Electronic Supplementary Material (ESI) for Chemical Communications.

Enantioselective formal [2+2+2] cycloaddition of 1,3,5-triazinanes to construct tetrahydropyrimidin-4-one derivatives

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Abstract: A chiral Lewis acid-catalyzed enantioselective formal [2+2+2] cycloaddition of 1,3,5-triazinanes with azlactones or β,γ-unsaturated pyrazole amides were developed to synthesize chiral tertiary/quaternary tetrahydropyrimidin-4-one derivatives with good yields and enantioselectivities. Two competitive reaction pathways were proposed based on experiments.

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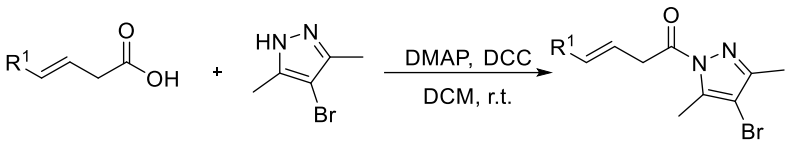
1. General Information

NMR characterization data were collected on bruker ASCENDTM operating at 400 MHz for ^{1}H NMR, 101 MHz for ^{13}C NMR (with complete proton decoupling), and 376 MHz for ^{19}F NMR (with complete proton decoupling). ^{1}H NMR and ^{13}C NMR: chemical shifts δ were recorded in ppm relative to tetramethylsilane and internally referenced to the residual solvent signal (for ^{1}H NMR: $CDCl_3$ = 7.26 ppm, DMSO-d_6 = 2.50 ppm; for ^{13}C NMR: $CDCl_3$ = 77.1 ppm, DMSO-d_6 = 39.6 ppm). Data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, td = triplet of doublets, dt = doublet of triplets, ddd = doublet of doublet of doublets, m = multiplet), coupling constants (Hz), integration. High performance liquid chromatography (HPLC) was performed on Alliance e2695 using Daicel Chiralcel IA, IE, IH, ADH, ODH at 23 °C with UV detector at 254 nm, supercritical fluid chromatography (SFC) was performed on Acquity UPC^2 using Daicel Chiralcel OX-3, IB-3 at 25 °C with UV detector at 254 nm, enantiomeric excesses (ee) and enantiomeric ratio (er) were determined in comparison with the authentic racemates. High resolution mass spectra (HRMS) were performed on Thermo Q-Exactive Focus (FTMS+c ESI) and data were reported as (m/z). Infrared spectra (IR) were recorded on Bruker Tensor II spectrometer with Plantium ATR accessory and the peaks are reported as absorption maxima (ν, cm^{-1}). Optical rotations were measured on Rudolph Research Analytic Automatic Polarimeter, and reported as follows: $[\alpha]^{T}_{D}$ (*c*: g/100 mL, solvent). Melting point ranges were determined on OptiMelt. X-ray crystallographic data were collected by a Bruker D8 Venture Photon II.
The experiments requiring chiral *N,N*'-dioxide ligands[1] and azlactone[2] were synthesized according to known procedures and purified by recrystallization prior to use. All of the starting materials including the metal salts were purchased from TCI, Aladdin, Adamas, Acros, Aldrich and other companies, and used without further purification. The 3/4/5Å MS was purchased from Acros, and oven-dried by the muffle furnace for 4 h prior to use. All the solvents including toluene, tetrahydrofuran, diethyl ether, dichloromethane, chloroform, 1,2-dichloroethane and so on were pre-dried over appropriate desiccants, and distilled prior to use. Reactions were monitored using thin-layer chromatography (TLC) on GF254 silica gel. Visualization of the developed plates was performed under UV light (254 nm) or using iodine, cobalt thiocyanate or $KMnO_4$. The products were purified by flash column chromatography with silicycle 300-400 mesh silica gel.

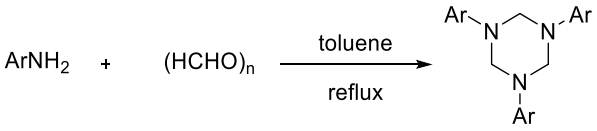
2. Substrates synthesis

2.1 General procedure for the synthesis of β,γ-unsaturated pyrazole amide compounds[3]



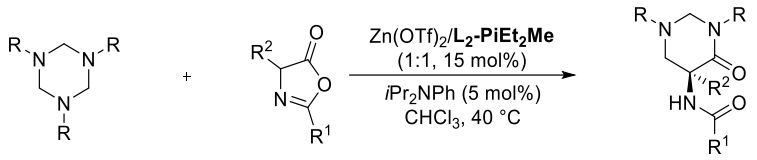
A solution of β,γ-unsaturated carboxylic acid (4.0 mmol) and the substituted pyrazole (4.2 mmol, 1.05 equiv) in CH_2Cl_2 (30 mL) at 0 °C was added DCC (1.2 equiv, 4.8 mmol in 10 mL of CH_2Cl_2) and 4-DMAP (4.0 mmol, 1.0 equiv) successively. The resulting mixture was stirred at 0 °C for 30 minutes, then gradually warmed to room temperature and further stirred at room temperature for 24 hours. Water (75 mL) was added to the crude product which was then extracted with CH_2Cl_2 (80 mL × 2). The combined organic phase was washed with water, brine, dried over Na_2SO_4, filtered, concentrated under reduced pressure and purified by column chromatography to afford the desired product.

2.2 General procedure for the synthesis of 1,3,5-triazinane compounds[4]



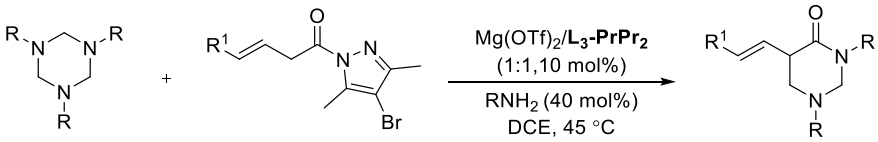
In a 100 mL round-bottomed flask equipped with a Dean-Stark apparatus, a mixture of aniline (30 mmol), paraformaldehyde (30 mmol), and toluene (50 mL) was heated with refluxing for 2 hours. Then the solvent was concentrated under reduced pressure at 50°C, a precipitate came out from the mixture. The precipitate was collected by filtration, washed with *n*-hexane several times, and dried to obtain 1,3,5-triazinane.

3. General procedure for the catalytic asymmetric cycloaddition



1 **2** **3**

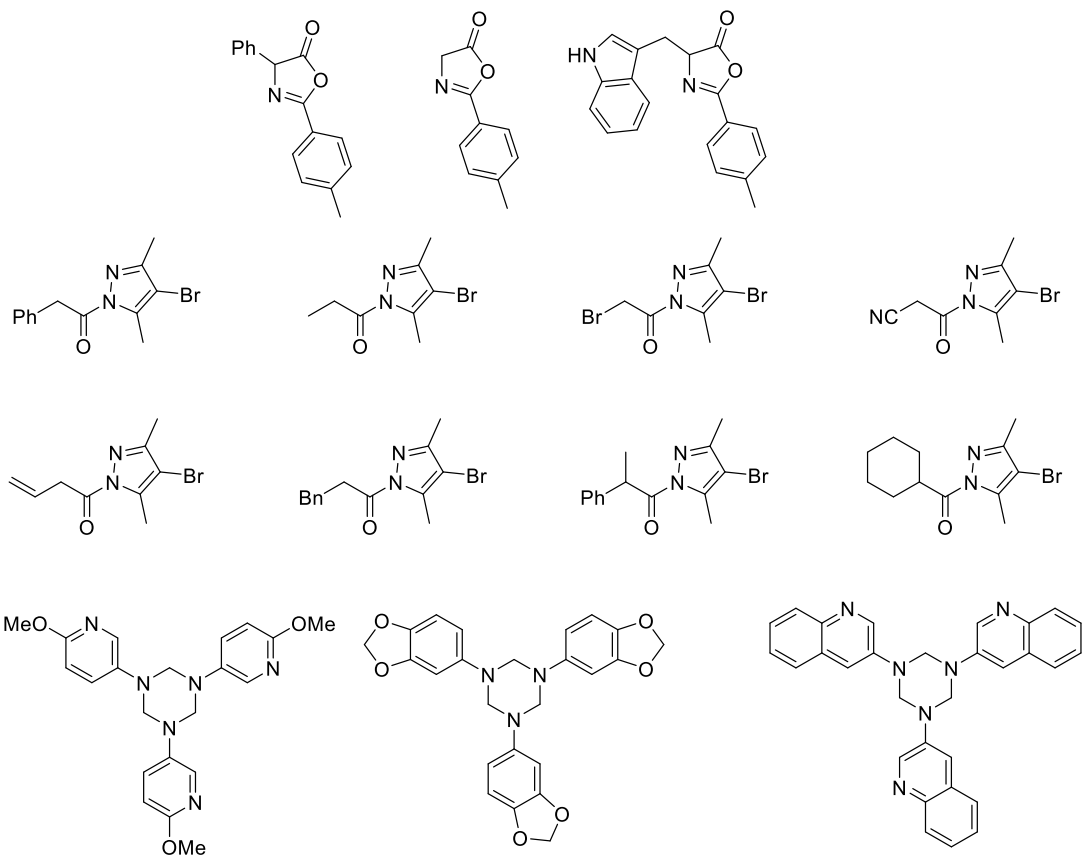
The reactions were carried out with Zn(OTf)$_2$/**L$_2$-PiEt$_2$Me** (1:1, 15 mol%) in THF (0.3 mL) under N_2 at 35 °C for 0.5 h. After removing the solvent in vacuo, 1,3,5-triazinane **1** (0.1 mmol), azlactone **2** (0.1 mmol), *i*Pr$_2$NPh (5 mol%) and solvent (1 mL) were added successively under N_2 and kept stirring at 40 °C for 38-72 h. The reaction mixture was subjected to column chromatography on silica gel and eluted with petroleum ether–ethyl acetate (v/v, 5:1) to afford the corresponding product **3**. The racemic product was prepared through the same process without catalyst or *i*Pr$_2$NPh.



1 **6** **7**

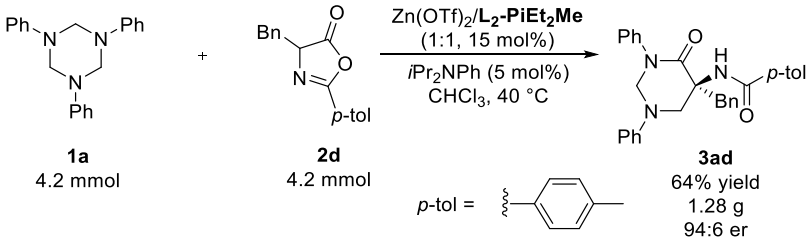
The reaction was conducted with Mg(OTf)$_2$ (0.01 mmol), **L$_3$-PrPr$_2$** (0.01 mmol) and pyrazole amide **6** (0.10 mmol) in 1.0 mL of DCE. The mixture was stirred at 35 °C under N_2 for 30 min. 1,3,5-Triazinane **1** (0.15 mmol) and RNH_2 were added, and the resulting mixture was stirred at 45 °C for 120 h. The reaction mixture was subjected to column chromatography on silica gel and eluted with petroleum ether–ethyl acetate (v/v, 4:1) to afford the corresponding product **7**. The racemic product was prepared using Mg(OTf)$_2$/(±)-**L$_3$-PrPr$_2$** complex as catalyst.

4. Scope limitation



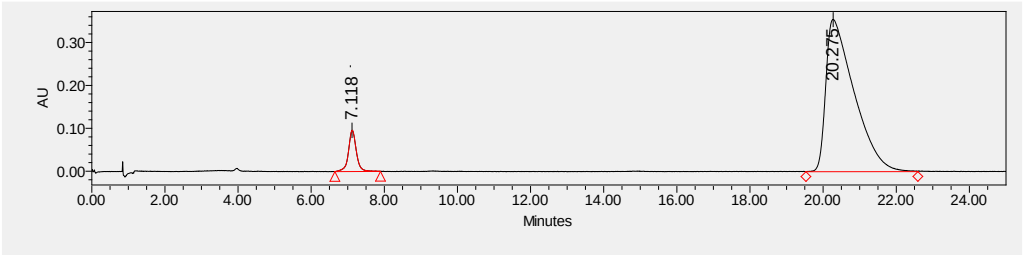
The substrates above had no reactivities in the catalytic system.

5. Experimental procedure for the scale-up reaction and further conversion

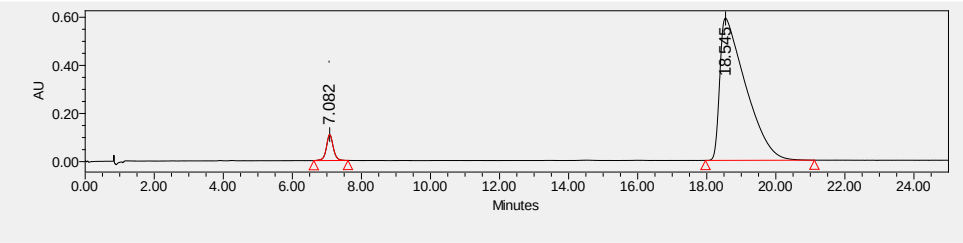


A dry 50 mL round-bottom flask was charged with $Zn(OTf)_2$ (15 mol%, 226.8 mg), **L_2-PiEt$_2$Me** (15 mol%, 382.2 mg). Then, THF (12.0 mL) was added and the mixture was stirred at 35 °C under N_2 for 10 h. After removing the solvent in vacuo, *N*-phenyl 1,3,5-triazinane **1a** (4.2 mmol, 1.32 g), azlactone **2d** (4.2 mmol, 1.05 g), *i*Pr_2NPh (5 mol%, 42 μL) and $CHCl_3$ (42 mL) were added successively under N_2 and kept stirring at 40°C for 60 h. The solvent was removed in vacuo and the residue was subjected to column chromatography on silica gel and eluted with petroleum ether–ethyl acetate (v/v, 5:1) to afford the corresponding product **3ad** (1.28 g, 64% yield, 94:6 er).

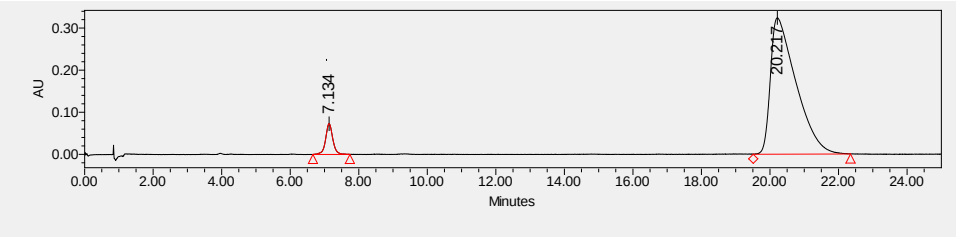
Self-disproportionation of enantiomers (SDE) was observed in the flash chromatography separation, but it did not attain the significant level. The products of the first four tubes, the next four tubes, and all were obtained in 93:7 er, 95:5 er, 94:6 er, separately.



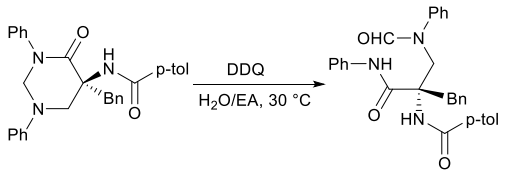
	Retention Time	Area	% Area
1	7.118	1439694	7.03
2	20.275	19043897	92.97



	Retention Time	Area	% Area
1	7.082	1495942	4.64
2	18.545	30759435	95.36



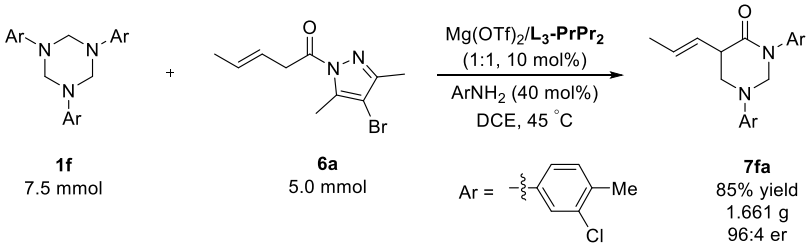
	Retention Time	Area	% Area
1	7.134	1068334	5.99
2	20.217	16781518	94.01



3ad
94:6 er

8ad
> 99% yield
94:6 er

3ad (0.1 mmol, 96:6 er) was dissolved in ethyl acetate (1 mL), and then H_2O (0.1 mmol, 1.8 μL), DDQ (0.2 mmol, 45.4 mg) were added successively. After stirring at 30 °C for 3 h, the reaction mixture was subjected to column chromatography on silica gel and eluted with petroleum ether–ethyl acetate (v/v, 3:1) to afford the corresponding product **8ad** with maintained optically activity.



1f
7.5 mmol

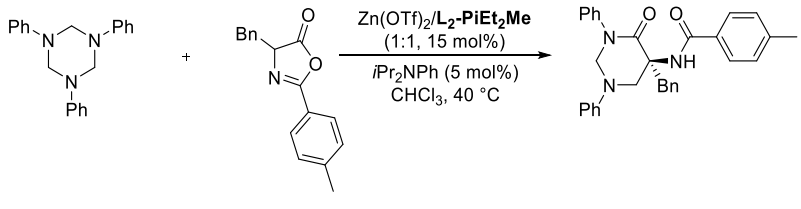
6a
5.0 mmol

7fa
85% yield
1.661 g
96:4 er

A dry reaction 50 mL round-bottom flask was charged with $Mg(OTf)_2$ (10 mol%, 161.0 mg), **L_3-PrPr$_2$** (10 mol%, 310.5 mg) and the substrate **6a** (5.0 mmol, 1.285 g). Then, DCE (50.0 mL) was added and the mixture was stirred at 35 °C under N_2 for 5 h. Subsequently, **1f** (1.5 equiv., 3.700 g) and $ArNH_2$ was added and the reaction mixture was stirred at 45 °C for 120 h. The solvent was removed in vacuo and the residue was subjected to column chromatography on silica gel and eluted with petroleum ether–ethyl acetate (v/v, 6:1) to afford the corresponding product **7fa** (1.701 g, 85% yield, 96:4 er).

6. The nonlinear effect study

Table S1 The relationship between ee of the ligand and ee of the product **3ad**



1a

2d

3ad

entry[a]	ee of **L$_2$-PiEt$_2$Me** (%)	yield (%)[b]	ee of **3ad** (%)[c]
1	0	79	0
2	20	67	18
3	40	75	30
4	60	73	46
5	80	74	65
6	100	63	90

[a] All reactions were carried out with Zn(OTf)$_2$/**L$_2$-PiEt$_2$Me** (1:1, 15 mol%), **1a** (0.1 mmol), **2d** (0.1 mmol), *i*Pr$_2$NPh (5 mol%) in DCE (1.0 mL) at 40 °C for 38 h. [b] Yield of isolated product. [c] Determined by SFC analysis on a chiral stationary phase.

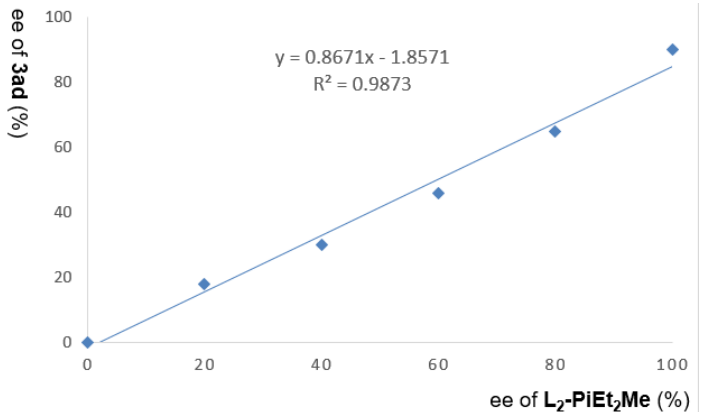
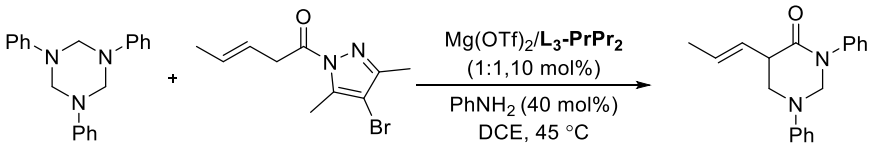


Figure S1 Linear relationship between ee of the ligand and ee of the product **3ad**.

Table S2. The relationship between ee of the ligand and ee of the product **7aa**



1a **6a** **7aa**

entry[a]	ee of **L$_3$-PrPr$_2$** (%)	yield (%)[b]	ee of **7aa** (%)[c]
1	0	89	0
2	20	89	21
3	40	89	42
4	60	89	55
5	80	90	76
6	100	90	92

[a] All reactions were carried out with Mg(OTf)$_2$/**L$_3$-PrPr$_2$** (1:1, 10 mol%), **6a** (0.10 mmol), **1a** (0.15 mmol), PhNH$_2$ (20 mol%) in DCE (1.0 mL) at 45 °C for 120 h. [b] Yield of isolated product. [c] Determined by HPLC analysis on a chiral stationary phase.

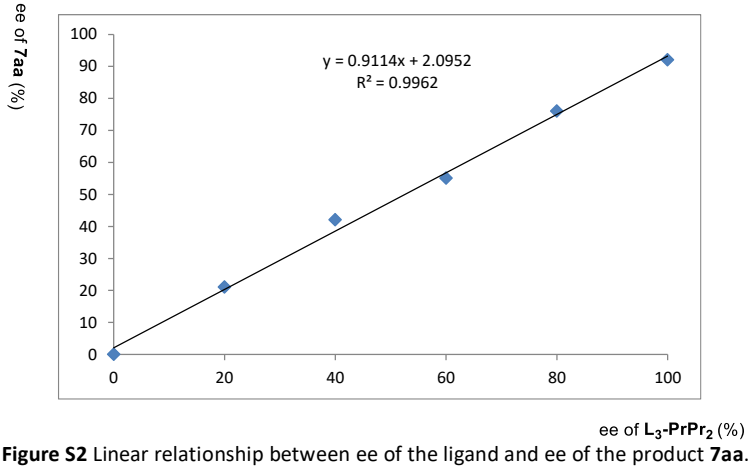
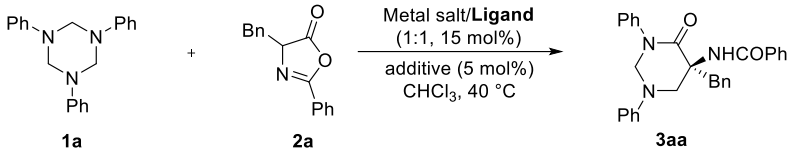
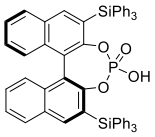
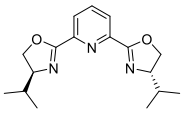
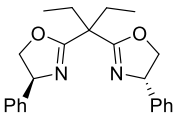


Figure S2 Linear relationship between ee of the ligand and ee of the product **7aa**.

7. Control experiments and other catalysts screening

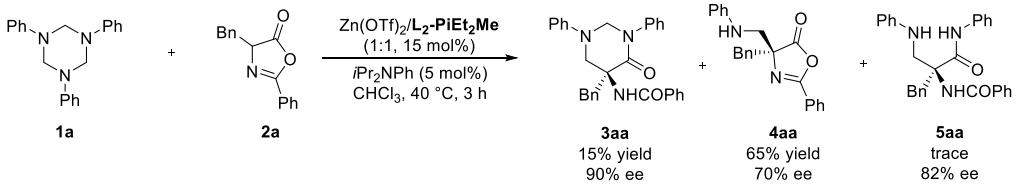
Table S3 Control experiments and other catalysts screening

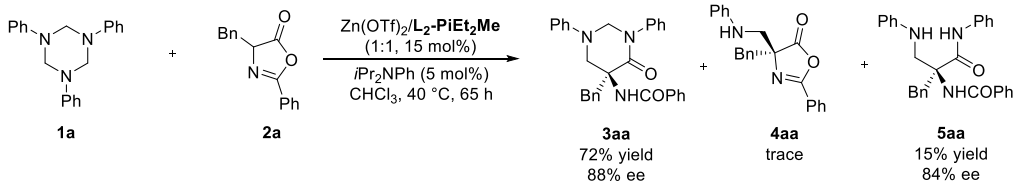


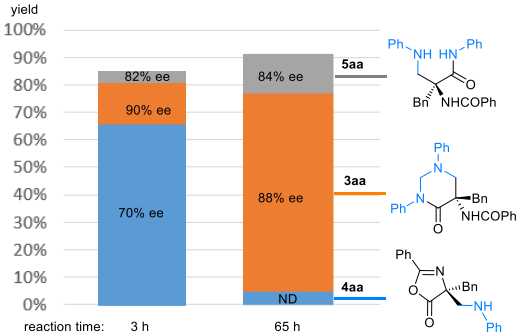
entry[a]	ligand	metal salt	additive	yield (%)[b]	er[c]
1	--	$Zn(OTf)_2$	iPr_2NPh	18	--
2	**L_2-PiEt$_2$Me**	--	iPr_2NPh	38	51:49
3	--	--	iPr_2NPh	50	--
4	**L_2-PiEt$_2$Me**	$Zn(OTf)_2$	--	62	95:5
5	--	--	--	73	--
6	**L_2-PiEt$_2$Me**	$Zn(OTf)_2$	iPr_2NPh	71	95:5
7		--	iPr_2NPh	72	50:50
8		$Zn(OTf)_2$	iPr_2NPh	66	50:50
9		$Zn(OTf)_2$	iPr_2NPh	57	54.5:45.5

[a] All reactions were carried out with **ligand** (0 or 15 mol%), metal salts (0 or 15 mol%), **1a** (0.1 mmol), **2a** (0.1 mmol), iPr_2NPh (0 or 5 mol%) in $CHCl_3$ (1.0 mL) at 40 °C for 38 h. [b] Isolated yield. [c] Determined by chiral SFC analysis.

Scheme S1 Control experiments about byproducts

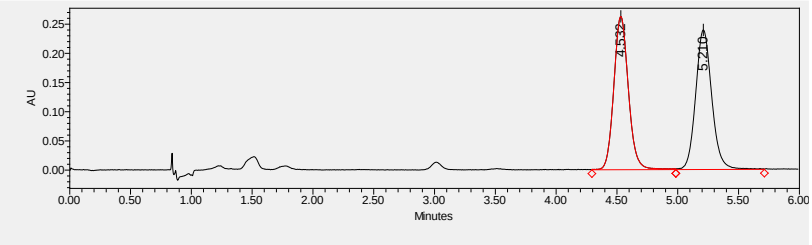




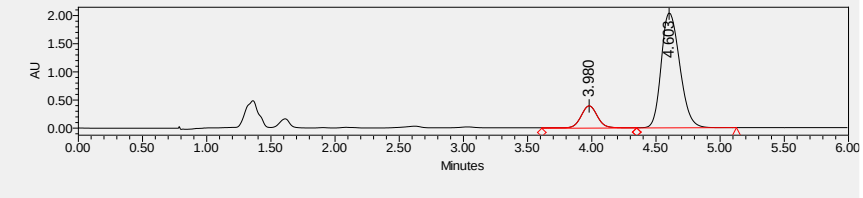


Under standard conditions, the reaction between **1a** and **2a** was ananlyzed at different reaction time.
It is testified by TLC analysis that the byproduct **4aa** is an intermediate to partly form **3aa**, leading to slightly lower yield of **3aa**. The **5aa** is formal hydrolysate of **3aa** via losing a formaldehyde. Moreover, the formal Mannich product **4aa** has distinctly lower ee than the correspongding **3aa** (70% vs. 90% ee at 3 h).

SFC analysis for **4aa**: dissolved **4aa** in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 4.53 min, t_{major} = 5.21 min. Reaction time = 3 h, ee= 70%.

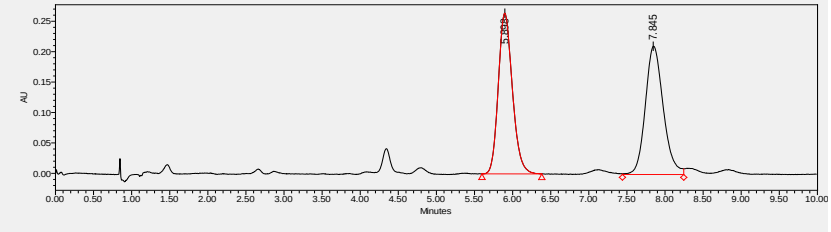


	Retention Time	Area	% Area
1	4.532	2191426	49.90
2	5.210	2200251	50.10

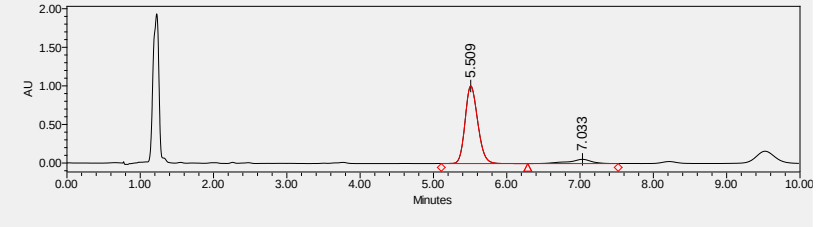


	Retention Time	Area	% Area
1	3.980	3644036	15.07
2	4.603	20543068	84.93

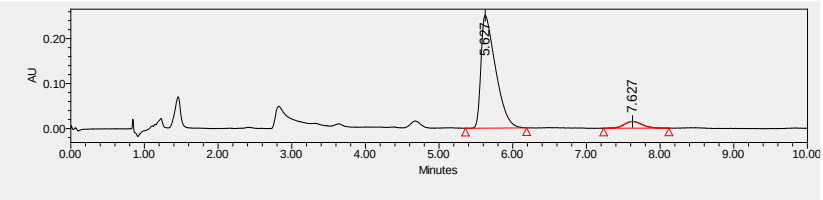
SFC analysis for **5aa**: dissolved **5aa** in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 5.90 min, t_{major} = 7.85 min. Reaction time = 3 h, ee= 82%; reaction time = 65 h ee= 84%.



	Retention Time	Area	% Area
1	5.898	3391136	49.56
2	7.845	3450978	50.44

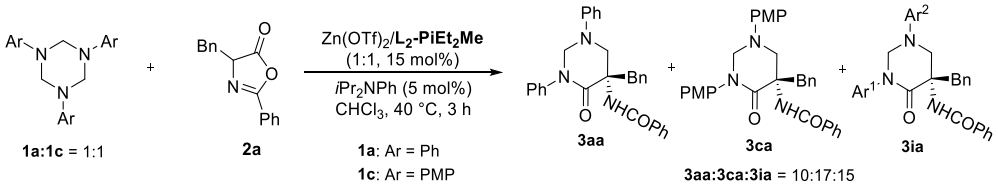


	Retention Time	Area	% Area
1	5.509	12020200	91.06
2	7.033	1180250	8.94



	Retention Time	Area	% Area
1	5.627	3464354	93.20
2	7.627	252639	6.80

Scheme S2 cross-over experiment

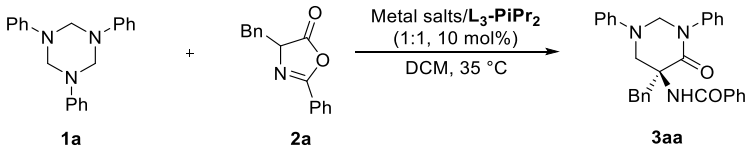


A cross-over experiment was performed by subjecting *N*-Ph triazinane **1a** and *N*-PMP triazinane **1c** with azlactone **2a** to the standard reaction conditions. Not only were the corresponding products **3aa** and **3ca** detected, but also so was the *N*-substitution cross-over product **3ia**, with these three products observed in a 10 : 17 : 15 ratio after 65 hours.

8. Optimazition reaction condition

Optimazition for the reaction with azlactone

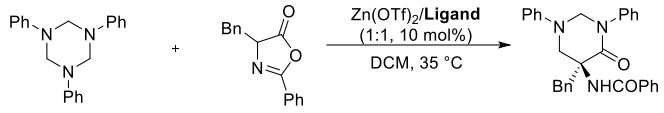
Table S4 Optimization of metal salts



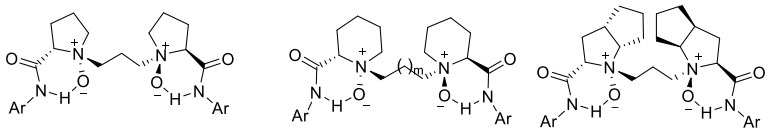
entry[a]	metal saltas	yield (%)[b]	er (%)[c]
1	$Sc(OTf)_3$	74	70:30
2	$Y(OTf)_3$	65	71:29
3	$Fe(OTf)_3$	31	57:43
4	$In(OTf)_3$	67	64:36
5	$Yb(OTf)_3$	69	72:28
6	$Ni(OTf)_2$	72	53:47
7	$Mg(OTf)_2$	72	54:46
8	$Zn(OTf)_2$	84	71:29

[a] Unless otherwise noted, all reactions were carried out with metal salts/**L_3-$PiPr_2$** (1:1, 10 mol%), **1a** (0.1 mmol), **2a** (0.1 mmol), in DCM (1.0 mL) at 35 °C for 24 h. [b] Isolated yield. [c] Determined by chiral SFC analysis.

Table S5 Optimization of the ligands



1a **2a** **3aa**



L3-PrPr2: Ar = 2,6-*i*Pr2C6H3 **L3-PiPr2**: Ar = 2,6-*i*Pr2C6H3, m = 1 **L3-RaPr2**: Ar = 2,6-*i*Pr2C6H3

L4-PiPr2: Ar = 2,6-*i*Pr2C6H3, m = 2

L2-PiPr2: Ar = 2,6-*i*Pr2C6H3, m = 0

L2-PiMe3: Ar = 2,4,6-Me3C6H2, m = 0

L2-PiPr3: Ar = 2,4,6-*i*Pr3C6H2, m = 0

L2-PiMe2: Ar = 2,6-Me2C6H3, m = 0

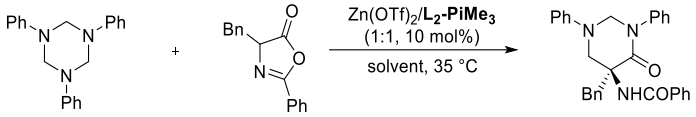
L2-PiEt2: Ar = 2,6-Et2C6H3, m = 0

L2-PiEt2Me: Ar = 2,6-Et2-4-MeC6H2, m = 0

entry[a]	**ligand**	yield (%)[b]	er (%)[c]
1	**L3-PrPr2**	78	58:42
2	**L3-RaPr2**	85	51:49
3	**L3-PiPr2**	84	71:29
4	**L4-PiPr2**	53	60:40
5	**L2-PiPr2**	54	83:17
6	**L2-PiEt2**	69	88:12
7	**L2-PiPr3**	57	79:21
8	**L2-PiMe3**	64	89:11

[a] Unless otherwise noted, all reactions were carried out with Zn(OTf)2/**ligand** (1:1, 10 mol%), **1a** (0.1 mmol), **2a** (0.1 mmol), in DCM (1.0 mL) at 35 °C for 24 h. [b] Isolated yield. [c] Determined by chiral SFC analysis.

Table S6 Optimization of solvents

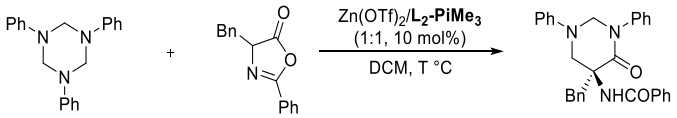


1a **2a** **3aa**

entry[a]	solvent	yield (%)[b]	er (%)[c]
1	DCM	64	89:11
2[d]	THF	43	59:41
3	PhMe	69	59:41
4	Et_2O	68	69.5:30.5
5[d]	$CHCl_3$	65	80.5:19.5
6[d]	DCE	60	88:12

[a] Unless otherwise noted, all reactions were carried out with Zn(OTf)2/**L2-PiMe3** (1:1, 10 mol%), **1a** (0.1 mmol), **2a** (0.1 mmol), in solvent (1.0 mL) at 35 °C for 24 h. [b] Isolated yield. [c] Determined by chiral SFC analysis. [d] The reaction time was 34-38 h.

Table S7 Optimization of temperature

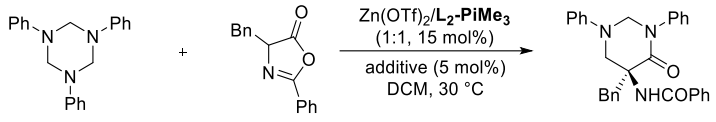


1a **2a** **3aa**

entry[a]	T (°C)	yield (%)[b]	er (%)[c]
1[d]	35	64	88.5:11.5
2	30	59	91.5:8.5
3	20	52	91:9
4	10	51	88.5:11.5
5[e]	30	57	92.5:7.5

[a] Unless otherwise noted, all reactions were carried out with Zn(OTf)2/**L2-PiMe3** (1:1, 10 mol%), **1a** (0.1 mmol), **2a** (0.1 mmol), in DCM (1.0 mL) at T °C for 38 h. [b] Isolated yield. [c] Determined by chiral SFC analysis. [d] The reaction time was 24 h. [e] 15 mol% catalyst loading.

Table S8: Optimization of additive

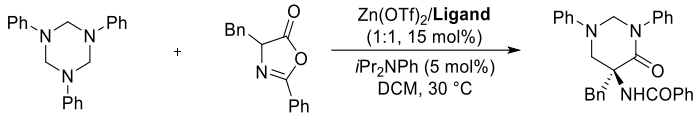


1a **2a** **3aa**

entry[a]	additive	yield (%)[b]	er (%)[c]
1	Et_3N	58	94.5:5.5
2	Ph_3N	60	91:9
3	*i*Bu_3N	63	93.5:6.5
4	*i*Pr_2EtN	57	93:7
5	*i*Pr_2NH	57	93:7
6	*i*Pr_2NPh	67	94.5:5.5
7[d]	Et_3N	62	93.5:6.5
8[d]	K_2CO_3	34	93.5:6.5
9[d]	*t*BuOK	54	91.5:8.5
10[e]	*i*Pr_2NPh	48	94:6
11[f]	*i*Pr_2NPh	60	94:6

[a] Unless otherwise noted, all reactions were carried out with $Zn(OTf)_2$/**L_2-PiMe$_3$** (1:1, 15 mol%), **1a** (0.1 mmol), **2a** (0.1 mmol) and additive (5 mol%) in DCM (1.0 mL) at 30 °C for 38 h. [b] Isolated yield. [c] Determined by chiral SFC analysis. [d] DCE was used instead of DCM. [e] 10 mol% additive loading. [f] 2.5 mol% additive loading.

Table S9 Reoptimization of ligands

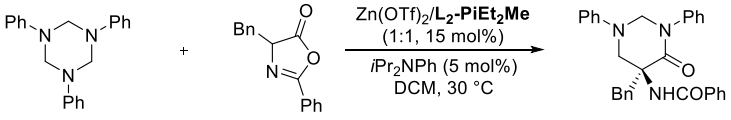


1a **2a** **3aa**

entry[a]	**Ligand**	yield (%)[b]	er (%)[c]
1	**L_2-PiMe$_3$**	67	94.5:5.5
2	**L_2-PiMe$_2$**	50	94:6
3	**L_2-PiEt$_2$**	60	94:6
4[d]	**L_2-PiEt$_2$Me**	66	94:6
5[d][e]	**L_2-PiEt$_2$Me**	63	94:6
6[d][f]	**L_2-PiEt$_2$Me**	57	94:6

[a] Unless otherwise noted, all reactions were carried out with $Zn(OTf)_2$/**Ligand** (1:1, 15 mol%), **1a** (0.1 mmol), **2a** (0.1 mmol) and *i*Pr_2NPh (5 mol%) in DCM (1.0 mL) at 30 °C for 38 h. [b] Isolated yield. [c] Determined by chiral SFC analysis. [d] The ligand involved can be purified more efficiently by recrystallization. [e] 12.5 mol% catalyst loading. [f] 10 mol% catalyst loading.

Table S10 Optimization of substrates ratio and concentration

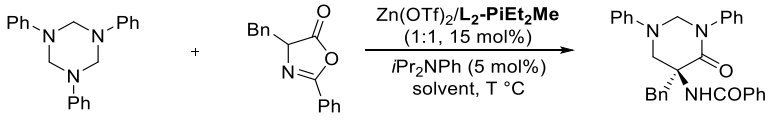


1a **2a** **3aa**

entry[a]	**1a** (X mmol)	**2a** (Y mmol)	yield (%)[b]	er (%)[c]
1	0.16	0.10	66	93.5:6.5
2	0.13	0.10	71	93.5:6.5
3	0.10	0.10	66	94:6
4	0.07	0.10	65	94:6
5	0.07	0.15	40	95:5

[a] Unless otherwise noted, all reactions were carried out with $Zn(OTf)_2$/**L_2-PiEt$_2$Me** (1:1, 15 mol%), **1a** (X mmol), **2a** (Y mmol) and *i*Pr_2NPh (5 mol%) in DCM (1.0 mL) at 30 °C for 38 h. [b] Isolated yield. [c] Determined by chiral SFC analysis.

Table S11 Optimization of solvent and temperature



1a **2a** **3aa**

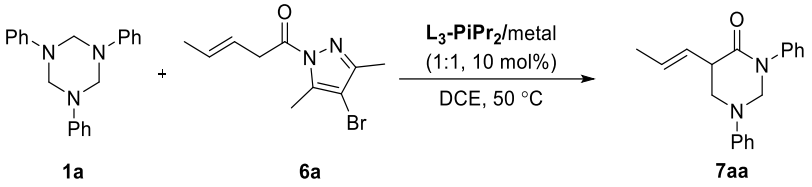
entry[a]	solvent	T (°C)	yield (%)[b]	er (%)[c]

1	DCM	30	66	94:6
2	DCE	30	64	93.5:6.5
3	$CHCl_3$	30	74	94.5:5.5
4	$CHCl_3$	40	71	95:5
5	$CHCl_3$	20	60	93:7

[a] Unless otherwise noted, all reactions were carried out with $Zn(OTf)_2$/**L_2-PiEt$_2$Me** (1:1, 15 mol%), **1a** (0.1 mmol), **2a** (0.1 mmol) and *i*Pr_2NPh (5 mol%) in solvent (1.0 mL) at T °C for 38 h. [b] Isolated yield. [c] Determined by chiral SFC analysis.

Optimazition for the reaction with β,γ-unsaturated pyrazole amide

Table S12 Metal salts effect

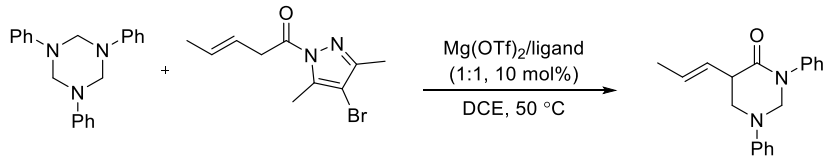


1a **6a** **7aa**

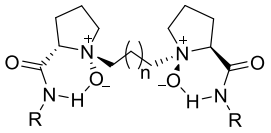
entry[a]	metal salt	yield (%)[b]	er (%)[c]
1	$Mg(OTf)_2$	65	86.5:13.5
2	$Ni(OTf)_2$	74	63.5:36.5
3	$Zn(OTf)_2$	35	69:31
4	$La(OTf)_3$	34	53.5:46.5
5	$Sc(OTf)_3$	36	60:40
6	$Y(OTf)_3$	39	75:25
7	$Yb(OTf)_3$	34	73.5:26.5
8	$In(OTf)_3$	33	52.5:47.5

[a] All reactions were carried out with metal salt/**L_3-PrPr$_2$** (1:1, 10 mol%), **6a** (0.10 mmol), **1a** (0.15 mmol) in DCE (1.0 mL) at 50 °C for 72 h. [b] Yield of isolated product. [c] Determined by HPLC analysis on a chiral stationary phase.

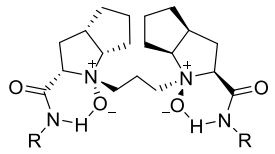
Table S13 Ligand effect



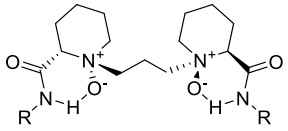
1a **6a** **7aa**



L_3-PrPr$_2$: R = 2,6-*i*$Pr_2C_6H_3$, n = 1
L_3-PrMe$_2$: R = 2,6-$Me_2C_6H_3$, n = 1
L_3-PrEt$_2$: R = 2,6-$Et_2C_6H_3$, n = 1
L_3-PrPh: R = C_6H_5, n = 1
L_3-PrBn: R = Bn, n = 1
L_3-Pr(OMe)$_2$: R = 2,6-$(OMe)_2C_6H_3$, n = 1
L_3-PrPr$_2$Ad: R = 2,6-*i*Pr_2-4-(1-Ad)C_6H_2, n = 1
L_2-PrPr$_2$: R = 2,6-*i*$Pr_2C_6H_3$, n = 0
L_4-PrPr$_2$: R = 2,6-*i*$Pr_2C_6H_3$, n = 2
L_5-PrPr$_2$: R = 2,6-*i*$Pr_2C_6H_3$, n = 3



L_3-RaPr$_2$: R = 2,6-*i*$Pr_2C_6H_3$
L_3-RaPr$_2$Ad: R = 2,6-*i*Pr_2-4-(1-Ad)C_6H_2



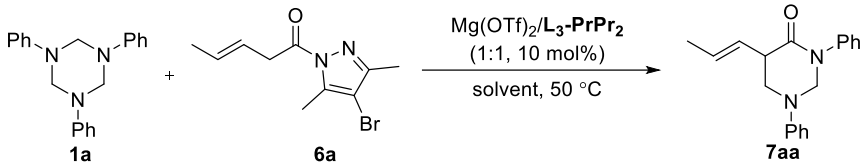
L_3-PiPr$_2$: R = 2,6-*i*$Pr_2C_6H_3$

entry[a]	ligand	yield (%)[b]	er (%)[c]
1	**L_3-PrPr$_2$**	73	93:7
2	**L_3-PiPr$_2$**	65	86.5:13.5
3	**L_3-RaPr$_2$**	64	90:10
4	**L_3-PrPh**	39	50:50
5	**L_3-PrMe$_2$**	50	88:12
6	**L_3-PrEt$_2$**	60	89.5:10.5
7	**L_2-PrPr$_2$**	26	80:20
8	**L_4-PrPr$_2$**	63	94:6
9	**L_5-PrPr$_2$**	66	92.5:7.5
10	**L_3-PrBn**	35	77.5:22.5

11	**L_3-Pr(OMe)$_2$**	1	68.5:31.5

[a] All reactions were carried out with Mg(OTf)$_2$/**ligand** (1:1, 10 mol%), **6a** (0.10 mmol), **1a** (0.15 mmol) in DCE (1.0 mL) at 50 °C for 72 h. [b] Yield of isolated product. [c] Determined by HPLC analysis on a chiral stationary phase.

Table S14 Solvent effect

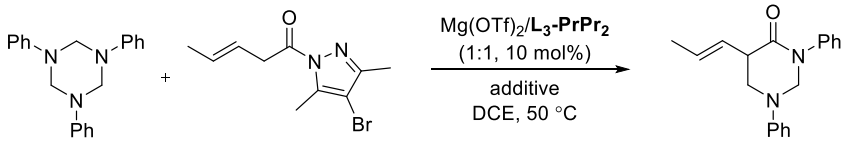


1a **6a** **7aa**

entry[a]	solvent	yield (%)[b]	er (%)[c]
1	DCE	73	93:7
2	EtOAc	N.R.	--
3	CH_3CN	62	82.5:17.5
4	Acetone	41	65:35
5	THF	N.R.	--
6	$ClCH_2CH_2Cl$	73	93:7
7	Toluene	N.R.	--
8	$CHCl_3$	71	92.5:7.5
9	$Cl_2CHCHCl_2$	59	92:8
10	Cl_2CHCH_2Cl	79	92:8

[a] All reactions were carried out with Mg(OTf)$_2$/**L_3-PrPr$_2$** (1:1, 10 mol%), **6a** (0.10 mmol), **1a** (0.15 mmol) in solvent (1.0 mL) at 50 °C for 72 h. [b] Yield of isolated product. [c] Determined by HPLC analysis on a chiral stationary phase.

Table S15 Additive effect

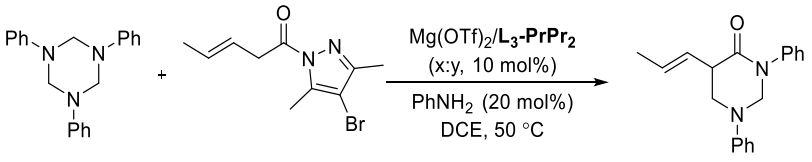


1a **6a** **7aa**

entry[a]	additive	yield (%)[b]	er (%)[c]
1	3Å MS (20 mg)	65	93.5:6.5
2	4Å MS (20 mg)	56	82:18
3	5Å MS (20 mg)	87	89:11
4	NH_4Cl (20 mol%)	68	90:10
5	PhCOOH (20 mol%)	63	50:50
6	Et_3N (20 mol%)	N.R.	--
7	K_2CO_3 (20 mol%)	N.R.	--
8	H_2O (20 mol%)	75	86.5:13.5
9	$PhNH_2$ (20 mol%)	88	93.5:6.5
10	--	73	93:7

[a] All reactions were carried out with Mg(OTf)$_2$/**L_3-PrPr$_2$** (1:1, 10 mol%), **6a** (0.10 mmol), **1a** (0.15 mmol), additive in DCE (1.0 mL) at 50 °C for 72 h. [b] Yield of isolated product. [c] Determined by HPLC analysis on a chiral stationary phase.

Table S16 Metal salt/ligand ratio effect

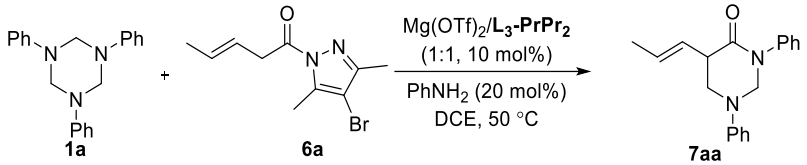


1a **6a** **7aa**

entry[a]	Mg(OTf)$_2$:**L_3-PrPr$_2$**	yield (%)[b]	er (%)[c]
1	2:1	69	90.5:9.5
2	1.1:1	73	93.5:6.5
3	1:1	88	93.5:6.5
4	1:1.1	81	93:7
5	1:1.2	80	93:7
6	1:1.5	69	93:7
7	1:2	55	93.5:6.5

[a] All reactions were carried out with Mg(OTf)$_2$/**L_3-PrPr$_2$** (g:h, 10 mol%), **6a** (0.10 mmol), **1a** (0.15 mmol), $PhNH_2$ (20 mol%) in DCE (1.0 mL) at 50 °C for 72 h. [b] Yield of isolated product. [c] Determined by HPLC analysis on a chiral stationary phase.

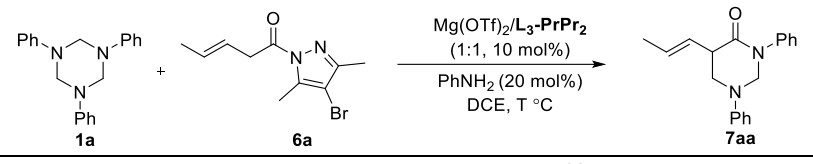
Table S17 Solvent dosage effect



entry[a]	DCE (mL)	yield (%)[b]	er (%)[c]
1	0.2	65	79:21
2	0.5	70	86.5:13.5
3	1.0	89	93.5:6.5
4	1.2	86	93:7
5	1.5	80	93:7
6	2.0	71	92.5:7.5

[a] All reactions were carried out with $Mg(OTf)_2$/**L_3-PrPr$_2$** (1:1, 10 mol%), **6a** (0.10 mmol), **1a** (0.15 mmol), $PhNH_2$ (20 mol%) in DCE at 50 °C for 72 h. [b] Yield of isolated product. [c] Determined by HPLC analysis on a chiral stationary phase.

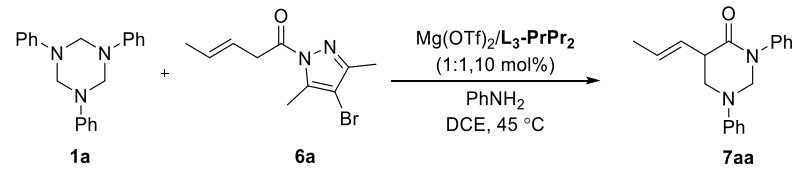
Table S18 Temperature effect



entry[a]	T (°C)	yield (%)[b]	er (%)[c]
1[d]	30	33	99:1
2[d]	35	45	99:1
3[d]	40	56	97.5:2.5
4[d]	45	66	96:4
5	50	88	93.5:6.5
6	55	90	86.5:13.5
7	60	85	75.5:24.5
8	70	74	53.5:46.5
9	80	messy	--

[a] All reactions were carried out with $Mg(OTf)_2$/**L_3-PrPr$_2$** (1:1, 10 mol%), **6a** (0.10 mmol), **1a** (0.15 mmol), $PhNH_2$ (20 mol%) in DCE (1.0 mL) at T °C for 120 h. [b] Yield of isolated product. [c] Determined by HPLC analysis on a chiral stationary phase. [d] The reaction time was longed to 120 h.

Table S19 The effect of the amount of $PhNH_2$

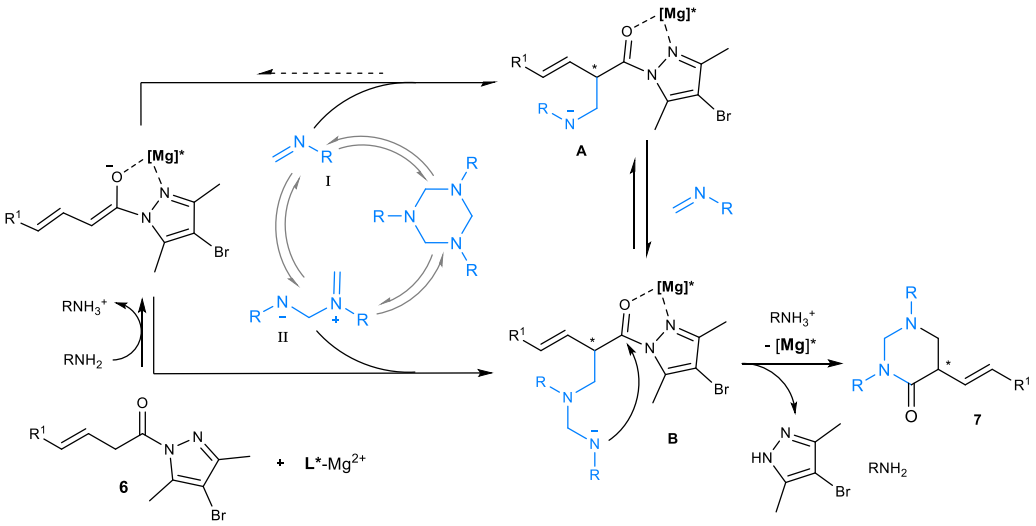


entry[a]	$PhNH_2$ (mol%)	yield (%)[b]	er (%)[c]
1	0	33	96:4
2	10	48	96:4
3	20	66	96:4
4	30	70	95.5:4.5
5	40	88	96:4
6	50	88	95:5
7	70	90	93:7
8	100	93	93.5:6.5

[a] All reactions were carried out with $Mg(OTf)_2$/**L_3-PrPr$_2$** (1:1, 10 mol%), **6a** (0.10 mmol), **1a** (0.15 mmol), $PhNH_2$ in DCE (1.0 mL) at 45 °C for 120 h. [b] Yield of isolated product. [c] Determined by HPLC analysis on a chiral stationary phase.

9. Possible reaction pathways for β,γ-unsaturated pyrazole amide

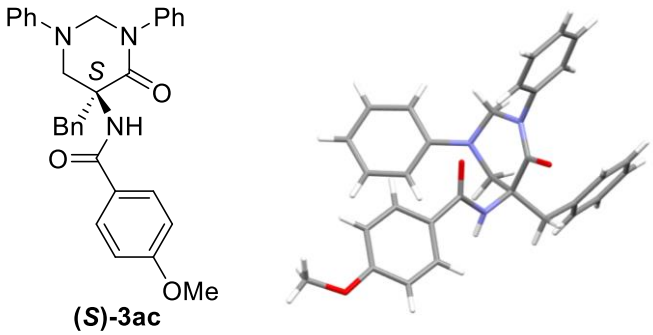
Based on experiments, reaction mechanism sililar to azlatone was proposed (Scheme S3). Reagents **I** and **II** competitively react with pyrazole aminde **6**, then intermediate **A** and **B** were respectively obtained. **A** and **B** could mutually transform via Mannich/retro-Mannich reaction.Losing a pyrazole as leaving group, substituted hydropyrimidin-4(1H)-one **7** was obtained.



Scheme S3 Possible reaction pathways for β,γ-unsaturated pyrazole amide

10. Determination of absolute configurations of the compounds

Crystals suitable for the X-ray crystal structure analysis were obtained from a solution of compound **3ac** in DCM (ca. 1 mL) and petroleum ether (1 mL) at r.t.The colourless crystal in flake-shape, with approximate dimensions of 0.046 × 0.083 × 0.451 mm^3, was selected and mounted for the single-crystal X-ray diffraction. The data set was collected by Bruker D8 Venture Photon II diffractometer at 172(2) K equipped with micro-focus Cu radiation source (K_α = 1.54178 Å). Applied with face-indexed numerical absorption correction, the structure solution was solved and refinement was processed by SHELXTL (version 6.14) and OLEX 2.3 program package[5,6,7,8]. The structure was analyzed by ADDSYM routine implemented in PLATON suite and no higher symmetry was suggested[9]. The crystal data and further details are listed in Table S20. CCDC 2096416 which contains the crystallographic data for the structure have been deposited with the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK, which can be obtained free of charge via Fax: +44 (0)1223 336033; E-Mail: deposit@ccdc.cam.ac.uk, https://www.ccdc.cam.ac.uk/structures/.



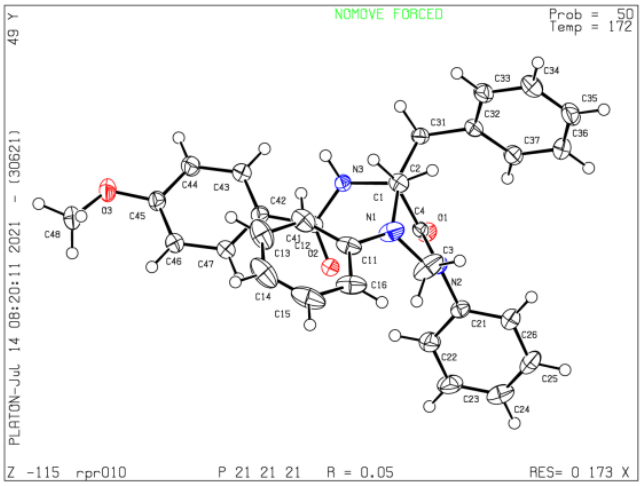


Figure S3 the thermal ellipsoid figure of 3ad with 50% probabilities

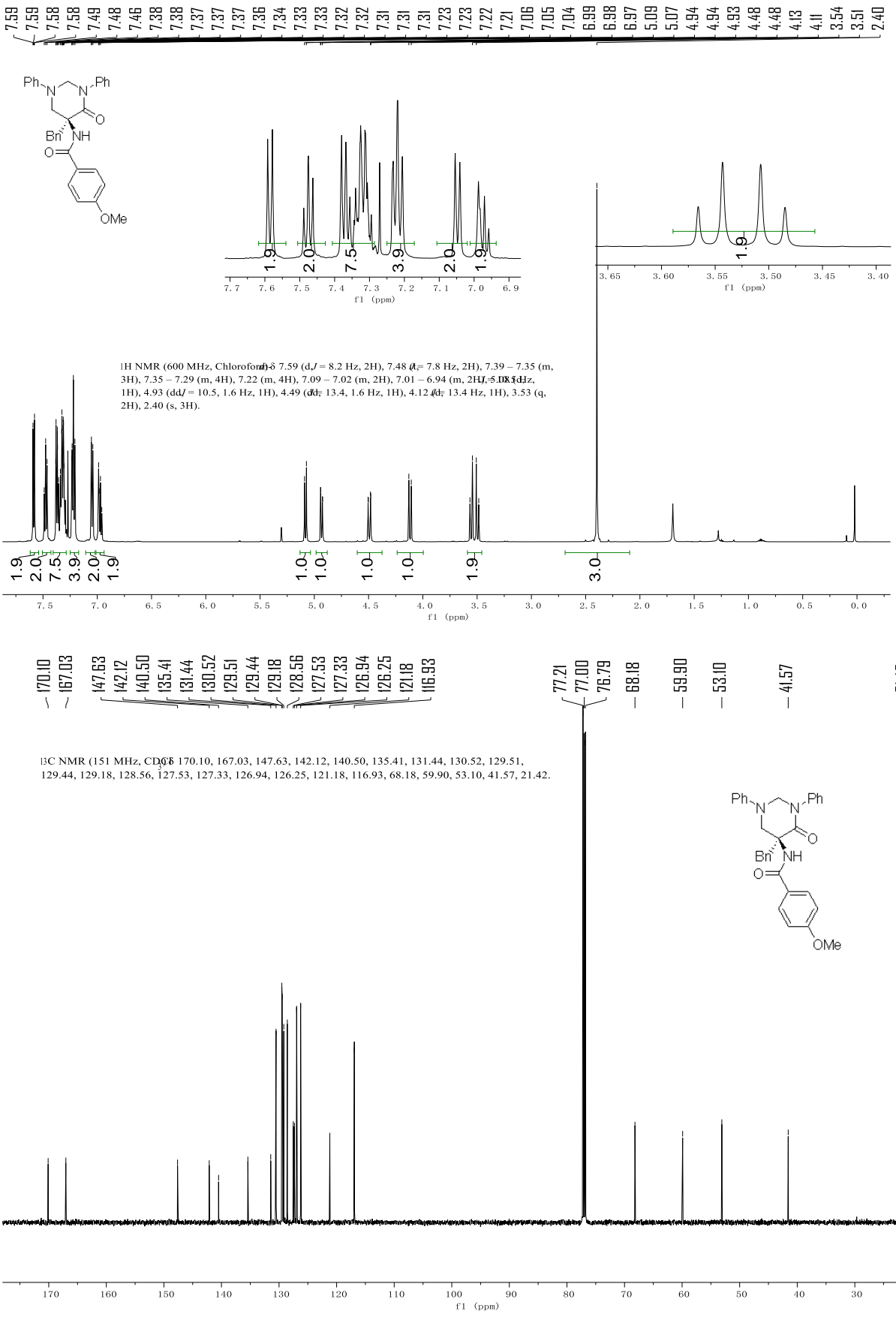
Table S20 Crystallographic Data for $C_{31}H_{29}N_3O_3$.

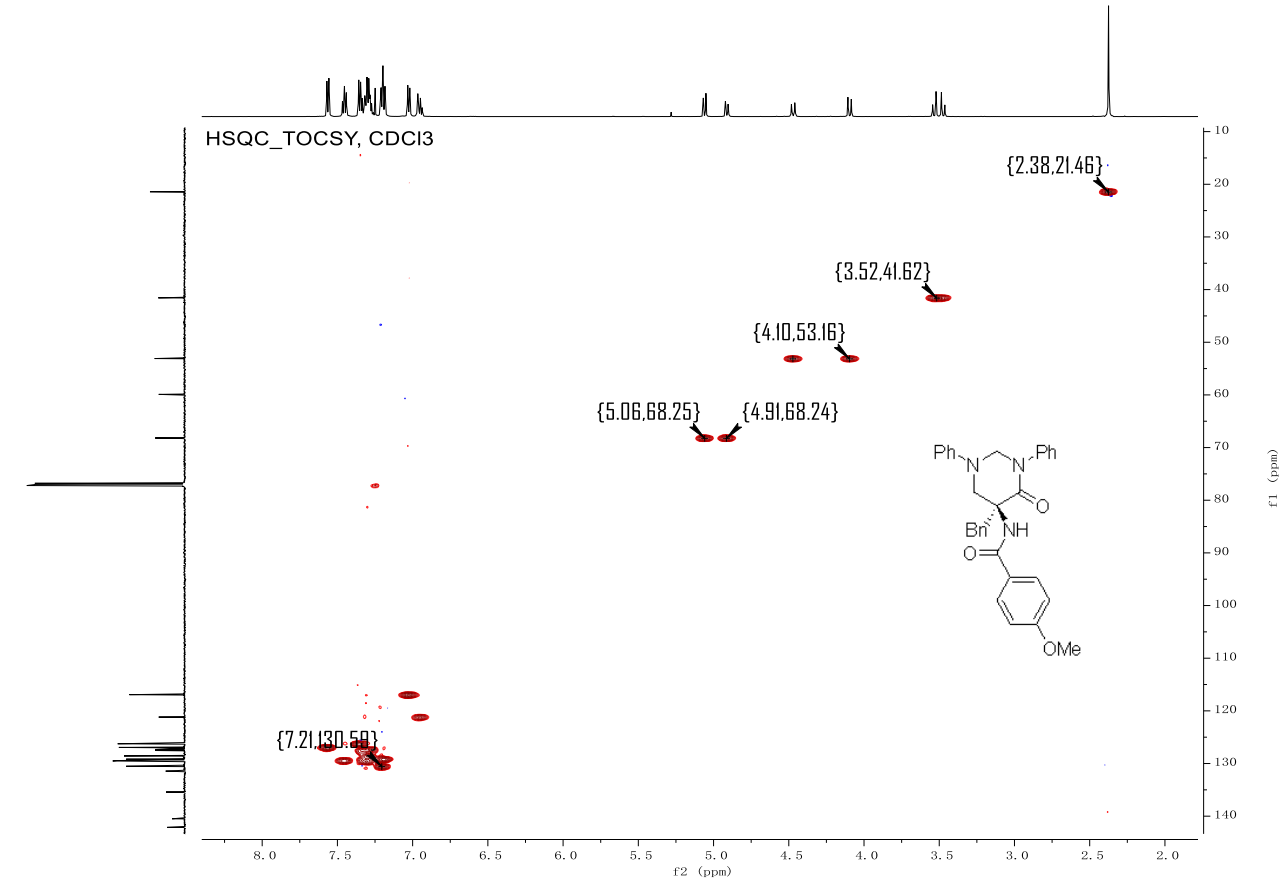
Formula	$C_{31}H_{29}N_3O_3$
Formula mass (amu)	491.57
Space group	$P2_12_12_1$
a (Å)	10.0721(3)
b (Å)	12.0628(4)
c (Å)	20.9044(6)
α (deg)	90
β (deg)	90
γ (deg)	90
V (Å^3)	2540.05(13)
Z	4
λ (Å)	1.54178
T (K)	172
ρ_{calcd} (g cm^{-3})	1.285
μ (mm^{-1})	0.667
Transmission factors	0.676–1.000
θ_{max} (deg)	68.129
No. of unique data, including $F_o^2 < 0$	4449
No. of unique data, with $F_o^2 > 2\sigma(F_o^2)$	4014
No. of variables	340
$R(F)$ for $F_o^2 > 2\sigma(F_o^2)$ [a]	0.0485
$R_w(F_o^2)$ [b]	0.1303
Goodness of fit	1.088

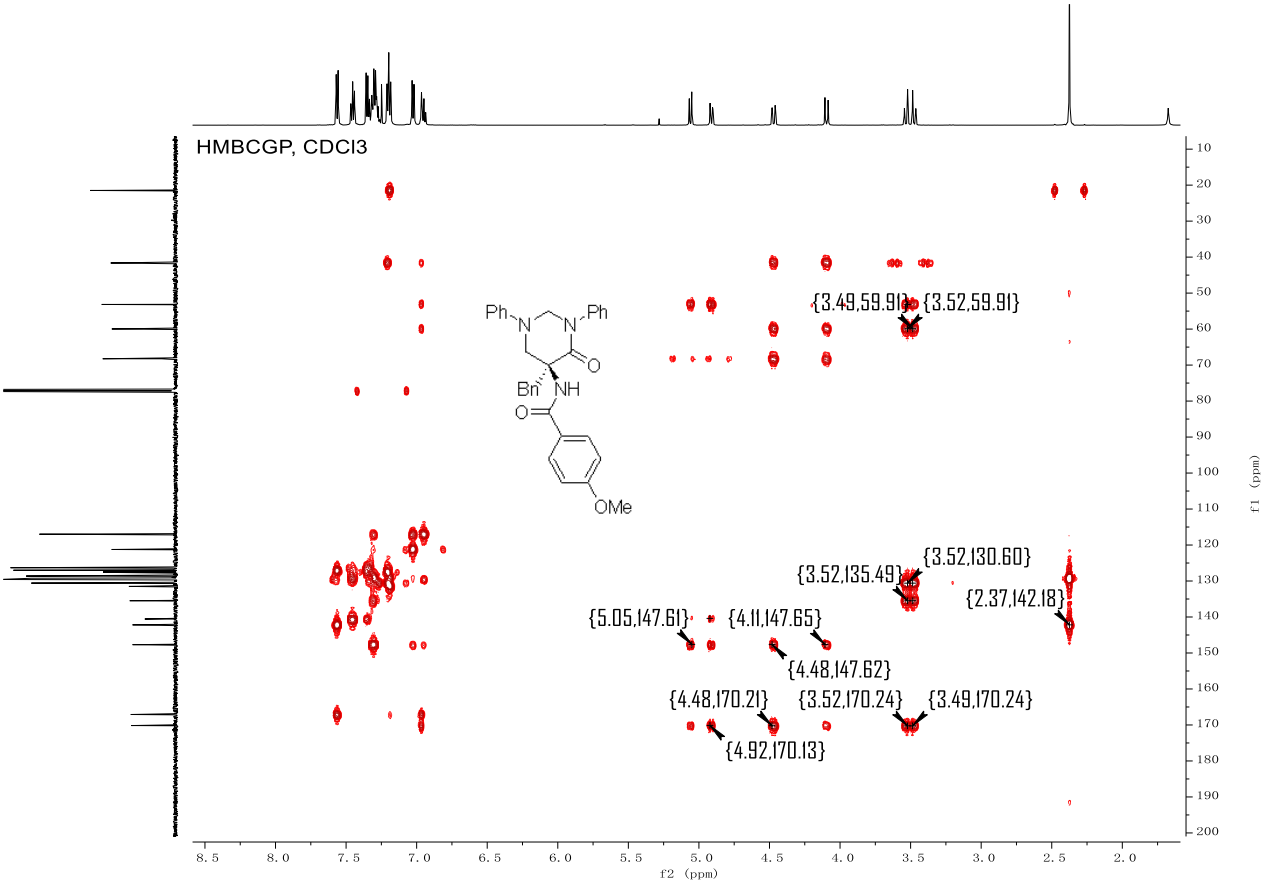
[a] $R(F) = \sum||F_o| - |F_c|| / \sum|F_o|$.

