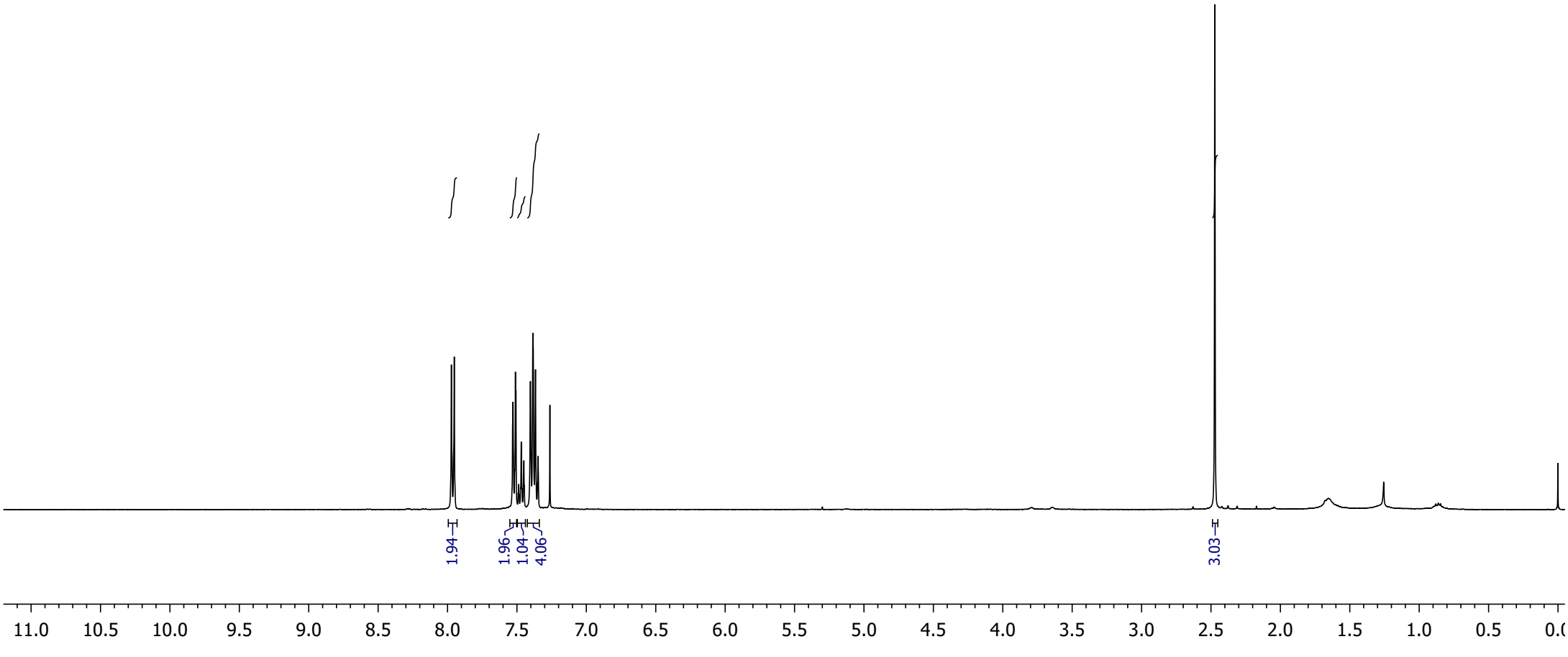


U0131-1
LJ3-36

7.97 7.95 7.53 7.51 7.51 7.49 7.47 7.47 7.45 7.45 7.40 7.39 7.38 7.37 7.35 7.26 2.47 0.00

Solvent $CDCl_3$
Spectrometer Frequency 400.28
Nucleus ^1H

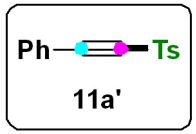


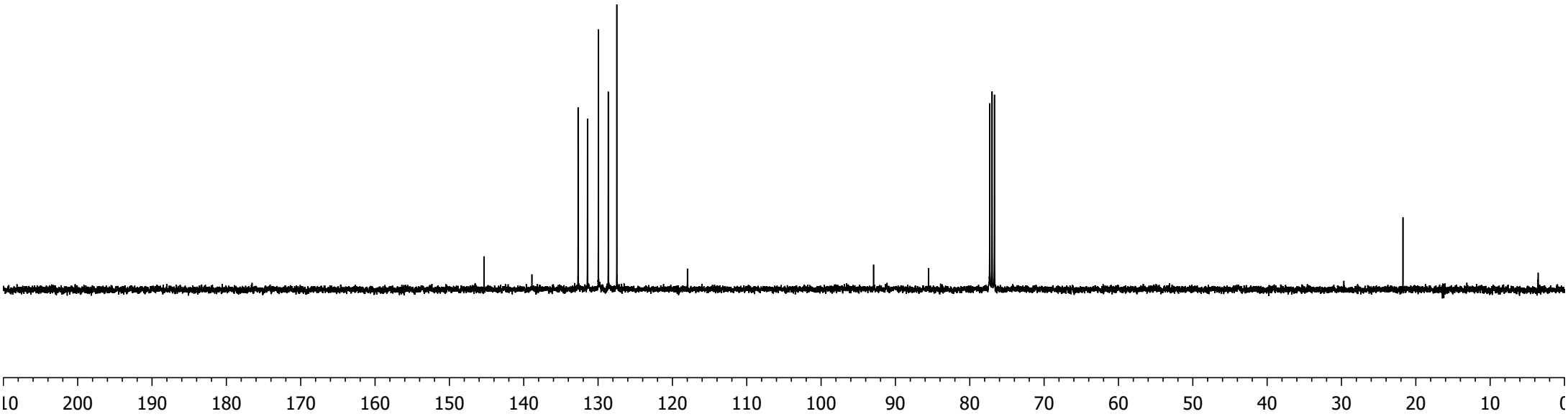


U0131-2
LJ3-36

145.3
138.9
132.7
131.4
130.0
128.6
127.5
118.0
92.9
85.6
77.3
77.0
76.7
21.7

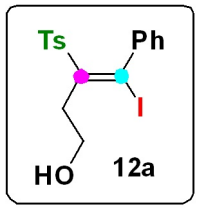
Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C





U0111-7
LJ3-29

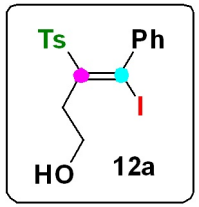
Solvent $CDCl_3$
Spectrometer Frequency 400.28
Nucleus ^1H



U0111-8
LJ3-29

146.1
143.9
142.4
137.2
129.3
128.6
127.6
127.5
117.4
77.3
77.0
76.7
60.9
42.1
21.5

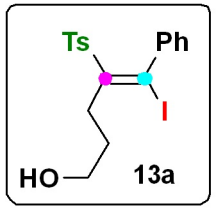
Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C



12a

M0394-1
LJ3-31

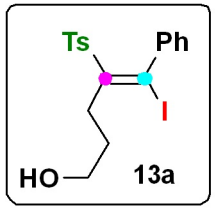
Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H



