Supporting Information

Gold-Catalyzed Bicyclic Annulations of *N*-(o-Alkynylphenyl)imines with α-Diazo Esters to Form 5,6-Dihydroindolo[2,1-a]isoquinolines.

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Contents:

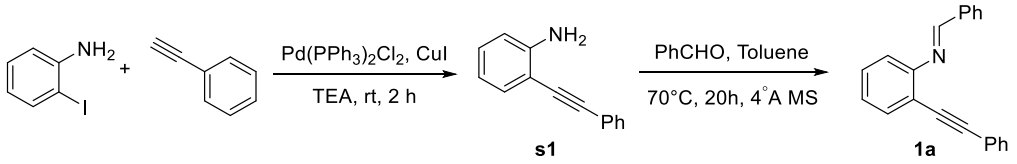
1. Representative Synthetic Procedures:

(a) General procedure:

Unless otherwise noted, all the preparations of substrates were performed in oven-dried glassware under nitrogen atmosphere with freshly distilled solvents. The catalytic reactions were performed under Nitrogen atmosphere. DCE, DCM and Ether were distilled from CaH_2 under nitrogen. THF were distilled from Na metal under nitrogen. All other commercial reagents were used without further purification, unless otherwise indicated. ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker 400, 600 MHz and Varian 700 MHz spectrometers using chloroform-*d* ($CDCl_3$) and DMSO-d_6 as the internal standards. High-resolution mass spectral analysis (HRMS) data were measured on JMS-T100LP4G (JEOL) mass spectrometer or a TOF mass analyzer equipped with the ESI source and Magnetic Sector Mass Analyzer (MStation) equipped with the EI source. Single-crystal X-ray diffraction intensity data were collected on a Bruker X8 APEX diffractometer equipped with a CCD area detector and Mo Kα radiation (λ = 0.71073 Å) at 100 K; all data calculations were performed by using the PC version of the APEX2 program package. Final R indices were obtained using those reflections I > 2σ(I).

(b) Preparation of (*E*)-1-Phenyl-N-(2-(phenylethynyl)phenyl)methanimine (1a).

All *N*-(*o*-alkynylphenyl)imines (**1a-1p**) were prepared from procedure reported in the literature.[s1]

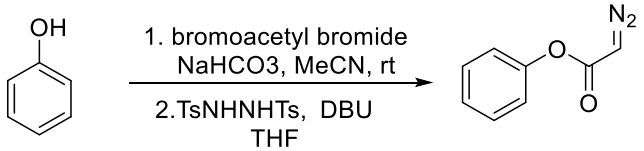


General procedure for synthesis of 2-(phenylethynyl)aniline (**s1**): To a 150 mL flask charged with 2-iodoaniline (2 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (0.04 mmol, 2 mol%), and CuI (0.08 mmol, 4 mol%) in degassed Et_3N (5 mL) was added ethynylbenzene (2.4 mmol, 1.2 equiv), and the resulting solution was stirred at room temperature for 2 h. Upon completion, the solvent was removed under reduced pressure, and the residue was extracted with EtOAc (3 x 5 mL). The combined organic layer was dried over Na_2SO_4 and concentrated. The residue was purified by a silica gel flash chromatography (petroleum ether / ethyl acetate = 10 / 1) to afford 2-(phenylethynyl)aniline **s1** as yellow solid (1.81 mmol, 91%).

To a stirred solution of 2-(phenylethynyl)aniline (**s1**) (2.6 mmol) in toluene (5.0 mL) were added 5Å MS (5.0 g) and benzaldehyde (2.7 mmol) at room temperature. The resulting mixture was heated at 70 °C in oil bath with stirring for 20 h, then this reaction mixture was filtered through a short celite bed and the filtrate was concentrated under reduced pressure to afford (E)-1-phenyl-N-(2-(phenylethynyl)phenyl)methanimine (**1a**). Crude product of **1a** was used directly for further catalytic reaction. Similarly, similar substrates **1b** to 1**p** were synthesized following similar synthetic procedure from their respective starting materials. which were used as such without further purification.

(c) Preparation of α-diazo esters.

All α-diazo esters (**2b-2e**) were prepared from the reported procedure in the literatures.[s2]



2e

To a solution of phenol (0.50 g, 5.3 mmol) and NaHCO3 (1.34 g, 15.9 mmol) in acetonitrile (20 mL) was added bromoacetyl bromide (1.61 g, 7.9 mmol) dropwise at 0 °C; the resulting mixture was stirred for 15 min at room temperature, quenched with water, and extracted with DCM. The organic layer was washed with brine, dried with $MgSO_4$, and concentrated in vacuo to give a bromoacetate product, which was used in the next step without purification. The bromoacetate product (0.98 g, 4.9 mmol) and N,N'-ditosylhydrazine (3.35 g, 9.8 mmol) were dissolved in THF (20 mL), and cooled to 0 °C, and to this mixture was added DBU (3.75 g, 24.6 mmol) was added slowly over 5-10 min. After stirring for 15 min at the same temperature, the reaction solution was quenched with saturated NaHCO3 and extracted with Et2O three times. Organic phase was washed with brine, dried with $MgSO_4$ and evaporate to dryness, affording diazo crude product which was purified on a silica column using ethyl acetate/hexane (20/80) as the eluent to give desired diazoacetate **2e** (0.78 g, 4.8 mmol, 91%) as a pale-yellow oil.

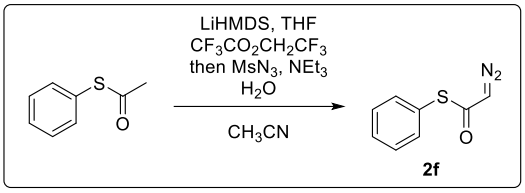
2a is commercially available from Sigma-Aldrich and other diazo esters (**2b-2d**) were prepared with the above procedure.

***tert*-butyl 2-diazoacetate (2b):** crude product which was purified on a silica column using ethyl acetate/hexane (20/80) as the eluent to give desired diazoacetate **2b** (0.68 g, 4.8 mmol, 71%) as a pale-yellow oil.

Isbutyl 2-diazoacetate(2c): crude product which was purified on a silica column using ethyl acetate/hexane (20/80) as the eluent to give desired diazoacetate **2c** (0.52 g, 366 mmol, 54%) as a pale-yellow oil.

Benzyl 2-diazoacetate (2d): crude product which was purified on a silica column using ethyl acetate/hexane (20/80) as the eluent to give desired diazoacetate **2d** (0.65 g, 3.69 mmol, 80%) as a pale-yellow oil.

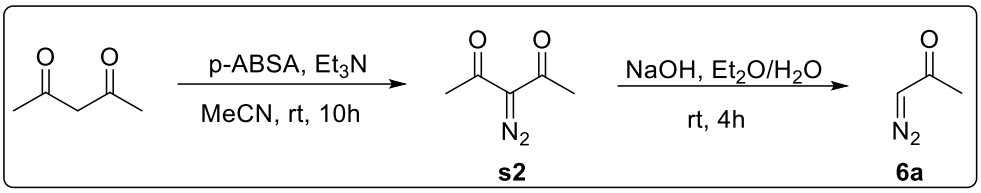
S-phenyl 2- diazoethanethioate (**2f):**



A 100-mL, three-necked, round bottomed flask was charged with 1,1,1,3,3,3-hexamethyldisilazane (1.15 g, 7.11 mmol) and 18 mL of THF and then cooled at 0°C while n-BuLi (2.40 M in hexane, 2.90 mL, 6.96 mmol) was added rapidly dropwise. The resulting solution was stirred at 0 °C for 15 min, and then cooled at -78 °C while a solution of phenyl thioacetate (1.0 g, 6.57 mmol) in 14 mL of THF was added dropwise over 15 min. The reaction mixture was stirred at -78 °C for 30 min, and then 2,2,2-trifluoroethyl trifluoroacetate (1.61 g, 8.21 mmol) was added rapidly in one portion. The reaction mixture was stirred at -78 °C for 10 min and then partitioned between 40 mL of 5% HCl solution and 50 mL of diethyl ether. The aqueous phase was separated and extracted with diethyl ether, and the combined organic phases were washed with 45 mL of brine and concentrated to afford 2.25 g of a yellow oil which was immediately dissolved in 25 mL of acetonitrile and transferred via cannula to a 100-mL, three necked round-bottomed flask. Water (0.118 g, 6.55 mmol) and triethylamine (1.02 g, 10.0 mmol) were added rapidly, and then a solution of methanesulfonyl azide (1.20 g, 9.89 mmol) in 28 mL of acetonitrile was added over 20 min. The resulting yellow solution was stirred at 25 °C for 2.5 h and then concentrated and residue was diluted with 50 mL of diethyl ether and washed with three 40-mL portions of 10% NaOH solution and 40 mL of brine, dried over MgSO4, filtered, and concentrated. The crude product was purified on a silica column using ethyl acetate/hexane (10/90) as the eluent to give desired diazoethanethioate **2f** (0.84 g, 4.7 mmol, 72%) as a yellow oil.

Spectral data of known compounds diazo esters (**2b-2f**) were already reported in the literatures.[s2, s3]

(d) **Preparation of α-diazo methyl ketone.**



The compound **6a** was prepared according to literature procedure[s4]. In a well-dried 200-mL flask, acetylacetone (0.5 g, 4.9 mmol) and Et_3N (0.76 mL, 5.5 mmol) was dissolved in 10 mL acetonitrile, and a solution of p-ABSA (1.2 g, 4.9 mmol) in 10 mL acetonitrile was added dropwise with stirring. The reaction mixture was stirred at room temperature under air atmosphere overnight (about 10 h), and the solvent was removed under reduced pressure with a rotary evaporator. The product was purified by column chromatography (silica gel) using petro ether (PE)/ ethyl acetate (EA) 5:1 as eluent to give (560mg, 4.4 mmol, 89%) of compound 3-diazopentane-2,4- dione (**s2**) as a yellow liquid. After that, a solution of compound **s2** (0.560 g, 4.4 mmol) in diethyl ether (10 mL) was added an aqueous NaOH solution (0.89 g, 22.2 mmol in 10 mL water), and the mixture was stirred at room temperature under nitrogen atmosphere for 4 h. The phases were divided using a separatory funnel, and the aqueous phase was extracted with dichloromethane 2 times. The combined organic phase was dried over Na2SO4 and filtered, and the filtrate was concentrated under reduced pressure to give the product **6a** as a volatile yellow liquid (0.24 g, 2.8mmol, 64% yield). Spectral data of this know compound reported in literature. [s4]

References

[s1] (a) Chakrabarty, I.; Inamdar, S. M.; Akram, M. O.; Gade, A. B.; Banerjee, S.; Bera, S.; Patil, N. T. *Chem. Commun.* **2017**, *53*, 196-199. (b) Kusama, H.; Miyashita, Y.; Takaya, J.; Iwasawa, N. *Org. Lett.* **2006**, *8*, 289-292; (c) Gabriele, B.; Veltri, L.; Salerno, G.; Mancuso, R.; Costa, M.. *Adv. Synth. Catal.* **2010**, *352*, 3355−3363. (d) Kusama, H.; Takaya, J.; Iwasawa, N. *J. Am. Chem. Soc.* 2002, 124, 11592-11593. (e) Takeda, A.; Kamijo, S.; Yamamoto, Y. *J. Am. Chem. Soc.* 2000, 122, 5662-5663.

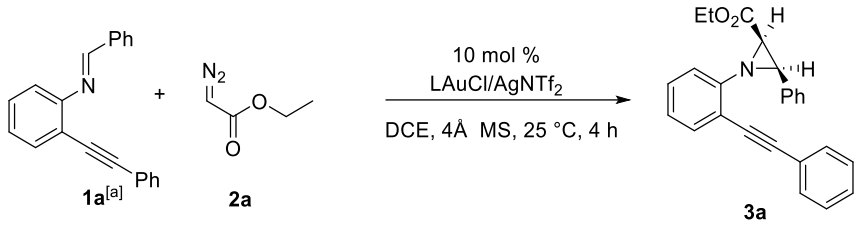
[s2] (a) Torna, T.; Shimokawa, J.; Fukuyama, T. *Org. Lett.* **2007**, *9*, 3195–3197; (b) Mao, H.; Lin, A.; Shi, Y.; Mao, Z.; Zhu, X.; Li, W.; Hu, H.; Cheng, Y.; Zhu, C. *Angew. Chem. Int. Ed.* **2013**, *52*, 6288–6292; (c) Doyle, M. P.; Dyatkin, A. B. *J. Org. Chem.* **1995,** *60*, 3035–3038; (d) Kardile, R. D.; Liu, R.-S. *Org. Lett.* **2019**, *21*, 6452−6456.

[s3] Lawlor, M. D.; Lee, T. W.; Danheiser, R. L. *J. Org. Chem.* **2000**, *65*, 4375–4384.

[s4] Liu, Q.; Li, M.; Xiong, R.; Mo, F. *Org. Lett.* **2017**, *19*, 6756.

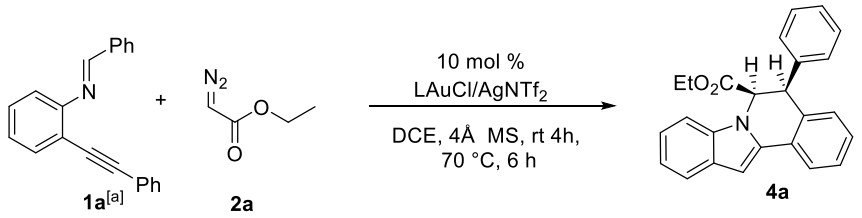
2. Standard procedures for catalytic operations.

(a) Typical procedure for the synthesis of ethyl (2R,3R)-3-phenyl-1-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate (3a):



A suspension of LAuCl L= P(*t*-Bu)$_2$(*o*-biphenyl (53 mg, 0.10 mmol) and AgNTf$_2$ (39 mg, 0.10 mmol) in dry DCE (1.0 mL) was fitted with a N_2 balloon, and the mixture was stirred at 25 °C for 5 min. To this mixture was added a dry DCE (2.0 mL) solution of 2-(phenylethynyl)benzaldehyde **1a** (281 mg, 1.0 mmol) and ethyl 2-diazoacetate **2a** (137 mg, 1.2 mmol) at 25 °C; the resulting mixture was stirred at 25 °C for 4 h. The solution was filtered over a short celite bed and evaporated under reduced pressure. The residue was purified on a silica gel column using ethyl acetate/hexane (05:95) as the eluent to give compound **3a** as yellow oil (327 mg, 0.89 mmol, 89%).

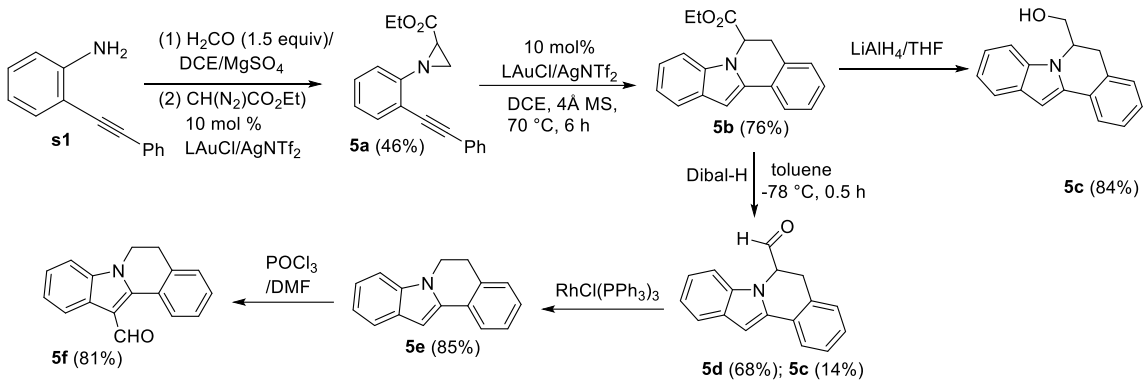
(b) Typical procedure for the synthesis of ethyl (5S,6R)-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4a):



A suspension of LAuCl L= P(*t*-Bu)$_2$(*o*-biphenyl (53 mg, 0.10 mmol) and AgNTf$_2$ (39 mg, 0.10 mmol) in dry DCE (1.0 mL) was fitted with a N_2 balloon, and the mixture was stirred at 25 °C for 5 min. To this mixture was added a dry DCE (2.0 mL) solution of 2-(phenylethynyl)benzaldehyde **1a** (281 mg, 1.0 mmol) and ethyl 2-diazoacetate **2a** (137 mg, 1.2 mmol) at 25 °C. The resulting mixture was stirred at 25 °C for 4 h, followed by heating at 70 °C (oil bath) for 4 h. The solution was filtered over a short celite bed and evaporated under reduced pressure. The residue was

purified on a silica gel column using ethyl acetate/hexane (05:95) as the eluent to give compound **4a** as yellow oil (301 mg, 0.81 mmol, 82%).

(3) Typical procedure for the synthesis of 5a- 5f:



(a) Typical procedure for the synthesis of ethyl 1-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate (5a):

An aqueous formaldehyde solution (37%) (63mg, 0.776 mmol) was added to a mixture of dry DCE (1 mL) and $MgSO_4$, and to this solution was added compound **s1** (100mg, 0.517mmol), and reaction mixture was stirred for 3 h at room temperature. To this reaction mixture, α-diazo ester (71mg, 0.620mmol) was added and then combine mixture was treated to a stirred suspension of LAuCl L= P(*t*-Bu)$_2$(*o*-biphenyl (27 mg, 0.052 mmol) and $AgNTf_2$ (20 mg, 0.052 mmol) in dry DCE (1.0 mL) was fitted with a N_2 balloon, and the mixture was stirred at 25 °C for 4 h. After completion of reaction, solution was filtered over a short celite bed and evaporated under reduced pressure. The residue was purified on a silica gel column using ethyl acetate/hexane (20:80) as eluent to give compound **5a** as brown oil (70 mg, 0.24 mmol, 46%).

(b) Typical procedure for the synthesis of ethyl 5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (5b):

A suspension of LAuCl L= P(*t*-Bu)$_2$(*o*-biphenyl (18 mg, 0.034mmol) and $AgNTf_2$ (13 mg, 0.034 mmol) in dry DCE (1.0 mL) was fitted with a N_2 balloon, and the mixture was stirred at 25 °C for 5 min. To this mixture was added a dry DCE (2.0 mL) solution of ethyl 1-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate **5a** (100 mg, 0.34 mmol) and heated at 70 °C (oil bath) for 6 h. The solution was filtered over a short celite bed and evaporated under reduced pressure. The residue was purified on a silica gel column using ethyl acetate/hexane (05:95) as eluent to give compound **5b** as yellow oil (76 mg, 0.26 mmol, 76%).

(c) Typical procedure for the synthesis of (5,6-dihydroindolo[2,1-a]isoquinolin-6-yl)methanol (5c):

To a stirred solution of ethyl 5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate **5b** (100mg, 0.34mmol) in dry THF (2mL) was added $LiAlH_4$ (30mg, 0.28 mmol) at 0 °C. The resulting mixture was sirred for 4 h at same temperature. The solution was quenched with a saturated solution of ammonium chloride at 0 °C; and the solution was then extracted with ethyl acetate three times. Organic phase was washed with brine, dried with $MgSO_4$ and concentrated under reduced pressure. The residue was purified on a silica column using ethyl acetate/hexane (30:70) as the eluent to give compound **5c** as Yellow oil (72 mg, 0.29 mmol, 84%).

(d) Typical procedure for the synthesis of 5,6-dihydroindolo[2,1-a]isoquinoline-6-carbaldehyde (5d):

A THF solution (2 mL) of ethyl 5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate **5b** (100mg, 0.34mmol) was cooled to -78 °C, and to this suspension was added 1.2 M DIBAl-H in hexane (0.29mL, 0.34mmol) over a period of 15 min. The mixture was stirred for 30 min at -78 °C. The resulting mixture was quenched with a saturated solution of potassium sodium tartrate; the solution was extracted with ethyl acetate two times. Organic phase was washed with brine, dried with $MgSO_4$ and concentrated under reduced pressure. The residue was purified on a silica gel column using ethyl acetate/hexane (20:80) as the eluent to give compound **5d** as a yellow oil (58 mg, 0.23 mmol, 68%)

(e) Typical procedure for the synthesis of 5,6-dihydroindolo[2,1-a]isoquinoline (5e)

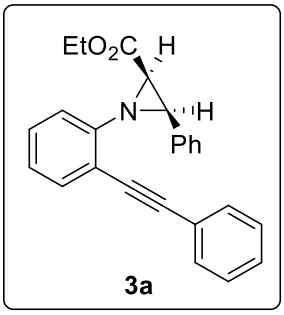
To a solution of 5,6-dihydroindolo[2,1-a]isoquinoline-6-carbaldehyde **5d** (100mg,) in benzene (2 mL) was added $RhCl(PPh_3)_3$ (374 mg, 0.40 mmol). The mixture was stirred for 8 h at 80 °C before the solution was filtered over a short celite bed and evaporated under reduced pressure. The residue was purified on a silica column using ethyl acetate/hexane (05:95) as the eluent to give compound **5e** as white solid (75 mg, 0.34 mmol, 85%).

(f) Typical procedure for the synthesis of 5,6-dihydroindolo[2,1-a]isoquinoline-12-carbaldehyde (5f):

$POCl_3$ (84mg, 0.55mmol) was added to dry DMF (1mL) at 0 °C and stirred for 30 min. To this mixture was added a dry DMF (3 mL) solution of 5,6-dihydroindolo[2,1-a]isoquinoline **5e** (100 mg, 0.46 mmol) at room temperatures. After stirring for 45 min at 35 °C in an oil bath, the reaction mixture was cooled and treated with water was (1.0 mL). The solution was then treated with by 30%. NaOH aqueous solution and heated to 35 °C for 30 min. The organic phase was extracted with diethyl ether twice. Organic phase was washed with brine, dried with $MgSO_4$ and concentrated under reduced pressure. The residue was purified on a silica gel column using ethyl acetate/hexane (20:80) as the eluent to give compound **5f** as white solid (91 mg, 0.37 mmol, 81%)

4. Spectral data for key compounds:

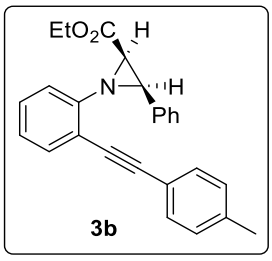
Spectral data of ethyl (2R,3R)-3-phenyl-1-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate (3a):



3a

Compound **3a** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Brown oil (327 mg, 0.89 mmol, 89%); ^{1}H NMR 400 MHz, $CDCl_3$): δ 7.56 (dd, *J* = 8.0, 1.2 Hz, 2H), 7.49 ~ 7.47 (m, 1H), 7.33 ~ 7.24 (m, 4H), 7.22 ~ 7.15 (m, 3H), 7.09 ~ 7.07 (m, 2H), 7.04 ~ 7.03 (m, 2H), 3.90 ~ 3.84 (m, 2H), 3.77 (d, *J* = 6.8 Hz, 1H), 3.34 (d, *J* = 6.8 Hz, 1H), 0.86 (t, *J* = 7.2 Hz, 3H), ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.5, 153.2, 134.6, 133.3, 131.4, 129.2, 128.1, 128.0, 127.9, 127.8, 127.7, 123.1, 123.0, 119.9, 115.6, 95.6, 86.1, 60.9, 47.9, 46.7, 13.8; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{21}NO_2$: 367.1572; found: 367.1574.

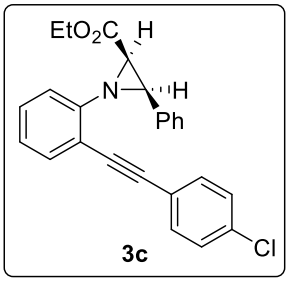
Spectral data of ethyl (2R,3R)-3-phenyl-1-(2-(p-tolylethynyl)phenyl)aziridine-2-carboxylate (3b):



3b

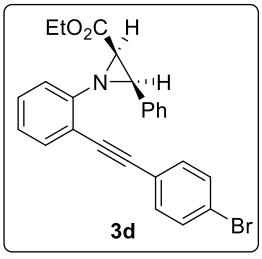
Compound **3b** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (54 mg, 0.14 mmol, 84%); ^{1}H NMR (700 MHz, $CDCl_3$): ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.55 (d, *J* = 7.0 Hz, 2H), 7.46 (dd, *J* = 7.7 , 0.7 Hz, 1H), 7.30 (t, *J* = 7 Hz, 2H), 7.26 (t, *J* = 7.0 Hz, 1H), 7.23 ~ 7.21 (m, 1H), 7.02 ~ 6.95 (m, 6H), 3.89 ~ 3.86 (m, 2H), 3.77 (d, *J* = 7.0 Hz, 1H), 3.33 (d, *J* = 6.3 Hz, 1H), 2.28 (s, 3H), 0.86 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.6, 153.1, 138.2, 134.6, 133.2, 131.3, 129.0, 128.7, 128.0, 127.9, 127.7, 123.1, 120.0, 119.9, 115.8, 95.9, 85.4, 60.9, 47.9, 46.7, 21.4, 13.8; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{26}H_{23}NO_2$: 381.1729; found: 381.1728.

Spectral data of ethyl (2R,3R)-1-(2-((4-chlorophenyl)ethynyl)phenyl)-3-phenylaziridine-2-carboxylate (3c):



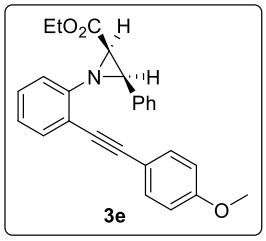
Compound **3c** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (51 mg, 0.13 mmol, 80%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.54 (d, *J* = 7.7 Hz, 2H), 7.45 (dd, *J* = 7.0 , 0.7 Hz, 1H), 7.30 ~ 7.23 (m, 4H), 7.12 (d, *J* = 8.4 Hz, 2H), 7.02 ~ 7.00 (m, 2H), 6.91 (d, *J* = 8.4 Hz, 2H), 3.91 ~ 3.86 (m, 2H), 3.76 (d, *J* = 6.3 Hz, 1H), 3.31 (d, *J* = 7.0 Hz, 1H), 0.88 (t, *J* = 7.7 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.5, 153.3, 134.6, 134.0, 133.2, 132.5, 129.4, 128.3, 128.0,127.9 127.8, 123.1, 121.5, 120.0, 115.2, 94.4, 87.1, 60.9, 47.9, 46.7, 13.8; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{20}ClNO_2$: 401.1183; found: 401.1186.

Spectral data of ethyl (2R,3R)-1-(2-((4-bromophenyl)ethynyl)phenyl)-3-phenylaziridine-2-carboxylate (3d):



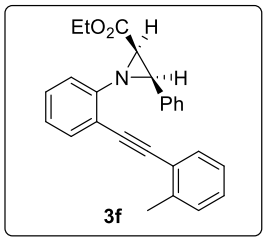
Compound **3d** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Brown Solid (53 mg, 0.12 mmol, 85%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.54 (d, *J* = 7.0 Hz, 2H), 7.45 (d, *J* = 7.7 Hz, 1H), 7.31 ~ 7.24 (m, 6H), 7.03 ~ 7.00 (m, 2H), 6.84 (d, *J* = 8.4 Hz, 2H), 3.91 ~ 3.88 (m, 2H), 3.77 (d, *J* = 7.0 Hz, 1H), 3.32 (d, *J* = 7.0 Hz, 1H), 0.89 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.5, 153.3, 134.6, 133.3, 132.8, 131.2, 129.4, 128.1, 127.9, 127.8, 123.2, 122.3, 122.0, 120.0, 115.2, 94.5, 87.3, 61.0, 47.9, 46.7, 13.8; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{20}BrNO_2$: 445.0677; found: 445.0677.

Spectral data of ethyl (2R,3R)-1-(2-((4-methoxyphenyl)ethynyl)phenyl)-3-phenylaziridine-2-carboxylate (3e):



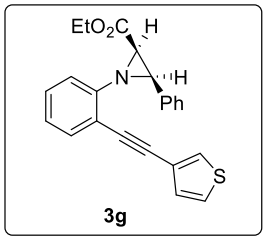
Compound **3e** was purified on silica gel column using ethyl acetate/hexane: (7 : 93) as the eluent; Brown oil (46 mg, 0.12 mmol, 72%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.55 (d, *J* = 7.7 Hz, 2H), 7.46 (dd, *J* = 7.7 , 0.7 Hz, 1H), 7.30 (t, *J* = 7.0 Hz, 2H), 7.26 (t, *J* = 7.0 Hz, 1H), 7.23 ~ 7.20 (m, 1H), 7.01 ~ 6.99 (m, 4H), 3.91 ~ 3.85 (m, 2H), 3.76 (d, *J* = 7.7 Hz, 1H), 3.75(s, 3H) 3.32 (d, *J* = 7 Hz, 1H), 0.87 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.6, 159.5, 153.0, 134.7, 133.1, 132.9, 128.8, 128.0, 127.9, 127.7, 127.1, 119.9, 115.9, 115.2, 113.6, 95.7, 85.8, 60.9, 55.2, 47.9, 46.7, 13.8; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{26}H_{23}NO_3$: 397.1678; found: 397.1677.

Spectral data of ethyl (2R,3R)-3-phenyl-1-(2-(o-tolylethynyl)phenyl)aziridine-2-carboxylate (3f):



Compound **3f** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (56 mg, 0.15 mmol, 87%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, *J* = 7.6 Hz, 2H), 7.48 (d, *J* = 8.0 Hz, 1H), 7.30 ~ 7.22 (m, 4H), 7.12 (d, *J* = 4.4 Hz, 2H), 7.04 ~ 6.95 (m, 3H), 6.83 (d, *J* = 7.6 Hz, 1H), 3.85 ~ 3.80 (m, 2H), 3.76 (d, *J* = 6.4 Hz, 1H), 3.35 (d, *J* = 6.8 Hz, 1H), 2.41 (s, 3H), 0.85 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.5, 153.0, 140.0, 134.5, 133.3, 131.6, 129.1, 128.1, 128.0 127.9, 127.7, 125.2, 123.1, 122.8, 120.0, 115.8, 94.6, 89.9, 60.9, 48.0, 46.7, 20.6, 13.8; one CH carbon merged with other peak; HRMS (EI-MS) m/z: [M]+ calcd. For $C_{26}H_{23}NO_2$: 381.1729; found: 381.1725.

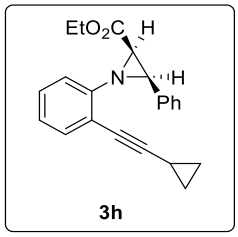
Spectral data of ethyl (2R,3R)-3-phenyl-1-(2-(thiophen-3-ylethynyl)phenyl)aziridine-2-carboxylate (3g):



3g

Compound **3g** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (56 mg, 0.15 mmol, 86%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.55 (d, *J* = 7.0 Hz, 2H), 7.45 ~ 7.44 (m, 1H), 7.31 ~ 7.21 (m, 4H), 7.13 ~ 7.11 (m, 1H), 7.01 ~ 6.96 (m, 3H), 6.78 (d, *J* = 4.2 Hz, 1H), 3.90 ~ 3.86 (m, 2H), 3.77 (d, *J* = 7.0 Hz, 1H), 3.31 (d, *J* = 6.3 Hz, 1H), 0.87 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.6, 153.2, 134.7, 133.0, 129.7, 129.1, 128.6, 128.0, 127.9, 127.8, 124.8, 123.1, 122.1, 119.9, 115.6, 90.9, 85.5, 60.9, 47.9, 46.7, 13.8; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{23}H_{19}NO_2S$: 373.1136; found: 373.1137.

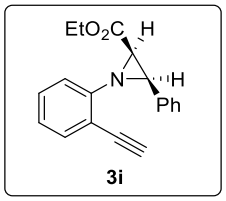
Spectral data of ethyl (2R,3R)-1-(2-(cyclopropylethynyl)phenyl)-3-phenylaziridine-2-carboxylate (3h):



3h

Compound **3h** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (59 mg, 0.18 mmol, 87%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (t, *J* = 6.8 Hz, 2H), 7.34 ~ 7.26 (m, 4H), 7.18 ~ 7.13 (m, 1H), 6.96 ~ 6.92 (m, 2H), 4.07 ~ 3.95 (m, 2H), 3.68 (d, *J* = 6.8 Hz, 1H), 3.25 (d, *J* = 6.8 Hz, 1H), 1.14 ~ 1.10 (m, 1H), 0.96 (t, *J* = 7.2 Hz, 3H), 0.63 ~ 0.60 (m, 2H), 0.49 ~ 0.45 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.7, 153.3, 134.7, 133.1, 127.8, 122.9, 119.7, 116.3, 99.9, 72.6, 60.9, 47.9, 46.6, 13.9, 08.1, 0.80, 0.14; two CH carbon merged with other peaks; HRMS (EI-MS) m/z: [M]+ calcd. For $C_{22}H_{21}NO_2$: 331.1572; found: 331.1571.

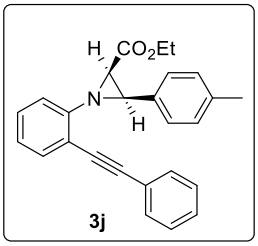
Spectral data of ethyl (2R,3R)-1-(2-ethynylphenyl)-3-phenylaziridine-2-carboxylate (3i):



3i

Compound **3i** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (30 mg, 0.10 mmol, 42%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.54 (d, *J* = 7.0 Hz, 2H), 7.43 (dd, *J* = 7.7, 0.7 Hz, 1H), 7.33 (t, *J* = 7.7 Hz, 2H), 7.29 ~ 7.25 (m, 2H), 7.00 ~ 6.98 (m, 2H), 4.08 ~ 4.03 (m, 1H), 3.99 ~ 3.95 (m, 1H), 3.73 (d, *J* = 7.0 Hz, 1H), 3.30 (d, *J* = 7.0 Hz, 1H), 3.16 (s, 1H), 0.99 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.7, 154.3, 134.5, 133.6, 129.7, 127.9, 127.8, 127.7, 123.0, 119.8, 114.5, 83.9, 80.4, 60.9, 47.8, 46.6, 13.9; HRMS (EI-MS) m/z: [M]+ calcd. For $C_{19}H_{17}NO_2$: 291.1259; found: 291.1255.

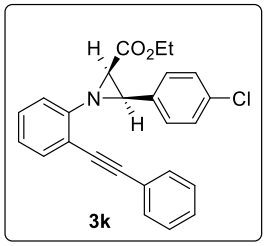
Spectral data of ethyl (2R,3R)-1-(2-(phenylethynyl)phenyl)-3-(p-tolyl)aziridine-2-carboxylate (3j):



3j

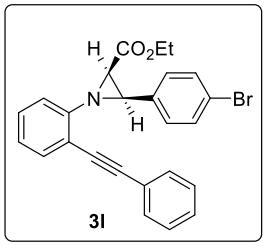
Compound **3j** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (46 mg, 0.12 mmol, 71%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48 ~ 7.42 (m, 3H), 7.25 ~ 7.15 (m, 4H), 7.11 ~ 7.07 (m, 4H), 7.03 ~ 6.99 (m, 2H), 3.90 ~ 3.85 (m, 2H), 3.72 (d, *J* = 6.8 Hz, 1H), 3.31 (d, *J* = 6.8 Hz, 1H), 2.32 (s, 3H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.6, 153.4, 137.5, 133.2, 131.5, 131.4, 129.2, 128.7, 128.0, 127.9, 127.8, 123.1, 123.0, 119.9, 115.6, 95.6, 86.1, 60.9, 48.0, 46.7, 21.2, 13.8; HRMS (EI-MS) m/z: [M]+ calcd. $C_{26}H_{23}NO_2$: 381.1729; found: 381.1725.

Spectral data of ethyl (2R,3R)-3-(4-chlorophenyl)-1-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate (3k):



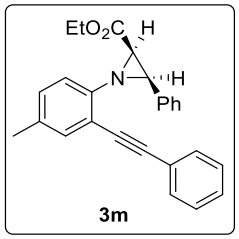
Compound **3k** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (52 mg, 0.13 mmol, 82%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.46 (d, *J* = 8.4 Hz, 2H), 7.44 (dd, *J* = 7.7 , 0.7 Hz, 1H), 7.22 ~ 7.18 (m, 4H), 7.16 (t, *J* = 7.7 Hz, 2H), 7.00 ~ 6.98 (m, 3H), 6.95 (d, *J* = 7.7 Hz, 1H), 3.86 (q, *J* = 7.0 Hz, 2H), 3.67 (d, *J* = 7.0 Hz, 1H), 3.29 (d, *J* = 7.0 Hz, 1H), 0.89 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.3, 153.0, 133.7, 133.3, 133.2, 131.3, 129.3, 129.2, 128.2, 128.1, 128.0, 123.3, 122.9, 119.8, 115.6, 95.7, 86.0, 61.1, 47.4, 46.7, 13.8; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{20}ClNO_2$: 401.1183; found: 401.1181.

Spectral data of ethyl (2R,3R)-3-(4-bromophenyl)-1-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate (3l):



Compound **3l** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow solid (49 mg, 0.11 mmol, 79%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.54 (d, *J* = 7.0 Hz, 2H), 7.46 (dd, *J* = 7.0, 0.7 Hz, 1H), 7.30 ~ 7.24 (m, 6H), 7.02 ~ 7.00 (m, 2H), 6.84 (d, *J* = 8.4 Hz, 2H), 3.92 ~ 3.87 (m, 2H), 3.76 (d, *J* = 7.0 Hz, 1H), 3.31 (d, *J* = 7.0 Hz, 1H), 0.89 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.5, 153.3, 134.6, 133.3, 132.8, 131.2, 129.4, 128.1, 127.9, 127.8, 123.1, 122.3, 122.0, 120.0, 115.2, 94.4, 87.3, 61.0, 47.9, 46.7, 13.8; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{20}BrNO_2$: 445.0677; found: 445.0677.

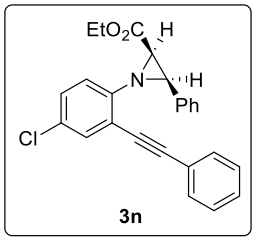
Spectral data of ethyl (2R,3R)-1-(4-methyl-2-(phenylethynyl)phenyl)-3-phenylaziridine-2-carboxylate (3m):



3m

Compound **3m** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (51mg, 0.13 mmol, 79%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.55 (d, *J* = 7.7 Hz, 2H), 7.29 (t, *J* = 7.0 Hz, 3H), 7.26 (d, *J* = 7.0 Hz, 1H), 7.20 (t, *J* = 7.0 Hz, 1H), 7.16 (t, *J* = 7.7 Hz, 2H), 7.07 ~ 7.03 (m, 3H), 6.90 (d, *J* = 8.4 Hz, 1H), 3.89 ~ 3.84 (m, 2H), 3.72 (d, *J* = 7.0 Hz, 1H), 3.29 (d, *J* = 7.0 Hz, 1H), 2.28 (s, 3H), 0.86 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.7, 151.0, 134.7, 133.6, 132.6, 131.4, 130.0, 128.0, 127.9, 127.9, 127.7, 123.1, 119.8, 115.3, 95.3, 86.2, 60.9, 48.1, 46.8, 20.6, 13.8; one CH carbon merged with other peaks; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{26}H_{23}NO_2$: 381.1729; found: 381.1726.

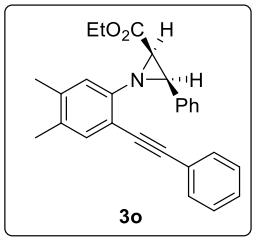
Spectral data of ethyl (2R,3R)-1-(4-chloro-2-(phenylethynyl)phenyl)-3-phenylaziridine-2-carboxylate (3n):



3n

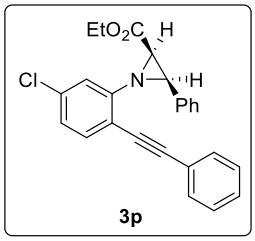
Compound **3n** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Brown oil (47 mg, 0.12 mmol, 74%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.53 (d, *J* = 7.0 Hz, 2H), 7.45 (d, *J* = 2.8 Hz, 1H), 7.31 ~ 7.26 (m, 3H), 7.23 (t, *J* = 7.7 Hz, 1H), 7.20 ~ 7.17 (m, 3H), 7.05 (d, *J* = 7.0 Hz, 2H), 6.94 (d, *J* = 8.4 Hz, 1H), 3.88 ~ 3.85 (m, 2H), 3.74 (d, *J* = 7.0 Hz, 1H), 3.31 (d, *J* = 7.0 Hz, 1H), 0.87 (t, *J* = 7.7 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.2, 151.9, 134.2, 132.6, 131.4, 129.1, 128.4, 128.1, 128.1,128.0, 127.9, 127.8, 122.5, 121.2, 117.1, 96.7, 84.7, 61.0, 48.1, 46.8, 13.8; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{20}ClNO_2$: 401.1183; found: 401.1177.

Spectral data of ethyl (2R,3R)-1-(4,5-dimethyl-2-(phenylethynyl)phenyl)-3-phenylaziridine-2-carboxylate (3o):



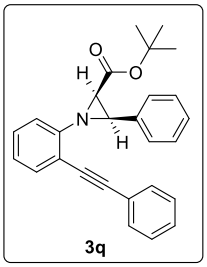
Compound **30** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Brown oil (45mg, 0.11 mmol, 70%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 7.2 Hz, 2H), 7.33 ~ 7.26 (m, 3H), 7.21 ~ 7.15 (m, 4H), 7.07 ~ 7.05 (m, 2H), 6.90 (d, *J* = 0.4 Hz, 1H), 3.88 ~ 3.82 (m, 2H), 3.81 (d, *J* = 6.8 Hz, 1H), 3.40 (d, *J* = 6.8 Hz, 1H), 2.24 (s, 3H), 2.22 (s, 3H), 0.86 (t, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.8, 148.9, 134.7, 132.4, 132.1, 131.8, 131.3, 129.8, 128.0, 127.9, 127.7, 123.3, 114.5, 95.1, 86.9, 60.8, 49.1, 48.4, 20.4, 18.7, 13.8; two CH carbon merged with other peaks; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{27}H_{25}NO_2$: 395.1885; found: 395.1889.

Spectral data of ethyl (2R,3R)-1-(5-chloro-2-(phenylethynyl)phenyl)-3-phenylaziridine-2-carboxylate (3p):



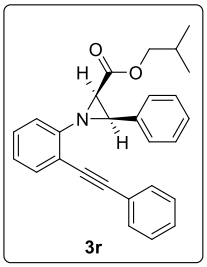
Compound **3p** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Brown oil (48 mg, 0.12 mmol, 75%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.53 (d, *J* = 7.0 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 1H), 7.31 ~ 7.26 (m, 3H), 7.22 (t, *J* = 7.7 Hz, 1H), 7.17 (t, *J* = 7.7 Hz, 2H), 7.06 (d, *J* = 7.0 Hz, 2H), 7.00 ~ 6.99 (m, 2H), 3.86 (q, *J* = 7.0 Hz, 2H), 3.78 (d, *J* = 7.0 Hz, 1H), 3.34 (d, *J* = 7.0 Hz, 1H), 0.86 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.1, 154.2, 134.7, 134.2, 134.1, 131.4, 128.3, 128.1, 128.0, 127.9, 127.8, 123.4, 122.7, 120.3, 114.2, 96.4, 85.1, 61.0, 48.0, 46.7, 13.8; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{20}ClNO_2$: 401.1183; found: 401.1186.

Spectral data of tert-butyl (2R,3R)-3-phenyl-1-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate (3q):



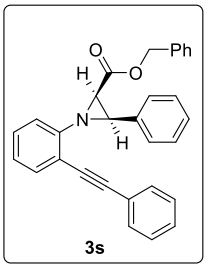
Compound **3q** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (57 mg, 0.14 mmol, 81%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.57 (d, *J* = 7.7 Hz, 2H), 7.48 (dd, *J* = 6.3, 1.4 Hz, 1H), 7.30 (t, *J* = 7.0 Hz, 2H), 7.27 ~ 7.23 (m, 2H), 7.19 (t, *J* = 7.0 Hz, 1H), 7.15 (t, *J* = 7.7 Hz, 2H), 7.03 ~ 7.00 (m, 4H), 3.74 (d, *J* = 7.0 Hz, 1H), 3.28 (d, *J* = 7.0 Hz, 1H), 1.09 (s, 9H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 166.6, 153.4, 134.9, 133.4, 131.4, 129.1, 128.0, 127.9, 127.9, 127.8, 127.5, 123.1, 122.8, 120.2, 115.4, 95.4, 86.1, 81.6, 47.6, 47.4, 27.6; HRMS (EI-MS) m/z: [M]+ calcd. For $C_{27}H_{25}NO_2$: 395.1885; found: 395.1888.

Spectral data of isobutyl (2R,3R)-3-phenyl-1-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate (3r):



Compound **3r** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Brown oil (58 mg, 0.15 mmol, 83%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.54 (d, *J* = 7.7 Hz, 2H), 7.47 (dd, *J* = 7.7, 0.7 Hz, 1H), 7.28 (t, *J* = 7.0 Hz, 2H), 7.25 ~ 7.19 (m, 3H), 7.15 (t, *J* = 5.6 Hz, 2H), 7.05 ~ 7.04 (m, 2H), 7.02 ~ 6.99 (m, 2H), 3.76 (d, *J* = 7.0 Hz, 1H), 3.64 (q, *J* = 7.0 Hz, 1H), 3.51 (q, *J* = 7.0 Hz, 1H), 3.34 (d, *J* = 7.0 Hz, 1H), 1.56 (m, 1H) 0.63 (d, *J* = 7.0 Hz, 3H), 0.60 (d, *J* = 7.0 Hz, 3H) ; ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.5, 153.4, 134.7, 133.3, 131.4, 129.2, 128.1, 128.0, 127.9, 127.8, 127.7, 123.1, 123.0, 119.9, 115.6, 95.6, 86.1, 71.0, 48.0, 46.9, 27.4, 18.8, 18.7; HRMS (EI-MS) m/z: [M]+ calcd. For $C_{27}H_{25}NO_2$: 395.1885; found: 395.1883.

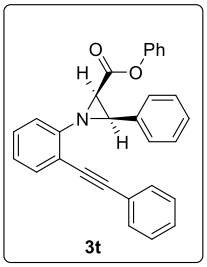
Spectral data of benzyl (2R,3R)-3-phenyl-1-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate (3s):



3s

Compound **3s** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Brown oil (64 mg, 0.15 mmol, 84%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.53 (m, 2H), 7.48 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.28 ~ 7.25 (m, 3H), 7.23 ~ 7.21 (m, 3H), 7.20 ~ 7.17 (m, 4H), 7.10 (d, *J* = 7.7 Hz, 2H), 7.04 ~ 7.00 (m, 2H), 6.91 (d, *J* = 7.0 Hz, 2H), 4.89 (d, *J* = 12.6 Hz, 1H), 4.75 (d, *J* = 12.6 Hz, 1H), 3.78 (d, *J* = 7.0 Hz, 1H), 3.39 (d, *J* = 7.0 Hz, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.4, 153.2, 135.2, 134.4, 133.3, 131.4, 129.2, 128.3, 128.1, 128.1,128.1 128.0, 127.9, 127.8, 127.8, 123.1, 123.0, 119.9, 115.6, 95.7, 86.1, 66.6, 48.1, 46.7; HRMS (EI-MS) m/z: [M]+ calcd. For $C_{30}H_{23}NO_2$: 429.1729; found: 429.1733.

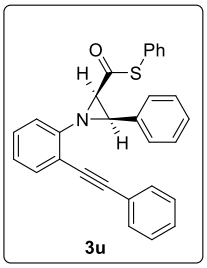
Spectral data of phenyl (2R,3R)-3-phenyl-1-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate (3t):



3t

Compound **3t** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Brown oil (48 mg, 0.12 mmol, 65%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.63 (d, *J* = 7.0 Hz, 2H), 7.51 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.35 ~ 7.31 (m, 3H), 7.28 ~ 7.26 (m, 1H), 7.24 ~ 7.21 (m, 1H), 7.17 ~ 7.12 (m, 4H), 7.11 ~ 7.06 (m, 5H), 6.32 (d, *J* = 7.7 Hz, 2H), 3.92 (d, *J* = 7.0 Hz, 1H), 3.58 (d, *J* = 7.0 Hz, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 166.3, 152.8, 150.1, 134.3, 133.4, 131.6, 129.3, 129.1, 128.3, 128.2, 128.1, 128.0, 127.9, 125.7, 123.3, 122.9, 121.3, 120.0, 115.6, 95.8, 86.0, 48.2, 46.6; HRMS (EI-MS) m/z: [M]+ calcd. For $C_{29}H_{21}NO_2$: 415.1572; found: 415.1562.

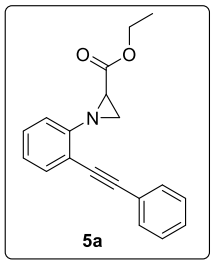
Spectral data of S-phenyl (2R,3R)-3-phenyl-1-(2-(phenylethynyl)phenyl)aziridine-2-carbothioate (3u):



3u

Compound **3u** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Brown oil (44 mg, 0.10 mmol, 58%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.60 (d, *J* = 7.0 Hz, 2H), 7.52 (d, *J* = 7.7 Hz, 1H), 7.34 (t, *J* = 7.0 Hz, 2H), 7.32 ~ 7.24 (m, 4H), 7.20 ~ 7.16 (m, 4H), 7.14 ~ 7.10 (m, 3H), 7.07 (t, *J* = 7.0 Hz, 1H), 6.79 (d, *J* = 7.7 Hz, 2H), 3.94 (d, *J* = 6.3 Hz, 1H), 3.61 (d, *J* = 6.3 Hz, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 194.4, 152.5, 134.4, 133.8, 133.6, 131.5, 129.3, 129.1, 128.9, 128.4, 128.2, 128.1, 128.0, 127.9, 127.5, 123.4, 123.1, 120.2, 115.6, 96.1, 86.1, 52.7, 49.3; HRMS (EI-MS) m/z: [M]+ calcd. For $C_{29}H_{21}NOS$: 431.1344; found: 431.1343.

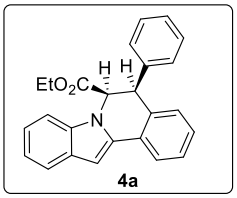
Spectral data of ethyl 1-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate (5a):



5a

Compound **5a** was purified on silica gel column using ethyl acetate/hexane: (15 : 85) as the eluent; Brown oil (26 mg, 0.12 mmol, 46%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.55 (dd, *J* = 7.7, 2.1 Hz, 2H), 7.47 (dd, *J* = 7.7, 0.7 Hz, 1H), 7.37 ~ 7.34 (m, 3H), 7.27 ~ 7.24 (m, 1H), 7.03 ~ 7.00 (m, 1H), 6.94 (d, *J* = 8.4 Hz, 1H), 4.13 ~ 4.09 (m, 1H), 3.98 ~ 3.96 (m, 1H), 2.94 (q, *J* = 7.0 Hz, 1H), 2.74 (q, *J* = 2.8 Hz, 1H), 2.50 (dd, *J* = 6.3, 1.4 Hz, 1H), 1.10 (t, *J* = 7.0 Hz, 3H) ; ^{13}C NMR (175 MHz, $CDCl_3$): δ 170.0, 153.2, 132.8, 131.6, 129.1, 128.3, 128.2, 123.3, 122.9, 119.9, 116.4, 95.6, 86.0, 61.3, 38.5, 34.2, 13.9; HRMS (ESI-TOF) m/z: [M+Na]+ calcd. for $C_{19}H_{17}NO_2Na$: 314.1157, found: 314.1157.

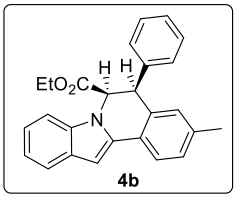
Spectral data of ethyl (5S,6R)-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4a):



4a

Compound **4a** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (54 mg, 0.15 mmol, 83%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.88 (d, *J* = 7.7 Hz, 1H), 7.64 (d, *J* = 7.7 Hz, 1H), 7.44 ~ 7.37 (m, 5H), 7.33 (t, *J* = 7.7 Hz, 1H), 7.27 (d, *J* = 7.7 Hz, 1H), 7.19 ~ 7.11 (m, 3H), 7.04 (d, *J* = 7.0 Hz, 1H), 7.00 (s, 1H), 5.29 (d, *J* = 7.0 Hz, 1H), 4.88 (d, *J* = 7.0 Hz, 1H), 3.89 ~ 3.84 (m, 1H), 3.77 ~ 3.72 (m, 1H), 0.88 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 168.9, 137.0, 136.5, 135.2, 131.7, 130.2, 129.2, 128.9, 128.8, 128.1, 127.6, 127.3, 127.3, 124.0, 122.2, 121.0, 120.7, 108.8, 98.1, 61.0, 59.4, 48.2, 13.6; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{21}NO_2$: 367.1572; found: 367.1577.

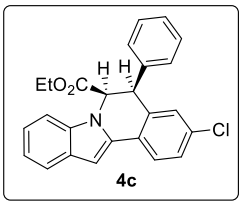
Spectral data of ethyl (5S,6R)-3-methyl-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4b):



4b

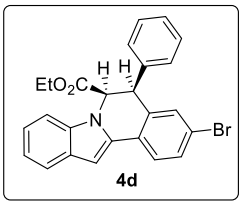
Compound **4b** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (52 mg, 0.14 mmol, 81%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, *J* = 8.0 Hz, 1H), 7.61 (d, *J* = 7.6 Hz, 1H), 7.43 ~ 7.36 (m, 5H), 7.25 (d, *J* = 7.2 Hz, 1H), 7.17 ~ 7.10 (m, 3H), 6.94 (s, 1H), 6.84 (s, 1H), 5.25 (d, *J* = 6.8 Hz, 1H), 4.85 (d, *J* = 6.8 Hz, 1H), 3.88 ~ 3.84 (m, 1H), 3.79 ~ 3.72 (m, 1H), 2.25 (s, 3H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 168.9, 137.3, 137.2, 136.4, 135.5, 131.6, 129.3, 128.8, 128.2, 128.1, 128.0, 126.2, 124.0, 121.9, 120.8, 120.6, 108.8, 97.3, 60.9, 59.4, 48.3, 21.5, 13.7; one CH carbon merged with other peaks HRMS (EI-MS) m/z: [M]+ calcd. for $C_{26}H_{23}NO_2$: 381.1729; found: 381.1727.

Spectral data of ethyl (5S,6R)-3-chloro-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4c):



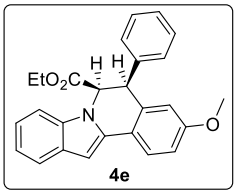
Compound **4c** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (49 mg, 0.12 mmol, 77%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (d, *J* = 8.4 Hz, 1H), 7.63 (d, *J* = 7.0 Hz, 1H), 7.45 ~ 7.41 (m, 3H), 7.36 ~ 7.34 (m, 2H), 7.30 ~ 7.25 (m, 2H), 7.21 ~ 7.17 (m, 1H), 7.13 ~ 7.11 (m, 1H), 7.02 (d, *J* = 0.8 Hz, 1H), 6.97 (s, 1H), 5.26 (d, *J* = 6.8 Hz, 1H), 4.82 (d, *J* = 6.4 Hz, 1H), 3.91 ~ 3.80 (m, 1H), 3.78 ~ 3.74 (m, 1H), 0.89 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 168.7, 136.5, 136.2, 134.2, 133.6, 133.0, 130.0, 129.1, 128.4, 127.7, 127.6, 127.5, 125.2, 122.6, 121.1, 120.9, 108.9, 98.5, 61.1, 59.2, 48.2, 13.7; one quaternary carbon merged with other peaks; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{20}ClNO_2$: 401.1183; found: 401.1182.

