Supporting Information

On-Water Accelerated Sulfenylation of Indole Derivatives under Visible Light Irradiation

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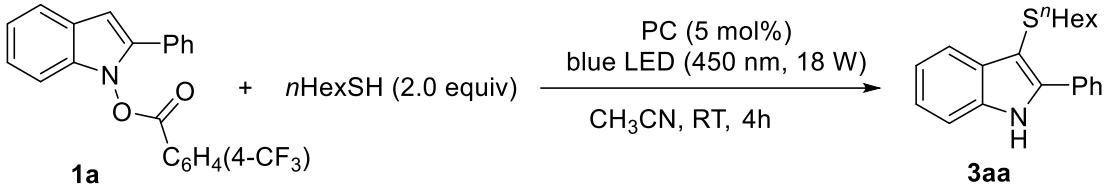
1. General Information

All solvents were dried and distilled according to standard methods before use. Synthesis of N-carboxyindoles **1a**, **1c-f**, and **1i-j** has been reported.[1-3] All other materials were purchased from TCI, Aldrich, or Alfa Aesar and were used as received. Photocatalysis was conducted with LED lamps and photoredox box from Hepatochem (365 nm lamp: HCK1012-01-011; 405 nm lamp: Part # HCK-1012-01-010; 450 nm lamp: Part # HCK-1012-01-002; 525 nm lamp: # HCK1012-01-004; Photobox: Part # HCK-1006-01-016). TLC (thin-layer chromatography) analyses were carried out on Merck silica gel 60 F254 TLC plates and was visualized with UV lamp and $KMnO_4$ solution. Flash chromatography was performed on Kieselgel 60 (230-400 mesh). ^{1}H and ^{13}C NMR spectra were recorded on a Bruker (400 MHz) spectrometer with TMS as an internal standard. High resolution mass spectra (HRMS) were obtained from Organic Chemistry Research Center in Sogang University.

2. Optimization Study

Table S1. Screening of Photocatalysts with nHexSH (**2a**).[a,b]



entry	PC	M*/M$^-$ (V)	M/M$^-$ (V)	M$^+$/M* (V)	M$^+$/M (V)	E_T (kcal/mol)	Conv (%)	Yield[c] (**3aa**)
1	$Ir(ppy)_2(dtbbpy)PF_6$	0.66[1]	-1.51[1]	-0.96[1]	1.21[1]	49.2[3]	>99	52%
2	Rhodamin B (in water)	0.84[4]	-0.96[4]	-0.89[4]	0.91[4]	41.5[4]	83	49%
3	Eosin Y (in MeOH)	0.83[4]	-1.08[4]	-1.15[4]	0.76[4]	44.0[4]	76	48%
4	Fluorescein	0.77[4]	-1.17[4]	-1.07[4]	0.87[4]	44.7[4]	68	46%
5	$Ru(bpy)_3(PF_6)_2$	0.77[1]	-1.33[1]	-0.81[1]	1.29[1]	46.5[3]	69	40%
6	$Ru(bpz)_3(PF_6)_2$	1.45[5]	-0.80[5]	-0.26[5]	1.86[5]	48.4[6]	75	39%
7	*fac*-$Ir(ppy)_3$	0.31[1]	-2.19[1]	-1.73[1]	0.77[1]	57.8[3]	>99	30%
8	Benzophenone	1.28[1]	-1.72[1]	-0.61[1]	2.39[1]	69.1[4]	28	17%

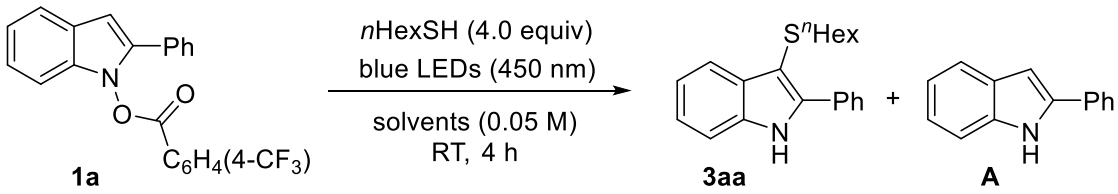
[a]**1a** (0.05 mmol), *n*HexSH (0.1 mmol) in CH_3CN (0.1 M); [b]Photophysical/electrochemical data (vs. SCE). [c]The yield was estimated by the crude ^1H NMR spectra with 2,5-dibromopyrimidine as an internal standard.

*References to the photophysical/electrochemical data in Table S1:

[1] K. Kwon, R. T. Simmons, M. Nandakumar, J. L. Roisen, *Chem. Rev*. **2022**, *122*, 2353.
[2] Vlcek, A. A.; Dodsworth, E. S.; Pietro, W. J.; Lever, A. B. P. *Inorg. Chem*. **1995**, *34*, 1906.
[3] Zhou, Q. –Q.; Zou, Y. –Q.; Lu, Z. –Q.; Xiao, W. –J. *Angew. Chem. Int. Ed*. **2019**, *58*, 1586.
[4] Romero, N. A.; Nicewicz, D. A. *Chem. Rev*. **2016**, *106*, 10075.
[5] Murray, P. R. D.; Cox, J. H.; Chiappini; N. D.; Roos, C. B.; McLoughlin, E. A.; Hejna, B. G.; Nguyen, S. T.; Ripberger, H. H.; Ganley, J. M.; Tsui, E.; Shin, N. Y.; Koronkiewicz, B.; Qiu, G.; Knowles, R. R. *Chem. Rev.* **2022**, *122*, 2017-2291.
[6] Dutta, S.; Erchinger, J. E.; Strieth-Kalthoff, F.; Kleinmans, R.; Glorius, F. *Chem. Soc. Rev*. **2024**, *53*, 1068–1089.

* No clear trends appeared between the reaction efficiency and the properties of the photocatalysts, such as redox potential and triplet energy.

Table S2. Screening of solvents and additives.[a]



Entry	Solvent	Conv (%)	**3aa** (%)[b]	**A** (%)
1	CH_3CN	58	33	10
2	THF	>99	64	5
3	MeOH	>99	35	-
4	EtOH	>99	50	17
5	CH_2Cl_2	82	47	11
6	MTBE	73	44	8
7	Ether	86	66	10
8	H_2O	59	48	8
9	H_2O: CH_3CN (1:3)	62	50	12
10	H_2O: CH_3CN (1:1)	70	50	13
11	H_2O: CH_3CN (3:1)	79	52	15
12	H_2O, CTAB (1.5 eq)[c]	86	14	9
13	H_2O, polysorbate 80 (1.5 eq)	48	3	4
14	H_2O, celite (50 mg)	>99	83	15
15	H_2O, SiO_2 (50 mg)	>99	77	19
16	H_2O, MS 4Å (50 mg)	92	70	13
17	H_2O (0.1 M), celite (50 mg)	>99	78	19
18	H_2O (0.01 M) celite (50 mg)	74	60	13
19[d]	H_2O, celite (50 mg)	>99	63	26
20[e]	H_2O, celite (50 mg)	0	-	-
21	H_2O, celite (50 mg) EtSH (0.1 mmol)	73	53	13
22	H_2O, celite (50 mg) EtSH (0.3 mmol)	>99	74	15
23	H_2O, celite (100 mg)	68	53	10
24	EtOH, celite (50 mg)	86	45	17
25	CH_3CN, celite (50 mg)	54	37	9
26	ether, celite (50 mg)	87	66	13

27	Neat	41	29	10

[a]**1a** (0.05 mmol), EtSH (0.2 mmol) in solvents (1.0 mL), unless otherwise noted. [b]The yield was estimated by the crude ^{1}H NMR spectra with 2,5-dibromopyrimidine as an internal standard. [c]CTAB: cetyltrimethylammonium bromide. [d]λ = 405 nm. [e]λ = 525 nm.

Fig. S1. Chromatogram of reaction mixtures of **1a** (0.05 mmol) and *n*HexSH (**2a**, 0.2 mmol) in different solvents (1 mL) after irradiation with 450 nm LEDs for 4 h.

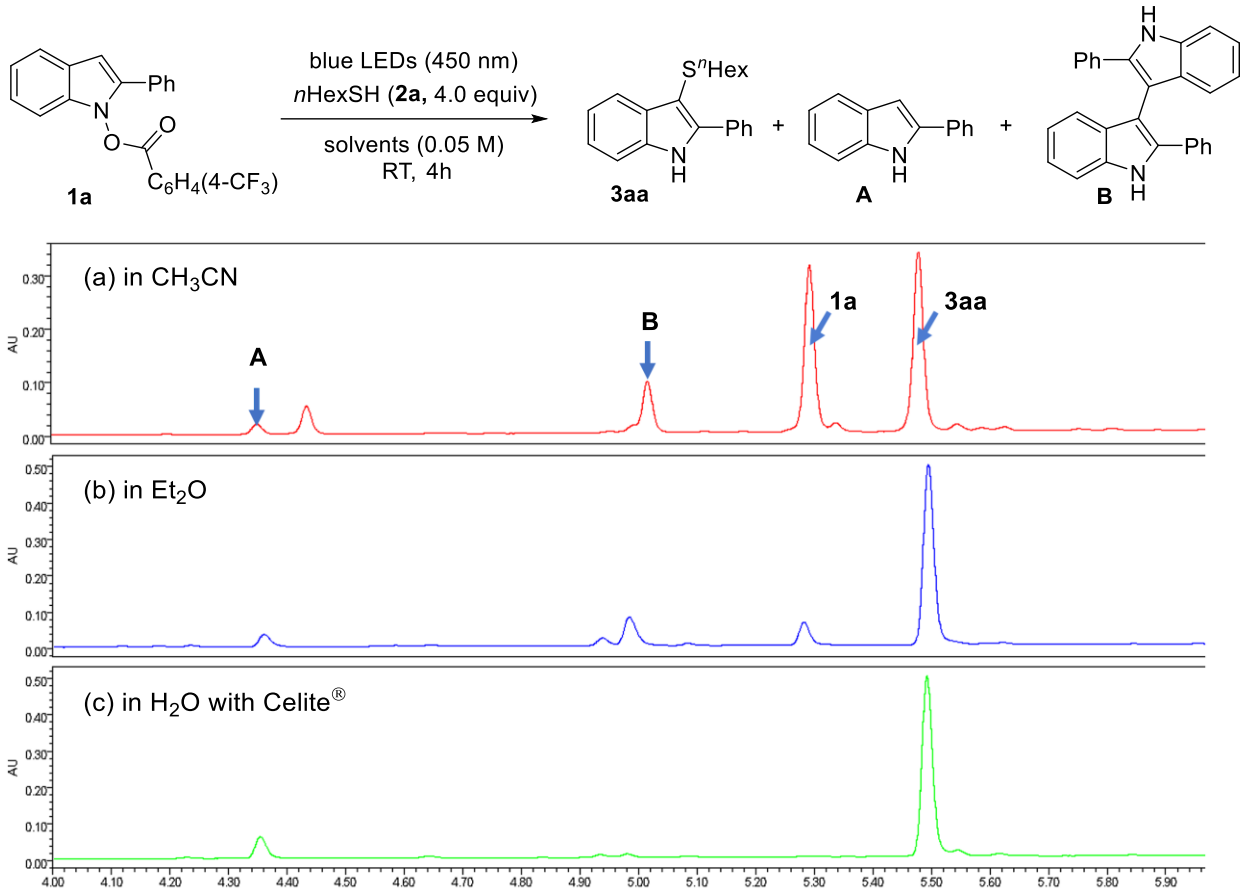
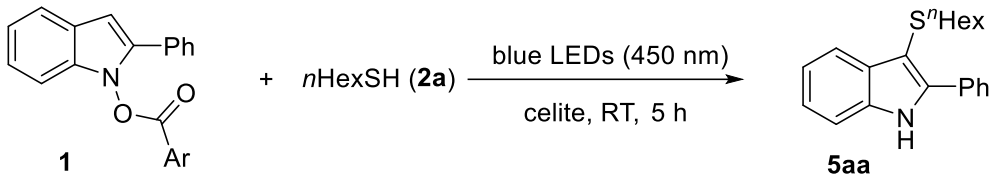


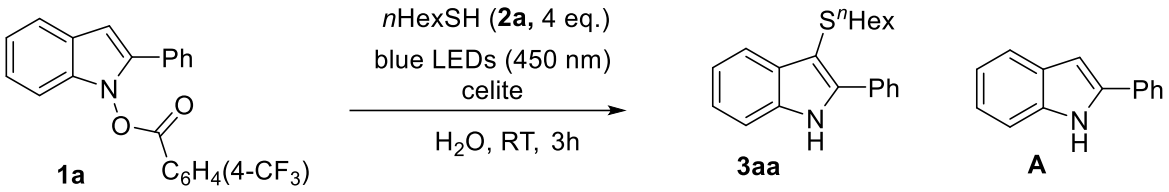
Table S3. The effect of leaving groups of **1a**.[a]



Entry	Substrate (**1**)	Ar	**3aa** (%)
1	**1a**	4-CF_3-C_6H_4	83
2	**1a-1**	4-Cl-C_6H_4	73
3	**1a-2**	4-CH_3-C_6H_4	74
4	**1a-3**	4-MeO-C_6H_4	64
5	**1a-4**	2,4,6-Me_3-C_6H_2	69

[a]**1a** (0.05 mmol), *n*HexSH (0.2 mmol) and Celite® in solvents (1.0 mL) under irradiation with blue LEDs; The yield of **3aa** was based on the ^{1}H NMR spectra.

Table S4. The effect of the amount of reagents and stirring speed.[a]



Entry	Stirring speed (rpm)[b]	Conv (%)	**3aa** (%)	**A** (%)
1	0	36	28	6
2	300	69	55	12
3	700	73	59	13
4	1200	74	57	12

[a]**1a** (0.05 mmol), *n*HexSH, and Celite (50 mg) in H_2O (0.5 mL). [b]The speed of magnetic stirrer.

3. Representative Procedures

3.1 Synthesis of C3-sulfenyl indoles

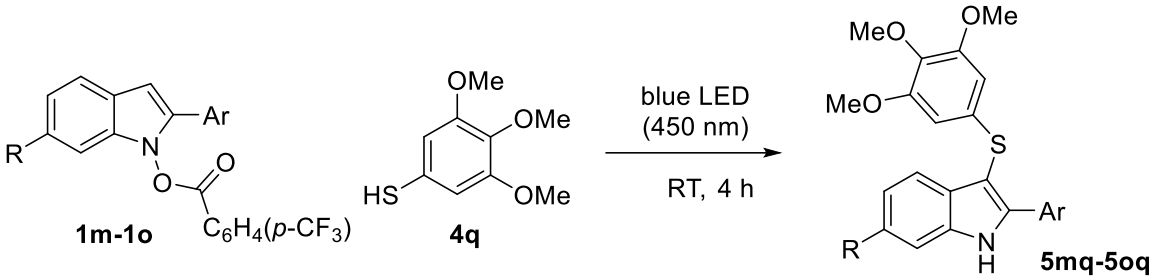
Condition A (Table 2, footnote a): Synthesis of **3aa** "on water"

In a 4 mL vial, indole derivative **1a** (38.1 mg, 0.1 mmol), *n*-hexanethiol **2a** (47.3 mg, 0.4 mmol) and Celite® (100.0 mg) was suspended in water (2.0 mL) and the vial was capped under air. The resulting suspension was stirred at 700 rpm and was irradiated with 450 nm LEDs (18W EvoluChem Spotlight LED from Hepatochem) inside a photoredox box kept under 25~28 ^{o}C. After TLC indicated a completion of reaction, the mixture was extracted with CH_2Cl_2 (2 mL x 3). The combined layers were dried ($MgSO_4$), filtered, and concentrated to dryness. The residue was purified on a silica gel (EtOAc:Hex = 1:20~1:4) to give **3aa** (25.1 mg, 81%) as a pale yellow oil.

Condition B (Table 2, footnote d; Table 3, footnote b): Synthesis of **3al** in CH_3CN

In a 4 mL vial, indole derivative **1a** (38.1 mg, 0.1 mmol), 2-thiazoline-2-thiol **2l** (47.6 mg, 0.4 mmol) and Celite® (100.0 mg) was dissolved in acetonitrile (2.0 mL) and the vial was capped under air. The resulting suspension was stirred at 700 rpm and was irradiated with 405 nm LEDs (18W EvoluChem Spotlight LED from Hepatochem) inside a photoredox box kept under 25~28 ^{o}C. After TLC indicated a completion of reaction, the mixture was extracted with CH_2Cl_2 (2 mL x 3). The combined layers were dried ($MgSO_4$), filtered, and concentrated to dryness. The residue was purified on a silica gel (EtOAc:Hex = 1:20~1:4) to give **3al** (14.3 mg, 52 % yield) as a yellow solid.

3.2.2 Synthesis of Biologically Active Thioindoles.

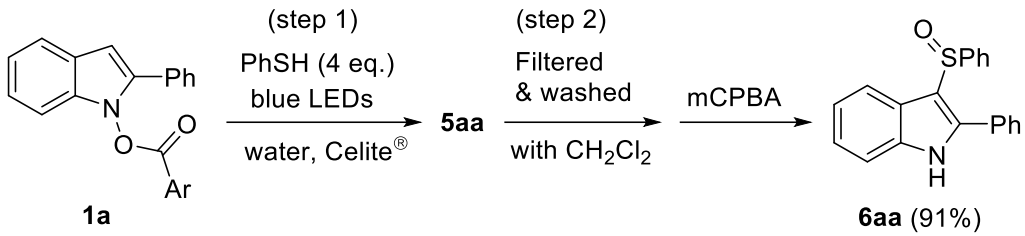


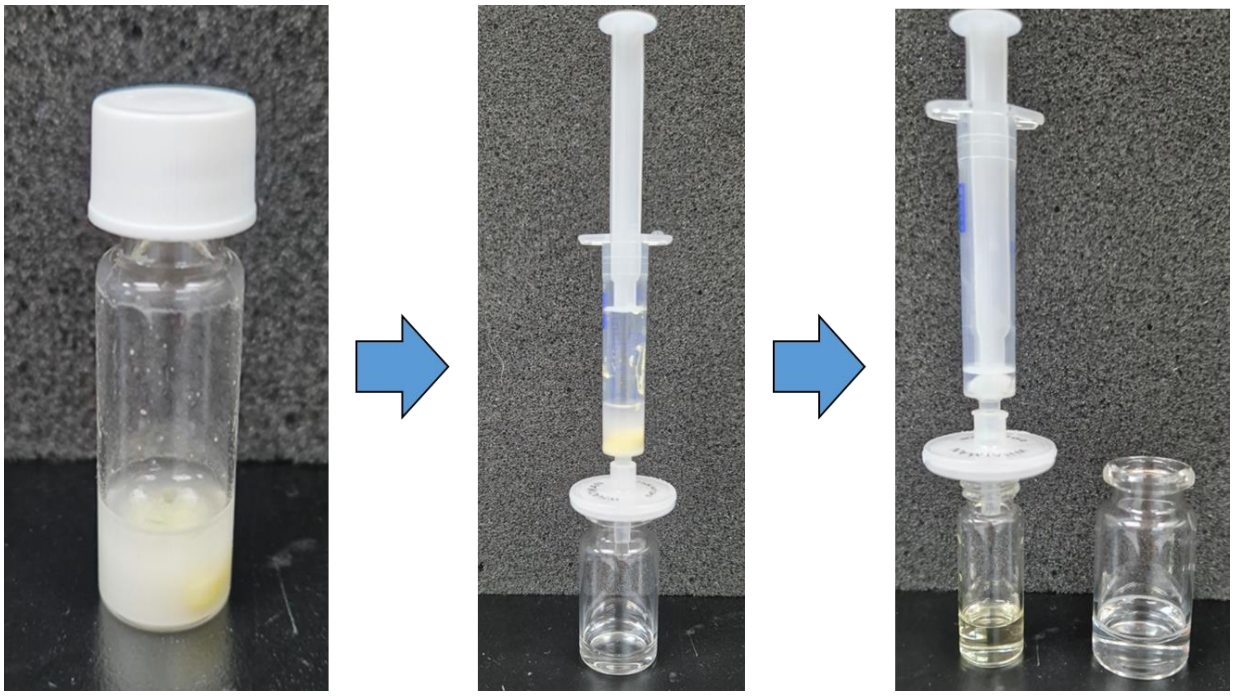
Representative Procedure (Condition A) was followed, employing substrates **1m-o** and **4q**

Conditions: **1m-1o** (0.1 mmol), **4q** (0.4 mmol), and Celite (100 mg) in water (2 mL)

3.2 Synthetic applications of sulfenylindoles

3.2.1 Isolation of **5aa** by filtration and in-situ oxidation into **6aa**.





(step 1)
after reaction

(step 2-1)
washed with H_2O
(1 mL x 2)

(step 2-2)
dissolved with CH_2Cl_2
(1 mL)

Fig. S2. Isolation of the crude product by filtration, followed by in-situ reaction.

In a 4 mL vial, **1a** (19.1 mg, 0.1 mmol), benzenethiol **2a** (22.0 mg, 0.4 mmol), and Celite® (50.0 mg) was suspended in water (1.0 mL). The resulting suspension was stirred at 700 rpm, while irradiated with 450 nm LEDs (18W EvoluChem Spotlight LED from Hepatochem) inside a photoredox box kept under 25~28 °C. After reaction, the reaction mixture was filtered through a syringe filter (pore size: 0.45 um, 25 mm diameter, Whatman, cat no. 6750-2504) and was

washed with water (1 mL x 2). The filtrant containing crude product (**5aa**) inside the syringe was dissolved into a vial with CH_2Cl_2 (1 mL x 2). The CH_2Cl_2 solution was cooled to 0°C and was treated with mCPBA (22.4 mg, 0.2 mmol). The mixture was allowed to warm to RT over 2h. After TLC indicated a completion of reaction, the mixture was washed with 1 mL of 5% aq. $NaHCO_3$ solution and the aqueous layer was extracted with CH_2Cl_2 (1 mLx2). The combined organic layers were dried ($MgSO_4$), filtered, and was concentrated to dryness. The residue was purified on a silica gel (EtOAc:Hex = 1:10~1:4) to give **6aa** (14.4 mg, 91%) as a pale yellow oil.

4. Mechanistic Study

4.1. Attempts to Measure the Eutectic Melting Temperature

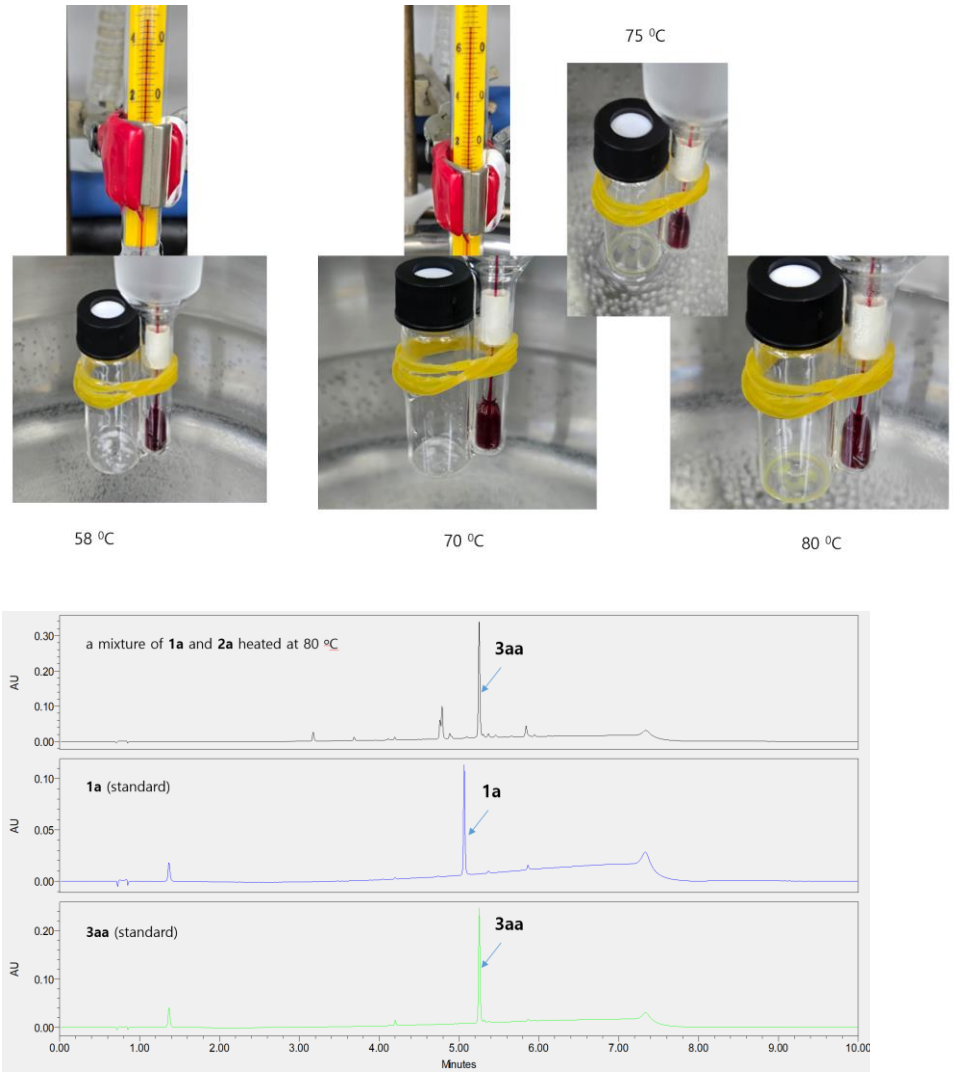
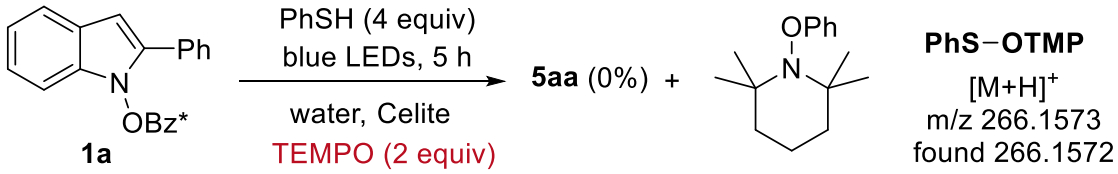


Fig. S3. Attempts to measure the eutectic melting temperature: a thermal reaction to **3aa**

A mixture of solid **1a** (9.6 mg) and liquid **2a** (12.0 mg, 4 equiv) was placed in a vial and was slowly heated in an oil bath. At room temperature, the solid in the mixture remains undissolved. Between 70~80 °C, the mixture underwent a thermal reaction to **3aa** (66% of **3aa** according to the HPLC chromatogram) and the color of the heterogeneous mixture turned to pale yellow. It was confirmed that eutectic melting did not occur until 70 °C.

4.2. Radical Trapping Experiment



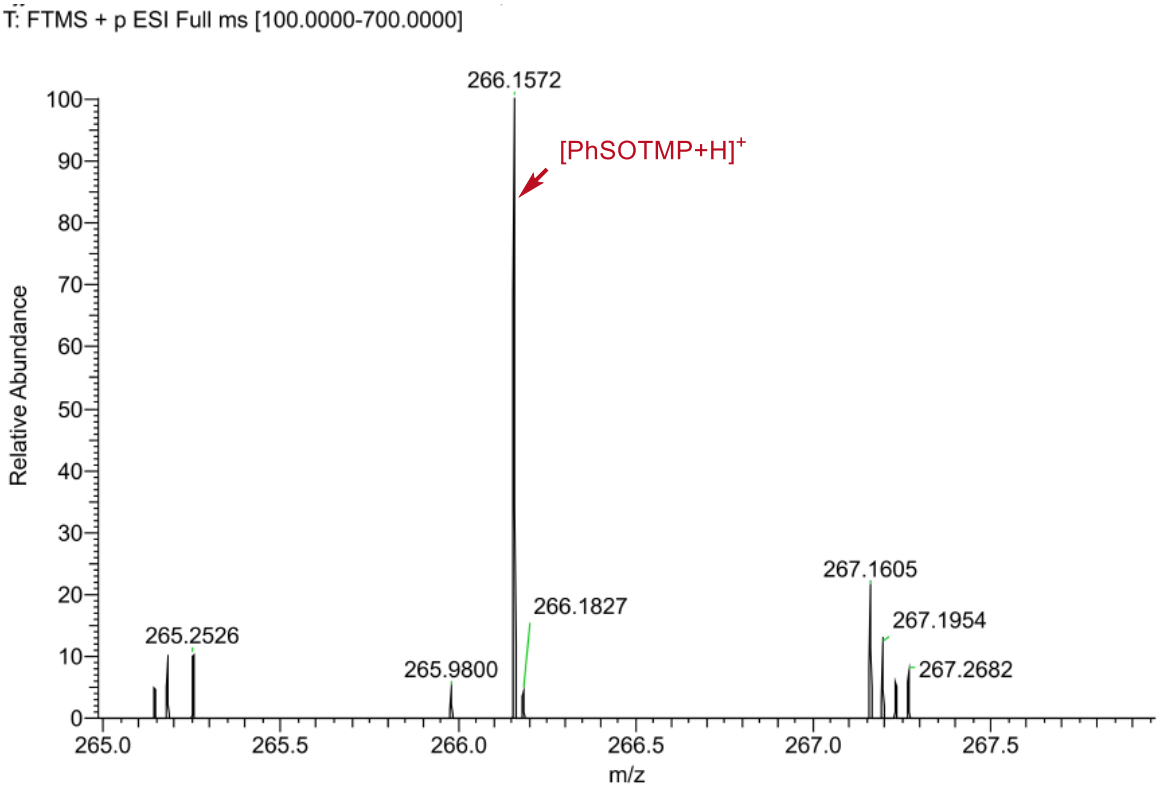
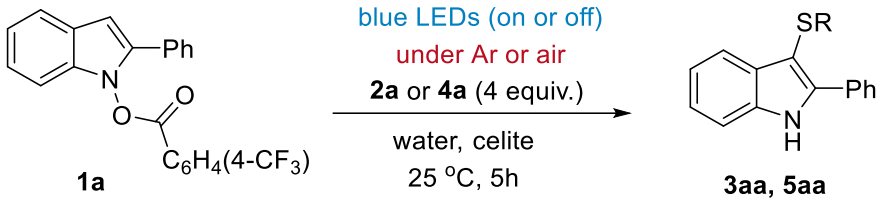


Fig. S4. Mass spectrum of a reaction mixture in the presence of TEMPO.

4.3. Effect of Air and Light

Table S5. The effect of degassing in the presence of 450 nm irradiation.[a]



Entry	Reactants	Blue LEDs	Headspace gas	**3aa** or **5aa** (%)
1	**2a** (*n*HexSH, 4 eq.)	On	Ar[b]	81
2		Off	Ar[b]	0
3		Off	air[c]	0
4	**4a** (PhSH, 4 eq.)	On	Ar[b]	93
5		Off	Ar[b]	1.5
6		Off	air[c]	10

[a]**1a** (0.05 mmol), **2a** or **4a** (0.2 mmol) and Celite® (50 mg) was suspended in water (1 mL). [b]The reaction flask was degassed by freeze-pump-thaw cycles (x3), and was capped under Ar atmosphere. [c]The mixture was stirred in an open tube.

* Under standard conditions, the reaction was performed in a vial capped under air. he reaction with nHexSH (**2a**) under blue LED irradiation proceeded equally well in an Ar atmosphere. However, with the blue LEDs off, the reaction did not proceed at all, even in an open tube, suggesting that the formation of a thiyl radical from aliphatic thiol is purely photochemical. Aromatic thiol, which oxidizes more easily, exhibited slightly different behavior: with the blue LEDs off, the reaction with PhSH (**4a**) produced 10% of **5aa** in an aerobic atmosphere, indicating that generation of a thiyl radical by air partially accounts for the conversion.

In summary, reactions of **1a** with both aliphatic and aromatic thiols proceeded efficiently only in the presence of LEDs irradiation, which suggested that the thiyl radical was generated photochemically.

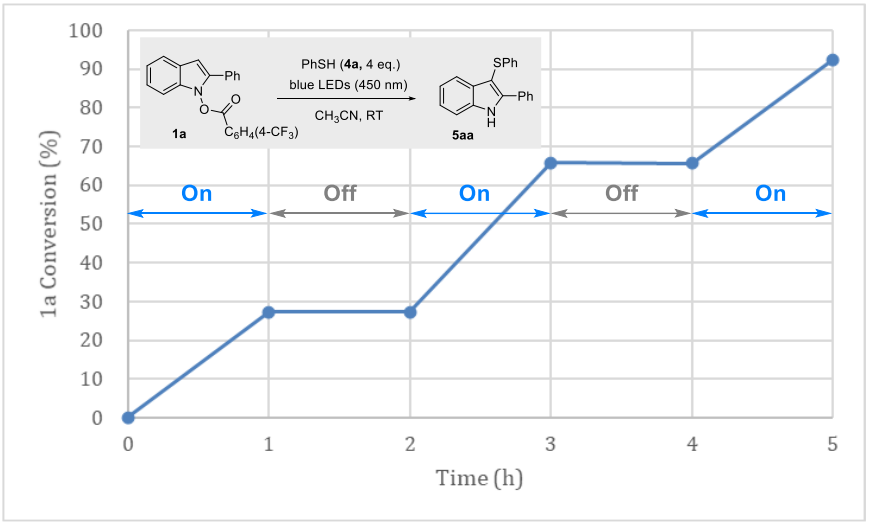


Fig. S5. Light on-off experiment using **1a** and **4a** in CH_3CN under irradiation with blue LEDs.

4.4. Measurement of UV-Vis Spectra

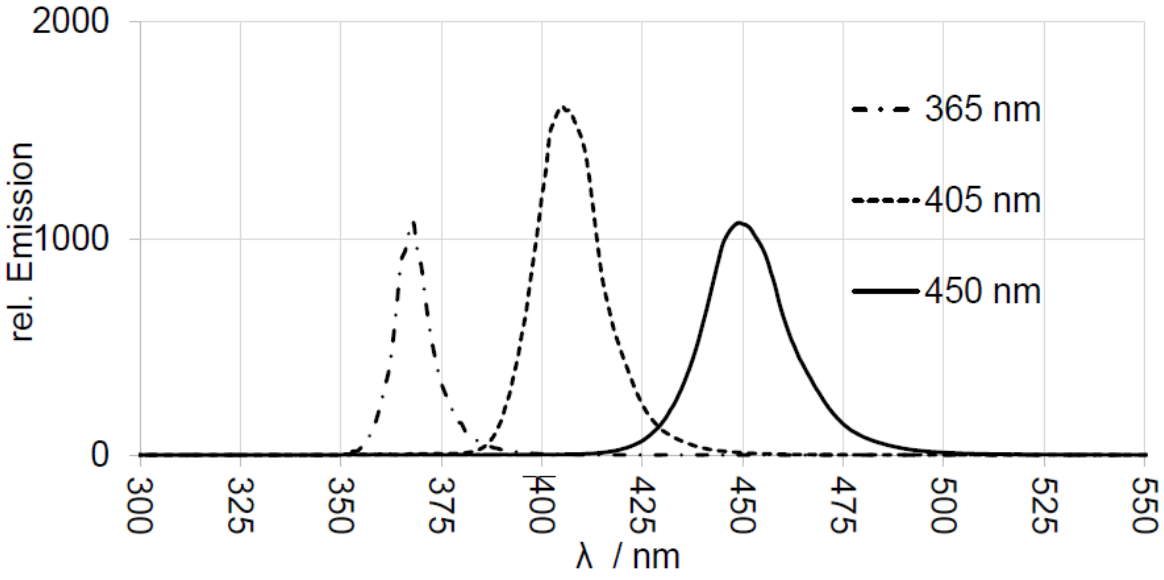


Fig. S6: Emission spectra of the EvoluChem™ LEDs used in the present study

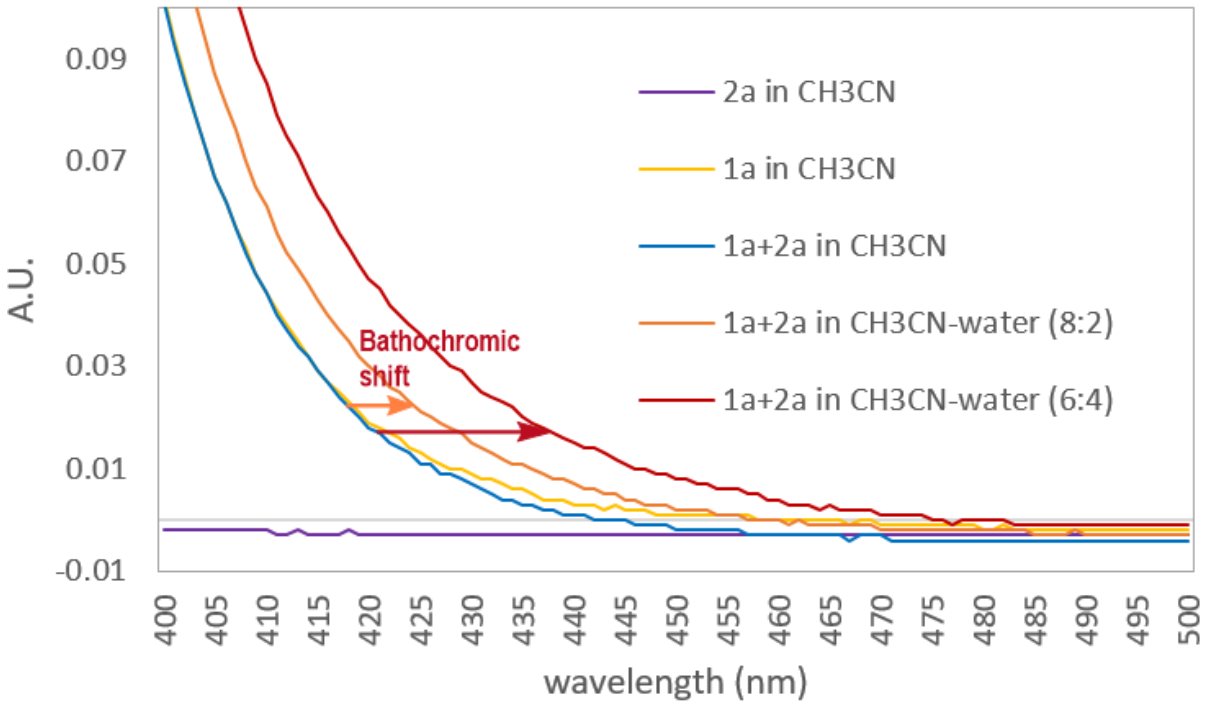


Fig. S7. Bathochromic shift of UV-Vis spectrum of **1a** and **2a** (*n*HexSH) in CH_3CN and H_2O.

(purple) **2a** (*n*HexSH, 0.05 mmol) in CH_3CN (1 mL)

(yellow) **1a** (0.0125 mmol) in CH_3CN (1 mL)

(blue) **1a** (0.0125 mmol) and **2a** (0.05 mmol) in CH_3CN (1 mL)

(orange) **1a** (0.0125 mmol) and **2a** (0.05 mmol) in CH_3CN-water (8:2, 1 mL)

(red) **1a** (0.0125 mmol) and **2a** (0.05 mmol) in CH_3CN-water (6:4, 1 mL)

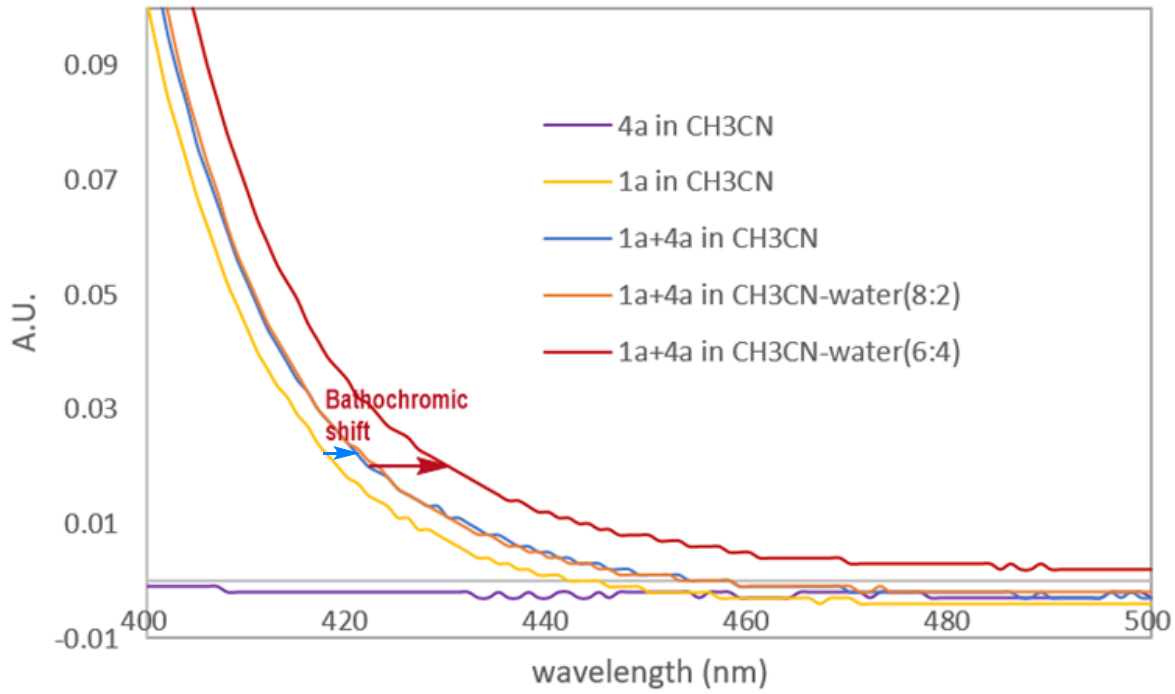


Fig. S8. Bathochromic shift of UV-Vis spectrum of **1a** and **4a** (PhSH) in CH_3CN and H_2O.

