Supporting Information

Relay Zn(II)- and Au(I)-Catalyzed Aziridination/Cyclization/Ring Expansion Sequence to form 3-Benzazepine Derivatives

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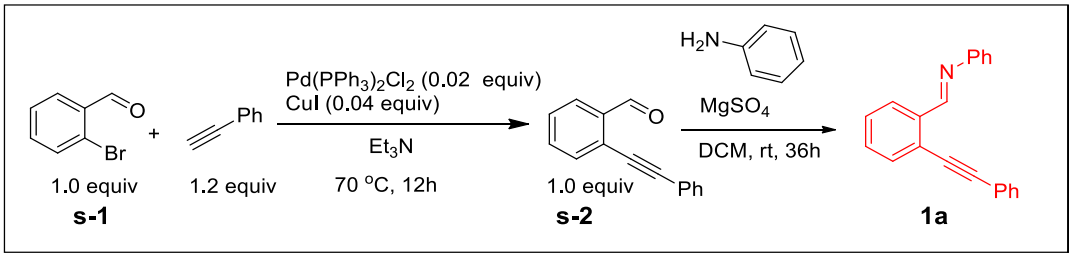
Content:

(1) Representative Synthetic Procedures:

(a) General procedure.

Unless otherwise noted, 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. DCM, DCE, and toluene were distilled from CaH_2 under nitrogen. THF was 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 Varian 500, Varian 700 MHz, Bruker 400 MHz spectrometers using chloroform-*d* ($CDCl_3$) as the internal standard. High-resolution mass spectral analysis (HRMS) data were measured on JMST100LP4G (JEOL) mass spectrometer or a TOF mass analyzer equipped with the ESI source, JEOL Model: JMS-T200GC AccuTOF GCx equipped with FD (field desorption) 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.

(b) Synthesis of (*E*)-*N*-(2-(phenylethynyl)benzylidene)aniline (1a). [s1]



Synthesis of 2-(phenylethynyl)benzaldehyde (s-2).

A round bottom flask was charged with $Pd(PPh_3)_2Cl_2$ (76.6 mg, 0.010 mmol, 0.02 equiv) and CuI (41.5 mg, 0.020 mmol, 1.25 mol%, 0.04 equiv). To this mixture was added Et_3N (10 mL). The solution was kept stirring and 2-bromobenzaldehyde **s-1** (1.0 g, 5.46 mmol, 1.0 equiv) was added. Finally, phenylacetylene (0.66 g, 6.55 mmol, 1.2 equiv.) was added using a syringe over 10 min at 25^0C. Then the mixture was stirred at room temperature for 12 h. After completion of the reaction, the reaction mixture was filtered through celite and concentrated under reduced pressure to afford crude. The residue was purified on a silica gel column to give 2-(phenylethynyl)benzaldehyde **s-2** (1.02 g, 4.97 mmol, 91%) as a brown oil.

Synthesis (*E*)-*N*-(2-(phenylethynyl)benzylidene)aniline (1a).

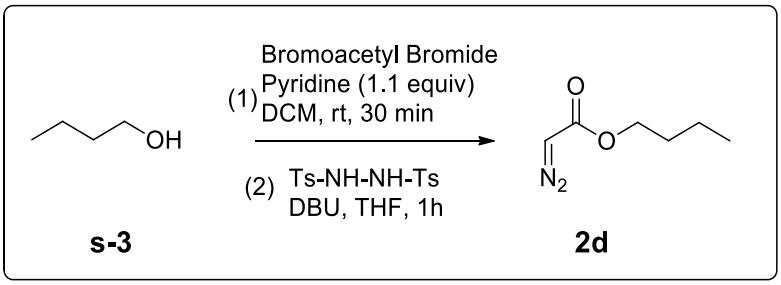
To a stirred solution of 2-(phenylethynyl)benzaldehyde **(s-2)** (0.5 g 2.4 mmo, l.0 equiv) in DCM (4.0 mL) were added $MgSO_4$ (1.0 g) and aniline (0.22 g, 2.4 mmol, 1.0 equiv) at room temperature. The resulting mixture was stirred at rt for 36 h, then this reaction mixture was filtered through a short celite bed and the filtrate was concentrated under reduced pressure to afford ((*E*)-*N*-(2-(phenylethynyl)benzylidene)aniline **(1a)** (0.491 g, 1.74 mmol, 72%). Crude product of **1a** was used directly for further catalytic reaction.

Other Substrates **1b-1t** were synthesized according to the same procedure of **b.**

All Imine **(1a-1n)** were prepared from procedure reported in the literatures.[s1]

Substrates **1u** was synthesized according to the literature procedure.[s2]

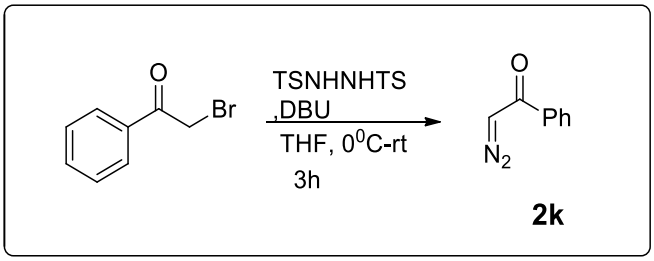
(c) Synthesis of α-diazo esters (2d).[s3]



To a solution of n-butyl alcohol **s-3** (0.50 g, 6.75 mmol, 1.0 equiv) and pyridine (0.58 g, 7.43 mmol, 1.1 equiv) in DCM (20 mL) was added bromoacetyl bromide (1.61 g, 8.1 mmol, 1.2 equiv) 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 (0.90 g, 4.6 mmol, 1.0 equiv) and *N*,*N'*-ditosylhydrazine (3.1 g, 9.2 mmol, 2.0 equiv) were dissolved in THF (20 mL), and cooled to 0 °C, and to this mixture was added DBU (1.05 g, 6.9 mmol, 1.5 equiv) was added slowly over 5 min. After stirring for 1h at the same temperature, the reaction solution was quenched with saturated $NaHCO_3$ and extracted with Et_2O 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 butyl 2-diazoacetate **2d** (0.579 g, 4.08 mmol, 88%) as a pale-yellow oil.

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

(d) Synthesis of 2-diazo-1-phenylethanone (2k).

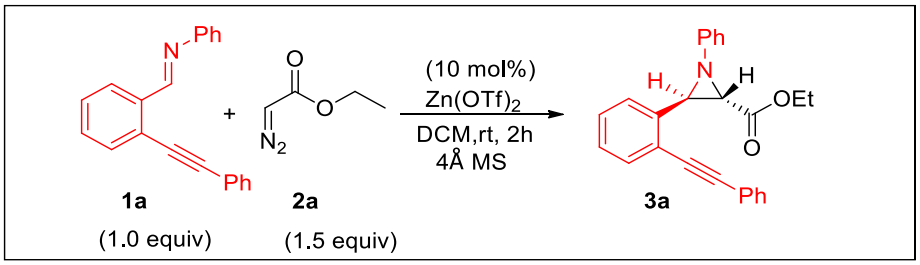


In a well-dried 200-mL flask, 2-bromo-1-phenylethanone (1.0 g, 5.0 mmol) and *N,N'*-ditosylhydrazine (3.4, 10 mmol) was dissolved in 30 mL THF, and a DBU (3.7 mL, 25.0 mmol) was added dropwise with stirring. The reaction mixture was stirred at room temperature for 3h. the reaction solution was quenched with saturated H_2O and extracted with Et_2O 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 (8/92) as the eluent to give desired 2-diazo-1-phenylethanone **2k** (0.554 g, 3.8 mmol, 76%) as a yellow solid.

(diazomethyl)trimethylsilane **2l** is commercially available from thermo scientific.

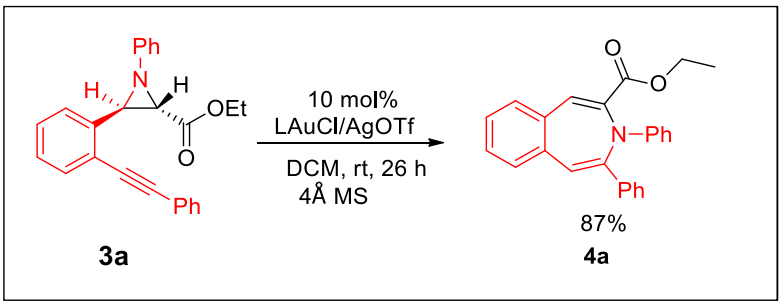
2. Standard procedures for catalytic operations:

(a) Typical procedure for (*2S,3R*)-ethyl 1-phenyl-3-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate (3a):



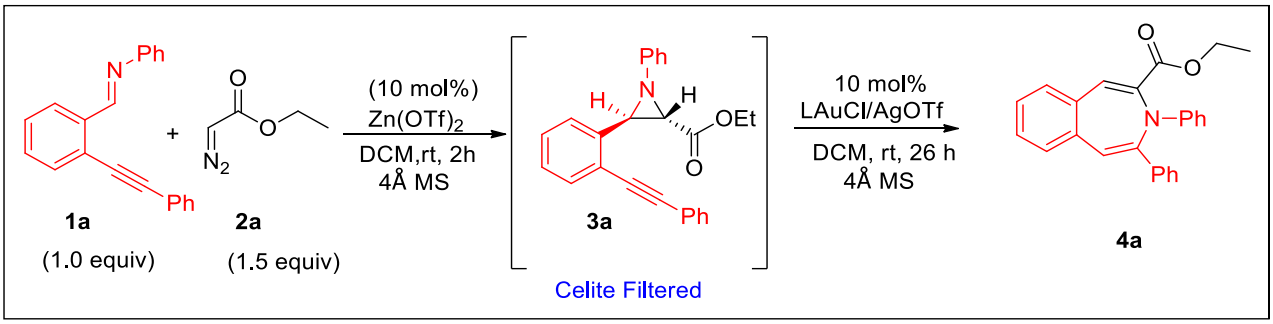
A suspension of $Zn(OTf)_2$ (7.7 mg, 0.021 mmol) and 4 Å MS (30-40 mg) in dry DCM (1.0 mL) was fitted with N_2 balloon and the mixture was stirred at 25^0C for 2 min at 25 °C. To this solution was added a DCM (3 mL) solution of (*E*)-N-(2-(phenylethynyl)benzylidene)aniline **1a** (60 mg, 0.21 mmol) and ethyl 2-diazoacetate **2a** (36.5 mg, 0.32 mmol) and the reaction was further stirred at 25°C for 2h. After completion of the reaction, the solution was filtered over a short celite bed. The solvent was evaporated to dryness under reduced pressure, and the residue was purified on a neutral alumina column using ethyl acetate/hexane (2:98) as the eluent to give compound (*2S,3R*)-ethyl 1-phenyl-3-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate **3a** (56 mg, 0.15 mmol, 71%) as off-white solid.

(b) Typical procedure for ethyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (4a):



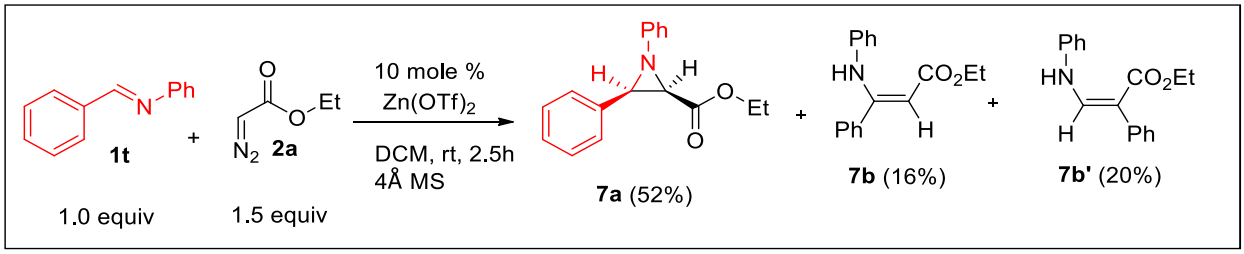
A suspension of LAuCl (L= P(*t*-Bu)$_2$(*o*-biphenyl)) (7.2 mg, 0.013 mmol) and AgOTf (3.5 mg, 0.013 mmol) in dry DCM (1.0 mL) was fitted with a N_2 balloon, and the mixture was stirred at 25 °C for 5 min. To this mixture was added a dry DCM (3.0 mL) solution of ethyl 1-phenyl-3-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate **3a** (50 mg, 0.13 mmol) at 25 °C. The resulting mixture was stirred at 25 °C for 26 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 (03:97) as the eluent to give ethyl 3,4-diphenyl-3H-benzo[d]azepine-2-carboxylate **4a** as yellow solid (43.5 mg, 0.11 mmol, 87%).

(c) Typical procedure for synthesis of (ethyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (4a):



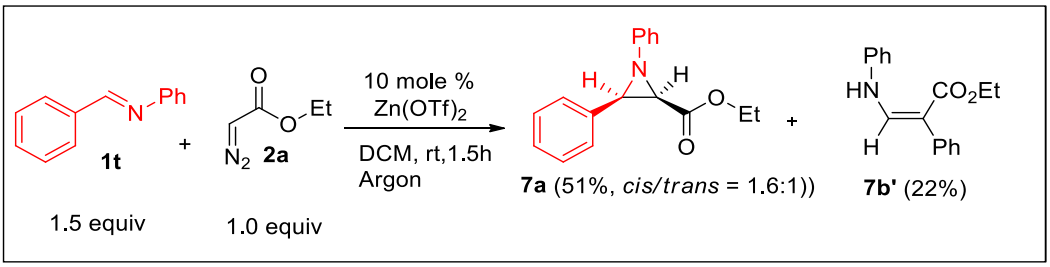
A suspension of Zn(OTf)$_2$ (7.7 mg, 0.021 mmol) and 4 Å MS (30-40 mg) in dry DCM (1.0 mL) was fitted with N_2 balloon and the mixture was stirred at 25^{0}C for 2 min at 25 °C. To this solution was added a DCM (3 mL) solution of (*E*)-N-(2-(phenylethynyl)benzylidene)aniline **1a** (60 mg, 0.21 mmol) and ethyl 2-diazoacetate **2a** (36.5 mg, 0.32 mmol) and the reaction was further stirred at 25°C for 2h. After completion of the reaction, the solution was filtered over a short celite bed; to the filtrate was added LAuCl (L= P(*t*-Bu)$_2$(*o*-biphenyl)) (11.3 mg, 0.021 mmol), AgOTf (5.4 mg, 0.021 mmol) and 4 Å MS in dry DCM (1.0 mL) under the N_2 atmosphere. The resulting mixture was stirred at 25 °C for 26 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 (03:97) as the eluent to give ethyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate **4a** as yellow solid (59.5 mg, 0.16 mmol, 76%).

(d) Typical procedure for (2R,3R)-ethyl 1,3-diphenylaziridine-2-carboxylate (7a):



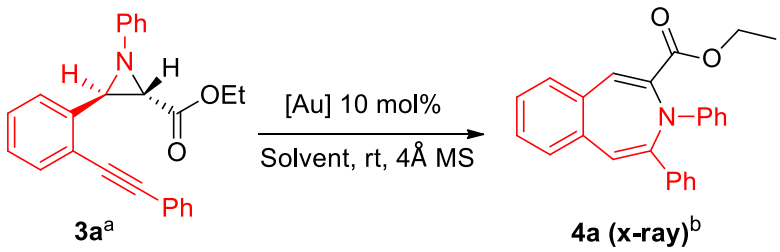
A suspension of $Zn(OTf)_2$ (16.0 mg, 0.044 mmol) and 4 Å MS (20-30 mg) in dry DCM (1.0 mL) was fitted with N_2 balloon and the mixture was stirred at 25^0C for 2 min at 25 °C. To this solution was added a DCM (3 mL) solution of (*E*)-N-benzylideneaniline **1t** (80 mg, 0.44 mmol) and ethyl 2-diazoacetate **2a** (75.5 mg, 0.66 mmol) and the reaction was further stirred at 25°C for 2.5h. After completion of the reaction, the solution was filtered over a short celite bed. The solvent was evaporated to dryness under reduced pressure to afford the crude product which was analyzed by ^{1}HNMR. Crude ^{1}H NMR showed only *cis*-aziridine product peaks and *trans*-aziridine product peaks not observed in crude data. The residue was purified on a silica column using ethyl acetate/hexane (3:97) as the eluent to give (*2R,3R*)-ethyl 1,3-diphenylaziridine-2-carboxylate **7a** (61.3 mg, 0.22 mmol, 52%) as white solid.

(e) Typical procedure for ethyl 1,3-diphenylaziridine-2-carboxylate (7a): (Jørgensen Procedure)[s4]



The $Zn(OTf)_2$ (25.5 mg,0.070 mmol) was added to a 10-ml Schlenk flask which was evacuated and filled twice with Ar. The DCM (4 ml) was added followed by the (*E*)-N-benzylideneaniline **1t** (190 mg, 1.05 mmol) and ethyl 2-diazoacetate **2a** (80 mg, 0.70 mmol). When the evolution of N_2 ceased the reaction mixture was filtered through a plug of silica gel, which was washed with additional CH_2Cl_2 (5 ml) and evaporated *in vacuo* to give the crude product which was analyzed by ^{1}H NMR. Crude ^{1}H NMR showed a mixture of *cis*/*trans* aziridine = 1.6:1. The crude product was purified by short flash column chromatography to give ethyl 1,3-diphenylaziridine-2-carboxylate (**7a**) (95.5 mg, 0.35 mmol, 51%).

3. Table S1: Optimization of Gold Catalysts:



3a[a] **4a (x-ray)**[b]

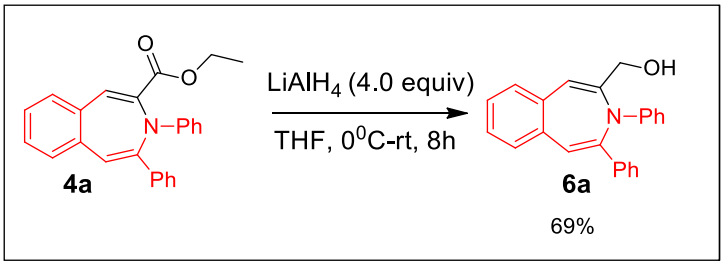
Entry	[Catalyst] (10 mol%)	Time (h)	Solvent	Yield (%)[b] **4a**
1	$PPh_3AuCl/AgOTf$	30	DCM	71%
2	$(PhO)_3PAuCl/AgOTf$	34	DCM	63%
3	(2,4-*t*-BuPhO)$_3$PAuCl/AgOTf	27	DCM	68%
4	IPrAuCl/AgOTf	28	DCM	78%
5	**LAuCl/AgOTf**	**26**	**DCM**	**87%**
6	$LAuCl/AgNTf_2$	30	DCM	80%
7	$LAuCl/AgSbF_6$	28	DCM	75%
8	LAuCl/ANaBARF	23	DCM	54%
9	AgOTf	31	DCM	69%
10	LAuCl/AgOTf	25	DCE	79%
11	LAuCl/AgOTf	32	Toluene	73%

[a]**1a=** 0.03M. [b]Product yields are obtained after purification from a silica column. L = P(*t*-Bu)$_2$(*o*-biphenyl). IPr=1,3-bis(diisopropylphenyl) imidazol-2-ylidene.

We optimized reaction condition for formation **4a**. Table **S1** shows the optimizations of reaction conditions of ethyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate **4a** using various catalysts; the results are summarized in Table **S1**. Our initial test of $PPh_3AuCl/AgOTf$ and $P(PhO)_3AuCl/AgOTf$ in dry DCM near 25 °C delivered **4a** in 63−71% yield, (entries 1-2). For (2,4-t-BuPhO)$_3$PAuCl/AgOTf in DCM, compound **4a** was obtained in 68% yield (entry 3). Next we tested, IPrAuCl/AgOTf in DCM, yielding compound **4a** in 78% yield (entry 4). With LAuCl/AgOTf, (L = P(*t*-Bu)$_2$(*o*-biphenyl)) we observed an enhanced efficiency, giving compound **4a** in 87% yield (entry 5). Variations of silver salts as in LAuCl/AgX, (X = NTf_2, SbF_6) in DCM afforded compound **4a** in 75-80% yields (entries 6-7). For LAuCl/NaBARF (L = P(*t*-Bu)$_2$(*o*-biphenyl)) in DCM, compound **4a** was obtained in 54% yield (entry 8). With AgOTf (10 mol %), the yield of compound **4a** in 69% yield (entry 9). For LAuCl/AgOTf, (L = P(*t*-Bu)$_2$(*o*-biphenyl)) in other solvents, the yields of compound **4a** were as follows: DCE (79%), toluene (73%) (entries 10−11).

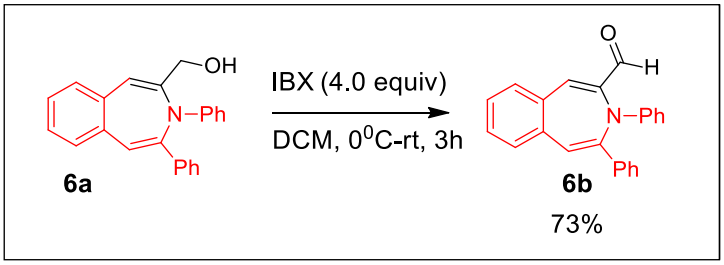
4. Synthetic procedures for chemical functionalizations:

(a) Synthesis of (3,4-diphenyl-3*H*-benzo[d]azepin-2-yl)methanol (6a):



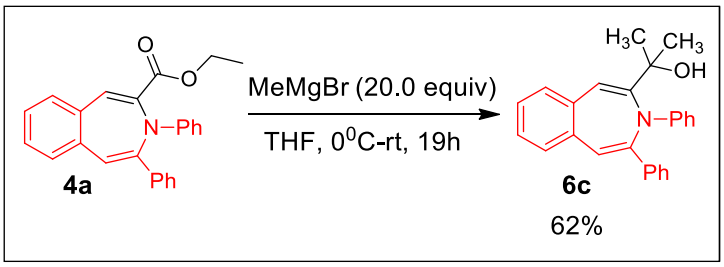
To a stirred solution of ethyl 3,4-diphenyl-3H-benzo[d]azepine-2-carboxylate **4a** (80 mg, 0.217 mmol) in dry THF (3.0 mL) at 0 ºC was added $LiAlH_4$ (21.6 mg, 0.58 mmol). The resulting mixture was stirred for 6h at 25^{0}C, confirmed the completion of reaction using TLC. Reaction mixture was quenched with saturated solution of NH_4Cl, followed by separation of organic and aqueous layer. The solvent was evaporated under reduced pressure, and eluted through a silica column with ethyl acetate/hexane (20:80) to yield compound ((3,4-diphenyl-3H-benzo[d]azepin-2-yl)methanol **6a** (49 mg, 0.15 mmol, 69 %) as light-yellow oil.

(b) Synthesis of 3,4-diphenyl-3*H*-benzo[d]azepine-2-carbaldehyde (6b):



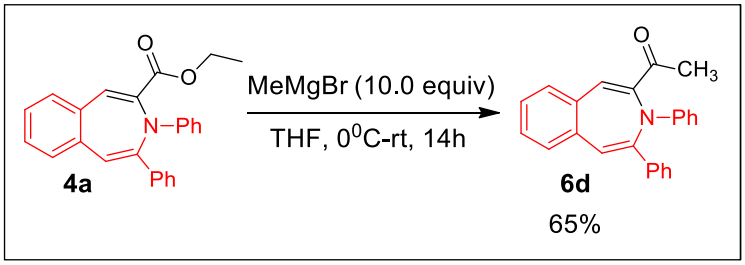
To a stirred solution of ((3,4-diphenyl-3H-benzo[d]azepin-2-yl)methanol **6a** (45 mg, 0.13 mmol) in dry DCM (4.0 mL) at 0 ºC was added IBX (155.0 mg, 0.55 mmol). The resulting mixture was stirred for 3h at 0-25^{0}C, confirmed the completion of reaction using TLC. Reaction mixture was filtered through celite and concentrated under reduced pressure, and eluted through a silica column with ethyl acetate/hexane (13:87) to afford compound 3,4-diphenyl-3*H*-benzo[d]azepine-2-carbaldehyde **6b** (32.6 mg, 0.10 mmol, 73 %) as light-yellow oil.

(c) Synthesis of 2-(3,4-diphenyl-3*H*-benzo[d]azepin-2-yl)propan-2-ol (6c) :



To a stirred solution of ethyl 3,4-diphenyl-3H-benzo[d]azepine-2-carboxylate **4a** (60 mg, 0.16 mmol) in dry THF (4.0 mL) at 0 ºC was added MeMgBr (1.0 mL, 3.0 M in Et_2O, 3.26 mmol). The resulting mixture was stirred for 19h at 0-25^{0}C, confirmed the completion of reaction using TLC. Reaction mixture was quenched with saturated solution of NH_4Cl, followed by the separation of organic and aqueous layer. The solvent was evaporated under reduced pressure, and eluted through a silica column with ethyl acetate/hexane (18:82) to yield compound 2-(3,4-diphenyl-3*H*-benzo[d]azepin-2-yl)propan-2-ol **6c** (36 mg, 0.10 mmol, 62 %) as yellow oil.

(d) Synthesis of 1-(3,4-diphenyl-3*H*-benzo[d]azepin-2-yl)ethanone (6d) :



To a stirred solution of ethyl 3,4-diphenyl-3H-benzo[d]azepine-2-carboxylate **4a** (60 mg, 0.16 mmol) in dry THF (4.0 mL) at 0 ºC was added MeMgBr (0.5 mL, 3.0 M in Et_2O, 1.6 mmol). The resulting mixture was stirred for 14h at 0-25^{0}C, confirmed the completion of reaction using TLC. Reaction mixture was quenched with saturated solution of NH_4Cl, followed by the separation of organic and aqueous layer. The solvent was evaporated under reduced pressure, and eluted through a silica column with ethyl acetate/hexane (10:90) to afford compound 1-(3,4-diphenyl-3*H*-benzo[d]azepin-2-yl)ethanone **6d** (35.8 mg, 0.10 mmol, 65 %) as yellow oil.

5. References:

[s1] (a) H. Luo, R. Liang, L. Chen, H. Jiang and S. Zhu, A silver-catalyzed three-component reaction via stabilized cation: synthesis of polysubstituted tetrahydronaphthols and tetrahydronaphthylamines. *Org. Chem. Front.*, 2018, **5,** 1160–1164; (b) T. Xiao, P. Peng, Y. Xie, Z.-Y. Wang and L. Zhou, Ag(I)-Catalyzed Three-Component Reaction of 2-Alkynylbenzaldehydes, Amines, and Diazo Compounds, *Org. Lett*., 2015, **17**, 4332−4335; (c) T. Y. Chaudhari, Urvashi, S. K. Ginotra, P. Yadav, G. Kumar and V. Tandon, Regioselective synthesis of functionalized dihydroisoquinolines from *o*-alkynylarylaldimines via the Reformatsky reaction, *Org. Biomol. Chem.*, 2016, **14,** 9896–9906; (d) N. Asao, Yudha S. S, T. Nogami and Y. Yamamoto, Direct Mannich and Nitro-Mannich Reactions with Non-Activated Imines: AgOTf-Catalyzed Addition of Pronucleophiles to ortho-Alkynylaryl Aldimines Leading to 1,2-Dihydroisoquinolines, *Angew. Chem.*, 2005, **117,** 5662 –5664.

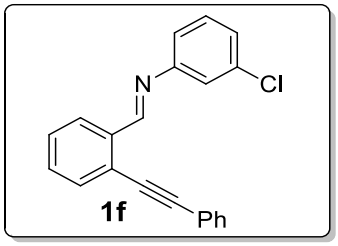
[s2] A. S. Narode and R-S. Liu, Gold-Catalyzed Bicyclic Annulations of *N*-(*o*-Alkynylphenyl)imines with α-Diazo Esters to Form 5,6-Dihydroindolo[2,1-a]isoquinolines; *Org. Lett*., 2022, **24,** 2165−2169.

[s3] **(**a) R. D. Kardile and R.-S. Liu, Two Distinct Ag(I)- and Au(I)-Catalyzed Olefinations between α-Diazo Esters and N-Boc-Derived Imines, *Org. Lett.*, 2019, **21,** 6452−6456; (b) A. S. Narode and R-S. Liu, Gold-Catalyzed Bicyclic Annulations of *N*-(*o*-Alkynylphenyl)imines with α-Diazo Esters to Form 5,6-Dihydroindolo[2,1-a]isoquinolines, *Org. Lett*., 2022, **24,** 2165−2169; (c) T. Toma, J. Shimokawa and T. Fukuyama, *N,N′*-Ditosylhydrazine: A Convenient Reagent for Facile Synthesis of Diazoacetates, *Org. Lett.*, 2007, **9,** 3195–3197; (d) H. Mao, A. Lin, Y. Shi, Z. Mao, X. Zhu, W. Li, H. Hu, Y. Cheng and C. Zhu, Construction of Enantiomerically Enriched Diazo Compounds UsingDiazo Esters as Nucleophiles: Chiral Lewis Base Catalysis, *Angew. Chem. Int., Ed*., 2013, **52,** 6288–6292; (e) D. M. Hodgson and D. Angrish, Highly Chemo- and Stereoselective Intermolecular Coupling of Diazoacetates To Give *cis*-Olefins by Using Grubbs Second-Generation Catalyst, *Chem. Eur. J*., 2007, **13,** 3470 – 3479; (f) D. M. Carminati, D. Intrieri, A. Caselli, S. Le Gac, B. Boitrel, L. Toma, L. Legnani, E. Gallo, Designing 'Totem' C2-Symmetrical Iron Porphyrin Catalysts for Stereoselective Cyclopropanations, *Chem. Eur. J*., 2016, **22,** 13599-13612; (g) H. S. A. Mandour, Y. Nakagawa, M. Tone, H. Inoue, N. Otog, I. Fujisawa, S. Chanthamath and S. Iwasa, Reusable and highly enantioselective water-soluble Ru(II)-Amm-Pheox catalyst for intramolecular cyclopropanation of diazo compounds, *Beilstein, J. Org. Chem*., 2019, **15,** 357–363.

[s4] K. G. Rasmussen and K. A. Jørgensen, Metal-catalysed reactions of imines with ethyl diazoacetate leading to aziridines, *J. Chem. Soc., Perkin Trans.1*., 1997, 1287-1291.

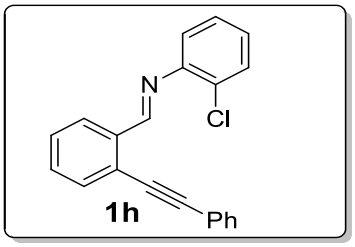
(6) Spectral data for key compounds:

Spectral data of (E)-3-chloro-N-(2-(phenylethynyl)benzylidene)aniline (1f):



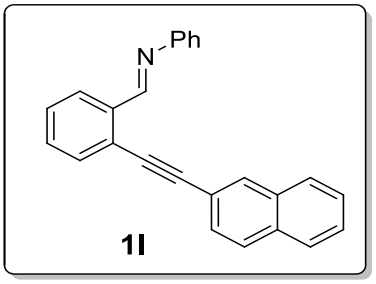
The compound **1f** was purified HPLC column using ethyl acetate/hexane:1/99 as the eluent; Brown oil (0.52 g, 1.65 mmol, 68%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.05 (s, 1H), 8.23 (d, *J* = 7.5 Hz, 1H), 7.60 (d, *J* = 7.0 Hz, 1H), 7.54 ~ 7.52 (m, 2H), 7.47 ~ 7.41 (m, 2H), 7.31 ~ 7.30 (m, 4H), 7.25 ~ 7.20 (m, 2H), 7.12 (d, *J* = 7.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.9, 153.4, 136.2, 134.7, 132.7, 131.5, 131.2, 130.2, 128.8, 128.7, 128.5, 126.7, 126.0, 125.3, 122.6, 121.2, 119.3, 95.7, 86.1; HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd. for $C_{21}H_{15}ClN$: 316.0893; found: 316.0894.

Spectral data of (E)-2-chloro-N-(2-(phenylethynyl)benzylidene)aniline (1h):



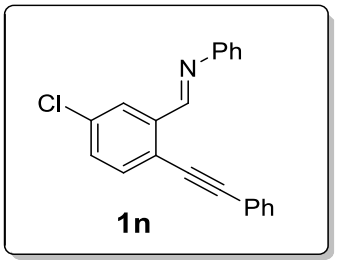
The compound **1h** was purified HPLC column using ethyl acetate/hexane:1/99 as the eluent; Brown oil (0.474 g, 1.50 mmol, 62%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.02 (s, 1H), 8.34 (d, *J* = 7.0 Hz, 1H), 7.62 ~ 7.60 (m, 1H), 7.53 ~ 7.51 (m, 2H), 7.48 ~ 7.43 (m, 3H), 7.36 ~ 7.34 (m, 3H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 7.08 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 160.4, 149.5, 136.2, 132.6, 131.5, 131.2, 129.9, 128.7, 128.46, 128.45, 128.0, 127.6, 126.9, 126.5, 125.2, 122.6, 120.0, 95.4, 86.0; HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd. for $C_{21}H_{15}ClN$: 316.0893; found: 316.0888.

Spectral data of (E)-N-(2-(naphthalen-2-ylethynyl)benzylidene)aniline (1l):



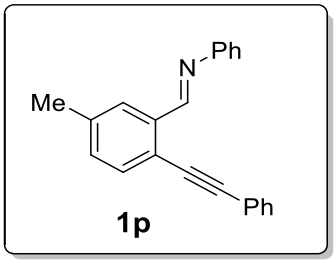
The compound **1l** was purified HPLC column using ethyl acetate/hexane: 2/98 as the eluent; Brown oil (0.465 g, 1.40 mmol, 72%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.16 (s, 1H), 8.29 (d, *J* = 6.8 Hz, 1H), 8.05 (s, 1H), 7.81 (d, *J* = 8.0 Hz, 3H), 7.66 ~ 7.63 (m, 1H), 8.55 (d, *J* = 7.2 Hz, 1H), 7.50 ~ 7.40 (m, 6H), 7.30 ~ 7.25 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.9, 152.2, 136.8, 133.04, 133.01, 132.8, 131.6, 130.9, 129.3, 128.7, 128.2, 127.8, 126.9, 126.7, 126.2, 125.1, 121.1, 120.1, 95.9, 86.7; HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd. for $C_{25}H_{18}N$: 332.1439; found: 332.1442.

Spectral data of (E)-N-(5-chloro-2-(phenylethynyl)benzylidene)aniline (1n):



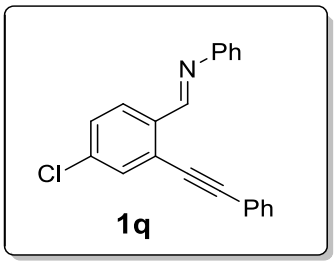
The compound **1n** was purified HPLC column using ethyl acetate/hexane: 2/98 as the eluent; Brown oil (0.452 g, 1.43 mmol, 69%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.01 (s, 1H), 8.26 (s, 1H), 7.53 ~ 7.50 (m, 3H), 7.43 ~ 7.34 (m, 6H), 7.27 ~ 7.24 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.2, 151.4, 138.0, 134.9, 133.7, 131.5, 130.8, 129.2, 128.8, 128.4, 126.55, 126.51, 123.2, 122.3, 121.0, 96.1, 85.2; HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd. for $C_{21}H_{15}N$: 316.0893; found: 316.0891.

Spectral data of (E)-N-(5-methyl-2-(phenylethynyl)benzylidene)aniline (1p):



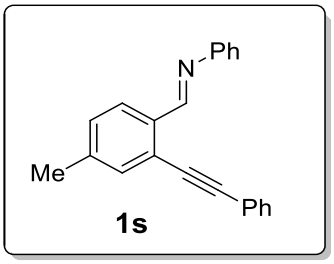
The compound **1p** was purified HPLC column using ethyl acetate/hexane:1/99 as the eluent; Brown oil (0.442 g, 1.49 mmol, 66%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.08 (s, 1H), 8.09 (s, 1H), 7.53 ~ 7.50 (m, 3H), 7.41 (t, *J* = 8.0 Hz, 2H), 7.35 ~ 7.34 (m, 3H), 7.28 ~ 7.25 (m, 4H), 2.44 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.1, 152.1, 139.0, 136.4, 132.6, 131.9, 131.4, 129.2, 128.5, 128.4, 126.8, 126.0, 122.9, 122.3, 121.0, 94.7, 86.4, 21.4; HRMS (FD) m/z: $[M]^+$ calcd. for $C_{22}H_{17}N$: 295.1366; found: 295.1361.

Spectral data of (E)-N-(4-chloro-2-(phenylethynyl)benzylidene)aniline (1q):



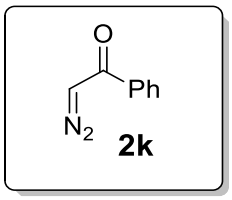
The compound **1q** was purified HPLC column using ethyl acetate/hexane:1/99 as the eluent; Brown oil (0.459 g, 1.45 mmol, 70%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.01 (s, 1H), 8.20 (d, *J* = 8.4 Hz, 1H), 7.58 (d, *J* = 2.4 Hz, 1H), 7.52 ~ 7.50 (m, 2H), 7.42 ~ 7.35 (m, 6H), 7.26 ~ 7.23 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.4, 151.7, 136.8, 135.1, 132.1, 131.6, 129.2, 129.0, 128.5, 127.9, 126.3, 122.2, 121.0, 96.4, 84.9; HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd. for $C_{21}H_{15}N$: 316.0893; found: 316.0894.

Spectral data of (E)-N-(4-methyl-2-(phenylethynyl)benzylidene)aniline (1s):



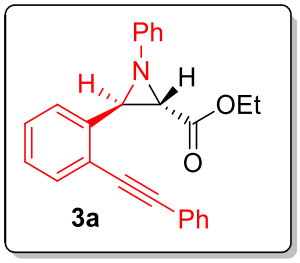
The compound **1s** was purified HPLC column using ethyl acetate/hexane:1/99 as the eluent; Brown oil (0.455 g, 1.54 mmol, 68%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.04 (s, 1H), 8.15 (d, *J* = 8.0 Hz, 1H), 7.52 ~ 7.50 (m, 2H), 7.42 ~ 7.33 (m, 6H), 7.25 ~ 7.20 (m, 4H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.8, 152.2, 141.3, 134.1, 133.0, 131.5, 129.8, 129.1, 128.6, 128.4, 126.5, 125.9, 124.9, 122.8, 121.0, 94.9, 86.4; HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd. for $C_{22}H_{18}N$: 296.1439; found: 296.1434.

Spectral data of 2-diazo-1-phenylethanone (2k):



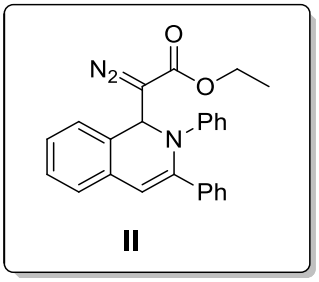
The compound **2k** was purified silica gel column using ethyl acetate/hexane:8/92 as the eluent; Yellow solid (0.554 g, 3.8 mmol, 76%); mp = 53-55°C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.74 (d, *J* = 7.0 Hz, 2H), 7.52 (t, *J* = 6.0 Hz, 1H), 7.42 (d, *J* = 6.0 Hz, 2H), 5.88 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 186.2, 136.5, 132.6, 128.5, 126.6, 54.1; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_8H_6N_2O$: 169.0377; found: 169.0380.

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



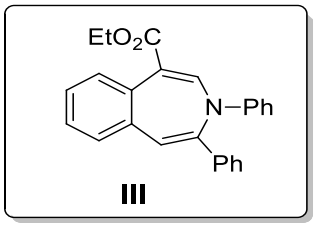
The compound **3a** was purified Neutral alumina column using ethyl acetate/hexane:2/98 as the eluent; Off-white solid (56 mg, 0.15 mmol, 71%); mp = 103-105°C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.55 ~ 7.54 (m, 1H), 7.48 ~ 7.46 (m, 2H), 7.37 ~ 7.28 (m, 6H), 7.22 (t, *J* = 8.0 Hz, 2H), 6.99 ~ 6.94 (m, 3H), 4.32 (d, *J* = 2.5 Hz, 1H), 4.06 ~ 3.95 (m, 2H), 3.19 (d, *J* = 2.0 Hz, 1H), 1.01 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.2, 148.8, 138.6, 131.58, 131.52, 128.8, 128.5, 128.4, 128.2, 127.6, 125.6, 122.9, 122.89, 122.80, 119.8, 94.9, 86.7, 61.1, 45.3, 45.2, 13.8; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{25}H_{21}NO_2Na$: 390.1470; found: 390.1473.

Spectral data of ethyl 2-diazo-2-(2,3-diphenyl-1,2-dihydroisoquinolin-1-yl)acetate(II):



The compound **II** was purified silica gel column using ethyl acetate/hexane:4/96 as the eluent; Yellow solid (64.0 mg, 0.16 mmol, 76 %); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.55 (d, *J* = 7.7 Hz, 2H), 7.34 ~ 7.30 (m, 2H), 7.27 ~ 7.17 (m, 7H), 7.12 (t, *J* = 7.7 Hz, 2H), 6.86 (t, *J* = 7.0 Hz, 1H), 6.63 (s, 1H), 6.16 (s, 1H), 4.36 (t, *J* = 5.6 Hz, 2H), 1.34 (s, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 166.1, 146.3, 141.9, 137.1, 132.0, 128.6, 128.37, 128.33, 128.1, 127.5, 127.3, 127.1, 125.7, 124.6, 122.5, 122.3, 112.5, 60.9, 60.3, 14.4; HRMS (ESI-TOF) m/z: $[M+H]^+$ $C_{25}H_{22}N_3O_2$: 396.1712; found: 396.1714.

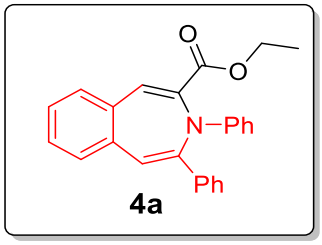
Spectral data of ethyl 3,4-diphenyl-3*H*-benzo[d]azepine-1-carboxylate (III):



III

The compound **III** was purified silica gel column using ethyl acetate/hexane:4/96 as the eluent; Yellow solid (25.0 mg, 0.06 mmol, 32 %); ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.03 (s, 1H), 7.47 (d, *J* = 7.7 Hz, 1H), 7.32 (d, *J* = 7.7 Hz, 2H), 7.28 ~ 7.20 (m, 6H), 7.07 (t, *J* = 8.4 Hz, 2H), 6.77 ~ 6.73 (m, 4H), 4.34 (q, *J* = 7.0 Hz, 2H), 1.37 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.2, 147.8, 147.4, 143.2, 137.2, 136.8, 133.9, 130.1, 128.8, 128.7, 128.6, 127.9, 127.3, 126.9, 126.3, 124.9, 124.5, 120.1, 114.3, 61.0, 14.3; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{25}H_{21}NO_2Na$: 390.1470.; found: 390.1459.

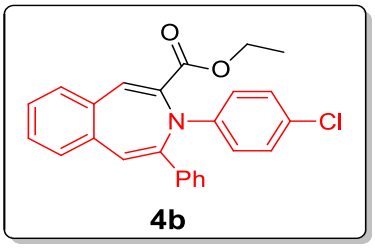
Spectral data of ethyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (4a):



4a

The compound **4a** was purified silica gel column using ethyl acetate/hexane:3/97 as the eluent; Yellow solid (59.5 mg, 0.16 mmol, 76%); mp = 130-132°C; ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.04 (d, *J* = 7.7 Hz, 2H), 8.00 (s, 1H), 7.41 ~ 7.29 (m, 7H), 7.23 (t, *J* = 7.7 Hz, 1H), 6.98 (t, *J* = 8.4 Hz, 2H), 6.65 (t, *J* = 7.0 Hz, 1H), 6.54 (d, *J* = 8.4 Hz, 2H), 4.35 (d, *J* = 7.0 Hz, 2H), 1.35 (t, *J* = 7.7 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.8, 145,7, 145,6, 138,8, 136.9, 136.8, 134.5, 133.2, 131.1, 130.3, 128.8, 128.6, 128.5, 127.0, 126.7, 124.2, 118.4, 112.5, 61.5, 14.3; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{25}H_{21}NO_2Na$: 390.1470; found: 390.1474.

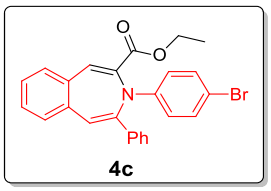
Spectral data of ethyl 3-(4-chlorophenyl)-4-phenyl-3*H*-benzo[d]azepine-2-carboxylate (4b):



4b

The compound **4b** was purified on silica gel column using ethyl acetate/hexane:3/97 as the eluent; Yellow solid (53 mg, 0.13 mmol, 69%); mp = 172-174°C; ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.02 ~ 8.01 (m, 3H), 7.42 ~ 7.31 (m, 7H), 7.26 (t, *J* = 7.7 Hz, 1H), 6.93 (d, *J* = 9.1 Hz, 2H), 6.47 (d, *J* = 9.1 Hz, 2H), 4.35 (s, 2H), 1.36 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.4, 145.3, 144.3, 139.2, 136.7, 136.5, 134.1, 133.1, 131.2, 130.4, 129.1, 128.8, 128.7, 128.4, 127.2, 126.6, 124.4, 123.4, 113.6, 61.6, 14.3; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{25}H_{20}ClNO_2Na$: 424.1080; found: 424.1084.

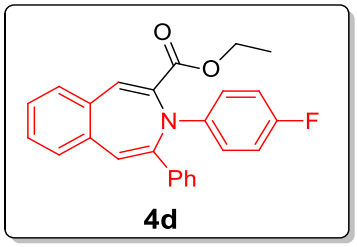
Spectral data of ethyl 3-(4-Bromophenyl)-4-phenyl-3*H*-benzo[d]azepine-2-carboxylate (4c):



4c

The compound **4c** was purified on silica gel column using ethyl acetate/hexane:3/97 as the eluent; Yellow solid (48 mg, 0.10 mmol, 64%); mp = 175-177°C; ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.99 ~ 7.98 (m, 3H), 7.41 ~ 7.29 (m, 7H), 7.25 (t, *J* = 7.0 Hz, 1H), 7.04 (d, *J* = 8.4 Hz, 2H), 6.39 (d, *J* = 9.1 Hz, 2H), 4.33 (s, 2H), 1.34 (t, *J* = 7.7 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.4, 145.1, 144.8, 139.2, 136.6, 136.4, 134.0, 133.0, 131.27, 131.21, 130.4, 129.1, 128.8, 128.7, 127.2, 126.6, 124.4, 114.1, 110.6, 61.6, 14.3; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{25}H_{20}BrNO_2Na$: 468.0575; found: 468.0579.

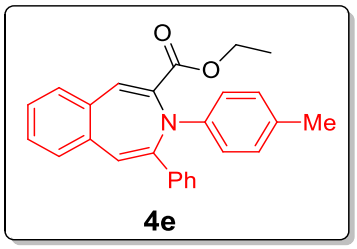
Spectral data of ethyl 3-(4-fluorophenyl)-4-phenyl-3*H*-benzo[d]azepine-2-carboxylate (4d):



4d

The compound **4d** was purified on silica gel column using ethyl acetate/hexane:4/96 as the eluent; Yellow oil (56 mg, 0.14 mmol, 73%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.03 (d, *J* = 7.7 Hz, 2H), 8.01 (s, 1H), 7.41 ~ 7.36 (m, 4H), 7.32 ~ 7.30 (m, 3H), 7.25 (t, *J* = 7.7 Hz, 1H), 6.69 (t, *J* = 8.4 Hz, 2H), 6.48 ~ 6.46 (m, 2H), 4.36 (q, *J* = 7.0 Hz, 2H), 1.36 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.6, 157.1, 155.8, 145.8, 141.96, 141.95, 139.1, 136.8, 134.6, 133.1, 131.1, 130.3, 128.9, 128.7, 128.6, 127.0, 126.6, 124.3, 115.0, 114.9, 113.19, 113.15 61.5, 14.2,; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{25}H_{20}FNO_2Na$: 408.1375; found: 408.1376.

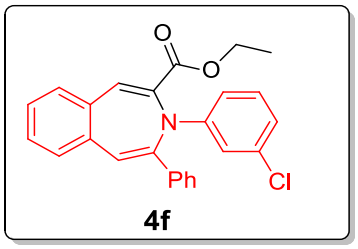
Spectral data of ethyl 4-phenyl-3-(*p*-tolyl)-3*H*-benzo[d]azepine-2-carboxylate (4e):



4e

The compound **4e** was purified on silica gel column using ethyl acetate/hexane:3/97 as the eluent; Yellow oil (52 mg, 0.13 mmol, 68%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.00 (d, *J* = 7.7 Hz, 2H), 7.94 (s, 1H), 7.35 ~ 7.30 (m, 4H), 7.27 ~ 7.24 (m, 3H), 7.20 ~ 7.17 (m, 1H), 6.75 (d, *J* = 9.1 Hz, 2H), 6.41 (d, *J* = 9.1 Hz, 2H), 4.31 (d, *J* = 6.3 Hz, 2H), 2.07 (s, 3H), 1.32 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.9, 146.0, 143.4, 138.8, 137.2, 136.9, 134.8, 133.3, 131.1, 130.3, 129.0, 128.8, 128.6, 128.4, 127.5, 126.9, 126.7, 124.1, 112.4, 61.4, 20.2, 14.3; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{26}H_{23}NO_2Na$: 404.1626; found: 404.1630.

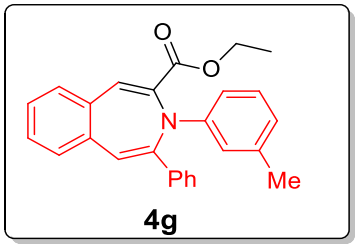
Spectral data of ethyl 3-(3-chlorophenyl)-4-phenyl-3*H*-benzo[d]azepine-2-carboxylate (4f):



4f

The compound **4f** was purified on silica gel column using ethyl acetate/hexane: 4/96 as the eluent; Yellow oil (49.6 mg, 0.12 mmol, 65%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.01 ~ 7.99 (m, 3H), 7.42 ~ 7.29 (m, 7H), 7.27 ~ 7.22 (m, 1H), 6.86 (t, *J* = 8.0 Hz, 1H), 6.60 (d, *J* = 8.0 Hz, 1H), 6.49 ~ 6.48 (m, 1H), 6.39 (dd, J = 2.5 Hz, 8.5 Hz, 1H), 4.34 (t, *J* = 7.0 Hz, 2H), 1.34 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.3, 146.9, 144.8, 139.3, 136.6, 134.3, 133.7, 133.0, 131.2, 130.4, 129.4, 129.1, 128.8, 128.7, 127.2, 126.6, 124.5, 118.5, 112.4, 110.8, 61.6, 14.3 ; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{25}H_{20}ClNO_2Na$: 424.1080; found: 424.1087.

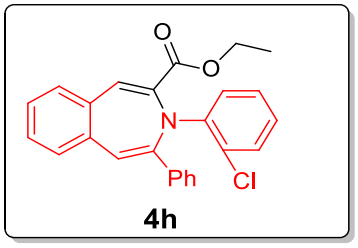
Spectral data of ethyl 4-phenyl-3-(*m*-tolyl)-3*H*-benzo[d]azepine-2-carboxylate (4g):



4g

The compound **4g** was purified on silica gel column using ethyl acetate/hexane:3/97 as the eluent; Yellow oil (47 mg, 0.12 mmol, 60%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.03 (d, *J* = 7.5 Hz, 2H), 7.98 (s, 1H), 7.40 ~ 7.27 (m, 7H), 7.23 ~ 7.20 (m, 1H), 6.85 (t, *J* = 7.5 Hz, 1H), 6.47 (d, *J* = 7.5 Hz, 1H), 6.34 ~ 6.32 (m, 2H), 4.34 (d, *J* = 7.0 Hz, 2H), 2.11 (s, 3H), 1.34 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.9, 145.75, 145.70, 138.7, 138.1, 137.0, 136.9, 134.6, 133.3, 131.1, 130.3, 128.8, 128.6, 128.4, 128.3, 126.9, 126.7, 124.2, 119.4, 113.0, 109.8, 61.4, 21.7, 14.3; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{26}H_{23}NO_2Na$: 404.1626; found: 404.1622.

Spectral data of ethyl ethyl 3-(2-chlorophenyl)-4-phenyl-3*H*-benzo[d]azepine-2-carboxylate (4h):

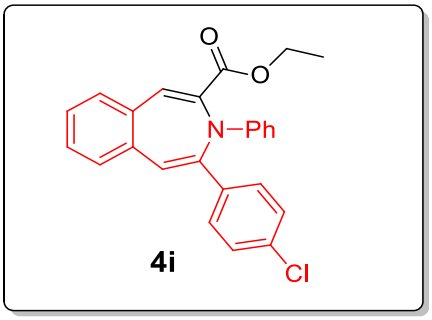


4h

The compound **4h** was purified on silica gel column using ethyl acetate/hexane:5/95 as the eluent; Yellow oil (43.5 mg, 0.10 mmol, 57%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.02 (d, *J* = 7.5 Hz, 2H),

7.88 (s, 1H), 7.50 (s, 1H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.36 ~ 7.30 (m, 4H), 7.28 ~ 7.26 (m, 1H), 7.08 (d, *J* = 7.5 Hz, 1H), 6.76 (d, *J* = 7.5 Hz, 1H), 6.69 (d, *J* = 7.5 Hz, 1H), 6.58 (t, *J* = 7.0 Hz, 1H), 4.28 (q, *J* = 7.0 Hz, 2H), 1.29 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 165.9, 145.1, 141.7, 137.1, 136.5, 136.0, 134.6, 133.4, 131.8, 130.9, 129.9, 128.6, 128.5, 128.4, 127.1, 126.8, 126.7, 124.9, 120.7, 120.2, 118.5, 61.2, 14.2; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{25}H_{20}ClNO_2Na$: 424.1080; found: 424.1077.

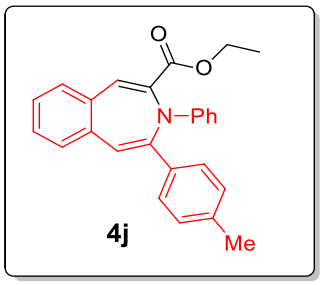
Spectral data of ethyl 4-(4-chlorophenyl)-3-phenyl-3*H*-benzo[d]azepine-2-carboxylate (4i):



4i

The compound **4i** was purified on silica gel column using ethyl acetate/hexane:3/97 as the eluent; Yellow soild (53 mg, 0.13 mmol, 70%); mp = 122-124°C; ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.98 ~ 7.97 (m, 3H), 7.40 ~ 7.36 (m, 2H), 7.32 ~ 7.29 (m, 4H), 7.24 (t, *J* = 7.7 Hz, 1H), 6.99 (t, *J* = 9.1 Hz, 2H), 6.66 (t, *J* = 7.7 Hz, 1H), 6.50 (d, *J* = 8.4 Hz, 2H), 4.34 (s, 2H), 1.35 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.7, 145.3, 144.5, 138.9, 136.6, 135.5, 134.4, 134.3, 133.2, 131.2, 130.3, 128.9, 128.8, 128.5, 128.0, 127.2, 124.5, 118.6, 112.4, 61.6, 14.3; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{25}H_{20}ClNO_2Na$: 424.1080; found: 424.1080.

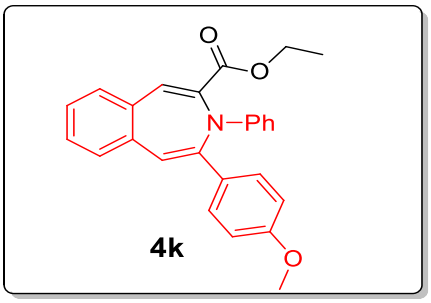
Spectral data of ethyl 3-phenyl-4-(*p*-tolyl)-3*H*-benzo[d]azepine-2-carboxylate (4j):



4j

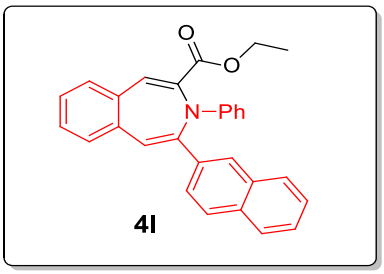
The compound **4j** was purified on silica gel column using ethyl acetate/hexane:4/96 as the eluent; Yellow soild (58 mg, 0.15 mmol, 72%); mp = 119-121°C; ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.99 (s, 1H), 7.92 (d, *J* = 8.4 Hz, 2H), 7.38 (q, *J* = 7.7 Hz, 2H), 7.29 ~ 7.28 (m, 2H), 7.23 ~ 7.21 (m, 1H), 7.16 (d, *J* = 7.7 Hz, 2H), 6.98 (t, *J* = 8.4 Hz, 2H), 6.64 (t, *J* = 7.7 Hz, 1H), 6.54 (d, *J* = 8.4 Hz, 2H), 4.34 (s, 2H), 2.35 (s, 3H), 1.35 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.8, 145.8, 145.6, 138.9, 138.5, 137.0, 134.4, 134.0, 133.2, 131.1, 130.2, 129.4, 128.8, 128.4, 126.8, 126.6, 123.3, 118.2, 112.4, 61.4, 21.2, 14.3; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{26}H_{23}NO_2Na$: 404.1626; found: 404.1634.

Spectral data of ethyl 4-(4-methoxyphenyl)-3-phenyl-3*H*-benzo[d]azepine-2-carboxylate (4k):



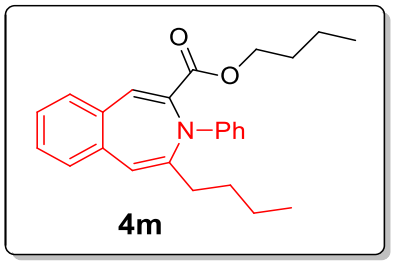
The compound **4k** was purified on silica gel column using ethyl acetate/hexane:4/96 as the eluent; Yellow oil (57 mg, 0.14 mmol, 74%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.99 ~ 7.96 (m, 3H), 7.39 ~ 7.35 (m, 2H), 7.28 (t, *J* = 7.0 Hz, 1H), 7.22 ~ 7.19 (m, 2H), 6.99 (t, *J* = 8.00 Hz, 2H), 6.88 (d, *J* = 9.0 Hz, 2H), 6.64 (t, *J* = 7.5 Hz, 1H), 6.54 (d, *J* = 8.0 Hz, 2H), 4.34 (s, 2H), 3.80 (s, 3H), 1.35 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.8, 159.9, 145.7, 145.3, 139.0, 137.1, 134.2, 133.0, 131.1, 130.1, 129.4, 128.8, 128.4, 128.2, 126.6, 122.2, 118.2, 114.0, 112.4, 61.4, 55.2, 14.3; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{26}H_{23}NO_3Na$: 420.1575; found: 420.1578.

Spectral data of ethyl 4-(naphthalen-2-yl)-3-phenyl-3*H*-benzo[d]azepine-2-carboxylate (4l):



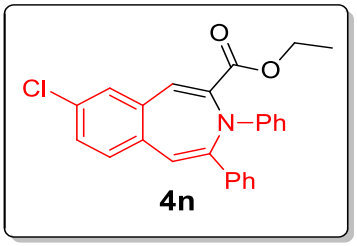
The compound **4l** was purified on silica gel column using ethyl acetate/hexane:3/97 as the eluent; Yellow soild (58 mg, 0.13 mmol, 76%); mp = 185-187°C; ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.59 (s, 1H), 8.12 (d, *J* = 8.4 Hz, 1H), 8.06 (s, 1H), 7.86 ~ 7.81 (m, 3H), 7.49 (s, 1H), 7.46 ~ 7.43 (m, 4H), 7.32 (t, *J* = 7.7 Hz, 1H), 7.26 ~ 7.23 (m, 1H), 7.00 (t, *J* = 7.7 Hz, 2H), 6.67 (t, *J* = 7.7 Hz, 1H), 6.62 (d, *J* = 8.4 Hz, 2H), 4.41 (q, *J* = 7.0 Hz, 2H), 1.41 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.8, 145.8, 145.6, 138.9, 136.8, 134.6, 134.2, 133.5, 133.3, 133.2, 131.1, 130.4, 128.9, 128.7, 128.5, 128.3, 127.5, 127.0, 126.6, 126.3, 126.1, 124.8, 124.0, 118.4, 112.5, 61.5, 14.4; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{29}H_{23}NO_2Na$: 440.1626; found: 440.1626.