Spectral data of ethyl (5S,6R)-3-bromo-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4d):



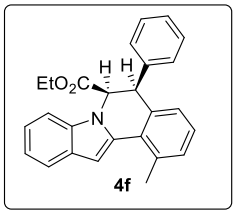
Compound **4d** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; off white solid (51 mg, 0.11 mmol, 82%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.72 (d, *J* = 8.4 Hz, 1H), 7.63 (d, *J* = 7.0 Hz, 1H), 7.46 ~ 7.41 (m, 4H), 7.35 (d, *J* = 7.0 Hz, 2H), 7.26 (d, *J* = 8.4 Hz, 1H), 7.21 ~ 7.18 (m, 2H), 7.13 (d, *J* = 7.7 Hz, 1H), 6.98 (s, 1H), 5.26 (d, *J* = 7.0 Hz, 1H), 4.82 (d, *J* = 7.0 Hz, 1H), 3.90 ~ 3.85 (m, 1H), 3.79 ~ 3.74 (m, 1H), 0.89 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 168.7, 136.5, 136.2, 134.2, 133.9, 130.5, 130.5, 129.1, 128.4, 127.9, 125.4, 122.6, 121.1, 121.0, 120.9, 108.9, 98.6, 61.1, 59.2, 48.1, 13.7; one quaternary carbon and one CH carbon merged with other peaks; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{20}BrNO_2$: 445.0677; found: 445.0678.

Spectral data of ethyl (5S,6R)-3-methoxy-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4e):



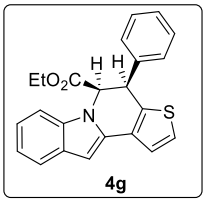
Compound **4e** was purified on silica gel column using ethyl acetate/hexane: (7 : 93) as the eluent; Yellow oil (44 mg, 0.11 mmol, 69%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.79 (d, *J* = 8.4 Hz, 1H), 7.59 (d, *J* = 7.7 Hz, 1H), 7.43 ~ 7.37 (m, 5H), 7.25 ~ 7.23 (m, 1H), 7.15 ~ 7.09 (m, 2H), 6.88 ~ 6.86 (m, 2H), 6.58 (d, *J* = 1.4 Hz, 1H), 5.25 (d, *J* = 7.0 Hz, 1H), 4.84 (d, *J* = 7.0 Hz, 1H), 3.88 ~ 3.85 (m, 1H), 3.77 ~ 3.74 (m, 1H), 3.70 (s, 3H), 0.88 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 168.9, 159.1, 136.9, 136.3, 135.4, 133.6, 130.2, 129.4, 128.8, 128.2, 125.4, 122.0, 121.7, 120.6, 120.5, 113.8, 112.6, 108.7, 96.5, 61.0, 59.4, 55.2, 48.4, 13.7; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{26}H_{23}NO_3$: 397.1678; found: 397.1677.

Spectral data of ethyl (5S,6R)-1-methyl-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4f):



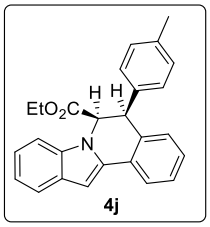
Compound **4f** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (52 mg, 0.14 mmol, 80%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.75 (d, *J* = 7.7 Hz, 1H), 7.62 ~ 7.60 (m, 1H), 7.23 ~ 7.21 (m, 1H), 7.11 (t, *J* = 2.8 Hz, 3H), 7.08 ~ 7.04 (m, 5H), 7.01 (d, *J* = 7.0 Hz, 1H), 6.93 (d, *J* = 8.4 Hz, 1H), 5.26 (d, *J* = 5.6 Hz, 1H), 4.74 (d, *J* = 5.6 Hz, 1H), 3.97 ~ 3.93 (m, 1H), 3.76 ~ 3.72 (m, 1H), 2.18 (s, 3H), 0.85 (t, *J* = 7.7 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 169.0, 138.1, 137.5, 136.2, 136.1, 131.4, 130.0, 129.9, 129.4, 128.2, 127.6, 127.3, 122.4, 121.6, 120.6, 120.4, 111.3, 98.5, 61.4, 60.9, 45.4, 19.5, 13.5; one quaternary carbon merged with other peaks; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{26}H_{23}NO_2$: 381.1729; found: 381.1734.

Spectral data of ethyl (4R,5R)-4-phenyl-4,5-dihydrothieno[3',2':3,4]pyrido[1,2-a]indole-5-carboxylate (4g):



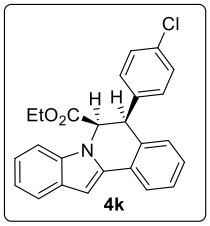
Compound **4g** was purified on silica gel column using ethyl acetate/hexane: (7 : 93) as the eluent; Yellow oil (29 mg, 0.08 mmol, 44%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 7.6 Hz, 1H), 7.41 ~ 7.37 (m, 5H), 7.07 ~ 7.15 (m, 4H), 6.71 (d, *J* = 4.8 Hz, 2H), 5.28 (d, *J* = 8.0 Hz, 1H), 4.87 (d, *J* = 7.6 Hz, 1H), 3.81 ~ 3.73 (m, 1H), 3.63 ~ 3.55 (m, 1H), 0.79 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 168.4, 137.5, 136.6, 133.1, 132.2, 129.8, 129.5, 128.7, 128.1, 127.3, 123.2, 122.4, 120.9, 128.8, 128.7, 108.7, 97.7, 60.8, 60.2, 45.9, 13.5; one quaternary carbon merged with other peaks; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{23}H_{19}NO_2S$: 373.1136; found: 373.1140.

Spectral data of ethyl (5S,6R)-5-(p-tolyl)-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4j):



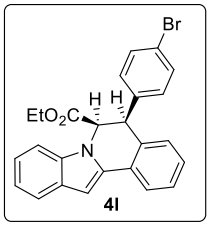
Compound **4j** was purified on silica gel column using ethyl acetate/hexane: 5 : 95) as the eluent; Yellow oil (44 mg, 0.12 mmol, 68%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.88 (d, *J* = 7.7 Hz, 1H), 7.65 (d, *J* = 7.7 Hz, 1H), 7.33 (s, *J* = 7.7 Hz, 1H), 7.29 ~ 7.24 (m, 5H), 7.20 ~ 7.13 (m, 3H), 7.06 (d, *J* = 7.7 Hz, 1H), 7.00 (s, 1H), 5.28 (d, *J* = 7.0 Hz, 1H), 4.86 (d, *J* = 7.0 Hz, 1H), 3.91 ~ 3.89 (m, 1H), 3.79 ~ 3.77 (m, 1H), 2.42 (s, 3H), 0.90 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 168.9, 137.8, 136.4, 135.3, 133.9, 132.0, 130.1, 129.5, 129.2, 128.9, 127.6, 127.3, 127.2, 123.9, 122.2, 120.9, 120.7, 108.9, 98.0, 61.0, 59.5, 47.9, 21.2, 13.7; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{26}H_{23}NO_2$: 381.1729; found: 381.1728.

Spectral data of ethyl (5S,6R)-5-(4-chlorophenyl)-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4k):



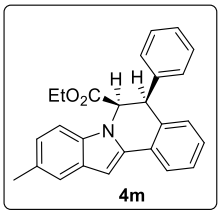
Compound **4k** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (50 mg, 0.12 mmol, 79%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, *J* = 7.6 Hz, 1H), 7.63 (d, *J* = 8.0 Hz, 1H), 7.42 ~ 7.39 (m, 2H), 7.35 ~ 7.24 (m, 4H), 7.20 ~ 7.11 (m, 3H), 6.99 ~ 6.97 (m, 2H), 5.25 (d, *J* = 6.8 Hz, 1H), 4.86 (d, *J* = 6.8 Hz, 1H), 3.91 ~ 3.84 (m, 1H), 3.78 ~ 3.73 (m, 1H), 0.91 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.7, 136.5, 135.7, 135.1, 134.1, 131.6, 131.2, 129.2, 129.0, 128.8, 127.5, 127.4, 124.1, 122.3, 121.0, 120.8, 108.8, 98.3, 61.1, 59.3, 47.6, 13.7; one CH carbon merged with other peaks; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{20}ClNO_2$: 401.1183; found: 401.1181.

Spectral data of ethyl (5S,6R)-5-(4-bromophenyl)-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4l):



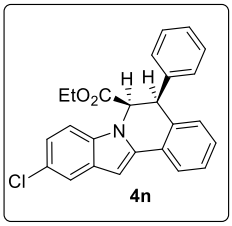
Compound **4l** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (47 mg, 0.11 mmol, 76%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.72 (d, *J* = 8.4 Hz, 1H), 7.63 (d, *J* = 7.7 Hz, 1H), 7.44 ~ 7.43 (m, 4H), 7.34 (d, *J* = 5.6 Hz, 2H), 7.25 (t, *J* = 7.7 Hz, 1H), 7.19 ~ 7.11 (m, 3H), 6.98 (s, 1H), 5.24 (d, *J* = 6.3 Hz, 1H), 4.83 (d, *J* = 6.3 Hz, 1H), 3.88 ~ 3.85 (m, 1H), 3.78 ~ 3.75 (m, 1H), 0.89 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 168.7, 136.5, 136.2, 134.3, 133.9, 130.5, 130.1, 129.1, 128.5, 127.9, 125.5, 122.6, 121.1, 120.9, 108.9, 98.6, 61.2, 59.2, 48.1, 13.7; one quaternary and two CH carbon merged with other peaks; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{20}BrNO_2$: 445.0677; found: 445.0674.

Spectral data of ethyl (5S,6R)-10-methyl-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4m):



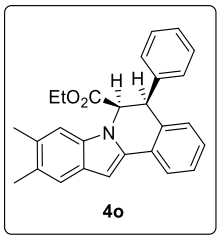
Compound **4m** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (49 mg, 0.13 mmol, 76%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.85 (d, *J* = 7.7 Hz, 1H), 7.42 ~ 7.36 (m, 6H), 7.30 (t, *J* = 7.7 Hz, 1H), 7.16 ~ 7.12 (m, 2H), 7.03 ~ 6.99 (m, 2H), 6.91 (s, 1H), 5.24 (d, *J* = 7.0 Hz, 1H), 4.87 (d, *J* = 7.0 Hz, 1H), 3.87 ~ 3.83 (m, 1H), 3.75 ~ 3.70 (m, 1H), 2.43 (s, 3H), 0.87 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 168.9, 137.2, 135.3, 134.9, 131.7, 130.2, 130.0, 129.5, 129.0, 128.8, 128.1, 127.6, 127.3, 127.1, 123.9, 123.8, 120.6, 108.5, 97.6, 60.9, 59.5, 48.2, 21.4, 13.7; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{26}H_{23}NO_2$: 381.1729; found: 381.1726.

Spectral data of ethyl (5S,6R)-10-chloro-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4n):



Compound **4n** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (44 mg, 0.11 mmol, 69%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.85 (d, *J* = 7.7 Hz, 1H), 7.59 (s, 1H), 7.43 ~ 7.35 (m, 5H), 7.33 (t, *J* = 7.7 Hz, 1H), 7.18 ~ 7.16 (m, 2H), 7.12 (d, *J* = 8.4 Hz, 1H), 7.04 (d, *J* = 7.7 Hz, 1H), 6.92 (s, 1H), 5.23 (d, *J* = 7.0 Hz, 1H), 4.88 (d, *J* = 7.0 Hz, 1H), 3.89 ~ 3.84 (m, 1H), 3.76 ~ 3.72 (m, 1H), 0.87 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 168.6, 136.8, 136.6, 134.9, 131.8, 130.3, 130.2, 128.9, 128.4, 128.2, 127.8, 127.7, 127.4, 126.3, 124.1, 122.4, 120.3, 109.8, 97.6, 61.1, 59.5, 48.1, 13.7; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{20}ClNO_2$: 401.1183; found: 401.1184.

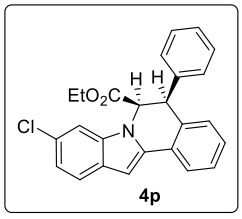
Spectral data of ethyl (5S,6R)-9,10-dimethyl-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4o):



4o

Compound **4o** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (40 mg, 0.10 mmol, 62%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.85 (d, *J* = 7.7 Hz, 1H), 7.41 ~ 7.28 (m, 6H), 7.24 (d, *J* = 9.1 Hz, 1H), 7.11 (t, *J* = 7.7 Hz, 1H), 6.97 (t, *J* = 7.7 Hz, 1H), 6.93 (s, 1H), 6.71 (s, 1H), 5.64 (d, *J* = 6.3 Hz, 1H), 4.92 (d, *J* = 6.3 Hz, 1H), 3.89 ~ 3.84 (m, 1H), 3.71 ~ 3.67 (m, 1H), 2.63 (s, 3H), 2.36 (s, 3H), 0.85 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 169.4, 137.4, 136.2, 134.0, 131.2, 130.3, 130.0, 129.4, 128.1, 127.4, 127.3, 127.2, 126.9, 123.7, 120.4, 118.6, 98.9, 61.5, 60.8, 49.1, 21.1, 19.9, 13.7; two CH carbon merged with other peaks; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{27}H_{25}NO_2$: 395.1885; found: 395.1889.

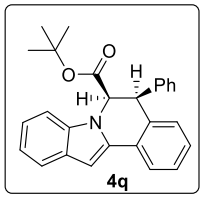
Spectral data of ethyl (5S,6R)-10-chloro-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4p):



4p

Compound **4p** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (46 mg, 0.11 mmol, 72%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.84 (d, *J* = 8.0 Hz, 1H), 7.52 (d, *J* = 8.5 Hz, 1H), 7.44 ~ 7.30 (m, 6H), 7.25 (d, *J* = 6.0 Hz, 1H), 7.18 ~ 7.15 (m, 1H), 7.08 ~ 7.04 (m, 2H), 6.94 (s, 1H), 5.20 (d, *J* = 7.0 Hz, 1H), 4.87 (d, *J* = 7.0 Hz, 1H), 3.91 ~ 3.85 (m, 1H), 3.79 ~ 3.72 (m, 1H), 0.88 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.6, 136.9, 136.8, 136.1, 131.7, 130.2, 128.8, 128.5, 128.2, 128.1, 127.8, 127.7, 127.6, 127.4, 124.0, 121.7, 121.4, 109.0, 98.0, 61.2, 59.5, 48.2, 13.6; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{25}H_{20}ClNO_2$: 401.1183; found: 401.1185.

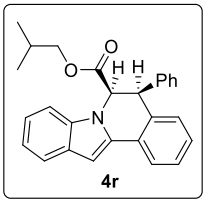
Spectral data of tert-butyl (5S,6R)-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4q):



4q

Compound **4q** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (55 mg, 0.14 mmol, 78%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.85 (d, *J* = 7.7 Hz, 1H), 7.62 (d, *J* = 7.7 Hz, 1H), 7.44 ~ 7.39 (m, 5H), 7.31 ~ 7.28 (m, 2H), 7.18 ~ 7.09 (m, 3H), 7.04 (d, *J* = 8.4 Hz, 1H), 6.98 (s, 1H), 5.16 (d, *J* = 7.0 Hz, 1H), 4.83 (d, *J* = 6.3 Hz, 1H), 1.07 (s, 9H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.8, 137.0, 136.5, 135.3, 132.1, 130.6, 129.2, 128.9, 128.8, 128.0, 127.4, 127.2, 127.1, 123.9, 122.1, 120.9, 120.6, 109.0, 98.0, 82.2, 60.5, 48.1, 27.7; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{27}H_{25}NO_2$: 395.1885; found: 395.1880.

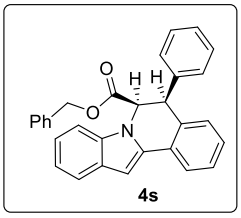
Spectral data of isobutyl (5S,6R)-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4r):



4r

Compound **4r** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (53 mg, 0.13 mmol, 75%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.87 (d, *J* = 7.7 Hz, 1H), 7.63 (d, *J* = 7.7 Hz, 1H), 7.43 ~ 7.35 (m, 5H), 7.32 ~ 7.28 (m, 2H), 7.19 ~ 7.11 (m, 3H), 7.03 (d, *J* = 7.7 Hz, 1H), 6.99 (s, 1H), 5.30 (d, *J* = 7.0 Hz, 1H), 4.88 (d, *J* = 7.0 Hz, 1H), 3.61 (q, *J* = 6.3 Hz, 1H), 3.45 (q, *J* = 6.3 Hz, 1H), 1.54 ~ 1.49 (m, 1H), 0.53 (d, *J* = 7.0 Hz, 6H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 168.8, 136.9, 136.5, 135.2, 132.0, 130.1, 129.2, 128.9, 128.9, 128.2, 127.6, 127.3, 127.2, 123.9, 122.3, 120.9, 120.7, 108.9, 98.1, 71.2, 59.8, 48.2, 27.1, 18.6; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{27}H_{25}NO_2$: 395.1885; found: 395.1882.

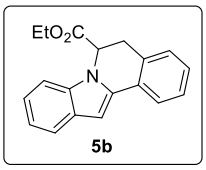
Spectral data of benzyl (5S,6R)-5-phenyl-5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (4s):



4s

Compound **4s** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (62 mg, 0.14 mmol, 81%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.87 (d, *J* = 7.7 Hz, 1H), 7.62 (d, *J* = 7.7 Hz, 1H), 7.34 ~ 7.32 (m, 4H), 7.26 ~ 7.19 (m, 4H), 7.16 ~ 7.09 (m, 5H), 7.00 ~ 6.99 (m, 2H), 6.19 (d, *J* = 7.0 Hz, 2H), 5.33 (d, *J* = 7.0 Hz, 1H), 4.88 (t, *J* = 6.3 Hz, 2H), 5.66 (d, *J* = 12.6 Hz, 1H) ; ^{13}C NMR (175 MHz, $CDCl_3$): δ 168.6, 136.8, 136.5, 135.2, 134.8, 131.8, 130.1, 130.0, 129.2, 128.9, 128.3, 128.2, 128.0, 127.8, 127.7, 127.4, 124.1, 122.3, 121.0, 120.8, 109.0, 98.2, 66.6, 59.7, 48.3; one CH carbon merged with other peaks; HRMS (EI-MS) m/z: [M]+ calcd. for $C_{30}H_{23}NO_2$: 429.1729; found: 429.1726

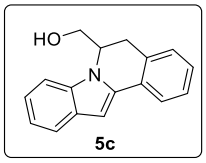
Spectral data of ethyl 5,6-dihydroindolo[2,1-a]isoquinoline-6-carboxylate (5b):



5b

Compound **5b** was purified on silica gel column using ethyl acetate/hexane: (5 : 95) as the eluent; Yellow oil (52 mg, 0.18 mmol, 76%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, *J* = 7.6 Hz, 1H), 7.61 (d, *J* = 8.0 Hz, 1H), 7.38 ~ 7.35 (m, 3H), 7.29 ~ 7.25 (m, 1H), 7.22 ~ 7.19 (m, 1H), 7.09 (t, *J* = 7.2 Hz, 1H), 6.86 (s, 1H), 4.82 (dd, *J* = 12.4, 4.0 Hz, 1H), 4.22 (dd, *J* = 12.4, 4.8 Hz, 1H), 4.10 ~ 4.02 (m, 3H), 1.10 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 171.2, 136.9, 134.7, 129.4, 129.2, 128.8, 128.7, 128.4, 127.5, 124.6, 122.0, 120.8, 120.0, 109.0, 97.1, 61.5, 45.1, 42.0, 14.0; HRMS (ESI-TOF) m/z: [M+Na]+ calcd. for $C_{19}H_{17}NO_2Na$: 314.1157, found: 314.1163.

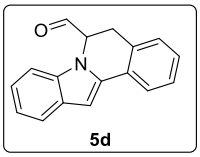
Spectral data of (5,6-dihydroindolo[2,1-a]isoquinolin-6-yl)methanol (5c):



5c

Compound **5c** was purified on silica gel column using ethyl acetate/hexane: (30 : 70) as the eluent; Yellow oil (36 mg, 0.14 mmol, 84%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, *J* = 8.0 Hz, 1H), 7.63 ~ 7.61 (m, 1H), 7.38 ~ 7.24 (m, 4H), 7.23 ~ 7.18 (m, 1H), 7.11 ~ 7.07 (m, 1H), 6.86 (s, 1H), 4.66 (dd, *J* = 12.4, 2.0 Hz, 1H), 4.07 (dd, *J* = 12.4, 4.4 Hz, 1H), 3.65 (q, *J* = 6.4 Hz, 1H), 3.55 (q, *J* = 9.2 Hz, 1H), 3.35 ~ 3.32 (m, 1H), 1.24 (broad s, 1H), ; ^{13}C NMR (175 MHz, $CDCl_3$): δ 137.1, 134.8, 132.3, 128.9, 128.8, 128.7, 127.9, 127.6, 124.6, 121.8, 120.7, 119.9, 109.0, 96.7, 63.7, 41.8, 40.6 ; HRMS (ESI-TOF) m/z: [M+H]+ calcd. for $C_{17}H_{15}NO$: 250.1232, found: 250.1229.

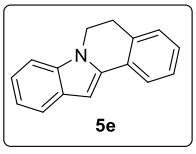
Spectral data of 5,6-dihydroindolo[2,1-a]isoquinoline-6-carbaldehyde (5d):



5d

Compound **5d** was purified on silica gel column using ethyl acetate/hexane: (20 : 80) as the eluent; Yellow oil (29 mg, 0.12 mmol, 68%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 9.62 (s, 1H), 7.83 (d, *J* = 8.4 Hz, 1H), 7.61 (d, *J* = 7.7 Hz, 1H), 7.43 ~ 7.39 (m, 3H), 7.34 (t, *J* = 7.0 Hz, 1H), 7.24 (t, *J* = 6.3 Hz, 1H), 7.12 (t, *J* = 7.7 Hz, 1H), 6.89 (s, 1H), 4.98 (dd, *J* = 11.9, 2.1 Hz, 1H), 4.17 (dd, *J* = 12.6, 4.9 Hz, 1H), 3.86 (d, *J* = 4.2 Hz, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 198.5, 137.1, 134.1, 129.3, 129.3, 128.9, 128.8, 128.0, 126.8, 124.8, 122.3, 120.9, 120.3, 109.1, 97.6, 51.8, 39.3 ; HRMS (ESI-TOF) m/z: [M+H]+ calcd. for $C_{17}H_{14}NO$: 248.1075, found: 248.1077.

Spectral data of 5,6-dihydroindolo[2,1-a]isoquinoline (5e):

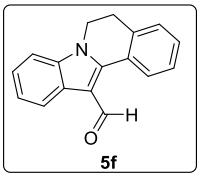


5e

Compound **5e** was purified on silica gel column using ethyl acetate/hexane: (05 : 95) as the eluent; white solid (38 mg, 0.17 mmol, 85%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.78 (d, *J* = 7.7 Hz, 1H),

7.66 (d, *J* = 7.7 Hz, 1H), 7.36 ~ 7.32 (m, 2H), 7.29 ~ 7.22 (m, 3H), 7.13 (t, *J* = 7.7 Hz, 1H), 6.90 (s, 1H), 4.28 (t, *J* = 7.0 Hz, 2H), 3.22 (t, *J* = 7.0 Hz, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 136.6, 135.6, 132.1, 129.0, 128.8, 128.3, 127.4, 127.2, 124.4, 121.6, 120.7, 119.9, 108.9, 96.4, 40.1, 29.1 ; ; HRMS (ESI-TOF) m/z: [M+Na]+ calcd. for $C_{16}H_{13}N$: 242.0946 found: 242.0945.

Spectral data of 5,6-dihydroindolo[2,1-a]isoquinoline-12-carbaldehyde (5f):



5f

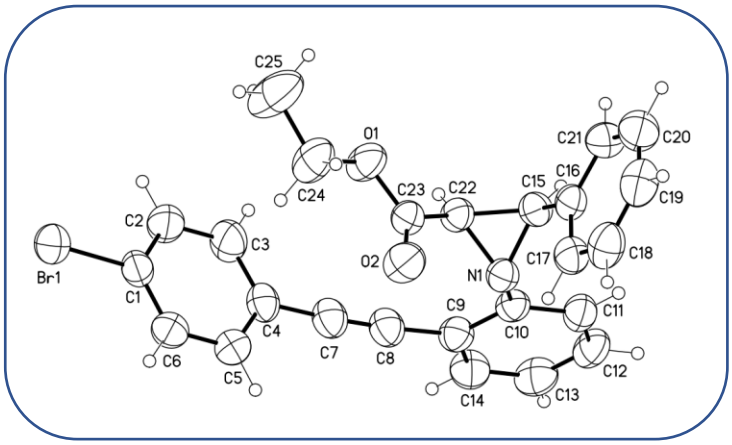
Compound **5f** was purified on silica gel column using ethyl acetate/hexane: (20 : 80) as the eluent; Yellow oil (46 mg, 0.18 mmol, 81%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 10.50 (s, 1H), 8.44 (d, *J* = 7.7 Hz, 1H), 7.96 ~ 7.94 (m, 1H), 7.42 ~ 7.36 (m, 4H), 7.34 ~ 7.29 (m, 2H), 4.24 (t, *J* = 6.3 Hz, 2H), 3.17 (t, *J* = 6.3 Hz, 2H) ; ^{13}C NMR (175 MHz, $CDCl_3$): δ 185.5, 142.9, 135.7, 134.9, 129.9, 129.0, 128.5, 127.8, 127.1, 126.7, 124.0, 123.2, 122.3, 113.4, 109.1, 40.1, 29.2 ; HRMS (ESI-TOF) m/z: [M+Na]+ calcd. for $C_{17}H_{13}NONa$: 270.0895, found: 270.0895.

4. X-ray crystallographic structure and data for compound 3d and 4d:

(a) X-ray crystallographic data of compound (3d).

Ellipsoid contour % probability level = 50%

Experimental: The sample was dissolved in appropriate amount of methanol followed by the addition of pentane to furnish a saturated solution. Afterwards, the mixture was allowed to stand at room temperature to form the crystals.



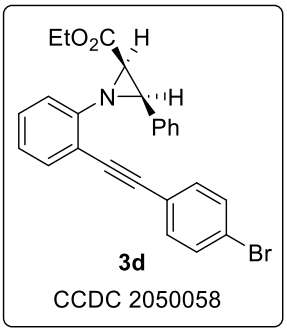


Table 1. Crystal data and structure refinement for 201138lt_0m_a.

Identification code	201138LT_0m_a	
Empirical formula	C25 H20 Br N O2	
Formula weight	446.33	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.0729(4) Å	α= 94.217(2)°.
	b = 11.4073(5) Å	β= 90.628(2)°.
	c = 18.7007(7) Å	γ = 100.546(2)°.
Volume	2106.13(15) $Å^3$	
Z	4	

Density (calculated)	1.408 Mg/m^3
Absorption coefficient	1.972 mm^{-1}
F(000)	912
Crystal size	0.10 x 0.06 x 0.01 mm^3
Theta range for data collection	1.092 to 26.439°.
Index ranges	-12<=h<=12, -14<=k<=14, -23<=l<=23
Reflections collected	36201
Independent reflections	8663 [R(int) = 0.0270]
Completeness to theta = 25.242°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7454 and 0.6763
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8663 / 0 / 525
Goodness-of-fit on F^2	1.029
Final R indices [I>2sigma(I)]	R1 = 0.0358, wR2 = 0.0885
R indices (all data)	R1 = 0.0591, wR2 = 0.0986
Extinction coefficient	n/a
Largest diff. peak and hole	0.386 and -0.301 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 201138lt_0m_a. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	5910(1)	8312(1)	-1121(1)	65(1)
Br(2)	10863(1)	8456(1)	6483(1)	80(1)
C(1)	5963(2)	8337(2)	-104(1)	49(1)
C(2)	4830(2)	8485(2)	269(1)	63(1)
C(3)	4860(3)	8474(2)	1007(1)	64(1)
C(4)	6014(2)	8316(2)	1374(1)	49(1)
C(5)	7147(2)	8185(2)	982(1)	54(1)
C(6)	7118(2)	8197(2)	245(1)	54(1)
C(7)	6007(2)	8258(2)	2140(1)	55(1)
C(8)	5939(2)	8154(2)	2771(1)	54(1)
C(9)	5786(2)	7961(2)	3515(1)	48(1)
C(10)	5318(2)	6813(2)	3729(1)	43(1)
C(11)	5174(2)	6645(2)	4455(1)	51(1)
C(12)	5500(2)	7595(2)	4965(1)	57(1)
C(13)	5961(3)	8726(2)	4760(1)	61(1)
C(14)	6093(3)	8903(2)	4048(1)	58(1)
C(15)	3908(2)	4894(2)	3228(1)	45(1)
C(16)	4050(2)	3620(2)	3150(1)	46(1)
C(17)	5278(2)	3260(2)	3038(1)	54(1)
C(18)	5337(3)	2051(3)	2979(1)	65(1)
C(19)	4175(3)	1205(2)	3023(1)	67(1)
C(20)	2970(3)	1560(2)	3133(1)	66(1)
C(21)	2900(2)	2755(2)	3199(1)	56(1)
C(22)	4124(2)	5741(2)	2636(1)	44(1)
C(23)	4581(2)	5370(2)	1913(1)	46(1)
C(24)	3985(3)	5114(3)	688(1)	70(1)
C(25)	2804(3)	5121(4)	225(2)	104(1)
C(26)	10913(2)	8377(2)	5469(1)	57(1)
C(27)	12080(3)	8232(2)	5136(1)	61(1)
C(28)	12132(3)	8196(2)	4396(1)	60(1)
C(29)	11011(2)	8305(2)	3987(1)	53(1)

C(30)	9833(3)	8434(3)	4338(2)	72(1)
C(31)	9782(3)	8472(3)	5075(2)	74(1)
C(32)	11052(3)	8259(2)	3218(1)	56(1)
C(33)	11026(2)	8186(2)	2580(1)	53(1)
C(34)	10909(2)	8041(2)	1809(1)	46(1)
C(35)	11267(2)	9008(2)	1391(1)	57(1)
C(36)	11143(3)	8870(2)	658(2)	63(1)
C(37)	10640(2)	7753(2)	318(1)	58(1)
C(38)	10275(2)	6790(2)	720(1)	48(1)
C(39)	10406(2)	6912(2)	1461(1)	40(1)
C(40)	8935(2)	5011(2)	1740(1)	43(1)
C(41)	9039(2)	3728(2)	1691(1)	42(1)
C(42)	7865(2)	2881(2)	1571(1)	51(1)
C(43)	7910(3)	1678(2)	1529(1)	59(1)
C(44)	9124(3)	1308(2)	1599(1)	58(1)
C(45)	10296(3)	2142(2)	1712(1)	58(1)
C(46)	10257(2)	3346(2)	1758(1)	51(1)
C(47)	9167(2)	5818(2)	2425(1)	43(1)
C(48)	9598(2)	5388(2)	3109(1)	47(1)
C(49)	8970(3)	5060(3)	4290(1)	85(1)
C(50)	7835(4)	5085(4)	4738(2)	114(1)
N(1)	5108(2)	5823(2)	3219(1)	43(1)
N(2)	10150(2)	5904(2)	1864(1)	40(1)
O(1)	3632(2)	5411(2)	1424(1)	60(1)
O(2)	5633(2)	5080(2)	1777(1)	64(1)
O(3)	8641(2)	5411(2)	3584(1)	67(1)
O(4)	10633(2)	5074(2)	3226(1)	68(1)

Table 3. Bond lengths [Å] and angles [°] for 201138lt_0m_a.

Br(1)-C(1)	1.900(2)
Br(2)-C(26)	1.892(2)
C(1)-C(6)	1.368(3)
C(1)-C(2)	1.374(3)
C(2)-C(3)	1.381(3)
C(2)-H(2)	0.9500
C(3)-C(4)	1.390(3)
C(3)-H(3)	0.9500
C(4)-C(5)	1.387(3)
C(4)-C(7)	1.440(3)
C(5)-C(6)	1.379(3)
C(5)-H(5)	0.9500
C(6)-H(6)	0.9500
C(7)-C(8)	1.196(3)
C(8)-C(9)	1.430(3)
C(9)-C(10)	1.398(3)
C(9)-C(14)	1.400(3)
C(10)-C(11)	1.391(3)
C(10)-N(1)	1.408(3)
C(11)-C(12)	1.378(3)
C(11)-H(11)	0.9500
C(12)-C(13)	1.374(4)
C(12)-H(12)	0.9500
C(13)-C(14)	1.365(3)
C(13)-H(13)	0.9500
C(14)-H(14)	0.9500
C(15)-N(1)	1.455(3)
C(15)-C(16)	1.484(3)
C(15)-C(22)	1.514(3)
C(15)-H(15)	1.0000
C(16)-C(21)	1.386(3)
C(16)-C(17)	1.386(3)
C(17)-C(18)	1.387(3)
C(17)-H(17)	0.9500

C(18)-C(19)	1.381(4)
C(18)-H(18)	0.9500
C(19)-C(20)	1.361(4)
C(19)-H(19)	0.9500
C(20)-C(21)	1.374(4)
C(20)-H(20)	0.9500
C(21)-H(21)	0.9500
C(22)-N(1)	1.451(3)
C(22)-C(23)	1.491(3)
C(22)-H(22)	1.0000
C(23)-O(2)	1.191(3)
C(23)-O(1)	1.324(3)
C(24)-O(1)	1.456(3)
C(24)-C(25)	1.465(4)
C(24)-H(24A)	0.9900
C(24)-H(24B)	0.9900
C(25)-H(25A)	0.9800
C(25)-H(25B)	0.9800
C(25)-H(25C)	0.9800
C(26)-C(27)	1.367(3)
C(26)-C(31)	1.375(4)
C(27)-C(28)	1.383(3)
C(27)-H(27)	0.9500
C(28)-C(29)	1.387(3)
C(28)-H(28)	0.9500
C(29)-C(30)	1.389(4)
C(29)-C(32)	1.436(3)
C(30)-C(31)	1.379(4)
C(30)-H(30)	0.9500
C(31)-H(31)	0.9500
C(32)-C(33)	1.190(3)
C(33)-C(34)	1.439(3)
C(34)-C(35)	1.395(3)
C(34)-C(39)	1.404(3)
C(35)-C(36)	1.371(4)
C(35)-H(35)	0.9500

C(36)-C(37)	1.386(4)
C(36)-H(36)	0.9500
C(37)-C(38)	1.374(3)
C(37)-H(37)	0.9500
C(38)-C(39)	1.384(3)
C(38)-H(38)	0.9500
C(39)-N(2)	1.407(3)
C(40)-N(2)	1.446(3)
C(40)-C(41)	1.482(3)
C(40)-C(47)	1.512(3)
C(40)-H(40)	1.0000
C(41)-C(46)	1.383(3)
C(41)-C(42)	1.388(3)
C(42)-C(43)	1.377(3)
C(42)-H(42)	0.9500
C(43)-C(44)	1.373(4)
C(43)-H(43)	0.9500
C(44)-C(45)	1.377(3)
C(44)-H(44)	0.9500
C(45)-C(46)	1.378(3)
C(45)-H(45)	0.9500
C(46)-H(46)	0.9500
C(47)-N(2)	1.447(3)
C(47)-C(48)	1.491(3)
C(47)-H(47)	1.0000
C(48)-O(4)	1.185(3)
C(48)-O(3)	1.320(3)
C(49)-C(50)	1.429(4)
C(49)-O(3)	1.460(3)
C(49)-H(49A)	0.9900
C(49)-H(49B)	0.9900
C(50)-H(50A)	0.9800
C(50)-H(50B)	0.9800
C(50)-H(50C)	0.9800

C(6)-C(1)-C(2)	121.1(2)

C(6)-C(1)-Br(1)	119.76(17)
C(2)-C(1)-Br(1)	119.17(18)
C(1)-C(2)-C(3)	119.1(2)
C(1)-C(2)-H(2)	120.5
C(3)-C(2)-H(2)	120.5
C(2)-C(3)-C(4)	121.0(2)
C(2)-C(3)-H(3)	119.5
C(4)-C(3)-H(3)	119.5
C(5)-C(4)-C(3)	118.5(2)
C(5)-C(4)-C(7)	121.4(2)
C(3)-C(4)-C(7)	120.1(2)
C(6)-C(5)-C(4)	120.6(2)
C(6)-C(5)-H(5)	119.7
C(4)-C(5)-H(5)	119.7
C(1)-C(6)-C(5)	119.8(2)
C(1)-C(6)-H(6)	120.1
C(5)-C(6)-H(6)	120.1
C(8)-C(7)-C(4)	176.1(3)
C(7)-C(8)-C(9)	176.1(3)
C(10)-C(9)-C(14)	118.0(2)
C(10)-C(9)-C(8)	120.4(2)
C(14)-C(9)-C(8)	121.6(2)
C(11)-C(10)-C(9)	119.5(2)
C(11)-C(10)-N(1)	120.2(2)
C(9)-C(10)-N(1)	120.05(18)
C(12)-C(11)-C(10)	120.8(2)
C(12)-C(11)-H(11)	119.6
C(10)-C(11)-H(11)	119.6
C(13)-C(12)-C(11)	120.2(2)
C(13)-C(12)-H(12)	119.9
C(11)-C(12)-H(12)	119.9
C(14)-C(13)-C(12)	119.5(2)
C(14)-C(13)-H(13)	120.2
C(12)-C(13)-H(13)	120.2
C(13)-C(14)-C(9)	122.0(2)
C(13)-C(14)-H(14)	119.0

C(9)-C(14)-H(14)	119.0
N(1)-C(15)-C(16)	119.40(18)
N(1)-C(15)-C(22)	58.46(13)
C(16)-C(15)-C(22)	124.86(17)
N(1)-C(15)-H(15)	114.2
C(16)-C(15)-H(15)	114.2
C(22)-C(15)-H(15)	114.2
C(21)-C(16)-C(17)	118.9(2)
C(21)-C(16)-C(15)	118.2(2)
C(17)-C(16)-C(15)	123.0(2)
C(16)-C(17)-C(18)	119.8(2)
C(16)-C(17)-H(17)	120.1
C(18)-C(17)-H(17)	120.1
C(19)-C(18)-C(17)	120.3(2)
C(19)-C(18)-H(18)	119.8
C(17)-C(18)-H(18)	119.8
C(20)-C(19)-C(18)	119.8(2)
C(20)-C(19)-H(19)	120.1
C(18)-C(19)-H(19)	120.1
C(19)-C(20)-C(21)	120.5(3)
C(19)-C(20)-H(20)	119.7
C(21)-C(20)-H(20)	119.7
C(20)-C(21)-C(16)	120.7(2)
C(20)-C(21)-H(21)	119.6
C(16)-C(21)-H(21)	119.6
N(1)-C(22)-C(23)	115.63(18)
N(1)-C(22)-C(15)	58.74(13)
C(23)-C(22)-C(15)	121.97(19)
N(1)-C(22)-H(22)	116.0
C(23)-C(22)-H(22)	116.0
C(15)-C(22)-H(22)	116.0
O(2)-C(23)-O(1)	123.7(2)
O(2)-C(23)-C(22)	126.5(2)
O(1)-C(23)-C(22)	109.71(18)
O(1)-C(24)-C(25)	107.9(2)
O(1)-C(24)-H(24A)	110.1

C(25)-C(24)-H(24A)	110.1
O(1)-C(24)-H(24B)	110.1
C(25)-C(24)-H(24B)	110.1
H(24A)-C(24)-H(24B)	108.4
C(24)-C(25)-H(25A)	109.5
C(24)-C(25)-H(25B)	109.5
H(25A)-C(25)-H(25B)	109.5
C(24)-C(25)-H(25C)	109.5
H(25A)-C(25)-H(25C)	109.5
H(25B)-C(25)-H(25C)	109.5
C(27)-C(26)-C(31)	120.6(2)
C(27)-C(26)-Br(2)	119.47(19)
C(31)-C(26)-Br(2)	119.90(19)
C(26)-C(27)-C(28)	119.8(2)
C(26)-C(27)-H(27)	120.1
C(28)-C(27)-H(27)	120.1
C(27)-C(28)-C(29)	120.8(2)
C(27)-C(28)-H(28)	119.6
C(29)-C(28)-H(28)	119.6
C(28)-C(29)-C(30)	118.3(2)
C(28)-C(29)-C(32)	121.1(2)
C(30)-C(29)-C(32)	120.5(2)
C(31)-C(30)-C(29)	120.9(2)
C(31)-C(30)-H(30)	119.5
C(29)-C(30)-H(30)	119.5
C(26)-C(31)-C(30)	119.6(2)
C(26)-C(31)-H(31)	120.2
C(30)-C(31)-H(31)	120.2
C(33)-C(32)-C(29)	176.8(3)
C(32)-C(33)-C(34)	176.1(3)
C(35)-C(34)-C(39)	118.4(2)
C(35)-C(34)-C(33)	121.5(2)
C(39)-C(34)-C(33)	120.2(2)
C(36)-C(35)-C(34)	121.4(2)
C(36)-C(35)-H(35)	119.3
C(34)-C(35)-H(35)	119.3

C(35)-C(36)-C(37)	119.9(2)
C(35)-C(36)-H(36)	120.0
C(37)-C(36)-H(36)	120.0
C(38)-C(37)-C(36)	119.6(2)
C(38)-C(37)-H(37)	120.2
C(36)-C(37)-H(37)	120.2
C(37)-C(38)-C(39)	121.2(2)
C(37)-C(38)-H(38)	119.4
C(39)-C(38)-H(38)	119.4
C(38)-C(39)-C(34)	119.6(2)
C(38)-C(39)-N(2)	120.76(19)
C(34)-C(39)-N(2)	119.47(18)
N(2)-C(40)-C(41)	119.08(17)
N(2)-C(40)-C(47)	58.51(12)
C(41)-C(40)-C(47)	123.94(18)
N(2)-C(40)-H(40)	114.6
C(41)-C(40)-H(40)	114.6
C(47)-C(40)-H(40)	114.6
C(46)-C(41)-C(42)	119.0(2)
C(46)-C(41)-C(40)	122.56(19)
C(42)-C(41)-C(40)	118.49(19)
C(43)-C(42)-C(41)	120.6(2)
C(43)-C(42)-H(42)	119.7
C(41)-C(42)-H(42)	119.7
C(44)-C(43)-C(42)	120.0(2)
C(44)-C(43)-H(43)	120.0
C(42)-C(43)-H(43)	120.0
C(43)-C(44)-C(45)	119.8(2)
C(43)-C(44)-H(44)	120.1
C(45)-C(44)-H(44)	120.1
C(44)-C(45)-C(46)	120.4(2)
C(44)-C(45)-H(45)	119.8
C(46)-C(45)-H(45)	119.8
C(45)-C(46)-C(41)	120.2(2)
C(45)-C(46)-H(46)	119.9
C(41)-C(46)-H(46)	119.9

N(2)-C(47)-C(48)	115.72(17)
N(2)-C(47)-C(40)	58.48(12)
C(48)-C(47)-C(40)	121.80(18)
N(2)-C(47)-H(47)	116.1
C(48)-C(47)-H(47)	116.1
C(40)-C(47)-H(47)	116.1
O(4)-C(48)-O(3)	123.9(2)
O(4)-C(48)-C(47)	126.6(2)
O(3)-C(48)-C(47)	109.44(19)
C(50)-C(49)-O(3)	108.6(2)
C(50)-C(49)-H(49A)	110.0
O(3)-C(49)-H(49A)	110.0
C(50)-C(49)-H(49B)	110.0
O(3)-C(49)-H(49B)	110.0
H(49A)-C(49)-H(49B)	108.3
C(49)-C(50)-H(50A)	109.5
C(49)-C(50)-H(50B)	109.5
H(50A)-C(50)-H(50B)	109.5
C(49)-C(50)-H(50C)	109.5
H(50A)-C(50)-H(50C)	109.5
H(50B)-C(50)-H(50C)	109.5
C(10)-N(1)-C(22)	120.50(17)
C(10)-N(1)-C(15)	121.10(16)
C(22)-N(1)-C(15)	62.80(13)
C(39)-N(2)-C(40)	121.14(16)
C(39)-N(2)-C(47)	121.10(16)
C(40)-N(2)-C(47)	63.02(13)
C(23)-O(1)-C(24)	114.90(18)
C(48)-O(3)-C(49)	114.67(19)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 201138lt_0m_a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	63(1)	81(1)	47(1)	11(1)	3(1)	4(1)
Br(2)	71(1)	113(1)	53(1)	7(1)	7(1)	8(1)
C(1)	52(1)	48(1)	44(1)	9(1)	4(1)	3(1)
C(2)	49(1)	84(2)	59(2)	19(1)	6(1)	19(1)
C(3)	57(2)	85(2)	56(1)	17(1)	17(1)	24(1)
C(4)	60(1)	39(1)	48(1)	7(1)	5(1)	6(1)
C(5)	49(1)	58(2)	54(1)	10(1)	-2(1)	10(1)
C(6)	45(1)	64(2)	55(1)	7(1)	7(1)	12(1)
C(7)	64(2)	46(1)	54(1)	7(1)	4(1)	4(1)
C(8)	60(1)	47(1)	53(1)	6(1)	4(1)	4(1)
C(9)	48(1)	51(1)	44(1)	2(1)	0(1)	9(1)
C(10)	39(1)	50(1)	41(1)	2(1)	0(1)	10(1)
C(11)	54(1)	57(1)	43(1)	8(1)	1(1)	13(1)
C(12)	60(2)	74(2)	38(1)	0(1)	-3(1)	17(1)
C(13)	63(2)	64(2)	53(1)	-13(1)	-6(1)	9(1)
C(14)	63(2)	49(1)	58(1)	-2(1)	-1(1)	6(1)
C(15)	43(1)	53(1)	40(1)	3(1)	6(1)	9(1)
C(16)	51(1)	51(1)	34(1)	4(1)	1(1)	8(1)
C(17)	52(1)	63(2)	49(1)	10(1)	5(1)	12(1)
C(18)	77(2)	72(2)	54(1)	10(1)	7(1)	32(2)
C(19)	95(2)	52(2)	53(1)	1(1)	-4(1)	17(2)
C(20)	73(2)	54(2)	66(2)	3(1)	-7(1)	-3(1)
C(21)	51(1)	57(2)	57(1)	2(1)	-3(1)	4(1)
C(22)	44(1)	49(1)	41(1)	3(1)	1(1)	11(1)
C(23)	47(1)	48(1)	42(1)	4(1)	0(1)	6(1)
C(24)	77(2)	92(2)	43(1)	-6(1)	-2(1)	30(2)
C(25)	86(2)	169(4)	62(2)	-17(2)	-18(2)	49(2)
C(26)	55(1)	61(2)	50(1)	0(1)	4(1)	3(1)
C(27)	52(1)	74(2)	56(1)	-3(1)	-5(1)	8(1)
C(28)	52(1)	67(2)	59(2)	-7(1)	4(1)	6(1)
C(29)	58(2)	42(1)	55(1)	-6(1)	-3(1)	3(1)

C(30)	61(2)	93(2)	62(2)	-3(2)	-9(1)	20(2)
C(31)	56(2)	102(2)	64(2)	-2(2)	4(1)	21(2)
C(32)	65(2)	43(1)	55(2)	-5(1)	-4(1)	1(1)
C(33)	55(1)	40(1)	60(2)	-2(1)	-1(1)	2(1)
C(34)	42(1)	44(1)	53(1)	4(1)	3(1)	8(1)
C(35)	58(1)	43(1)	70(2)	10(1)	9(1)	8(1)
C(36)	60(2)	58(2)	76(2)	29(1)	13(1)	12(1)
C(37)	56(1)	75(2)	47(1)	16(1)	10(1)	17(1)
C(38)	47(1)	52(1)	46(1)	3(1)	4(1)	11(1)
C(39)	35(1)	44(1)	45(1)	6(1)	3(1)	11(1)
C(40)	41(1)	45(1)	43(1)	4(1)	-2(1)	6(1)
C(41)	45(1)	41(1)	38(1)	2(1)	0(1)	5(1)
C(42)	44(1)	49(1)	60(1)	2(1)	2(1)	7(1)
C(43)	54(1)	46(1)	71(2)	2(1)	4(1)	-4(1)
C(44)	73(2)	41(1)	62(2)	4(1)	3(1)	12(1)
C(45)	58(2)	54(2)	65(2)	0(1)	-1(1)	20(1)
C(46)	45(1)	51(1)	55(1)	0(1)	-3(1)	6(1)
C(47)	42(1)	43(1)	43(1)	2(1)	0(1)	9(1)
C(48)	50(1)	46(1)	45(1)	3(1)	2(1)	7(1)
C(49)	91(2)	120(3)	54(2)	33(2)	15(2)	34(2)
C(50)	97(3)	184(4)	79(2)	53(2)	32(2)	57(3)
N(1)	45(1)	46(1)	38(1)	2(1)	2(1)	6(1)
N(2)	41(1)	38(1)	41(1)	3(1)	0(1)	4(1)
O(1)	55(1)	86(1)	41(1)	-3(1)	-2(1)	21(1)
O(2)	56(1)	88(1)	52(1)	-1(1)	7(1)	26(1)
O(3)	60(1)	97(1)	50(1)	21(1)	10(1)	24(1)
O(4)	61(1)	91(1)	59(1)	17(1)	-2(1)	28(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 201138lt_0m_a.

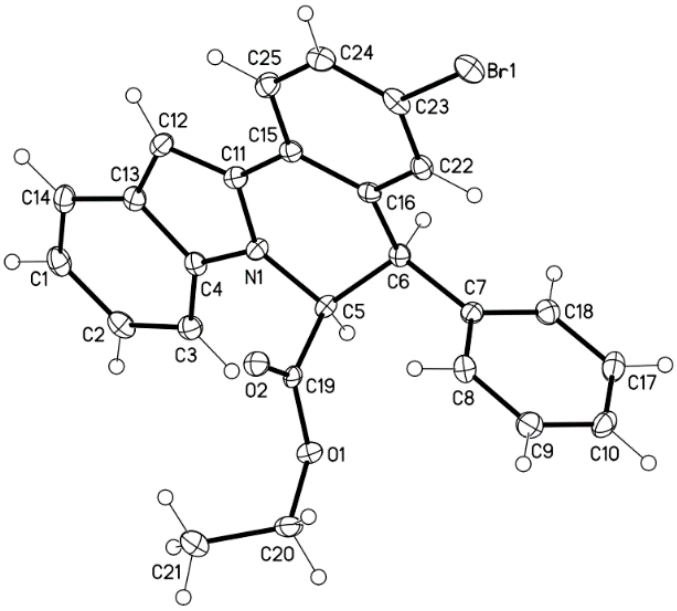
	x	y	z	U(eq)
H(2)	4037	8594	22	75
H(3)	4080	8576	1268	77
H(5)	7949	8087	1224	64
H(6)	7898	8108	-19	65
H(11)	4848	5867	4602	61
H(12)	5405	7466	5459	68
H(13)	6186	9381	5110	73
H(14)	6404	9690	3910	69
H(15)	3228	5073	3586	55
H(17)	6077	3839	3001	65
H(18)	6180	1804	2909	78
H(19)	4217	377	2976	80
H(20)	2172	977	3165	79
H(21)	2053	2990	3280	67
H(22)	3559	6377	2653	53
H(24A)	4240	4314	645	84
H(24B)	4761	5708	545	84
H(25A)	2045	4523	368	156
H(25B)	3019	4929	-275	156
H(25C)	2558	5915	273	156
H(27)	12853	8155	5412	73
H(28)	12944	8097	4166	72
H(30)	9051	8498	4065	86
H(31)	8971	8562	5310	89
H(35)	11604	9778	1620	69
H(36)	11400	9539	383	76
H(37)	10549	7654	-190	70
H(38)	9927	6026	486	58
H(40)	8268	5222	1394	51
H(42)	7023	3132	1519	62

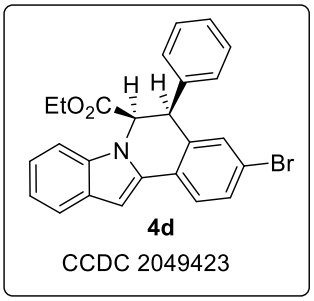
H(43)	7101	1104	1450	71
H(44)	9156	479	1571	70
H(45)	11136	1886	1757	69
H(46)	11070	3916	1837	61
H(47)	8627	6471	2473	51
H(49A)	9184	4245	4246	102
H(49B)	9770	5618	4502	102
H(50A)	7643	5898	4788	171
H(50B)	8037	4837	5212	171
H(50C)	7046	4536	4522	171

(b) X-ray crystallographic data of compound (4d).

Ellipsoid contour % probability level = 50%

Experimental: The sample was dissolved in appropriate amount of methanol followed by the addition of pentane to furnish a saturated solution. Afterwards, the mixture was allowed to stand at room temperature to form the crystals.





.Table 1. Crystal data and structure refinement for 201206lt_0m.

Identification code	201206LT_0m	
Empirical formula	C25 H20 Br N O2	
Formula weight	446.33	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 8.1912(2) Å	α= 90°.
	b = 9.0033(2) Å	β= 90.4360(10)°.

	c = 26.7875(6) Å γ = 90°.
Volume	1975.46(8) $Å^3$
Z	4
Density (calculated)	1.501 Mg/m^3
Absorption coefficient	2.103 mm^{-1}
F(000)	912
Crystal size	0.20 x 0.18 x 0.15 mm^3
Theta range for data collection	1.520 to 26.360°.
Index ranges	-10<=h<=10, -11<=k<=9, -32<=l<=33
Reflections collected	17645
Independent reflections	4041 [R(int) = 0.0235]
Completeness to theta = 25.242°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7454 and 0.6495
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4041 / 0 / 263
Goodness-of-fit on F^2	1.072
Final R indices [I>2sigma(I)]	R1 = 0.0232, wR2 = 0.0628
R indices (all data)	R1 = 0.0263, wR2 = 0.0642
Extinction coefficient	n/a
Largest diff. peak and hole	0.345 and -0.407 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 201206lt_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	13177(1)	180(1)	332(1)	19(1)
O(1)	5923(1)	1332(1)	2275(1)	15(1)
O(2)	7905(1)	2692(1)	1912(1)	16(1)
N(1)	5585(2)	2562(1)	1083(1)	12(1)
C(1)	1740(2)	5500(2)	1062(1)	18(1)
C(2)	1585(2)	4270(2)	1382(1)	17(1)
C(3)	2819(2)	3226(2)	1424(1)	15(1)
C(4)	4199(2)	3428(2)	1128(1)	13(1)
C(5)	6002(2)	1306(2)	1397(1)	12(1)
C(6)	7168(2)	257(2)	1111(1)	12(1)
C(7)	7717(2)	-1044(2)	1433(1)	13(1)
C(8)	8775(2)	-865(2)	1839(1)	16(1)
C(9)	9157(2)	-2056(2)	2148(1)	18(1)
C(10)	8523(2)	-3453(2)	2046(1)	19(1)
C(11)	6622(2)	3204(2)	739(1)	13(1)
C(12)	5905(2)	4468(2)	556(1)	15(1)
C(13)	4368(2)	4642(2)	798(1)	14(1)
C(14)	3100(2)	5700(2)	773(1)	17(1)
C(15)	8189(2)	2493(2)	640(1)	12(1)
C(16)	8522(2)	1086(2)	846(1)	12(1)
C(17)	7518(2)	-3658(2)	1633(1)	19(1)
C(18)	7111(2)	-2457(2)	1332(1)	16(1)
C(19)	6739(2)	1872(2)	1888(1)	12(1)
C(20)	6579(2)	1665(2)	2772(1)	18(1)
C(21)	6234(2)	3246(2)	2923(1)	24(1)
C(22)	10013(2)	412(2)	753(1)	14(1)
C(23)	11138(2)	1126(2)	451(1)	16(1)
C(24)	10825(2)	2488(2)	237(1)	17(1)
C(25)	9343(2)	3174(2)	337(1)	16(1)

Table 3. Bond lengths [Å] and angles [°] for 201206lt_0m.