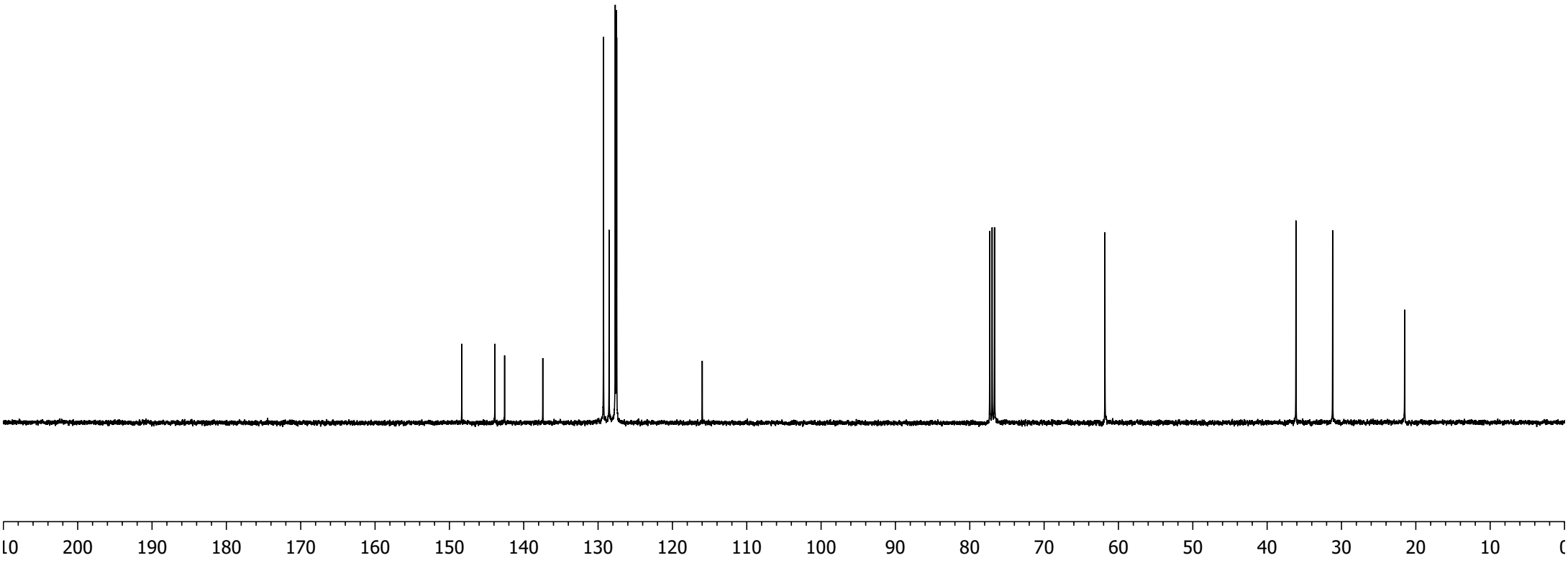
M0394-2
LJ3-31

148.3
143.9
142.6
137.4
129.3
128.5
127.7
127.5
127.5
116.0
77.3
77.0
76.7
61.8
36.1
31.2
21.5

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



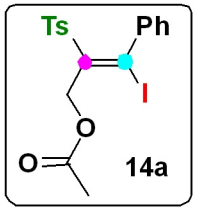
13a



V0035-9
LJ3-21

7.31 7.29 7.26 7.25 7.24 7.24 7.23 7.21 7.20 7.19 7.12 7.10 7.06 7.05 5.29 2.37 2.11

Solvent $CDCl_3$
Spectrometer Frequency 597.22
Nucleus ^1H



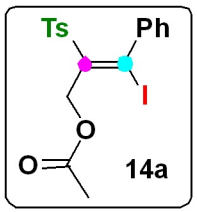
2.00 3.13 2.00 2.00 2.00 3.00 3.00

11.0 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

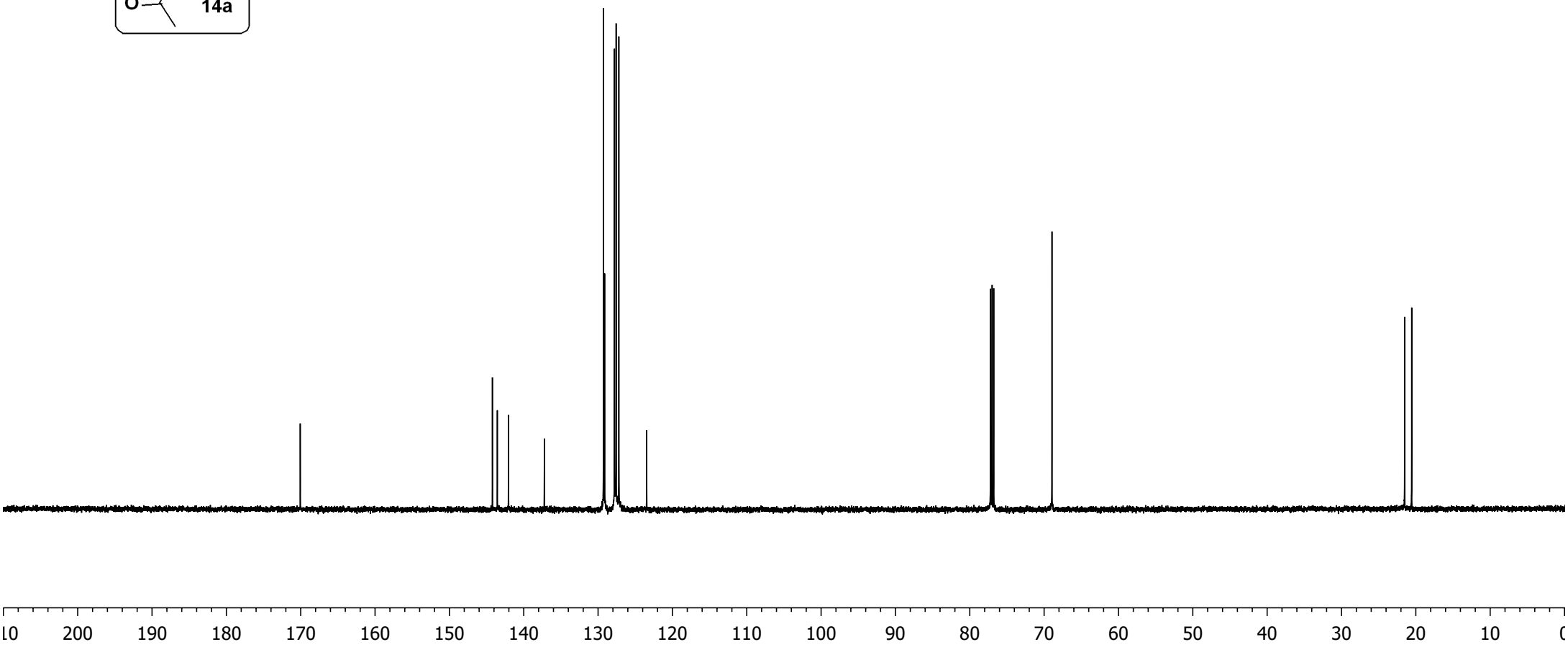
V0035-10
LJ3-21

170.1
144.2
143.6
142.0
137.2
129.3
129.1
127.8
127.6
127.2
123.5
77.2
77.0
76.8
68.9
21.5
20.6

Solvent $CDCl_3$
Spectrometer Frequency 150.19
Nucleus ^{13}C



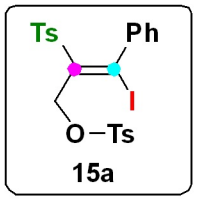
14a



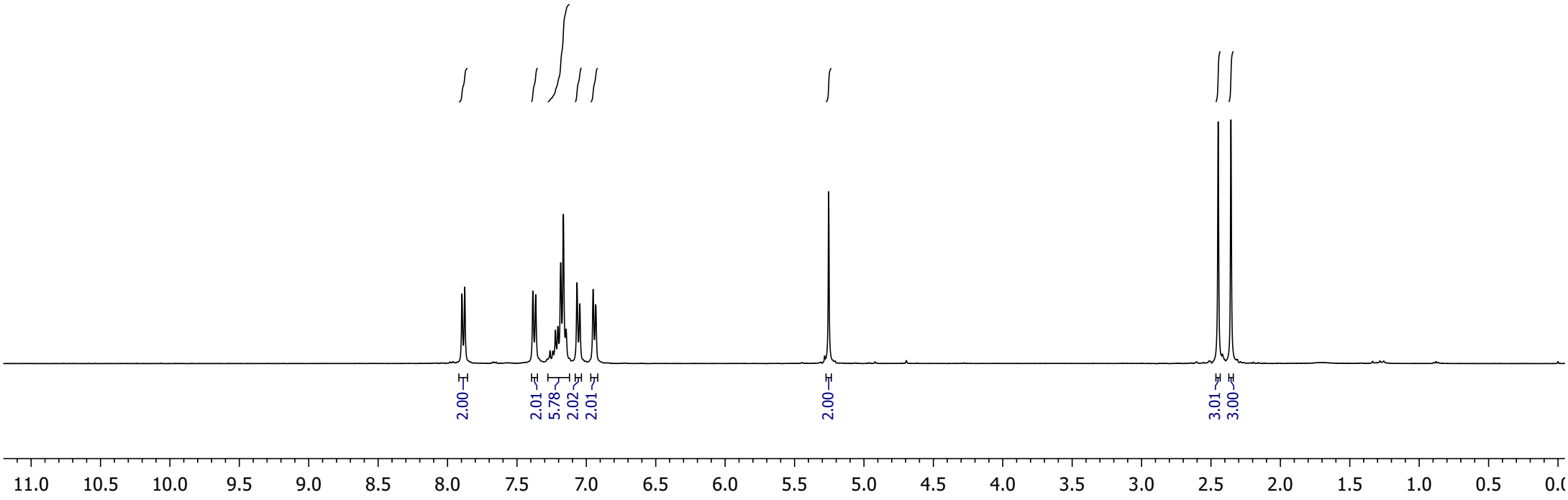
U0067-13
LJ3-22

7.90 7.88 7.39 7.37 7.26 7.24 7.22 7.21 7.18 7.17 7.15 7.07 7.05 6.95 6.93 5.25 2.45 2.36