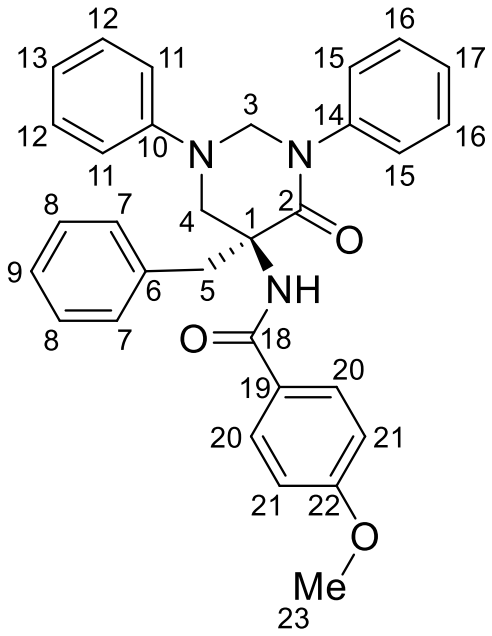
[b] $R_w(F_o^2) = [\sum[w(F_o^2 - F_c^2)^2] / \sum wF_o^4]^{1/2}$; $w^{-1} = [\sigma^2(F_o^2) + (Ap)^2 + Bp]$, where $p = [\max(F_o^2,0) + 2F_c^2] / 3$.

11. Analysis results of 2D NMR spectra of the product 3ac




HSQC_TOCSY, CDCl3
{2.38,21.46}
{3.52,41.62}
{4.10,53.16}
{5.06,68.25}
{4.91,68.24}
{7.21,130.58}
Ph
N
Ph
O
Bn
NH
O
OMe
f2 (ppm)
f1 (ppm)

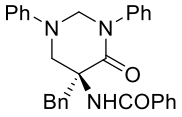

HMBCGP, CDCl3
{3.49,59.91}
{3.52,59.91}
{3.52,130.60}
{3.52,135.49}
{2.37,142.18}
{5.05,147.61}
{4.11,147.65}
{4.48,147.62}
{4.48,170.21}
{3.52,170.24}
{3.49,170.24}
{4.92,170.13}
Ph
N
Ph
O
Bn
NH
O
OMe
f2 (ppm)
f1 (ppm)



Number of Atom	H (ppm)	C (ppm)
1	--	59.9
2	--	170.2
3	4.93(dd), 5.08(d)	68.2
4	4.12(d), 4.49(dd)	53.2
5	3.53(q)	41.6
6	--	135.5
7	7.22(t)	130.6
8	7.02-7.59	116.0-131.5
9	7.02-7.59	116.0-131.5
10	--	147.6
11	7.02-7.59	116.0-131.5
12	7.02-7.59	116.0-131.5
13	7.02-7.59	116.0-131.5
14	--	140.4
15	7.02-7.59	116.0-131.5
16	7.02-7.59	116.0-131.5
17	7.02-7.59	116.0-131.5
18	--	167.0
19	--	116.0-131.5
20	7.02-7.59	116.0-131.5
21	7.02-7.59	116.0-131.5
22	--	142.2
23	2.40 (s)	21.5

12. Characterization of products

(*S*)-*N*-(5-benzyl-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)benzamide (3aa)



32.8 mg, 71% yield; white solid, melting point: 67.1-72.6 °C, $[\alpha]^{26}_{D}$ = -41.0 (*c* = 0.65, CH_2Cl_2).

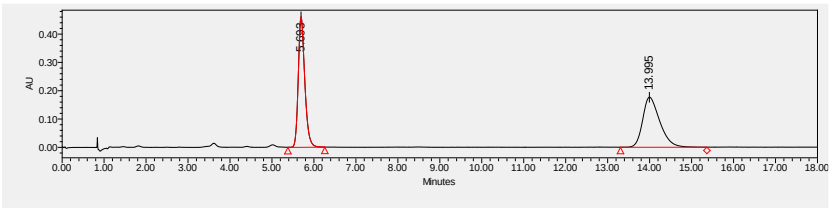
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 5.72 min, t_{major} = 13.66 min. er = 95:5.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.72 – 7.60 (m, 2H), 7.52 – 7.40 (m, 3H), 7.40 – 7.25 (m, 10H), 7.23 – 7.15 (m, 2H), 7.06 – 6.97 (m, 3H), 6.94 (t, *J* = 7.3, 1.1 Hz, 1H), 5.04 (d, *J* = 10.5 Hz, 1H), 4.89 (dd, *J* = 10.6, 1.6 Hz, 1H), 4.45 (dd, *J* = 13.4, 1.6 Hz, 1H), 4.10 (d, *J* = 13.4 Hz, 1H), 3.48 (m, 2H).

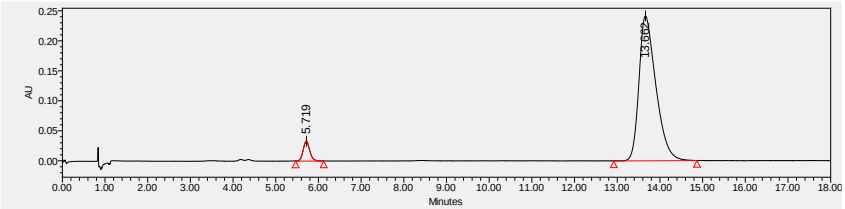
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.1, 167.1, 147.7, 140.5, 135.4, 134.3, 131.7, 130.6, 129.6, 129.5, 128.6, 128.6, 127.6, 127.4, 127.0, 126.3, 121.3, 117.0, 68.2, 60.0, 53.1, 41.6.

IR (neat): *ν* (cm^{-1}): 3318, 3060, 3032, 1651, 1490.

HRMS (ESI-TOF) calcd for $C_{30}H_{28}N_3O_2^+$ ([M]+H$^+$) = 462.2176, found 462.2170.

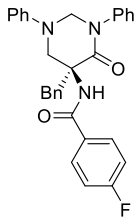


	Retention Time	Area	% Area
1	5.693	4959840	49.76
2	13.995	5008233	50.24



	Retention Time	Area	% Area
1	5.719	359738	5.14
2	13.662	6643964	94.86

(*S*)-*N*-(5-benzyl-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)-4-fluorobenzamide (3ab)



25.0 mg, 52% yield; white solid, melting point: 71.0-74.2 °C, $[\alpha]^{27}_{D}$ = -45.7 (*c* = 0.23, CH_2Cl_2).
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 4.13 min, t_{major} = 9.57 min. er = 93:7.

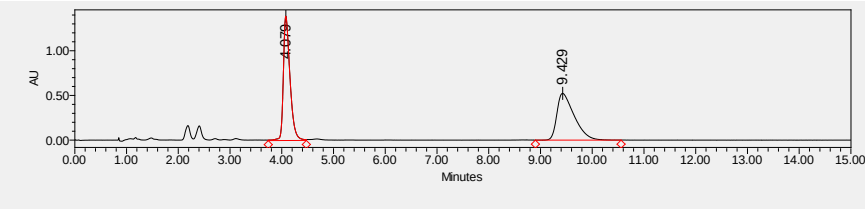
^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.72 – 7.60 (m, 2H), 7.54 – 7.41 (m, 2H), 7.39 – 7.28 (m, 8H), 7.24 – 7.16 (m, 2H), 7.12 – 7.00 (m, 4H), 7.00 – 6.89 (m, 2H), 5.05 (d, *J* = 10.6 Hz, 1H), 4.92 (dd, *J* = 10.6, 1.5 Hz, 1H), 4.47 (dd, *J* = 13.5, 1.6 Hz, 1H), 4.12 (d, *J* = 13.4 Hz, 1H), 3.53 (q, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) *δ* 170.1 , 166.1 , 147.7 , 140.5 , 135.4 , 130.6 , 129.7 (d, J_{C-F} = 6.5 Hz), 129.4 (d, J_{C-F} = 9.0 Hz), 128.7 , 127.7 , 127.5 , 126.3 , 117.0 , 115.6 (d, J_{C-F} = 22.0 Hz), 68.3 , 60.2 , 53.1 , 41.7.

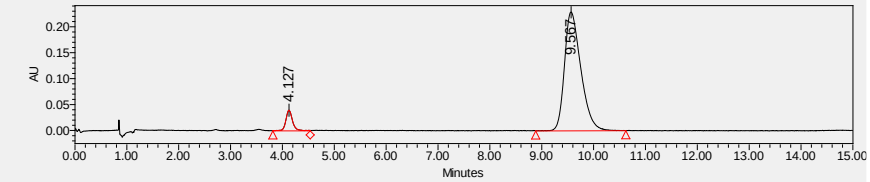
^{19}F NMR (376 MHz, $CDCl_3$) *δ* -107.79.

IR (neat): ν (cm^{-1}): 3317, 3062, 2922, 1652, 1492.

HRMS (ESI-TOF) calcd for $C_{30}H_{27}FN_3O_2^+$ ([M]+H^+) = 480.2082, found 480.2076.

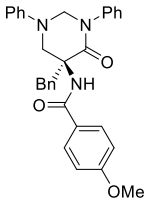


	Retention Time	Area	% Area
1	4.079	12382440	50.49
2	9.429	12140715	49.51



	Retention Time	Area	% Area
1	4.127	360461	6.69
2	9.567	5030362	93.31

(*S*)-*N*-(5-benzyl-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)-4-methoxybenzamide (3ac)



33.0 mg, 67% yield; white solid, melting point: 65.7-77.4 °C, $[\alpha]^{27}_D$ = -45.4 (*c* = 0.52, CH_2Cl_2).

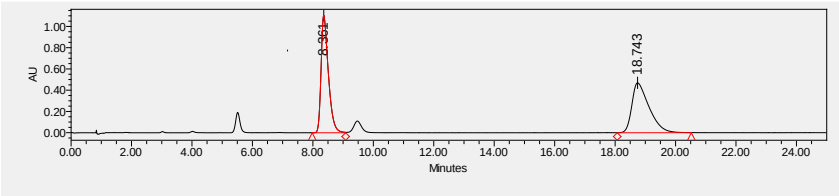
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 8.50 min, t_{major} = 19.18 min. er = 96:4.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.63 – 7.52 (m, 2H), 7.51 – 7.41 (m, 2H), 7.41 – 7.28 (m, 7H), 7.24 – 7.15 (m, 4H), 7.09 – 6.91 (m, 4H), 5.07 (d, *J* = 10.6 Hz, 1H), 4.92 (dd, *J* = 10.5, 1.6 Hz, 1H), 4.48 (dd, *J* = 13.4, 1.7 Hz, 1H), 4.10 (d, *J* = 13.4 Hz, 1H), 3.51 (q, *J* = 13.6 Hz, 2H), 2.39 (s, 3H).

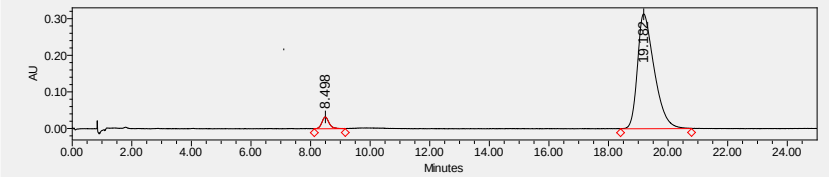
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.2, 167.1, 147.7, 142.2, 135.5, 131.5, 130.6, 129.6, 129.5, 129.3, 128.7, 127.6, 127.4, 127.0, 126.3, 121.3, 117.0, 68.3, 60.0, 53.2, 41.7, 21.5.

IR (neat): *ν* (cm^{-1}) 3332, 3032, 2929, 1650, 1602, 1492.

HRMS (ESI-TOF) calcd for $C_{31}H_{30}N_3O_3^+$ ([M]+H$^+$) = 492.2282, found 492.2292.

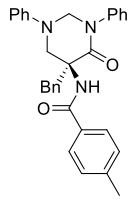


	Retention Time	Area	% Area
1	8.361	18411899	49.94
2	18.743	18458405	50.06



	Retention Time	Area	% Area
1	8.498	545674	4.27
2	19.182	12234418	95.73

(*S*)-*N*-(5-benzyl-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)-4-methylbenzamide (3ad)



37.1 mg, 78% yield; white solid, melting point: 64.2-68.4 ℃, $[\alpha]^{27}_{D}$ = -42.6 (*c* = 0.57, CH_2Cl_2).

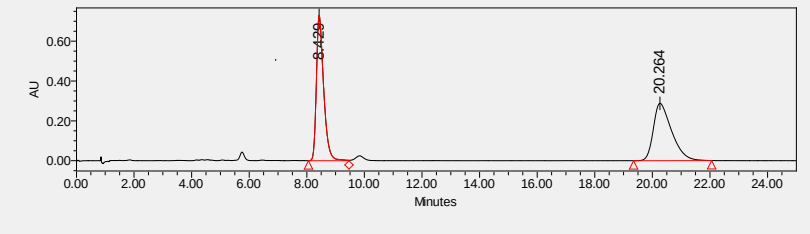
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 8.52 min, t_{major} = 19.97 min. er = 96:4.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.69 – 7.59 (m, 2H), 7.51 – 7.42 (m, 2H), 7.40 – 7.28 (m, 7H), 7.25 – 7.18 (m, 2H), 7.07 – 6.99 (m, 2H), 6.99 – 6.93 (m, 1H), 6.92 – 6.86 (m, 3H), 5.07 (d, *J* = 10.5 Hz, 1H), 4.92 (dd, *J* = 10.6, 1.6 Hz, 1H), 4.46 (dd, *J* = 13.4, 1.6 Hz, 1H), 4.09 (d, *J* = 13.4 Hz, 1H), 3.84 (s, 3H), 3.51 (q, *J* = 13.6 Hz, 2H).

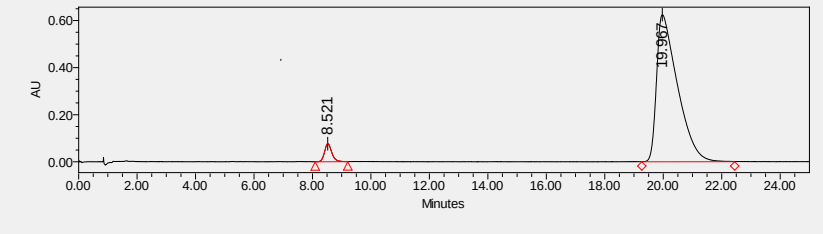
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.3, 166.7, 162.4, 147.7, 140.6, 135.5, 130.6, 129.6, 129.5, 128.9, 128.7, 127.6, 127.4, 126.6, 126.3, 121.3, 117.0, 113.8, 68.3, 59.9, 55.5, 53.2, 41.7.

IR (neat): ν (cm^{-1}) 3390, 3032, 2931, 1650, 1602, 1492.

HRMS (ESI-TOF) calcd for $C_{24}H_{23}N_3O_2^+$ ([M]-$PhCH_2$+H^+) = 385.1785, found 385.1790.

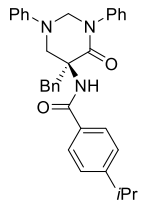


	Retention Time	Area	% Area
1	8.429	12768769	50.13
2	20.264	12703611	49.87



	Retention Time	Area	% Area
1	8.521	1327123	4.11
2	19.967	30992290	95.89

(*S*)-*N*-(5-benzyl-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)-4-isopropylbenzamide (3ae)



35.3 mg, 70% yield; white solid, melting point: 71.0-74.0 °C, $[\alpha]^{27}_{D}$ = -45.5 (*c* = 0.47, CH_2Cl_2).

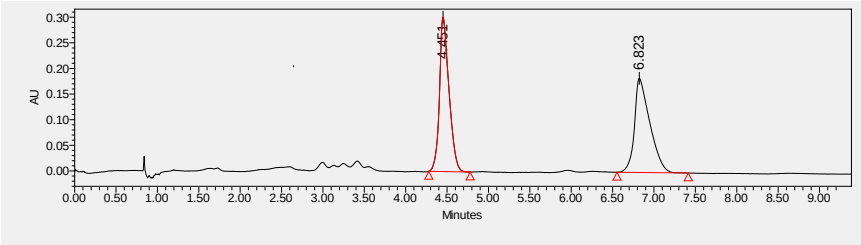
Dissolved in MeOH for SFC; SFC (Daicel chiralcel IB-3, CO_2/MeOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{major} = 4.46 min, t_{minor} = 6.93 min. er = 95:5.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.63 – 7.53 (m, 2H), 7.49 – 7.39 (m, 2H), 7.37 – 7.26 (m, 7H), 7.25 – 7.16 (m, 4H), 7.06 – 6.89 (m, 4H), 5.05 (d, *J* = 10.5 Hz, 1H), 4.89 (dd, *J* = 10.5, 1.6 Hz, 1H), 4.45 (dd, *J* = 13.4, 1.6 Hz, 1H), 4.07 (d, *J* = 13.4 Hz, 1H), 3.50 (q, 2H), 3.02 – 2.81 (m, 1H), 1.23 (d, *J* = 7.0 Hz, 6H).

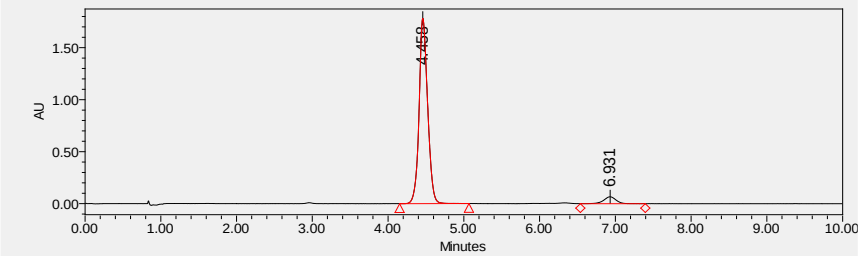
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.2, 167.1, 153.0, 147.7, 140.6, 135.5, 131.9, 130.6, 129.6, 129.5, 128.7, 127.6, 127.4, 127.2, 126.7, 126.3, 121.3, 117.0, 68.3, 60.0, 53.2, 41.7, 34.2, 23.8.

IR (neat): ν (cm^{-1}) 3390, 2961,1654, 1492.

HRMS (ESI-TOF) calcd for $C_{33}H_{34}N_3O_2^+$ ([M]+H$^+$) = 504.2646, found 504.2630.

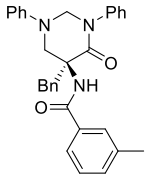


	Retention Time	Area	% Area
1	4.451	2411795	50.38
2	6.823	2375627	49.62



	Retention Time	Area	% Area
1	4.458	14003887	94.88
2	6.931	755634	5.12

(*S*)-*N*-(5-benzyl-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)-3-methylbenzamide (3af)



31.9 mg, 67% yield; white solid, melting point: 60.6-66.6 °C, $[\alpha]^{27}_D$ = -45.0 (*c* = 0.34, CH_2Cl_2).

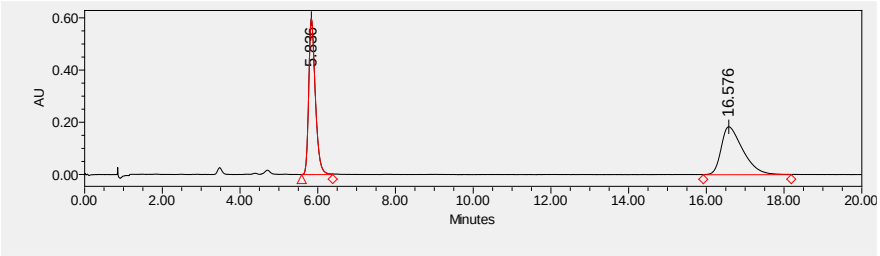
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 5.88 min, t_{major} = 16.24 min. er = 94:6.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.55 – 7.40 (m, 4H), 7.40 – 7.28 (m, 9H), 7.25 – 7.18 (m, 2H), 7.08 – 6.92 (m, 4H), 5.06 (d, *J* = 10.6 Hz, 1H), 4.92 (dd, *J* = 10.6, 1.6 Hz, 1H), 4.49 (dd, *J* = 13.4, 1.6 Hz, 1H), 4.13 (d, *J* = 13.4 Hz, 1H), 3.53 (dd, *J* =21.4, 13.6 Hz, 2H), 2.37 (s, 3H).

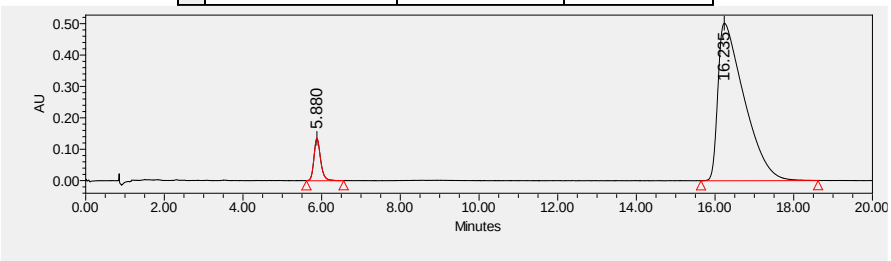
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.1, 167.4, 147.7, 140.6, 138.5, 135.5, 134.3, 132.5, 130.6, 129.6, 129.6, 128.7, 128.5, 127.8, 127.7, 127.5, 126.3, 124.0, 121.3, 117.0, 68.2, 60.1, 53.2, 41.7, 21.4.

IR (neat): ν (cm^{-1}) 3387, 3033, 1652, 1496.

HRMS (ESI-TOF) calcd for $C_{31}H_{30}N_3O_2^+$ ([M]+H$^+$) = 476.2333, found 476.2330.

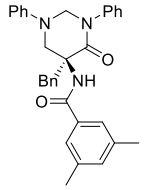


	Retention Time	Area	% Area
1	5.836	7025064	49.68
2	16.576	7116983	50.32



	Retention Time	Area	% Area
1	5.880	1591790	6.41
2	16.235	23236653	93.59

(*S*)-*N*-(5-benzyl-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)-3,5-dimethylbenzamide (3ag)



32.3 mg, 66% yield; white solid, melting point: 68.1-72.9 °C, $[\alpha]^{27}_{D}$ = -53.9 (*c* = 0.08, CH_2Cl_2).

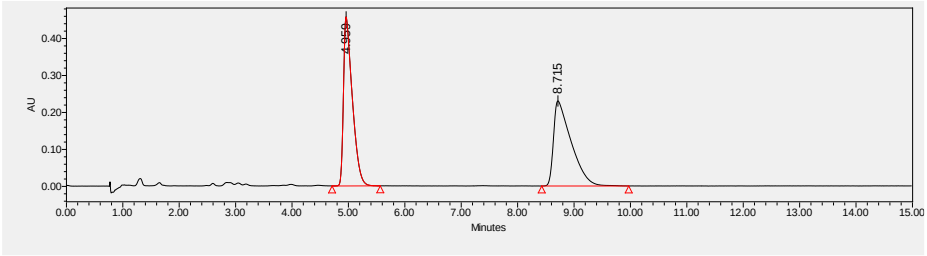
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 5.08 min, t_{major} = 8.97 min. er = 95:5.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.48 – 7.38 (m, 2H), 7.37 – 7.26 (m, 7H), 7.25 – 7.22 (m, 3H), 7.22 – 7.13 (m, 2H), 7.11 – 7.06 (m, 1H), 7.03 – 6.89 (m, 4H), 5.03 (d, *J* = 10.6 Hz, 1H), 4.88 (dd, *J* = 10.6, 1.5 Hz, 1H), 4.47 (dd, *J* = 13.5, 1.6 Hz, 1H), 4.10 (d, *J* = 13.4 Hz, 1H), 3.51 (dd, *J* = 20.5, 13.6 Hz, 2H), 2.29 (s, 6H).

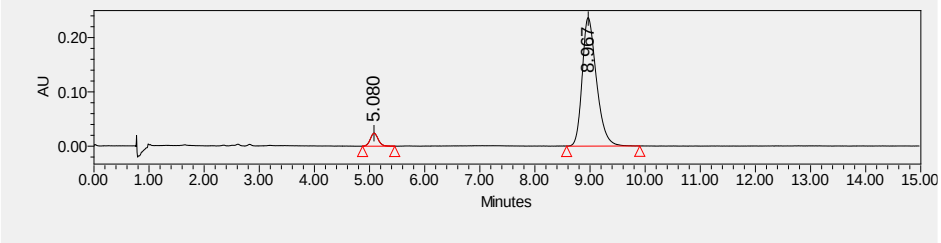
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.1, 167.5, 147.7, 140.5, 138.3, 135.6, 134.3, 133.3, 130.6, 129.6, 129.5, 128.6, 127.6, 127.4, 126.3, 124.8, 121.2, 116.9, 68.2, 60.1, 53.2, 41.7, 21.3, 21.3.

IR (neat): *ν* (cm^{-1}) 3330, 2923, 2859, 1656, 1599, 1497.

HRMS (ESI-TOF) calcd for $C_{32}H_{32}N_3O_2^+$ ([M]+H$^+$) = 490.2489, found 490.2482.

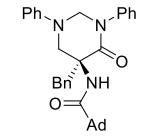


	Retention Time	Area	% Area
1	4.959	4961644	49.79
2	8.715	5004022	50.21



	Retention Time	Area	% Area
1	5.080	239816	5.33
2	8.967	4256797	94.67

(*S*)-N-(-5-benzyl-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)adamantane-1-carboxamide (3ah)



Ad = adamantan-1-yl

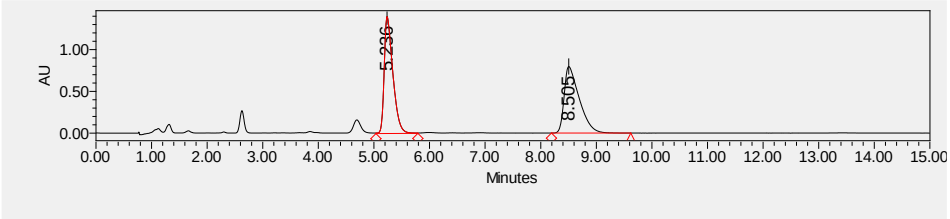
38.0 mg, 73% yield; white solid, melting point: 69.0-76.0 °C, $[\alpha]^{28}_{D}$ = -44.2 (*c* = 0.43, CH_2Cl_2).
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 5.33 min, t_{major} = 8.58 min. er = 93:7.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.51 – 7.39 (m, 2H), 7.37 – 7.26 (m, 8H), 7.23 – 7.09 (m, 2H), 7.04 – 6.89 (m, 3H), 6.45 (s, 1H), 5.03 (d, *J* = 10.5 Hz, 1H), 4.87 (dd, *J* = 10.5, 1.6 Hz, 1H), 4.33 (dd, *J* = 13.4, 1.7 Hz, 1H), 3.93 (d, *J* = 13.4 Hz, 1H), 3.37 (s, 2H), 2.01 (s, 1H), 1.84 – 1.59 (m, 14H).

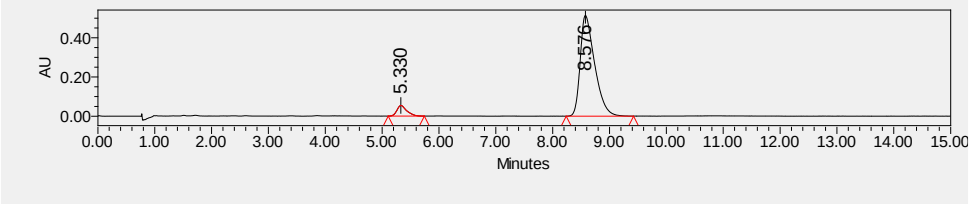
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 177.9, 170.4, 147.8, 140.7, 135.5, 130.7, 129.6, 129.5, 128.6, 127.5, 127.4, 126.2, 121.1, 117.0, 68.1, 59.1, 53.2, 41.6, 41.0, 39.1, 36.5, 28.2.

IR (neat): *ν* (cm^{-1}) 3400, 2906, 2851, 1655, 1495.

HRMS (ESI-TOF) calcd for $C_{34}H_{38}N_3O_2^+$ ([M]+H$^+$) = 520.2959, found 520.2949.

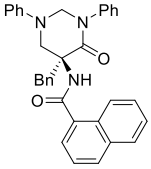


	Retention Time	Area	% Area
1	5.236	15090400	50.14
2	8.505	15008171	49.86



	Retention Time	Area	% Area
1	5.330	653564	6.72
2	8.576	9070412	93.28

(*S*)-N-(5-benzyl-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)-1-naphthamide (3ai)



33.8 mg, 66% yield; white solid, melting point: 71.3-78.3 °C, $[\alpha]^{27}_D$ = -51.1 (*c* = 0.54, CH_2Cl_2).

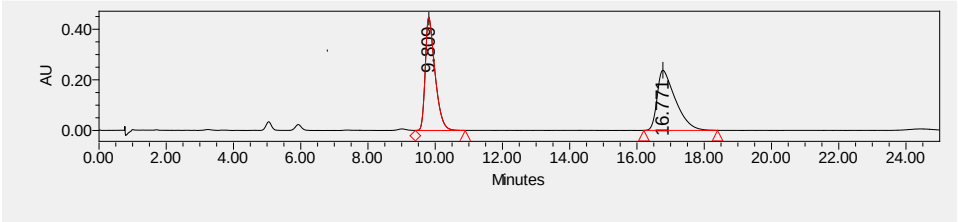
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 9.92 min, t_{major} = 16.30 min. er = 94:6.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 8.41 – 8.26 (m, 1H), 7.91 – 7.81 (m, 2H), 7.54 – 7.41 (m, 5H), 7.42 – 7.29 (m, 6H), 7.30 – 7.26 (m, 2H), 7.25 – 7.16 (m, 3H), 7.12 – 7.03 (m, 2H), 6.98 (t, *J* = 7.3 Hz, 1H), 6.75 (s, 1H), 5.09 (d, *J* = 10.7 Hz, 1H), 4.89 (d, *J* = 10.7 Hz, 1H), 4.41 (s, 2H), 3.54 (s, 2H).

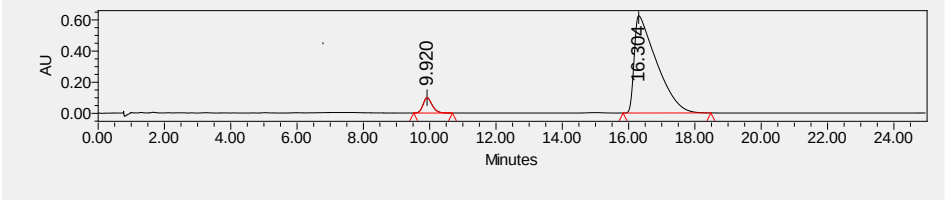
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.1, 169.5, 147.8, 140.6, 135.3, 133.8, 133.7, 131.0, 130.7, 130.2, 129.7, 129.6, 128.7, 128.3, 127.7, 127.5, 127.3, 126.5, 126.4, 125.5, 125.4, 124.7, 121.4, 117.2, 68.3, 60.6, 53.0, 42.0.

IR (neat): *ν* (cm^{-1}) 3385, 3299, 3056, 1651, 1491.

HRMS (ESI-TOF) calcd for $C_{34}H_{30}N_3O_2^+$ ([M]+H$^+$) = 512.2333, found 512.2325.

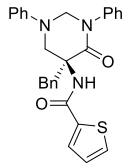


	Retention Time	Area	% Area
1	9.809	8943175	50.01
2	16.771	8938845	49.99



	Retention Time	Area	% Area
1	9.920	1894526	6.09
2	16.304	29237953	93.91

(*S*)-*N*-(5-benzyl-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)thiophene-2-carboxamide (3aj)



24.8 mg, 53% yield; white solid, melting point: 67.6-74.1 °C, $[\alpha]^{27}_{D}$ = -40.5 (*c* = 0.42, CH_2Cl_2).

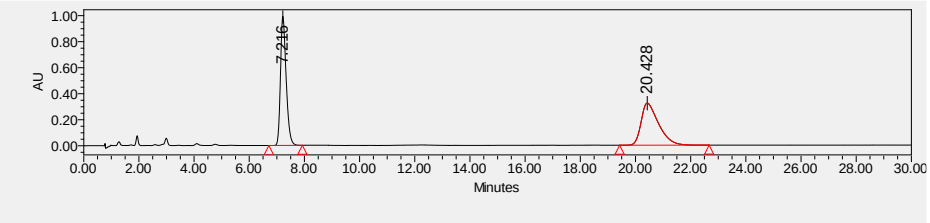
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 7.28 min, t_{major} = 20.50 min. er = 90:10.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.52 – 7.41 (m, 3H), 7.40 – 7.29 (m, 8H), 7.24 – 7.12 (m, 2H), 7.07 – 7.00 (m, 3H), 6.96 (t, *J* = 7.4 Hz, 1H), 6.86 (s, 1H), 5.06 (d, *J* = 10.5 Hz, 1H), 4.91 (dd, *J* = 10.5, 1.7 Hz, 1H), 4.44 (dd, *J* = 13.4, 1.8 Hz, 1H), 4.05 (d, *J* = 13.4 Hz, 1H), 3.49 (q, *J* = 13.6 Hz, 2H).

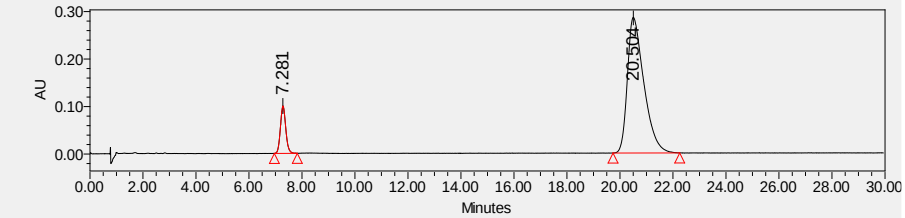
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 169.9, 161.6, 147.7, 140.5, 138.9, 135.3, 130.6, 130.5, 129.6, 129.6, 128.7, 128.4, 127.7, 127.7, 127.5, 126.4, 121.4, 117.1, 68.4, 60.0, 53.2, 41.7.

IR (neat): *ν* (cm^{-1}) 3324, 3062, 3032, 1645, 1494.

HRMS (ESI-TOF) calcd for $C_{28}H_{26}N_3O_2S^+$ ([M]+H$^+$) = 468.1740, found 468.1734.

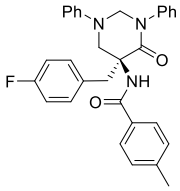


	Retention Time	Area	% Area
1	7.216	14188270	50.07
2	20.428	14149765	49.93



	Retention Time	Area	% Area
1	7.281	1396948	10.18
2	20.504	12321657	89.82

(*S*)-*N*-(5-(4-fluorobenzyl)-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)-4-methylbenzamide(3ak)



29.6 mg, 60% yield; white solid, melting point: 71.1-73.7 °C, $[\alpha]^{26}_{D}$ = -50.0 (*c* = 0.38, CH_2Cl_2).

Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 5.24 min, t_{major} = 12.87 min. er = 93:7.

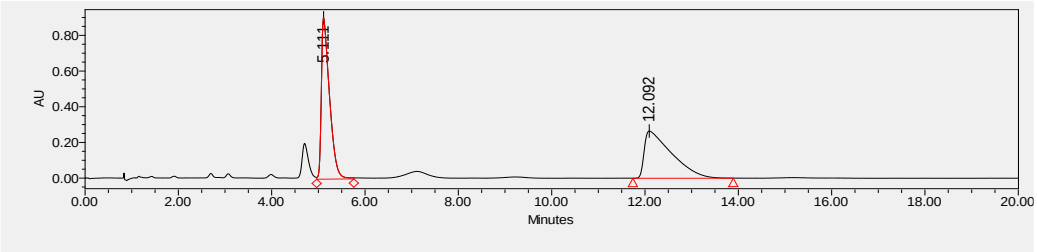
^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.62 – 7.53 (m, 2H), 7.51 – 7.43 (m, 2H), 7.41 – 7.28 (m, 5H), 7.25 – 7.09 (m, 4H), 7.07 – 6.89 (m, 6H), 5.04 (d, *J* = 10.5 Hz, 1H), 4.95 (dd, *J* = 10.5, 1.5 Hz, 1H), 4.61 (dd, *J* = 13.4, 1.6 Hz, 1H), 4.09 (d, *J* = 13.4 Hz, 1H), 3.59 (d, *J* = 13.8 Hz, 1H), 3.46 (d, *J* = 13.7 Hz, 1H), 2.39 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) *δ* 170.0, 167.3, 163.4, 160.9, 147.5, 142.3, 140.4, 132.0 (d, $J_{C\text{-}F}$ = 8.0 Hz), 131.5, 131.3(d, $J_{C\text{-}F}$ = 3.3 Hz), 129.6, 129.3, 127.8, 127.0, 126.3, 121.3, 116.8, 115.3 (d, $J_{C\text{-}F}$ = 21.2 Hz), 68.2, 60.4, 53.6, 40.9, 21.5 (d, $J_{C\text{-}F}$ = 2.3 Hz).

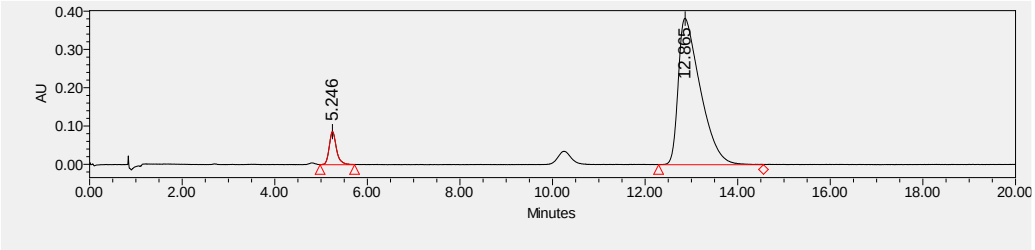
^{19}F NMR (376 MHz, $CDCl_3$) *δ* -115.32.

IR (neat): *ν* (cm^{-1}) 3330, 3037, 2923, 1648, 1496.

HRMS (ESI-TOF) calcd for $C_{31}H_{29}N_3O_2F^+$ ([M]+H^+) = 494.2238, found 494.2232.

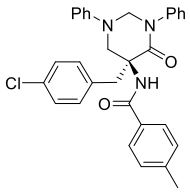


	Retention Time	Area	% Area
1	5.111	10946264	50.43
2	12.092	10761127	49.57



	Retention Time	Area	% Area
1	5.246	943050	6.95
2	12.865	12635540	93.05

(*S*)-*N*-(5-(4-chlorobenzyl)-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)-4-methylbenzamide(3al)



29.6 mg, 58% yield; white solid, melting point: 81.3-85.4 °C, $[\alpha]^{27}_{D}$ = -48.4 (*c* = 0.55, CH_2Cl_2).

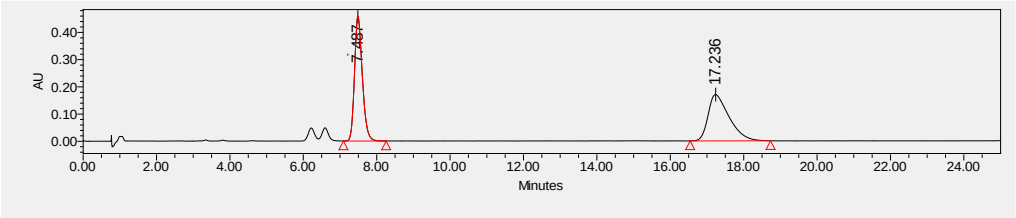
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 7.52 min, t_{major} = 16.66 min. er = 90:10.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.61 – 7.54 (m, 2H), 7.52 – 7.43 (m, 2H), 7.41 – 7.27 (m, 5H), 7.25 – 7.18 (m, 4H), 7.13 – 7.06 (m, 2H), 7.04 – 6.90 (m, 2H), 5.04 (d, *J* = 10.4 Hz, 1H), 4.96 (dd, *J* = 10.5, 1.5 Hz, 1H), 4.63 (dd, *J* = 13.5, 1.6 Hz, 1H), 4.07 (d, *J* = 13.4 Hz, 1H), 3.61 (d, *J* = 13.6 Hz, 1H), 3.46 (d, *J* = 13.6 Hz, 1H), 2.39 (s, 3H).

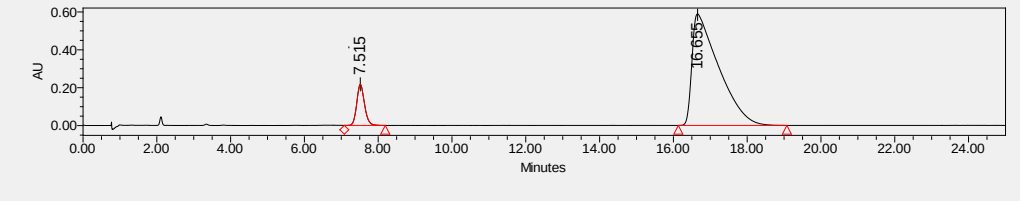
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 169.8, 167.3, 147.5, 142.4, 140.3, 134.1, 133.2, 131.8, 131.5, 129.6, 129.3, 128.6, 127.8, 127.0, 126.2, 121.3, 116.8, 68.3, 60.4, 53.7, 41.0, 21.6.

IR (neat): ν (cm^{-1}) 3330, 3035, 2923, 1650, 1493.

HRMS (ESI-TOF) calcd for $C_{31}H_{29}N_3O_2Cl^+$ ([M]+H$^+$) = 510.1943, 511, 512.1913, found 510.1933, 512.1912.

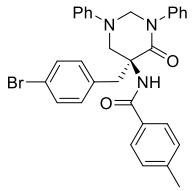


	Retention Time	Area	% Area
1	7.487	6768734	50.08
2	17.236	6747364	49.92



	Retention Time	Area	% Area
1	7.515	3163558	9.65
2	16.655	29635922	90.35

(*S*)-*N*-(5-(4-bromobenzyl)-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)-4-methylbenzamide(3am)



29.4 mg, 53% yield; white solid, melting point: 85.0-89.2 °C, $[\alpha]^{26}_{D}$ = -40.0 (*c* = 0.57, CH_2Cl_2).

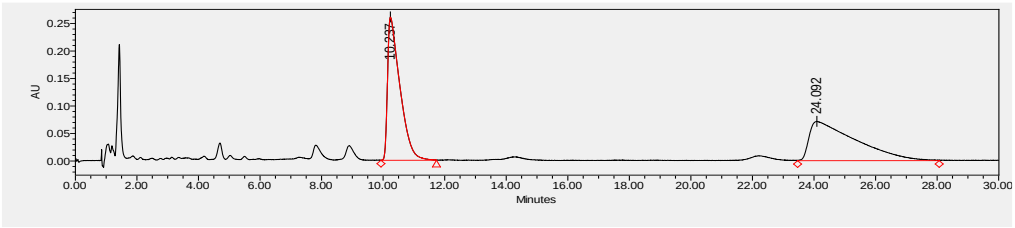
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 10.52 min, t_{major} = 24.59 min. er = 89:11.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.63 – 7.53 (m, 2H), 7.51 – 7.43 (m, 2H), 7.41 – 7.27 (m, 7H), 7.25 – 7.18 (m, 2H), 7.10 – 6.91 (m, 6H), 5.03 (d, *J* = 10.4 Hz, 1H), 4.96 (dd, *J* = 10.4, 1.6 Hz, 1H), 4.63 (dd, *J* = 13.5, 1.6 Hz, 1H), 4.06 (d, *J* = 13.4 Hz, 1H), 3.60 (d, *J* = 13.6 Hz, 1H), 3.44 (d, *J* = 13.6 Hz, 1H), 2.39 (s, 3H).

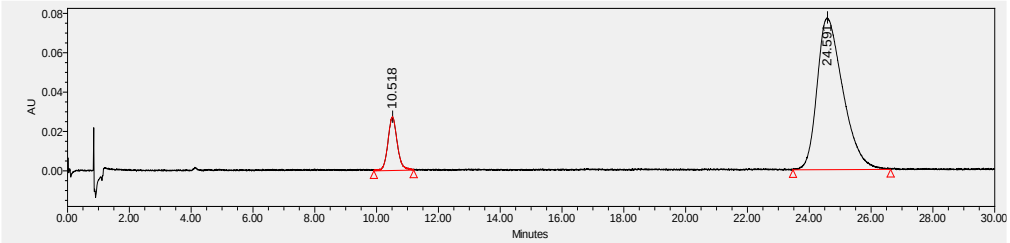
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 169.8, 167.3, 147.5, 142.4, 140.3, 134.7, 132.2, 131.5, 131.5, 129.6, 129.3, 127.8, 127.0, 126.2, 121.4, 121.3, 116.8, 68.3, 60.4, 53.7, 41.1, 21.6.

IR (neat): *ν* (cm^{-1}) 3328, 3035, 2922, 2855, 1649, 1492.

HRMS (ESI-TOF) calcd for $C_{31}H_{29}N_3O_2Br^+$ ([M]+H^+) = 554.1438, 556.1417, found 554.1428, 556.1407.

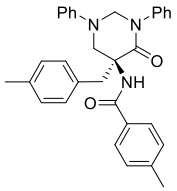


	Retention Time	Area	% Area
1	10.237	7262825	49.19
2	24.092	7501355	50.81



	Retention Time	Area	% Area
1	10.518	574070	11.39
2	24.591	4467462	88.61

(*S*)-4-methyl-*N*-(5-(4-methylbenzyl)-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)benzamide(3an)



37.8 mg, 71% yield; white solid, melting point: 68.2-73.6 °C, $[\alpha]^{27}_{D}$ = -46.7 (*c* = 0.55, CH_2Cl_2).

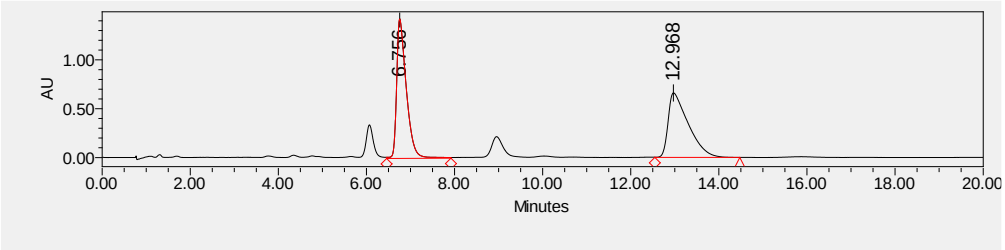
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 6.88 min, t_{major} = 12.80 min. er = 94:6.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.62 – 7.54 (m, 2H), 7.50 – 7.42 (m, 2H), 7.41 – 7.27 (m, 4H), 7.24 – 7.17 (m, 2H), 7.16 – 7.07 (m, 4H), 7.08 – 7.01 (m, 2H), 6.99 – 6.92 (m, 2H), 5.10 (d, J = 10.6 Hz, 1H), 4.95 (dd, J = 10.6, 1.6 Hz, 1H), 4.43 (dd, J = 13.4, 1.7 Hz, 1H), 4.07 (d, J = 13.3 Hz, 1H), 3.48 (dd, *J* = 30.0, 13.7 Hz, 2H), 2.39 (s, 3H), 2.34 (s, 3H).

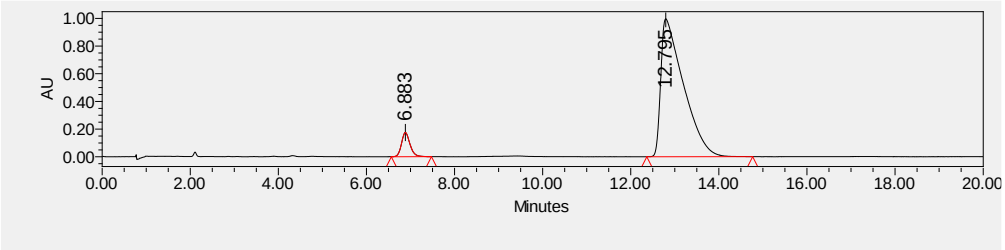
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.3, 167.0, 147.8, 142.2, 140.7, 137.1, 132.3, 131.5, 130.4, 129.6, 129.5, 129.4, 129.3, 127.6, 127.0, 126.4, 121.2, 117.1, 68.3, 59.9, 53.1, 41.1, 21.5, 21.2.

IR (neat): ν (cm^{-1}) 3389, 3031, 2922, 1651, 1490.

HRMS (ESI-TOF) calcd for $C_{32}H_{32}N_3O_2^+$ ([M]+H$^+$) = 490.2489, found 490.2480.

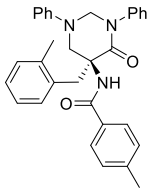


	Retention Time	Area	% Area
1	6.756	21630002	50.77
2	12.968	20975753	49.23



	Retention Time	Area	% Area
1	6.883	2344167	6.20
2	12.795	35448342	93.80

(*S*)-4-methyl-*N*-(5-(2-methylbenzyl)-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)benzamide(3ao)



25.5mg, 52% yield; white solid, melting point: 66.8-72.0 °C, $[\alpha]^{27}_D$ = -50.5 (c = 0.44, CH_2Cl_2).

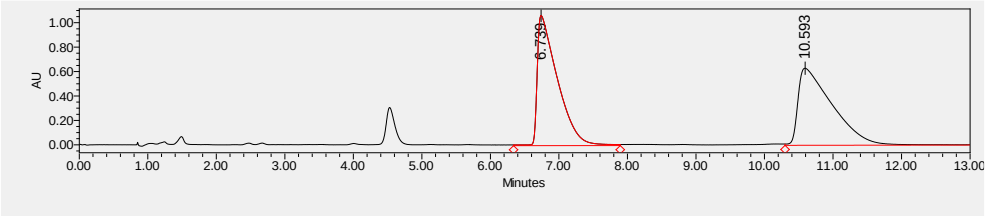
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 7.00 min, t_{major} = 10.14 min. er = 97:3.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.59 – 7.51 (m, 2H), 7.50 – 7.42 (m, 2H), 7.42 – 7.27 (m, 5H), 7.23 – 7.09 (m, 6H), 7.08 – 7.00 (m, 2H), 6.98 – 6.89 (m, 2H), 5.01 (d, *J* = 10.4 Hz, 1H), 4.91 (dd, *J* = 10.4, 1.6 Hz, 1H), 4.48 (dd, *J* = 13.4, 1.6 Hz, 1H), 4.15 (d, *J* = 13.4 Hz, 1H), 3.68 (d, *J* = 14.0 Hz, 1H), 3.49 (d, *J* = 14.1 Hz, 1H), 2.37 (d, 6H).

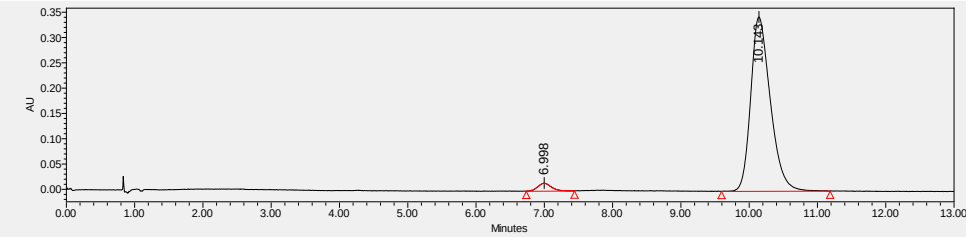
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.3, 167.1, 147.8, 142.2, 140.5, 137.8, 134.1, 131.5, 131.1, 130.9, 129.6, 129.5, 129.3, 127.7, 127.5, 127.0, 126.4, 126.1, 121.4, 117.1, 68.5, 60.4, 53.6, 38.1, 21.5, 20.1.

IR (neat): ν (cm^{-1}) 3395, 3031, 2922, 1650, 1488.

HRMS (ESI-TOF) calcd for $C_{32}H_{32}N_3O_2^+$ ([M]+H$^+$) = 490.2489, found 490.2479.

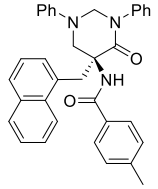


	Retention Time	Area	% Area
1	6.739	21778355	49.73
2	10.593	22011822	50.27



	Retention Time	Area	% Area
1	6.998	223165	3.14
2	10.143	6892722	96.86

(*S*)-4-methyl-*N*-(5-(naphthalen-1-ylmethyl)-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)benzamide(3ap)



27.4 mg, 52% yield; white solid, melting point: 77.5-84.5 °C, $[\alpha]^{26}_{D}$ = -51.1 (*c* = 0.56, CH_2Cl_2).

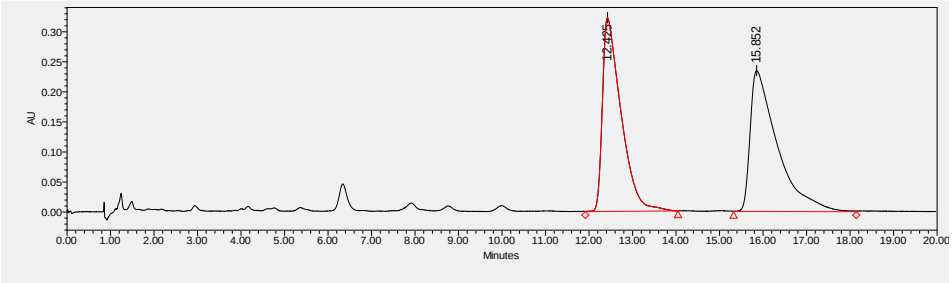
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 12.59 min, t_{major} = 15.67 min. er = 97:3.

^{1}H NMR (400 MHz, Chloroform-d) *δ* 8.38 (d, *J* = 8.4 Hz, 1H), 7.90 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.98 – 7.73 (m, 2H), 7.56 – 7.28 (m, 13H), 7.15 – 7.03 (m, 4H), 7.04 – 6.94 (m, 1H), 6.86 (s, 1H), 5.10 (d, *J* = 10.4 Hz, 1H), 4.94 (dd, *J* = 10.4, 1.8 Hz, 1H), 4.32 (dd, *J* = 13.2, 1.9 Hz, 1H), 4.18 (d, *J* = 14.1 Hz, 1H), 4.02 (d, *J* = 13.1 Hz, 1H), 3.82 (d, *J* = 14.1 Hz, 1H), 2.34 (s, 3H).

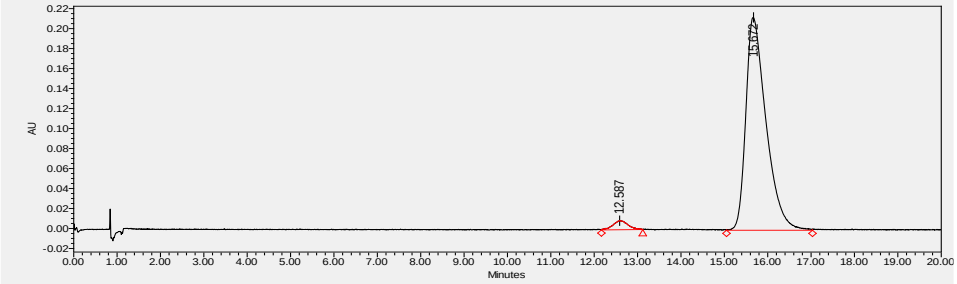
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.6, 166.9, 147.9, 142.1, 140.6, 134.1, 133.2, 131.8, 131.1, 129.6, 129.4, 129.2, 129.1, 128.4, 127.5, 127.0, 126.9, 126.5, 126.0, 125.5, 124.3, 121.7, 117.7, 69.0, 60.3, 52.9, 37.3, 21..

IR (neat): ν (cm^{-1}) 3434, 2922, 1655, 1491.

HRMS (ESI-TOF) calcd for $C_{35}H_{32}N_3O_2^+$ ([M]+H$^+$) = 526.2489, found 526.2483.

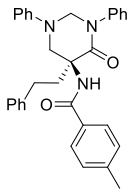


	Retention Time	Area	% Area
1	12.425	9592780	48.50
2	15.852	10185389	51.50



	Retention Time	Area	% Area
1	12.587	218607	3.07
2	15.672	6892713	96.93

(*S*)-4-methyl-*N*-(4-oxo-5-phenethyl-1,3-diphenylhexahydropyrimidin-5-yl)benzamide(3aq)



31.9 mg, 65% yield; white solid, melting point: 80.3-85.7 °C, $[\alpha]^{27}_{D}$ = -52.4 (*c* = 0.54, CH_2Cl_2).

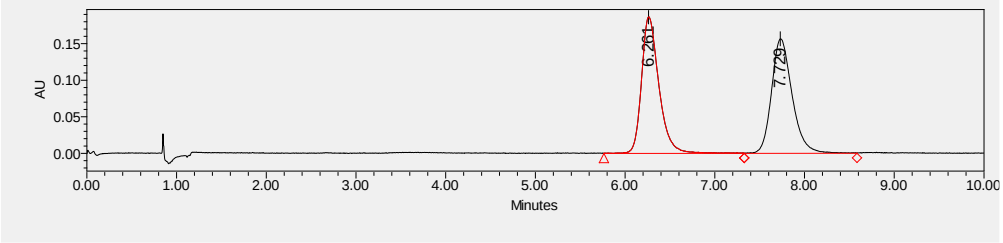
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 6.30 min, t_{major} = 7.64 min. er = 93:7.

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.56 (s, 1H), 7.80 (d, *J* = 8.2 Hz, 2H), 7.50 – 7.37 (m, 4H), 7.36 – 7.25 (m, 5H), 7.24 – 7.07 (m, 5H), 7.02 – 6.94 (m, 2H), 6.90 (t, *J* = 7.3 Hz, 1H), 5.30 (d, *J* = 11.5 Hz, 1H), 5.01 (dd, *J* = 11.5, 2.3 Hz, 1H), 4.25 (dd, *J* = 13.7, 2.4 Hz, 1H), 4.03 (d, *J* = 13.7 Hz, 1H), 2.82 (td, *J* = 12.9, 4.6 Hz, 1H), 2.64 (td, *J* = 12.9, 4.4 Hz, 1H), 2.37 (s, 3H), 2.13 – 2.00 (m, 1H), 1.85 (td, *J* = 13.5, 4.6 Hz, 1H).

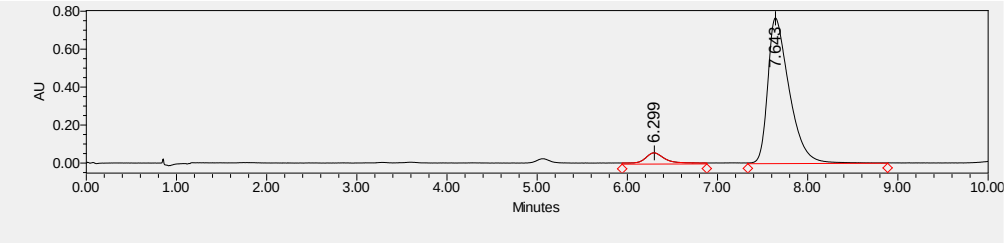
^{13}C NMR (101 MHz, DMSO-d_6) δ 171.5, 167.5, 148.6, 142.1, 142.0, 141.8, 131.6, 129.9, 129.4, 129.2, 128.7, 128.6, 128.4, 127.0, 126.7, 126.2, 120.5, 116.8, 66.6, 59.0, 53.6, 29.8, 21.5.

IR (neat): ν (cm^{-1}) 3298, 3032, 2925, 1645, 1492.

HRMS (ESI-TOF) calcd for $C_{32}H_{32}N_3O_2^+$ ([M]+H$^+$) = 490.2489, found 490.2481.

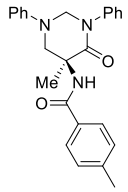


	Retention Time	Area	% Area
1	6.261	2516272	50.21
2	7.729	2494779	49.79



	Retention Time	Area	% Area
1	6.299	1025301	7.43
2	7.643	12766352	92.57

(*S*)-4-methyl-*N*-(5-methyl-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)benzamide(3ar)



24.8 mg, 62% yield; white solid, melting point: 85.6-88.7 °C, $[\alpha]^{23}_{D}$ = -46.8 (*c* = 0.53, CH_2Cl_2).

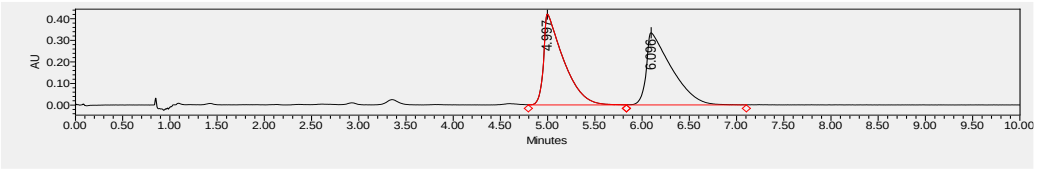
Dissolved in MeOH for SFC; SFC (Daicel chiralcel IB-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{major} = 5.12 min, t_{minor} = 6.30 min. er = 88:12.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.69 (d, *J* = 8.3 Hz, 2H), 7.51 – 7.38 (m, 4H), 7.37 – 7.27 (m, 3H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.01 (d, 2H), 6.94 (m, 1H), 6.82 (s, 1H), 5.24 (d, *J* = 10.9 Hz, 1H), 5.01 (dd, *J* = 10.9, 2.3 Hz, 1H), 4.30 (dd, *J* = 13.4, 2.4 Hz, 1H), 4.09 (d, *J* = 13.3 Hz, 1H), 2.39 (s, 3H), 1.63 (s, 3H).

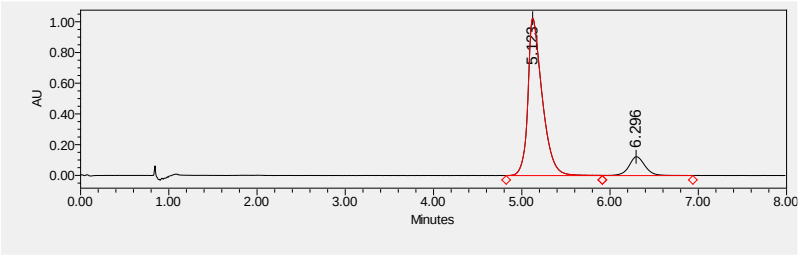
^{13}C NMR (101 MHz, $CDCl_3$) δ 171.7, 167.4, 148.0, 142.3, 140.6, 131.2, 129.6, 129.5, 129.2, 127.5, 127.2, 126.5, 121.0, 116.6, 67.4, 56.7, 56.5, 25.0, 21.5.

IR (neat): ν (cm^{-1}) 3316, 3035, 2986, 2925, 1644, 1496.

HRMS (ESI-TOF) calcd for $C_{25}H_{26}N_3O_2^+$ ([M]+H$^+$) = 400.2020, found 400.2016.

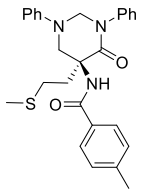


	Retention Time	Area	% Area
1	4.997	6172509	49.93
2	6.096	6190805	50.07



	Retention Time	Area	% Area
1	5.123	11534460	87.93
2	6.296	1583777	12.07

(*S*)-4-methyl-*N*-(5-(2-(methylthio)ethyl)-4-oxo-1,3-diphenylhexahydropyrimidin-5-yl)benzamide(3as)



29.4 mg, 64% yield; white solid, melting point: 59.4-63.0 °C, $[\alpha]^{26}_{D}$ = -47.0 (*c* = 0.53, CH_2Cl_2).

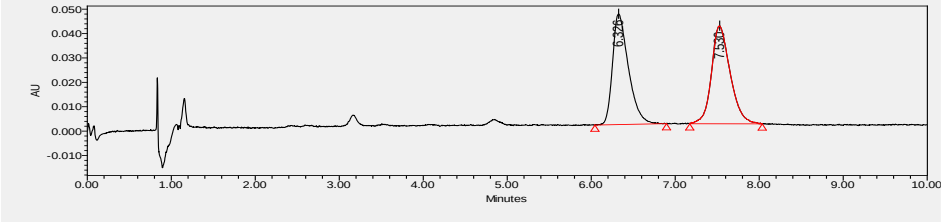
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 6.38 min, t_{major} = 7.47 min. er = 87:13.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 (d, *J* = 8.4 Hz, 3H), 7.52 – 7.27 (m, 7H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.05 – 6.90 (m, 3H), 5.18 (d, *J* = 10.6 Hz, 1H), 5.01 (dd, *J* = 10.6, 2.1 Hz, 1H), 4.44 (dd, *J* = 13.5, 2.1 Hz, 1H), 4.06 (d, *J* = 13.4 Hz, 1H), 2.85 – 2.57 (m, 2H), 2.52 – 2.25 (m, 5H), 2.07 (s, 3H).

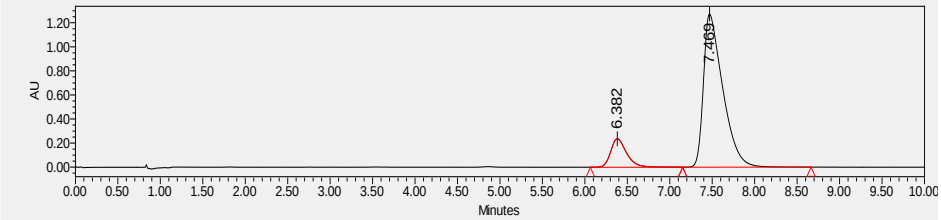
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.5, 167.2, 147.8, 142.3, 140.5, 131.1, 129.7, 129.6, 129.3, 127.7, 127.2, 126.5, 121.2, 116.6, 67.7, 59.6, 54.8, 35.1, 28.7, 21.5, 15.7.

IR (neat): ν (cm^{-1}) 3327, 3035, 2918, 1647, 1529.

HRMS (ESI-TOF) calcd for $C_{27}H_{30}N_3O_2S^+$ ([M]+H^+) = 460.2053, found 460.2051.

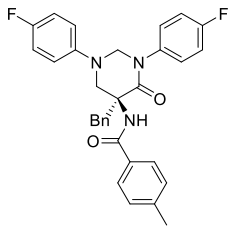


	Retention Time	Area	% Area
1	6.326	597246	49.07
2	7.530	619807	50.93

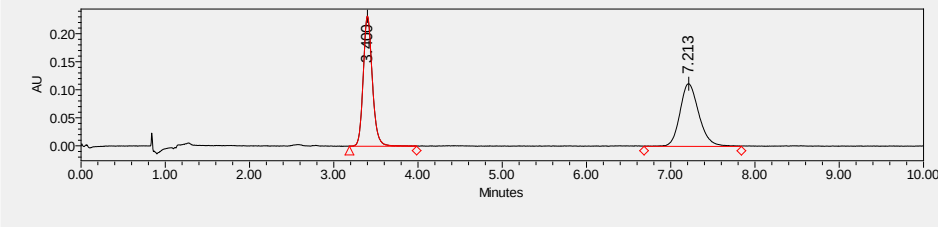


	Retention Time	Area	% Area
1	6.382	3080735	13.31
2	7.469	20058271	86.69

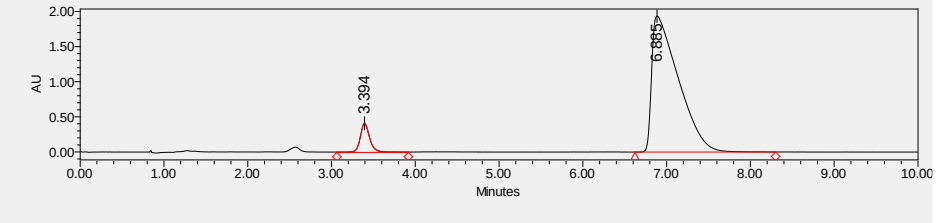
(*S*)-*N*-(5-benzyl-1,3-bis(4-fluorophenyl)-4-oxohexahydropyrimidin-5-yl)-4-methylbenzamide(3bd)



34.8 mg, 68% yield; white solid, melting point: 68.7-71.8 °C, $[\alpha]^{26}_{D}$ = -38.1 (*c* = 0.67, CH_2Cl_2).
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 3.39 min, t_{major} = 6.89 min. er = 93:7.
^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.57 (d, *J* = 8.2 Hz, 2H), 7.40 – 7.28 (m, 5H), 7.23 – 7.09 (m, 6H), 7.07 – 6.94 (m, 4H), 6.87 (s, 1H), 4.98 (d, *J* = 10.3 Hz, 1H), 4.76 (dd, *J* = 10.2, 1.8 Hz, 1H), 4.19 (dd, *J* = 13.2, 1.8 Hz, 1H), 4.02 (d, *J* = 13.1 Hz, 1H), 3.49 (dd, *J* = 31.2, 13.5 Hz, 2H), 2.38 (s, 3H).
^{13}C NMR (101 MHz, Chloroform-d) *δ* 170.4, 167.1, 162.9, 160.4 , 159.4, 157.0, 144.2 (d, $J_{C\text{-}F}$ = 2.6 Hz), 142.4, 136.5 (d, $J_{C\text{-}F}$ = 3.3 Hz), 135.3, 131.2 , 130.6, 129.3, 128.8, 128.3 (d, $J_{C\text{-}F}$ = 8.6 Hz), 127.6, 127.0, 119.5 (d, $J_{C\text{-}F}$ = 7.7 Hz), 116.5, 116.3, 116.1, 69.8, 59.6, 53.8, 41.9, 21.5.
^{19}F NMR (376 MHz, $CDCl_3$) *δ* -113.79, -121.65.
IR (neat): *ν* (cm^{-1}) 3332, 3058, 2925, 1648, 1503.
HRMS (ESI-TOF) calcd for $C_{31}H_{28}N_3O_2F_2^+$ ([M]+H$^+$) = 512.2144, found 512.2150.

