Electronic Supplementary Material (ESI) for ChemComm.

Visible-Light-Induced Regioselective Synthesis of Polyheteroaromatic Compounds

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Supporting Information

Contents

General Considerations

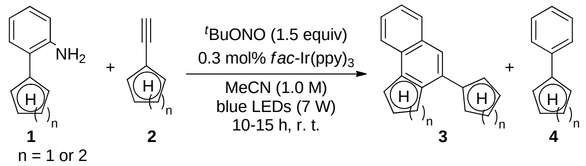
General Reagent Information

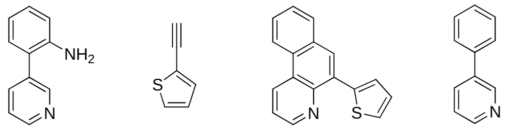
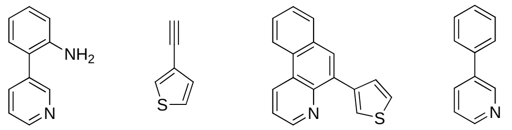
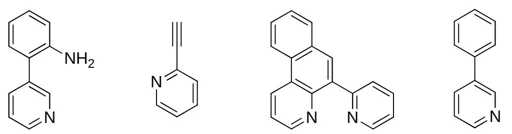
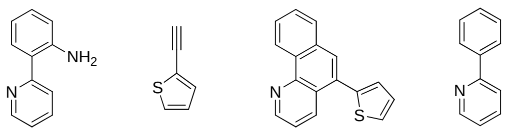
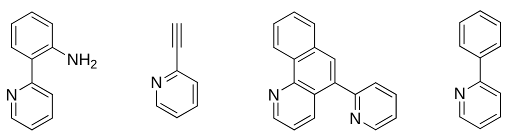
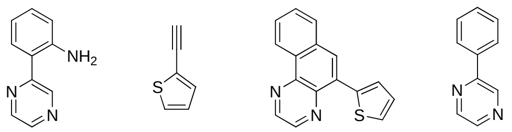
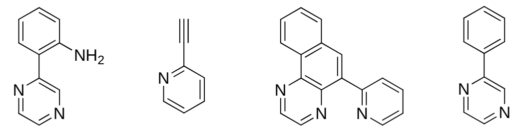
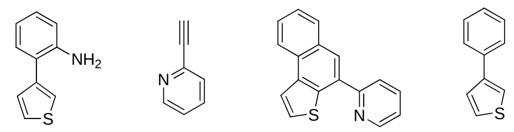
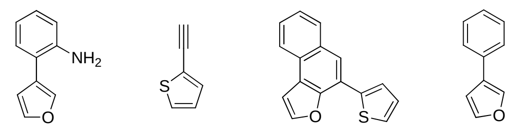
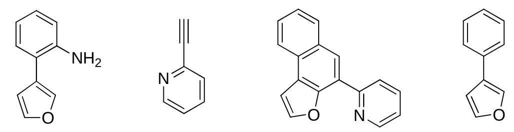
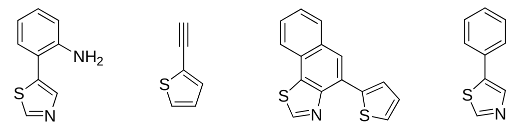
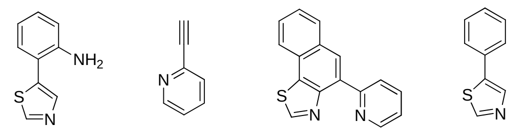
Acetonitrile was purchased from Sigma-Aldrich chemical company in Sure-Seal bottles and degassed by repeated sonication under light vacuum and replenishing the atmosphere with argon. *fac*-Ir(ppy)$_3$ was purchased from Sigma-Aldrich company. All other reagents were purchased from Sigma-Aldrich, Alfa Aesar, Acros Organics, or Combi-Blocks companies. Flash column chromatography was performed using Merck silica gel 60 (70-230 mesh).

General Analytical Information

The amino substrates and polyheteroaromatic products were characterized by ^{1}H, ^{13}C NMR, and FT-IR spectroscopy. NMR spectra were recorded on a Bruker 600 MHz instrument (600 MHz for ^{1}H NMR and 151 MHz for ^{13}C NMR). Copies of ^{1}H and ^{13}C NMR spectra can be found at the end of the Supporting Information. ^{1}H NMR experiments are reported in units, parts per million (ppm), and were measured relative to residual chloroform (7.26 ppm) in the deuterated solvent. ^{13}C NMR spectra are reported in ppm relative to deuterochloroform (77.23 ppm), and all were obtained with ^{1}H decoupling. Coupling constants were reported in Hz. FT-IR spectra were recorded on a Nicolet iS 10 ThermoFisher FT-IR spectrometer. Reactions were monitored by GC-MS using the Agilent GC 7890B/5977A inert MSD with Triple-Axis Detector. Mass spectral data of all unknown compounds were obtained from the Korea Basic Science Institute (Daegu) on a Jeol JMS 700 high resolution mass spectrometer or the organic chemistry research center (Sogang University) on a Bruker compact high resolution mass spectrometer. UV–vis spectra were recorded on a Scinco S-3100 spectrophotometer equipped with a Peltier temperature controller. Fluorescence spectra were obtained on a PTI QuantaMaster steady-state spectrofluorometer.

Table S1. Reaction scope including the yields of deaminated products[a]



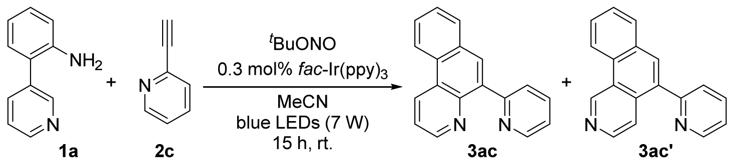
entry	amine (1)	alkyne (2)	polyheteroaromatics (3)	deaminated products (4)[b]
1	 1a	2a	3aa, 84%	4a, trace
2	 1a	2b	3ab, 80%[c]	4a, trace
3	 1a	2c	3ac, 75%[d]	4a, 14%
4	 1b	2a	3ba, 65%	4b, 11%
5	 1b	2c	3bc, 61%	4b, 19%
6	 1c	2a	3ca, 60%	4c, 11%
7	 1c	2c	3cc, 53%	4c, 20%
8	 1d	2c	3dc, 52%	4d, 12%
9	 1e	2a	3ea, 70%	4e, 10%
10	 1e	2c	3ec, 65%	4e, 15%
11	 1f	2a	3fa, 63%	4f, 20%
12	 1f	2c	3fc, 61%	4f, 20%

[a]Reaction scale: **1** (0.5 mmol), **2** (0.75 mmol); [b]yields were determined by gas chromatography; [c]regioisomer ratio (91:9); [d]regioisomer ratio (**3ac**:**3ac'** = 73:27).

Concentration dependence on regioselectivity of the reaction between 1a and 2c

The conditions that give increased levels of regioselectivity in the reaction between **1a** and **2c** were further identified by determining the effects of reagent concentrations and stoichiometries (Table *S2*). Decreasing the reactant concentrations from 1.0 to 0.25 M significantly increased the **3ac**:**3ac'** regioisomer ratio from 73:27 to 92:8 (entries 1–4). An increase in the amount of ^{t}BuONO did not affect the reaction outcome, but increasing the amount of alkyne **2c** to 3 equiv. improved the overall yield (77%) while retaining a high level of regioselectivity (entries 5–7). Because regioisomers **3ac** and **3ac'** can be readily separated by column chromatography, both sets of conditions (entries 1 and 7) can be used to synthesize **3ac** and **3ac'**.

***Table S2.* Concentration dependence on regioselectivity**[a]



entry	concentration	^{t}BuONO (equiv)	**2c** (equiv)	ratio[b] (**3ac** : **3ac'**)	yield (%)[c] (**3ac** + **3ac'**)
1	**1.0 M**	**1.5**	**1.5**	**73 : 27**	**75**
2	2.0 M	1.5	1.5	72 : 28	76
3	0.5 M	1.5	1.5	91 : 9	51
4	0.25 M	1.5	1.5	92 : 8	28
5	0.5 M	3.0	1.5	91 : 9	55
6	0.5 M	3.0	3.0	91 : 9	76
7	**0.5 M**	**1.5**	**3.0**	**91 : 9**	**77**

[a]Reaction scale: **1a** (0.1 mmol); [b,c]regioisomer ratios and yields were determined using gas chromatography.

We speculate that the regioselectivity and its dependence on reaction concentration may arise from the stabilization of intermediate vinyl radicals by *intramolecular* and *intermolecular* H-bond formations. Addition of 2-(3-pyridyl) aryl radical (**9a**) to 2-ethynyl pyridine (**2c**) produces the corresponding vinyl radical as represented by either **13** or **13'** (Figure *S1*). The vinyl radical can have different conformations by C-C single bond rotations, among which **13** and **13'** are lower-energy conformations due to *intramolecular* H-bonding between pyridine N and pyridyl or vinyl C-H as indicated by dotted lines in Figure *S1*. In general, conformer **13** is likely to be more stable than **13'** because there are two *intramolecular* H-bonding interactions in **13** while **13'** can have one *intramolecular* H-bonding. Therefore, the radical cyclization is likely to be more favorable at the C-2 of pyridine ring compared to the C-4, always giving **3ac** as a major isomer regardless of the reaction concentration. However, the ratio of **3ac** and **3ac'** is reduced at high reaction concentrations, possibly, due to additional stabilization of **13'**, an intermediate to **3ac'**, by *intermolecular* H-bonding as shown in Figure *S1*.

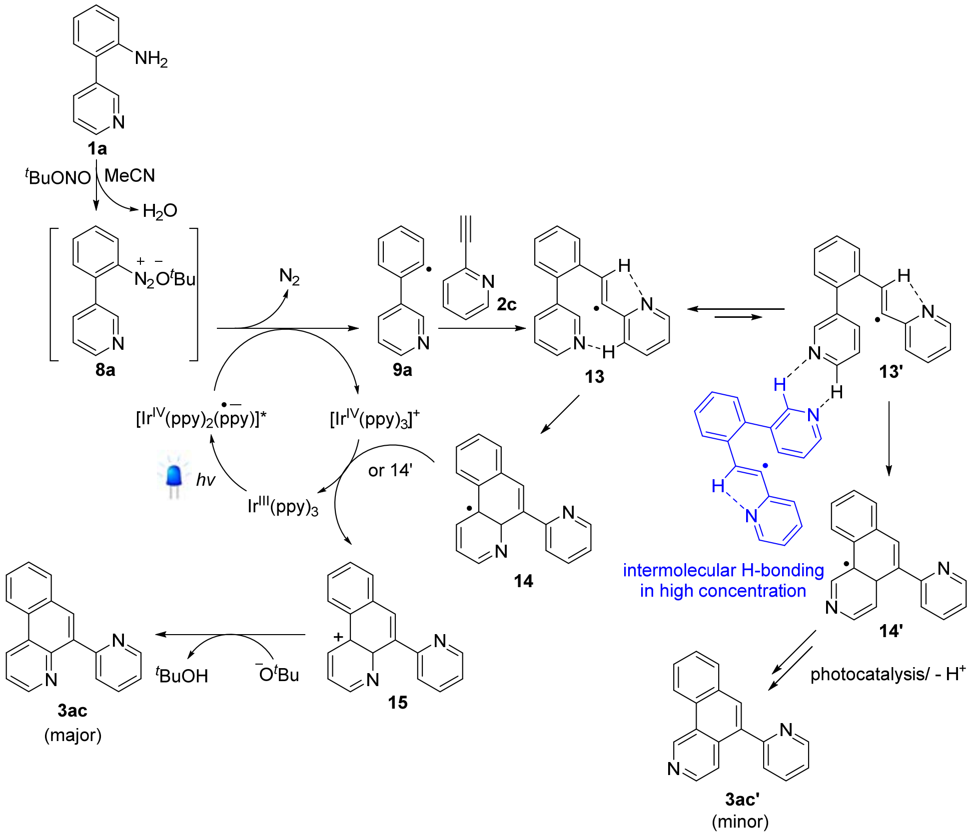
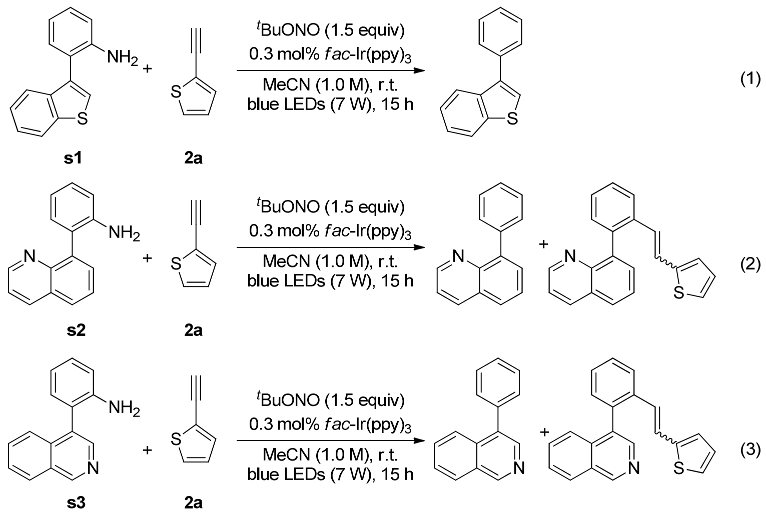


Figure S1. **Interpretation of the regioselectivity for the formation of 3ac and 3ac'.**

Reactions of Benzo-fused Heterocycle-Substituted Anilines

(Benzo[*b*]thiophen-3-yl)aniline (**s1**) is not a suitable substrate for this process, because its reaction with **2a** produces only the deamination product [Scheme *S1*.(1)]. Similarly, reactions of 2-(quinolin-8-yl)aniline (**s2**) and 2-(isoquinolin-4-yl)aniline (**s3**) with **2a** do not yield the desired annulated products, but rather the respective deamination and mono-addition products are formed exclusively [Scheme *S1*.(2) and (3)].

***Scheme S1*. Additional Reactions of Benzo-fused Heterocycle-Substituted Anilines**

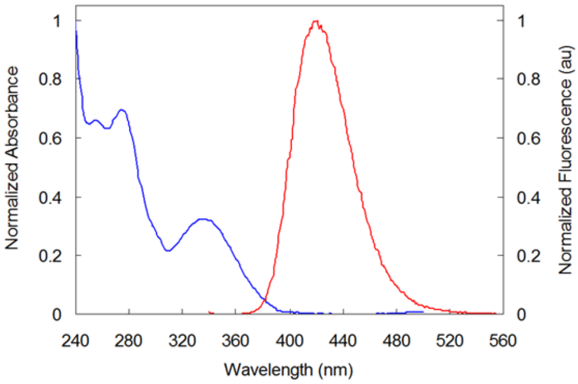


Photophysical Properties of Polyheteroaromatic Compounds (3):

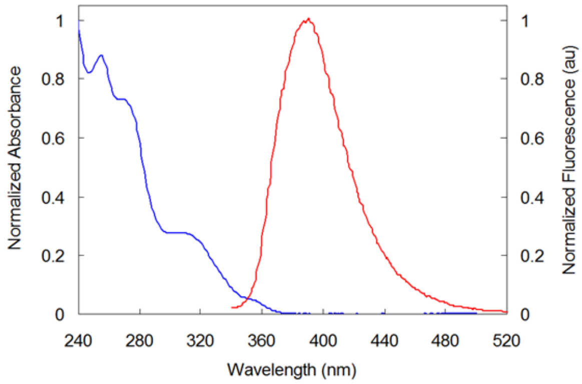
***Table S3*. Absorption and Emission Spectroscopic Data of Compounds 3**

	UV-vis		Fluorescence	
	λ (nm)	ε ($M^{-1}cm^{-1}$)	λ (nm)	Φ (quantum yield)
3aa	255, 274, 335	24000, 25300, 11800	421	**0.16**
3ab	255, 269, 304	33400, 28900, 10600	391	0.022
3ac	248, 270, 296, 351	46500, 27500, 11800, 2400	360, 377	0.006
3ac'	252, 300	35000, 9500	357, 369, 373	0.011
3ba	253, 271, 308, 351	33800, 40800, 12500, 3000	395	0.005
3bc	249, 269, 297, 334, 351	26100, 24600, 9300, 2600, 2200	355, 373	0.058
3ca	257, 289, 335	27300, 32200, 9700	485	**0.35**
3cc	244, 279, 366	25000, 19100, 3500	403	0.008
3dc	250, 322	35600, 14200	380	0.022
3ea	244, 276, 326	22200, 25400, 20200	359, 375	**0.36**
3ec	257, 309, 335	40700, 17800, 8200	365, 379	**0.22**
3fa	288, 298, 341	8800, 8600, 9800	385	0.003
3fc	265, 285, 342	39900, 27300, 2200	359, 375, 393	0.01
3ga	248, 281, 304, 354	36700, 33000, 14700, 3300	391	0.015

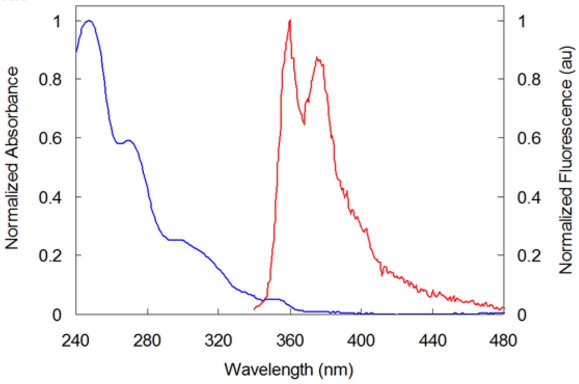
3aa



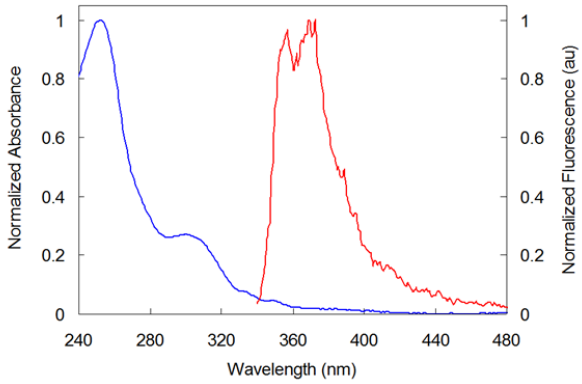
3ab



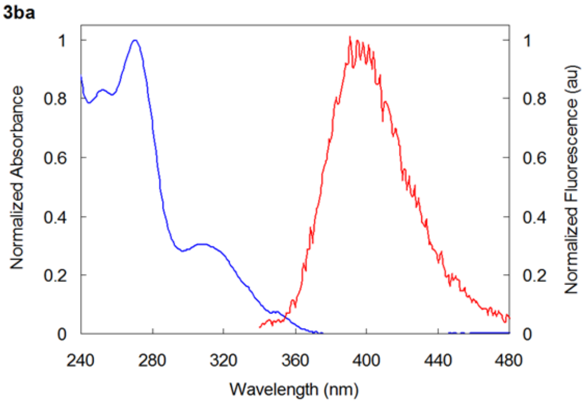
3ac



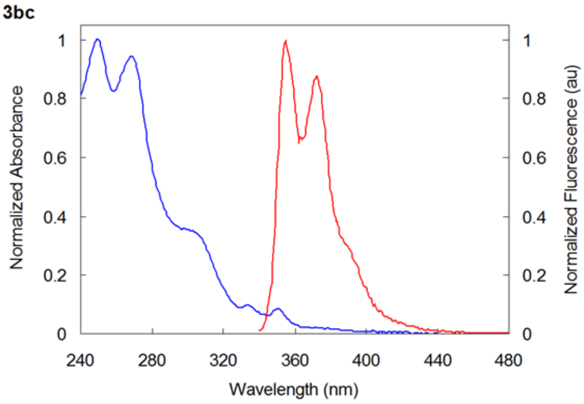
3ac'



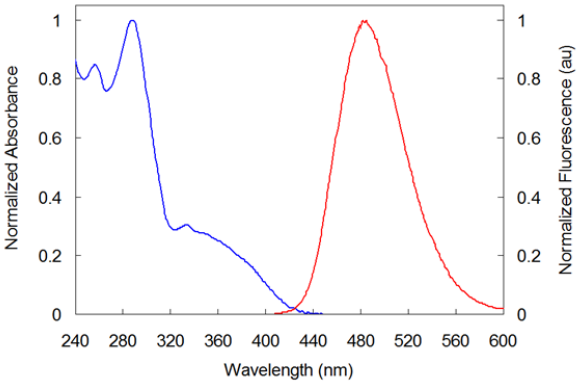
3ba



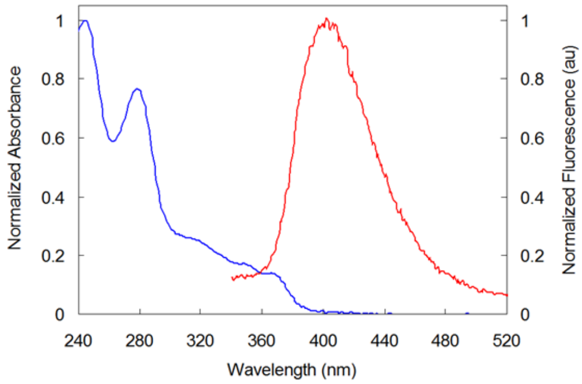
3bc



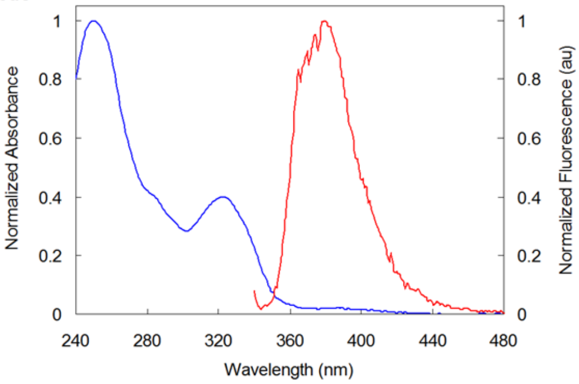
3ca



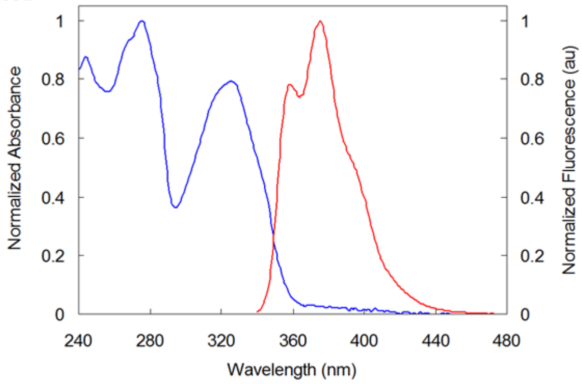
3cc



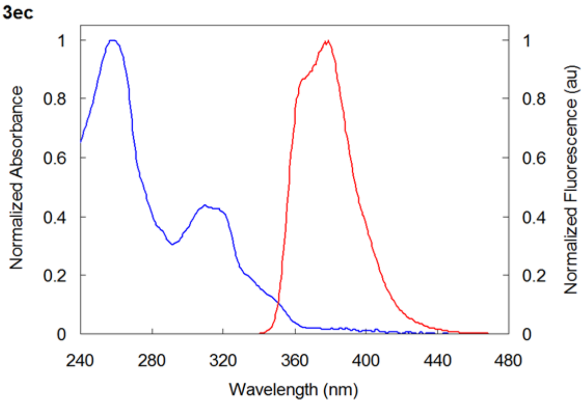
3dc



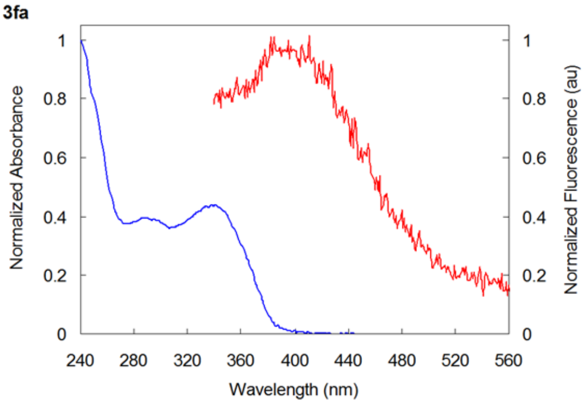
3ea



3ec



3fa



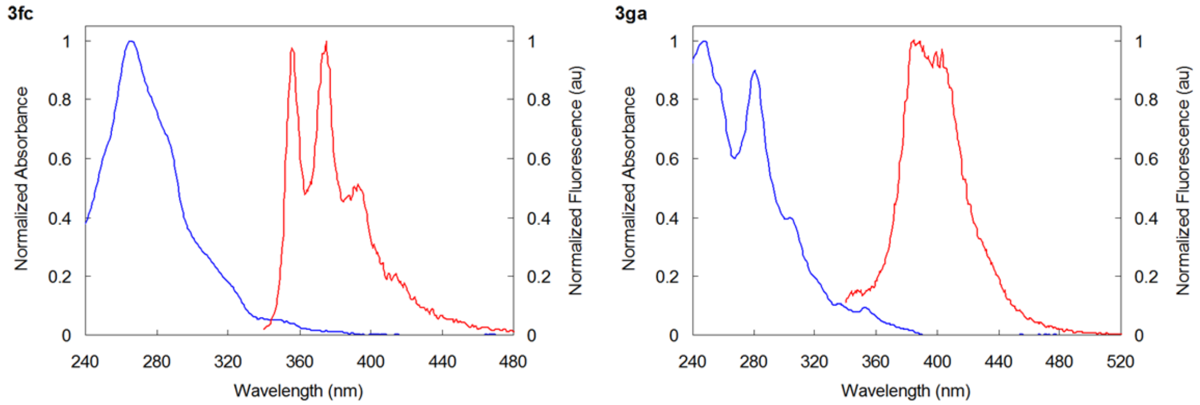
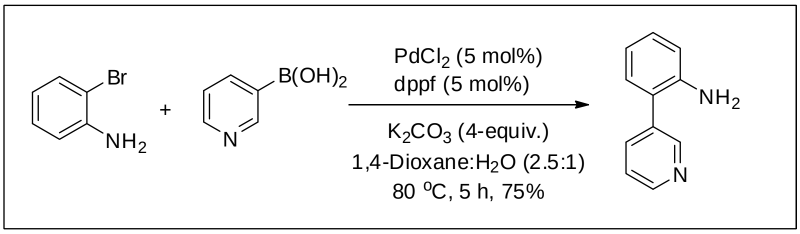


Figure S2. **Absorption and Emission Spectra of Compounds 3. [compounds 3] = 1.0×10^{-5} M in dichloromethane. λ_{ex} = 300 nm. Red and blue lines are absorbance and fluorescence emission spectra, respectively.**

Experimental Details

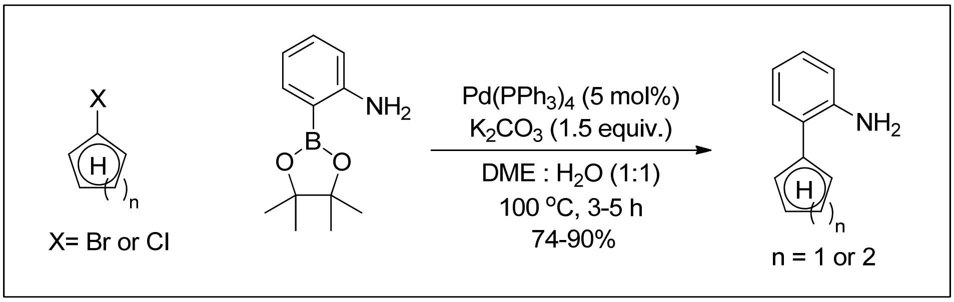
Experimental Procedures for the Synthesis of Amino Substrates:

Synthesis of 2-(pyridin-3-yl)aniline, 1a:



The synthesis of **1a** followed a previously reported procedure.[1] An anhydrous solution of $PdCl_2$ (44 mg, 5 mol%) and dppf (139 mg, 5 mol%) in 25 mL of dioxane under an argon atmosphere was stirred at room temperature for 10 min. 2-Boromoaniline (860 mg, 5 mmol), 3-pyridineboronic acid (800 mg, 1.3 equiv.), K_2CO_3 (2.76 g, 4 equiv.) and H_2O (10 mL) were then added and the resulting mixture was stirred at 80 °C for 5 h, cooled to room temperature, and diluted with a 1.5 N aq. NaOH and 30 mL of CH_2Cl_2. The aqueous phase was extracted with 3 × 30 mL of CH_2Cl_2. The combined CH_2Cl_2 extracts were dried over $MgSO_4$ and concentrated in vacuum to afford brown oil that was subjected to column chromatography on silica gel (hexanes/EtOAc 4:1 (v/v)) to afford 2-(pyridin-3-yl)aniline (**1a**) as a brown solid (637 mg, 75%).

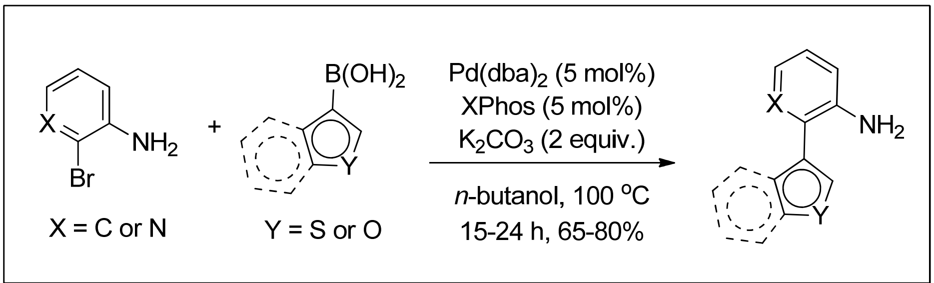
Synthesis of 1b, 1c, 1f, s2, and s3:



The synthesis of **1b** followed a previously reported procedure.[2] To 2-bromopyridine (790 mg, 5 mmol) in DME–H_2O (1:1, 18 mL) at 23 ºC were added K_2CO_3 (1.35 g, 1.50 equiv), 2-aminophenylboronic acid pinacol ester (1.97 g, 1.00 equiv), and tetrakis(triphenylphosphine)-palladium (289 mg, 5.00 mol%). The reaction mixture was stirred at 100 °C for 3.0 h. After cooling to 23 ºC, the phases were separated and the aqueous phase was extracted with EtOAc (3 × 30 mL). The combined organic phases were washed with brine (30 mL) and dried ($MgSO_4$). The filtrate was concentrated in vacuo and the residue was purified by chromatography on silica gel eluting with hexanes/EtOAc 4:1 (v/v) to afford 2-(pyridin-2-yl)aniline (**1b**) as a brown viscous liquid (722 mg, 85%).