Solvent $CDCl_3$
Spectrometer Frequency 400.28
Nucleus ^1H



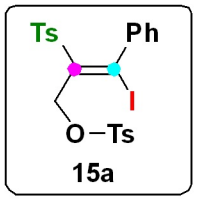
15a



U0067-14
LJ3-22

145.2
144.4
142.0
141.7
136.7
132.1
129.8
129.3
128.3
127.8
127.5
127.0
126.3
77.3
77.0
76.7
73.3
21.7
21.5

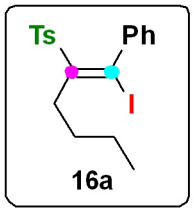
Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C



15a

U0100-1
LJ3-26

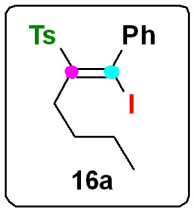
Solvent $CDCl_3$
Spectrometer Frequency 400.28
Nucleus ^1H



U0100-2
LJ3-26

149.0
143.6
142.7
137.8
129.2
128.4
127.8
127.5
115.0
77.3
77.0
76.7
39.5
30.3
22.7
21.5
13.7

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

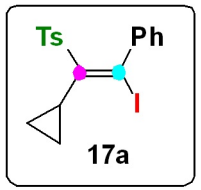


16a

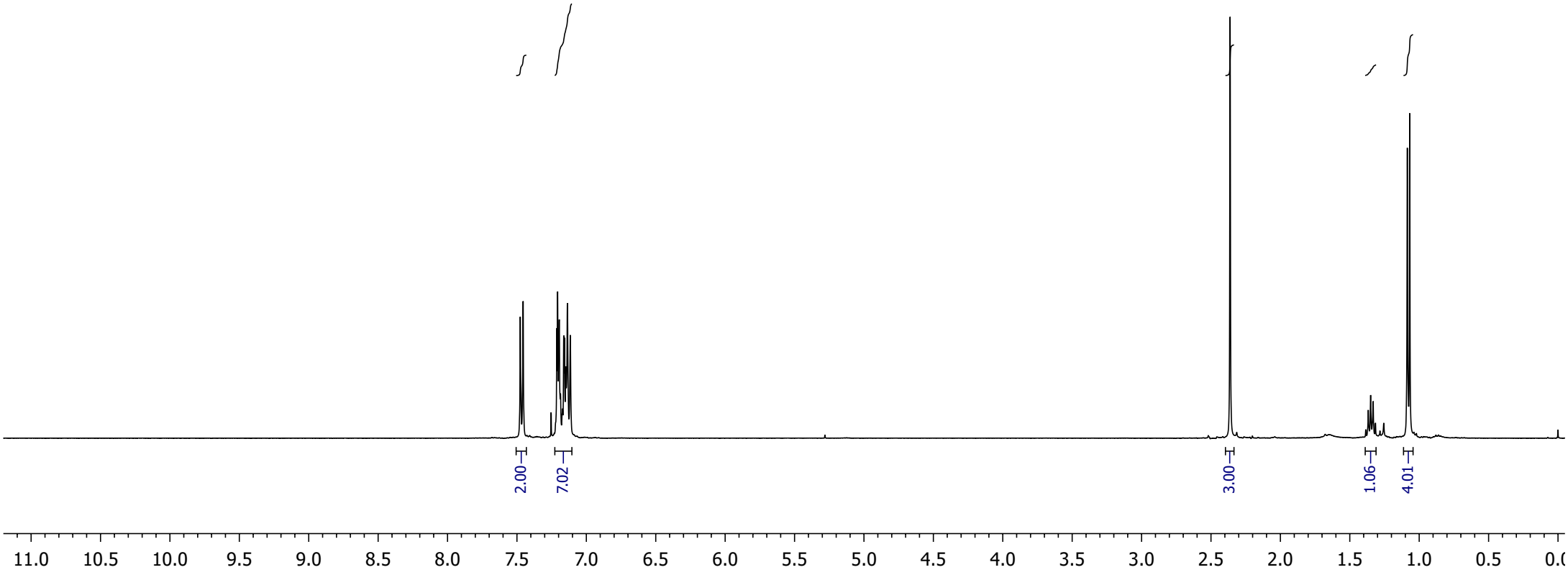
U0111-1
LJ3-27

7.48 7.46 7.21 7.21 7.20 7.16 7.16 7.15 7.14 7.12 2.36 1.38 1.37 1.35 1.33 1.32 1.09 1.07

Solvent $CDCl_3$
Spectrometer Frequency 400.28
Nucleus ^1H



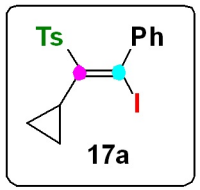
17a



U0111-2
LJ3-27

147.6
143.6
142.8
137.6
129.1
128.9
128.2
127.8
127.6
119.8
77.3
77.0
76.7
21.5
20.6
12.0

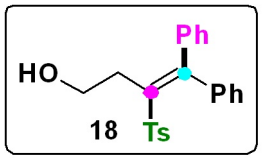
Solvent $CDCl_3$
Spectrometer Frequency 100.66
Nucleus ^{13}C



17a

M0394-4
LJ3-App-2

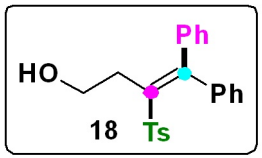
Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H

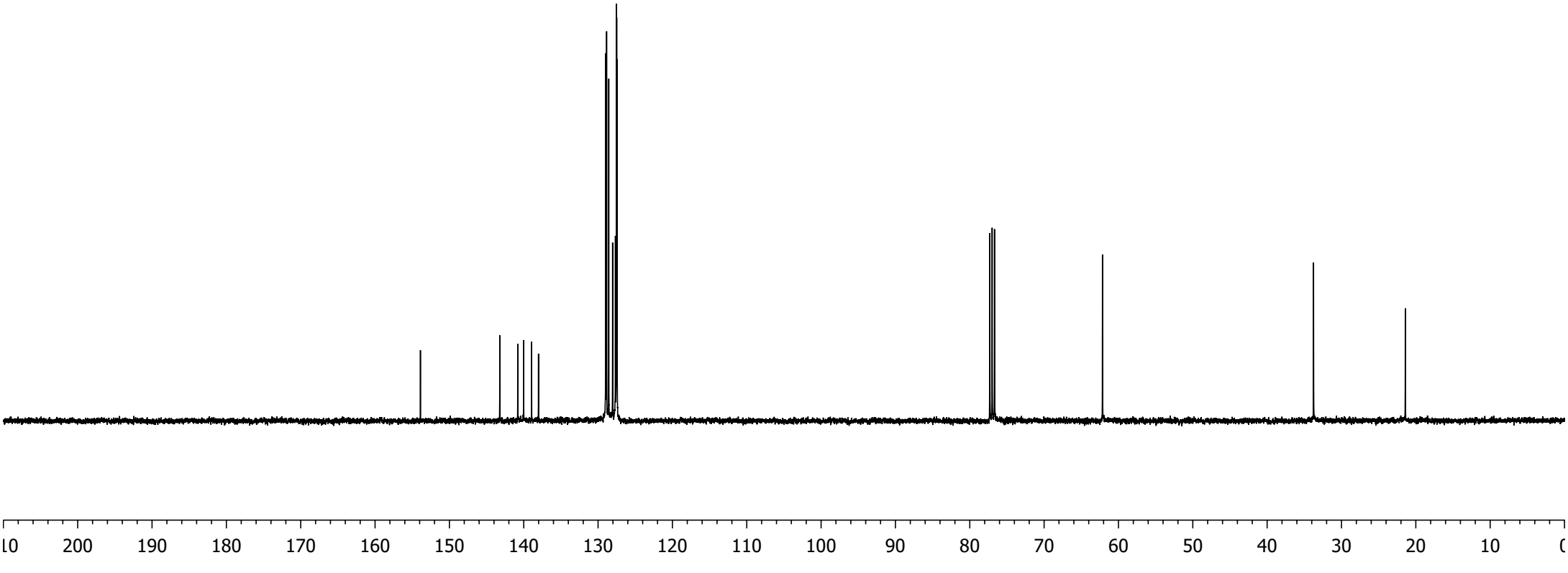


M0394-5
LJ3-App-2

153.9
143.2
140.8
140.0
138.9
138.0
129.0
128.9
128.6
128.0
127.7
127.5
127.5
127.4
77.3
77.0
76.7
62.2
33.8
21.4