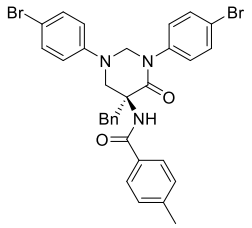


	Retention Time	Area	% Area
1	3.400	1746982	49.98
2	7.213	1748425	50.02

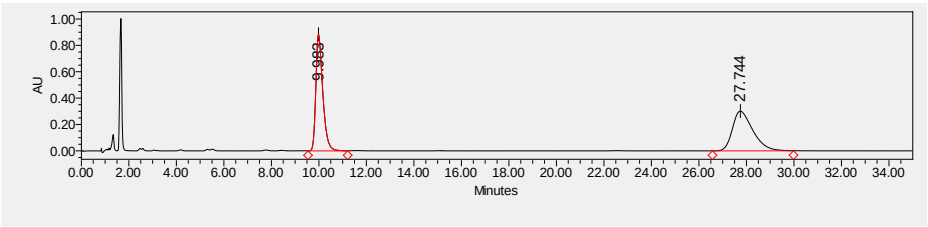


	Retention Time	Area	% Area
1	3.394	3364571	7.27
2	6.885	42896525	92.73

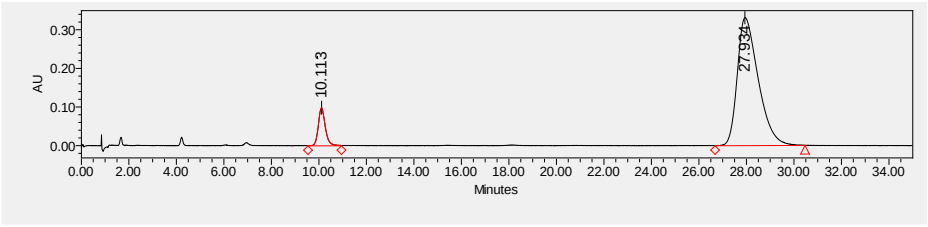
(*S*)-*N*-(5-benzyl-1,3-bis(4-bromophenyl)-4-oxohexahydropyrimidin-5-yl)-4-methylbenzamide(3cd)



43.7 mg, 69% yield; white solid, melting point: 88.7-93.4 °C, $[\alpha]^{26}_{D}$ = -47.5 (*c* = 0.68, CH_2Cl_2).
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 10.11 min, t_{major} = 27.93 min. er = 91:9.
^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.64 – 7.50 (m, 4H), 7.45 – 7.36 (m, 2H), 7.36 – 7.27 (m, 3H), 7.26 – 7.12 (m, 6H), 6.95 – 6.78 (m, 3H), 5.02 (d, *J* = 10.6 Hz, 1H), 4.80 (dd, *J* = 10.6, 1.7 Hz, 1H), 4.28 (dd, *J* = 13.5, 1.8 Hz, 1H), 4.06 (d, *J* = 13.4 Hz, 1H), 3.44 (dd, *J* = 19.1, 13.6 Hz, 2H), 2.39 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.2, 167.2, 146.7, 142.5, 139.5, 135.1, 132.7, 132.5, 131.2, 130.5, 129.3, 128.8, 128.0, 127.7, 127.0, 121.3, 118.8, 113.9, 68.1, 59.7, 53.0, 41.9, 21.6.
IR (neat): ν (cm^{-1}) 3331, 3031, 2924, 1650, 1487.
HRMS (ESI-TOF) calcd for $C_{31}H_{28}N_3O_2Br_2^+$ ([M]+H$^+$) = 632.0543, 634.0522, 636.0502, found 632.0538, 634.0515, 636.0501.

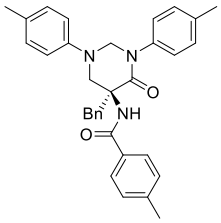


	Retention Time	Area	% Area
1	9.983	17959307	49.95
2	27.744	17994153	50.05

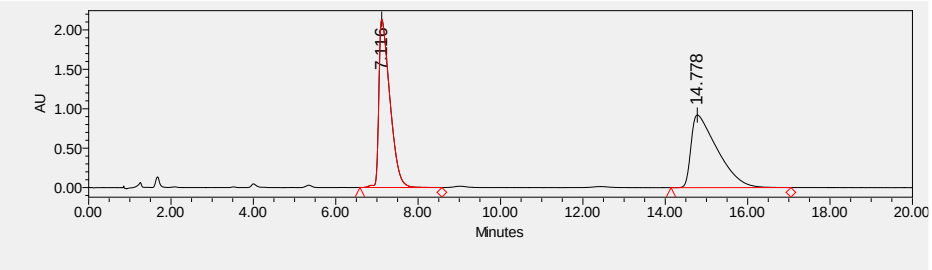


	Retention Time	Area	% Area
1	10.113	2018927	9.04
2	27.934	20314156	90.96

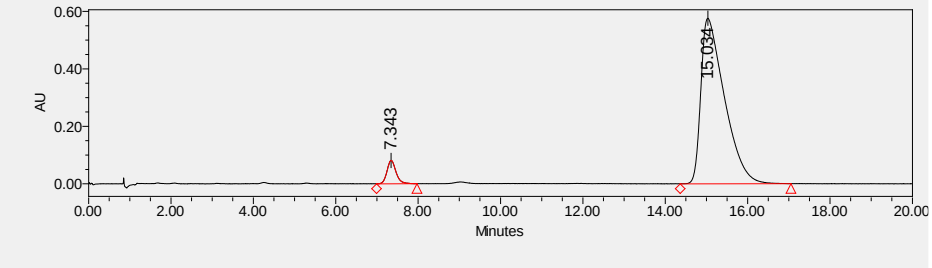
(*S*)-*N*-(5-benzyl-4-oxo-1,3-di-p-tolylhexahydropyrimidin-5-yl)-4-methylbenzamide(3dd)



33.8 mg, 67% yield; white solid, melting point: 67.9-72.4 °C, $[\alpha]^{27}_{D}$ = -44.7 (*c* = 0.47, CH_2Cl_2).
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 7.34 min, t_{major} = 15.03 min. er = 95:5.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 (d, *J* = 8.2 Hz, 2H), 7.33 – 7.14 (m, 11H), 7.10 (d, *J* = 8.2 Hz, 2H), 6.98 – 6.89 (m, 3H), 4.97 (d, *J* = 10.5 Hz, 1H), 4.82 (dd, *J* = 10.4, 1.6 Hz, 1H), 4.39 (dd, *J* = 13.4, 1.6 Hz, 1H), 4.05 (d, *J* = 13.3 Hz, 1H), 3.50 (q, *J* = 13.5 Hz, 2H), 2.37 (s, 6H), 2.28 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.2, 167.1, 145.5, 142.1, 138.0, 137.5, 135.6, 131.6, 130.8, 130.7, 130.1, 129.2, 128.6, 127.3, 127.0, 126.1, 117.3 , 68.9, 60.0, 53.5, 41.7, 21.5, 21.2, 20.6.
IR (neat): ν (cm^{-1}) 3388, 3029, 2922, 1651, 1515, 1486.
HRMS (ESI-TOF) calcd for $C_{33}H_{34}N_3O_2^+$ ([M]+H$^+$) = 504.2646, found 504.2638.

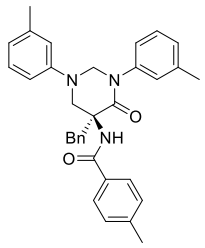


	Retention Time	Area	% Area
1	7.116	39676836	49.31
2	14.778	40788216	50.69



	Retention Time	Area	% Area
1	7.343	1239674	5.14
2	15.034	22871848	94.86

(*S*)-*N*-(5-benzyl-4-oxo-1,3-di-m-tolylhexahydropyrimidin-5-yl)-4-methylbenzamide(3ed)



28.7 mg, 57% yield; white solid, melting point: 49.7-54.9 °C, $[\alpha]^{25}_{D}$ = -35.8 (*c* = 0.61, CH_2Cl_2).

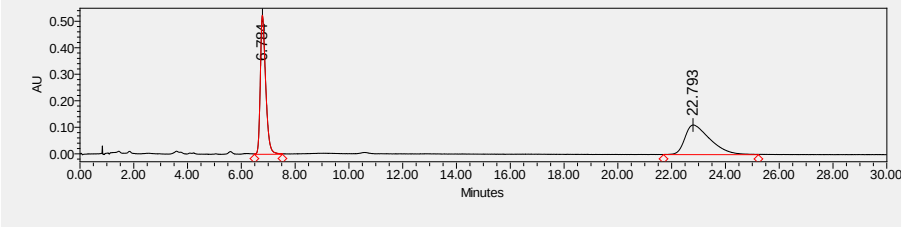
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 6.88 min, t_{major} = 23.57 min. er = 91:9.

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.59 (d, *J* = 8.2 Hz, 2H), 7.39 – 7.27 (m, 4H), 7.25 – 7.10 (m, 8H), 7.01 (s, 1H), 6.89 – 6.74 (m, 3H), 5.03 (d, *J* = 10.5 Hz, 1H), 4.90 (dd, *J* = 10.5, 1.5 Hz, 1H), 4.51 (dd, *J* = 13.3, 1.6 Hz, 1H), 4.07 (d, *J* = 13.3 Hz, 1H), 3.53 (q, *J* = 13.6 Hz, 2H), 2.40 (d, 6H), 2.33 (s, 3H).

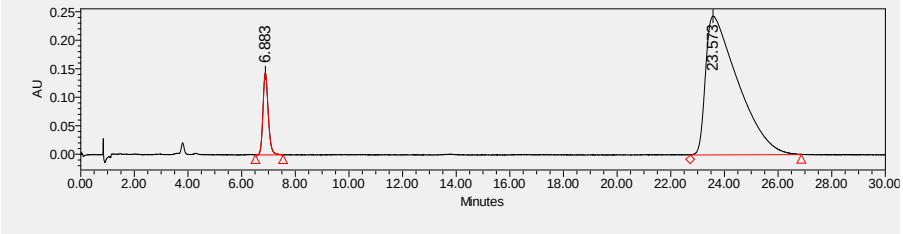
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.1, 167.1, 147.7, 142.2, 140.5, 139.5, 139.4, 135.6, 131.6, 130.6, 129.4, 129.3, 129.2, 128.6, 128.5, 127.4, 127.0, 127.0, 123.4, 122.0, 117.7, 113.9, 68.3, 60.1, 53.1, 41.6, 21.8, 21.5.

IR (neat): ν (cm^{-1}) 3416, 3031, 2922, 1815, 1651, 1511.

HRMS (ESI-TOF) calcd for $C_{33}H_{34}N_3O_2^+$ ([M]+H$^+$) = 504.2646, found 504.2643.

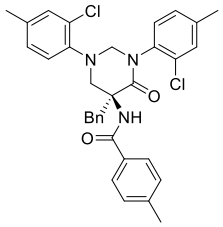


	Retention Time	Area	% Area
1	6.784	7234704	49.89
2	22.793	7266379	50.11



	Retention Time	Area	% Area
1	6.883	1960563	8.74
2	23.573	20478250	91.26

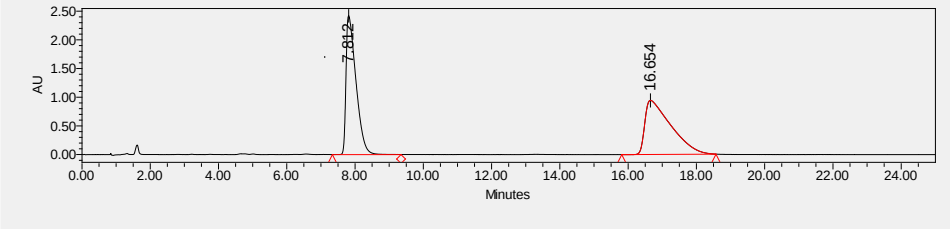
(*S*)-*N*-(5-benzyl-1,3-bis(2-chloro-4-methylphenyl)-4-oxohexahydropyrimidin-5-yl)-4-methylbenzamide(3fd)



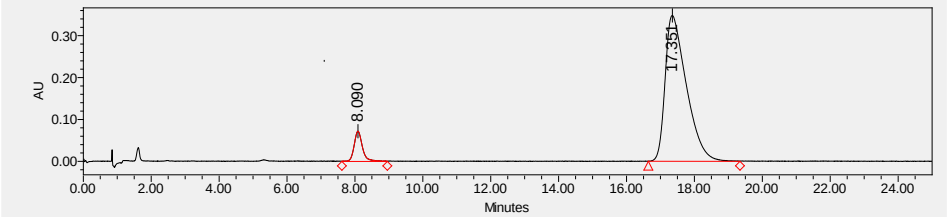
45.8 mg, 80% yield; white solid, melting point: 71.8-75.9 °C, $[\alpha]^{27}_{D}$ = -40.3 (*c* = 0.67, CH_2Cl_2).
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 8.09 min, t_{major} = 17.35 min. er = 92:8.
^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.66 – 7.47 (m, 2H), 7.43 – 7.28 (m, 5H), 7.25 – 7.08 (m, 6H), 7.05 – 6.86 (m, 2H), 6.86 – 6.77 (m, 1H), 4.99 (d, *J* = 10.5 Hz, 1H), 4.79 (dd, *J* = 10.5, 1.7 Hz, 1H), 4.31 (dd, *J* = 13.4, 1.7 Hz, 1H), 4.03 (d, *J* = 13.4 Hz, 1H), 3.46 (dd, *J* = 19.7, 13.6 Hz, 2H), 2.52 – 2.25 (m, 9H).
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.2, 167.1, 146.7, 142.3, 139.1, 135.7, 135.2, 134.9, 131.7, 131.6, 131.3, 130.6, 129.3, 128.8, 127.6, 127.0, 126.9, 124.7, 117.9, 115.6 , 68.3, 59.8, 53.1, 41.9, 21.5, 19.8, 19.2.

IR (neat): ν (cm^{-1}) 3345, 3030, 2923, 2859,1651, 1494.

HRMS (ESI-TOF) calcd for $C_{33}H_{32}N_3O_2Cl_2^+$ ([M]+H$^+$) = 572.1866, 574.1837, 576.1807, found 572.1859, 574.1835, 576.1812.

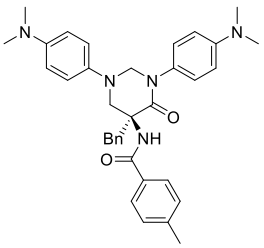


	Retention Time	Area	% Area
1	7.812	48863247	49.52
2	16.654	49806419	50.48



	Retention Time	Area	% Area
1	8.090	1226374	7.52
2	17.351	15088653	92.48

(*S*)-*N*-(5-benzyl-1,3-bis(4-(dimethylamino)phenyl)-4-oxohexahydropyrimidin-5-yl)-4-methylbenzamide(3gd)



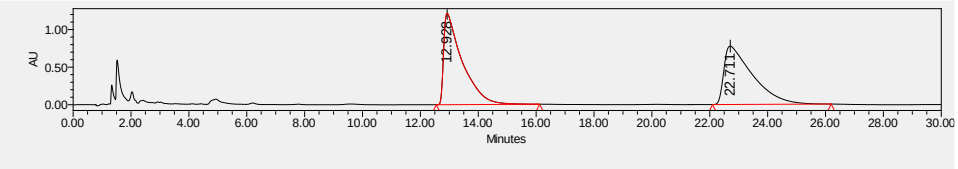
21.4 mg, 38% yield; white solid, melting point: 77.5-81.6 °C, $[\alpha]^{26}_{D}$ = -37.7 (*c* = 0.42, CH_2Cl_2).
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{minor} = 13.75 min, t_{major} = 23.58 min. er = 90:10.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.2 Hz, 2H), 7.34 – 7.26 (m, 3H), 7.25 – 7.14 (m, 6H), 7.03 – 6.96 (m, 3H), 6.79 – 6.70 (m, 4H), 4.85 (d, *J* = 10.2 Hz, 1H), 4.70 (dd, *J* = 10.2, 1.5 Hz, 1H), 4.27 (dd, *J* = 13.2, 1.5 Hz, 1H), 4.01 (d, *J* = 13.1 Hz, 1H), 3.58 (dd, *J* = 30.2, 13.5 Hz, 2H), 2.97 (s, 6H), 2.89 (s, 6H), 2.38 (s, 3H).

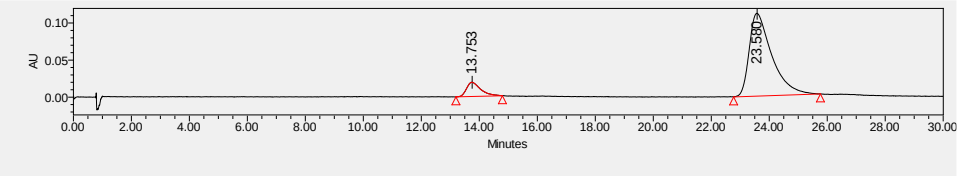
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 167.0, 149.8, 146.5, 142.0, 139.1, 136.0, 131.8, 130.8, 129.6, 129.2, 128.5, 127.2, 127.1, 119.7, 114.3, 113.0, 70.4, 60.1, 54.4, 42.0, 41.4, 40.8, 21.5.

IR (neat): ν (cm^{-1}) 3385, 2853, 2798, 1650, 1518.

HRMS (ESI-TOF) calcd for $C_{35}H_{40}N_5O_2^+$ ([M]+H$^+$) = 562.3177, found 562.3173.

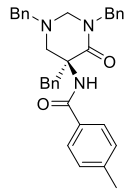


	Retention Time	Area	% Area
1	12.928	53861782	50.24
2	22.711	53353450	49.76



	Retention Time	Area	% Area
1	13.753	650881	9.73
2	23.580	6037897	90.27

(*S*)-4-methyl-*N*-(1,3,5-tribenzyl-4-oxohexahydropyrimidin-5-yl)benzamide(3hd)



38.8mg, 77% yield; white solid, melting point: 60.5-62.8 °C, $[\alpha]^{26}_D$ = -47.5 (*c* = 0.81, CH_2Cl_2).

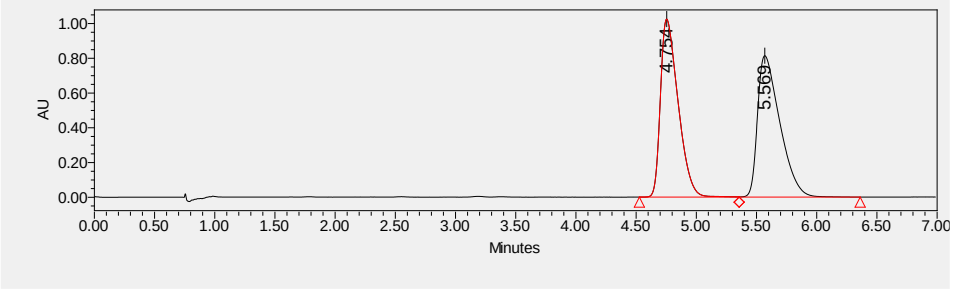
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{major} = 4.78 min, t_{minor} = 5.70 min. er = 90:10.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 7.61 – 7.48 (m, 2H), 7.43 – 7.27 (m, 10H), 7.25 – 7.13 (m, 5H), 6.94 – 6.84 (m, 2H), 6.68 (s, 1H), 4.86 (d, *J* = 15.1 Hz, 1H), 4.51 (d, *J* = 15.1 Hz, 1H), 4.01 (dd, *J* = 8.6, 1.8 Hz, 1H), 3.95 (d, *J* = 8.7 Hz, 1H), 3.67 (d, *J* = 12.8 Hz, 1H), 3.57 (d, *J* = 13.3 Hz, 1H), 3.50 (d, *J* = 12.8 Hz, 1H), 3.34 (dd, *J* = 11.7, 1.8 Hz, 1H), 3.30 (d, *J* = 13.4 Hz, 1H), 3.04 (d, *J* = 11.8 Hz, 1H), 2.37 (s, 3H).

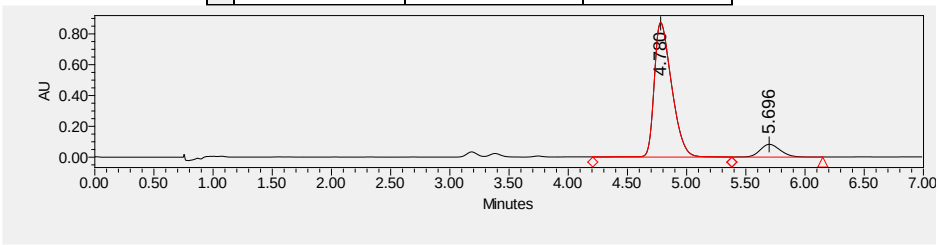
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 170.1, 166.5, 142.1, 136.9, 136.6, 135.6, 131.4, 130.5, 129.5, 129.2, 128.7, 128.7, 128.6, 128.0, 127.7, 127.5, 127.2, 127.0, 68.8, 58.8, 54.8, 48.9, 41., 21.5.

IR (neat): ν (cm^{-1}) 3435, 3030, 2921, 2812, 1647, 1486.

HRMS (ESI-TOF) calcd for $C_{33}H_{34}N_3O_2^+$ ([M]+H$^+$) = 504.2646, found 504.2641.

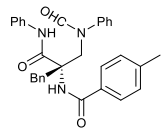


	Retention Time	Area	% Area
1	4.754	10421475	49.94
2	5.569	10447815	50.06



	Retention Time	Area	% Area
1	4.780	8906945	90.07
2	5.696	981497	9.93

(*S*)-*N*-(2-benzyl-1-oxo-1-(phenylamino)-3-(*N*-phenylformamido)propan-2-yl)-4-methylbenzamide (8ad)



50.5 mg, >99% yield; white solid, melting point: 93.6-96.9 °C, $[\alpha]^{25}_{D}$ = -22.0 (*c* = 0.53, CH_2Cl_2).

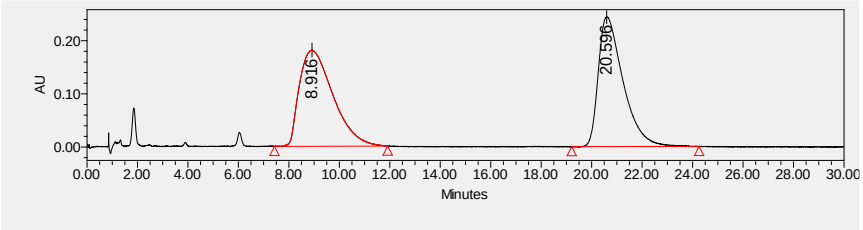
Dissolved in MeOH for SFC; SFC (Daicel chiralcel OX-3, CO_2/EtOH = 80/20, flow rate = 1.5 mL/min, λ = 254 nm) retention time: t_{major} = 8.64 min, t_{minor} = 20.80 min. er = 94:6.

^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 8.84 (s, 1H), 8.22 (s, 1H), 7.55 (s, 1H), 7.43 (d, *J* = 7.9 Hz, 2H), 7.33 (t, *J* = 7.8 Hz, 2H), 7.24 (d, *J* = 6.5 Hz, 2H), 7.21 – 7.01 (m, 12H), 5.10 (d, *J* = 15.1 Hz, 1H), 4.86 (d, *J* = 15.1 Hz, 1H), 3.87 (d, *J* = 13.7 Hz, 1H), 3.40 (d, *J* = 13.7 Hz, 1H), 2.34 (s, 3H).

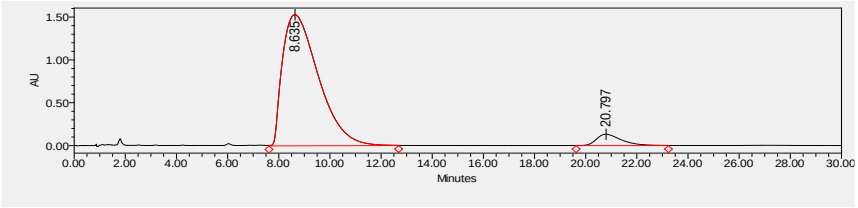
^{13}C NMR (101 MHz, $CDCl_3$) *δ* 168.8, 167.7, 164.7, 142.0, 140.3, 137.2, 134.7, 131.8, 130.2, 129.6, 129.0, 128.9, 128.3, 127.5, 127.3, 126.8, 124.9, 124.8, 120.8, 67.1, 46.3, 39.8, 21.5.

IR (neat): *ν* (cm^{-1}) 3318, 3060, 3032, 2927, 1651, 1598, 1490.

HRMS (ESI-TOF) calcd for $C_{24}H_{23}N_3O_2^+$ ([M]-$PhCH_2$+H^+) = 399.1577, found 399.1567.

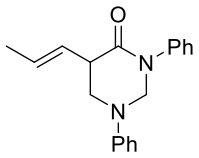


	Retention Time	Area	% Area
1	8.916	16714785	50.14
2	20.596	16620593	49.86



	Retention Time	Area	% Area
1	8.635	149172062	94.27
2	20.797	9060010	5.73

(*E*)-1,3-Diphenyl-5-(prop-1-en-1-yl)tetrahydropyrimidin-4(1H)-one (7aa)



25.7 mg, 88% yield; light yellow oil; $[\alpha]^{24}_{D}$ = -30.5 (*c* = 0.42, CH_2Cl_2).

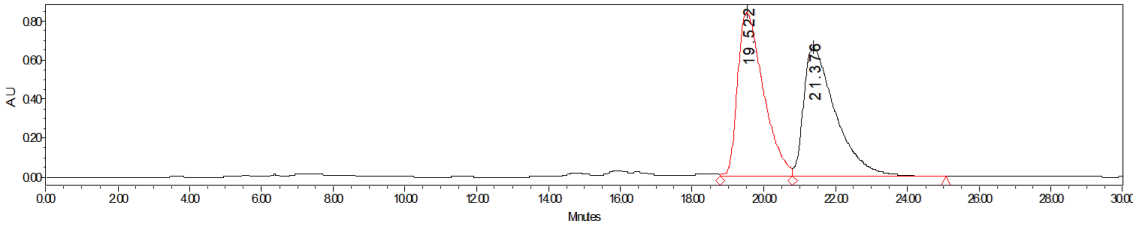
Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel ODH, hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{minor} = 20.19 min, t_{major} = 21.61 min, er = 96:4.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42 (m, 2H), 7.31 (m, 5H), 6.99 – 6.90 (m, 3H), 5.75 – 5.56 (m, 2H), 5.14 (d, *J* = 11.6 Hz, 1H), 5.04 (dd, *J* = 11.6, 2.0 Hz, 1H), 3.97 (m, 1H), 3.59 (m, 1H), 3.40 (m, 1H), 1.74 (d, *J* = 4.0 Hz, 3H).

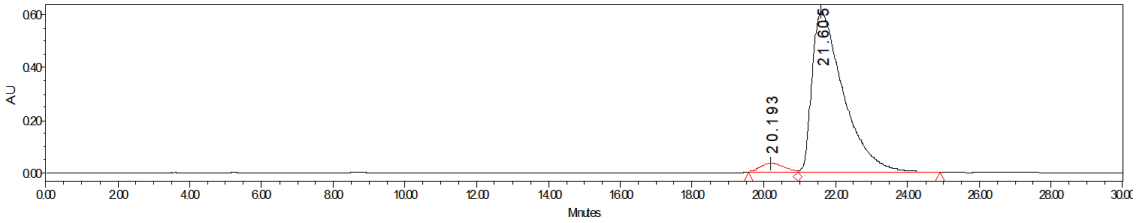
^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.8, 147.2, 140.9, 129.6, 129.4, 129.3, 127.0, 126.3, 125.8, 120.6, 116.0, 66.4, 52.4, 43.45, 18.2.

IR (neat): ν (cm^{-1}) 2914, 236, 1658, 1597, 1497, 1429, 1380, 1216, 1117, 1073, 965.

HRMS (ESI-TOF) calcd for $C_{19}H_{20}N_2NaO^+$ ([M]+Na^+) = 315.1468, found 315.1468.

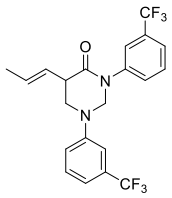


Peak	Retention Time	Area	% Area
1	19.522	42250064	50.62
2	21.376	41216792	49.38



Peak	Retention Time	Area	% Area
1	20.193	1514327	3.93
2	21.605	36990405	96.07

(*E*)-5-(Prop-1-en-1-yl)-1,3-bis(3-(trifluoromethyl)phenyl)tetrahydropyrimidin-4(1H)-one (7ba)



21.4 mg, 51% yield; light yellow oil; $[\alpha]^{24}_{D}$ = -25.9 (*c* = 2.17, CH_2Cl_2).

Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel IH, hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{minor} = 7.21 min, t_{major} = 11.47 min, er = 98:2.

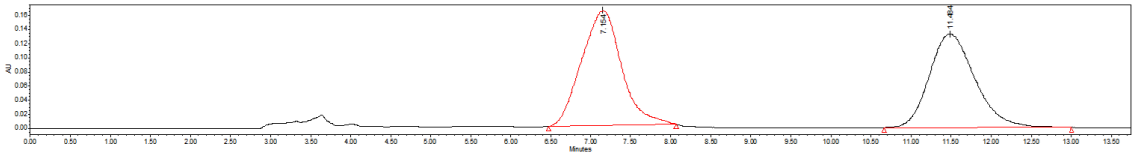
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.63 – 7.34 (m, 5H), 7.18 (d, *J* = 7.6 Hz, 1H), 7.12 – 6.99 (m, 2H), 5.83 – 5.51 (m, 2H), 5.21 (d, *J* = 12.0 Hz, 1H), 5.10 (d, *J* = 12.0 Hz, 1H), 3.97 (m, 1H), 3.63 (m, 1H), 3.51 – 3.36 (m, 1H), 1.75 (d, *J* = 6.0 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.8, 147.3, 141.1, 131.9 (q, $J_{C\text{-}F}$ = 58.58), 131.9 (d, $J_{C\text{-}F}$ = 7.07), 130.3 , 130.2, 129.9, 129.1, 125.4, 125.0, 123,8 (q, $J_{C\text{-}F}$ = 4.04), 122.5 (q, J_{CF} = 4.04), 122.3 , 118.5 , 117.0 (q, $J_{C\text{-}F}$ = 4.04) , 111.9 (q, $J_{C\text{-}F}$ = 4.04) , 65.4, 51.8, 43.9, 18.1.

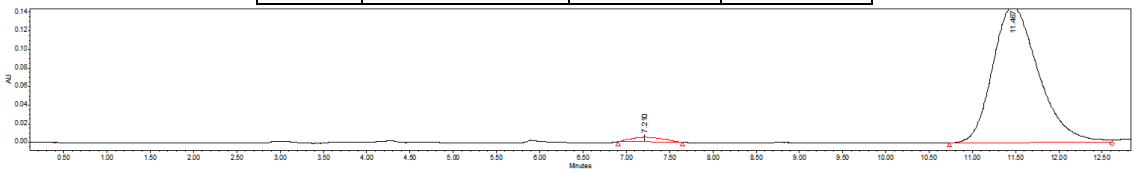
^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.68.

IR (neat): ν (cm^{-1}) 2919, 2336, 1663, 1592, 1479, 1438, 1382, 1313, 1264, 967.

HRMS (ESI-TOF) calcd for $C_{21}H_{18}F_6N_2NaO^+$ ([M]+Na^+) = 451.1216, found 451.1219.

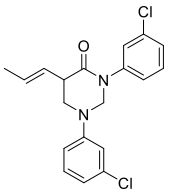


Peak	Retention Time	Area	% Area
1	7.154	5653073	51.82
2	11.484	5256031	48.18



Peak	Retention Time	Area	% Area
1	7.210	121330	2.23
2	11.467	5317243	97.77

(*E*)-1,3-Bis(3-chlorophenyl)-5-(prop-1-en-1-yl)tetrahydropyrimidin-4(1H)-one (7ca)



31.0 mg, 86% yield; colorless oil; $[\alpha]^{24}_{D}$ = -8.8 (*c* = 0.63, CH_2Cl_2).

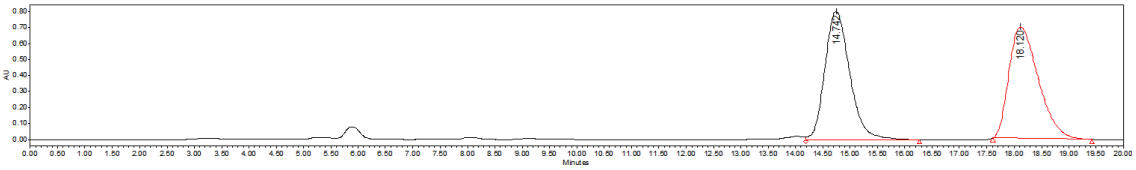
Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel ADH, hexane/*i*PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{major} = 14.91 min, t_{minor} = 18.54 min, er = 96:4.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 – 7.25 (m, 3H), 7.20 (q, *J* = 8.4 Hz, 2H), 6.94 – 6.82 (m, 2H), 6.75 (dd, *J* = 8.4, 2.0 Hz, 1H), 5.75 – 5.57 (m, 2H), 5.10 (d, *J* = 11.6 Hz, 1H), 5.04 – 4.97 (m, 1H), 3.89 (dd, *J* = 12.8, 6.4 Hz, 1H), 3.55 (dd, *J* = 12.8, 10.0 Hz, 1H), 3.46 – 3.34 (m, 1H), 1.75 (d, *J* = 5.6 Hz, 3H).

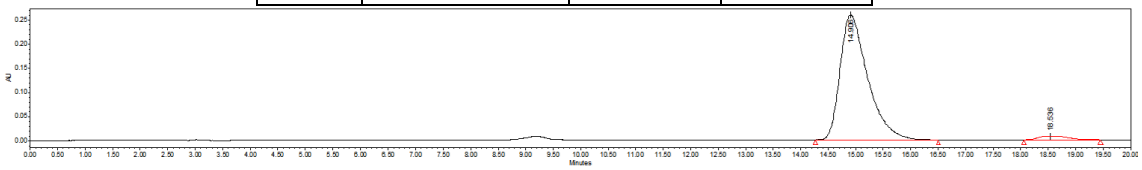
^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.8, 148.2, 141.8, 135.5, 134.8, 130.6, 130.3, 130.01, 127.3, 126.1, 125.6, 123.9, 120.5, 115.7, 113.5, 65.6, 51.8, 43.8, 18.2.

IR (neat): ν (cm^{-1}) 2916, 2367, 1663, 1591, 1478, 1438, 1381, 1256, 1168, 966, 770.

HRMS (ESI-TOF) calcd for $C_{19}H_{19}Cl_2N_2O^+$ ([M]+H$^+$) = 448.9859, 450.9838, 452.9818, found 448.9861, 450.9838, 452.9816.

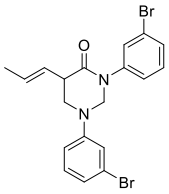


Peak	Retention Time	Area	% Area
1	14.742	25131481	49.16
2	18.120	25994315	50.84



Peak	Retention Time	Area	% Area
1	14.906	9079777	96.41
2	18.536	338277	3.59

(*E*)-1,3-Bis(3-bromophenyl)-5-(prop-1-en-1-yl)tetrahydropyrimidin-4(1H)-one (7da)



25.7 mg, 57% yield; pale yellow oil; $[\alpha]^{24}_{D}$ = -5.1 (*c* = 1.20, CH_2Cl_2).

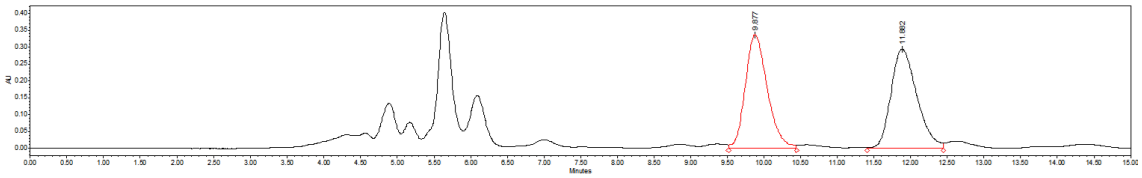
Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel ADH, hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{major} = 9.93 min, t_{minor} = 11.97 min, er = 96:4.

^{1}H NMR (400 MHz, Chloroform-*d*) δ7.48 – 7.40 (m, 2H), 7.30 – 7.20 (m, 2H), 7.14 (t, *J* = 8.0 Hz, 1H), 7.07 – 6.99 (m, 2H), 6.79 (dd, *J* = 8.4, 2.0 Hz, 1H), 5.74 – 5.57 (m, 2H), 5.11 – 4.95 (m, 2H), 3.88 (dd, *J* = 12.8, 6.4 Hz, 1H), 3.54 (dd, *J* = 12.8, 10.4 Hz, 1H), 3.44 – 3.36 (m, 1H), 1.74 (d, *J* = 5.6 Hz, 3H).

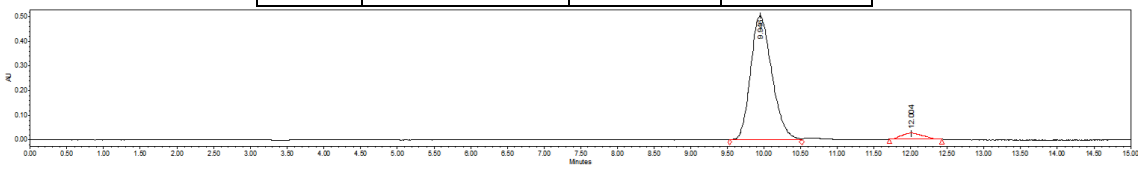
^{13}C NMR (101 MHz, Chloroform-*d*) δ169.7, 148.3, 141.9, 130.9, 130.53 , 130.2, 130.0, 128.9, 125.6, 124.4, 123.6, 123.4, 122.6, 118.6, 114.0, 65.6, 51.7, 43.8, 18.2.

IR (neat): ν (cm^{-1}) 2915, 2361, 1660, 1588, 1476, 1436, 1352, 1217, 1070, 965, 766.

HRMS (ESI-TOF) calcd for $C_{19}H_{19}Br_2N_2O^+$ ([M]+H$^+$) = 448.9859, 450.9838, 452.9818, found 448.9861, 450.9838, 452.9816.

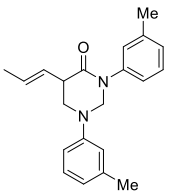


Peak	Retention Time	Area	% Area
1	9.877	7039592	49.40
2	11.882	7209412	50.60



Peak	Retention Time	Area	% Area
1	9.976	13406726	95.56
2	12.067	623212	4.44

(*E*)-5-(Prop-1-en-1-yl)-1,3-di-m-tolyltetrahydropyrimidin-4(1H)-one (7ea)



19.5 mg, 61% yield; pale yellow oil; $[\alpha]^{24}_{D}$ = -26.6 (*c* = 0.66, CH_2Cl_2).

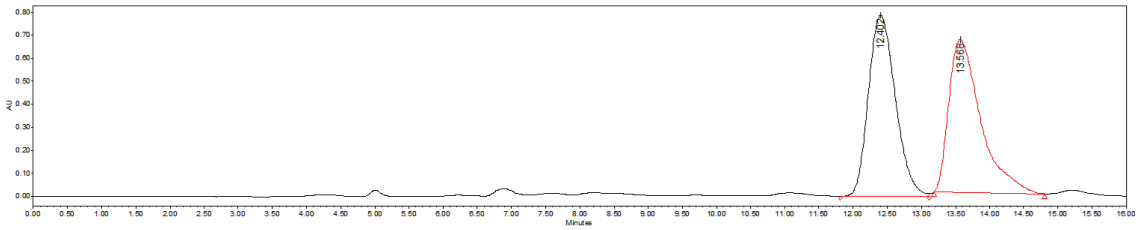
Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel ADH, hexane/*i*PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{major} = 12.39 min, t_{minor} = 13.65 min, er = 92:8.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.74 (t, *J* = 7.6 Hz, 1H), 7.66 – 7.59 (m, 1H), 7.57 – 7.48 (m, 3H), 7.19 (s, 3H), 6.16 – 6.04 (m, 2H), 5.52 (d, *J* = 11.6 Hz, 1H), 5.42 (dd, *J* = 11.6, 1.6 Hz, 1H), 4.37 (m, 1H), 3.99 (dd, *J* = 13.0, 10.0 Hz, 1H), 3.81 (m, 1H), 2.78 (d, *J* = 17.1 Hz, 6H), 2.17 (d, *J* = 3.8 Hz, 3H).

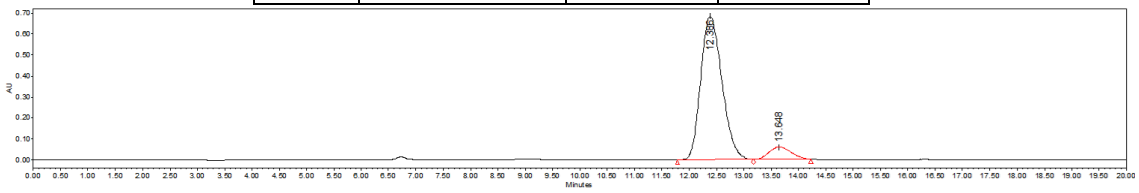
^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.5, 147.9, 141.4, 140.0, 139.8, 130.0, 129.9, 129.7, 128.5, 127.4, 127.0, 123.5, 122.1, 117.5, 113.8, 67.3, 52.98 , 44.1, 22.4, 22.0, 18.8.

IR (neat): ν (cm^{-1}) 2361, 1659, 1603, 1491, 1441, 1261, 1159, 966, 744.

HRMS (ESI-TOF) calcd for $C_{21}H_{25}N_2O^+$ ([M]+H$^+$) = 321.1961, found 321.1961.

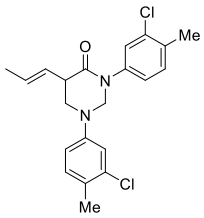


Peak	Retention Time	Area	% Area
1	12.402	21491067	49.85
2	13.568	21621080	50.15



Peak	Retention Time	Area	% Area
1	12.386	18496192	91.64
2	13.648	1688427	8.36

(*E*)-1,3-Bis(3-chloro-4-methylphenyl)-5-(prop-1-en-1-yl)tetrahydropyrimidin-4(1H)-one (7fa)



31.4 mg, 81% yield; colorless oil; $[\alpha]^{24}_{D}$ = -14.2 (*c* = 0.82, CH_2Cl_2).

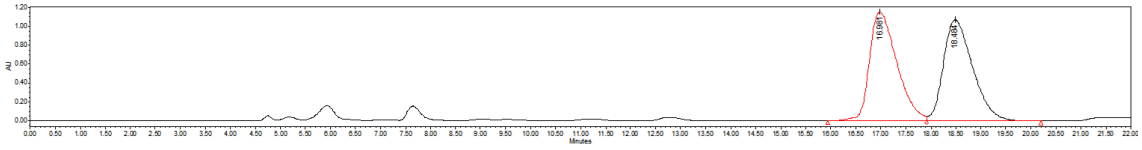
Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel ADH, hexane/*i*PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{major} = 18.55 min, t_{minor} = 17.18 min, er = 95:5.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.23 (m, 2H), 7.14 – 7.05 (m, 2H), 6.90 (d, *J* = 2.4 Hz, 1H), 6.70 (dd, *J* = 8.4, 2.4 Hz, 1H), 5.73 – 5.52 (m, 2H), 5.03 (d, *J* = 11.6 Hz, 1H), 4.91 (dd, *J* = 11.6, 1.6 Hz, 1H), 3.86 (m, 1H), 3.51 (dd, *J* = 13.0, 10.4 Hz, 1H), 3.34 (dt, *J* = 10.4, 6.0 Hz, 1H), 2.32 (d, *J* = 30.0 Hz, 6H), 1.72 (d, *J* = 5.2 Hz, 3H) .

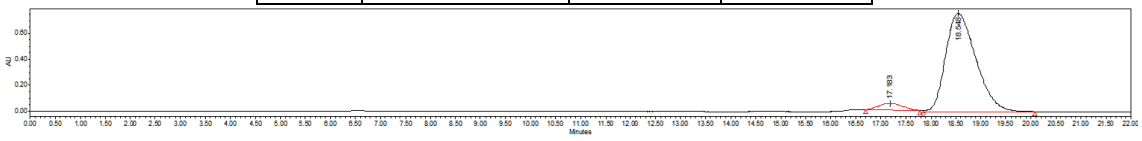
^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.7, 146.2, 139.4, 135.3, 135.1, 134.7, 131.6, 131.4, 129.8, 128.1, 126.4, 125.9, 124.2, 116.8, 114.4, 66.5, 52.4, 43.5, 19.7, 19.0, 18.2.

IR (neat): ν (cm^{-1}) 2361, 1660, 1609, 1563, 1497, 1433, 1381, 1222, 1004, 737.

HRMS (ESI-TOF) calcd for $C_{21}H_{23}Cl_2N_2O^+$ ([M]+H^+) = 389.1182, 391.1152, 393.1123, found 389.1183, 391.1152, 393.1119.

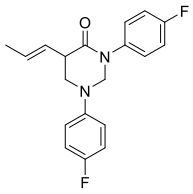


Peak	Retention Time	Area	% Area
1	16.981	43686545	50.31
2	18.484	43144762	49.69



Peak	Retention Time	Area	% Area
1	17.183	1631113	4.89
2	18.548	31738654	95.11

(*E*)-1,3-Bis(4-fluorophenyl)-5-(prop-1-en-1-yl)tetrahydropyrimidin-4(1H)-one (7ga)



27.2 mg, 83% yield; yellow oil; $[\alpha]^{24}_{D}$ = -49.5 (*c* = 1.09, CH_2Cl_2).

Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel ADH, hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{major} = 14.82 min, t_{minor} = 10.63 min, er = 97:3.

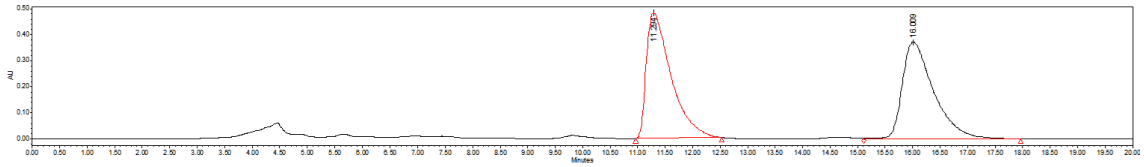
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.25 (dd, *J* = 8.4, 4.8 Hz, 2H), 7.10 (t, *J* = 8.4 Hz, 2H), 7.00 (t, *J* = 8.8 Hz, 2H), 6.90 (dd, *J* = 9.2, 4.4 Hz, 2H), 5.73 – 5.53 (m, 2H), 5.09 – 4.87 (m, 2H), 3.87 (dd, *J* = 13.2, 6.4 Hz, 1H), 3.54 (dd, *J* = 13.2, 10.4 Hz, 1H), 3.34 (dt, *J* = 10.0, 6.0 Hz, 1H), 1.72 (d, *J* = 4.4 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.8, 143.8 (d, J_{CF} = 2.02 Hz), 136.7 (d, J_{CF} = 2.02 Hz), 129.6, 127.7 (d, J_{CF} = 9.09 Hz), 126.1, 118.2 (d, J_{CF} = 7.07 Hz), 116.3(d, J_{CF} = 5.05 Hz), 116.1(d, J_{CF} = 4.04 Hz), 76.8 , 67.7, 53.4, 43.4, 18.2.

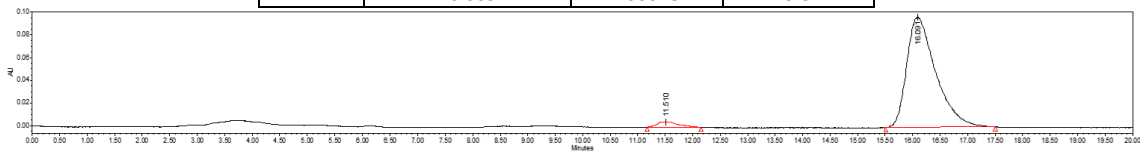
^{19}F NMR (376 MHz, Chloroform-*d*) δ -114.51, -122.76.

IR (neat): ν (cm^{-1}) 2361, 1658, 1601, 1508, 1258, 966, 752.

HRMS (ESI-TOF) calcd for $C_{19}H_{19}F_2N_2O^+$ ([M]+H^+) = 329.1460, found 329.1460.

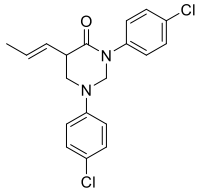


Peak	Retention Time	Area	% Area
1	11.294	14988032	50.66
2	16.009	14600182	49.34



Peak	Retention Time	Area	% Area
1	11.510	120742	3.30
2	16.091	3540428	96.70

(*E*)-1,3-Bis(4-chlorophenyl)-5-(prop-1-en-1-yl)tetrahydropyrimidin-4(1H)-one (7ha)



28.2 mg, 78% yield; colorless oil; $[\alpha]^{24}_{D}$ = -11.43 (*c* = 0.29, CH_2Cl_2).

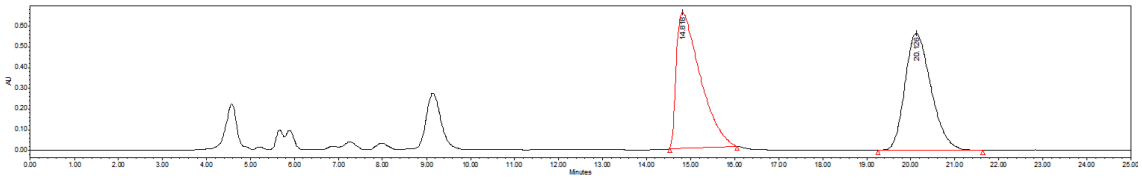
Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel ADH, hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{major} = 20.16 min, t_{minor} = 15.17 min, er = 96:4.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 (d, *J* = 8.4 Hz, 2H), 7.23 (dd, *J* = 13.2, 8.8 Hz, 4H), 6.82 (d, *J* = 8.8 Hz, 2H), 5.75 – 5.54 (m, 2H), 5.09 (d, *J* = 11.6 Hz, 1H), 4.97 (d, *J* = 11.6 Hz, 1H), 3.90 (dd, *J* = 13.2, 6.4 Hz, 1H), 3.55 (dd, *J* = 13.2, 10.4 Hz, 1H), 3.41 – 3.32 (m, 1H), 1.73 (d, *J* = 5.2 Hz, 3H).

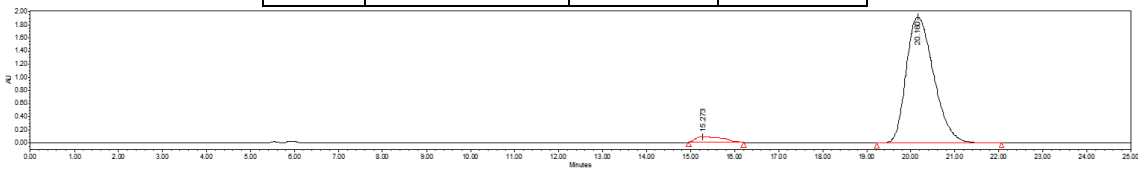
^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.7, 145.7, 139.2, 132.6, 129.9 , 129.6, 129.5, 127.0, 125.8, 117.2, 66.2 , 52.4, 43.6, 18.2.

IR (neat): ν (cm^{-1}) 2361, 1661, 1595, 1493, 1437, 1264, 1009, 739.

HRMS (ESI-TOF) calcd for $C_{19}H_{19}Cl_2N_2O^+$ ([M]+H$^+$) = 361.0869, 363.0839, 365.0810, found 361.0872, 363.9840, 365.0807.

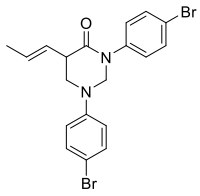


Peak	Retention Time	Area	% Area
1	15.200	10849588	50.01
2	20.359	10844629	49.99



Peak	Retention Time	Area	% Area
1	15.273	3892297	4.46
2	20.160	83360189	95.54

(*E*)-1,3-Bis(4-bromophenyl)-5-(prop-1-en-1-yl)tetrahydropyrimidin-4(1H)-one (7ia)



38.7 mg, 86% yield; yellow oil; $[\alpha]^{24}_{D}$ = -8.4 (*c* = 0.70, CH_2Cl_2).

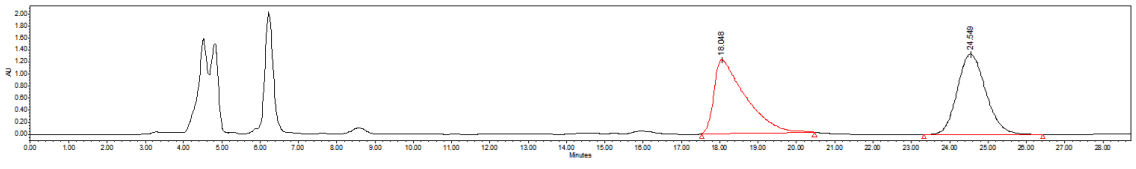
Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel ADH, hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{major} = 23.61 min, t_{minor} = 18.75 min, er = 94:6.

^{1}H NMR (400 MHz, Chloroform-*d*) δ = 7.53 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.8 Hz, 2H), 7.15 (d, *J* = 8.4 Hz, 2H), 6.76 (d, *J* = 8.8 Hz, 2H), 5.75 – 5.55 (m, 2H), 5.09 (d, *J* = 12.0 Hz, 1H), 5.00 – 4.93 (m, 1H), 3.89 (m, 1H), 3.54 (dd, *J* = 13.2, 10.4 Hz, 1H), 3.37 (dt, *J* = 10.0, 6.4 Hz, 1H), 1.73 (d, *J* = 5.6 Hz, 3H).

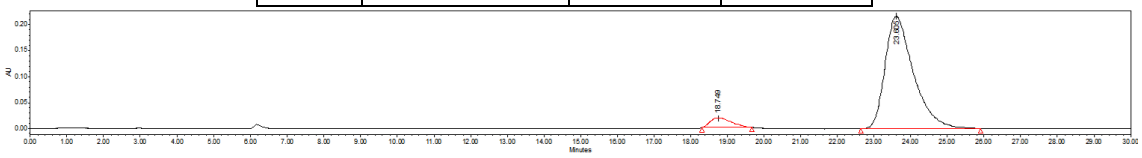
^{13}C NMR (101 MHz, Chloroform-*d*) δ = 169.7, 146.1, 139.7, 132.5, 132.4, 130.0, 127.3, 125.7, 117.4, 113.0, 65.9, 52.2, 43.6, 18.2.

IR (neat): ν (cm^{-1}) 2361, 1661, 1595, 1493, 1437, 1278, 1009, 753.

HRMS (ESI-TOF) calcd for $C_{19}H_{19}Br_2N_2O^+$ ([M]+H$^+$) = 448.9859, 450.9838, 452.9818, found 448.9865, 450.9840, 452.9816.

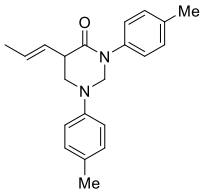


Peak	Retention Time	Area	% Area
1	18.048	69085489	50.29
2	24.549	68275791	49.71



Peak	Retention Time	Area	% Area
1	18.749	764290	6.16
2	23.605	11633203	93.84

(*E*)-5-(Prop-1-en-1-yl)-1,3-di-p-tolyltetrahydropyrimidin-4(1H)-one (7ja)



25.9 mg, 81% yield; colorless oil; $[\alpha]^{24}_D$ = -19.8 (*c* = 0.86, CH_2Cl_2).

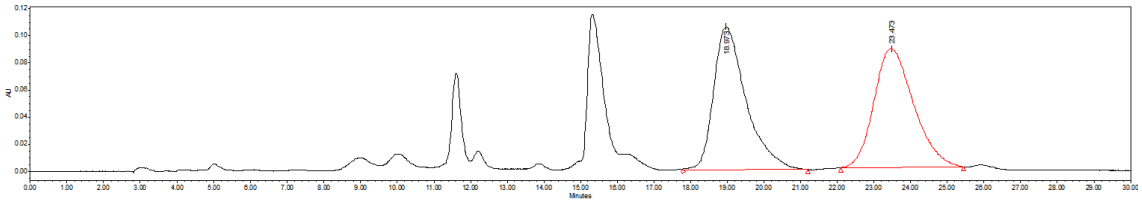
Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel ODH, hexane/*i*PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{major} = 18.68 min, t_{minor} = 23.69 min, er = 95:5.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.26 – 7.15 (m, 4H), 7.11 (d, *J* = 6.4 Hz, 2H), 6.87 (d, *J* = 6.4 Hz, 2H), 5.65 (s, 2H), 5.09 – 4.91 (m, 2H), 3.92 (d, *J* = 11.6 Hz, 1H), 3.54 (t, *J* = 11.6 Hz, 1H), 3.36 (s, 1H), 2.33 (d, *J* = 28.0 Hz, 6H), 1.73 (s, 3H).

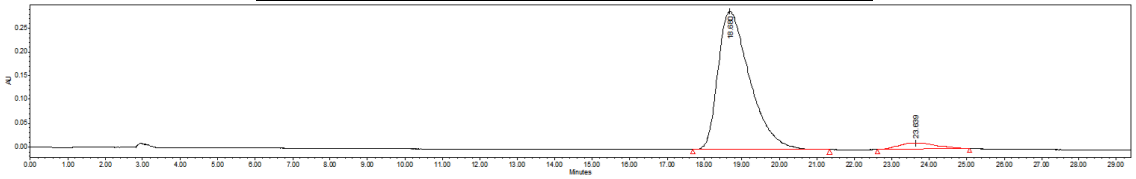
^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.8, 145.0, 136.8, 130.3, 130.1, 129.9, 129.2, 126.6, 125.8, 116.5, 67.2, 52.9, 43.3, 21.1, 20.5, 18.2.

IR (neat): ν (cm^{-1}) 2920, 1658, 1613, 1513, 1438, 1221,1002, 814.

HRMS (ESI-TOF) calcd for $C_{21}H_{25}N_2O^+$ ([M]+H^+) = 321.1961, found 321.1959.

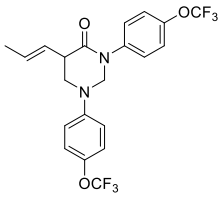


Peak	Retention Time	Area	% Area
1	18.973	6438594	49.59
2	23.473	6545091	50.41



Peak	Retention Time	Area	% Area
1	18.680	17134946	95.01
2	23.639	900255	4.99

(*E*)-5-(Prop-1-en-1-yl)-1,3-bis(4-(trifluoromethoxy)phenyl)tetrahydropyrimidin-4(1H)-one (7ka)



35.4 mg, 77% yield; yellow oil; $[\alpha]^{24}_{D}$ = -16.7 (*c* = 1.67, CH_2Cl_2).

Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel ODH, hexane/*i*PrOH = 90/10, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{major} = 9.66 min, t_{minor} = 12.01 min, er = 93:7.

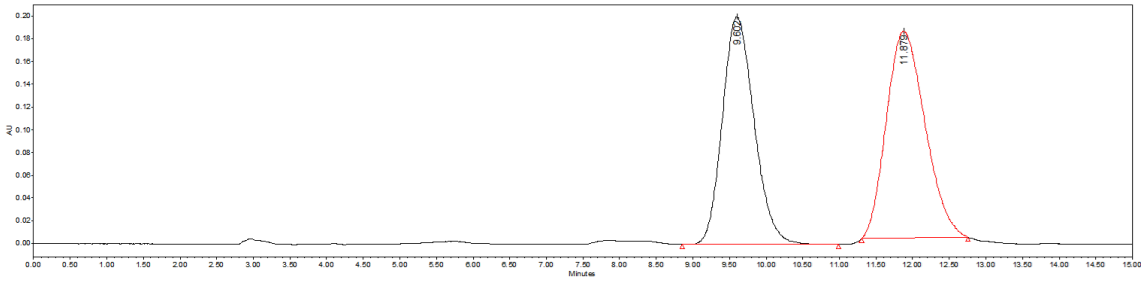
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 – 7.24 (m, 4H), 7.16 (d, *J* = 8.4 Hz, 2H), 6.89 (d, *J* = 8.8 Hz, 2H), 5.78 – 5.54 (m, 2H), 5.11 (s, 1H), 5.01 (d, *J* = 11.6 Hz, 1H), 3.92 (dd, *J* = 13.2, 6.4 Hz, 1H), 3.58 (dd, *J* = 12.8, 10.0 Hz, 1H), 3.45 – 3.35 (m, 1H), 1.74 (d, *J* = 5.6 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.8, 147.6, 145.9, 139.2, 130.0, 127.1, 125.8, 122.6, 121.9, 116.70 , 66.3, 52.4, 43.7, 18.2.

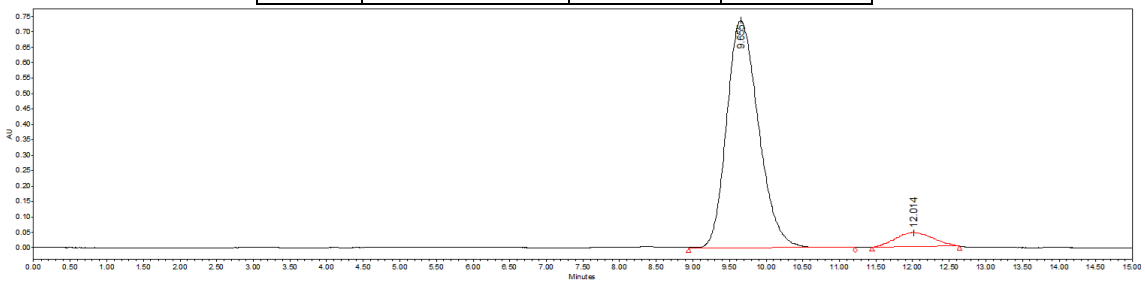
^{19}F NMR (376 MHz, Chloroform-*d*) δ -57.96, -58.33.

IR (neat): ν (cm^{-1}) 2361, 1656, 1614, 1513, 1440, 1265, 966, 736.

HRMS (ESI-TOF) calcd for $C_{21}H_{19}F_6N_2O_3^+$ ([M]+H$^+$) = 461.1294, found 461.1295.

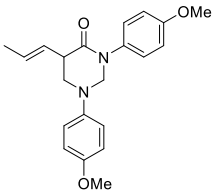


Peak	Retention Time	Area	% Area
1	9.602	6037537	48.04
2	11.879	6530334	51.96



Peak	Retention Time	Area	% Area
1	9.655	22238697	93.30
2	12.014	1595874	6.70

(*E*)-1,3-Bis(4-methoxyphenyl)-5-(prop-1-en-1-yl)tetrahydropyrimidin-4(1H)-one (7la)



17.6 mg, 50% yield; colorless oil; $[\alpha]^{24}_{D}$ = -18.1 (*c* = 1.11, CH_2Cl_2).

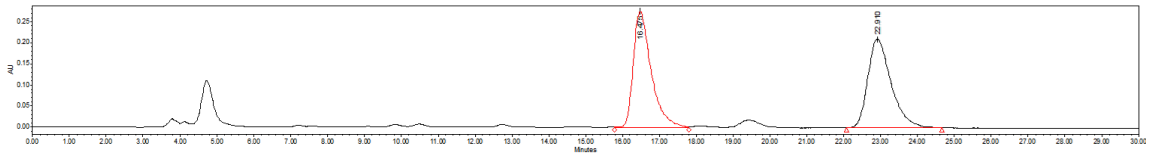
Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel IA, hexane/*i*PrOH = 70/30, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{major} = 23.30 min, t_{minor} = 16.94 min, er = 90:10.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24 – 7.17 (m, 2H), 6.94 (m, 4H), 6.89 – 6.83 (m, 2H), 5.75 – 5.51 (m, 2H), 5.01 (d, *J* = 11.6 Hz, 1H), 4.84 (dd, *J* = 11.6, 1.6 Hz, 1H), 3.88 – 3.75 (m, 7H), 3.51 (dd, *J* = 13.2, 10.0 Hz, 1H), 3.31 (dt, *J* = 10.4, 5.6 Hz, 1H), 1.72 (d, *J* = 4.0 Hz, 3H).

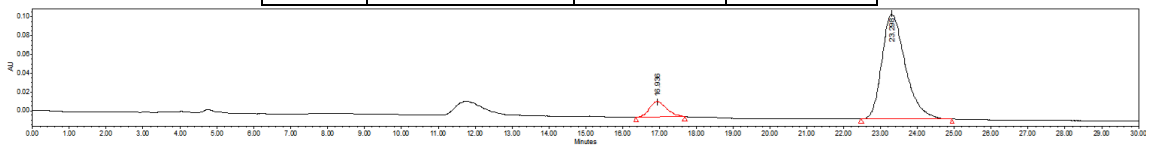
^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.8, 158.3, 154.5, 141.4, 133.6, 129.1, 127.2, 126.6, 118.7, 114.8, 114.6, 68.5, 55.5, 53.9, 43.1, 18.2.

IR (neat): ν (cm^{-1}) 2361, 1658, 1590, 1506, 1487, 1262, 1227, 745.

HRMS (ESI-TOF) calcd for $C_{21}H_{25}N_2O_3^+$ ([M]+H$^+$) = 353.1860, found 353.1858.

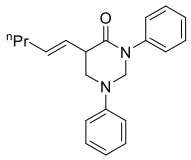


Peak	Retention Time	Area	% Area
1	16.475	9285959	50.12
2	22.910	9242540	49.88



Peak	Retention Time	Area	% Area
1	16.936	528581	9.65
2	23.298	4946728	90.35

(*E*)-5-(Pent-1-en-1-yl)-1,3-diphenyltetrahydropyrimidin-4(1H)-one (7ab)



11.9 mg, 39% yield; pale yellow oil; $[\alpha]^{24}_{D}$ = -114.6 (*c* = 0.60, CH_2Cl_2).

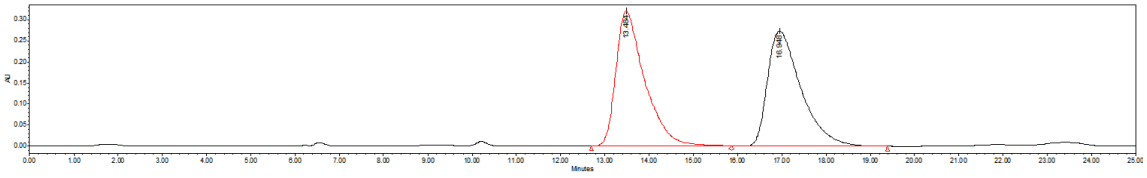
Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel ODH, hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{major} = 17.18 min, t_{minor} = 13.82 min, er = 84:16.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 – 7.37 (m, 2H), 7.34 – 7.25 (m, 5H), 6.99 – 6.89 (m, 3H), 5.73 – 5.54 (m, 2H), 5.15 (d, *J* = 11.6 Hz, 1H), 5.05 (m, 1H), 3.98 (m, 1H), 3.60 (dd, *J* = 13.2, 10.4 Hz, 1H), 3.40 (m, 1H), 2.05 (q, *J* = 7.6 Hz, 2H), 1.41 m, 2H), 0.91 (t, *J* = 7.6 Hz, 3H).

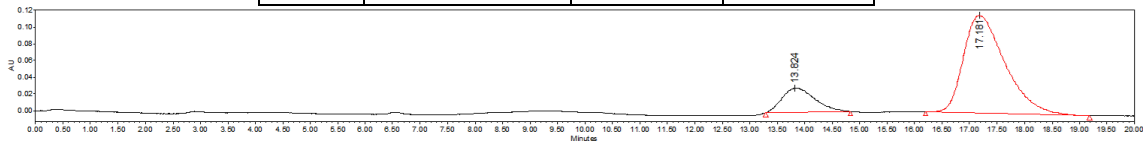
^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.9, 147.2, 134.6, 129.6, 129.3, 127.0, 125.8, 125.2, 120.7, 116.0, 66.4, 52.5, 43.5, 34.8 , 22.3, 13.7.

IR (neat): ν (cm^{-1}) 2361, 1660, 1266, 749.

HRMS (ESI-TOF) calcd for $C_{21}H_{25}N_2O^+$ ([M]+H^+) = 321.1961, found 321.1962.

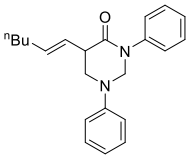


Peak	Retention Time	Area	% Area
1	13.484	14505855	50.14
2	16.948	14426743	49.86



Peak	Retention Time	Area	% Area
1	13.824	1204849	16.45
2	17.181	6117992	83.55

(*E*)-5-(Hex-1-en-1-yl)-1,3-diphenyltetrahydropyrimidin-4(1H)-one (7ac)



23.7 mg, 71% yield; pale yellow oil; $[\alpha]^{24}_{D}$ = -38.9 (*c* = 0.28, CH_2Cl_2).

