Supporting Information

Polycyclic heteroaromatic ring construction driven by silver/cobalt co-catalyzed desulfonylative and defluorinative fragment-recombination of enol nonaflates with amidines

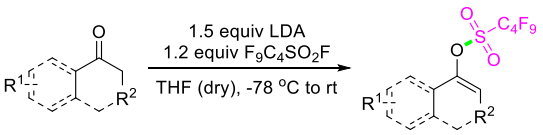
Ting Xie, Yao-Wei Zhang, Li-Li Liu, Zhi-Liang Shen, Teck-Peng Loh,* and Xue-Qiang Chu*

Table of Contents

1. General information

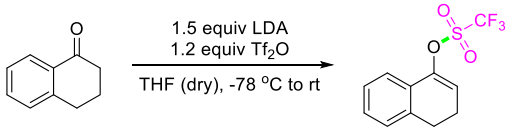
Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. All reactions were carried out under air using undistilled solvent, without the need of precautions to exclude air and moisture unless otherwise noted. Melting points were recorded on an Electrothermal digital melting point apparatus. IR spectra were recorded on a FT-IR spectrophotometer using KBr optics. ^{1}H, ^{19}F, and ^{13}C NMR spectra were recorded in $CDCl_3$ or DMSO-d_6 on Bruker Avance or Joel 400 MHz spectrometers. The chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. High resolution mass spectra (HRMS) were obtained using a commercial apparatus (ESI or EI Source). Column chromatography was generally performed on silica gel (300-400 mesh) or alkali alumina (200-300 mesh) and reactions were monitored by thin layer chromatography (TLC) using UV light to visualize the course of the reactions.

2. General procedures for the synthesis of enol nonaflates[1-2]



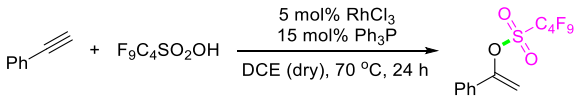
The solution of ketone (5 mmol) in dry THF (25 mL) was cooled to -78 °C and then lithium diisopropylamide (LDA, 3.75 mL, 7.5 mmol, 2.0 mol/L in THF/Hexane) was dropwise added to the reaction mixture. Nonafluorobutanesulfonyl fluoride (1.1 mL, 6 mmol) was added slowly by a syringe over 10 min. The reaction mixture was warmed to room temperature and stirred overnight. The reaction was then quenched by saturated NH_4Cl solution (20 mL) and diluted with EtOAc (20 mL). The organic layer was washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (300-400 mesh) on Et_3N-treated silica gel eluting with petroleum ether to afford enol nonaflate.

3. General procedures for the synthesis of enol triflate 1m[1-2]



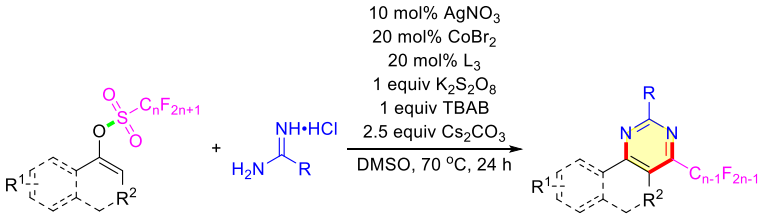
The solution of 3,4-dihydronaphthalen-1(2*H*)-one (0.73 g, 5 mmol) in dry THF (25 mL) was cooled to -78 °C and then lithium diisopropylamide (LDA, 3.75 mL, 7.5 mmol, 2.0 mol/L in THF/Hexane) was dropwise added to the reaction mixture. Trifluoromethanesulfonic anhydride (1.7 g, 6 mmol) was added slowly by a syringe over 10 min. The reaction mixture was warmed to room temperature and stirred overnight. The reaction was then quenched by saturated NH_4Cl solution (20 mL) and diluted with EtOAc (20 mL). The organic layer was washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (300-400 mesh) on Et_3N-treated silica gel eluting with petroleum ether to afford 3,4-dihydronaphthalen-1-yl trifluoromethanesulfonate (0.97g, 70%).

4. General procedures for the synthesis of 1-phenylvinyl 1,1,2,2,3,3,4,4,5,5,5-undecafluoropentane-1-sulfonate[3]



$RhCl_3$ (52 mg, 0.25 mmol) and PPh_3 (197 mg, 0.75 mmol) were filled into a Schlenk tube under N_2 protection. 1,2-Dichloroethane (5 mL), ethynylbenzene (1.1 mL, 10.0 mmol), and nonafluoro-1-butanesulfonic acid (1.5 g, 5.0 mmol) were added consecutively under N_2 protection. Then the reaction mixture was heated to 70 °C with an oil bath and stirred for 24 h. The resulting mixture was concentrated and purified by column chromatography on silica gel (300-400 mesh) to yield the pure 1-phenylvinyl 1,1,2,2,3,3,4,4,5,5,5-undecafluoropentane-1-sulfonate (382 mg, 19%).

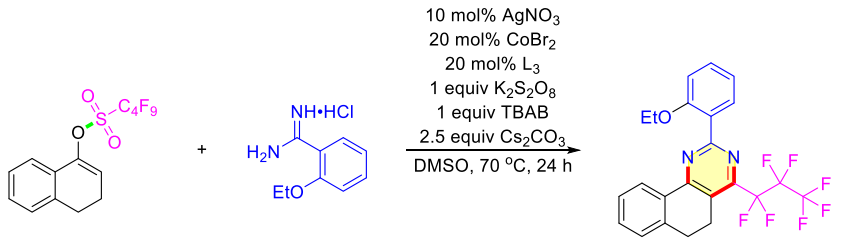
5. General procedures for the synthesis of perfluoroalkylated pyrimidine derivatives



1 2 3-4

A solution of enol sulfonate **1** (0.45 mmol), amidine **2** (0.3 mmol), $AgNO_3$ (5 mg, 0.03 mmol), $CoBr_2$ (13 mg, 0.06 mmol), 4,7-diphenyl-1,10-phenanthroline (20 mg , 0.06 mmol, L_3), $K_2S_2O_8$ (81 mg, 0.3 mmol), tetrabutylammonium bromide (97 mg, 0.3 mmol, TBAB), and Cs_2CO_3 (244 mg, 0.75 mmol) in DMSO (2.0 mL) was stirred under nitrogen atmosphere at 70 °C for 12-24 h. The reaction was then quenched by saturated NH_4Cl solution (20 mL) and diluted with EtOAc (20 mL). The organic layer was washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (300-400 mesh) using petroleum ether/ethyl acetate as eluent to afford the pure product **3** or **4**.

6. 1 mmol scale synthesis of perfluoroalkylated pyrimidine 3ag

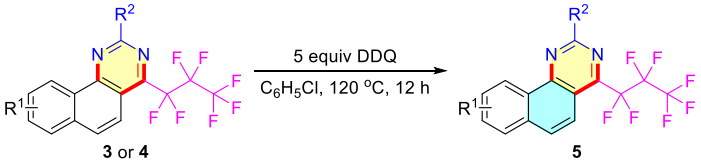


1a 2g 3ag

A solution of enol sulfonate **1a** (642 mg, 1.5 mmol), amidine **2g** (201 mg, 1 mmol), $AgNO_3$ (17 mg, 0.1 mmol), $CoBr_2$ (44 mg, 0.2 mmol), 4,7-diphenyl-1,10-phenanthroline (66 mg , 0.2 mmol, L_3), $K_2S_2O_8$ (270 mg, 1 mmol), tetrabutylammonium bromide (322 mg, 1 mmol, TBAB), and Cs_2CO_3 (815 mg, 2.5 mmol) in DMSO (5.0 mL) was stirred at 70 °C for 24 h under nitrogen atmosphere.

The reaction was then quenched by saturated NH_4Cl solution (20 mL) and diluted with EtOAc (20 mL). The organic layer was washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (300-400 mesh) using petroleum ether/ethyl acetate as eluent to afford the pure product **3ag** in 60% yield (282 mg).

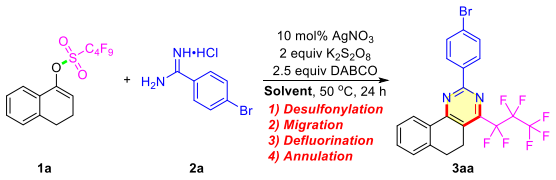
7. General procedures for the synthesis of perfluoroalkylated benzo[*h*]quinazoline derivatives



Dihydrobenzo[*h*]quinazoline **3** or **4** (0.1 mmol), 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (0.5 mmol, 5 equiv, DDQ), and PhCl (2 mL) was stirred at 120 °C for 12 h. Upon completion of the reaction (indicated by TLC), solvent was removed under vacuum and the residue was purified by flash silica gel column chromatography (300-400 mesh) using petroleum ether/ethyl acetate as eluent to afford the pure products **5**.

8. Optimization of reaction conditions

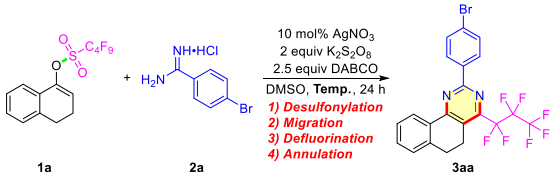
Table S1. Optimization of the reaction solvent[a]



Entry	**Solvent**	Yield of **3aa** (%)[b]
1	MeCN	0
2	toluene	0
3	EtOH	0
4	1,4-dioxane	0
5	DMF	0
6	DCE	0
7	DME	0
8	acetone	0
9	PhCl	0
10	$(CH_2OH)_2$	0
11	$CF_3CH(OH)CF_3$	0
12	^{t}BuOH	0
13	**DMSO**	**5**

[a] Reaction conditions: 3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (**1a**, 0.36 mmol), 4-bromobenzamidine hydrochloride (**2a**, 0.3 mmol), $AgNO_3$ (0.03 mmol), $K_2S_2O_8$ (0.6 mmol), and DABCO (0.75 mmol) in solvent (2.0 mL) at 50 °C for 24 h under N_2. [b] Yields were determined by NMR analysis with 1,4-dimethoxybenzene as an internal standard.

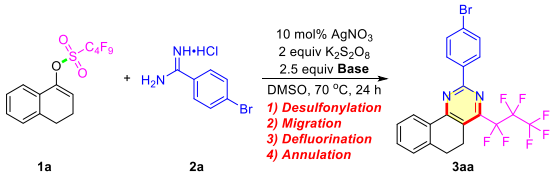
Table S2. Optimization of the reaction temperature[a]



Entry	**Temp.** (°C)	Yield of **3aa** (%)[b]
1	30	0
2	**70**	**9**
3	90	4
4	110	8

[a] Reaction conditions: 3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (**1a**, 0.36 mmol), 4-bromobenzamidine hydrochloride (**2a**, 0.3 mmol), $AgNO_3$ (0.03 mmol), $K_2S_2O_8$ (0.6 mmol), and DABCO (0.75 mmol) in DMSO (2.0 mL) for 24 h under N_2. [b] Yields were determined by NMR analysis with 1,4-dimethoxybenzene as an internal standard.

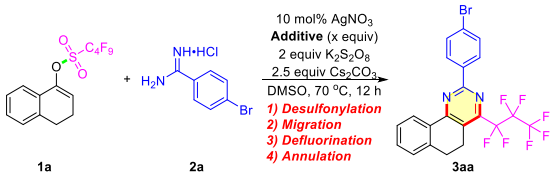
Table S3. Optimization of the reaction base[a]



Entry	**Base**	Yield of **3aa** (%)[b]
1	$NaHCO_3$	<5
2	NaOAc	0
3	**Cs_2CO_3**	**22**
4	K_2CO_3	<5
5	Na_2CO_3	<5
6	NaOH	0
7	K_3PO_4	<5
8	DBU	<5
9	Et_3N	0
10	sodium ascorbate	trace
11	TMEDA	0
12	$^{i}Pr_2NH$	0
13	^{t}BuOLi	8
14	DMAP	0
15	pyridine	0
16	CsOAc	<5
17	cesium pivalate	<5

[a] Reaction conditions: 3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (**1a**, 0.36 mmol), 4-bromobenzamidine hydrochloride (**2a**, 0.3 mmol), $AgNO_3$ (0.03 mmol), $K_2S_2O_8$ (0.6 mmol), and base (0.75 mmol) in DMSO (2.0 mL) at 70 °C for 24 h under N_2. [b] Yields were determined by NMR analysis with 1,4-dimethoxybenzene as an internal standard.

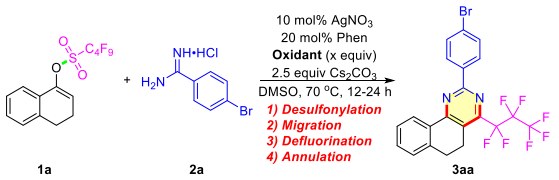
Table S4. Optimization of the reaction additive[a]



Entry	**Additive (x equiv)**	Yield of **3aa** (%)[b]
1	AIBN (0.05)	18
2	BPO (0.05)	22
3	$Co(acac)_2$ (0.05)	24
4	$Fe(OAc)_2$ (0.05)	17
5	In (0.2)	25
6	*n*-C_4F_9I (0.05)	20
7	**Phen (0.2)**	**32**

[a] Reaction conditions: 3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (**1a**, 0.36 mmol), 4-bromobenzamidine hydrochloride (**2a**, 0.3 mmol), $AgNO_3$ (0.03 mmol), $K_2S_2O_8$ (0.6 mmol), Cs_2CO_3 (0.75 mmol), and additives (0.06 or 0.015 mmol) in DMSO (2.0 mL) at 70 °C for 12 h under N_2. [b] Yields were determined by NMR analysis with 1,4-dimethoxybenzene as an internal standard.

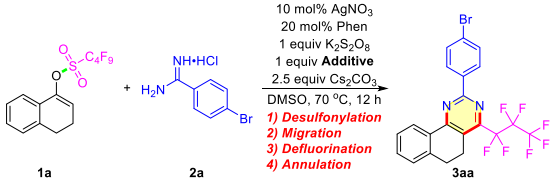
Table S5. Optimization of the reaction oxidant[a]



Entry	**Oxidant (x equiv)**	Time (h)	Yield of **3aa** (%)[b]
1	oxone (2)	24	0
2	$(NH_4)_2S_2O_8$ (2)	24	7
3	$Na_2S_2O_8$ (2)	24	31
4	**$K_2S_2O_8$ (1)**	**12**	**42**
5	$K_2S_2O_8$ (3)	12	<10
6	$K_2S_2O_8$ (0.5)	12	<10
7	$Cu(OAc)_2$ (1)	12	0
8	$PhI(OAc)_2$ (1)	12	0

[a] Reaction conditions: 3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (**1a**, 0.36 mmol), 4-bromobenzamidine hydrochloride (**2a**, 0.3 mmol), $AgNO_3$ (0.03 mmol), oxidant (0.15-0.9 mmol), Cs_2CO_3 (0.75 mmol), and Phen (0.06 mmol) in DMSO (2.0 mL) at 70 °C under N_2 for 12-24 h. [b] Yields were determined by NMR analysis with 1,4-dimethoxybenzene as an internal standard.

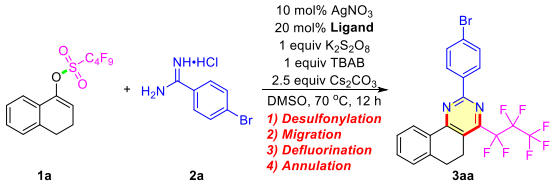
Table S6. Optimization of the phase transfer additives[a]



Entry	**Phase transfer additive**	Yield of **3aa** (%)[b]
1	TBAI	56
2	TBAHS	19
3	**TBAB**	**75**
4	TOMAC	33
5	BTEAB	60
6	CTAB	35

[a] Reaction conditions: 3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (**1a**, 0.45 mmol), 4-bromobenzamidine hydrochloride (**2a**, 0.3 mmol), $AgNO_3$ (0.03 mmol), $K_2S_2O_8$ (0.3 mmol), Cs_2CO_3 (0.75 mmol), Phen (0.06 mmol), and phase transfer additive (0.3 mmol) in DMSO (2.0 mL) at 70 °C for 12 h under N_2. [b] Yields were determined by NMR analysis with 1,4-dimethoxybenzene as an internal standard.

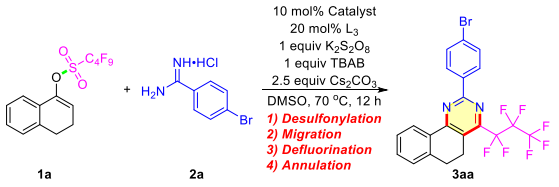
Table S7. Optimization of the reaction ligand[a]



Entry	**Ligand**	Yield of **3aa** (%)[b]
1	1,10-Phenanthroline (L_1)	75
2	2,2'-Bipyridine (L_2)	37
3	**4,7-Diphenyl-1,10-Phenanthroline (L_3)**	**80**
4	2,9-Dimethyl-1,10-Phenanthroline (L_4)	39
5	4,7-Dimethoxy-1,10-Phenanthroline (L_5)	65

[a] Reaction conditions: 3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (**1a**, 0.45 mmol), 4-bromobenzamidine hydrochloride (**2a**, 0.3 mmol), $AgNO_3$ (0.03 mmol), $K_2S_2O_8$ (0.3 mmol), Cs_2CO_3 (0.75 mmol), ligand (0.06 mmol), and TBAB (0.3 mmol) in DMSO (2.0 mL) at 70 °C for 12 h under N_2. [b] Yields were determined by NMR analysis with 1,4-dimethoxybenzene as an internal standard.

Table S8. Optimization of the reaction catalyst[a]

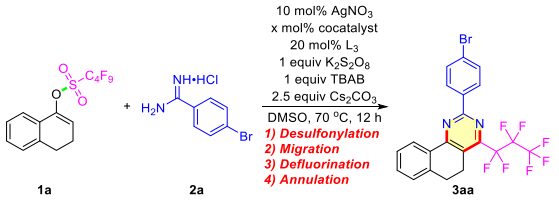


Entry	**Catalyst**	Yield of **3aa** (%)[b]
1	**$AgNO_3$**	**80 (56)[c]**

2	$AgNO_3$	60[d]
3	$AgNO_3$	35[e]
4	Ag_2CO_3	56
5	AgOAc	59
6	Ag_2O	56
7	Ag_2SO_4	33
8	AgI	49
9	$Fe(NO_3)_3·9H_2O$	<10
10	$Cu(NO_3)_2·2H_2O$	<10
11	$Co(acac)_2$	62
12	$Zn(NO_3)_2·6H_2O$	34
13	$AgNO_3$	<10[f]
14	$AgNO_3$	33[g]

[a] Reaction conditions: 3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (**1a**, 0.45 mmol), 4-bromobenzamidine hydrochloride (**2a**, 0.3 mmol), catalyst (0.03 mmol), $K_2S_2O_8$ (0.3 mmol), Cs_2CO_3 (0.75 mmol), 4,7-diphenyl-1,10-phenanthroline (L_3, 0.06 mmol), and TBAB (0.3 mmol) in DMSO (2.0 mL) at 70 °C for 12 h under N_2. [b] Yields were determined by NMR analysis with 1,4-dimethoxybenzene as an internal standard. [c] Isolated yield. [d] 0.54 mmol of **1a** was used. [e] 0.6 mmol of **1a** was used. [f] 0.45 mmol of Cs_2CO_3 was used. [g] 1.05 mmol of Cs_2CO_3 was used.

Table S9. Optimization of the cocatalyst[a]

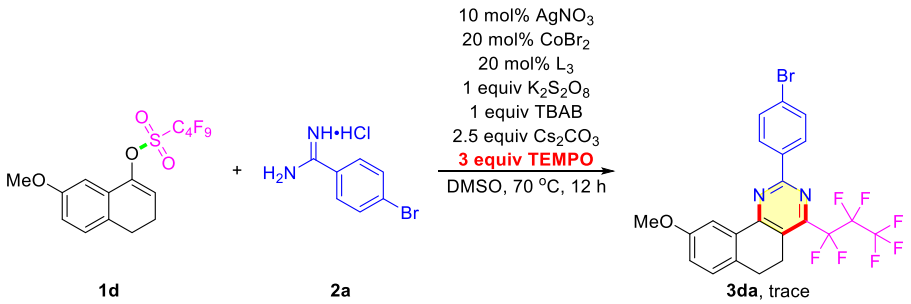


Entry	**cocatalyst (x mol%)**	Yield of **3aa** (%)[b]
1	--	80 (56)[c]
2	$Ir(ppy)_3$ (1)	<20[d]
3	$Ru(bipy)_3Cl_2·6H_2O$ (1)	<20[d]
4	$Co(OAc)_2$ (10)	(48)[c]
5	$Co(PPh_3)_2Cl_2$ (10)	83
6	$Co(acac)_2$ (10)	78
7	$Co(acac)_3$ (10)	<10
8	$CoBr_2$ (10)	(56)[c,d]
9	**$CoBr_2$ (20)**	**90 (63)[c,d,e]**
10	$CoBr_2$ (30)	78

[a] Reaction conditions: 3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (**1a**, 0.45 mmol), benzamidine hydrochloride (**2a**, 0.3 mmol), $AgNO_3$ (0.03 mmol), catalyst (0.03 mmol), $K_2S_2O_8$ (0.3 mmol), Cs_2CO_3 (0.75 mmol), 4,7-diphenyl-1,10-phenanthroline (L_3, 0.06 mmol), and TBAB (0.3 mmol) in DMSO (2.0 mL) at 70 °C for 12 h under N_2. [b] Yields were determined by NMR analysis with 1,4-dimethoxybenzene as an internal standard. [c] Isolated yield. [d] Irradiation with blue LEDs (8 W). [e] For 24 h

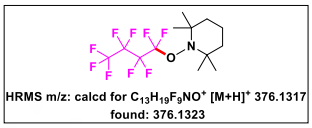
9. Mechanistic studies

1) Trapping experiment with 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO)



A solution of 7-methoxy-3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (206 mg, 0.45 mmol, **1d**), 4-bromobenzamidine hydrochloride (71 mg, 0.3 mmol, **2a**), 2,2,6,6-tetramethylpiperidin-1-oxyl (0.9 mmol, TEMPO), $AgNO_3$ (5 mg, 0.03 mmol), $CoBr_2$ (13 mg, 0.06 mmol), 4,7-diphenyl-1,10-phenanthroline (20 mg , 0.06 mmol, L_3), $K_2S_2O_8$ (81 mg, 0.3 mmol), tetrabutylammonium bromide (97 mg, 0.3 mmol, TBAB), and Cs_2CO_3 (244 mg, 0.75 mmol) in DMSO (2.0 mL) was stirred at 70 °C for 24 h under nitrogen atmosphere.

Elemental Composition Report Page 1



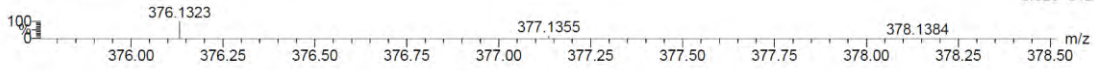
HRMS m/z: calcd for $C_{13}H_{19}F_9NO^+$ $[M+H]^+$ 376.1317
found: 376.1323

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
64 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 12-17 H: 10-19 N: 0-5 O: 0-2 F: 6-10

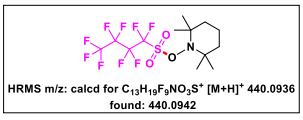
CXQ-X (1.836) Is (1.00,1.00) C13H19F9NO
1: TOF MS ES+

8.62e+012



Minimum:	80.00				-1.5				
Maximum:	100.00		2.0	5.0	50.0				
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
376.1323	100.00	376.1323	0.0	0.0	0.5	41.8	0.000	100.00	C13 H19 N O F9
		376.1312	1.1	2.9	4.5	72.3	30.533	0.00	C16 H18 N F8

Elemental Composition Report Page 1



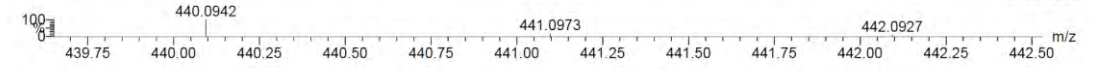
HRMS m/z: calcd for $C_{13}H_{19}F_9NO_3S^+$ $[M+H]^+$ 440.0936
found: 440.0942

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
66 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 12-15 H: 15-20 N: 0-2 O: 2-5 S: 0-2 F: 7-10

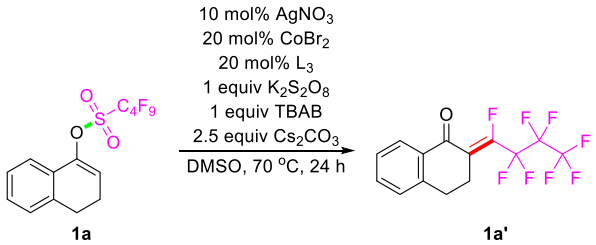
CXQ-X (2.486) Is (1.00,1.00) C13H19F9NO3S
1: TOF MS ES+

8.15e+012

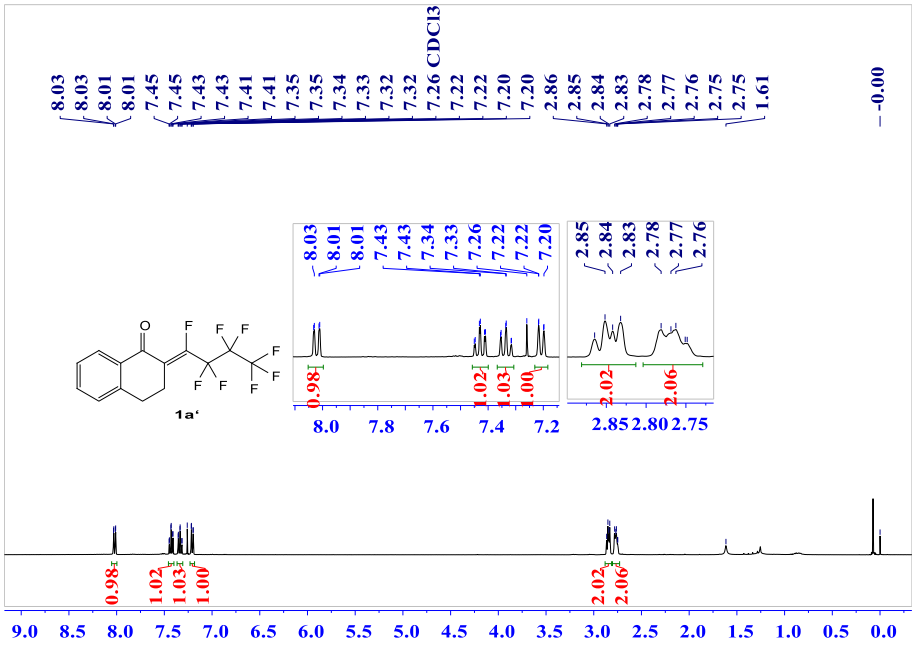


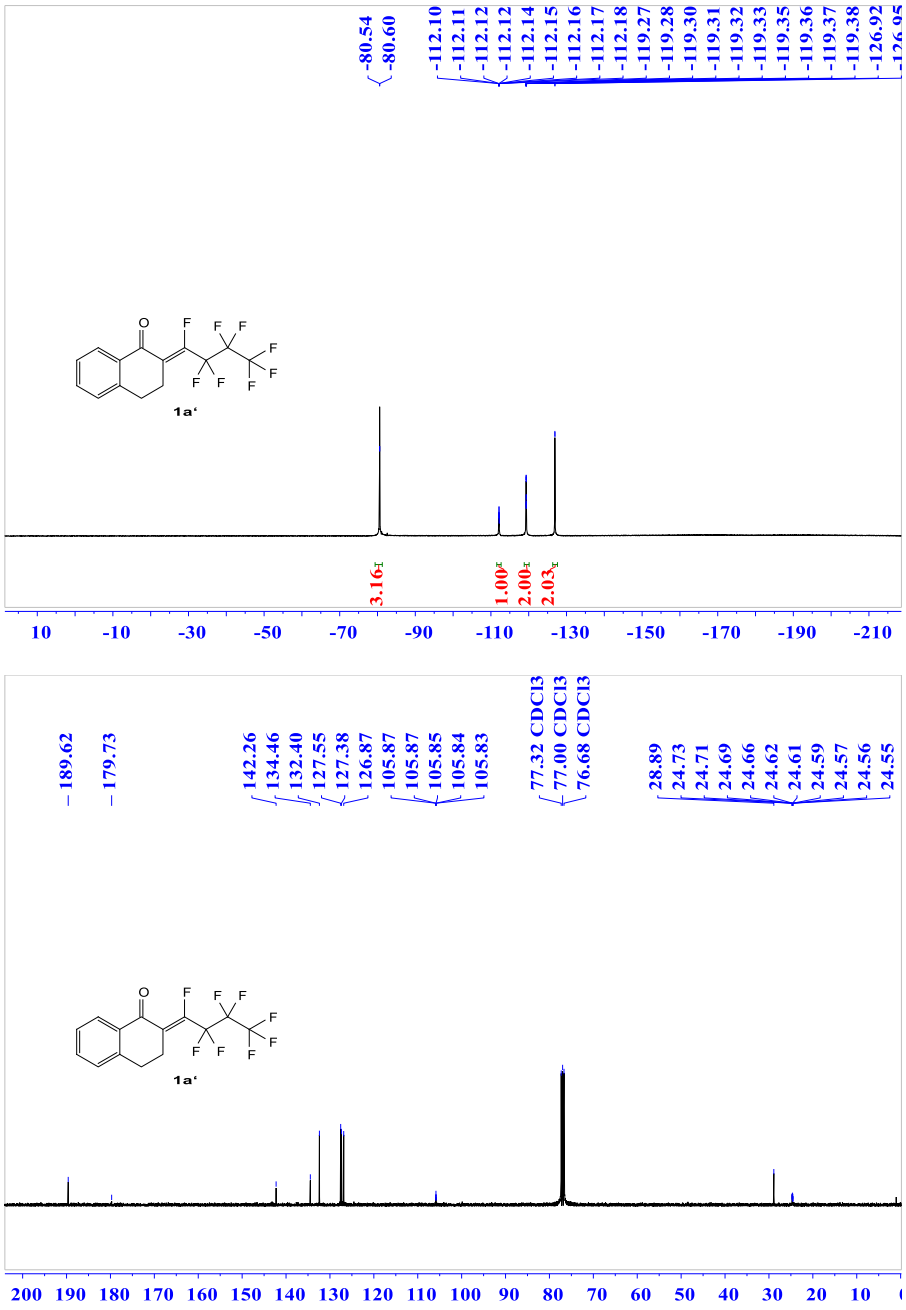
Minimum:	80.00				-1.5				
Maximum:	100.00		2.0	5.0	50.0				
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
440.0942	100.00	440.0942	0.0	0.0	0.5	46.5	n/a	n/a	C13 H19 N O3 S F9

2) Detection of the by-product (*Z*)-2-(perfluoropentylidene)-3,4-dihydronaphthalen-1(2*H*)-one (1a')

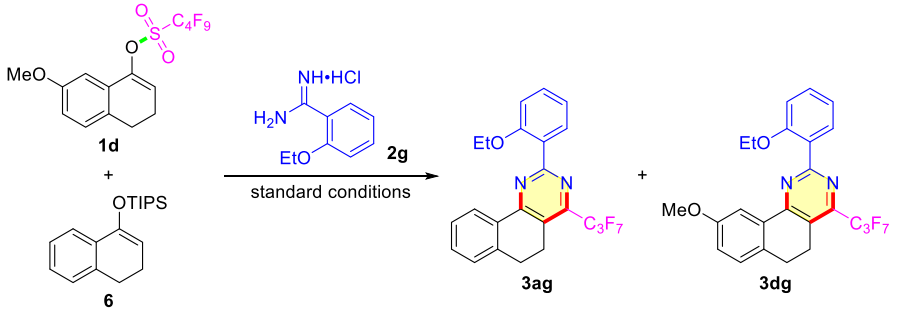


A solution of 3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (129 mg, 0.45 mmol, **1a**), $AgNO_3$ (5 mg, 0.03 mmol), $CoBr_2$ (13 mg, 0.06 mmol), 4,7-diphenyl-1,10-phenanthroline (20 mg, 0.06 mmol, L_3), $K_2S_2O_8$ (81 mg, 0.3 mmol), tetrabutylammonium bromide (97 mg, 0.3 mmol, TBAB), and Cs_2CO_3 (244 mg, 0.75 mmol) in DMSO (2.0 mL) was stirred at 70 °C for 24 h under nitrogen atmosphere. The reaction was then quenched by saturated NH_4Cl solution (20 mL) and diluted with EtOAc (20 mL). The organic layer was washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (300-400 mesh) using petroleum ether/ethyl acetate as eluent to afford by-product (*Z*)-2-(perfluoropentylidene)-3,4-dihydronaphthalen-1(2*H*)-one (<5%, **1a'**).

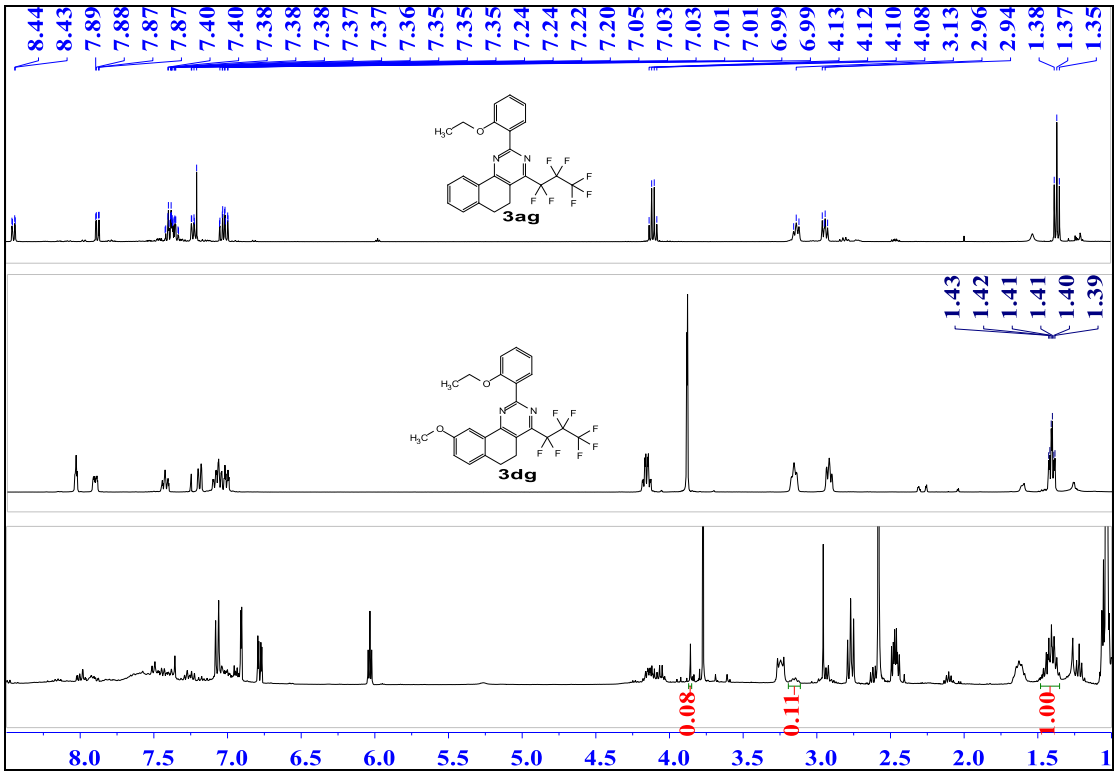




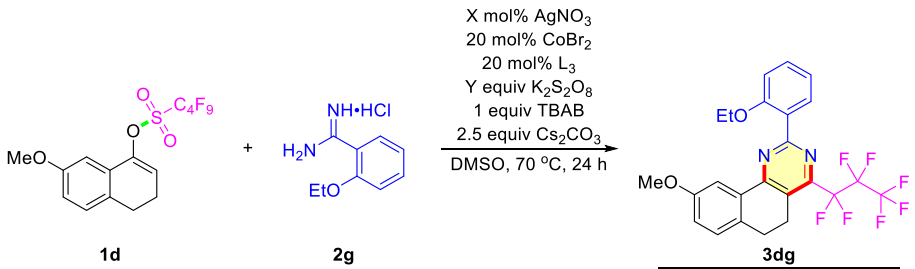
3) Crossover experiment with an equimolar amount of 7-methoxy-3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (1d) and ((3,4-dihydronaphthalen-1-yl)oxy)triisopropylsilane (6)



A solution of 7-methoxy-3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (103 mg, 0.225 mmol, **1d**), ((3,4-dihydronaphthalen-1-yl)oxy)triisopropylsilane (68 mg, 0.225 mmol, **6**), 2-ethoxybenzimidamide hydrochloride (60 mg, 0.3 mmol, **2g**), $AgNO_3$ (5 mg, 0.03 mmol), $CoBr_2$ (13 mg, 0.06 mmol), 4,7-diphenyl-1,10-phenanthroline (20 mg, 0.06 mmol, L_3), $K_2S_2O_8$ (81 mg, 0.3 mmol), tetrabutylammonium bromide (97 mg, 0.3 mmol, TBAB), and Cs_2CO_3 (244 mg, 0.75 mmol) in DMSO (2.0 mL) was stirred at 70 °C for 24 h under nitrogen atmosphere. The reaction was then quenched by saturated NH_4Cl solution (20 mL) and diluted with EtOAc (20 mL). The organic layer was washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The ratio of two possible products **3ag** and **3dg** was determined by NMR analysis (**3ag**/**3dg** = 1:1).



4) Control experiment without $K_2S_2O_8$ or $AgNO_3$

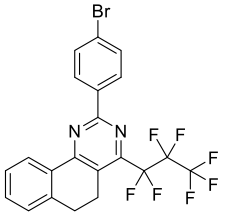


X	Y	Yield of **3dg** (%)
10	0	trace
0	1	<15

A solution of 7-methoxy-3,4-dihydronaphthalen-1-yl 1,1,2,2,3,3,4,4,4-nonafluorobutane-1-sulfonate (206 mg, 0.45 mmol, **1d**), 2-ethoxybenzimidamide hydrochloride (60 mg, 0.3 mmol, **2g**), $AgNO_3$ (0-0.3 mmol), $CoBr_2$ (13 mg, 0.06 mmol), 4,7-diphenyl-1,10-phenanthroline (20 mg, 0.06

mmol, L_3), $K_2S_2O_8$ (0-0.3 mmol), tetrabutylammonium bromide (97 mg, 0.3 mmol, TBAB), and Cs_2CO_3 (244 mg, 0.75 mmol) in DMSO (2.0 mL) was stirred at 70 °C for 24 h under nitrogen atmosphere. The reaction was then quenched by saturated NH_4Cl solution (20 mL) and diluted with EtOAc (20 mL). The organic layer was washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure.

10. Characterization data for perfluoroalkylated pyrimidine derivatives



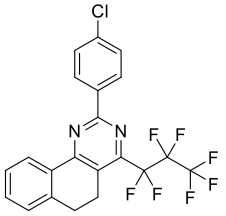
2-(4-Bromophenyl)-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3aa):

Yield = 63% (95 mg). White solid. M.p. 111.1–112.9 °C.

IR (KBr): ν = 3072, 2943, 1556, 1399, 1227, 930, 770, 743, 606 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.53 (dd, J = 7.4, 1.7 Hz, 1H), 8.47 – 8.43 (m, 2H), 7.67 – 7.61 (m, 2H), 7.51 – 7.43 (m, 2H), 7.32 – 7.27 (m, 1H), 3.21 – 3.14 (m, 2H), 3.00 – 2.97 (m, 2H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.64 (t, J = 9.5 Hz, 3F), -110.17 (q, J = 9.6 Hz, 2F), -125.44 (d, J = 3.6 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 162.6, 161.2, 152.1 (t, $J_{C\text{-}F}$ = 25.3 Hz), 139.4, 135.7, 132.0, 131.9, 131.8, 129.8, 127.9, 127.5, 126.4, 126.3, 125.9, 26.8, 22.9 (m) ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{21}H_{13}BrF_7N_2$ $[M+H]^+$ 505.0145, found: 505.0163.



2-(4-Chlorophenyl)-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3ab):

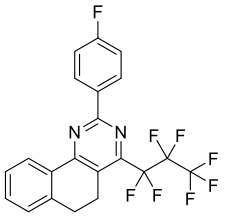
Yield = 58% (80 mg). White solid. M.p. 107.4–108.9 °C.

IR (KBr): ν = 2954, 1553, 1397, 931, 766 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.54 – 8.48 (m, 3H), 7.50 – 7.41 (m, 4H), 7.30 – 7.26 (m, 1H), 3.16 (dd, J = 8.4, 6.4 Hz, 2H), 2.97 (dd, J = 8.7, 5.8 Hz, 2H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.78 (t, J = 9.9 Hz, 3F), -110.30 (q, J = 10.1 Hz, 2F), -125.55 – -125.62 (m, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 162.6, 161.1 (t, $J_{C\text{-}F}$ = 1.3 Hz), 152.1 (t, $J_{C\text{-}F}$ = 24.0 Hz), 139.4, 137.3, 135.2,

132.0, 131.9, 129.6, 128.8, 127.9, 127.5, 126.4, 126.2, 26.8 (t, $J_{C\text{-}F}$ = 1.0 Hz), 22.9 (m) ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{21}H_{13}ClF_7N_2$ $[M+H]^+$ 461.0650, found: 461.0657.