The same experimental procedure was applied to synthesize 2-(pyrazin-2-yl)aniline (**1c**) from 2-chloro pyrazine (90%), 2-(thiazol-5-yl)aniline (**1f**) from 5-bromothiazole (74%), 2-(quinolin-8-yl)aniline (**s2**) from 8-bromoquinoline (80%), and 2-(isoquinolin-4-yl)aniline (**s3**) from 4-bromoisoquinoline (78%).

Synthesis of 1d, 1e, 1g, s1, and 5:



The synthesis of **1d** followed a previously reported procedure.[3] An anhydrous solution of $Pd(dba)_2$ (143 mg, 5 mol%) and XPhos (119 mg, 5 mol%) in 10 mL of *n*-butanol under an argon atmosphere was stirred at room temperature for 10 min. 2-Boromoaniline (860 mg, 5 mmol), thiophene-3-ylboronic acid (960 mg, 1.5 equiv.), and K_2CO_3 (1.38 g, 2 equiv.) were then added and the resulting mixture was stirred at 100 °C for 16 h. After cooling to room temperature, the crude reaction mixture was extracted with EtOAc (3 × 30 mL). The combined organic phases were washed with brine (30 mL) and dried ($MgSO_4$). The filtrate was concentrated in vacuo and the residue was purified by chromatography on silica gel eluting with hexanes/EtOAc 8:1 (v/v) to afford 2-(thiophen-3-yl)aniline (**1d**) as a brown solid (673 mg, 77%).

The same experimental procedure was applied to synthesize 2-(furan-3-yl)aniline (**1e**) in 80% yield using furan-3-ylboronic acid as the coupling partner, 2-(benzo[*b*]thiophen-2-yl)aniline (**1g**) in 70% yield using benzo[*b*]thiophen-2-ylboronic acid as the coupling partner, 2-(benzo[*b*]thiophen-3-yl)aniline (**s1**) in 77% yield using benzo[*b*]thiophen-3-ylboronic acid as the coupling partner and 2-(furan-3-yl)pyridin-3-amine (**5**) in 65% yield using 3-amino-2-bromopyridine and furan-3-ylboronic acid as the starting materials.

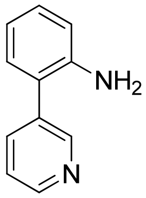
Experimental Procedure for the Synthesis of Polyheteroaromatics

Representative experimental procedure: the synthesis of 3aa

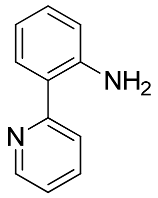
An oven-dried tube equipped with a magnetic stir bar was charged with **1a** (85 mg, 0.5 mmol, 1.0 equiv.), **2a** (81 mg, 0.75 mmol, 1.5 equiv.), *fac*-Ir(ppy)$_3$ (1 mg, 0.3 mol%) in 0.5 mL MeCN (1.0 M). Then ^{t}BuONO (77 mg, 0.75 mmol, 1.5 equiv.) was added to the mixture and the tube was sealed with a silicone septum screw cap and then placed near blue LEDs (7 W) at room temperature. Reaction progress was monitored by using both TLC and gas chromatography. After complete disappearance of **1a**, the mixture was diluted with dichloromethane (DCM) and filtered. The filtrate was concentrated in vacuo giving a residue that was subjected to flash column chromatography (SiO_2) (hexane/ethyl acetate) to furnish pure 5-(thiophen-2-yl)benzo[*f*]quinoline, **3aa** (109 mg, 84%).

Analytic Data for Synthesized Compounds:

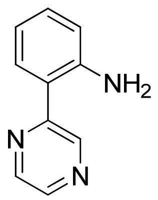
2-Heteroaryl Substituted Amino Substrates (1a-1g, s1-s3, and 5):



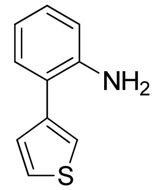
2-(Pyridin-3-yl)aniline, **1a**:[1] Brown solid, m. p. 42.2-43.7 °C; **^{1}H NMR (600 MHz, CDCl$_3$)** δ 8.73 (s, 1H), 8.61 (s, 1H), 7.80 (d, *J* = 7.8 Hz, 1H), 7.40-7.35 (m, 1H), 7.19 (ddd, *J* = 7.8, 7.2, 1.8 Hz, 1H), 7.10 (dd, *J* = 7.8, 1.8 Hz, 1H), 6.85 (dd, *J* = 7.2, 1.2 Hz, 1H), 6.78 (dd, *J* = 7.8, 1.2 Hz, 1H), 3.70 (bs, 2H); **^{13}C NMR (151 MHz, CDCl$_3$)** δ 150.22, 148.56, 143.93, 136.70, 135.49, 130.74, 129.53, 123.92, 123.78, 119.10, 116.05; **IR (neat)**: ν_{max} = 3327, 3203, 3025, 1616, 1497, 1406, 1297, 746 cm^{-1}; ***R$_f$*** 0.30 (hex/EtOAc, 1/2).



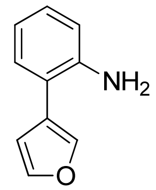
2-(Pyridin-2-yl)aniline, **1b**:[2] Brown viscous liquid; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.56 (d, *J* = 4.8 Hz, 1H), 7.67 (dd, *J* = 8.4, 1.8, 1H), 7.59 (d, *J* = 7.8 Hz, 1H), 7.48 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.13 (dd, *J* = 8.4, 1.8, 1H), 7.10 (ddd, *J* = 7.2, 4.8, 1.2 Hz, 1H), 6.75 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 6.71 (d, *J* = 8.4 Hz, 1H) 5.70 (bs, 2H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 159.48, 147.88, 146.66, 136.93, 129.95, 129.46, 122.25, 122.23, 120.99, 117.64, 117.25; **IR (neat)**: ν_{max} = 3446, 3315, 1610, 1584, 1476, 1159, 747 cm^{-1}; ***R_f*** 0.33 (hex/EtOAc, 4/1).



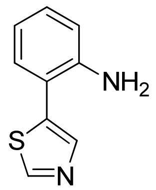
2-(Pyrazin-2-yl)aniline, **1c**:[4] Pale green solid; m. p. 58.5-60.1 °C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.99 (s, 1H), 8.52 (d, *J* = 2.4 Hz, 1H), 8.43 (d, *J* = 2.4 Hz, 1H),7.60 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.22 (ddd, *J* = 8.4, 7.2, 1.8 Hz, 1H), 6.81 (ddd, *J* = 7.8, 7.2, 1.2 Hz, 1H), 6.77 (dd, *J* = 8.4, 1.2 Hz, 1H), 5.69 (bs, 2H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 154.98, 147.35, 143.90, 142.01, 141.26, 131.17, 129.15, 118.67, 117.98, 117.62; **IR (neat)**: ν_{max} = 3446, 3315, 1610, 1584, 1476, 1425, 747 cm^{-1}; ***R_f*** 0.45 (hex/EtOAc, 2/1).



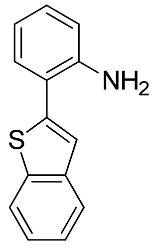
2-(Thiophen-3-yl)aniline, **1d**:[5] Brown solid; m. p. 36.8-37.6 °C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.43 (dd, *J* = 4.8, 1.8 Hz, 1H), 7.39 (s, 1H), 7.29 (dd, *J* = 4.8, 1.8 Hz, 1H), 7.23 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.16 (ddd, *J* = 8.4, 7.8, 1.8 Hz, 1H), 6.83 (ddd, *J* = 8.4, 7.8, 1.2 Hz, 1H), 6.78 (dd, *J* = 7.8, 1.2 Hz, 1H), 3.86 (bs, 2H).; **^{13}C NMR (151 MHz, $CDCl_3$)** δ 144.06, 139.99, 130.36, 128.64, 128.54, 126.16, 122.65, 122.62, 118.71, 115.86; **IR (neat)**: ν_{max} = 3444, 3361, 3099, 1615, 1489, 750 cm^{-1}; ***R_f*** 0.56 (hex/EtOAc, 4/1).



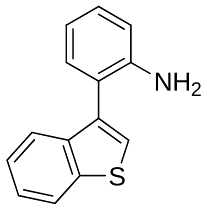
2-(Furan-3-yl)aniline, **1e**:[6] Brown oil; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.81 (s, 1H), 7.64-7.38 (m, 1H), 7.38 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.27 (dd, *J* = 7.2 Hz, 1.8 Hz, 1H), 6.96 (dd, *J* = 7.2, 1.2 Hz, 1H), 6.85 (dd, *J* = 7.8, 1.2 Hz, 1H), 6.82 – 6.76 (m, 1H), 3.95 (bs, 2H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 144.07, 143.16, 139.67, 129.64, 128.29, 123.46, 118.56, 118.21, 115.73, 110.76; ***R_f*** 0.50 (hex/EtOAc, 4/1).



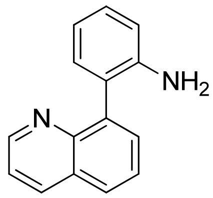
2-(Thiazol-5-yl)aniline, **1f:** Red-brown oil; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.84 (s, 1H), 7.98 (s, 1H), 7.23 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.19 (dd, *J* = 7.8, 1.8 Hz, 1H), 6.81 (d, *J* = 8.4 Hz, 1H), 6.79 (d, *J* = 8.4 Hz, 1H), 3.92 (bs, 2H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 153.15, 144.54, 141.54, 135.90, 131.56, 130.12, 118.95, 116.29, 116.11; **IR (neat)**: ν_{max} = 3339, 3212, 3077, 1618, 1485, 872, 749 cm^{-1}; **HRMS** m/z (EI) calc. for $C_9H_8N_2S$ [M+] 176.0408, found 176.0409; ***R_f*** 0.55 (hex/EtOAc, 1/2).



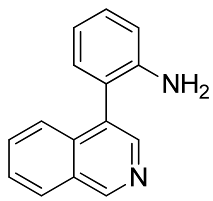
2-(Benzo[*b*]thiophen-2-yl)aniline, **1g**:[7] Off-white solid; m. p. 116.2-117.5 ^{o}C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.86 (d, *J* = 7.8 Hz, 1H), 7.80 (d, *J* = 7.2 Hz, 1H), 7.43 (s, 1H), 7.41–7.32 (m, 3H), 7.19 (ddd, *J* = 7.8, 7.2, 1.8 Hz, 1H), 6.84 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 6.81 (dd, *J* = 8.4, 1.2 Hz, 1H), 4.12 (bs, 2H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 144.46, 141.78, 140.57, 140.00, 131.28, 129.73, 124.66, 124.42, 123.64, 122.64, 122.32, 119.97, 118.81, 116.21; **IR (neat)**: ν_{max} = 3468, 3380, 3054, 1611, 1488, 1301, 742 cm^{-1}; ***R_f*** 0.52 (hex/EtOAc, 4/1).



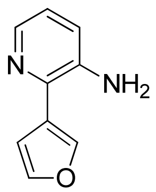
2-(Benzo[*b*]thiophen-3-yl)aniline, **s1**:[8] Brown viscous liquid; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.99 (dd, *J* = 7.2, 1.2 Hz, 1H), 7.72 (dd, *J* = 7.2, 1.2 Hz, 1H), 7.48 (s, 1H), 7.45-7.42 (m, 2H), 7.33 – 7.27 (m, 2H), 6.93 (ddd, *J* = 7.8, 7.2, 1.2 Hz 1H), 6.86 (dd, *J* = 7.8, 1.2 Hz, 1H), 3.71 (bs, 2H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 144.80, 140.43, 138.31, 135.03, 131.24, 129.18, 124.81, 124.67, 124.39, 123.54, 122.92, 121.02, 118.36, 115.62; **IR (neat)**: ν_{max} = 3377, 3062, 1615, 1488, 1299, 762 cm^{-1}; ***R_f*** 0.60 (hex/EtOAc, 4/1).



2-(Quinolin-8-yl)aniline, **s2**: Red solid; m. p. 100.4-101.9 ^{o}C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.94 (d, *J* = 4.2 Hz, 1H), 8.21 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.85 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.71 (d, *J* = 7.2 Hz, 1H), 7.62 (dd, *J* = 8.4, 7.2 Hz, 1H), 7.41 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.27 – 7.19 (m, 2H), 6.91 (dd, *J* = 7.8, 7.2 Hz, 1H), 6.85 (d, *J* = 7.8 Hz, 1H), 3.76 (bs, 2H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 150.75, 146.35, 144.91, 139.54, 136.82, 131.87, 131.70, 128.99, 128.73, 128.02, 126.96, 126.92, 121.22, 118.97, 116.50; **IR (neat)**: ν_{max} = 3334, 3028, 1615, 1490, 1295, 965, 745 cm^{-1}; ***R_f*** 0.58 (hex/EtOAc, 1/2).

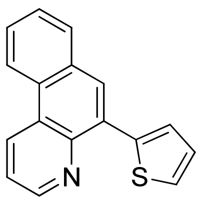


2-(Isoquinolin-4-yl)aniline, **s3**:[9] Brown solid, m. p. 114.4-116.1 ^{o}C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 9.26 (s, 1H), 8.49 (s, 1H), 8.03 (d, *J* = 8.4 Hz, 1H), 7.71 – 7.56 (m, 3H), 7.27 (ddd, *J* = 7.8, 7.2, 1.8 Hz, 1H), 7.15 (dd, *J* = 7.8, 1.8 Hz, 1H), 6.88 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 6.84 (dd, *J* = 8.4, 1.2 Hz, 1H), 3.53 (bs, 2H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 152.54, 144.83, 143.82, 134.56, 131.58, 130.83, 130.53, 129.60, 128.60, 128.06, 127.58, 125.28, 122.02, 118.58, 115.64; **IR (neat)**: ν_{max} = 3326, 3199, 3027, 1616, 1490, 1300, 749 cm^{-1}; ***R_f*** 0.43 (hex/EtOAc, 1/2).

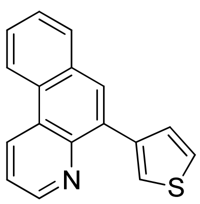


2-(Furan-3-yl)pyridin-3-amine, **5**: Pale yellow solid, m. p. 53.8-55.4 °C; **^{1}H NMR (600 MHz, CDCl3)** δ 8.01 (dd, *J* = 4.2, 1.8 Hz, 1H), 7.87 (s, 1H), 7.45 (dd, *J* = 4.2, 1.8 Hz, 1H), 6.97 – 6.87 (m, 3H), 3.92 (bs, 2H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 143.21, 140.76, 140.41, 139.79, 138.06, 124.64, 122.89, 122.47, 110.34; **IR (neat)**: ν_{max} = 3326, 1617, 1457, 1158, 801, 761 cm^{-1}; **HRMS** m/z (EI) calc. for $C_9H_8N_2O$ [M+] 160.0637, found 160.0639; $\boldsymbol{R_f}$ 0.63 (hex/EtOAc, 1/2).

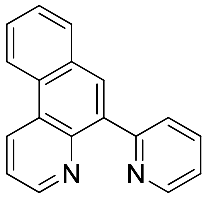
Polyheteroaromatic Compounds (3 and 6):



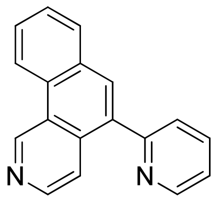
5-(Thiophen-2-yl)benzo[*f*]quinoline, **3aa:** Off-white solid; m. p. 83.9-85.0 °C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 9.06 (d, *J* = 3.6 Hz, 1H), 8.95 (d, *J* = 8.4 Hz, 1H), 8.55 (d, *J* = 7.2 Hz, 1H), 8.31 (s, 1H), 7.93 (d, *J* = 7.2 Hz, 1H), 7.88 (d, *J* = 3.6 Hz, 1H), 7.65-7.63 (m, 2H), 7.58 (dd, *J* = 8.4, 3.6 Hz, 1H), 7.53 (d, *J* = 4.8 Hz, 1H), 7.23 (dd, *J* = 4.8, 3.6 Hz, 1H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 148.76, 145.42, 139.98, 131.62, 131.56, 131.05, 129.29, 129.06, 128.98, 128.11, 127.76, 127.17, 126.99, 126.71, 125.89, 122.53, 121.50; **IR (neat)**: ν_{max} = 2926, 1421, 1264, 896, 731 cm^{-1}; **HRMS** m/z (ESI) calc. for $C_{17}H_{12}NS$ [M+1] 262.0685, found 262.0686; $\boldsymbol{R_f}$ 0.68 (hex/EtOAc, 2/1).



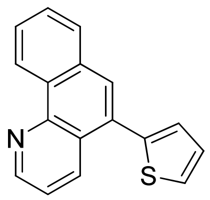
5-(Thiophen-3-yl)benzo[*f*]quinoline, **3ab:** Off-white solid; m. p. 123.9-124.5 °C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 9.03 (d, *J* = 4.2 Hz, 1H), 9.01 (d, *J* = 8.4 Hz, 1H), 8.62 (d, *J* = 7.8 Hz, 1H), 8.14 (s, 1H), 7.95 (d, *J* = 7.8 Hz, 2H), 7.71 (d, *J* = 4.2 Hz, 1H) 7.70 – 7.63 (m, 2H), 7.59 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.47 (dd, *J* = 4.2, 2.4 Hz, 1H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 149.46, 146.76, 139.90, 133.86, 131.72, 131.04, 130.56, 130.09, 129.57, 128.93, 127.73, 127.15, 126.04, 124.94, 124.71, 122.61, 121.39; **IR (neat)**: ν_{max} = 3070, 2918, 2849, 1589, 1485, 1381, 794, 749 cm^{-1}; **HRMS** m/z (ESI) calc. for $C_{17}H_{12}NS$ [M+1] 262.0685, found 262.0686; $\boldsymbol{R_f}$ 0.69 (hex/EtOAc, 2/1).



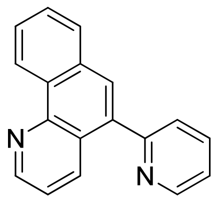
5-(Pyridin-2-yl)benzo[*f*]quinoline, **3ac:** White solid; m. p. 69.9-70.8 °C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 9.03 (dd, *J* = 8.4, 1.2 Hz, 1H), 8.99 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.82 (d, *J* = 4.2 Hz, 1H), 8.64 (d, *J* = 7.8 Hz, 1H), 8.41 (s, 1H), 8.09 (d, *J* = 7.8 Hz, 1H), 8.02 (d, *J* = 8.8 Hz, 1H), 7.83 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.71 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.67 (dd, *J* = 8.8, 1.2 Hz, 1H), 7.59 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.34 (ddd, *J* = 8.4, 4.2, 1.2 Hz, 1H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 157.41, 149.71, 149.46, 146.46, 137.43, 135.69, 132.71, 131.52, 131.16, 130.12, 129.63, 127.75, 127.68, 127.07, 126.06, 122.58, 122.45, 121.39; **IR (neat)**: ν_{max} = 3058, 1586, 1471, 799, 746 cm^{-1}; **HRMS** m/z (ESI) calc. for $C_{18}H_{13}N_2$ [M+1] 257.1073, found 257.1075; ***Rf*** 0.46 (hex/EtOAc, 1/4).



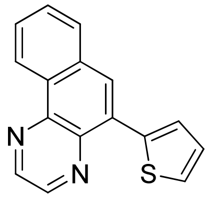
5-(Pyridin-2-yl)benzo[*h*]isoquinoline, **3ac′**: Off white solid; m. p. 63.4-64.1 °C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 10.14 (s, 1H), 8.88–8.81 (m, 2H), 8.70 (d, *J* = 5.4 Hz, 1H), 8.09 (s, 1H), 8.02 (d, *J* = 5.4 Hz, 1H), 7.99 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.90 (ddd, *J* = 9.6, 7.8, 1.8 Hz, 1H), 7.78 (ddd, *J* = 9.6, 7.8, 1.2 Hz, 1H), 7.70 (ddd, *J* = 9.0, 7.8, 1.2 Hz, 1H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.41 (ddd, *J* = 9.0, 7.8, 1.2 Hz, 1H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 157.99, 149.95, 147.25, 145.54, 137.08, 135.57, 134.59, 132.96, 131.73, 129.71, 129.60, 128.60, 127.97, 125.63, 125.09, 122.81, 122.09, 119.45; **IR (neat)**: ν_{max} = 3047, 2918, 2849, 1585, 1471, 1431, 746 cm^{-1}; **HRMS** m/z (ESI) calc. for $C_{18}H_{13}N_2$ [M+1] 257.1073, found 257.1075; $\boldsymbol{R_f}$ 0.30 (hex/EtOAc, 1/4).



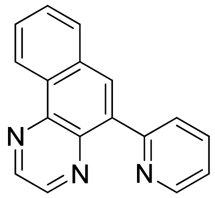
5-(Thiophen-2-yl)benzo[*h*]quinolone, **3ba**: Brown solid; m. p. 95.5-96.8 °C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 9.31 (d, *J* = 7.8 Hz, 1H), 9.03 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.55 (dd, *J* = 8.4, 1.8 Hz, 1H), 8.02–7.84 (m, 2H), 7.76 (ddd, *J* = 8.4, 7.8, 1.8 Hz, 1H), 7.71 (ddd, *J* = 8.4, 7.8, 1.8 Hz, 1H), 7.53 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.47 (d, *J* = 4.8 Hz, 1H), 7.27 (d, *J* = 4.2 Hz, 1H), 7.22 (dd, *J* = 4.8, 4.2 Hz, 1H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 148.99, 146.99, 140.73, 134.30, 132.99, 131.51, 130.11, 129.51, 128.80, 128.16, 127.97, 127.68, 127.64, 126.17, 125.95, 124.72, 122.01; **IR (neat)**: ν_{max} = 3062, 2918, 2850, 1722, 1397, 1260, 695 cm^{-1}; **HRMS** m/z (ESI) calc. for $C_{17}H_{12}NS$ [M+1] 262.0685, found 262.0686; $\boldsymbol{R_f}$ 0.73 (hex/EtOAc, 2/1).



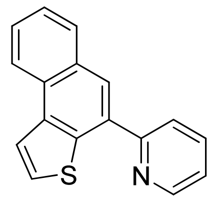
5-(Pyridin-2-yl)benzo[*h*]quinolone, **3bc**: Brown solid; m. p. 63.8-64.5 °C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 9.33 (d, *J* = 8.4 Hz, 1H), 9.03 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.82 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.54 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.99–7.91 (m, 2H), 7.89 (ddd, *J* = 9.6, 7.8, 1.8 Hz, 1H), 7.78 (ddd, J = 9.6, 8.4, 1.2 Hz, 1H), 7.73 (ddd, *J* = 8.4, 7.8, 1.2 Hz, 1H), 7.67 (d, *J* = 7.8 Hz, 1H), 7.52 (dd, *J* = 8.4, 4.8 Hz, 1H), 7.39 (ddd, *J* = 7.8, 4.8, 1.2 Hz, 1H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 158.37, 149.49, 148.74, 146.89, 136.86, 135.87, 134.28, 132.79, 131.62, 128.91, 128.90, 128.46, 128.17, 127.54, 124.94, 124.50, 122.36, 121.74; **IR (neat)**: ν_{max} = 3054, 2923, 2852, 1586, 1471, 1261, 748 cm^{-1}; **HRMS** m/z (ESI) calc. for $C_{18}H_{13}N_2$ [M+1] 257.1073, found 257.1075; $\boldsymbol{R_f}$ 0.33 (hex/EtOAc, 2/1).



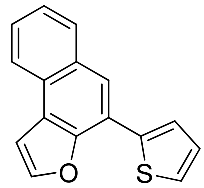
5-(Thiophen-2-yl)benzo[*f*]quinoxaline, **3ca**: Pale green solid,; m. p. 68.1-69.2 ^{o}C; ^{1}**H NMR (600 MHz, CDCl$_3$)** δ 9.20 (dd, *J* = 9.0, 2.4 Hz, 1H), 9.01 (d, *J* = 2.4 Hz, 1H), 8.97 (d, *J* = 2.4 Hz, 1H), 8.36 (s, 1H), 7.97 (dd, *J* = 9.0, 2.4 Hz, 1H), 7.87 (d, *J* = 3.6 Hz, 1H), 7.79 – 7.73 (m, 2H), 7.51 (d, *J* = 4.8 Hz, 1H), 7.22 (dd, *J* = 4.8, 3.6 Hz, 1H); ^{13}**C NMR (151 MHz, CDCl$_3$)** δ 143.49, 143.24, 142.19, 140.59, 139.07, 133.23, 130.80, 130.61, 129.85, 129.62, 128.31, 127.90, 127.80, 127.64, 127.17, 124.77; **IR (neat)**: ν_{max} = 3054, 1264, 896, 731 cm^{-1}; **HRMS** m/z (EI) calc. for $C_{16}H_{10}N_2S$ [M+] 262.0565, found 262.0564; ***R$_f$*** 0.70 (hex/EtOAc, 2/1).



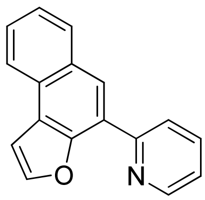
5-(Pyridin-2-yl)benzo[*f*]quinoxaline, **3cc**: Pale yellow solid,; m. p. 100.2-101.4 ^{o}C; ^{1}**H NMR (600 MHz, CDCl$_3$)** δ 9.25 (dd, *J* = 7.8, 1.8 Hz, 1H), 8.96 (dd, *J* = 9.6, 1.8 Hz, 2H), 8.84 (dd, *J* = 4.8, 1.8, Hz, 1H), 8.52 (s, 1H), 8.07 (dd, *J* = 8.4, 1.2 Hz, 1H), 8.05 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.85 (ddd, *J* = 9.6, 8.4, 1.8 Hz, 1H) 7.82–7.75 (m, 2H), 7.37 (ddd, *J* = 7.8, 4.8, 1.8 Hz, 1H); ^{13}**C NMR (151 MHz, CDCl$_3$)** δ 156.23, 149.90, 143.96, 143.15, 142.17, 141.32, 136.29, 135.97, 133.34, 133.06, 131.38, 129.54, 128.94, 128.26, 126.91, 124.74, 122.77; **IR (neat)**: ν_{max} = 3051, 2924, 2851, 1587, 1467, 1264, 731 cm^{-1}; **HRMS** m/z (EI) calc. for $C_{17}H_{11}N_3$ [M+] 257.0953, found 257.0955; ***R$_f$*** 0.80 (hex/EtOAc, 1/4).



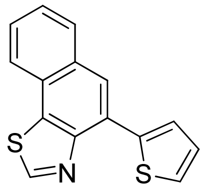
2-(Naphtho[2,1-*b*]thiophen-4-yl)pyridine, **3dc**: Brown solid, m. p. 111.3-112.2 ^{o}C; ^{1}**H NMR (600 MHz, CDCl$_3$)** δ 8.86 (d, *J* = 5.4 Hz, 1H), 8.40 (d, *J* = 8.4 Hz, 1H), 8.26 (s, 1H), 8.11 – 8.01 (m, 3H), 7.85 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.73 (d, *J* = 5.4 Hz, 1H), 7.65 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.56 (dd, *J* = 7.8, 6.6 Hz, 1H), 7.32 (dd, *J* = 7.8, 6.6 Hz, 1H); ^{13}**C NMR (151 MHz, CDCl$_3$)** δ 156.79, 148.85, 137.67, 136.90, 135.19, 132.31, 131.53, 129.81, 129.21, 127.24, 125.78, 123.86, 123.84, 122.61, 121.50, 121.50, 121.36; **IR (neat)**: ν_{max} = 3059, 2922, 2851, 1589, 1470, 1159, 720 cm^{-1}; **HRMS** m/z (ESI) calc. for $C_{17}H_{12}NS$ [M+1] 262.0685, found 262.0684; ***R$_f$*** 0.68 (hex/EtOAc, 2/1).



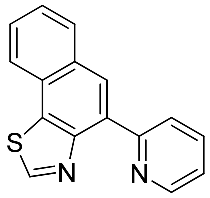
4-(Thiophen-2-yl)naphtho[2,1-*b*]furan, **3ea**: Pale green solid; m. p. 140.3-141.6 ^{o}C; ^{1}**H NMR (600 MHz, CDCl$_3$)** δ 8.12 (d, *J* = 8.4 Hz, 1H), 7.99 (s, 1H), 7.97 (d, *J* = 3.6 Hz, 1H), 7.96 (d, *J* = 4.8 Hz, 1H), 7.88 (d, *J* = 2.4 Hz, 1H), 7.57 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.50 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.42 (dd, *J* = 7.2, 1.2 Hz, 1H), 7.33 (d, *J* = 2.4 Hz, 1H), 7.22 (dd, *J* = 4.8, 3.6 Hz, 1H); ^{13}**C NMR (151 MHz, CDCl$_3$)** δ 149.47, 144.58, 138.89, 130.90, 128.86, 128.21, 127.28, 126.97, 126.46, 125.63, 125.27, 123.71, 123.46, 122.35, 120.29, 106.04; **IR (neat)**: ν_{max} = 3058, 2919, 2849, 1593, 1514, 1372, 1128, 744, 693 cm^{-1}; **HRMS** m/z (EI) calc. for $C_{16}H_{10}OS$ [M+] 250.0452, found 250.0455; $\boldsymbol{R_f}$ 0.40 (in hexane).