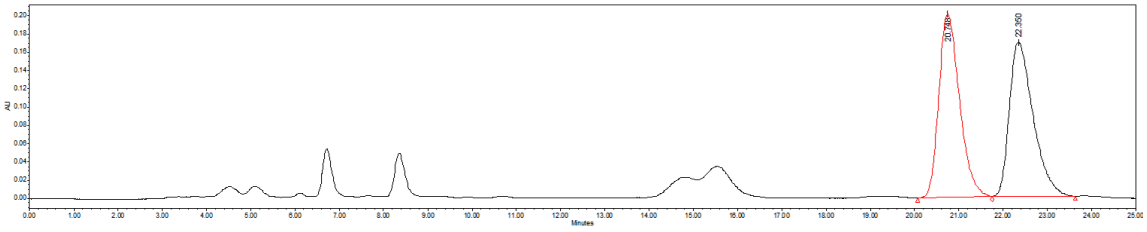
Dissolved in *i*PrOH for HPLC; HPLC (Chiralcel IE, hexane/*i*PrOH = 80/20, flow rate 1.0 mL/min, λ = 254 nm) retention time: t_{major} = 20.27 min, t_{minor} = 22.23 min, er = 85:15.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 – 7.38 (m, 2H), 7.30 (q, *J* = 7.2 Hz, 5H), 6.95 (d, *J* = 8.8 Hz, 3H), 5.72 – 5.58 (m, 2H), 5.15 (d, *J* = 11.6 Hz, 1H), 5.04 (m, 1H), 3.98 (m, 1H), 3.60 (m, 1H), 3.45 – 3.36 (m, 1H), 2.08 (m, 2H), 1.42 – 1.29 (m, 4H), 0.90 (t, *J* = 7.2 Hz, 3H).

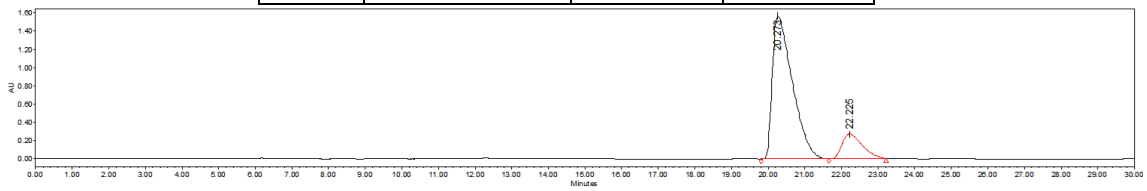
^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.8, 147.2, 140.9, 134.7, 120.6, 116.0, 66.3, 52.4, 43.4, 32.4, 31.3, 22.2, 14.0.

IR (neat): ν (cm^{-1}) 2361, 1659, 1458, 1266, 751.

HRMS (ESI-TOF) calcd fo $C_{22}H_{27}N_2O^+$ ([M]+H^+) = 335.2118, found 335.2116.



Peak	Retention Time	Area	% Area
1	20.748	6525884	50.82
2	22.350	6315460	49.18

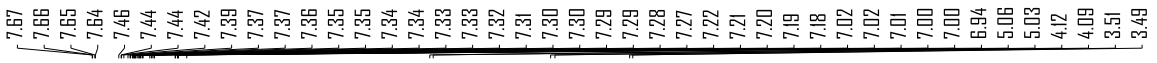


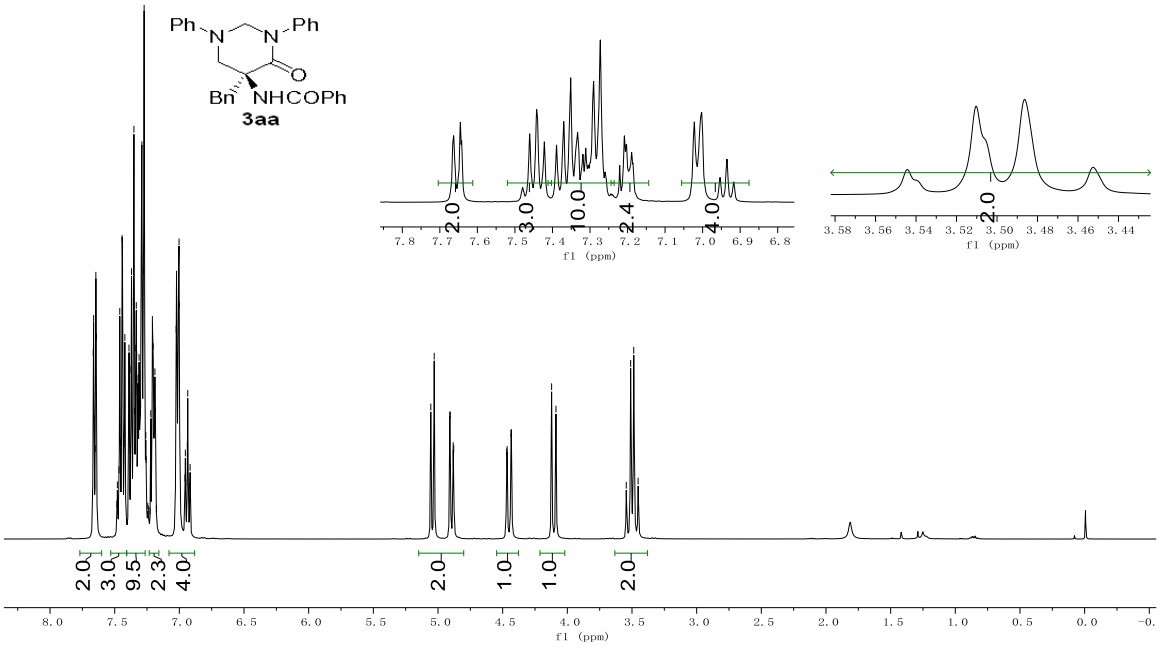
Peak	Retention Time	Area	% Area
1	20.273	59173325	85.40
2	22.225	10117239	14.60

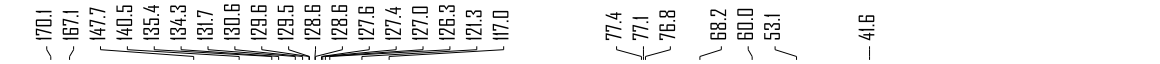
13. References

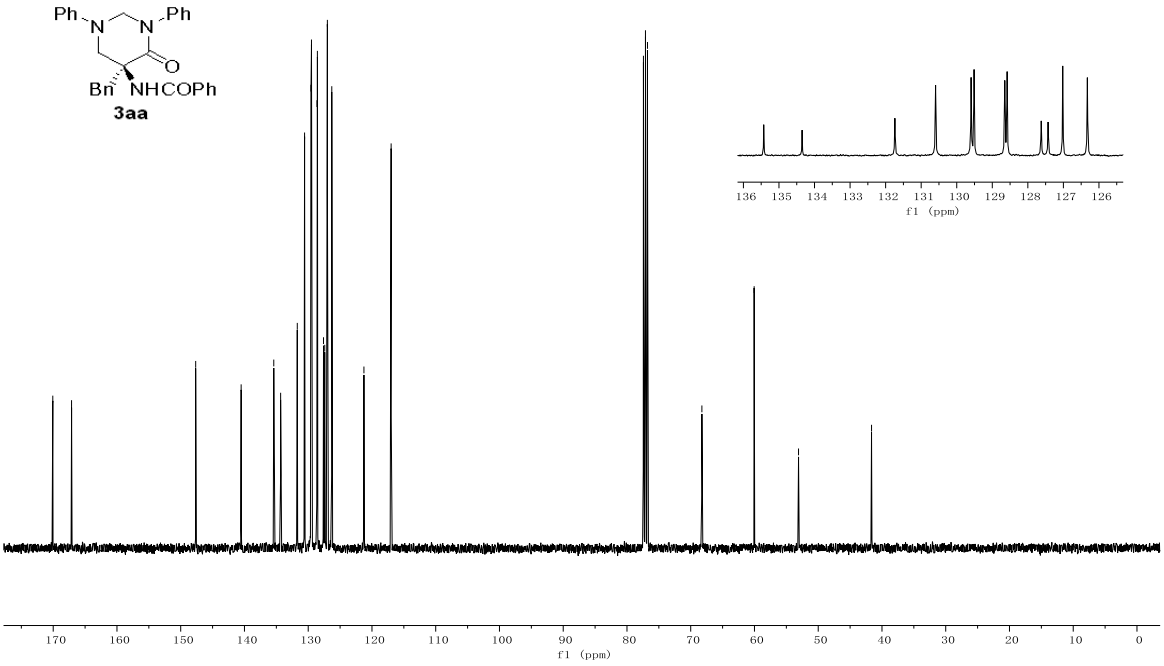
1. Y. H. Wen, X. Huang, J. L. Huang, Y. Xiong, B. Qin, X. M. Feng, *Synlett,* 2005, 2445.
2. (a) S. X. Dong, X. H. Liu, X. M. Feng, *J. Am. Chem. Soc.,* 2010, **132**, 10650; (b) L. H. Xie, Y. Li, S. X. Dong, X. M. Feng, X. H. Liu, *Chem. Commun.,* 2021, **57**, 239; (c) J. Z. Li, Y. H. Mo, L. Q. Yan, X. M. Feng, Z. S. Su, X. H. Liu, *CCS Chemistry,* 2021, 1.
3. N. F. Hu, H. Jung, Y. Zheng, J. Lee, L. L. Zhang, Z. Ullah, X. L. Xie, K. Harms, M.-H. Baik, E. Meggers, *Angew. Chem., Int. Ed.,* 2018, **57**, 6242.
4. C. H. Zhu, G. Y. Xu, J. T. Sun, *Angew. Chem., Int. Ed.,* 2016, **55**, 11867–11871.
5. G. M. Sheldrick, *Acta Cryst*., 2008, **A64**, 112–122.
6. G. M. Sheldrick, *Acta Cryst.,* 2015, **A71**, 3–8.
7. G. M. Sheldrick, *Acta Cryst.,* 2015, **C71**, 3–8.
8. O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard, H. J Puschmann, *Appl. Cryst.,* 2009, **42**, 339-341.
9. A. L. J. Spek, *Appl. Cryst.,* 2003, **36**, 7–13.

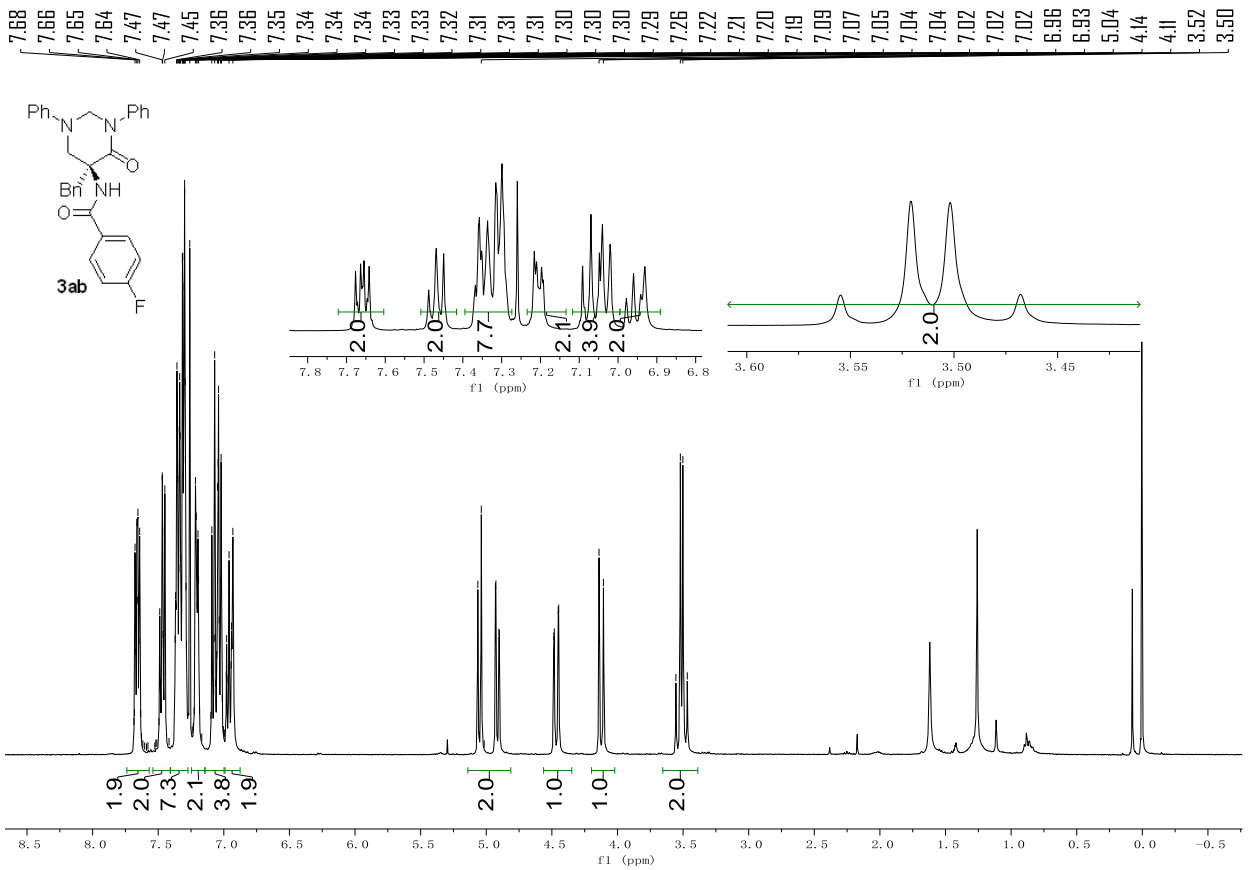
14. Copies of NMR spectra for products







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Ph N N Ph O Bn NH O F
3ab
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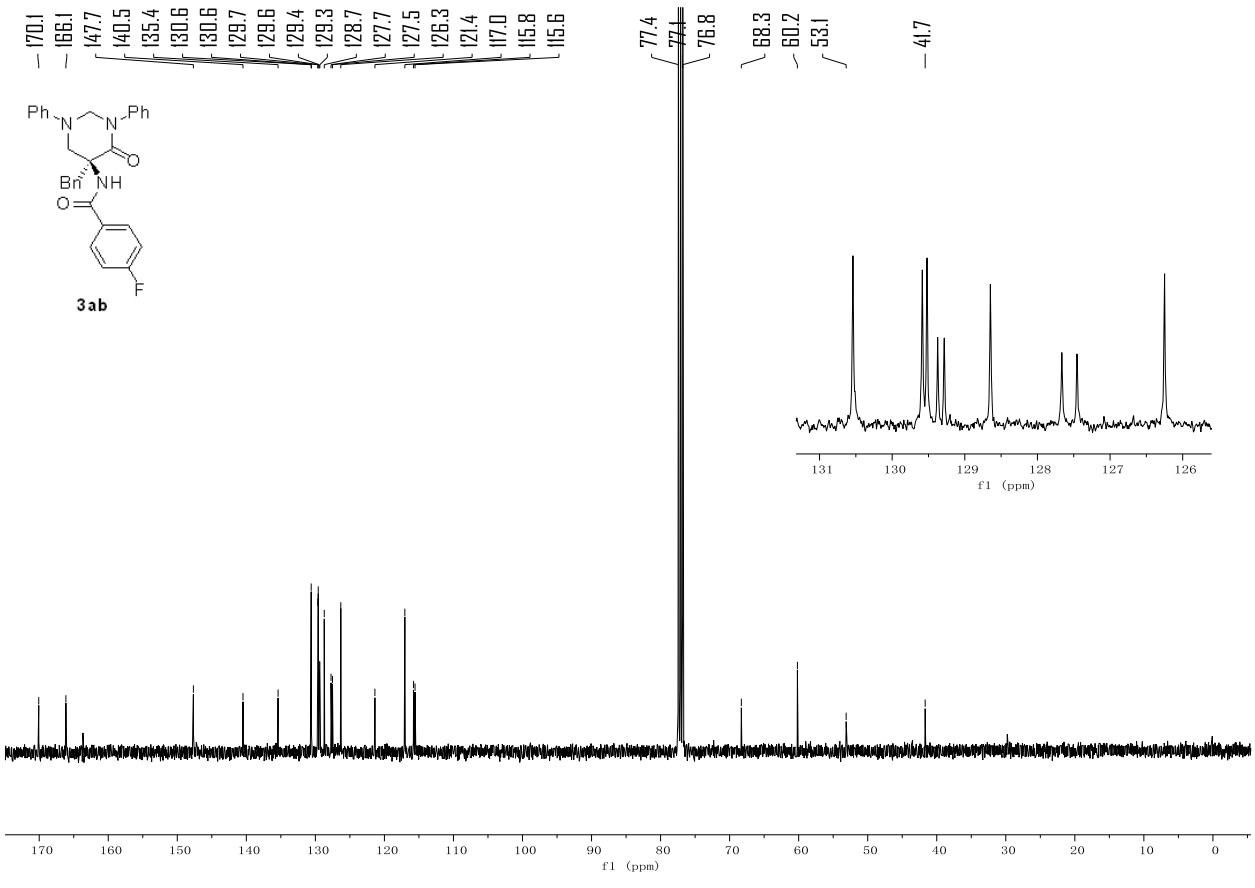
f1 (ppm)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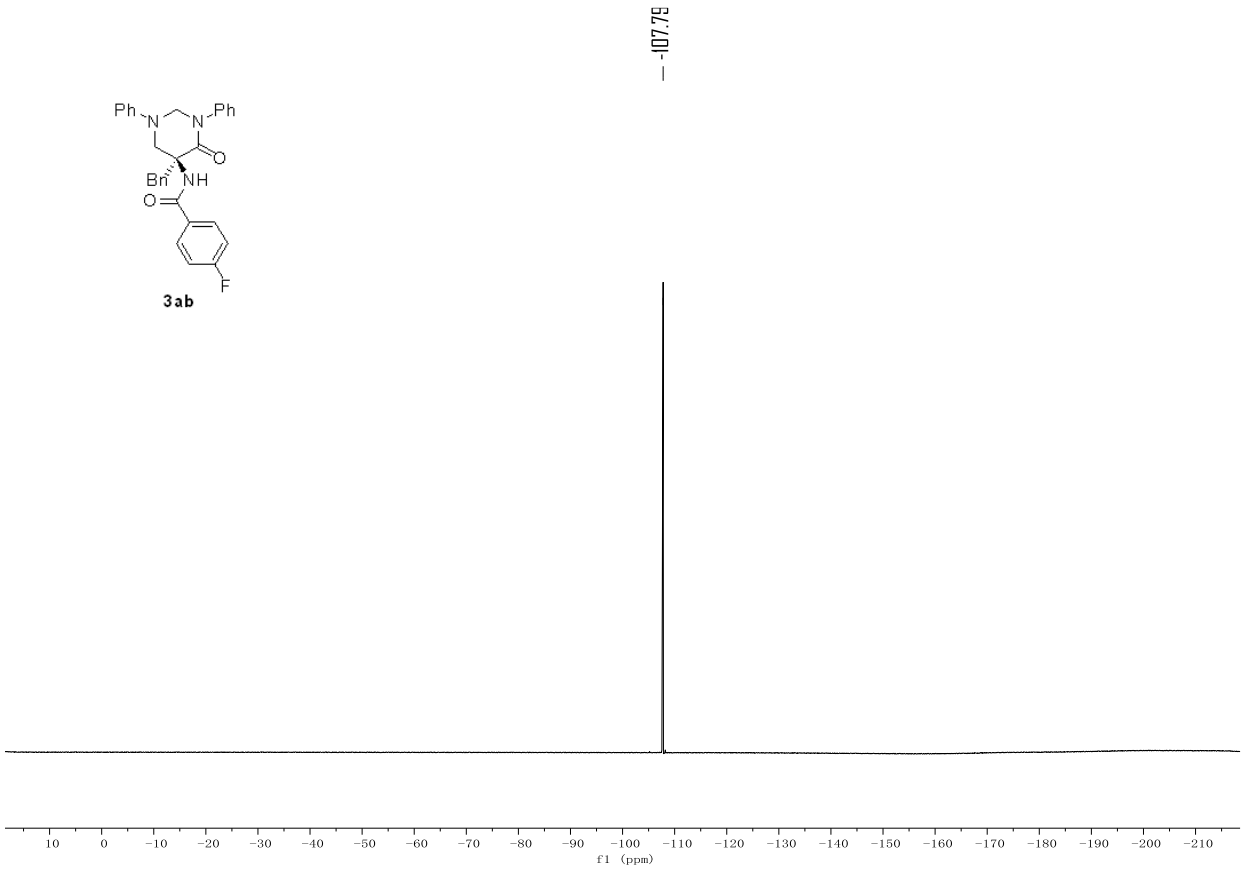
3ab
f1 (ppm)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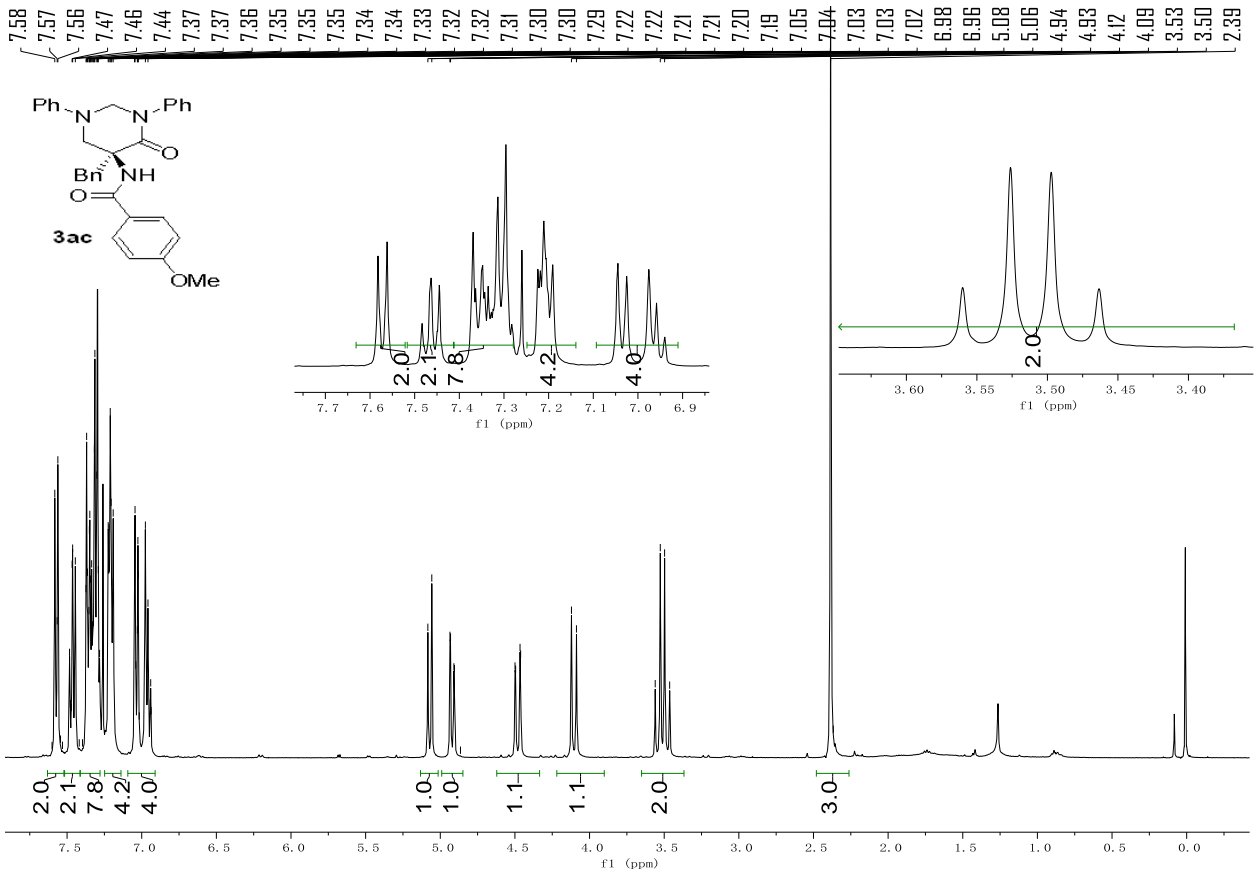
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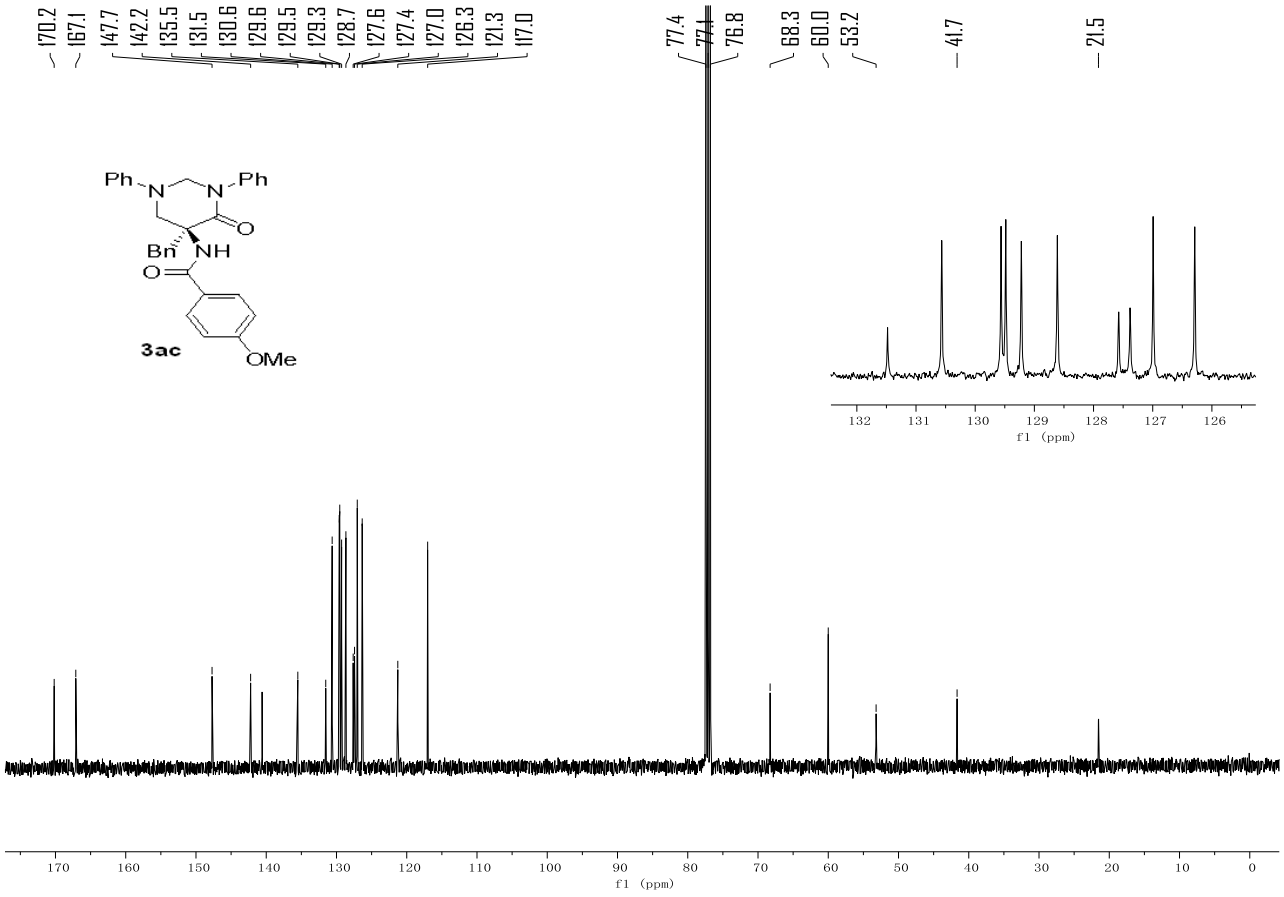
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Ph
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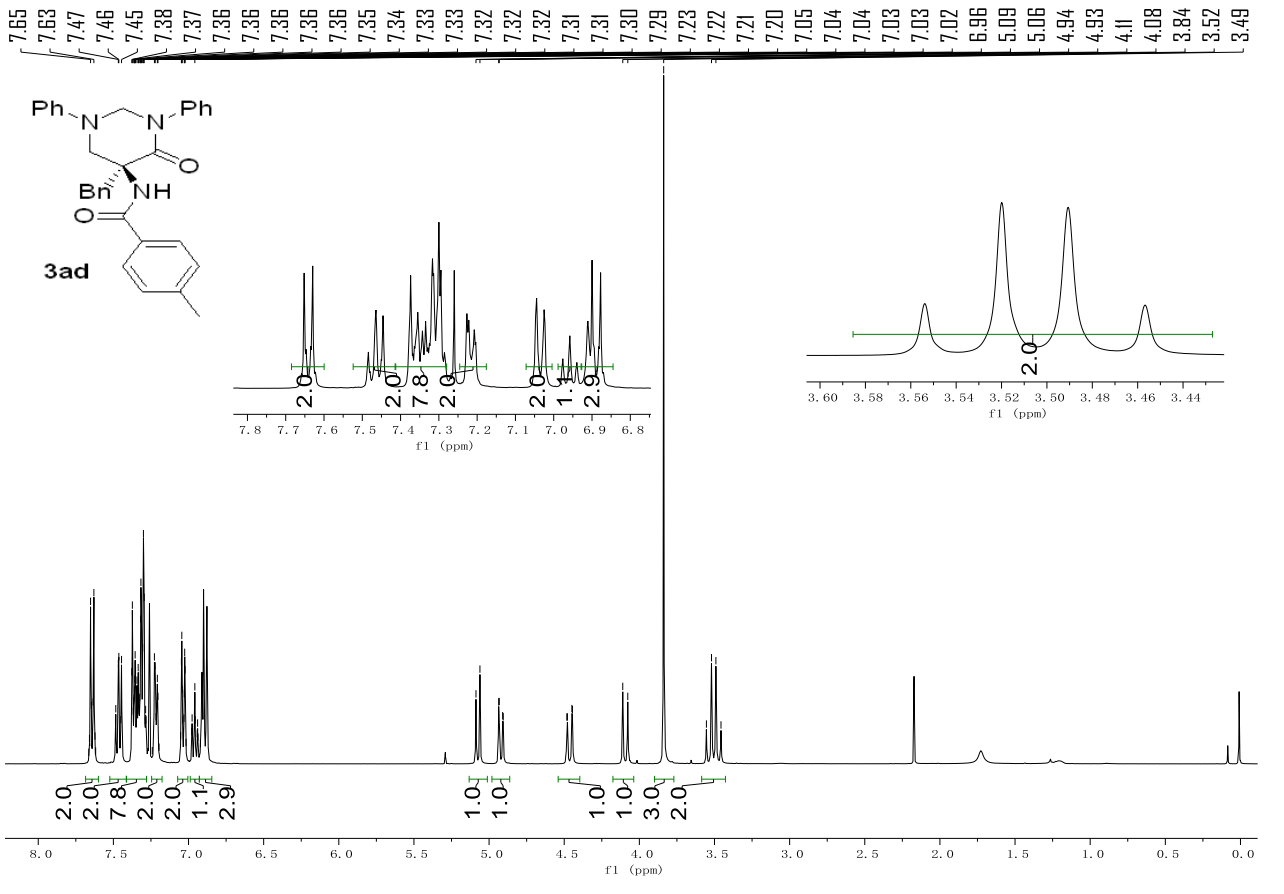
Bn
NH
O
3ac
OMe
f1 (ppm)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7.34
7.33
7.33
7.32
7.32
7.32
7.31
7.31
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7.29
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7.22
7.21
7.20
7.05
7.04
7.04
7.03
7.03
7.02
6.96
5.09
5.06
4.94
4.93
4.11
4.08
3.84
3.52
3.49
Ph
N
Ph
O
Bn
NH
O
3ad
2.0
2.0
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2.0
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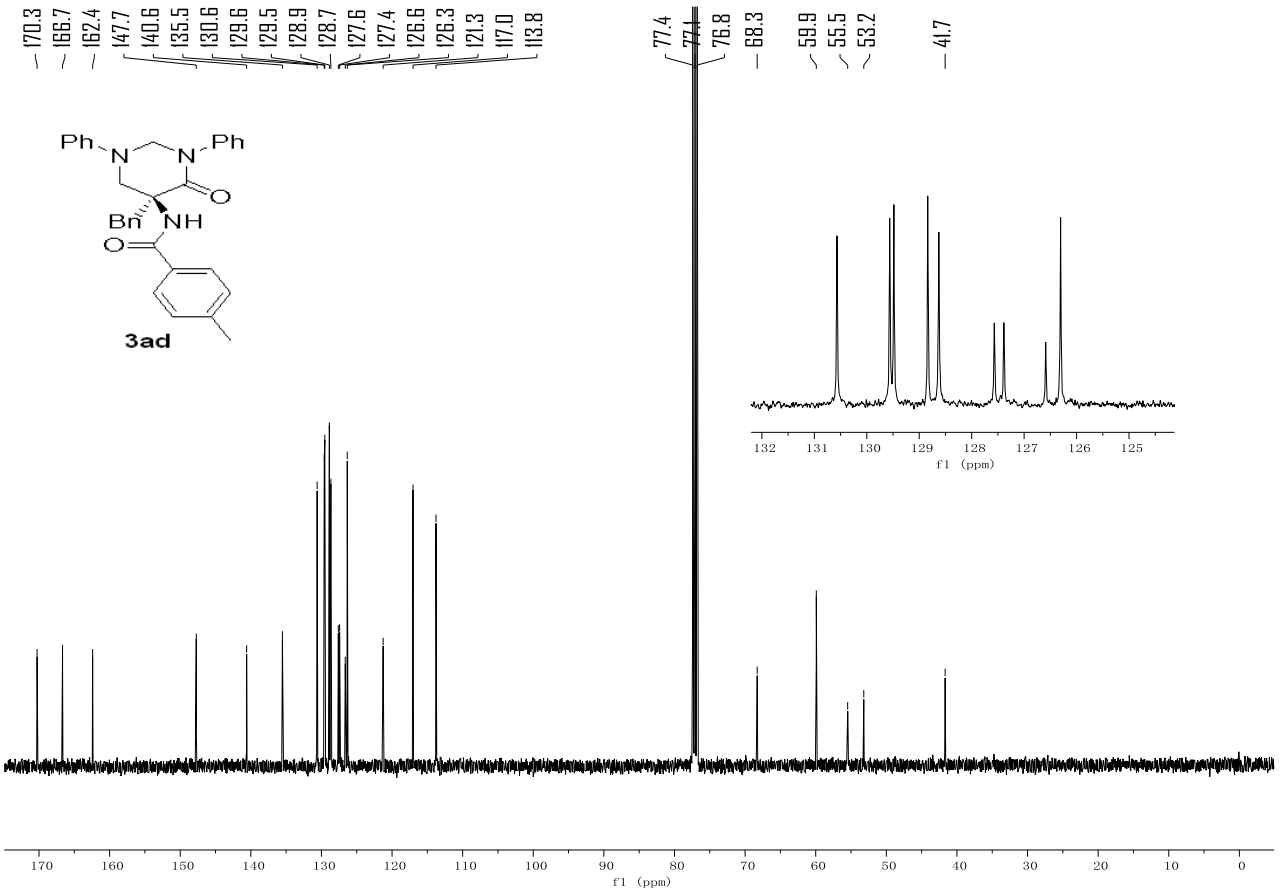
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127.4
126.6
126.3
121.3
117.0
113.8
77.4
77.1
76.8
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41.7
Ph
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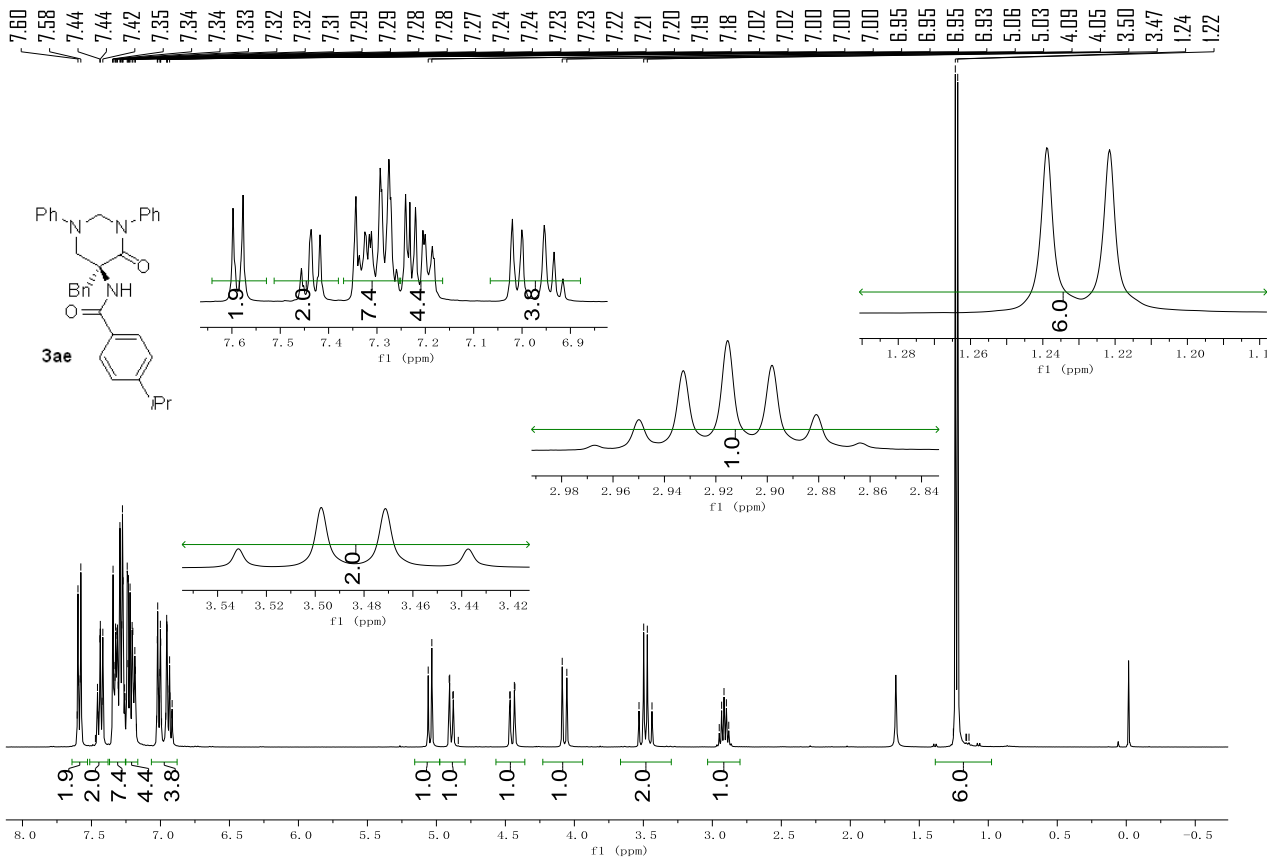
Ph
O
Bn
NH
O
3ad
f1 (ppm)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7.29
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7.24
7.23
7.23
7.22
7.21
7.20
7.19
7.18
7.02
7.02
7.00
7.00
7.00
6.95
6.95
6.95
6.93
5.06
5.03
4.09
4.05
3.50
3.47
1.24
1.22
Ph
N
N
Ph
O
Bn
NH
O
3ae
iPr
1.9
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3.8
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1.0
1.0
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2.0
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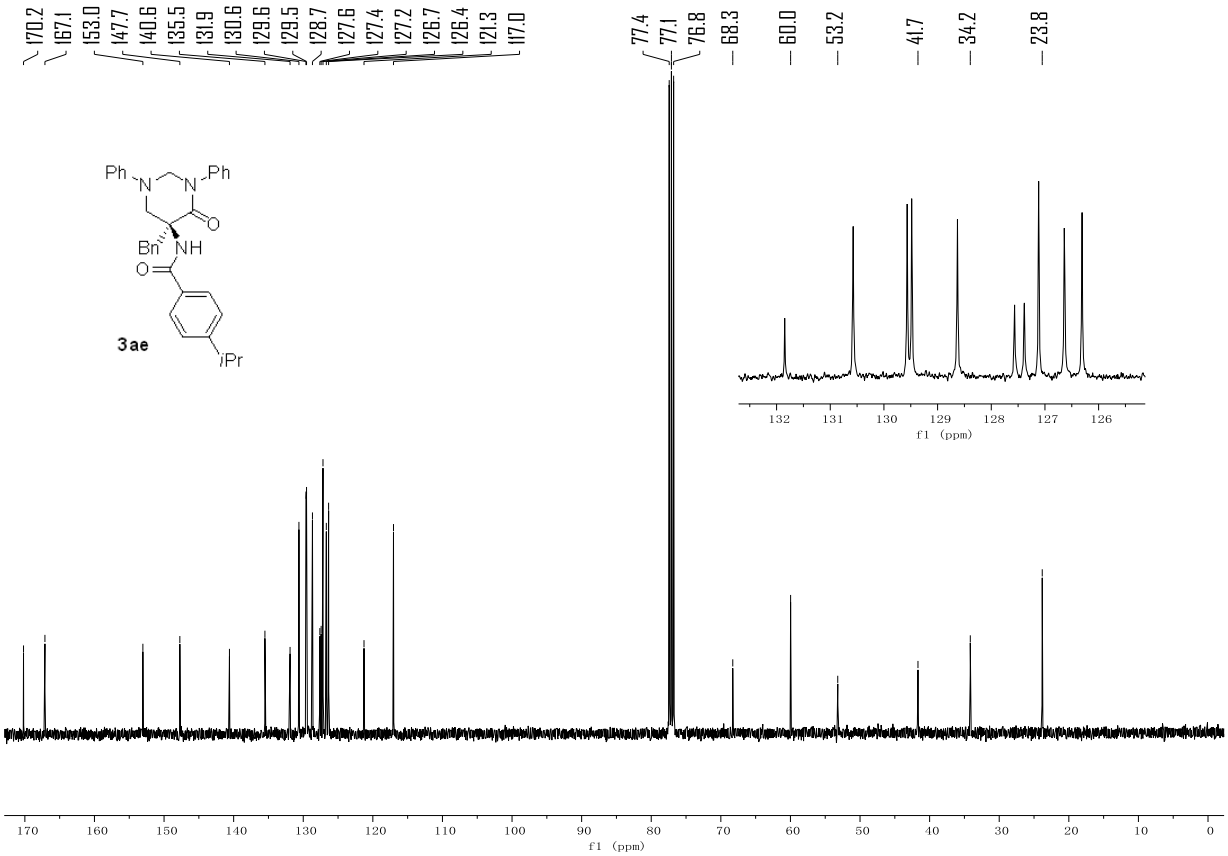
6.0
f1 (ppm)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127.4
127.2
126.7
126.4
121.3
117.0
77.4
77.1
76.8
68.3
60.0
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23.8
Ph
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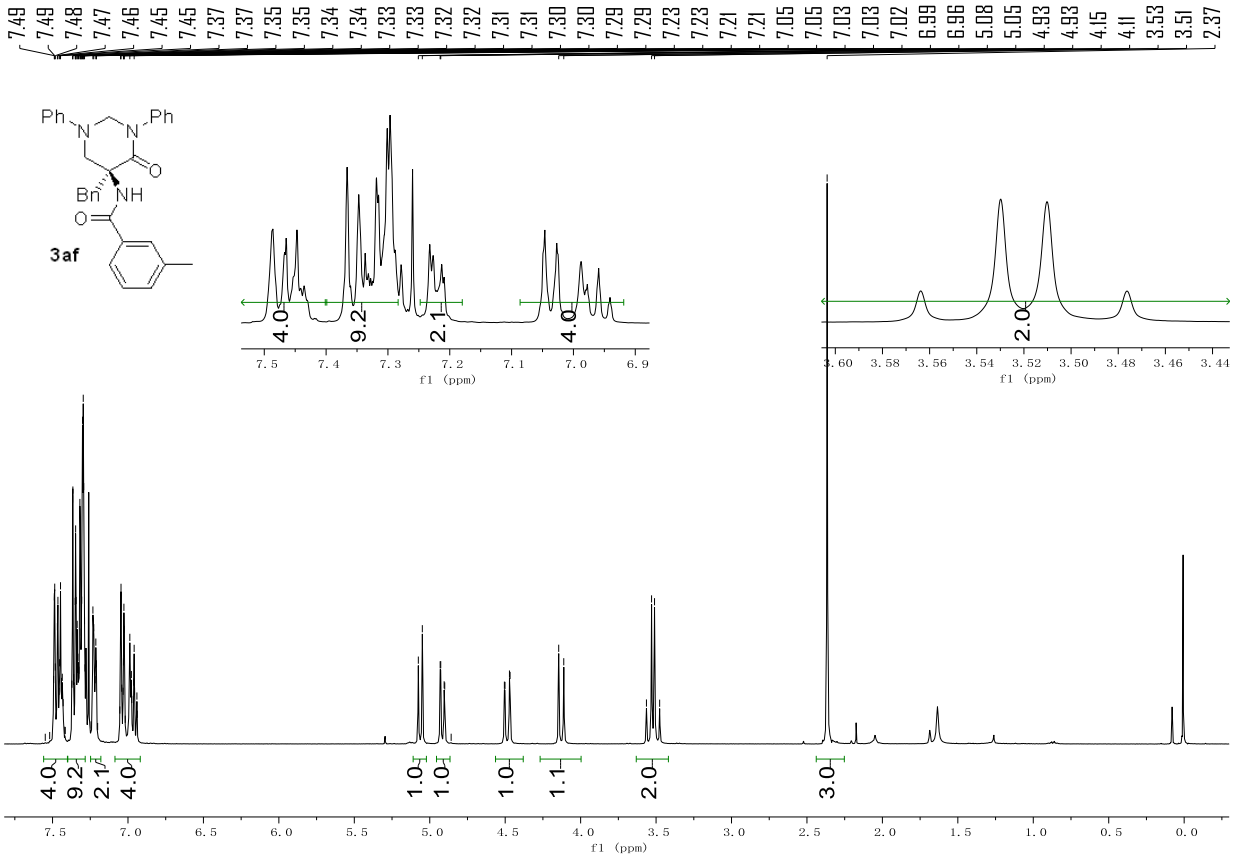
Ph
O
Bn
NH
O
3ae
iPr
f1 (ppm)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7.23
7.21
7.21
7.05
7.05
7.03
7.03
7.02
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5.08
5.05
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4.93
4.15
4.11
3.53
3.51
2.37
Ph
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Ph
O
Bn
NH
O
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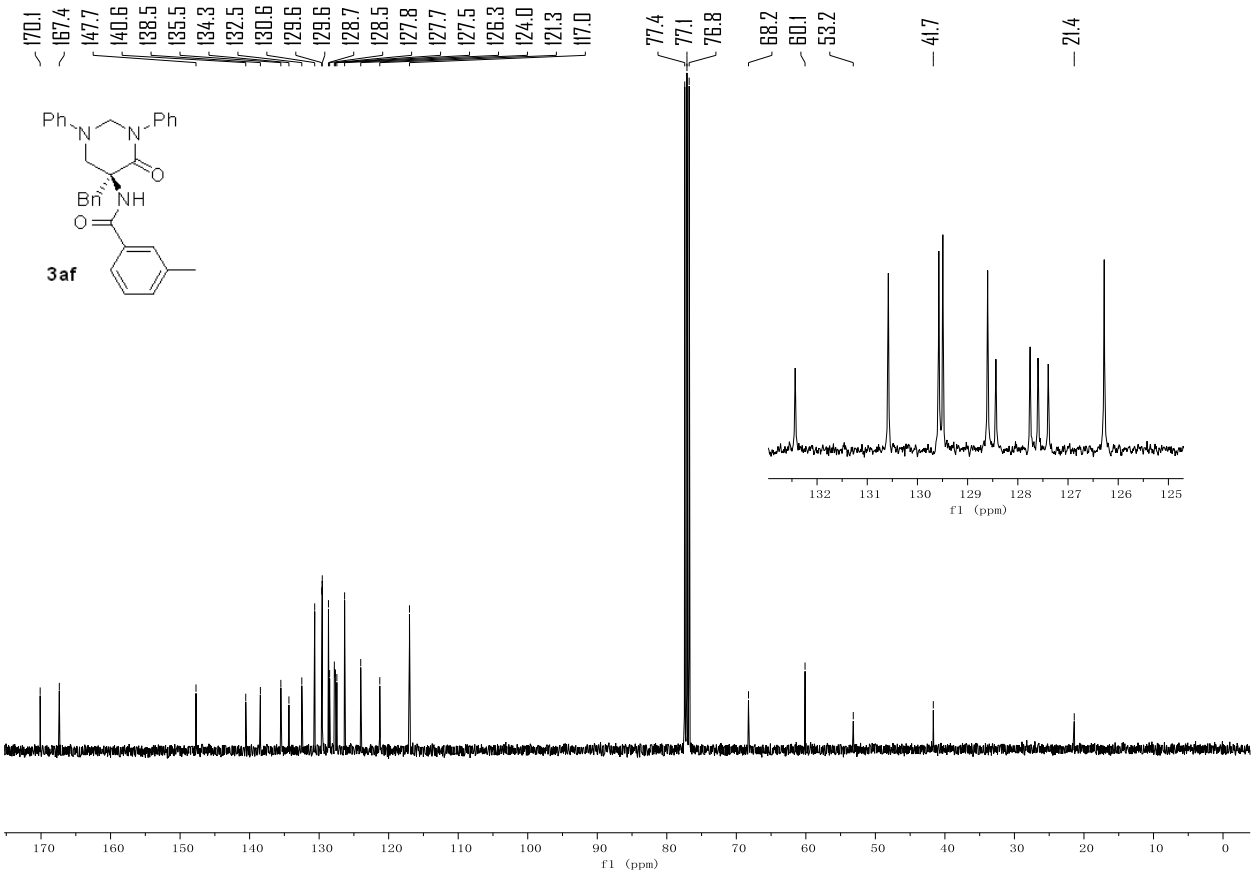
2.0
3.0
f1 (ppm)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128.5
127.8
127.7
127.5
126.3
124.0
121.3
117.0
77.4
77.1
76.8
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41.7
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Ph
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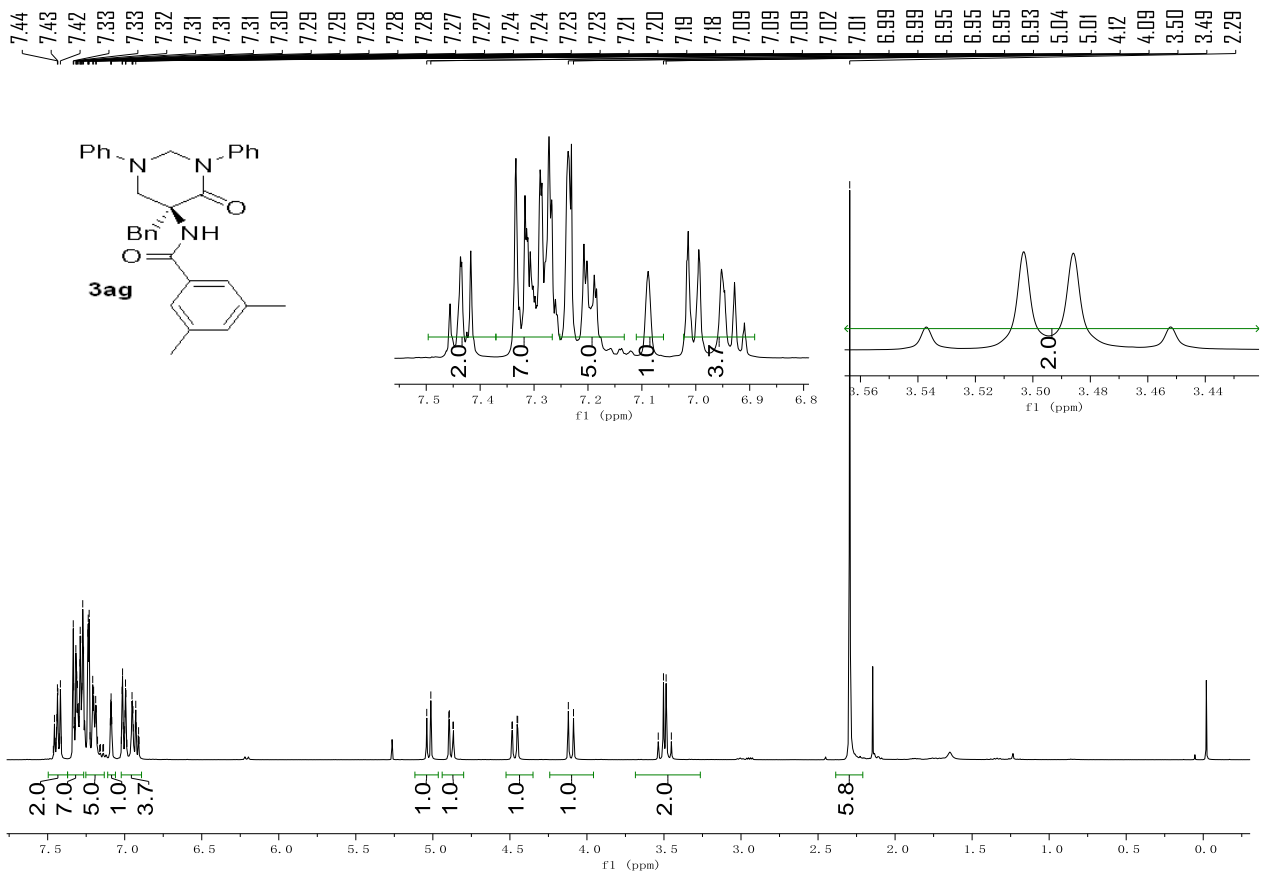
Ph
O
Bn
NH
O
3af
f1 (ppm)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7.20
7.19
7.18
7.09
7.09
7.09
7.02
7.01
6.99
6.99
6.95
6.95
6.95
6.93
5.04
5.01
4.12
4.09
3.50
3.49
2.29
Ph
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N
Ph
O
Bn
NH
O
3ag
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3.7
2.0
1.0
1.0
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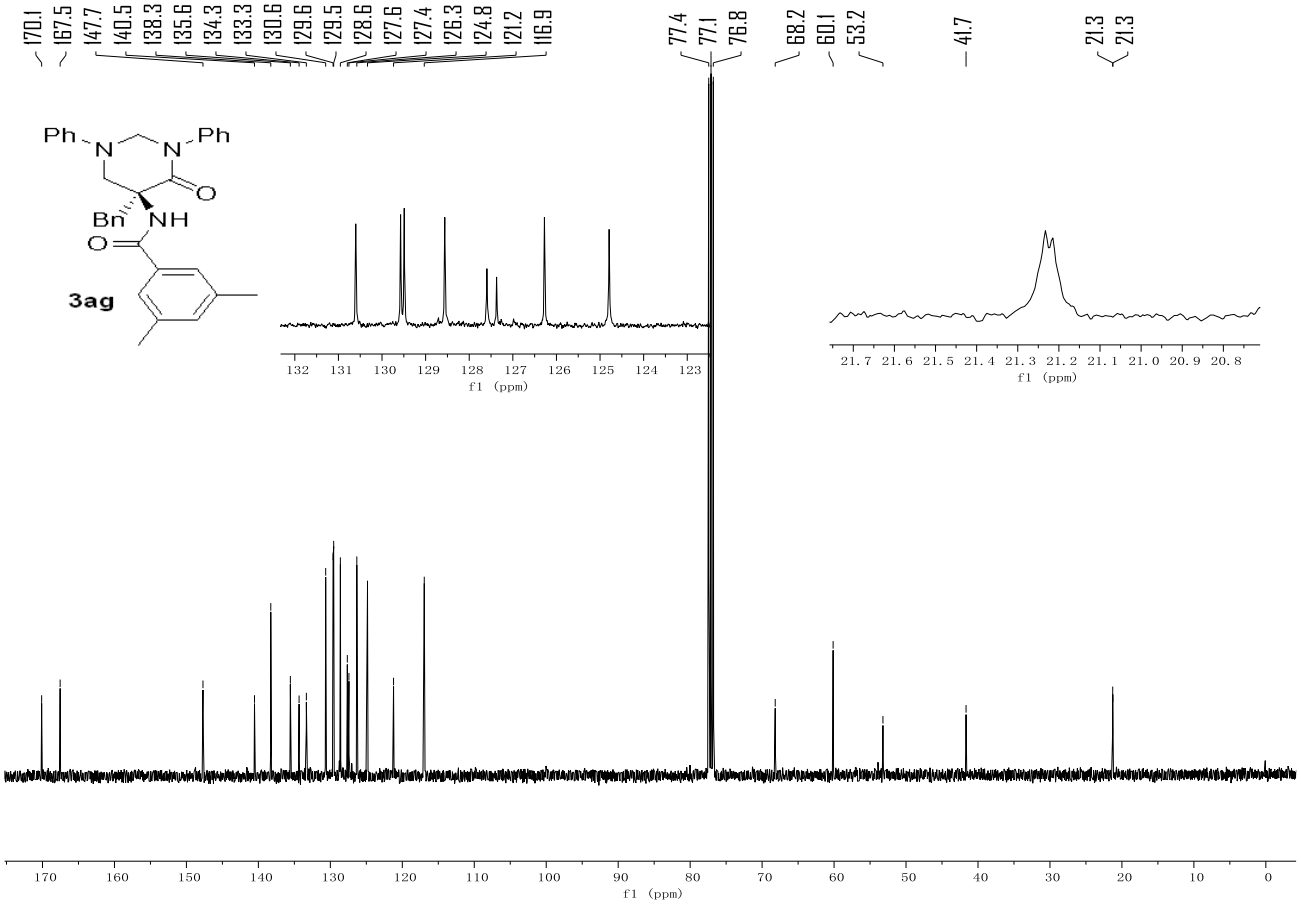
1.0
2.0
5.8
f1 (ppm)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135.6
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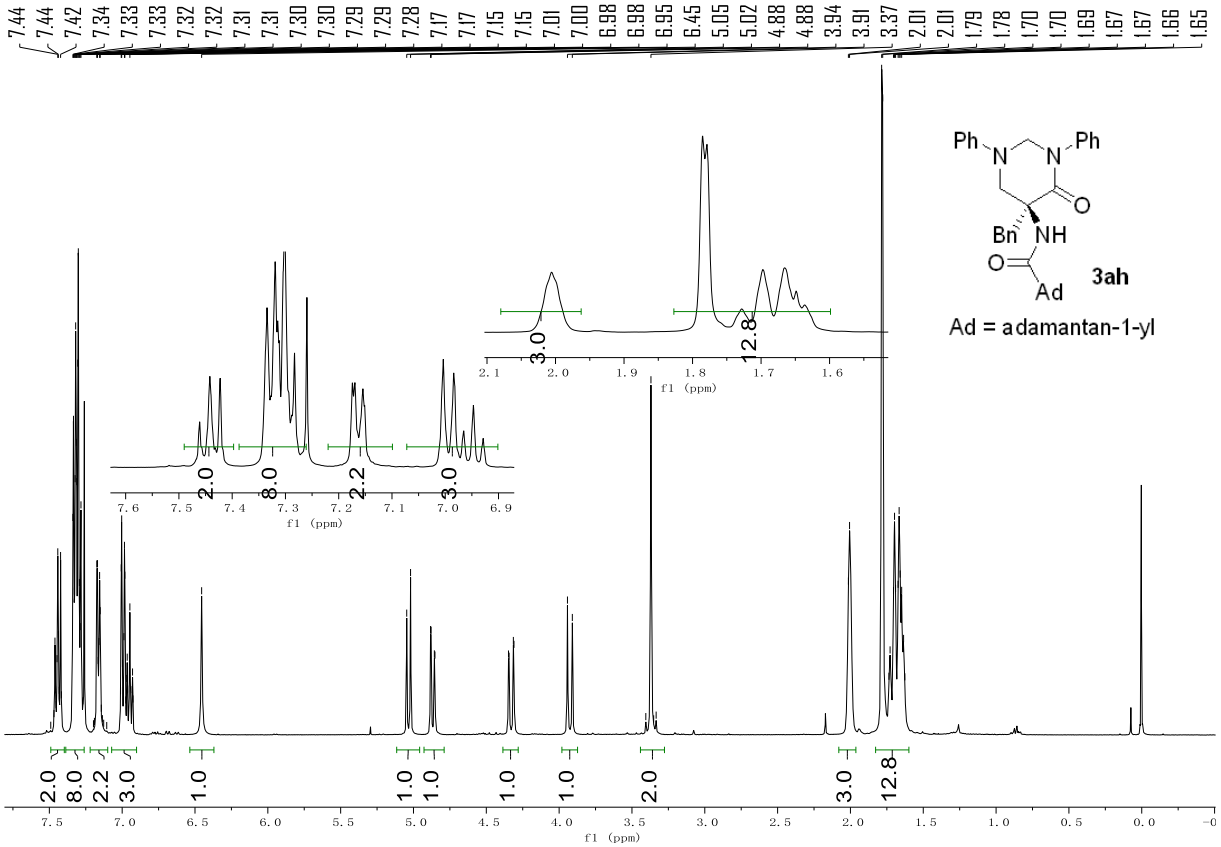
128.6
127.6
127.4
126.3
124.8
121.2
116.9
77.4
77.1
76.8
68.2
60.1
53.2
41.7
21.3
21.3
Ph
N
N
Ph
O
Bn
NH
O
3ag
f1 (ppm)
f1 (ppm)
f1 (ppm)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7.01
7.00
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6.45
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5.02
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3.91
3.37
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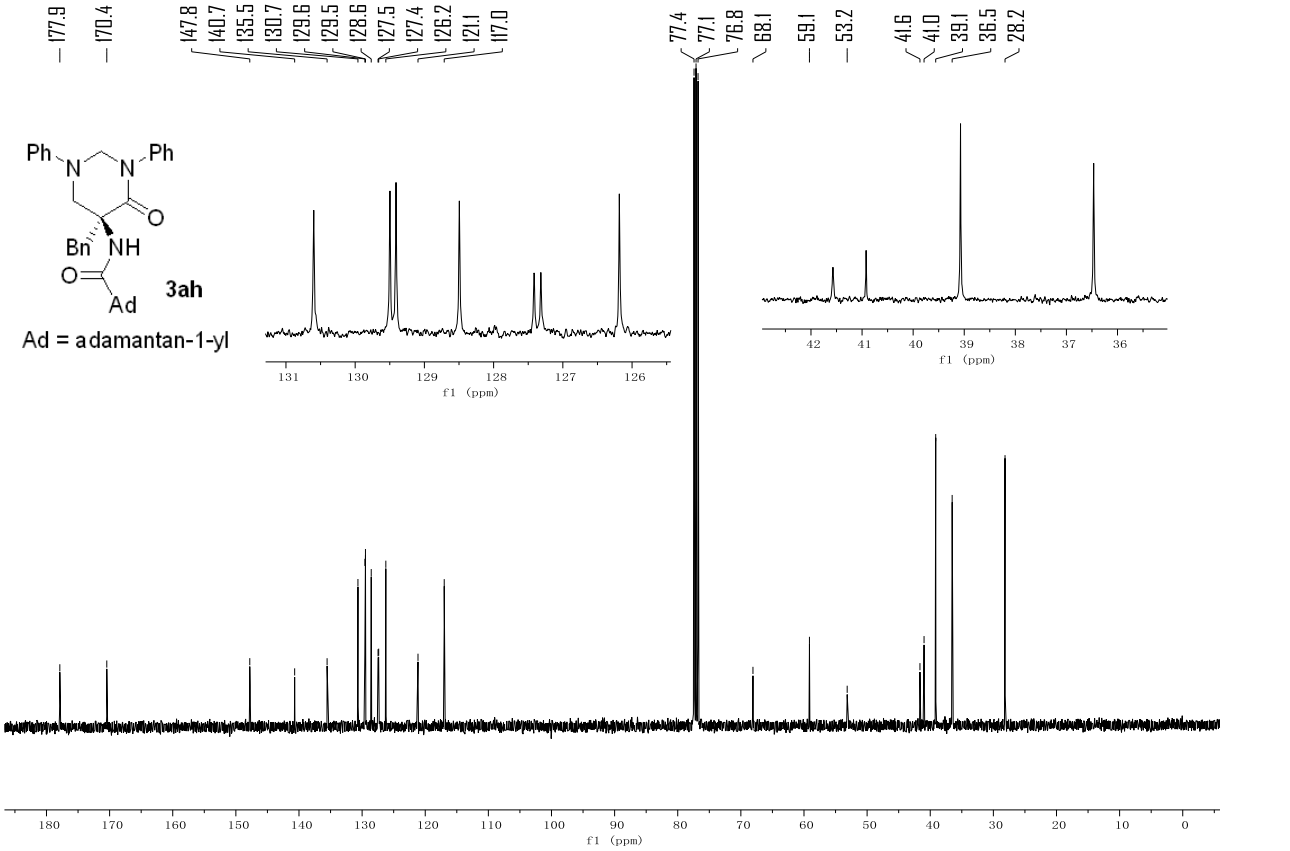
1.78
1.70
1.70
1.69
1.67
1.67
1.66
1.65
Ph
N
N
Ph
O
Bn
NH
O
Ad
3ah
Ad = adamantan-1-yl
2.0
8.0
2.2
3.0
1.0
1.0
1.0
1.0
1.0
2.0
3.0
12.8
f1 (ppm)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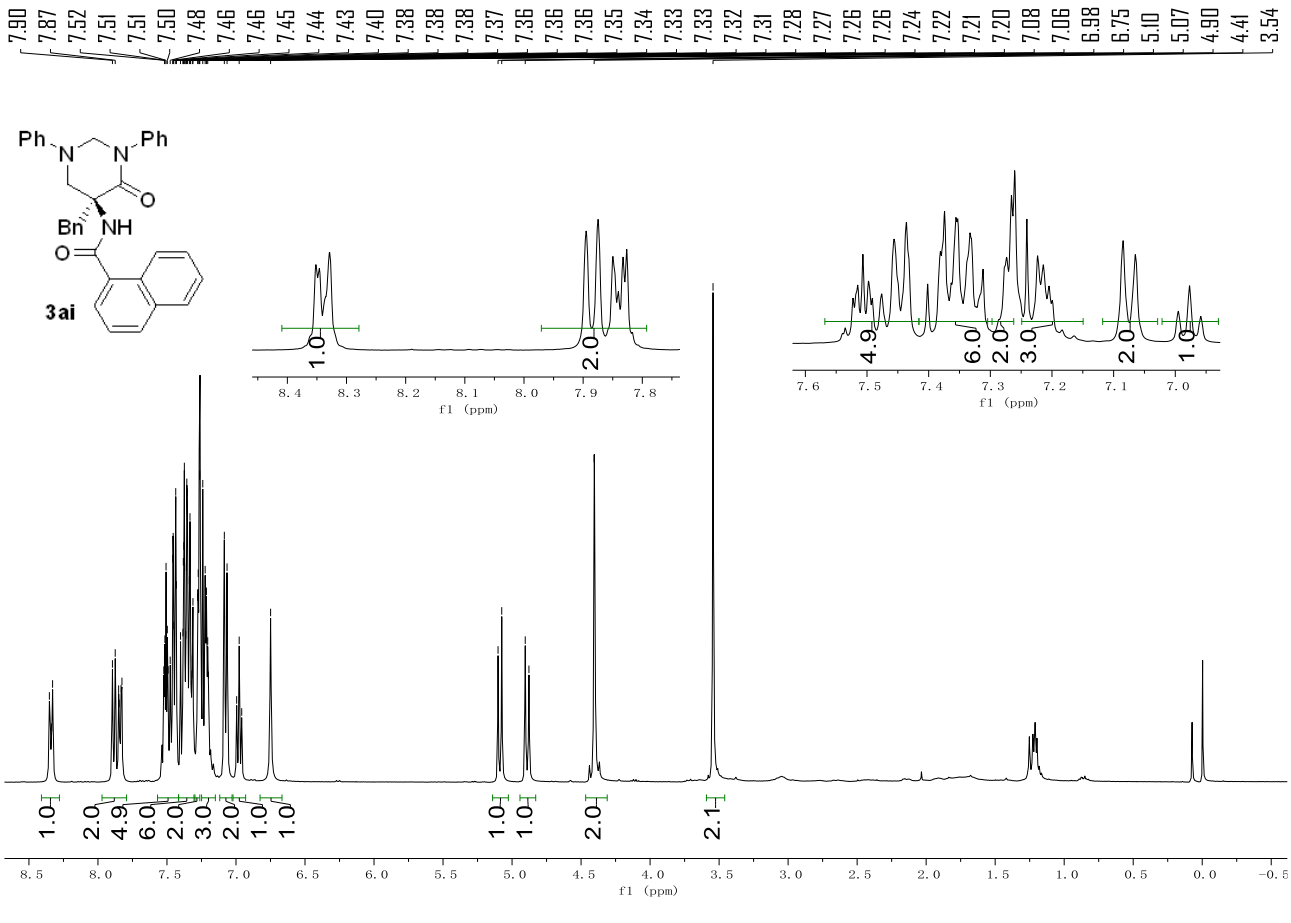
170.4
147.8
140.7
135.5
130.7
129.6
129.5
128.6
127.5
127.4
126.2
121.1
117.0
77.4
77.1
76.8
68.1
59.1
53.2
41.6
41.0
39.1
36.5
28.2
Ph
N
N
Ph
O
Bn
NH
O
Ad
3ah
Ad = adamantan-1-yl
f1 (ppm)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7.37
7.36
7.36
7.36
7.35
7.34
7.33
7.33
7.32
7.31
7.28
7.27
7.26
7.26
7.24
7.22
7.21
7.20
7.08
7.06
6.98
6.75
5.10
5.07
4.90
4.41
3.54
Ph
N
N
Ph
O
Bn
NH
O
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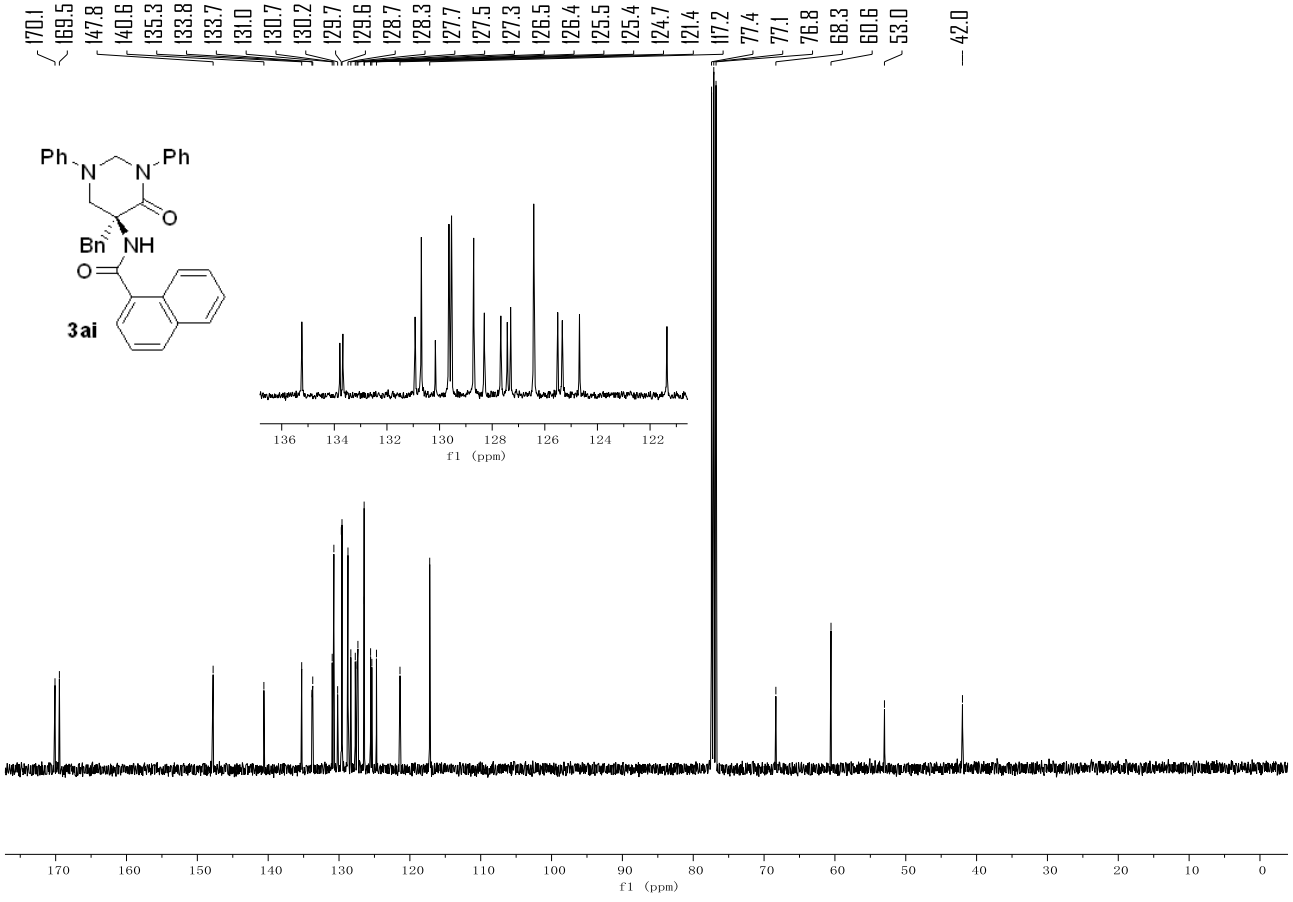
2.0
4.9
6.0
2.0
3.0
2.0
1.0
f1 (ppm)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133.8
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131.0
130.7
130.2
129.7
129.6
128.7
128.3
127.7
127.5
127.3
126.5
126.4
125.5
125.4
124.7
121.4
117.2
77.4
77.1
76.8
68.3
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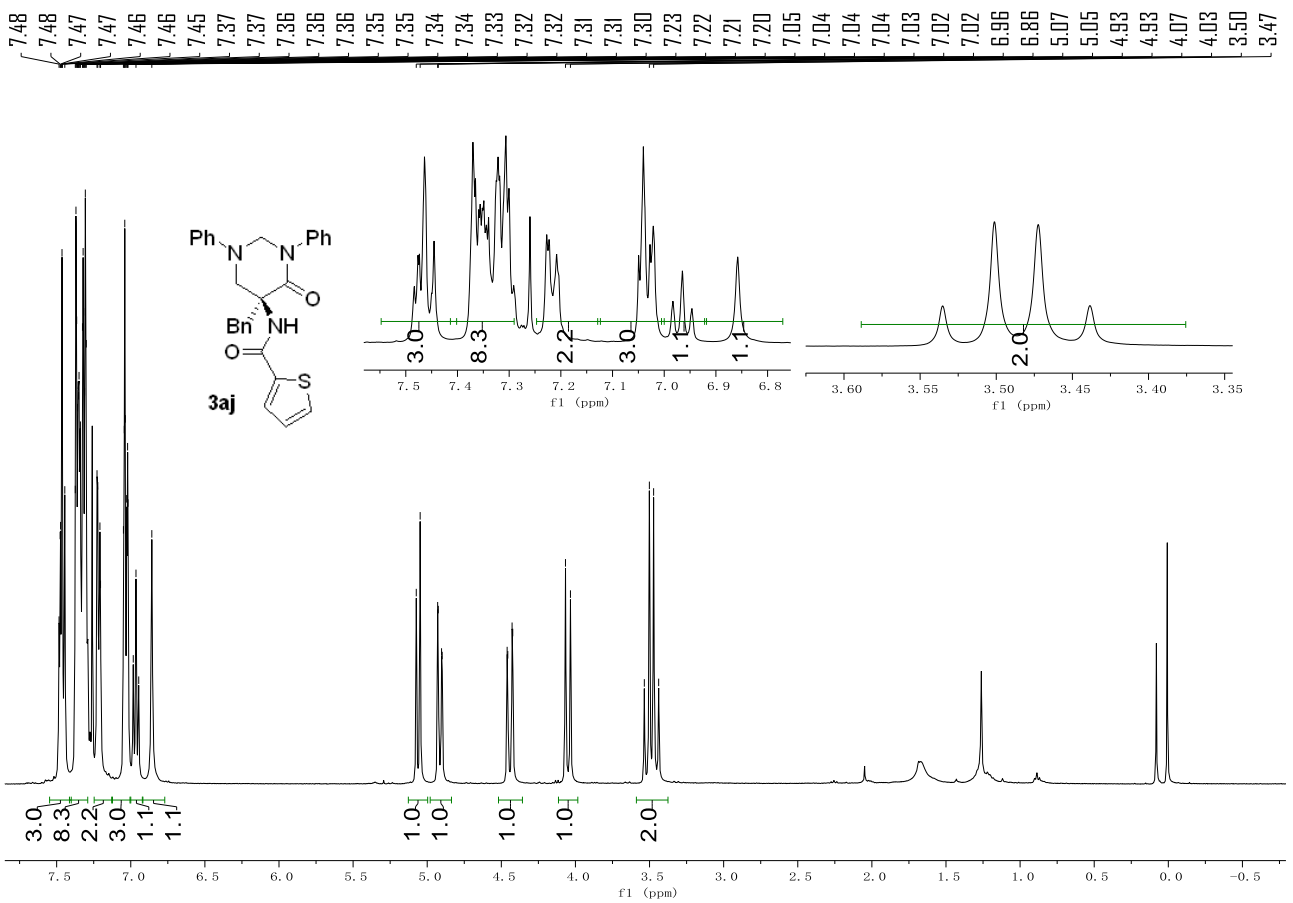
Ph
O
Bn
NH
O
3ai
f1 (ppm)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7.34
7.34
7.33
7.32
7.32
7.31
7.31
7.30
7.23
7.22
7.21
7.20
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7.04
7.04
7.04
7.03
7.02
7.02
6.96
6.86
5.07
5.05
4.93
4.93
4.07
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N
N
Ph
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S
3aj
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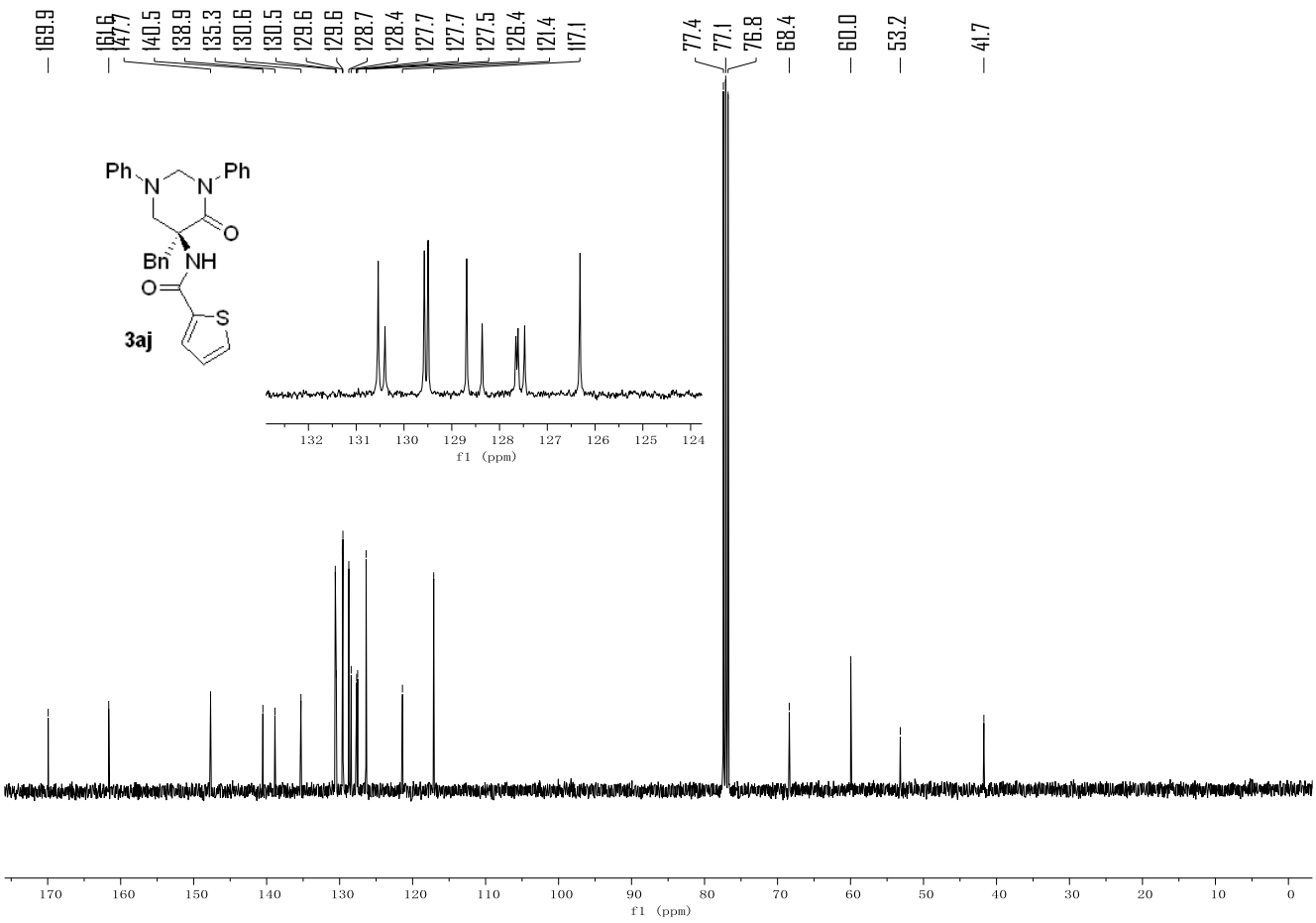
1.1
1.1
2.0
f1 (ppm)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135.3
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130.5
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129.6
128.7
128.4
127.7
127.7
127.5
126.4
121.4
117.1
77.4
77.1
76.8
68.4
60.0
53.2
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Ph
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Ph
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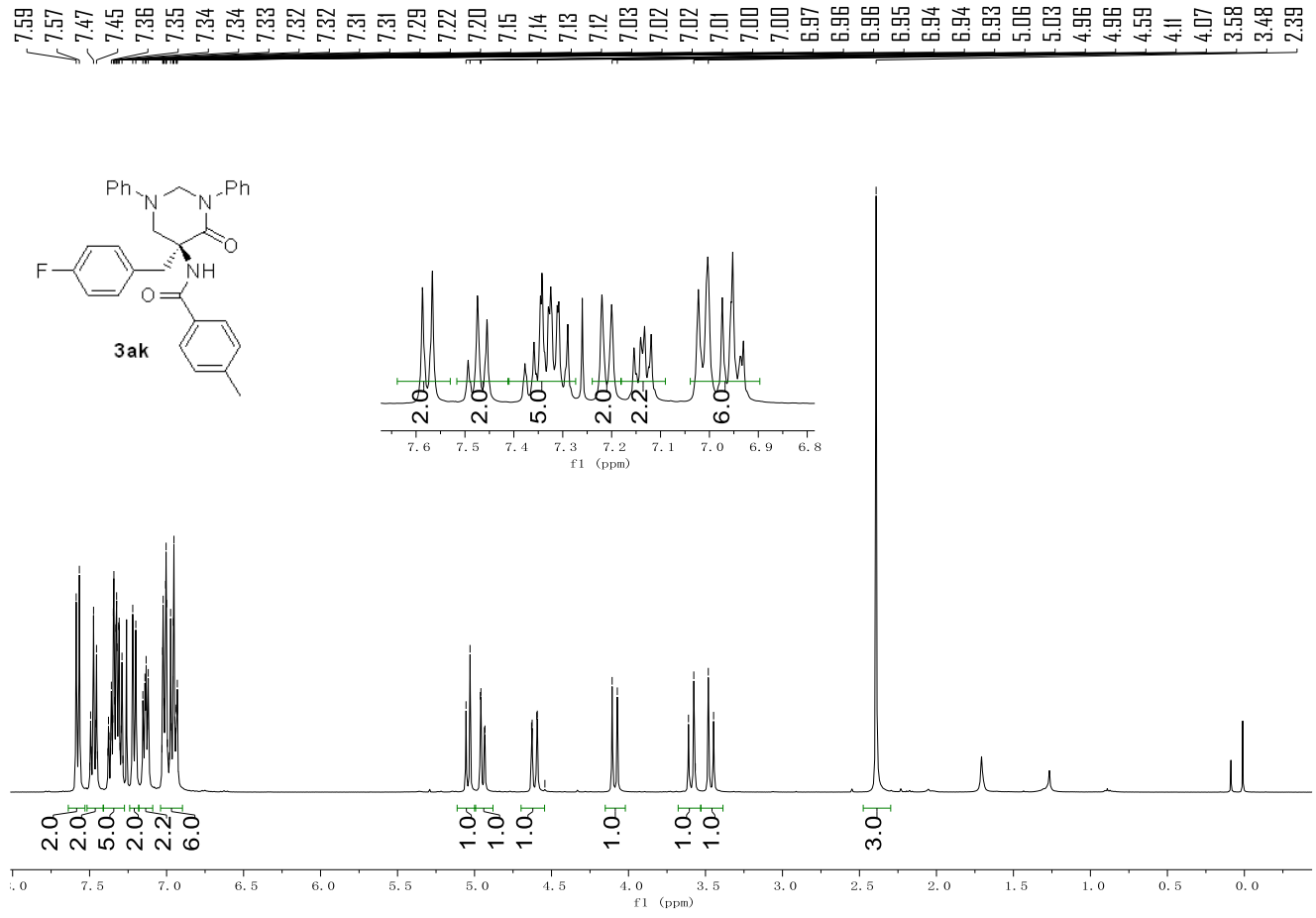
Bn
NH
O
S
3aj
f1 (ppm)
f1 (ppm)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7.29
7.22
7.20
7.15
7.14
7.13
7.12
7.03
7.02
7.02
7.01
7.00
7.00
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6.96
6.96
6.95
6.94
6.94
6.93
5.06
5.03
4.96
4.96
4.59
4.11
4.07
3.58
3.48
2.39
Ph
N
N
Ph
O
F
NH
O
3ak
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2.0
5.0
2.0
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1.0
1.0
1.0
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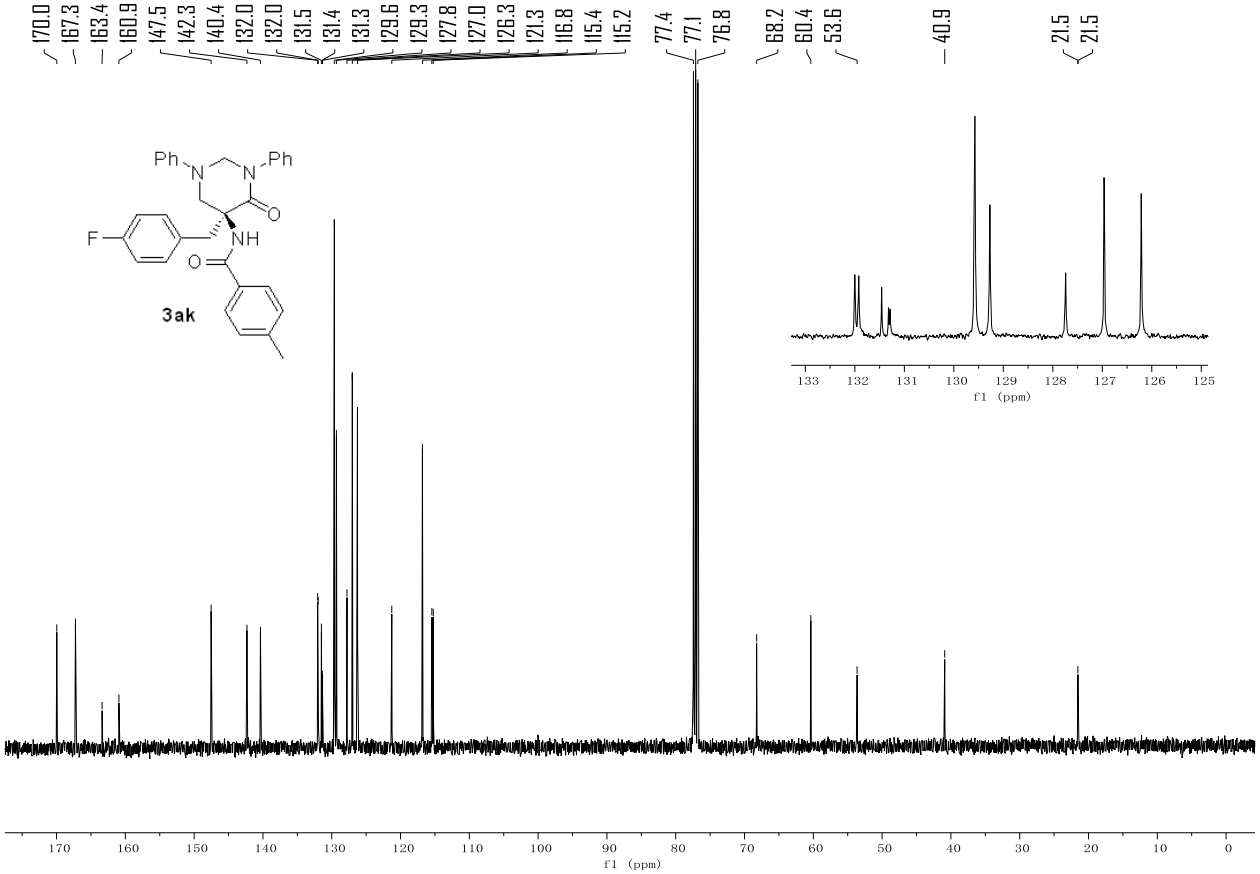
1.0
3.0
f1 (ppm)
f1 (ppm)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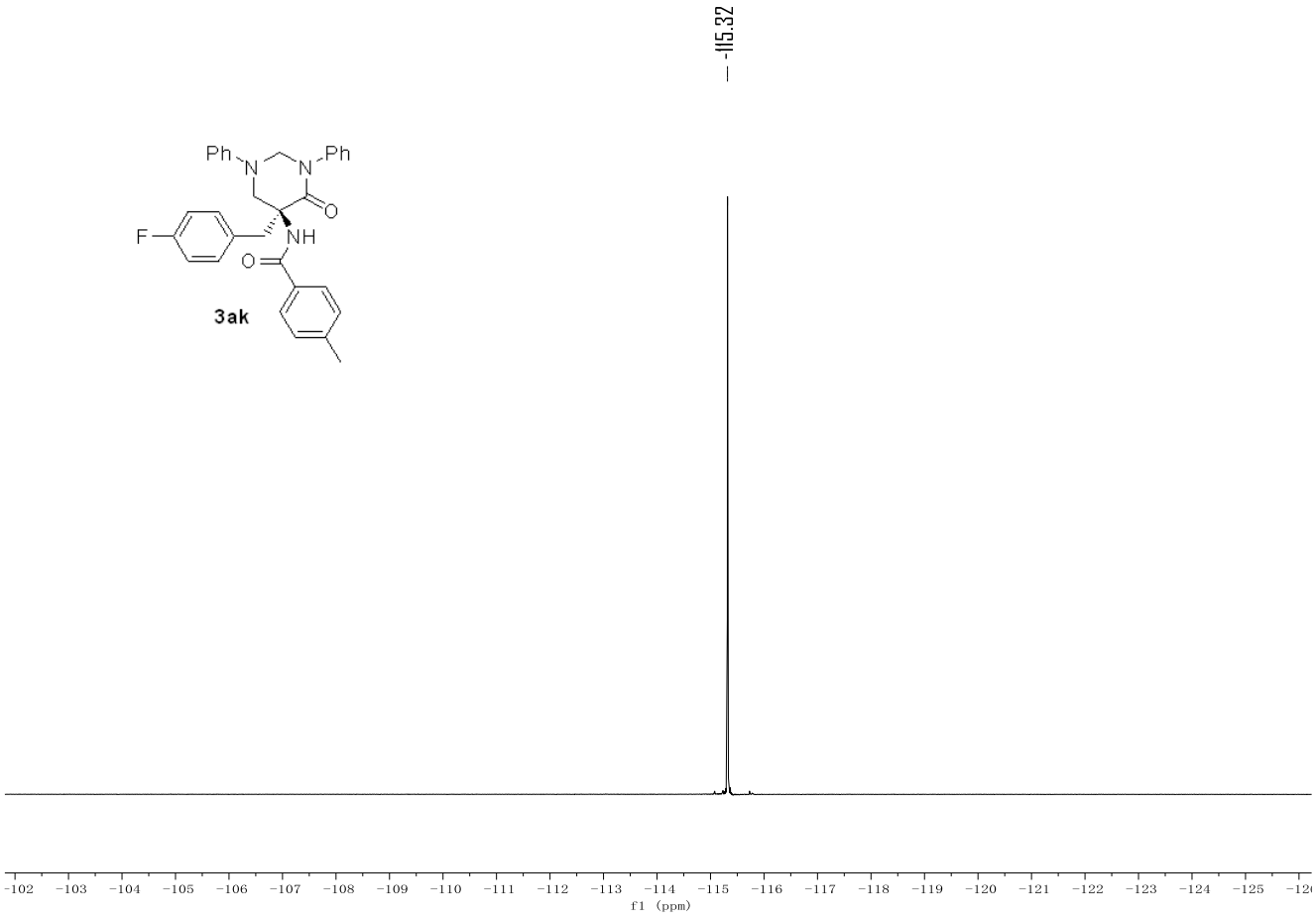
160.9
147.5
142.3
140.4
132.0
132.0
131.5
131.4
131.3
129.6
129.3
127.8
127.0
126.3
121.3
116.8
115.4
115.2
77.4
77.1
76.8
68.2
60.4
53.6
40.9
21.5
21.5
Ph
N
N
Ph
O
F
NH
O
3ak
f1 (ppm)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Ph
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F
NH
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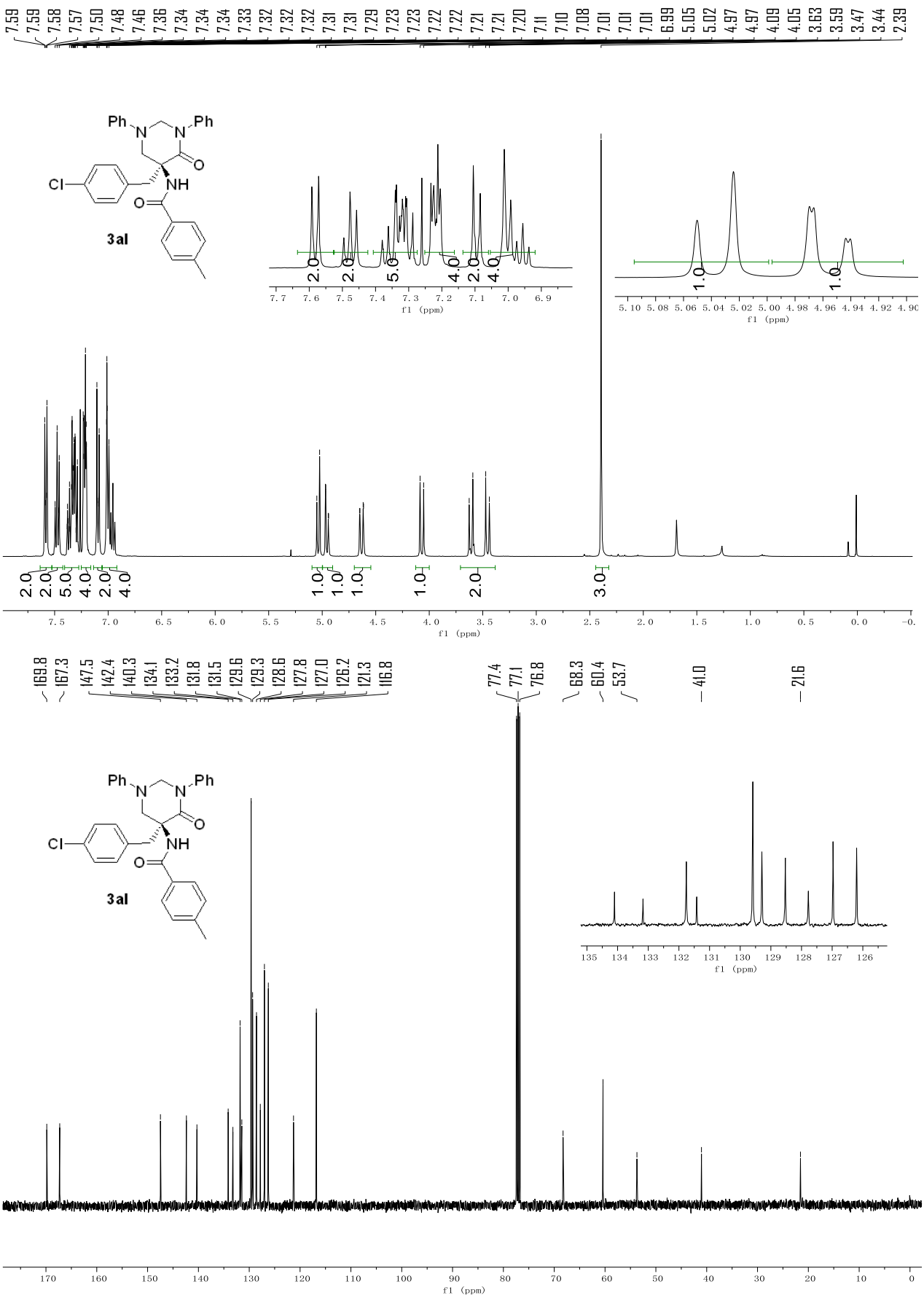
f1 (ppm)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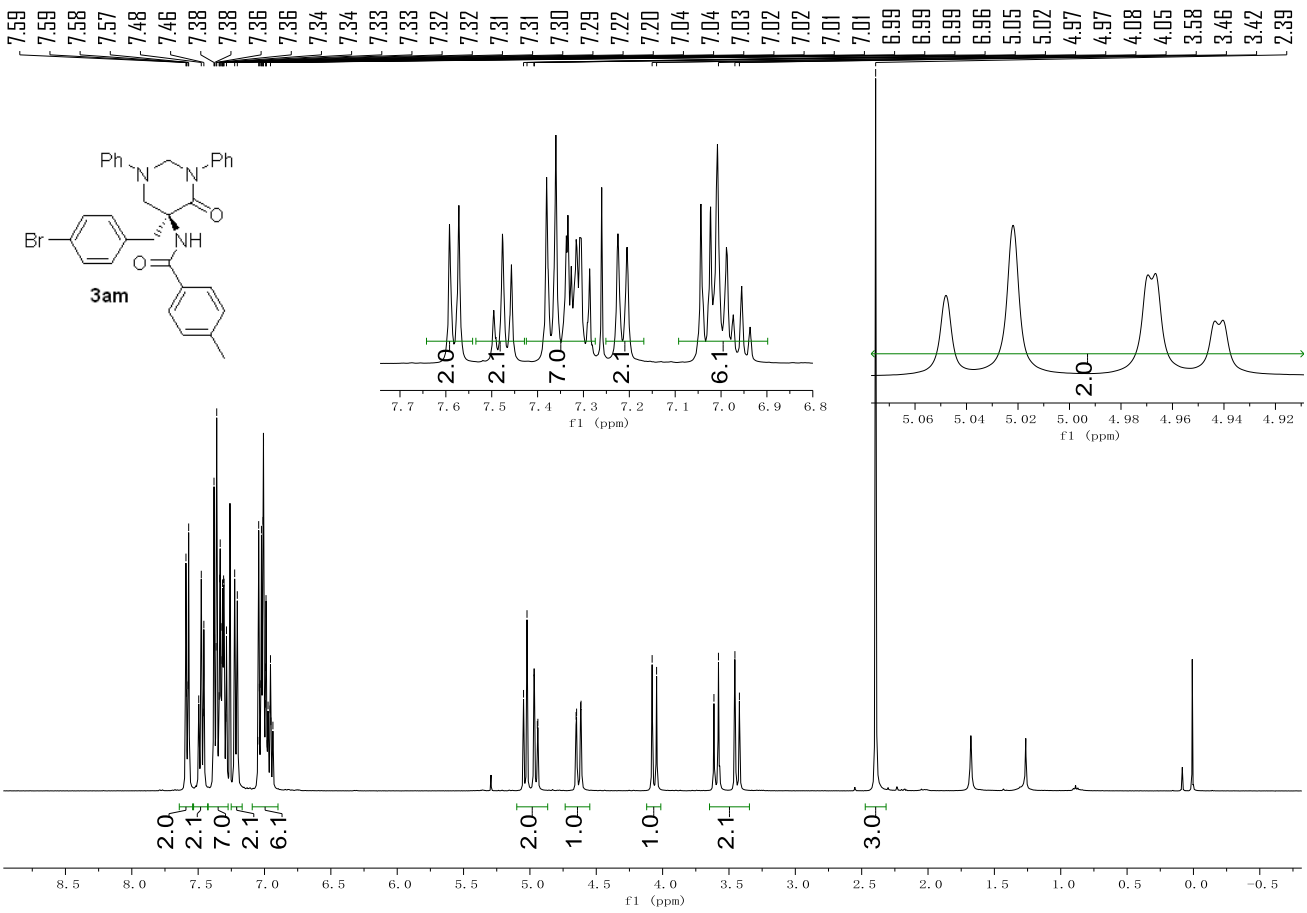
Ph
N
N
Ph
O
Cl
NH
O
3al
2.0 2.0 5.0 4.0 2.0 4.0
1.0 1.0 1.0 1.0 2.0 3.0
f1 (ppm)
169.8 167.3 147.5 142.4 140.3 134.1 133.2 131.8 131.5 129.6 129.3 128.6 127.8 127.0 126.2 121.3 116.8
77.4 77.1 76.8 68.3 60.4 53.7 41.0 21.6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Ph
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Ph
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NH
O
Br
3am
2.0 2.1 7.0 2.1 6.1
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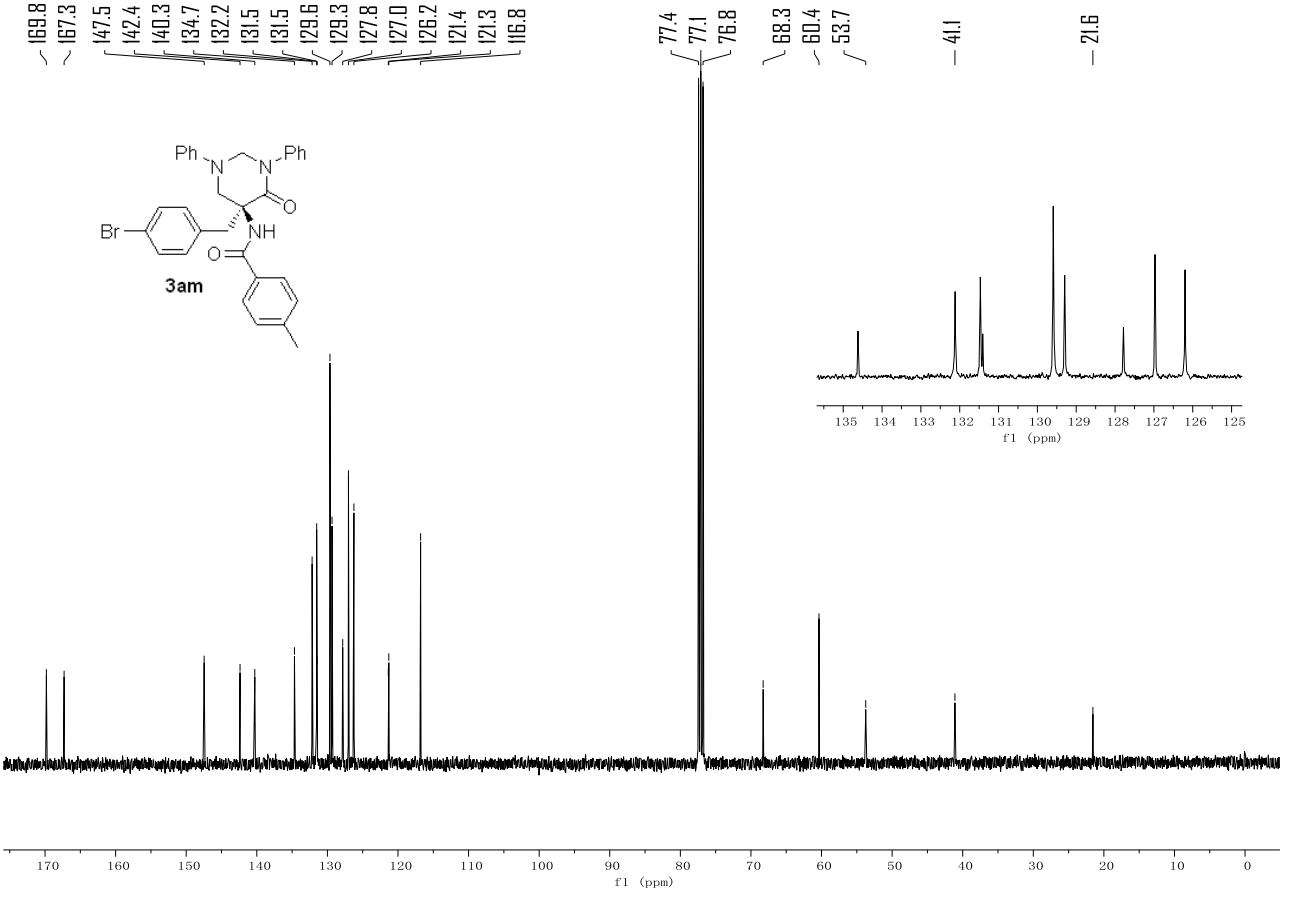
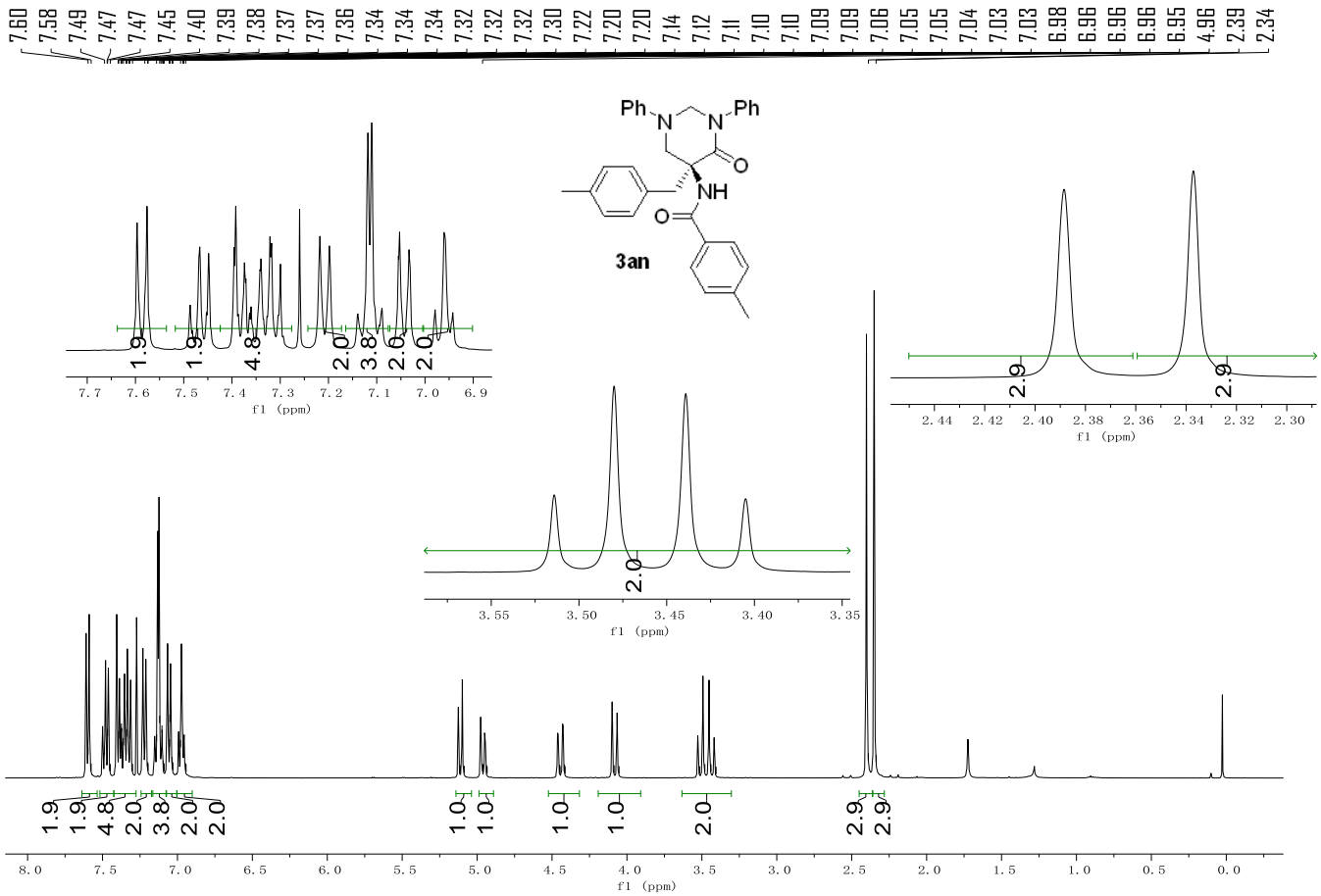
f1 (ppm)
2.0 2.1 7.0 2.1 6.1 2.0 1.0 1.0 2.1 3.0
f1 (ppm)