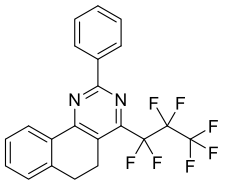
2-(4-Fluorophenyl)-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3ac):

Yield = 49% (63 mg). White solid. M.p. 114.6–116.6 °C.

IR (KBr): ν = 2936, 1613, 1397, 931, 775 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.63 – 8.57 (m, 2H), 8.56 – 8.53 (m, 1H), 7.52 – 7.43 (m, 2H), 7.32 – 7.29 (m, 1H), 7.23 – 7.16 (m, 2H), 3.21 – 3.13 (m, 2H), 2.99 (dd, J = 8.5, 6.0 Hz, 2H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.65 (t, J = 9.5 Hz, 3F), -109.52 – -109.59 (m, 1F), -110.11 – -110.24 (m, 2F), -125.47 (d, J = 4.7 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 166.1, 163.6, 162.5, 161.1 (d, $J_{C\text{-}F}$ = 0.9 Hz), 152.1 (t, $J_{C\text{-}F}$ = 26.1 Hz) 139.4, 132.9 (d, $J_{C\text{-}F}$ = 2.9 Hz), 132.1, 131.9, 130.4 (d, $J_{C\text{-}F}$ = 8.7 Hz), 127.7 (d, $J_{C\text{-}F}$ = 39.22 Hz), 126.4, 125.8 (d, $J_{C\text{-}F}$ = 0.6 Hz), 115.6 (d, $J_{C\text{-}F}$ = 21.6 Hz), 26.9, 22.8 (m) ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{21}H_{13}F_8N_2$ $[M+H]^+$ 445.0946, found: 445.0951.



4-(Perfluoropropyl)-2-phenyl-5,6-dihydrobenzo[*h*]quinazoline (3ad):

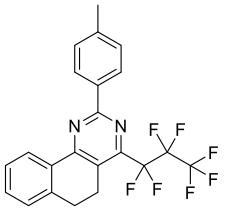
Yield = 53% (68 mg). White solid. M.p. 129.1–130.6 °C.

IR (KBr): ν = 2936, 1622, 931, 731 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.64 – 8.55 (m, 3H), 7.54 – 7.49 (m, 3H), 7.50 – 7.42 (m, 2H), 7.31 – 7.27 (m, 1H), 3.18 (t, J = 7.2 Hz, 2H), 2.99 (dd, J = 8.4, 6.1 Hz, 2H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.64 (t, J = 9.5 Hz, 3F), -110.13 (q, J = 9.5 Hz, 2F), -125.45 (d, J = 4.6 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 162.4, 162.0 (t, $J_{C\text{-}F}$ = 1.7 Hz), 152.0 (t, $J_{C\text{-}F}$ = 24.0 Hz),

139.4, 136.7, 132.2, 131.8, 131.0, 128.6, 128.3, 127.8, 127.5, 126.4, 125.9, 26.9, 22.8 (m) ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{21}H_{14}F_7N_2$ $[M+H]^+$ 427.1040, found: 427.1038.



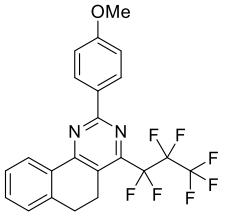
4-(Perfluoropropyl)-2-(*p*-tolyl)-5,6-dihydrobenzo[h]quinazoline (3ae):

Yield = 50% (60 mg). White solid. M.p. 90.4–92.3 °C.

IR (KBr): ν = 2954, 1613, 1553, 931, 792 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.59 – 8.53 (m, 1H), 8.47 (d, J = 8.2 Hz, 2H), 7.49 – 7.40 (m, 2H), 7.31 (d, J = 8.0 Hz, 2H), 7.29 – 7.25 (m, 1H), 3.15 (t, J = 7.1 Hz, 2H), 3.00 – 2.93 (m, 2H), 2.44 (s, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.77 (t, J = 9.9 Hz, 3F), -110.27 (q, J = 10.8 Hz, 2F), -125.59 (d, J = 4.7 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 162.3, 162.1 (t, $J_{C\text{-}F}$ = 1.0 Hz), 152.0 (t, $J_{C\text{-}F}$ = 24.4 Hz), 141.4, 139.4, 134.1, 132.3, 131.7, 129.3, 128.2, 127.8, 127.4, 126.4, 125.5, 26.9, 22.8 (m), 21.5 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{22}H_{16}F_7N_2$ $[M+H]^+$ 441.1196, found: 441.1202.



2-(4-Methoxyphenyl)-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3af):

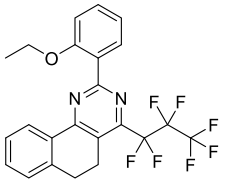
Yield = 56% (77 mg). Light brown solid. M.p. 97.0–99.0 °C.

IR (KBr): ν = 2945, 1648, 1622, 931, 792 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.64 – 8.49 (m, 3H), 7.50 – 7.42 (m, 2H), 7.31 – 7.27 (m, 1H), 7.06 – 7.01 (m, 2H), 3.90 (s, 3H), 3.20 – 3.12 (m, 2H), 3.01 – 2.94 (m, 2H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.64 (t, J = 10.0 Hz, 3F), -110.16 (q, J = 10.3 Hz, 2F), -125.47 (d, J = 4.3 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 162.2, 162.1, 161.8 (t, $J_{C\text{-}F}$ = 1.2 Hz), 151.9 (t, $J_{C\text{-}F}$ = 23.2 Hz), 139.4, 132.3, 131.6, 129.9, 129.5, 127.8, 127.4, 126.3, 125.0, 113.9, 55.3, 26.9, 22.8 (m)

ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{22}H_{16}F_7N_2O$ $[M+H]^+$ 457.1145, found: 457.1151.



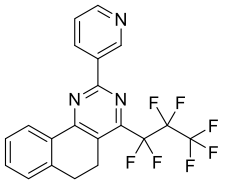
2-(2-Ethoxyphenyl)-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3ag):

Yield = 72% (102 mg). Yellow solid. M.p. 87.6–88.9 °C.

IR (KBr): ν = 2842, 1622, 1562, 922, 766 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.42 (dd, J = 7.6, 1.4 Hz, 1H), 7.85 (dd, J = 7.6, 1.8 Hz, 1H), 7.40 – 7.30 (m, 3H), 7.23 – 7.19 (m, 1H), 7.03 – 6.96 (m, 2H), 4.08 (q, J = 7.0 Hz, 2H), 3.14 – 3.08 (m, 2H), 2.91 (dd, J = 8.5, 6.0 Hz, 2H), 1.34 (t, J = 7.0 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -80.20 – -80.81 (m, 3F), -110.70 – -111.69 (m, 2F), -126.21 (s, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 163.4, 162.2, 157.8, 151.5 (t, $J_{C\text{-}F}$ = 23.4 Hz), 139.3, 132.3, 132.0, 131.6, 131.3, 127.7, 127.6, 127.4, 126.6, 125.4, 120.6, 113.6, 64.5, 26.9, 22.9 (m), 14.8 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{23}H_{18}F_7N_2O$ $[M+H]^+$ 471.1302, found: 471.1307.



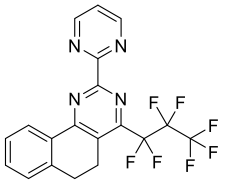
4-(Perfluoropropyl)-2-(pyridin-3-yl)-5,6-dihydrobenzo[*h*]quinazoline (3ah):

Yield = 44% (56 mg). Yellow solid. M.p. 109.4–111.0 °C.

IR (KBr): ν = 2901, 1609, 1553, 931, 749 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.69 (s, 1H), 8.73 – 8.70 (m, 1H), 8.66 (d, J = 3.6 Hz, 1H), 8.45 – 8.43 (m, 1H), 7.42 – 7.34 (m, 3H), 7.22 – 7.18 (m, 1H), 3.10 (t, J = 7.2 Hz, 2H), 2.93 – 2.87 (m, 2H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.24 – -80.12 (m, 3F), -109.43 – -110.91 (m, 2F), -125.47 (d, J = 16.2 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 162.7, 160.2, 152.2 (t, $J_{C\text{-}F}$ = 25.3 Hz), 151.5, 149.8, 139.4, 135.5, 132.2 (m), 132.1, 131.7, 127.9, 127.5, 126.8, 126.4, 123.4 (m), 26.7, 22.8 (m) ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{20}H_{13}F_7N_3$ $[M+H]^+$ 428.0992, found: 428.0998.



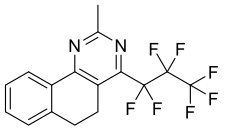
4-(Perfluoropropyl)-2-(pyrimidin-2-yl)-5,6-dihydrobenzo[*h*]quinazoline (3ai):

Yield = 18% (23 mg). Brown solid. M.p. 162.6–164.3 °C.

IR (KBr): ν = 2954, 1553, 939, 887 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.99 (d, J = 4.9 Hz, 2H), 8.52 (dd, J = 7.6, 1.4 Hz, 1H), 7.44 – 7.34 (m, 3H), 7.25 – 7.21 (m, 1H), 3.18 (t, J = 7.2 Hz, 2H), 2.97 – 2.91 (m, 2H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.05 – -80.25 (m, 3F), -109.74 – -111.73 (m, 2F), -125.25 (d, J = 17.1 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 163.7, 162.5, 160.9, 160.8, 158.0, 139.4, 132.2, 131.6, 129.0, 127.7, 127.7, 127.2, 121.3, 26.7, 23.4 (m) ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{19}H_{12}F_7N_4$ $[M+H]^+$ 429.0945, found: 429.0950.



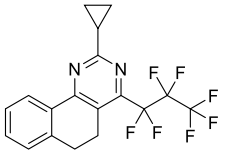
2-Methyl-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3aj):

Yield = 42 % (46 mg). Brown solid. M.p. 80.4–82.4 °C.

IR (KBr): ν = 2936, 1605, 1562, 740, 602 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.39 (dd, J = 7.6, 1.5 Hz, 1H), 7.47 – 7.38 (m, 2H), 7.29 – 7.26 (m, 1H), 3.13 – 3.07 (m, 2H), 2.93 (dd, J = 8.6, 5.9 Hz, 2H), 2.82 (s, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.76 (t, J = 9.5 Hz, 3F), -110.71 (q, J = 9.5 Hz, 2F), -125.59 (d, J = 4.6 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 166.0 (t, $J_{C\text{-}F}$ = 1.0 Hz), 162.3, 151.4 (t, $J_{C\text{-}F}$ = 23.7 Hz), 139.4, 131.9, 131.7, 127.8, 127.5, 126.3, 125.3, 26.9 (t, $J_{C\text{-}F}$ = 1.4 Hz), 25.8, 22.8 (m) ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{16}H_{12}F_7N_2$ $[M+H]^+$ 365.0883, found: 365.0889.



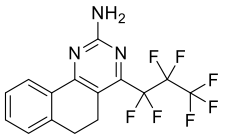
2-Cyclopropyl-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3ak):

Yield = 57% (67 mg). Yellow oil.

IR (KBr): ν = 2936, 1605, 1562, 749 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.36 (dd, J = 7.5, 1.3 Hz, 1H), 7.45 – 7.35 (m, 2H), 7.27 – 7.22 (m, 1H), 3.07 (t, J = 7.0 Hz, 2H), 2.95 – 2.87 (m, 2H), 2.38 – 2.32 (m, 1H), 1.25 – 1.20 (m, 2H), 1.14 – 1.07 (m, 2H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.71 (t, J = 9.4 Hz, 3F), -110.44 (q, J = 9.4 Hz, 2F), -125.58 (s, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 169.9 (t, $J_{C\text{-}F}$ = 0.8 Hz), 161.9, 151.4 (t, $J_{C\text{-}F}$ = 23.5 Hz), 139.4, 132.1, 131.5, 127.7, 127.3, 126.2, 124.6, 27.0, 22.6 (m), 17.9, 10.9 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{18}H_{14}F_7N_2$ $[M+H]^+$ 391.1040, found: 391.1045.



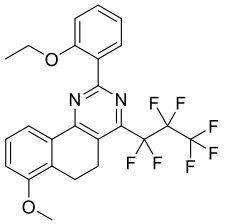
4-(Perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazolin-2-amine (3al):

Yield = 34% (37 mg). Yellow solid. M.p. 126.7–128.0 ºC.

IR (KBr): ν = 2954, 1640, 1622, 800, 749 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.18 (dd, J = 7.7, 1.4 Hz, 1H), 7.36 – 7.32 (m, 1H), 7.30 – 7.26 (m, 1H), 7.20 – 7.15 (m, 1H), 5.33 (s, 2H), 2.89 (dd, J = 9.9, 4.4 Hz, 2H), 2.80 (dd, J = 8.7, 5.2 Hz, 2H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -78.17 (t, J = 9.9 Hz, 3F), -109.57 (q, J = 10.0 Hz, 2F), -124.06 (d, J = 4.9 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 163.7, 161.2 (t, $J_{C\text{-}F}$ = 1.6 Hz), 152.1 (t, $J_{C\text{-}F}$ = 24.6 Hz), 139.9, 132.0, 131.5, 127.8, 127.2, 126.1, 118.3, 27.5 (t, $J_{C\text{-}F}$ = 1.5 Hz), 22.3 (m) ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{15}H_{11}F_7N_3$ $[M+H]^+$ 366.0836, found: 366.0841.



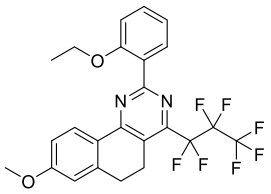
2-(2-Ethoxyphenyl)-7-methoxy-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3bg):

Yield = 42% (63 mg). Yellow solid. M.p. 87.6–88.5 ºC.

IR (KBr): ν = 2842, 1648, 1613, 766, 740 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.05 (d, J = 7.9 Hz, 1H), 7.84 (dd, J = 7.6, 1.8 Hz, 1H), 7.35 – 7.31 (m, 1H), 7.26 (t, J = 8.0 Hz, 1H), 7.01 – 6.91 (m, 3H), 4.07 (q, J = 7.0 Hz, 2H), 3.80 (s, 3H), 3.05 (t, J = 7.2 Hz, 2H), 2.90 (dd, J = 8.6, 6.0 Hz, 2H), 1.33 (t, J = 7.0 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -78.82 – -80.60 (m, 3F), -109.19 – -111.57 (m, 2F), -125.42 (d, J = 16.6 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 163.2 (t, $J_{C\text{-}F}$ = 1.0 Hz), 162.2, 157.7, 156.0, 151.4 (t, $J_{C\text{-}F}$ = 24.5 Hz), 133.3, 132.0, 131.3, 128.1, 127.6, 127.5, 125.4, 120.6, 118.7, 113.6, 113.1, 64.5, 55.6, 22.4 (m), 19.2, 14.8 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{24}H_{20}F_7N_2O_2$ $[M+H]^+$ 501.1408, found: 501.1413.



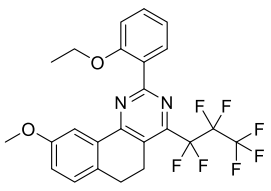
2-(2-Ethoxyphenyl)-8-methoxy-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3cg):

Yield = 87% (131 mg). Yellow oil.

IR (KBr): ν = 2936, 1613, 1553, 757, 740 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.44 (d, J = 8.7 Hz, 1H), 7.92 – 7.89 (m, 1H), 7.42 – 7.38 (m, 1H), 7.10 – 7.01 (m, 2H), 6.91 (dd, J = 8.7, 2.6 Hz, 1H), 6.77 (d, J = 2.5 Hz, 1H), 4.14 (q, J = 7.0 Hz, 2H), 3.86 (s, 3H), 3.15 (t, J = 7.2 Hz, 2H), 2.94 (dd, J = 8.5, 6.0 Hz, 2H), 1.41 (t, J = 7.0 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.40 – -80.21 (m, 3F), -109.83 – -110.86 (m, 2F), -125.42 (d, J = 17.4 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 163.2 (t, $J_{C\text{-}F}$ = 1.2 Hz), 162.4, 162.0, 157.7, 151.0 (t, $J_{C\text{-}F}$ = 24.0 Hz), 141.4, 132.0, 131.2, 128.6, 127.8, 125.2, 124.4, 120.5, 113.6, 113.1, 112.6, 64.5, 55.3, 27.3, 23.0 (m), 14.8 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{24}H_{20}F_7N_2O_2$ $[M+H]^+$ 501.1408, found: 501.1413.



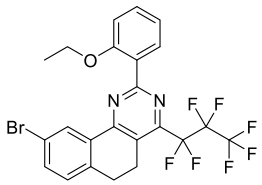
2-(2-Ethoxyphenyl)-9-methoxy-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3dg):

Yield = 37% (56 mg). Yellow oil.

IR (KBr): ν = 2936, 1605, 1562, 809, 757 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.03 (t, J = 3.0 Hz, 1H), 7.92 – 7.89 (m, 1H), 7.45 – 7.40 (m, 1H), 7.22 – 7.17 (m, 1H), 7.11 – 7.04 (m, 2H), 7.03 – 7.00 (m, 1H), 4.19 – 4.13 (m, 2H), 3.89 (d, J = 2.1 Hz, 3H), 3.16 (t, J = 6.6 Hz, 2H), 2.92 (t, J = 6.9 Hz, 2H), 1.43 – 1.39 (m, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.75 (t, J = 9.8 Hz, 3F), -110.30 (q, J = 8.3 Hz, 2F), -125.34 (s, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 163.3 (t, $J_{C\text{-}F}$ = 1.0 Hz), 162.1, 159.0, 157.7, 151.6 (t, $J_{C\text{-}F}$ = 24.1 Hz), 133.2, 132.1, 131.7, 131.3, 128.8, 127.7, 125.7, 120.6, 118.4, 113.6, 110.5, 64.5, 55.5, 26.1, 23.2 (m), 14.8 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{24}H_{20}F_7N_2O_2$ $[M+H]^+$ 501.1408, found: 501.1413.



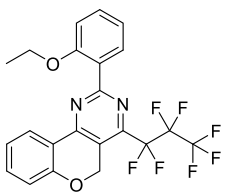
9-Bromo-2-(2-ethoxyphenyl)-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3eg):

Yield = 52% (86 mg). Yellow solid. M.p. 82.4–83.0 °C.

IR (KBr): ν = 2971, 1639, 1613, 809, 757 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.64 (d, J = 1.7 Hz, 1H), 7.97 (d, J = 7.6 Hz, 1H), 7.53 (dd, J = 8.1, 1.8 Hz, 1H), 7.47 – 7.40 (m, 1H), 7.14 (d, J = 8.1 Hz, 1H), 7.11 – 7.04 (m, 2H), 4.17 (q, J = 7.0 Hz, 2H), 3.16 (t, J = 7.2 Hz, 2H), 2.97 – 2.88 (m, 2H), 1.48 (t, J = 6.9 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.71 (t, J = 9.6 Hz, 3F), -110.31 (q, J = 9.5 Hz, 2F), -125.35 (d, J = 4.7 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 163.4 (t, $J_{C\text{-}F}$ = 0.9 Hz), 160.8, 157.9, 151.9 (t, $J_{C\text{-}F}$ = 24.1 Hz), 137.9, 134.3, 134.1, 132.1, 131.6, 129.4, 129.4, 126.8, 125.2, 121.3, 120.5, 113.4, 64.4, 26.3, 22.7 (m), 14.9 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{23}H_{17}BrF_7N_2O$ $[M+H]^+$ 549.0407, found: 549.0395.



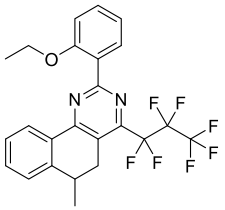
2-(2-Ethoxyphenyl)-4-(perfluoropropyl)-5H-chromeno[4,3-*d*]pyrimidine (3gg):

Yield = 41% (58 mg). Light yellow solid. M.p. 69.0–71.0 °C.

IR (KBr): ν = 2998, 1622, 1613, 766, 731 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.36 (dd, J = 7.8, 1.7 Hz, 1H), 7.92 (dd, J = 7.7, 1.8 Hz, 1H), 7.46 – 7.41 (m, 2H), 7.16 – 7.10 (m, 1H), 7.10 – 6.98 (m, 3H), 5.43 (s, 2H), 4.14 (q, J = 7.0 Hz, 2H), 1.40 (t, J = 6.9 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.80 (t, J = 10.3 Hz, 3F), -111.60 (q, J = 10.0 Hz, 2F), -125.83 (d, J = 16.3 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 165.0, 158.3, 157.8, 157.7, 149.5 (t, $J_{C\text{-}F}$ = 25.2 Hz), 134.1, 132.1, 131.7, 127.1, 126.1, 122.7, 120.9, 120.6, 119.6, 117.2, 113.5, 64.5, 63.9 (m), 14.7 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{22}H_{16}F_7N_2O_2$ $[M+H]^+$ 473.1095, found: 473.1100.



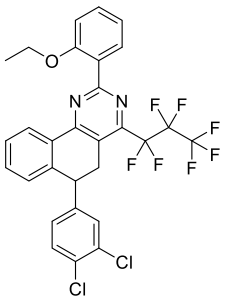
2-(2-Ethoxyphenyl)-6-methyl-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3hg):

Yield = 31% (45 mg). Yellow oil.

IR (KBr): ν = 2971, 1604, 1570, 757, 731 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.52 (dd, J = 7.7, 1.1 Hz, 1H), 7.96 (dd, J = 7.6, 1.8 Hz, 1H), 7.50 – 7.46 (m, 1H), 7.45 – 7.37 (m, 2H), 7.32 (d, J = 7.5 Hz, 1H), 7.12 – 7.03 (m, 2H), 4.24 – 4.11 (m, 2H), 3.27 – 3.06 (m, 3H), 1.43 (t, J = 7.0 Hz, 3H), 1.26 (d, J = 6.6 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.77 (t, J = 9.6 Hz, 3F), -109.14 – -111.14 (m, 2F), -125.32 (d, J = 4.5 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 163.3 (t, $J_{C\text{-}F}$ = 1.1 Hz), 161.8, 157.8, 152.3 (t, $J_{C\text{-}F}$ = 21.8 Hz), 144.3, 132.1, 131.9, 131.4, 131.2, 127.5, 127.5, 127.2, 126.8, 126.5, 124.2, 120.6, 113.6, 64.5, 31.4, 30.3 (m), 20.4, 14.8 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{24}H_{20}F_7N_2O$ $[M+H]^+$ 485.1458, found: 485.1464.



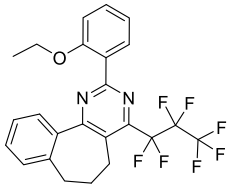
6-(3,4-Dichlorophenyl)-2-(2-ethoxyphenyl)-4-(perfluoropropyl)-5,6-dihydrobenzo[*h*]quinazoline (3ig):

Yield = 22% (41 mg). Yellow solid. M.p. 129–130 °C.

IR (KBr): ν = 2920, 1597, 1544, 792, 740 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.66 – 8.60 (m, 1H), 7.96 (dd, J = 7.6, 1.8 Hz, 1H), 7.51 – 7.42 (m, 3H), 7.36 (d, J = 8.3 Hz, 1H), 7.22 (d, J = 2.1 Hz, 1H), 7.11 – 7.01 (m, 3H), 6.96 (dd, J = 8.3, 2.1 Hz, 1H), 4.31 (t, J = 6.9 Hz, 1H), 4.20 – 4.14 (m, 2H), 3.46 (d, J = 7.0 Hz, 2H), 1.43 (t, J = 7.0 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.76 (t, J = 10.0 Hz, 3F), -108.94 – -111.50 (m, 2F), -125.44 (s, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 163.7, 161.7, 157.8, 150.2 (t, $J_{C\text{-}F}$ = 22.0 Hz), 142.1, 140.1, 132.8, 132.3, 132.2, 132.1, 131.6, 131.2, 130.6, 130.1, 128.2, 128.0, 127.5, 127.1, 127.0, 123.2, 120.6, 113.5, 64.5, 42.0, 30.9 (m), 14.8 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{29}H_{20}Cl_2F_7N_2O$ $[M+H]^+$ 615.0835, found: 615.0841.



2-(2-Ethoxyphenyl)-4-(perfluoropropyl)-6,7-dihydro-5*H*-benzo[6,7]cyclohepta[1,2-*d*]pyrimidine (3jg):

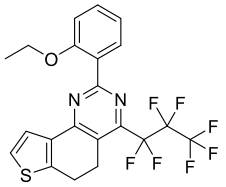
Yield = 46% (67 mg). Yellow oil.

IR (KBr): ν = 2945, 1666, 1640, 766, 740 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.98 – 7.95 (m, 1H), 7.94 – 7.89 (m, 1H), 7.49 – 7.38 (m, 3H), 7.31 – 7.24 (m, 1H), 7.09 – 7.01 (m, 2H), 4.20 – 4.12 (m, 2H), 2.71 (t, J = 6.4 Hz, 2H), 2.61 (t, J = 7.0 Hz, 2H), 2.36 – 2.29 (m, 2H), 1.47 – 1.43 (m, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.68 (t, J = 10.5 Hz, 3F), -108.24 – -108.97 (m, 2F), -124.56 (s, 2F) ppm. **^{13}C NMR** (100 MHz,

$CDCl_3$): δ = 169.6, 163.1, 157.7, 149.7 (t, $J_{C\text{-}F}$ = 23.0 Hz), 139.8, 137.7, 132.2, 131.4, 130.7, 129.6, 128.7, 128.6, 127.2, 127.1, 120.5, 113.4, 64.4, 32.9, 30.8, 24.9 (m), 14.8 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{24}H_{20}F_7N_2O$ $[M+H]^+$ 485.1458, found: 485.1464.



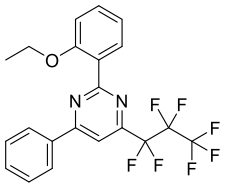
2-(2-Ethoxyphenyl)-4-(perfluoropropyl)-5,6-dihydrothieno[2,3-*h*]quinazoline (3kg):

Yield = 45% (64 mg). Yellow oil.

IR (KBr): ν = 2936, 1605, 1570, 853, 749 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.87 (dd, J = 7.6, 1.7 Hz, 1H), 7.75 (d, J = 5.2 Hz, 1H), 7.44 – 7.37 (m, 1H), 7.20 (d, J = 5.2 Hz, 1H), 7.10 – 7.01 (m, 2H), 4.13 (q, J = 7.0 Hz, 2H), 3.30 (t, J = 7.5 Hz, 2H), 3.11 (t, J = 7.6 Hz, 2H), 1.39 (t, J = 7.0 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.76 (t, J = 9.6 Hz, 3F), -110.51 (tq, J = 9.6, 4.9 Hz, 2F), -125.44 (d, J = 4.7 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 163.6 (t, $J_{C\text{-}F}$ = 1.3 Hz), 159.8, 157.7, 150.9 (t, $J_{C\text{-}F}$ = 24.4 Hz), 145.9, 135.2, 131.9, 131.3, 127.7, 125.0, 123.7, 122.9, 120.6, 113.7, 64.6, 22.9 (m), 22.7, 14.7 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{21}H_{16}F_7N_2OS$ $[M+H]^+$ 477.0866, found: 477.0872.



2-(2-Ethoxyphenyl)-4-(perfluoropropyl)-6-phenylpyrimidine (3lg):

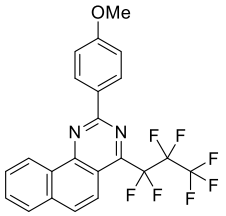
Yield = 58% (77mg). Yellow oil.

IR (KBr): ν = 3411, 2979, 1585, 1374, 1234, 753 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.50 (s, 1H), 8.44 (dd, J = 8.0, 1.5 Hz, 2H), 7.80 (dd, J = 7.6, 1.7 Hz, 1H), 7.68–7.58 (m, 3H), 7.57–7.50 (m, 1H), 7.22 (d, J = 8.2 Hz, 1H), 7.12 (t, J = 7.1 Hz, 1H), 4.13 (q, J = 6.9 Hz, 2H), 1.28 (t, J = 6.9 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, DMSO-d_6): δ = -79.69 (t, J = 9.0 Hz, 3F), -115.42 (q, J = 9.0 Hz, 2F), -125.67 (s, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 166.3, 165.9, 157.9, 156.2 (t, $J_{C\text{-}F}$ = 26.0 Hz), 136.0, 132.2, 131.7, 131.7, 129.0, 127.5,

127.4, 120.6, 113.5, 111.2 (t, $J_{C\text{-}F}$ = 5.0 Hz), 64.5, 14.7 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{21}H_{16}F_7N_2O$ $[M+H]^+$ 445.1145, found: 445.1133.



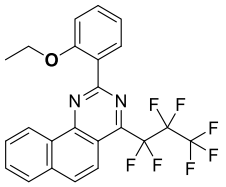
2-(4-Methoxyphenyl)-4-(perfluoropropyl)benzo[*h*]quinazoline (5af):

Yield = 79% (36 mg). White solid. M.p. 121.6–123.0 °C.

IR (KBr): ν = 2833, 1648, 1579, 809, 775 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.42 – 9.35 (m, 1H), 8.72 – 8.63 (m, 2H), 8.04 – 7.97 (m, 1H), 7.88 – 7.82 (m, 1H), 7.81 – 7.73 (m, 3H), 7.06 – 6.98 (m, 2H), 3.90 (s, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.52 (t, J = 10.0 Hz, 3F), -107.99 (q, J = 10.4 Hz, 2F), -124.68 (d, J = 6.5 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 164.1, 162.3, 159.2 (t, $J_{C\text{-}F}$ = 1.2 Hz), 153.3, 134.9, 130.8, 130.4, 130.2, 129.7, 129.2 (t, $J_{C\text{-}F}$ = 1.4 Hz), 127.8, 127.8, 125.3, 119.9 (m), 118.4, 114.0, 55.4 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{22}H_{14}F_7N_2O$ $[M+H]^+$ 455.0989, found: 455.0994.



2-(2-Ethoxyphenyl)-4-(perfluoropropyl)benzo[*h*]quinazoline (5ag):

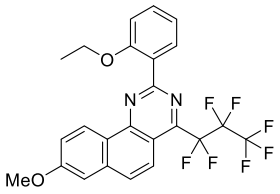
Yield = 86% (40 mg). Light yellow solid. M.p. 130.7–132.5 °C.

IR (KBr): ν = 2989, 1657, 1613, 792, 740 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.47 – 9.41 (m, 1H), 8.14 (dd, J = 7.7, 1.8 Hz, 1H), 8.11 – 8.05 (m, 1H), 7.94 – 7.87 (m, 2H), 7.85 – 7.73 (m, 2H), 7.49 – 7.45 (m, 1H), 7.18 – 7.08 (m, 2H), 4.20 (q, J = 7.0 Hz, 2H), 1.44 (t, J = 7.0 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.60 (t, J = 10.0 Hz, 3F), -107.52 – -109.19 (m, 2F), -124.52 (q, J = 7.1, 6.0 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 160.7 (t, $J_{C\text{-}F}$ = 10.9 Hz), 158.1, 153.3, 152.6 (t, $J_{C\text{-}F}$ = 24.2 Hz), 134.7, 132.6, 131.6, 130.8, 130.4, 129.9 (t, $J_{C\text{-}F}$ = 1.3 Hz), 127.9, 127.8, 127.6, 125.6, 120.7, 119.9 (m), 118.4, 113.6,

64.5, 14.8 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{23}H_{16}F_7N_2O$ $[M+H]^+$ 469.1145, found: 469.1151.



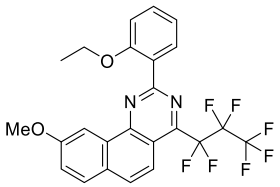
2-(2-Ethoxyphenyl)-8-methoxy-4-(perfluoropropyl)benzo[*h*]quinazoline (5cg):

Yield = 93% (46 mg). Yellow solid. M.p. 106.9–108.5 °C.

IR (KBr): ν = 2936, 1613, 1544, 922, 853 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.34 (d, J = 9.1 Hz, 1H), 8.11 (dd, J = 7.7, 1.8 Hz, 1H), 8.10 – 8.06 (m, 1H), 7.84 (d, J = 9.3 Hz, 1H), 7.49 – 7.45 (m, 1H), 7.38 (dd, J = 9.1, 2.6 Hz, 1H), 7.28 (d, J = 2.6 Hz, 1H), 7.16 – 7.08 (m, 2H), 4.20 (q, J = 7.0 Hz, 2H), 4.01 (s, 3H), 1.43 (t, J = 7.0 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.64 (t, J = 10.1 Hz, 3F), -107.76 – -109.40 (m, 2F), -124.63 (s, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 161.8, 160.7 (t, $J_{C\text{-}F}$ = 0.9 Hz), 158.1, 153.1, 136.7, 132.6, 131.5, 129.4 (t, $J_{C\text{-}F}$ = 1.4 Hz), 127.8, 127.6, 124.6, 120.7, 118.4, 117.4, 116.3 (m), 113.6, 107.9, 64.5, 55.6, 14.8 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{24}H_{18}F_7N_2O_2$ $[M+H]^+$ 499.1251, found: 499.1256.



2-(2-Ethoxyphenyl)-9-methoxy-4-(perfluoropropyl)benzo[*h*]quinazoline (5dg):

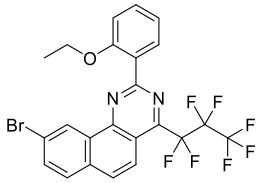
Yield = 84% (42 mg). Light yellow solid. M.p. 102.6–105.0 °C.

IR (KBr): ν = 2989, 1613, 1553, 835, 809 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.81 (d, J = 2.6 Hz, 1H), 8.06 (dd, J = 7.6, 1.8 Hz, 1H), 7.97 (d, J = 9.2 Hz, 1H), 7.86 (dd, J = 9.0, 6.0 Hz, 2H), 7.52 – 7.42 (m, 2H), 7.18 – 7.08 (m, 2H), 4.20 (q, J = 7.0 Hz, 2H), 4.05 (s, 3H), 1.41 (t, J = 7.0 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.63 (t, J = 9.7 Hz, 3F), -108.40 – -108.50 (m, 2F), -124.53 (d, J = 4.8 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 160.2 (t, $J_{C\text{-}F}$ = 1.2 Hz), 159.4, 157.9, 152.5, 132.6, 132.0, 131.4, 129.6, 129.5, 129.4, 128.0, 121.8, 120.7, 118.9, 117.4 (m), 113.6, 105.3, 64.5, 55.7, 14.8 ppm; carbons corresponding to

the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{24}H_{18}F_7N_2O_2$ $[M+H]^+$ 499.1251, found: 499.1256.



9-Bromo-2-(2-ethoxyphenyl)-4-(perfluoropropyl)benzo[*h*]quinazoline (5eg):

Yield = 92% (50 mg). Light yellow solid. M.p. 136.6–138.0 °C.

IR (KBr): ν = 2998, 1631, 1622, 835, 749 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.60 (d, J = 2.1 Hz, 1H), 8.21 (dd, J = 7.7, 1.8 Hz, 1H), 8.12 – 8.08 (m, 1H), 7.91 (dd, J = 8.5, 2.1 Hz, 1H), 7.87 (d, J = 9.2 Hz, 1H), 7.80 (d, J = 8.5 Hz, 1H), 7.52 – 7.47 (m, 1H), 7.19 – 7.09 (m, 2H), 4.23 (q, J = 7.0 Hz, 2H), 1.52 (t, J = 7.0 Hz, 3H) ppm. **^{19}F NMR** (376 MHz, $CDCl_3$): δ = -79.61 (t, J = 9.7 Hz, 3F), -108.36 – -108.50 (m, 2F), -124.59 (d, J = 5.5 Hz, 2F) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 160.7 (t, $J_{C\text{-}F}$ = 1.1 Hz), 158.3, 152.9, 152.1, 134.0, 133.2, 132.7, 132.0, 131.7, 129.4, 129.1 (t, $J_{C\text{-}F}$ = 1.3 Hz), 128.3, 126.8, 122.5, 120.7, 120.4 (m), 118.5, 113.4, 64.5, 14.9 ppm; carbons corresponding to the C_3F_7 group cannot be identified due to C-F coupling.

HRMS m/z: calcd for $C_{23}H_{15}BrF_7N_2O$ $[M+H]^+$ 547.0250, found: 547.0238.

11. References

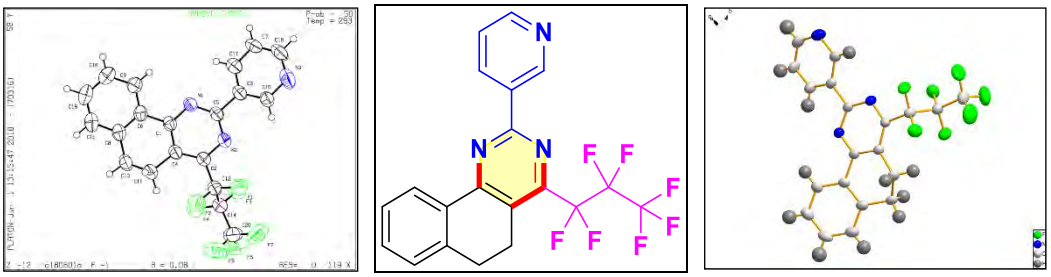
[1] X. Su, H. Huang, Y. Yuan, and Y. Li, *Angew. Chem., Int. Ed.* **2017**, *56*, 1338–1341.

[2] S. Liu, J. Jie, J. Yu, X. Yang, *Adv. Synth. Catal.* **2018**, *360*, 267–271.

[3] Y. Yang, E. G. Moschetta, R. M. Rioux, *ChemCatChem.* **2013**, *5*, 3005–3013.

[4] A. Herrera, R. Martínez-Alvarez, M. Chioua, R. Chatt, R. Chioua, A. Sánchez, J. Almy, *Tetrahedron* **2006**, *62*, 2799–2811.