(purple) **4a** (PhSH, 0.05 mmol) in CH_3CN (1 mL)

(yellow) **1a** (0.0125 mmol) in CH_3CN (1 mL)

(blue) **1a** (0.0125 mmol) and **4a** (0.05 mmol) in CH_3CN (1 mL)

(orange) **1a** (0.0125 mmol) and **4a** (0.05 mmol) in CH_3CN-water (8:2, 1 mL)

(red) **1a** (0.0125 mmol) and **4a** (0.05 mmol) in CH_3CN-water (6:4, 1 mL)

It was noted that the mixture of **1a** and **2a** (or **1a** and **4a**) remained homogeneous in CH_3CN and water (6:4)

4.5. NMR Spectroscopic Analysis to Probe Distribution of Reactants

4.4.1. Distribution of **1a** and *n*HexSH (**2a**) in D_2O

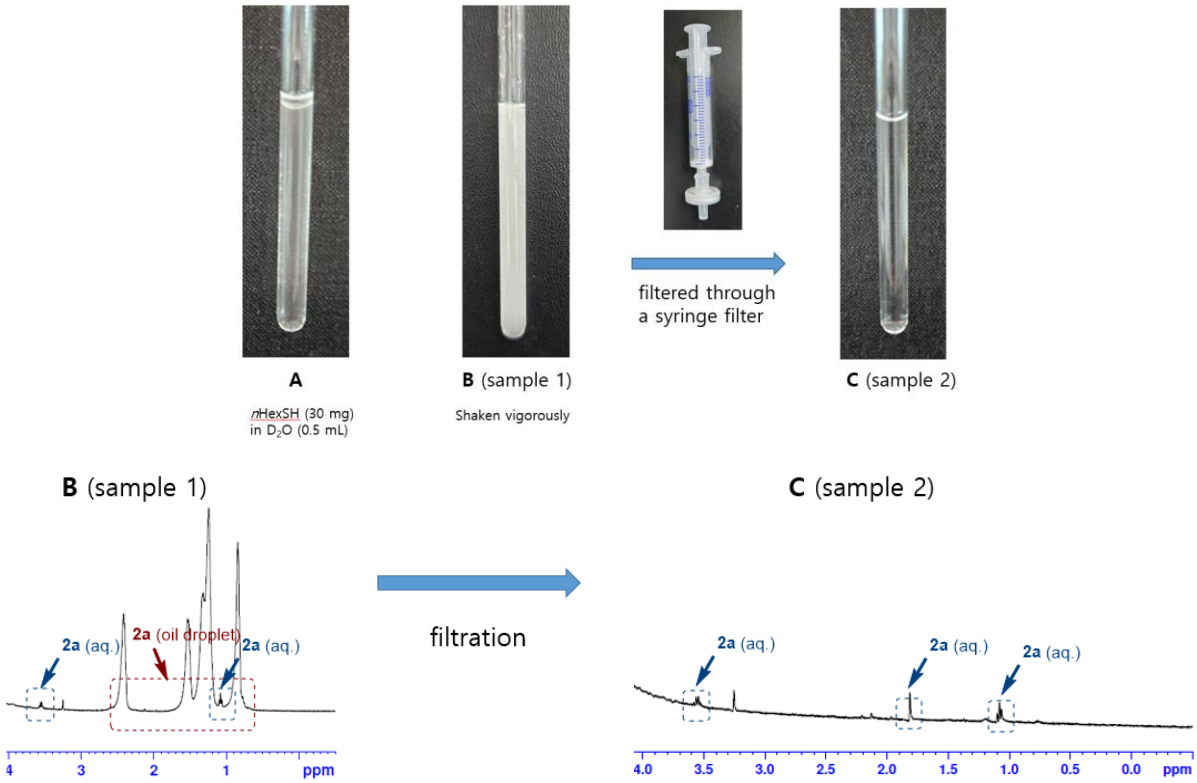


Fig. S9. ^{1}H NMR spectra of a mixture of *n*HexSH (**2a**) and D_2O.

A mixture of *n*HexSH (**2a**, 30 mg) in D_2O (0.5 mL) forms two layers (**A**). When shaken, it forms an emulsion (**B**). In the ^{1}H NMR spectrum of **B** (sample 1), we observed two distinct species (left spectrum): **2a** dissolved in D_2O, and **2a** in oil droplet which is separated from D_2O.

When this emulsion was filtered through a syringe filter (pore size: 0.45 um, 25 mm diameter, Whatman, cat no. 6750-2504), a clear solution was obtained (**C**). The ^{1}H NMR spectrum of **C** (sample 2) indicated that **2a** in the oil droplet was removed by filtration, and showed only **2a** dissolved in aqueous phase (**a**).

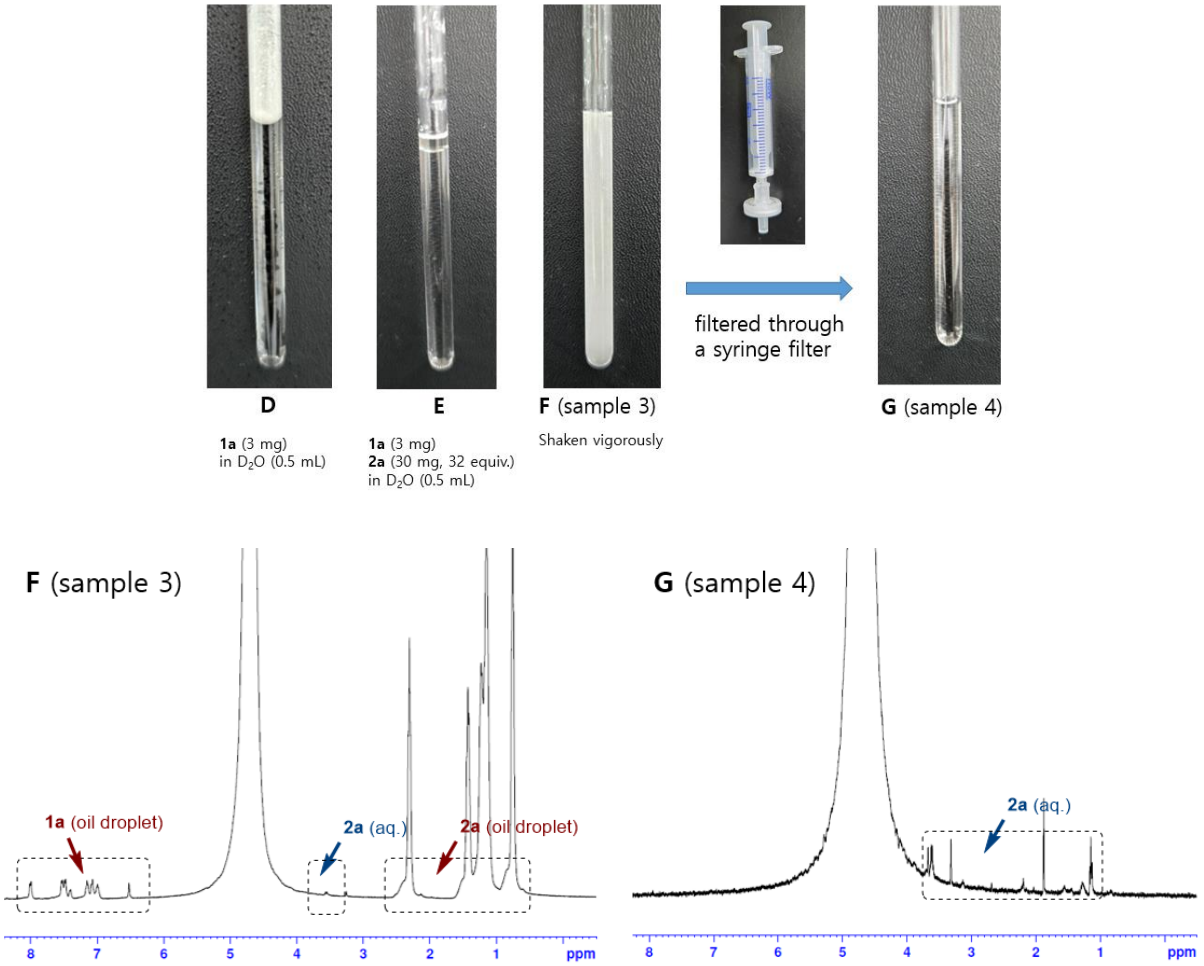


Fig. S10. ^{1}H NMR spectra of a mixture of **1a** and *n*HexSH (**2a**) in D_2O.

A mixture of **1a** (3.0 mg) in D_2O (0.5 mL) did not dissolve at all (^{1}H NMR spectrum of **D** showed no peaks except solvent peaks). To this was added **2a** (*n*HexSH, 30.0 mg, 32 equiv.) until it forms a eutectic mixture (**E**) in which all solid particles of **1a** disappeared and formed two layers. When shaken, it forms an emulsion (**F**). In the ^{1}H NMR spectrum of **F** (sample 3), we observed three distinct species (left spectrum): **1a** in oil droplet, **2a** dissolved in D_2O and **2a** in oil droplet which is separated from D_2O.

When this emulsion was filtered through a syringe filter (pore size: 0.45 um, 25 mm diameter, Whatman, cat no. 6750-2504), a clear solution was obtained (**G**). The ^{1}H NMR spectrum of **G**

(sample 4) showed only **2a** dissolved in aqueous phase. This suggested that peaks of **1a** existed in an oil droplet, and did not dissolve in the aqueous phase. On the other hand, the filtered materials remaining in the syringe filter and syringe was rinsed with $CDCl_3$ and its ^1H NMR spectra showed a clean mixture of **1a** and **2a**.

A COSY spectrum of **G** (sample 2) was taken to confirm the identity of **2a** dissolved in the aqueous phase (Fig. S11).

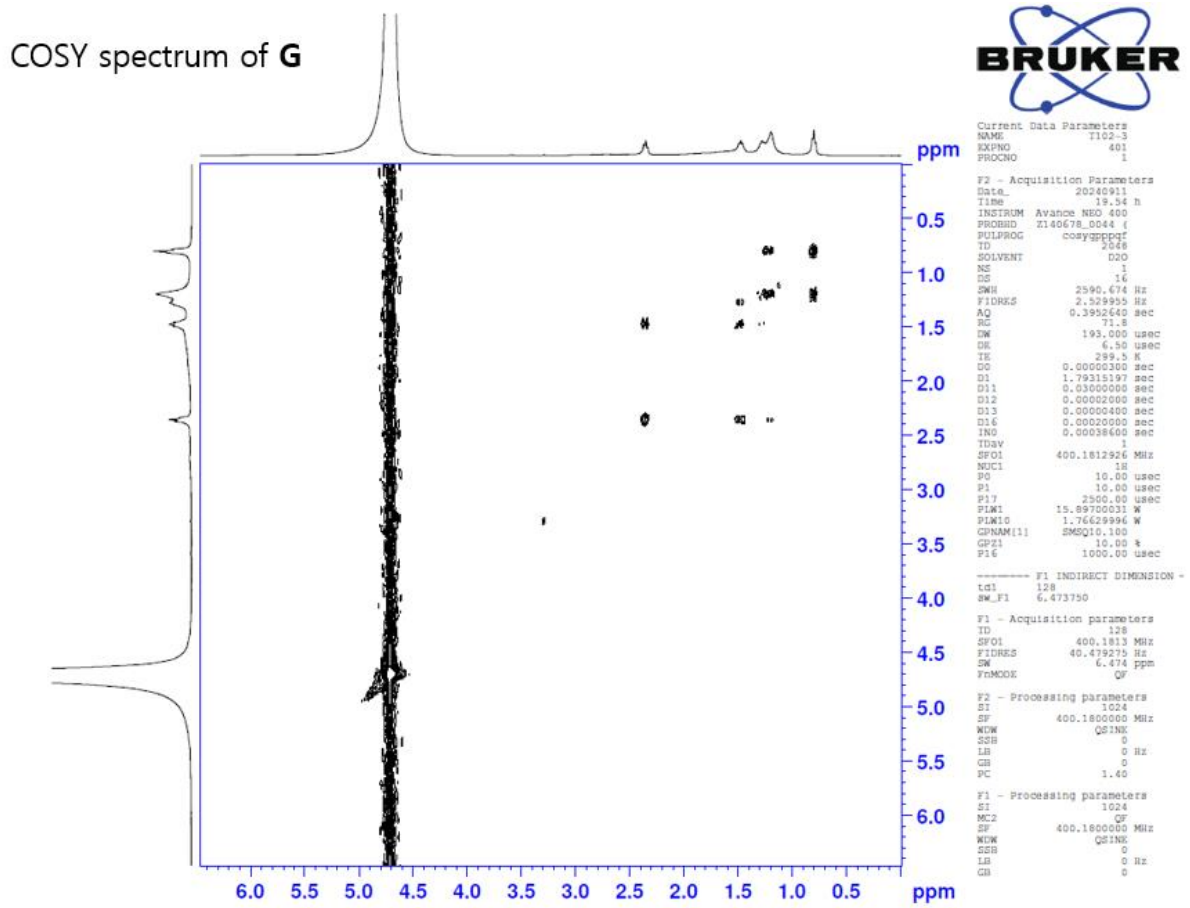
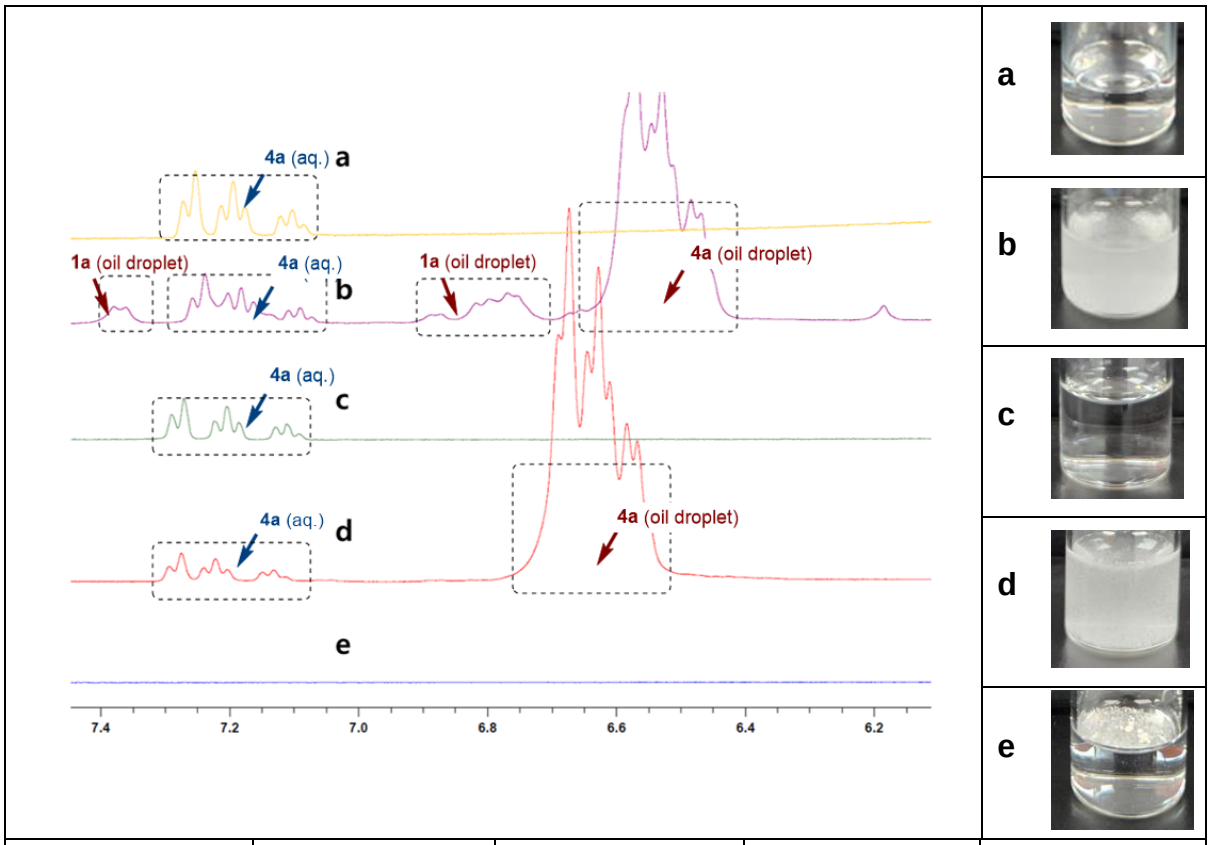


Fig. S11. COSY spectrum of **G** (sample a mixture of **1a** and *n*HexSH (**2a**) in D_2O

4.4.2. Distribution of **1a** and PhSH (**4a**) in D_2O



a	b	c	d	e
1a (1 mg) and PhSH (10 mg, ~30 eq.), filtered	**1a** (1 mg) and PhSH (10 mg, ~30 eq.), shaken	PhSH (10 mg) filtered	PhSH (10 mg) shaken	**1a** (1 mg) insoluble

Fig. S12. ^1H NMR spectra of a mixture of **1a** and PhSH (**4a**) in D_2O.

Distribution of **1a** and PhSH (**4a**) was probed similarly. Spectra of **c** and **d** showed distribution of PhSH in D_2O. The ^1H NMR spectrum (**d**) of a shaken sample of PhSH in D_2O showed two species: PhSH dissolved in aqueous and in oil drop. When filtered (**c**), PhSH in oil drop was removed and only PhSH (aq.) remained in D_2O phase.

Likewise, a shaken mixture of **1a** and PhSH (~30 equiv) in D_2O (**b**) showed PhSH dissolved in D_2O as well as **1a** and PhSH in oil droplet. When filtered (**a**), **1a** and PhSH in oil droplet were removed and only PhSH (aq.) remained in D_2O phase.

4.6. Determination of Quantum Yield (Φ)

According to the procedure of Yoon[9] the photon flux of the blue LED (λ_{max} = 450 nm) was determined by standard ferrioxalate actinometry. A 0.15 M solution of ferrioxalate was prepared by dissolving potassium ferrioxalate hydrate (0.737 g) in H_2SO_4 (10 mL of a 0.050 M solution). A buffered solution of 1,10-phenanthroline was prepared by dissolving 1,10-phenanthroline (25.0 mg) and sodium acetate (5.626 g) in 25 mL of 0.5 M H_2SO_4. Both solutions were stored in the dark. To determine the photon flux of the LED, 2.0 mL of ferrioxalate solution was placed in a cuvette and irradiated for 90 seconds at λ_{max} = 450 nm. After irradiation, 0.35 mL of phenanthroline solution was added to the cuvette and the mixture was allowed to stir in the dark for 1 h to allow the ferrous ions to completely coordinate to the phenanthroline. The absorbance of the solution was measured at 510 nm. A non-irradiated sample was also prepared and the absorbance at 510 nm was measured. Conversion was calculated using Eq. S1.

	Non-irradiated	Irradiated-1	Irradiated-2	Irradiated-3
A(510 nm)	0.0216	0.8669	0.7278	0.9322

Avarage *A*(510 nm) of irradiated sample: 0.8423

Mol of Fe^{2+} = $\frac{V*\Delta A(510\ nm)}{l*\varepsilon}$ = $\frac{0.00235\ (L)*\ 0.8207}{1\ cm*\ 11{,}100\frac{L}{mol.cm}}$ = 1.74 x 10^{-7} mol (S1)

V is the total volume (0.00235 L) of the solution after addition of phenanthroline, ΔA is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions, l is the path length (1.00 cm), and ε is the molar absorptivity of the ferrioxalate actinometer at 510 nm (11,100 $Lmol^{-1}cm^{-1}$). The photon flux can be calculated using Eq. S2.

Photon flux = $\frac{mol\ of\ Fe2+}{\Phi * t * f}$ = $\frac{1.74 * 10^{-7}}{0.9 * 90(s) * 0.98}$ = 2.19 x 10^{-9} einstein/s (S2)

Where Φ is the quantum yield for the ferrioxalate actinometer (0.9 at λ = 457.9 nm),[10] t is the irradiation time (90 s), and f is the fraction of light absorbed at 450 nm by the ferrioxalate actinometer. This value is calculated using Eq. S3 where A(450 nm) is the absorbance of the ferrioxalate solution at 450 nm. An absorption spectrum gave an A(450 nm) value of 1.702,

indicating that the fraction of absorbed light (f):

$$f = 1 - 10^{-A(450nm)} = 1 - 10^{-(1.702)} = 0.98 \qquad (S3)$$

Determination of the reaction quantum yield.

A cuvette was charged with **1a** (38.1 mg, 0.1 mmol), **4a** (0.4 mmol) and Celite (100 mg) in water (2 mL). The cuvette was then capped with a PTFE stopper. The cuvette was placed in a photoreactor with an identical distance from the light source to the above actinometry measurement. Three identical reaction mixtures were prepared and was stirred and irradiated (λ = 450 nm). The conversions at 0.2 h, 0.5 h, 1 h were analyzed by HPLC. After 720 sec, the yield of product was determined to be 85% (Fig. S13), which indicated that the initial yield after 90 sec is over 10.6 % (1.06 x 10^{-5} mol).

The quantum yield can be estimated using Eq. S5, where the fraction of light (f) absorbed by the substrates can be determined according to Eq. S4. In acetonitrile, the absorbance (A) of a mixture of **1a** (0.05 M) and **4a** (0.20 M) was A = 0.004, and f = 0.0092 was determined. From this, higher limit of the quantum yield (Φ) was determined to be 5800. The actual absorbance of the reaction mixture in water could not be determined accurately because of the heterogeneity and the scattering effect, but was expected to be much higher in CH_3CN (A > 0.004), due to the bathochromic shift (Figure S8). Therefore, fraction of light must be 0.004 < f < 1, and the estimated range of quantum yield Φ (54~5800) strongly supported the presence of radical chain mechanism.

$$f = 1 - 10^{-A(450nm)} = 1 - 10^{-(0.004)} = 0.0092 \qquad (S4)$$

$$\Phi = \frac{mol\ product}{flux * t * f} = \frac{1.06 * 10^{-5}}{2.19*10^{-9} * 90(s) * 0.0092} = 5800 \qquad (S5)$$

$$\Phi = \frac{mol\ product}{flux * t * f} = \frac{1.06 * 10^{-5}}{2.19*10^{-9} * 90(s) * 1} = 54 \qquad (S6)$$

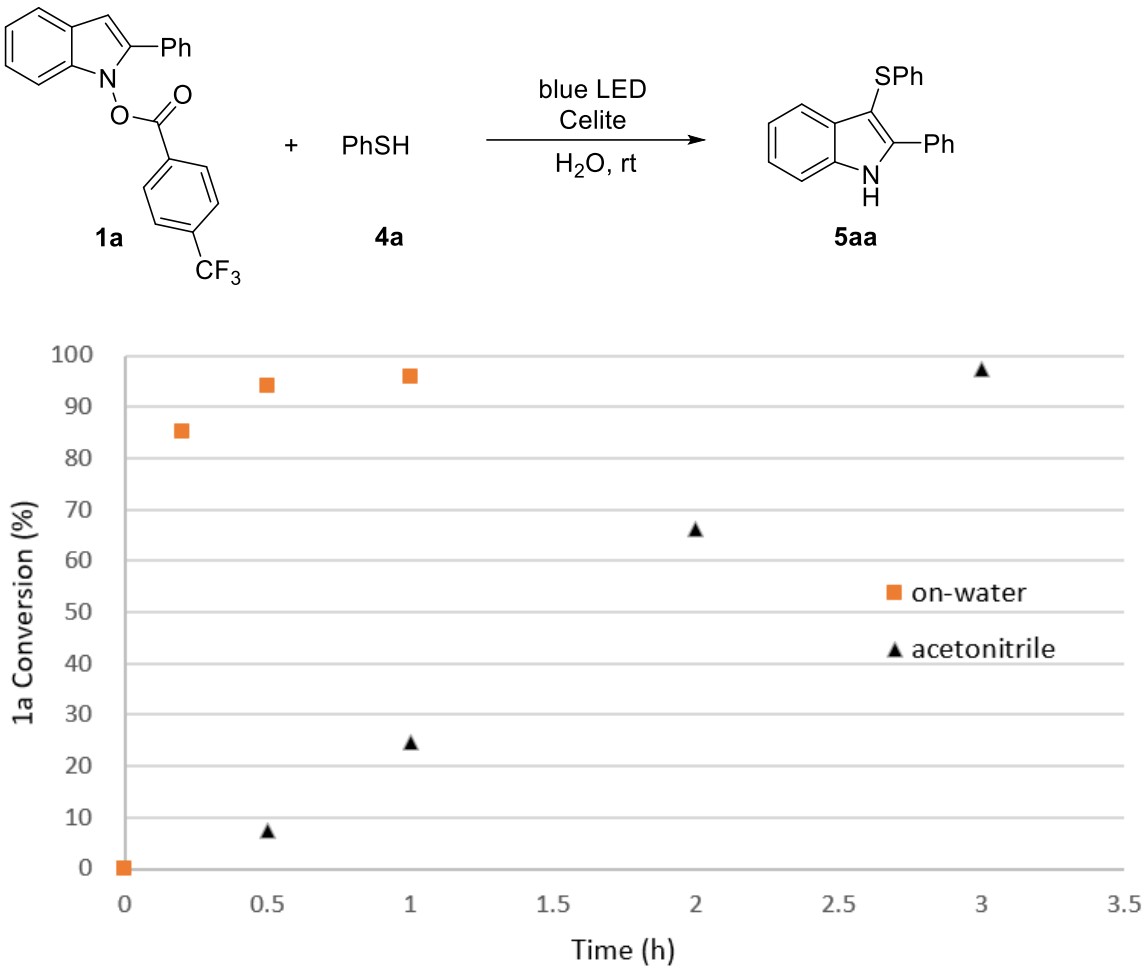
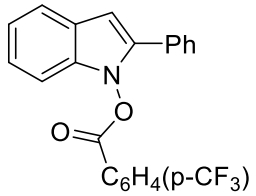
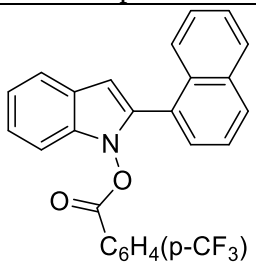
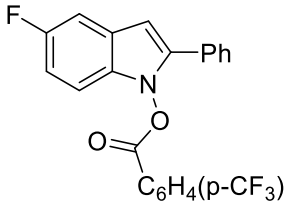
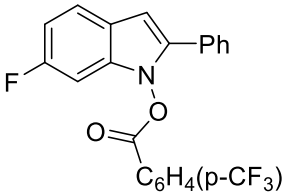
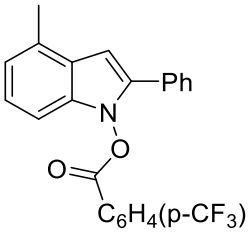
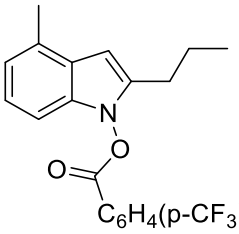
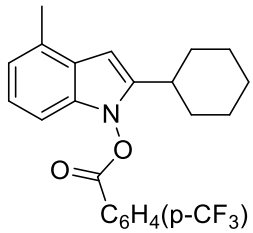
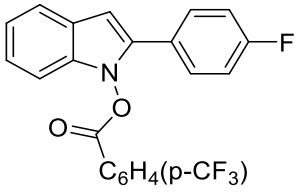


Fig. S13. Reaction of **1a** and **4a** on-water under standard conditions vs. reaction in acetonitrile.

5. Characterization of Substrates

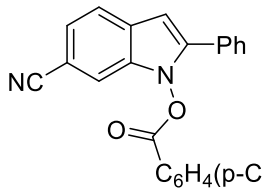
List of known compounds

Compound	Reference	Compound	reference
 **1a**	1	 **1c**	3
 **1d**	3	 **1e**	3
 **1f**	3	 **1i**	3
 **1j**	3		

 **1b**

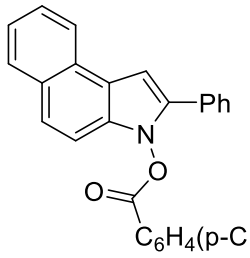
Yellow solid (EtOAc:Hex = 1:30, Rf = 0.20), mp 118 - 121 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ

8.20 (d, *J* = 8.1 Hz, 2H), 7.76 (d, *J* = 8.2 Hz, 2H), 7.66 – 7.55 (m, 3H), 7.30 – 7.15 (m, 3H), 7.07 (t, *J* = 8.6 Hz, 2H), 6.67 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.7, 162.8 (d, J_{CF} = 247.1 Hz), 140.2, 137.8, 135.9 (q, J_{CF} = 32.7 Hz), 130.6, 129.6, 129.5, 129.4 (d, J_{CF} = 8.3 Hz), 126.4, (d, J_{CF} = 3.2 Hz) 126.0 (q, J_{CF} = 3.7 Hz), 125.6, 123.8, 123.3 (q, J_{CF} = 271.4 Hz), 122.5, 121.3, 116.0 (d, J_{CF} = 21.7 Hz), 109.7, 101.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.37, -112.74; IR (ATR): ṽ = 1777, 1500, 1449, 1412, 1320, 1229, 1174, 1159, 1133, 1067, 1041, 1004, 860, 841, 794, 766, 740, 698 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{14}F_3NO_2^+$ $[M+1]^+$: 400.0955, found 400.0954.



1g

Yellow solid (EtOAc:Hex = 1:10, Rf = 0.35), mp 154 - 158 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (d, *J* = 8.1 Hz, 2H), 7.83 (d, *J* = 8.2 Hz, 2H), 7.74 (d, *J* = 8.2 Hz, 1H), 7.69 (d, *J* = 7.1 Hz, 2H), 7.61 (s, 1H), 7.52 – 7.35 (m, 4H), 6.81 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4, 143.6, 136.3 (q, J_{CF} = 32.9 Hz), 135.6, 130.7, 129.4, 129.0, 128.9, 128.3, 127.9, 126.2 (q, J_{CF} = 3.7 Hz), 125.0, 123.2 (d, J_{CF} = 271 Hz), 121.9, 119.9, 113.7, 106.0, 100.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.42; IR (ATR): ṽ = 2224, 1776, 1617, 1486, 1456, 1411, 1322, 1229, 1165, 1126, 1112, 1065, 1042, 1028, 998, 858, 839, 822, 763, 694cm^{-1}; HRMS (ESI) Calcd for $C_{23}H_{14}F_3N_2O_2^+$ $[M+1]^+$: 407.1002, found 407.1000.

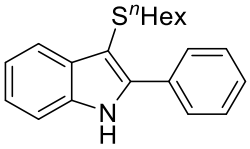


1h

white solid (EtOAc:Hex = 1:10, Rf = 0.50), mp 172 - 176 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (d, *J* = 8.1 Hz, 3H), 7.95 (d, J = 8.1 Hz, 1H), 7.83 (d, *J* = 8.1 Hz, 2H), 7.74 (d, *J* = 7.6 Hz, 2H), 7.70 (d, *J* = 8.8 Hz, 1H), 7.64 (t, *J* = 7.5 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.44 (t, *J* = 7.0 Hz, 3H), 7.34 (t, *J* = 7.4 Hz, 1H) 7.29 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.6, 137.6, 136.0 (q, J_{CF} = 32.7 Hz), 132.8, 130.7, 130.2, 130.1, 129.5, 128.9, 128.8, 128.1, 127.7, 127.6,

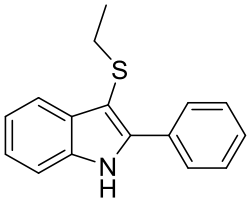
126.5, 126.1 (q, J_{CF} = 3.7 Hz), 124.6, 124.2, 123.3 (d, J_{CF} = 271.1 Hz), 122.8, 122.0, 120.0, 109.9, 99.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.35; IR (ATR): ṽ = 1777, 1718, 1653, 1603, 1559, 1507, 1457, 1410, 1319, 1226, 1166, 1129, 1111, 1047, 1028, 1006, 852, 802, 758, 759, 724, 694 cm^{-1}; HRMS (ESI) Calcd for $C_{26}H_{17}F_3NO_2^+$ $[M+1]^+$: 432.1206, found 432.1203

6. Characterization of Products



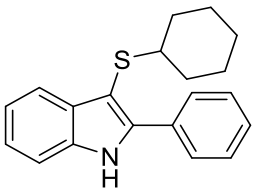
3aa

Yellow oil (25.1 mg, 81 % yield), (EtOAc:Hex = 1:10 Rf = 0.30); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (s, 1H), 7.89 (d, *J* = 7.1 Hz, 2H), 7.82 (d, *J* = 7.1 Hz, 1H), 7.49 (t, *J* = 7.5 Hz, 2H), 7.43-7.38 (m, 2H), 7.27 (td, *J* = 7.1, 1.5 Hz, 1H), 7.22 (td, *J* = 7.1, 1.2 Hz, 1H), 2.67 (t, *J* = 7.2 Hz, 2H), 1.46 -1.36 (m, 2H), 1.31-1.03 (m, 6H), 0.81 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.6, 135.6, 132.2, 131.6, 128.6, 128.3, 128.2, 122.4, 120.6, 119.9, 111.0, 103.6, 36.5, 31.4, 29.5, 28.2, 22.5, 14.0; IR (ATR): ṽ = 3399, 2954, 2926, 2855, 1741, 1603, 1482, 1455, 1323, 1298, 1225, 1007, 1032, 927, 741, 692 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{24}NS^+$ $[M+1]^+$: 310.1624, found 310.1618.



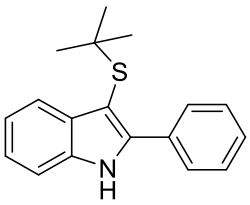
3ab

Yellow oil (21.3 mg, 84 % yield), (EtOAc:Hex = 1:10 Rf = 0.30); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (s, 1H), 7.91 (d, *J* = 8.0 Hz, 2H), 7.85 (d, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.47 – 7.37 (m, 2H), 7.33 – 7.19 (m, 2H), 2.73 (q, *J* = 7.4 Hz, 2H), 1.13 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.6, 135.6, 132.1, 131.7, 128.6, 128.3, 123.0, 120.7, 119.9, 111.0, 103.2, 30.4, 15.0; IR (ATR): ṽ = 3414, 2961, 2923, 1603, 1481, 1453, 1444, 1400, 1352, 1323, 1225, 1071, 1032, 1006, 968, 764, 694 cm^{-1}; HRMS (ESI) Calcd for $C_{16}H_{16}NS^+$ $[M+1]^+$: 254.0998, found 254.0999.



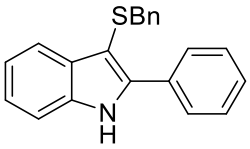
3ac

Brown oil (23.3 mg, 76 % yield), (EtOAc:Hex = 1:10, Rf = 0.30); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (s, 1H), 7.91 (d, *J* = 7.1 Hz, 2H), 7.83 (d, *J* = 7.2 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 2H), 7.42-7.36 (m, 2H), 7.25 (td, *J* = 7.1, 1.5 Hz, 1H), 7.21 (td, *J* = 7.1, 1.2 Hz, 1H), 2.89-2.79 (m, 1H), 1.88 – 1.74 (m, 2H), 1.69-1.58 (m, 2H), 1.52-1.45 (s, 1H), 1.33-1.17 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.0, 135.5, 132.4, 132.3, 128.6, 128.4, 128.2, 122.9, 120.6, 120.2, 110.9, 102.7, 48.2, 33.6, 26.1, 25.7; IR (ATR): ṽ = 3403, 3057, 2926, 2851, 1603, 1481, 1444, 1399, 1352, 1323, 1262, 1298, 1262, 1225, 1032, 996, 742, 693 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{22}NS^{+}$ $[M+1]^{+}$: 308.1467, found 308.1469.



3ad

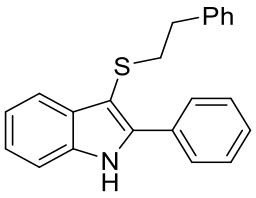
Yellow oil (14.6 mg, 52 % yield), (EtOAc:Hex = 1:6, Rf = 0.30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.39 (s, 1H), 7.92 (d, *J* = 7.1 Hz, 2H), 7.85 (d, *J* = 6.9 Hz, 1H), 7.46 (t, J = 7.5 Hz, 2H), 7.41-7.34 (m, 2H), 7.24 (td, *J* = 7.1, 1.6 Hz, 1H), 7.22 (td, *J* = 7.1, 1.3 Hz, 1H), 1.10 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.4, 135.5, 133.3, 132.8, 128.8, 128.4, 128.2, 122.8, 120.8, 120.7, 110.7, 102.4, 48.5, 31.1; IR (ATR): ṽ = 3405, 2958, 2922, 2856, 1753, 1661, 1604, 1525, 1455, 1445, 1399, 1363, 1323, 1296, 1225, 1164, 1034, 1008, 921, 768, 743, 694 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{20}NS^{+}$ $[M+1]^{+}$: 282.1311, found 282.1306.



3ae (ref. 4)

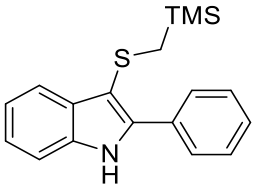
Yellow oil (18.2 mg, 58% yield), (EtOAc:Hex = 1:4, Rf = 0.30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.28 (s, 1H), 7.75 (d, *J* = 7.7 Hz, 1H), 7.54 (dd, *J* = 6.5, 1.7 Hz, 2H), 7.41-7.30 (m, 4H), 7.25 (td, *J* = 7.1, 1.3 Hz, 1H), 7.29 (td, *J* = 7.8, 1.3 Hz, 1H), 7.11-7.04 (m, 3H), 6.98-6.93 (m, 2H),

3.83 (s, 2H).



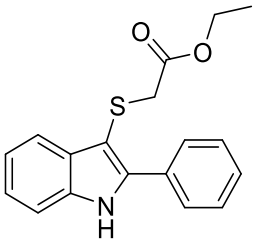
3af

Yellow oil (23.0 mg, 70 % yield), (EtOAc:Hex = 1:6, Rf = 0.20); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.35 (s, 1H), 7.85 (d, *J* = 7.7 Hz, 2H), 7.81 (d, *J* = 7.5 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.43-7.37 (m, 2H), 7.30-7.10 (m, 5H), 6.96 (d, *J* = 6.9 Hz, 2H), 2.93 (t, *J* = 7.8 Hz, 2H), 2.70 (t, *J* = 7.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.7, 140.6, 135.7, 132.0, 131.5, 128.7, 128.5, 128.4, 128.39, 128.38, 126.1, 123.1, 120.8, 119.9, 111.1, 103.1, 37.5, 36.2; IR (ATR): ṽ = 3407, 3060, 2919, 1738, 1603, 1581, 1527, 1496, 1454, 1400, 1352, 1324, 1298, 1225, 1134, 1076, 1007, 927, 744, 695 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{20}NS^{+}$ $[M+1]^{+}$: 330.1311, found 330.1307.



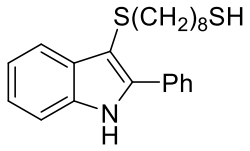
3ag

Brown oil (17.1 mg, 55 % yield), (EtOAc:Hex = 1:4, Rf = 0.30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.28 (s, 1H), 7.89 (d, *J* = 7.1Hz, 2H), 7.81 (d, *J* = 6.9 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.42-7.36 (m, 2H), 7.25 (td, *J* = 7.0, 1.3 Hz, 1H), 7.21 (td, *J* = 7.1, 0.7 Hz, 1H), 2.07 (s, 2H), 0.06 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.0, 137.5, 134.0, 132.9, 130.5, 130.0, 129.9, 124.8, 122.4, 121.6, 112.9, 108.8, 25.0, 0.0; IR (ATR): ṽ = 3409, 2955, 1603, 1487, 1455, 1400, 1327, 1301, 1248, 1225, 1036, 1007, 844, 742, 694 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{22}NSSi^{+}$ $[M+1]^{+}$: 312.1237, found 312.1232 .



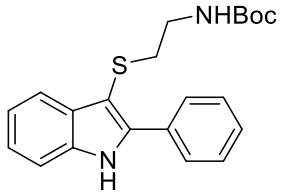
3ah

Yellow oil (13.1 mg, 42 % yield), (EtOAc:Hex = 1:6, Rf = 0.28); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (s, 1H), 7.90 (d, *J* = 7.7 Hz, 2H), 7.83 (d, *J* = 7.0 Hz, 1H), 7.52 (t, *J* = 7.3 Hz, 2H), 7.44 (t, *J* = 7.9 Hz, 2H), 7.33 – 7.20 (m, 2H), 3.84 (q, *J* = 7.1 Hz, 2H), 3.37 (s, 2H), 1.02 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.1, 141.4, 135.6, 131.7, 131.1, 128.7, 128.5, 128.3, 123.2, 120.9, 119.5, 111.2, 101.5 ,61.1, 38.2, 13.8; IR (ATR): ṽ = 3350, 2981, 1709, 1604, 1479, 1445, 1405, 1367, 1325, 1286, 1227, 1126, 1030, 919, 769, 744, 694 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{17}NNaO_2S^+$ $[M+Na]^+$: 334.0872, found 334.0869.



3ai

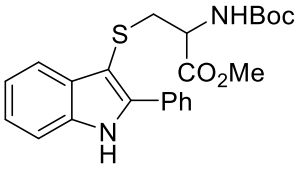
Yellow oil (28.8 mg, 78 % yield), (EtOAc:Hex = 1:3, Rf = 0.45); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (s, 1H), 7.91 (d, *J* = 7.5 Hz, 2H), 7.84 (d, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.43 (t, *J* = 6.8 Hz, 2H), 7.33 – 7.17 (m, 2H), 2.69 (t, *J* = 7.1 Hz, 2H), 2.51 (q, *J* = 7.4 Hz, 2H), 1.63 – 1.50 (m, 2H), 1.49 – 1.21 (m, 7H), 1.19 – 1.02 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.6, 135.6, 132.1, 131.6, 128.6, 128.3, 128.3, 123.0, 120.7, 119.8, 111.0, 103.5, 36.4, 34.0, 29.5, 29.0, 28.9, 28.4, 28.3, 24.6; IR (ATR): ṽ = 3405, 3054, 2923, 2851, 1601, 1481, 1453, 1444, 1399, 1322, 1295, 1224, 1072, 1032, 1006, 803, 693 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{27}NNaS_2^+$ $[M+Na]^+$: 392.1477, found 392.1475.