Br(1)-C(23)	1.9038(15)
O(1)-C(19)	1.3310(19)
O(1)-C(20)	1.4626(19)
O(2)-C(19)	1.2086(19)
N(1)-C(4)	1.383(2)
N(1)-C(11)	1.385(2)
N(1)-C(5)	1.4475(19)
C(1)-C(14)	1.373(2)
C(1)-C(2)	1.406(2)
C(1)-H(1)	0.9500
C(2)-C(3)	1.384(2)
C(2)-H(20)	0.9500
C(3)-C(4)	1.398(2)
C(3)-H(3)	0.9500
C(4)-C(13)	1.414(2)
C(5)-C(19)	1.530(2)
C(5)-C(6)	1.550(2)
C(5)-H(16)	1.0000
C(6)-C(16)	1.517(2)
C(6)-C(7)	1.521(2)
C(6)-H(6)	1.0000
C(7)-C(18)	1.391(2)
C(7)-C(8)	1.395(2)
C(8)-C(9)	1.388(2)
C(8)-H(10)	0.9500
C(9)-C(10)	1.387(2)
C(9)-H(9)	0.9500
C(10)-C(17)	1.386(2)
C(10)-H(2)	0.9500
C(11)-C(12)	1.370(2)
C(11)-C(15)	1.461(2)
C(12)-C(13)	1.430(2)
C(12)-H(5)	0.9500
C(13)-C(14)	1.410(2)

C(14)-H(4)	0.9500
C(15)-C(25)	1.393(2)
C(15)-C(16)	1.408(2)
C(16)-C(22)	1.388(2)
C(17)-C(18)	1.388(2)
C(17)-H(8)	0.9500
C(18)-H(7)	0.9500
C(20)-C(21)	1.507(2)
C(20)-H(12)	0.9900
C(20)-H(11)	0.9900
C(21)-H(15)	0.9800
C(21)-H(13)	0.9800
C(21)-H(14)	0.9800
C(22)-C(23)	1.388(2)
C(22)-H(19)	0.9500
C(23)-C(24)	1.377(2)
C(24)-C(25)	1.390(2)
C(24)-H(18)	0.9500
C(25)-H(17)	0.9500

C(19)-O(1)-C(20)	116.85(12)
C(4)-N(1)-C(11)	109.30(13)
C(4)-N(1)-C(5)	125.45(13)
C(11)-N(1)-C(5)	124.68(13)
C(14)-C(1)-C(2)	121.61(15)
C(14)-C(1)-H(1)	119.2
C(2)-C(1)-H(1)	119.2
C(3)-C(2)-C(1)	121.03(15)
C(3)-C(2)-H(20)	119.5
C(1)-C(2)-H(20)	119.5
C(2)-C(3)-C(4)	117.29(15)
C(2)-C(3)-H(3)	121.4
C(4)-C(3)-H(3)	121.4
N(1)-C(4)-C(3)	130.09(14)
N(1)-C(4)-C(13)	107.30(13)
C(3)-C(4)-C(13)	122.60(14)

N(1)-C(5)-C(19)	109.14(12)
N(1)-C(5)-C(6)	109.45(12)
C(19)-C(5)-C(6)	112.73(12)
N(1)-C(5)-H(16)	108.5
C(19)-C(5)-H(16)	108.5
C(6)-C(5)-H(16)	108.5
C(16)-C(6)-C(7)	115.48(13)
C(16)-C(6)-C(5)	112.76(12)
C(7)-C(6)-C(5)	111.69(13)
C(16)-C(6)-H(6)	105.3
C(7)-C(6)-H(6)	105.3
C(5)-C(6)-H(6)	105.3
C(18)-C(7)-C(8)	118.41(15)
C(18)-C(7)-C(6)	119.39(14)
C(8)-C(7)-C(6)	122.17(14)
C(9)-C(8)-C(7)	120.71(15)
C(9)-C(8)-H(10)	119.6
C(7)-C(8)-H(10)	119.6
C(10)-C(9)-C(8)	120.10(16)
C(10)-C(9)-H(9)	120.0
C(8)-C(9)-H(9)	120.0
C(17)-C(10)-C(9)	119.77(15)
C(17)-C(10)-H(2)	120.1
C(9)-C(10)-H(2)	120.1
C(12)-C(11)-N(1)	108.81(14)
C(12)-C(11)-C(15)	132.32(15)
N(1)-C(11)-C(15)	118.87(14)
C(11)-C(12)-C(13)	107.73(14)
C(11)-C(12)-H(5)	126.1
C(13)-C(12)-H(5)	126.1
C(14)-C(13)-C(4)	118.46(15)
C(14)-C(13)-C(12)	134.69(16)
C(4)-C(13)-C(12)	106.85(14)
C(1)-C(14)-C(13)	118.98(16)
C(1)-C(14)-H(4)	120.5
C(13)-C(14)-H(4)	120.5

C(25)-C(15)-C(16)	119.63(14)
C(25)-C(15)-C(11)	120.97(14)
C(16)-C(15)-C(11)	119.39(14)
C(22)-C(16)-C(15)	119.37(14)
C(22)-C(16)-C(6)	121.11(14)
C(15)-C(16)-C(6)	119.07(13)
C(10)-C(17)-C(18)	119.89(15)
C(10)-C(17)-H(8)	120.1
C(18)-C(17)-H(8)	120.1
C(17)-C(18)-C(7)	121.06(15)
C(17)-C(18)-H(7)	119.5
C(7)-C(18)-H(7)	119.5
O(2)-C(19)-O(1)	125.56(15)
O(2)-C(19)-C(5)	123.90(14)
O(1)-C(19)-C(5)	110.54(13)
O(1)-C(20)-C(21)	111.69(13)
O(1)-C(20)-H(12)	109.3
C(21)-C(20)-H(12)	109.3
O(1)-C(20)-H(11)	109.3
C(21)-C(20)-H(11)	109.3
H(12)-C(20)-H(11)	107.9
C(20)-C(21)-H(15)	109.5
C(20)-C(21)-H(13)	109.5
H(15)-C(21)-H(13)	109.5
C(20)-C(21)-H(14)	109.5
H(15)-C(21)-H(14)	109.5
H(13)-C(21)-H(14)	109.5
C(23)-C(22)-C(16)	119.45(15)
C(23)-C(22)-H(19)	120.3
C(16)-C(22)-H(19)	120.3
C(24)-C(23)-C(22)	122.19(15)
C(24)-C(23)-Br(1)	119.31(12)
C(22)-C(23)-Br(1)	118.51(12)
C(23)-C(24)-C(25)	118.37(15)
C(23)-C(24)-H(18)	120.8
C(25)-C(24)-H(18)	120.8

C(24)-C(25)-C(15)	120.97(15)
C(24)-C(25)-H(17)	119.5
C(15)-C(25)-H(17)	119.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 201206lt_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

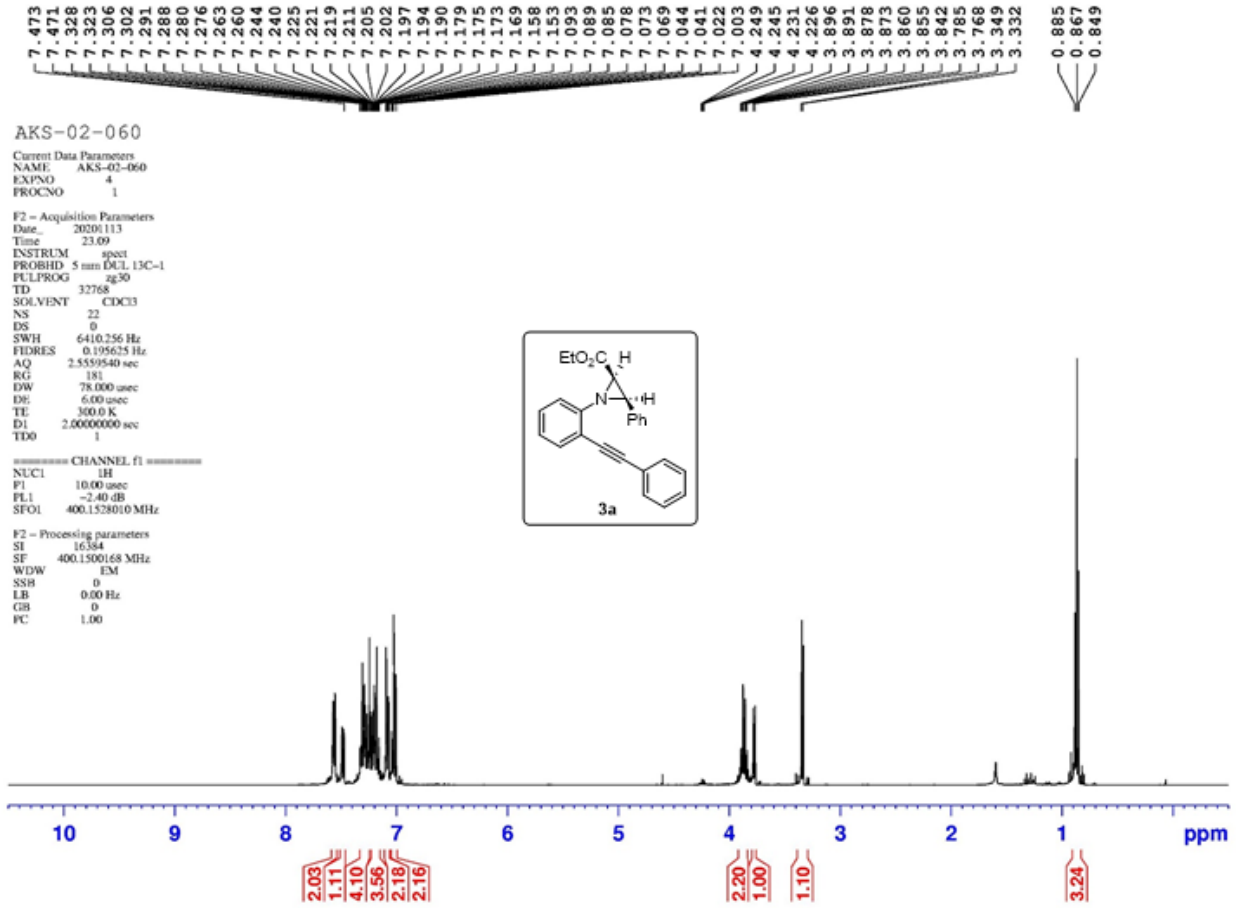
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	12(1)	25(1)	19(1)	-5(1)	1(1)	2(1)
O(1)	16(1)	16(1)	13(1)	2(1)	1(1)	-1(1)
O(2)	16(1)	16(1)	16(1)	0(1)	0(1)	-4(1)
N(1)	11(1)	12(1)	13(1)	2(1)	1(1)	0(1)
C(1)	17(1)	17(1)	20(1)	-5(1)	-4(1)	5(1)
C(2)	14(1)	21(1)	17(1)	-5(1)	0(1)	1(1)
C(3)	16(1)	16(1)	15(1)	-1(1)	-1(1)	-2(1)
C(4)	12(1)	13(1)	13(1)	-2(1)	-4(1)	1(1)
C(5)	11(1)	11(1)	14(1)	3(1)	-1(1)	0(1)
C(6)	12(1)	12(1)	13(1)	-1(1)	-1(1)	0(1)
C(7)	11(1)	14(1)	13(1)	1(1)	3(1)	1(1)
C(8)	16(1)	13(1)	19(1)	-2(1)	-1(1)	1(1)
C(9)	18(1)	21(1)	17(1)	0(1)	-2(1)	5(1)
C(10)	19(1)	17(1)	21(1)	7(1)	4(1)	7(1)
C(11)	12(1)	14(1)	12(1)	0(1)	-1(1)	-4(1)
C(12)	17(1)	13(1)	14(1)	2(1)	-1(1)	-1(1)
C(13)	15(1)	13(1)	14(1)	-2(1)	-4(1)	-2(1)
C(14)	19(1)	12(1)	18(1)	-1(1)	-5(1)	2(1)
C(15)	12(1)	14(1)	11(1)	-1(1)	-2(1)	-2(1)
C(16)	12(1)	14(1)	10(1)	-2(1)	-1(1)	-2(1)
C(17)	19(1)	13(1)	26(1)	1(1)	3(1)	1(1)
C(18)	14(1)	16(1)	17(1)	-1(1)	1(1)	0(1)
C(19)	12(1)	10(1)	13(1)	2(1)	1(1)	4(1)
C(20)	21(1)	21(1)	11(1)	2(1)	1(1)	1(1)
C(21)	32(1)	22(1)	18(1)	-4(1)	7(1)	1(1)
C(22)	14(1)	14(1)	14(1)	-2(1)	-2(1)	1(1)
C(23)	10(1)	21(1)	16(1)	-6(1)	-2(1)	2(1)
C(24)	13(1)	21(1)	16(1)	-1(1)	3(1)	-5(1)
C(25)	17(1)	15(1)	17(1)	1(1)	0(1)	-3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 201206lt_0m.

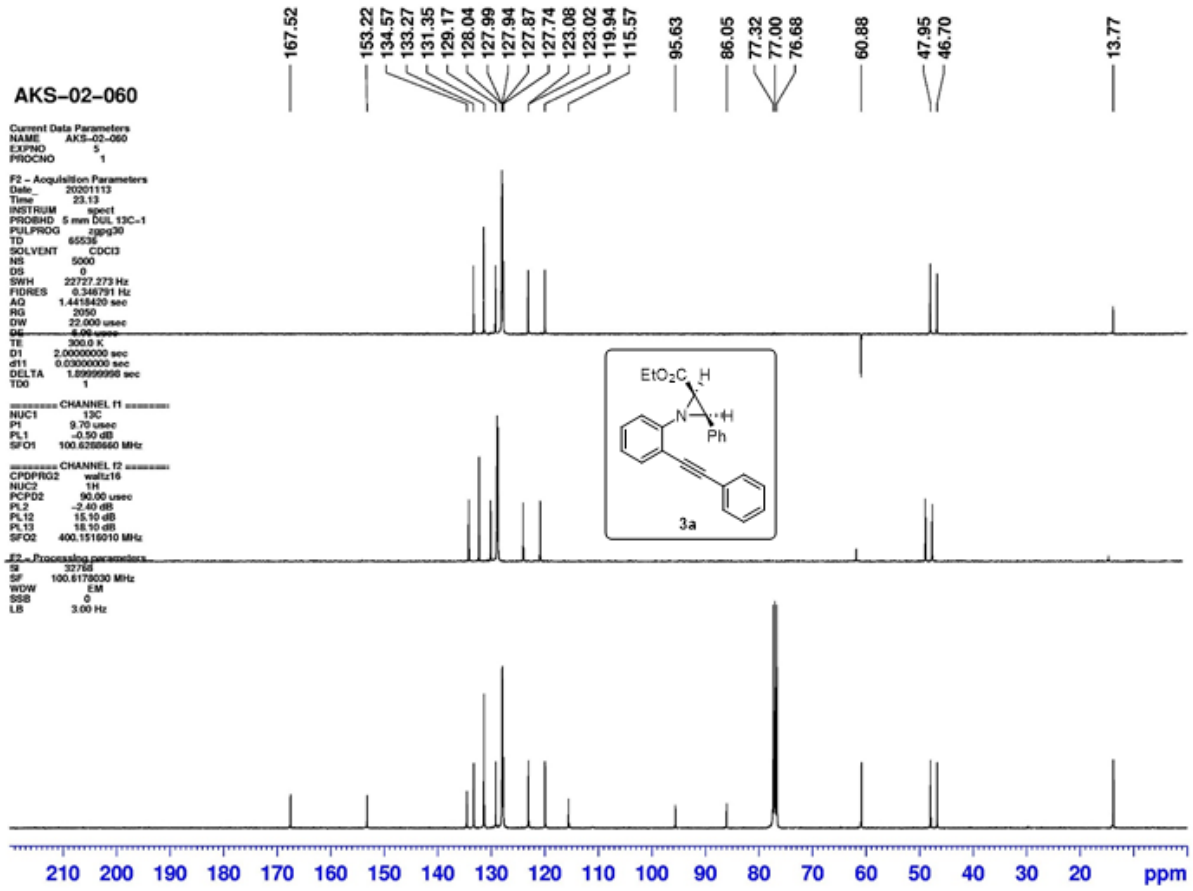
	x	y	z	U(eq)
H(1)	884	6210	1046	22
H(20)	619	4154	1572	21
H(3)	2730	2406	1645	19
H(16)	4978	749	1473	14
H(6)	6487	-191	839	15
H(10)	9240	83	1905	19
H(9)	9854	-1914	2429	22
H(2)	8777	-4267	2259	23
H(5)	6349	5112	311	18
H(4)	3184	6538	559	20
H(8)	7107	-4619	1557	23
H(7)	6407	-2602	1053	19
H(12)	7773	1496	2774	21
H(11)	6086	981	3018	21
H(15)	6850	3924	2709	36
H(13)	6565	3394	3272	36
H(14)	5064	3448	2886	36
H(19)	10261	-530	894	17
H(18)	11603	2947	26	20
H(17)	9114	4121	197	19

6. ^{1}H and ^{13}C spectra of key compounds:

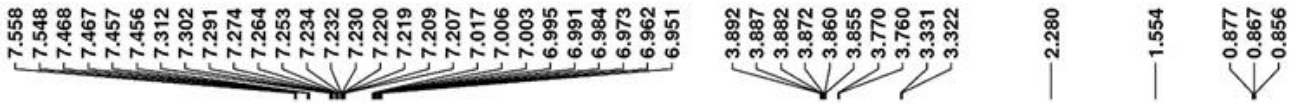
Solvent: $CDCl_3$
SFO1: 400 MHz



Solvent: $CDCl_3$
SFO1: 400 MHz

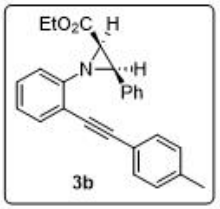


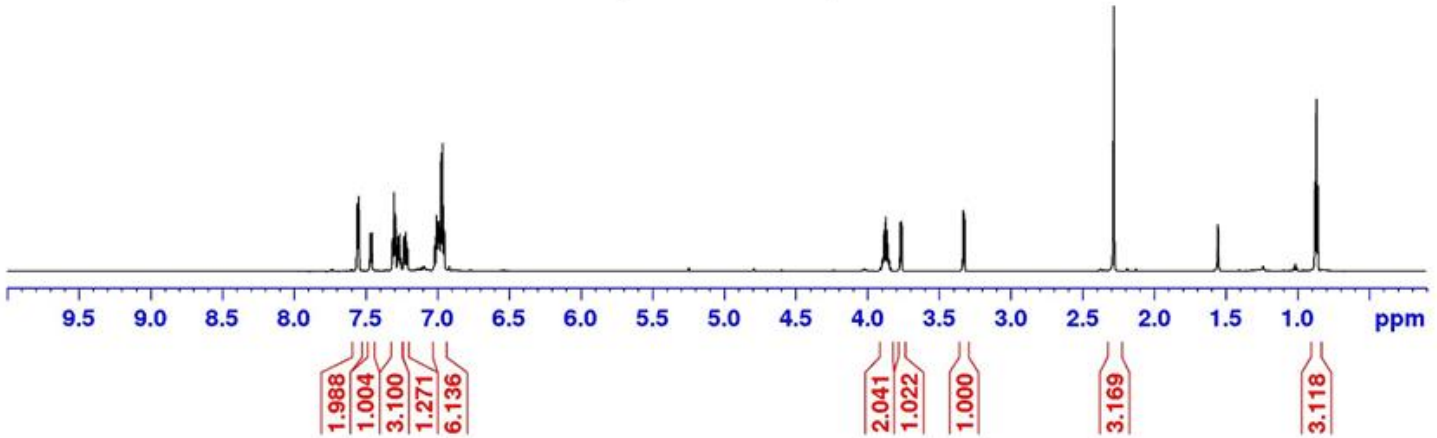
Solvent: $CDCl_3$
SFO1: 700 MHz

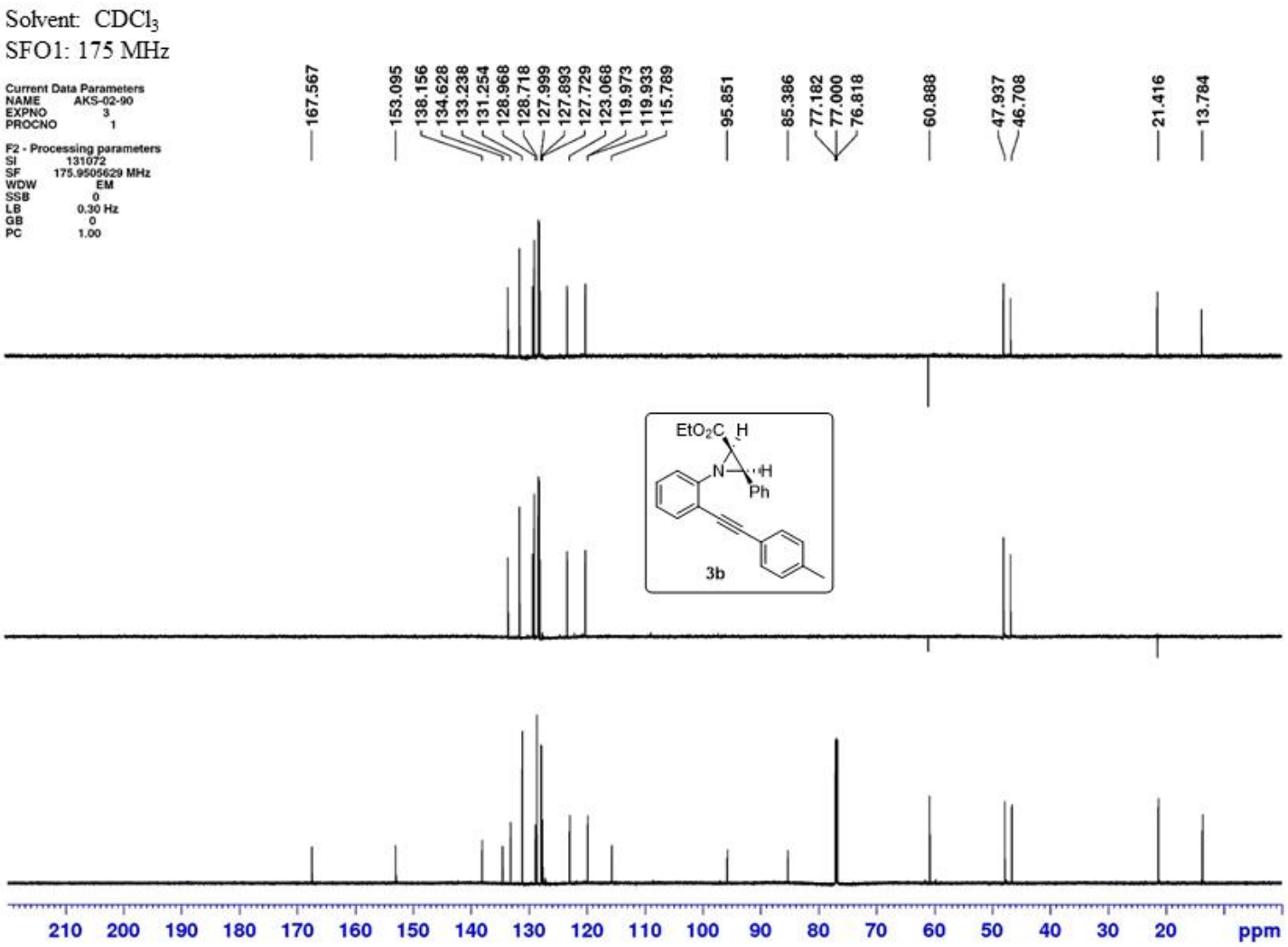


Current Data Parameters
NAME AKS-02-90-H.fid
EXPNO 3
PROCNO 1

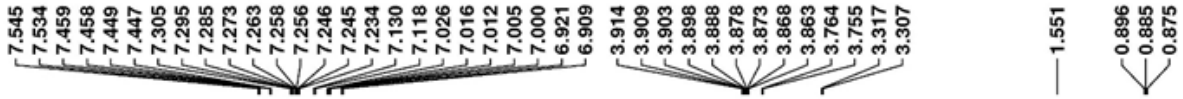
F2 - Processing parameters
SI 65536
SF 699.7431031 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





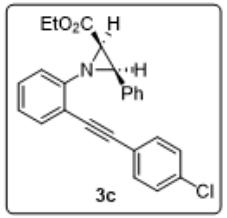

Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-90
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505629 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
167.567
153.095
138.156
134.628
133.238
131.254
128.968
128.718
127.999
127.893
127.729
123.068
119.973
119.933
115.789
95.851
85.386
77.182
77.000
76.818
60.888
47.937
46.708
21.416
13.784
EtO2C
H
N
H
Ph
3b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

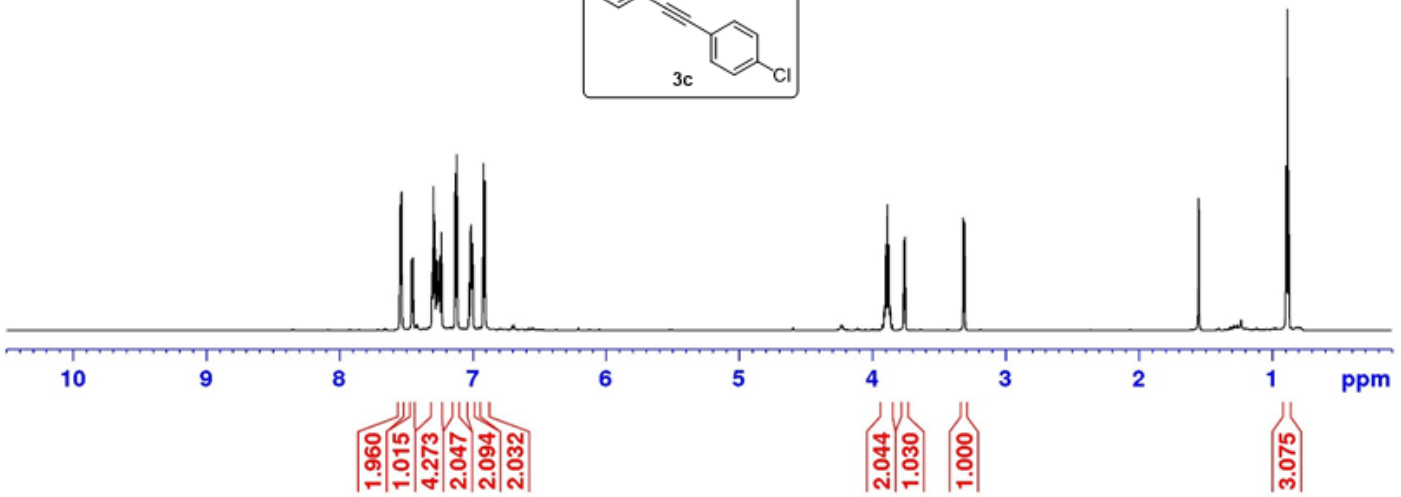
Solvent: $CDCl_3$
SFO1: 700 MHz



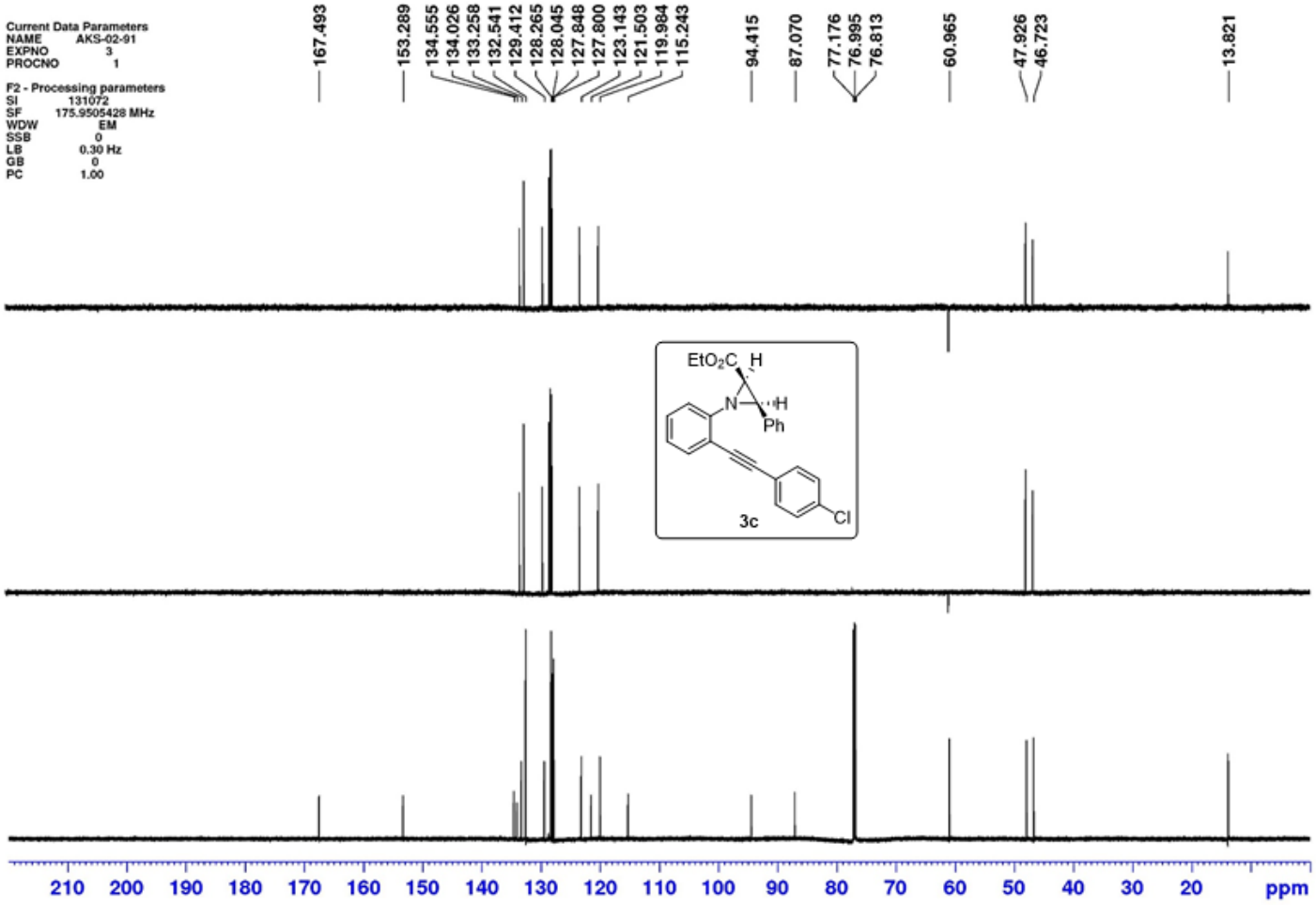
Current Data Parameters
NAME AKS-02-91
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431031 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





Solvent: $CDCl_3$
SFO1: 175 MHz



Solvent: $CDCl_3$
SFO1: 700 MHz

7.548 7.538 7.463 7.452 7.311 7.301 7.291 7.279 7.270 7.266 7.264 7.253 7.240 7.032 7.021 7.017 7.010 7.006 6.850 6.838

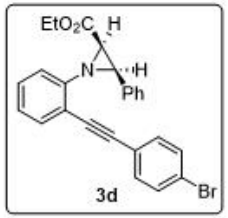
3.916 3.911 3.906 3.895 3.885 3.880 3.875 3.770 3.760 3.321 3.311

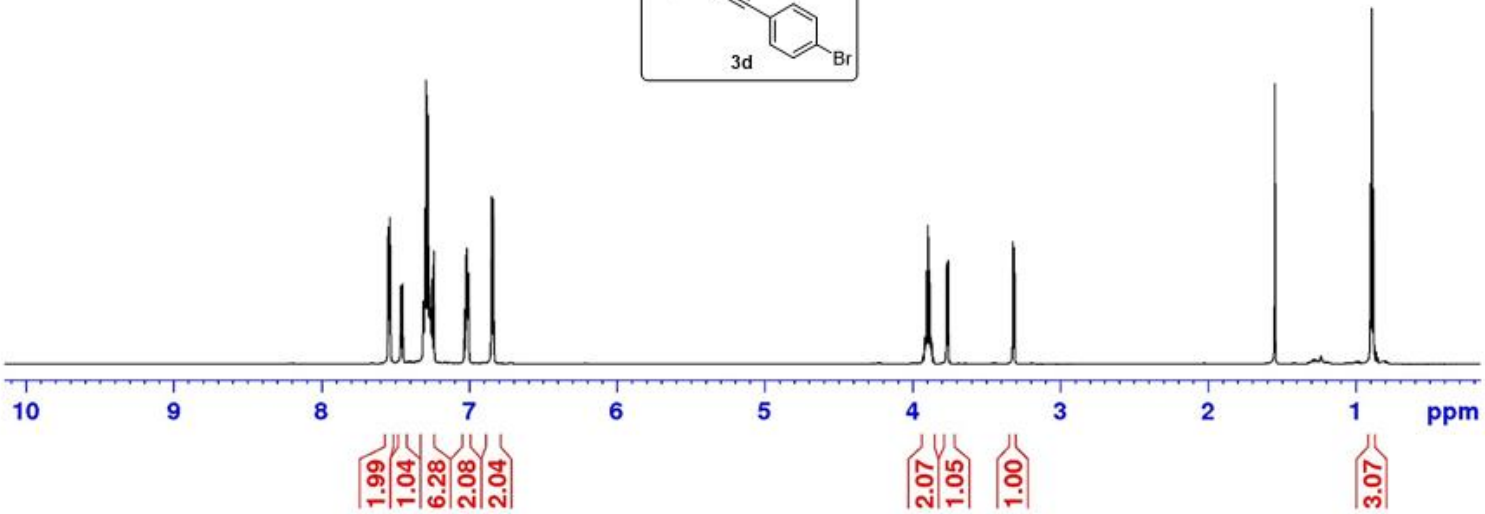
1.548

0.901 0.891 0.881

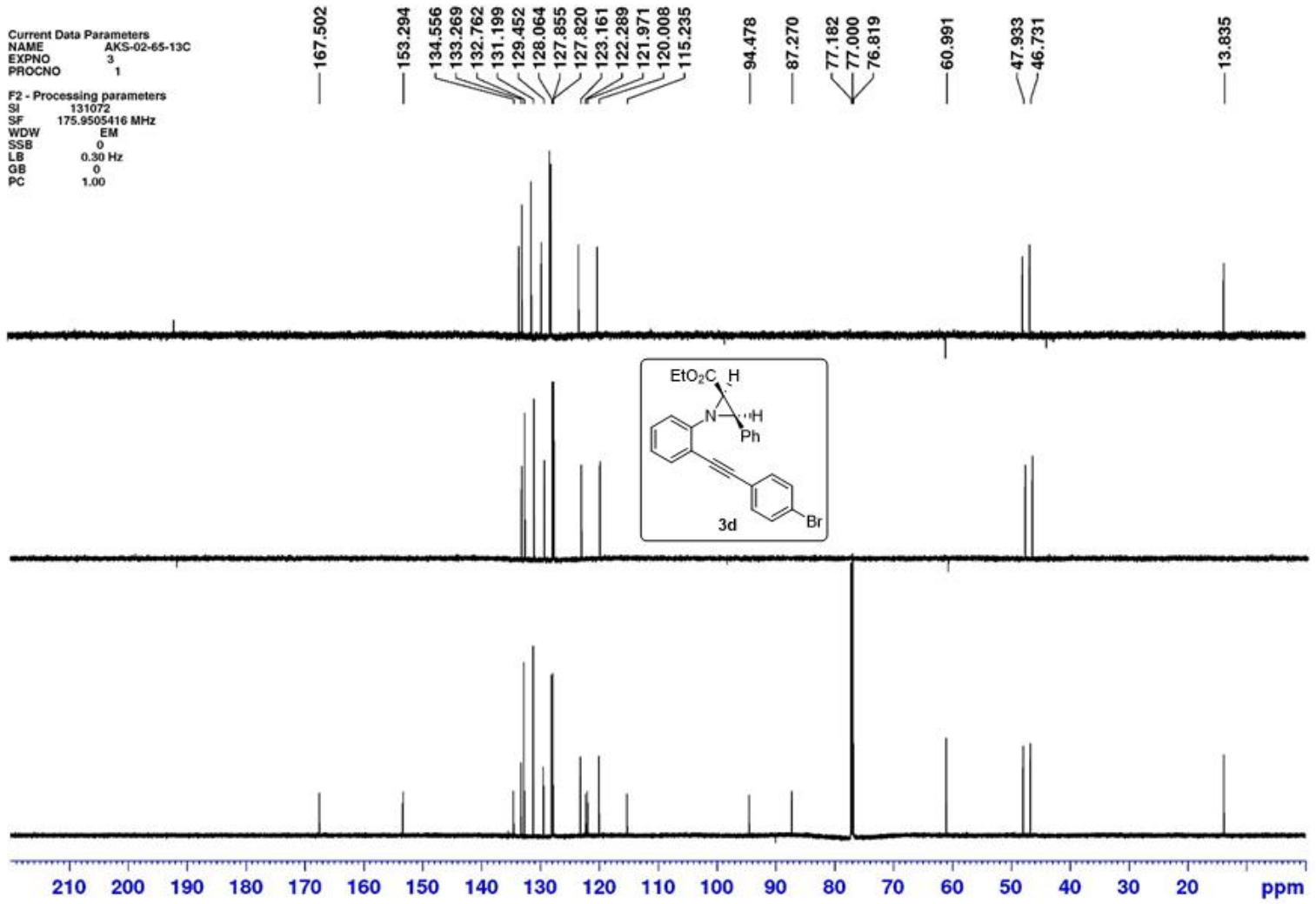
Current Data Parameters
NAME AKS-02-65-H.fid
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431027 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

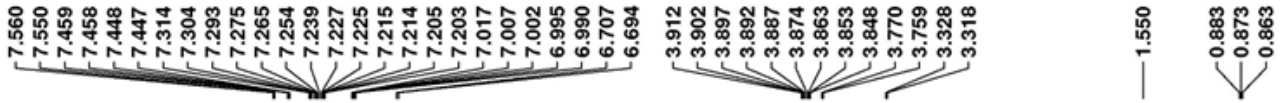




Solvent: $CDCl_3$
SFO1: 175 MHz

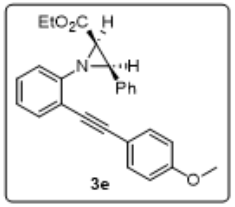


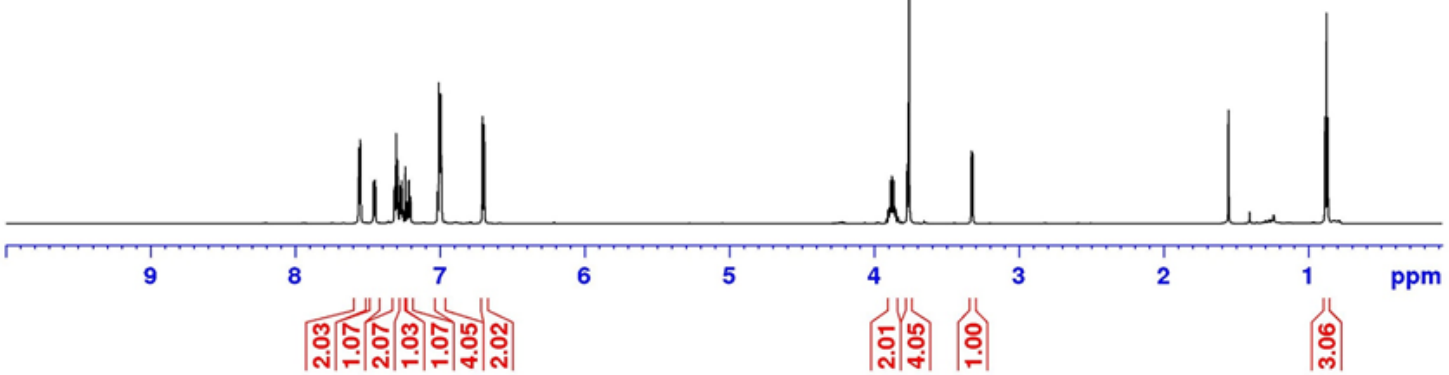
Solvent: $CDCl_3$
SFO1: 700 MHz

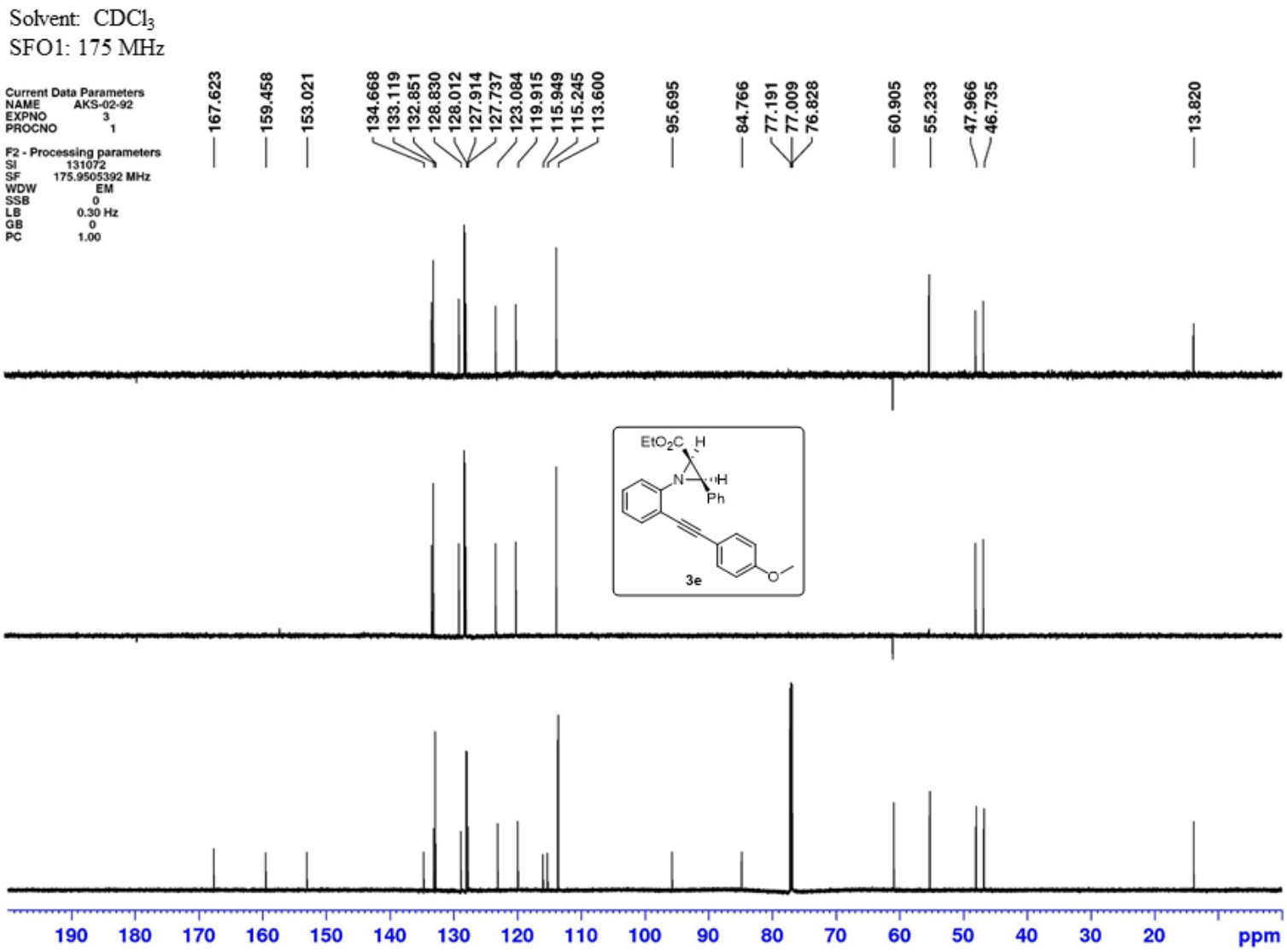


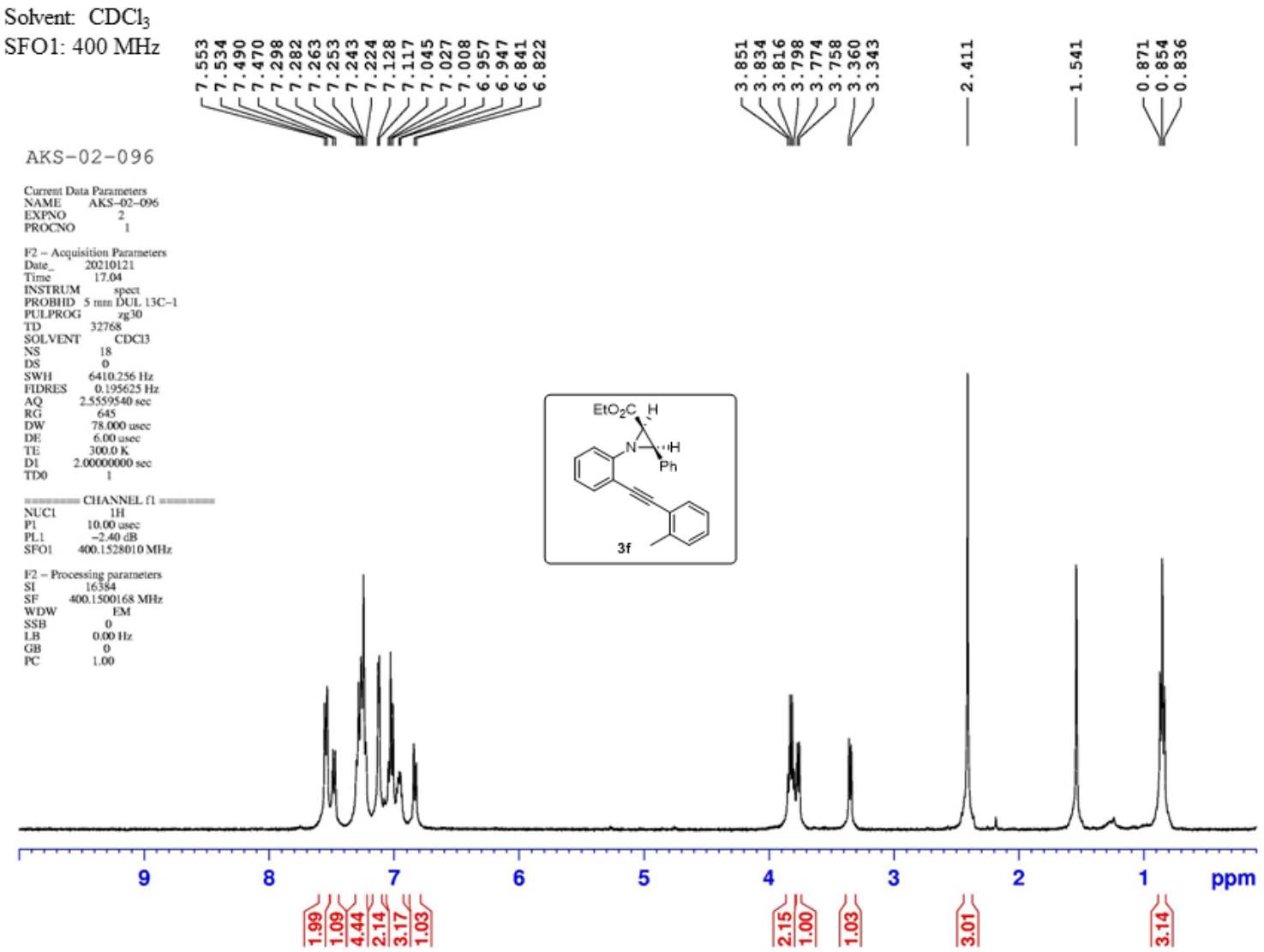
Current Data Parameters
NAME AKS-02-92-H.fid
EXPNO 1
PROCNO 1

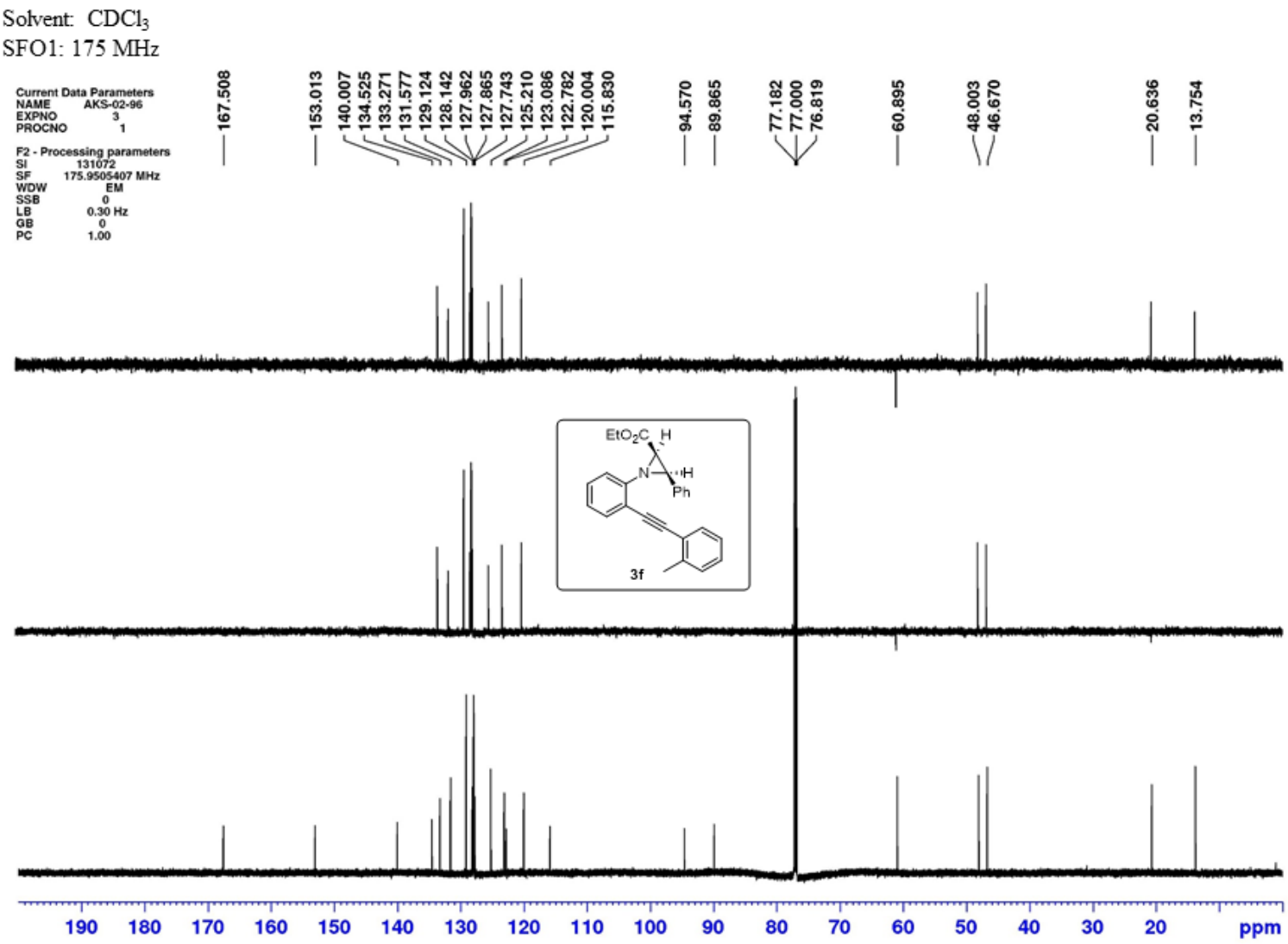
F2 - Processing parameters
SI 65536
SF 699.7430991 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





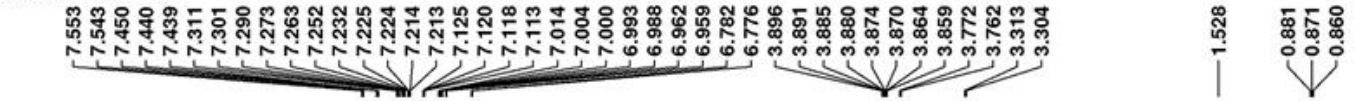

Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-92
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505392 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
167.623
159.458
153.021
134.668
133.119
132.851
128.830
128.012
127.914
127.737
123.084
119.915
115.949
115.245
113.600
95.695
84.766
77.191
77.009
76.828
60.905
55.233
47.966
46.735
13.820
EtO2C
H
N
H
Ph
O
3e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Solvent: $CDCl_3$
SFO1: 400 MHz
7.553
7.534
7.490
7.470
7.298
7.282
7.263
7.253
7.243
7.224
7.128
7.117
7.045
7.027
7.008
6.957
6.947
6.841
6.822
3.851
3.834
3.816
3.798
3.774
3.758
3.360
3.343
2.411
1.541
0.871
0.854
0.836
AKS-02-096
Current Data Parameters
NAME AKS-02-096
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20210121
Time 17.04
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 18
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 645
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 – Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
EtO2C
H
N
H
Ph
3f
9
8
7
6
5
4
3
2
1
ppm
1.99
1.09
4.44
2.14
3.17
1.03
2.15
1.00
1.03
3.01
3.14


Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-96
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505407 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
167.508
153.013
140.007
134.525
133.271
131.577
129.124
128.142
127.962
127.865
127.743
125.210
123.086
122.782
120.004
115.830
94.570
89.865
77.182
77.000
76.819
60.895
48.003
46.670
20.636
13.754
EtO2C
H
N
H
Ph
3f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