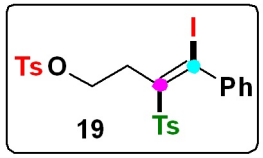
Solvent $CDCl_3$
Spectrometer Frequency 100.69
Nucleus ^{13}C

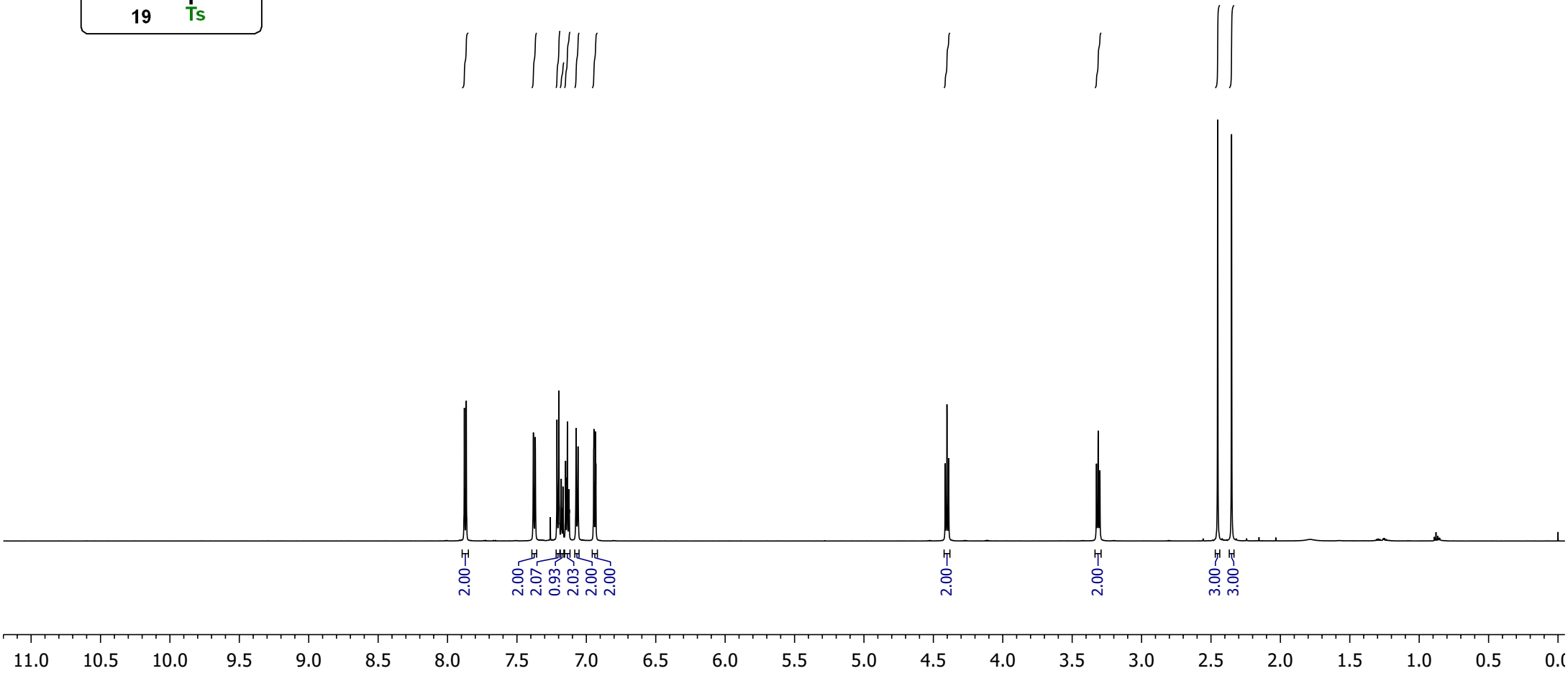




V0118-4
LJ3-App-1

Solvent $CDCl_3$
Spectrometer Frequency 597.22
Nucleus ^1H

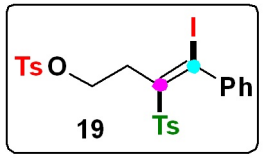


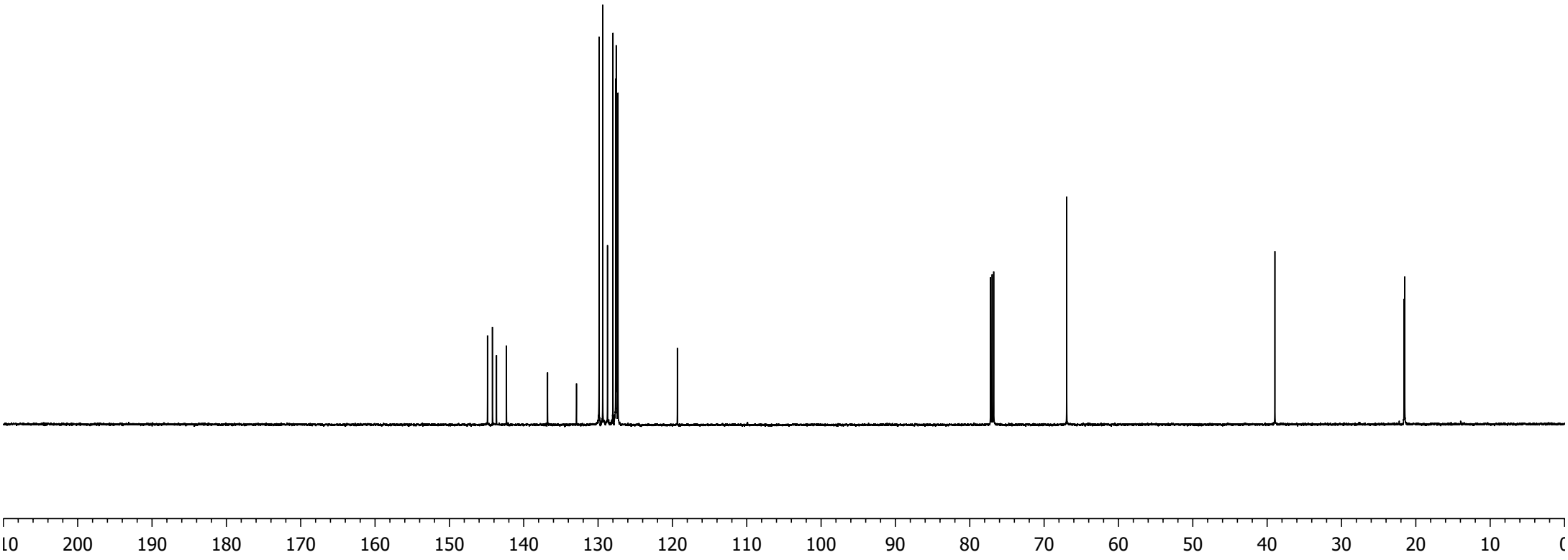


V0118-5
LJ3-App-1

144.9
144.2
143.7
142.3
129.9
129.4
128.7
128.0
127.6
127.6
127.3
119.3
77.2
77.0
76.8
67.0
39.0
21.6
21.5

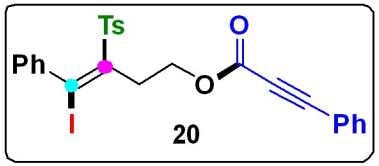
Solvent $CDCl_3$
Spectrometer Frequency 150.19
Nucleus ^{13}C





M0401-1
LJ3-App-3

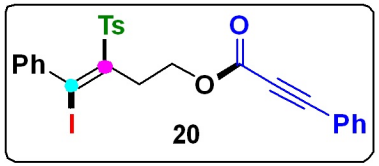
Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H