3aj

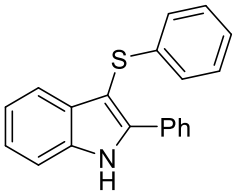
Brown oil (32.8 mg, 89 % yield), (EtOAc:Hex = 1:3, Rf = 0.42); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.66 (s, 1H), 7.82 (d, *J* = 7.2 Hz, 2H), 7.77 (d, *J* = 6.8 Hz, 1H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.42-7.35 (m, 2H), 7.25 (td, *J* = 7.1, 1.5 Hz, 1H), 7.22 (td, *J* = 7.1, 1.4 Hz, 1H), 4.56 (br s, 1H), 2.98 (q, *J* = 6.0 Hz, 2H), 2.69 (t, *J* = 6.1 Hz, 2H), 1.39 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.7, 141.5, 135.7, 131.7, 131.2, 128.8, 128.6, 128.5, 123.2, 121.0, 119.5, 111.3, 101.4, 79.1, 39.2, 36.0, 28.4; IR (ATR): ṽ = 3294, 2977, 2928, 1487, 1506, 1446, 1392, 1366, 1326, 1250, 1225, 1164, 1033, 1007, 952, 862, 742, 695 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{23}N_2O_2S^-$ [M-

1]$^{-}$: 367.1486, found 367.1484.



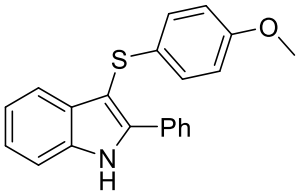
3ak

Yellow oil (33.2 mg, 78 % yield) (EtOAc:Hex = 1:3, Rf = 0.30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.63 (s, 1H), 7.84 (d, *J* = 7.3 Hz, 2H), 7.82-7.76 (m, 1H), 7.51 (t, *J* = 7.4 Hz, 2H), 7.47 - 7.35 (m, 2H), 7.28-7.22 (m, 2H), 5.04 (d, *J* = 7.9 Hz, 1H), 4.38 - 4.23 (m, 1H), 3.32 (dd, *J* = 13.7, 4.4 Hz, 1H), 3.20 (s, 3H), 3.03 (dd, *J* = 13.7, 5.0 Hz, 1H), 1.36 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.9, 155.1, 141.5, 135.7, 131.5, 130.8, 130.5, 128.7, 128.64, 128.58, 123.1, 120.9, 120.0, 111.3, 100.8, 79.7, 52.9, 52.8, 37.3, 28.2; IR (ATR): ṽ = 3333, 2979, 1589, 1499, 1447, 1396, 1367, 1324, 1291, 1251, 1226, 1165, 1062, 1019, 911 , 863, 745, 695 cm^{-1}; HRMS (ESI) Calcd for $C_{23}H_{27}N_2O_4S^+$ $[M+1]^+$: 427.1686, found 427.1680.



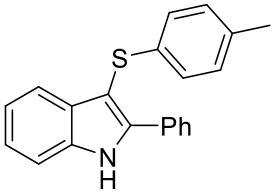
5aa (ref. 4)

Yellow oil; (28.6 mg, 95% yield) (EtOAc:Hex = 1:10, Rf = 0.27); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.47 (s, 1H), 7.72 (d, *J* = 7.1 Hz, 2H), 7.62 (d, *J* = 7.9 Hz, 1H), 7.44-7.32 (m, 4H), 7.25 (t, *J* = 7.6 Hz, 1H), 7.19 - 7.12 (m, 3H), 7.18 – 7.06 (m, *J* = 7.5, 0.7 Hz, 2H), 7.03 (t, *J* = 7.1 Hz, 1H).



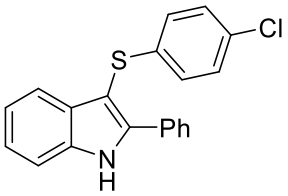
5ab (ref. 6)

Yellow solid (32.1 mg, 97% yield) (EtOAc:Hex = 1:10 Rf = 0.22), mp 156 - 188 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.48 (s, 1H), 7.78 (d, *J* = 7.0 Hz, 2H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.48 - 7.35 (m, 4H), 7.26 (td, *J* = 7.1, 1.2 Hz, 1H), 7.16 (td, *J* = 8.0, 0.9 Hz, 1H), 7.06 (dt, *J* = 8.8, 2.6 Hz, 2H), 6.72 (dt, *J* = 8.8, 2.6 Hz, 2H), 3.70 (s, 3H).



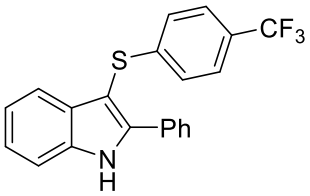
5ac (ref. 4)

Yellow oil; (31.2 mg, 99% yield) (EtOAc:Hex = 1:6, Rf = 0.27); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.48 (s, 1H), 7.75 (d, *J* = 7.1 Hz, 2H), 7.63 (d, *J* = 7.9 Hz, 1H), 7.47 - 7.34 (m, 4H), 7.26 (td, *J* = 7.3, 1.2 Hz, 1H), 7.15 (td, *J* = 7.5, 0.7 Hz, 1H), 7.03 - 6.94 (m, 4H), 2.24 (s, 3H).



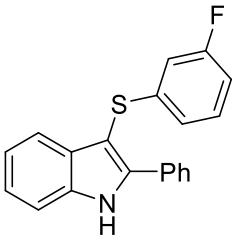
5ad (ref. 4)

Yellow oil (30.5 mg, 91% yield) (EtOAc:Hex = 1:6, Rf = 0.34); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.52 (s, 1H), 7.71 (d, *J* = 6.9 Hz, 2H), 7.59 (d, *J* = 7.9 Hz, 1H), 7.48 - 7.35 (m, 4H), 7.27 (td, *J* = 7.2 Hz, 1.1 1H), 7.18 (td, *J* = 7.5, 0.9 Hz 1H), 7.11 (dt, *J* = 8.7, 2.3 Hz, 2H), 7.00 (dt, J = 8.7, 2.3 Hz, 2H).



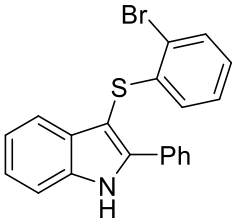
5ae (ref. 6)

Yellow oil (34.3 mg, 93% yield) (EtOAc:Hex = 1:6, Rf = 0.21); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.56 (s, 1H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.57 (d, *J* = 7.7 Hz, 1H), 7.49 - 7.34 (m, 6H), 7.29 (t, *J* = 7.6, 1.0 Hz, 1H), 7.18 (t, *J* = 7.9 Hz, 1H), 7.14 (d, *J* = 8.3 Hz, 2H).



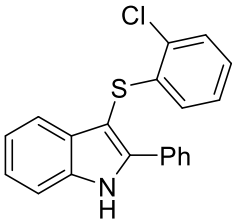
5af (ref. 7)

Yellow oil (27.1 mg, 85% yield), (EtOAc:Hex = 1:10, Rf = 0.25); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.55 (s, 1H), 7.73 (d, *J* = 6.9 Hz, 2H), 7.61 (d, *J* = 7.6 Hz, 1H), 7.49 - 7.36 (m, 4H), 7.28 (td, *J* = 7.6, 1.2 Hz 1H), 7.19 (t, *J* = 7.5 Hz, 1.0 1H), 7.12 (dd, *J* = 13.8, 6.0 Hz, 1H), 6.90 (d, *J* = 7.9 Hz, 1H), 6.77-6.69 (m, 2H).



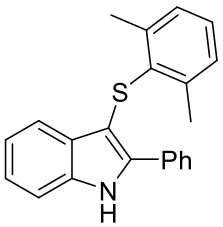
5ag (ref. 4)

Yellow oil(36.8 mg, 97% yield), (EtOAc:Hex = 1:10, Rf = 0.28); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.73 (s, 1H), 7.72 (d, *J* = 6.8 Hz, 2H), 7.61 (d, *J* = 7.9 Hz, 1H), 7.52 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.49 - 7.36 (m, 4H), 7.30 (dd, *J* = 8.2, 7.1 Hz, 1H), 7.19 (dd, *J* = 8.0, 7.2 Hz, 1H), 6.98 (td, *J* = 7.6, 1.4 Hz, 1H), 6.92 (td, *J* = 7.7, 1.7 Hz, 1H), 6.69 (dd, *J* = 7.9, 1.7 Hz, 1H).



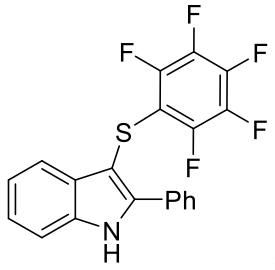
5ah (ref. 4)

Brown oil (29.8 mg, 89% yield) (EtOAc:Hex = 1:10, Rf = 0.27); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.60 (s, 1H), 7.71 (d, *J* = 8.3 Hz, 2H), 7.59 (d, *J* = 7.6 Hz, 1H), 7.48 - 7.35 (m, 4H), 7.32 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.28 (dd, *J* = 8.2, 7.1 Hz, 1H), 7.17 (dd, *J* = 8.0, 7.2 Hz, 1H), 6.98 (td, *J* = 7.4, 1.7 Hz, 1H), 6.92 (td, *J* = 7.9, 1.6 Hz, 1H), 6.68 (d, *J* = 7.8, 1.5 Hz, 1H).



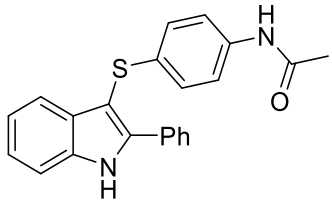
5ai (ref. 6)

Brown oil (28.6 mg, 87% yield) (EtOAc:Hex = 1:6, Rf = 0.43); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (s, 1H), 7.75 (d, *J* = 7.1 Hz, 2H), 7.49 (t, *J* = 7.5 Hz, 2H), 7.43 – 4.37 (m, 1H), 7.33 (d, *J* = 8.1 Hz, 1H), 7.17 - 7.09(m, 2H), 7.01 (dd, *J* = 8.7, 5.9 Hz, 1H), 6.99 - 6.93(m, 3H), 2.31 (s, 6H).



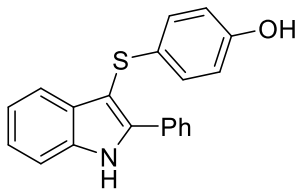
5aj

Yellow oil (35.6 mg, 91 % yield), (EtOAc:Hex = 1:6, Rf =0.31); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (s, 1H), 7.81 (d, *J* = 7.0 Hz, 2H), 7.72 (d, *J* = 7.6 H, 1H), 7.56 - 7.43 (m, 3H), 7.39 (d, *J* = 7.2 Hz, 1H), 7.26 (td, *J* = 7.1, 1.4 Hz, 1H), 7.22 (td, *J* = 7.2, 1.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.5, 135.4, 131.3, 130.6, 129.0, 128.7, 128.6, 123.4, 121.4, 119.3, 111.3; 98.6 and others (the ^{13}C NMR spectrum show complex splitting patterns due to multiple C–F couplings, complicating the identification of individual coupling constants). ^{19}F NMR (376 MHz, $CDCl_3$) δ -133.24 (dd, *J* = 23.8, 7.1 Hz), -154.08 (t, *J* = 21.2 Hz), -161.08 – -161.65 (m); IR (ATR): ṽ = 3402, 1640, 1513, 1484, 1449, 1393, 1326, 1297, 1227, 1089, 980, 909, 852, 747, 697 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{11}F_5NS^+$ $[M+1]^+$: 392.0527, found 392.0517.



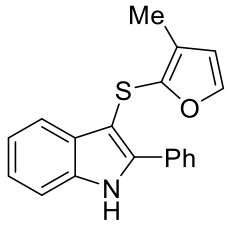
5ak

Brown soild (25.4 mg, 71 % yield), (EtOAc:Hex = 1:6 Rf = 0.12,; mp 72 - 76 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.86 (s, 1H), 7.73 (d, *J* = 7.0 Hz, 2H), 7.60 (d, *J* = 7.9 Hz, 1H), 7.46-7.32 (m, 4H), 7.30-7.21 (m, 4H), 7.15 (t, *J* = 7.4 Hz, 1H), 7.02 (d, *J* = 8.6 Hz, 2H), 2.09 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.3, 142.0, 135.9, 134.87, 134.79, 131.4, 131.1, 128.77, 128.70, 128.2, 126.4, 123.3, 121.1, 120.8, 119.9, 112.2, 99.5, 24.5; IR (ATR): ṽ = 3285, 3929, 1665, 1590, 1516, 1492, 1446, 1400, 1371, 1311, 1259, 1230, 1181, 1086, 1011, 909, 821, 736, 697 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{18}N_2NaOS^+$ $[M+Na]^+$: 381.1032, found 381.1027.



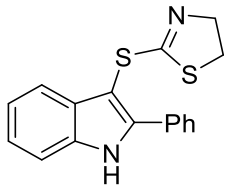
5al

Brown solid (29.8 mg, 94 % yield), (EtOAc:Hex = 1:6 Rf = 0.15), mp 170 - 172 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49 (s, 1H), 7.77 (d, *J* = 7.0 Hz, 2H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.49-7.35 (m, 4H), 7.26 (td, *J* = 7.1, 1.2 Hz, 1H), 7.17 (td, *J* = 8.0, 0.9 Hz, 1H), 7.02 (dt, *J* = 8.8, 2.6 Hz, 2H), 6.65 (dt, *J* = 8.8, 2.6 Hz, 2H), 4.55 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.4, 141.6, 135.8, 131.5, 131.2, 130.0, 128.78, 128.66, 128.2, 128.0, 123.3, 121.1, 120.0, 116.0, 111.1, 100.9; IR (ATR): ṽ = 3405, 3060, 2925, 2855, 1597, 1491, 1448, 1400, 1326, 1228, 1169, 1098, 1012, 909, 823, 748, 697 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{15}NNaOS^{+}$ $[M+Na]^{+}$: 340.0767, found 340.0765.



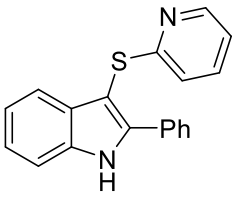
5am

Pale yellow oil (21.7 mg, 71 % yield), (EtOAc:Hex = 1:6 Rf = 0.24); ^{1}H NMR (400 MHz, $CDCl_3$): 8.33 (s, 1H), 7.83 (d, *J* = 7.1 Hz, 2H), 7.78 (d, *J* = 7.7 Hz, 1H), 7.50 (t, *J* = 7.2 Hz, 2H), 7.45 - 7.36 (m, 2H), 7.26 - 7.17 (m, 2H), 7.07 (d, *J* = 1.9 Hz, 1H), 6.06 (d, *J* = 1.7 Hz, 1H), 2.25 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.6, 140.5, 140.2, 135.5, 131.9, 131.2, 128.7, 128.6, 128.5, 123.1, 120.9, 119.8, 113.9, 113.1, 111.0, 103.3, 11.9; IR (ATR): ṽ = 3409, 1592, 1516, 1444, 1400, 1323, 1224, 1127, 1086, 934, 887, 740, 695 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{16}NOS^{+}$ $[M+1]^{+}$: 306.0947, found 306.0952.



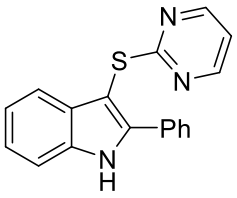
5an

Yellow solid (14.3 mg, 46 % yield), (EtOAc:Hex = 1:3, Rf = 0.30), mp 190 - 194 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.76 (s, 1H), 7.80-7.74 (m, 3H), 7.52-7.37 (m, 4H), 7.31-7.22 (m, 2H), 4.28 (t, *J* = 8.2 Hz, 2H), 3.20 (t, *J* = 8.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.2, 143.6, 135.4, 131.3, 131.1, 128.9, 128.8, 128.5, 123.5, 121.5, 119.7, 111.3, 98.1, 66.1, 34.5; IR (ATR): ṽ = 3061, 2926, 1563, 1480, 1446, 1409, 1327, 1302, 1264, 1228, 997, 927, 769, 743, 695 cm^{-1}; HRMS (ESI) Calcd for $C_{17}H_{15}N_2S_2^{+}$ $[M+1]^{+}$: 311.0671, found 311.0664.



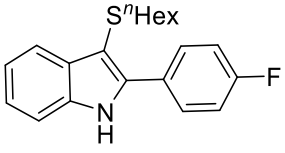
5ao (ref. 5)

Brown soild (18.7 mg, 62% yield) (EtOAc:Hex = 1:6, Rf = 0.12), mp 182 - 186 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.95 (s, 1H), 8.44 – 8.35 (m, 1H), 7.76 (d, J = 7.1 Hz, 2H), 7.64 (d, J = 7.9 Hz, 1H), 7.48 - 7.24 (m, 6H), 7.18 (t, J = 7.5 Hz, 1H), 6.93 (dd, J = 7.4, 4.9 Hz, 1H), 6.75 (d, J = 8.1, 1H).



5ap

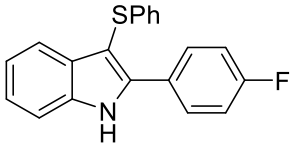
Brown soild (12.4 mg, 41yield), (EtOAc:Hex = 1:6 Rf = 0.11), mp 244 - 248 ^{o}C ^{1}H NMR (400 MHz, $CDCl_3$): 8.63 (s, 1H), 8.44 (d, *J* = 4.8 Hz, 2H), 7.74 (d, *J* = 8.0 Hz, 2H), 7.61 (d, *J* = 7.9 Hz, 1H), 7.44 - 7.34 (m, 4H), 7.30 - 7.22 (m, 1H), 7.16 (t, *J* = 7.1 Hz, 1H), 6.92 (t, *J* = 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 173.4, 157.6, 142.2, 135.8, 131.5, 131.1, 128.72, 128.68, 128.3, 123.3, 121.1, 119.7, 116.8, 111.4, 97.7; IR (ATR): ṽ = 3243, 2992, 2853,1564, 1549, 1457, 1374, 1182, 802, 771, 737, 685 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{12}N_3S^-$ $[M-1]^-$: 302.0757, found 302.0752.



3ba

Yellow oil (23.9 mg, 73 % yield) (EtOAc:Hex = 1:10, Rf = 0.32); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (s, 1H), 7.92 – 7.79 (m, 3H), 7.42 (d, *J* = 7.7 Hz, 1H), 7.33 – 7.14 (m, 4H), 2.68 (t, *J* = 7.2 Hz, 2H), 1.51 – 1.37 (m, 2H), 1.32 – 1.08 (m, 6H), 0.83 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.7 (d, J_{CF} = 247.0 Hz), 139.7, 135.6, 131.5, 130.2 (d, J_{CF} = 8.1 Hz), 128.3

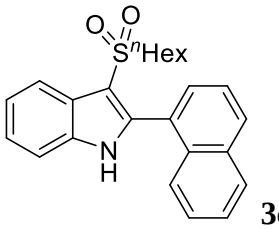
(d, J_{CF} = 3.0 Hz), 123.1, 120.8, 119.9, 115.6 (d, J_{CF} = 21.5 Hz), 111.0, 103.7, 36.4, 31.4, 29.5, 28.2, 22.5, 14.0.; ^{19}F NMR (376 MHz, $CDCl_3$) δ -112.92; IR (ATR): ṽ = 3404, 2956, 2926, 2855, 1607, 1541, 1483, 1452, 1419, 1392, 1351, 1321, 1296, 1229, 1158, 1095, 835, 795, 743 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{23}FNS^+$ [M+1]$^+$: 328.1530, found 328.1525.



5bb (ref. 6)

Brown oil. (31.3 mg, 98% yield) (EtOAc:Hex = 1:10, Rf = 0.35); ^{1}H NMR (400 MHz, $CDCl_3$):

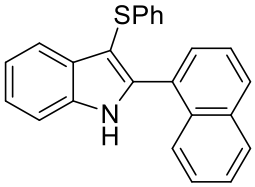
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (s, 1H), 7.75 (dd, *J* = 8.0, 6.0 Hz, 2H), 7.66 (d, *J* = 7.9 Hz, 1H), 7.48 (d, *J* = 8.1 Hz, 1H), 7.35 – 7.28 (m, 1H), 7.24 – 7.15 (m, 4H), 7.15 – 7.04 (m, 4H).



3ca'

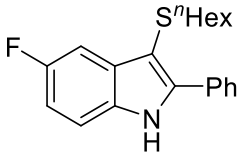
The reaction of **1c** and *n*HexSH, **2a** gave **3ca** in a poor yield along with a significant amount of the NH indole from (**A** in Table 1) which was inseparable from **3ca** by silica chromatography. Therefore, after measuring the crude yield of **3ca** (13%) from the ^{1}H NMR spectrum, the partially purified sample was oxidized by treating with excess *m*CPBA into the corresponding sulfone **3ca'** and was characterized as such.

Yellow oil (4.7 mg, 13 % yield), (EtOAc:Hex = 1:3, Rf = 0.50); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.75 (s, 1H), 8.23 (d, *J* = 7.8 Hz, 1H), 8.01 (d, *J* = 7.9 Hz, 1H), 7.94 (d, *J* = 8.1 Hz, 1H), 7.75 (d, *J* = 7.0 Hz, 1H), 7.65 – 7.51 (m, 3H), 7.51 – 7.45 (m, 2H), 7.45 – 7.34 (m, 2H), 2.93– 2.89 (m, 2H), 1.71 – 1.46 (m, 2H), 1.21 – 0.95 (m, 6H), 0.78 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.8, 134.6, 133.4, 132.5, 130.4, 130.2, 128.6, 127.0, 126.3, 125.0, 124.8, 124.2, 122.7, 120.8, 111.2, 57.09, 31.1, 27.8, 22.7, 22.2, 13.8. IR (ATR): ṽ = 3254, 2945, 2927, 2856, 1534, 1504, 1453, 1407, 1304, 1113, 1082, 803, 776, 749 cm^{-1}; HRMS (ESI) Calcd for $C_{24}H_{26}NO_2S^+$ [M+1]$^+$: 392.1679, found 392.1678.



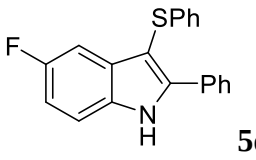
5cb

Yellow oil (19.3 mg, 55 % yield), (EtOAc:Hex = 1:10, Rf = 0.30); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (s, 1H), 7.95 (t, *J* = 7.4 Hz, 2H), 7.88 (d, *J* = 8.4 Hz, 1H), 7.71 (d, *J* = 7.9 Hz, 1H), 7.60 (d, *J* = 7.0 Hz, 1H), 7.57 – 7.48 (m, 3H), 7.49 – 7.42 (m, 1H), 7.35 (t, *J* = 7.6 Hz, 1H), 7.25 (t, *J* = 7.5 Hz, 1H), 7.18 – 7.07 (m, 4H), 7.04 (t, *J* = 6.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.7, 139.1, 136.0, 133.7, 132.3, 130.2, 129.6, 129.2, 129.0, 128.6, 128.5, 126.7, 126.2, 125.9, 125.6, 125.2, 124. 6, 123.3, 121.1, 120.1, 111.2, 102.3. IR (ATR): ṽ = 3393, 3057, 1581, 1503, 1477, 1425, 1438, 1352, 1269, 1078, 1023, 907, 802, 733, 689 cm^{-1}; HRMS (ESI) Calcd for $C_{24}H_{18}NS^{+}$ $[M+1]^{+}$: 352.1154, found 352.1146.



3da

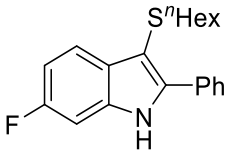
Brown oil (18.0 mg, 55 % yield), (EtOAc:Hex = 1:10, Rf = 0.25); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.35 (s, 1H), 7.87 (d, *J* = 7.1 Hz, 2H), 7.53-7.38 (m, 4H), 7.31 (dd, *J* = 8.7, 4.2 Hz, 1H), 6.98 (td, *J* = 9.1, 2.5 Hz, 1H), 2.63 (t, *J* = 7.2 Hz, 2H), 1.44 - 1.34 (m, 2H), 1.28 - 1.03 (m, 6H), 0.80 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.6 (d, J_{CF} = 234.0 Hz), 142.4, 132.6 (d, J_{CF} = 10.0 Hz), 132.0, 131.8, 128.7, 128.5, 128.3, 111.7 (d, J_{CF} = 9.0 Hz), 111.3 (d, J_{CF} = 26.0 Hz), 104.9 (d, J_{CF} = 24.0 Hz), 103.7, 36.4, 31.4, 29.5, 28.2, 22.5, 14.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ -122.94; IR (ATR): ṽ = 3420, 2927, 2856, 1627, 1582, 1479, 1454, 1441, 1317, 1272, 1216, 1156, 1112, 1032, 954, 857, 797, 766, 694, 607 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{23}FNS^{+}$ $[M+1]^{+}$: 328.1530, found 328.1524.



5db (ref. 4)

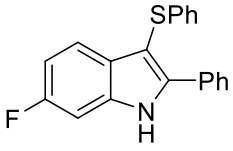
Yellow oil (28.1 mg, 88% yield) (EtOAc:Hex = 1:10 Rf =0.30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.56 (s, 1H), 7.74 (d, *J* = 6.8 Hz 2H), 7.47 - 7.34 (m, 4H), 7.27 (dd, *J* = 9.2, 2.5 Hz, 1H), 7.20

- 7.14 (m, 2H), 7.11 - 7.04 (m, 3H), 7.00 (td, *J* = 9.1, 2.5 Hz, 1H).



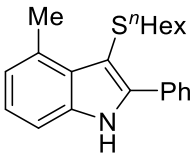
3ea

Brown oil (18.6 mg, 57 % yield), (EtOAc:Hex = 1:10, Rf = 0.21); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.32 (s, 1H), 7.88 (d, *J* = 7.7 Hz, 2H), 7.74 (dd, *J* = 8.6, 5.4 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 7.42 (t, *J* = 7.4 Hz, 1H), 7.11 (d, *J* = 9.1 Hz, 1H), 7.00 (t, *J* = 8.7 Hz, 1H) 2.64 (t, *J* = 7.2 Hz, 2H), 1.48 - 1.36 (m, 2H), 1.33 - 1.02 (m, 6H), 0.80 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.4 (d, J_{CF} = 237.0 Hz), 140.9 (d, J_{CF} = 3.5 Hz), 135.5, 135.4 131.9, 128.7, 128.3, 128.1, 120.7 (d, J_{CF} = 9.9 Hz), 109.3 (d, J_{CF} = 24.2 Hz), 103.7, 97.5 (d, J_{CF} = 26.3 Hz), 36.5, 31.4, 29.5, 28.2, 22.5, 14.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ -119.67; IR (ATR): $\tilde{\nu}$ = 3414, 2958, 2927, 2857, 1712, 1626, 1598, 1483, 1445, 1382, 1348, 1302, 1246, 1218, 1141, 1107, 1037, 958, 912, 834, 805, 765, 695 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{23}FNS^{+}$ $[M+1]^{+}$: 328.1530, found 328.1525.



5eb (ref. 4)

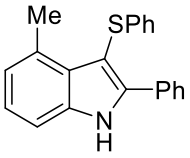
Yellow oil (31.6 mg, 99% yield) (EtOAc:Hex = 1:10 Rf =0.25); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (s, 1H), 7.73 (d, *J* = 6.9 Hz, 2H), 7.52 (dd, *J* = 8.8, 5.3 Hz, 2H), 7.46 - 7.35 (m, 3H), 7.20-7.11 (m, 3H), 7.10-7.03 (m, 3H), 6.92 (ddd, *J* = 8.8, 2.3, 0.9 Hz, 1H).



3fa

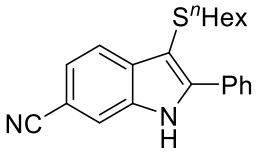
Brown oil (26.8 mg, 83 % yield), (EtOAc:Hex = 1:10, Rf = 0.43); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.30 (s, 1H), 7.81 (d, *J* = 7.0 Hz, 2H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.43 - 7.37 (m, 1H), 7.23 (d, *J* = 8.1 Hz, 1H), 7.12 (t, *J* = 7.6 Hz, 1H), 6.93 (d, *J* = 7.2 Hz, 1H), 2.98 (s, 3H), 2.56 (t, *J* = 7.2 Hz, 2H), 1.37 - 1.28 (m, 2H), 1.20 - 0.98 (m, 6H), 0.78 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.8, 135.9, 132.5, 132.2, 129.0, 128.7, 128.5, 128.2, 122.7, 108.9, 103.8,

38.9, 31.4, 28.7, 28.3, 22.5, 19.3, 14.0; IR (ATR): ṽ = 3393, 2925, 2855, 1602, 1574, 1502, 1480, 1447, 1414, 1396, 1325, 1245, 1157, 1074, 1032, 911, 768, 748, 694 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{26}NS^+$ $[M+1]^+$: 324.1780, found 324.1769.



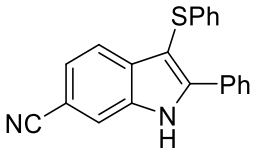
5fb

Yellow solid (29.6 mg, 94 % yield), (EtOAc:Hex = 1:10, Rf = 0.28), mp 142 - 144 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.58 (s, 1H), 7.65 (dd, *J* = 8.3, 1.7 Hz, 2H), 7.43 - 7.32 (m, 3H), 7.23 (d, *J* = 8.1 Hz, 1H), 7.20 - 7.11 (m, 3H), 7.09 - 7.01 (m, 3H), 6.89 (dt, *J* = 7.2, 0.8 Hz, 1H), 2.67 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.0, 141.8, 136.2, 132.4, 131.7, 128.9, 128.7, 128.7, 128.6, 128.6, 125.0, 124.4, 123.2, 123.0, 98.9, 18.9; IR (ATR): ṽ = 3403, 3050, 1582, 1504, 1478, 1447, 1439, 1415, 1395, 1352, 1325, 1265, 1247, 1157, 1081, 1024, 769, 748, 692 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{18}NS^+$ $[M+1]^+$: 316.1154, found 316.1151.



3ga

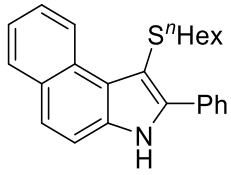
Yellow oil (13.0 mg, 39 % yield), (EtOAc:Hex = 1:3, Rf = 0.45); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.67 (s, 1H), 7.93 (d, *J* = 7.5 Hz, 2H), 7.90 (d, *J* = 8.4 Hz, 1H), 7.75 (s, 1H), 7.55 (t, *J* = 7.4 Hz, 2H), 7.49 (t, *J* = 7.7 Hz, 2H), 2.66 (t, *J* = 7.1 Hz, 2H), 1.42 - 1.33 (m, 2H), 1.29 - 1.02(m, 6H), 0.80 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ ; 144.2, 135.0, 134.4, 131.0, 129.2, 128.9, 128.4, 123.8, 120.7, 120.4, 115.7, 105.3, 104.8, 36.5, 31.3, 29.5, 28.1, 22.5, 14.0; IR (ATR): ṽ = 3306, 2926, 2855, 2221, 1620, 1580, 1536, 1459, 1381, 1348, 1318, 1226, 1138, 1034, 1012, 909, 872, 816, 769, 732, 693 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{23}N_2S^+$ $[M+1]^+$: 335.1576, found 335.1580.



5gb

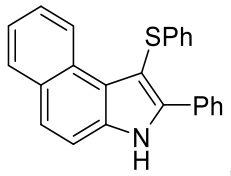
White solid (16.3 mg, 50 % yield), (EtOAc:Hex = 1:3, Rf = 0.35); mp 164 - 168 °C; ^{1}H NMR

(400 MHz, $CDCl_3$): δ 8.90 (s, 1H), 7.82-7.76 (m, 3H), 7.68 (d, *J* = 8.2 Hz, 1H), 7.51-7.43 (m, 3H), 7.39 (dd, *J* = 8.2, 1.2 Hz, 1H), 7.18 (t, *J* = 7.2 Hz, 2H), 7.11 - 7.03 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.6, 138.2, 134.7, 134.6, 130.3, 129.6, 129.0, 128.3, 125.7, 125.1, 124.2, 120.8, 120.3, 115.9, 105.6, 100.8; IR (ATR): ṽ = 3290, 2922, 2853, 2221, 1730, 1621, 1585, 1538, 1480, 1459, 1447, 1378, 1345, 1287, 1229, 1123, 1080, 1036, 873, 804, 767, 726, 683, 658, 621 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{15}N_2S^+$ $[M+1]^+$: 327.0950, found 327.0957.



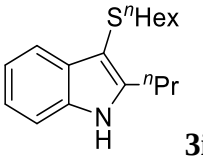
3ha

Yellow oil (6.1 mg, 17 % yield), (EtOAc:Hex = 1:10, Rf = 0.35); ^1H NMR (400 MHz, $CDCl_3$): δ 9.66 (d, *J* = 8.4 Hz, 1H), 8.65 (s, 1H), 7.92 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 7.7 Hz, 2H), 7.67 - 7.59 (m, 2H), 7.55 - 7.37 (m, 5H), 2.71 (t, *J* = 7.1 Hz, 2H), 1.41 - 1.32 (m, 2H), 1.23 - 0.97 (m, 6H), 0.77 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 77.0): δ 140.0, 132.5, 132.3, 130.1, 129.3, 128.9, 128.7, 128.5, 128.0, 125.9, 124.3, 123.6, 123.3, 123.1, 112.4, 105.3, 36.9, 31.4, 28.8, 28.3, 22.5, 14.0; IR (ATR): ṽ = 3403, 2926, 2853, 1603, 1523, 1453, 1410, 1377, 1334, 1243, 1203, 1069, 1018, 978, 803, 764, 695 cm^{-1}; HRMS (ESI) Calcd for $C_{24}H_{26}NS^+$ $[M+1]^+$: 360.1780, found 360.1777.



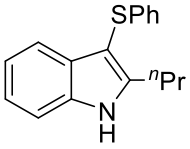
5hb

Yellow oil (27.0 mg, 77 % yield), (EtOAc:Hex = 1:10, Rf = 0.30); ^1H NMR (400 MHz, $CDCl_3$): δ 9.20 (dd, *J* = 8.0, 1.6 Hz, 1H), 8.81 (s, 1H), 7.88 (d, *J* = 7.4 Hz, 1H), 7.71 - 7.65 (m, 3H), 7.54 (d, *J* = 8.8 Hz, 1H), 7.45 - 7.34 (m, 5H), 7.19 - 7.11 (m, 4H), 7.05 - 7.00 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.2, 139.7, 132.8, 131.6, 130.2, 129.0, 128.9, 128.7, 128.6, 128.5, 126.2, 125.4, 124.9, 124.7, 123.9, 123.5, 123.3, 112.4, 100.7; IR (ATR): ṽ = 3400, 3057, 1582, 1476, 1454, 1439, 1356, 1264, 1081, 1024, 978, 766, 737, 670 cm^{-1}; HRMS (ESI) Calcd for $C_{24}H_{18}NS^+$ $[M+1]^+$: 352.1154, found 352.1148.



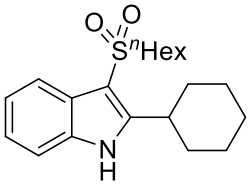
3ia

Yellow oil (9.4 mg, 34 % yield), (EtOAc:Hex = 1:10, Rf = 0.37); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (s, 1H), 7.78 – 7.65 (m, 1H), 7.36 – 7.30 (m, 1H), 7.24-7.12 (m, 2H), 2.95 (t, *J* = 7.6 Hz, 2H), 2.67 (t, *J* = 7.3 Hz, 2H), 1.81 – 1.67 (m, 2H), 1.60 – 1.49 (m, 2H), 1.45 – 1.34 (m, 2H), 1.34 – 1.15 (m, 4H), 1.03 (t, *J* = 7.3 Hz, 3H), 0.89 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.8, 135.3, 130.7, 121.8, 120.2, 119.0, 110.5, 102.7, 36.4, 31.5, 30.0, 28.4, 28.4, 23.0, 22.6, 14.0, 13.9.IR (ATR): ν̃ = 3284, 2952, 2930, 2872, 1534, 1457, 1423, 1297, 1281, 1145, 1112, 1096, 1070, 1014, 771, 748 cm^{-1}; HRMS (ESI) Calcd for $C_{17}H_{26}NS^+$ $[M+1]^+$: 276.1780, found 276.1781.



5ib (ref. 7)

Yellow oil (16.0 mg, 60% yield) (EtOAc:Hex = 1:10, Rf = 0.43); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (s, 1H), 7.52 (d, J = 8.0 Hz, 1H), 7.31 (d, J = 8.0 Hz, 1H), 7.17 (t, J = 8.0 Hz, 1H), 7.10 (q, J = 6.8 Hz, 3H), 7.06 (d, *J* = 7.4 Hz, 3H), 2.75 (d, J = 6.8 Hz, 2H), 7.06 (d, *J* = 7.4 Hz, 3H), 2.92 (t, *J* = 7.6 Hz, 2H), 1.78 – 1.66 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H).

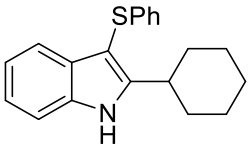


3ja' (characterized after oxidation into a sulfone)

The reaction of **1j** and *n*HexSH, **2a** gave **3ja** which was inseparable from the NH indole from **1c** by silica chromatography. Therefore, after measuring the crude yield of **3ja** (39%) from the ^{1}H NMR spectrum, the partially purified sample was oxidized by treating with excess *m*CPBA into the corresponding sulfone **3ja'** and was characterized as such.

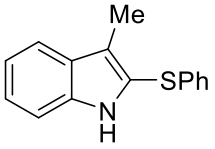
Yellow oil (12.3 mg, 39 % yield), (EtOAc:Hex = 1:3, Rf = 0.20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (s, 1H), 8.04 – 7.92 (m, 1H), 7.44 – 7.35 (m, 1H), 7.33 – 7.22 (m, 2H), 3.81 – 3.73 (td,

J = 11.6, 2.7 Hz 1H), 3.17 (d, *J* = 8.1 Hz, 2H), 2.12 – 2.01 (m, 2H),1.94 – 1.71 (m, 5H), 1.60 – 1.41 (m, 4H), 1.41 – 1.32 (m, 2H), 1.32 – 1.19 (m, 5H), 0.86 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.2, 134.3, 125.7, 123.2, 122.0, 119.9, 111.0, 108.2, 57.5, 35.2, 33.3, 31.3, 28.0, 26.2, 25.9, 22.8, 22.3, 13.9. IR (ATR): ṽ = 3297, 2927, 2854, 1600, 1449, 1424, 1299, 1280, 1247, 1136, 1124, 1112, 1124, 1075, 954, 748, 696 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{30}NO_2S^+$ $[M+1]^+$: 348.1992, found 348.1988.



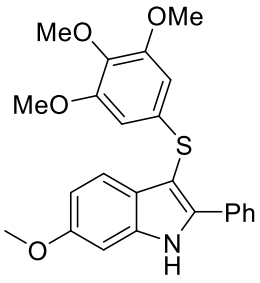
5jb (ref. 4)

Yellow oil (5.8 mg, 19% yield), (EtOAc:Hex = 1:10, Rf = 0.25); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (s, 1H), 7.55 (d, *J* = 7.8 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.19 – 7.10 (m, 3H), 7.06 (d, *J* = 7.6 Hz, 3H), 3.30 – 3.18 (m, 1H), 2..00 – 1.83 (m, 2H), 1.88 – 1.75 (m, 4H), 1.45 – 1.38 (m, 2H), 1.34 – 1.18 (m, 2H).



5ka (ref. 8)

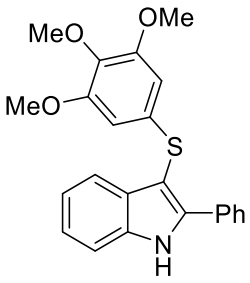
White solid (10.7 mg, 45% yield) (EtOAc:Hex = 1:10, Rf = 0.30), mp 75 – 78 ºC, ^{1}H NMR (400 MHz, $CDCl_3$): 8.02 (s, 1H), 7.64 (d, *J* = 7.9 Hz, 1H), 7.29 – 7.05 (m, 6H), 7.09 (d, *J* = 7.9 Hz, 2H), 2.43 (s, 3H).



5mq (ref. 11)

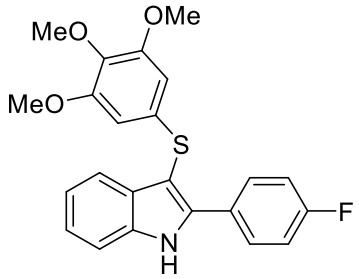
White solid (21.5 mg, 51% yield) (EtOAc:Hex = 1:3, Rf = 0.25), mp 120 – 124 °C, ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.89 (s, 1H), 7.85 (d, *J* = 7.9 Hz, 2H), 7.50 (t, *J* = 7.2 Hz, 2H), 7.43 – 7.30 (m, 2H), 6.98 (s, 1H), 6.77 (d, *J* = 8.7 Hz, 1H), 6.31 (s, 2H), 3.82 (s,

3H), 3.60 (s, 3H), 3.55 (s, 6H).



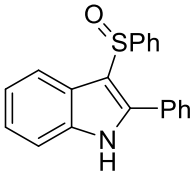
5nq (ref. 12)

White solid (35.6 mg, 91% yield) (EtOAc:Hex = 1:3, Rf = 0.20), mp 158 – 160 °C. ^{1}H NMR(400 MHz, $CDCl_3$): δ 8.64 (s, 1H), 7.82 (d, *J* = 7.4 Hz, 2H), 7.71 (d, *J* = 7.8 Hz, 1H), 7.52 – 7.39 (m, 3H), 7.34 – 7.26 (m, 2H), 7.22 (t, *J* = 7.2 Hz, 1H), 6.38 (s, 2H), 3.79 (s, 3H), 3.65 (s, 6H).



5oq (ref. 12)

White solid (36.0 mg, 88% yield) (EtOAc:Hex = 1:3, Rf = 0.25), mp 170 – 175 °C , ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.11 (s, 1H), 7.97 – 7.85 (m, 2H), 7.50 (t, *J* = 7.7 Hz, 2H), 7.39 (t, *J* = 8.7 Hz, 2H), 7.23 (t, *J* = 7.6 Hz, 1H), 7.11 (d, *J* = 7.5 Hz, 1H), 6.30 (s, 2H), 3.57 (s, 3H), 3.53 (s, 6H).