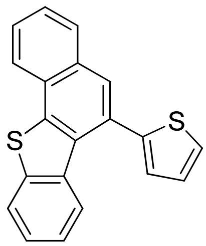
2-(Naphtho[2,1-*b*]furan-4-yl)pyridine, **3ec**: Yellow solid; m. p. 100.1-101.5 °C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.83 (d, *J* = 4.8 Hz, 1H) 8.56 (s, 1H), 8.39 (dd, *J* = 7.8, 1.2 Hz, 1H), 8.16 (dd, *J* = 8.4, 1.2 Hz, 1H), 8.08 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.87 (d, *J* = 2.4 Hz, 1H), 7.86 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.61 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.52 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.36 (d, *J* = 2.4 Hz, 1H), 7.32 (ddd, *J* = 7.2, 4.8, 1.2 Hz, 1H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 154.12, 150.42, 150.06, 144.36, 136.88, 130.89, 129.82, 128.25, 127.06, 125.45, 125.16, 124.95, 124.26, 124.03, 123.38, 122.81, 106.01; **IR (neat)**: ν_{max} = 3057, 2923, 2851, 1585, 1471, 1363, 1211, 742 cm^{-1}; **HRMS** m/z (ESI) calc. for $C_{17}H_{12}NO$ [M+1] 246.0913, found 246.0915; $\boldsymbol{R_f}$ 0.39 (hex/EtOAc, 2/1).



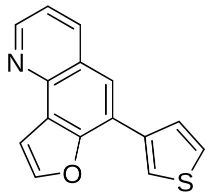
4-(Thiophen-2-yl)naphtho[2,1-*d*]thiazole, **3fa**: Brown solid, m. p. 160.1-162.2 °C (decomposed); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.60 (s, 1H), 7.52 (s, 1H), 7.40–7.36 (m, 2H), 7.29-7.25 (m, 3H), 7.05 (d, *J* = 3.6 Hz, 1H), 7.00 (dd, *J* = 4.8, 3.6 Hz, 1H); **^{13}C NMR (151 MHz, $CDCl_3$)** δ 174.15, 170.46, 142.71, 141.70, 138.40, 136.30, 130.05, 130.00, 128.23, 127.36, 126.16, 125.32, 124.40, 122.05 (Overlapped peaks present); **IR (neat)**: ν_{max} = 2923, 2853, 1584, 1457, 1129, 985, 698 cm^{-1}; $\boldsymbol{R_f}$ 0.63 (hex/EtOAc, 1/1).



4-(Pyridin-2-yl)naphtho[2,1-*d*]thiazole, **3fc**: Pale yellow solid, m. p. 121.3-122.4 °C; **^{1}H NMR (600 MHz, $CDCl_3$)** δ 9.14 (s, 1H), 8.82 (dd, *J* = 4.8, 1.2 Hz, 1H), 8.56 (s, 1H), 8.49 (dd, *J* = 7.8, 1.2 Hz, 1H), 8.13–8.05 (m, 2H), 7.87 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.63 (ddd, *J* = 7.8, 7.2, 1.8 Hz, 1H), 7.59 (ddd, *J* = 7.8, 7.2, 1.8 Hz, 1H), 7.33 (dd, *J* = 7.2, 4.8 Hz, 1H) ; **^{13}C NMR (151 MHz, $CDCl_3$)** δ 156.15, 152.34, 149.88, 149.48, 136.54, 133.17, 133.03, 131.60, 129.97, 128.41, 128.37, 127.73, 126.89, 125.72, 125.32, 122.77; **IR (neat)**: ν_{max} = 3046, 2922, 2851, 1586, 1261, 748 cm^{-1}; **HRMS** m/z (ESI) calc. for $C_{16}H_{11}N_2S$ [M+1] 263.0637, found 263.0638; $\boldsymbol{R_f}$ 0.56 (hex/EtOAc, 1/1).



6-(Thiophen-2-yl)benzo[*b*]naphtho[2,1-*d*]thiophene, **3ga**: White solid; m. p. 95.3-97.1 °C ; **^{1}H NMR (600 MHz, CDCl$_3$)** δ 8.19 (d, *J* = 8.4 Hz, 1H), 7.94 (ddd, *J* = 8.4, 7.8, 1.2 Hz, 2H), 7.84 (s, 1H), 7.65 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.59 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.52 (dd, *J* = 6.0, 1.2 Hz, 1H), 7.40 (ddd, *J* = 7.8, 6.0, 1.2 Hz, 1H), 7.25 – 7.23 (m, 2H), 7.23 – 7.20 (m, 2H); **^{13}C NMR (151 MHz, CDCl$_3$)** δ 141.95, 139.34, 138.40, 136.63, 131.66, 131.14, 129.36, 129.07, 128.92, 128.86, 127.60, 127.51, 127.36, 127.06, 126.19, 125.99, 124.82, 124.53, 124.36, 122.90; **IR (neat)**: ν_{max} = 3060, 2918, 2849, 1496, 1420, 1230, 754, 696 cm^{-1}; **HRMS** m/z (EI) calc. for $C_{20}H_{12}S_2$ [M+] 316.0380, found 316.0377; ***R$_f$*** 0.48 (in hexane).



6-(Thiophen-3-yl)furo[2,3-*h*]quinoline, **6**: Pale yellow sticky solid; **^{1}H NMR (600 MHz, CDCl$_3$)** δ 8.94 (dd, *J* = 4.2, 1.8, Hz, 1H), 8.29 (dd, *J* = 7.8, 1.8 Hz, 1H), 8.17 (s, 1H), 7.91 (s, 1H), 7.91 (d, *J* = 2.4 Hz, 1H), 7.79 (ddd, *J* = 5.4, 4.2, 1.2 Hz, 1H), 7.63 (d, *J* = 2.4 Hz, 1H), 7.50 (dd, *J* = 5.4, 2.4, 1H), 7.45 (dd, *J* = 7.8, 4.2 Hz, 1H); **^{13}C NMR (151 MHz, CDCl$_3$)** δ 152.40, 149.46, 144.74, 136.48, 135.96, 126.92, 125.85, 125.65, 124.85, 124.38, 122.14, 121.49, 121.48, 120.36, 106.39; **IR (neat)**: ν_{max} = 2923, 2853, 1510, 1375, 1057, 794 cm^{-1}; **HRMS** m/z (EI) calc. for $C_{15}H_9NOS$ [M+] 251.0405, found 251.0407; ***R$_f$*** 0.53 (hex/EtOAc, 1/1).

References

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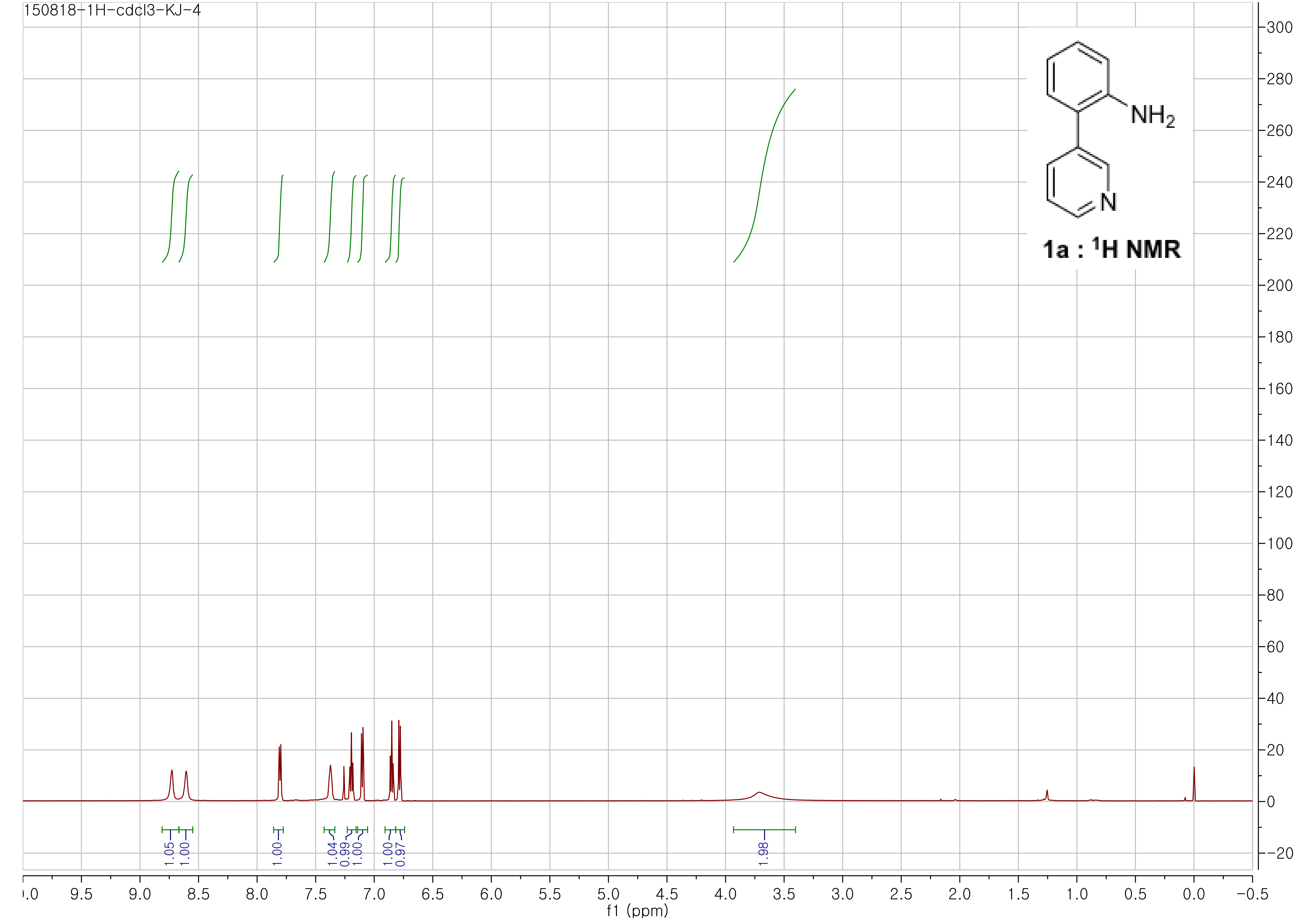
150818-1H-cdcl3-KJ-4
NH2
N
1a : 1H NMR
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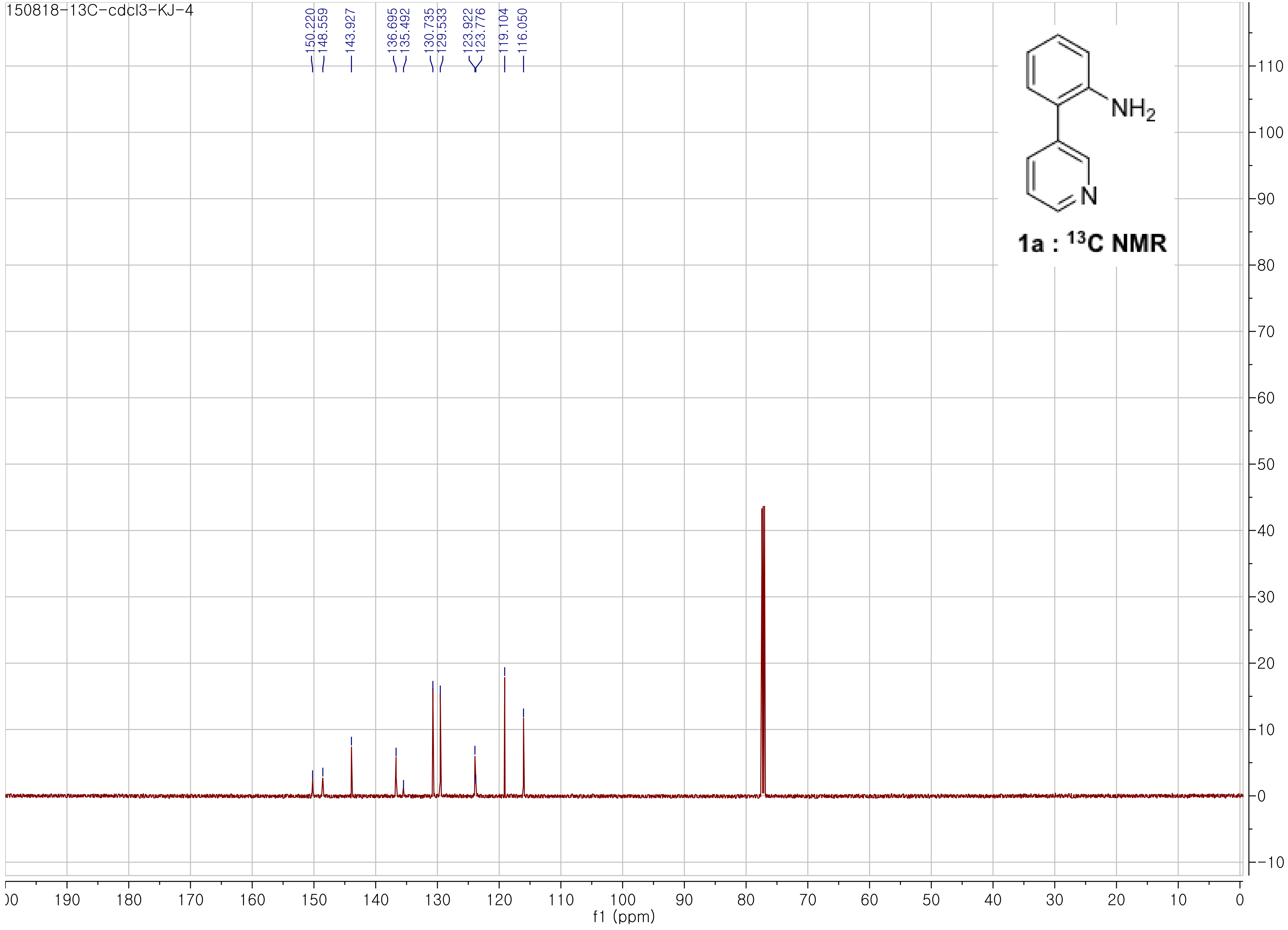
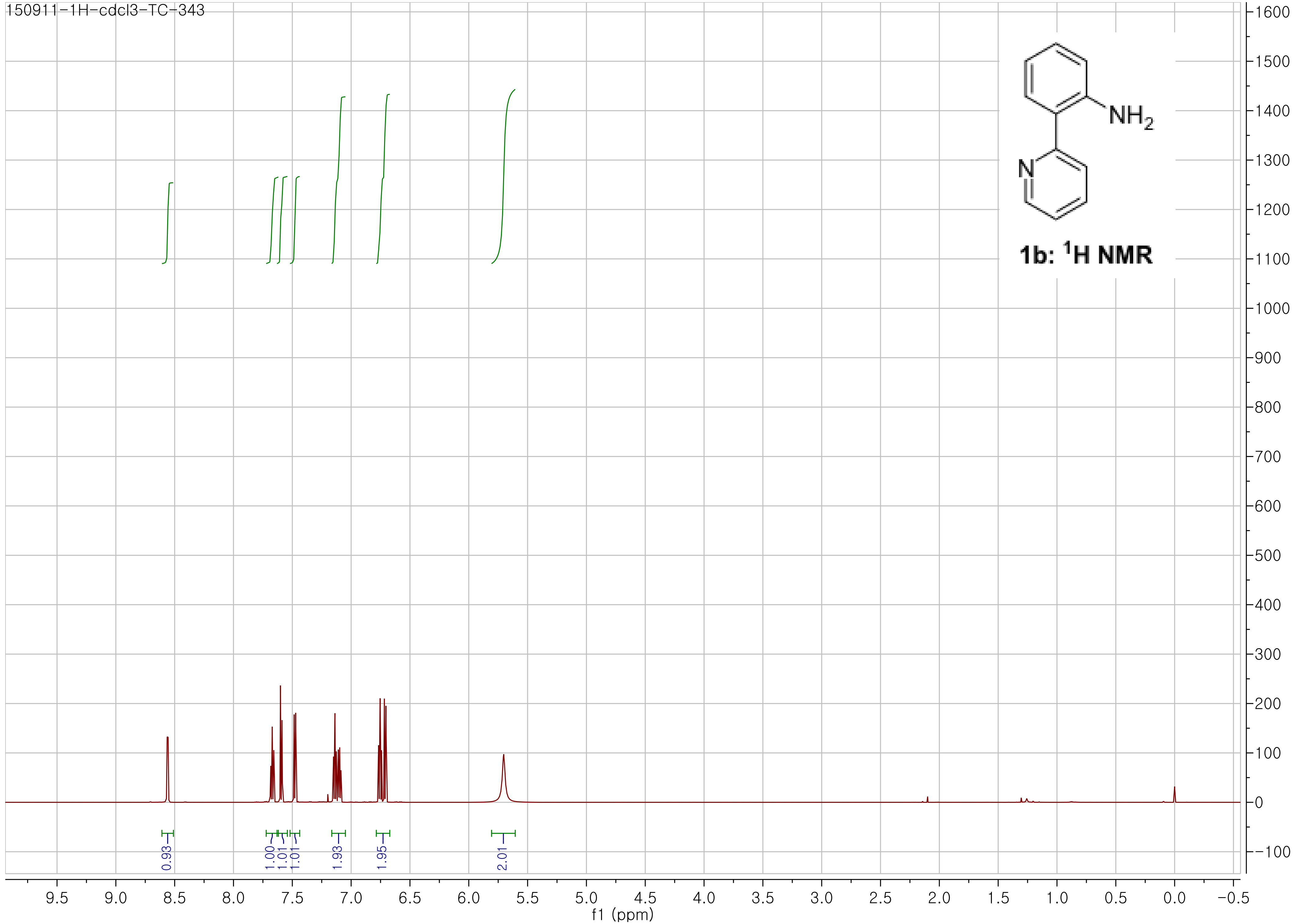
1.00
1.00
1.04
0.99
1.00
1.00
0.97
1.98
f1 (ppm)


150818-13C-cdcl3-KJ-4
150.220
148.559
143.927
136.695
135.492
130.735
129.533
123.922
123.776
119.104
116.050
NH2
N
1a : 13C NMR
f1 (ppm)


150911-1H-cdcl3-TC-343
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N
1b: 1H NMR
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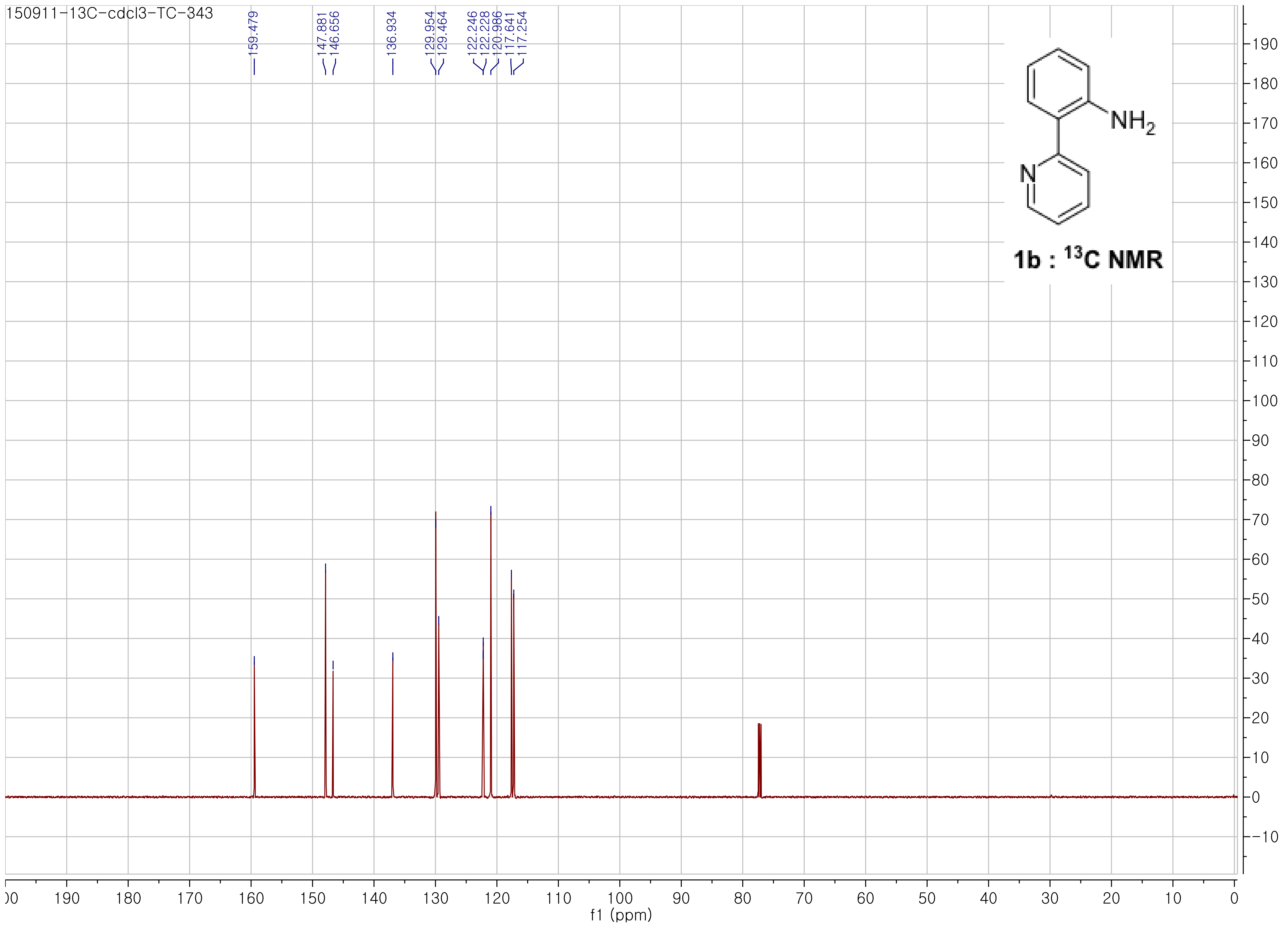
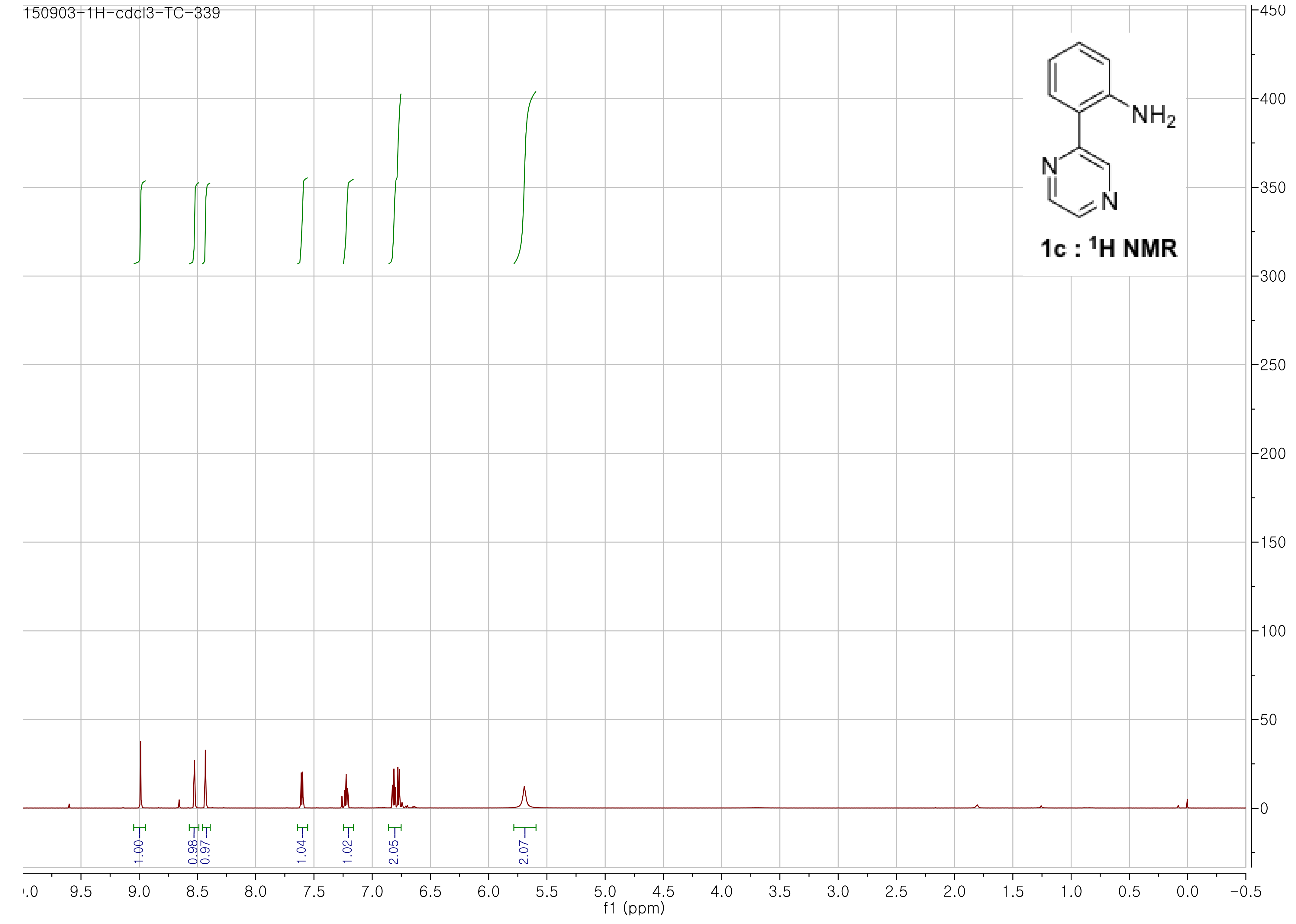
1.00
1.01
1.01
1.93
1.95
2.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
1600 1500 1400 1300 1200 1100 1000 900 800 700 600 500 400 300 200 100 0 -100


150911-13C-cdcl3-TC-343
159.479
147.881
146.656
136.934
129.954
129.464
122.246
122.228
120.986
117.641
117.254
NH2
N
1b : 13C NMR
f1 (ppm)


