

Supporting Information

Synthesis of *meta*-functionalized phenols and anilines

*Corresponding author, e-mail:

Contents

1. General Information.....	S3
2. Reaction Optimization	S4
2.1 Condition optimization for synthesis of <i>meta</i> -functionalized phenols	S4
2.2 Condition optimization for synthesis of <i>meta</i> -functionalized phenols	S8
3. Procedure for the Synthesis of Starting Materials and Products.....	S10
3.1 Procedure for the synthesis of starting materials	S10
3.2 General procedure for the synthesis of <i>meta</i> -functionalized phenols	S13
3.3 Gram-scale for the synthesis of <i>meta</i> -functionalized phenols	S14
3.4 General procedure for the synthesis of <i>meta</i> -functionalized anilines	S14
4. Characterization Data of Starting Materials and Products.....	S15
4.1 Characterization data of starting materials	S15
4.2 Characterization data of products	S23
5. Preliminary Mechanistic Studies	S55
5.1 ¹⁸ O-labeled water experiment	S55
5.2 Deuterium-labeling experiments.....	S55
5.2.1 H/D exchange experiments–investigations of γ -C–H activation	S55
5.2.2 H/D exchange experiments – investigations of α -C–H activation	S56
5.3 KIE experiments	S57
5.4 Intermediate trapping experiments and kinetic profiles.....	S60
5.4.1 Intermediate trapping experiments	S60
5.4.2 Kinetic profiles.....	S64
5.5 Kinetic studies.....	S65
5.5.1 Determination of kinetic rate constants of the first step: k_1	S65
5.5.2 Determination of kinetic rate constants of the second step: k_2	S66
5.5.3 Determination of kinetic rate constants of the third step: k_3	S67
5.5.4 Determination of kinetic rate constants of the fourth step: k_4	S68
5.6 Proposed mechanism	S70
6. X-Ray Crystallographic Data.....	S72
7. NMR Spectra	S77
8. References.....	S190

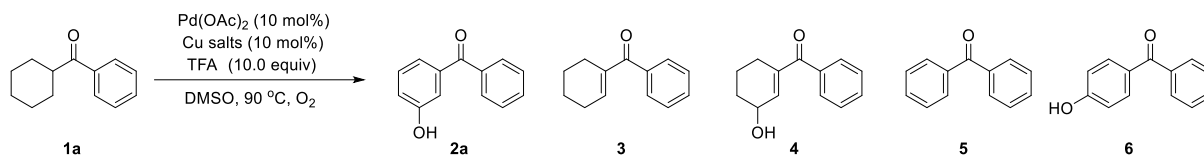
1. General Information

Unless noted otherwise, commercially available chemicals were used without further purification. Flash chromatography was performed with silica gel (200-300 mesh). Oil bath served as the heat source. NMR spectra were acquired on Bruker 400 MHz (^1H at 400 MHz, ^{13}C at 101 MHz) or Jeol 400 MHz (^1H at 400 MHz, ^{13}C at 101 MHz). Chemical shifts are reported in parts per million (ppm, δ), downfield from tetramethylsilane (TMS, $\delta = 0.00$ ppm). The residual solvent signals were used as references for ^1H and ^{13}C NMR spectra (CDCl_3 : $\delta_{\text{H}} = 7.26$ ppm, $\delta_{\text{C}} = 77.2$ ppm; Acetone- d_6 : $\delta_{\text{H}} = 2.05$ ppm, $\delta_{\text{C}} = 29.8, 206.3$ ppm). Coupling constants, J were reported in Hertz unit (Hz). Data for ^1H NMR spectra were reported as follows: chemical shift (ppm, referenced to protium, s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, td = triplet of doublets, m = multiplet, br = broad, coupling constant (Hz), and integration). Infrared (IR) data were acquired on a Bruker Invenio-R FT-IR spectrometer. Absorbance frequencies are reported in reciprocal centimeters (cm^{-1}). Mass spectra were acquired on a BrukerDaltonics S2 MicroTof-Q II mass spectrometer. X-ray crystal structure analyses were measured on Bruker Smart APEXIICCD instrument using Mo- $K\alpha$ radiation ($\lambda = 0.71073$ Å). The structures were solved and refined using the SHELXTL software package.

2. Reaction Optimization

2.1 Condition optimization for synthesis of *meta*-functionalized phenols

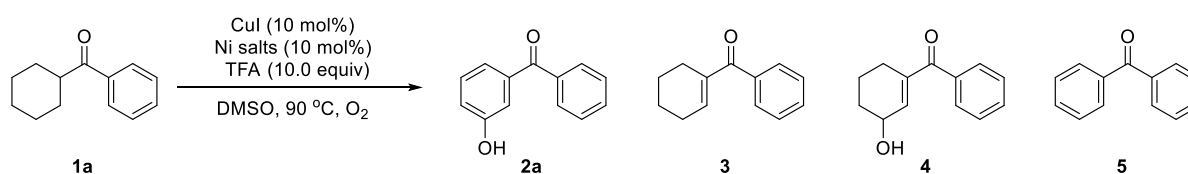
Table S1. Screening of Cu salts^{a,b}



Entry	Catalyst	Cu salts	2a (%)	3 (%)	4 (%)	5 (%)	6 (%)
1	Pd(OAc) ₂	Cu(OAc) ₂	6	trace	trace	11	7
2	Pd(OAc) ₂	CuI	16	trace	trace	7	trace
3	Pd(OAc) ₂	Cu(OAc)	6	trace	trace	14	6
4	Pd(OAc) ₂	CuBr ₂	5	trace	trace	6	trace
5	Pd(OAc) ₂	CuBr	trace	trace	trace	8	trace
6	Pd(OAc) ₂	CuCl ₂	-	-	-	-	-
7	Pd(OAc) ₂	CuCl	trace	trace	trace	7	trace
8	Pd(OAc) ₂	CuSO ₄	6	trace	trace	13	7
9	Pd(OAc) ₂	Cu(OTf) ₂	7	trace	trace	16	9
10	Pd(OAc) ₂	Cu ₂ O	5	trace	trace	10	7
11	Pd(OAc) ₂	-	trace	trace	trace	12	5
12	-	CuI	41	trace	trace	<5	n.d.

^aReaction conditions: **1a** (0.25 mmol), Pd(OAc)₂ (10 mol%), Cu salts (10 mol%), TFA (10.0 equiv), DMSO (1 mL), under O₂ at 90 °C for 60 hours. ^bThe yields were determined by ¹H NMR using dibromomethane as the internal standard.

Table S2. Screening of Ni salts^{a,b}

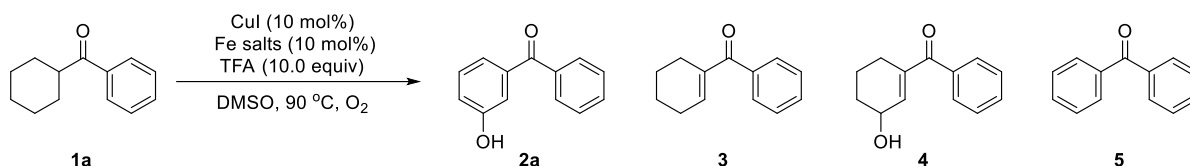


Entry	Ni salts	2a (%)	3 (%)	4 (%)	5 (%)
1	NiI ₂	19	trace	trace	<5
2	NiCl ₂	33	trace	trace	<5
3	NiBr ₂	32	7	trace	<5
4	Ni(OAc) ₂ ·4H ₂ O	35	9	trace	<5
5	NiBr ₂ (dme)	38	8	trace	<5
6	Ni(OTf) ₂	33	10	trace	<5
7	Ni(acac) ₂ (II)	26	trace	trace	<5
8	NiCp ₂	29	trace	trace	<5
9	Ni(PPh ₃) ₂ Cl ₂	36	trace	trace	<5
10	Ni(PPh ₃) ₂ Br ₂	28	trace	trace	<5

11 Ni(PCy₃)₂Cl₂ 30 trace trace <5

^aReaction conditions: **1a** (0.25 mmol), CuI (10 mol%), Ni salts (10 mol%), TFA (10.0 equiv), DMSO (1 mL), under O₂ at 90 °C for 60 hours. ^bThe yields were determined by ¹H NMR using dibromomethane as the internal standard.

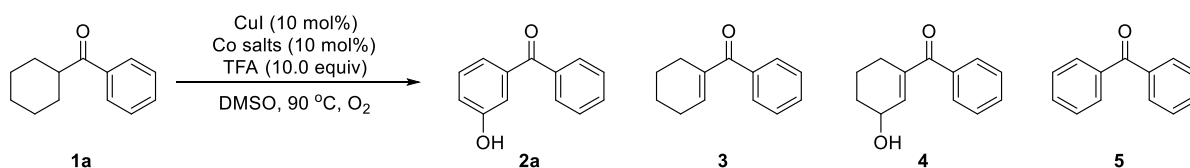
Table S3. Screening of Fe salts^{a,b}



Entry	Fe salts	2a (%)	3 (%)	4 (%)	5 (%)
1	Ferrocene	24	6	trace	<5
2	K ₃ [Fe(CN) ₆]	21	8	trace	<5
3	FeCl ₂	26	trace	trace	<5
4	Fe(CO) ₁₂	19	5	trace	<5
5	FeSO ₄ · 7H ₂ O	31	7	trace	<5
6	FeSO ₄	28	7	11	<5
7	Fe(acac) ₃ (III)	17	5	7	<5
8	Fe ₂ (C ₂ O ₄) ₃ · 6H ₂ O	25	5	9	<5
9	Fe ₂ (CO) ₉	23	trace	8	<5

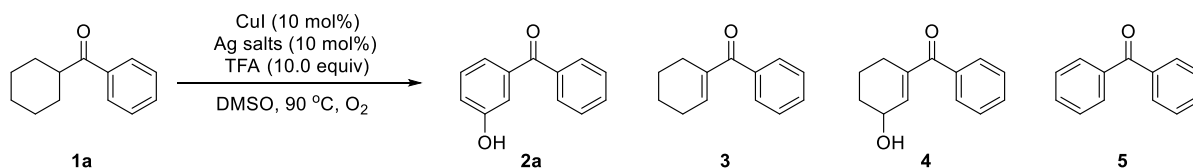
^aReaction conditions: **1a** (0.25 mmol), CuI (10 mol%), Fe salts (10 mol%), TFA (10.0 equiv), DMSO (1 mL), under O₂ at 90 °C for 60 hours. ^bThe yields were determined by ¹H NMR using dibromomethane as the internal standard.