6aa (ref. 4)

White solid (14.5 mg, 91% yield), (EtOAc:Hex = 1:3, Rf = 0.20), ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.75 (s, 1H), 7.74 – 7.59 (m, 4H), 7.51 – 7.31 (m, 7H), 7.25 (d, *J* = 8.1 Hz, 1H), 7.12 (t, *J* = 7.7 Hz, 1H), 6.94 (t, *J* = 7.6 Hz, 1H).

6. References

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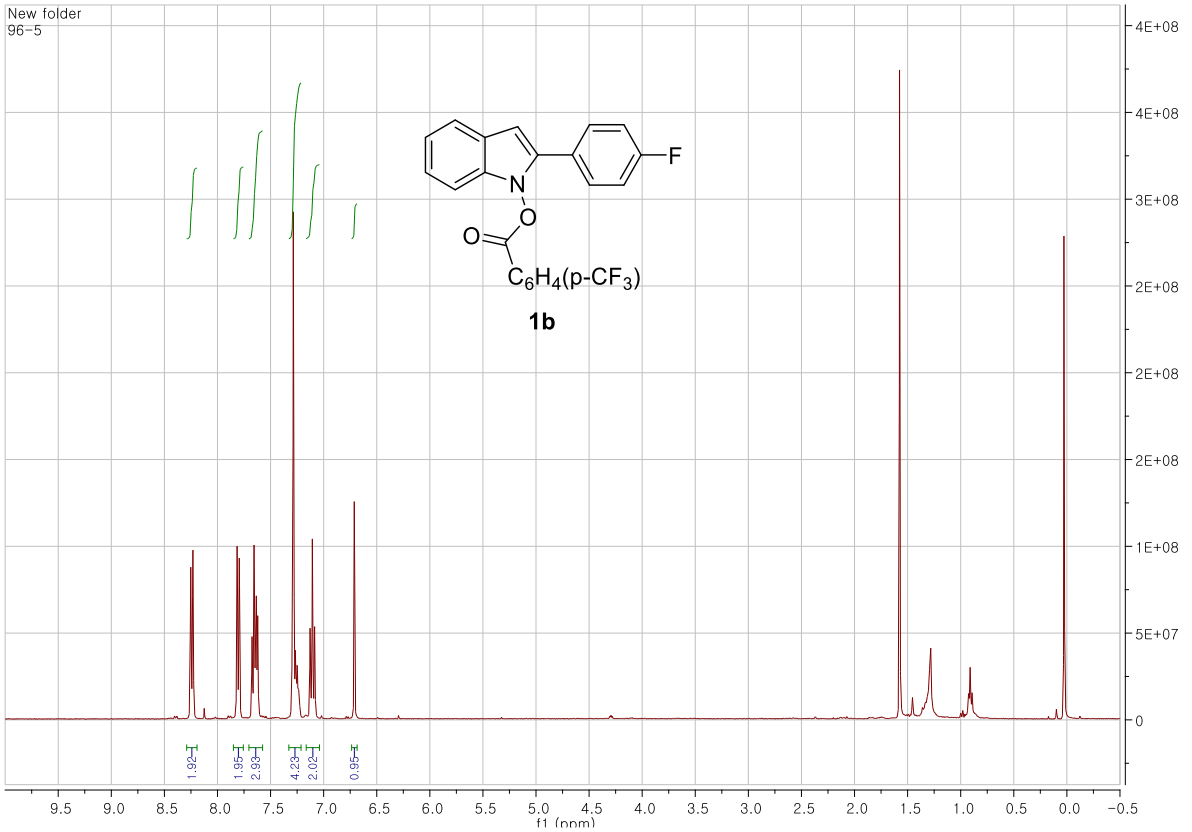
Scanned Spectra of Unknown Compounds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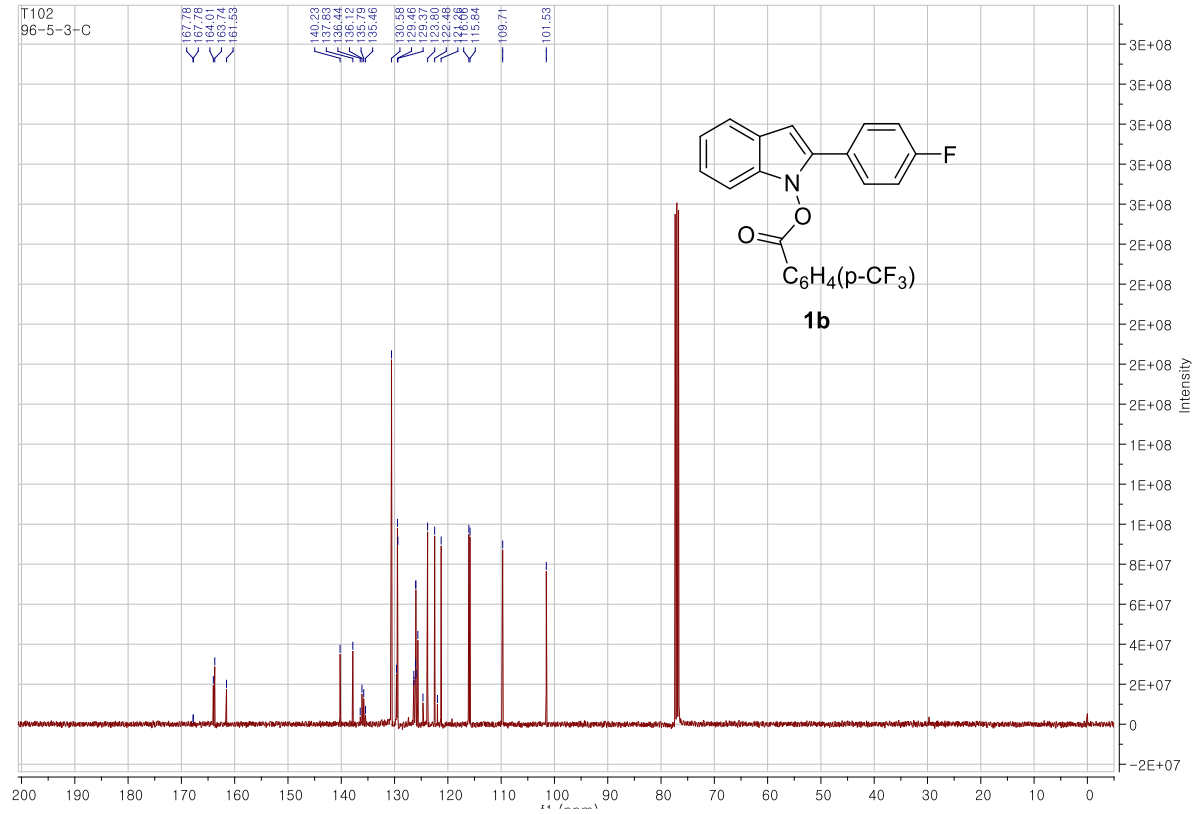
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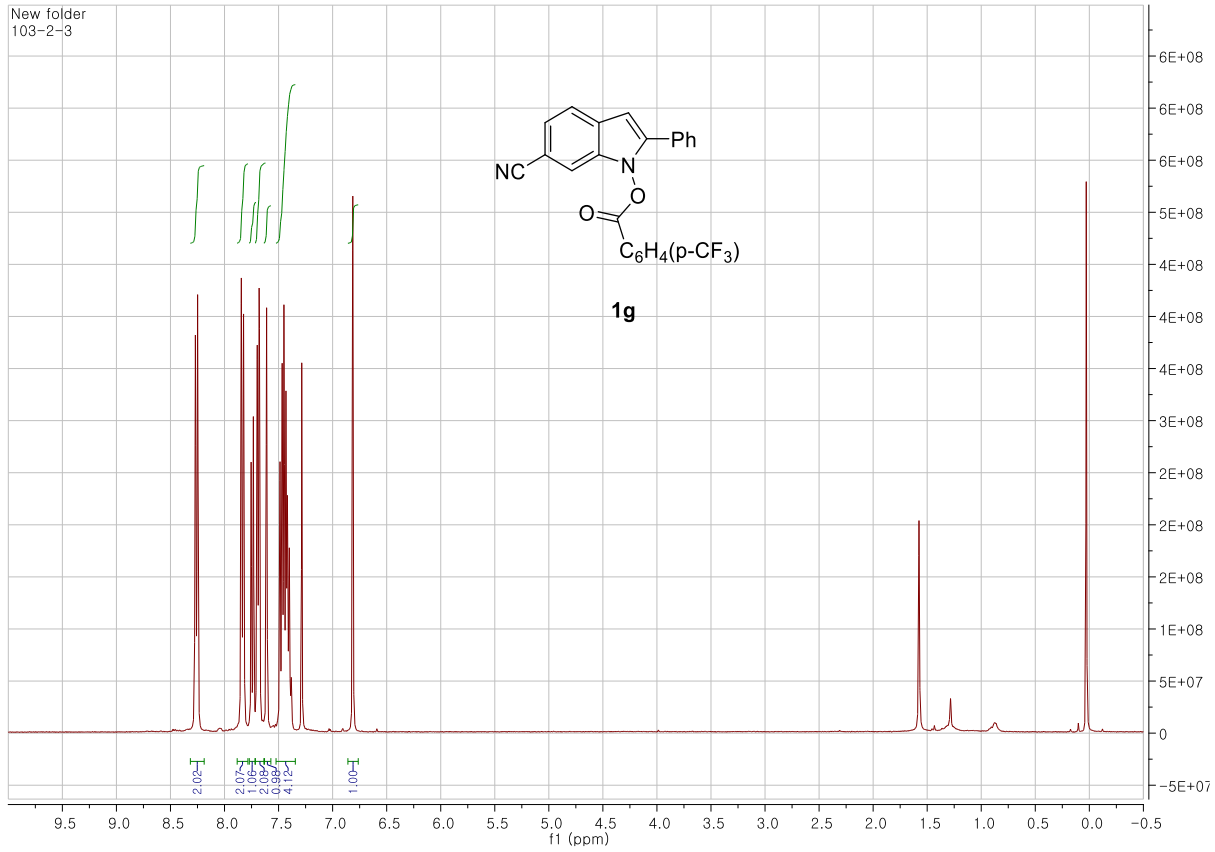
Intensity
f1 (ppm)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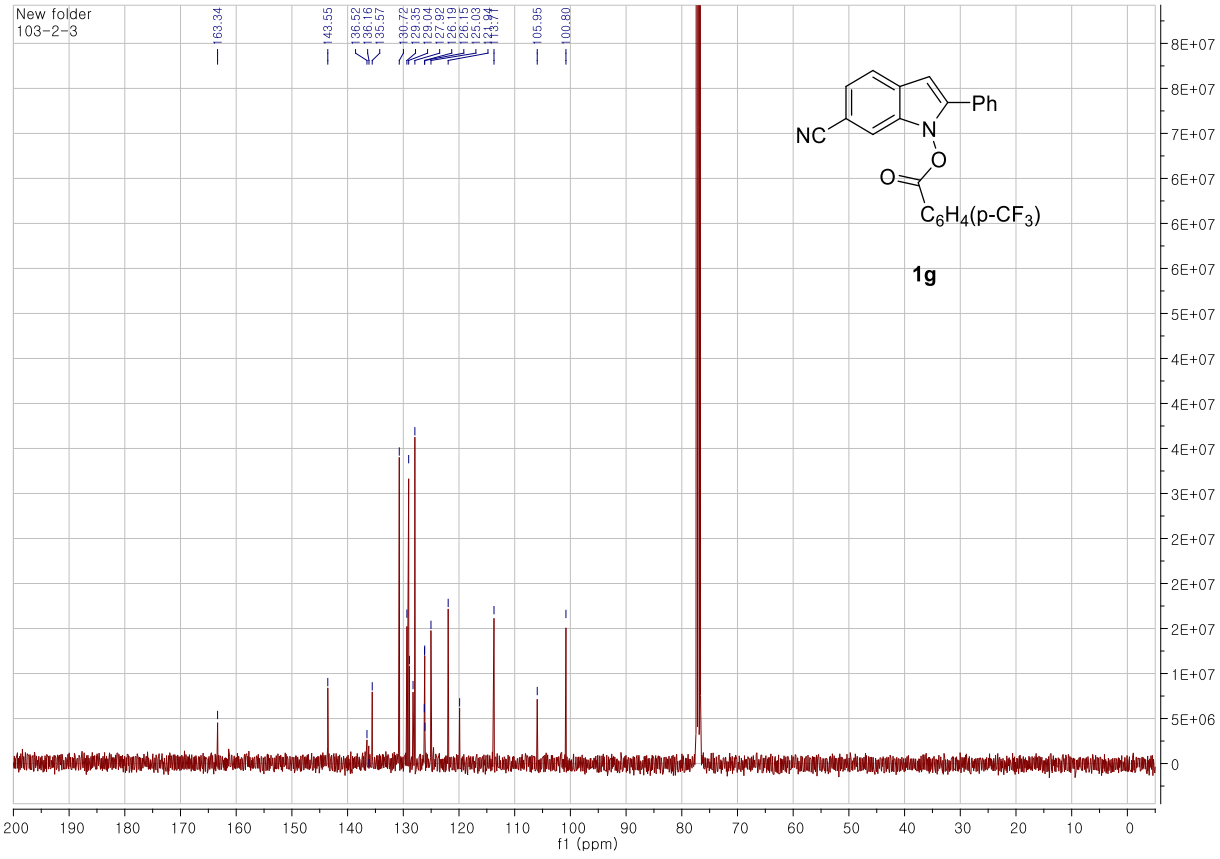
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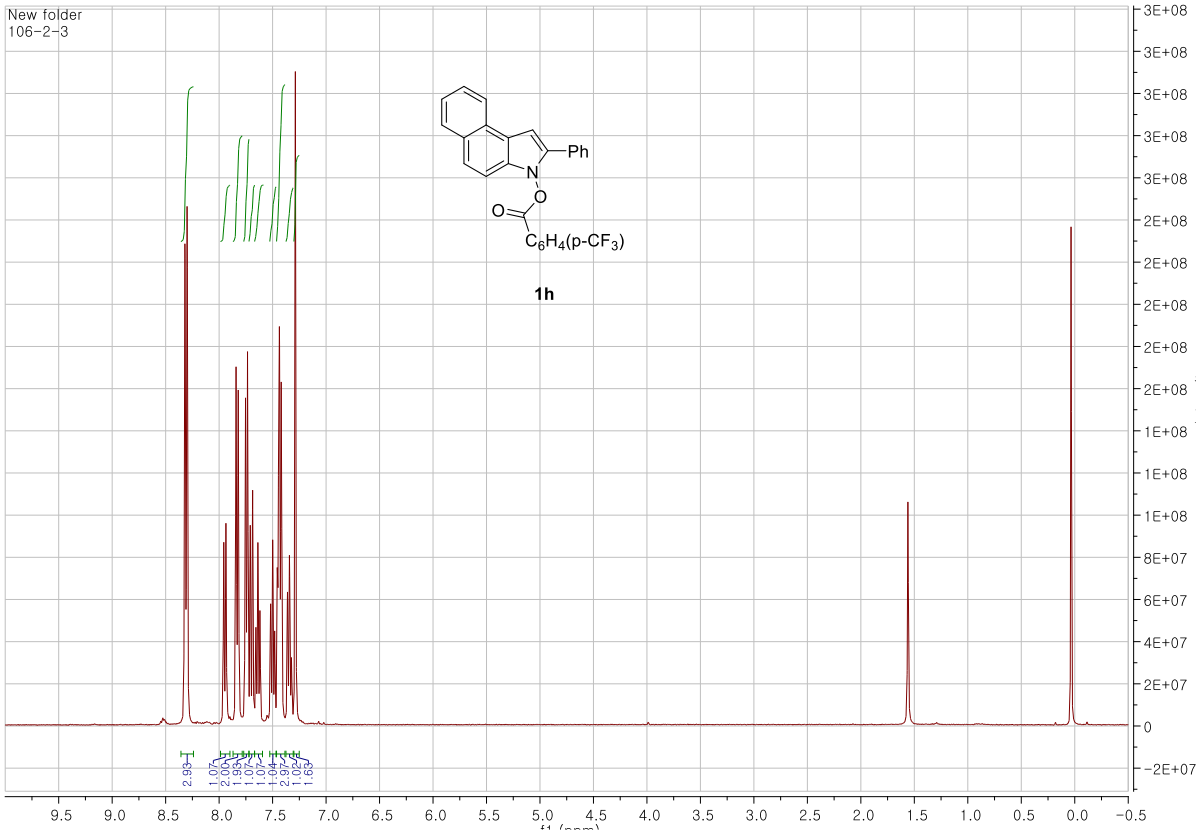
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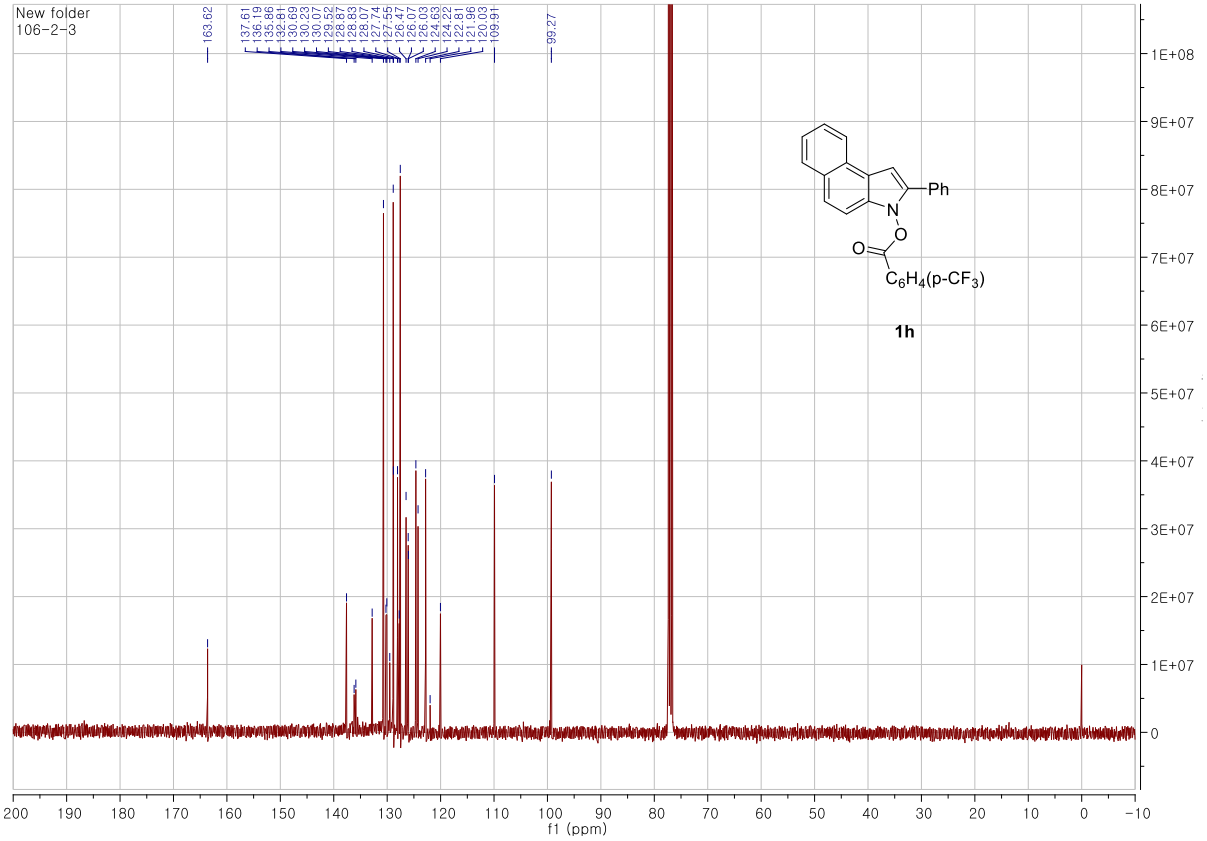
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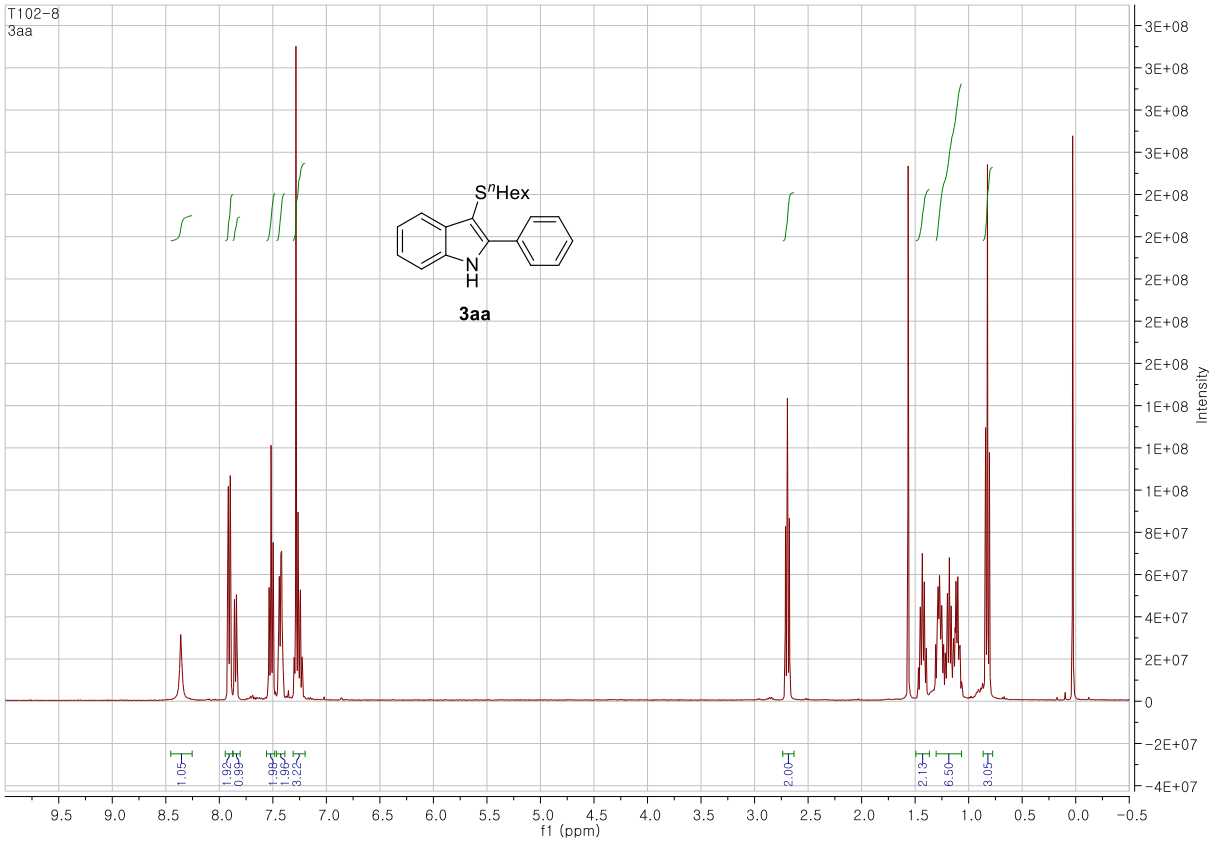
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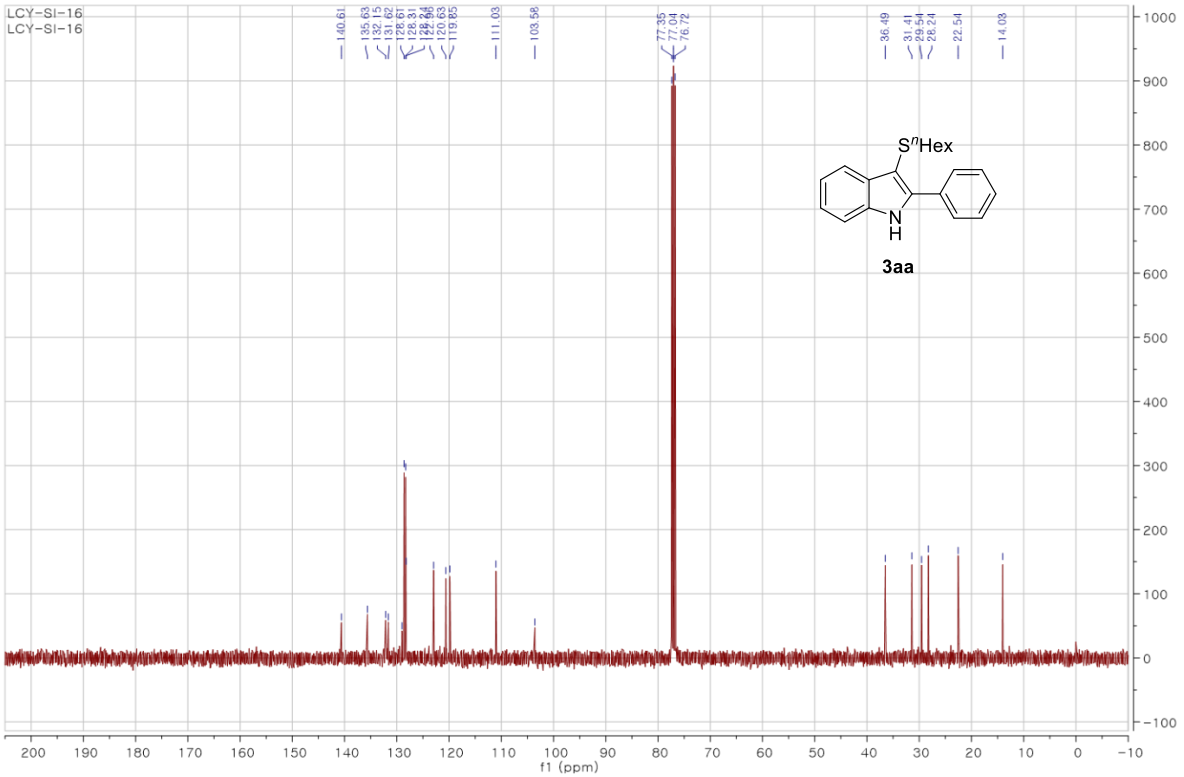
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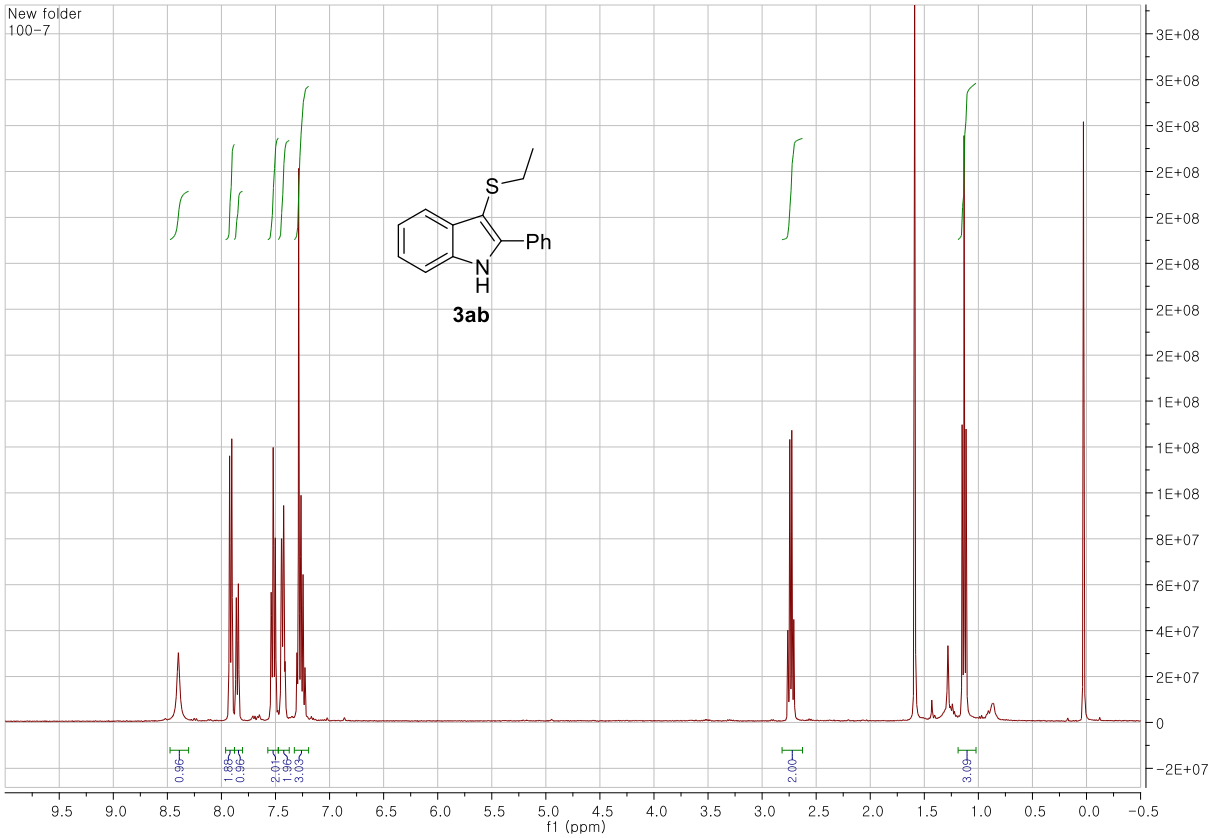
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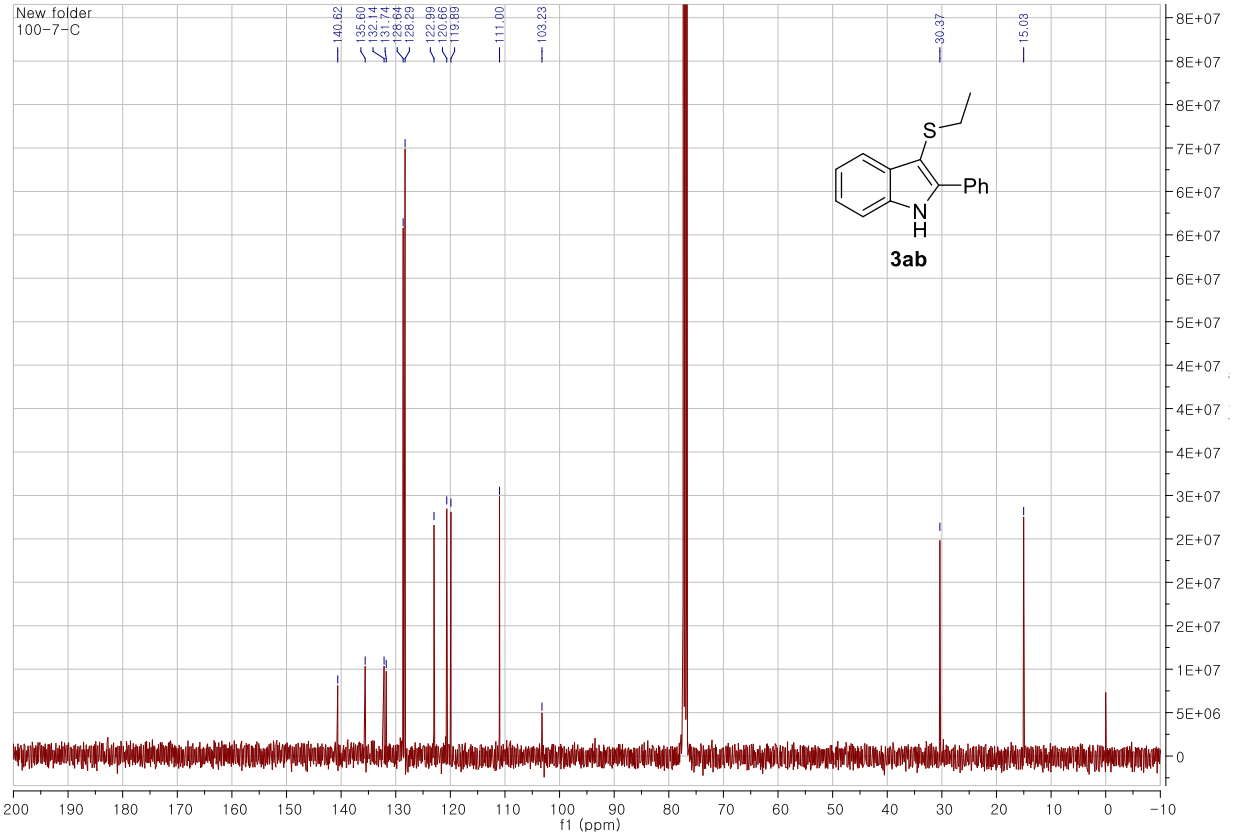
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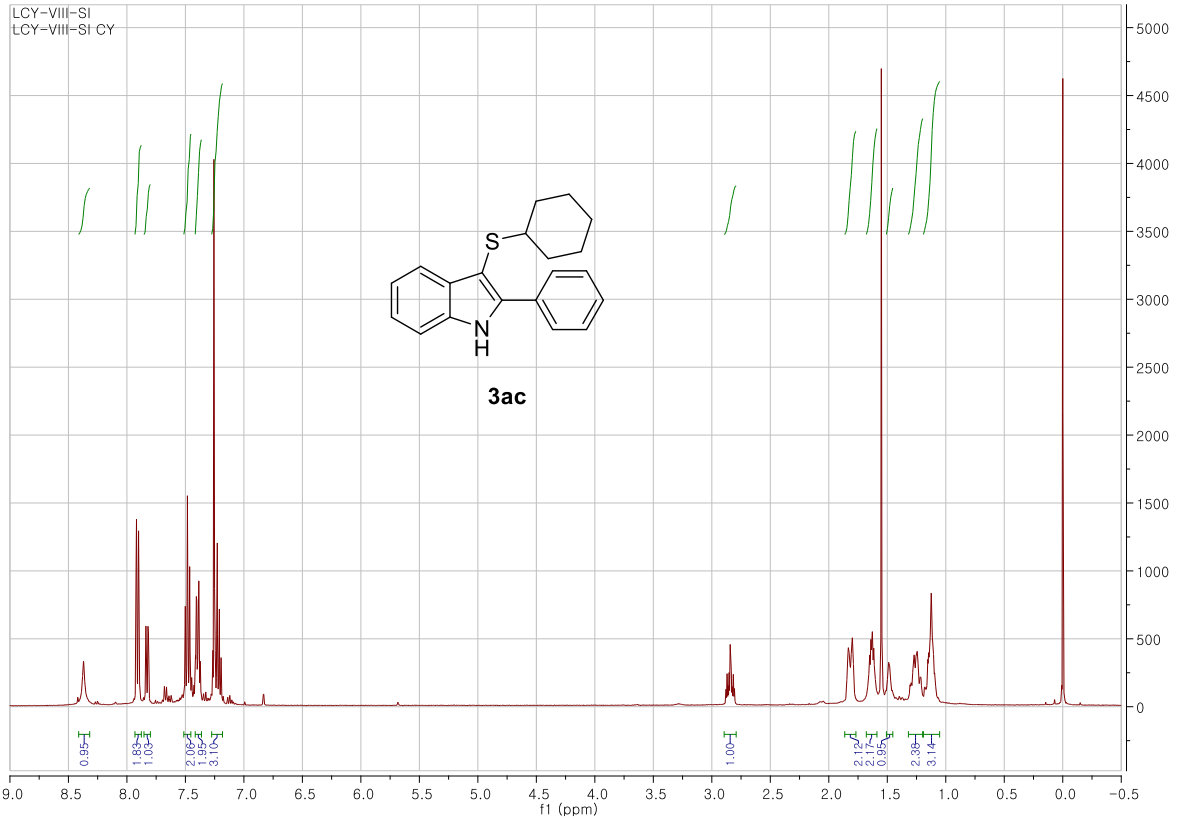
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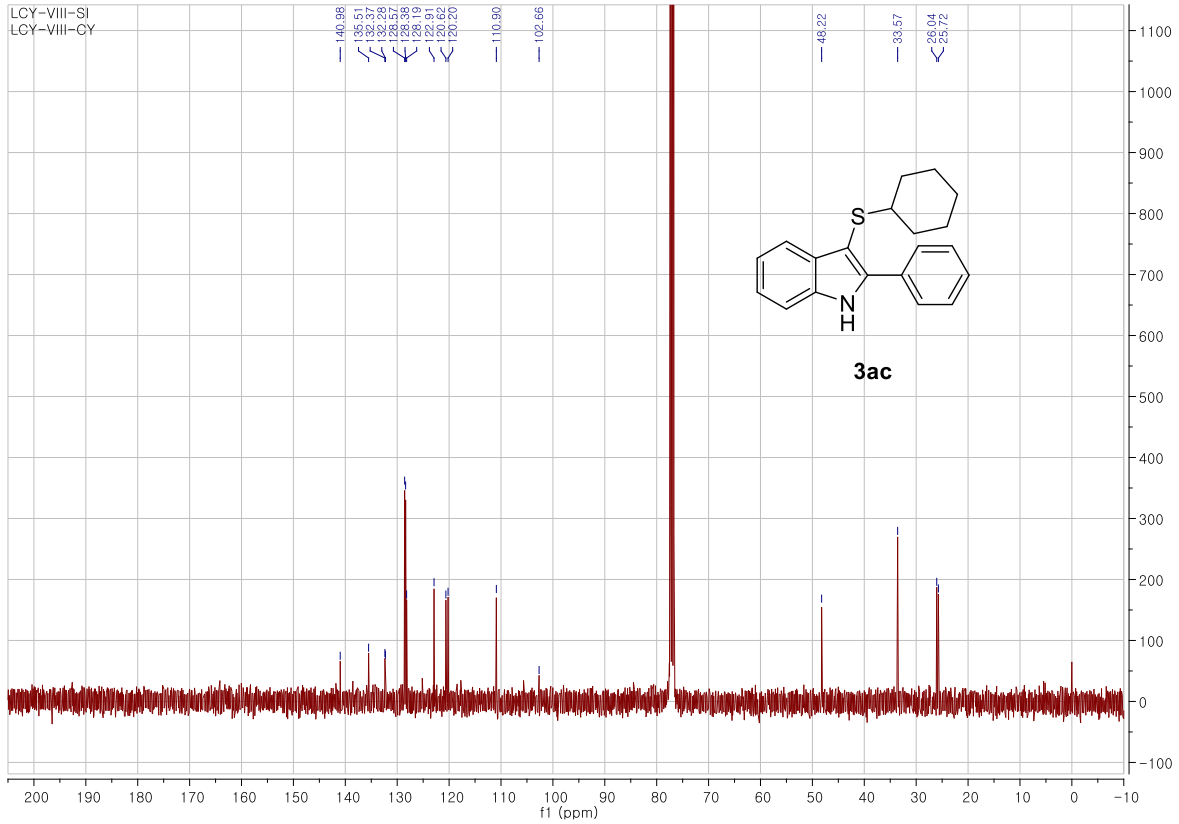
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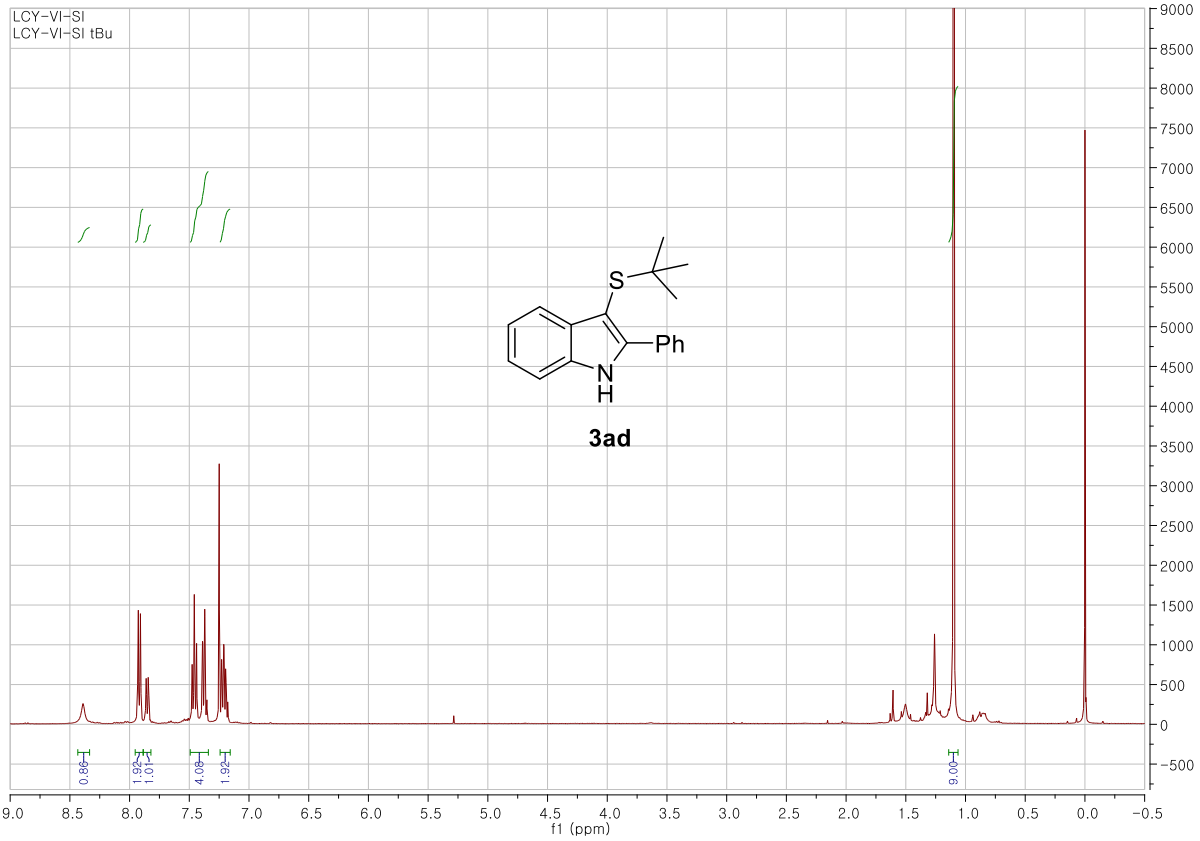
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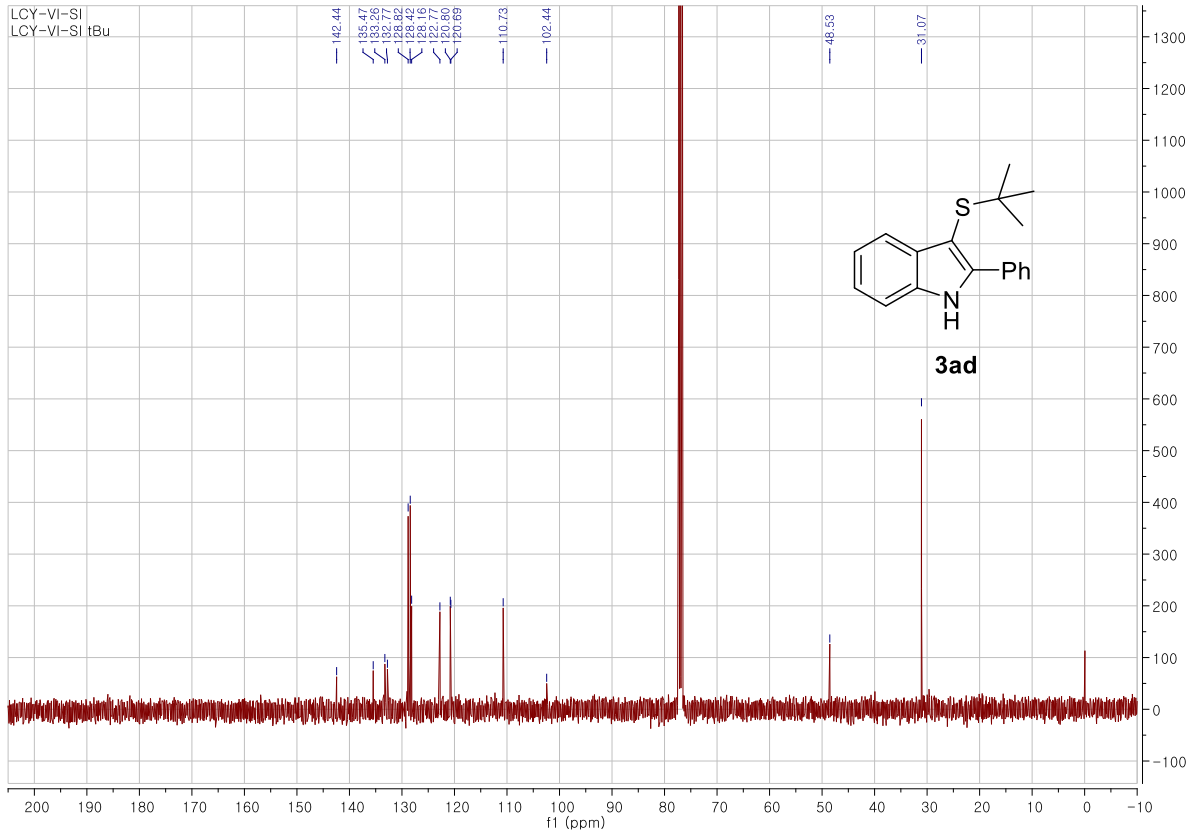
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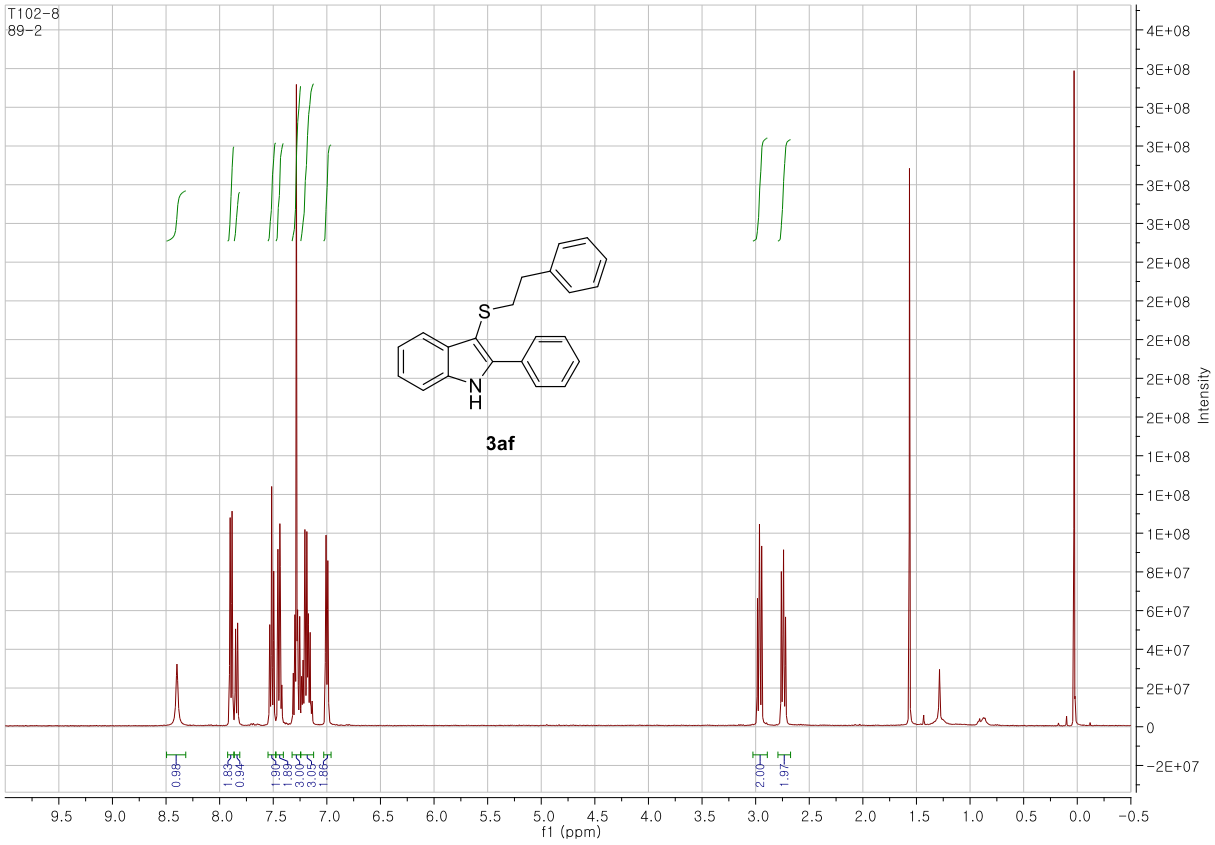
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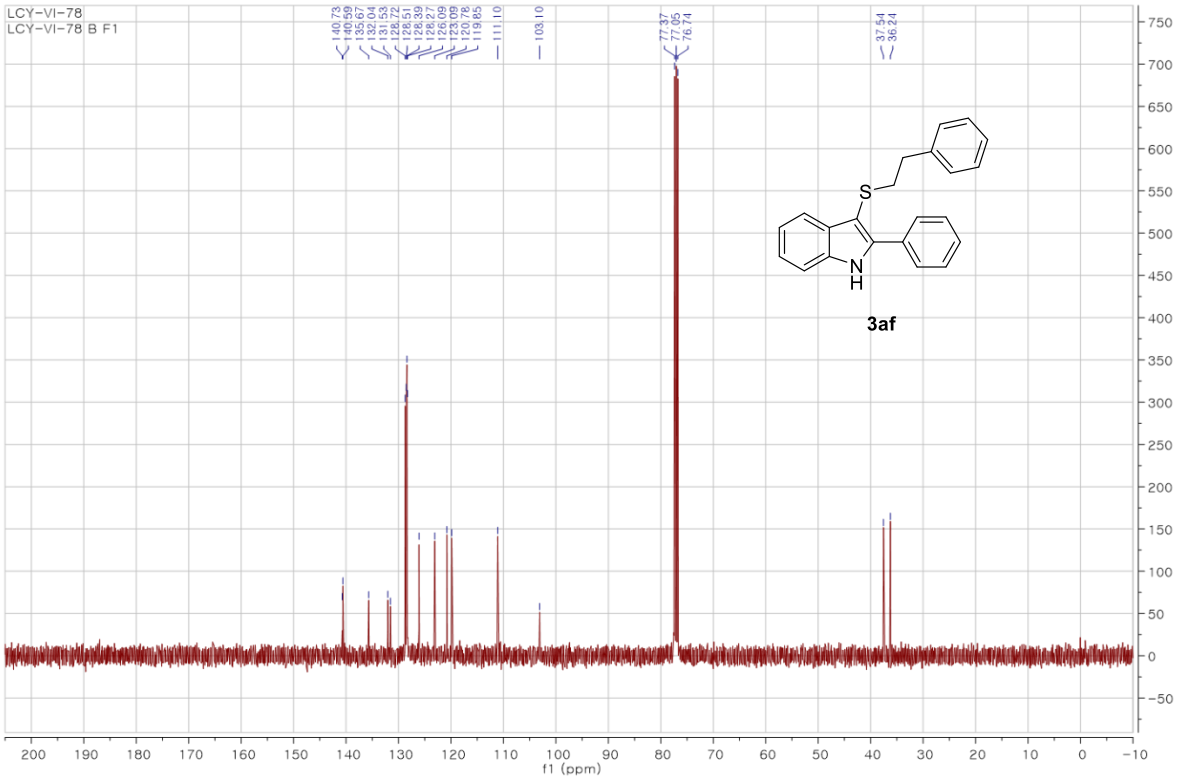
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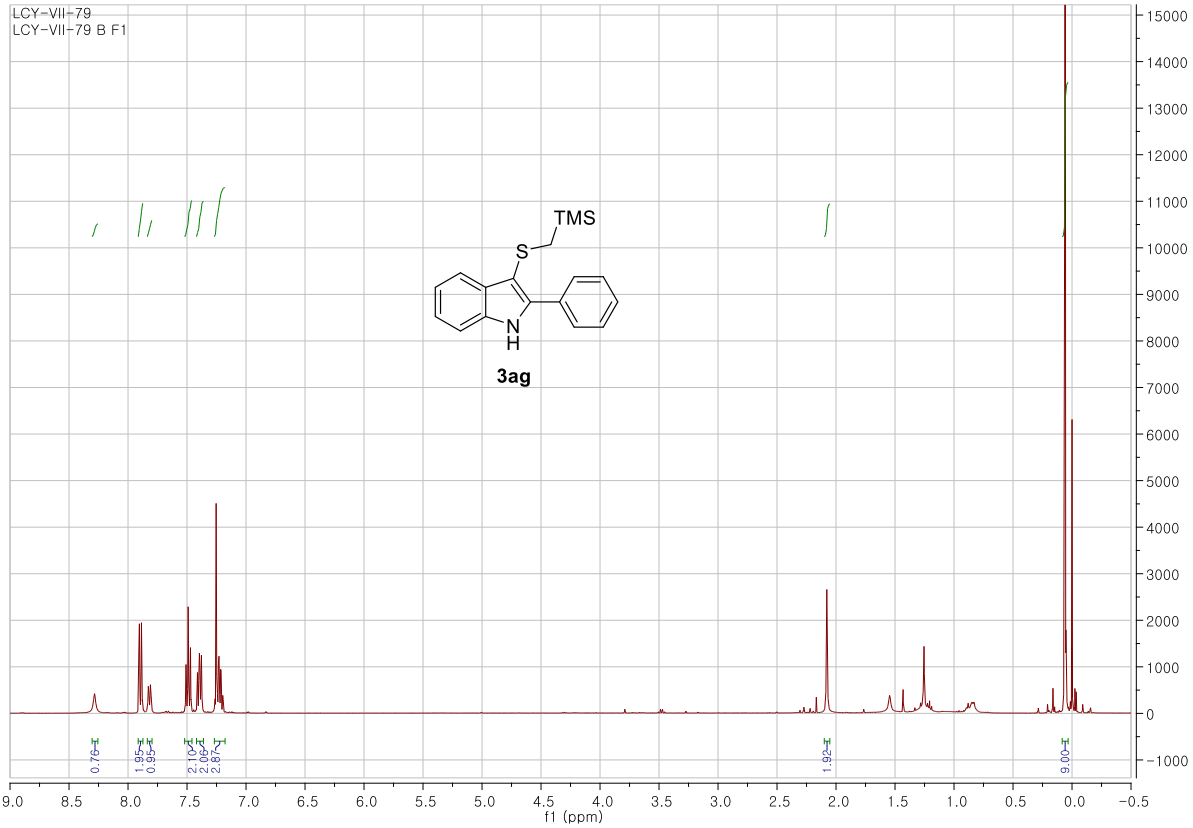
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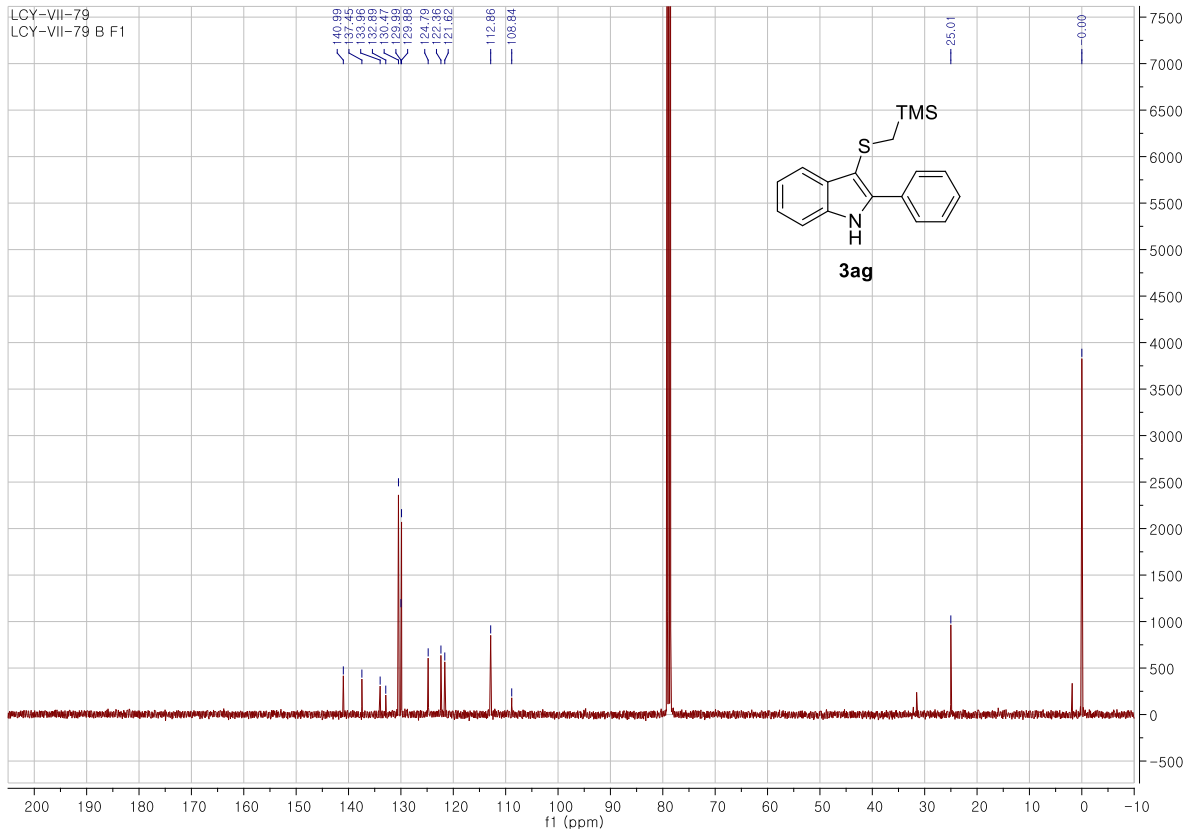
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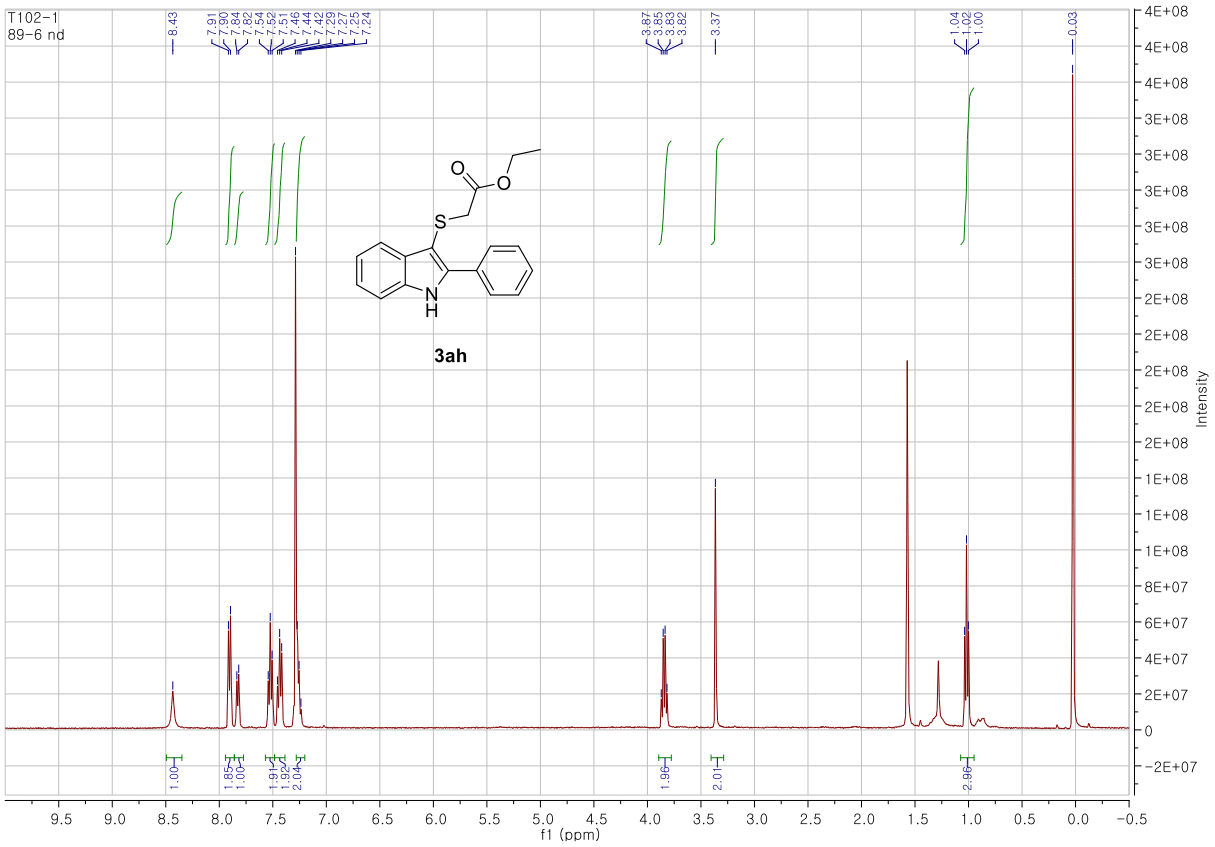
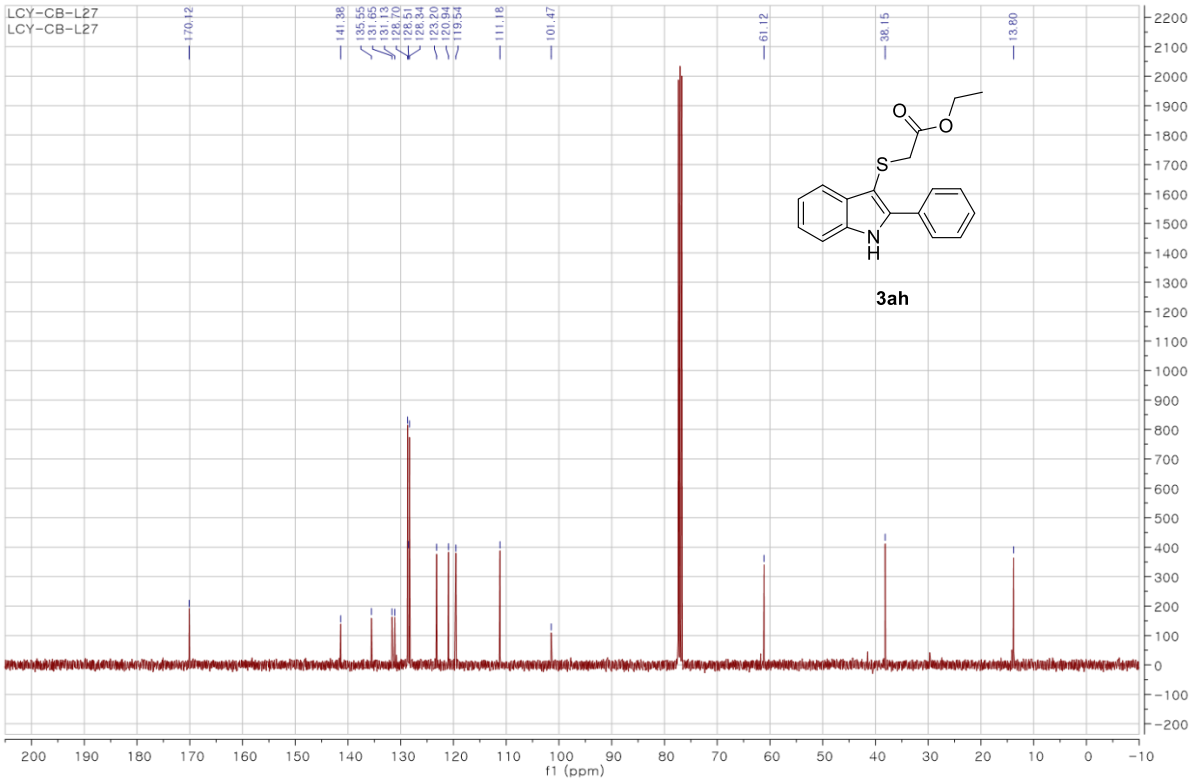
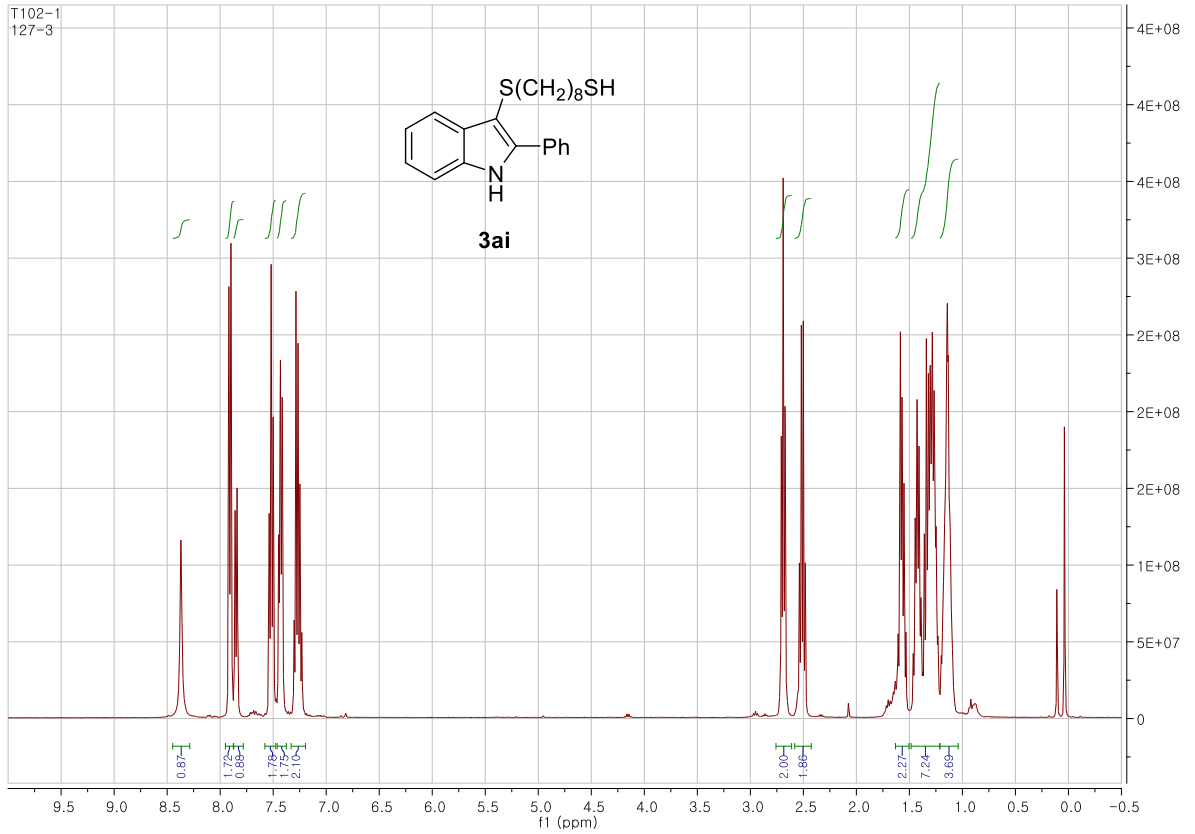
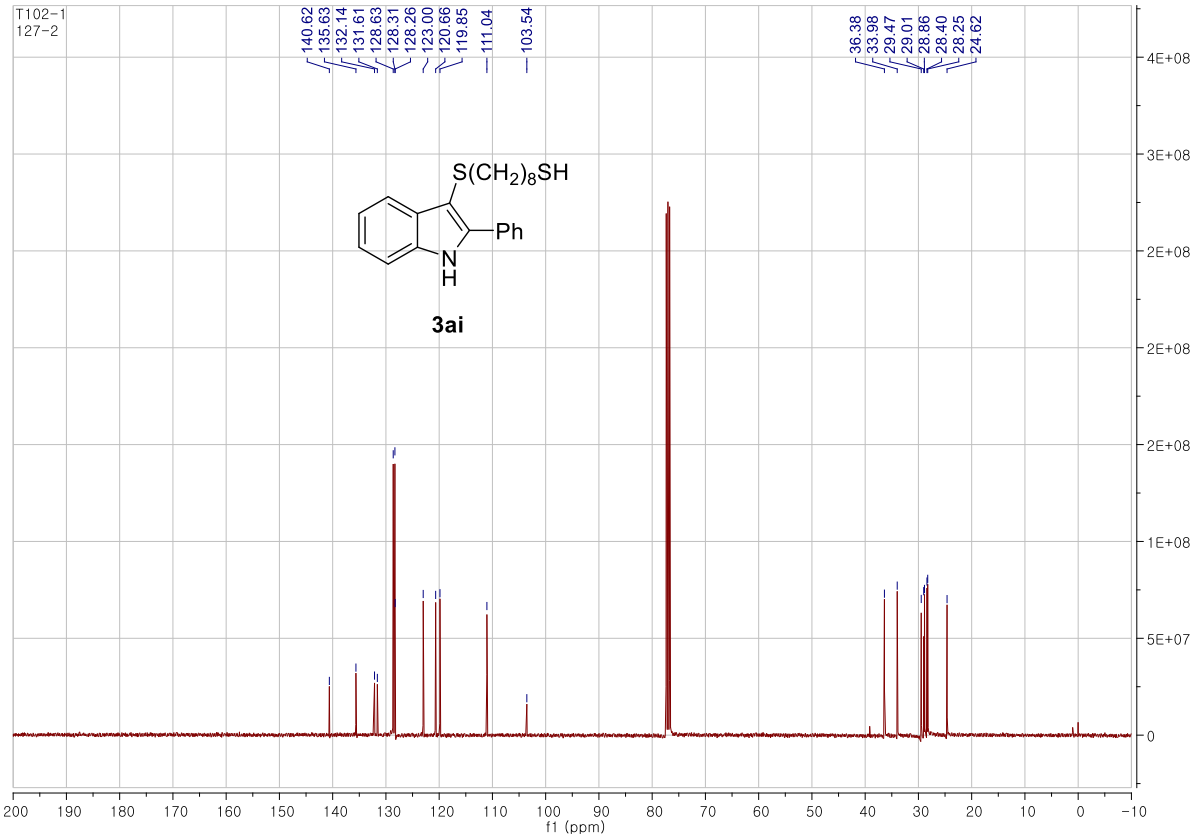
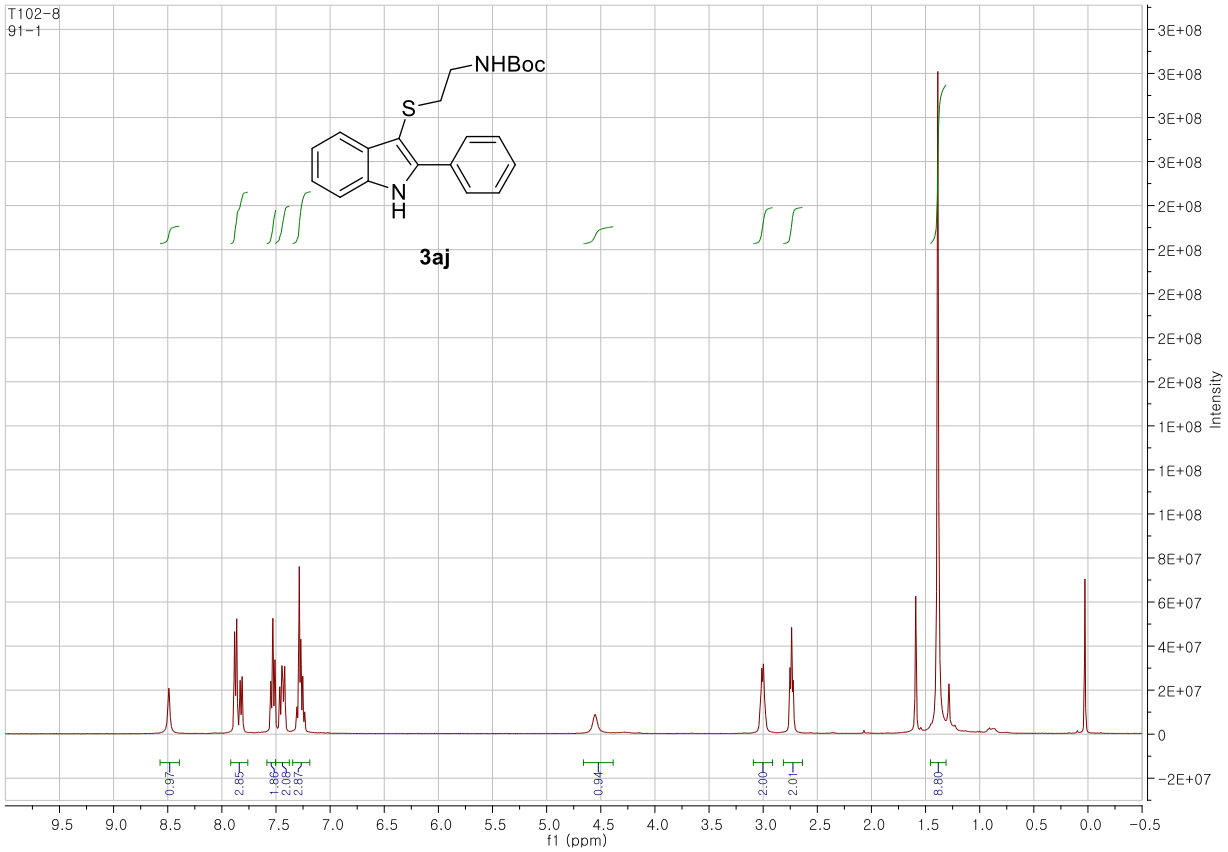
3ag
f1 (ppm)