12. The X-ray crystal structure of product 3ah



4-(Perfluoropropyl)-2-(pyridin-3-yl)-5,6-dihydrobenzo[*h*]quinazoline (3ah)

Crystal Number: CCDC 1847355

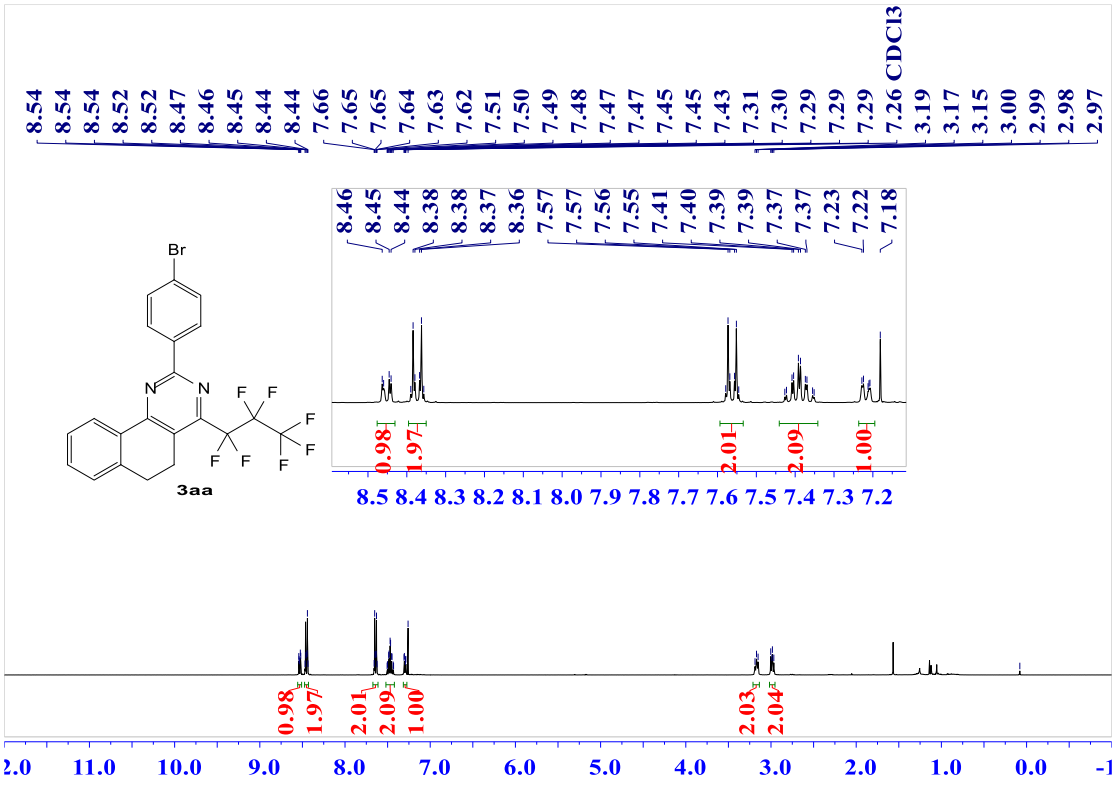
Empirical formula: $C_{20}H_{12}F_7N_3$

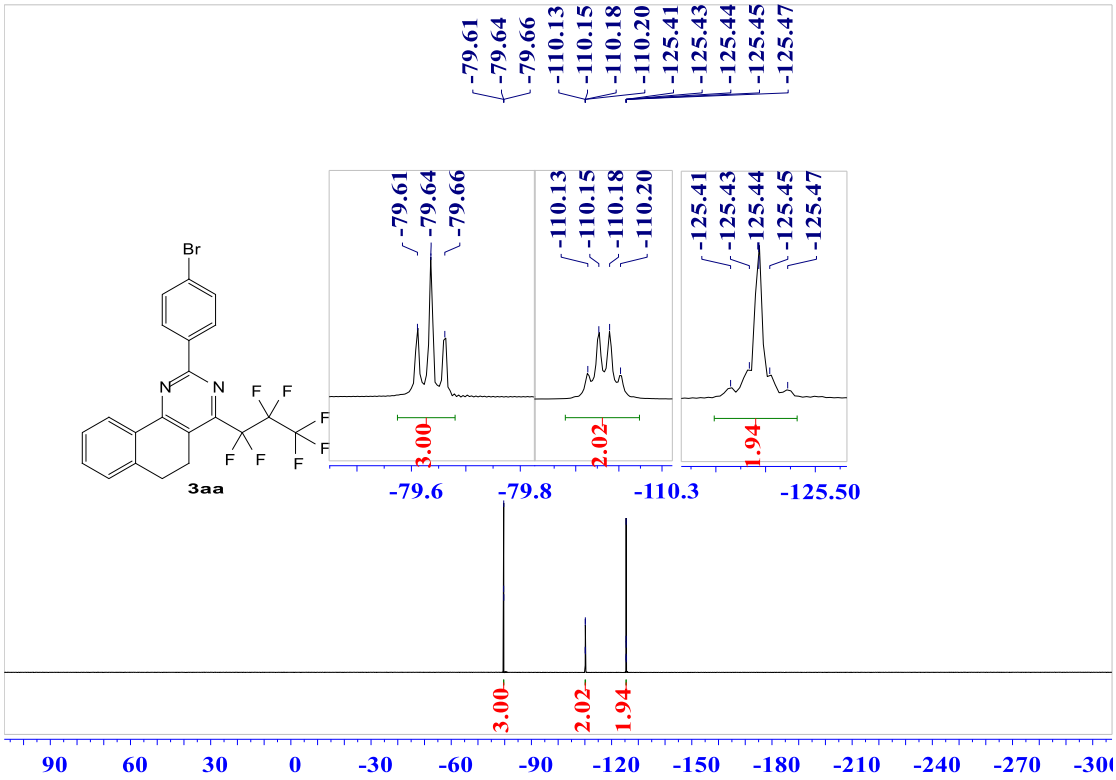
Formula weight: 427.3258

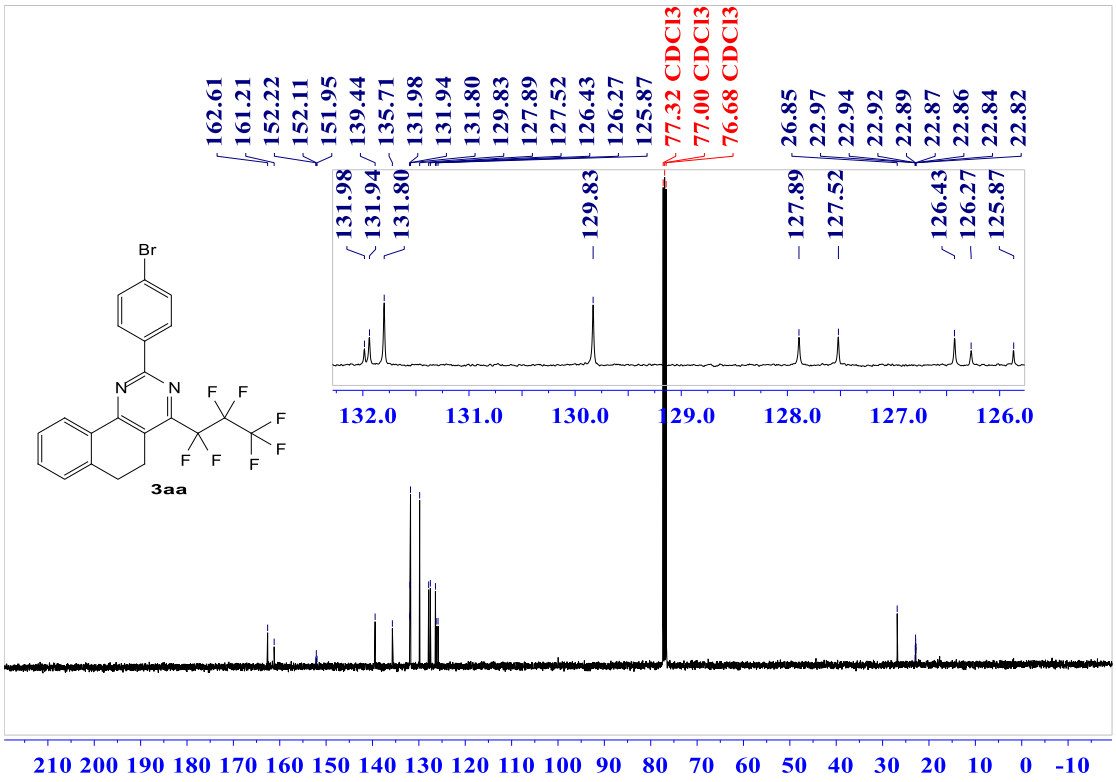
Space Group: P 1 (2)

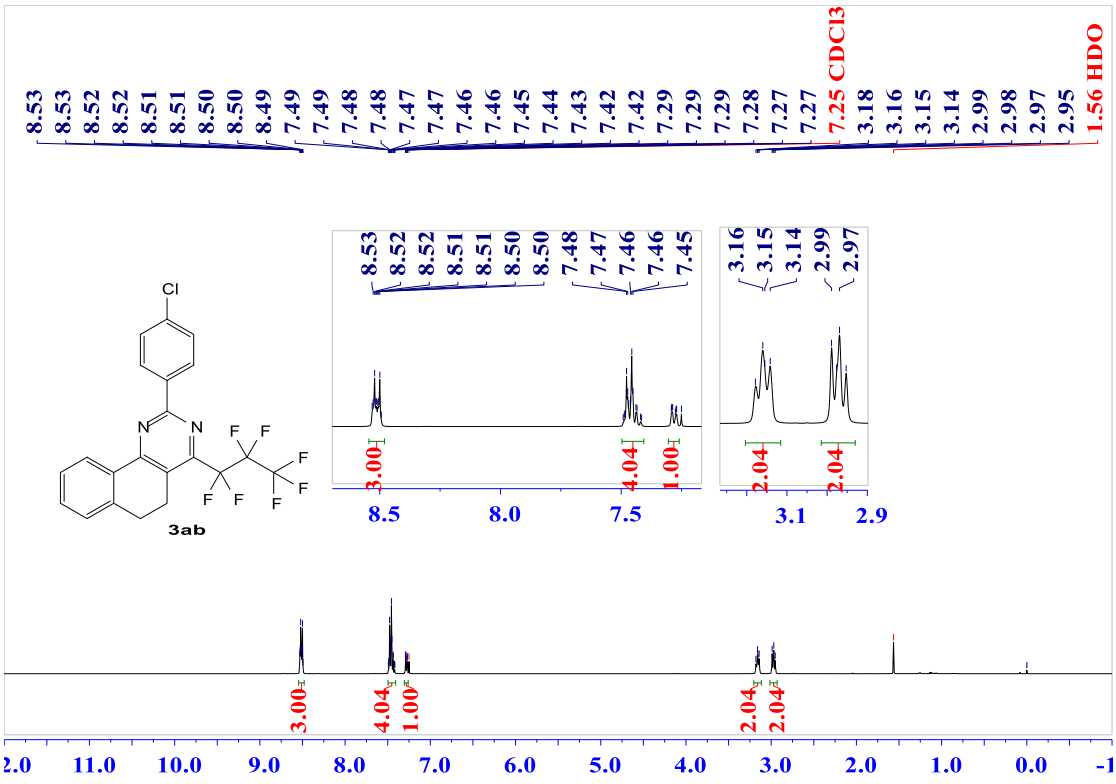
Cell: a 8.5757(7)Å b 10.7240(9)Å c 11.5979(7)Å, α 64.620(7)° β 77.985(7)° γ 71.245(7)°

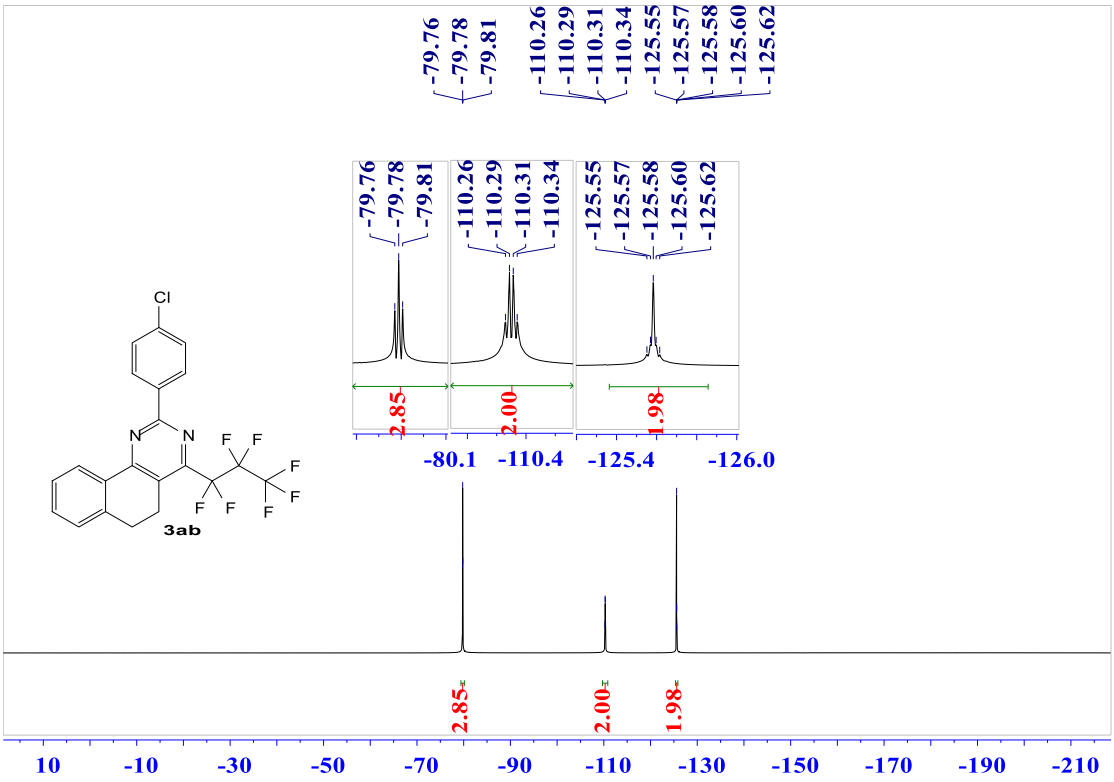
13. The ^{1}H , ^{19}F, ^{13}C spectra of products 3-5:

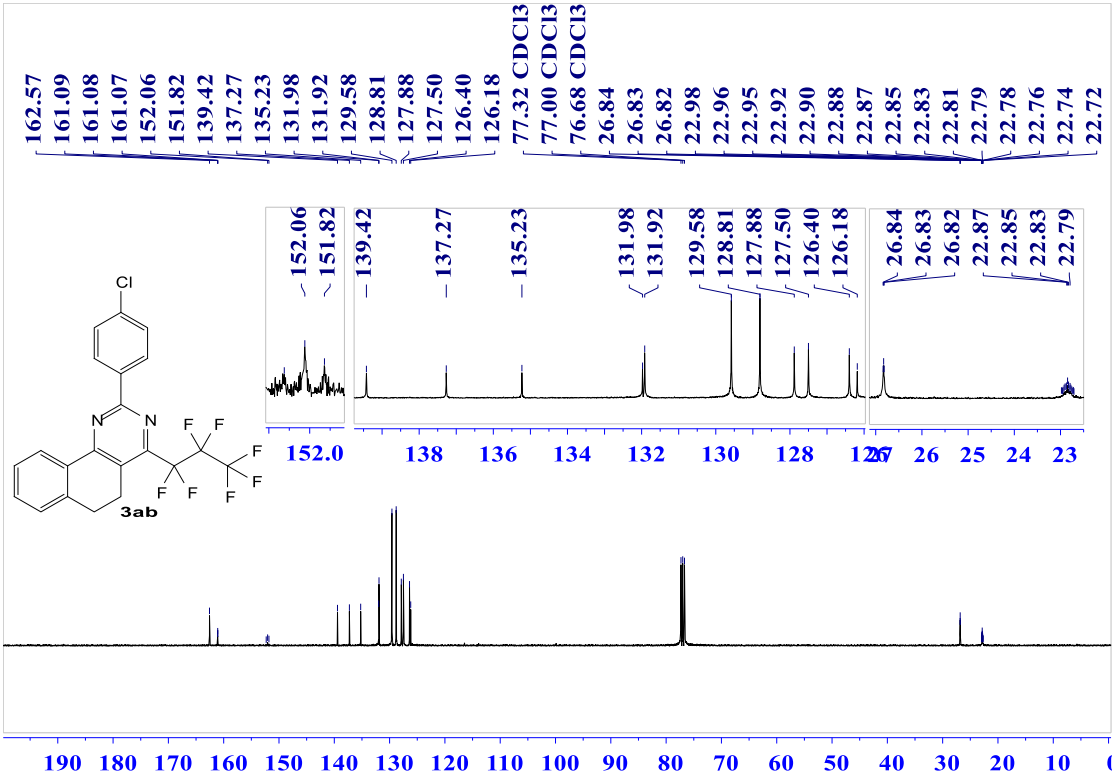


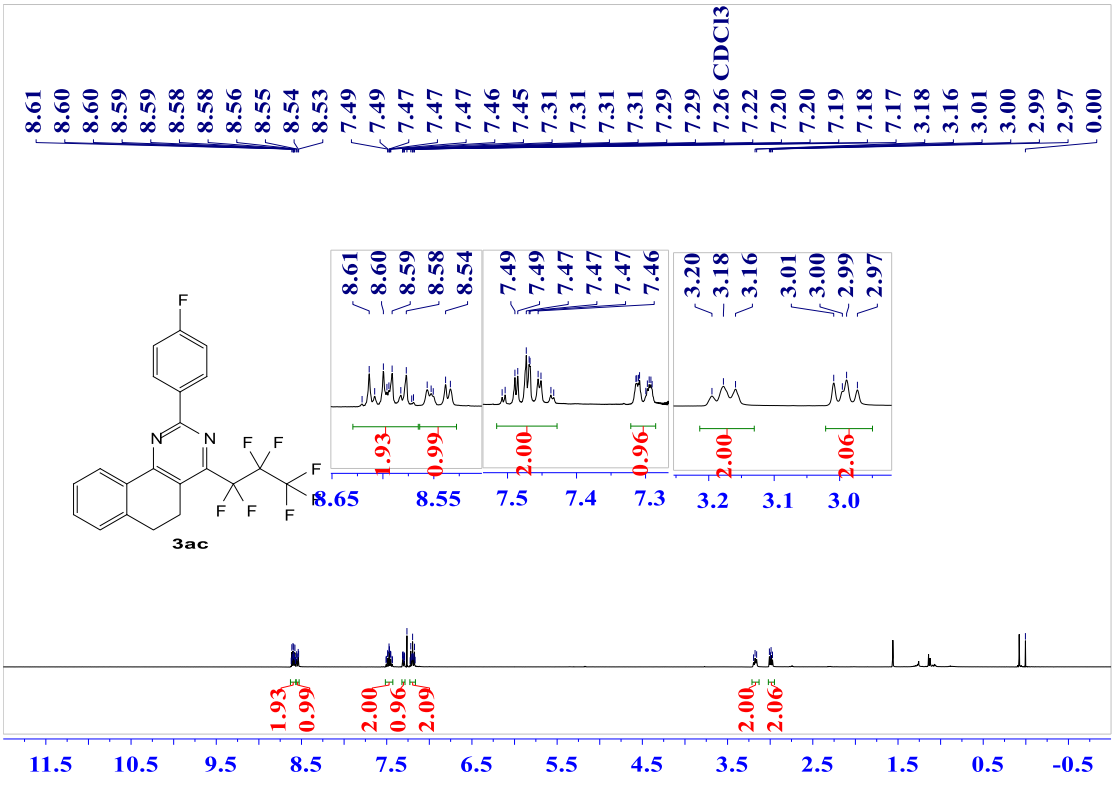


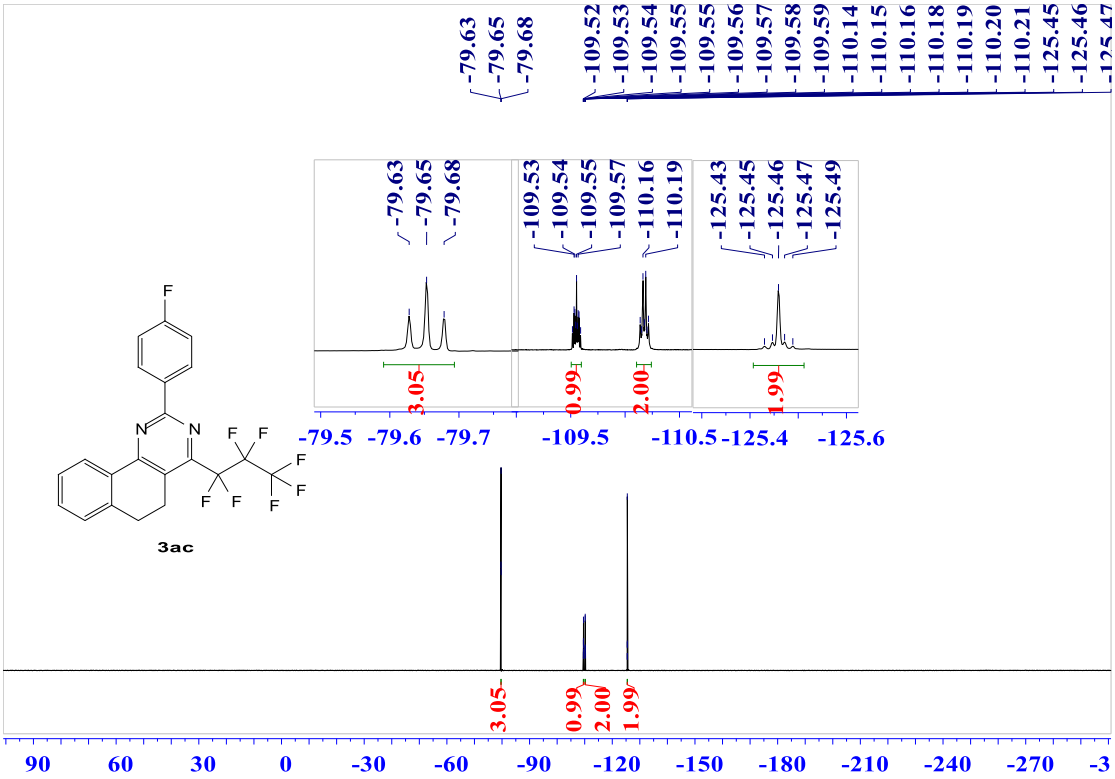

162.61
161.21
152.22
152.11
151.95
139.44
135.71
131.98
131.94
131.80
129.83
127.89
127.52
126.43
126.27
125.87
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
26.85
22.97
22.94
22.92
22.89
22.87
22.86
22.84
22.82
Br
N
N
F
F
F
F
F
F
3aa
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

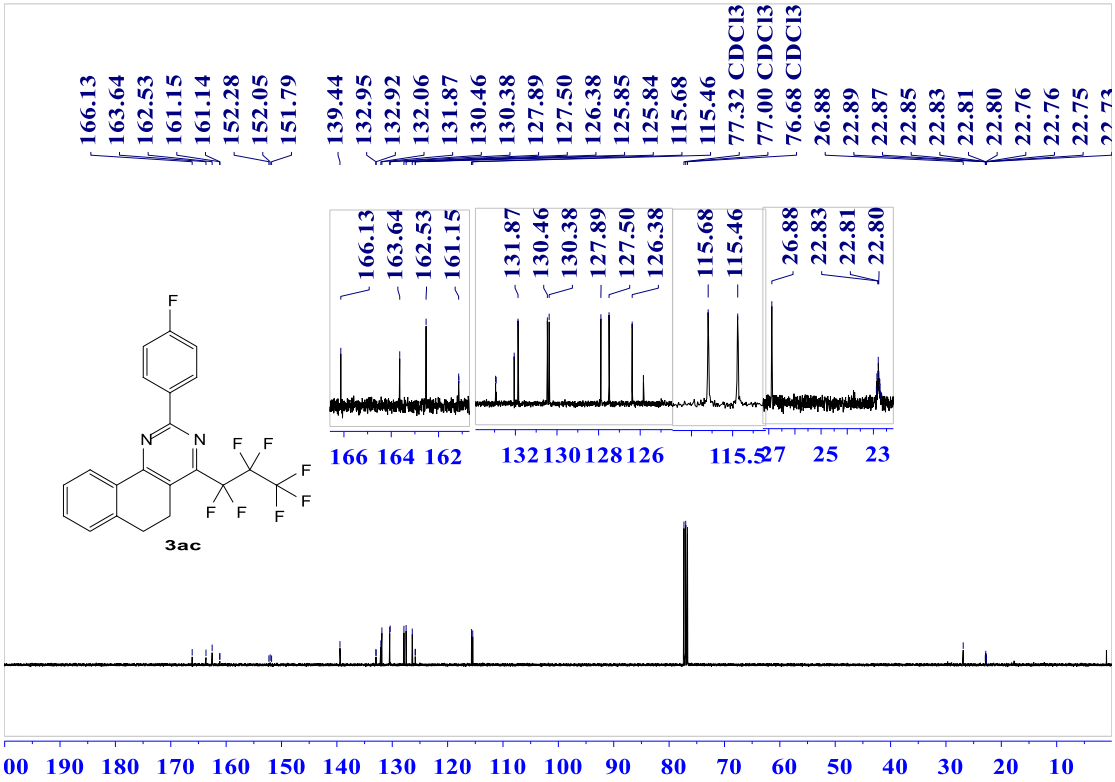

8.53
8.53
8.52
8.52
8.51
8.51
8.50
8.50
8.49
7.49
7.49
7.48
7.48
7.47
7.47
7.46
7.46
7.45
7.44
7.43
7.42
7.42
7.29
7.29
7.29
7.28
7.27
7.27
7.25 CDCl3
3.18
3.16
3.15
3.14
2.99
2.98
2.97
2.95
1.56 HDO
Cl
N
N
F
F
F
F
F
F
3ab
3.00
4.04
1.00
2.04
2.04
8.5
8.0
7.5
3.1
2.9
2.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1

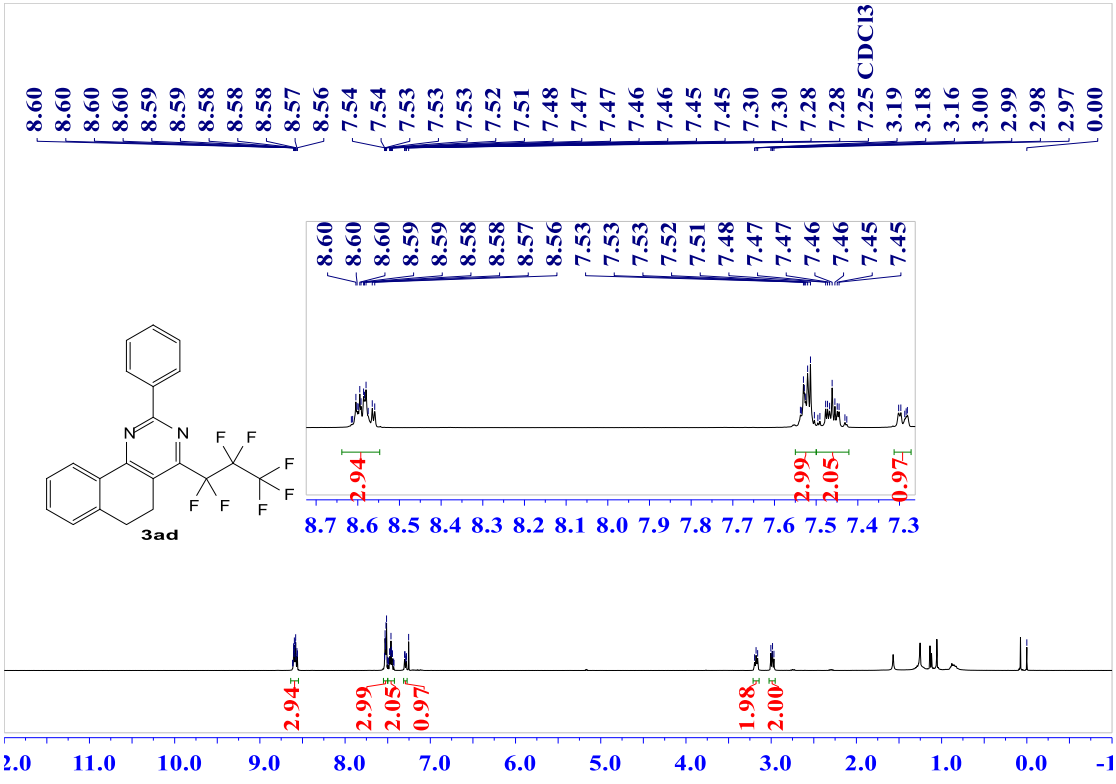

-79.76
-79.78
-79.81
-110.26
-110.29
-110.31
-110.34
-125.55
-125.57
-125.58
-125.60
-125.62
Cl
N
N
F
F
F
F
F
F
F
3ab
2.85
2.00
1.98
-80.1
-110.4
-125.4
-126.0
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210

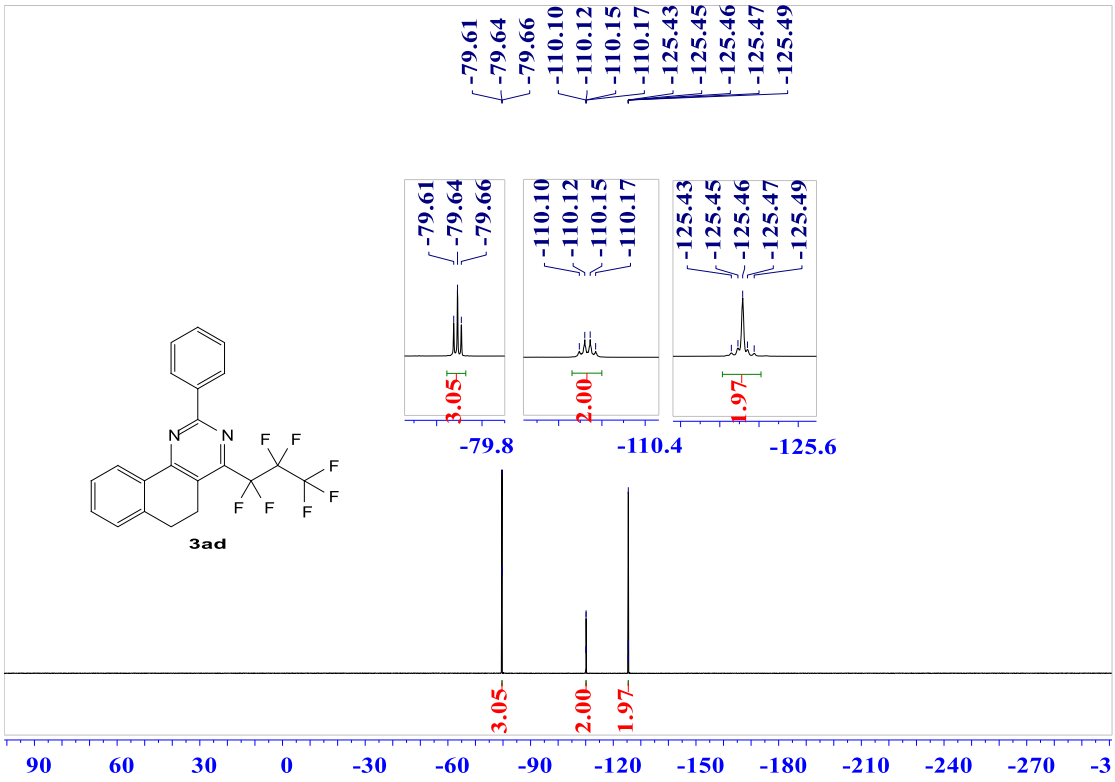

162.57
161.09
161.08
161.07
152.06
151.82
139.42
137.27
135.23
131.98
131.92
129.58
128.81
127.88
127.50
126.40
126.18
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
26.84
26.83
26.82
22.98
22.96
22.95
22.92
22.90
22.88
22.87
22.85
22.83
22.81
22.79
22.78
22.76
22.74
22.72
Cl
N
N
F
F
F
F
F
F
F
3ab
152.0
138
136
134
132
130
128
26
25
24
23
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

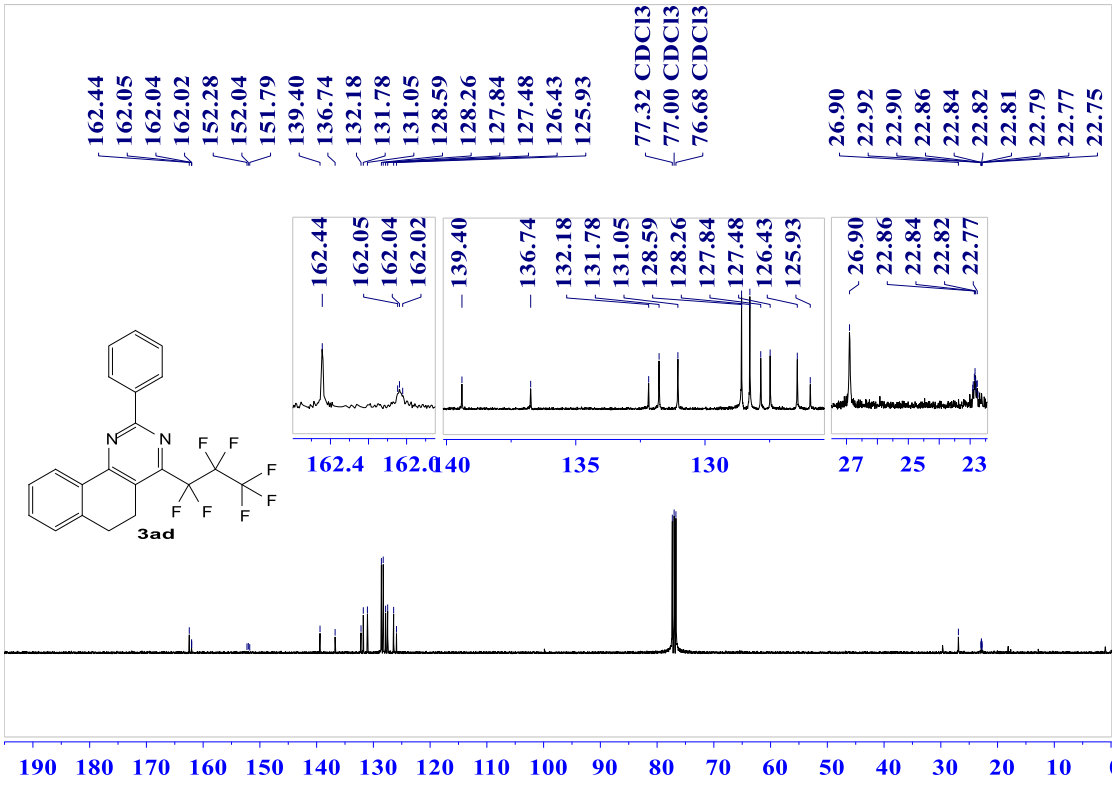

8.61 8.60 8.60 8.59 8.59 8.58 8.58 8.56 8.55 8.54 8.53 7.49 7.49 7.47 7.47 7.47 7.46 7.45 7.31 7.31 7.31 7.31 7.29 7.29 7.26 CDCl3 7.22 7.20 7.20 7.19 7.18 7.17 3.18 3.16 3.01 3.00 2.99 2.97 0.00
3ac
1.93 0.99 2.00 0.96 2.09 2.00 2.06
11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5

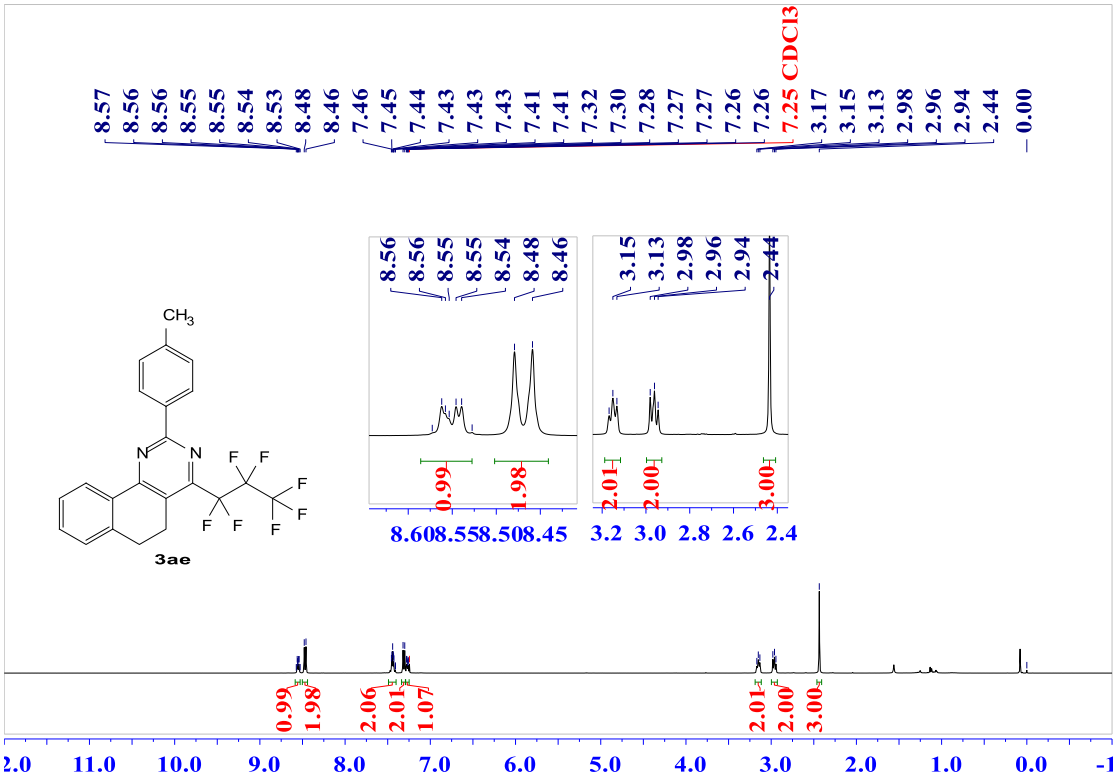

-79.63 -79.65 -79.68 -109.52 -109.53 -109.54 -109.55 -109.55 -109.56 -109.57 -109.58 -109.59 -110.14 -110.15 -110.16 -110.18 -110.19 -110.20 -110.21 -125.45 -125.46
3ac
3.05 0.99 2.00 1.99
-79.5 -79.6 -79.7 -109.5 -110.5 -125.4 -125.6
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270 -3

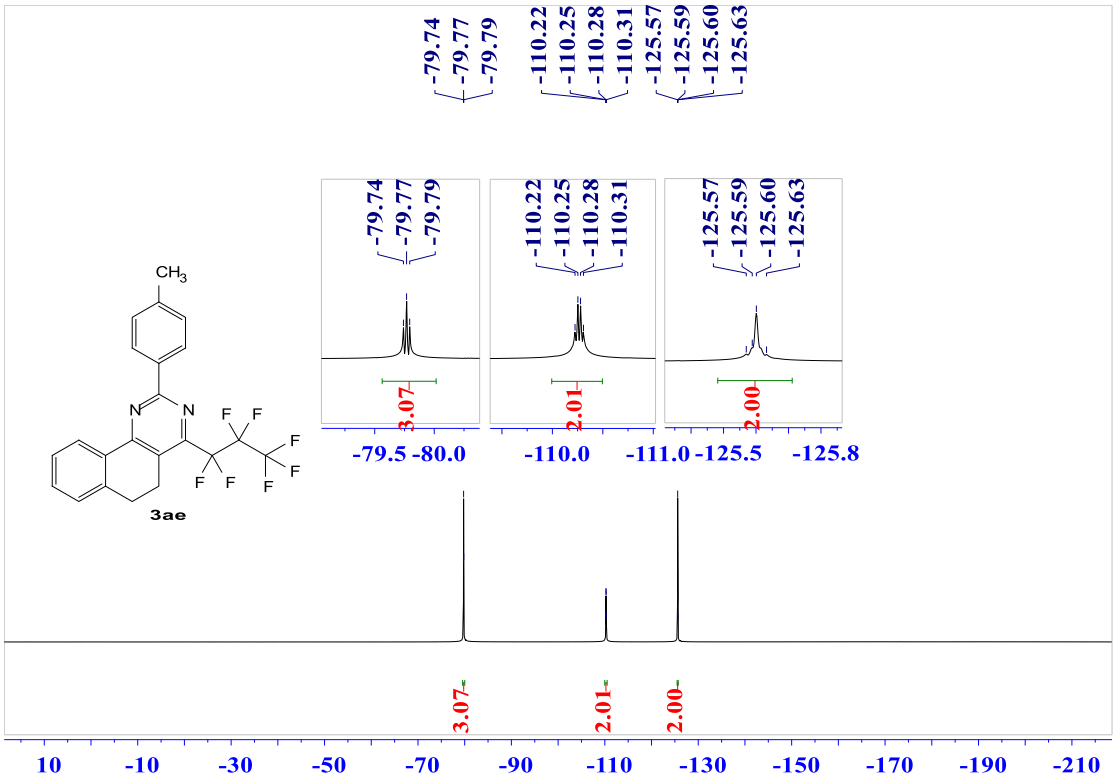

166.13
163.64
162.53
161.15
161.14
152.28
152.05
151.79
139.44
132.95
132.92
132.06
131.87
130.46
130.38
127.89
127.50
126.38
125.85
125.84
115.68
115.46
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
26.88
22.89
22.87
22.85
22.83
22.81
22.80
22.76
22.76
22.75
3ac

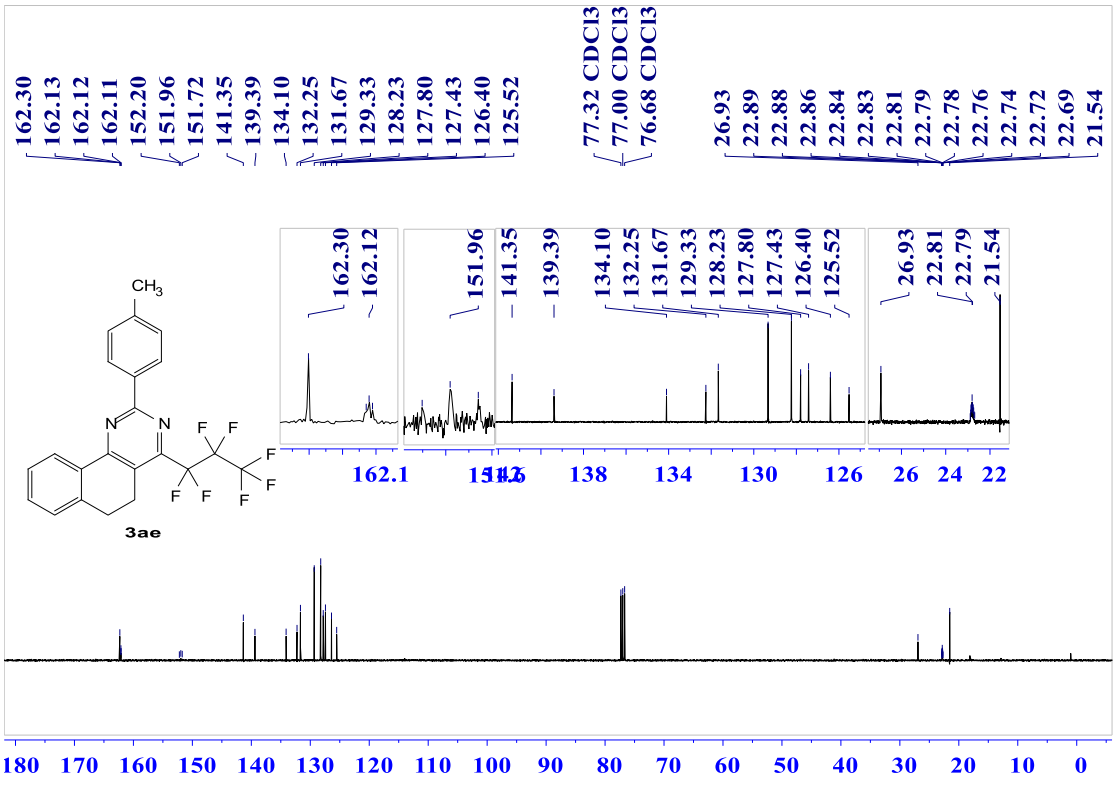

8.60
8.60
8.60
8.60
8.59
8.59
8.58
8.58
8.58
8.57
8.56
7.54
7.54
7.53
7.53
7.53
7.52
7.51
7.48
7.47
7.47
7.46
7.46
7.45
7.45
7.30
7.30
7.28
7.28
7.25 CDCl3
3.19
3.18
3.16
3.00
2.99
2.98
2.97
0.00
2.94
2.99
2.05
0.97
1.98
2.00
3ad