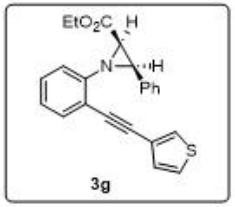
Solvent: $CDCl_3$

SFO1: 700 MHz

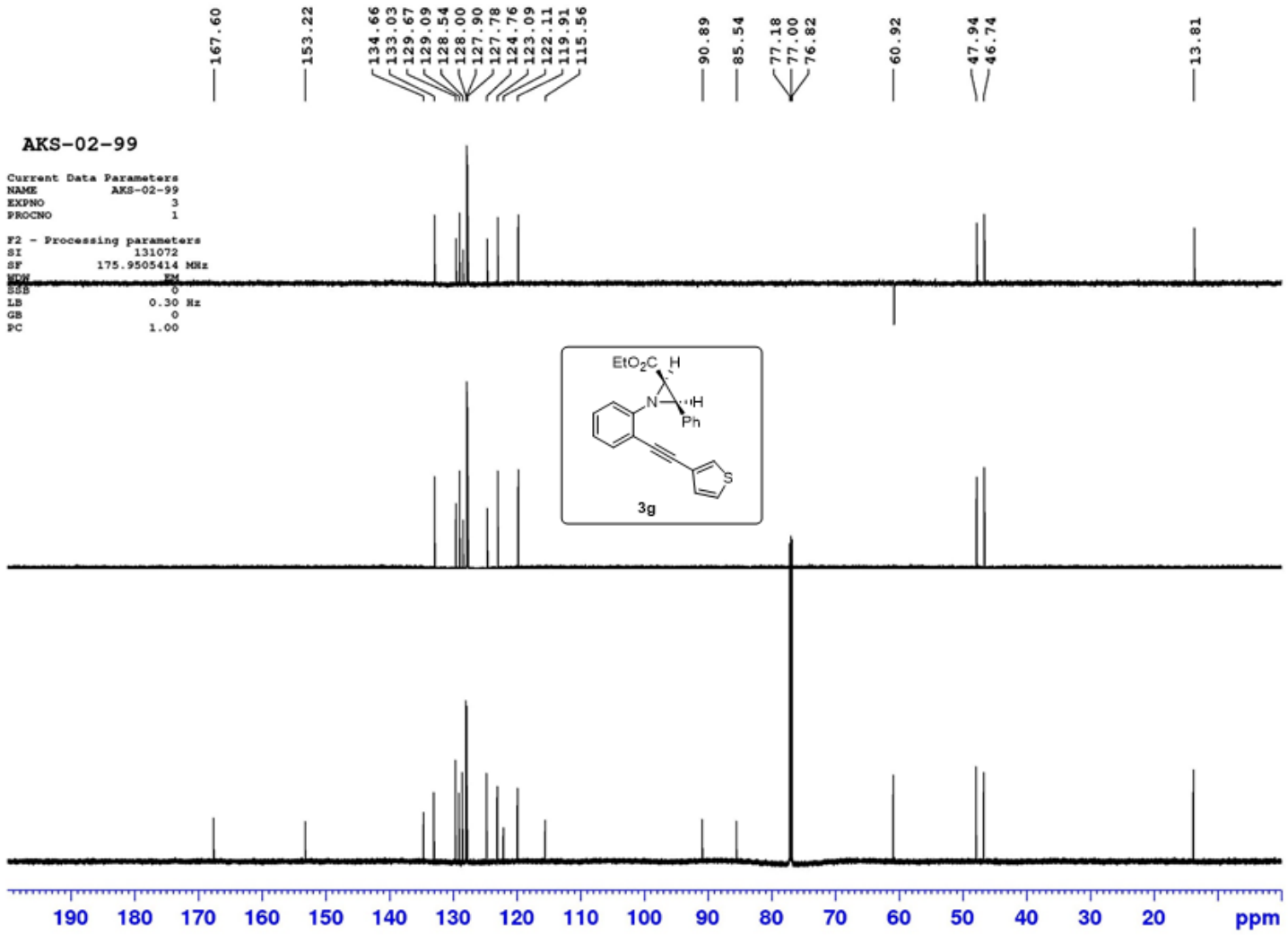


Current Data Parameters
NAME AKS-02-99
EXPNO 1
PROCNO 1

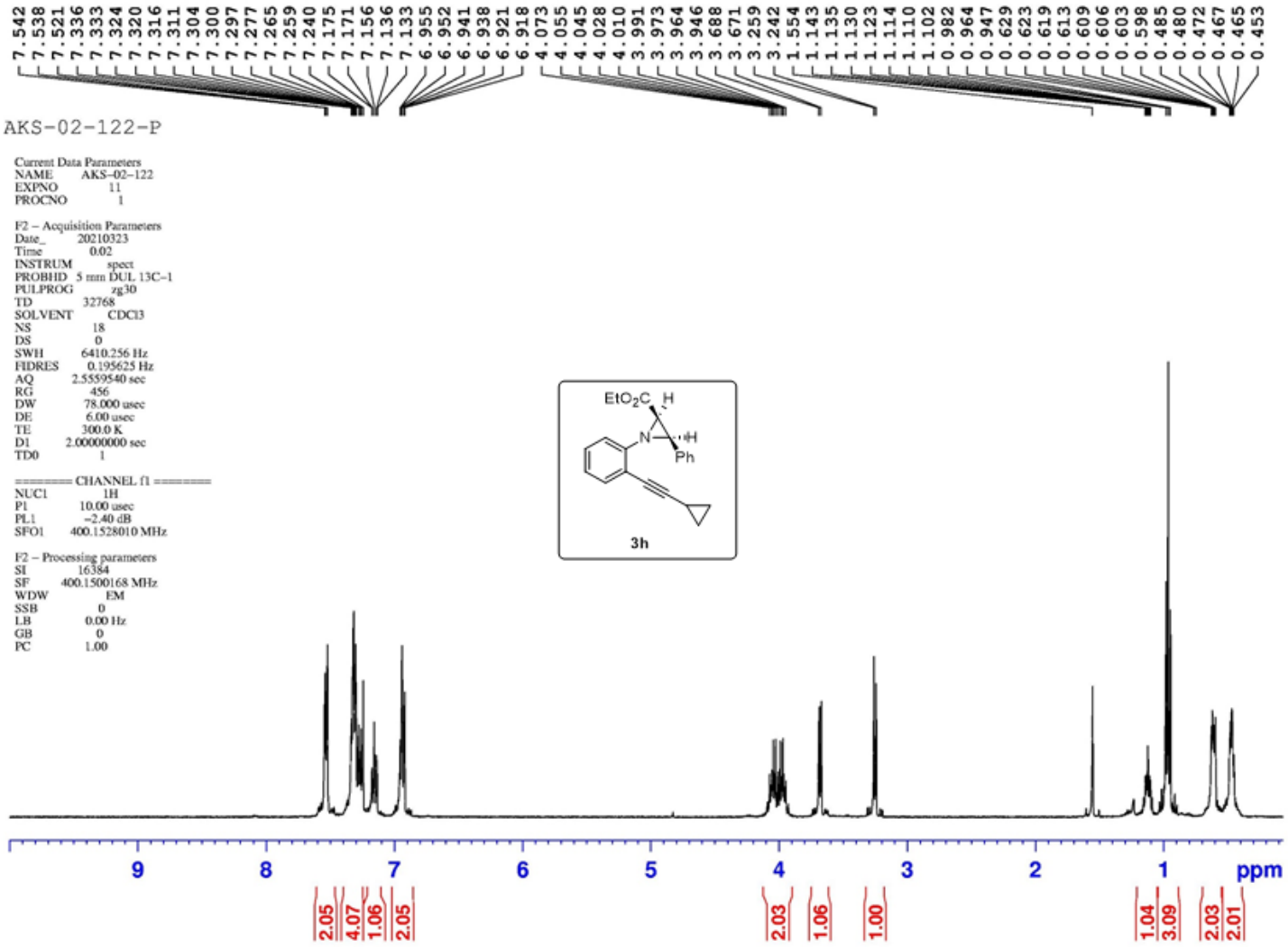
F2 - Processing parameters
SI 65536
SF 699.7431083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

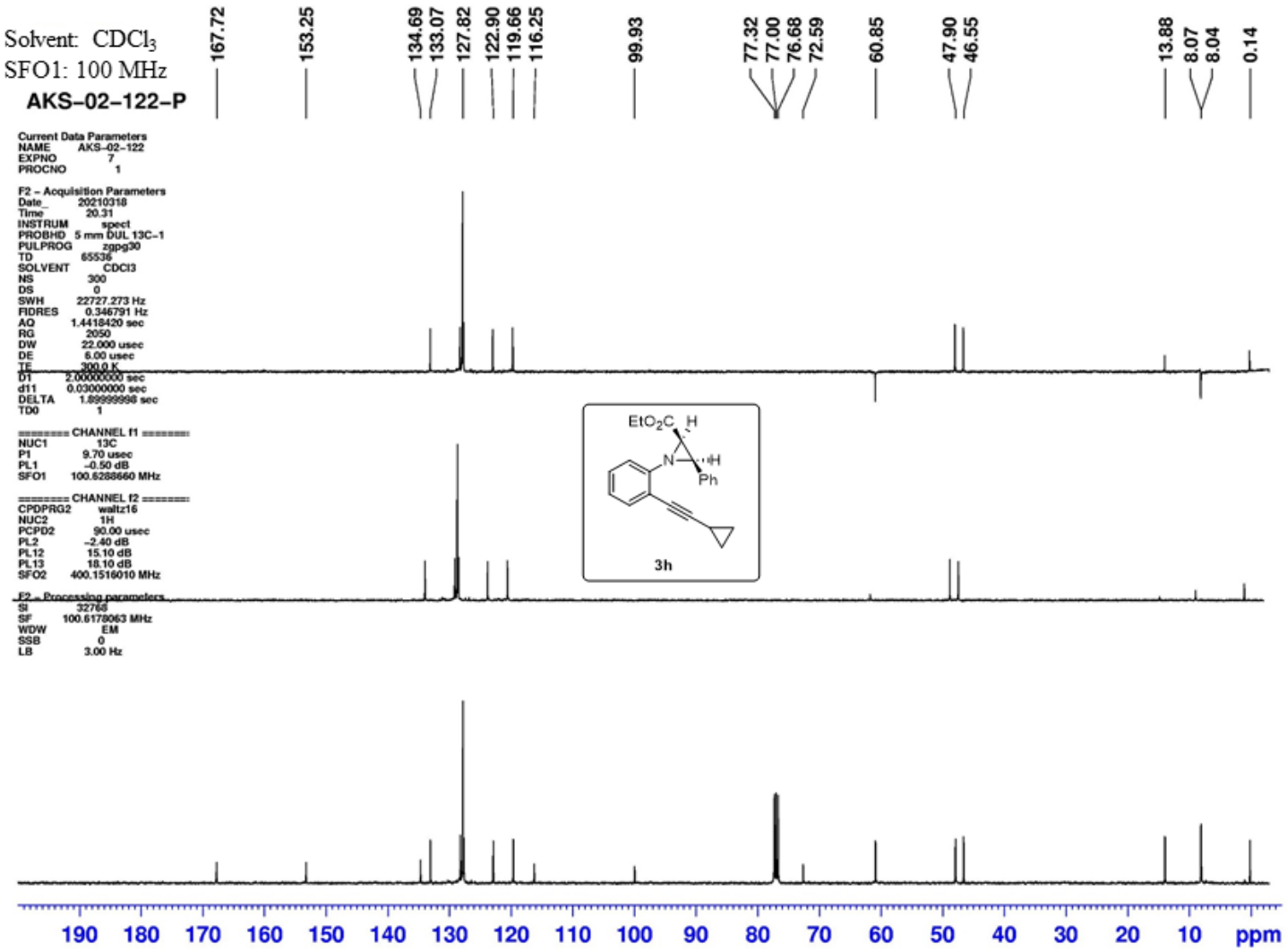


Solvent: $CDCl_3$
SFO1: 175 MHz



Solvent: $CDCl_3$
SFO1: 400 MHz

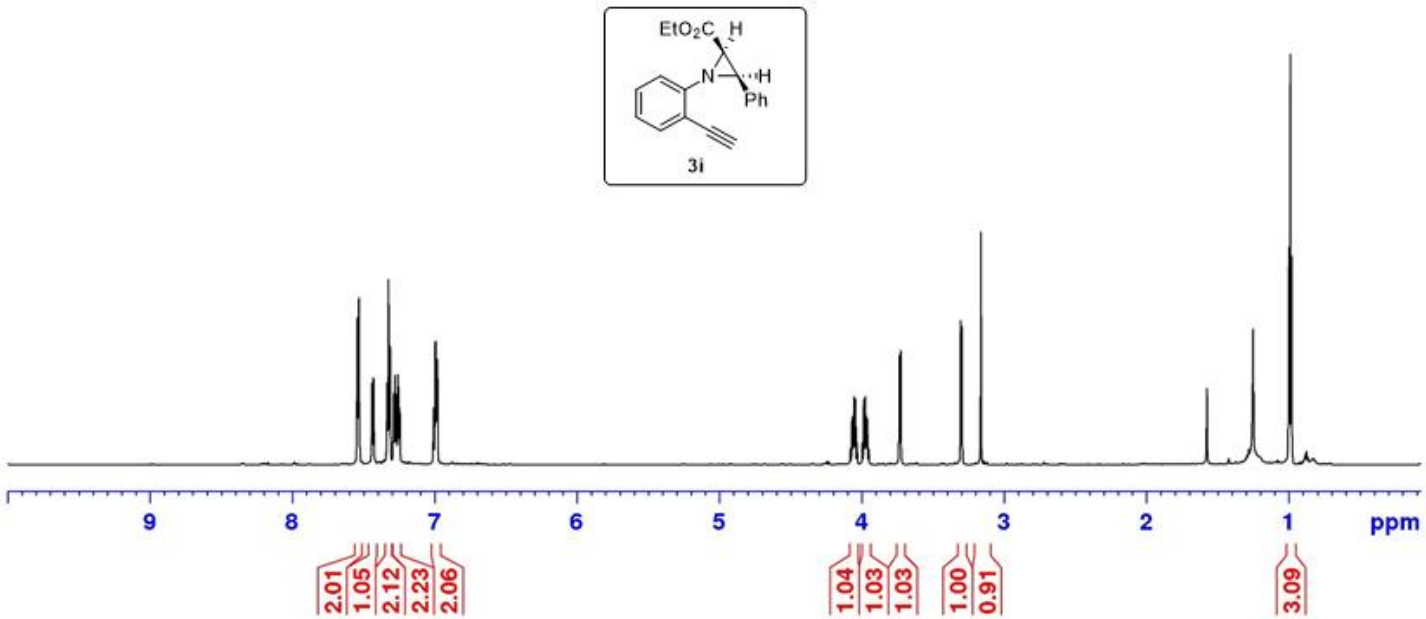


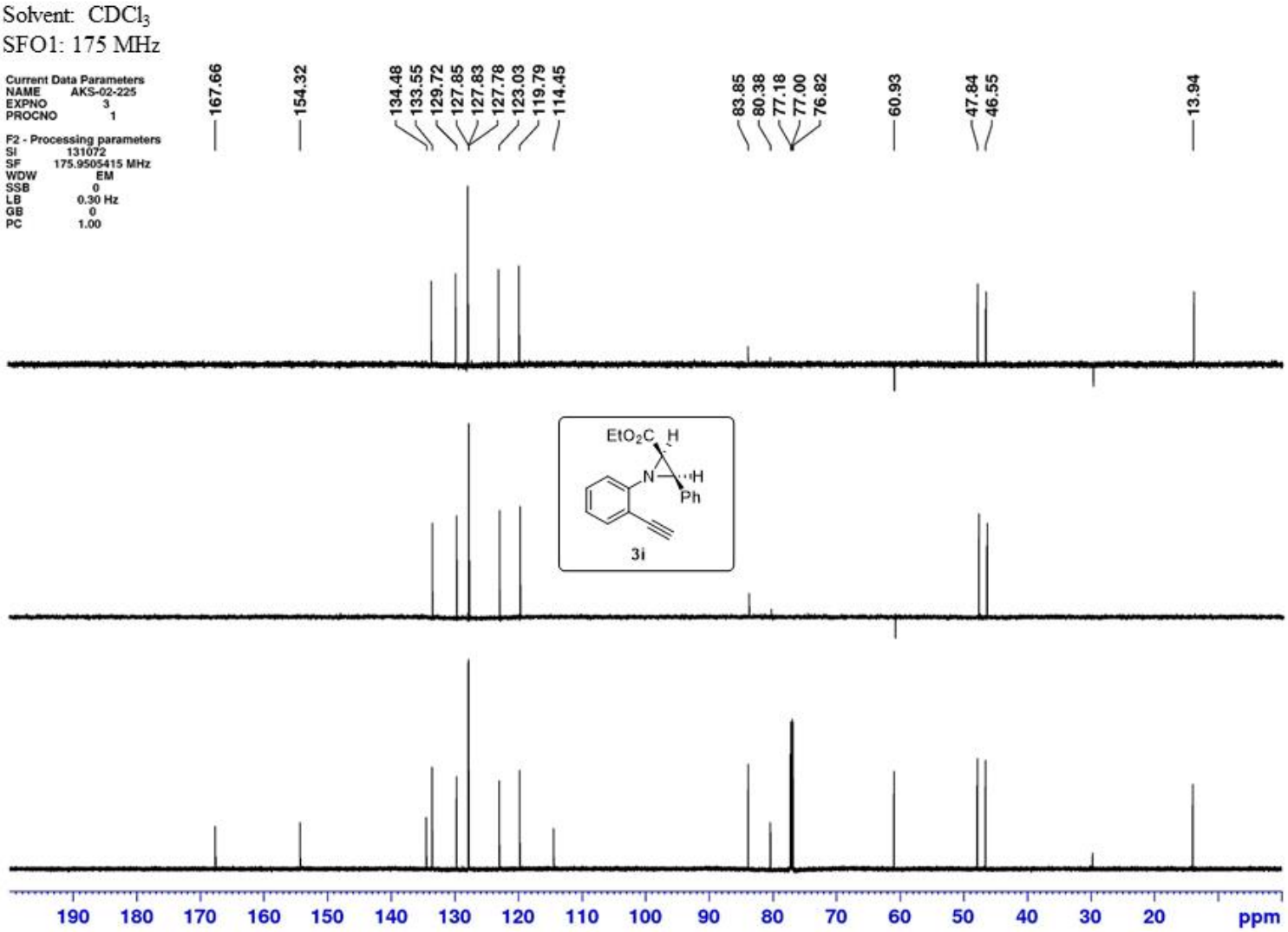

Solvent: CDCl3
SFO1: 100 MHz
AKS-02-122-P
167.72
153.25
134.69
133.07
127.82
122.90
119.66
116.25
99.93
77.32
77.00
76.68
72.59
60.85
47.90
46.55
13.88
8.07
8.04
0.14
Current Data Parameters
NAME AKS-02-122
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210318
Time 20.31
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 2050
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178063 MHz
WDW EM
SSB 0
LB 3.00 Hz
EtO2C
H
N
H
Ph
3h
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Solvent: $CDCl_3$
SFO1: 700 MHz

Current Data Parameters
NAME AKS-02-225
EXPNO 1
PROCNO 1

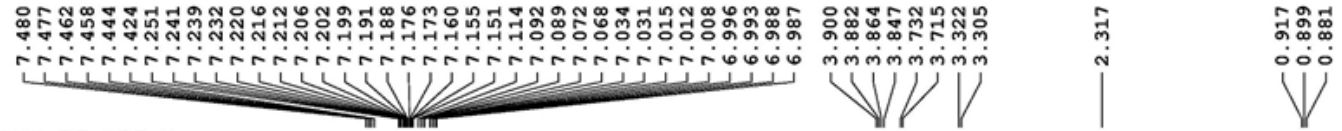
F2 - Processing parameters
SI 65536
SF 699.7430945 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-225
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505415 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
167.66
154.32
134.48
133.55
129.72
127.85
127.83
127.78
123.03
119.79
114.45
83.85
80.38
77.18
77.00
76.82
60.93
47.84
46.55
13.94
EtO2C
H
N
H
Ph
3i
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Solvent: $CDCl_3$

SFO1: 400 MHz



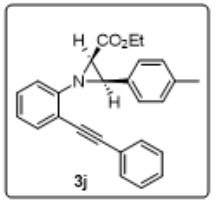
AKS-02-102-P

Current Data Parameters
NAME AKS-02-102
EXPNO 9
PROCNO 1

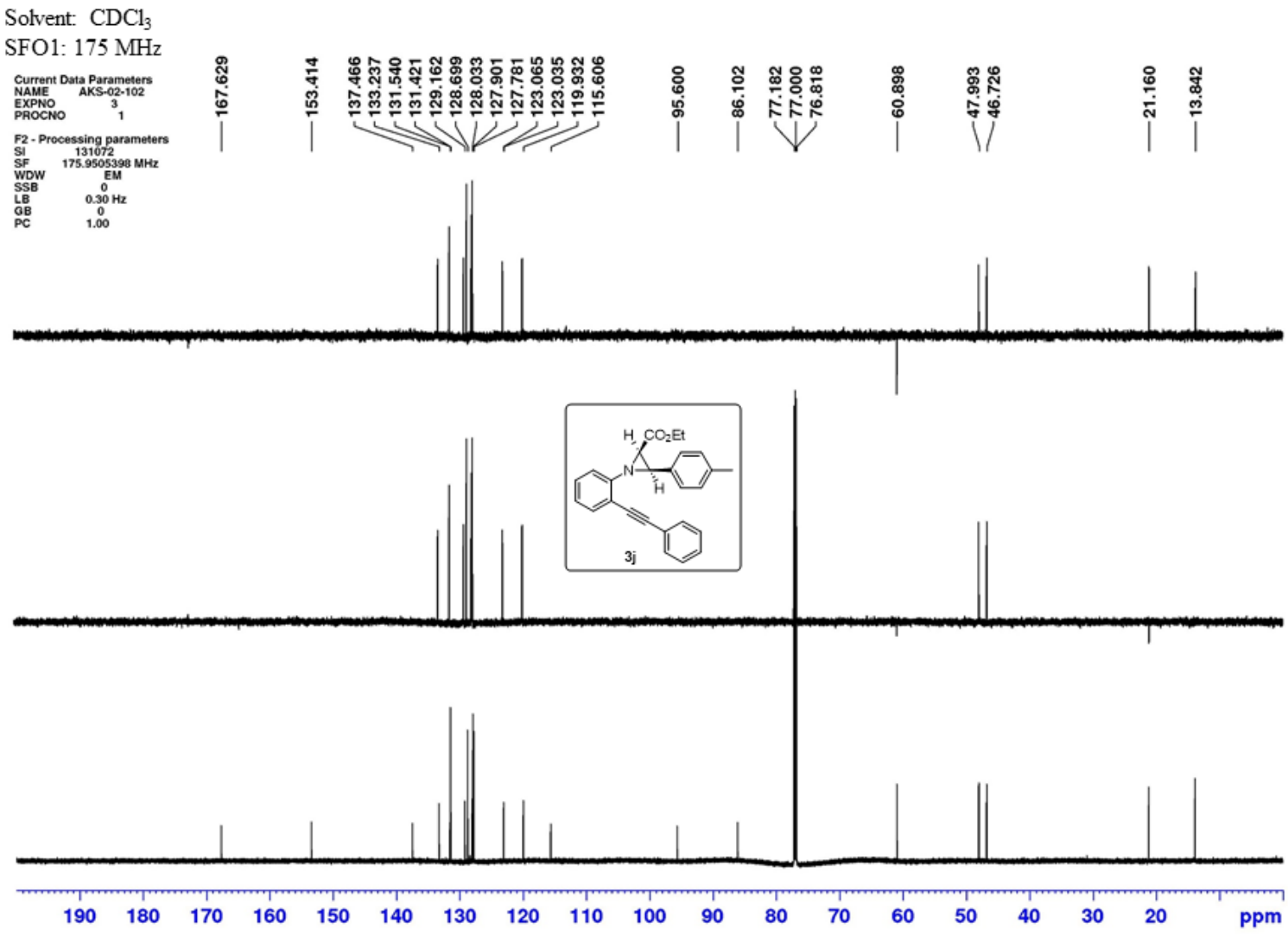
F2 – Acquisition Parameters
Date_ 20210220
Time 17.45
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 19
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 724
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz

F2 – Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



3j


Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-102
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505398 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
167.629
153.414
137.466
133.237
131.540
131.421
129.162
128.699
128.033
127.901
127.781
123.065
123.035
119.932
115.606
95.600
86.102
77.182
77.000
76.818
60.898
47.993
46.726
21.160
13.842
H
CO2Et
N
H
3j
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Solvent: $CDCl_3$

SFO1: 700 MHz

7.464 7.452 7.441 7.440 7.430 7.429 7.223 7.217 7.211 7.207 7.206 7.203 7.195 7.184 7.166 7.155 7.144 7.007 6.997 6.995 6.993 6.982 6.981 6.951 6.940

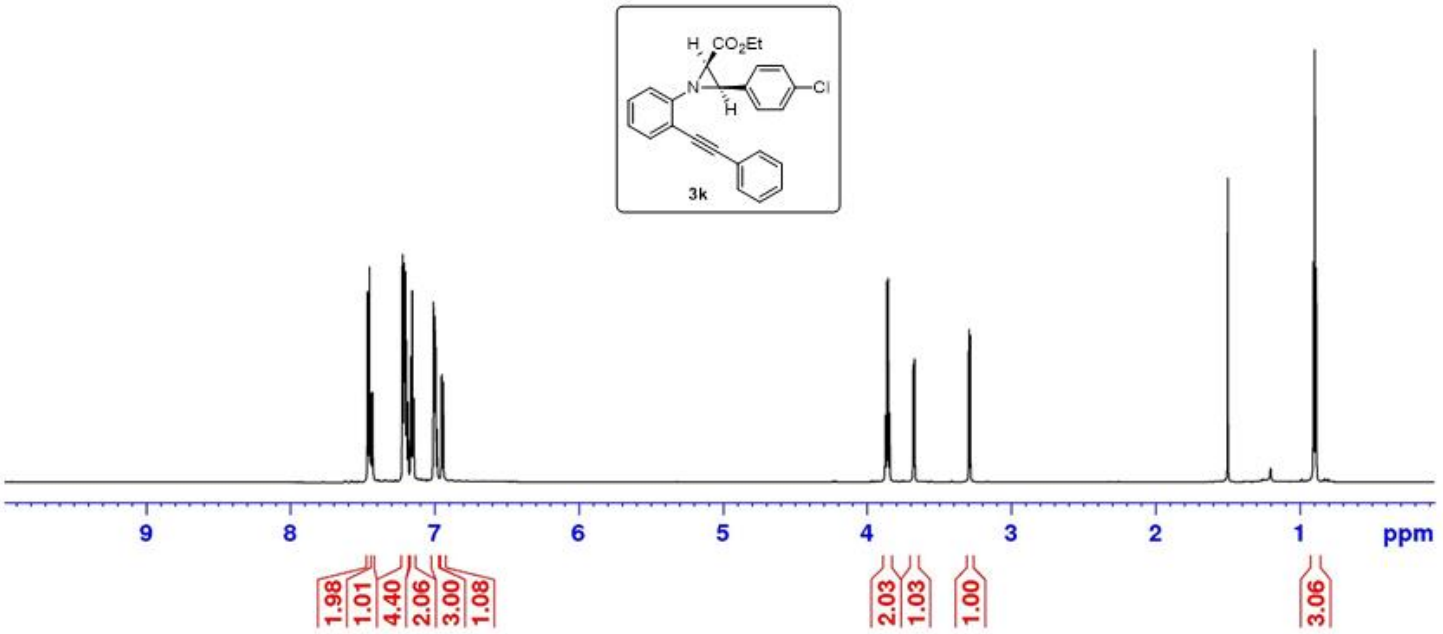
3.873 3.863 3.853 3.843 3.679 3.669 3.293 3.283

1.497

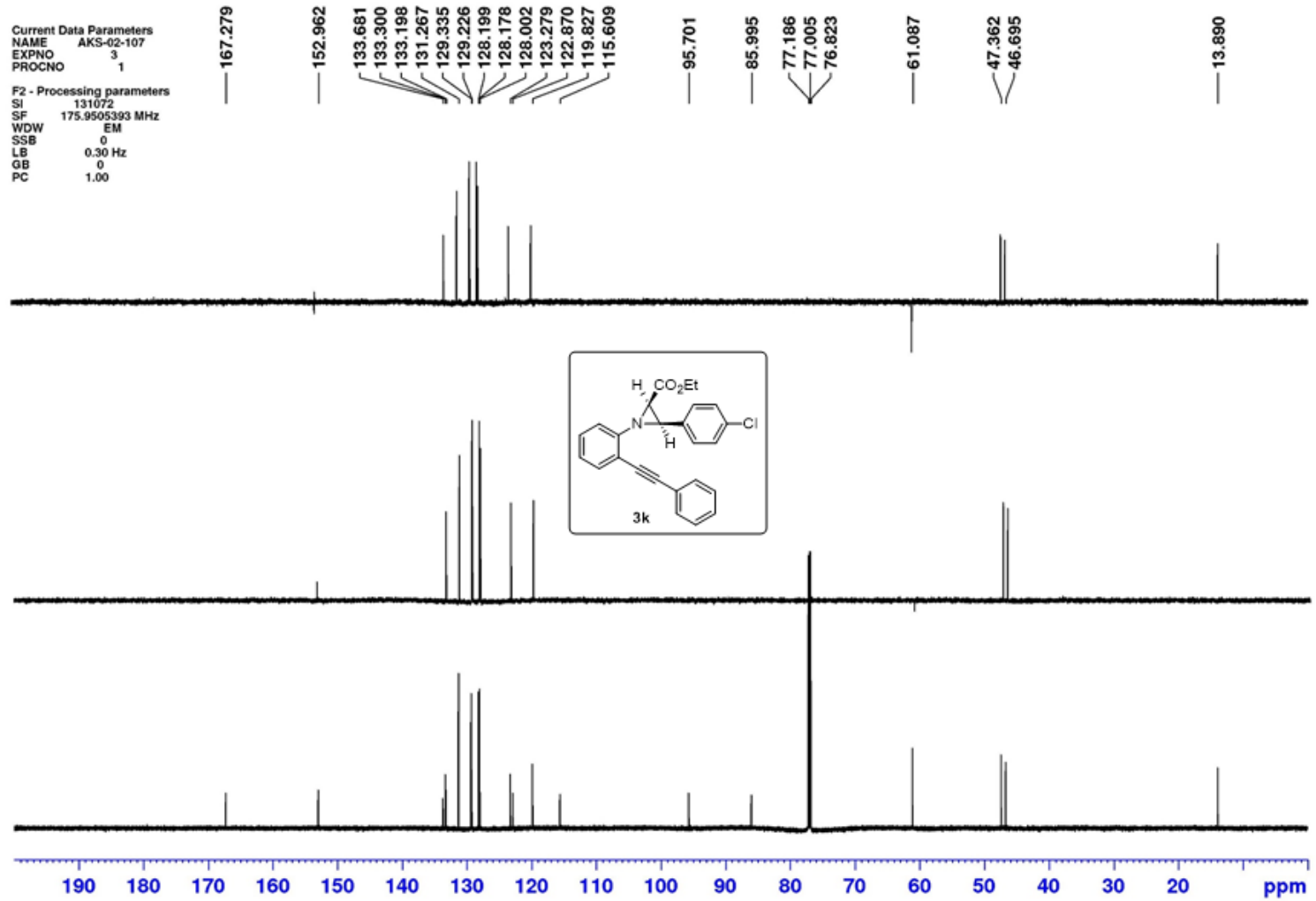
0.903 0.893 0.883

Current Data Parameters
NAME AKS-02-107-A-H.fid
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431271 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

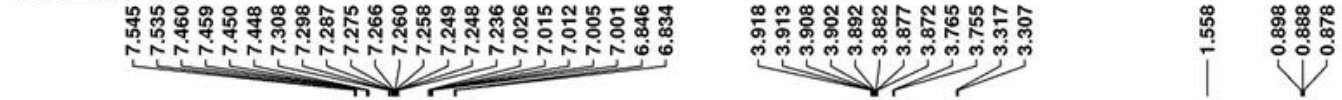


Solvent: $CDCl_3$
SFO1: 175 MHz



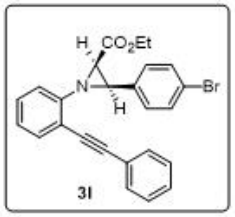
Solvent: $CDCl_3$

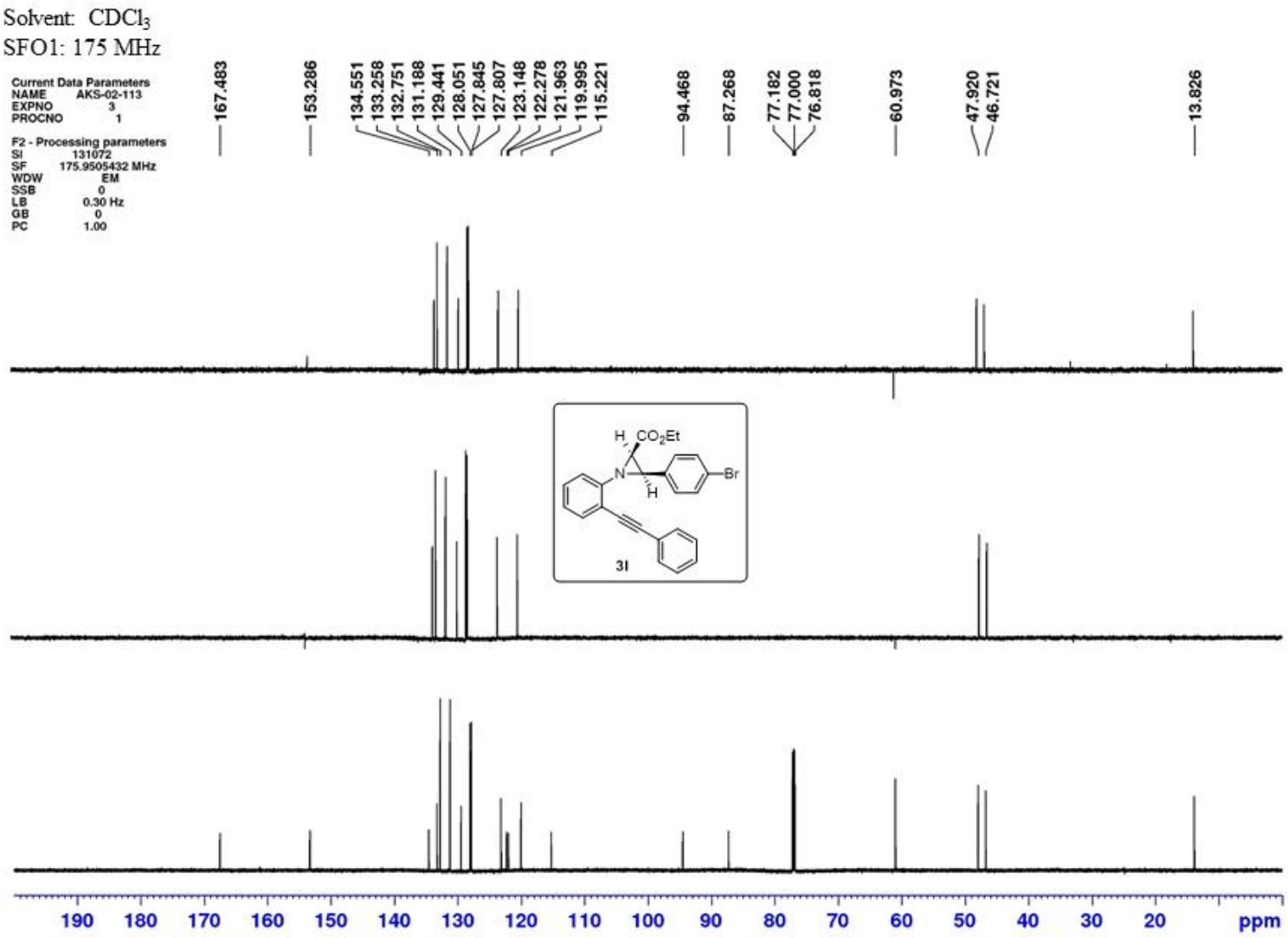
SFO1: 700 MHz



Current Data Parameters
NAME AKS-02-113
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431025 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



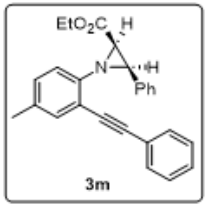

Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-113
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505432 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
167.483
153.286
134.551
133.258
132.751
131.188
129.441
128.051
127.845
127.807
123.148
122.278
121.963
119.995
115.221
94.468
87.268
77.182
77.000
76.818
60.973
47.920
46.721
13.826
H
CO_2Et
N
H
Br
3l
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

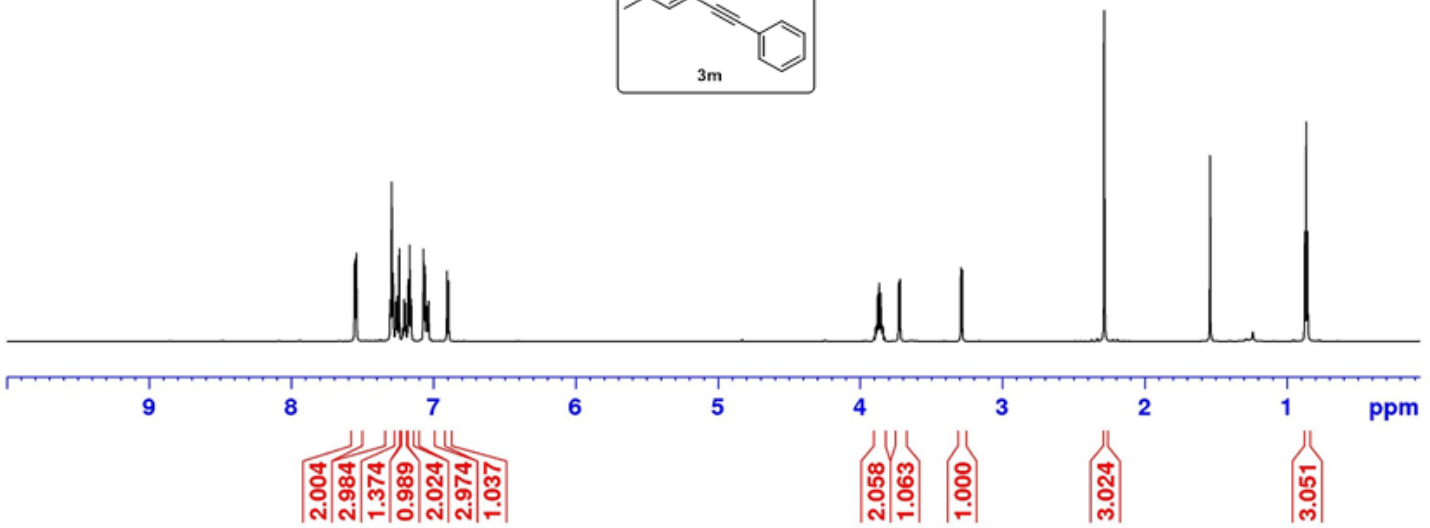
Solvent: $CDCl_3$

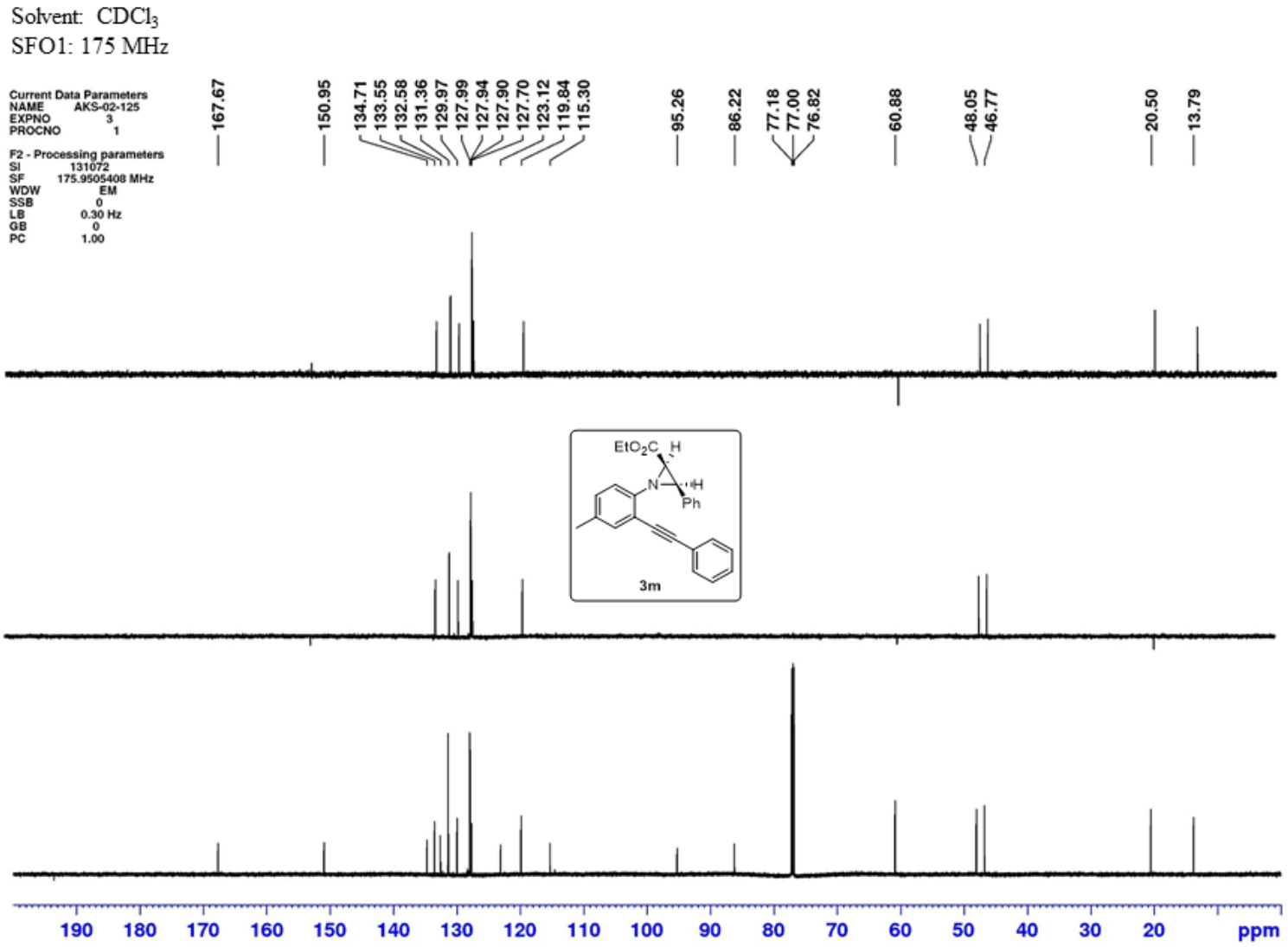
SFO1: 700 MHz

Current Data Parameters
NAME AKS-02-125
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431016 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





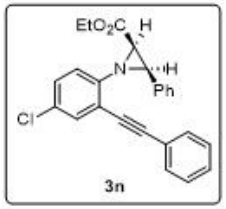

Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-125
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505408 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
167.67
150.95
134.71
133.55
132.58
131.36
129.97
127.99
127.94
127.90
127.70
123.12
119.84
115.30
95.26
86.22
77.18
77.00
76.82
60.88
48.05
46.77
20.50
13.79
EtO2C
H
N
H
Ph
3m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Solvent: $CDCl_3$

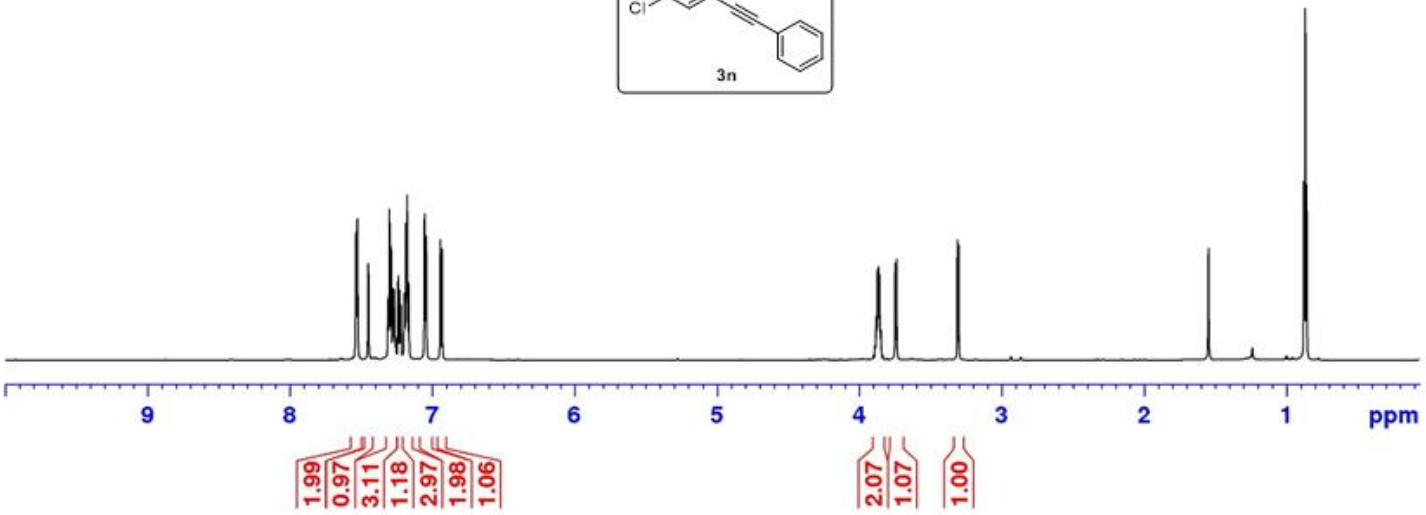
SFO1: 700 MHz

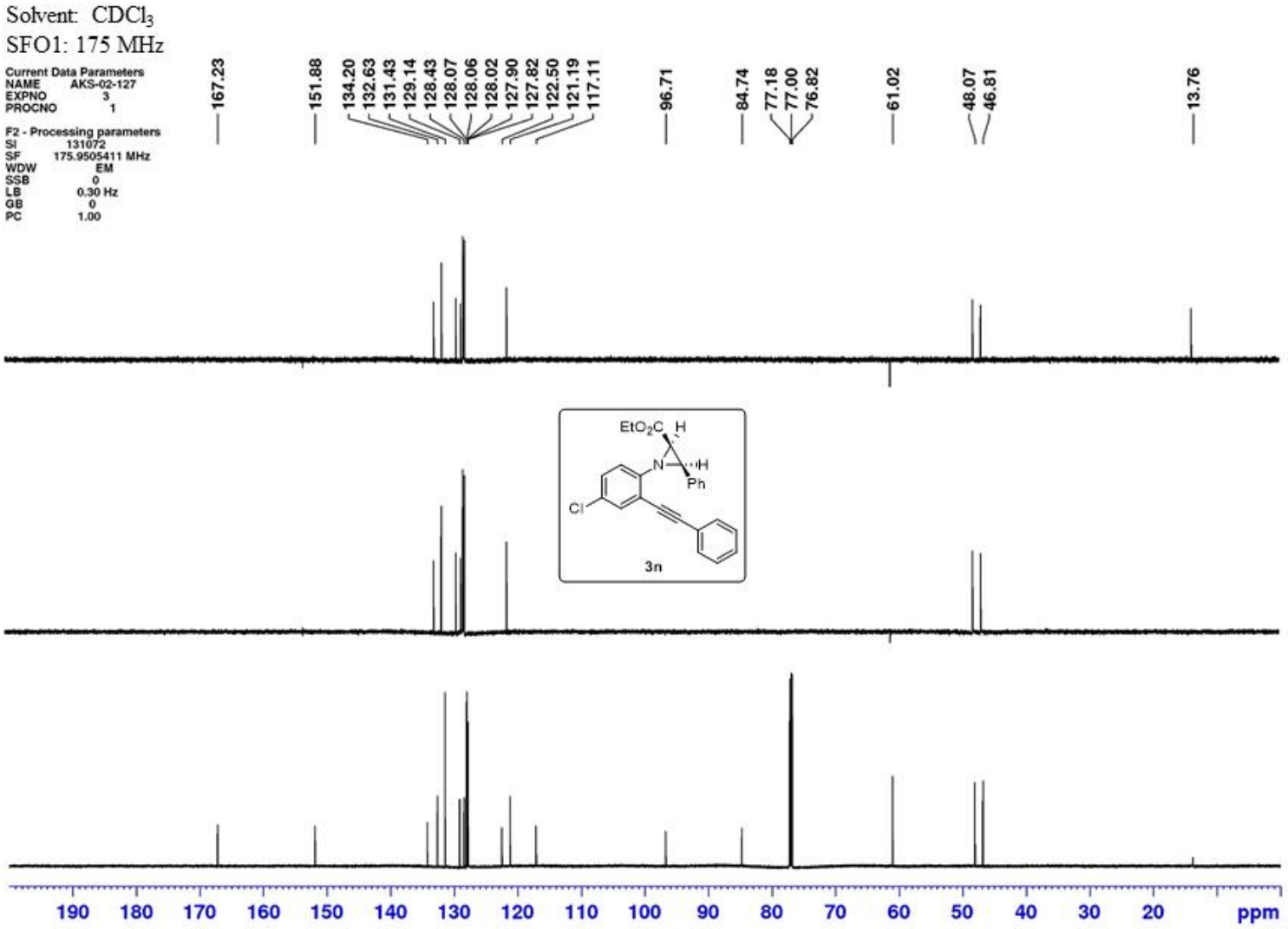
Current Data Parameters
NAME AKS-02-127
EXPNO 1
PROCNO 1

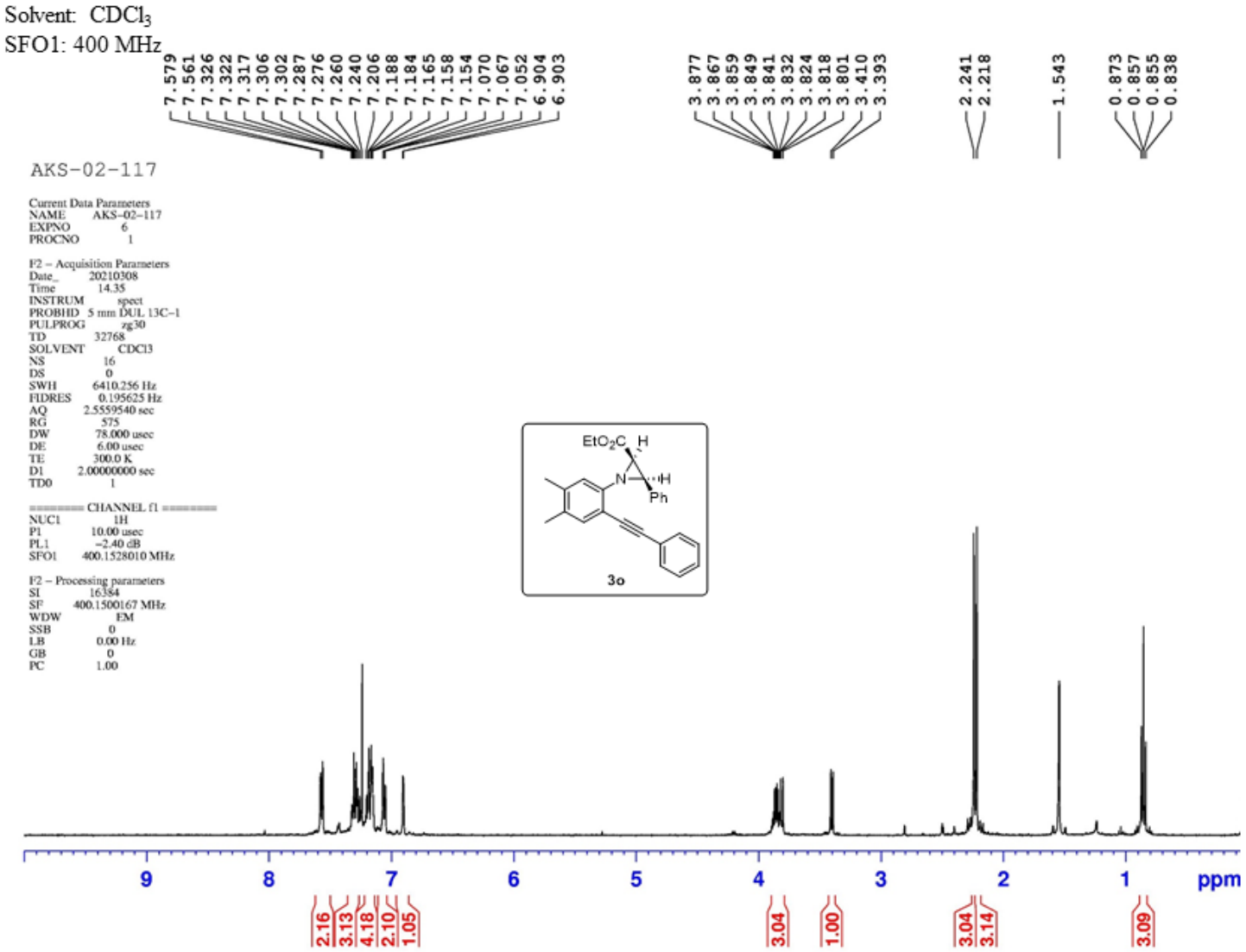
F2 - Processing parameters
SI 65536
SF 699.7431016 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