Spectral data of butyl 4-butyl-3-phenyl-3*H*-benzo[d]azepine-2-carboxylate (4m):



The compound **4m** was purified on silica gel column using ethyl acetate/hexane:4/96 as the eluent; Yellow oil (57 mg, 0.15 mmol, 66%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (s, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 7.25 ~ 7.23 (m, 2H), 7.19 ~ 7.15 (m, 1H), 7.07 (t, *J* = 8.8 Hz, 2H), 6.68 (t, *J* = 7.2 Hz, 1H), 6.58 ~ 6.52 (m, 3H), 4.24 (t, *J* = 6.8 Hz, 2H), 2.54 (t, *J* = 7.6 Hz, 2H), 1.70 ~ 1..61 (m, 4H), 1.39 ~ 1.31 (m, 4H), 0.93 ~ 0.87 (m, 6H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 165.0, 149.6, 145.0, 137.3, 137.0, 134.4, 133.1, 130.9, 129.6, 128.6, 128.5, 126.3, 124.9, 118.0, 111.6, 65.1, 34.4, 30.71, 30.70, 22.4, 19.0, 13.9, 13.6; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd. for $C_{25}H_{29}NO_2Na$: 398.2096; found: 398.2098.

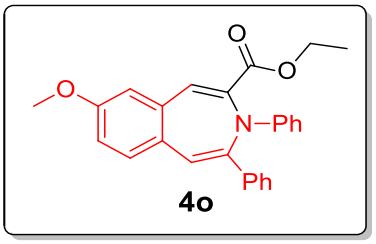
Spectral data of ethyl 8-chloro-3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (4n):



The compound **4n** was purified silica gel column using ethyl acetate/hexane:3/97 as the eluent;

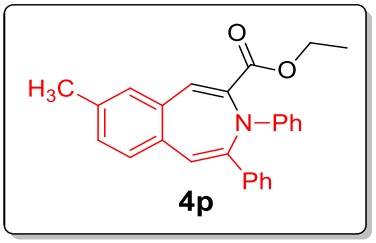
Yellow solid (54 mg, 0.13 mmol, 71%); mp = 191-193°C; ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.02 (d, *J* = 7.0 Hz, 2H), 7.91 (s, 1H), 7.39 ~ 7.35 (m, 3H), 7.31 (t, *J* = 7.0 Hz, 2H), 7.28 (s, 1H), 7.26 ~ 7.24 (m, 1H), 6.99 (t, *J* = 7.0 Hz, 2H), 6.68 (t, *J* = 7.7 Hz, 1H), 6.52 (d, *J* = 7.7 Hz, 2H), 4.35 (q, *J* = 6.3 Hz, 2H), 1.35 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.4, 146.2, 145.4, 137.5, 136.7, 135.7, 135.2, 134.7, 132.6, 131.6, 130.4, 128.9, 128.7, 128.5, 126.7, 123.3, 118.7, 112.5, 61.6, 14.2; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{25}H_{20}ClNO_2Na$: 424.1080; found: 424.1074.

Spectral data of ethyl 8-methoxy-3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (4o):



The compound **4o** was purified silica gel column using ethyl acetate/hexane:3/97 as the eluent; Yellow oil (57.4 mg, 0.14 mmol, 75%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.02 (d, *J* = 7.0 Hz, 2H), 7.97 (s, 1H), 7.35 (t, *J* = 7.7 Hz, 2H), 7.32 ~ 7.28 (m, 3H), 6.99 (t, *J* = 7.7 Hz, 2H), 6.89 ~ 6.87 (m, 2H), 6.66 (t, *J* = 7.0 Hz, 1H), 6.55 (d, *J* = 8.4 Hz, 2H), 4.35 (s, 2H), 3.78 (s, 3H), 1.35 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.8, 158.1, 145.7, 143.2, 138.6, 137.0, 134.5, 134.3, 131.8, 129.9, 128.6, 128.4, 128.1, 126.4, 124.0, 118.3, 116.3, 114.3, 112.3, 61.5, 55.3, 14.2; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{26}H_{23}NO_3Na$: 420.1575; found: 420.1576.

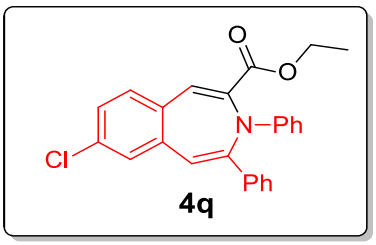
Spectral data of ethyl 8-methyl-3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (4p):



The compound **4p** was purified silica gel column using ethyl acetate/hexane:4/96 as the eluent; Yellow solid (57 mg, 0.14 mmol, 74%); mp = 170-172°C; ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.04 (d, *J* = 7.7 Hz, 2H), 7.98 (s, 1H), 7.37 ~ 7.33 (m, 3H), 7.31 ~ 7.28 (m, 2H), 7.22 (s, 1H), 7.11 (d,

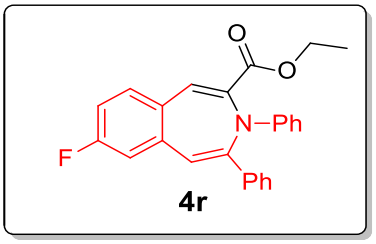
J = 8.4 Hz, 1H), 6.99 (t, *J* = 8.4 Hz, 2H), 6.65 (t, *J* = 7.0 Hz, 1H), 6.54 (d, *J* = 8.4 Hz, 2H), 4.35 (s, 2H), 2.32 (s, 3H), 1.35 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.9, 145.8, 144.6, 139.0, 137.0, 136.9, 134.2, 134.1, 133.1, 131.4, 130.3, 129.9, 128.6, 128.4, 128.3, 126.5, 124.2, 118.2, 112.4, 61.4, 21.0, 14.2; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{26}H_{23}NO_2Na$: 404.1626; found: 404.1626.

Spectral data of ethyl 7-chloro-3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (4q):



The compound **4q** was purified silica gel column using ethyl acetate/hexane:4/96 as the eluent; Yellow solid (59.5 mg, 0.14 mmol, 78%); mp = 188-190°C; ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.04 (d, *J* = 7.7 Hz, 2H), 7.95 (s, 1H), 7.38 (t, *J* = 7.0 Hz, 3H), 7.34 ~ 7.31 (m, 2H), 7.25 (s, 1H), 7.19 (dd, *J* = 2.1 Hz, 8.4 Hz, 1H), 7.01 (t, *J* = 8.4 Hz, 2H), 6.69 (t, *J* = 7.0 Hz, 1H), 6.54 (d, *J* = 7.7 Hz, 2H), 4.36 (q, *J* = 7.0 Hz, 2H), 1.36 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.5, 147.0, 145.4, 138.3, 137.9, 136.6, 134.9, 134.6, 132.3, 131.6, 129.8, 128.8, 128.7, 128.5, 127.2, 126.8, 123.0, 118.7, 112.5, 61.6, 14.2; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{25}H_{20}ClNO_2Na$: 424.1080; found: 424.1082.

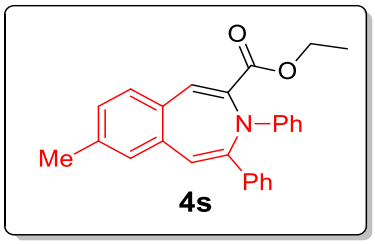
Spectral data of ethyl 7-fluoro-3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (4r):



The compound **4r** was purified silica gel column using ethyl acetate/hexane:5/95 as the eluent; Yellow oil (60 mg, 0.15 mmol, 77%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.05 (d, *J* = 7.0 Hz, 2H), 7.97 (s, 1H), 7.39 ~ 7.36 (m, 3H), 7.34 ~ 7.32 (m, 1H), 7.26 (s, 1H), 7.08 (dd, *J* = 2.1 Hz, 9.1 Hz, 1H), 7.01 (t, *J* = 8.4 Hz, 2H), 6.96 ~ 6.93 (m, 1H), 6.68 (t, *J* = 7.0 Hz, 1H), 6.55 (d, *J* = 7.7 Hz, 2H), 4.35 (d, *J* = 7.0 Hz, 2H), 1.36 (t, *J* = 7.7 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.6,

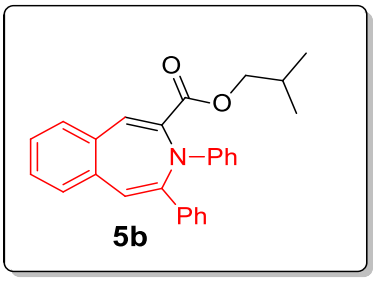
162.9, 161.5, 146.7, 145.4, 139.18, 139.13, 138.0, 136.6, 134.0, 133.3, 133.2, 129.6, 129.5, 128.8, 128.7, 128.5, 126.8, 123.2, 118.6, 116.2, 116.1, 114.8, 114.6, 112.5, 61.5, 14.2; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{25}H_{20}FNO_2Na$: 408.1375; found: 408.1380.

Spectral data of ethyl 7-methyl-3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (4s):



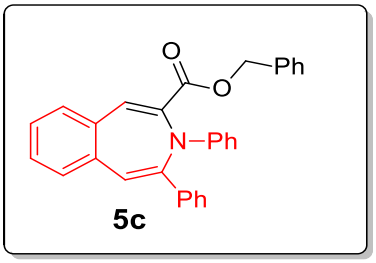
The compound **4s** was purified silica gel column using ethyl acetate/hexane:3/97 as the eluent; Yellow oil (33 mg, 0.08 mmol, 42%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.03 (d, *J* = 7.5 Hz, 2H), 7.98 (s, 1H), 7.36 ~ 7.29 (m, 5H), 7.20 (s, 1H), 7.05 (t, *J* = 7.5 Hz, 1H), 6.97 (t, *J* = 8.5 Hz, 2H), 6.63 (t, *J* = 7.5 Hz, 1H), 6.52 (d, *J* = 8.5 Hz, 2H)**,** 4.33 (s, 2H), 2.33 (s, 3H), 1.34 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.9, 145.8, 145.2, 139.1, 138.9, 137.0, 136.8, 133.5, 131.2, 130.7, 128.7, 128.5, 128.1, 126.6, 124.3, 118.3, 112.4, 61.4, 21.3, 14.3; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{26}H_{23}NO_2Na$: 404.1626; found: 404.1616.

Spectral data of isobutyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (5b):



The compound **5b** was purified silica gel column using ethyl acetate/hexane:3/97 as the eluent; Yellow oil (61 mg, 0.15 mmol, 73%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 ~ 7.99 (m, 3H), 7.41 ~ 7.23 (m, 8H), 6.98 (t, *J* = 7.2 Hz, 2H), 6.65 (t, *J* = 7.2 Hz, 1H), 6.54 (d, *J* = 8.8 Hz, 2H)**,** 4.07 (d, *J* = 6.8 Hz, 2H)**,** 2.06 ~ 2.00 (m, 1H), 0.93 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.9, 145.7, 145.6, 138.8, 137.0, 136.8, 134.6, 133.2, 131.1, 130.3, 128.8, 128.7, 128.4, 127.0, 126.6, 124.3, 118.4, 112.5, 71.8, 27.8, 19.2, 19.1; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{27}H_{25}NO_2Na$: 418.1783; found: 418.1785.

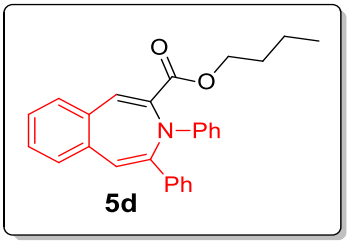
Spectral data of benzyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (5c):



5c

The compound **5c** was purified silica gel column using ethyl acetate/hexane:3/97 as the eluent; Yellow solid (63 mg, 0.14 mmol, 70%); mp = 133-135°C; ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.03 (s, 1H), 7.97 (d, *J* = 7.7 Hz, 2H), 7.39 ~ 7.21 (m, 13H), 6.98 (t, *J* = 7.7 Hz, 2H), 6.66 (t, *J* = 7.0 Hz, 1H), 6.54 (d, *J* = 7.7 Hz, 2H)**,** 5.32 (s, 2H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.6, 145.8, 145.5, 139.3, 136.9, 135.5, 134.3, 133.2, 131.2, 130.3, 128.9, 128.6, 128.58, 128.56, 128.52, 128.4, 128.3, 127.0, 126.6, 124.2, 118.5, 67.4; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{30}H_{23}NO_2Na$: 452.1626; found: 452.1621.

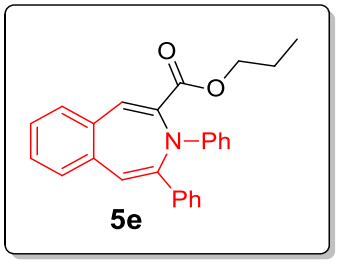
Spectral data of butyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (5d):



5d

The compound **5d** was purified silica gel column using ethyl acetate/hexane:2/98 as the eluent; Yellow solid (67 mg, 0.16 mmol, 79%); mp = 132-134°C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (d, *J* = 8.0 Hz, 2H), 8.01 (s, 1H), 7.43 ~ 7.30 (m, 7H), 7.27 ~ 7.23 (m, 1H), 7.01 (t, *J* = 8.8 Hz, 2H), 6.67 (t, *J* = 8.4 Hz, 1H), 6.56 (d, *J* = 8.4 Hz, 2H), 4.31 (t, *J* = 5.6 Hz, 2H), 1.76 ~ 1.69 (m, 2H), 1.43 ~ 1.34 (m, 2H), 0.94 (t, *J* = 7.6 Hz, 3H),; ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.9, 145.7, 145.6, 138.7, 137.0, 136.8, 134.6, 133.2, 131.1, 130.3, 128.8, 128.6, 128.4, 127.0, 126.6, 124.3, 118.4, 112.5, 65.4, 30.7, 19.1, 13.6; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{27}H_{25}NO_2Na$: 418.1783; found: 418.1780.

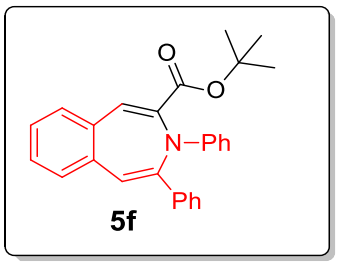
Spectral data of propyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (5e):



5e

The compound **5e** was purified silica gel column using ethyl acetate/hexane: 3/97 as the eluent; Yellow solid (62 mg, 0.16 mmol, 76%); mp = 130-132°C; ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.07 (d, *J* = 8.4 Hz, 2H), 8.03 (s, 1H), 7.43 ~ 7.37 (m, 4H), 7.35 ~ 7.30 (m, 3H), 7.25 (t, *J* = 7.0 Hz, 1H), 7.01 (t, *J* = 8.4 Hz, 2H), 6.68 (t, *J* = 7.7 Hz, 1H), 6.58 (d, *J* = 8.4 Hz, 2H), 4.28 (s, 2H), 1.79 ~ 1.76 (m, 2H), 0.96 (t, *J* = 7.7 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.9, 145.7, 145.6, 138.8, 137.0, 136.8, 134.6, 133.2, 131.1, 130.3, 128.8, 128.6, 128.4, 126.9, 126.6, 124.3, 118.4, 112.5, 67.1, 22.0, 10.4; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{26}H_{23}NO_2Na$: 404.1626; found: 404.1633.

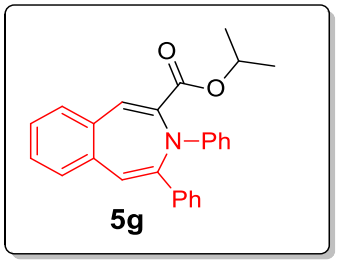
Spectral data of *tert*-butyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (5f):



5f

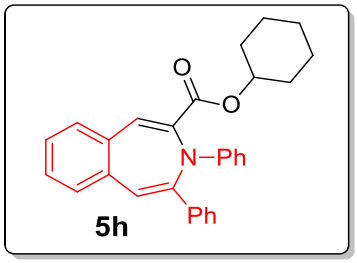
The compound **5f** was purified silica gel column using ethyl acetate/hexane:4/96 as the eluent; Yellow oil (51 mg, 0.12 mmol, 60%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.04 (d, *J* = 7.5 Hz, 2H), 7.89 (s, 1H), 7.38 ~ 7.26 (m, 7H), 7.22 ~ 7.20 (m, 1H), 6.98 (t, *J* = 9.0 Hz, 2H), 6.64 (t, *J* = 7.5 Hz, 1H), 6.53 (d, *J* = 8.0 Hz, 2H), 1.55 (s, 9H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.0, 145.7, 145.6, 137.6, 137.1, 136.7, 136.2, 133.5, 131.0, 130.2, 128.6, 128.5, 128.4, 128.3, 126.9, 126.7, 124.2, 118.2, 112.5, 82.0, 28.1; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{27}H_{25}NO_2Na$: 418.1783; found: 418.1783.

Spectral data of isopropyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (5g):



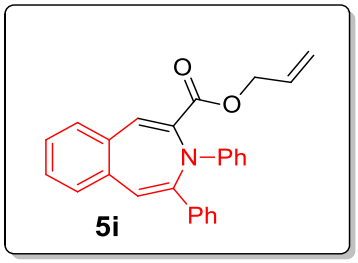
The compound **5g** was purified silica gel column using ethyl acetate/hexane:3/97 as the eluent; Yellow oil (58 mg, 0.15 mmol, 71%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.07 (d, *J* = 8.0 Hz, 2H), 8.00 (s, 1H), 7.43 ~ 7.30 (m, 7H), 7.26 ~ 7.25 (m, 1H), 7.00 (t, *J* = 8.0 Hz, 2H), 6.67 (t, *J* = 7.5 Hz, 1H), 6.55 (d, *J* = 8.5 Hz, 2H), 5.25 ~ 5.20 (m, 1H), 1.35 (d, *J* = 6.0 Hz, 6H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.3, 145.7, 145.6, 138.5, 137.0, 136.8, 135.0, 133.3, 131.1, 130.3, 128.8, 128.6, 128.46, 128.43, 126.9, 126.7, 124.2, 118.3, 112.5, 69.1, 21.8; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{26}H_{23}NO_2Na$: 404.1626; found: 404.1626.

Spectral data of cyclohexyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (5h):



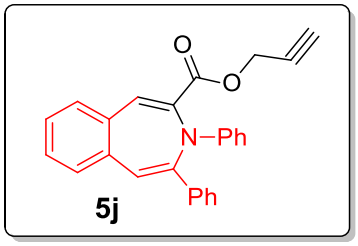
The compound **5h** was purified silica gel column using ethyl acetate/hexane:4/96 as the eluent; Yellow oil (64 mg, 0.15 mmol, 71%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.08 (d, *J* = 7.0 Hz, 2H), 8.01 (s, 1H), 7.43 ~ 7.30 (m, 7H), 7.26 ~ 7.24 (m, 1H), 7.00 (t, *J* = 7.7 Hz, 2H), 6.67 (t, *J* = 7.7 Hz, 1H), 6.57 (d, *J* = 8.4 Hz, 2H), 5.02 ~ 4.98 (m, 1H), 1.97 (s, 2H), 1.75 (s, 2H), 1.58 ~ 1.56 (m, 3H), 1.44 ~ 1.39 (m, 2H), 1.31 ~ 1.26 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.2, 145.7, 145.6, 138.5, 137.1, 136.8, 135.1, 133.3, 131.1, 130.3, 128.8, 128.6, 128.46, 128.42, 127.0, 126.7, 124.3, 118.3, 112.5, 74.1, 31.6, 25.3, 23.7; HRMS (ESI-TOF) m/z: $[M+H]^+$ $C_{29}H_{28}NO_2$: 422.2120; found: 422.2123.

Spectral data of allyl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (5i):



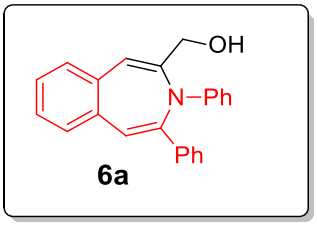
The compound **5i** was purified silica gel column using ethyl acetate/hexane:5/95 as the eluent; Yellow oil (55.0 g, 0.14 mmol, 68%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.06 ~ 8.05 (m, 3H), 7.42 ~ 7.30 (m, 7H), 7.26 ~ 7.24 (m, 1H), 7.00 (t, *J* = 7.7 Hz, 2H), 6.67 (t, *J* = 7.7 Hz, 1H), 6.57 (d, *J* = 8.4 Hz, 2H), 6.04 ~ 5.98 (m, 1H), 5.36 (dd, *J* = 1.4 Hz, 17.5 Hz, 1H), 5.27 (dd, *J* = 0.7 Hz, 10.5 Hz, 1H), 4.81 (d, *J* = 4.2 Hz, 2H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 164.3, 145.7, 145.6, 139.3, 136.96, 136.92, 134.2, 133.1, 131.9, 131.1, 130.3, 128.9, 128.7, 128.5, 127.0, 126.6, 124.2, 118.7, 118.4, 112.5, 66.1; HRMS (FD) m/z: $[M]^+$ $C_{26}H_{21}NO_2$: 379.1577; found: 379.1582.

Spectral data of prop-2-yn-1-yl 3,4-diphenyl-3*H*-benzo[d]azepine-2-carboxylate (5j):



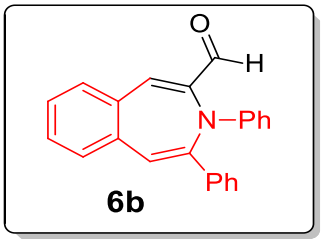
The compound **5j** was purified silica gel column using ethyl acetate/hexane:4/96 as the eluent; Yellow oil (52 mg, 0.13 mmol, 65%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.06 ~ 8.05 (m, 3H), 7.42 ~ 7.36 (m, 4H), 7.34 ~ 7.30 (m, 3H), 7.27 ~ 7.23 (m, 1H), 7.00 (t, *J* = 8.5 Hz, 2H), 6.67 (t, *J* = 7.5 Hz, 1H), 6.56 (d, *J* = 8.0 Hz, 2H), 4.88 (s, 2H), 2.53 (s, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 163.8, 145.8, 145.5, 140.1, 137.0, 136.8, 133.4, 133.0, 131.2, 130.3, 129.1, 128.7, 128.5, 127.0, 126.6, 124.2, 118.6, 112.5, 77.4, 75.2, 52.8; HRMS (FD) m/z: $[M]^+$ $C_{26}H_{19}NO_2$: 377.1421; found: 377.1420.

Spectral data of (3,4-diphenyl-3*H*-benzo[d]azepin-2-yl)methanol (6a):



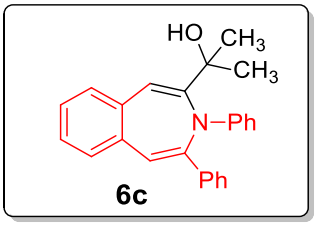
The compound **6a** was purified silica gel column using ethyl acetate/hexane:20/80 as the eluent; Pale yellow oil (49 mg, 0.15 mmol, 69 %); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.75 (d, *J* = 7.5 Hz, 2H), 7.46 (s, 1H), 7.40 ~ 7.31 (m, 5H), 7.20 ~ 7.18 (m, 2H), 7.05 (s, 1H), 6.97 (t, *J* = 8.5 Hz, 2H), 6.65 ~ 6.60 (m, 3H), 4.46 (s, 2H), 1.91 (s, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 146.4, 145.6, 143.2, 137.2, 135.1, 134.7, 130.2, 129.7, 128.9, 128.6, 128.3, 126.8, 126.7, 126.1, 125.8, 125.2, 118.1, 112.2, 63.5; HRMS (FD) m/z: $[M]^+$ $C_{24}H_{19}NO_3$: 325.1472; found: 325.1473.

Spectral data of 3,4-diphenyl-3*H*-benzo[d]azepine-2-carbaldehyde (6b):



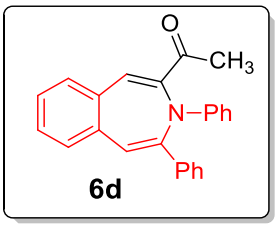
The compound **6b** was purified silica gel column using ethyl acetate/hexane:9/91 as the eluent; Pale Yellow oil (32 mg, 0.10 mmol, 73%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 9.78 (s, 1H), 8.01 (d, *J* = 8.4 Hz, 2H), 7.75 (s, 1H), 7.44 ~ 7.26 (m, 7H), 7.21 (s, 1H), 6.99 (t, *J* = 8.4 Hz, 2H), 6.68 (t, *J* = 7.0 Hz, 1H), 6.52 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 188.8, 147.4, 146.1, 144.7, 144.2, 137.3, 137.0, 132.9, 131.2, 130.5, 129.9, 128.9, 128.8, 128.6, 127.2, 126.1, 124.0, 118.8, 112.8; HRMS (FD) m/z: $[M]^+$ $C_{23}H_{17}NO$: 323.1315; found: 323.1320.

Spectral data of 2-(3,4-diphenyl-3*H*-benzo[d]azepin-2-yl)propan-2-ol (6c):



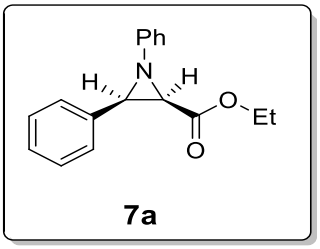
The compound **6c** was purified silica gel column using ethyl acetate/hexane:12/88 as the eluent; Yellow oil (36 mg, 0.10 mmol, 62 %); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.75 (d, *J* = 8.4 Hz, 2H), 7.49 (s, 1H), 7.41 (t, *J* = 7.0 Hz, 2H), 7.36 ~ 7.35 (m, 3H), 7.17 ~ 7.14 (m, 2H), 7.10 (s, 1H), 6.93 (t, *J* = 7.7 Hz, 2H), 6.78 (d, *J* = 9.1 Hz, 2H), 6.60 (t, *J* = 7.0 Hz, 1H), 2.42 (s, 1H), 1.55 (s, 3H), 1.33 (s, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 152.4, 148.5, 145.1, 138.0, 134.7, 134.4, 130.2, 129.9, 128.9, 128.5, 128.1, 126.7, 126.6, 126.55, 126.50, 124.7, 118.7, 113.5, 73.6, 30.3, 29.1; HRMS (FD) m/z: $[M]^+$ $C_{25}H_{23}NO$: 353.1785; found: 353.1792.

Spectral data of 1-(3,4-diphenyl-3*H*-benzo[d]azepin-2-yl)ethanone (6d):



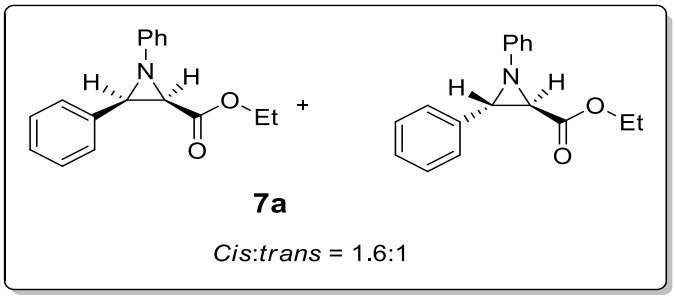
The compound **6d** was purified silica gel column using ethyl acetate/hexane:10/90 as the eluent; Yellow oil (35.8 mg, 0.10 mmol, 65 %); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.95 ~ 7.93 (m, 3H), 7.45 ~ 7.37 (m, 5H), 7.33 (q, *J* = 7.0 Hz, 2H), 7.25 (t, *J* = 7.7 Hz, 1H), 6.98 (t, *J* = 7.7 Hz, 2H), 6.65 (t, *J* = 7.7 Hz, 1H), 6.52 (d, *J* = 7.0 Hz, 2H), 2.49 (s, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 196.4, 146.2, 145.0, 142.3, 138.4, 136.88, 136.80, 133.0, 131.4, 130.5, 129.1, 128.76, 128.70, 128.5, 127.1, 126.8, 125.2, 118.5, 112.4, 27.0; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{24}H_{19}NONa$: 360.1364; found: 360.1364.

Spectral data of (2R,3R)-ethyl 1,3-diphenylaziridine-2-carboxylate (7a):



The compound **7a** was purified silica gel column using ethyl acetate/hexane:3/97 as the eluent; White solid (61.3 mg, 0.22 mmol, 52 %); mp = 63-65°C; ^{1}H NMR for *Cis* isomer (400 MHz, $CDCl_3$): 7.50 (d, *J* = 8.4 Hz, 2H), 7.35 ~ 7.24 (m, 5H), 7.06 ~ 7.01 (m, 3H), 4.06 ~ 3.94 (m, 2H), 3.58 (d, *J* = 6.8 Hz, 1H), 3.19 (d, *J* = 6.8 Hz, 1H), 0.97 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.6, 152.3, 134.6, 129.1, 128.0, 127.8, 127.6, 123.4, 119.9, 60.9, 47.1, 45.5, 13.8; HRMS (ESI-TOF) m/z: $[M+Na]^+$ $C_{17}H_{17}NO_2Na$: 290.1157.; found: 290.1154.

Spectral data of ethyl 1,3-diphenylaziridine-2-carboxylate (7a):[s4]



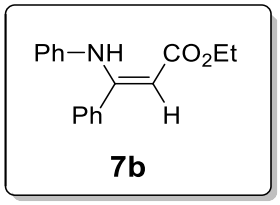
7a

Cis:*trans* = 1.6:1

This obtained crude ^{1H} NMR data consistent with reported literature data **S5.**

Crude ^{1}H NMR for *trans*- aziridine (400 MHz, $CDCl_3$): 7.27 ~ 7.17 (m, 2H), 7.02 ~ 6.85 (m, 3H), 4.12 (q, *J* = 7.2 Hz, 2H), 3.81(d, *J* = 2.4 Hz, 1H), 3.23 (d, *J* = 2.4 Hz, 1H), 1.16 (t, *J* = 7.2 Hz, 3H); rest of peak merged with aromatic regions.

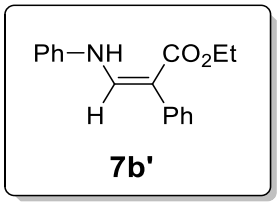
Spectral data of (Z)-ethyl 3-phenyl-3-(phenylamino)acrylate (7b):



7b

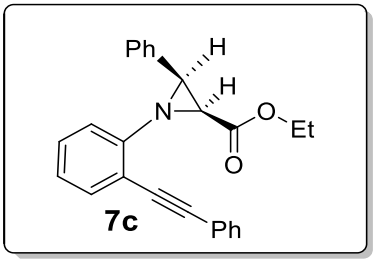
The compound **7b** was purified silica gel column using ethyl acetate/hexane:3/97 as the eluent; Pale yellow oil (14.0 mg, 0.05 mmol, 16%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 10.27 (s, 1H), 7.33 ~ 7.30 (m, 3H), 7.27 ~ 7.25 (m, 2H), 7.05 (t, *J* = 7.7 Hz, 2H), 6.88 (t, *J* = 7.0 Hz, 1H), 6.64 (d, *J* = 8.4 Hz, 2H), 4.97 (s, 1H), 4.19 (q, *J* = 7.0 Hz, 2H), 1.30 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 170.1, 159.0, 140.3, 135.9, 129.4, 128.5, 128.4, 128.2, 122.9, 122.1, 91.1, 59.2, 14.5; HRMS (ESI-TOF) m/z: $[M+H]^+$ $C_{17}H_{18}NO_2$: 268.1337.; found: 268.1338.

Spectral data of (Z)-ethyl 2-phenyl-3-(phenylamino)acrylate (7b'):



The compound **7b'** was purified silica gel column using ethyl acetate/hexane:3/97 as the eluent; Pale yellow oil (14.0 mg, 0.05 mmol, 20%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.33 (br d, 12.4Hz, 1H), 7.41 (t, *J* = 12.8 Hz, 1H), 7.53 ~ 7.24 (m, 7H), 7.01 ~ 6.99 (m, 3H), 4.26 (q, *J* = 6.8 Hz, 2H), 1.29 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.2, 143.6, 140.7, 137.9, 129.6, 129.5, 127.9, 126.0, 122.6, 115.5, 102.9, 59.8, 14.3; HRMS (ESI-TOF) m/z: $[M]^+$ $C_{17}H_{17}NO_2$: 267.1259; found: 267.1207

Spectral data of (2S,3S)-ethyl 3-phenyl-1-(2-(phenylethynyl)phenyl)aziridine-2-carboxylate (7c):



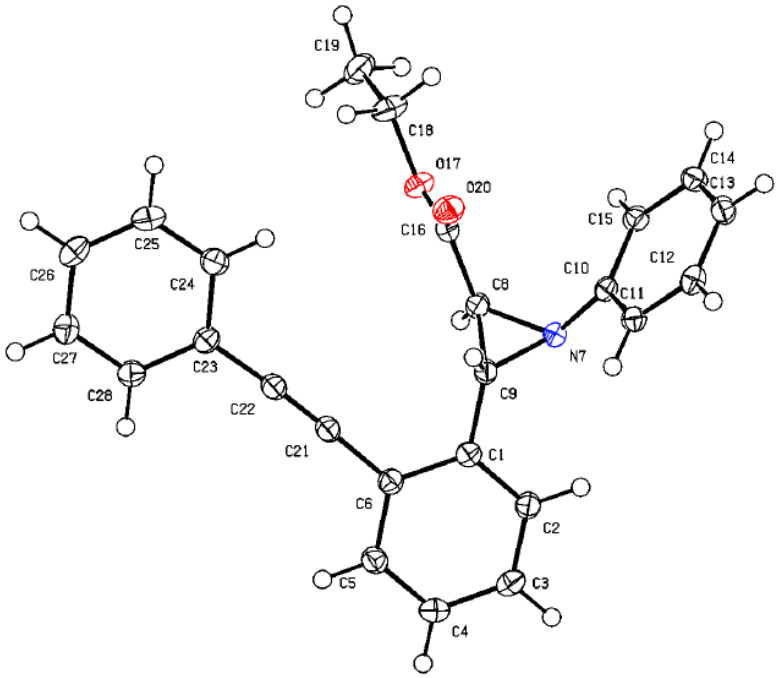
The compound **7c** was purified silica gel column using ethyl acetate/hexane:3/97 as the eluent; Brown oil (42.3 mg, 0.11 mmol, 54%); ^{1}H NMR (400 MHz, CDCl3): δ 7.55 (d, *J* = 7.6, 2H), 7.47 (d, *J* = 7.6, 2H), 7.32 ~ 7.15 (m, 7H), 7.08 ~ 7.06 (m, 2H), 7.04 ~ 7.00 (m, 2H), 3.89 ~ 3.84 (m, 2H), 3.77 (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, CDCl3): δ 167.5, 153.2, 134.6, 133.2, 131.3, 129.1, 128.05, 128.0, 127.9, 127.8, 127.7, 123.1, 123.0, 119.9, 115.6, 95.6, 86.06, 60.9, 47.9, 46.7, 13.7; HRMS (EI-MS) m/z: [M+Na]+ calcd. for $C_{25}H_{21}NO_2Na$: 390.1470; found: 390.1466.

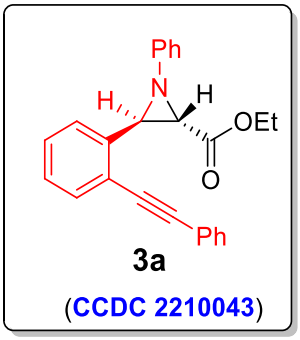
(7) X-ray crystallographic data of compound:

7.1 X-ray crystallographic structure and data of compound (3a):

Experimental: The sample was dissolved in appropriate amount of ethyl acetate 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.

Crystal measurements:





220613lt_auto

Table S2: Crystal data and structure refinement for 220613lt_auto.

Identification code	220613lt_auto
Empirical formula	$C_{25}H_{21}NO_2$
Formula weight	367.43
Temperature/K	100.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	9.03855(12)
b/Å	10.22281(12)
c/Å	10.74746(12)
α/°	90.6728(9)
β/°	100.9741(10)
γ/°	93.6105(10)
Volume/Å^3	972.68(2)

Z	2
ρ_{calc}g/cm^3	1.255
μ/mm^{-1}	0.626
F(000)	388.0
Crystal size/mm^3	0.21 × 0.2 × 0.16
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	8.67 to 149.736
Index ranges	-10 ≤ h ≤ 11, -12 ≤ k ≤ 10, -12 ≤ l ≤ 13
Reflections collected	10355
Independent reflections	3748 [R_{int} = 0.0129, R_{sigma} = 0.0142]
Data/restraints/parameters	3748/0/255
Goodness-of-fit on F^2	1.030
Final R indexes [I>=2σ (I)]	R_1 = 0.0328, wR_2 = 0.0831
Final R indexes [all data]	R_1 = 0.0342, wR_2 = 0.0841
Largest diff. peak/hole / e Å^{-3}	0.22/-0.16

Table S3: Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 220613lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C1	4145.2(11)	6553.1(9)	2727.1(9)	18.4(2)
C2	2714.0(11)	5976.1(10)	2732.6(10)	20.9(2)
C3	1850.0(12)	5397.1(10)	1635.0(10)	23.3(2)
C4	2399.2(12)	5404.5(10)	514.4(10)	23.0(2)
C5	3820.8(12)	5979.6(10)	490.0(10)	21.6(2)
C6	4717.8(11)	6546.6(10)	1595.8(10)	19.2(2)
C8	6264.4(11)	6489.6(10)	4742.2(9)	19.1(2)
C9	5082.3(11)	7207.0(10)	3883.7(9)	18.3(2)
C10	4734.5(11)	7682.0(9)	6093.7(9)	17.9(2)
C11	3796.5(11)	8716.7(10)	5854.3(10)	19.9(2)
C12	3535.7(12)	9484.8(10)	6855.3(10)	22.2(2)
C13	4229.7(12)	9248.8(10)	8091.5(10)	23.4(2)
C14	5195.7(12)	8236.4(11)	8322.1(10)	23.1(2)
C15	5442.2(11)	7445.9(10)	7332.1(10)	20.7(2)
C16	7673.7(11)	7283.8(10)	5327.0(9)	19.8(2)
C18	10227.7(12)	7155.5(12)	6318.8(12)	32.0(3)
C19	11331.5(12)	6125.3(13)	6659.1(12)	32.8(3)
C21	6216.3(11)	7096.2(10)	1576.0(9)	20.3(2)
C22	7469.2(12)	7538.6(10)	1524.1(9)	21.0(2)

Table S3: Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 220613lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C23	8941.6(11)	8065.5(10)	1388.0(10)	20.3(2)
C24	9924.4(12)	8728.7(10)	2395.6(10)	24.1(2)
C25	11336.2(12)	9233.2(11)	2235.3(11)	26.2(2)
C26	11788.7(12)	9067.8(11)	1084.7(11)	26.3(2)
C27	10821.4(12)	8420.0(11)	80.9(11)	25.2(2)
C28	9400.8(12)	7925.0(10)	223.4(10)	22.2(2)
N7	4818.3(9)	6773.2(8)	5105.6(8)	17.97(19)
O17	8795.3(8)	6497.9(7)	5709.9(7)	23.66(18)
O20	7795.2(8)	8462.4(7)	5442.8(7)	25.87(19)

Table S4: Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 220613lt_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	18.4(5)	17.2(5)	19.4(5)	1.4(4)	3.1(4)	2.6(4)
C2	19.7(5)	22.8(5)	20.9(5)	2.1(4)	5.2(4)	2.0(4)
C3	17.4(5)	24.1(5)	27.2(5)	1.9(4)	2.0(4)	-1.2(4)
C4	21.6(5)	23.2(5)	21.8(5)	-1.5(4)	-1.7(4)	1.1(4)
C5	22.6(5)	23.2(5)	19.3(5)	-0.3(4)	4.2(4)	3.8(4)
C6	18.6(5)	19.3(5)	19.9(5)	0.6(4)	3.9(4)	3.1(4)
C8	17.1(5)	20.0(5)	20.3(5)	-1.4(4)	4.6(4)	0.2(4)
C9	18.2(5)	19.3(5)	18.0(5)	0.5(4)	5.6(4)	0.4(4)
C10	16.1(5)	18.8(5)	18.9(5)	0.4(4)	5.0(4)	-3.2(4)
C11	18.8(5)	21.7(5)	18.9(5)	2.8(4)	3.5(4)	-0.6(4)
C12	20.3(5)	20.3(5)	27.0(5)	0.8(4)	7.7(4)	0.3(4)
C13	25.1(5)	24.3(5)	21.7(5)	-4.1(4)	8.7(4)	-4.2(4)
C14	22.5(5)	28.3(5)	17.3(5)	1.4(4)	2.4(4)	-4.1(4)
C15	18.5(5)	22.2(5)	21.3(5)	2.9(4)	3.4(4)	0.2(4)
C16	19.7(5)	23.2(5)	17.0(5)	-0.8(4)	5.1(4)	0.3(4)
C18	19.0(5)	34.9(6)	37.8(7)	-3.9(5)	-3.8(5)	-3.7(5)
C19	17.8(5)	43.6(7)	36.4(6)	9.5(5)	4.2(5)	0.3(5)
C21	21.6(5)	22.7(5)	16.9(5)	-0.7(4)	4.1(4)	2.4(4)
C22	22.3(5)	23.4(5)	17.4(5)	-0.5(4)	4.0(4)	2.8(4)
C23	18.5(5)	19.7(5)	22.8(5)	1.5(4)	3.5(4)	2.1(4)
C24	24.2(5)	26.2(5)	21.6(5)	-1.0(4)	2.7(4)	3.6(4)
C25	22.3(5)	23.7(5)	29.3(6)	-2.1(4)	-2.9(4)	-0.4(4)
C26	19.1(5)	23.0(5)	36.2(6)	3.3(4)	4.8(4)	-1.2(4)

Table S4: Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 220613lt_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C27	24.5(5)	25.9(5)	26.8(6)	1.6(4)	9.2(4)	0.0(4)
C28	21.7(5)	21.6(5)	22.5(5)	-1.7(4)	3.0(4)	-1.1(4)
N7	16.2(4)	20.4(4)	17.4(4)	0.5(3)	3.6(3)	0.8(3)
O17	16.1(4)	25.2(4)	28.0(4)	-0.9(3)	0.9(3)	-0.4(3)
O20	23.8(4)	22.2(4)	29.9(4)	-3.3(3)	2.5(3)	-2.2(3)

Table S5: Bond Lengths for 220613lt_auto.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	C2	1.3886(14)	C12	C13	1.3878(15)
C1	C6	1.4093(14)	C13	C14	1.3902(15)
C1	C9	1.4893(14)	C14	C15	1.3883(15)
C2	C3	1.3879(15)	C16	O17	1.3394(12)
C3	C4	1.3872(15)	C16	O20	1.2058(13)
C4	C5	1.3840(15)	C18	C19	1.4969(17)
C5	C6	1.4013(14)	C18	O17	1.4539(13)
C6	C21	1.4374(14)	C21	C22	1.2043(15)
C8	C9	1.5067(14)	C22	C23	1.4382(14)
C8	C16	1.4932(14)	C23	C24	1.4008(15)
C8	N7	1.4780(12)	C23	C28	1.4002(15)
C9	N7	1.4483(12)	C24	C25	1.3881(15)
C10	C11	1.3928(14)	C25	C26	1.3862(16)
C10	C15	1.3935(14)	C26	C27	1.3849(16)
C10	N7	1.4187(13)	C27	C28	1.3868(15)
C11	C12	1.3891(14)			

Table S6: Bond Angles for 220613lt_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	C1	C6	119.46(9)	C12	C13	C14	119.40(10)
C2	C1	C9	121.58(9)	C15	C14	C13	120.58(10)
C6	C1	C9	118.95(9)	C14	C15	C10	119.75(10)
C3	C2	C1	120.47(10)	O17	C16	C8	110.22(8)
C4	C3	C2	120.31(10)	O20	C16	C8	125.31(9)
C5	C4	C3	120.01(10)	O20	C16	O17	124.47(9)
C4	C5	C6	120.34(10)	O17	C18	C19	107.59(9)
C1	C6	C21	120.53(9)	C22	C21	C6	178.02(11)

Table S6: Bond Angles for 220613lt_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C5	C6	C1	119.39(9)	C21	C22	C23	176.87(11)
C5	C6	C21	120.08(9)	C24	C23	C22	121.30(9)
C16	C8	C9	116.24(8)	C28	C23	C22	119.53(9)
N7	C8	C9	58.05(6)	C28	C23	C24	119.17(10)
N7	C8	C16	119.44(8)	C25	C24	C23	120.07(10)
C1	C9	C8	121.06(8)	C26	C25	C24	120.24(10)
N7	C9	C1	117.88(8)	C27	C26	C25	120.10(10)
N7	C9	C8	59.98(6)	C26	C27	C28	120.25(10)
C11	C10	C15	119.87(9)	C27	C28	C23	120.16(10)
C11	C10	N7	119.92(9)	C9	N7	C8	61.97(6)
C15	C10	N7	119.73(9)	C10	N7	C8	122.38(8)
C12	C11	C10	119.83(9)	C10	N7	C9	121.41(8)
C13	C12	C11	120.53(10)	C16	O17	C18	115.54(8)

Table S7: Torsion Angles for 220613lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C1	C2	C3	C4	-0.90(16)	C11	C12	C13	C14	0.20(15)
C1	C9	N7	C8	111.62(10)	C12	C13	C14	C15	-1.46(15)
C1	C9	N7	C10	-135.69(9)	C13	C14	C15	C10	1.04(15)
C2	C1	C6	C5	1.20(14)	C15	C10	C11	C12	-1.89(14)
C2	C1	C6	C21	-177.80(9)	C15	C10	N7	C8	-63.05(12)
C2	C1	C9	C8	96.26(12)	C15	C10	N7	C9	-137.69(10)
C2	C1	C9	N7	26.27(14)	C16	C8	C9	C1	143.82(9)
C2	C3	C4	C5	0.64(16)	C16	C8	C9	N7	-109.78(9)
C3	C4	C5	C6	0.55(15)	C16	C8	N7	C9	104.27(10)
C4	C5	C6	C1	-1.47(15)	C16	C8	N7	C10	-6.92(14)
C4	C5	C6	C21	177.54(9)	C19	C18	O17	C16	-179.26(9)
C6	C1	C2	C3	-0.03(15)	C22	C23	C24	C25	-179.66(10)
C6	C1	C9	C8	-85.08(12)	C22	C23	C28	C27	-179.55(9)
C6	C1	C9	N7	-155.07(9)	C23	C24	C25	C26	-0.90(16)
C8	C9	N7	C10	112.69(10)	C24	C23	C28	C27	1.02(15)
C8	C16	O17	C18	-178.95(9)	C24	C25	C26	C27	1.27(16)
C9	C1	C2	C3	178.63(9)	C25	C26	C27	C28	-0.48(16)
C9	C1	C6	C5	-177.49(9)	C26	C27	C28	C23	-0.66(16)
C9	C1	C6	C21	3.51(14)	C28	C23	C24	C25	-0.23(15)
C9	C8	C16	O17	-159.90(8)	N7	C8	C9	C1	-106.40(10)
C9	C8	C16	O20	20.15(15)	N7	C8	C16	O17	133.63(9)
C9	C8	N7	C10	-111.19(10)	N7	C8	C16	O20	-46.32(14)

Table S7: Torsion Angles for 220613lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C10	C11	C12	C13	1.47(15)	N7	C10	C11	C12	170.16(9)
C11	C10	C15	C14	0.64(15)	N7	C10	C15	C14	-171.42(9)
C11	C10	N7	C8	124.89(10)	O20	C16	O17	C18	1.00(15)
C11	C10	N7	C9	50.25(13)					

Table S8: Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 220613lt_auto.

Atom	*x*	*y*	*z*	U(eq)
H2	2323.37	5977.75	3493	25
H3	878.01	4993.88	1651.25	28
H4	1799.4	5014.66	-236.38	28
H5	4190.47	5989.97	-280.52	26
H8	6380.83	5560.63	4496.53	23
H9	5315.8	8170.71	3822.87	22
H11	3336.35	8896.63	5008.61	24
H12	2877.05	10177.27	6692.37	27
H13	4046.15	9774.45	8773.96	28
H14	5691.6	8084.07	9164.52	28
H15	6090.89	6746.72	7498.34	25
H18A	10105.07	7642.2	7090.56	38
H18B	10593.1	7785.69	5734.53	38
H19A	11506.75	5698.87	5883.55	49
H19B	10923.06	5470.59	7184.6	49
H19C	12287.47	6532.14	7132.08	49
H24	9624.45	8833.33	3189.51	29
H25	11994.6	9693.55	2916.63	31
H26	12763.74	9399.37	984.53	32
H27	11132.28	8314.28	-708.32	30
H28	8737.55	7489.52	-470.8	27

Experimental

Single crystals of $C_{25}H_{21}NO_2$ **[220613lt_auto]** were **[]**. A suitable crystal was selected and **[]** on a **XtaLAB Synergy R, DW system, HyPix-Arc 150** diffractometer. The crystal was kept at 100.00(10) K during data collection. Using Olex2 [1], the structure was solved with the SHELXT [2] structure solution program using Intrinsic Phasing and refined with the SHELXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.

2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [220613lt_auto]

Crystal Data for $C_{25}H_{21}NO_2$ (M =367.43 g/mol): triclinic, space group P-1 (no. 2), a = 9.03855(12) Å, b = 10.22281(12) Å, c = 10.74746(12) Å, α = 90.6728(9)°, β = 100.9741(10)°, γ = 93.6105(10)°, V = 972.68(2) Å^3, Z = 2, T = 100.00(10) K, μ(Cu Kα) = 0.626 mm^{-1}, *Dcalc* = 1.255 g/cm^3, 10355 reflections measured (8.67° ≤ 2Θ ≤ 149.736°), 3748 unique (R_{int} = 0.0129, R_{sigma} = 0.0142) which were used in all calculations. The final R_1 was 0.0328 (I > 2σ(I)) and wR_2 was 0.0841 (all data).

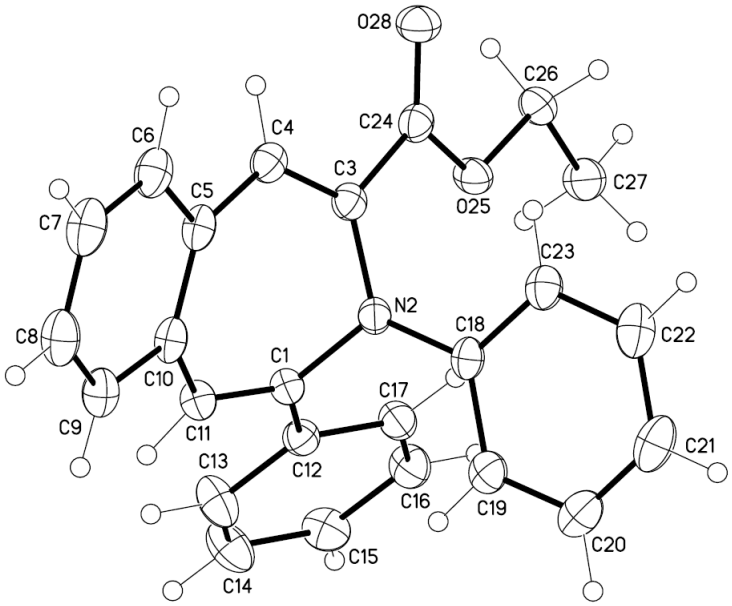
Refinement model description

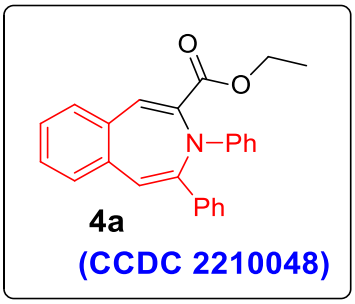
Number of restraints - 0, number of constraints - unknown.
Details:
1. Fixed Uiso
At 1.2 times of:
All C(H) groups, All C(H,H) groups
At 1.5 times of:
All C(H,H,H) groups
2.a Ternary CH refined with riding coordinates:
C8(H8), C9(H9)
2.b Secondary CH2 refined with riding coordinates:
C18(H18A,H18B)
2.c Aromatic/amide H refined with riding coordinates:
C2(H2), C3(H3), C4(H4), C5(H5), C11(H11), C12(H12), C13(H13), C14(H14), C15(H15), C24(H24), C25(H25), C26(H26), C27(H27), C28(H28)
2.d Idealised Me refined as rotating group:
C19(H19A,H19B,H19C)

7.2 X-ray crystallographic structure and data of compound (4a):

Experimental: The sample was dissolved in appropriate amount of ethyl acetate 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.

Crystal Measurement:





211143LT_auto

Table S9: Crystal data and structure refinement for 211143LT_auto.

Identification code	211143LT_auto
Empirical formula	$C_{25}H_{21}NO_2$
Formula weight	367.43
Temperature/K	100.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	9.6318(3)
b/Å	10.8319(4)
c/Å	11.3759(4)
α/°	92.501(3)
β/°	114.674(3)
γ/°	115.008(3)

Volume/Å^3	941.83(7)
Z	2
ρ_{calc}g/cm^3	1.296
μ/mm^{-1}	0.646
F(000)	388.0
Crystal size/mm^3	0.17 × 0.14 × 0.12
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	8.878 to 145.99
Index ranges	-11 ≤ h ≤ 11, -11 ≤ k ≤ 13, -13 ≤ l ≤ 12
Reflections collected	10151
Independent reflections	3521 [R_{int} = 0.0217, R_{sigma} = 0.0248]
Data/restraints/parameters	3521/0/255
Goodness-of-fit on F^2	1.055
Final R indexes [I>=2σ (I)]	R_1 = 0.0363, wR_2 = 0.0950
Final R indexes [all data]	R_1 = 0.0403, wR_2 = 0.0975
Largest diff. peak/hole / e Å^{-3}	0.55/-0.17

Table S10: Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 211143LT_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C1	5097.0(15)	6021.4(12)	2651.1(12)	20.2(3)
C3	4658.9(15)	6613.3(12)	4500.3(12)	20.2(3)
C4	6210.0(16)	7032.7(12)	5579.2(12)	22.3(3)
C5	7914.2(16)	7715.5(12)	5627.9(12)	23.2(3)
C6	9375.2(17)	8582.8(13)	6876.1(13)	28.6(3)
C7	11041.5(17)	9279.4(14)	7025.4(14)	32.4(3)
C8	11304.0(17)	9090.9(14)	5933.9(15)	31.6(3)
C9	9897.1(16)	8198.3(13)	4706.8(14)	27.4(3)
C10	8180.4(16)	7503.1(12)	4517.3(12)	23.2(3)
C11	6810.2(16)	6451.3(13)	3235.4(12)	22.6(3)
C12	3742.2(16)	4791.6(12)	1460.7(12)	21.2(3)
C13	4186.5(17)	3944.4(14)	887.8(14)	31.7(3)
C14	2913.7(18)	2783.9(15)	-206.5(15)	34.5(3)
C15	1169.2(17)	2429.7(14)	-753.3(13)	28.9(3)
C16	710.1(16)	3256.4(14)	-198.0(13)	27.2(3)
C17	1982.4(16)	4426.1(13)	899.2(12)	23.5(3)
C18	4178.6(14)	7821.4(12)	2725.4(12)	20.3(3)
C19	4169.7(15)	8054.1(13)	1521.0(12)	23.8(3)

Table S10: Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 211143LT_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C20	3874.4(16)	9127.0(14)	1044.3(14)	29.9(3)
C21	3579.4(18)	9988.9(14)	1742.2(14)	34.0(3)
C22	3564.3(17)	9748.2(14)	2924.2(14)	30.6(3)
C23	3853.9(16)	8682.4(13)	3417.5(12)	24.3(3)
C24	3032.4(16)	5976.2(12)	4633.6(12)	21.6(3)
C26	-55.7(16)	4607.3(13)	3453.1(12)	24.4(3)
C27	-1464.8(16)	3801.8(14)	2034.1(13)	29.0(3)
N2	4495.4(13)	6762.3(10)	3224.9(9)	19.4(2)
O25	1598.9(11)	5311.5(9)	3432.9(8)	23.1(2)
O28	3025.8(12)	6060.0(10)	5695.3(9)	28.3(2)

Table S11: Anisotropic Displacement Parameters (Å^2×10^3) for 211143LT_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	20.7(6)	21.5(6)	21.4(6)	6.4(5)	10.9(5)	11.8(5)
C3	21.8(6)	18.6(6)	20.1(6)	3.8(5)	9.2(5)	10.8(5)
C4	24.8(6)	19.2(6)	21.2(6)	4.2(5)	8.9(5)	11.7(5)
C5	20.8(6)	18.7(6)	25.2(6)	4.6(5)	5.6(5)	10.9(5)
C6	26.5(7)	23.8(6)	27.2(7)	2.7(5)	5.2(6)	13.5(5)
C7	21.8(6)	22.0(6)	34.0(7)	0.0(5)	0.5(6)	8.4(5)
C8	18.7(6)	23.3(6)	42.8(8)	7.3(6)	8.6(6)	8.2(5)
C9	21.3(6)	23.6(6)	34.6(7)	8.6(5)	10.8(6)	11.3(5)
C10	20.1(6)	18.8(6)	27.4(6)	6.4(5)	7.2(5)	11.0(5)
C11	22.1(6)	22.6(6)	24.6(6)	5.5(5)	11.2(5)	12.1(5)
C12	20.6(6)	21.8(6)	21.0(6)	5.9(5)	10.2(5)	10.0(5)
C13	21.5(6)	30.2(7)	38.0(8)	-2.3(6)	12.4(6)	11.2(6)
C14	31.1(7)	30.5(7)	39.8(8)	-3.1(6)	18.6(7)	12.9(6)
C15	25.4(7)	26.0(6)	25.2(7)	-0.6(5)	10.2(6)	6.8(5)
C16	19.8(6)	29.3(7)	25.2(7)	2.8(5)	7.7(5)	9.6(5)
C17	21.9(6)	26.3(6)	22.6(6)	4.2(5)	10.4(5)	12.3(5)
C18	12.3(5)	18.6(6)	22.5(6)	3.1(5)	4.1(5)	5.9(4)
C19	14.6(5)	24.8(6)	24.8(6)	5.2(5)	6.8(5)	6.6(5)
C20	20.7(6)	28.4(7)	29.9(7)	12.2(6)	8.1(5)	6.8(5)
C21	29.4(7)	23.3(6)	37.4(8)	10.3(6)	6.5(6)	12.4(6)
C22	25.8(7)	22.6(6)	32.0(7)	-0.4(5)	4.2(6)	12.8(5)
C23	20.7(6)	22.4(6)	23.2(6)	2.1(5)	5.6(5)	10.2(5)

Table S11: Anisotropic Displacement Parameters ($Å^2×10^3$) for 211143LT_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C24	23.4(6)	21.1(6)	21.2(6)	5.6(5)	9.4(5)	12.8(5)
C26	20.7(6)	29.9(6)	25.9(6)	8.9(5)	13.5(5)	12.7(5)
C27	20.0(6)	36.4(7)	26.3(7)	7.9(6)	9.9(5)	11.5(6)
N2	18.9(5)	20.5(5)	19.2(5)	4.6(4)	8.1(4)	11.2(4)
O25	17.9(4)	28.2(5)	20.4(4)	4.2(3)	9.1(4)	9.3(4)
O28	29.0(5)	35.3(5)	21.0(5)	6.0(4)	12.7(4)	15.3(4)

Table S12: Bond Lengths for 211143LT_auto.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	C11	1.3376(17)	C13	C14	1.3853(19)
C1	C12	1.4862(16)	C14	C15	1.3853(19)
C1	N2	1.4384(15)	C15	C16	1.3838(18)
C3	C4	1.3384(17)	C16	C17	1.3918(17)
C3	C24	1.4990(16)	C18	C19	1.4010(17)
C3	N2	1.4167(14)	C18	C23	1.4055(17)
C4	C5	1.4623(17)	C18	N2	1.3930(15)
C5	C6	1.4059(17)	C19	C20	1.3898(18)
C5	C10	1.4176(18)	C20	C21	1.389(2)
C6	C7	1.3825(19)	C21	C22	1.386(2)
C7	C8	1.390(2)	C22	C23	1.3869(17)
C8	C9	1.3838(19)	C24	O25	1.3378(14)
C9	C10	1.4082(17)	C24	O28	1.2100(14)
C10	C11	1.4640(17)	C26	C27	1.5019(17)
C12	C13	1.4009(17)	C26	O25	1.4583(14)
C12	C17	1.3944(17)			

Table S13: Bond Angles for 211143LT_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C11	C1	C12	124.52(11)	C14	C13	C12	120.86(12)
C11	C1	N2	118.56(11)	C15	C14	C13	120.65(12)
N2	C1	C12	116.90(10)	C16	C15	C14	119.16(12)
C4	C3	C24	119.54(11)	C15	C16	C17	120.51(12)
C4	C3	N2	122.14(11)	C16	C17	C12	120.88(11)
N2	C3	C24	118.32(10)	C19	C18	C23	118.29(11)
C3	C4	C5	126.27(11)	N2	C18	C19	121.00(11)

Table S13: Bond Angles for 211143LT_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C6	C5	C4	117.14(11)	N2	C18	C23	120.71(11)
C6	C5	C10	118.72(12)	C20	C19	C18	120.35(12)
C10	C5	C4	124.07(11)	C21	C20	C19	121.15(12)
C7	C6	C5	121.57(13)	C22	C21	C20	118.59(12)
C6	C7	C8	119.81(12)	C21	C22	C23	121.22(13)
C9	C8	C7	119.76(12)	C22	C23	C18	120.39(12)
C8	C9	C10	121.63(13)	O25	C24	C3	111.38(10)
C5	C10	C11	123.44(11)	O28	C24	C3	124.01(11)
C9	C10	C5	118.43(12)	O28	C24	O25	124.61(11)
C9	C10	C11	117.56(11)	O25	C26	C27	107.32(9)
C1	C11	C10	127.41(11)	C3	N2	C1	116.06(9)
C13	C12	C1	121.12(11)	C18	N2	C1	121.40(9)
C17	C12	C1	120.91(11)	C18	N2	C3	120.76(10)
C17	C12	C13	117.95(11)	C24	O25	C26	115.75(9)

Table S14: Torsion Angles for 211143LT_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C1	C12	C13	C14	178.79(12)	C12	C13	C14	C15	-0.4(2)
C1	C12	C17	C16	-178.62(10)	C13	C12	C17	C16	-0.12(18)
C3	C4	C5	C6	154.20(12)	C13	C14	C15	C16	0.4(2)
C3	C4	C5	C10	-28.78(19)	C14	C15	C16	C17	-0.25(19)
C3	C24	O25	C26	177.78(9)	C15	C16	C17	C12	0.10(19)
C4	C3	C24	O25	-164.79(10)	C17	C12	C13	C14	0.3(2)
C4	C3	C24	O28	15.41(18)	C18	C19	C20	C21	-0.11(19)
C4	C3	N2	C1	60.93(15)	C19	C18	C23	C22	-1.11(17)
C4	C3	N2	C18	-104.09(13)	C19	C18	N2	C1	10.07(17)
C4	C5	C6	C7	-179.73(11)	C19	C18	N2	C3	174.28(10)
C4	C5	C10	C9	-178.42(11)	C19	C20	C21	C22	-0.7(2)
C4	C5	C10	C11	-7.29(18)	C20	C21	C22	C23	0.7(2)
C5	C6	C7	C8	-2.13(19)	C21	C22	C23	C18	0.27(19)
C5	C10	C11	C1	33.13(19)	C23	C18	C19	C20	1.04(17)
C6	C5	C10	C9	-1.45(17)	C23	C18	N2	C1	-170.12(10)
C6	C5	C10	C11	169.68(11)	C23	C18	N2	C3	-5.91(16)
C6	C7	C8	C9	-0.49(19)	C24	C3	C4	C5	-177.70(10)
C7	C8	C9	C10	2.12(19)	C24	C3	N2	C1	-119.67(11)
C8	C9	C10	C5	-1.11(18)	C24	C3	N2	C18	75.31(14)
C8	C9	C10	C11	-172.77(11)	C27	C26	O25	C24	-173.88(10)
C9	C10	C11	C1	-155.67(12)	N2	C1	C11	C10	8.25(18)

Table S14: Torsion Angles for 211143LT_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C10	C5	C6	C7	3.09(18)	N2	C1	C12	C13	-175.55(11)
C11	C1	C12	C13	2.52(19)	N2	C1	C12	C17	2.90(16)
C11	C1	C12	C17	-179.02(11)	N2	C3	C4	C5	1.69(19)
C11	C1	N2	C3	-67.97(14)	N2	C3	C24	O25	15.80(14)
C11	C1	N2	C18	96.94(13)	N2	C3	C24	O28	-164.00(11)
C12	C1	C11	C10	-169.79(11)	N2	C18	C19	C20	-179.15(11)
C12	C1	N2	C3	110.22(11)	N2	C18	C23	C22	179.07(11)
C12	C1	N2	C18	-84.86(13)	O28	C24	O25	C26	-2.43(17)

Table S15: Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 211143LT_auto.