Table S4. Screening of Co salts^{a,b}



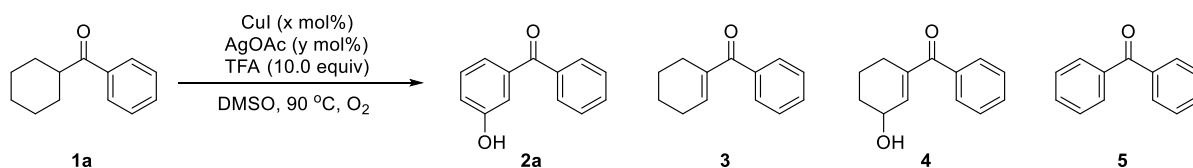
Entry	Co salts	2a (%)	3 (%)	4 (%)	5 (%)
1	Co(OAc) ₂	23	6	trace	<5
2	Co(acac) ₂	16	5	trace	<5
3	CoBr ₂	20	5	trace	<5
4	Co(PPh ₃) ₃ Cl	21	7	trace	<5
5	Co(C ₂ O ₄) · 2H ₂ O	18	8	trace	<5

^aReaction conditions: **1a** (0.25 mmol), CuI (10 mol%), Co salts (10 mol%), TFA (10.0 equiv), DMSO (1 mL), under O₂ at 90 °C for 60 hours. ^bThe yields were determined by ¹H NMR using dibromomethane as the internal standard.

Table S5. Screening of Ag salts^{a,b}

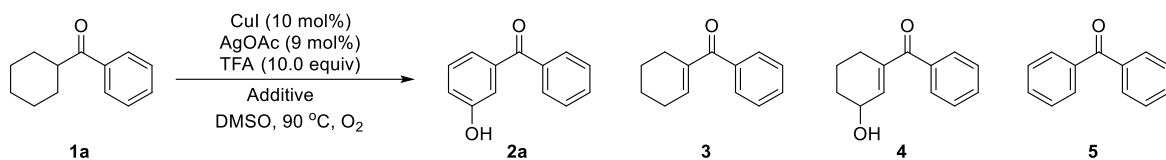
Entry	Catalyst	Ag salts	2a (%)	3 (%)	4 (%)	5 (%)
1	CuI	AgOAc	53	17	13	6
2	CuI	AgTFA	47	18	15	7
3	CuI	AgOTf	50	17	14	7
4	CuI	AgNO ₃	48	19	16	6
5	CuI	Ag ₂ O	-	-	-	-
6	CuI	Ag ₂ CO ₃	-	-	-	-
7	CuI	Ag ₃ PO ₄	-	-	-	-

^aReaction conditions: **1a** (0.25 mmol), CuI (10 mol%), Ag salts (10 mol%), TFA (10.0 equiv), DMSO (1 mL), under O₂ at 90 °C for 60 hours. ^bThe yields were determined by ¹H NMR using dibromomethane as the internal standard.

Table S6. Screening of the ratio of AgOAc to CuI^{a,b}

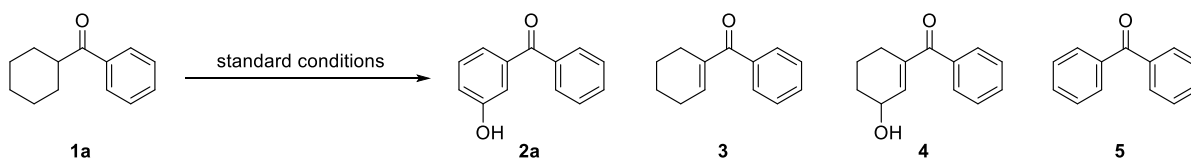
Entry	CuI (mol%)	AgOAc (mol%)	2a (%)	3 (%)	4 (%)	5 (%)
1	10	10	53	17	13	6
2	10	11	32	21	14	7
3	10	12	-	-	-	-
4	10	20	-	-	-	-
5	20	10	45	trace	trace	<5
6	10	9	64	15	8	<5
7	10	8	60	13	6	<5
8	10	7	57	12	5	<5
9	10	6	58	10	trace	<5
10	10	5	54	8	trace	<5
11	10	2.5	47	7	trace	<5

^aReaction conditions: **1a** (0.25 mmol), CuI (x mol%), Ag(OAc)₂ (y mol%), TFA (10.0 equiv), DMSO (1 mL), under O₂ at 90 °C for 60 hours. ^bThe yields were determined by ¹H NMR using dibromomethane as the internal standard.

Table S7. Screening of water additive^{a,b}

Entry	H ₂ O (uL)	2a (%)	3 (%)	4 (%)	5 (%)
1	25	71	9	trace	<5
2	50	76 (71) ^c	trace	trace	<5
3	75	70	trace	trace	<5
4	100	63	6	trace	<5
5	200	54	17	trace	<5
6	300	51	10	trace	<5

^aReaction conditions: **1a** (0.25 mmol), CuI (10 mol%), Ag(OAc)₂ (9 mol%), TFA (10.0 equiv), DMSO (1 mL), H₂O (x uL), under O₂ at 90 °C for 60 hours. ^bThe yields were determined by ¹H NMR using dibromomethane as the internal standard. ^cIsolated yield.

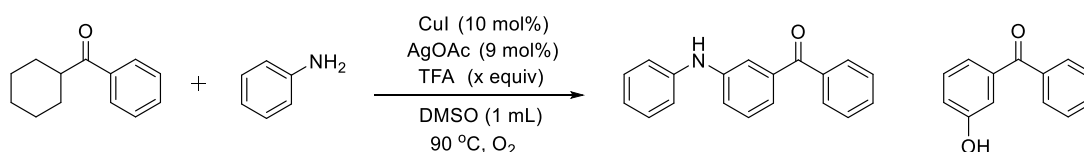
Table S8. Control experiments^{a,b}

Entry	Catalyst	Oxidant	TFA (equiv)	additive	2a (%)	3 (%)	4 (%)	5 (%)
1	CuI	AgOAc	10.0	H ₂ O	76 (71) ^c	trace	trace	<5
2	CuI	-	10.0	H ₂ O	54	trace	trace	<5
3	-	AgOAc	10.0	H ₂ O	-	-	-	-
4	CuI	AgOAc	-	H ₂ O	-	-	-	-
5	CuI	AgOAc	10.0	-	64	15	8	<5
6 ^d	CuI	AgOAc	10.0	H ₂ O	68	trace	8	<5
7 ^e	CuI	TBHP	10.0	-	73	trace	trace	<5

^aReaction conditions: **1a** (0.25 mmol), CuI (10 mol%), Ag(OAc)₂ (9 mol%), TFA (10.0 equiv), DMSO (1 mL), H₂O (50 uL), under O₂ at 90 °C for 60 hours. ^bThe yields were determined by ¹H NMR using dibromomethane as the internal standard. ^cIsolated yield. ^dUnder air. ^eTBHP (2.2 equiv) instead of AgOAc and O₂.

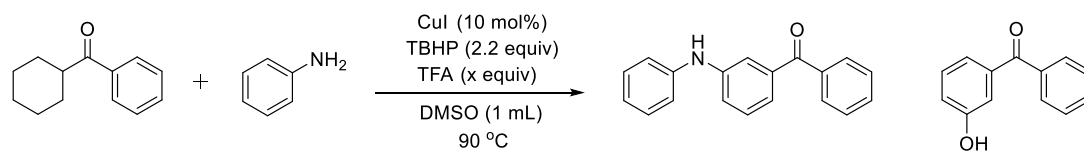
2.2 Condition optimization for synthesis of *meta*-functionalized phenols

Table S9. Screening of the ratio of aniline to TFA^{a,b}

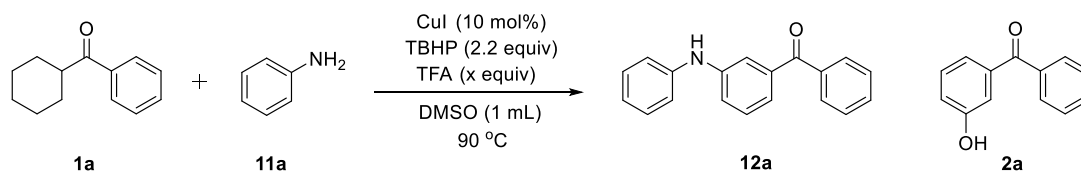
				
Entry	Aniline (equiv)	TFA (equiv)	12a (%)	2a (%)
1	1	1.0	14	5
2	1	2.0	21	9
3	1	3.0	39	12
4	1	4.0	43	15
5	1	5.0	48	13
6	1	6.0	45	10
7	1	8.0	31	<5
8	1.5	5.0	trace	-
9	5	10.0	nr	-
10	4	10.0	nr	-

^aReaction conditions: Reaction conditions: **1a** (0.25 mmol), **11a** (y equiv), CuI (10 mol%), Ag(OAc)₂ (9 mol%), TFA (x equiv), DMSO (1 mL), under O₂ at 90 °C for 60 hours. ^bThe yields were determined by ¹H NMR using dibromomethane as the internal standard.

Table S10. Screening of the ratio of aniline to TFA under silver-free conditions^{a,b}

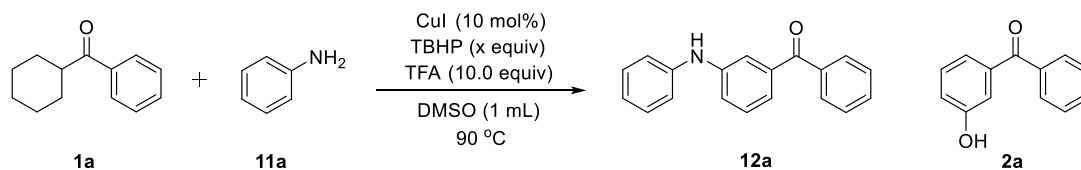
				
Entry	Aniline (equiv)	TFA (equiv)	12a (%)	2a (%)
1	1	10	34	9
2	1.5	10	59	<5
3	2	10	78 (73) ^c	trace
4	3	10	62	trace
5	4	10	60	trace
6	5	10	51	trace
7	1.5	5	53	trace
8	2	5	61	trace
9	2.5	5	36	trace
10	3	5	22	trace

^aReaction conditions: **1a** (0.25 mmol), **11a** (y equiv), CuI (10 mol%), TBHP (2.2 equiv), TFA (x equiv), DMSO (1 mL), at 90 °C for 60 hours. ^bThe yields were determined by ¹H NMR using dibromomethane as the internal standard. ^cIsolated yield.

Table S11. Screening of the TFA loading^{a,b}

Entry	Aniline (equiv)	TFA (equiv)	12a (%)	2a (%)
1	2	2	trace	-
2	2	4	58	trace
3	2	6	62	trace
4	2	7	65	trace
5	2	8	67	trace
6	2	9	71	trace
7	2	10	78 (73) ^c	trace

^aReaction conditions: **1a** (0.25 mmol), **11a** (0.5 mmol), CuI (10 mol%), TBHP (2.2 equiv), TFA (x equiv), DMSO (1 mL), at 90 °C for 60 hours. ^bThe yields were determined by ¹H NMR using dibromomethane as the internal standard. ^cIsolated yield.