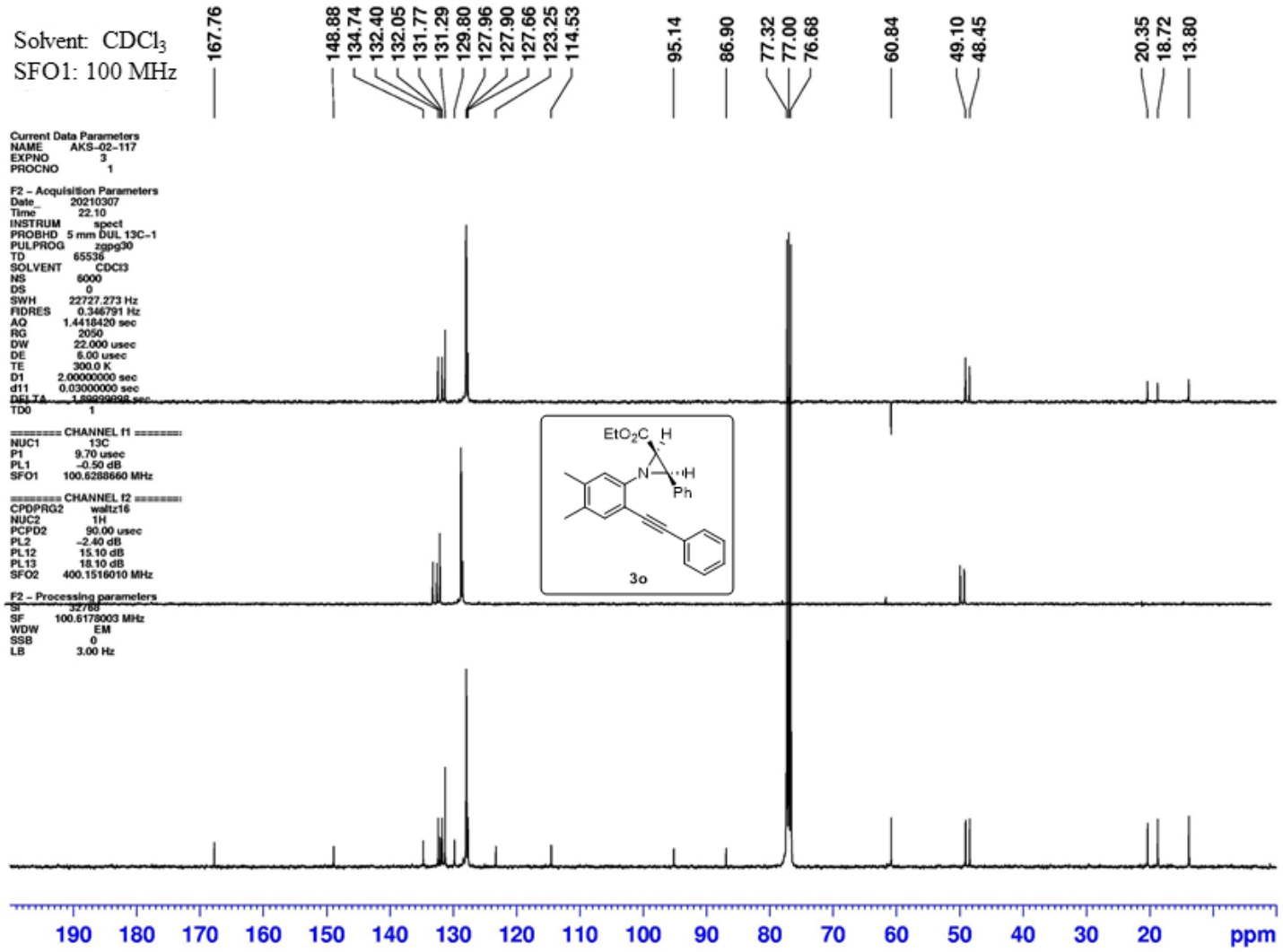


3n

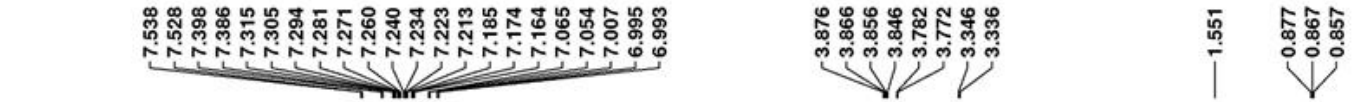



Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-127
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505411 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
167.23
151.88
134.20
132.63
131.43
129.14
128.43
128.07
128.06
128.02
127.90
127.82
122.50
121.19
117.11
96.71
84.74
77.18
77.00
76.82
61.02
48.07
46.81
13.76
EtO2C
H
N
H
Ph
Cl
3n
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Solvent: CDCl3
SFO1: 400 MHz
7.579
7.561
7.326
7.322
7.317
7.306
7.302
7.287
7.276
7.260
7.240
7.206
7.188
7.184
7.165
7.158
7.154
7.070
7.067
7.052
6.904
6.903
3.877
3.867
3.859
3.849
3.841
3.832
3.824
3.818
3.801
3.410
3.393
2.241
2.218
1.543
0.873
0.857
0.855
0.838
AKS-02-117
Current Data Parameters
NAME AKS-02-117
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210308
Time 14.35
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 575
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500167 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
EtO2C
H
N
H
Ph
3o
9
8
7
6
5
4
3
2
1
ppm
2.16
3.13
4.18
2.10
1.05
3.04
1.00
3.04
3.14
3.09

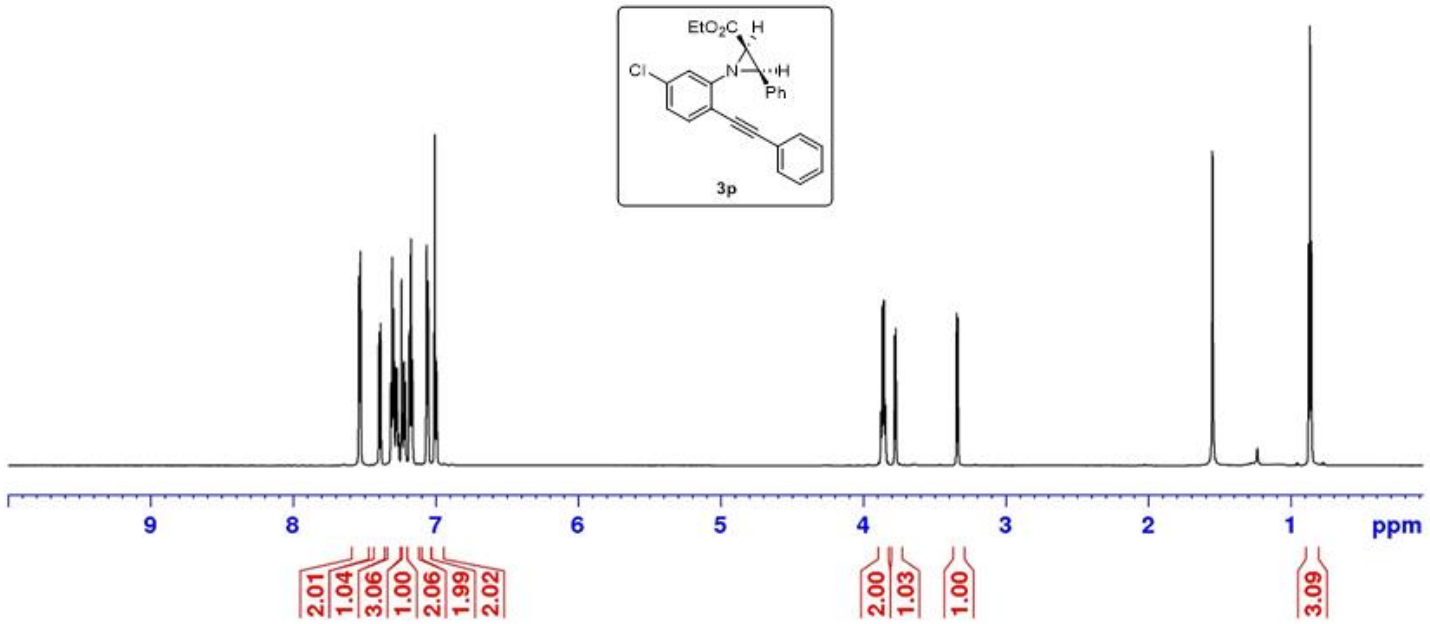

Solvent: $CDCl_3$
SFO1: 100 MHz
167.76
148.88
134.74
132.40
132.05
131.77
131.29
129.80
127.96
127.90
127.66
123.25
114.53
95.14
86.90
77.32
77.00
76.68
60.84
49.10
48.45
20.35
18.72
13.80
Current Data Parameters
NAME AKS-02-117
EXPNO 3
PROCNO 1
F2 – Acquisition Parameters
Date_ 20210307
Time 22.10
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6000
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 2050
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.59999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 – Processing parameters
SI 32768
SF 100.6178003 MHz
WDW EM
SSB 0
LB 3.00 Hz
EtO2C
H
N
H
Ph
3o
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

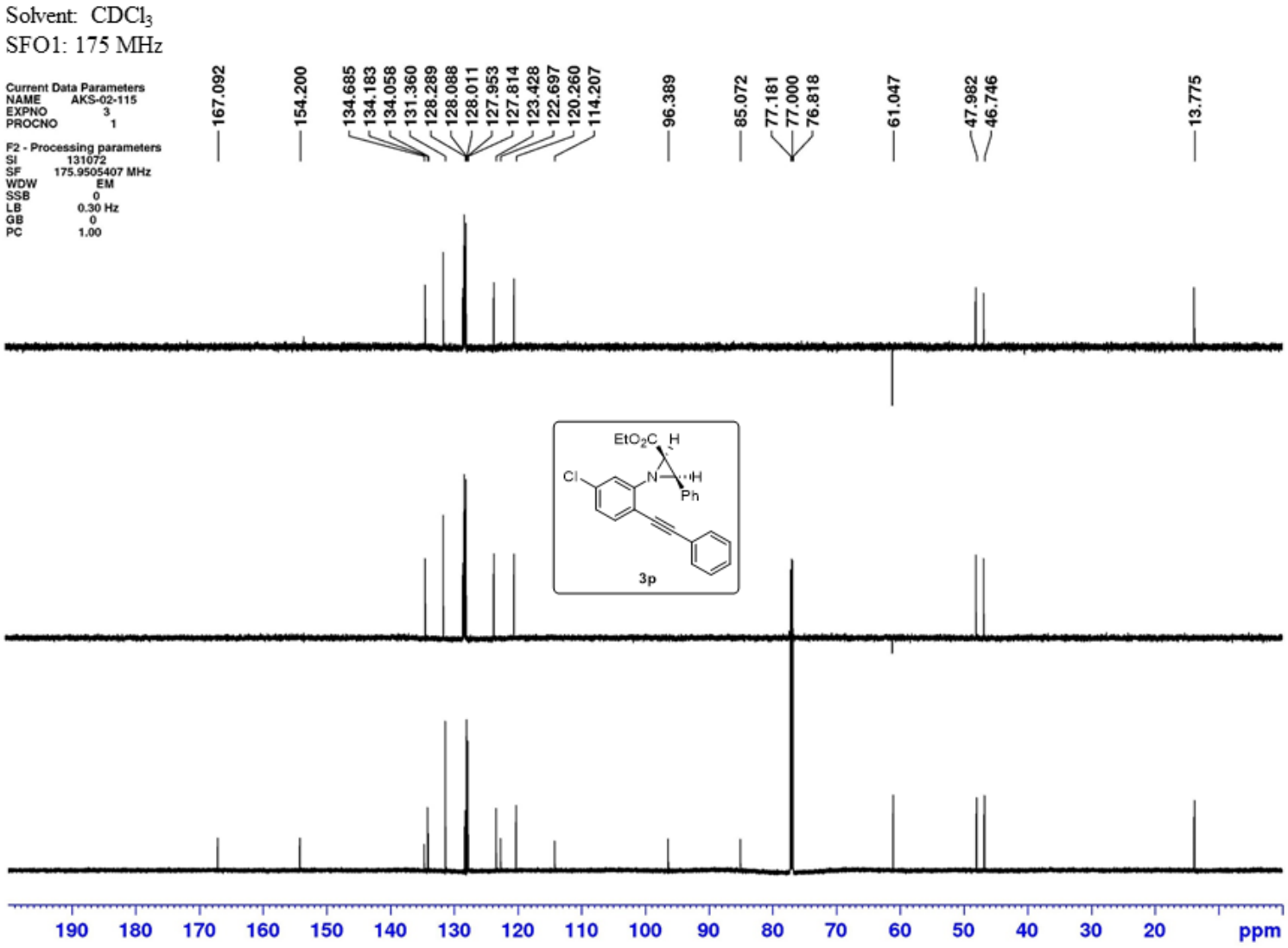
Solvent: $CDCl_3$
SFO1: 700 MHz



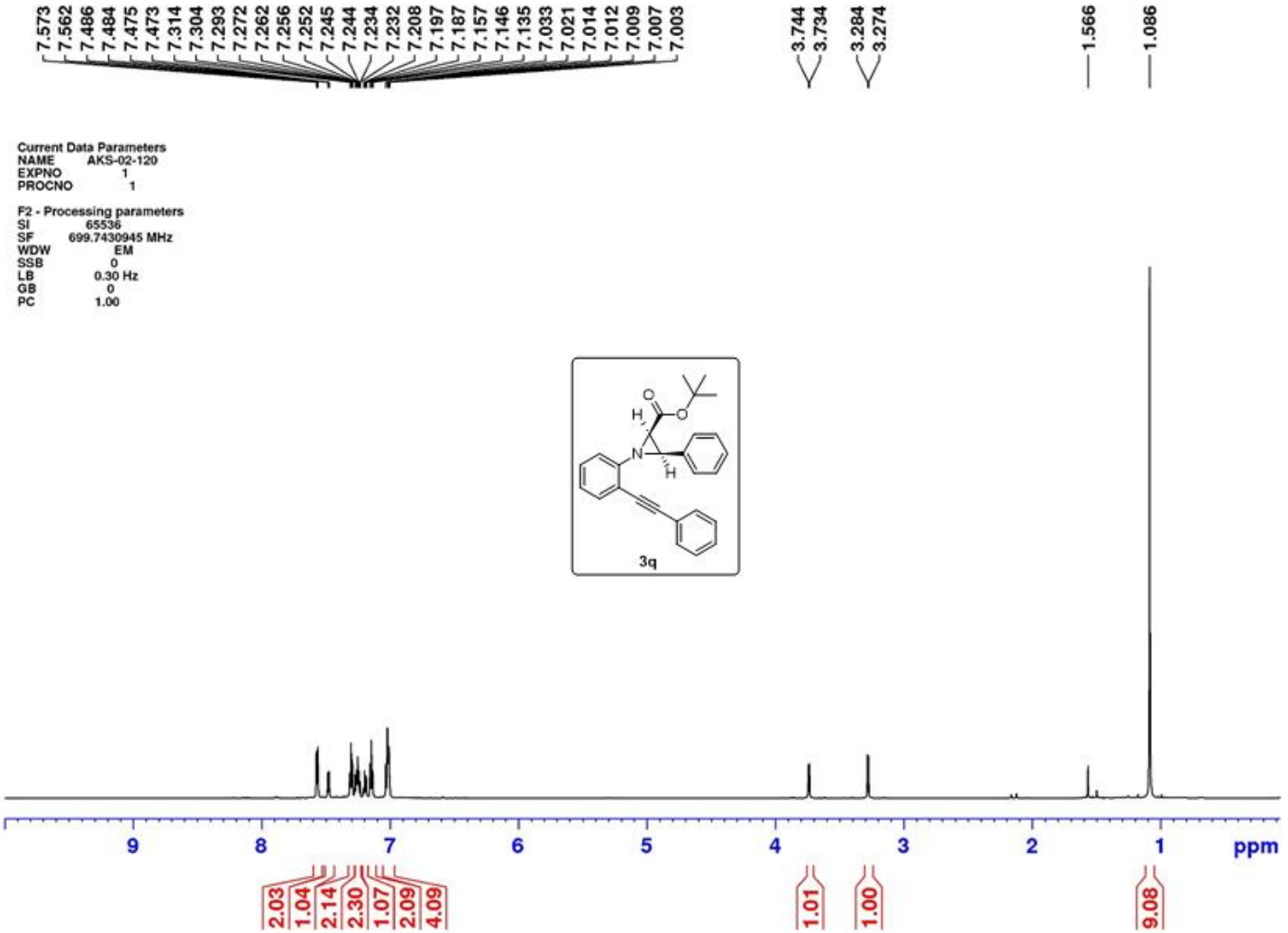
Current Data Parameters
NAME AKS-02-115
EXPNO 1
PROCNO 1

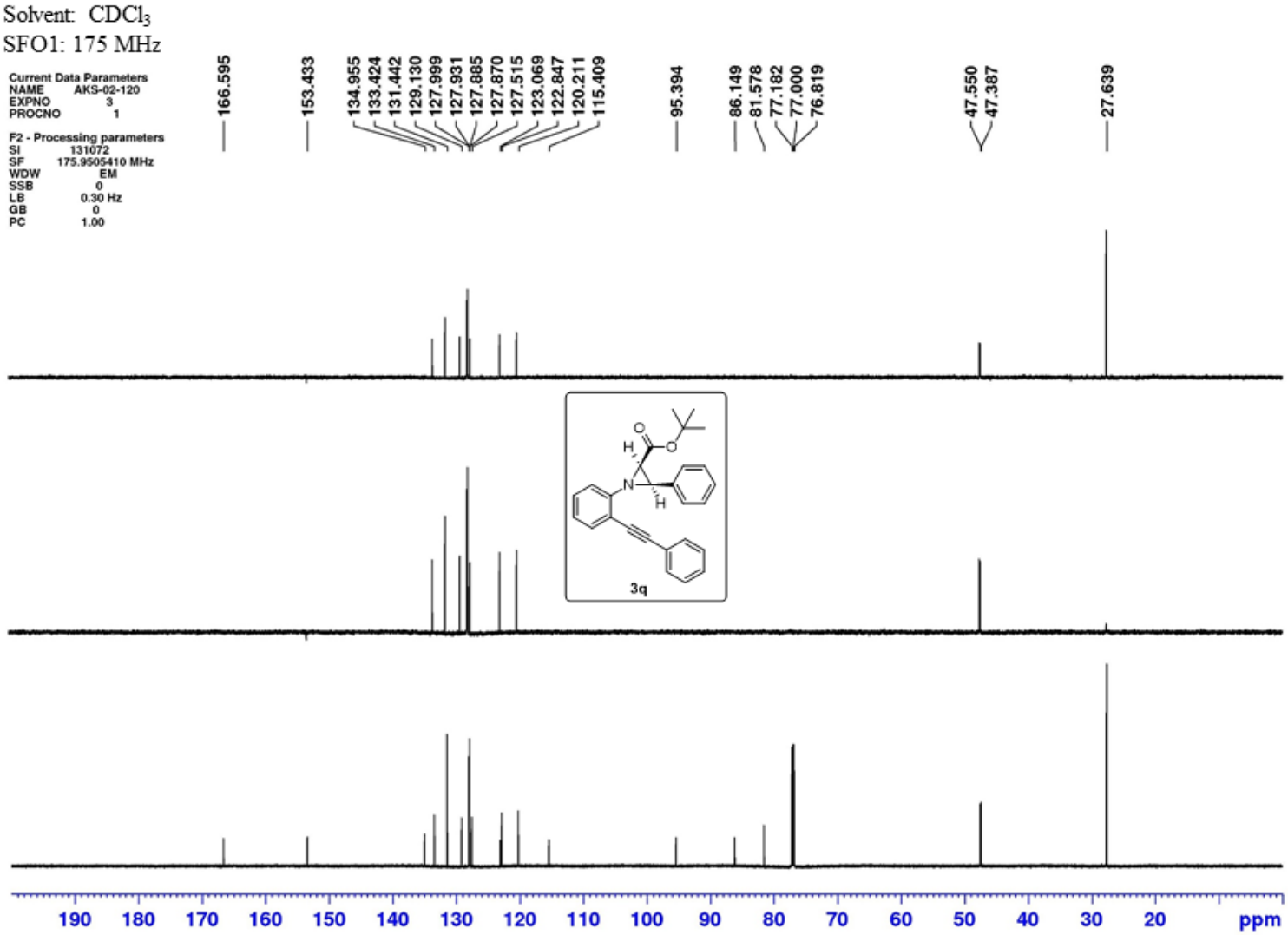
F2 - Processing parameters
SI 65536
SF 699.7431020 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



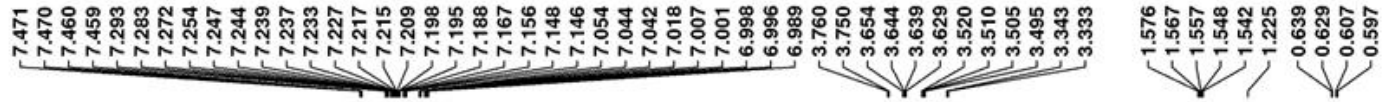

Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-115
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505407 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
167.092
154.200
134.685
134.183
134.058
131.360
128.289
128.088
128.011
127.953
127.814
123.428
122.697
120.260
114.207
96.389
85.072
77.181
77.000
76.818
61.047
47.982
46.746
13.775
EtO2C
H
N
H
Cl
Ph
3p
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Solvent: $CDCl_3$
SFO1: 700 MHz



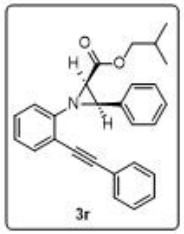

Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-120
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505410 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
166.595
153.433
134.955
133.424
131.442
129.130
127.999
127.931
127.885
127.870
127.515
123.069
122.847
120.211
115.409
95.394
86.149
81.578
77.182
77.000
76.819
47.550
47.387
27.639
3q
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

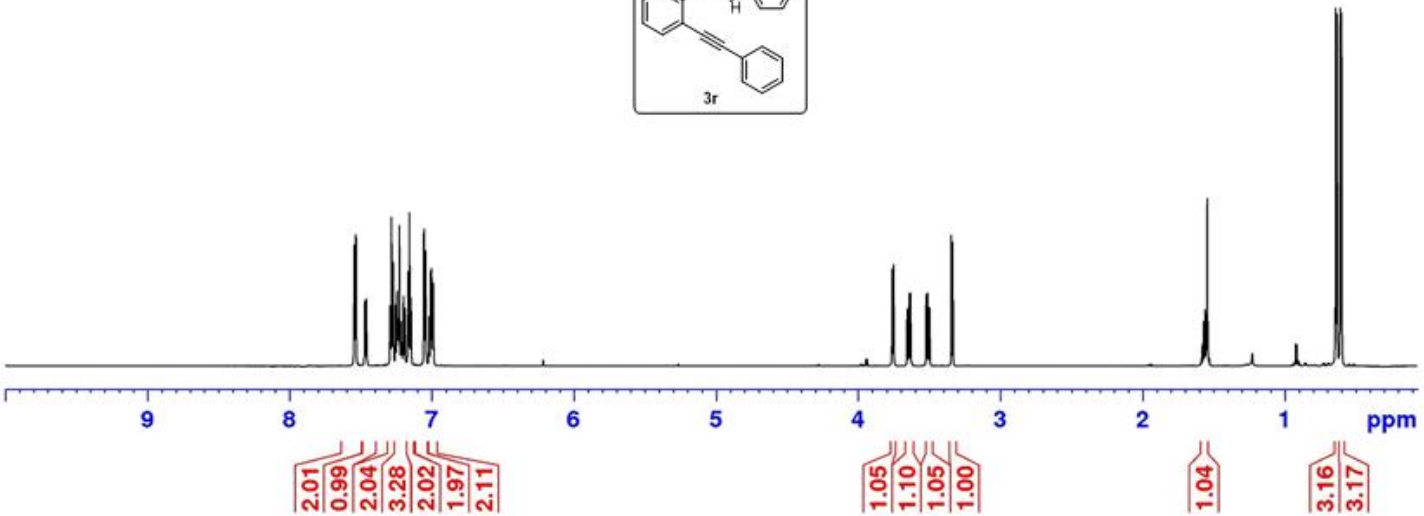
Solvent: $CDCl_3$
SFO1: 700 MHz

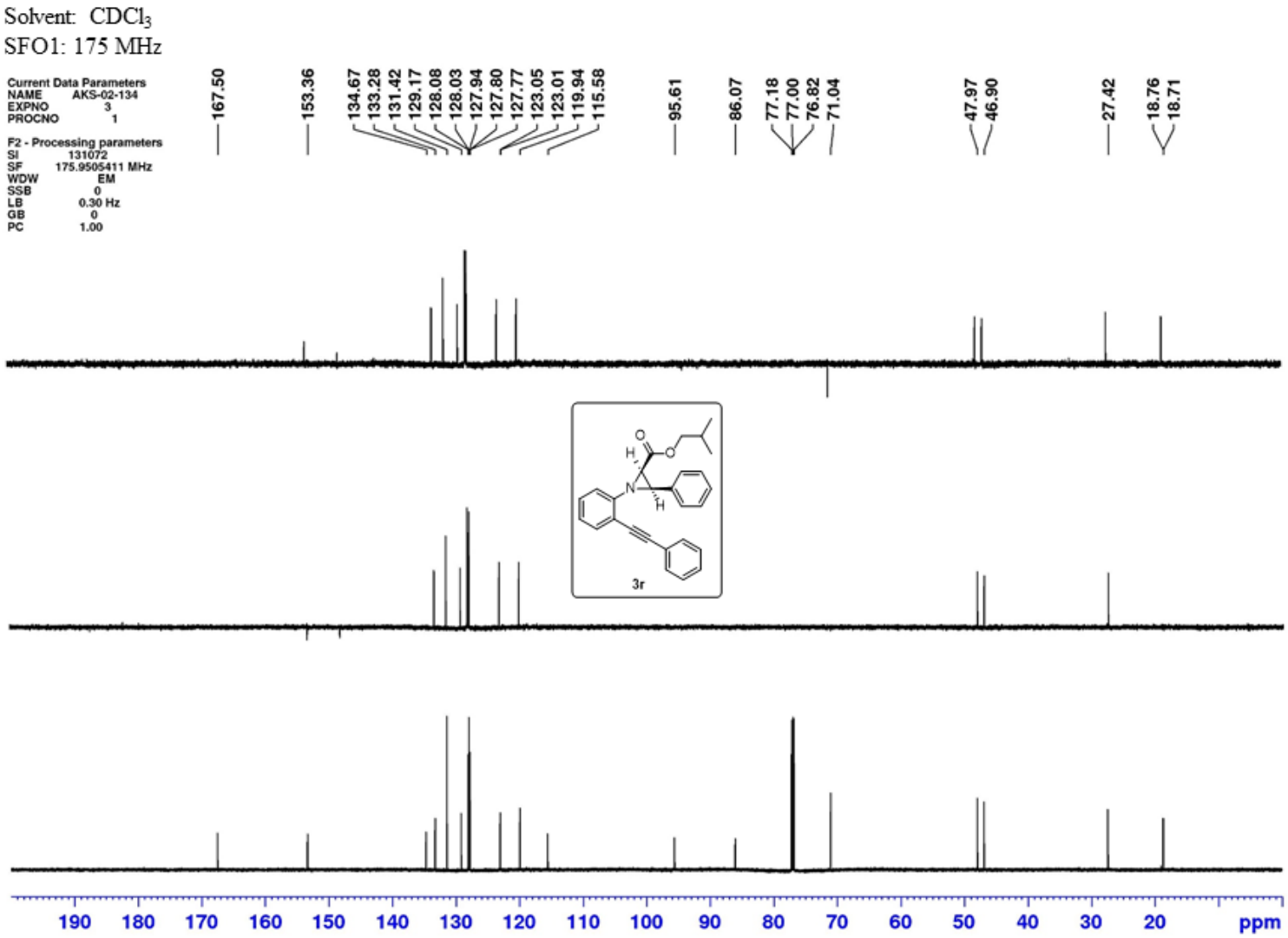


Current Data Parameters
NAME AKS-02-134
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

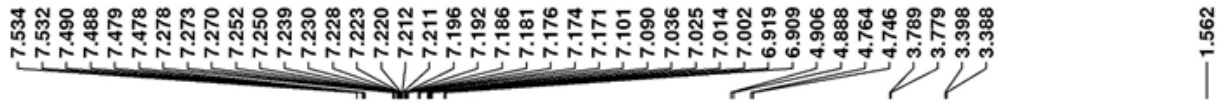





Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-134
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505411 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
167.50
153.36
134.67
133.28
131.42
129.17
128.08
128.03
127.94
127.80
127.77
123.05
123.01
119.94
115.58
95.61
86.07
77.18
77.00
76.82
71.04
47.97
46.90
27.42
18.76
18.71
3r
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

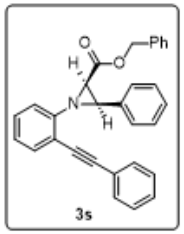
Solvent: $CDCl_3$

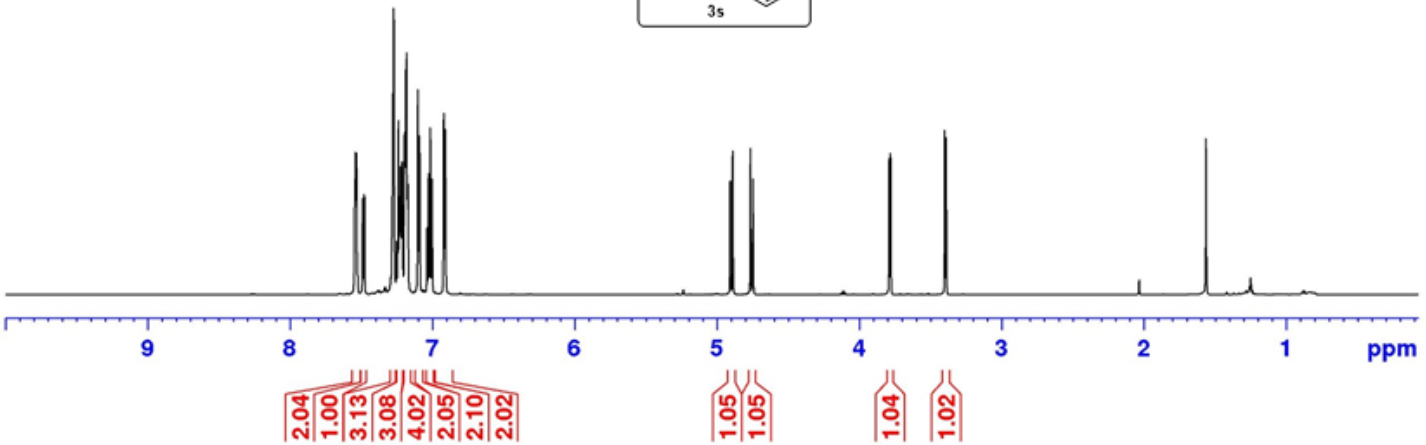
SFO1: 700 MHz



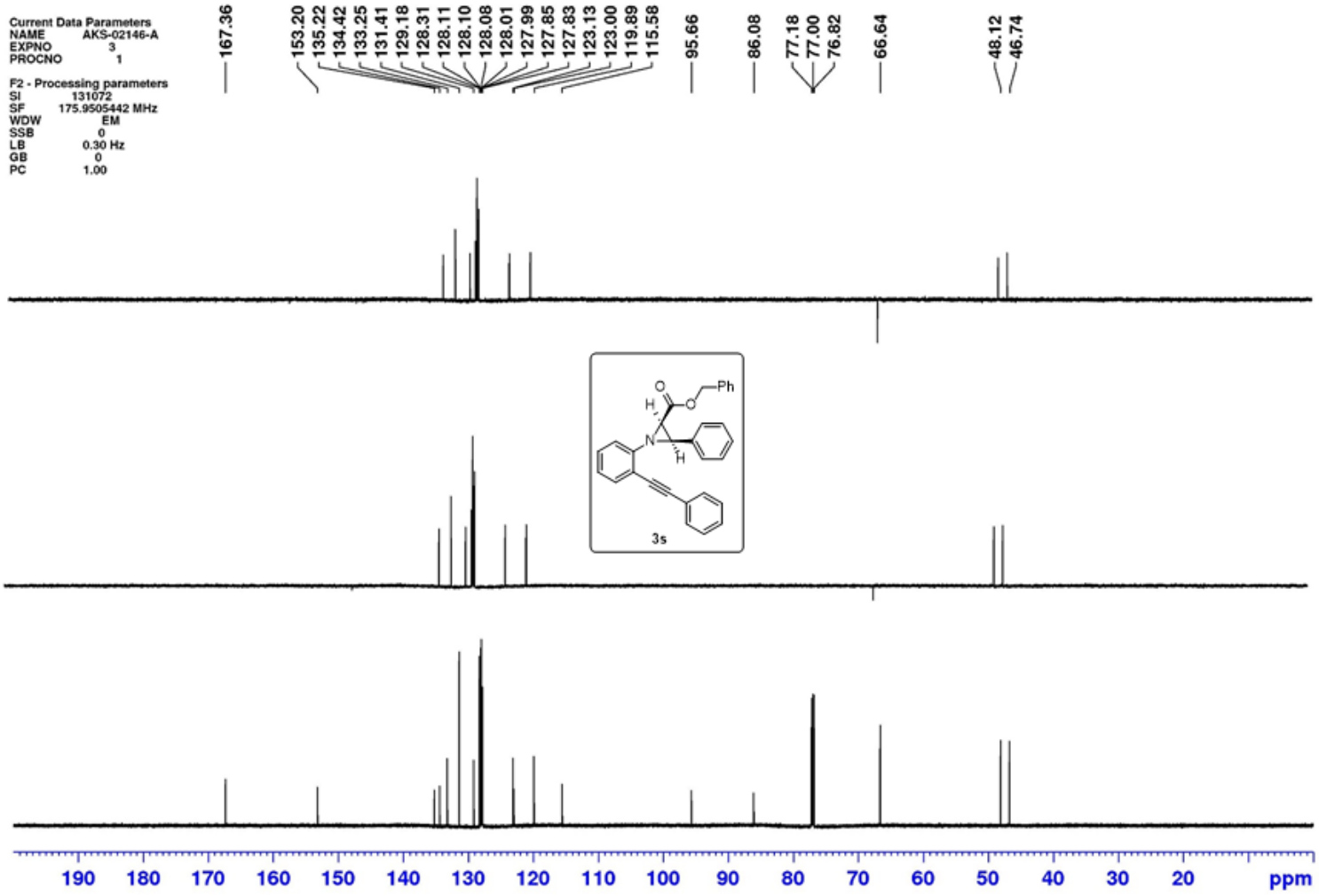
Current Data Parameters
NAME AKS-02-146-A
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7430991 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



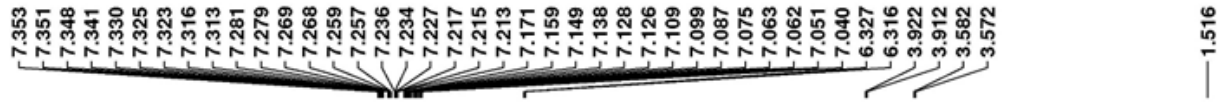


Solvent: $CDCl_3$
SFO1: 175 MHz



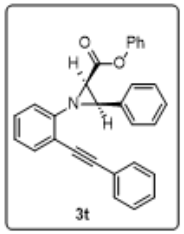
Solvent: $CDCl_3$

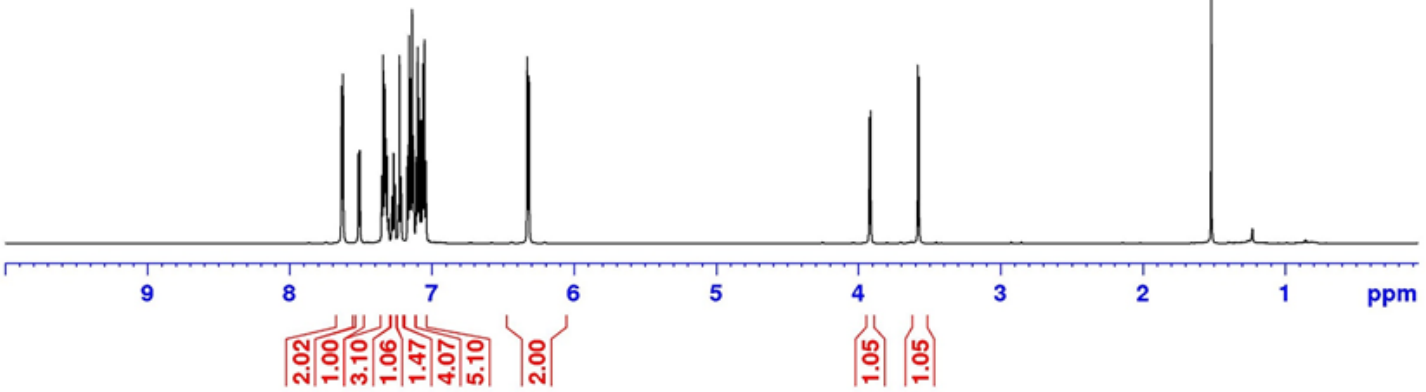
SFO1: 700 MHz

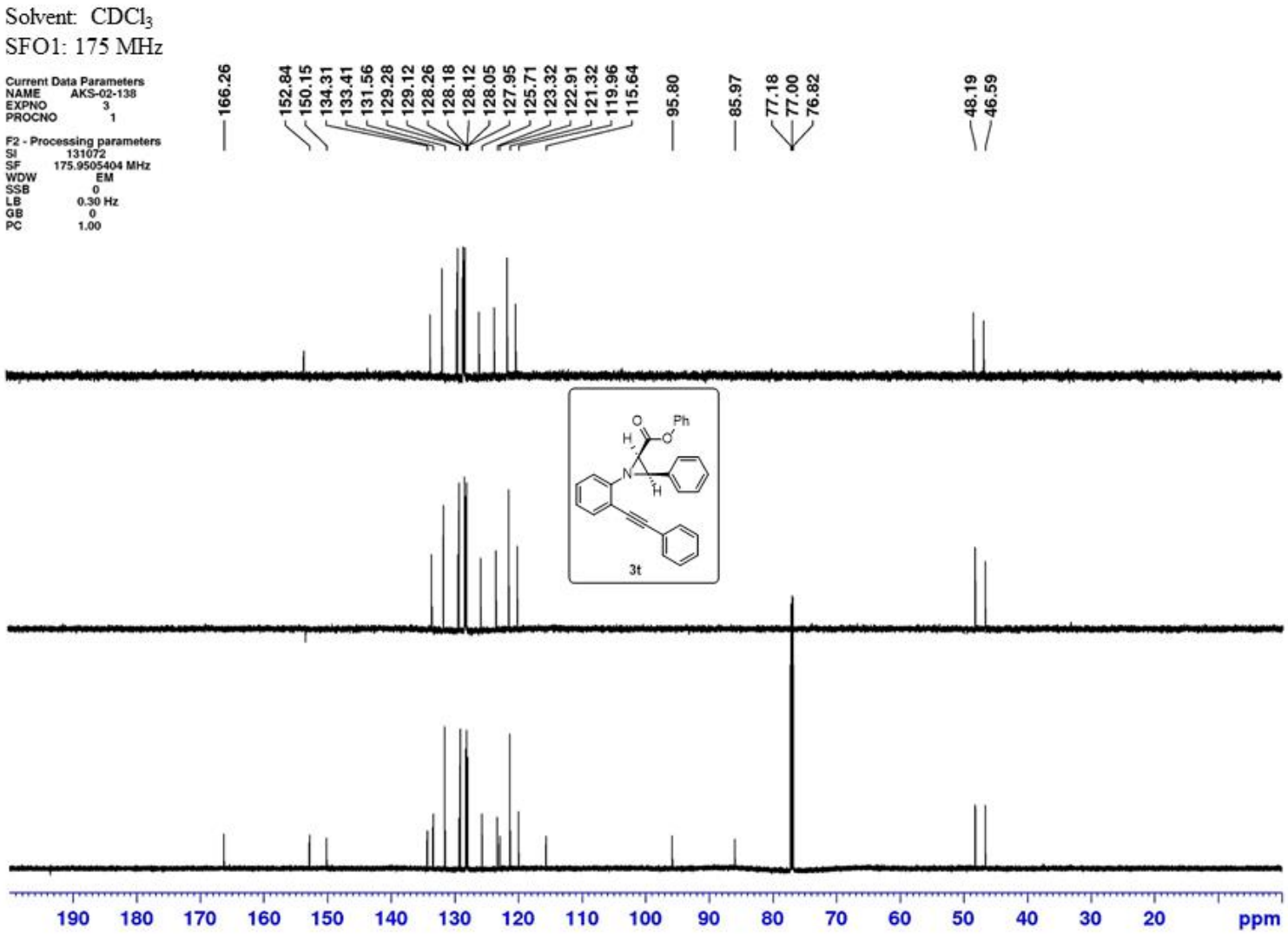


Current Data Parameters
NAME AKS-02-138
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



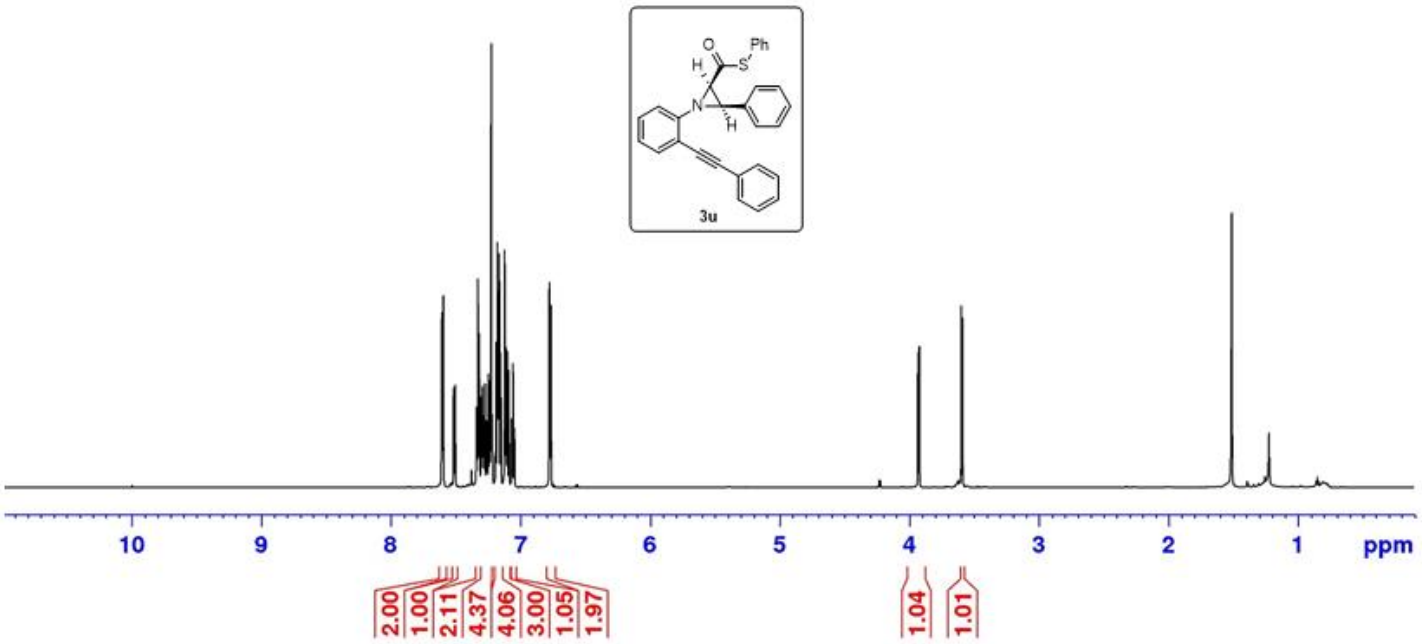


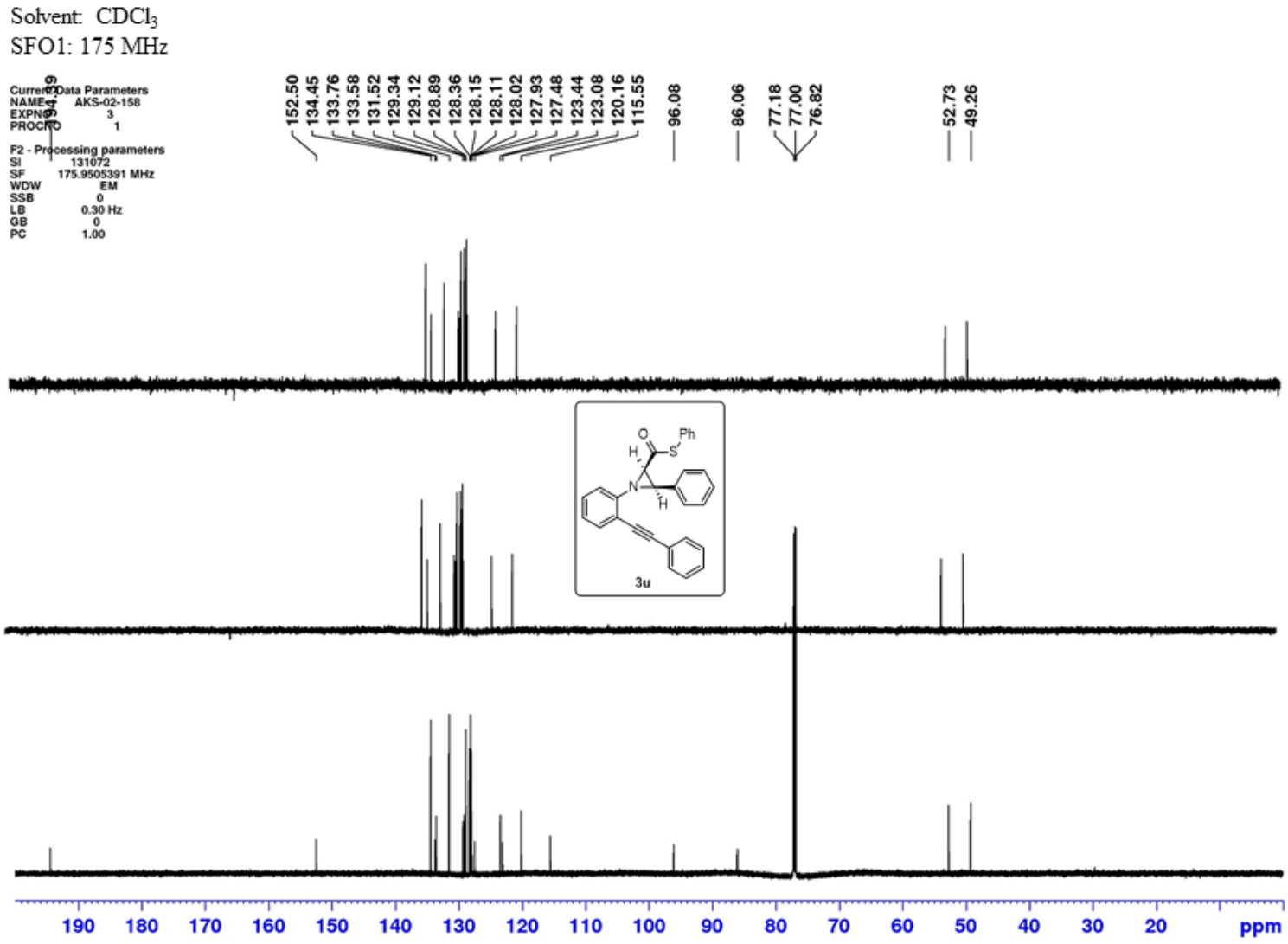

Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-138
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505404 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
166.26
152.84
150.15
134.31
133.41
131.56
129.28
129.12
128.26
128.18
128.12
128.05
127.95
125.71
123.32
122.91
121.32
119.96
115.64
95.80
85.97
77.18
77.00
76.82
48.19
46.59
Ph
3t
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Solvent: $CDCl_3$
SFO1: 700 MHz

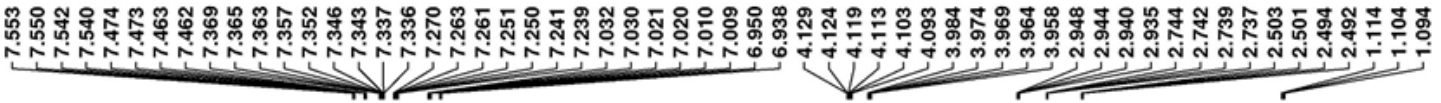
Current Data Parameters
NAME AKS-02-158-A
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



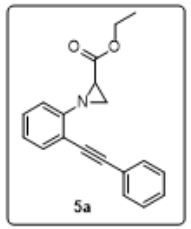

Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-158
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505391 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
152.50
134.45
133.76
133.58
131.52
129.34
129.12
128.89
128.36
128.15
128.11
128.02
127.93
127.48
123.44
123.08
120.16
115.55
96.08
86.06
77.18
77.00
76.82
52.73
49.26
3u
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

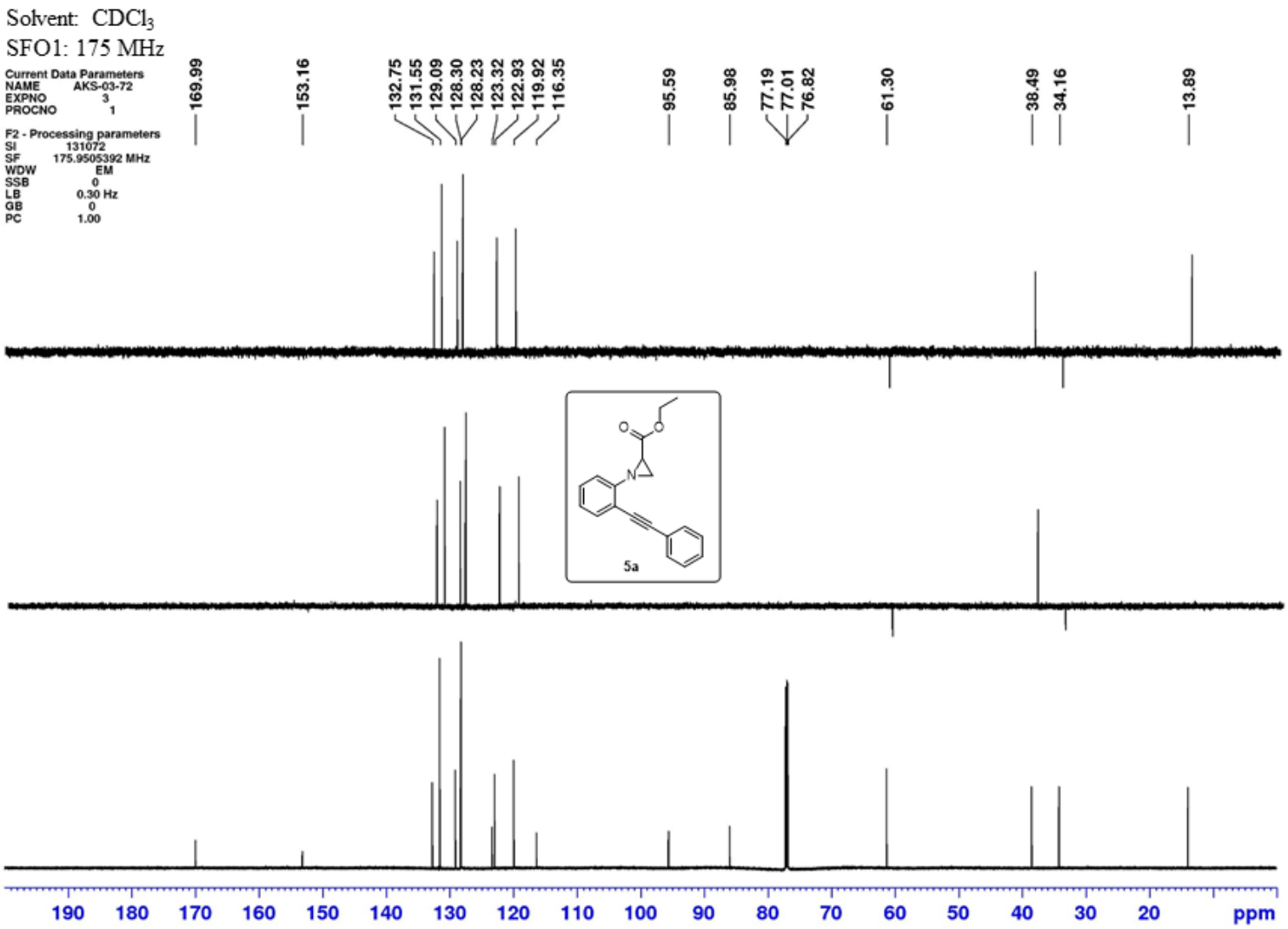
Solvent: $CDCl_3$
SFO1: 700 MHz



Current Data Parameters
NAME AKS-03-72-1H
EXPNO 1
PROCNO 1

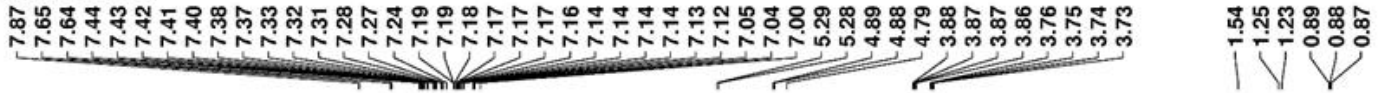
F2 - Processing parameters
SI 131072
SF 699.7430808 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-03-72
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505392 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
169.99
153.16
132.75
131.55
129.09
128.30
128.23
123.32
122.93
119.92
116.35
95.59
85.98
77.19
77.01
76.82
61.30
38.49
34.16
13.89
5a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

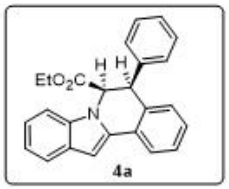
Solvent: $CDCl_3$

SFO1: 700 MHz

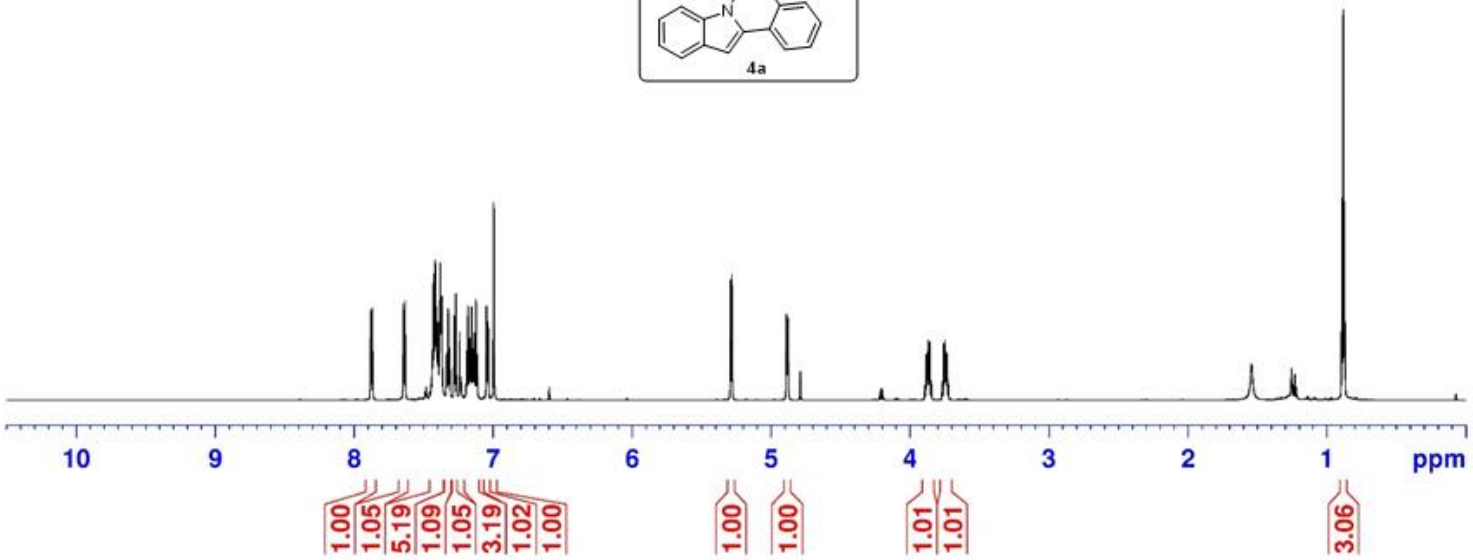


Current Data Parameters
NAME AKS–02–69
EXPNO 1
PROCNO 1

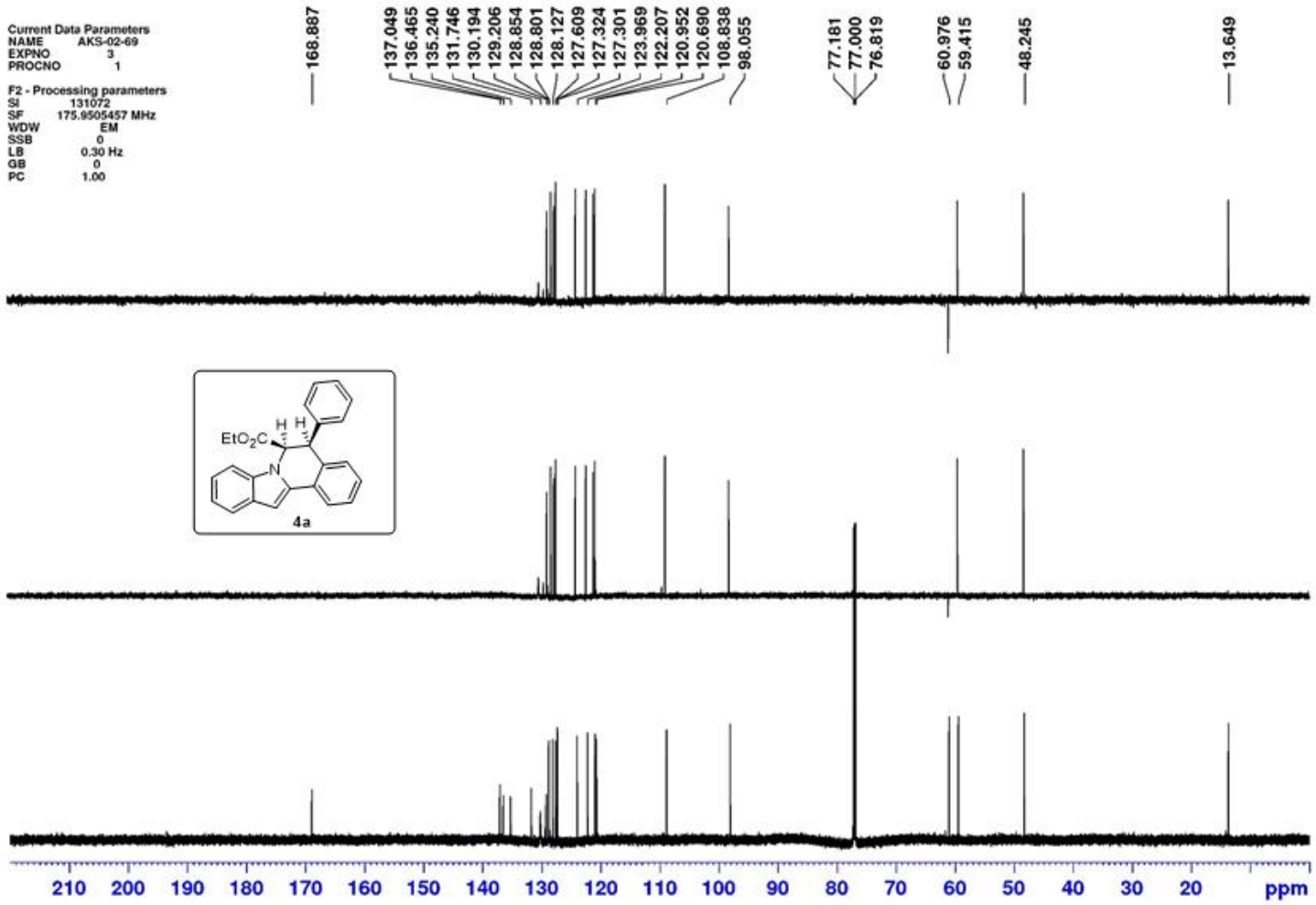
F2 – Processing parameters
SI 65536
SF 699.7438028 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4a