169.8 167.3 147.5 142.4 140.3 134.7 132.2 131.5 131.5 129.6 129.3 127.8 127.0 126.2 121.4 121.3 116.8
77.4 77.1 76.8 68.3 60.4 53.7 41.1 21.6
Ph
N
N
Ph
O
NH
O
Br
3am
f1 (ppm)
f1 (ppm)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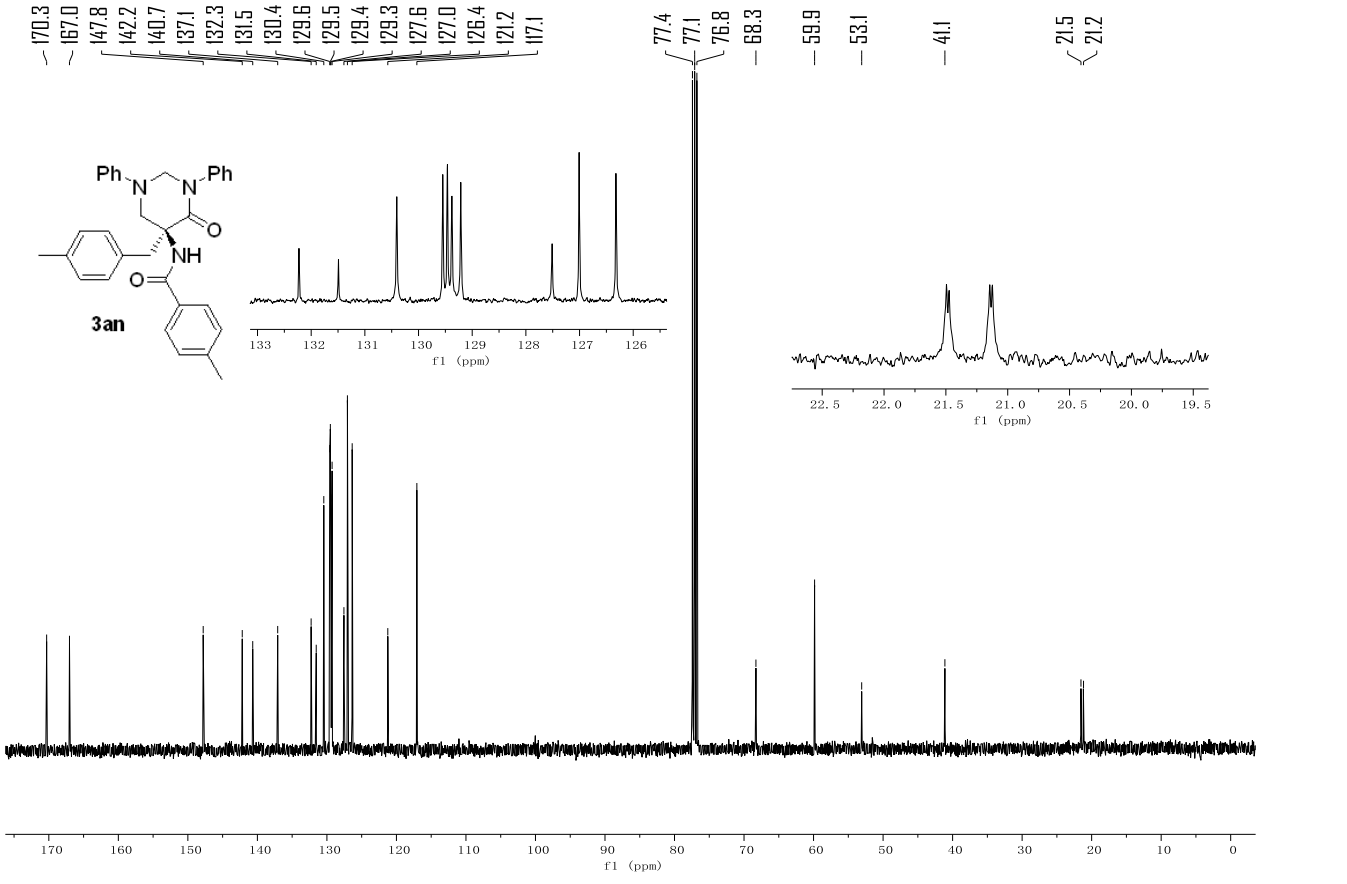
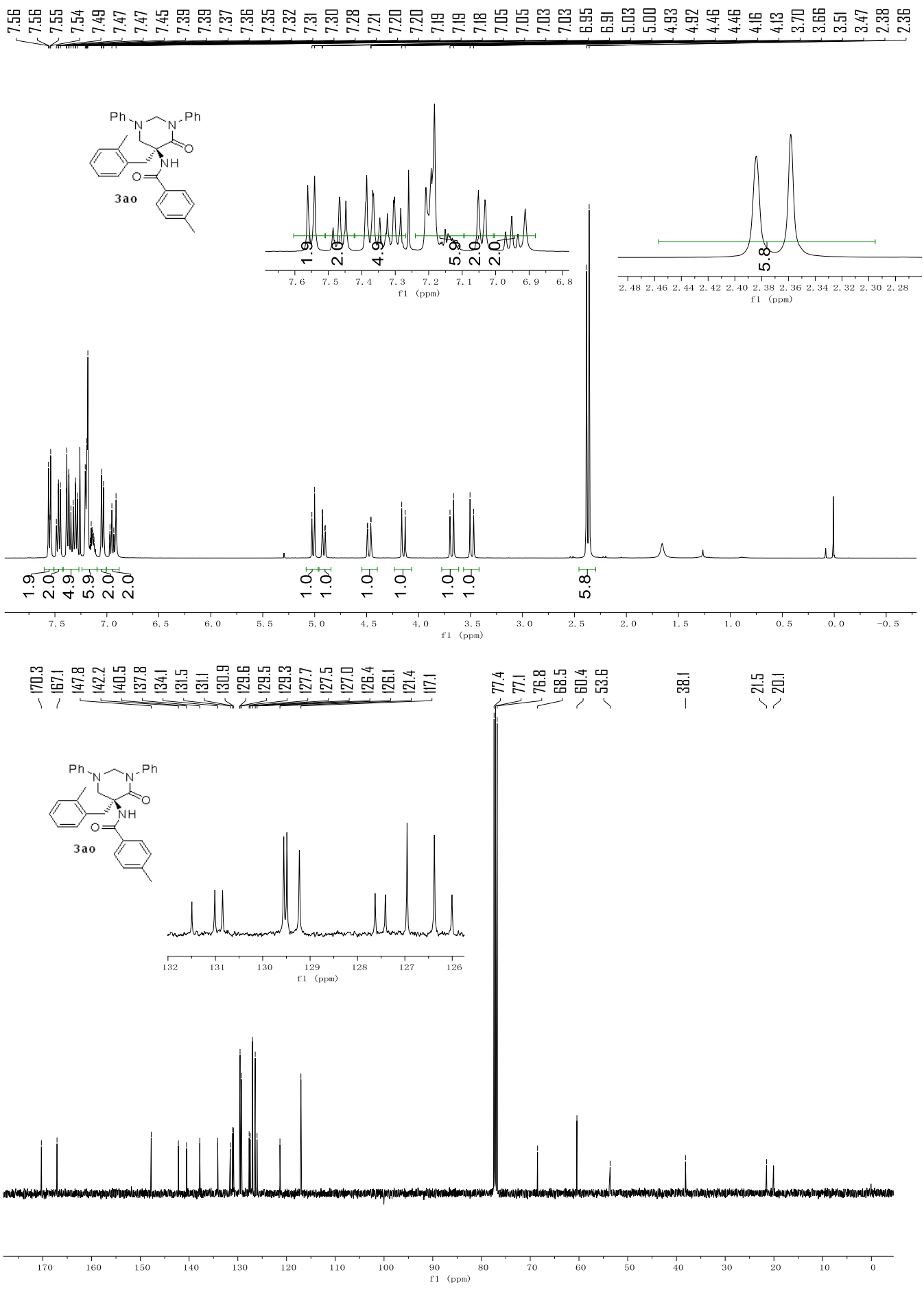
Ph N N Ph O NH O
3an
1.9 1.9 4.8 2.0 3.8 2.6 2.0
2.9 2.9
2.0
1.9 1.9 4.8 2.0 3.8 2.0 2.0 1.0 1.0 1.0 1.0 2.0 2.9 2.9
f1 (ppm)


170.3 167.0 147.8 142.2 140.7 137.1 132.3 131.5 130.4 129.6 129.5 129.4 129.3 127.6 127.0 126.4 121.2 117.1
77.4 77.1 76.8 68.3 59.9 53.1 41.1 21.5 21.2
Ph N N Ph O NH O
3an
f1 (ppm)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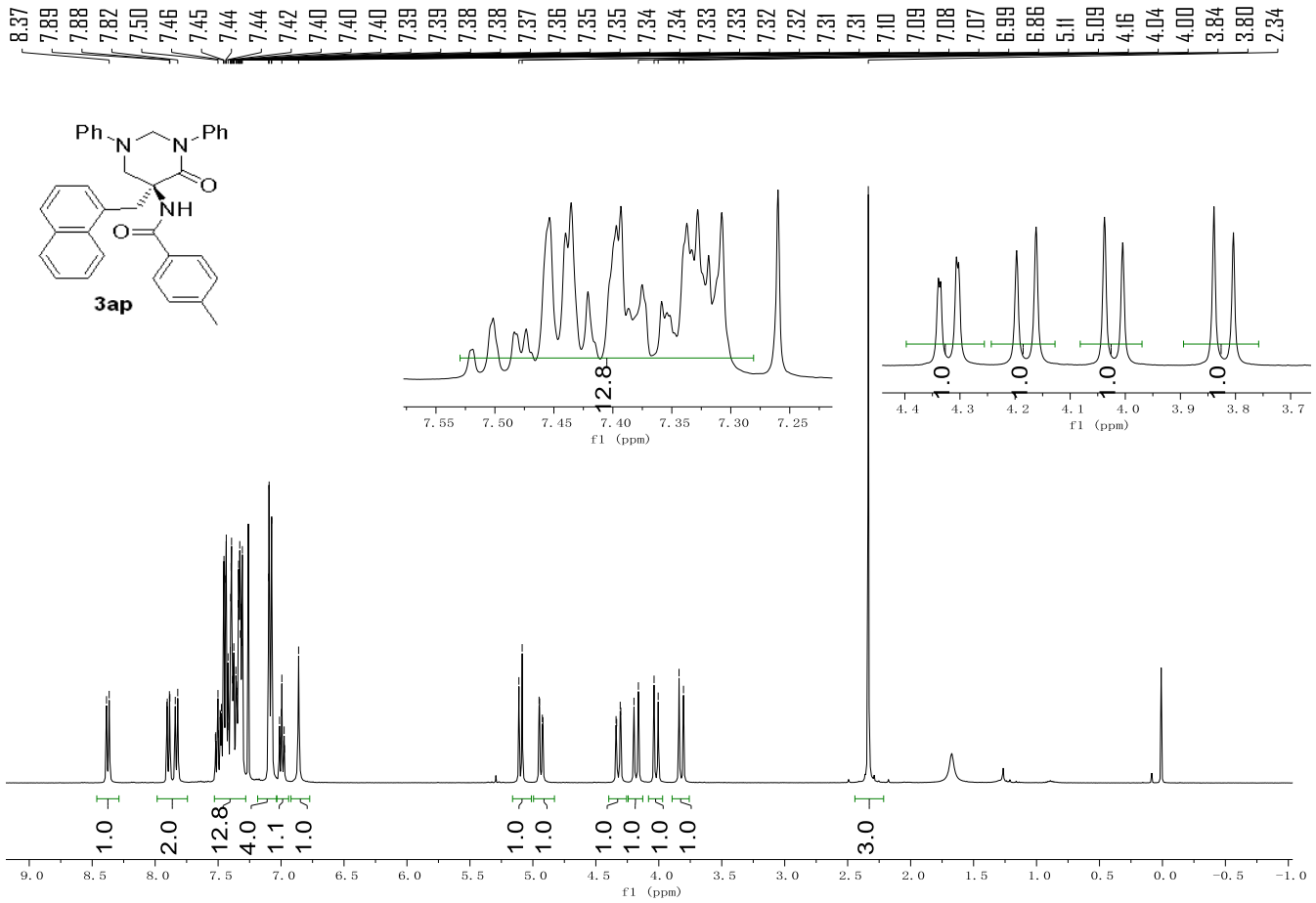
Ph N N Ph O NH O
3ao
1.9 2.0 4.9 5.9 2.0 2.0
1.0 1.0 1.0 1.0 1.0 1.0
5.8
f1 (ppm)
170.3 167.1 147.8 142.2 140.5 137.8 134.1 131.5 131.1 130.9 129.6 129.5 129.3 127.7 127.5 127.0 126.4 126.1 121.4 117.1
77.4 77.1 76.8 68.5 60.4 53.6 38.1 21.5 20.1
f1 (ppm)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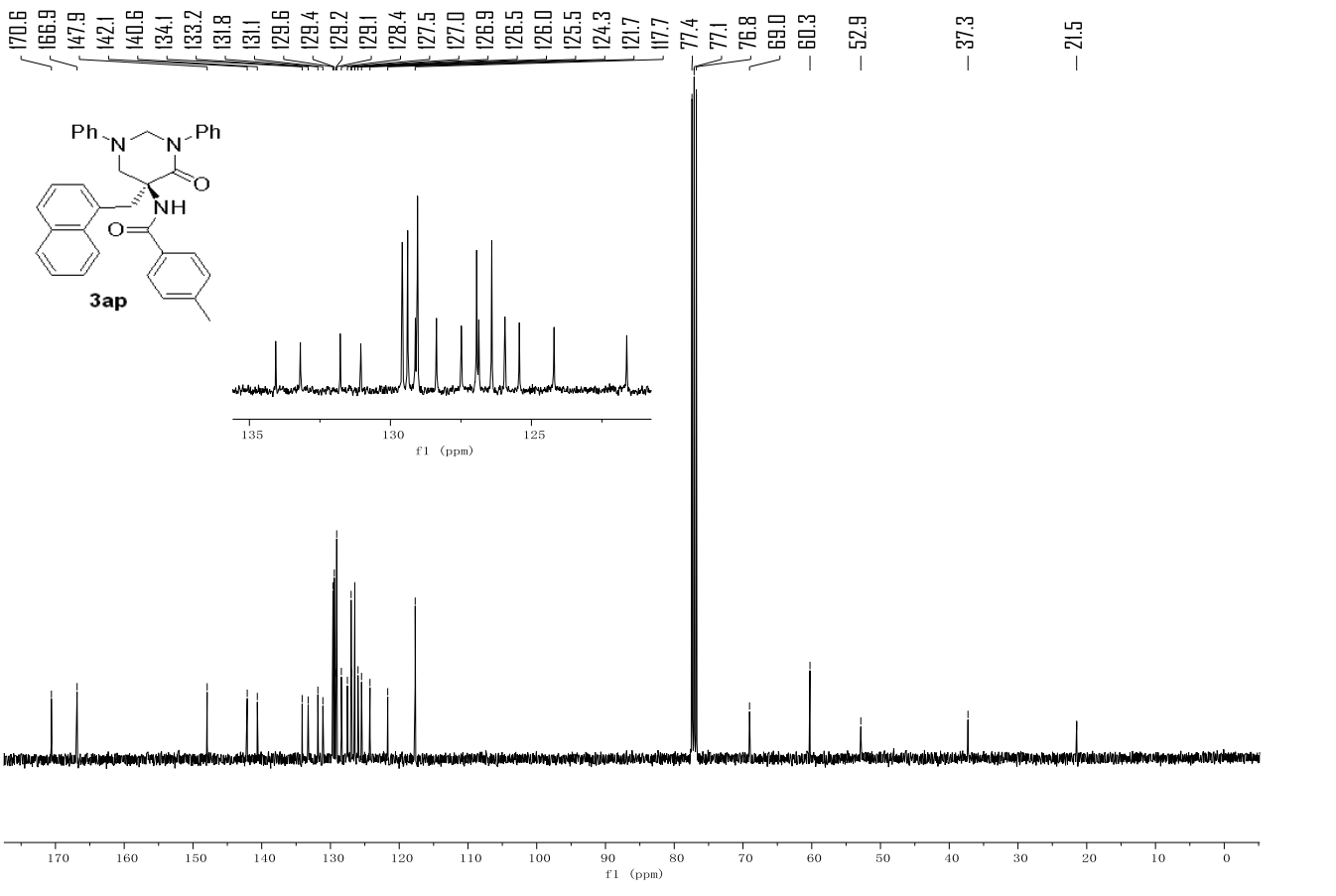
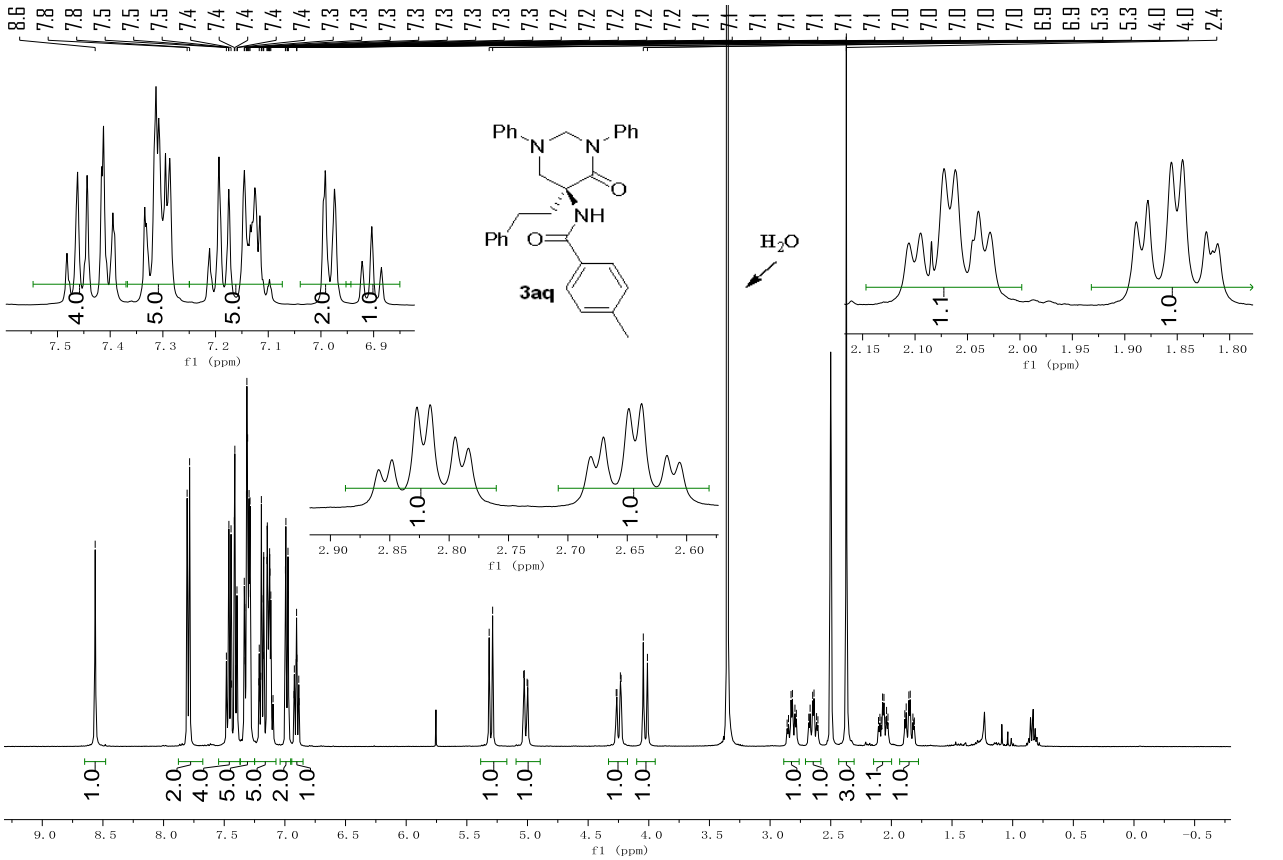
Ph N N Ph O NH O
3ap
12.8
1.0 1.0 1.0 1.0
1.0 2.0 12.8 4.0 1.1 1.0 1.0 1.0 1.0 1.0 1.0 1.0 3.0
f1 (ppm)


170.6 166.9 147.9 142.1 140.6 134.1 133.2 131.8 131.1 129.6 129.4 129.2 129.1 128.4 127.5 127.0 126.9 126.5 126.0 125.5 124.3 121.7 117.7 77.4 77.1 76.8 69.0 60.3 52.9 37.3 21.5
Ph N N Ph O NH O
3ap
f1 (ppm)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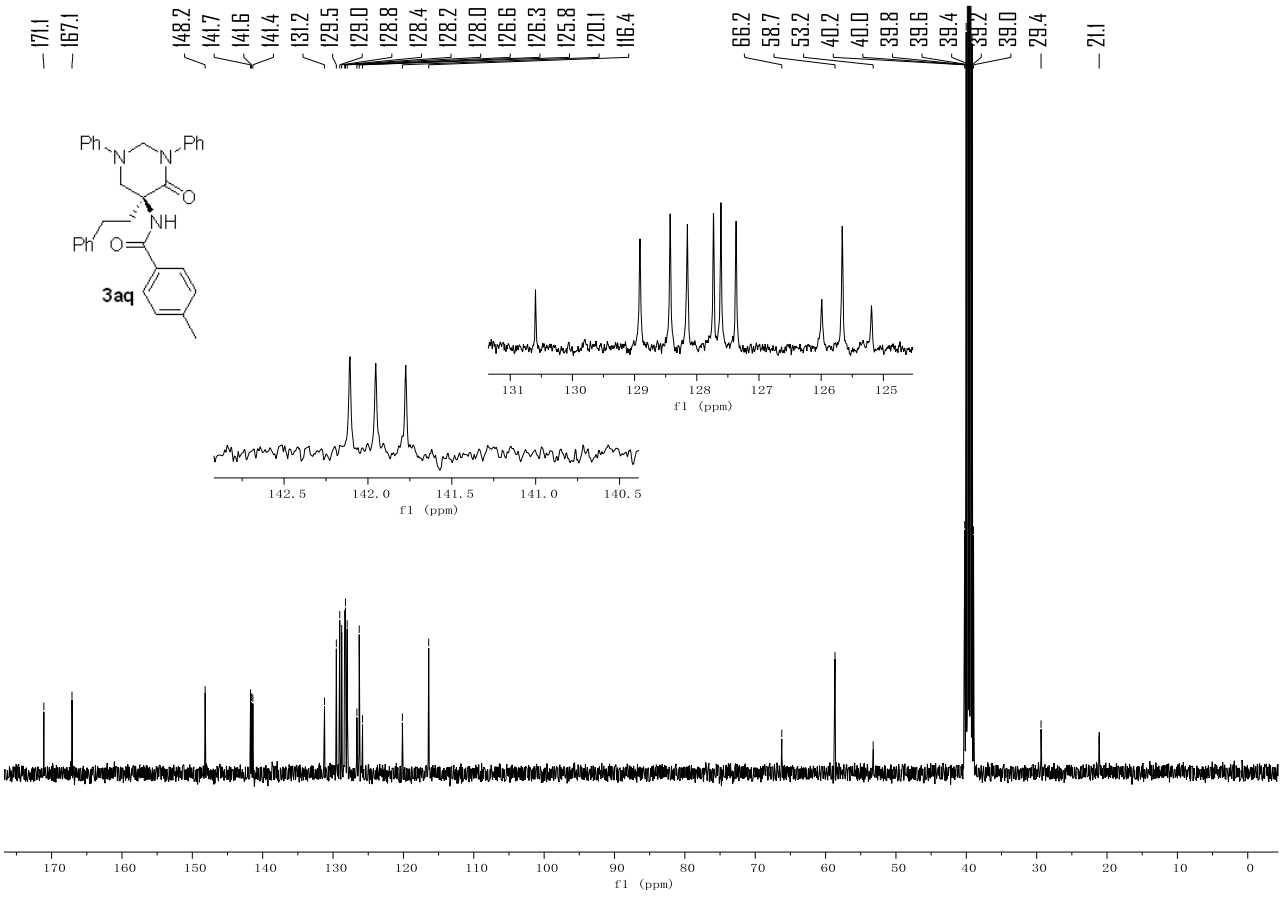
H2O
f1 (ppm)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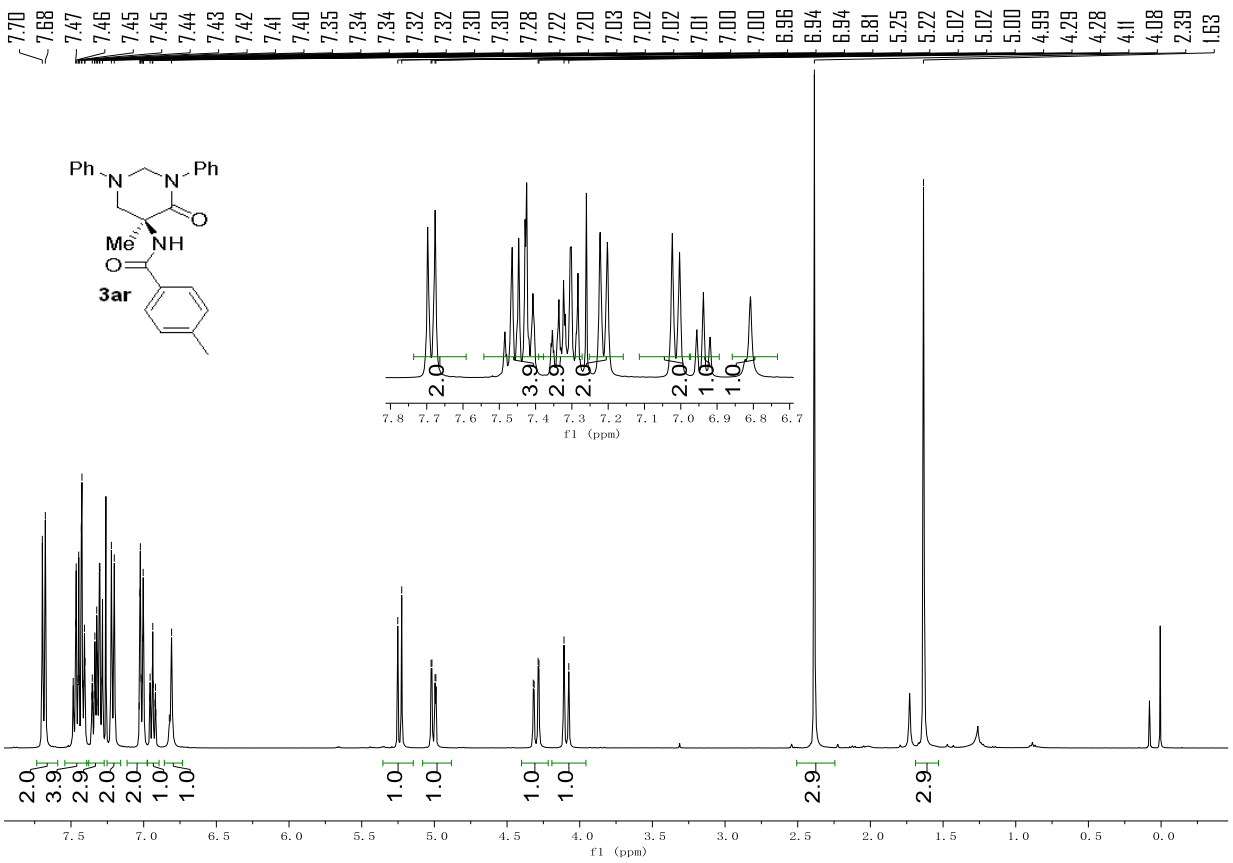
f1 (ppm)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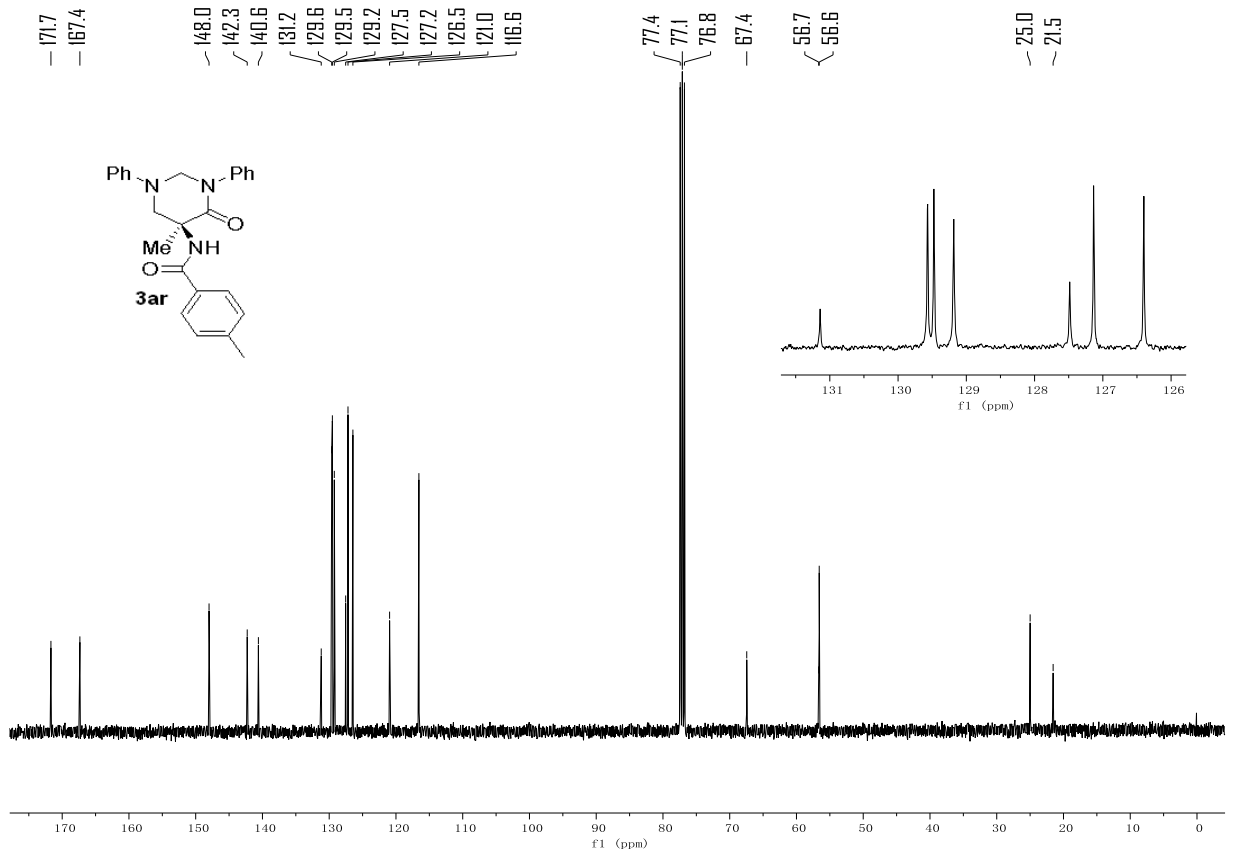
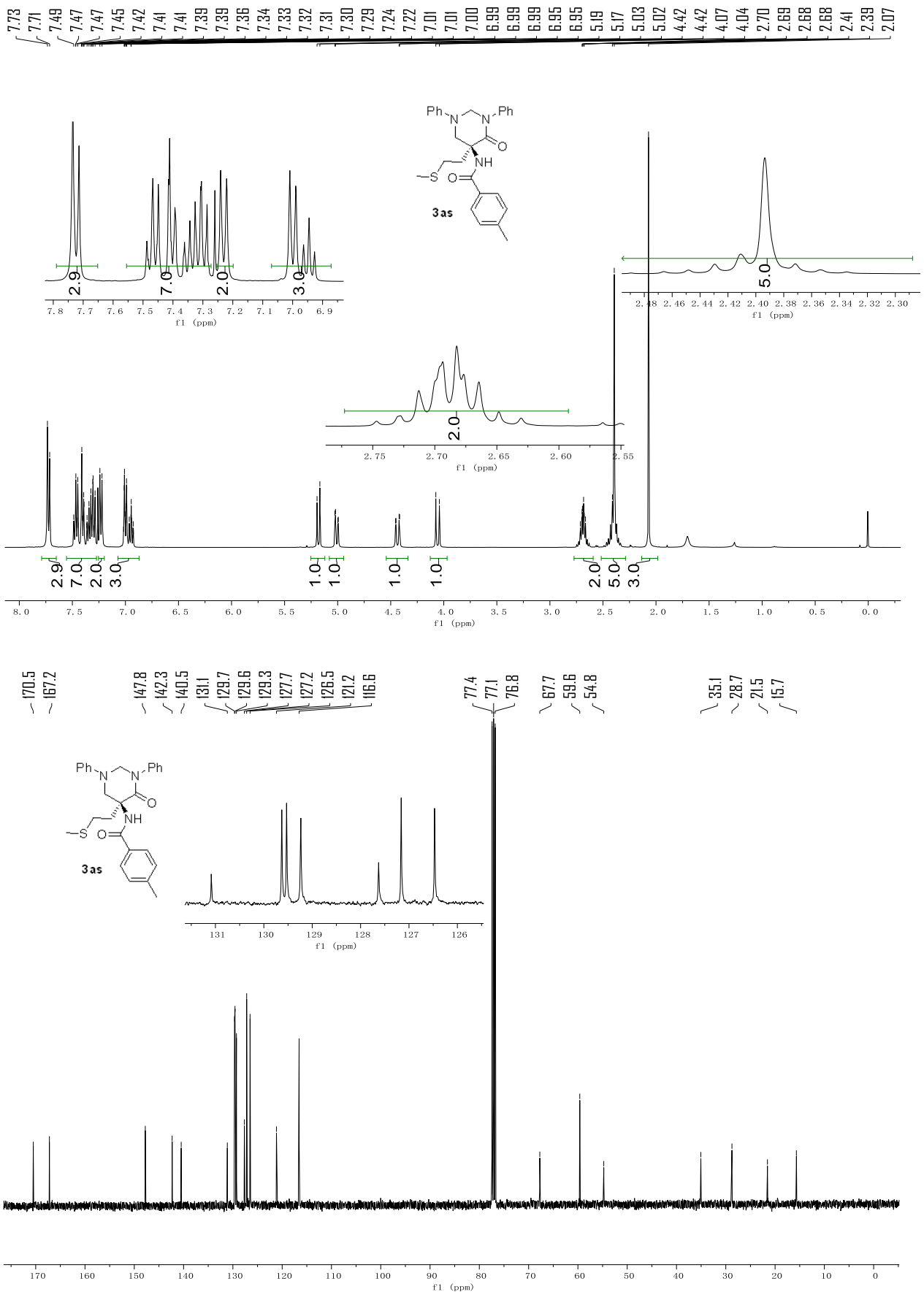
Ph N N Ph O Me NH O
3ar
2.0 3.9 2.9 2.0 2.0 1.0 1.0 1.0 1.0 1.0 1.0 2.9 2.9
f1 (ppm)