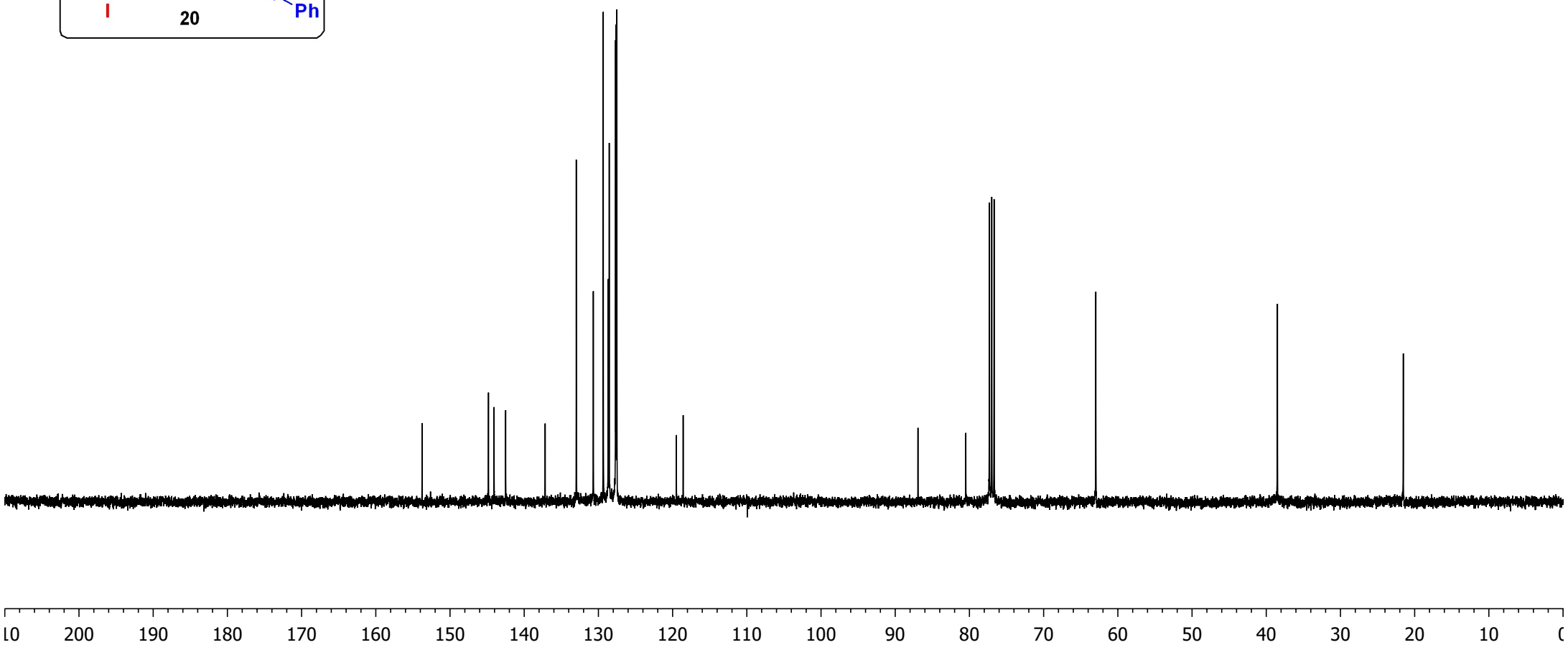


M0401-2
LJ3-App-3

153.8
144.8
144.1
142.5
133.0
129.3
128.7
128.5
127.7
127.6
127.5
119.5
118.6
86.9
80.5
77.3
77.0
76.7
63.0
38.5
21.5

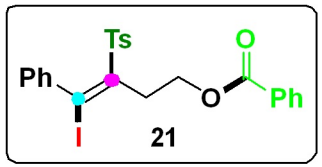
Solvent $CDCl_3$
Spectrometer Frequency 100.69
Nucleus ^{13}C

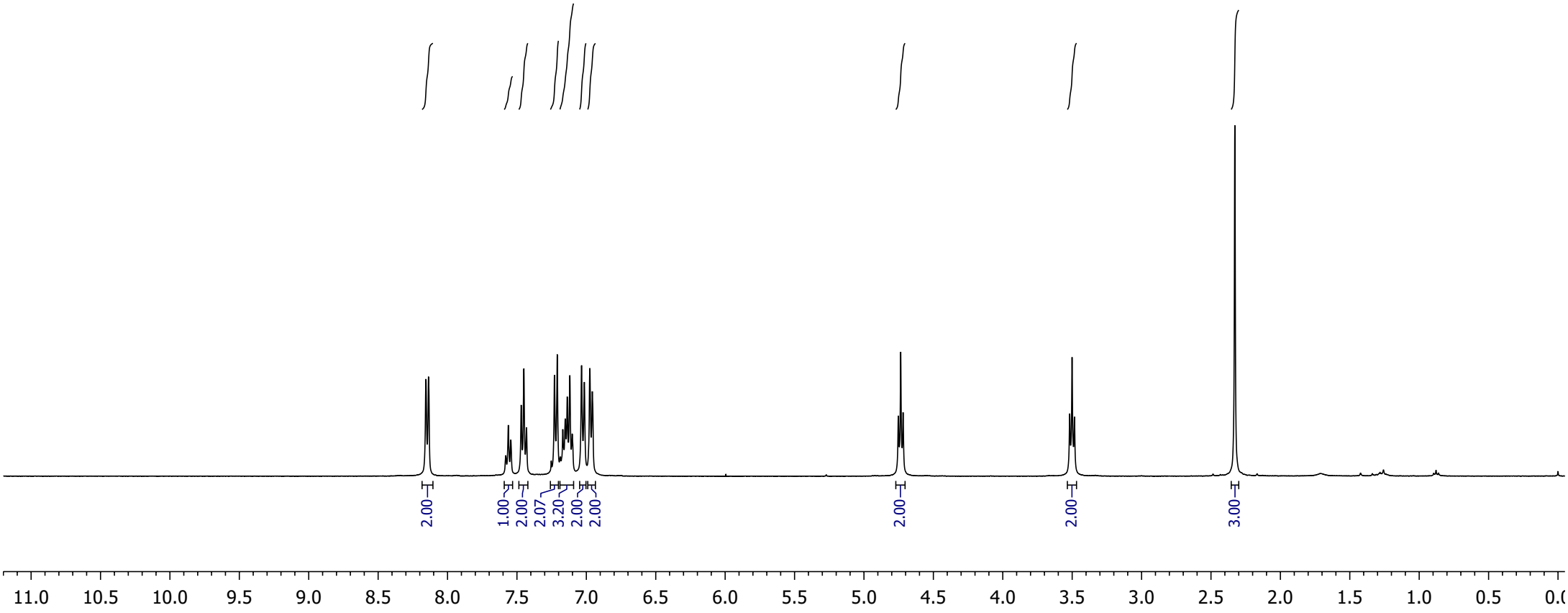




M0415-1
LJ3-App-4

Solvent $CDCl_3$
Spectrometer Frequency 400.40
Nucleus ^1H

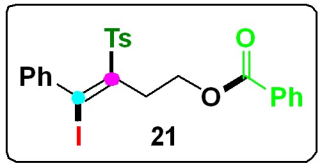




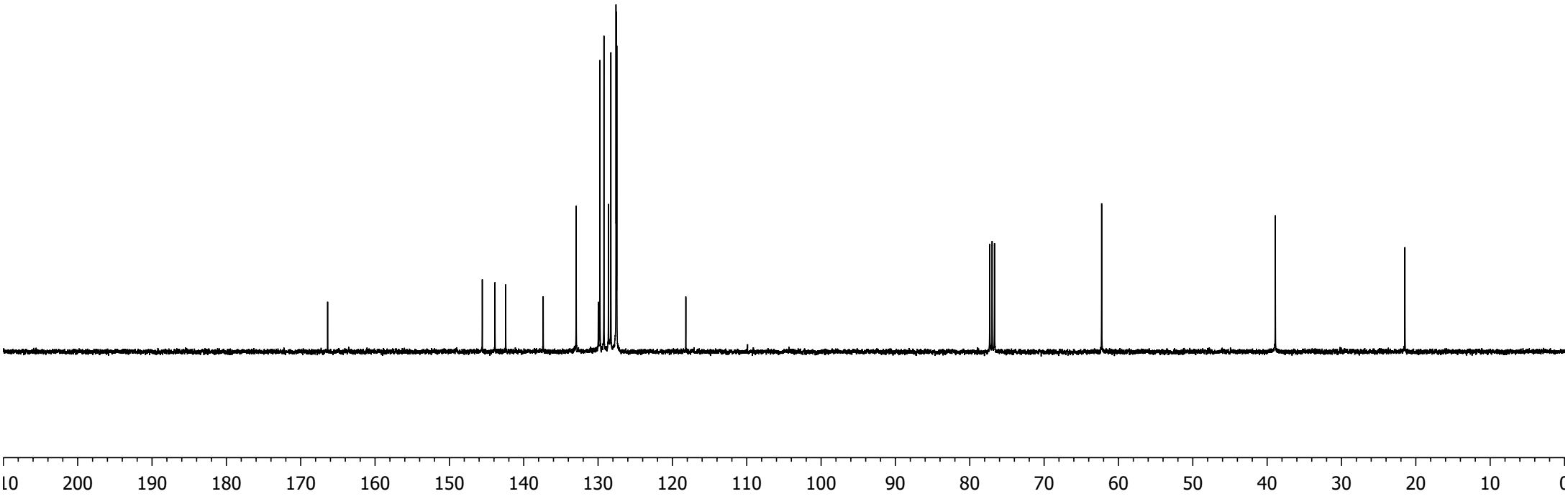
M0415-2
LJ3-App-4

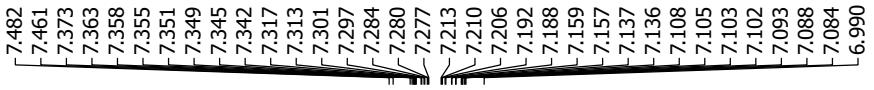
166.4
145.6
143.9
142.4
132.9
129.8
129.2
128.6
128.3
127.6
127.5
118.2
77.3
77.0
76.7
62.3
38.9
21.5

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



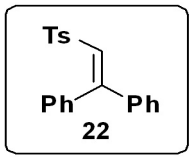
21

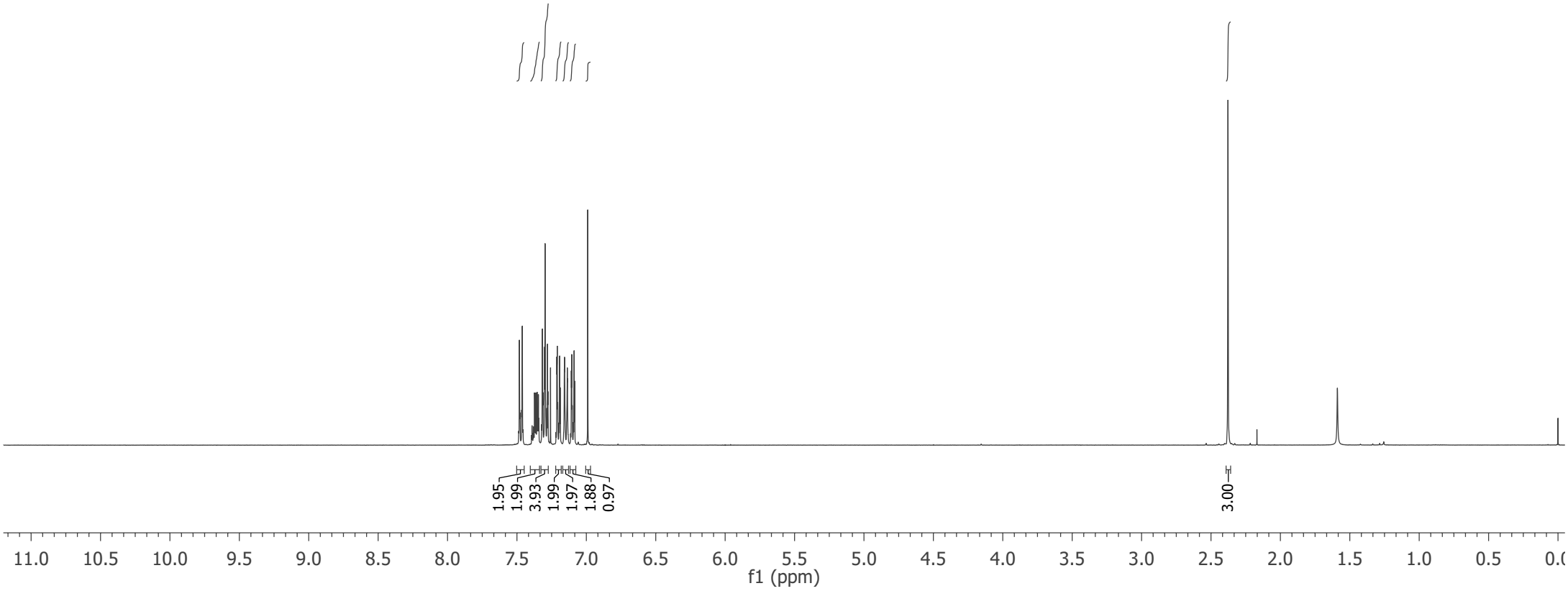




Solvent $CDCl_3$

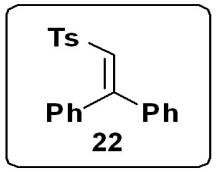
Spectrometer Frequency 400.40





Solvent $CDCl_3$

Spectrometer Frequency 100.69



22

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) k10910-jjw-bmr-11-c-1

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: k10910-jjw-bmr-11-c-1

Bond precision: C-C = 0.0084 A Wavelength=0.71073

Cell: a=9.2544(2) b=9.7531(2) c=30.4790(9)
alpha=89.135(2) beta=88.150(2) gamma=74.562(2)
Temperature: 113 K

	Calculated	Reported
Volume	2650.31(11)	2650.31(11)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C28 H24 I N O4 S2 [+ solvent]	2(C28 H24 I N O4 S2)
Sum formula	C28 H24 I N O4 S2 [+ solvent]	C56 H48 I2 N2 O8 S4
Mr	629.50	1259.00
Dx,g cm-3	1.578	1.578
Z	4	2
Mu (mm-1)	1.401	1.401
F000	1264.0	1264.0
F000'	1263.57	
h,k,lmax	11,11,36	11,11,36
Nref	9304	9290
Tmin,Tmax	0.663,0.756	0.307,1.000
Tmin'	0.565	

Correction method= # Reported T Limits: Tmin=0.307 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.998 Theta(max)= 24.998

R(reflections)= 0.0533(8370)

wR2(reflections)= 0.1063(9290)

S = 1.273 Npar= 653

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

PLAT342_ALERT_3_C	Low Bond Precision on C-C Bonds	0.00837	Ang.
PLAT906_ALERT_3_C	Large K Value in the Analysis of Variance	6.996	Check
PLAT911_ALERT_3_C	Missing FCF Refl Between Thmin & STh/L= 0.595	14	Report
PLAT977_ALERT_2_C	Check Negative Difference Density on H11 .	-0.39	eA-3

Alert level G

PLAT045_ALERT_1_G	Calculated and Reported Z Differ by a Factor ...	2	Check
PLAT083_ALERT_2_G	SHELXL Second Parameter in WGHT Unusually Large	15.11	Why ?
PLAT154_ALERT_1_G	The s.u.'s on the Cell Angles are Equal ..(Note)	0.002	Degree
PLAT431_ALERT_2_G	Short Inter HL..A Contact I1 ..O2 .	2.97	Ang.
	1-x,-y,2-z =	2_657	Check
PLAT605_ALERT_4_G	Largest Solvent Accessible VOID in the Structure	21	A**3
PLAT883_ALERT_1_G	No Info/Value for _atom_sites_solution_primary .	Please	Do !
PLAT909_ALERT_3_G	Percentage of I>2sig(I) Data at Theta(Max) Still	75%	Note
PLAT910_ALERT_3_G	Missing # of FCF Reflection(s) Below Theta(Min).	2	Note
PLAT933_ALERT_2_G	Number of HKL-OMIT Records in Embedded .res File	5	Note
PLAT967_ALERT_5_G	Note: Two-Theta Cutoff Value in Embedded .res ..	50.0	Degree
PLAT978_ALERT_2_G	Number C-C Bonds with Positive Residual Density.	1	Info

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0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
4 **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

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
5 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. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

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Publication of your CIF in other journals