Atom	*x*	*y*	*z*	U(eq)
H4	6203.25	6871.38	6391.39	27
H6	9213.62	8692.68	7634.92	34
H7	12004.25	9885.2	7871.83	39
H8	12445.25	9573.05	6029.82	38
H9	10094.17	8050.84	3974.05	33
H11	7179.95	6025.49	2764.96	27
H13	5375.96	4168.68	1254.52	38
H14	3240.49	2225.45	-585.56	41
H15	299.02	1629.51	-1500.07	35
H16	-482.5	3023.84	-568.6	33
H17	1647.34	4982.79	1270.69	28
H19	4366.61	7475.4	1027.71	29
H20	3874.13	9273.68	226.92	36
H21	3392.11	10727.54	1416	41
H22	3351.56	10323.8	3405.55	37
H23	3832.69	8533.45	4227.98	29
H26A	-280.11	5313	3808.44	29
H26B	-22.96	3953.74	4033.34	29
H27A	-1561.83	4467.37	1489.18	44
H27B	-2581.26	3240.92	2022.16	44
H27C	-1170.68	3171.47	1665.09	44

Experimental

Single crystals of $C_{25}H_{21}NO_2$ **[211143LT_auto]** were **[]**. A suitable crystal was selected and **[]** on a **XtaLAB Synergy R, DW system, HyPix-Arc 150** diffractometer. The crystal was kept at 100.00(10) K during data collection. Using Olex2 [1], the structure was solved with the SHELXT

[2] structure solution program using Intrinsic Phasing and refined with the SHELXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [211143LT_auto]

Crystal Data for $C_{25}H_{21}NO_2$ (M =367.43 g/mol): triclinic, space group P-1 (no. 2), a = 9.6318(3) Å, b = 10.8319(4) Å, c = 11.3759(4) Å, α = 92.501(3)°, β = 114.674(3)°, γ = 115.008(3)°, V = 941.83(7) Å^3, Z = 2, T = 100.00(10) K, μ(Cu Kα) = 0.646 mm^{-1}, *Dcalc* = 1.296 g/cm^3, 10151 reflections measured (8.878° ≤ 2Θ ≤ 145.99°), 3521 unique (R_{int} = 0.0217, R_{sigma} = 0.0248) which were used in all calculations. The final R_1 was 0.0363 (I > 2σ(I)) and wR_2 was 0.0975 (all data).

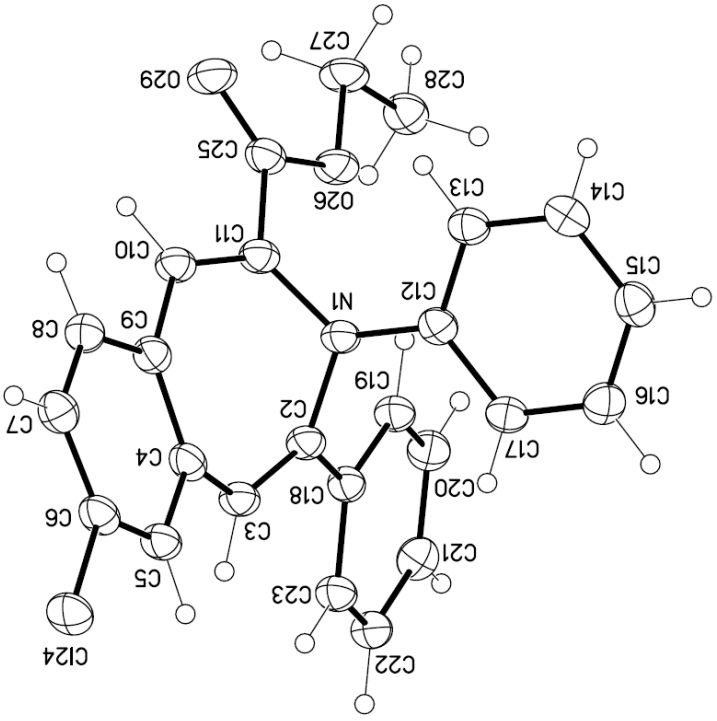
Refinement model description

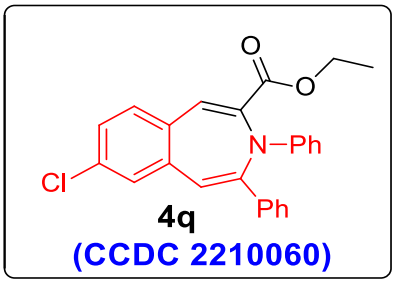
Number of restraints - 0, number of constraints - unknown.
Details:
1. Fixed Uiso
At 1.2 times of:
All C(H) groups, All C(H,H) groups
At 1.5 times of:
All C(H,H,H) groups
2.a Secondary CH2 refined with riding coordinates:
C26(H26A,H26B)
2.b Aromatic/amide H refined with riding coordinates:
C4(H4), C6(H6), C7(H7), C8(H8), C9(H9), C11(H11), C13(H13), C14(H14),
C15(H15), C16(H16), C17(H17), C19(H19), C20(H20), C21(H21), C22(H22), C23(H23)
2.c Idealised Me refined as rotating group:
C27(H27A,H27B,H27C)

7.3 X-ray crystallographic structure and data of compound (4q):

Experimental: The sample was dissolved in appropriate amount of ethyl acetate 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.

Crystal Measurement:





2205108lt_auto

Table S16: Crystal data and structure refinement for 2205108lt_auto.

Identification code	2205108lt_auto
Empirical formula	$C_{25}H_{20}ClNO_2$
Formula weight	401.87
Temperature/K	100.01(10)
Crystal system	monoclinic
Space group	C2/c
a/Å	11.3472(3)
b/Å	18.7364(4)
c/Å	18.9688(3)
α/°	90
β/°	103.100(2)
γ/°	90

Volume/Å^3	3927.94(14)
Z	8
ρ_{calc}g/cm^3	1.359
μ/mm^{-1}	1.890
F(000)	1680.0
Crystal size/mm^3	0.14 × 0.05 × 0.02
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	9.29 to 150.384
Index ranges	-14 ≤ h ≤ 14, -22 ≤ k ≤ 22, -22 ≤ l ≤ 12
Reflections collected	15232
Independent reflections	3835 [R_{int} = 0.0349, R_{sigma} = 0.0281]
Data/restraints/parameters	3835/0/263
Goodness-of-fit on F^2	1.104
Final R indexes [I>=2σ (I)]	R_1 = 0.0447, wR_2 = 0.1255
Final R indexes [all data]	R_1 = 0.0492, wR_2 = 0.1327
Largest diff. peak/hole / e Å^{-3}	0.45/-0.71

Table S17: Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 2205108lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C2	5236.3(14)	5999.3(9)	4172.7(8)	22.9(3)
C3	4316.1(14)	5591.3(9)	3818.2(8)	24.3(3)
C4	4386.9(14)	4870.9(9)	3535.2(8)	23.9(3)
C5	3453.7(15)	4666.1(9)	2942.4(8)	25.7(3)
C6	3368.5(15)	3966.7(10)	2708.8(8)	26.4(4)
C7	4148.3(15)	3441.4(9)	3059.1(9)	27.2(4)
C8	5065.9(15)	3639.2(9)	3639.3(9)	26.1(4)
C9	5232.0(14)	4349.2(9)	3869.8(8)	23.6(3)
C10	6243.9(14)	4495.3(9)	4479.8(8)	23.8(3)
C11	6796.1(14)	5122.6(9)	4656.3(8)	23.1(3)
C12	7095.9(14)	5963.1(8)	3724.4(8)	22.3(3)
C13	8239.3(15)	5673.9(9)	3724.7(9)	25.0(3)
C14	8874.2(15)	5899.2(9)	3217.8(9)	28.5(4)
C15	8390.2(16)	6407.5(9)	2701.0(9)	28.9(4)
C16	7255.1(16)	6693.8(9)	2698.9(9)	28.0(4)
C17	6612.7(15)	6475.9(9)	3201.8(8)	24.9(3)
C18	5064.4(14)	6700.3(9)	4498.3(8)	23.1(3)
C19	6023.7(15)	7022.0(9)	4995.0(8)	25.1(3)

Table S17: Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 2205108lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C20	5859.2(16)	7661.3(9)	5328.5(9)	27.7(4)
C21	4747.5(16)	8007.7(9)	5168.1(9)	27.7(4)
C22	3792.9(15)	7701.3(9)	4666.7(9)	27.5(4)
C23	3944.1(15)	7058.4(9)	4338.5(9)	25.4(3)
C25	7811.4(14)	5146.1(9)	5321.8(9)	24.9(3)
C27	9045.9(16)	5886.9(10)	6201.1(9)	30.3(4)
C28	9018.1(16)	6649.4(10)	6441.1(9)	29.7(4)
Cl24	2234.8(4)	3728.2(2)	1962.6(2)	31.04(16)
N1	6459.5(12)	5754.1(7)	4244.1(7)	22.5(3)
O26	8104.5(10)	5812.7(6)	5538.4(6)	27.5(3)
O29	8293.8(11)	4620.5(7)	5624.6(6)	32.3(3)

Table S18: Anisotropic Displacement Parameters (Å^2×10^3) for 2205108lt_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C2	20.1(7)	29.7(8)	17.5(7)	3.2(6)	1.1(6)	0.9(6)
C3	20.1(7)	29.8(9)	21.4(7)	2.3(6)	1.3(6)	1.6(6)
C4	19.2(7)	30.7(9)	20.6(7)	0.2(6)	2.2(6)	-4.4(6)
C5	20.5(7)	31.9(9)	22.9(8)	1.8(7)	1.3(6)	-0.8(6)
C6	21.3(8)	34.9(9)	21.3(8)	-1.3(7)	1.3(6)	-4.7(7)
C7	26.1(8)	28.8(9)	26.2(8)	-3.5(7)	4.8(7)	-4.1(7)
C8	24.0(8)	28.5(9)	25.1(8)	2.0(6)	3.9(7)	-1.2(6)
C9	21.2(7)	29.1(8)	20.2(7)	1.0(6)	4.0(6)	-1.5(6)
C10	21.0(8)	29.2(8)	19.9(7)	1.6(6)	1.9(6)	1.4(6)
C11	19.7(7)	28.2(8)	19.8(7)	0.6(6)	1.3(6)	1.2(6)
C12	21.6(8)	24.2(8)	19.2(7)	-3.3(6)	0.6(6)	-2.6(6)
C13	22.4(8)	26.6(8)	23.9(8)	-1.5(6)	1.2(6)	-0.3(6)
C14	22.5(8)	33.0(9)	29.8(9)	-5.1(7)	5.5(7)	-0.5(6)
C15	27.9(9)	33.8(9)	25.9(8)	-1.2(7)	7.9(7)	-4.7(7)
C16	29.7(9)	30.4(9)	22.1(8)	0.6(6)	2.0(6)	-1.9(7)
C17	21.3(8)	28.2(8)	22.9(8)	-1.3(6)	0.5(6)	0.0(6)
C18	20.4(7)	27.5(8)	20.3(7)	2.4(6)	2.5(6)	-0.8(6)
C19	21.3(8)	29.7(9)	22.1(8)	1.9(6)	0.3(6)	-0.5(6)
C20	27.0(8)	30.5(9)	23.0(8)	-0.7(6)	0.5(6)	-2.3(7)
C21	31.7(9)	26.8(9)	24.2(8)	-0.6(6)	5.8(7)	-0.3(7)
C22	23.9(8)	30.3(9)	27.5(8)	4.3(7)	4.5(6)	2.6(6)

Table S18: Anisotropic Displacement Parameters ($Å^2×10^3$) for 2205108lt_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C23	20.9(8)	29.9(9)	23.5(8)	1.4(6)	0.9(6)	0.2(6)
C25	19.5(7)	30.3(9)	23.4(8)	0.3(6)	1.9(6)	-0.7(6)
C27	23.3(8)	37.1(10)	24.1(8)	0.1(7)	-8.3(7)	0.9(7)
C28	27.0(8)	36.6(10)	23.3(8)	-0.8(7)	1.2(6)	-2.0(7)
Cl24	26.3(2)	37.2(3)	25.5(2)	-3.71(15)	-2.71(17)	-5.74(15)
N1	18.7(6)	26.0(7)	21.3(6)	2.0(5)	1.3(5)	1.6(5)
O26	23.1(6)	30.6(6)	23.6(6)	0.4(5)	-6.0(5)	-1.5(5)
O29	27.8(6)	31.8(7)	31.4(6)	2.8(5)	-5.7(5)	3.5(5)

Table S19: Bond Lengths for 2205108lt_auto.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C2	C3	1.345(2)	C12	C17	1.400(2)
C2	C18	1.483(2)	C12	N1	1.403(2)
C2	N1	1.439(2)	C13	C14	1.391(2)
C3	C4	1.461(2)	C14	C15	1.388(3)
C4	C5	1.412(2)	C15	C16	1.395(2)
C4	C9	1.415(2)	C16	C17	1.387(2)
C5	C6	1.380(2)	C18	C19	1.404(2)
C6	C7	1.388(2)	C18	C23	1.408(2)
C6	Cl24	1.7414(16)	C19	C20	1.387(2)
C7	C8	1.383(2)	C20	C21	1.390(2)
C8	C9	1.400(2)	C21	C22	1.393(2)
C9	C10	1.460(2)	C22	C23	1.384(2)
C10	C11	1.338(2)	C25	O26	1.333(2)
C11	C25	1.505(2)	C25	O29	1.207(2)
C11	N1	1.422(2)	C27	C28	1.502(2)
C12	C13	1.406(2)	C27	O26	1.4601(18)

Table S20: Bond Angles for 2205108lt_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C3	C2	C18	123.45(15)	N1	C12	C13	120.92(14)
C3	C2	N1	119.25(15)	C14	C13	C12	120.34(15)
N1	C2	C18	117.30(13)	C15	C14	C13	120.82(16)
C2	C3	C4	127.41(15)	C14	C15	C16	118.91(15)
C5	C4	C3	116.73(15)	C17	C16	C15	120.92(16)

Table S20: Bond Angles for 2205108lt_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C5	C4	C9	118.63(15)	C16	C17	C12	120.44(15)
C9	C4	C3	124.15(14)	C19	C18	C2	120.31(14)
C6	C5	C4	119.96(15)	C19	C18	C23	117.68(15)
C5	C6	C7	121.94(15)	C23	C18	C2	122.00(14)
C5	C6	Cl24	119.34(13)	C20	C19	C18	120.86(15)
C7	C6	Cl24	118.72(13)	C19	C20	C21	120.85(15)
C8	C7	C6	118.30(16)	C20	C21	C22	118.94(16)
C7	C8	C9	121.80(16)	C23	C22	C21	120.60(16)
C4	C9	C10	123.88(15)	C22	C23	C18	121.04(15)
C8	C9	C4	119.14(15)	O26	C25	C11	112.10(14)
C8	C9	C10	116.87(15)	O29	C25	C11	123.63(15)
C11	C10	C9	127.06(15)	O29	C25	O26	124.27(15)
C10	C11	C25	117.50(15)	O26	C27	C28	106.86(13)
C10	C11	N1	122.76(14)	C11	N1	C2	116.78(13)
N1	C11	C25	119.74(14)	C12	N1	C2	118.93(13)
C17	C12	C13	118.57(15)	C12	N1	C11	120.10(13)
C17	C12	N1	120.50(14)	C25	O26	C27	115.93(13)

Table S21: Torsion Angles for 2205108lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C2	C3	C4	C5	-153.82(16)	C13	C12	C17	C16	-0.3(2)
C2	C3	C4	C9	34.3(2)	C13	C12	N1	C2	-172.77(14)
C2	C18	C19	C20	-177.00(14)	C13	C12	N1	C11	-16.5(2)
C2	C18	C23	C22	177.86(14)	C13	C14	C15	C16	0.3(3)
C3	C2	C18	C19	165.53(15)	C14	C15	C16	C17	-0.2(3)
C3	C2	C18	C23	-13.0(2)	C15	C16	C17	C12	0.2(2)
C3	C2	N1	C11	-64.37(19)	C17	C12	C13	C14	0.4(2)
C3	C2	N1	C12	92.69(18)	C17	C12	N1	C2	8.5(2)
C3	C4	C5	C6	-171.28(15)	C17	C12	N1	C11	164.74(14)
C3	C4	C9	C8	167.05(15)	C18	C2	C3	C4	-174.26(14)
C3	C4	C9	C10	-9.0(2)	C18	C2	N1	C11	115.83(15)
C4	C5	C6	C7	2.8(2)	C18	C2	N1	C12	-87.11(17)
C4	C5	C6	Cl24	-178.40(12)	C18	C19	C20	C21	-1.4(2)
C4	C9	C10	C11	-25.3(3)	C19	C18	C23	C22	-0.7(2)
C5	C4	C9	C8	-4.7(2)	C19	C20	C21	C22	0.2(2)
C5	C4	C9	C10	179.23(14)	C20	C21	C22	C23	0.7(2)
C5	C6	C7	C8	-2.9(2)	C21	C22	C23	C18	-0.4(2)
C6	C7	C8	C9	-0.9(2)	C23	C18	C19	C20	1.6(2)

Table S21: Torsion Angles for 2205108lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C7	C8	C9	C4	4.7(2)	C25	C11	N1	C2	-119.77(16)
C7	C8	C9	C10	-178.94(14)	C25	C11	N1	C12	83.45(18)
C8	C9	C10	C11	158.58(16)	C28	C27	O26	C25	-166.47(14)
C9	C4	C5	C6	1.1(2)	Cl24	C6	C7	C8	178.31(12)
C9	C10	C11	C25	178.90(14)	N1	C2	C3	C4	5.9(2)
C9	C10	C11	N1	-1.0(3)	N1	C2	C18	C19	-14.7(2)
C10	C11	C25	O26	-165.61(14)	N1	C2	C18	C23	166.79(14)
C10	C11	C25	O29	13.9(2)	N1	C11	C25	O26	14.3(2)
C10	C11	N1	C2	60.1(2)	N1	C11	C25	O29	-166.23(15)
C10	C11	N1	C12	-96.66(19)	N1	C12	C13	C14	-178.35(15)
C11	C25	O26	C27	177.31(13)	N1	C12	C17	C16	178.51(15)
C12	C13	C14	C15	-0.5(2)	O29	C25	O26	C27	-2.2(2)

Table S22: Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 2205108lt_auto.

Atom	*x*	*y*	*z*	U(eq)
H3	3530.44	5794.51	3743.23	29
H5	2884.82	5009.59	2704.6	31
H7	4054.19	2958.32	2904.41	33
H8	5597.96	3283.41	3888.13	31
H10	6542.9	4102.52	4784.8	29
H13	8580.06	5322.82	4072.87	30
H14	9648.8	5702.54	3225.65	34
H15	8824.97	6557.89	2354.23	35
H16	6917.08	7043.14	2348.13	34
H17	5839.69	6675.82	3191.6	30
H19	6795.52	6798.92	5104.01	30
H20	6515.06	7865.05	5670.98	33
H21	4640.28	8446.38	5396.72	33
H22	3031.04	7935.71	4548.97	33
H23	3281.7	6855.62	4000.22	31
H27A	9847.71	5770.05	6107.77	36
H27B	8887.23	5560.62	6579.56	36
H28A	9198.55	6965.41	6067.52	45
H28B	9624.51	6718.91	6893.96	45
H28C	8213.27	6761.42	6518.42	45

Experimental

Single crystals of $C_{25}H_{20}ClNO_2$ **[2205108lt_auto]** were **[]**. A suitable crystal was selected and **[]** on a **XtaLAB Synergy R, DW system, HyPix-Arc 150** diffractometer. The crystal was kept at 100.01(10) K during data collection. Using Olex2 [1], the structure was solved with the SHELXT [2] structure solution program using Intrinsic Phasing and refined with the SHELXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [2205108lt_auto]

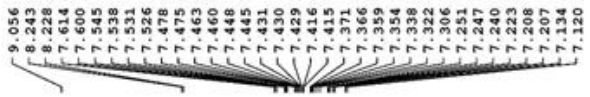
Crystal Data for $C_{25}H_{20}ClNO_2$ (M =401.87 g/mol): monoclinic, space group C2/c (no. 15), a = 11.3472(3) Å, b = 18.7364(4) Å, c = 18.9688(3) Å, β = 103.100(2)°, V = 3927.94(14) Å^3, Z = 8, T = 100.01(10) K, μ(Cu Kα) = 1.890 mm^{-1}, *Dcalc* = 1.359 g/cm^3, 15232 reflections measured (9.29° ≤ 2Θ ≤ 150.384°), 3835 unique (R_{int} = 0.0349, R_{sigma} = 0.0281) which were used in all calculations. The final R_1 was 0.0447 (I > 2σ(I)) and wR_2 was 0.1327 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.
Details:
1. Fixed Uiso
At 1.2 times of:
All C(H) groups, All C(H,H) groups
At 1.5 times of:
All C(H,H,H) groups
2.a Secondary CH2 refined with riding coordinates:
C27(H27A,H27B)
2.b Aromatic/amide H refined with riding coordinates:
C3(H3), C5(H5), C7(H7), C8(H8), C10(H10), C13(H13), C14(H14), C15(H15), C16(H16), C17(H17), C19(H19), C20(H20), C21(H21), C22(H22), C23(H23)
2.c Idealised Me refined as rotating group:
C28(H28A,H28B,H28C)

(8) ^{1}H and ^{13}C spectra key compounds:

Solvent: $CDCl_3$
SFO1: 500 MHz



DT-03-108-3CL Imine-HPL

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PROCNO                   1

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PC                    1.00
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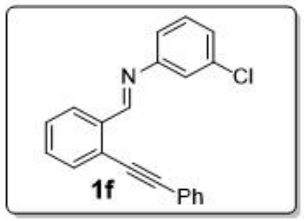

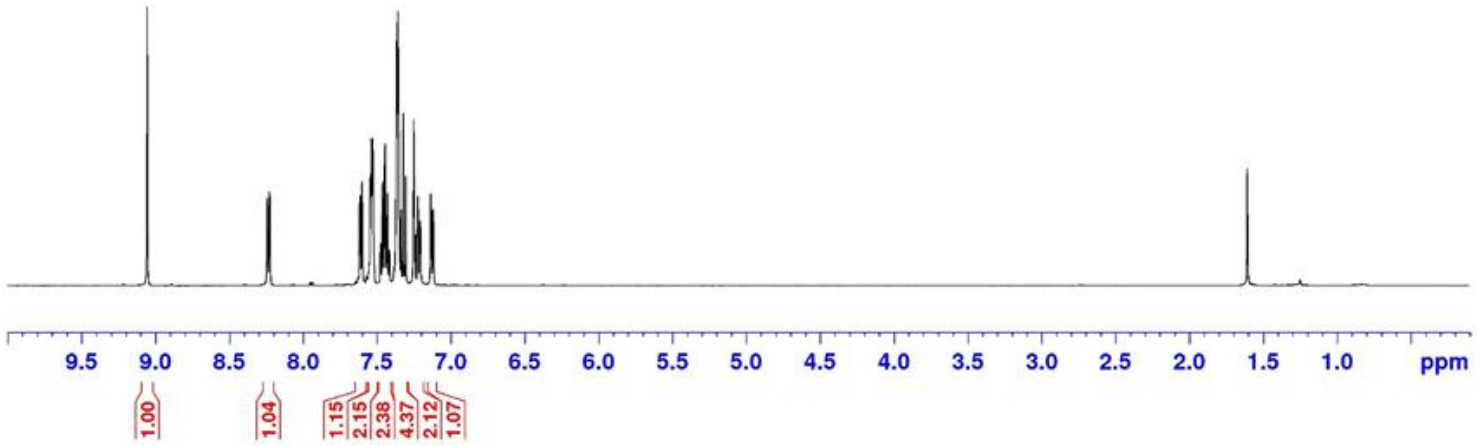

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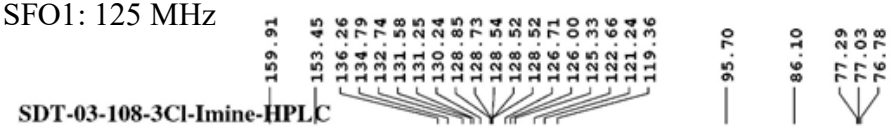
SFO1: 125 MHz



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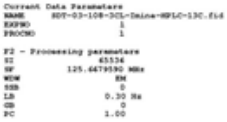
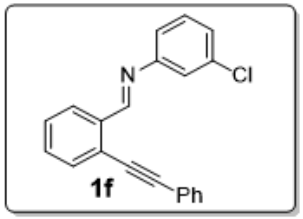
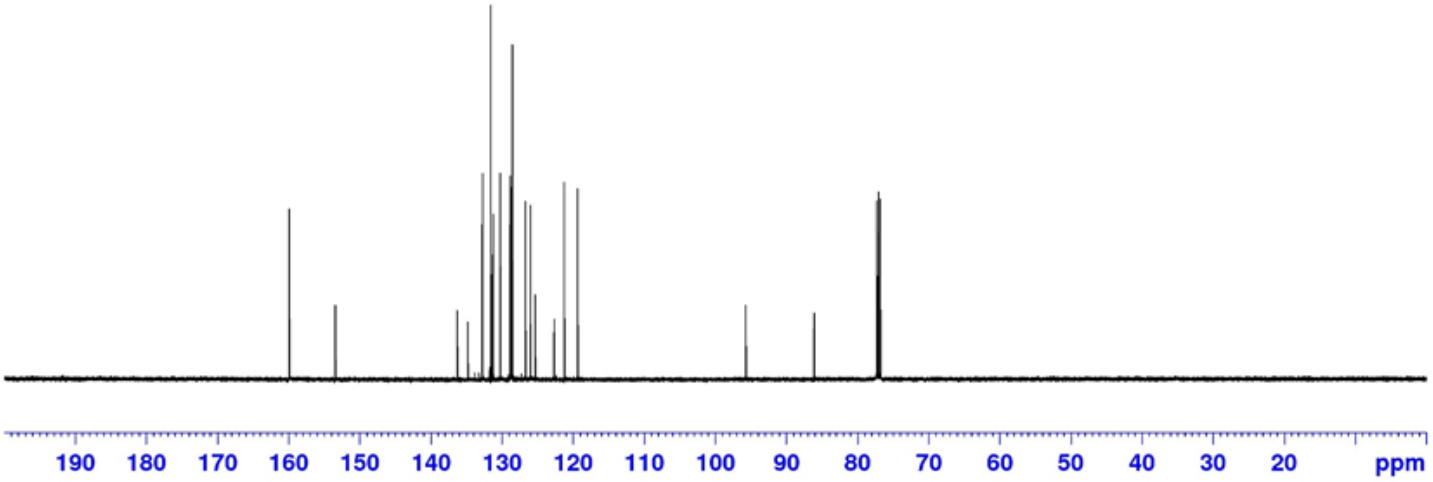
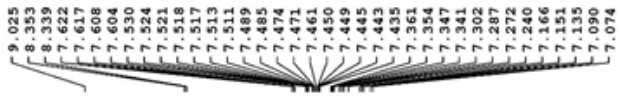






Solvent: $CDCl_3$
SFO1: 500 MHz



SDT-03-108-2Cl-Imine-HPLC

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PROCNO                   1

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SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
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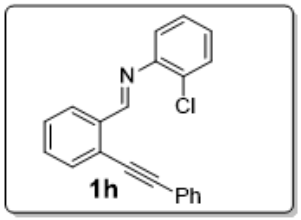
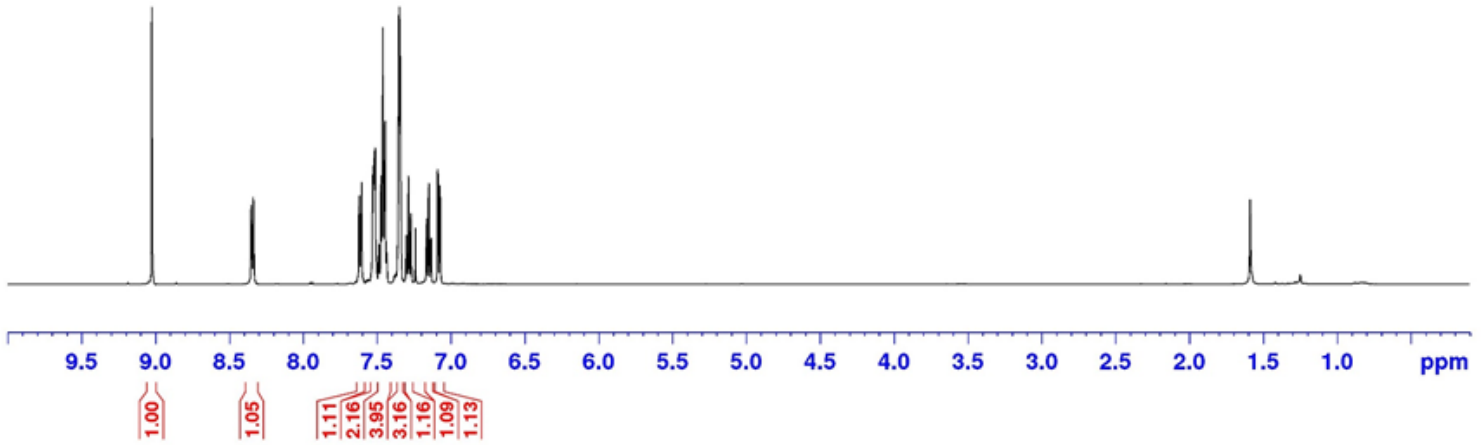
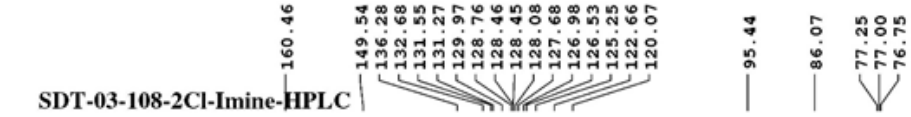
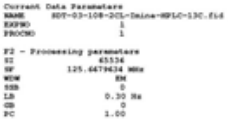
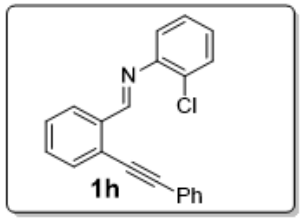


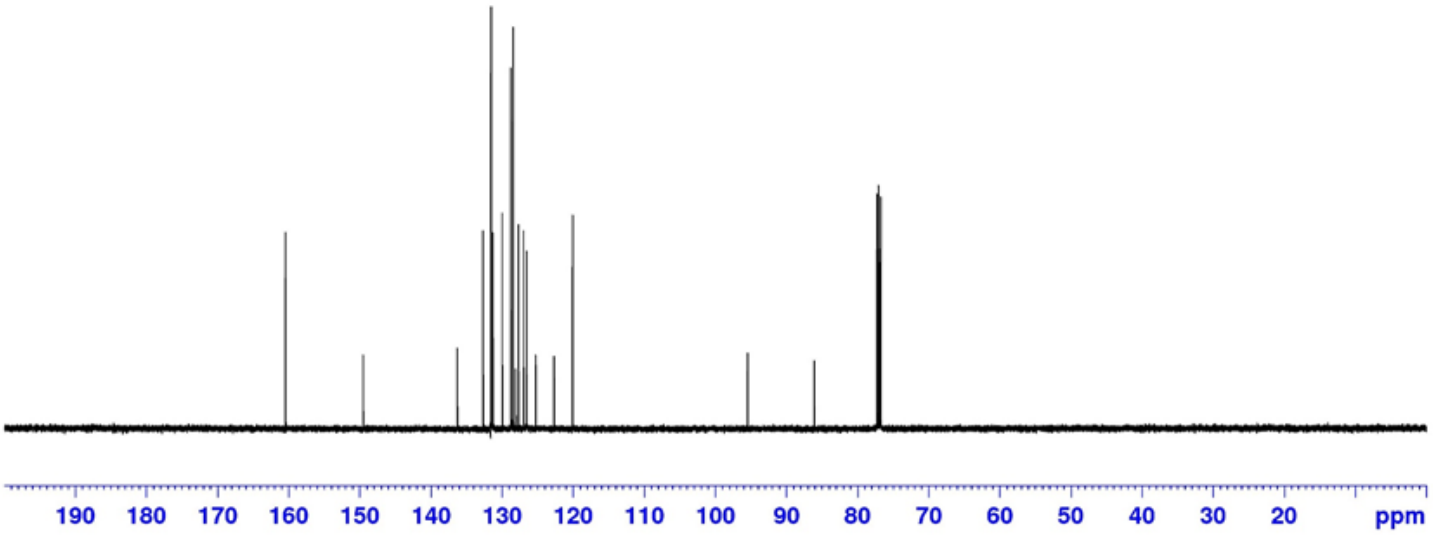


Solvent: $CDCl_3$
SFO1: 125 MHz





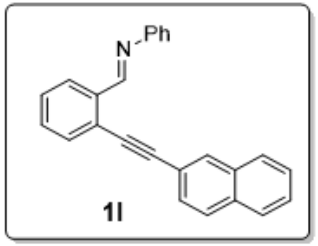


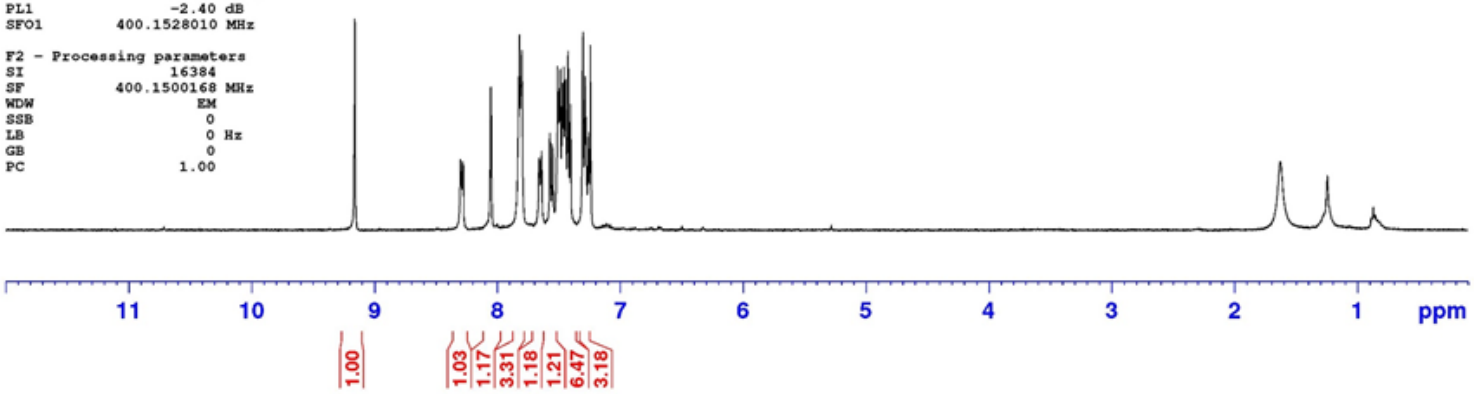


Solvent: $CDCl_3$
SFO1: 400 MHz

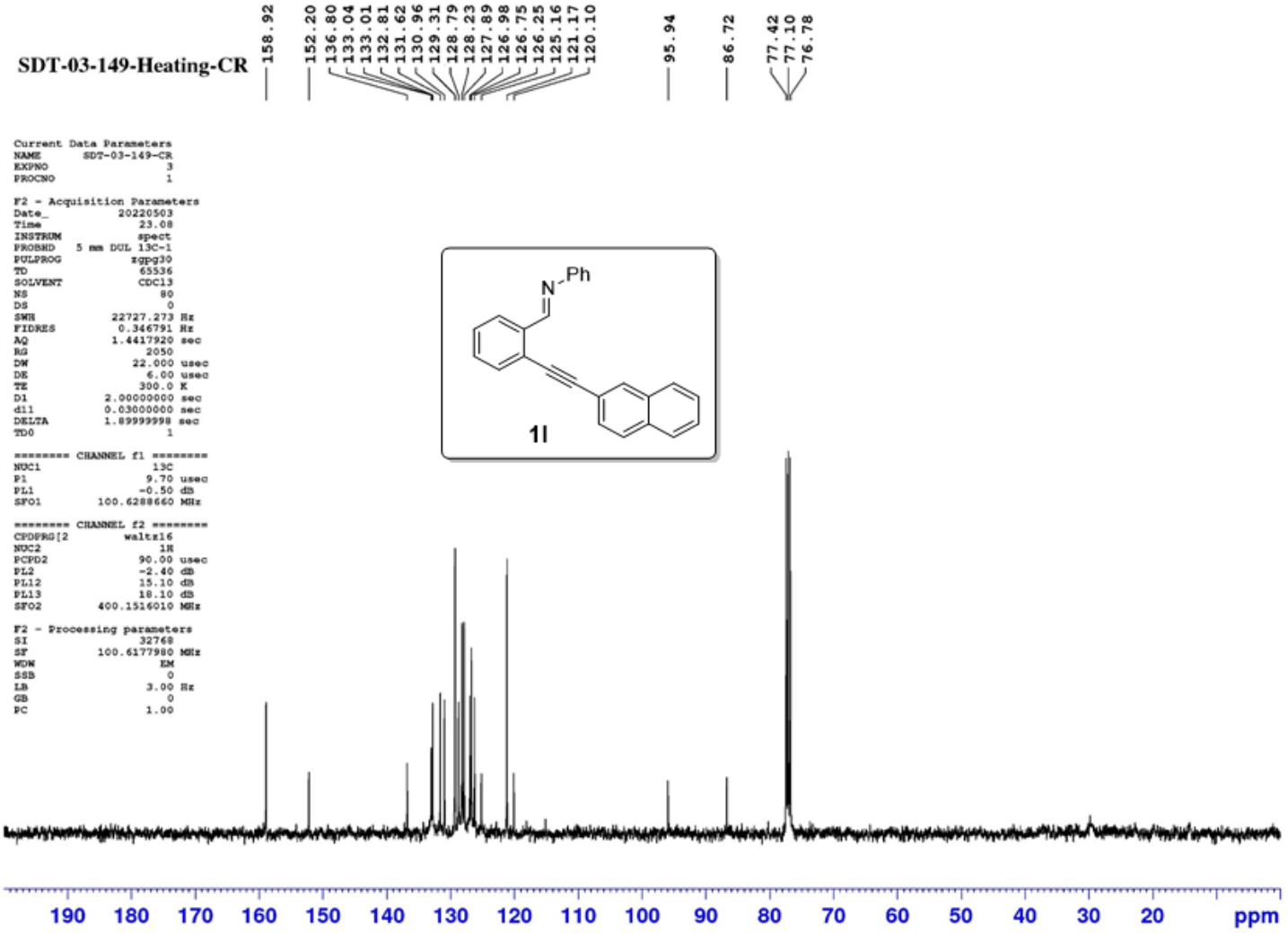
SDT-03-149-P

```
Current Data Parameters
NAME        SDT-03-149-P
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220523
Time              22.18
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    10
DS                     0
SWH            6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559039 sec
RG                   512
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 Hz
GB                     0
PC                  1.00
```

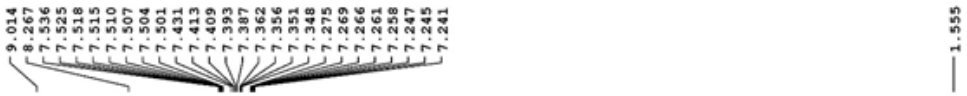




Solvent: $CDCl_3$
SFO1: 100 MHz

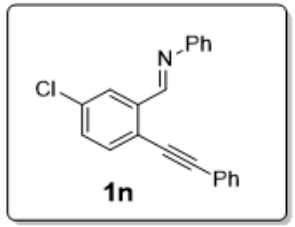


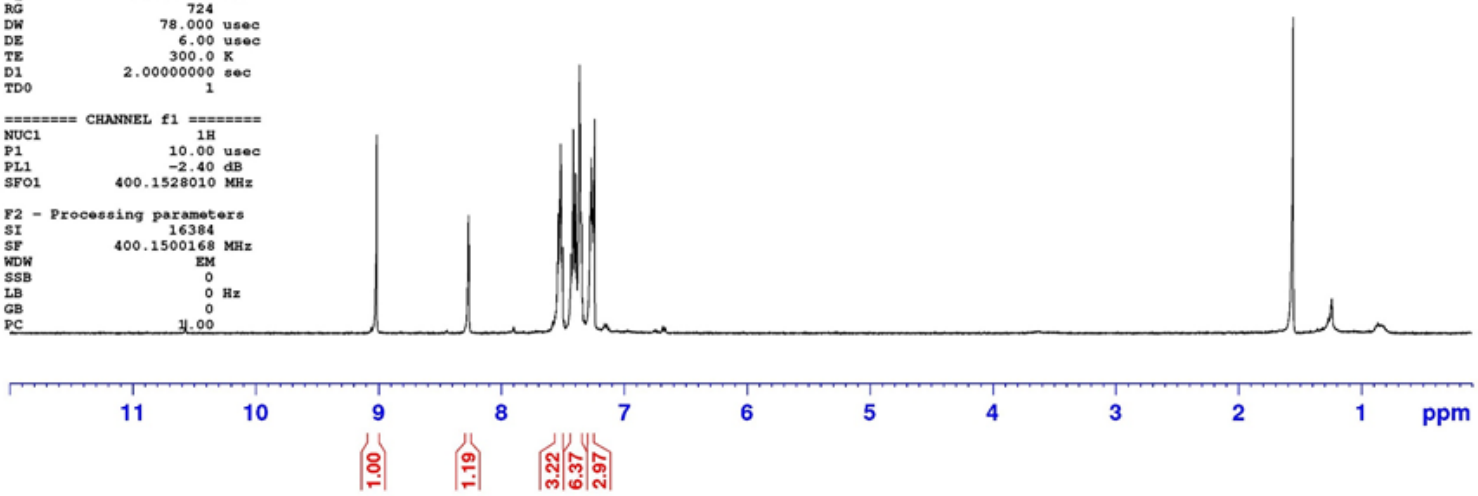
Solvent: $CDCl_3$
SFO1: 400 MHz



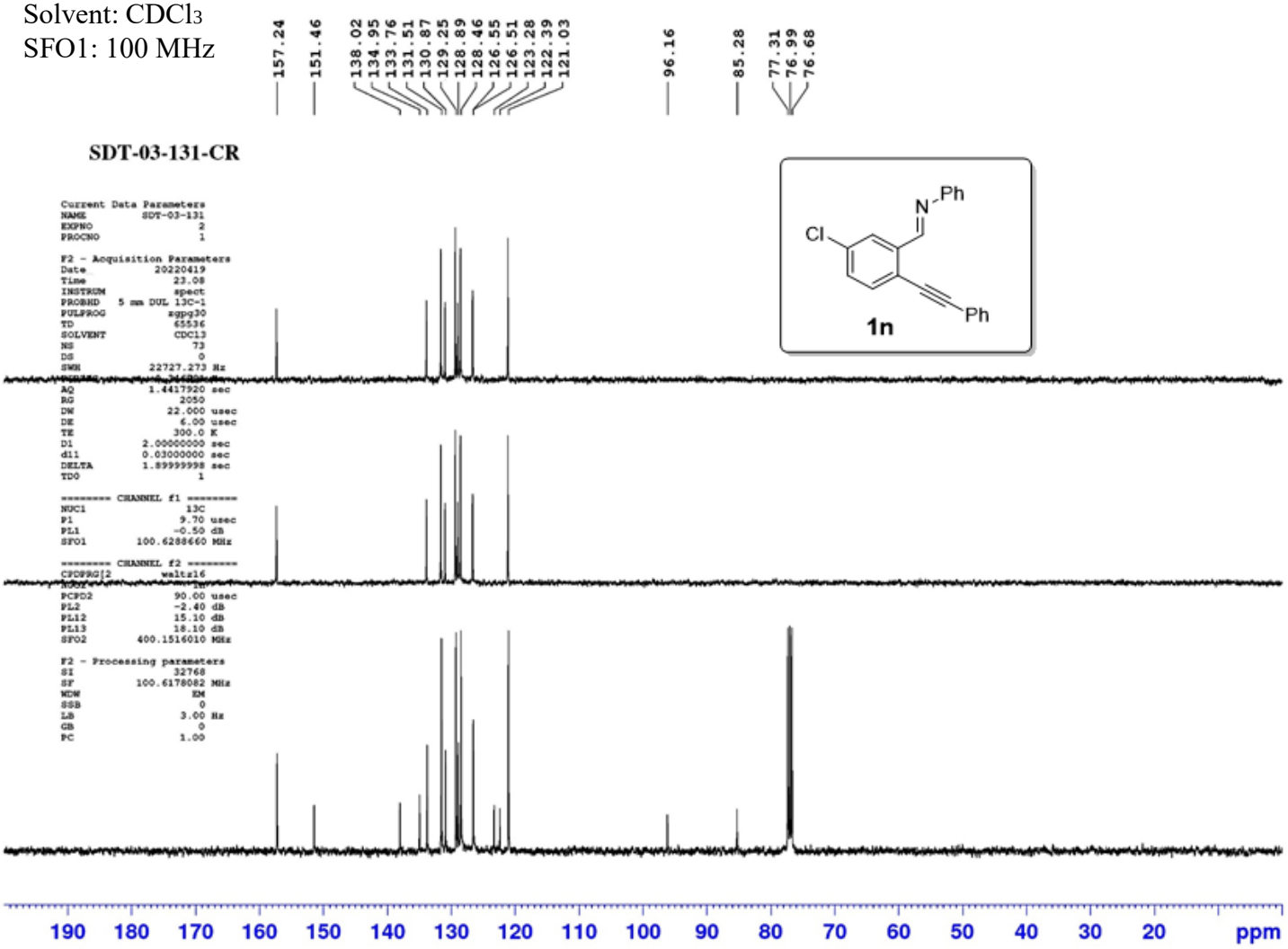
SDT-03-131-CR

```
Current Data Parameters
NAME          SDT-03-131
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20220415
Time               15.03
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    10
DS                     0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ               2.5559039 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 Hz
GB                     0
PC                  1.00
```