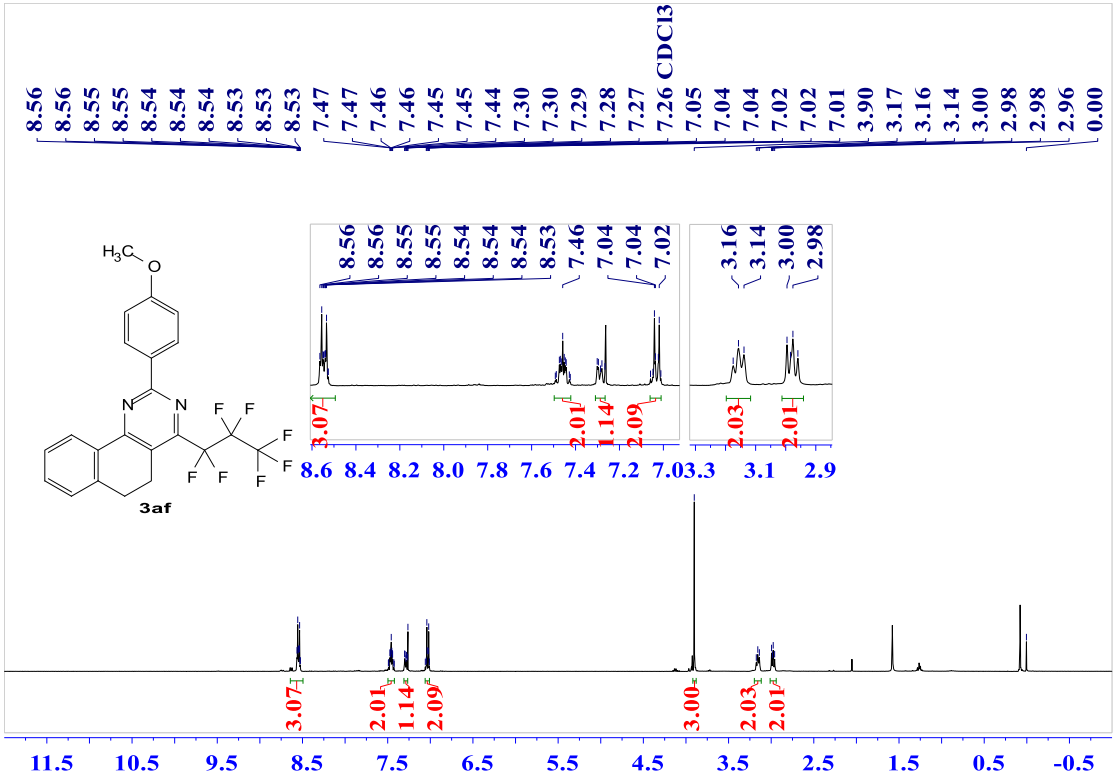

-79.61
-79.64
-79.66
-110.10
-110.12
-110.15
-110.17
-125.43
-125.45
-125.46
-125.47
-125.49
3.05
2.00
1.97
-79.8
-110.4
-125.6
3ad
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270 -3

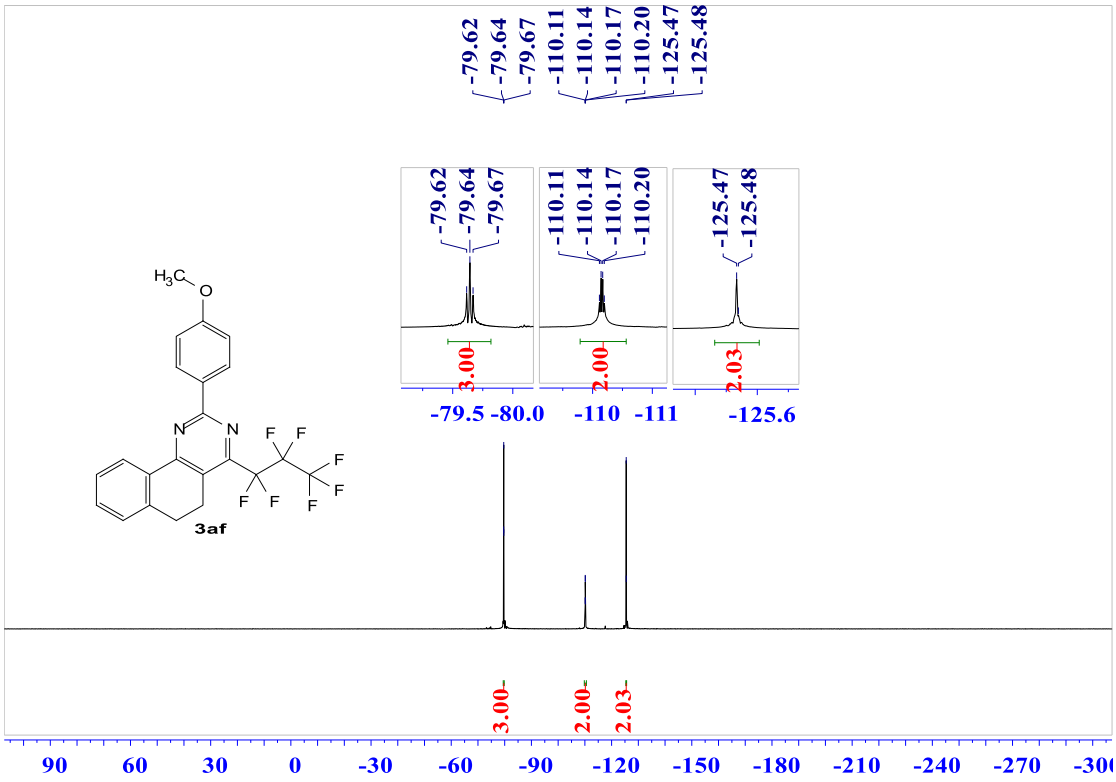

162.44
162.05
162.04
162.02
152.28
152.04
151.79
139.40
136.74
132.18
131.78
131.05
128.59
128.26
127.84
127.48
126.43
125.93
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
26.90
22.92
22.90
22.86
22.84
22.82
22.81
22.79
22.77
22.75
3ad
162.4 162.0 140 135 130 27 25 23
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

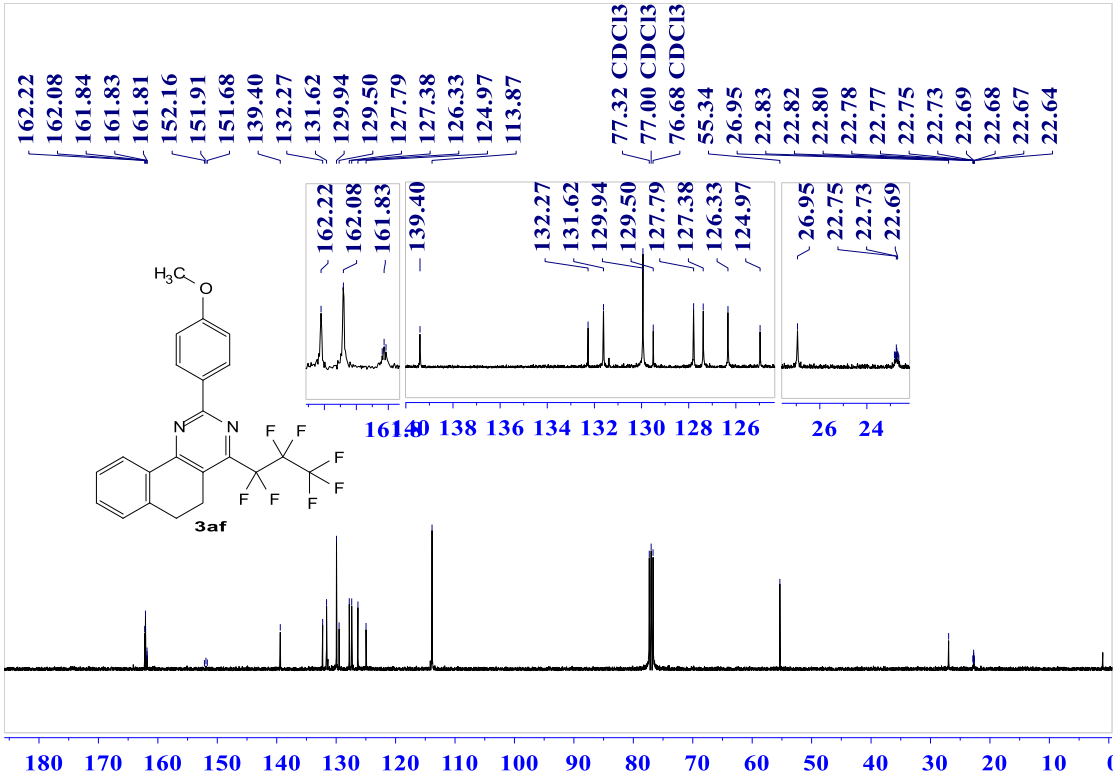

8.57 8.56 8.56 8.55 8.55 8.54 8.53 8.48 8.46 7.46 7.45 7.44 7.43 7.43 7.43 7.41 7.41 7.32 7.30 7.28 7.27 7.27 7.26 7.26 7.25 CDCl3 3.17 3.15 3.13 2.98 2.96 2.94 2.44 0.00
CH3
N N F F F F F F F F
3ae
0.99 1.98 2.01 2.00 3.00
8.608.558.508.45 3.2 3.0 2.8 2.6 2.4
0.99 1.98 2.06 2.01 1.07 2.01 2.00 3.00
2.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

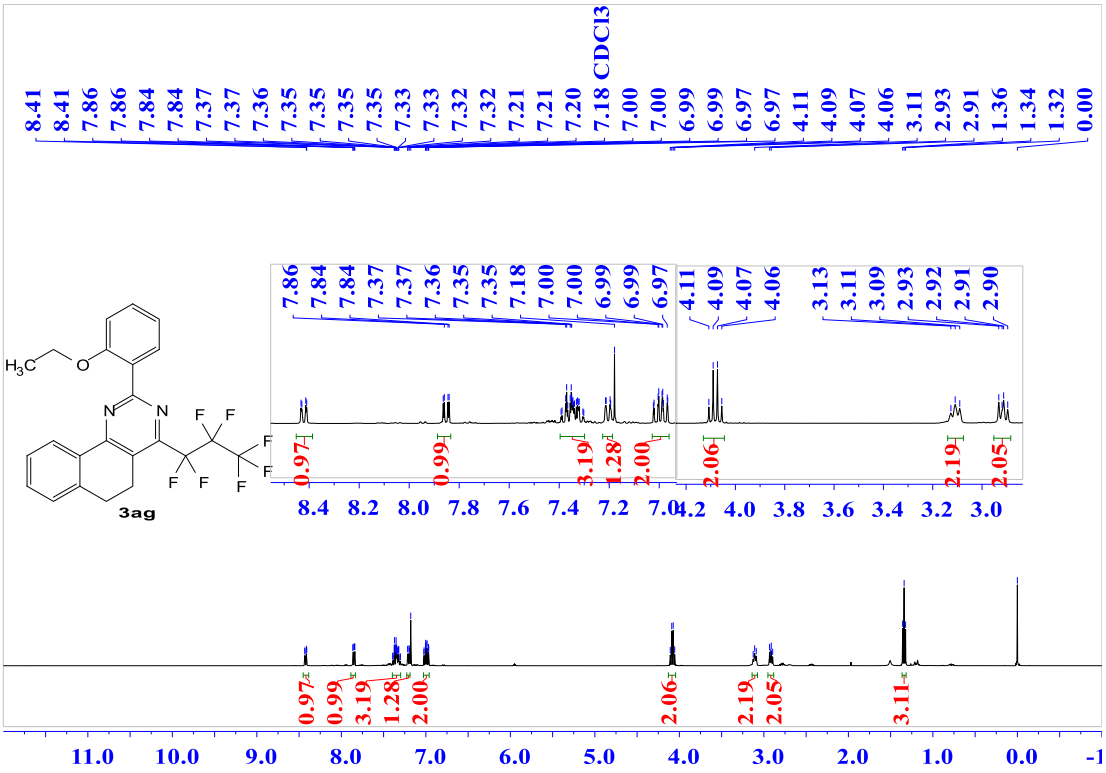

-79.74 -79.77 -79.79 -110.22 -110.25 -110.28 -110.31 -125.57 -125.59 -125.60 -125.63
CH3
N N F F F F F F F F
3ae
3.07 2.01 2.00
-79.5 -80.0 -110.0 -111.0 -125.5 -125.8
3.07 2.01 2.00
10 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210

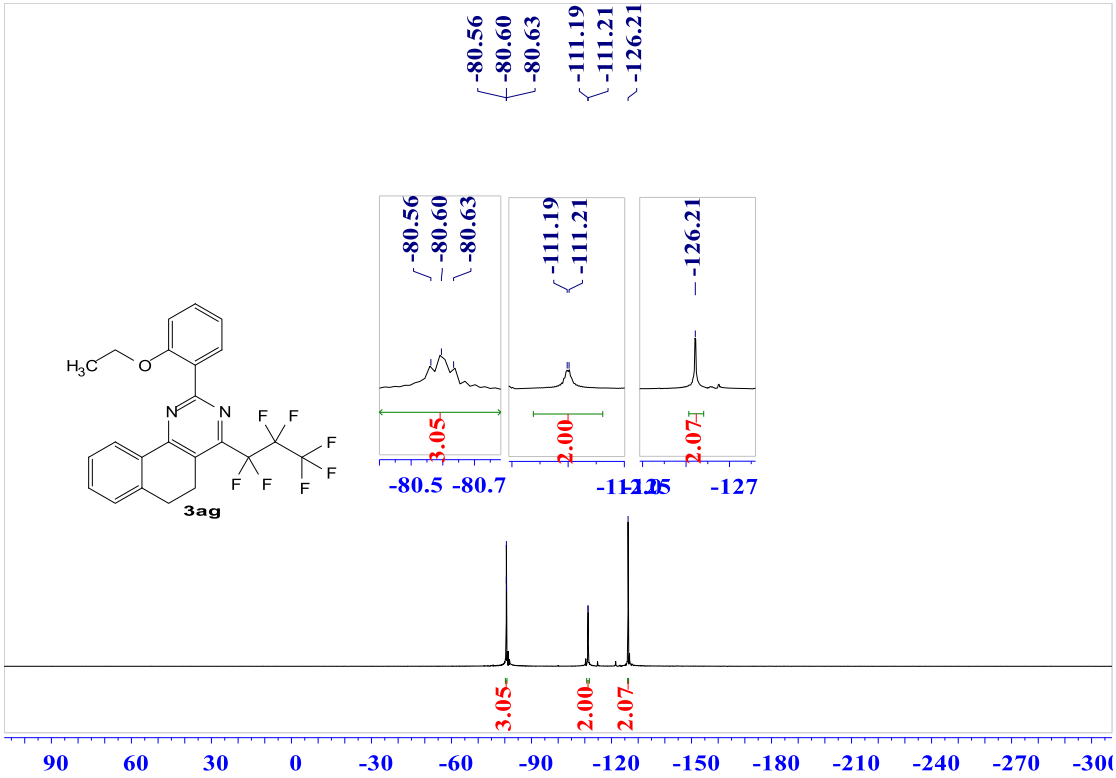

162.30
162.13
162.12
162.11
152.20
151.96
151.72
141.35
139.39
134.10
132.25
131.67
129.33
128.23
127.80
127.43
126.40
125.52
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
26.93
22.89
22.88
22.86
22.84
22.83
22.81
22.79
22.78
22.76
22.74
22.72
22.69
21.54
CH3
N
N
F
F
F
F
F
F
F
3ae
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

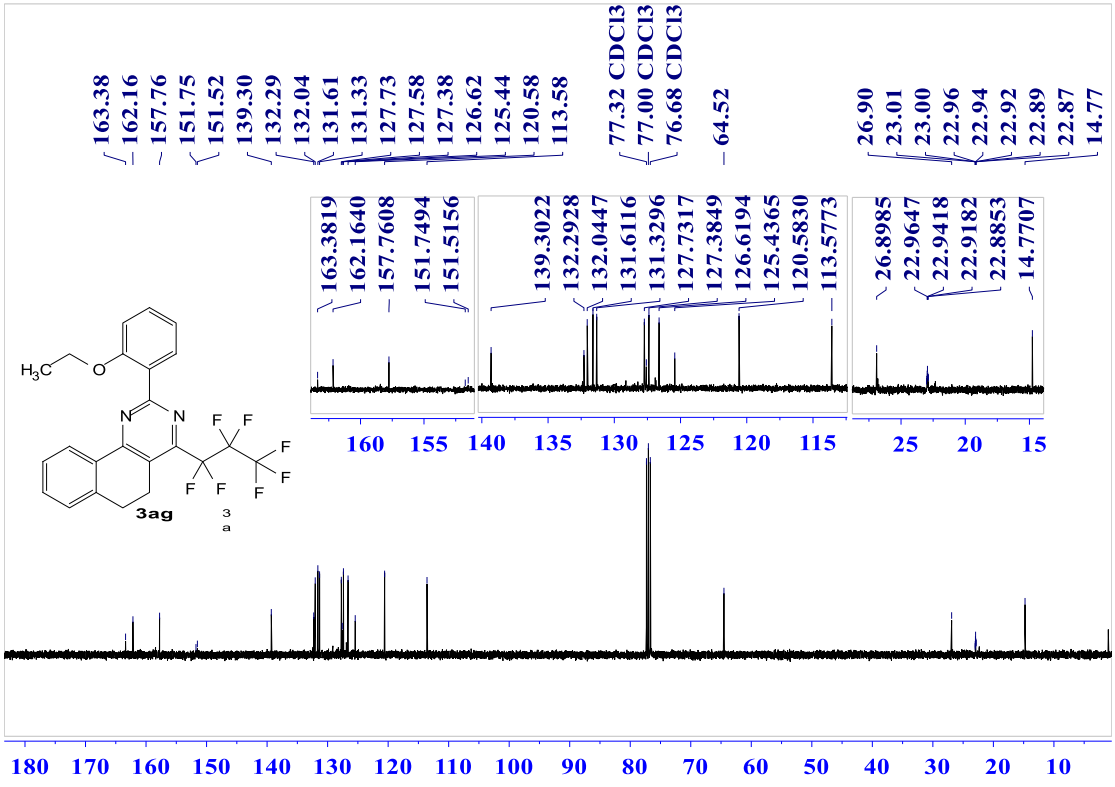

8.56
8.56
8.55
8.55
8.54
8.54
8.54
8.53
8.53
8.53
7.47
7.47
7.46
7.46
7.45
7.45
7.44
7.30
7.30
7.29
7.28
7.27
7.26 CDCl3
7.05
7.04
7.04
7.02
7.02
7.01
3.90
3.17
3.16
3.14
3.00
2.98
2.98
2.96
0.00
H3C
O
N
N
F
F
F
F
F
F
F
3af
3.07
2.01
1.14
2.09
3.00
2.03
2.01
11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5

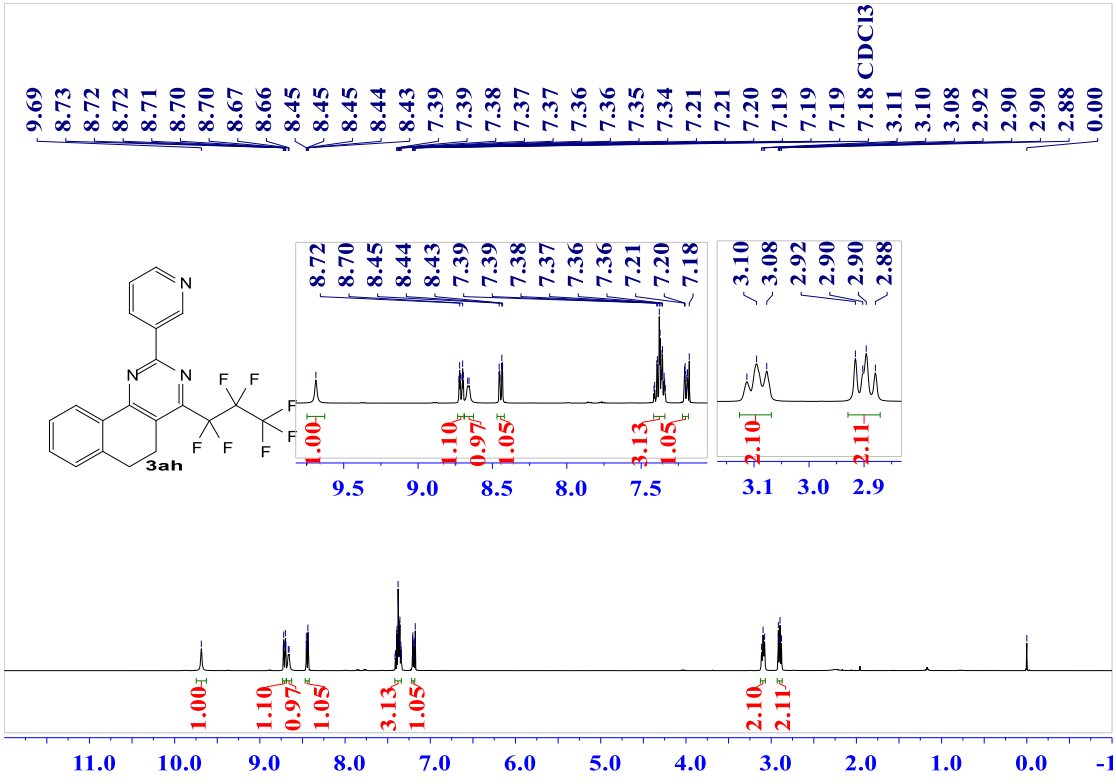

-79.62
-79.64
-79.67
-110.11
-110.14
-110.17
-110.20
-125.47
-125.48
3.00
2.00
2.03
-79.5 -80.0
-110
-111
-125.6
3af
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270

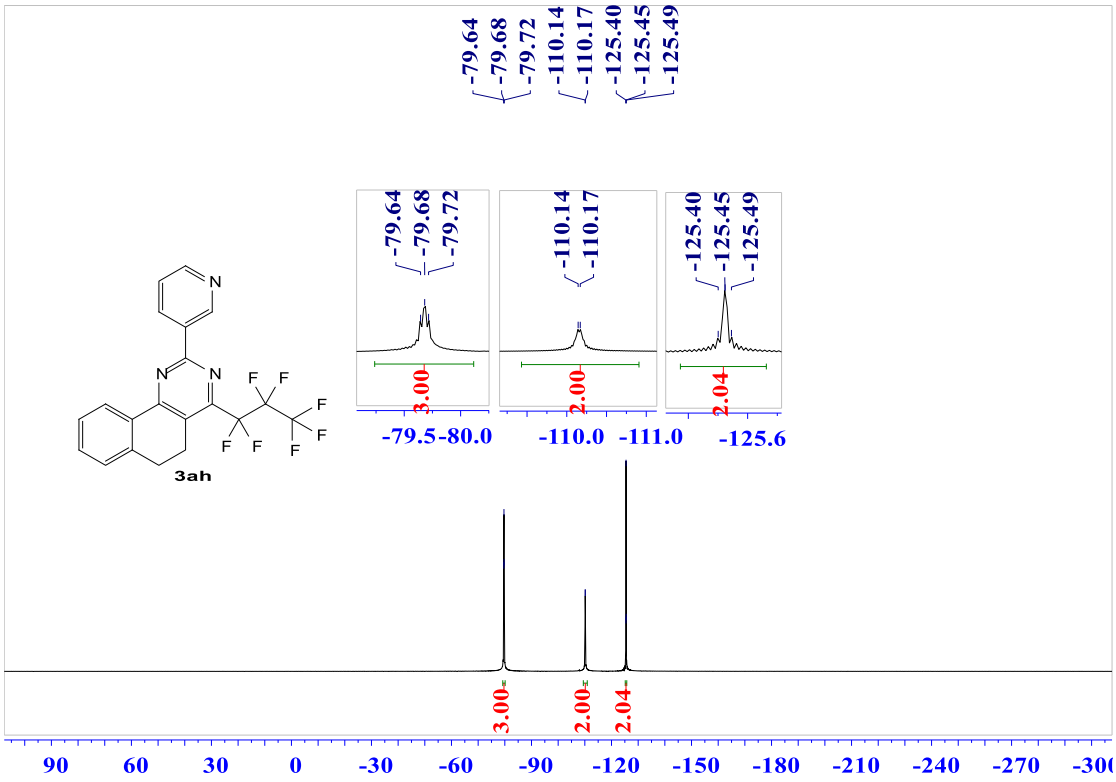

162.22
162.08
161.84
161.83
161.81
152.16
151.91
151.68
139.40
132.27
131.62
129.94
129.50
127.79
127.38
126.33
124.97
113.87
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
55.34
26.95
22.83
22.82
22.80
22.78
22.77
22.75
22.73
22.69
22.68
22.67
22.64
3af
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

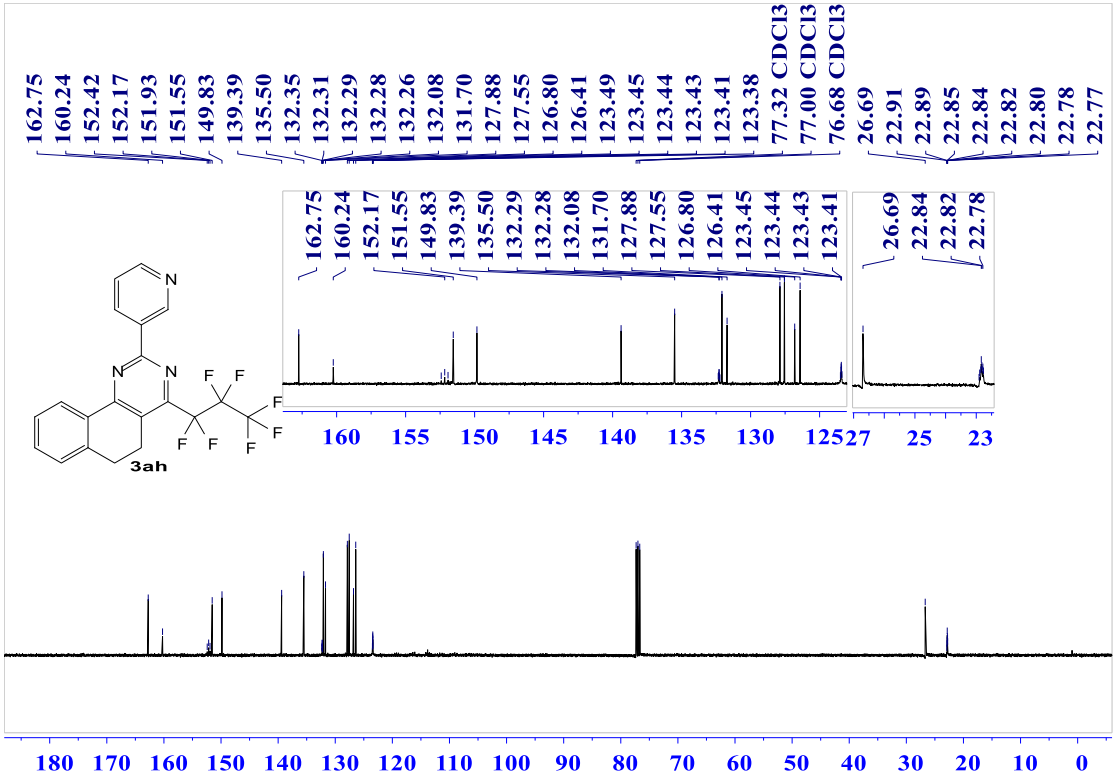

8.41 8.41 7.86 7.86 7.84 7.84 7.37 7.37 7.36 7.35 7.35 7.35 7.35 7.33 7.33 7.32 7.32 7.21 7.21 7.20 7.18 CDCl3 7.00 7.00 6.99 6.99 6.97 6.97 4.11 4.09 4.07 4.06 3.11 2.93 2.91 1.36 1.34 1.32 0.00
3ag
0.97 0.99 3.19 1.28 2.00 2.06 2.19 2.05 3.11
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

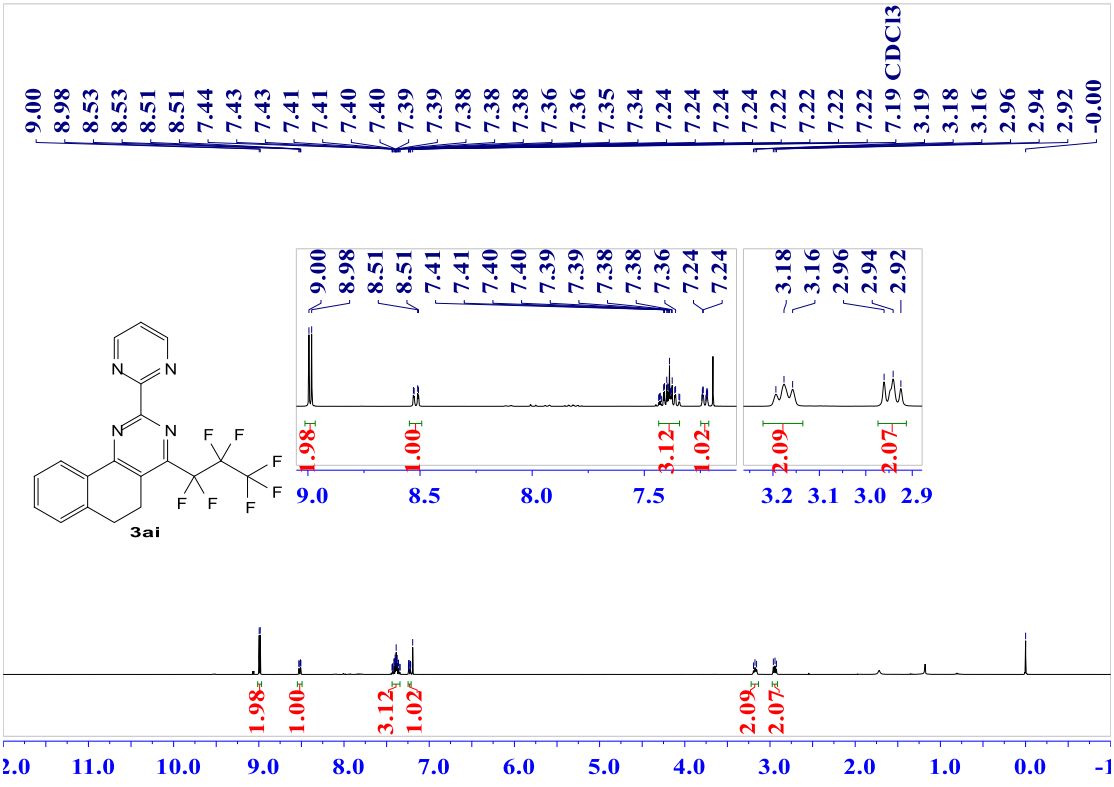

-80.56 -80.60 -80.63 -111.19 -111.21 -126.21
3ag
3.05 2.00 2.07
-80.5 -80.7 -127
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270

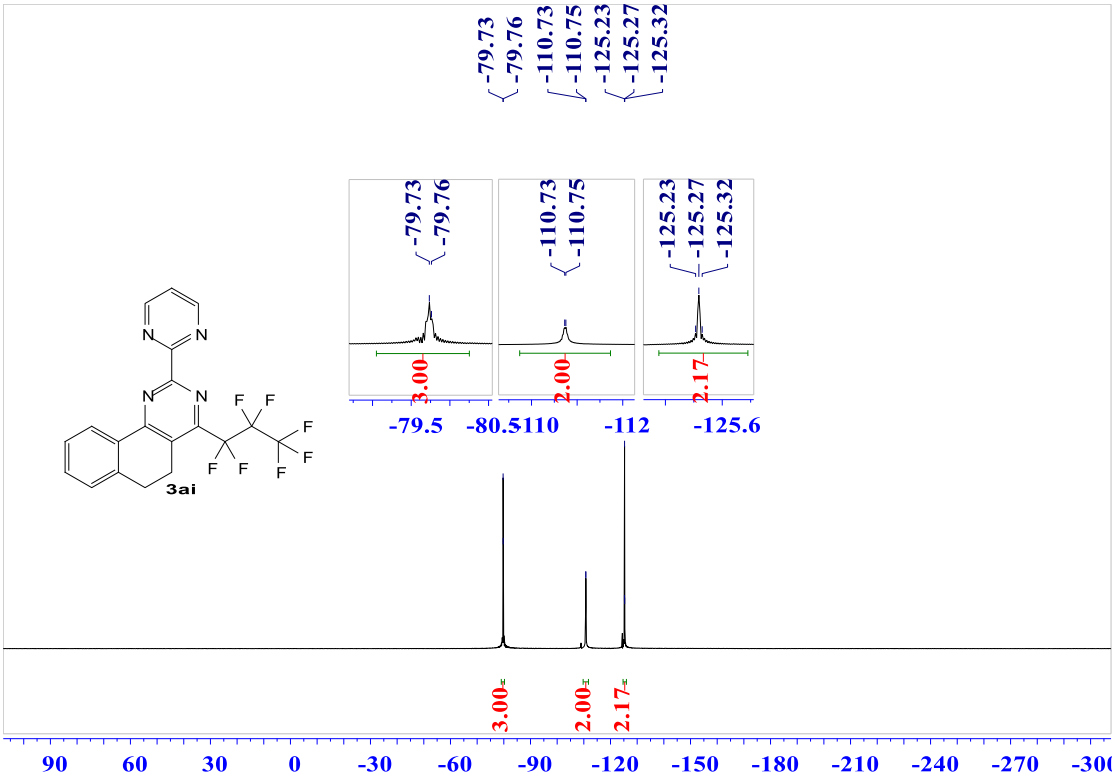

163.38
162.16
157.76
151.75
151.52
139.30
132.29
132.04
131.61
131.33
127.73
127.58
127.38
126.62
125.44
120.58
113.58
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.52
26.90
23.01
23.00
22.96
22.94
22.92
22.89
22.87
14.77
3ag
3a

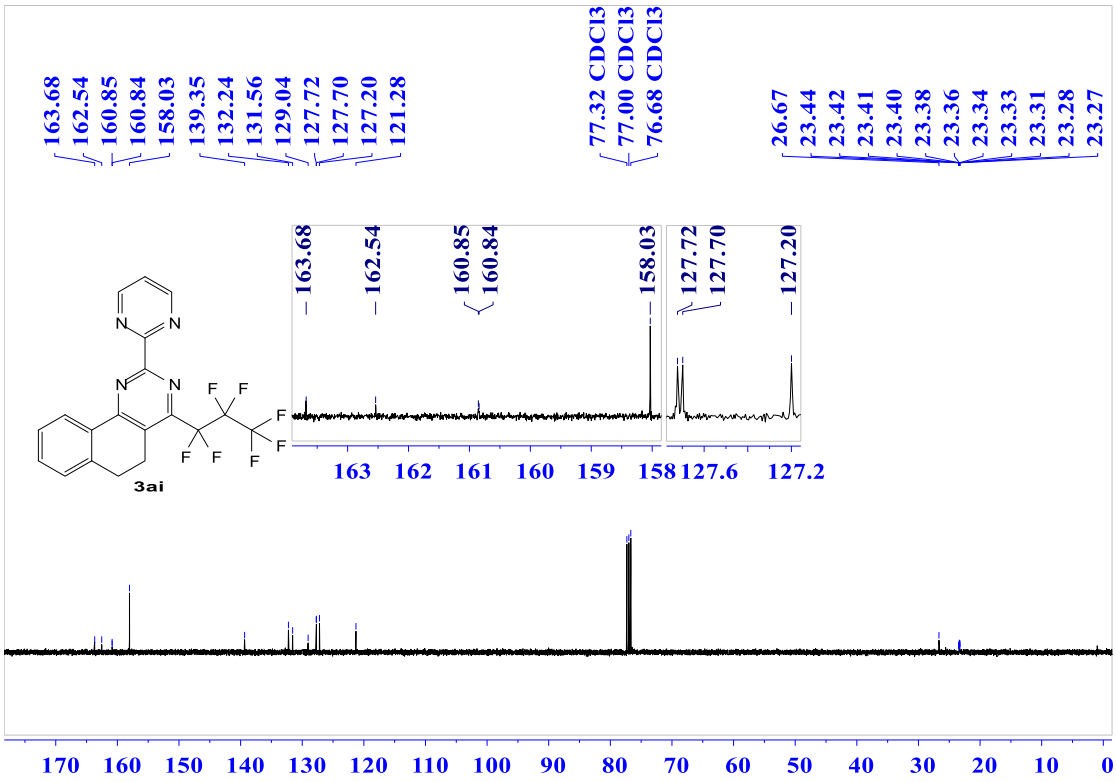

9.69
8.73
8.72
8.72
8.71
8.70
8.70
8.67
8.66
8.45
8.45
8.45
8.44
8.43
7.39
7.39
7.38
7.37
7.37
7.36
7.36
7.35
7.34
7.21
7.21
7.20
7.19
7.19
7.19
7.18 CDCl3
3.11
3.10
3.08
2.92
2.90
2.90
2.88
0.00
3ah

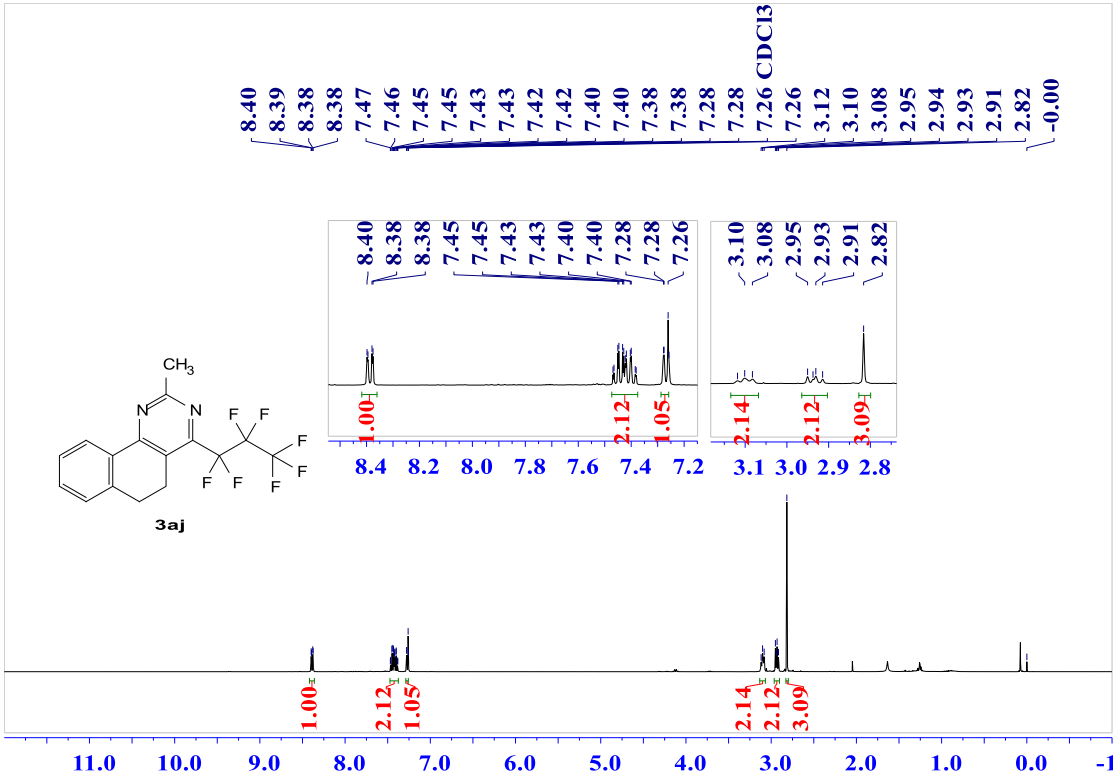

-79.64
-79.68
-79.72
-110.14
-110.17
-125.40
-125.45
-125.49
3ah
3.00
2.00
2.04
-79.5 -80.0
-110.0 -111.0
-125.6
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270 -300