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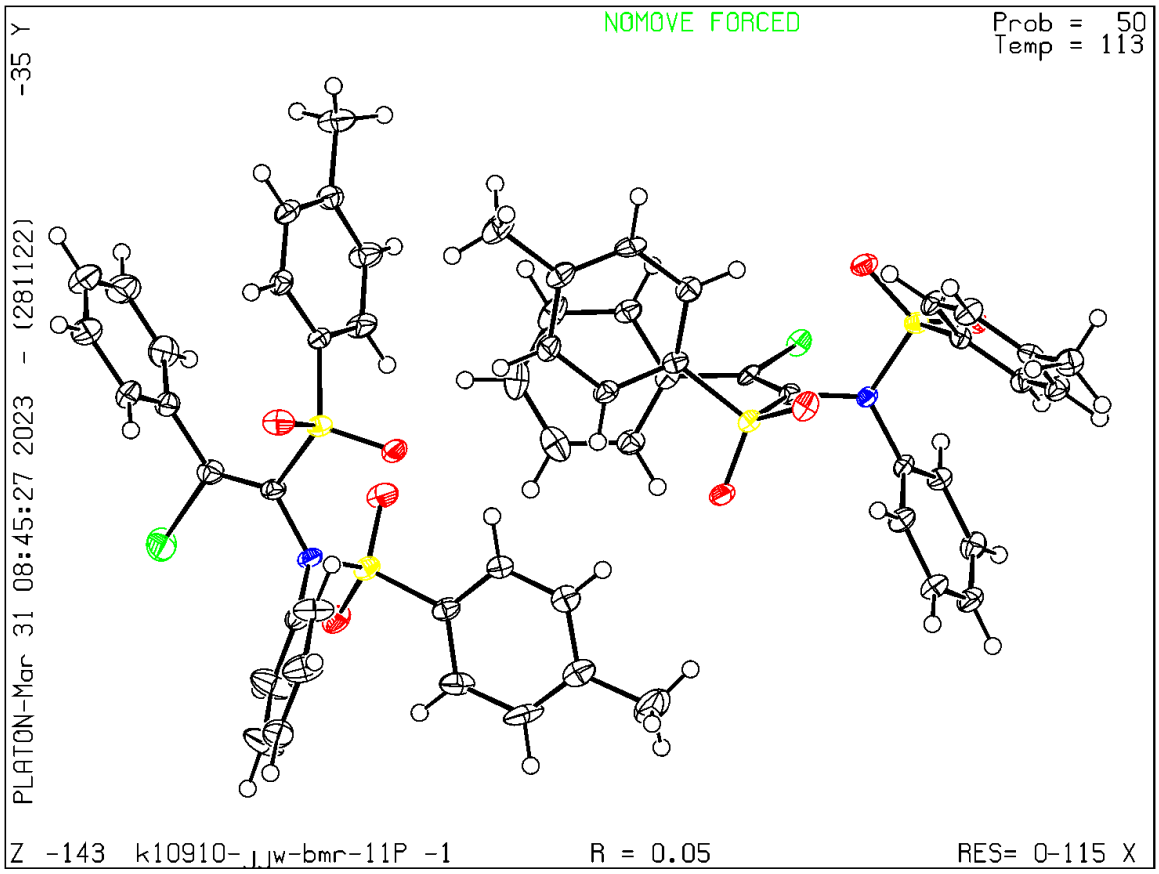
PLATON version of 28/11/2022; check.def file version of 28/11/2022

Datablock k10910-jjw-bmr-11-c-1 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) k11101-jjw-c

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: k11101-jjw-c

Bond precision: C-C = 0.0147 A Wavelength=0.71073

Cell: a=8.5557(4) b=10.3154(5) c=29.9202(16)
alpha=93.058(4) beta=96.826(4) gamma=104.064(4)
Temperature: 113 K

	Calculated	Reported
Volume	2534.0(2)	2534.0(2)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C25 H26 I N O4 S2 [+ solvent]	2(C25 H26 I N O4 S2)
Sum formula	C25 H26 I N O4 S2 [+ solvent]	C50 H52 I2 N2 O8 S4
Mr	595.49	1190.97
Dx,g cm-3	1.561	1.561
Z	4	2
Mu (mm-1)	1.460	1.460
F000	1200.0	1200.0
F000'	1199.54	
h,k,lmax	10,12,35	10,12,35
Nref	8921	8908
Tmin,Tmax	0.769,0.803	0.244,1.000
Tmin'	0.747	

Correction method= # Reported T Limits: Tmin=0.244 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.999 Theta(max)= 25.000

R(reflections)= 0.0909(5053) wR2(reflections)= 0.2357(8908)

S = 1.028 Npar= 611

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

RINTA01_ALERT_3_C The value of Rint is greater than 0.12
Rint given 0.150

PLAT020_ALERT_3_C	The Value of Rint is Greater Than 0.12	0.150	Report
PLAT342_ALERT_3_C	Low Bond Precision on C-C Bonds	0.01469	Ang.
PLAT906_ALERT_3_C	Large K Value in the Analysis of Variance	10.645	Check
PLAT906_ALERT_3_C	Large K Value in the Analysis of Variance	2.152	Check
PLAT911_ALERT_3_C	Missing FCF Refl Between Thmin & STh/L= 0.595	11	Report
PLAT971_ALERT_2_C	Check Calcd Resid. Dens. 0.15Ang From I2	2.14	eA-3
PLAT971_ALERT_2_C	Check Calcd Resid. Dens. 0.28Ang From I1	2.12	eA-3
PLAT971_ALERT_2_C	Check Calcd Resid. Dens. 0.96Ang From C12	1.65	eA-3
PLAT971_ALERT_2_C	Check Calcd Resid. Dens. 0.99Ang From I1	1.57	eA-3
PLAT971_ALERT_2_C	Check Calcd Resid. Dens. 0.99Ang From I2	1.53	eA-3
PLAT975_ALERT_2_C	Check Calcd Resid. Dens. 0.98Ang From O1 .	0.77	eA-3
PLAT977_ALERT_2_C	Check Negative Difference Density on H16 .	-0.33	eA-3
PLAT977_ALERT_2_C	Check Negative Difference Density on H41 .	-0.31	eA-3
PLAT977_ALERT_2_C	Check Negative Difference Density on H49 .	-0.36	eA-3

Alert level G

PLAT003_ALERT_2_G	Number of Uiso or Uij Restrained non-H Atoms ...	67	Report
PLAT045_ALERT_1_G	Calculated and Reported Z Differ by a Factor ...	2	Check
PLAT072_ALERT_2_G	SHELXL First Parameter in WGHT Unusually Large	0.11	Report
PLAT154_ALERT_1_G	The s.u.'s on the Cell Angles are Equal ..(Note)	0.004	Degree
PLAT178_ALERT_4_G	The CIF-Embedded .res File Contains SIMU Records	1	Report
PLAT188_ALERT_3_G	A Non-default SIMU Restraint Value has been used	0.0100	Report
PLAT301_ALERT_3_G	Main Residue Disorder .....(Resd 1)	3%	Note
PLAT431_ALERT_2_G	Short Inter HL..A Contact I1 ..O1 . 1-x,1-y,-z =	3.32 2_665	Ang. Check
PLAT605_ALERT_4_G	Largest Solvent Accessible VOID in the Structure	9	A**3
PLAT860_ALERT_3_G	Number of Least-Squares Restraints	1998	Note
PLAT883_ALERT_1_G	No Info/Value for _atom_sites_solution_primary .	Please Do !	
PLAT910_ALERT_3_G	Missing # of FCF Reflection(s) Below Theta(Min).	2	Note
PLAT933_ALERT_2_G	Number of HKL-OMIT Records in Embedded .res File	3	Note
PLAT967_ALERT_5_G	Note: Two-Theta Cutoff Value in Embedded .res ..	50.0	Degree
PLAT978_ALERT_2_G	Number C-C Bonds with Positive Residual Density.	0	Info

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15 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
15 **ALERT level G** = General information/check it is not something unexpected

3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data

```
 14 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
  2 ALERT type 4 Improvement, methodology, query or suggestion
  1 ALERT type 5 Informative message, check
```

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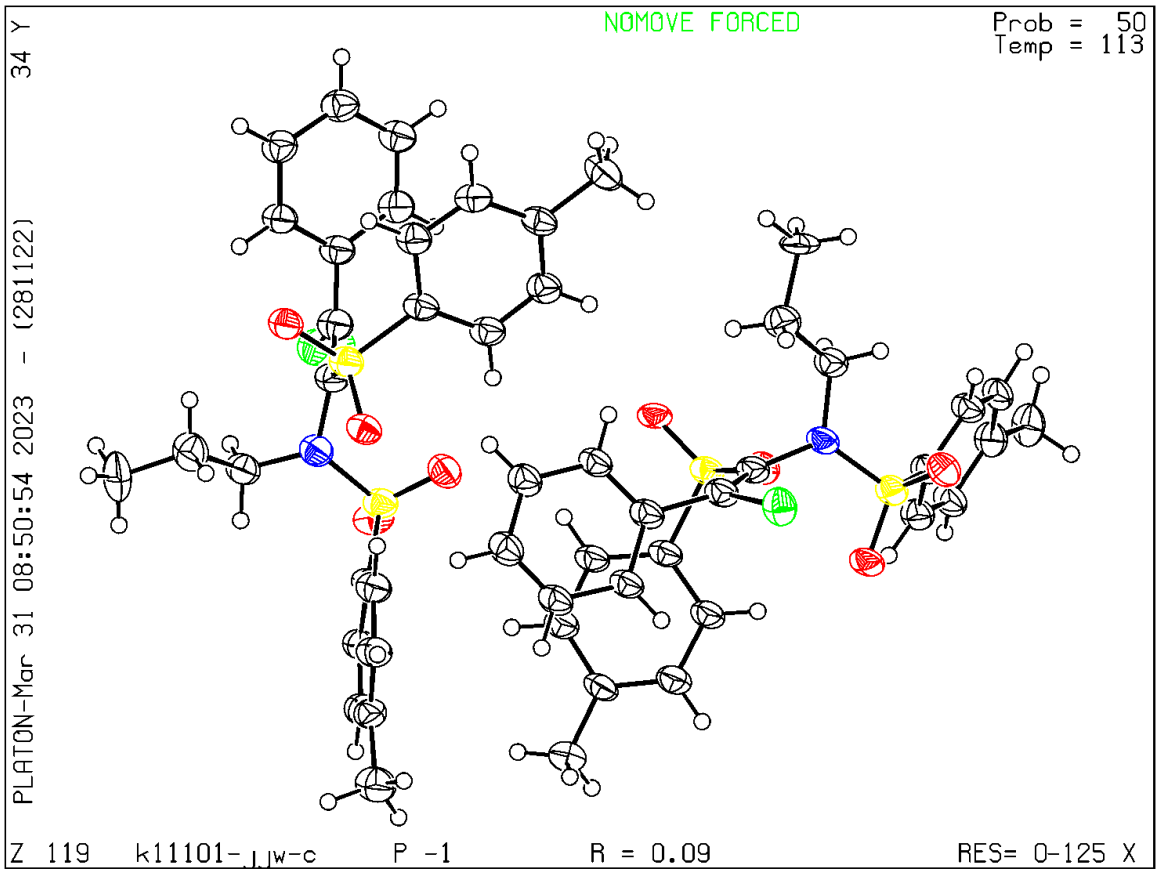
PLATON version of 28/11/2022; check.def file version of 28/11/2022