150903-1H-cdcl3-TC-339
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N
N
1c : 1H NMR
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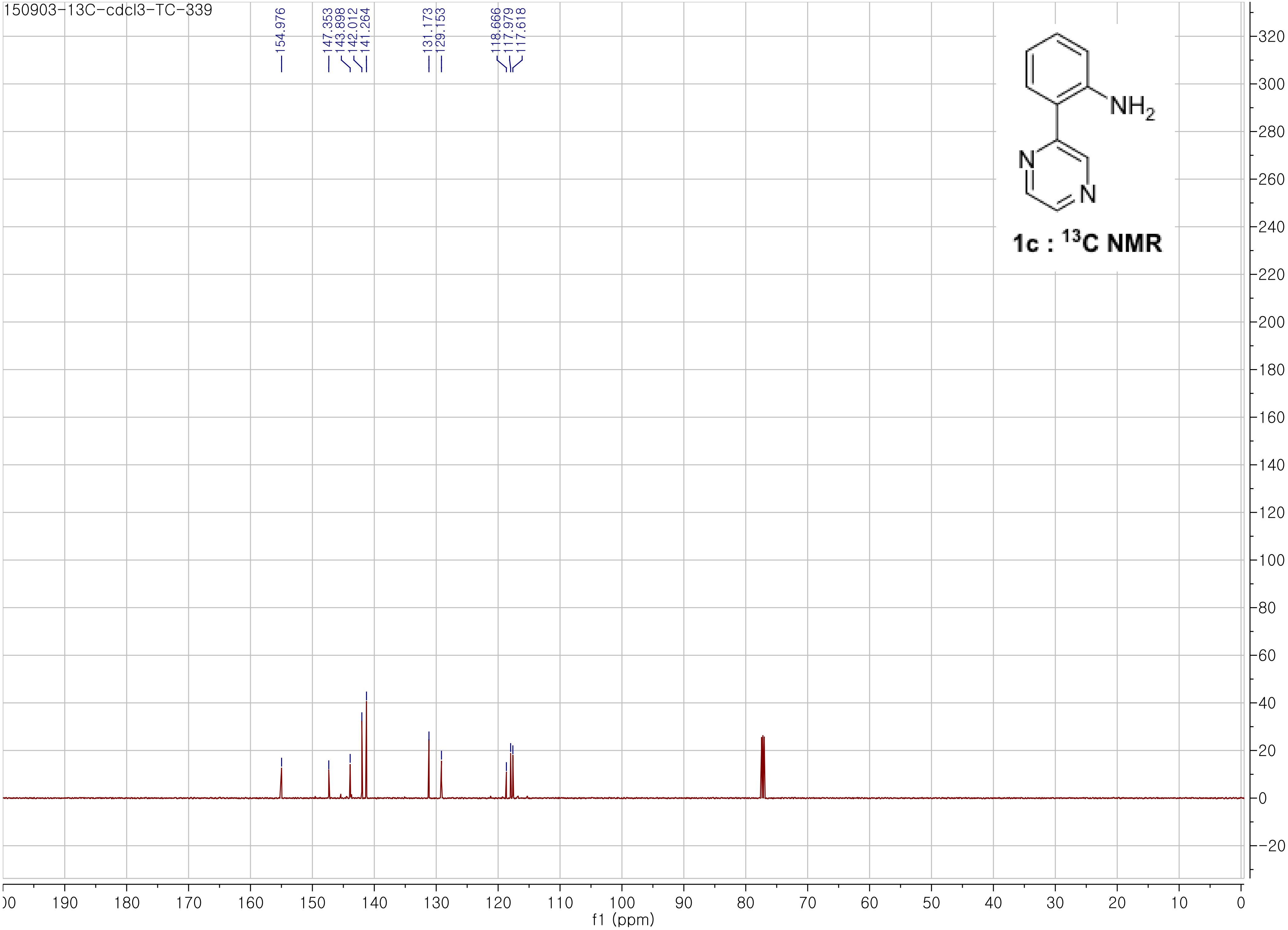
0.98
0.97
1.04
1.02
2.05
2.07
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
450
400
350
300
250
200
150
100
50
0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147.353
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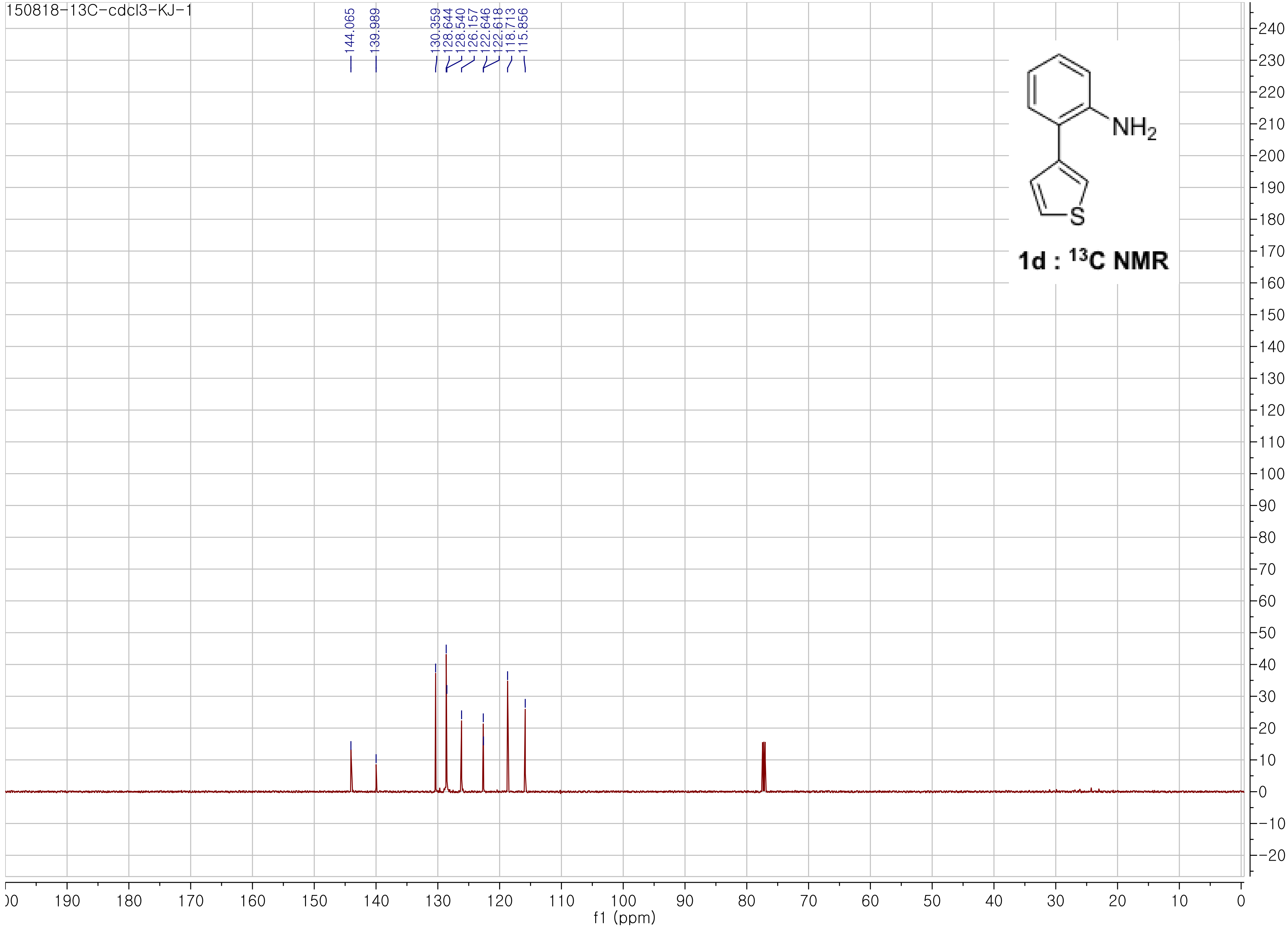
142.012
141.264
131.173
129.153
118.666
117.979
117.618
NH2
N
N
1c : 13C NMR
f1 (ppm)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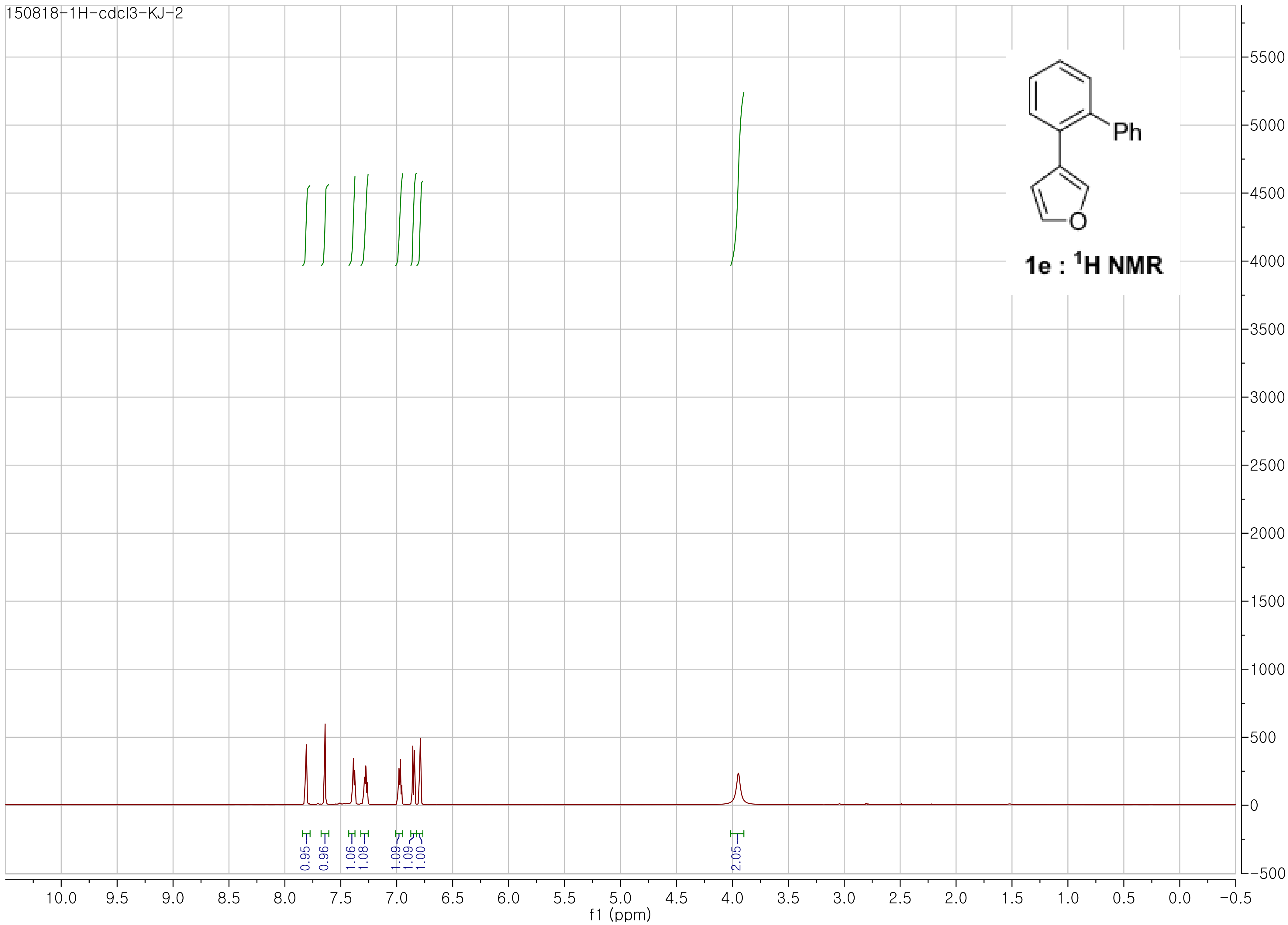
139.989
130.359
128.644
128.540
126.157
122.646
122.618
118.713
115.856
NH2
S
1d : 13C NMR
f1 (ppm)



150818-1H-cdcl3-KJ-2
Ph
O
1e : ¹H NMR
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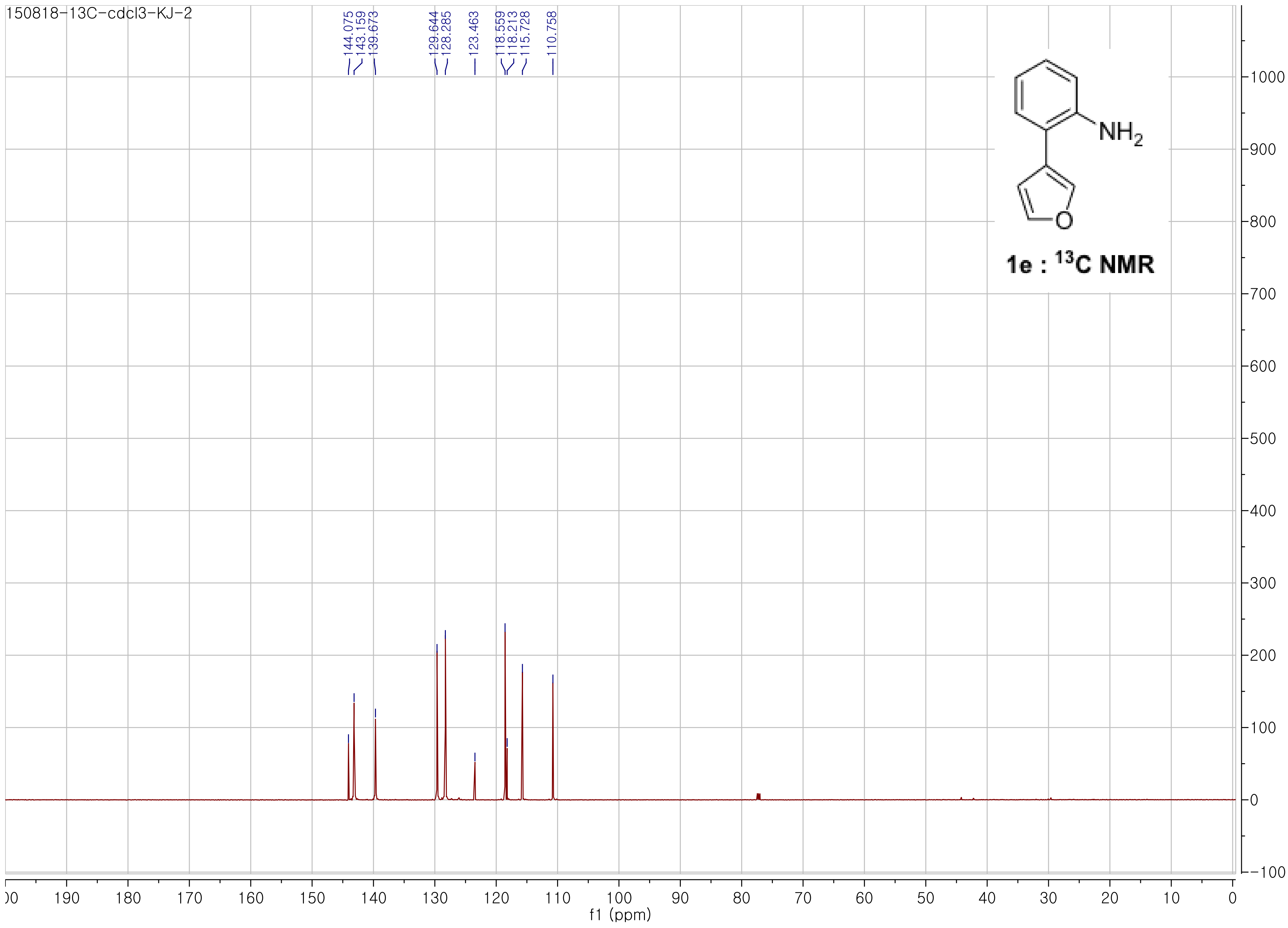
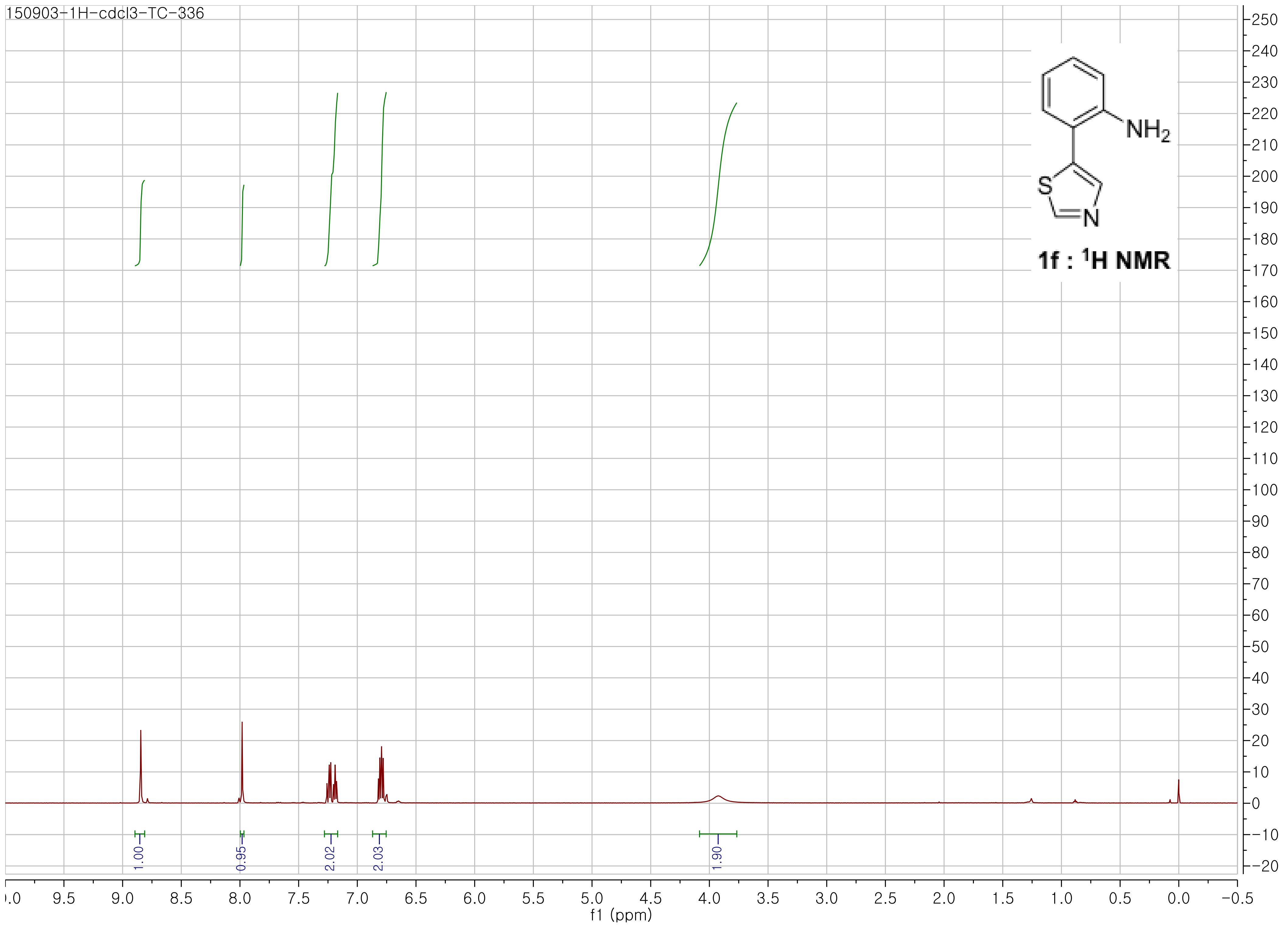
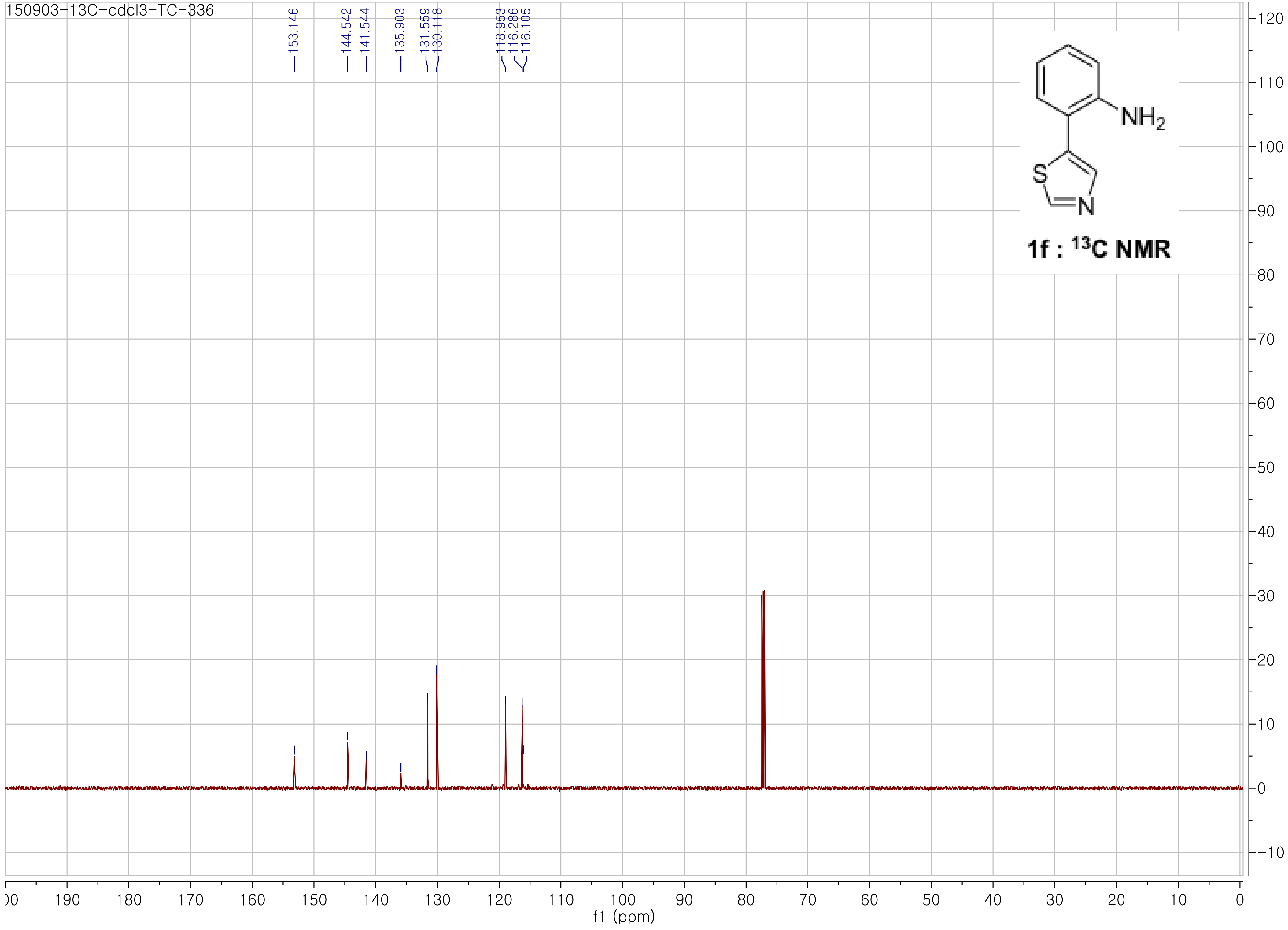
0.96
1.06
1.08
1.09
1.09
1.00
2.05
f1 (ppm)


150818-13C-cdcl3-KJ-2
144.075
143.159
139.673
129.644
128.285
123.463
118.559
118.213
115.728
110.758
NH2
O
1e : 13C NMR
f1 (ppm)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
1000 900 800 700 600 500 400 300 200 100 0 -100



150903-1H-cdcl3-TC-336
NH2
S
N
1f : 1H NMR
1.00
0.95
2.02
2.03
1.90
f1 (ppm)


150903-13C-cdcl3-TC-336
153.146
144.542
141.544
135.903
131.559
130.118
118.953
116.286
116.105
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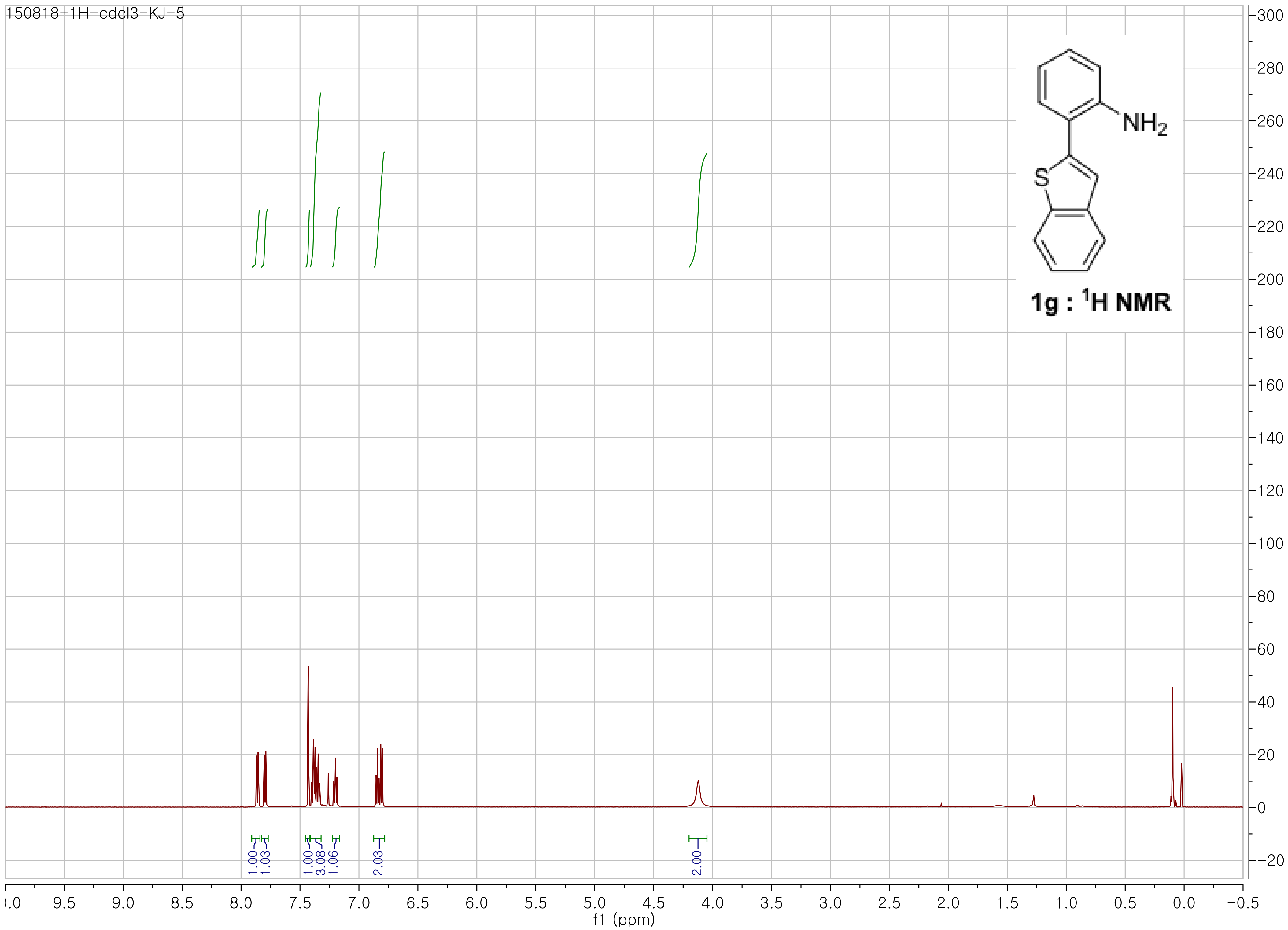
S
N
1f : 13C NMR
f1 (ppm)


150818-1H-cdcl3-KJ-5
NH2
S
1g : 1H NMR
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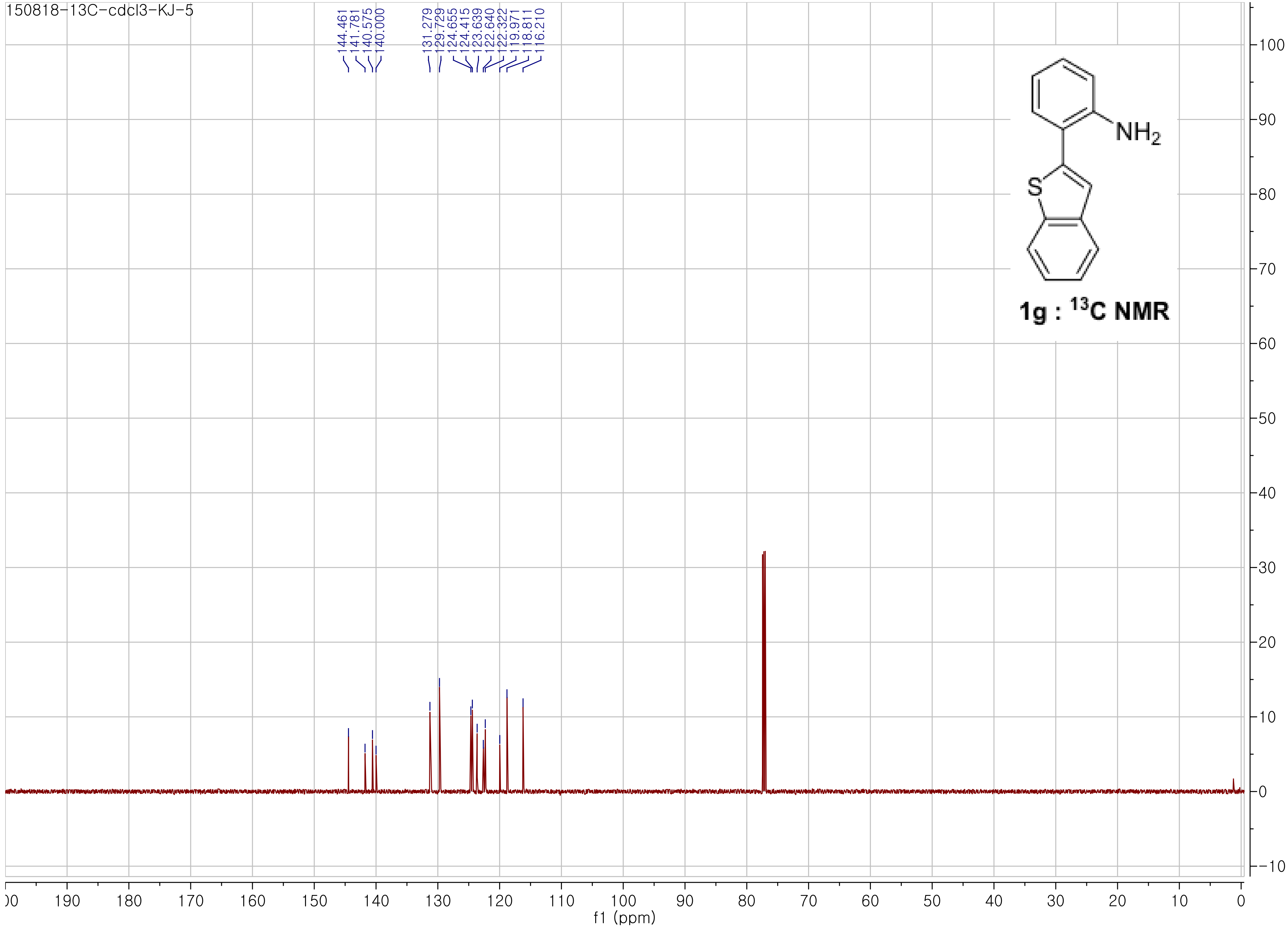
1.03
1.00
3.08
1.06
2.03
2.00
f1 (ppm)



150818-13C-cdcl3-KJ-5
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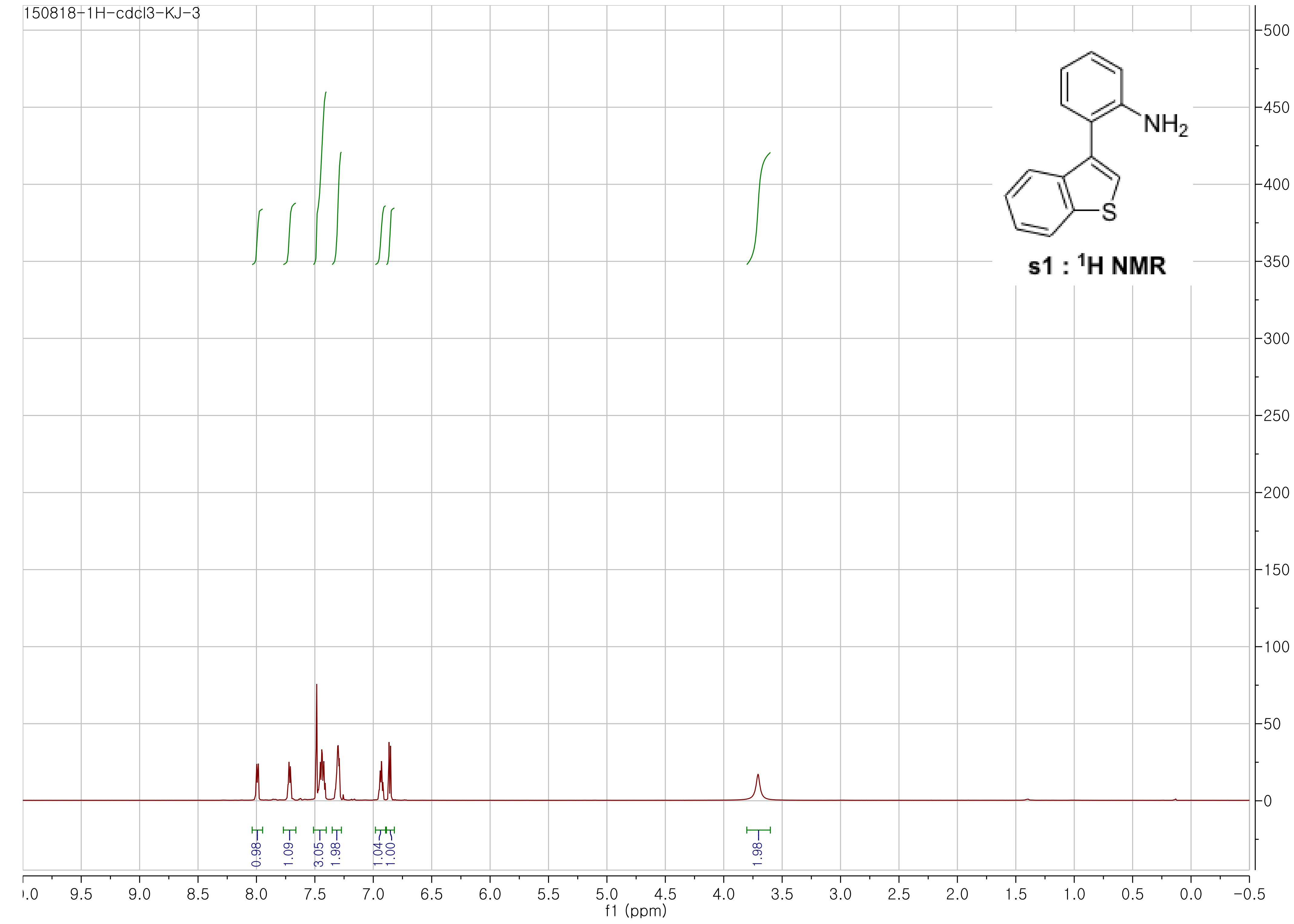
140.575
140.000
131.279
129.729
124.655
124.415
123.639
122.640
122.322
119.971
118.811
116.210
NH2
S
1g : 13C NMR
f1 (ppm)