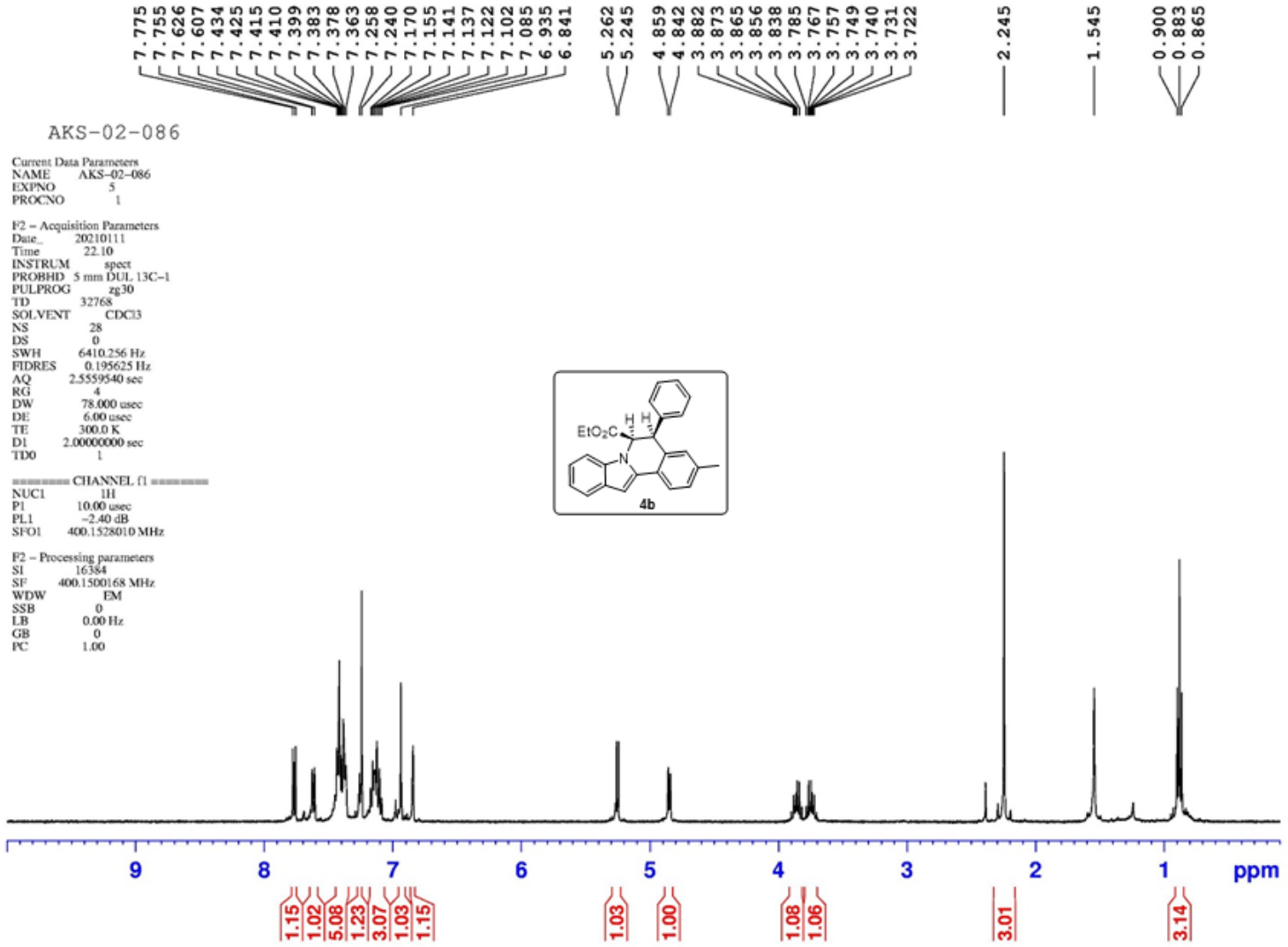


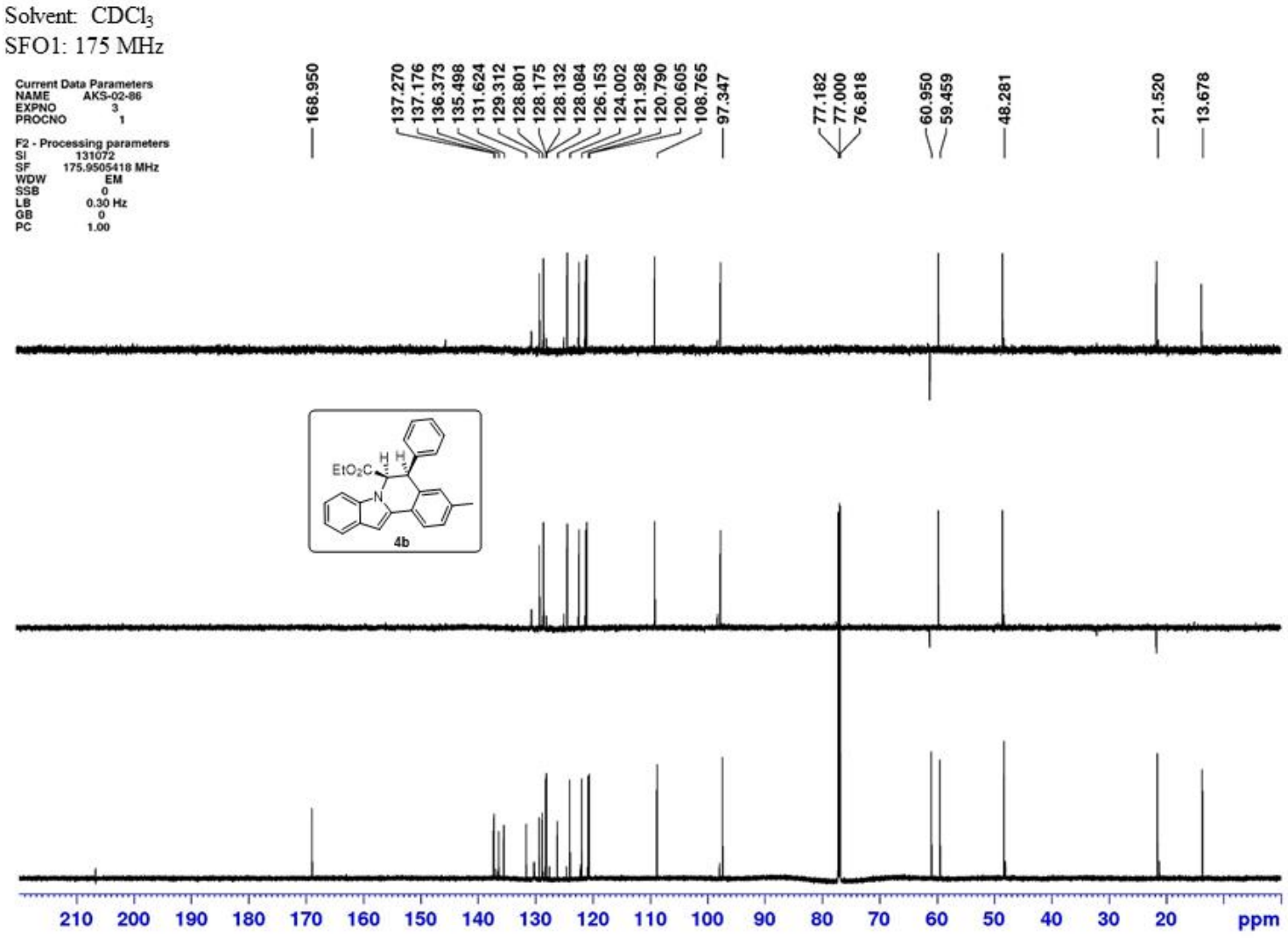
Solvent: $CDCl_3$
SFO1: 175 MHz



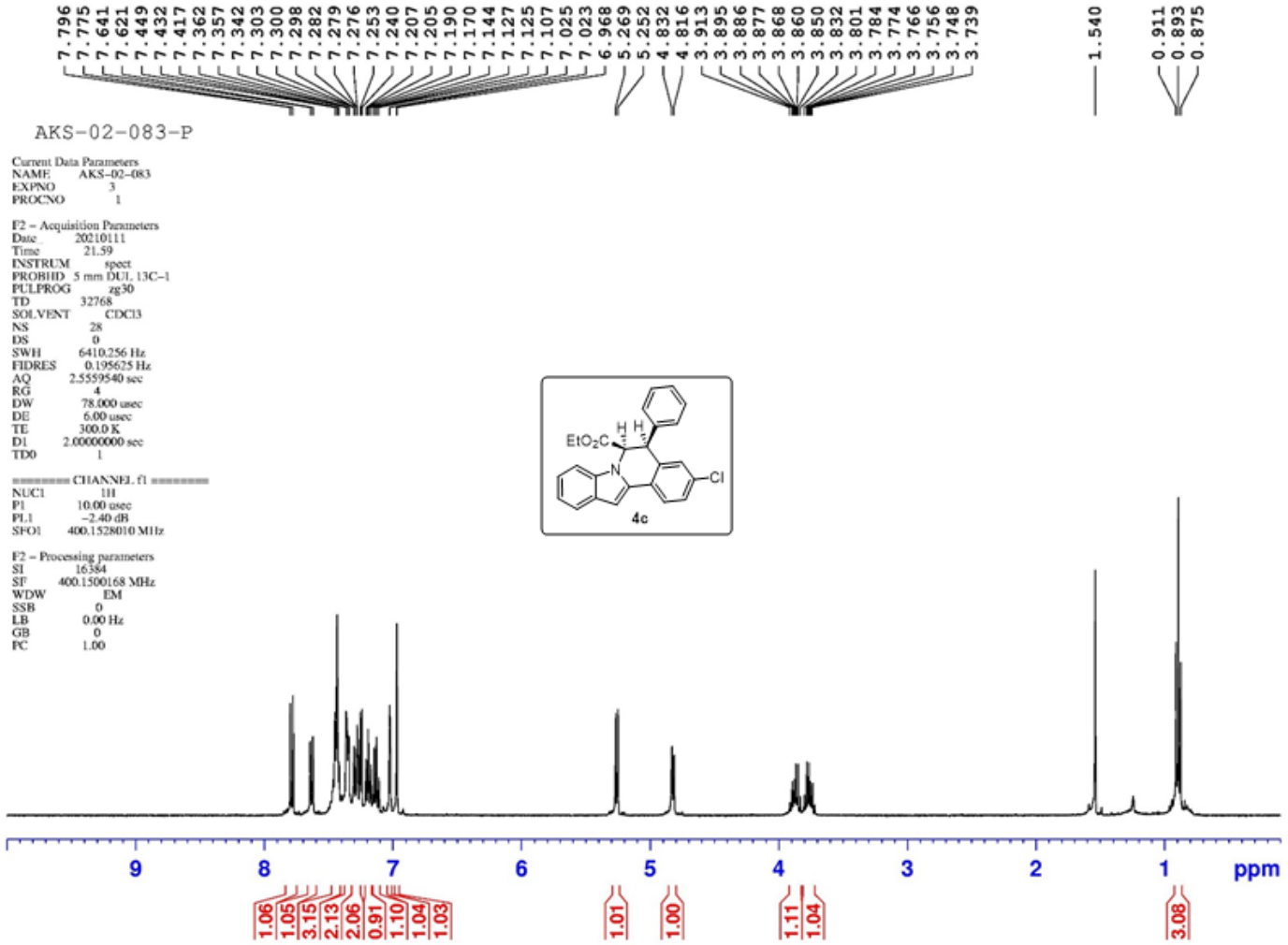
Solvent: $CDCl_3$

SFO1: 400 MHz

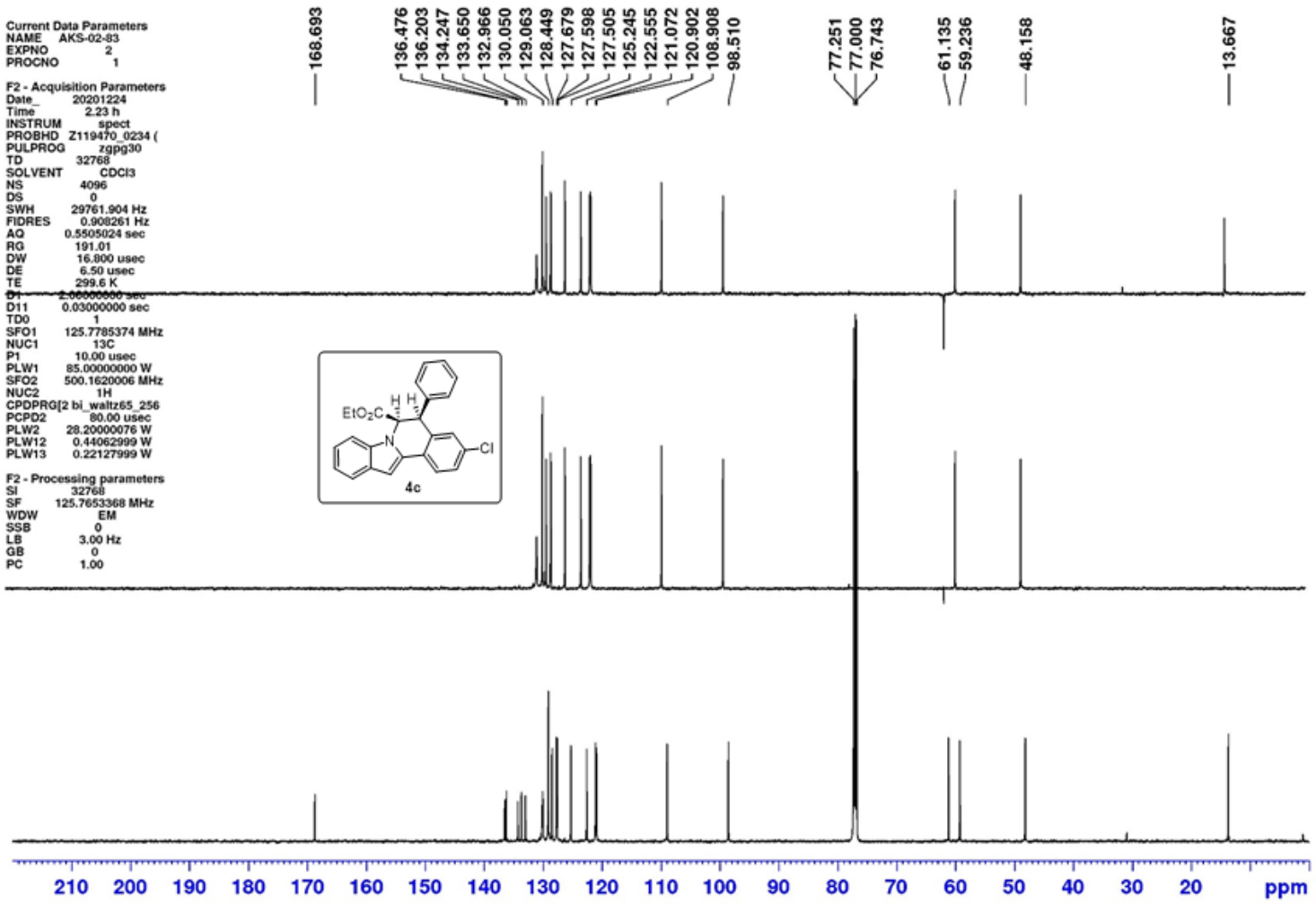



Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-86
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505418 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
168.950
137.270
137.176
136.373
135.498
131.624
129.312
128.801
128.175
128.132
128.084
126.153
124.002
121.928
120.790
120.605
108.765
97.347
77.182
77.000
76.818
60.950
59.459
48.281
21.520
13.678
EtO2C
H
H
N
4b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

Solvent: $CDCl_3$
SFO1: 400 MHz

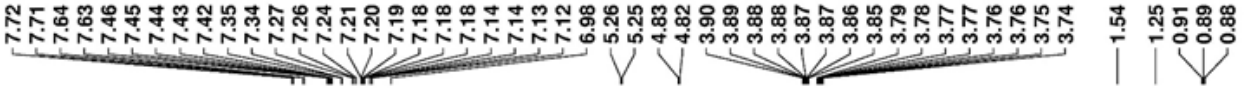


Solvent: $CDCl_3$
SFO1: 125 MHz



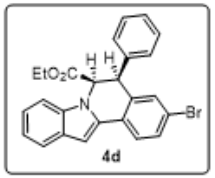
Solvent: $CDCl_3$

SFO1: 700 MHz

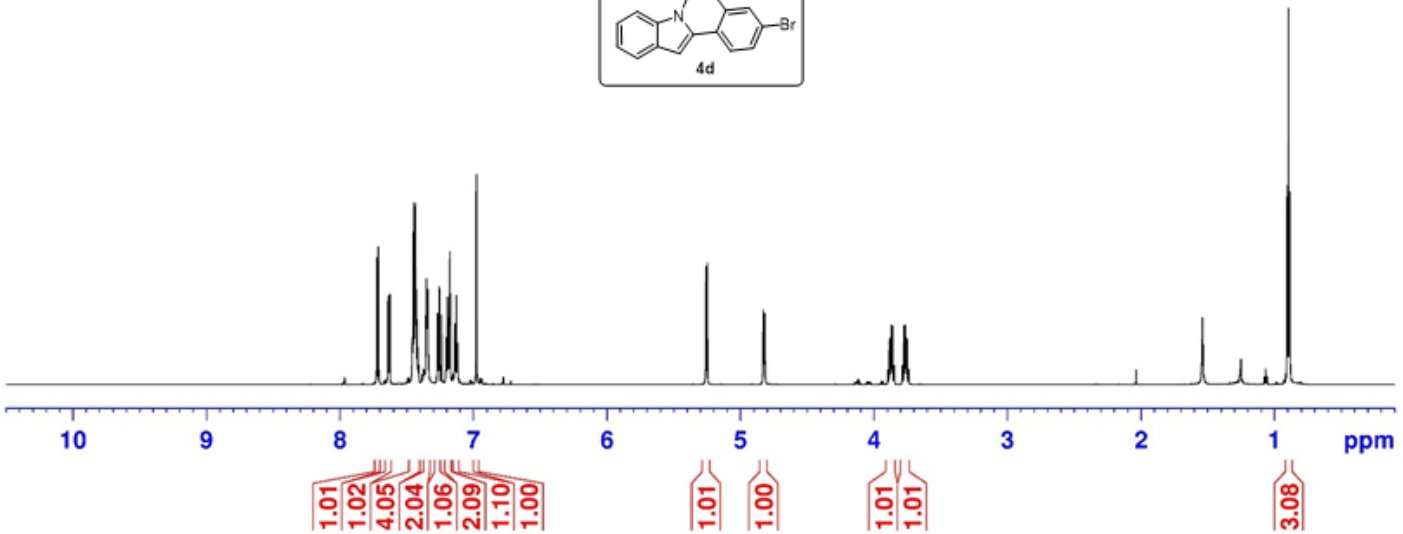


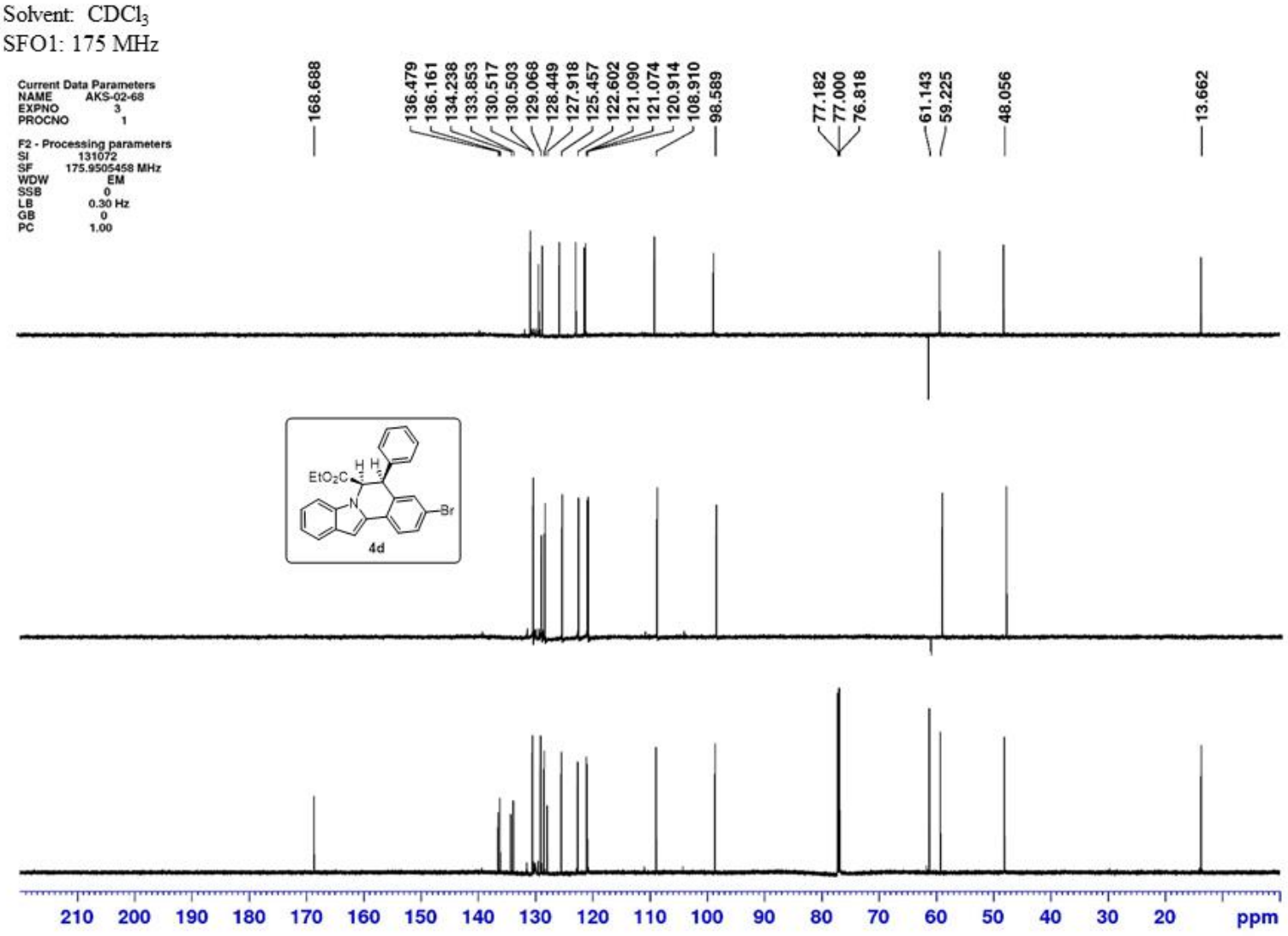
Current Data Parameters
NAME AKS–02–68
EXPNO 1
PROCNO 1

F2 – Processing parameters
SI 65536
SF 699.7438026 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



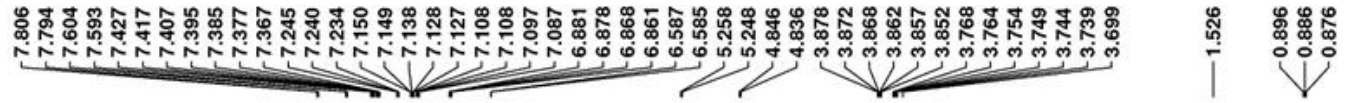
4d




Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-68
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505458 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
168.688
136.479
136.161
134.238
133.853
130.517
130.503
129.068
128.449
127.918
125.457
122.602
121.090
121.074
120.914
108.910
98.589
77.182
77.000
76.818
61.143
59.225
48.056
13.662
EtO2C
H
H
N
Br
4d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

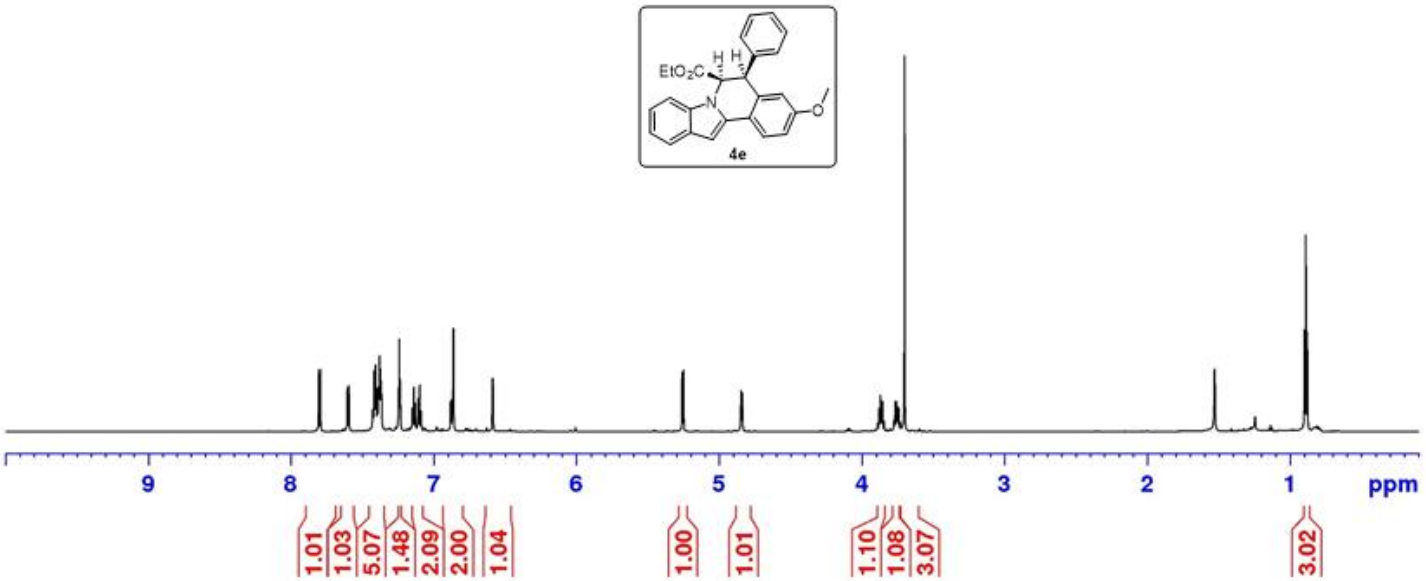
Solvent: $CDCl_3$

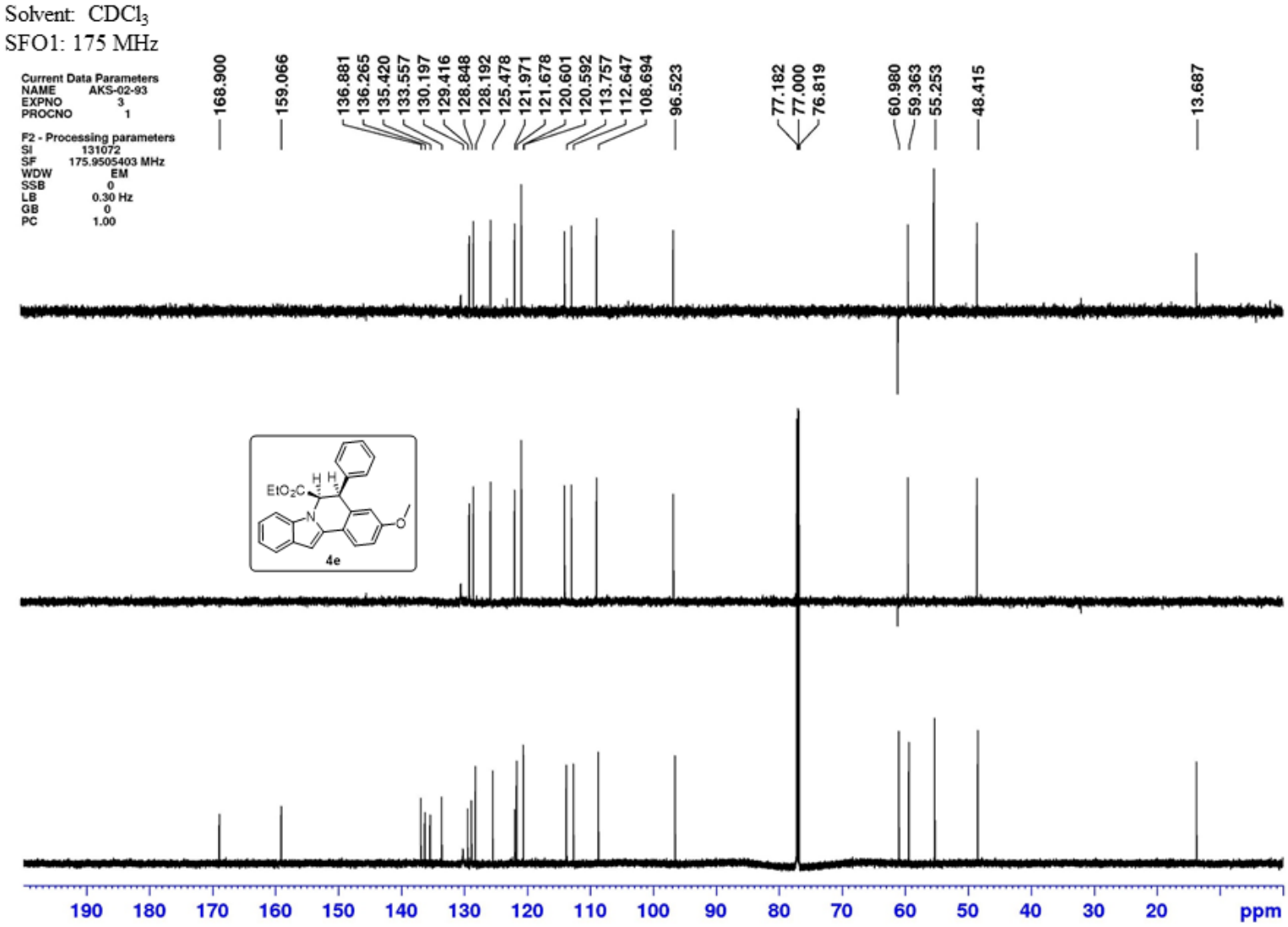
SFO1: 700 MHz



Current Data Parameters
NAME AKS-02-93
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7430995 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

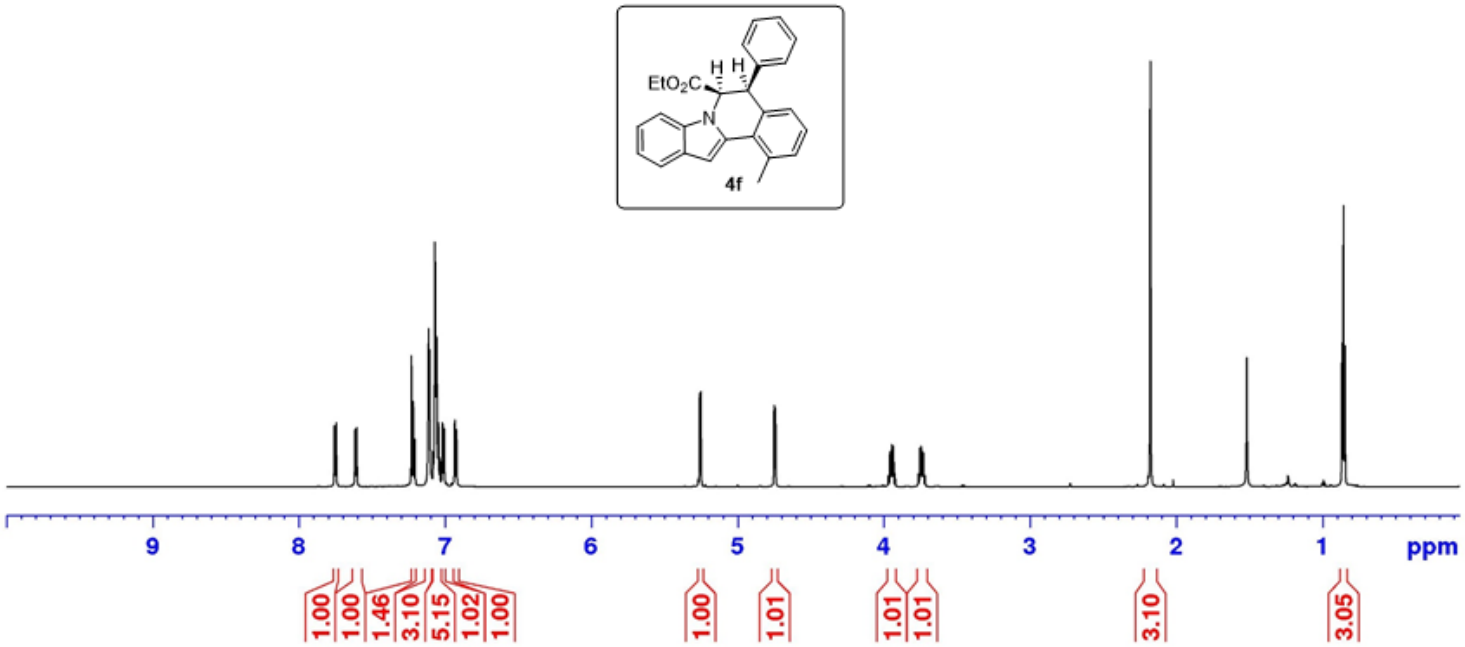


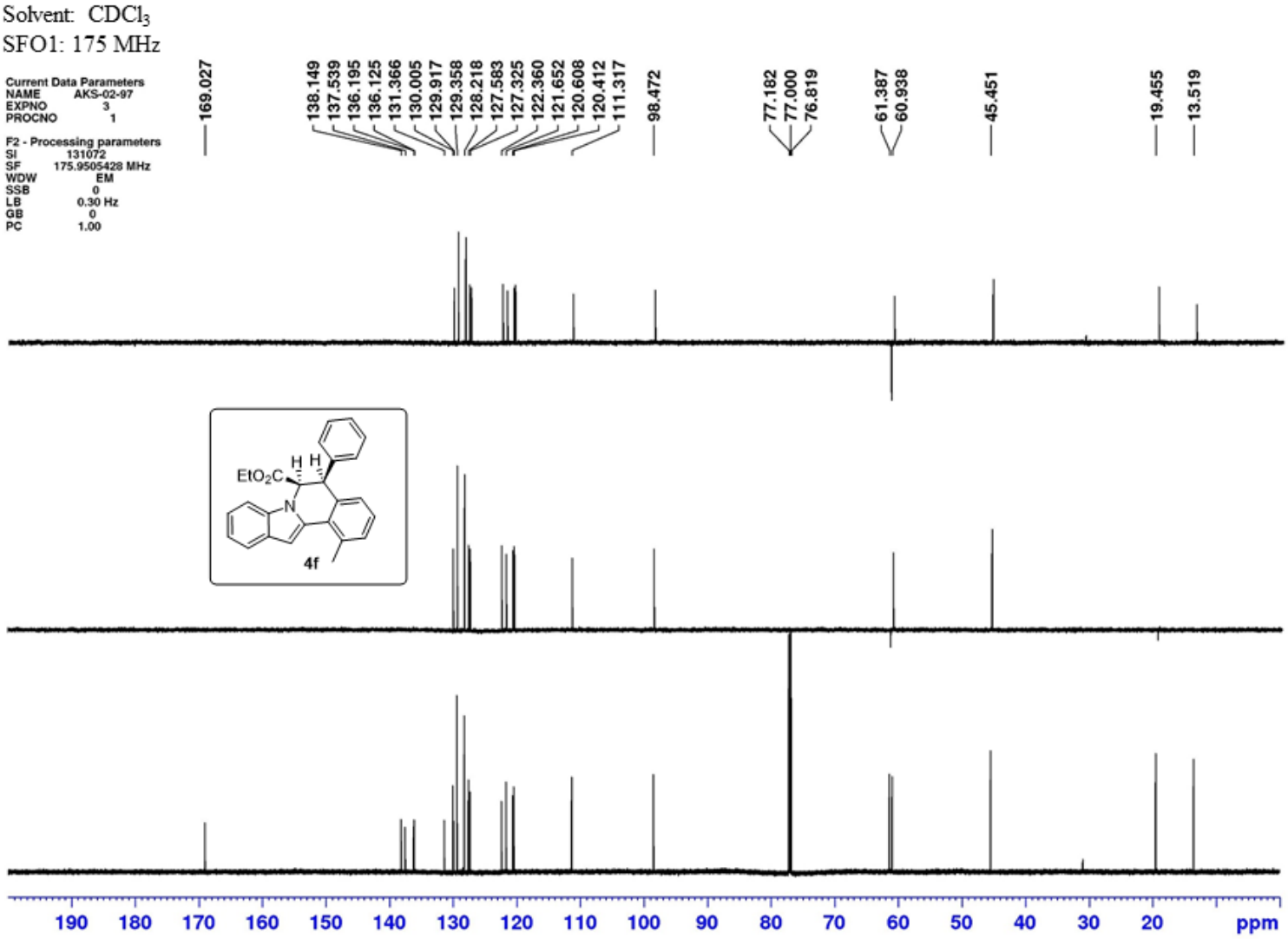

Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-93
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505403 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
168.900
159.066
136.881
136.265
135.420
133.557
130.197
129.416
128.848
128.192
125.478
121.971
121.678
120.601
120.592
113.757
112.647
108.694
96.523
77.182
77.000
76.819
60.980
59.363
55.253
48.415
13.687
EtO2C
H
H
N
O
4e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Solvent: $CDCl_3$
SFO1: 700 MHz

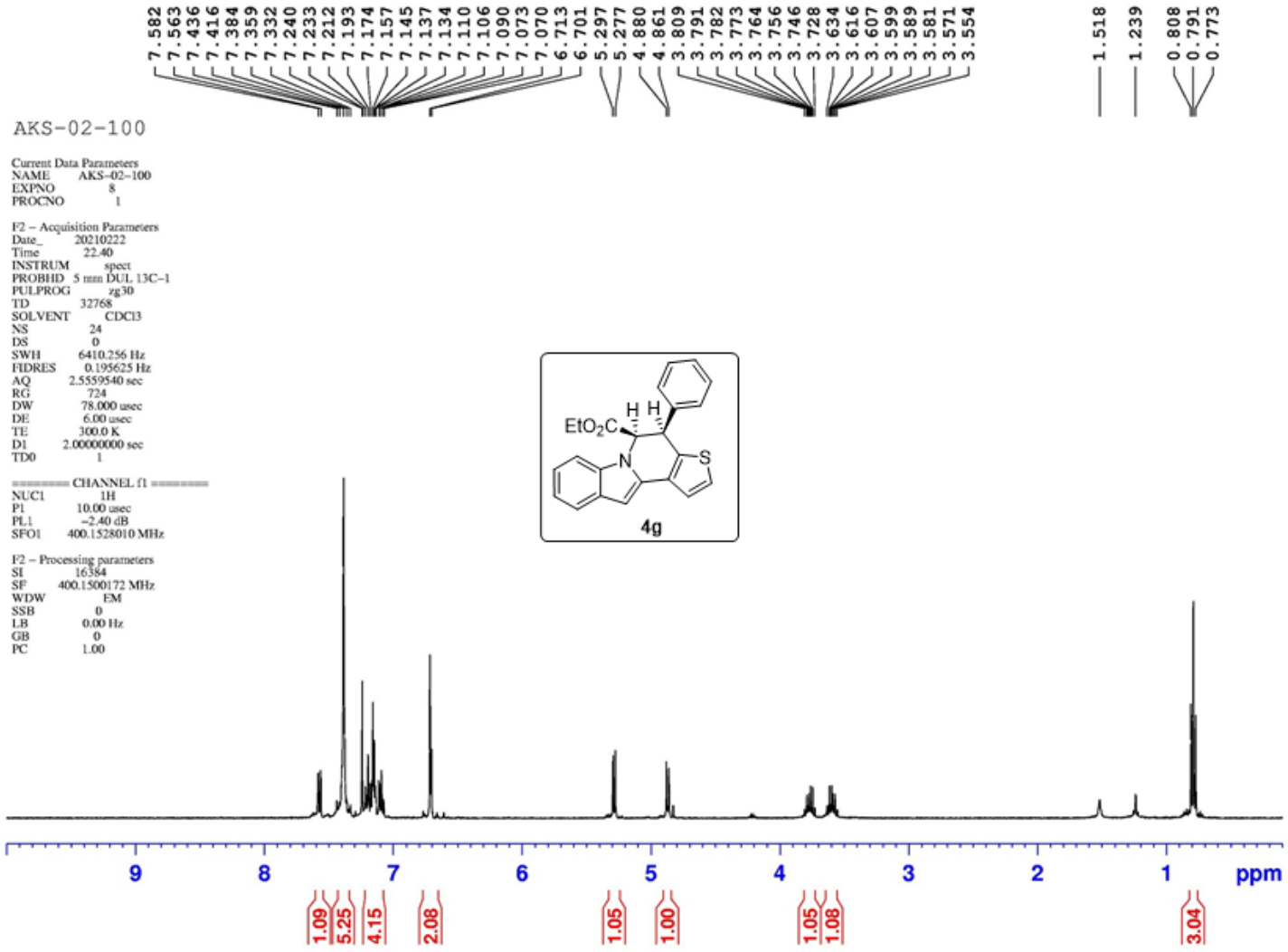
Current Data Parameters
NAME AKS-02-97
EXPNO 1
PROCNO 1

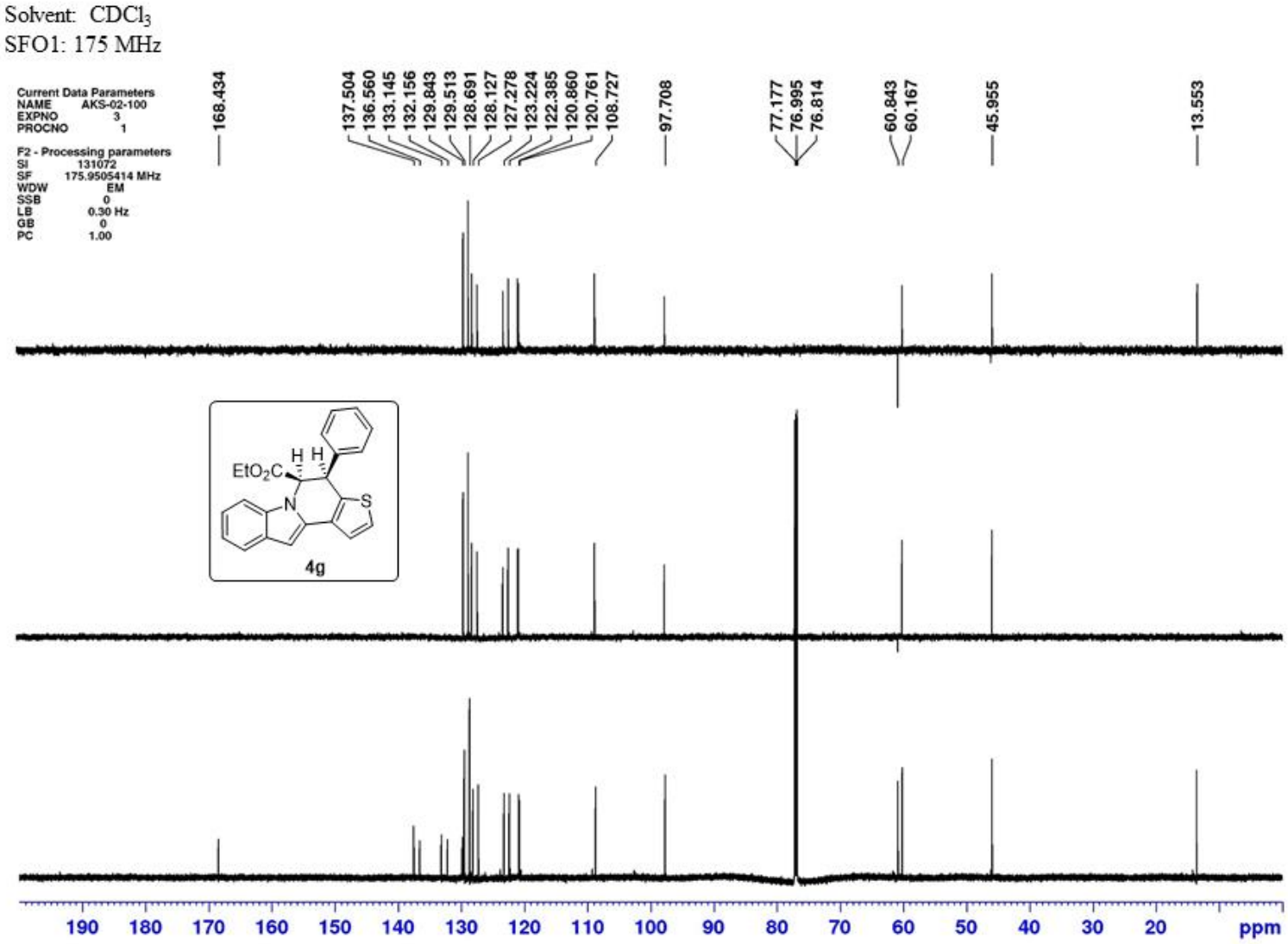
F2 - Processing parameters
SI 65536
SF 699.7431083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-97
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505428 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
169.027
138.149
137.539
136.195
136.125
131.366
130.005
129.917
129.358
128.218
127.583
127.325
122.360
121.652
120.608
120.412
111.317
98.472
77.182
77.000
76.819
61.387
60.938
45.451
19.455
13.519
EtO_2C
H
H
N
4f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Solvent: $CDCl_3$
SFO1: 400 MHz




Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-100
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505414 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
168.434
137.504
136.560
133.145
132.156
129.843
129.513
128.691
128.127
127.278
123.224
122.385
120.860
120.761
108.727
97.708
77.177
76.995
76.814
60.843
60.167
45.955
13.553
EtO2C
H H
N
S
4g
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

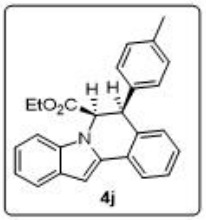
Solvent: $CDCl_3$

SFO1: 700 MHz

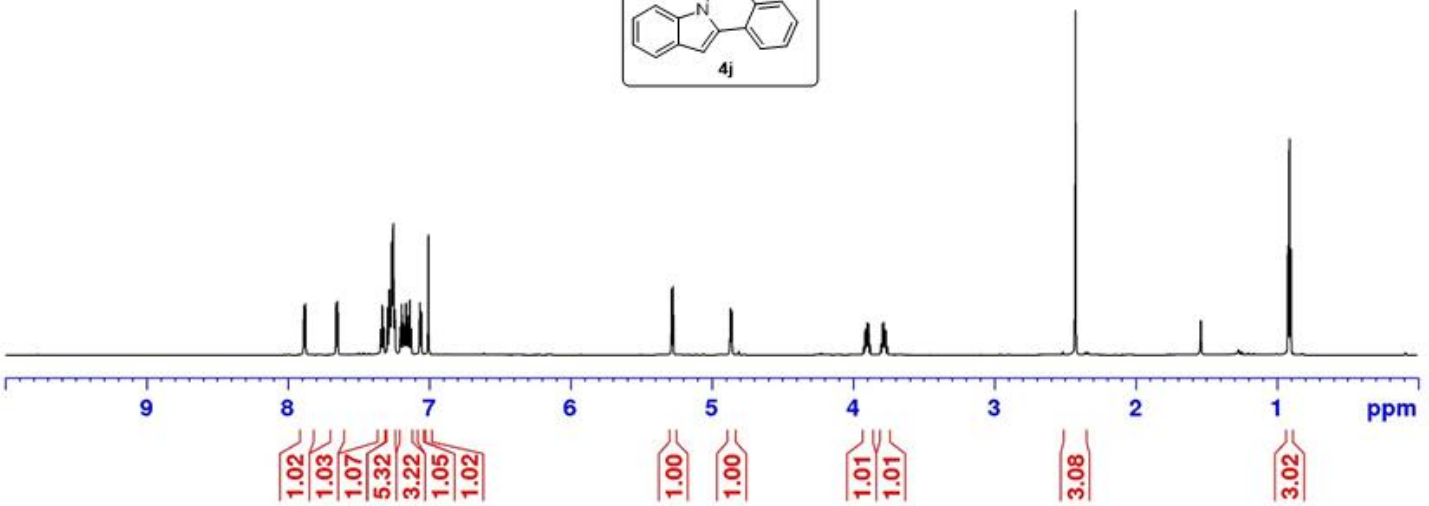
7.887 7.876 7.659 7.648 7.342 7.332 7.321 7.294 7.283 7.276 7.265 7.261 7.254 7.243 7.205 7.194 7.183 7.175 7.164 7.154 7.148 7.137 7.126 7.066 7.055 7.005 5.283 5.273 4.867 4.857 3.914 3.908 3.904 3.898 3.893 3.888 3.791 3.786 3.781 3.776 3.771 3.766 2.424 1.537 0.920 0.909 0.899

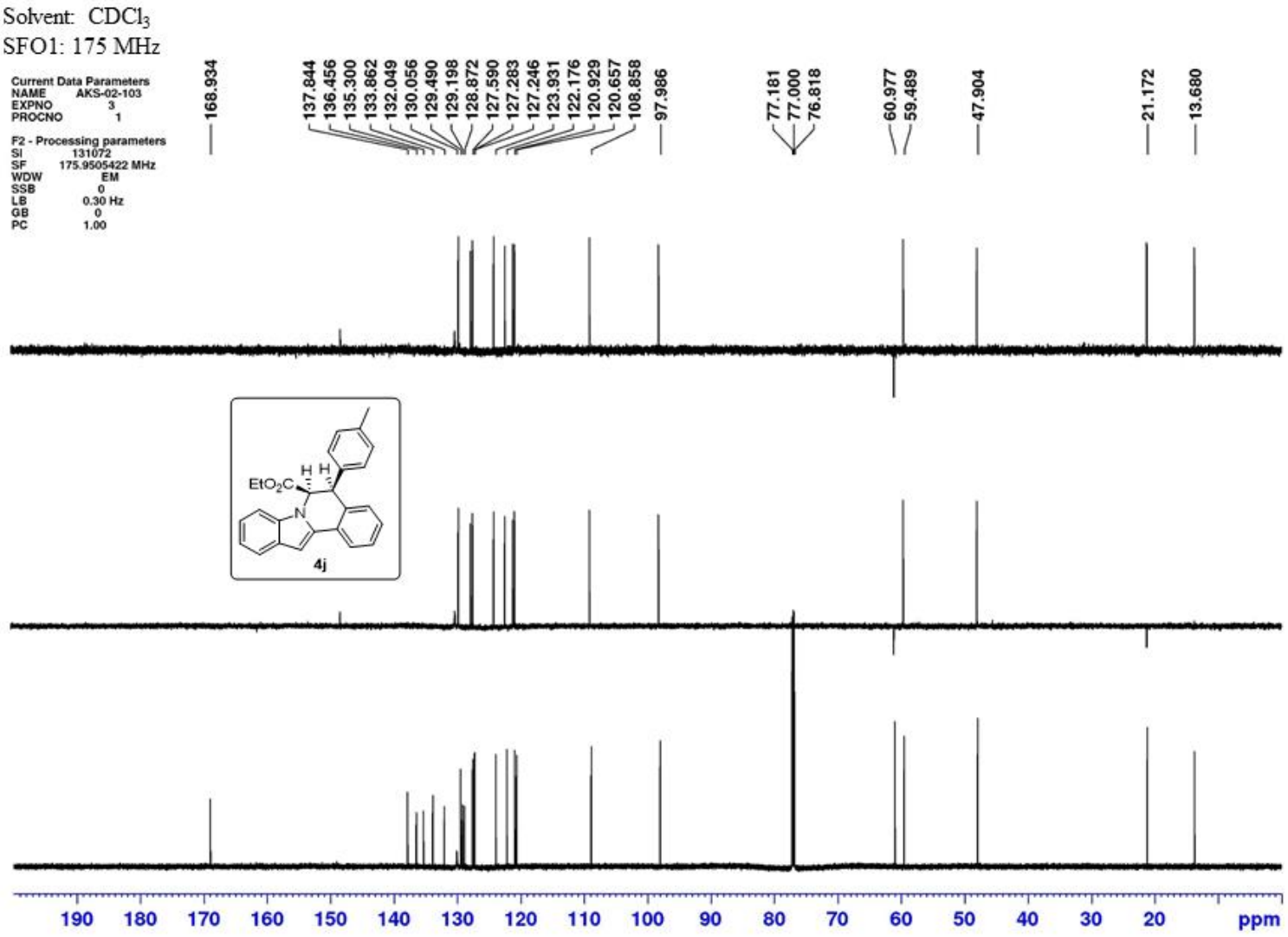
Current Data Parameters
NAME AKS-02-103-P1-H.fid
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7430879 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