Datablock k11101-jjw-c - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) k11007-jjw-c

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: k11007-jjw-c

Bond precision: C-C = 0.0049 A Wavelength=0.71073

Cell: a=6.7880(2) b=12.1573(2) c=13.6372(3)
alpha=96.973(2) beta=96.425(2) gamma=102.531(2)
Temperature: 113 K

	Calculated	Reported
Volume	1079.30(5)	1079.30(4)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C22 H19 I O4 S2 [+ solvent]	C22 H19 I O4 S2
Sum formula	C22 H19 I O4 S2 [+ solvent]	C22 H19 I O4 S2
Mr	538.39	538.39
Dx,g cm-3	1.657	1.657
Z	2	2
Mu (mm-1)	1.703	1.703
F000	536.0	536.0
F000'	535.76	
h,k,lmax	8,14,16	8,14,16
Nref	3796	3786
Tmin,Tmax	0.671,0.711	0.590,1.000
Tmin'	0.501	

Correction method= # Reported T Limits: Tmin=0.590 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.997 Theta(max)= 24.996

R(reflections)= 0.0310(3475) wR2(reflections)= 0.0810(3786)

S = 1.060 Npar= 264

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

PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.595 9 Report

---

Alert level G

PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note) 0.002 Degree
PLAT431_ALERT_2_G Short Inter HL..A Contact I1 ..O1 . 3.08 Ang.
2-x,2-y,1-z = 2_776 Check
PLAT605_ALERT_4_G Largest Solvent Accessible VOID in the Structure 0 A**3
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still 84% Note
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 2 Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File 3 Note
PLAT967_ALERT_5_G Note: Two-Theta Cutoff Value in Embedded .res .. 50.0 Degree
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 0 Info

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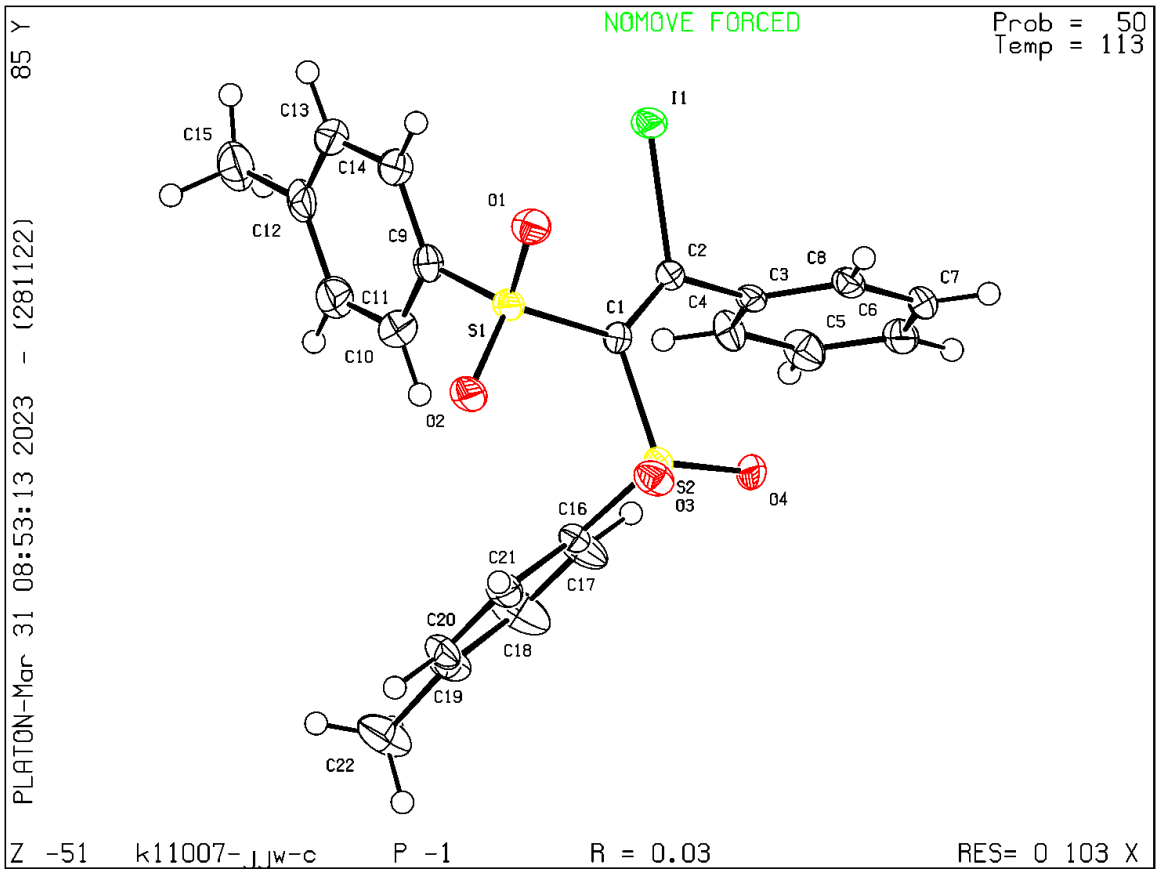
PLATON version of 28/11/2022; check.def file version of 28/11/2022

Datablock k11007-jjw-c - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) k11104-jjw-alj3-7

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: k11104-jjw-alj3-7

```
Bond precision:   C-C = 0.0031 A          Wavelength=0.71073

Cell:           a=7.9819(1)        b=9.8435(1)       c=13.6204(2)
                alpha=82.881(1)    beta=74.573(1)    gamma=74.145(1)
Temperature:    113 K

                Calculated                Reported
Volume          990.83(2)                 990.83(2)
Space group     P -1                      P -1
Hall group      -P 1                      -P 1
Moiety formula  C22 H17 I O4 S            C22 H17 I O4 S
Sum formula     C22 H17 I O4 S            C22 H17 I O4 S
Mr              504.32                    504.31
Dx,g cm-3       1.690                     1.690
Z               2                         2
Mu (mm-1)       1.747                     1.747
F000            500.0                     500.0
F000'           499.51
h,k,lmax        9,11,16                   9,11,16
Nref            3494                      3490
Tmin,Tmax       0.664,0.705               0.468,1.000
Tmin'           0.640

Correction method= # Reported T Limits: Tmin=0.468 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.999           Theta(max)= 25.000

R(reflections)= 0.0196( 3334)                wR2(reflections)=
                                             0.0468( 3490)
S = 1.078                  Npar= 254
```

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

PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.595 3 Report

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Alert level G

PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 6 Report
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note) 0.001 Degree
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records 1 Report
PLAT188_ALERT_3_G A Non-default SIMU Restraint Value has been used 0.0100 Report
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 60 Note
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still 90% Note
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 1 Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File 1 Note
PLAT967_ALERT_5_G Note: Two-Theta Cutoff Value in Embedded .res .. 50.0 Degree
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 12 Info

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

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
3 ALERT type 2 Indicator that the structure model may be wrong or deficient
5 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. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

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

Publication of your CIF in other journals

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

PLATON version of 28/11/2022; check.def file version of 28/11/2022

Datablock k11104-jjw-alj3-7 - ellipsoid plot