150818-1H-cdcl3-KJ-3
NH2
S
s1 : 1H NMR
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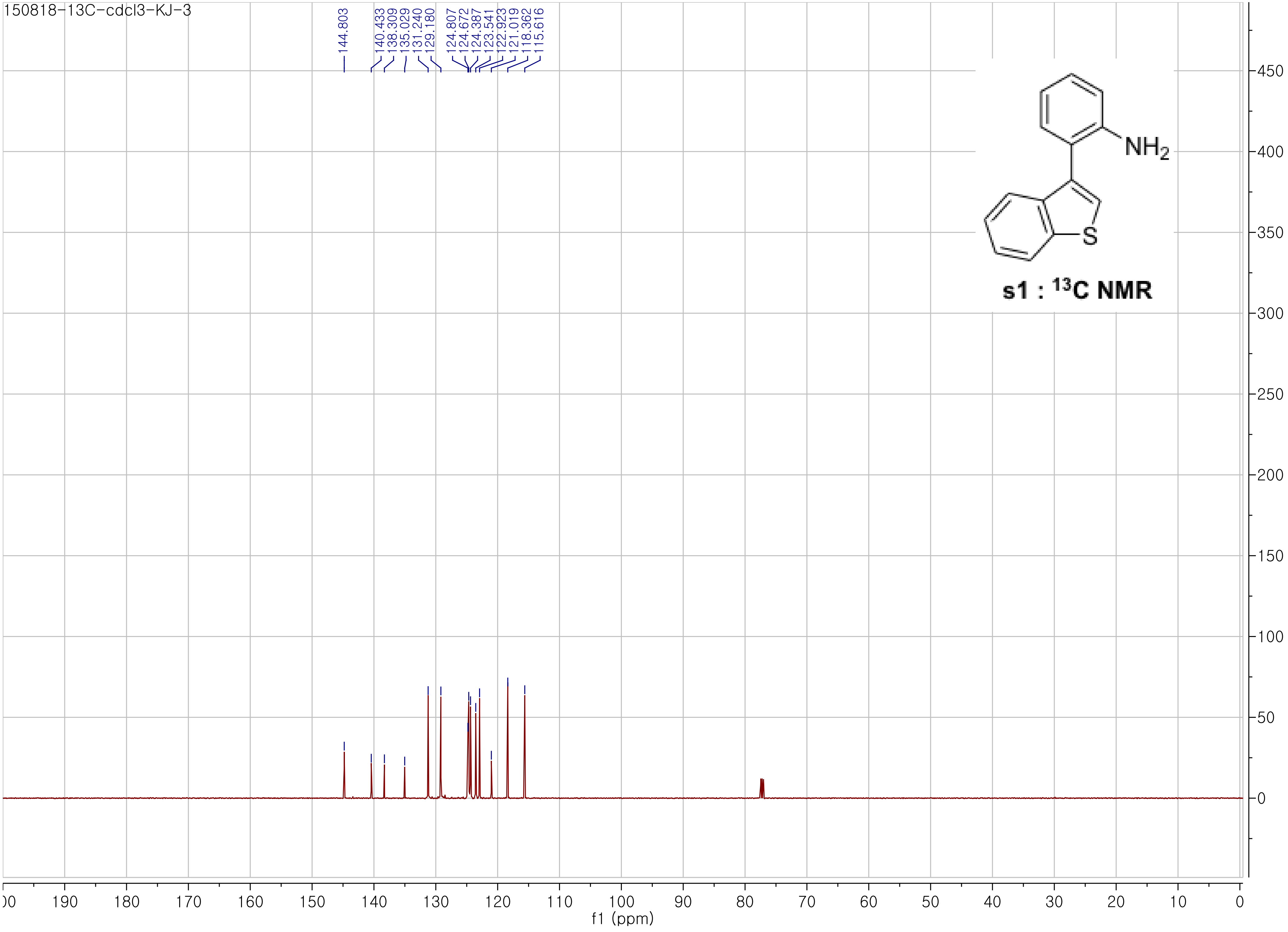
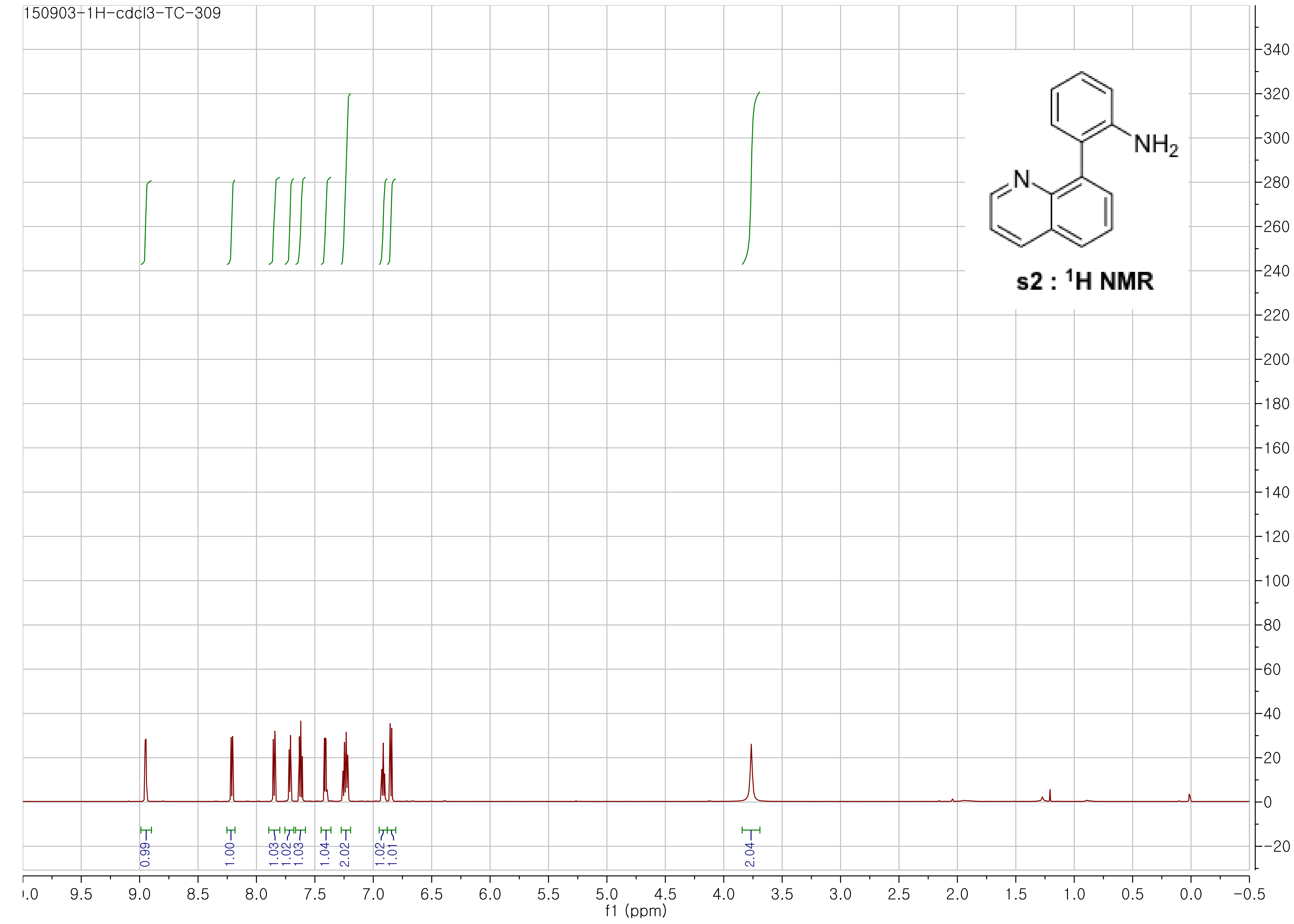
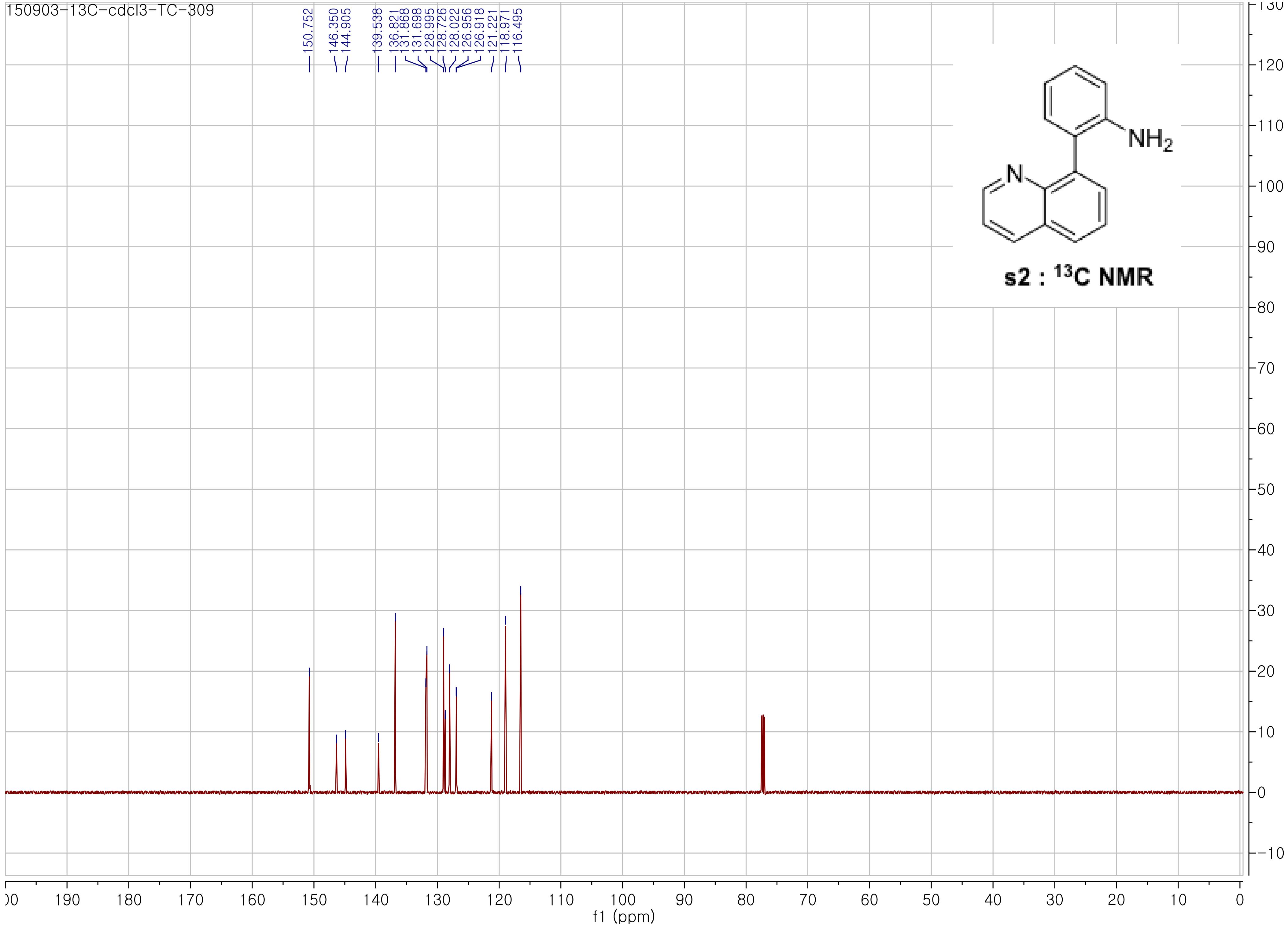
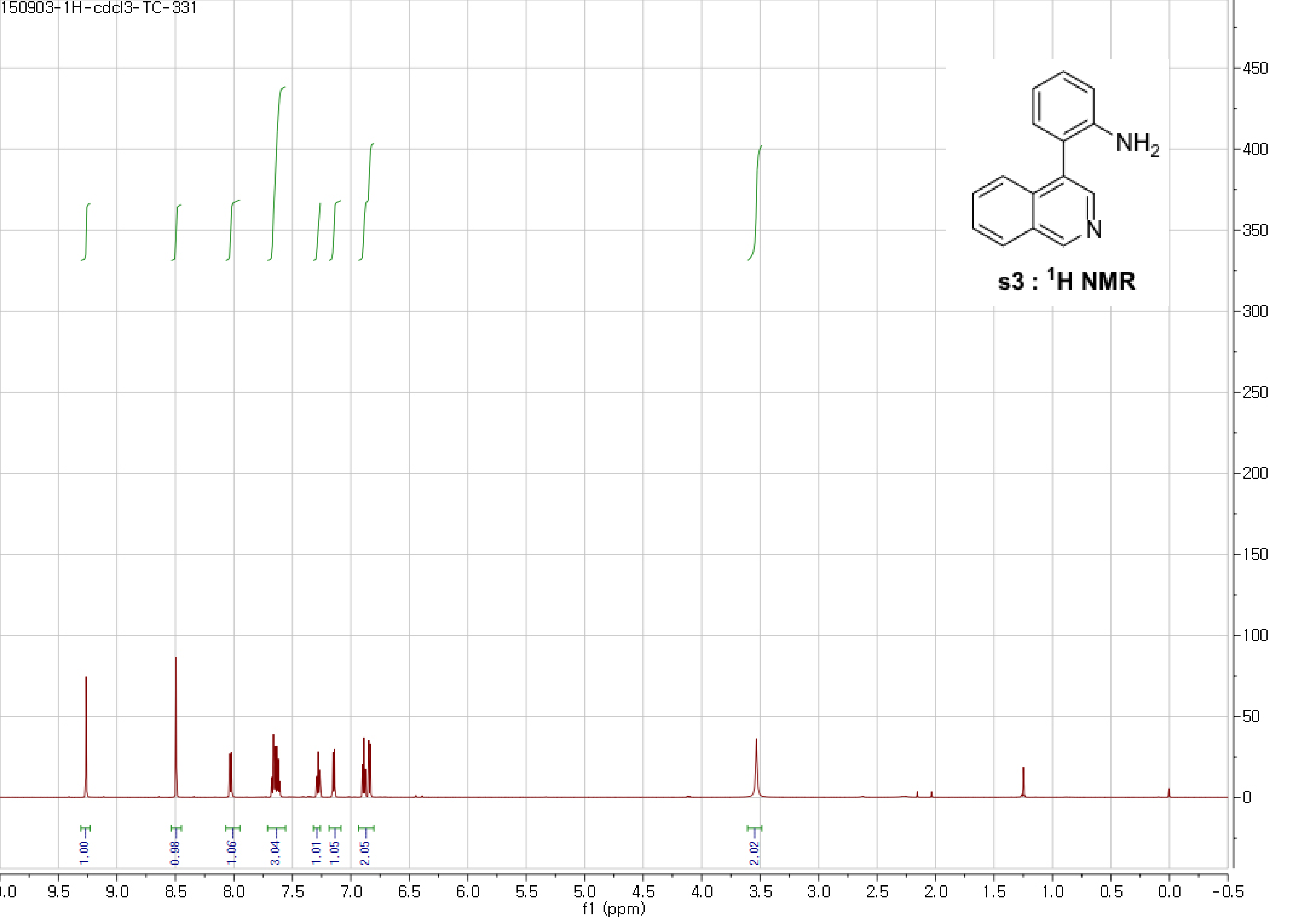
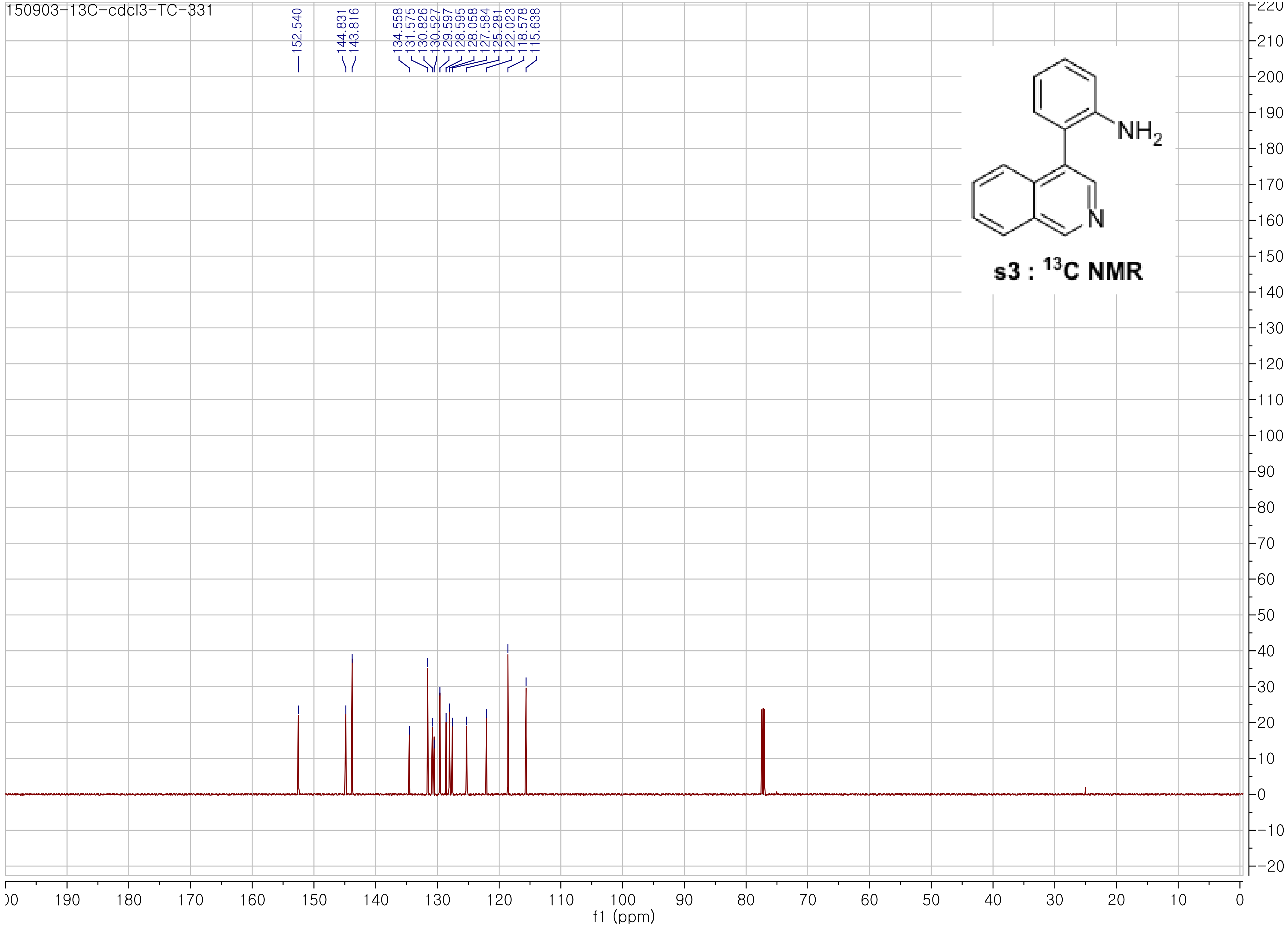
1.09
3.05
1.98
1.04
1.00
1.98
f1 (ppm)


150818-13C-cdcl3-KJ-3
144.803
140.433
138.309
135.029
131.240
129.180
124.807
124.672
124.387
123.541
122.923
121.019
118.362
115.616
NH2
S
s1 : 13C NMR
f1 (ppm)

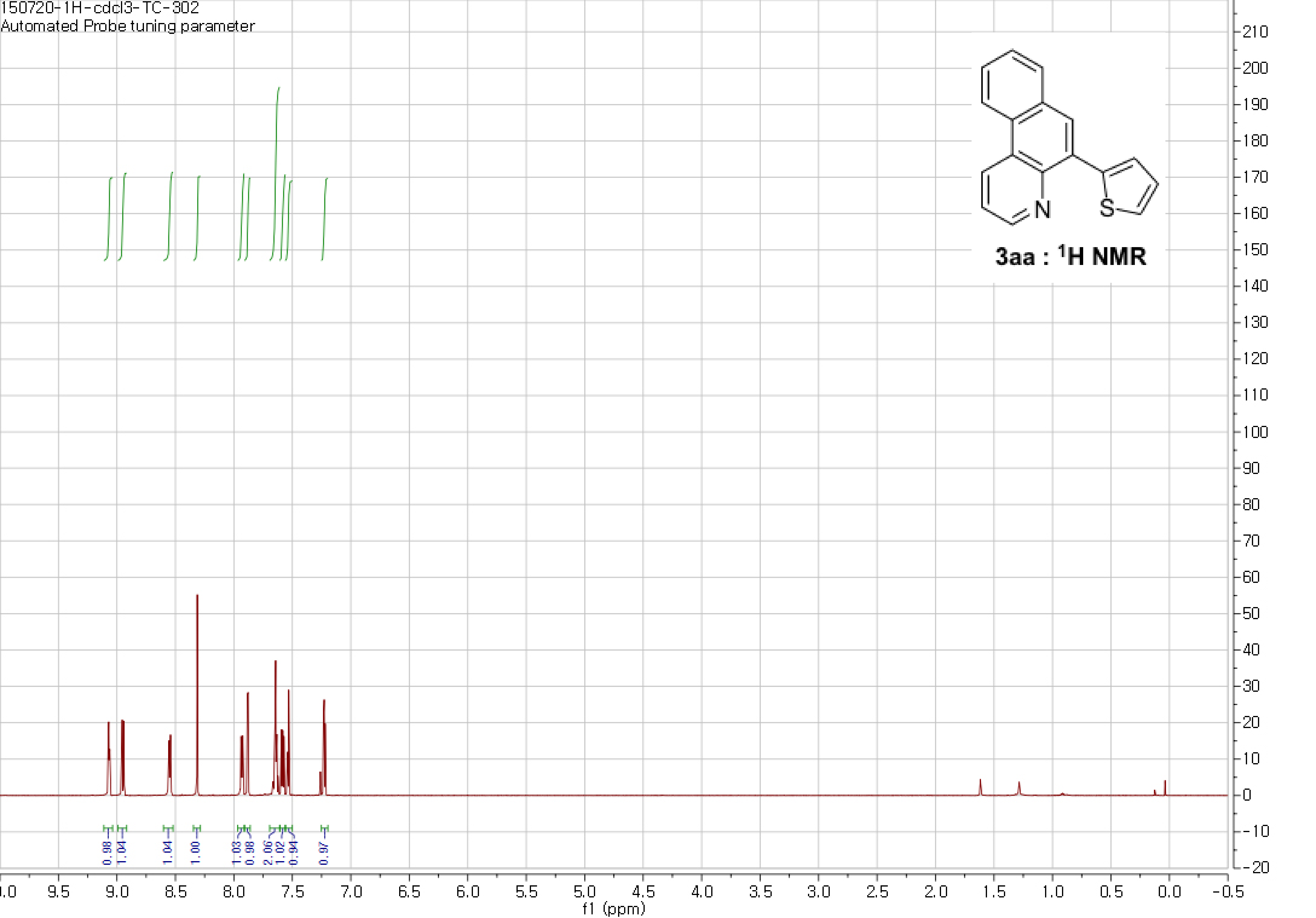

150903-1H-cdcl3-TC-309
NH2
N
s2 : 1H NMR
0.99
1.00
1.03
1.02
1.03
1.04
2.02
1.02
1.01
2.04
f1 (ppm)

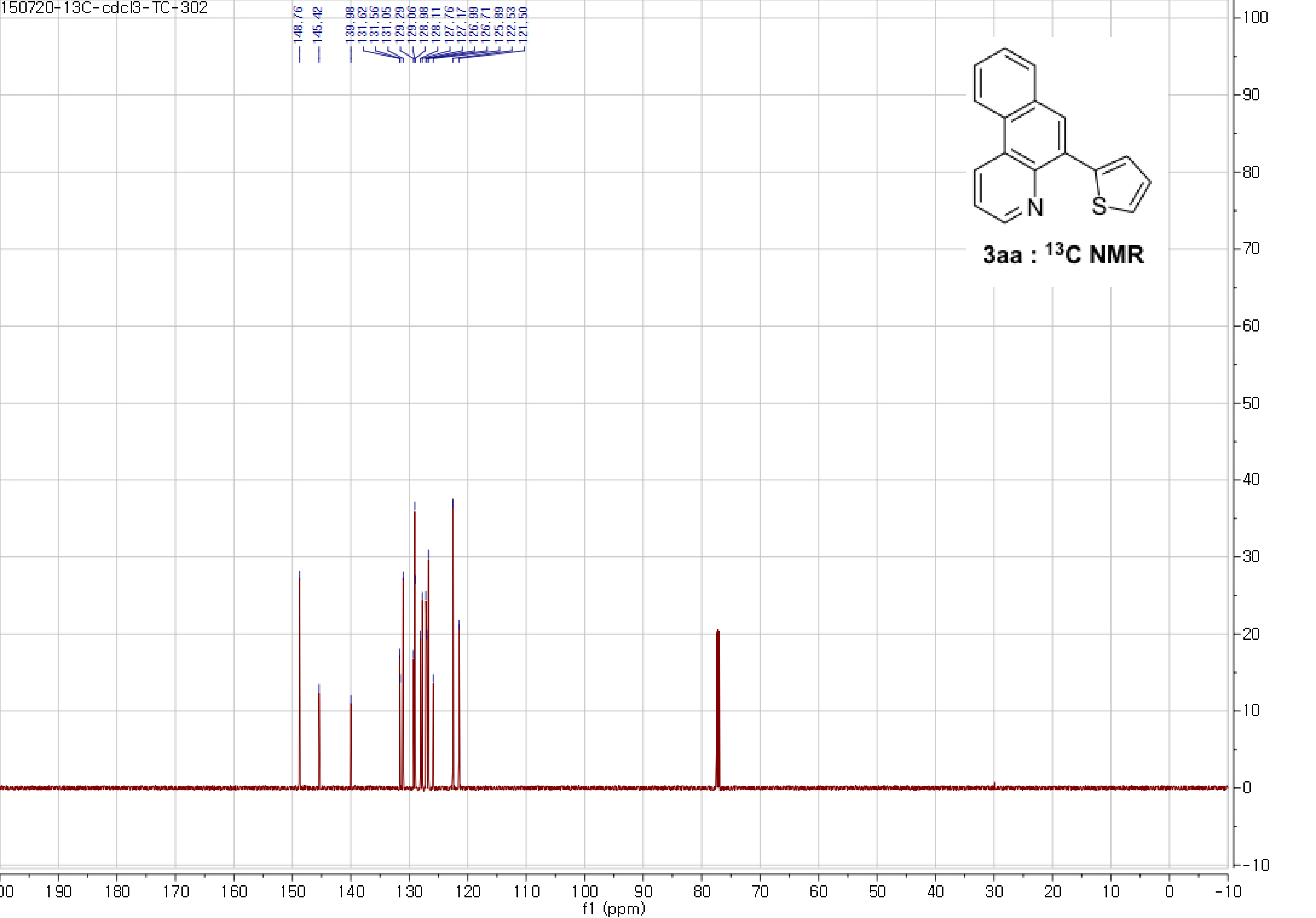

150903-13C-cdcl3-TC-309
150.752
146.350
144.905
139.538
136.821
131.868
131.698
128.995
128.726
128.022
126.956
126.918
121.221
118.971
116.495
NH2
N
s2 : 13C NMR
f1 (ppm)


150903-1H-cdcl3-TC-331
NH2
N
s3 : 1H NMR
1.00
0.98
1.06
3.04
1.01
1.05
2.05
2.02
f1 (ppm)

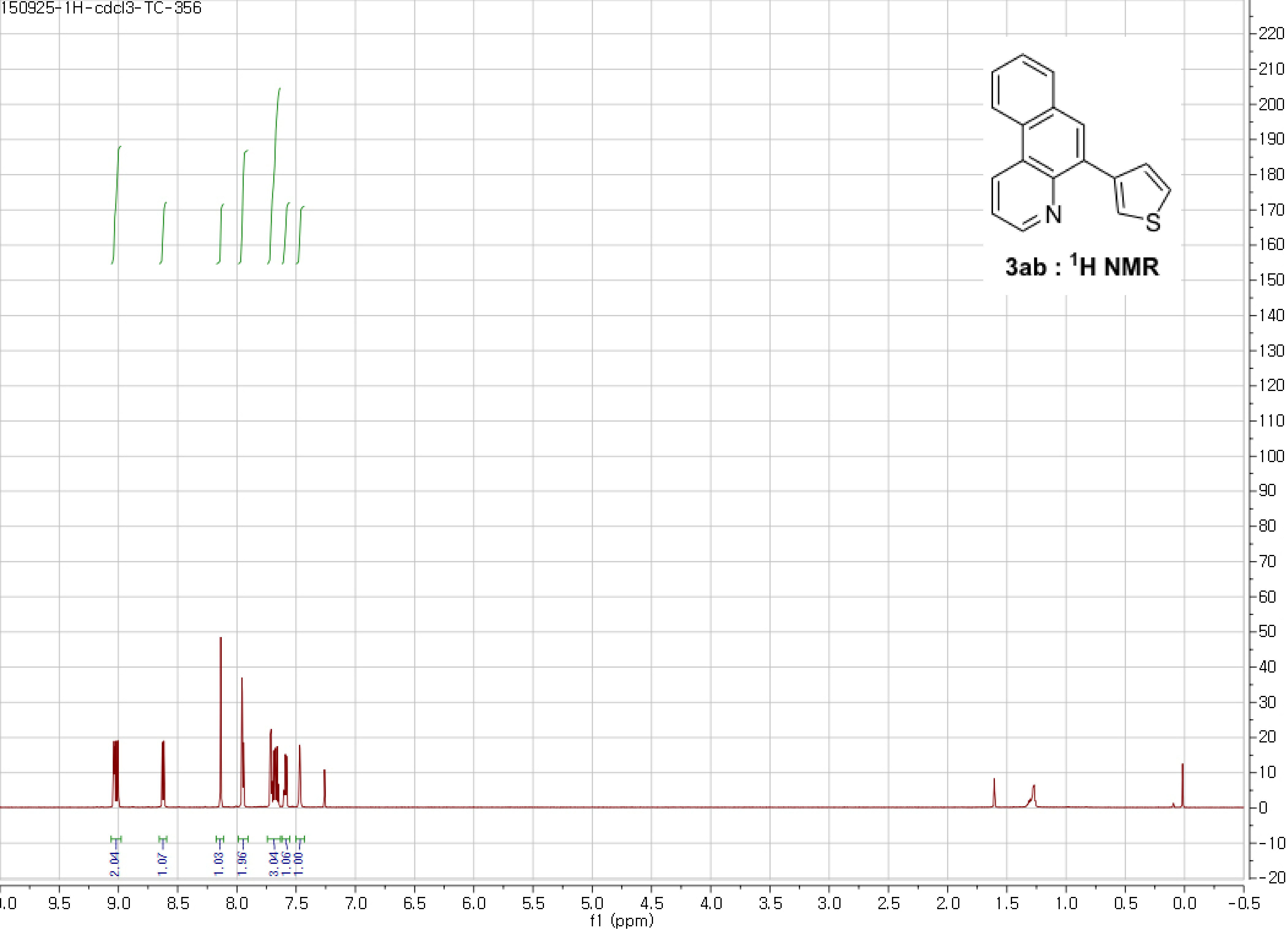


150903-13C-cdcl3-TC-331
152.540
144.831
143.816
134.558
131.575
130.826
130.527
129.597
128.595
128.058
127.584
125.281
122.023
118.578
115.638
NH2
N
s3 : 13C NMR
f1 (ppm)