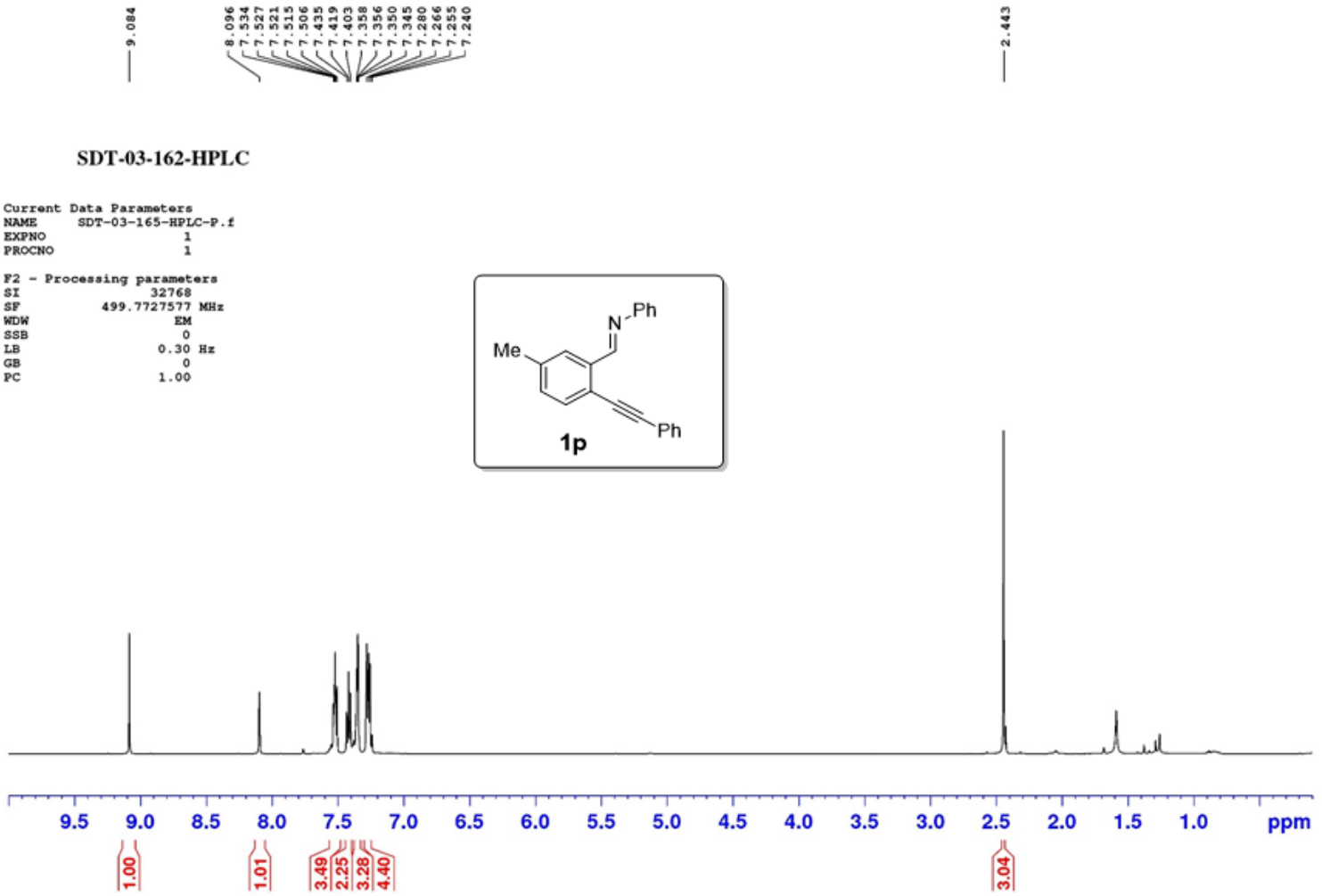




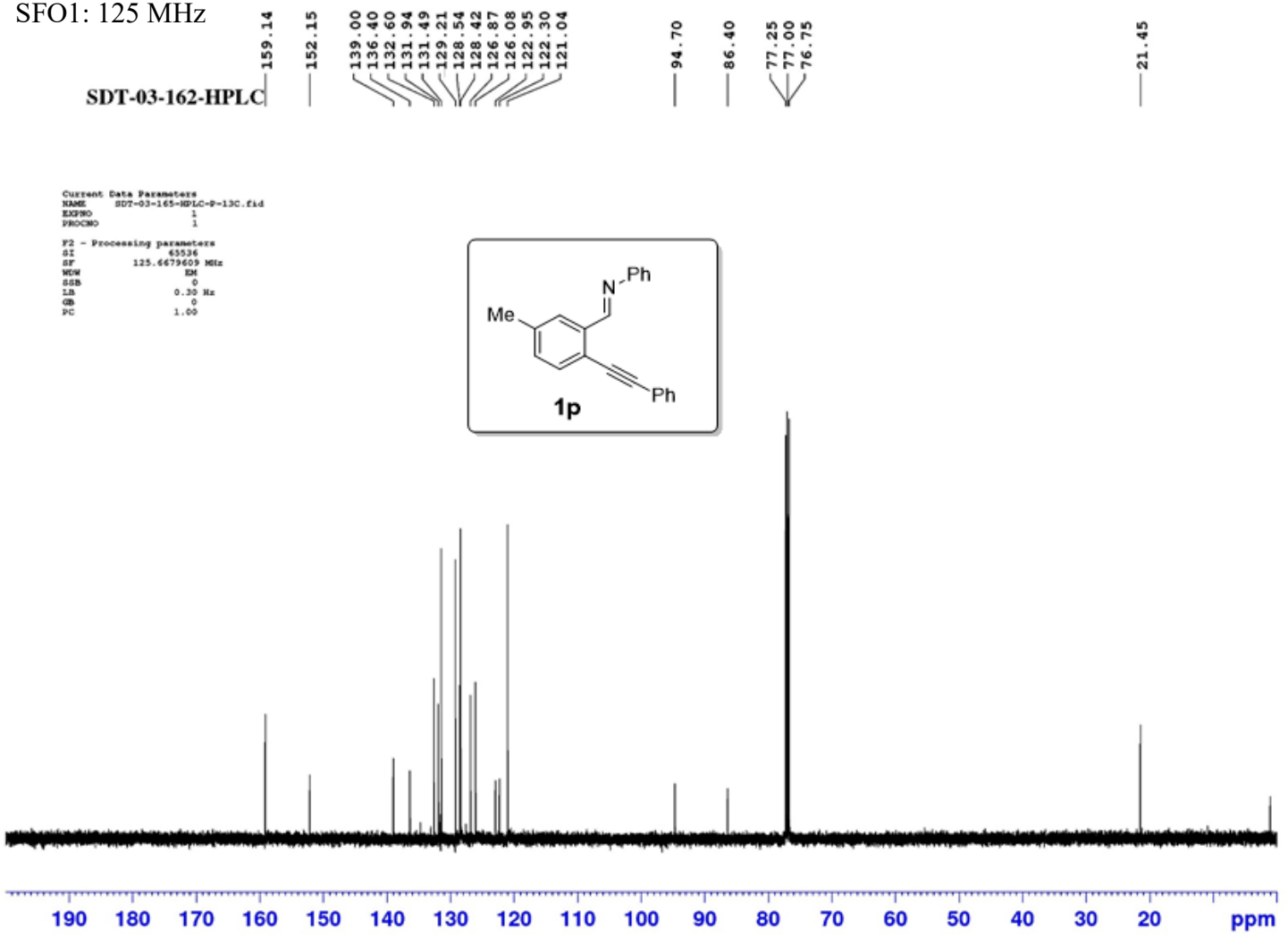
Solvent: $CDCl_3$
SFO1: 100 MHz



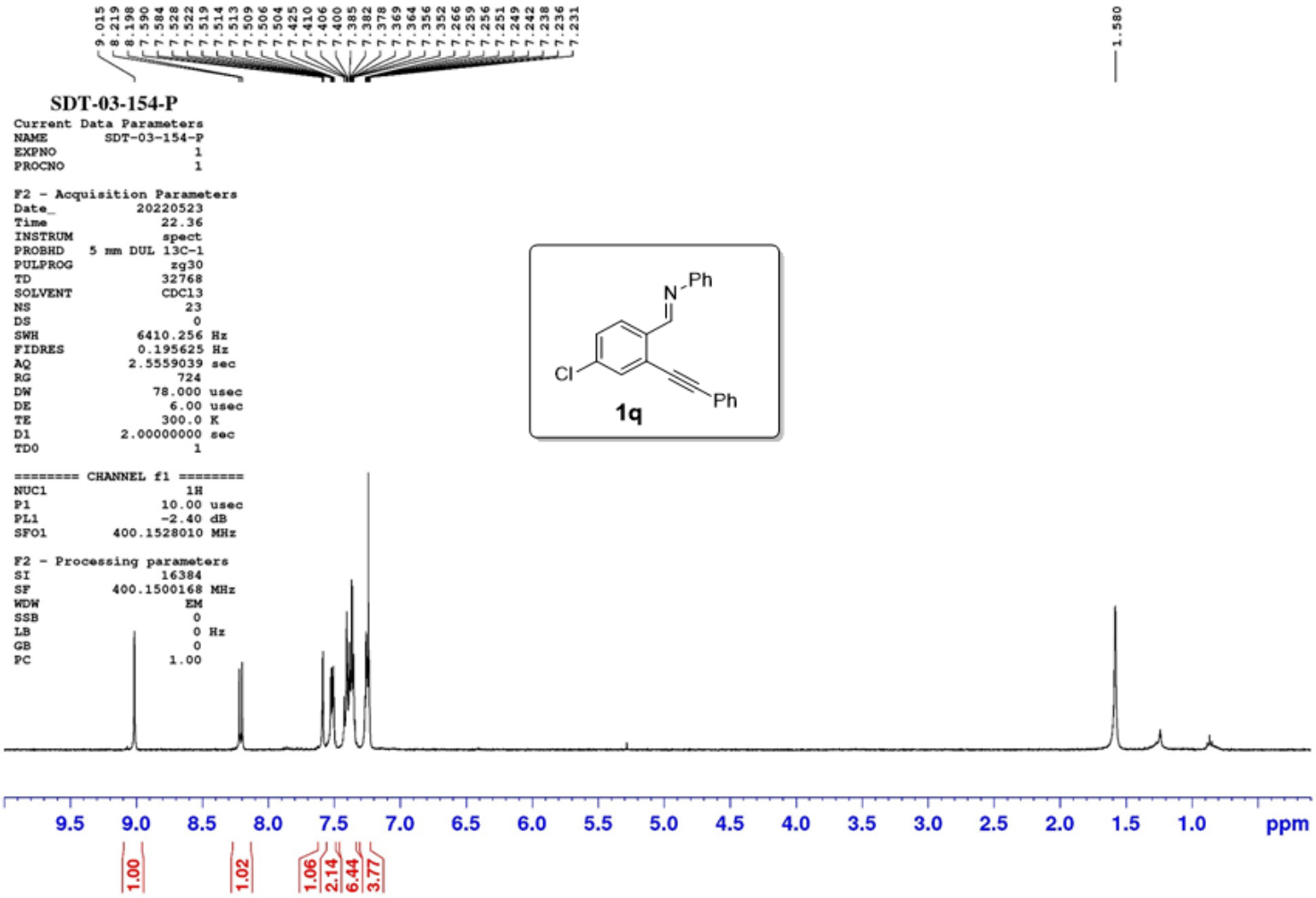
Solvent: $CDCl_3$
SFO1: 500 MHz



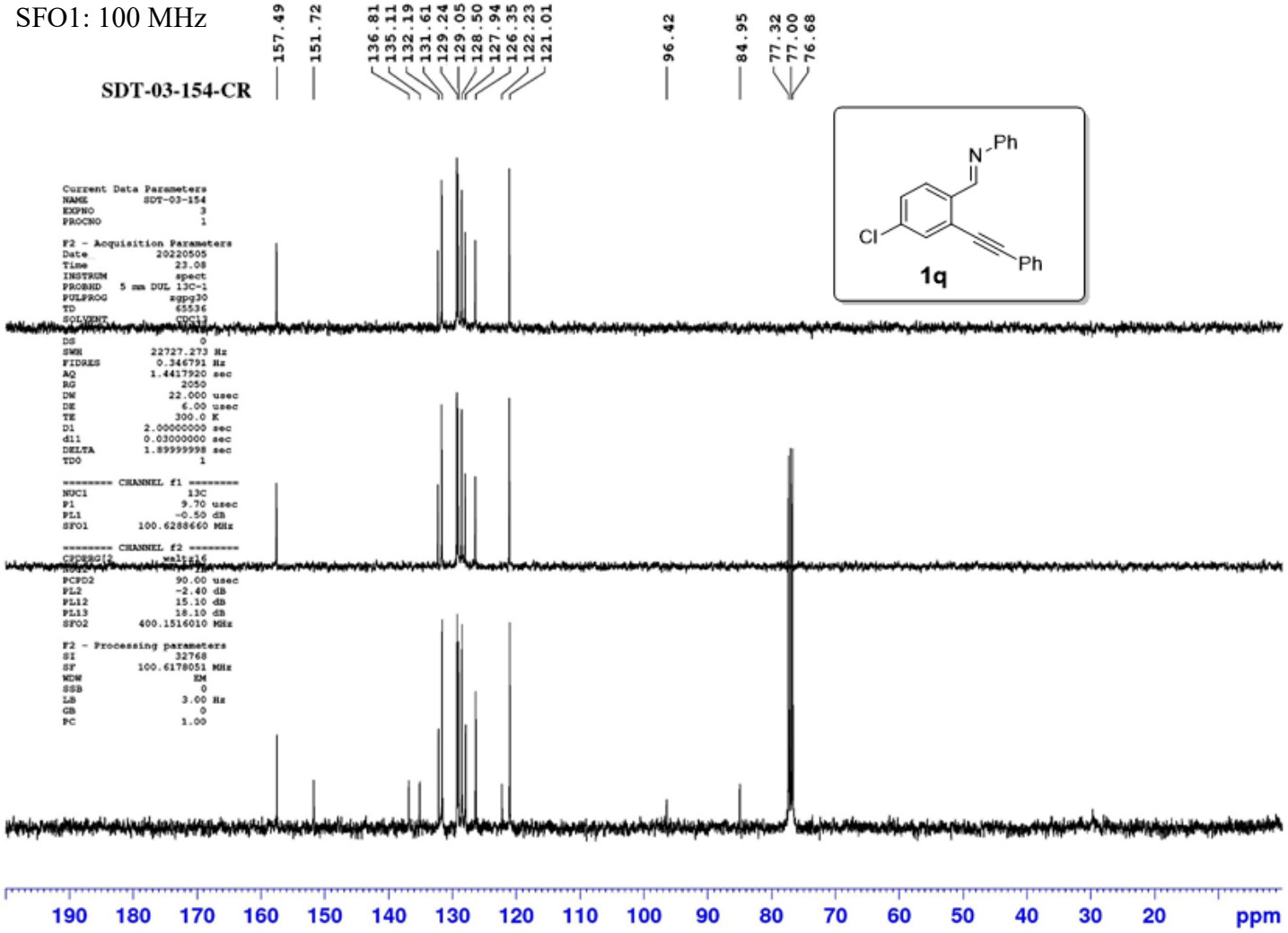
Solvent: $CDCl_3$
SFO1: 125 MHz



Solvent: $CDCl_3$
SFO1: 400 MHz

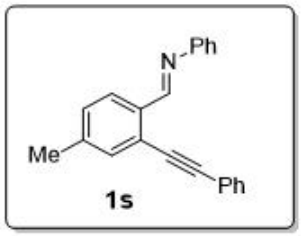


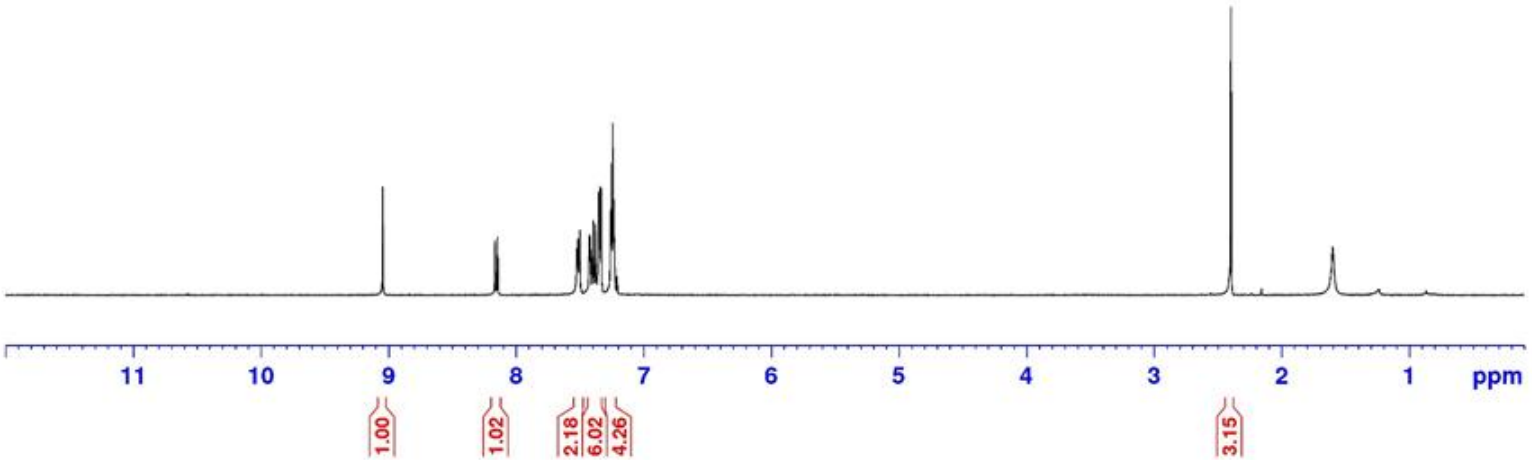
Solvent: $CDCl_3$
SFO1: 100 MHz



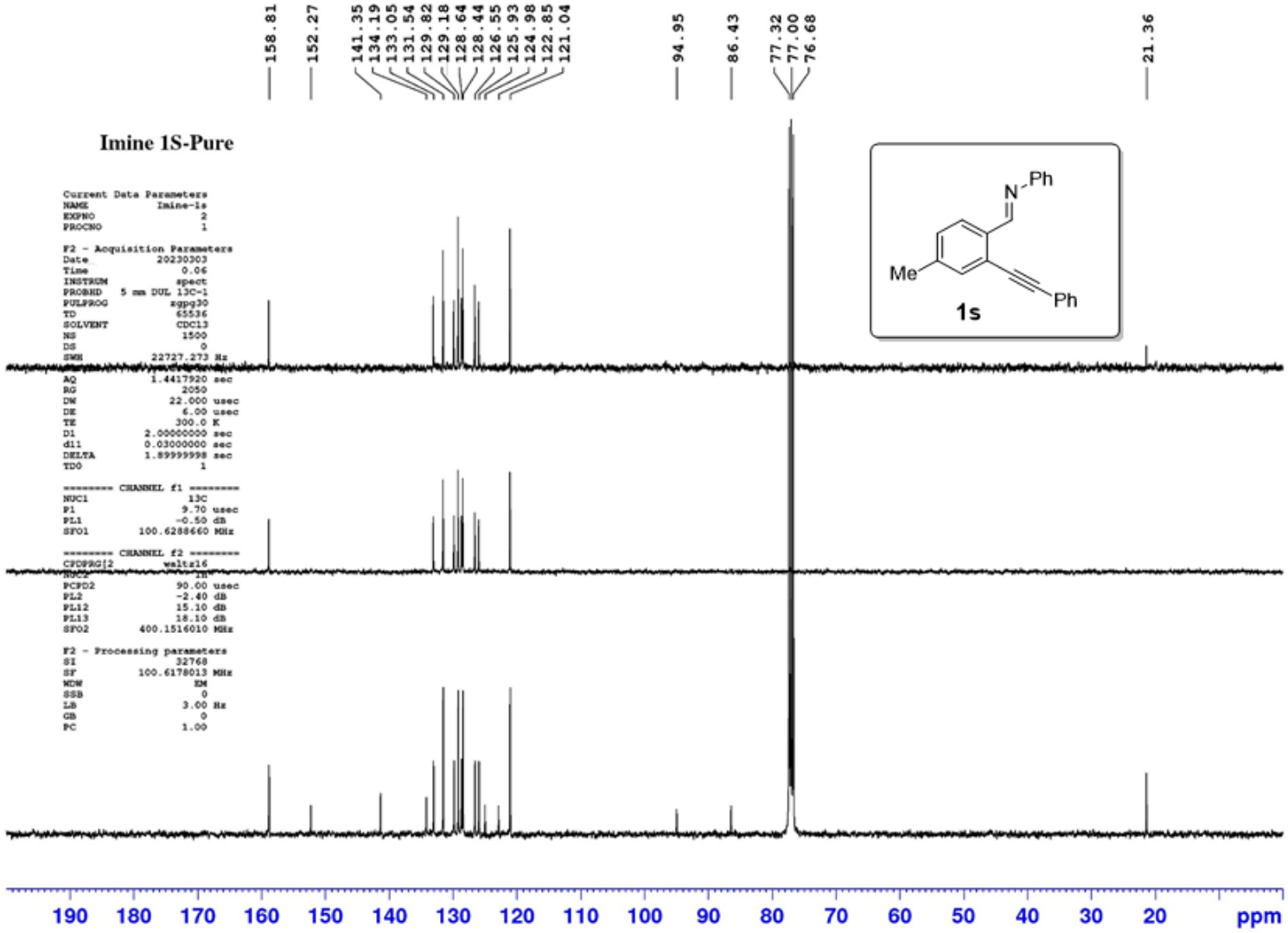
Solvent: $CDCl_3$
SFO1: 500 MHz

Imine 1S-Pure

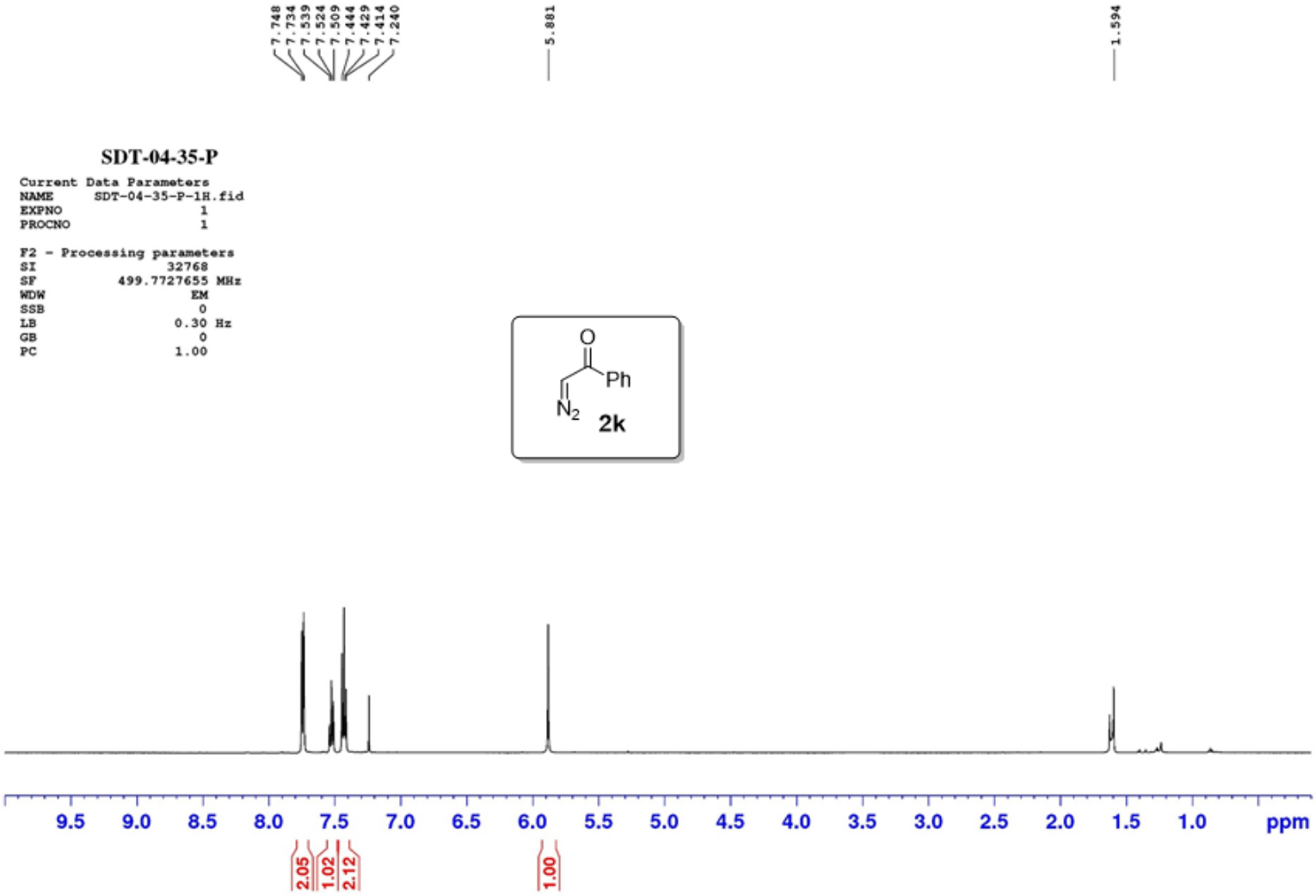




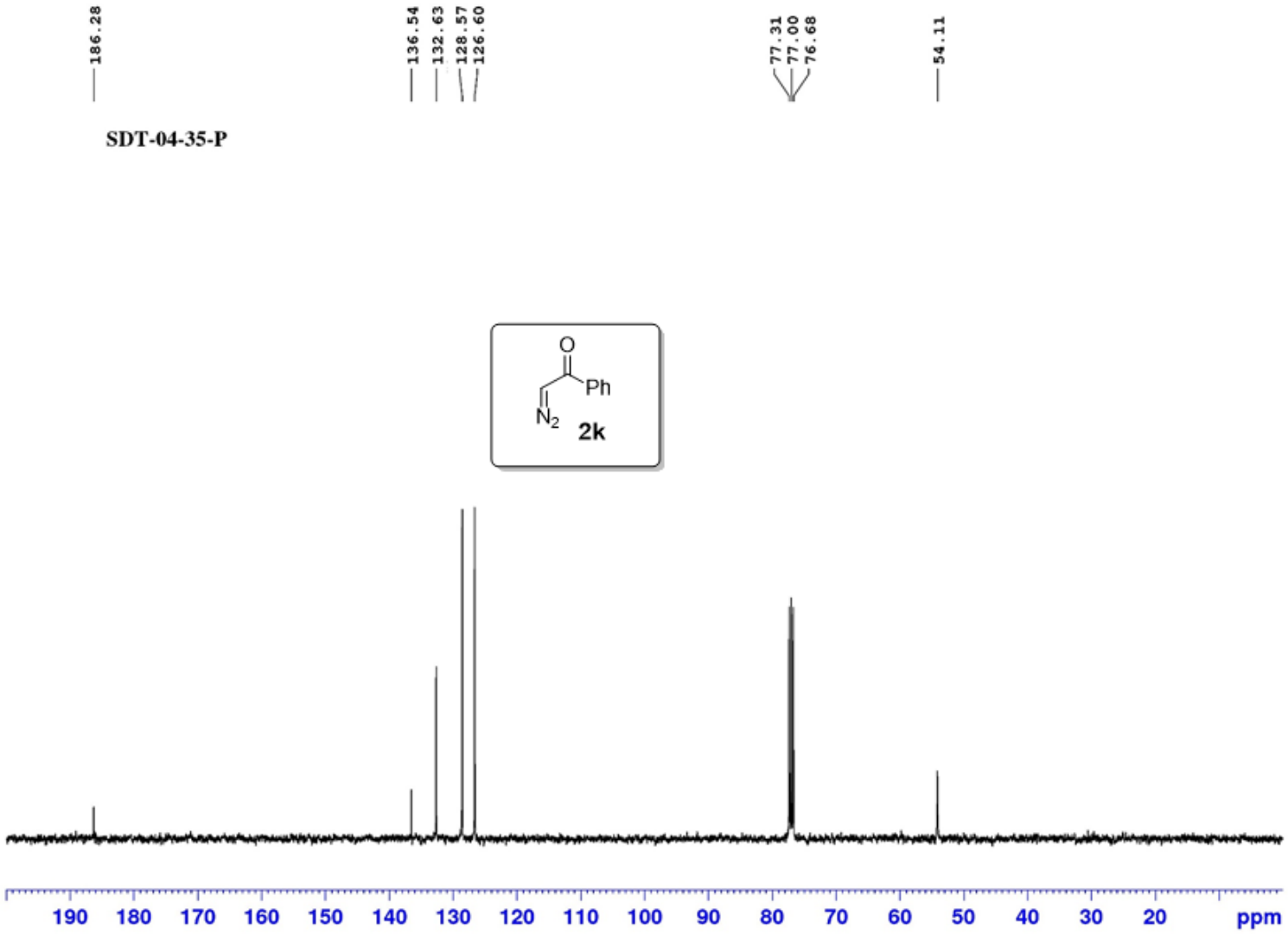
Solvent: $CDCl_3$
SFO1: 100 MHz



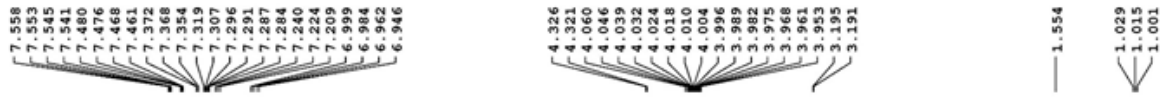
Solvent: $CDCl_3$
SFO1: 500 MHz



Solvent: $CDCl_3$
SFO1: 100 MHz

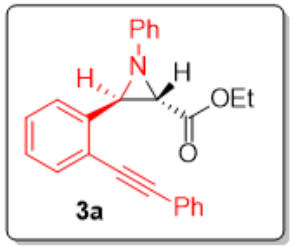


Solvent: $CDCl_3$
SFO1: 500 MHz

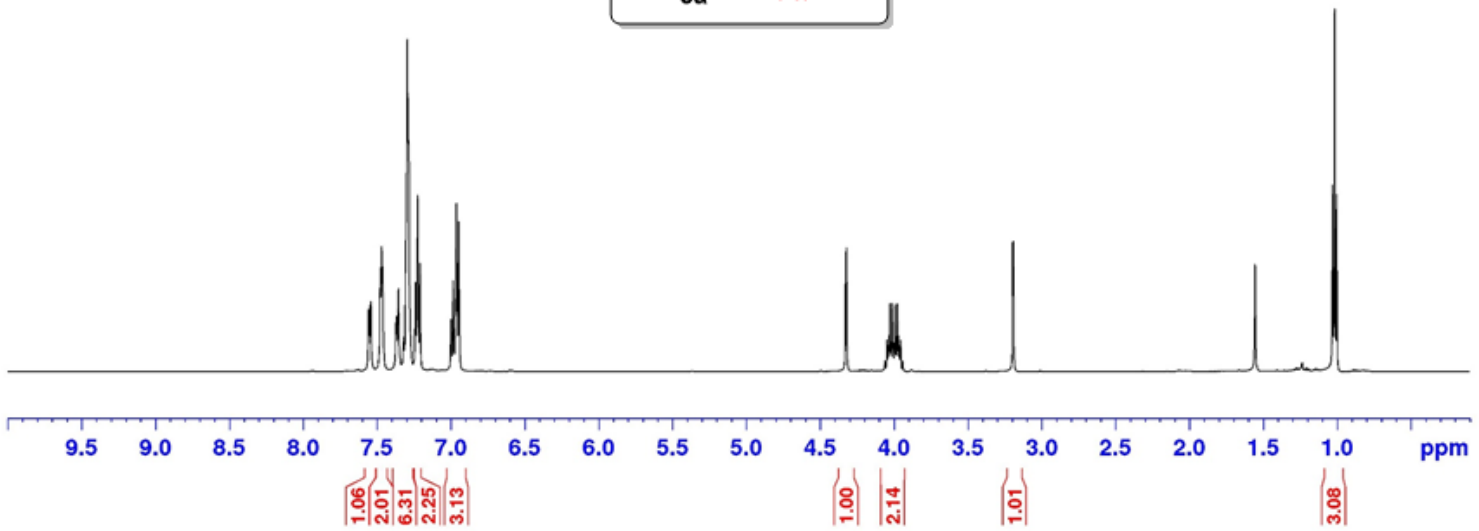


SDt-03-179-AP-C

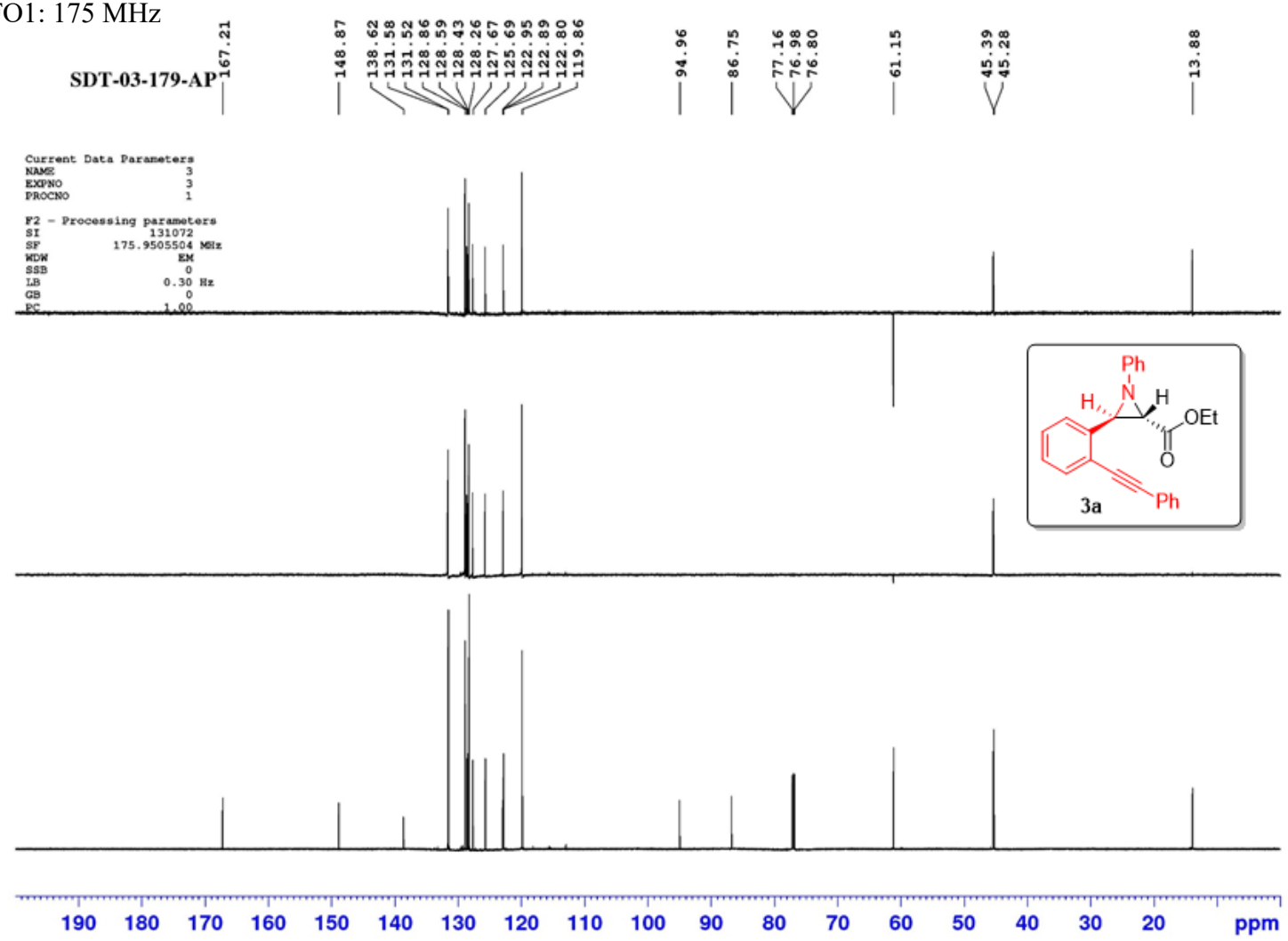
```
Current Data Parameters
NAME      SDT-03-179-AP-C-Int
EXPNO                       1
PROCNO                      1

F2 - Processing parameters
SI                      32768
SF          499.7727705 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



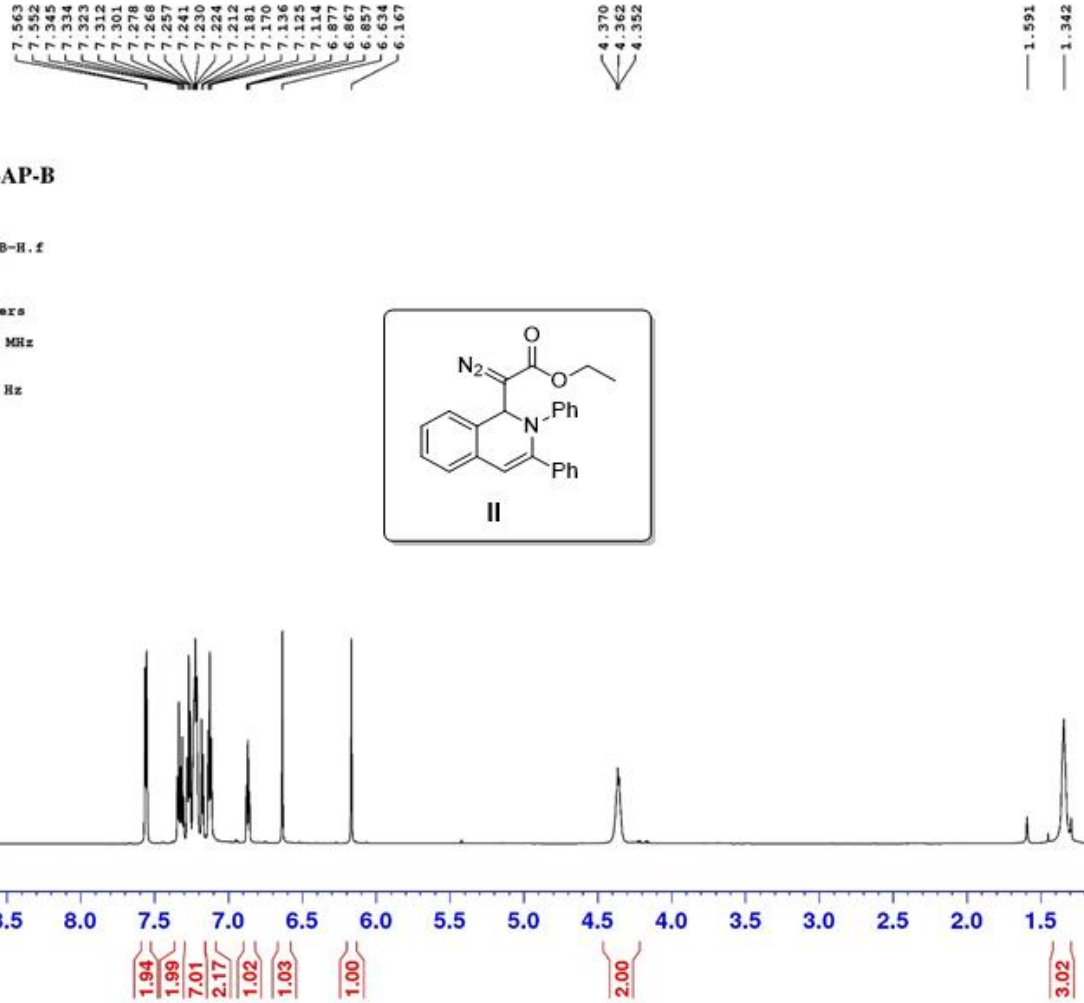
3a



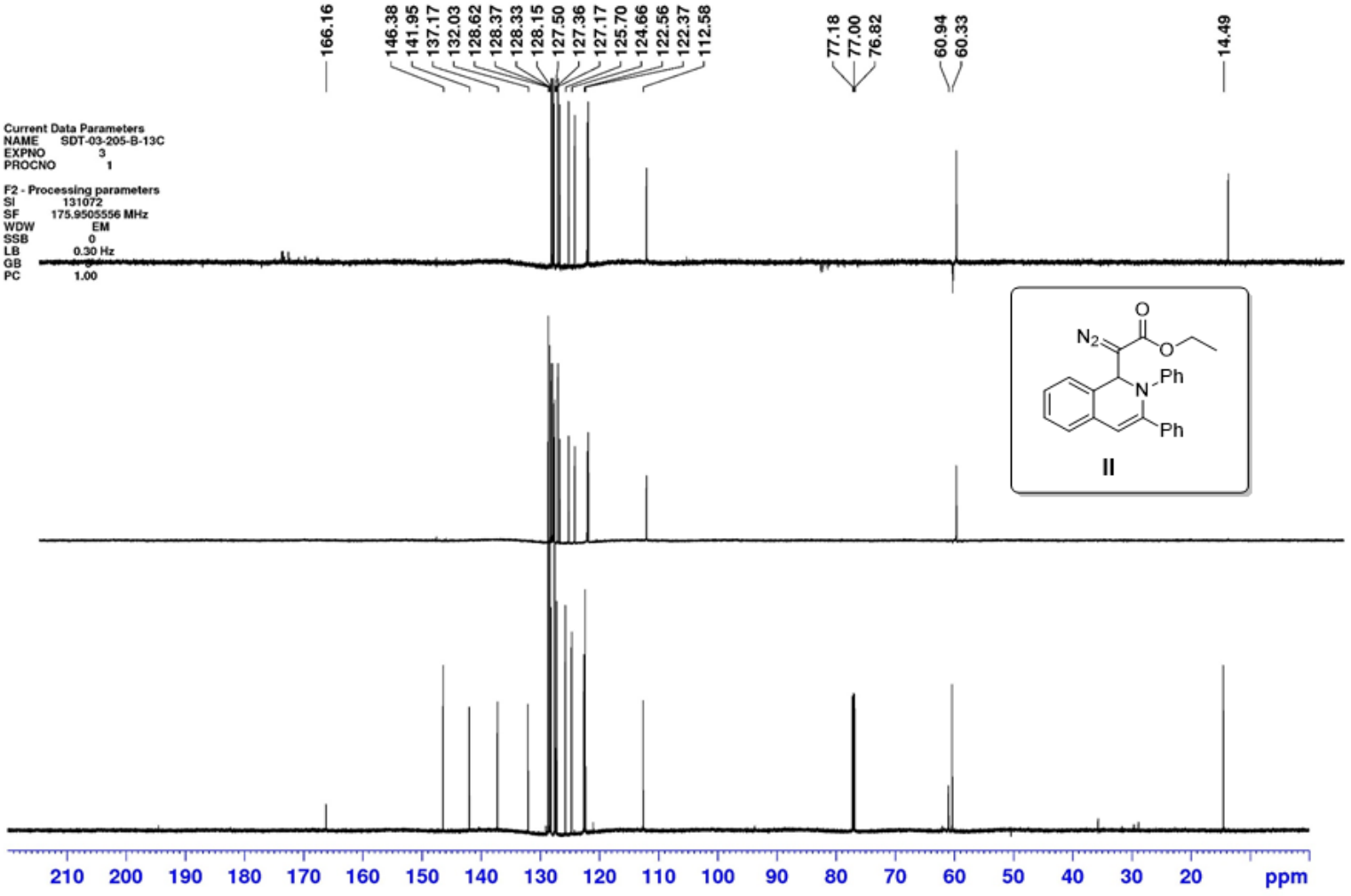
Solvent: $CDCl_3$
SFO1: 175 MHz



Solvent: $CDCl_3$
SFO1: 700 MHz



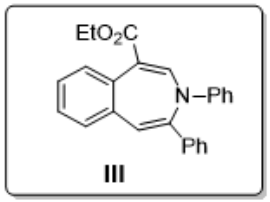
Solvent: $CDCl_3$
SFO1: 175 MHz



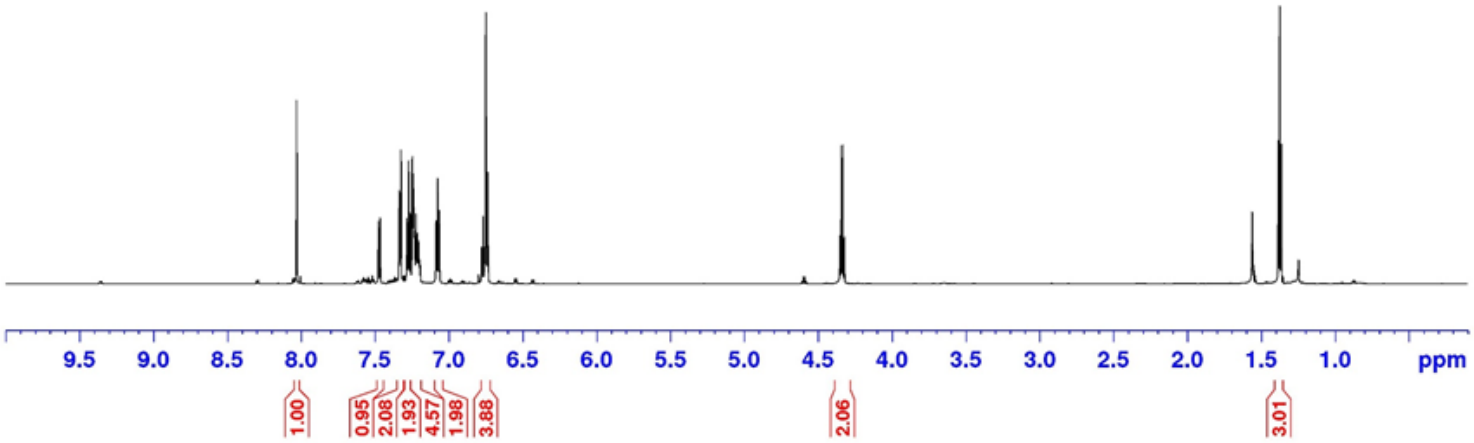
Solvent: $CDCl_3$
SFO1: 700 MHz

SDT-03-211-AP

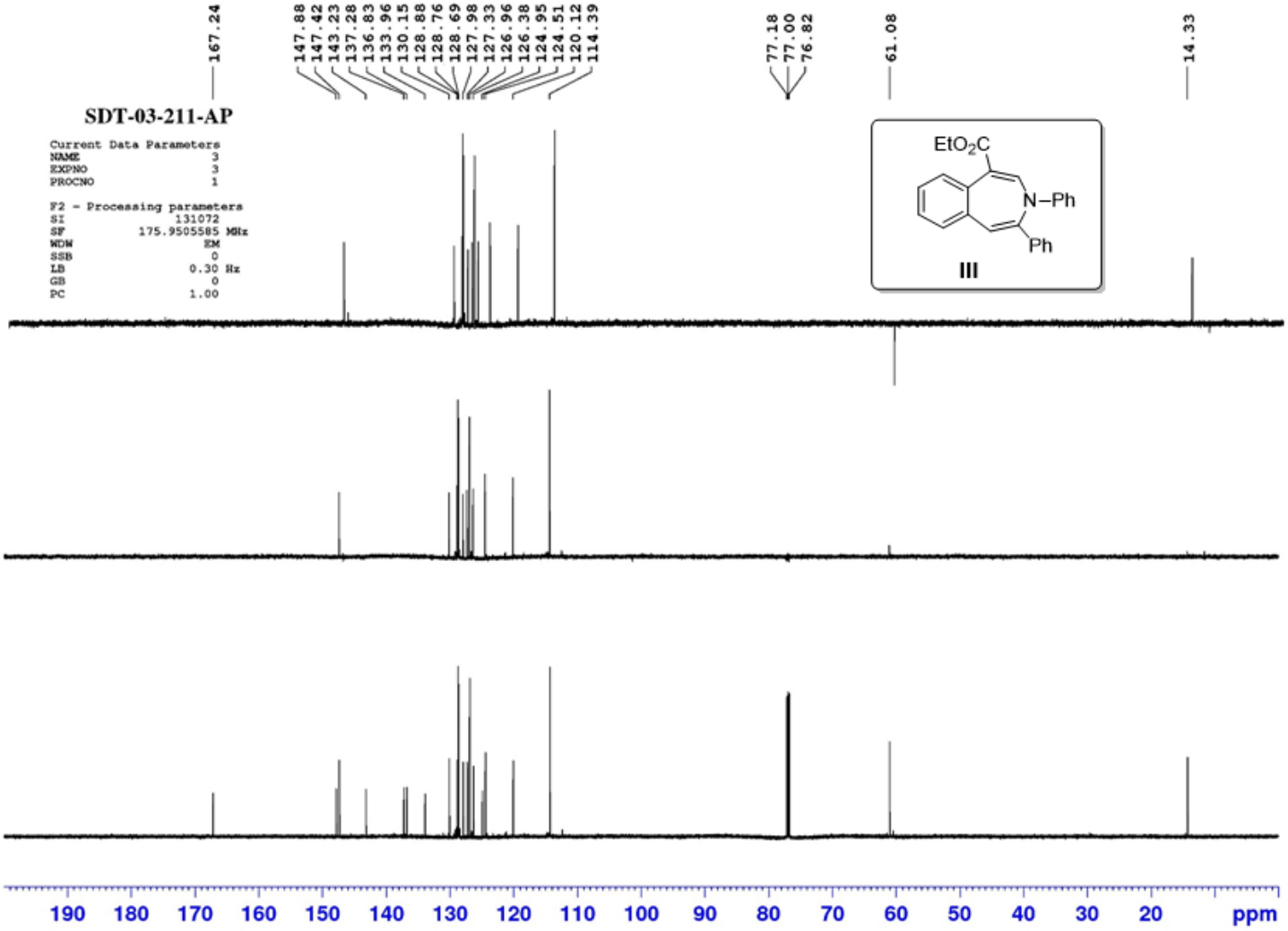
```
Current Data Parameters
NAME      SDT-03-211-AP-H.fid
EXPNO                       1
PROCNO                      1

F2 - Processing parameters
SI                      65536
SF          699.7431036 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



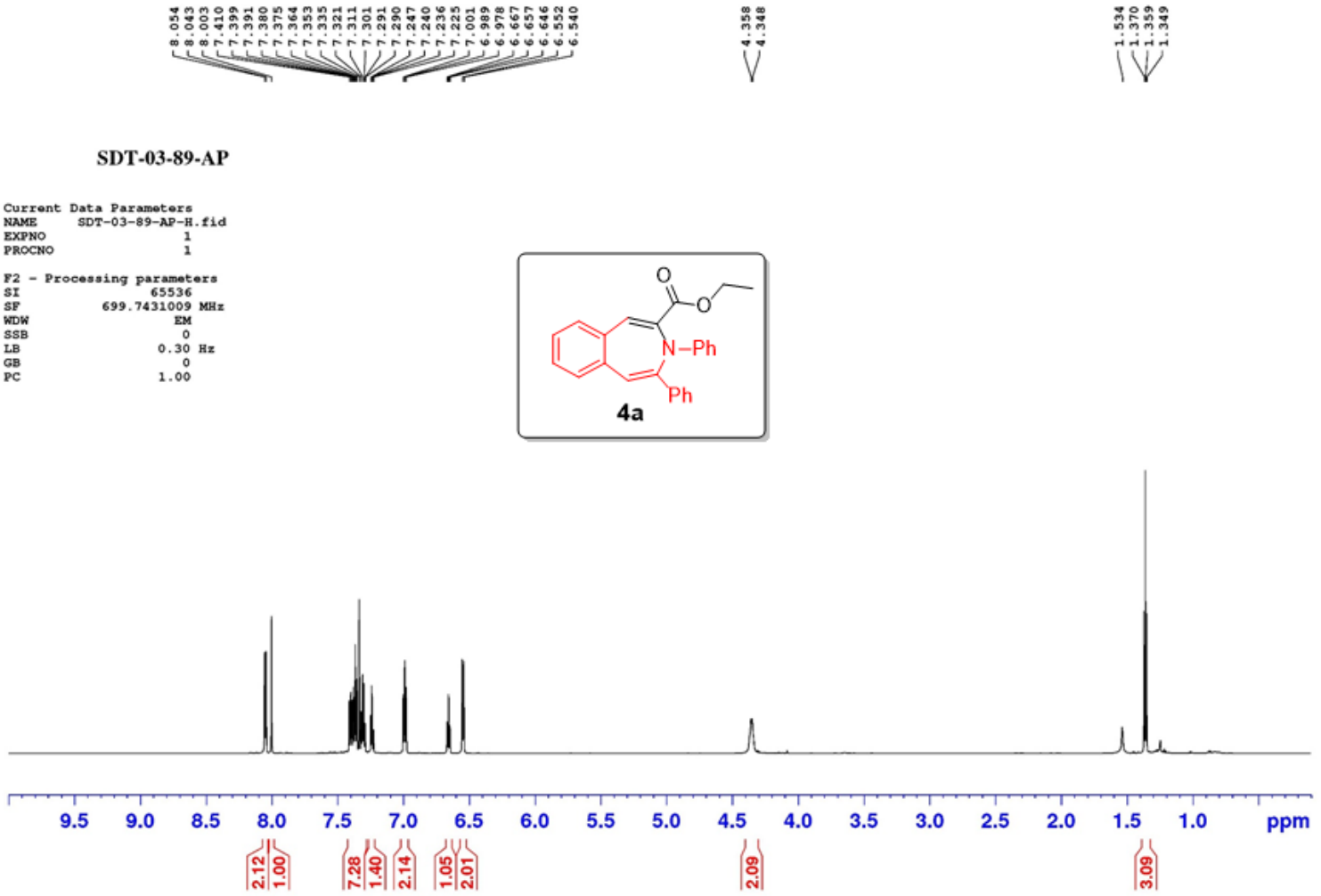
III



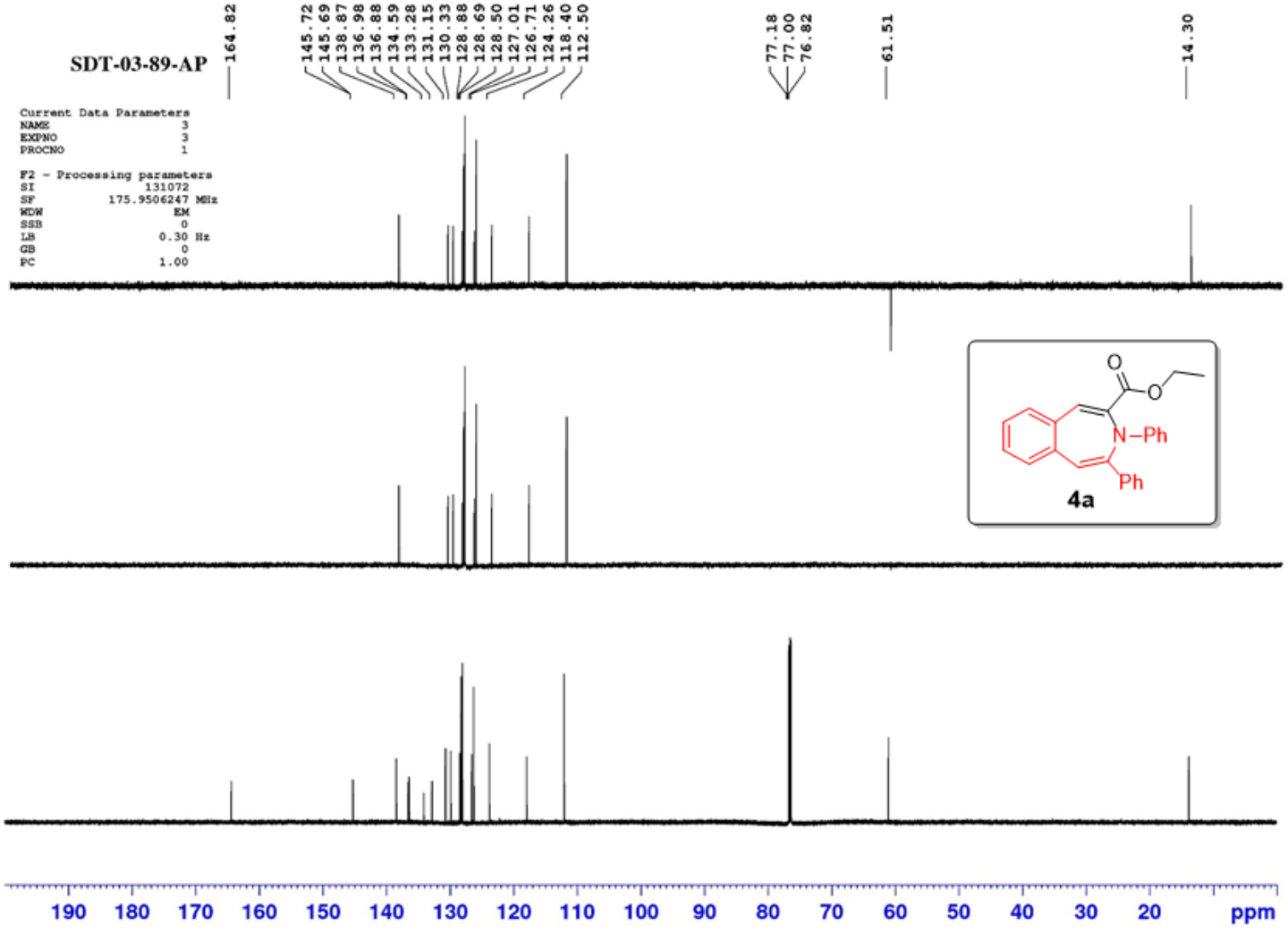
Solvent: $CDCl_3$
SFO1: 175 MHz



Solvent: $CDCl_3$
SFO1: 700 MHz



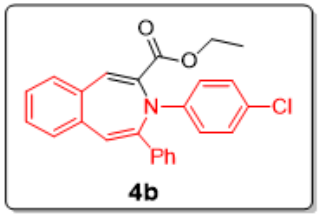
Solvent: $CDCl_3$
SFO1: 175 MHz



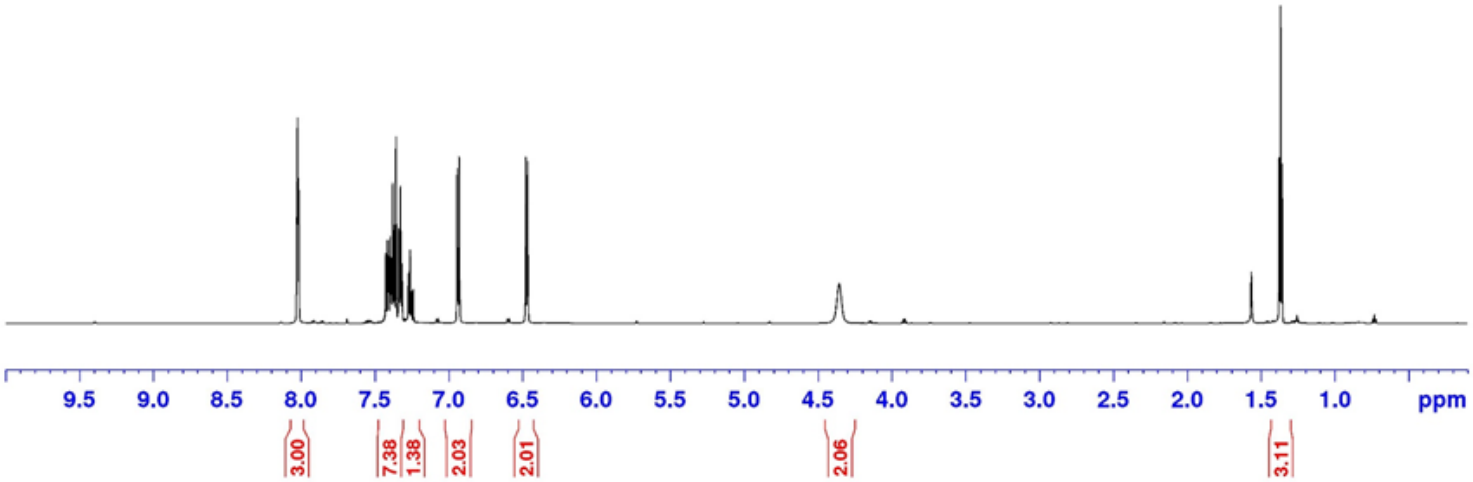
Solvent: $CDCl_3$
SFO1: 700 MHz

SDT-03-90-2

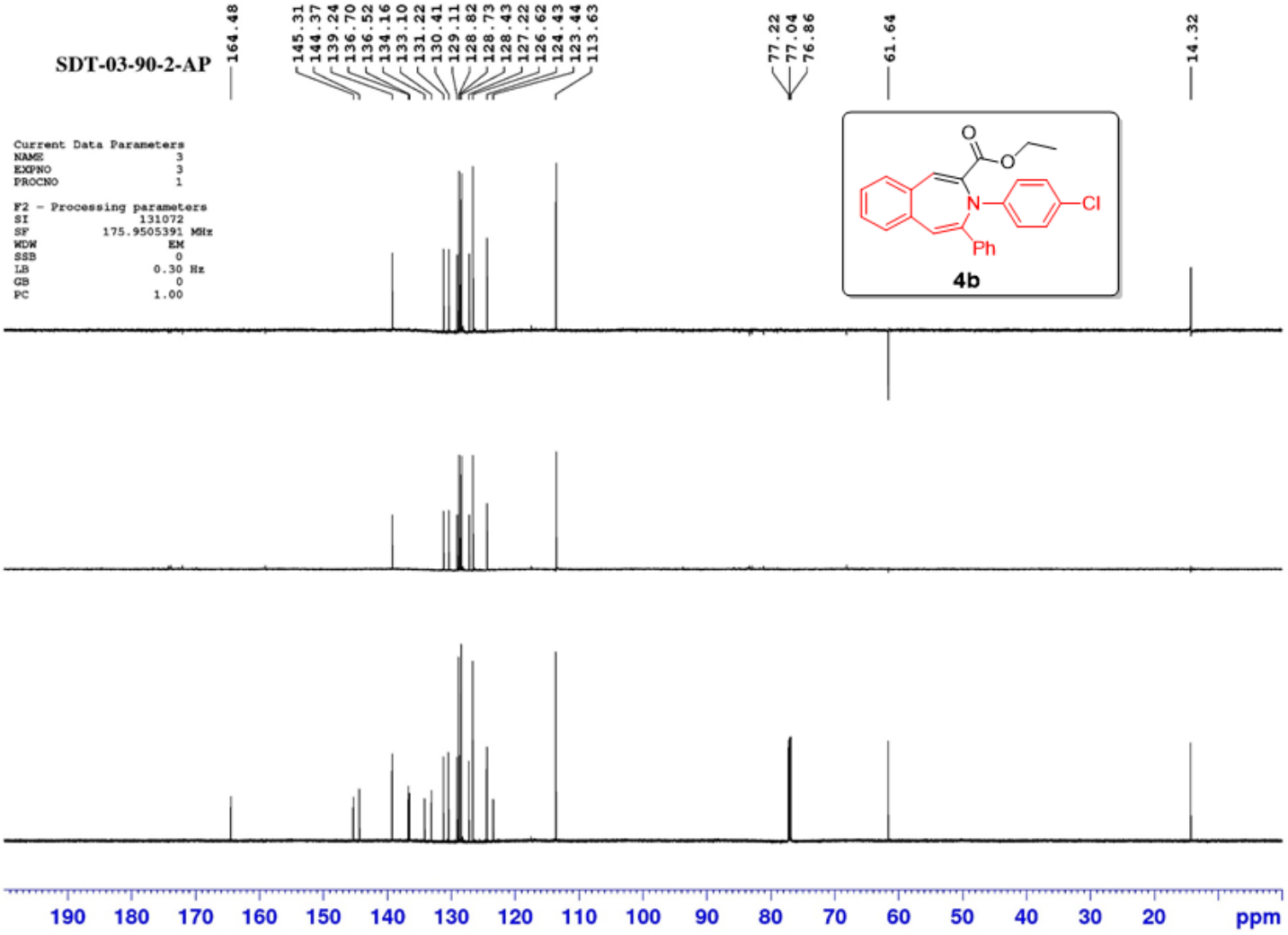
```
Current Data Parameters
NAME      SDT-03-90-2-AP-H.fi
EXPNO                      1
PROCNO                     1

F2 - Processing parameters
SI                     65536
SF           699.7431022 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



4b



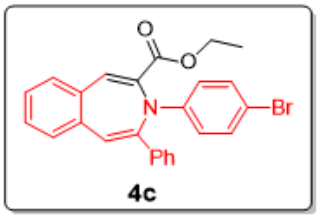
Solvent: $CDCl_3$
SFO1: 175 MHz



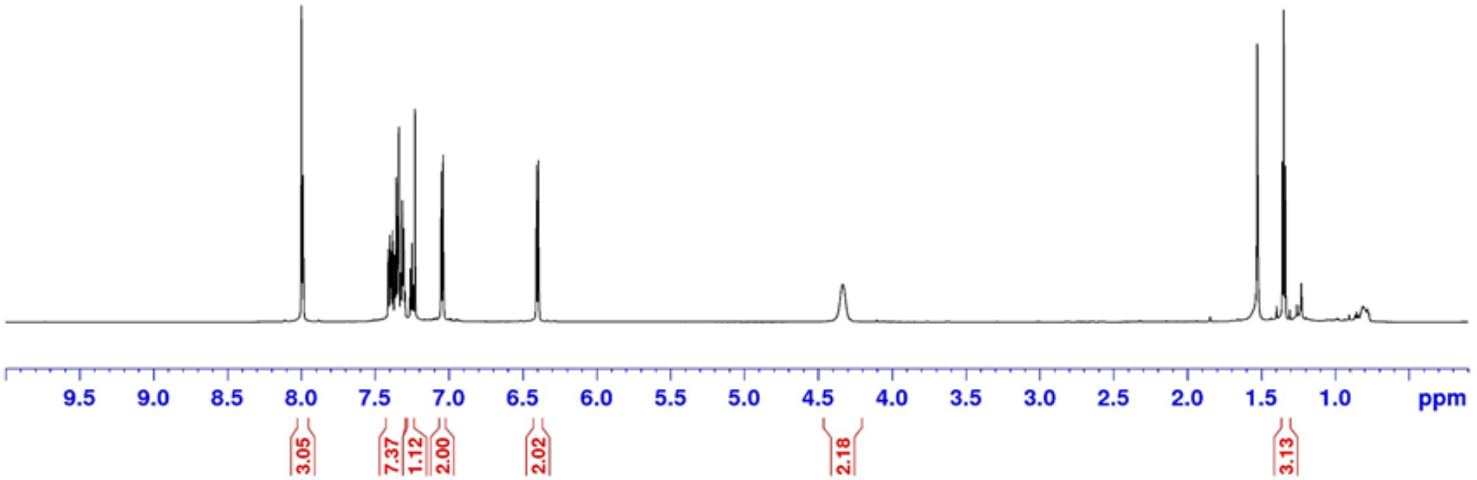
Solvent: $CDCl_3$
SFO1: 700 MHz

SDT-03-100-AP-r

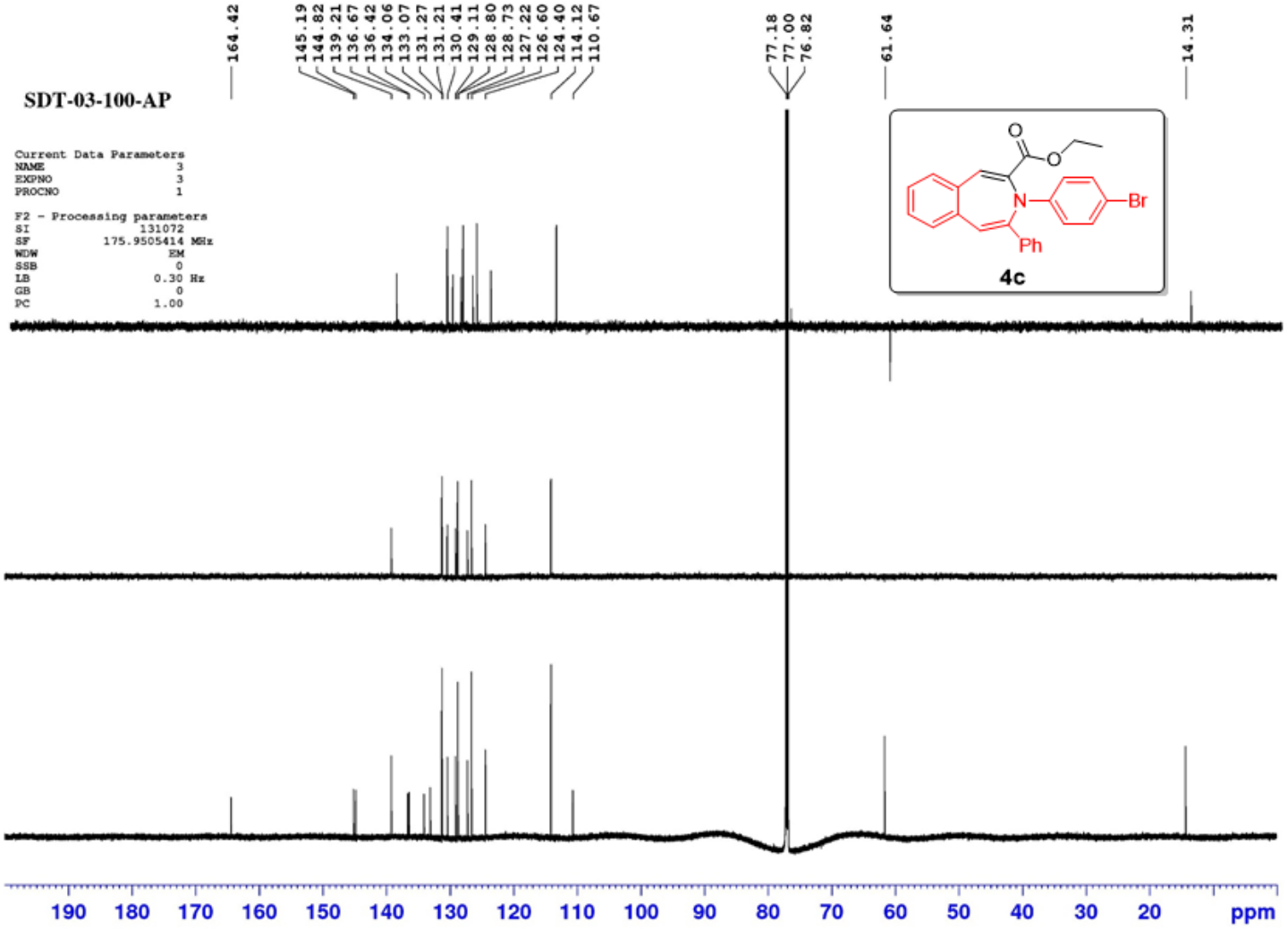
```
Current Data Parameters
NAME      SDT-03-100-AP-r-H.f
EXPNO                      1
PROCNO                     1

F2 - Processing parameters
SI                     65536
SF           699.7431058 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



4c



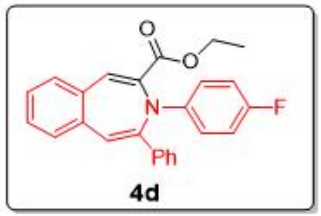
Solvent: $CDCl_3$
SFO1: 175 MHz



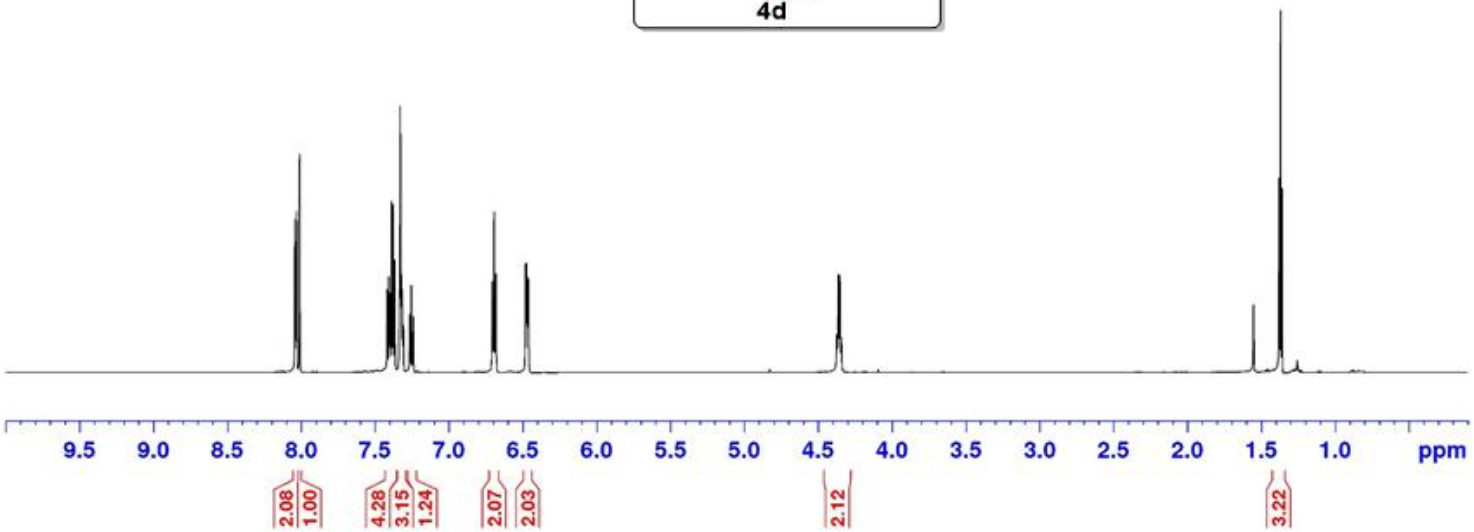
Solvent: $CDCl_3$
SFO1: 700 MHz

SDT-03-69-AP

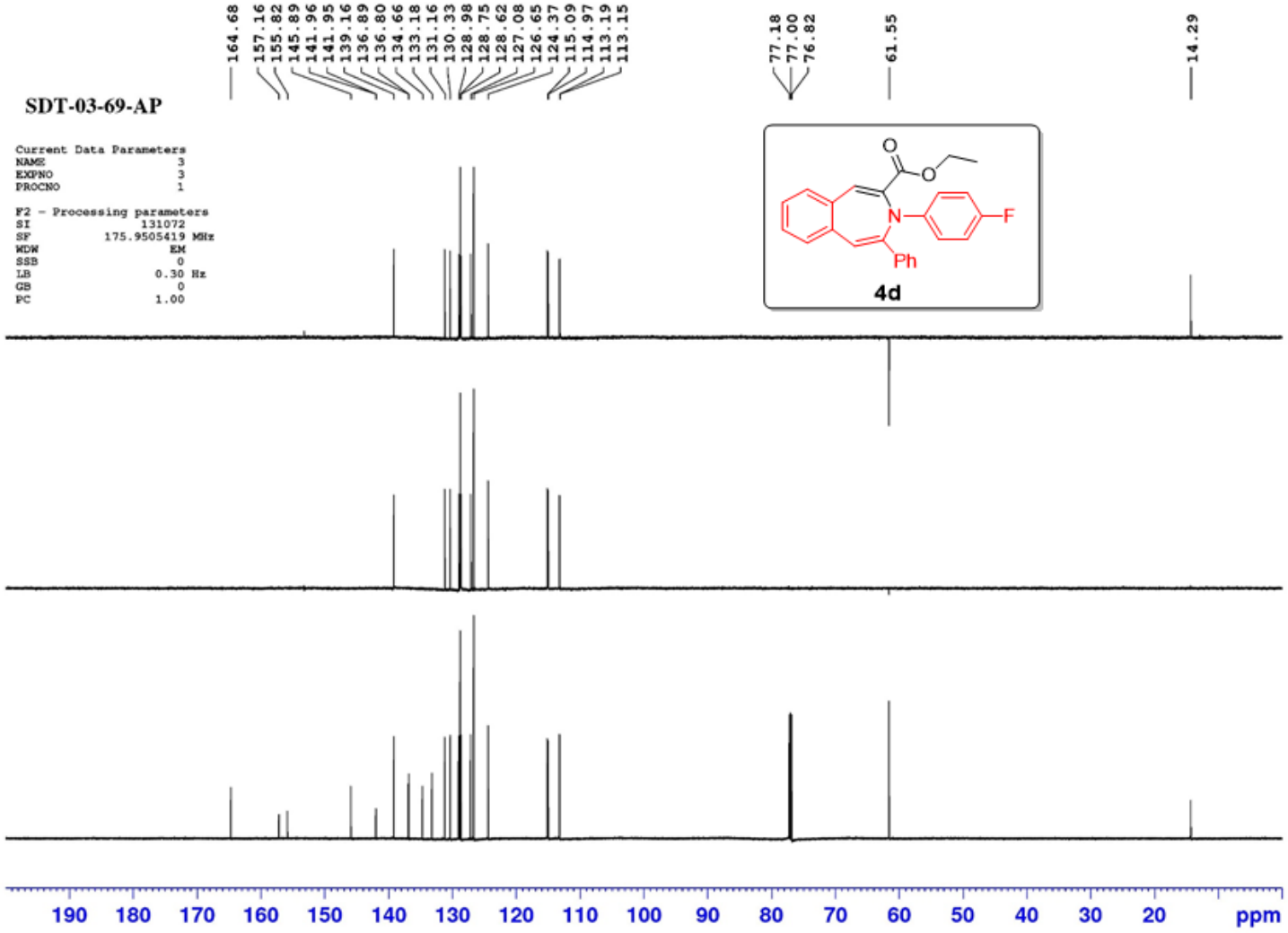
```
Current Data Parameters
NAME      SDT-03-109-AP-H.fid
EXPNO                       1
PROCNO                      1

F2 - Processing parameters
SI                      65536
SF             699.7431026 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



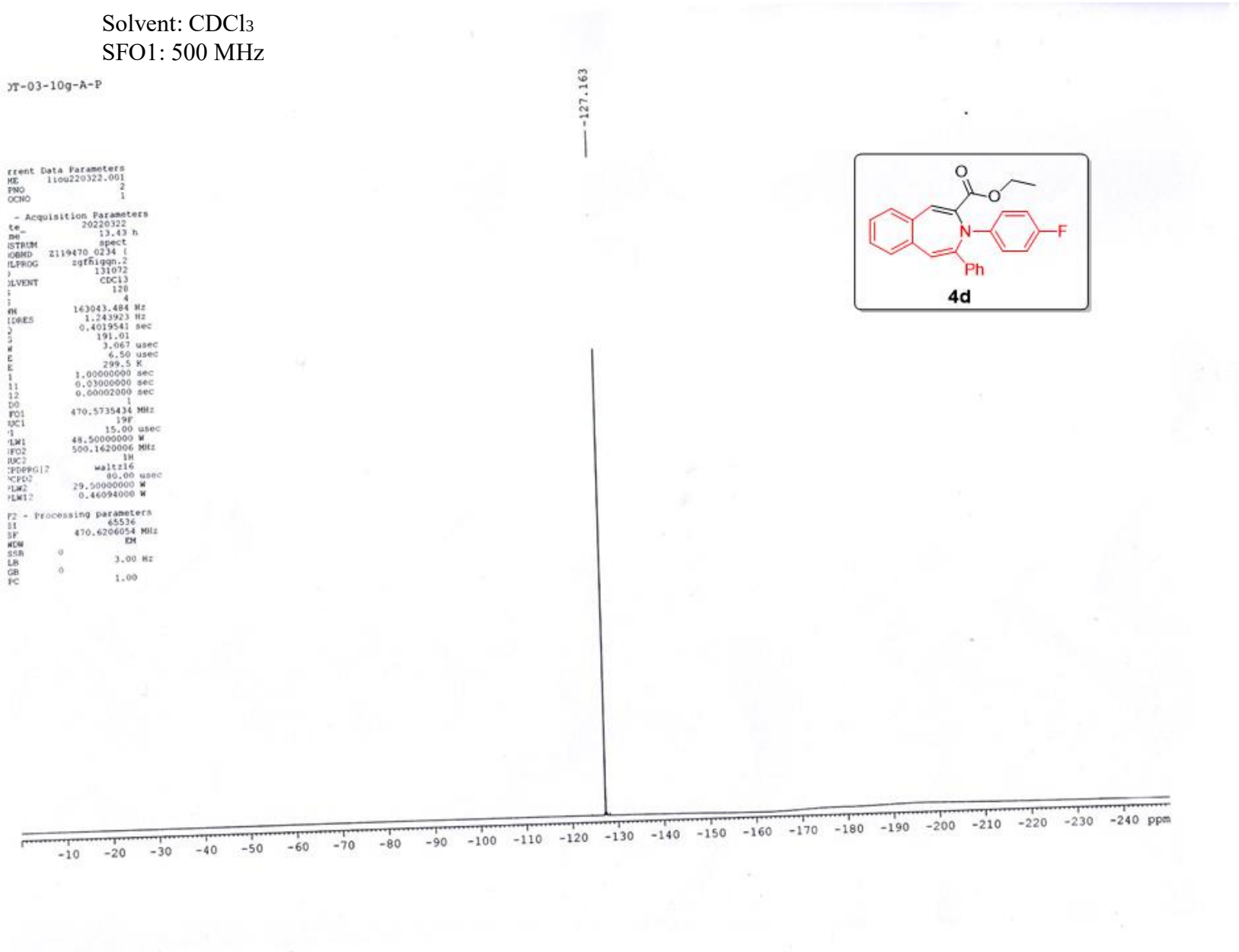
4d



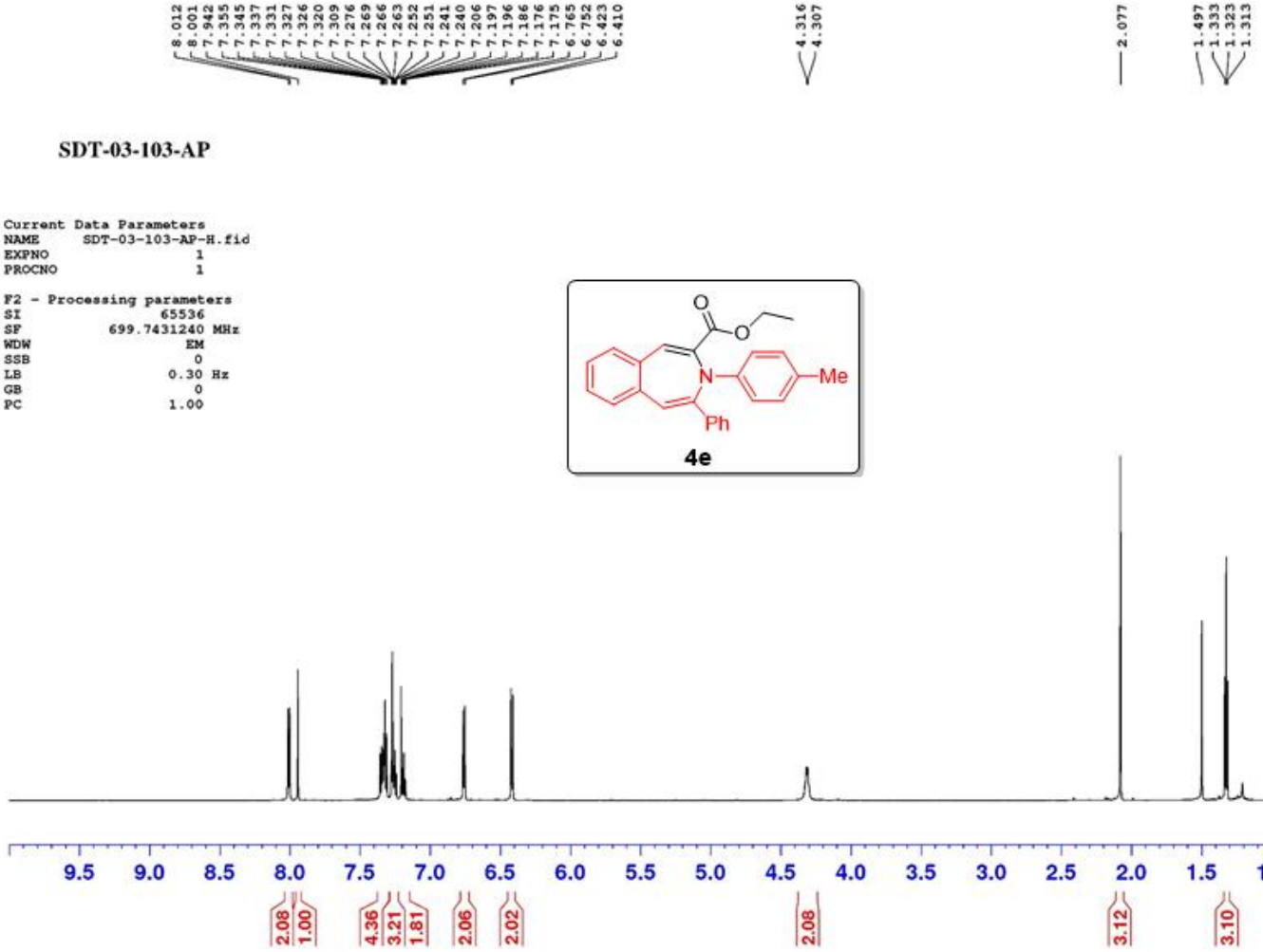
Solvent: $CDCl_3$
SFO1: 175 MHz



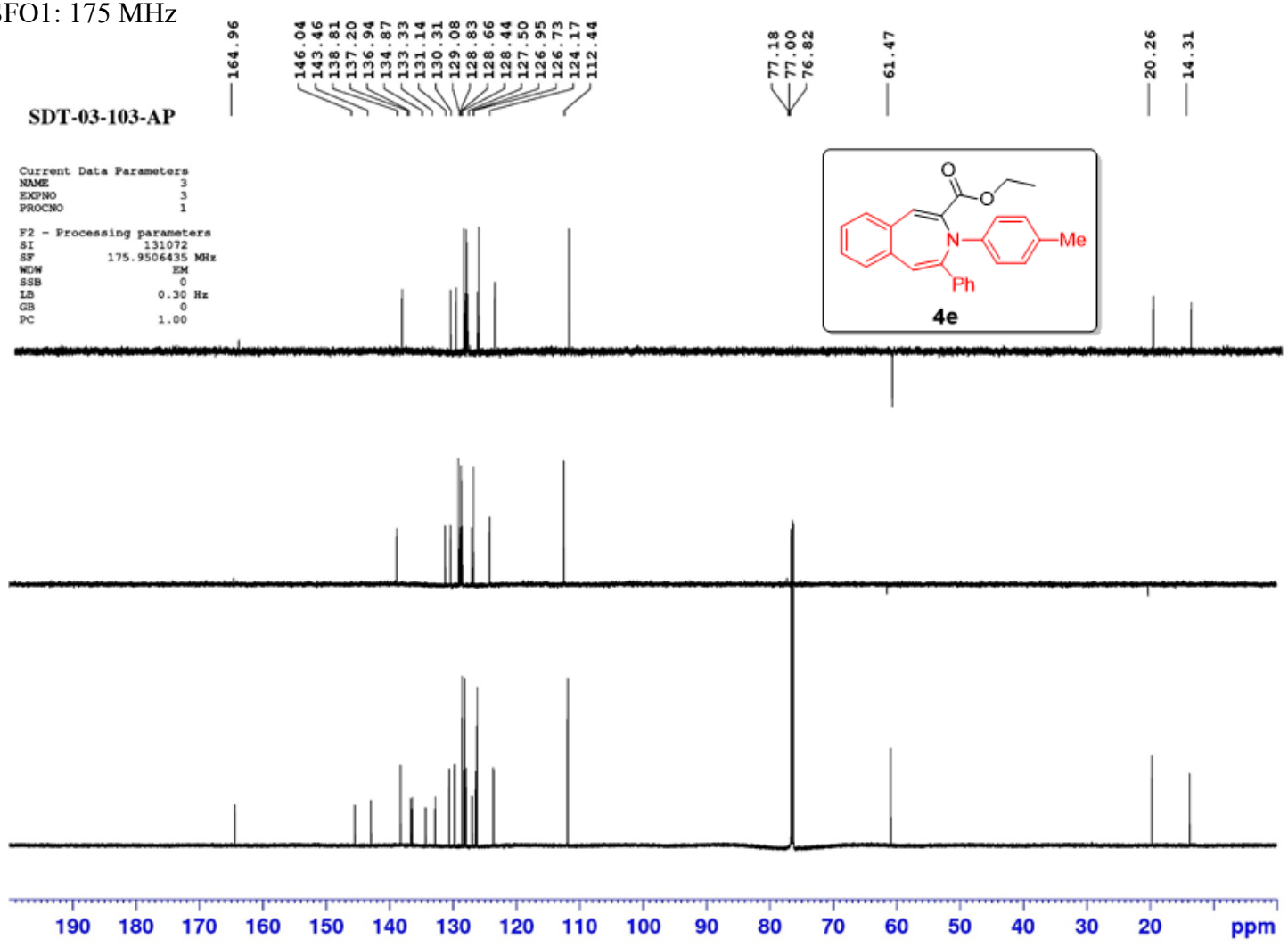
Solvent: $CDCl_3$
SFO1: 500 MHz



Solvent: $CDCl_3$
SFO1: 700 MHz



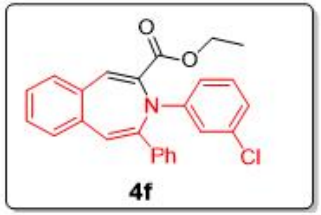
Solvent: $CDCl_3$
SFO1: 175 MHz



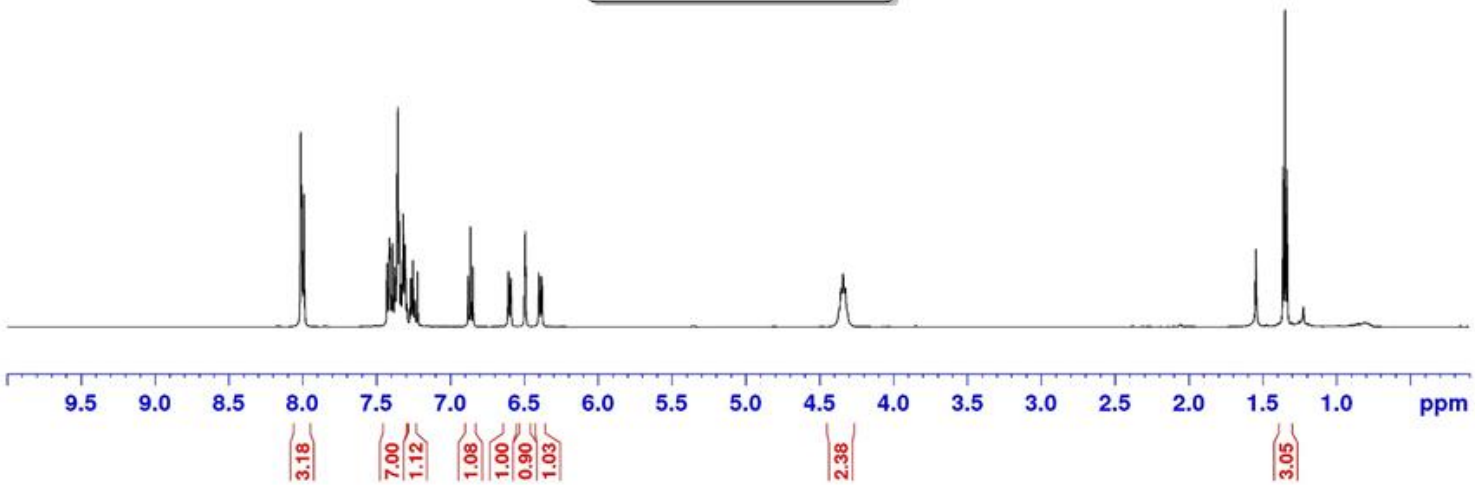
Solvent: $CDCl_3$
SFO1: 500 MHz

SDT-03-112-AP

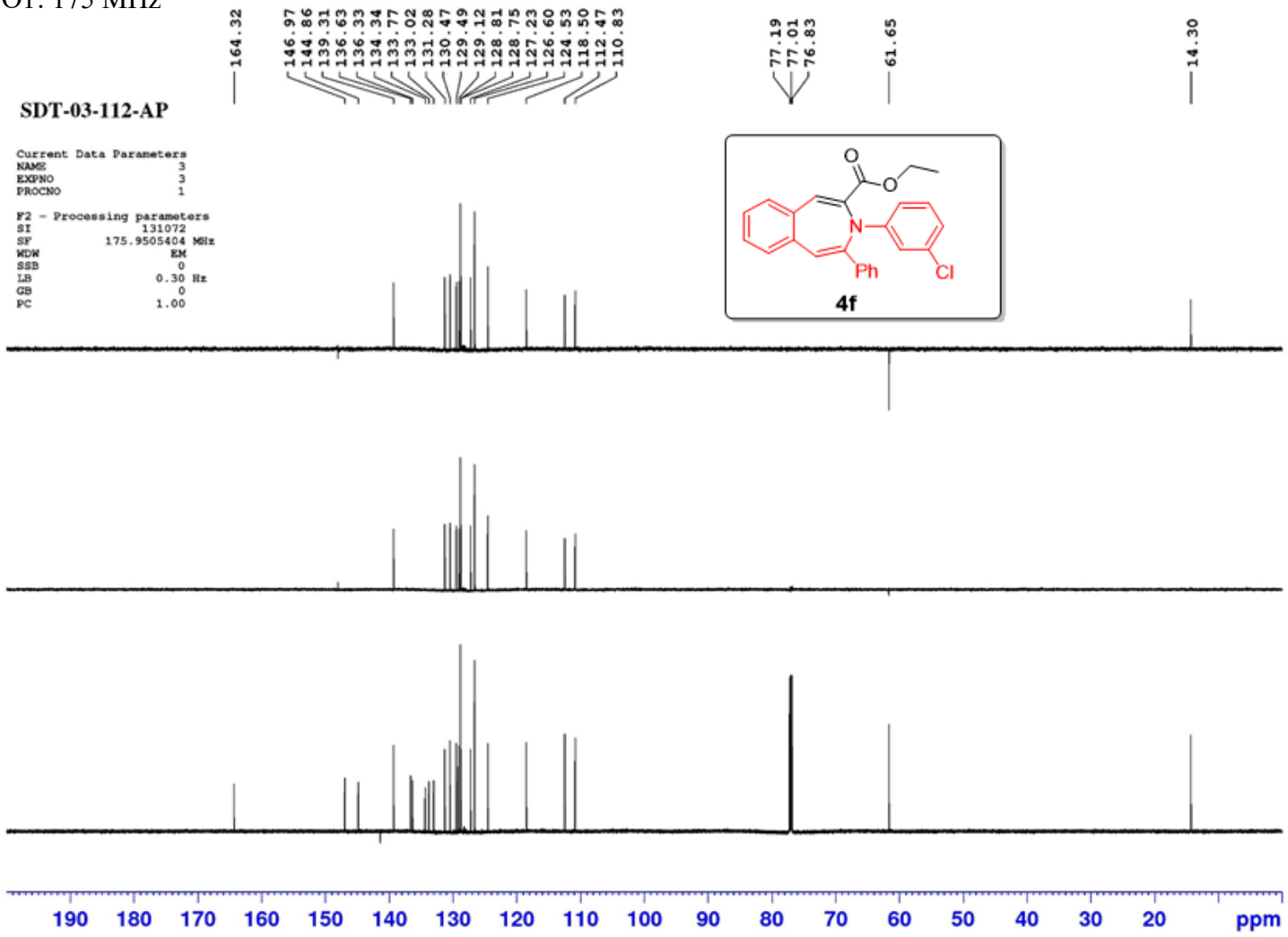
```
Current Data Parameters
NAME      SDT-03-112-Final-AF
EXPNO                       1
PROCNO                      1

F2 - Processing parameters
SI                      32768
SF          499.7727748 MHz
WDW                        EM
SSB                         0
LB                   0.30 Hz
GB                          0
PC                       1.00
```



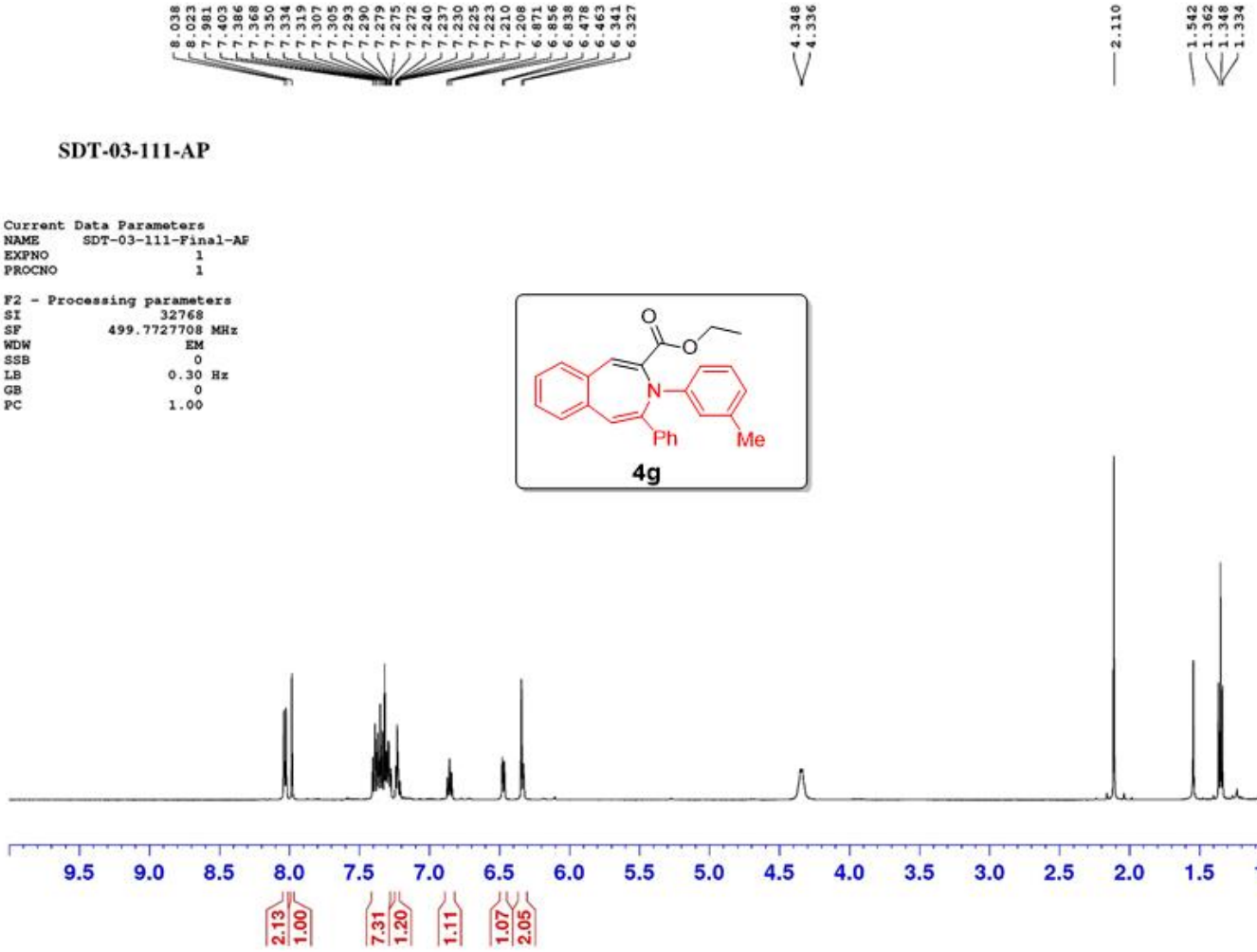
4f



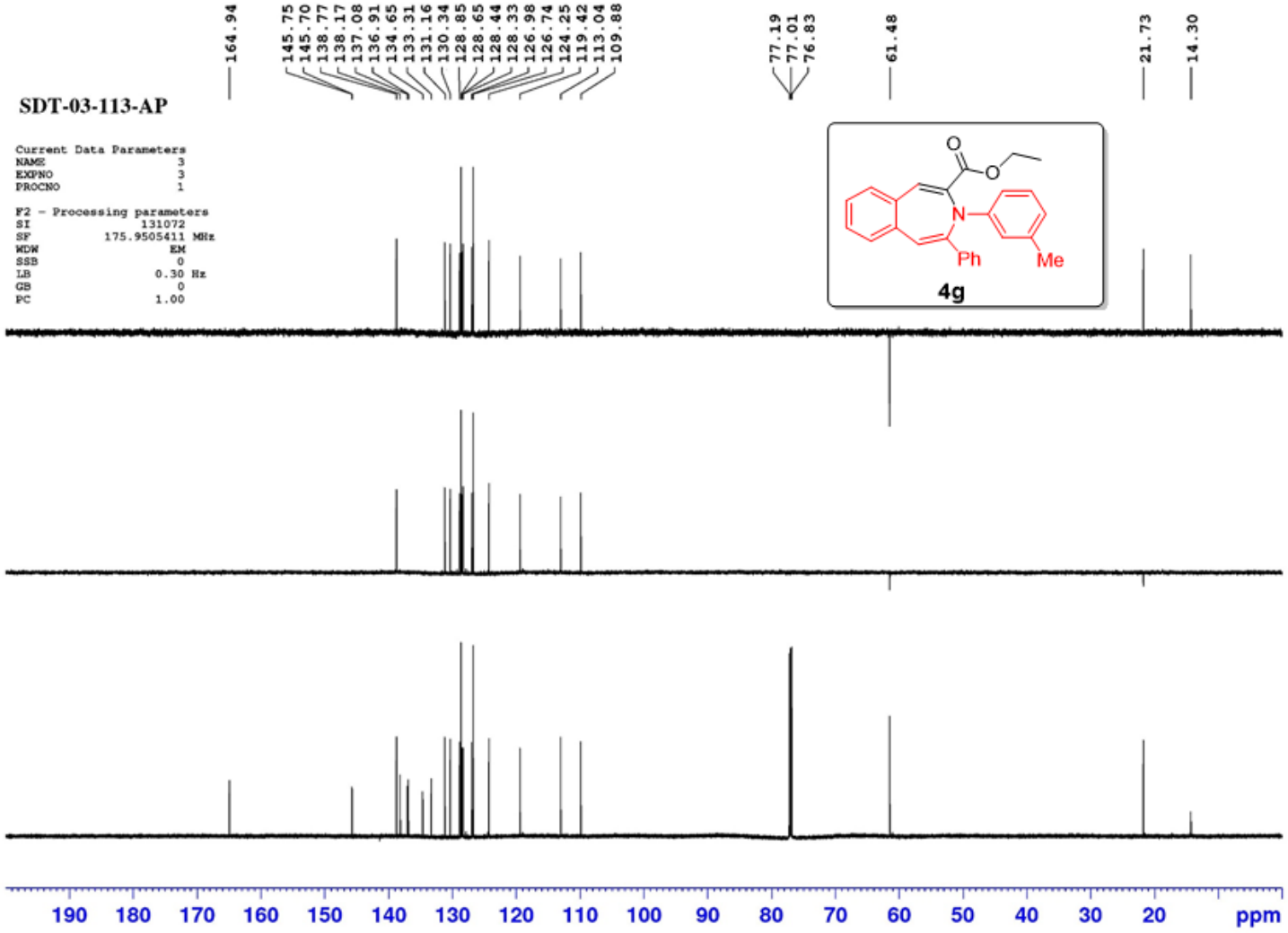
Solvent: $CDCl_3$
SFO1: 175 MHz



Solvent: $CDCl_3$
SFO1: 500 MHz



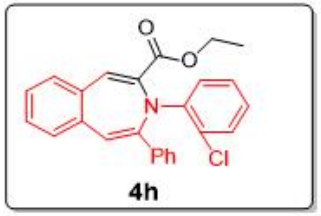
Solvent: $CDCl_3$
SFO1: 175 MHz



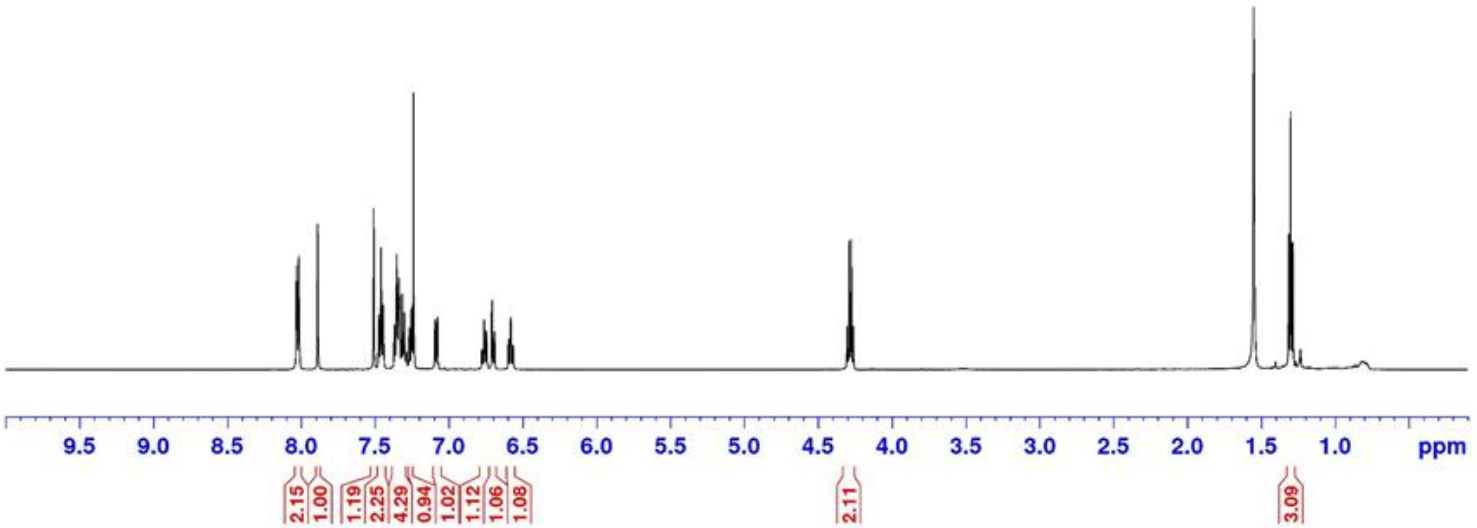
Solvent: $CDCl_3$
SFO1: 500 MHz

SDT-03-113-AP

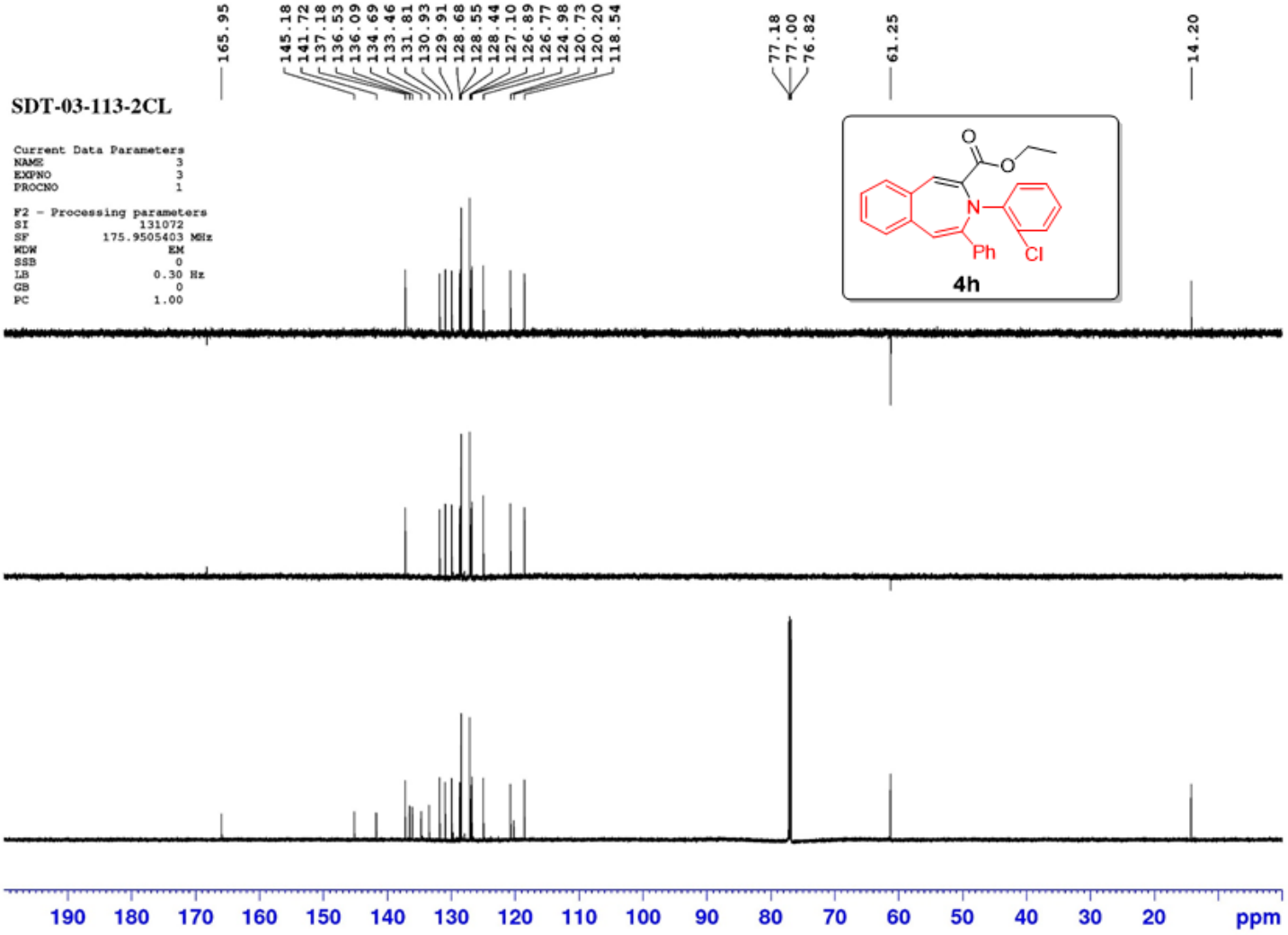
```
Current Data Parameters
NAME      SDT-03-113-Final-AF
EXPNO                       1
PROCNO                      1

F2 - Processing parameters
SI                      32768
SF          499.7727656 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