171.7 167.4 148.0 142.3 140.6 131.2 129.6 129.5 129.2 127.5 127.2 126.5 121.0 116.6 77.4 77.1 76.8 67.4 56.7 56.6 25.0 21.5
Ph N N Ph O Me NH O
3ar
f1 (ppm)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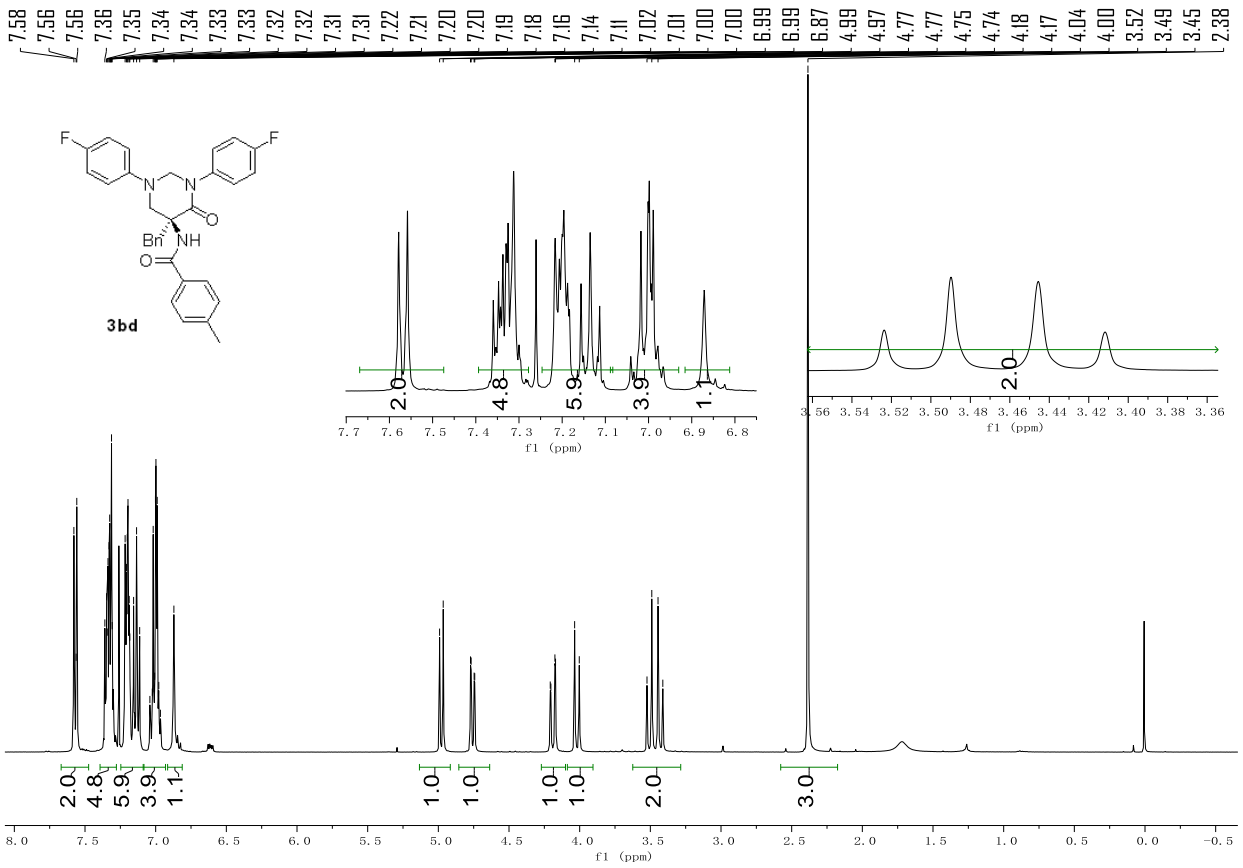
Ph N N Ph O NH S O
3as
2.9 7.0 2.0 3.0
5.0
2.0
1.0 1.0 1.0 1.0 2.0 5.0 3.0
f1 (ppm)

170.5 167.2 147.8 142.3 140.5 131.1 129.7 129.6 129.3 127.7 127.2 126.5 121.2 116.6 77.4 77.1 76.8 67.7 59.6 54.8 35.1 28.7 21.5 15.7
Ph N N Ph O NH S O
3as
f1 (ppm)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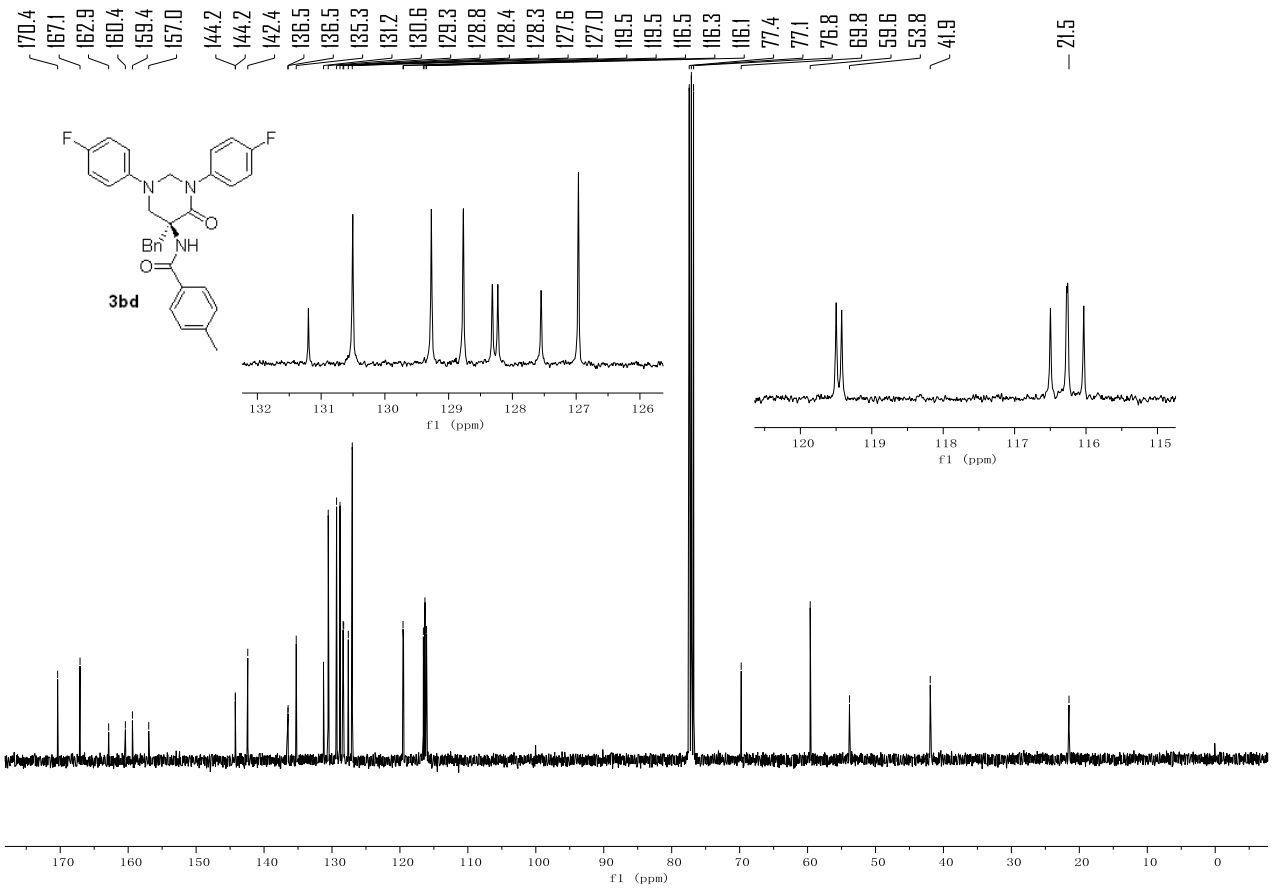
F
F
N
N
O
Bn
NH
O
3bd
2.0 4.8 5.9 3.9 1.1
2.0
f1 (ppm)
2.0 4.8 5.9 3.9 1.1 1.0 1.0 1.0 1.0 2.0 3.0
f1 (ppm)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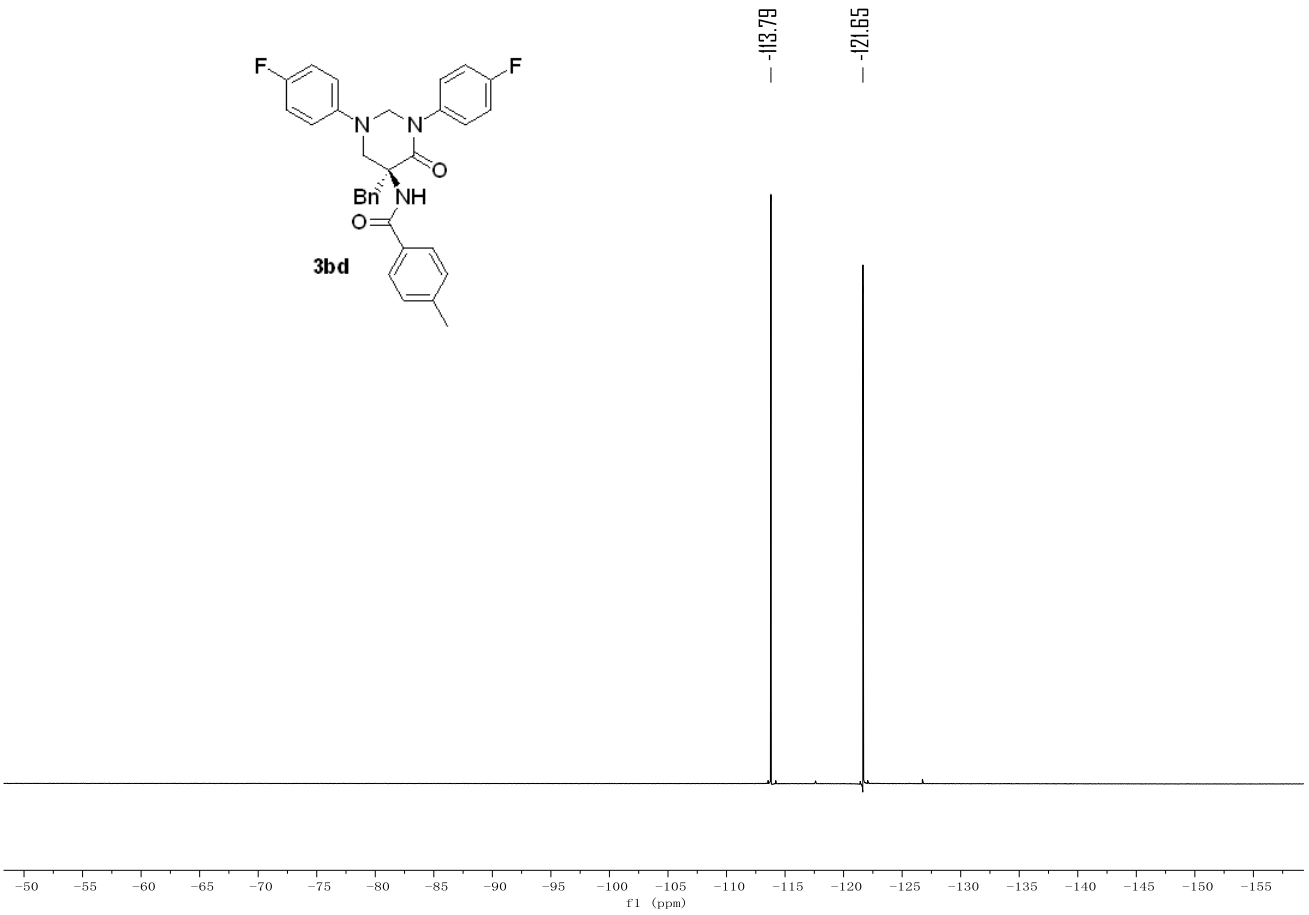
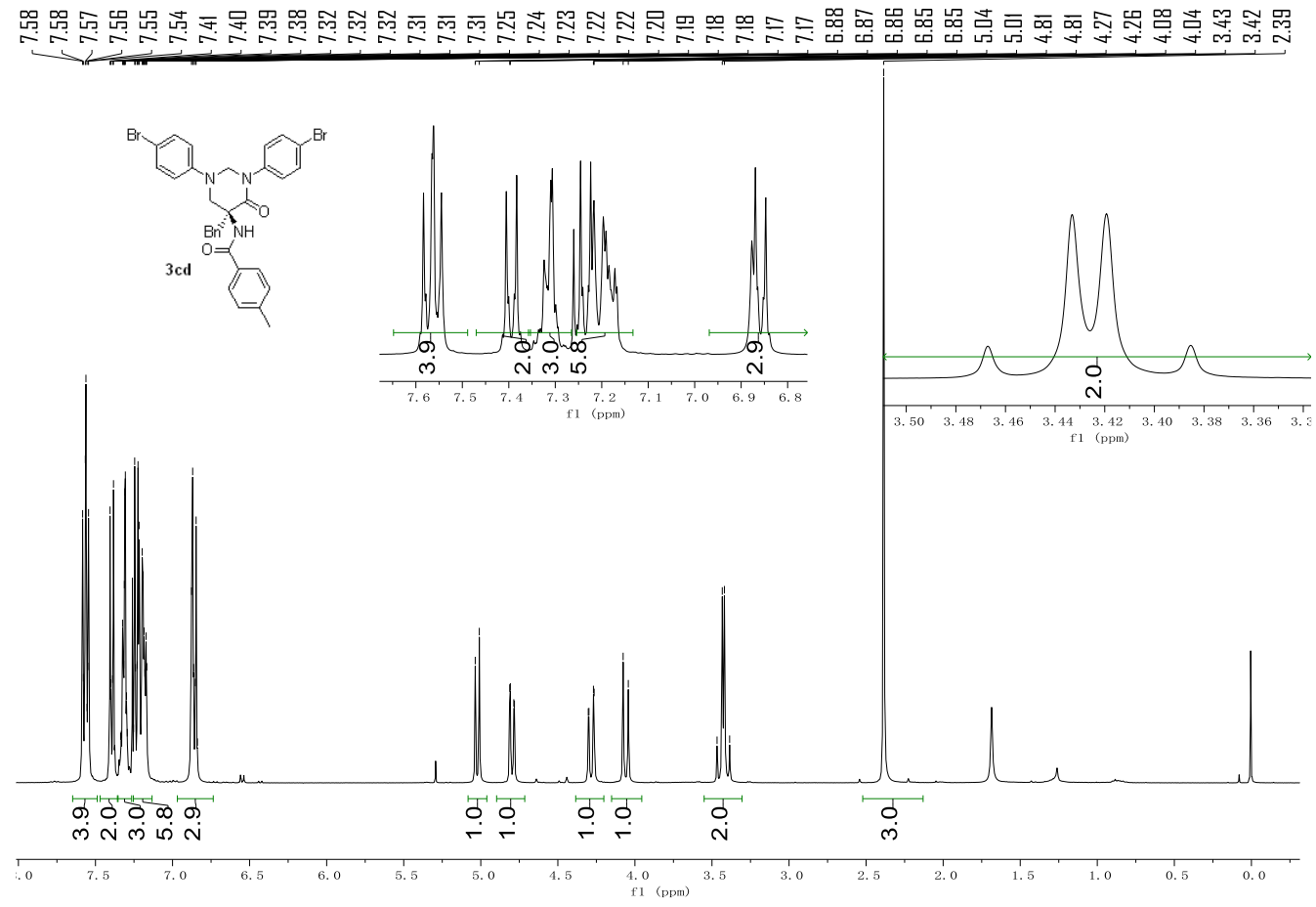
F
F
N
N
O
Bn
NH
O
3bd
f1 (ppm)
f1 (ppm)
f1 (ppm)


F
F
N
N
O
Bn
NH
O
3bd
-113.79
-121.65
-50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150 -155
f1 (ppm)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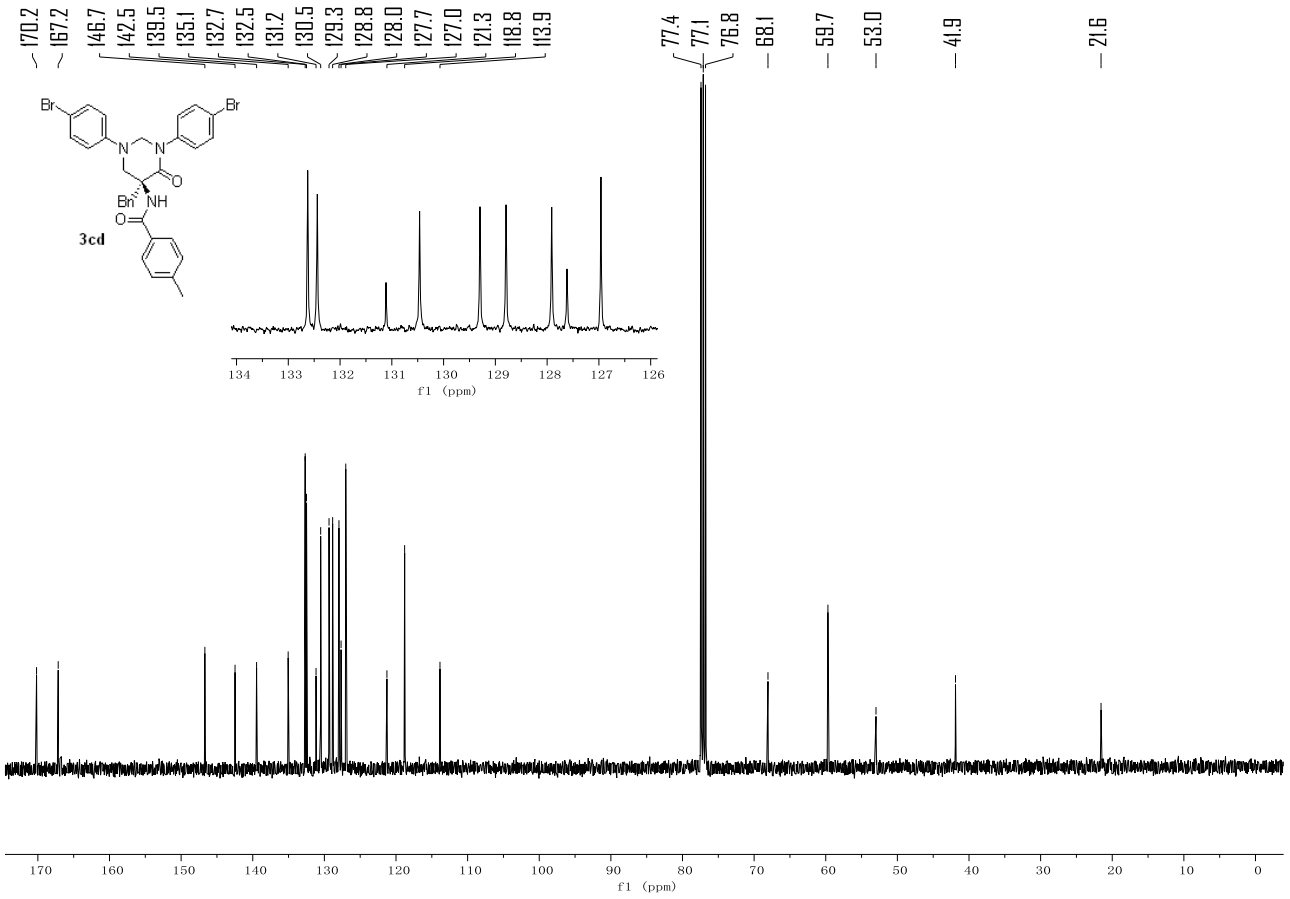
Br
Br
N
N
O
Bn
NH
O
3cd
3.9 2.0 3.0 5.8 2.9
7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8
f1 (ppm)
2.0
3.50 3.48 3.46 3.44 3.42 3.40 3.38 3.36
f1 (ppm)
3.9 2.0 3.0 5.8 2.9 1.0 1.0 1.0 1.0 2.0 3.0
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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135.1
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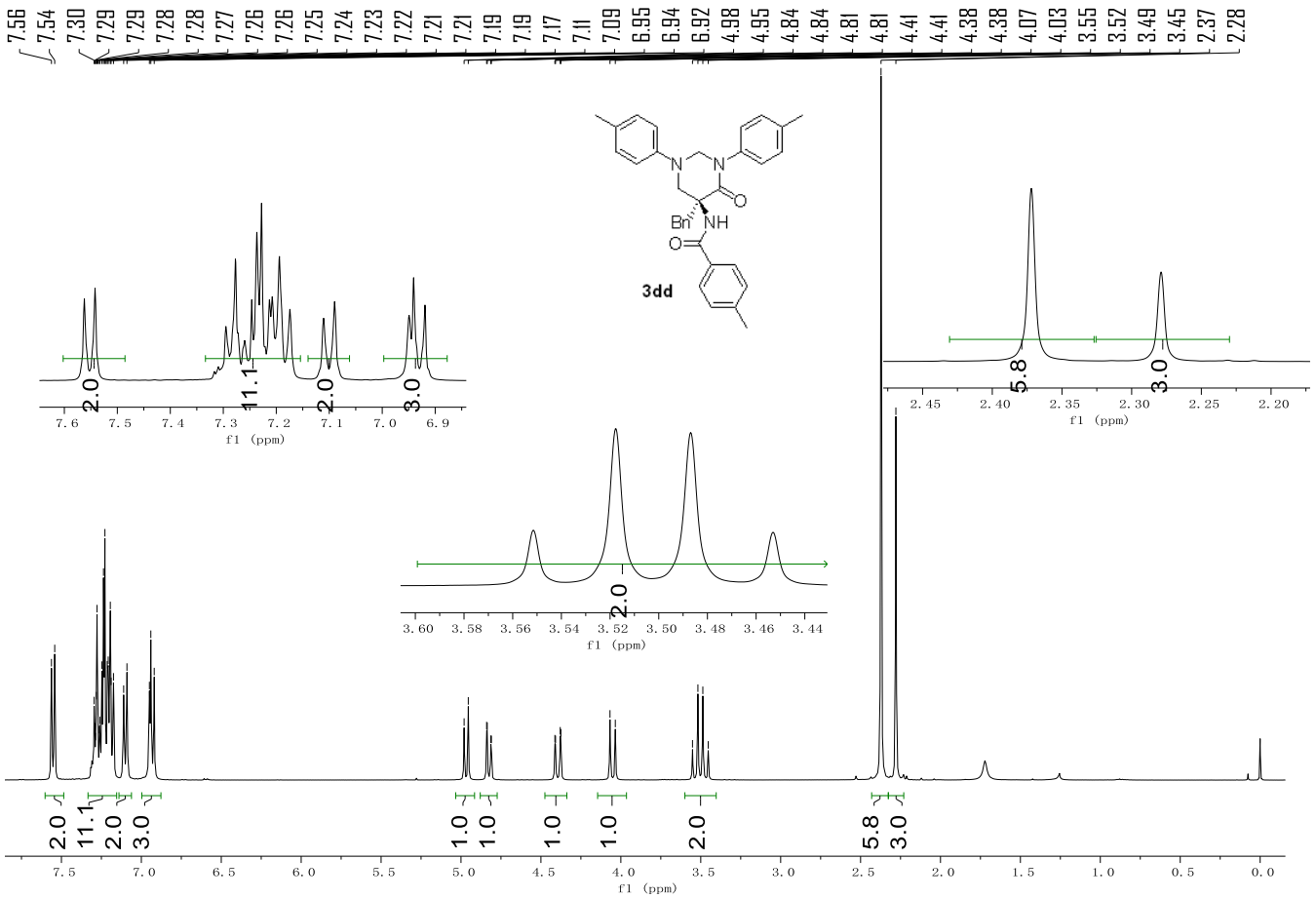
132.5
131.2
130.5
129.3
128.8
128.0
127.7
127.0
121.3
118.8
113.9
77.4
77.1
76.8
68.1
59.7
53.0
41.9
21.6
Br
Br
N
N
O
Bn
NH
O
3cd
134 133 132 131 130 129 128 127 126
f1 (ppm)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.17
7.11
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4.84
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4.81
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4.41
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4.38
4.07
4.03
3.55
3.52
3.49
3.45
2.37
2.28
N
N
O
Bn
NH
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3.0
7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9
f1 (ppm)
5.8
3.0
2.45 2.40 2.35 2.30 2.25 2.20
f1 (ppm)
2.0
3.60 3.58 3.56 3.54 3.52 3.50 3.48 3.46 3.44
f1 (ppm)
2.0
11.1
2.0
3.0
1.0
1.0
1.0
1.0
2.0
5.8
3.0
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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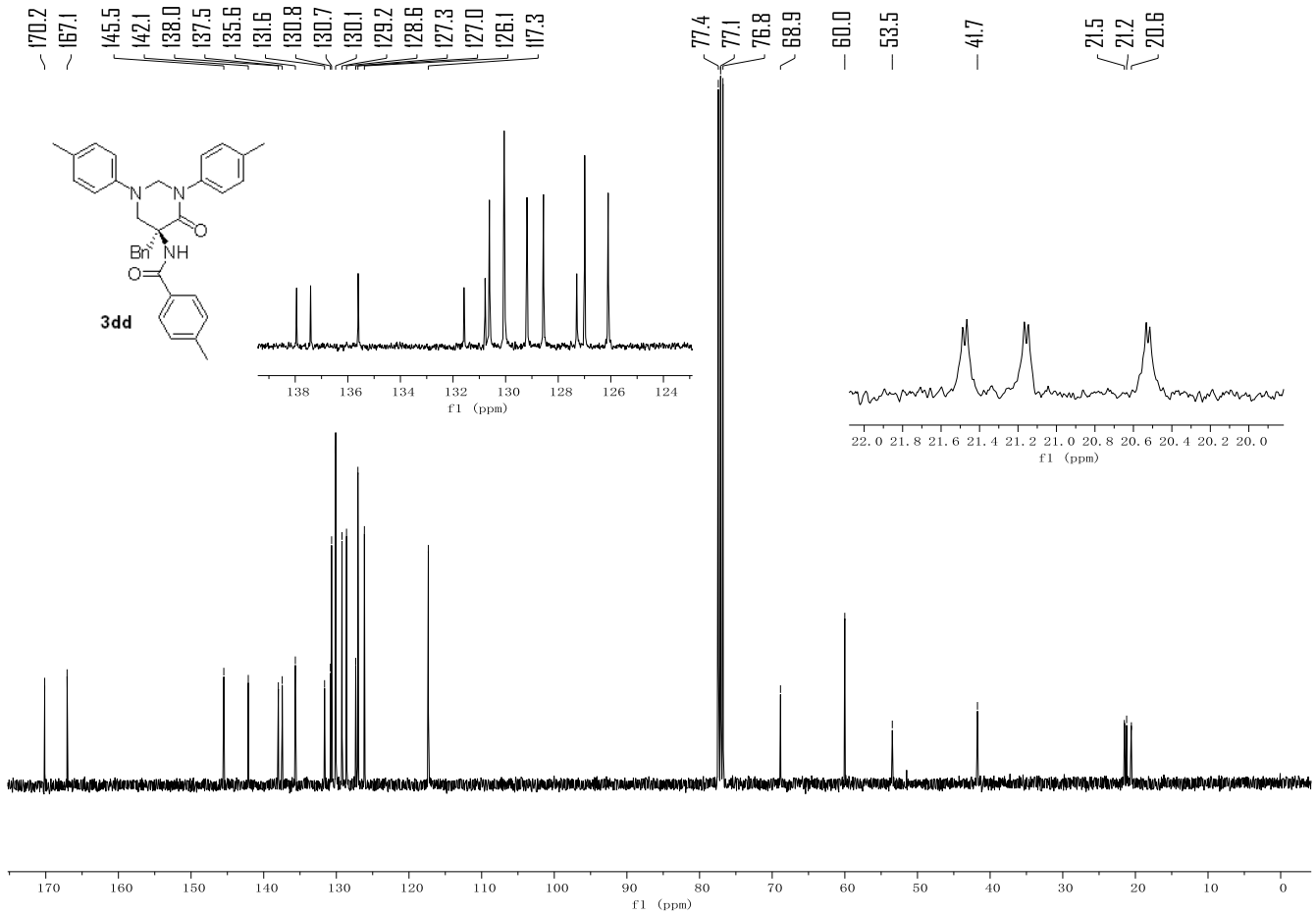
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130.8
130.7
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129.2
128.6
127.3
127.0
126.1
117.3
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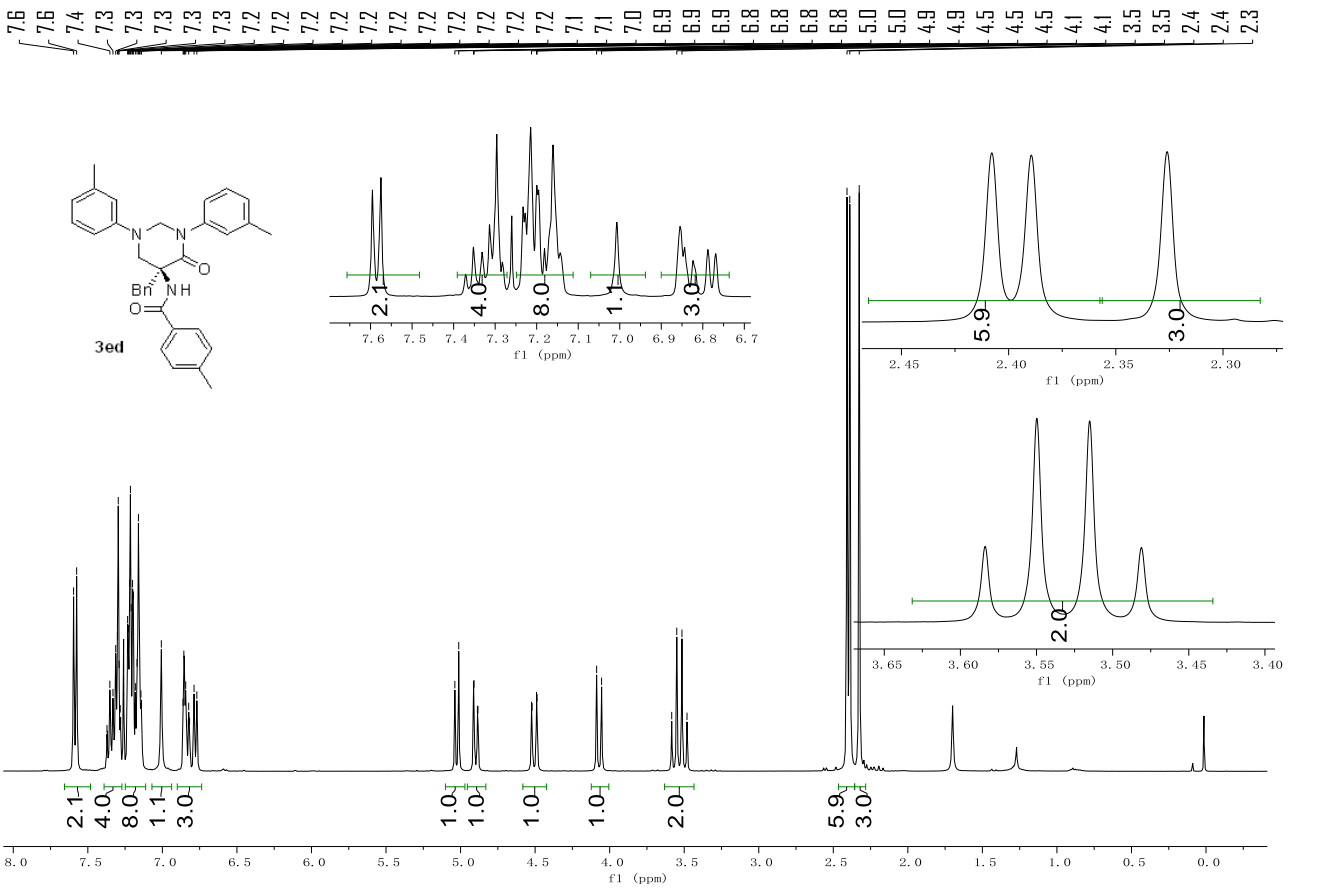
77.1
76.8
68.9
60.0
53.5
41.7
21.5
21.2
20.6
3dd
f1 (ppm)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7.2
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7.2
7.2
7.2
7.2
7.2
7.1
7.1
7.0
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6.9
6.9
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6.8
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5.0
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4.9
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3.0
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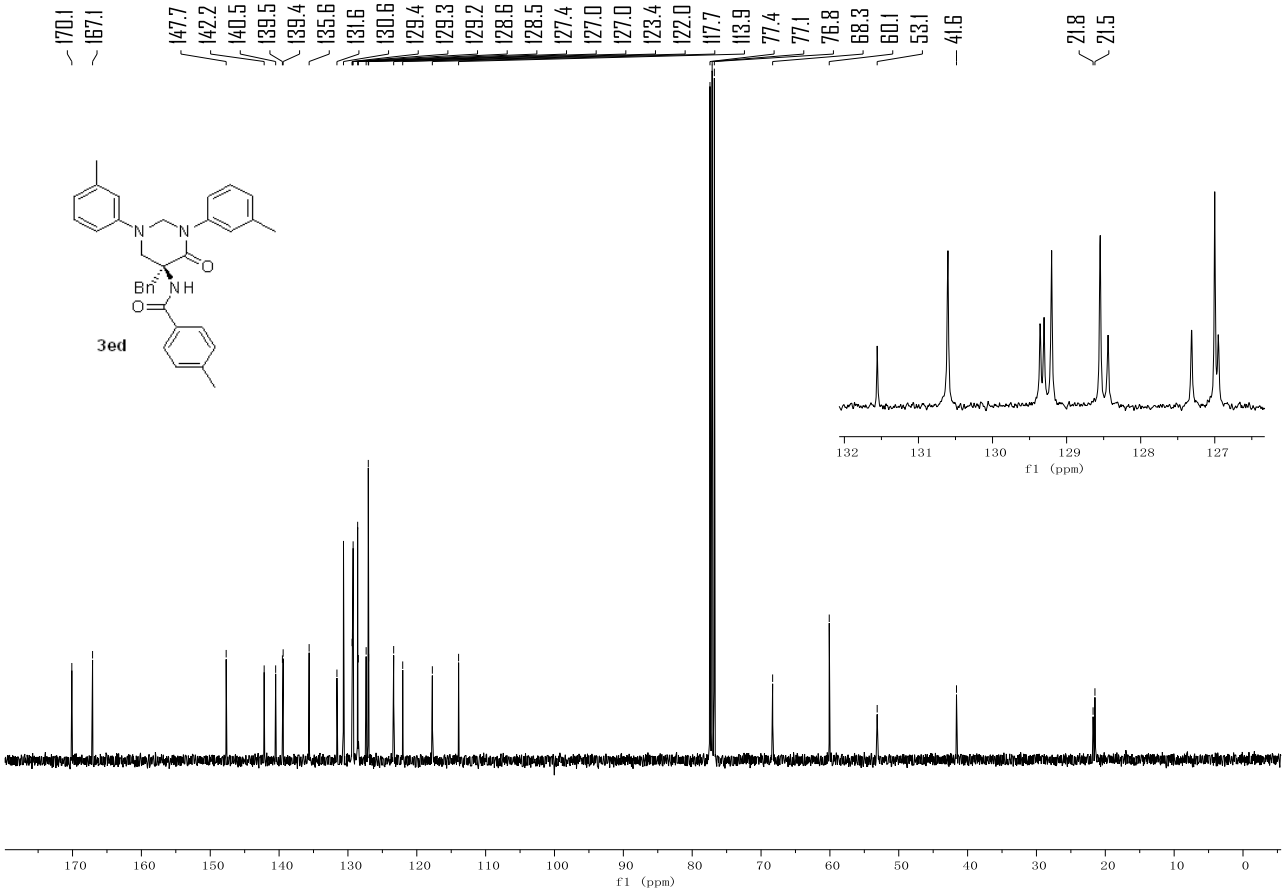
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127.4
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127.0
123.4
122.0
117.7
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53.1
41.6
21.8
21.5
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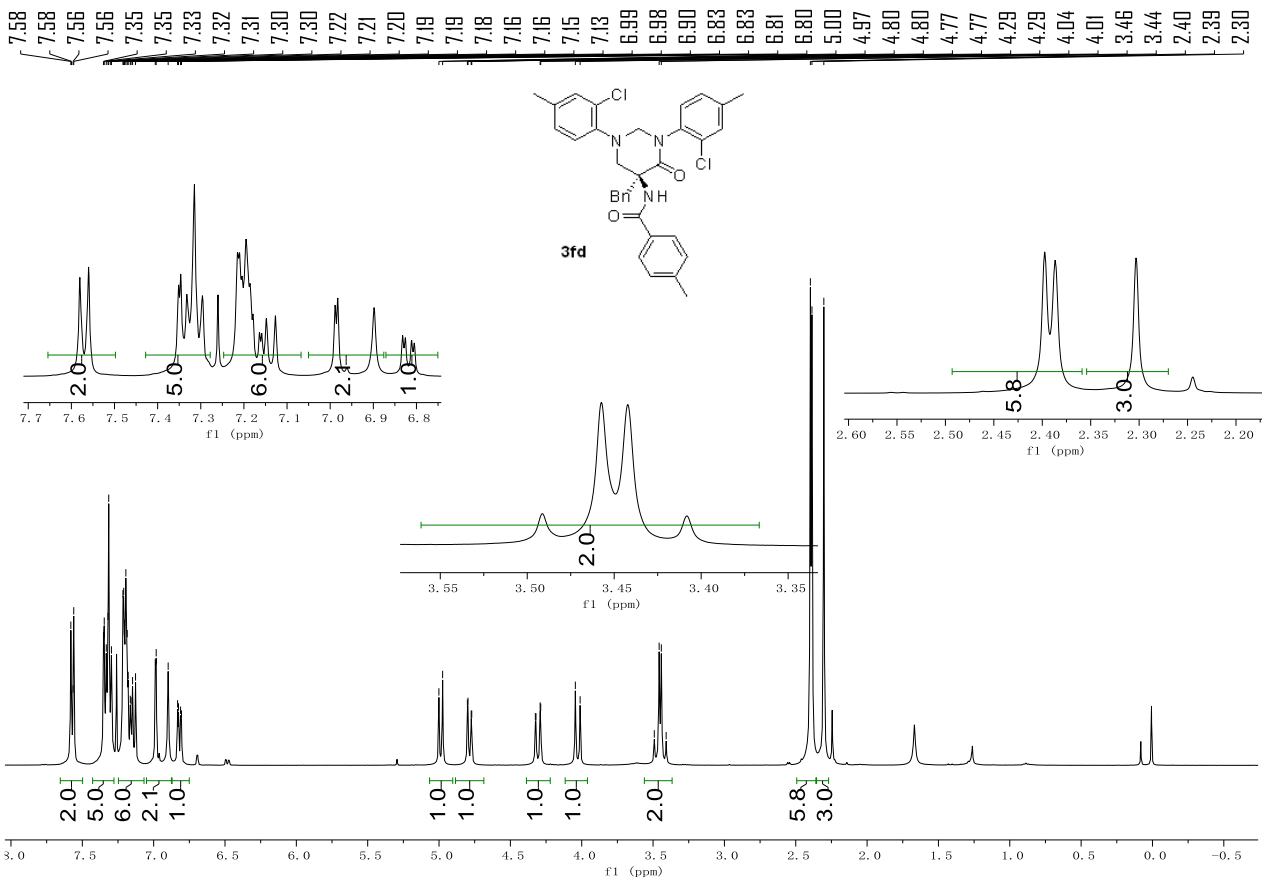
f1 (ppm)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7.19
7.19
7.18
7.16
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7.15
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6.98
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6.83
6.83
6.81
6.80
5.00
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4.80
4.80
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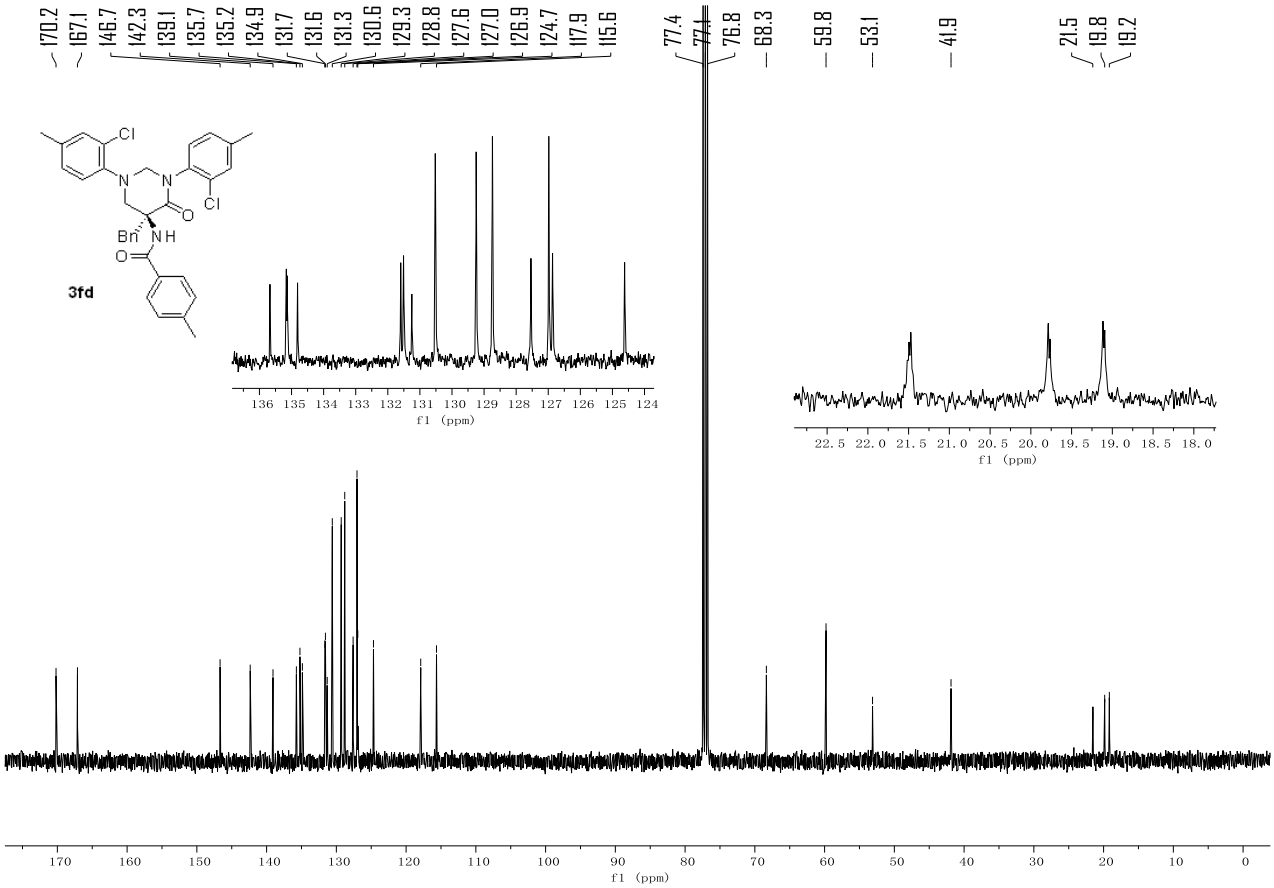
f1 (ppm)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127.6
127.0
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124.7
117.9
115.6
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21.5
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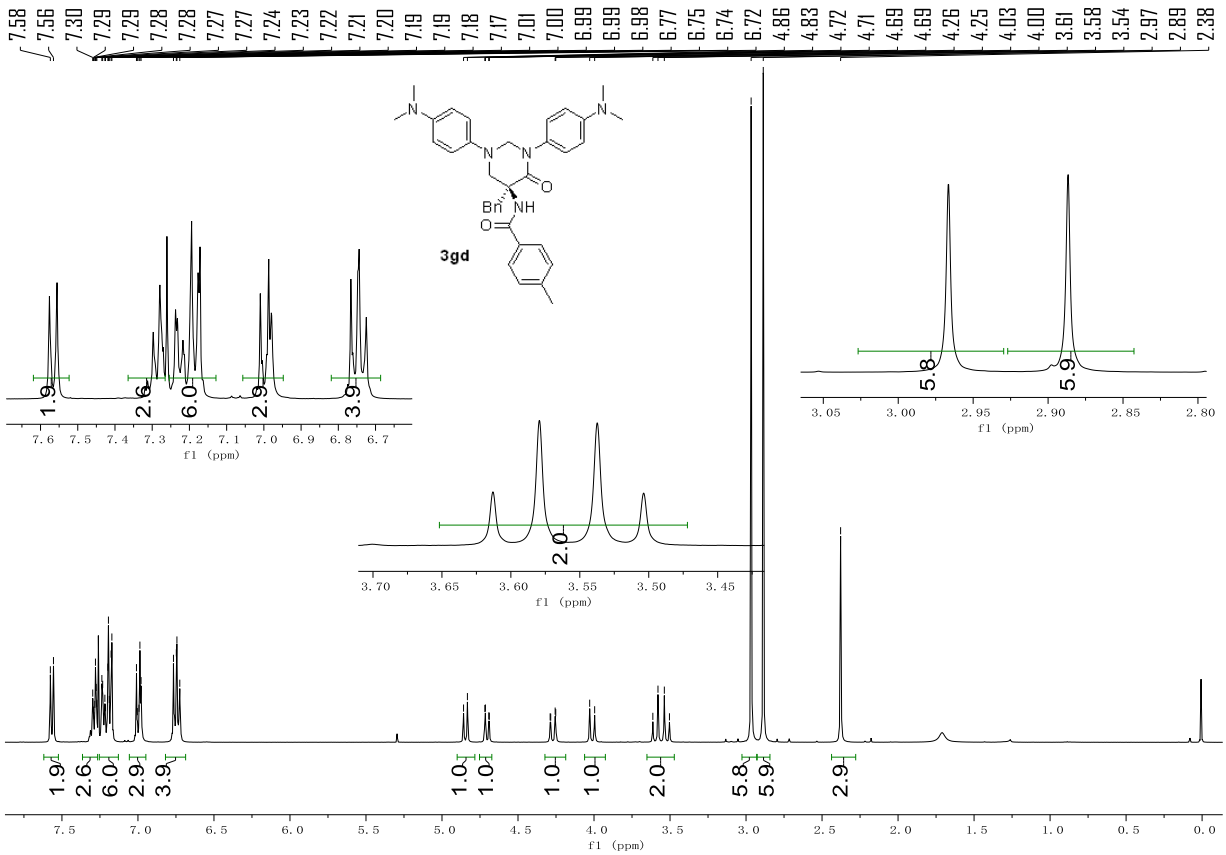
f1 (ppm)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6.75
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4.69
4.69
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4.25
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3.54
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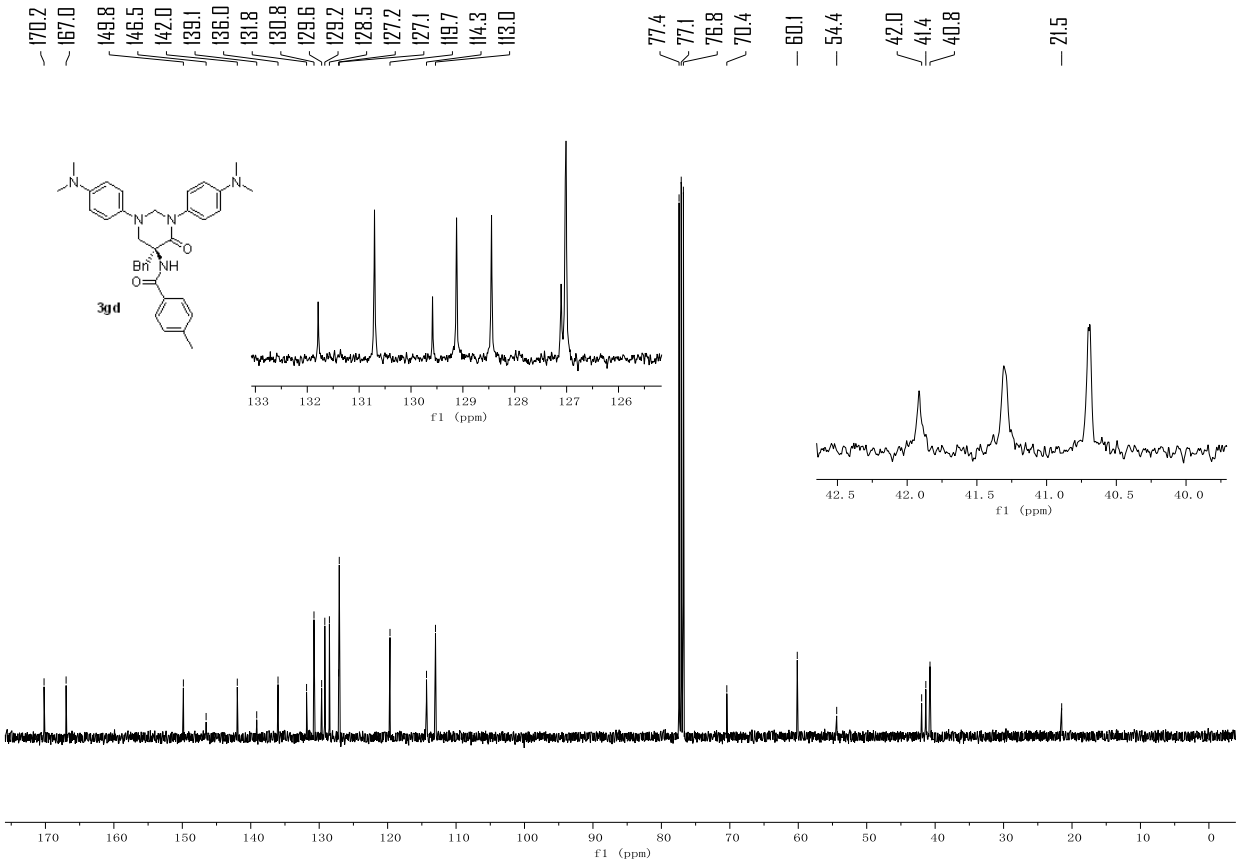
f1 (ppm)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127.2
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119.7
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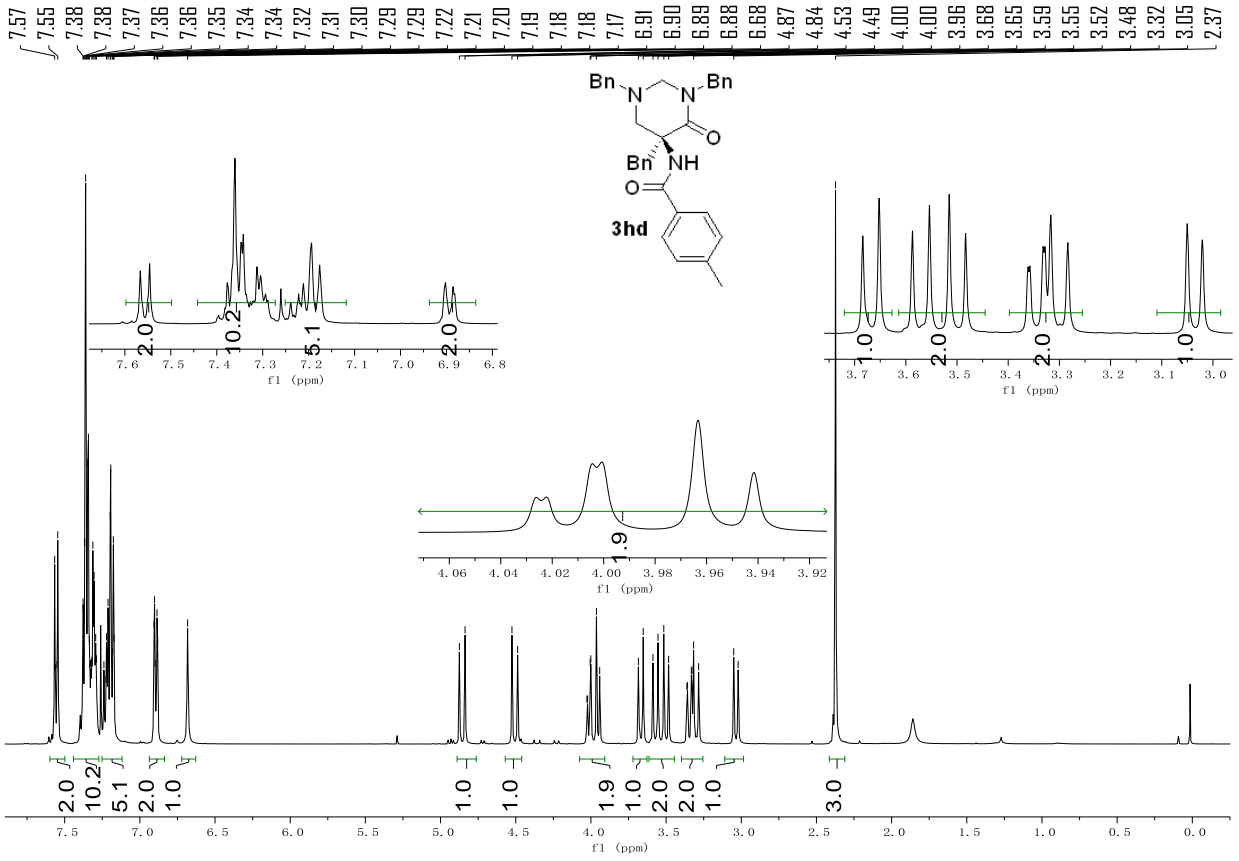
f1 (ppm)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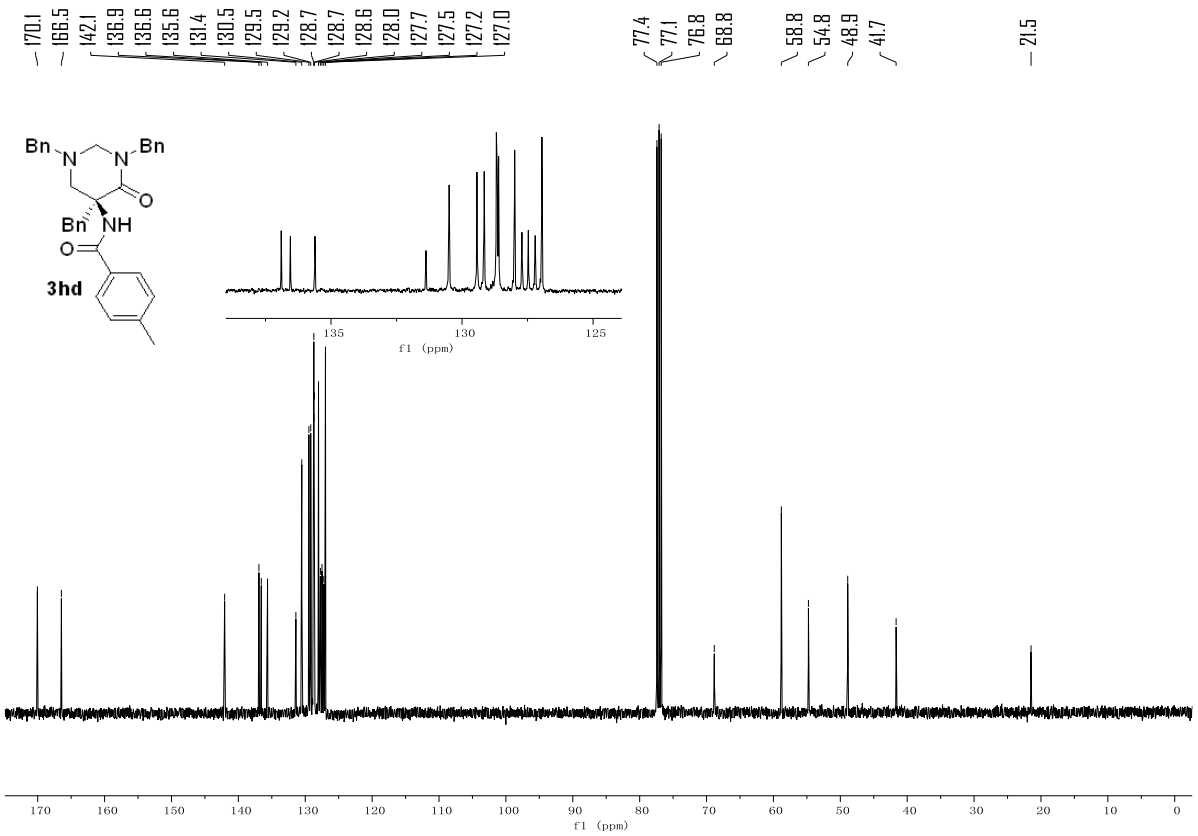
f1 (ppm)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76.8
68.8
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54.8
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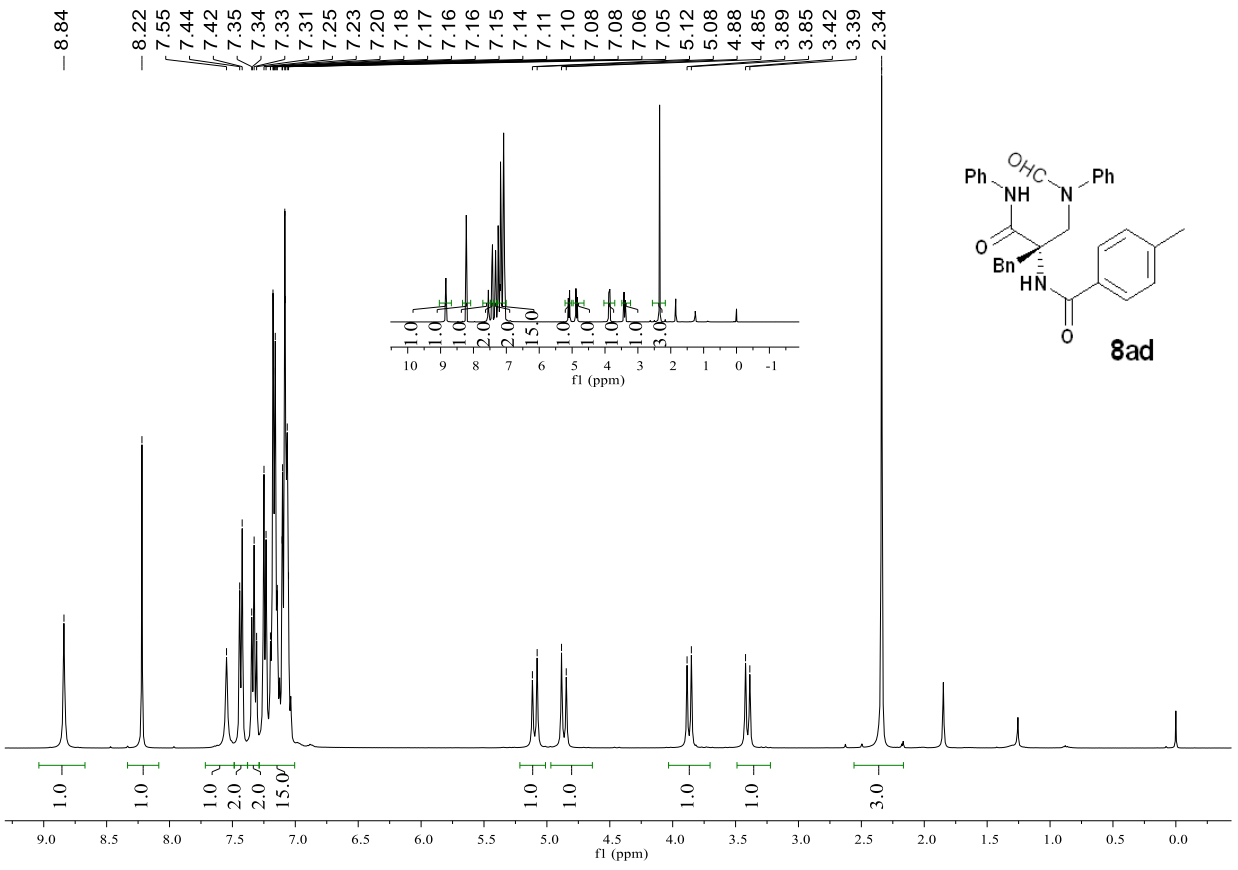
41.7
21.5
Bn
N
N
Bn
O
Bn
NH
O
3hd
f1 (ppm)
f1 (ppm)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7.16
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7.14
7.11
7.10
7.08
7.08
7.06
7.05
5.12
5.08
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4.85
3.89
3.85
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3.39
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Ph
NH
OHC
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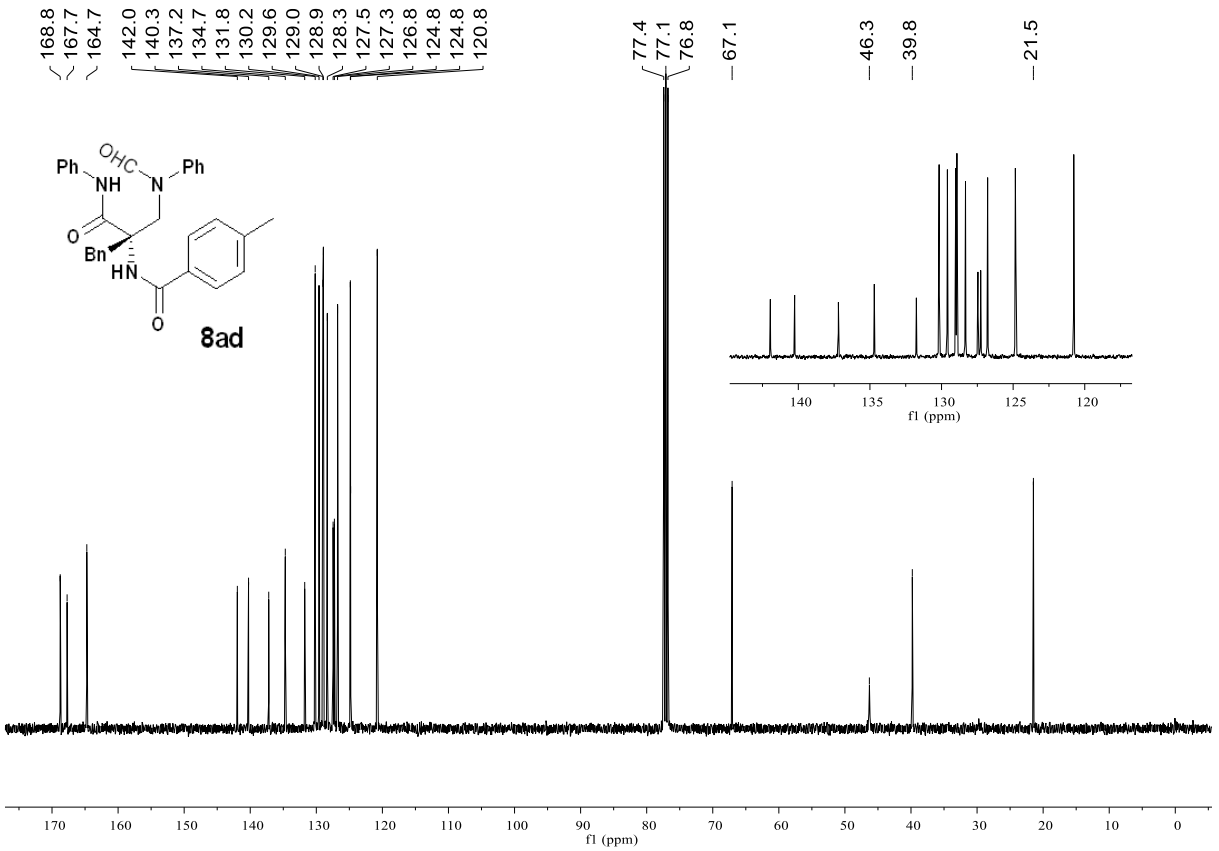
Ph
O
Bn
HN
O
8ad
f1 (ppm)
f1 (ppm)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128.9
128.3
127.5
127.3
126.8
124.8
124.8
120.8
77.4
77.1
76.8
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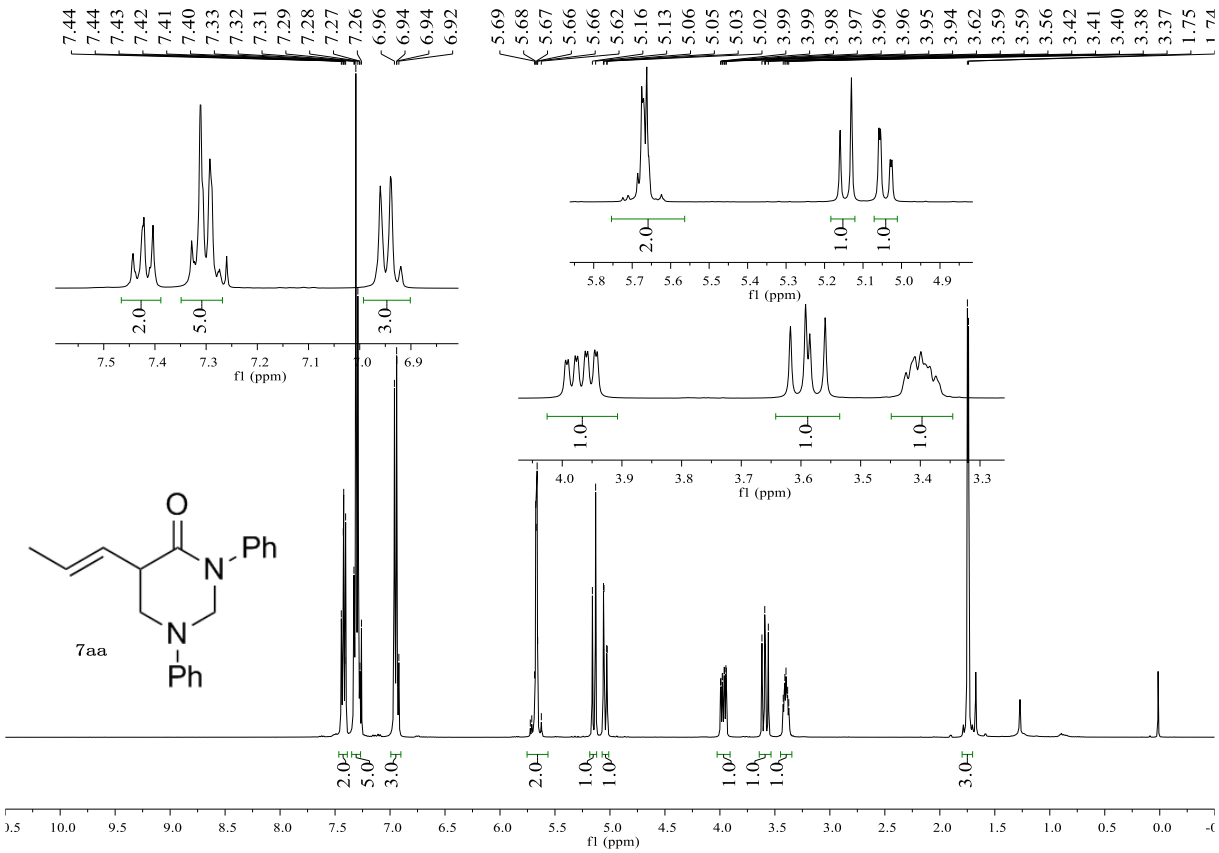
39.8
21.5
Ph
NH
OHC
N
Ph
O
Bn
HN
O
8ad
f1 (ppm)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3.0
2.0
1.0
1.0
1.0
1.0
1.0
1.0
1.0
1.0
1.0
2.0
5.0
3.0
2.0
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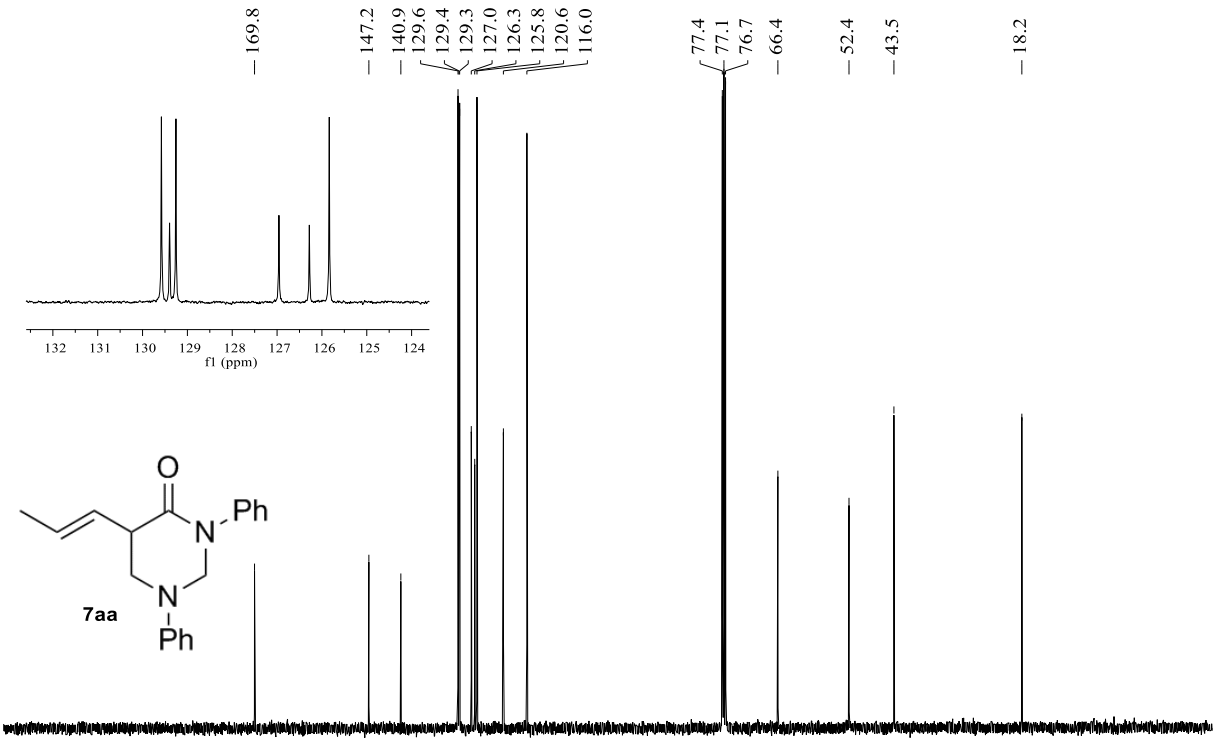
1.0
1.0
1.0
3.0
O
N
Ph
N
Ph
7aa
f1 (ppm)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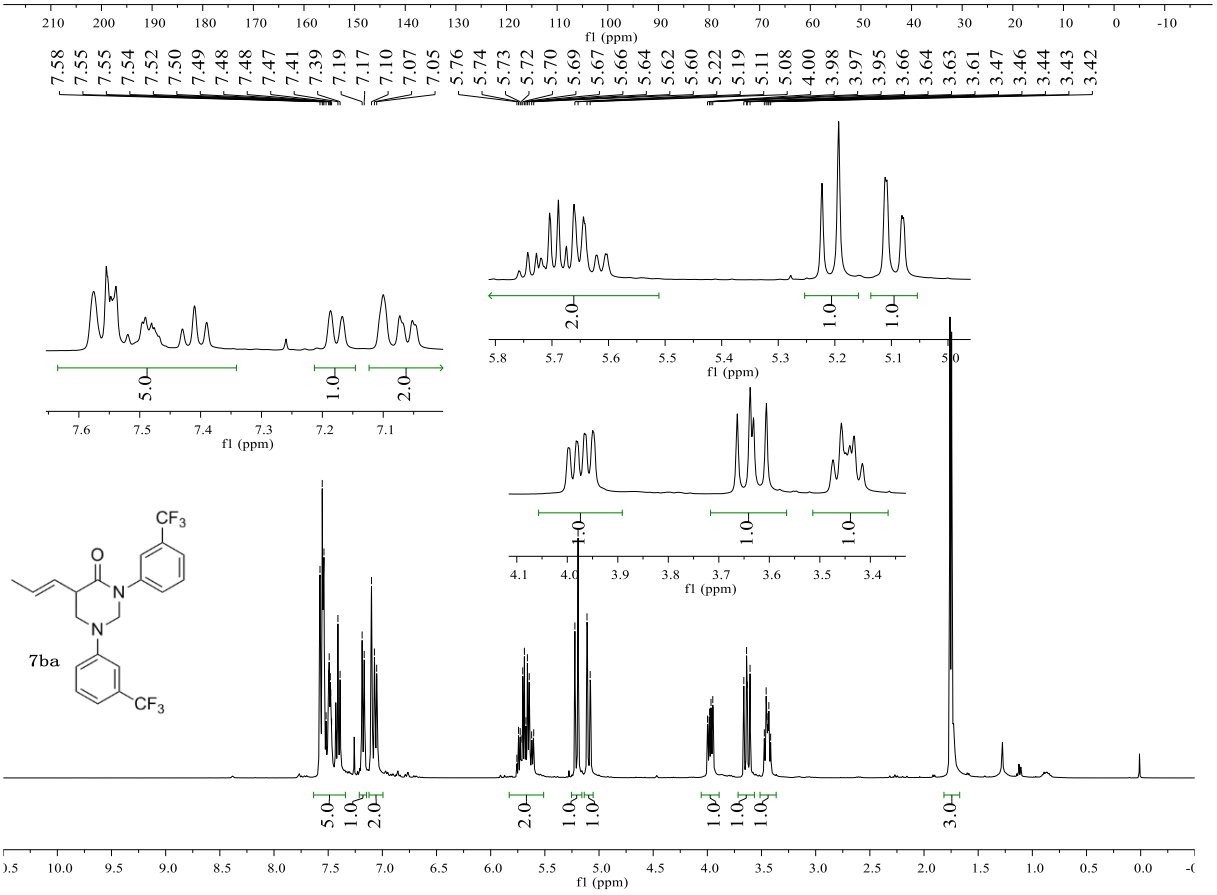
129.4
129.3
127.0
126.3
125.8
120.6
116.0
77.4
77.1
76.7
66.4
52.4
43.5
18.2
fl (ppm)
O
N
Ph
N
Ph
7aa