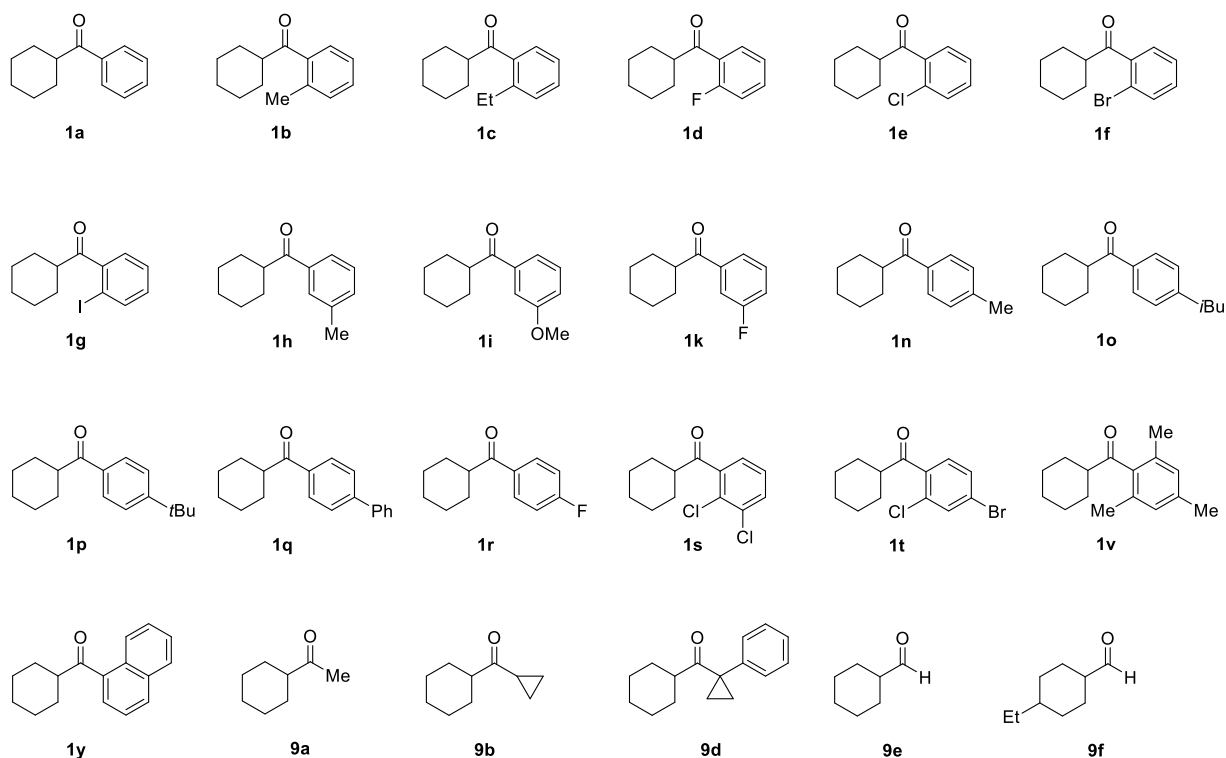
Table S12. Screening of the TBHP loading^{a,b}

Entry	Aniline (equiv)	TBHP (equiv)	12a (%)	2a (%)
1	2	2.2	78 (73) ^c	trace
2	2	1.65	67	trace
3	2	1.1	62	trace
4	2	0.55	57	trace

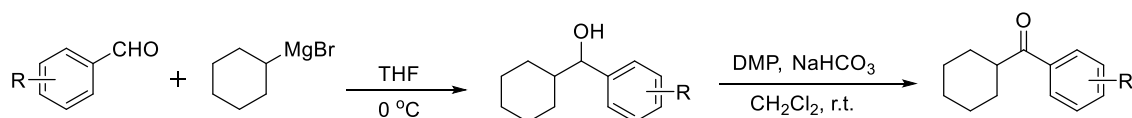
^aReaction conditions: **1a** (0.25 mmol), **11a** (0.5 mmol), CuI (10 mol%), TBHP (x equiv), TFA (10.0 equiv), DMSO (1 mL), at 90 °C for 60 hours. ^bThe yields were determined by ¹H NMR using dibromomethane as the internal standard. ^cIsolated yield.

3. Procedure for the Synthesis of Starting Materials and Products

3.1 Procedure for the synthesis of starting materials



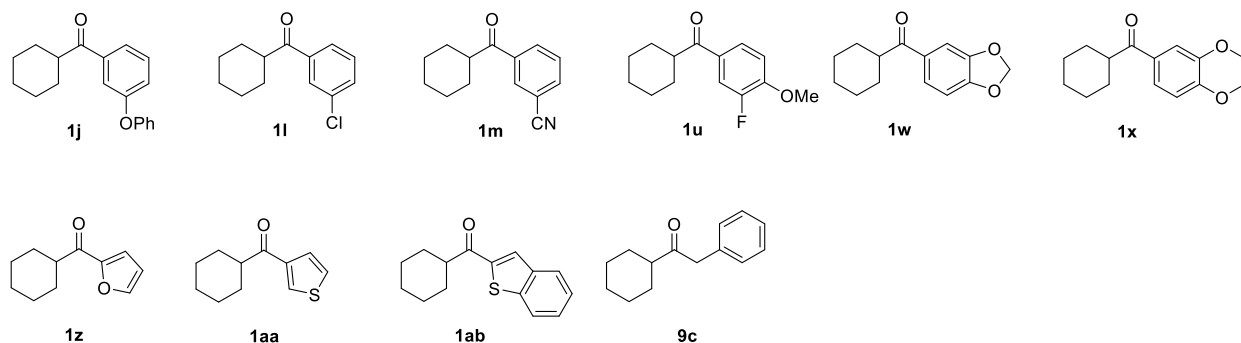
1a, 9a, 9e are commercially available. **1b-1i, 1k, 1n-1t, 1v, 1y, 9b, 9d** were prepared according to reported procedure¹. **1b**², **1c**³, **1d**⁴, **1e-1g**², **1h**⁵, **1i**⁶, **1k**⁷, **1n**², **1p**⁸, **1q**⁵, **1r**², **1v**⁹, **1y**¹⁰, **9b**⁷, **9d**¹¹, **9f**¹² are known compounds and their characterization data were consistent with these reported in the literature. Other ketones were prepared by general procedure A.



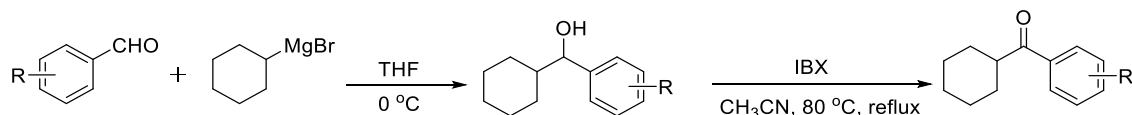
1st Step: To a solution of the corresponding aldehyde (15 mmol, 1.0 equiv) in anhydrous THF (25 mL) at 0 °C was added cyclohexylmagnesium bromide (1.0 M in THF, 19.5 mmol, 19.5 mL, 1.3 equiv) dropwise under argon. Upon completion, the mixture was quenched with saturated NH₄Cl (20 mL), extracted with EtOAc (3 × 50 mL). The combined extracts were washed with brine, dried over anhydrous Na₂SO₄, and concentrated to give the residue, which was further purified by silica gel column chromatography to afford the desired corresponding alcohols.

2nd Step¹: To a solution of the preceding alcohols in dry DCM (30 mL) were added solid NaHCO₃ (2 equiv) and Dess-Martin periodinane (DMP, 1.5 equiv). The solution was stirred at room temperature and consumption of starting material was monitored by TLC. Upon completion, the reaction was quenched by adding saturated NaHCO₃ (30 mL) and saturated Na₂S₂O₃ (30 mL) and stirred for 2 h. The mixture was extracted with CH₂Cl₂ (3 × 60 mL). The

combined organic extracts were dried with Na₂SO₄, and concentrated to give the residue, which was further purified by silica gel column chromatography to afford the desired ketones.

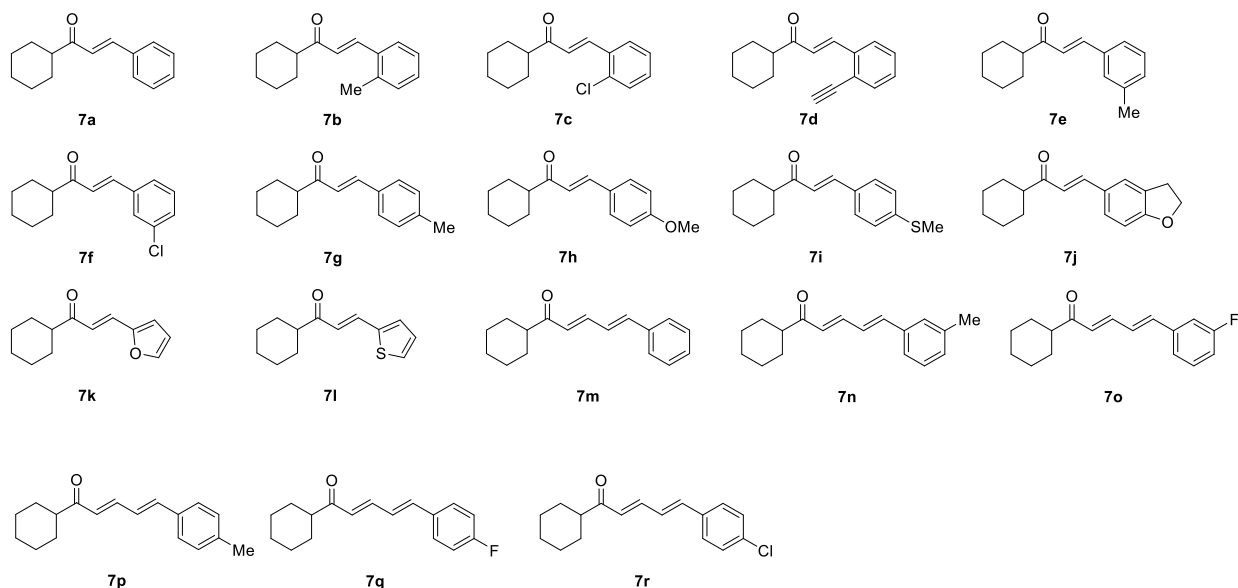


1j, 1l, 1m, 1u, 1w, 1x, 1z, 1aa, 1ab, 9c were prepared according to reported procedure¹³. **1l**¹⁴, **1w**⁷, **1z**⁷, **1aa**⁶, **1ab**¹⁵, **9c**¹⁶ are known compounds and their characterization data were consistent with these reported in the literature. Other ketones were prepared by general procedure B.

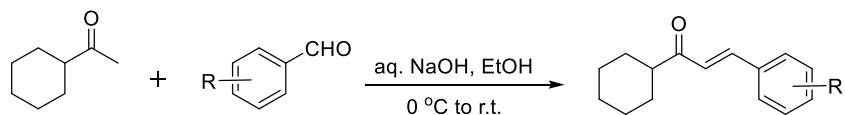


1st Step: To a solution of the corresponding aldehyde (15 mmol, 1.0 equiv) in anhydrous THF (25 mL) at 0 °C was added cyclohexylmagnesium bromide (1.0 M in THF, 19.5 mmol, 19.5 mL, 1.3 equiv) dropwise under argon. Upon completion, the mixture was quenched with saturated NH₄Cl (20 mL), extracted with EtOAc (3 × 50 mL). The combined extracts were washed with brine, dried over anhydrous Na₂SO₄, and concentrated to give the residue, which was further purified by silica gel column chromatography to afford the desired corresponding alcohols.

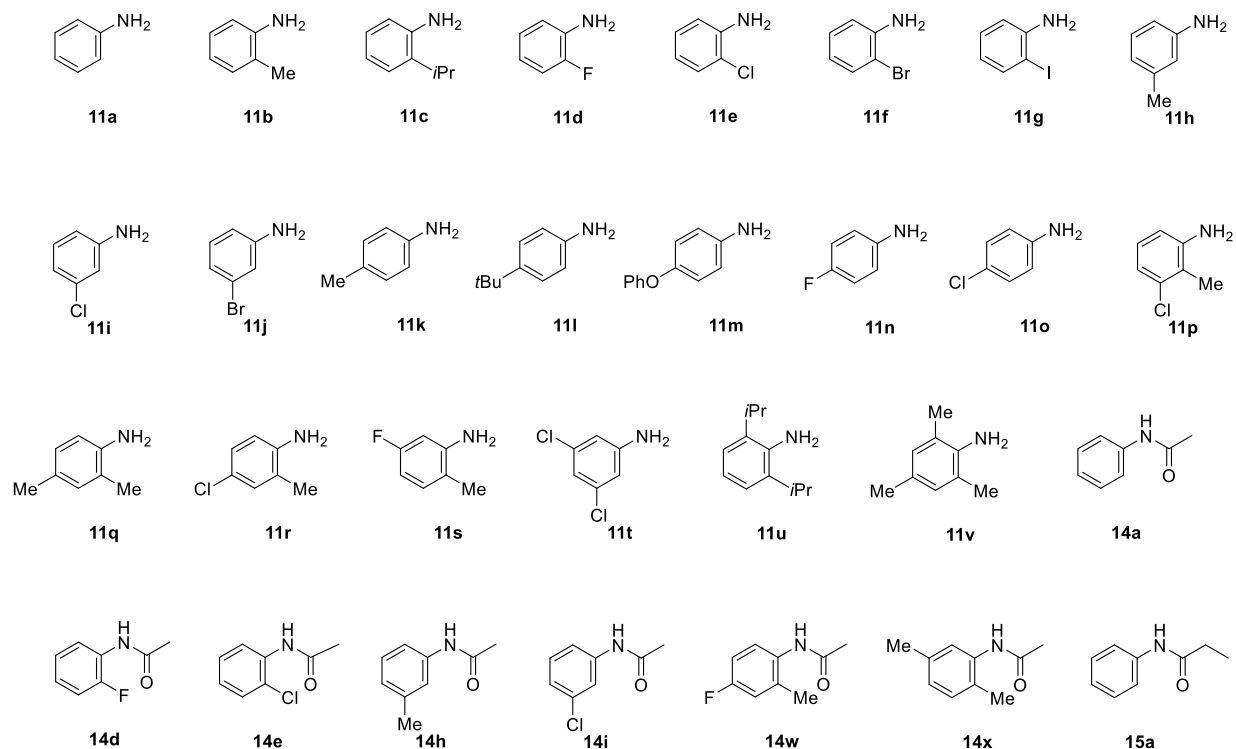
2nd Step¹³: To a solution of the preceding alcohols in CH₃CN (0.25 M) were added 2-Iodoxybenzoic acid (IBX, 2.0 equiv), then the mixture was refluxed and stirred for 1 h. Upon completion, the reaction mixture was cooled to room temperature. The mixture was filtered through a short path of silica gel and washed with EtOAc. Then the solution was concentrated by evaporation to give the residue, which was further purified by silica gel column chromatography to afford the desired ketones.



7a-7r were prepared according to reported procedure¹⁷. **7a**⁵, **7b**¹⁸, **7e**¹⁸, **7g**¹⁸, **7h**¹⁹, **7l**¹⁸ are known compounds and their characterization data were consistent with these reported in the literature. Other ketones were prepared by general procedure C.



To a solution of 1-cyclohexylethan-1-one (554 mg, 4.4 mmol, 1.1 equiv) in EtOH (3 mL) at 0 °C was added dropwise a solution of NaOH (320 mg, 8 mmol, 2.0 equiv) in water (2 mL) over 15 min. The resulting solution was stirred for 30 min at 0 °C before the corresponding aldehyde (4 mmol, 1.0 equiv) in EtOH (1 mL) was added dropwise. The reaction mixture was warmed to room temperature gradually and stirred overnight. The solid product was collected by suction filtration on a Buchner funnel and washed repeatedly with cold ethanol. Recrystallization from ethanol or purification by silica gel chromatography for liquid products.



11a-11v, 14a, 14d, 14e, 14h, 14i, 14w, 15a are commercially available. **14x** was prepared according to reported procedure²⁰. **14x**²¹ is known compounds and their characterization data were consistent with these reported in the literature.

Note: the NMR spectroscopy of new compounds **1j, 1m, 1o, 1s, 1t, 1u, 1x, 7c, 7d, 7f, 7i, 7j, 7k, 7m, 7n, 7o, 7p, 7q, 7r** were offered.

3.2 General procedure for the synthesis of *meta*-functionalized phenols

General procedure D: A 15 mL sealed tube containing a magnetic stir bar was charged with ketones (0.25 mmol), CuI (4.8 mg, 10 mol%), AgOAc (3.7 mg, 9 mol%) and DMSO (1 mL) sequentially. The tube was evacuated and backfilled with O₂ three times. Subsequently, H₂O (50 uL, 11.1 equiv) and TFA (186 uL, 10.0 equiv) was added. The tube was sealed and the mixture was stirred at 90 °C for 60 h. After cooling to room temperature, the reaction mixture was diluted with EtOAc (50 mL) and washed with H₂O (3 × 1 mL) and brine (3 × 1 mL). The combined organic layer was dried over anhydrous Na₂SO₄, and concentrated to give the residue, which was further purified by silica gel column chromatography to afford the desired product.

Procedure for the synthesis of **10a**

A 15 mL sealed tube containing a magnetic stir bar was charged with **9a** (0.25 mmol), CuI (4.8 mg, 10 mol%), Pd(OAc)₂ (8.4 mg, 15 mol%) and DMSO (1 mL) sequentially. The tube was evacuated and backfilled with O₂ three times. Subsequently, H₂O (50 uL, 11.1 equiv) and TFA (186 uL, 10.0 equiv) was added. The tube was sealed and the mixture was stirred at 90 °C for 60 h. After cooling to room temperature, the reaction mixture was diluted with EtOAc (50 mL) and

washed with H₂O (3 × 1 mL) and brine (3 × 1 mL). The combined organic layer was dried over anhydrous Na₂SO₄, and concentrated to give the residue, which was further purified by silica gel column chromatography to afford the desired product.

3.3 Gram-scale for the synthesis of *meta*-functionalized phenols

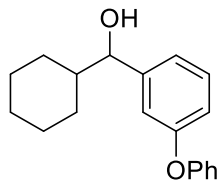
A 48 mL sealed tube containing a magnetic stir bar was charged with **1a** (5.5 mmol), CuI (105.6 mg, 10 mol%), AgOAc (81.4 mg, 9 mol%) and DMSO (22 mL) sequentially. The tube was evacuated and backfilled with O₂ three times. Subsequently, H₂O (1.1 mL, 11.1 equiv) and TFA (4.08 mL, 10.0 equiv) was added. The tube was sealed and the mixture was stirred at 90 °C for 72 h. After cooling to room temperature, the reaction mixture was diluted with EtOAc (800 mL) and washed with H₂O (3 × 15 mL) and brine (3 × 15 mL). The combined organic layer was dried over anhydrous Na₂SO₄, and concentrated to give the residue, which was further purified by silica gel column chromatography to afford the desired product.

3.4 General procedure for the synthesis of *meta*-functionalized anilines

General procedure E: A 15 mL sealed tube containing a magnetic stir bar was charged with ketones (0.25 mmol), anilines (0.5 mmol), CuI (4.8 mg, 10 mol%), DMSO (1 mL) and TFA (186 uL, 10.0 equiv) sequentially. Subsequently, TBHP (100 uL, 2.2 equiv) was added. The tube was sealed and the mixture was stirred at 90 °C for 60 h. After cooling to room temperature, the reaction mixture was diluted with EtOAc (50 mL) and washed with H₂O (3 × 1 mL) and brine (3 × 1 mL). The combined organic layer was dried over anhydrous Na₂SO₄, and concentrated to give the residue, which was further purified by silica gel column chromatography to afford the desired product.

4. Characterization Data of Starting Materials and Products

4.1 Characterization data of starting materials

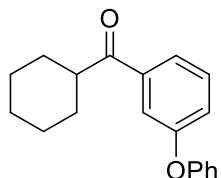


cyclohexyl(3-phenoxyphenyl)methanol (1j-1). Prepared according to general procedure B and purified by flash column chromatography to afford the product as a yellow liquid (2.83 g, 67% yield). Eluant: ethyl acetate/petroleum ether (1:8, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.36 – 7.29 (m, 2H), 7.29 – 7.24 (m, 1H), 7.12 – 7.06 (m, 1H), 7.05 – 7.00 (m, 2H), 7.00 – 6.96 (m, 2H), 6.89 (dd, J = 8.1, 2.5 Hz, 1H), 4.33 (d, J = 7.0 Hz, 1H), 1.98 – 1.86 (m, 2H), 1.79 – 1.72 (m, 1H), 1.71 – 1.52 (m, 3H), 1.44 – 1.36 (m, 1H), 1.27 – 0.87 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.4, 157.2, 145.9, 129.9 (2C), 129.6, 123.3, 121.6, 118.9 (2C), 117.8, 117.3, 79.1, 45.1, 29.4, 28.8, 26.5, 26.2, 26.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{22}\text{O}_2\text{Na}$ 305.1512; Found 305.1507.

IR: 3372, 2921, 2850, 1581, 1484, 1443, 1239, 1212, 1072, 1022, 748, 691 cm^{-1} .

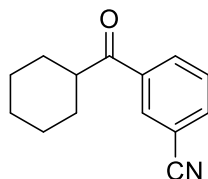


cyclohexyl(3-phenoxyphenyl)methanone (1j). Prepared according to general procedure B and purified by flash column chromatography to afford the product as a colorless liquid (2.48 g, 59% yield for two steps). Eluant: ethyl acetate/petroleum ether (1:25, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.69 – 7.64 (m, 1H), 7.57 (dd, J = 2.5, 1.7 Hz, 1H), 7.44 – 7.38 (m, 1H), 7.38 – 7.31 (m, 2H), 7.22 – 7.10 (m, 2H), 7.05 – 6.99 (m, 2H), 3.19 (tt, J = 11.5, 3.2 Hz, 1H), 1.93 – 1.79 (m, 4H), 1.76 – 1.68 (m, 1H), 1.54 – 1.19 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.3, 157.9, 156.8, 138.3, 130.0 (3C), 123.9, 123.1, 123.0, 119.2 (2C), 118.4, 45.9, 29.5 (2C), 26.0, 25.9 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{20}\text{O}_2\text{Na}$ 303.1356; Found 303.1362.

IR: 3065, 2929, 2853, 1680, 1579, 1488, 1434, 1253, 1230, 910, 751, 692 cm^{-1} .

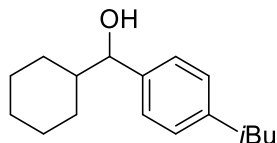


3-(cyclohexanecarbonyl)benzonitrile (1m). Prepared according to general procedure B and purified by flash column chromatography to afford the product as a yellow liquid (1.95 g, 61% yield for two steps). Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 8.23 – 8.20 (m, 1H), 8.19 – 8.14 (m, 1H), 7.86 – 7.81 (m, 1H), 7.65 – 7.59 (m, 1H), 3.22 (tt, J = 11.2, 3.1 Hz, 1H), 1.93 – 1.83 (m, 4H), 1.80 – 1.72 (m, 1H), 1.56 – 1.21 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.8, 137.2, 135.7, 132.4, 132.1, 129.8, 118.2, 113.2, 45.8, 29.3 (2C), 25.9, 25.8 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{15}\text{NONa}$ 236.1046; Found 236.1056.

IR: 3072, 2929, 2854, 2231, 1684, 1598, 1578, 1449, 1255, 1154, 983, 811, 753 cm^{-1} .

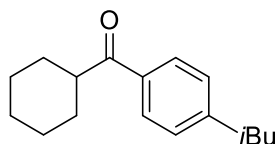


cyclohexyl(4-isobutylphenyl)methanol (1o-1). Prepared according to general procedure A and purified by flash column chromatography to afford the product as a white solid (2.62 g, 71% yield). m.p. = 52 – 54 °C. Eluant: ethyl acetate/petroleum ether (1:10, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.22 – 7.17 (m, 2H), 7.13 – 7.08 (m, 2H), 4.31 (d, J = 7.3 Hz, 1H), 2.46 (d, J = 7.1 Hz, 2H), 2.05 – 1.96 (m, 1H), 1.92 – 1.80 (m, 2H), 1.79 – 1.71 (m, 1H), 1.70 – 1.55 (m, 3H), 1.39 – 1.31 (m, 1H), 1.29 – 0.94 (m, 5H), 0.90 (d, J = 6.6 Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 141.04, 141.01, 129.1 (2C), 126.5 (2C), 79.5, 45.3, 45.0, 30.4, 29.5, 29.1, 26.6, 26.23, 26.15, 22.5 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{26}\text{ONa}$ 269.1876; Found 269.1881.

IR: 3380, 2952, 2923, 2851, 1464, 1450, 1015, 847 cm^{-1} .

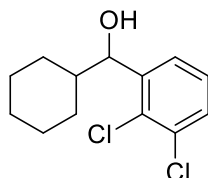


cyclohexyl(4-isobutylphenyl)methanone (1o). Prepared according to general procedure A and purified by flash column chromatography to afford the product as a colorless liquid (2.38 g, 65% yield for two steps). Eluant: ethyl acetate/petroleum ether (1:30, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.90 – 7.84 (m, 2H), 7.25 – 7.18 (m, 2H), 3.25 (tt, J = 11.5, 3.2 Hz, 1H), 2.52 (d, J = 7.2 Hz, 2H), 1.96 – 1.79 (m, 5H), 1.78 – 1.67 (m, 1H), 1.58 – 1.19 (m, 5H), 0.91 (d, J = 6.6 Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.7, 147.3, 134.2, 129.4 (2C), 128.4 (2C), 45.7, 45.5, 30.2, 29.6 (2C), 26.1, 26.0 (2C), 22.5 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{24}\text{ONa}$ 267.1719; Found 267.1722.

IR: 2929, 2854, 1678, 1606, 1464, 1449, 1414, 1251, 1172, 975 cm^{-1} .

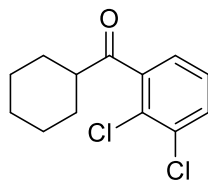


cyclohexyl(2,3-dichlorophenyl)methanol (1s-1). Prepared according to general procedure A and purified by flash column chromatography to afford the product as a white solid (2.67 g, 69% yield). m.p. = 68 – 70 °C. Eluant: ethyl acetate/petroleum ether (1:8, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.41 (dd, J = 7.8, 1.6 Hz, 1H), 7.37 (dd, J = 7.9, 1.7 Hz, 1H), 7.24 – 7.19 (m, 1H), 4.94 (d, J = 5.8 Hz, 1H), 2.03 (br, 1H), 1.83 – 1.70 (m, 3H), 1.69 – 1.57 (m, 2H), 1.51 – 1.43 (m, 1H), 1.27 – 1.06 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.7, 133.0, 130.6, 129.1, 127.3, 126.4, 75.5, 43.8, 29.7, 27.4, 26.5, 26.4, 26.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{OCl}_2\text{Na}$ 281.0470; Found 281.0456.

IR: 3370, 2924, 2850, 1448, 1419, 1177, 1154, 1043, 1016, 782, 739, 727 cm^{-1} .

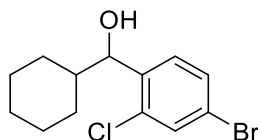


cyclohexyl(2,3-dichlorophenyl)methanone (1s). Prepared according to general procedure A and purified by flash column chromatography to afford the product as a white solid (2.38 g, 62% yield for two steps). m.p. = 51 – 53 °C. Eluant: ethyl acetate/petroleum ether (1:25, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.51 (dd, J = 8.0, 1.6 Hz, 1H), 7.29 – 7.25 (m, 1H), 7.17 (dd, J = 7.6, 1.6 Hz, 1H), 2.98 (tt, J = 11.3, 3.5 Hz, 1H), 1.97 – 1.88 (m, 2H), 1.85 – 1.76 (m, 2H), 1.73 – 1.64 (m, 1H), 1.52 – 1.38 (m, 2H), 1.36 – 1.17 (m, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 206.8, 142.4, 134.0, 131.6, 128.8, 127.7, 126.1, 50.3, 28.4 (2C), 25.9, 25.7 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{14}\text{OCl}_2\text{Na}$ 279.0314; Found 279.0322.

IR: 2927, 2853, 1700, 1448, 1408, 1248, 1180, 1107, 983, 796, 746, 734 cm^{-1} .

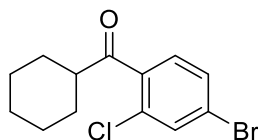


(4-bromo-2-chlorophenyl)(cyclohexyl)methanol (1t-1). Prepared according to general procedure A and purified by flash column chromatography to afford the product as a white solid (3.08 g, 68% yield). m.p. = 66 – 68 °C. Eluant: ethyl acetate/petroleum ether (1:10, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.48 (d, J = 1.9 Hz, 1H), 7.42 – 7.34 (m, 2H), 4.85 (d, J = 6.2 Hz, 1H), 2.00 (br, 1H), 1.88 – 1.68 (m, 3H), 1.68 – 1.57 (m, 2H), 1.47 – 1.39 (m, 1H), 1.23 – 1.03 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.4, 133.3, 131.9, 130.1, 129.6, 121.1, 74.6, 44.0, 29.5, 27.6, 26.44, 26.36, 26.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{OClBrNa}$ 324.9965; Found 324.9973.

IR: 3369, 2922, 2850, 1582, 1556, 1466, 1448, 1378, 1080, 1047, 1015, 819, 743 cm^{-1} .

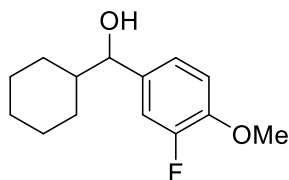


(4-bromo-2-chlorophenyl)(cyclohexyl)methanone (1t). Prepared according to general procedure A and purified by flash column chromatography to afford the product as a colorless liquid (2.70 g, 60% yield for two steps). Eluant: ethyl acetate/petroleum ether (1:50, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.58 (d, J = 1.8 Hz, 1H), 7.45 (dd, J = 8.2, 1.8 Hz, 1H), 7.21 (d, J = 8.2 Hz, 1H), 3.02 (tt, J = 11.2, 3.4 Hz, 1H), 1.96 – 1.85 (m, 2H), 1.84 – 1.75 (m, 2H), 1.72 – 1.62 (m, 1H), 1.51 – 1.37 (m, 2H), 1.36 – 1.17 (m, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 206.4, 138.7, 133.0, 131.8, 130.2, 129.7, 124.5, 50.0, 28.5 (2C), 25.9, 25.7 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{14}\text{OClBrNa}$ 322.9809; Found 322.9805.

IR: 2926, 2852, 1694, 1576, 1549, 1463, 1447, 1366, 1201, 1083, 1065, 816, 788, 759 cm^{-1} .

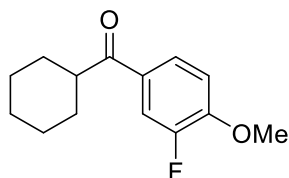


cyclohexyl(3-fluoro-4-methoxyphenyl)methanol (1u-1). Prepared according to general procedure B and purified by flash column chromatography to afford the product as a white solid (2.71 g, 76% yield). m.p. = 57 – 59 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.04 (dd, J = 12.3, 2.1 Hz, 1H), 7.00 – 6.95 (m, 1H), 6.94 – 6.87 (m, 1H), 4.29 (d, J = 7.1 Hz, 1H), 3.88 (s, 3H), 2.02 – 1.90 (m, 2H), 1.82 – 1.72 (m, 1H), 1.71 – 1.60 (m, 2H), 1.59 – 1.48 (m, 1H), 1.41 – 1.32 (m, 1H), 1.31 – 0.83 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 152.3 (C-F, $^1J_{\text{C-F}}$ = 245.7 Hz), 146.9 (C-F, $^2J_{\text{C-F}}$ = 10.8 Hz), 136.9 (C-F, $^3J_{\text{C-F}}$ = 5.4 Hz), 122.4 (C-F, $^3J_{\text{C-F}}$ = 3.6 Hz), 114.4 (C-F, $^2J_{\text{C-F}}$ = 18.4 Hz), 113.0 (C-F, $^4J_{\text{C-F}}$ = 2.1 Hz), 78.6, 56.4, 45.0, 29.3, 28.9, 26.5, 26.13, 26.05.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{19}\text{O}_2\text{FNa}$ 261.1261; Found 261.1252.

IR: 3376, 2922, 2850, 1622, 1586, 1511, 1443, 1267, 1220, 1118, 1025, 813, 752 cm^{-1} .

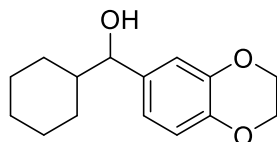


cyclohexyl(3-fluoro-4-methoxyphenyl)methanone (1u). Prepared according to general procedure B and purified by flash column chromatography to afford the product as a white solid (2.45 g, 69% yield for two steps). m.p. = 79 – 81 °C. Eluant: ethyl acetate/petroleum ether (1:15, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.78 – 7.72 (m, 1H), 7.69 (dd, J = 12.0, 2.1 Hz, 1H), 7.04 – 6.97 (m, 1H), 3.95 (s, 3H), 3.17 (tt, J = 11.4, 3.2 Hz, 1H), 1.91 – 1.80 (m, 4H), 1.78 – 1.69 (m, 1H), 1.56 – 1.19 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.6 (C-F, $^4J_{\text{C-F}}$ = 1.8 Hz), 152.2 (C-F, $^1J_{\text{C-F}}$ = 248.4 Hz), 151.7 (C-F, $^2J_{\text{C-F}}$ = 11.0 Hz), 129.6 (C-F, $^3J_{\text{C-F}}$ = 4.7 Hz), 125.5 (C-F, $^3J_{\text{C-F}}$ = 3.3 Hz), 116.1 (C-F, $^2J_{\text{C-F}}$ = 18.8 Hz), 112.4 (C-F, $^4J_{\text{C-F}}$ = 1.9 Hz), 56.4, 45.5, 29.6 (2C), 26.03, 25.95 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{17}\text{O}_2\text{FNa}$ 259.1105; Found 259.1117.

IR: 2929, 2853, 1672, 1609, 1580, 1514, 1430, 1314, 1267, 1114, 817, 761 cm^{-1} .

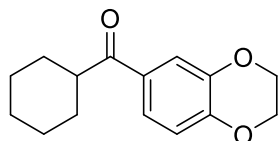


cyclohexyl(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)methanol (1x-1). Prepared according to general procedure B and purified by flash column chromatography to afford the product as a colorless liquid (2.86 g, 77% yield). Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 6.83 – 6.78 (m, 2H), 6.75 (dd, J = 8.3, 2.0 Hz, 1H), 4.27 – 4.20 (m, 5H), 2.03 – 1.94 (m, 1H), 1.87 (br, 1H), 1.80 – 1.71 (m, 1H), 1.70 – 1.59 (m, 2H), 1.59 – 1.49 (m, 1H), 1.41 – 1.33 (m, 1H), 1.30 – 0.82 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.4, 142.9, 137.3, 119.8, 117.0, 115.6, 79.1, 64.50, 64.46, 45.0, 29.4, 29.1, 26.6, 26.2, 26.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{15}\text{H}_{20}\text{O}_3\text{Na}$ 271.1305; Found 271.1299.

IR: 3400, 2921, 2850, 1590, 1503, 1449, 1432, 1281, 1255, 1100, 816, 736 cm^{-1} .

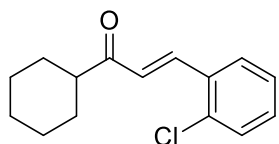


cyclohexyl(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)methanone (1x). Prepared according to general procedure B and purified by flash column chromatography to afford the product as a colorless liquid (2.58 g, 70% yield for two steps). Eluant: ethyl acetate/petroleum ether (1:10, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.53 – 7.46 (m, 2H), 6.93 – 6.88 (m, 1H), 4.34 – 4.25 (m, 4H), 3.17 (tt, J = 11.5, 3.2 Hz, 1H), 1.90 – 1.79 (m, 4H), 1.77 – 1.68 (m, 1H), 1.55 – 1.19 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 202.4, 147.8, 143.4, 130.2, 122.4, 117.9, 117.3, 64.8, 64.2, 45.4, 29.7 (2C), 26.1, 26.0 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{15}\text{H}_{18}\text{O}_3\text{Na}$ 269.1148; Found 269.1154.

IR: 2926, 2852, 1668, 1603, 1579, 1504, 1449, 1427, 1282, 1256, 1064, 884, 775, 734 cm^{-1} .

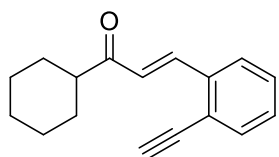


(*E*)-3-(2-chlorophenyl)-1-cyclohexylprop-2-en-1-one (7c). Prepared according to general procedure C and purified by flash column chromatography to afford the product as a white solid (0.91 g, 92% yield). m.p. = 62 – 64 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, J = 16.1 Hz, 1H), 7.65 (dd, J = 7.3, 2.2 Hz, 1H), 7.42 (dd, J = 7.4, 1.9 Hz, 1H), 7.36 – 7.24 (m, 2H), 6.76 (d, J = 16.1 Hz, 1H), 2.71 (tt, J = 11.2, 3.4 Hz, 1H), 1.96 – 1.88 (m, 2H), 1.88 – 1.79 (m, 2H), 1.76 – 1.65 (m, 1H), 1.52 – 1.17 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.1, 138.1, 135.4, 133.2, 131.1, 130.3, 127.7, 127.4, 127.2, 49.2, 28.9 (2C), 26.0, 25.8 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{15}\text{H}_{17}\text{OClNa}$ 271.0860; Found 271.0865.

IR: 2926, 2852, 1686, 1659, 1605, 1468, 1441, 1143, 1009, 976, 752 cm^{-1} .

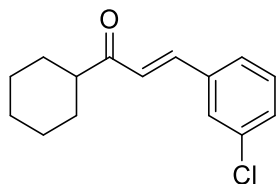


(*E*)-1-cyclohexyl-3-(2-ethynylphenyl)prop-2-en-1-one (7d). Prepared according to general procedure C and purified by flash column chromatography to afford the product as a yellow solid (0.84 g, 88% yield). m.p. = 65 – 67 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 8.09 (d, J = 16.2 Hz, 1H), 7.66 (dd, J = 7.6, 1.7 Hz, 1H), 7.54 (dd, J = 7.4, 1.7 Hz, 1H), 7.42 – 7.29 (m, 2H), 6.85 (d, J = 16.1 Hz, 1H), 3.44 (s, 1H), 2.71 (tt, J = 11.2, 3.4 Hz, 1H), 1.96 – 1.88 (m, 2H), 1.87 – 1.79 (m, 2H), 1.76 – 1.66 (m, 1H), 1.50 – 1.17 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.3, 139.8, 136.9, 133.6, 129.8, 129.2, 126.7, 126.1, 123.2, 83.5, 81.3, 49.3, 28.9 (2C), 26.0, 25.9 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{18}\text{ONa}$ 261.1250; Found 261.1245.

IR: 3296, 3241, 2926, 2851, 1683, 1656, 1606, 1592, 1474, 1447, 1318, 978, 754 cm^{-1} .

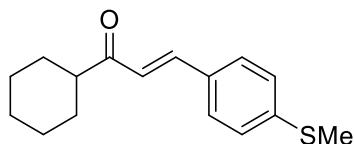


(E)-3-(3-chlorophenyl)-1-cyclohexylprop-2-en-1-one (7f). Prepared according to general procedure C and purified by flash column chromatography to afford the product as a white solid (0.90 g, 91% yield). m.p. = 52 – 54 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.59 – 7.48 (m, 2H), 7.42 (dt, J = 6.9, 1.7 Hz, 1H), 7.38 – 7.29 (m, 2H), 6.81 (d, J = 16.0 Hz, 1H), 2.63 (tt, J = 11.2, 3.4 Hz, 1H), 1.97 – 1.87 (m, 2H), 1.87 – 1.79 (m, 2H), 1.77 – 1.67 (m, 1H), 1.50 – 1.17 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 202.8, 140.7, 136.8, 135.0, 130.3, 130.2, 127.9, 126.7, 125.9, 49.8, 28.7 (2C), 26.0, 25.9 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{15}\text{H}_{17}\text{OClNa}$ 271.0860; Found 271.0866.

IR: 2925, 2852, 1686, 1657, 1609, 1563, 1197, 1144, 1010, 979, 781 cm^{-1} .

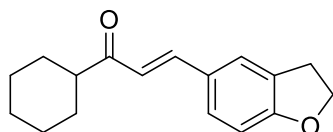


(E)-1-cyclohexyl-3-(4-(methylthio)phenyl)prop-2-en-1-one (7i). Prepared according to general procedure C and purified by flash column chromatography to afford the product as a yellow solid (0.90 g, 87% yield). m.p. = 79 – 81 °C. Eluant: ethyl acetate/petroleum ether (1:15, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.54 (d, J = 16.0 Hz, 1H), 7.49 – 7.44 (m, 2H), 7.25 – 7.20 (m, 2H), 6.77 (d, J = 16.0 Hz, 1H), 2.64 (tt, J = 11.3, 3.3 Hz, 1H), 2.50 (s, 3H), 1.94 – 1.86 (m, 2H), 1.86 – 1.78 (m, 2H), 1.75 – 1.67 (m, 1H), 1.49 – 1.16 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.2, 142.1, 141.8, 131.3, 128.7 (2C), 126.1 (2C), 123.8, 49.5, 28.9 (2C), 26.0, 25.9 (2C), 15.3.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{16}\text{H}_{20}\text{OSNa}$ 283.1127; Found 283.1134.

IR: 2924, 2853, 1678, 1600, 1589, 1493, 1404, 1320, 1082, 1014, 983, 810, 747 cm^{-1} .

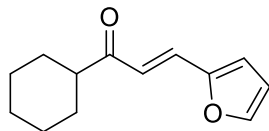


(E)-1-cyclohexyl-3-(2,3-dihydrobenzofuran-5-yl)prop-2-en-1-one (7j). Prepared according to general procedure C and purified by flash column chromatography to afford the product as a yellow solid (0.94 g, 92% yield). m.p. = 110 – 112 °C. Eluant: ethyl acetate/petroleum ether (1:15, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.55 (d, J = 16.0 Hz, 1H), 7.46 – 7.43 (m, 1H), 7.34 (dd, J = 8.4, 1.9 Hz, 1H), 6.78 (d, J = 8.2 Hz, 1H), 6.67 (d, J = 15.9 Hz, 1H), 4.62 (t, J = 8.7 Hz, 2H), 3.23 (t, J = 8.7 Hz, 2H), 2.63 (tt, J = 11.3, 3.3 Hz, 1H), 1.93 – 1.78 (m, 4H), 1.77 – 1.65 (m, 1H), 1.52 – 1.17 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.4, 162.5, 142.6, 130.0, 128.2, 127.6, 124.8, 122.0, 109.8, 72.0, 49.5, 29.4, 29.0 (2C), 26.0, 25.9 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{20}\text{O}_2\text{Na}$ 279.1356; Found 279.1361.

IR: 2927, 2853, 1678, 1649, 1586, 1490, 1442, 1241, 981, 815 cm^{-1} .

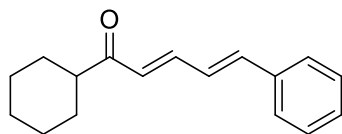


(E)-1-cyclohexyl-3-(furan-2-yl)prop-2-en-1-one (7k). Prepared according to general procedure C and purified by flash column chromatography to afford the product as a brown solid (0.63 g, 77% yield). m.p. = 56 – 58 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.48 (d, J = 1.4 Hz, 1H), 7.36 (d, J = 15.7 Hz, 1H), 6.73 (d, J = 15.7 Hz, 1H), 6.65 (d, J = 3.4 Hz, 1H), 6.48 (dd, J = 3.4, 1.8 Hz, 1H), 2.57 (tt, J = 11.3, 3.4 Hz, 1H), 1.98 – 1.86 (m, 2H), 1.86 – 1.78 (m, 2H), 1.74 – 1.66 (m, 1H), 1.49 – 1.16 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 202.9, 151.5, 144.8, 128.6, 122.1, 115.6, 112.6, 50.0, 28.8 (2C), 26.0, 25.9 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_2\text{Na}$ 227.1043; Found 227.1048.

IR: 2927, 2853, 1680, 1607, 1553, 1449, 1315, 1199, 1144, 1013, 973, 746 cm^{-1} .

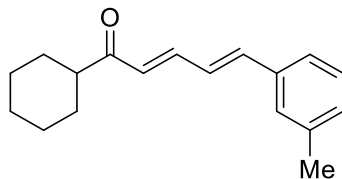


(2E,4E)-1-cyclohexyl-5-phenylpenta-2,4-dien-1-one (7m). Prepared according to general procedure C and purified by flash column chromatography to afford the product as a yellow solid (0.51 g, 53% yield). m.p. = 43 – 45 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.50 – 7.44 (m, 2H), 7.41 – 7.28 (m, 4H), 7.01 – 6.83 (m, 2H), 6.35 (d, J = 15.2 Hz, 1H), 2.59 (tt, J = 11.4, 3.3 Hz, 1H), 1.91 – 1.77 (m, 4H), 1.74 – 1.69 (m, 1H), 1.47 – 1.16 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.6, 142.4, 141.3, 136.3, 129.2, 129.0 (2C), 128.4, 127.3 (2C), 127.0, 49.3, 28.9 (2C), 26.0, 25.9 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{20}\text{ONa}$ 263.1406; Found 263.1401.

IR: 2925, 2851, 1677, 1650, 1614, 1584, 1447, 1120, 1064, 997, 751, 690 cm^{-1} .

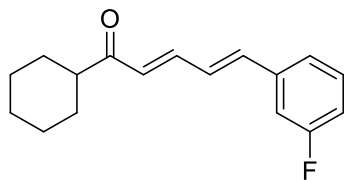


(2E,4E)-1-cyclohexyl-5-(m-tolyl)penta-2,4-dien-1-one (7n). Prepared according to general procedure C and purified by flash column chromatography to afford the product as a yellow solid (0.79 g, 78% yield). m.p. = 51 – 53 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.41 – 7.32 (m, 1H), 7.31 – 7.24 (m, 3H), 7.15 – 7.10 (m, 1H), 6.96 – 6.82 (m, 2H), 6.34 (d, J = 15.3 Hz, 1H), 2.59 (tt, J = 11.3, 3.3 Hz, 1H), 2.36 (s, 3H), 1.90 – 1.78 (m, 4H), 1.74 – 1.66 (m, 1H), 1.47 – 1.15 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.6, 142.5, 141.4, 138.6, 136.2, 130.1, 128.8, 128.2, 128.0, 126.8, 124.5, 49.3, 28.9 (2C), 26.0, 25.9 (2C), 21.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{18}\text{H}_{22}\text{ONa}$ 277.1563; Found 277.1556.

IR: 2924, 2852, 1701, 1604, 1585, 1449, 1245, 1141, 998, 779, 689 cm^{-1} .

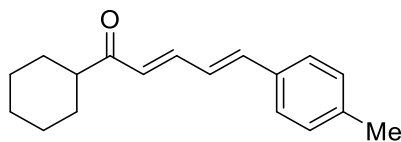


(2E,4E)-1-cyclohexyl-5-(3-fluorophenyl)penta-2,4-dien-1-one (7o). Prepared according to general procedure C and purified by flash column chromatography to afford the product as a yellow liquid (0.86 g, 83% yield). Eluant: ethyl acetate/petroleum ether (1:15, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.39 – 7.29 (m, 2H), 7.25 – 7.20 (m, 1H), 7.19 – 7.13 (m, 1H), 7.04 – 6.97 (m, 1H), 6.96 – 6.82 (m, 2H), 6.38 (d, J = 15.3 Hz, 1H), 2.58 (tt, J = 11.3, 3.3 Hz, 1H), 1.92 – 1.76 (m, 4H), 1.74 – 1.66 (m, 1H), 1.48 – 1.16 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.4, 163.2 (C-F, $^1J_{\text{C-F}}$ = 246.2 Hz), 141.7, 139.6 (C-F, $^4J_{\text{C-F}}$ = 2.9 Hz), 138.5 (C-F, $^3J_{\text{C-F}}$ = 7.7 Hz), 130.4 (C-F, $^3J_{\text{C-F}}$ = 8.4 Hz), 129.1, 128.2, 123.3 (C-F, $^4J_{\text{C-F}}$ = 2.8 Hz), 116.0 (C-F, $^2J_{\text{C-F}}$ = 21.6 Hz), 113.4 (C-F, $^2J_{\text{C-F}}$ = 22.0 Hz), 49.4, 28.8 (2C), 26.0, 25.9 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{17}\text{H}_{20}\text{OF}$ 259.1493; Found 259.1499.

IR: 2927, 2854, 1698, 1613, 1584, 1486, 1447, 1246, 1142, 966, 782, 750 cm^{-1} .

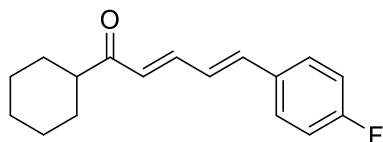


(2E,4E)-1-cyclohexyl-5-(p-tolyl)penta-2,4-dien-1-one (7p). Prepared according to general procedure C and purified by flash column chromatography to afford the product as a yellow solid (0.81 g, 80% yield). m.p. = 92 – 94 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.40 – 7.32 (m, 3H), 7.19 – 7.14 (m, 2H), 6.96 – 6.78 (m, 2H), 6.33 (d, J = 15.3 Hz, 1H), 2.58 (tt, J = 11.4, 3.3 Hz, 1H), 2.36 (s, 3H), 1.90 – 1.77 (m, 4H), 1.73 – 1.69 (m, 1H), 1.46 – 1.15 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.6, 142.7, 141.4, 139.5, 133.5, 129.7 (2C), 127.8, 127.3 (2C), 126.0, 49.3, 28.9 (2C), 26.0, 25.9 (2C), 21.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{18}\text{H}_{22}\text{ONa}$ 277.1563; Found 277.1553.

IR: 2923, 2851, 1673, 1582, 1446, 1140, 1063, 1000, 833, 802 cm^{-1} .

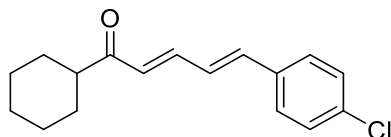


(2E,4E)-1-cyclohexyl-5-(4-fluorophenyl)penta-2,4-dien-1-one (7q). Prepared according to general procedure C and purified by flash column chromatography to afford the product as a yellow liquid (0.74 g, 72% yield). Eluant: ethyl acetate/petroleum ether (1:15, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.47 – 7.41 (m, 2H), 7.35 (dd, J = 15.2, 10.7 Hz, 1H), 7.09 – 7.01 (m, 2H), 6.94 – 6.75 (m, 2H), 6.35 (d, J = 15.3 Hz, 1H), 2.58 (tt, J = 11.4, 3.3 Hz, 1H), 1.95 – 1.78 (m, 4H), 1.74 – 1.69 (m, 1H), 1.47 – 1.16 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.5, 163.2 (C-F, $^1J_{\text{C-F}}$ = 249.9 Hz), 142.2, 139.8, 132.5 (C-F, $^4J_{\text{C-F}}$ = 3.5 Hz), 129.0 (C-F, $^3J_{\text{C-F}}$ = 8.2 Hz, 2C), 128.3, 126.7 (C-F, $^5J_{\text{C-F}}$ = 2.5 Hz), 116.0 (C-F, $^2J_{\text{C-F}}$ = 21.8 Hz, 2C), 49.4, 28.9 (2C), 26.0, 25.9 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{19}\text{OFNa}$ 281.1312; Found 281.1307.

IR: 2926, 2853, 1677, 1618, 1581, 1507, 1449, 1231, 1157, 1141, 997, 840 cm^{-1} .



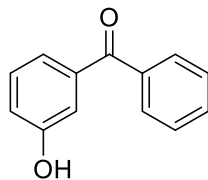
(2E,4E)-5-(4-chlorophenyl)-1-cyclohexylpenta-2,4-dien-1-one (7r). Prepared according to general procedure C and purified by flash column chromatography to afford the product as a yellow solid (0.94 g, 86% yield). m.p. = 124 – 126 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.42 – 7.28 (m, 5H), 6.93 – 6.78 (m, 2H), 6.36 (d, J = 15.3 Hz, 1H), 2.58 (tt, J = 11.4, 3.3 Hz, 1H), 1.90 – 1.77 (m, 4H), 1.75 – 1.69 (m, 1H), 1.48 – 1.14 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 203.4, 142.0, 139.7, 134.9, 134.7, 129.2 (2C), 128.7, 128.4 (2C), 127.5, 49.4, 28.8 (2C), 26.0, 25.9 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{19}\text{OClNa}$ 297.1017; Found 297.1026.