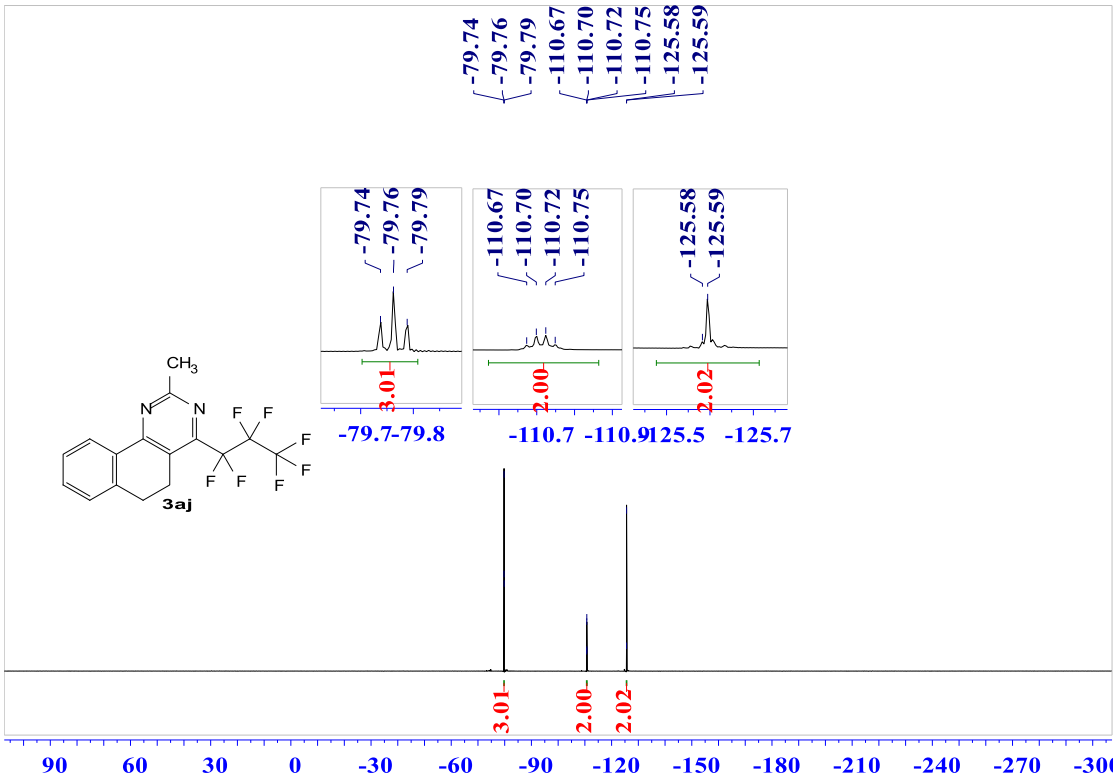

162.75
160.24
152.42
152.17
151.93
151.55
149.83
139.39
135.50
132.35
132.31
132.29
132.28
132.26
132.08
131.70
127.88
127.55
126.80
126.41
123.49
123.45
123.44
123.43
123.41
123.38
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
26.69
22.91
22.89
22.85
22.84
22.82
22.80
22.78
22.77
3ah
160 155 150 145 140 135 130 125
27 25 23
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

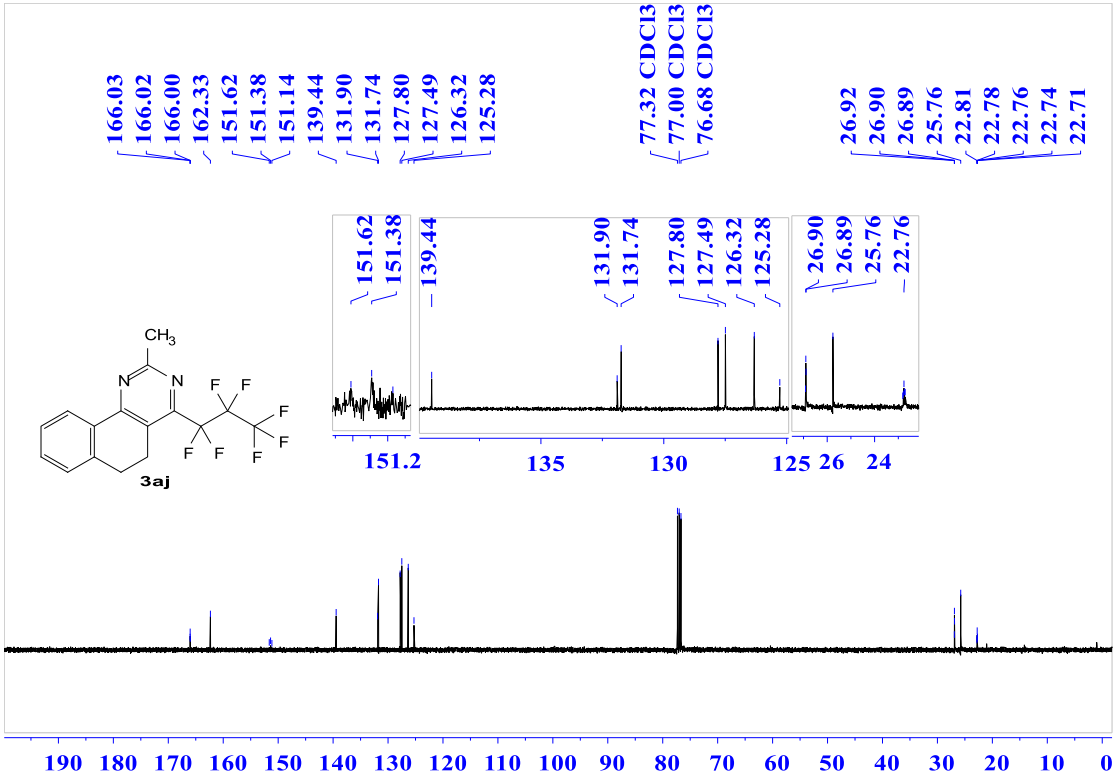

9.00 8.98 8.53 8.53 8.51 8.51 7.44 7.43 7.43 7.41 7.41 7.40 7.40 7.39 7.39 7.38 7.38 7.38 7.36 7.36 7.35 7.34 7.24 7.24 7.24 7.24 7.22 7.22 7.22 7.22 7.19 CDCl3 3.19 3.18 3.16 2.96 2.94 2.92 -0.00
3ai
1.98 1.00 3.12 1.02 2.09 2.07

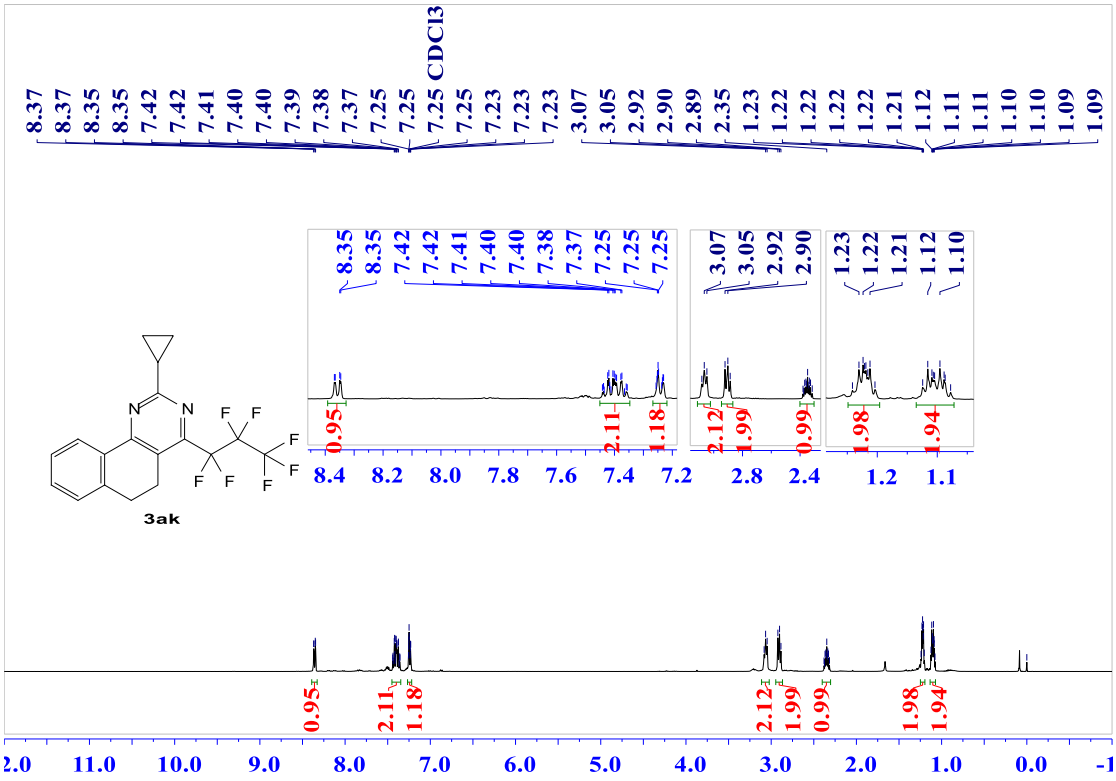

-79.73 -79.76 -110.73 -110.75 -125.23 -125.27 -125.32
3ai
3.00 2.00 2.17

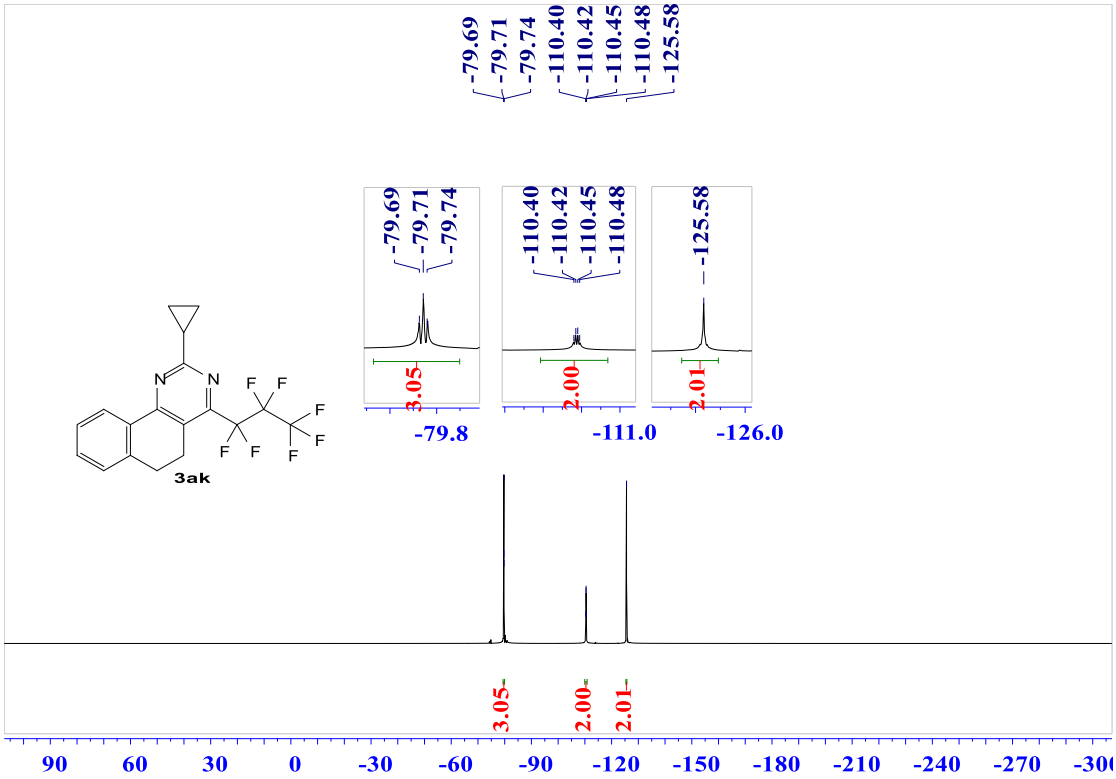

163.68
162.54
160.85
160.84
158.03
139.35
132.24
131.56
129.04
127.72
127.70
127.20
121.28
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
26.67
23.44
23.42
23.41
23.40
23.38
23.36
23.34
23.33
23.31
23.28
23.27
3ai
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

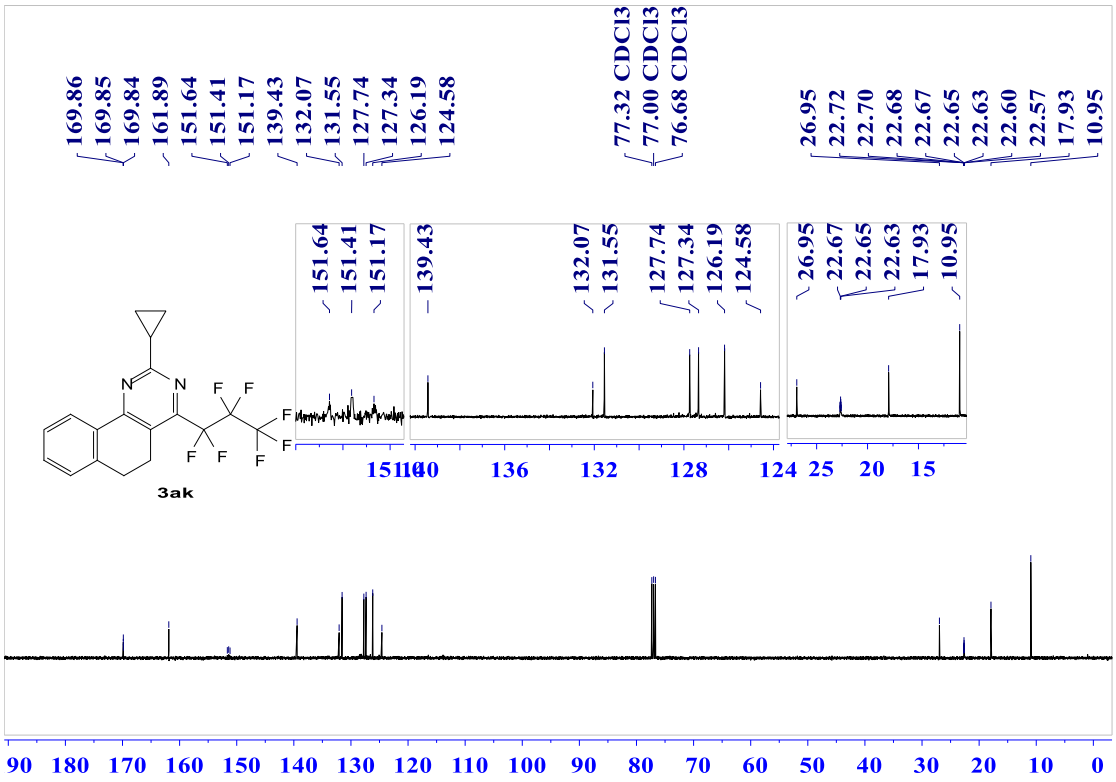

8.40
8.39
8.38
8.38
7.47
7.46
7.45
7.45
7.43
7.43
7.42
7.42
7.40
7.40
7.38
7.38
7.28
7.28
7.26 CDCl3
7.26
3.12
3.10
3.08
2.95
2.94
2.93
2.91
2.82
-0.00
1.00
2.12
1.05
2.14
2.12
3.09
3aj
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1

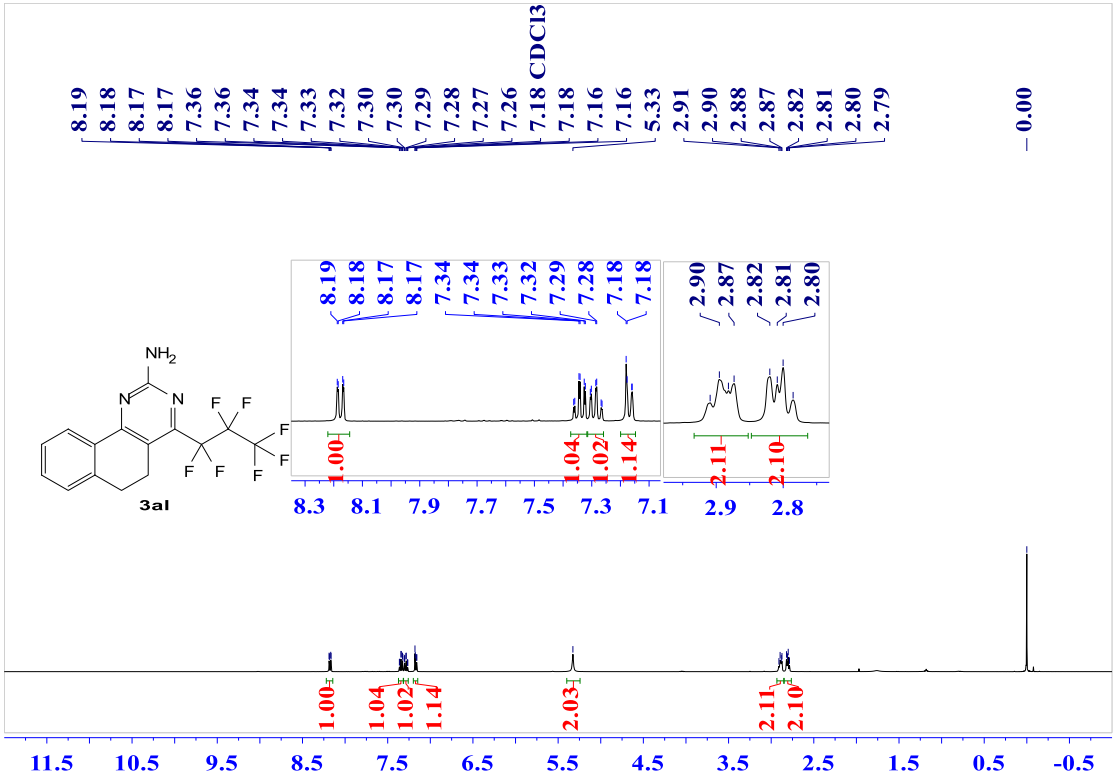

-79.74
-79.76
-79.79
-110.67
-110.70
-110.72
-110.75
-125.58
-125.59
3.01
2.00
2.02
-79.7-79.8
-110.7
-110.9
125.5
-125.7
CH3
N
N
F
F
F
F
F
F
F
3aj
90
60
30
0
-30
-60
-90
-120
-150
-180
-210
-240
-270

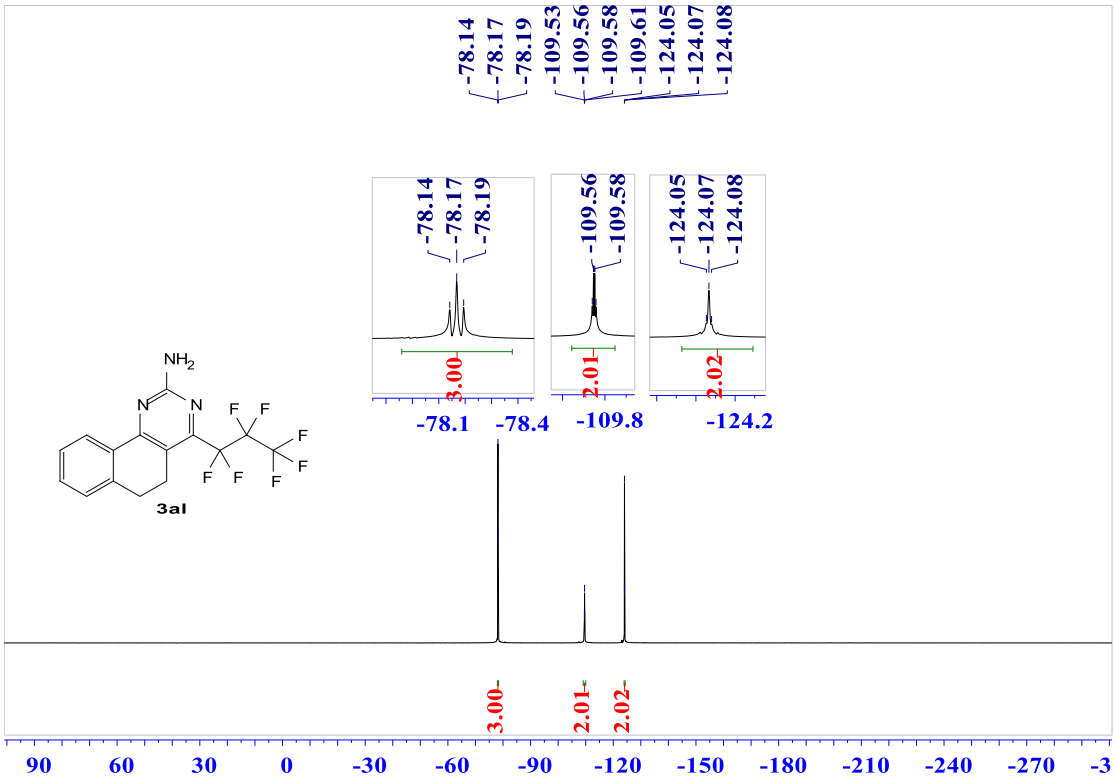

166.03
166.02
166.00
162.33
151.62
151.38
151.14
139.44
131.90
131.74
127.80
127.49
126.32
125.28
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
26.92
26.90
26.89
25.76
22.81
22.78
22.76
22.74
22.71
151.2
135
130
125
26
24
CH3
N
N
F
F
F
F
F
F
F
3aj
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

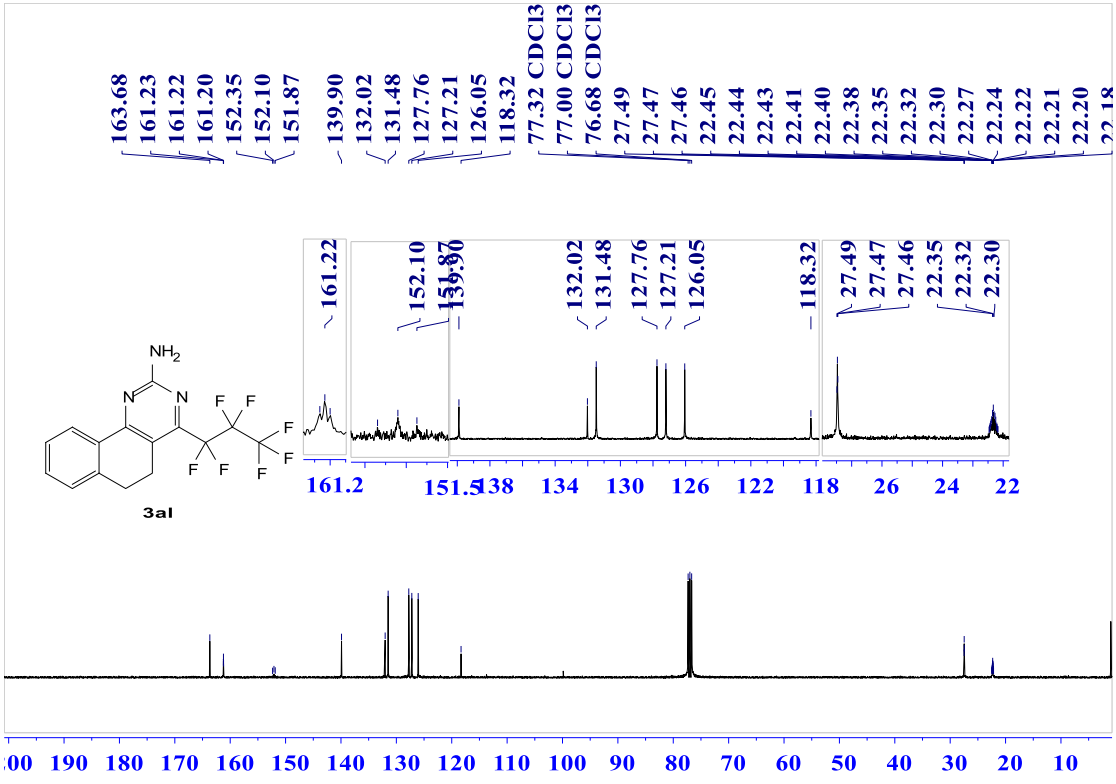

8.37 8.37 8.35 8.35 7.42 7.42 7.41 7.40 7.40 7.39 7.38 7.37 7.25 7.25 7.25 CDCl3 7.25 7.23 7.23 7.23 3.07 3.05 2.92 2.90 2.89 2.35 1.23 1.22 1.22 1.22 1.22 1.21 1.12 1.11 1.11 1.10 1.10 1.09 1.09
3ak
0.95 2.11 1.18 2.12 1.99 0.99 1.98 1.94
8.4 8.2 8.0 7.8 7.6 7.4 7.2 2.8 2.4 1.2 1.1
2.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

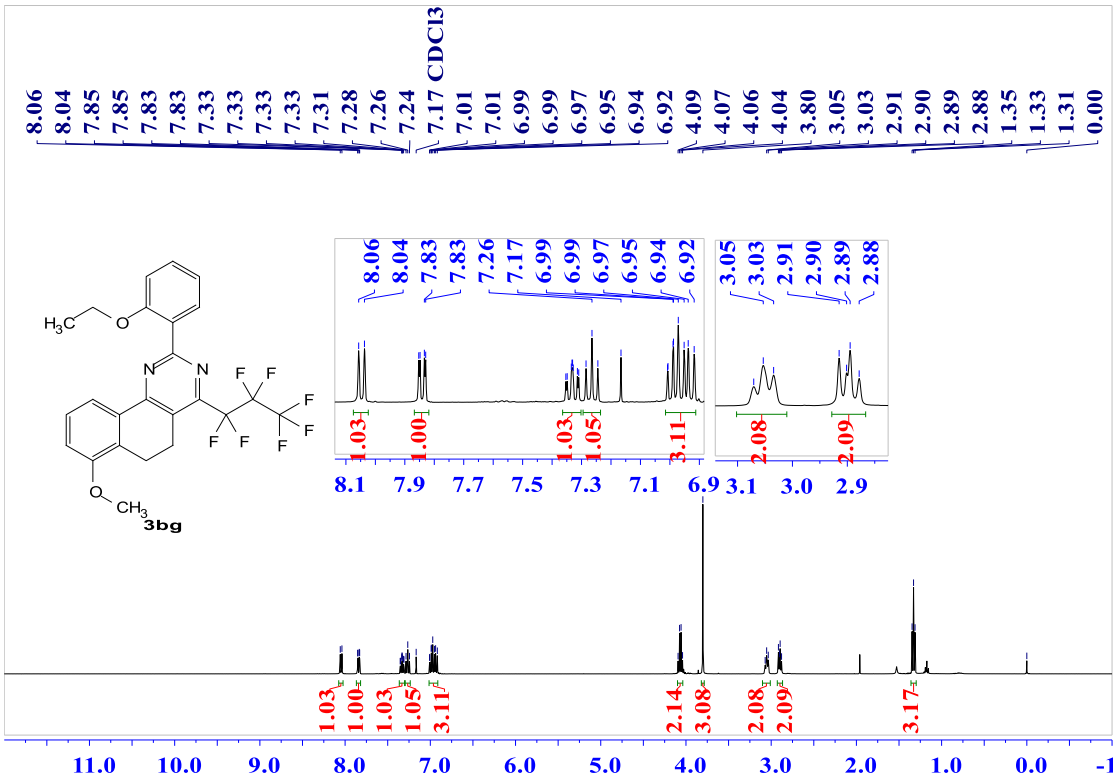

-79.69 -79.71 -79.74 -110.40 -110.42 -110.45 -110.48 -125.58
3ak
3.05 2.00 2.01
-79.8 -111.0 -126.0
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270

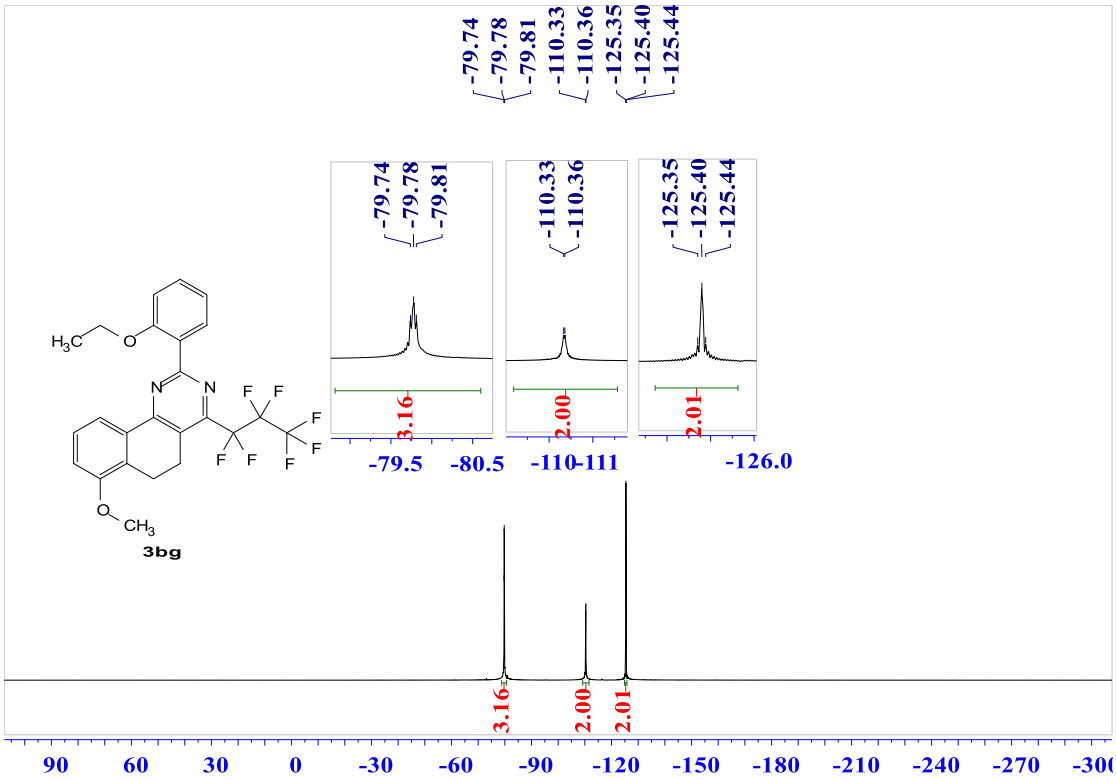

169.86
169.85
169.84
161.89
151.64
151.41
151.17
139.43
132.07
131.55
127.74
127.34
126.19
124.58
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
26.95
22.72
22.70
22.68
22.67
22.65
22.63
22.60
22.57
17.93
10.95
3ak

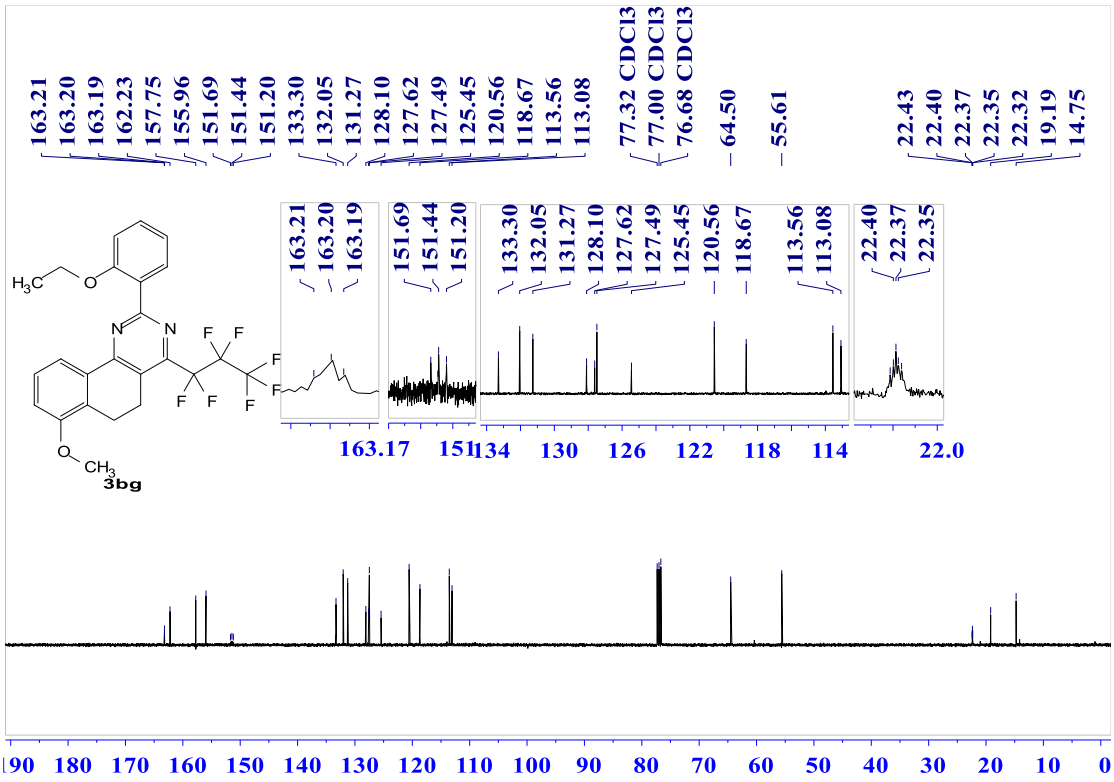

8.19
8.18
8.17
8.17
7.36
7.36
7.34
7.34
7.33
7.32
7.30
7.30
7.29
7.28
7.27
7.26
7.18 CDCl3
7.18
7.16
7.16
5.33
2.91
2.90
2.88
2.87
2.82
2.81
2.80
2.79
0.00
3al

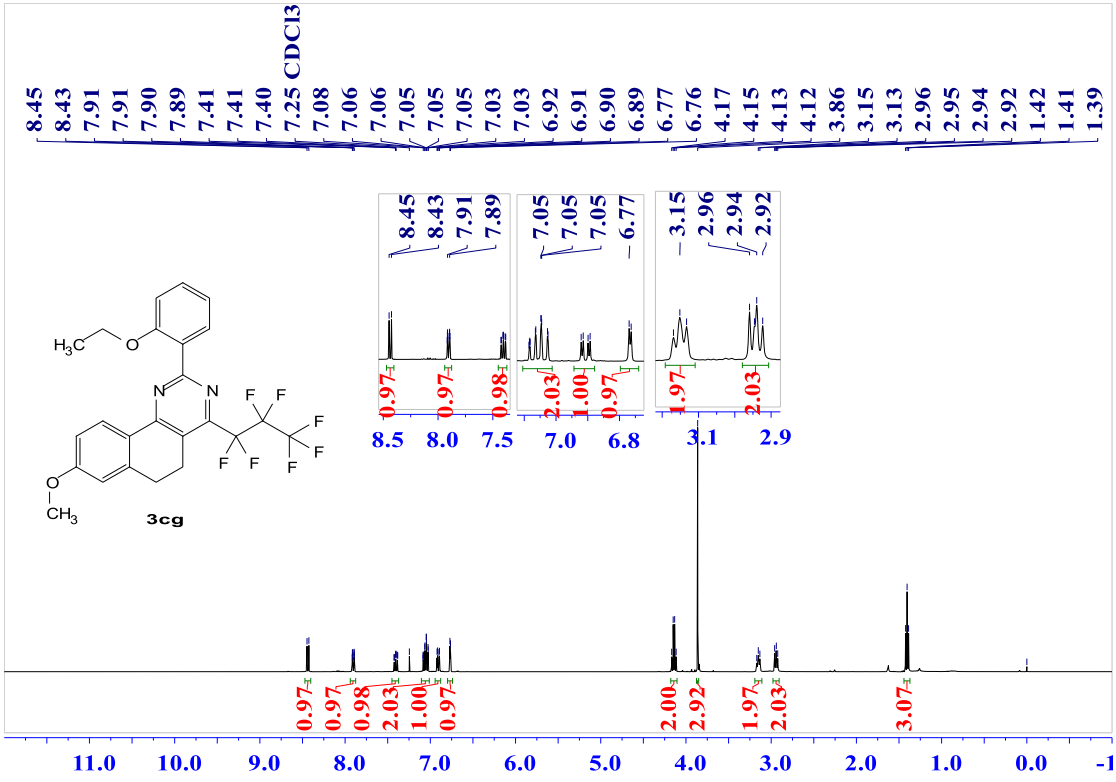

-78.14
-78.17
-78.19
-109.53
-109.56
-109.58
-109.61
-124.05
-124.07
-124.08
3.00
2.01
2.02
-78.1
-78.4
-109.8
-124.2
NH2
N
N
F
F
F
F
F
F
F
3al
90
60
30
0
-30
-60
-90
-120
-150
-180
-210
-240
-270

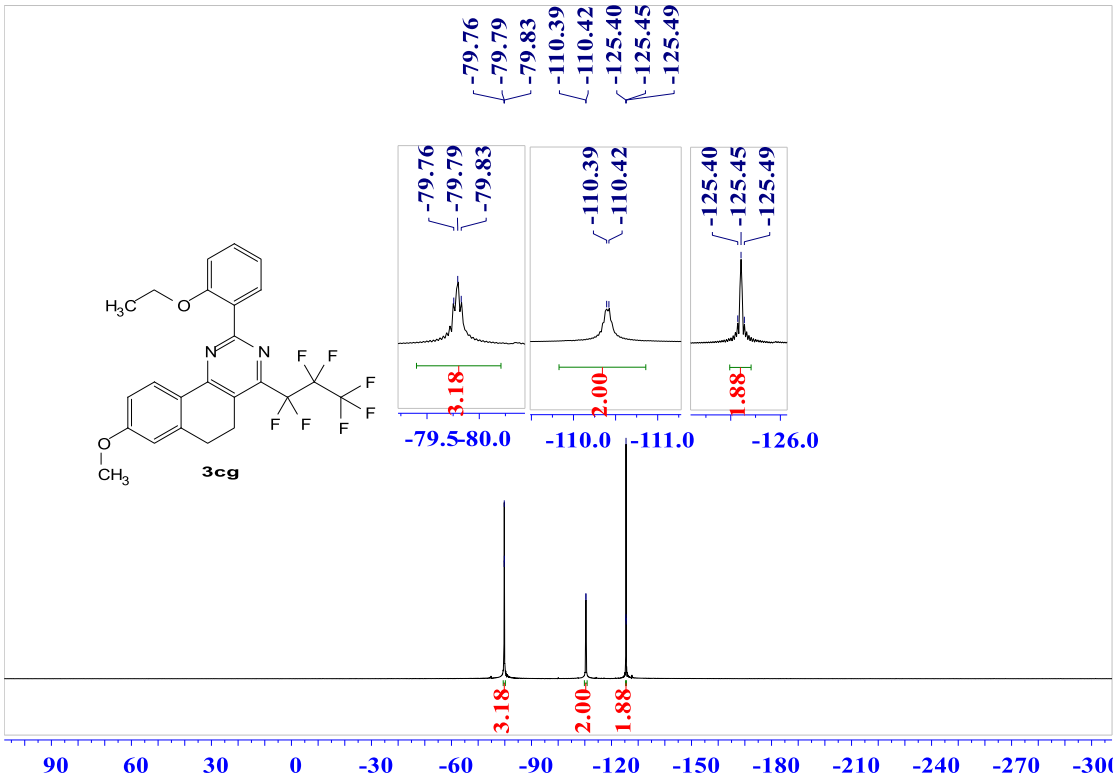

163.68
161.23
161.22
161.20
152.35
152.10
151.87
139.90
132.02
131.48
127.76
127.21
126.05
118.32
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
27.49
27.47
27.46
22.45
22.44
22.43
22.41
22.40
22.38
22.35
22.32
22.30
22.27
22.24
22.22
22.21
22.20
NH2
N
N
F
F
F
F
F
F
F
3al
161.2
151.5
138
134
130
126
122
118
26
24
22
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

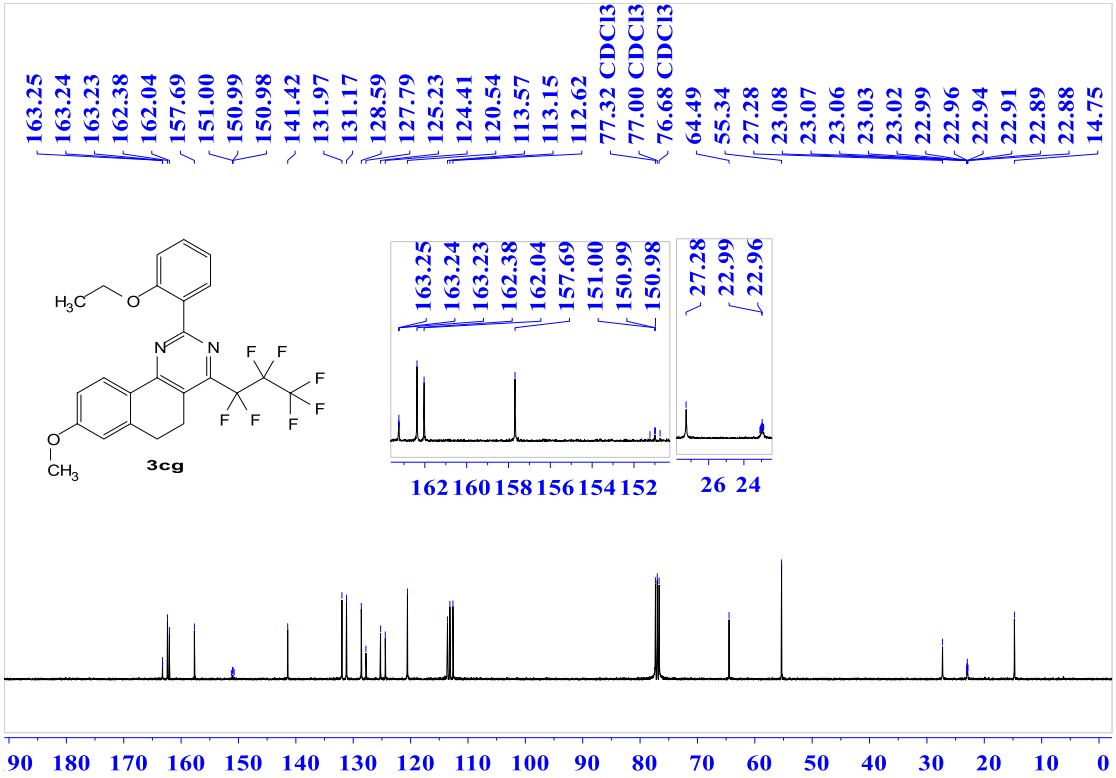

8.06 8.04 7.85 7.85 7.83 7.83 7.33 7.33 7.33 7.33 7.31 7.28 7.26 7.24 7.17 CDCl3 7.01 7.01 6.99 6.99 6.97 6.95 6.94 6.92 4.09 4.07 4.06 4.04 3.80 3.05 3.03 2.91 2.90 2.89 2.88 1.35 1.33 1.31 0.00
3bg
1.03 1.00 1.03 1.05 3.11 2.14 3.08 2.08 2.09 3.17
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