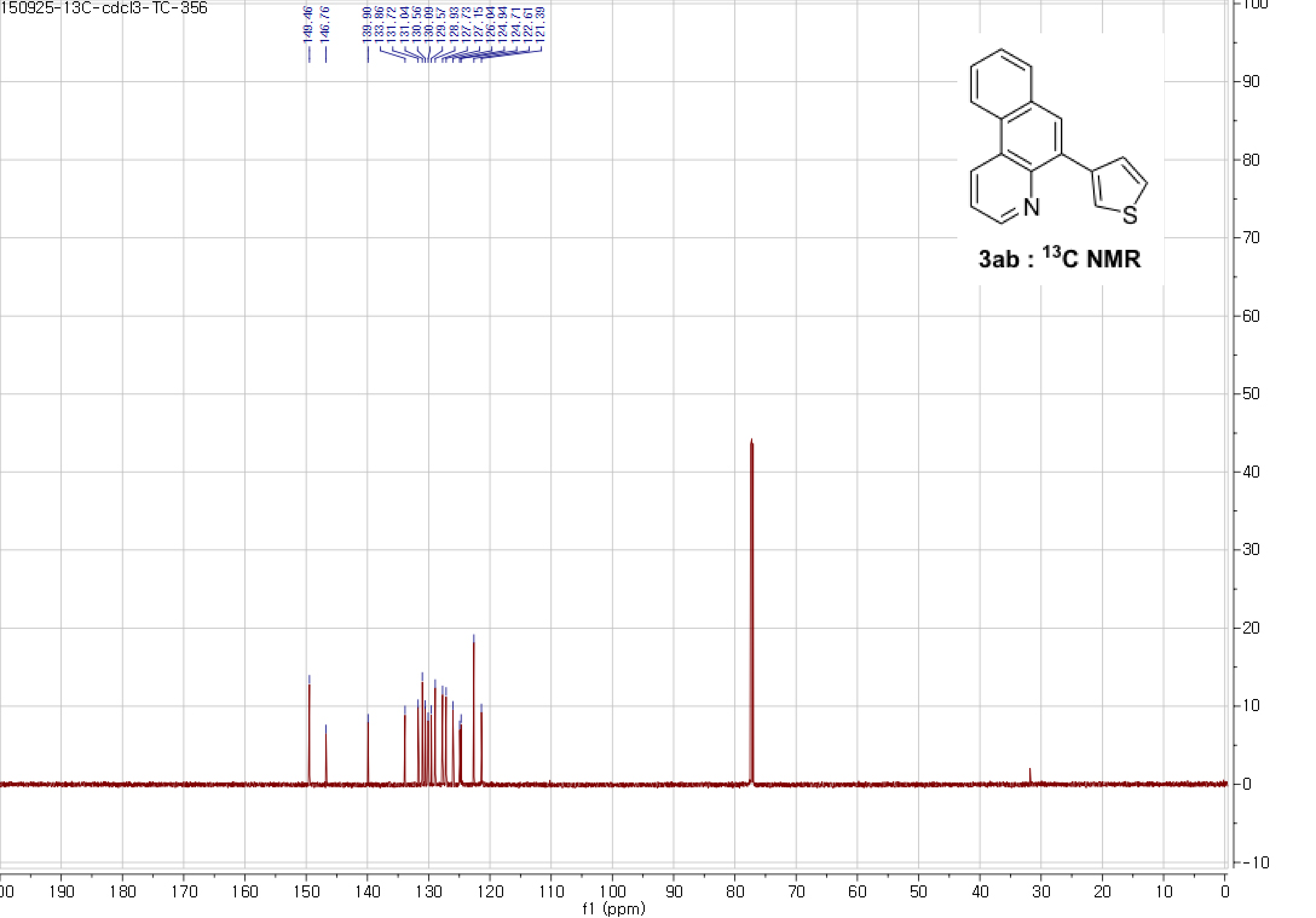

150720-1H-cdcl3-TC-302
Automated Probe tuning parameter
N
S
3aa : 1H NMR
0.98
1.04
1.04
1.00
1.03
0.98
2.06
1.02
0.94
0.97
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20

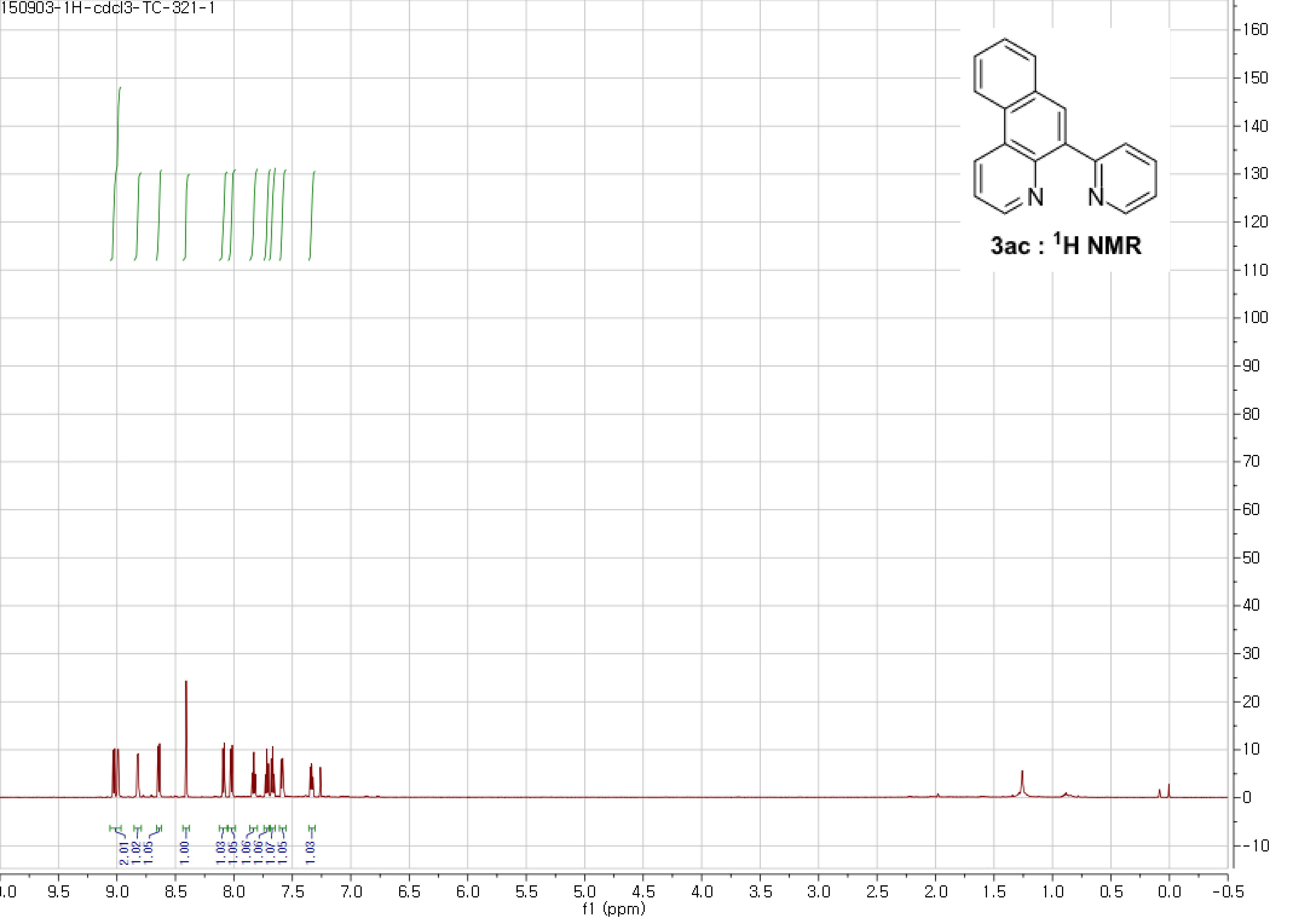


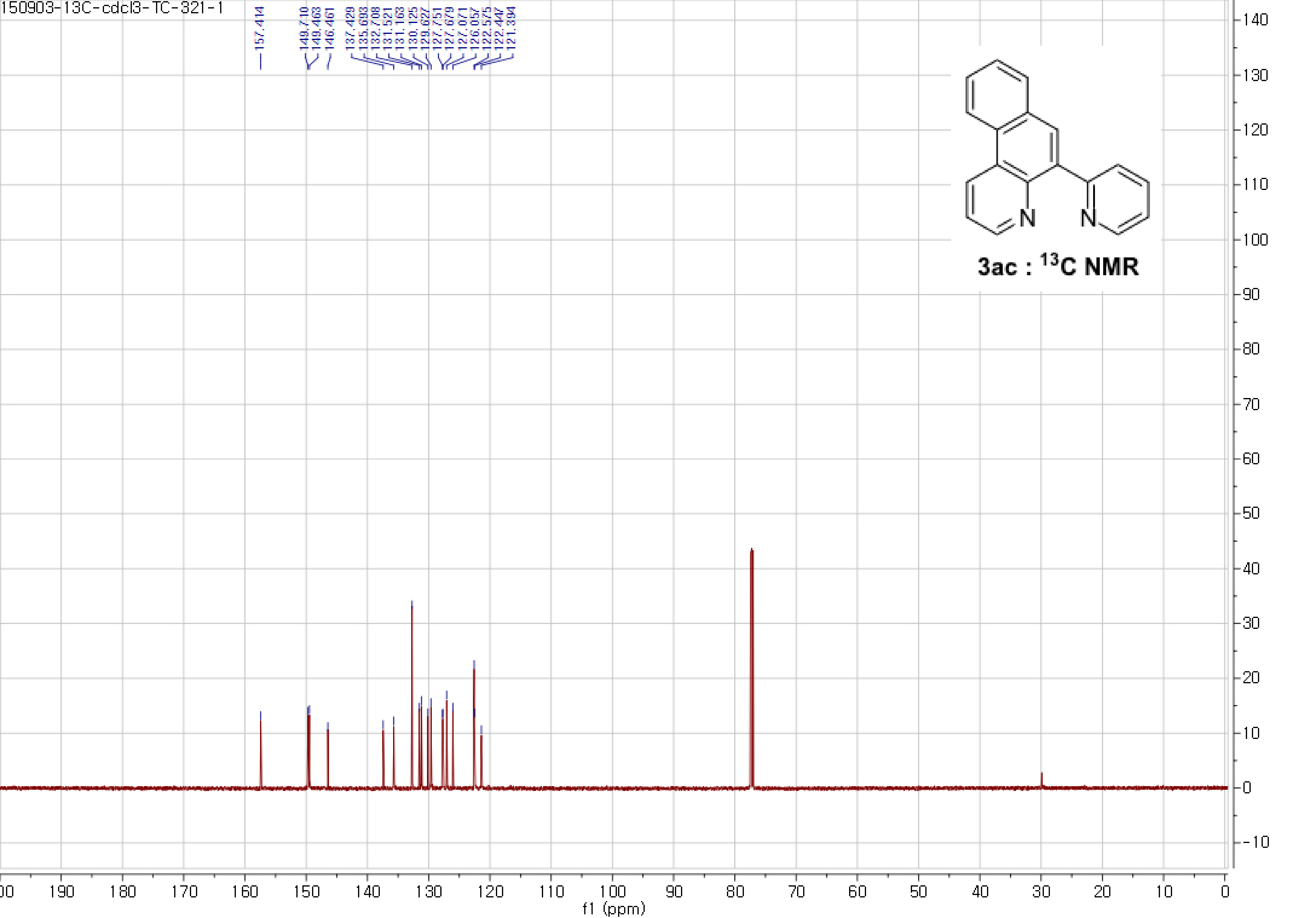
150720-13C-cdcl3-TC-302
148.76
145.42
139.98
131.62
131.56
131.05
129.29
129.06
128.98
128.11
127.76
127.17
126.99
126.71
125.89
122.53
121.50
N
S
3aa : 13C NMR
f1 (ppm)

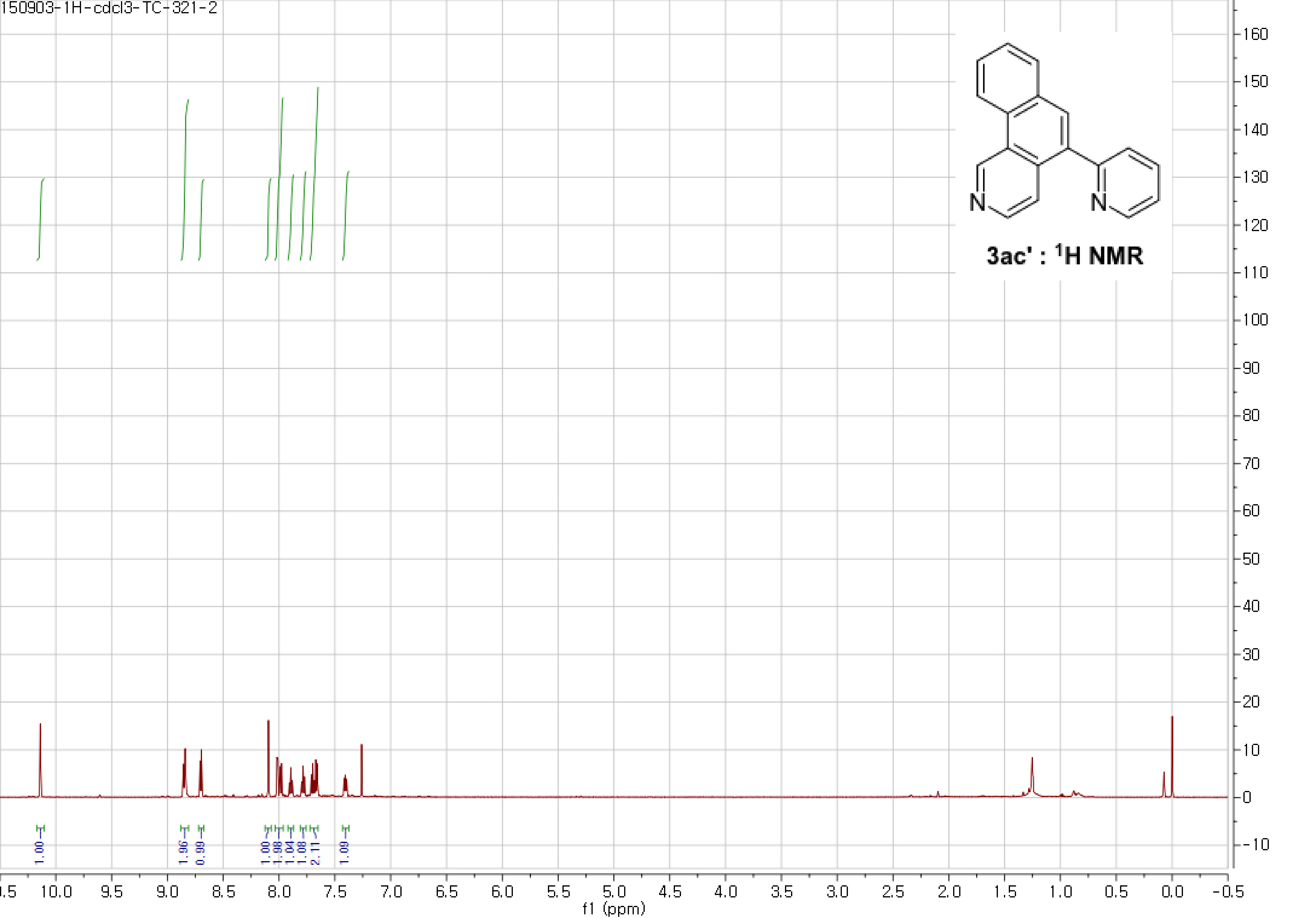


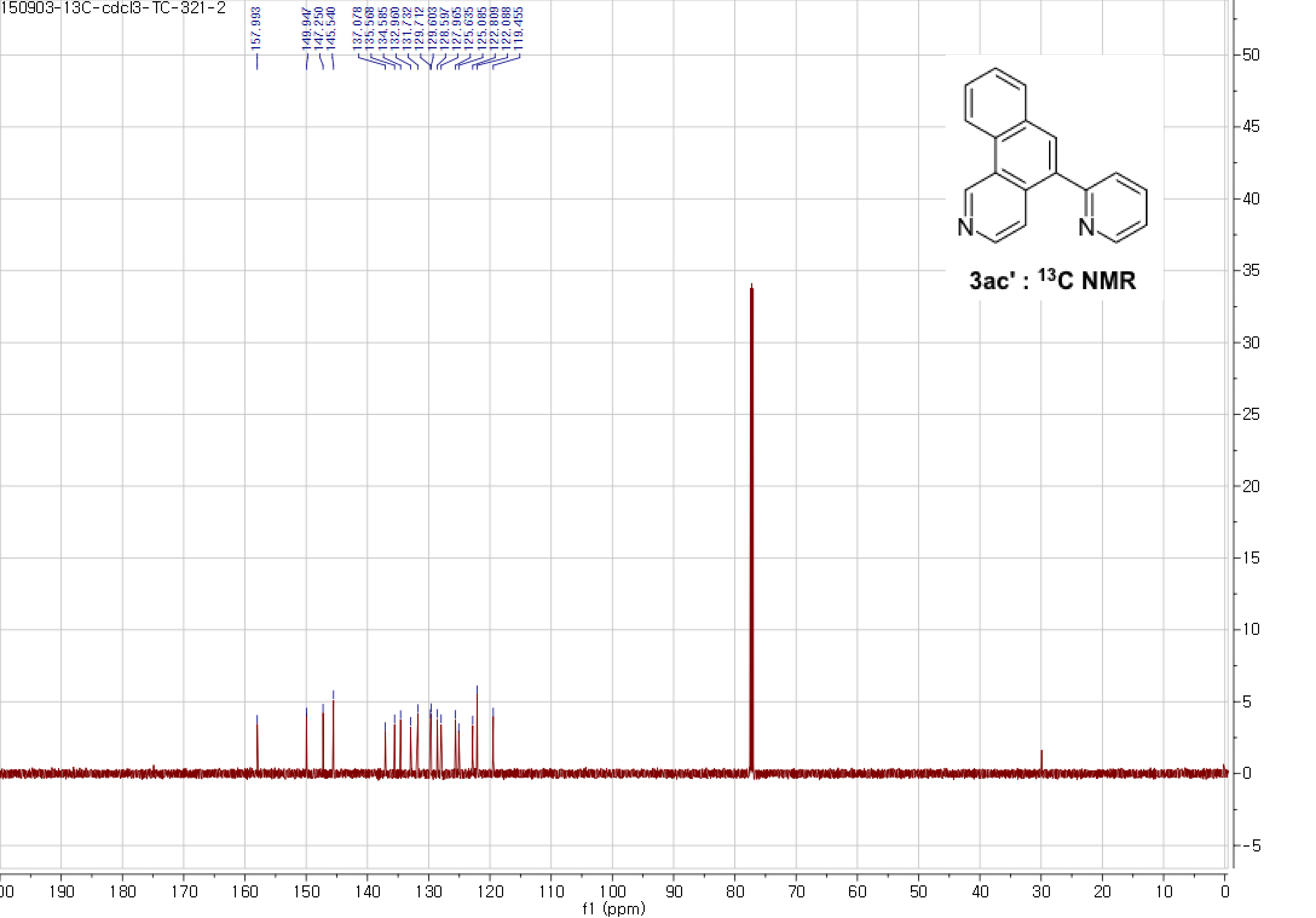
150925-1H-cdcl3-TC-356
N
S
3ab : 1H NMR
2.04
1.07
1.03
1.96
3.04
1.06
1.00
f1 (ppm)


150925-13C-cdcl3-TC-356
149.46
146.76
139.90
133.86
131.72
131.04
130.56
130.09
129.57
128.93
127.73
127.15
126.04
124.94
124.71
122.61
121.39
N
S
3ab : 13C NMR
f1 (ppm)

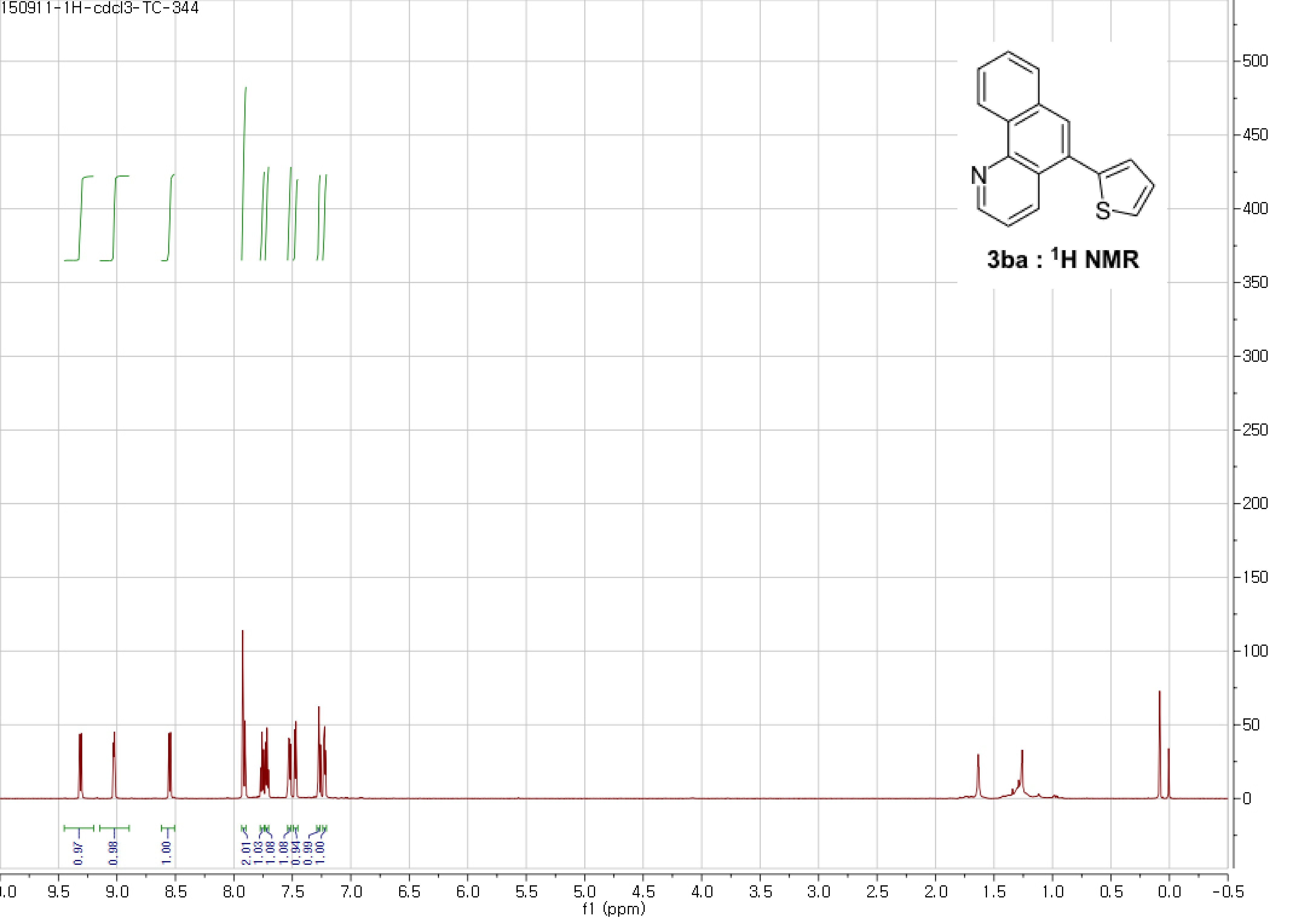

150903-1H-cdcl3-TC-321-1
3ac : 1H NMR
2.01
1.02
1.05
1.00
1.03
1.05
1.06
1.06
1.07
1.05
1.03
f1 (ppm)

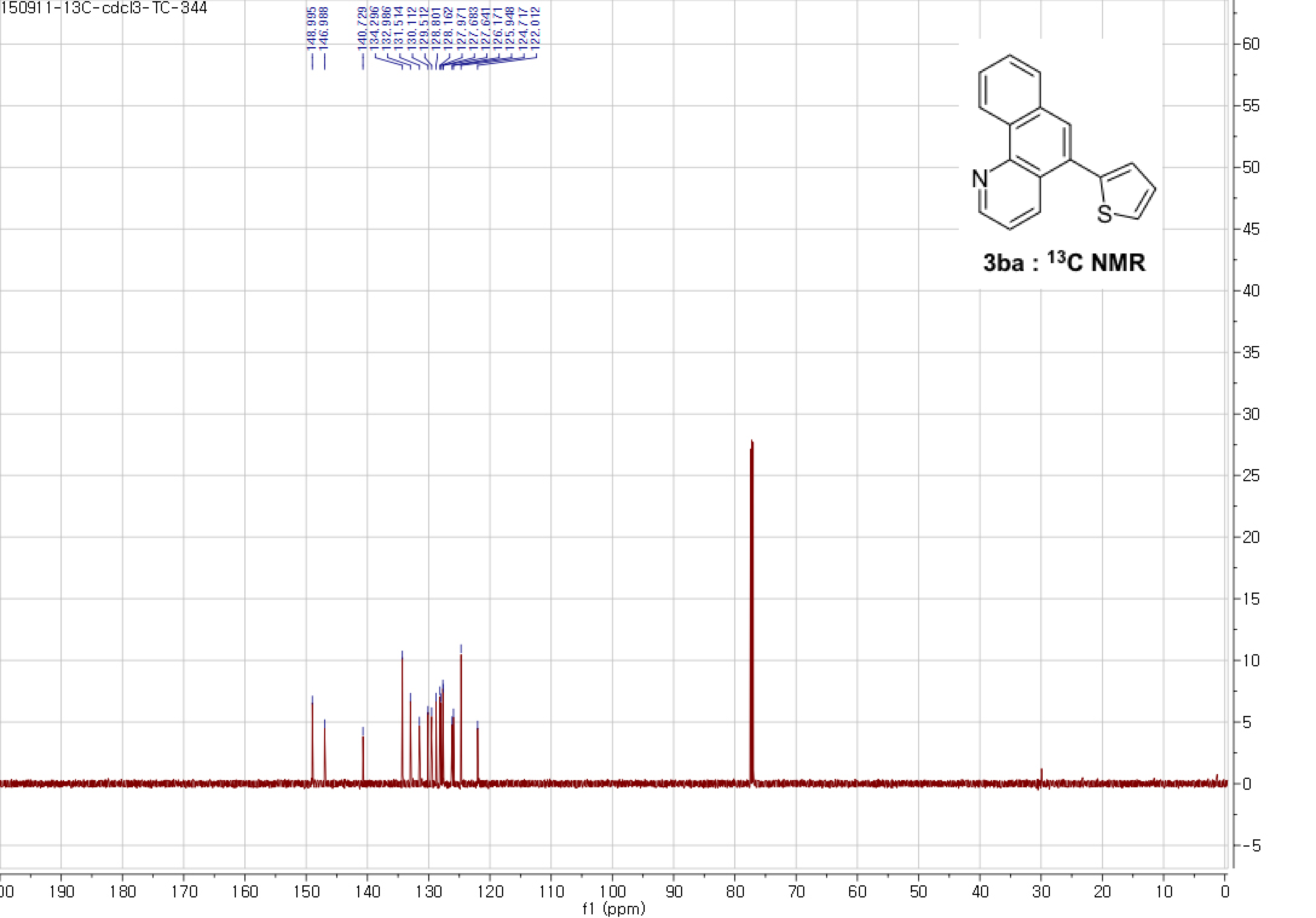

150903-13C-cdcl3-TC-321-1
157.414
149.710
149.463
146.461
137.429
135.693
132.708
131.521
131.163
130.125
129.627
127.751
127.679
127.071
126.057
122.575
122.447
121.394
N
N
3ac : 13C NMR
f1 (ppm)