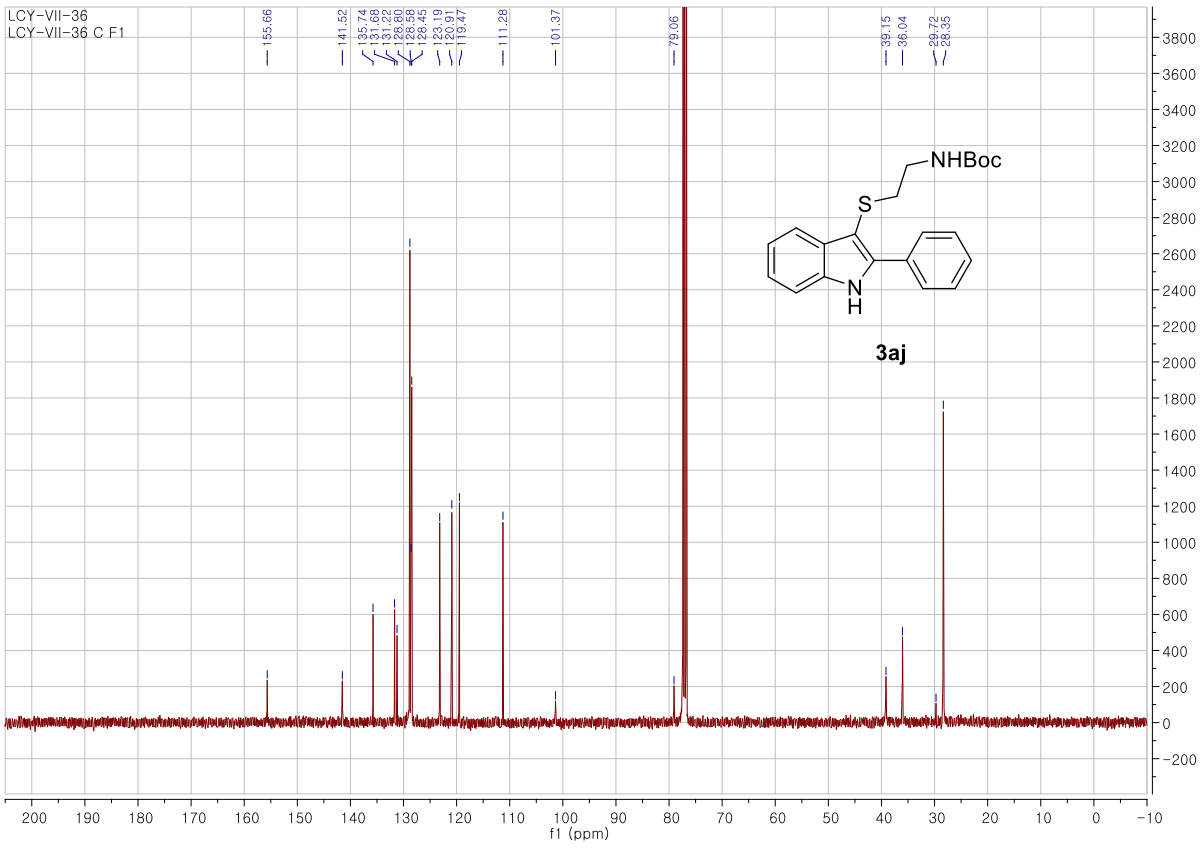

T102-1
89-6 nd
3ah
f1 (ppm)
Intensity

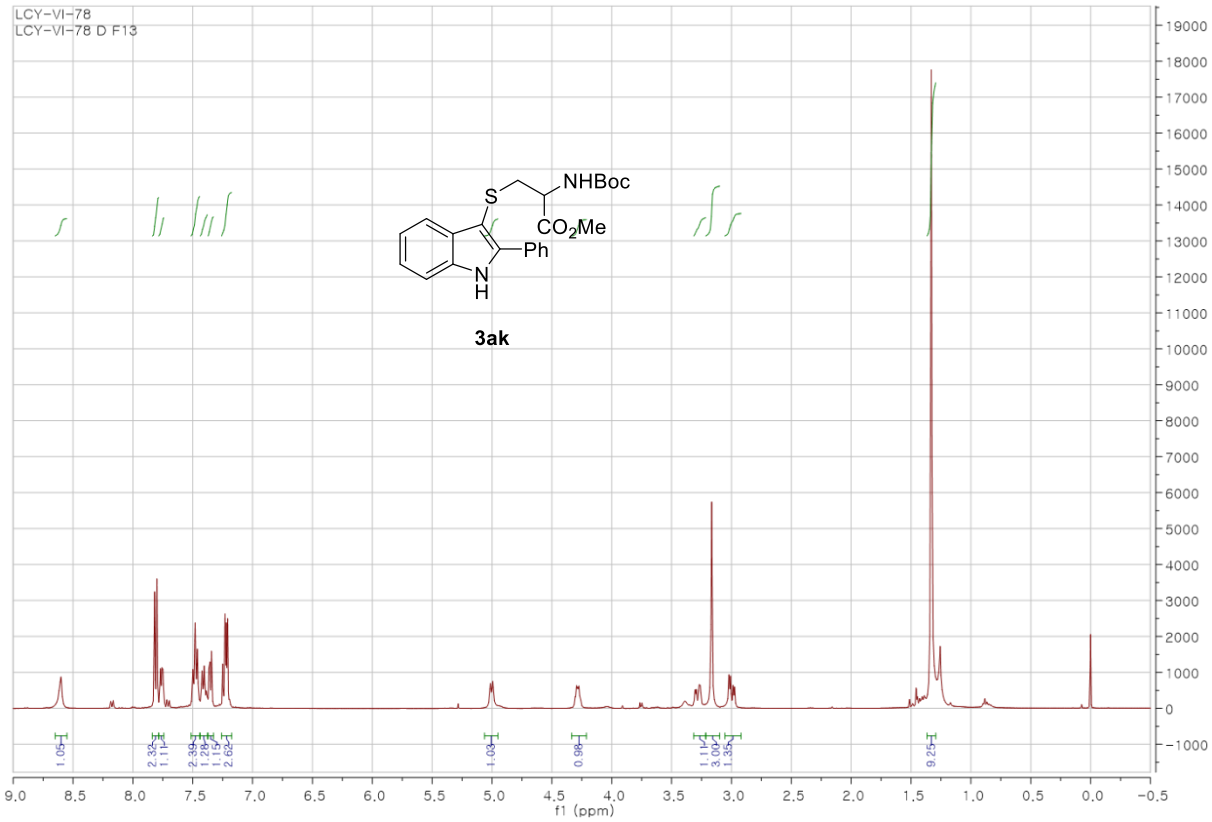

LCY-CB-L27
LCY-CB-L27
3ah
f1 (ppm)

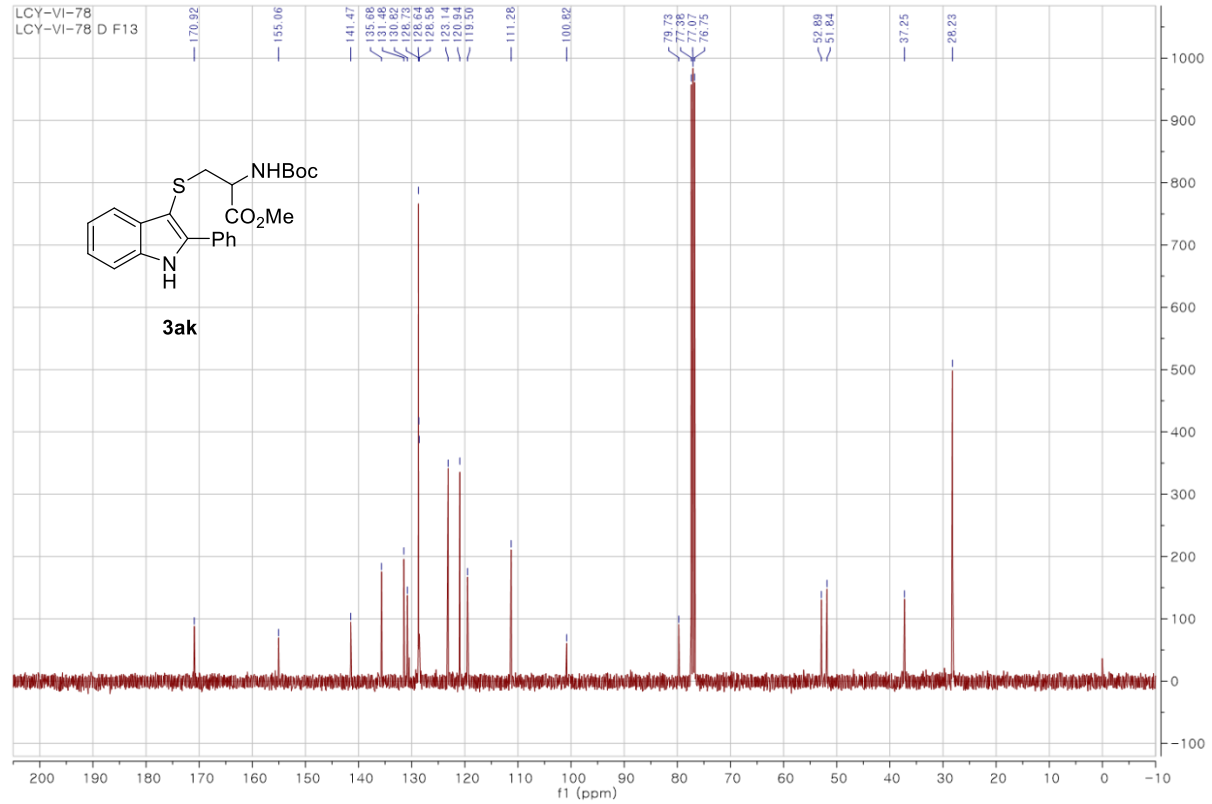

T102-1
127-3
S(CH2)8SH
Ph
N
H
3ai
f1 (ppm)