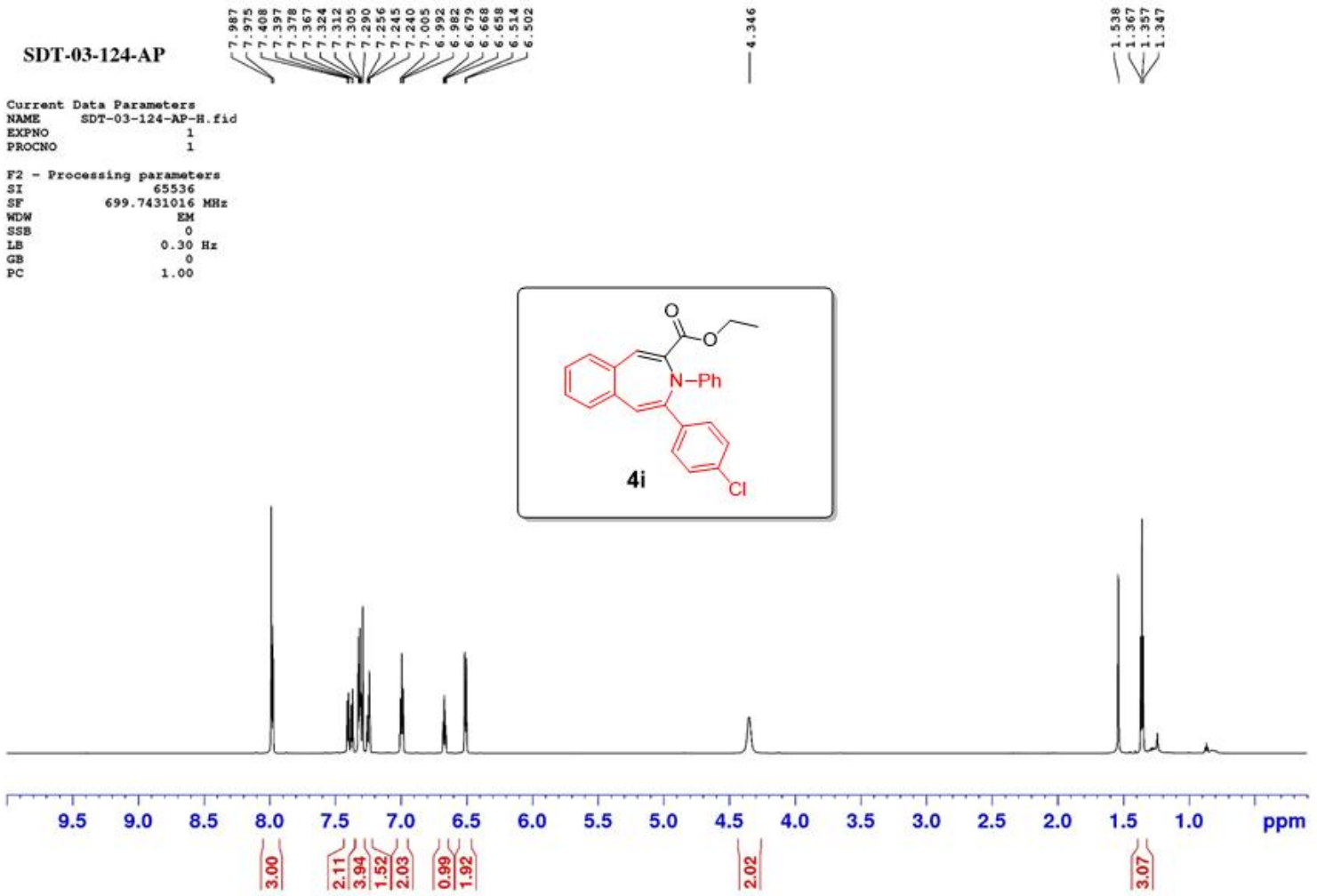
4h



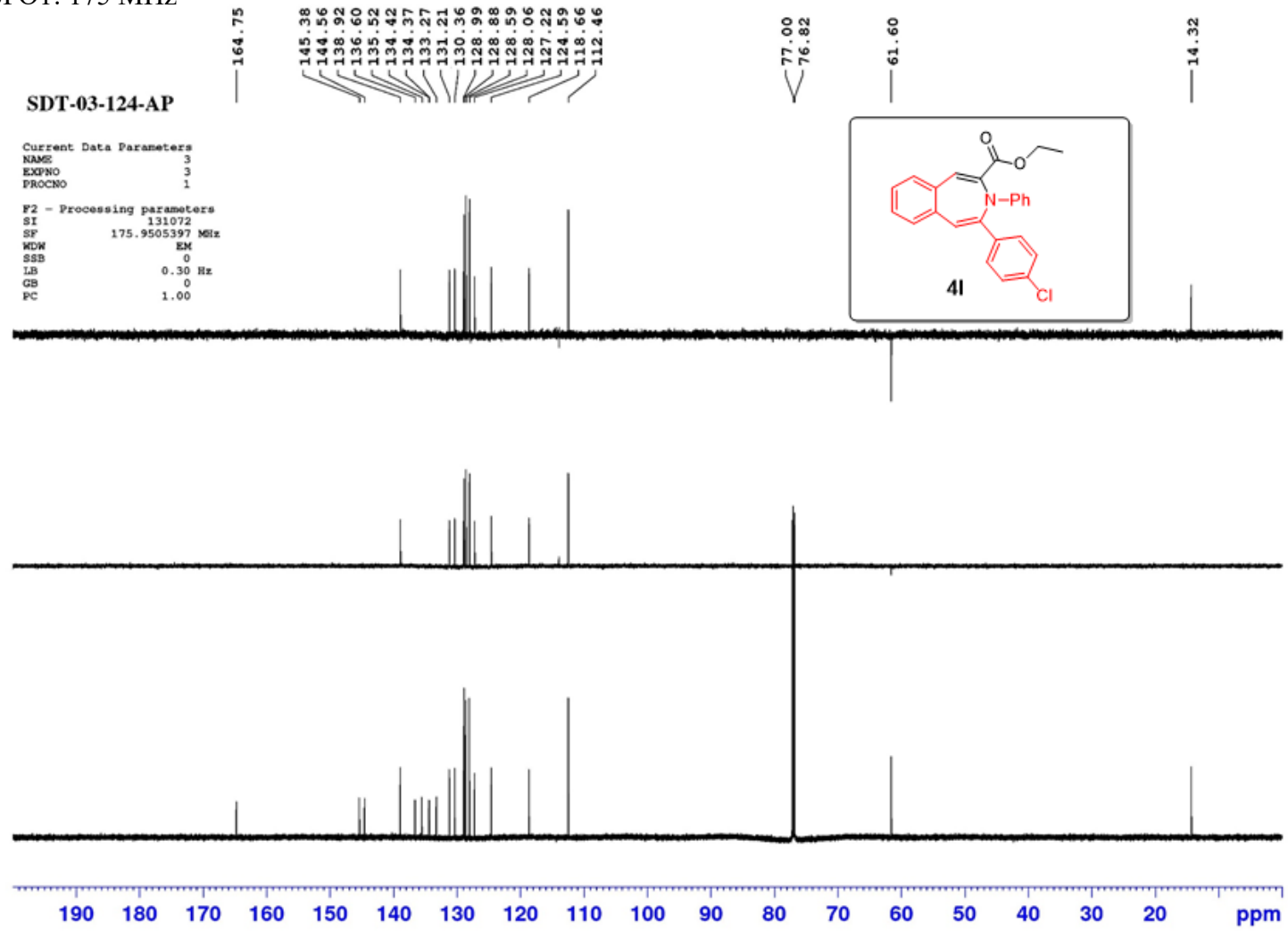
Solvent: $CDCl_3$
SFO1: 175 MHz



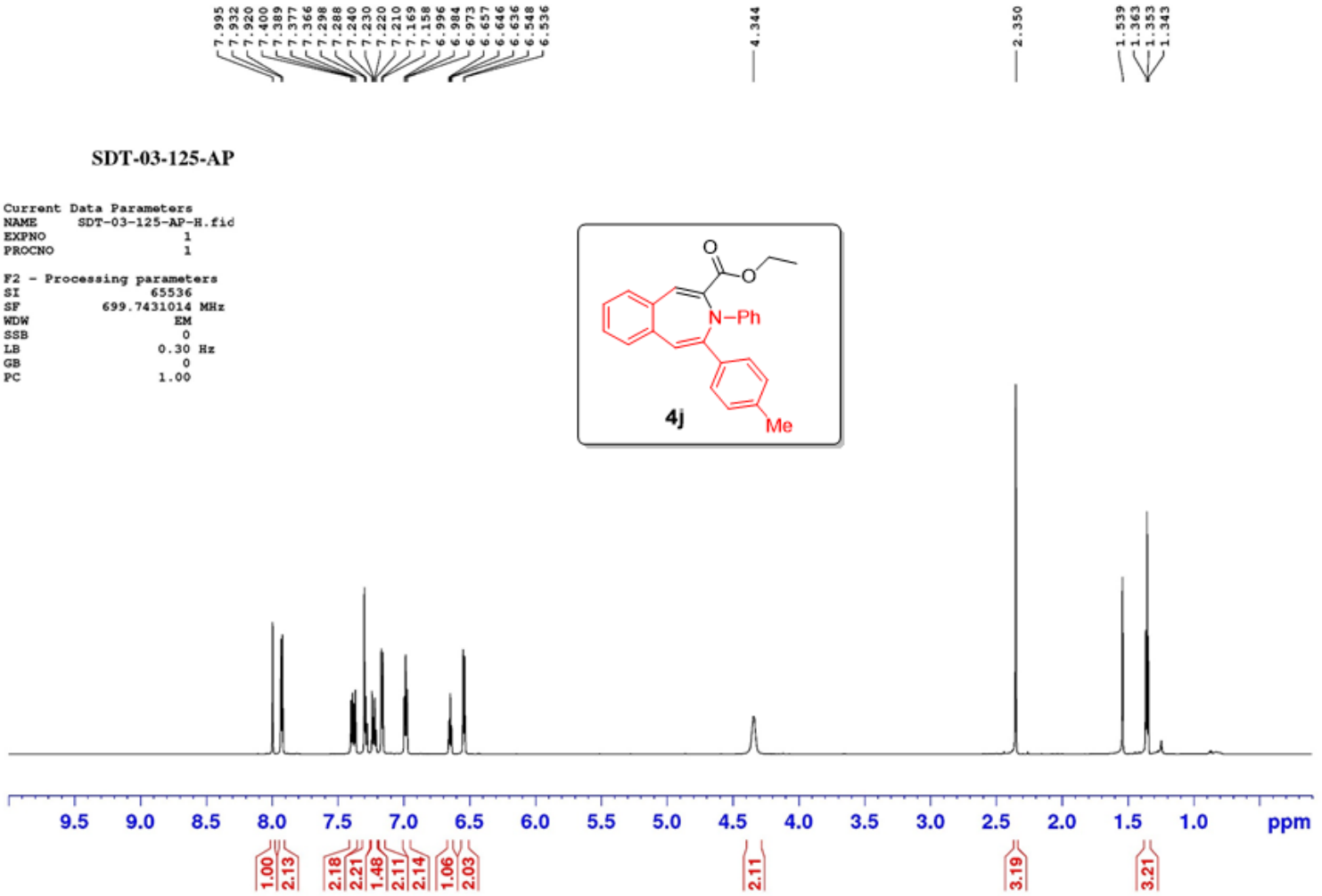
Solvent: $CDCl_3$
SFO1: 700 MHz



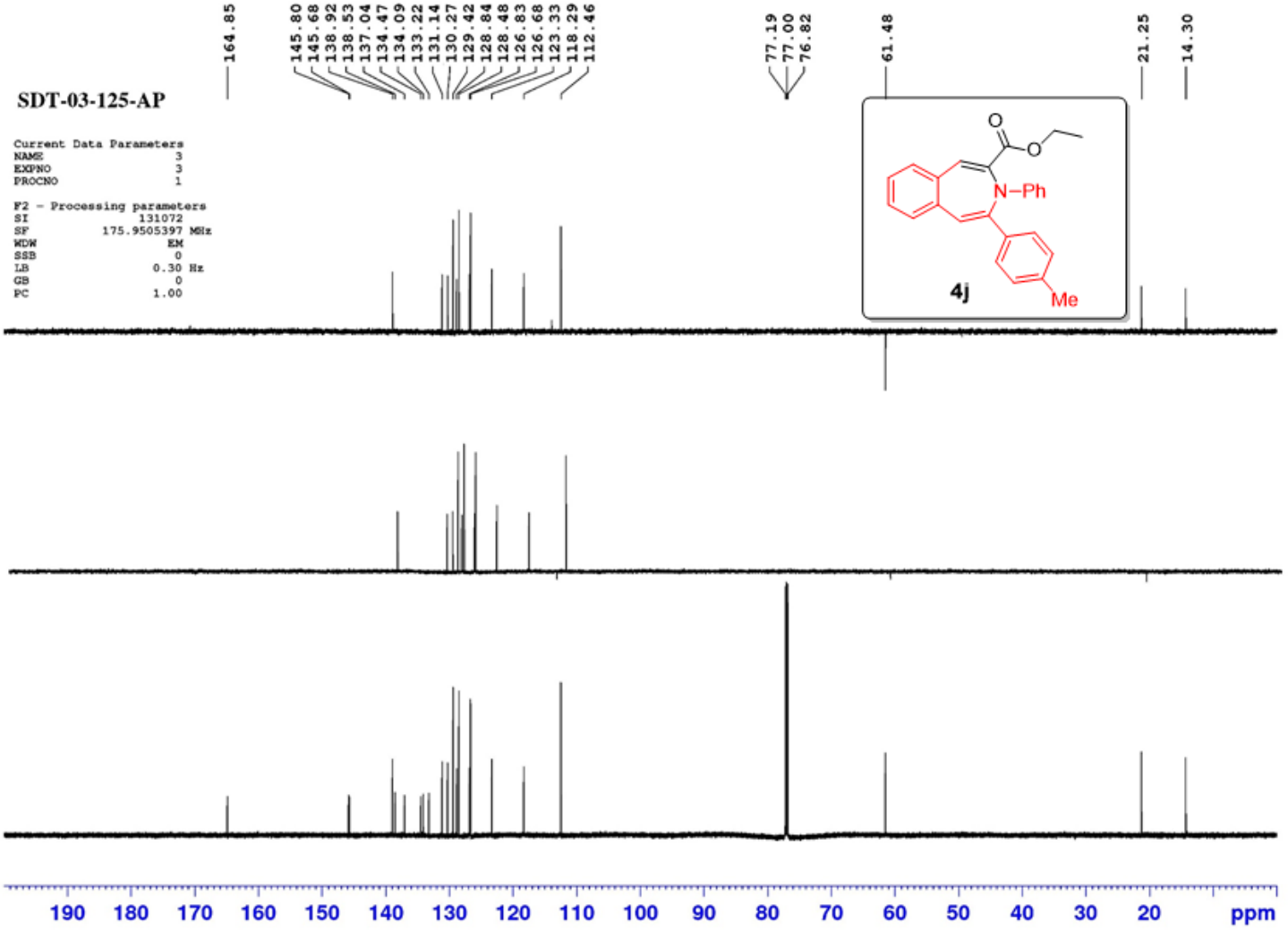
Solvent: $CDCl_3$
SFO1: 175 MHz



Solvent: $CDCl_3$
SFO1: 700 MHz



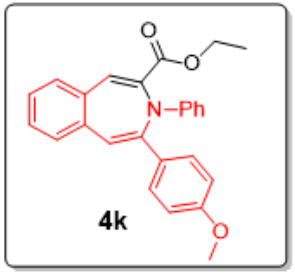
Solvent: $CDCl_3$
SFO1: 175 MHz

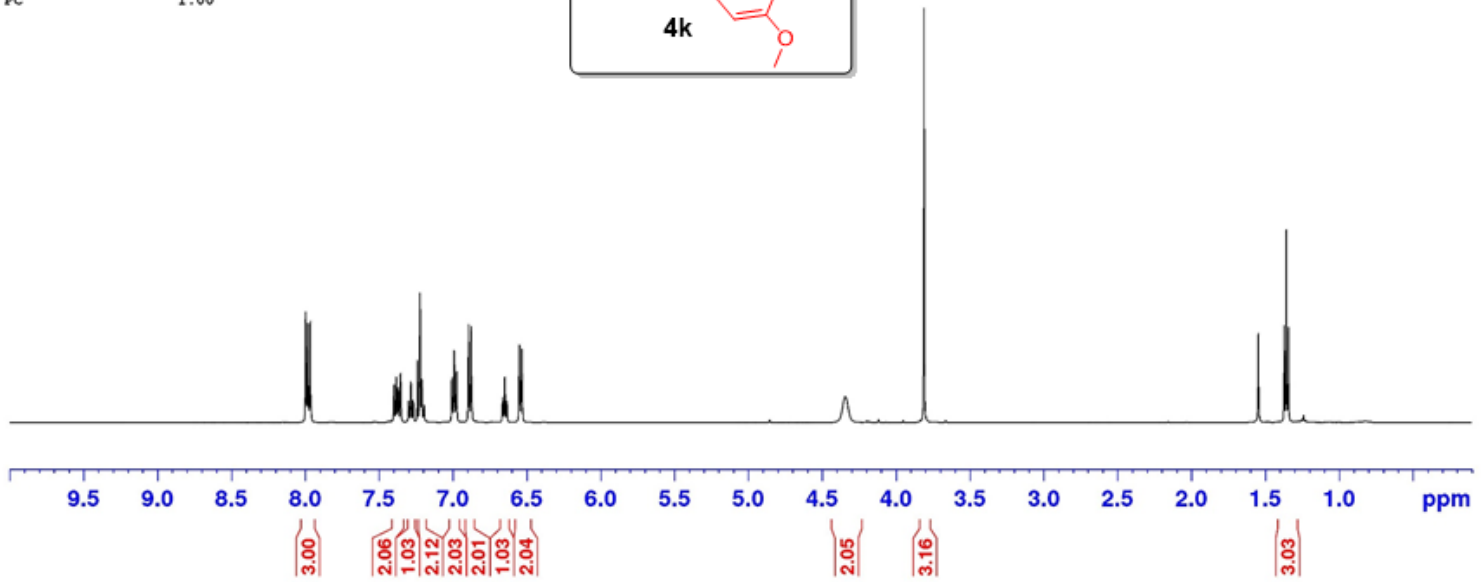


Solvent: $CDCl_3$
SFO1: 500 MHz

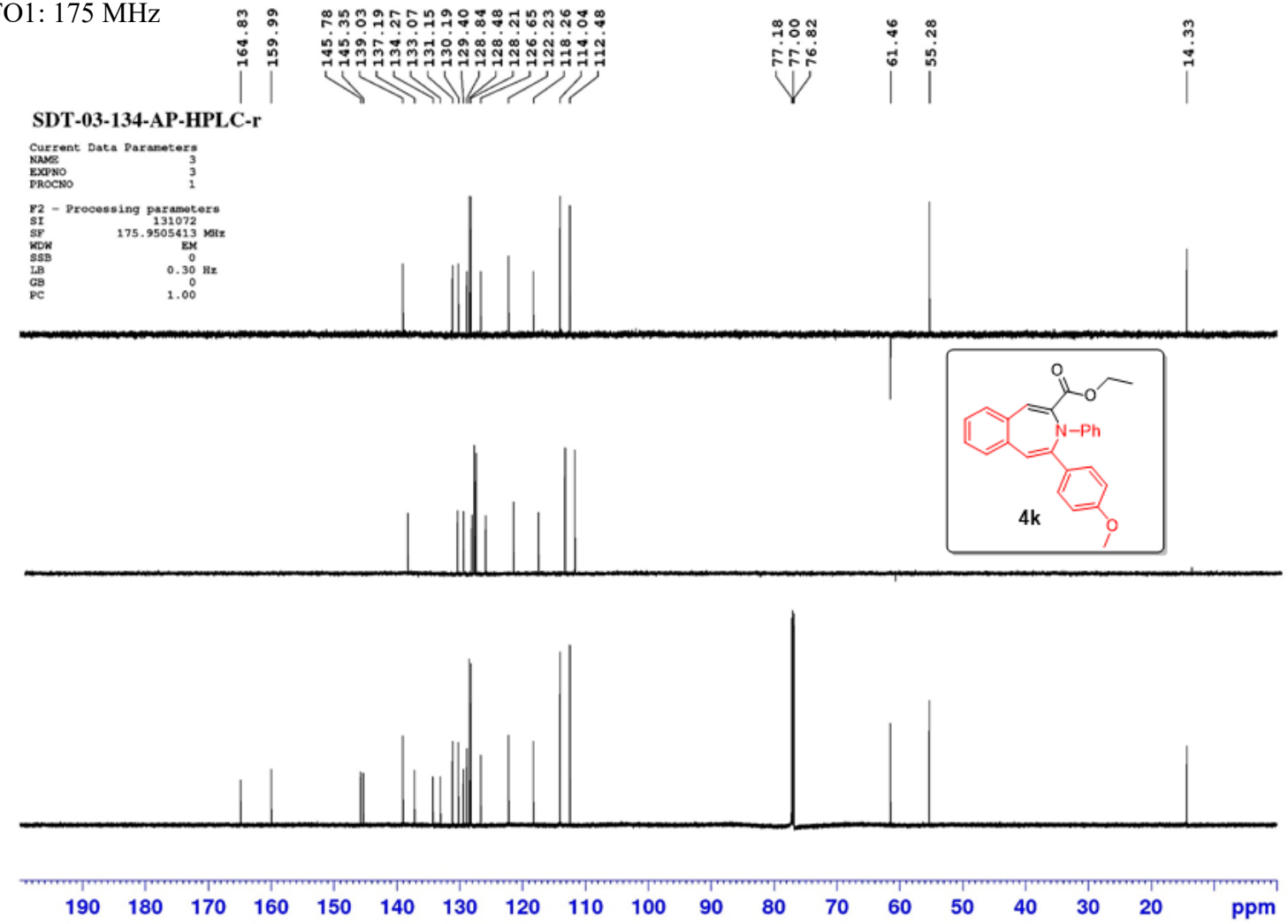
SDT-03-134-AP

```
Current Data Parameters
NAME      SDT-03-134-HPLC-AP.
EXPNO                      1
PROCNO                     1

F2 - Processing parameters
SI                     32768
SF           499.7727655 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

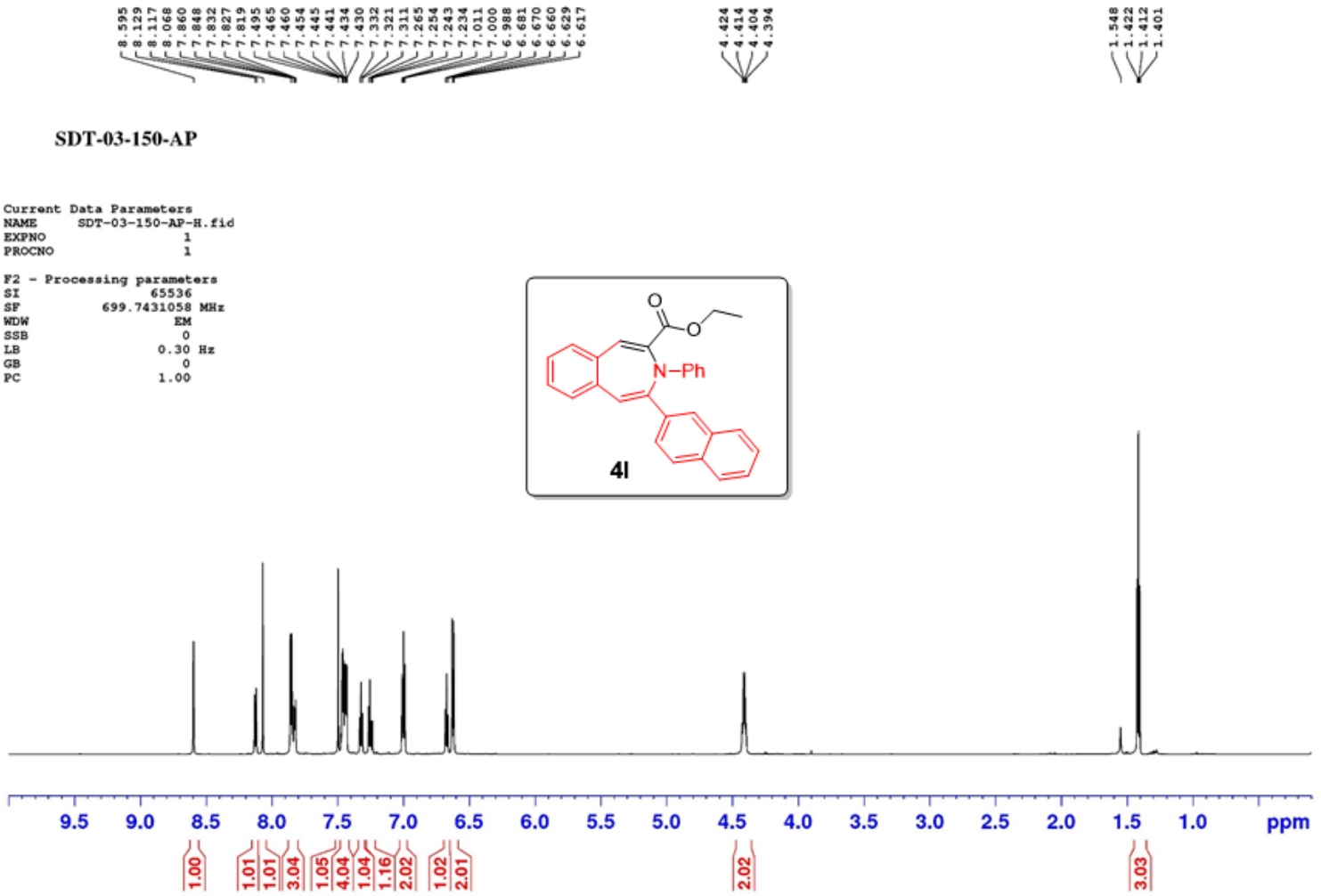




Solvent: $CDCl_3$
SFO1: 175 MHz

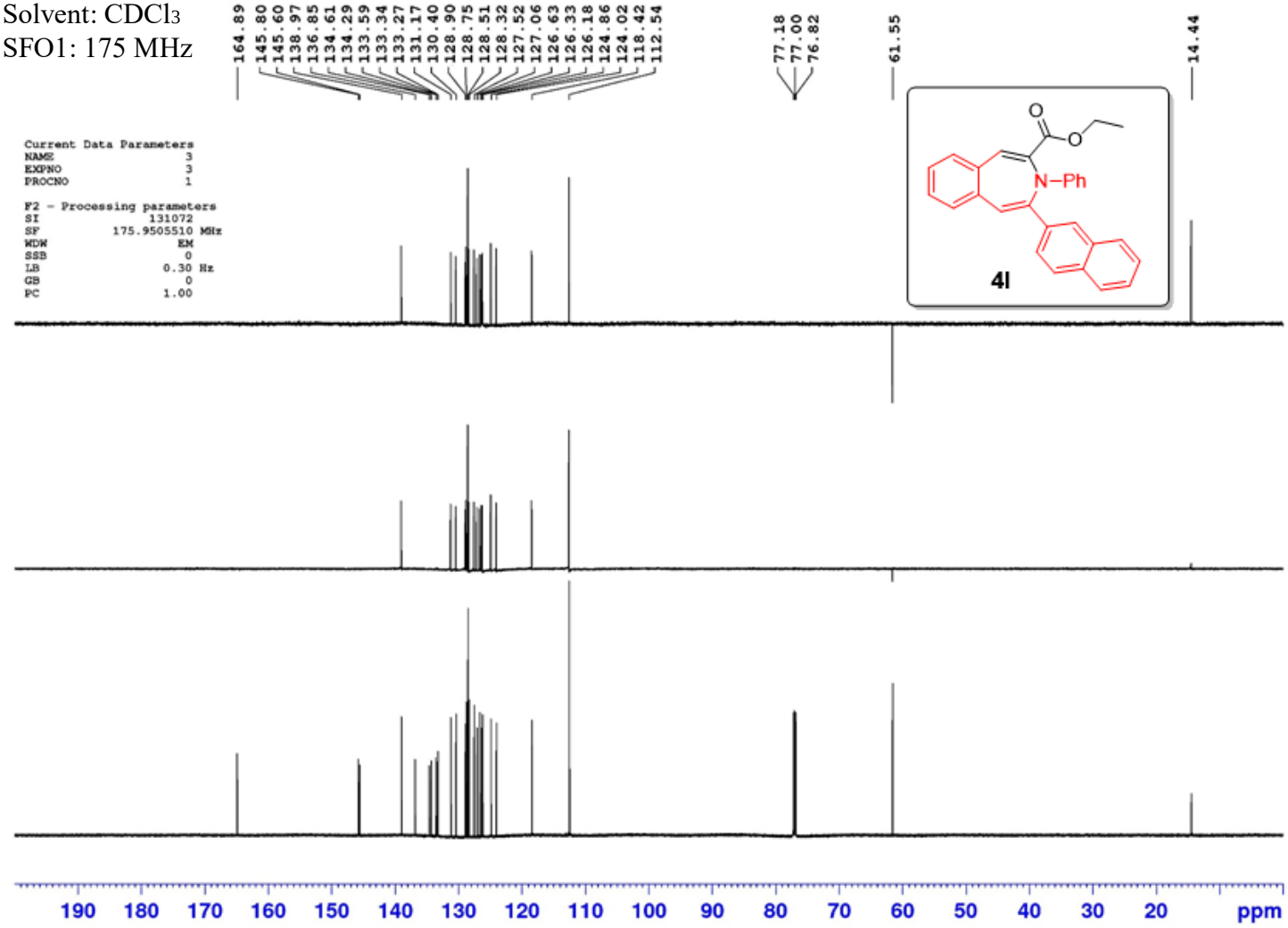


Solvent: $CDCl_3$
SFO1: 700 MHz



SDT-03-150-AP

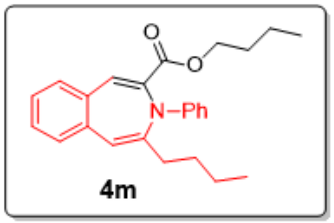
Solvent: $CDCl_3$
SFO1: 175 MHz

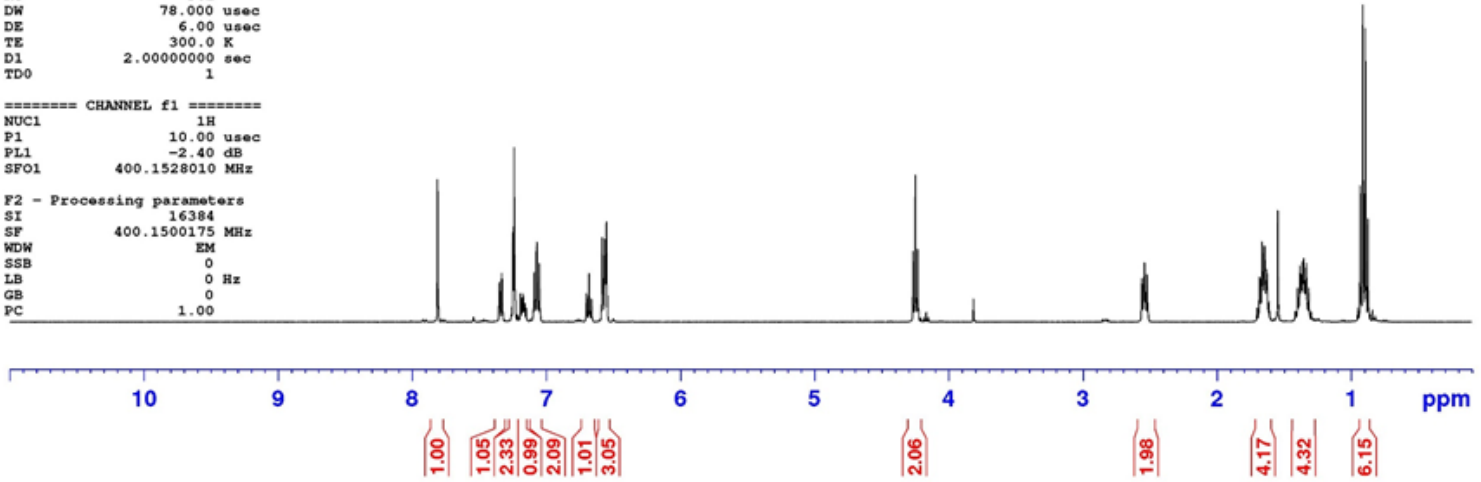


Solvent: $CDCl_3$
SFO1: 400 MHz

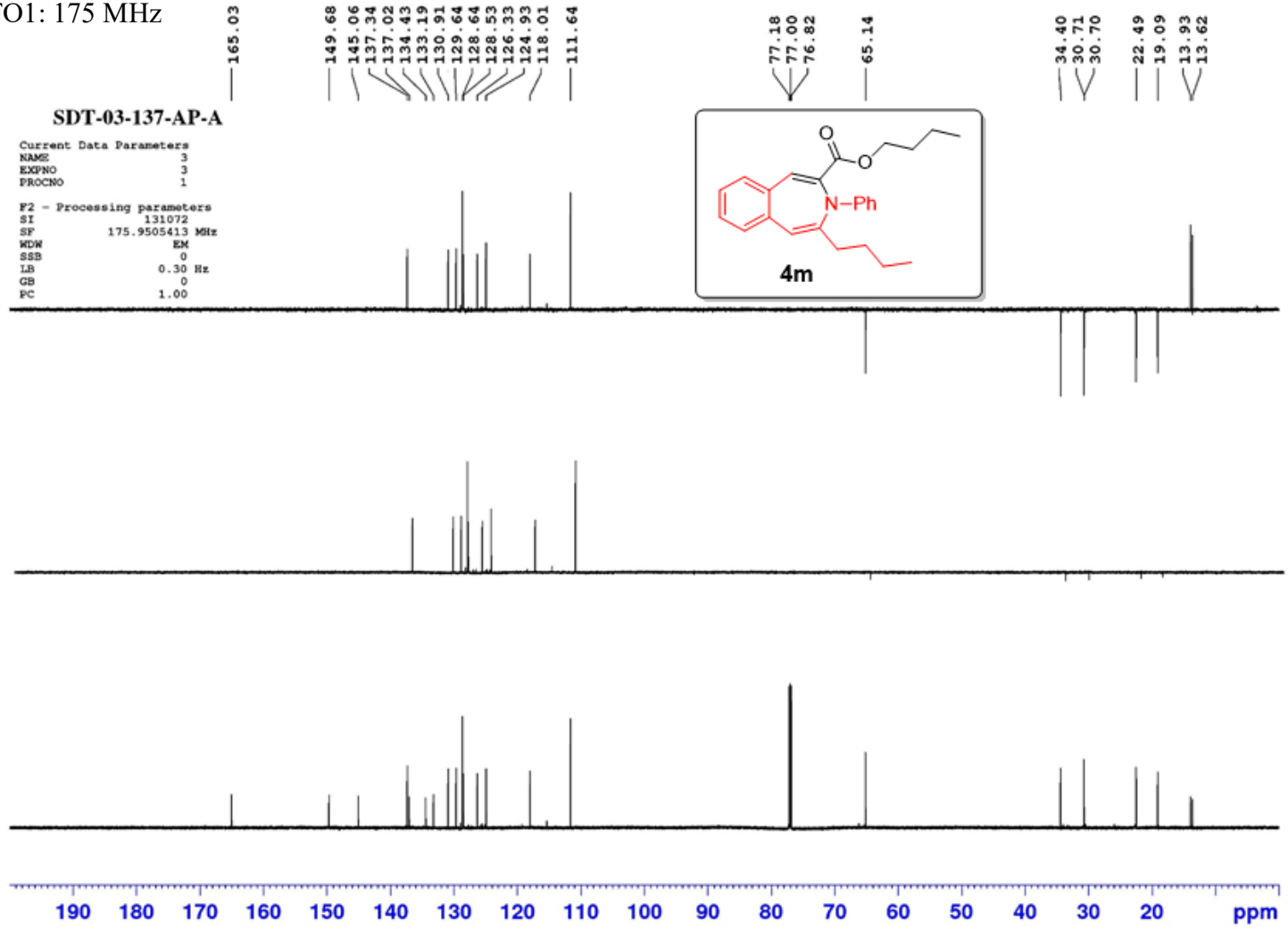
SDT-03-137-AP-A

```
Current Data Parameters
NAME      SDT-03-137-AP-A
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20220422
Time              22.48
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     6
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ              2.5559039 sec
RG                   362
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           400.1500175 MHz
WDW                   EM
SSB                    0
LB                     0 Hz
GB                     0
PC                  1.00
```

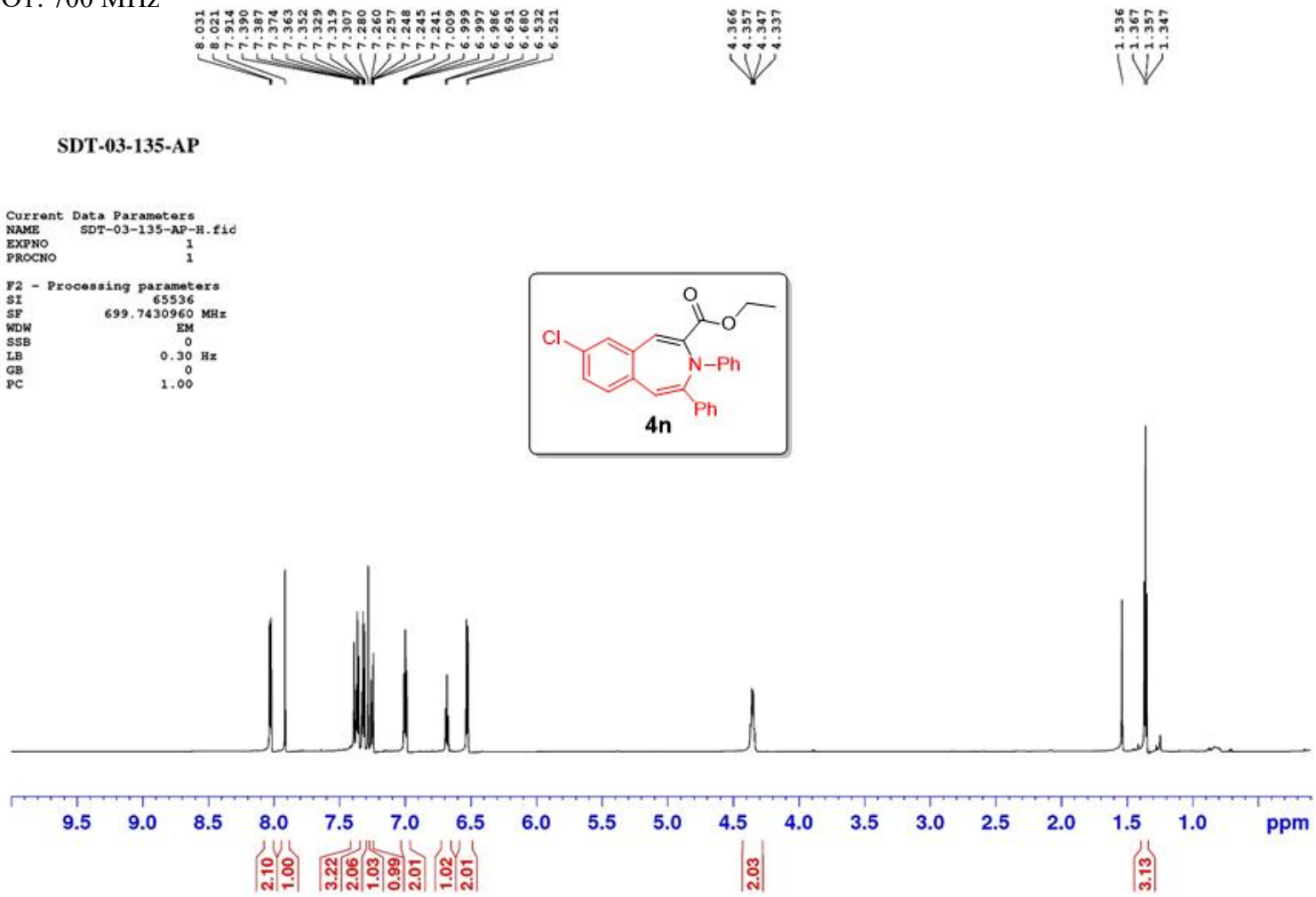




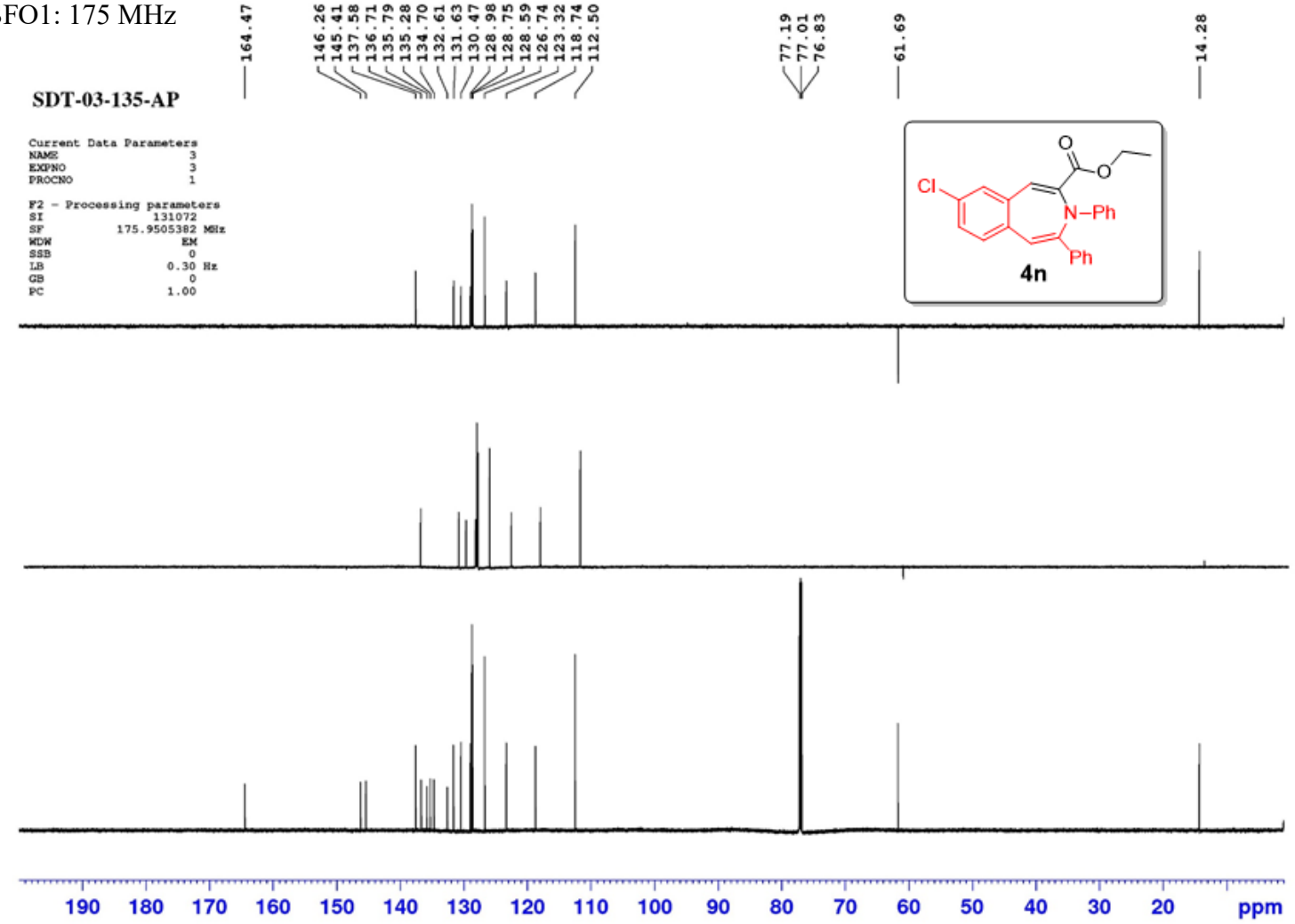
Solvent: $CDCl_3$
SFO1: 175 MHz



Solvent: $CDCl_3$
SFO1: 700 MHz



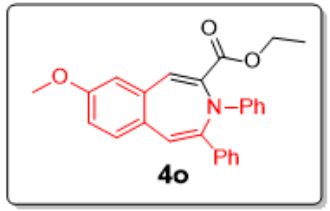
Solvent: $CDCl_3$
SFO1: 175 MHz



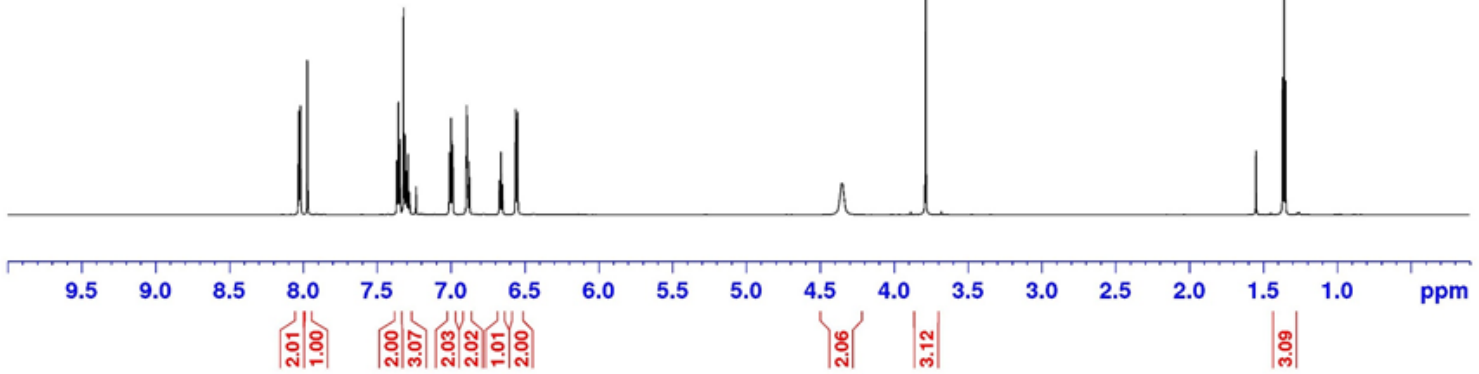
Solvent: $CDCl_3$
SFO1: 700 MHz

SDT-03-148-AP-r

```
Current Data Parameters
NAME      SDT-03-148-AP-r-H.f
EXPNO                    1
PROCNO                   1

F2 - Processing parameters
SI                   65536
SF           699.7431048 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

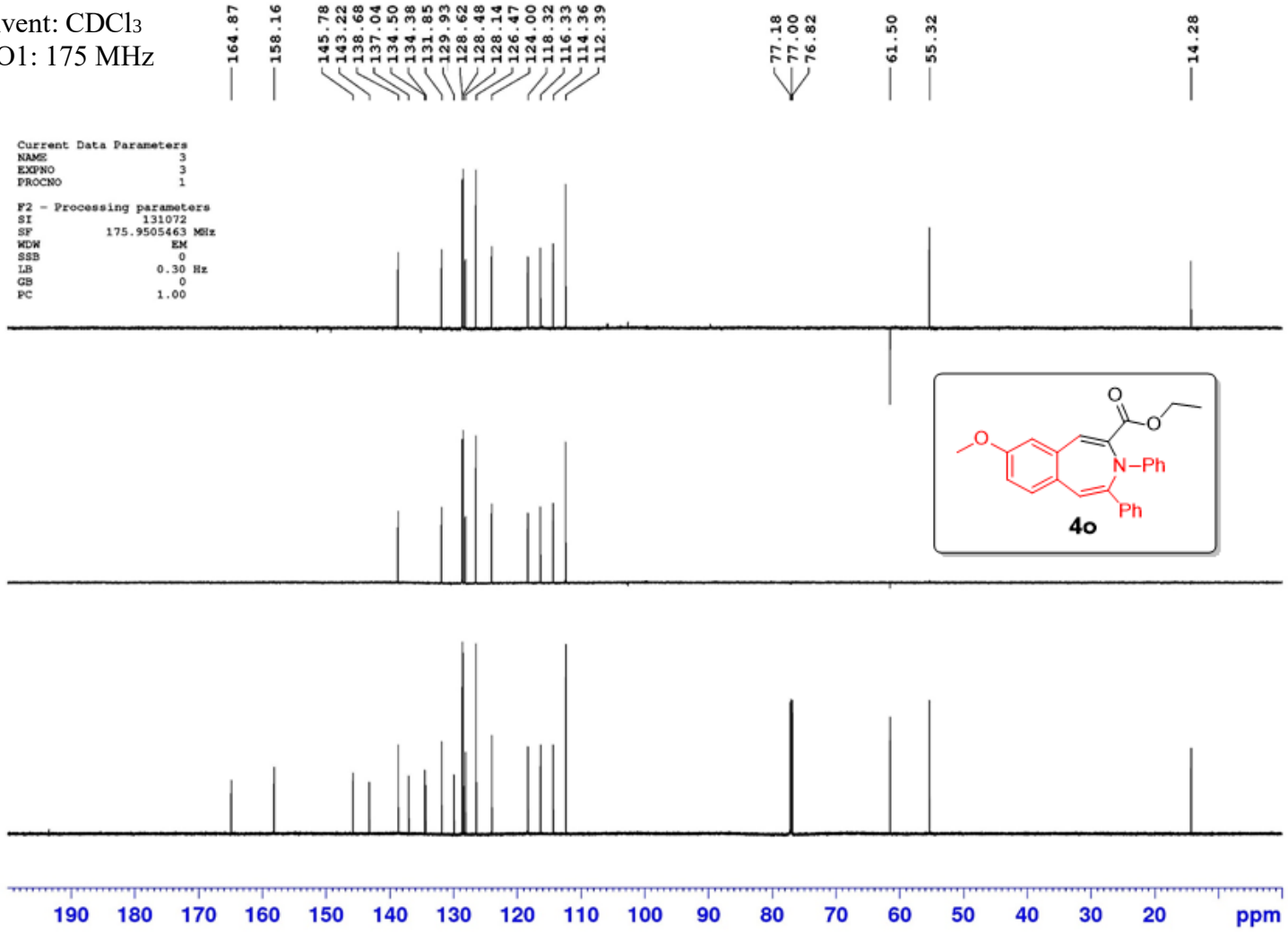


4o



SDT-03-148-AP-r

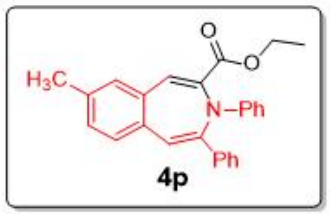
Solvent: $CDCl_3$
SFO1: 175 MHz



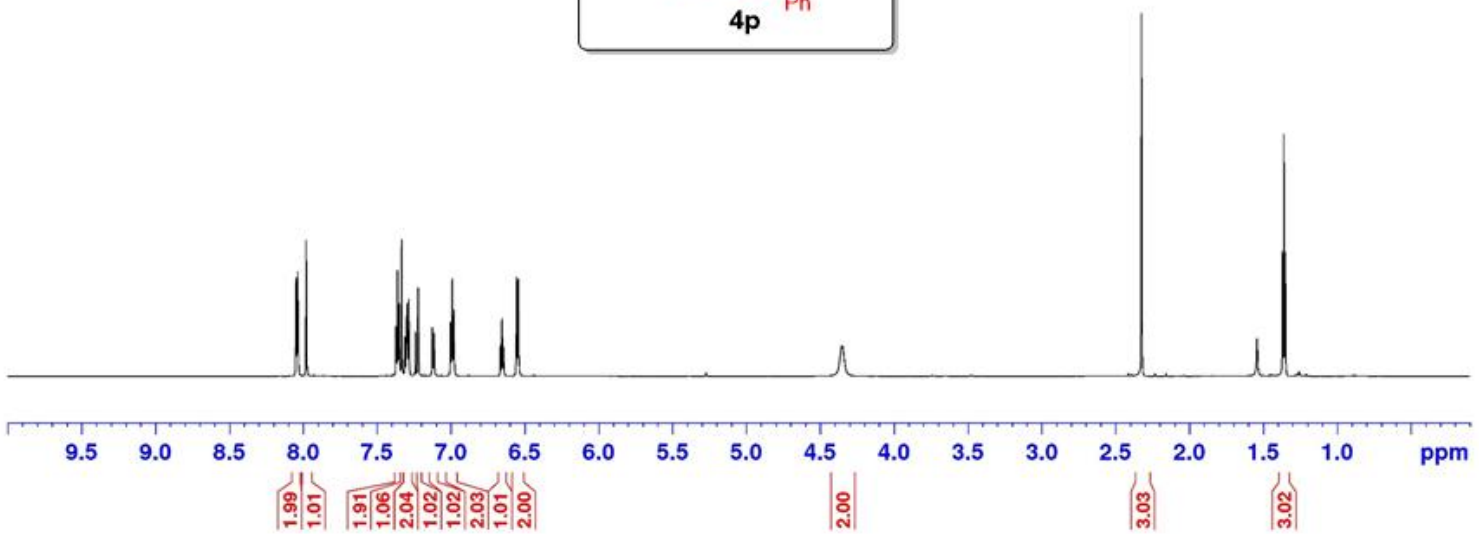
Solvent: $CDCl_3$
SFO1: 700 MHz

SDT-03-168-AP

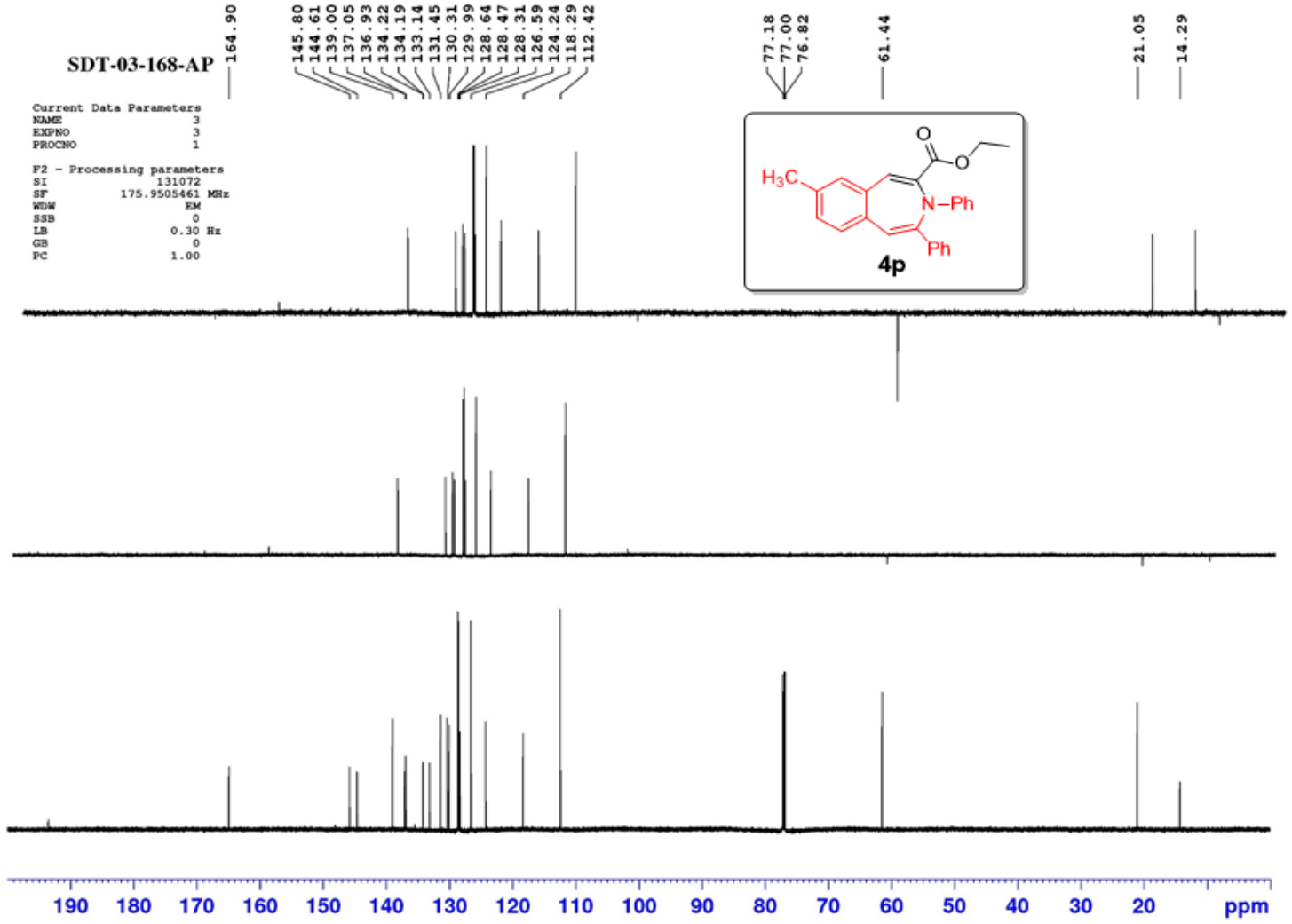
```
Current Data Parameters
NAME      SDT-03-168-AP-H.fid
EXPNO                      1
PROCNO                     1

F2 - Processing parameters
SI                     65536
SF           699.7431023 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



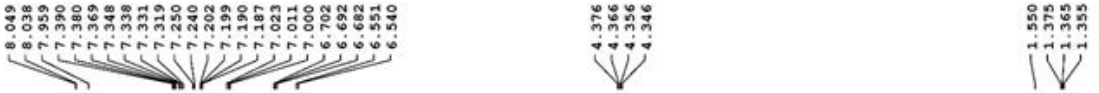
4p



Solvent: $CDCl_3$
SFO1: 175 MHz

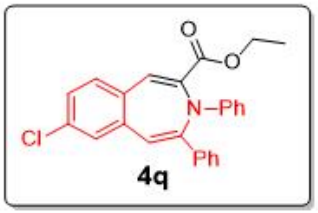


Solvent: $CDCl_3$
SFO1: 700 MHz

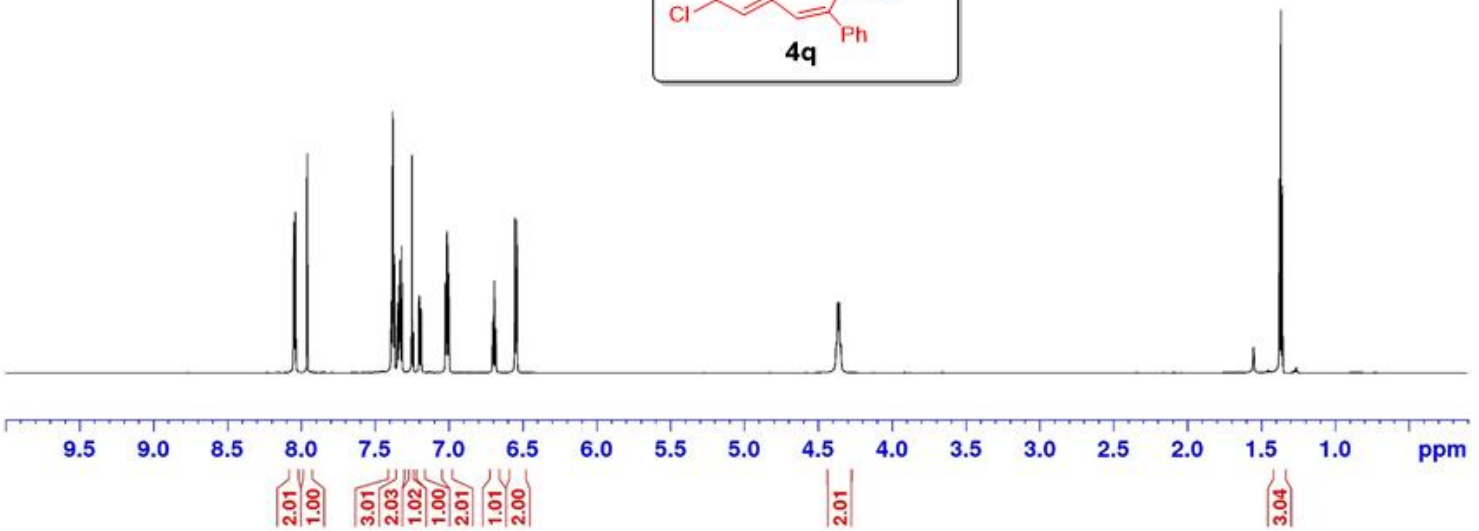


SDT-03-157-AP

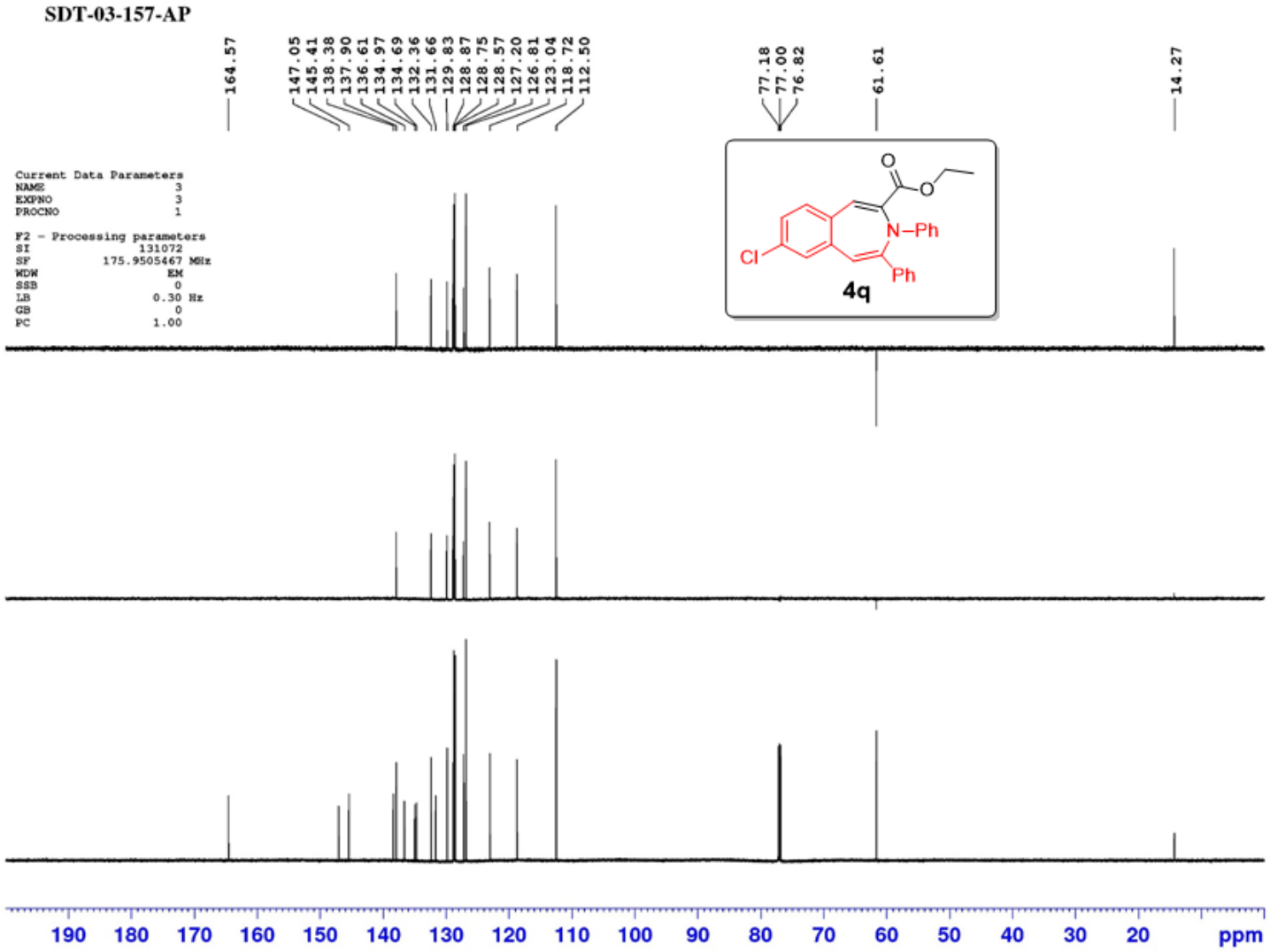
```
Current Data Parameters
NAME      SDT-03-157-AP-H.fid
EXPNO                       1
PROCNO                      1

F2 - Processing parameters
SI                      65536
SF           699.7431019 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



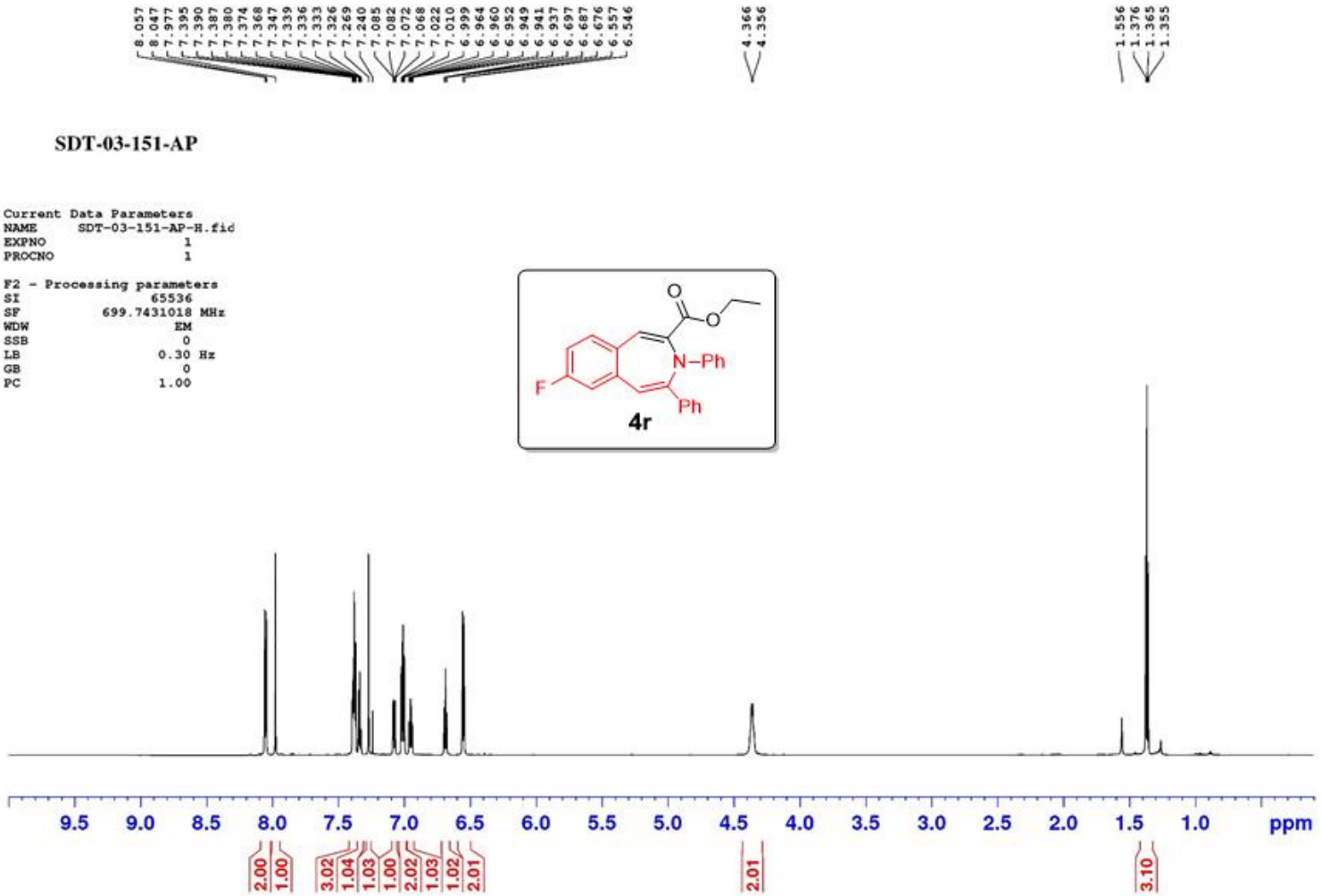
4q



Solvent: $CDCl_3$
SFO1: 175 MHz

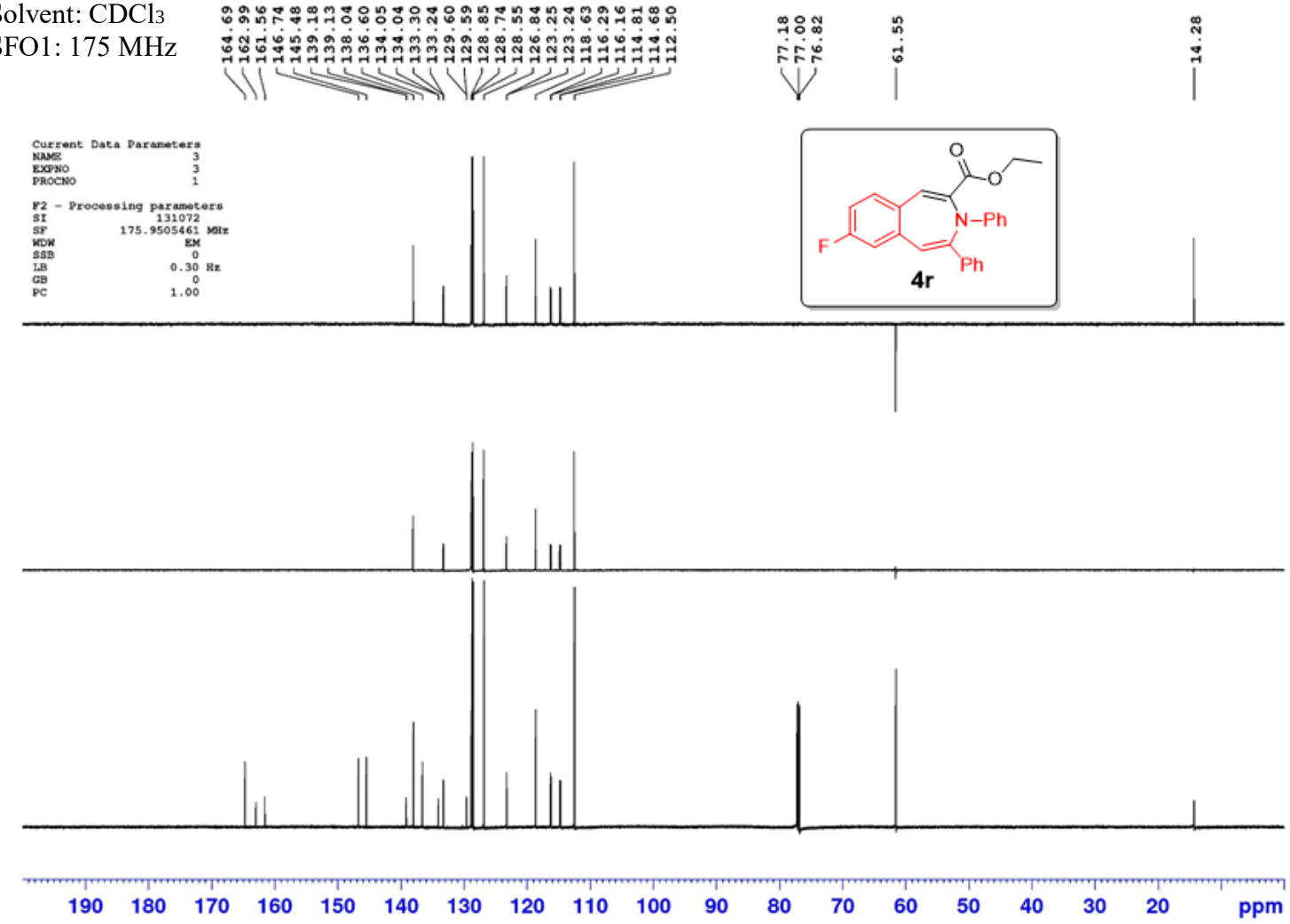


Solvent: $CDCl_3$
SFO1: 700 MHz

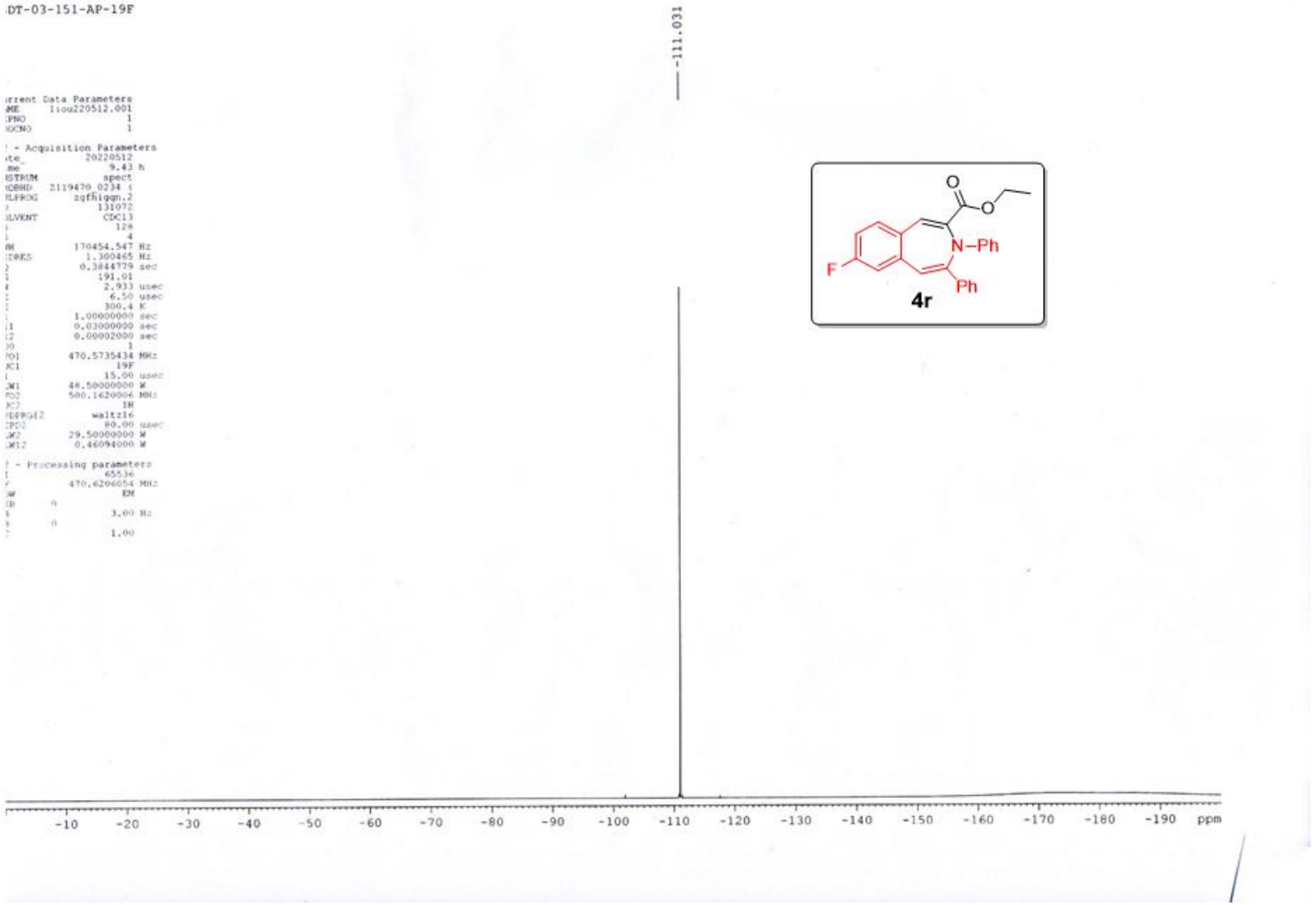


SDT-03-151-AP

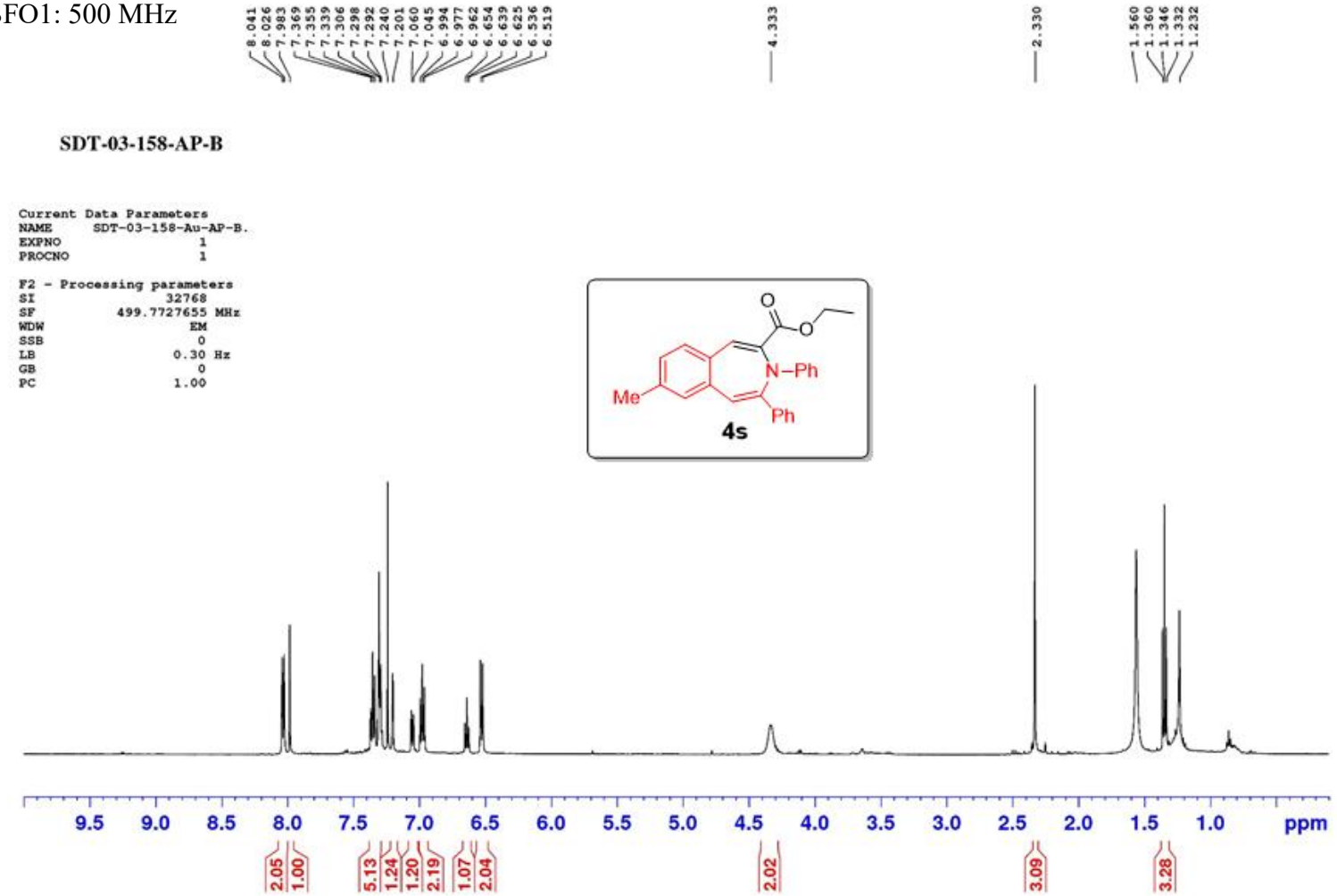
Solvent: $CDCl_3$
SFO1: 175 MHz



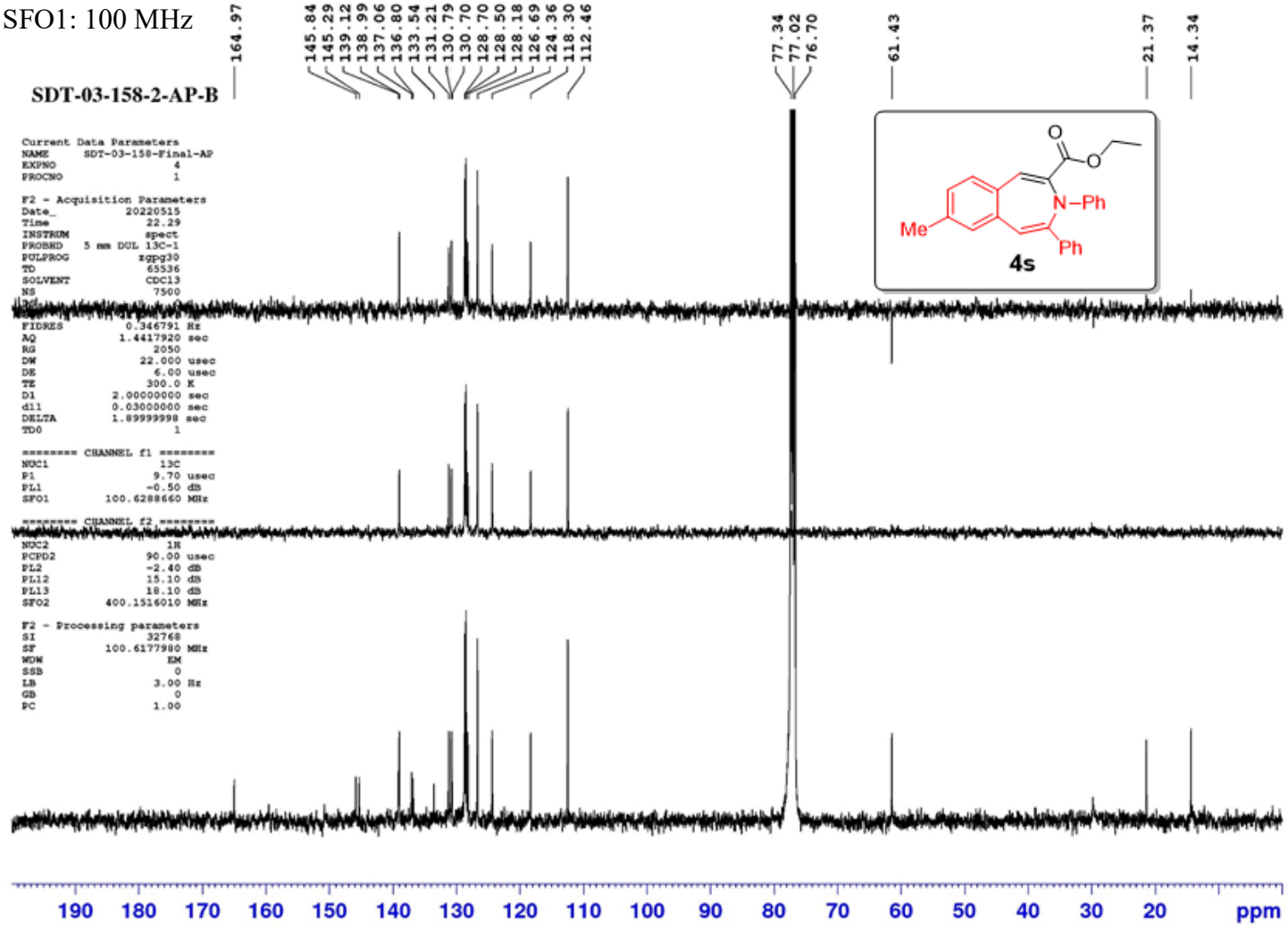
Solvent: $CDCl_3$
SFO1: 500 MHz



Solvent: $CDCl_3$
SFO1: 500 MHz



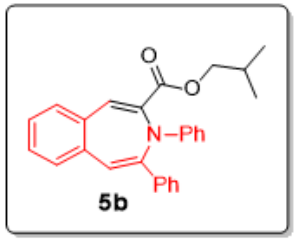
Solvent: $CDCl_3$
SFO1: 100 MHz



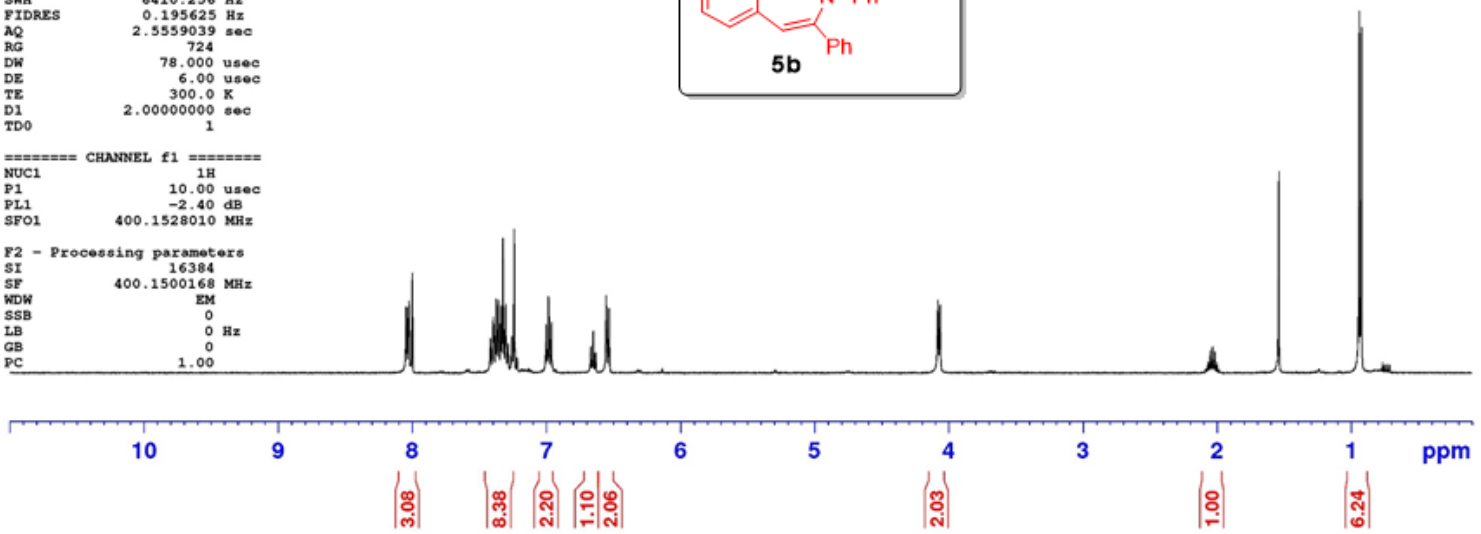
Solvent: $CDCl_3$
SFO1: 400 MHz

SDT-03-93-IBu-AP

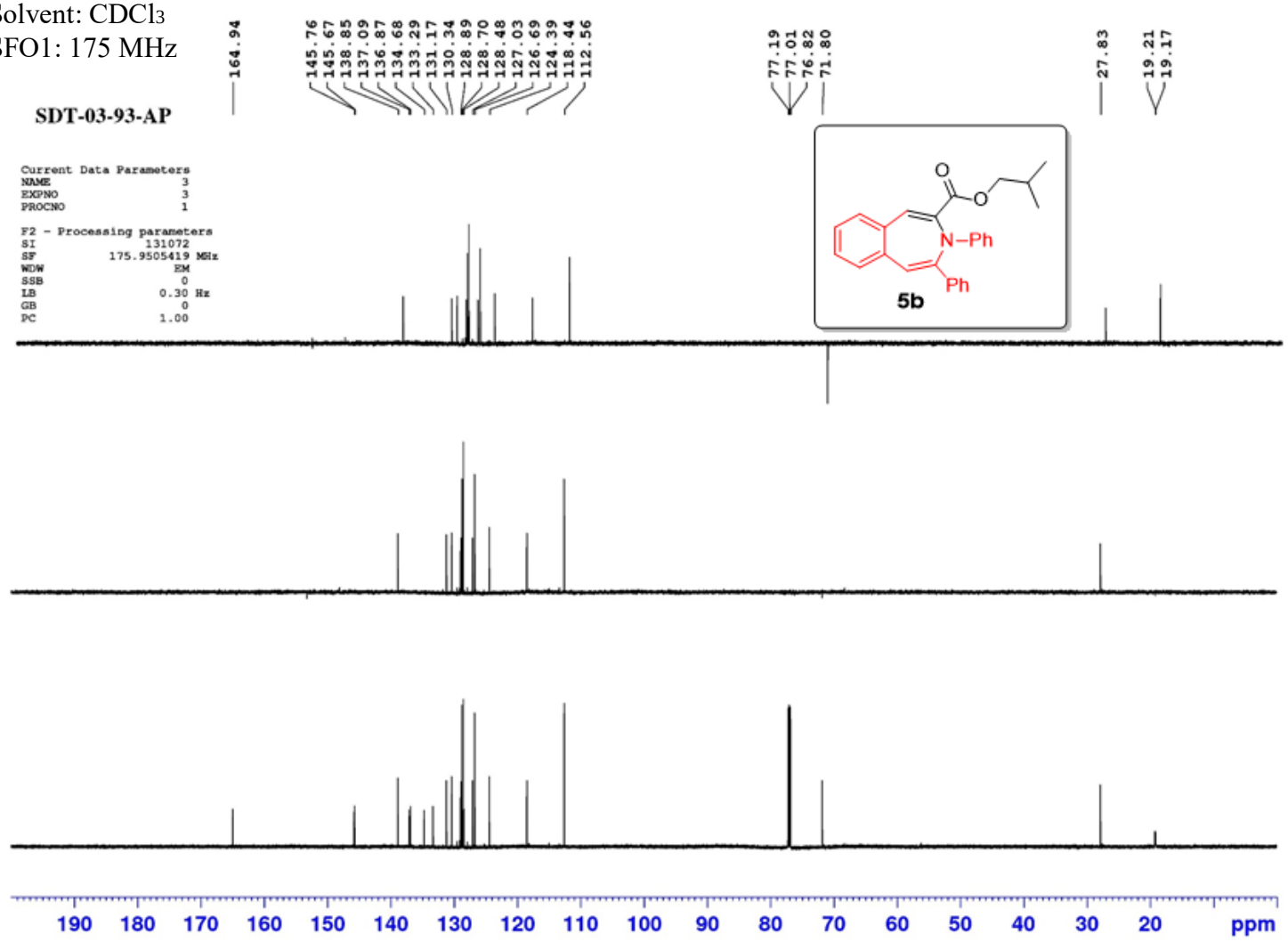
```
Current Data Parameters
NAME      SDT-03-93-IBu-AP
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20220309
Time              16.28
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                   7
DS                   0
SWH           6410.256 Hz
FIDRES        0.195625 Hz
AQ            2.5559039 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 Hz
GB                   0
PC                1.00
```