fl (ppm)
7.58
7.55
7.55
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7.50
7.49
7.48
7.48
7.47
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7.39
7.19
7.17
7.10
7.07
7.05
5.76
5.74
5.73
5.72
5.70
5.69
5.67
5.66
5.64
5.62
5.60
5.22
5.19
5.11
5.08
4.00
3.98
3.97
3.95
3.66
3.64
3.63
3.61
3.47
3.46
3.44
3.43
3.42
5.0
1.0
2.0
2.0
1.0
1.0
1.0
1.0
1.0
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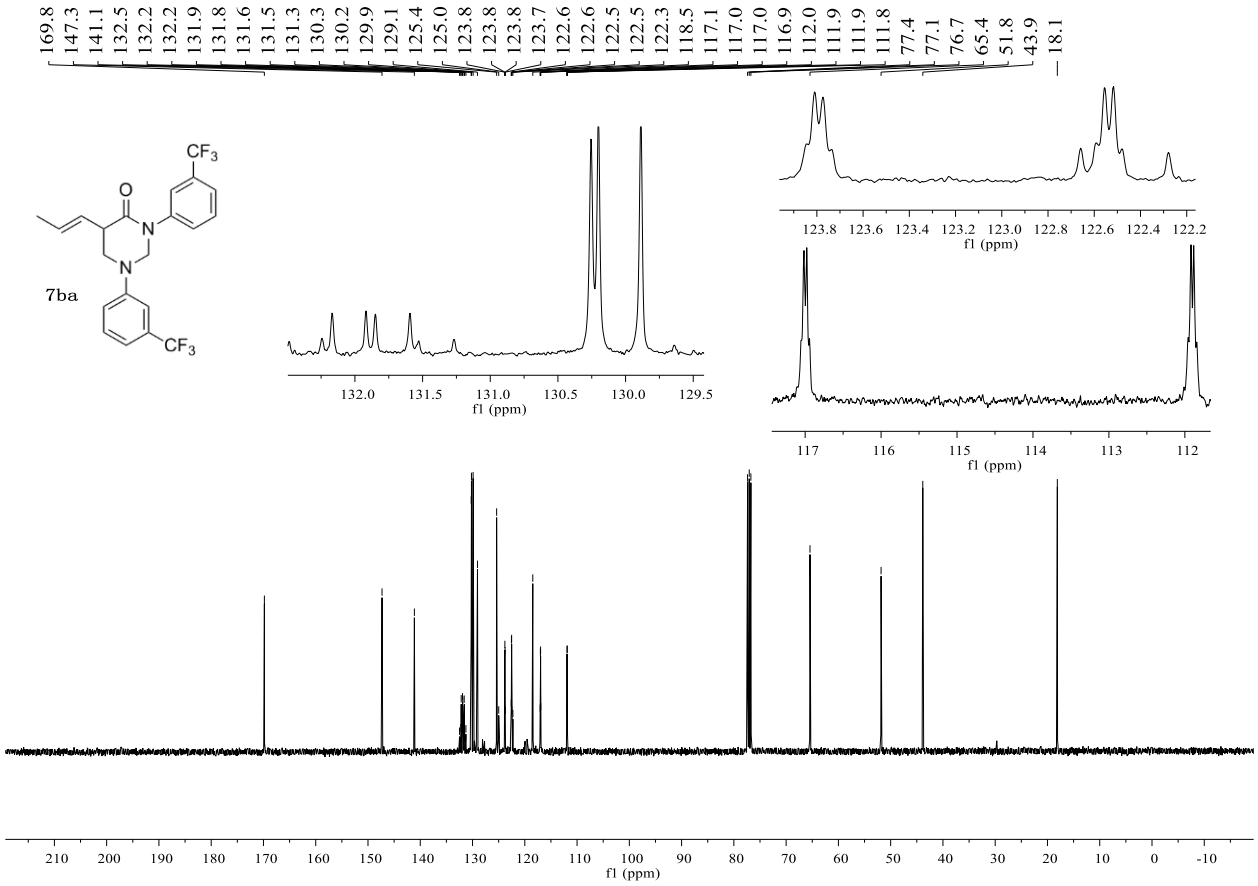
1.0
1.0
1.0
1.0
3.0
CF3
O
N
N
CF3
7ba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141.1
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132.2
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131.8
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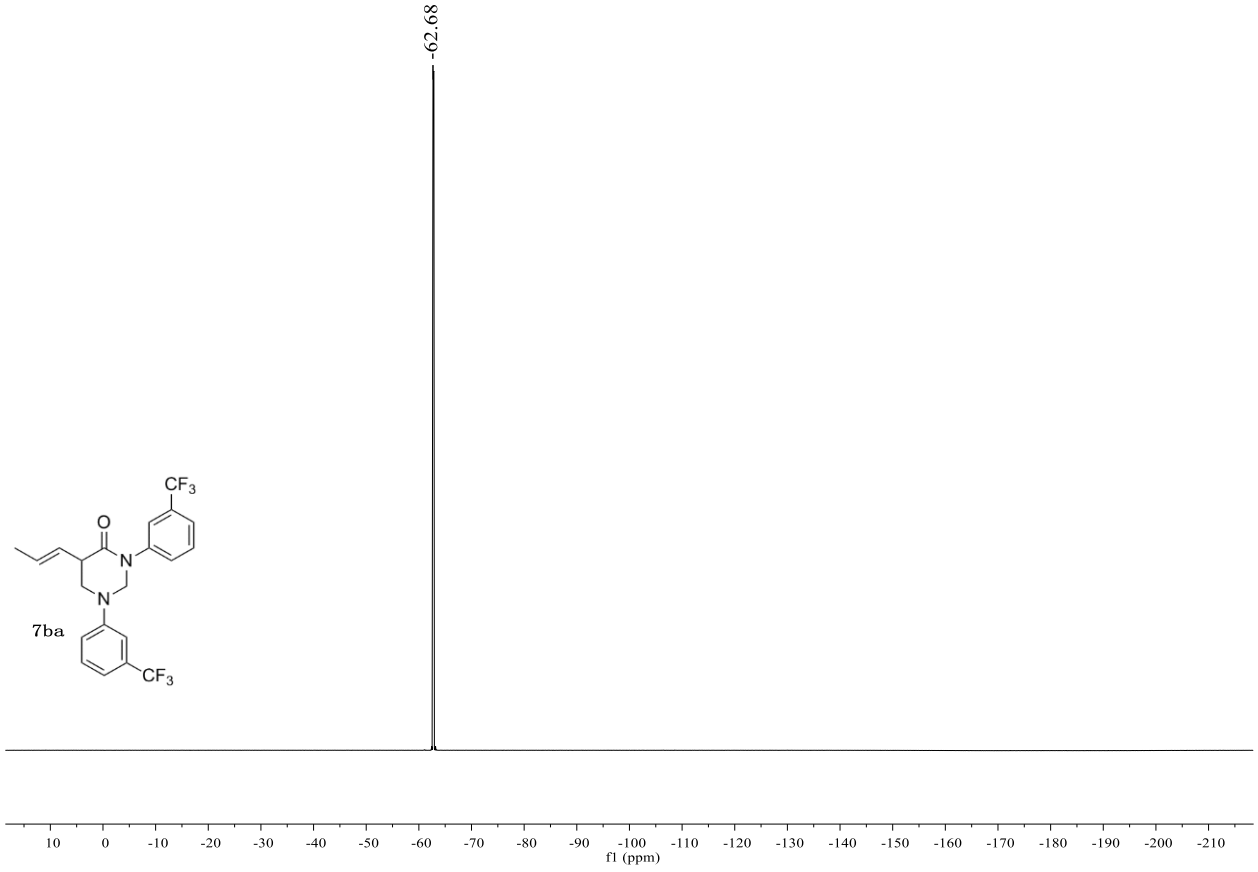
131.3
130.3
130.2
129.9
129.1
125.4
125.0
123.8
123.8
123.8
123.7
122.6
122.6
122.5
122.5
122.3
118.5
117.1
117.0
117.0
116.9
112.0
111.9
111.9
111.8
77.4
77.1
76.7
65.4
51.8
43.9
18.1
CF3
O
N
N
CF3
7ba
132.0
131.5
131.0
130.5
130.0
129.5
f1 (ppm)
123.8
123.6
123.4
123.2
123.0
122.8
122.6
122.4
122.2
f1 (ppm)
117
116
115
114
113
112
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


-62.68
CF3
O
N
N
7ba
CF3
10
0
-10
-20
-30
-40
-50
-60
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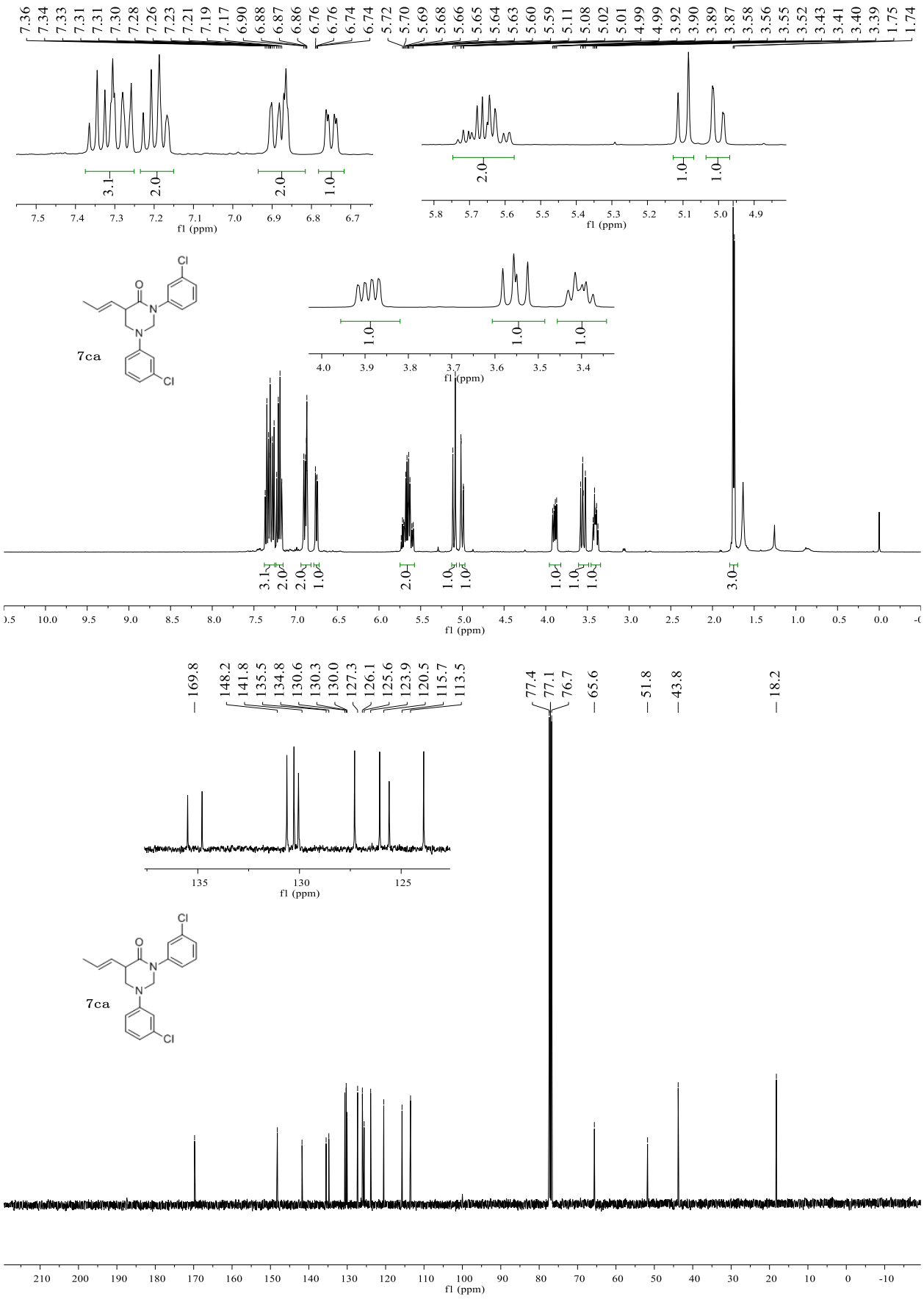
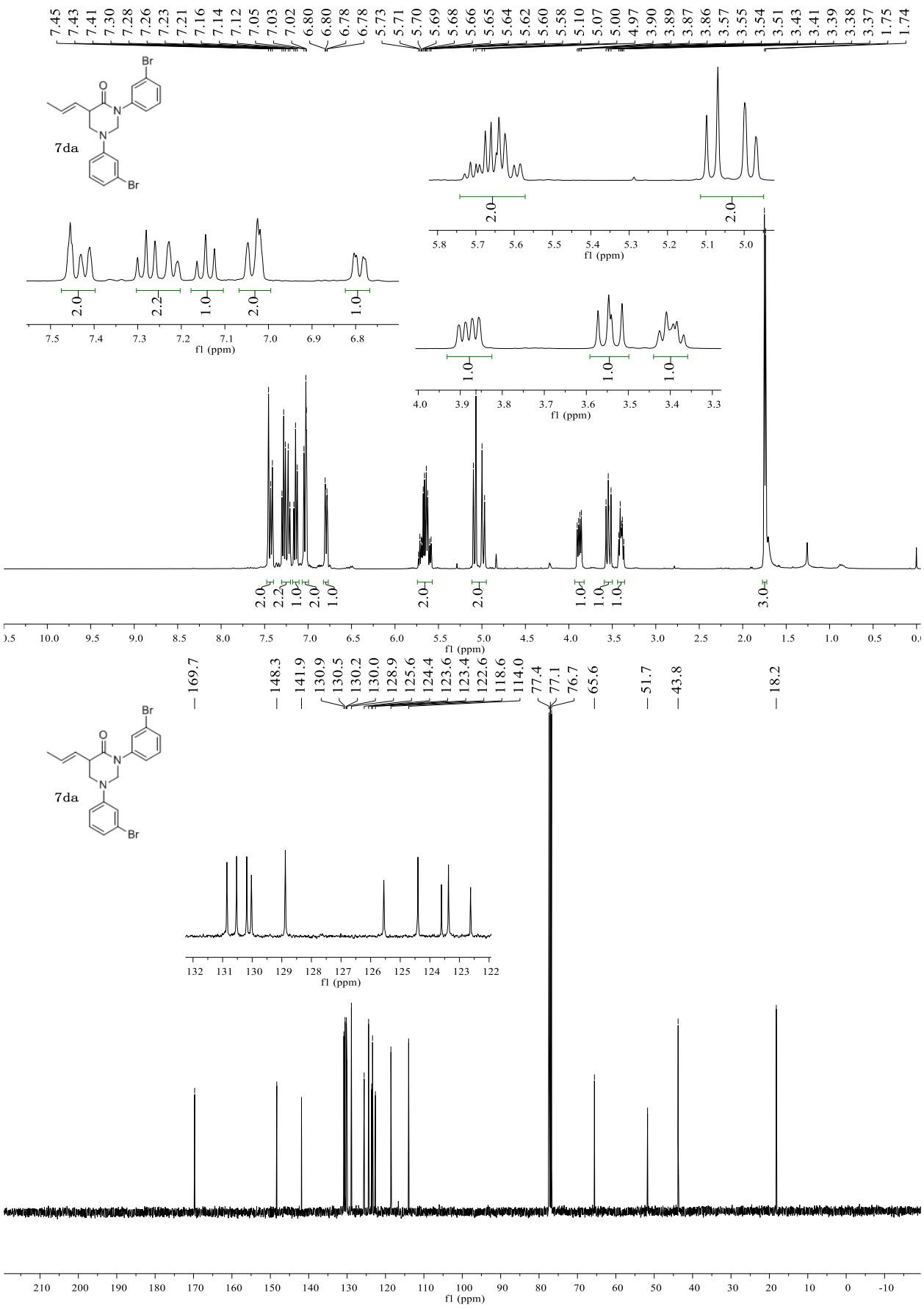
-170
-180
-190
-200
-210
f1 (ppm)


7.36 7.34 7.33 7.31 7.31 7.30 7.28 7.26 7.23 7.21 7.19 7.17 6.90 6.88 6.87 6.86 6.76 6.76 6.74 5.72 5.70 5.69 5.68 5.66 5.65 5.64 5.63 5.60 5.59 5.11 5.08 5.02 5.01 4.99 4.99 3.92 3.90 3.89 3.87 3.58 3.56 3.55 3.52 3.43 3.41 3.40 3.39 1.75 1.74
f1 (ppm)
7ca
Cl
O
N
N
Cl

169.8 148.2 141.8 135.5 134.8 130.6 130.3 130.0 127.3 126.1 125.6 123.9 120.5 115.7 113.5 77.4 77.1 76.7 65.6 51.8 43.8 18.2
f1 (ppm)
7ca
Cl
O
N
N
Cl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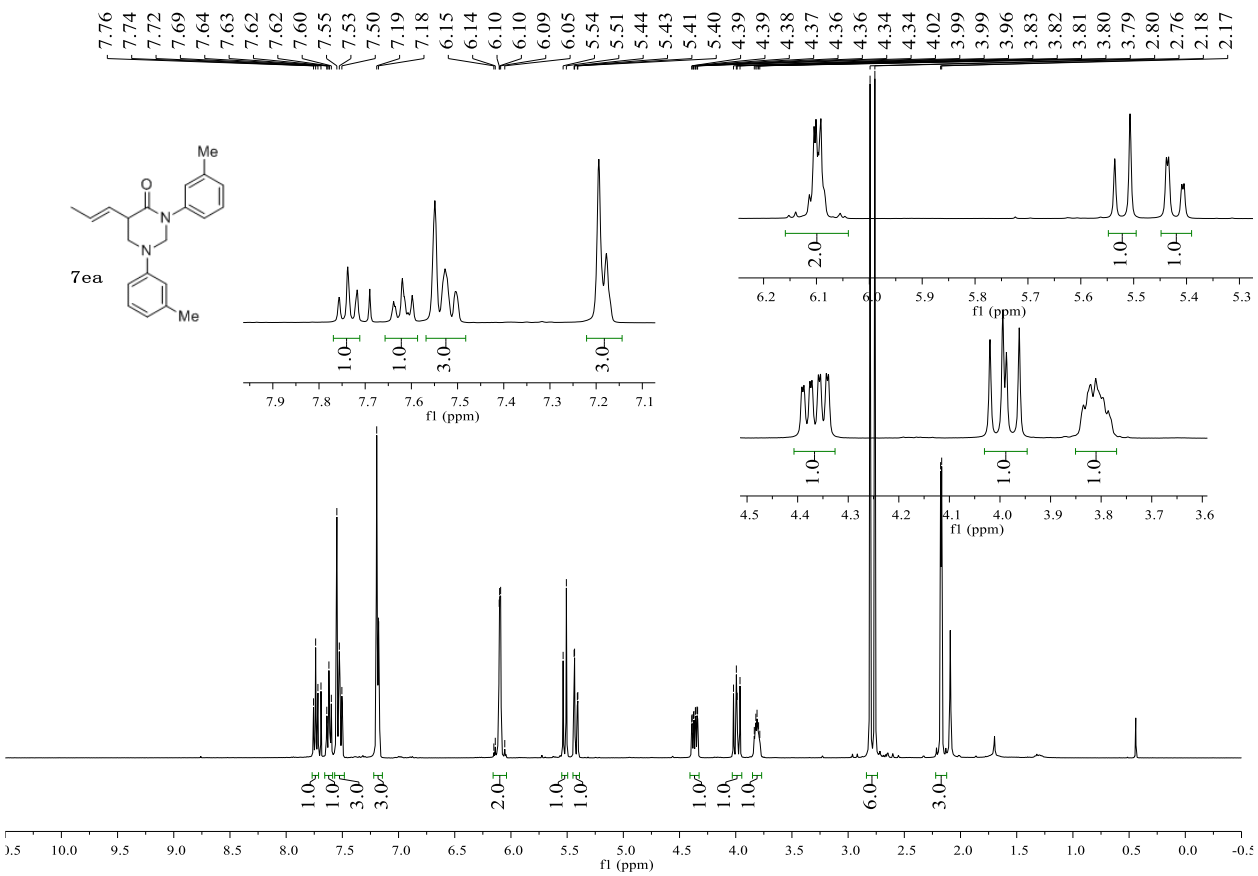
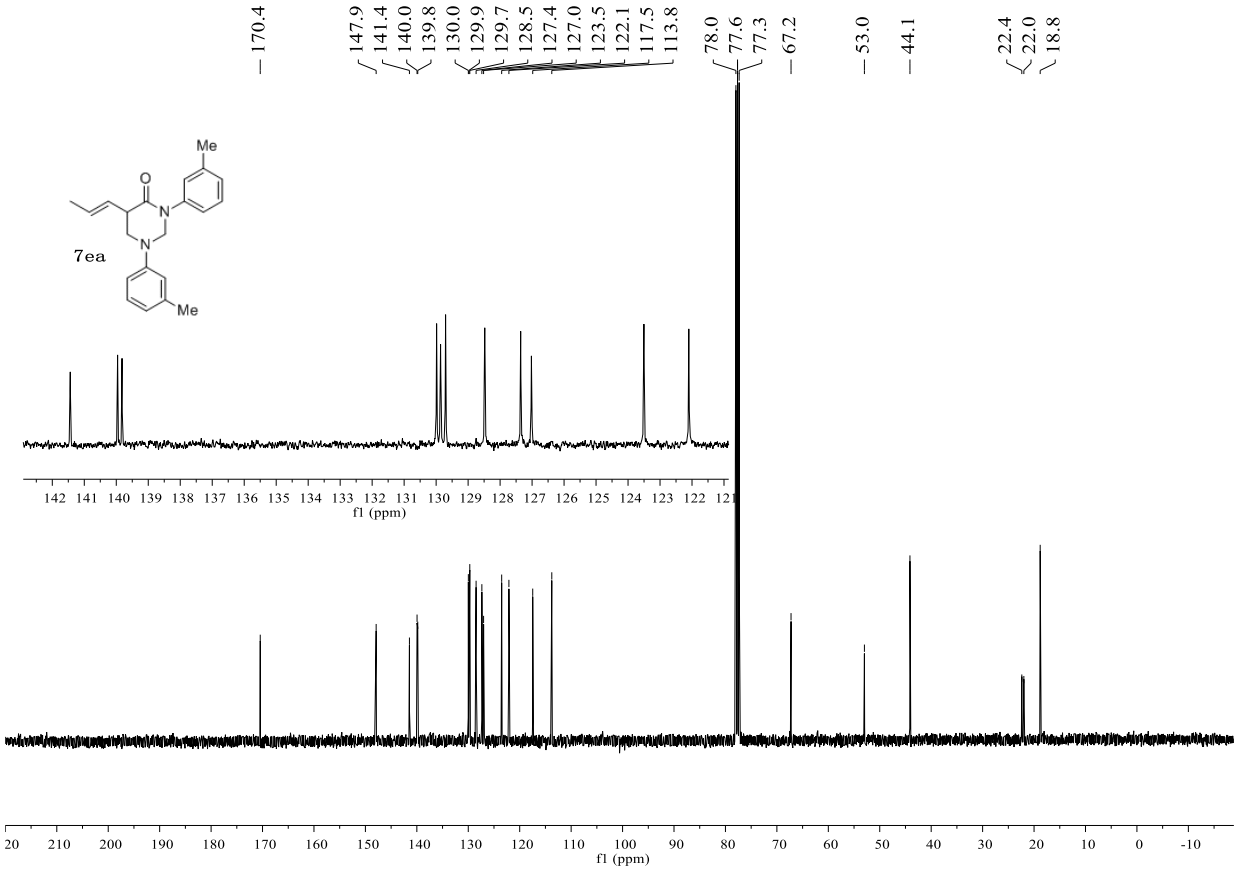
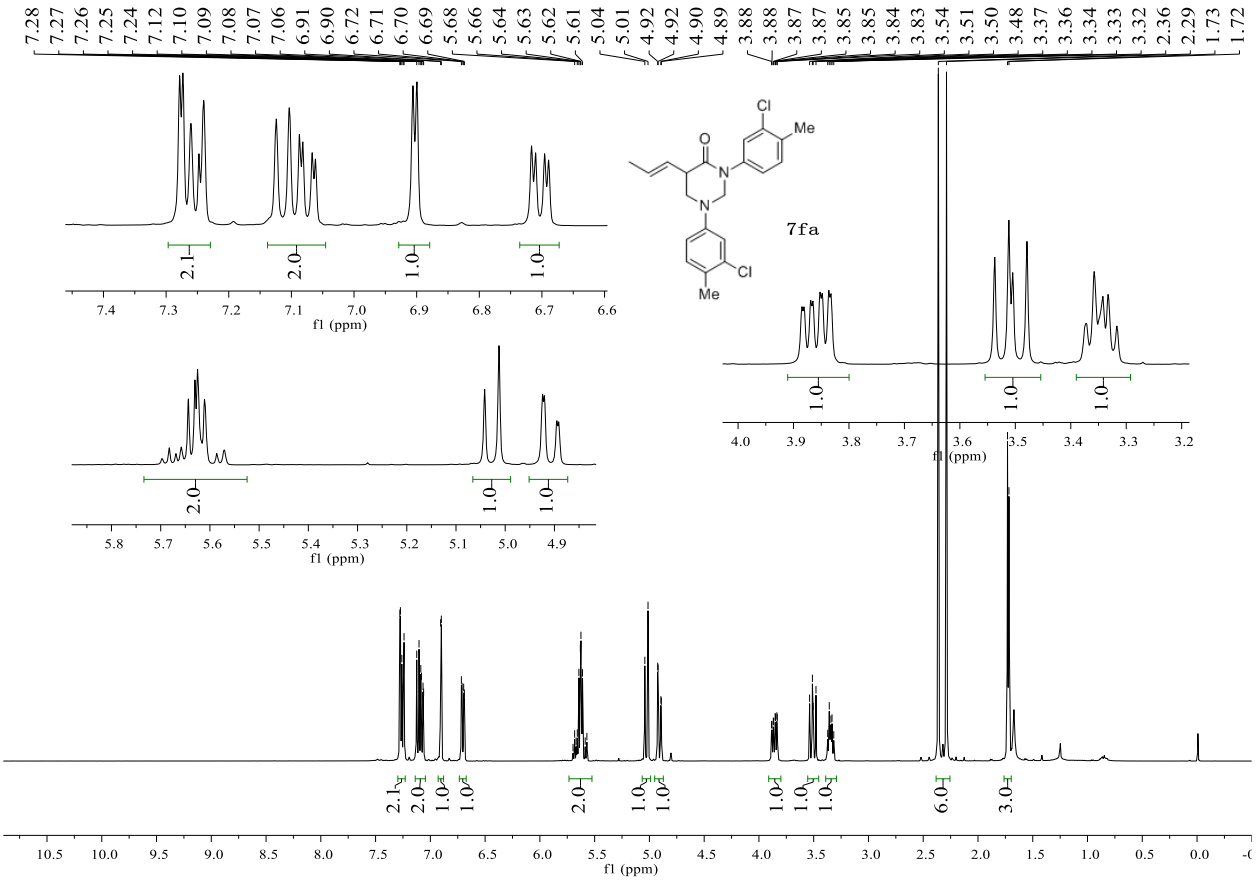
5.73 5.71 5.70 5.69 5.68 5.66 5.65 5.64 5.62 5.60 5.58 5.10 5.07 5.00 4.97 3.90 3.89 3.87 3.86 3.57 3.55 3.54 3.51 3.43 3.41 3.39 3.38 3.37 1.75 1.74
7da
Br
Br
f1 (ppm)
169.7 148.3 141.9 130.9 130.5 130.2 130.0 128.9 125.6 124.4 123.6 123.4 122.6 118.6 114.0 77.4 77.1 76.7 65.6 51.7 43.8 18.2
7da
Br
Br
f1 (ppm)


7.76 7.74 7.72 7.69 7.64 7.63 7.62 7.62 7.60 7.55 7.53 7.50 7.19 7.18 6.15 6.14 6.10 6.10 6.09 6.05 5.54 5.51 5.44 5.43 5.41 5.40 4.39 4.39 4.38 4.37 4.36 4.36 4.34 4.34 4.02 3.99 3.99 3.96 3.83 3.82 3.81 3.80 3.79 2.80 2.76 2.18 2.17
Me
O
N
N
7ea
Me
1.0 1.0 3.0 3.0
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1
f1 (ppm)
2.0 1.0 1.0
6.2 6.1 6.0 5.9 5.8 5.7 5.6 5.5 5.4 5.3
f1 (ppm)
1.0 1.0 1.0
4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6
f1 (ppm)
1.0 1.0 1.0 3.0 2.0 1.0 1.0 1.0 1.0 1.0 6.0 3.0
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


170.4
147.9 141.4 140.0 139.8 130.0 129.9 129.7 128.5 127.4 127.0 123.5 122.1 117.5 113.8
78.0 77.6 77.3
67.2
53.0
44.1
22.4 22.0 18.8
Me
O
N
N
7ea
Me
142 141 140 139 138 137 136 135 134 133 132 131 130 129 128 127 126 125 124 123 122
f1 (ppm)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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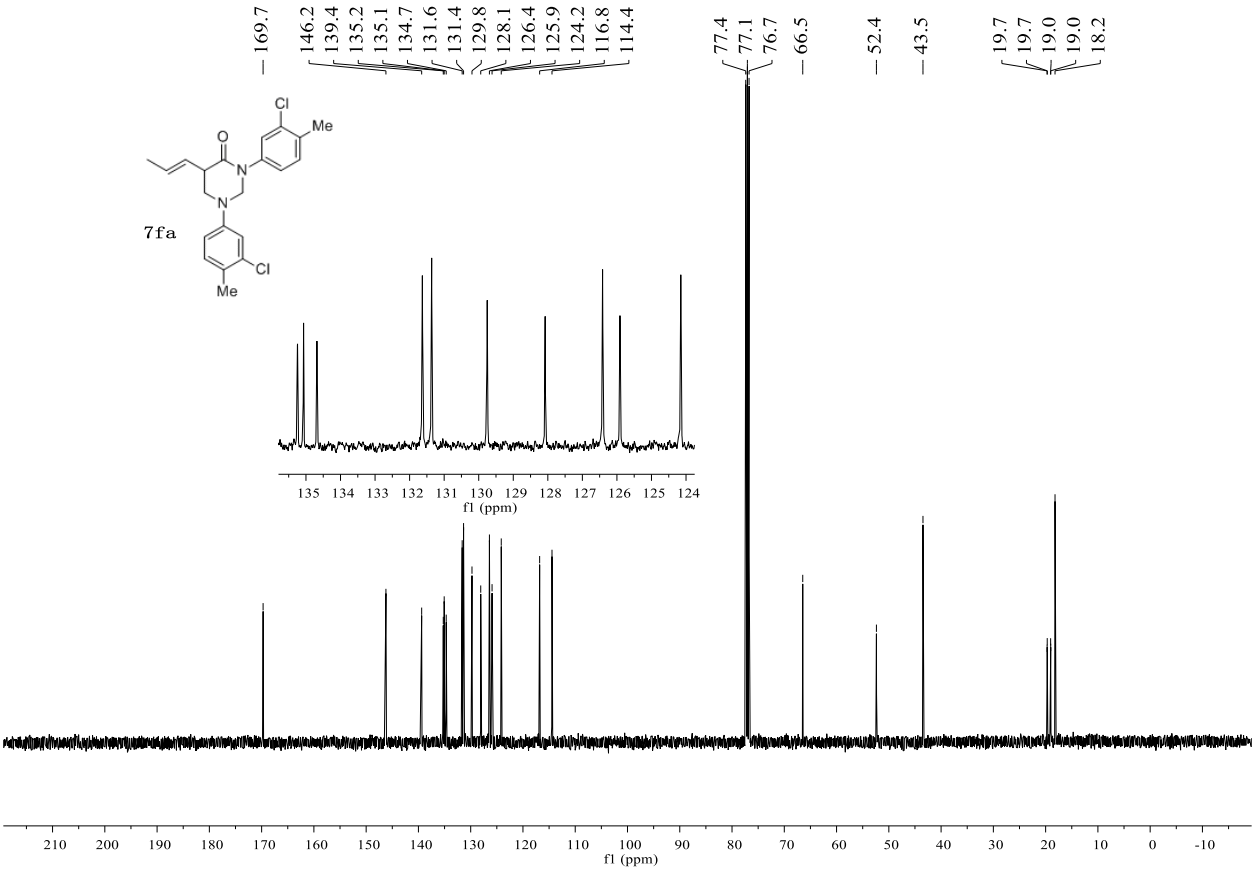
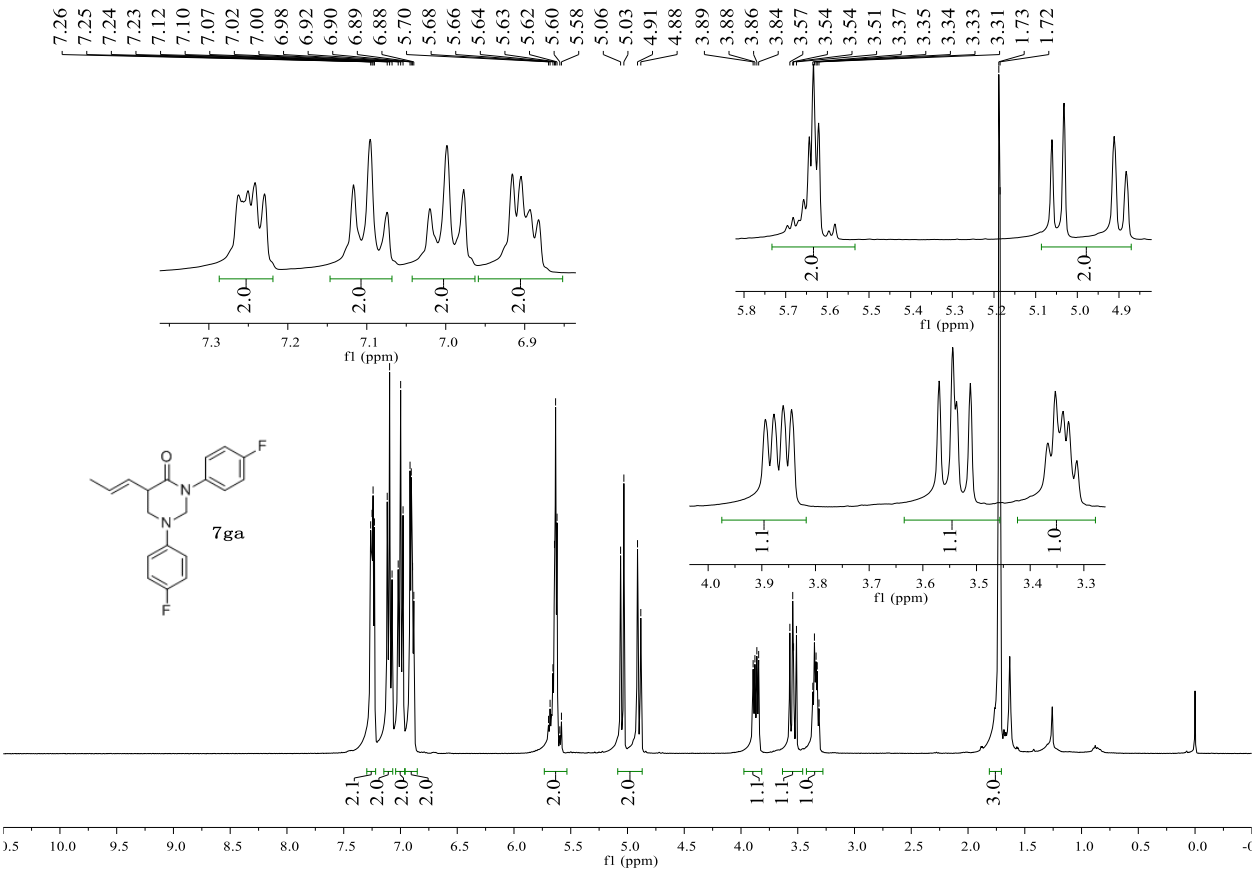
Cl
Me
O
N
N
Cl
Me
7fa
f1 (ppm)


169.7 146.2 139.4 135.2 135.1 134.7 131.6 131.4 129.8 128.1 126.4 125.9 124.2 116.8 114.4 77.4 77.1 76.7 66.5 52.4 43.5 19.7 19.7 19.0 19.0 18.2
Cl
Me
O
N
N
7fa
Cl
Me
f1 (ppm)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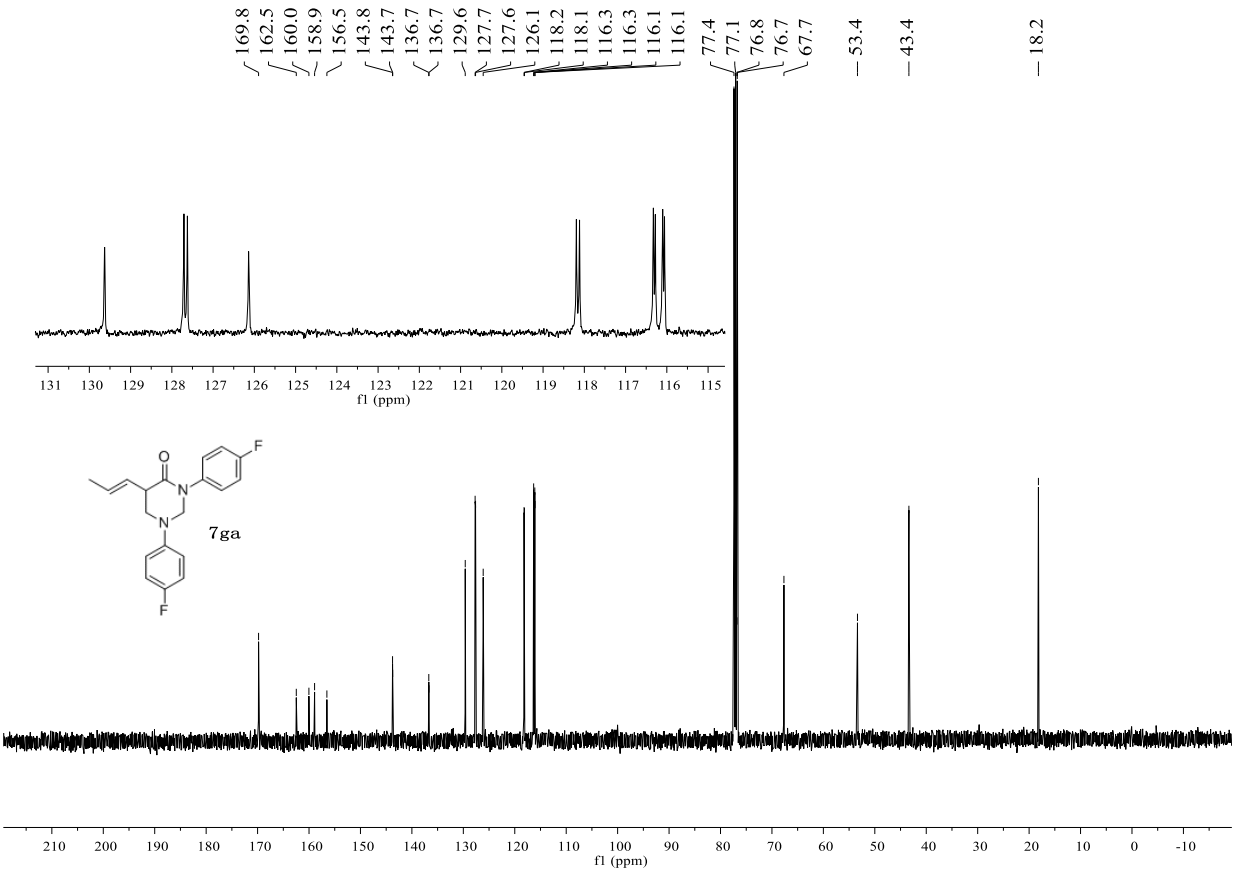
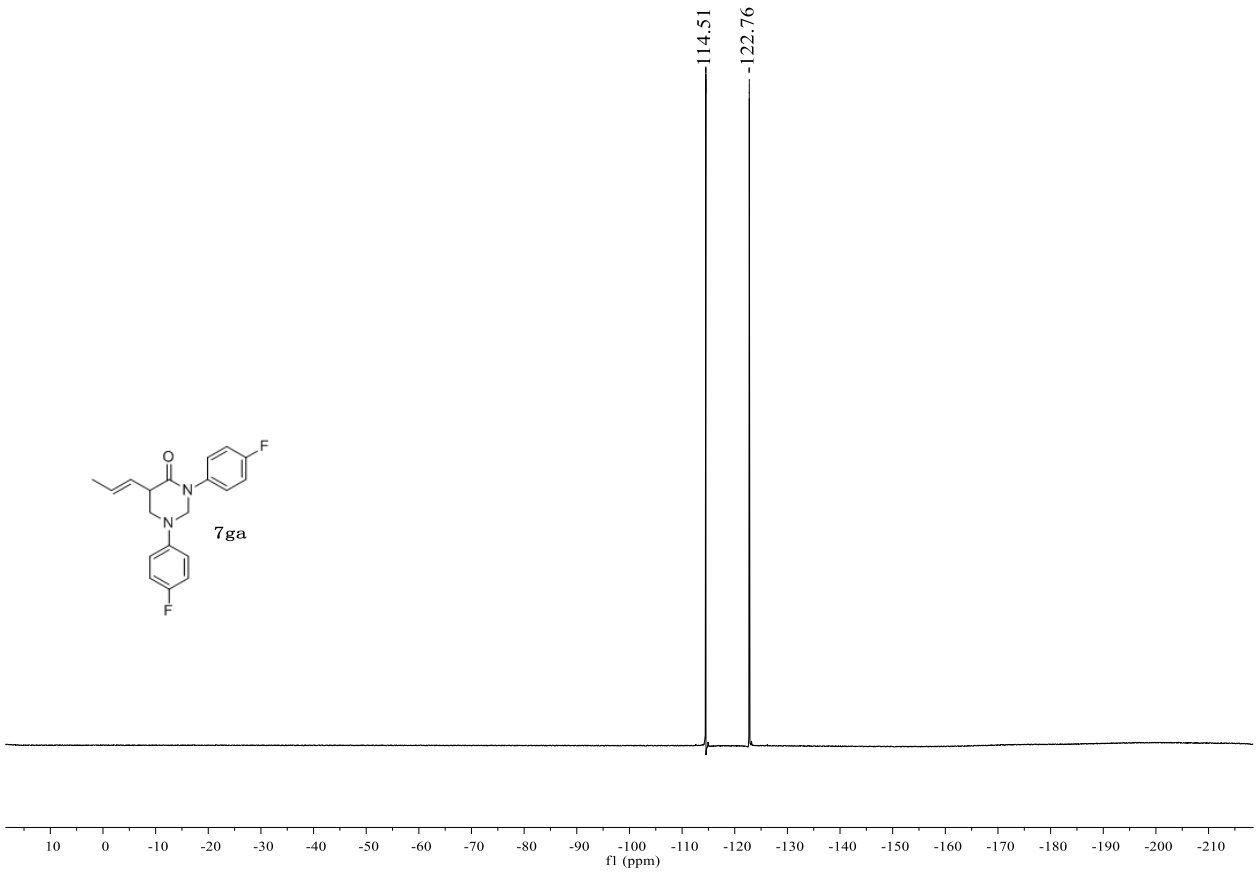
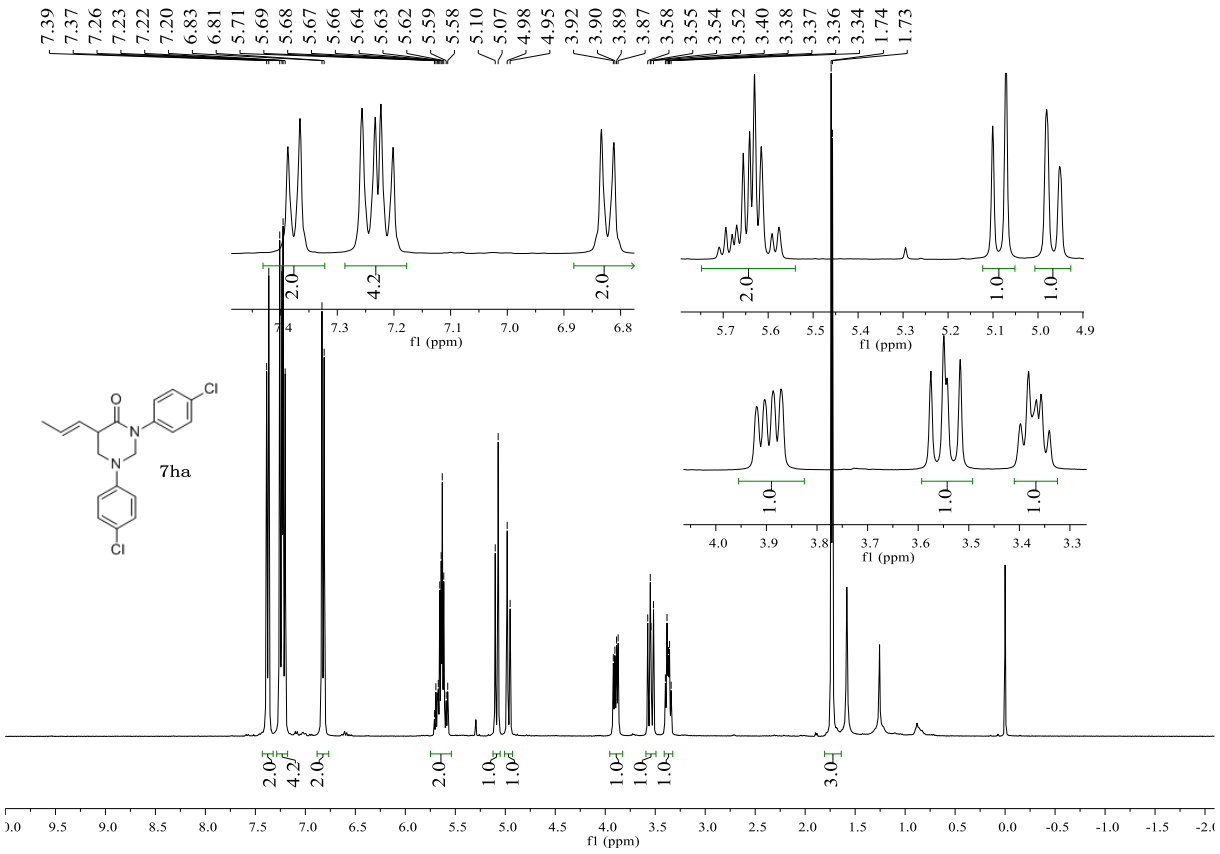
2.0 2.0 2.0 2.0
f1 (ppm)
2.0 2.0
f1 (ppm)
1.1 1.1 1.0
f1 (ppm)
7ga
2.1 2.0 2.0 2.0 2.0 2.0 1.1 1.1 1.0 3.0
f1 (ppm)


169.8 162.5 160.0 158.9 156.5 143.8 143.7 136.7 136.7 129.6 127.7 127.6 126.1 118.2 118.1 116.3 116.3 116.1 116.1 77.4 77.1 76.8 76.7 67.7 53.4 43.4 18.2
f1 (ppm)
7ga
f1 (ppm)


114.51
-122.76
7ga
f1 (ppm)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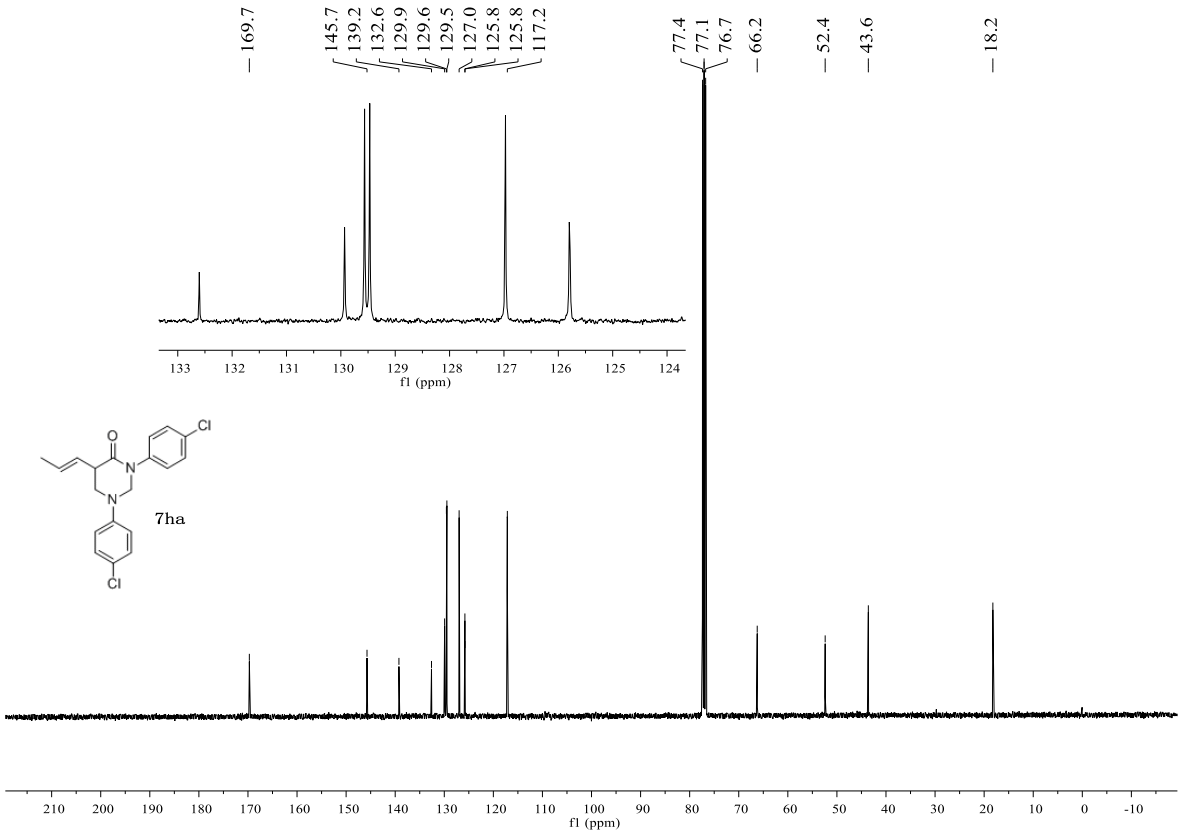
5.66
5.64
5.63
5.62
5.59
5.58
5.10
5.07
4.98
4.95
3.92
3.90
3.89
3.87
3.58
3.55
3.54
3.52
3.40
3.38
3.37
3.36
3.34
1.74
1.73
7ha
f1 (ppm)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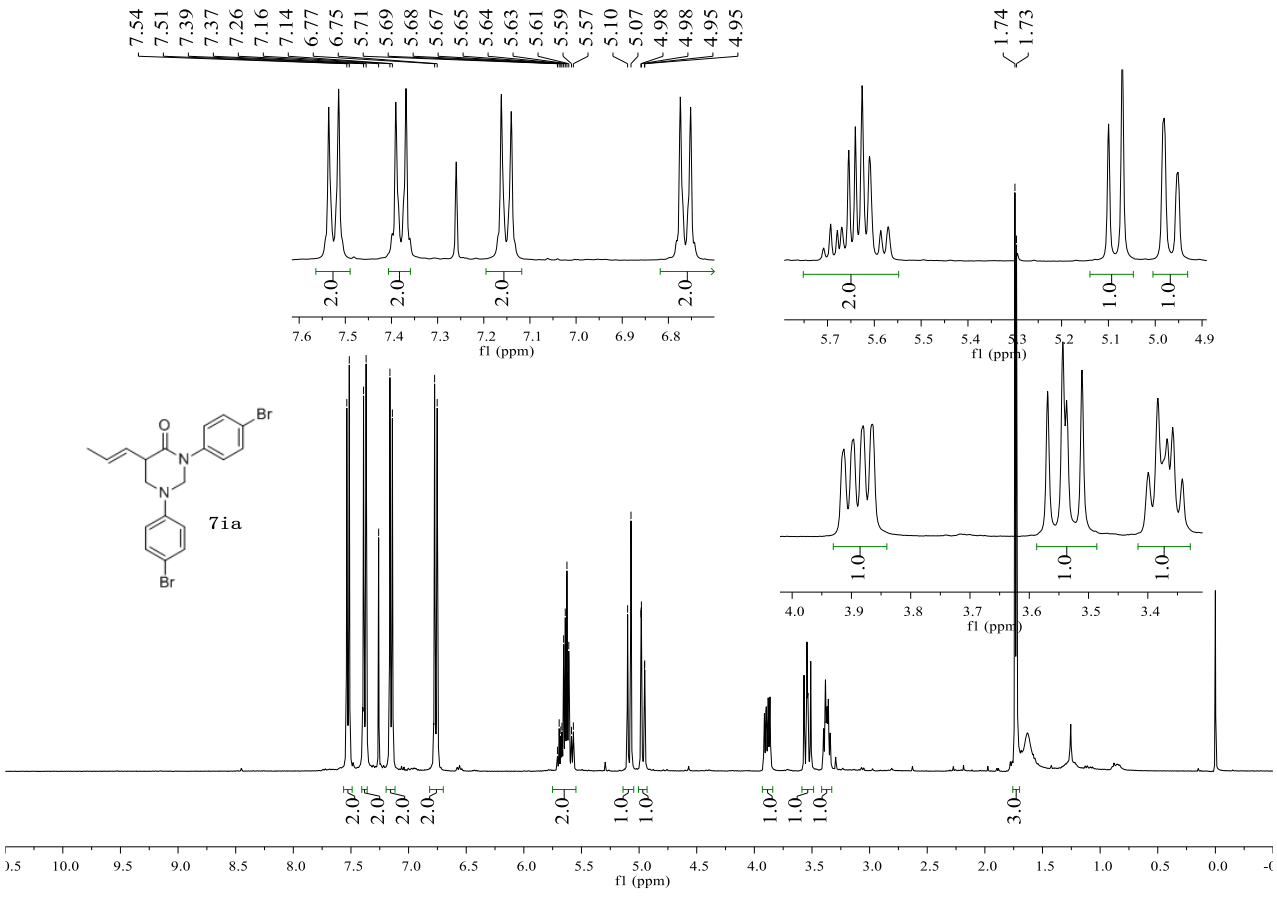
132.6
129.9
129.6
129.5
127.0
125.8
125.8
117.2
77.4
77.1
76.7
66.2
52.4
43.6
18.2
7ha
f1 (ppm)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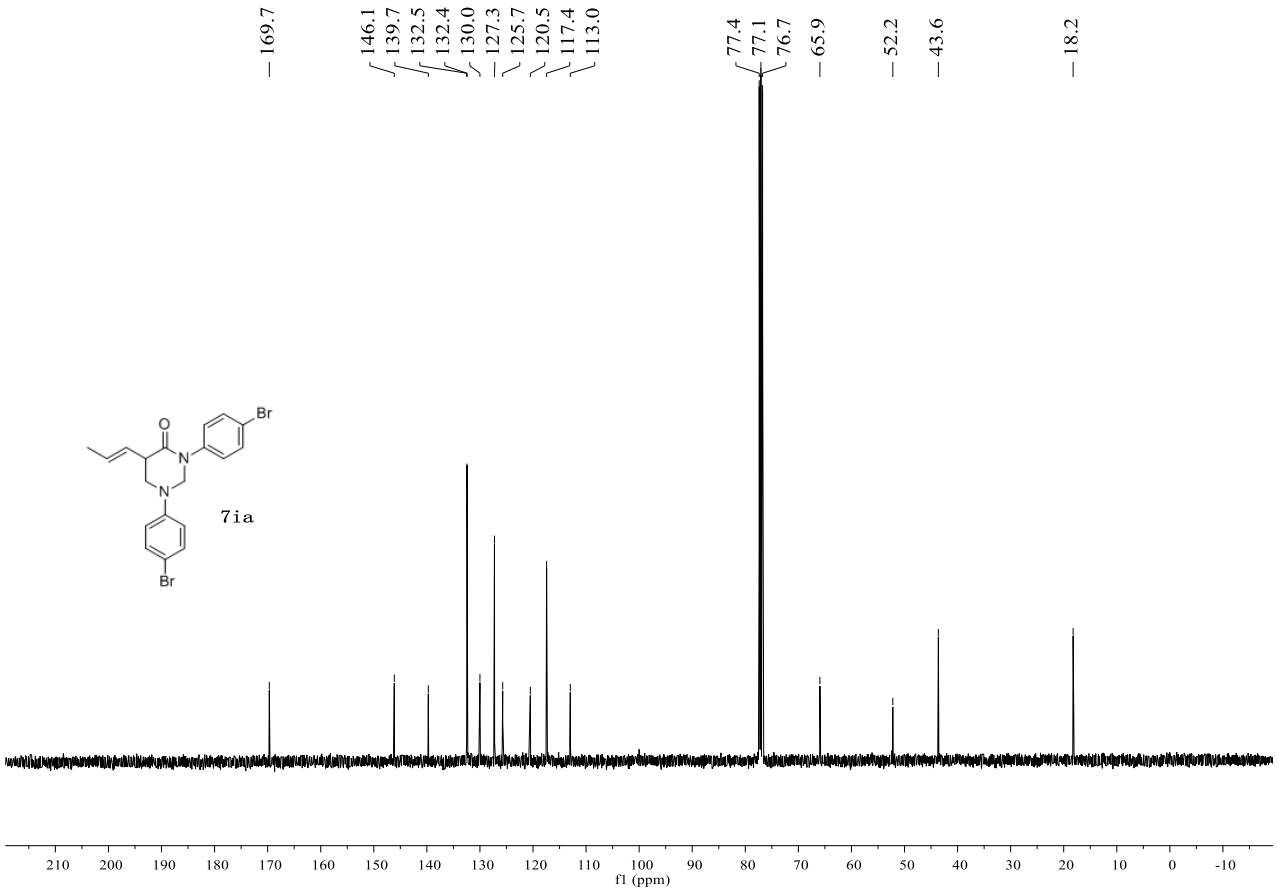
5.65
5.64
5.63
5.61
5.59
5.57
5.10
5.07
4.98
4.98
4.95
4.95
1.74
1.73
7ia
f1 (ppm)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139.7
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132.4
130.0
127.3
125.7
120.5
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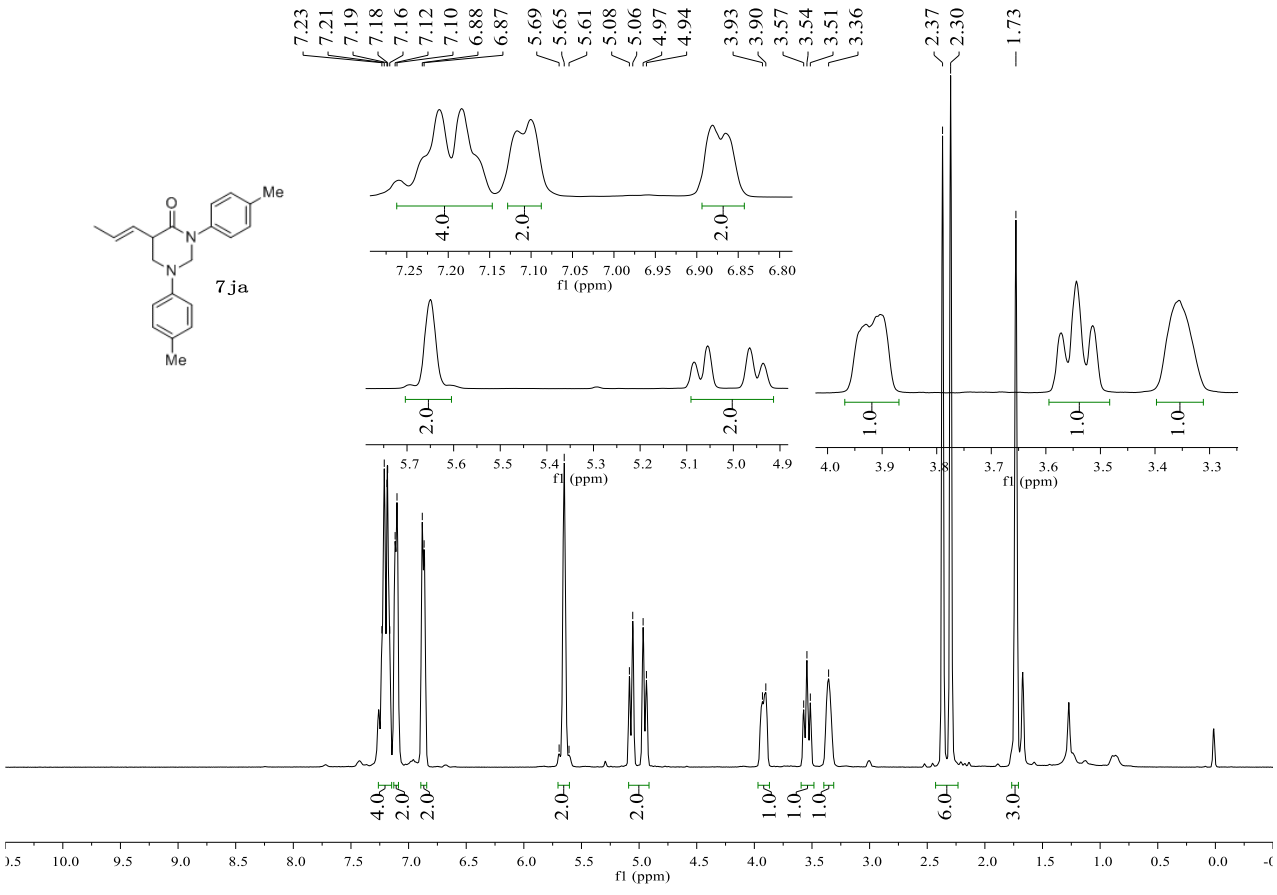
113.0
77.4
77.1
76.7
65.9
52.2
43.6
18.2
Br
Br
7ia
f1 (ppm)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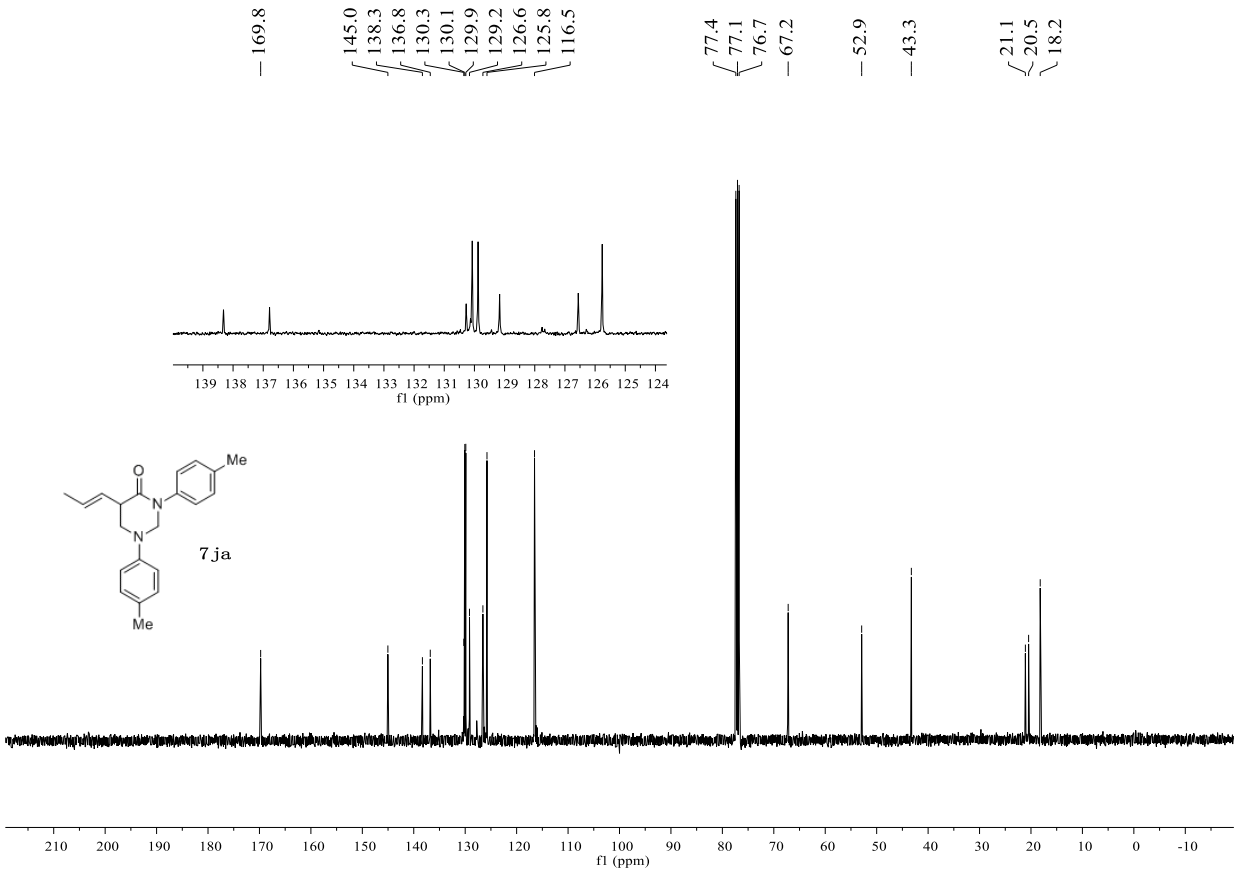
5.65
5.61
5.08
5.06
4.97
4.94
3.93
3.90
3.57
3.54
3.51
3.36
2.37
2.30
1.73
Me
Me
7ja
4.0
2.0
2.0
2.0
2.0
2.0
2.0
1.0
1.0
1.0
6.0
3.0
f1 (ppm)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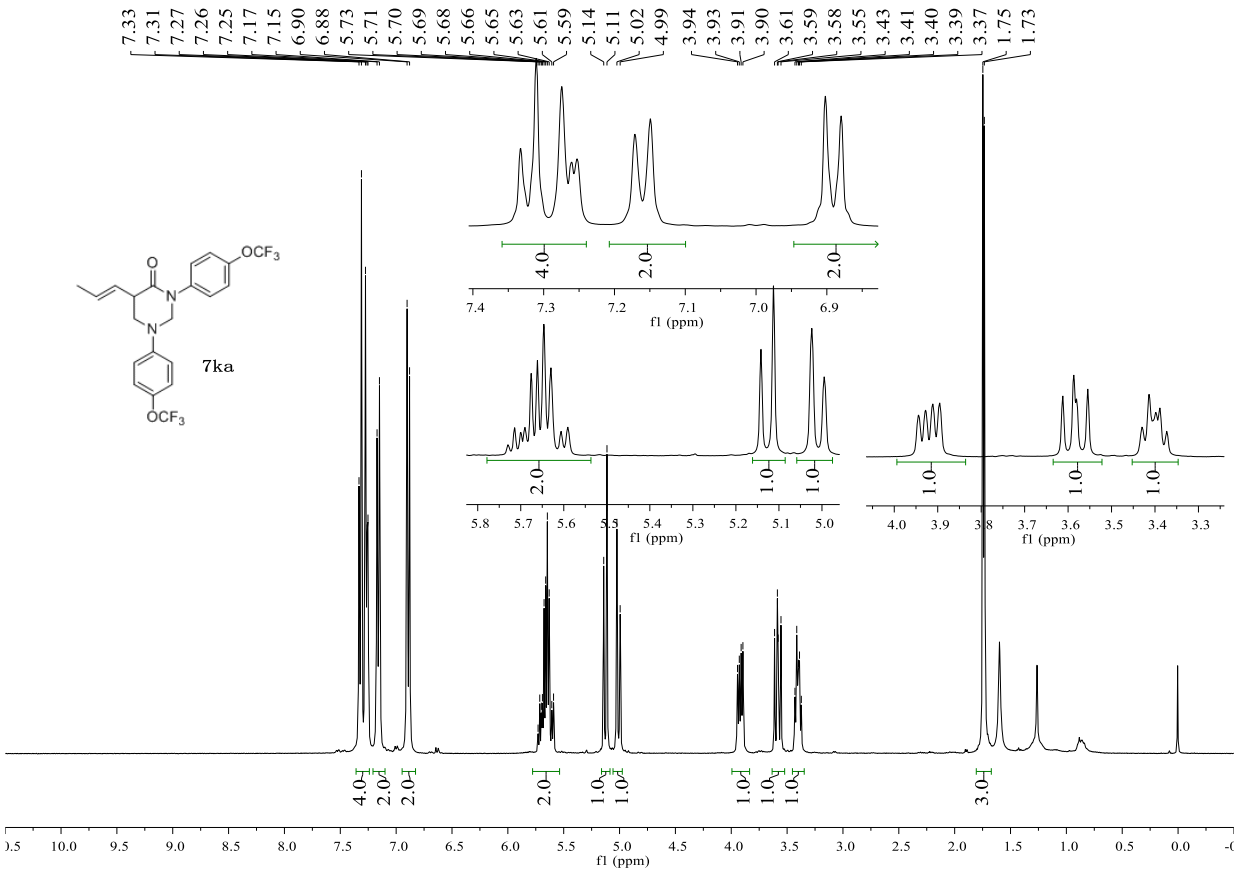
145.0
138.3
136.8
130.3
130.1
129.9
129.2
126.6
125.8
116.5
77.4
77.1
76.7
67.2
52.9
43.3
21.1
20.5
18.2
Me
Me
7ja
f1 (ppm)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7.17
7.15
6.90
6.88
5.73
5.71
5.70
5.69
5.68
5.66
5.65
5.63
5.61
5.59
5.14
5.11
5.02
4.99
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3.93
3.91
3.90
3.61
3.59
3.58
3.55
3.43
3.41
3.40
3.39
3.37
1.75
1.73
OCF3
OCF3
7ka
4.0
2.0
2.0
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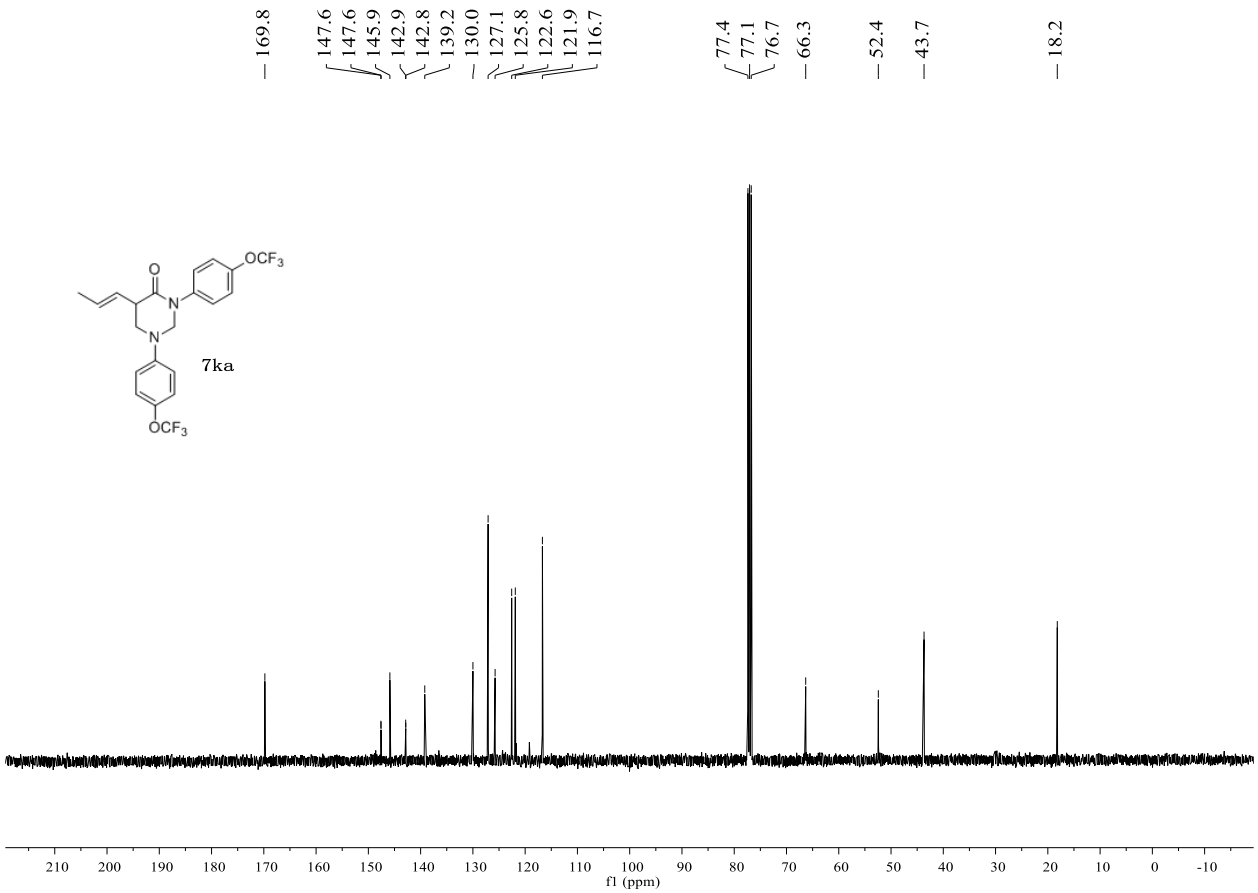
1.0
1.0
1.0
1.0
1.0
1.0
1.0
3.0
f1 (ppm)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147.6
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142.9
142.8
139.2
130.0
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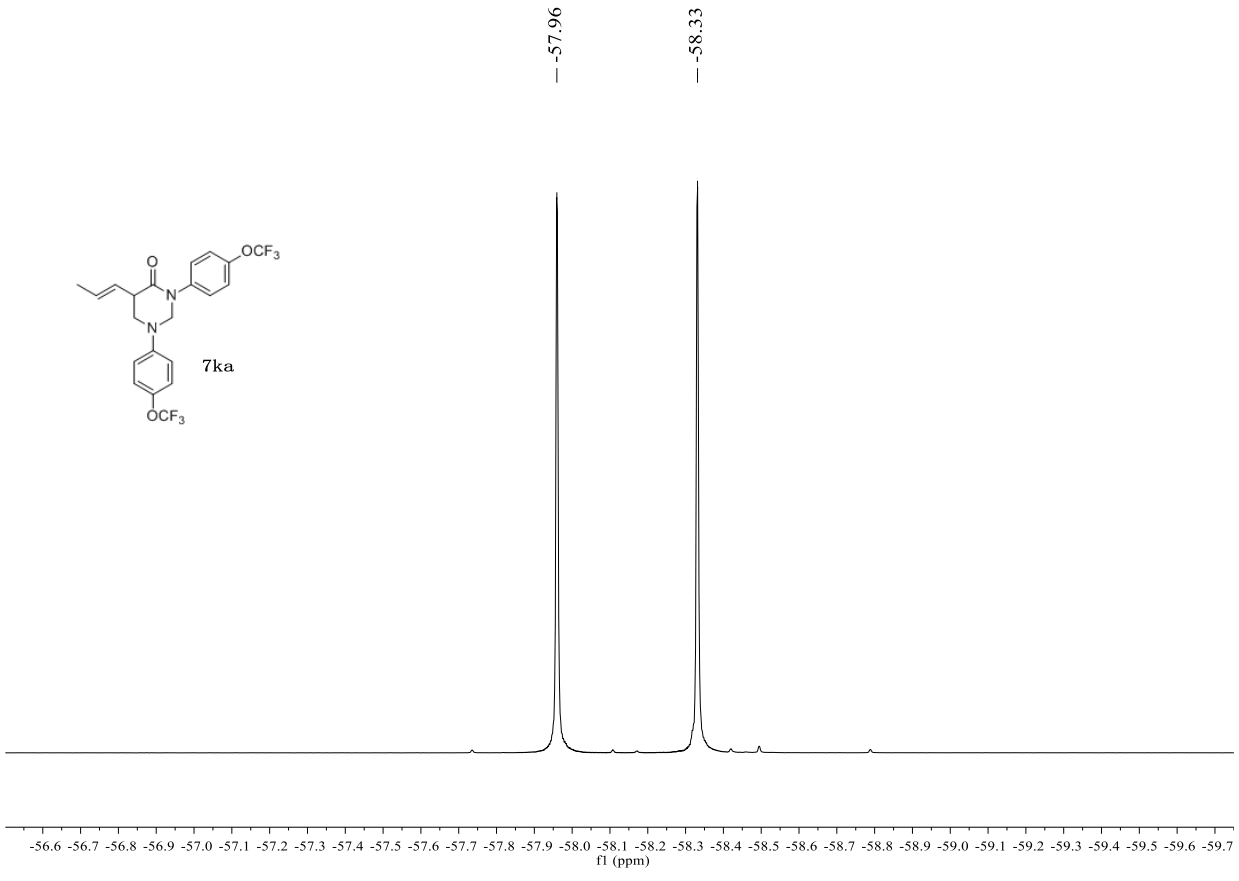
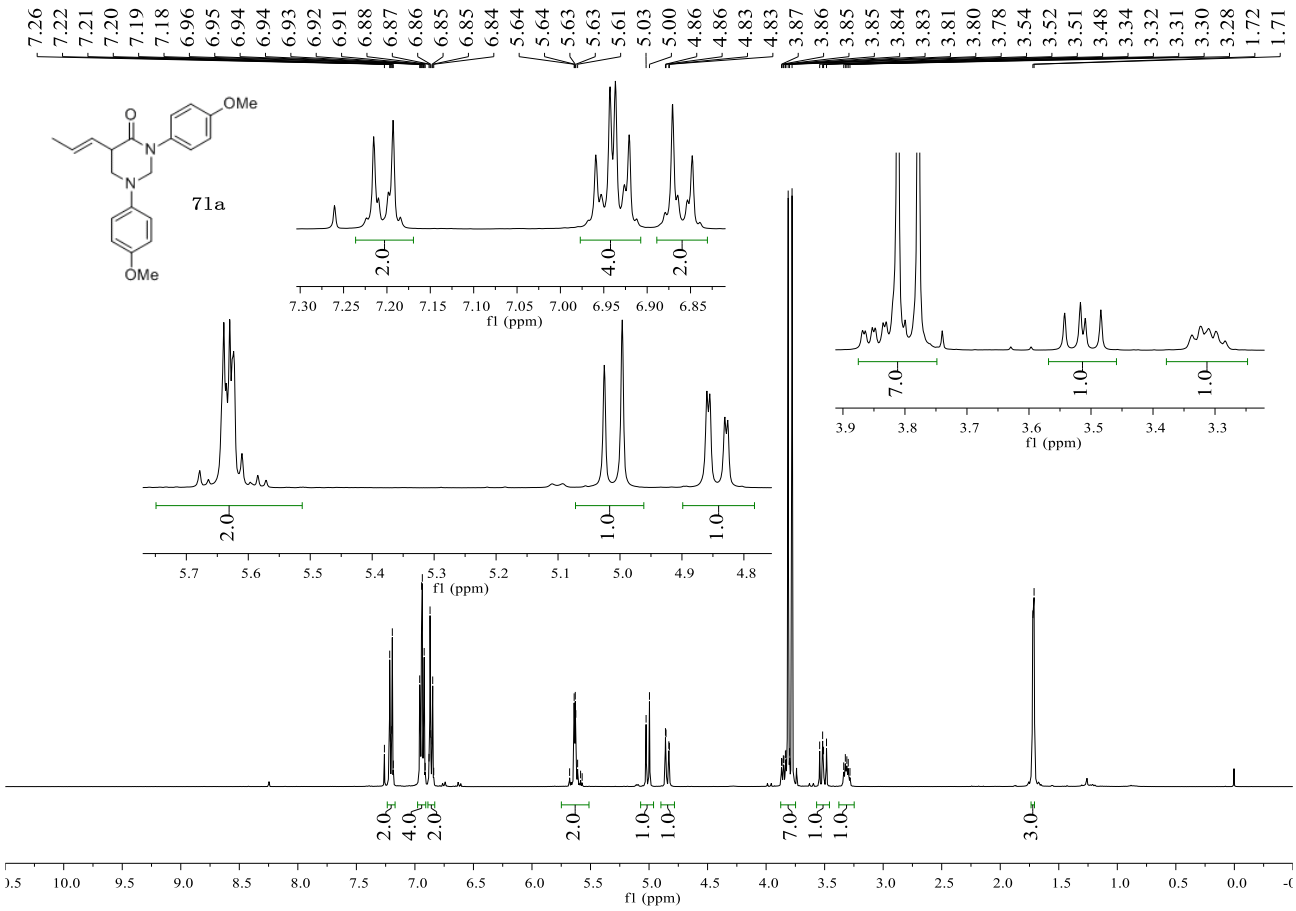
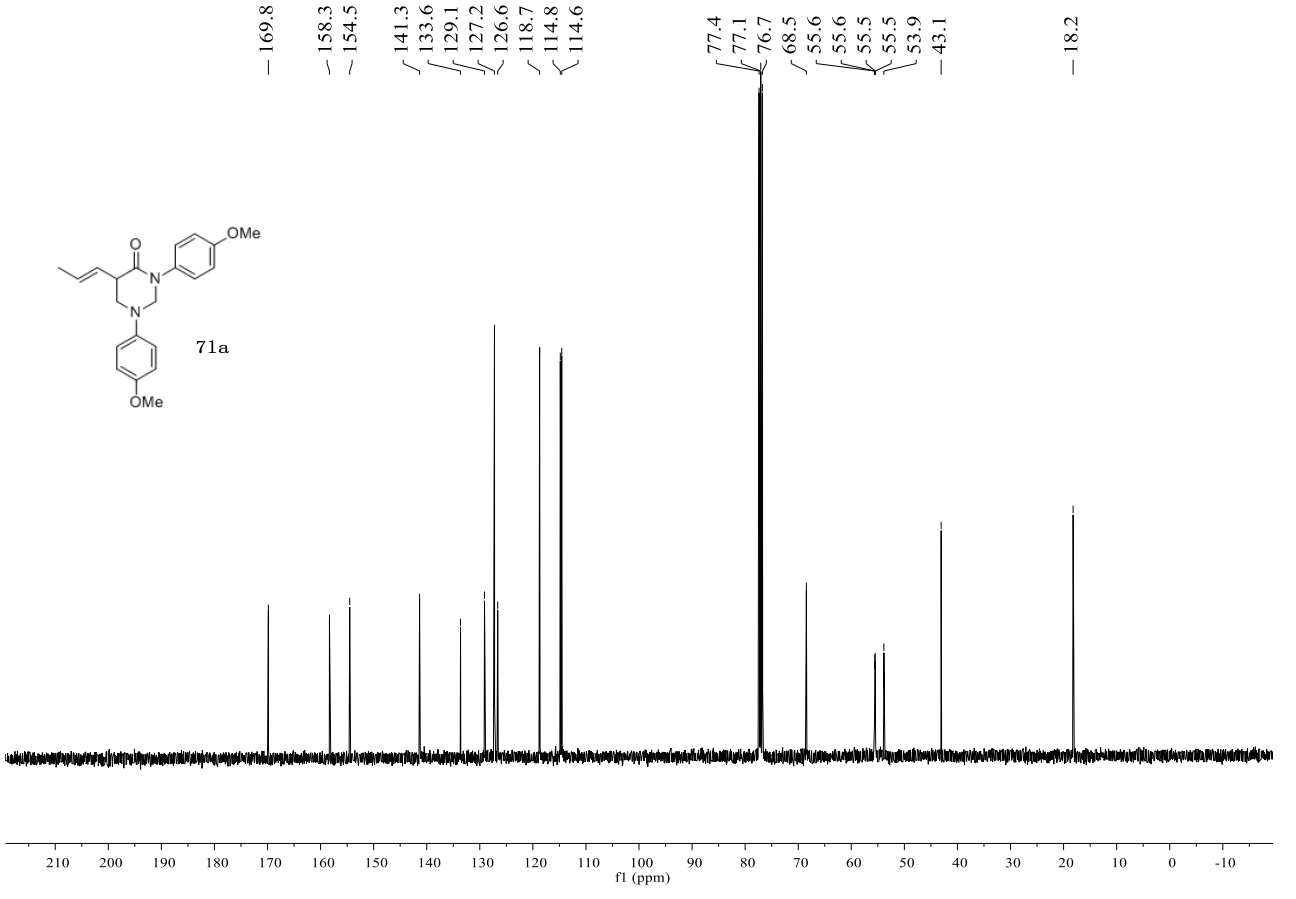
125.8
122.6
121.9
116.7
77.4
77.1
76.7
66.3
52.4
43.7
18.2
OCF3
7ka
OCF3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-57.96
-58.33
OCF3
7ka
OCF3
-56.6 -56.7 -56.8 -56.9 -57.0 -57.1 -57.2 -57.3 -57.4 -57.5 -57.6 -57.7 -57.8 -57.9 -58.0 -58.1 -58.2 -58.3 -58.4 -58.5 -58.6 -58.7 -58.8 -58.9 -59.0 -59.1 -59.2 -59.3 -59.4 -59.5 -59.6 -59.7
f1 (ppm)


7.26 7.22 7.21 7.20 7.19 7.18 6.96 6.95 6.94 6.94 6.93 6.92 6.91 6.88 6.87 6.86 6.85 6.85 6.84
5.64 5.64 5.63 5.63 5.61 5.03 5.00 4.86 4.86 4.83 4.83
3.87 3.86 3.85 3.85 3.84 3.83 3.81 3.80 3.78 3.54 3.52 3.51 3.48 3.34 3.32 3.31 3.30 3.28 1.72 1.71
OMe
O
N
N
OMe
71a
2.0
4.0
2.0
7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85
f1 (ppm)
7.0
1.0
1.0
3.9 3.8 3.7 3.6 3.5 3.4 3.3
f1 (ppm)
2.0
1.0
1.0
5.7 5.6 5.5 5.4 5.3 5.2 5.1 5.0 4.9 4.8
f1 (ppm)
2.0 4.0 2.0 2.0 1.0 1.0 7.0 1.0 1.0 3.0
f1 (ppm)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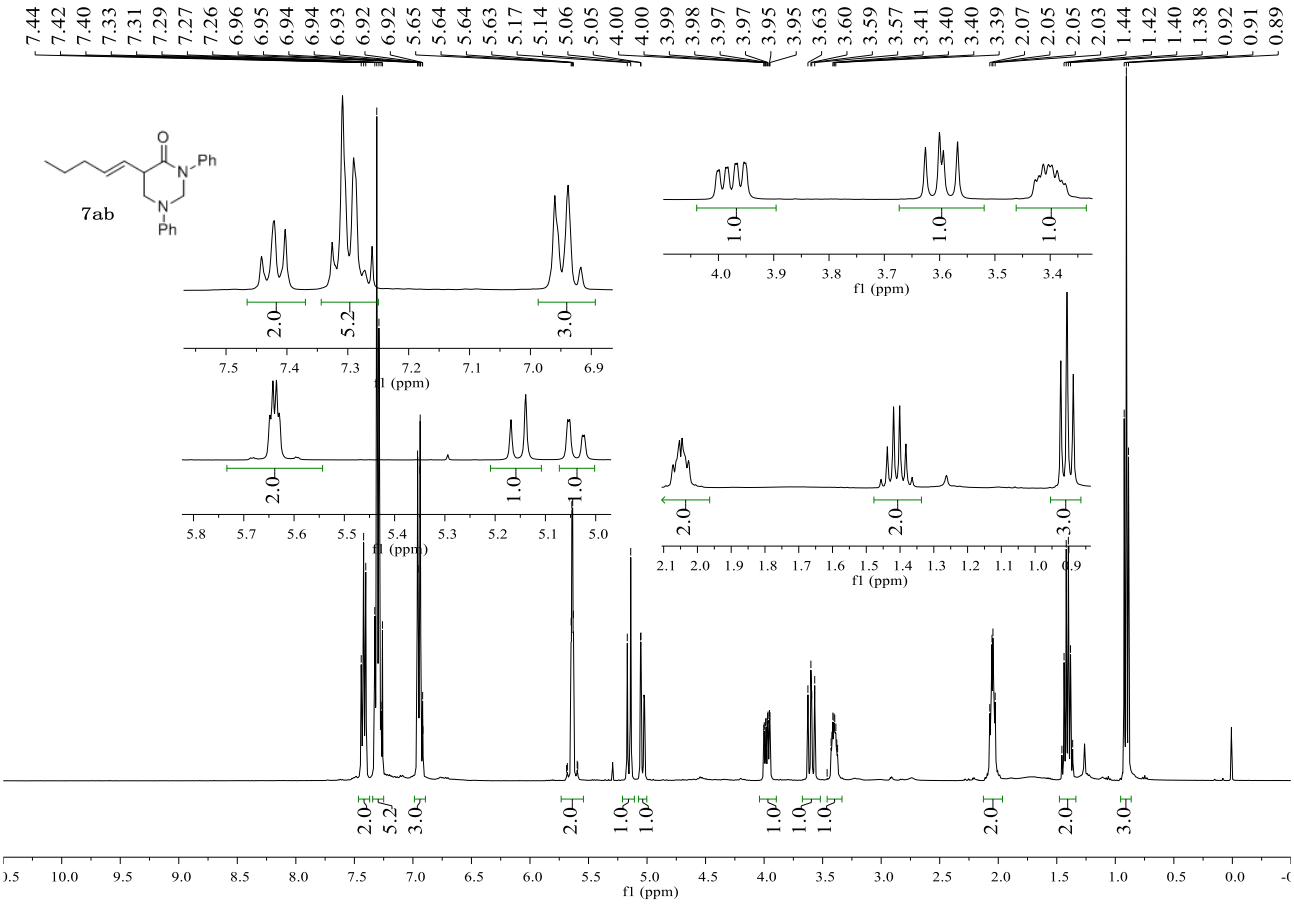
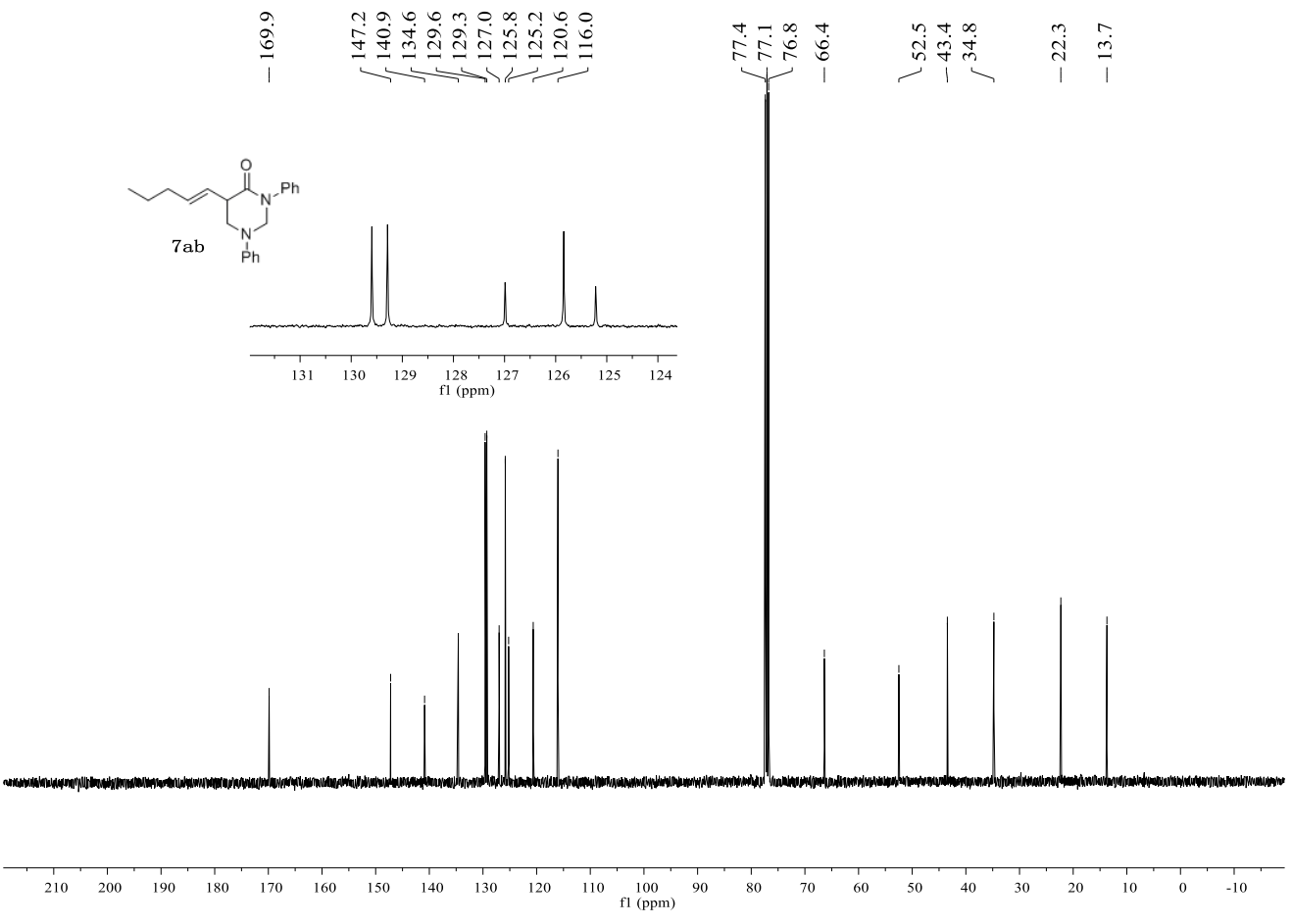
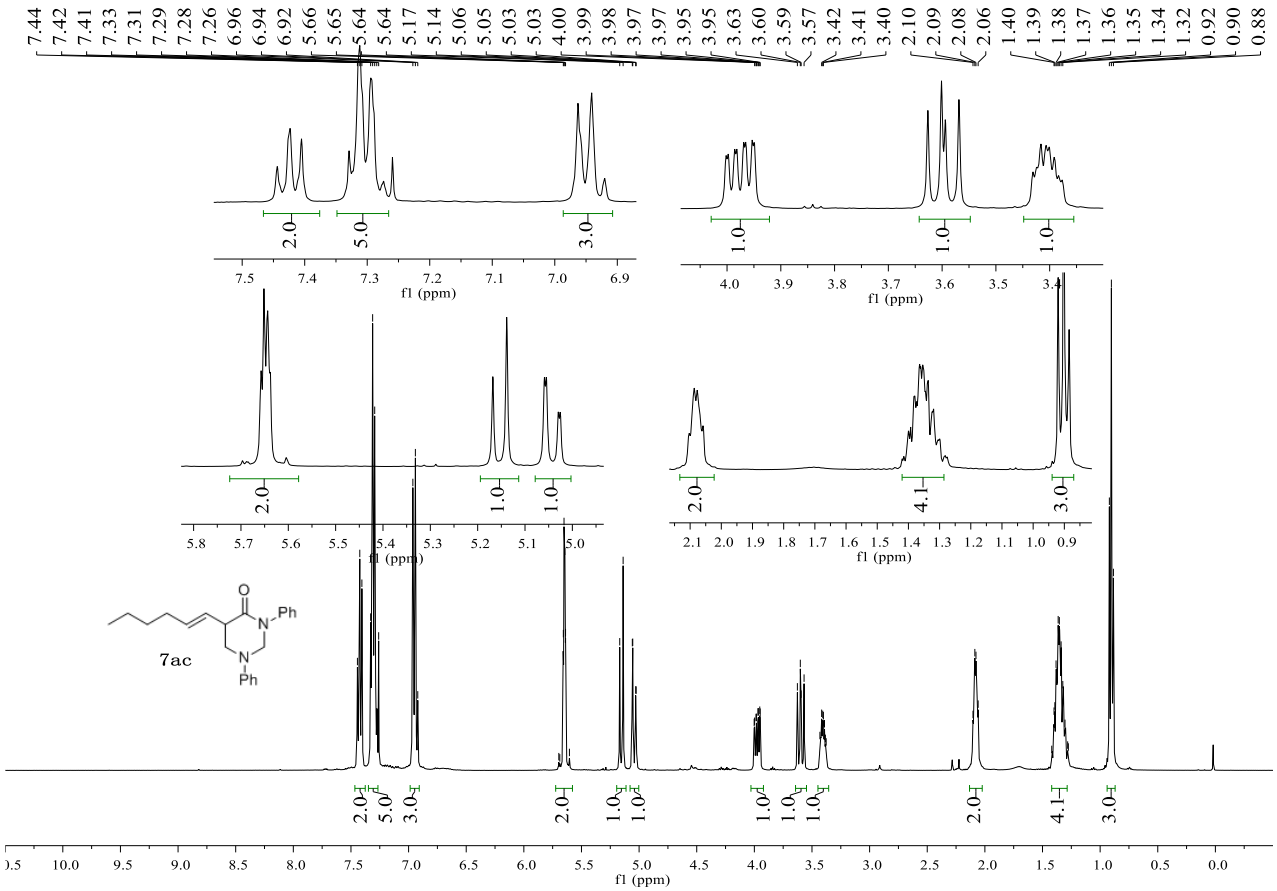
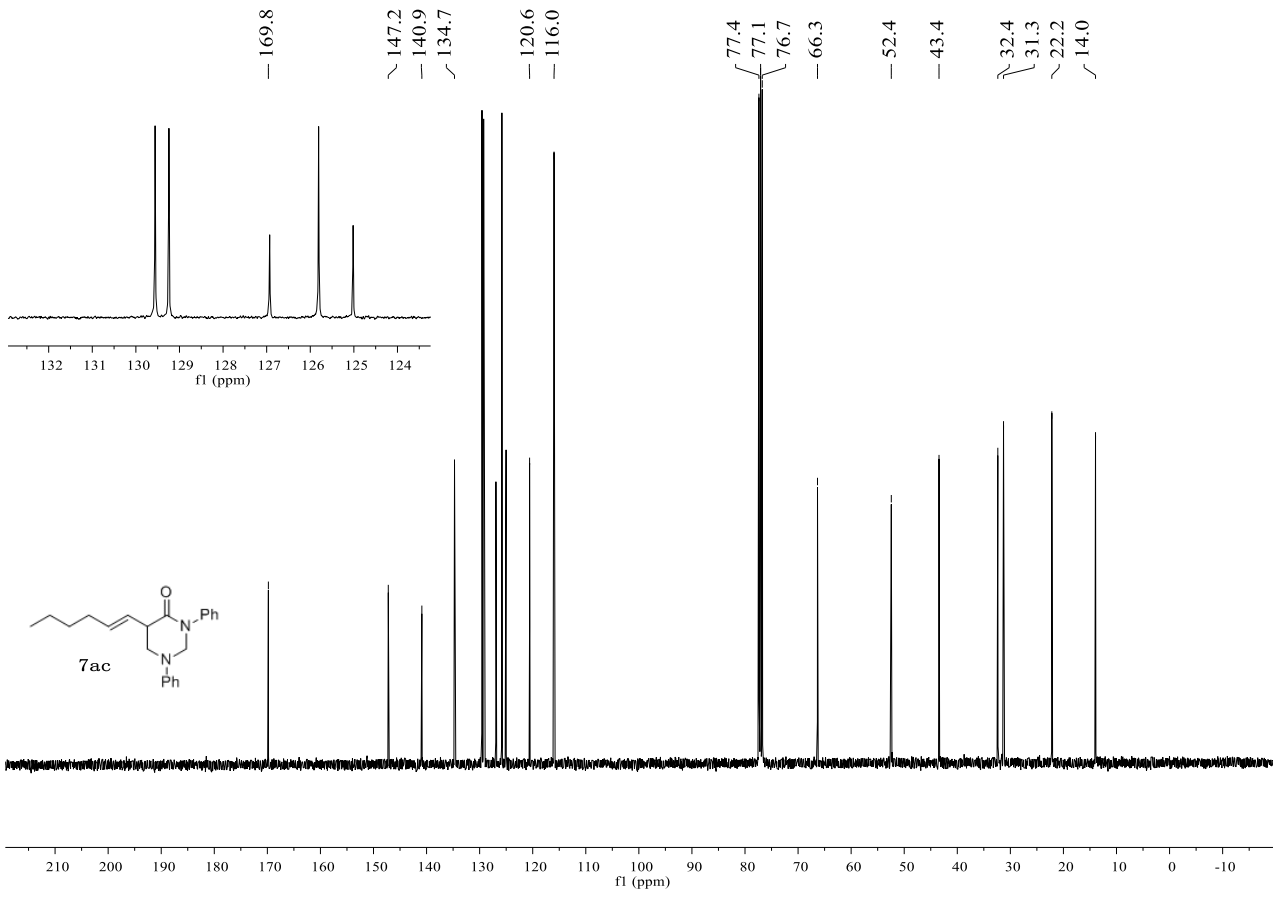
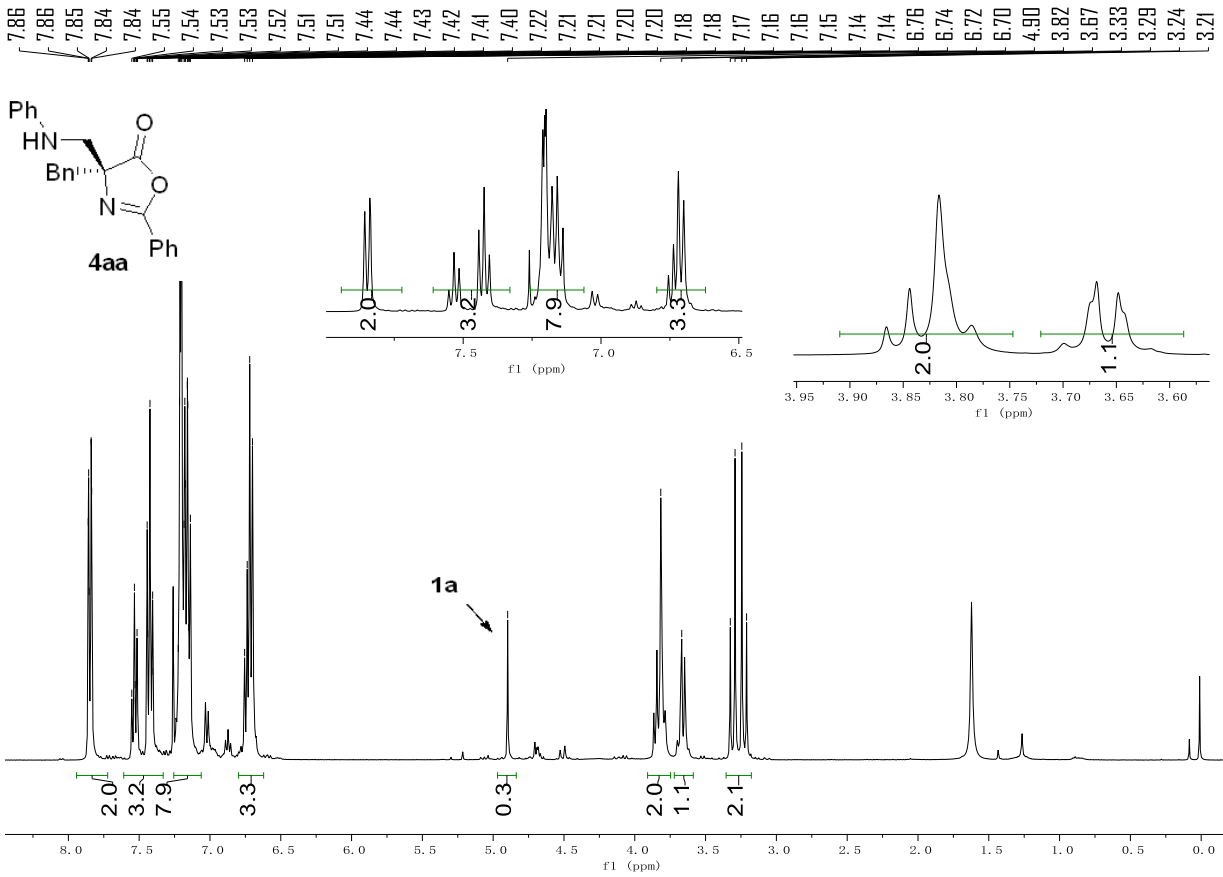
158.3
154.5
141.3
133.6
129.1
127.2
126.6
118.7
114.8
114.6
77.4
77.1
76.7
68.5
55.6
55.6
55.5
55.5
53.9
43.1
18.2
OMe
O
N
N
OMe
71a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

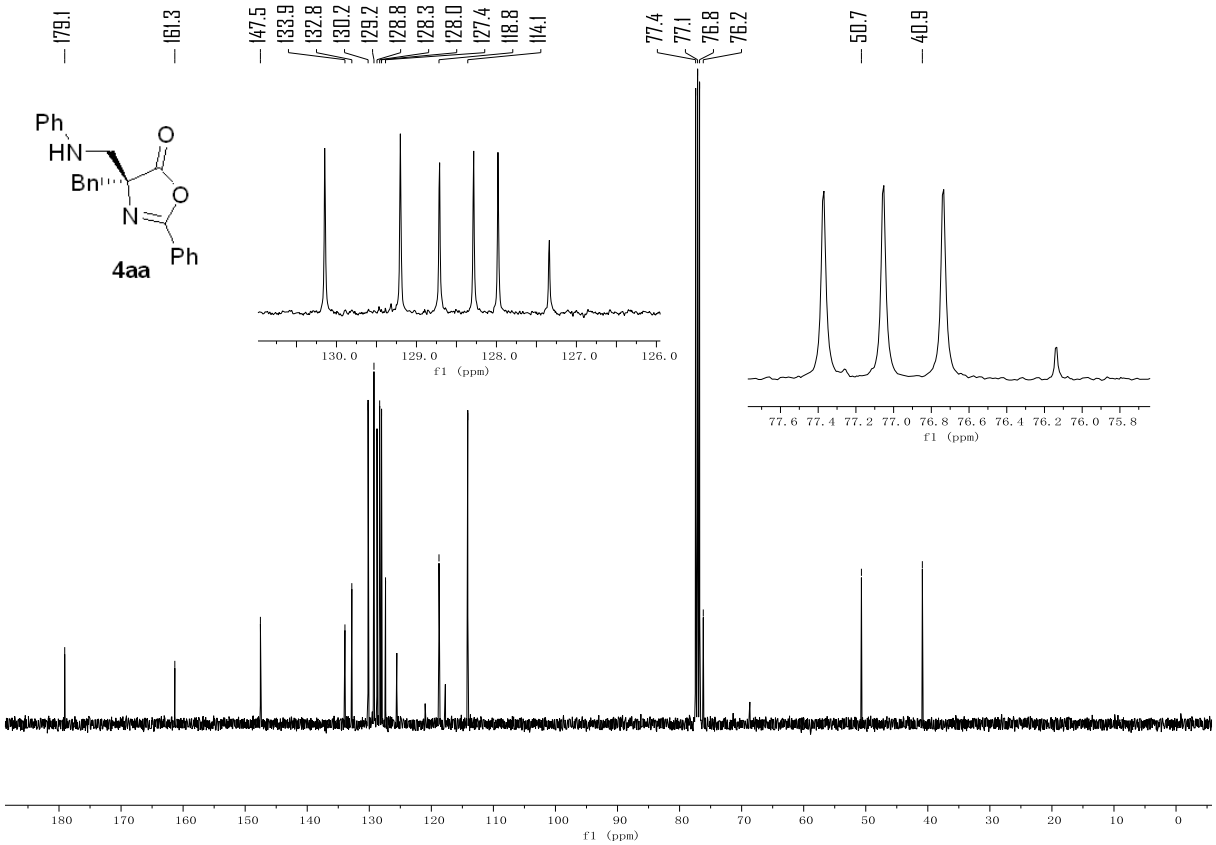

7.44 7.42 7.40 7.33 7.31 7.29 7.27 7.26 6.96 6.95 6.94 6.94 6.93 6.92 5.65 5.64 5.64 5.63 5.17 5.14 5.06 5.05 4.00 4.00 3.99 3.98 3.97 3.97 3.95 3.95 3.63 3.60 3.59 3.57 3.41 3.40 3.40 3.39 2.07 2.05 2.05 2.03 1.44 1.42 1.40 1.38 0.92 0.91 0.89
7ab
f1 (ppm)

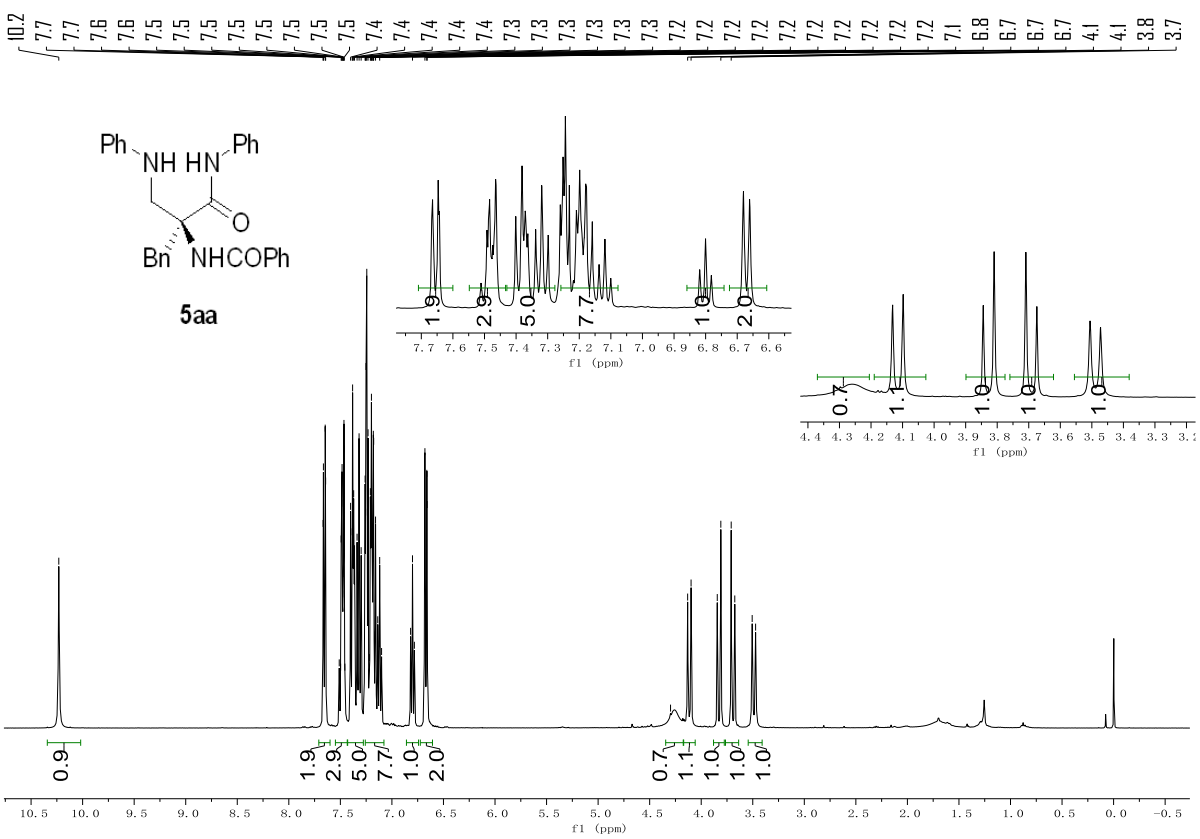

169.9 147.2 140.9 134.6 129.6 129.3 127.0 125.8 125.2 120.6 116.0 77.4 77.1 76.8 66.4 52.5 43.4 34.8 22.3 13.7
7ab
f1 (ppm)

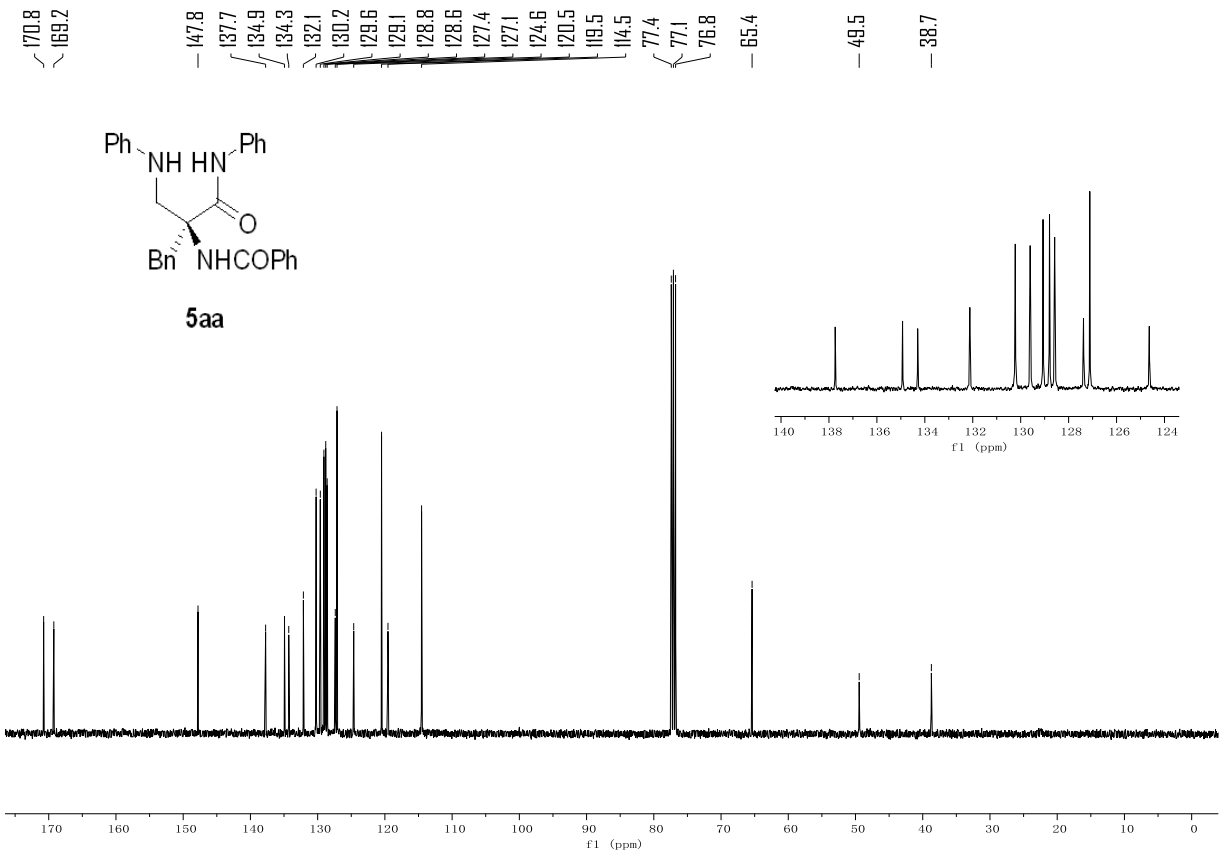

7.44 7.42 7.41 7.33 7.31 7.29 7.28 7.26 6.96 6.94 6.92 5.66 5.65 5.64 5.64 5.17 5.14 5.06 5.05 5.03 5.03 4.00 3.99 3.98 3.97 3.97 3.95 3.95 3.63 3.60 3.59 3.57 3.42 3.41 3.40 2.10 2.09 2.08 2.06 1.40 1.39 1.38 1.37 1.36 1.35 1.34 1.32 0.92 0.90 0.88
2.0 5.0 3.0
1.0 1.0 1.0
2.0 1.0 1.0
2.0 4.1 3.0
2.0 5.0 3.0 2.0 1.0 1.0 1.0 1.0 1.0 2.0 4.1 3.0
f1 (ppm)
7ac

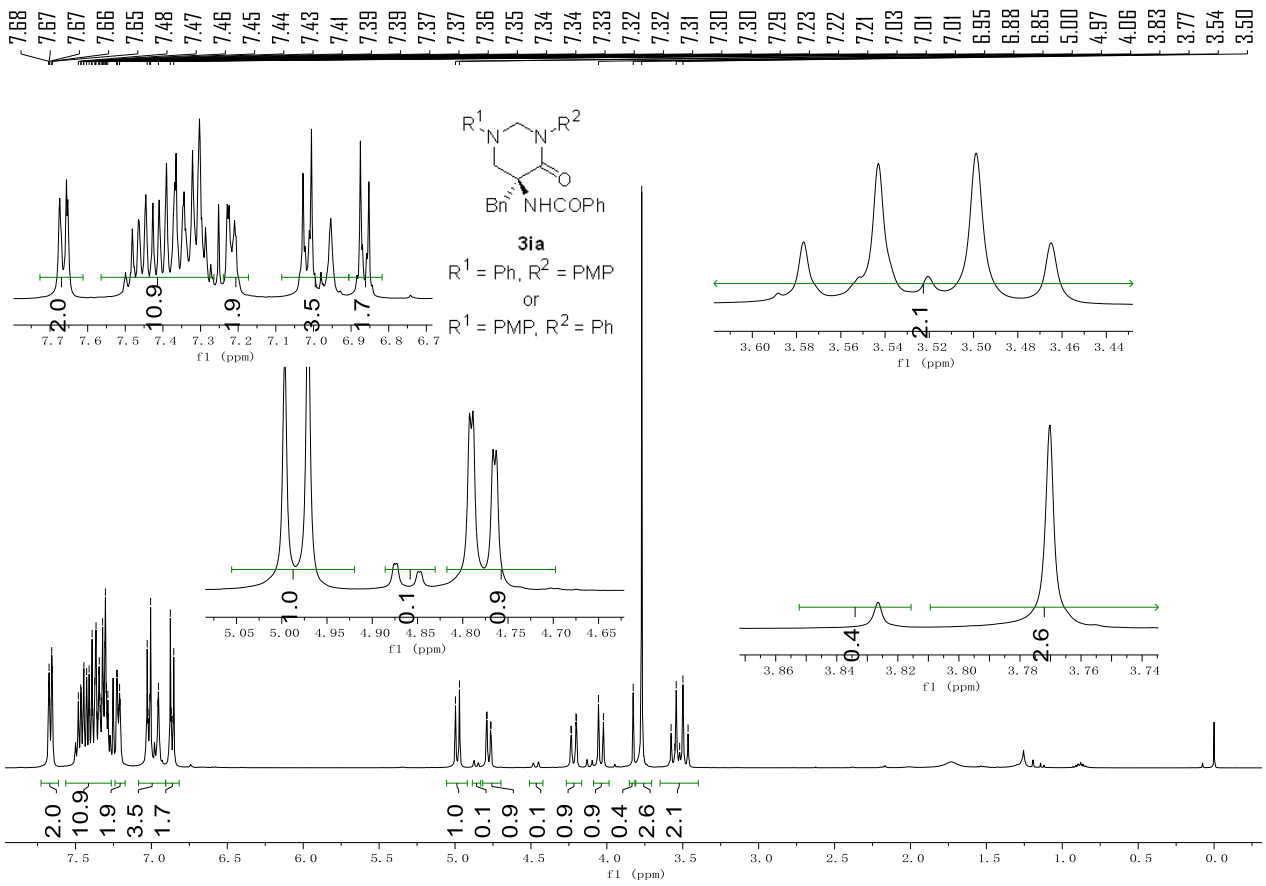

169.8
147.2 140.9 134.7
120.6 116.0
77.4 77.1 76.7
66.3
52.4
43.4
32.4 31.3 22.2 14.0
f1 (ppm)
7ac

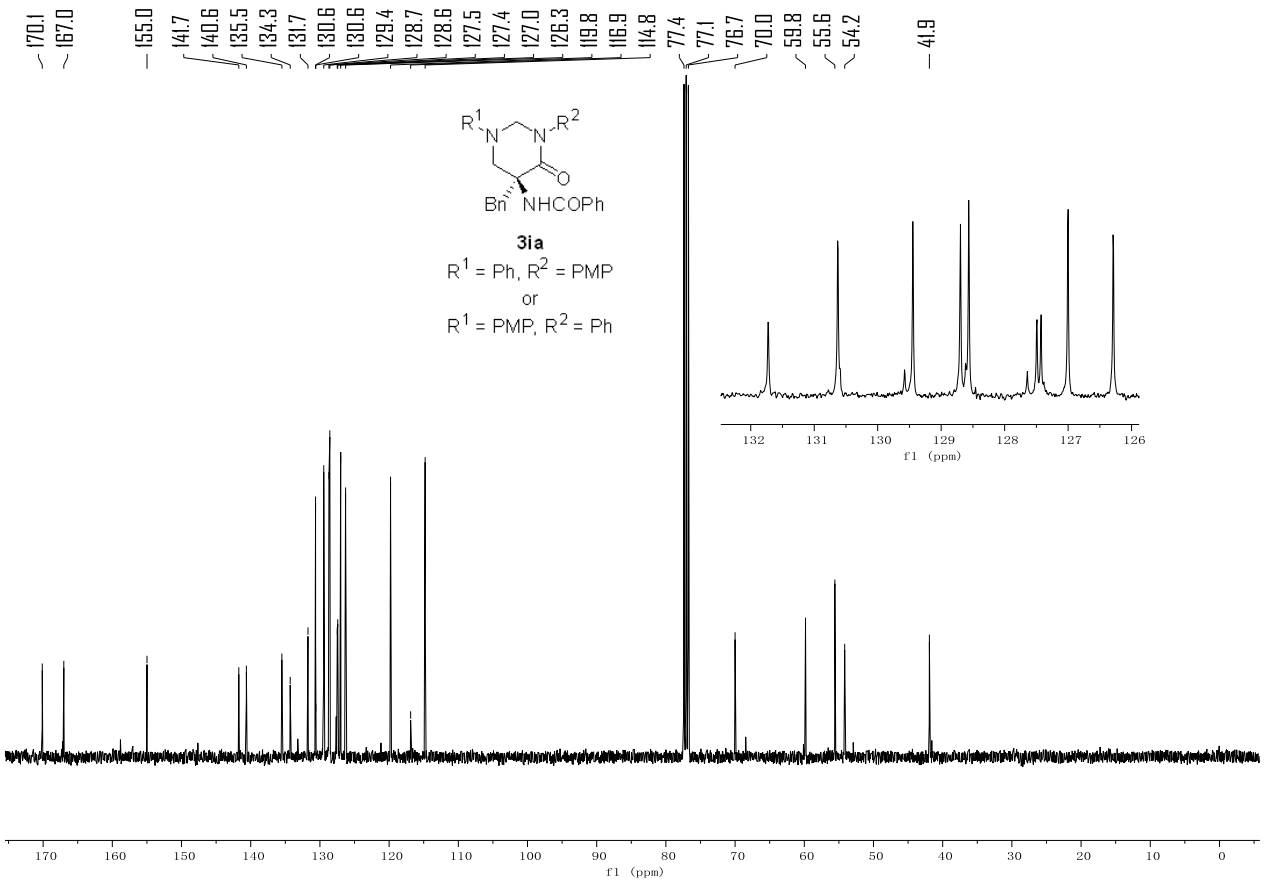

7.86 7.86 7.85 7.84 7.84 7.55 7.54 7.53 7.53 7.52 7.51 7.51 7.44 7.44 7.43 7.42 7.41 7.40 7.22 7.21 7.21 7.20 7.20 7.18 7.18 7.17 7.16 7.16 7.15 7.14 7.14 6.76 6.74 6.72 6.70 4.90 3.82 3.67 3.33 3.29 3.24 3.21
Ph
HN
Bn
O
O
N
Ph
4aa
2.0
3.2
7.9
3.3
2.0
1.1
1a
2.0
3.2
7.9
3.3
0.3
2.0
1.1
2.1
f1 (ppm)

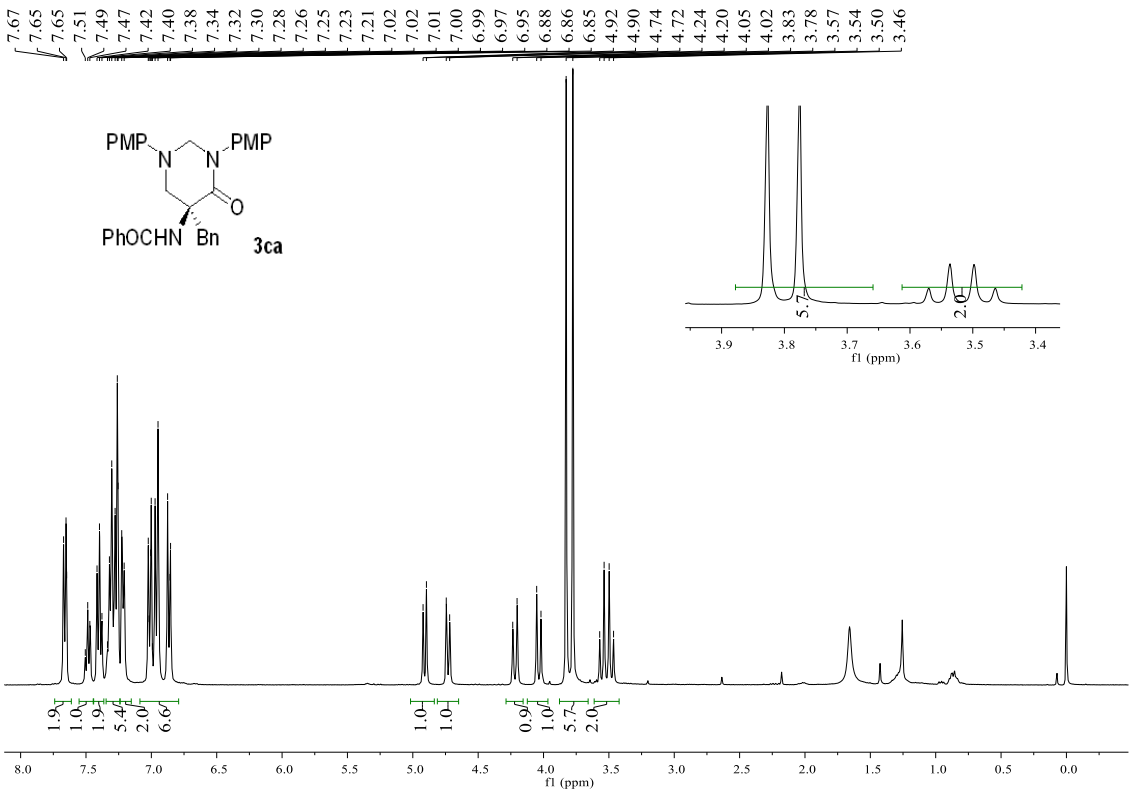

179.1
161.3
147.5
133.9
132.8
130.2
129.2
128.8
128.3
128.0
127.4
118.8
114.1
77.4
77.1
76.8
76.2
50.7
40.9
Ph
HN
Bn
O
O
N
Ph
4aa
f1 (ppm)

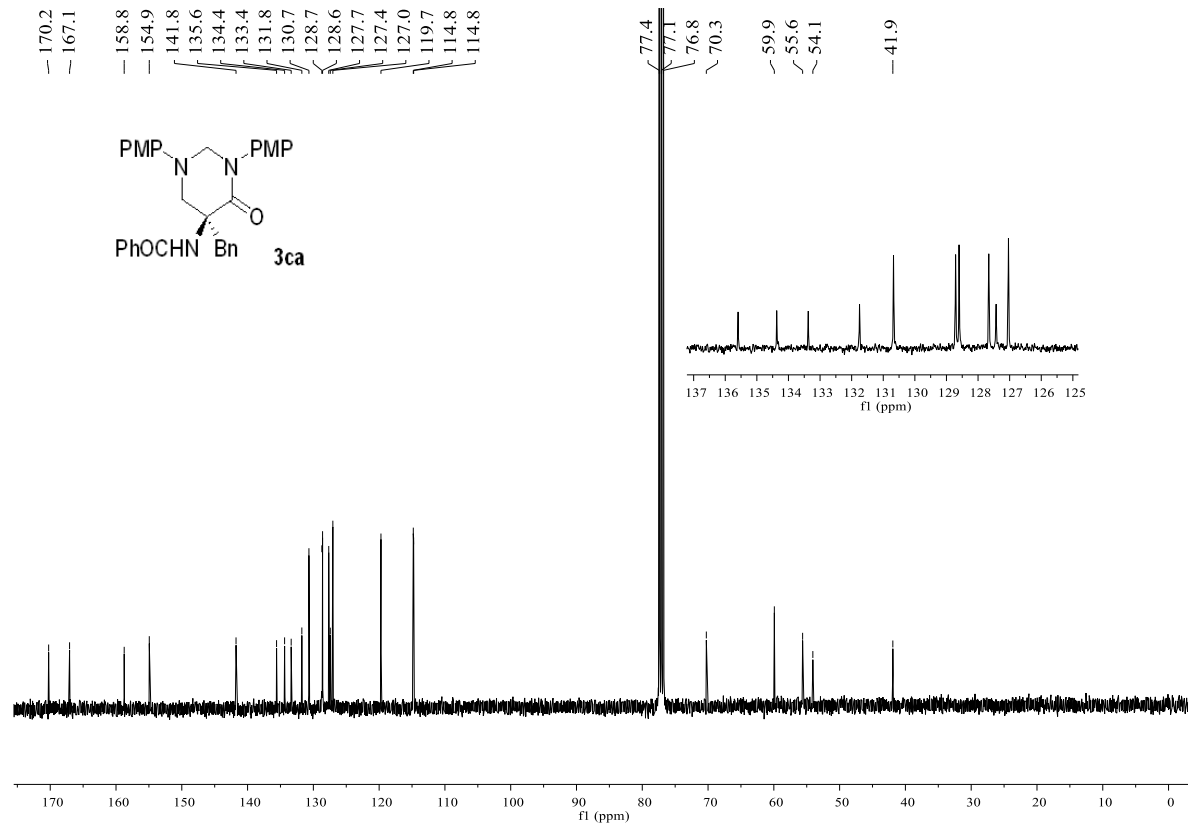

Ph
NH HN
Ph
O
Bn
NHCOPh
5aa
f1 (ppm)


Ph
NH HN
Ph
O
Bn
NHCOPh
5aa
f1 (ppm)


7.68 7.67 7.67 7.66 7.65 7.48 7.47 7.46 7.45 7.44 7.43 7.41 7.39 7.39 7.37 7.37 7.36 7.35 7.34 7.34 7.33 7.32 7.32 7.31 7.30 7.30 7.29 7.23 7.22 7.21 7.03 7.01 7.01 6.95 6.88 6.85 5.00 4.97 4.06 3.83 3.77 3.54 3.50
Bn NHCOPh
3ia
R1 = Ph, R2 = PMP
or
R1 = PMP, R2 = Ph
2.0 10.9 1.9 3.5 1.7
1.0 0.1 0.9 0.1 0.9 0.9 0.4 2.6 2.1
f1 (ppm)


170.1 167.0 155.0 141.7 140.6 135.5 134.3 131.7 130.6 130.6 129.4 128.7 128.6 127.5 127.4 127.0 126.3 119.8 116.9 114.8 77.4 77.1 76.7 70.0 59.8 55.6 54.2 41.9
Bn NHCOPh
3ia
R1 = Ph, R2 = PMP
or
R1 = PMP, R2 = Ph
f1 (ppm)


7.67
7.65
7.65
7.51
7.49
7.47
7.42
7.40
7.38
7.34
7.32
7.30
7.28
7.26
7.25
7.23
7.21
7.02
7.02
7.01
7.00
6.99
6.97
6.95
6.88
6.86
6.85
4.92
4.90
4.74
4.72
4.24
4.20
4.05
4.02
3.83
3.78
3.57
3.54
3.50
3.46
PMP
PMP
N
N
O
PhOCHN Bn
3ca
5.7
2.0
3.9
3.8
3.7
3.6
3.5
3.4
f1 (ppm)
1.9
1.0
1.9
5.4
2.0
6.6
1.0
1.0
0.9
1.0
5.7
2.0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


170.2
167.1
158.8
154.9
141.8
135.6
134.4
133.4
131.8
130.7
128.7
128.6
127.7
127.4
127.0
119.7
114.8
114.8
77.4
77.1
76.8
70.3
59.9
55.6
54.1
41.9
PMP
PMP
N
N
O
PhOCHN Bn
3ca
137
136
135
134
133
132
131
130
129
128
127
126
125
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)