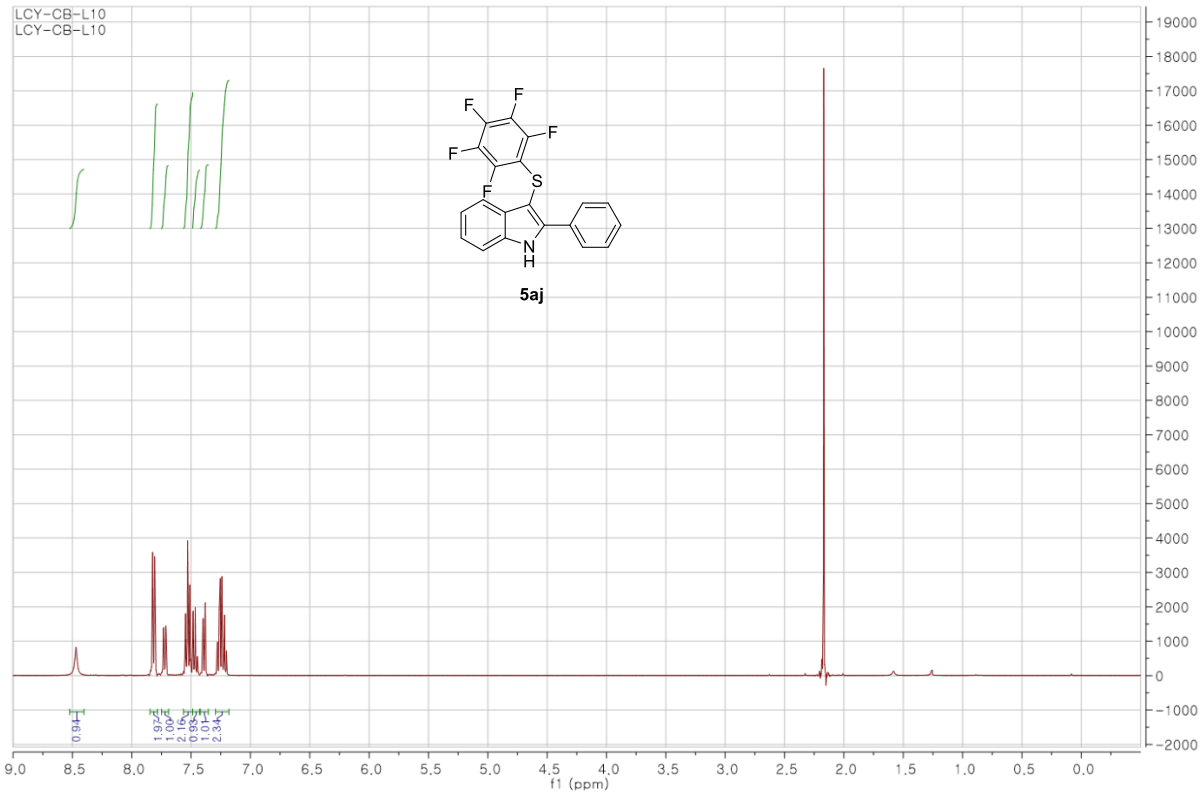

T102-1
127-2
140.62
135.63
132.14
131.61
128.63
128.31
128.26
123.00
120.66
119.85
111.04
103.54
36.38
33.98
29.47
29.01
28.86
28.40
28.25
24.62
S(CH2)8SH
Ph
N
H
3ai
f1 (ppm)

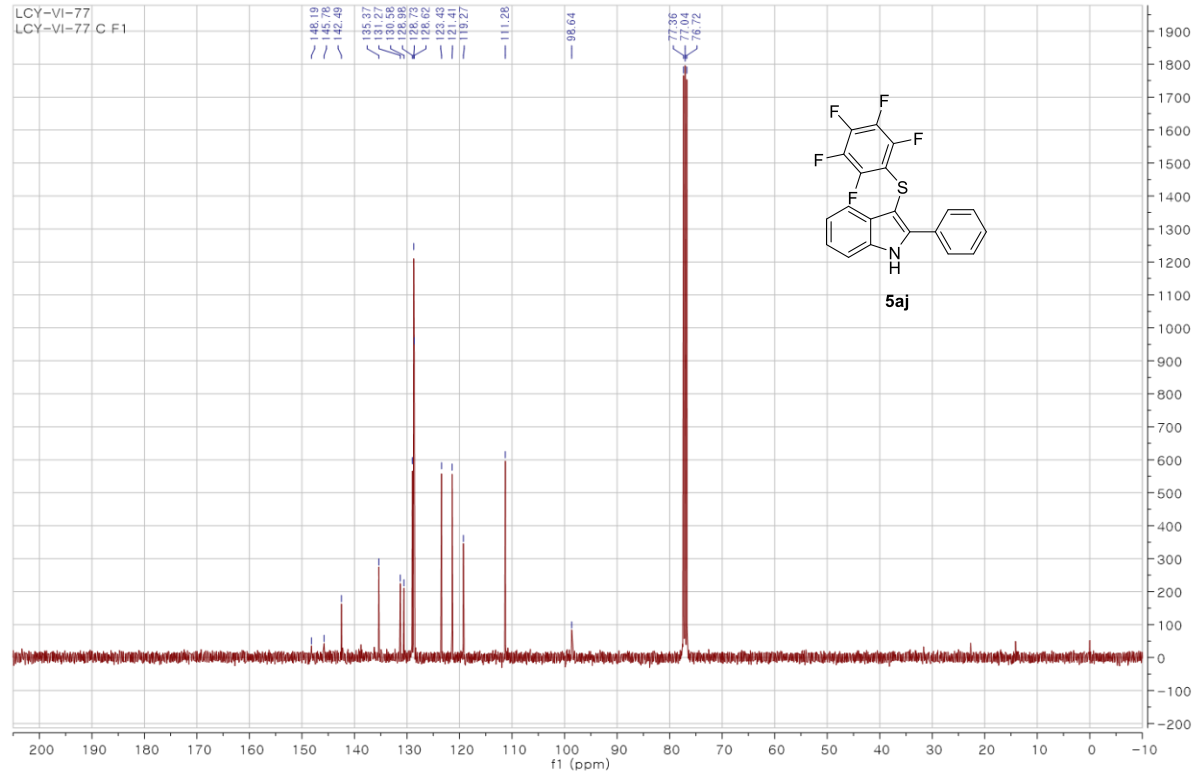

T102-8
91-1
NHBoc
S
N
H
3aj
Intensity
f1 (ppm)

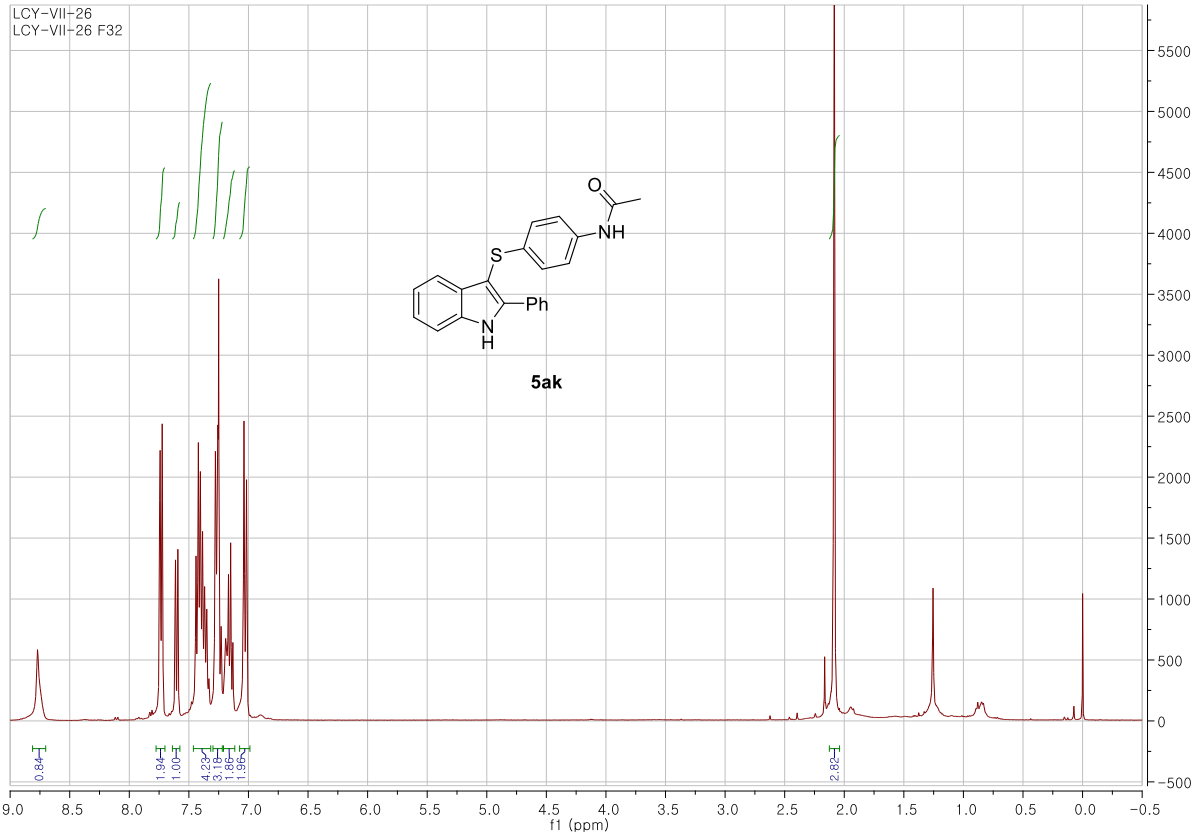

LCY-VII-36
LCY-VII-36 C F1
155.66
141.52
135.74
131.69
131.22
128.80
128.58
128.45
123.19
120.91
119.47
111.28
101.37
79.06
39.15
36.04
29.72
28.35
NHBoc
S
N
H
3aj
f1 (ppm)

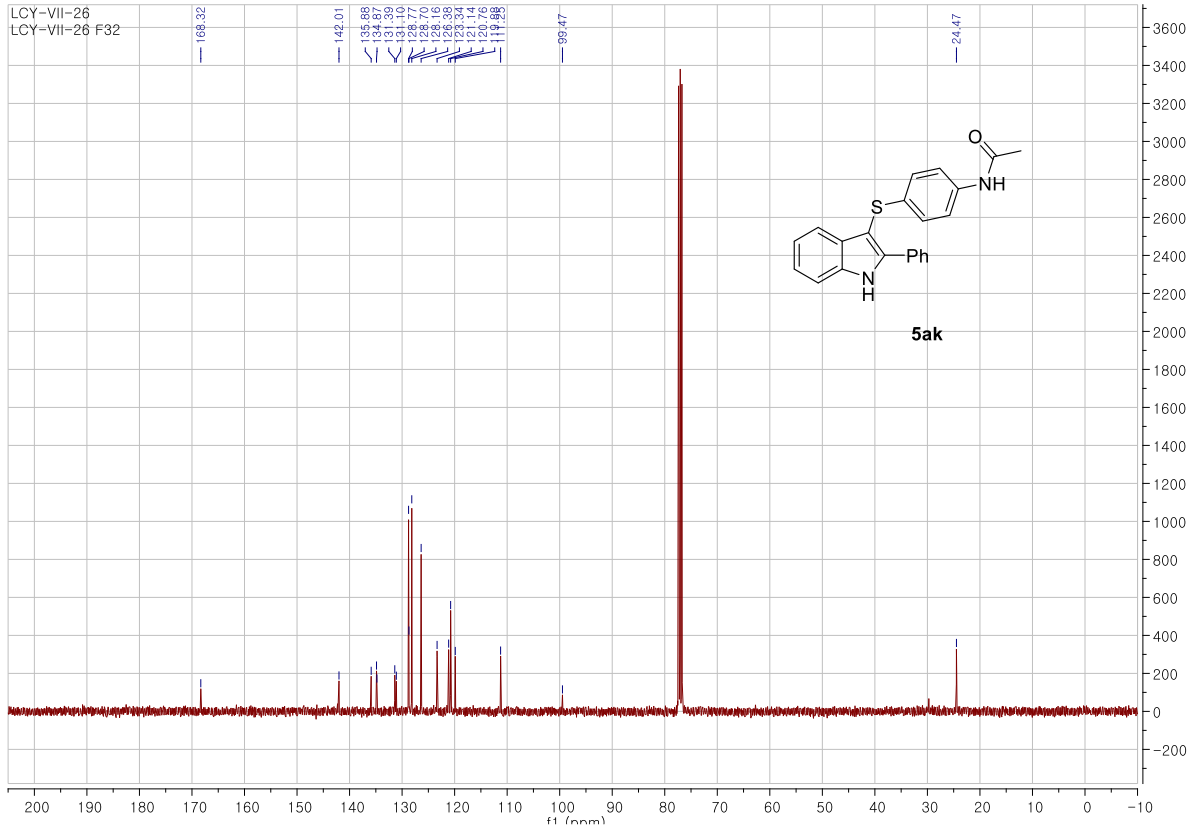

LCY-VI-78
LCY-VI-78 D F13
NHBoc
S
CO2Me
Ph
N
H
3ak
f1 (ppm)

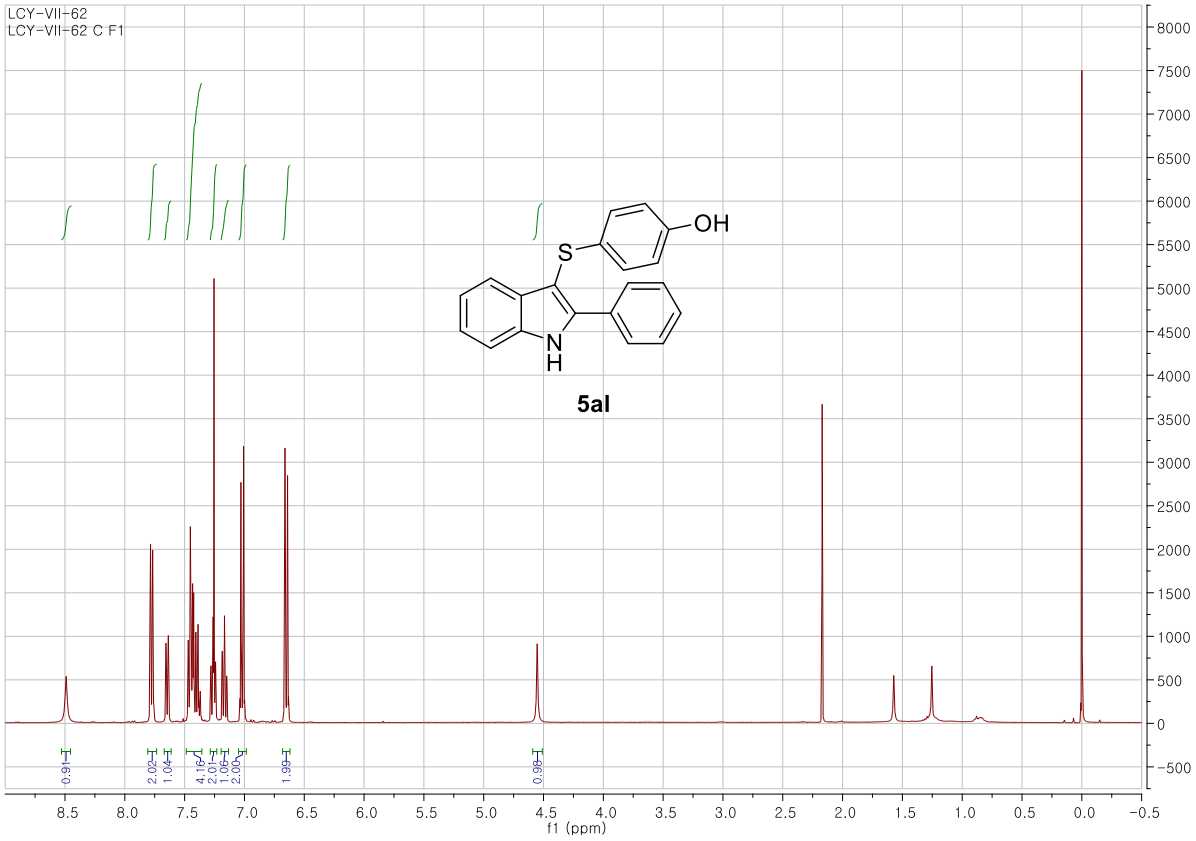

LCY-VI-78
LCY-VI-78 D F13
170.92
155.06
141.47
135.68
131.48
130.82
128.73
128.64
126.58
123.14
120.94
119.50
111.28
100.82
79.73
77.38
77.17
76.75
52.88
51.84
37.25
28.23
NHBoc
S
CO2Me
Ph
N
H
3ak
f1 (ppm)

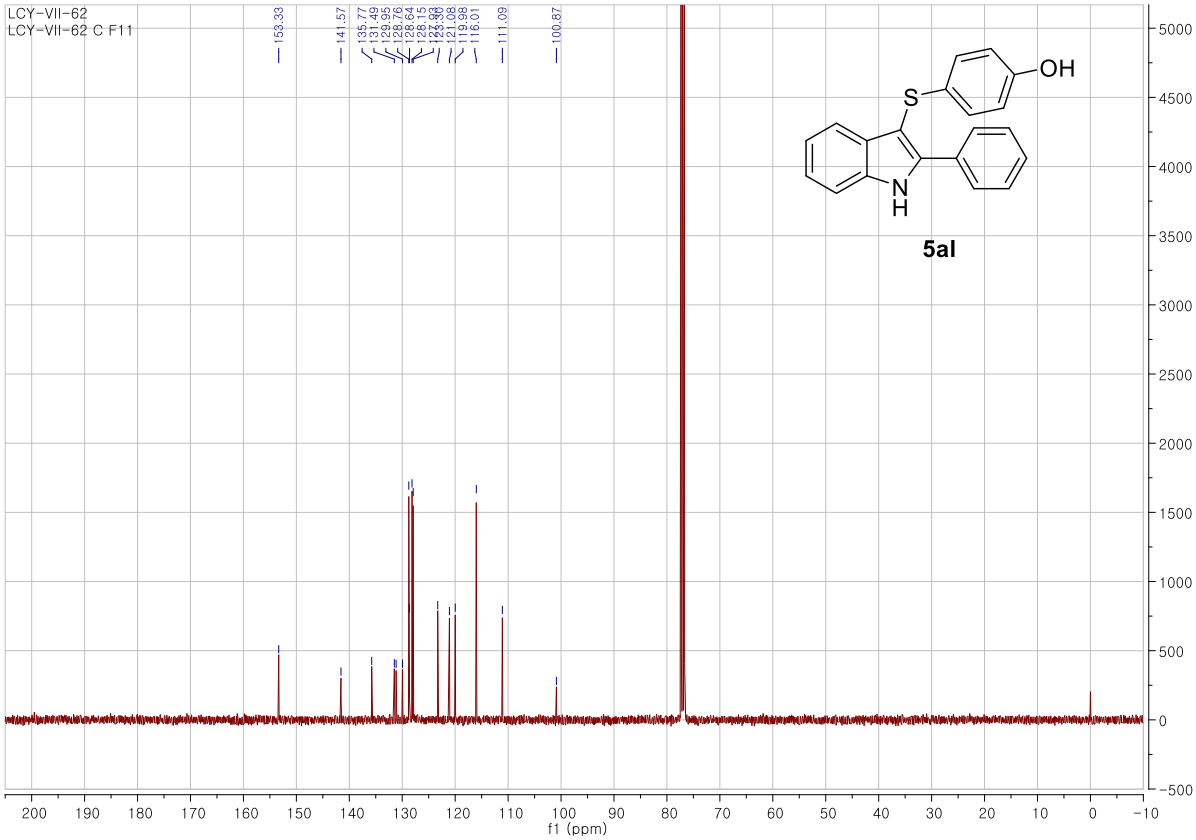

LCY-CB-L10
LCY-CB-L10
F
F
F
F
F
S
N
H
5aj
f1 (ppm)

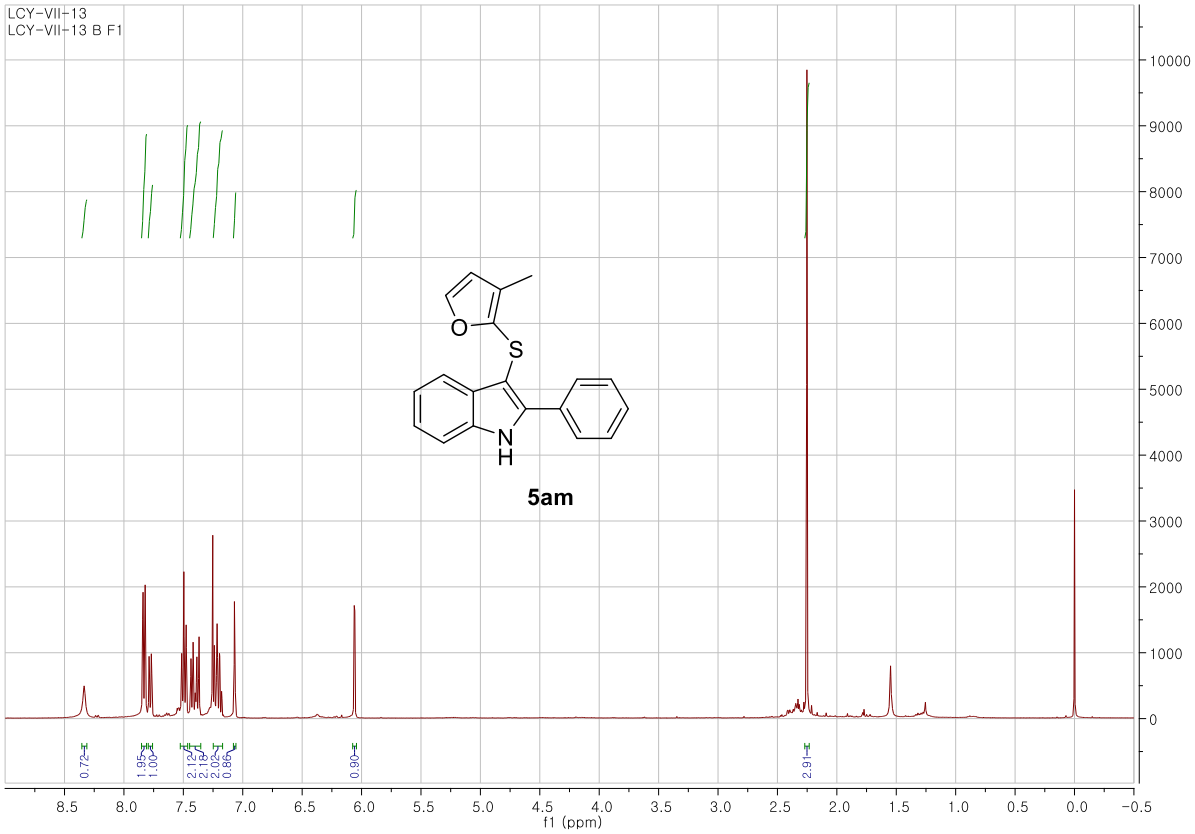

LCY-VI-77
LCY-VI-77 C F1
148.19
145.78
142.49
135.37
131.27
130.58
128.98
128.73
128.62
123.48
123.41
119.27
111.28
98.64
77.36
77.04
76.72
F
F
F
F
F
S
N
H
5aj
f1 (ppm)

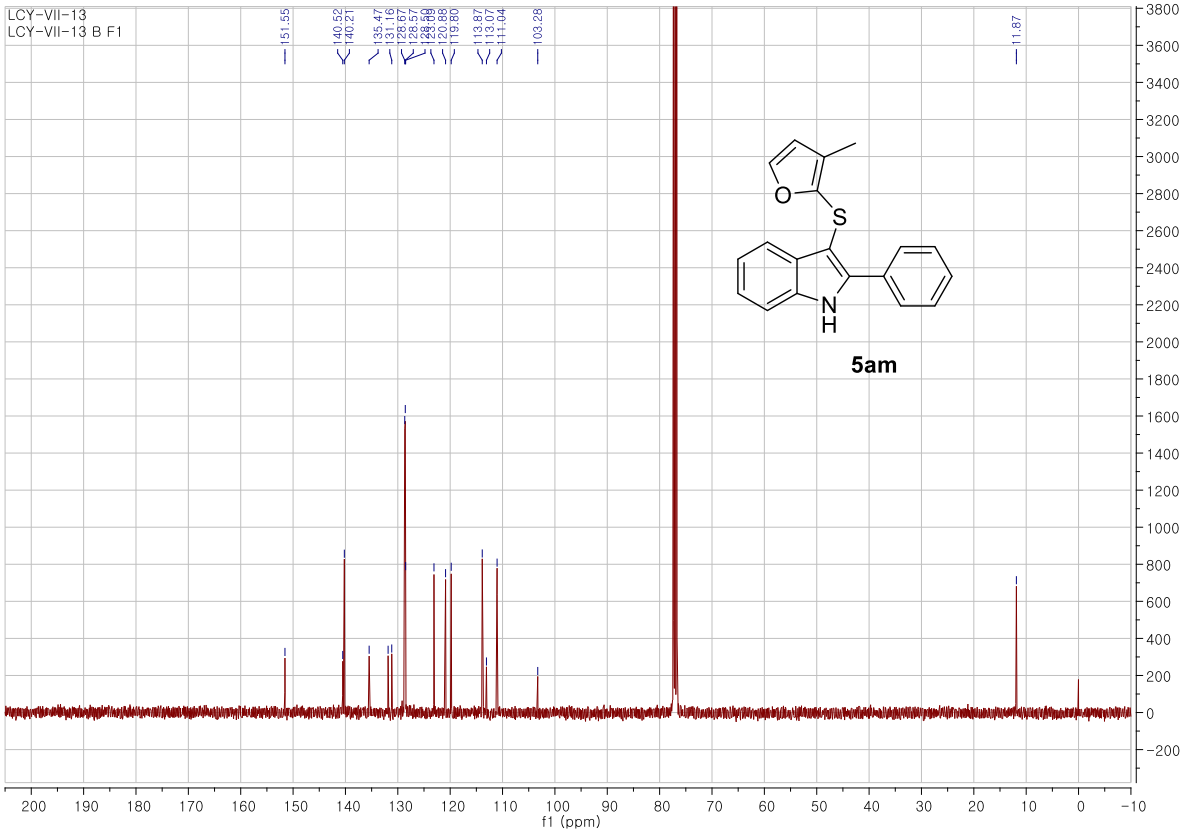

LCY-VII-26
LCY-VII-26 F32
O
NH
S
Ph
N
H
5ak
f1 (ppm)

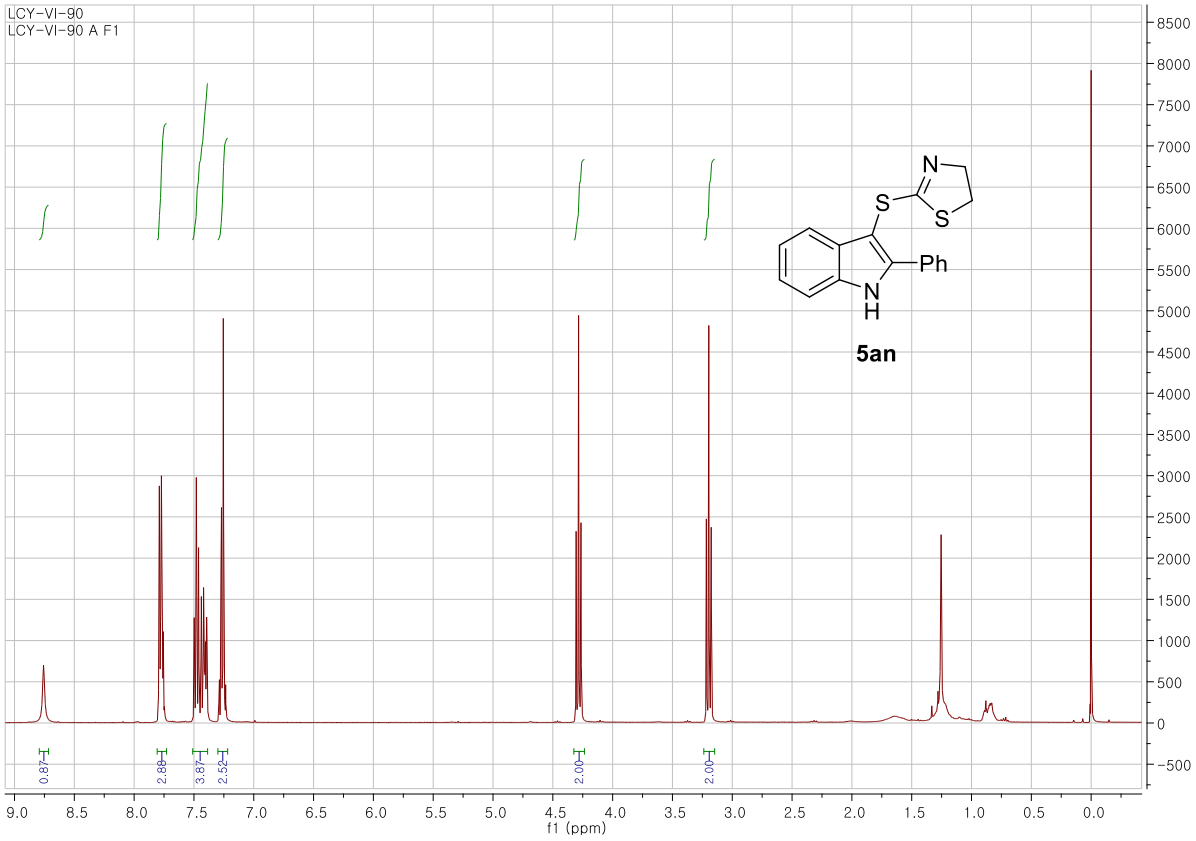

LCY-VII-26
LCY-VII-26 F32
168.32
142.01
135.88
134.87
131.39
131.10
128.77
128.70
128.16
126.38
123.34
121.14
120.76
119.88
99.47
24.47
O
NH
S
Ph
N
H
5ak
f1 (ppm)

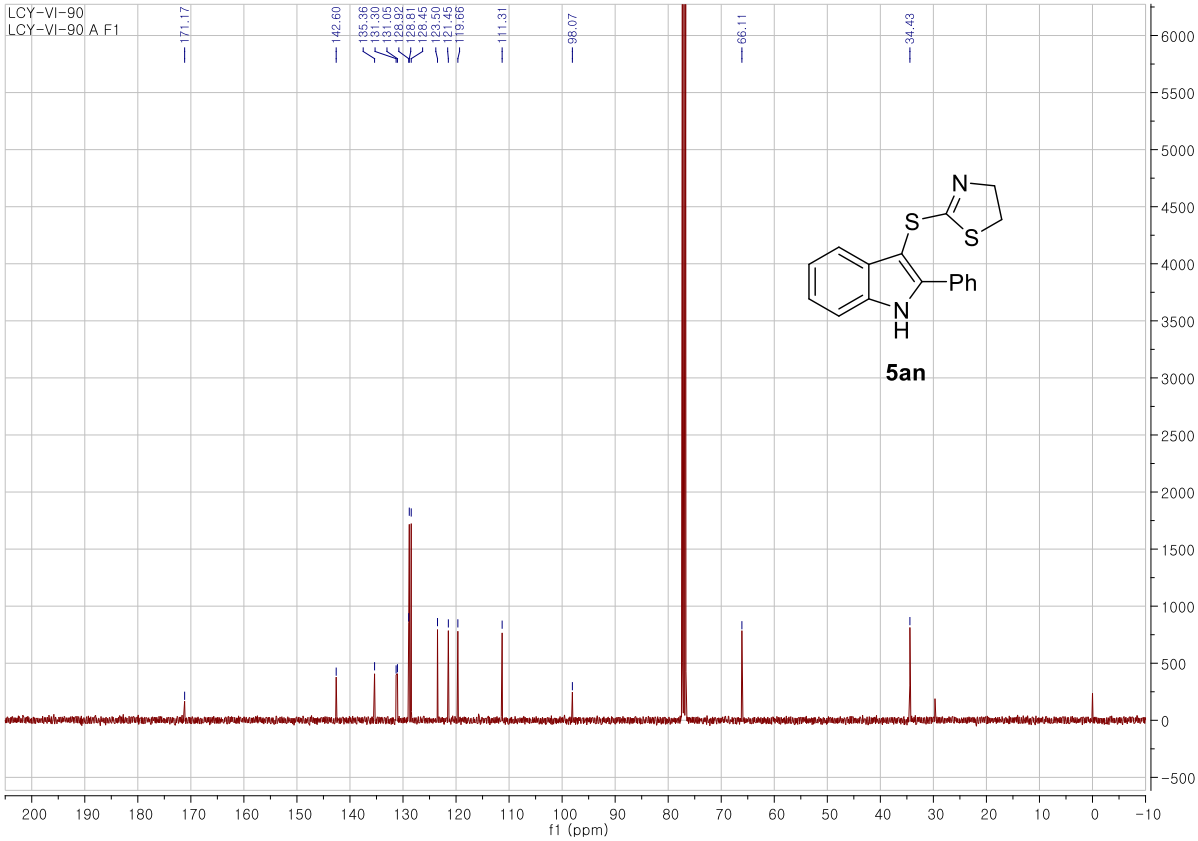

LCY-VII-62
LCY-VII-62 C F1
OH
S
N
H
5al
f1 (ppm)

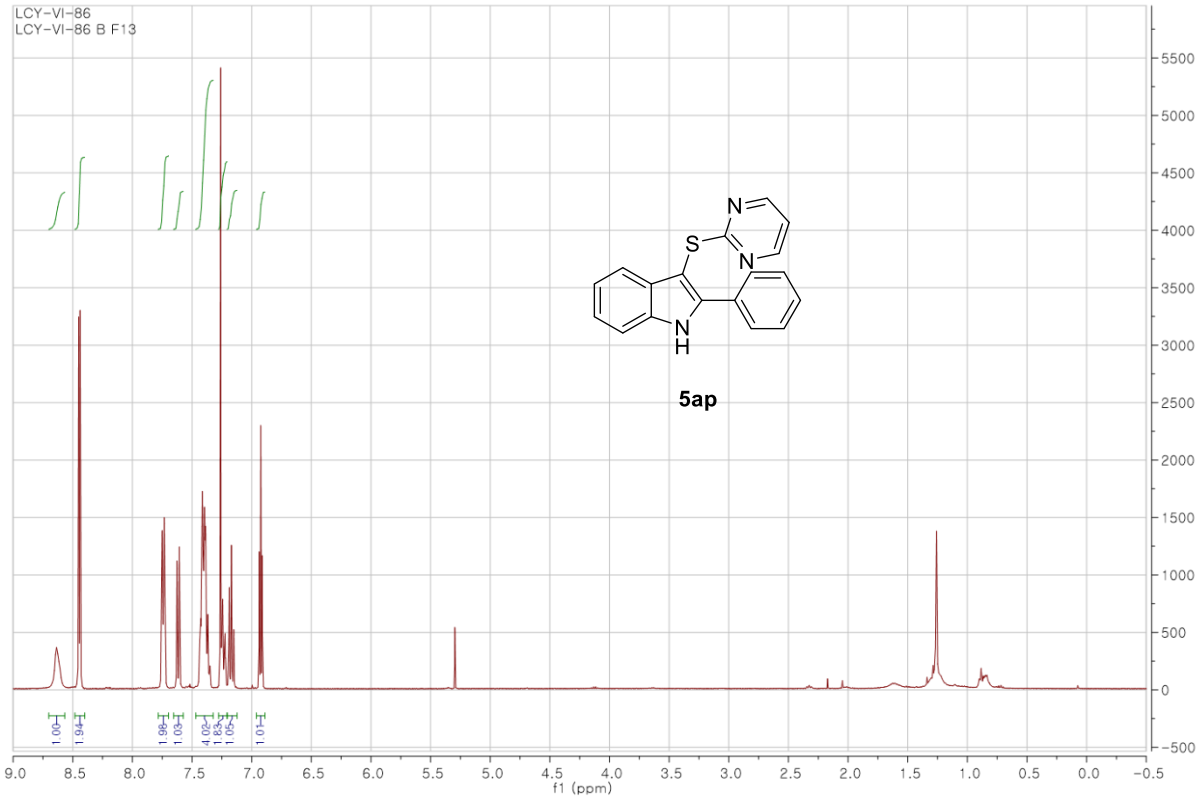

LCY-VII-62
LCY-VII-62 C F11
158.33
141.57
135.77
131.49
129.95
128.76
128.64
126.15
123.30
121.08
119.98
116.01
111.09
100.87
OH
S
N
H
5al
f1 (ppm)

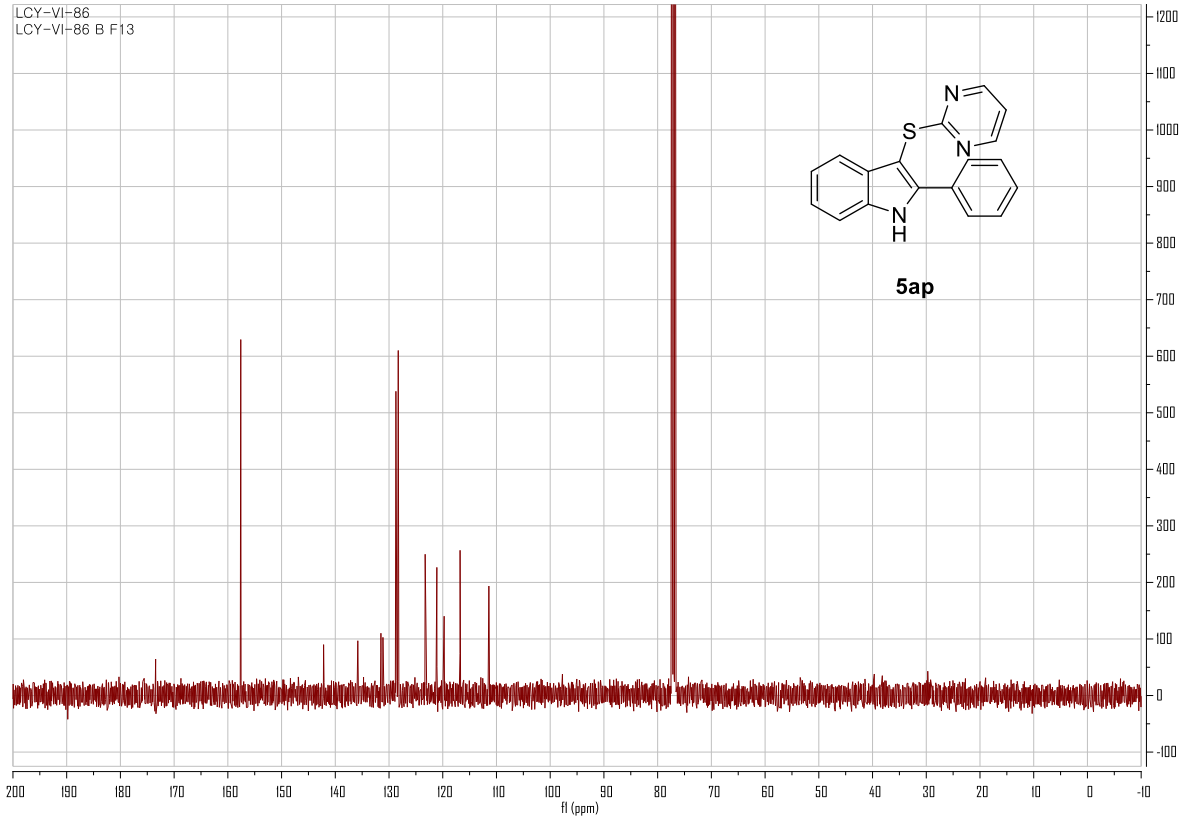

LCY-VII-13
LCY-VII-13 B F1
5am
f1 (ppm)

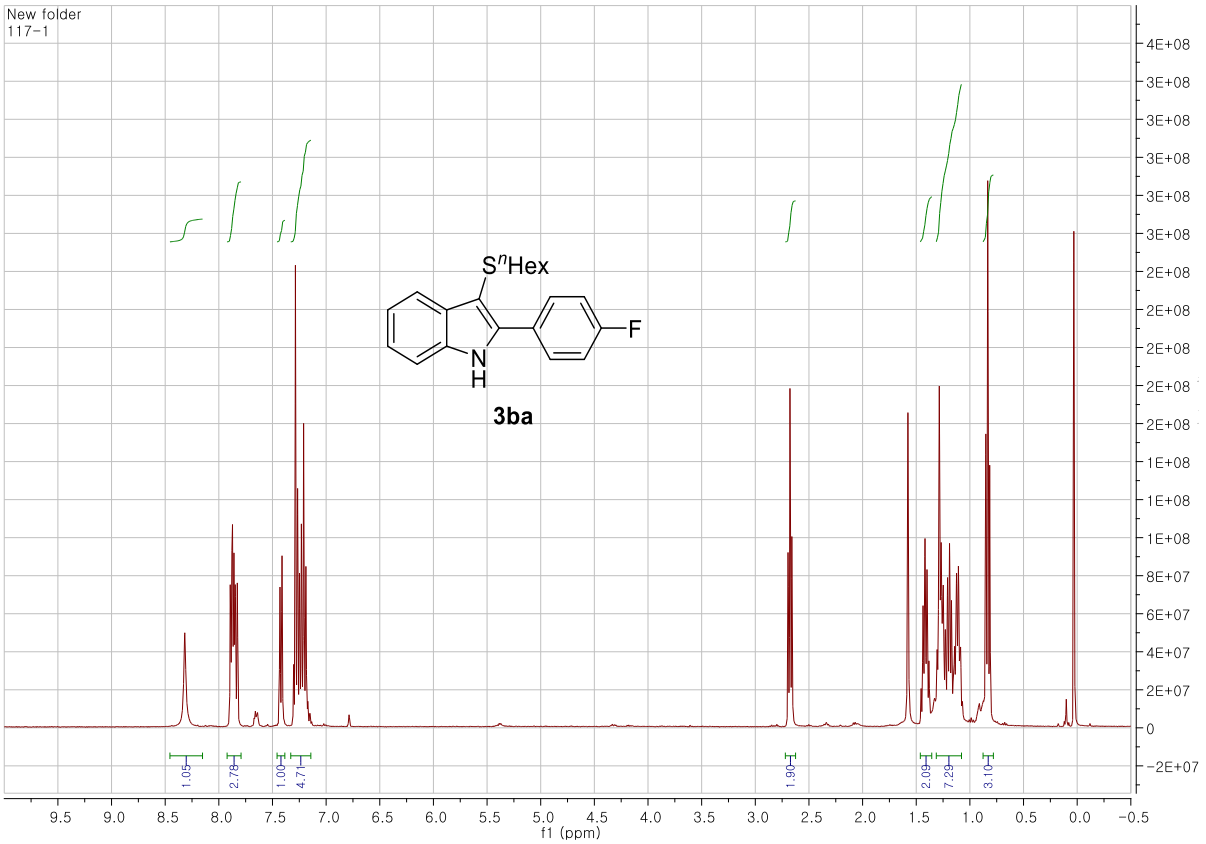

LCY-VII-13
LCY-VII-13 B F1
151.55
140.52
140.21
135.47
131.15
128.67
128.57
120.88
119.80
113.87
113.07
111.94
103.28
11.87
5am
f1 (ppm)

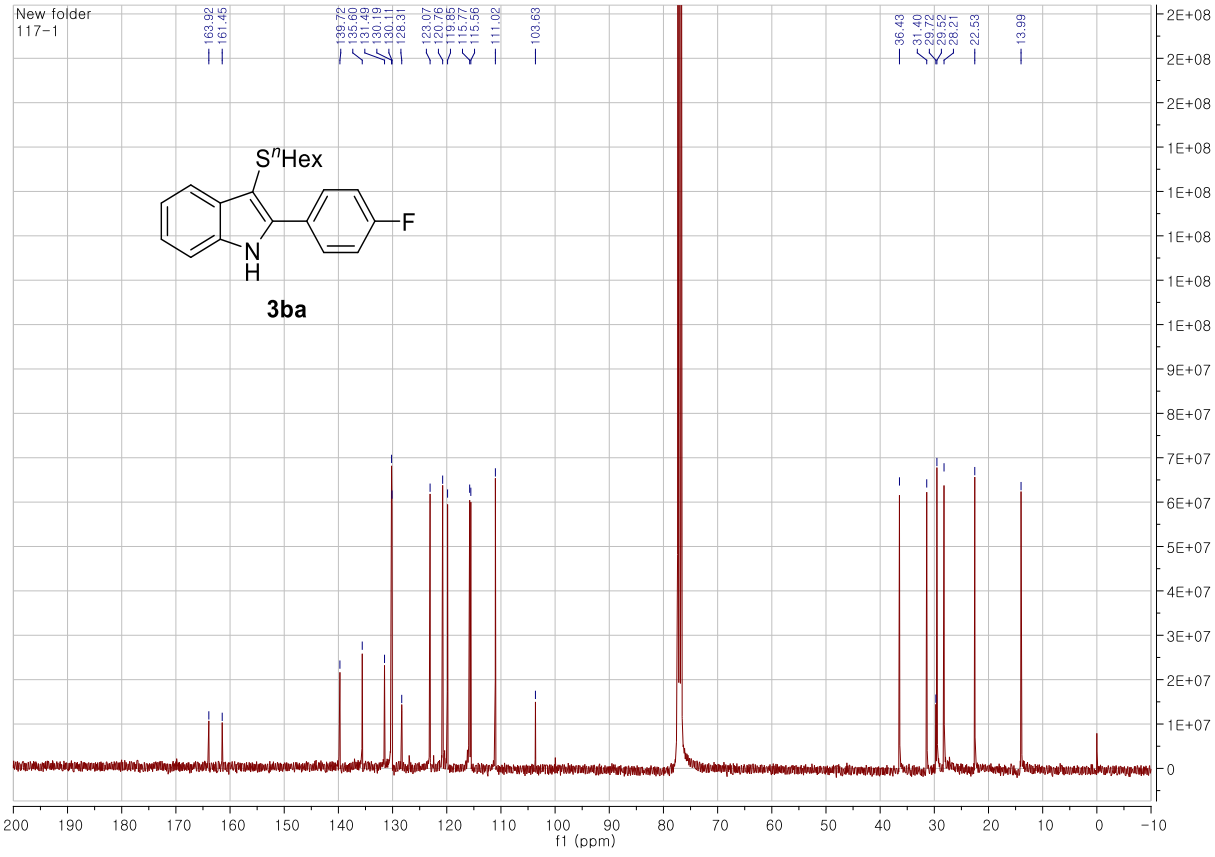

LCY-VI-90
LCY-VI-90 A F1
N
S
S
Ph
N
H
5an
0.87
2.88
3.87
2.52
2.00
2.00
f1 (ppm)

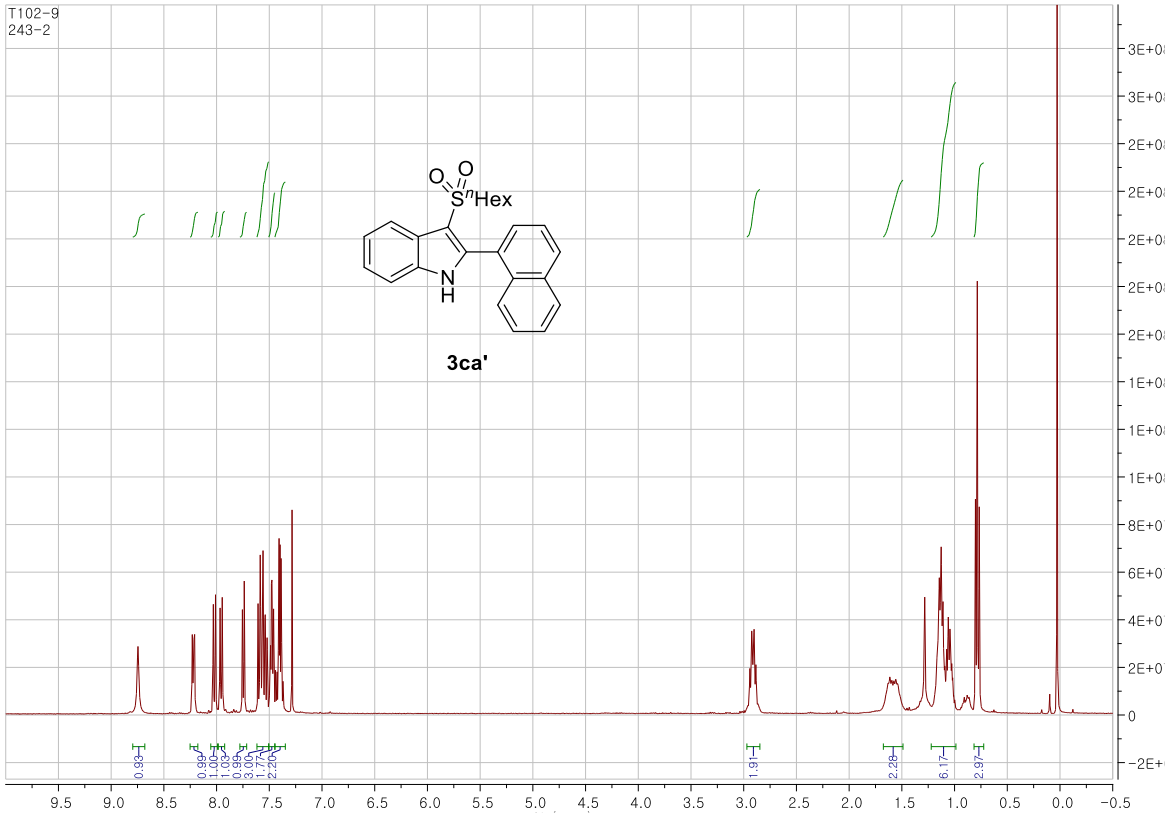

LCY-VI-90
LCY-VI-90 A F1
171.17
142.60
135.36
131.30
131.05
128.92
128.81
128.45
123.50
121.45
119.66
111.31
98.07
66.11
34.43
N
S
S
Ph
N
H
5an
f1 (ppm)

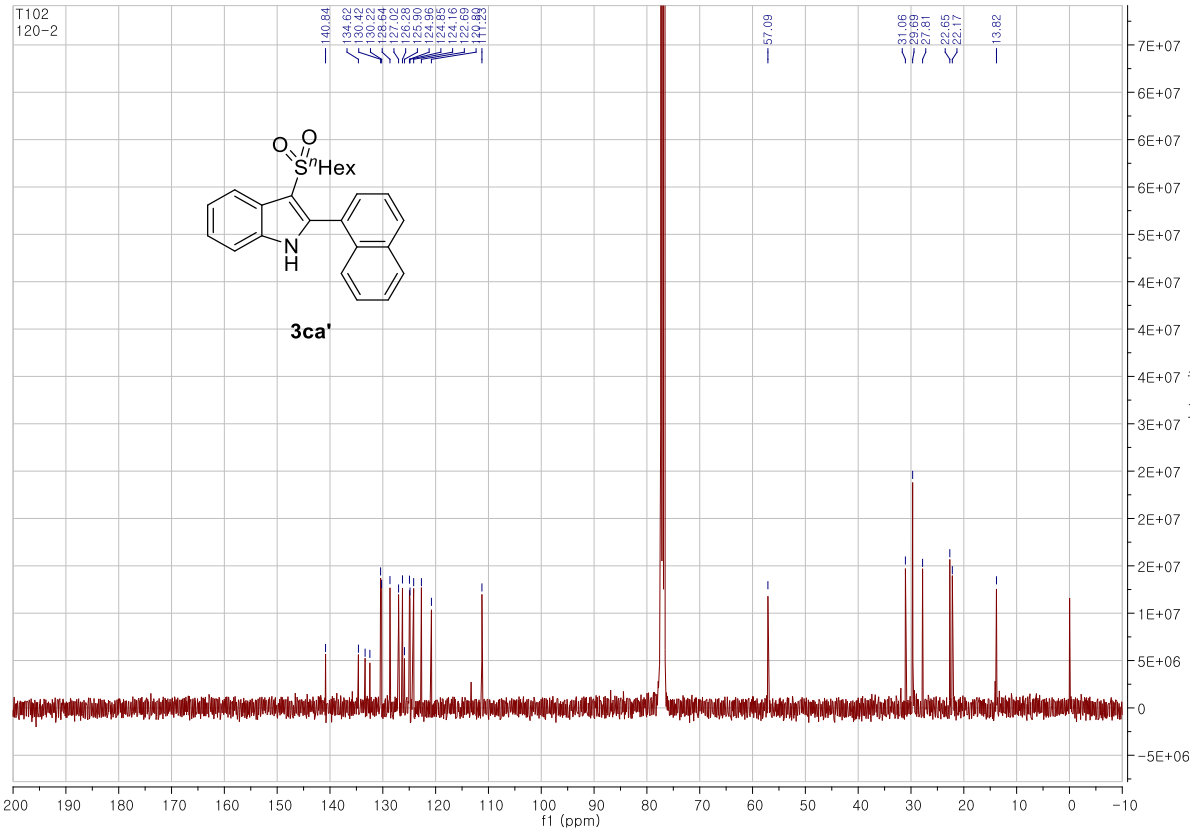

LCY-VI-86
LCY-VI-86 B F13
5ap
f1 (ppm)

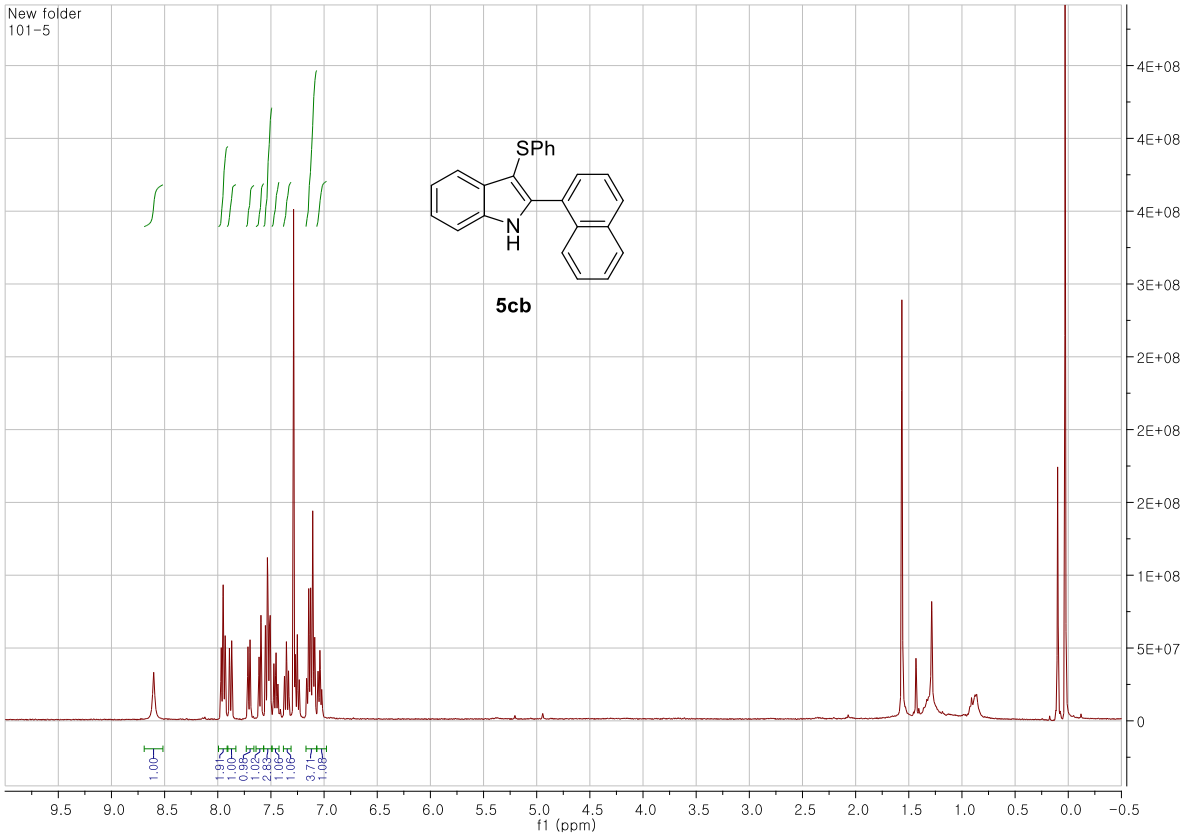

LCY-VI-86
LCY-VI-86 B F13
5ap
f1 (ppm)

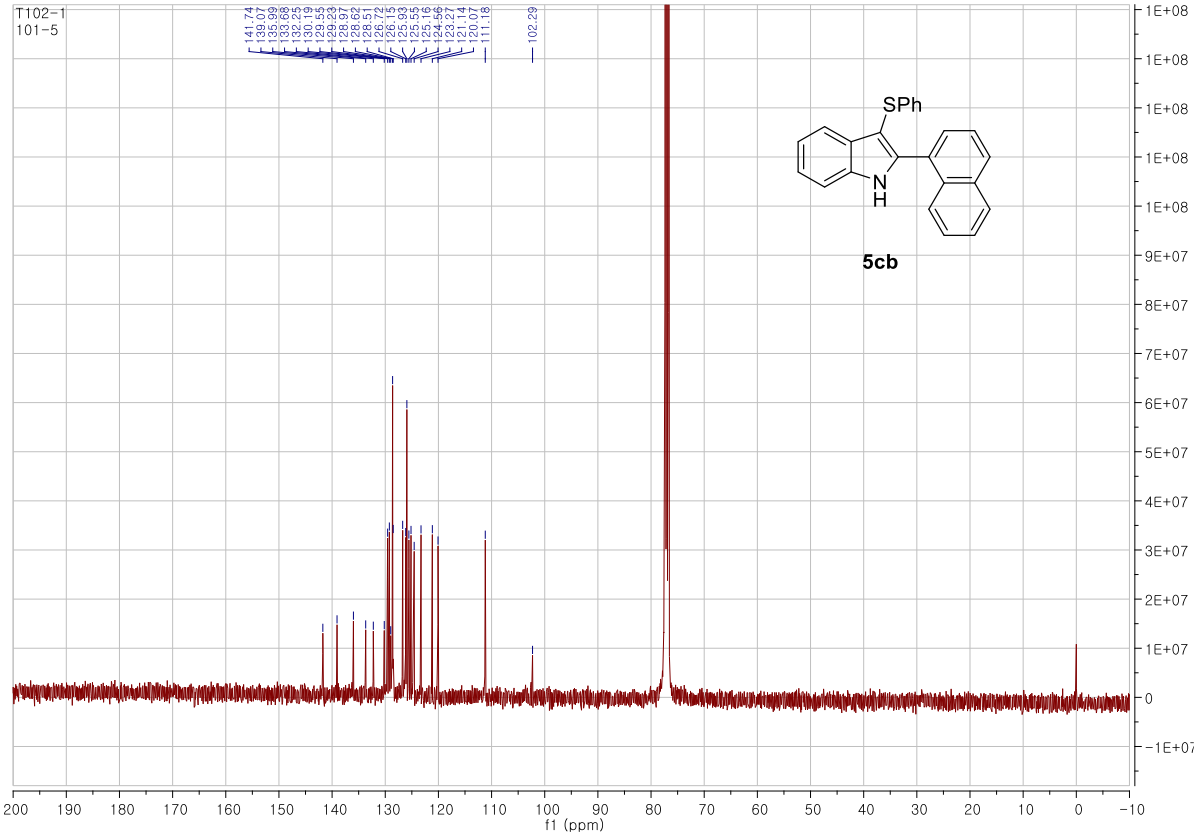

New folder
117-1
S^{n}Hex
F
N
H
3ba
1.05
2.78
1.00
4.71
1.90
2.09
7.29
3.10
f1 (ppm)

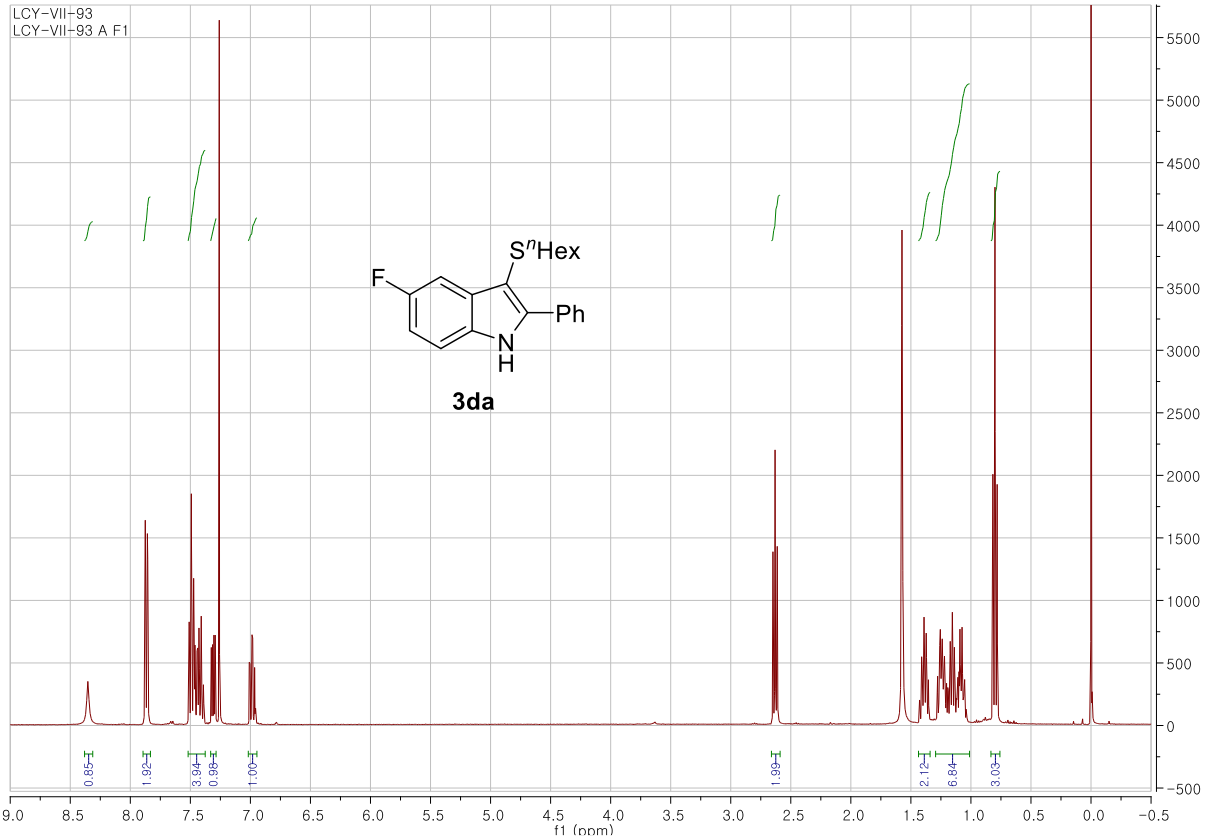

New folder
117-1
163.92
161.45
139.72
135.60
131.49
130.19
130.11
128.31
123.07
120.76
119.85
115.77
115.56
111.02
103.63
36.43
31.40
29.52
28.21
22.53
13.99
S^{n}Hex
F
N
H
3ba
f1 (ppm)

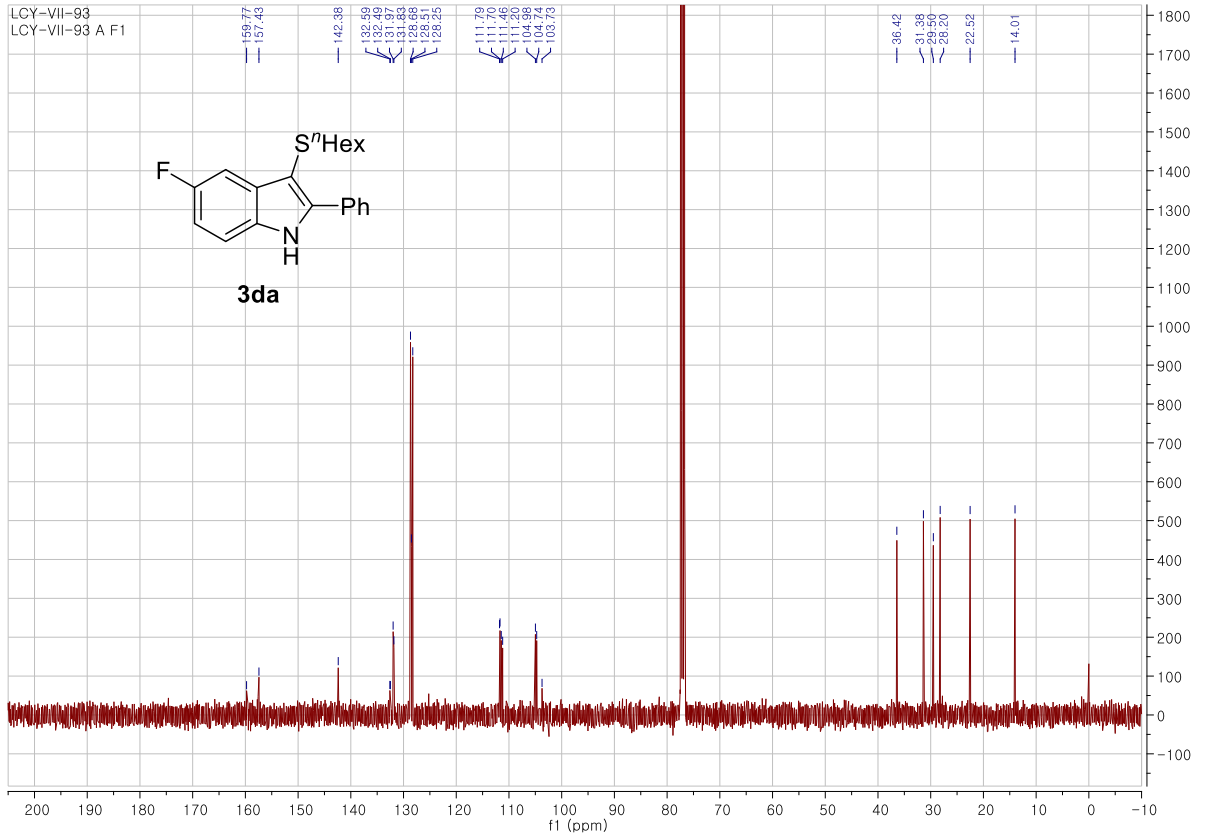

T102-9
243-2
O
O
S
nHex
N
H
3ca'

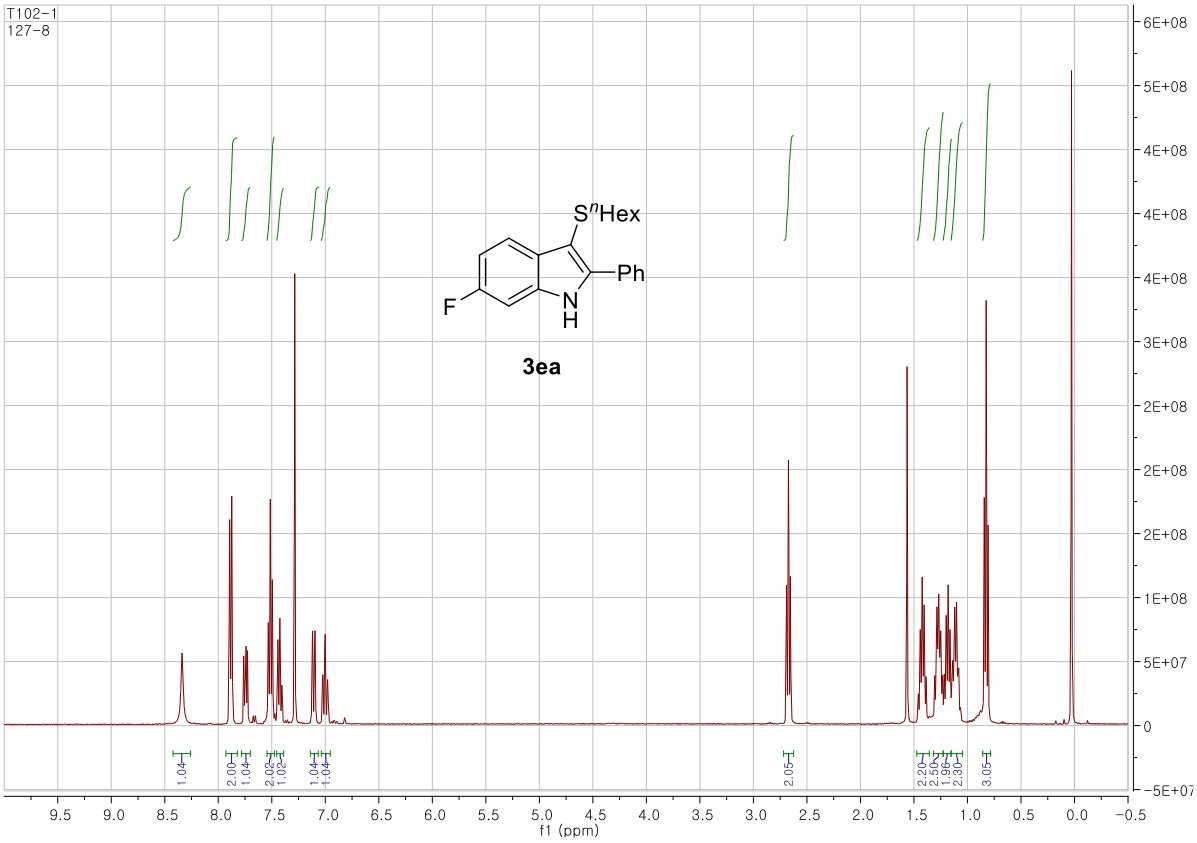

T102
120-2
140.84
134.62
130.42
128.22
127.64
127.02
126.28
125.90
124.96
124.85
124.11
122.69
120.89
111.23
57.09
31.06
29.69
27.81
22.65
22.17
13.82
O
O
S
nHex
N
H
3ca'
f1 (ppm)

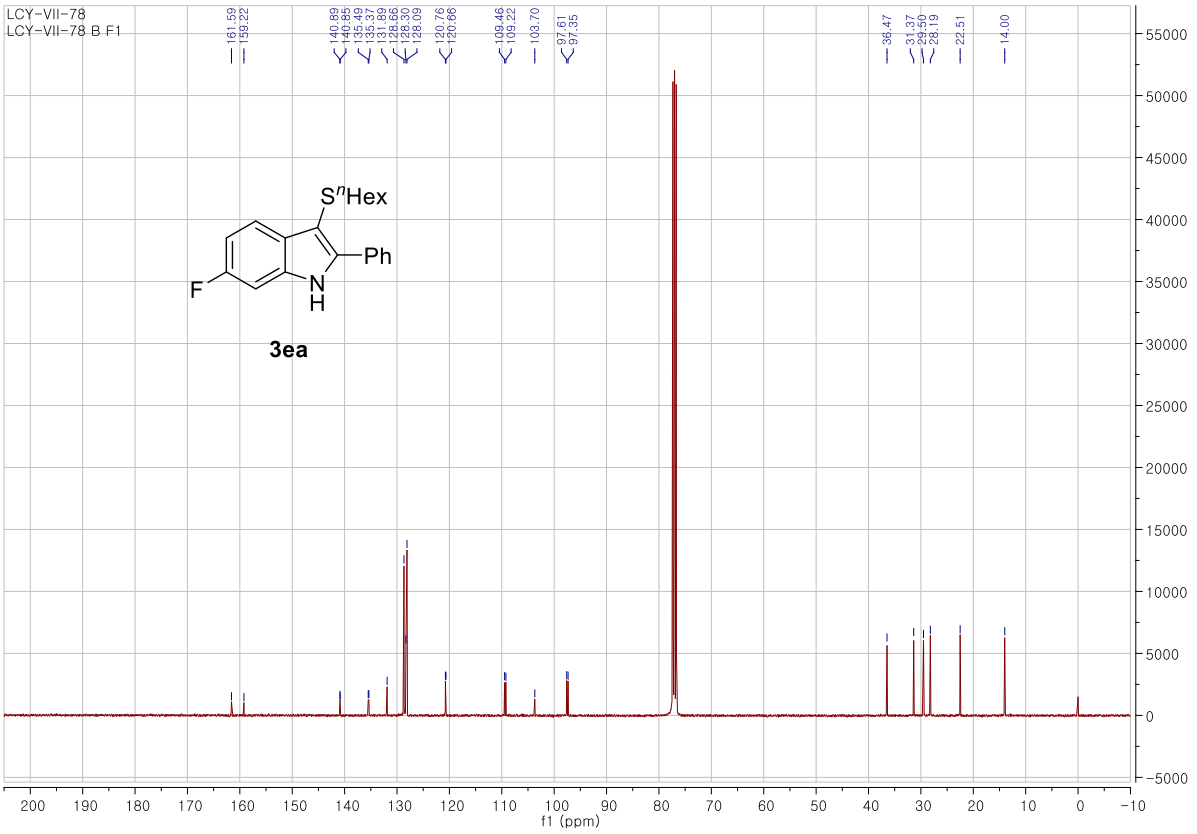

New folder
101-5
SPh
N
H
5cb
f1 (ppm)

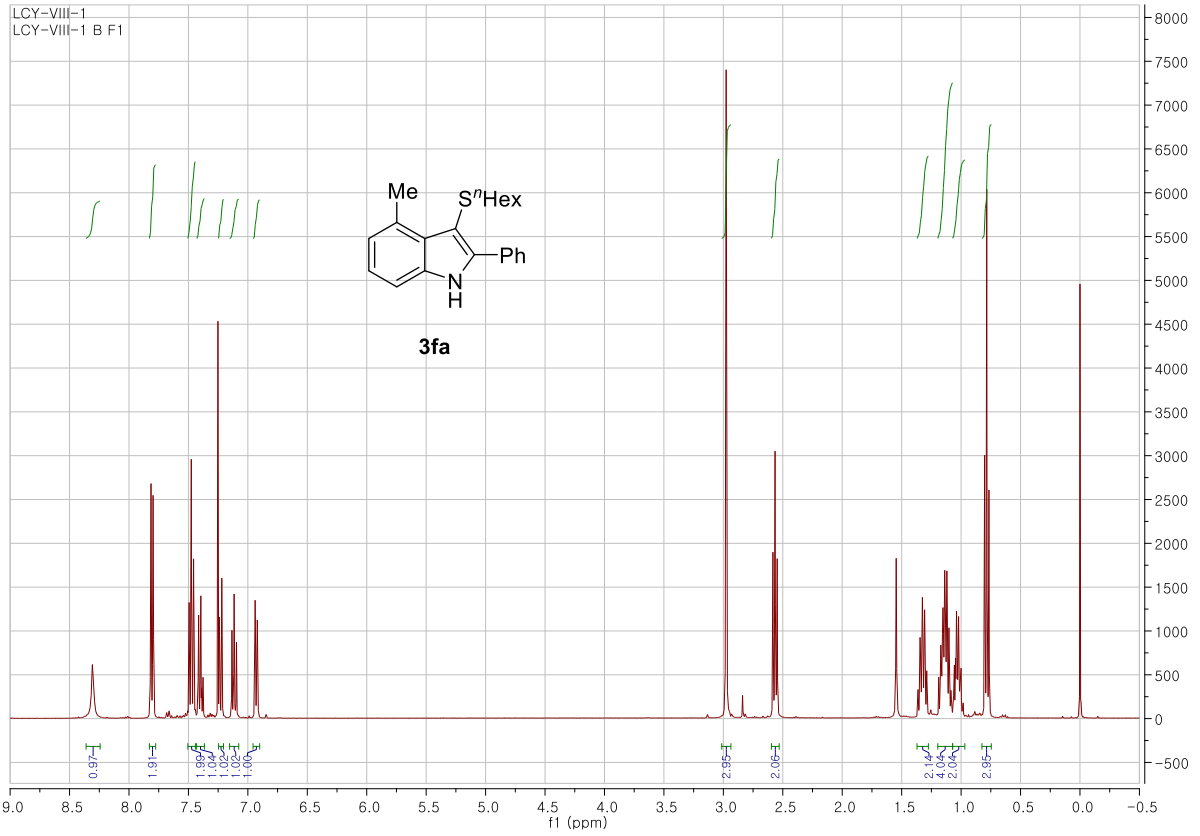

T102-1
101-5
141.74
139.07
135.99
133.86
132.25
130.19
129.55
129.23
128.97
128.62
128.51
126.72
126.15
125.93
125.55
125.15
124.66
123.27
121.14
120.07
111.18
102.29
SPh
N
H
5cb
f1 (ppm)

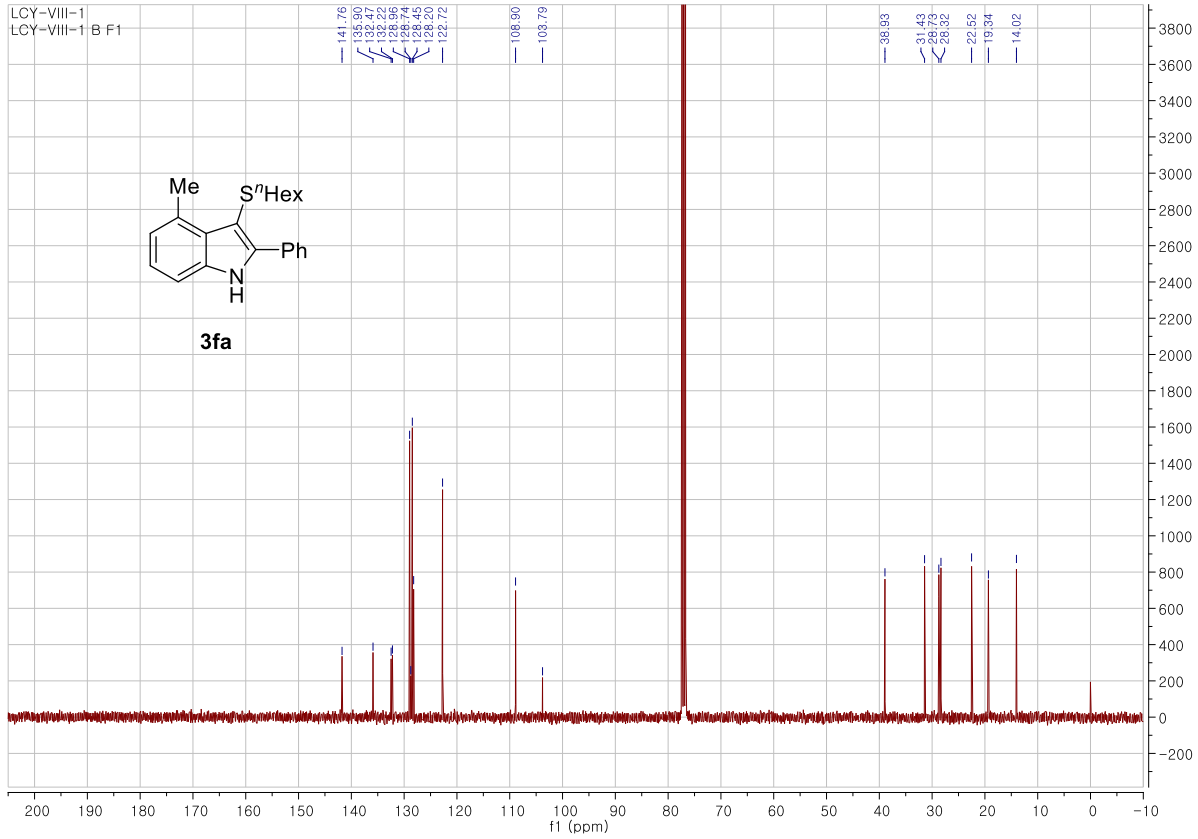

LCY-VII-93
LCY-VII-93 A F1
S^{n}Hex
F
Ph
N
H
3da
0.85
1.92
3.94
0.99
1.00
1.99
2.12
6.84
3.03
f1 (ppm)

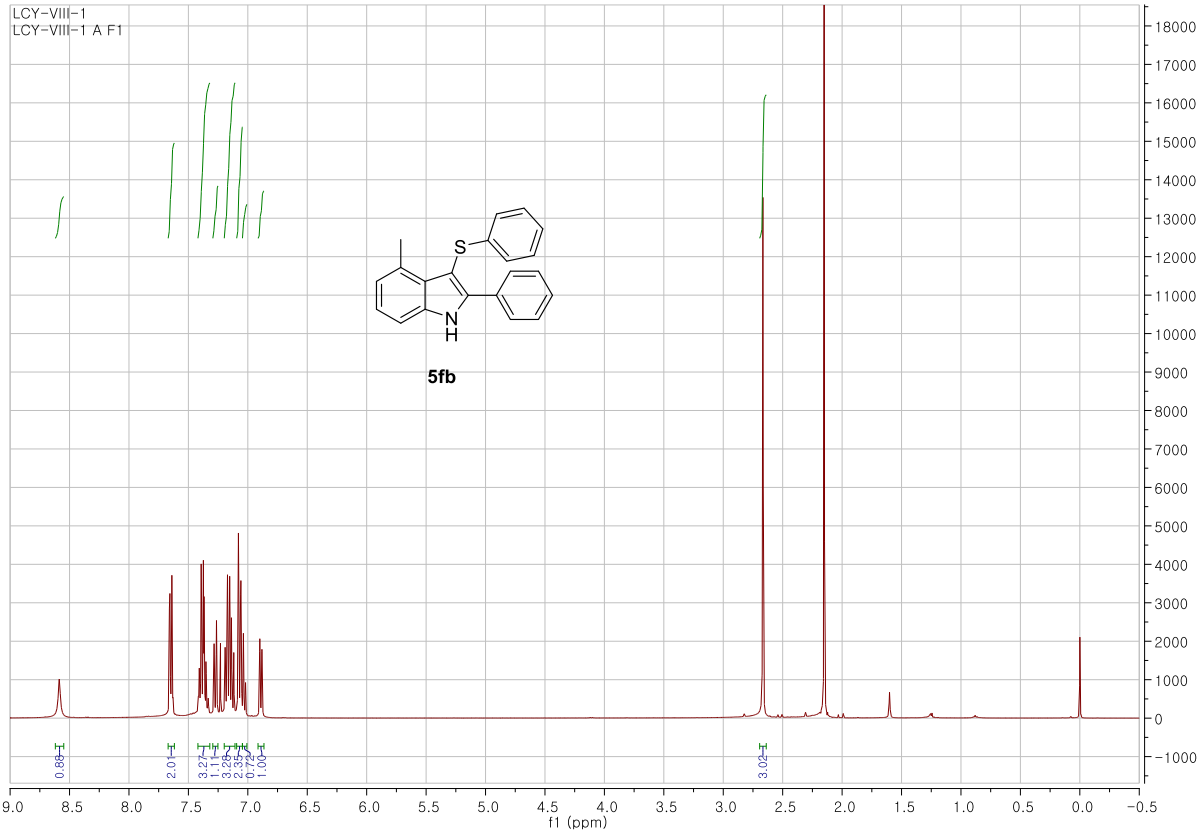

LCY-VII-93
LCY-VII-93 A F1
159.77
157.43
142.38
132.59
132.49
131.97
131.83
128.68
128.51
128.25
111.79
111.70
111.46
111.20
104.98
104.74
103.73
36.42
31.38
29.50
28.20
22.52
14.01
S^{n}Hex
F
Ph
N
H
3da
f1 (ppm)

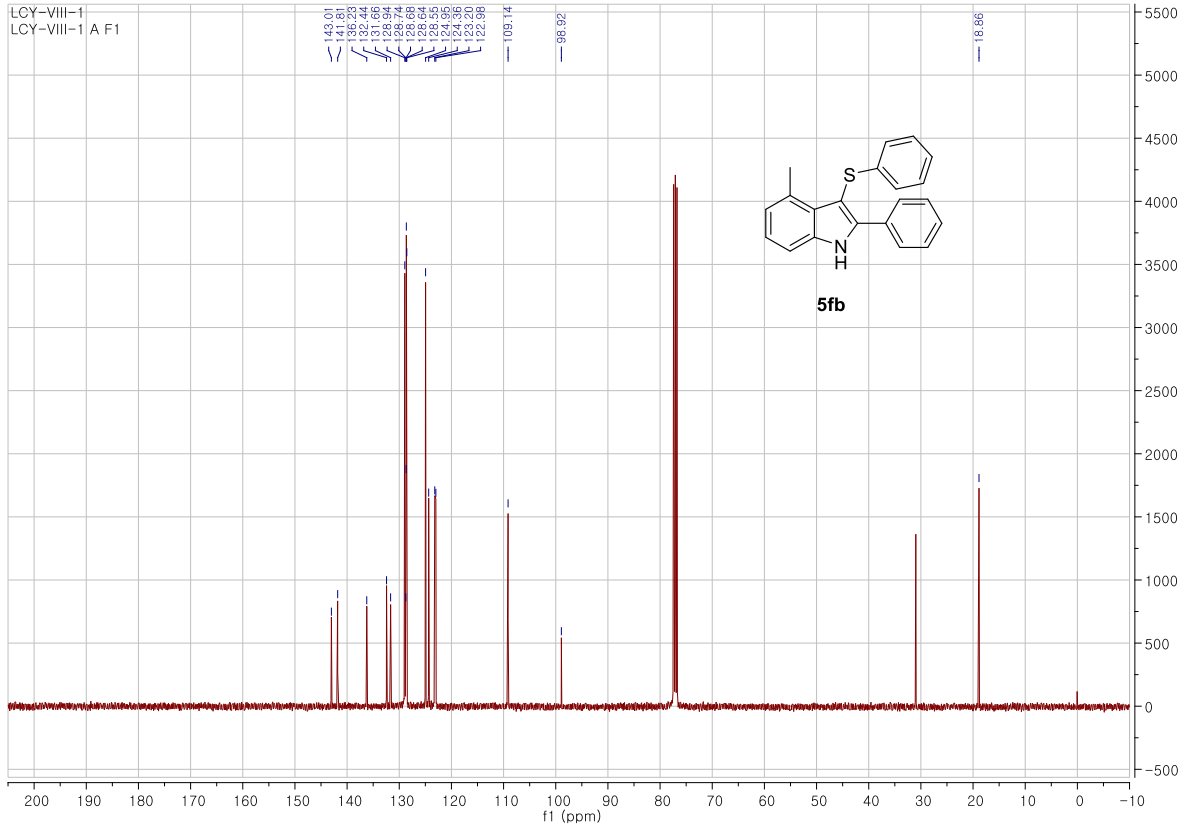

T102-1
127-8
S^{n}Hex
Ph
F
N
H
3ea
f1 (ppm)