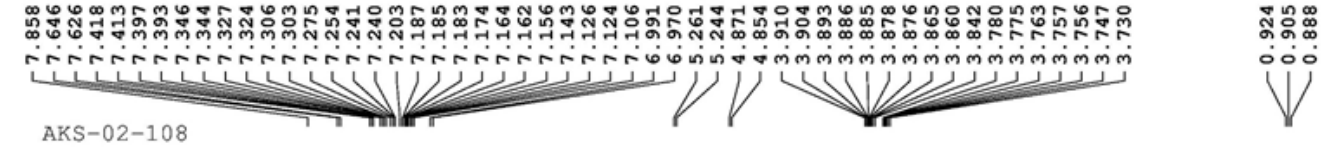
4j




Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-103
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505422 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
168.934
137.844
136.456
135.300
133.862
132.049
130.056
129.490
129.198
128.872
127.590
127.283
127.246
123.931
122.176
120.929
120.657
108.858
97.986
77.181
77.000
76.818
60.977
59.489
47.904
21.172
13.680
EtO2C
H
H
N
4j
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Solvent: $CDCl_3$

SFO1: 400 MHz

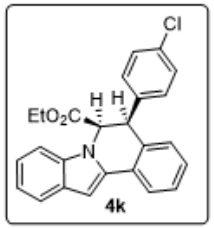


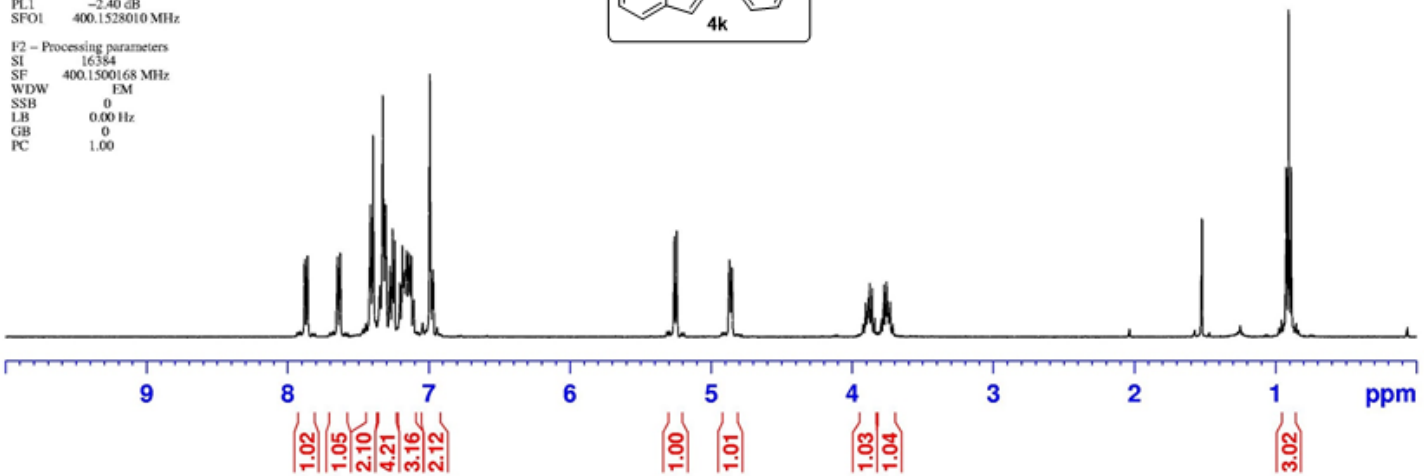
Current Data Parameters
NAME AKS-02-108-1
EXPNO 4
PROCNO 1

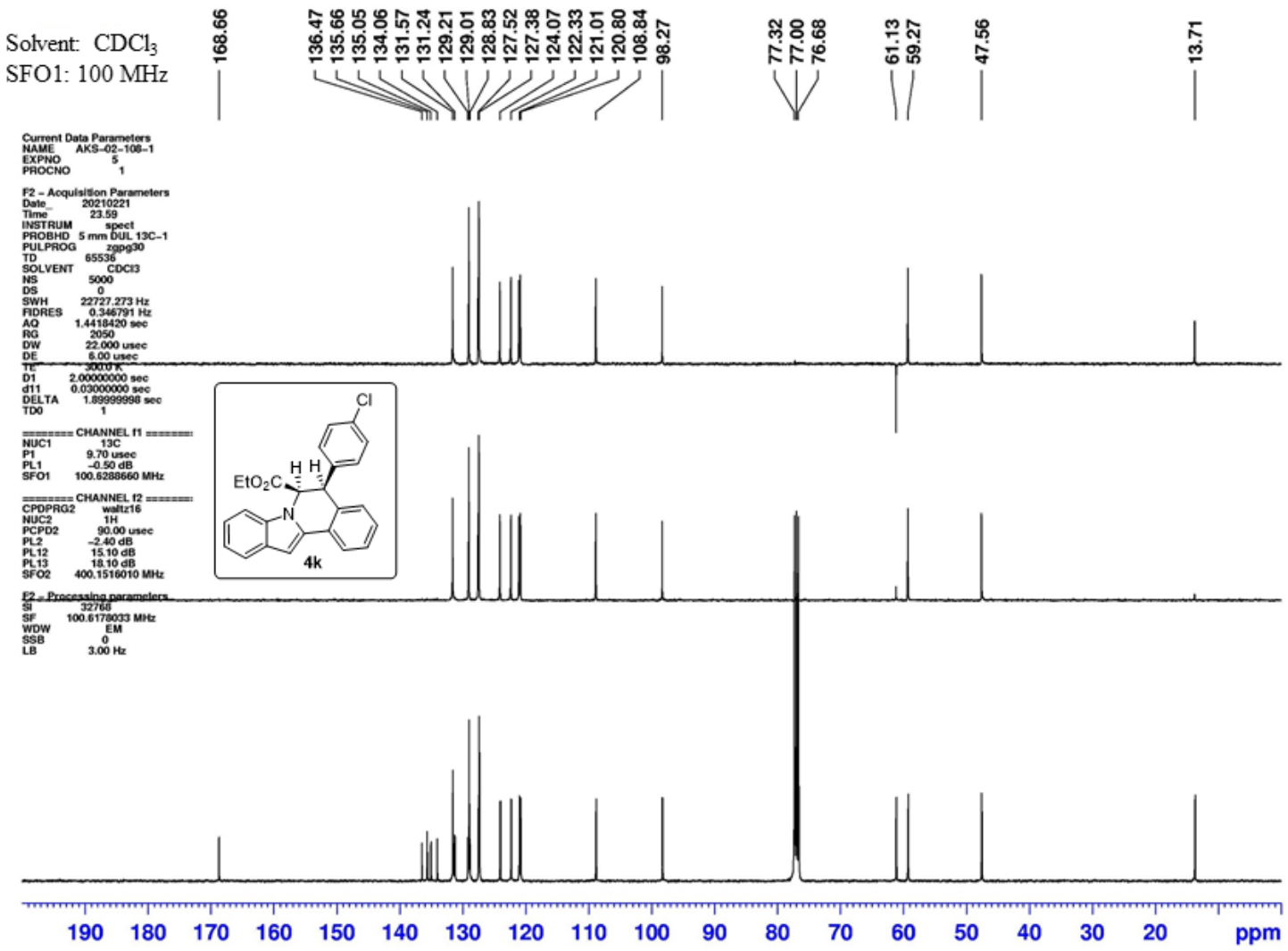
F2 - Acquisition Parameters
Date_ 20210220
Time 17.40
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 14
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 456
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz

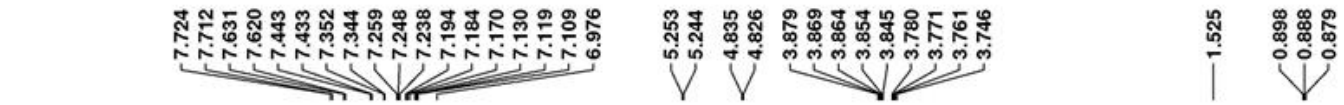
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00





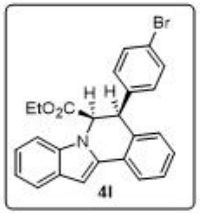

Solvent: $CDCl_3$
SFO1: 100 MHz
168.66
136.47
135.66
135.05
134.06
131.57
131.24
129.21
129.01
128.83
127.52
127.38
124.07
122.33
121.01
120.80
108.84
98.27
77.32
77.00
76.68
61.13
59.27
47.56
13.71
Current Data Parameters
NAME AKS-02-108-1
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210221
Time 23.59
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5000
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 2050
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178033 MHz
WDW EM
SSB 0
LB 3.00 Hz
Cl
H H
EtO_2C
N
4k
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

Solvent: $CDCl_3$
SFO1: 700 MHz

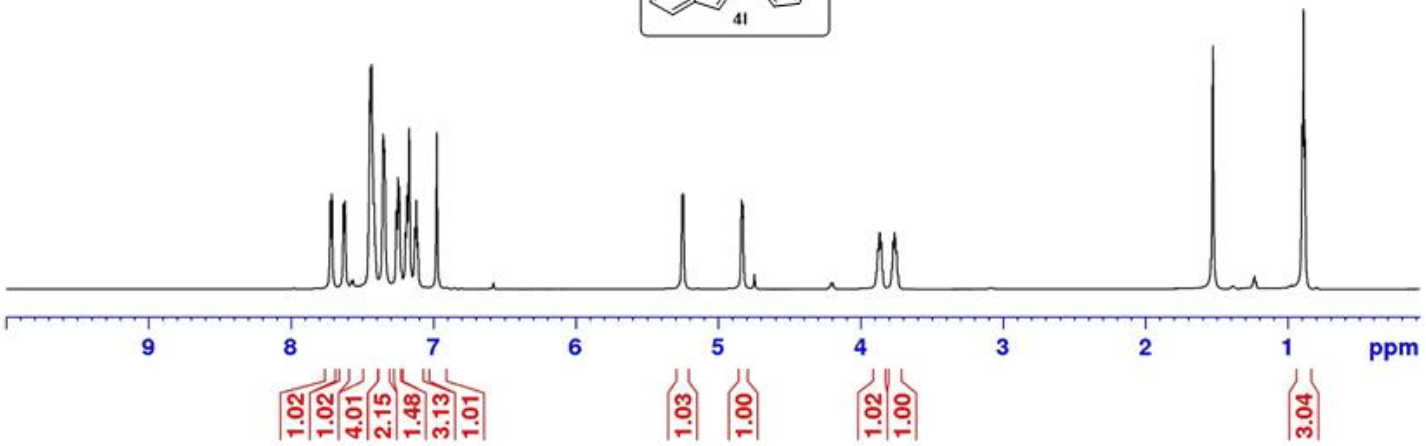


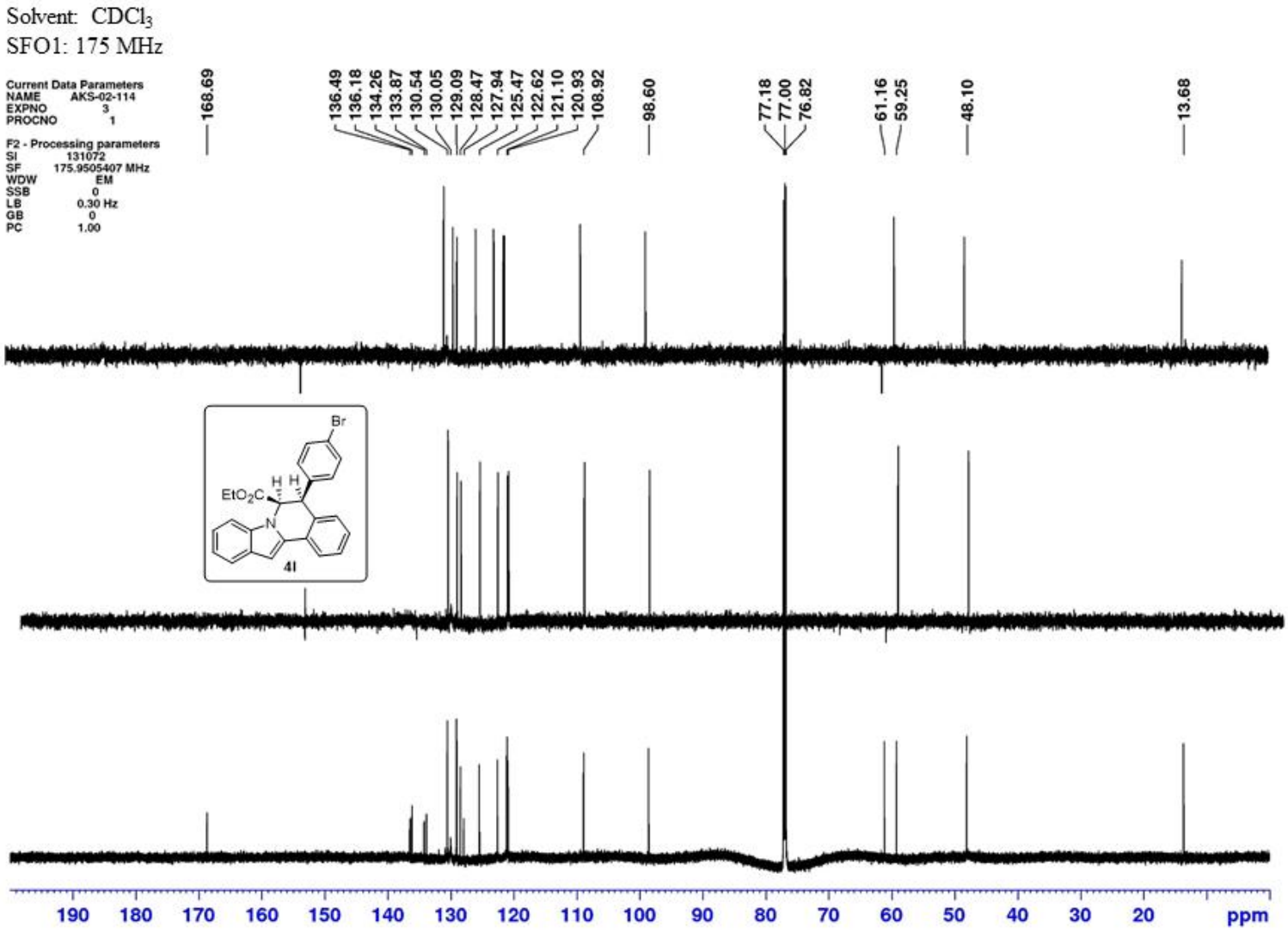
Current Data Parameters
NAME AKS-02-114
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431025 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4l




Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-114
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505407 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
168.69
136.49
136.18
134.26
133.87
130.54
130.05
129.09
128.47
127.94
125.47
122.62
121.10
120.93
108.92
98.60
77.18
77.00
76.82
61.16
59.25
48.10
13.68
Br
H H
EtO2C
N
4l
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

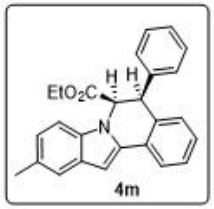
Solvent: $CDCl_3$

SFO1: 700 MHz

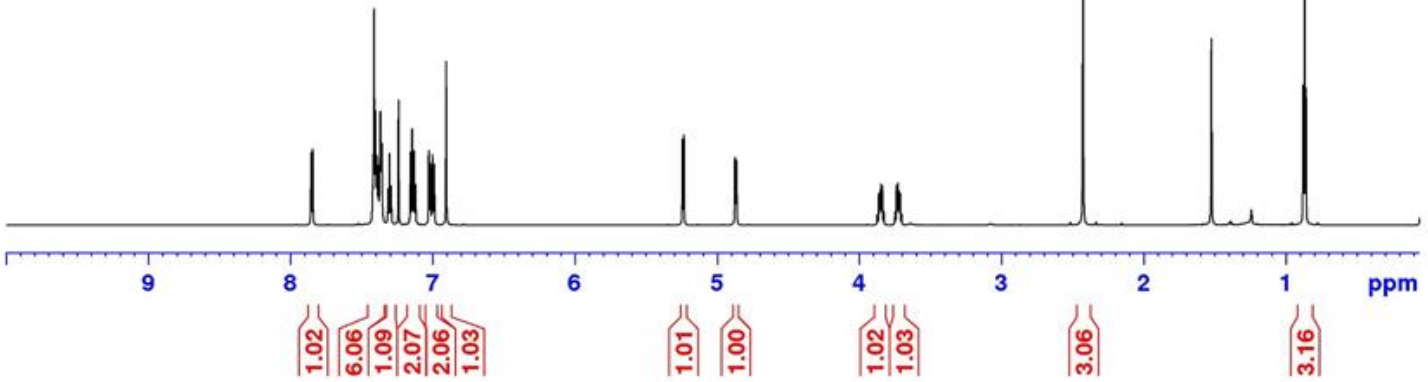
7.855 7.844 7.422 7.413 7.402 7.393 7.383 7.368 7.358 7.314 7.303 7.292 7.240 7.157 7.145 7.131 7.121 7.027 7.016 7.002 6.990 6.906 5.243 5.233 4.874 4.864 3.873 3.863 3.857 3.853 3.847 3.842 3.837 3.827 3.748 3.738 3.733 3.726 3.723 3.718 3.713 3.702 2.427 1.523 0.878 0.868 0.858

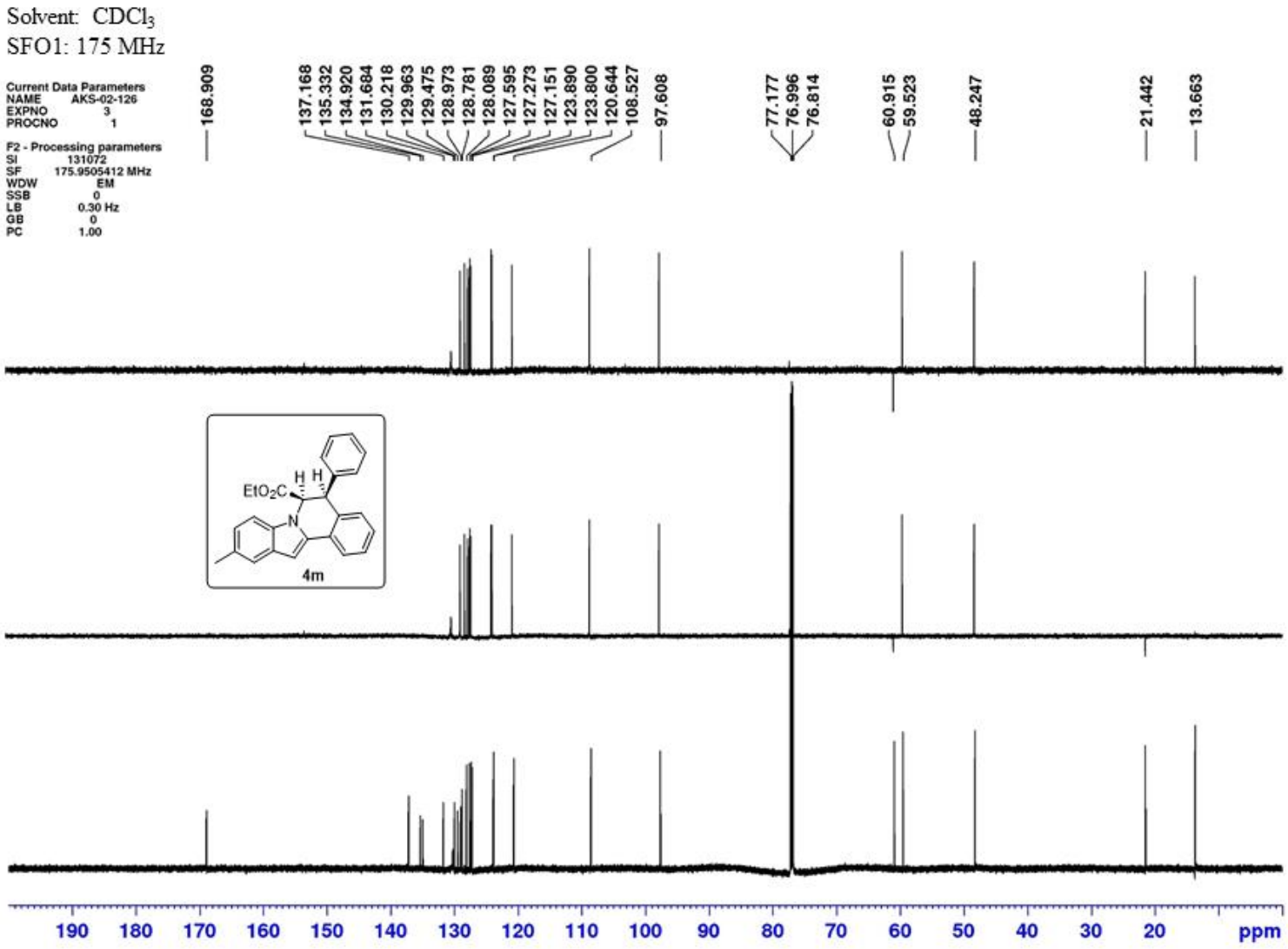
Current Data Parameters
NAME AKS-02-126
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431005 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4m



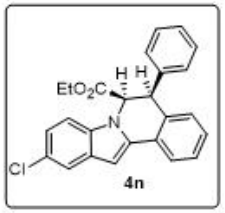

Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-126
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505412 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
168.909
137.168
135.332
134.920
131.684
130.218
129.963
129.475
128.973
128.781
128.089
127.595
127.273
127.151
123.890
123.800
120.644
108.527
97.608
77.177
76.996
76.814
60.915
59.523
48.247
21.442
13.663
EtO2C
H
H
N
4m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Solvent: $CDCl_3$

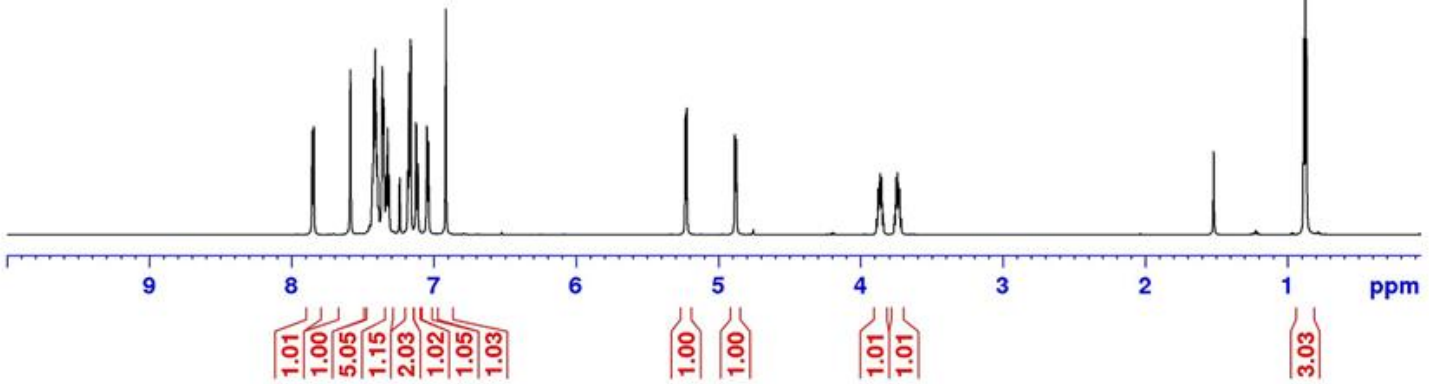
SFO1: 700 MHz

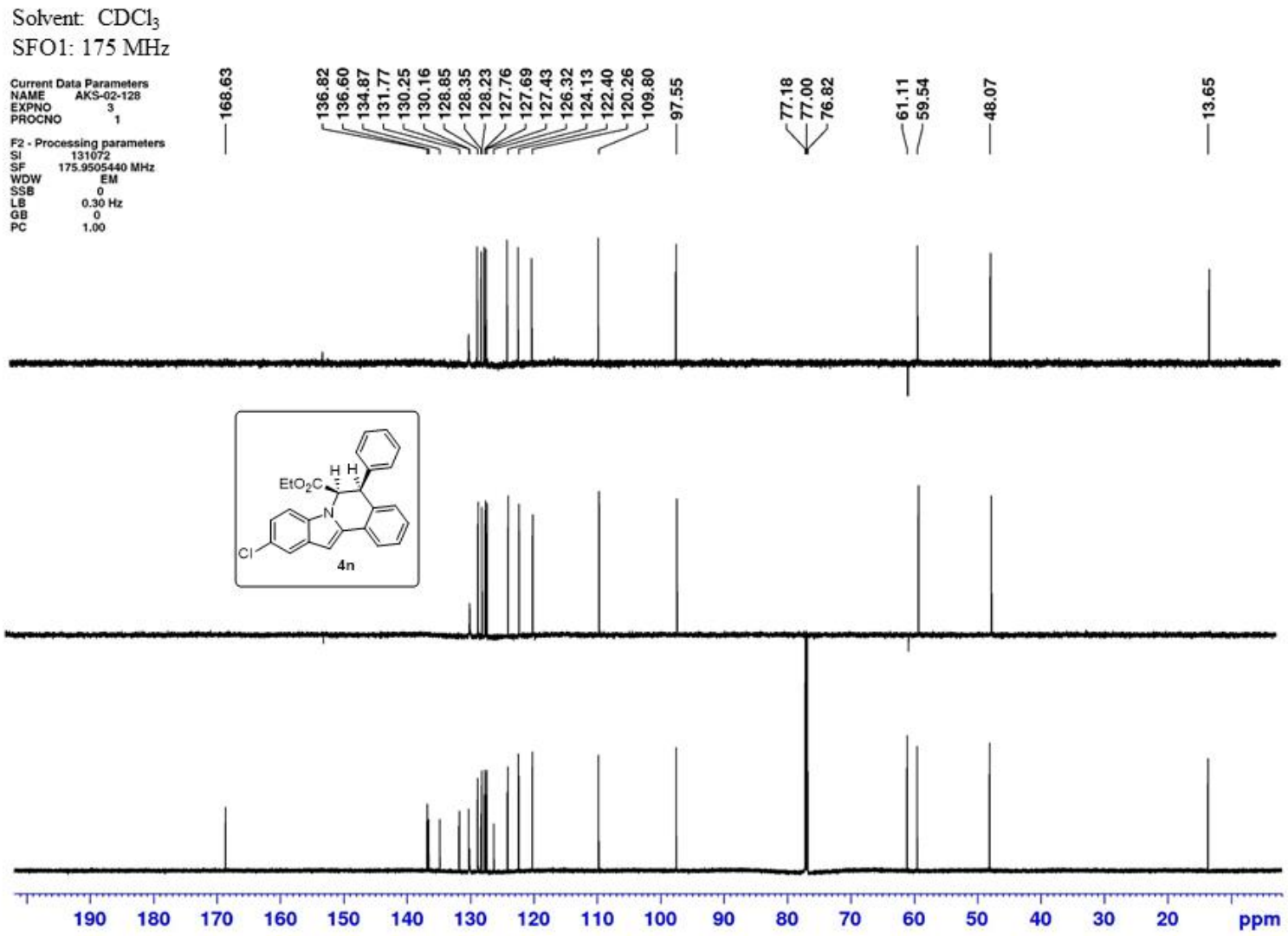
Current Data Parameters
NAME AKS-02-128
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431028 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4n



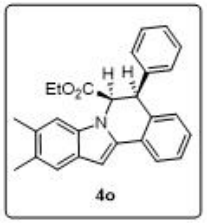

Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-128
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505440 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
168.63
136.82
136.60
134.87
131.77
130.25
130.16
128.85
128.35
128.23
127.76
127.69
127.43
126.32
124.13
122.40
120.26
109.80
97.55
77.18
77.00
76.82
61.11
59.54
48.07
13.65
EtO2C
H
H
N
Cl
4n
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Solvent: $CDCl_3$
SFO1: 700 MHz

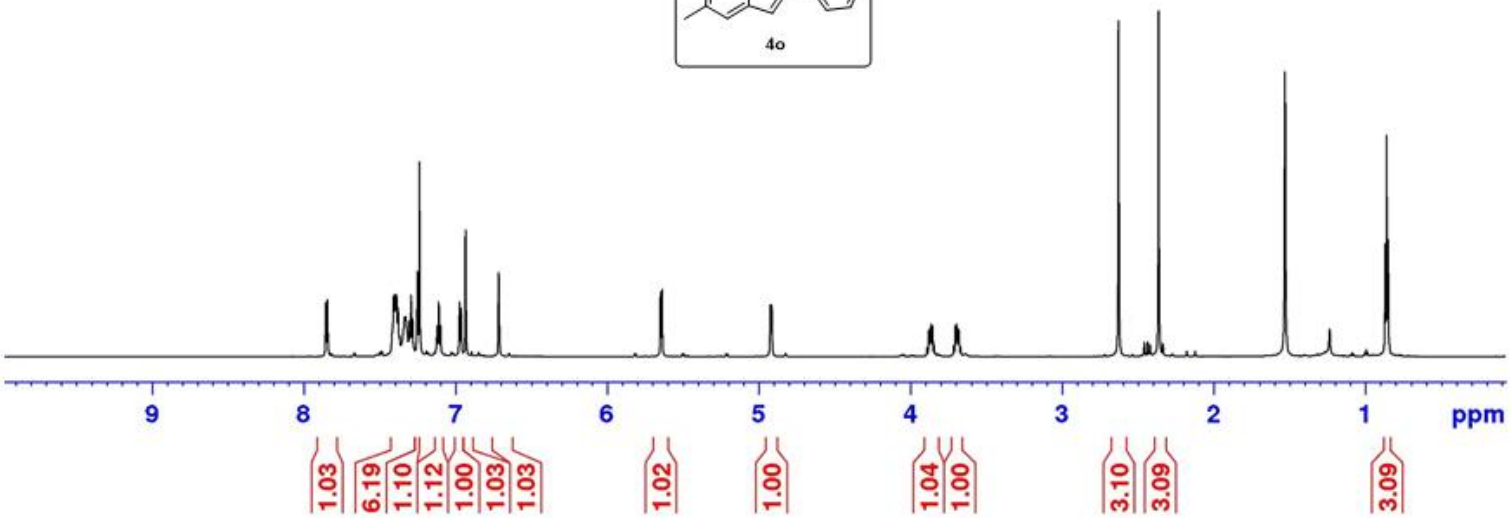
7.856 7.845 7.407 7.397 7.388 7.379 7.328 7.304 7.293 7.282 7.251 7.238 7.121 7.110 7.100 6.974 6.963 6.933 6.715 5.647 5.638 4.923 4.914 3.889 3.879 3.873 3.868 3.863 3.859 3.853 3.843 3.714 3.704 3.698 3.693 3.688 3.683 3.678 3.668 2.627 2.362 1.528 0.868 0.858 0.848

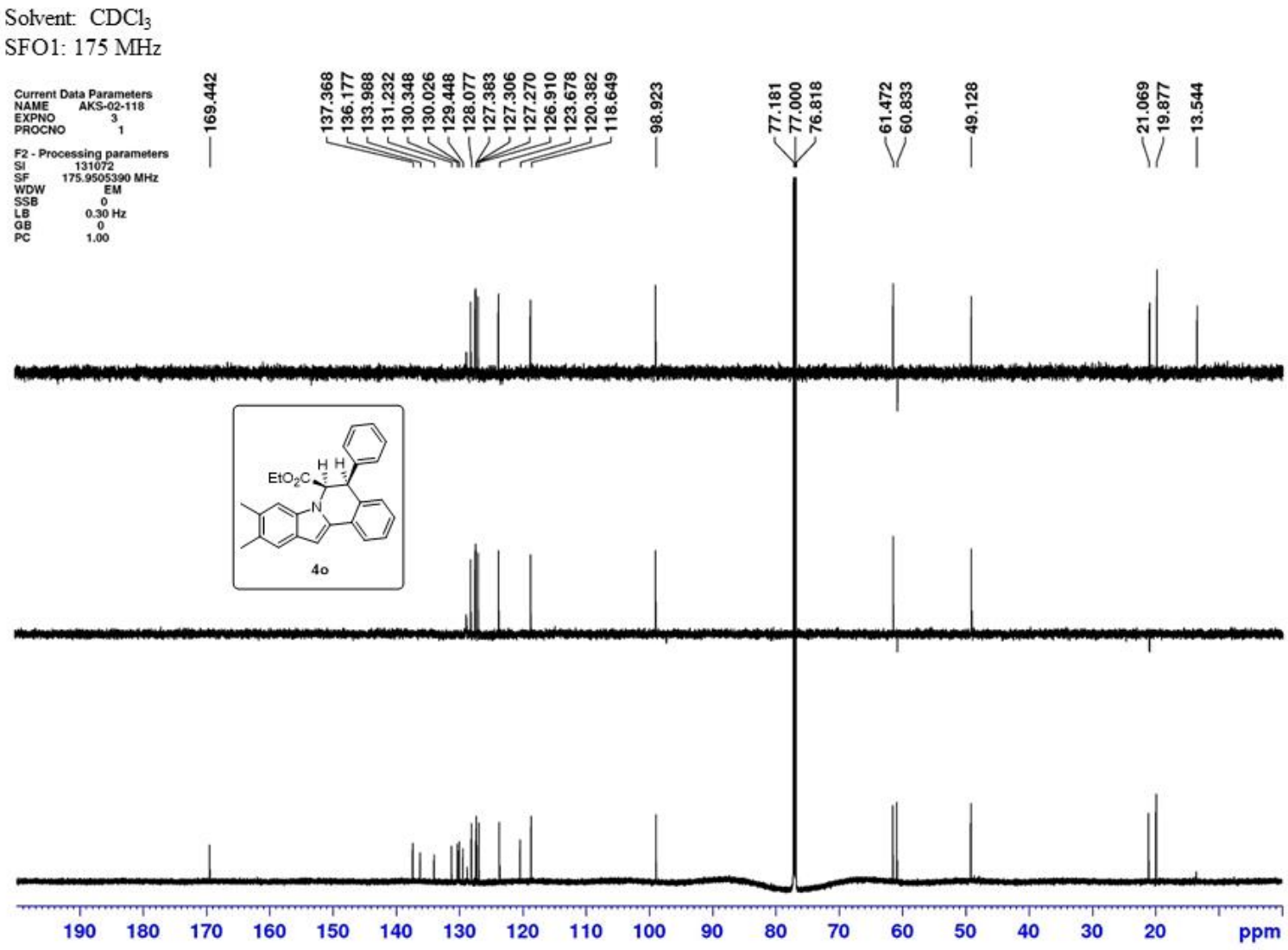
Current Data Parameters
NAME AKS-02-118
EXPNO 1
PROCNO 1

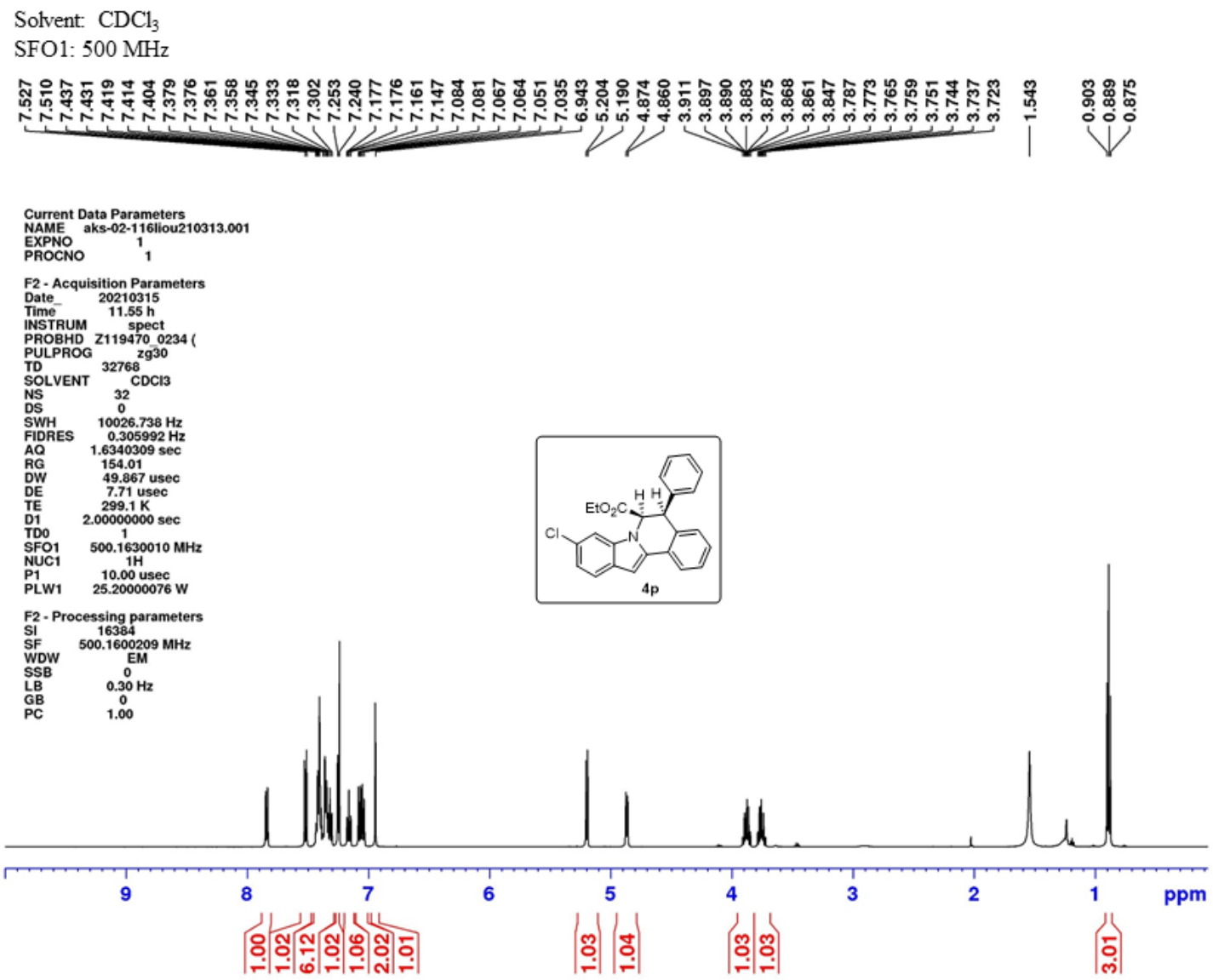
F2 - Processing parameters
SI 65536
SF 699.7431013 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

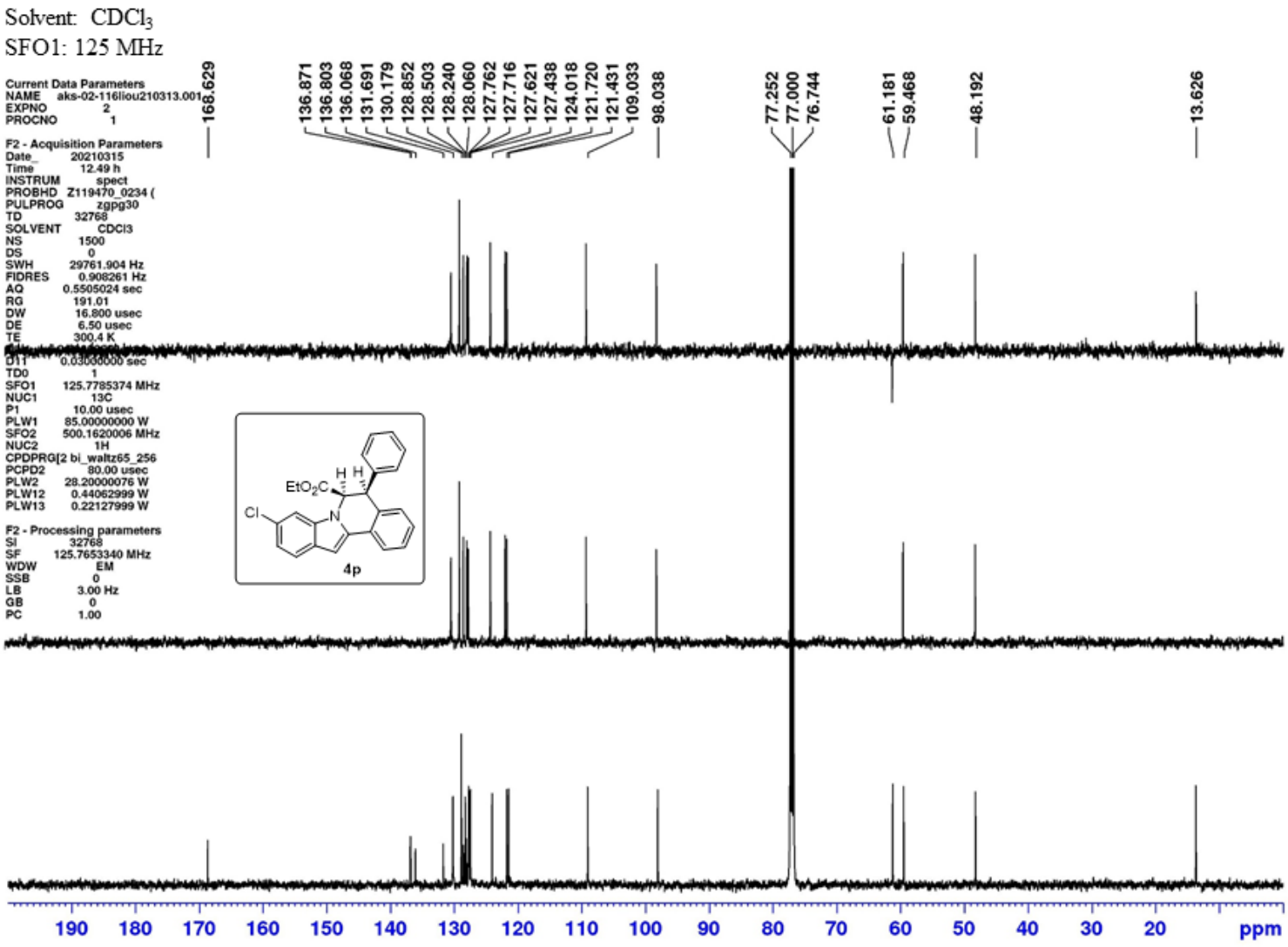


4o




Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-118
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505390 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
169.442
137.368
136.177
133.988
131.232
130.348
130.026
129.448
128.077
127.383
127.306
127.270
126.910
123.678
120.382
118.649
98.923
77.181
77.000
76.818
61.472
60.833
49.128
21.069
19.877
13.544
EtO2C
H
H
N
4o
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

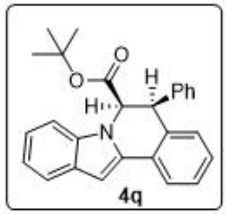

Solvent: CDCl3
SFO1: 500 MHz
7.527
7.510
7.437
7.431
7.419
7.414
7.404
7.379
7.376
7.361
7.358
7.345
7.333
7.318
7.302
7.253
7.240
7.177
7.176
7.161
7.147
7.084
7.081
7.067
7.064
7.051
7.035
6.943
5.204
5.190
4.874
4.860
3.911
3.897
3.890
3.883
3.875
3.868
3.861
3.847
3.787
3.773
3.765
3.759
3.751
3.744
3.737
3.723
1.543
0.903
0.889
0.875
Current Data Parameters
NAME aks-02-116liou210313.001
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210315
Time 11.55 h
INSTRUM spect
PROBHD Z119470_0234 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 10026.738 Hz
FIDRES 0.305992 Hz
AQ 1.6340309 sec
RG 154.01
DW 49.867 usec
DE 7.71 usec
TE 299.1 K
D1 2.00000000 sec
TD0 1
SFO1 500.1630010 MHz
NUC1 1H
P1 10.00 usec
PLW1 25.20000076 W
F2 - Processing parameters
SI 16384
SF 500.1600209 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
EtO2C
H
H
Cl
N
4p
9
8
7
6
5
4
3
2
1
ppm
1.00
1.02
6.12
1.02
1.06
2.02
1.01
1.03
1.04
1.03
1.03
3.01


Solvent: $CDCl_3$
SFO1: 125 MHz
Current Data Parameters
NAME aks-02-116liou210313.001
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210315
Time 12.49 h
INSTRUM spect
PROBHD Z119470_0234 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1500
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 191.01
DW 16.800 usec
DE 6.50 usec
TE 300.4 K
D11 0.03000000 sec
TD0 1
SFO1 125.7785374 MHz
NUC1 13C
P1 10.00 usec
PLW1 85.00000000 W
SFO2 500.1620006 MHz
NUC2 1H
CPDPRG[2 bi_waltz65_256
PCPD2 80.00 usec
PLW2 28.20000076 W
PLW12 0.44062999 W
PLW13 0.22127999 W
F2 - Processing parameters
SI 32768
SF 125.7653340 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
168.629
136.871
136.803
136.068
131.691
130.179
128.852
128.503
128.240
128.060
127.762
127.716
127.621
127.438
124.018
121.720
121.431
109.033
98.038
77.252
77.000
76.744
61.181
59.468
48.192
13.626
EtO2C
H
H
Cl
N
4p
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

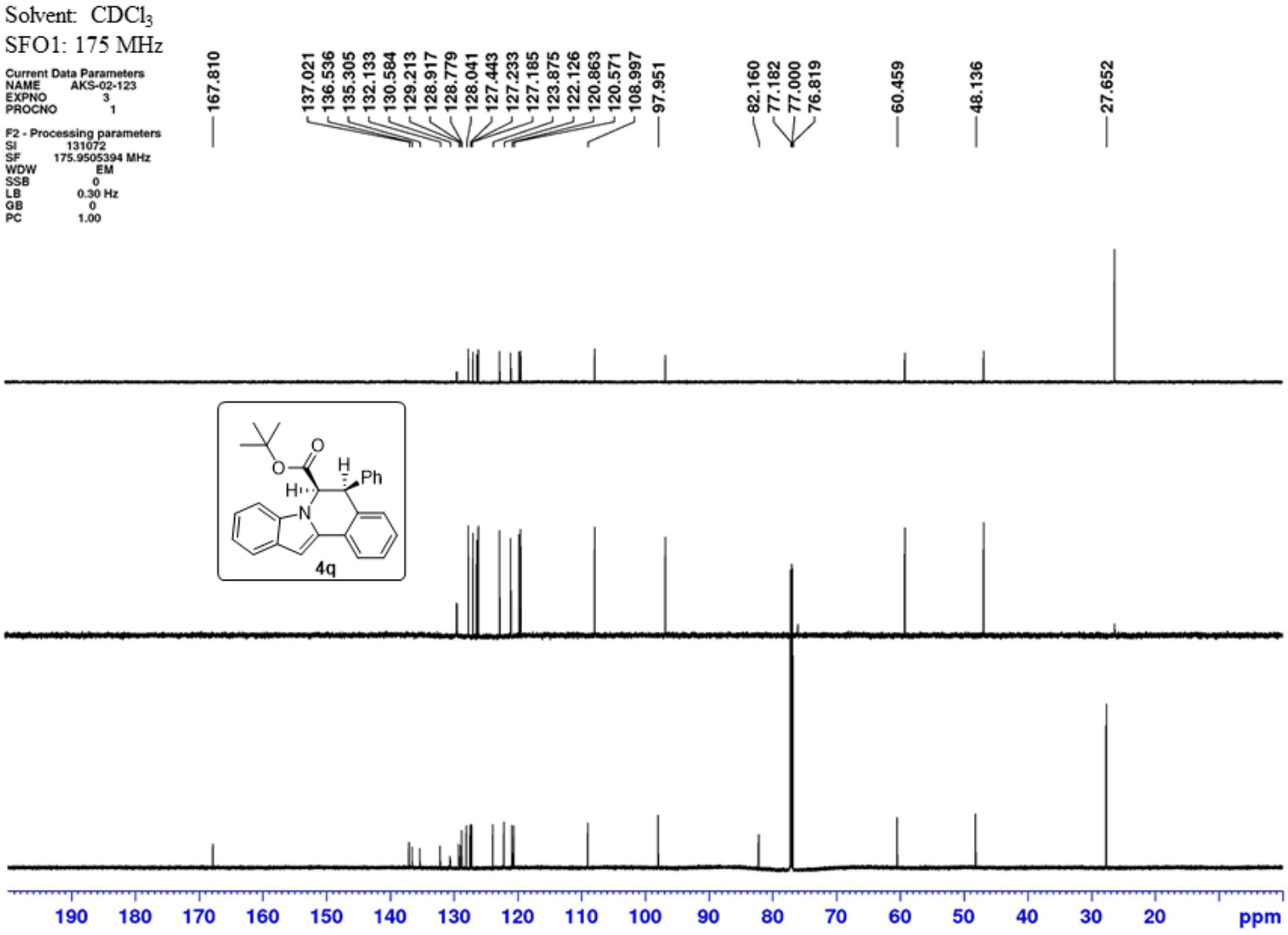
Solvent: $CDCl_3$
SFO1: 700 MHz

Current Data Parameters
NAME AKS-02-123
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431013 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4q


Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-123
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505394 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
167.810
137.021
136.536
135.305
132.133
130.584
129.213
128.917
128.779
128.041
127.443
127.233
127.185
123.875
122.126
120.863
120.571
108.997
97.951
82.160
77.182
77.000
76.819
60.459
48.136
27.652
4q
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

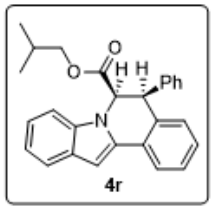
Solvent: $CDCl_3$

SFO1: 700 MHz

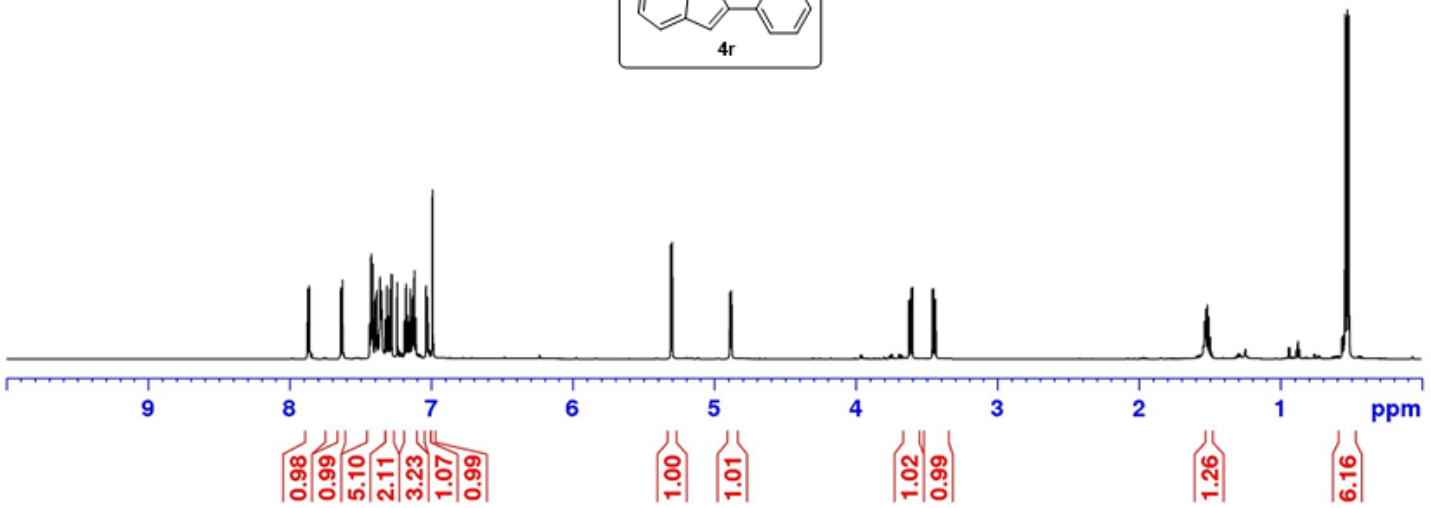
7.628 7.434 7.424 7.413 7.397 7.386 7.376 7.361 7.351 7.324 7.313 7.303 7.291 7.280 7.240 7.189 7.188 7.178 7.167 7.166 7.158 7.157 7.146 7.136 7.135 7.129 7.119 7.108 7.036 7.025 6.991 5.307 5.297 4.889 4.879 3.624 3.615 3.609 3.600 3.459 3.450 3.444 3.435 1.543 1.533 1.524 1.514 1.505 1.495 0.542 0.532 0.524 0.515

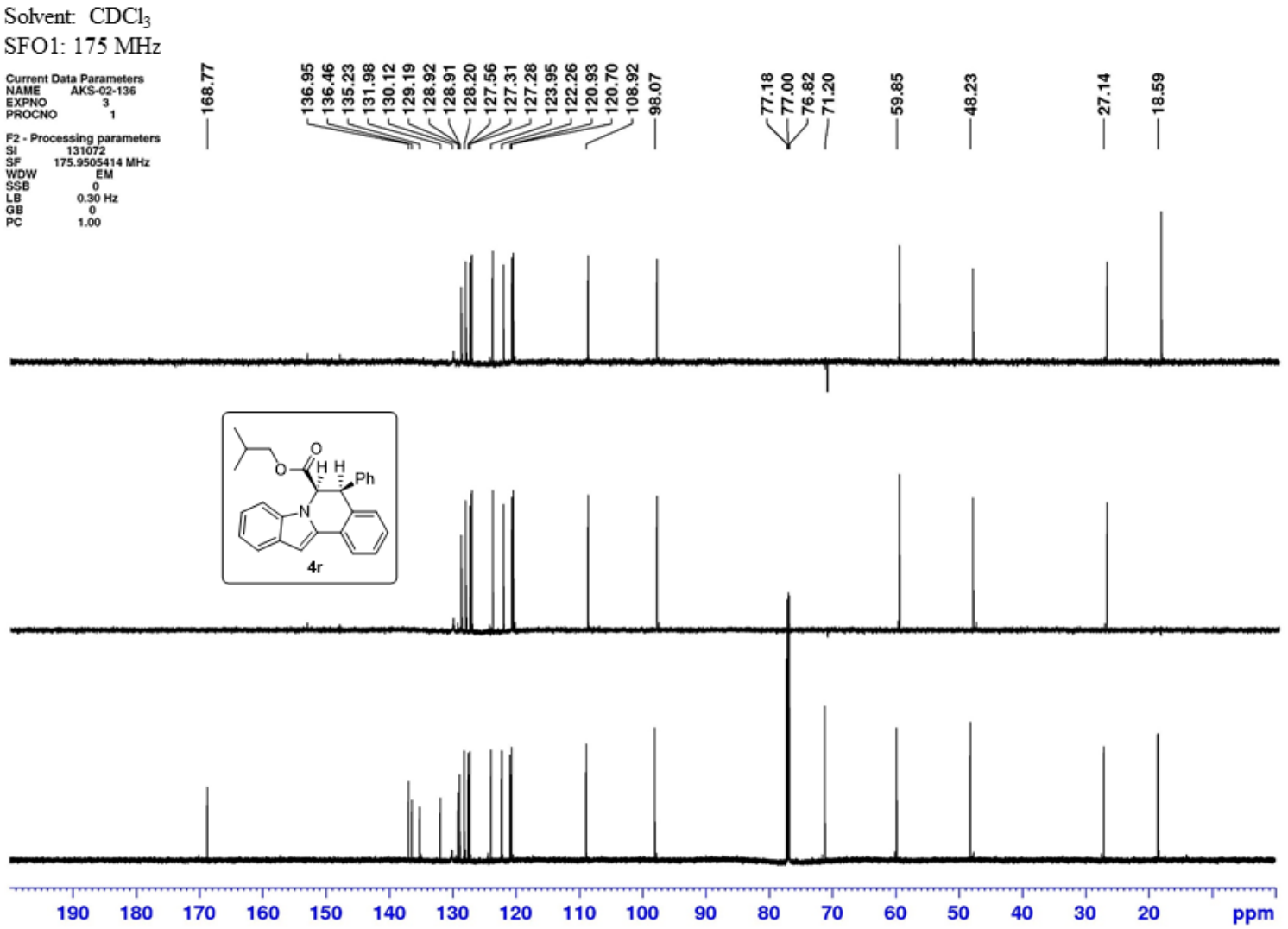
Current Data Parameters
NAME AKS-02-136
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431005 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4r




Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-136
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505414 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
168.77
136.95
136.46
135.23
131.98
130.12
129.19
128.92
128.91
128.20
127.56
127.31
127.28
123.95
122.26
120.93
120.70
108.92
98.07
77.18
77.00
76.82
71.20
59.85
48.23
27.14
18.59
4r
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

Solvent: $CDCl_3$

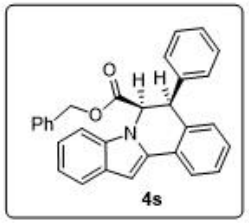
SFO1: 700 MHz

7.879 7.868 7.630 7.619 7.341 7.331 7.321 7.257 7.245 7.227 7.211 7.201 7.190 7.158 7.148 7.137 7.119 7.108 7.098 7.002 6.992 6.794 6.784 5.338 5.328 4.888 4.879 4.869 4.677 4.659

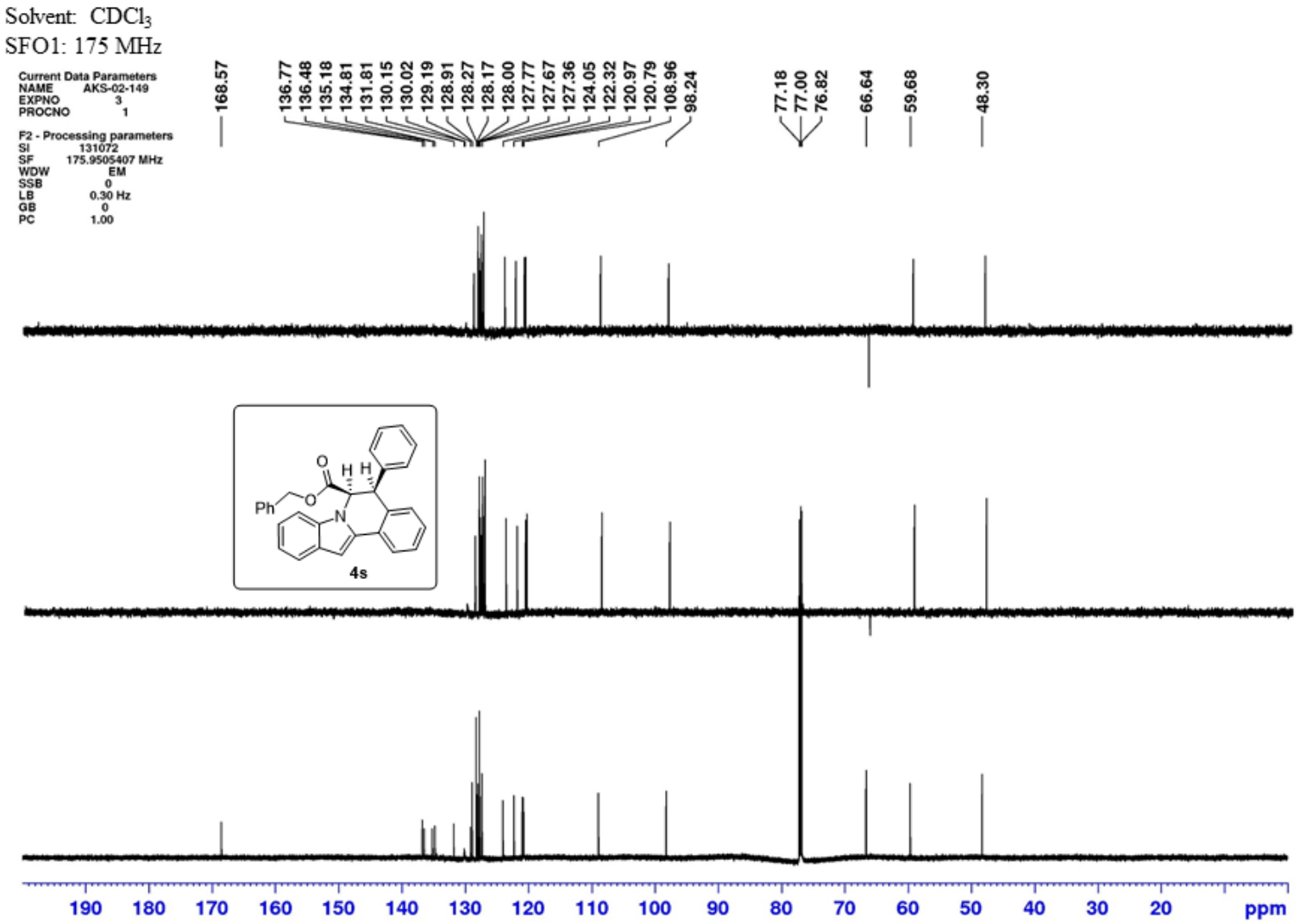
1.512

Current Data Parameters
NAME AKS-02-149
EXPNO 1
PROCNO 1

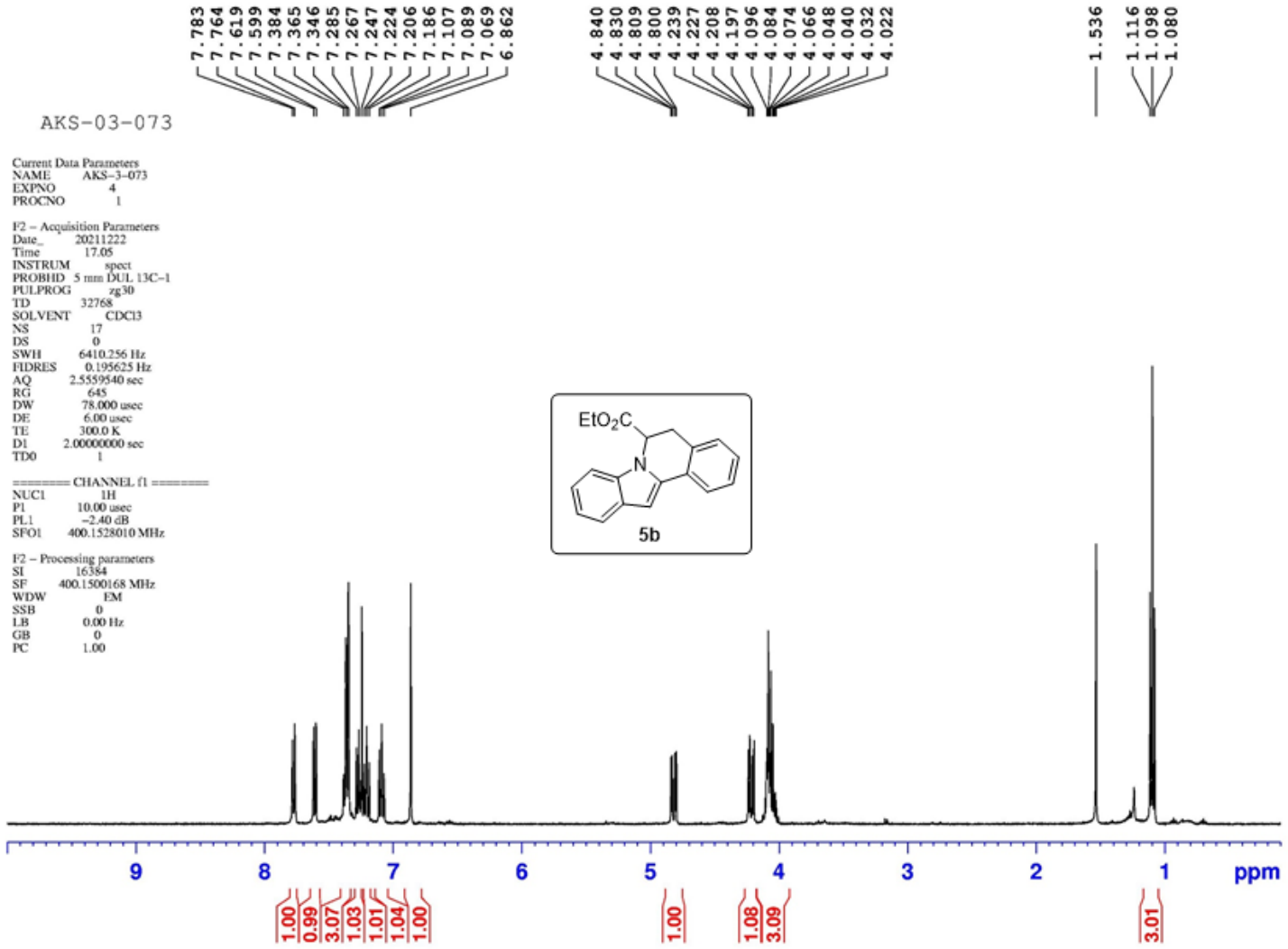
F2 - Processing parameters
SI 65536
SF 699.7431092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

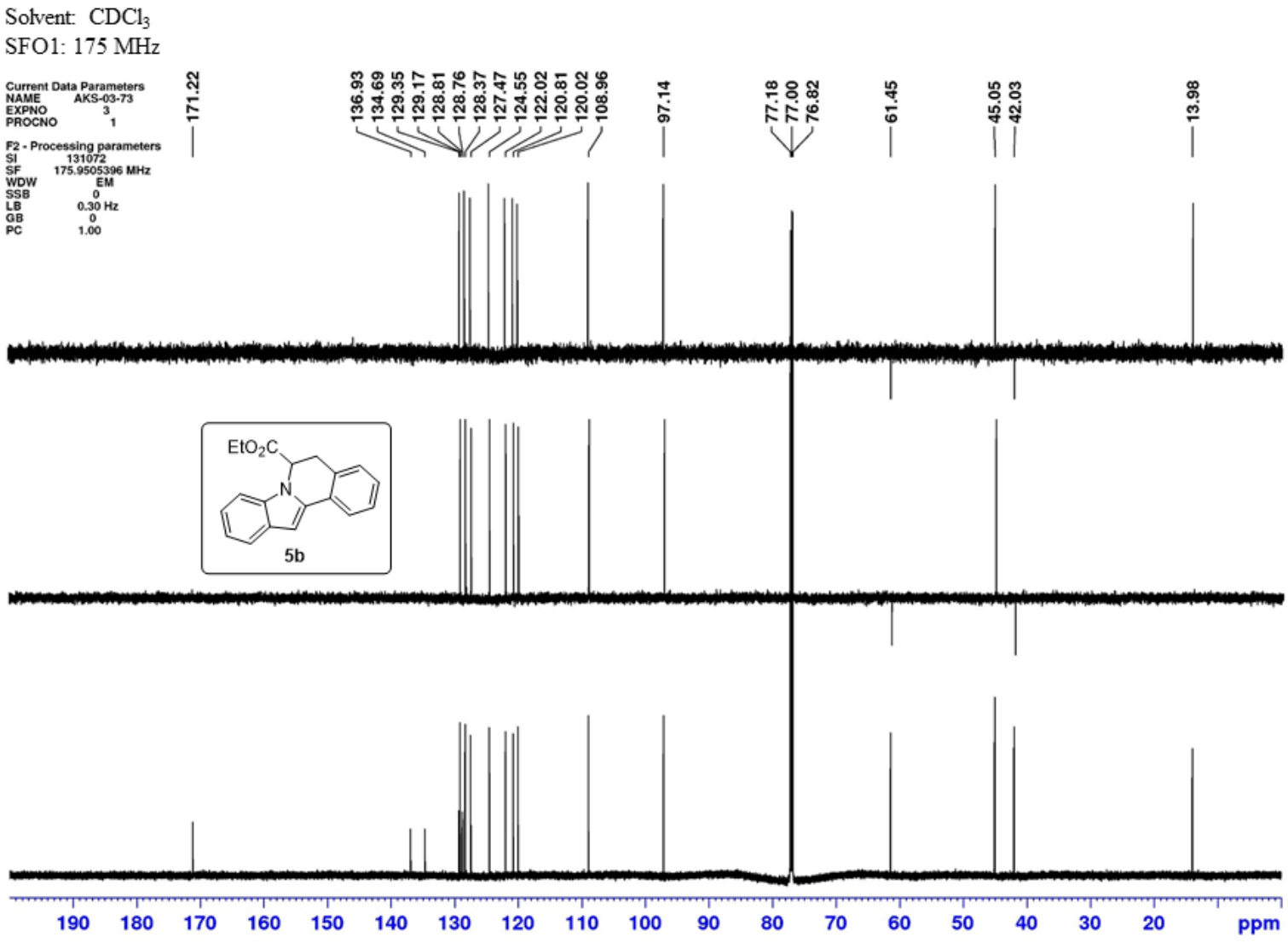


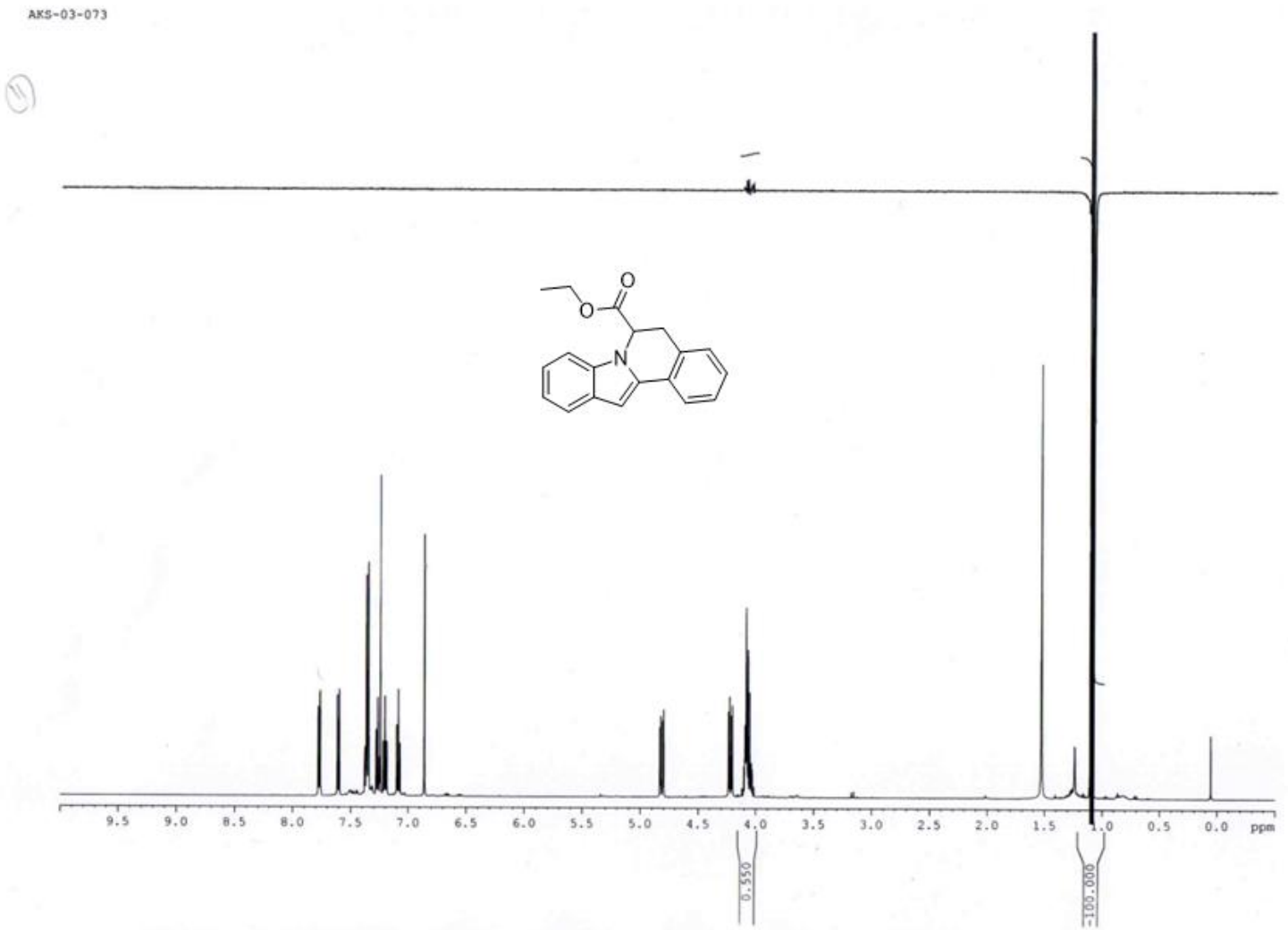
4s

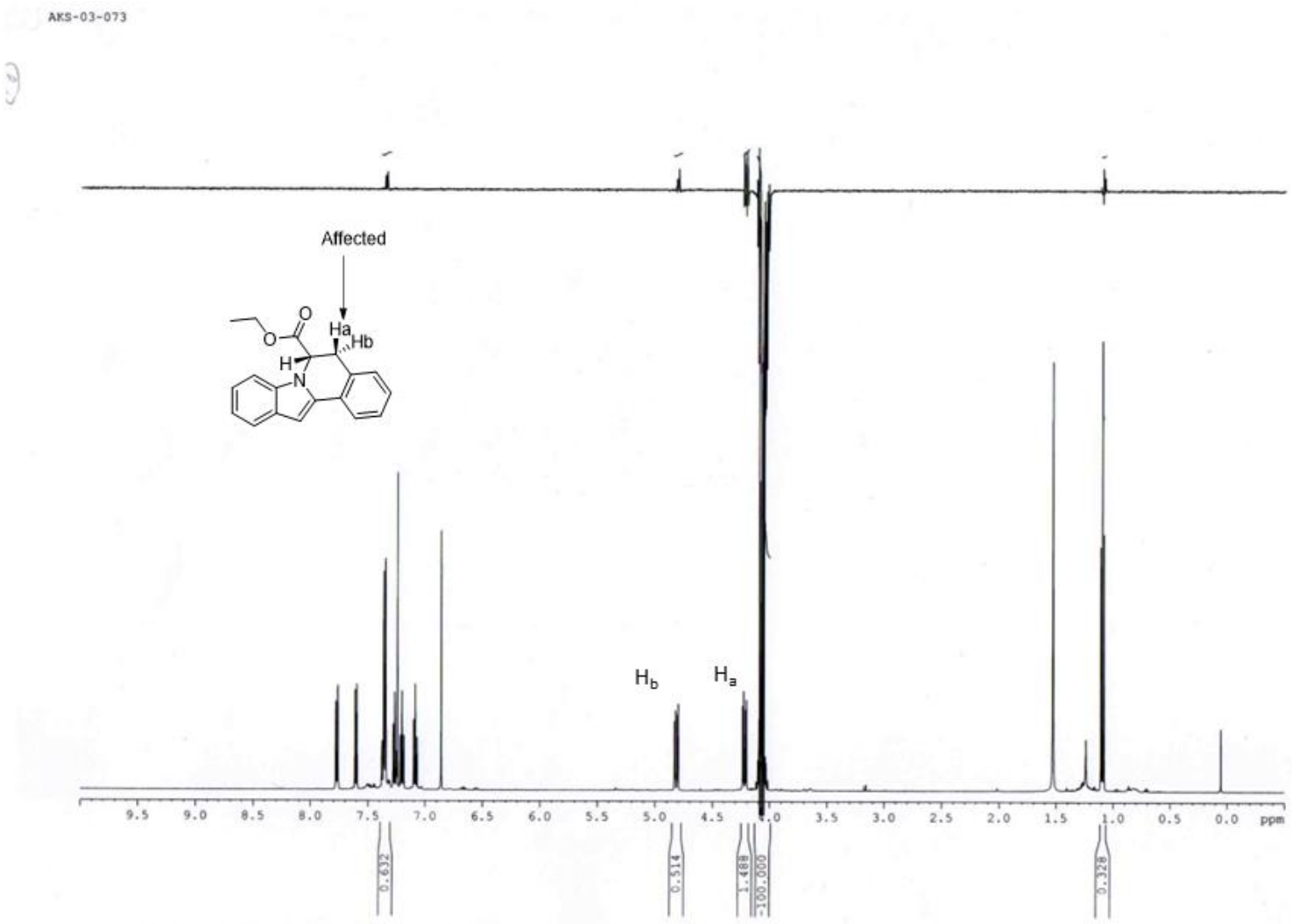

Solvent: $CDCl_3$
SFO1: 175 MHz
Current Data Parameters
NAME AKS-02-149
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505407 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
168.57
136.77
136.48
135.18
134.81
131.81
130.15
130.02
129.19
128.91
128.27
128.17
128.00
127.77
127.67
127.36
124.05
122.32
120.97
120.79
108.96
98.24
77.18
77.00
76.82
66.64
59.68
48.30
4s
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

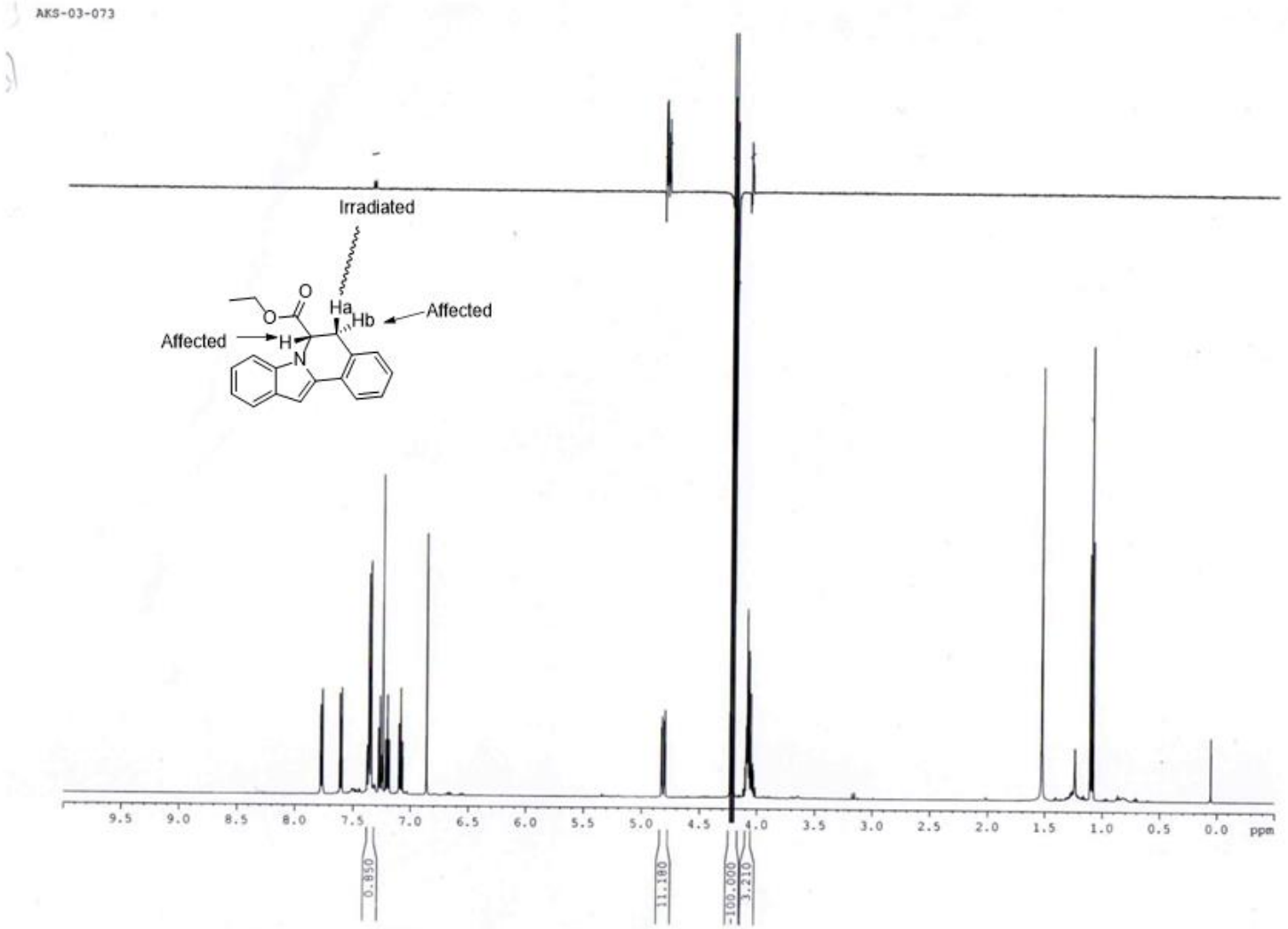
Solvent: $CDCl_3$
SFO1: 400 MHz

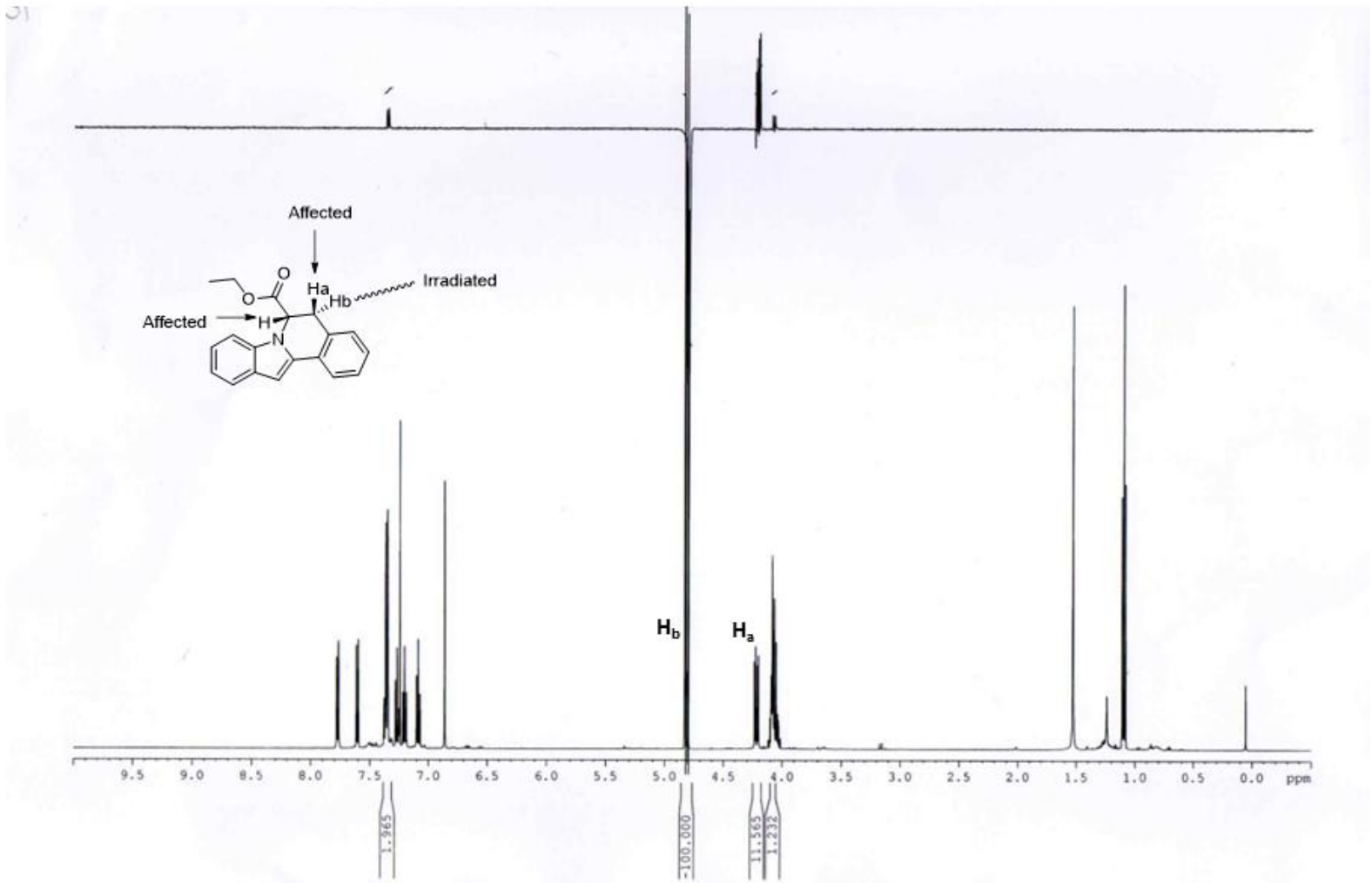



Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-03-73
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505396 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
171.22
136.93
134.69
129.35
129.17
128.81
128.76
128.37
127.47
124.55
122.02
120.81
120.02
108.96
97.14
77.18
77.00
76.82
61.45
45.05
42.03
13.98
EtO2C
N
5b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

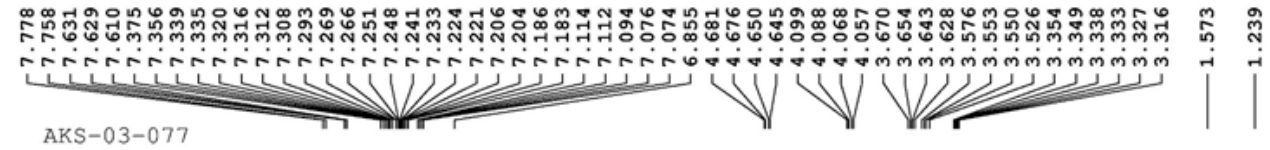

AKS-03-073
O
O
N
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.550
100.000


AKS-03-073
Affected
Ha
Hb
H
N
O
O
Hb
Ha
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
ppm
0.632
0.514
1.488
100.000
0.328


AKS-03-073
Irradiated
Affected
Affected
Ha
Hb
O
O
H
N
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
ppm
0.850
11.180
100.000
3.210


Affected
Ha
Hb
Irradiated
Affected
O
O
H
N
Hb
Ha
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
ppm
1.966
100.000
11.565
1.932

Solvent: $CDCl_3$
SFO1: 400 MHz

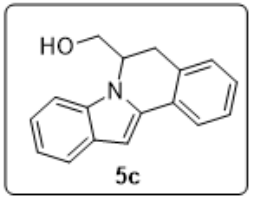


Current Data Parameters
NAME AKS-3-077
EXPNO 4
PROCNO 1

F2 – Acquisition Parameters
Date_ 20220104
Time 15.58
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 23
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 724
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

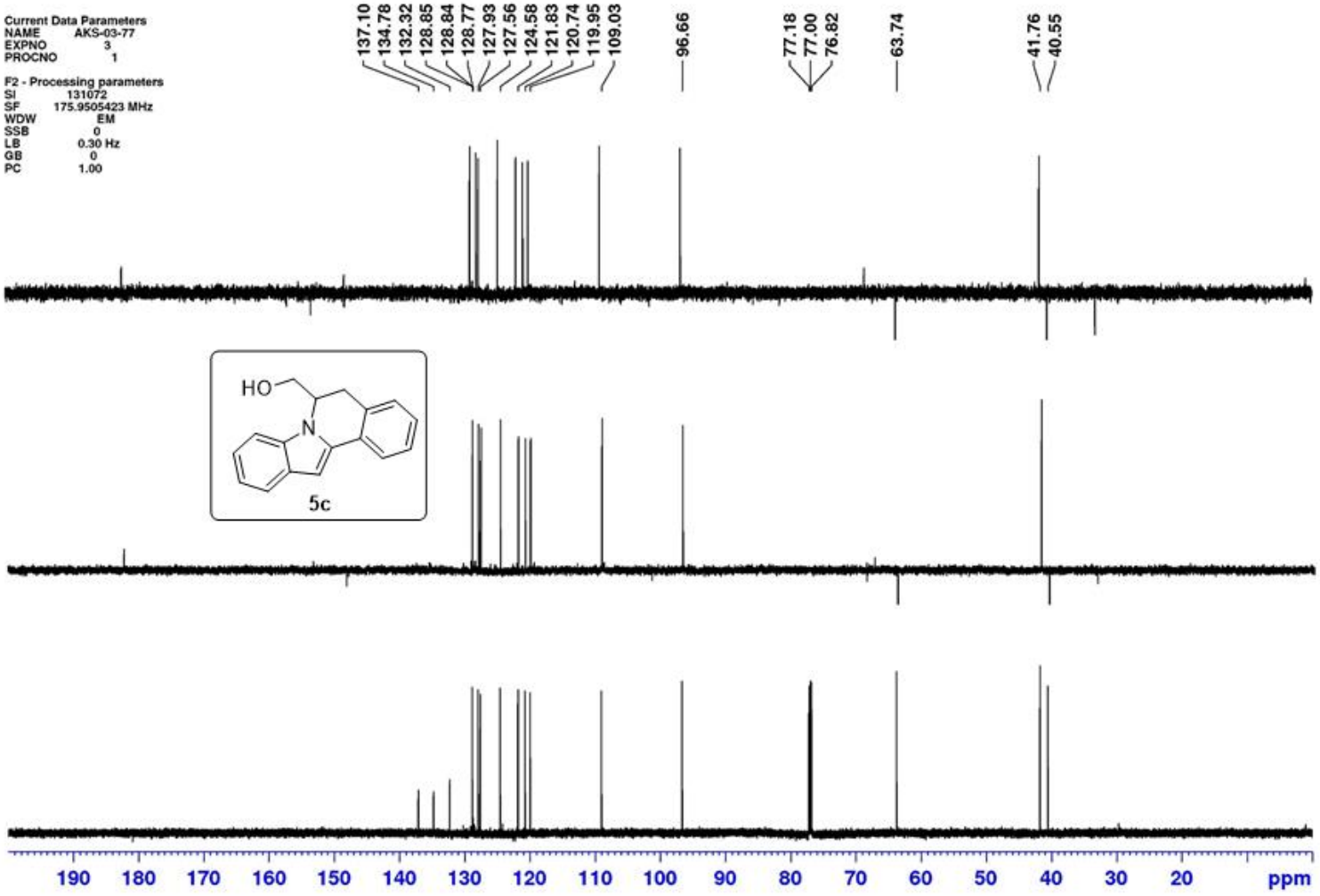
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz

F2 – Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

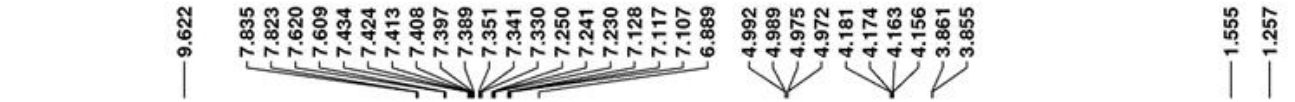


5c

Solvent: $CDCl_3$
SFO1: 175 MHz

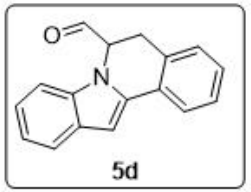


Solvent: $CDCl_3$
SFO1: 700 MHz

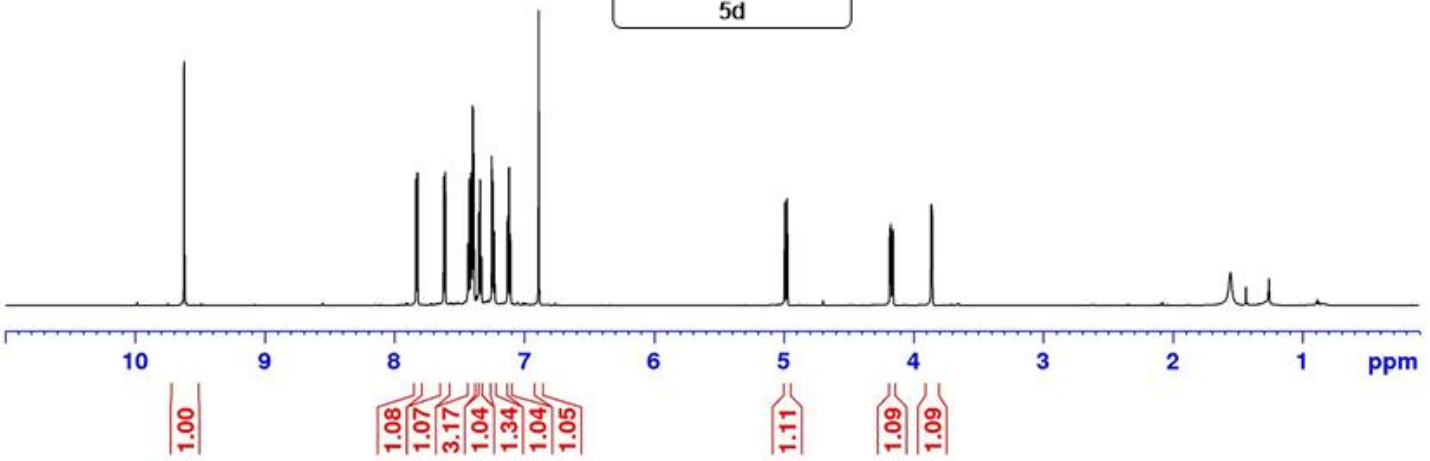


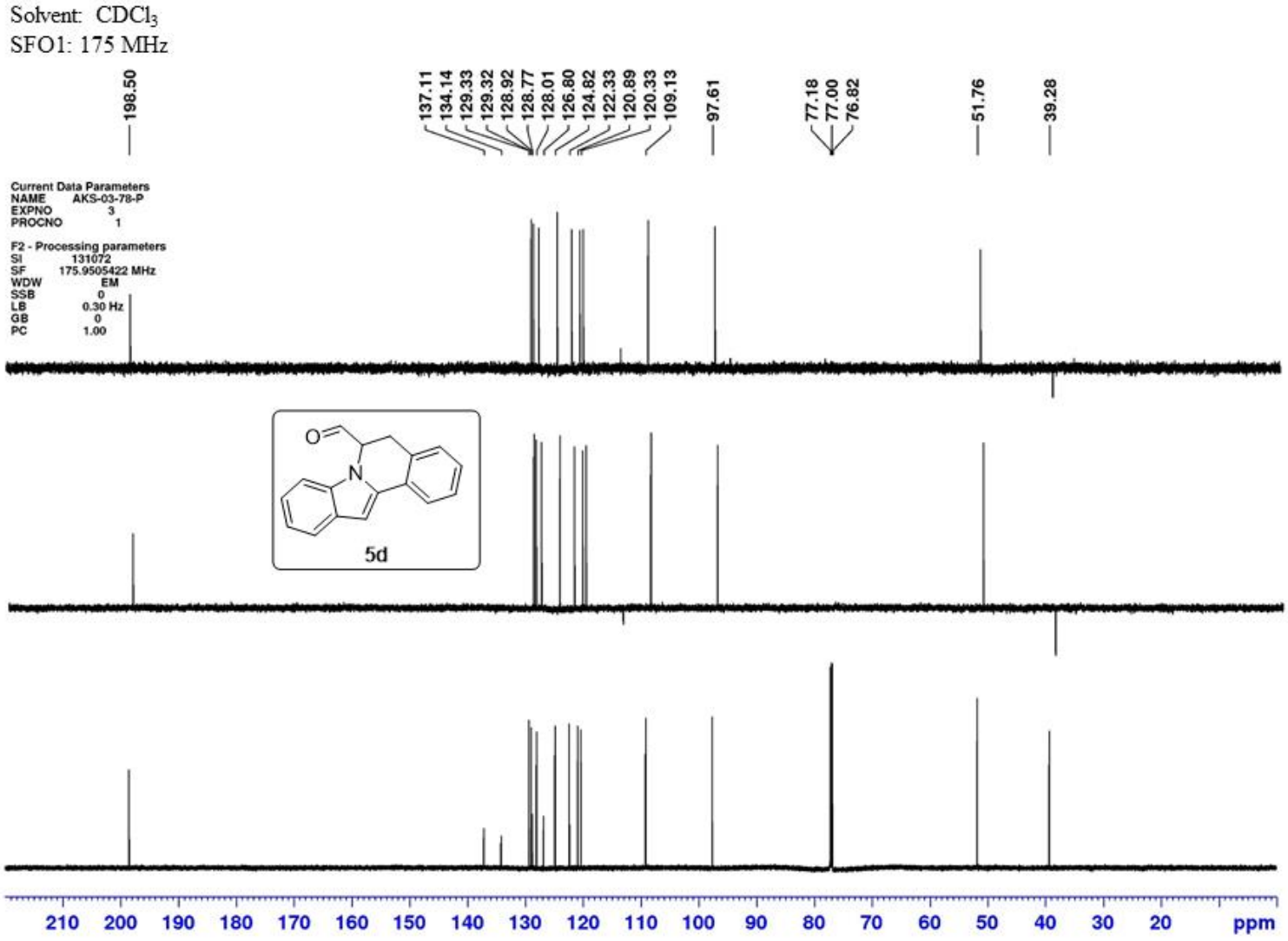
Current Data Parameters
NAME AKS-03-78-P
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7430945 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

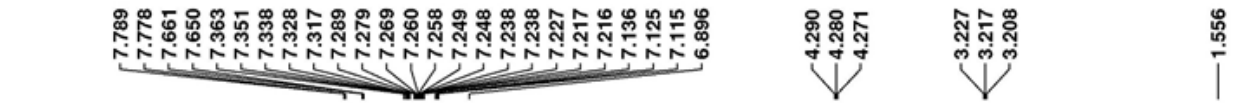


5d



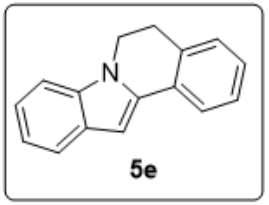

Solvent: $CDCl_3$
SFO1: 175 MHz
198.50
137.11
134.14
129.33
129.32
128.92
128.77
128.01
126.80
124.82
122.33
120.89
120.33
109.13
97.61
77.18
77.00
76.82
51.76
39.28
Current Data Parameters
NAME AKS-03-78-P
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505422 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
5d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

Solvent: $CDCl_3$
SFO1: 700 MHz

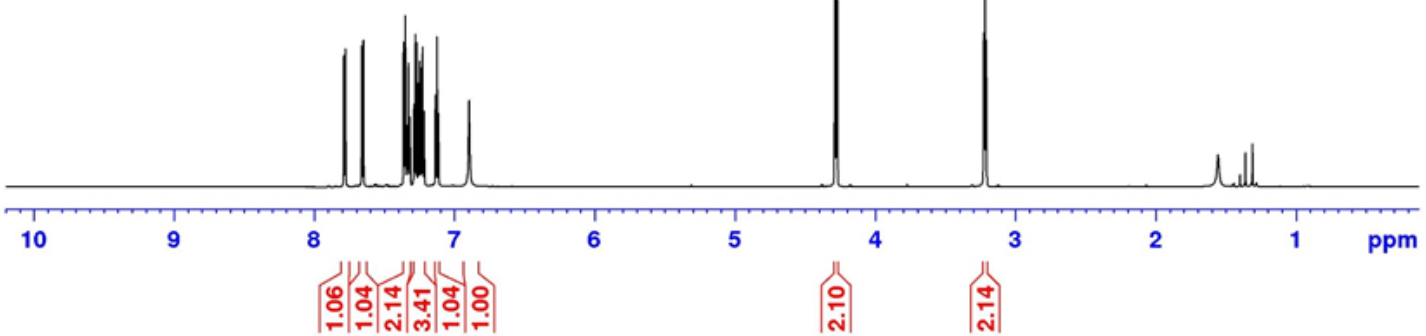


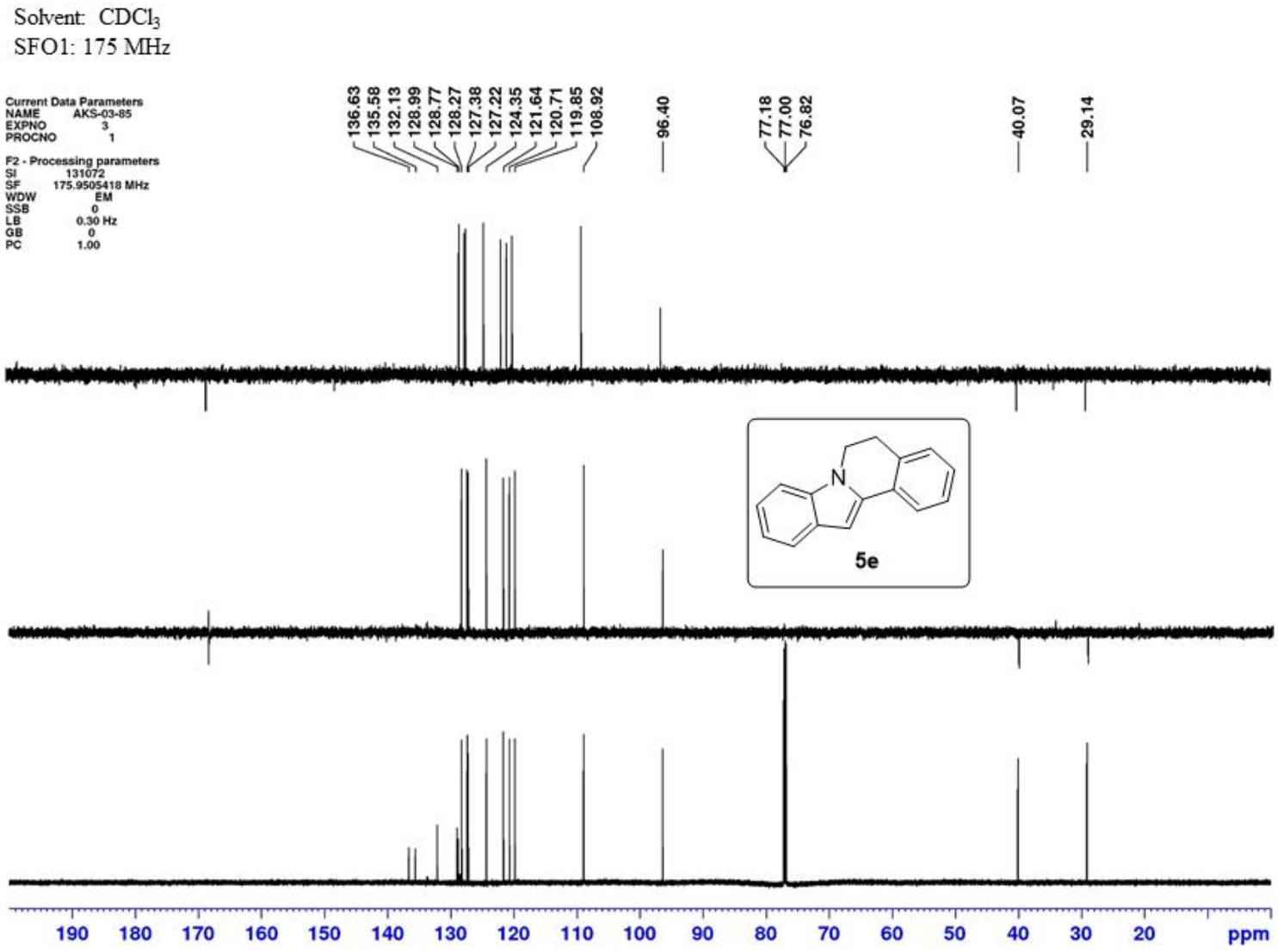
Current Data Parameters
NAME AKS-03-85
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7430808 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

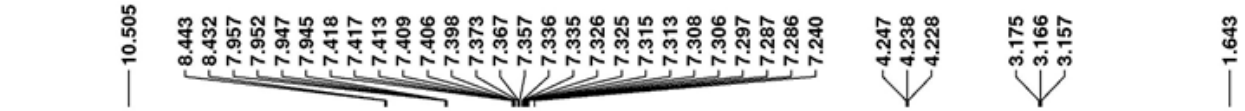


5e



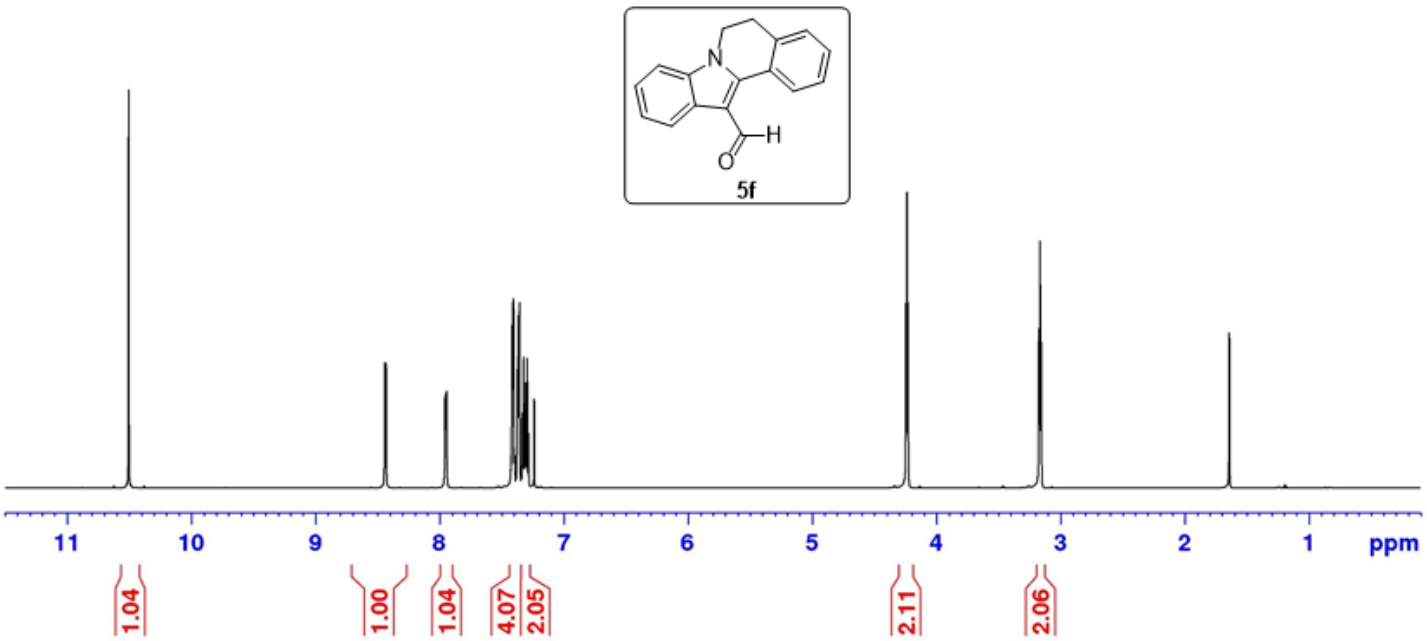

Solvent: CDCl3
SFO1: 175 MHz
Current Data Parameters
NAME AKS-03-85
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505418 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
136.63
135.58
132.13
128.99
128.77
128.27
127.38
127.22
124.35
121.64
120.71
119.85
108.92
96.40
77.18
77.00
76.82
40.07
29.14
N
5e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

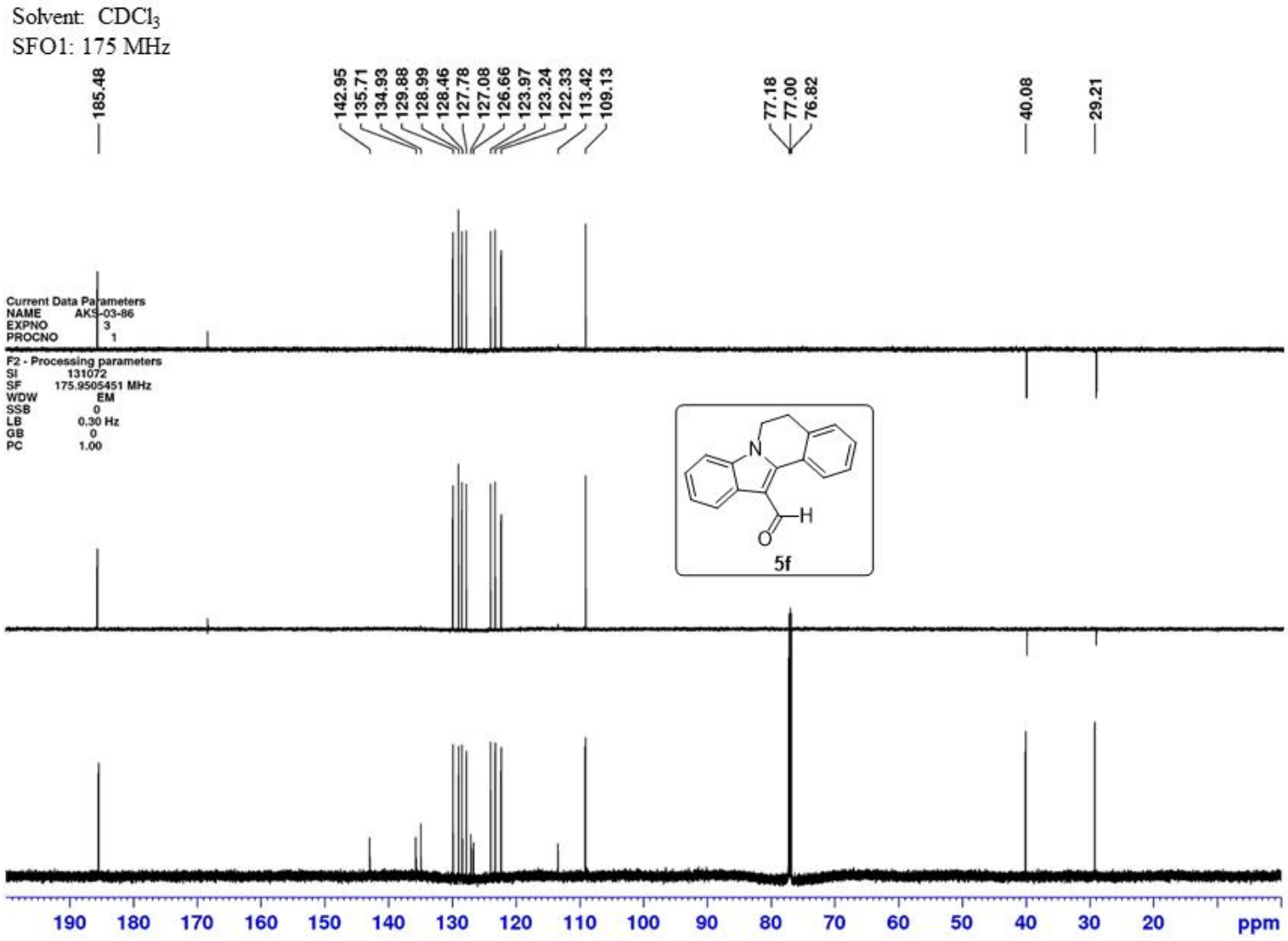
Solvent: $CDCl_3$
SFO1: 700 MHz



Current Data Parameters
NAME AKS-03-86
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431009 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




Solvent: $CDCl_3$
SFO1: 175 MHz
185.48
142.95
135.71
134.93
129.88
128.99
128.46
127.78
127.08
126.66
123.97
123.24
122.33
113.42
109.13
77.18
77.00
76.82
40.08
29.21
Current Data Parameters
NAME AKS-03-86
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505451 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
H
O
5f
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm