

Supporting Information

Table of Contents

1. General experiment information.	S1
Table S1. Optimization of acyloxylation reaction with metal catalysts	S2
Table S2. Optimization of acyloxylation reaction with silver salts	S2
Table S3. Optimization of acyloxylation reaction with other conditions	S3
2. Preliminary mechanistic studies.	S4
2.1 Kinetic isotope effect of this transformation	S4
2.2 The effects of addition of radical scavengers of this reaction	S7
2.3 Study of intermediate.	S8
3. Gram-scale experiments	S9
4. Synthetic transformation	S9
Scheme S1. Pathways to obtain acetamide intermediate	S11
Scheme S2. Failure synthetic strategies to obtain macrocyclic compound.	S11
5. Single crystal data of product 3aa-8	S12
6. Characterization data of silver carboxylates 2a-1–2d-12	S22
7. Characterization data of products 3aa-1–3wa	S33
8. Copies of ^1H , ^{13}C and ^{19}F NMR spectra of silver carboxylates 2a-1–2d-12	S59
9. Copies of ^1H , ^{13}C and ^{19}F NMR spectra of products 3aa-1–3wa	S131
10. Failure examples of C–H bond acyloxylation	S215
11. References	S216

1. General experiment information.

General experimental. Unless otherwise noted, reactions were carried out in single-neck or two-neck flask round bottom flasks, with magnetic stirring. Air- or water-sensitive liquids and solutions were transferred *via* syringe. Organic solutions were concentrated by rotary evaporation at 23–40 °C under 40 Torr (house vacuum). Analytical thin layer chromatography (TLC) was performed with Silicycle normal phase glass plates (0.25 mm, 60-A pore size, 230–400 mesh). Visualization was done under a 254 nm UV light source. Purification of reaction products was generally done by flash chromatography with Silicycle 200–300 mesh silica gel.

Materials. Unless otherwise indicated, all reagents and solvents were purchased for commercial suppliers and used without additional purification. Distilled water was used in the reactions. Picolinamides **1a–1z** and **D-1a** were prepared according to literature procedures.^[S1]

Instrumentation. ¹H NMR, ¹³C NMR and ¹⁹F NMR spectra were recorded on a 400 MHz spectrometer (400 MHz for ¹H; 100 MHz for ¹³C; 376 MHz for ¹⁹F NMR) at room temperature. All chemical shift values are quoted in ppm referenced residual CHCl₃ at 7.26 ppm, Pyridine at 8.72 and DMSO at 2.50 ppm for ¹H NMR; relative to residual CHCl₃ at 77.0 ppm, Pyridine at 123.44 and DMSO at 40.0 ppm for ¹³C unless otherwise noted. HRMS (ion trap) were obtained from mass spectrometer (ESI). Melting points were recorded on an electrothermal digital melting point apparatus and were uncorrected.

The general procedure for silver carboxylates of 2a-1–2d-16: The substrate carboxylic acid **1** (6 mmol) and silver oxide (2 mmol) were added to 25.0 mL round-bottomed flask, followed by addition of 20.0 mL acetonitrile. The mixture was stirred at room temperature for 12 h. This solid was recovered by filtration and washed with 10 mL of cold acetonitrile (stored in dark). Subsequent drying under vacuum led to the desired product **2**.

The general procedure for new compounds of 3aa-1–3wa: The substrate picolinamides **1** (0.2 mmol), substrate silver carboxylates **2** (0.44 mmol) and CoCl₂ (0.02 mmol, 2.6 mg) were added to 5.0 mL round-bottomed flask, followed by addition of 2.0 mL toluene. The mixture was stirred at 110 °C for 12 h. After cooling to room temperature, the reaction mixture was added with 10 mL of water and then extracted by ethyl acetate (10 mL×3), the organic layers were combined, dried with anhydrous Na₂SO₄, and then filtered. The filtrate was concentrated under vacuum, and the resulting residue was purified by column chromatography (Silica Gel: 200–300 mesh) to afford the desired product **3**.

Table S1. Optimization of acyloxylation reaction with metal catalysts^[a]

Entry	Catalyst (x mol%)	Yield (%) ^[b]	r.r. (%) ^{[b],[c]}
1	none	N.R.	>95%
2	CuCl ₂ ·2H ₂ O (20)	N.R.	>95%
3	PdCl ₂ (20)	N.R.	>95%
4	CuI (20)	N.R.	70%
5	NiCl ₂ (20)	N.R.	>95%
6	[Rh(COD)Cl] ₂ (20)	N.R.	70%
7	[Ir(COD)Cl] ₂ (20)	N.R.	60%
8	Co(acac) ₃ (20)	N.R.	90%
9	Co(OAc)₂ (20)	90%	8%

^[a] The reaction was carried out with picolinamide **1a** (0.2 mmol, 49.6 mg), **catalyst** (20 mol%) and AgOAc (0.4 mmol, 56.6mg) in 2.0 mL of toluene at 100 °C for 24 h. ^[b] Determined by ¹H-NMR. ^[c] r.r. = Recovery rate.

Table S2. Optimization of acyloxylation reaction with silver salts^[a]

Entry	CoX ₂	AgX	Yield (%) ^[b]	r.r. (%) ^{[b],[c]}
1	CoCl ₂	AgF	N.R.	>95%
2	CoCl ₂	AgCl	N.R.	90%
3	CoBr ₂	AgBr	N.R.	>95%
4	CoI ₂	AgI	N.R.	90%
5	Co(OAc) ₂	AgOAc	90	<5%
6 ^[d]	CoCl ₂	AgOTfa	N.R.	>95%
7 ^[d]	CoCl ₂	AgOTf	N.R.	>95%
8	CoCl ₂	AgSCN	N.R.	>95%
9	CoCl ₂	AgSeCN	N.R.	>95%
10	CoCl ₂	AgOCN	N.R.	>95%
11	CoCl ₂	AgOMs	N.R.	90%
12	CoCl ₂	AgOTs	N.R.	90%
13	CoCl ₂	Tol-SOOAg	N.R.	95%
15	CoCl ₂	AgNBn ₂	N.R.	>95%

20	CoCl ₂	CH ₃ COSAg	N.R.	>95%
21	CoCl ₂		N.R.	>95%
22	CoCl ₂	AgNTf ₂	N.R.	>95%
23	CoCl ₂	AgBF ₄	N.R.	>95%
24	CoCl ₂	Ag ₂ WO ₃	N.R.	>95%

^[a]The reaction was carried out with picolinamide **1a** (0.2 mmol, 49.6 mg), Co catalyst (20 mol%) and AgX (0.4 mmol, 2.0 eq) in 2.0 mL of toluene at 100 °C for 24 h. ^[b]Determined by ¹H-NMR. ^[c]r.r. = Recovery rate. ^[d]Quenched with water before ¹H-NMR analysis.

Table S3. Optimization of acyloxylation reaction with other conditions^[a]

Entry	CoX ₂ (x mol%)	AgOAc (x eq.)	solvent	additive (x eq.)	Yield (%) ^[b]	
					Yield (%) ^[b]	Yield (%) ^[b]
1	Co(OAc) ₂ (20)	2.0	DMSO	-	9%	
2	Co(OAc) ₂ (20)	2.0	DME	-	82%	
3	Co(OAc) ₂ (20)	2.0	1,4-dioxane	-	58%	
4	Co(OAc) ₂ (20)	2.0	18-crown-6	-	20%	
5	Co(OAc) ₂ (20)	2.0	toluenen	-	90%	
6	Co(OAc) ₂ (20)	2.0	toluenen	Cs ₂ CO ₃ (2.0)	2%	
7	Co(OAc) ₂ (20)	2.0	toluenen	KOAc (2.0)	50%	
8	Co(OAc) ₂ (20)	2.0	toluenen	DMAP (2.0)	N.R.	
9	Co(OAc) ₂ (20)	2.0	toluenen	H ₂ O (5.0)	29%	
10 ^[c]	Co(OAc) ₂ (20)	2.0	toluenen	-	91%	
11 ^[d]	Co(OAc) ₂ (20)	2.0	toluenen	-	88%	
12 ^[e]	Co(OAc) ₂ (20)	2.0	toluenen	-	87%	
13	CoF ₂ (20)	2.0	toluenen	-	3%	
14	CoCl ₂ (20)	2.0	toluenen	-	91%	
15	CoBr ₂ (20)	2.0	toluenen	-	71%	
16	CoI ₂ (20)	2.0	toluenen	-	74%	
17	CoCO ₃ (20)	2.0	toluenen	-	<1%	
18	Co(SCN) ₂ (20)	2.0	toluenen	-	67%	
19	CoSO ₄ (20)	2.0	toluenen	-	8%	
20	Co(acac) ₃ (20)	2.0	toluenen	-	N.R.	
21 ^[f]	CoCl ₂ (1)	2.0	toluenen	-	12%	
22 ^[f]	CoCl ₂ (5)	2.0	toluenen	-	32%	
23 ^[f]	CoCl ₂ (10)	2.0	toluenen	-	83%	

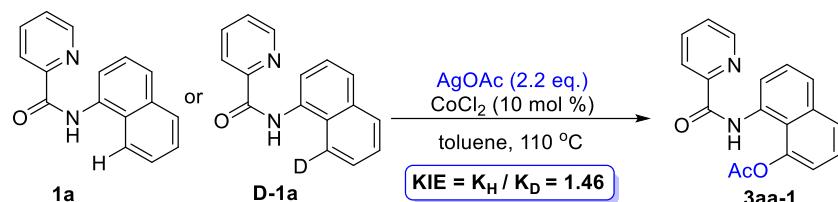
24	CoCl ₂ (10)	2.0	toluenen	-	60%
25	CoCl ₂ (10)	0.6	toluenen	-	23%
26	CoCl ₂ (10)	1.0	toluenen	-	43%
27	CoCl ₂ (10)	2.2	toluenen	-	82%
28 ^{[g],[h]}	CoCl ₂ (10)	2.2	toluenen	-	92%

^[a]Unless other notes, the reaction was carried out with picolinamide **1a** (0.2 mmol, 49.6 mg), Co catalyst (20 mmol%) and AgOAc (0.4 mmol, 66.8 mg, 2.0 eq) in 2.0 mL of solvent at 100 °C for 24 h.

^[b]Determined by ¹H-NMR. ^[c]Under O₂ atmosphere. ^[d]Under N₂ atmosphere. ^[e]Aged Co(OAc)₂. ^[f]For 48 h. ^[g]For 12 h. ^[h]At 110 °C.

2. Preliminary mechanistic studies.

2.1 Kinetic isotope effect (KIE) of the transformation



The picolinamides (**2a**, 0.2 mmol, 49.6 mg or **D-2a**, 0.2 mmol, 49.8 mg), AgOAc (**2a-1**, 0.44 mmol, 73.5 mg) and CoCl₂ (0.02 mmol, 2.6 mg) were added to 5.0 mL round-bottomed flask, followed by addition of 2.0 mL xylene as solvent. The mixture was stirred at 110 °C. An aliquot of each reaction mixture was taken at 1 h, 2 h, 3 h and 4 h. After the solvent of each aliquot (0.5 mL) was removed under reduced pressure conditions and analyzed by ¹H NMR spectrum in CDCl₃ (see Figure S1 and Figure S2). The relative yield of **1a** and **D-1a** were shown in Table S4. A sample plot of the initial rate data for the reaction of both **1a** and **D-1a** was shown in Figure S3 and Figure S4. The reaction progress in the early stage (0-4 h) indicated a kinetic isotope effect (KIE) of 1.46.

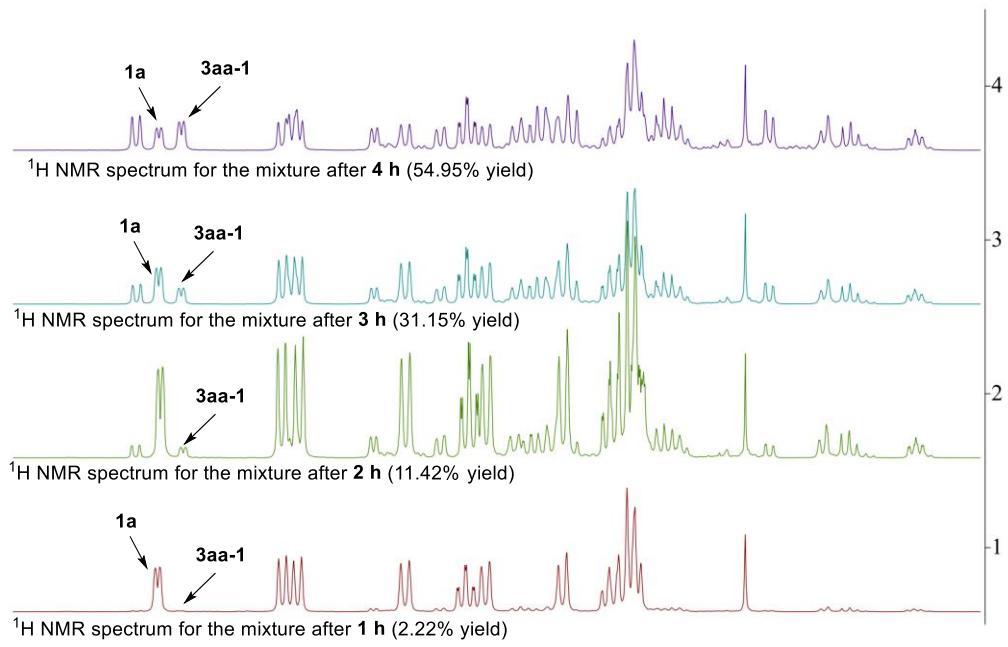


Figure S1. The conversion of **1a** was monitored by ¹H NMR method

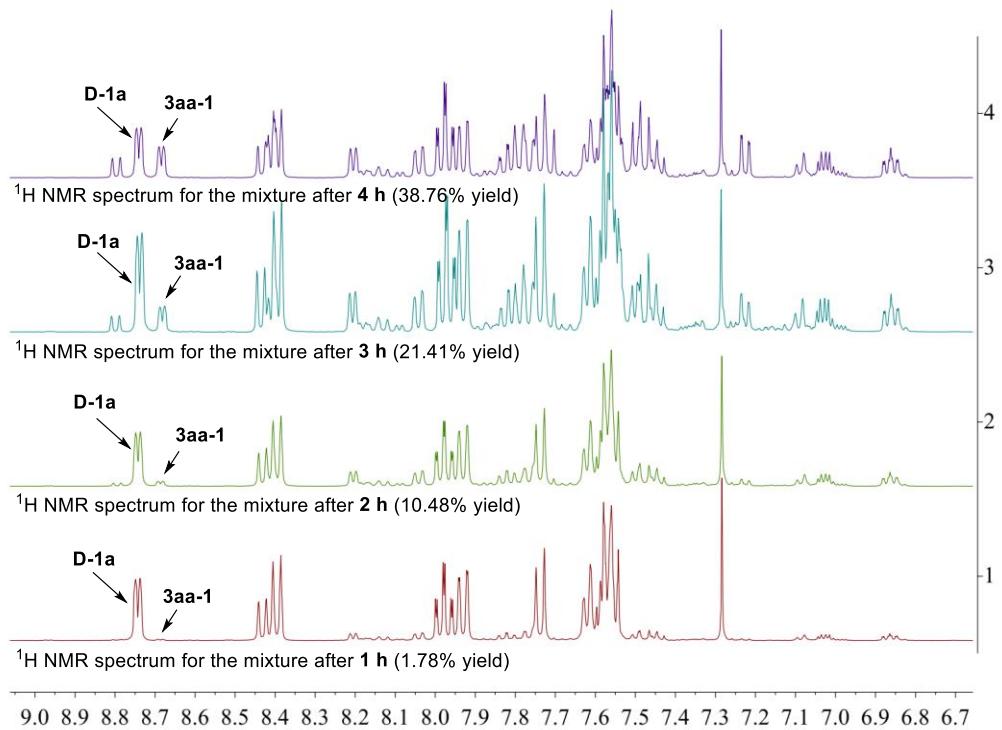


Figure S2. The conversion of **D-1a** was monitored by ¹H NMR method

Table S4. The relative yields (%) of 3aa-1 was monitored by ^1H NMR method.

Time [h]	1	2	3	4
^1H NMR yield of 3aa from 2a [%]	2.22	11.42	31.15	54.95
^1H NMR yield of 3aa from D-2a [%]	1.78	10.48	21.41	38.76

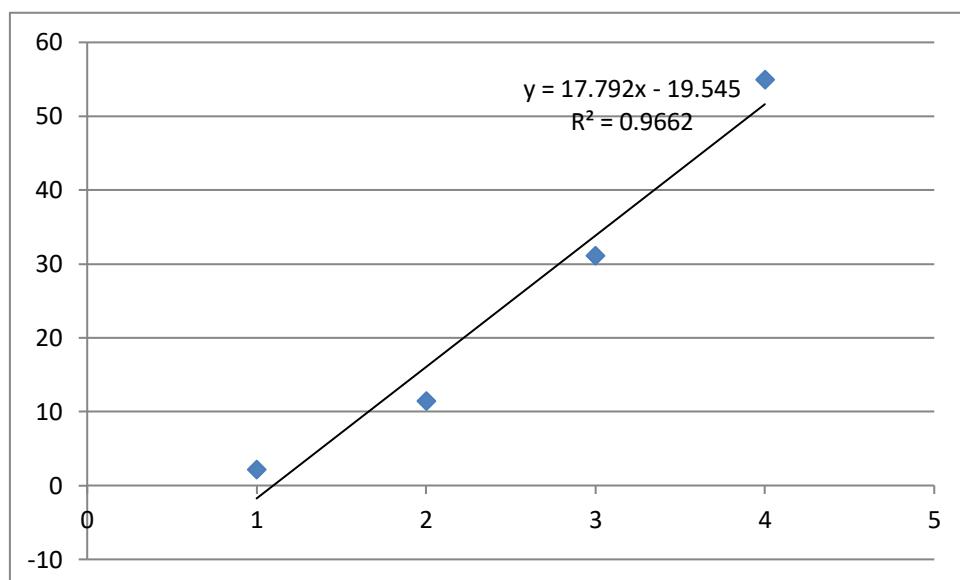


Figure S3. The plot of initial rates for the conversion of **1a**

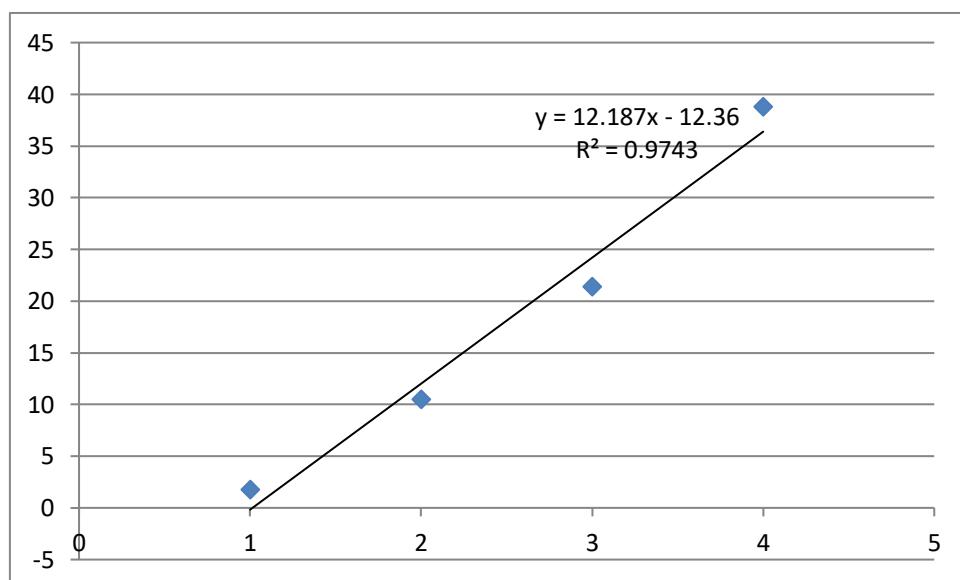
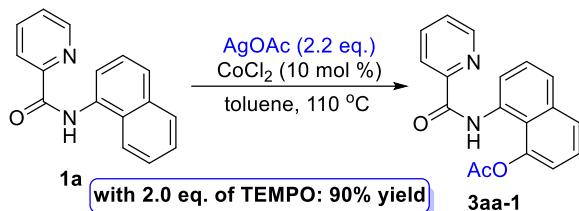
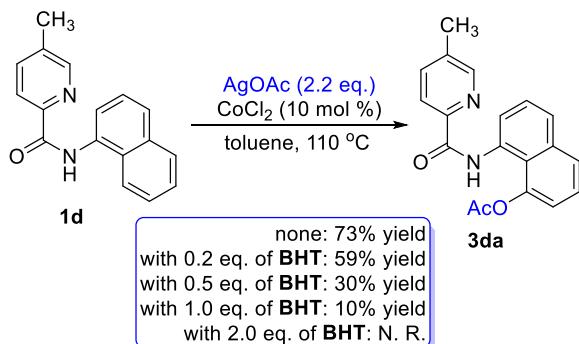


Figure S4. The plot of initial rates for the conversion of **D-1a**

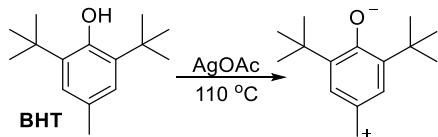
2.2 The effects of addition of radical scavengers of the reaction.



The picolinamides **1a** (0.2 mmol, 49.6 mg), CoCl_2 (0.02 mmol, 2.6 mg), AgOAc (0.44 mmol, 73.5 mg) and **TEMPO** (0.4 mmol, 62.5 mg, 2.0 eq.) were added to 5.0 mL round-bottomed flask, followed by addition of 2.0 mL toluene as solvent. The mixture was stirred at 110 °C for 12 h. After cooling to room temperature, the reaction mixture was added with 10 mL of water and then extracted by ethyl acetate (10 mL×3), the organic layers were combined, dried with anhydrous Na_2SO_4 , and then filtered. The filtrate was concentrated under vacuum, and the yield of **3aa-1** was determined by $^1\text{H-NMR}$ in CDCl_3 . (**The result indicated that radical process should not involve in the acyloxylation reaction.**)

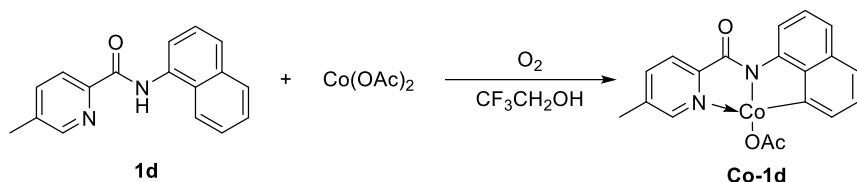


The picolinamides **1a** (0.2 mmol, 49.6 mg), CoCl_2 (0.02 mmol, 2.6 mg), AgOAc (0.44 mmol, 73.5 mg) and **BHT** were added to 5.0 mL round-bottomed flask, followed by addition of 2.0 mL toluene as solvent. The mixture was stirred at 110 °C for 12 h. After cooling to room temperature, the reaction mixture was added with 10 mL of water and then extracted by ethyl acetate (10 mL×3), the organic layers were combined, dried with anhydrous Na_2SO_4 , and then filtered. The filtrate was concentrated under vacuum, and the yields of **3da** with different loading amount of BTH was determined by $^1\text{H-NMR}$ in CDCl_3 . (**The result indicated that radical process should not involve in the acyloxylation reaction. BHT might be oxidized by AgOAc.**)

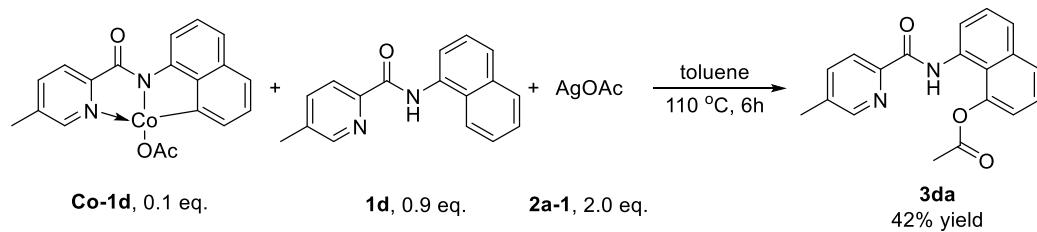


In order to figure out the relationship/reaction between AgOAc and BHT, the following reaction was carried out: AgOAc (0.2 mmol, 53.4 mg) and BHT (0.2 mmol, 44.0 mg) were added to 5.0 mL round-bottomed flask, followed by addition of 2.0 mL toluene as solvent. The mixture was stirred at 110 °C for 12 h. After cooling to room temperature, the reaction mixture was filtered. The filtrate was concentrated under vacuum. A crude ¹H-NMR analysis in CDCl₃ was tested without further purification. (The result of the ¹H-NMR analysis indicated that BHT could be oxidized by AgOAc. The oxidation reaction is similar to the reaction between BHT and Ag₂O in hexane reported by Prof. Richard Cosstick.^[S2])

2.3 Study of intermediate.

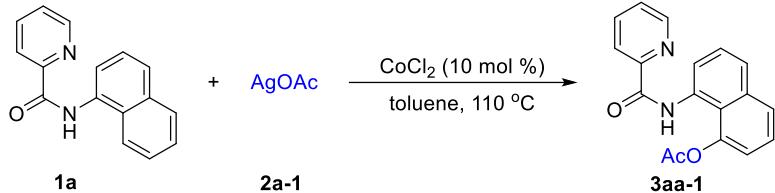


The picolinamides **1a** (0.5 mmol, 124.0 mg) and Co(OAc)₂ (0.5 mmol, 88.5 mg) were added to 25.0 mL round-bottomed flask, followed by addition of 5.0 mL CF₃CH₂OH as solvent. The mixture was stirred at 70 °C for 12 h under O₂ atmosphere. After cooling to room temperature, the reaction mixture was concentrated under vacuum to remove CF₃CH₂OH, and the resulting residue was purified by column chromatography (PE:EA=1:1, then 100% of EA) to afford the intermediate **Co-1d**.^[S3]



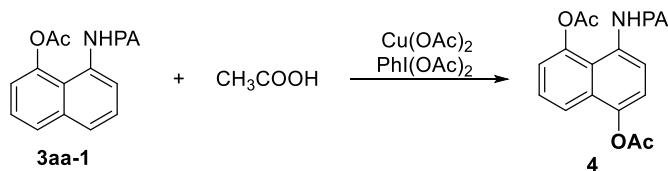
The intermediate **Co-1d** (0.02 mmol, 7.6 mg), picolinamides **1d** (0.18 mmol, 47.2 mg) and AgOAc (0.4 mmol, 66.8 mg) were added to 25.0 mL round-bottomed flask, followed by addition of 5.0 mL toluene as solvent. The mixture was stirred at 110 °C for **6 h**. After cooling to room temperature, the reaction mixture was added with 10 mL of water and then extracted by ethyl acetate (10 mL×3), the organic layers were combined, dried with anhydrous Na₂SO₄, and then filtered. The filtrate was concentrated under vacuum, and the resulting residue was purified by column chromatography to afford the desired product **3da** in 42% yield (vs the 73% yield under the optimized condition for **12 h**). (The result indicated that complex **Co-1d** should be the key intermediate for the further acyloxylation reaction to form product **3da**.)

3. Gram-scale experiments

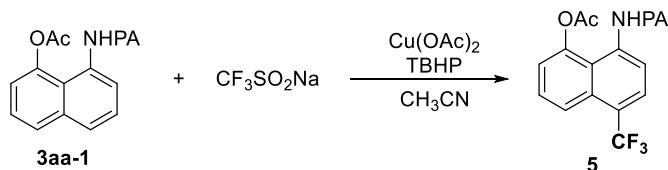


The picolinamides **1a** (0.5 mmol, 1.984 g), CoCl_2 (0.8 mmol, 0.104 g) and AgOAc (17.6 mmol, 2.939 g) were added to 100.0 mL round-bottomed flask, followed by addition of 50.0 mL toluene as solvent. The mixture was stirred at 110 °C for 12 h. After cooling to room temperature, the reaction mixture was added with 100 mL of water and then extracted by ethyl acetate (50 mL×3), the organic layers were combined, dried with anhydrous Na_2SO_4 , and then filtered. The filtrate was concentrated under vacuum, and the resulting residue was purified by column chromatography to afford the desired product **3aa-1** in 80% yield.

4. Synthetic transformation.

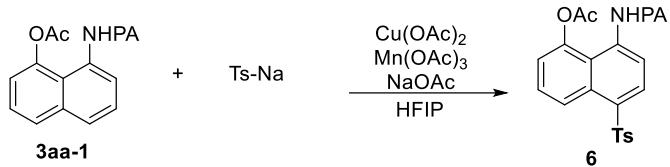


3aa-1 (61.2 mg, 0.2 mmol, 1.0 equiv.), $\text{Cu}(\text{OAc})_2$ (8.0 mg, 0.04 mmol, 0.2 equiv.), $\text{PhI}(\text{OAc})_2$ (128.8 mg, 0.4 mmol, 2.0 equiv.) and CH_3COOH (2 mL) were successively added into a sealed tube. The mixture was stirred at 80 °C under air for 12 h. After cooling to ambient temperature, the resulting mixture was filtered through a pad of tripolite and washed with ethyl acetate. The filtrate was concentrated under vacuum and purified by column chromatography (Silica Gel: 200–300 mesh) to afford the desired product **4**.

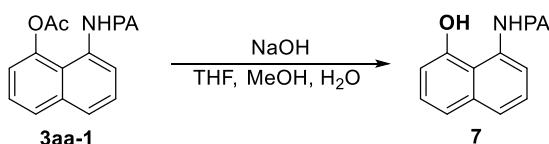


3aa-1 (61.2 mg, 0.2 mmol, 1.0 equiv.), $\text{Cu}(\text{OAc})_2$ (8.0 mg, 0.04 mmol, 0.2 equiv.), TBHP (5.5 mol / L in decane, 0.2 mmol, 1.0 equiv.), $\text{CF}_3\text{SO}_2\text{Na}$ (93.6 mg, 0.6 mmol, 3.0 equiv.), and CH_3CN (2 mL) were successively added into a sealed tube. The mixture was stirred at 80 °C under air for 48 h. After cooling to ambient temperature, the resulting mixture was filtered through a pad of tripolite and washed

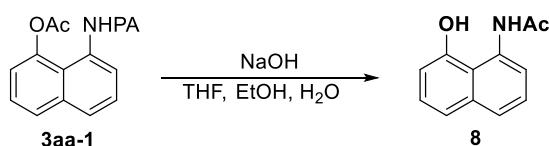
with ethyl acetate. The filtrate was concentrated under vacuum and purified by column chromatography (Silica Gel: 200–300 mesh) to afford the desired product **5**.



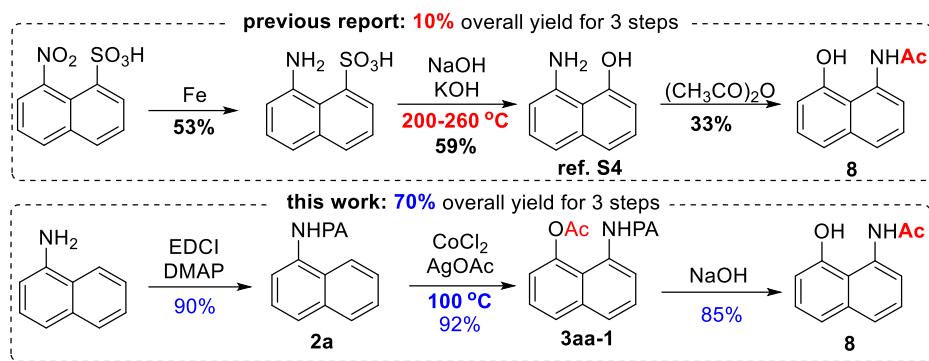
3aa-1 (61.2 mg, 0.2 mmol, 1.0 equiv.), Cu(OAc)₂ (8.0 mg, 0.04 mmol, 0.2 equiv.), Mn(OAc)₃ (185.6 mg, 0.8 mmol, 4.0 equiv.), NaOAc (49.2 mg, 0.6 mmol, 3.0 equiv.), TsNa (142.4 mg, 0.8 mmol, 4.0 equiv.), and HFIP (2 mL) were added into a sealed tube. The mixture was stirred at 60 °C for 2 h. After cooling to ambient temperature, the resulting mixture was filtered through a pad of tripolite and washed with ethyl acetate. The filtrate was concentrated under vacuum and purified by column chromatography (Silica Gel: 200–300 mesh) to afford the desired product **6**.



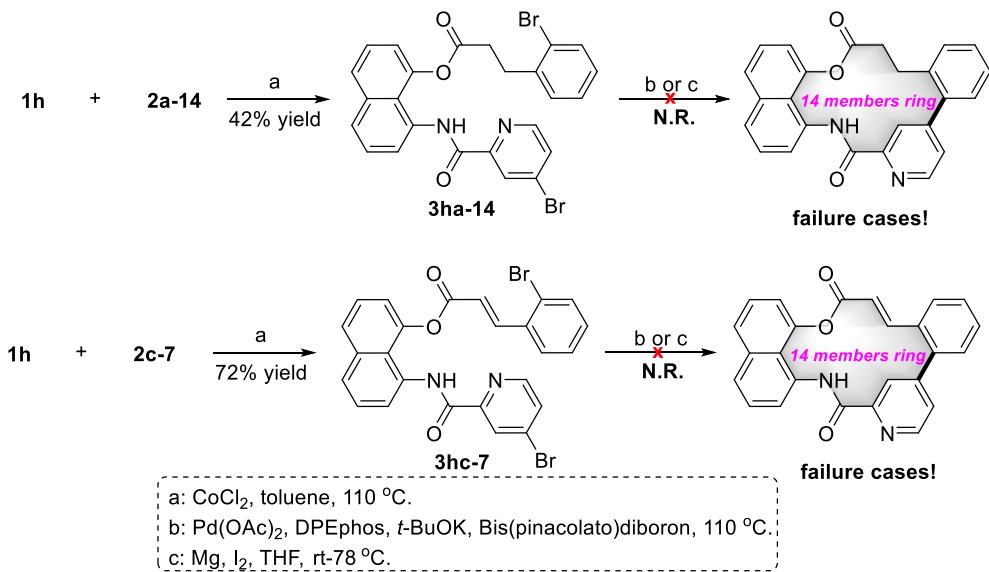
3aa-1 (61.2 mg, 0.2 mmol, 1.0 equiv.), NaOH (16.0 mg, 0.4 mmol, 2.0 equiv.), and solvent (THF/MeOH/H₂O, v:v:v=2:1:1, 2 mL) were successively added into a sealed tube. The mixture was stirred at room temperature for 12 h. The reaction mixture was added with 10 mL of H₂O and then extracted by DCM (10 mL×3), the organic layers were combined, dried with anhydrous Na₂SO₄, and then filtered. The filtrate was concentrated under vacuum, and the resulting residue was purified by column chromatography to afford the desired product **7**.



3aa-1 (61.2 mg, 0.2 mmol, 1.0 equiv.), NaOH (64.0 mg, 1.6 mmol, 8.0 equiv.), and solvent (THF/EtOH/H₂O, v:v:v=2:1:1, 2 mL) were successively added into a sealed tube. The mixture was stirred at 100 °C for 48 h under N₂ atmosphere. The reaction mixture was added with 10 mL of H₂O and then extracted by ethyl acetate (10 mL×3), the organic layers were combined, dried with anhydrous Na₂SO₄, and then filtered. The filtrate was concentrated under vacuum, and the resulting residue was purified by column chromatography to afford the desired product **8**.



Scheme S1. Pathways to obtain acetamide intermediate **8**



Scheme S2. Failure synthetic strategies to obtain macrocyclic compound

5. Single crystal data of product 3aa-8 (CCDC 2160352)

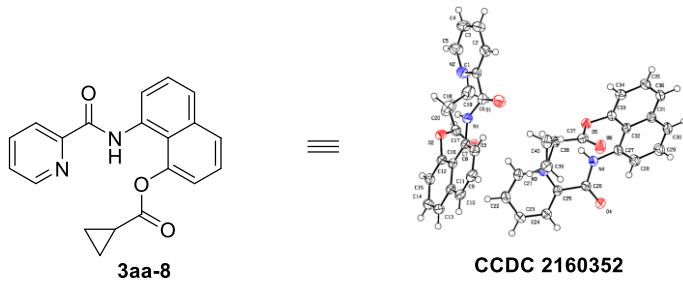


Figure S5. The single crystal structure of **3aa**

Table S3. Crystal data and structure refinement for **3aa-8**

Identification code	0912CHACC3_0m (3aa-8)	
Empirical formula	C ₂₀ H ₁₆ N ₂ O ₃	
Formula weight	332.35	
Temperature	150 K	
Wavelength	0.71073 Å	
Space group Crystal system	triclinic	
Space group IT number	2	
Space group name H-M alt	P -1	
Unit cell dimensions	a = 10.6196(8) Å	alpha = 75.187(2) deg.
	b = 12.7360(9) Å	beta = 70.684(2) deg.
	c = 13.6446(9) Å	gamma = 69.536(2) deg.
Volume	1611.1(2) Å ³	
Z, Calculated density	4, 1.370 Mg/m ³	
Absorption coefficient	0.094 mm ⁻¹	
F(000)	696	
Crystal size	0.15 x 0.08 x 0.05 mm	
Theta range for data collection	2.124 to 26.436 deg.	
Limiting indices	-13<=h<=13, -15<=k<=15, -17<=l<=14	
Reflections collected	4253	
Completeness to theta = 25.242	99.7%	
Absorption correction	SADABS-2016/2 (Bruker,2016/2)	
The ratio of min. to max. transmission	0.8704	
Refinement method	none	
Data / restraints / parameters	6559 / 0 / 451	
Goodness-of-fit on F ²	1.048	
Final R indices [Fo>4sigma(Fo)]	R1 = 0.0532	
R indices (all data)	R1 = 0.0991, wR2 = 0.1295	
Absolute structure parameter	n/a	
Largest diff. peak and hole	0.21 and -0.26 e.Å ⁻³	

Table S4. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3aa-8**

Atom	x/a	y/b	z/c	U(eq)
O6	0.9303(2)	1.0408(1)	0.8505(1)	0.0453(5)
O5	0.7635(2)	0.9509(1)	0.9163(1)	0.0279(4)
O4	0.5028(2)	1.2659(1)	0.7073(1)	0.0357(4)
O3	0.5618(2)	0.7053(1)	0.6329(1)	0.0406(4)
O2	0.7443(2)	0.5550(1)	0.5824(1)	0.0282(4)
O1	1.0380(2)	0.5891(2)	0.7826(1)	0.0500(5)
N4	0.5673(2)	1.1096(2)	0.8276(1)	0.0274(4)
N3	0.6761(2)	0.9781(2)	0.6807(1)	0.0300(4)
N2	0.8121(2)	0.4237(2)	0.8281(1)	0.0340(5)
N1	0.9361(2)	0.5601(2)	0.6710(1)	0.0281(4)
H9	1.24650	0.73480	0.45410	0.0440
H8	1.14890	0.63650	0.61540	0.0390
H5	0.69610	0.31920	0.88800	0.0510
H4A	0.61160	1.03650	0.83320	0.0330
H40B	1.14390	0.76480	0.72500	0.0460
H40A	1.15520	0.87100	0.76910	0.0460
H4	0.78200	0.22880	1.03300	0.0540
H39B	1.03440	1.00530	0.64770	0.0500
H39A	1.02310	0.89910	0.60370	0.0500
H38	0.89170	0.83510	0.77720	0.0350
H36	0.55160	1.14290	1.24470	0.0370
H35	0.74380	0.98560	1.23490	0.0400
H34	0.84220	0.90160	1.08110	0.0370
H30	0.37150	1.28510	1.16920	0.0400
H3	0.95060	0.28740	1.06310	0.0520
H29	0.26980	1.36390	1.03180	0.0440
H28	0.35980	1.28430	0.87720	0.0390
H24	0.54020	1.20470	0.53270	0.0350
H23	0.62070	1.07740	0.41230	0.0390
H22	0.73560	0.88750	0.46350	0.0400
H21	0.77080	0.83090	0.63010	0.0420
H20B	0.35920	0.47830	0.73550	0.0650
H20A	0.34700	0.61490	0.68860	0.0650
H2	1.03340	0.43200	0.94000	0.0430
H19B	0.42710	0.62630	0.82790	0.0660
H19A	0.43930	0.48970	0.87480	0.0660
H18	0.60750	0.43690	0.71280	0.0400
H15	0.67020	0.63250	0.41500	0.0360

H14	0.76650	0.74180	0.25960	0.0410
H13	0.95990	0.79300	0.24650	0.0390
H10	1.14400	0.78730	0.31800	0.0410
H1	0.87500	0.52800	0.67100	0.0340
C9	1.1644(3)	0.7154(2)	0.4630(2)	0.0364(6)
C8	1.1063(2)	0.6556(2)	0.5595(2)	0.0326(5)
C7	0.9892(2)	0.6242(2)	0.5747(2)	0.0267(5)
C6	0.9679(2)	0.5421(2)	0.7635(2)	0.0312(5)
C5	0.7665(3)	0.3418(2)	0.8984(2)	0.0425(6)
C40	1.1019(2)	0.8442(2)	0.7383(2)	0.0385(6)
C4	0.8161(3)	0.2880(2)	0.9858(2)	0.0448(7)
C39	1.0274(3)	0.9269(2)	0.6636(2)	0.0416(6)
C38	0.9450(2)	0.8883(2)	0.7716(2)	0.0291(5)
C37	0.8843(2)	0.9687(2)	0.8467(2)	0.0296(5)
C36	0.5900(2)	1.1090(2)	1.1828(2)	0.0310(5)
C35	0.7039(3)	1.0164(2)	1.1771(2)	0.0331(5)
C34	0.7624(2)	0.9661(2)	1.0855(2)	0.0312(5)
C33	0.7044(2)	1.0100(2)	1.0029(2)	0.0262(5)
C32	0.5850(2)	1.1059(2)	1.0040(2)	0.0246(5)
C31	0.5278(2)	1.1558(2)	1.0984(2)	0.0282(5)
C30	0.4092(2)	1.2524(2)	1.1066(2)	0.0333(5)
C3	0.9159(3)	0.3217(2)	1.0030(2)	0.0435(6)
C29	0.3490(3)	1.2988(2)	1.0256(2)	0.0365(6)
C28	0.4033(2)	1.2510(2)	0.9329(2)	0.0329(5)
C27	0.5176(2)	1.1576(2)	0.9209(2)	0.0265(5)
C26	0.5540(2)	1.1639(2)	0.7305(2)	0.0257(5)
C25	0.6088(2)	1.0863(2)	0.6504(2)	0.0244(5)
C24	0.5870(2)	1.1270(2)	0.5516(2)	0.0293(5)
C23	0.6348(2)	1.0521(2)	0.4807(2)	0.0329(5)
C22	0.7026(2)	0.9408(2)	0.5105(2)	0.0337(6)
C21	0.7221(2)	0.9078(2)	0.6104(2)	0.0346(6)
C20	0.3951(3)	0.5430(2)	0.7270(3)	0.0545(8)
C2	0.9644(3)	0.4068(2)	0.9308(2)	0.0357(6)
C19	0.4445(3)	0.5499(2)	0.8124(2)	0.0547(8)
C18	0.5495(2)	0.5180(2)	0.7121(2)	0.0334(5)
C17	0.6131(2)	0.6043(2)	0.6417(2)	0.0286(5)
C16	0.8040(2)	0.6249(2)	0.4936(2)	0.0266(5)
C15	0.7486(2)	0.6551(2)	0.4101(2)	0.0299(5)
C14	0.8068(2)	0.7192(2)	0.3171(2)	0.0338(5)
C13	0.9212(2)	0.7491(2)	0.3096(2)	0.0324(5)
C12	0.9235(2)	0.6529(2)	0.4911(2)	0.0265(5)
C11	0.9836(2)	0.7159(2)	0.3940(2)	0.0291(5)

C10	1.1046(2)	0.7458(2)	0.3825(2)	0.0338(6)
C1	0.9102(2)	0.4541(2)	0.8452(2)	0.0285(5)

Table S5. Bond lengths [Å] and angles [deg] for **3aa-8**

O2-C17	1.375(3)
O2-C16	1.413(2)
O5-C33	1.410(2)
O5-C37	1.374(3)
O4-C26	1.222(2)
O3-C17	1.201(3)
O6-C37	1.200(3)
O1-C6	1.225(3)
N1-H1	0.8800
N1-C7	1.412(3)
N1-C6	1.355(3)
N4-H4A	0.8800
N4-C26	1.357(3)
N4-C27	1.414(3)
N3-C25	1.341(3)
N3-C21	1.336(3)
N2-C1	1.335(3)
N2-C5	1.333(3)
C33-C32	1.419(3)
C33-C34	1.363(3)
C26-C25	1.496(3)
C25-C24	1.381(3)
C32-C27	1.435(3)
C32-C31	1.436(3)
C11-C12	1.435(3)
C11-C13	1.416(3)
C11-C10	1.413(3)
C12-C7	1.435(3)
C12-C16	1.421(3)
C7-C8	1.373(3)
C17-C18	1.462(3)
C1-C6	1.505(3)
C1-C2	1.377(3)
C37-C38	1.467(3)
C27-C28	1.369(3)
C15-H15	0.9500
C15-C16	1.361(3)
C15-C14	1.401(3)

C31-C36	1.413(3)
C31-C30	1.417(3)
C8-H8	0.9500
C8-C9	1.405(3)
C24-H24	0.9500
C24-C23	1.383(3)
C13-H13	0.9500
C13-C14	1.361(3)
C34-H34	0.9500
C34-C35	1.406(3)
C35-H35	0.9500
C35-C36	1.359(3)
C38-H38	1.0000
C38-C40	1.508(3)
C38-C39	1.502(3)
C23-H23	0.9500
C23-C22	1.370(3)
C14-H14	0.9500
C10-H10	0.9500
C10-C9	1.353(3)
C28-H28	0.9500
C28-C29	1.403(3)
C36-H36	0.9500
C22-H22	0.9500
C22-C21	1.382(3)
C2-H2	0.9500
C2-C3	1.386(3)
C30-H30	0.9500
C30-C29	1.360(3)
C18-H18	1.0000
C18-C20	1.509(3)
C18-C19	1.496(3)
C21-H21	0.9500
C29-H29	0.9500
C9-H9	0.9500
C40-H40A	0.9900
C40-H40B	0.9900
C40-C39	1.475(3)
C39-H39A	0.9900
C39-H39B	0.9900
C3-H3	0.9500
C3-C4	1.377(4)

C5-H5	0.9500
C5-C4	1.384(3)
C4-H4	0.9500
C20-H20A	0.9900
C20-H20B	0.9900
C20-C19	1.459(4)
C19-H19A	0.9900
C19-H19B	0.9900
C17-O2-C16	116.98(16)
C37-O5-C33	117.10(15)
C7-N1-H1	116.100
C6-N1-H1	116.100
C6-N1-C7	127.88(18)
C26-N4-H4A	116.500
C26-N4-C27	127.07(19)
C27-N4-H4A	116.500
C21-N3-C25	116.93(19)
C5-N2-C1	116.9(2)
O5-C33-C32	120.13(18)
C34-C33-O5	116.7(2)
C34-C33-C32	123.0(2)
O4-C26-N4	125.2(2)
O4-C26-C25	121.29(19)
N4-C26-C25	113.49(19)
N3-C25-C26	116.73(19)
N3-C25-C24	123.3(2)
C24-C25-C26	120.0(2)
C33-C32-C27	126.74(19)
C33-C32-C31	115.78(19)
C27-C32-C31	117.5(2)
C13-C11-C12	120.1(2)
C10-C11-C12	120.2(2)
C10-C11-C13	119.7(2)
C7 C12-C11	117.54(19)
C16 C12-C11	115.75(19)
C16 C12-C7	126.71(19)
N1-C7-C12	119.83(18)
C8-C7-N1	120.3(2)
C8-C7-C12	119.9(2)
O2-C17-C18	110.97(18)
O3-C17-O2	122.2(2)
O3-C17-C18	126.8(2)

N2-C1-C6	117.3(2)
N2-C1-C2	123.8(2)
C2-C1-C6	118.8(2)
O5-C37-C38	110.67(18)
O6-C37-O5	122.5(2)
O6-C37-C38	126.8(2)
N4-C27-C32	119.63(19)
C28-C27-N4	120.1(2)
C28-C27-C32	120.2(2)
C16-C15-H15	119.800
C16-C15-C14	120.3(2)
C14-C15-H15	119.800
C36-C31-C32	120.1(2)
C36-C31-C30	120.0(2)
C30-C31-C32	119.9(2)
O2-C16-C12	120.00(19)
C15-C16-O2	116.85(19)
C15-C16-C12	122.95(19)
C7-C8-H8	119.300
C7-C8-C9	121.4(2)
C9-C8-H8	119.300
C25-C24-H24	120.800
C25-C24-C23	118.5(2)
C23-C24-H24	120.800
C11-C13-H13	119.400
C14-C13-C11	121.2(2)
C14-C13-H13	119.400
O1-C6-N1	125.8(2)
O1-C6-C1	120.5(2)
N1-C6-C1	113.64(19)
C33-C34-H34	120.000
C33-C34-C35	120.0(2)
C35-C34-H34	120.000
C34-C35-H35	120.100
C36-C35-C34	119.8(2)
C36-C35-H35	120.100
C37-C38-H38	117.000
C37-C38-C40	116.79(19)
C37-C38-C39	117.44(19)
C40-C38-H38	117.000
C39-C38-H38	117.000
C39-C38-C40	58.70(15)

C24-C23-H23	120.400
C22-C23-C24	119.1(2)
C22-C23-H23	120.400
C15-C14-H14	120.200
C13-C14-C15	119.7(2)
C13-C14-H14	120.200
C11-C10-H10	119.900
C9-C10-C11	120.3(2)
C9-C10-H10	119.900
C27-C28-H28	119.200
C27-C28-C29	121.5(2)
C29-C28-H28	119.200
C31-C36-H36	119.400
C35-C36-C31	121.2(2)
C35-C36-H36	119.400
C23-C22-H22	120.700
C23-C22-C21	118.6(2)
C21-C22-H22	120.700
C1-C2-H2	120.700
C1-C2-C3	118.5(2)
C3-C2-H2	120.700
C31-C30-H30	119.700
C29-C30-C31	120.6(2)
C29-C30-H30	119.700
C17-C18-H18	117.400
C17-C18-C20	115.9(2)
C17-C18-C19	117.5(2)
C20-C18-H18	117.400
C19-C18-H18	117.400
C19-C18-C20	58.07(18)
N3-C21-C22	123.6(2)
N3-C21-H21	118.200
C22-C21-H21	118.200
C28-C29-H29	119.900
C30-C29-C28	120.3(2)
C30-C29-H29	119.900
C8-C9-H9	119.600
C10-C9-C8	120.7(2)
C10-C9-H9	119.600
C38-C40-H40A	117.700
C38-C40-H40B	117.700
H40A-C40-H40B	114.800

C39-C40-C38	60.45(15)
C39-C40-H40A	117.700
C39-C40-H40B	117.700
C38-C39-H39A	117.700
C38-C39-H39B	117.700
C40-C39-C38	60.84(15)
C40-C39-H39A	117.700
C40-C39-H39B	117.700
H39A-C39-H39B	114.800
C2-C3-H3	120.800
C4-C3-C2	118.4(2)
C4-C3-H3	120.800
N2-C5-H5	118.300
N2-C5-C4	123.5(2)
C4-C5-H5	118.300
C3-C4-C5	118.8(2)
C3-C4-H4	120.600
C5-C4-H4	120.600
C18-C20-H20A	117.700
C18-C20-H20B	117.700
H20A-C20-H20B	114.800
C19-C20-C18	60.52(17)
C19-C20-H20A	117.700
C19-C20-H20B	117.700
C18-C19-H19A	117.600
C18-C19-H19B	117.600
C20-C19-C18	61.41(18)
C20-C19-H19A	117.600
C20-C19-H19B	117.600
H19A-C19-H19B	114.700

Table S6. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3aa-8**

Atom	U11	U22	U33	U12	U13	U23
O2	0.0248(8)	0.0250(8)	0.0319(8)	-0.0072(7)	-0.0052(7)	-0.0025(7)
O5	0.0278(9)	0.0282(8)	0.0264(8)	-0.0085(7)	-0.0029(7)	-0.0074(7)
O4	0.0491(11)	0.0242(9)	0.0324(9)	-0.0062(8)	-0.0139(8)	-0.0047(7)
O3	0.0481(11)	0.0242(9)	0.0395(10)	-0.0064(8)	-0.0016(8)	-0.0070(7)
O6	0.0536(11)	0.0415(10)	0.0433(10)	-0.0287(9)	0.0061(9)	-0.0150(8)
O1	0.0767(14)	0.0540(11)	0.0395(10)	-0.0414(11)	-0.0229(10)	0.0002(9)
N1	0.0300(11)	0.0279(10)	0.029(1)	-0.0109(8)	-0.0087(8)	-0.0040(8)
N4	0.0317(11)	0.0252(10)	0.0249(10)	-0.0053(8)	-0.0085(8)	-0.0062(8)
N3	0.0319(11)	0.0265(10)	0.0295(10)	-0.0063(9)	-0.0070(9)	-0.0056(8)

N2	0.0309(11)	0.0386(11)	0.0306(11)	-0.0122(9)	-0.0040(9)	-0.0047(9)
C33	0.0283(12)	0.0272(12)	0.0245(11)	-0.0121(10)	-0.0018(10)	-0.0074(10)
C26	0.0263(12)	0.0281(12)	0.0253(11)	-0.0107(10)	-0.0074(10)	-0.0039(10)
C25	0.0233(12)	0.0259(11)	0.0268(11)	-0.0101(9)	-0.0055(9)	-0.0061(9)
C32	0.0262(12)	0.0240(11)	0.0246(11)	-0.0114(10)	-0.0021(9)	-0.0057(9)
C11	0.0320(13)	0.0196(11)	0.0314(12)	-0.0052(10)	-0.0049(10)	-0.0047(10)
C12	0.0292(12)	0.0192(11)	0.0281(12)	-0.0033(9)	-0.0055(10)	-0.0066(9)
C7	0.0281(13)	0.0199(11)	0.0295(12)	-0.0061(9)	-0.0037(10)	-0.0058(9)
C17	0.0285(13)	0.0308(13)	0.0283(12)	-0.008(1)	-0.0096(10)	-0.0061(10)
C1	0.0292(13)	0.0268(12)	0.0274(12)	-0.0046(10)	-0.0059(10)	-0.0079(10)
C37	0.0331(13)	0.0252(12)	0.0284(12)	-0.0092(10)	-0.0076(11)	-0.0003(10)
C27	0.0285(13)	0.0275(12)	0.0257(11)	-0.0102(10)	-0.0053(10)	-0.0077(10)
C15	0.0299(13)	0.0272(12)	0.0323(13)	-0.0036(10)	-0.0095(11)	-0.0093(10)
C31	0.0307(13)	0.0298(12)	0.0271(12)	-0.0138(10)	-0.0038(10)	-0.0078(10)
C16	0.0277(12)	0.0205(11)	0.0270(11)	-0.0057(9)	-0.0031(10)	-0.0033(9)
C8	0.0367(14)	0.0296(12)	0.0349(13)	-0.0112(11)	-0.0108(11)	-0.007(1)
C24	0.0304(13)	0.0299(12)	0.0268(12)	-0.0084(10)	-0.0068(10)	-0.0047(10)
C13	0.0356(14)	0.0267(12)	0.0264(12)	-0.0038(10)	-0.0035(10)	-0.0037(10)
C6	0.0371(14)	0.0269(12)	0.0308(12)	-0.0085(11)	-0.0094(11)	-0.0072(10)
C34	0.0335(13)	0.0302(12)	0.0302(12)	-0.009(1)	-0.0104(11)	-0.0031(10)
C35	0.0411(15)	0.0357(13)	0.0269(12)	-0.0148(12)	-0.0121(11)	-0.0033(11)
C38	0.0279(13)	0.0287(12)	0.0293(12)	-0.0074(10)	-0.0044(10)	-0.0076(10)
C23	0.0354(14)	0.0398(14)	0.0250(12)	-0.0114(11)	-0.0086(11)	-0.0064(11)
C14	0.0383(14)	0.0316(13)	0.0263(12)	-0.0033(11)	-0.0094(11)	-0.0042(10)
C10	0.0373(14)	0.0258(12)	0.0354(13)	-0.0121(11)	-0.0051(11)	-0.0018(10)
C28	0.0314(13)	0.0358(13)	0.0313(13)	-0.0061(11)	-0.0105(11)	-0.0074(11)
C36	0.0376(14)	0.0330(13)	0.0261(12)	-0.0150(11)	-0.0047(11)	-0.0093(10)
C22	0.0354(14)	0.0348(13)	0.0312(13)	-0.0083(11)	-0.0048(11)	-0.0135(11)
C2	0.0403(15)	0.0371(14)	0.0318(13)	-0.0101(11)	-0.0119(11)	-0.0076(11)
C30	0.0350(14)	0.0343(13)	0.0297(12)	-0.0084(11)	-0.0025(11)	-0.0142(11)
C18	0.0259(13)	0.0283(12)	0.0437(14)	-0.0079(10)	-0.0057(11)	-0.0067(11)
C21	0.0382(14)	0.0289(12)	0.0346(13)	-0.0042(11)	-0.0082(11)	-0.0111(11)
C29	0.0342(14)	0.0348(13)	0.0357(13)	-0.0016(11)	-0.0073(11)	-0.0123(11)
C9	0.0339(14)	0.0318(13)	0.0452(15)	-0.0158(11)	-0.0062(12)	-0.0060(11)
C40	0.0297(14)	0.0394(14)	0.0461(15)	-0.0089(11)	-0.0045(12)	-0.0155(12)
C39	0.0438(16)	0.0402(15)	0.0324(13)	-0.0110(12)	0.0013(12)	-0.0083(12)
C3	0.0535(17)	0.0423(15)	0.0308(13)	-0.0122(13)	-0.0135(12)	0.0011(12)
C5	0.0414(16)	0.0516(16)	0.0362(14)	-0.0240(13)	-0.0056(12)	-0.0017(13)
C4	0.0495(17)	0.0447(15)	0.0349(14)	-0.0212(14)	-0.0038(13)	0.0035(12)
C20	0.0289(15)	0.0395(15)	0.093(2)	-0.0095(12)	-0.0150(15)	-0.0102(15)
C19	0.0538(18)	0.0462(16)	0.0552(18)	-0.0239(14)	0.0101(15)	-0.0124(14)

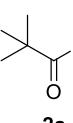
6. Characterization data of silver carboxylates **2a-1–2d-16**

 **2a-1** Silver(I) acetate (**2a-1**). Following the general procedure, **2a-1** was obtained as a gray solid (752 mg, 90%). ^1H NMR (400 MHz, Pyridine- d_5) δ 2.45 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, Pyridine- d_5) δ 176.4, 24.6.

 **2a-2** Silver(I) propionate (**2a-2**). Following the general procedure, **2a-2** was obtained as a gray solid (819 mg, 91%). ^1H NMR (400 MHz, DMSO- d_6) δ 2.12 (q, $J = 7.6$ Hz, 2H), 1.01 (t, $J = 7.6$ Hz, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 178.2, 30.2, 11.7.

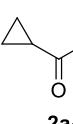
 **2a-3** Silver(I) butyrate (**2a-3**). Following the general procedure, **2a-3** was obtained as a gray solid (873 mg, 90%). ^1H NMR (400 MHz, DMSO- d_6) δ 2.10 (t, $J = 7.4$ Hz, 2H), 1.58-1.46 (m, 2H), 0.86 (t, $J = 7.4$ Hz, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 177.4, 39.3, 20.2, 14.6.

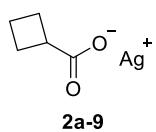
 **2a-4** Silver(I) isobutyrate (**2a-4**). Following the general procedure, **2a-4** was obtained as a gray solid (922 mg, 95%). ^1H NMR (400 MHz, DMSO- d_6) δ 2.39 (sep, $J = 6.8$ Hz, 1H), 1.05 (d, $J = 6.8$ Hz, 6H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 180.9, 36.2, 21.2.

 **2a-5** Silver(I) pivalate (**2a-5**). Following the general procedure, **2a-5** was obtained as a gray solid (1009 mg, 97%). ^1H NMR (400 MHz, DMSO- d_6) δ 1.10 (s, 9H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 182.3, 39.6, 29.4.

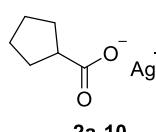
 **2a-6** Silver(I) nonanoate (**2a-6**). Following the general procedure, **2a-6** was obtained as a white solid (2124 mg, 80%). ^1H NMR (400 MHz, Pyridine- d_5) δ 2.82 (t, $J = 7.6$ Hz, 2H), 2.12-2.04 (m, 2H), 1.57-1.47 (m, 2H), 1.35-1.27 (m, 2H), 1.25-1.11 (m, 6H), 0.79 (t, $J = 6.8$ Hz, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, Pyridine- d_5) δ 179.0, 38.5, 32.0, 30.4, 30.0, 29.6, 28.0, 22.8, 14.2.

 **2a-7** Silver(I) adamantane-1-carboxylate (**2a-7**). Following the general procedure, **2a-7** was obtained as a gray solid (1148 mg, 80%). ^1H NMR (400 MHz, Pyridine- d_5) δ 2.49-2.43 (m, 6H), 2.03 (s, 3H), 1.81 (s, 1H), 1.78 (s, 2H), 1.73 (s, 2H), 1.70 (s, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, Pyridine- d_5) δ 183.2, 42.1, 41.6, 37.5, 39.3.

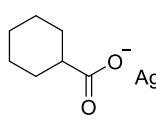
 **2a-8** Silver(I) cyclopropanecarboxylate (**2a-8**). Following the general procedure, **2a-8** was obtained as a gray solid (682 mg, 71%). ^1H NMR (400 MHz, DMSO- d_6) δ 1.51-1.38 (m, 1H), 0.69-0.54 (m, 4H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 178.5, 15.6, 7.3.



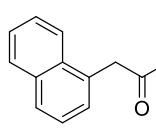
Silver(I) cyclobutanecarboxylate (**2a-9**). Following the general procedure, **2a-9** was obtained as a gray solid (618 mg, 60%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 3.03-2.91 (m, 1H), 2.16-2.00 (m, 4H), 1.88-1.68 (m, 2H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 179.4, 40.8, 26.7, 18.2.



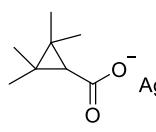
Silver(I) cyclopentanecarboxylate (**2a-10**). Following the general procedure, **2a-10** was obtained as a gray solid (880 mg, 80%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 2.63-2.53 (m, 1H), 1.80-1.36 (m, 8H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 180.3, 46.5, 31.2, 25.9.



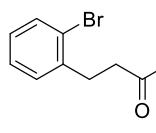
Silver(I) cyclohexanecarboxylate (**2a-11**). Following the general procedure, **2a-11** was obtained as a gray solid (995 mg, 85%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 2.14-2.04 (m, 1H), 1.78-1.74 (m, 2H), 1.70-1.60 (m, 2H), 1.59-1.52 (m, 1H), 1.35-1.13 (m, 5H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 179.8, 45.5, 30.9, 26.4, 26.1.



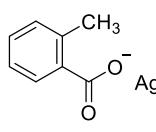
Silver(I) 2-(naphthalen-1-yl)acetate (**2a-12**). Following the general procedure, **2a-12** was obtained as a gray solid (1241 mg, 85%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.12-7.99 (m, 1H), 7.94-7.83 (m, 1H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.51-7.43 (m, 2H), 7.43-7.31 (m, 2H), 3.91 (s, 2H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 175.4, 135.3, 133.8, 132.7, 128.6, 127.9, 126.8, 126.1, 125.9, 125.8, 125.2, 42.3.



Silver(I) 2,2,3,3-tetramethylcyclopropane-1-carboxylate (**2a-13**). Following the general procedure, **2a-13** was obtained as a gray solid (930 mg, 75%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 1.19 (s, 6H), 1.13 (s, 1H), 1.08 (s, 6H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 176.7, 26.3, 24.4, 17.9.

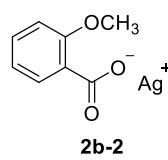


Silver(I) 3-(2-bromophenyl)propanoate (**2a-14**). Following the general procedure, **2a-14** was obtained as a gray solid (1598 mg, 80%). ¹H NMR (400 MHz, Pyridine-*d*₅) δ 7.57-7.49 (m, 2H), 7.14 (t, *J* = 7.2 Hz, 1H), 6.99 (t, *J* = 7.6 Hz, 1H), 3.54 (t, *J* = 8.0 Hz, 2H), 3.11 (t, *J* = 8.0 Hz, 2H). ¹³C{¹H} NMR (100 MHz, Pyridine-*d*₅) δ 176.3, 141.6, 131.3, 129.5, 126.4, 126.2, 123.4, 36.8, 33.0.

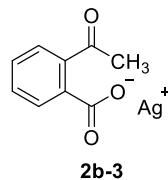


Silver(I) 2-methylbenzoate (**2b-1**). Following the general procedure, **2b-1** was obtained as a gray solid (1150 mg, 95%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.65 (d, *J* = 8.0 Hz, 1H), 7.24-7.19 (m, 1H), 7.18-7.08 (m, 2H), 2.49 (s, 3H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 173.2, 138.8,

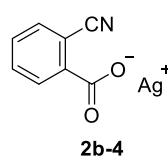
136.9, 130.9, 129.7, 129.0, 125.5, 21.8.



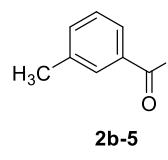
Silver(I) 2-methoxybenzoate (**2b-2**). Following the general procedure, **2b-2** was obtained as a gray solid (1226 mg, 95%). ^1H NMR (400 MHz, DMSO- d_6) δ 7.43 (dd, J = 7.6, 2.0 Hz, 1H), 7.31-7.25 (m, 1H), 6.98 (dd, J = 8.4, 1.2 Hz, 1H), 6.89 (dt, J = 7.6, 1.2 Hz, 1H), 3.73 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 171.8, 156.7, 129.9, 129.7, 129.4, 120.2, 112.2, 55.8.



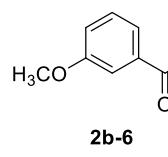
Silver(I) 2-acetylbenzoate (**2b-3**). Following the general procedure, **2b-3** was obtained as a gray solid (1283 mg, 95%). ^1H NMR (400 MHz, DMSO- d_6) δ 8.03 (d, J = 8.4 Hz, 2H), 7.95 (d, J = 8.4 Hz, 2H), 2.59 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 198.3, 169.7, 141.8, 138.2, 130.0, 128.2, 27.4.



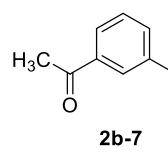
Silver(I) 2-cyanobenzoate (**2b-4**). Following the general procedure, **2b-4** was obtained as a gray solid (1201 mg, 95%). ^1H NMR (400 MHz, DMSO- d_6) δ 8.02 (dd, J = 7.6, 1.2 Hz, 1H), 7.77 (dd, J = 7.6, 1.2 Hz, 1H), 7.66 (dt, J = 7.6, 1.2 Hz, 1H), 7.55 (dd, J = 7.6, 1.2 Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 168.2, 141.5, 134.3, 132.7, 131.0, 130.4, 119.7, 112.1.



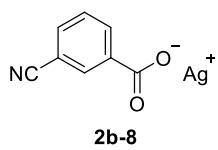
Silver(I) 3-methylbenzoate (**2b-5**). Following the general procedure, **2b-5** was obtained as a gray solid (1138 mg, 94%). ^1H NMR (400 MHz, DMSO- d_6) δ 7.78 (s, 1H), 7.75 (d, J = 6.4 Hz, 1H), 7.29-7.21 (m, 2H), 2.34 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 170.8, 137.21, 137.19, 131.3, 130.7, 128.1, 127.2, 21.5.



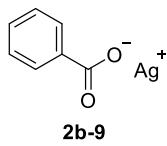
Silver(I) 3-methoxybenzoate (**2b-6**). Following the general procedure, **2b-6** was obtained as a gray solid (1226 mg, 95%). ^1H NMR (400 MHz, DMSO- d_6) δ 7.60-7.40 (m, 2H), 7.32-7.23 (m, 1H), 7.05-6.92 (m, 1H), 3.77 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 170.4, 159.3, 136.2, 129.3, 122.4, 116.6, 114.9, 55.5.



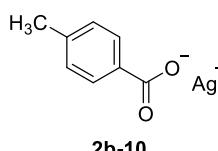
Silver(I) 3-acetylbenzoate (**2b-7**). Following the general procedure, **2b-7** was obtained as a gray solid (1242 mg, 92%). ^1H NMR (400 MHz, DMSO- d_6) δ 8.51 (s, 1H), 8.18 (d, J = 7.6 Hz, 1H), 8.02 (d, J = 7.6 Hz, 1H), 7.54 (t, J = 7.6 Hz, 1H), 2.60 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 198.3, 169.7, 137.9, 137.0, 134.4, 130.3, 129.5, 128.7, 27.3.



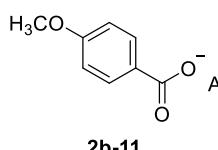
Silver(I) 3-cyanobenzoate (**2b-8**). Following the general procedure, **2b-8** was obtained as a gray solid (1213 mg, 96%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.24-8.18 (m, 2H), 7.90-7.85 (m, 1H), 7.60 (t, *J* = 8.0 Hz, 1H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 168.5, 139.0, 134.4, 133.9, 133.2, 129.7, 129.3, 111.3.



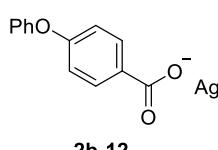
Silver(I) benzoate (**2b-9**). Following the general procedure, **2b-9** was obtained as a gray solid (1053 mg, 93%). ¹H NMR (400 MHz, Pyridine-*d*₅) δ 8.85 (d, *J* = 8.0 Hz, 2H), 7.49-7.38 (m, 3H). ¹³C{¹H} NMR (100 MHz, Pyridine-*d*₅) δ 171.8, 140.2, 130.6, 129.5, 127.8.



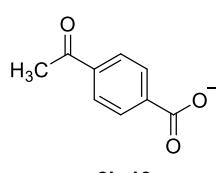
Silver(I) 4-methylbenzoate (**2b-10**). Following the general procedure, **2b-10** was obtained as a gray solid (1150 mg, 95%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.85 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 2.33 (s, 3H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 170.7, 140.3, 134.5, 130.1, 128.8, 21.5.



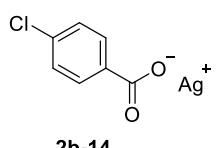
Silver(I) 4-methoxybenzoate (**2b-11**). Following the general procedure, **2b-11** was obtained as a gray solid (1225 mg, 95%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.89 (d, *J* = 9.0 Hz, 2H), 6.89 (d, *J* = 9.0 Hz, 2H), 3.78 (s, 3H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 170.5, 161.4, 131.8, 129.7, 113.4, 55.6.



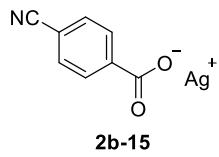
Silver(I) 4-phenoxybenzoate (**2b-12**). Following the general procedure, **2b-12** was obtained as a gray solid (1225 mg, 95%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.96 (d, *J* = 8.8 Hz, 2H), 7.42 (dd, *J* = 8.8, 7.2 Hz, 2H), 7.18 (t, *J* = 7.2 Hz, 1H), 7.07 (d, *J* = 7.2 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 170.0, 159.2, 156.4, 132.2, 132.1, 130.6, 124.4, 119.8, 117.4.



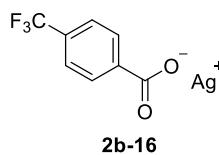
Silver(I) 4-acetylbenzoate (**2b-13**). Following the general procedure, **2b-13** was obtained as a gray solid (1245 mg, 92%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.04 (d, *J* = 8.2 Hz, 2H), 7.98 (d, *J* = 8.2 Hz, 2H), 2.60 (s, 3H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 198.3, 168.8, 139.6, 138.9, 130.0, 128.4, 27.4.



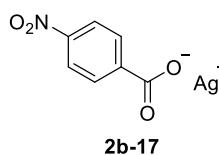
Silver(I) 4-chlorobenzoate (**2b-14**). Following the general procedure, **2b-14** was obtained as a gray solid (1179 mg, 90%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.95 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 2H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 169.1, 136.0, 135.1, 131.8, 128.5.



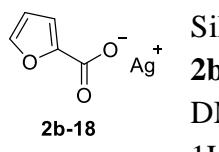
Silver(I) 4-cyanobenzoate (**2b-15**). Following the general procedure, **2b-15** was obtained as a gray solid (1175 mg, 93%). ^1H NMR (400 MHz, DMSO- d_6) δ 8.06 (d, $J = 8.0$ Hz, 2H), 7.84 (d, $J = 8.0$ Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 169.0, 142.0, 132.4, 130.5, 119.3, 112.8.



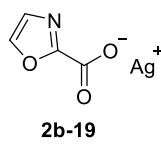
Silver(I) 4-(trifluoromethyl)benzoate (**2b-16**). Following the general procedure, **2b-16** was obtained as a gray solid (1406 mg, 95%). ^1H NMR (400 MHz, DMSO- d_6) δ 8.13 (d, $J = 8.0$ Hz, 2H), 7.73 (d, $J = 8.0$ Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 169.3, 141.4, 130.7 ($q, J_{\text{C}-\text{F}} = 31.3$ Hz), 130.6, 125.2 ($q, J_{\text{C}-\text{F}} = 14.0$ Hz), 124.7 ($q, J_{\text{C}-\text{F}} = 270.6$ Hz). ^{19}F NMR (376 MHz, DMSO- d_6) δ -61.1.



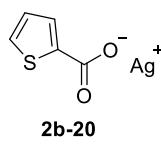
Silver(I) 4-nitrobenzoate (**2b-17**). Following the general procedure, **2b-17** was obtained as a gray solid (1256 mg, 92%). ^1H NMR (400 MHz, DMSO- d_6) δ 8.21 (d, $J = 8.8$ Hz, 2H), 8.12 (d, $J = 8.8$ Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 168.6, 148.8, 144.2, 130.9, 123.4.



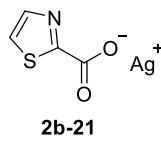
Silver(I) furan-2-carboxylate (**2b-18**). Following the general procedure, **2b-18** was obtained as a gray solid (872 mg, 80%). ^1H NMR (400 MHz, DMSO- d_6) δ 7.65 (dd, $J = 1.6, 0.8$ Hz, 1H), 6.81 (dd, $J = 3.2, 0.8$ Hz, 1H), 6.48 (dd, $J = 3.2, 1.6$ Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 163.2, 151.7, 144.2, 113.8, 111.6.



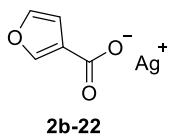
Silver(I) oxazole-2-carboxylate (**2b-19**). Following the general procedure, **2b-19** was obtained as a gray solid (876 mg, 80%). ^1H NMR (400 MHz, Pyridine- d_5) δ 7.97 (s, 1H), 7.24 (s, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, Pyridine- d_5) δ 162.9, 159.6, 139.9, 127.5.



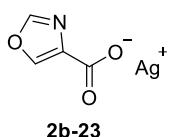
Silver(I) thiophene-2-carboxylate (**2b-20**). Following the general procedure, **2b-20** was obtained as a gray solid (936 mg, 80%). ^1H NMR (400 MHz, DMSO- d_6) δ 7.58 (dd, $J = 4.8, 1.2$ Hz, 1H), 7.47 (dd, $J = 3.6, 1.2$ Hz, 1H), 7.05 (dd, $J = 4.8, 3.6$ Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 166.2, 143.0, 130.6, 130.3, 127.9.



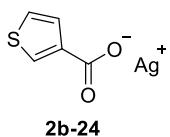
Silver(I) thiazole-2-carboxylate (**2b-21**). Following the general procedure, **2b-21** was obtained as a gray solid (940 mg, 80%). ^1H NMR (400 MHz, DMSO- d_6) δ 7.91 (d, $J = 3.2$ Hz, 1H), 7.88 (d, $J = 3.2$ Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 172.4, 160.8, 143.0, 125.5.



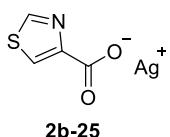
Silver(I) furan-3-carboxylate (**2b-22**). Following the general procedure, **2b-22** was obtained as a gray solid (894 mg, 83%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.94 (dd, *J* = 1.6, 0.8 Hz, 1H), 7.60 (t, *J* = 1.6 Hz, 1H), 6.60 (dd, *J* = 1.6, 0.8 Hz, 1H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 167.3, 146.1, 143.6, 125.5, 111.7.



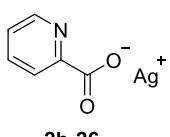
Silver(I) oxazole-4-carboxylate (**2b-23**). Following the general procedure, **2b-23** was obtained as a gray solid (876 mg, 80%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.52 (d, *J* = 0.8 Hz, 1H), 8.45 (d, *J* = 0.8 Hz, 1H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 163.1, 152.7, 143.5, 136.3.



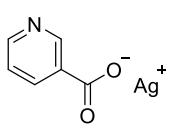
Silver(I) thiophene-3-carboxylate (**2b-24**). Following the general procedure, **2b-24** was obtained as a gray solid (937 mg, 80%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.91 (dd, *J* = 3.2, 0.8 Hz, 1H), 7.44 (dd, *J* = 4.8, 3.2 Hz, 1H), 7.37 (dd, *J* = 4.8, 0.8 Hz, 1H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 167.3, 141.3, 129.9, 129.5, 125.8.



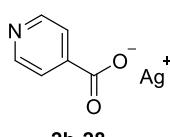
Silver(I) thiazole-4-carboxylate (**2b-25**). Following the general procedure, **2b-25** was obtained as a gray solid (942 mg, 80%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.20 (d, *J* = 2.0 Hz, 1H), 8.39 (d, *J* = 2.0 Hz, 1H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 163.6, 156.7, 150.8, 127.0.



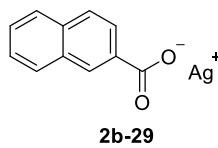
Silver(I) picolinate (**2b-26**). Following the general procedure, **2b-26** was obtained as a gray solid (1031 mg, 91%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.69 (d, *J* = 4.4 Hz, 1H), 8.06 (d, *J* = 7.6 Hz, 1H), 7.99 (dt, *J* = 7.6, 1.6 Hz, 1H), 7.66-7.59 (m, 1H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 166.6, 150.0, 149.3, 138.1, 127.4, 125.1.



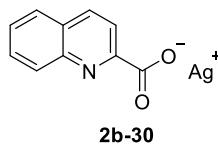
Silver(I) nicotinate (**2b-27**). Following the general procedure, **2b-27** was obtained as a gray solid (912 mg, 80%). ¹H NMR (400 MHz, Pyridine-*d*₅) δ 10.10 (d, *J* = 2.0 Hz, 1H), 8.91 (dt, *J* = 7.6, 2.0 Hz, 1H), 8.72 (dd, *J* = 4.8, 2.0 Hz, 1H), 7.27 (dd, *J* = 7.6, 4.8 Hz, 1H). ¹³C{¹H} NMR (100 MHz, Pyridine-*d*₅) δ 170.1, 152.4, 150.4, 137.5, 123.0.



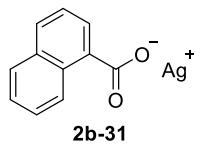
Silver(I) isonicotinate (**2b-28**). Following the general procedure, **2b-28** was obtained as a gray solid (1005 mg, 88%). ¹H NMR (400 MHz, Pyridine-*d*₅) δ 8.85 (dt, *J* = 4.0, 2.0 Hz, 2H), 8.52 (dt, *J* = 4.0, 2.0 Hz, 2H). ¹³C{¹H} NMR (100 MHz, Pyridine-*d*₅) δ 169.9, 150.2, 147.8, 124.7.



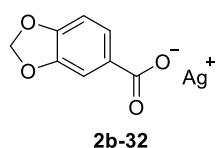
Silver(I) 2-naphthoate (**2b-29**). Following the general procedure, **2b-29** was obtained as a gray solid (1181 mg, 85%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.55 (s, 1H), 8.07 (dd, *J* = 8.4, 1.4 Hz, 1H), 8.05-8.00 (m, 1H), 7.96-7.92 (m, 1H), 7.90 (d, *J* = 8.8 Hz, 1H), 7.60-7.50 (m, 2H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 170.5, 134.5, 134.3, 132.9, 130.0, 129.4, 127.9, 127.6, 127.5, 127.3, 126.6.



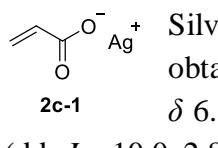
Silver(I) quinoline-2-carboxylate (**2b-30**). Following the general procedure, **2b-30** was obtained as a gray solid (1173 mg, 84%). ¹H NMR (400 MHz, Pyridine-*d*₅) δ 9.03 (d, *J* = 8.8 Hz, 1H), 8.64 (d, *J* = 8.4 Hz, 1H), 8.32 (d, *J* = 8.4 Hz, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.70-7.64 (m, 1H), 7.53-7.47 (m, 1H). ¹³C{¹H} NMR (100 MHz, Pyridine-*d*₅) δ 168.2, 156.6, 146.1, 137.8, 130.4, 130.3, 129.3, 128.1, 127.6, 123.0.



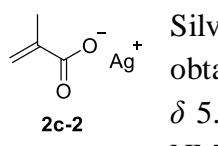
Silver(I) 1-naphthoate (**2b-31**). Following the general procedure, **2b-31** was obtained as a gray solid (1188 mg, 86%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.97-8.88 (m, 1H), 7.98-7.87 (m, 3H), 7.55-7.44 (m, 3H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 172.7, 136.9, 133.9, 131.2, 129.7, 128.4, 127.8, 127.7, 126.3, 125.9, 125.5.



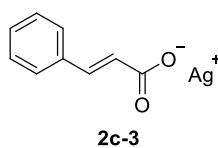
Silver(I) benzo[d][1,3]dioxole-5-carboxylate (**2b-32**). Following the general procedure, **2b-32** was obtained as a gray solid (1292 mg, 95%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.54 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.38 (d, *J* = 1.6 Hz, 1H), 6.89 (d, *J* = 8.0 Hz, 1H), 6.05 (s, 2H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 169.7, 149.6, 147.3, 131.1, 124.8, 109.9, 107.9, 101.7.



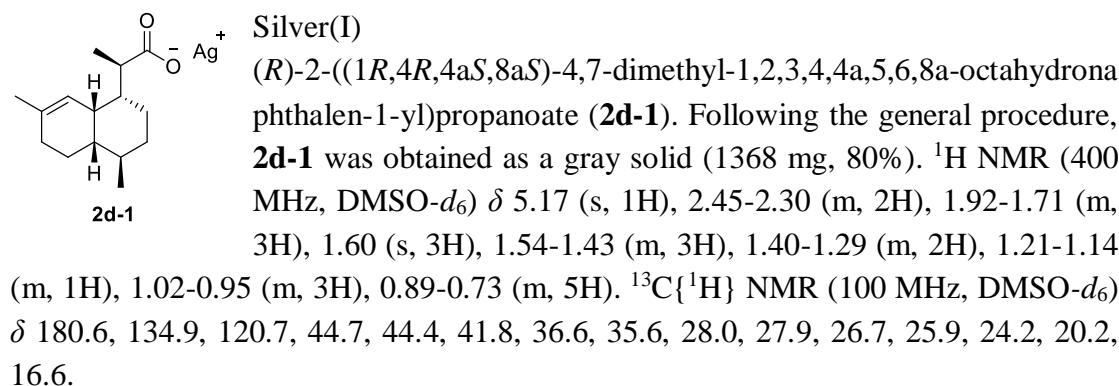
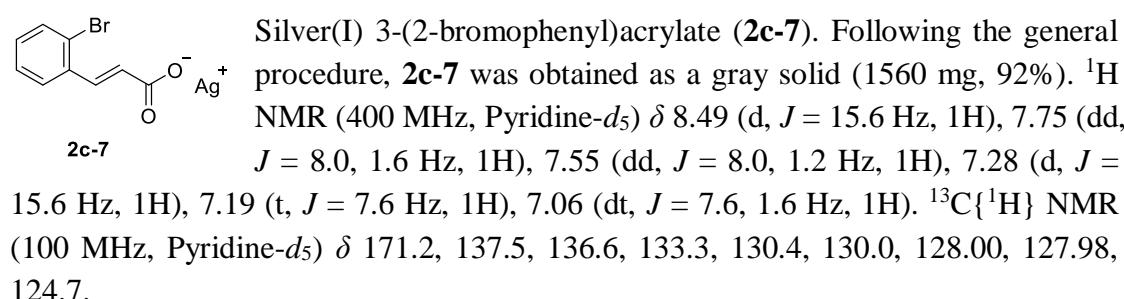
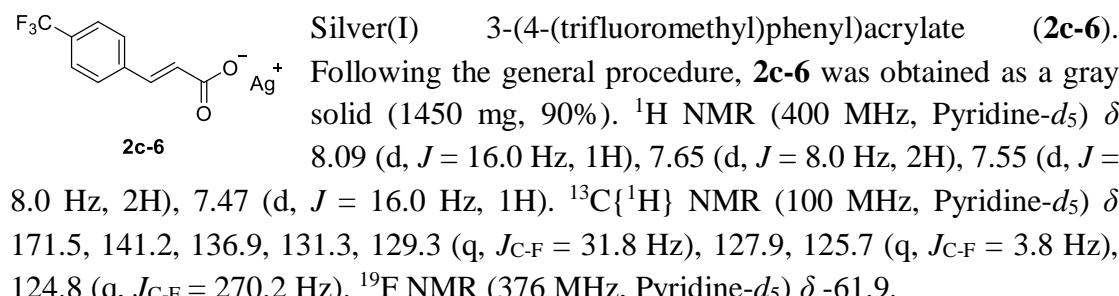
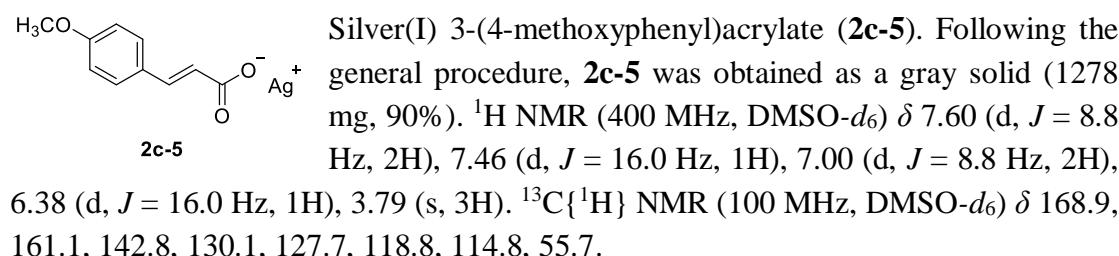
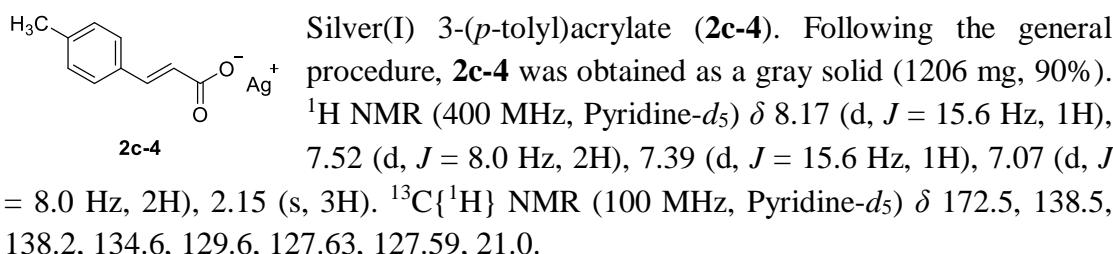
Silver(I) acrylate (**2c-1**). Following the general procedure, **2c-1** was obtained as a gray solid (712 mg, 80%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 6.15 (dd, *J* = 17.2, 10.0 Hz, 1H), 5.97 (dd, *J* = 17.2, 2.8 Hz, 1H), 5.52 (dd, *J* = 10.0, 2.8, 1H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 170.7, 135.7, 125.4.

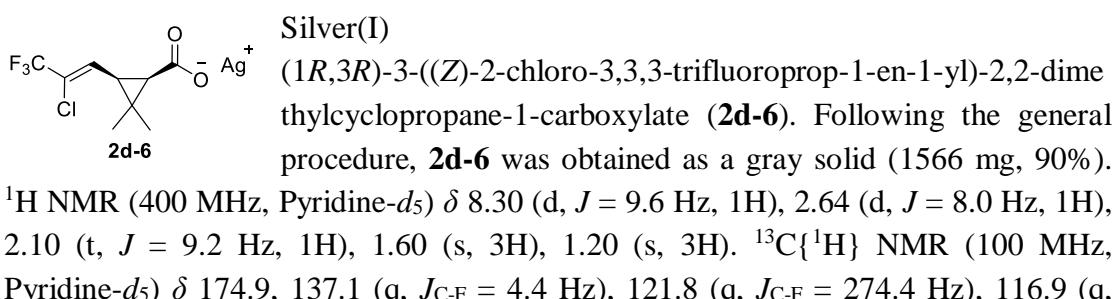
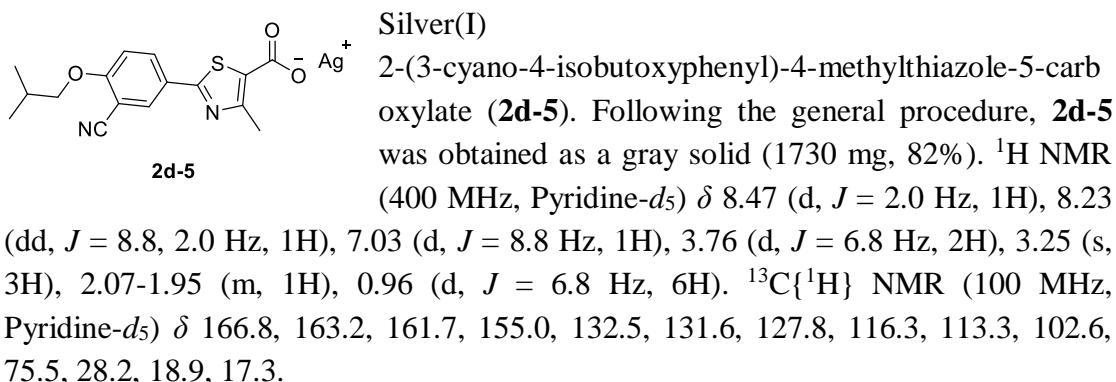
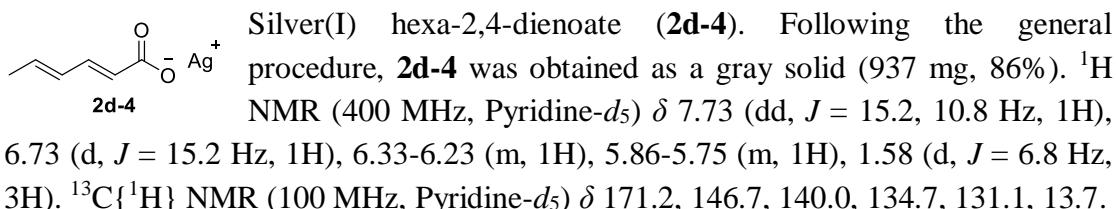
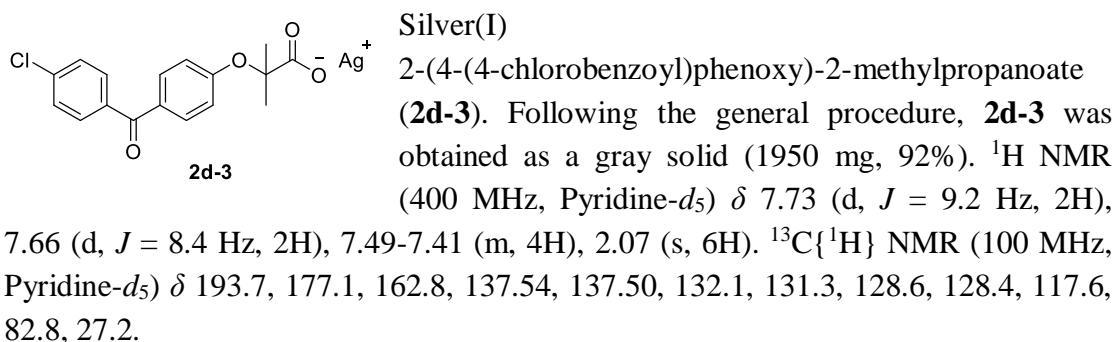
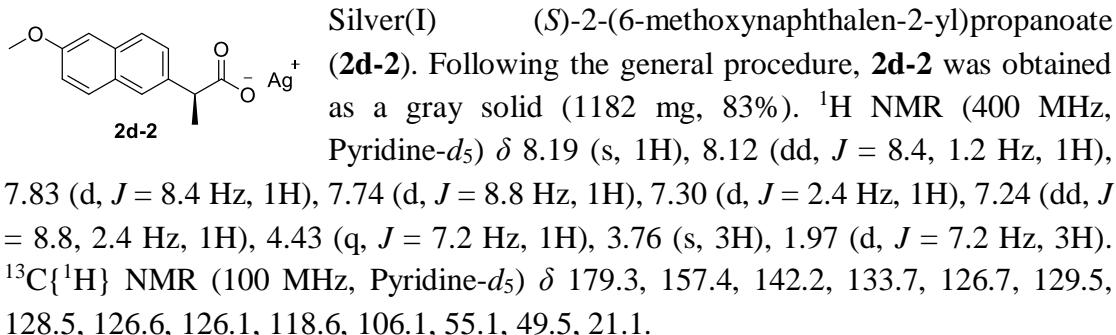


Silver(I) methacrylate (**2c-2**). Following the general procedure, **2c-2** was obtained as a gray solid (772 mg, 81%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 5.76 (dd, *J* = 7.2, 4.8, 1H), 5.26-5.22 (m, 1H), 1.86 (s, 3H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 172.0, 143.6, 120.6, 20.8.

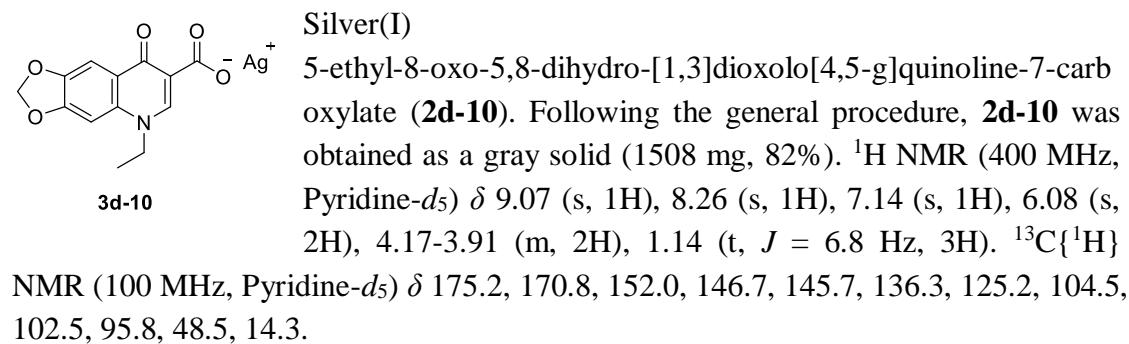
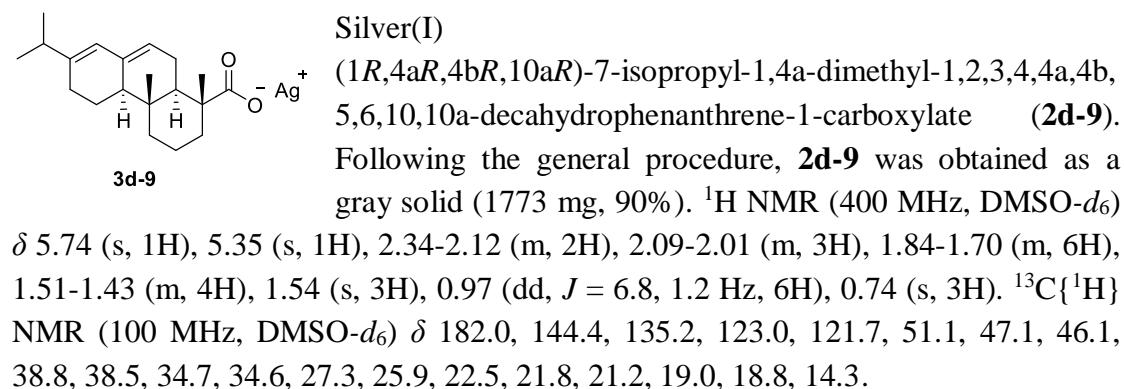
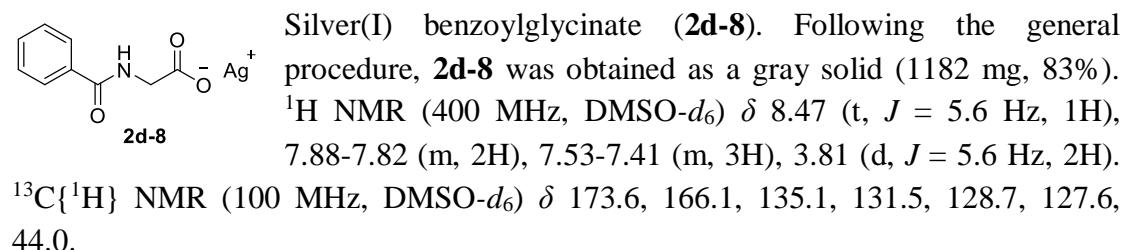
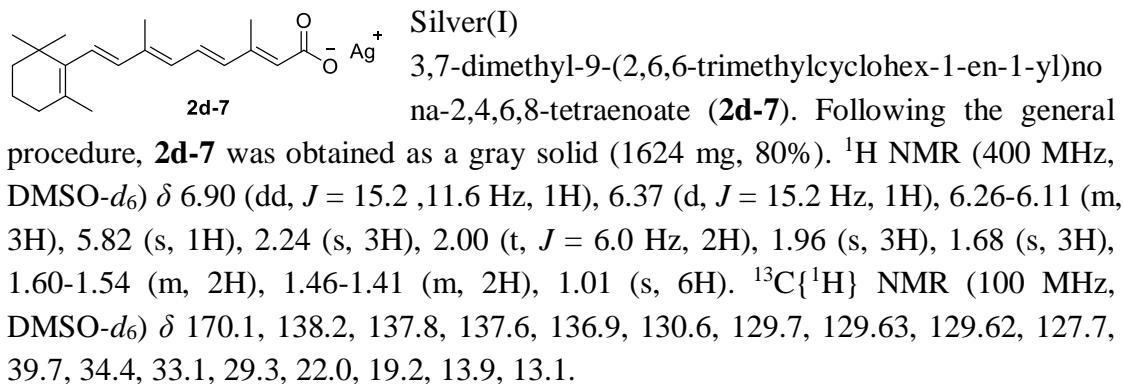


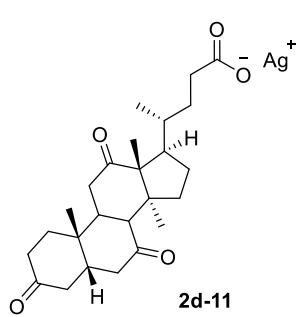
Silver(I) cinnamate (**2c-3**). Following the general procedure, **2c-3** was obtained as a gray solid (1143 mg, 90%). ¹H NMR (400 MHz, Pyridine-*d*₅) δ 8.17 (d, *J* = 16.0 Hz, 1H), 7.59 (d, *J* = 7.2 Hz, 2H), 7.43 (d, *J* = 16.0 Hz, 1H), 7.29-7.18 (m, 3H). ¹³C{¹H} NMR (100 MHz, Pyridine-*d*₅) δ 172.6, 138.1, 137.5, 129.3, 128.9, 128.3, 127.5.





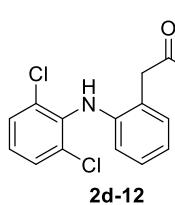
$J_{\text{C}-\text{F}} = 36.5$ Hz), 39.5, 30.3, 28.9, 27.3, 16.3. ^{19}F NMR (376 MHz, Pyridine-*d*₅) δ -67.2.





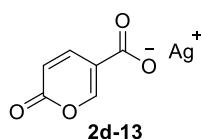
Silver(I)

(*R*)-4-((5*S*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-10,13-dimethyl-3,7,12-trioxohexadecahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl)pentanoate (**2d-11**). Following the general procedure, **2d-11** was obtained as a gray solid (2088 mg, 80%). ^1H NMR (400 MHz, DMSO-*d*₆) δ 3.07-2.94 (m, 2H), 2.83 (t, *J* = 12.6 Hz, 1H), 2.43 (dt, *J* = 12.4, 5.2 Hz, 1H), 2.34-1.92 (m, 9H), 1.87-1.63 (m, 6H), 1.49 (dt, *J* = 14.4, 4.0 Hz, 1H), 1.32 (s, 3H), 1.29-1.19 (m, 4H), 1.00 (s, 3H), 0.75 (d, *J* = 5.6 Hz, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO-*d*₆) δ 212.8, 210.4, 210.3, 178.2, 55.8, 51.8, 48.6, 46.6, 46.2, 45.1, 44.7, 43.1, 39.0, 36.7, 36.2, 35.9, 35.1, 34.5, 32.9, 27.9, 25.2, 21.7, 19.5, 12.1.



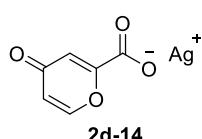
Silver(I)

2-((2,6-dichlorophenyl)amino)phenylacetate (**2d-12**). Following the general procedure, **2d-12** was obtained as a gray solid (1602 mg, 80%). ^1H NMR (400 MHz, DMSO-*d*₆) δ 8.98 (s, 1H), 7.47 (d, *J* = 8.0 Hz, 2H), 7.15-7.07 (m, 2H), 6.98 (dt, *J* = 8.0, 1.6 Hz, 1H), 6.79 (dt, *J* = 7.2, 1.6 Hz, 1H), 6.28 (dd, *J* = 8.0, 1.2 Hz, 1H), 3.56 (s, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO-*d*₆) δ 176.0, 143.3, 138.1, 130.7, 129.6, 129.4, 127.6, 126.8, 124.8, 120.9, 116.4, 42.6.



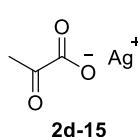
Silver(I)

2-oxo-2*H*-pyran-5-carboxylate (**2d-13**). Following the general procedure, **2d-13** was obtained as a gray solid (984 mg, 80%). ^1H NMR (400 MHz, DMSO-*d*₆) δ 8.26 (dd, *J* = 2.6, 1.0 Hz, 1H), 7.88 (dd, *J* = 9.6, 2.6 Hz, 1H), 6.28 (dd, *J* = 9.6, 1.0 Hz). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO-*d*₆) δ 166.4, 161.4, 156.7, 145.7, 117.4, 114.0.



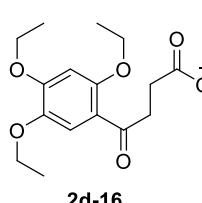
Silver(I)

4-oxo-4*H*-pyran-2-carboxylate (**2d-14**). Following the general procedure, **2d-14** was obtained as a gray solid (1082 mg, 88%). ^1H NMR (400 MHz, Pyridine-*d*₅) δ 8.04 (d, *J* = 5.6 Hz, 1H), 7.62 (d, *J* = 2.8 Hz, 1H), 6.45 (dd, *J* = 5.6, 2.8 Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, Pyridine-*d*₅) δ 179.9, 163.5, 161.0, 155.8, 117.7, 117.5.



Silver(I)

2-oxopropanoate (**2d-15**). Following the general procedure, **2d-15** was obtained as a gray solid (873 mg, 90%). ^1H NMR (400 MHz, DMSO-*d*₆) δ 2.20 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, DMSO-*d*₆) δ 203.0, 169.5, 27.8.

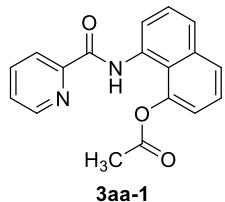


Silver(I)

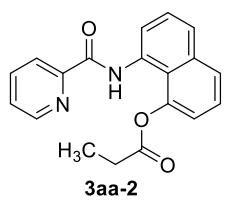
4-oxo-4-(2,4,5-triethoxyphenyl)butanoate (**2d-16**). Following the general procedure, **2d-16** was obtained as a gray solid (1893 mg, 91%). ^1H NMR (400 MHz, Pyridine-*d*₅) δ 7.80 (s, 1H), 6.65 (s, 1H), 4.06 (q, *J* = 6.8 Hz, 2H), 4.01 (q, *J* = 6.8

Hz, 2H), 3.92-3.85 (m, 4H), 3.37 (t, J = 6.8 Hz, 2H), 1.37 (t, J = 6.8 Hz, 3H), 1.32 (t, J = 6.8 Hz, 3H), 1.23 (t, J = 6.8 Hz, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, Pyridine-*d*₅) δ 200.5, 178.1, 154.8, 153.7, 142.9, 120.7, 115.8, 99.5, 65.0, 64.9, 64.5, 42.3, 33.1, 15.0, 14.83, 14.76.

7. Characterization data of products **3aa-1–3wa**



8-(Picolinamido)naphthalen-1-yl acetate (3aa-1**).** Following the general procedure, **3aa-1** was obtained as a colorless solid (56.4 mg, 92%). R_f = 0.40 (n-hexane/EtOAc 3:1). m.p. 160.0–162.0 °C. ^1H NMR (400 MHz, CDCl₃) δ 11.52 (s, 1H), 8.77 (d, J = 7.6 Hz, 1H), 8.68-8.62 (m, 1H), 8.38 (d, J = 8.0 Hz, 1H), 7.97-7.10 (m, 1H), 7.76 (d, J = 8.0 Hz, 1H), 7.69 (d, J = 8.0 Hz, 1H), 7.56-7.49 (m, 2H), 7.46 (t, J = 8.0 Hz, 1H), 7.20 (d, J = 7.6 Hz, 1H), 2.46 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl₃) δ 169.6, 162.3, 150.3, 147.6, 145.8, 137.8, 136.4, 132.4, 127.2, 126.6, 126.4, 125.2, 125.0, 122.9, 120.4, 119.5, 119.2, 21.6. HRMS (ESI): m/z [M+H]⁺ calcd for C₁₈H₁₅N₂O₃: 307.1083; found: 307.1077. FT-IR (neat, cm⁻¹) ν 3338, 2920, 1759, 1673, 1529, 1495, 1182, 724.

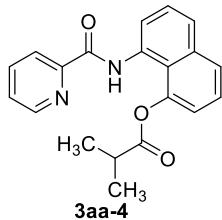


8-(Picolinamido)naphthalen-1-yl propionate (3aa-2**).** Following the general procedure, **3aa-2** was obtained as a colorless solid (49.9 mg, 78%). R_f = 0.48 (n-hexane/EtOAc 3:1). m.p. 136.0–138.0 °C. ^1H NMR (400 MHz, CDCl₃) δ 11.51 (s, 1H), 8.77 (d, J = 8.0 Hz, 1H), 8.64 (d, J = 4.4 Hz, 1H), 8.38 (d, J = 8.0 Hz, 1H), 7.95 (dd, J = 8.0, 1.6 Hz, 1H), 7.76 (d, J = 8.0 Hz, 1H), 7.68 (d, J = 8.0 Hz, 1H), 7.55-7.50 (m, 2H), 7.46 (t, J = 8.0 Hz, 1H), 7.18 (dd, J = 7.6, 1.2 Hz, 1H), 2.84 (q, J = 7.6 Hz, 2H), 1.19 (t, J = 7.6 Hz, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl₃) δ 173.2, 162.2, 150.3, 147.5, 126.0, 137.8, 136.4, 132.5, 127.1, 126.6, 126.4, 125.3, 125.0, 123.0, 120.3, 119.4, 119.3, 28.0, 8.9. HRMS (ESI): m/z [M+H]⁺ calcd for C₁₉H₁₇N₂O₃: 321.1239; found: 321.1234. FT-IR (neat, cm⁻¹) ν 3343, 2925, 1761, 1674, 1530, 1496, 1108, 748.

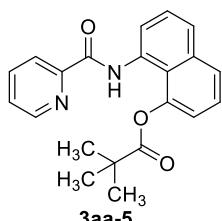


8-(Picolinamido)naphthalen-1-yl butyrate (3aa-3**).** Following the general procedure, **3aa-3** was obtained as a yellow solid (34.7 mg, 52%). R_f = 0.52 (n-hexane/EtOAc 3:1). m.p. 104.0–106.0 °C. ^1H NMR (400 MHz, CDCl₃) δ 11.50 (s, 1H), 8.76 (d, J = 7.6 Hz, 1H), 8.66 (d, J = 4.4 Hz, 1H), 8.38 (d, J = 8.0 Hz, 1H), 7.95 (t, J = 7.6 Hz, 1H), 7.75 (d, J = 7.6 Hz, 1H), 7.68 (d, J = 8.0 Hz, 1H), 7.56-7.50 (m, 2H), 7.46 (t, J = 7.6 Hz, 1H), 7.17 (dd, J = 7.6, 0.8 Hz, 1H), 2.77 (t, J = 7.6 Hz, 2H), 1.77-1.67 (m, 2H), 0.92 (t, J = 7.6 Hz, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl₃) δ 172.3, 162.2, 150.4, 147.6, 146.0, 137.8, 136.4, 132.5, 127.1, 126.6, 126.4, 125.3, 125.0, 123.0, 120.3,

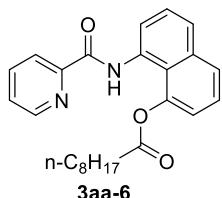
119.4, 119.3, 36.3, 18.2, 13.5. HRMS (ESI): m/z [M+H]⁺ calcd for C₂₀H₁₉N₂O₃: 335.1396; found: 335.1390. FT-IR (neat, cm⁻¹) ν 3338, 2925, 1759, 1677, 1529, 1496, 748.



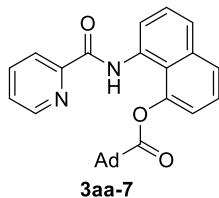
8-(Picolinamido)naphthalen-1-yl isobutyrate (3aa-4). Following the general procedure, **3aa-4** was obtained as a yellow solid (24.7 mg, 37%). R_f = 0.52 (n-hexane/EtOAc 3:1). m.p. 100.0–102.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 11.52 (s, 1H), 8.78 (d, J = 7.6 Hz, 1H), 8.63 (d, J = 4.8 Hz, 1H), 8.38 (d, J = 7.6 Hz, 1H), 7.95 (dt, J = 7.6, 1.6 Hz, 1H), 7.75 (d, J = 8.4 Hz, 1H), 7.68 (d, J = 8.0 Hz, 1H), 7.55–7.50 (m, 2H), 7.45 (t, J = 8.0 Hz, 1H), 7.13 (dd, J = 7.6, 0.8 Hz, 1H), 3.29–3.20 (m, 1H), 1.27 (d, J = 7.2 Hz, 6H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 176.1, 162.3, 150.4, 147.5, 146.3, 137.8, 136.4, 132.6, 127.0, 126.5, 126.4, 125.3, 125.0, 123.0, 120.1, 119.3, 33.9, 18.8. HRMS (ESI): m/z [M+H]⁺ calcd for C₂₀H₁₉N₂O₃: 335.1396; found: 335.1390. FT-IR (neat, cm⁻¹) ν 3346, 2923, 1763, 1684, 1530, 1497, 1075, 754.



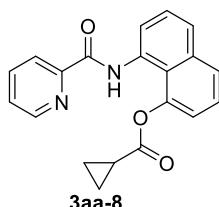
8-(Picolinamido)naphthalen-1-yl pivalate (3aa-5). Following the general procedure, **3aa-5** was obtained as a yellow oil (45.2 mg, 65%). R_f = 0.52 (n-hexane/EtOAc 3:1). ¹H NMR (400 MHz, CDCl₃) δ 10.72 (s, 1H), 8.66 (d, J = 4.0 Hz, 1H), 8.47 (d, J = 7.6 Hz, 1H), 8.35 (d, J = 8.0 Hz, 1H), 7.93 (dt, J = 7.6, 1.6 Hz, 1H), 7.76 (d, J = 7.6 Hz, 1H), 7.72 (d, J = 8.0 Hz, 1H), 7.55–7.48 (m, 2H), 7.45 (t, J = 7.6 Hz, 1H), 7.04 (dd, J = 7.6, 1.2 Hz, 1H), 1.26 (s, 9H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 178.1, 162.7, 150.3, 147.9, 147.3, 137.7, 136.4, 132.1, 127.0, 126.5, 126.3, 125.6, 125.4, 123.0, 121.0, 120.9, 120.0, 39.5, 26.9. HRMS (ESI): m/z [M+H]⁺ calcd for C₂₁H₂₁N₂O₃: 349.1552; found: 349.1547. FT-IR (neat, cm⁻¹) ν 3349, 2925, 1752, 1683, 1528, 1495, 1086, 752.



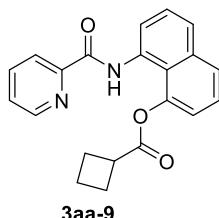
8-(Picolinamido)naphthalen-1-yl nonanoate (3aa-6). Following the general procedure, **3aa-6** was obtained as a colorless solid (38.8 mg, 48%). R_f = 0.75 (n-hexane/EtOAc 3:1). m.p. 94.0–96.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 11.50 (s, 1H), 8.76 (dd, J = 7.6, 0.8 Hz, 1H), 8.65 (d, J = 4.8 Hz, 1H), 8.38 (d, J = 8.0 Hz, 1H), 7.95 (dt, J = 7.6, 1.6 Hz, 1H), 7.76 (dd, J = 8.0, 1.2 Hz, 1H), 7.68 (dd, J = 8.0, 1.2 Hz, 1H), 7.56–7.50 (m, 2H), 7.46 (t, J = 8.0 Hz, 1H), 7.17 (dd, J = 7.6, 0.8 Hz, 1H), 2.77 (t, J = 7.6 Hz, 2H), 1.70–1.64 (m, 2H), 1.32–1.21 (m, 10H), 0.88 (t, J = 6.8 Hz, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 172.5, 162.2, 150.4, 147.6, 146.0, 137.8, 136.4, 132.5, 127.1, 126.5, 126.4, 125.3, 125.0, 123.0, 120.3, 119.4, 119.3, 34.5, 31.7, 29.2, 29.1, 24.7, 22.6, 14.1. HRMS (ESI): m/z [M+H]⁺ calcd for C₂₅H₂₉N₂O₃: 405.2178; found: 405.2173. FT-IR (neat, cm⁻¹) ν 3361, 2922, 1766, 1683, 1529, 1496, 1091, 750.



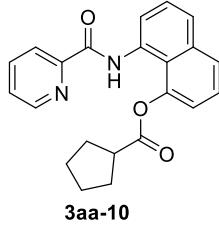
8-(picolinamido)naphthalen-1-yl adamantane-1-carboxylate (**3aa-7**). Following the general procedure, **3aa-7** was obtained as a yellow solid (50.3 mg, 59%). $R_f = 0.52$ (n-hexane/EtOAc 3:1). m.p. 204.0–206.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 10.53 (s, 1H), 8.69 (d, $J = 4.4$ Hz, 1H), 8.36 (d, $J = 7.6$ Hz, 2H), 7.93 (dt, $J = 7.6, 1.6$ Hz, 1H), 7.78–7.68 (m, 2H), 7.55–7.47 (m, 2H), 7.44 (t, $J = 8.0$ Hz, 1H), 7.00 (dd, $J = 7.6, 1.2$ Hz, 1H), 2.10–1.96 (m, 1H), 1.94–1.86 (m, 8H), 1.66–1.60 (m, 3H), 1.52–1.46 (m, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 177.1, 162.7, 150.5, 148.0, 147.3, 137.7, 136.4, 132.0, 126.9, 126.5, 126.2, 125.8, 125.5, 123.1, 121.4, 121.2, 120.2, 41.4, 38.3, 36.2, 27.7. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{27}\text{H}_{27}\text{N}_2\text{O}_3$: 427.2022; found: 427.2016. FT-IR (neat, cm^{-1}) ν 3365, 2914, 1746, 1684, 1527, 1496, 1198, 1032, 752.



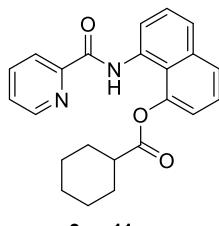
8-(Picolinamido)naphthalen-1-yl cyclopropanecarboxylate (**3aa-8**). Following the general procedure, **3aa-8** was obtained as a colorless solid (39.9 mg, 60%). $R_f = 0.46$ (n-hexane/EtOAc 3:1). m.p. 148.0–150.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.55 (s, 1H), 8.68 (dd, $J = 8.0, 1.2$ Hz, 1H), 8.60 (d, $J = 4.4$ Hz, 1H), 8.38 (d, $J = 8.0$ Hz, 1H), 7.94 (dt, $J = 7.6, 1.6$ Hz, 1H), 7.76 (dd, $J = 8.0, 1.2$ Hz, 1H), 7.69 (dd, $J = 8.0, 1.2$ Hz, 1H), 7.53 (t, $J = 8.0$ Hz, 1H), 7.51–7.47 (m, 1H), 7.45 (t, $J = 8.0$ Hz, 1H), 7.19 (dd, $J = 7.6, 1.2$ Hz, 1H), 2.19–2.12 (m, 1H), 1.04–0.98 (m, 2H), 0.73–0.67 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 173.8, 162.3, 150.5, 147.6, 146.2, 137.6, 136.4, 132.4, 127.1, 126.41, 126.31, 125.5, 125.1, 122.8, 120.4, 119.8, 119.6, 13.5, 9.8. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{20}\text{H}_{17}\text{N}_2\text{O}_3$: 333.1239; found: 333.1234. FT-IR (neat, cm^{-1}) ν 3357, 2923, 1752, 1683, 1530, 1498, 1125, 1086, 752.



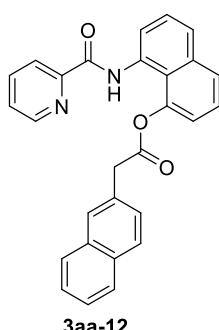
8-(Picolinamido)naphthalen-1-yl cyclobutanecarboxylate (**3aa-9**). Following the general procedure, **3aa-9** was obtained as a colorless solid (33.2 mg, 48%). $R_f = 0.52$ (n-hexane/EtOAc 3:1). m.p. 127.0–129.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.46 (s, 1H), 8.77–8.69 (m, 2H), 8.37 (d, $J = 8.0$ Hz, 1H), 7.95 (dt, $J = 7.6, 1.6$ Hz, 1H), 7.76 (dd, $J = 8.0, 1.2$ Hz, 1H), 7.68 (dd, $J = 8.0, 1.2$ Hz, 1H), 7.57–7.50 (m, 2H), 7.46 (t, $J = 8.0$ Hz, 1H), 7.17 (dd, $J = 7.6, 1.2$ Hz, 1H), 3.81–3.70 (m, 1H), 2.43–2.32 (m, 2H), 2.18–2.08 (m, 2H), 2.00–1.92 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 174.3, 162.2, 150.5, 147.5, 146.1, 137.8, 136.4, 132.5, 127.0, 126.6, 126.4, 125.3, 125.0, 123.0, 120.2, 119.4, 119.3, 38.2, 25.3, 18.5. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{21}\text{H}_{19}\text{N}_2\text{O}_3$: 347.1396; found: 347.1390. FT-IR (neat, cm^{-1}) ν 3345, 2924, 1758, 1682, 1529, 1496, 1108, 1032, 753.



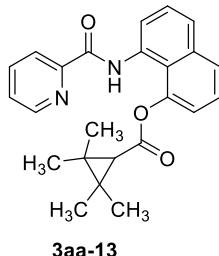
8-(Picolinamido)naphthalen-1-yl cyclopentanecarboxylate (**3aa-10**). Following the general procedure, **3aa-10** was obtained as a yellow solid (30.2 mg, 42%). $R_f = 0.60$ (n-hexane/EtOAc 3:1). m.p. 116.0–118.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.53 (s, 1H), 8.76 (d, $J = 7.6$ Hz, 1H), 8.62 (d, $J = 3.6$ Hz, 1H), 8.38 (d, $J = 7.6$ Hz, 1H), 7.95 (t, $J = 7.6$ Hz, 1H), 7.75 (d, $J = 8.0$ Hz, 1H), 7.68 (d, $J = 8.0$ Hz, 1H), 7.57-7.49 (m, 2H), 7.45 (t, $J = 8.0$ Hz, 1H), 7.15 (d, $J = 7.6$ Hz, 1H), 3.46-3.35 (m, 1H), 1.94-1.82 (m, 4H), 1.79-1.71 (m, 2H), 1.61-1.53 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 175.7, 162.2, 150.5, 147.4, 146.2, 137.8, 136.4, 132.6, 127.0, 126.5, 126.4, 125.3, 125.0, 123.0, 120.2, 119.4, 119.3, 43.8, 30.2, 25.8. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{22}\text{H}_{21}\text{N}_2\text{O}_3$: 361.1552; found: 361.1547. FT-IR (neat, cm^{-1}) ν 3339, 2928, 1760, 1682, 1529, 1496, 1105, 752.



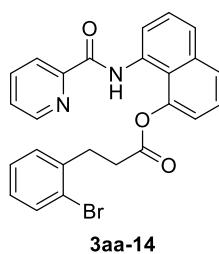
8-(Picolinamido)naphthalen-1-yl cyclohexanecarboxylate (**3aa-11**). Following the general procedure, **3aa-11** was obtained as a colorless solid (56.1 mg, 75%). $R_f = 0.65$ (n-hexane/EtOAc 3:1). m.p. 148.0–150.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.42 (s, 1H), 8.74-8.67 (m, 2H), 8.38 (d, $J = 8.0$ Hz, 1H), 7.96 (dt, $J = 8.0, 1.6$ Hz, 1H), 7.75 (d, $J = 8.0$ Hz, 1H), 7.69 (d, $J = 8.0$ Hz, 1H), 7.56-7.50 (m, 2H), 7.45 (t, $J = 7.6$ Hz, 1H), 7.10 (dd, $J = 7.6, 1.2$ Hz, 1H), 2.95-2.86 (m, 1H), 1.97-1.90 (m, 2H), 1.83-1.68 (m, 3H), 1.66-1.61 (m, 1H), 1.59-1.50 (m, 2H), 1.20-1.12 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 175.0, 162.2, 150.5, 147.7, 146.3, 137.8, 136.4, 132.5, 127.0, 126.5, 126.4, 125.3, 125.1, 123.0, 120.2, 119.6, 119.5, 42.7, 28.8, 25.6, 25.3. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{23}\text{H}_{23}\text{N}_2\text{O}_3$: 375.1709; found: 375.1703. FT-IR (neat, cm^{-1}) ν 3344, 2928, 1761, 1684, 1531, 1497, 1106, 1023, 751.



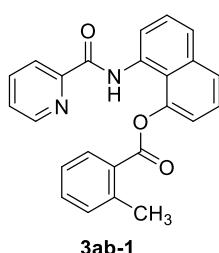
8-(Picolinamido)naphthalen-1-yl 2-(naphthalen-2-yl)acetate (**3aa-12**). Following the general procedure, **3aa-12** was obtained as a colorless solid (78.6 mg, 91%). $R_f = 0.48$ (n-hexane/EtOAc 3:1). m.p. 178.0–180.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.67 (s, 1H), 8.81 (dd, $J = 8.0, 1.2$ Hz, 1H), 8.68 (d, $J = 4.4$ Hz, 1H), 8.42 (d, $J = 7.6$ Hz, 1H), 8.10-8.04 (m, 1H), 7.93 (dt, $J = 7.6, 1.6$ Hz, 1H), 7.90-7.85 (m, 1H), 7.82 (d, $J = 8.0$ Hz, 1H), 7.73 (d, $J = 7.6$ Hz, 1H), 7.69 (d, $J = 8.0$ Hz, 1H), 7.58-7.41 (m, 6H), 7.36 (t, $J = 8.0$ Hz, 1H), 6.99 (dd, $J = 7.6, 1.2$ Hz, 1H), 4.58 (s, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 170.2, 162.3, 150.4, 147.7, 146.0, 137.9, 136.4, 133.9, 132.4, 132.0, 129.7, 128.8, 128.4, 127.9, 127.2, 126.7, 126.6, 126.5, 125.9, 125.5, 125.2, 125.1, 123.6, 123.1, 120.2, 119.6, 119.2, 39.1. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{28}\text{H}_{21}\text{N}_2\text{O}_3$: 433.1552; found: 433.1547. FT-IR (neat, cm^{-1}) ν 3349, 2923, 1766, 1682, 1530, 1497, 1101, 753.



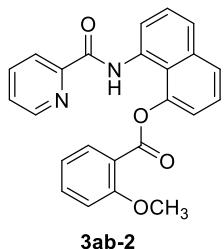
8-(Picolinamido)naphthalen-1-yl 2,2,3,3-tetramethylcyclopropane-1-carboxylate (**3aa-13**). Following the general procedure, **3aa-13** was obtained as a white solid (38.1 mg, 49%). $R_f = 0.70$ (n-hexane/EtOAc 3:1). m.p. 172.0–174.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.70 (s, 1H), 8.77 (d, $J = 7.2$ Hz, 1H), 8.67 (d, $J = 4.8$ Hz, 1H), 8.40 (d, $J = 8.0$ Hz, 1H), 7.93 (dt, $J = 8.0, 1.6$ Hz, 1H), 7.74 (d, $J = 8.0$ Hz, 1H), 7.66 (d, $J = 7.6$ Hz, 1H), 7.54–7.43 (m, 3H), 7.18 (dd, $J = 7.6, 0.8$ Hz, 1H), 1.79 (s, 1H), 1.16 (s, 6H), 0.90 (s, 6H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 170.1, 162.0, 150.5, 147.9, 145.9, 137.7, 136.3, 132.8, 126.7, 126.4, 126.3, 125.2, 124.7, 122.9, 121.0, 119.5, 118.6, 35.2, 32.0, 22.9, 16.3. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{24}\text{H}_{25}\text{N}_2\text{O}_3$: 389.1865; found: 389.1860. FT-IR (neat, cm^{-1}) ν 3336, 2923, 1750, 1685, 1533, 1499, 1098, 1030, 752.



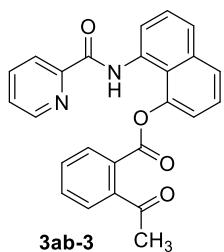
8-(picolinamido)naphthalen-1-yl 3-(2-bromophenyl)propanoate (**3aa-14**). Following the general procedure, **3aa-14** was obtained as a white solid (59.9 mg, 63%). $R_f = 0.50$ (n-hexane/EtOAc 3:1). m.p. 124.0–126.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.48 (s, 1H), 8.78 (d, $J = 7.6$ Hz, 1H), 8.46 (d, $J = 4.4$ Hz, 1H), 8.34 (d, $J = 8.0$ Hz, 1H), 7.91 (t, $J = 7.2$ Hz, 1H), 7.76 (d, $J = 8.0$ Hz, 1H), 7.68 (d, $J = 8.4$ Hz, 1H), 7.57–7.49 (m, 2H), 7.47–7.38 (m, 2H), 7.24–7.14 (m, 2H), 7.13–7.07 (m, 2H), 3.19–3.09 (m, 4H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 171.4, 162.2, 150.2, 147.7, 145.8, 139.3, 137.7, 136.4, 132.9, 132.5, 130.5, 128.3, 127.6, 127.2, 126.51, 126.45, 125.2, 125.0, 124.3, 122.9, 120.3, 119.4, 119.1, 34.3, 31.0. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{25}\text{H}_{20}\text{BrN}_2\text{O}_3$: 475.0657; found: 475.0652. FT-IR (neat, cm^{-1}) ν 3344, 2921, 1765, 1682, 1529, 1496, 1100, 750.



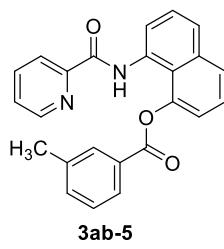
8-(Picolinamido)naphthalen-1-yl 2-methylbenzoate (**3ab-1**). Following the general procedure, **3ab-1** was obtained as a yellow solid (64.9 mg, 85%). $R_f = 0.50$ (n-hexane/EtOAc 3:1). m.p. 128.0–130.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.43 (s, 1H), 8.66 (d, $J = 7.6$ Hz, 1H), 8.26 (dd, $J = 7.6, 0.8$ Hz, 1H), 8.16 (d, $J = 7.6$ Hz, 1H), 7.81 (d, $J = 8.0$ Hz, 1H), 7.75–7.68 (m, 2H), 7.56 (t, $J = 8.0$ Hz, 1H), 7.50 (t, $J = 8.0$ Hz, 1H), 7.43 (dt, $J = 7.6, 1.2$ Hz, 1H), 7.30–7.22 (m, 3H), 7.18 (d, $J = 7.6$ Hz, 1H), 7.13–7.07 (m, 1H), 2.47 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 166.3, 162.4, 149.6, 147.1, 146.9, 141.7, 137.1, 136.5, 132.7, 132.4, 131.8, 131.7, 129.0, 127.3, 126.5, 125.81, 125.80, 125.5, 125.2, 122.0, 120.2, 120.1, 120.0, 21.6. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{24}\text{H}_{19}\text{N}_2\text{O}_3$: 383.1396; found: 383.1390. FT-IR (neat, cm^{-1}) ν 3334, 2922, 1741, 1685, 1531, 1497, 1218, 1031, 737.



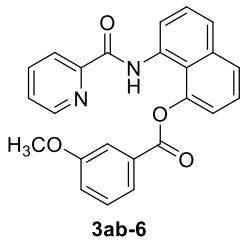
8-(Picolinamido)naphthalen-1-yl 2-methoxybenzoate (**3ab-2**). Following the general procedure, **3ab-2** was obtained as a yellow solid (39.8 mg, 50%). $R_f = 0.20$ (n-hexane/EtOAc 3:1). m.p. 144.0–146.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.54 (s, 1H), 8.69 (d, $J = 7.6$ Hz, 1H), 8.18 (d, $J = 7.6$ Hz, 1H), 8.06 (dd, $J = 7.6, 1.6$ Hz, 1H), 7.78 (d, $J = 8.0$ Hz, 1H), 7.75–7.68 (m, 2H), 7.55 (t, $J = 8.0$ Hz, 1H), 7.52–7.45 (m, 2H), 7.38 (d, $J = 7.6$ Hz, 1H), 7.29 (d, $J = 7.2$ Hz, 1H), 7.15–7.08 (m, 1H), 6.98 (t, $J = 7.6$ Hz, 1H), 6.87 (d, $J = 8.4$ Hz, 1H), 3.72 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 164.7, 162.4, 159.9, 149.8, 147.2, 146.9, 137.0, 136.4, 134.3, 132.7, 132.6, 127.0, 126.4, 125.7, 125.4, 125.0, 122.0, 120.0, 119.9, 119.7, 119.5, 111.9, 55.8. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{24}\text{H}_{19}\text{N}_2\text{O}_4$: 399.1345; found: 399.1339. FT-IR (neat, cm^{-1}) ν 3336, 2925, 1750, 1684, 1531, 1495, 1214, 1025, 754.



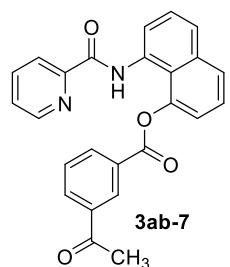
8-(Picolinamido)naphthalen-1-yl 2-acetylbenzoate (**3ab-3**). Following the general procedure, **3ab-3** was obtained as a yellow solid (50.0 mg, 61%). $R_f = 0.20$ (n-hexane/EtOAc 3:1). m.p. 176.0–178.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.02 (s, 1H), 8.46 (d, $J = 7.6$ Hz, 1H), 8.25 (d, $J = 8.0$ Hz, 2H), 8.14 (d, $J = 6.8$ Hz, 1H), 7.91 (d, $J = 8.0$ Hz, 2H), 7.83 (d, $J = 7.6$ Hz, 1H), 7.77 (d, $J = 8.4$ Hz, 1H), 7.69 (t, $J = 6.0$ Hz, 1H), 7.56 (t, $J = 7.6$ Hz, 1H), 7.49 (t, $J = 7.6$ Hz, 1H), 7.44 (d, $J = 4.0$ Hz, 1H), 7.24 (d, $J = 7.2$ Hz, 1H), 7.15–7.03 (m, 1H), 2.64 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 197.3, 164.9, 162.4, 149.5, 147.0, 146.5, 140.4, 137.2, 136.5, 133.4, 131.8, 130.8, 128.0, 127.6, 126.5, 125.9, 125.8, 125.5, 122.3, 121.4, 120.6, 120.1, 26.9. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{25}\text{H}_{19}\text{N}_2\text{O}_4$: 411.1345; found: 411.1339. FT-IR (neat, cm^{-1}) ν 3351, 2922, 1742, 1685, 1529, 1496, 1226, 1055, 754.



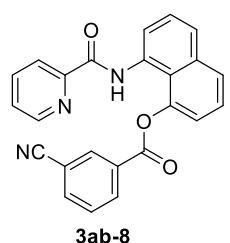
8-(picolinamido)naphthalen-1-yl 3-methylbenzoate (**3ab-5**). Following the general procedure, **3ab-5** was obtained as a yellow solid (63.1 mg, 71%). $R_f = 0.50$ (n-hexane/EtOAc 3:1). m.p. 156.0–158.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.31 (s, 1H), 8.59 (d, $J = 7.6$ Hz, 1H), 8.17 (d, $J = 7.6$ Hz, 1H), 8.04 (d, $J = 7.6$ Hz, 1H), 8.00 (s, 1H), 7.81 (d, $J = 8.0$ Hz, 1H), 7.77–7.69 (m, 2H), 7.56 (t, $J = 8.0$ Hz, 1H), 7.49 (t, $J = 8.0$ Hz, 1H), 7.40 (d, $J = 7.6$ Hz, 1H), 7.35–7.30 (m, 2H), 7.23 (dd, $J = 7.6, 0.4$ Hz, 1H), 7.15–7.09 (m, 1H), 2.37 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 166.1, 162.6, 149.7, 147.2, 147.1, 138.2, 137.0, 136.5, 134.3, 132.3, 131.2, 129.8, 128.3, 128.0, 127.3, 126.5, 125.8, 125.5, 125.4, 122.1, 120.5, 120.4, 120.1, 21.2. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{24}\text{H}_{19}\text{N}_2\text{O}_3$: 383.1396; found: 383.1390. FT-IR (neat, cm^{-1}) ν 3344, 2920, 1739, 1684, 1530, 1496, 1260, 1177, 1050, 748.



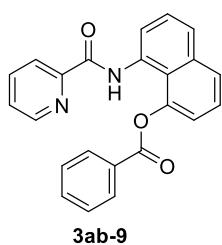
8-(Picolinamido)naphthalen-1-yl 3-methoxybenzoate (3ab-6**).** Following the general procedure, **3ab-6** was obtained as a yellow solid (50.9 mg, 64%). $R_f = 0.30$ (n-hexane/EtOAc 3:1). m.p. 142.0–144.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.26 (s, 1H), 8.57 (d, $J = 7.6$ Hz, 1H), 8.16 (d, $J = 7.6$ Hz, 1H), 7.84 (d, $J = 7.6$ Hz, 1H), 7.81 (d, $J = 8.0$ Hz, 1H), 7.77–7.65 (m, 3H), 7.56 (t, $J = 7.6$ Hz, 1H), 7.49 (t, $J = 7.6$ Hz, 1H), 7.44 (d, $J = 4.4$ Hz, 1H), 7.33 (t, $J = 8.0$ Hz, 1H), 7.23 (dd, $J = 7.6, 0.8$ Hz, 1H), 7.16–7.08 (m, 2H), 3.80 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 165.7, 162.5, 159.4, 149.6, 147.2, 146.9, 137.0, 136.4, 132.2, 131.0, 129.3, 127.3, 126.5, 125.9, 125.5, 125.4, 123.2, 122.1, 120.7, 120.4, 120.3, 120.0, 114.7, 55.4. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{24}\text{H}_{19}\text{N}_2\text{O}_4$: 399.1345; found: 399.1339. FT-IR (neat, cm^{-1}) ν 3351, 2916, 1737, 1683, 1529, 1492, 1263, 1206, 1032, 748.



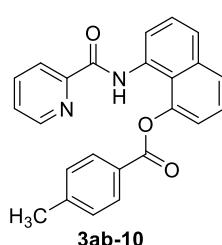
8-(Picolinamido)naphthalen-1-yl 3-acetylbenzoate (3ab-7**).** Following the general procedure, **3ab-7** was obtained as a yellow solid (49.2 mg, 60%). $R_f = 0.20$ (n-hexane/EtOAc 3:1). m.p. 147.0–149.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.00 (s, 1H), 8.67 (s, 1H), 8.43 (d, $J = 7.6$ Hz, 1H), 8.38 (d, $J = 7.6$ Hz, 1H), 8.16–8.10 (m, 2H), 7.83 (d, $J = 8.0$ Hz, 1H), 7.76 (d, $J = 7.6$ Hz, 1H), 7.67 (dt, $J = 7.6, 1.2$ Hz, 1H), 7.56 (t, $J = 7.6$ Hz, 1H), 7.50 (dt, $J = 7.6, 2.4$ Hz, 2H), 7.40 (d, $J = 4.4$ Hz, 1H), 7.24 (dd, $J = 7.6, 0.8$ Hz, 1H), 7.08 (dd, $J = 7.6, 4.4$ Hz, 1H), 2.59 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 196.9, 164.9, 162.3, 149.4, 147.0, 146.5, 137.1, 136.4, 134.8, 132.6, 131.7, 130.5, 130.2, 128.8, 127.5, 126.5, 125.9, 125.8, 125.5, 122.2, 121.6, 120.7, 120.1, 26.7. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{25}\text{H}_{19}\text{N}_2\text{O}_4$: 411.1345; found: 411.1339. FT-IR (neat, cm^{-1}) ν 3344, 2920, 1743, 1686, 1529, 1496, 1211, 749.



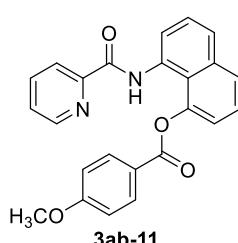
8-(Picolinamido)naphthalen-1-yl 3-cyanobenzoate (3ab-8**).** Following the general procedure, **3ab-8** was obtained as a yellow solid (35.4 mg, 45%). $R_f = 0.20$ (n-hexane/EtOAc 3:1). m.p. 176.0–178.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 10.8 (s, 1H), 8.45 (s, 1H), 8.35 (dt, $J = 8.8, 1.6$ Hz, 1H), 8.31 (d, $J = 7.6$ Hz, 1H), 8.17 (d, $J = 8.0$ Hz, 1H), 7.86 (dd, $J = 8.4, 1.2$ Hz, 1H), 7.82–7.74 (m, 3H), 7.72 (d, $J = 4.8$ Hz, 1H), 7.57 (t, $J = 8.0$ Hz, 1H), 7.53–7.46 (m, 2H), 7.25–7.20 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 163.8, 162.4, 149.5, 147.2, 146.2, 137.4, 136.5, 136.3, 134.5, 134.0, 131.4, 130.9, 129.3, 127.9, 126.6, 126.21, 126.18, 125.5, 122.5, 122.3, 121.0, 120.1, 117.5, 112.8. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{24}\text{H}_{16}\text{N}_3\text{O}_3$: 394.1192; found: 394.1186. FT-IR (neat, cm^{-1}) ν 3362, 2918, 1745, 1684, 1529, 1495, 1258, 1166, 749.



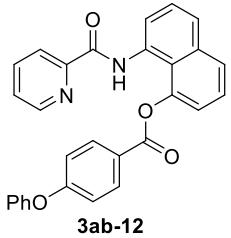
8-(Picolinamido)naphthalen-1-yl benzoate (3ab-9**).** Following the general procedure, **3ab-9** was obtained as a yellow solid (62.6 mg, 85%). m.p. 148.0–150.0 °C. R_f = 0.38 (n-hexane/EtOAc 3:1). ^1H NMR (400 MHz, CDCl_3) δ 11.34 (s, 1H), 8.63 (d, J = 7.6 Hz, 1H), 8.23 (d, J = 7.2 Hz, 2H), 8.16 (d, J = 7.6 Hz, 1H), 7.81 (d, J = 8.0 Hz, 1H), 7.76–7.67 (m, 2H), 7.63–7.54 (m, 2H), 7.49 (t, J = 8.0 Hz, 1H), 7.44 (t, J = 8.0 Hz, 2H), 7.31 (t, J = 4.4 Hz, 1H), 7.23 (dd, J = 7.6, 0.8 Hz, 1H), 7.13–7.08 (m, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 165.9, 162.5, 149.6, 147.2, 147.0, 137.1, 136.5, 133.5, 132.3, 130.7, 129.9, 128.4, 127.3, 126.5, 125.8, 125.5, 125.4, 122.1, 120.4, 120.3, 120.0. HRMS (ESI): m/z [M+H]⁺ calcd for $\text{C}_{23}\text{H}_{17}\text{N}_2\text{O}_3$: 369.1239; found: 369.1234. FT-IR (neat, cm^{-1}) ν 3345, 2921, 1740, 1683, 1529, 1495, 1220, 1049, 752.



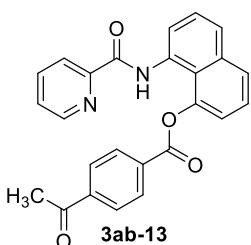
8-(Picolinamido)naphthalen-1-yl 4-methylbenzoate (3ab-10**).** Following the general procedure, **3ab-10** was obtained as a yellow solid (71.8 mg, 94%). R_f = 0.38 (n-hexane/EtOAc 3:1). m.p. 159.0–161.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.33 (s, 1H), 8.62 (d, J = 7.6 Hz, 1H), 8.15 (d, J = 7.6 Hz, 1H), 8.10 (d, J = 7.6 Hz, 2H), 7.80 (d, J = 8.4 Hz, 1H), 7.75–7.68 (m, 2H), 7.56 (t, J = 8.0 Hz, 1H), 7.48 (t, J = 8.0 Hz, 1H), 7.37 (d, J = 4.4 Hz, 1H), 7.24–7.18 (m, 3H), 7.15–7.09 (m, 1H), 2.43 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 165.6, 162.5, 149.6, 147.2, 147.0, 144.3, 137.0, 136.4, 132.3, 130.8, 129.0, 127.2, 127.1, 126.4, 125.6, 125.5, 125.3, 122.1, 120.31, 120.29, 120.0, 21.7. HRMS (ESI): m/z [M+H]⁺ calcd for $\text{C}_{24}\text{H}_{19}\text{N}_2\text{O}_3$: 383.1396; found: 383.1390. FT-IR (neat, cm^{-1}) ν 3345, 2920, 1737, 1683, 1529, 1496, 1219, 1049, 748.



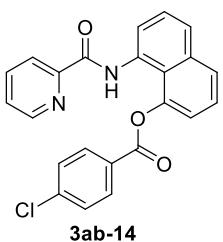
8-(Picolinamido)naphthalen-1-yl 4-methoxybenzoate (3ab-11**).** Following the general procedure, **3ab-11** was obtained as a white solid (54.1 mg, 68%). R_f = 0.50 (n-hexane/EtOAc 3:1). m.p. 209.0–211.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.36 (s, 1H), 8.59 (dd, J = 7.6, 0.8 Hz, 1H), 8.22–8.14 (m, 3H), 7.80 (dd, J = 8.4, 1.2 Hz, 1H), 7.77–7.72 (m, 2H), 7.56 (t, J = 8.0 Hz, 1H), 7.52–7.46 (m, 2H), 7.22 (dd, J = 7.6, 1.2 Hz, 1H), 7.20–7.15 (m, 1H), 6.92–6.86 (m, 2H), 3.90 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 165.6, 163.8, 162.4, 149.7, 147.2, 147.1, 137.3, 136.5, 133.0, 132.3, 127.1, 126.5, 125.8, 125.5, 125.4, 122.3, 122.2, 120.5, 120.4, 120.1, 113.6, 55.6. HRMS (ESI): m/z [M+H]⁺ calcd for $\text{C}_{24}\text{H}_{19}\text{N}_2\text{O}_4$: 399.1345; found: 399.1339. FT-IR (neat, cm^{-1}) ν 3337, 2922, 1731, 1680, 1527, 1496, 1247, 1028, 754.



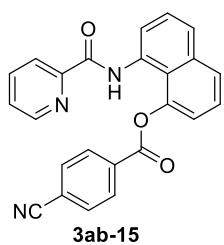
8-(Picolinamido)naphthalen-1-yl 4-phenoxybenzoate (3ab-12). Following the general procedure, **3ab-12** was obtained as a white solid (88.3 mg, 96%). R_f = 0.38 (n-hexane/EtOAc 3:1). m.p. 137.0–139.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.31 (s, 1H), 8.62 (dd, J = 8.0, 1.2 Hz, 1H), 8.24–8.17 (m, 3H), 7.84–7.74 (m, 3H), 7.65–7.61 (m, 1H), 7.58 (t, J = 8.0 Hz, 1H), 7.53–7.43 (m, 3H), 7.29–7.22 (m, 3H), 7.13–7.07 (m, 2H), 7.00–7.95 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 165.2, 162.4, 162.2, 155.3, 149.7, 147.2, 146.9, 137.1, 136.4, 132.9, 132.2, 130.1, 127.2, 126.4, 125.8, 125.5, 125.4, 124.7, 123.8, 122.2, 120.5, 120.3, 120.1, 120.0, 117.1. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{29}\text{H}_{21}\text{N}_2\text{O}_4$: 461.1501; found: 461.1496. FT-IR (neat, cm^{-1}) ν 3344, 2922, 1737, 1684, 1530, 1494, 1240, 1048, 751.



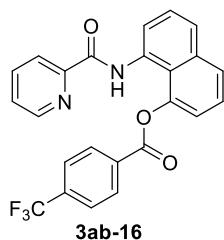
8-(Picolinamido)naphthalen-1-yl 4-acetylbenzoate (3ab-13). Following the general procedure, **3ab-13** was obtained as a yellow solid (25.4 mg, 31%). R_f = 0.20 (n-hexane/EtOAc 3:1). m.p. 173.0–175.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.01 (s, 1H), 8.46 (d, J = 8.0 Hz, 1H), 8.26 (d, J = 8.0 Hz, 2H), 8.14 (d, J = 8.0 Hz, 1H), 7.93 (d, J = 8.0 Hz, 2H), 7.84 (d, J = 8.0 Hz, 1H), 7.78 (d, J = 8.0 Hz, 1H), 7.71 (t, J = 7.2 Hz, 1H), 7.57 (t, J = 8.0 Hz, 1H), 7.51 (t, J = 8.0 Hz, 1H), 7.46 (d, J = 4.8 Hz, 1H), 7.25 (d, J = 8.0 Hz, 1H), 7.13–7.07 (m, 1H), 2.65 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 197.3, 164.9, 162.5, 149.6, 147.1, 146.6, 140.5, 137.2, 136.5, 133.4, 131.8, 130.9, 128.0, 127.6, 126.6, 125.9, 125.8, 125.5, 122.3, 121.5, 120.6, 120.1, 26.9. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{25}\text{H}_{19}\text{N}_2\text{O}_4$: 411.1345; found: 411.1339. FT-IR (neat, cm^{-1}) ν 3355, 2917, 1741, 1684, 1529, 1496, 1228, 1053, 754.



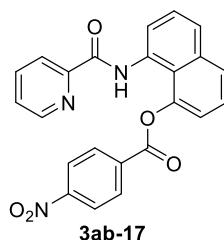
8-(Picolinamido)naphthalen-1-yl 4-chlorobenzoate (3ab-14). Following the general procedure, **3ab-14** was obtained as a yellow solid (70.8 mg, 88%). R_f = 0.48 (n-hexane/EtOAc 3:1). m.p. 143.0–145.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.00 (s, 1H), 8.45 (d, J = 7.6 Hz, 1H), 8.16 (d, J = 7.6 Hz, 1H), 8.09 (d, J = 7.6 Hz, 2H), 7.82 (d, J = 8.4 Hz, 1H), 7.78–7.71 (m, 2H), 7.59–7.53 (m, 2H), 7.48 (t, J = 7.6 Hz, 1H), 7.32 (d, J = 7.6 Hz, 2H), 7.24–7.18 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 146.9, 162.4, 149.5, 147.1, 146.6, 139.9, 137.2, 136.4, 131.9, 131.8, 128.6, 128.1, 127.5, 126.5, 125.9, 125.8, 125.5, 122.3, 121.5, 120.7, 120.1. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{23}\text{H}_{16}\text{ClN}_2\text{O}_3$: 403.0849; found: 403.0844. FT-IR (neat, cm^{-1}) ν 3349, 2926, 1740, 1683, 1528, 1492, 1219, 1051, 750.



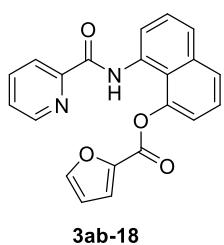
8-(Picolinamido)naphthalen-1-yl 4-cyanobenzoate (**3ab-15**). Following the general procedure, **3ab-15** was obtained as a white solid (55.0 mg, 70%). $R_f = 0.25$ (n-hexane/EtOAc 3:1). m.p. 163.0–165.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 10.78 (s, 1H), 8.35 (d, $J = 7.2$ Hz, 1H), 8.25 (d, $J = 8.0$ Hz, 2H), 8.15 (d, $J = 7.2$ Hz, 1H), 7.85 (d, $J = 7.6$ Hz, 1H), 7.82–7.72 (m, 2H), 7.63 (d, $J = 8.0$ Hz, 2H), 7.60–7.54 (m, 2H), 7.51 (t, $J = 8.0$ Hz, 1H), 7.25–7.19 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 164.1, 162.4, 149.5, 147.0, 146.2, 137.4, 136.5, 133.4, 132.0, 131.4, 131.0, 127.9, 126.6, 126.2, 126.1, 125.5, 122.5, 122.3, 120.8, 120.1, 117.7, 116.7. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{24}\text{H}_{16}\text{N}_3\text{O}_3$: 394.1192; found: 394.1186. FT-IR (neat, cm^{-1}) ν 3352, 2921, 1745, 1685, 1530, 1496, 1251, 1222, 1061, 754.



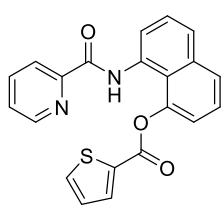
8-(Picolinamido)naphthalen-1-yl 4-(trifluoromethyl)benzoate (**3ab-16**). Following the general procedure, **3ab-16** was obtained as a yellow solid (49.7 mg, 57%). $R_f = 0.52$ (n-hexane/EtOAc 3:1). m.p. 150.0–152.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 10.80 (s, 1H), 8.33 (d, $J = 7.6$ Hz, 1H), 8.24 (d, $J = 8.0$ Hz, 2H), 8.13 (d, $J = 8.0$ Hz, 1H), 7.85 (d, $J = 8.0$ Hz, 1H), 7.79 (d, $J = 8.0$ Hz, 1H), 7.71 (dt, $J = 7.6, 1.6$ Hz, 1H), 7.61–7.48 (m, 5H), 7.25 (dd, $J = 7.6, 0.8$ Hz, 1H), 7.17–7.11 (m, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 164.5, 162.5, 149.5, 147.1, 146.4, 137.3, 136.6, 134.7 (q, $J_{\text{C}-\text{F}} = 32.6$ Hz), 132.9, 131.6, 130.9, 127.8, 126.6, 126.2, 126.1, 125.3, 125.2 (q, $J_{\text{C}-\text{F}} = 3.7$ Hz), 123.5 (q, $J_{\text{C}-\text{F}} = 271.1$ Hz), 122.4, 121.0, 120.2. ^{19}F NMR (376 MHz, CDCl_3) δ -63.2. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{24}\text{H}_{16}\text{F}_3\text{N}_2\text{O}_3$: 437.1113; found: 437.1108. FT-IR (neat, cm^{-1}) ν 3345, 2920, 1744, 1684, 1528, 1494, 1321, 1063, 752.



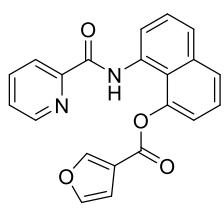
8-(Picolinamido)naphthalen-1-yl 4-nitrobenzoate (**3ab-17**). Following the general procedure, **3ab-17** was obtained as a yellow solid (57.8 mg, 70%). $R_f = 0.28$ (n-hexane/EtOAc 3:1). m.p. 187.0–189.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 10.72 (s, 1H), 8.30 (d, $J = 8.8$ Hz, 3H), 8.13 (d, $J = 8.8$ Hz, 3H), 7.86 (dd, $J = 8.0, 1.2$ Hz, 1H), 7.80 (dd, $J = 8.0, 1.2$ Hz, 1H), 7.72 (dt, $J = 7.6, 1.6$ Hz, 1H), 7.63–7.60 (m, 1H), 7.57 (t, $J = 8.0$ Hz, 1H), 7.52 (t, $J = 8.0$ Hz, 1H), 7.25 (dd, $J = 7.6, 1.2$ Hz, 1H), 7.18–7.13 (m, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 163.8, 162.4, 150.5, 149.5, 147.0, 146.1, 137.4, 136.5, 134.9, 131.6, 131.3, 127.9, 126.6, 126.3, 126.1, 125.5, 123.2, 122.6, 122.5, 120.9, 120.1. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{23}\text{H}_{16}\text{N}_3\text{O}_5$: 414.1090; found: 414.1084. FT-IR (neat, cm^{-1}) ν 3342, 2923, 1742, 1673, 1523, 1496, 1240, 1063, 753.



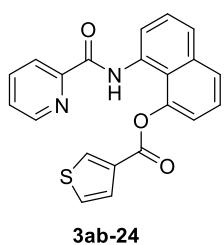
8-(Picolinamido)naphthalen-1-yl furan-2-carboxylate (**3ab-18**). Following the general procedure, **3ab-18** was obtained as a white solid (40.1 mg, 56%). $R_f = 0.25$ (n-hexane/EtOAc 3:1). m.p. 167.0–169.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.19 (s, 1H), 8.51 (d, $J = 7.6$ Hz, 1H), 8.22 (d, $J = 8.0$ Hz, 1H), 7.92 (d, $J = 4.0$ Hz, 1H), 7.83–7.72 (m, 3H), 7.59–7.52 (m, 2H), 7.48 (t, $J = 7.6$ Hz, 1H), 7.37 (d, $J = 7.6$ Hz, 1H), 7.29–7.21 (m, 2H), 6.50–6.42 (m, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 162.5, 157.2, 149.8, 147.4, 147.1, 146.0, 144.2, 137.3, 136.4, 132.0, 127.5, 126.5, 125.9, 125.6, 125.4, 122.3, 121.1, 120.4, 120.2, 120.0, 112.2. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{21}\text{H}_{15}\text{N}_2\text{O}_4$: 359.1032; found: 359.1026. FT-IR (neat, cm^{-1}) ν 3347, 2921, 1741, 1683, 1530, 1496, 1066, 753.



8-(Picolinamido)naphthalen-1-yl thiophene-2-carboxylate (**3ab-20**). Following the general procedure, **3ab-20** was obtained as a white solid (66.6 mg, 89%). $R_f = 0.30$ (n-hexane/EtOAc 3:1). m.p. 180.0–182.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.34 (s, 1H), 8.62 (d, $J = 7.6$ Hz, 1H), 8.20 (d, $J = 7.6$ Hz, 1H), 8.00 (d, $J = 3.6$ Hz, 1H), 7.82 (d, $J = 8.0$ Hz, 1H), 7.78–7.72 (m, 2H), 7.63 (d, $J = 4.4$ Hz, 2H), 7.58 (t, $J = 7.6$ Hz, 1H), 7.49 (t, $J = 7.6$ Hz, 1H), 7.29–7.24 (m, 1H), 7.22–7.17 (m, 1H), 7.10 (t, $J = 4.4$ Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 162.5, 161.1, 149.6, 147.1, 146.5, 137.1, 136.4, 135.2, 133.9, 133.3, 132.1, 127.8, 127.4, 126.4, 125.9, 125.40, 125.36, 122.1, 120.6, 120.3, 120.0. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{21}\text{H}_{15}\text{N}_2\text{O}_3\text{S}$: 375.0803; found: 375.0798. FT-IR (neat, cm^{-1}) ν 3333, 2917, 1717, 1679, 1490, 1420, 1252, 1218, 736.

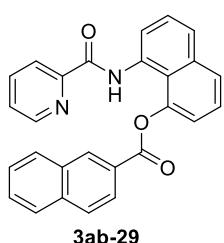


8-(Picolinamido)naphthalen-1-yl furan-3-carboxylate (**3ab-22**). Following the general procedure, **3ab-22** was obtained as a white solid (55.5 mg, 72%). $R_f = 0.30$ (n-hexane/EtOAc 3:1). m.p. 147.0–149.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.25 (s, 1H), 8.60 (d, $J = 8.0$ Hz, 1H), 8.30–8.21 (m, 2H), 7.95 (d, $J = 4.4$ Hz, 1H), 7.84–7.78 (m, 2H), 7.75 (d, $J = 7.8$ Hz, 1H), 7.58 (t, $J = 8.0$ Hz, 1H), 7.50 (t, $J = 8.0$ Hz, 1H), 7.42 (t, $J = 1.6$ Hz, 1H), 7.35–7.30 (m, 1H), 7.24 (dd, $J = 7.8, 0.8$ Hz, 1H), 6.85 (d, $J = 1.6$ Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 162.5, 161.7, 149.8, 149.5, 147.2, 146.2, 143.6, 137.3, 136.4, 132.1, 127.3, 126.4, 126.1, 125.5, 125.4, 122.4, 120.7, 120.3, 120.1, 118.9, 110.4. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{21}\text{H}_{15}\text{N}_2\text{O}_4$: 359.1032; found: 359.1026. FT-IR (neat, cm^{-1}) ν 3333, 2921, 1735, 1680, 1491, 1428, 1114, 750.



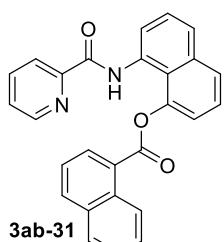
3ab-24

8-(picolinamido)naphthalen-1-yl thiophene-3-carboxylate (**3ab-24**). Following the general procedure, **3ab-24** was obtained as a white solid (68.1 mg, 91%). $R_f = 0.30$ (n-hexane/EtOAc 3:1). m.p. 162.0–164.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.29 (s, 1H), 8.58 (d, $J = 7.8$ Hz, 1H), 8.32 (d, $J = 1.6$ Hz, 1H), 8.18 (d, $J = 8.0$ Hz, 1H), 7.82–7.71 (m, 3H), 7.65 (t, $J = 4.4$ Hz, 2H), 7.55 (t, $J = 8.0$ Hz, 1H), 7.47 (t, $J = 8.0$ Hz, 1H), 7.30–7.26 (m, 1H), 7.24–7.16 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 162.5, 161.5, 149.6, 147.3, 146.6, 137.1, 136.4, 134.8, 133.0, 132.2, 128.7, 127.3, 126.4, 126.0, 125.9, 125.4, 125.3, 122.2, 120.5, 120.3, 120.0. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{21}\text{H}_{15}\text{N}_2\text{O}_3\text{S}$: 375.0803; found: 375.0798. FT-IR (neat, cm^{-1}) ν 3328, 2916, 1728, 1678, 1529, 1494, 1238, 1188, 746.



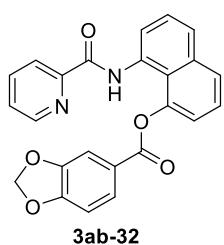
3ab-29

8-(Picolinamido)naphthalen-1-yl 2-naphthoate (**3ab-29**). Following the general procedure, **3ab-29** was obtained as a white solid (62.7 mg, 75%). $R_f = 0.48$ (n-hexane/EtOAc 3:1). m.p. 191.0–193.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.16 (s, 1H), 8.76 (s, 1H), 8.48 (d, $J = 7.6$ Hz, 1H), 8.17 (d, $J = 7.6$ Hz, 1H), 8.07 (d, $J = 7.6$ Hz, 1H), 7.89 (t, $J = 7.2$ Hz, 2H), 7.83 (t, $J = 8.0$ Hz, 2H), 7.78 (d, $J = 8.0$ Hz, 1H), 7.64 (t, $J = 7.6$ Hz, 1H), 7.60–7.45 (m, 4H), 7.30 (d, $J = 7.2$ Hz, 1H), 7.09 (d, $J = 4.4$ Hz, 1H), 6.73–6.65 (m, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 166.0, 162.6, 149.4, 146.92, 146.89, 136.8, 136.5, 135.6, 132.4, 132.2, 132.0, 129.4, 128.7, 128.1, 127.7, 127.3, 126.9, 126.8, 126.4, 125.8, 125.7, 125.6, 125.4, 122.0, 121.3, 120.8, 120.2. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{27}\text{H}_{19}\text{N}_2\text{O}_3$: 419.1396; found: 419.1390. FT-IR (neat, cm^{-1}) ν 3349, 2924, 1737, 1684, 1530, 1497, 1217, 1186, 755.

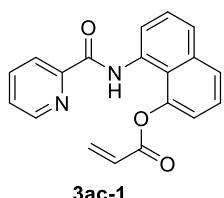


3ab-31

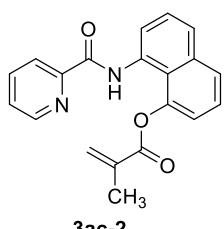
8-(Picolinamido)naphthalen-1-yl 1-naphthoate (**3ab-31**). Following the general procedure, **3ab-31** was obtained as a yellow solid (66.8 mg, 80%). $R_f = 0.48$ (n-hexane/EtOAc 3:1). m.p. 172.0–174.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.28 (s, 1H), 8.77–8.69 (m, 1H), 8.56 (d, $J = 7.6$ Hz, 1H), 8.52 (d, $J = 7.2$ Hz, 1H), 7.99 (d, $J = 8.0$ Hz, 1H), 7.90 (d, $J = 8.0$ Hz, 1H), 7.85 (d, $J = 8.0$ Hz, 1H), 7.82–7.75 (m, 2H), 7.61–7.45 (m, 5H), 7.41 (t, $J = 7.6$ Hz, 1H), 7.35 (dd, $J = 7.6, 1.2$ Hz, 1H), 6.91 (d, $J = 4.4$ Hz, 1H), 6.78–6.71 (m, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 166.1, 162.3, 149.2, 146.7, 146.5, 136.5, 136.4, 134.1, 133.5, 132.2, 131.5, 131.4, 128.22, 128.19, 127.4, 126.5, 126.4, 126.3, 125.54, 125.51, 125.4, 125.3, 124.3, 121.7, 120.7, 120.5, 120.2. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{27}\text{H}_{19}\text{N}_2\text{O}_3$: 419.1396; found: 419.1390. FT-IR (neat, cm^{-1}) ν 3343, 2917, 1735, 1684, 1530, 1497, 1108, 752.



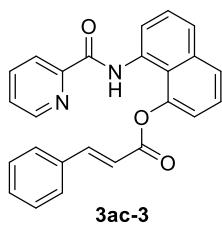
8-(Picolinamido)naphthalen-1-yl benzo[d][1,3]dioxole-5-carboxylate (**3ab-32**). Following the general procedure, **3ab-32** was obtained as a white solid (61.8 mg, 75%). $R_f = 0.25$ (n-hexane/EtOAc 3:1). m.p. 181.0–183.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.6 (s, 1H), 8.51 (d, $J = 7.6$ Hz, 1H), 8.18 (d, $J = 7.6$ Hz, 1H), 7.85–7.73 (m, 5H), 7.61 (d, $J = 1.6$ Hz, 1H), 7.55 (t, $J = 8.0$ Hz, 1H), 7.48 (t, $J = 8.0$ Hz, 1H), 7.25–7.18 (m, 2H), 6.79 (d, $J = 8.0$ Hz, 1H), 6.04 (s, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 165.1, 162.5, 152.0, 149.8, 147.5, 147.3, 146.9, 137.1, 136.5, 132.1, 127.2, 126.9, 126.4, 125.8, 125.6, 125.5, 123.6, 122.3, 120.9, 120.6, 120.1, 110.5, 107.9, 101.9. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{24}\text{H}_{17}\text{N}_2\text{O}_5$: 413.1137; found: 413.1132. FT-IR (neat, cm^{-1}) ν 3340, 2924, 1733, 1684, 1531, 1495, 1258, 1034, 753.



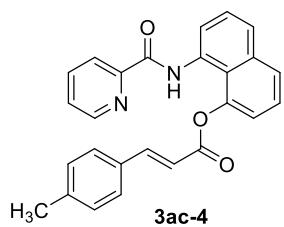
8-(picolinamido)naphthalen-1-yl acrylate (**3ac-1**). Following the general procedure, **3ac-1** was obtained as a white solid (24.8 mg, 39%). $R_f = 0.48$ (n-hexane/EtOAc 3:1). m.p. 159.0–161.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.48 (s, 1H), 8.73 (d, $J = 7.6$ Hz, 1H), 8.61 (d, $J = 4.4$ Hz, 1H), 8.33 (d, $J = 7.6$ Hz, 1H), 7.91 (dt, $J = 7.6$, 1.6 Hz, 1H), 7.78 (d, $J = 8.0$ Hz, 1H), 7.70 (d, $J = 8.0$ Hz, 1H), 7.54 (t, $J = 8.0$ Hz, 1H), 7.51–7.44 (m, 2H), 7.23 (d, $J = 7.2$ Hz, 1H), 6.60 (d, $J = 5.2$ Hz, 1H), 6.59 (s, 1H), 5.91 (dd, $J = 7.6$, 4.0 Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 164.8, 162.3, 150.2, 147.6, 145.9, 137.6, 136.4, 132.7, 132.4, 128.0, 127.3, 126.5, 126.4, 125.3, 125.1, 122.8, 120.1, 120.0, 119.4. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{19}\text{H}_{15}\text{N}_2\text{O}_3$: 319.1083; found: 319.1077. FT-IR (neat, cm^{-1}) ν 3340, 2921, 1745, 1675, 1533, 1497, 1121, 757.



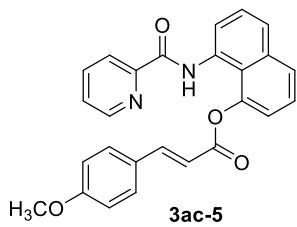
8-(picolinamido)naphthalen-1-yl methacrylate (**3ac-2**). Following the general procedure, **3ac-2** was obtained as a white solid (27.9 mg, 42%). $R_f = 0.48$ (n-hexane/EtOAc 3:1). m.p. 169.0–171.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.21 (s, 1H), 8.60 (dd, $J = 8.0$, 1.2 Hz, 1H), 8.48 (d, $J = 4.8$ Hz, 1H), 8.30 (dt, $J = 8.0$, 1.2 Hz, 1H), 7.88 (dt, $J = 7.6$, 1.2 Hz, 1H), 7.77 (dd, $J = 8.4$, 1.2 Hz, 1H), 7.72 (dd, $J = 8.4$, 1.2 Hz, 1H), 7.54 (t, $J = 7.6$ Hz, 1H), 7.48–7.42 (m, 2H), 7.13 (dd, $J = 7.6$, 1.2 Hz, 1H), 6.42 (s, 1H), 5.70 (t, $J = 1.2$ Hz, 1H), 1.99 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 166.5, 162.4, 150.2, 147.4, 146.9, 137.5, 136.4, 135.9, 132.2, 127.9, 127.2, 126.4, 126.3, 125.4, 125.3, 122.5, 120.4, 120.3, 120.0, 18.5. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{20}\text{H}_{17}\text{N}_2\text{O}_3$: 333.1239; found: 333.1234. FT-IR (neat, cm^{-1}) ν 3324, 2924, 1730, 1675, 1533, 1494, 1104, 759.



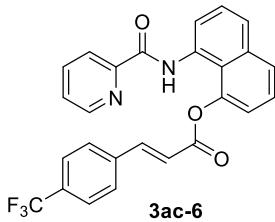
8-(picolinamido)naphthalen-1-yl cinnamate (3ac-3**).** Following the general procedure, **3ac-3** was obtained as a yellow solid (63.0 mg, 80%). $R_f = 0.48$ (n-hexane/EtOAc 3:1). m.p. 131.0–133.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.44 (s, 1H), 8.63 (d, $J = 7.6$ Hz, 1H), 8.41 (d, $J = 4.4$ Hz, 1H), 8.28 (d, $J = 7.6$ Hz, 1H), 7.83–7.70 (m, 4H), 7.55 (t, $J = 8.0$ Hz, 1H), 7.49 (t, $J = 8.0$ Hz, 1H), 7.44–7.35 (m, 5H), 7.27 (d, $J = 8.0$ Hz, 1H), 7.22–7.17 (m, 1H), 6.79 (d, $J = 15.6$ Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 165.6, 162.2, 150.1, 147.6, 146.9, 146.2, 137.5, 136.4, 134.0, 132.3, 130.8, 128.9, 128.2, 127.1, 126.4, 126.1, 125.4, 125.3, 122.7, 122.6, 120.2, 119.8, 117.5. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{25}\text{H}_{19}\text{N}_2\text{O}_3$: 395.1396; found: 395.1390. FT-IR (neat, cm^{-1}) ν 3342, 2925, 1736, 1683, 1530, 1496, 1113, 753.



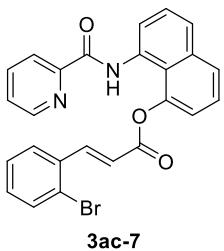
8-(picolinamido)naphthalen-1-yl 3-(p-tolyl)acrylate (3ac-4**).** Following the general procedure, **3ac-4** was obtained as a white solid (67.7 mg, 83%). $R_f = 0.60$ (n-hexane/EtOAc 3:1). m.p. 171.0–173.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.49 (s, 1H), 8.66 (d, $J = 7.6$ Hz, 1H), 8.41 (d, $J = 4.4$ Hz, 1H), 8.26 (d, $J = 8.0$ Hz, 1H), 7.82–7.73 (m, 3H), 7.69 (d, $J = 8.4$ Hz, 1H), 7.53 (t, $J = 8.0$ Hz, 1H), 7.47 (t, $J = 8.0$ Hz, 1H), 7.32 (d, $J = 8.0$ Hz, 2H), 7.27–7.21 (m, 2H), 7.18 (d, $J = 8.0$ Hz, 2H), 6.75 (d, $J = 16.0$ Hz, 1H), 2.39 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 165.8, 162.3, 150.2, 147.7, 147.0, 146.3, 141.4, 137.5, 136.4, 132.4, 131.3, 129.6, 128.3, 127.0, 126.4, 126.1, 125.4, 125.2, 122.7, 120.2, 119.9, 119.7, 116.4, 21.5. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{26}\text{H}_{21}\text{N}_2\text{O}_3$: 409.1552; found: 409.1547. FT-IR (neat, cm^{-1}) ν 3343, 2925, 1737, 1684, 1531, 1498, 1110, 753.



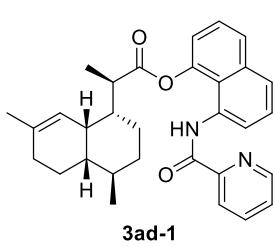
8-(picolinamido)naphthalen-1-yl 3-(4-methoxyphenyl)acrylate (3ac-5**).** Following the general procedure, **3ac-5** was obtained as a yellow solid (67.8 mg, 80%). $R_f = 0.30$ (n-hexane/EtOAc 3:1). m.p. 149.0–151.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.51 (s, 1H), 8.67 (d, $J = 8.0$ Hz, 1H), 8.42 (d, $J = 4.8$ Hz, 1H), 8.27 (d, $J = 7.6$ Hz, 1H), 7.81–7.73 (m, 3H), 7.70 (d, $J = 8.0$ Hz, 1H), 7.54 (t, $J = 8.0$ Hz, 1H), 7.48 (t, $J = 8.0$ Hz, 1H), 7.39 (d, $J = 8.4$ Hz, 2H), 7.27–7.21 (m, 2H), 6.90 (d, $J = 8.4$ Hz, 2H), 6.66 (d, $J = 15.6$ Hz, 1H), 3.86 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 165.9, 162.3, 161.8, 150.2, 147.7, 146.6, 146.3, 137.4, 136.4, 132.4, 130.0, 127.0, 126.7, 126.4, 126.1, 125.4, 125.1, 122.6, 120.2, 119.9, 119.7, 114.9, 114.3, 55.4. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{26}\text{H}_{21}\text{N}_2\text{O}_4$: 425.1501; found: 425.1496. FT-IR (neat, cm^{-1}) ν 3341, 2927, 1735, 1684, 1600, 1532, 1510, 1108, 757.



8-(picolinamido)naphthalen-1-yl
3-(4-(trifluoromethyl)phenyl)acrylate (3ac-6). Following the general procedure, **3ac-6** was obtained as a white solid (42.5 mg, 46%). $R_f = 0.70$ (n-hexane/EtOAc 3:1). m.p. 209.0–211.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.30 (s, 1H), 8.59 (d, $J = 7.6$ Hz, 1H), 8.41 (d, $J = 4.8$ Hz, 1H), 8.29 (d, $J = 7.6$ Hz, 1H), 7.82–7.71 (m, 4H), 7.63 (d, $J = 8.0$ Hz, 2H), 7.55 (t, $J = 8.0$ Hz, 1H), 7.52–7.46 (m, 3H), 7.27 (d, $J = 8.0$ Hz, 1H), 7.25–7.21 (m, 1H), 6.84 (d, $J = 16.0$ Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 165.0, 162.2, 150.2, 147.6, 146.0, 144.8, 137.6, 137.4, 137.3, 136.5, 132.2 (q, $J_{\text{C}-\text{F}} = 32.5$ Hz), 132.1, 128.3, 127.3, 126.5, 126.1, 125.8 (q, $J_{\text{C}-\text{F}} = 3.8$ Hz), 125.5, 125.4, 123.7 (q, $J_{\text{C}-\text{F}} = 270.5$ Hz), 122.8, 120.6, 120.2, 119.8. ^{19}F NMR (376 MHz, CDCl_3) δ -62.9. HRMS (ESI): m/z [M+H]⁺ calcd for $\text{C}_{26}\text{H}_{18}\text{F}_3\text{N}_2\text{O}_3$: 463.1270; found: 463.1264. FT-IR (neat, cm^{-1}) ν 3350, 2922, 1737, 1683, 1532, 1500, 1324, 1114, 750.

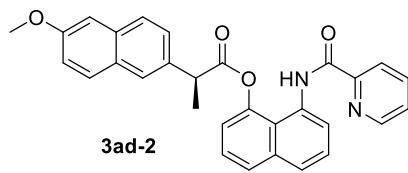


8-(picolinamido)naphthalen-1-yl 3-(2-bromophenyl)acrylate
(3ac-7). Following the general procedure, **3ac-7** was obtained as a yellow solid (62.3 mg, 66%). $R_f = 0.48$ (n-hexane/EtOAc 3:1). m.p. 151.0–153.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.35 (s, 1H), 8.61 (d, $J = 7.6$ Hz, 1H), 8.38 (d, $J = 4.4$ Hz, 1H), 8.30 (d, $J = 7.6$ Hz, 1H), 8.15 (d, $J = 15.6$ Hz, 1H), 7.82–7.71 (m, 3H), 7.64–7.59 (m, 1H), 7.55 (t, $J = 8.0$ Hz, 1H), 7.49 (t, $J = 8.0$ Hz, 1H), 7.39–7.34 (m, 1H), 7.31–7.25 (m, 3H), 7.21–7.15 (m, 1H), 6.73 (d, $J = 16.0$ Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 164.9, 162.3, 150.2, 147.6, 146.1, 145.1, 137.5, 136.4, 134.1, 133.6, 132.2, 131.6, 127.7, 127.6, 127.2, 126.4, 126.0, 125.6, 125.37, 125.36, 122.8, 120.4, 120.3, 120.2, 119.8. HRMS (ESI): m/z [M+H]⁺ calcd for $\text{C}_{25}\text{H}_{18}\text{BrN}_2\text{O}_3$: 473.0501; found: 473.0495. FT-IR (neat, cm^{-1}) ν 3341, 2922, 1735, 1680, 1527, 1494, 1114, 750.



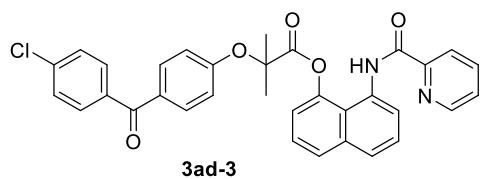
8-(picolinamido)naphthalen-1-yl
(R)-2-((1R,4R,4aS,8aS)-4,7-dimethyl-1,2,3,4,4a,5,6,8a-octahydronaphthalen-1-yl)propanoate (3ad-1). Following the general procedure, **3ad-1** was obtained as a white solid (74.2 mg, 77%). $R_f = 0.84$ (n-hexane/EtOAc 3:1). m.p. 154.0–156.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.6 (s, 1H), 8.79 (d, $J = 7.6$ Hz, 1H), 8.77 (d, $J = 4.4$ Hz, 1H), 8.38 (d, $J = 7.6$ Hz, 1H), 7.95 (dt, $J = 7.6, 1.2$ Hz, 1H), 7.75 (d, $J = 7.6$ Hz, 1H), 7.67 (d, $J = 8.0$ Hz, 1H), 7.57–7.49 (m, 2H), 7.46 (t, $J = 8.0$ Hz, 1H), 7.20 (dd, $J = 7.6, 0.8$ Hz, 1H), 5.03 (s, 1H), 3.16–3.06 (m, 1H), 2.45 (s, 1H), 1.97–1.72 (m, 4H), 1.68 (s, 3H), 1.58–1.44 (m, 3H), 1.38–1.29 (m, 1H), 1.24–1.19 (m, 1H), 1.09 (d, $J = 7.2$ Hz, 3H), 0.97–0.87 (m, 2H), 0.84 (d, $J = 6.4$ Hz, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 176.3, 162.1, 150.6, 147.8, 145.8, 137.8, 136.4, 136.0, 132.6, 127.0, 126.5, 126.4, 125.2, 124.8, 123.0, 120.5, 119.5, 119.2, 119.0, 44.1, 41.8, 41.5, 36.5, 35.1, 27.6, 27.4, 26.5, 25.6, 23.9, 19.6, 15.0. HRMS (ESI): m/z [M+H]⁺

calcd for C₃₁H₃₅N₂O₃: 483.2648; found: 483.2642. FT-IR (neat, cm⁻¹) ν 3342, 2918, 1764, 1686, 1532, 1498, 1105, 1040, 753.



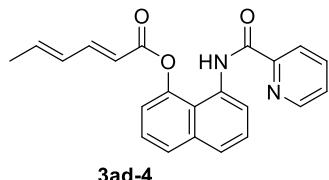
8-(picolinamido)naphthalen-1-yl
(*S*)-2-(6-methoxynaphthalen-2-yl)propanoate
(3ad-2). Following the general procedure, **3ad-2** was obtained as a white solid (87.6 mg, 92%). R_f = 0.50 (n-hexane/EtOAc 3:1). m.p. 146.0–148.0 °C.

¹H NMR (400 MHz, CDCl₃) δ 11.58 (s, 1H), 8.84–8.71 (m, 2H), 8.41 (d, J = 7.6 Hz, 1H), 7.97 (dt, J = 7.6, 1.6 Hz, 1H), 7.79–7.64 (m, 5H), 7.59–7.51 (m, 3H), 7.31 (t, J = 8.0 Hz, 1H), 7.21–7.12 (m, 2H), 6.77 (dd, J = 7.6, 1.2 Hz, 1H), 4.67 (q, J = 7.2 Hz, 1H), 3.93 (s, 3H), 1.53 (d, J = 7.2 Hz, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 173.4, 162.2, 157.8, 150.5, 147.6, 146.3, 137.9, 136.3, 134.6, 133.9, 132.4, 129.3, 129.0, 127.5, 127.1, 126.7, 126.4, 126.2, 126.1, 125.2, 125.0, 123.1, 119.7, 119.5, 119.3, 119.2, 105.6, 55.3, 45.1, 18.0. HRMS (ESI): m/z [M+H]⁺ calcd for C₃₀H₂₅N₂O₄: 477.1814; found: 477.1809. FT-IR (neat, cm⁻¹) ν 3355, 2933, 1763, 1685, 1531, 1497, 1109, 753.



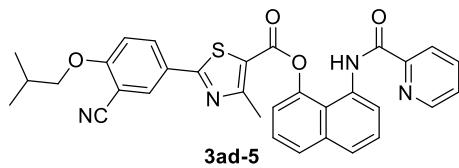
8-(picolinamido)naphthalen-1-yl
2-(4-(4-chlorobenzoyl)phenoxy)-2-methylpropionate
(3ad-3). Following the general procedure, **3ad-3** was obtained as a yellow solid (83.5 mg, 74%). R_f = 0.48

(n-hexane/EtOAc 3:1). m.p. 163.0–165.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 10.82 (s, 1H), 8.67 (d, J = 4.4 Hz, 1H), 8.51 (d, J = 7.6 Hz, 1H), 8.27 (d, J = 7.6 Hz, 1H), 7.91 (t, J = 7.6 Hz, 1H), 7.79 (d, J = 8.0 Hz, 1H), 7.72–7.65 (m, 5H), 7.55–7.43 (m, 5H), 7.14 (d, J = 7.2 Hz, 1H), 6.86 (d, J = 8.8 Hz, 2H), 1.68 (s, 6H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 194.2, 172.8, 162.6, 159.0, 150.1, 147.9, 146.0, 138.5, 137.7, 136.5, 136.2, 132.0, 131.9, 131.2, 131.1, 128.6, 127.6, 126.6, 126.5, 125.5, 125.2, 122.9, 120.4, 120.2, 120.0, 118.5, 79.9, 25.2. HRMS (ESI): m/z [M-H]⁻ calcd for C₃₃H₂₄ClN₂O₅: 563.1374; found: 563.1379. FT-IR (neat, cm⁻¹) ν 3341, 2923, 1737, 1652, 1594, 1543, 1504, 1251, 1145, 758.

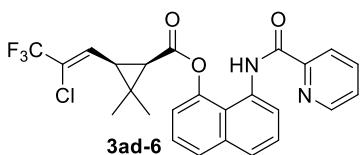


8-(Picolinamido)naphthalen-1-yl
(2*E*, 4*E*)-hexa-2,4-dienoate
(3ad-4). Following the general procedure, **3ad-4** was obtained as a yellow solid (27.2 mg, 38%). R_f = 0.50 (n-hexane/EtOAc 3:1). m.p. 145.0–147.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 11.56 (s, 1H), 8.74 (d, J = 7.6 Hz, 1H), 8.56 (d, J = 4.0 Hz, 1H), 8.31 (d, J = 7.6 Hz, 1H), 7.87 (t, J = 7.6 Hz, 1H), 7.75 (d, J = 8.0 Hz, 1H), 7.68 (d, J = 8.0 Hz, 1H), 7.53 (t, J = 8.0 Hz, 1H), 7.48–7.36 (m, 3H), 7.21 (d, J = 7.6 Hz, 1H), 6.26–6.10 (m, 3H), 1.87 (d, J = 4.8 Hz, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 165.9, 162.3, 150.3, 147.6, 147.3, 146.3,

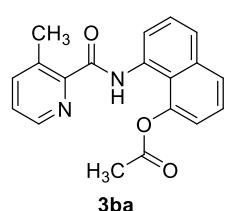
141.3, 137.5, 136.4, 132.6, 129.4, 126.9, 126.4, 126.2, 125.3, 125.0, 122.7, 120.1, 119.50, 119.46, 118.4, 18.8. HRMS (ESI): m/z [M+H]⁺ calcd for C₂₂H₁₉N₂O₃: 359.1396; found: 359.1390. FT-IR (neat, cm⁻¹) ν 3332, 2923, 1724, 1677, 1526, 1494, 1111, 1000, 740.



8-(Picolinamido)naphthalen-1-yl
2-(3-cyano-4-isobutoxyphenyl)-4-methylthiazole-5-carboxylate (**3ad-5**). Following the general procedure, **3ad-5** was obtained as a yellow solid (30.7 mg, 28%). R_f = 0.23 (n-hexane/EtOAc 3:1). m.p. 190.0–192.0 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.7 (s, 1H), 8.19 (d, *J* = 2.4 Hz, 1H), 8.10 (dd, *J* = 8.8, 2.4 Hz, 1H), 8.08-8.04 (m, 1H), 8.02-7.94 (m, 3H), 7.87 (d, *J* = 7.2 Hz, 1H), 7.78 (dt, *J* = 7.8, 1.6 Hz, 1H), 7.65-7.56 (m, 2H), 7.14 (dd, *J* = 7.2, 1.2 Hz, 1H), 7.37 (d, *J* = 8.8, 1H), 7.26-7.21 (m, 1H), 4.02 (d, *J* = 6.8 Hz, 2H), 2.55 (s, 3H), 2.16-2.06 (m, 1H), 1.04 (d, *J* = 6.8 Hz, 6H). ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 168.1, 163.0, 162.7, 162.1, 160.8, 149.6, 147.9, 145.9, 138.0, 136.4, 133.7, 132.1, 132.0, 128.0, 127.1, 126.8, 126.7, 126.3, 125.4, 125.0, 122.6, 122.3, 121.3, 121.2, 115.8, 114.4, 102.1, 75.6, 28.1, 19.2, 17.8. HRMS (ESI): m/z [M+H]⁺ calcd for C₃₂H₂₇N₄O₄S: 563.1753; found: 563.1748. FT-IR (neat, cm⁻¹) ν 3356, 2928, 1731, 1686, 1531, 1500, 1430, 1289, 1246, 1030, 753.

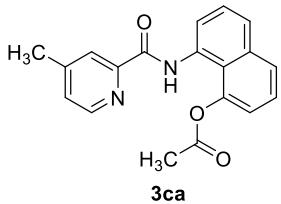


8-(picolinamido)naphthalen-1-yl
(1*R*,3*R*)-3-((*Z*)-2-chloro-3,3,3-trifluoroprop-1-en-1-yl)-2,2-dimethylcyclopropane-1-carboxylate (**3ad-6**). Following the general procedure, **3ad-6** was obtained as a white solid (76.1 mg, 78%). R_f = 0.64 (n-hexane/EtOAc 3:1). m.p. 123.0–125.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 11.46 (s, 1H), 8.69 (d, *J* = 7.6 Hz, 1H), 8.64 (d, *J* = 4.4 Hz, 1H), 8.40 (d, *J* = 7.6 Hz, 1H), 7.96 (t, *J* = 7.6 Hz, 1H), 7.78 (d, *J* = 8.4 Hz, 1H), 7.70 (d, *J* = 8.4 Hz, 1H), 7.57-7.51 (m, 2H), 7.47 (t, *J* = 8.4 Hz, 1H), 7.18 (d, *J* = 7.6 Hz, 1H), 6.76 (d, *J* = 9.2 Hz, 1H), 2.57 (d, *J* = 8.0 Hz, 1H), 1.96 (t, *J* = 8.4 Hz, 1H), 1.22 (s, 3H), 1.04 (s, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 168.9, 161.9, 150.3, 147.7, 145.6, 137.9, 136.4, 132.3, 128.9 (q, J_{C-F} = 4.4 Hz), 127.3, 126.6, 126.5, 125.2, 125.1, 123.1, 122.4 (q, J_{C-F} = 37.6 Hz), 120.6, 120.1 (q, J_{C-F} = 269.8 Hz), 119.7, 119.4, 32.6, 31.8, 29.9, 27.9, 14.6. ¹⁹F NMR (376 MHz, CDCl₃) δ -68.8. HRMS (ESI): m/z [M+H]⁺ calcd for C₂₅H₂₁ClF₃N₂O₃: 489.1193; found: 489.1187. FT-IR (neat, cm⁻¹) ν 3348, 2932, 1751, 1687, 1532, 1499, 1275, 1115, 1059, 753.

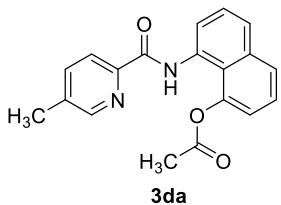


8-(3-methylpicolinamido)naphthalen-1-yl acetate (**3ba**). Following the general procedure, **3ba** was obtained as a white solid (31.4 mg, 49%). R_f = 0.50 (n-hexane/EtOAc 3:1). m.p. 147.0–149.0 °C. ¹H

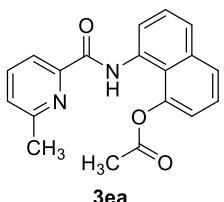
NMR (400 MHz, CDCl₃) δ 11.49 (s, 1H), 8.65 (d, *J* = 7.6 Hz, 1H), 8.50 (d, *J* = 4.0 Hz, 1H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 2H), 7.52 (t, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.39 (dd, *J* = 7.6, 4.8 Hz, 1H), 7.17 (dd, *J* = 7.6, 0.8 Hz, 1H), 2.85 (s, 3H), 2.33 (s, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 169.5, 163.8, 147.4, 145.9, 145.1, 141.4, 136.5, 136.4, 132.6, 127.2, 126.4, 126.1, 125.2, 124.9, 120.3, 119.63, 119.57, 21.5, 21.0. HRMS (ESI): m/z [M+H]⁺ calcd for C₁₉H₁₇N₂O₃: 321.1239; found: 321.1234. FT-IR (neat, cm⁻¹) ν 3346, 2924, 1769, 1682, 1524, 1494, 1430, 1081, 759.



8-(4-methylpicolinamido)naphthalen-1-yl acetate (**3ca**). Following the general procedure, **3ca** was obtained as a white solid (39.7 mg, 62%). R_f = 0.48 (n-hexane/EtOAc 3:1). m.p. 159.0–161.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 11.51 (s, 1H), 8.76 (dd, *J* = 8.0, 0.8 Hz, 1H), 8.49 (d, *J* = 4.8 Hz, 1H), 8.19 (s, 1H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.53 (t, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 4.8 Hz, 1H), 7.19 (dd, *J* = 7.6, 1.2 Hz, 1H), 2.48 (s, 3H), 2.45 (s, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 169.6, 162.5, 150.0, 149.3, 147.4, 145.9, 136.4, 132.5, 127.4, 127.2, 126.4, 125.2, 124.9, 123.7, 120.4, 119.4, 119.2, 21.6, 21.2. HRMS (ESI): m/z [M+H]⁺ calcd for C₁₉H₁₇N₂O₃: 321.1239; found: 321.1234. FT-IR (neat, cm⁻¹) ν 3337, 2934, 1763, 1671, 1528, 1496, 1432, 1179, 1024, 759.

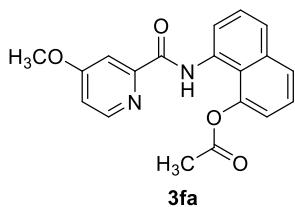


8-(5-methylpicolinamido)naphthalen-1-yl acetate (**3da**). Following the general procedure, **3da** was obtained as a white solid (46.7 mg, 73%). R_f = 0.46 (n-hexane/EtOAc 3:1). m.p. 155.0–157.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 11.46 (s, 1H), 8.77 (d, *J* = 7.6 Hz, 1H), 8.45 (s, 1H), 8.25 (d, *J* = 8.0 Hz, 1H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.72 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.52 (t, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.19 (d, *J* = 7.6 Hz, 1H), 2.46 (s, 3H), 2.44 (s, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 169.6, 162.5, 148.1, 147.9, 145.9, 138.1, 136.9, 136.4, 132.6, 127.2, 126.4, 125.2, 124.9, 122.5, 120.3, 119.3, 119.1, 21.7, 18.6. HRMS (ESI): m/z [M+H]⁺ calcd for C₁₉H₁₇N₂O₃: 321.1239; found: 321.1234. FT-IR (neat, cm⁻¹) ν 3331, 2922, 1766, 1673, 1530, 1499, 1185, 757.

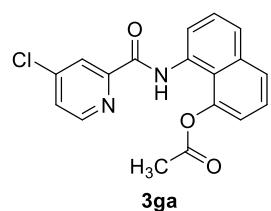


8-(6-methylpicolinamido)naphthalen-1-yl acetate (**3ea**). Following the general procedure, **3ea** was obtained as a gray solid (14.7 mg, 23%). R_f = 0.46 (n-hexane/EtOAc 3:1). m.p. 168.0–170.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 11.19 (s, 1H), 8.53 (d, *J* = 7.6 Hz, 1H), 8.19 (d, *J* = 7.6 Hz, 1H), 7.83 (t, *J* = 7.6 Hz, 1H), 7.78 (d, *J* = 7.6 Hz, 1H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.53 (t, *J* = 8.0 Hz, 1H), 7.47 (t, *J* = 8.0 Hz, 1H), 7.38 (d, *J* = 7.6 Hz, 1H), 7.19 (dd, *J* = 7.2, 0.8 Hz, 1H), 2.69 (s, 3H),

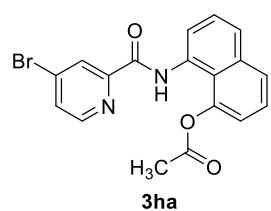
2.22 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.4, 162.5, 157.0, 149.6, 145.8, 138.0, 136.4, 132.0, 127.3, 126.4, 125.4, 125.3, 120.6, 120.5, 120.2, 120.0, 24.2, 21.2. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{19}\text{H}_{17}\text{N}_2\text{O}_3$: 321.1239; found: 321.1234. FT-IR (neat, cm^{-1}) ν 3362, 2924, 1772, 1687, 1530, 1497, 1179, 746.



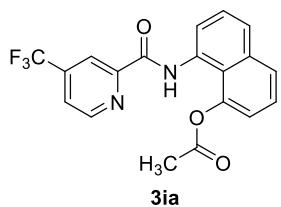
8-(4-methoxypicolinamido)naphthalen-1-yl acetate (**3fa**). Following the general procedure, **3fa** was obtained as a yellow solid (52.4 mg, 78%). $R_f = 0.40$ (n-hexane/EtOAc 3:1). m.p. 157.0–159.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.53 (s, 1H), 8.75 (dd, $J = 8.0, 1.2$ Hz, 1H), 8.43 (d, $J = 5.6$ Hz, 1H), 7.90 (d, $J = 2.8$ Hz, 1H), 7.76 (dd, $J = 8.0, 0.8$ Hz, 1H), 7.68 (d, $J = 8.0, 0.8$ Hz, 1H), 7.53 (t, $J = 8.0$ Hz, 1H), 7.45 (t, $J = 8.0$ Hz, 1H), 7.19 (dd, $J = 7.6, 1.2$ Hz, 1H), 7.00 (dd, $J = 5.6, 2.8$ Hz, 1H), 3.95 (s, 3H), 2.45 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.6, 167.3, 162.2, 152.3, 148.8, 145.9, 136.4, 132.4, 127.2, 126.4, 125.2, 125.0, 120.4, 119.4, 119.2, 113.4, 107.9, 55.6, 21.7. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{19}\text{H}_{17}\text{N}_2\text{O}_4$: 337.1188; found: 337.1183. FT-IR (neat, cm^{-1}) ν 3342, 2927, 1772, 1682, 1529, 1496, 1180, 1030, 758.



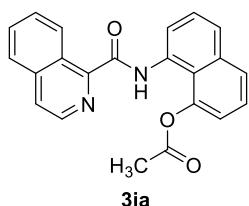
8-(4-chloropicolinamido)naphthalen-1-yl acetate (**3ga**). Following the general procedure, **3ga** was obtained as a yellow solid (48.3 mg, 71%). $R_f = 0.72$ (n-hexane/EtOAc 3:1). m.p. 184.0–186.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.44 (s, 1H), 8.75 (d, $J = 7.6$ Hz, 1H), 8.53 (d, $J = 5.2$ Hz, 1H), 8.37 (d, $J = 2.0$ Hz, 1H), 7.76 (d, $J = 8.0$ Hz, 1H), 7.69 (d, $J = 8.0$ Hz, 1H), 7.55–7.50 (m, 2H), 7.46 (t, $J = 8.0$ Hz, 1H), 7.20 (dd, $J = 7.6, 0.8$ Hz, 1H), 2.44 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.4, 161.1, 151.8, 148.6, 146.5, 145.8, 136.4, 132.2, 127.3, 126.8, 126.5, 125.4, 125.3, 123.6, 120.5, 119.5, 119.1, 21.7. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{18}\text{H}_{14}\text{ClN}_2\text{O}_3$: 341.0693; found: 341.0687. FT-IR (neat, cm^{-1}) ν 3350, 2930, 1763, 1672, 1542, 1501, 1194, 1027, 754.



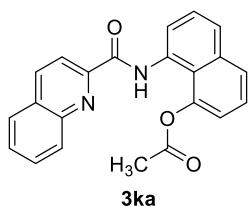
8-(4-bromopicolinamido)naphthalen-1-yl acetate (**3ha**). Following the general procedure, **3ha** was obtained as a yellow solid (55.4 mg, 72%). $R_f = 0.72$ (n-hexane/EtOAc 3:1). m.p. 178.0–180.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.42 (s, 1H), 8.74 (d, $J = 7.6$ Hz, 1H), 8.53 (d, $J = 1.6$ Hz, 1H), 8.44 (d, $J = 4.8$ Hz, 1H), 7.76 (d, $J = 8.4$ Hz, 1H), 7.71–7.65 (m, 2H), 7.53 (t, $J = 8.0$ Hz, 1H), 7.46 (t, $J = 8.0$ Hz, 1H), 7.19 (d, $J = 7.6$ Hz, 1H), 2.44 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.4, 160.9, 151.4, 148.3, 145.7, 136.3, 135.0, 132.1, 129.8, 127.2, 126.6, 126.4, 125.32, 125.30, 120.5, 119.5, 119.0, 21.7. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{18}\text{H}_{14}\text{BrN}_2\text{O}_3$: 385.0188; found: 385.0182. FT-IR (neat, cm^{-1}) ν 3346, 2952, 1759, 1668, 1538, 1497, 1189, 1024, 753.



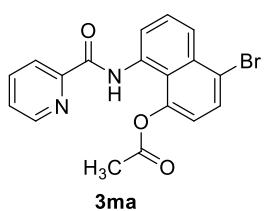
8-(4-(trifluoromethyl)picolinamido)naphthalen-1-yl acetate (**3ia**). Following the general procedure, **3ia** was obtained as a gray solid (53.2 mg, 78%). $R_f = 0.75$ (n-hexane/EtOAc 3:1). m.p. 170.0–172.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.47 (s, 1H), 8.84–8.80 (m, 1H), 8.78 (dd, $J = 8.0, 1.2$ Hz, 1H), 6.62 (s, 1H), 7.78–7.73 (m, 2H), 7.70 (d, $J = 8.0$ Hz, 1H), 7.54 (t, $J = 8.0$ Hz, 1H), 7.47 (t, $J = 8.0$ Hz, 1H), 7.21 (d, $J = 7.6$ Hz, 1H), 2.45 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.3, 160.8, 151.9, 148.7, 145.7, 140.4 (q, $J = 34.8$ Hz), 136.4, 132.0, 127.3, 126.4, 125.45, 125.36, 122.4 (q, $J = 271.9$ Hz), 122.2 (q, $J = 3.4$ Hz), 125.5, 119.5, 119.1 (q, $J = 3.5$ Hz), 119.0, 21.7. ^{19}F NMR (376 MHz, CDCl_3) δ -64.8. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{19}\text{H}_{14}\text{F}_3\text{N}_2\text{O}_3$: 375.0957; found: 375.0951. FT-IR (neat, cm^{-1}) ν 3348, 2924, 1774, 1685, 1534, 1501, 1408, 1330, 1174, 1139, 757.



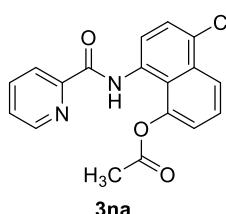
8-(isoquinoline-1-carboxamido)naphthalen-1-yl acetate (**3ja**). Following the general procedure, **3ja** was obtained as a yellow solid (62.7 mg, 88%). $R_f = 0.70$ (n-hexane/EtOAc 3:1). m.p. 159.0–161.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.64 (s, 1H), 9.80–9.70 (m, 1H), 8.73 (dd, $J = 8.0, 1.2, 1$ H), 8.58 (d, $J = 5.6$ Hz, 1H), 7.94–7.87 (m, 2H), 7.80–7.69 (m, 4H), 7.57 (t, $J = 8.0$ Hz, 1H), 7.47 (t, $J = 8.0$ Hz, 1H), 7.20 (dd, $J = 7.8, 1.2$ Hz, 1H), 2.30 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.5, 163.9, 148.2, 145.9, 139.6, 137.6, 136.4, 132.5, 130.8, 129.1, 127.9, 127.33, 127.25, 127.0, 126.4, 125.3, 125.15, 125.08, 120.4, 119.8, 119.6, 21.5. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{22}\text{H}_{17}\text{N}_2\text{O}_3$: 357.1239; found: 357.1234. FT-IR (neat, cm^{-1}) ν 3338, 2928, 1768, 1676, 1524, 1491, 1181, 754.



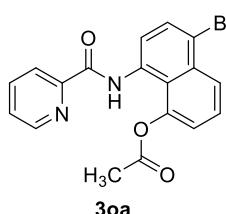
8-(quinoline-2-carboxamido)naphthalen-1-yl acetate (**3ka**). Following the general procedure, **3ka** was obtained as a white solid (14.2 mg, 20%). $R_f = 0.68$ (n-hexane/EtOAc 3:1). m.p. 184.0–186.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.51 (s, 1H), 8.66 (d, $J = 7.6$ Hz, 1H), 8.49 (d, $J = 8.4$ Hz, 1H), 8.42 (d, $J = 8.4$ Hz, 1H), 8.26 (d, $J = 8.4$ Hz, 1H), 7.96 (d, $J = 8.4$ Hz, 1H), 7.87–7.82 (m, 1H), 7.80 (d, $J = 8.0$ Hz, 1H), 7.73–7.66 (m, 2H), 7.56 (t, $J = 8.0$ Hz, 1H), 7.49 (t, $J = 8.0$ Hz, 1H), 7.22 (dd, $J = 7.2, 0.8, 1$ H), 2.21 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.5, 162.3, 150.2, 146.1, 145.9, 138.0, 136.4, 132.2, 130.6, 129.5, 129.4, 128.2, 128.1, 127.3, 126.5, 125.4, 125.3, 120.6, 120.2, 119.8, 119.2, 21.5. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{22}\text{H}_{17}\text{N}_2\text{O}_3$: 357.1239; found: 357.1234. FT-IR (neat, cm^{-1}) ν 3325, 2924, 1768, 1682, 1529, 1493, 1184, 758.



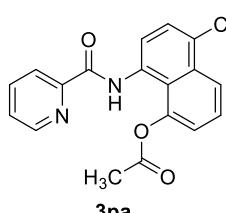
4-bromo-8-(picolinamido)naphthalen-1-yl acetate (**3ma**). Following the general procedure, **3ma** was obtained as a white solid (62.4 mg, 81%). $R_f = 0.50$ (n-hexane/EtOAc 3:1). m.p. 161.0–163.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.51 (s, 1H), 8.82 (dd, $J = 8.0, 1.2$ Hz, 1H), 8.67–8.62 (m, 1H), 8.37 (d, $J = 7.6$ Hz, 1H), 8.14 (dd, $J = 8.4, 0.8$ Hz, 1H), 7.95 (dt, $J = 7.6, 1.6$ Hz, 1H), 7.79 (d, $J = 8.4$ Hz, 1H), 7.65 (t, $J = 8.0$ Hz, 1H), 7.55–7.50 (m, 1H), 7.06 (d, $J = 8.0$ Hz, 1H), 2.43 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.2, 162.3, 150.1, 147.7, 145.6, 137.9, 134.1, 132.9, 129.5, 127.9, 126.7, 124.5, 123.0, 121.0, 120.6, 120.5, 120.4, 21.5. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{18}\text{H}_{14}\text{BrN}_2\text{O}_3$: 385.0188; found: 385.0182. FT-IR (neat, cm^{-1}) ν 3337, 2923, 1770, 1685, 1533, 1493, 1174, 741.



5-chloro-8-(picolinamido)naphthalen-1-yl acetate (**3na**). Following the general procedure, **3na** was obtained as a white solid (37.4 mg, 55%). $R_f = 0.50$ (n-hexane/EtOAc 3:1). m.p. 169.0–171.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.54 (s, 1H), 8.73 (d, $J = 8.4$ Hz, 1H), 8.64 (d, $J = 4.8$ Hz, 1H), 8.35 (d, $J = 8.0$ Hz, 1H), 8.26 (d, $J = 8.4$ Hz, 1H), 7.94 (dt, $J = 7.6, 1.2$ Hz, 1H), 7.63 (d, $J = 8.4$ Hz, 1H), 7.58 (t, $J = 8.4$ Hz, 1H), 7.54–7.49 (m, 1H), 7.26 (d, $J = 7.6$ Hz, 1H), 2.45 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.3, 162.2, 150.0, 147.6, 146.1, 137.9, 133.1, 131.9, 127.8, 126.9, 126.7, 126.3, 123.6, 123.0, 121.4, 120.3, 119.0, 21.6. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{18}\text{H}_{14}\text{ClN}_2\text{O}_3$: 341.0693; found: 341.0687. FT-IR (neat, cm^{-1}) ν 3348, 2922, 1755, 1686, 1531, 1499, 1198, 750.

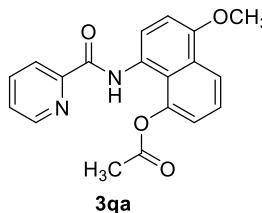


5-bromo-8-(picolinamido)naphthalen-1-yl acetate (**3oa**). Following the general procedure, **3oa** was obtained as a white solid (67.0 mg, 87%). $R_f = 0.50$ (n-hexane/EtOAc 3:1). m.p. 177.0–179.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.58 (s, 1H), 8.69 (d, $J = 8.4$ Hz, 1H), 8.66 (d, $J = 4.4$ Hz, 1H), 8.36 (d, $J = 8.0$ Hz, 1H), 8.26 (dd, $J = 8.4, 0.8$ Hz, 1H), 7.96 (dt, $J = 7.6, 1.6$ Hz, 1H), 7.84 (d, $J = 8.4$ Hz, 1H), 7.58 (t, $J = 7.6$ Hz, 1H), 7.55–7.51 (m, 1H), 7.27 (dd, $J = 7.6, 0.8$ Hz, 1H), 2.46 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.3, 162.2, 150.0, 147.7, 146.0, 137.9, 134.1, 132.6, 130.7, 126.8, 126.6, 126.5, 123.0, 121.4, 120.4, 119.5, 118.4, 21.6. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{18}\text{H}_{14}\text{BrN}_2\text{O}_3$: 385.0188; found: 385.0182. FT-IR (neat, cm^{-1}) ν 3339, 2927, 1771, 1683, 1522, 1493, 1176, 750.

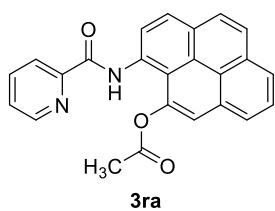


5-methyl-8-(picolinamido)naphthalen-1-yl acetate (**3pa**). Following the general procedure, **3pa** was obtained as a white

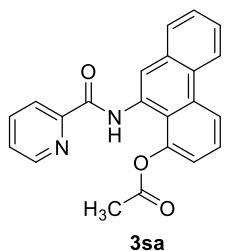
solid (55.0 mg, 86%). R_f = 0.48 (n-hexane/EtOAc 3:1). m.p. 154.0–156.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.40 (s, 1H), 8.64 (d, J = 4.4 Hz, 1H), 8.60 (d, J = 7.6 Hz, 1H), 8.36 (d, J = 8.0 Hz, 1H), 8.00–7.89 (m, 2H), 7.53–7.47 (m, 2H), 7.39 (d, J = 8.0 Hz, 1H), 7.20 (d, J = 8.0 Hz, 1H), 2.67 (s, 3H), 2.41 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.6, 162.1, 150.3, 147.6, 146.3, 137.7, 135.2, 131.1, 130.7, 127.3, 126.5, 125.0, 123.2, 122.8, 120.2, 119.6, 119.5, 21.6, 20.0. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{19}\text{H}_{17}\text{N}_2\text{O}_3$: 321.1239; found: 321.1234. FT-IR (neat, cm^{-1}) ν 3335, 2925, 1764, 1676, 1528, 1505, 1181, 747.



5-methoxy-8-(picolinamido)naphthalen-1-yl acetate (3qa**).** Following the general procedure, **3qa** was obtained as a yellow solid (55.1 mg, 82%). R_f = 0.37 (n-hexane/EtOAc 3:1). m.p. 138.0–140.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.14 (s, 1H), 8.66–8.59 (m, 1H), 8.51 (d, J = 8.8 Hz, 1H), 8.35 (d, J = 7.6 Hz, 1H), 8.26 (dd, J = 8.8, 1.6 Hz, 1H), 7.91 (dd, J = 7.6, 1.6 Hz, 1H), 7.51–7.42 (m, 2H), 7.22 (d, J = 7.6, 1.2 Hz, 1H), 6.88 (d, J = 8.4 Hz, 1H), 4.00 (s, 3H), 2.36 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.6, 162.1, 152.6, 150.4, 147.6, 145.7, 137.7, 128.0, 126.4, 124.9, 124.7, 122.7, 121.2, 120.92, 120.87, 120.7, 104.2, 55.7, 21.5. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{19}\text{H}_{17}\text{N}_2\text{O}_4$: 337.1188; found: 337.1183. FT-IR (neat, cm^{-1}) ν 3350, 2933, 1763, 1670, 1538, 1506, 1185, 1038, 749.

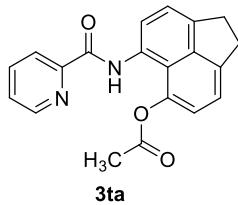


3-(picolinamido)pyren-4-yl acetate (3ra**).** Following the general procedure, **3ra** was obtained as a yellow solid (62.3 mg, 82%). R_f = 0.26 (n-hexane/EtOAc 3:1). m.p. 214.0–216.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.75 (s, 1H), 9.25 (d, J = 8.8 Hz, 1H), 8.70 (d, J = 4.4 Hz, 1H), 8.42 (d, J = 7.6 Hz, 1H), 8.24 (d, J = 8.8 Hz, 1H), 8.13 (d, J = 7.6 Hz, 1H), 8.07 (d, J = 7.2 Hz, 1H), 8.04–7.92 (m, 4H), 7.76 (s, 1H), 7.56–7.09 (m, 1H), 2.49 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.6, 162.3, 150.3, 147.8, 145.0, 137.9, 131.6, 131.3, 130.4, 128.5, 127.6, 127.1, 126.7, 126.6, 126.5, 125.3, 124.4, 123.6, 123.1, 120.8, 120.5, 116.1, 21.8. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{24}\text{H}_{17}\text{N}_2\text{O}_3$: 381.1239; found: 381.1234. FT-IR (neat, cm^{-1}) ν 3340, 2927, 1770, 1682, 1517, 1492, 1180, 746.

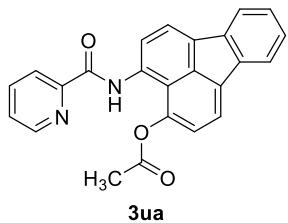


10-(picolinamido)phenanthren-1-yl acetate (3sa**).** Following the general procedure, **3sa** was obtained as a white solid (57.0 mg, 80%). R_f = 0.27 (n-hexane/EtOAc 3:1). m.p. 191.0–193.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.58 (s, 1H), 9.11 (s, 1H), 8.71–8.65 (m, 2H), 8.62–8.56 (m, 1H), 8.40 (d, J = 8.0 Hz, 1H), 7.95 (dt, J = 8.0, 1.2 Hz, 1H), 7.92–7.87 (m, 1H), 7.66 (t, J = 8.0 Hz, 1H), 7.63–7.57 (m, 2H), 7.55–7.50 (m, 1H), 7.32 (d, J = 7.6 Hz, 1H), 2.46 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.6, 162.5, 150.3, 147.7, 146.5, 137.9, 133.6, 131.9,

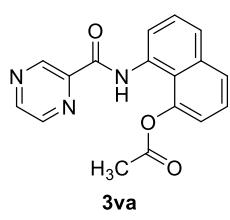
130.0, 128.6, 127.7, 127.5, 126.6, 126.4, 126.2, 122.9, 122.7, 121.7, 121.6, 119.3, 119.2, 21.6. HRMS (ESI): m/z [M+H]⁺ calcd for C₂₂H₁₇N₂O₃: 357.1239; found: 357.1234. FT-IR (neat, cm⁻¹) ν 3347, 2940, 1771, 1683, 1523, 1169, 755.



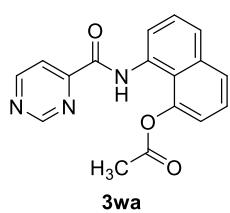
6-(picolinamido)-1,2-dihydroacenaphthylen-5-yl acetate (**3ta**). Following the general procedure, **3ta** was obtained as a white solid (35.5 mg, 53%). R_f = 0.30 (n-hexane/EtOAc 3:1). m.p. 161.0–163.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 11.28 (s, 1H), 8.74 (d, J = 8.0 Hz, 1H), 8.64 (d, J = 4.4 Hz, 1H), 8.37 (d, J = 8.0 Hz, 1H), 7.94 (dt, J = 7.6, 1.6 Hz, 1H), 7.53-7.49 (m, 1H), 7.33 (d, J = 8.0 Hz, 1H), 7.25 (d, J = 7.26 Hz, 1H), 7.16 (d, J = 7.6 Hz, 1H), 3.39 (s, 4H), 2.52 (s, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 169.9, 162.1, 150.4, 147.6, 144.4, 142.6, 142.1, 141.5, 137.8, 129.1, 126.4, 122.8, 121.4, 120.4, 120.1, 119.1, 117.3, 30.3, 30.1, 21.6. HRMS (ESI): m/z [M+H]⁺ calcd for C₂₀H₁₇N₂O₃: 333.1239; found: 333.1234. FT-IR (neat, cm⁻¹) ν 3335, 2929, 1767, 1671, 1528, 1498, 1179, 753.



4-(picolinamido)fluoranthen-3-yl acetate (**3ua**). Following the general procedure, **3ua** was obtained as a yellow solid (31.9 mg, 42%). R_f = 0.27 (n-hexane/EtOAc 3:1). m.p. 225.0–227.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 11.62 (s, 1H), 9.05 (d, J = 8.0 Hz, 1H), 8.64 (d, J = 4.8 Hz, 1H), 8.40 (d, J = 8.0 Hz, 1H), 8.00-7.83 (m, 5H), 7.56-7.51 (m, 1H), 7.40-7.31 (m, 3H), 2.64 (s, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 169.3, 162.3, 150.2, 147.6, 146.1, 139.1, 138.4, 137.9, 135.1, 134.1, 133.8, 132.6, 127.5, 127.1, 126.7, 123.0, 121.7, 121.3, 121.2, 121.1, 120.0, 119.0, 115.8, 21.8. HRMS (ESI): m/z [M+H]⁺ calcd for C₂₄H₁₇N₂O₃: 381.1239; found: 381.1234. FT-IR (neat, cm⁻¹) ν 3333, 2924, 1762, 1676, 1529, 1443, 1187, 1039, 750.

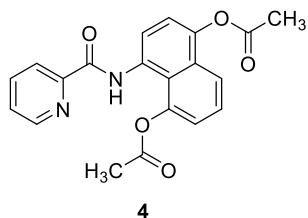


8-(pyrazine-2-carboxamido)naphthalen-1-yl acetate (**3va**). Following the general procedure, **3va** was obtained as a white solid (17.8 mg, 29%). R_f = 0.22 (n-hexane/EtOAc 3:1). m.p. 171.0–173.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 11.32 (s, 1H), 9.60 (s, 1H), 8.85 (d, J = 2.0 Hz, 1H), 8.78 (d, J = 8.0 Hz, 1H), 8.61 (s, 1H), 7.77 (d, J = 8.4 Hz, 1H), 7.71 (d, J = 8.4 Hz, 1H), 7.54 (t, J = 8.0 Hz, 1H), 7.47 (t, J = 8.0 Hz, 1H), 7.21 (d, J = 7.2 Hz, 1H), 2.46 (s, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 169.2, 160.8, 147.6, 145.7, 145.3, 144.9, 141.9, 136.4, 132.0, 127.3, 126.4, 125.5, 125.4, 120.6, 119.5, 118.9, 21.7. HRMS (ESI): m/z [M+H]⁺ calcd for C₁₇H₁₄N₃O₃: 308.1035; found: 308.1030. FT-IR (neat, cm⁻¹) ν 3337, 2925, 1764, 1680, 1534, 1498, 1174, 1020, 754.

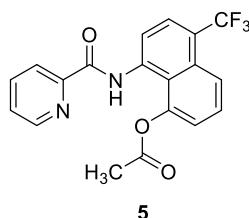


8-(pyrimidine-4-carboxamido)naphthalen-1-yl acetate (**3wa**). Following the general procedure, **3wa** was obtained as a yellow solid (35.6 mg, 58%). R_f = 0.22 (n-hexane/EtOAc 3:1). m.p.

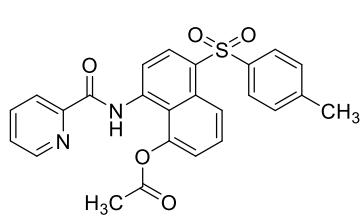
189.0–191.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.50 (s, 1H), 9.34 (s, 1H), 9.06 (d, J = 4.8 Hz, 1H), 8.77 (d, J = 8.0 Hz, 1H), 8.29 (dd, J = 4.8, 1.6 Hz, 1H), 7.77 (d, J = 8.0 Hz, 1H), 7.71 (d, J = 8.0 Hz, 1H), 7.53 (t, J = 7.6 Hz, 1H), 7.47 (t, J = 8.0 Hz, 1H), 7.22 (dd, J = 7.8, 1.6 Hz, 1H), 2.50 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.3, 160.4, 159.7, 157.4, 157.8, 145.7, 136.3, 131.8, 124.3, 126.4, 125.7, 125.5, 120.7, 119.5, 119.1, 118.9, 21.8. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{17}\text{H}_{14}\text{N}_3\text{O}_3$: 308.1035; found: 308.1030. FT-IR (neat, cm^{-1}) ν 3328, 2922, 1761, 1676, 1529, 1493, 1180, 756.



4-(picolinamido)naphthalene-1,5-diyI diacetate (**4**). Following the general procedure, **4** was obtained as a gray solid (29.8 mg, 41%). R_f = 0.23 (n-hexane/EtOAc 3:1). m.p. 163.0–165.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.51 (s, 1H), 8.78 (d, J = 8.4 Hz, 1H), 8.65 (d, J = 4.4 Hz, 1H), 8.37 (d, J = 7.6 Hz, 1H), 7.95 (t, J = 7.6 Hz, 1H), 7.83 (d, J = 8.4 Hz, 1H), 7.56–7.48 (m, 2H), 7.33 (d, J = 8.4 Hz, 1H), 7.25 (d, J = 7.2 Hz, 1H), 2.47 (s, 3H), 2.46 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.4, 169.3, 162.2, 150.2, 147.7, 146.2, 143.0, 137.9, 130.7, 129.4, 126.7, 125.9, 123.0, 121.1, 120.2, 120.0, 119.0, 118.9, 21.6, 21.0. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{20}\text{H}_{17}\text{N}_2\text{O}_5$: 365.1137; found: 365.1132. FT-IR (neat, cm^{-1}) ν 3345, 2926, 1761, 1678, 1530, 1502, 1177, 749.

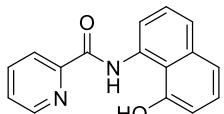


8-(picolinamido)-5-(trifluoromethyl)naphthalen-1-yl acetate (**5**). Following the general procedure, **5** was obtained as a gray solid (33.6 mg, 45%). R_f = 0.25 (n-hexane/EtOAc 3:1). m.p. 144.0–146.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 10.05 (s, 1H), 8.71 (d, J = 4.4 Hz, 1H), 8.35 (d, J = 7.6 Hz, 1H), 8.01–7.92 (m, 2H), 7.86 (d, J = 8.0 Hz, 1H), 7.78 (d, J = 8.8 Hz, 1H), 7.62–7.53 (m, 2H), 7.22 (d, J = 7.6 Hz, 1H), 1.79 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 170.1, 164.2, 149.0, 148.4, 146.7, 137.7, 137.6, 130.7 (q, J = 2.1 Hz), 129.0, 128.0, 127.4, 127.0, 126.3 (q, J = 28.9 Hz), 124.9, 123.7 (q, J = 272.3 Hz), 122.9, 122.2, 122.1 (q, J = 5.0 Hz), 20.6. ^{19}F NMR (376 MHz, CDCl_3) δ -60.6. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{19}\text{H}_{14}\text{F}_3\text{N}_2\text{O}_3$: 375.0957; found: 375.0951. FT-IR (neat, cm^{-1}) ν 3342, 2936, 1765, 1695, 1495, 1181, 1130, 752.

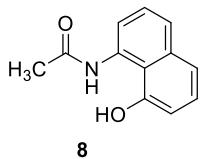


8-(picolinamido)-5-tosylnaphthalen-1-yl acetate (**6**). Following the general procedure, **6** was obtained as a gray solid (50.2 mg, 51%). R_f = 0.13 (n-hexane/EtOAc 3:1). m.p. 215.0–217.0 °C. ^1H NMR (400 MHz, CDCl_3)

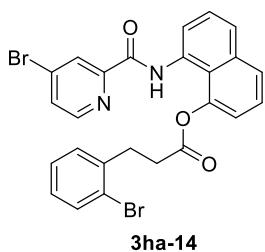
δ 11.95 (s, 1H), 9.06 (d, J = 8.8 Hz, 1H), 8.68-8.54 (m, 3H), 8.35 (d, J = 7.6 Hz, 1H), 7.95 (t, J = 7.6 Hz, 1H), 7.82 (d, J = 8.0 Hz, 2H), 7.58-7.49 (m, 2H), 7.27-7.18 (m, 3H), 2.49 (s, 3H), 2.35 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 169.1, 162.7, 149.5, 147.7, 146.4, 144.0, 138.8, 138.7, 138.0, 131.5, 131.4, 131.3, 129.7, 127.5, 127.4, 127.1, 123.25, 123.19, 121.5, 119.2, 116.0, 21.6, 21.5. HRMS (ESI): m/z [M+H]⁺ calcd for $\text{C}_{25}\text{H}_{21}\text{N}_2\text{O}_5\text{S}$: 461.1171; found: 461.1166. FT-IR (neat, cm^{-1}) ν 3335, 2925, 1778, 1693, 1521, 1174, 1146, 750.



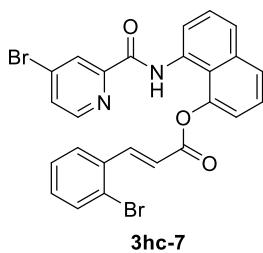
N-(8-hydroxynaphthalen-1-yl)picolinamide (**7**). Following the general procedure, **7** was obtained as a gray solid (42.2 mg, 80%). R_f = 0.20 (n-hexane/EtOAc 3:1). m.p. 226.0–228.0 °C. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 13.26 (s, 1H), 11.44 (s, 1H), 8.86 (d, J = 7.6 Hz, 1H), 8.74 (d, J = 4.8 Hz, 1H), 8.23 (d, J = 7.6 Hz, 1H), 8.10 (t, J = 7.6 Hz, 1H), 7.68 (dd, J = 7.6, 4.8 Hz, 1H), 7.57 (d, J = 8.4 Hz, 1H), 7.46 (t, J = 8.0 Hz, 1H), 7.37 (d, J = 8.0 Hz, 1H), 7.32 (t, J = 8.0 Hz, 1H), 6.95 (d, J = 7.6 Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, $\text{DMSO}-d_6$) δ 126.7, 154.0, 150.8, 149.1, 138.7, 136.7, 135.7, 127.4, 127.0, 126.6, 124.0, 122.8, 120.3, 115.8, 115.3, 110.6. HRMS (ESI): m/z [M+H]⁺ calcd for $\text{C}_{16}\text{H}_{13}\text{N}_2\text{O}_2$: 265.0977; found: 265.0972. FT-IR (neat, cm^{-1}) ν 2922, 2852, 1655, 1545, 817, 751.



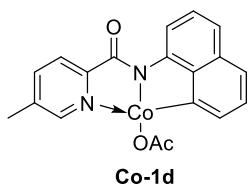
N-(8-hydroxynaphthalen-1-yl)acetamide (**8**). Following the general procedure, **8** was obtained as a white solid (44.8 mg, 85%). R_f = 0.10 (n-hexane/EtOAc 3:1). m.p. 156.0–158.0 °C. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 11.25 (s, 1H), 11.08 (s, 1H), 8.40 (d, J = 7.6 Hz, 1H), 7.49 (dd, J = 8.0, 0.8 Hz, 1H), 7.38-7.32 (m, 2H), 7.28 (t, J = 7.8 Hz, 1H), 6.89 (dd, J = 7.8, 1.2 Hz, 1H), 2.15 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, $\text{DMSO}-d_6$) δ 168.1, 153.8, 136.6, 136.1, 126.8, 126.5, 123.3, 120.4, 115.6, 115.1, 110.6, 25.8. HRMS (ESI): m/z [M+H]⁺ calcd for $\text{C}_{12}\text{H}_{12}\text{NO}_2$: 202.0868; found: 202.0863. FT-IR (neat, cm^{-1}) ν 2926, 2854, 1659, 1554, 1433, 1300, 818, 759.



8-(4-bromopicolinamido)naphthalen-1-yl 3-(2-bromophenyl)propanoate (**3ha-14**). Following the general procedure, **3ha-14** was obtained as a white solid (33.7 mg, 42%). R_f = 0.70 (n-hexane/EtOAc 3:1). m.p. 134.0–136.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.36 (s, 1H), 8.73 (d, J = 7.6 Hz, 1H), 8.48 (d, J = 1.6 Hz, 1H), 8.24 (d, J = 5.2 Hz, 1H), 7.76 (d, J = 8.4 Hz, 1H), 7.69 (d, J = 8.0 Hz, 1H), 7.58-7.49 (m, 3H), 7.45 (t, J = 7.6 Hz, 1H), 7.25-7.08 (m, 4H), 3.12 (s, 4H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 171.3, 160.8, 151.3, 148.4, 145.7, 139.3, 136.4, 134.9, 132.9, 132.2, 123.5, 129.7, 128.3, 127.7, 127.3, 126.6, 126.5, 125.31, 125.27, 124.2, 120.5, 119.5, 119.0, 34.4, 31.0. HRMS (ESI): m/z [M+H]⁺ calcd for $\text{C}_{25}\text{H}_{19}\text{Br}_2\text{N}_2\text{O}_3$: 552.9762; found: 552.9757. FT-IR (neat, cm^{-1}) ν 3347, 2926, 1766, 1685, 1529, 1499, 1101, 753.

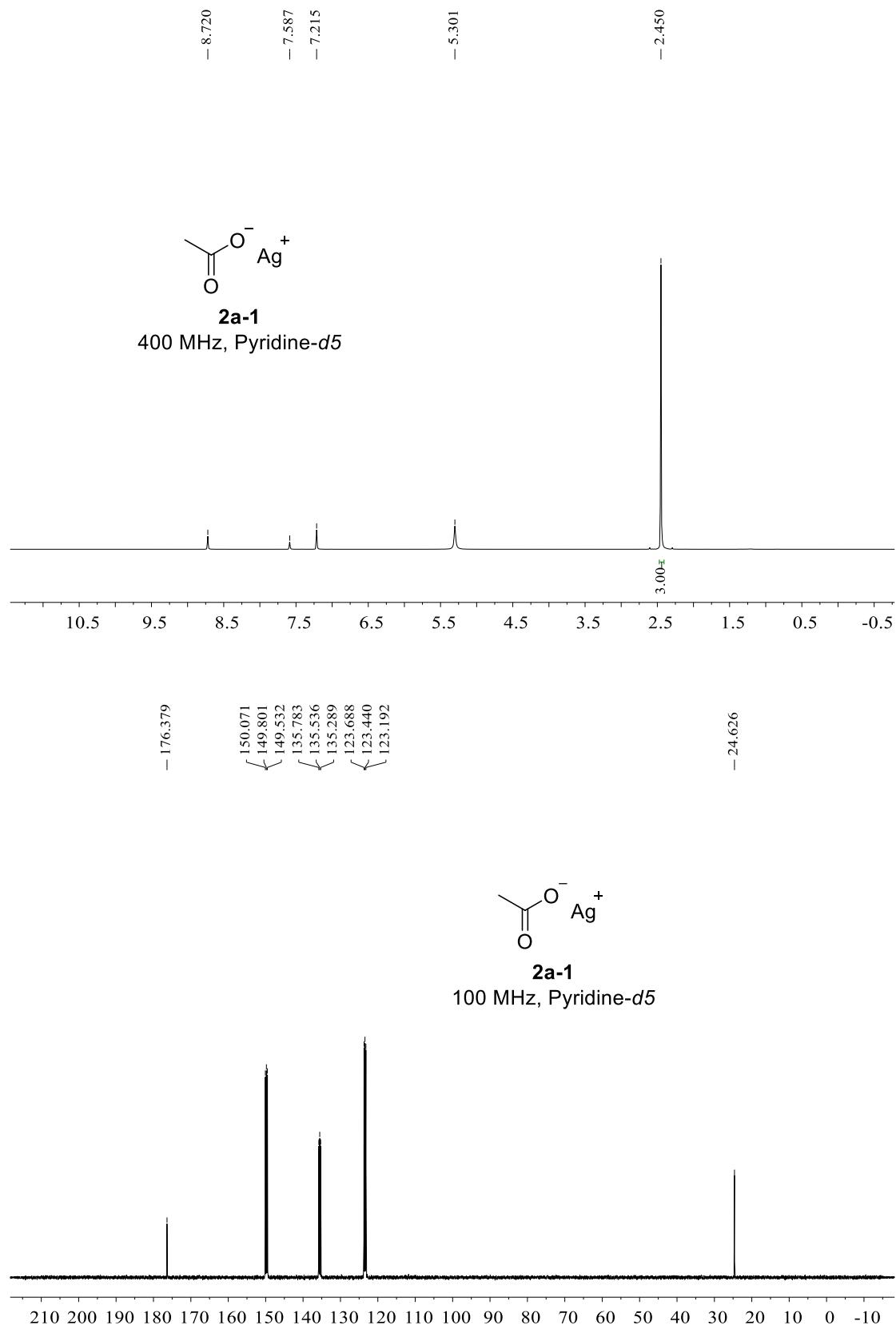


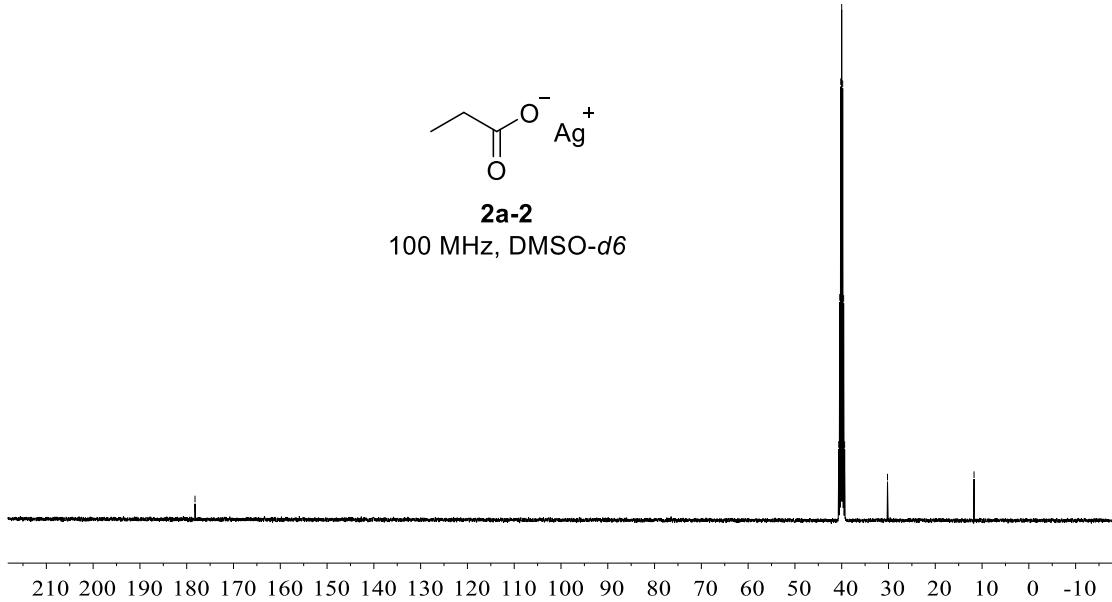
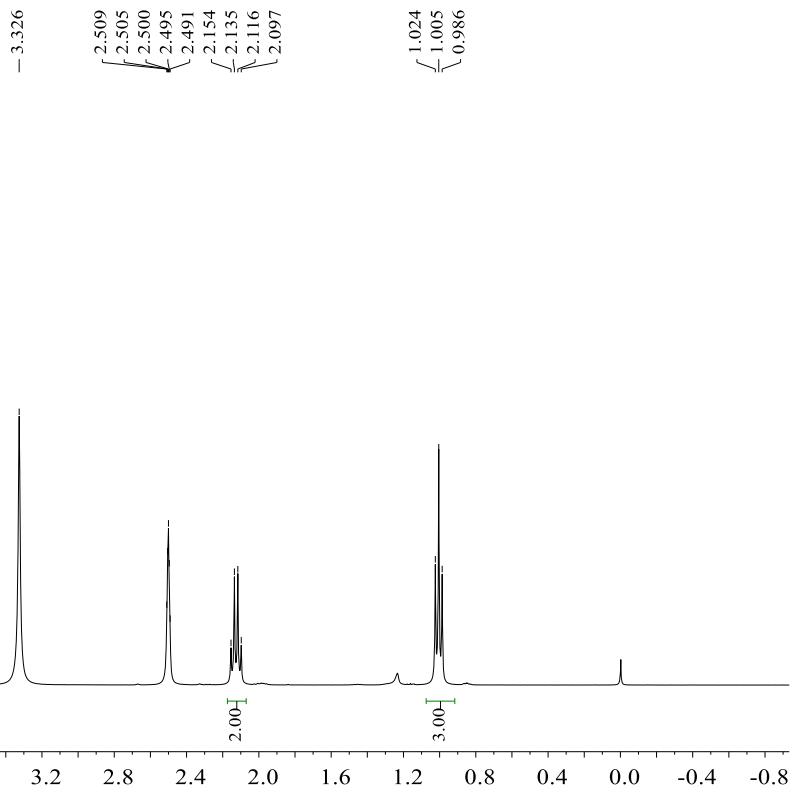
8-(4-bromopicolinamido)naphthalen-1-yl-3-(2-bromophenyl)acrylate (**3hc-7**). Following the general procedure, **3hc-7** was obtained as a yellow solid (79.5 mg, 72%). $R_f = 0.50$ (n-hexane/EtOAc 3:1). m.p. 157.0–159.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 11.16 (s, 1H), 8.53 (d, $J = 7.6$ Hz, 1H), 8.45 (d, $J = 1.2$ Hz, 1H), 8.18–8.07 (m, 2H), 7.80 (d, $J = 8.0$ Hz, 1H), 7.74 (d, $J = 8.4$ Hz, 1H), 7.64 (d, $J = 7.6$ Hz, 1H), 7.55 (t, $J = 8.0$ Hz, 1H), 7.50 (t, $J = 8.0$ Hz, 1H), 7.41–7.38 (m, 1H), 7.35–7.27 (m, 4H), 6.66 (d, $J = 16.0$ Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 164.8, 161.0, 151.3, 148.2, 146.0, 145.1, 136.4, 134.6, 134.0, 133.7, 131.77, 131.75, 129.2, 127.7, 127.5, 127.3, 126.4, 125.7, 125.6, 125.5, 120.8, 120.3, 120.2, 119.9. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{25}\text{H}_{17}\text{Br}_2\text{N}_2\text{O}_3$: 550.9606; found: 550.9600. FT-IR (neat, cm^{-1}) ν 3349, 2924, 1741, 1685, 1530, 1497, 1118, 756.

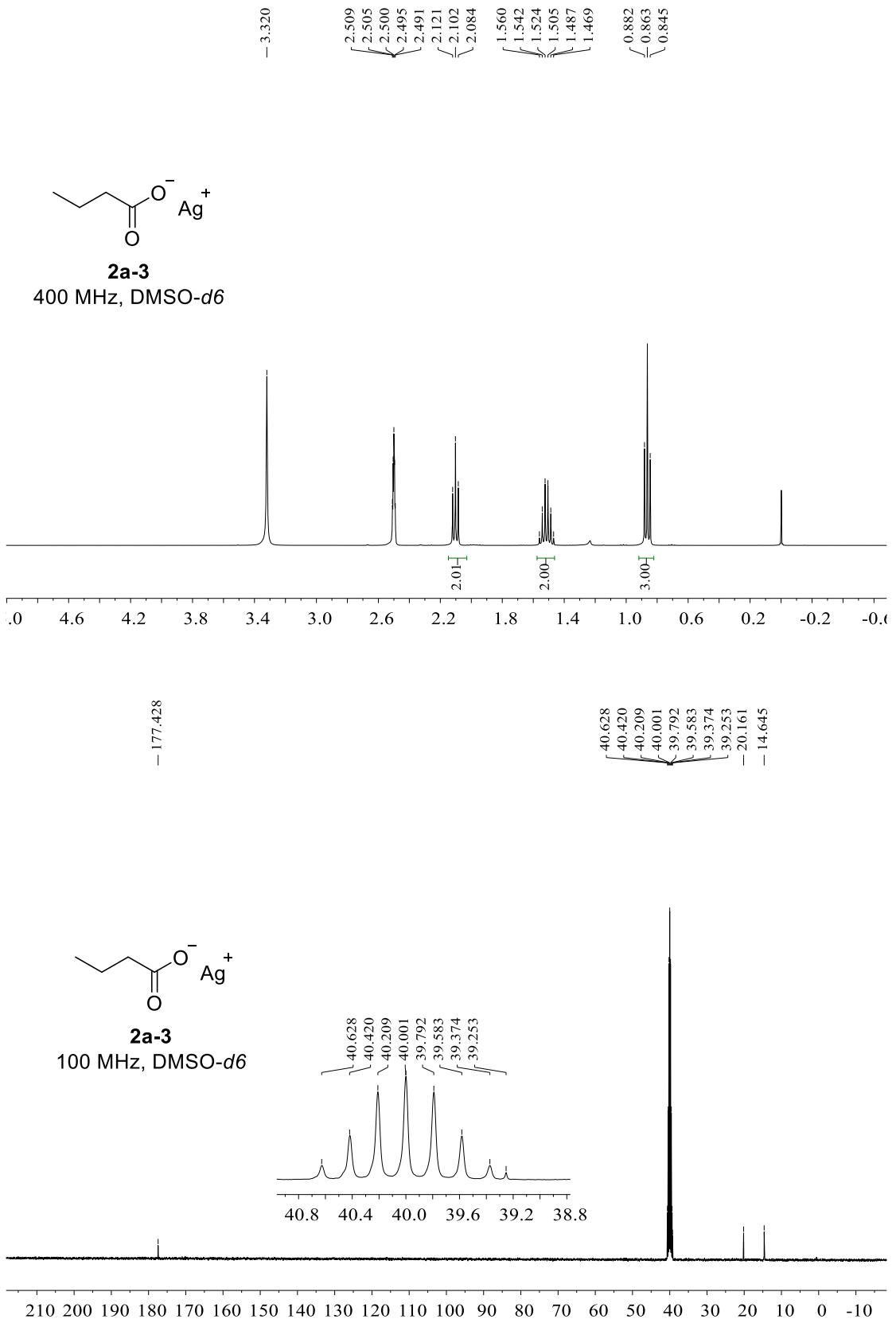


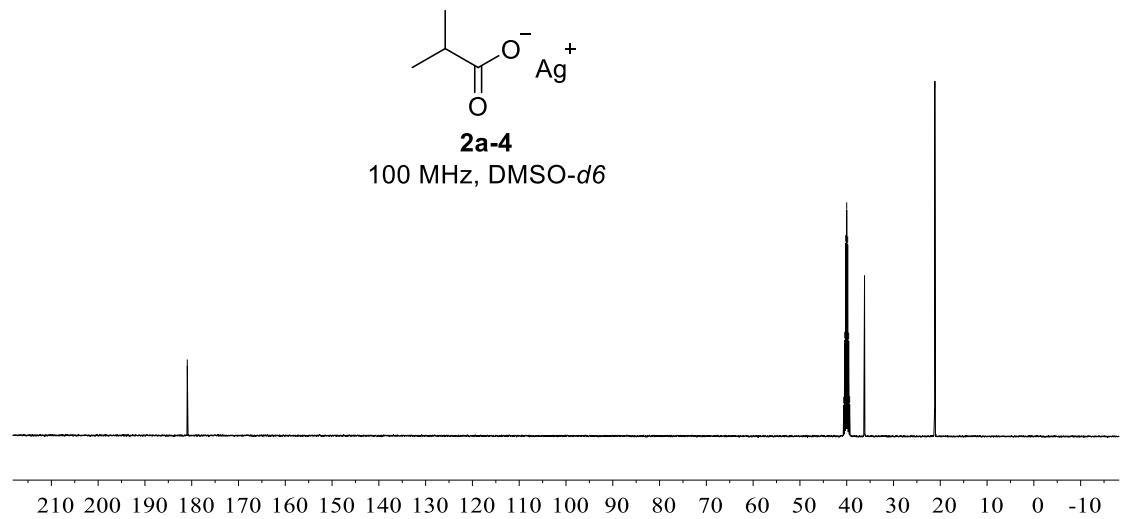
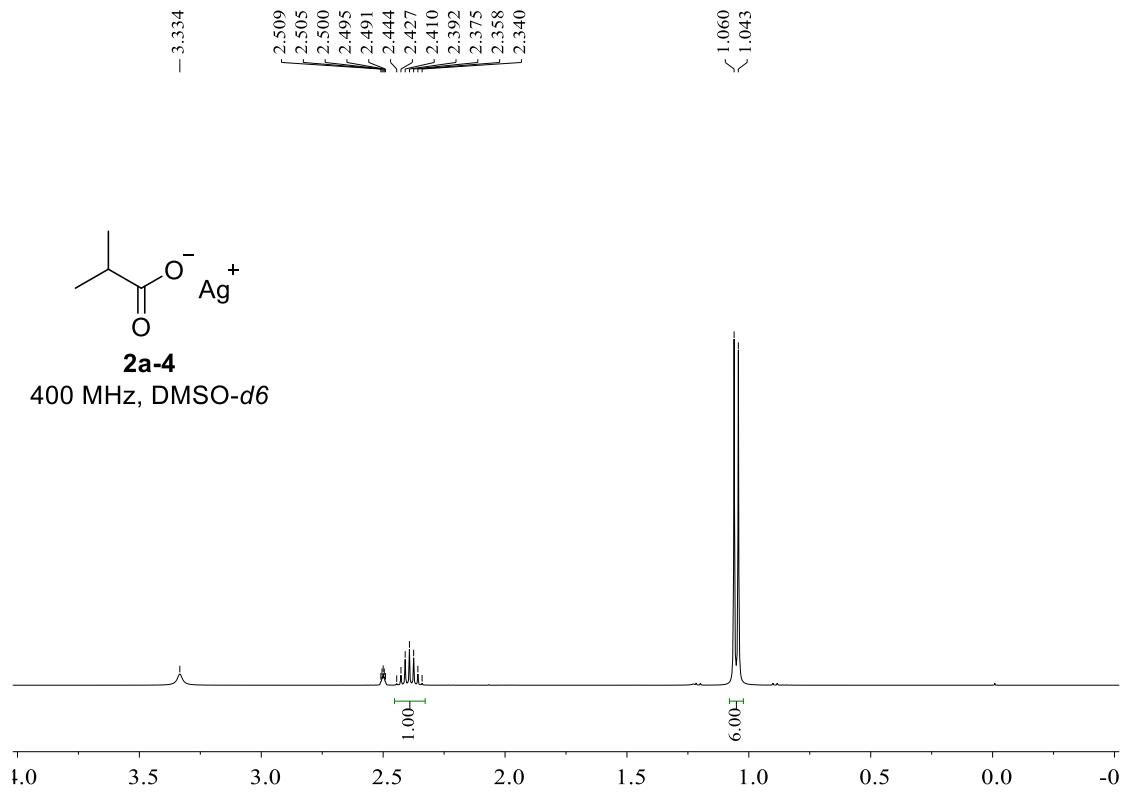
Co-1d was obtained as a red oil. $R_f = 0.50$ (EtOAc). ^1H NMR (400 MHz, CDCl_3) δ 7.90 (s, 1H), 7.87–7.82 (m, 1H), 7.77 (d, $J = 7.6$ Hz, 1H), 7.58–7.51 (m, 2H), 7.41 (t, $J = 7.2$ Hz, 1H), 7.33–7.17 (m, 1H), 7.04–6.89 (m, 2H), 1.93–1.64 (m, 6H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 192.1, 169.6, 154.3, 150.1, 141.5, 139.8, 137.1, 134.1, 131.7, 127.7, 126.4, 125.9, 125.5, 124.9, 124.7, 124.2, 23.8, 18.2. HRMS (ESI): m/z [M+H] $^+$ calcd for $\text{C}_{19}\text{H}_{16}\text{CoN}_2\text{O}_3$: 379.0493; found: 379.0487. FT-IR (neat, cm^{-1}) ν 3048, 2923, 1634, 1606, 1475, 1398, 796, 773.

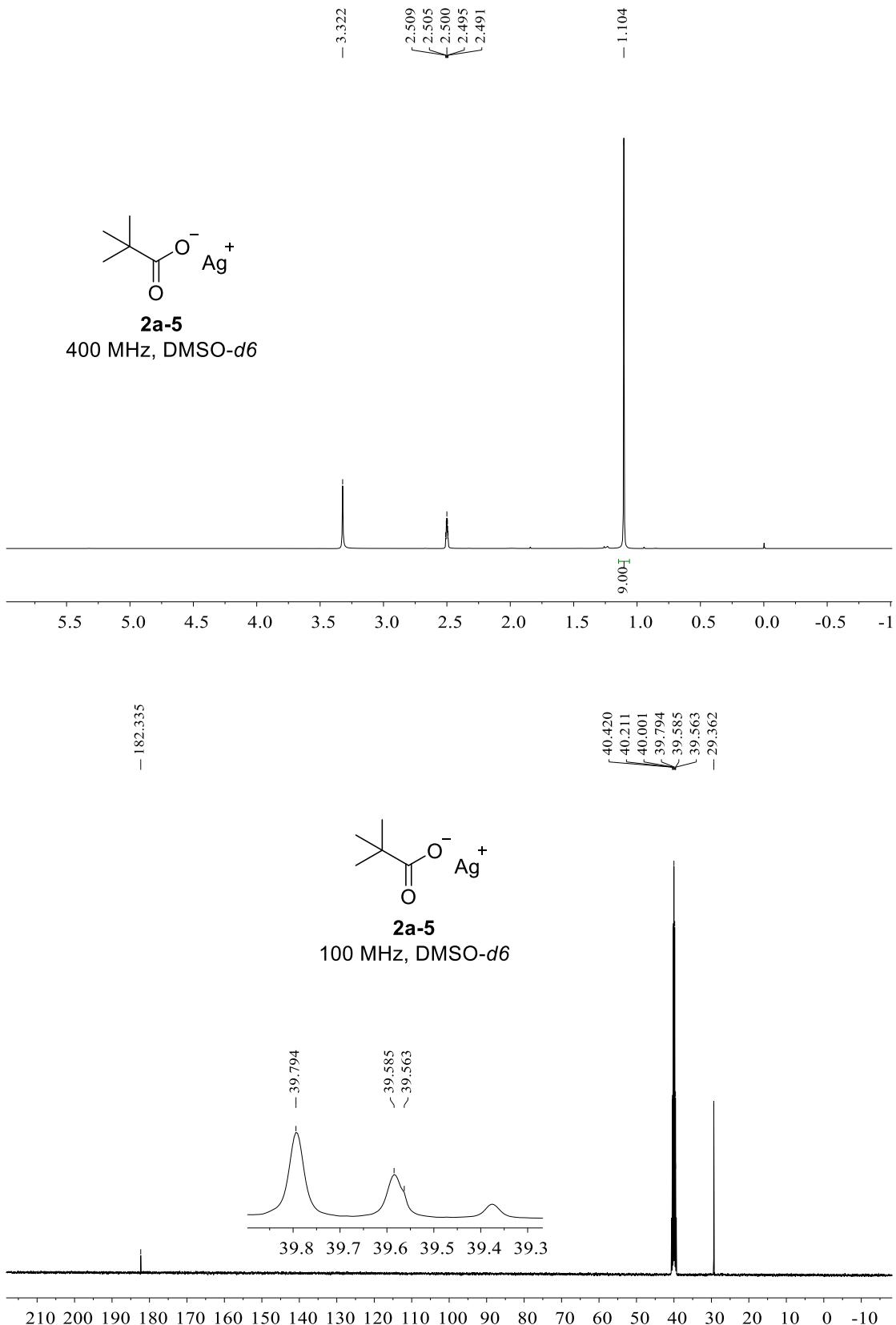
8. Copies of ^1H , ^{13}C and ^{19}F NMR spectra of silver carboxylates **2a-1–2d-16**

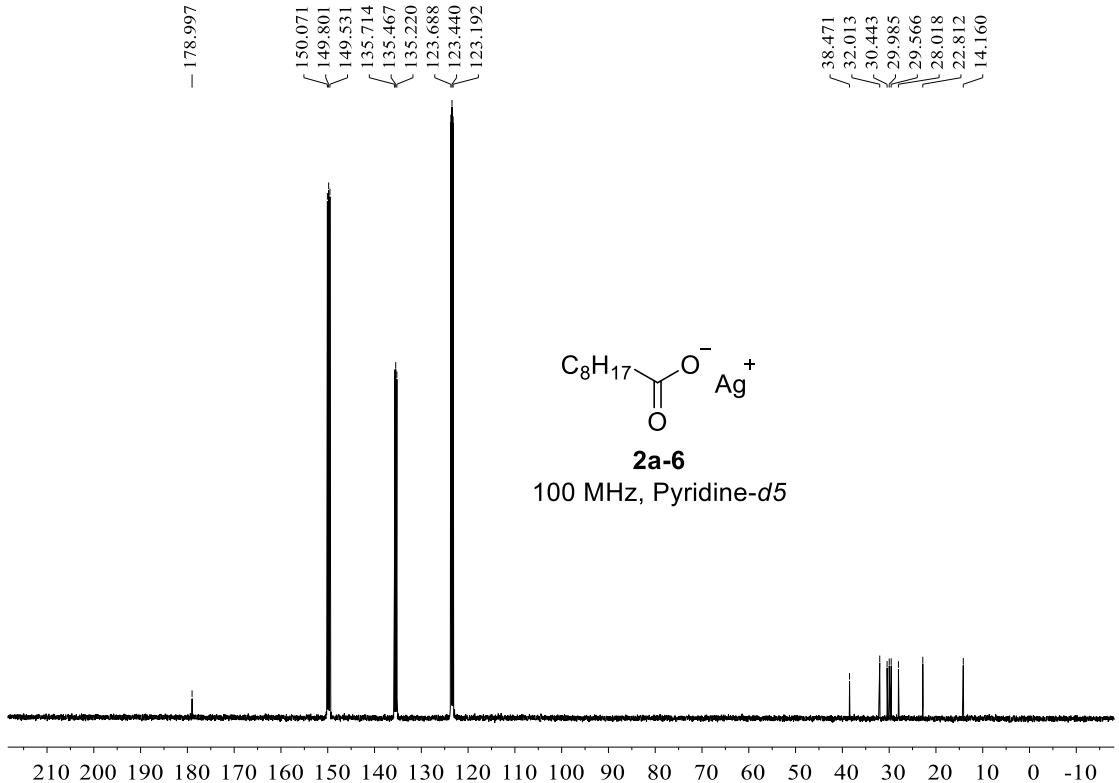
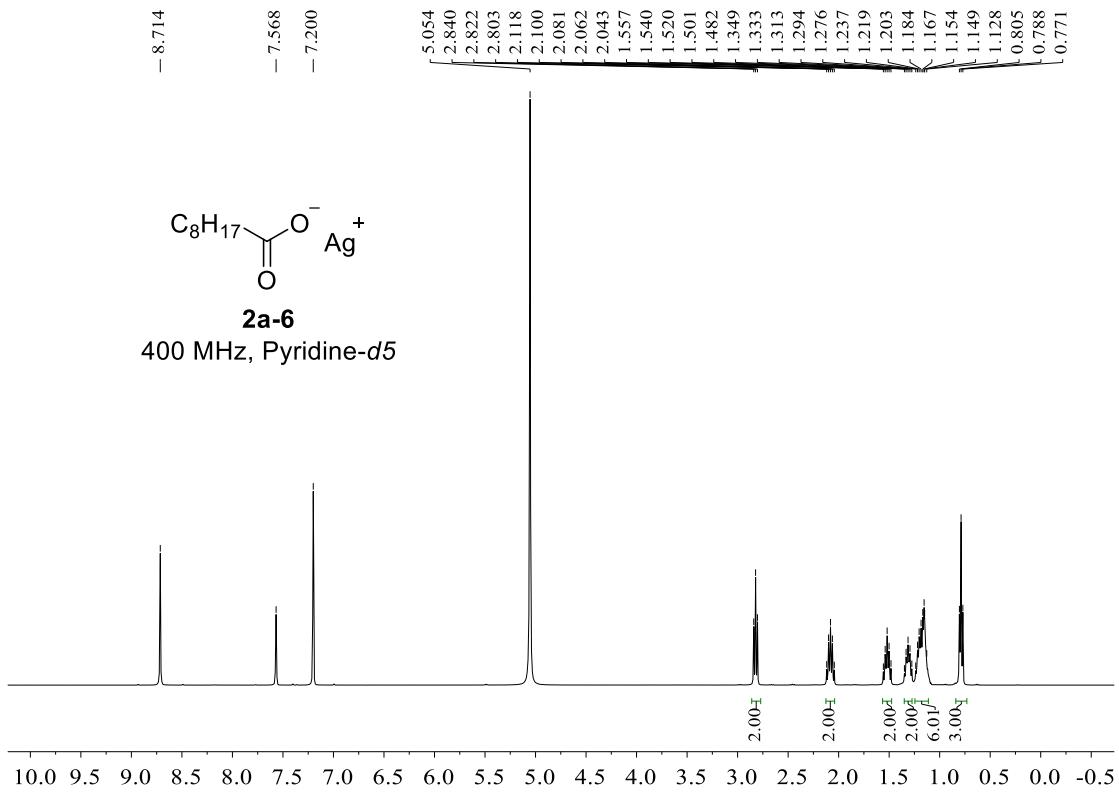


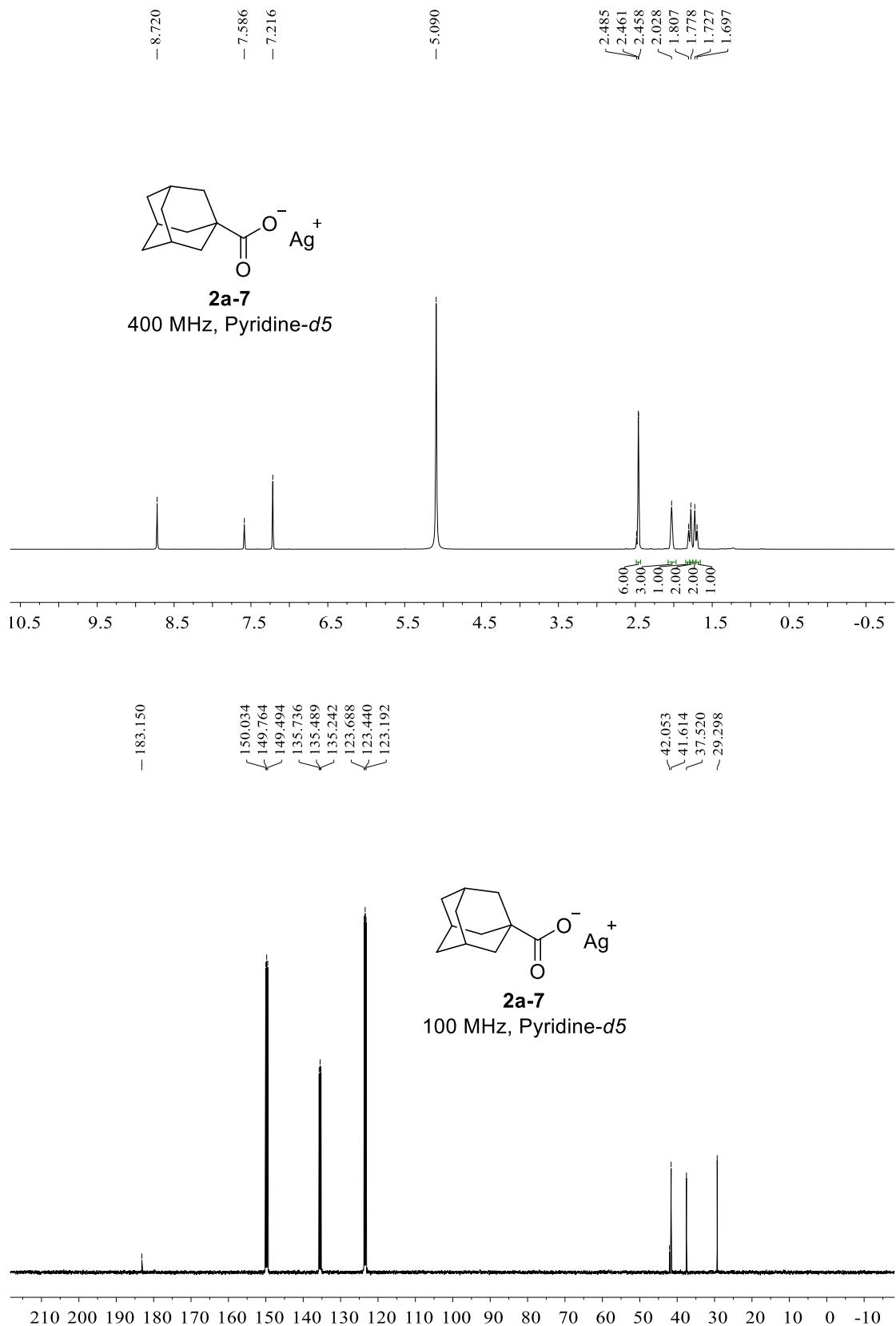


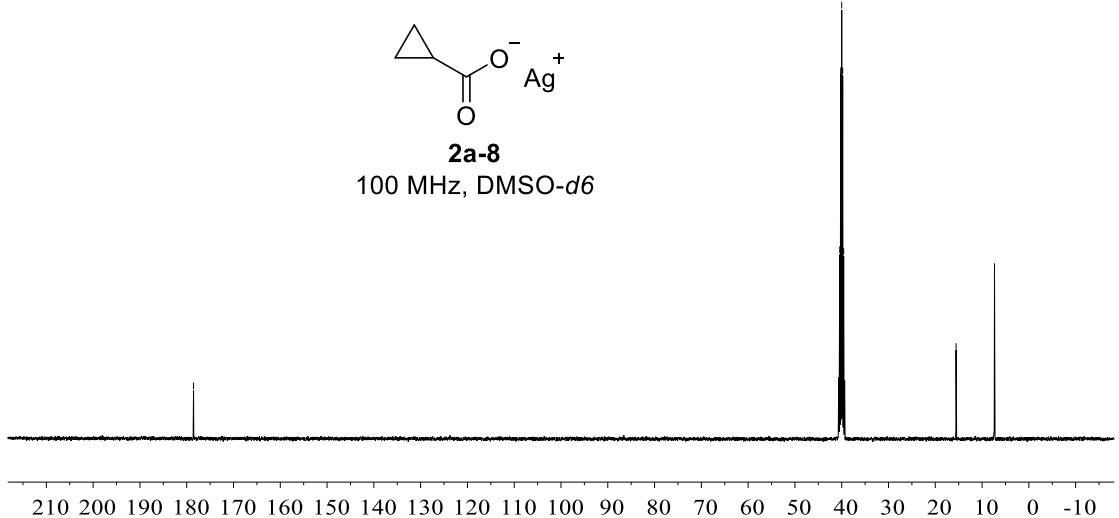
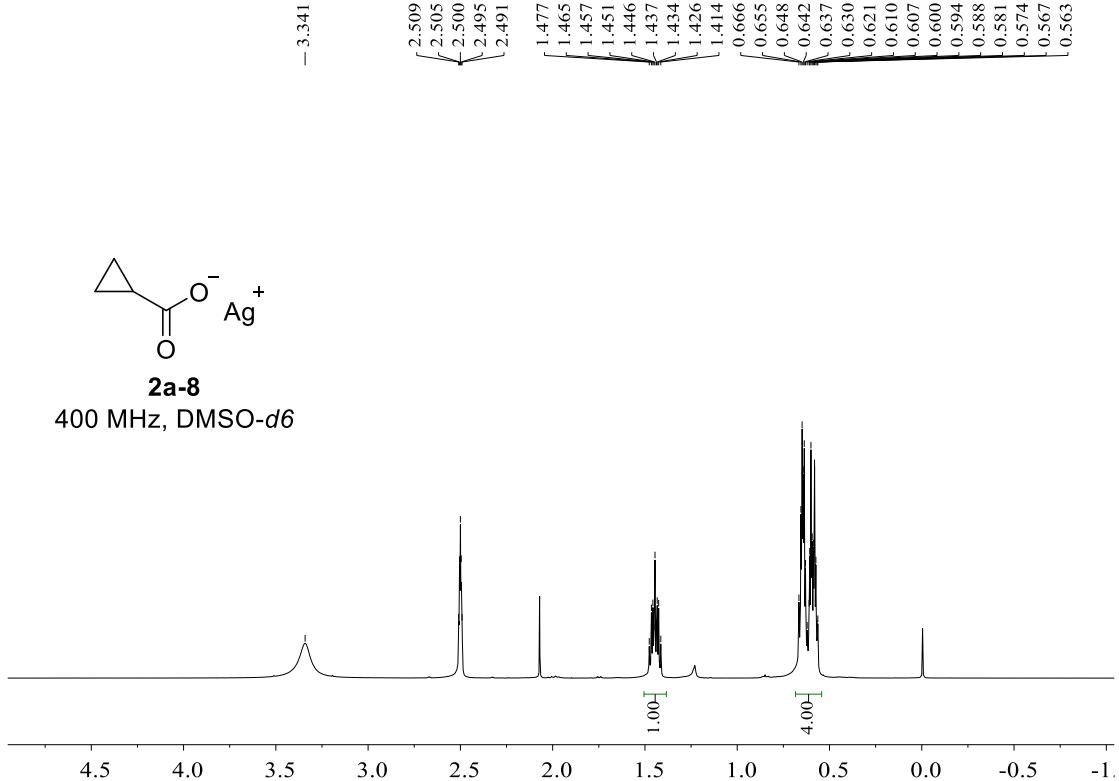


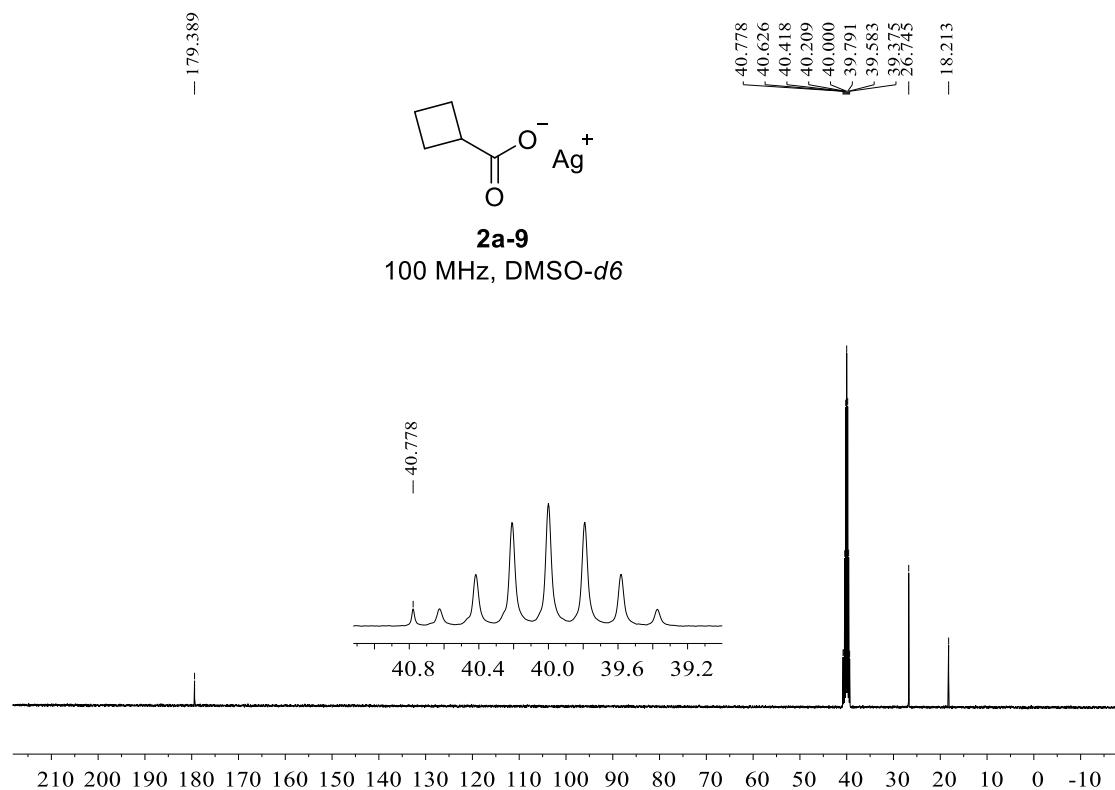
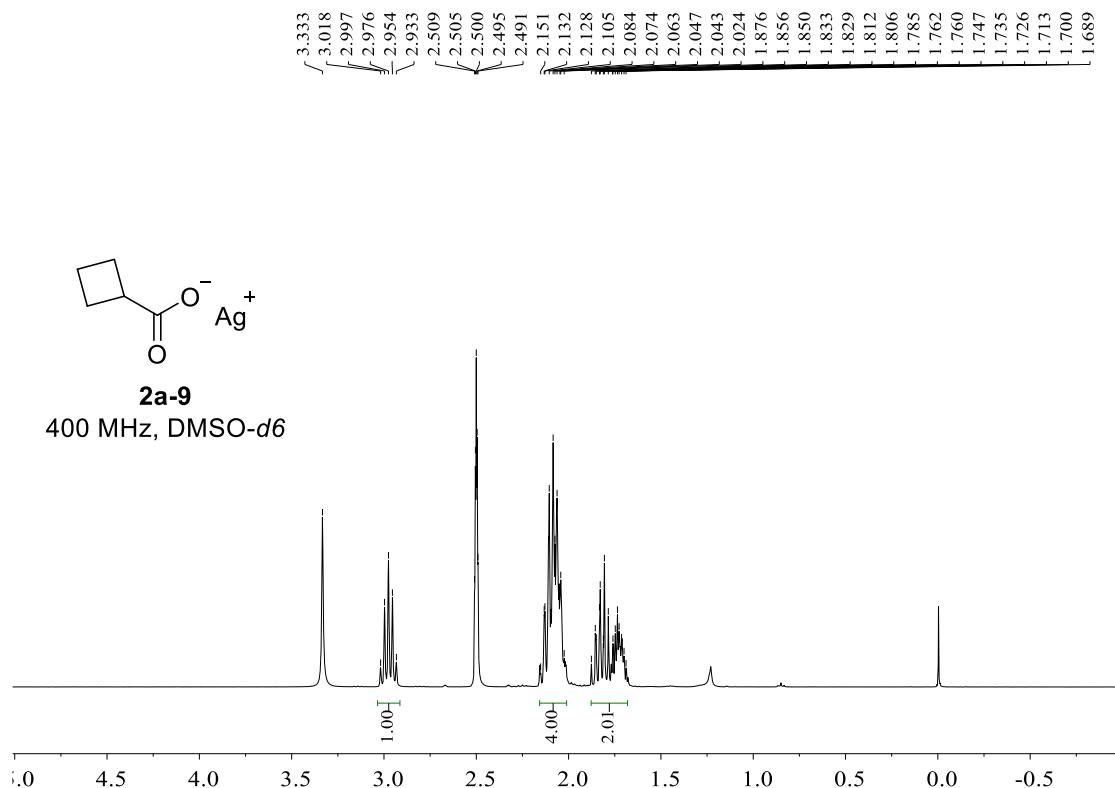


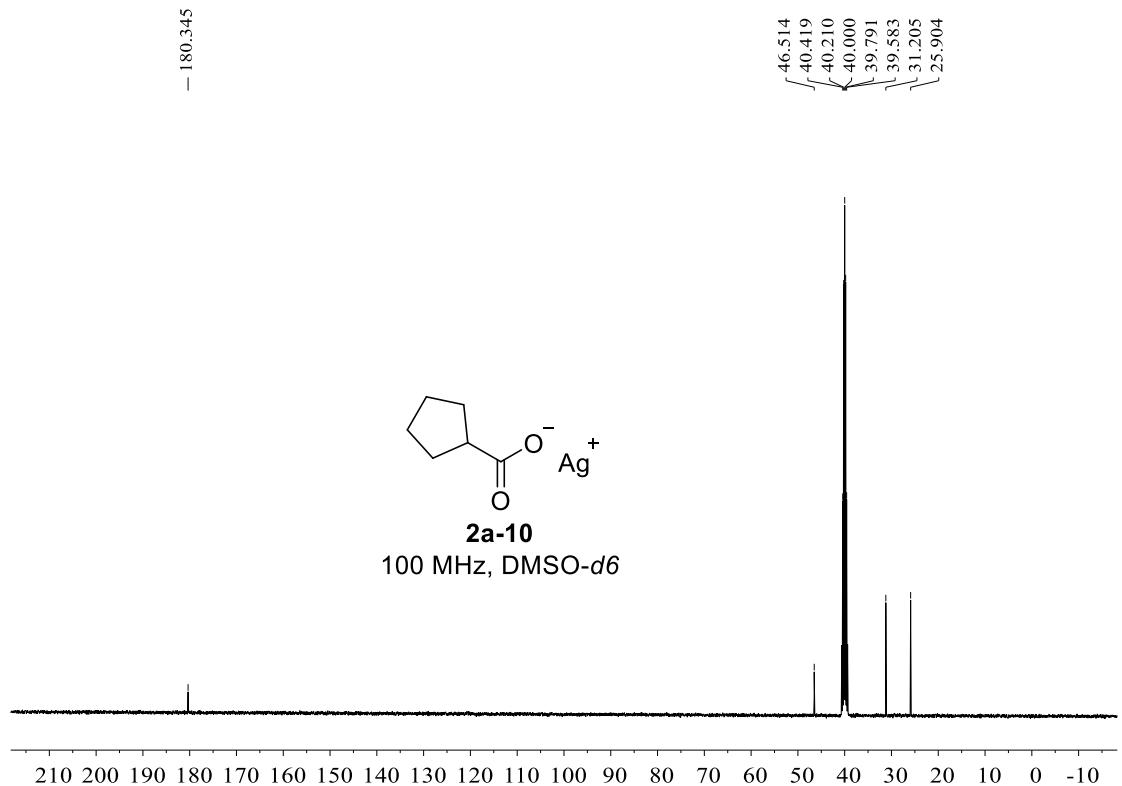
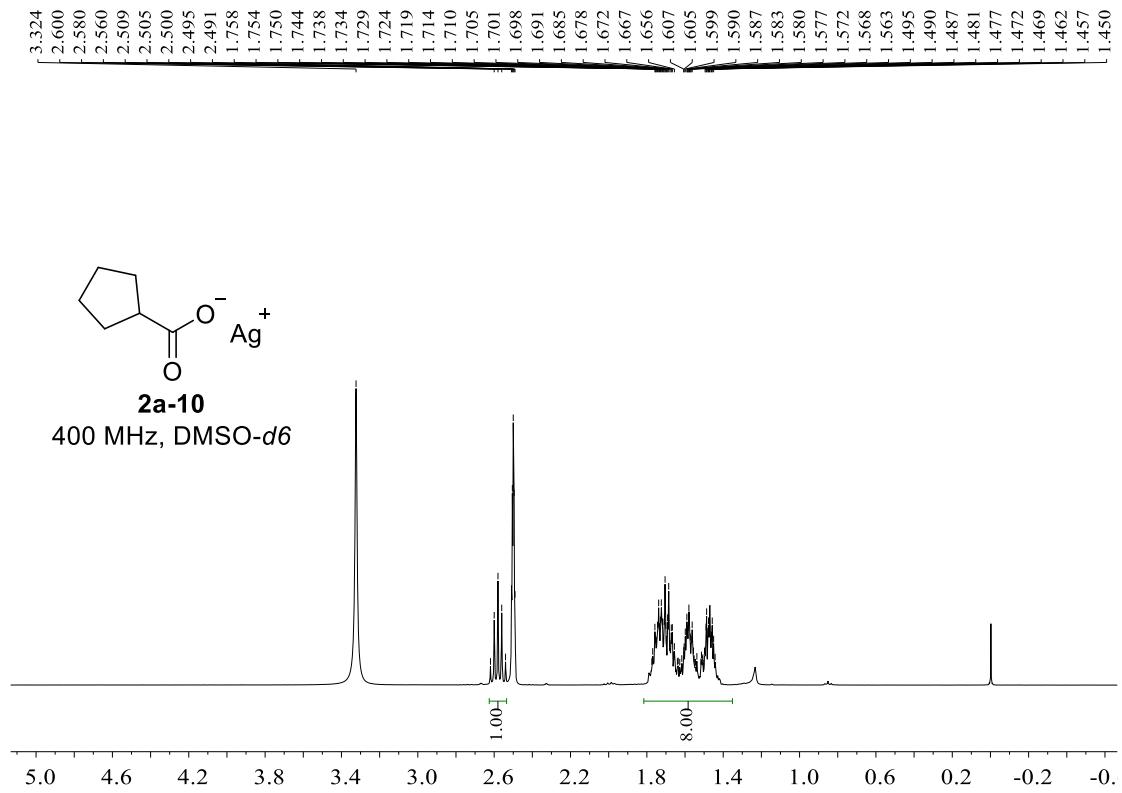


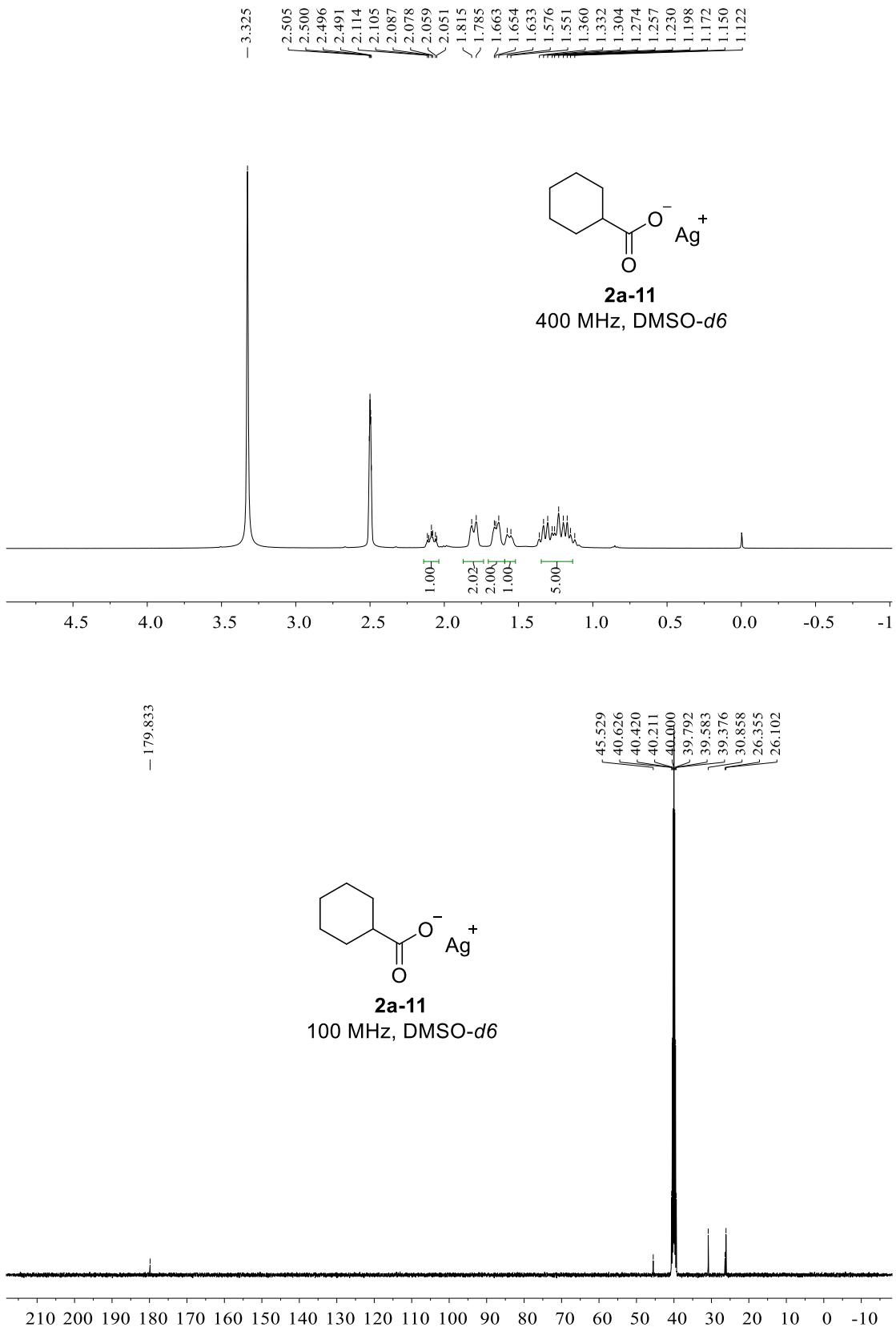


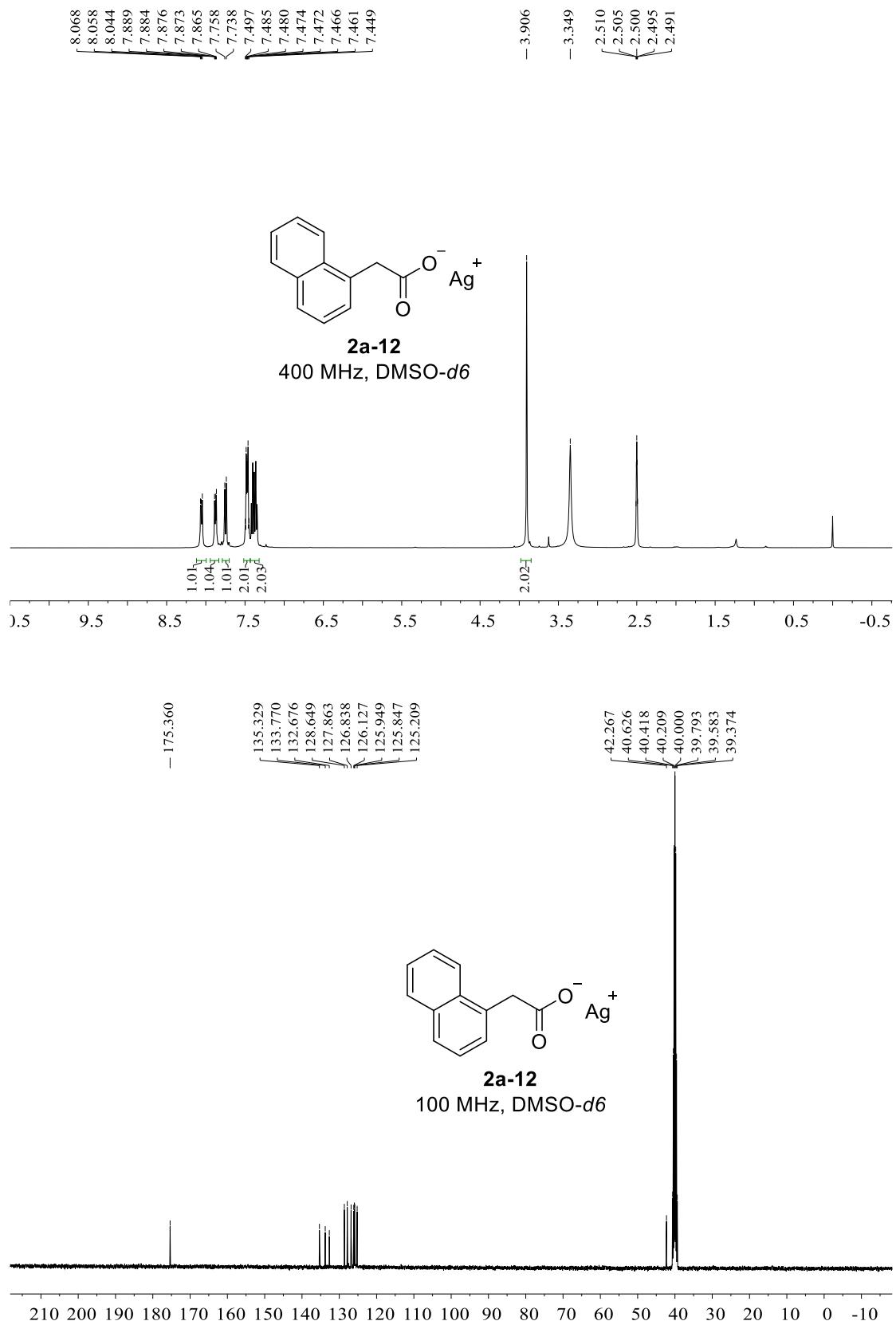


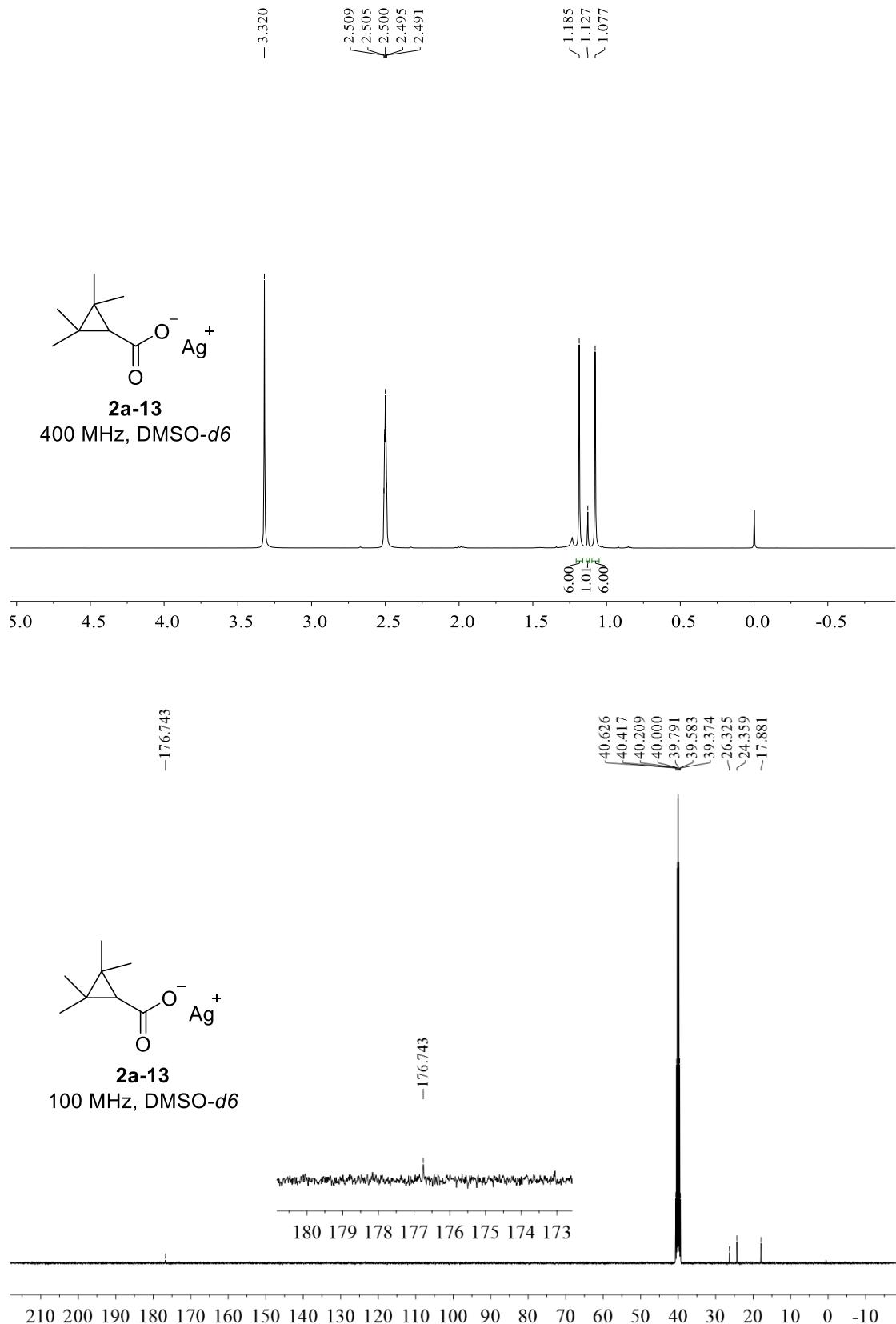


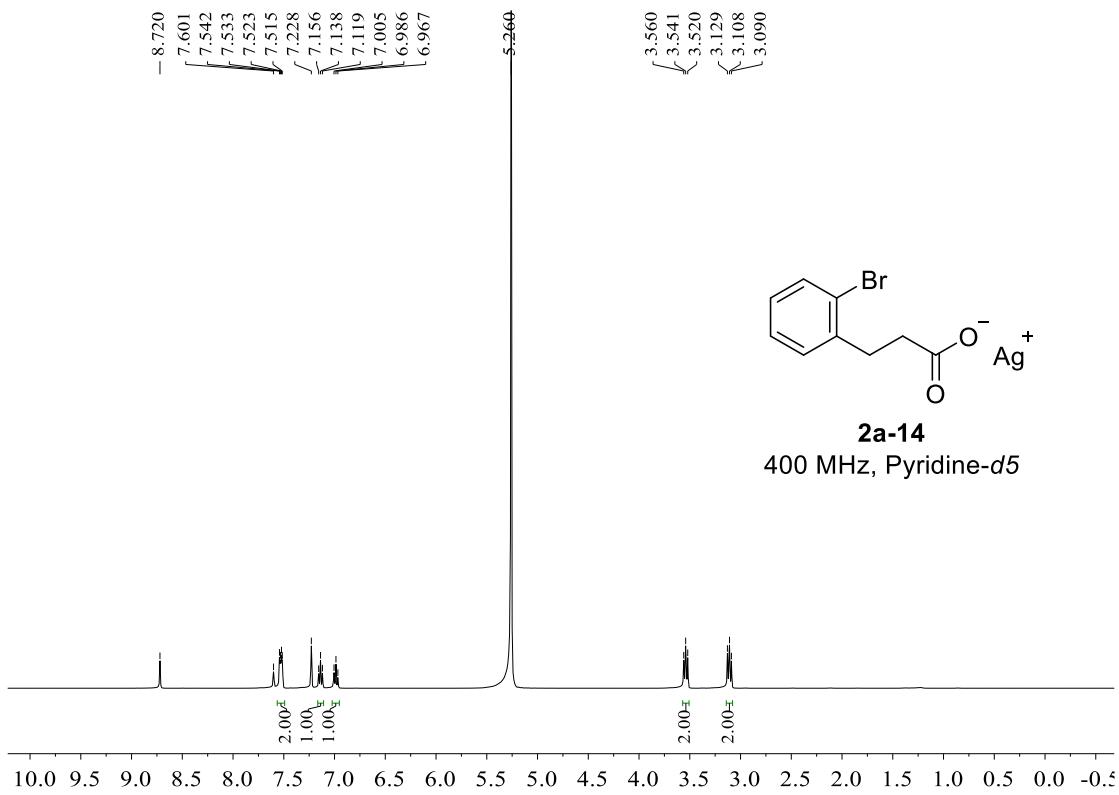








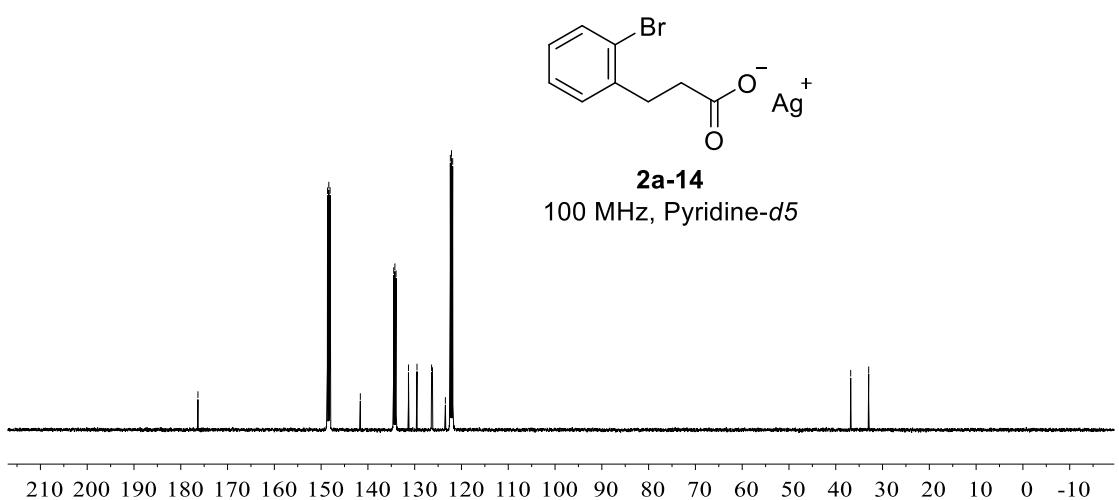


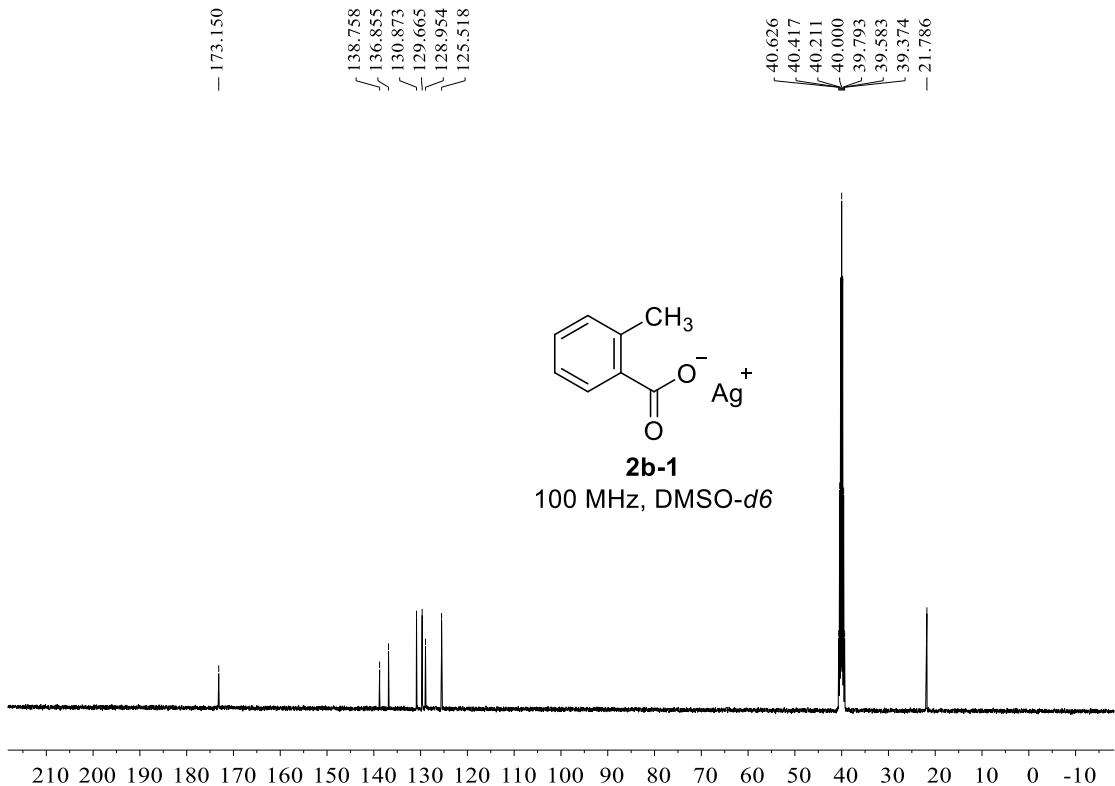
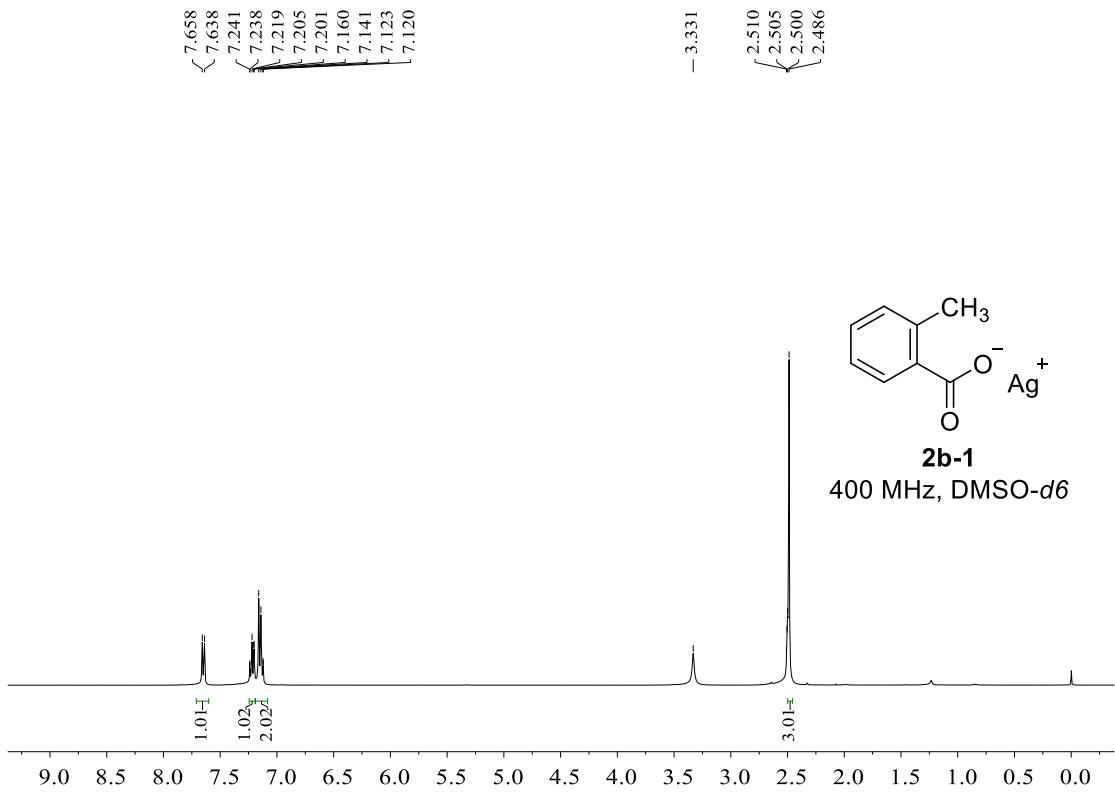


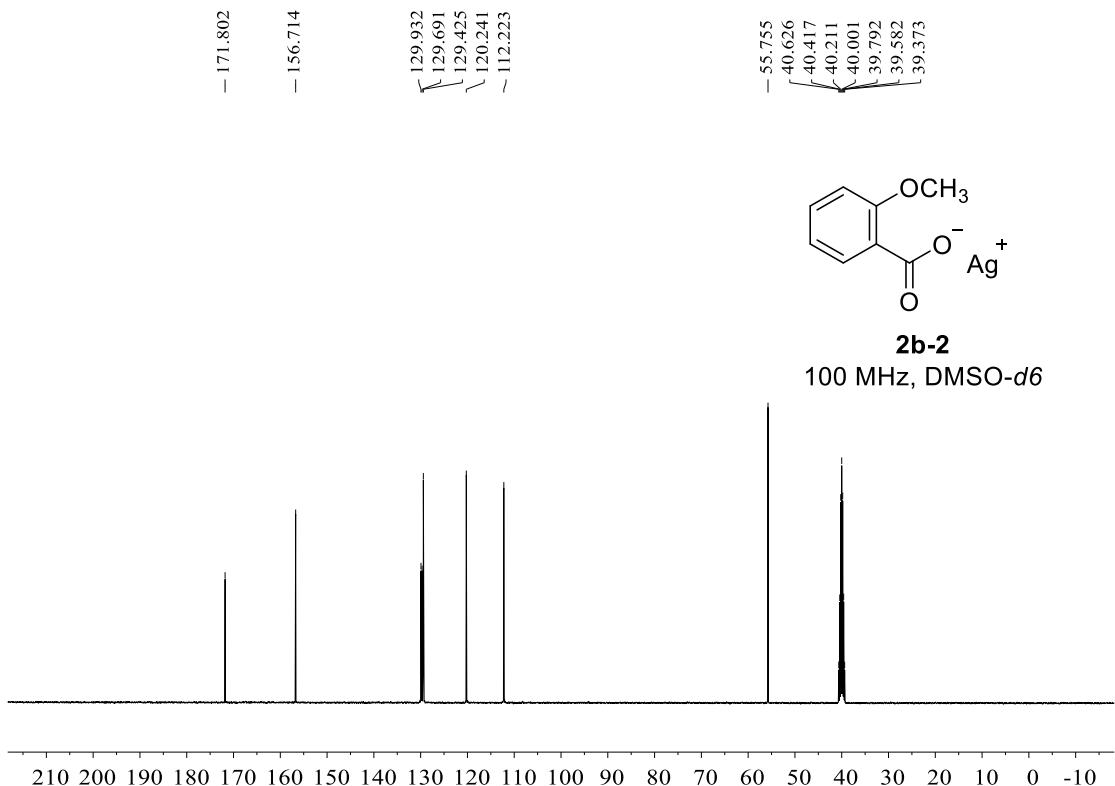
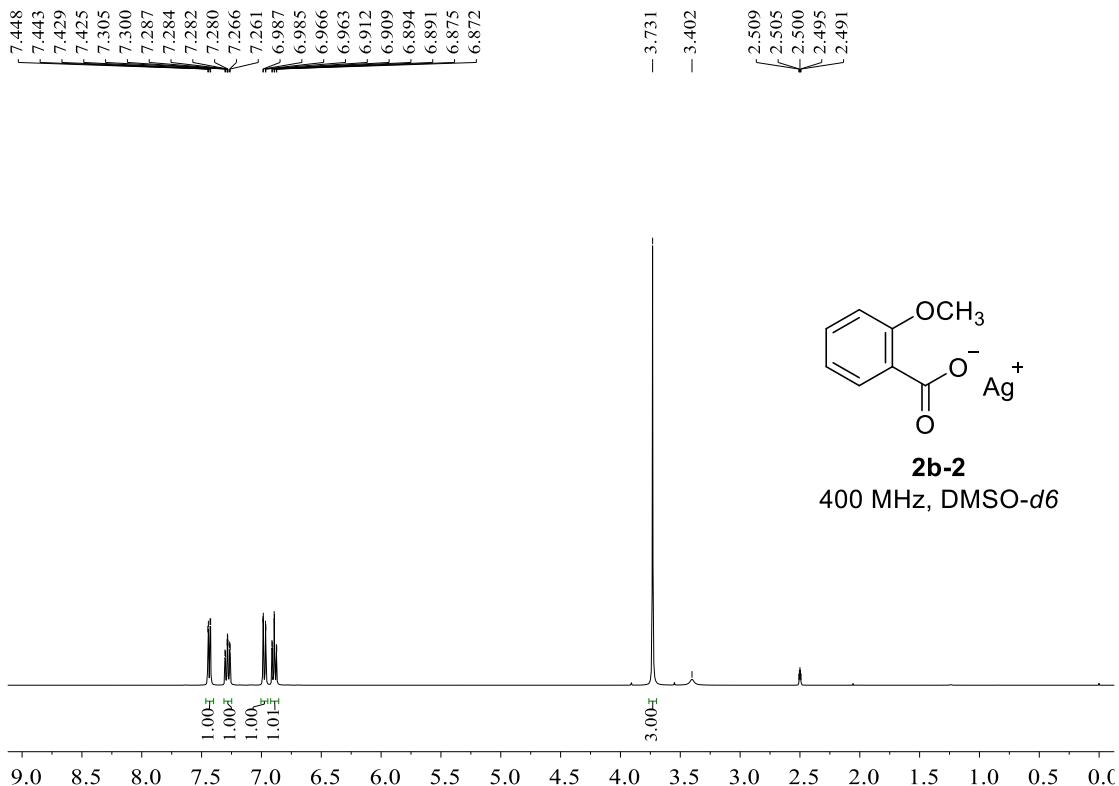
— 176.333
 — 148.627
 — 148.357
 — 148.087
 — 141.620
 — 134.481
 — 134.233
 — 133.987
 — 131.330
 — 129.508
 — 126.389
 — 126.205
 — 123.440
 — 122.378
 — 122.129
 — 121.881

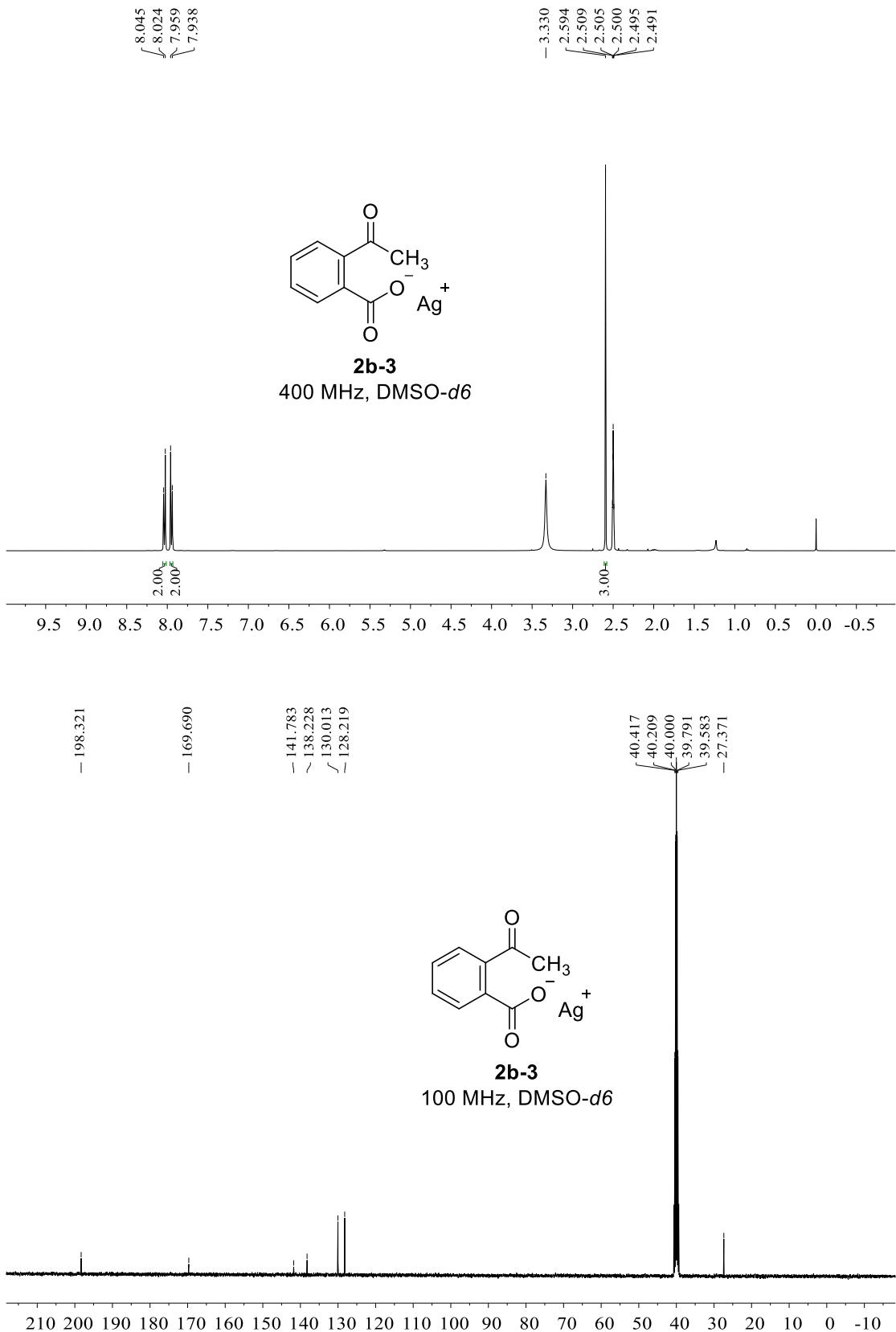
— 3.560
 { 3.541
 { 3.520
 { 3.129
 { 3.108
 { 3.090

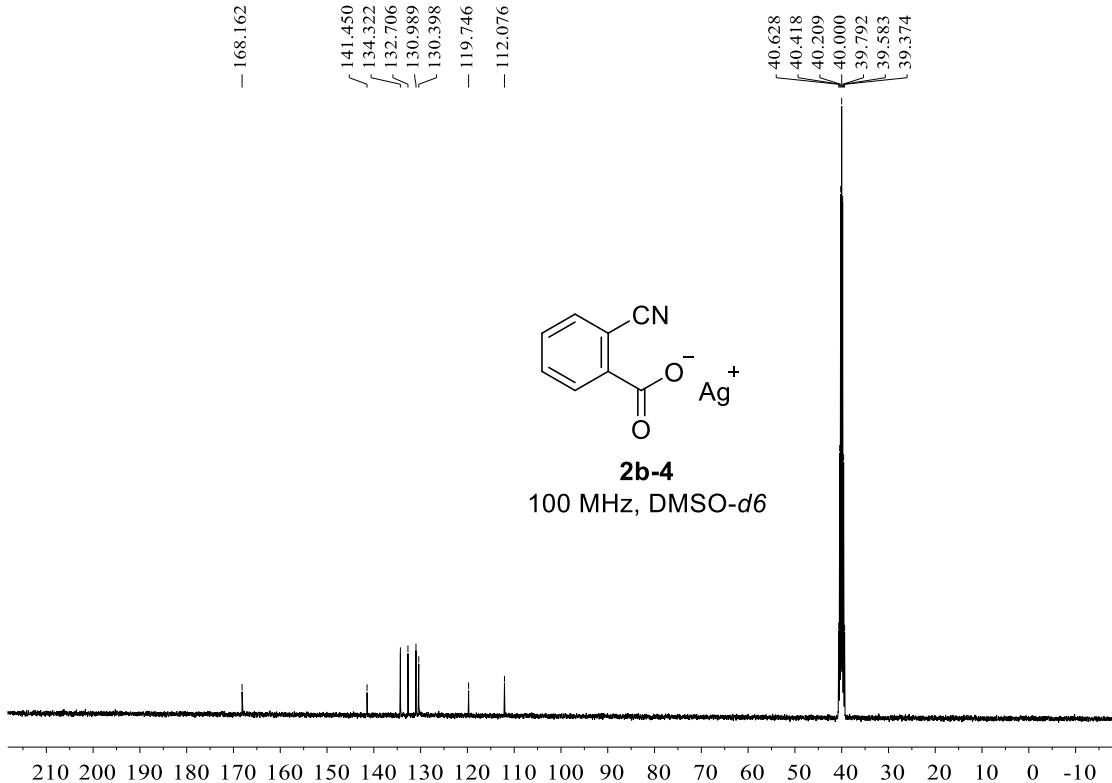
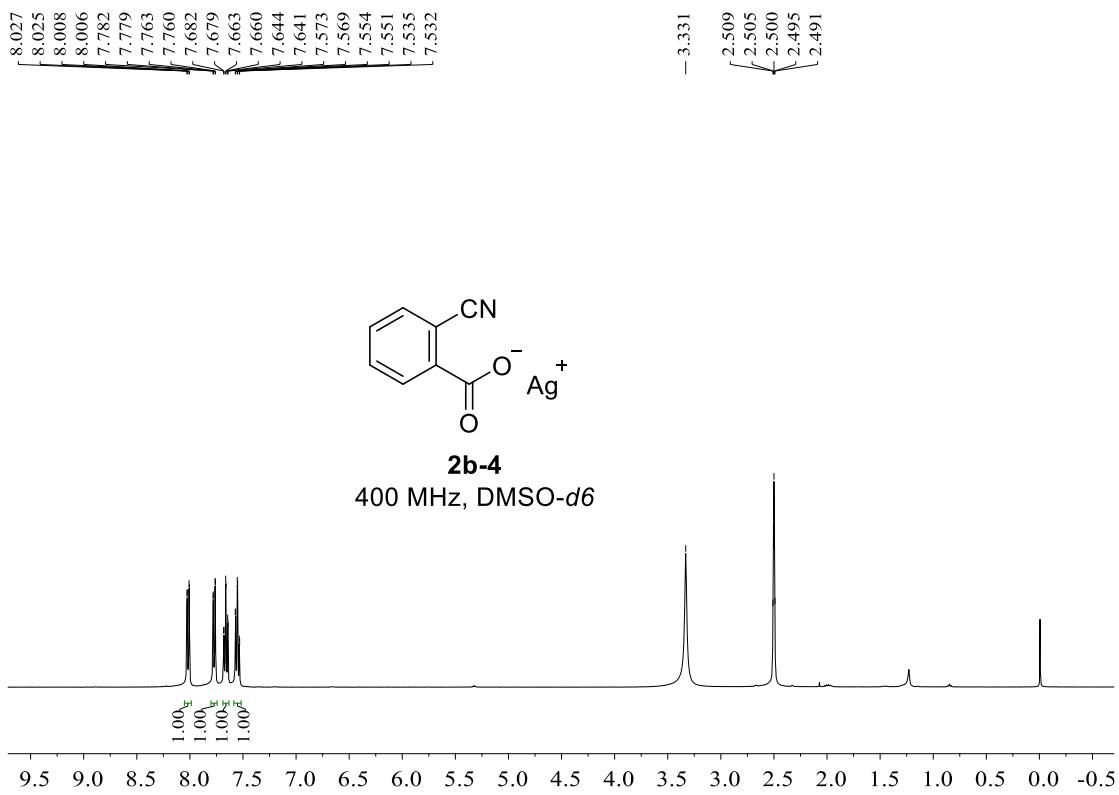
— 2.00_z
 — 1.00_b
 — 1.00_a

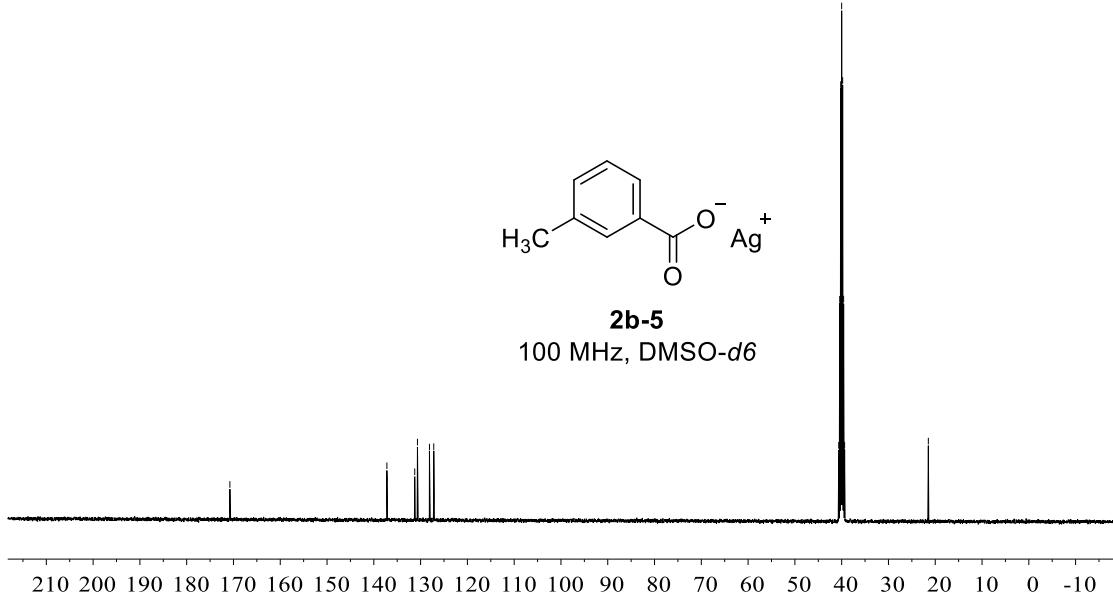
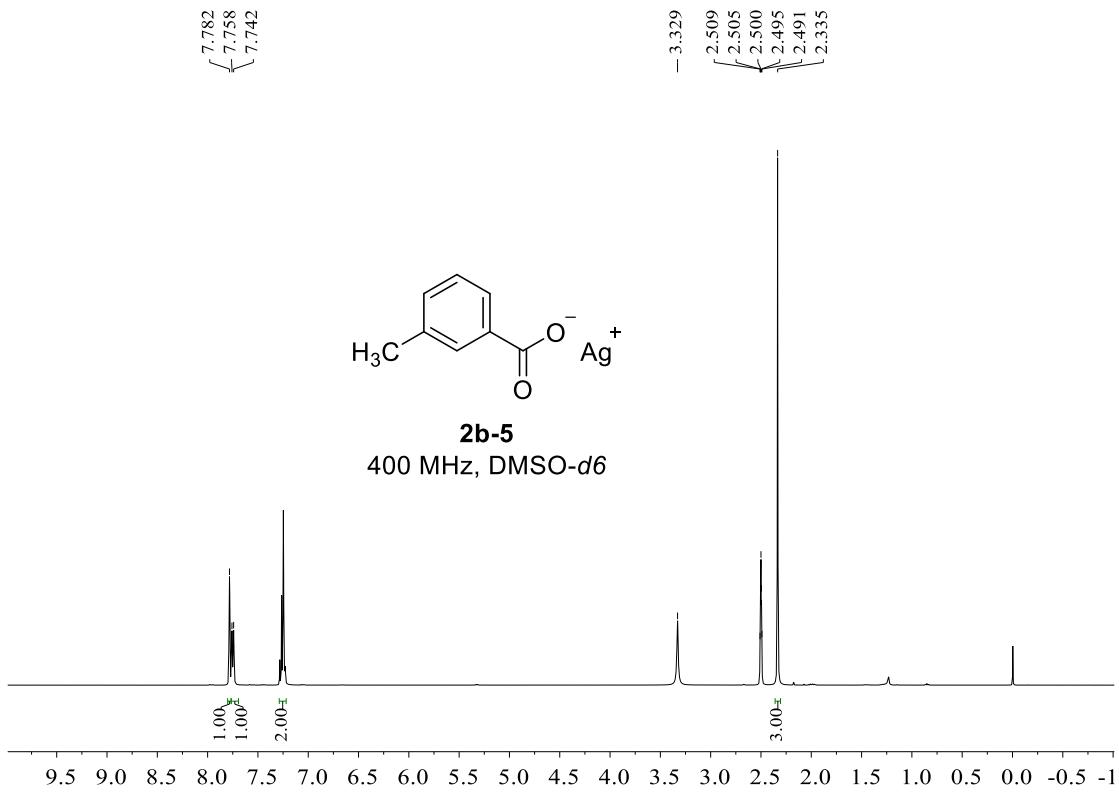


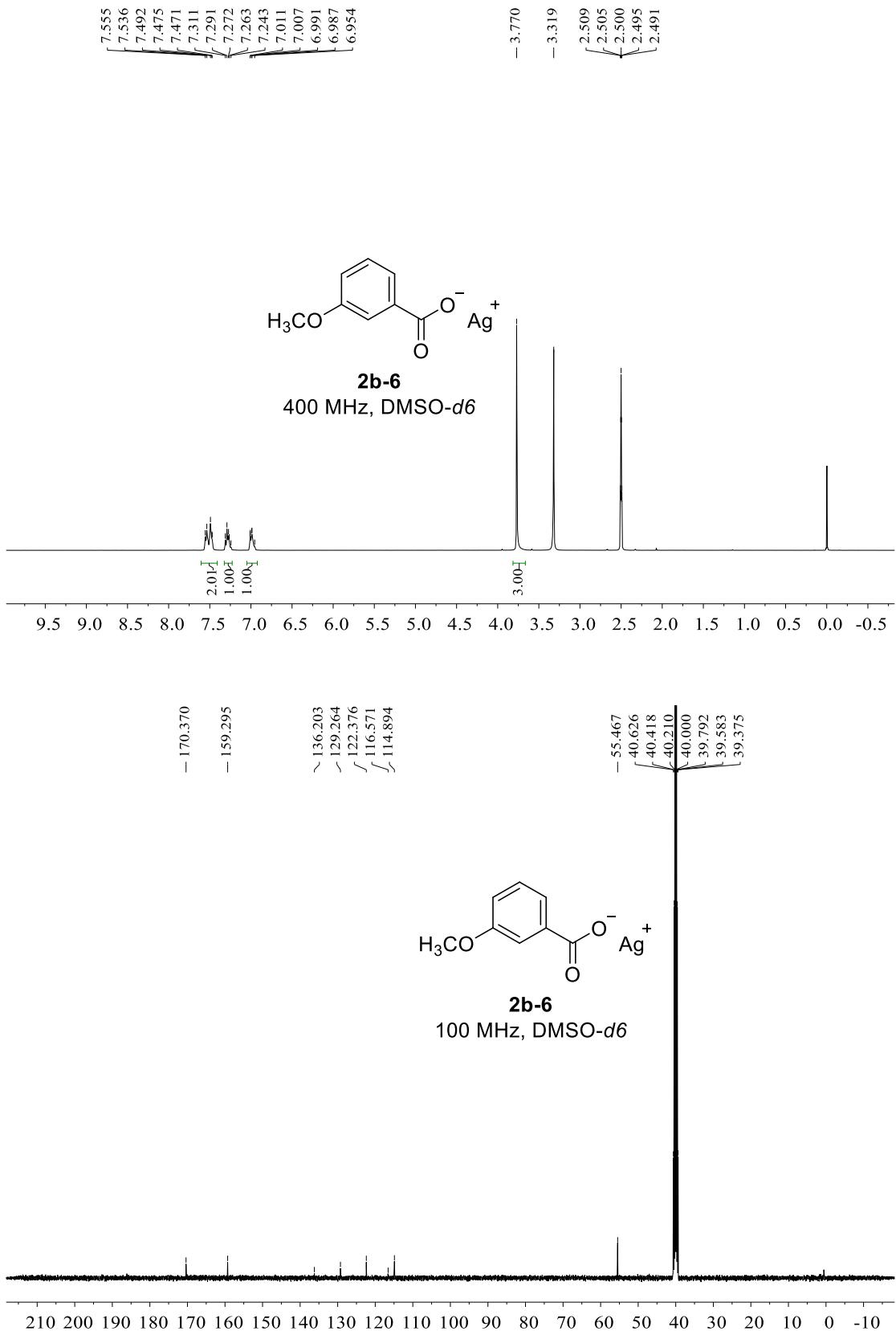


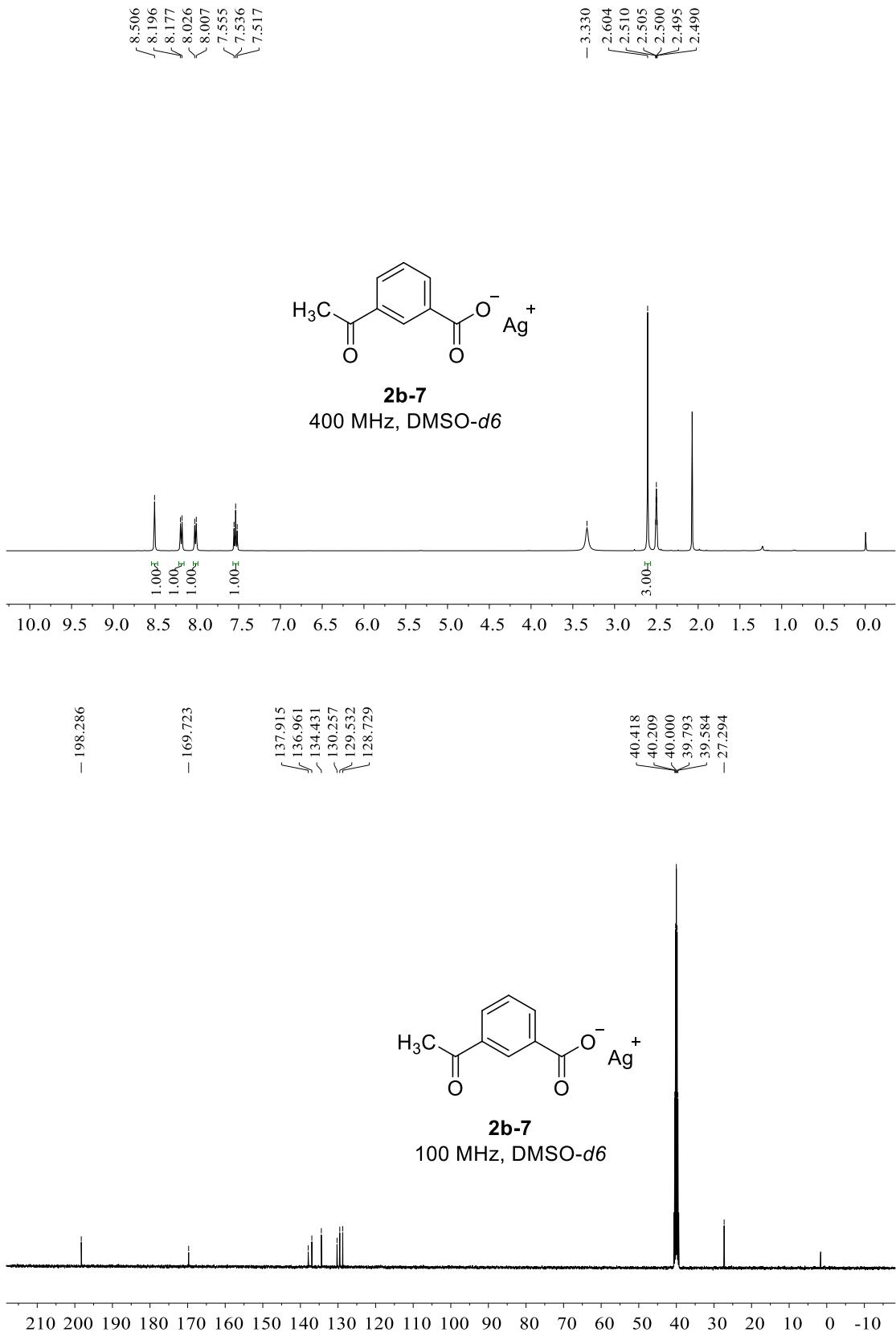


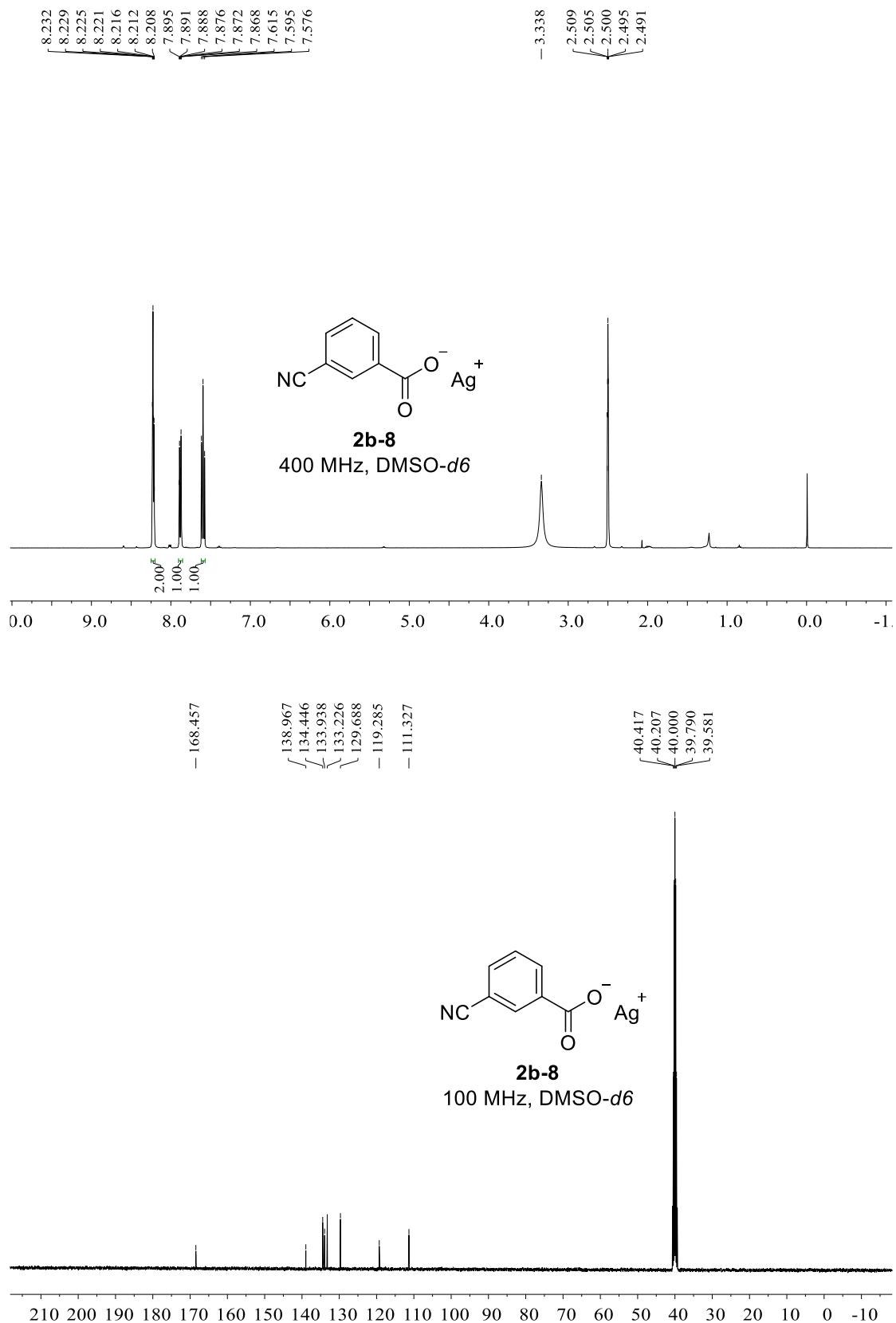


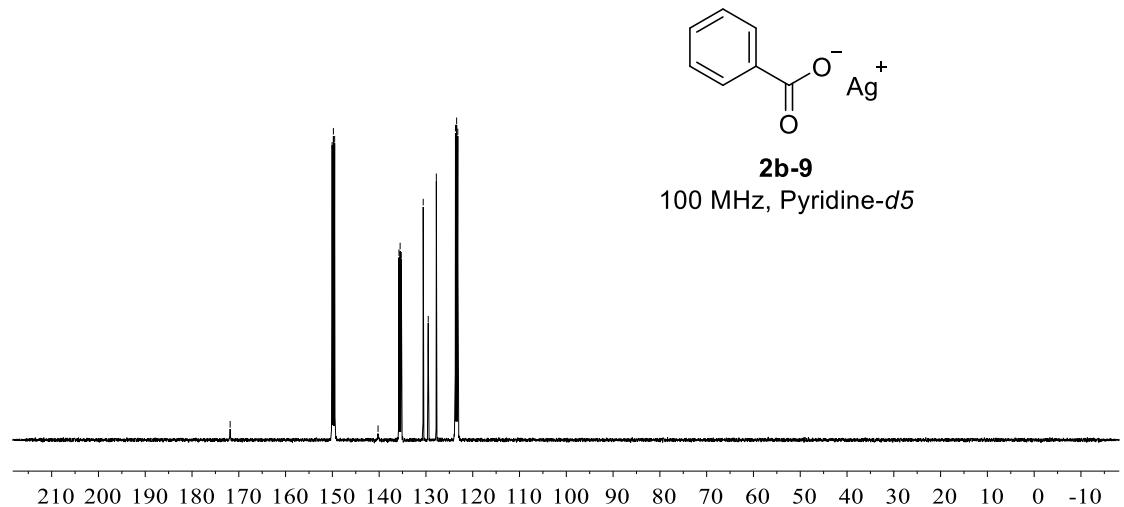
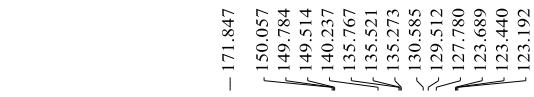
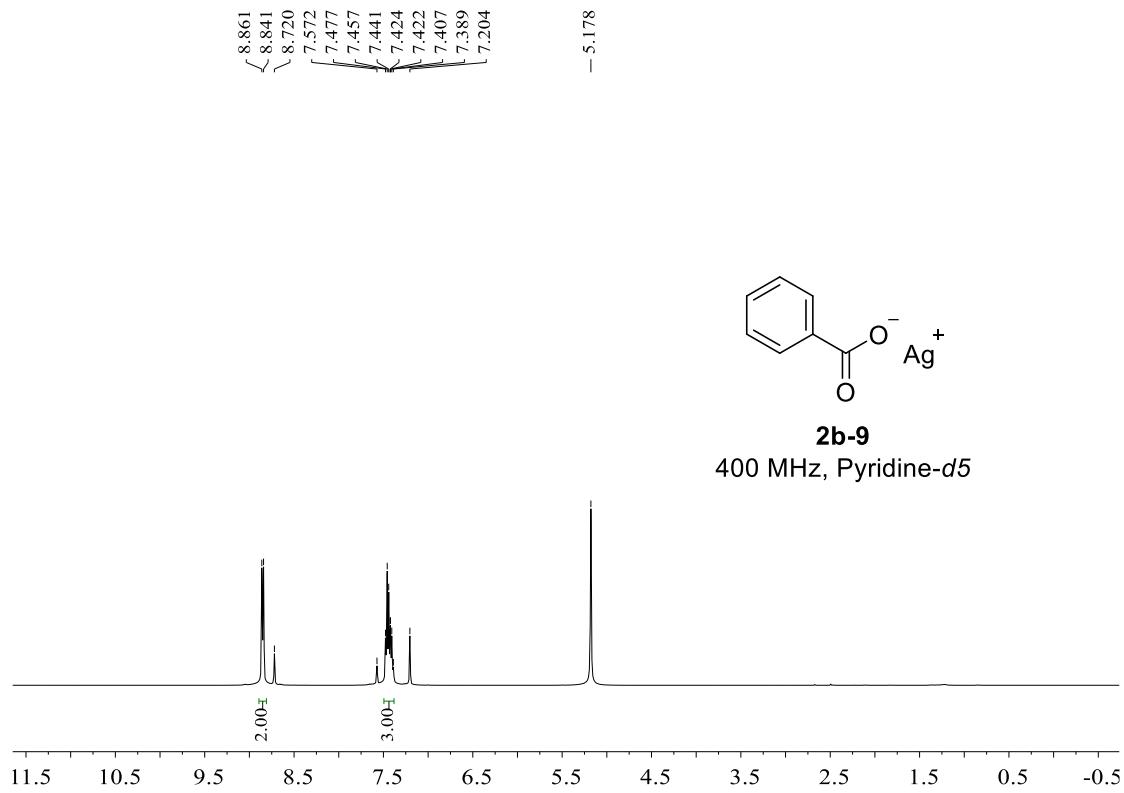


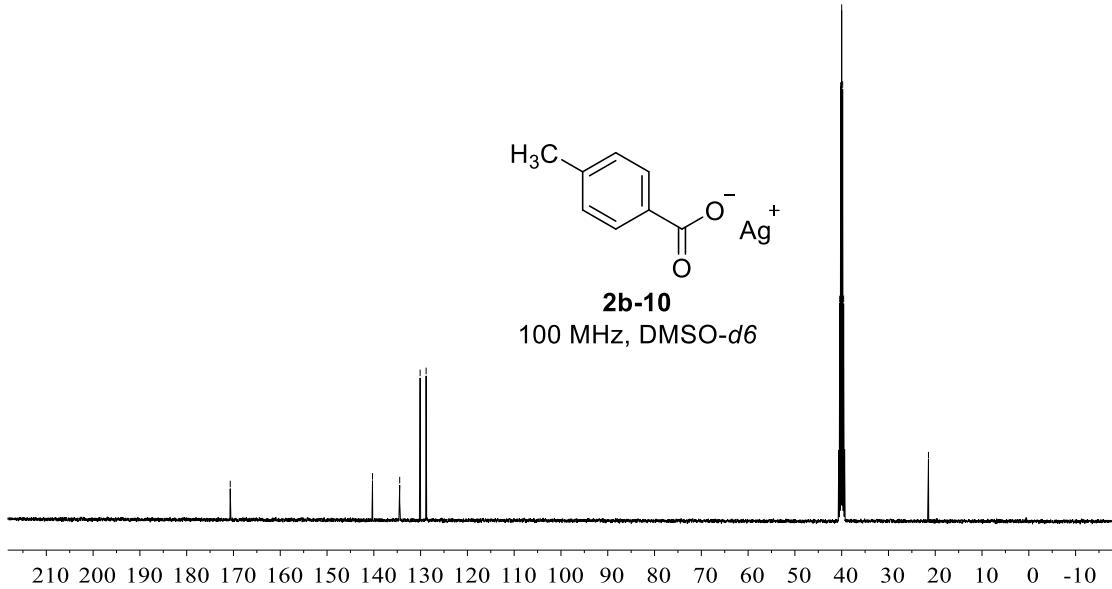
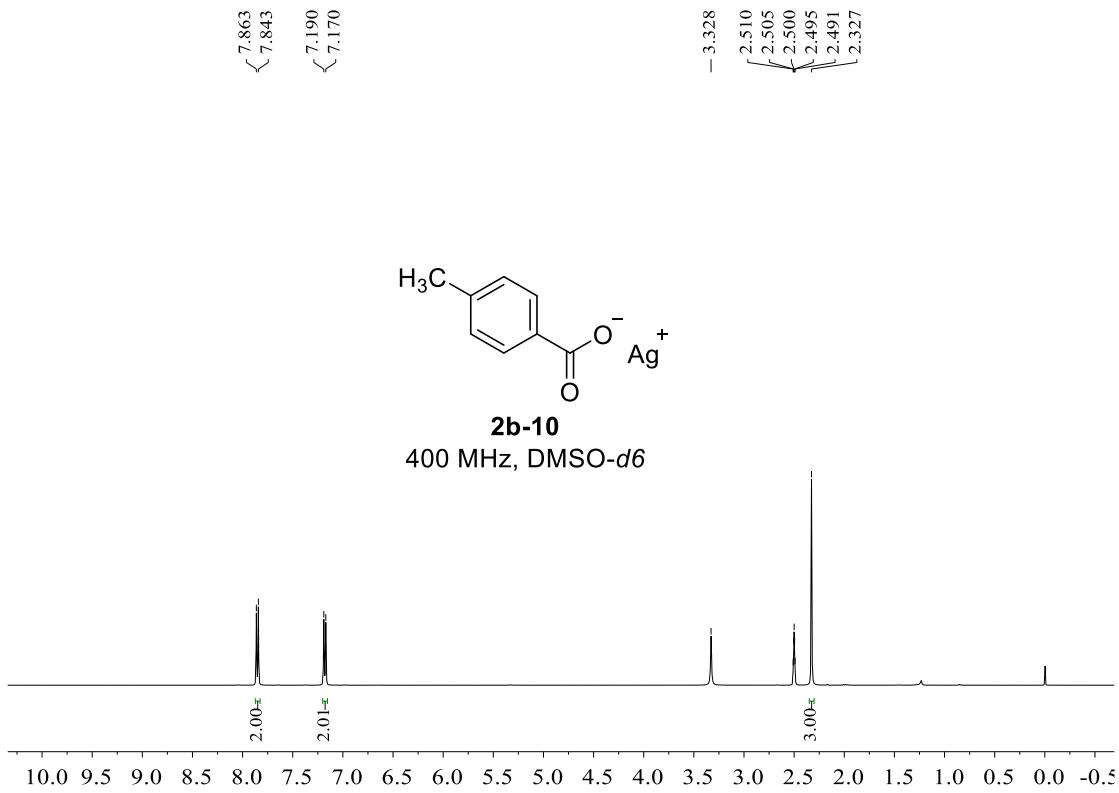


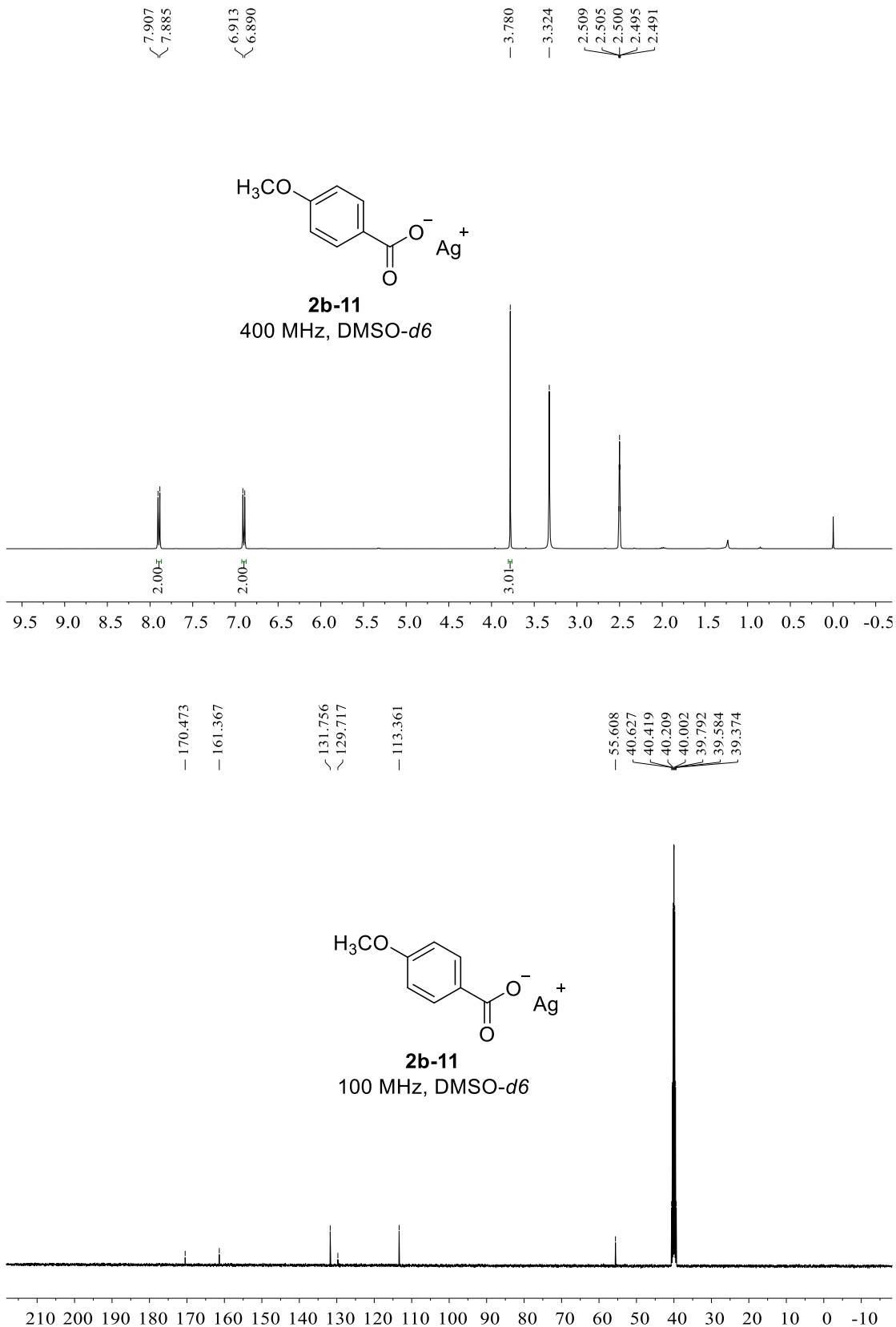


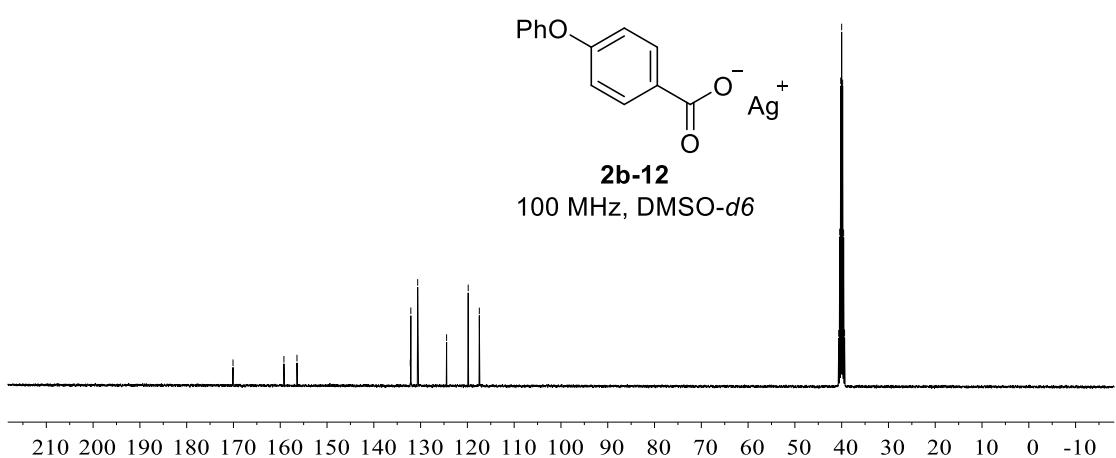
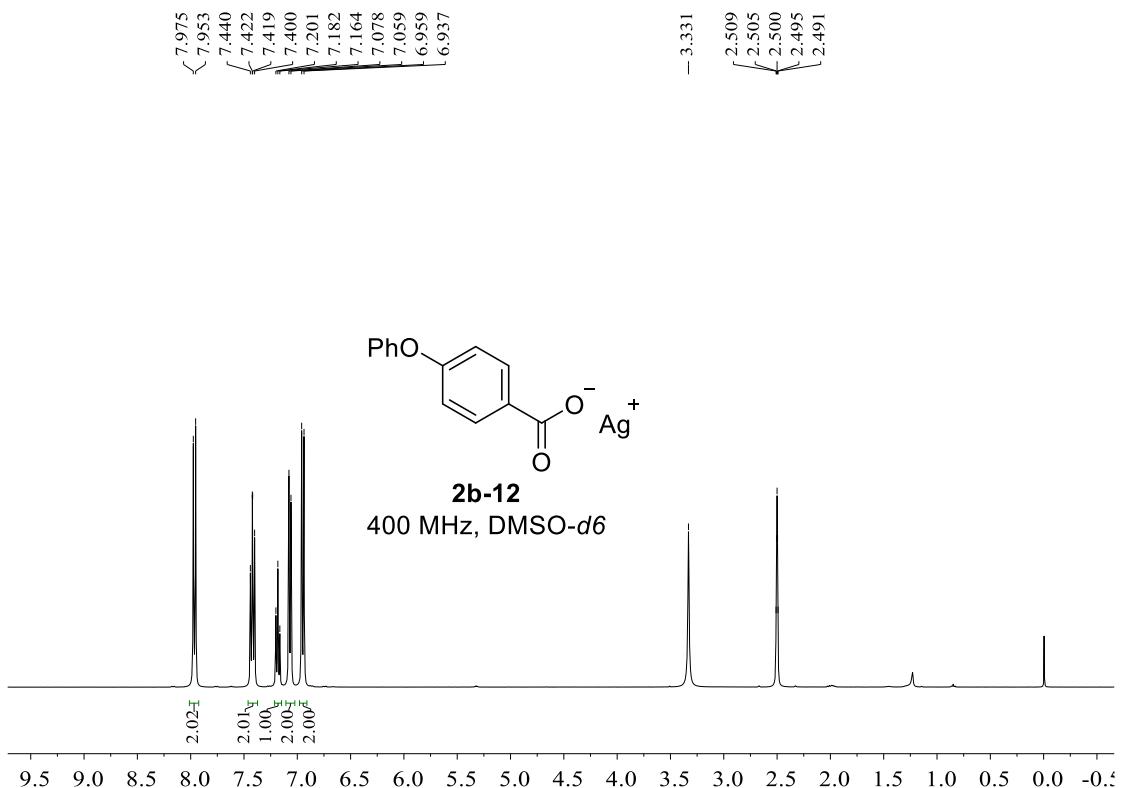


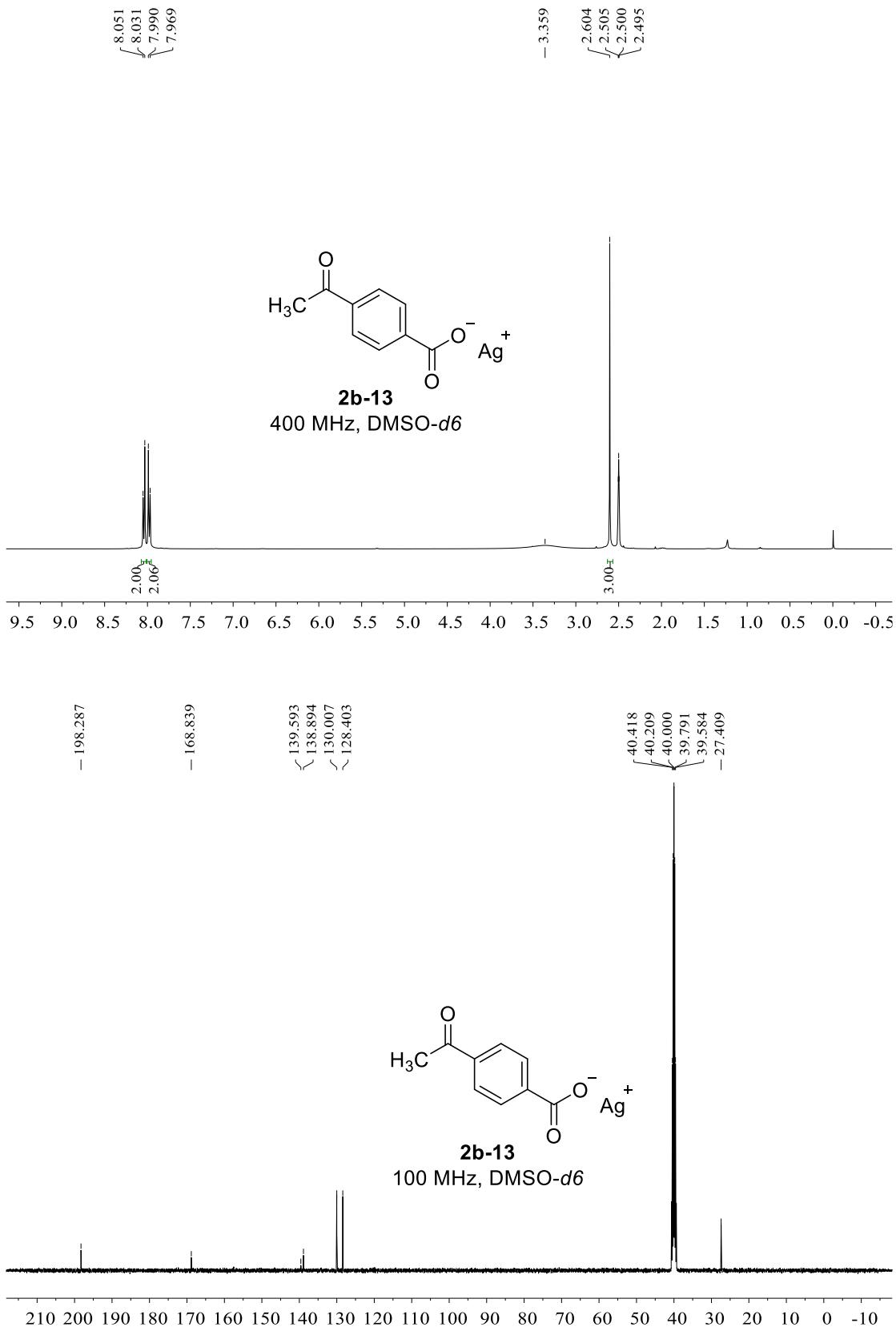


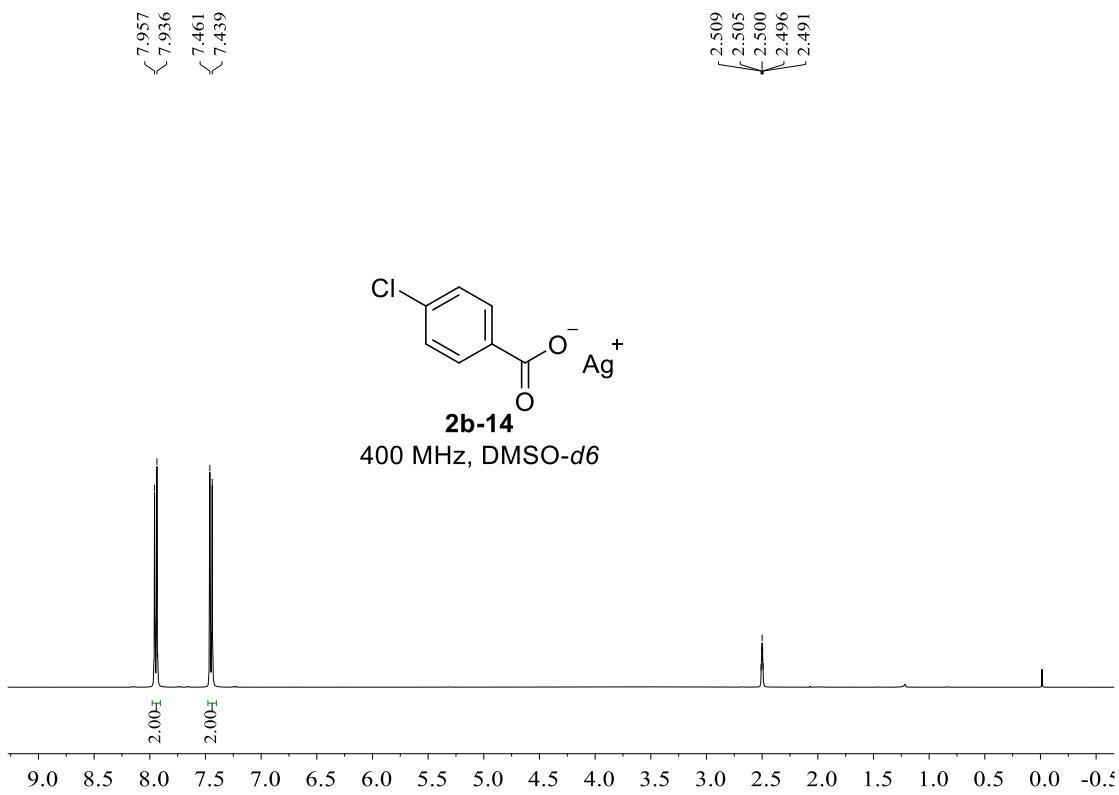


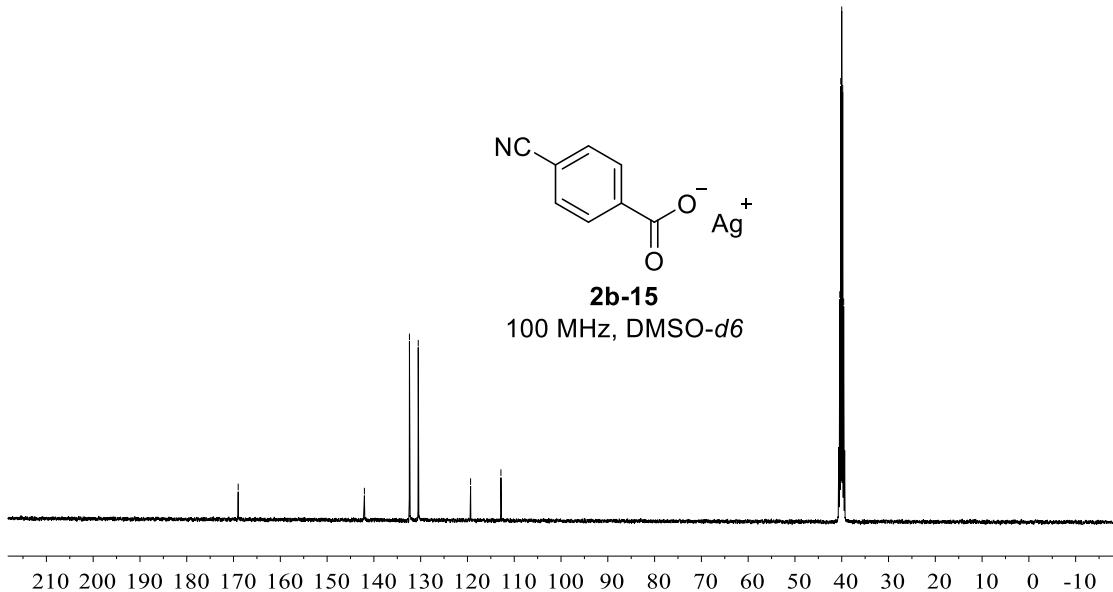
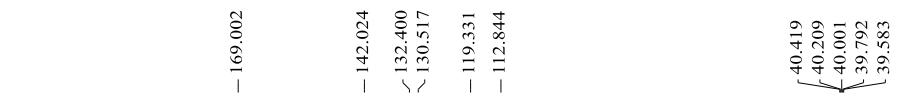
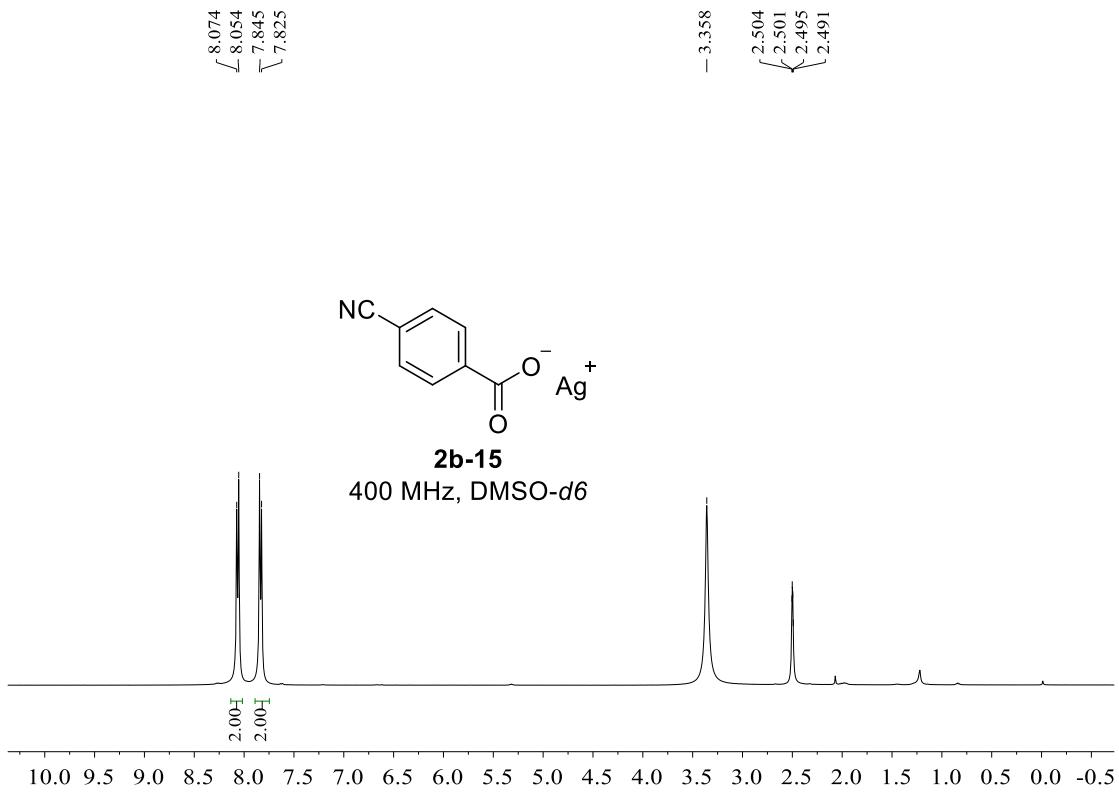


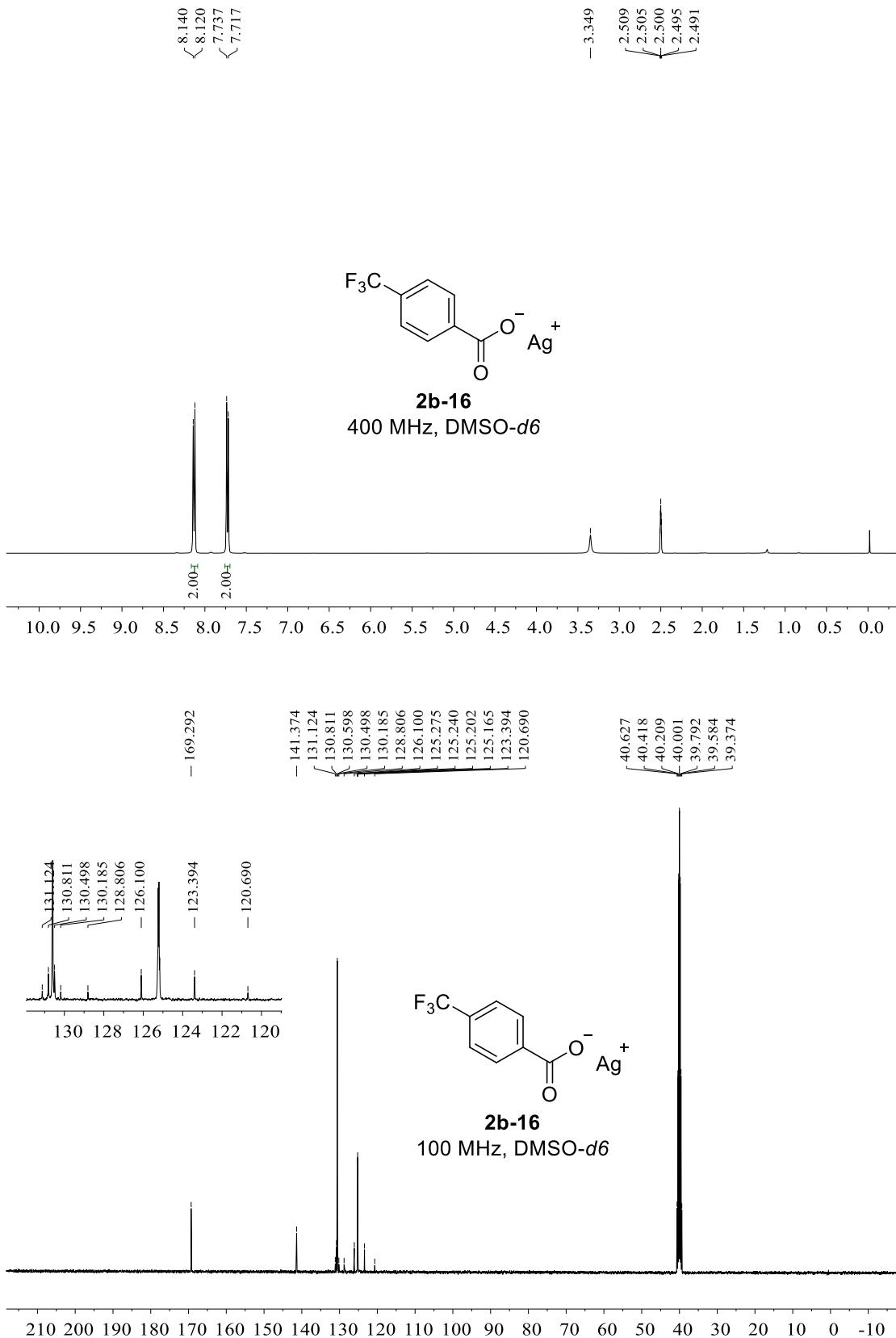




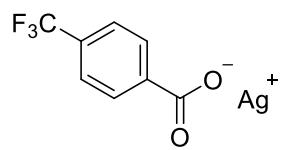




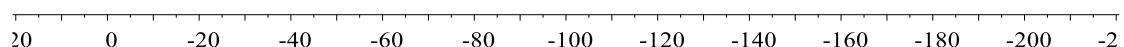


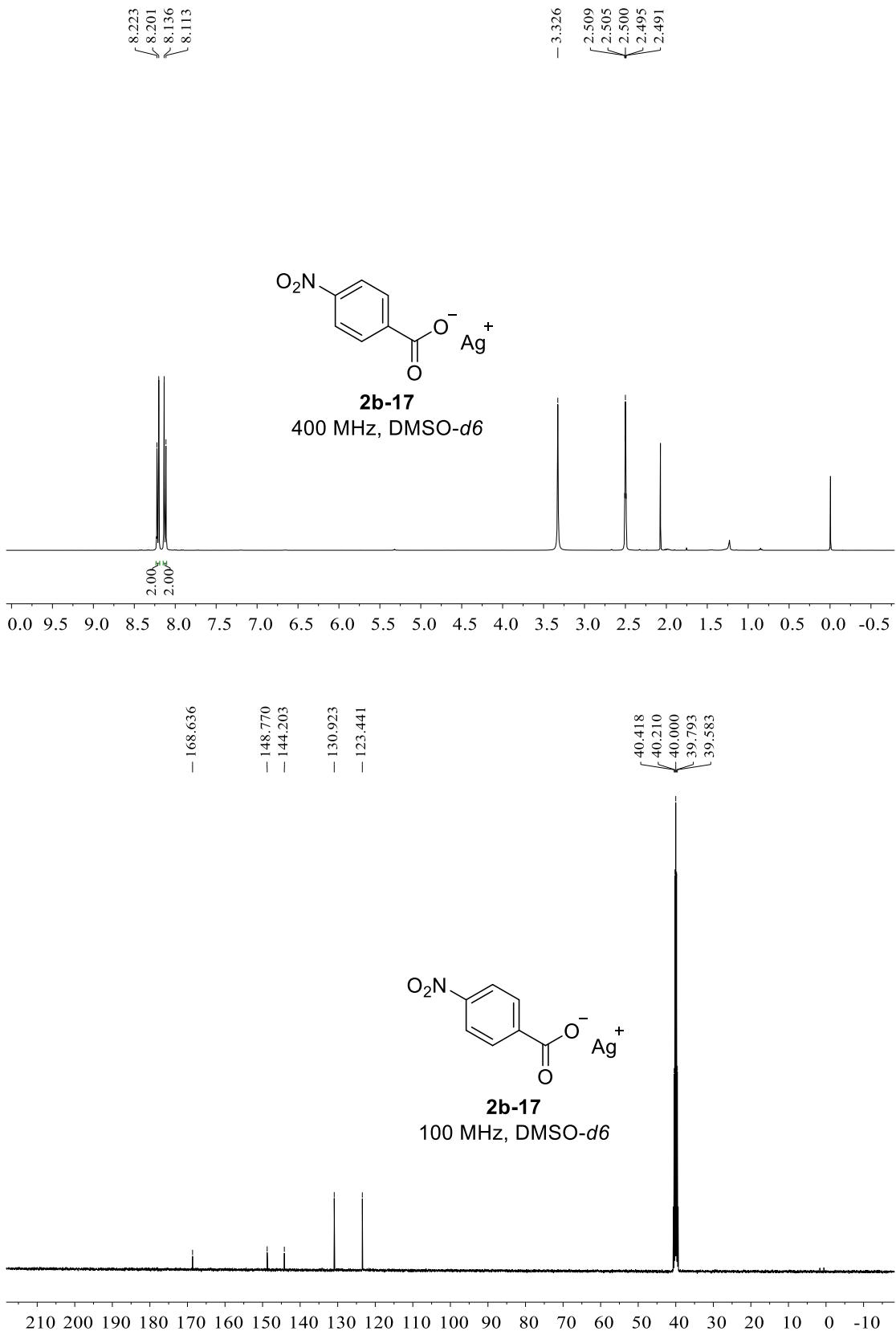


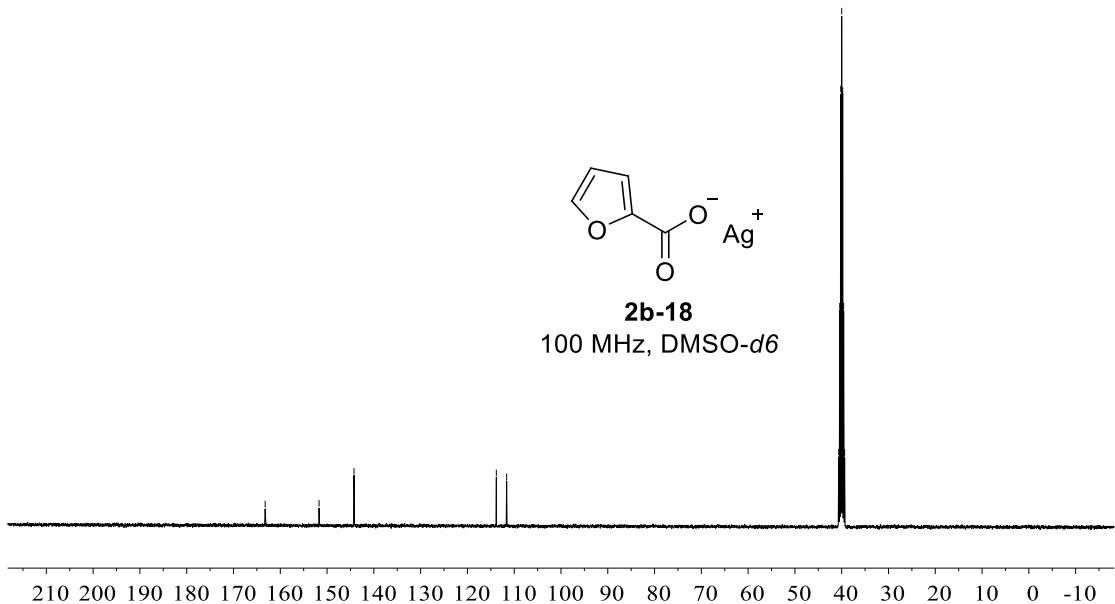
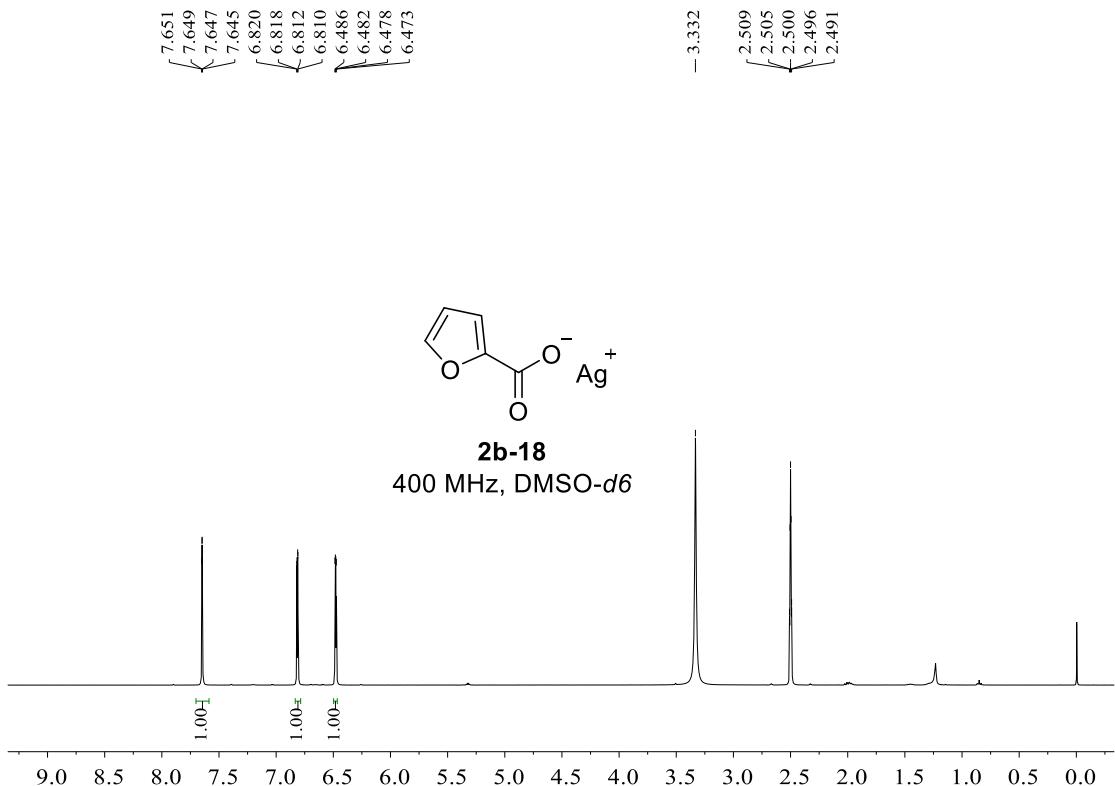
- -61.108

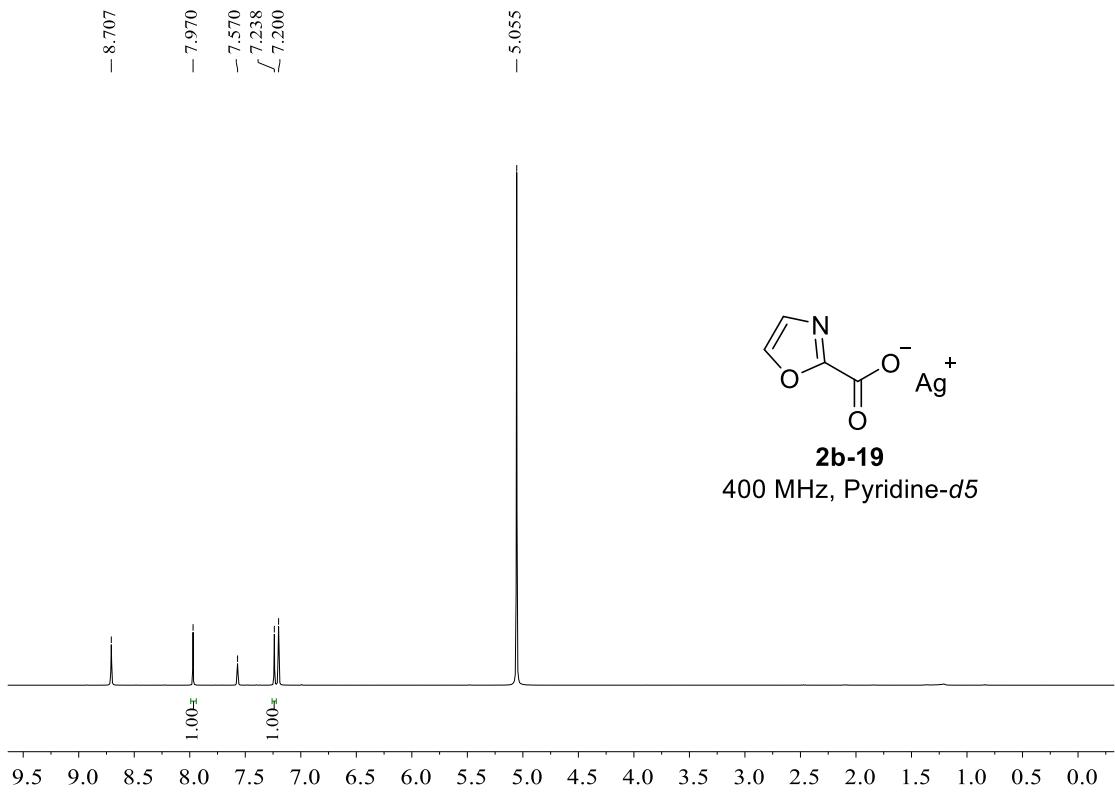


2b-16
376 MHz, DMSO-*d*6

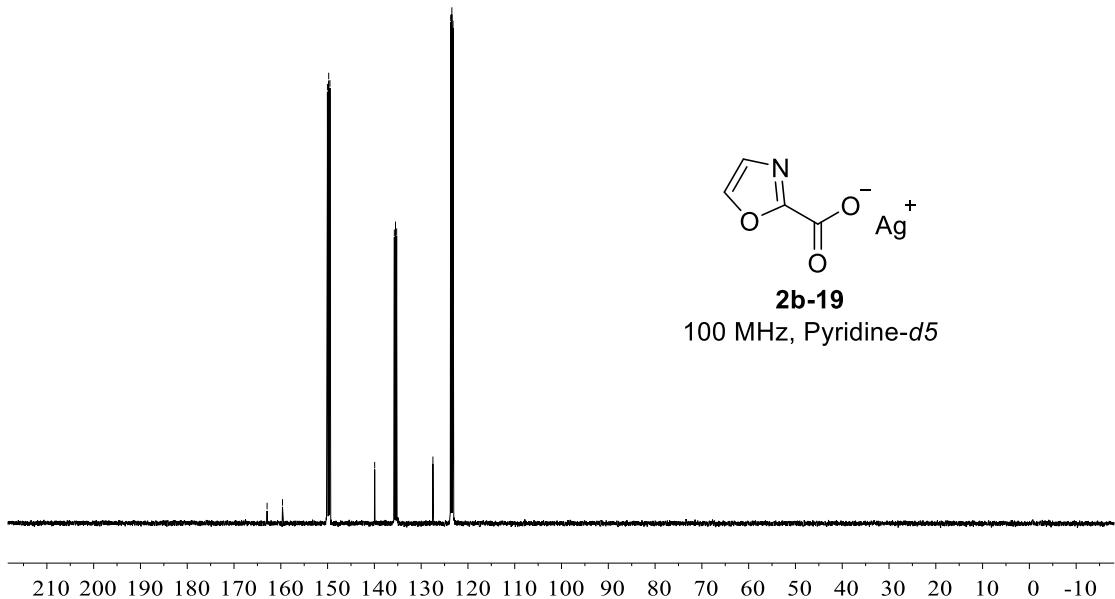


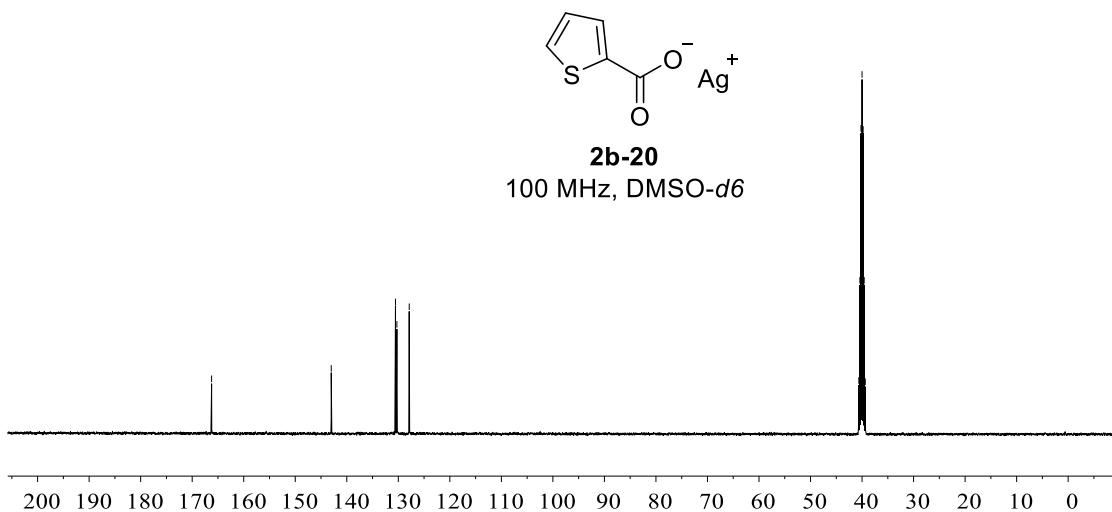
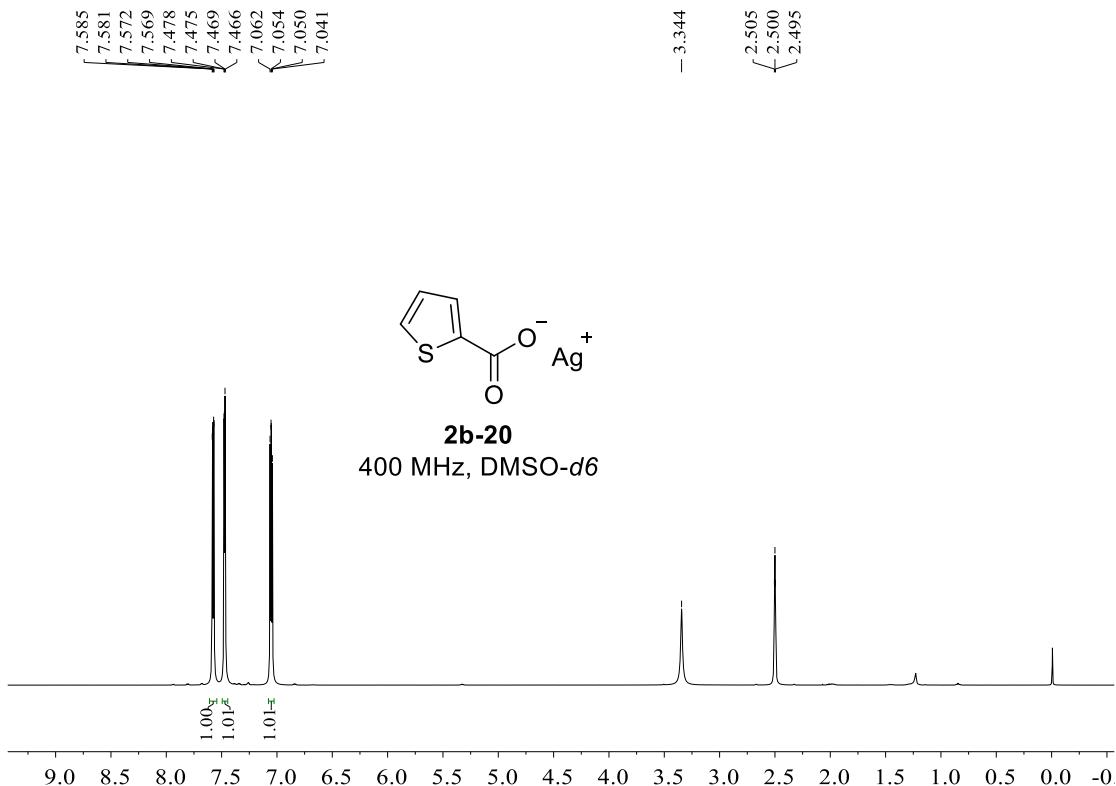


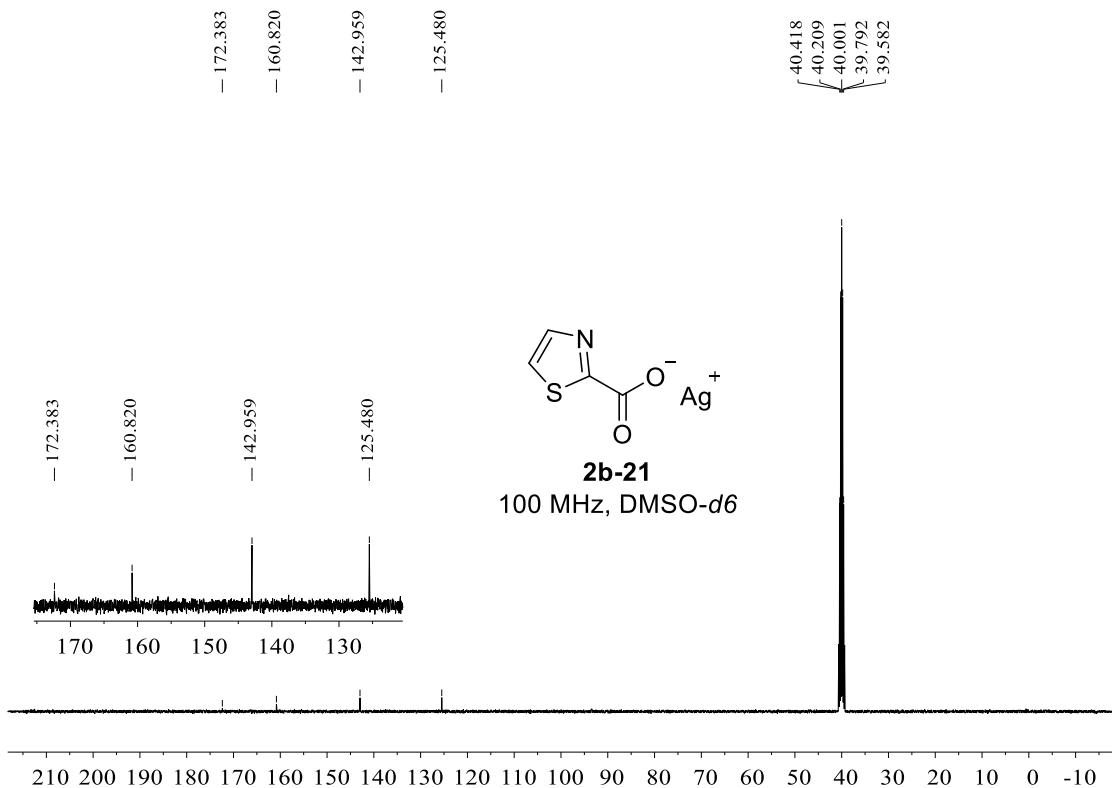
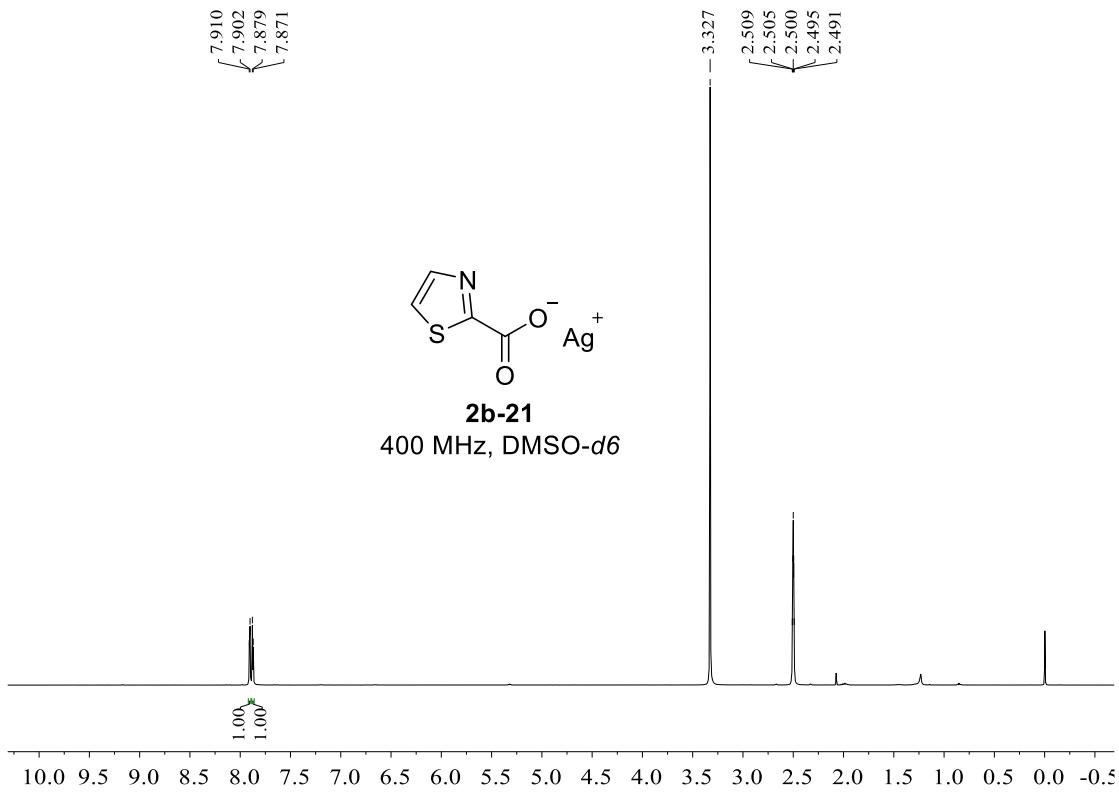


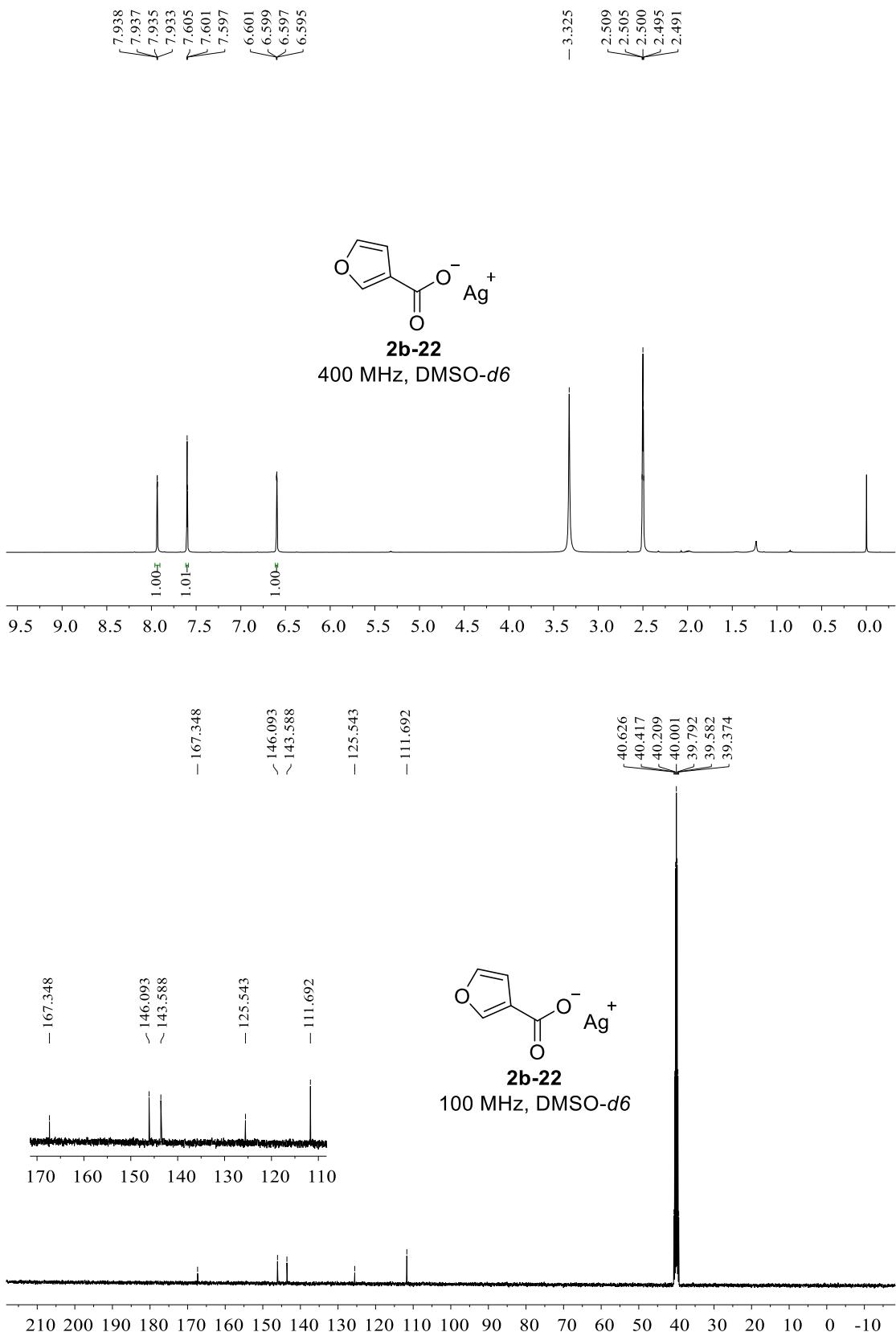


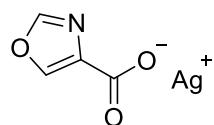
162.932
 159.643
 150.035
 149.765
 149.495
 139.934
 135.732
 135.485
 135.238
 127.487
 123.688
 123.440
 123.192





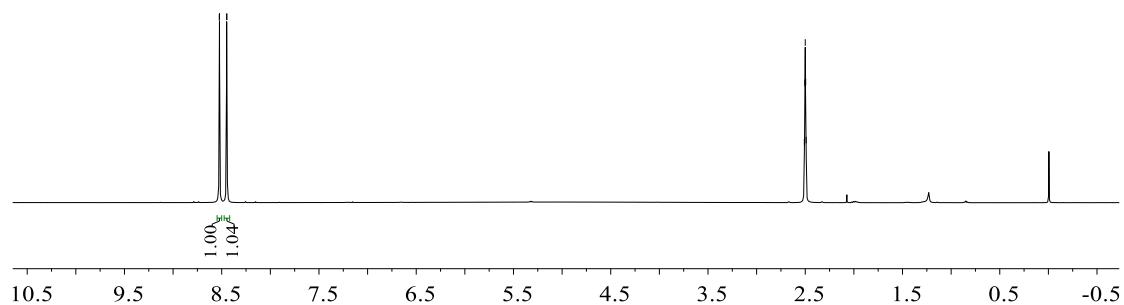






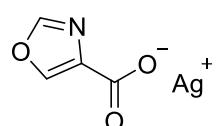
2b-23

400 MHz, DMSO-*d*6



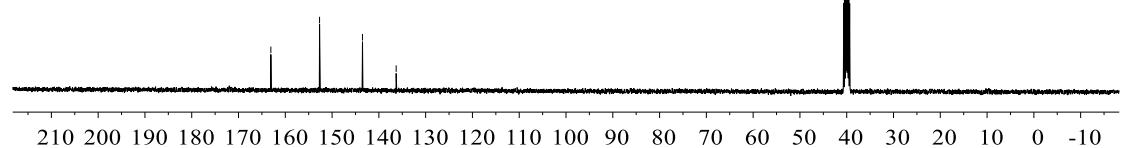
- 163.086
 \ 152.675
 \ 143.500
 / 136.301

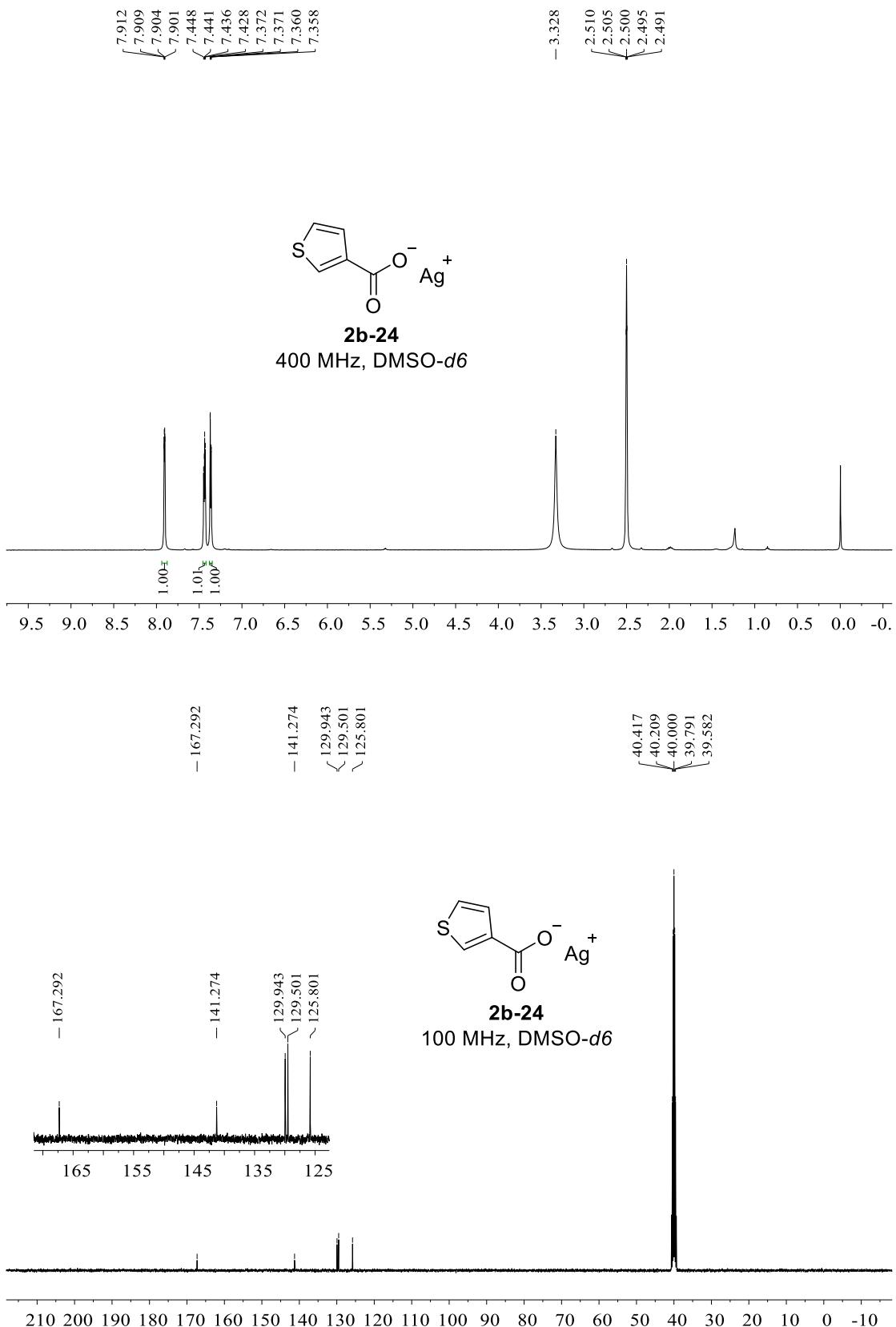
40.417
 40.210
 40.001
 39.791
 39.583

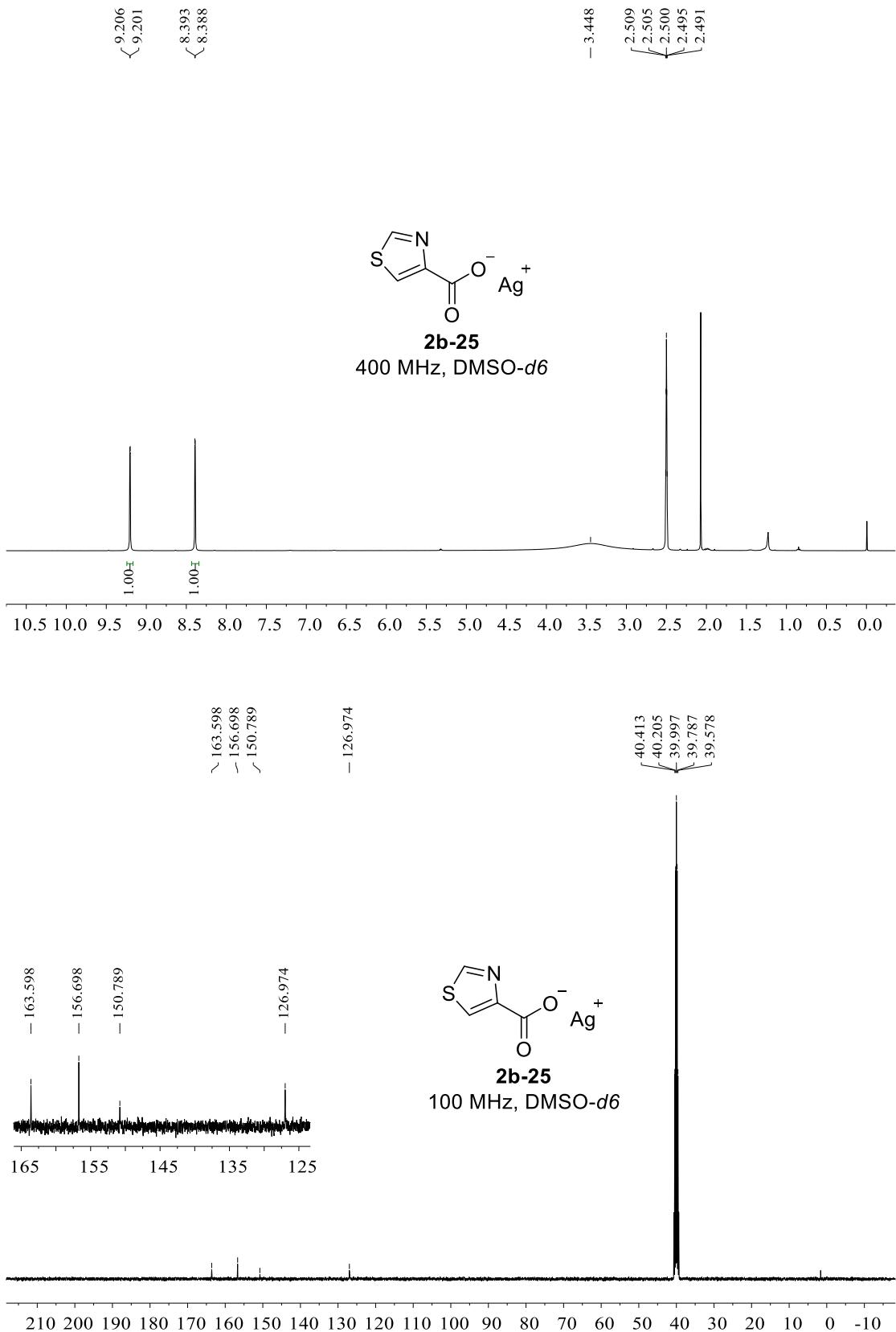


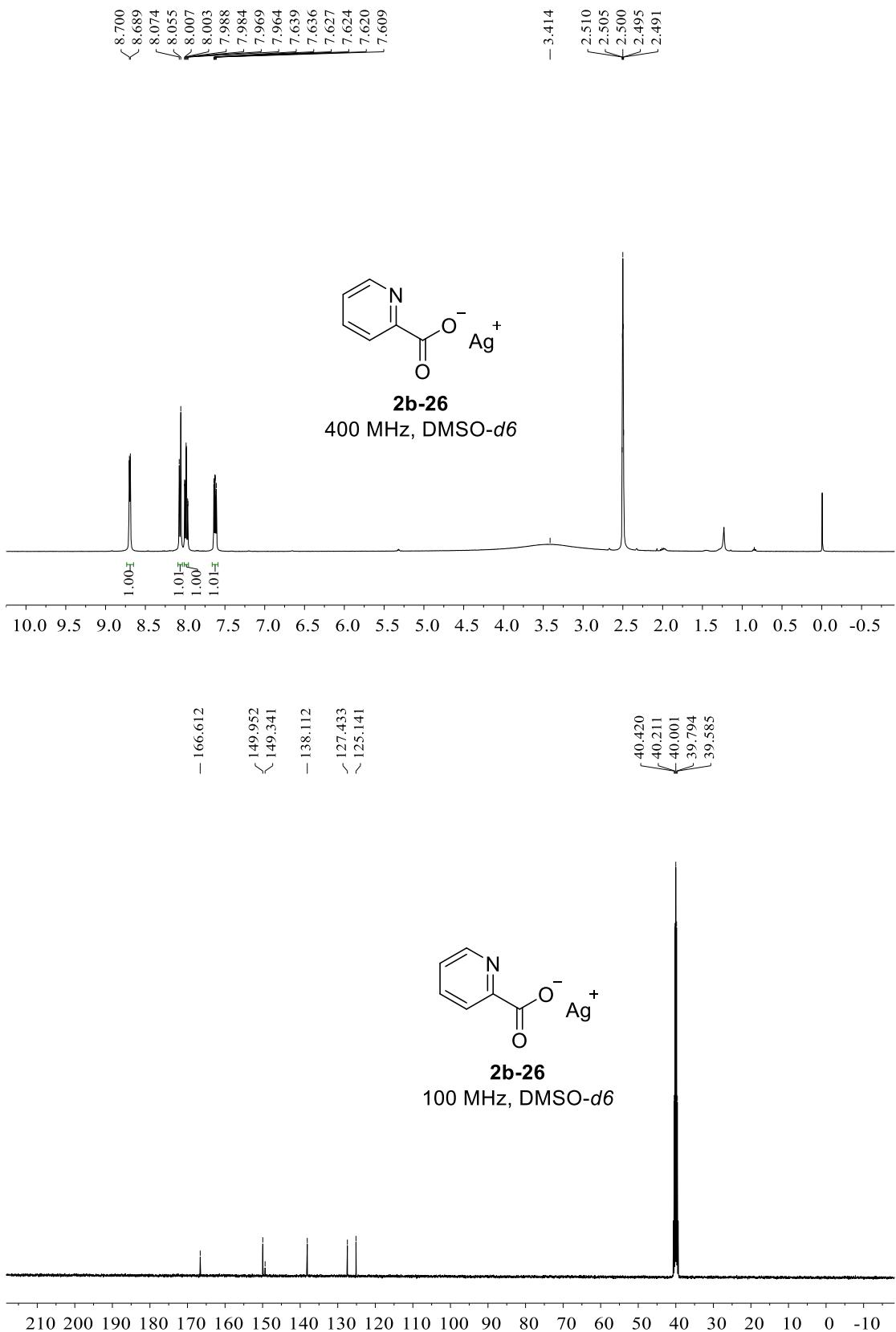
2b-23

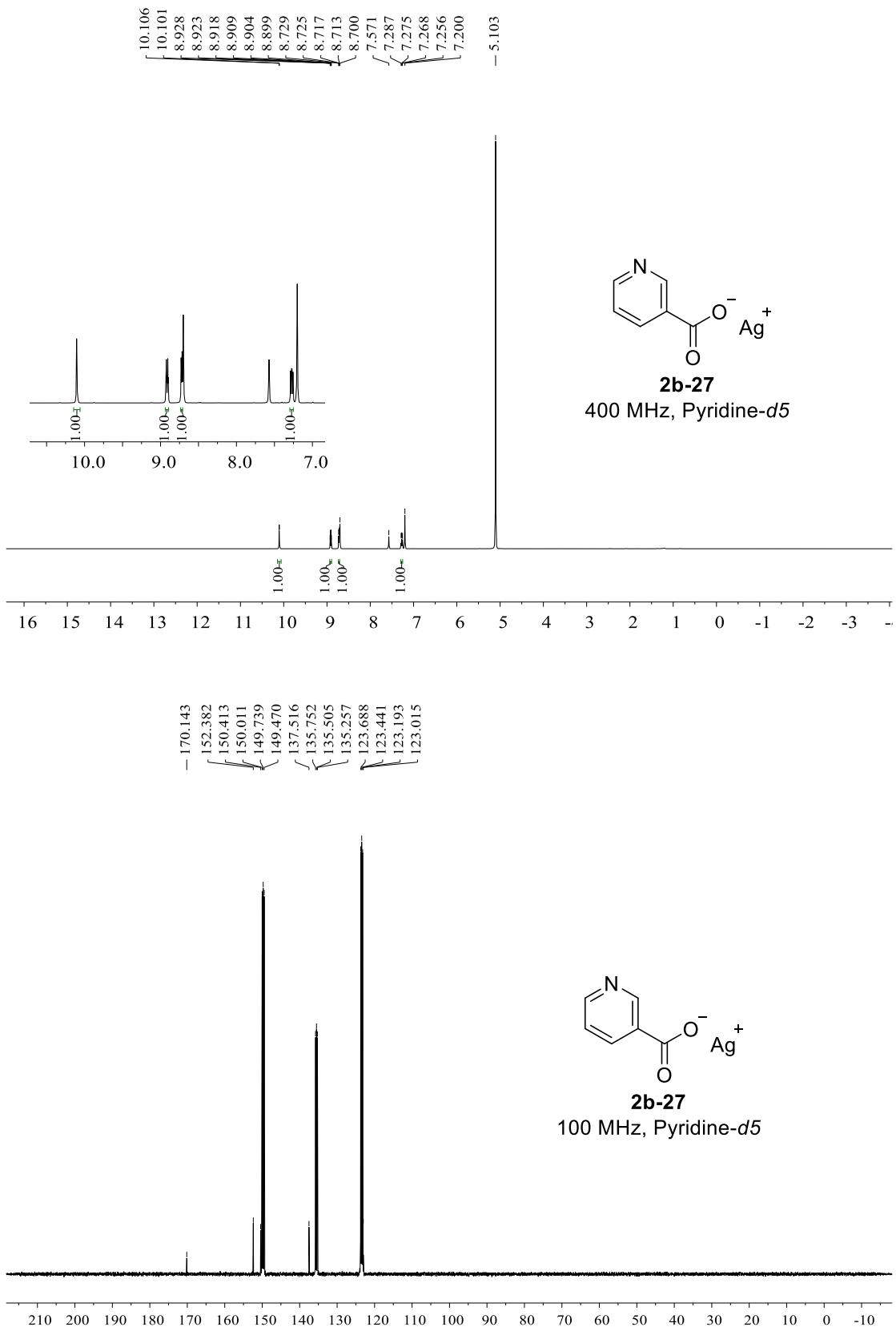
100 MHz, DMSO-*d*6

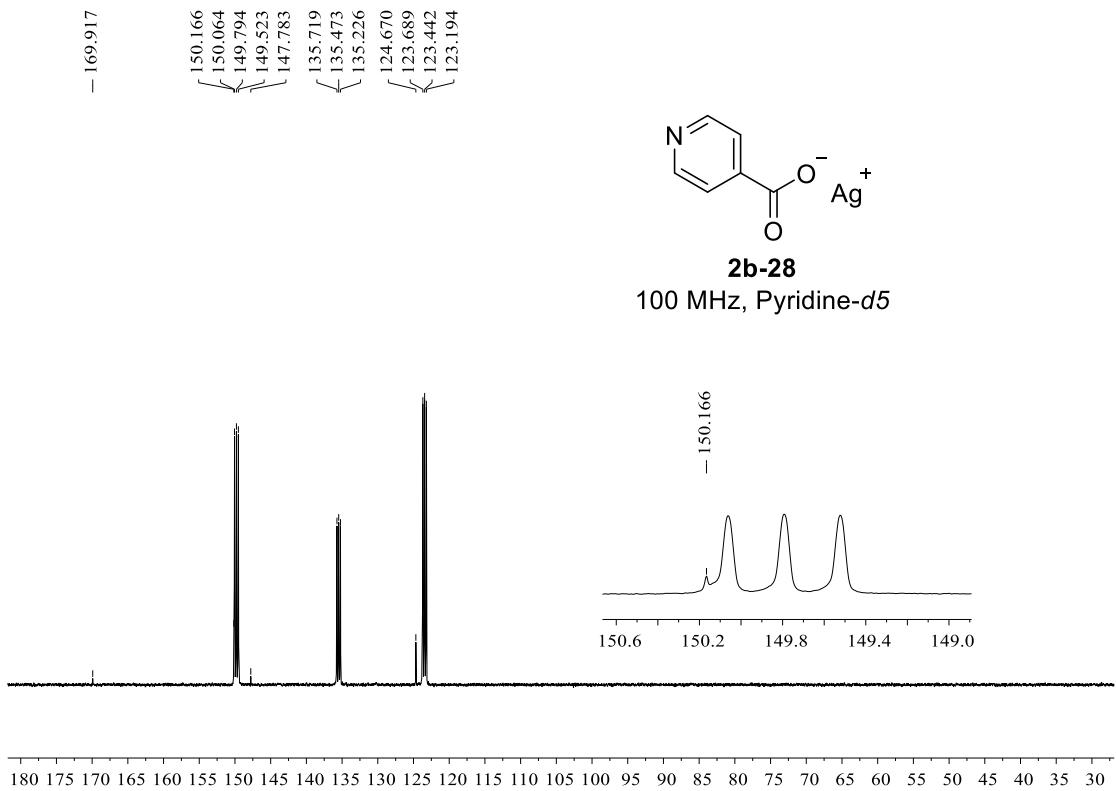
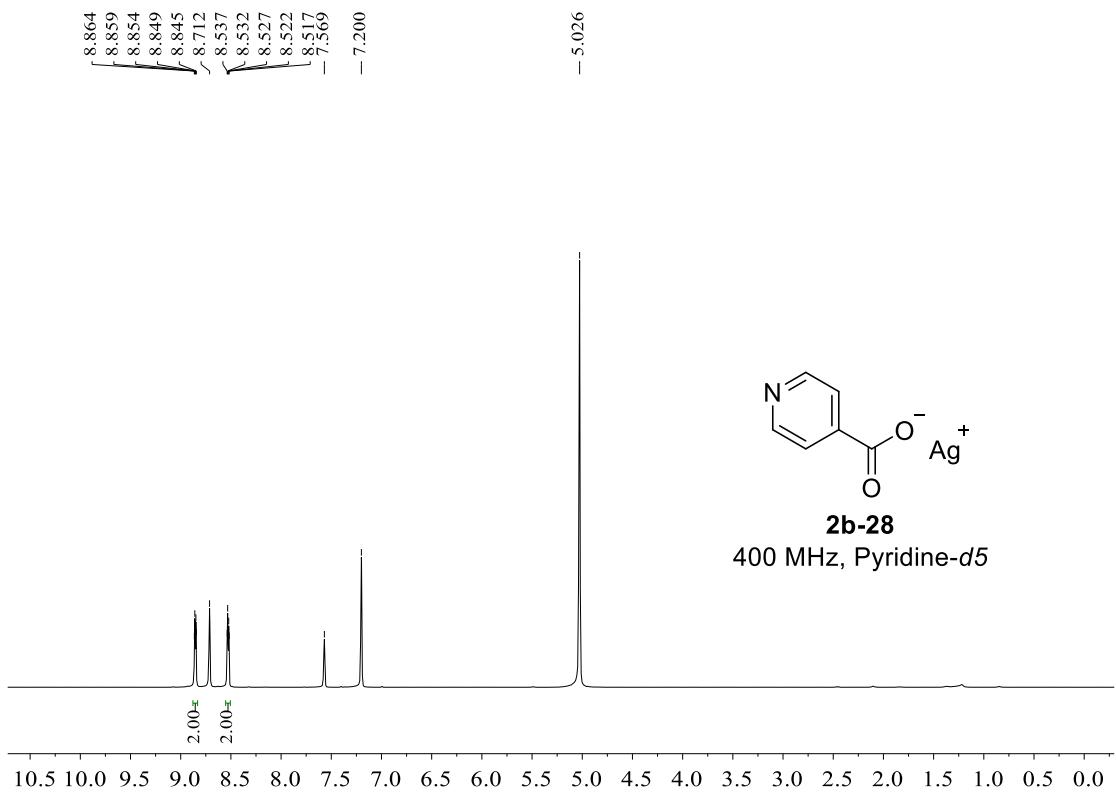


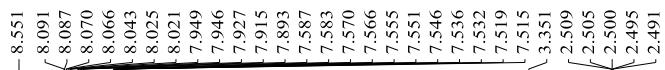




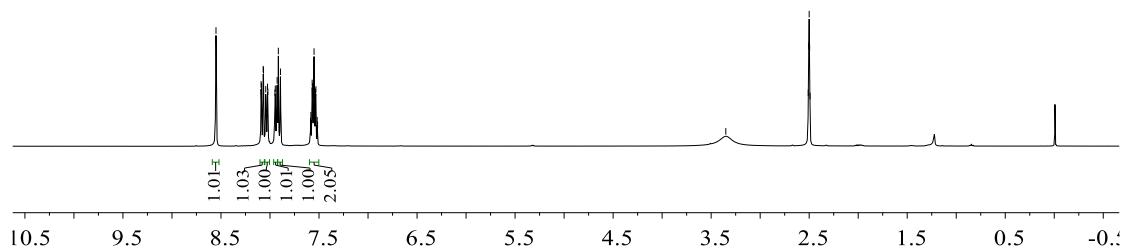




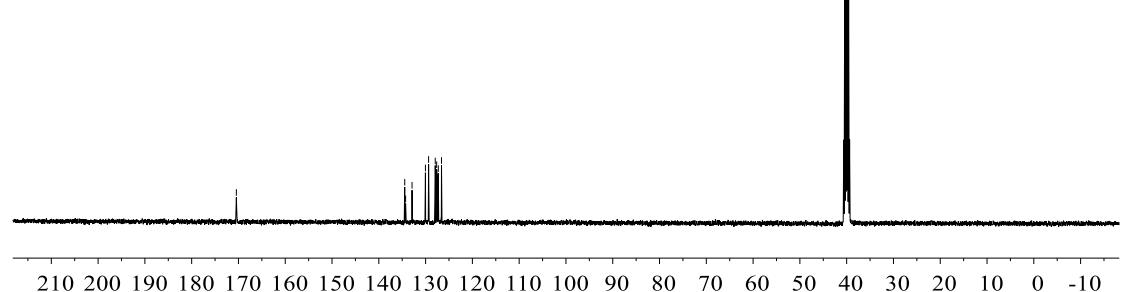


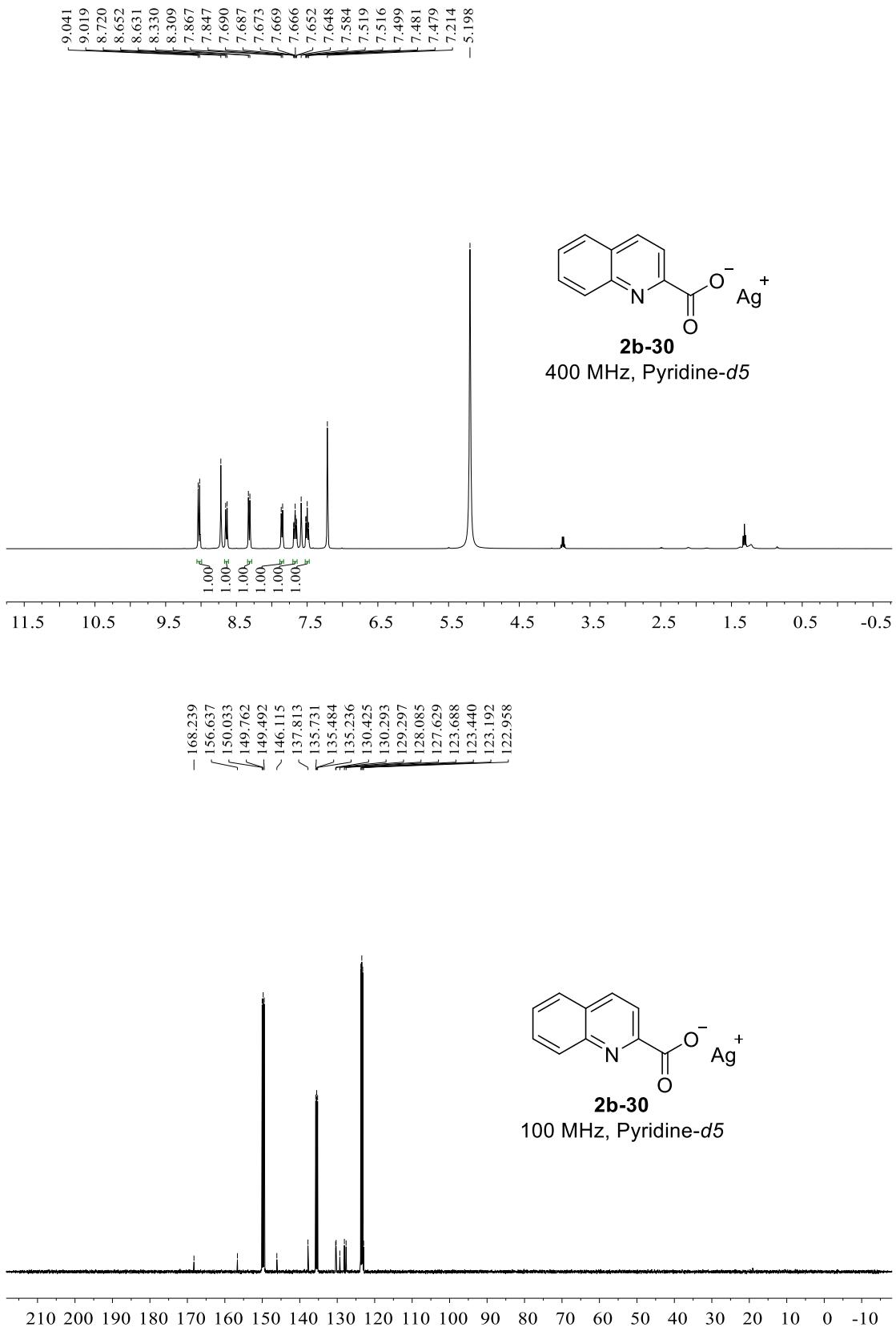


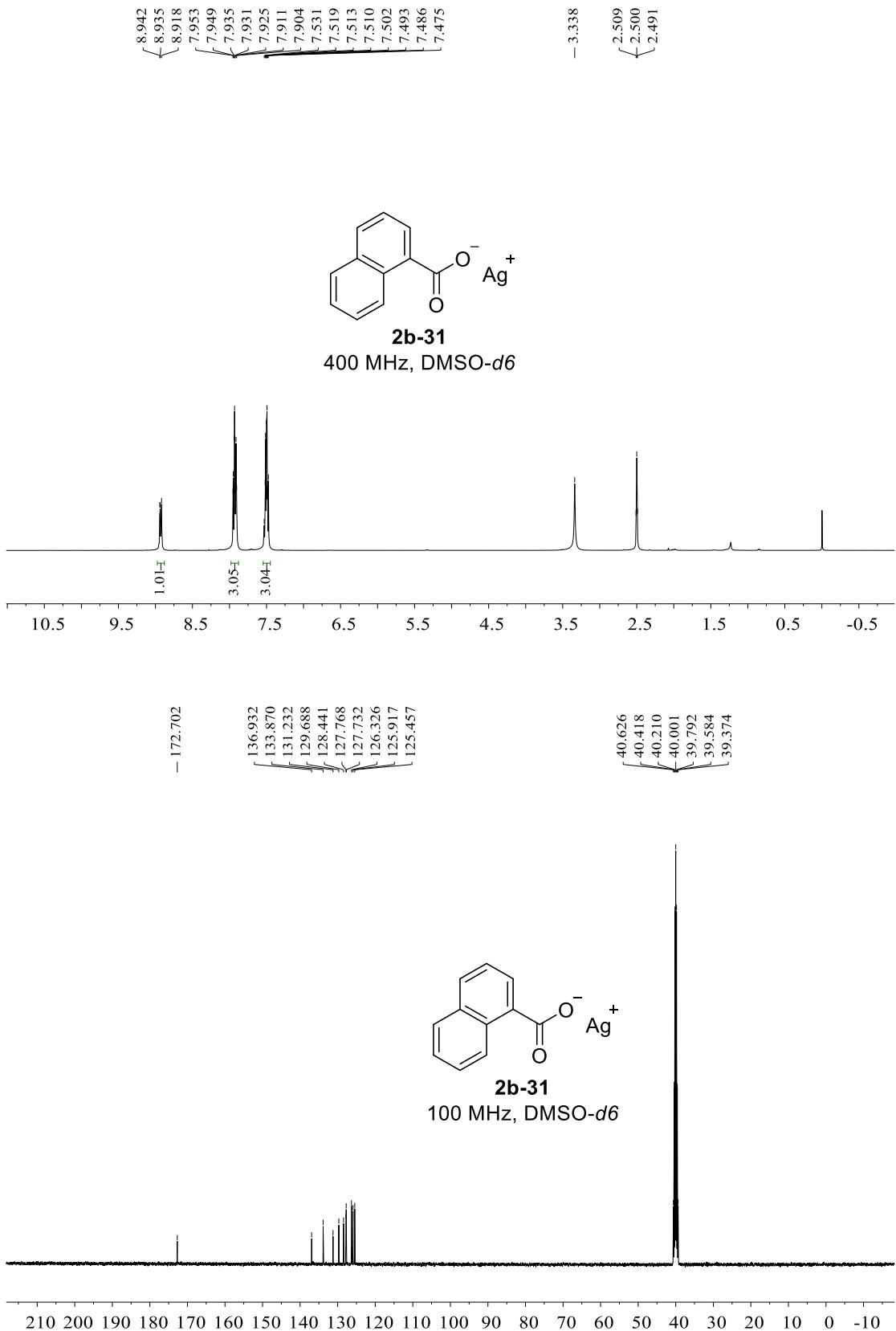
2b-29
400 MHz, DMSO-*d*6

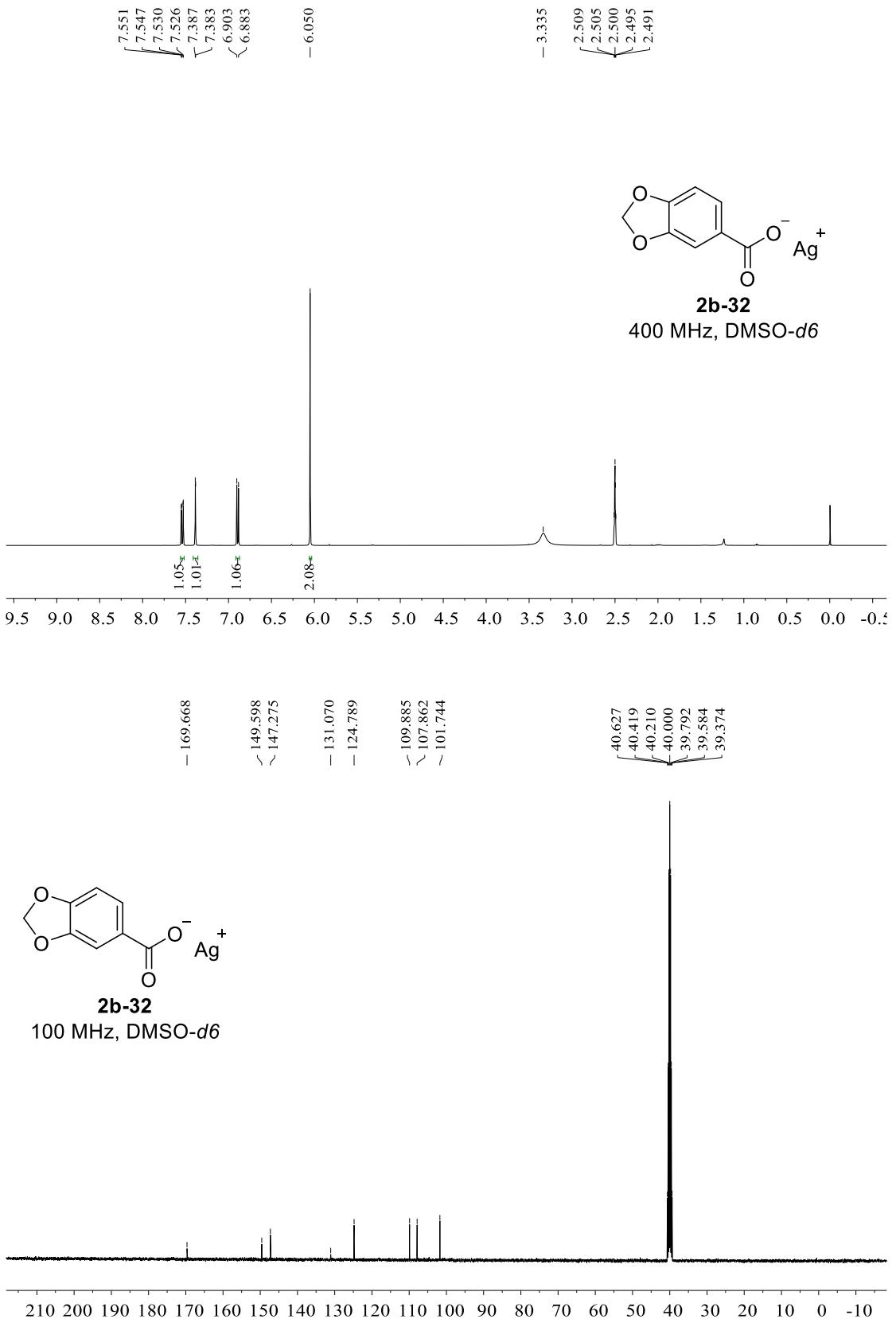


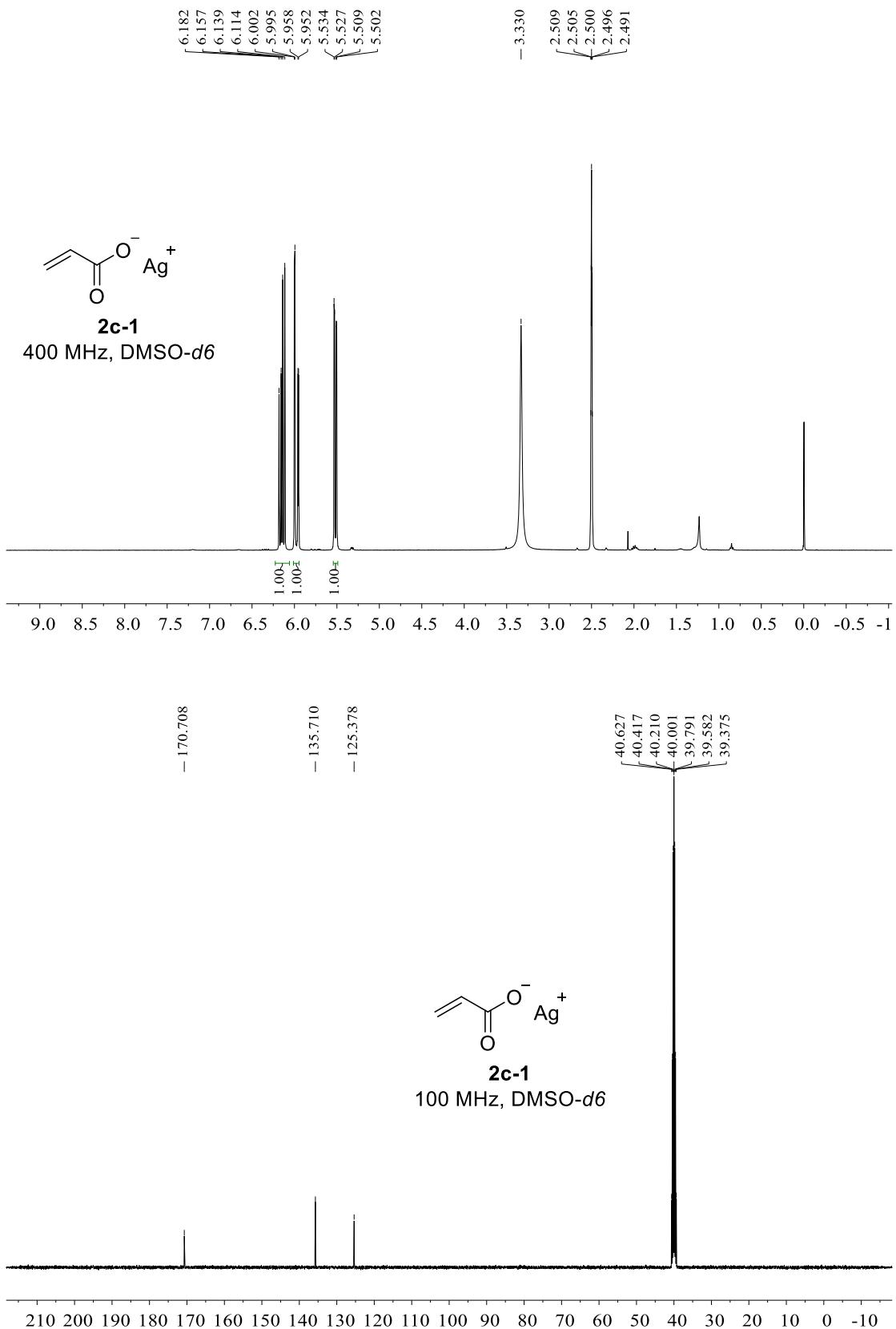
2b-29
100 MHz, DMSO-*d*6

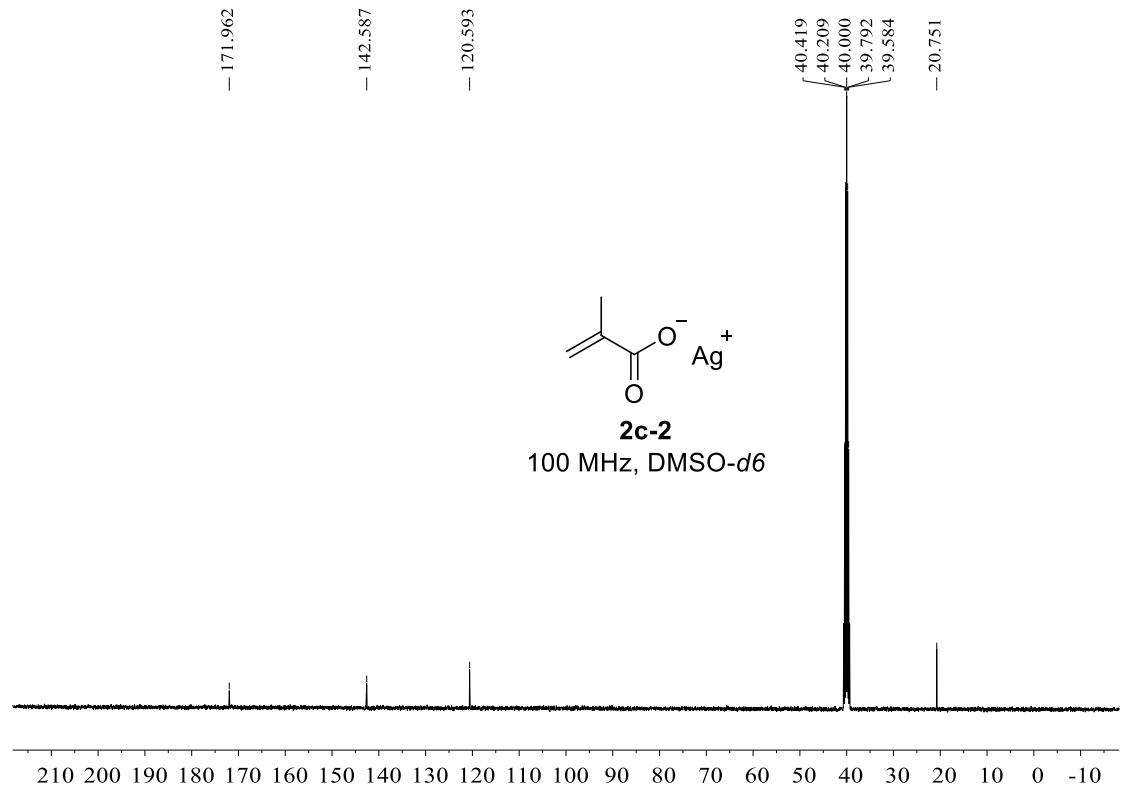
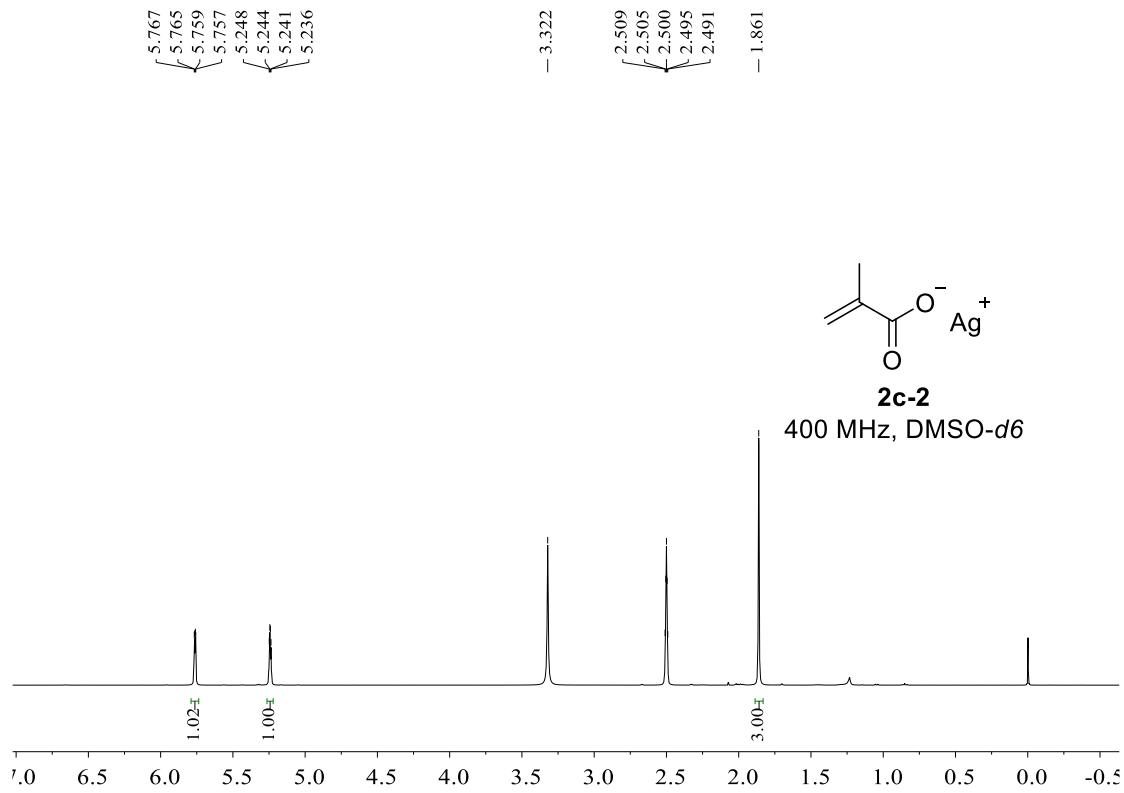


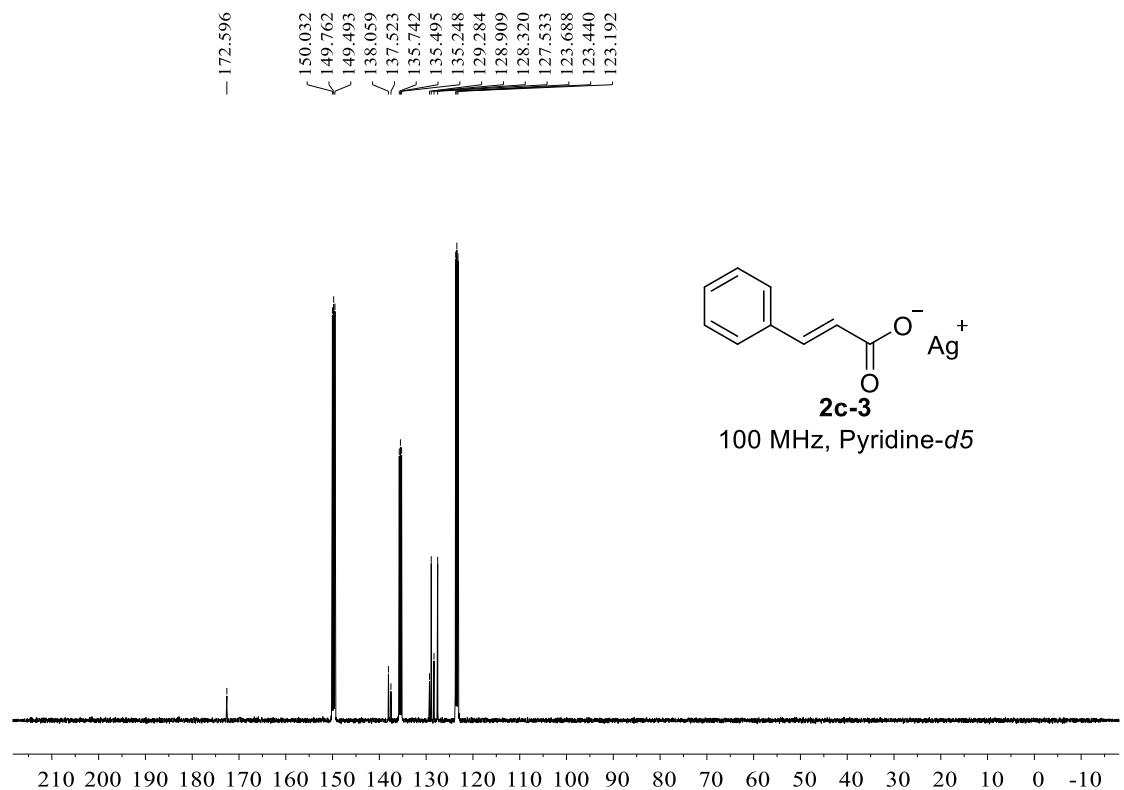
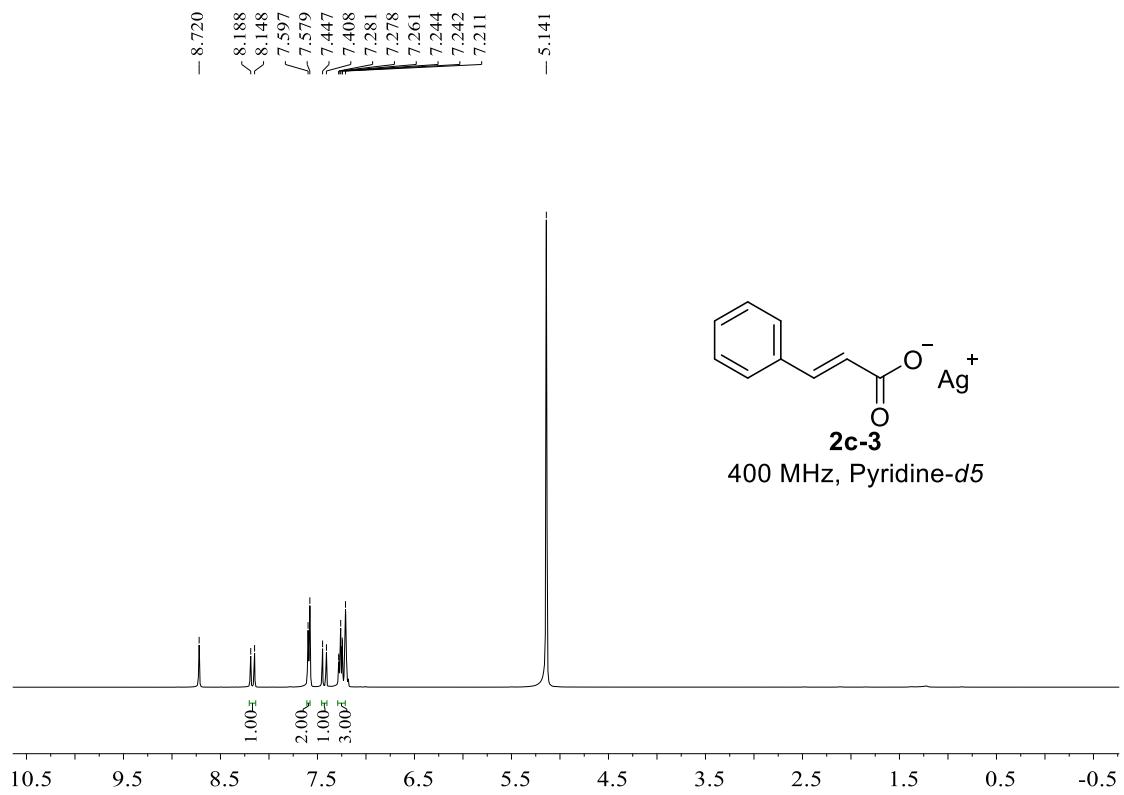


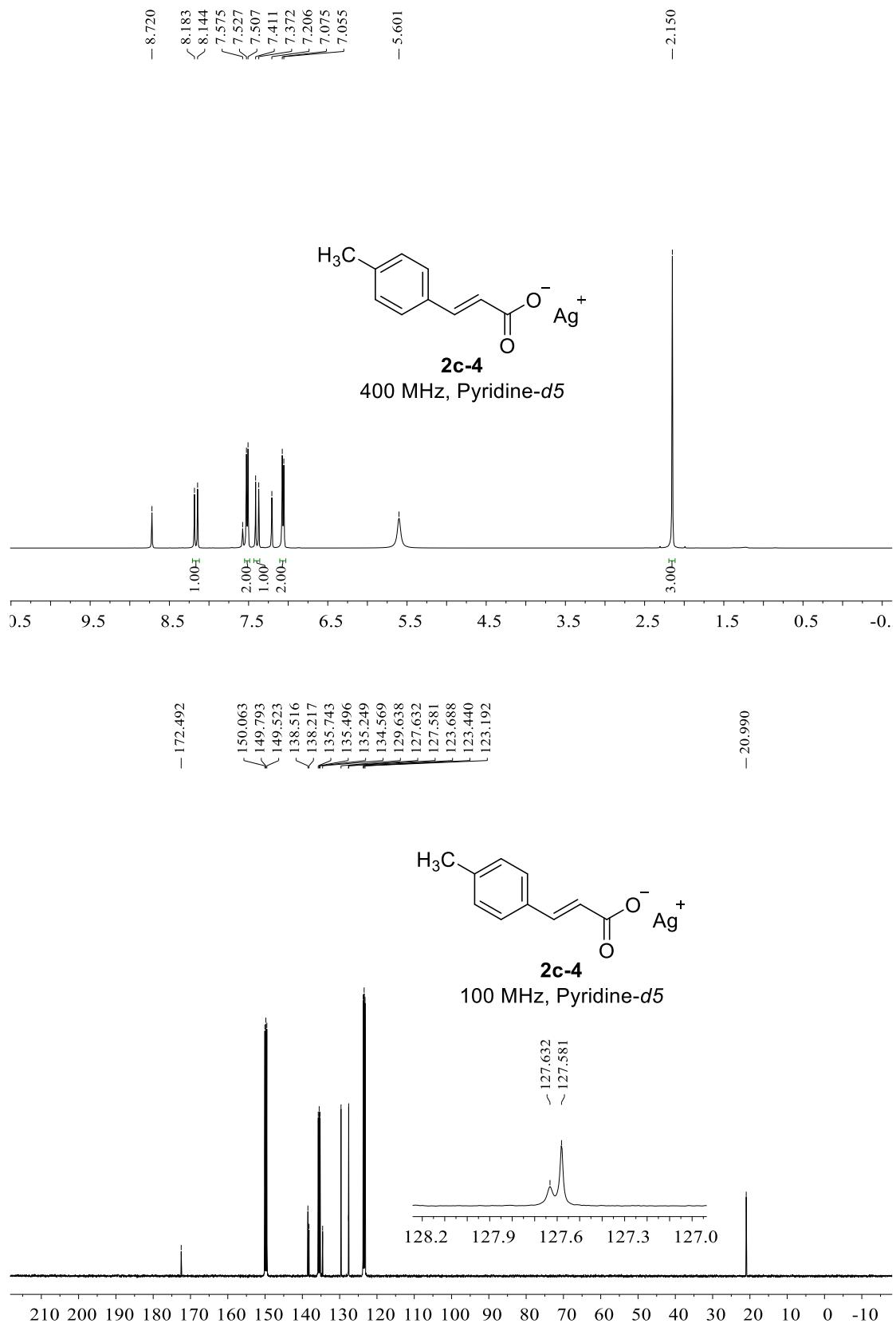


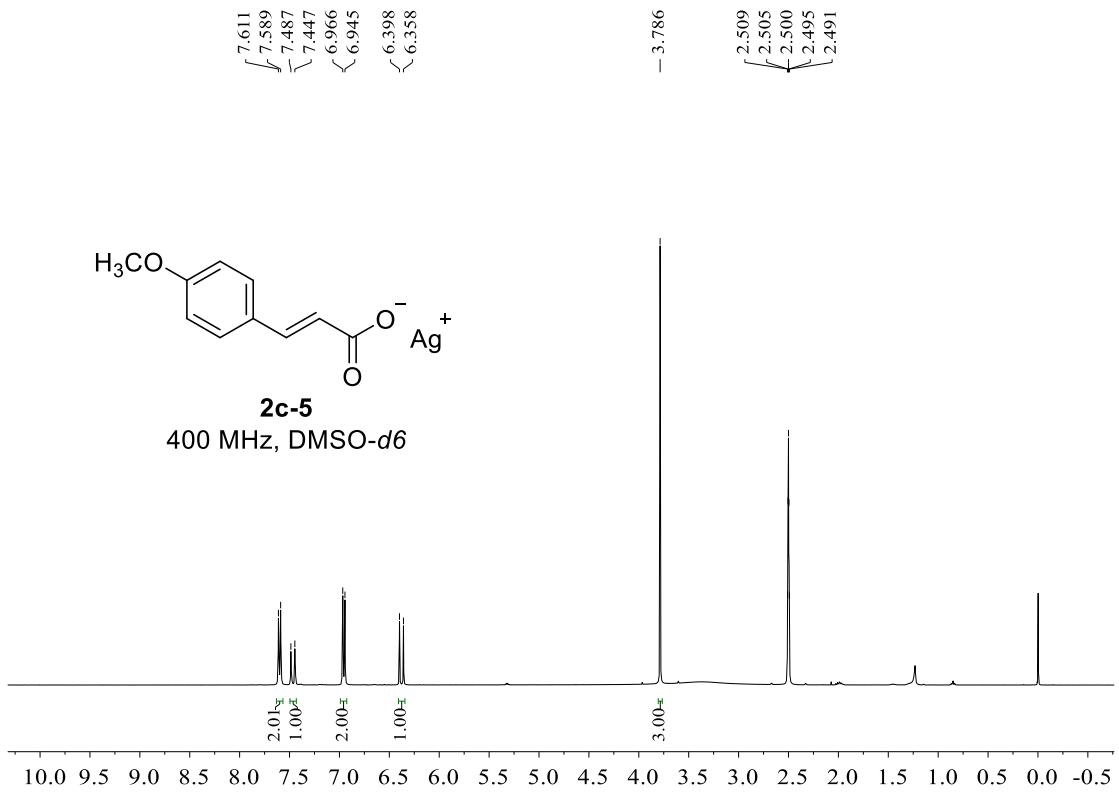


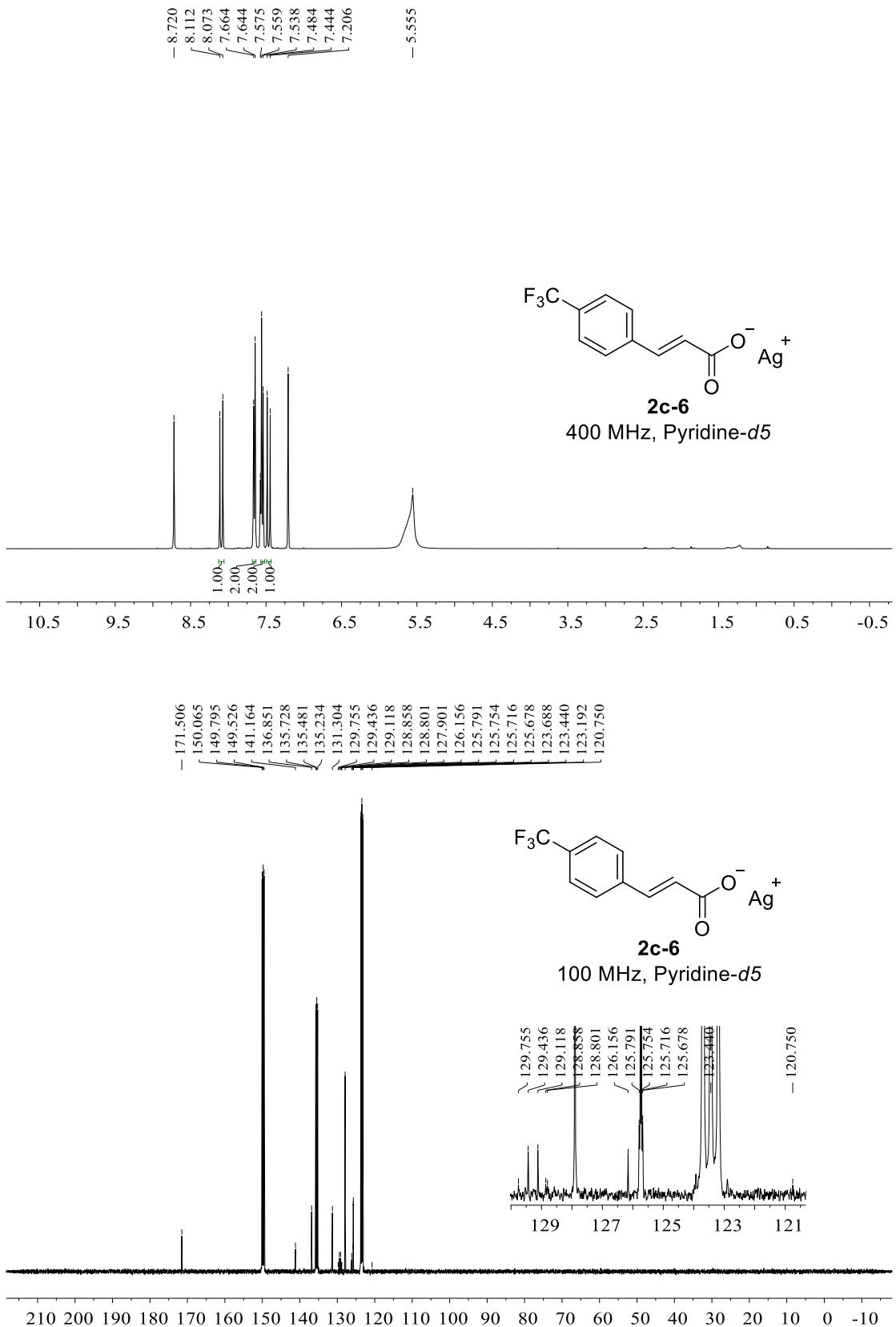




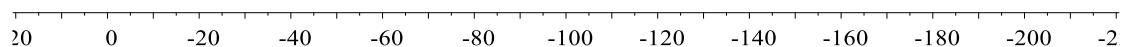
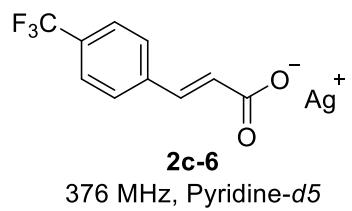


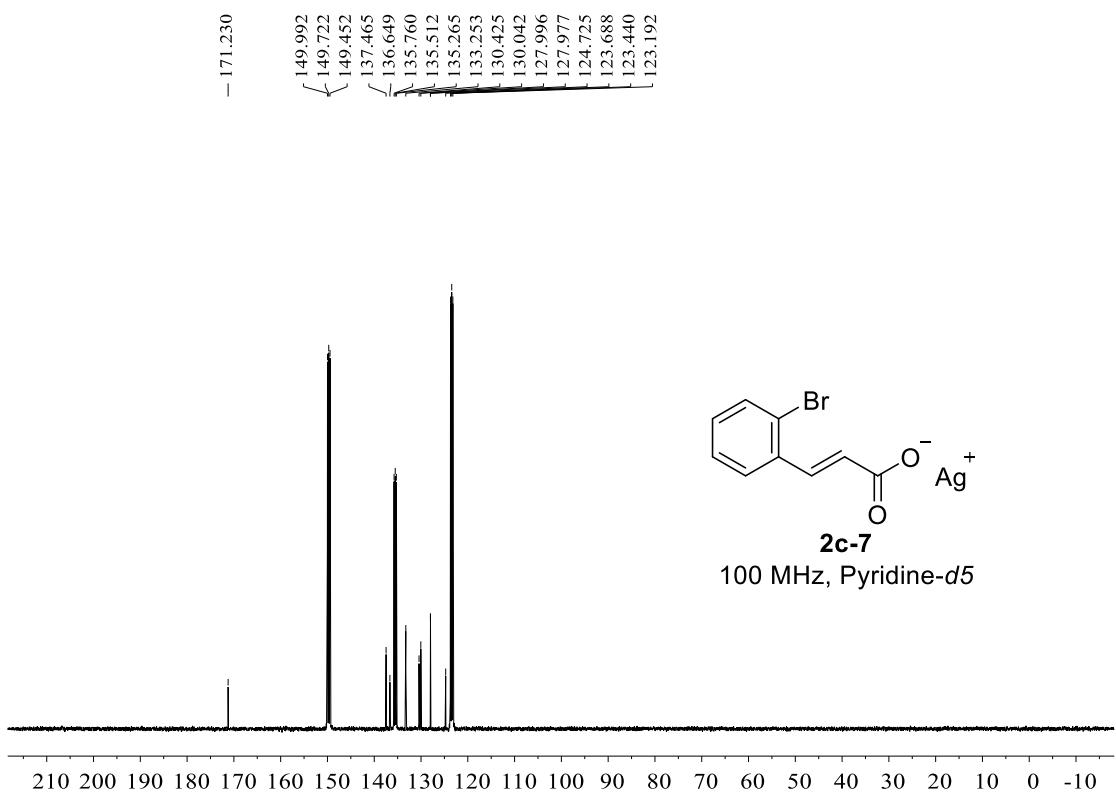
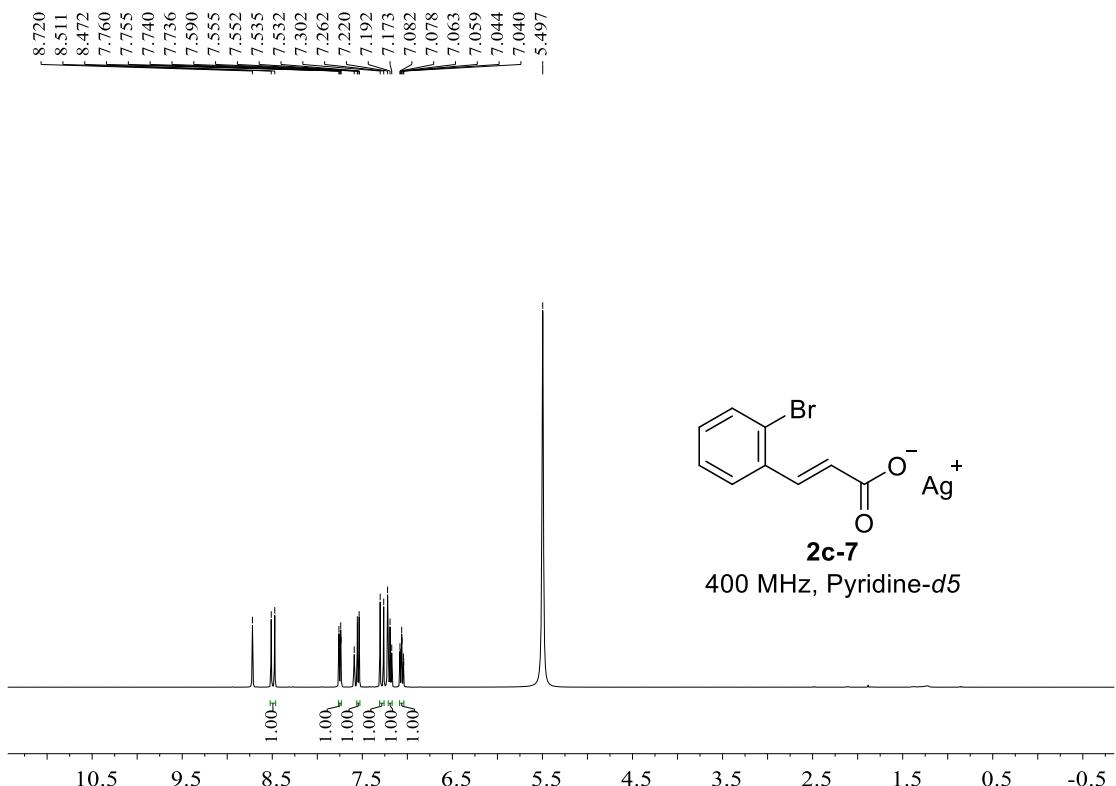


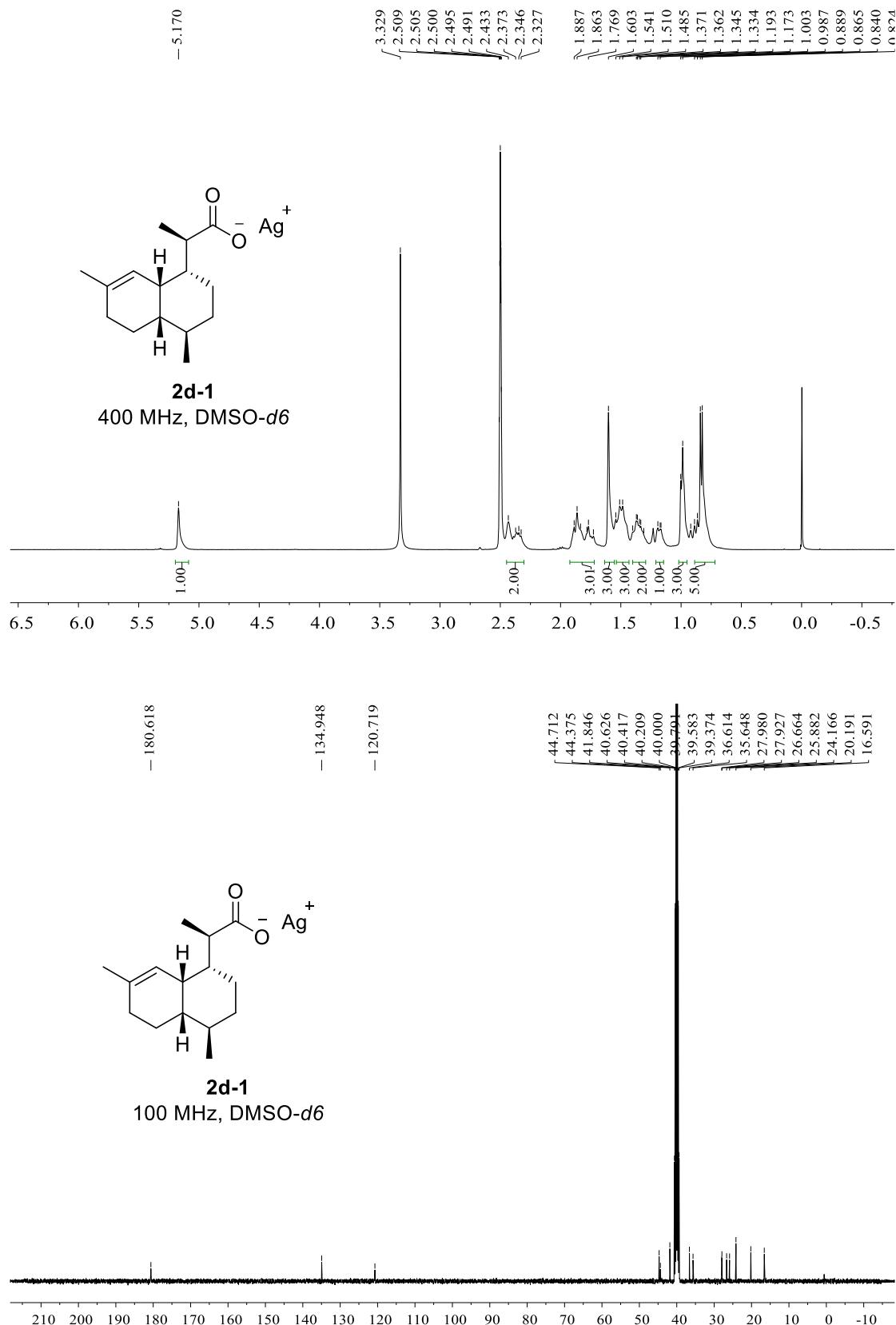


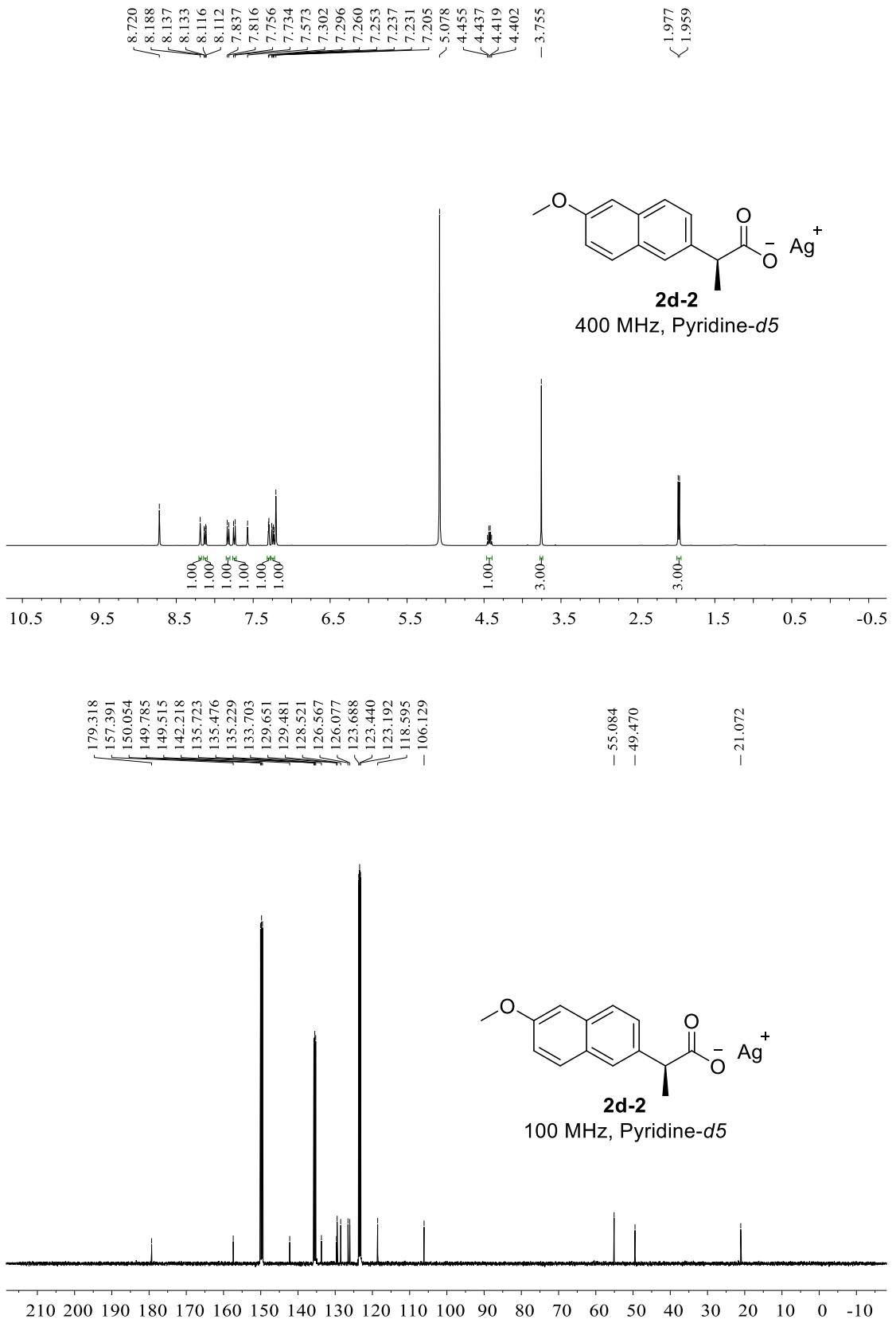


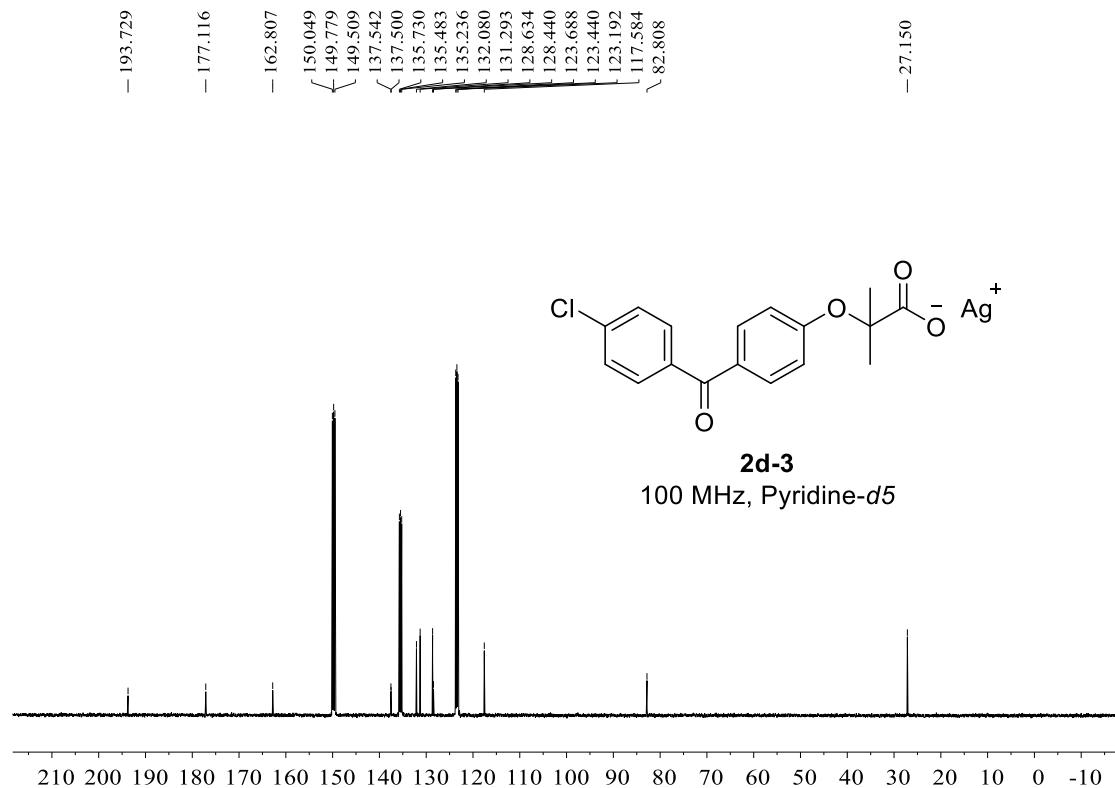
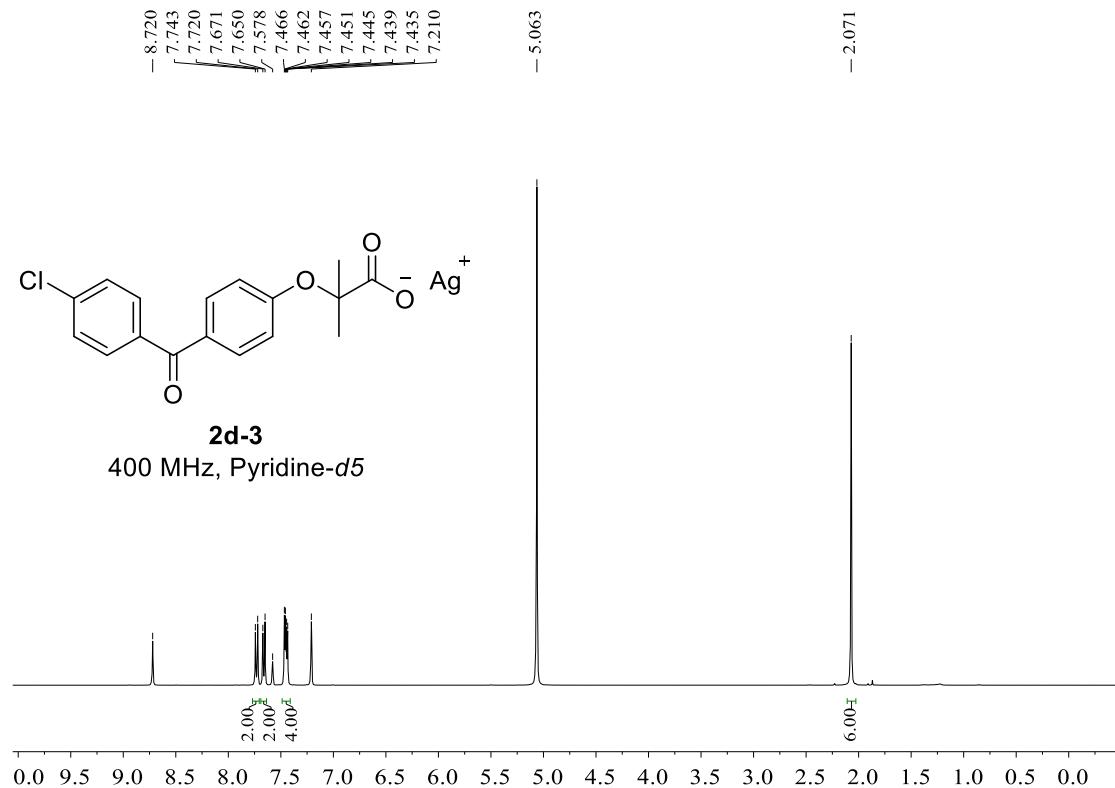
- -61.912

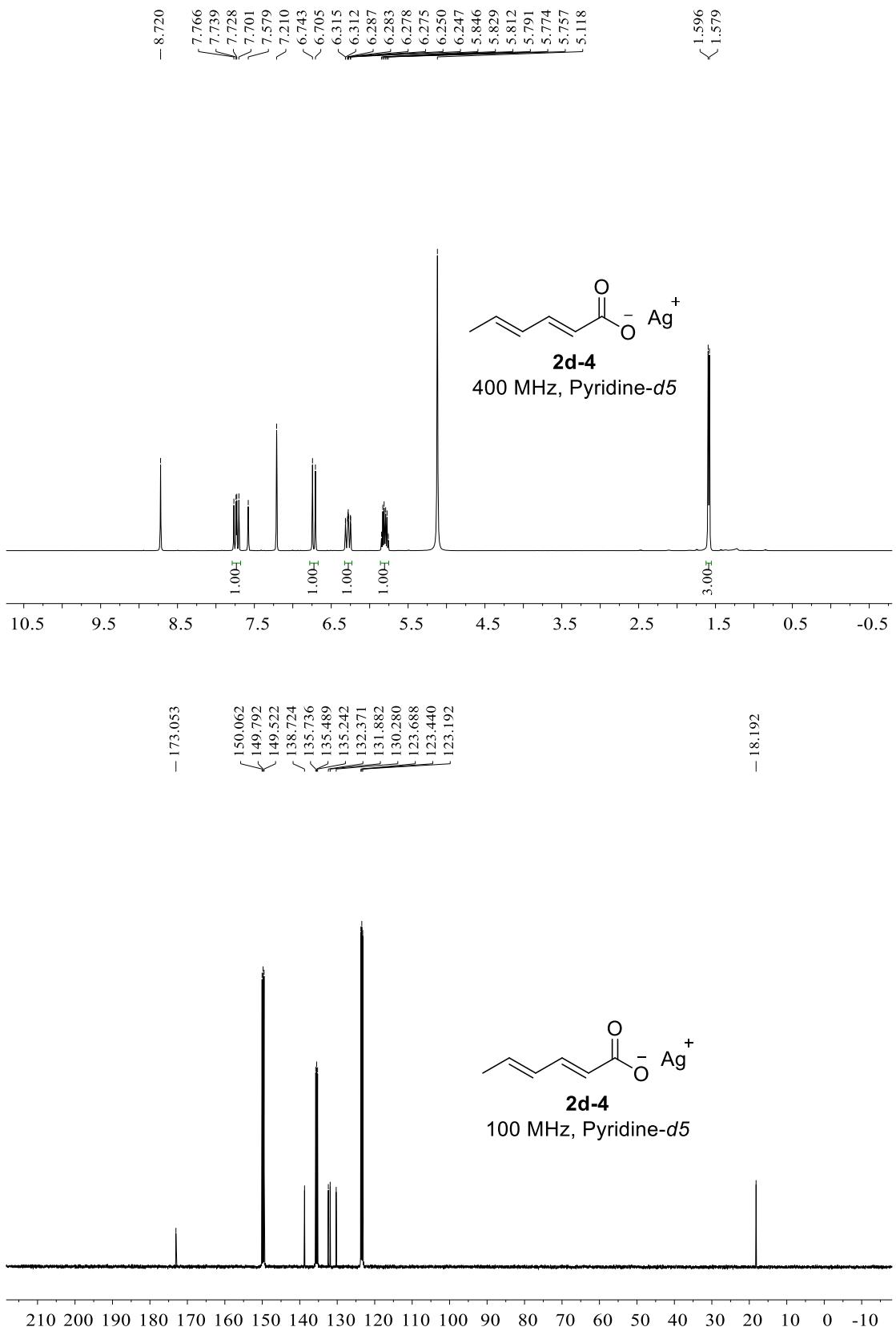


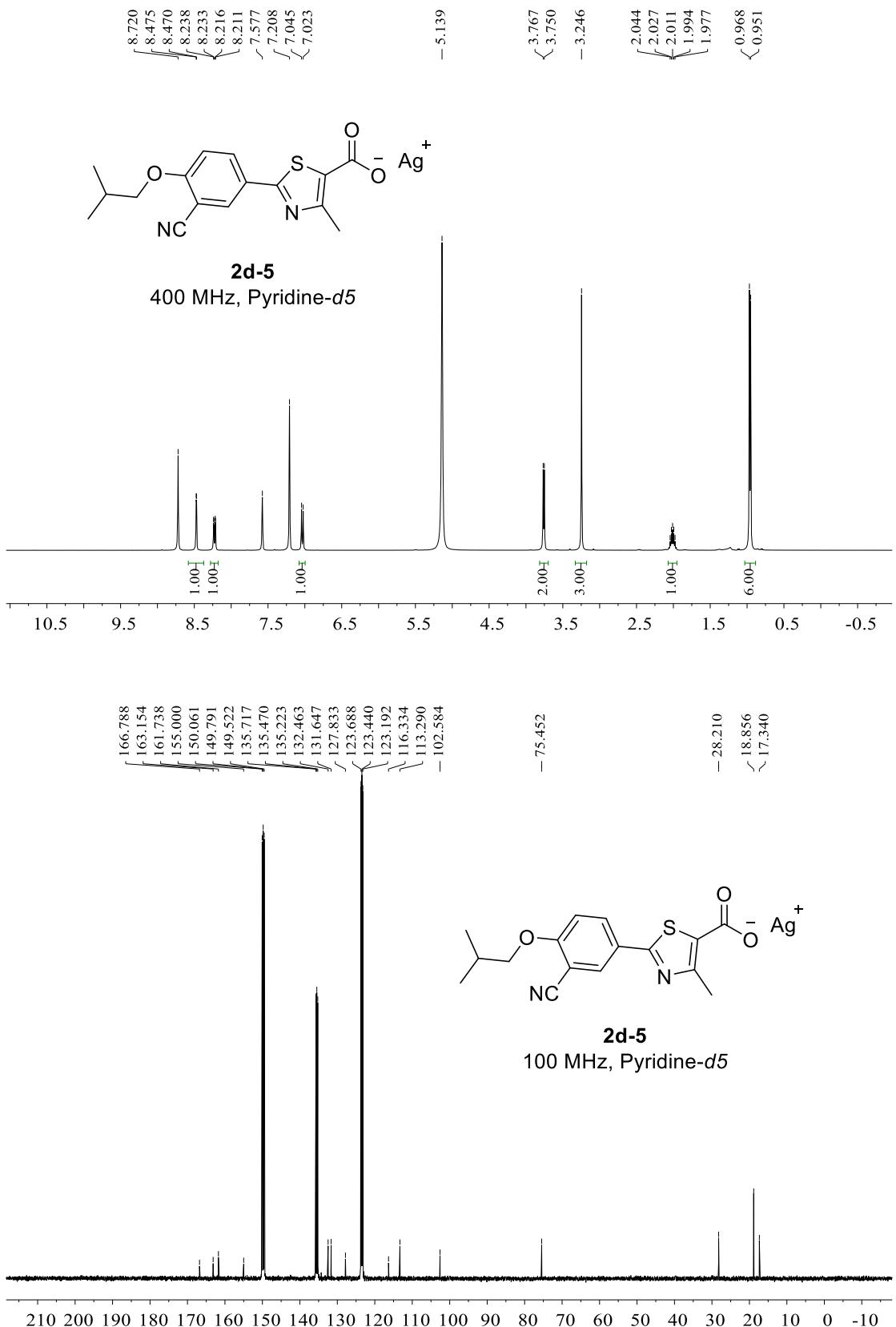


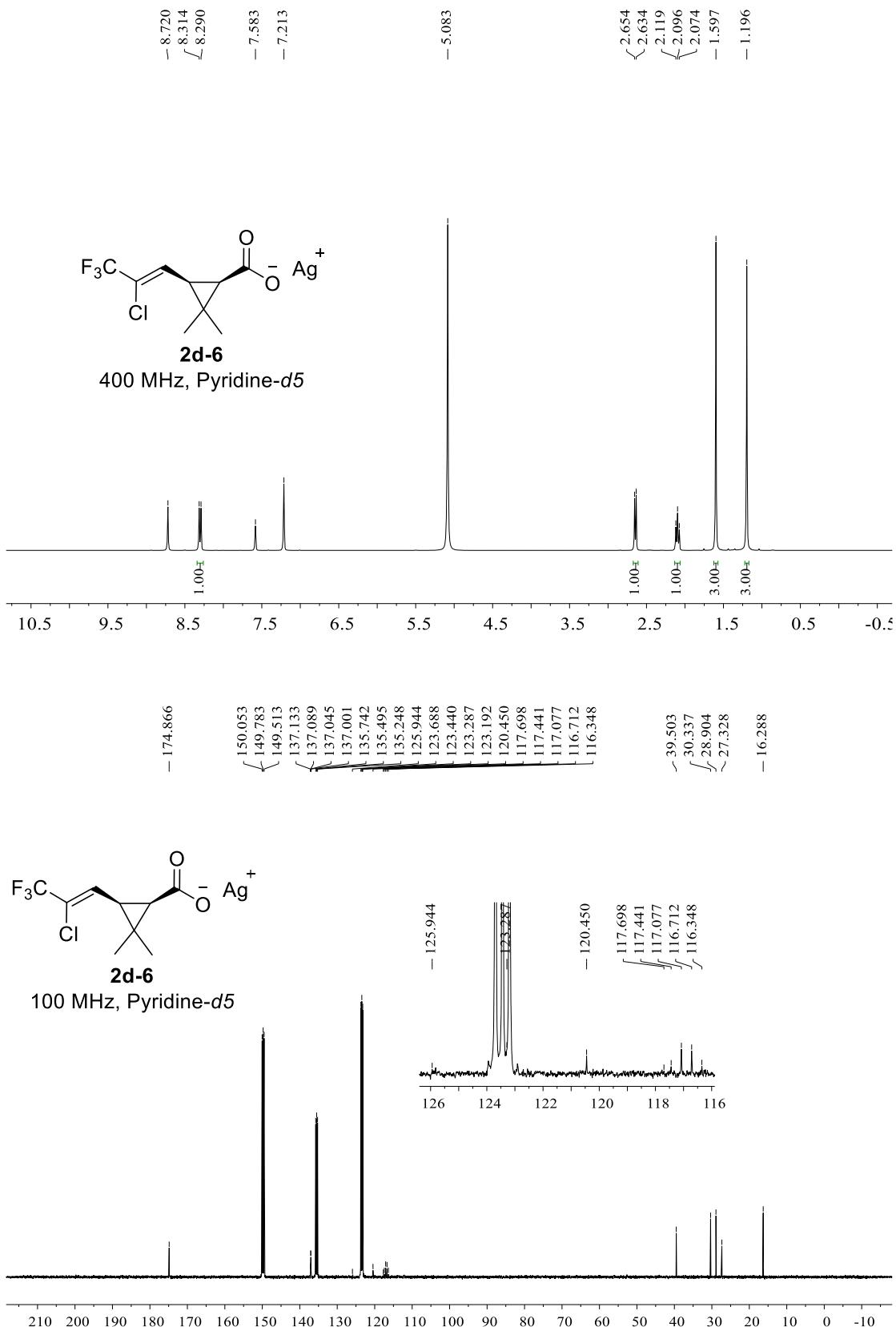




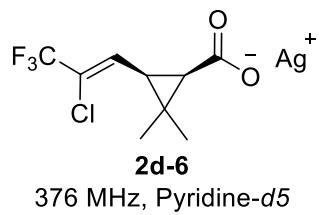








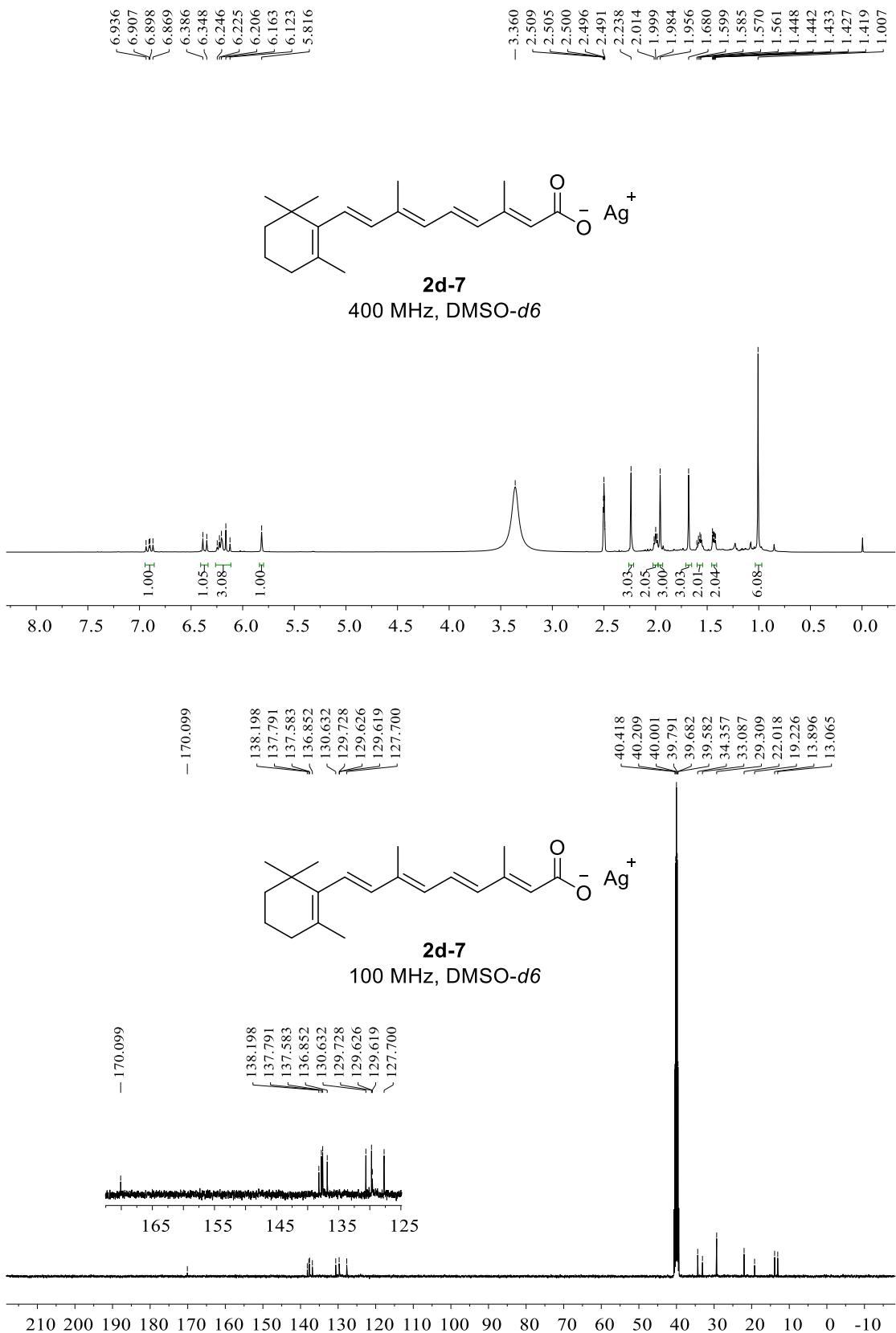
-67.231

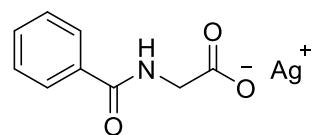
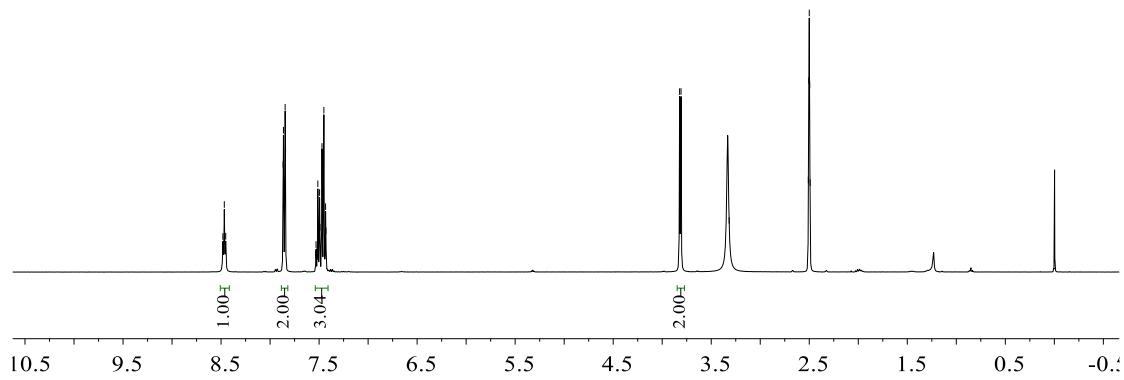
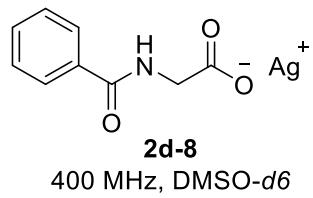
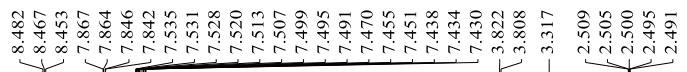


2d-6

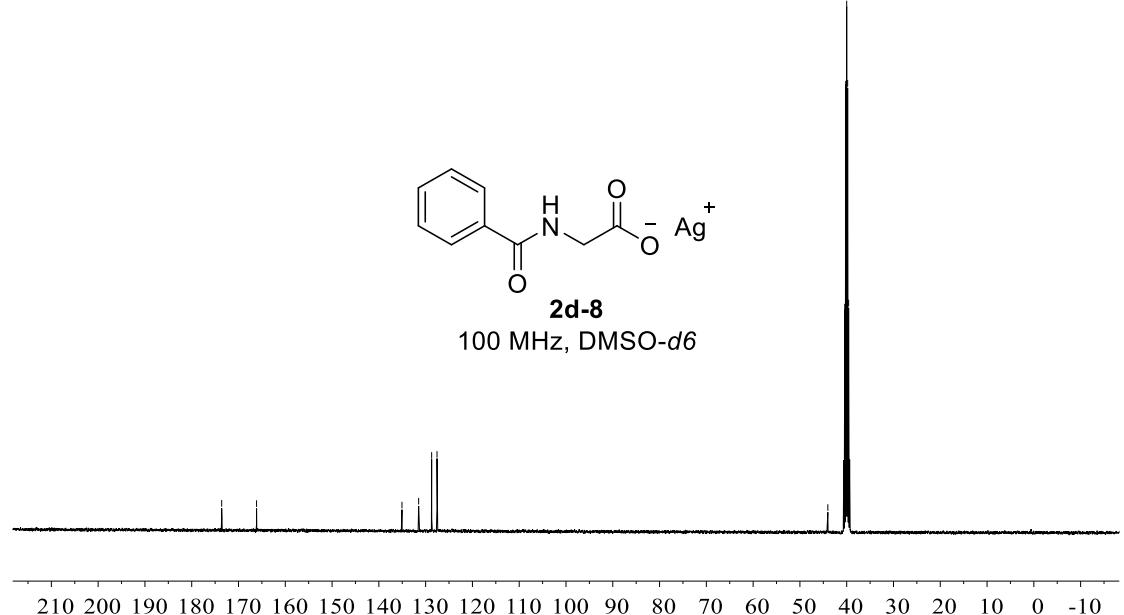
376 MHz, Pyridine-*d*5

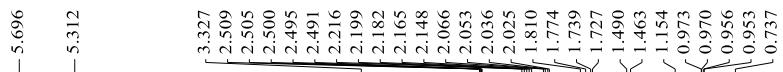
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -2



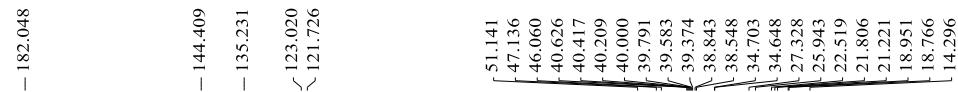
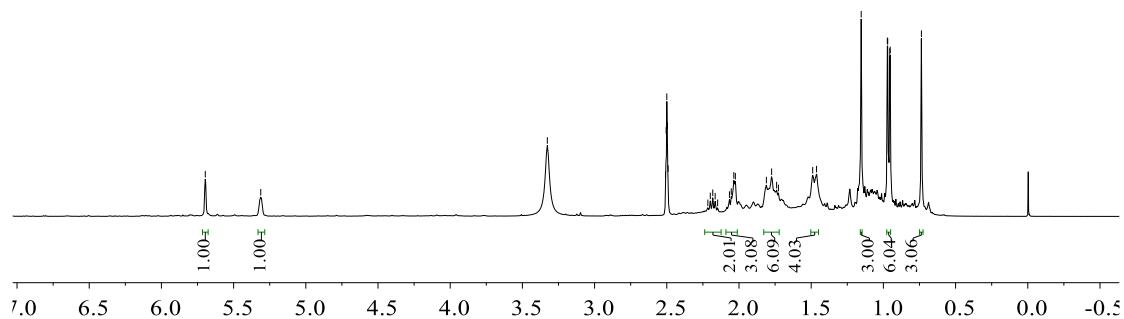


100 MHz, DMSO-*d*6

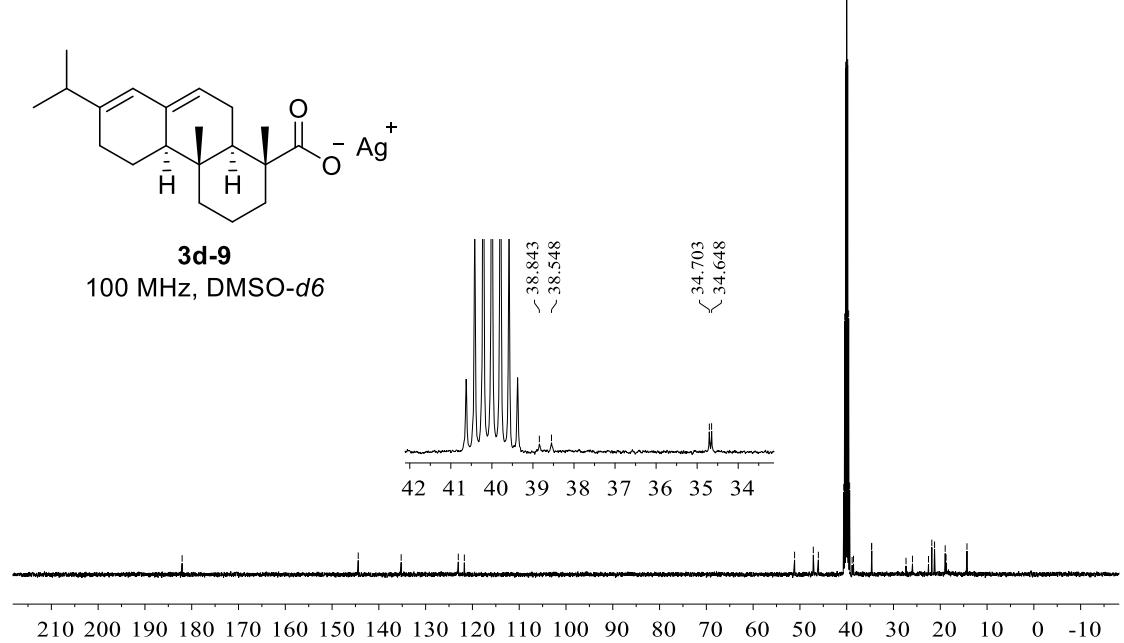




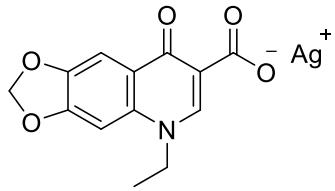
3d-9
400 MHz, DMSO-*d*6



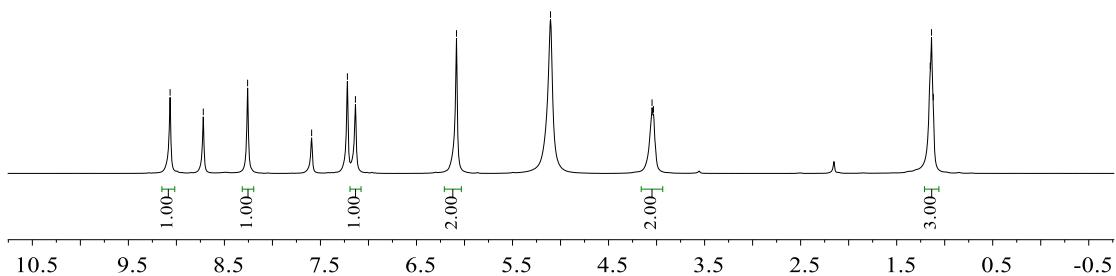
3d-9
100 MHz, DMSO-*d*6



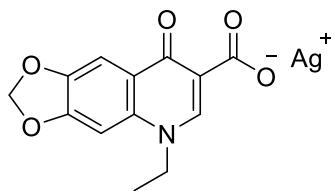
-9.066
 -8.720
 -8.259
 -7.592
 / 7.219
 / 7.136



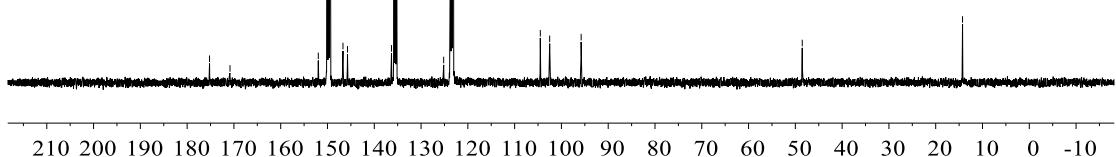
3d-10
400 MHz, Pyridine-*d*5

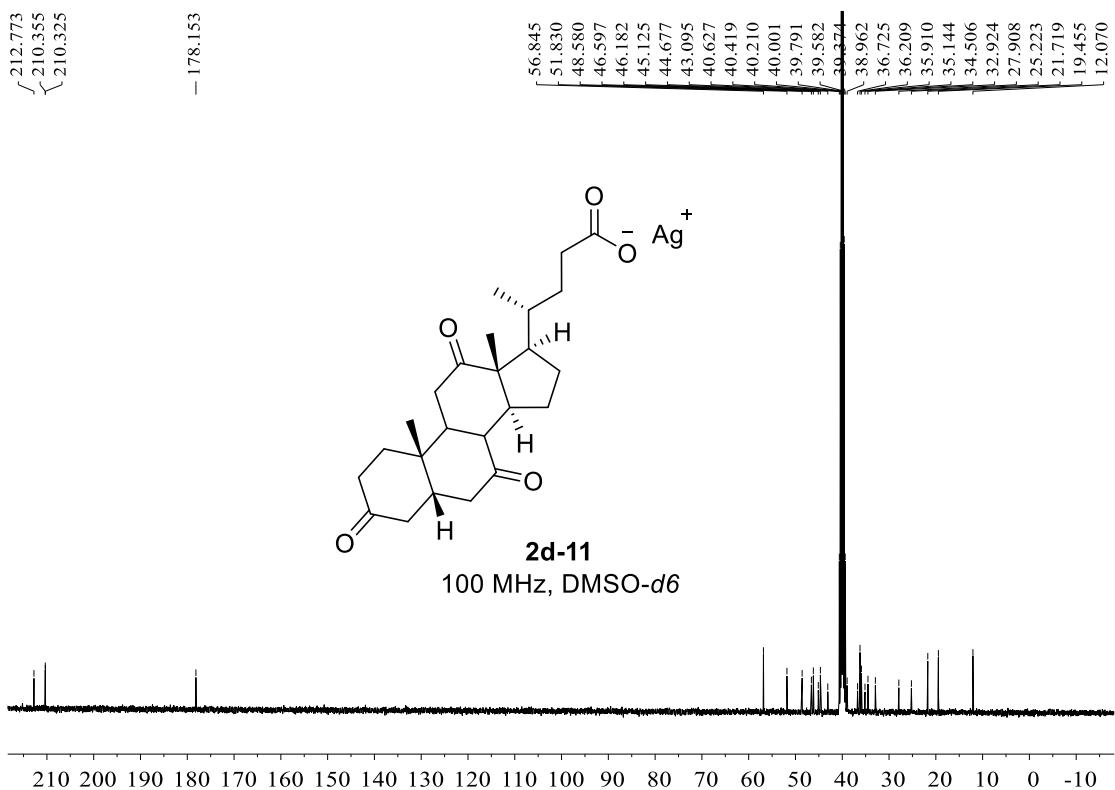
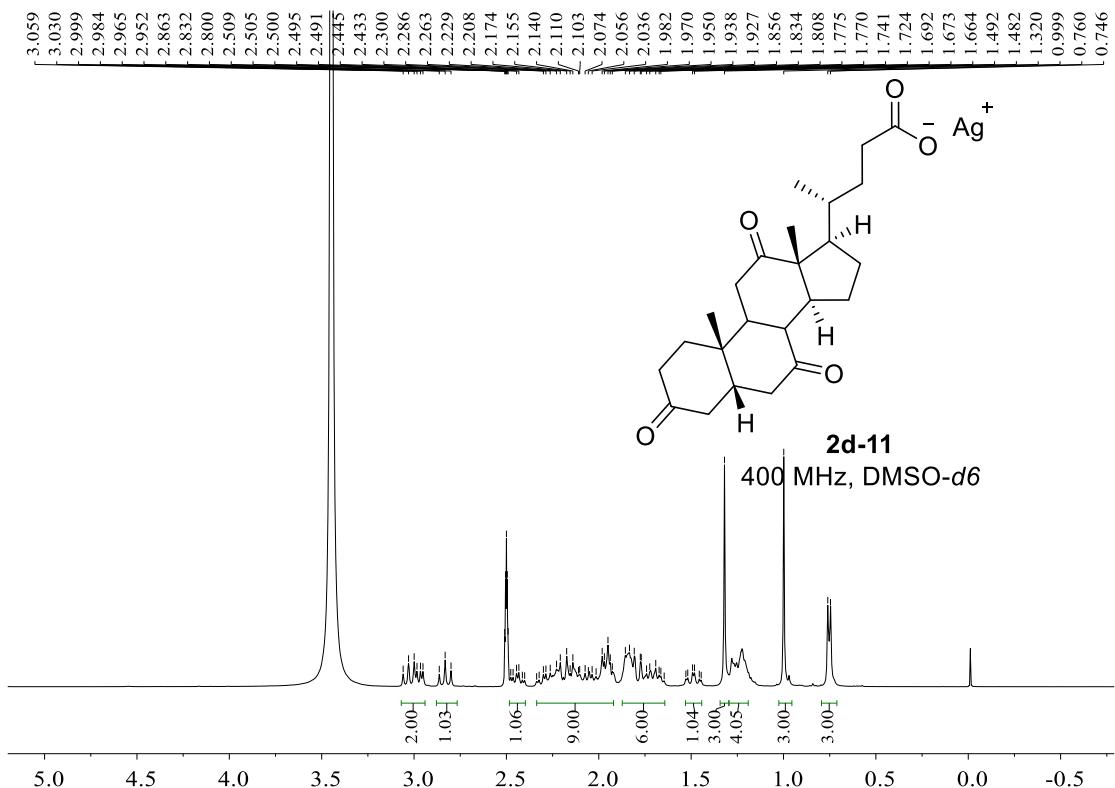


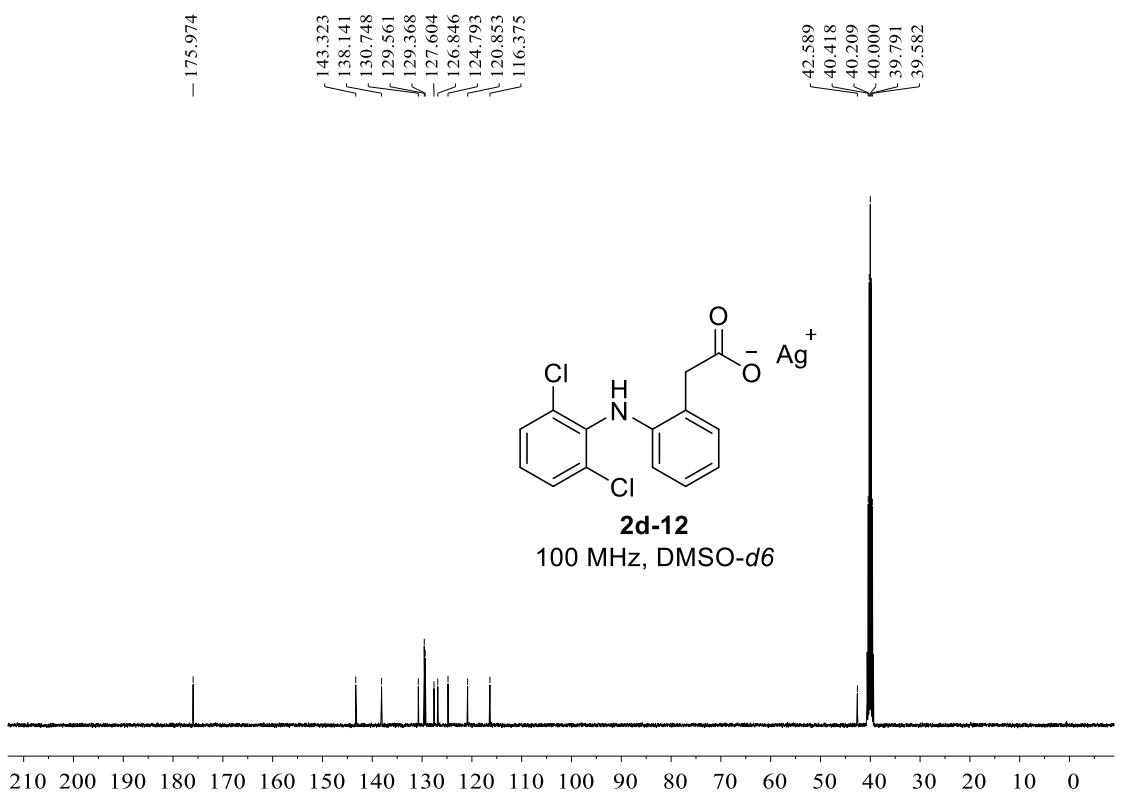
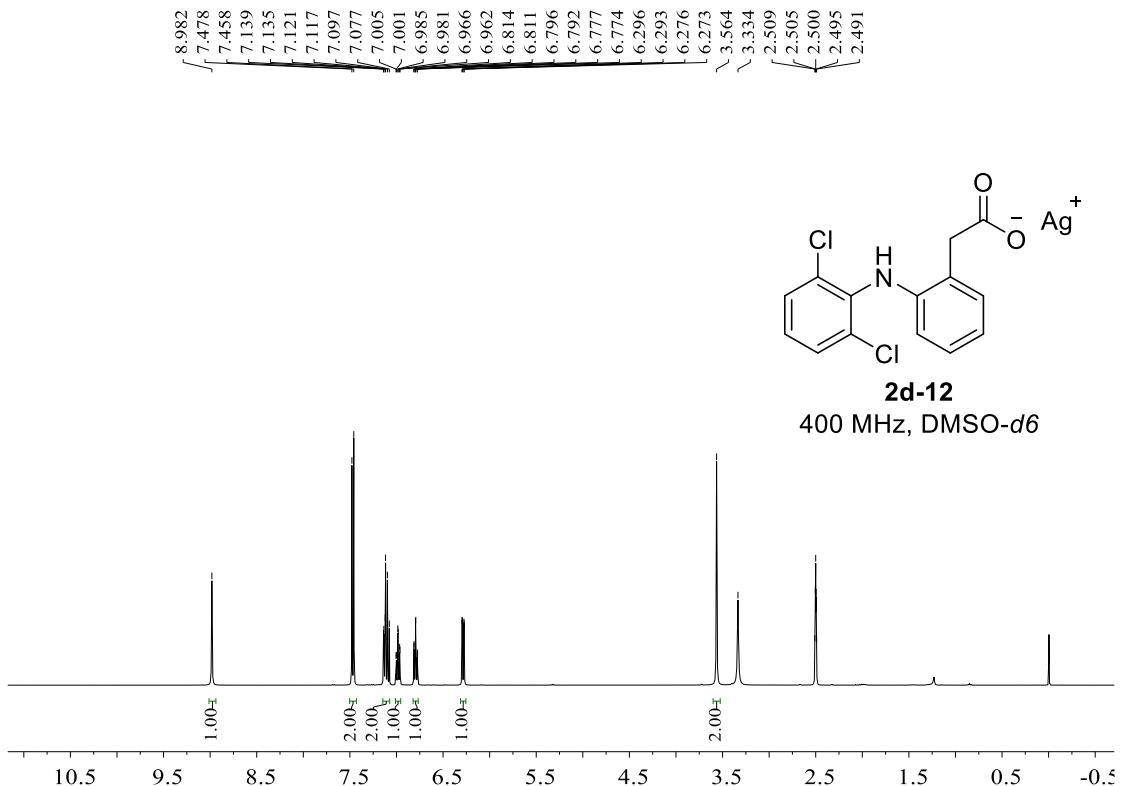
\ 175.209
 \ 170.840
 \ 151.965
 \ 150.028
 \ 149.759
 \ 149.489
 \ 146.692
 \ 145.727
 \ 136.320
 \ 135.751
 \ 135.504
 \ 135.257
 \ 125.169
 \ 123.688
 \ 123.440
 \ 123.192
 \ 104.513
 \ 102.513
 \ 95.786
 - 48.524
 - 14.265



3d-10
100 MHz, Pyridine-*d*5



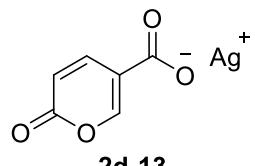




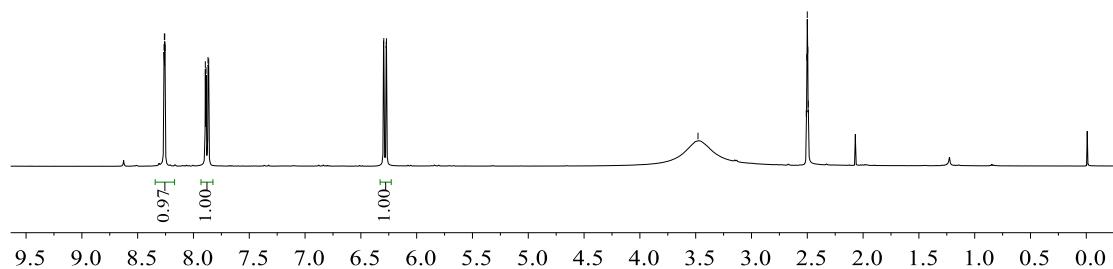
8.263
 8.261
 8.257
 8.255
 7.894
 7.887
 7.870
 7.863

6.297
 6.294
 6.273
 6.270

-3.479
 2.509
 2.505
 2.500
 2.496
 2.491

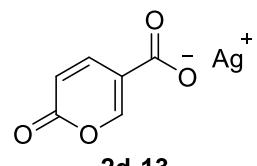


2d-13
400 MHz, DMSO-*d*6

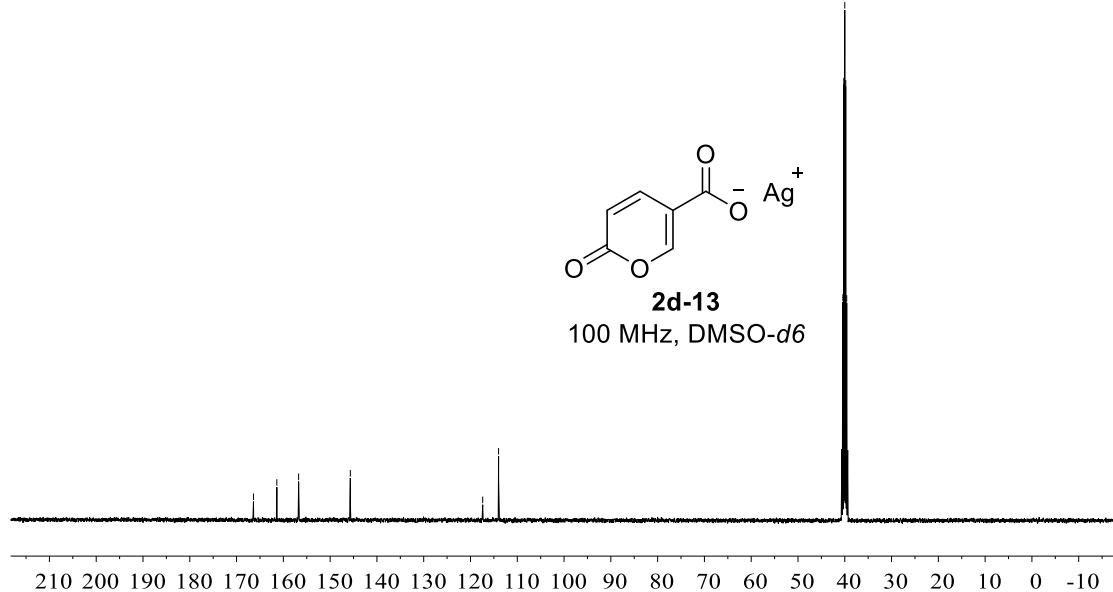


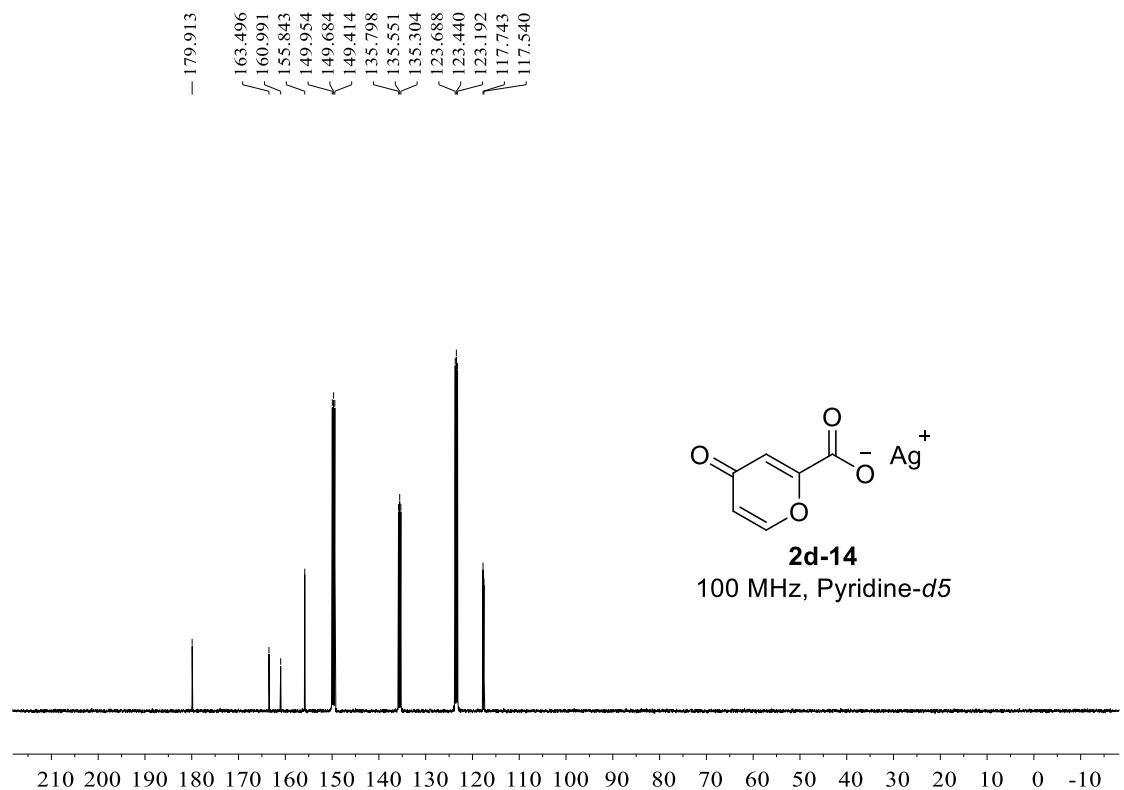
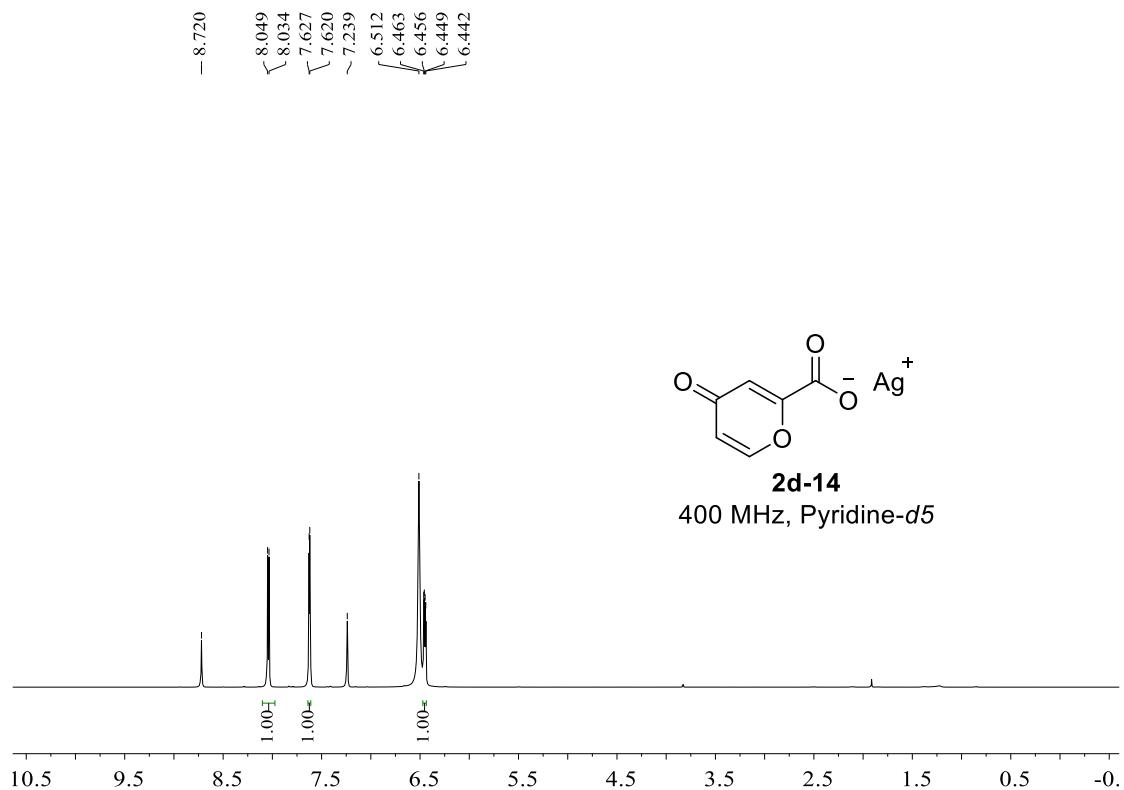
- 166.388
 - 161.391
 - 156.734
 - 145.684
 ~ 117.391
 ~ 113.997

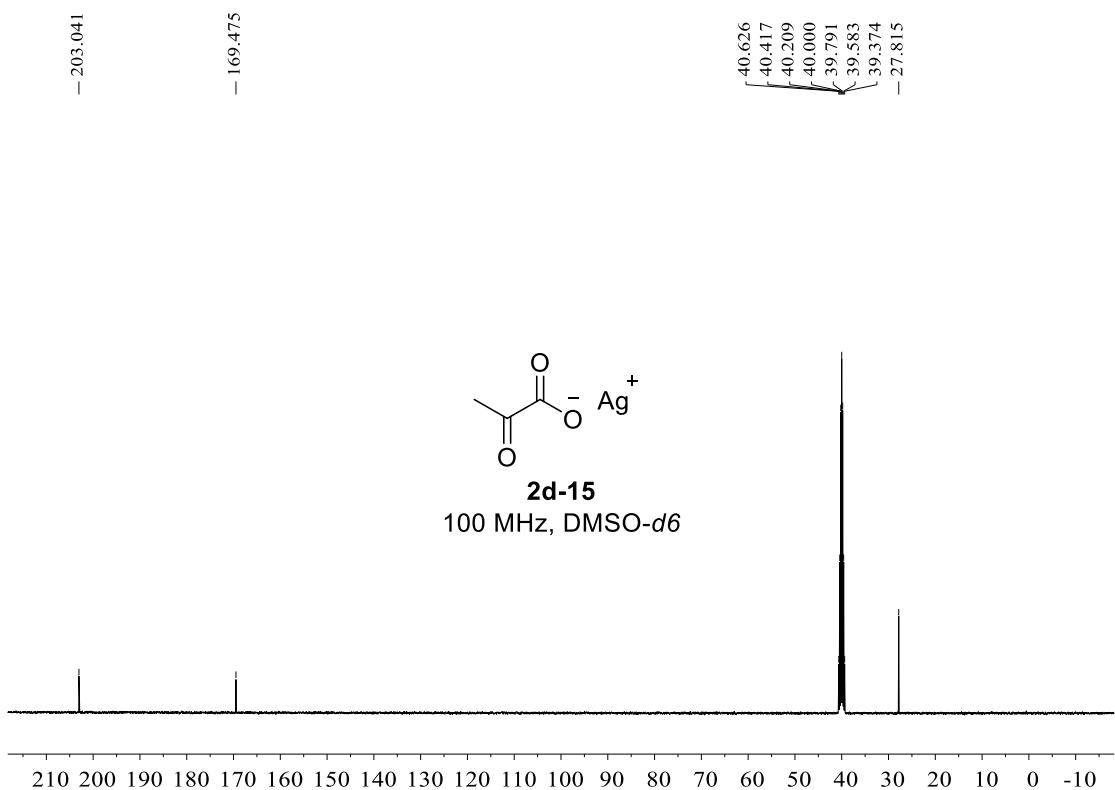
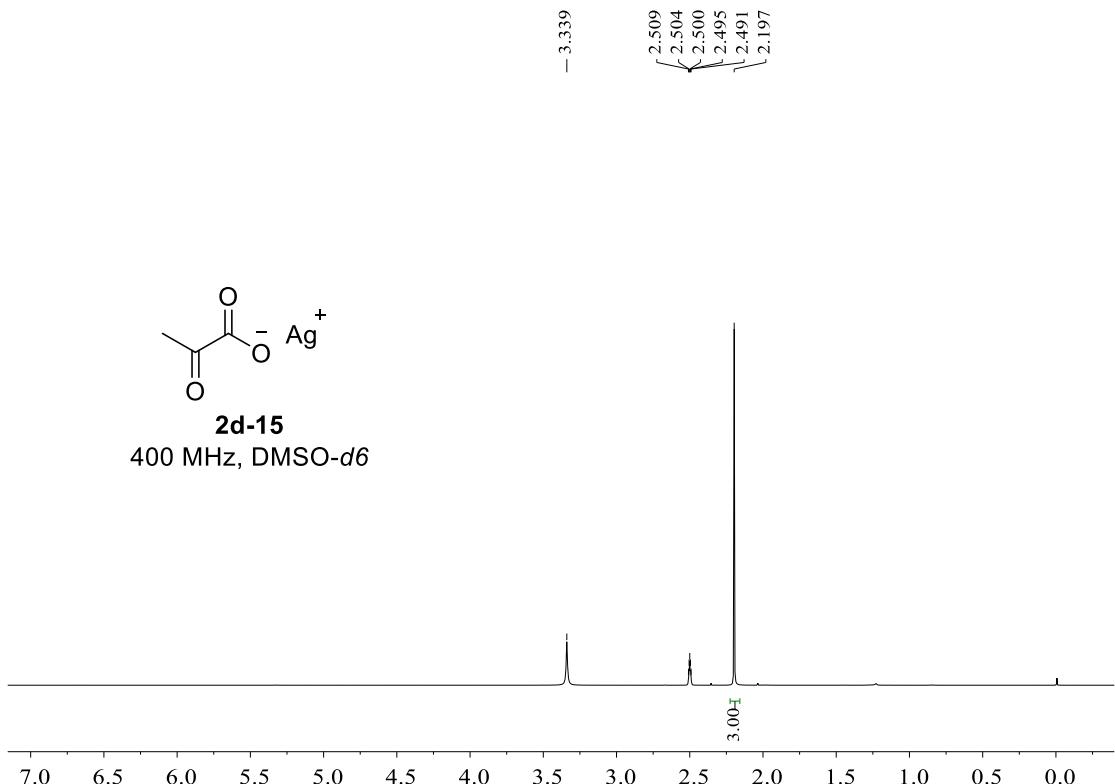
40.417
 40.209
 40.000
 39.791
 39.583

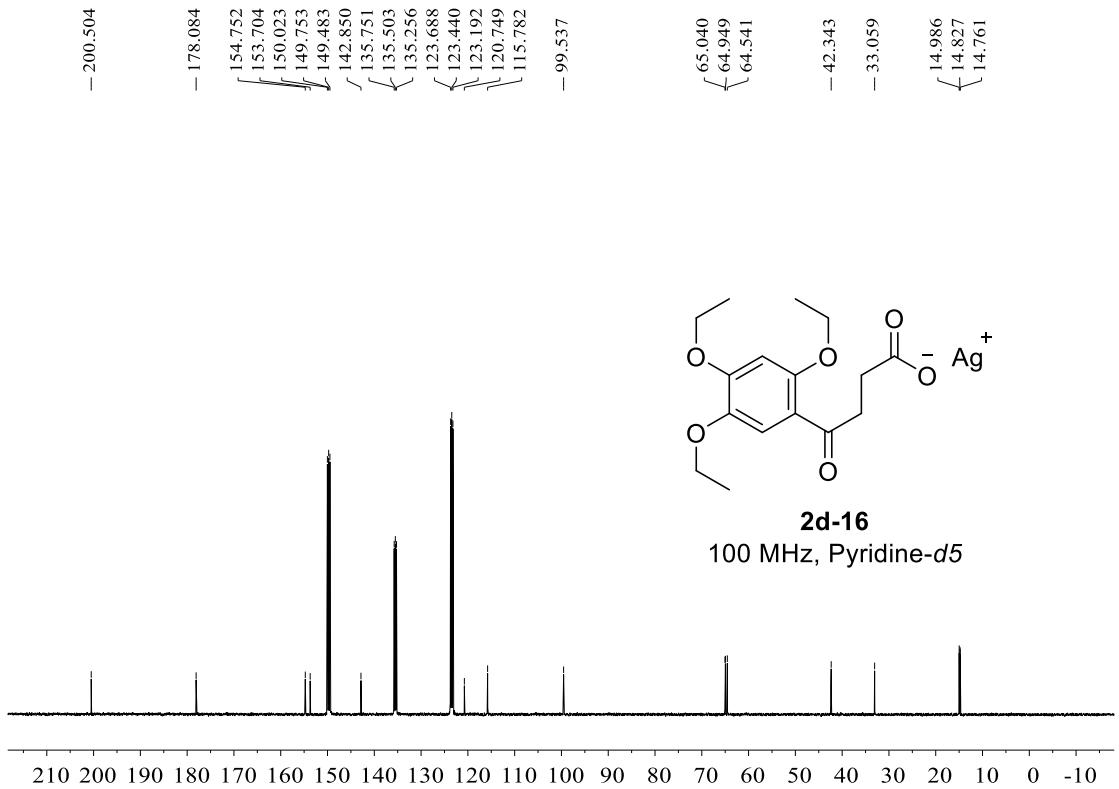
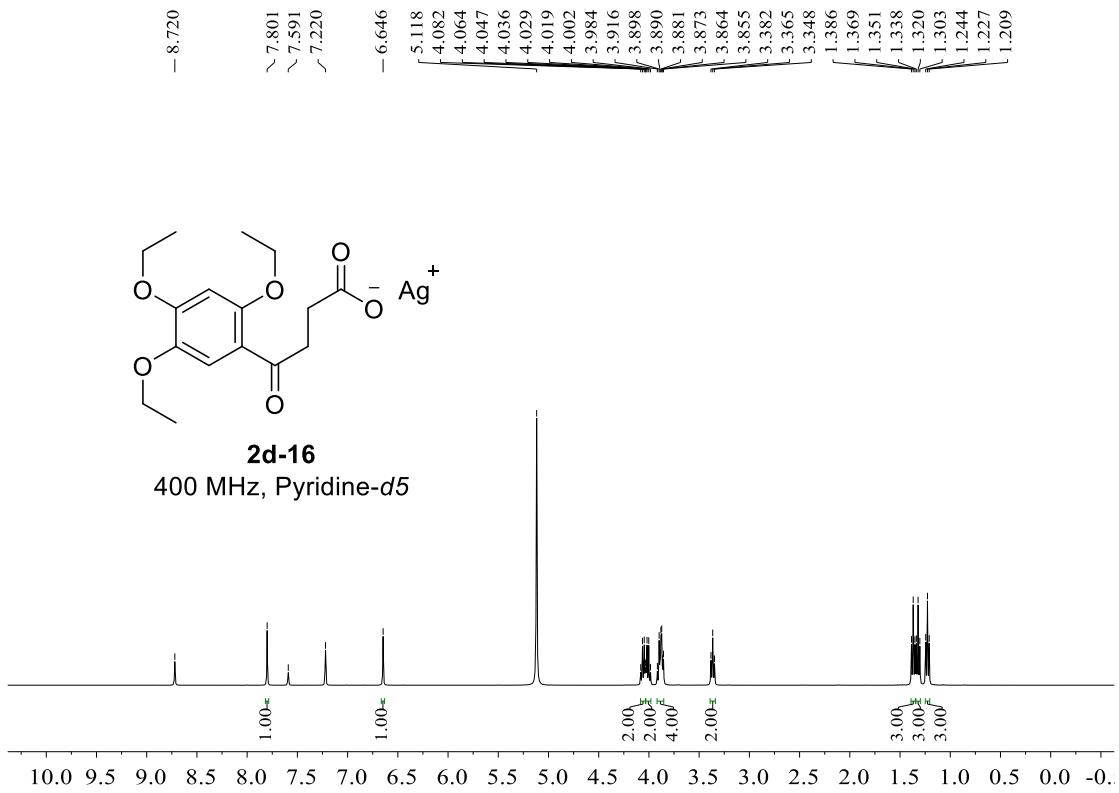


2d-13
100 MHz, DMSO-*d*6

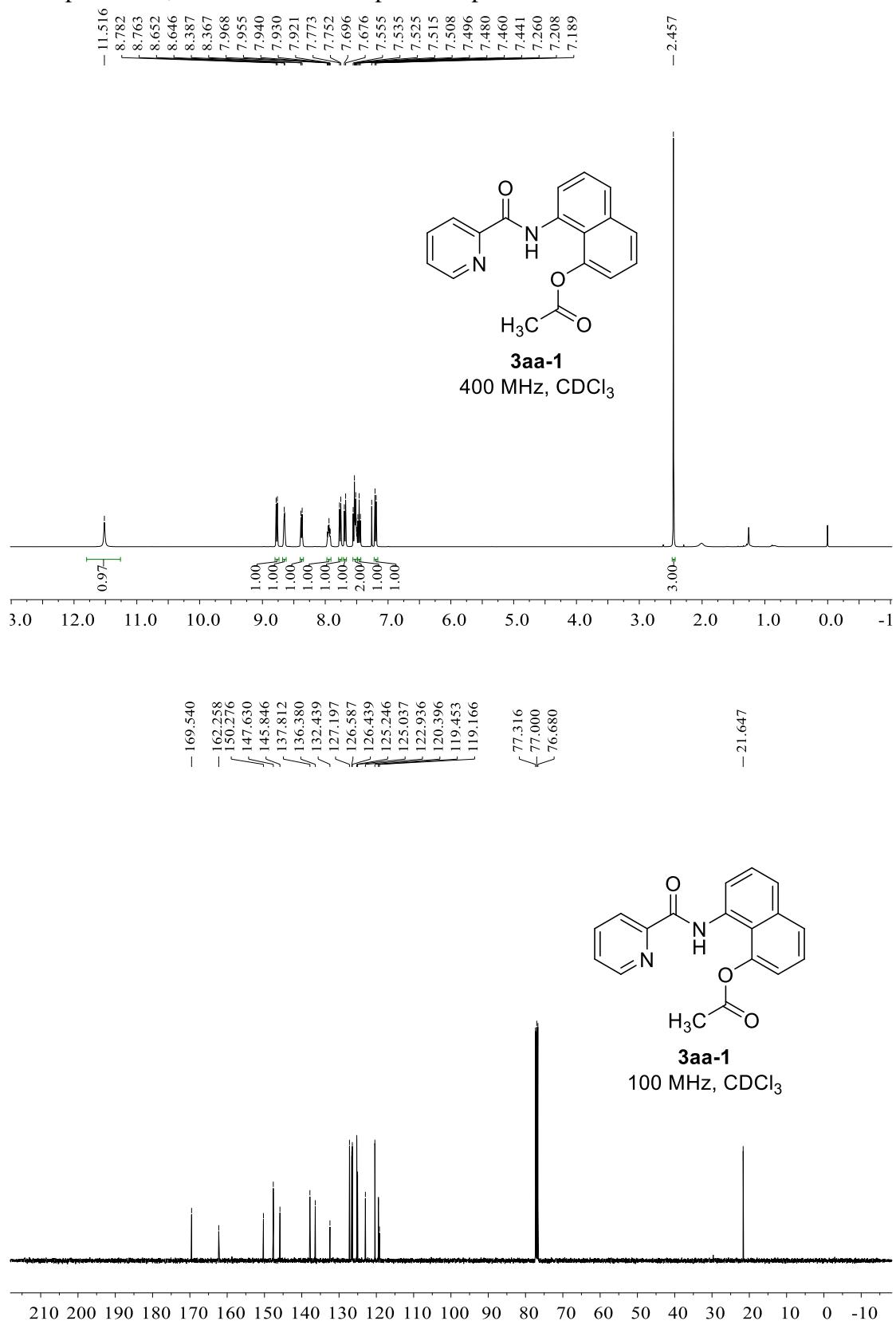


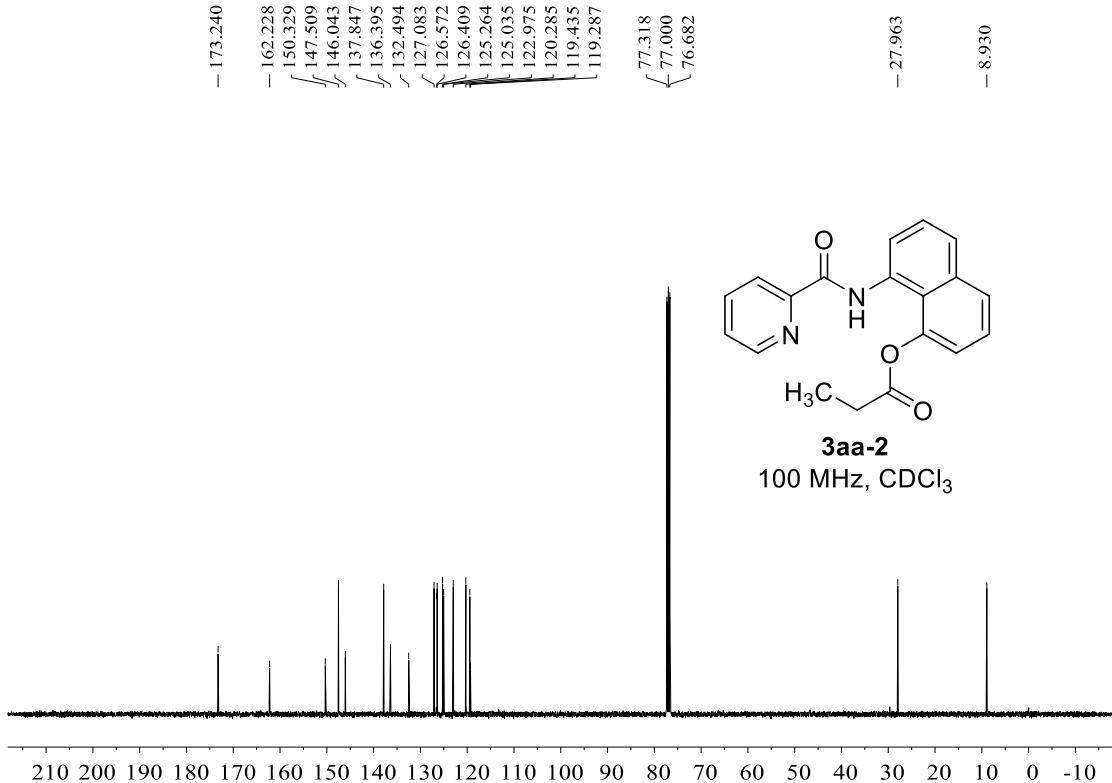
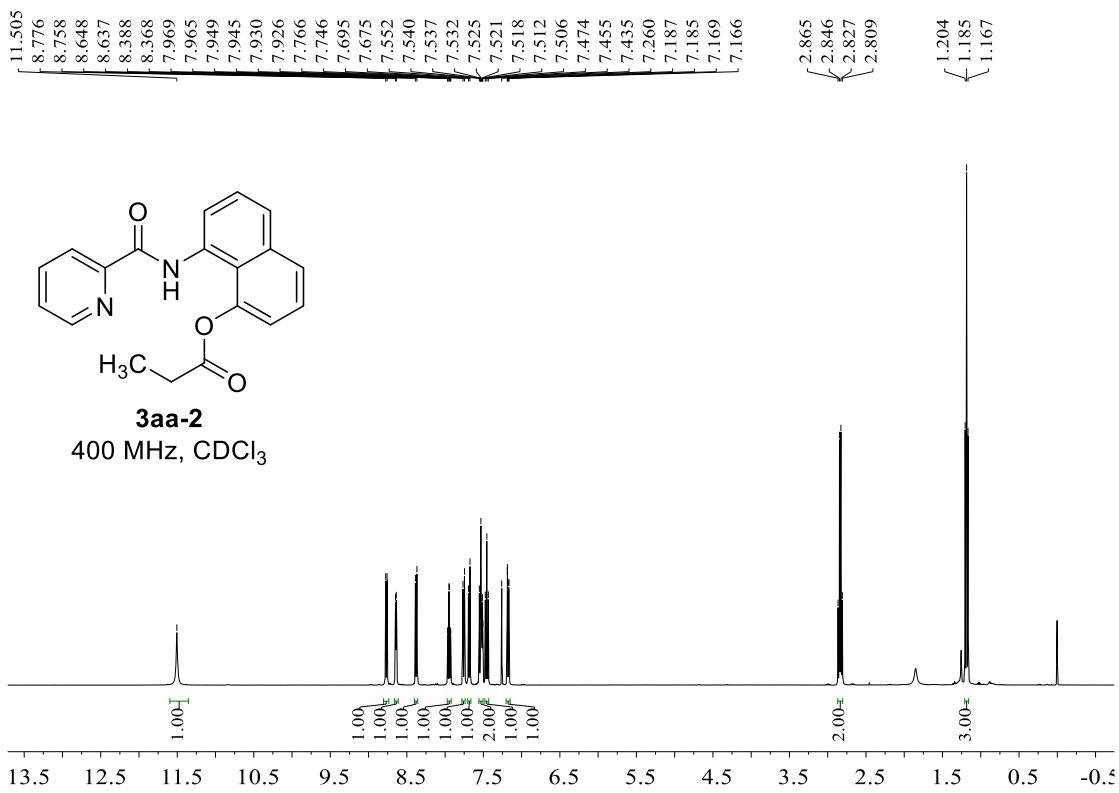


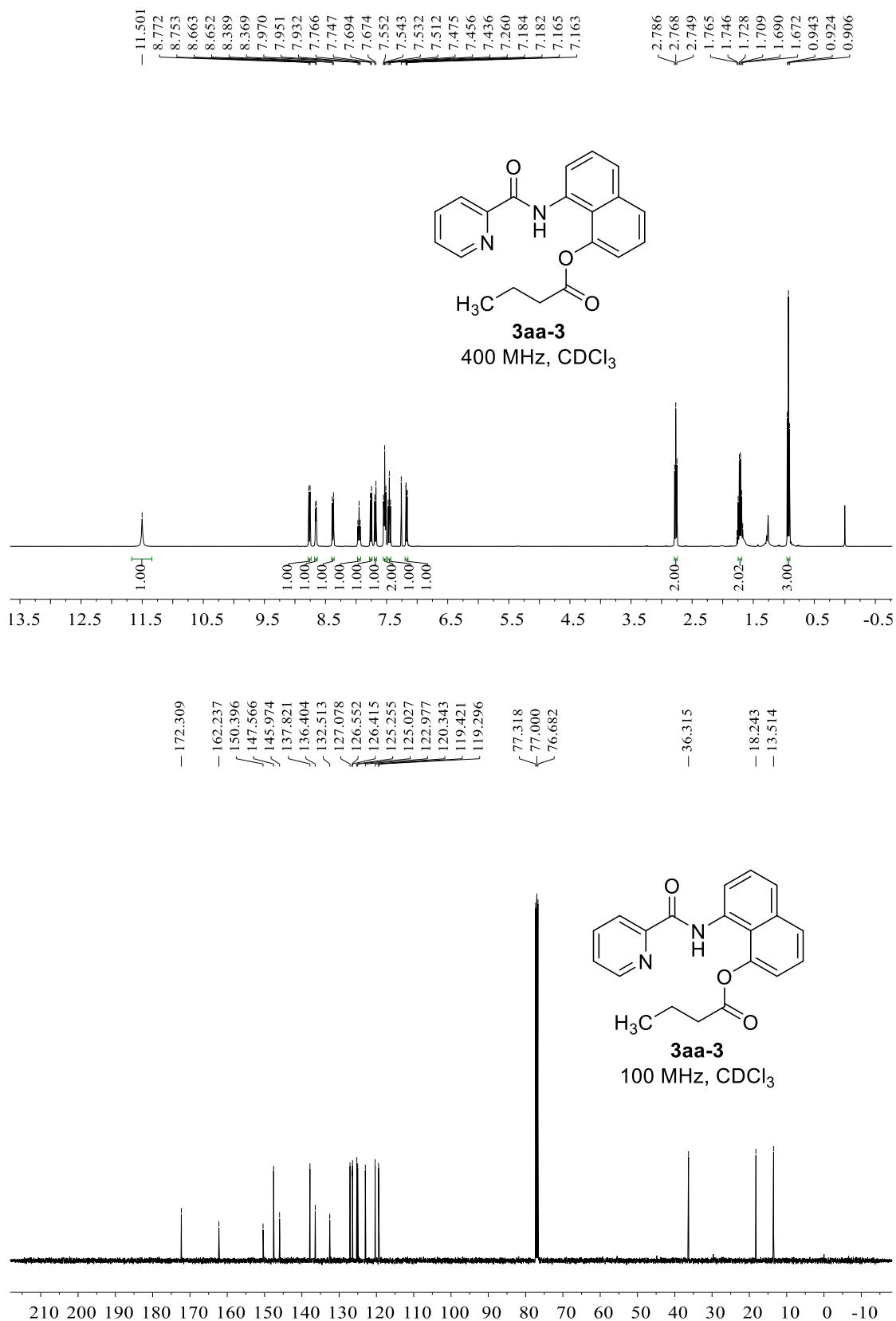


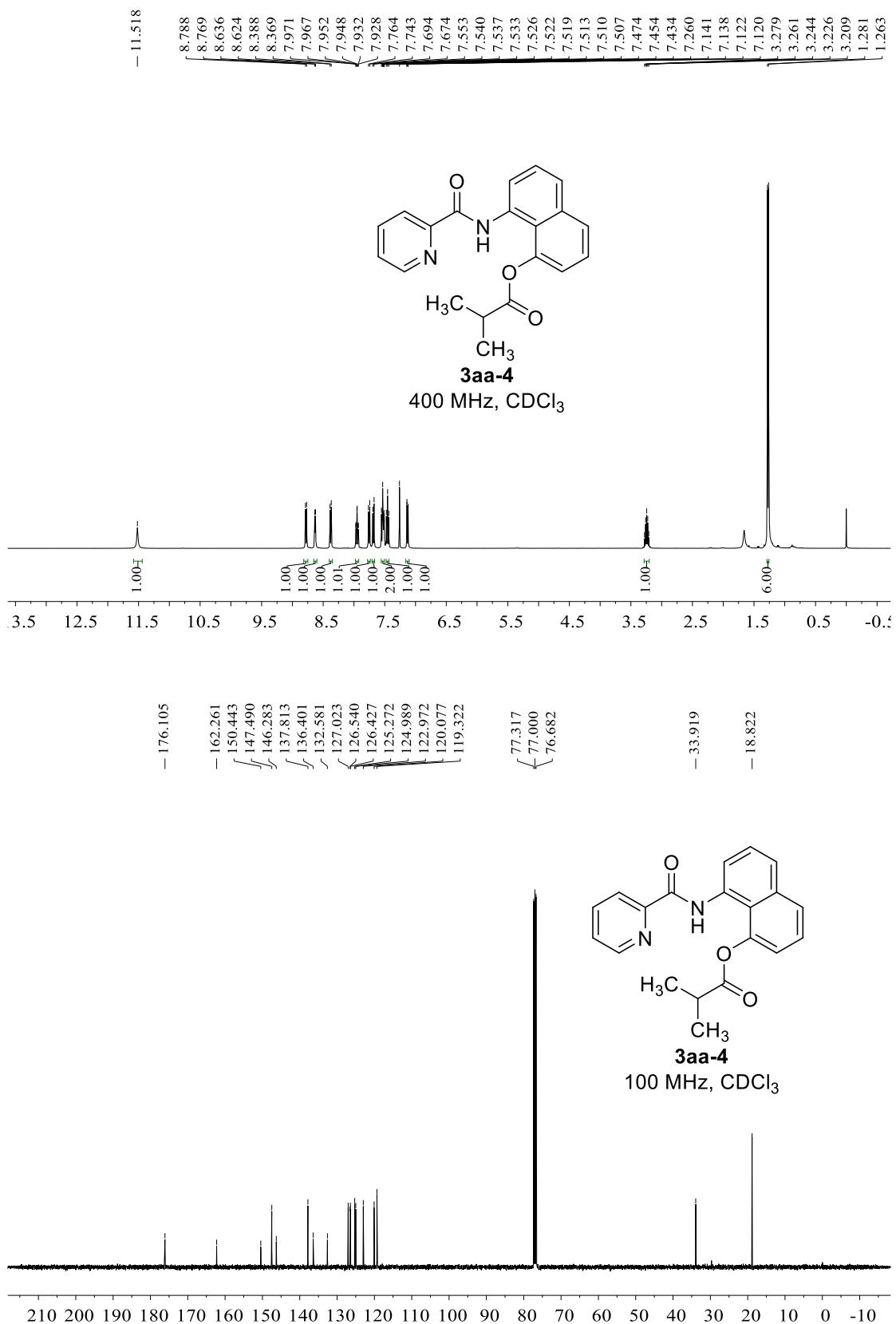


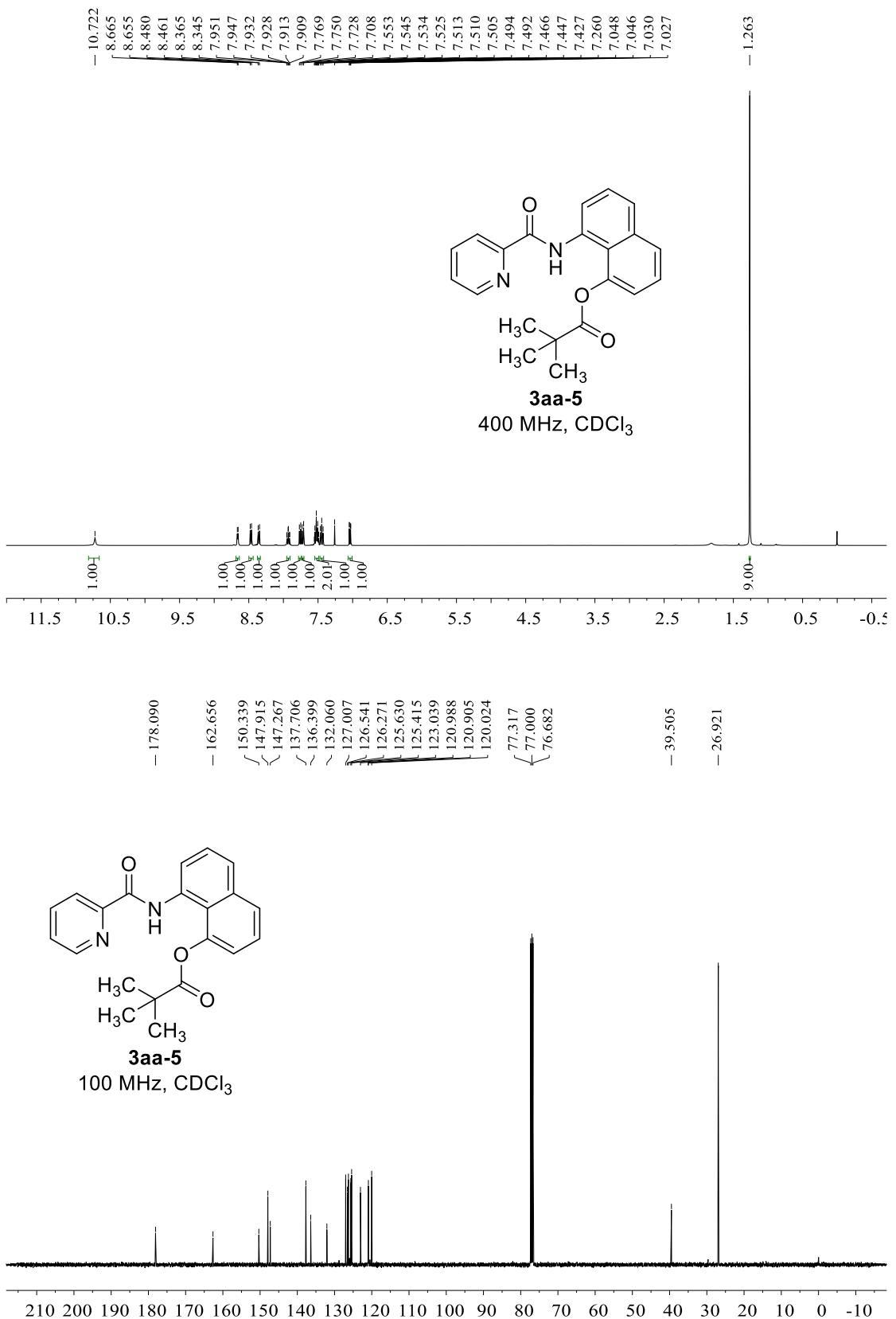
9. Copies of ^1H , ^{13}C and ^{19}F NMR spectra of products **3aa-1–3wa**

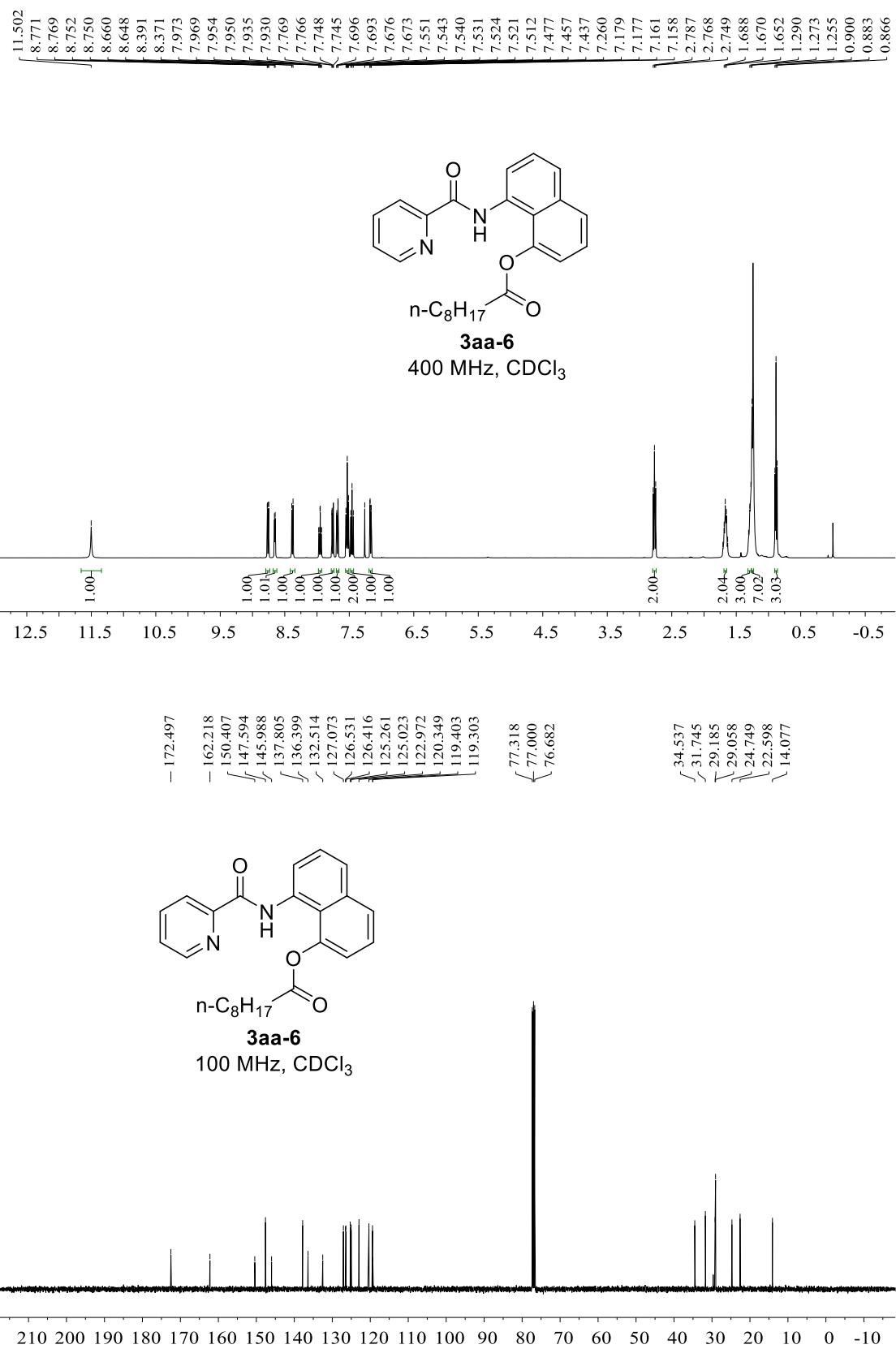


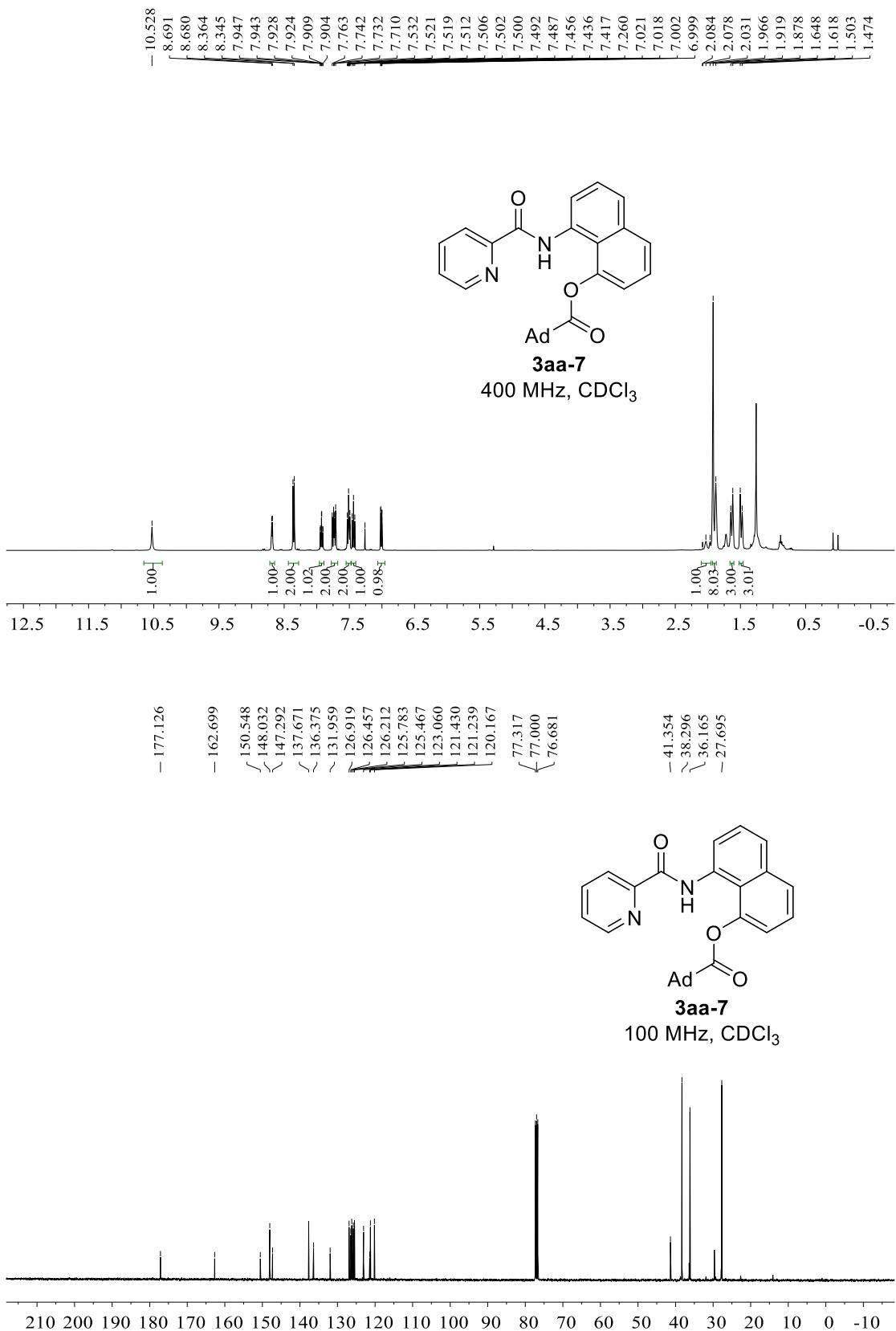


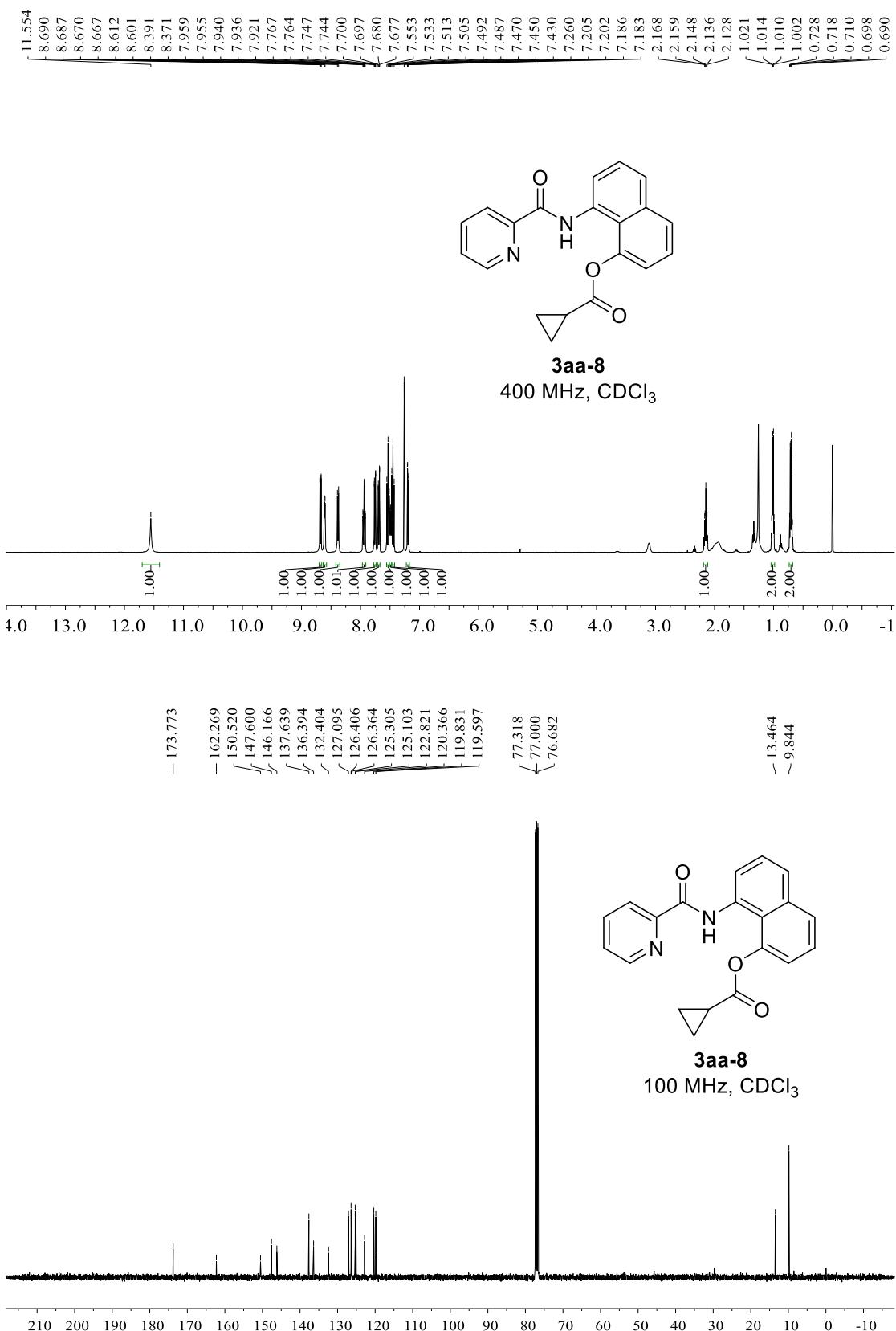


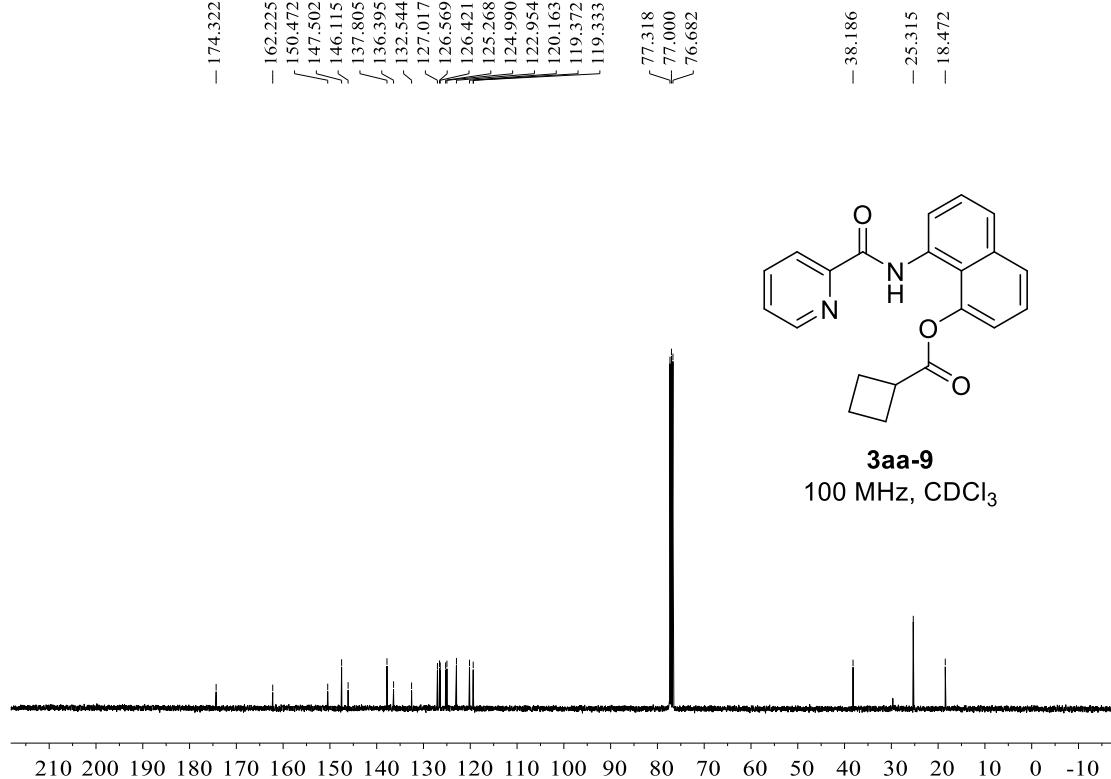
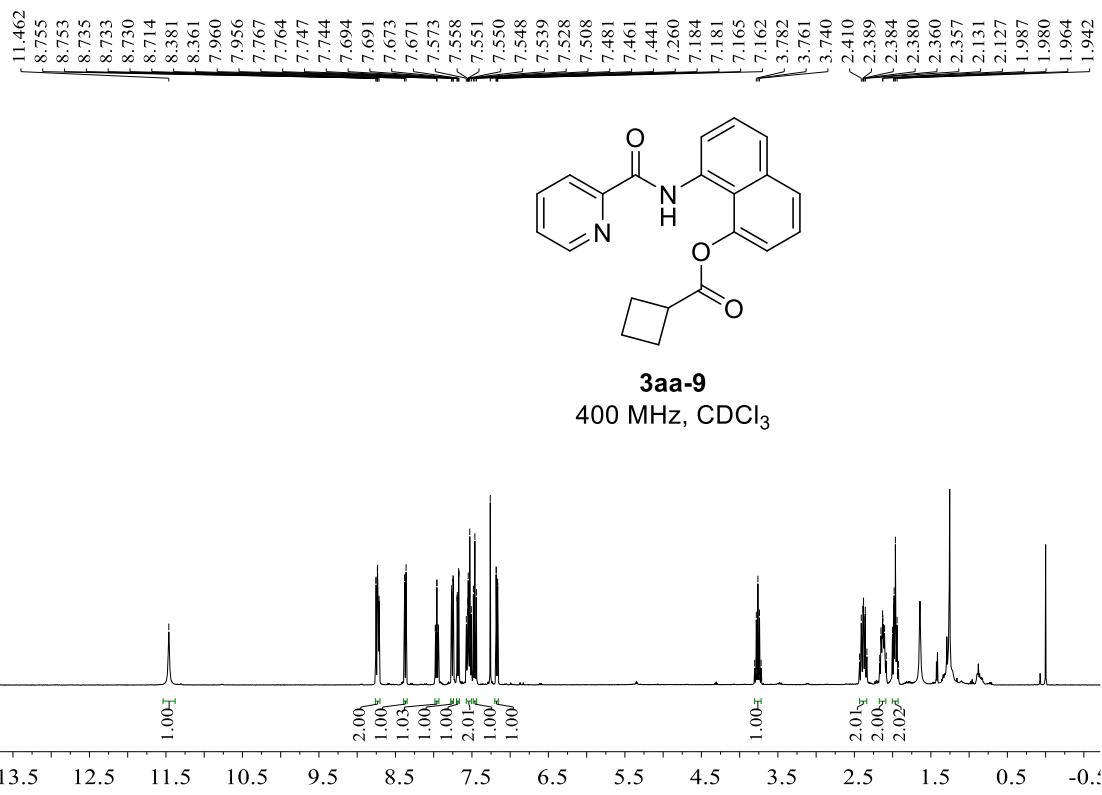


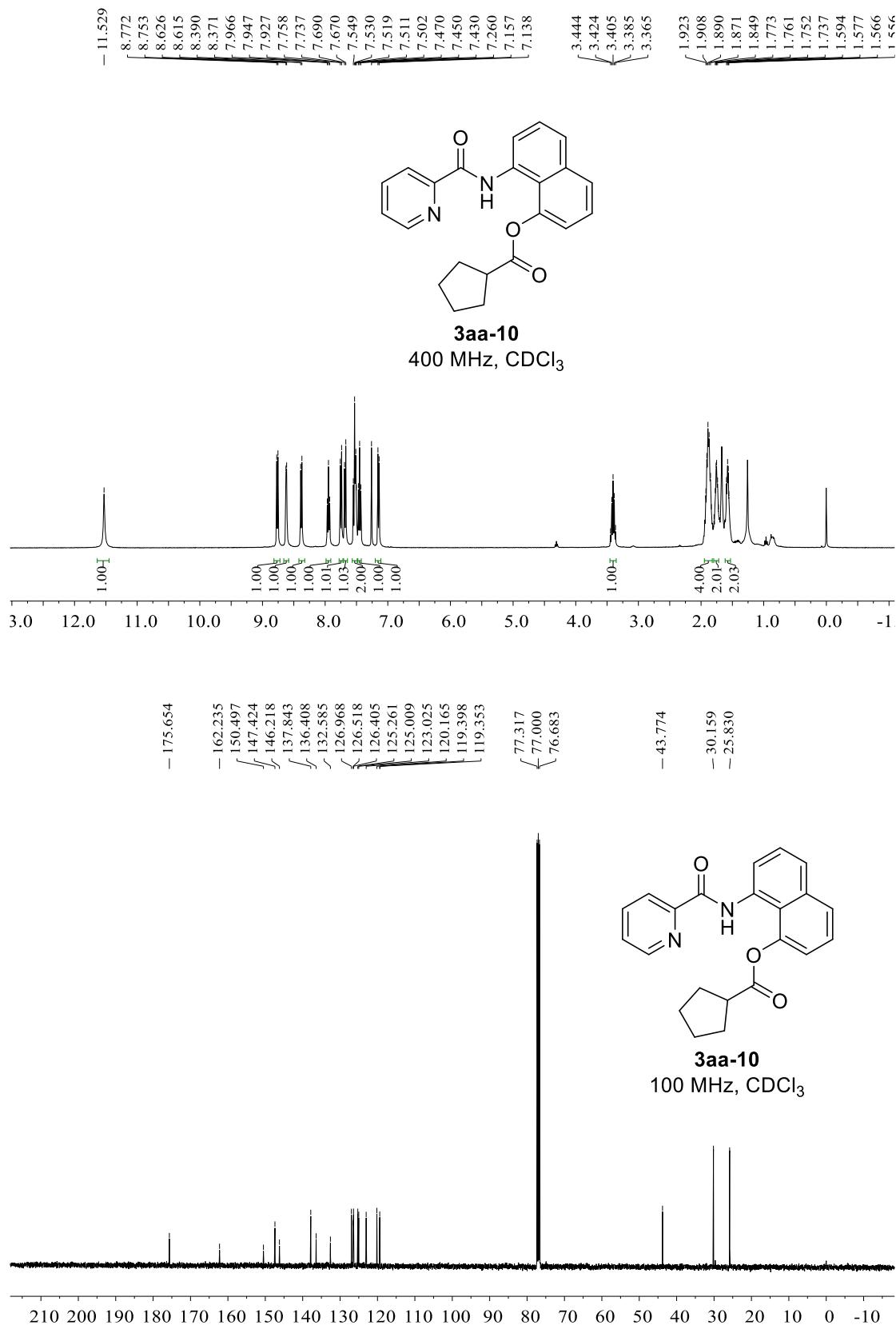


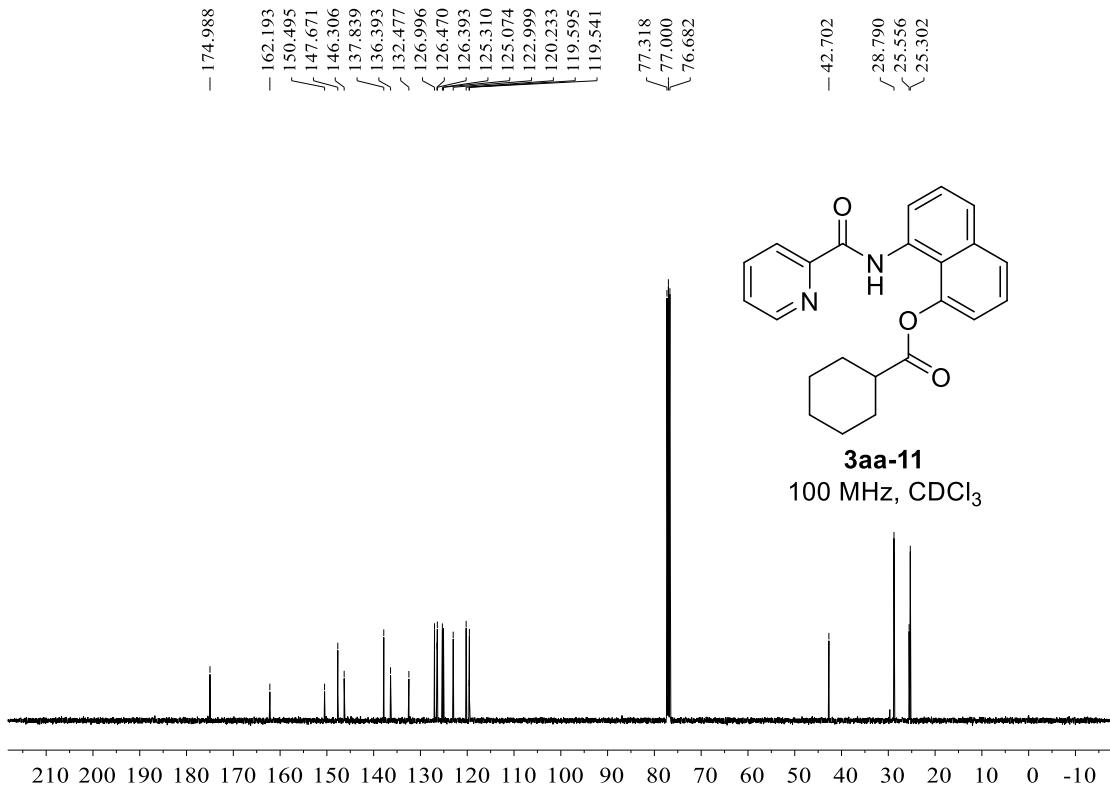
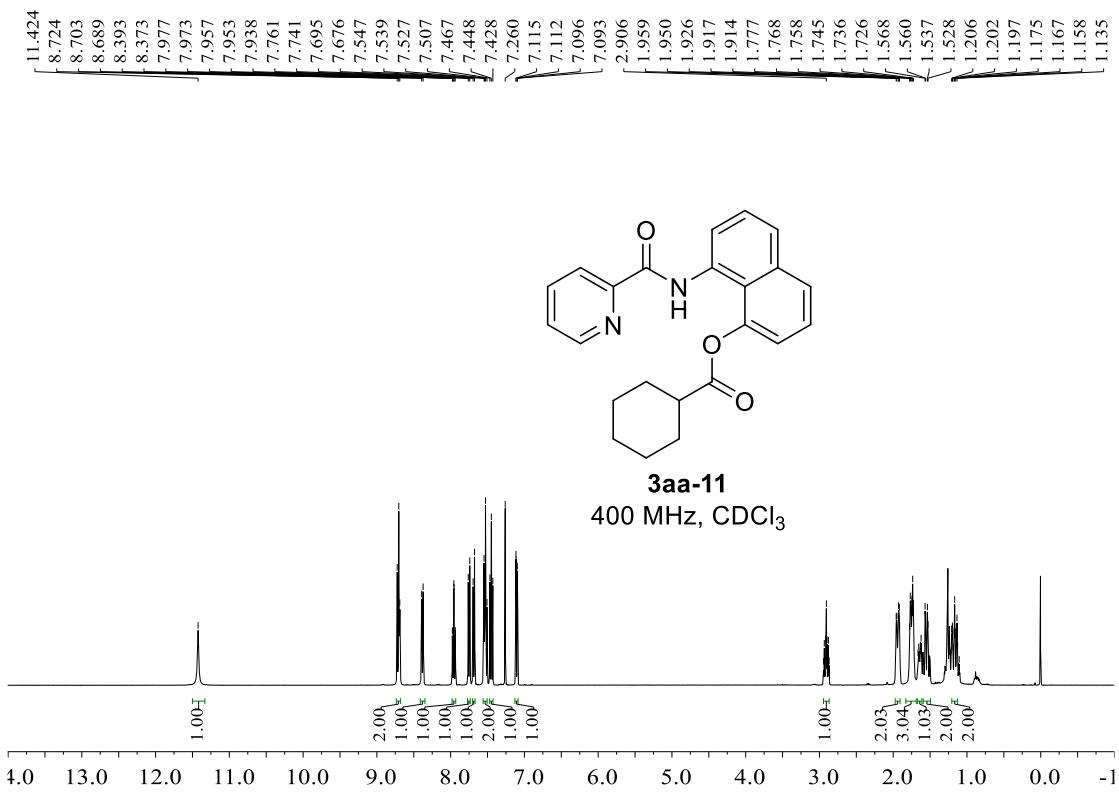


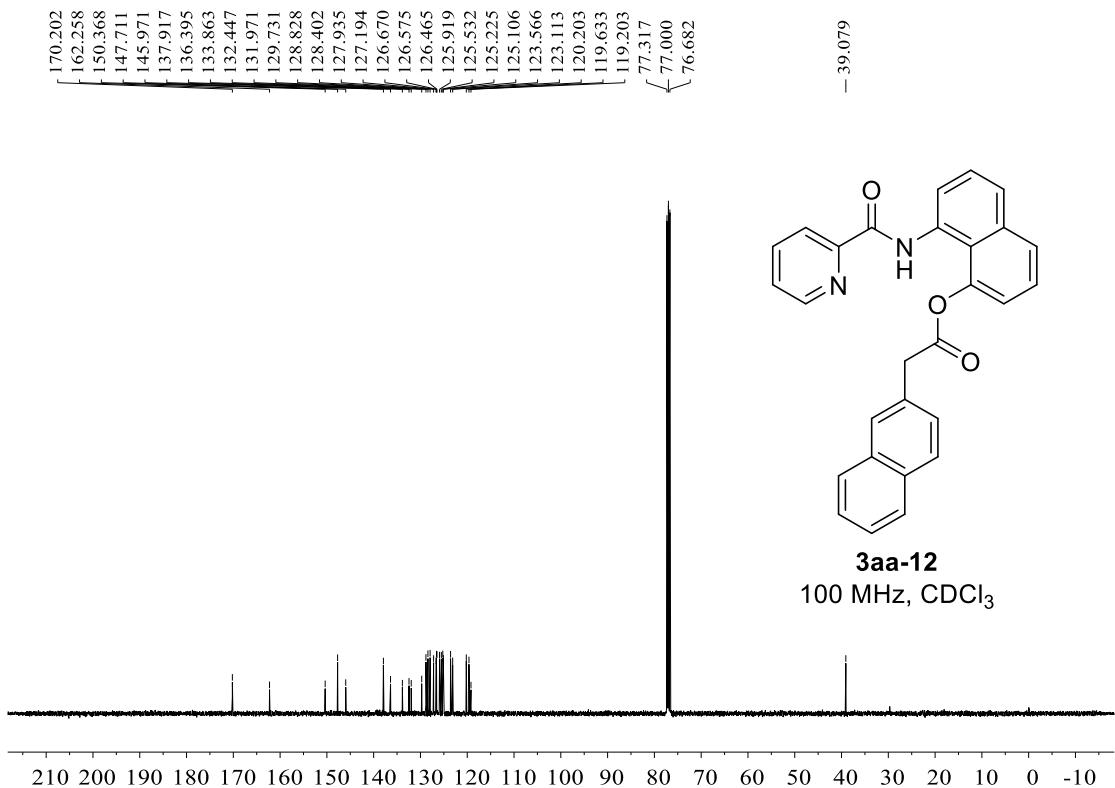
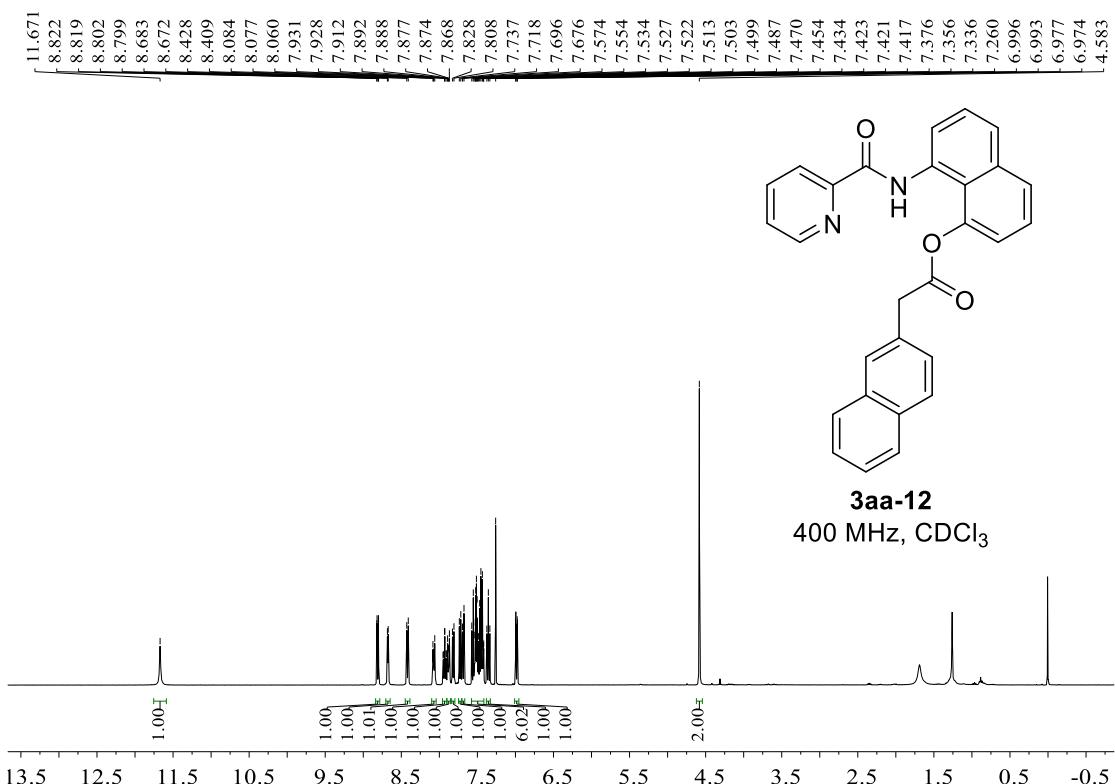


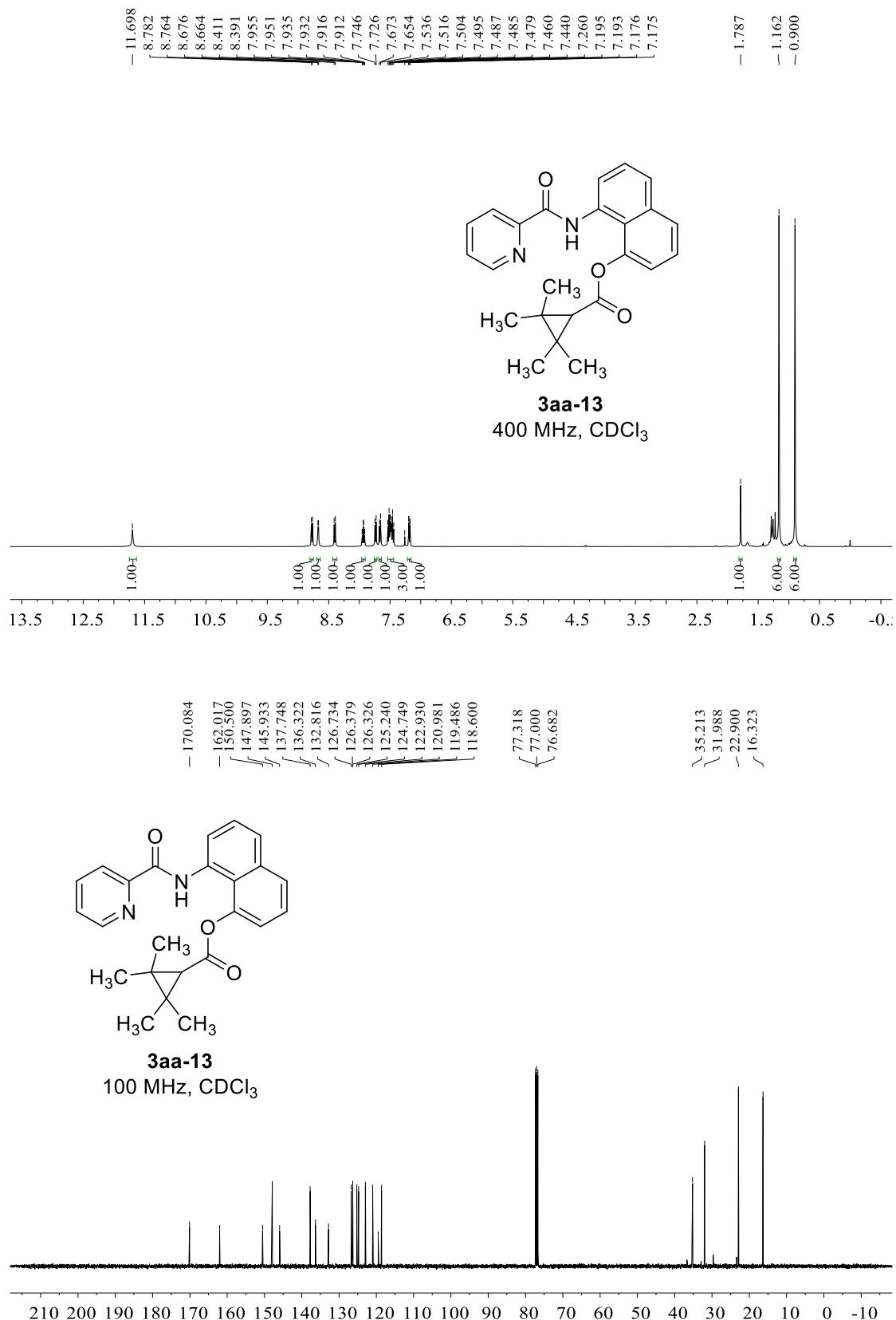


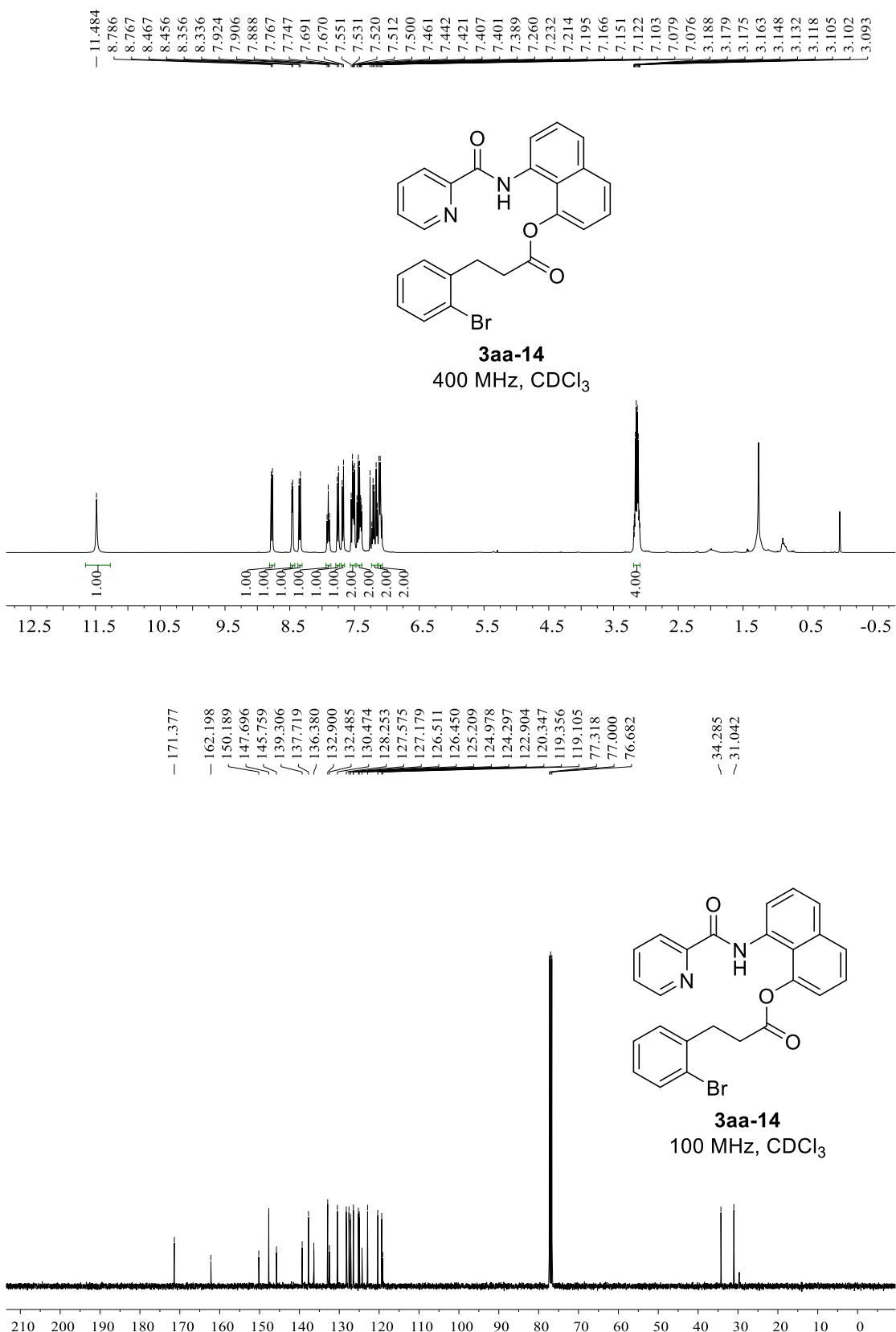


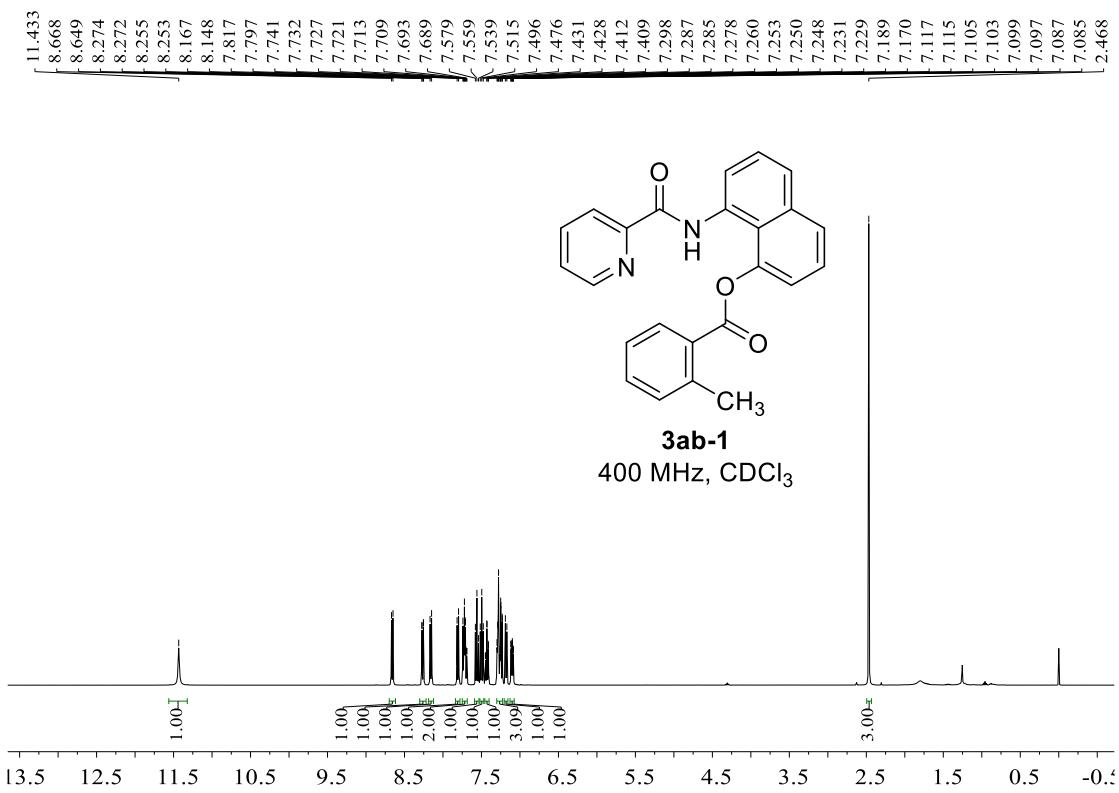




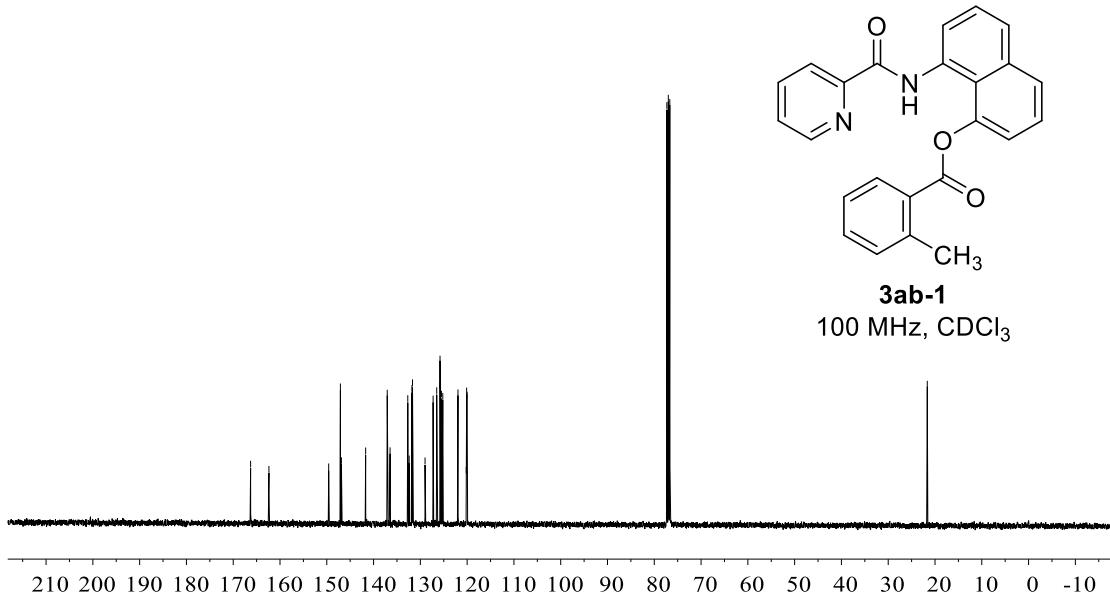


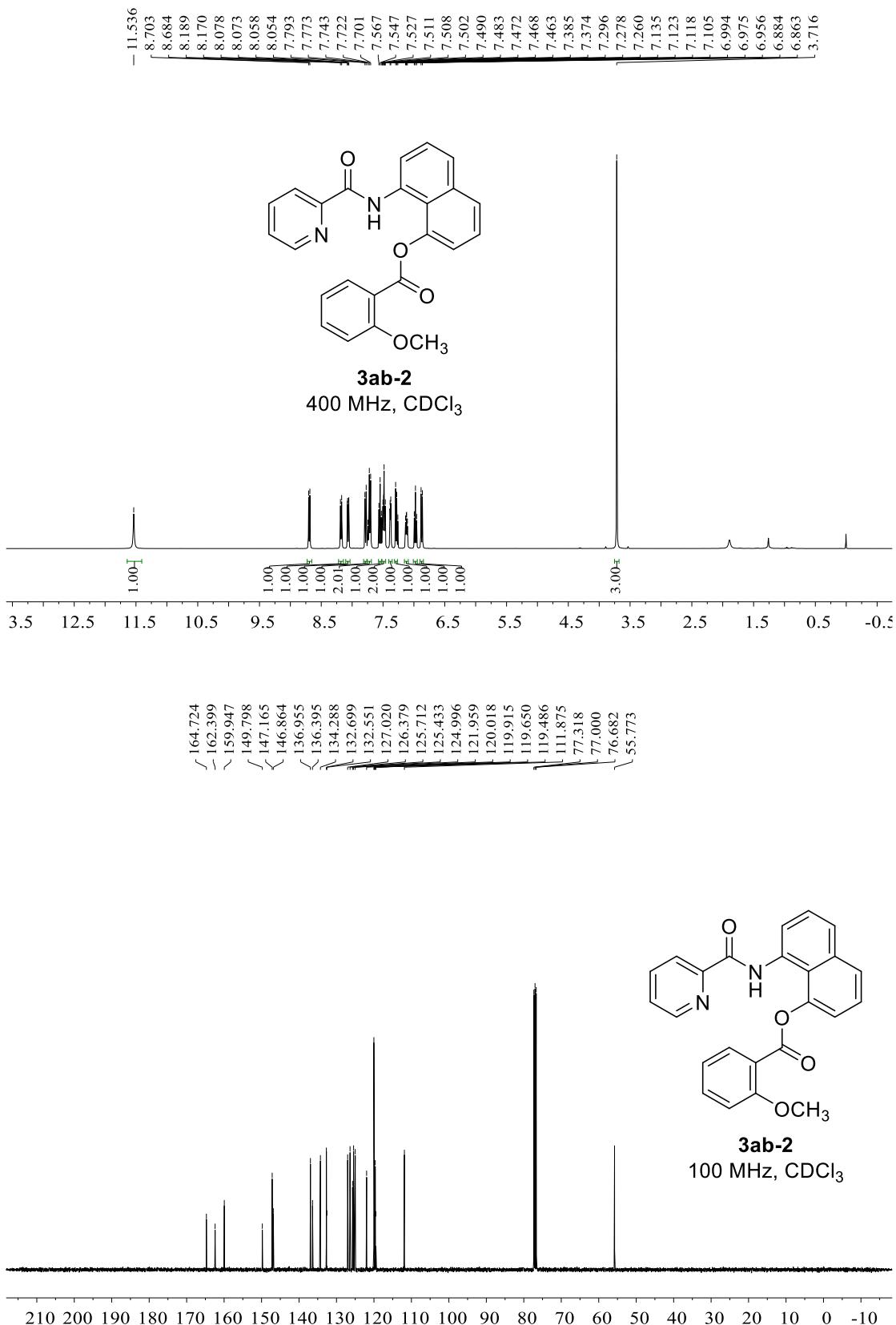


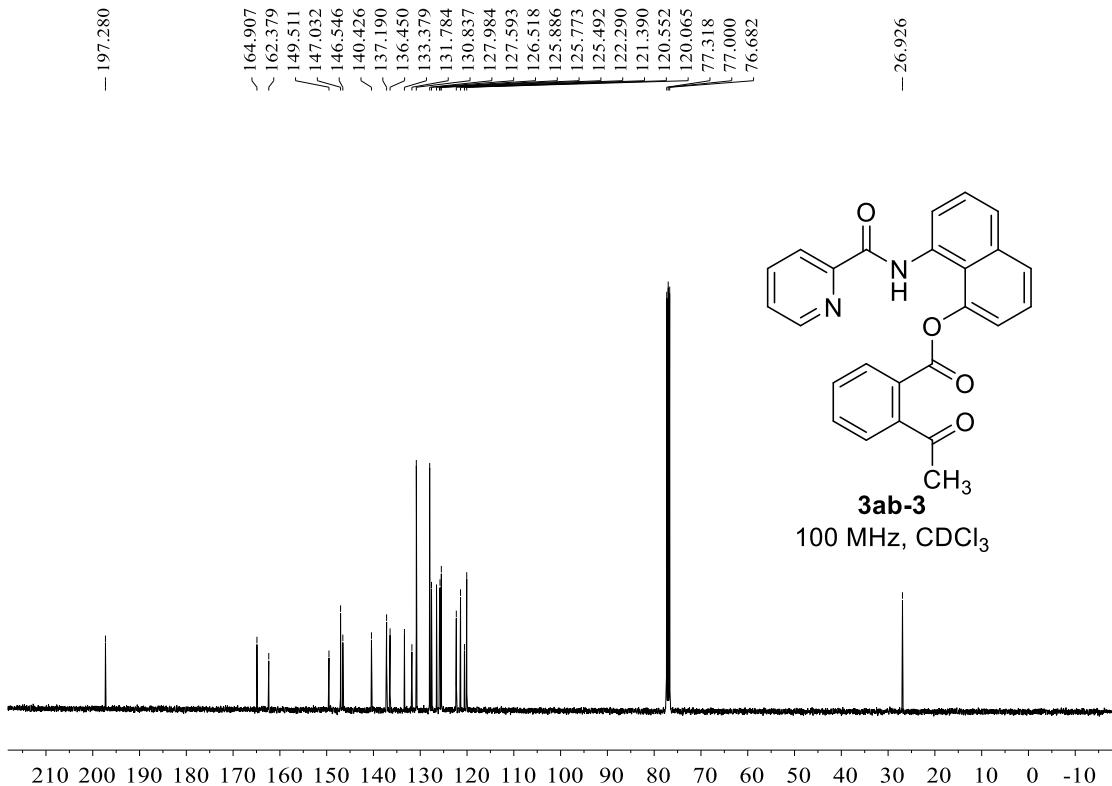
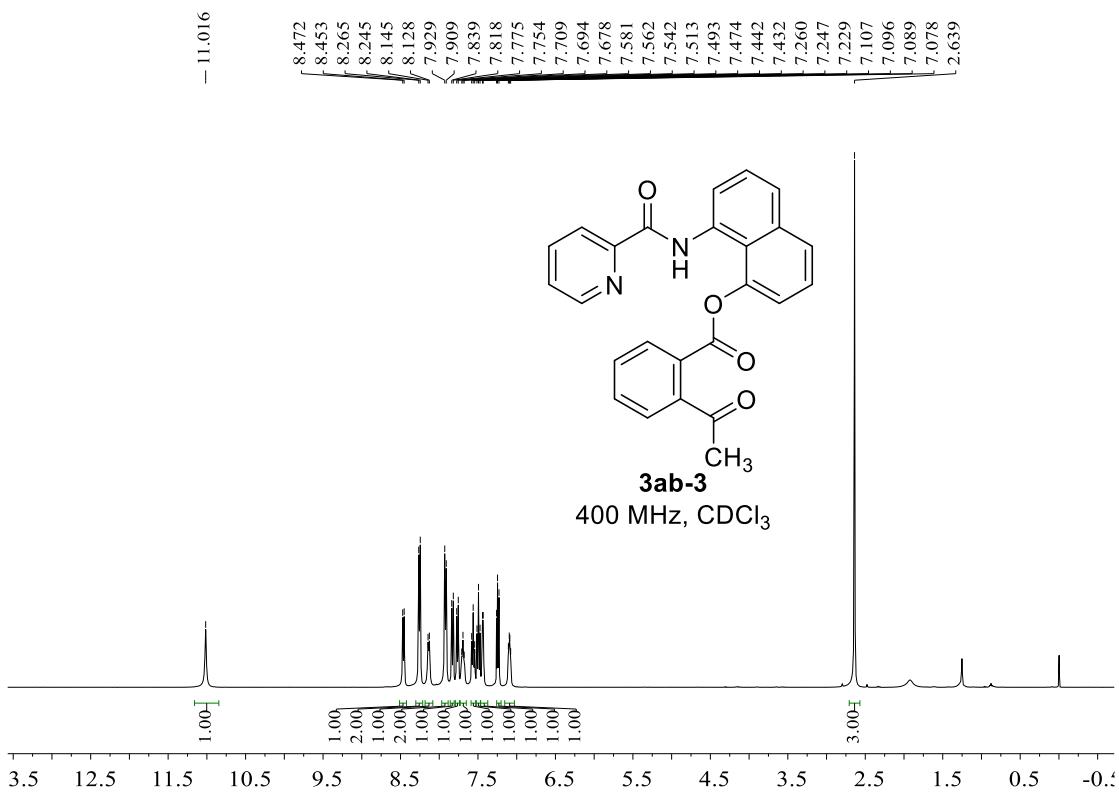


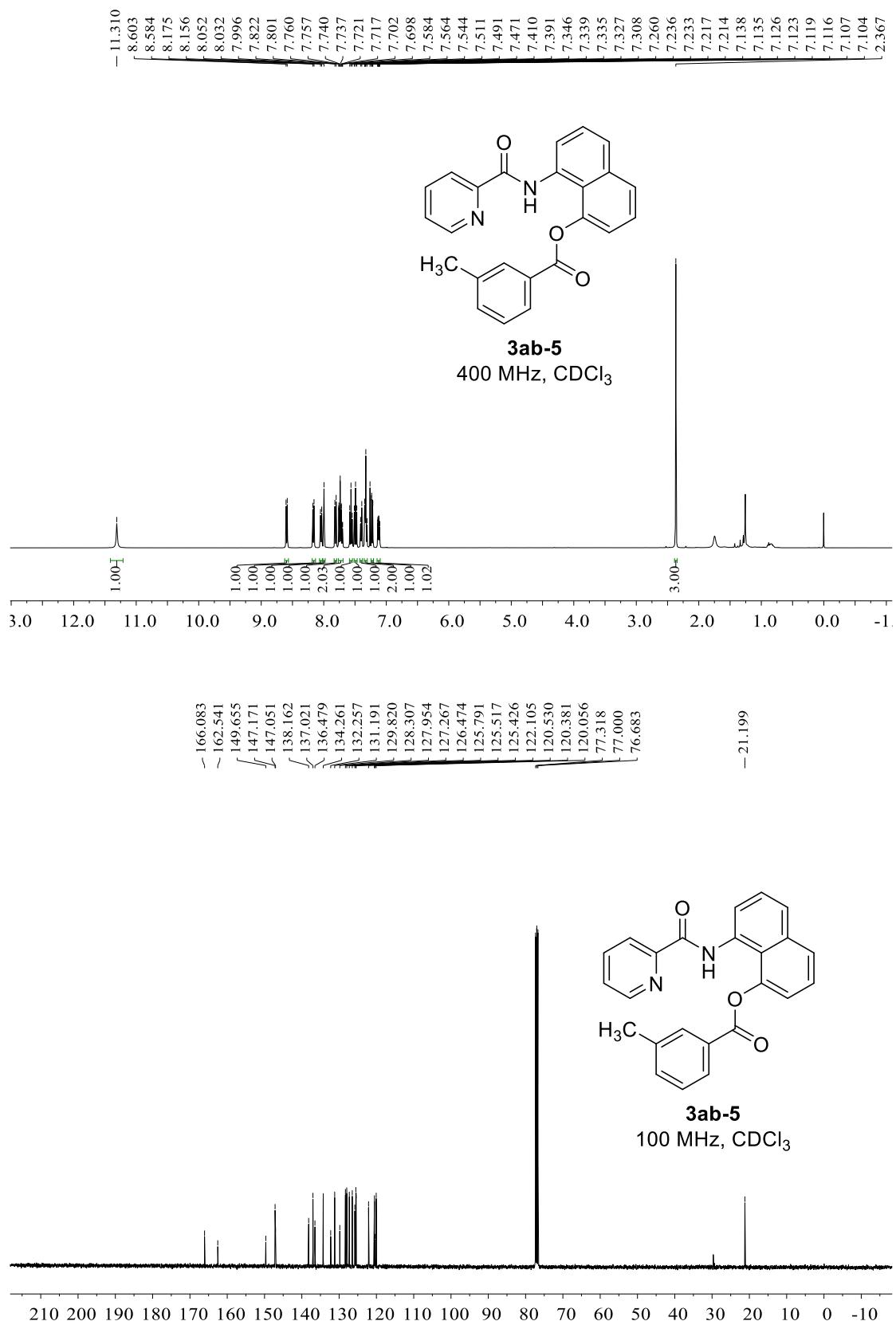


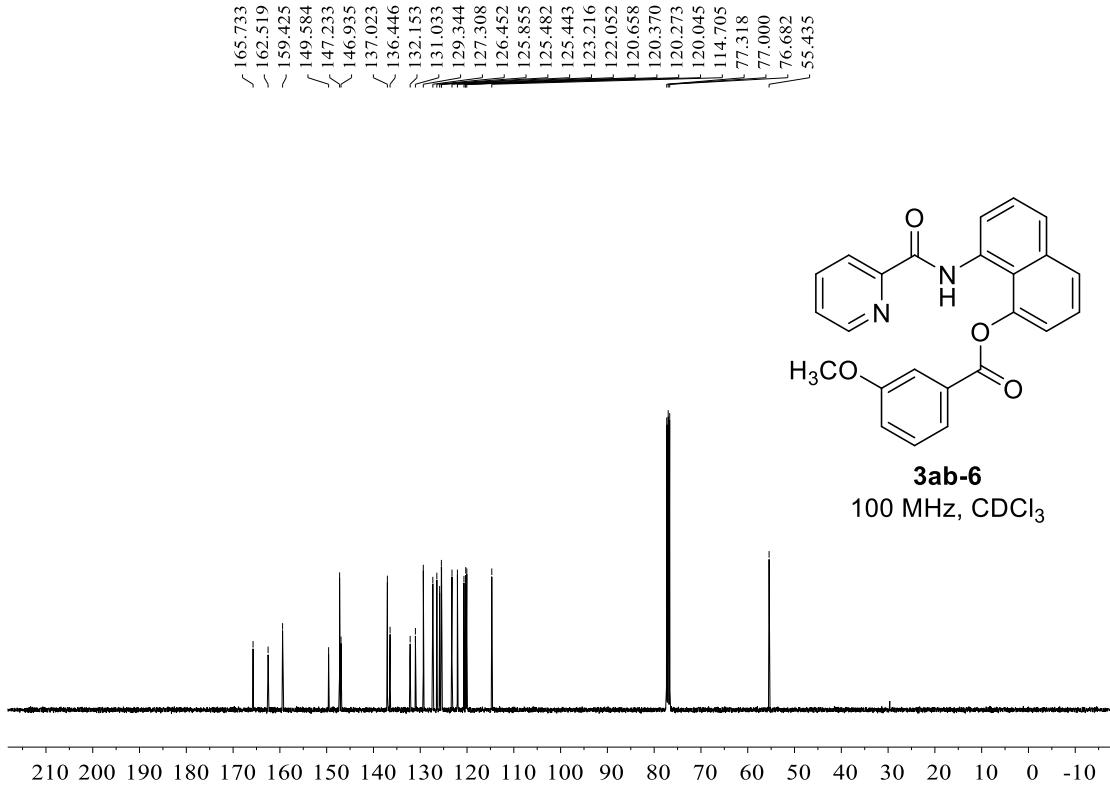
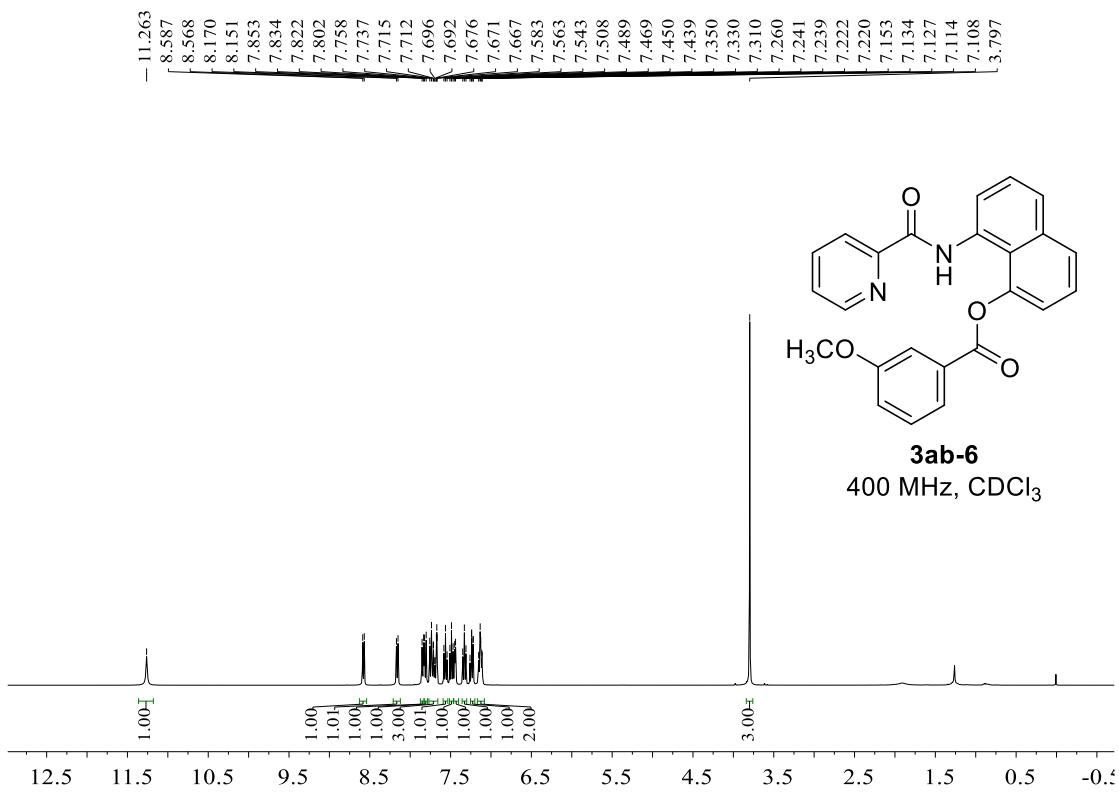
- 166.284
 - 162.366
 - 149.591
 - 147.089
 - 146.874
 > 141.694
 < 137.078
 < 136.474
 < 132.688
 - 132.397
 - 131.809
 - 131.679
 - 128.997
 - 127.274
 - 126.501
 - 125.811
 - 125.796
 - 125.492
 - 125.209
 - 121.962
 - 120.191
 - 120.104
 - 120.008
 - 77.317
 - 77.000
 - 76.682
 - 21.649

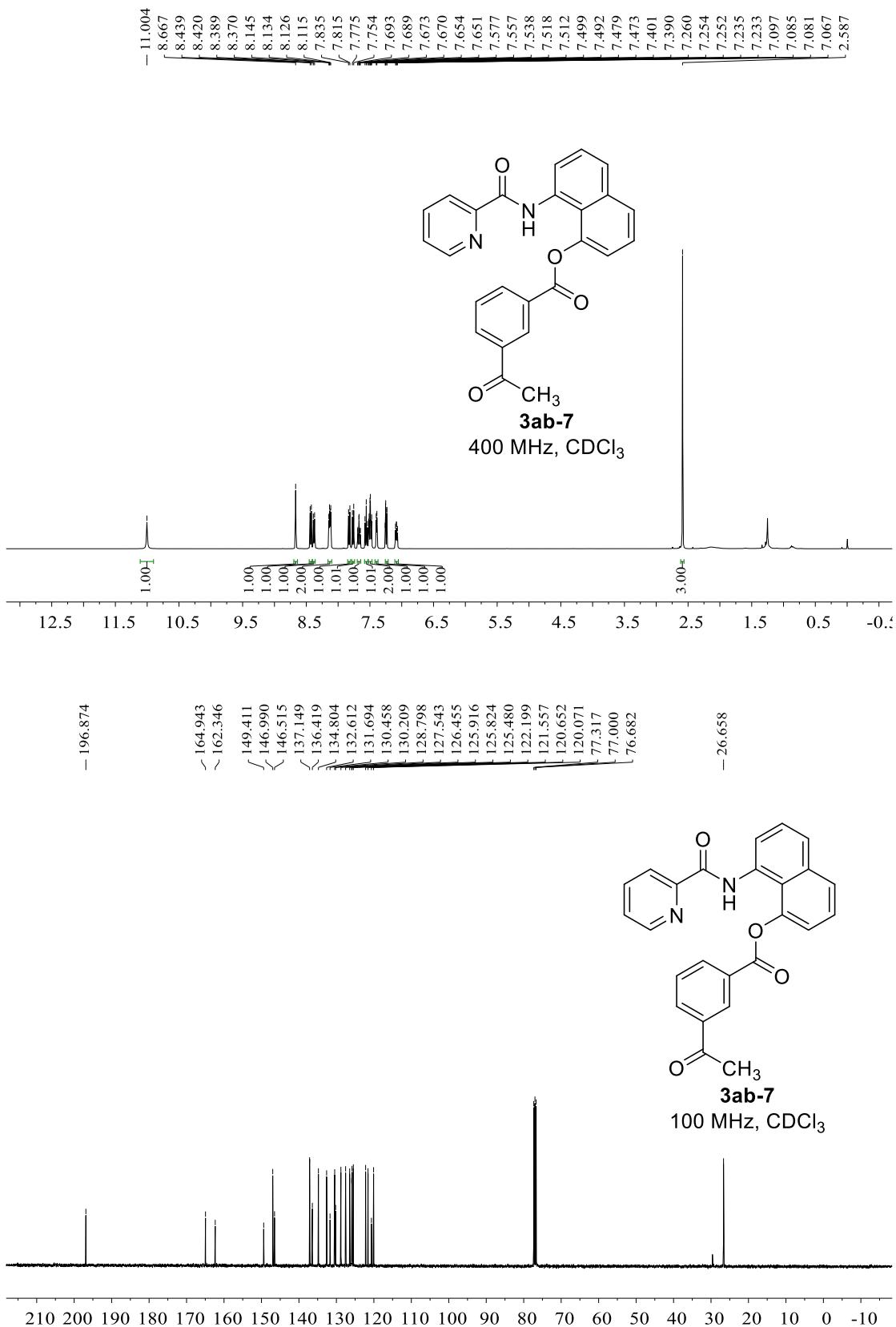


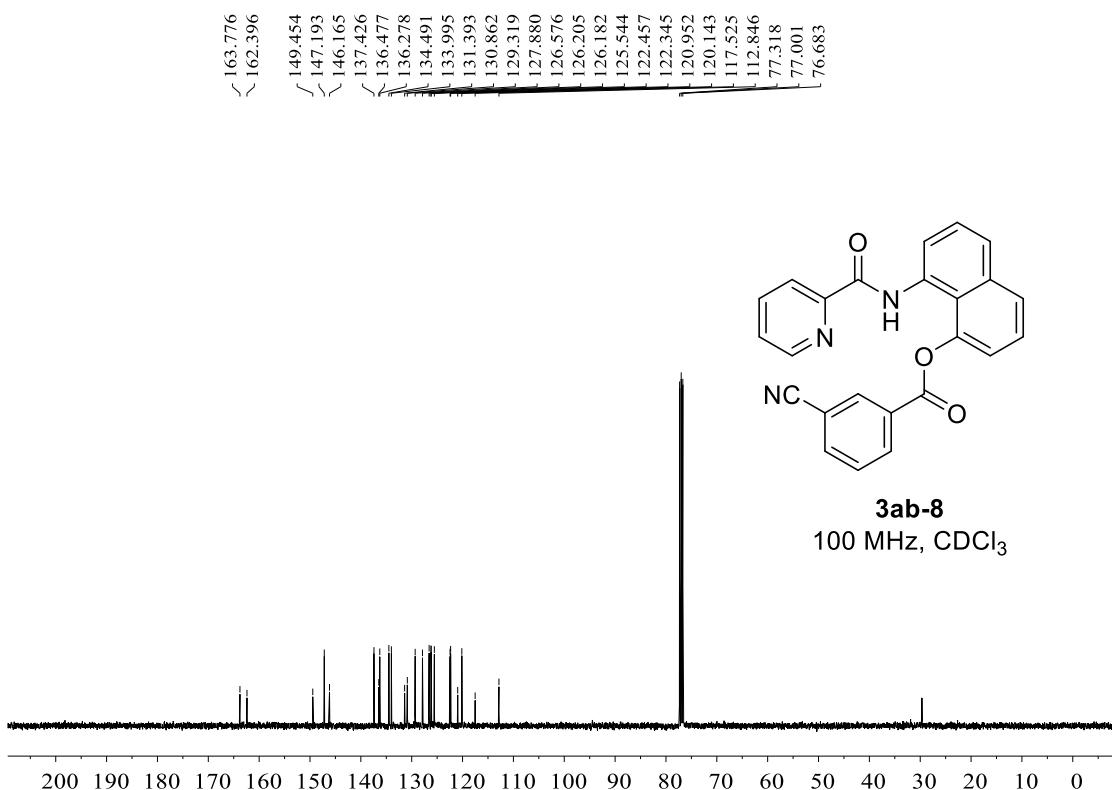
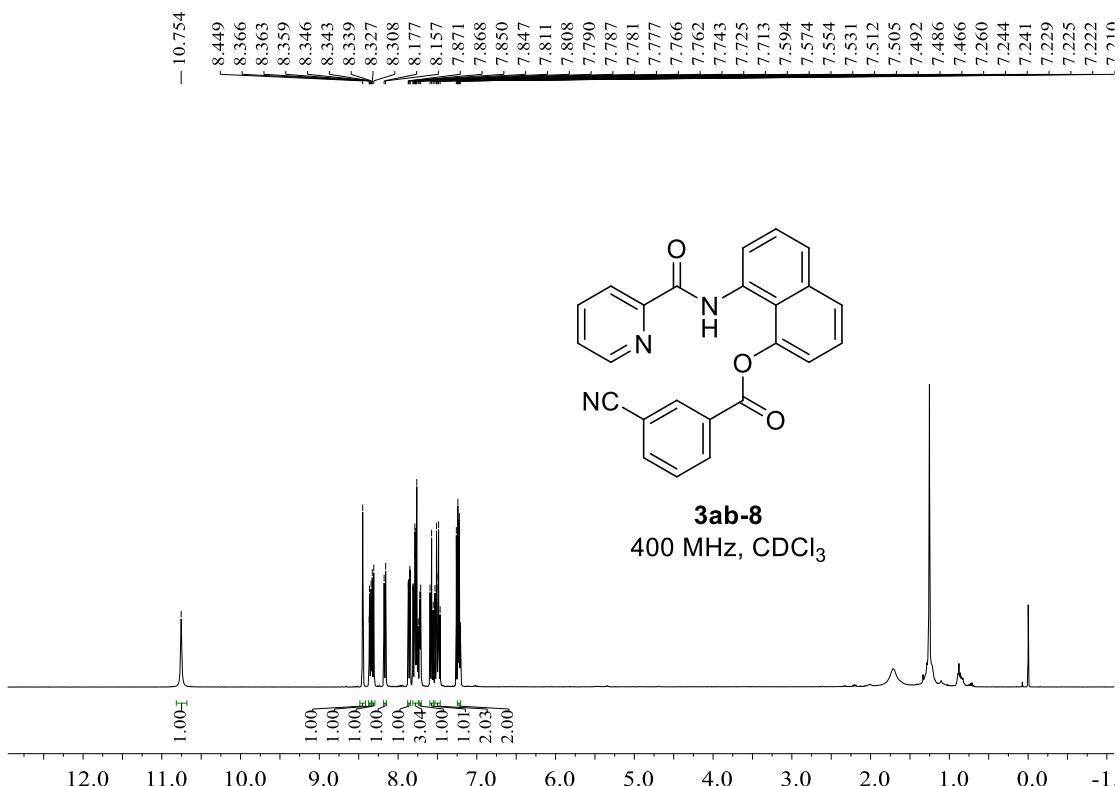


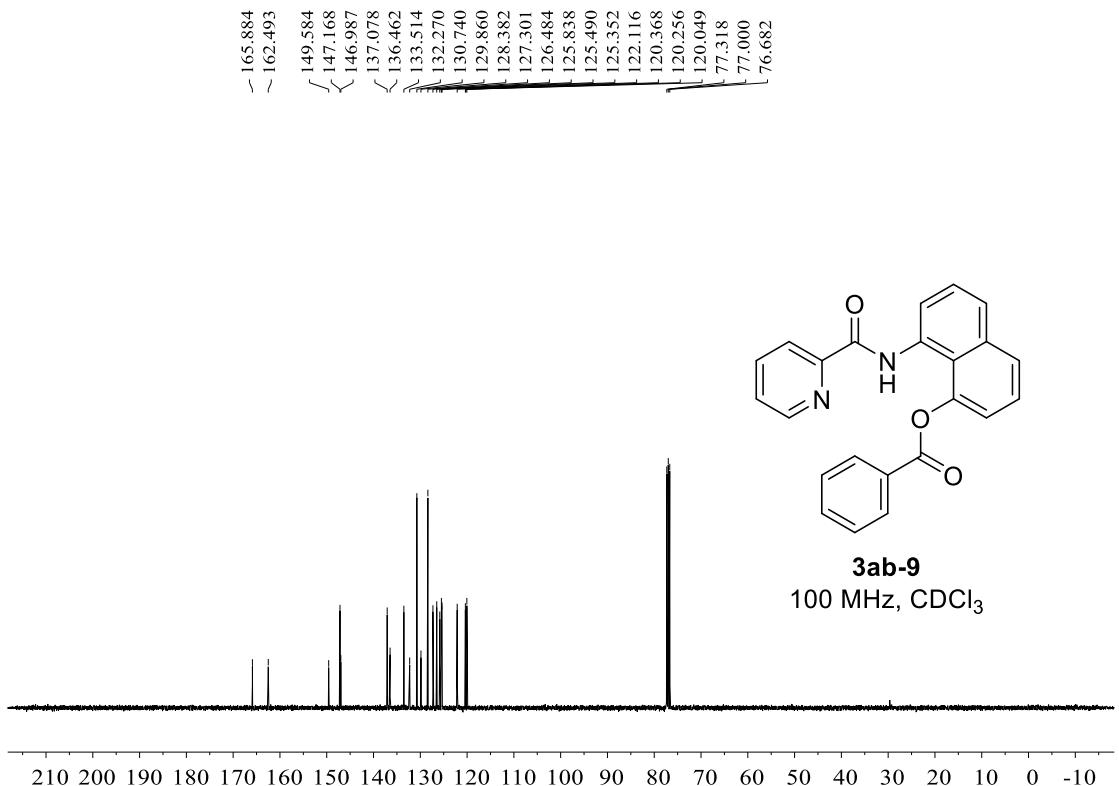
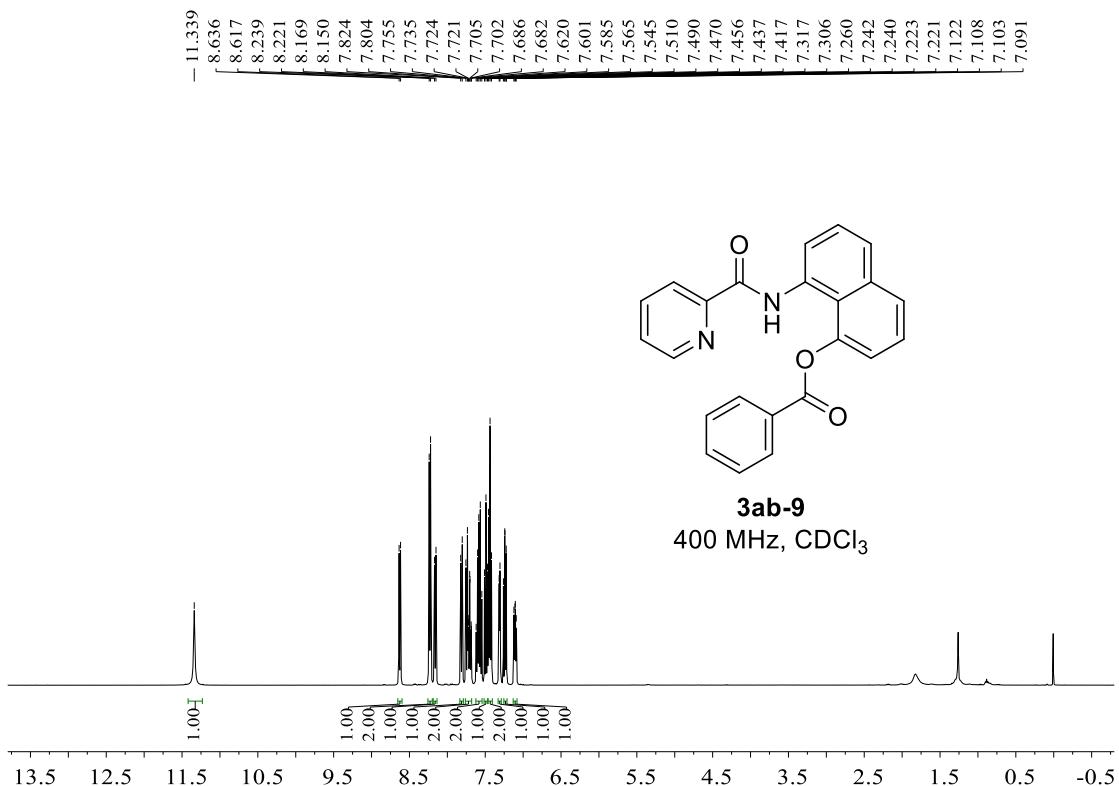


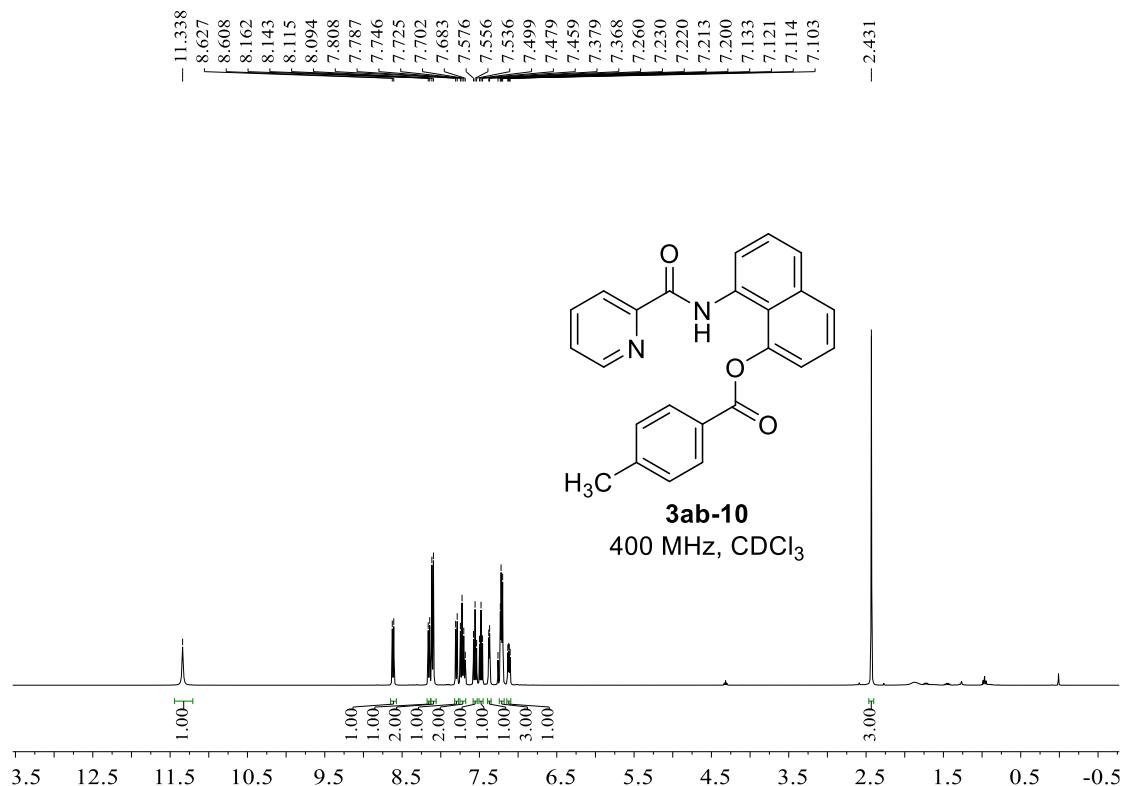




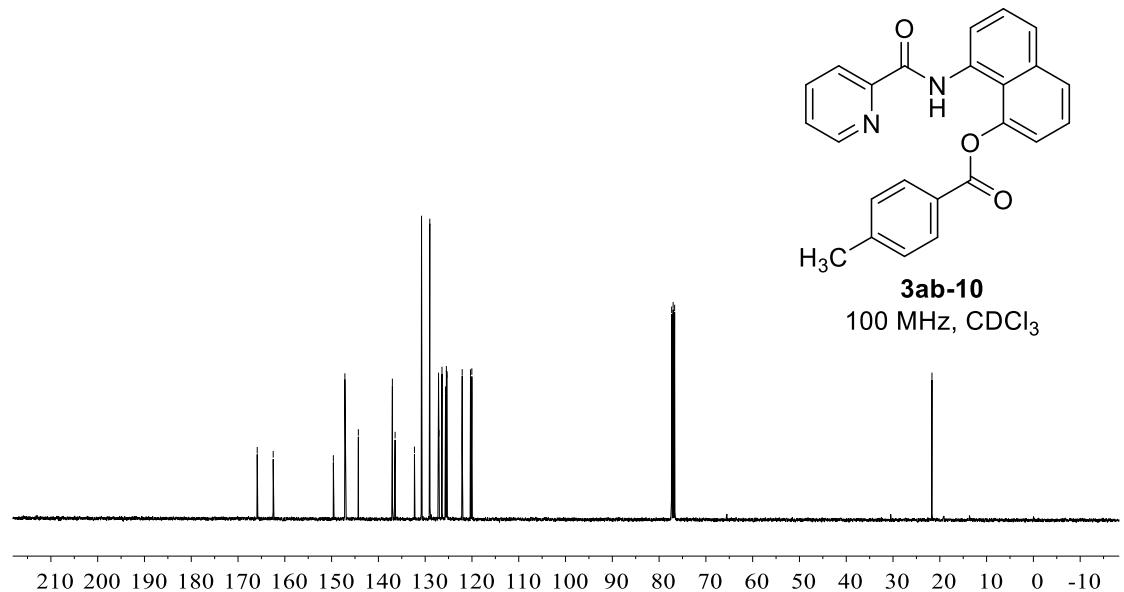


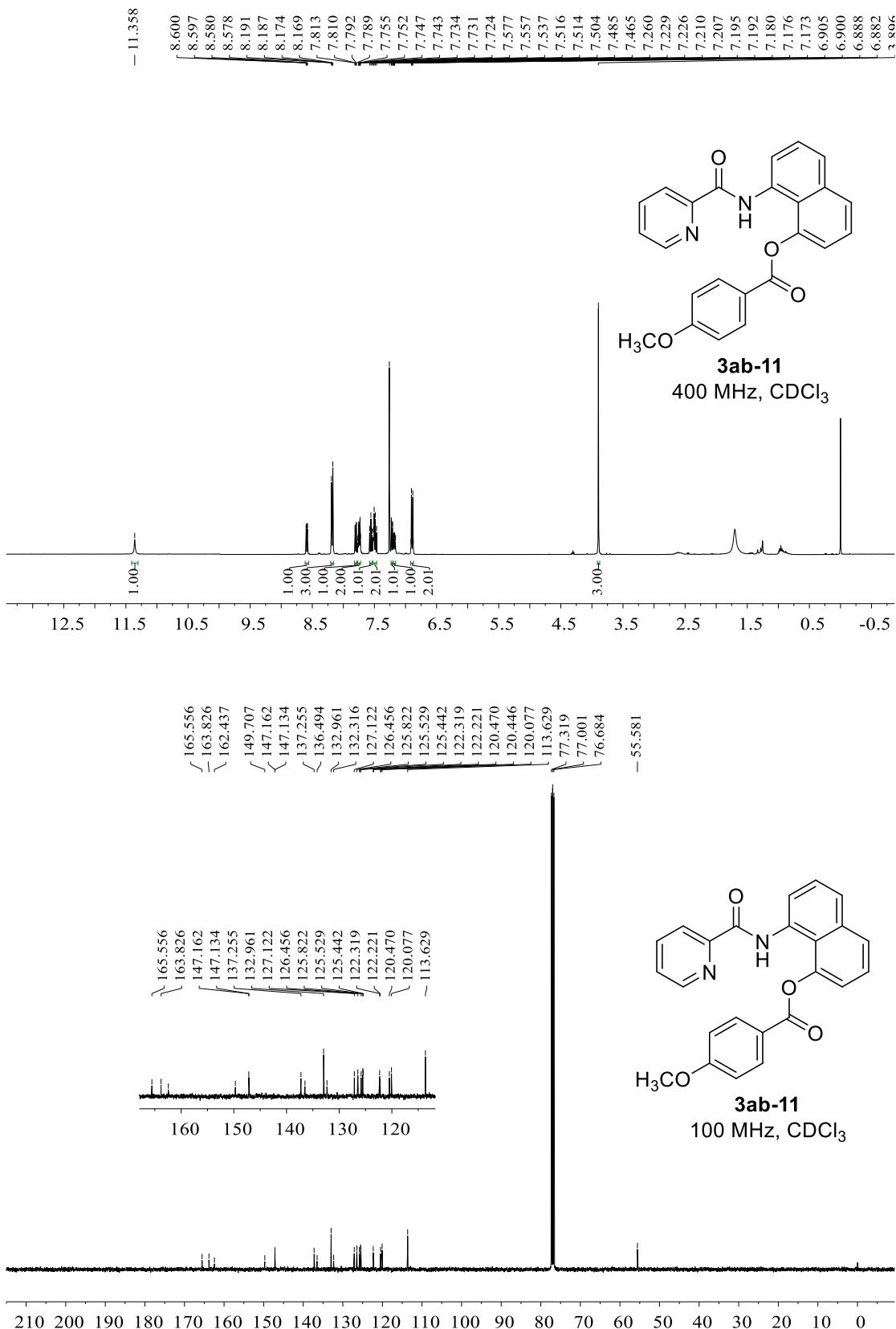


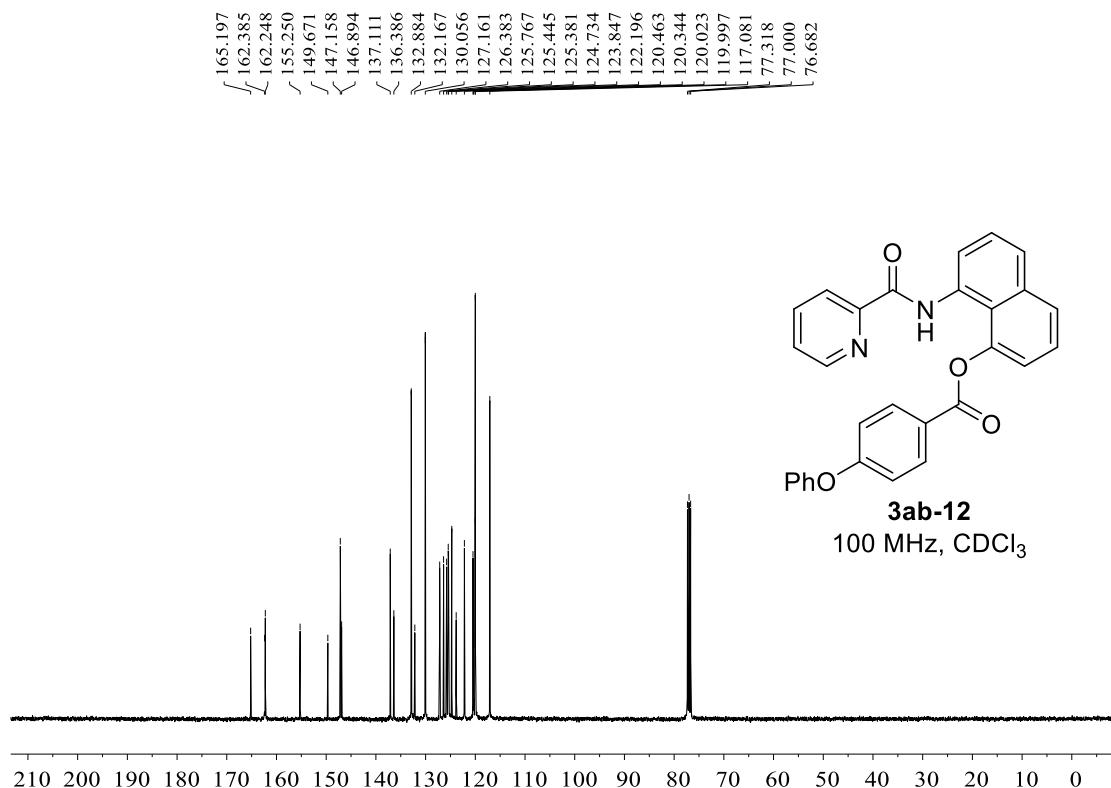
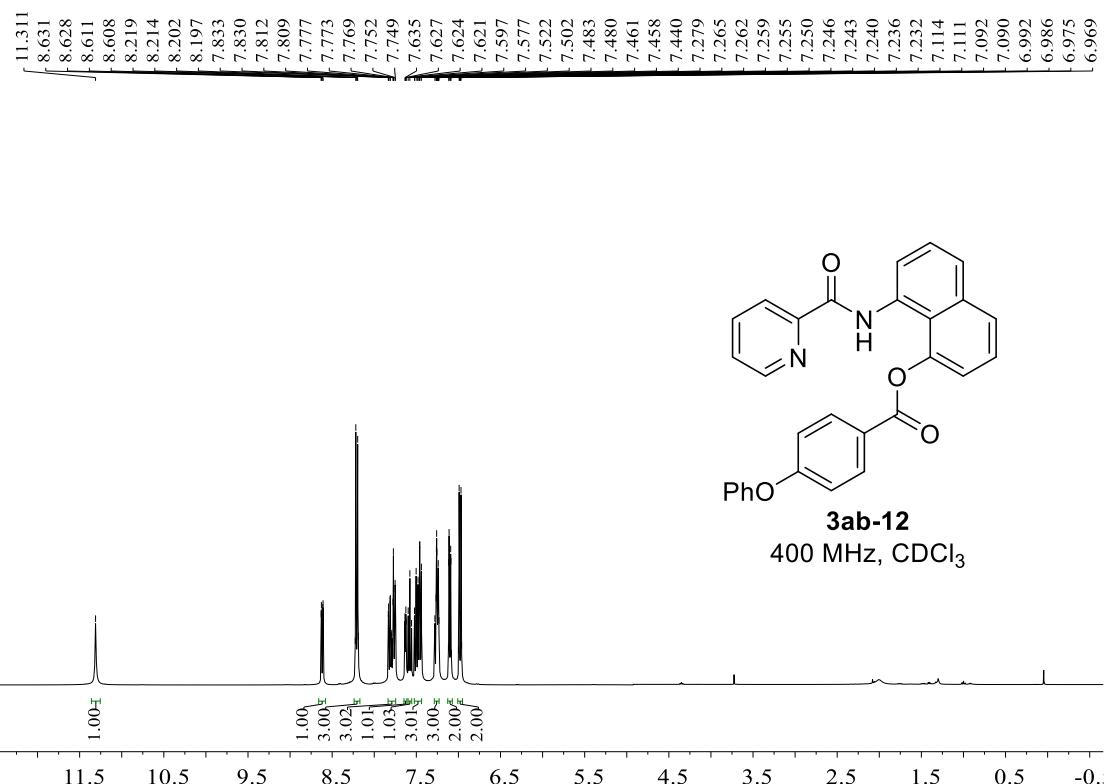


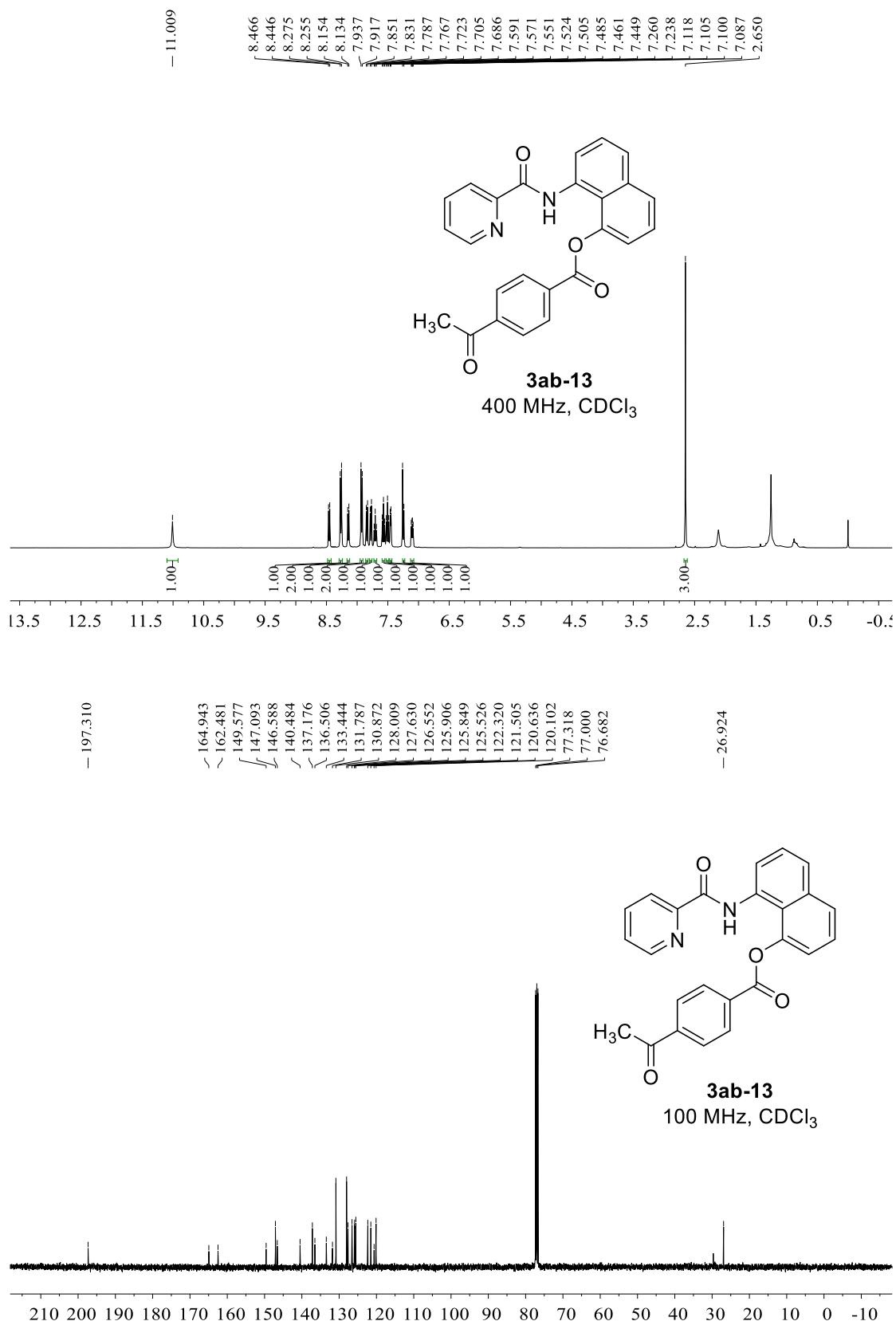


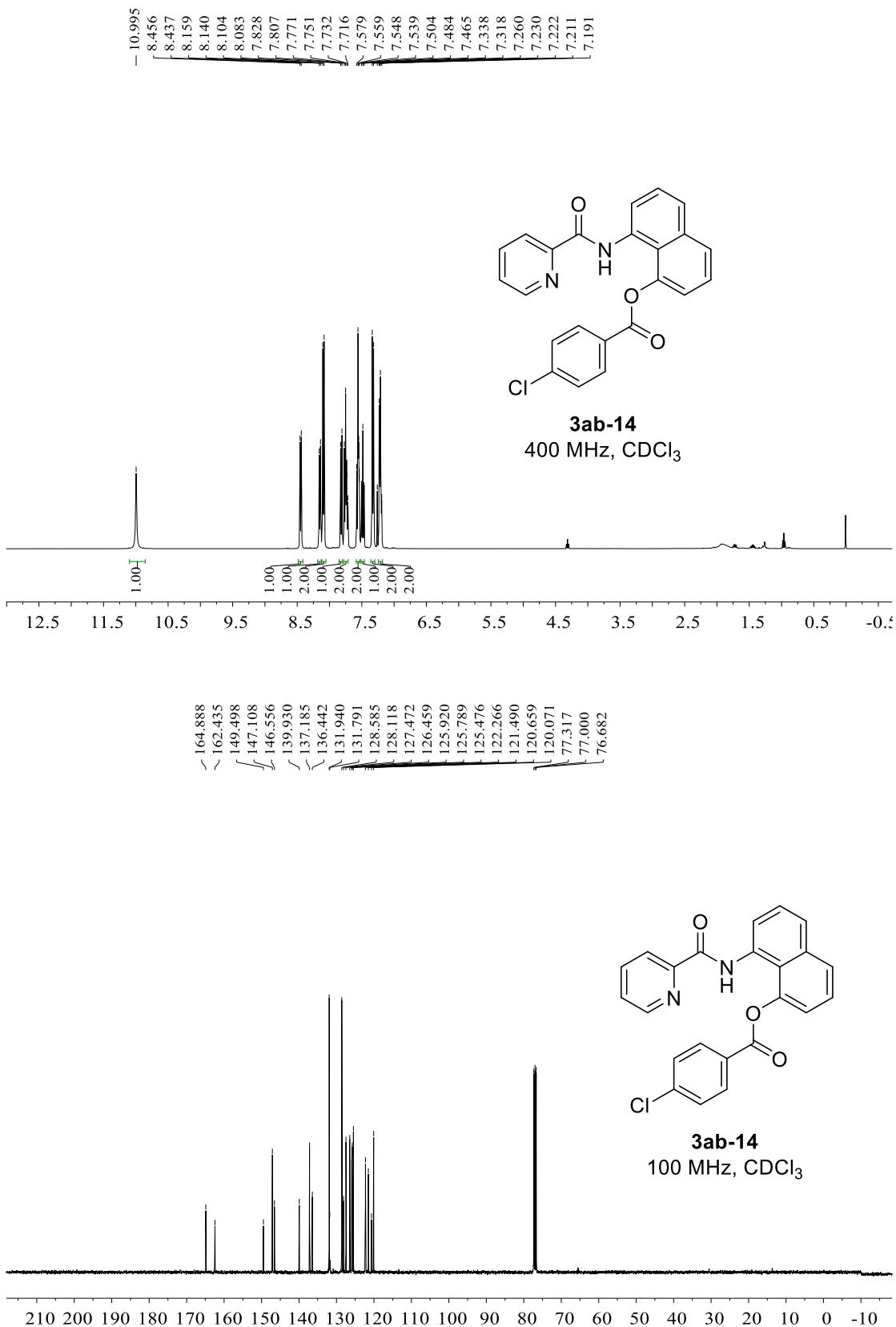
δ (ppm): 11.338, 8.627, 8.608, 8.162, 8.143, 8.115, 8.094, 7.808, 7.787, 7.746, 7.725, 7.702, 7.683, 7.576, 7.556, 7.536, 7.499, 7.479, 7.459, 7.379, 7.368, 7.260, 7.230, 7.220, 7.213, 7.200, 7.133, 7.121, 7.114, 7.103.

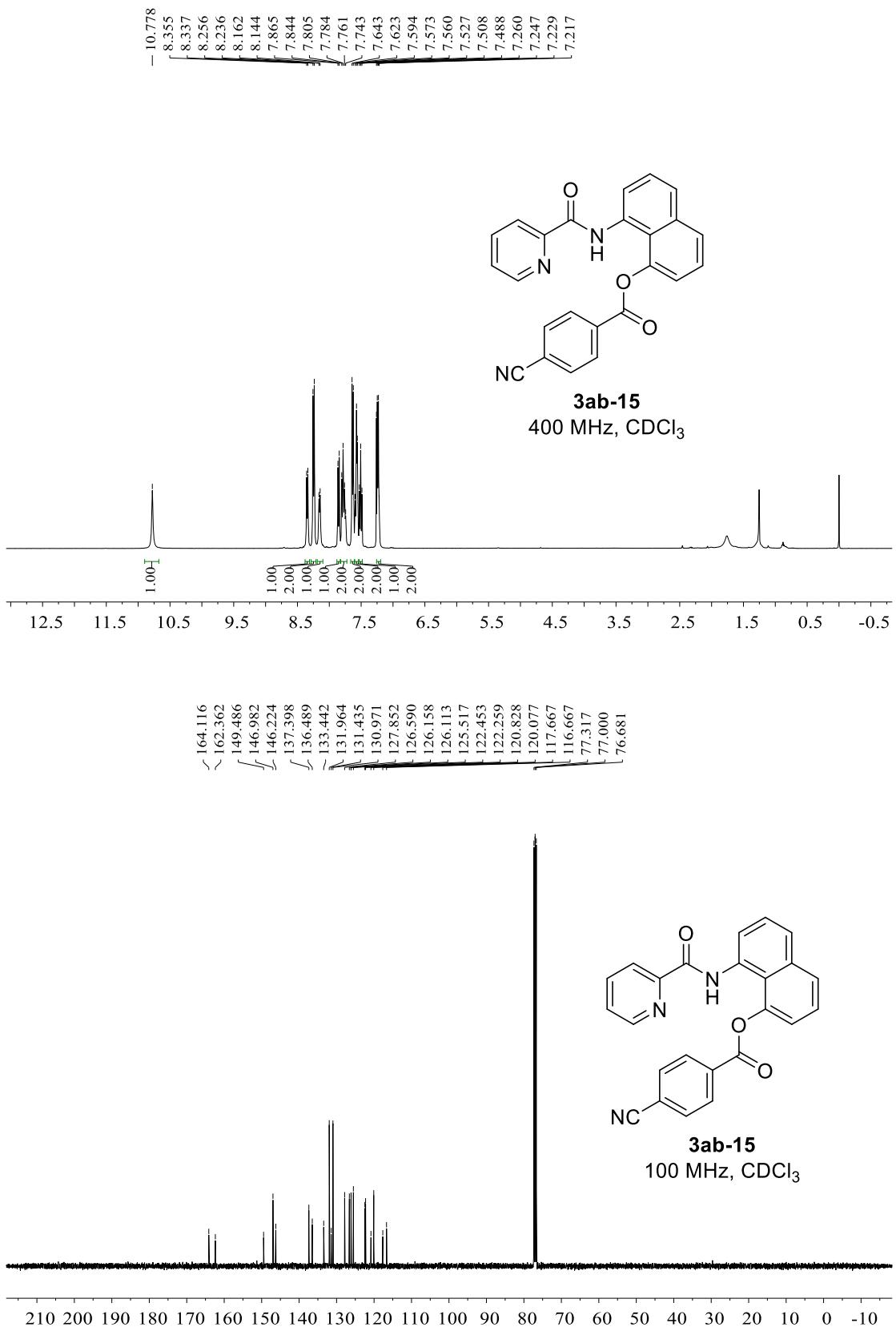


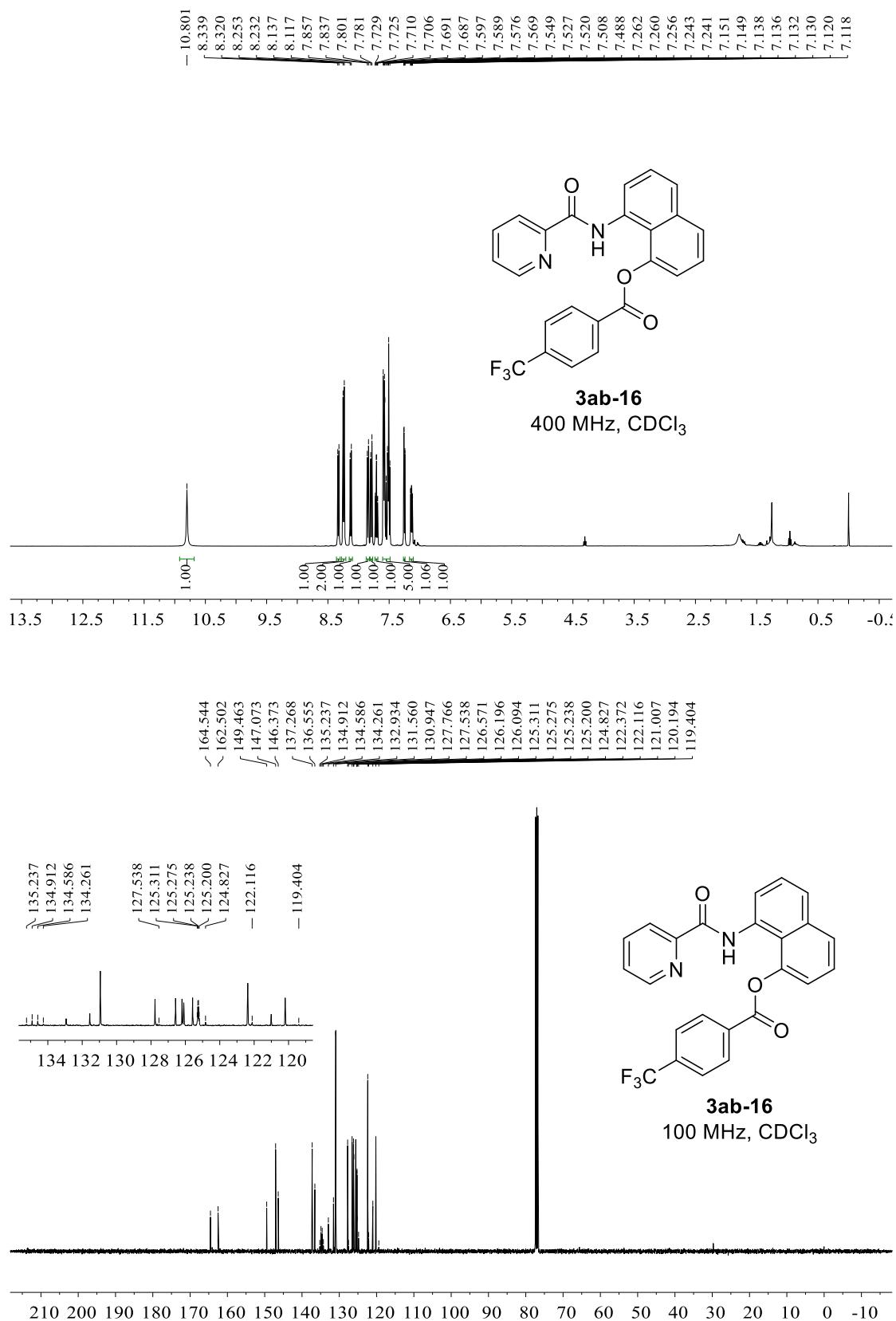




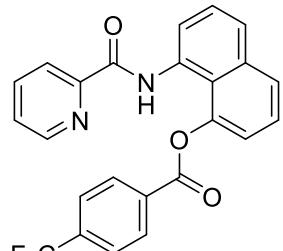




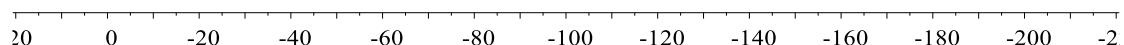


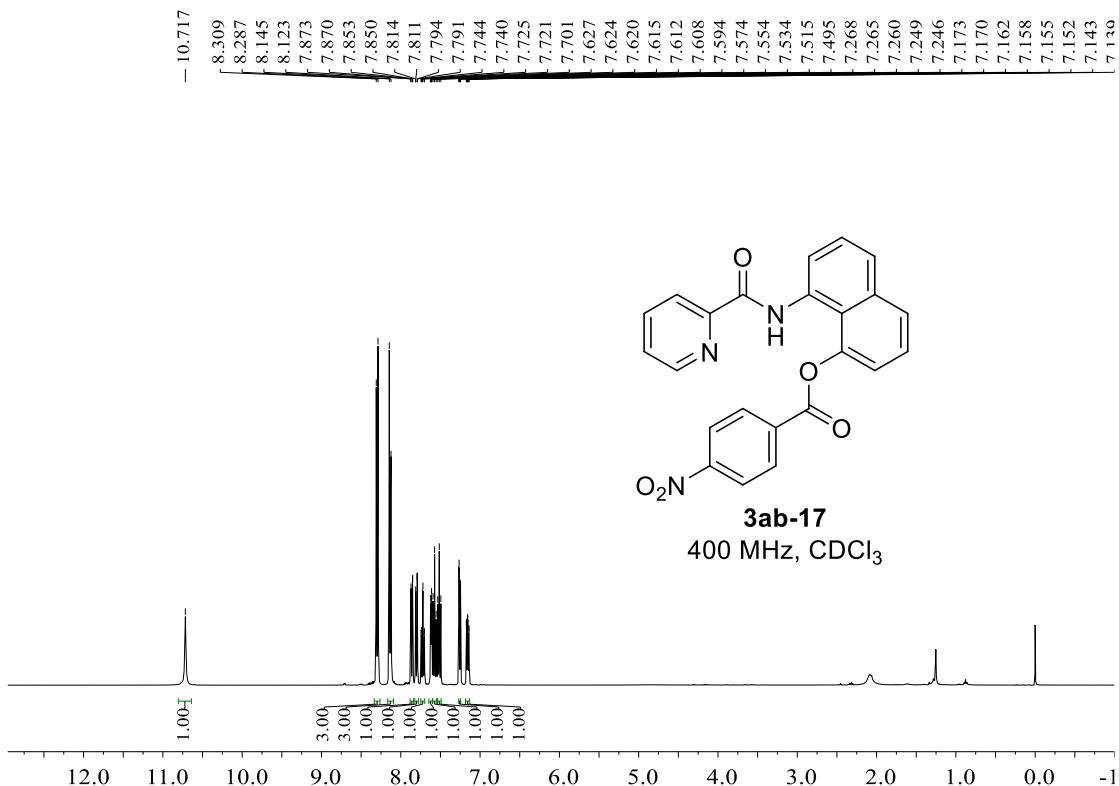


- -63.229

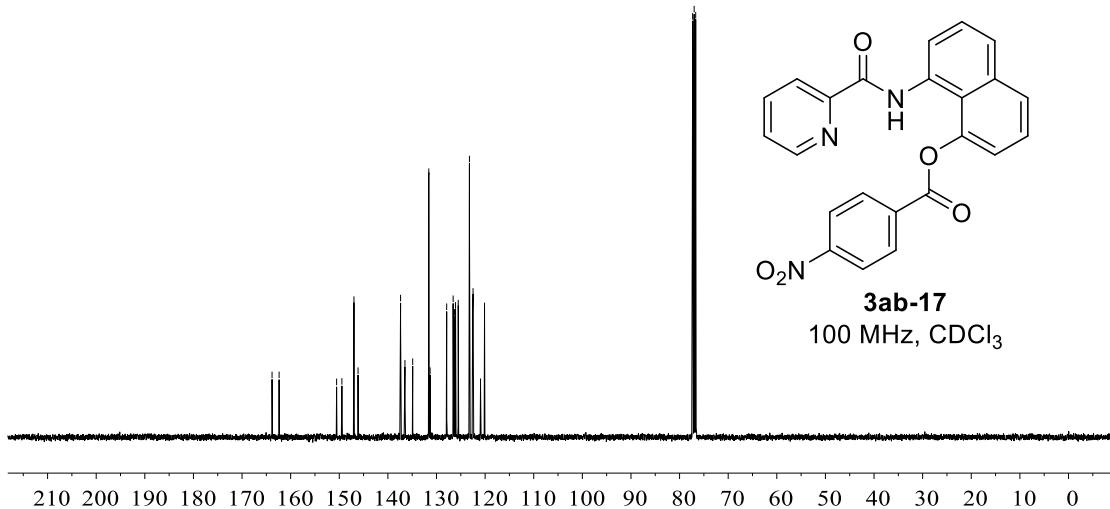


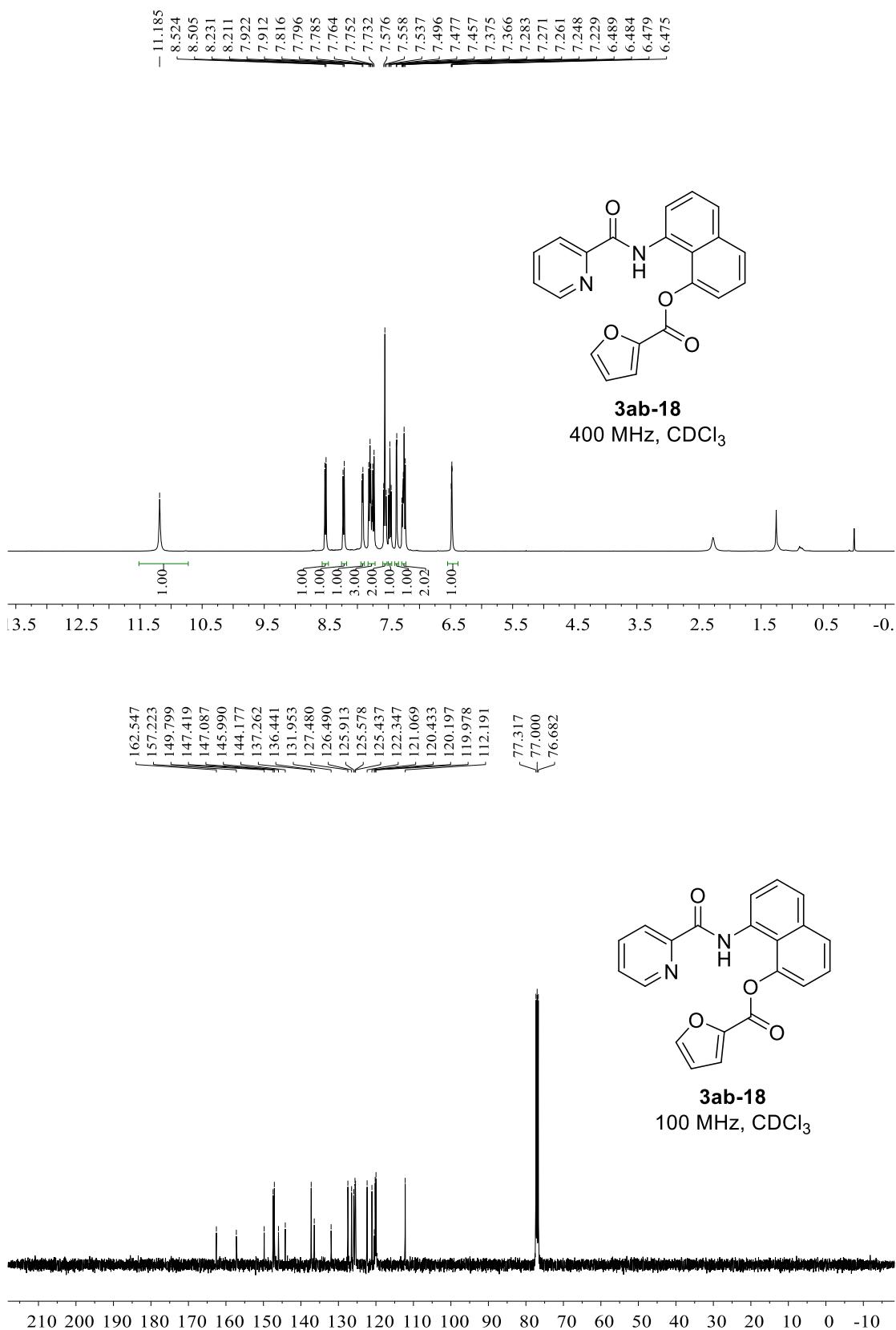
3ab-16
376 MHz, CDCl₃

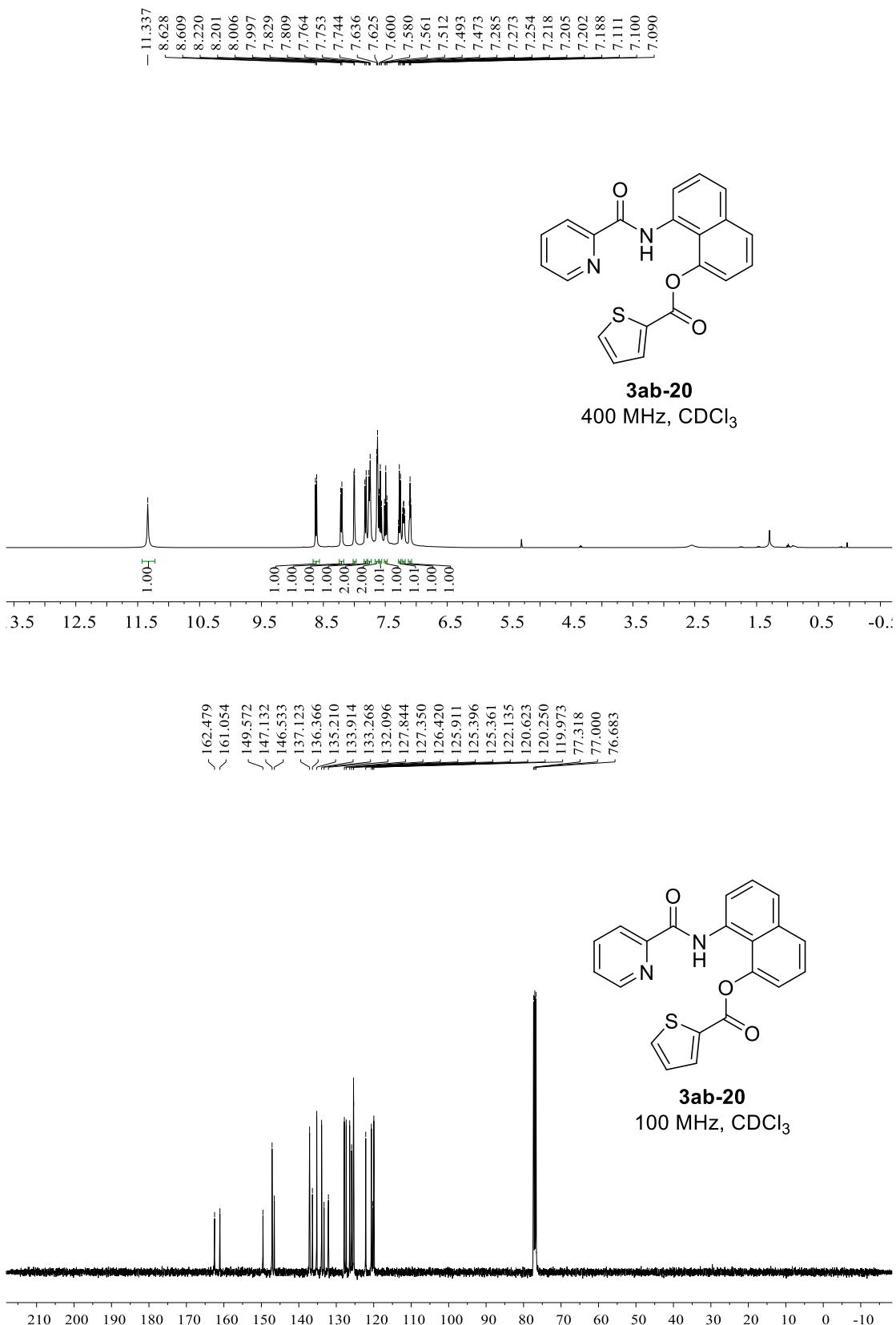


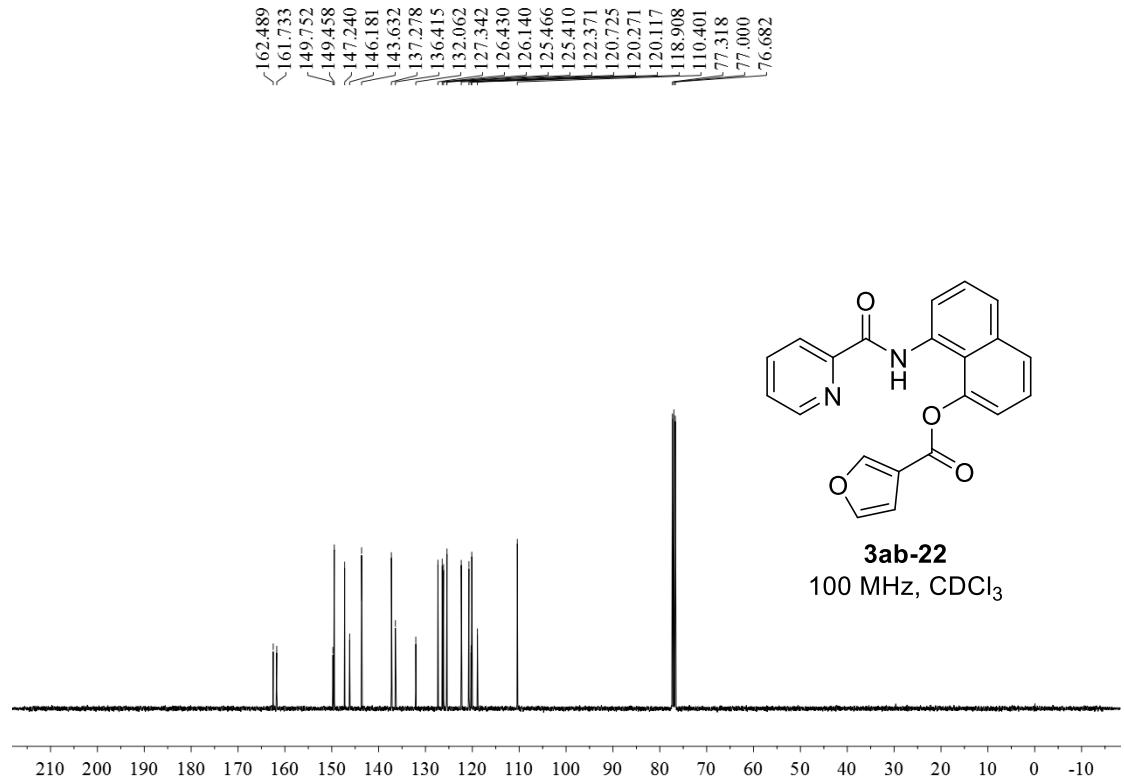
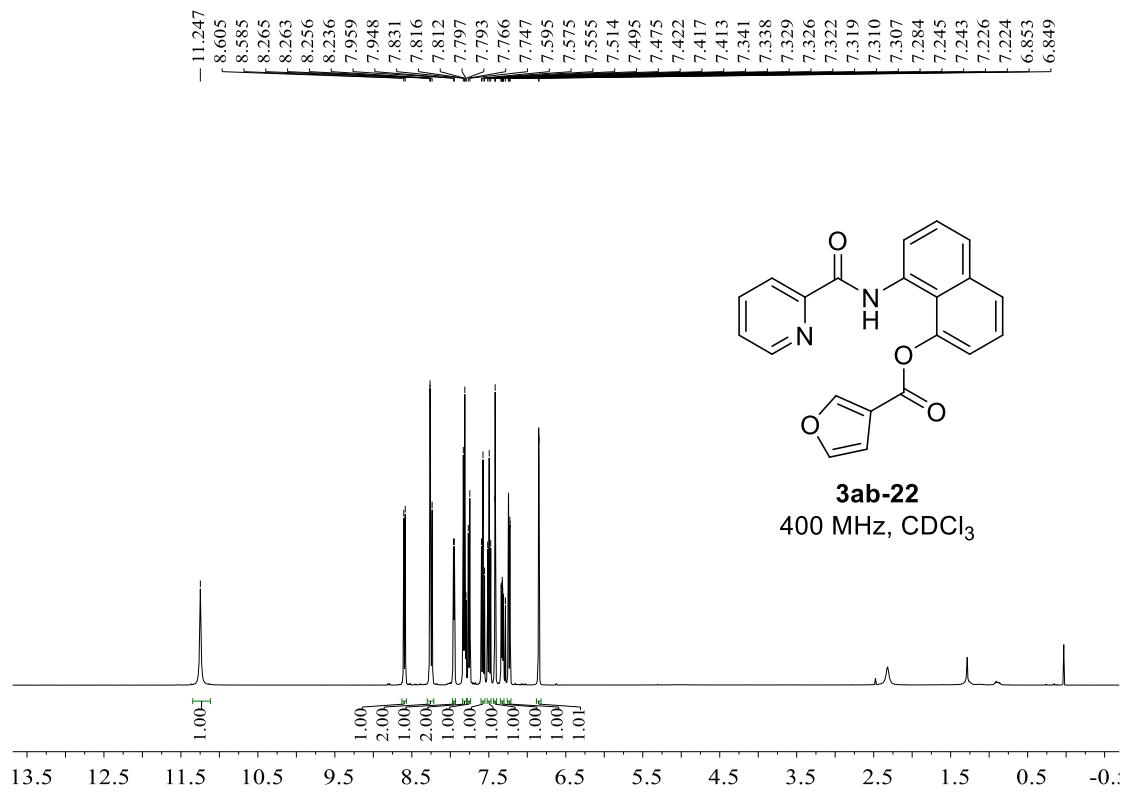


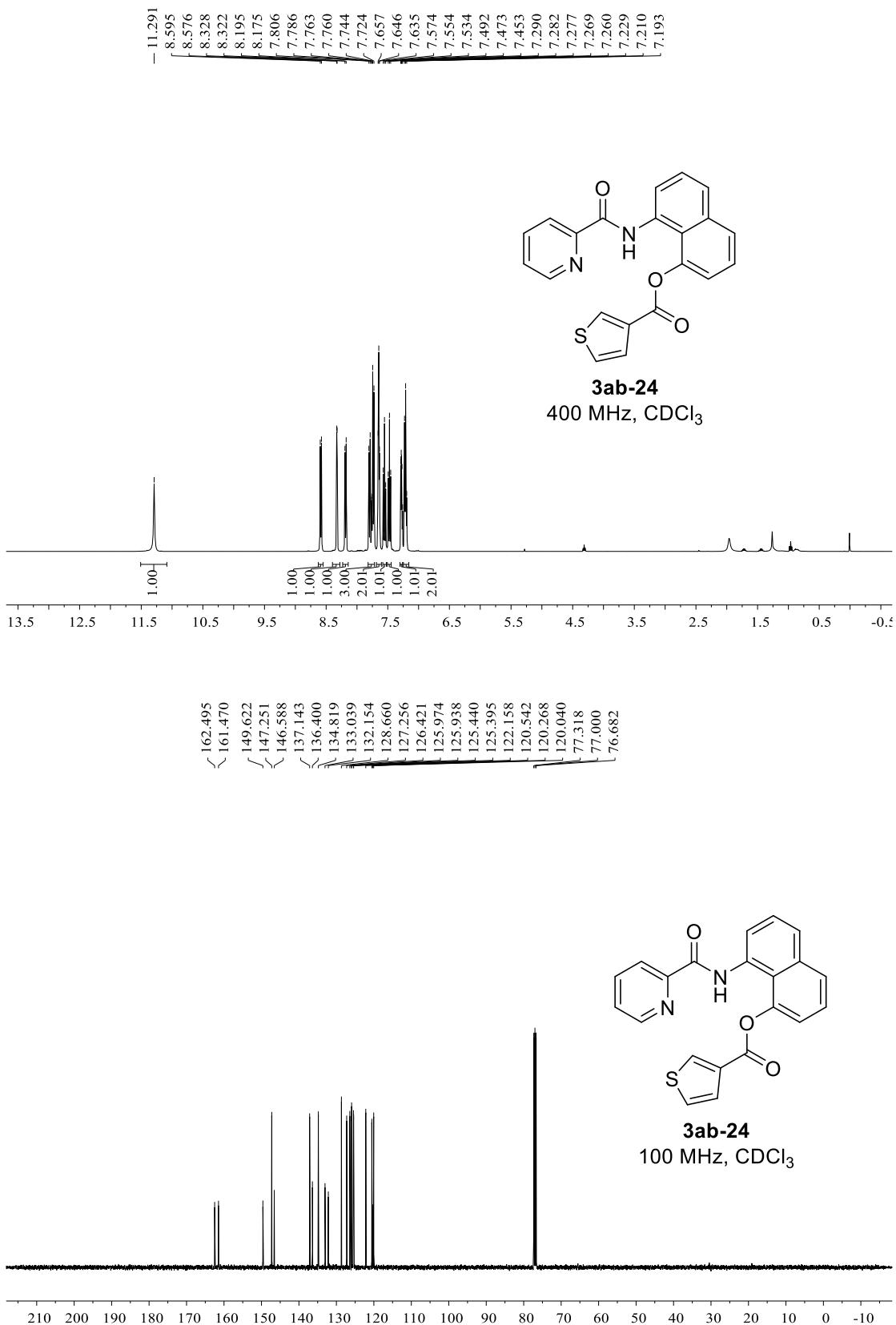
163.829
 < 162.377
 150.548
 149.460
 > 146.976
 < 146.139
 137.391
 < 136.493
 134.896
 131.583
 131.312
 127.905
 126.580
 126.289
 126.085
 125.530
 123.225
 122.560
 122.482
 120.942
 120.121
 77.317
 77.000
 76.682

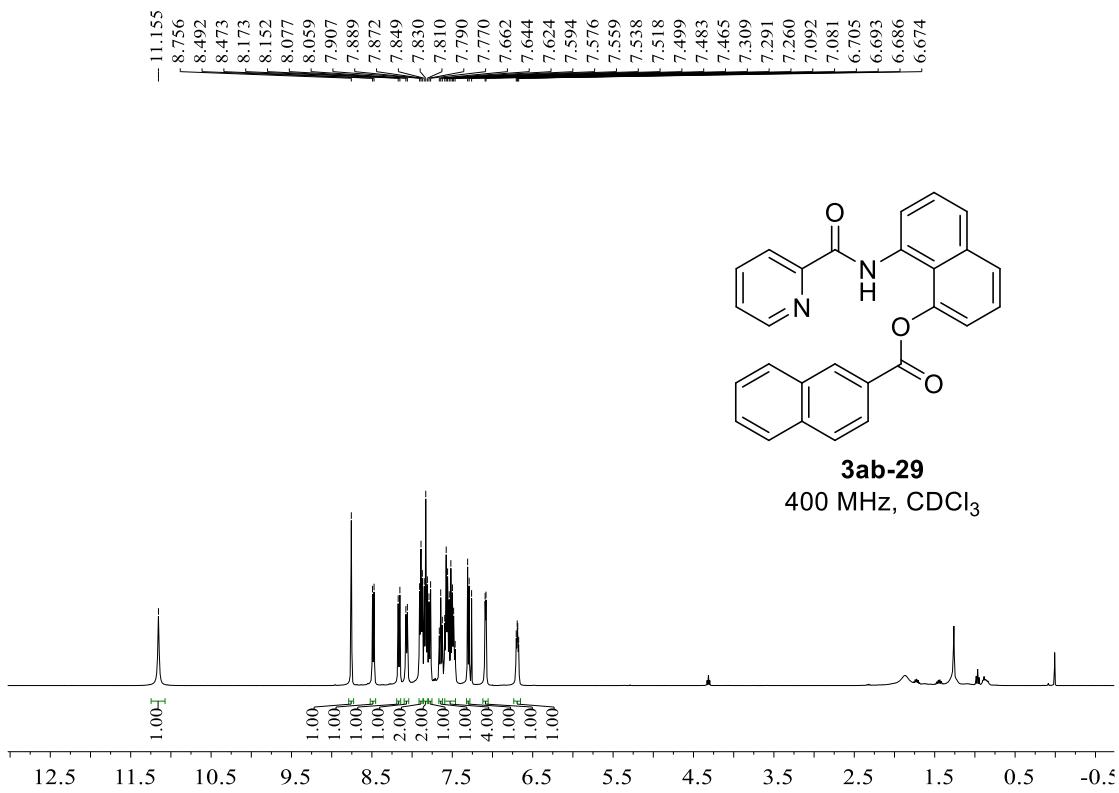




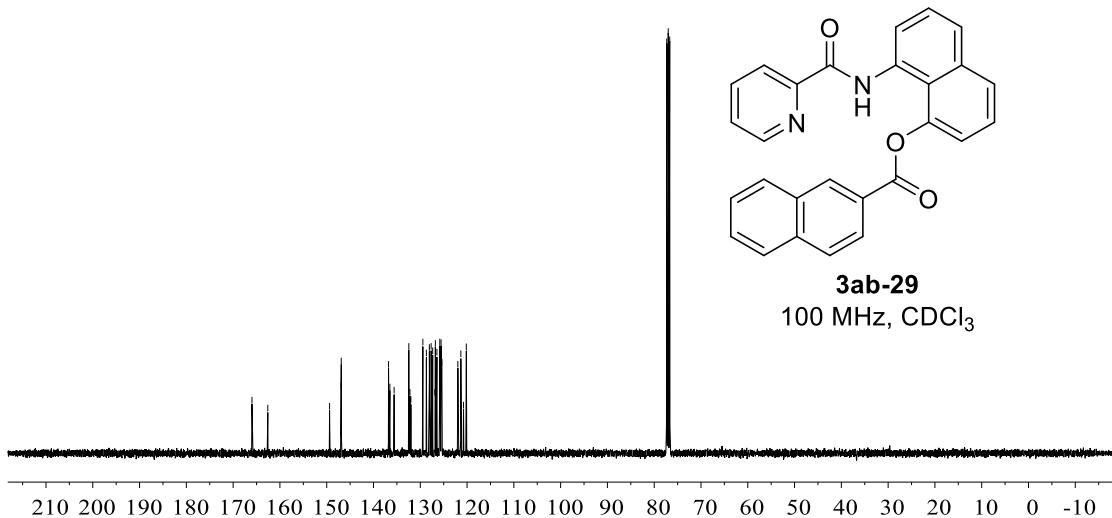


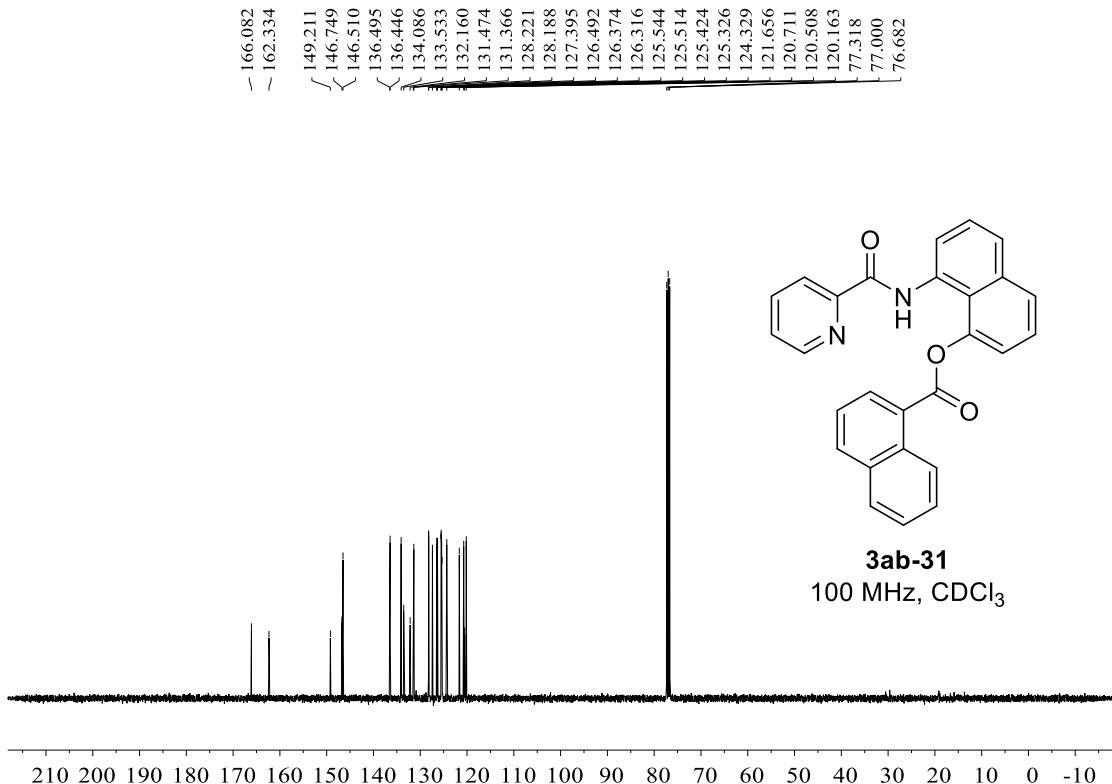
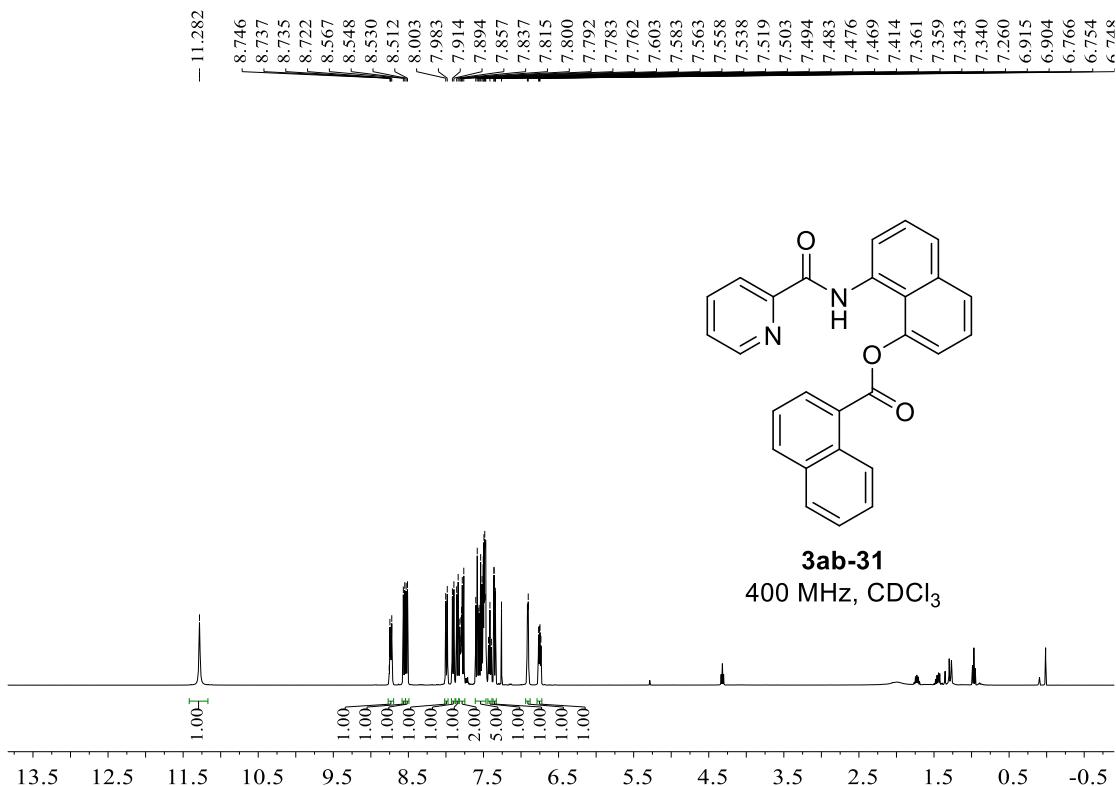


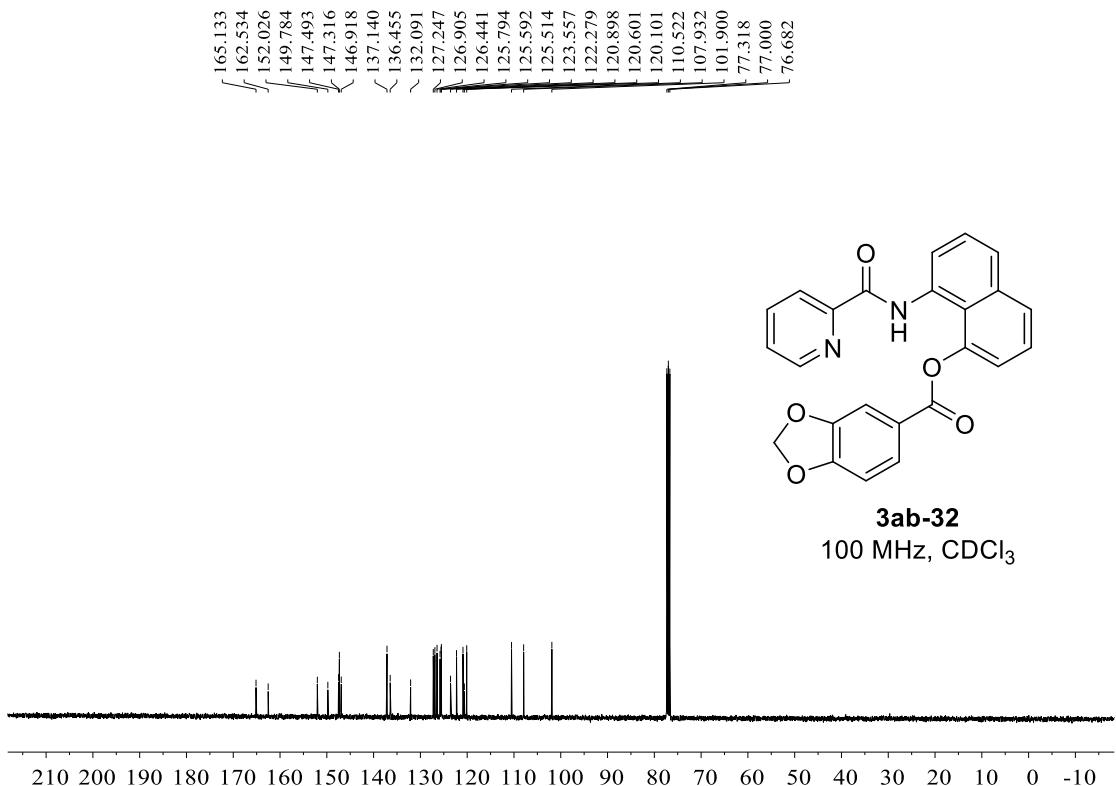
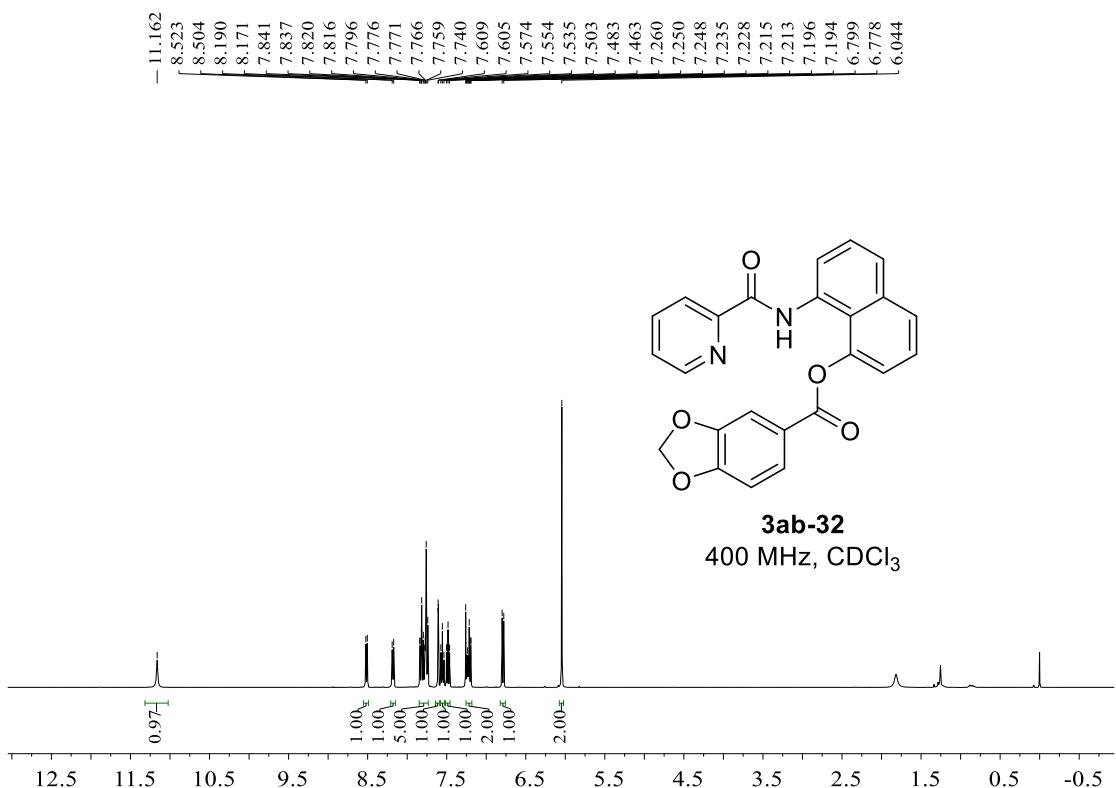


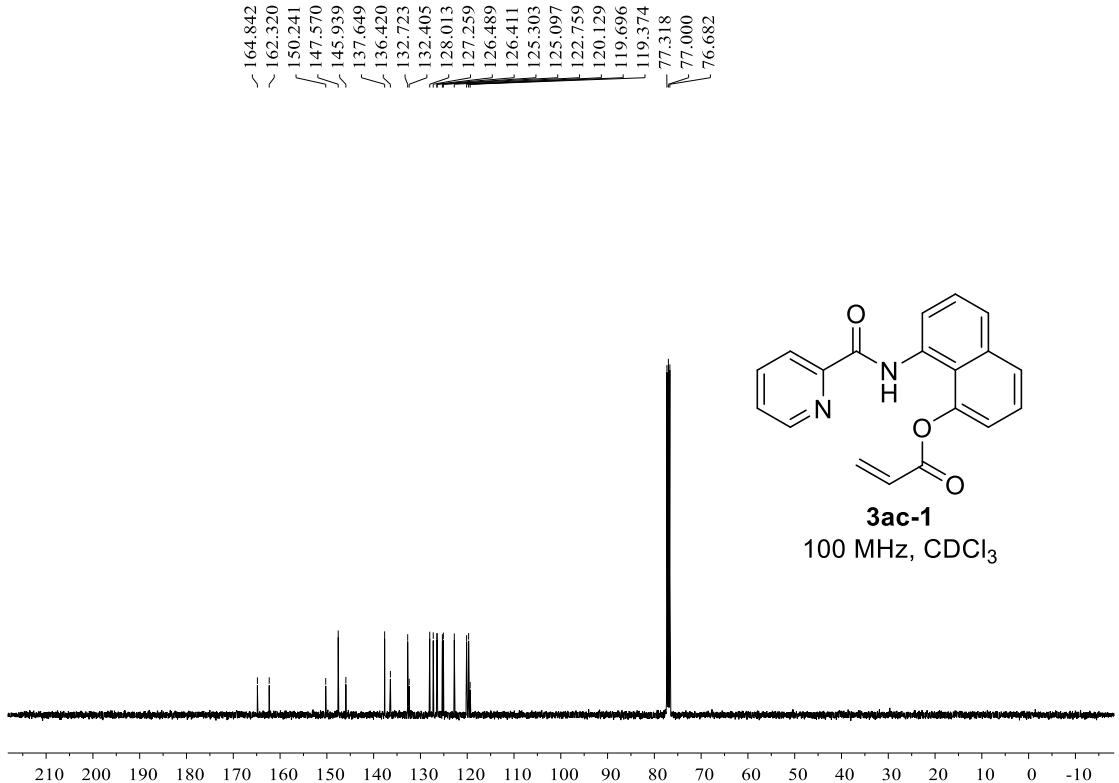
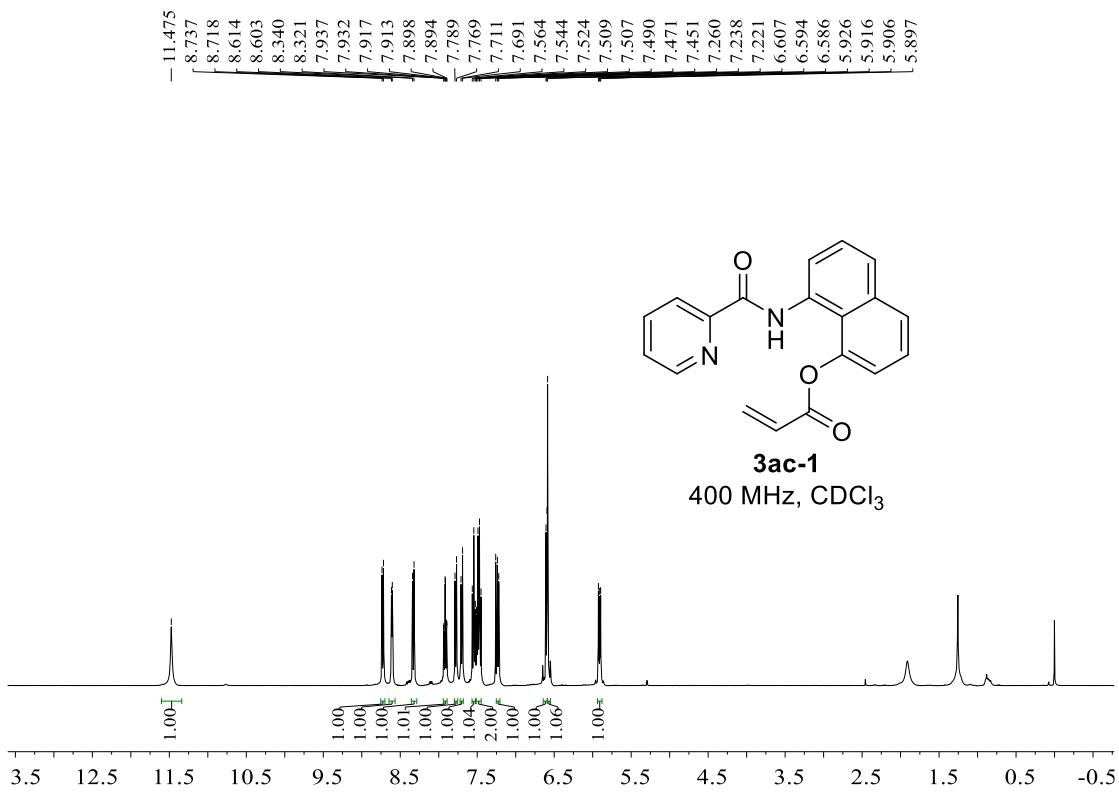


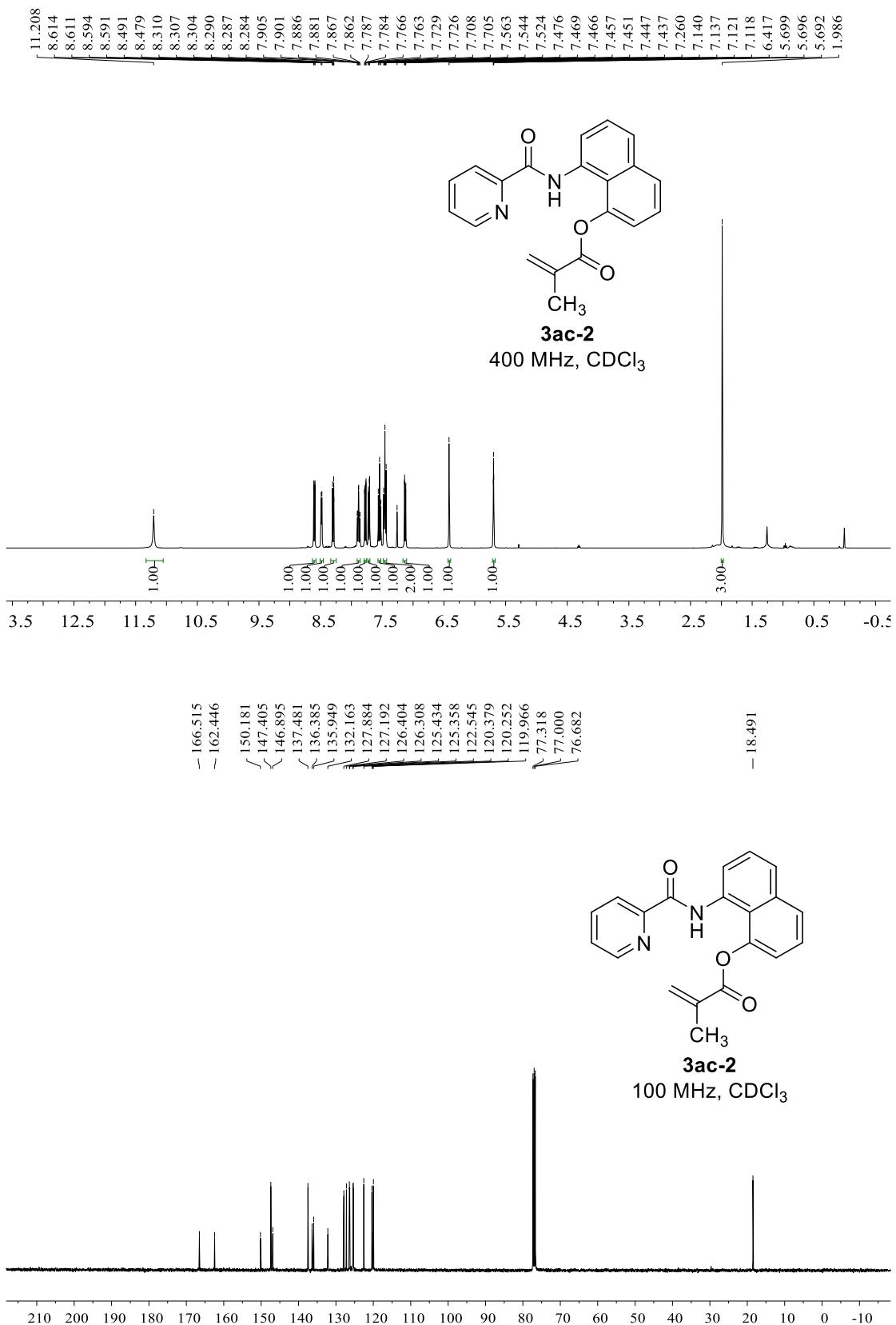
165.976
 < 146.922
 < 146.893
 136.781
 < 136.494
 < 135.590
 132.449
 132.209
 132.009
 129.449
 128.696
 128.056
 127.681
 127.348
 126.888
 126.776
 126.446
 125.823
 125.731
 125.551
 125.383
 121.965
 121.328
 120.755
 120.160
 77.318
 77.000
 76.682

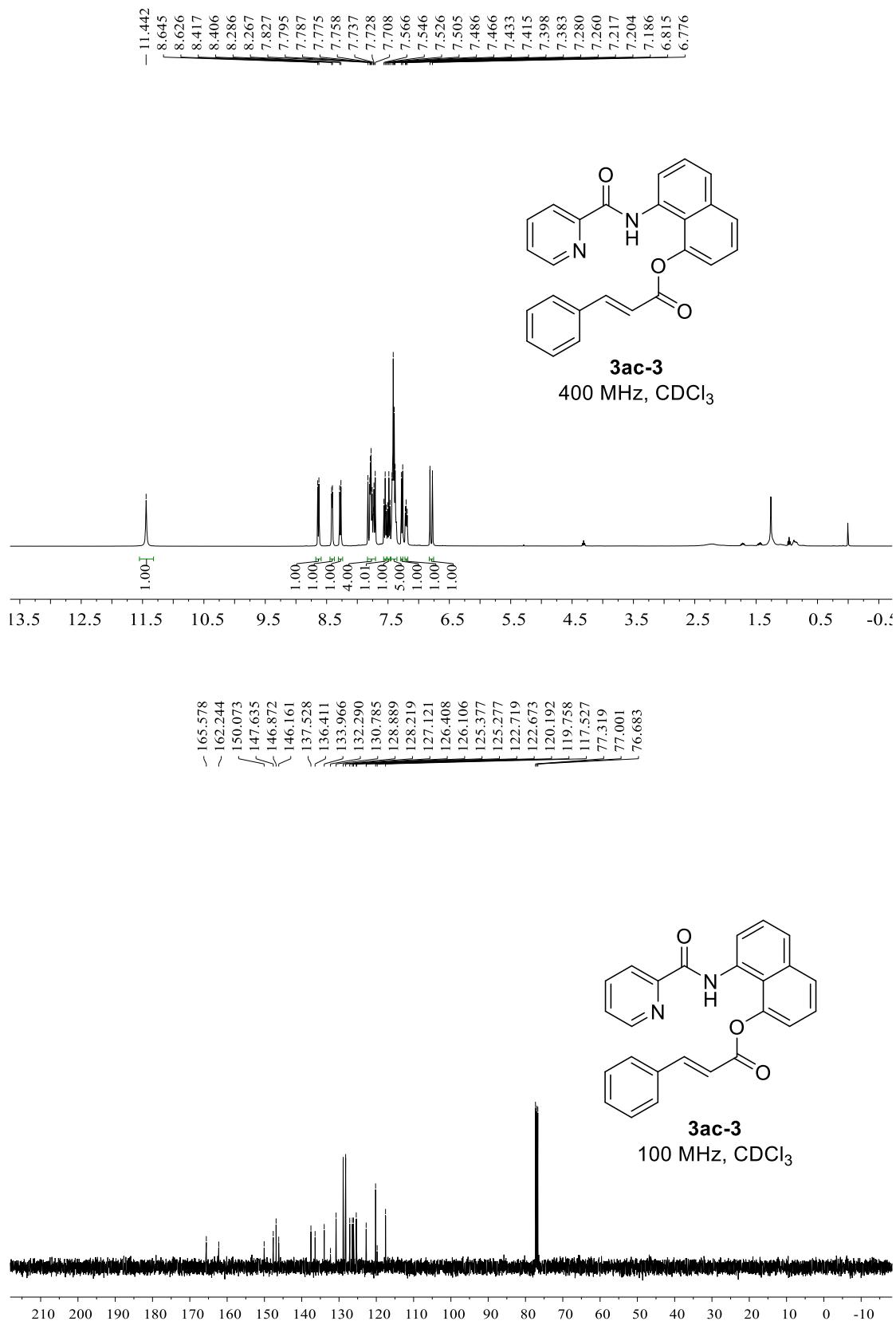


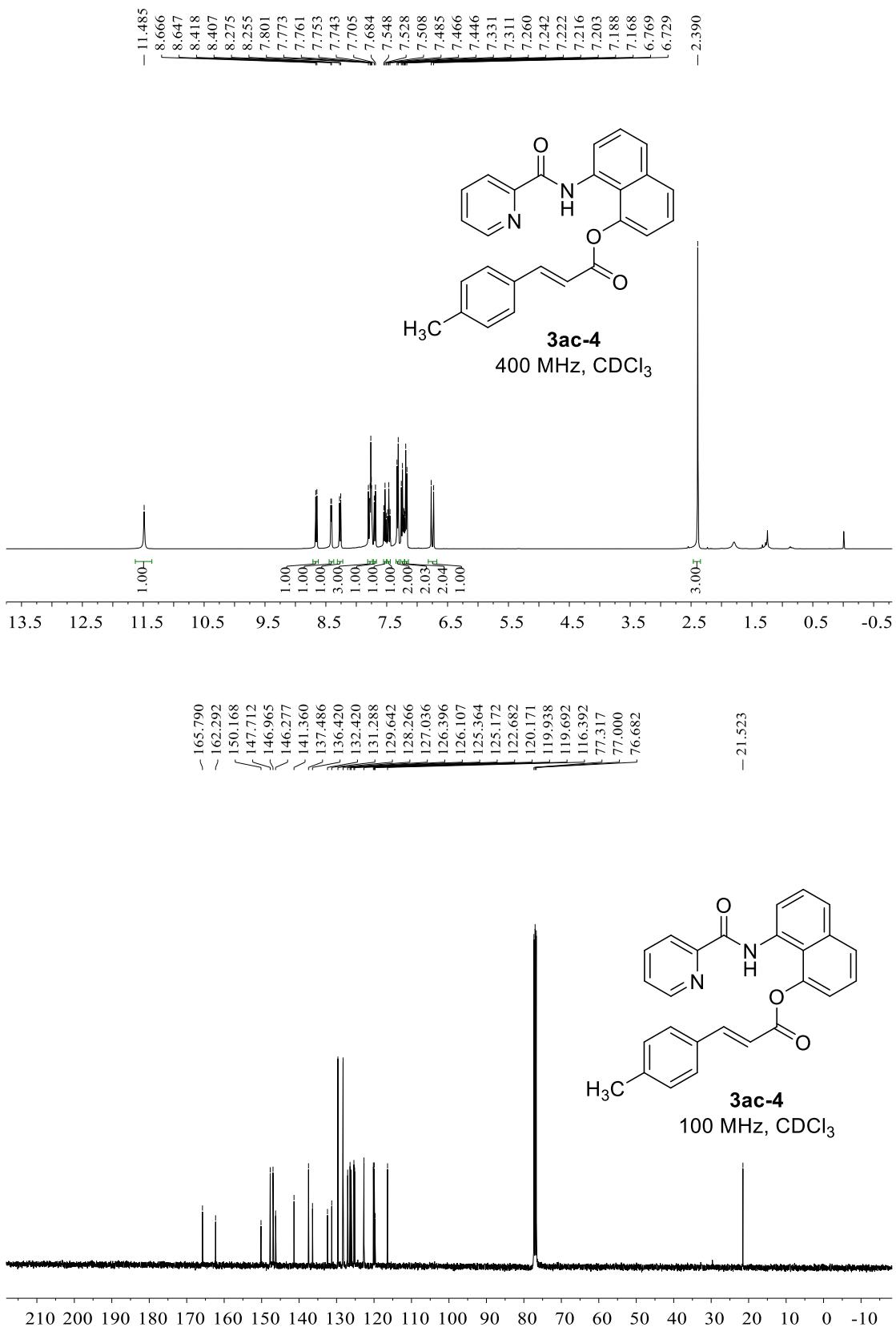


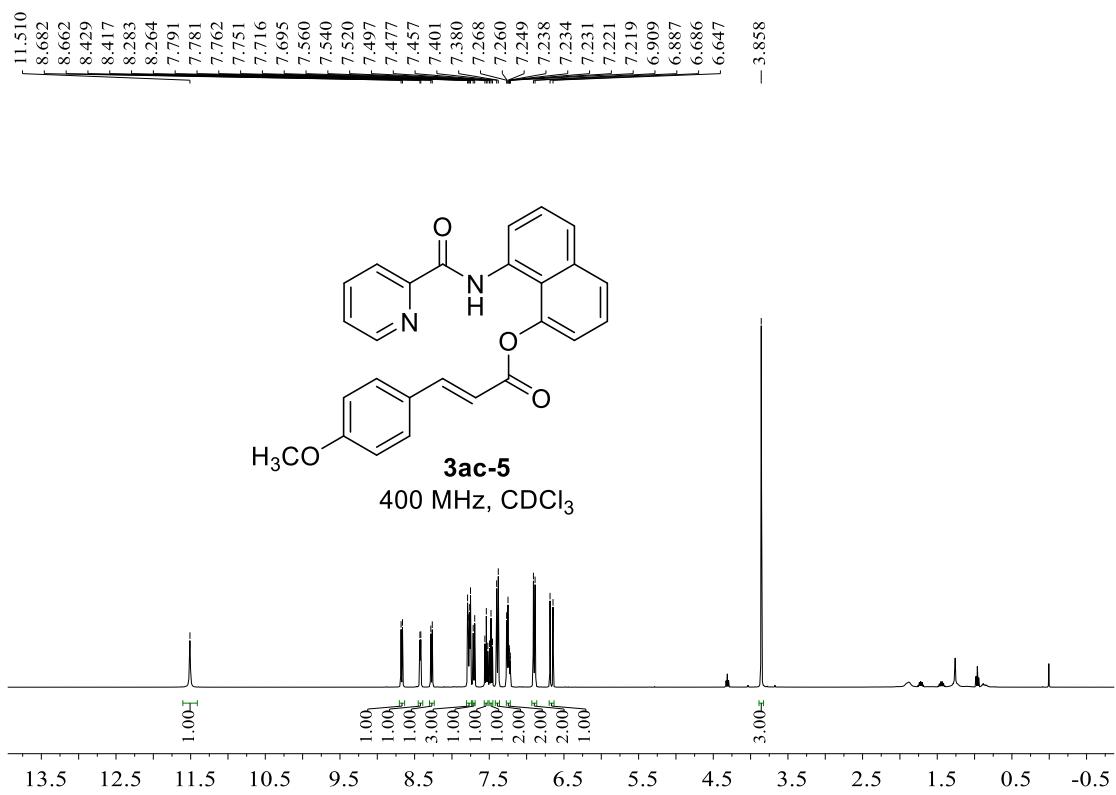




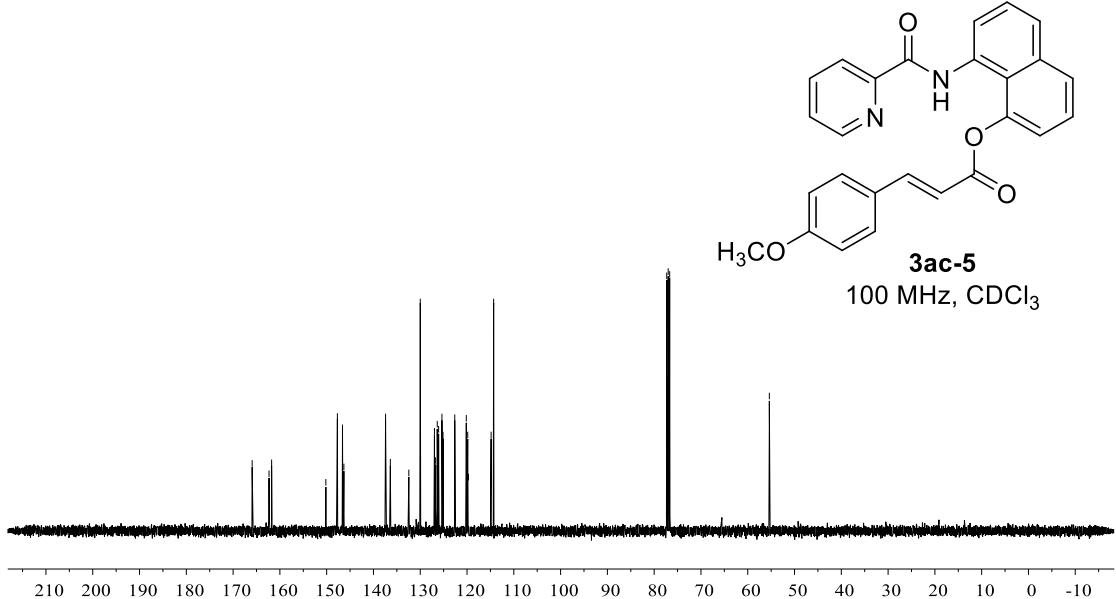


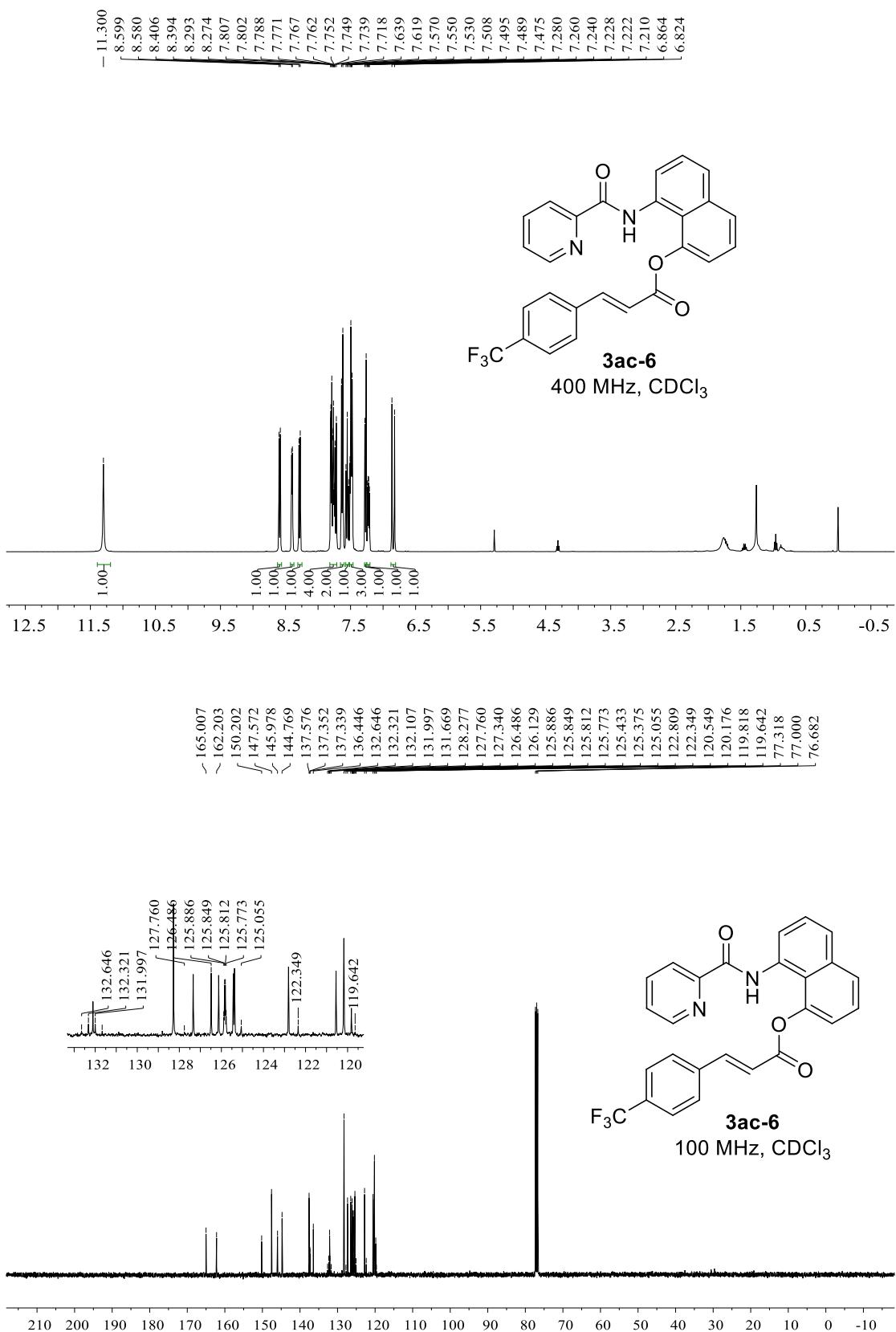




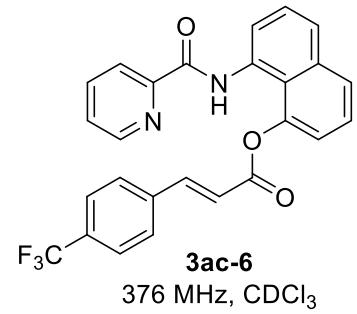


165.928
 162.321
 161.763
 150.175
 147.712
 146.616
 146.321
 137.414
 136.391
 132.440
 129.994
 126.963
 126.711
 126.362
 126.072
 125.361
 125.136
 122.621
 120.157
 119.852
 119.681
 114.852
 114.311
 77.318
 77.000
 76.683
 55.384



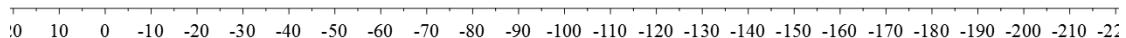


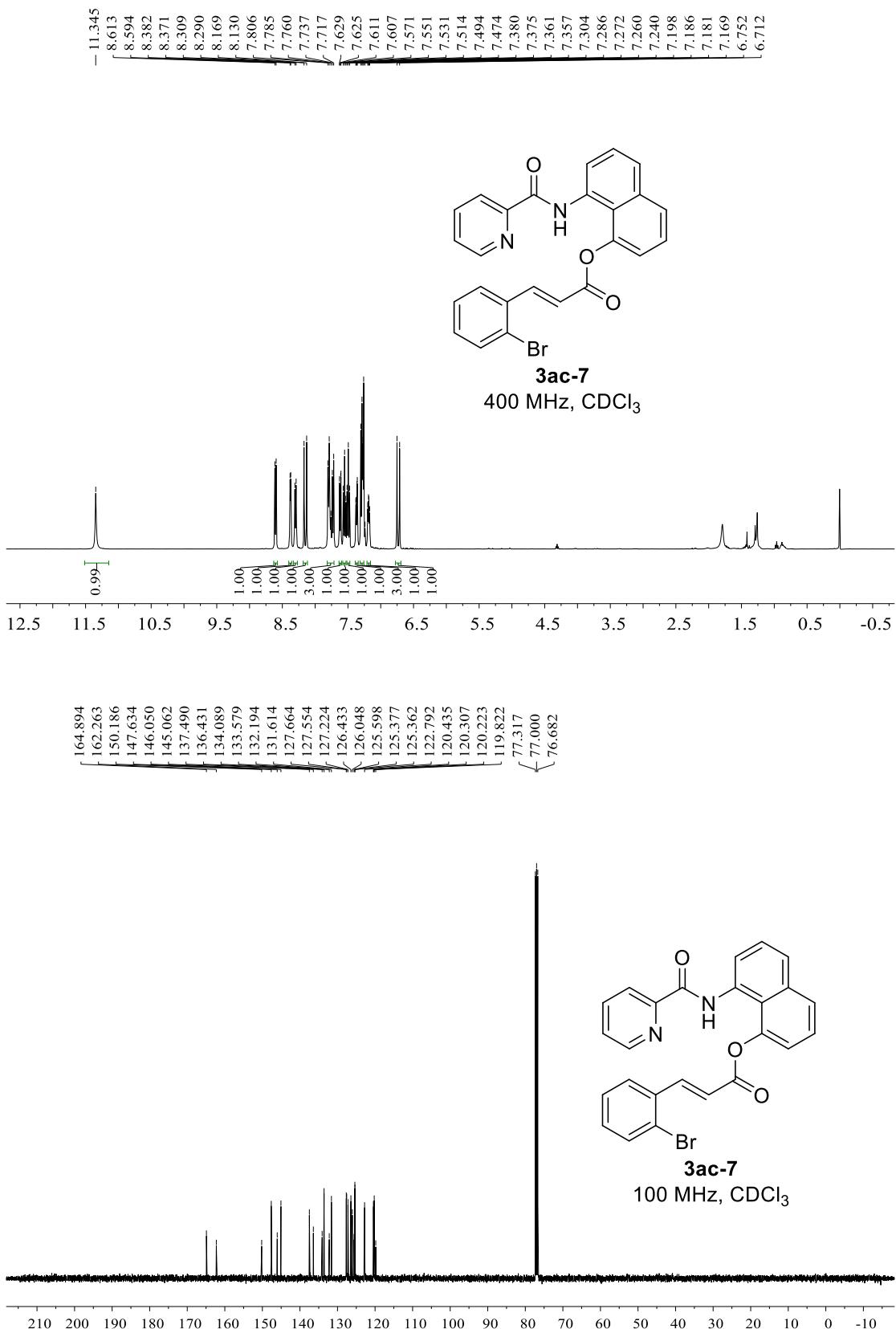
-62.853

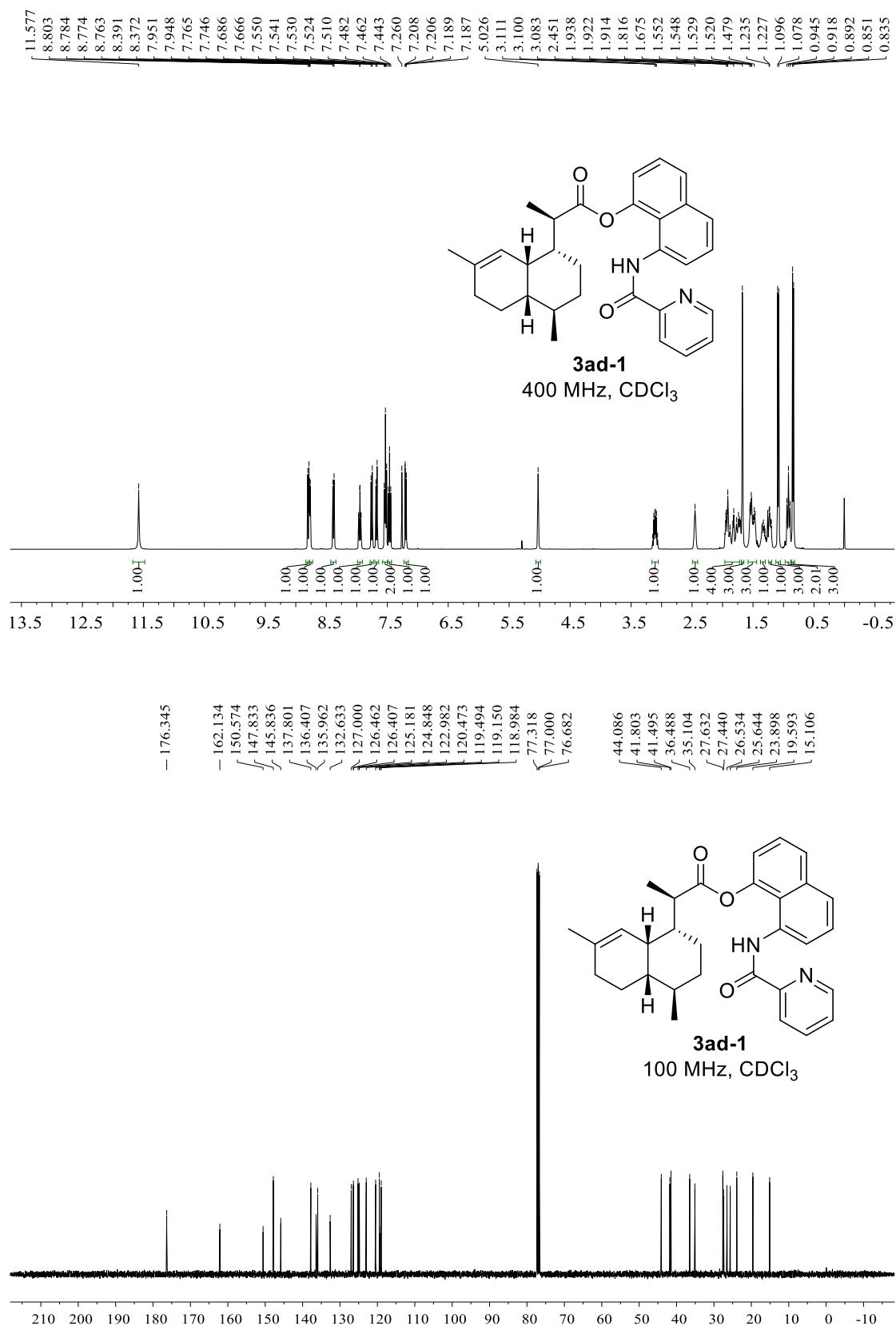


3ac-6

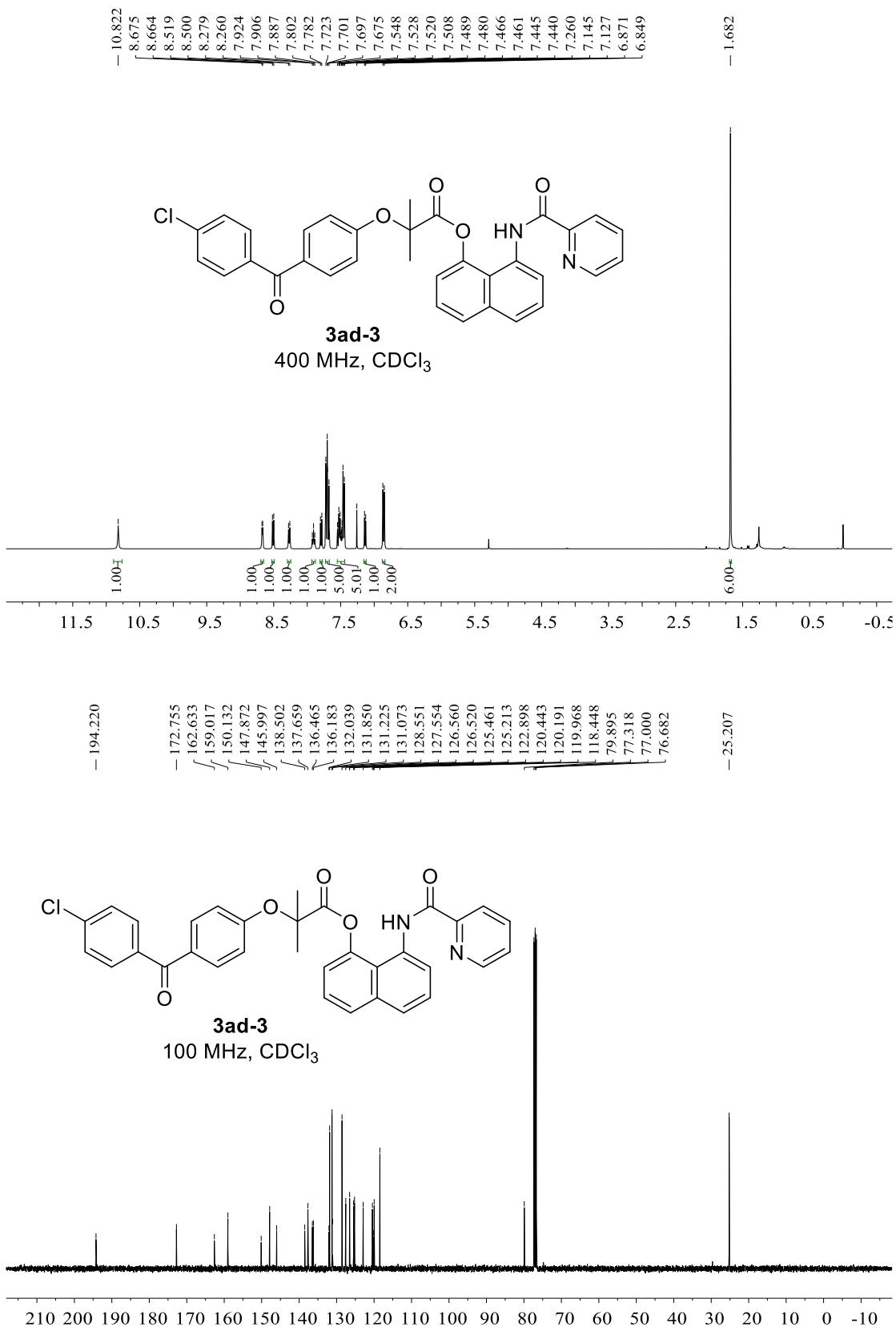
376 MHz, CDCl₃

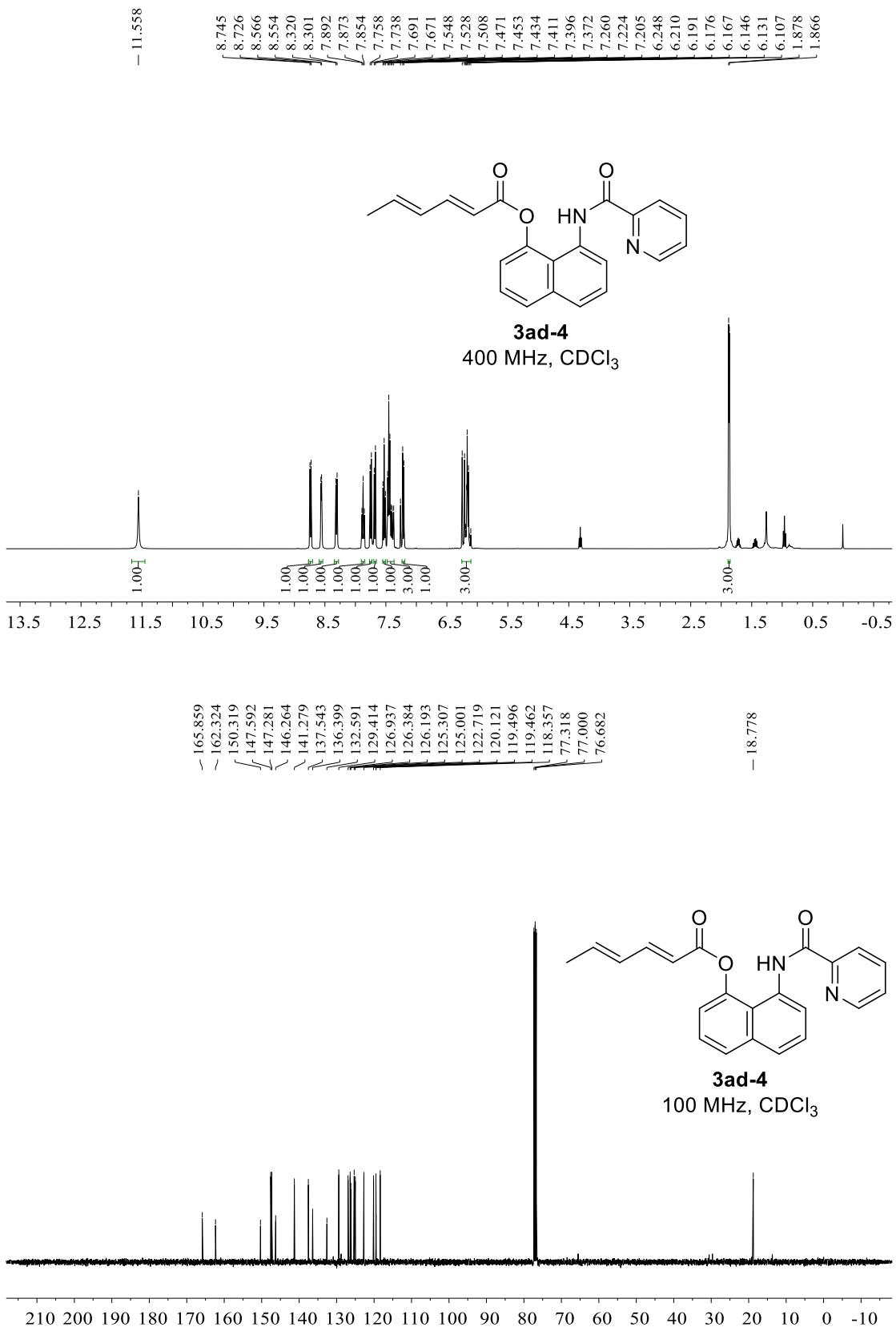


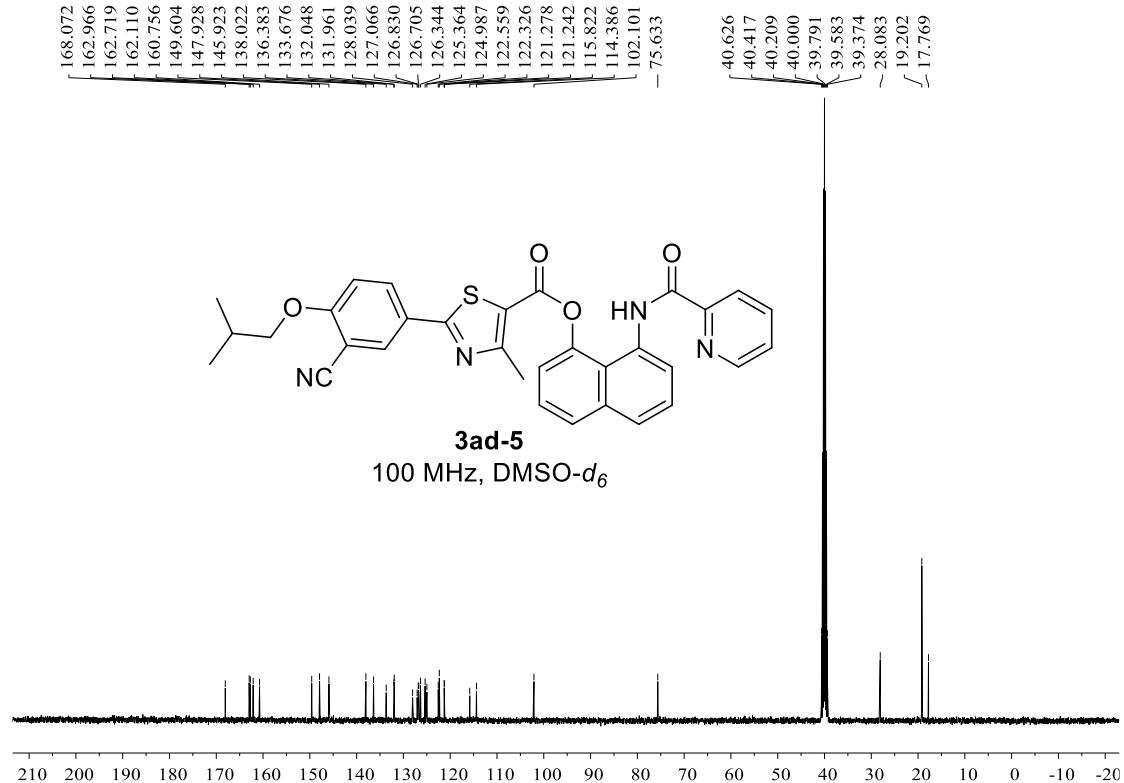
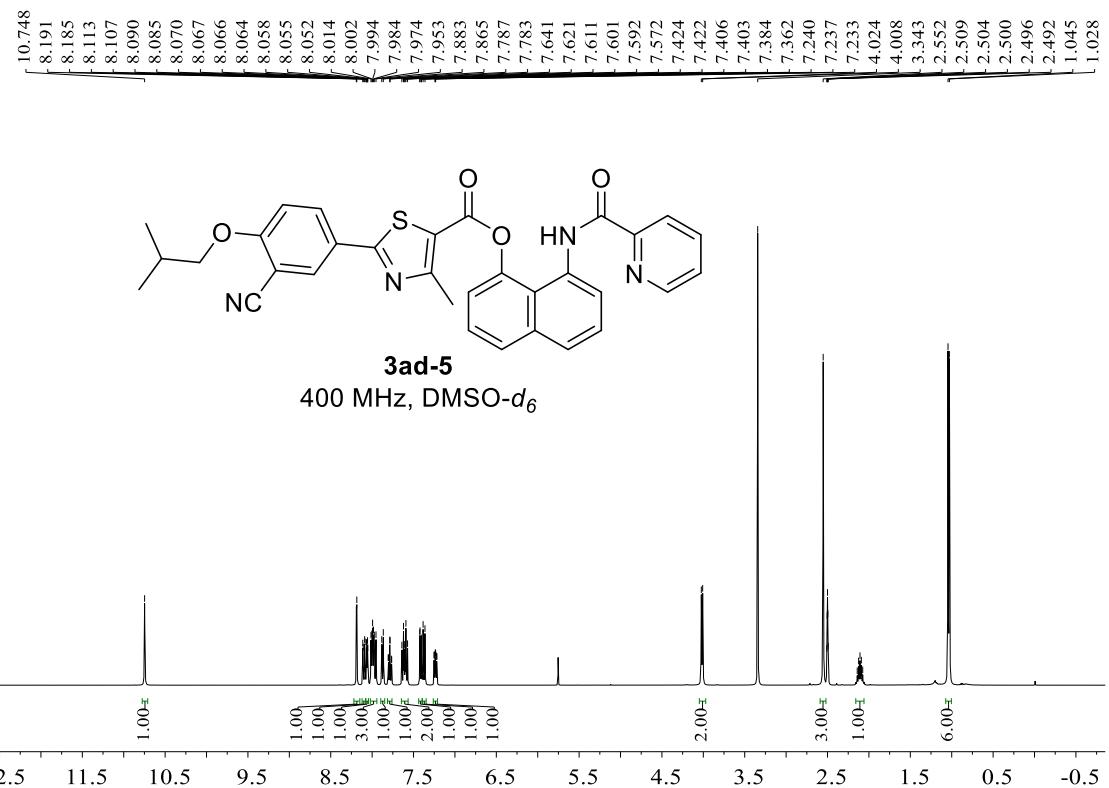


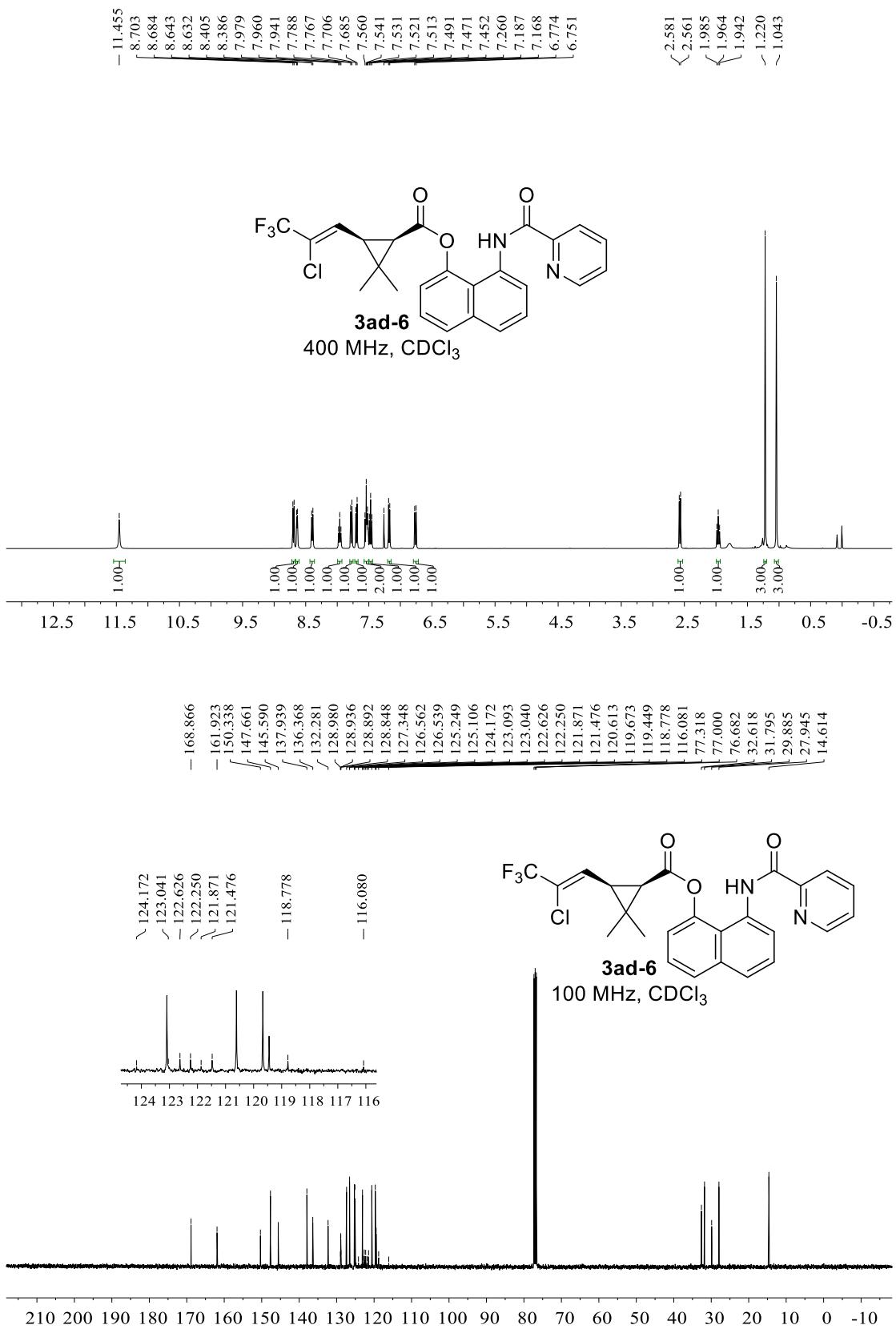


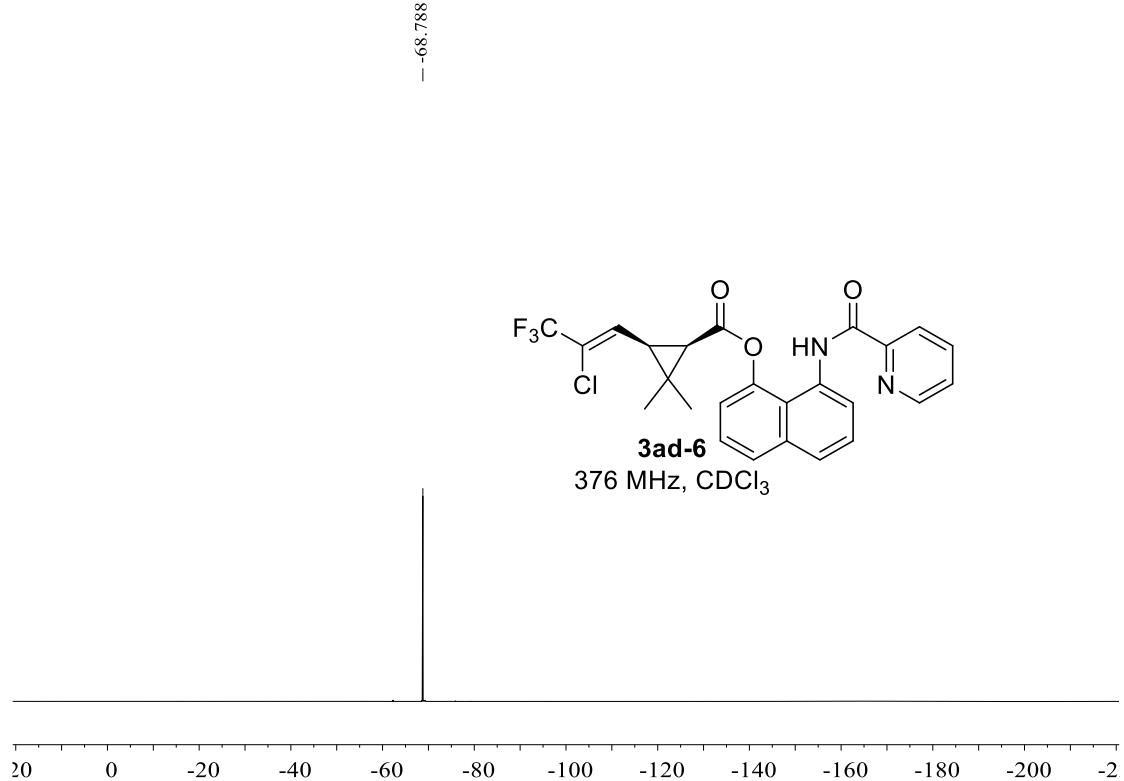


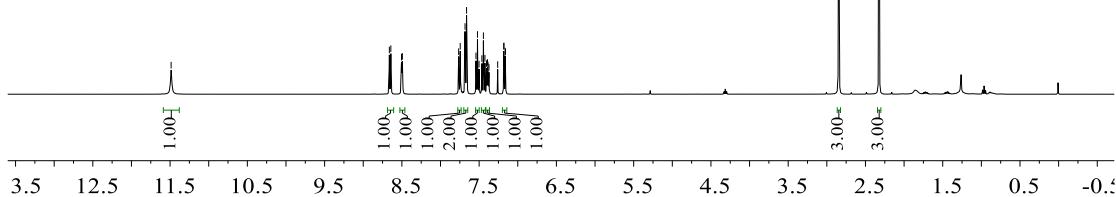
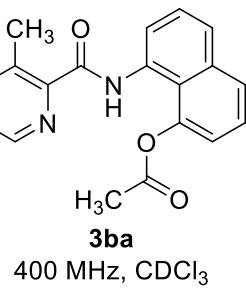






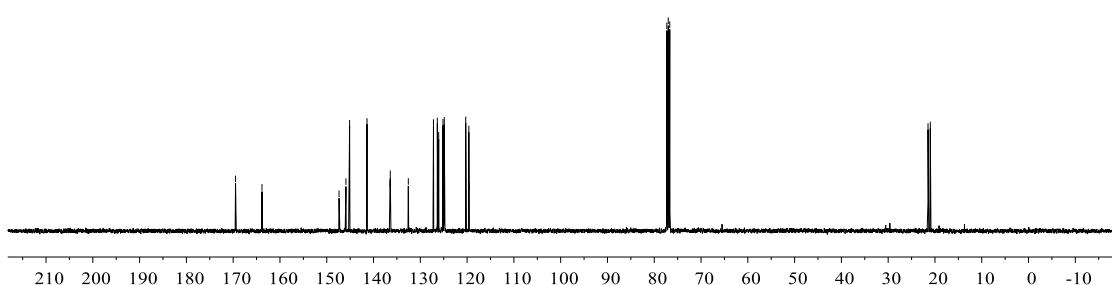
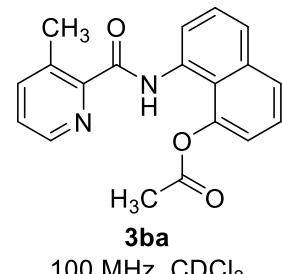


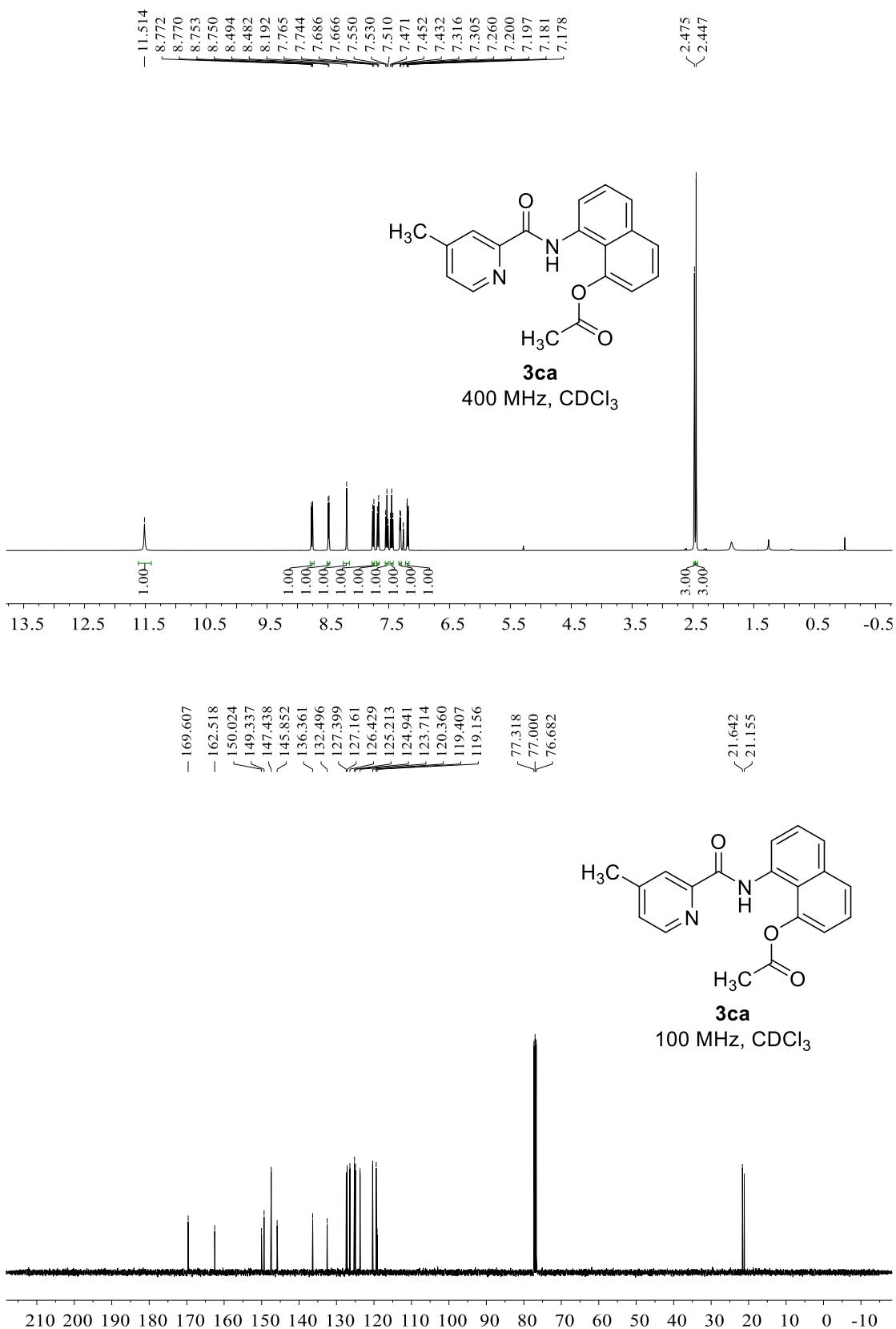


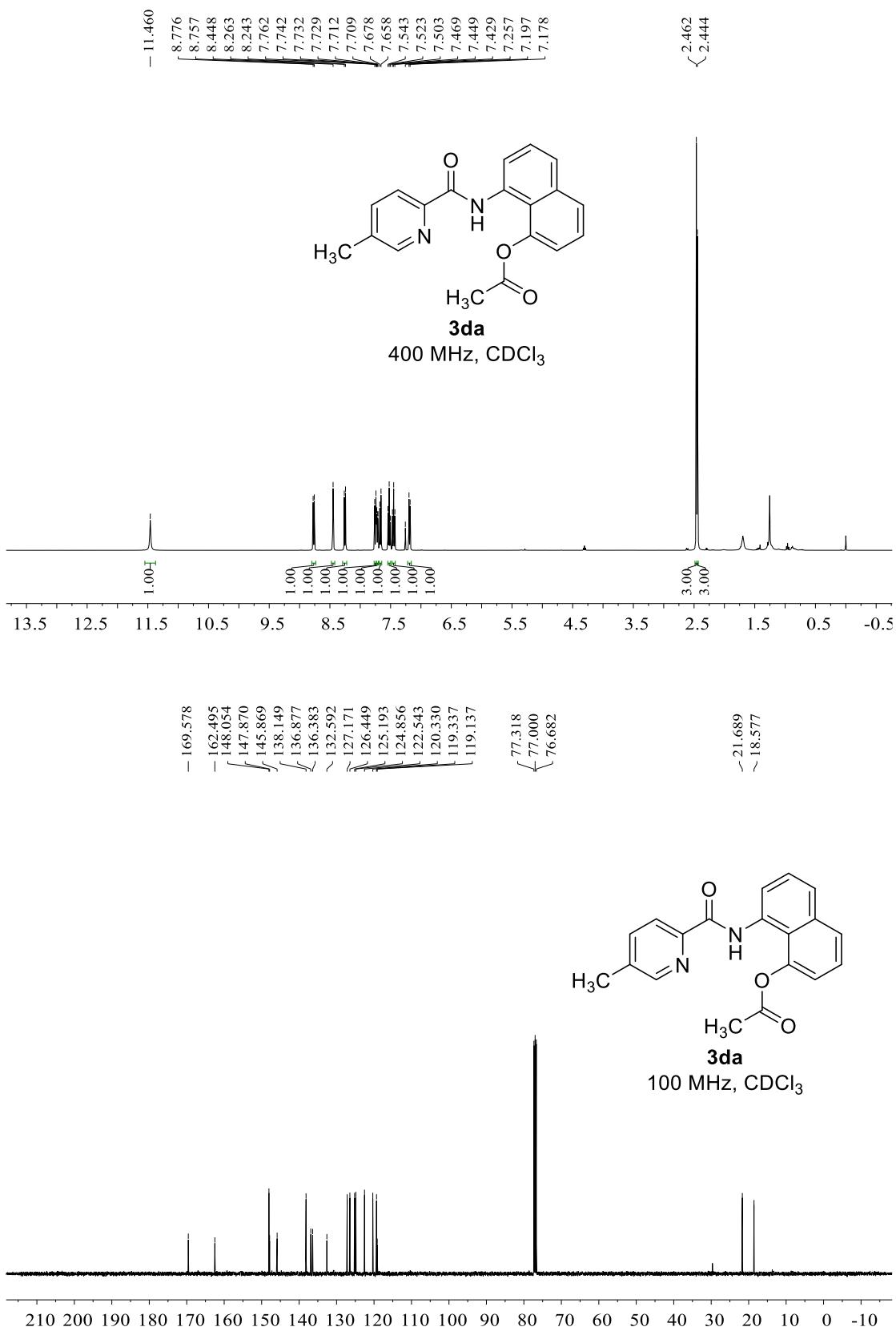


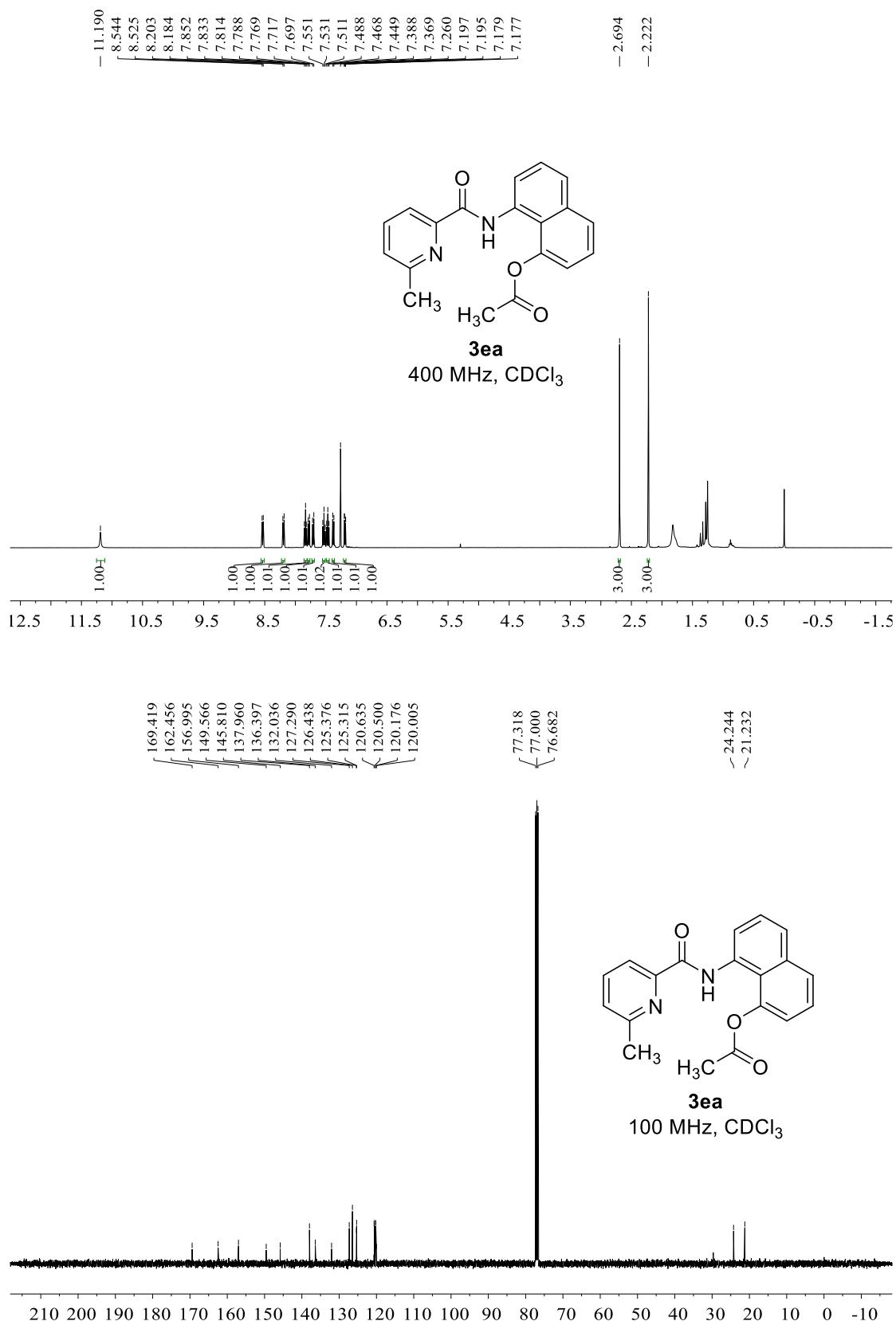
— 169.496
 — 163.843
 — 147.368
 — 145.898
 — 145.112
 — 141.398
 — 136.478
 — 136.416
 > 132.566
 — 127.188
 — 126.362
 — 126.071
 — 125.155
 — 124.882
 — 120.292
 — 119.626
 — 119.572

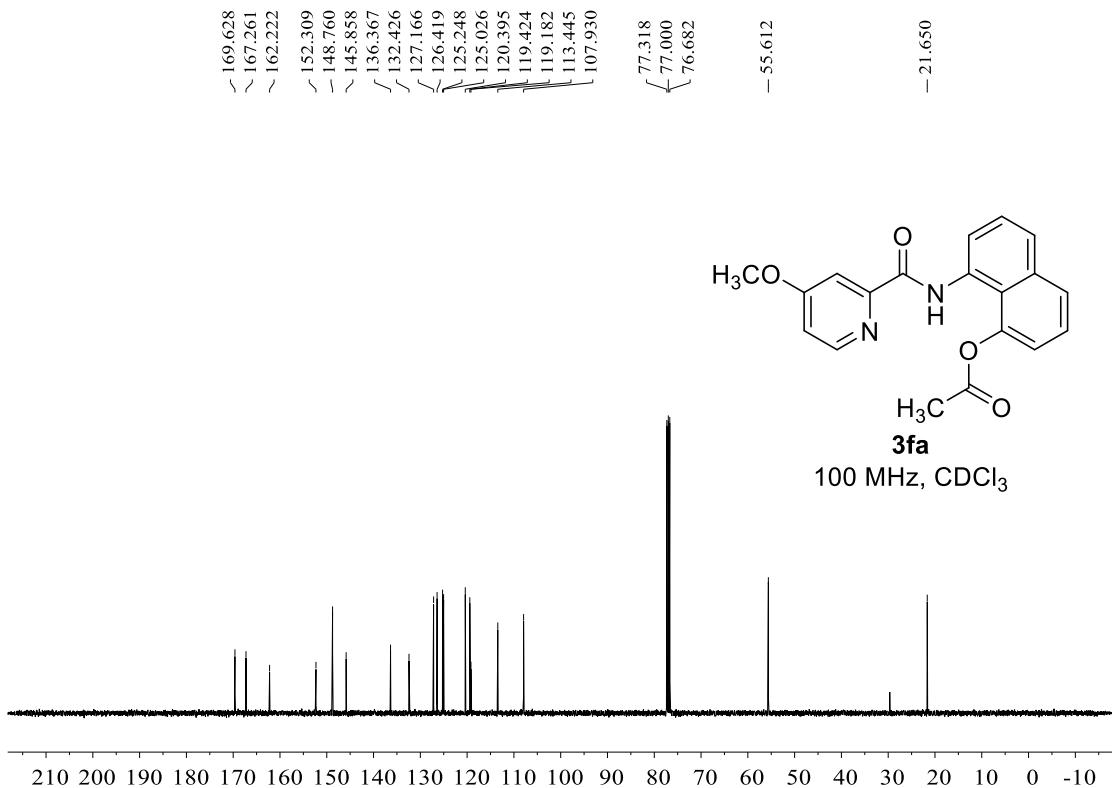
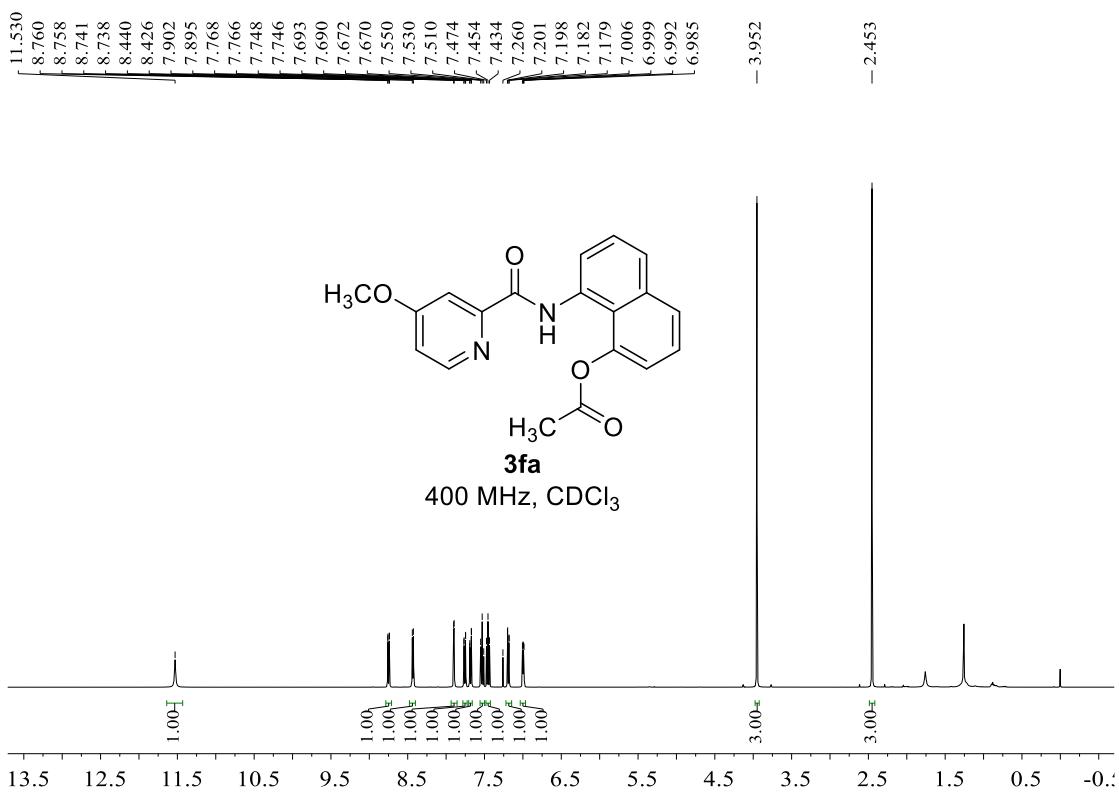
 — 77.318
 — 77.000
 — 76.682

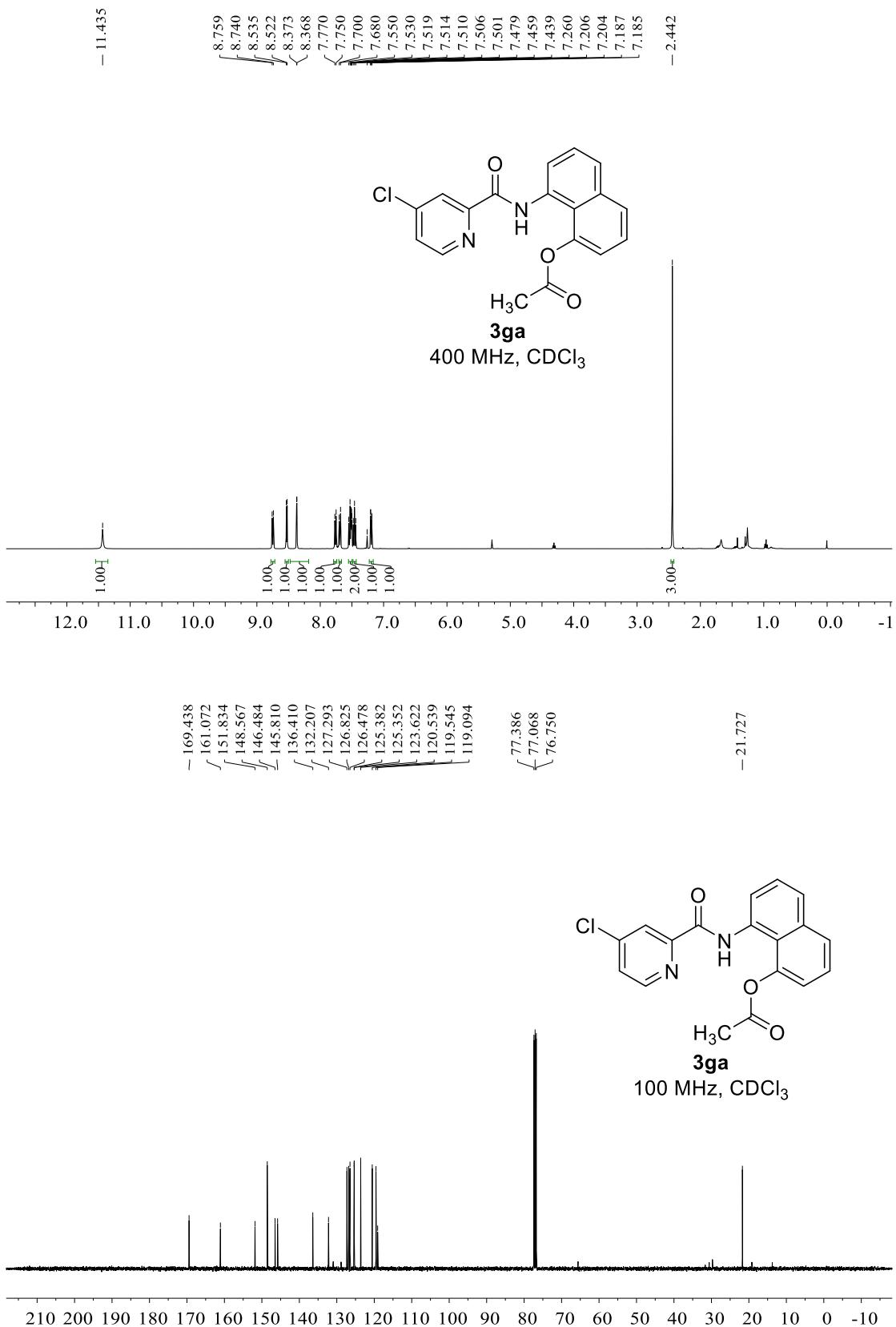


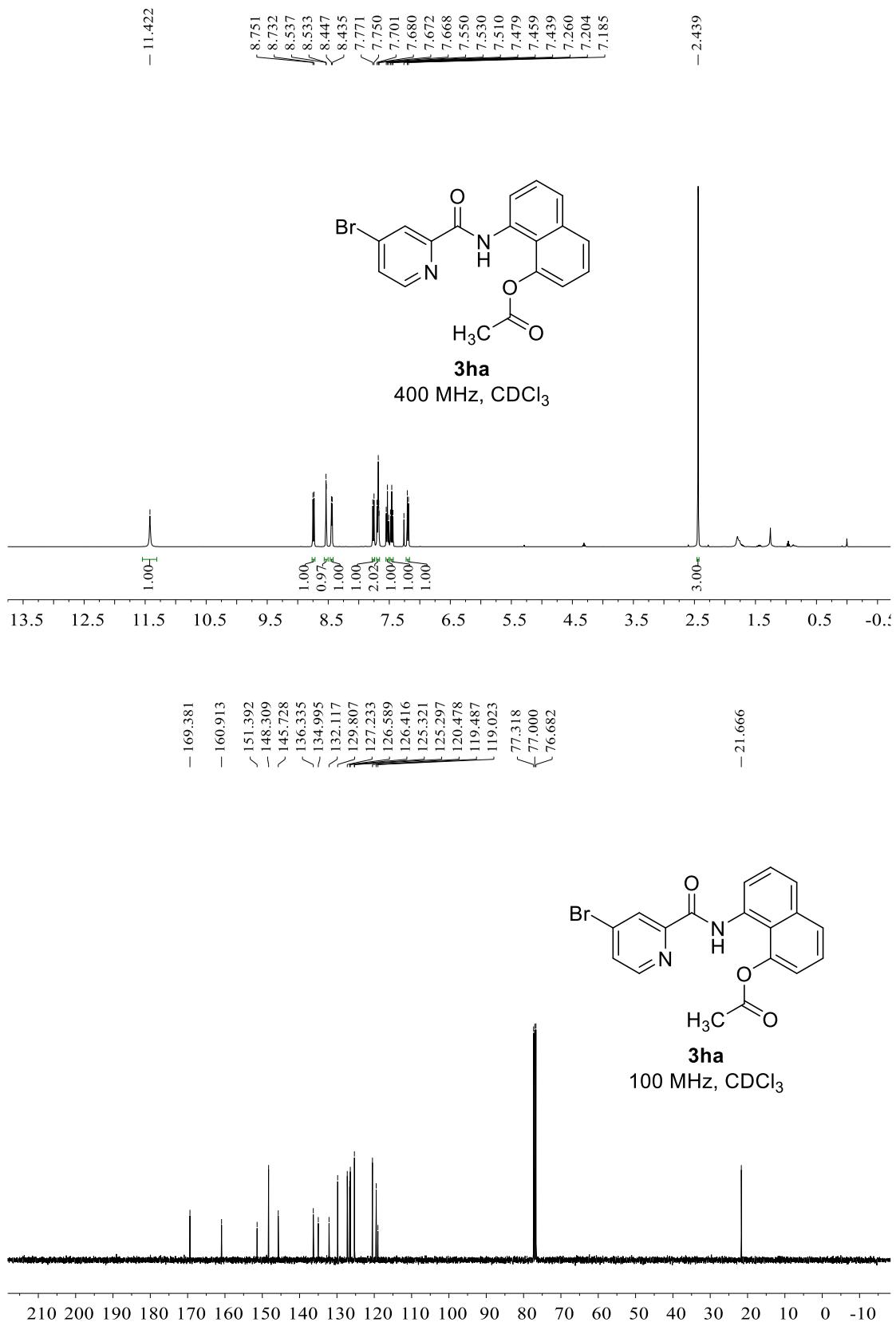


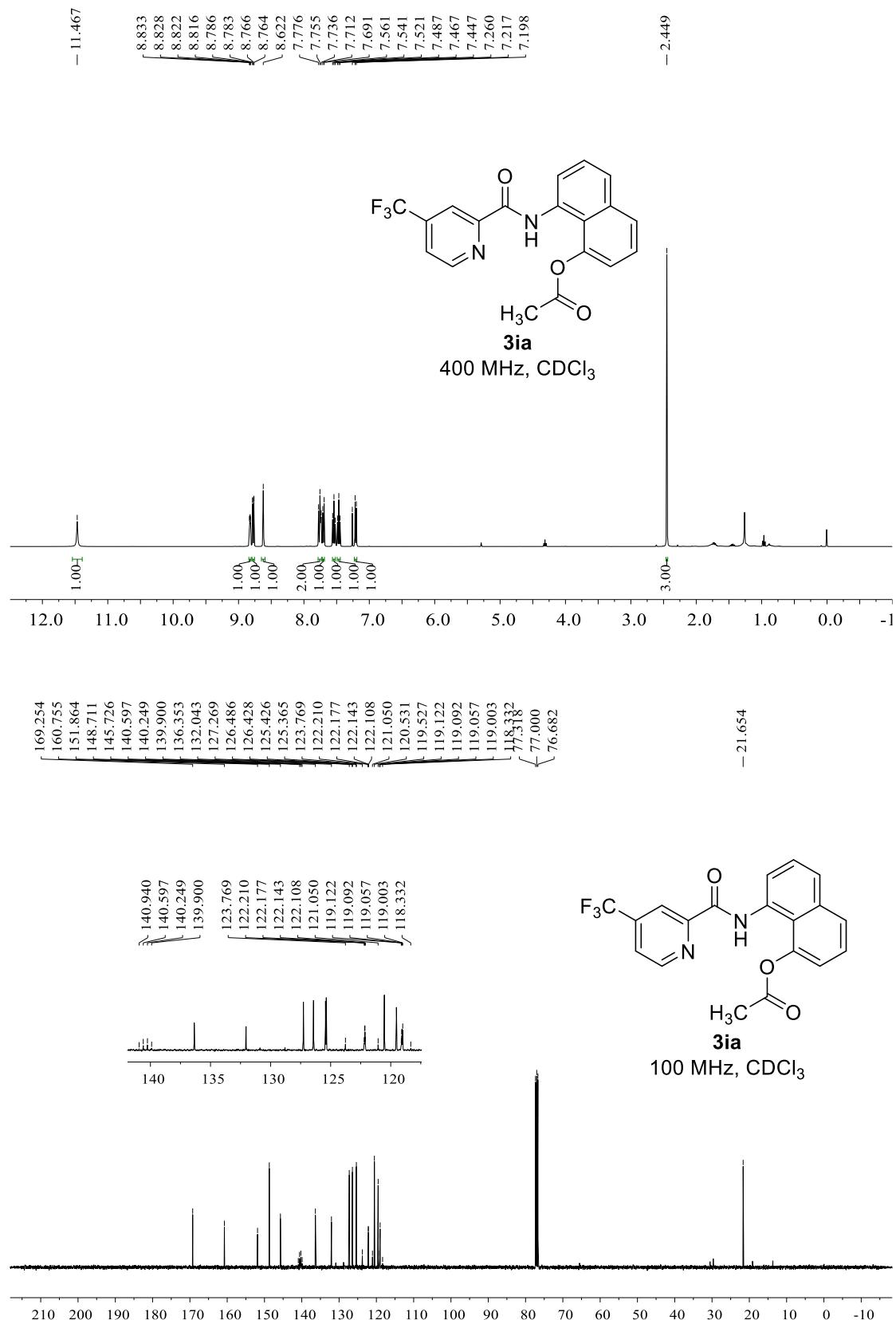




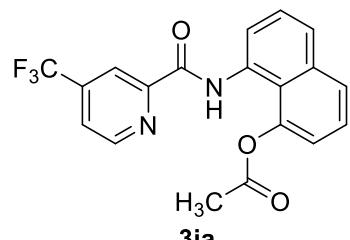




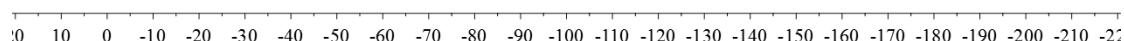


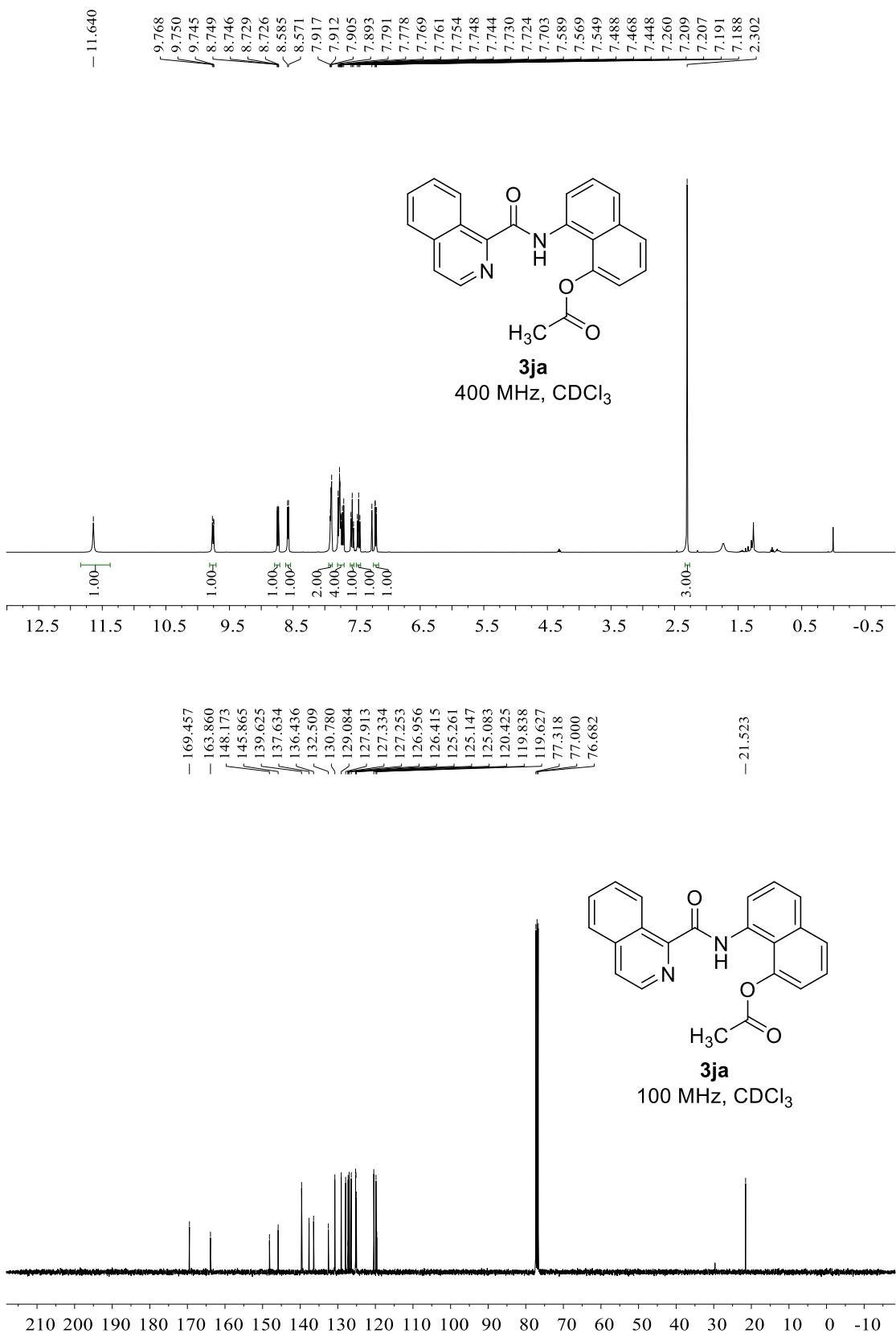


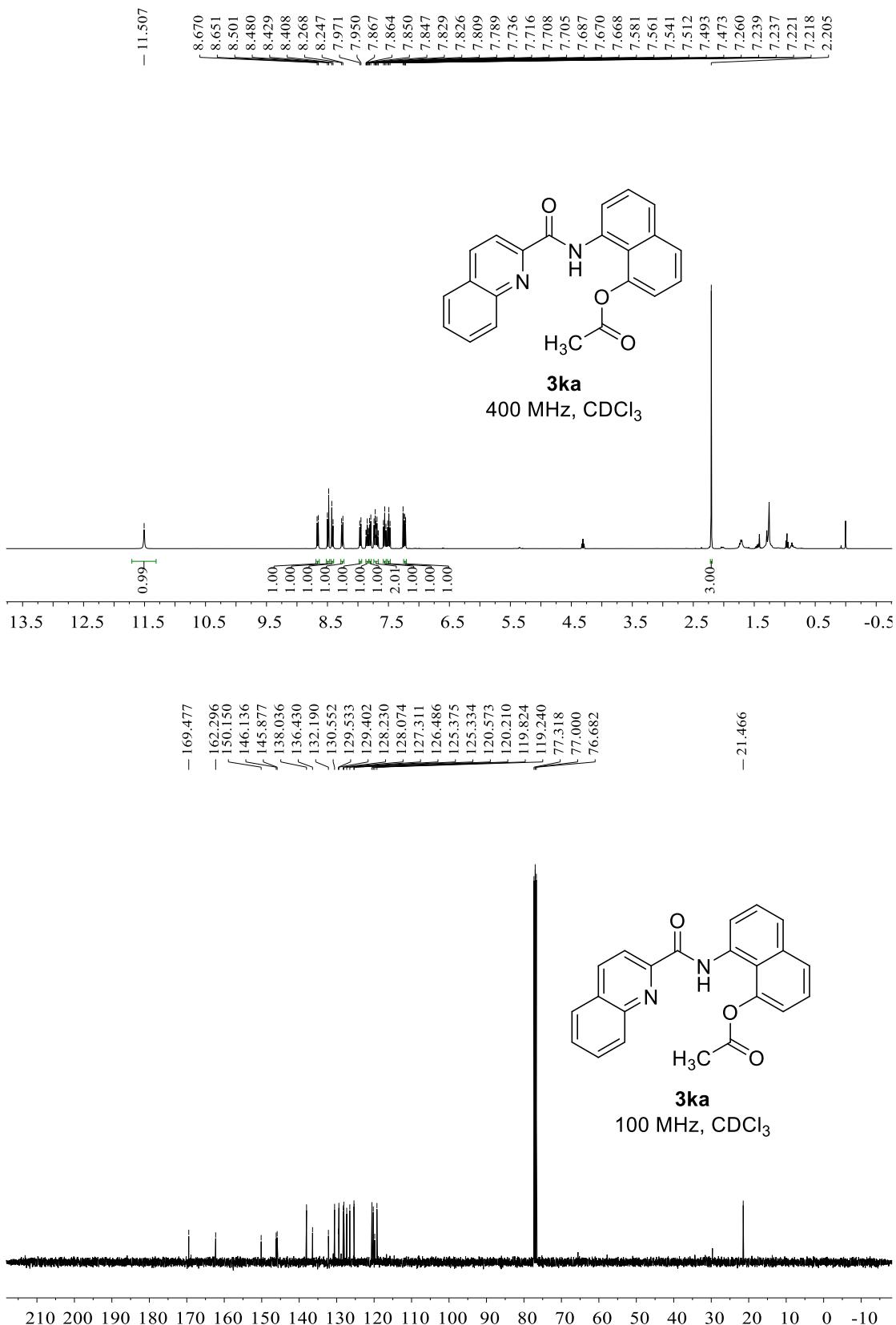
-64.785

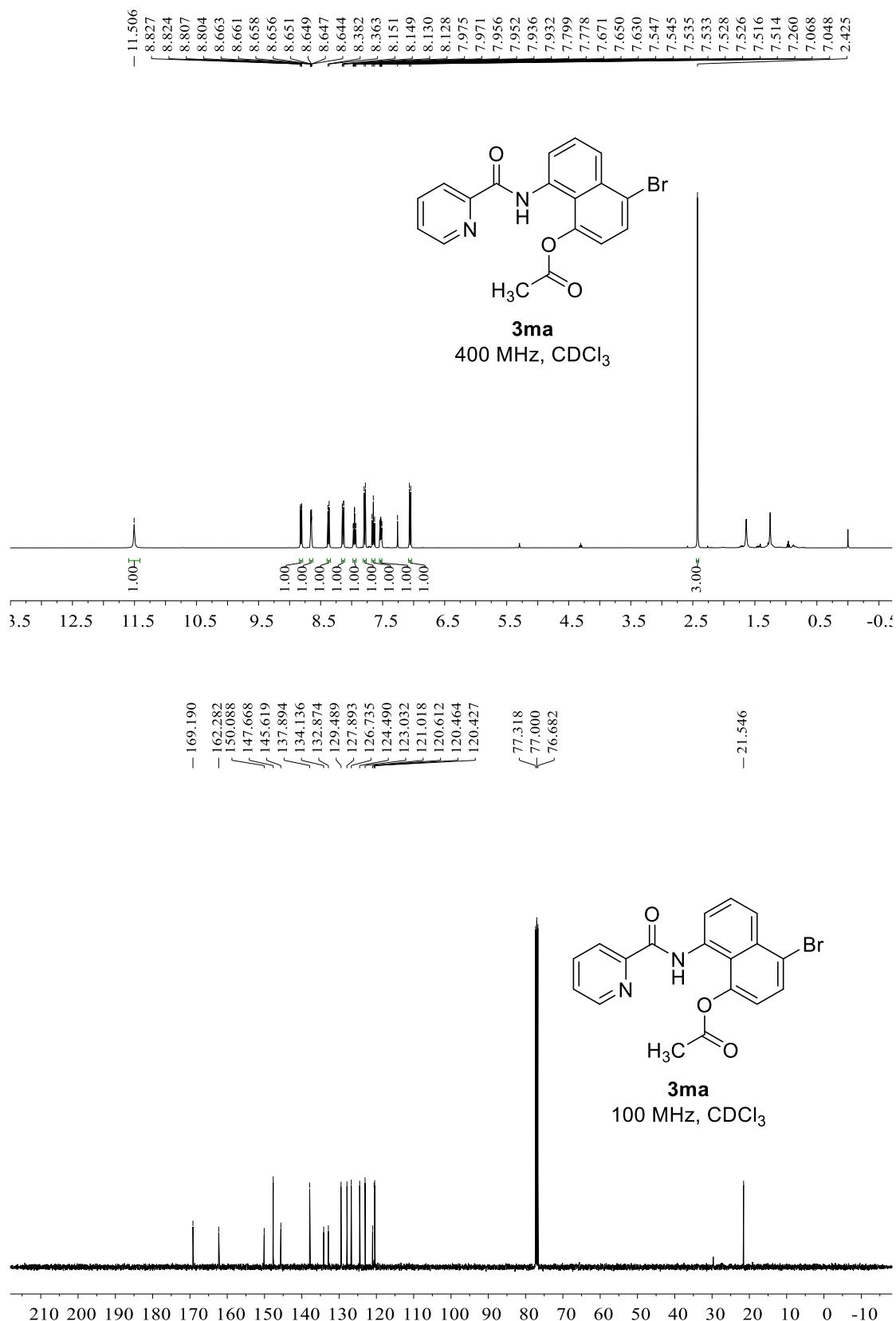


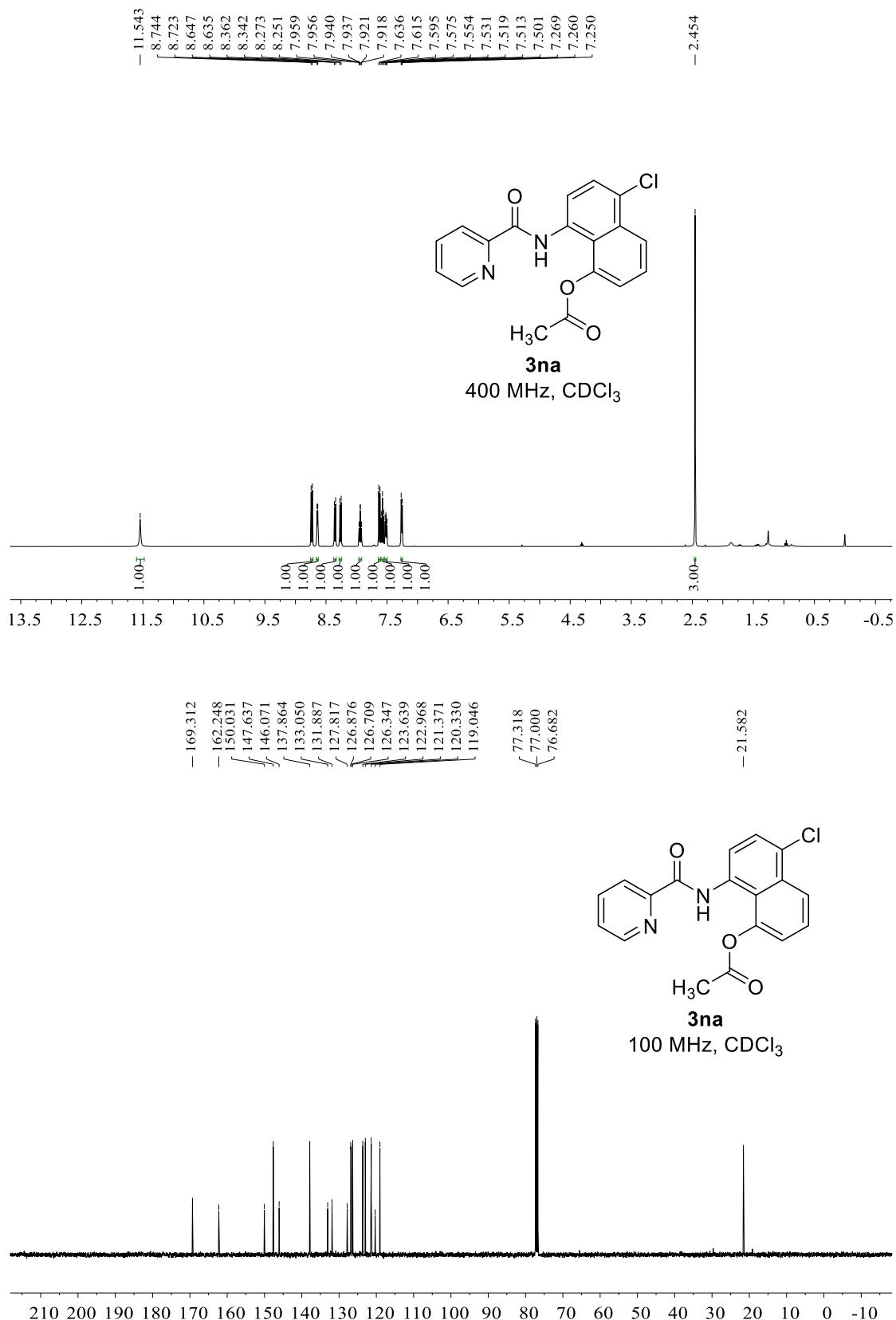
376 MHz, CDCl₃

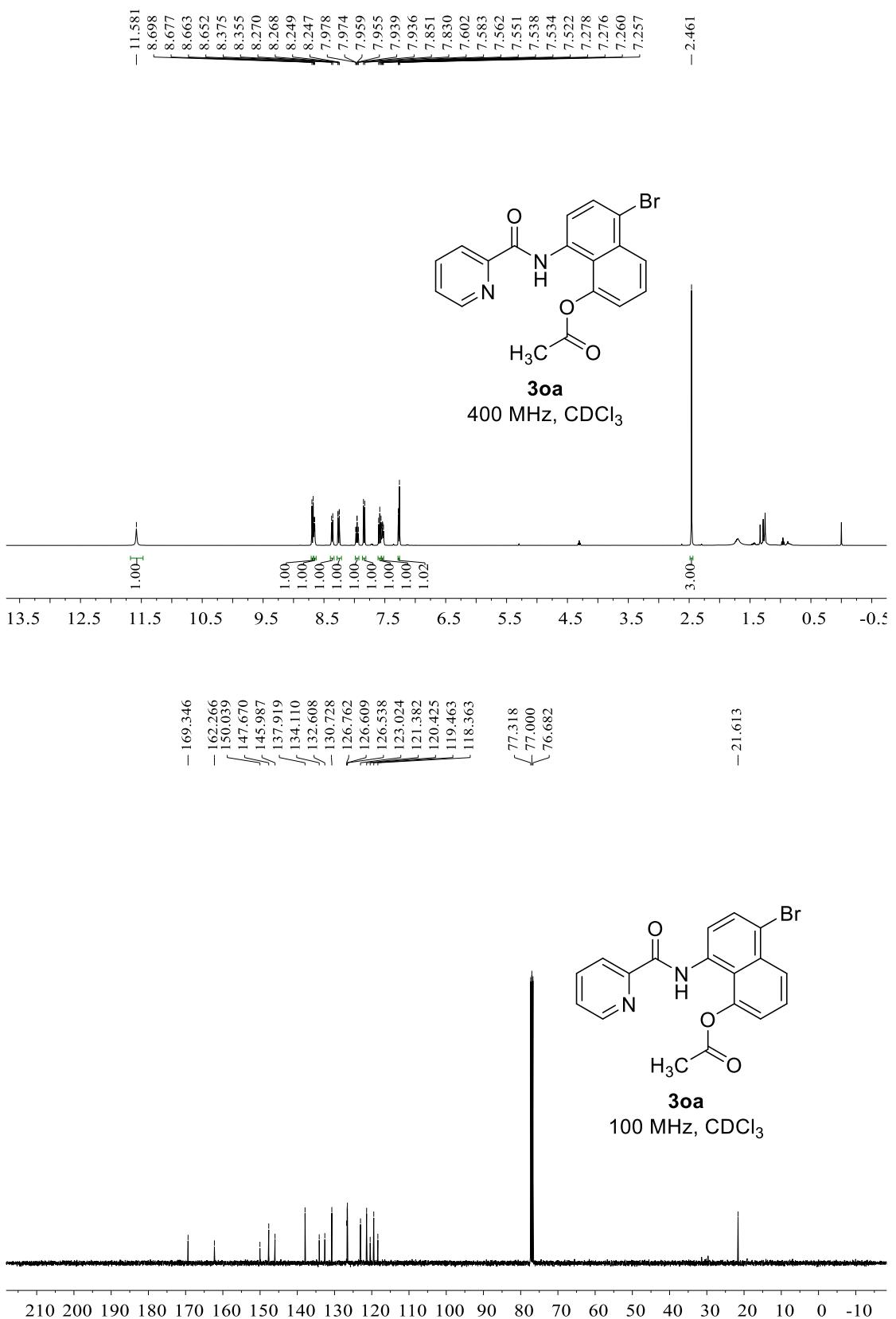


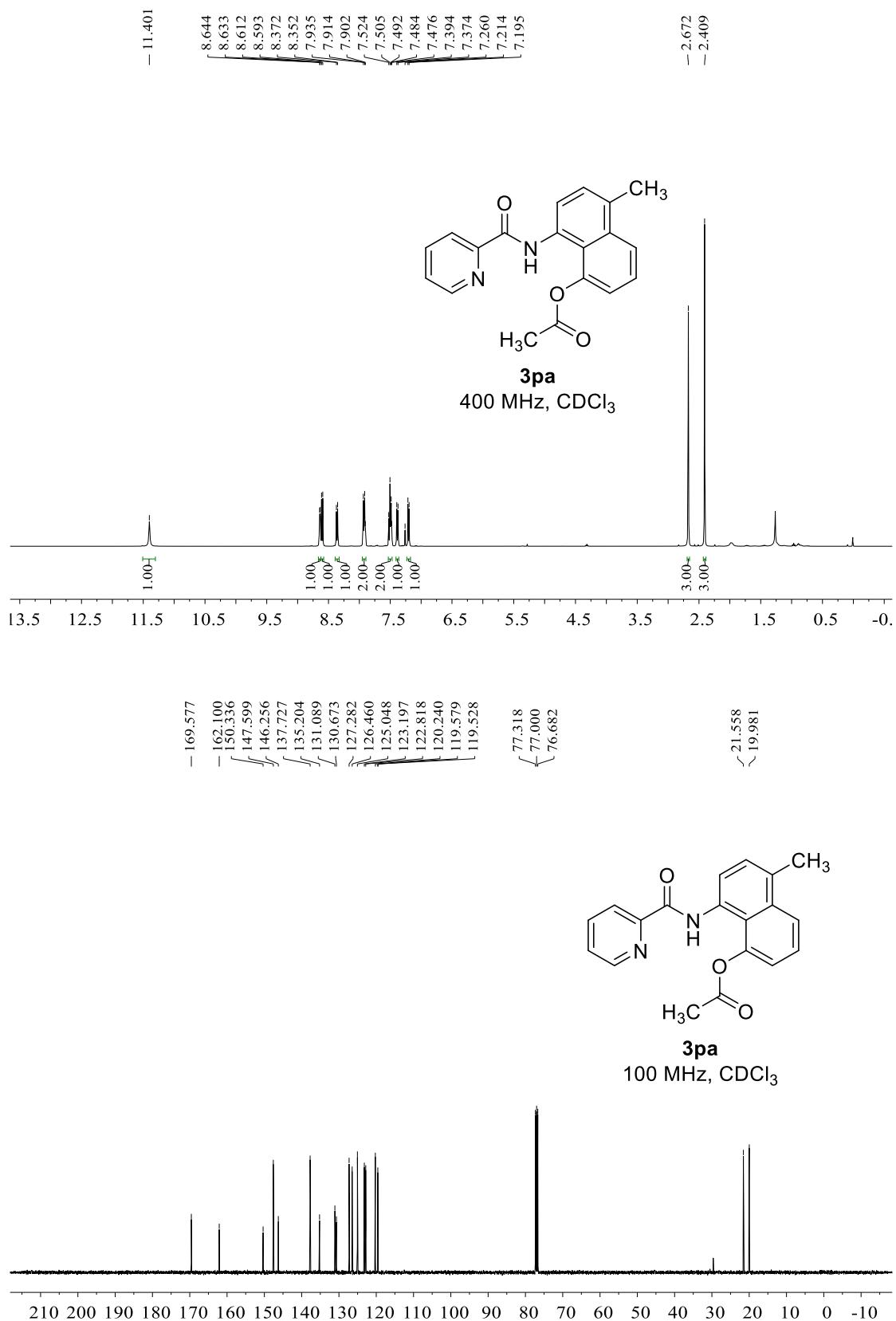


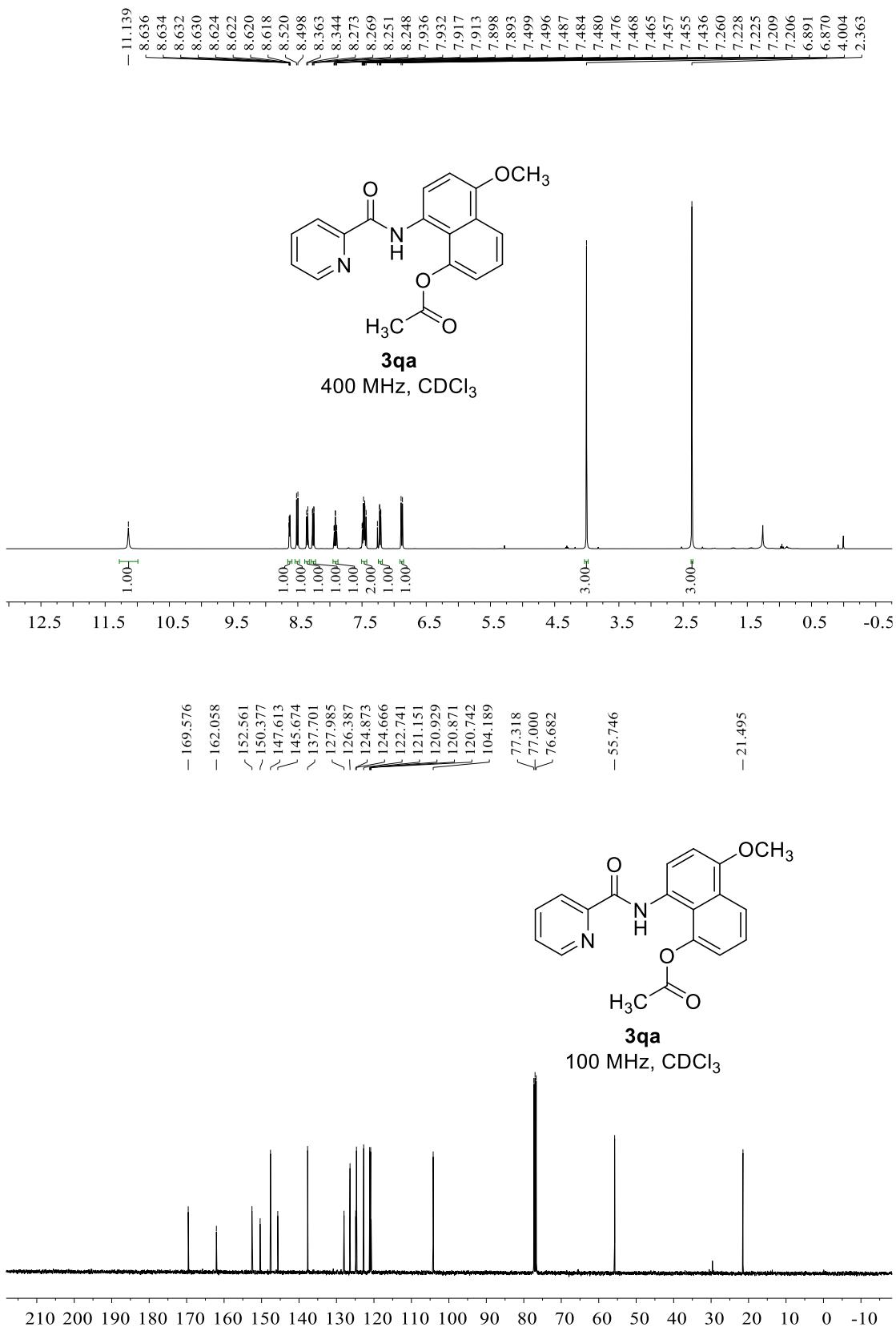


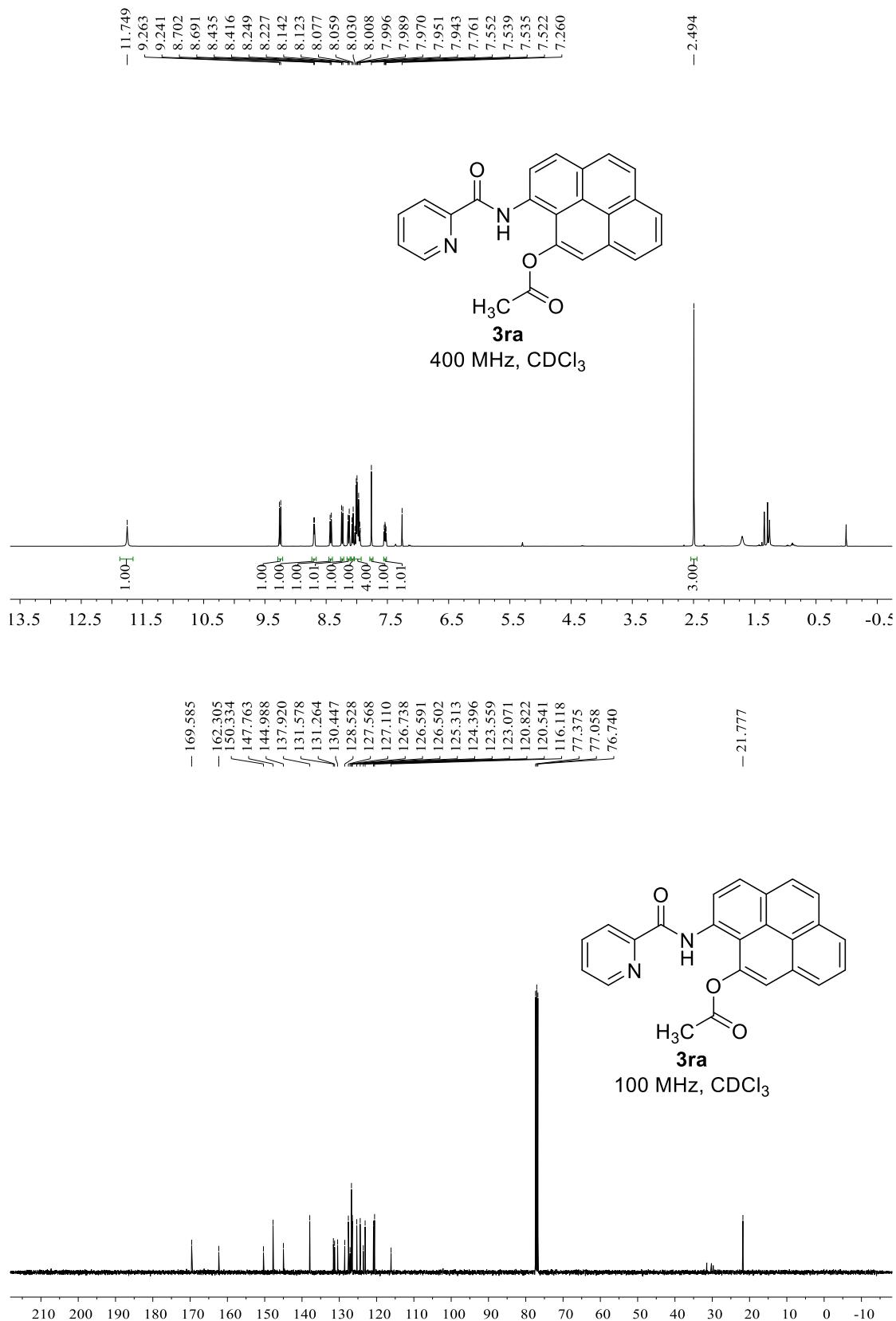


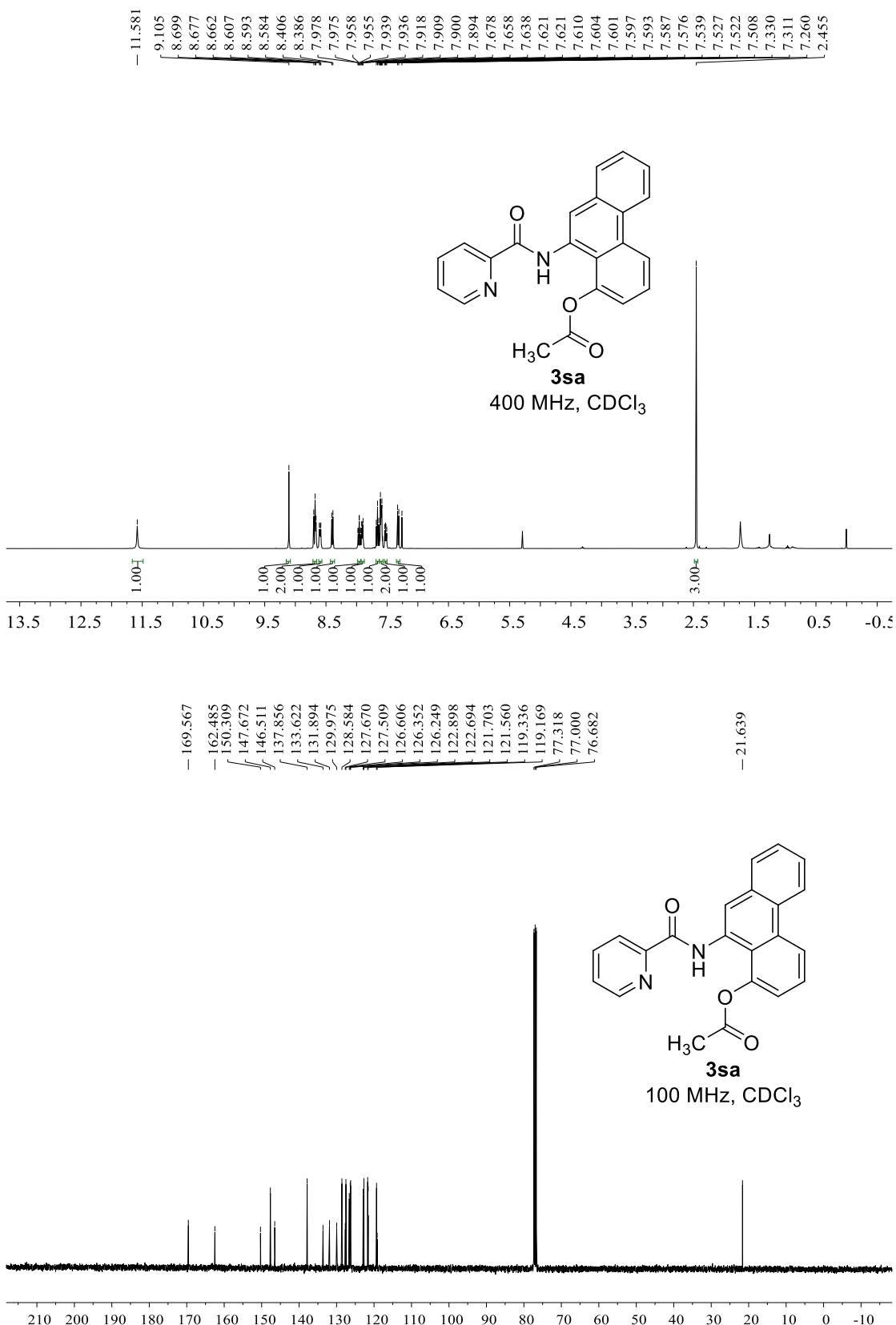


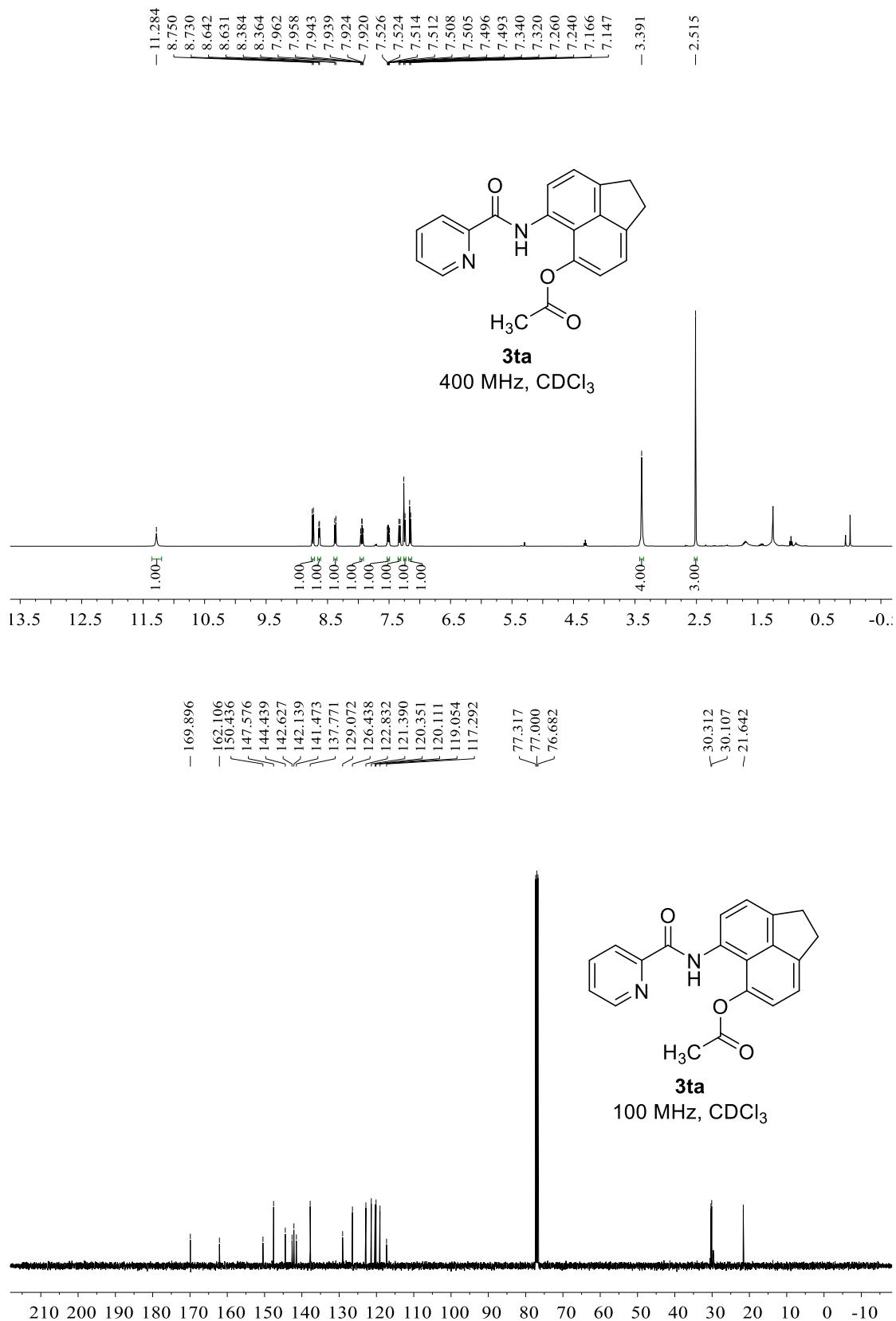


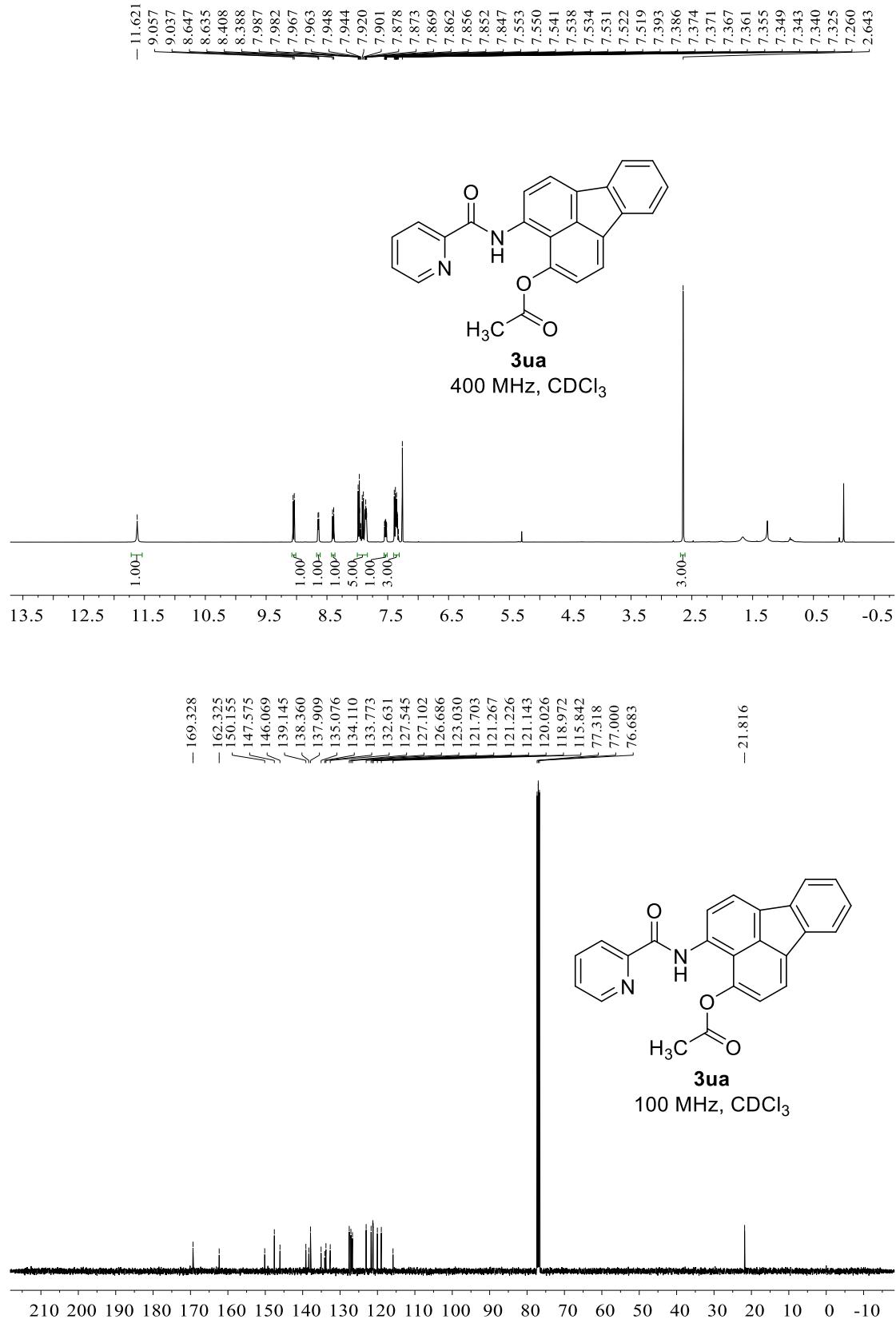


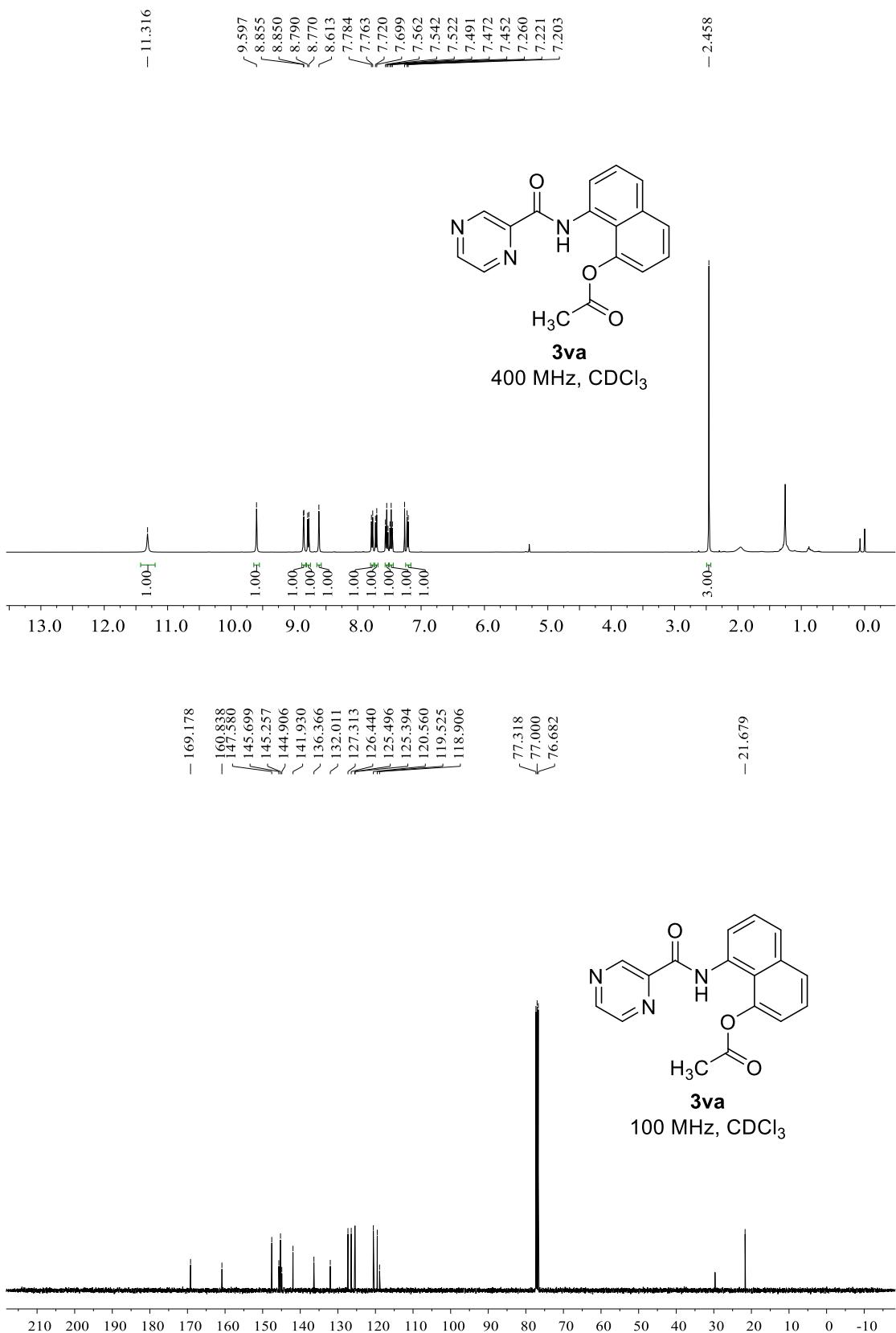


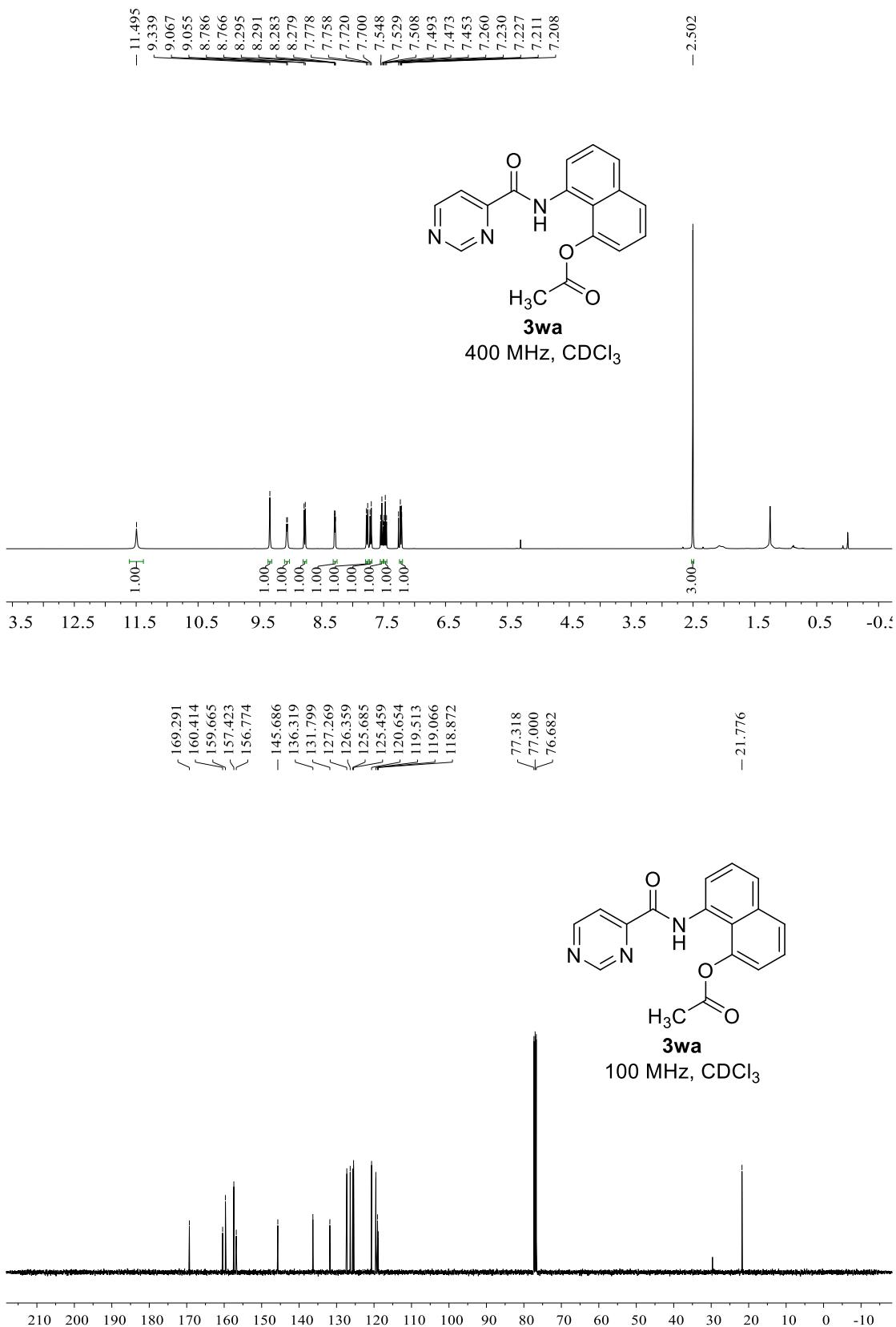


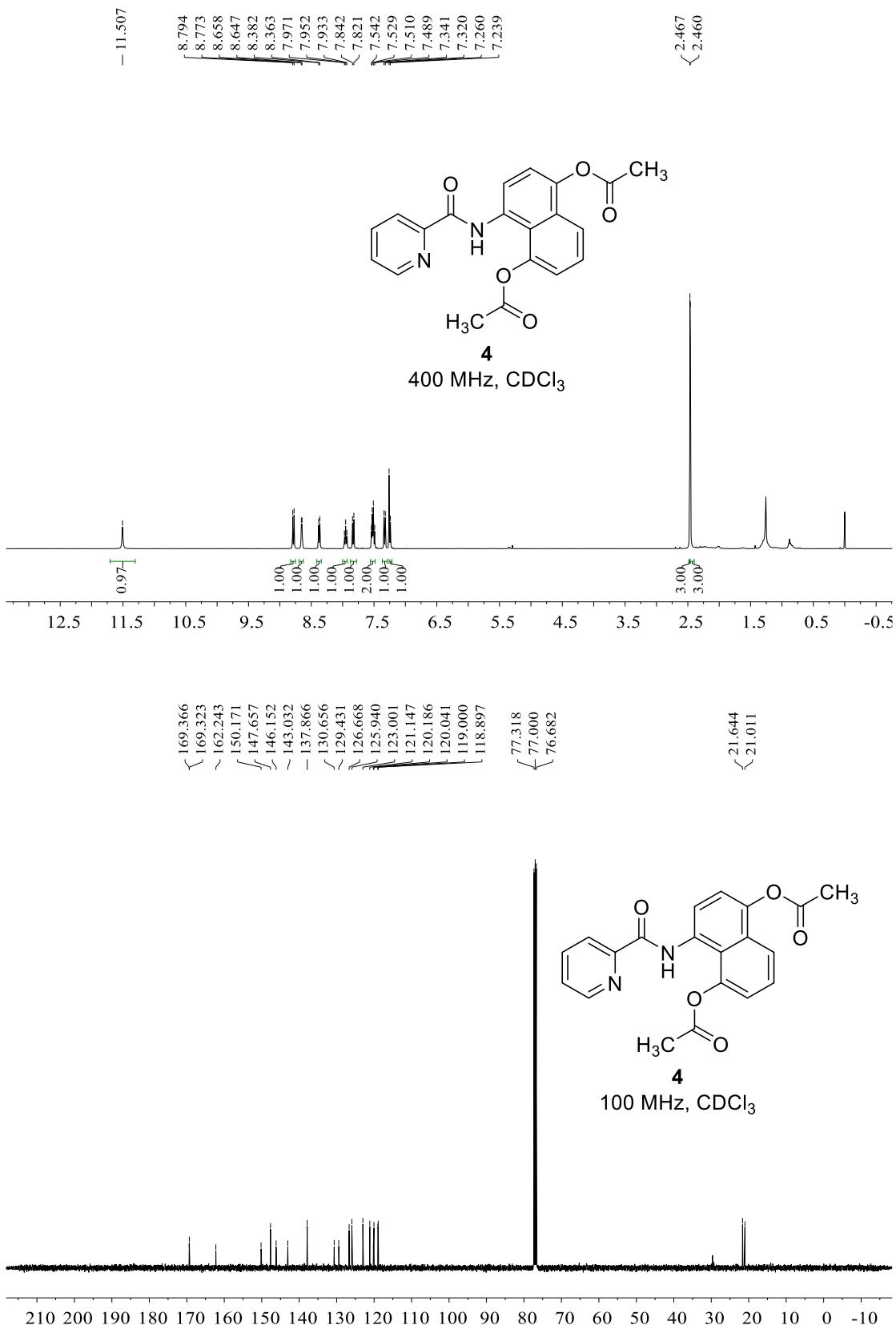


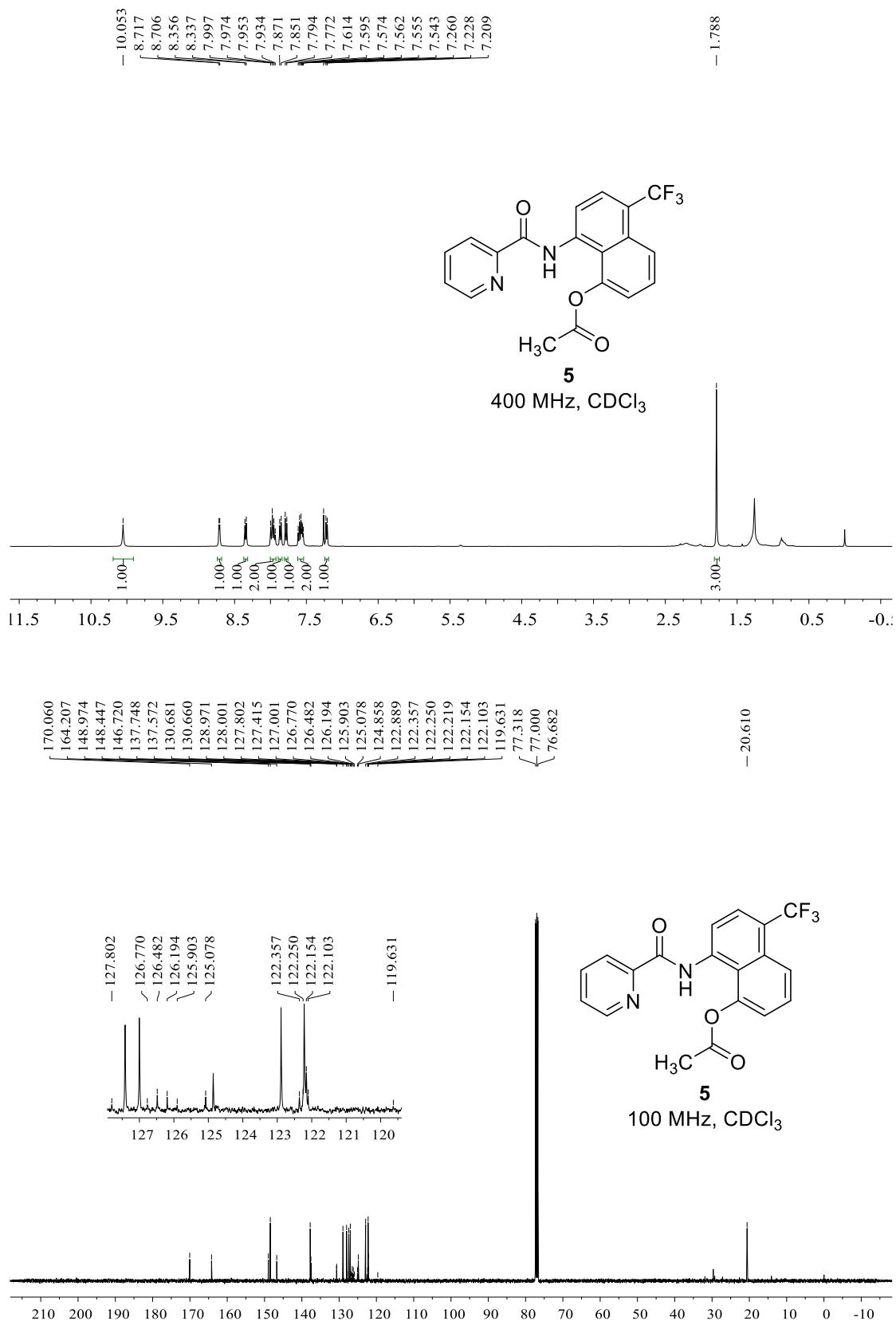




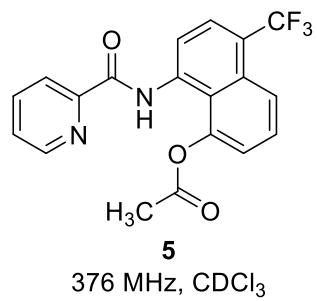




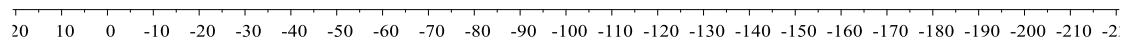


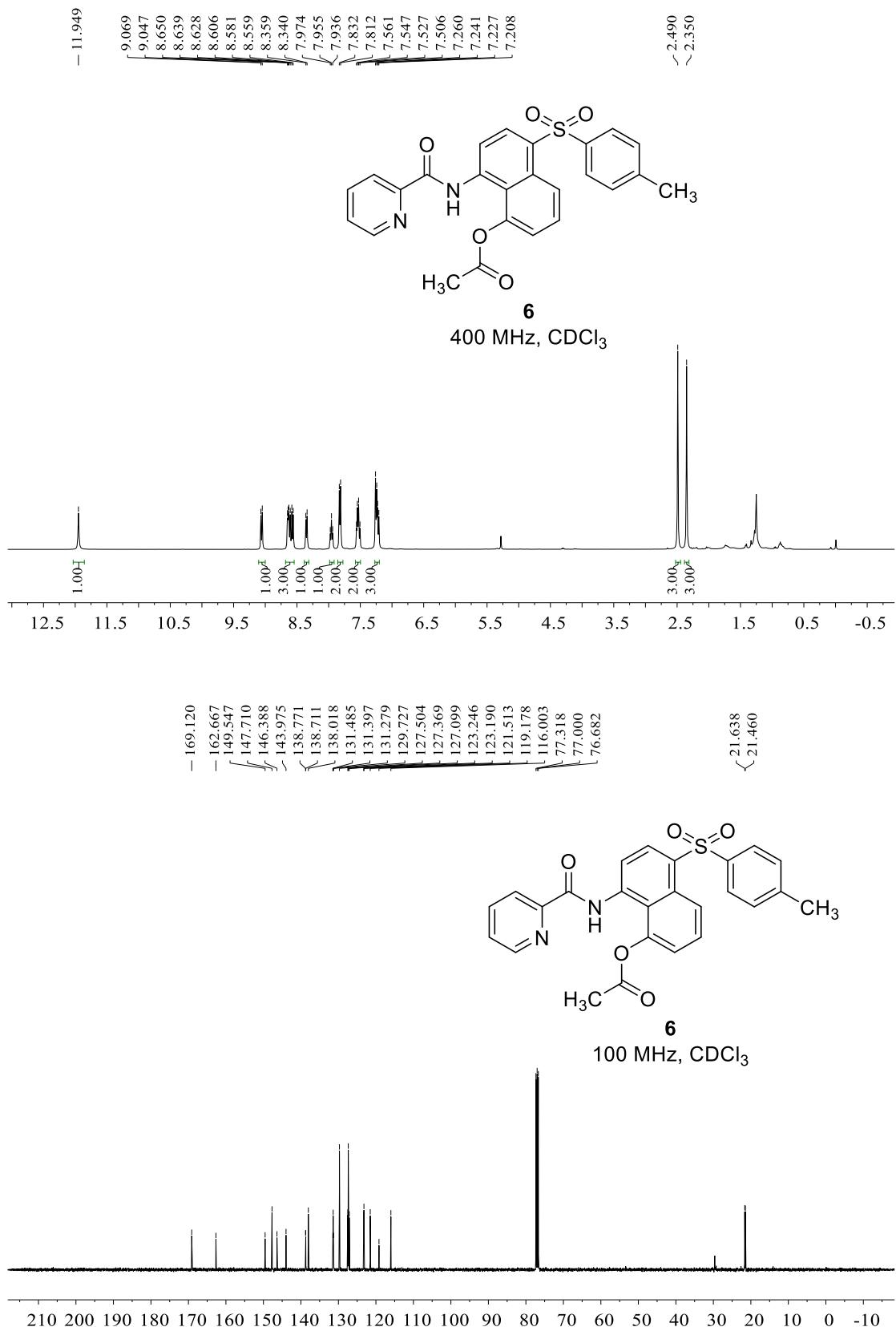


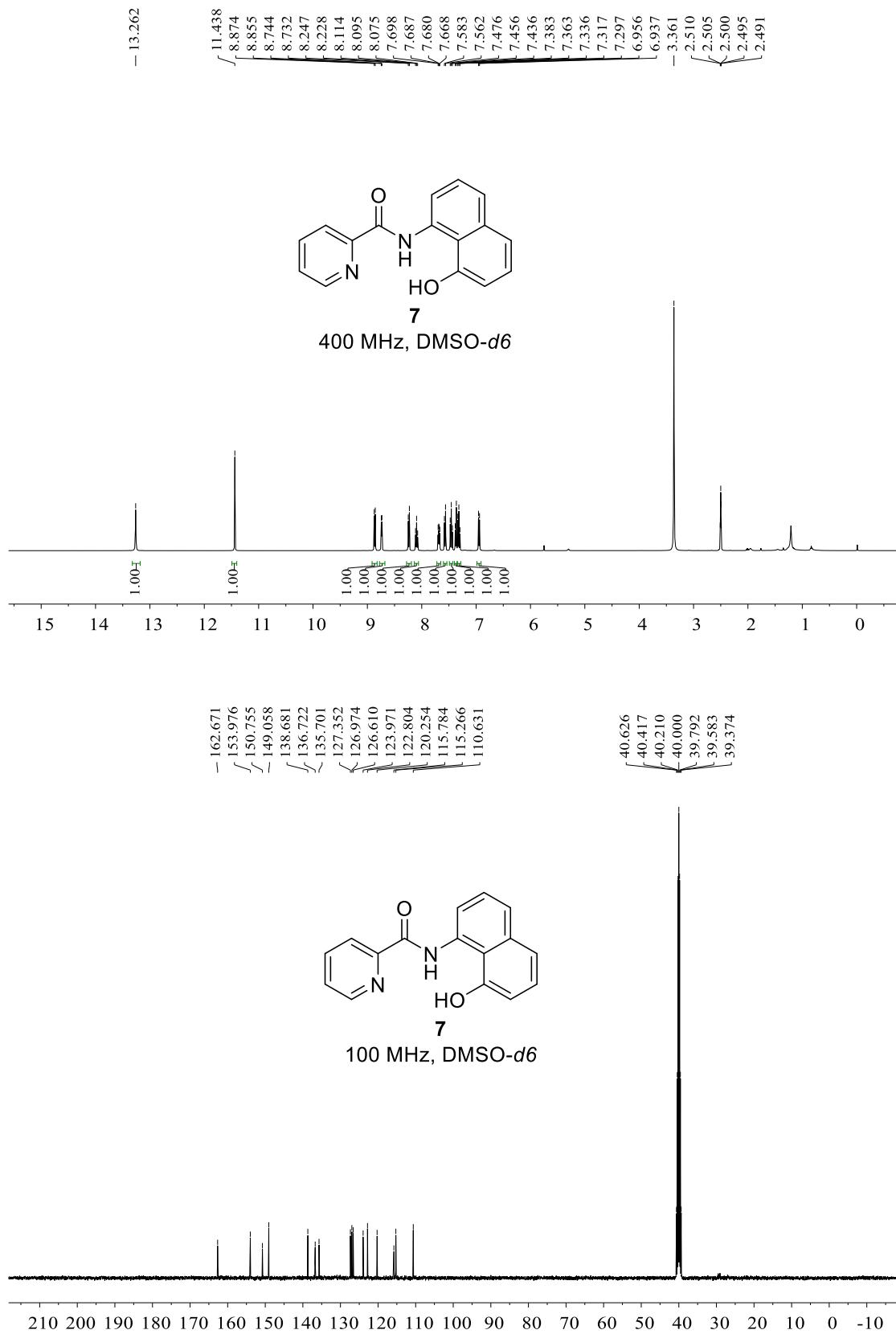
- -60.585

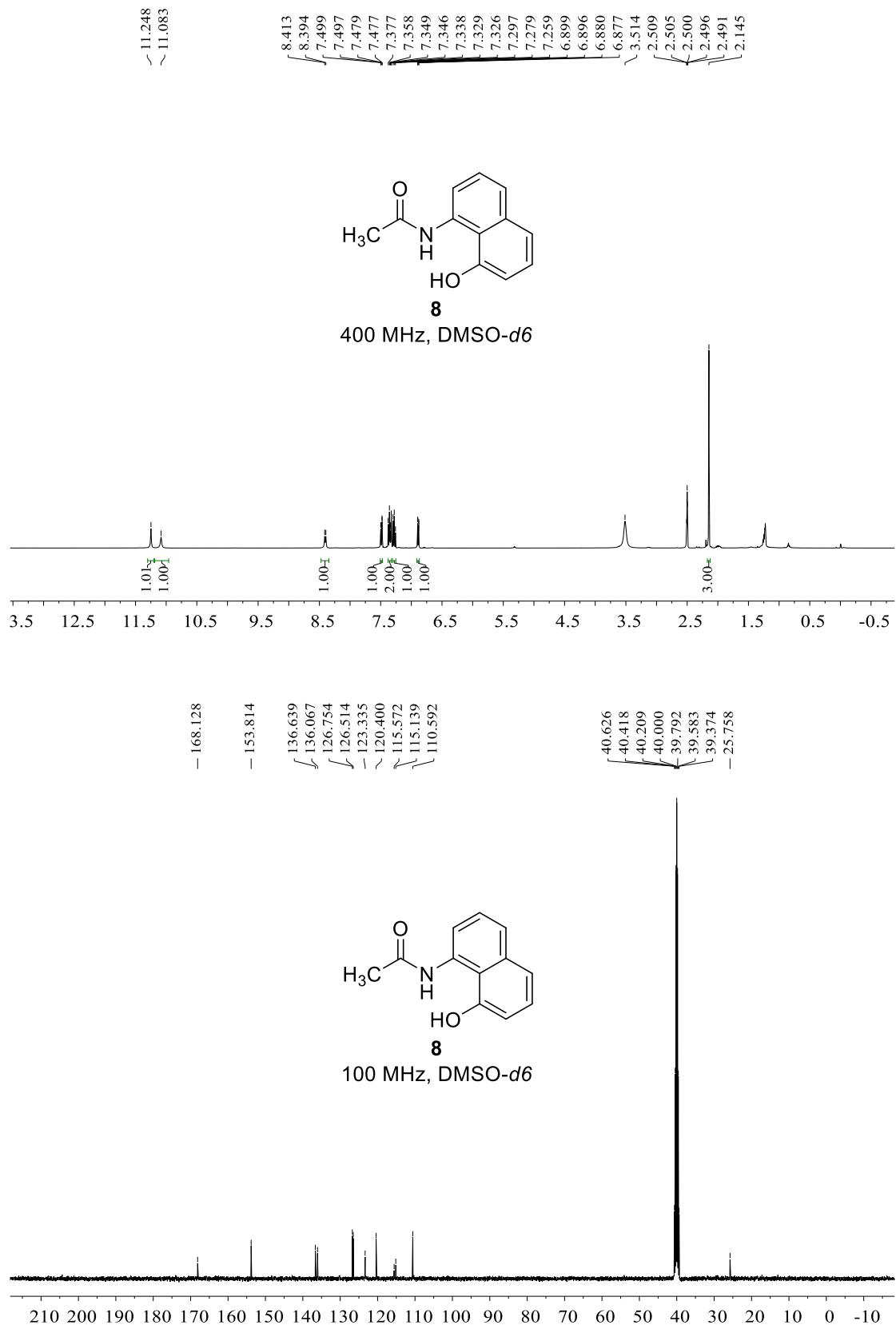


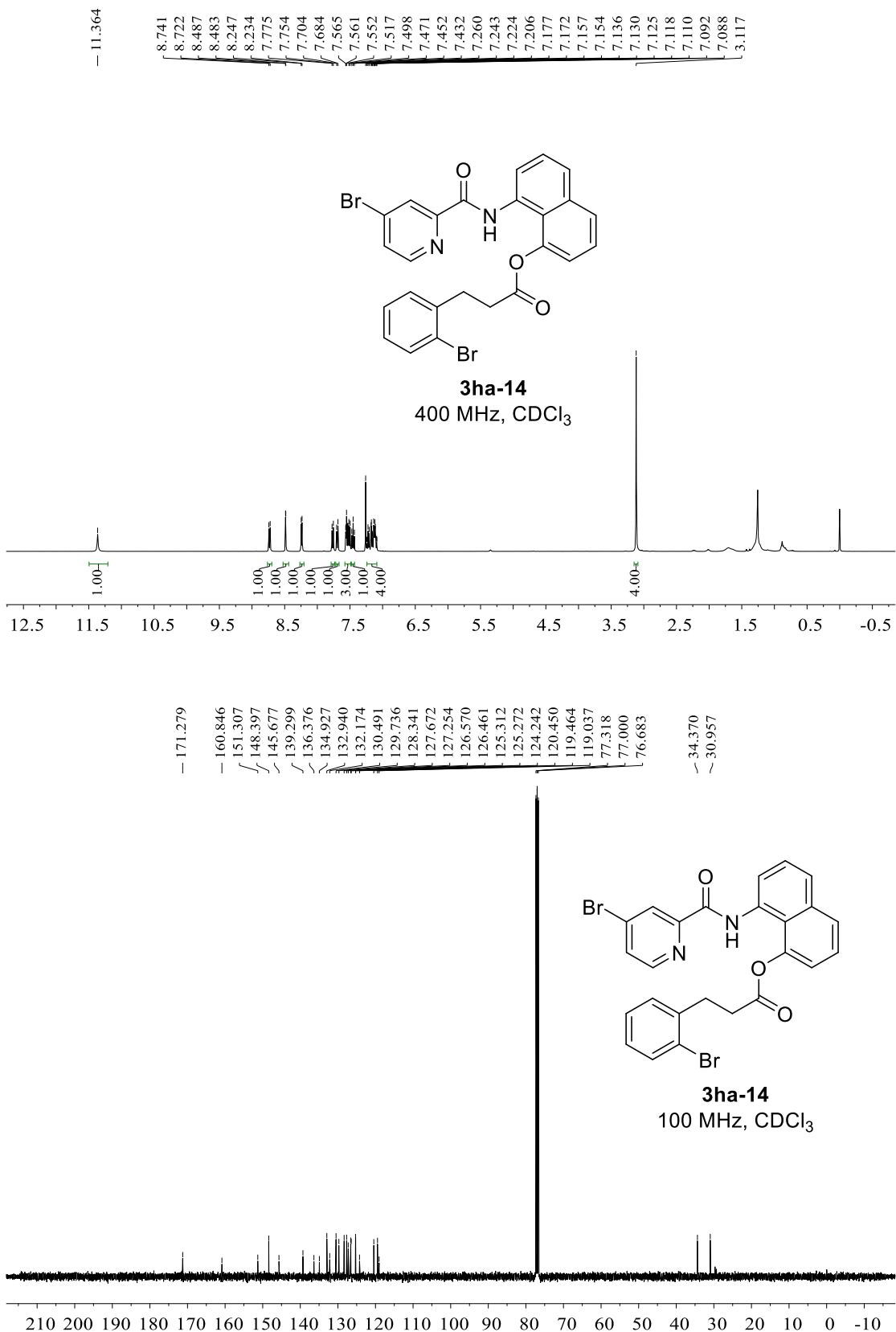
376 MHz, CDCl₃

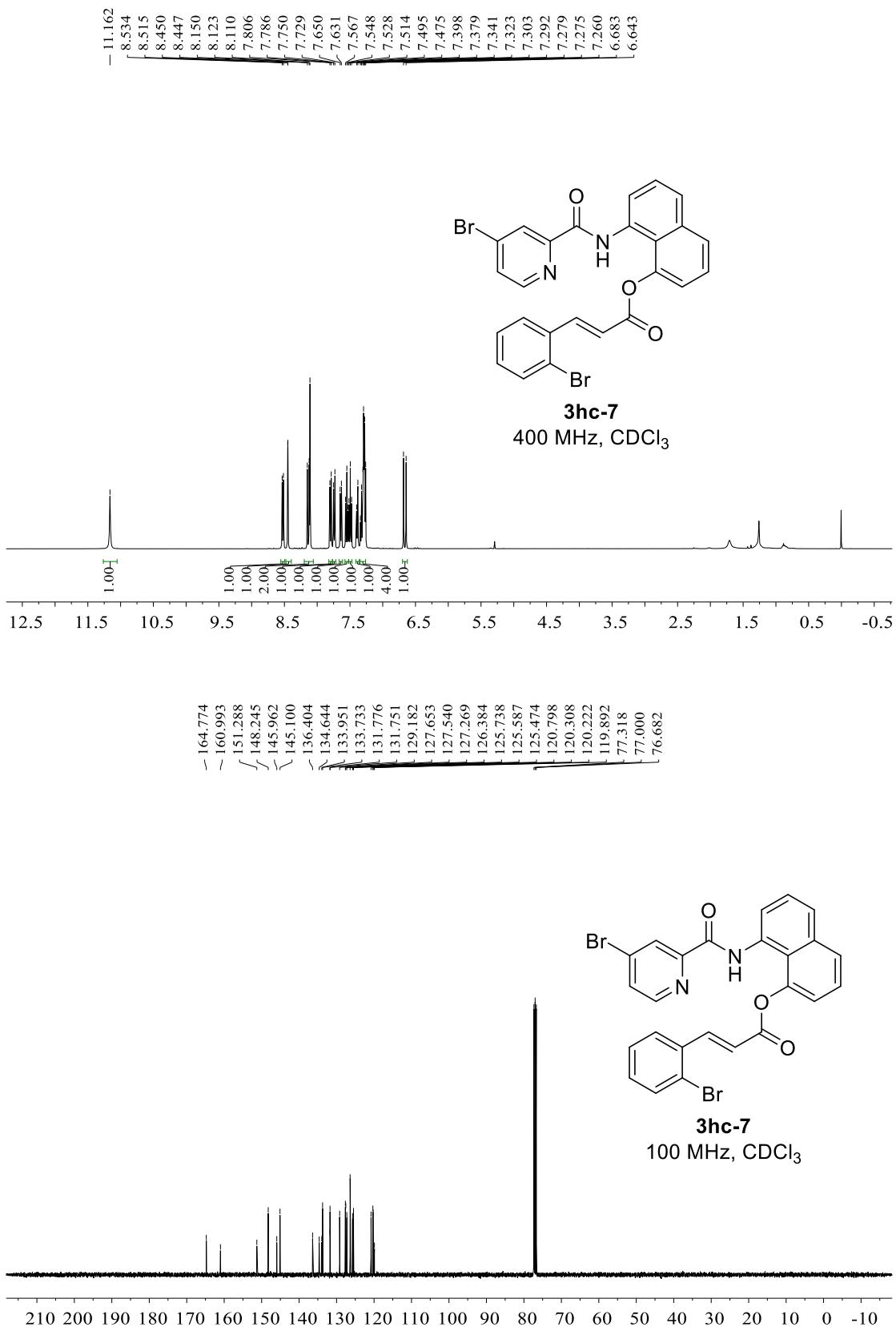


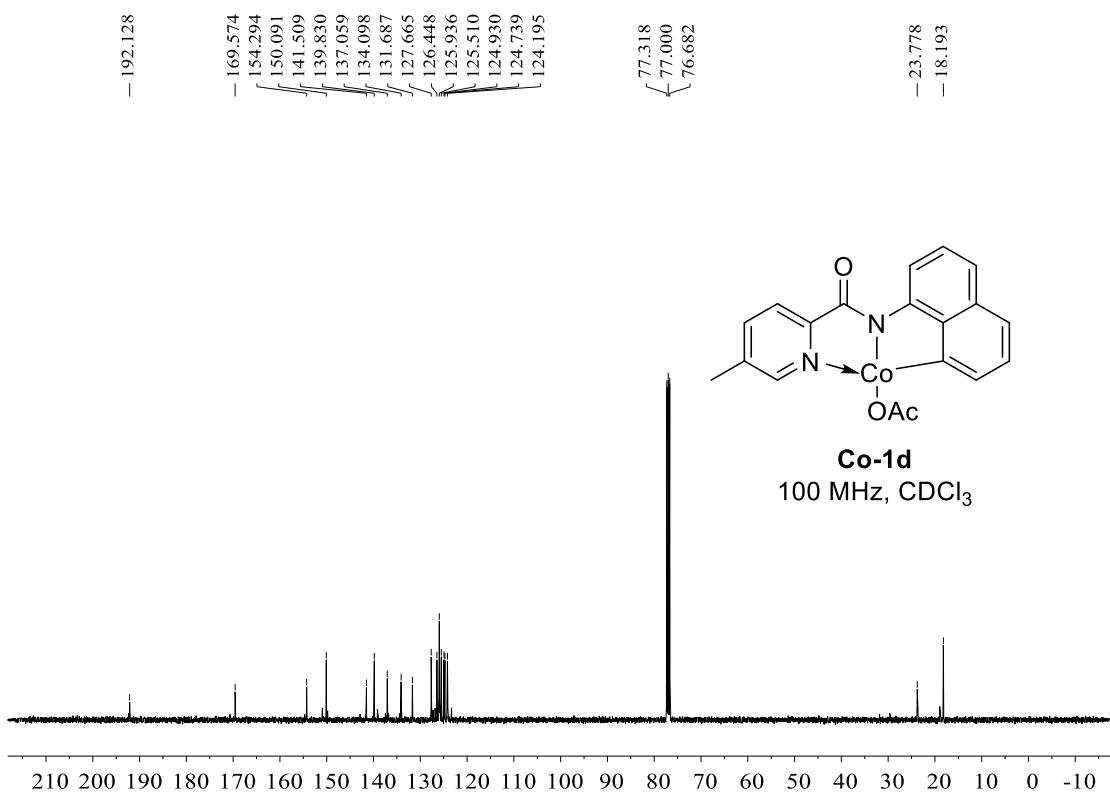
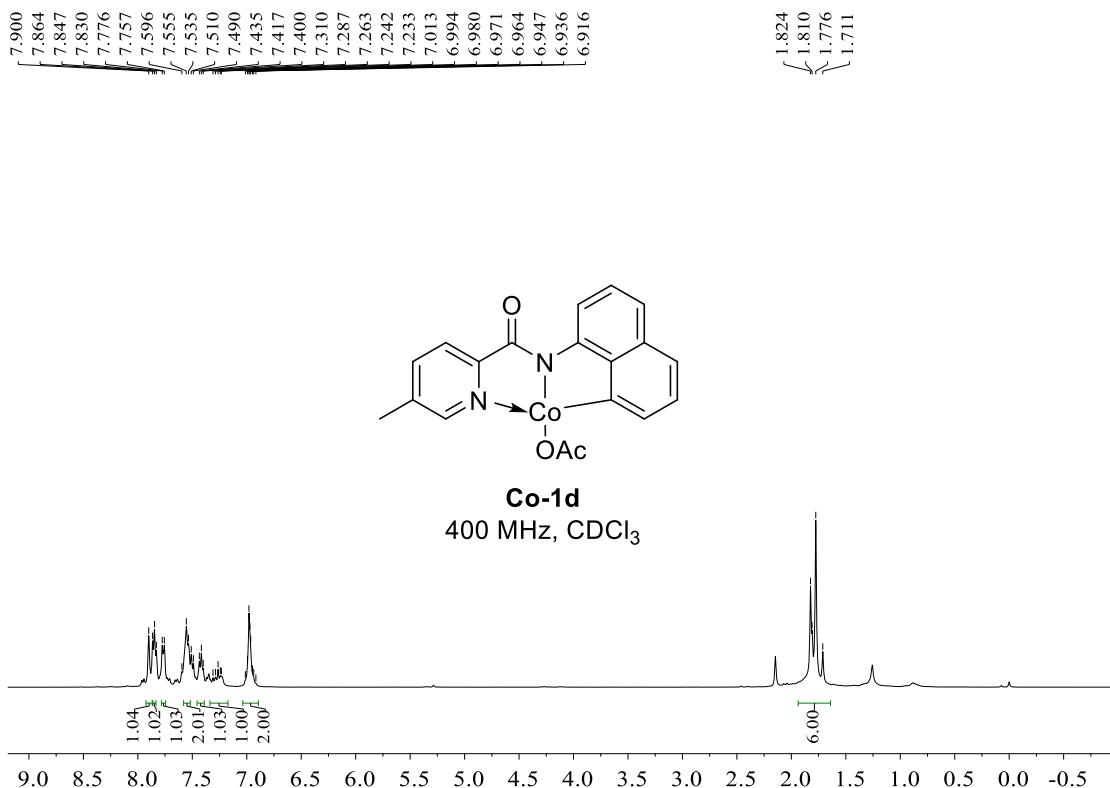




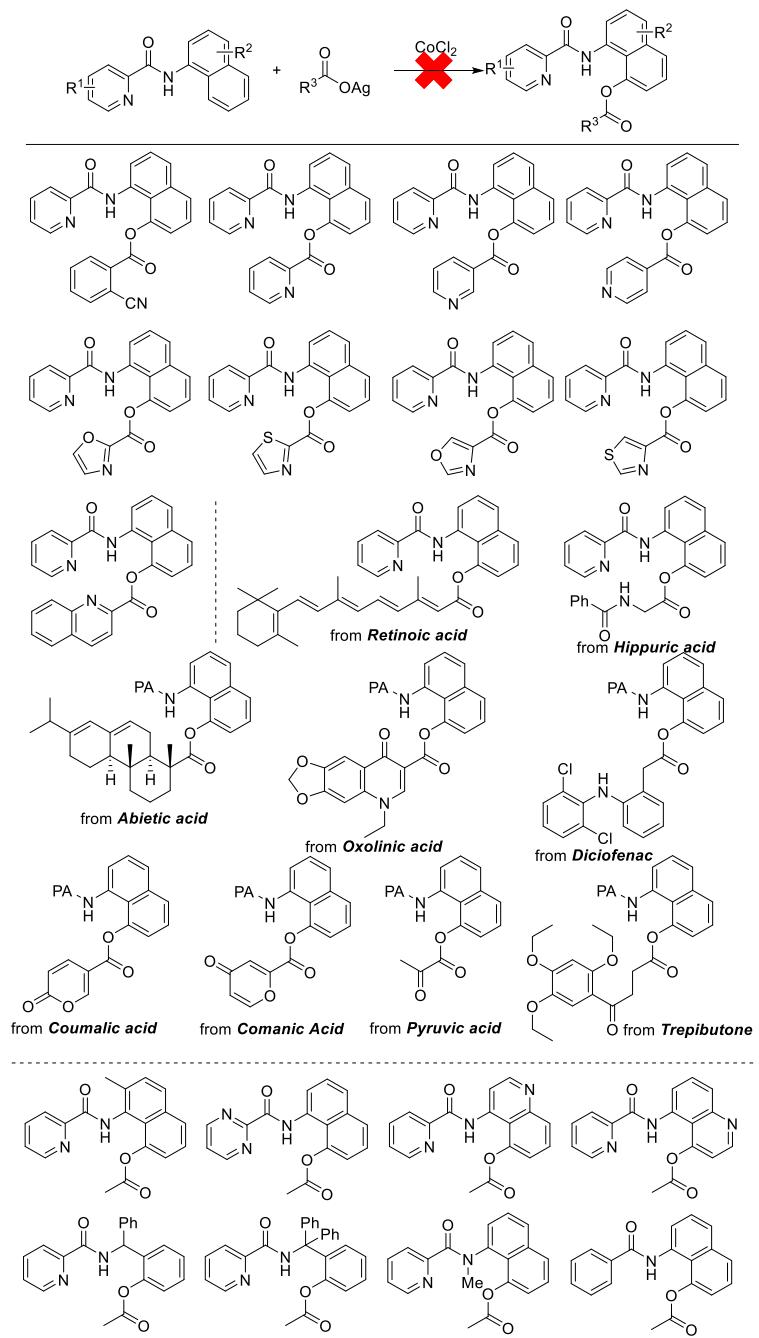








10. Failure examples of C–H bond acyloxylation.



11. References

- 1 (a) J.-Y. Lan, H.-S. Xie, X.-X. Lu, Y.-F. Deng, H.-F. Jiang, W. Zeng, *Org. Lett.* **2017**, *19*, 4279; (b) J. Ying, L.-Y. Fu, G.-Q. Zhong, X.-F. Wu, *Org. Lett.* **2019**, *21*, 5694; (c) Y. Yao, Q. Lin, W. Yang, W. Yang, F. Gu, W. Guo, D. Yang, *Chem. Eur. J.* **2020**, *26*, 5607.
- 2 K. J. Alexander, M. McConville, K. R. Williams, K. V. Luzyanin, I. A. O’Neil, R. Cosstick, *Chem. Eur. J.* **2018**, *24*, 3013.
- 3 L. Grigorjeva, O. Daugulis, *Angew. Chem. Int. Ed.* **2014**, *53*, 10209.
- 4 L. Wang, C. W. Barth, M. Sibrian-Vazquez, J. O. Escobedo, M. Lowry, J. Muschler, H. Li, S. L. Gibbs, R. M. Strongin, *ACS Omega* **2017**, *2*, 154.