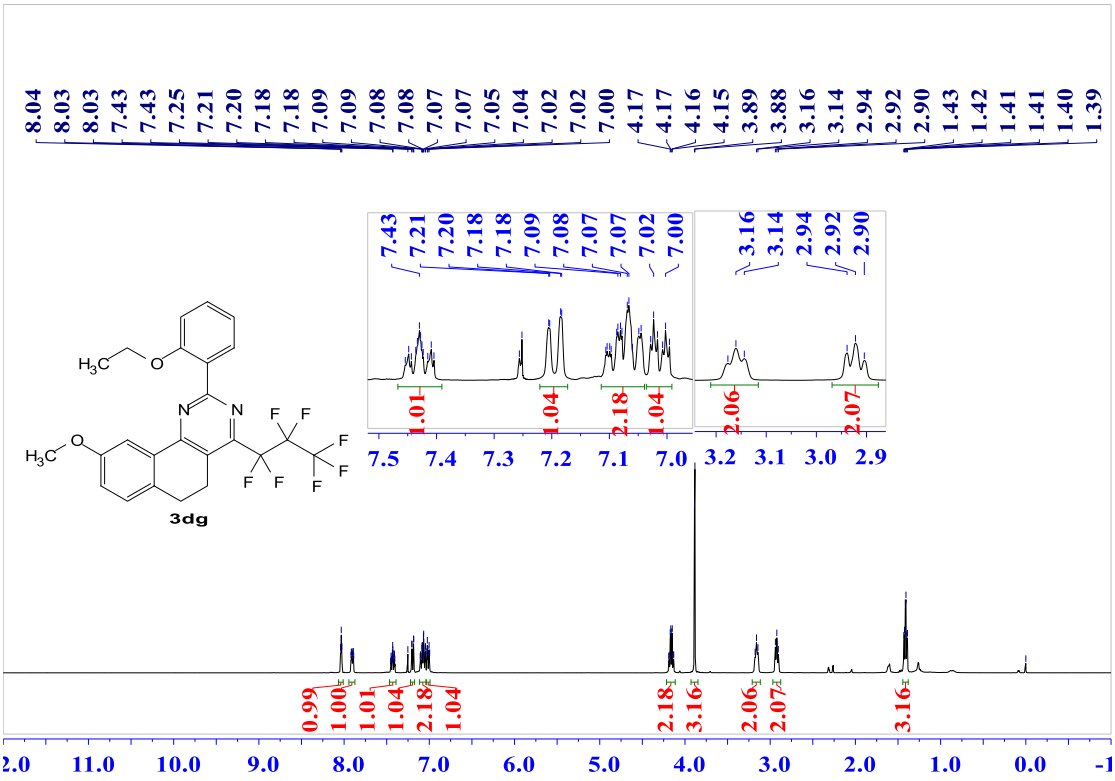

-79.74 -79.78 -79.81 -110.33 -110.36 -125.35 -125.40 -125.44
3bg
3.16 2.00 2.01
-79.5 -80.5 -110 -111 -126.0
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270

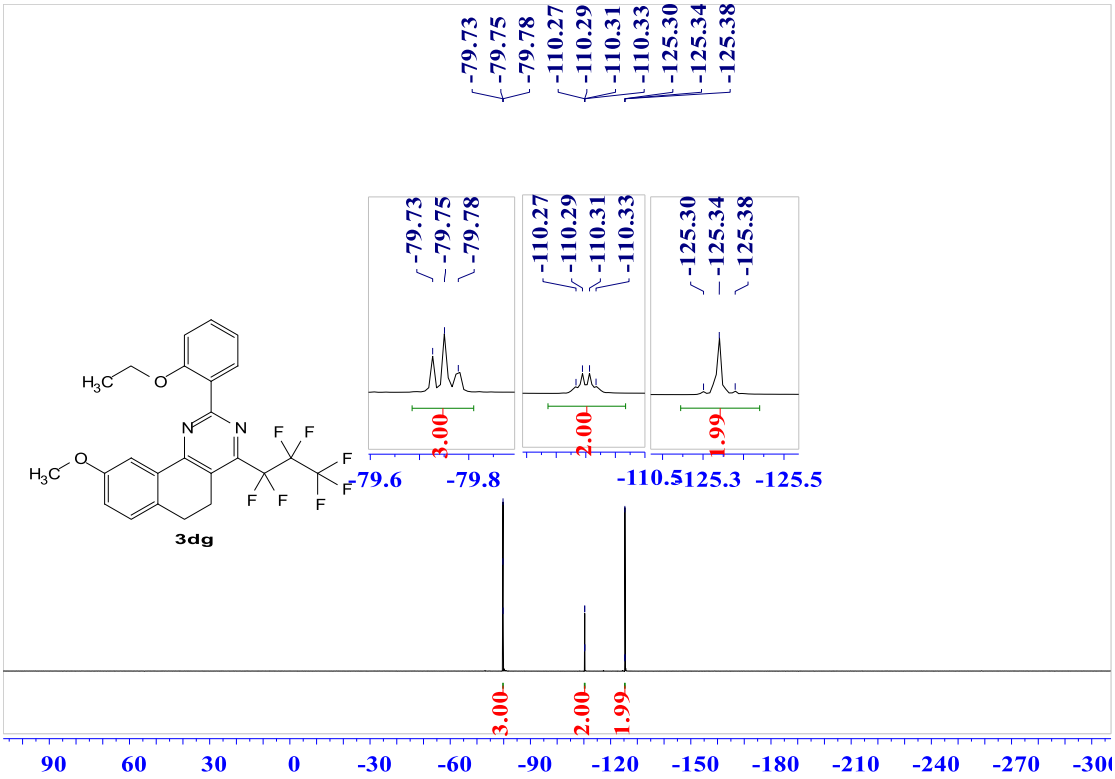

163.21
163.20
163.19
162.23
157.75
155.96
151.69
151.44
151.20
133.30
132.05
131.27
128.10
127.62
127.49
125.45
120.56
118.67
113.56
113.08
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.50
55.61
22.43
22.40
22.37
22.35
22.32
19.19
14.75
3bg
163.17
151
134
130
126
122
118
114
22.0
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

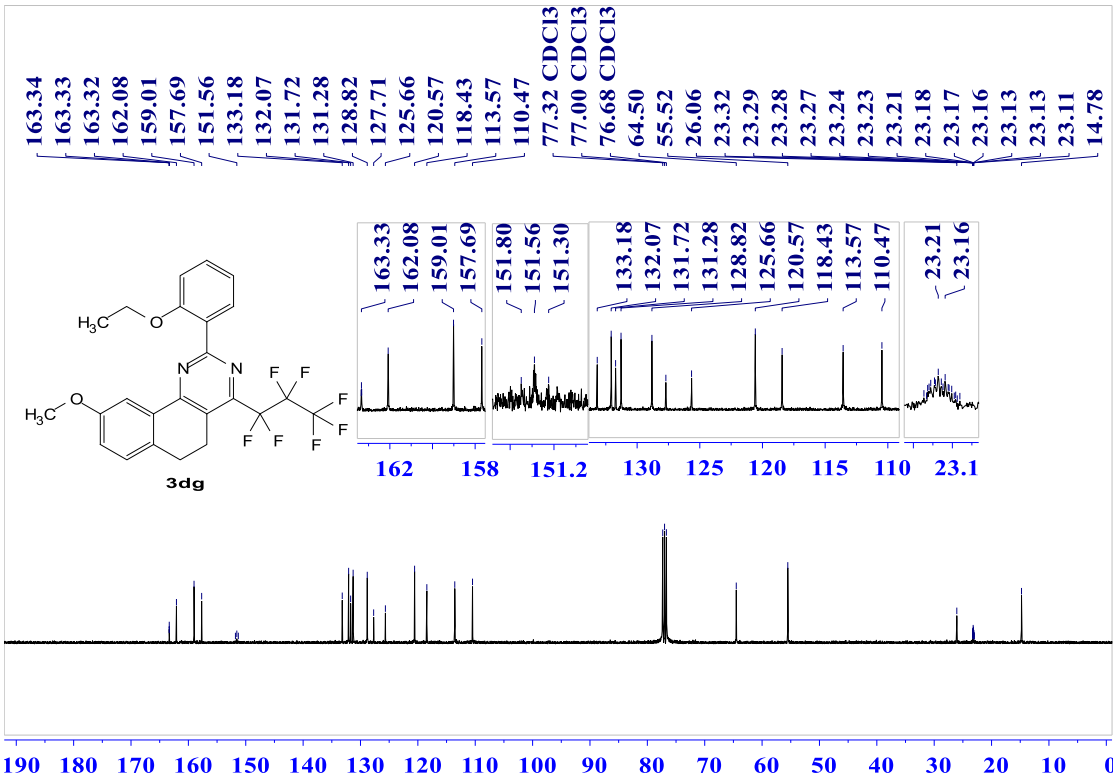

8.45
8.43
7.91
7.91
7.90
7.89
7.41
7.41
7.40
7.25 CDCl3
7.08
7.06
7.06
7.05
7.05
7.05
7.03
7.03
6.92
6.91
6.90
6.89
6.77
6.76
4.17
4.15
4.13
4.12
3.86
3.15
3.13
2.96
2.95
2.94
2.92
1.42
1.41
1.39
3cg
0.97
0.97
0.98
2.03
1.00
0.97
2.00
2.92
1.97
2.03
3.07
8.5
8.0
7.5
7.0
6.8
3.1
2.9
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1

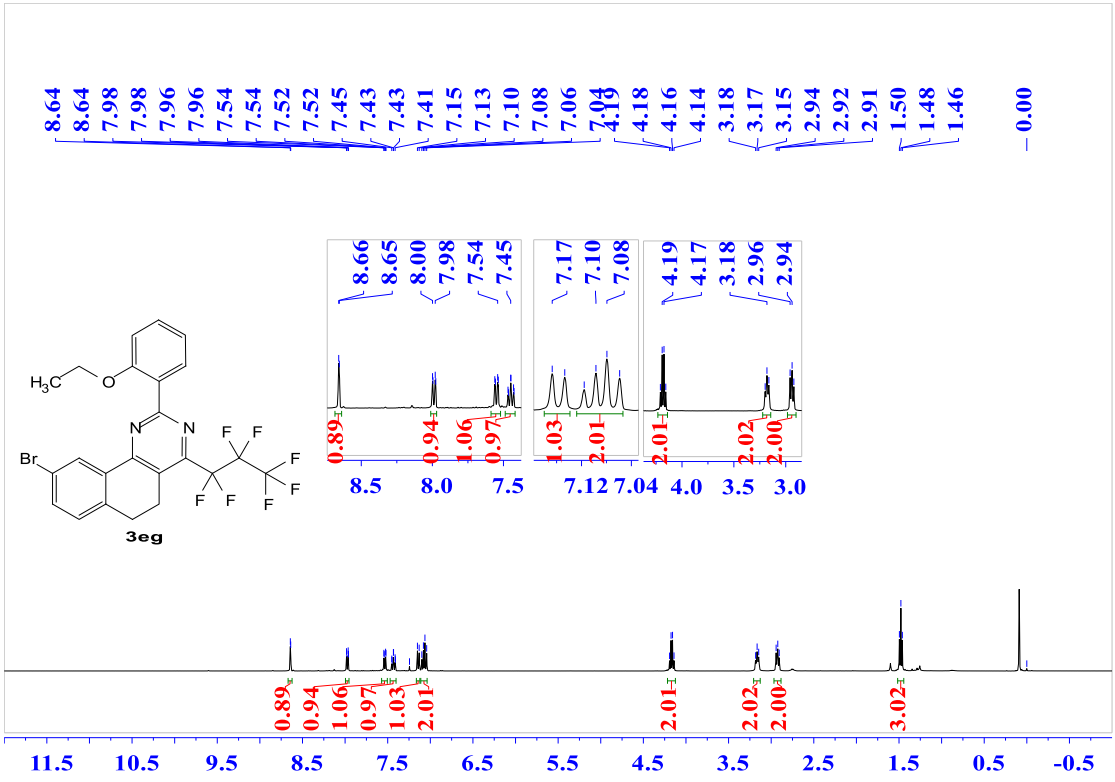

-79.76
-79.79
-79.83
-110.39
-110.42
-125.40
-125.45
-125.49
3.18
2.00
1.88
-79.5-80.0
-110.0
-111.0
-126.0
3cg
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270

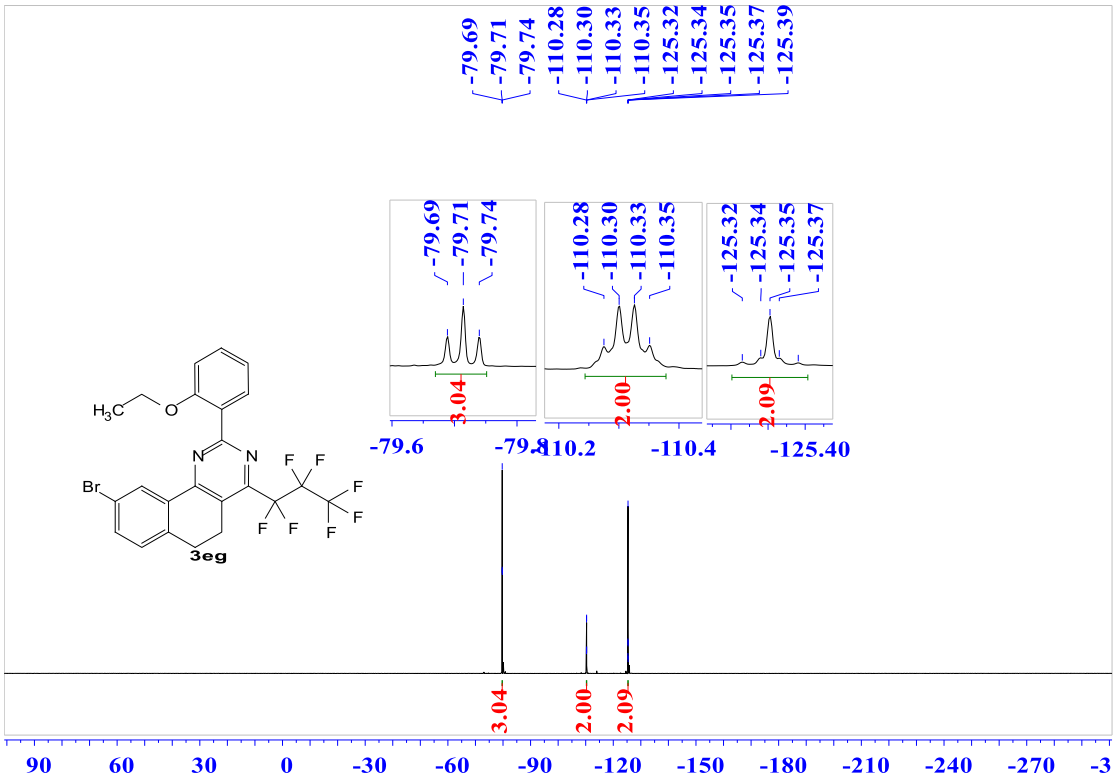

163.25
163.24
163.23
162.38
162.04
157.69
151.00
150.99
150.98
141.42
131.97
131.17
128.59
127.79
125.23
124.41
120.54
113.57
113.15
112.62
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.49
55.34
27.28
23.08
23.07
23.06
23.03
23.02
22.99
22.96
22.94
22.91
22.89
22.88
14.75
3cg
162 160 158 156 154 152
26 24
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

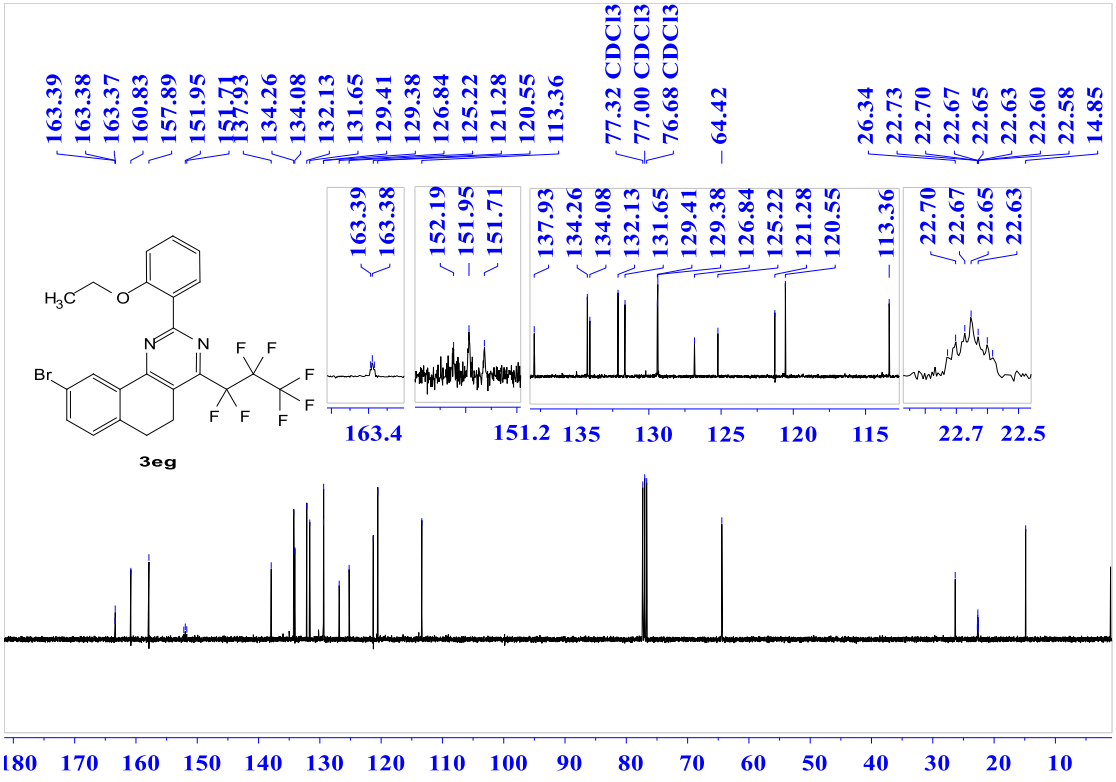

8.04 8.03 8.03 7.43 7.43 7.25 7.21 7.20 7.18 7.18 7.09 7.09 7.08 7.08 7.07 7.07 7.05 7.04 7.02 7.02 7.00 4.17 4.17 4.16 4.15 3.89 3.88 3.16 3.14 2.94 2.92 2.90 1.43 1.42 1.41 1.41 1.40 1.39
3dg
0.99 1.00 1.01 1.04 2.18 1.04 2.18 3.16 2.06 2.07 3.16

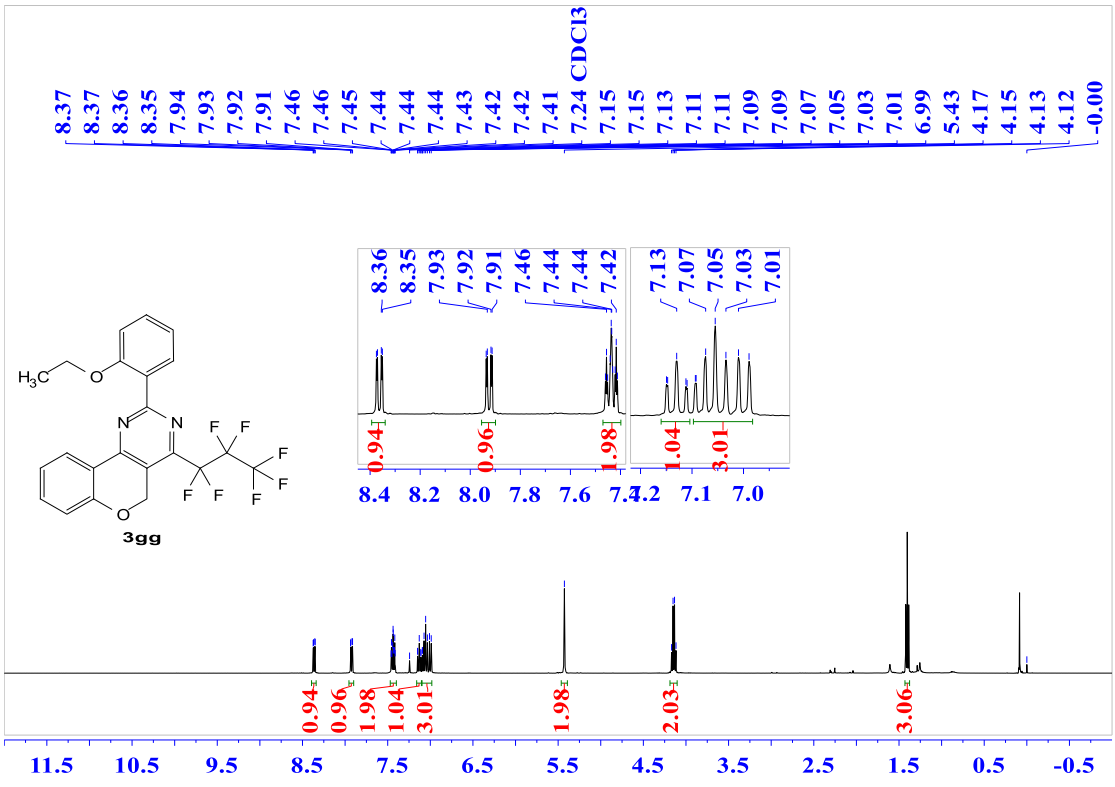

-79.73 -79.75 -79.78 -110.27 -110.29 -110.31 -110.33 -125.30 -125.34 -125.38
3dg
3.00 2.00 1.99
3.00 2.00 1.99

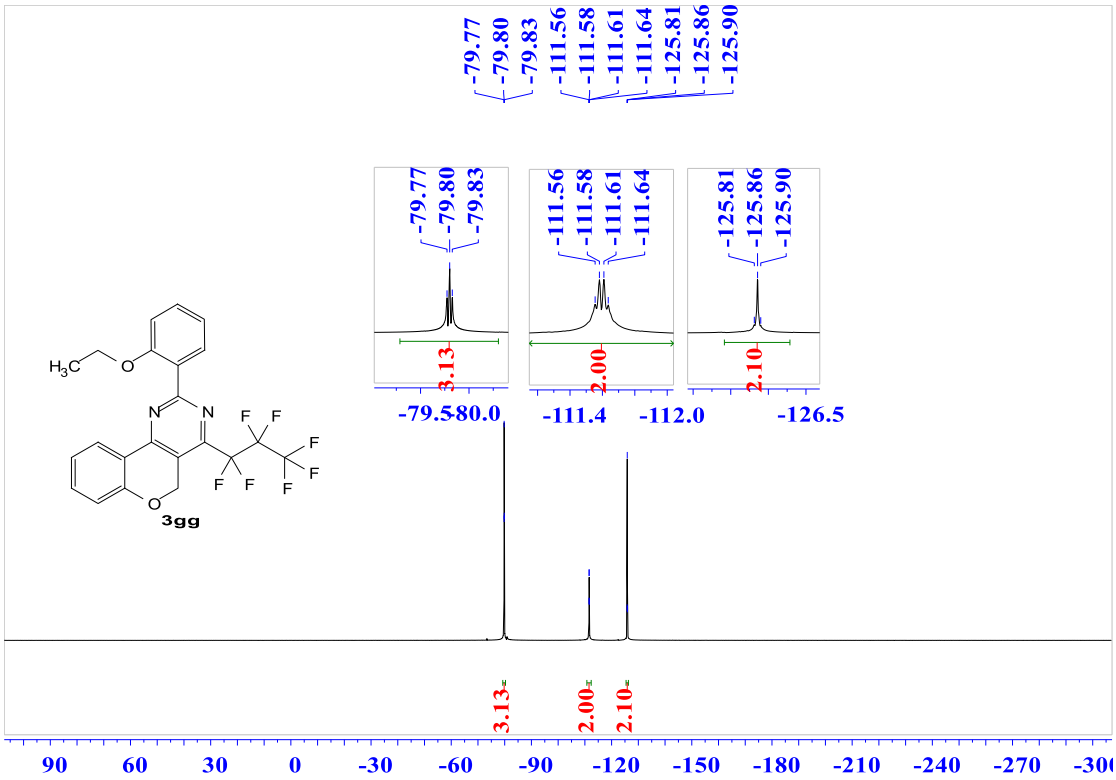

163.34
163.33
163.32
162.08
159.01
157.69
151.56
133.18
132.07
131.72
131.28
128.82
127.71
125.66
120.57
118.43
113.57
110.47
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.50
55.52
26.06
23.32
23.29
23.28
23.27
23.24
23.23
23.21
23.18
23.17
23.16
23.13
23.13
23.11
14.78
3dg
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

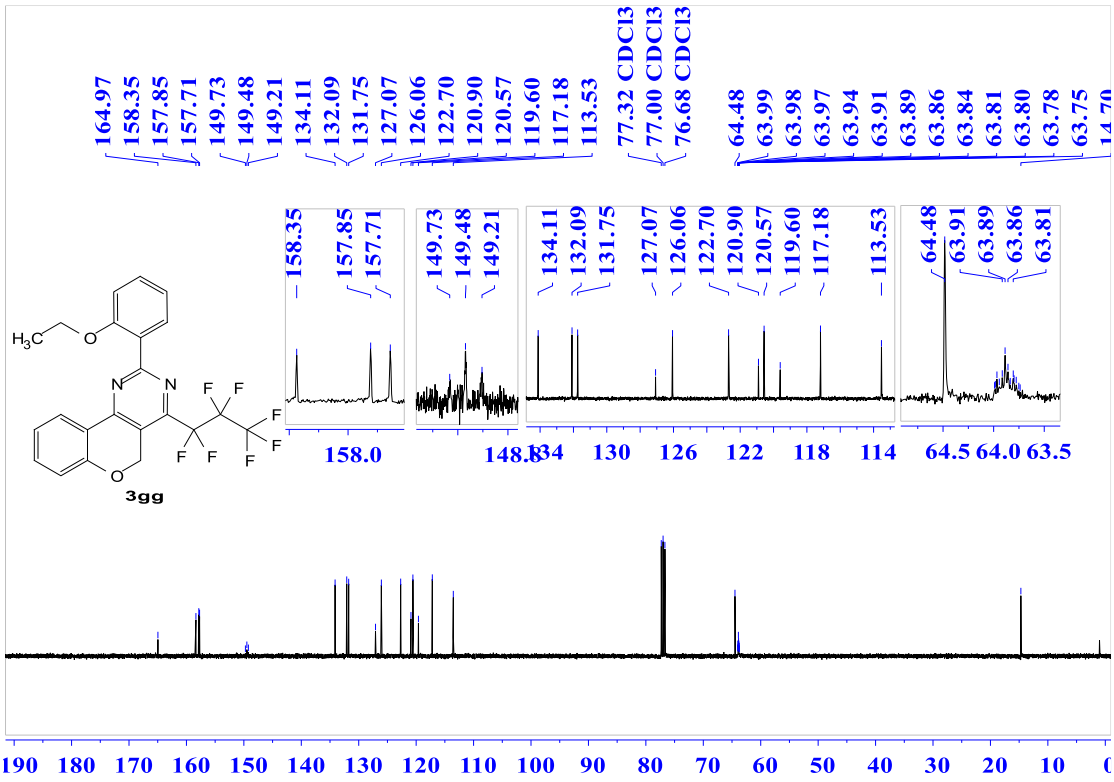

8.64
8.64
7.98
7.98
7.96
7.96
7.54
7.54
7.52
7.52
7.45
7.43
7.43
7.41
7.15
7.13
7.10
7.08
7.06
7.04
4.19
4.18
4.16
4.14
3.18
3.17
3.15
2.94
2.92
2.91
1.50
1.48
1.46
0.00
3eg
0.89
0.94
1.06
0.97
1.03
2.01
2.01
2.02
2.00
3.02
11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5

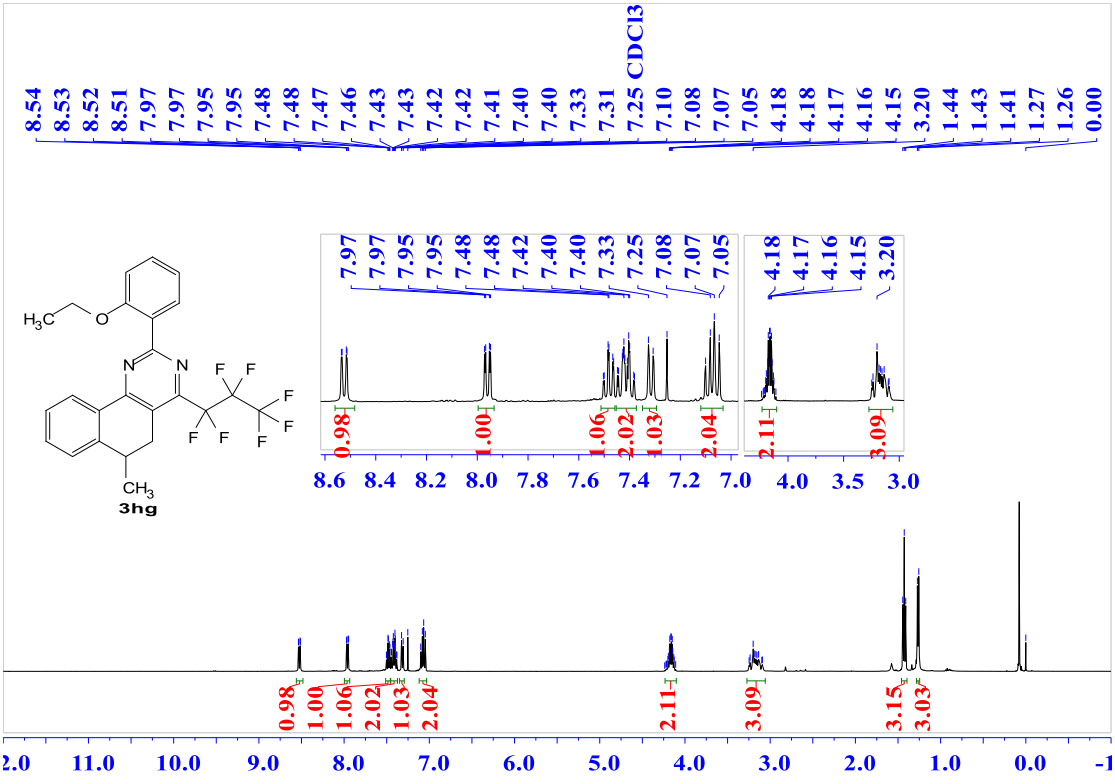

-79.69
-79.71
-79.74
-110.28
-110.30
-110.33
-110.35
-125.32
-125.34
-125.35
-125.37
-125.39
3.04
2.00
2.09
-79.6
-79.8
-110.2
-110.4
-125.40
Br
F
3eg
90
60
30
0
-30
-60
-90
-120
-150
-180
-210
-240
-270

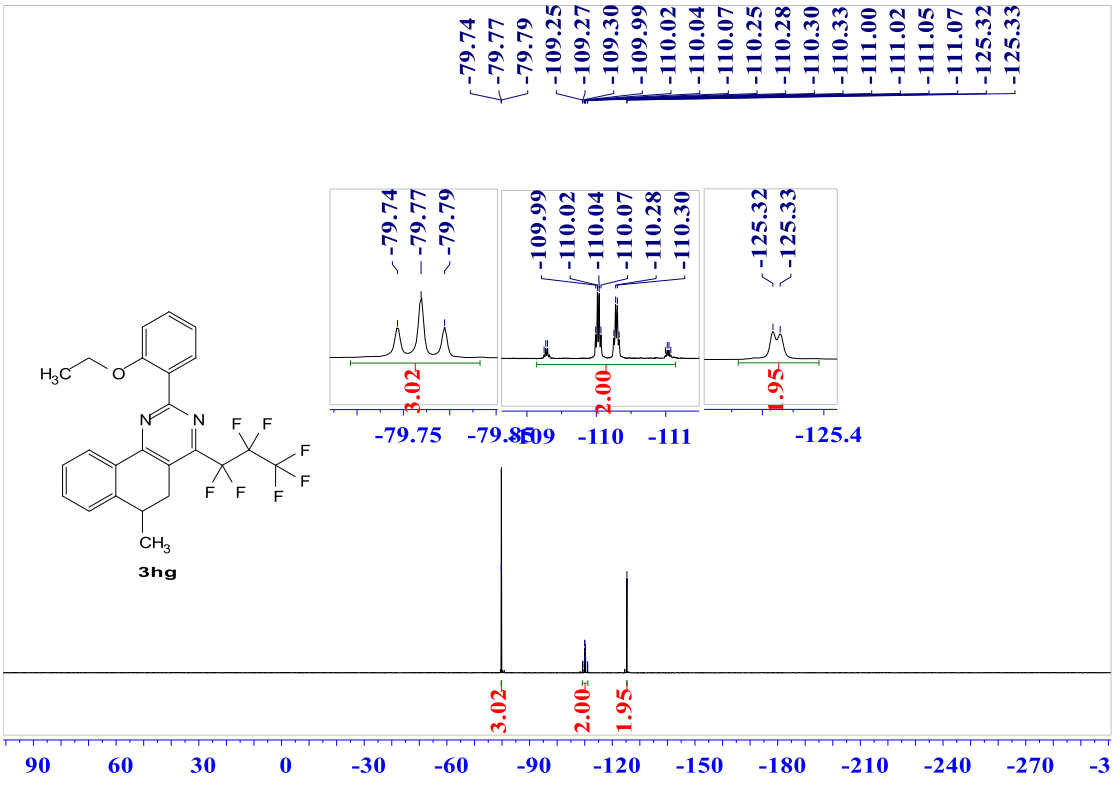

163.39
163.38
163.37
160.83
157.89
151.95
151.71
137.93
134.26
134.08
132.13
131.65
129.41
129.38
126.84
125.22
121.28
120.55
113.36
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.42
26.34
22.73
22.70
22.67
22.65
22.63
22.60
22.58
14.85
Br
F
3eg
163.4
151.2
135
130
125
120
115
22.7
22.5
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

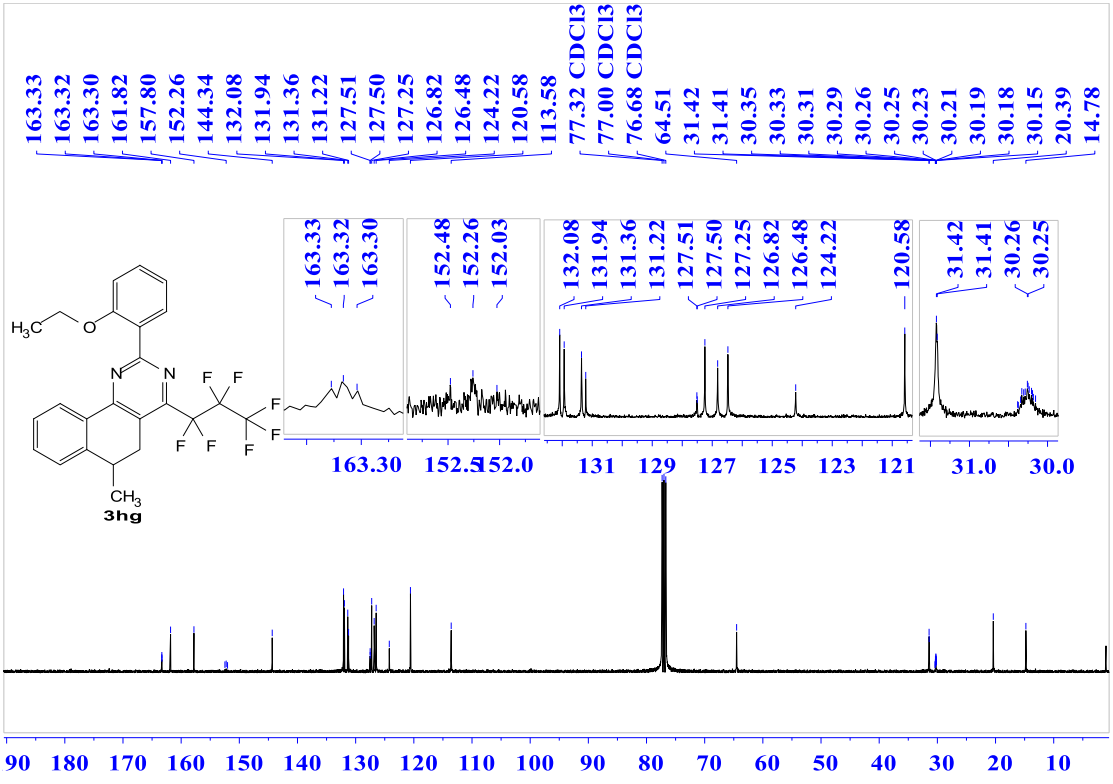

CDCl3
3gg

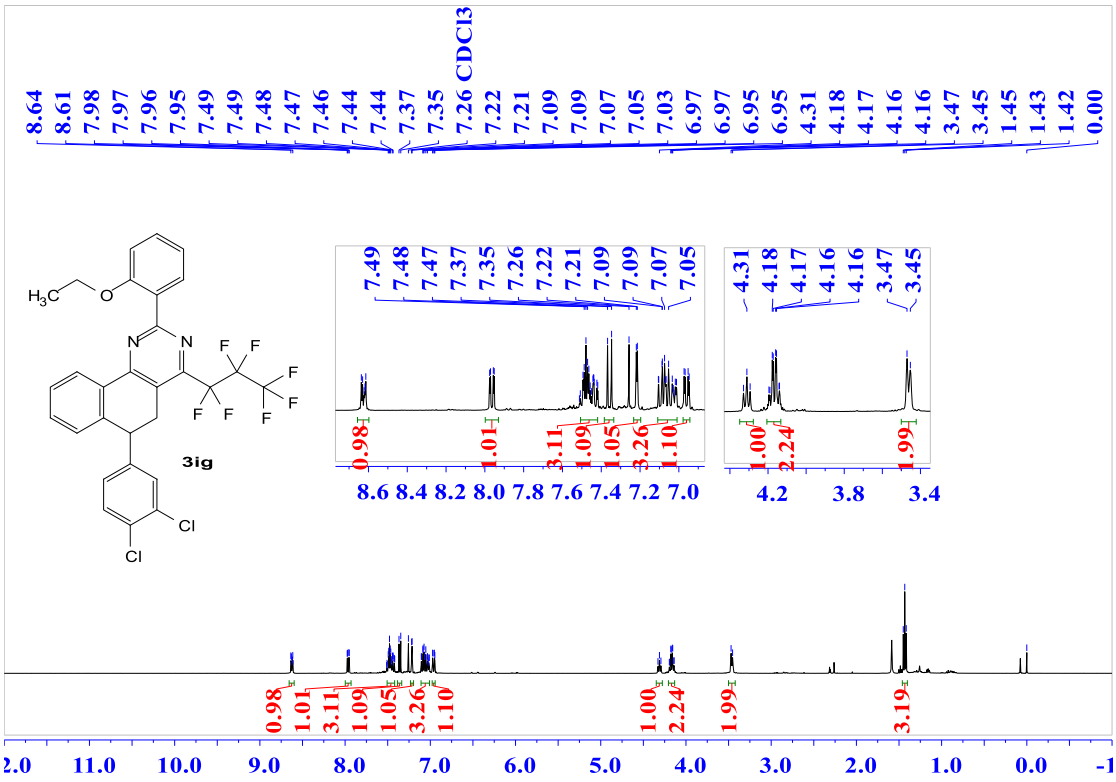

3gg

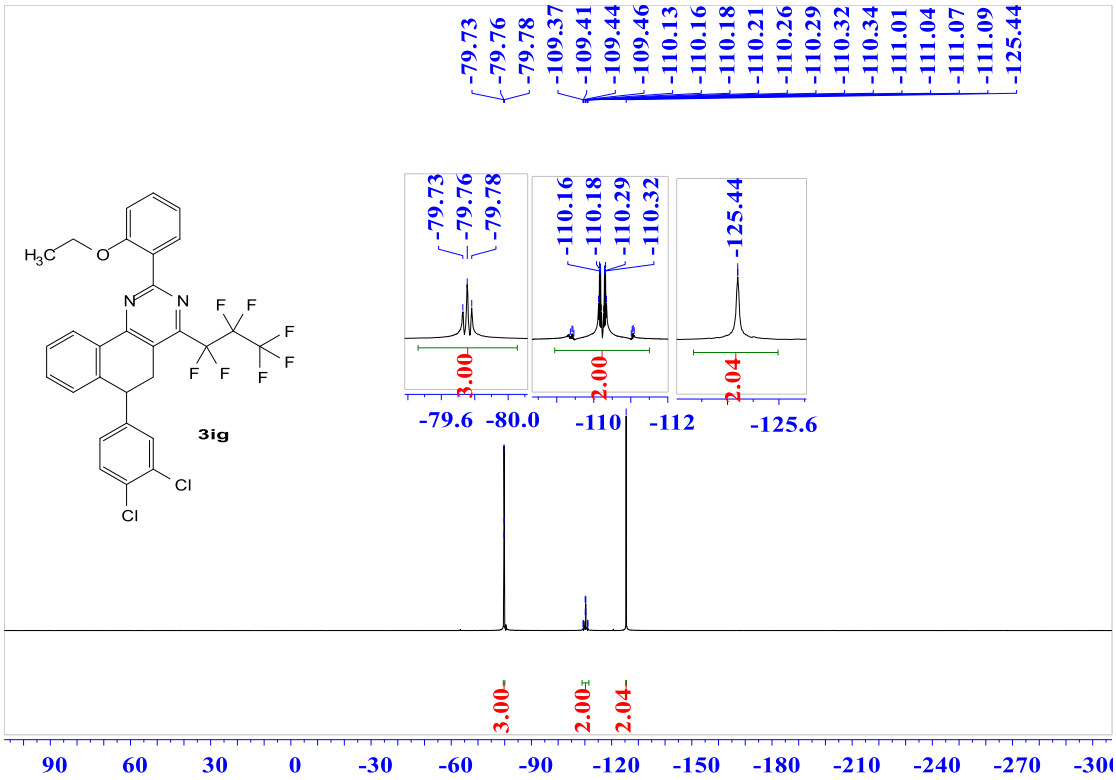

164.97
158.35
157.85
157.71
149.73
149.48
149.21
134.11
132.09
131.75
127.07
126.06
122.70
120.90
120.57
119.60
117.18
113.53
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.48
63.99
63.98
63.97
63.94
63.91
63.89
63.86
63.84
63.81
63.80
63.78
63.75
3gg
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