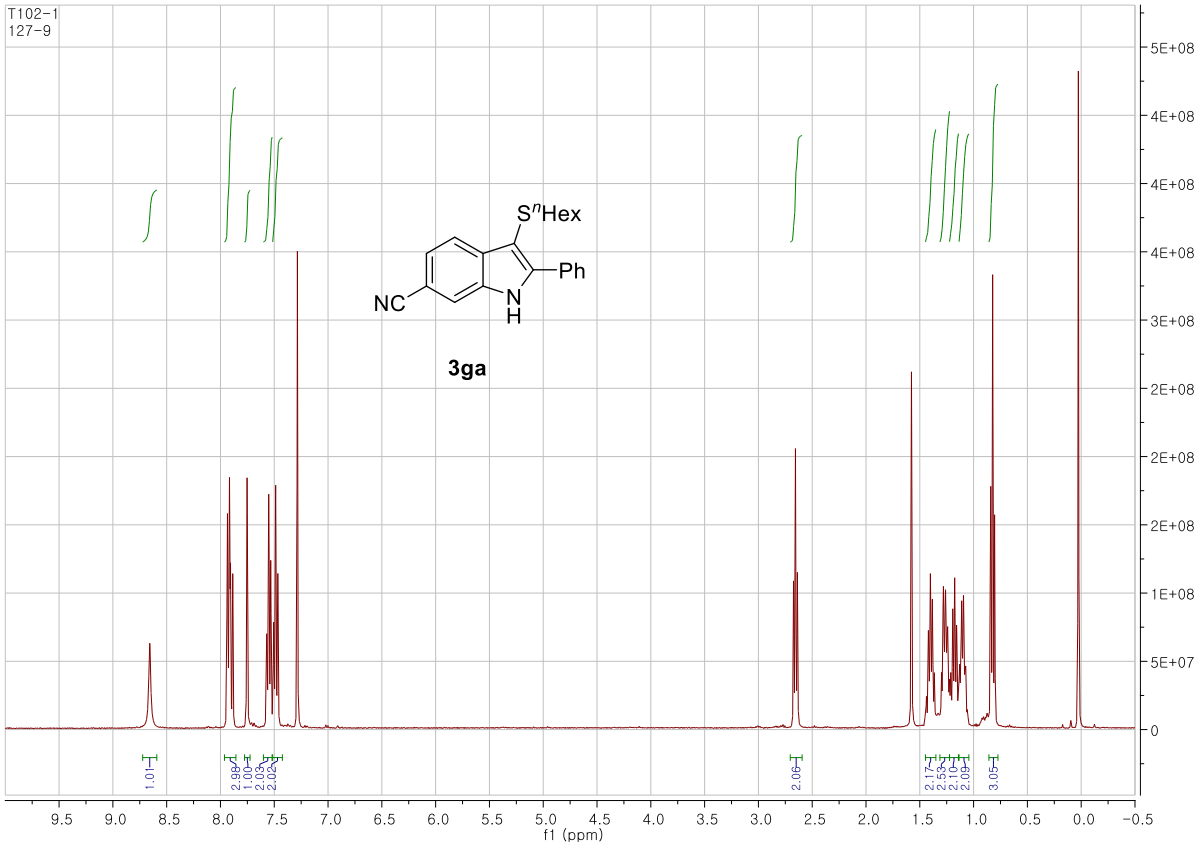

LCY-VII-78
LCY-VII-78 B F1
161.59
159.22
140.89
140.85
135.49
135.37
131.89
128.66
128.30
128.09
120.76
120.66
109.46
109.22
103.70
97.61
97.35
36.47
31.37
29.50
28.19
22.51
14.00
S^{n}Hex
Ph
F
N
H
3ea
f1 (ppm)

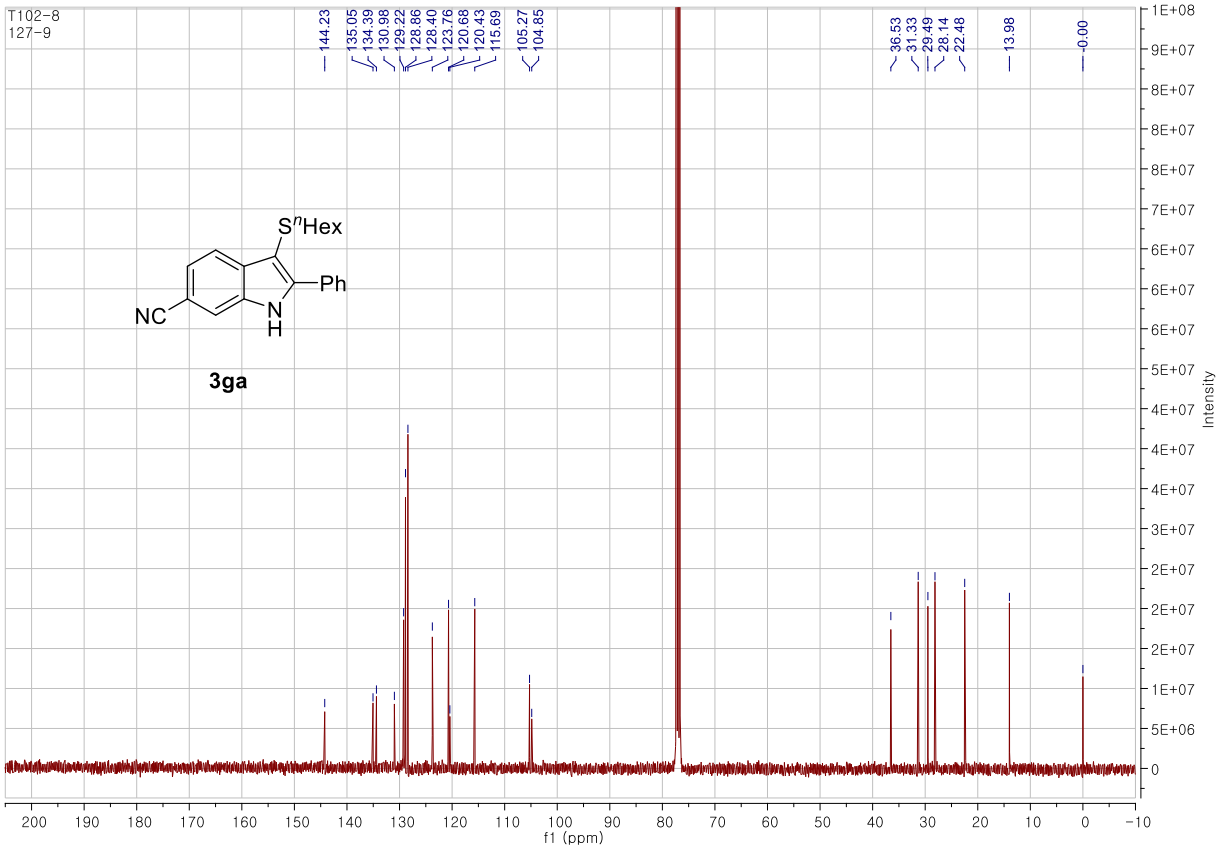

LCY-VIII-1
LCY-VIII-1 B F1
Me
S^{n}Hex
Ph
N
H
3fa
f1 (ppm)

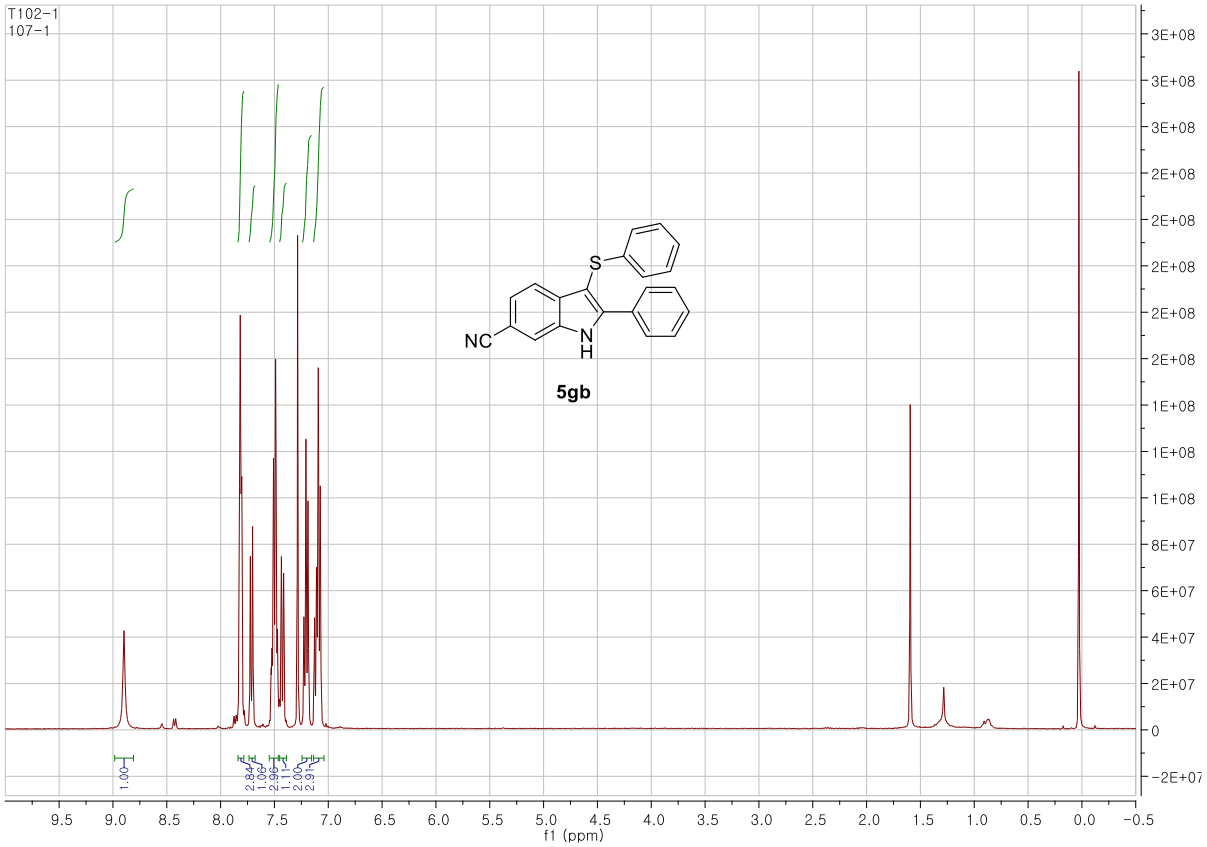

LCY-VIII-1
LCY-VIII-1 B F1
141.76
135.90
132.47
132.22
128.96
128.94
128.45
128.20
122.72
108.90
103.79
38.93
31.43
28.73
28.32
22.52
19.34
14.02
Me
S^{n}Hex
Ph
N
H
3fa
f1 (ppm)

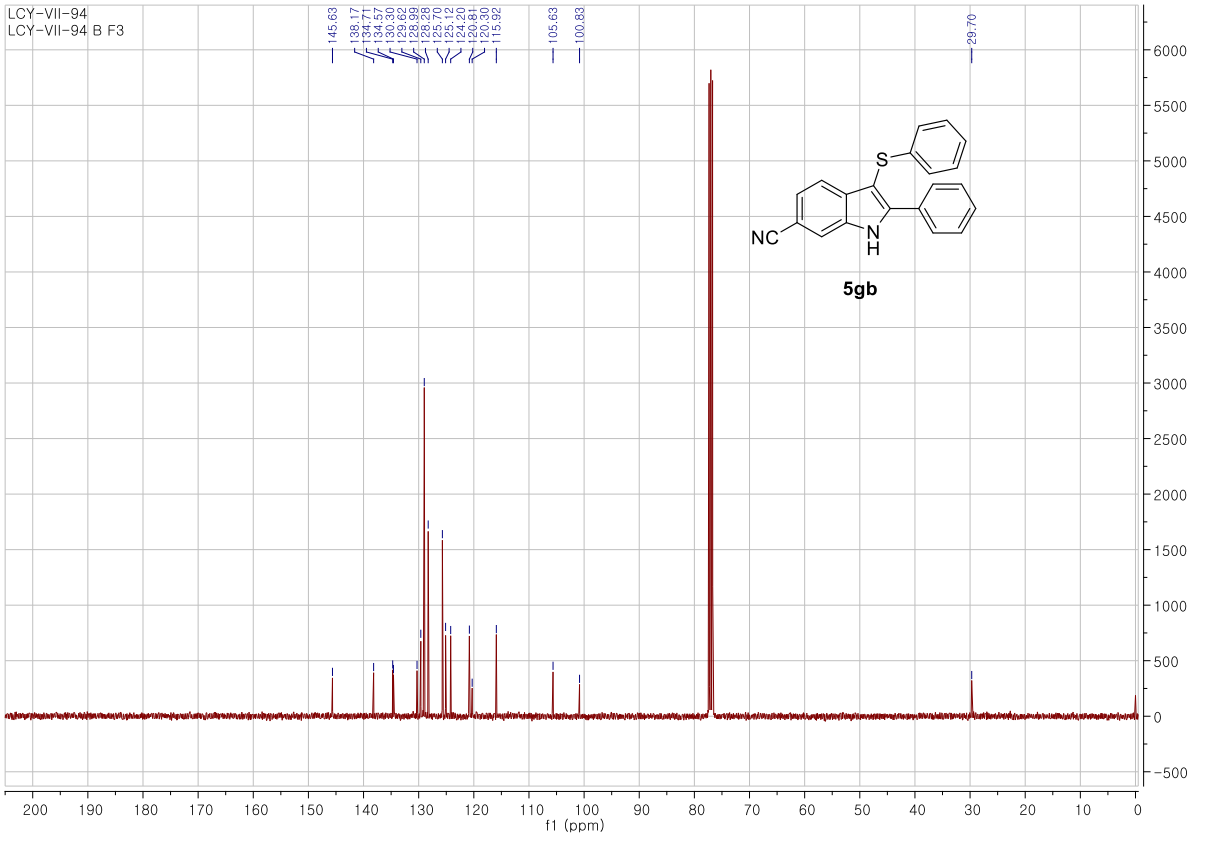

LCY-VIII-1
LCY-VIII-1 A F1
S
N
H
5fb
f1 (ppm)

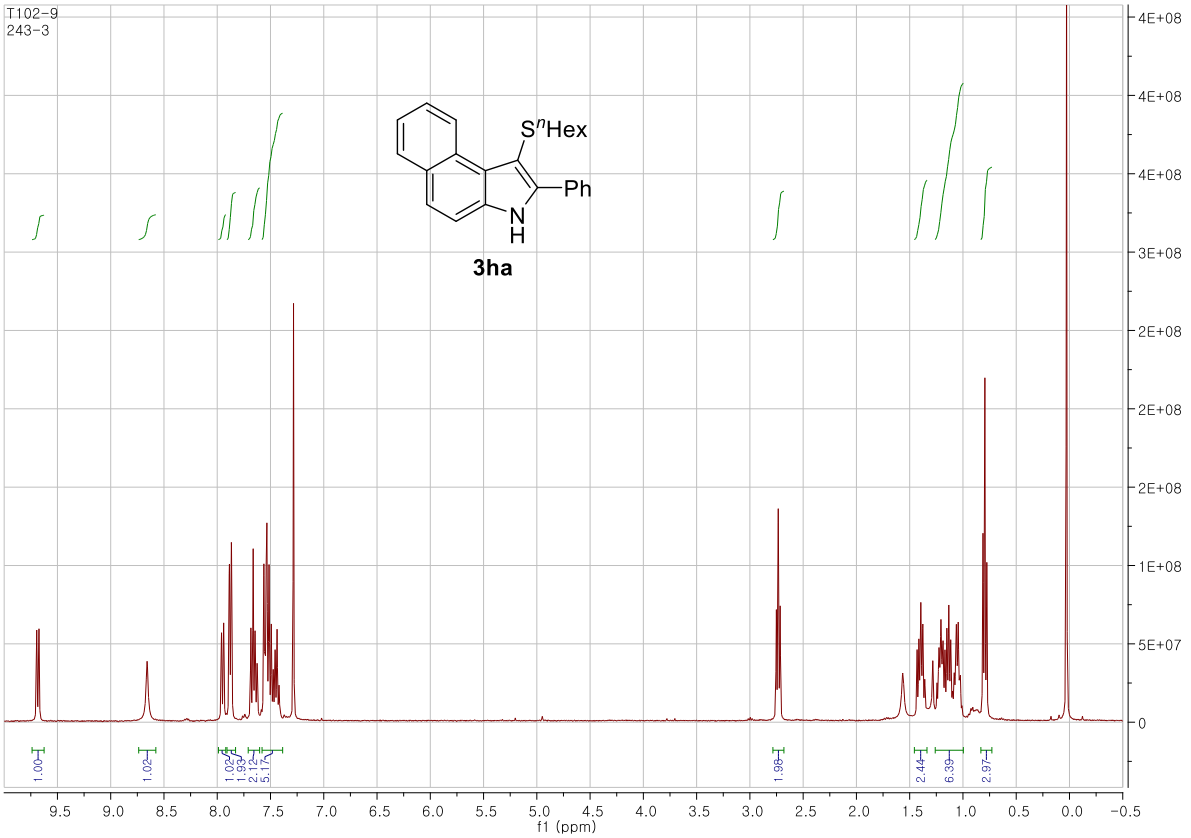

LCY-VIII-1
LCY-VIII-1 A F1
143.01
141.81
135.23
132.44
131.66
128.94
128.74
128.68
128.64
128.55
124.95
124.36
123.20
122.98
109.14
98.92
18.86
S
N
H
5fb
f1 (ppm)

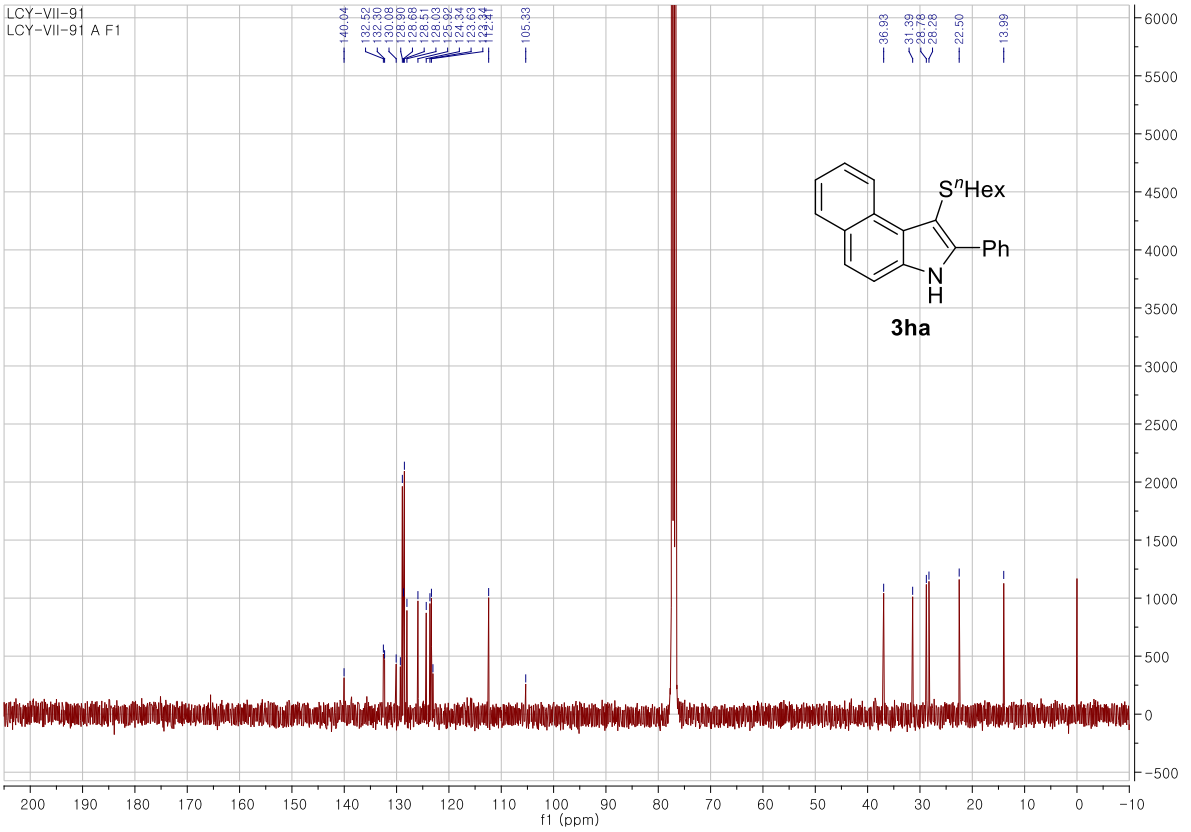

T102-1
127-9
S^{n}Hex
Ph
NC
N
H
3ga
f1 (ppm)

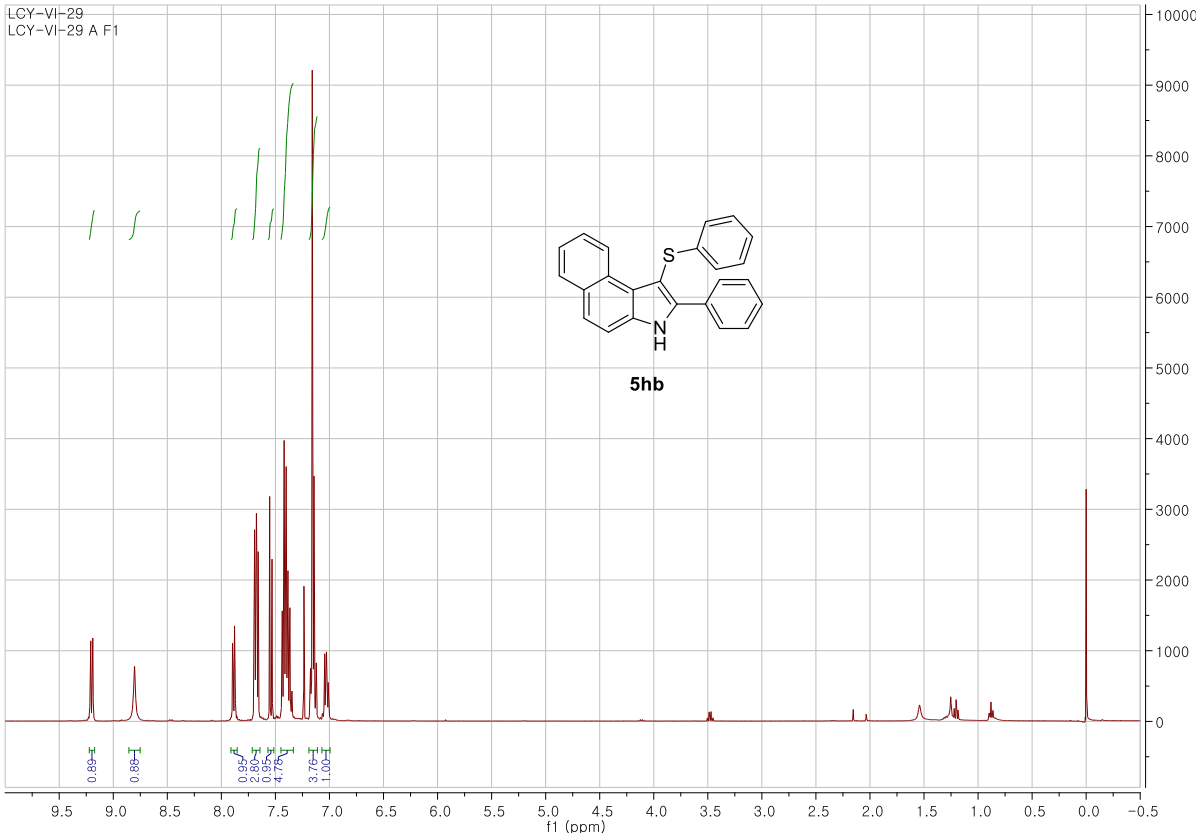

T102-8
127-9
144.23
135.05
134.39
130.98
129.22
128.86
128.40
123.76
120.68
120.43
115.69
105.27
104.85
36.53
31.33
29.49
28.14
22.48
13.98
0.00
S^{n}Hex
Ph
NC
N
H
3ga
f1 (ppm)
Intensity

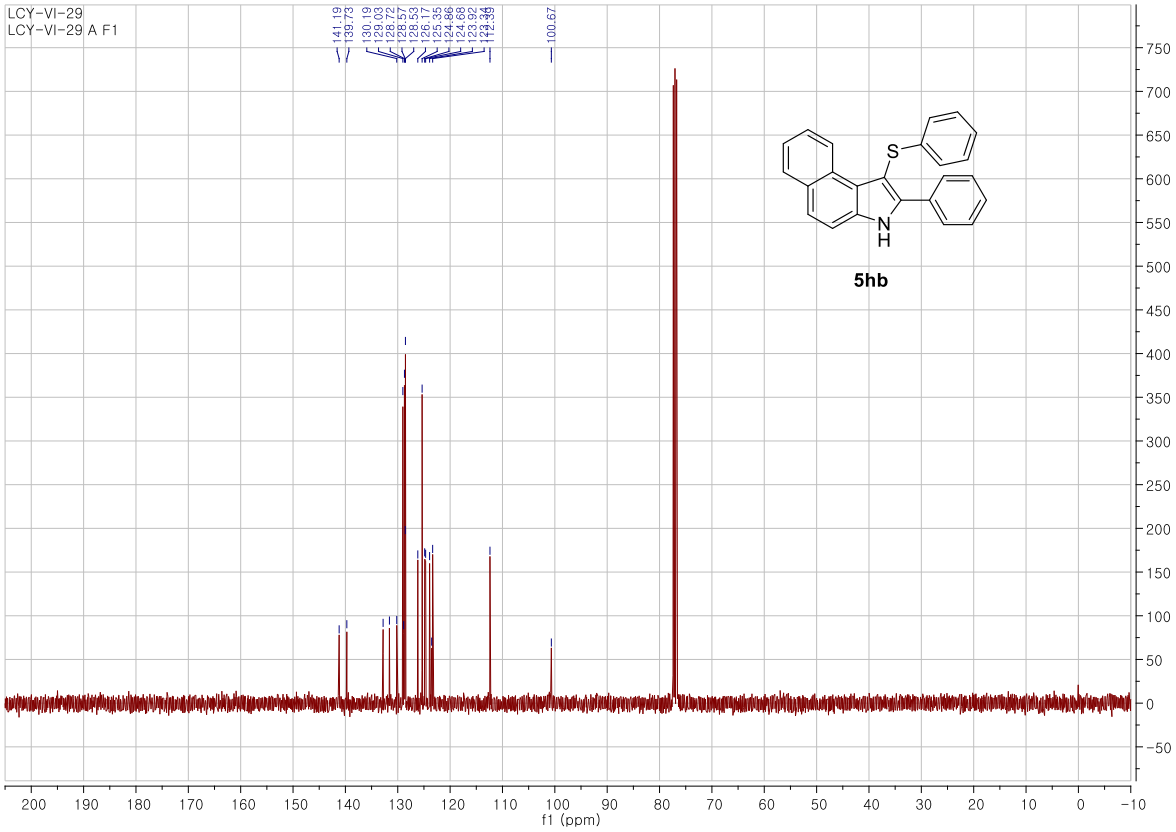

T102-1
107-1
S
NC
N
H
5gb
f1 (ppm)

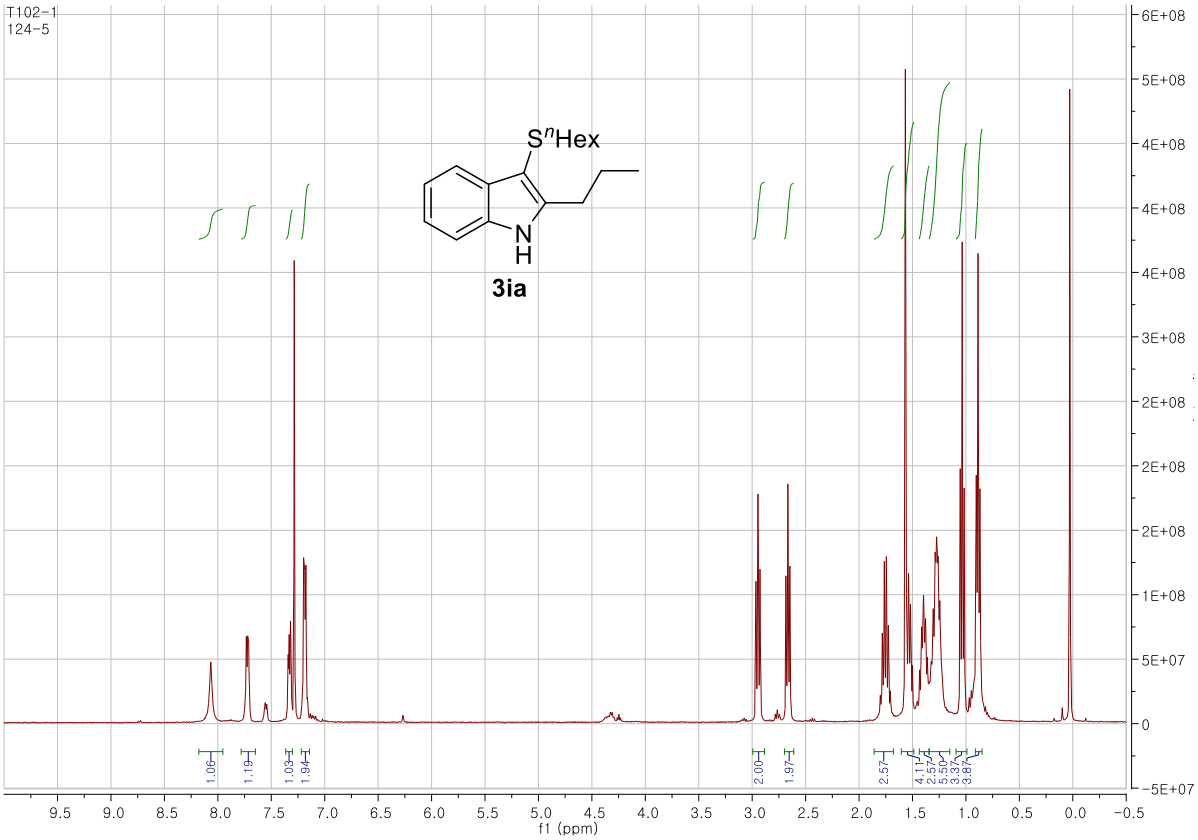

LCY-VII-94
LCY-VII-94 B F3
145.63
138.17
134.71
134.57
133.30
129.62
128.99
128.28
125.70
125.12
124.80
124.61
120.30
115.92
105.63
100.83
29.70
S
NC
N
H
5gb
f1 (ppm)

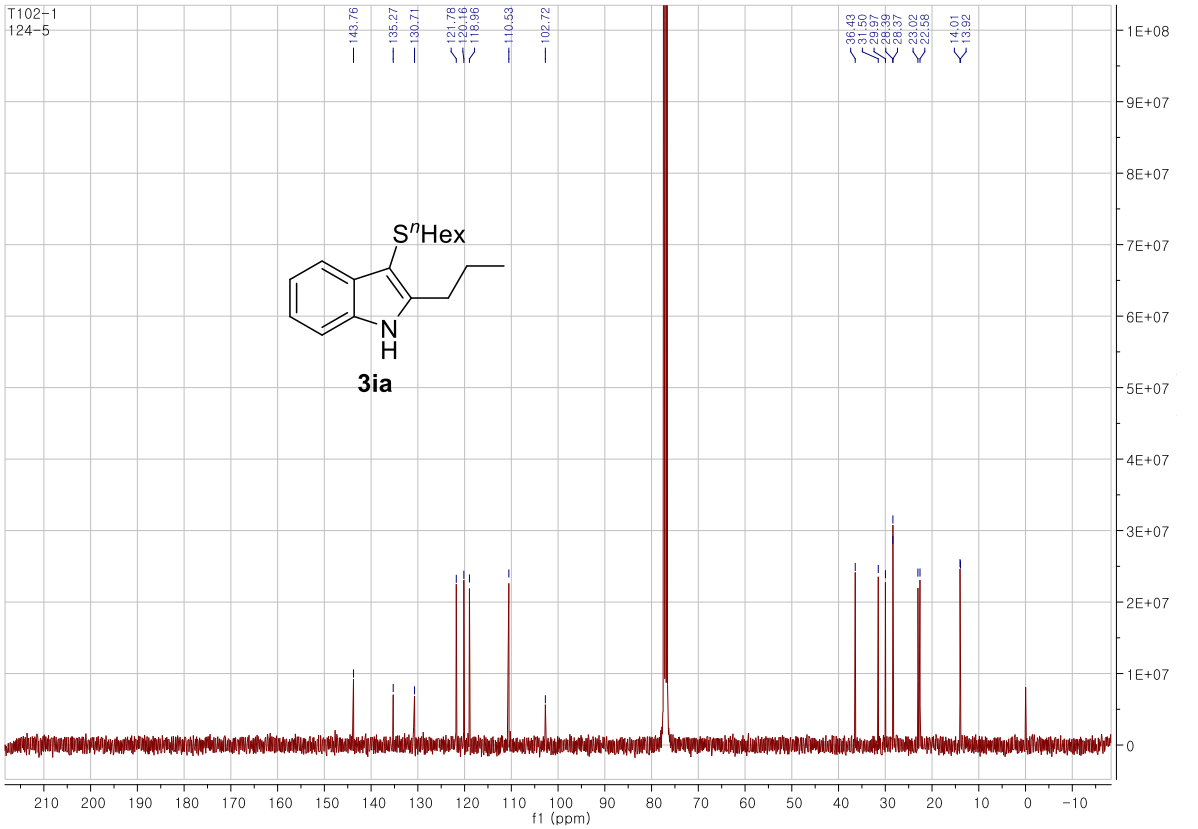

T102-9
243-3
S^{n}Hex
Ph
N
H
3ha
f1 (ppm)

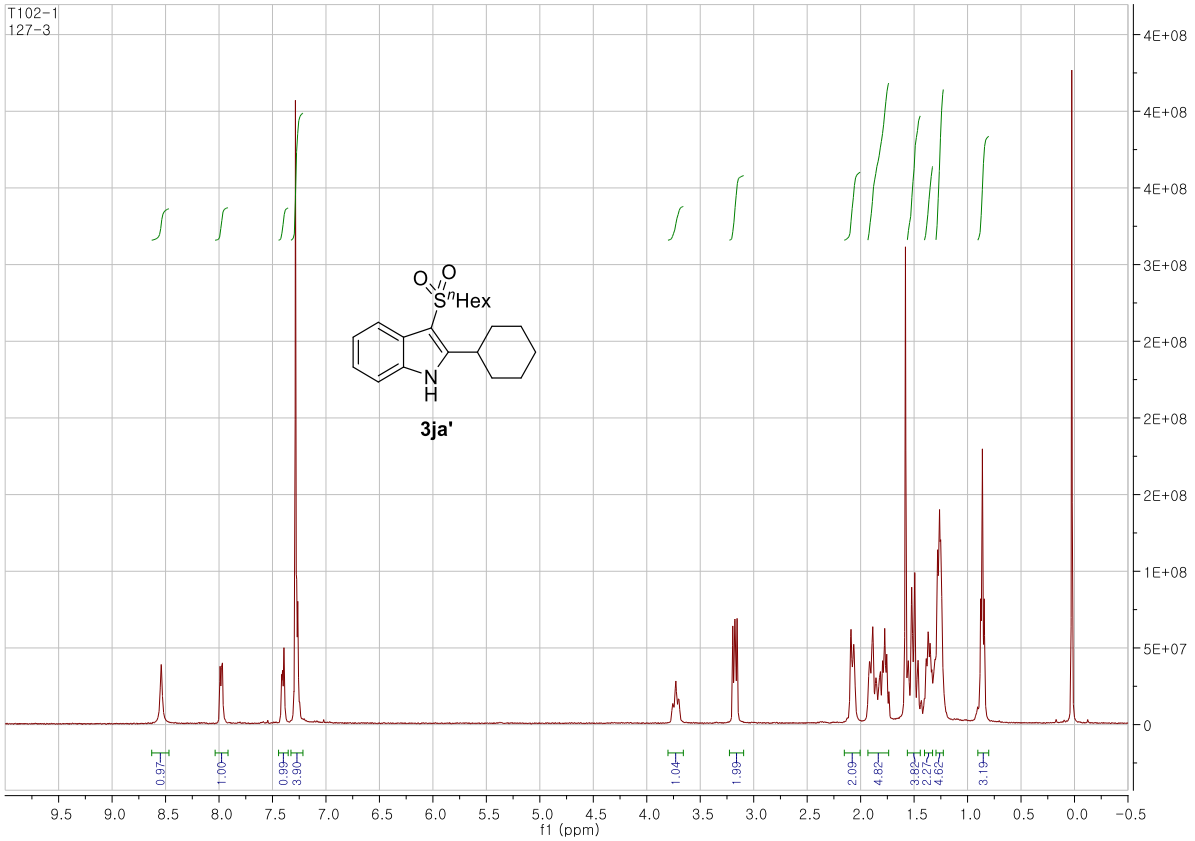

LCY-VII-91
LCY-VII-91 A F1
S^{n}Hex
Ph
N
H
3ha
f1 (ppm)

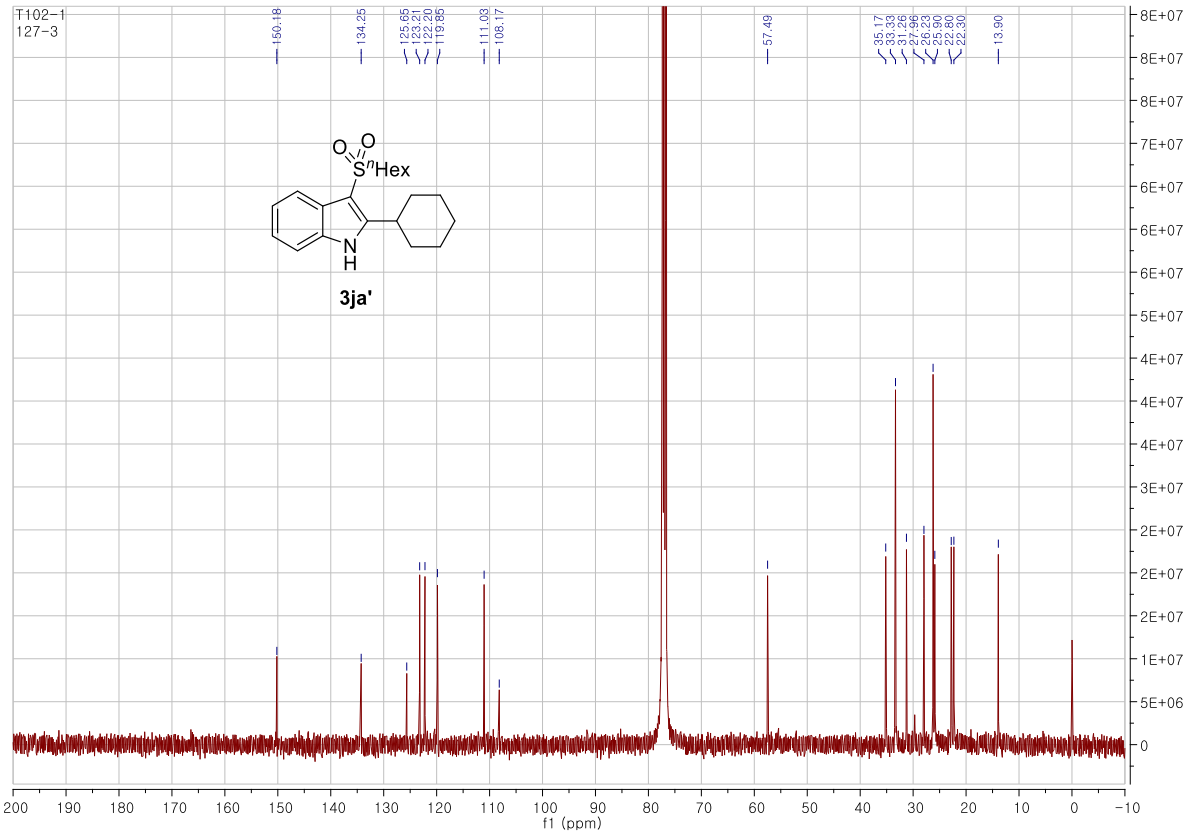

LCY-VI-29
LCY-VI-29 A F1
S
N
H
5hb
f1 (ppm)


LCY-VI-29
LCY-VI-29 A F1
141.19
139.73
130.19
129.03
128.72
128.57
128.53
126.17
125.35
124.86
124.68
123.92
123.34
112.39
100.67
S
N
H
5hb
f1 (ppm)


T102-1
124-5
S^{n}Hex
N
H
3ia
f1 (ppm)


T102-1
124-5
143.76
135.27
130.71
121.78
120.16
118.96
110.53
102.72
36.43
31.50
29.97
28.99
28.37
23.02
22.58
14.01
13.92
S^{n}Hex
N
H
3ia
f1 (ppm)


T102-1
127-3
O
O
S
n
Hex
N
H
3ja'
0.97
1.00
0.99
3.90
1.04
1.99
2.09
4.82
3.82
2.27
4.62
3.19
f1 (ppm)


T102-1
127-3
160.18
134.25
125.65
123.21
122.20
119.85
111.03
108.17
57.49
35.17
33.33
31.66
27.46
26.23
25.90
22.80
22.30
13.90
O
O
S
n
Hex
N
H
3ja'
f1 (ppm)