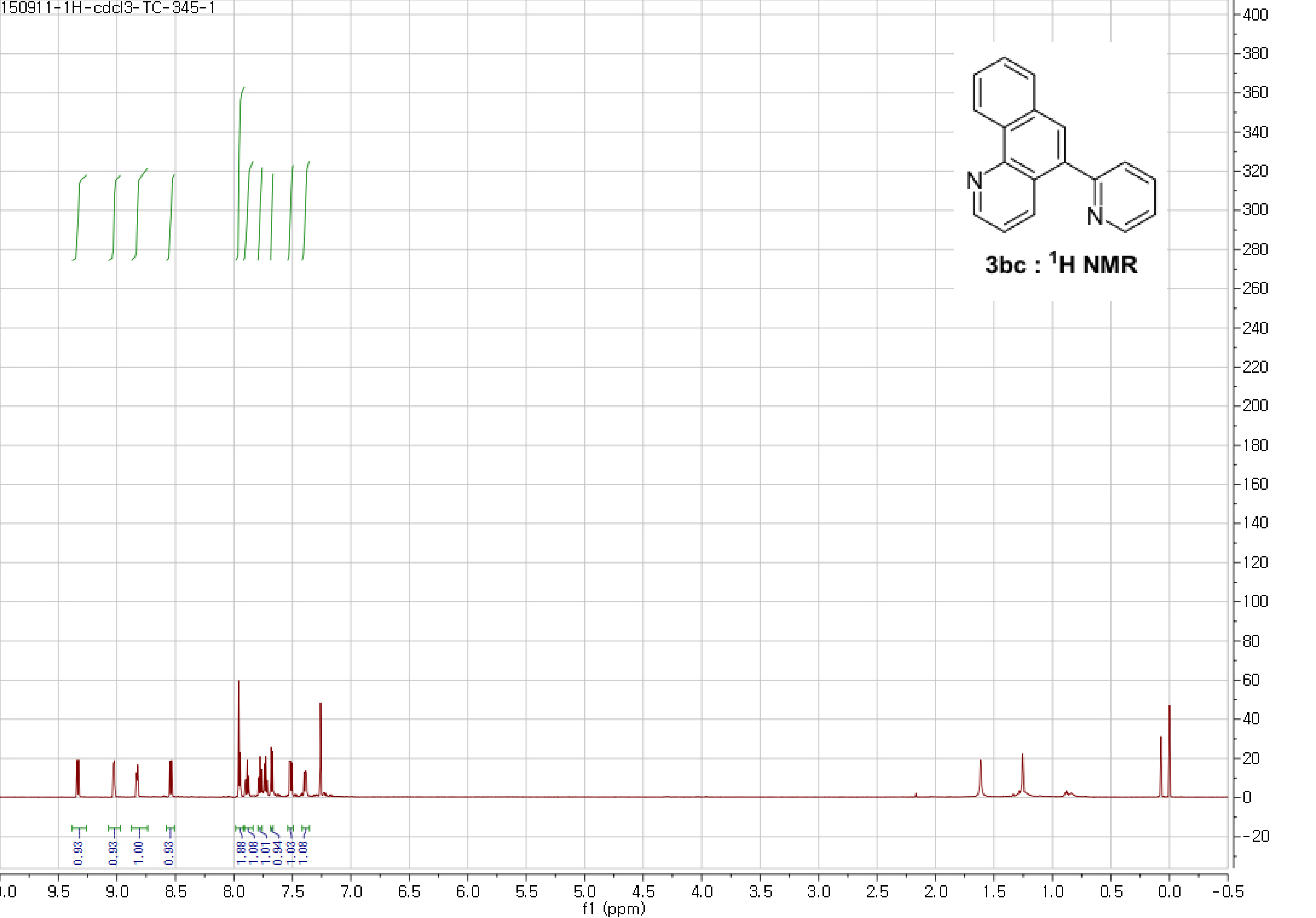

150903-1H-cdcl3-TC-321-2
3ac' : 1H NMR
N
N
1.00
1.96
0.99
1.00
1.98
1.04
1.08
2.11
1.09
f1 (ppm)

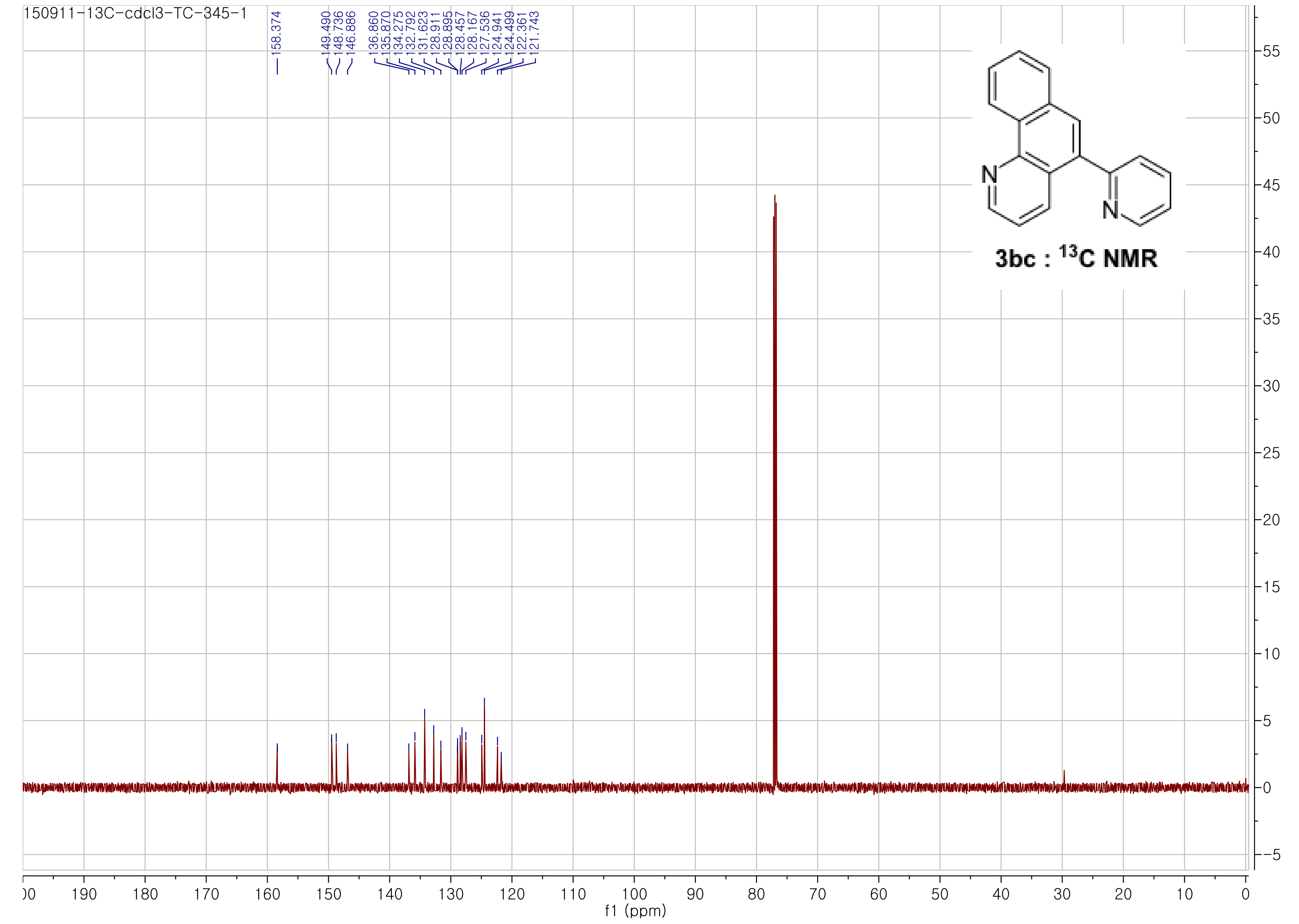


150903-13C-cdcl3-TC-321-2
157.993
149.947
147.250
145.540
137.078
135.568
134.585
132.960
131.732
129.712
129.603
128.597
127.965
125.635
125.085
122.809
122.088
119.455
N
N
3ac' : 13C NMR
f1 (ppm)

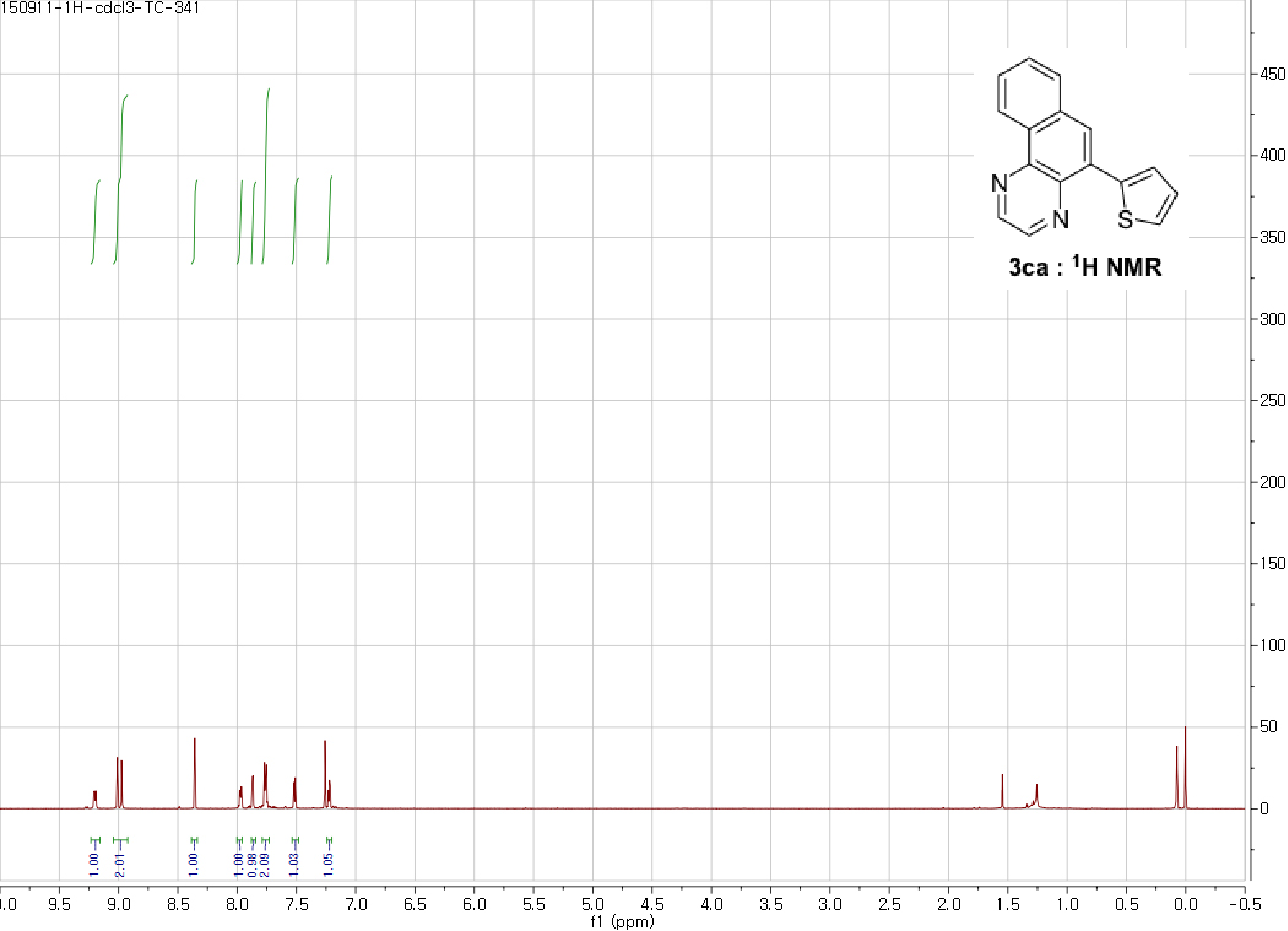

150911-1H-cdcl3-TC-344
3ba : 1H NMR
N
S
0.97
0.98
1.00
2.01
1.03
1.08
1.08
0.94
0.99
1.00
f1 (ppm)

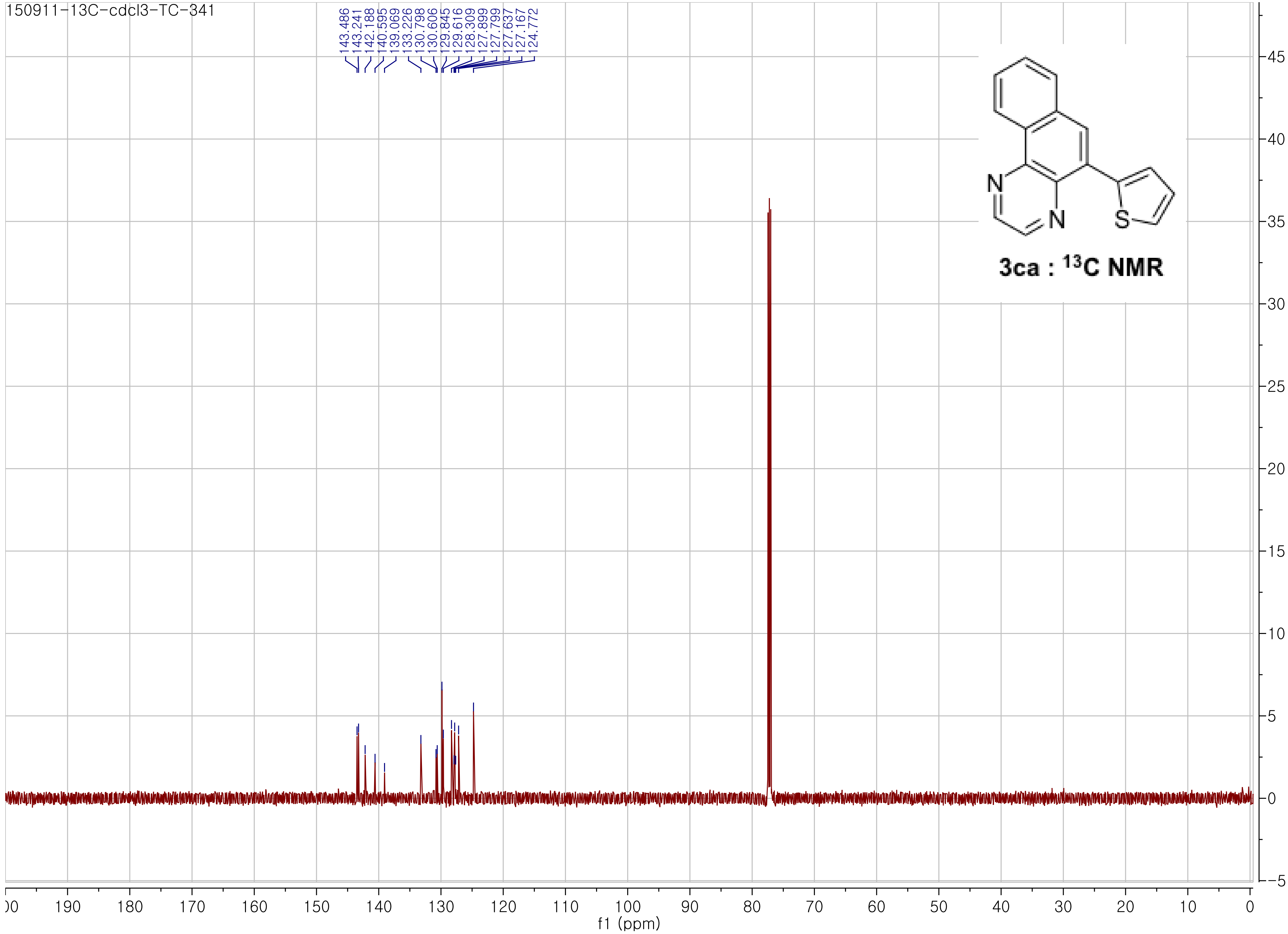

150911-13C-cdcl3-TC-344
148.995
146.988
140.729
134.296
132.986
131.514
130.112
129.512
128.801
128.162
127.971
127.683
127.641
126.171
125.948
124.717
122.012
N
S
3ba : 13C NMR
f1 (ppm)

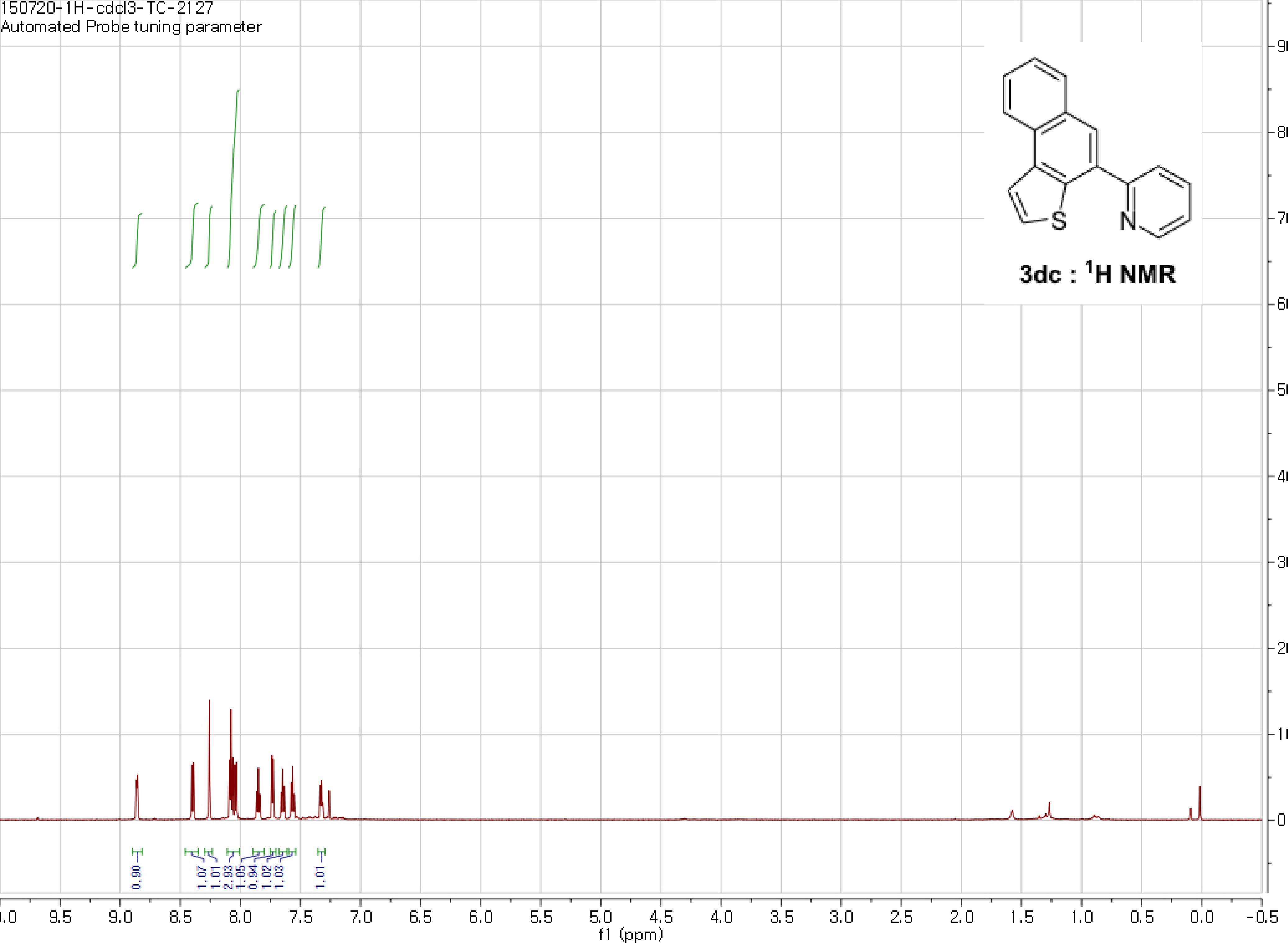

150911-1H-cdcl3-TC-345-1
3bc : 1H NMR
0.93
0.93
1.00
0.93
1.88
1.08
1.01
0.94
1.03
1.08
f1 (ppm)

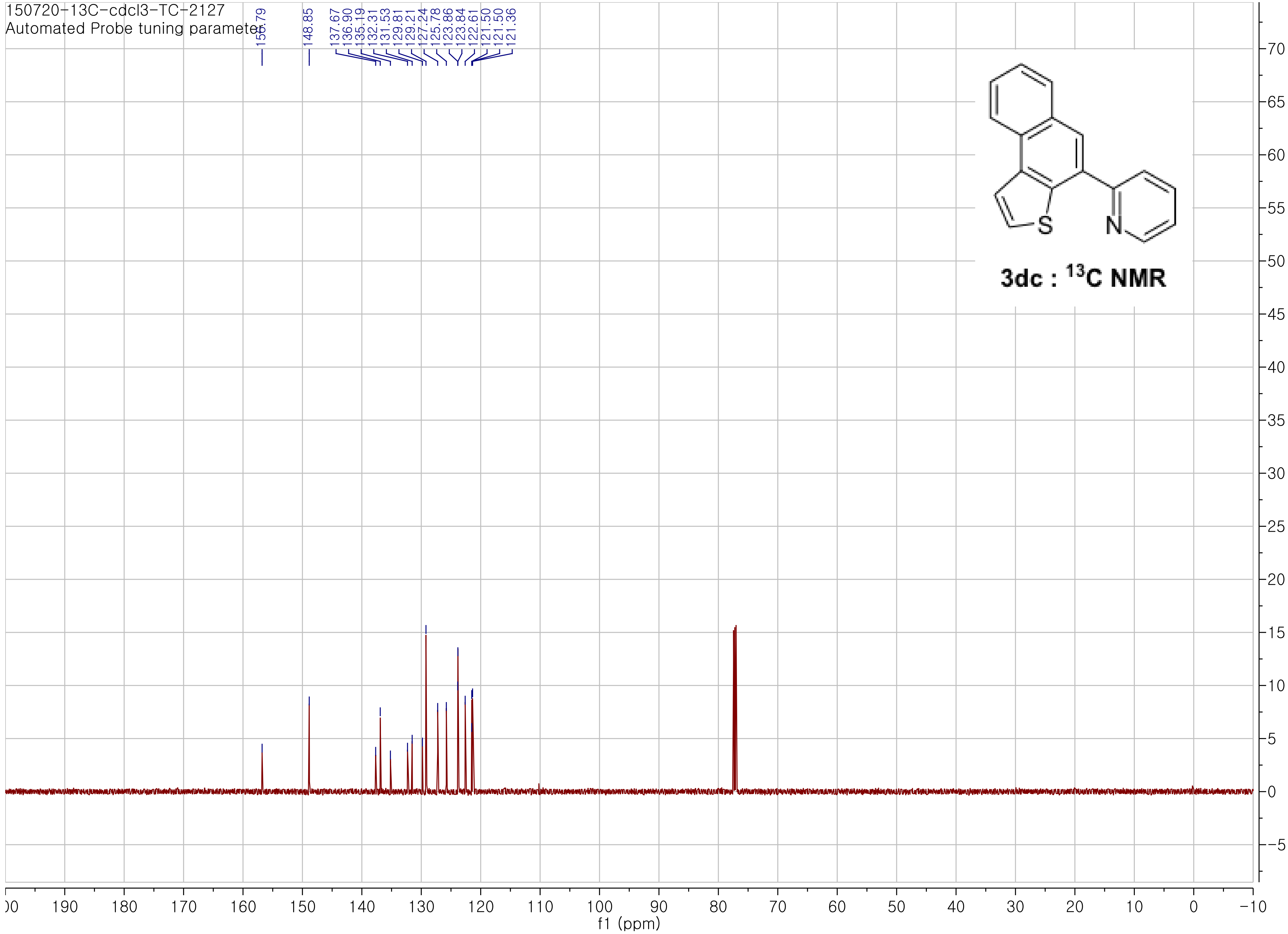


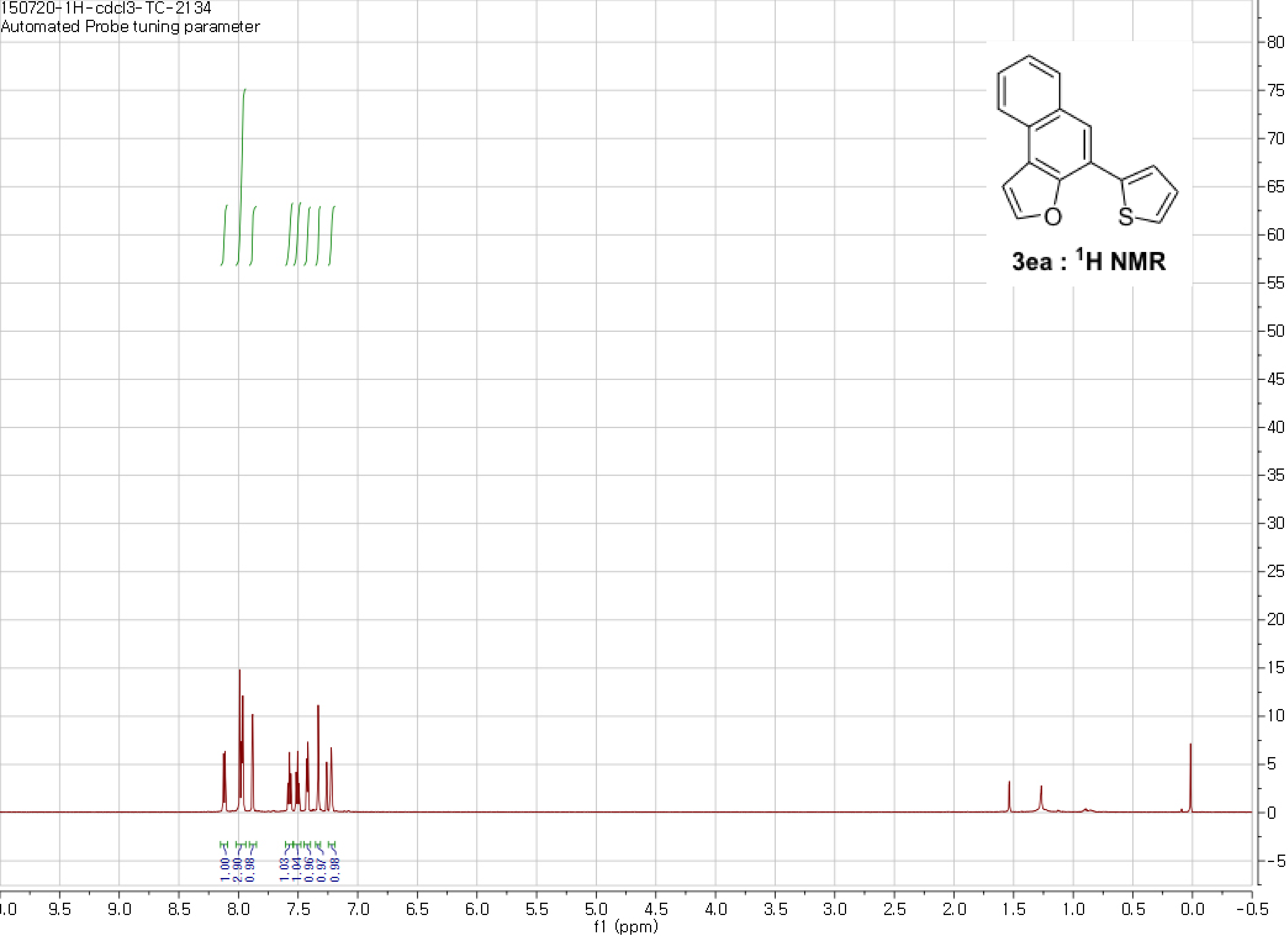
150911-13C-cdcl3-TC-345-1
158.374
149.490
148.736
146.886
136.860
135.870
134.275
132.792
131.623
128.911
128.895
128.457
128.167
127.536
124.941
124.499
122.361
121.743
N
N
3bc : ¹³C NMR
f1 (ppm)

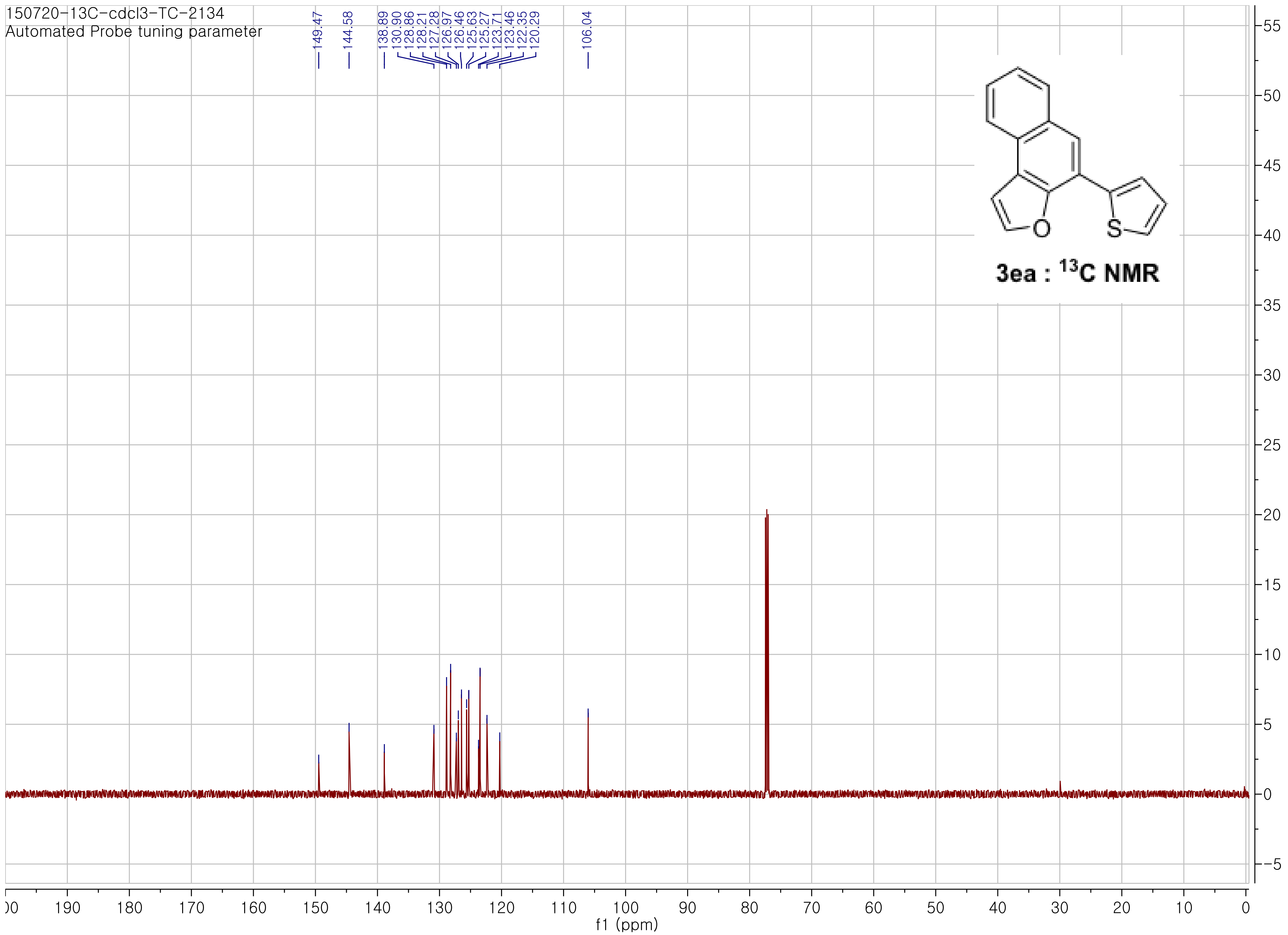

150911-1H-cdcl3-TC-341
3ca : 1H NMR
N
N
S
1.00
2.01
1.00
1.00
0.98
2.09
1.03
1.05
450
400
350
300
250
200
150
100
50
0
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

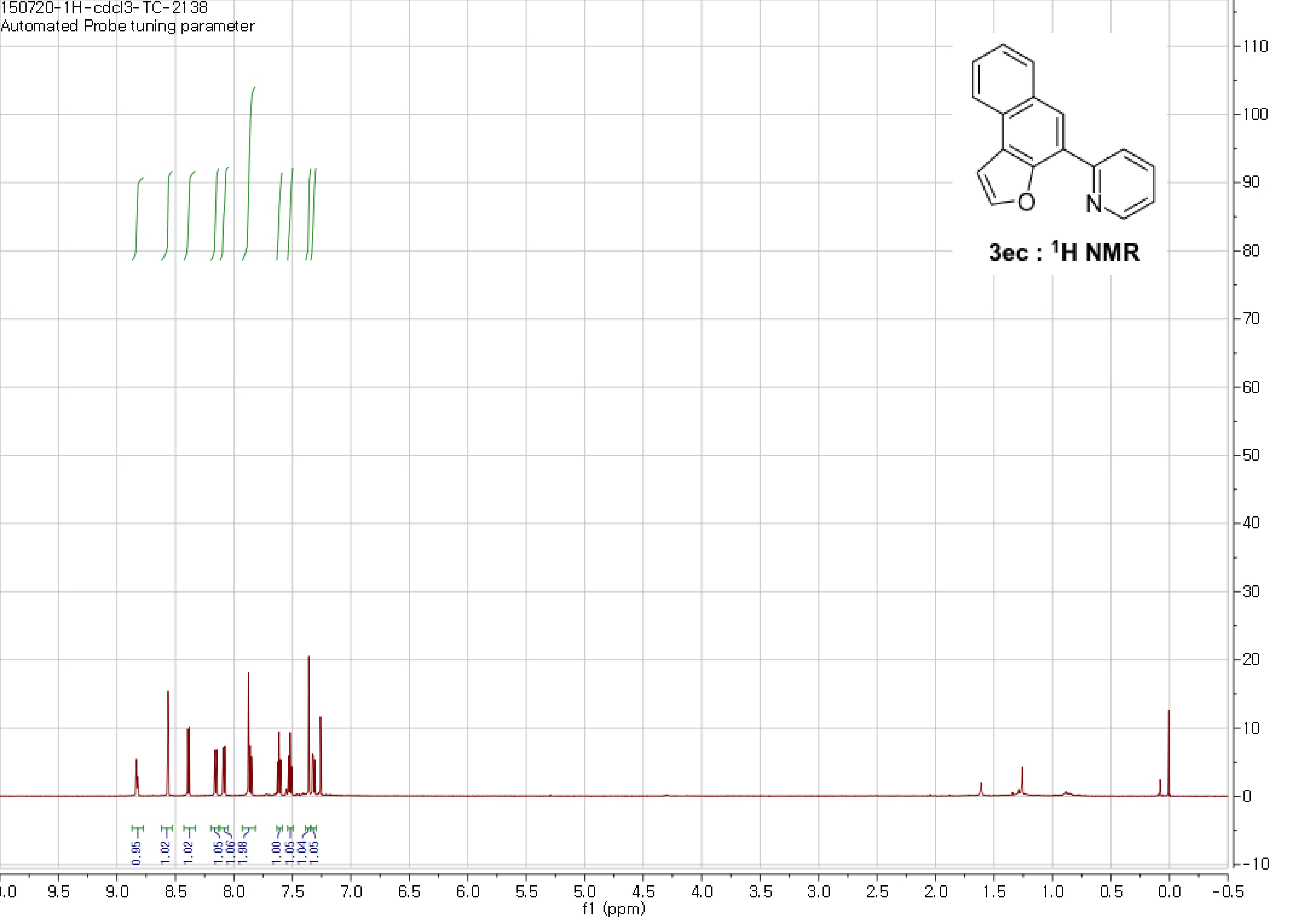

150911-13C-cdcl3-TC-341
143.486
143.241
142.188
140.595
139.069
133.226
130.798
130.606
129.845
129.616
128.309
127.899
127.799
127.637
127.167
124.772
N
N
S
3ca : ^{13}C NMR
f1 (ppm)

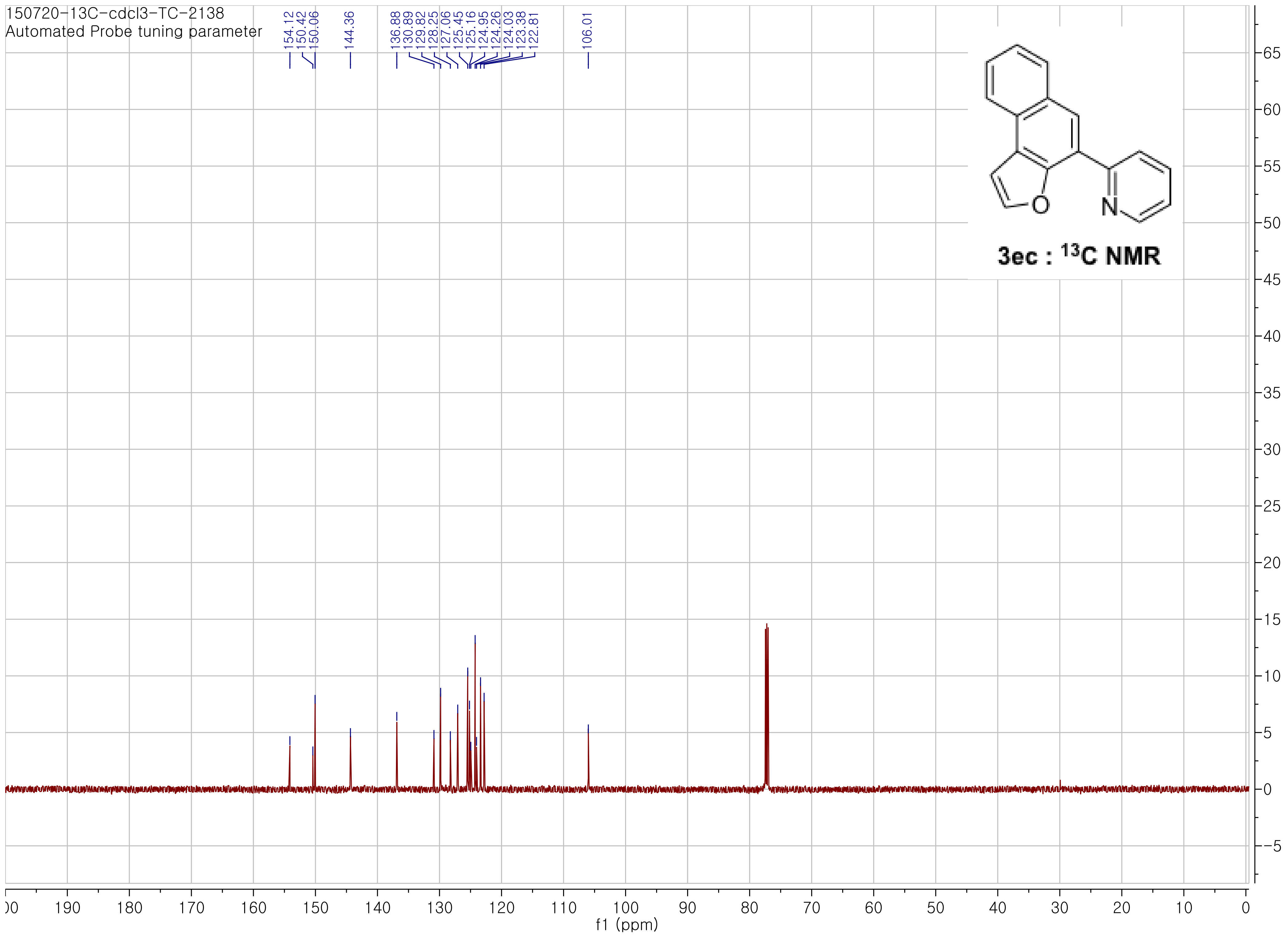

150720-1H-cdcl3-TC-2127
Automated Probe tuning parameter
S
N
3dc : ^{1}H NMR
0.90
1.07
1.01
2.93
1.05
0.94
1.02
1.03
1.01
f1 (ppm)


150720-13C-cdcl3-TC-2127
Automated Probe tuning parameter
156.79
148.85
137.67
136.90
135.19
132.31
131.53
129.81
129.21
127.24
125.78
123.86
123.84
122.61
121.50
121.50
121.36
S
N
3dc : 13C NMR
f1 (ppm)

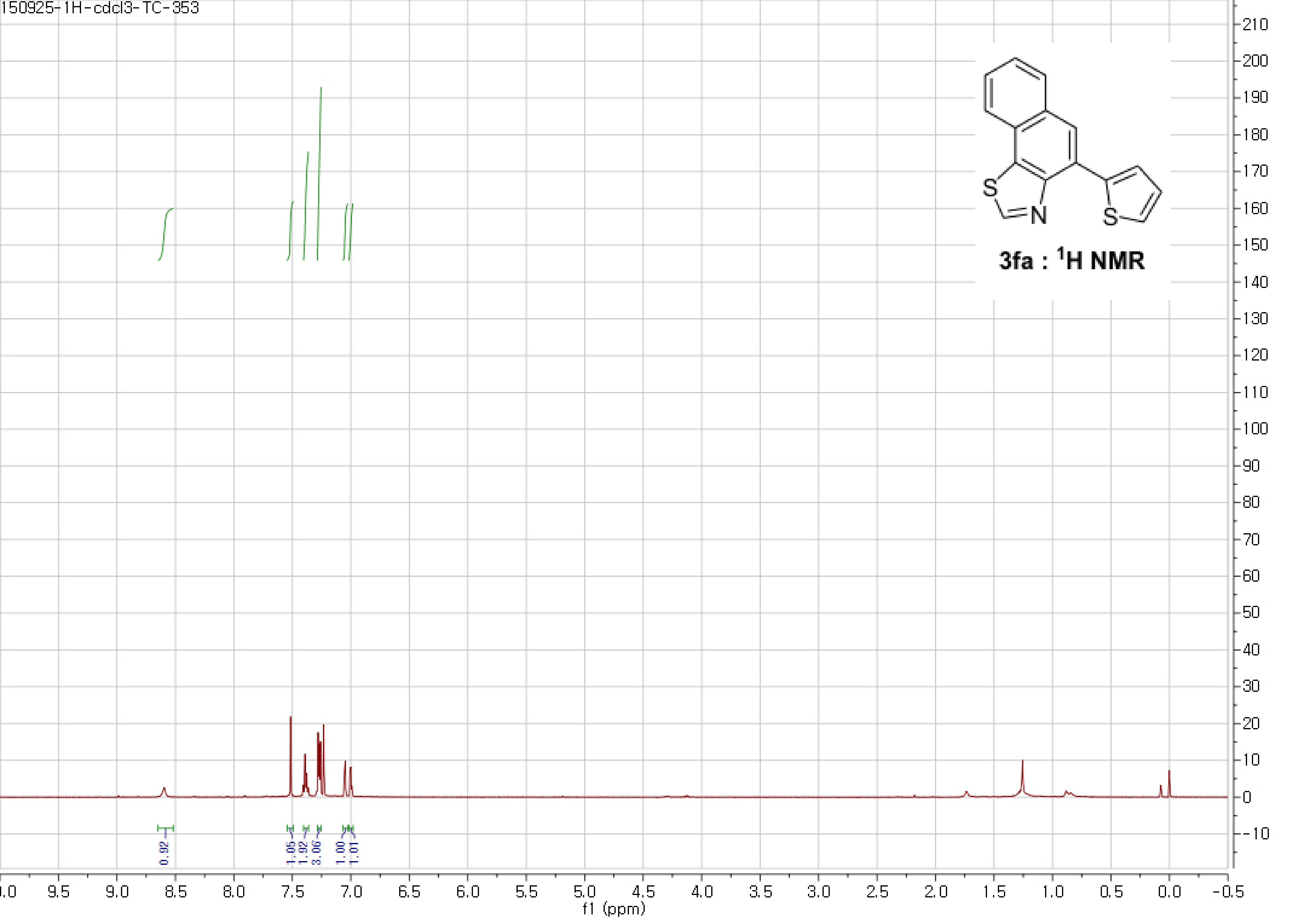

150720-1H-cdcl3-TC-2134
Automated Probe tuning parameter
3ea : ¹H NMR
O
S
1.00
2.90
0.98
1.03
1.04
0.96
0.97
0.98
f1 (ppm)

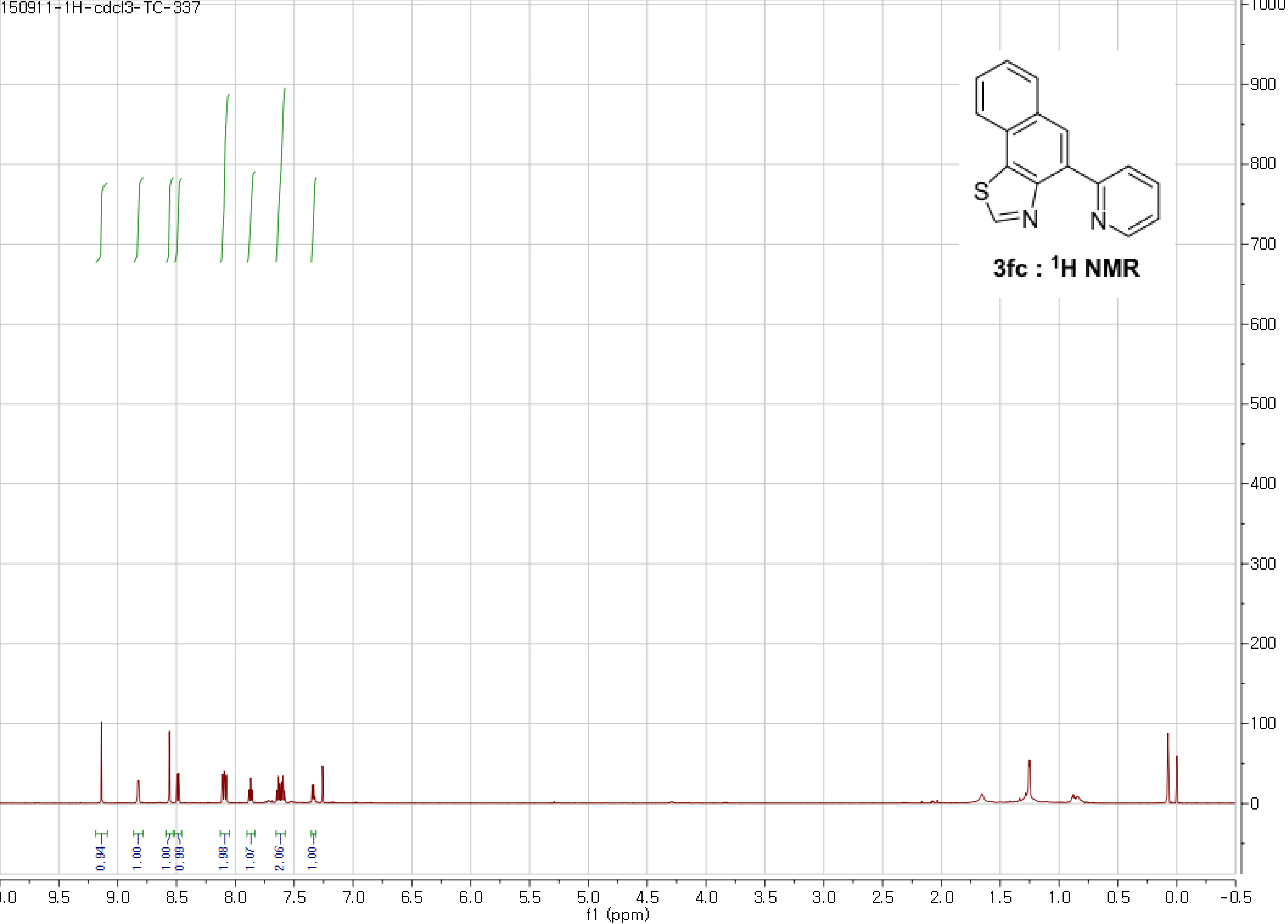

150720-13C-cdcl3-TC-2134
Automated Probe tuning parameter
149.47
144.58
138.89
130.90
128.86
128.21
127.28
126.97
126.46
125.63
125.27
123.71
123.46
122.35
120.29
106.04
O
S
3ea : 13C NMR
f1 (ppm)

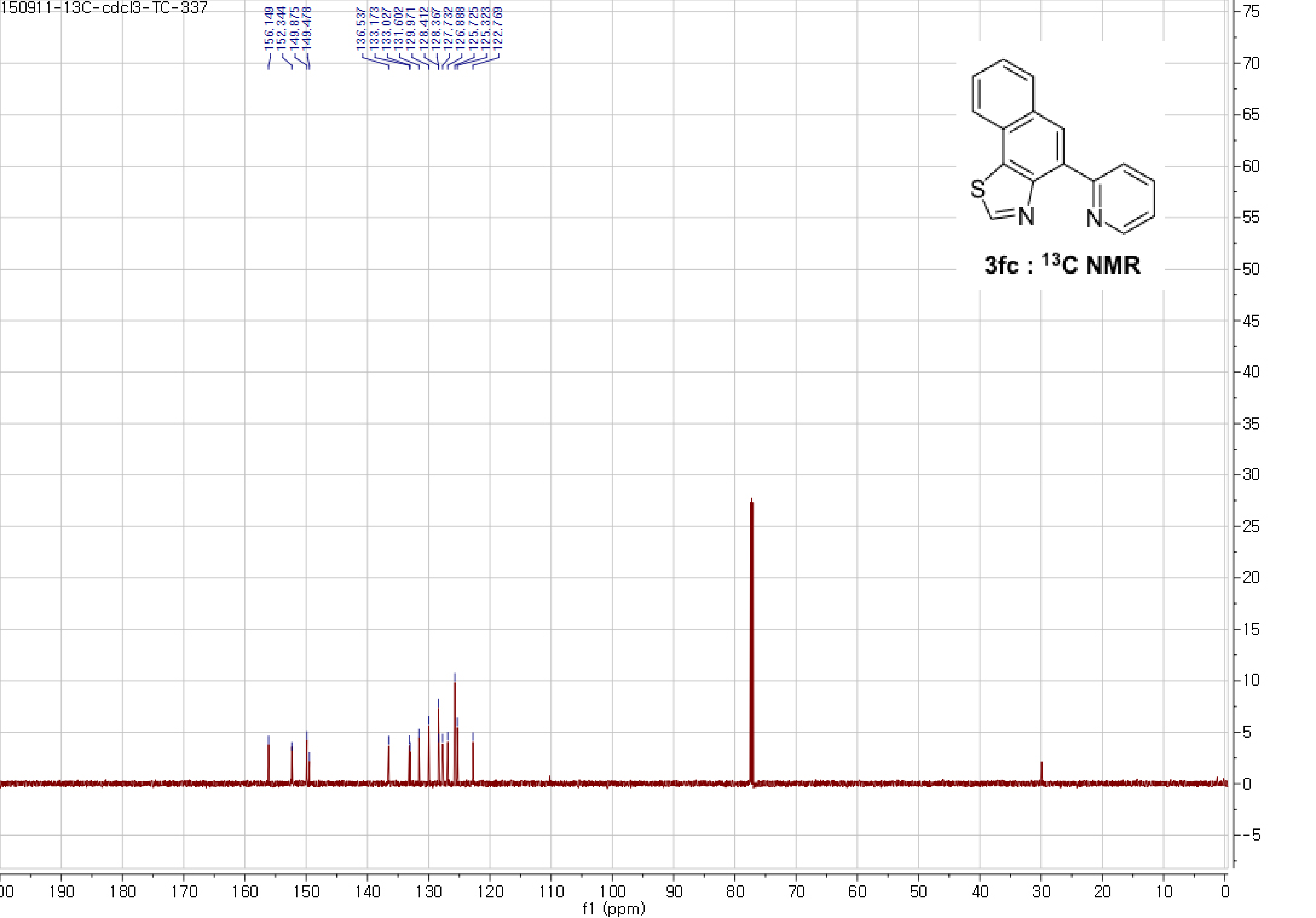

150720-1H-cdcl3-TC-2138
Automated Probe tuning parameter
3ec : 1H NMR
O
N
0.95
1.02
1.02
1.05
1.06
1.98
1.00
1.05
1.04
1.05
f1 (ppm)


150720-13C-cdcl3-TC-2138
Automated Probe tuning parameter
154.12
150.42
150.06
144.36
136.88
130.89
129.82
128.25
127.06
125.45
125.16
124.95
124.26
124.03
123.38
122.81
106.01
f1 (ppm)
O
N

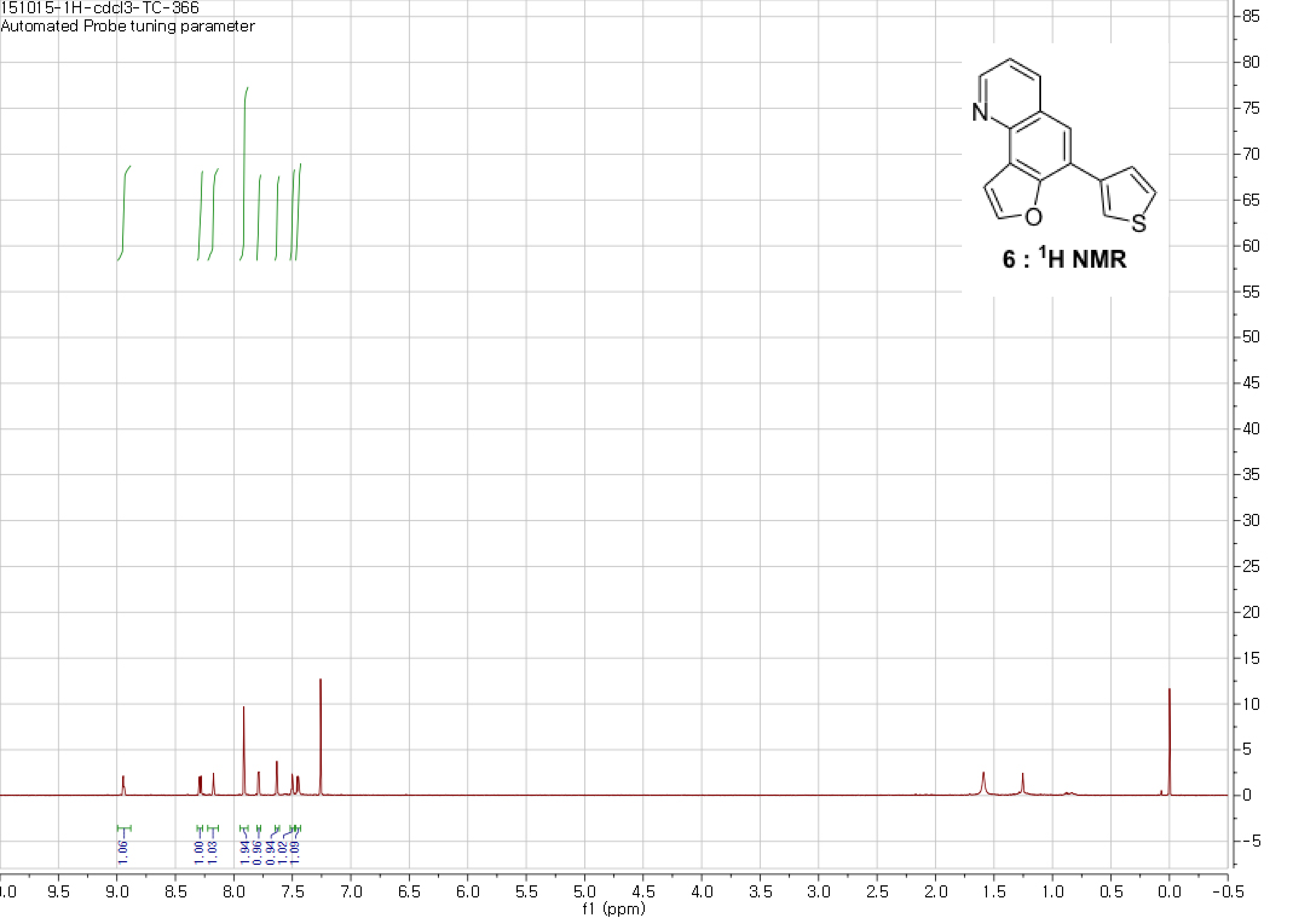
3ec : ^{13}C NMR


150925-1H-cdcl3-TC-353
S
N
S
3fa : ¹H NMR
0.92
1.05
1.92
3.06
1.00
1.01
f1 (ppm)

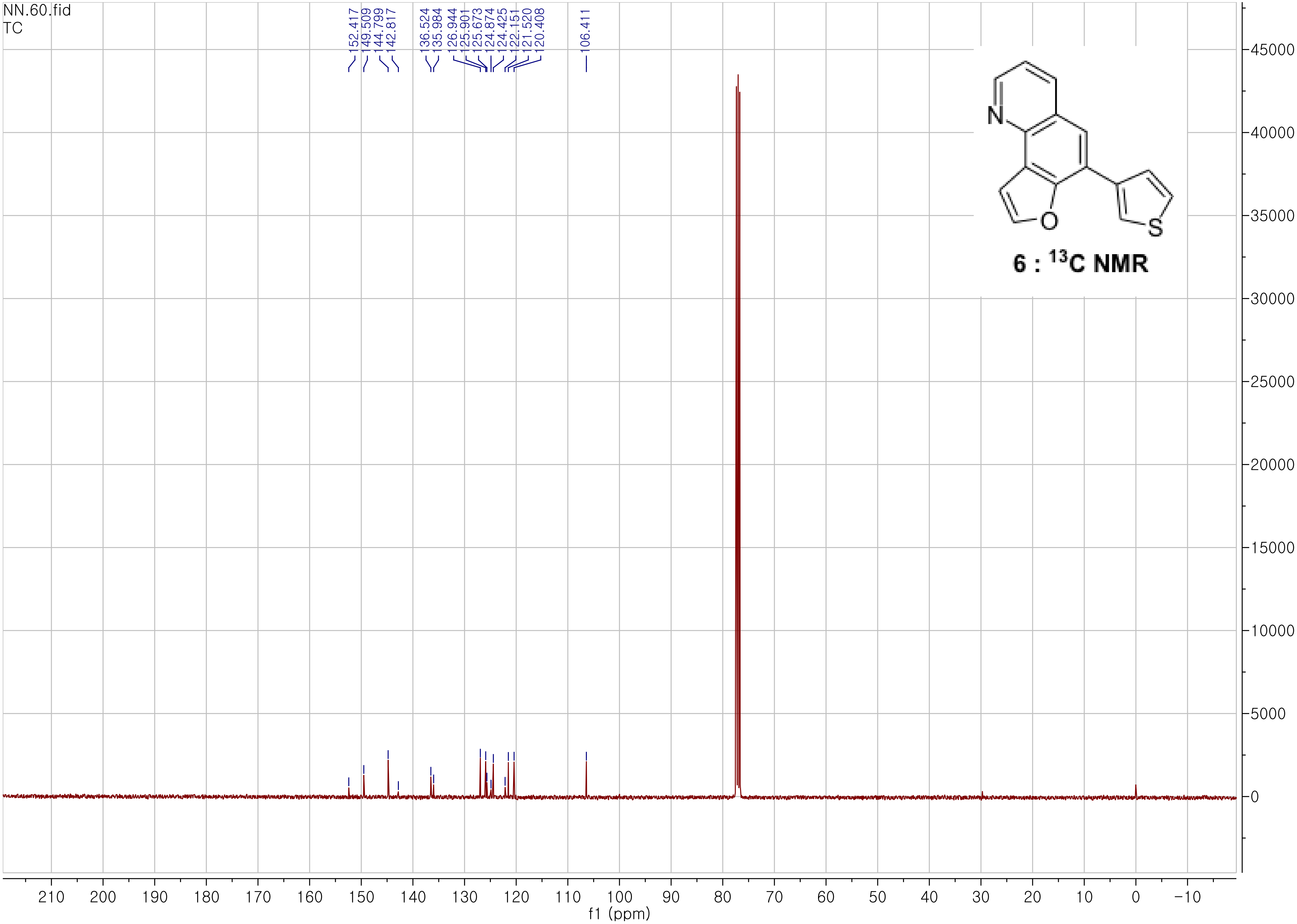

150911-1H-cdcl3-TC-337
3fc : 1H NMR
S
N
N
0.94
1.00
1.00
0.99
1.98
1.07
2.06
1.00
f1 (ppm)



150911-13C-cdcl3-TC-337
156.149
152.344
149.875
149.478
136.537
133.173
133.027
131.602
129.971
128.412
128.367
127.732
126.888
125.725
125.323
122.769
S
N
N
3fc : 13C NMR
f1 (ppm)



151015-1H-cdcl3-TC-366
Automated Probe tuning parameter
6 : 1H NMR
1.06
1.00
1.03
1.94
0.96
0.94
1.02
1.09
f1 (ppm)


NN.60.fid
TC
152.417
149.509
144.799
142.817
136.524
135.984
126.944
125.901
125.673
124.874
124.425
122.151
121.520
120.408
106.411
6 : 13C NMR
N
O
S
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)