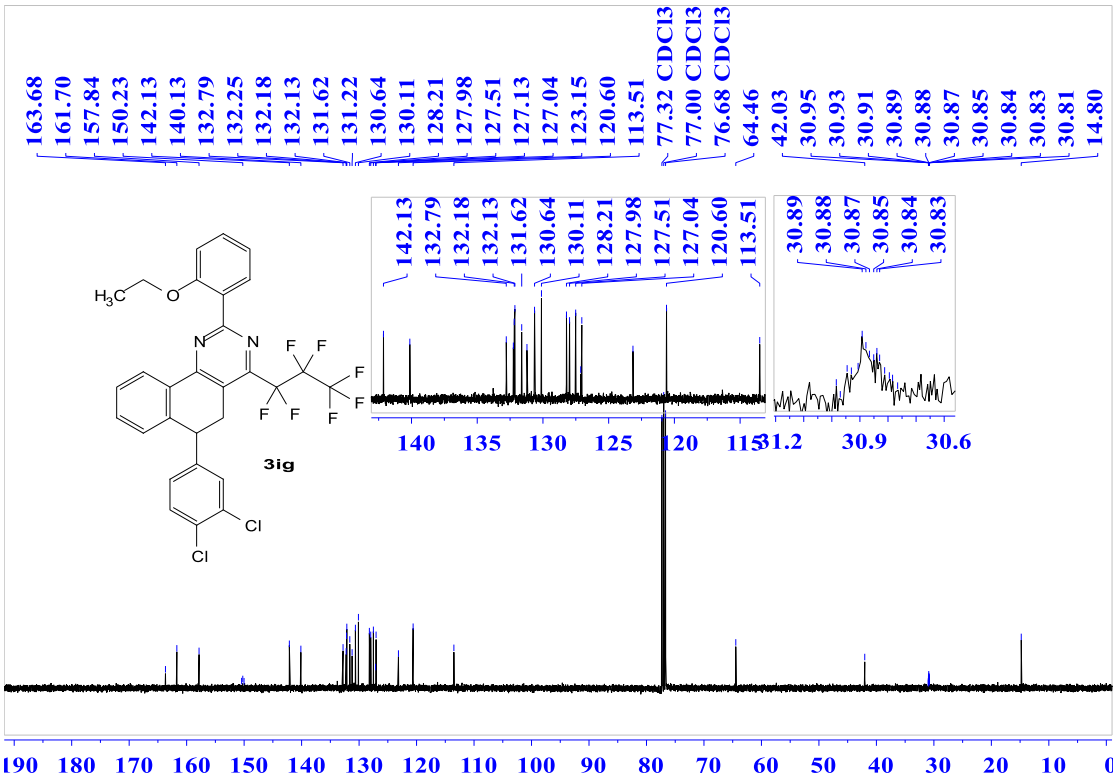

CDCl3
8.54
8.53
8.52
8.51
7.97
7.95
7.48
7.47
7.46
7.43
7.42
7.41
7.40
7.33
7.31
7.25
7.10
7.08
7.07
7.05
4.18
4.17
4.16
4.15
3.20
1.44
1.43
1.41
1.27
1.26
0.00
0.98
1.00
1.06
2.02
1.03
2.04
2.11
3.09
3.15
3.03
3hg
8.6 8.4 8.2 8.0 7.8 7.6 7.4 7.2 7.0 4.0 3.5 3.0
2.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

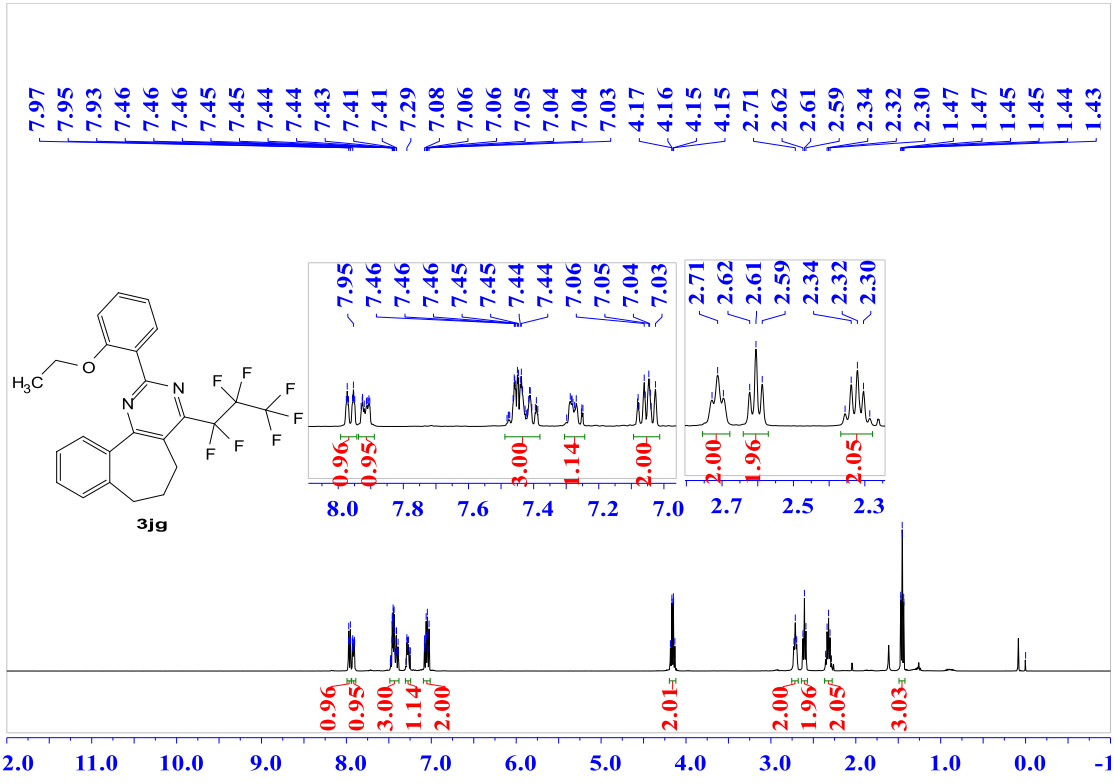

-79.74
-79.77
-79.79
-109.25
-109.27
-109.30
-109.99
-110.02
-110.04
-110.07
-110.25
-110.28
-110.30
-110.33
-111.00
-111.02
-111.05
-111.07
-125.32
-125.33
3.02
2.00
1.95
-79.75
-110
-111
-125.4
3hg
90
60
30
0
-30
-60
-90
-120
-150
-180
-210
-240
-270

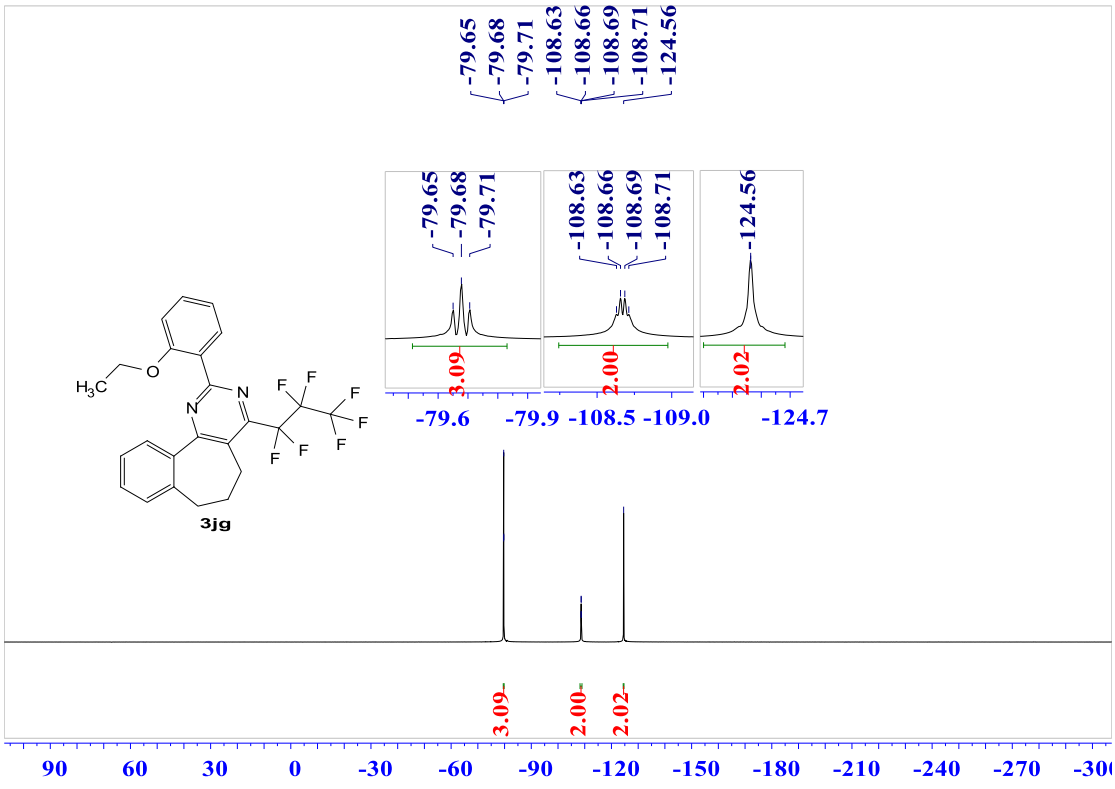

163.33
163.32
163.30
161.82
157.80
152.26
144.34
132.08
131.94
131.36
131.22
127.51
127.50
127.25
126.82
126.48
124.22
120.58
113.58
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.51
31.42
31.41
30.35
30.33
30.31
30.29
30.26
30.25
30.23
30.21
30.19
30.18
30.15
20.39
14.78
152.48
152.26
152.03
132.08
131.94
131.36
131.22
127.51
127.50
127.25
126.82
126.48
124.22
120.58
31.42
31.41
30.26
30.25
163.30
152.5
152.0
131
129
127
125
123
121
31.0
30.0
3hg
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

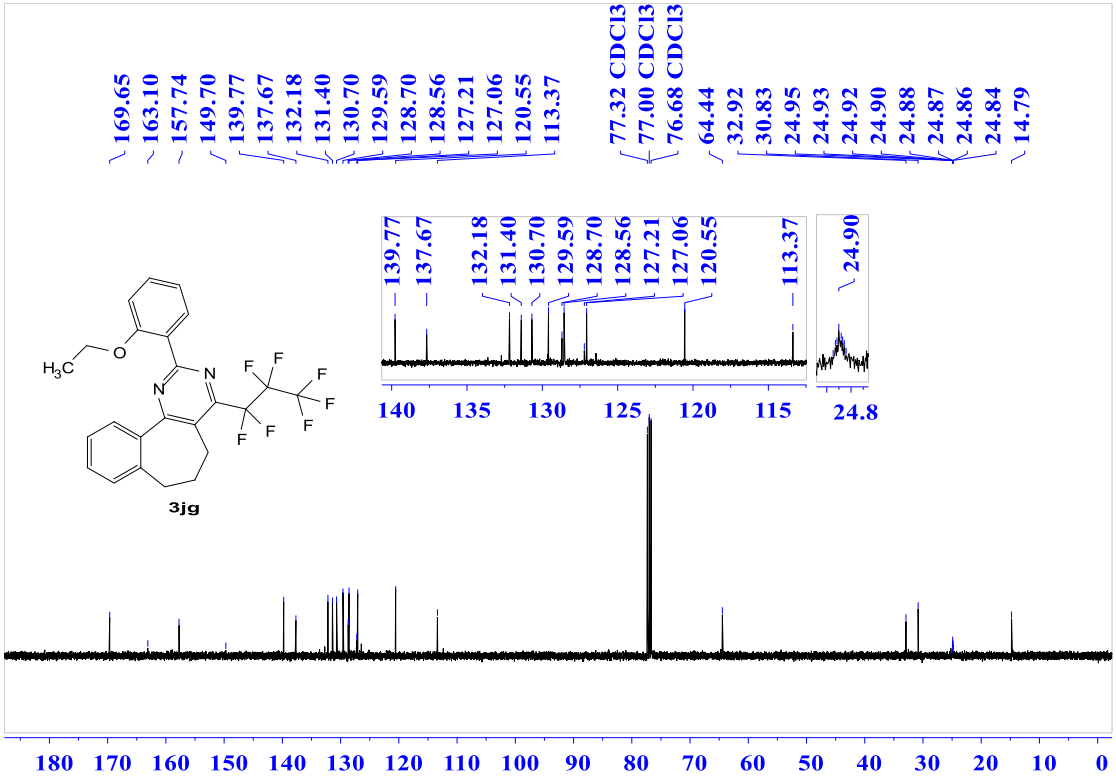

CDCl3
3ig

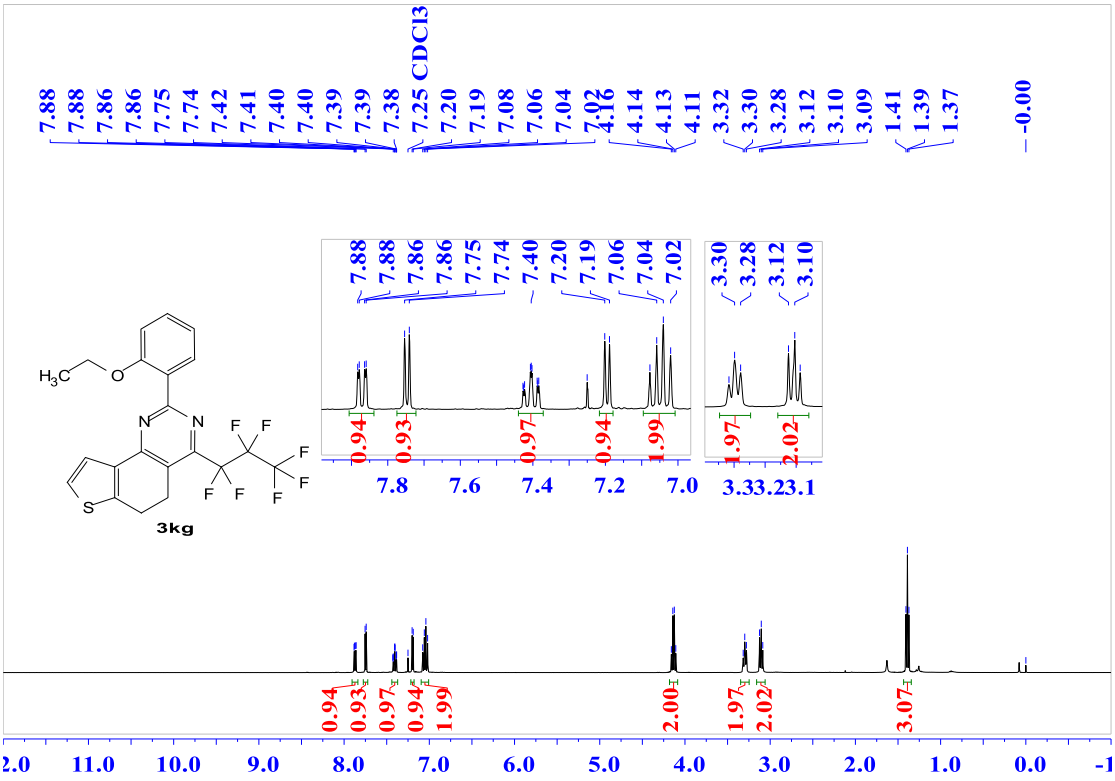

3ig

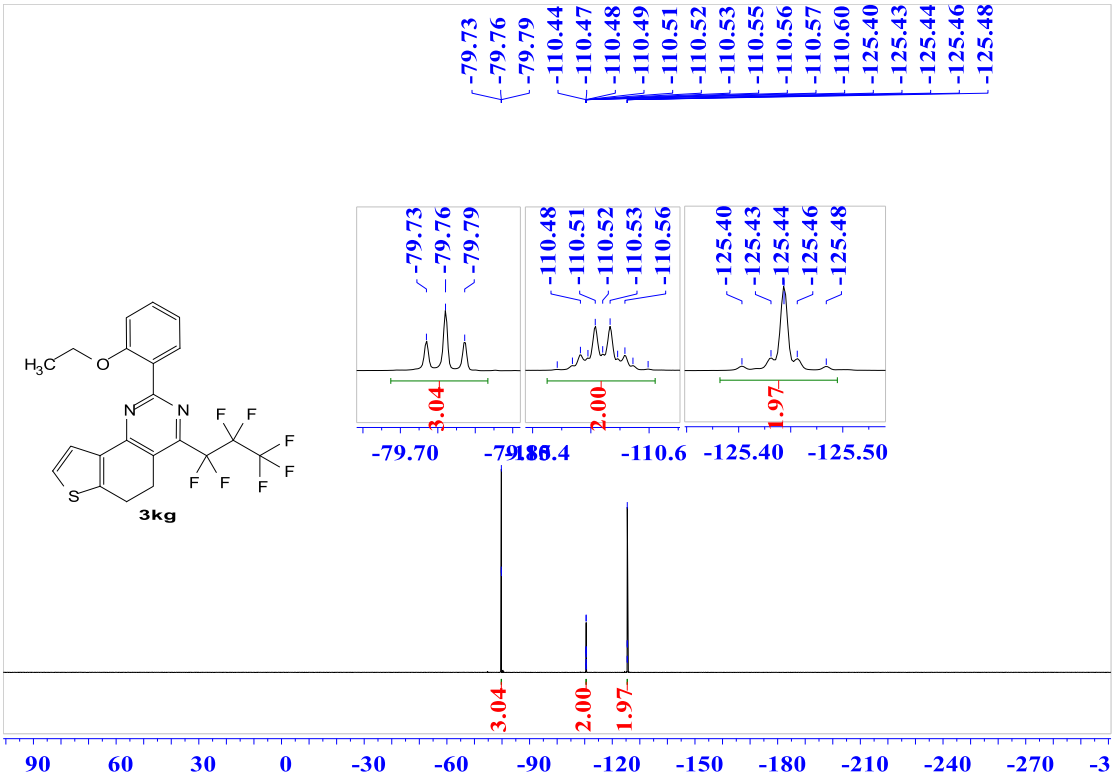

163.68 161.70 157.84 150.23 142.13 140.13 132.79 132.25 132.18 132.13 131.62 131.22 130.64 130.11 128.21 127.98 127.51 127.13 127.04 123.15 120.60 113.51 77.32 CDCl3 77.00 CDCl3 76.68 CDCl3 64.46 42.03 30.95 30.93 30.91 30.89 30.88 30.87 30.85 30.84 30.83 30.81 14.80
3ig
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

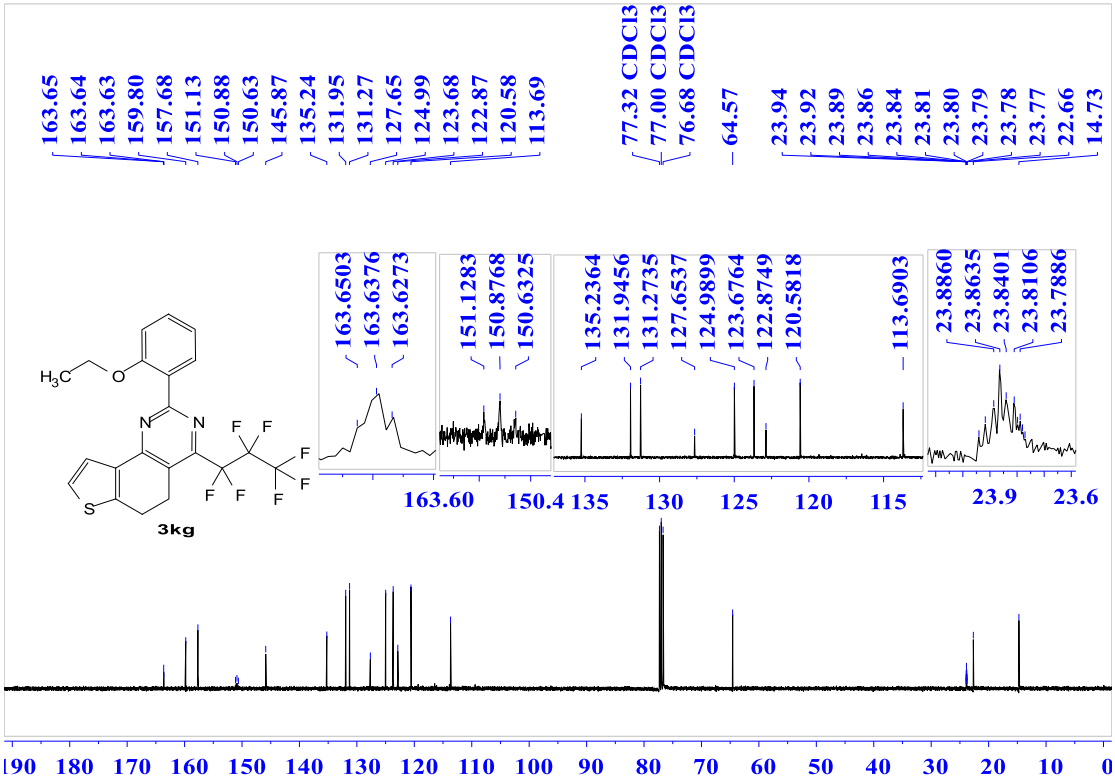

7.97 7.95 7.93 7.46 7.46 7.46 7.45 7.45 7.44 7.44 7.43 7.41 7.41 7.29 7.08 7.06 7.06 7.05 7.04 7.04 7.03 4.17 4.16 4.15 4.15 2.71 2.62 2.61 2.59 2.34 2.32 2.30 1.47 1.47 1.45 1.45 1.44 1.43
3jg
0.96 0.95 3.00 1.14 2.00 2.01 2.00 1.96 2.05 3.03
2.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

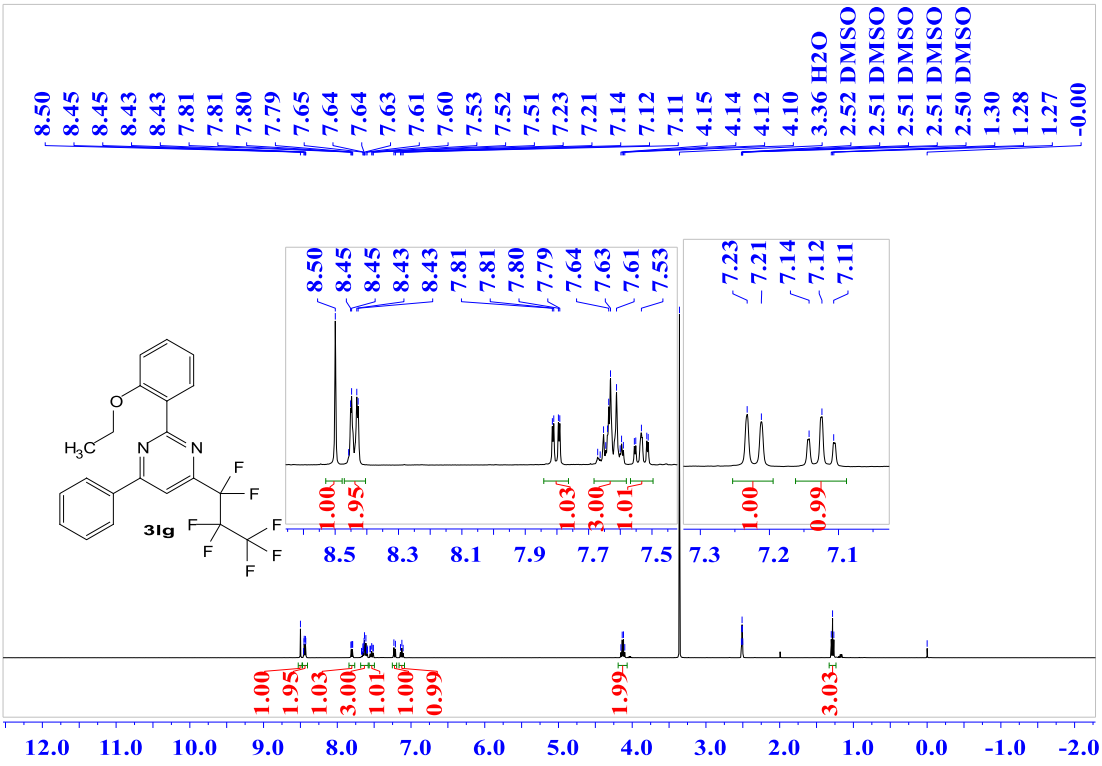

-79.65
-79.68
-79.71
-108.63
-108.66
-108.69
-108.71
-124.56
3.09
2.00
2.02
-79.6
-79.9
-108.5
-109.0
-124.7
3jg
90
60
30
0
-30
-60
-90
-120
-150
-180
-210
-240
-270

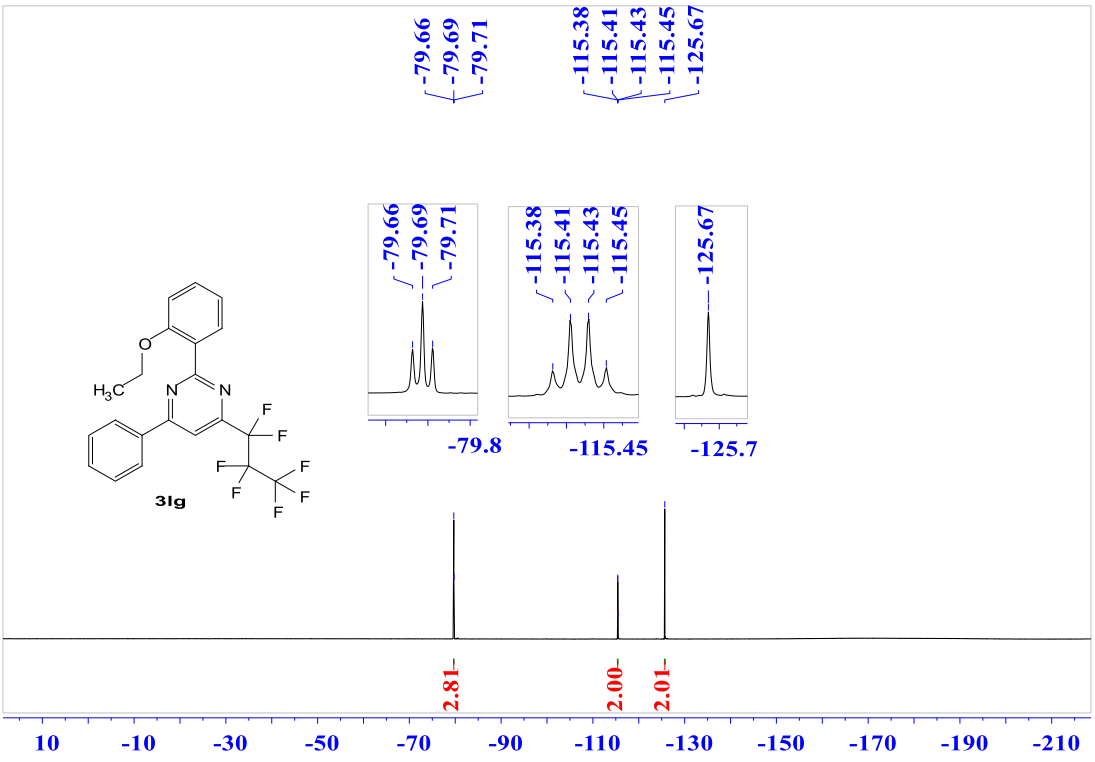

169.65
163.10
157.74
149.70
139.77
137.67
132.18
131.40
130.70
129.59
128.70
128.56
127.21
127.06
120.55
113.37
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.44
32.92
30.83
24.95
24.93
24.92
24.90
24.88
24.87
24.86
24.84
14.79
24.90
140
135
130
125
120
115
24.8
3jg
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

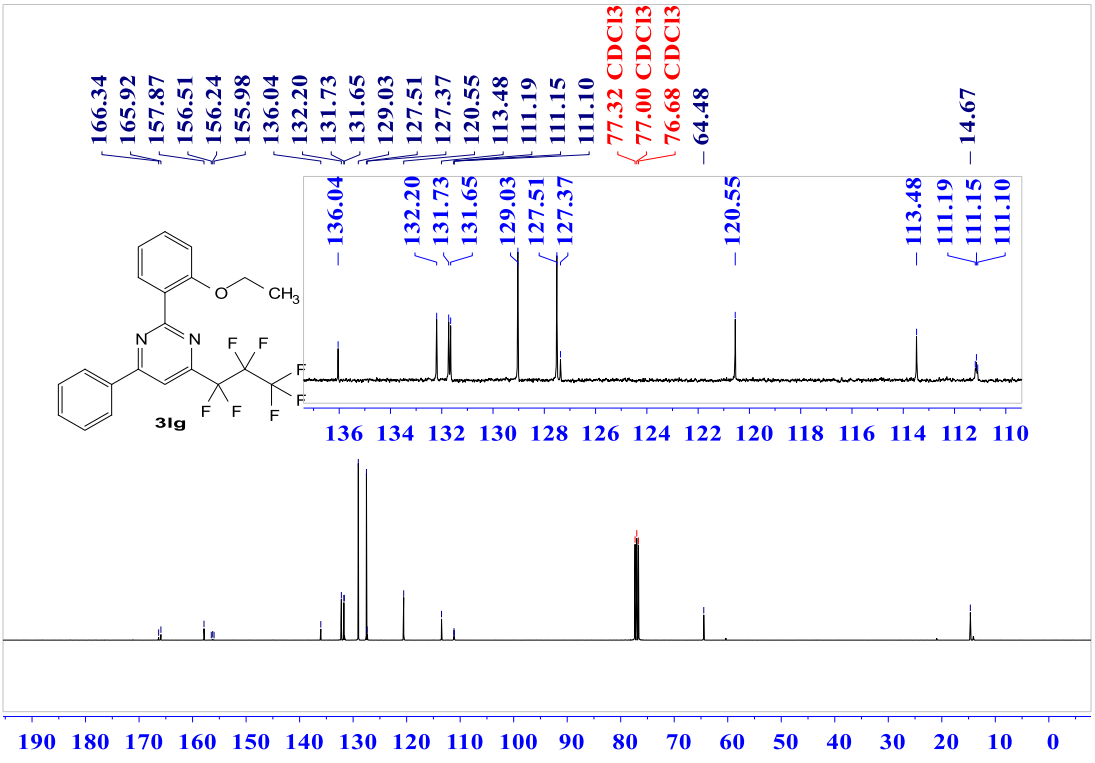

7.88 7.88 7.86 7.86 7.75 7.74 7.42 7.41 7.40 7.40 7.39 7.39 7.38 7.25 CDCl3 7.20 7.19 7.08 7.06 7.04 7.02 4.16 4.14 4.13 4.11 3.32 3.30 3.28 3.12 3.10 3.09 1.41 1.39 1.37 -0.00
3kg
0.94 0.93 0.97 0.94 1.99 2.00 1.97 2.02 3.07

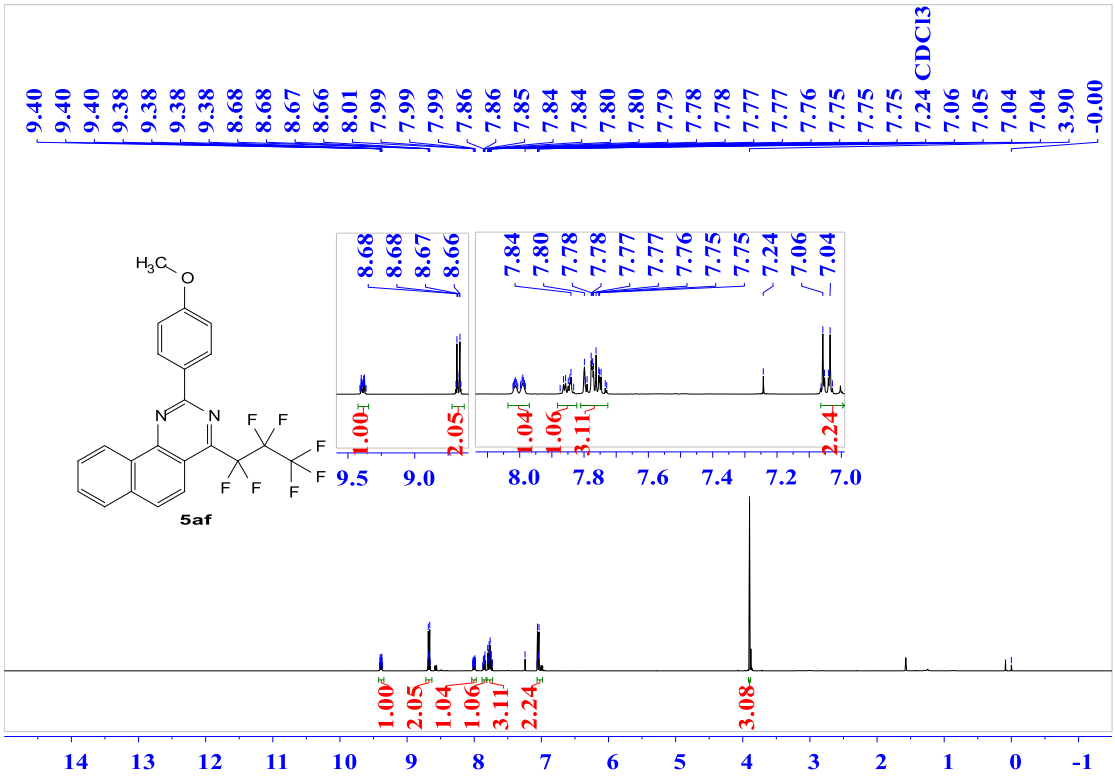

-79.73 -79.76 -79.79 -110.44 -110.47 -110.48 -110.49 -110.51 -110.52 -110.53 -110.55 -110.56 -110.57 -110.60 -125.40 -125.43 -125.44 -125.46 -125.48
3kg
3.04 2.00 1.97

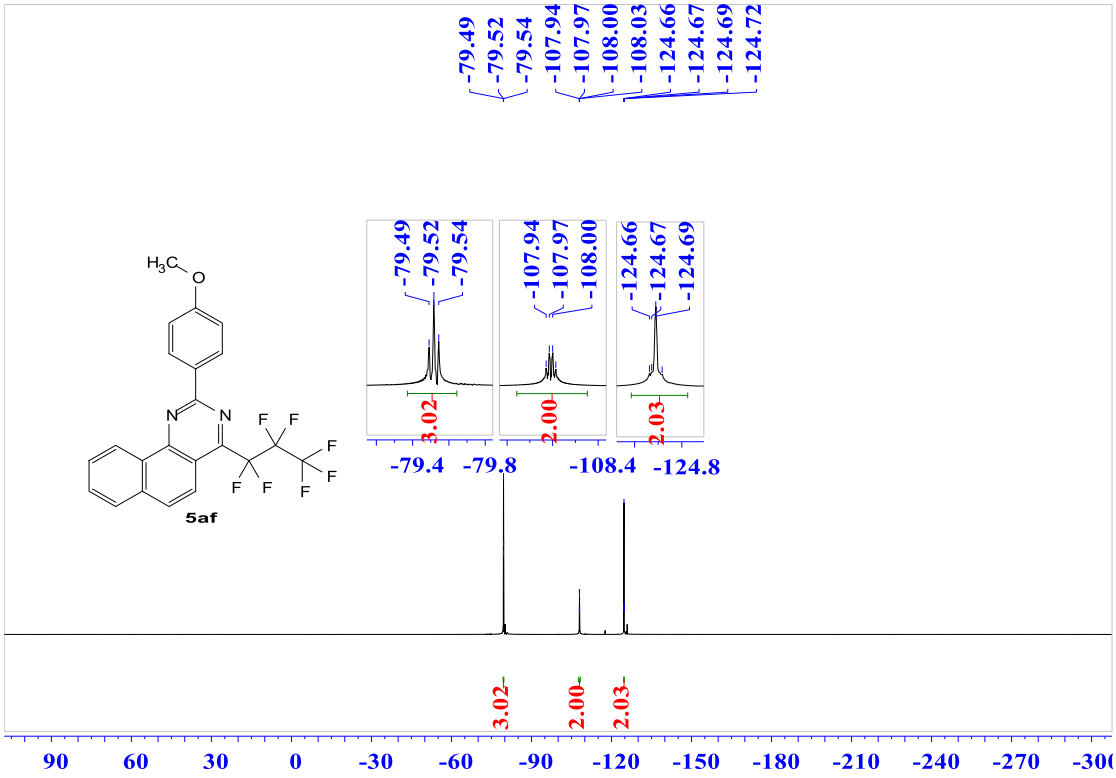

163.65
163.64
163.63
159.80
157.68
151.13
150.88
150.63
145.87
135.24
131.95
131.27
127.65
124.99
123.68
122.87
120.58
113.69
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.57
23.94
23.92
23.89
23.86
23.84
23.81
23.80
23.79
23.78
23.77
22.66
14.73
3kg
163.6503
163.6376
163.6273
151.1283
150.8768
150.6325
135.2364
131.9456
131.2735
127.6537
124.9899
123.6764
122.8749
120.5818
113.6903
23.8860
23.8635
23.8401
23.8106
23.7886