5b



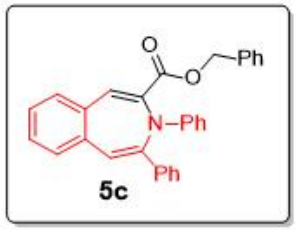
Solvent: $CDCl_3$
SFO1: 175 MHz



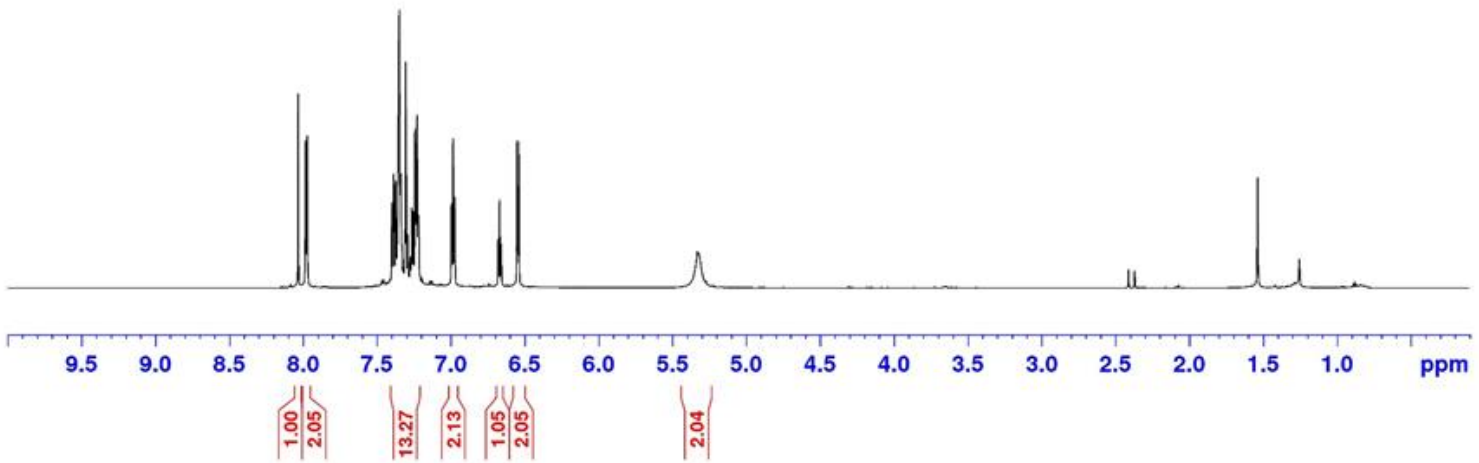
Solvent: $CDCl_3$
SFO1: 700 MHz

SDT-03-106-AP

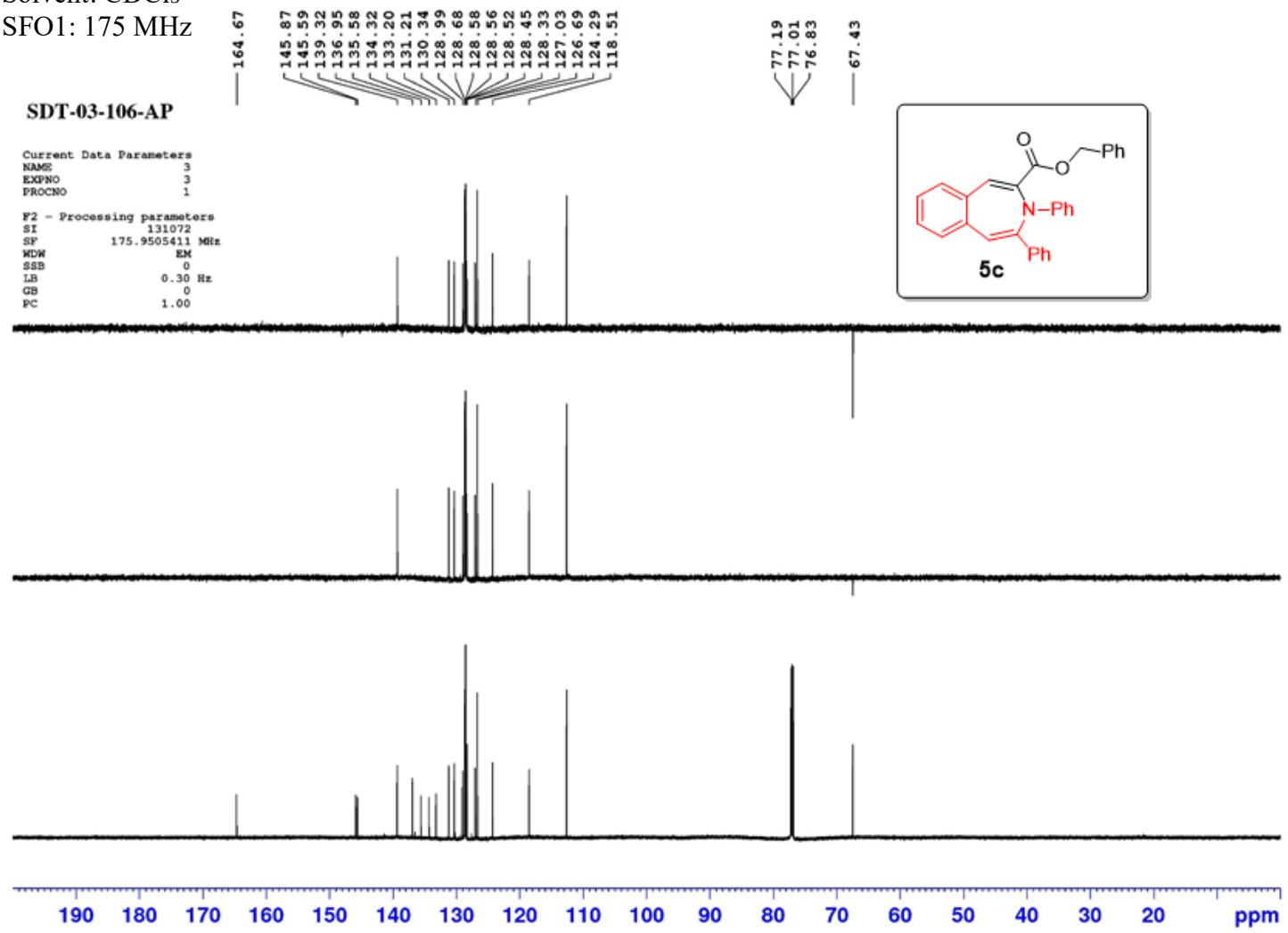
```
Current Data Parameters
NAME      SDT-03-106-AP-H.fid
EXPNO                      1
PROCNO                     1

F2 - Processing parameters
SI                     65536
SF           699.7430993 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



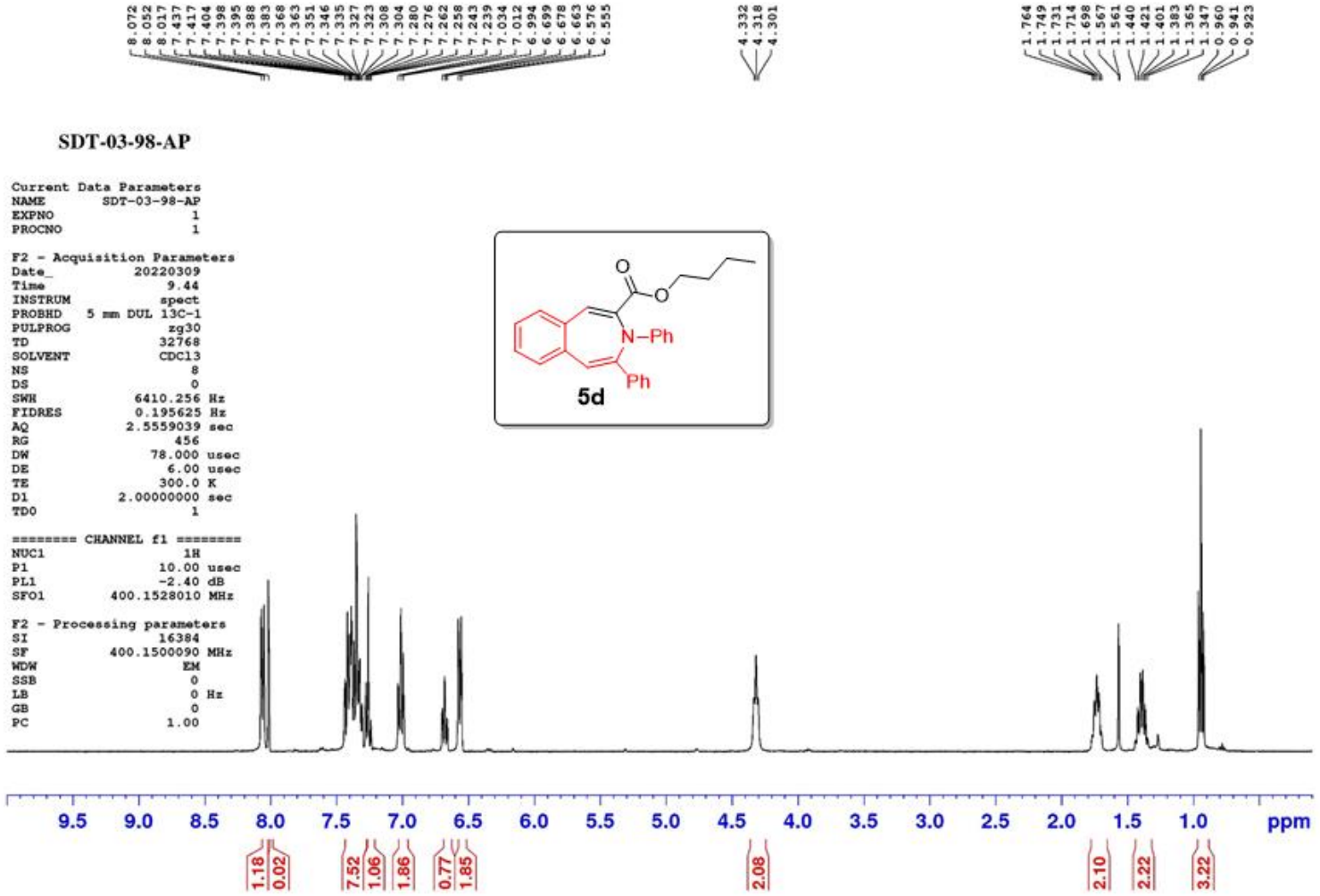
5c



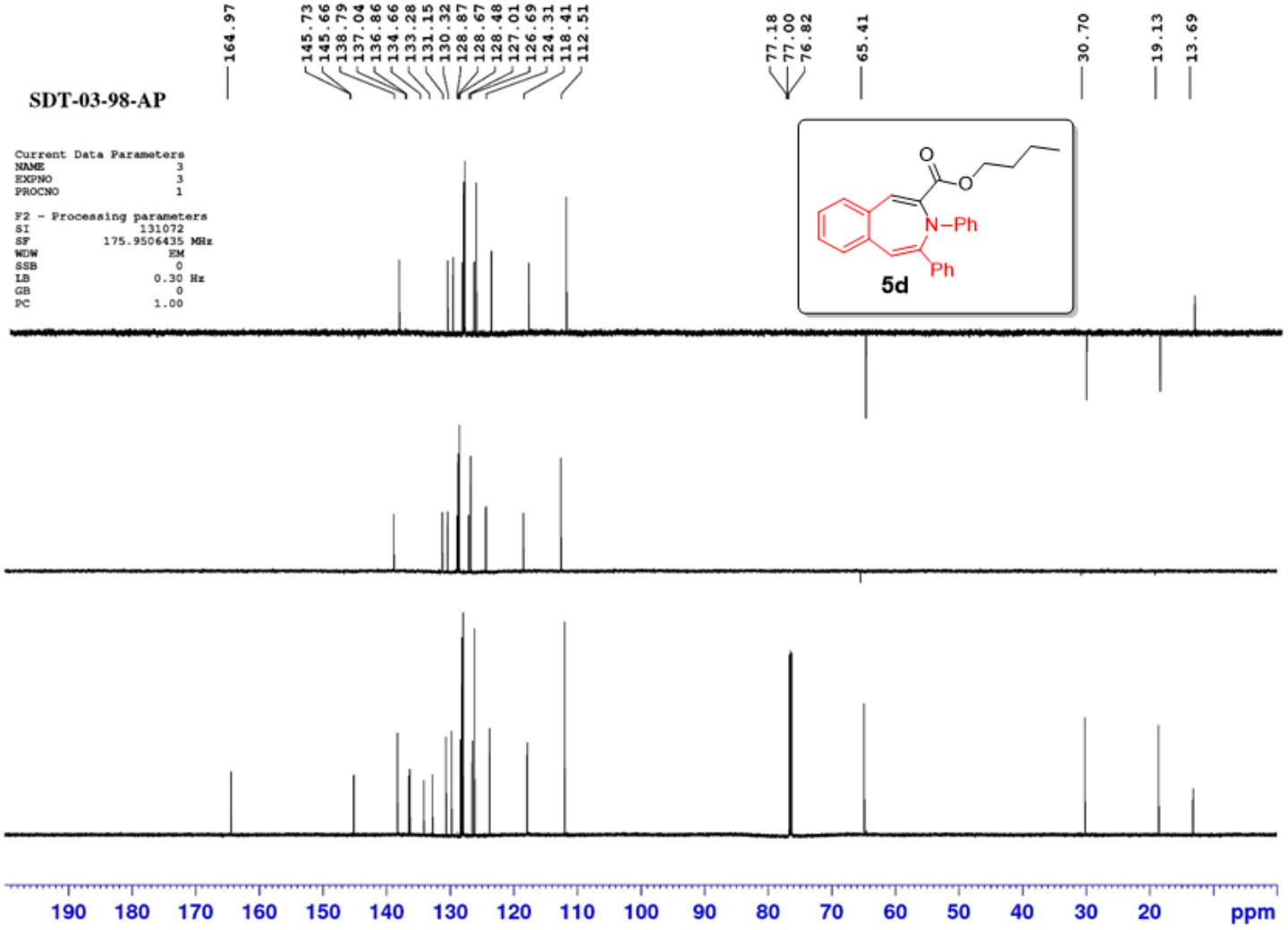
Solvent: $CDCl_3$
SFO1: 175 MHz



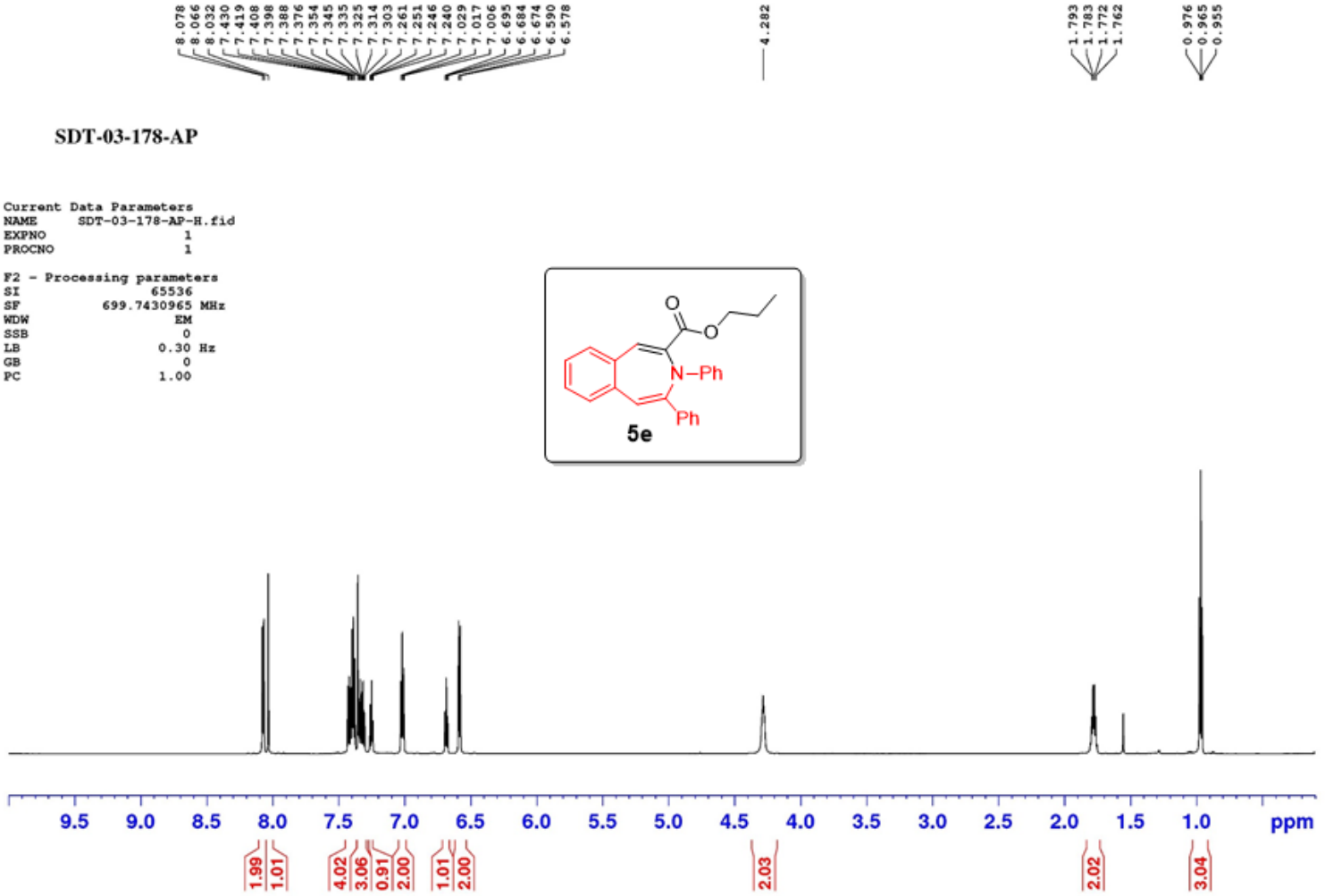
Solvent: $CDCl_3$
SFO1: 400 MHz



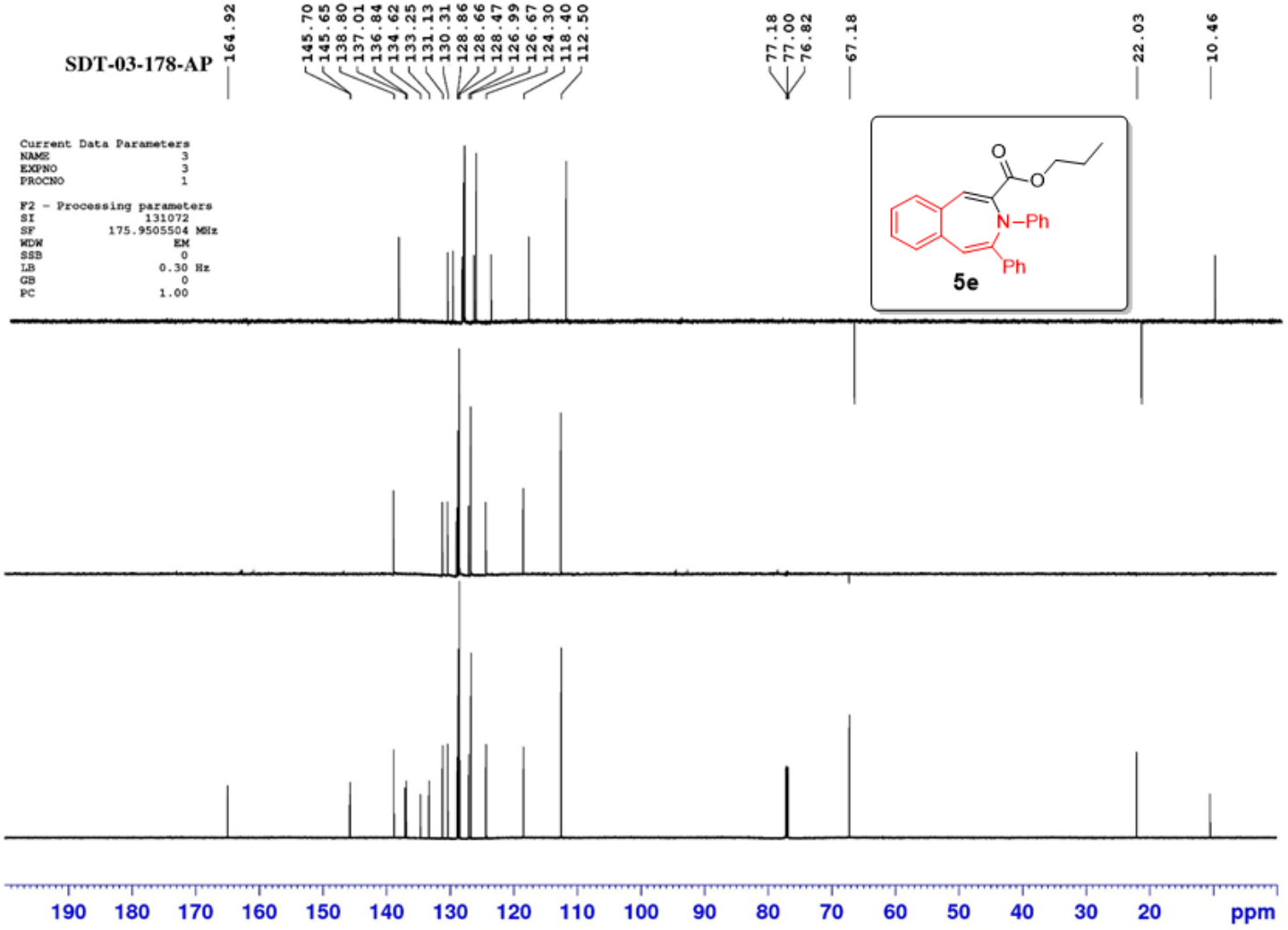
Solvent: $CDCl_3$
SFO1: 175 MHz



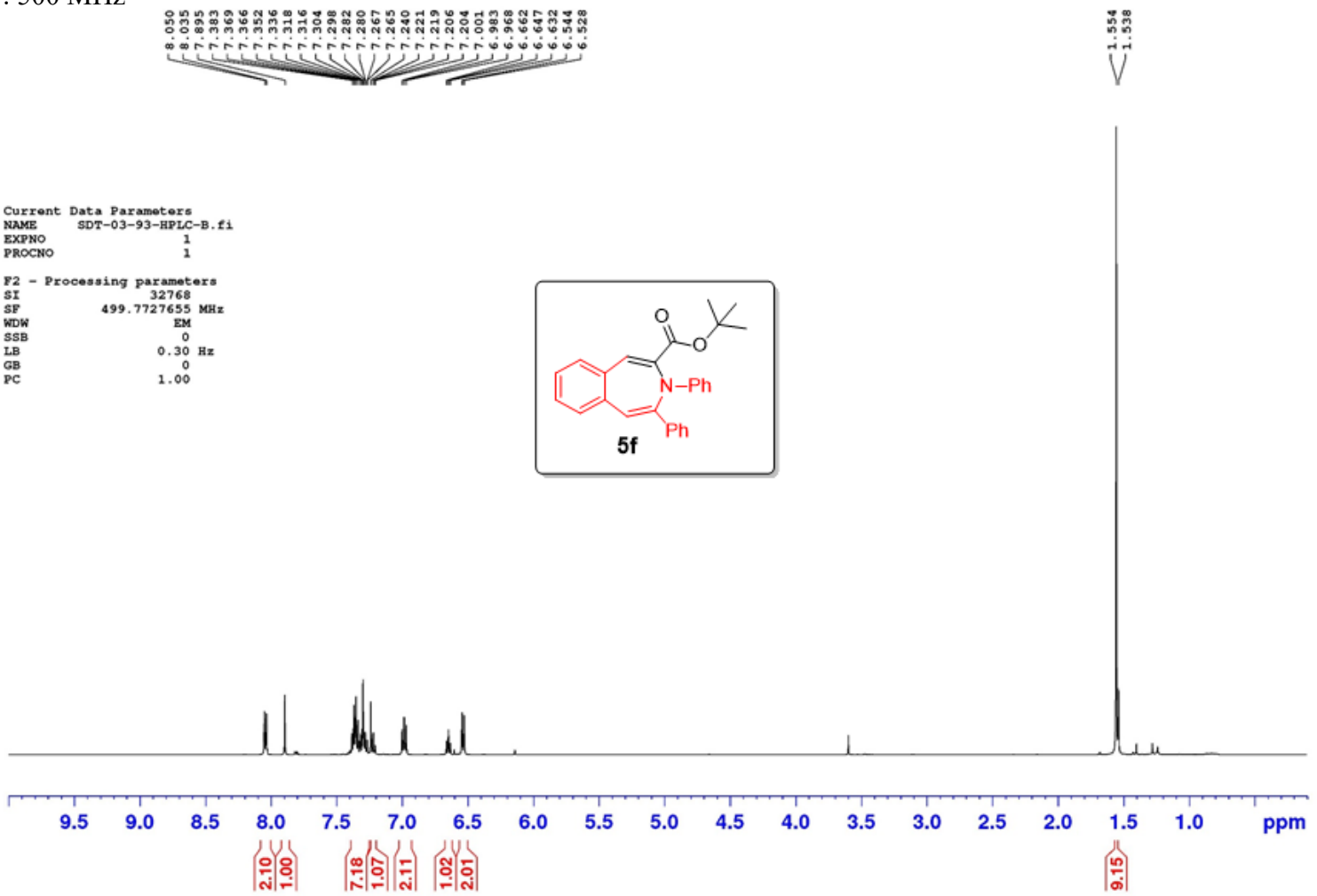
Solvent: $CDCl_3$
SFO1: 700 MHz



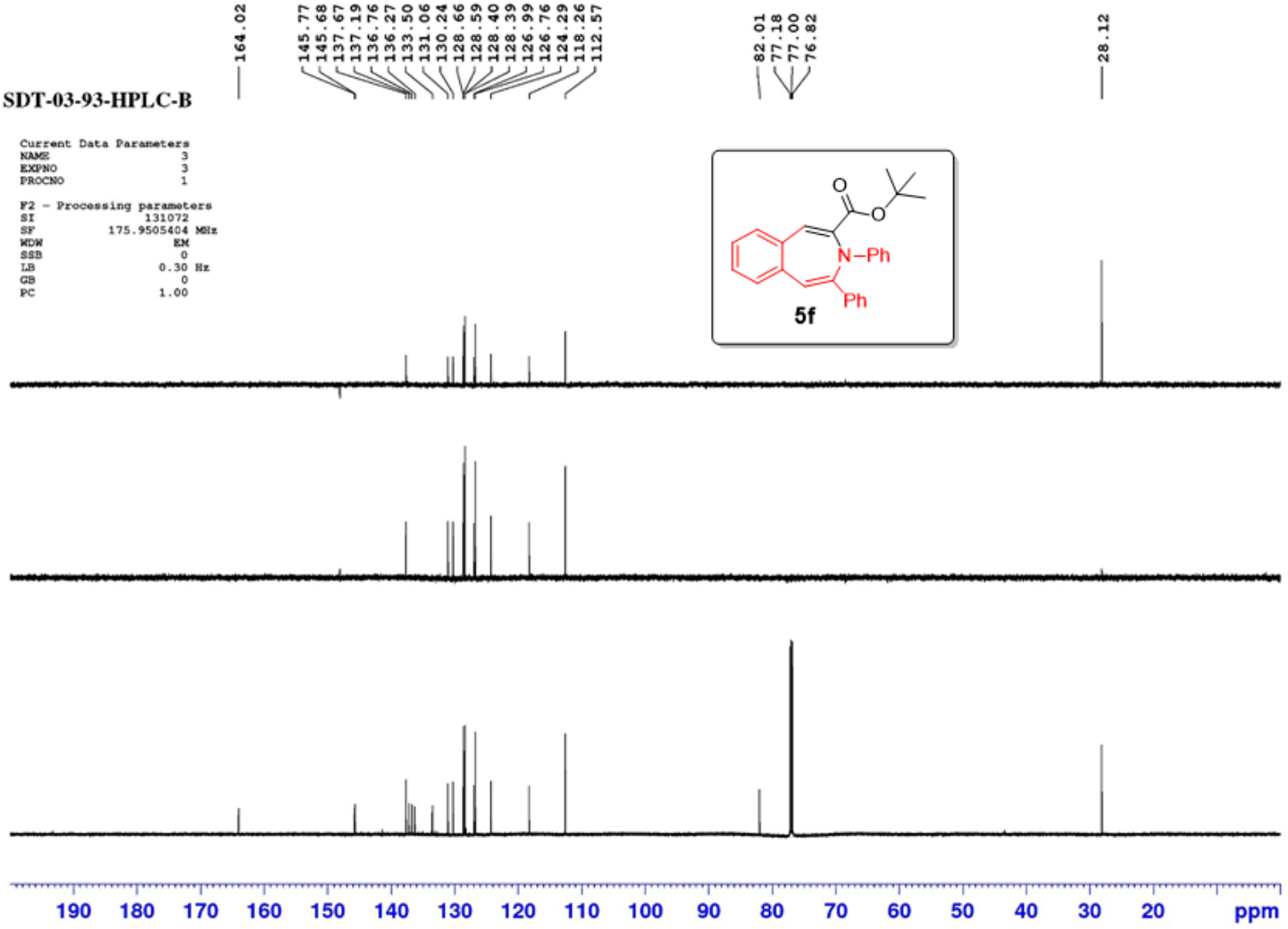
Solvent: $CDCl_3$
SFO1: 175 MHz



Solvent: $CDCl_3$
SFO1: 500 MHz



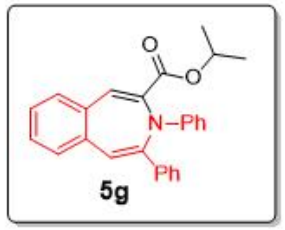
Solvent: $CDCl_3$
SFO1: 175 MHz



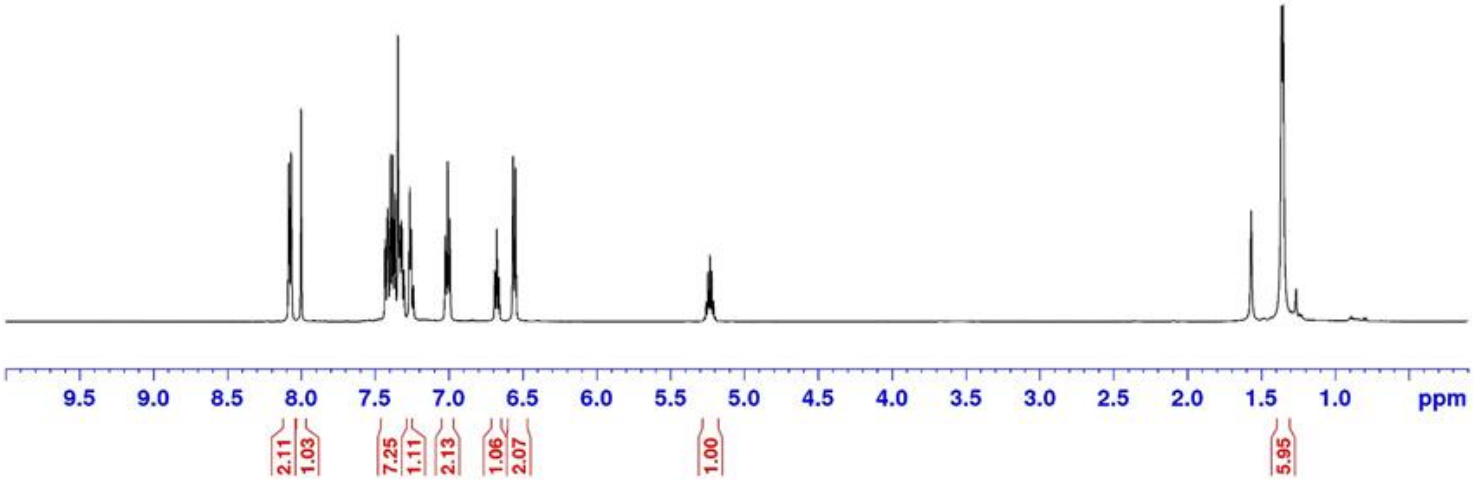
Solvent: $CDCl_3$
SFO1: 500 MHz

SDT-03-160-AP

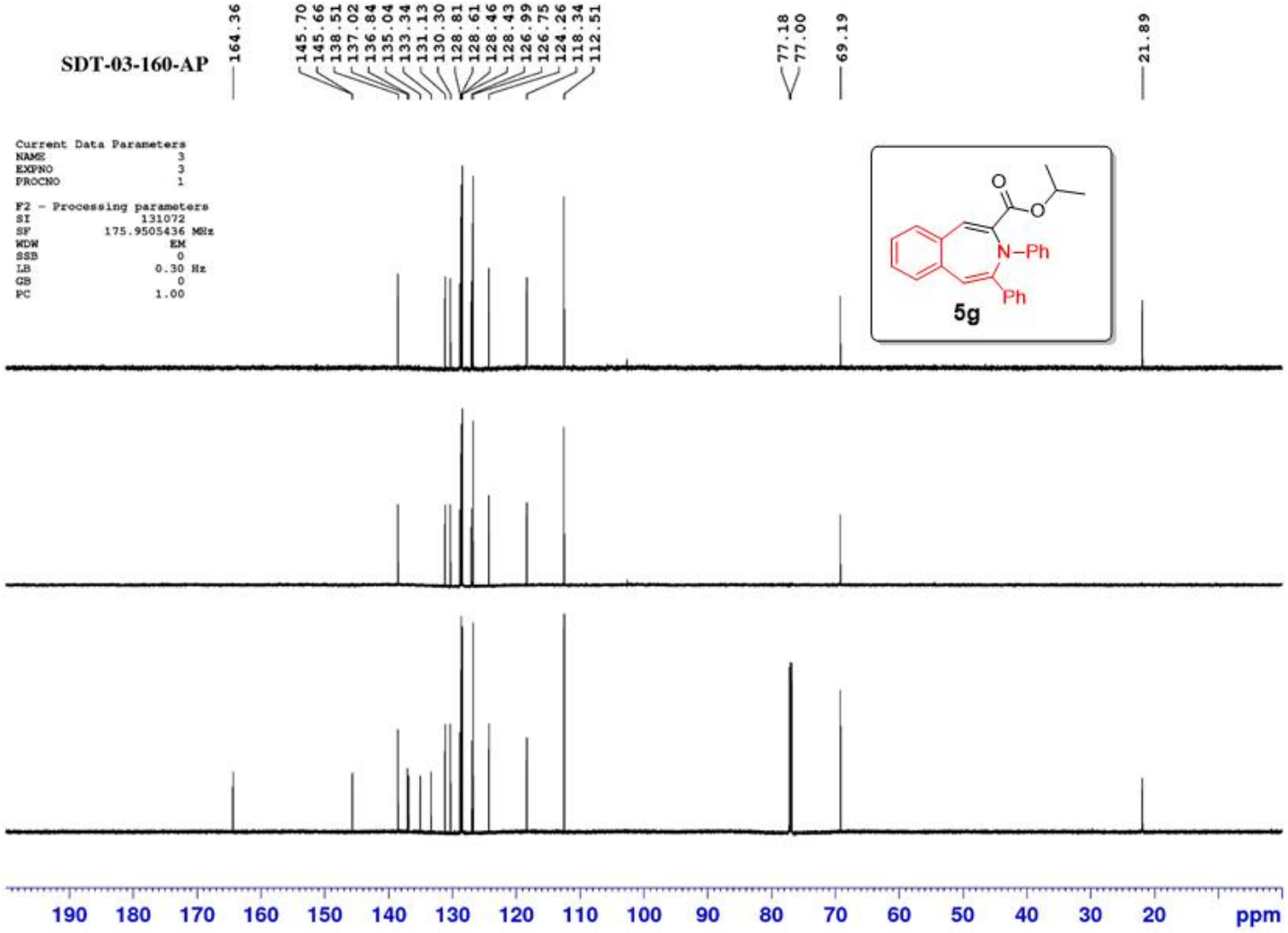
```
Current Data Parameters
NAME      SDT-03-160-Final-AP
EXPNO                      1
PROCNO                     1

F2 - Processing parameters
SI                     32768
SF           499.7727540 MHz
WDW                       EM
SSB                        0
LB                  0.30 Hz
GB                         0
PC                      1.00
```



5g



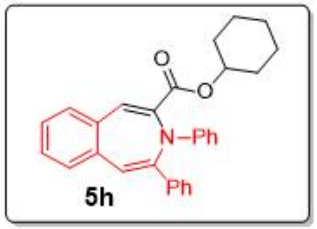
Solvent: $CDCl_3$
SFO1: 175 MHz



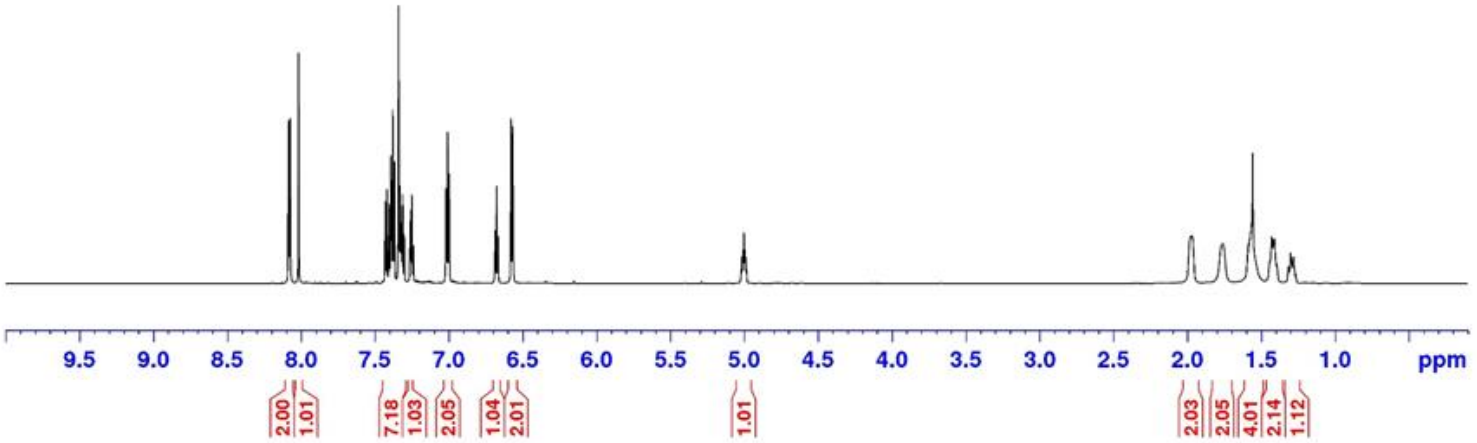
Solvent: $CDCl_3$
SFO1: 700 MHz

SDT-03-171-AP

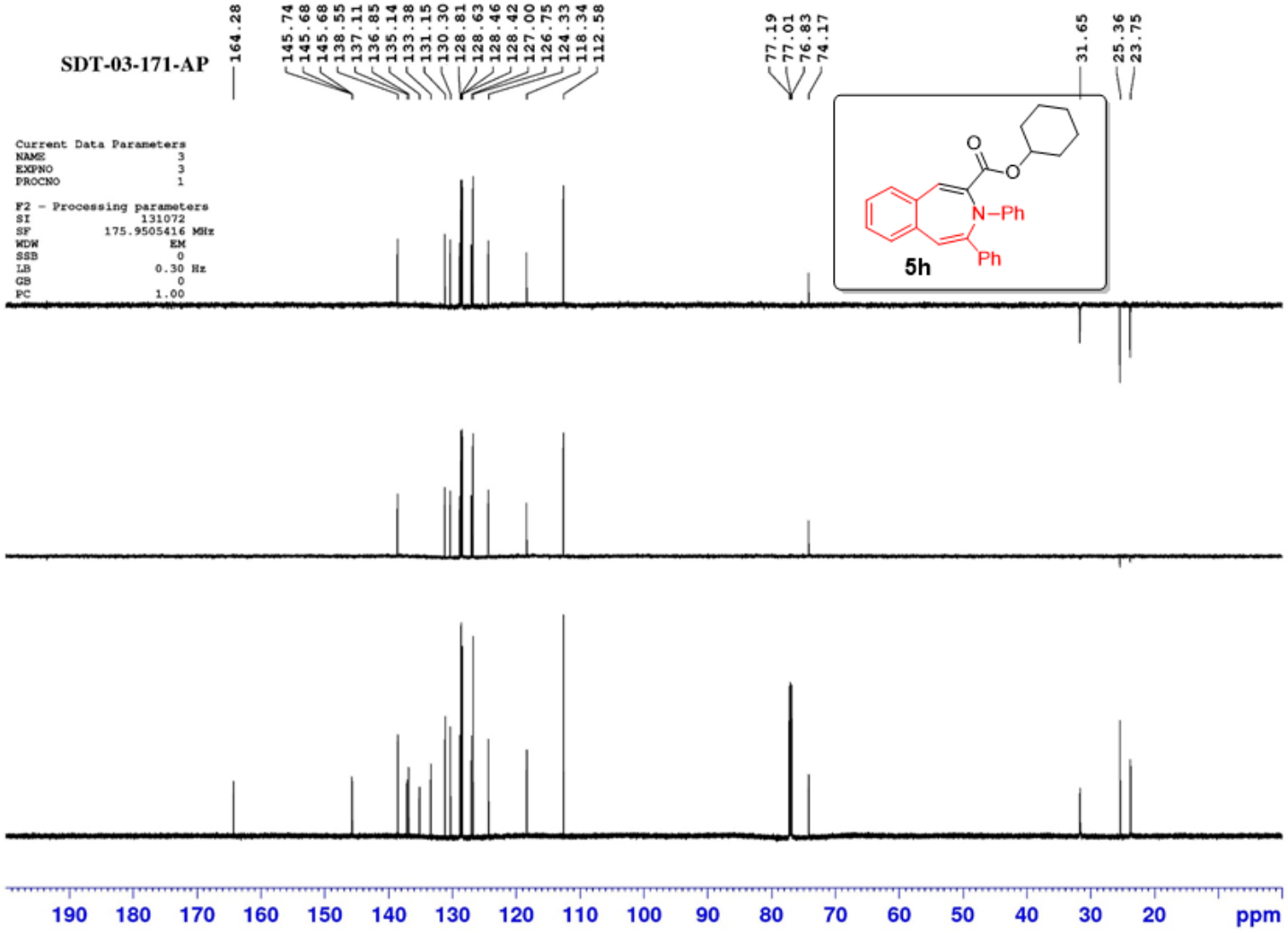
```
Current Data Parameters
NAME      SDT-03-171-AP-H.fid
EXPNO                       1
PROCNO                      1

F2 - Processing parameters
SI                      65536
SF             699.7430877 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



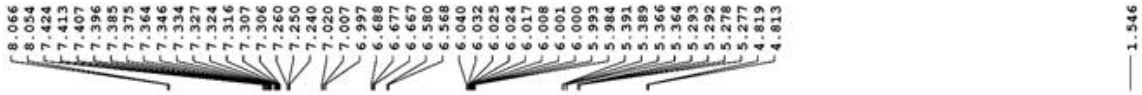
5h



Solvent: $CDCl_3$
SFO1: 175 MHz

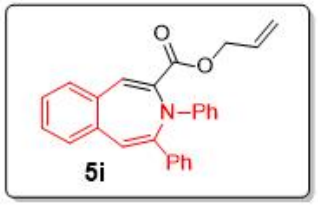


Solvent: $CDCl_3$
SFO1: 700 MHz

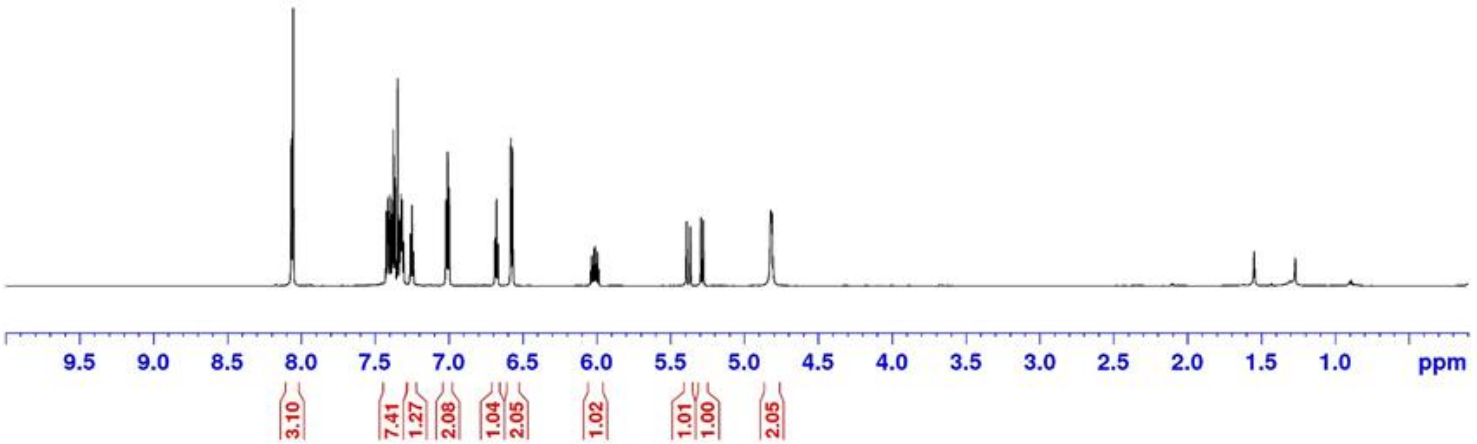


SDT-03-173-AP

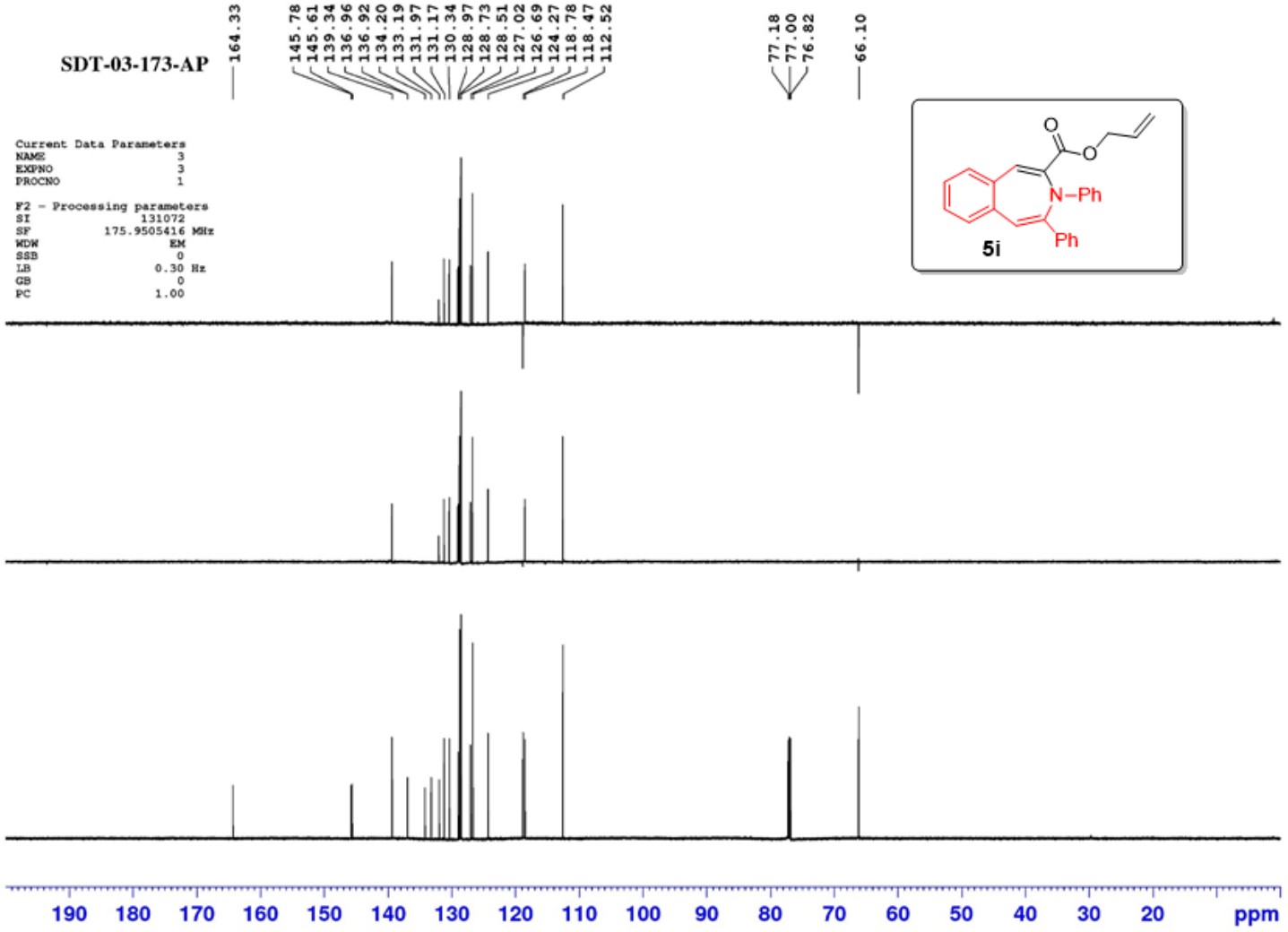
```
Current Data Parameters
NAME      SDT-03-173-AP-H.fid
EXPNO                       1
PROCNO                      1

F2 - Processing parameters
SI                      65536
SF             699.7430953 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



5i



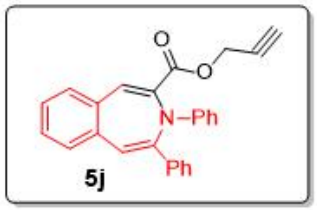
Solvent: $CDCl_3$
SFO1: 175 MHz



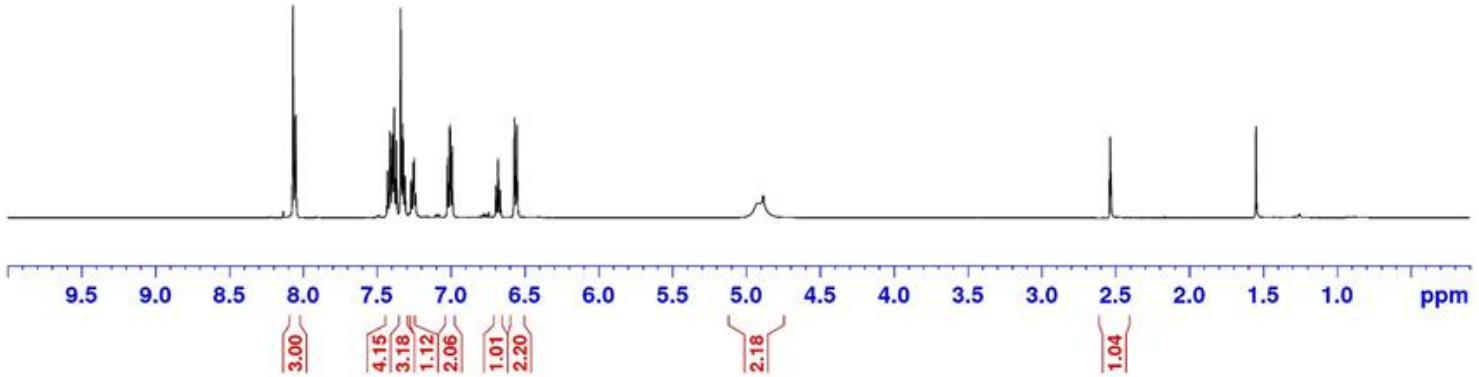
Solvent: $CDCl_3$
SFO1: 500 MHz

SDT-03-175-AP

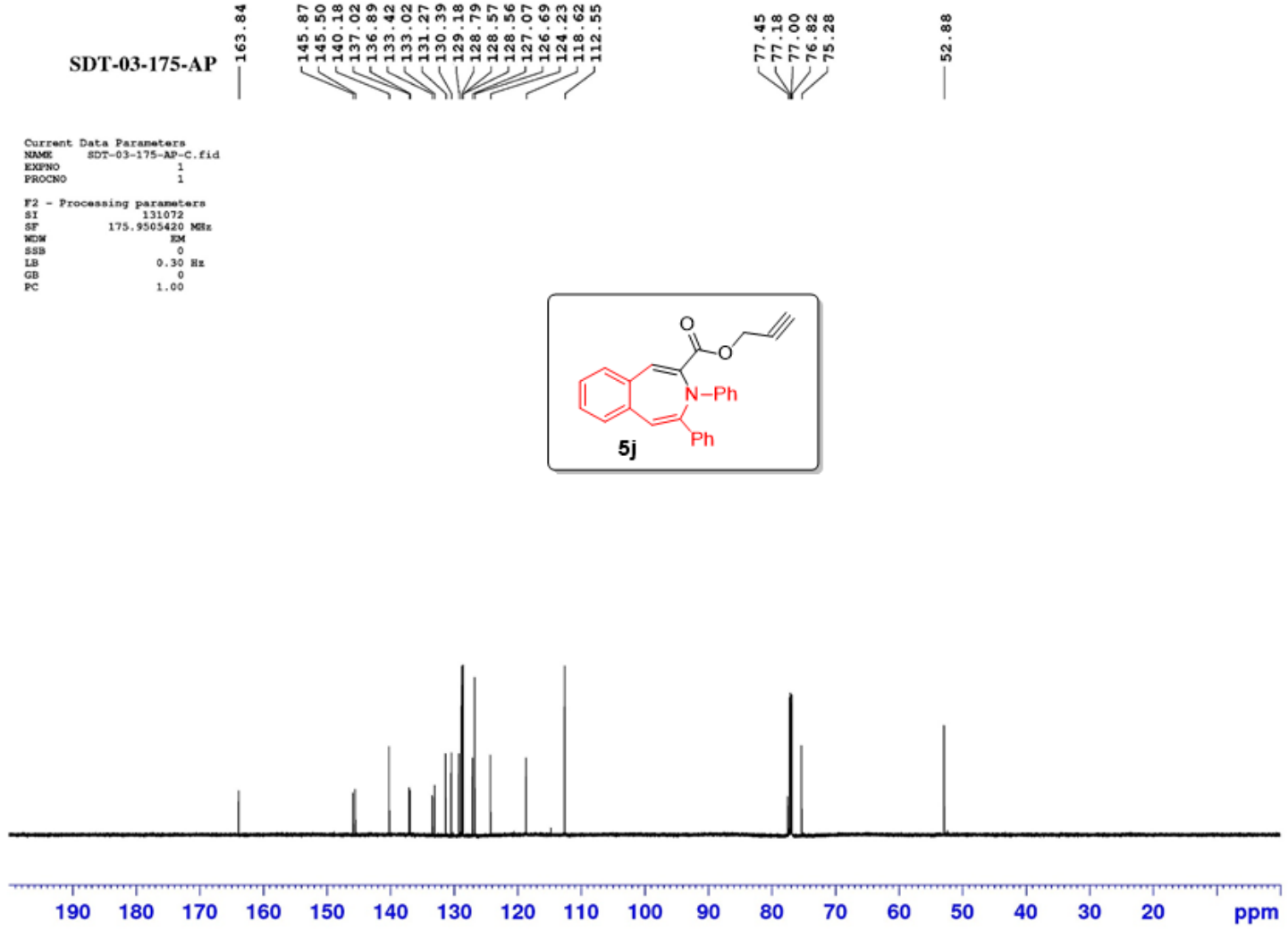
```
Current Data Parameters
NAME      SDT-03-175-Final-AP
EXPNO                      1
PROCNO                     1

F2 - Processing parameters
SI                     32768
SF           499.7727620 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



5j



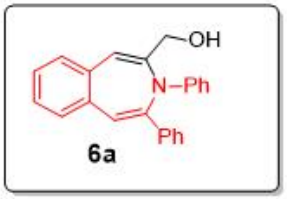
Solvent: $CDCl_3$
SFO1: 175 MHz



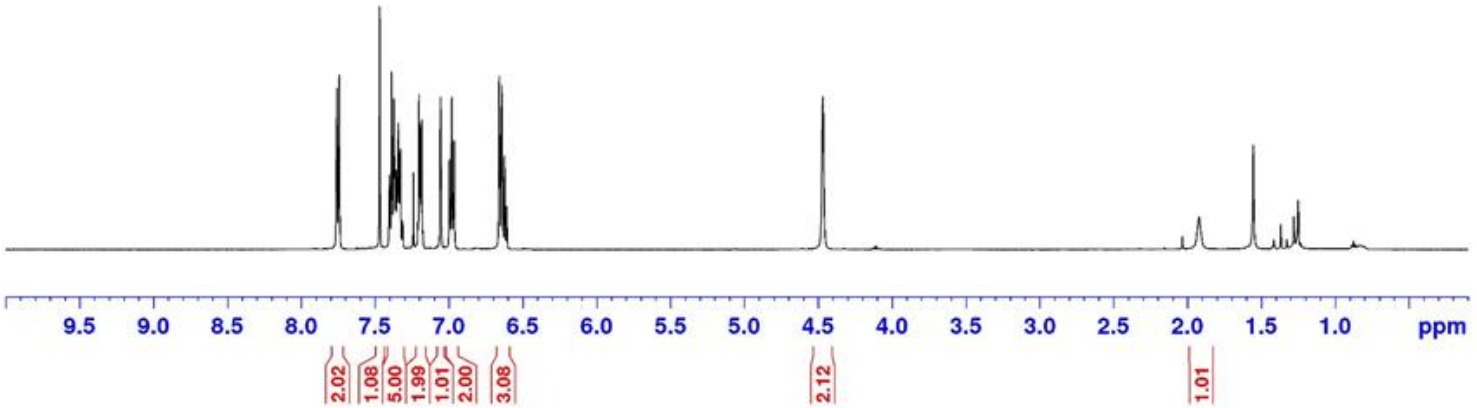
Solvent: $CDCl_3$
SFO1: 500 MHz

SDT-03-191-AP

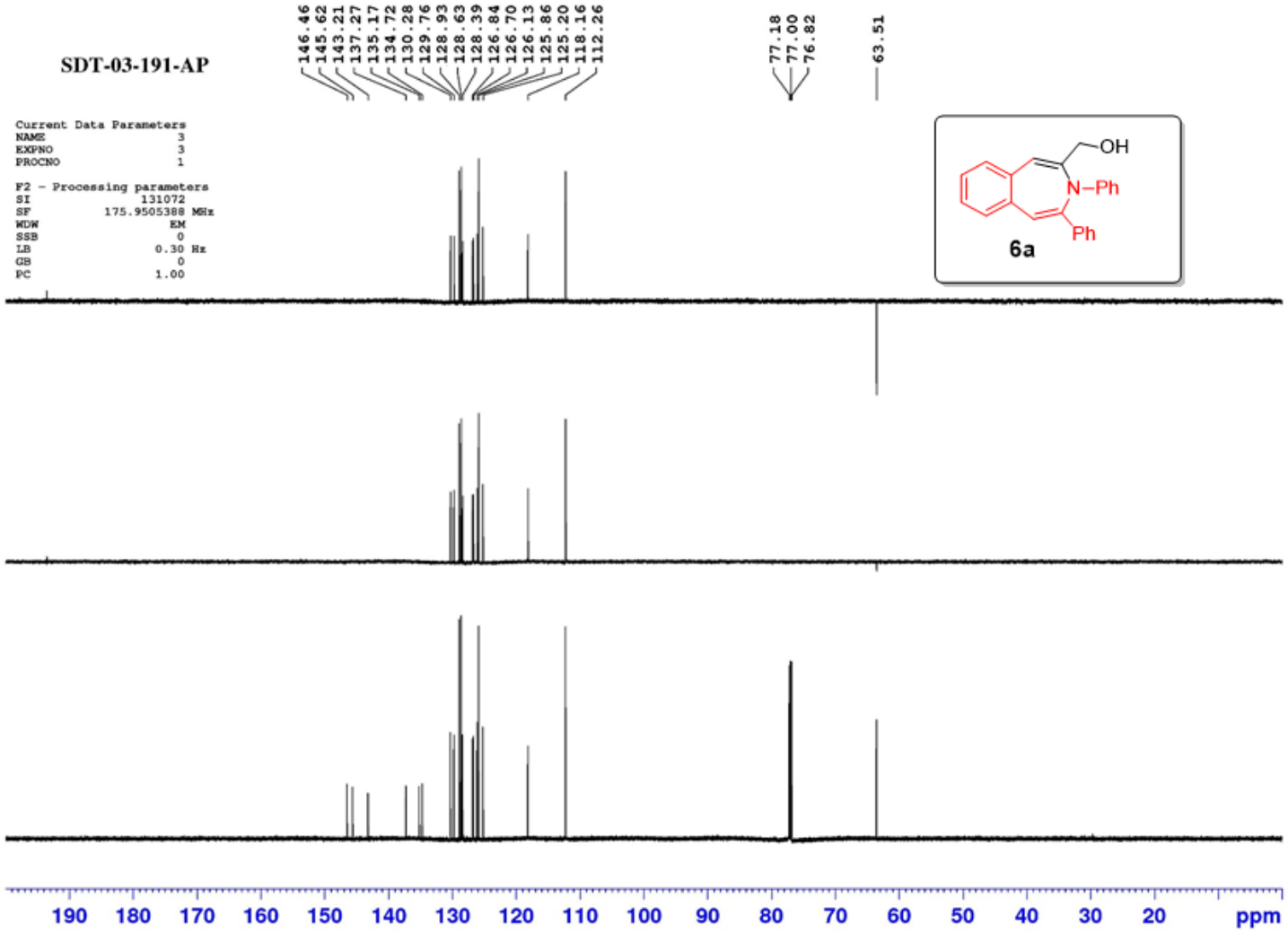
```
Current Data Parameters
NAME      SDT-03-191-AP.fid
EXPNO                    1
PROCNO                   1

F2 - Processing parameters
SI                   32768
SF           499.7727656 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



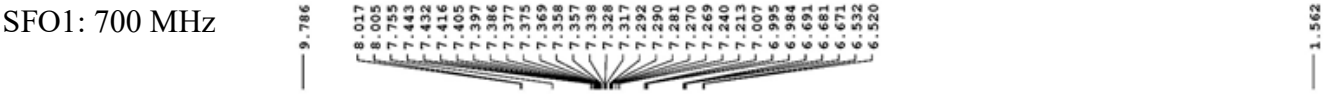
6a



Solvent: $CDCl_3$
SFO1: 175 MHz

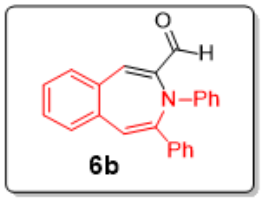


Solvent: $CDCl_3$
SFO1: 700 MHz

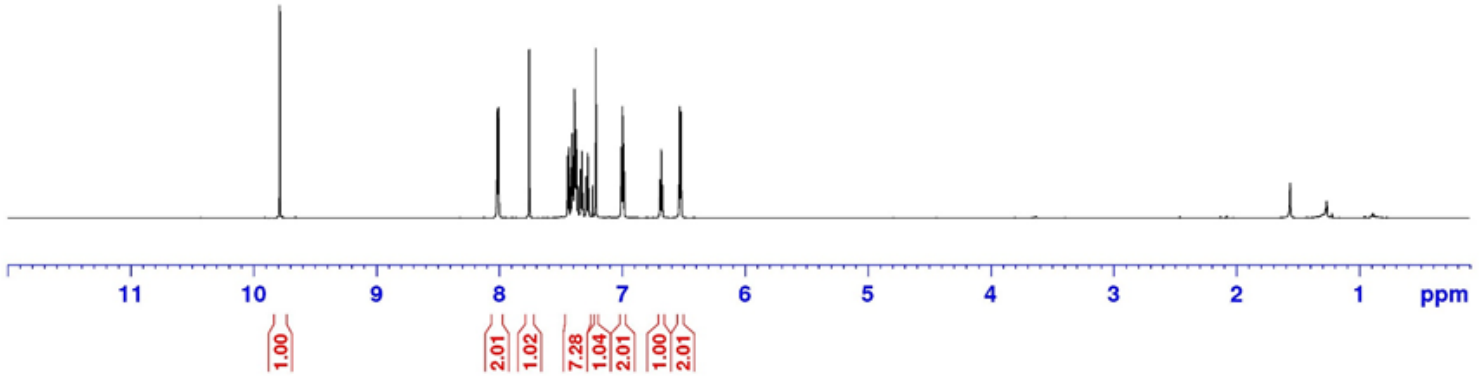


SDT-03-193-AP

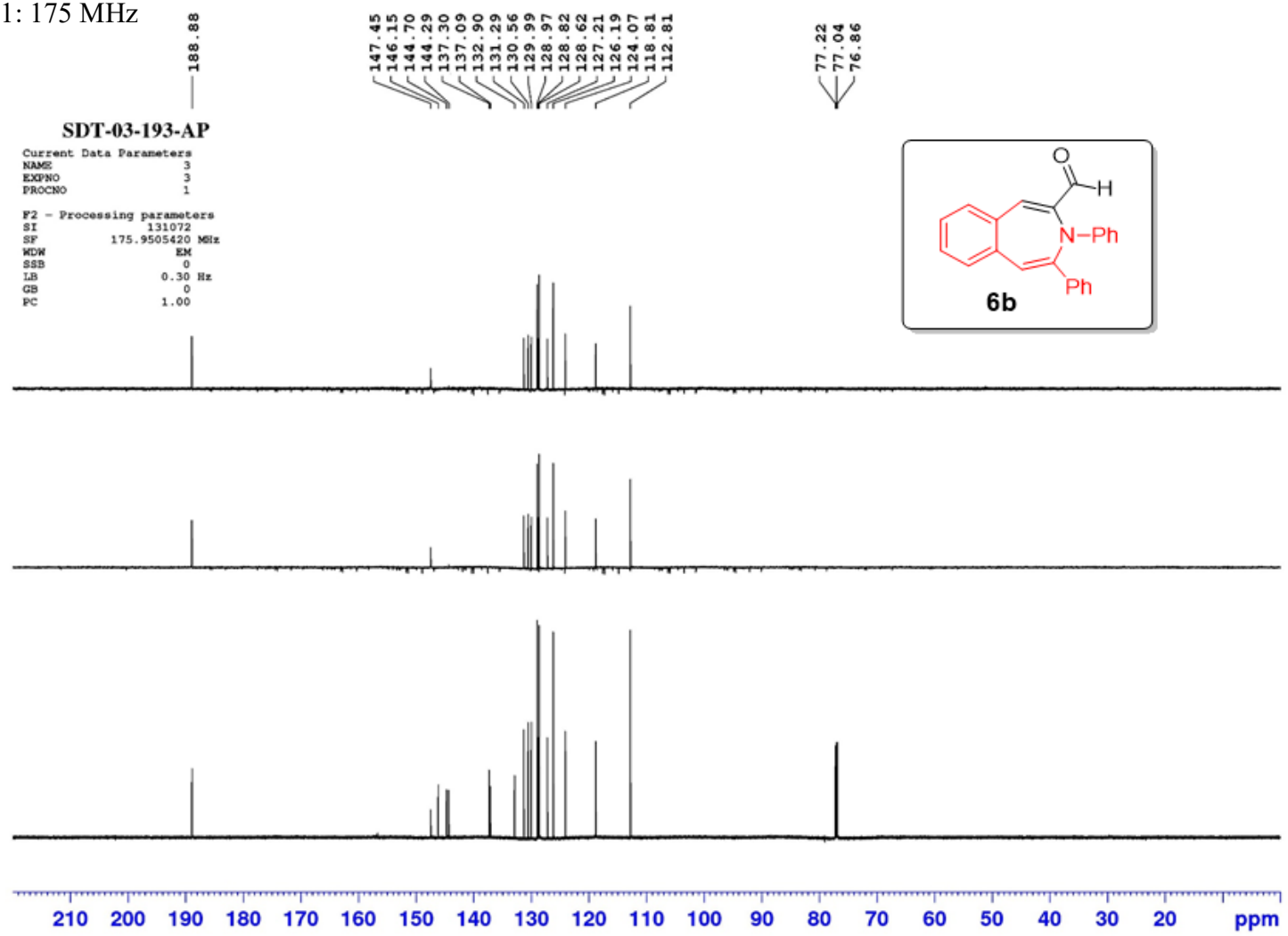
```
Current Data Parameters
NAME      SDT-03-193-AP-H.fid
EXPNO                      1
PROCNO                     1

F2 - Processing parameters
SI                     65536
SF           699.7431042 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



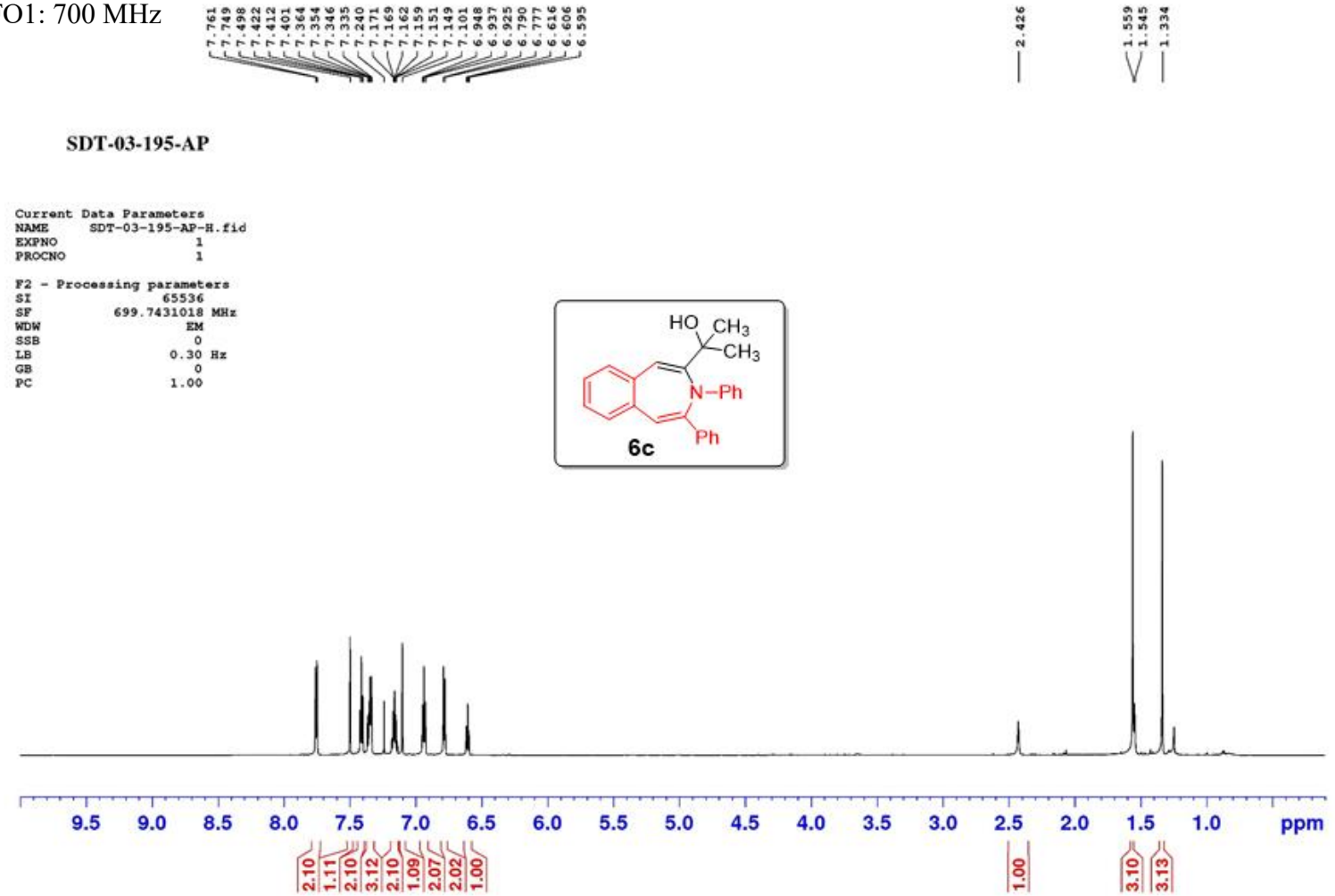
6b



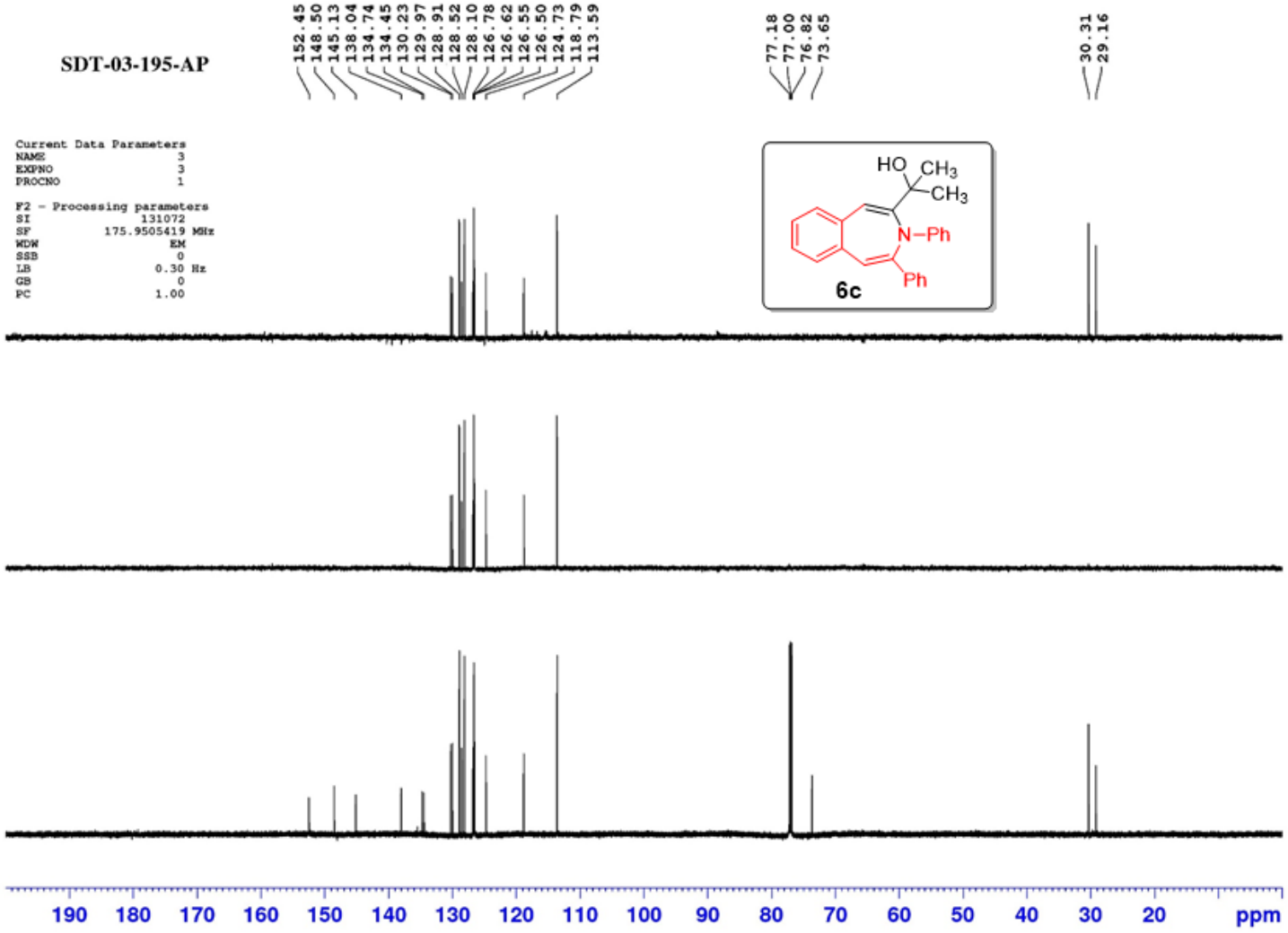
Solvent: $CDCl_3$
SFO1: 175 MHz



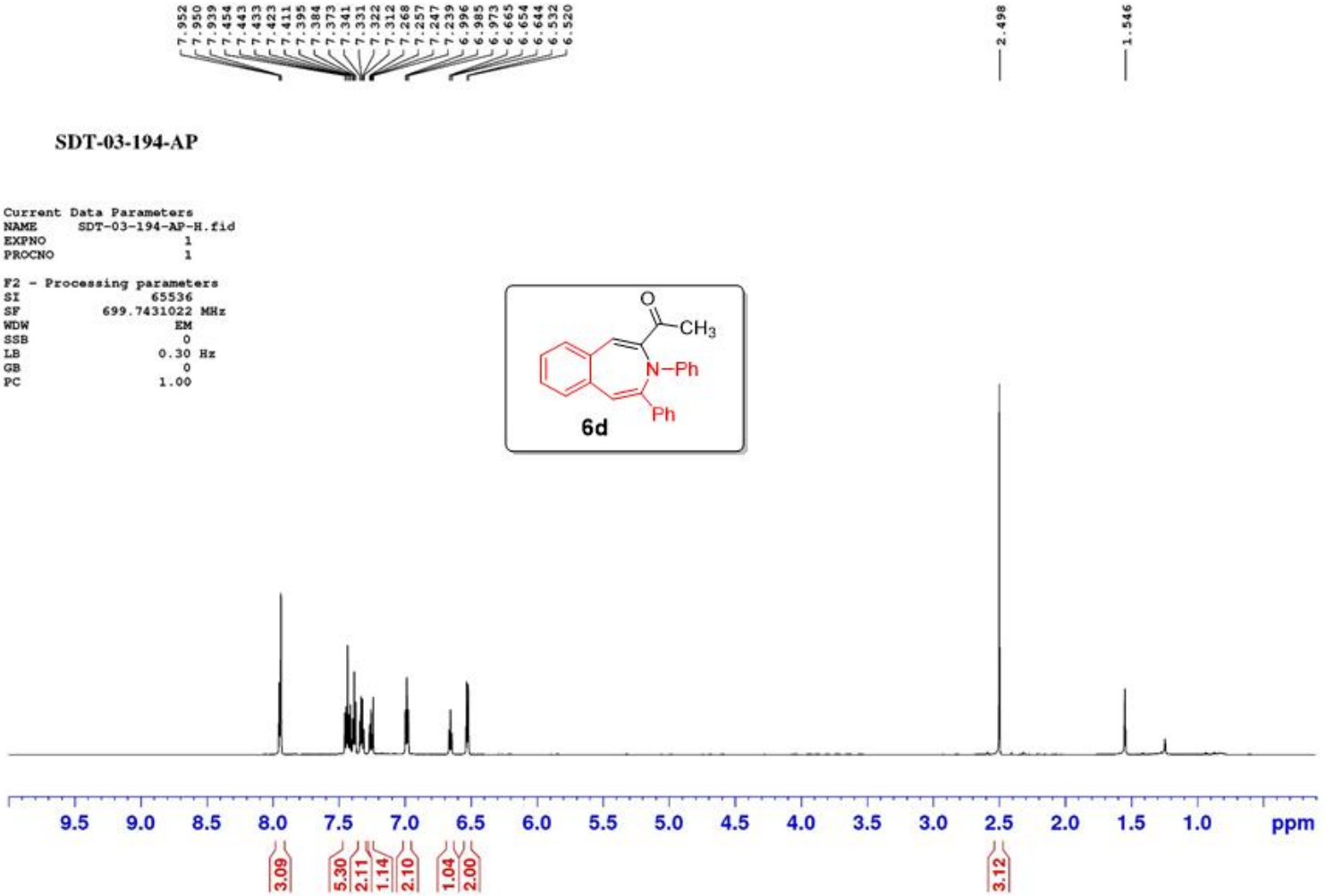
Solvent: $CDCl_3$
SFO1: 700 MHz



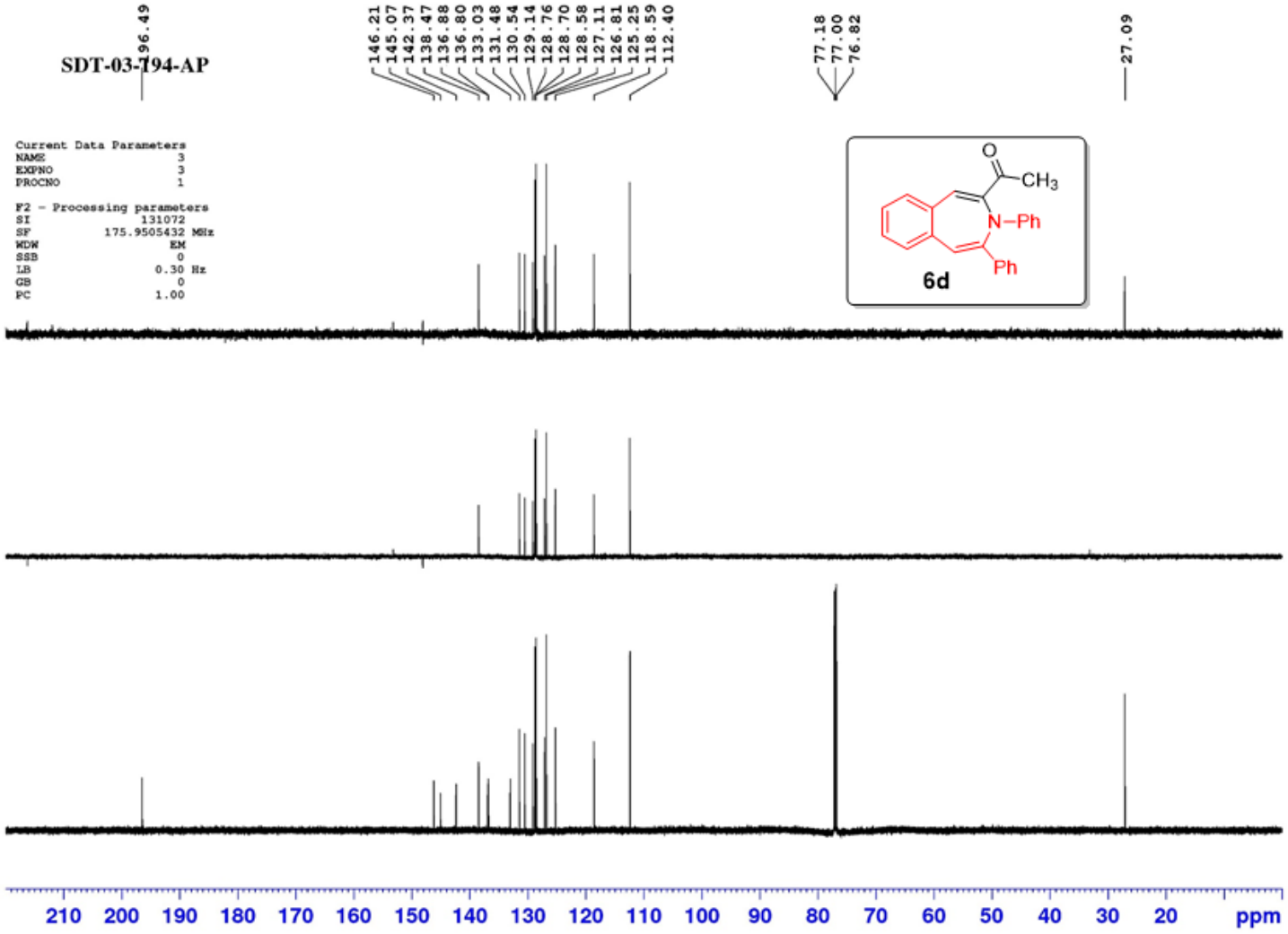
Solvent: $CDCl_3$
SFO1: 175 MHz



Solvent: $CDCl_3$
SFO1: 700 MHz



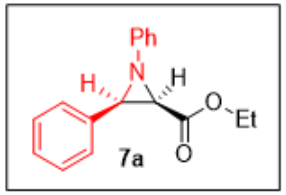
Solvent: $CDCl_3$
SFO1: 175 MHz

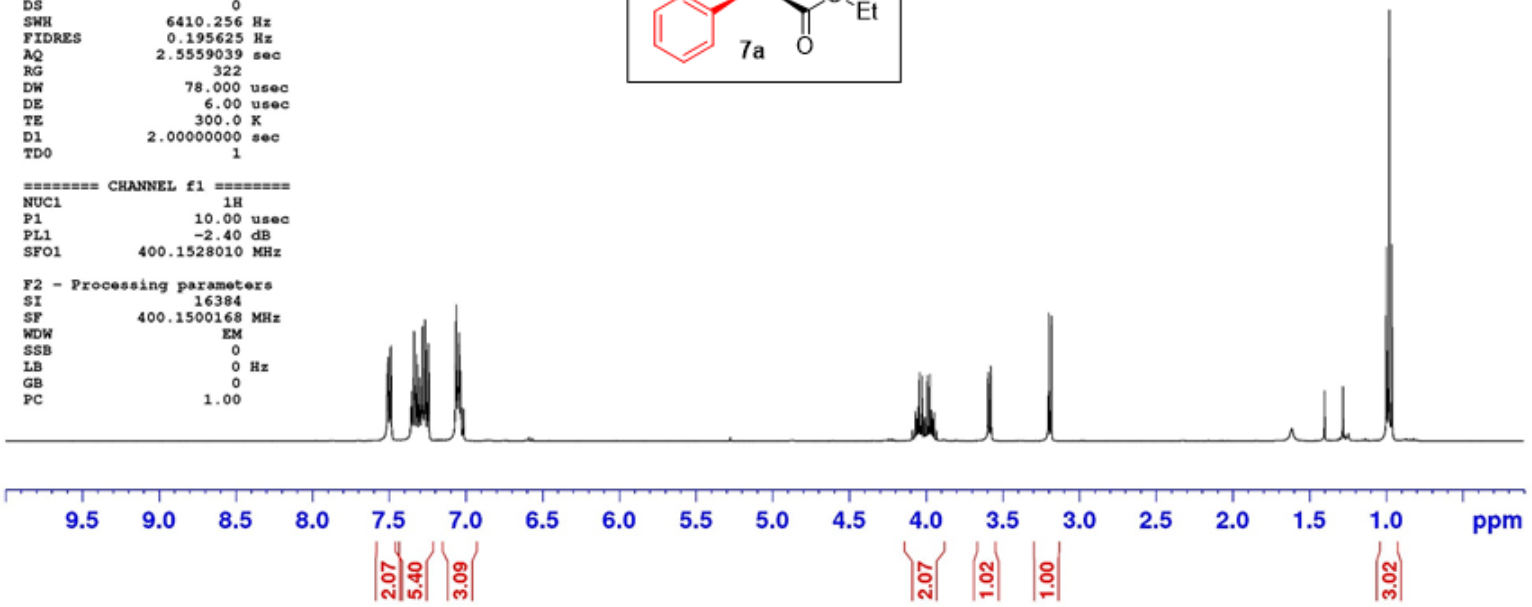


Solvent: $CDCl_3$
SFO1: 400 MHz

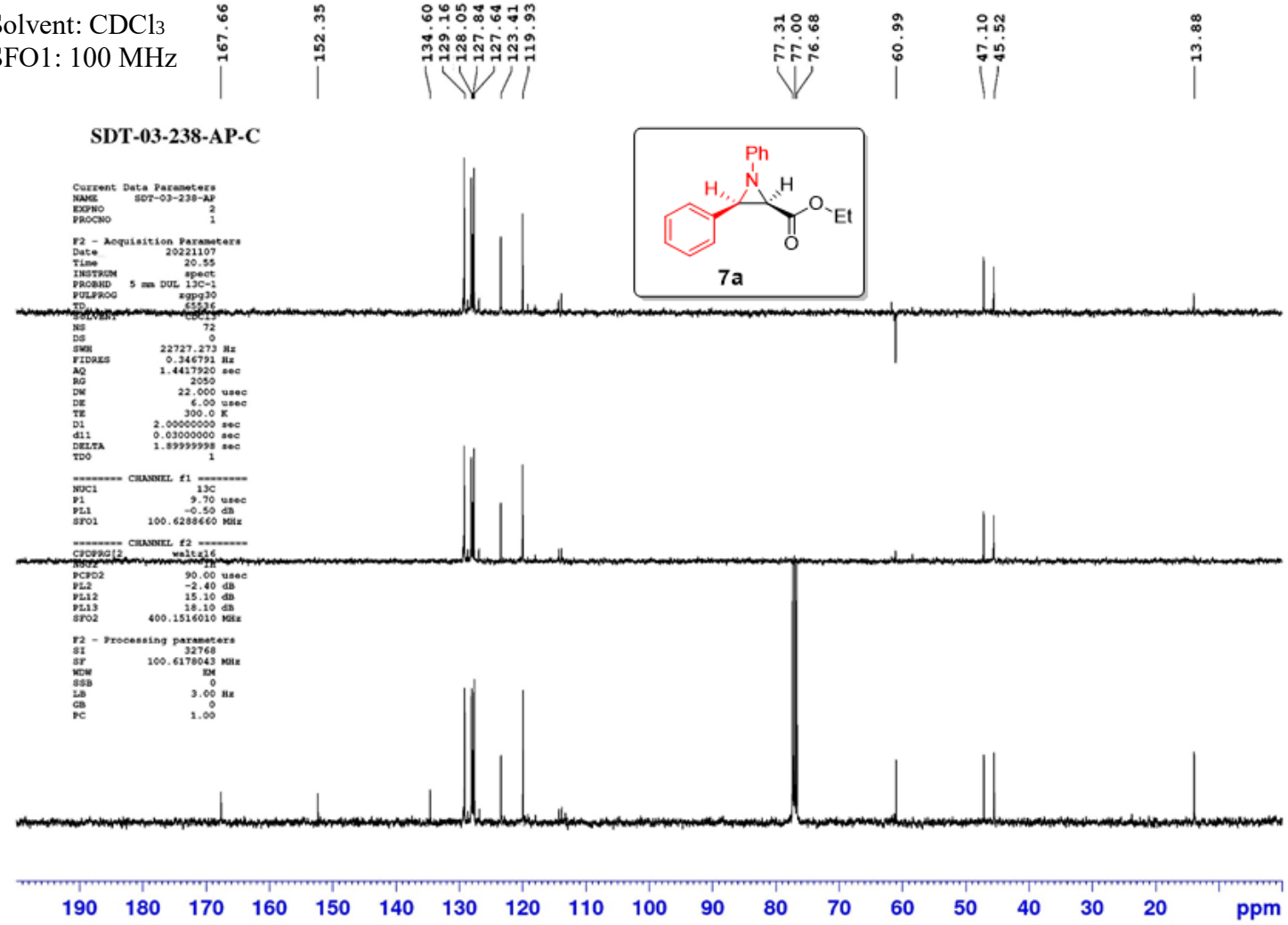
SDT-04-30-AP-C

```
Current Data Parameters
NAME        SDT-04-30-AP
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230107
Time               16.57
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    12
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559039 sec
RG                   322
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 Hz
GB                     0
PC                  1.00
```



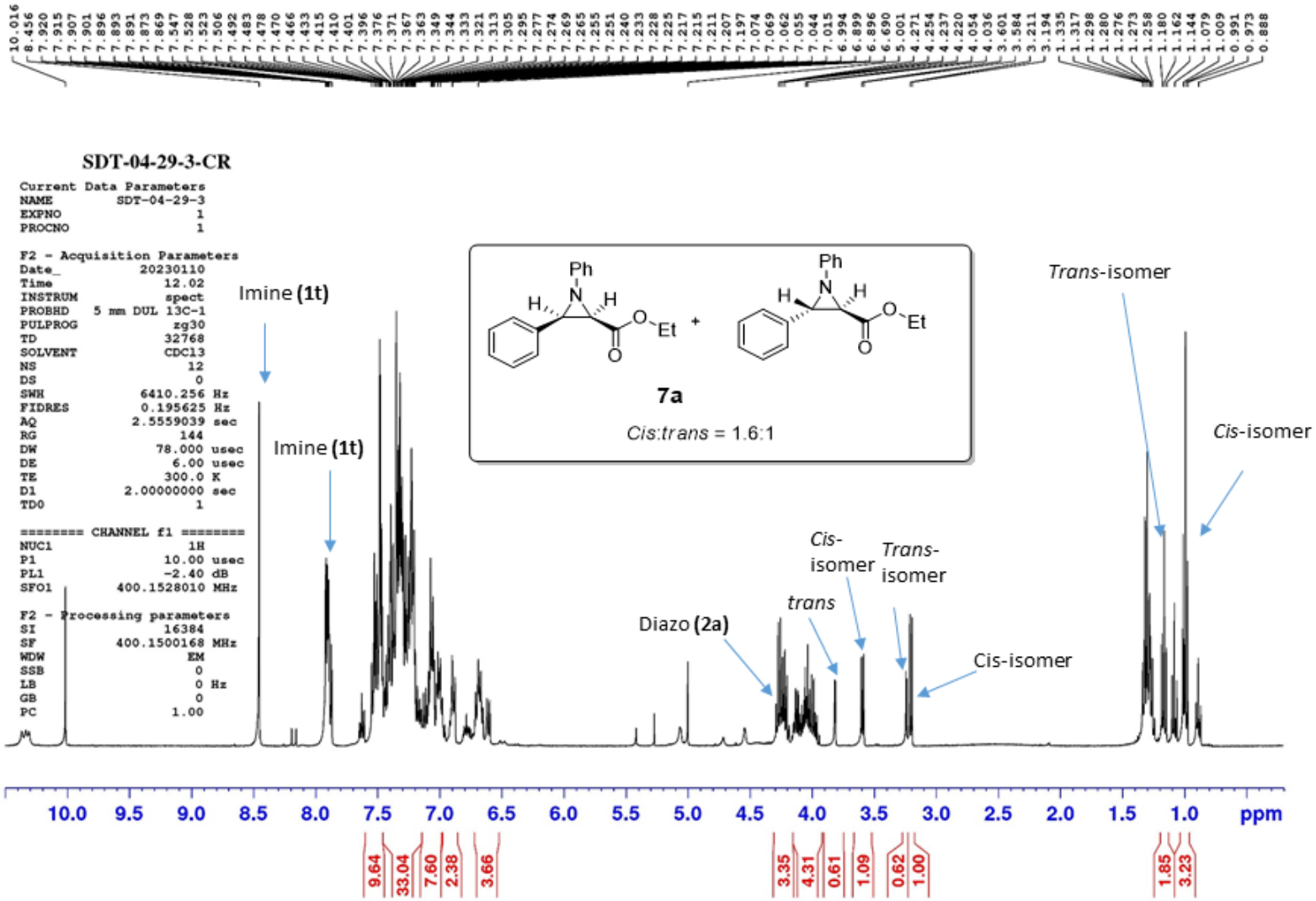


Solvent: $CDCl_3$
SFO1: 100 MHz

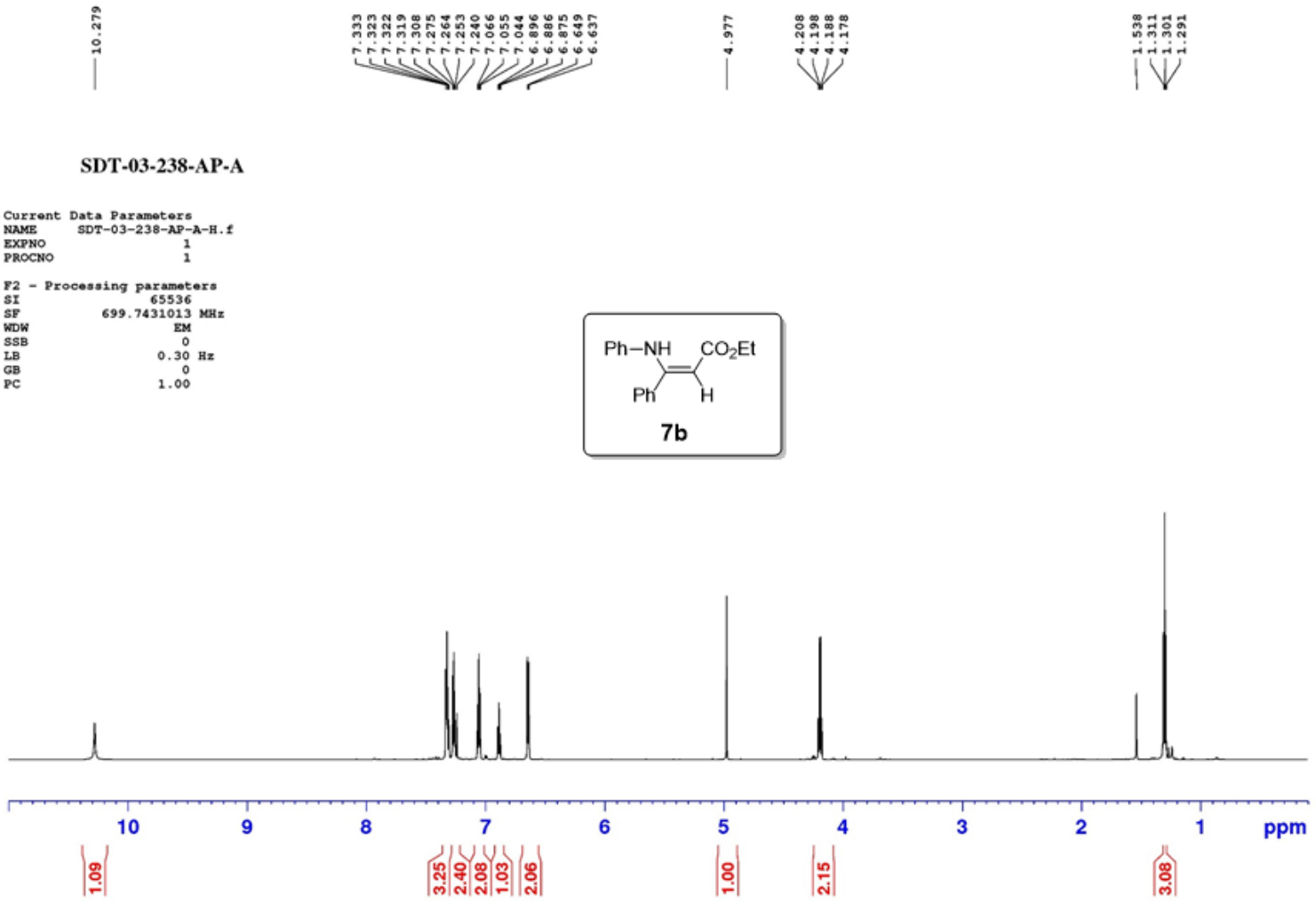


Solvent: $CDCl_3$
SFO1: 400 MHz

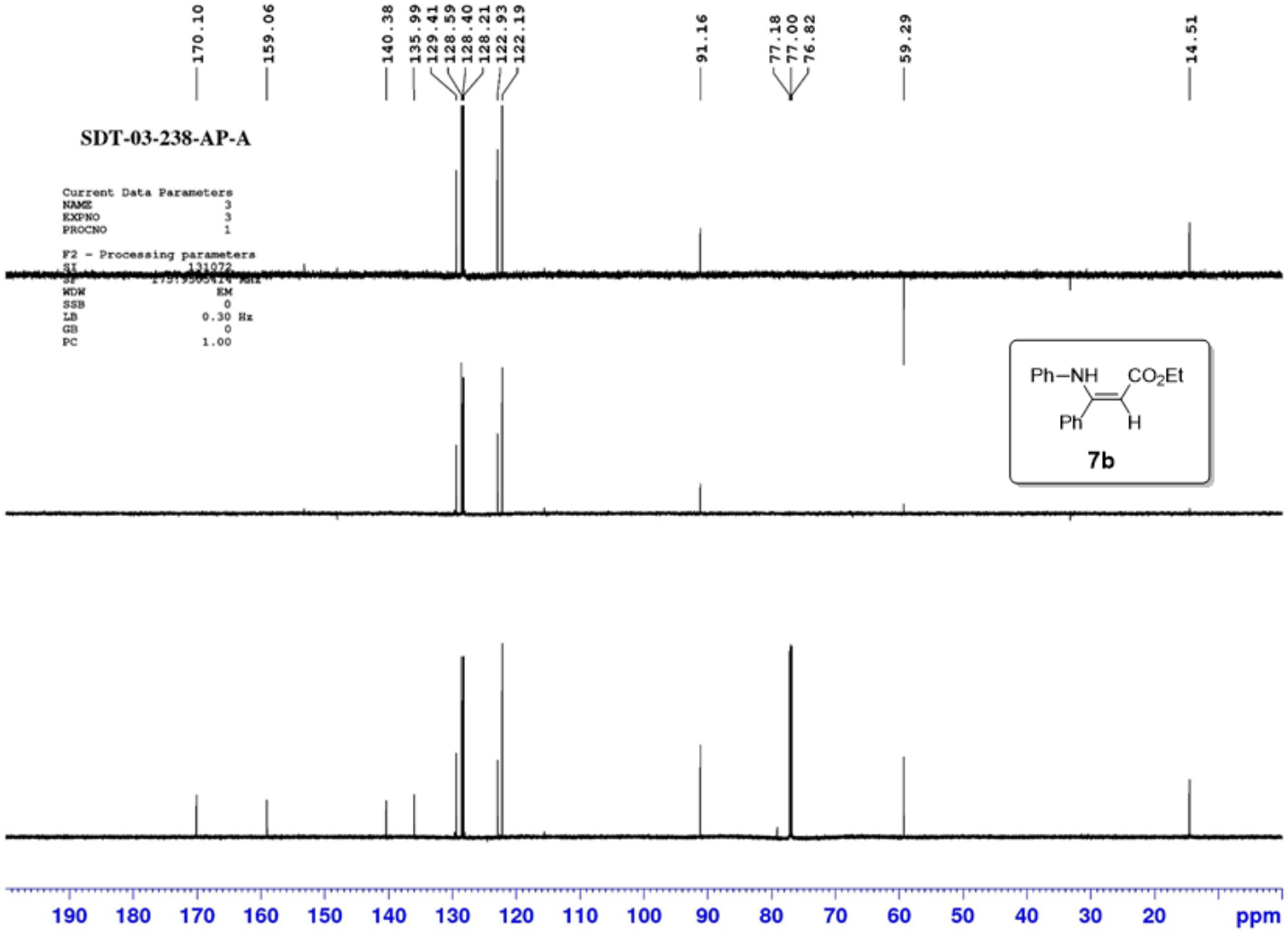
Jørgensen procedure Crude ^{1}H NMR Data



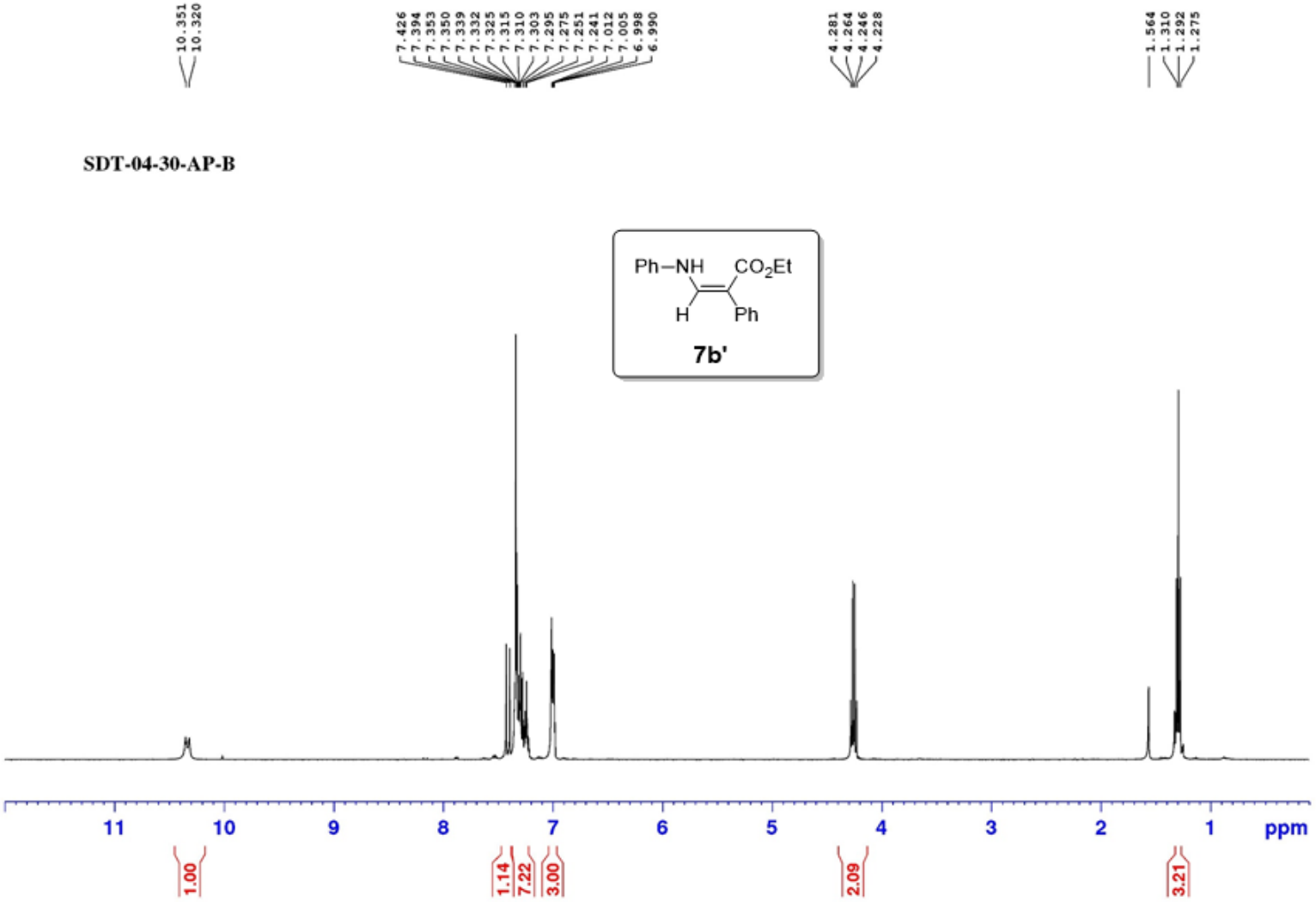
Solvent: $CDCl_3$
SFO1: 700 MHz



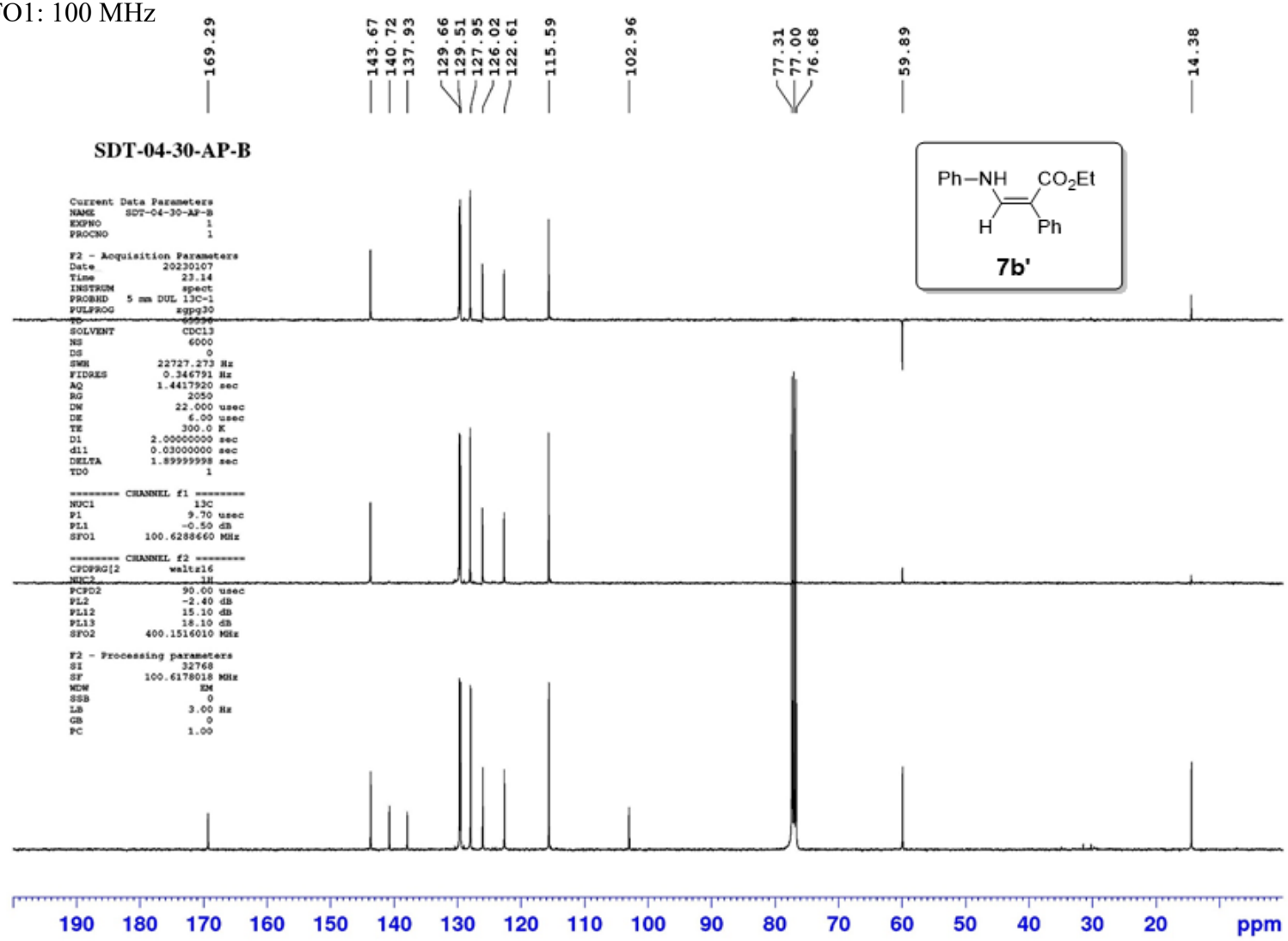
Solvent: $CDCl_3$
SFO1: 175 MHz



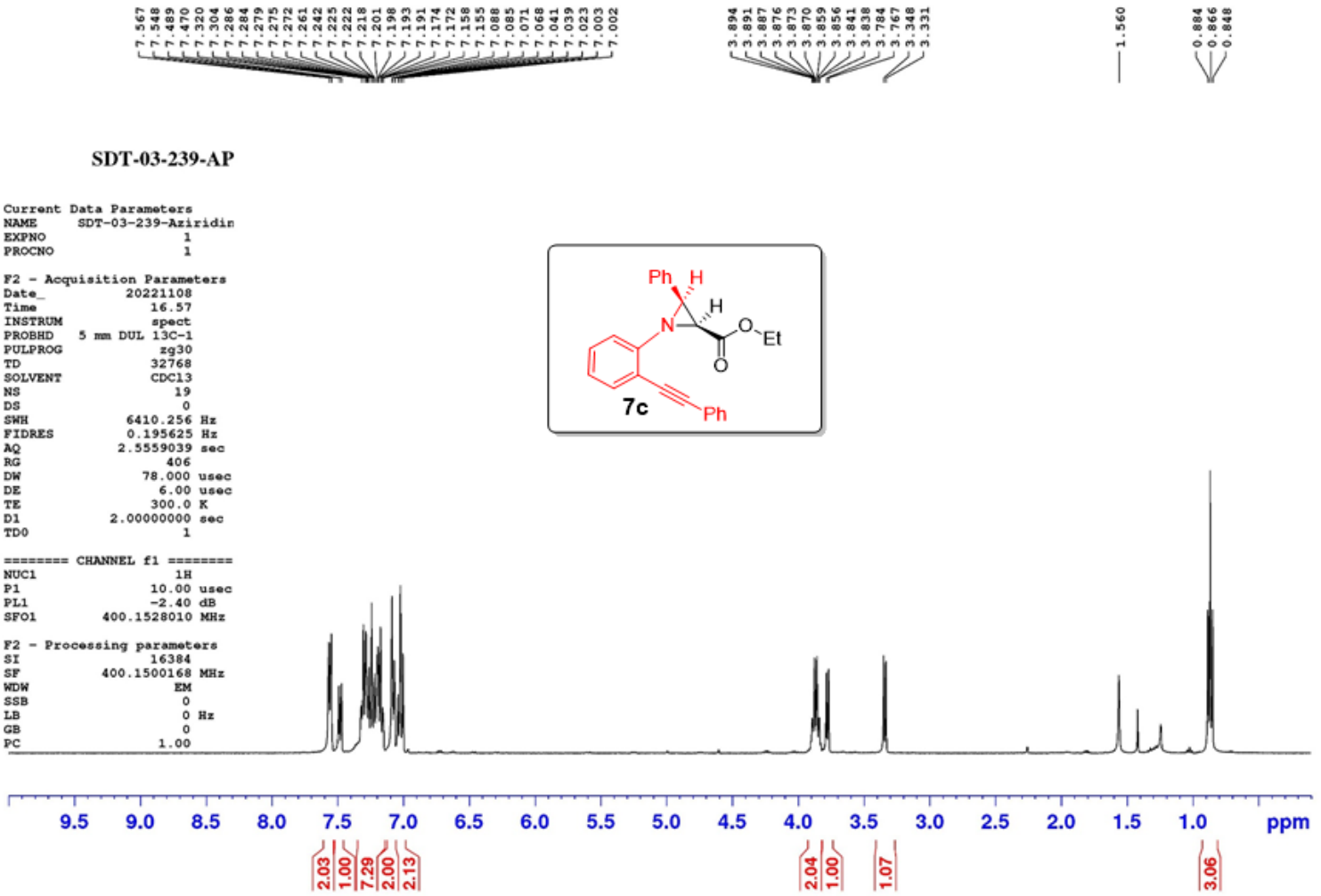
Solvent: $CDCl_3$
SFO1: 400 MHz



Solvent: $CDCl_3$
SFO1: 100 MHz



Solvent: $CDCl_3$
SFO1: 400 MHz



Solvent: $CDCl_3$
SFO1: 100 MHz