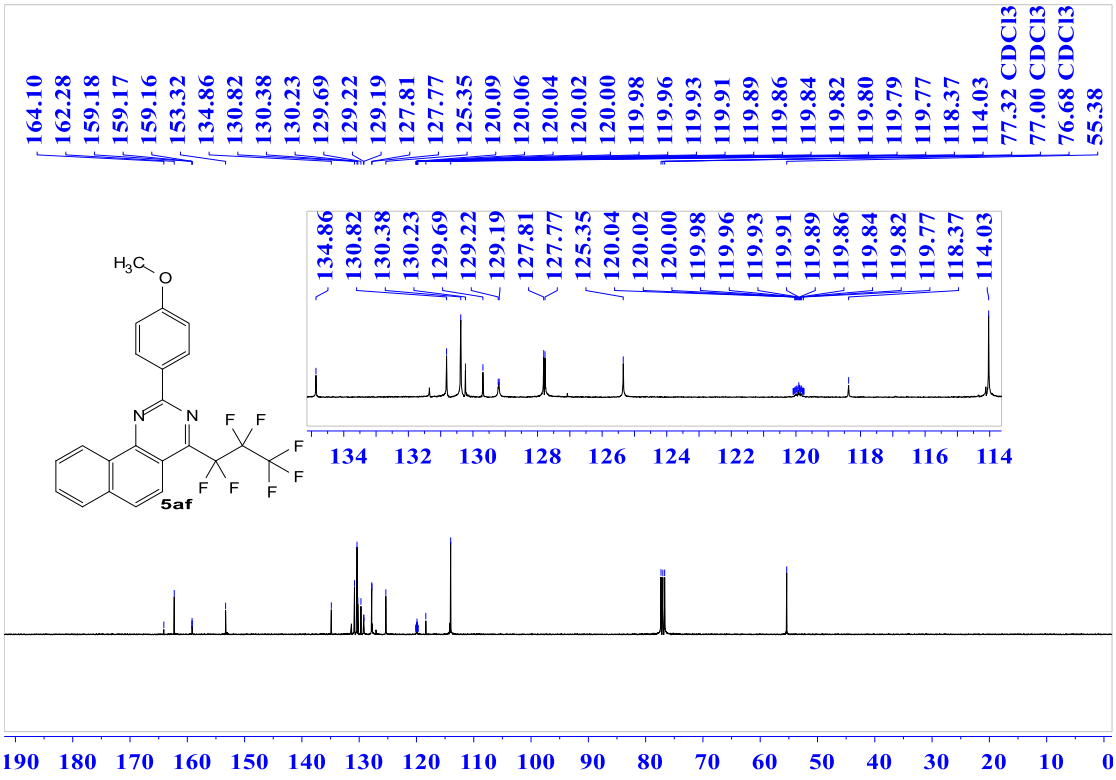

8.50
8.45
8.45
8.43
8.43
7.81
7.81
7.80
7.79
7.65
7.64
7.64
7.63
7.61
7.60
7.53
7.52
7.51
7.23
7.21
7.14
7.12
7.11
4.15
4.14
4.12
4.10
3.36 H2O
2.52 DMSO
2.51 DMSO
2.51 DMSO
2.51 DMSO
2.50 DMSO
1.30
1.28
1.27
-0.00
3lg
1.00
1.95
1.03
3.00
1.01
1.00
0.99
1.99
3.03

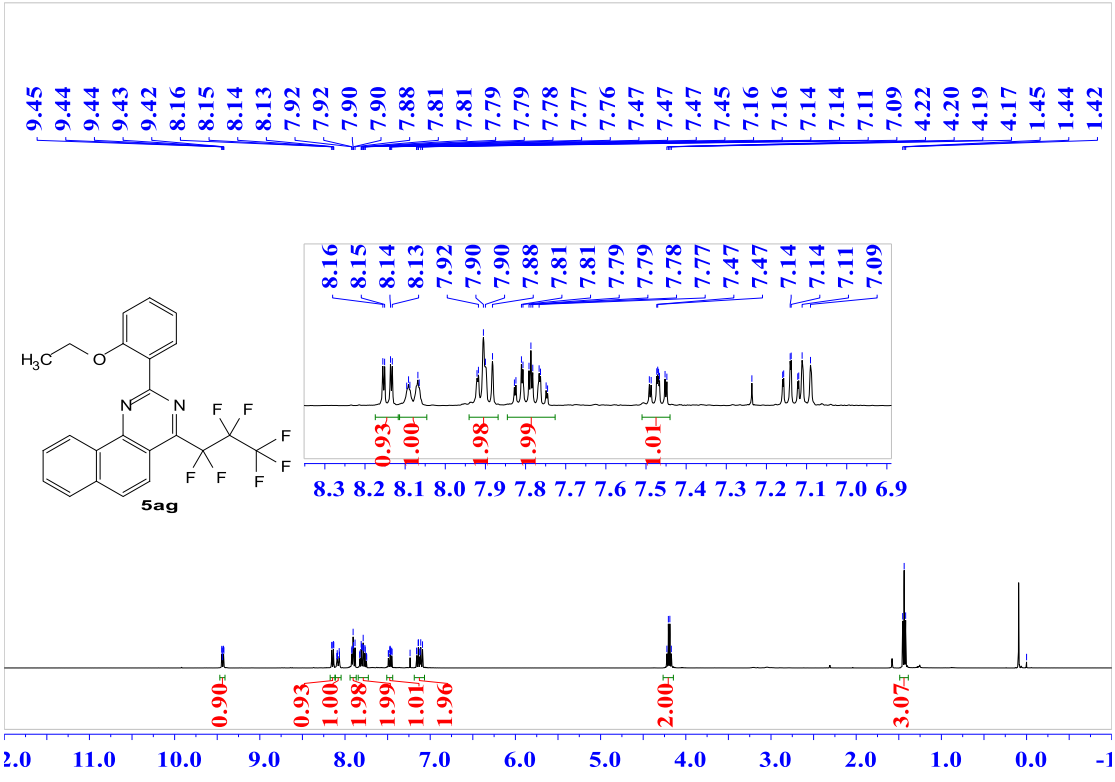

-79.66
-79.69
-79.71
-115.38
-115.41
-115.43
-115.45
-125.67
-79.8
-115.45
-125.7
3Ig
2.81
2.00
2.01
10 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210

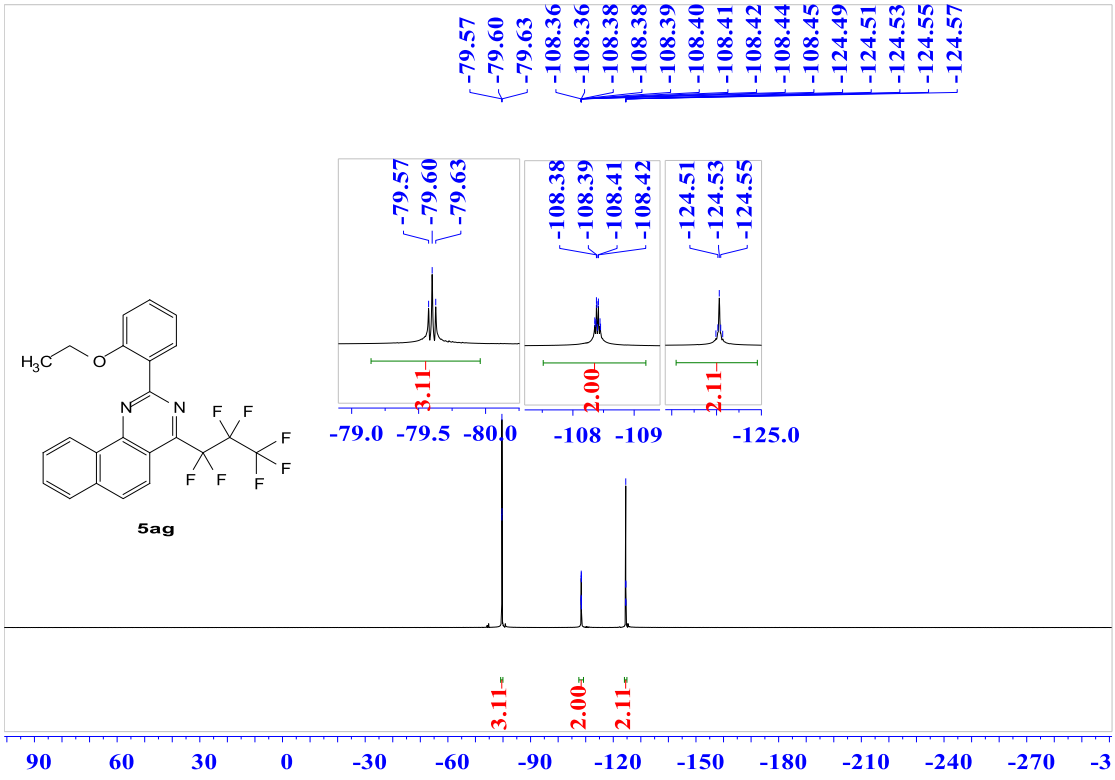

166.34
165.92
157.87
156.51
156.24
155.98
136.04
132.20
131.73
131.65
129.03
127.51
127.37
120.55
113.48
111.19
111.15
111.10
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.48
14.67
3Ig
136 134 132 130 128 126 124 122 120 118 116 114 112 110
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

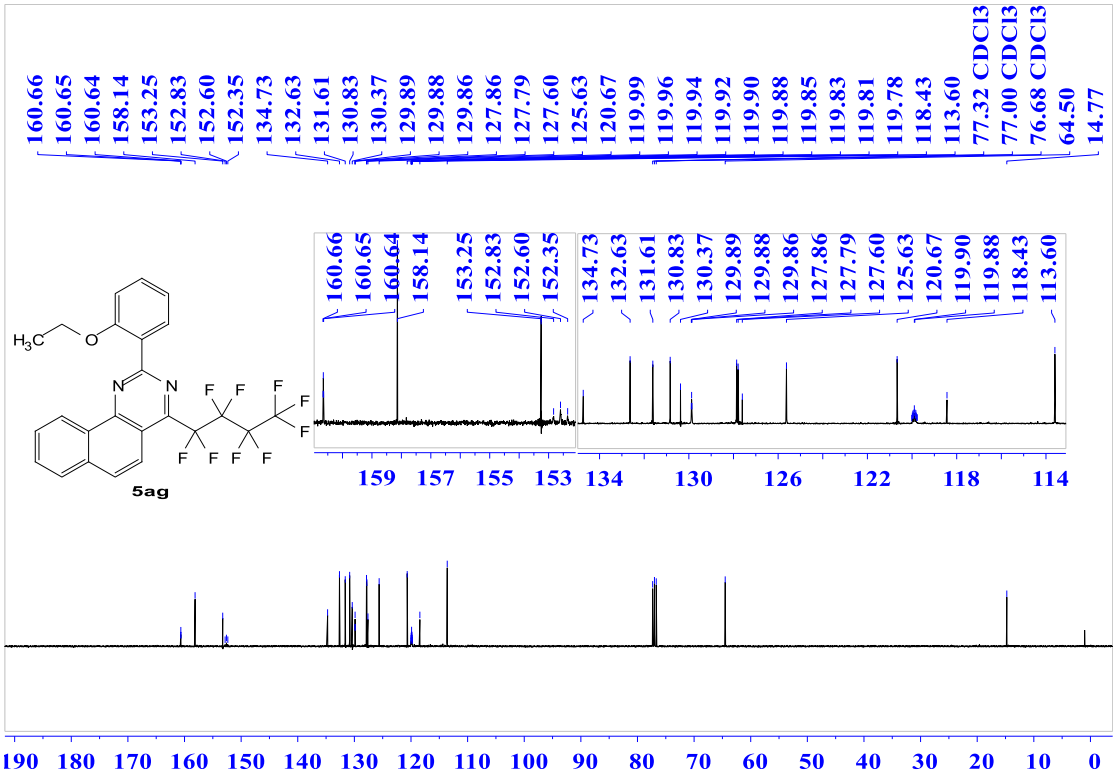

9.40 9.40 9.40 9.38 9.38 9.38 9.38 8.68 8.68 8.67 8.66 8.01 7.99 7.99 7.99 7.86 7.86 7.85 7.84 7.84 7.80 7.80 7.79 7.78 7.78 7.77 7.77 7.76 7.75 7.75 7.75 7.24 CDCl3 7.06 7.05 7.04 7.04 3.90 -0.00
8.68 8.68 8.67 8.66
7.84 7.80 7.78 7.78 7.77 7.77 7.76 7.75 7.75 7.24 7.06 7.04
1.00 2.05 1.04 1.06 3.11 2.24
9.5 9.0 8.0 7.8 7.6 7.4 7.2 7.0
5af
1.00 2.05 1.04 1.06 3.11 2.24 3.08
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1

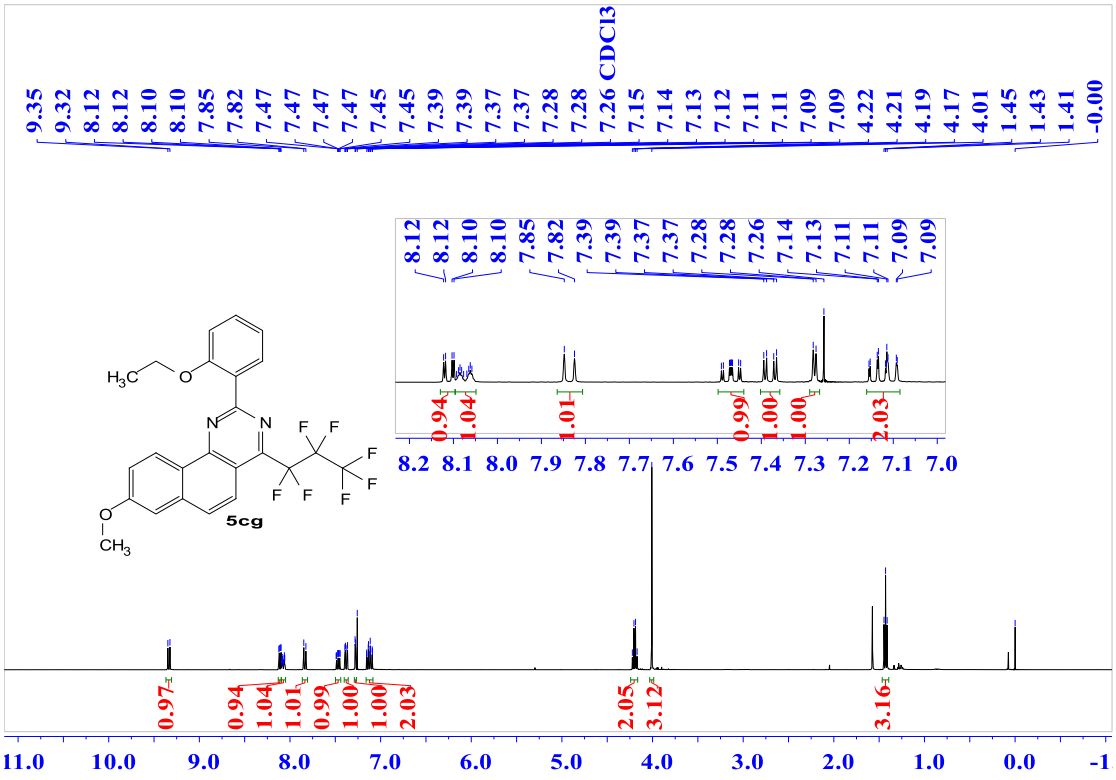

-79.49 -79.52 -79.54 -107.94 -107.97 -108.00 -108.03 -124.66 -124.67 -124.69 -124.72
-79.49 -79.52 -79.54
-107.94 -107.97 -108.00
-124.66 -124.67 -124.69
3.02 2.00 2.03
-79.4 -79.8 -108.4 -124.8
5af
3.02 2.00 2.03
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270 -300

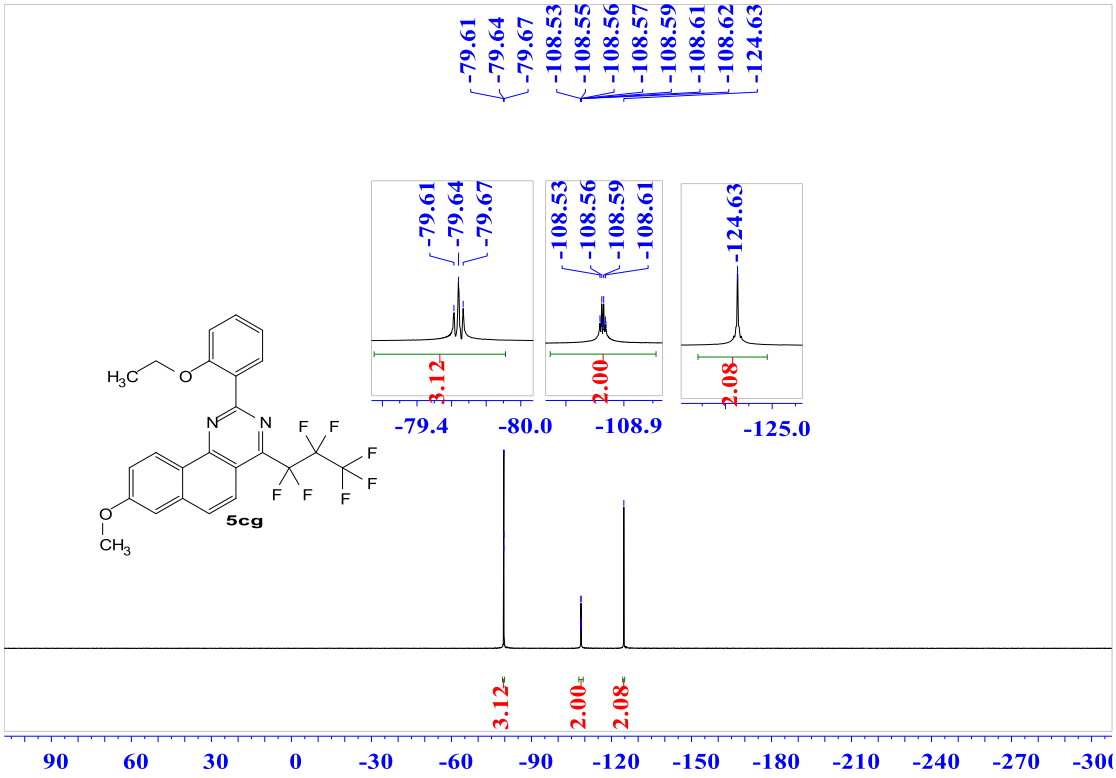

164.10
162.28
159.18
159.17
159.16
153.32
134.86
130.82
130.38
130.23
129.69
129.22
129.19
127.81
127.77
125.35
120.09
120.06
120.04
120.02
120.00
119.98
119.96
119.93
119.91
119.89
119.86
119.84
119.82
119.80
119.79
119.77
118.37
114.03
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
55.38
H3C
O
N
N
F
F
F
F
F
F
F
5af
134 132 130 128 126 124 122 120 118 116 114
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

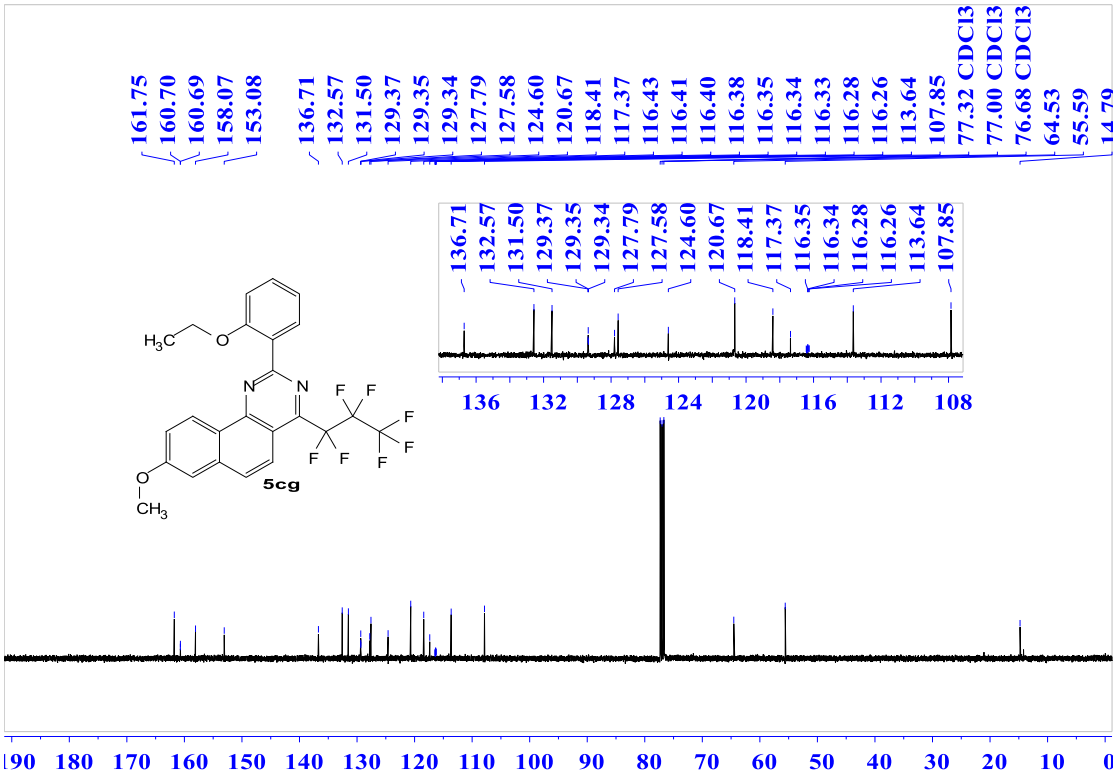

9.45
9.44
9.44
9.43
9.42
8.16
8.15
8.14
8.13
7.92
7.92
7.90
7.90
7.88
7.81
7.81
7.79
7.79
7.78
7.77
7.76
7.47
7.47
7.47
7.45
7.16
7.16
7.14
7.14
7.11
7.09
4.22
4.20
4.19
4.17
1.45
1.44
1.42
H3C
O
N
N
F
F
F
F
F
F
F
5ag
0.93
1.00
1.98
1.99
1.01
8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9
0.90
0.93
1.00
1.98
1.99
1.01
1.96
2.00
3.07
2.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1

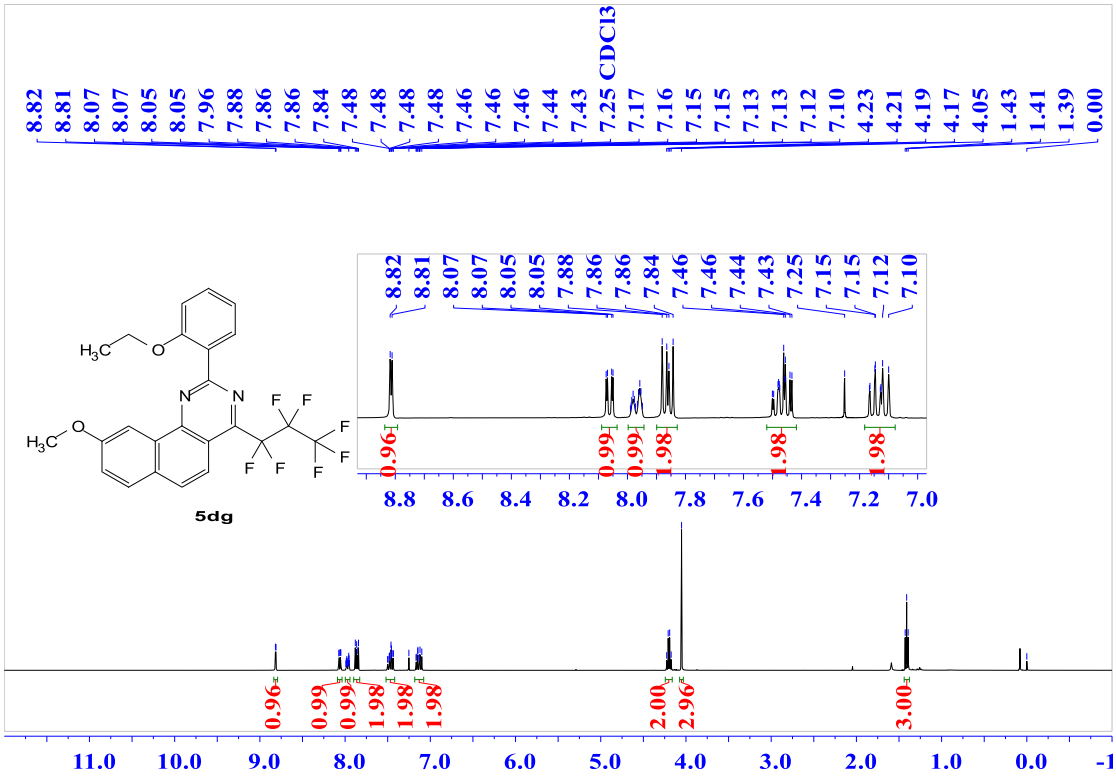

-79.57
-79.60
-79.63
-108.36
-108.36
-108.38
-108.38
-108.39
-108.40
-108.41
-108.42
-108.44
-108.45
-124.49
-124.51
-124.53
-124.55
-124.57
3.11
2.00
2.11
-79.0 -79.5 -80.0
-108 -109
-125.0
5ag
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270 -3

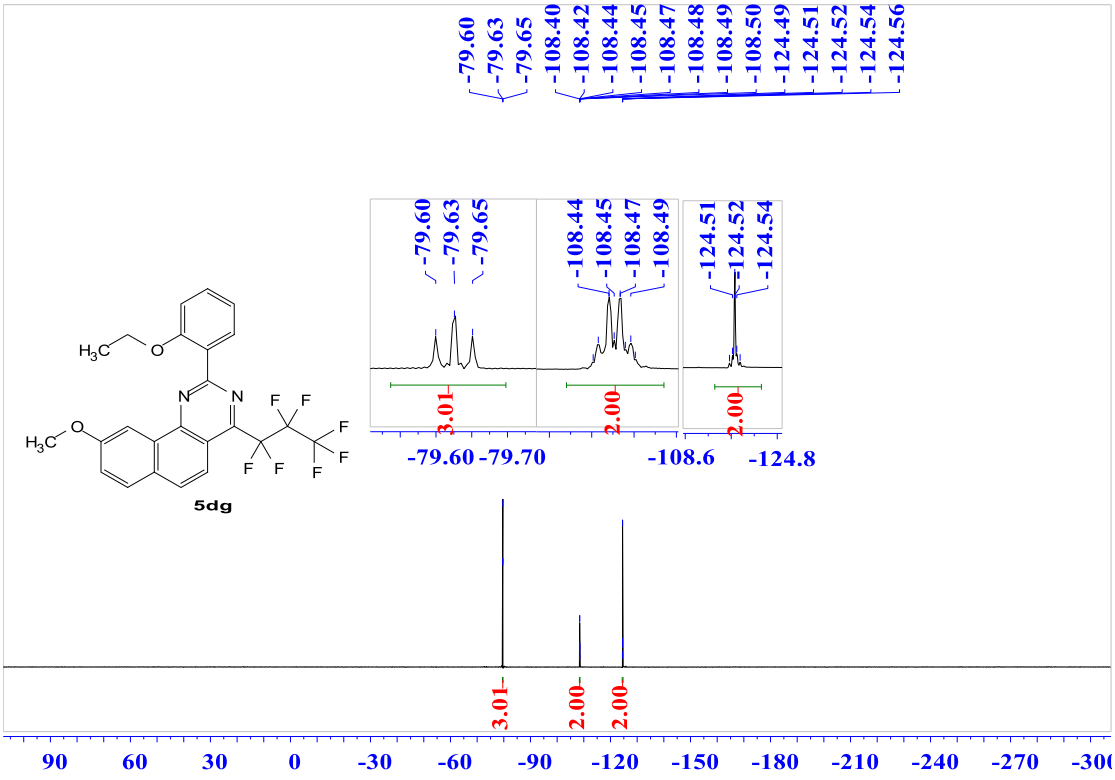

160.66
160.65
160.64
158.14
153.25
152.83
152.60
152.35
134.73
132.63
131.61
130.83
130.37
129.89
129.88
129.86
127.86
127.79
127.60
125.63
120.67
119.99
119.96
119.94
119.92
119.90
119.88
119.85
119.83
119.81
119.78
118.43
113.60
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.50
14.77
5ag
159 157 155 153 134 130 126 122 118 114
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

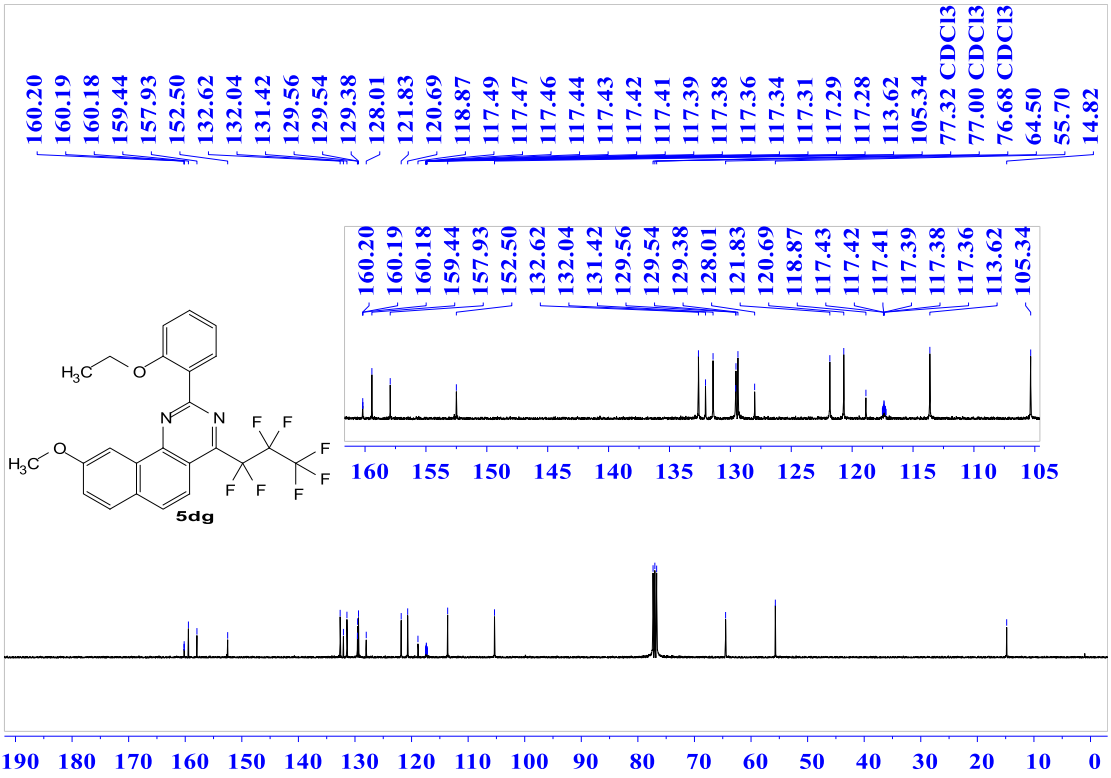

9.35 9.32 8.12 8.12 8.10 8.10 7.85 7.82 7.47 7.47 7.47 7.47 7.45 7.45 7.39 7.39 7.37 7.37 7.28 7.28 7.26 CDCl3 7.15 7.14 7.13 7.12 7.11 7.11 7.09 7.09 4.22 4.21 4.19 4.17 4.01 1.45 1.43 1.41 -0.00
5cg
0.97 0.94 1.04 1.01 0.99 1.00 1.00 2.03 2.05 3.12 3.16
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

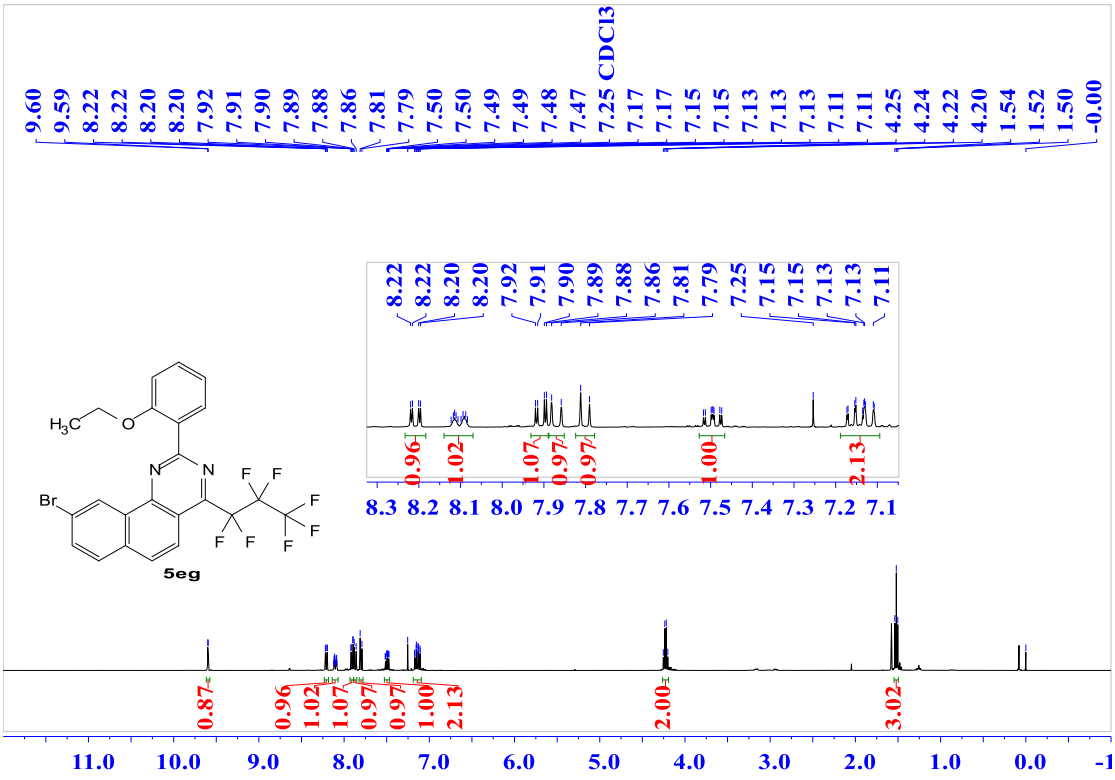

-79.61 -79.64 -79.67 -108.53 -108.55 -108.56 -108.57 -108.59 -108.61 -108.62 -124.63
5cg
3.12 2.00 2.08
-79.4 -80.0 -108.9 -125.0
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270

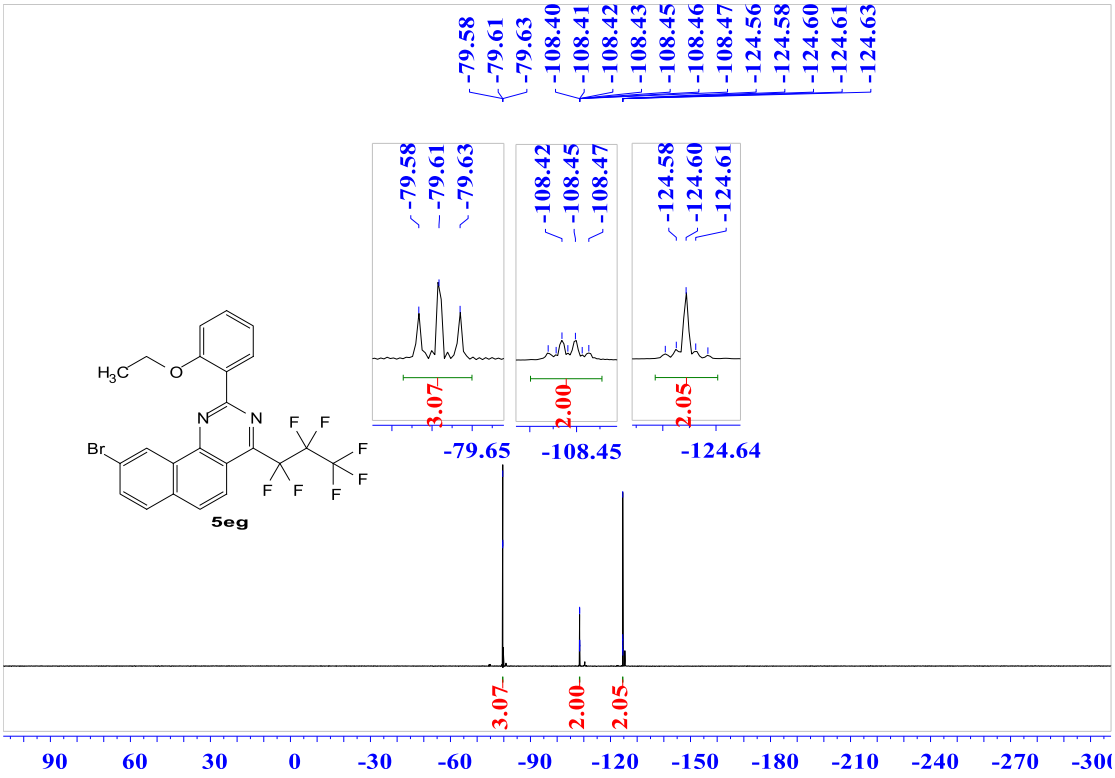

161.75
160.70
160.69
158.07
153.08
136.71
132.57
131.50
129.37
129.35
129.34
127.79
127.58
124.60
120.67
118.41
117.37
116.43
116.41
116.40
116.38
116.35
116.34
116.33
116.28
116.26
113.64
107.85
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.53
55.59
14.79
5cg

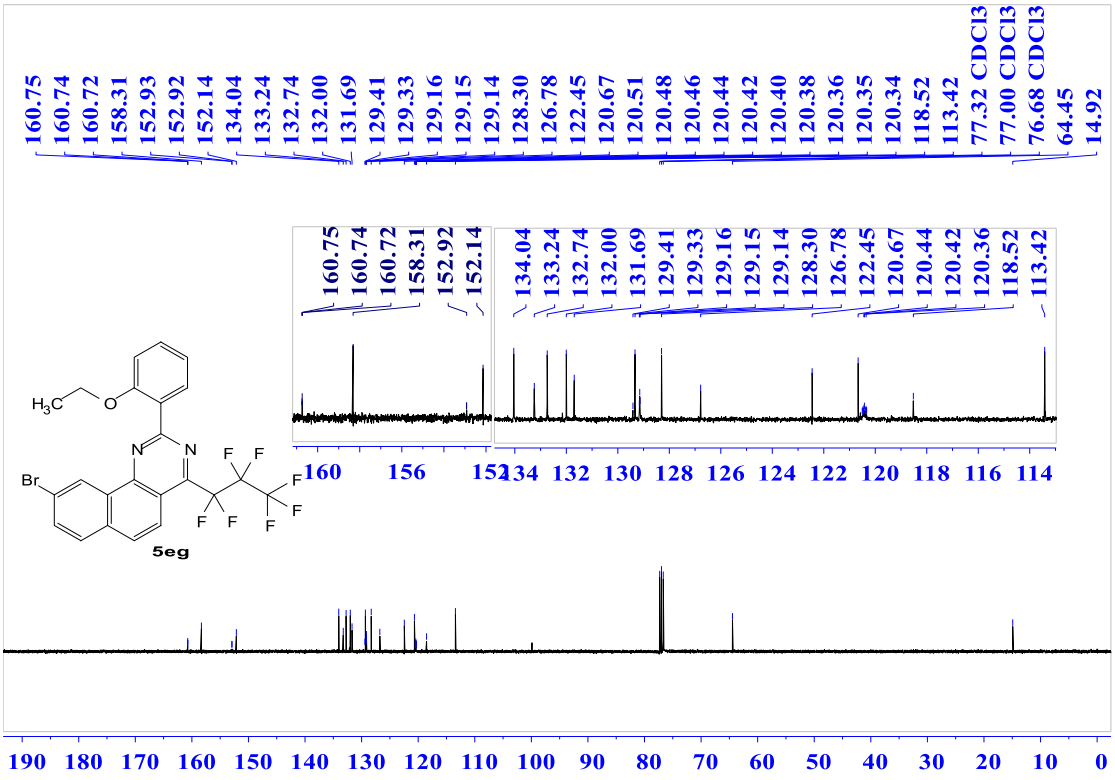

8.82
8.81
8.07
8.07
8.05
8.05
7.96
7.88
7.86
7.86
7.84
7.48
7.48
7.48
7.46
7.46
7.46
7.44
7.43
7.25 CDCl3
7.17
7.16
7.15
7.15
7.13
7.13
7.12
7.10
4.23
4.21
4.19
4.17
4.05
1.43
1.41
1.39
0.00
0.96
0.99
0.99
1.98
1.98
1.98
2.00
2.96
3.00
5dg


-79.60
-79.63
-79.65
-108.40
-108.42
-108.44
-108.45
-108.47
-108.48
-108.49
-108.50
-124.49
-124.51
-124.52
-124.54
-124.56
3.01
2.00
2.00
-79.60 -79.70
-108.6
-124.8
5dg
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270 -300


160.20
160.19
160.18
159.44
157.93
152.50
132.62
132.04
131.42
129.56
129.54
129.38
128.01
121.83
120.69
118.87
117.49
117.47
117.46
117.44
117.43
117.42
117.41
117.39
117.38
117.36
117.34
117.31
117.29
117.28
113.62
105.34
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.50
55.70
14.82
5dg
160 155 150 145 140 135 130 125 120 115 110 105
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


9.60 9.59 8.22 8.22 8.20 8.20 7.92 7.91 7.90 7.89 7.88 7.86 7.81 7.79 7.50 7.50 7.49 7.49 7.48 7.47 7.25 CDCl3 7.17 7.17 7.15 7.15 7.13 7.13 7.13 7.11 7.11 4.25 4.24 4.22 4.20 1.54 1.52 1.50 -0.00
5eg
0.87 0.96 1.02 1.07 0.97 0.97 1.00 2.13 2.00 3.02
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0


-79.58 -79.61 -79.63 -108.40 -108.41 -108.42 -108.43 -108.45 -108.46 -108.47 -124.56 -124.58 -124.60 -124.61 -124.63
5eg
3.07 2.00 2.05
-79.65 -108.45 -124.64
90 60 30 0 -30 -60 -90 -120 -150 -180 -210 -240 -270


160.75
160.74
160.72
158.31
152.93
152.92
152.14
134.04
133.24
132.74
132.00
131.69
129.41
129.33
129.16
129.15
129.14
128.30
126.78
122.45
120.67
120.51
120.48
120.46
120.44
120.42
120.40
120.38
120.36
120.35
120.34
118.52
113.42
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
64.45
14.92
H3C
O
N
N
F
F
F
F
F
F
Br
5eg
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0