IR: 2923, 2852, 1675, 1583, 1490, 1448, 1239, 1141, 1087, 1062, 998, 837, 809 cm^{-1} .

4.2 Characterization data of products

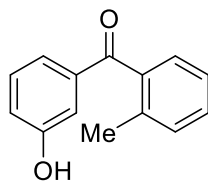


(3-hydroxyphenyl)(phenyl)methanone (2a). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (35.1 mg, 71% yield). m.p. = 108 – 110 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.76 (m, 2H), 7.63 – 7.55 (m, 1H), 7.50 – 7.43 (m, 2H), 7.42 – 7.38 (m, 1H), 7.36 – 7.27 (m, 2H), 7.13 – 7.08 (m, 1H), 6.42 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.4, 156.2, 138.9, 137.5, 132.8, 130.3 (2C), 129.7, 128.5 (2C), 123.0, 120.2, 116.7.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{10}\text{O}_2\text{Na}$ 221.0573; Found 221.0566.

IR: 3352, 3061, 2924, 1643, 1594, 1581, 1448, 1319, 1288, 1234, 843, 725, 707 cm^{-1} .

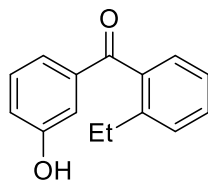


(3-hydroxyphenyl)(o-tolyl)methanone (2b). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (42.9 mg, 81% yield). m.p. = 99 – 101 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.3).

^1H NMR (400 MHz, CDCl_3) δ 7.43 – 7.36 (m, 2H), 7.34 – 7.21 (m, 5H), 7.11 – 7.06 (m, 1H), 5.77 (br, 1H), 2.33 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 199.3, 156.3, 139.2, 138.5, 137.0, 131.2, 130.6, 129.9, 128.8, 125.3, 123.3, 120.9, 116.4, 20.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{12}\text{O}_2\text{Na}$ 235.0730; Found 235.0722.

IR: 3329, 3062, 2925, 1646, 1595, 1583, 1479, 1448, 1296, 1229, 768, 742 cm^{-1} .

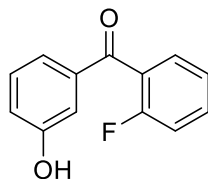


(2-ethylphenyl)(3-hydroxyphenyl)methanone (2c). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a light yellow solid (46.9 mg, 83% yield). m.p. = 48 – 50 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.47 – 7.43 (m, 1H), 7.41 (dd, J = 7.2, 1.7 Hz, 1H), 7.35 – 7.30 (m, 1H), 7.30 – 7.21 (m, 4H), 7.12 – 7.07 (m, 1H), 6.32 (br, 1H), 2.66 (q, J = 7.6 Hz, 2H), 1.15 (t, J = 7.6 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 199.4, 156.3, 143.2, 139.3, 138.3, 130.6, 129.8, 129.5, 128.5, 125.3, 123.4, 121.0, 116.4, 26.5, 16.0.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{15}\text{H}_{14}\text{O}_2\text{Na}$ 249.0886; Found 249.0882.

IR: 3328, 3063, 2967, 2932, 1646, 1594, 1582, 1479, 1447, 1291, 1224, 847, 750 cm^{-1} .

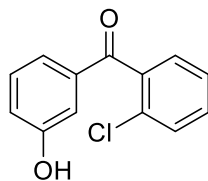


(2-fluorophenyl)(3-hydroxyphenyl)methanone (2d). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a light yellow solid (38.3 mg, 71% yield). m.p. = 110 – 112 °C. Eluant: ethyl acetate/petroleum ether (1:4, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.57 – 7.49 (m, 2H), 7.43 – 7.39 (m, 1H), 7.36 – 7.30 (m, 2H), 7.29 – 7.23 (m, 1H), 7.19 – 7.09 (m, 2H), 5.83 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 193.8, 160.3 (C-F, $^1J_{\text{C-F}}$ = 253.1 Hz), 156.1, 138.9, 133.3 (C-F, $^3J_{\text{C-F}}$ = 8.4 Hz), 130.9 (C-F, $^4J_{\text{C-F}}$ = 2.8 Hz), 129.9, 127.0 (C-F, $^2J_{\text{C-F}}$ = 14.5 Hz), 124.4 (C-F, $^3J_{\text{C-F}}$ = 3.7 Hz), 123.0, 121.1, 116.5 (C-F, $^2J_{\text{C-F}}$ = 21.6 Hz), 116.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_9\text{O}_2\text{FNa}$ 239.0479; Found 239.0471.

IR: 3354, 2925, 2853, 1651, 1610, 1597, 1584, 1482, 1451, 1301, 1222, 754 cm^{-1} .

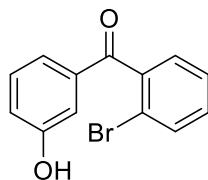


(2-chlorophenyl)(3-hydroxyphenyl)methanone (2e). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a light yellow solid (38.9 mg, 67% yield). m.p. = 112 – 114 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.49 – 7.41 (m, 2H), 7.40 – 7.35 (m, 3H), 7.34 – 7.28 (m, 2H), 7.13 – 7.08 (m, 1H), 5.44 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 195.5, 156.2, 138.5, 138.0, 131.5, 131.4, 130.3, 130.1, 129.3, 126.8, 123.3, 121.3, 116.2.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_9\text{O}_2\text{ClNa}$ 255.0183; Found 255.0173.

IR: 3352, 2924, 2853, 1655, 1586, 1449, 1434, 1294, 1226, 1058, 767, 748 cm^{-1} .

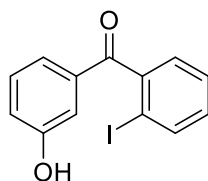


(2-bromophenyl)(3-hydroxyphenyl)methanone (2f). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a light yellow solid (49.5 mg, 72% yield). m.p. = 90 – 92 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.64 (d, J = 7.7 Hz, 1H), 7.44 – 7.38 (m, 2H), 7.38 – 7.27 (m, 4H), 7.13 – 7.09 (m, 1H), 5.75 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.3, 156.3, 140.5, 137.6, 133.4, 131.4, 130.1, 129.1, 127.3, 123.5, 121.5, 119.7, 116.3.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_9\text{O}_2\text{BrNa}$ 298.9678; Found 298.9687.

IR: 3357, 2926, 1654, 1585, 1449, 1294, 1225, 1048, 1026, 971, 746 cm^{-1} .

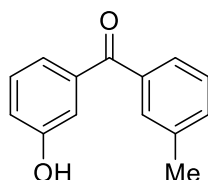


(3-hydroxyphenyl)(2-iodophenyl)methanone (2g). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a light yellow solid (58.9 mg, 73% yield). m.p. = 92 – 94 °C. Eluant: ethyl acetate/petroleum ether (1:8, R_f = 0.3).

^1H NMR (400 MHz, Acetone- d_6) δ 8.81 (br, 1H), 8.00 (dd, J = 7.9, 1.1 Hz, 1H), 7.61 – 7.55 (m, 1H), 7.41 – 7.35 (m, 2H), 7.34 – 7.28 (m, 1H), 7.27 – 7.24 (m, 1H), 7.23 – 7.18 (m, 1H), 7.18 – 7.13 (m, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 197.2, 158.6, 145.9, 140.3, 138.0, 132.0, 130.8, 129.0, 128.9, 122.5, 121.8, 117.0, 92.4.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_9\text{O}_2\text{INa}$ 346.9539; Found 346.9549.

IR: 3320, 2923, 2852, 1650, 1580, 1447, 1428, 1289, 1215, 1135, 1016, 970, 841, 742 cm^{-1} .

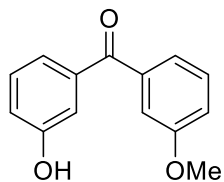


(3-hydroxyphenyl)(*m*-tolyl)methanone (2h). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a light yellow solid (41.3 mg, 78% yield). m.p. = 65 – 67 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.64 – 7.61 (m, 1H), 7.60 – 7.56 (m, 1H), 7.44 – 7.29 (m, 5H), 7.11 – 7.07 (m, 1H), 5.68 (br, 1H), 2.42 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.4, 156.1, 139.1, 138.3, 137.5, 133.6, 130.7, 129.6, 128.3, 127.6, 123.0, 120.0, 116.7, 21.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{12}\text{O}_2\text{Na}$ 235.0730; Found 235.0736.

IR: 3349, 2923, 2854, 1642, 1594, 1580, 1479, 1447, 1295, 1230, 1193, 784, 743 cm^{-1} .

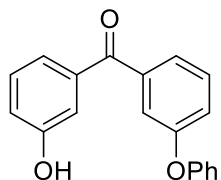


(3-hydroxyphenyl)(3-methoxyphenyl)methanone (2i). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (41.0 mg, 72% yield). m.p. = 96 – 98 °C. Eluant: ethyl acetate/petroleum ether (1:4, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.39 – 7.37 (m, 1H), 7.37 – 7.29 (m, 5H), 7.15 – 7.08 (m, 2H), 6.54 (br, 1H), 3.84 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.4, 159.6, 156.3, 138.8, 138.7, 129.6, 129.4, 123.2, 122.8, 120.3, 119.3, 116.8, 114.6, 55.6.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{12}\text{O}_3\text{Na}$ 251.0679; Found 251.0690.

IR: 3313, 2923, 2837, 1638, 1577, 1482, 1447, 1429, 1286, 1257, 1208, 1040, 994, 782, 747, 701 cm^{-1} .

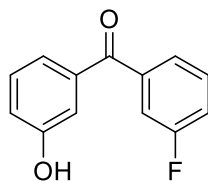


(3-hydroxyphenyl)(3-phenoxyphenyl)methanone (2j). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a light yellow solid (54.4 mg, 75% yield). m.p. = 94 – 96 °C. Eluant: ethyl acetate/petroleum ether (1:4, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.76 (br, 1H), 7.60 – 7.48 (m, 2H), 7.45 – 7.39 (m, 2H), 7.39 – 7.33 (m, 2H), 7.32 – 7.22 (m, 3H), 7.21 – 7.15 (m, 1H), 7.14 – 7.07 (m, 3H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 195.6, 158.4, 158.3, 157.5, 140.4, 139.6, 131.0 (2C), 130.9, 130.4, 125.4, 124.8, 123.2, 122.0, 120.6, 120.1 (2C), 119.8, 117.0.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{14}\text{O}_3\text{Na}$ 313.0835; Found 313.0827.

IR: 3329, 3064, 2926, 1642, 1577, 1487, 1436, 1288, 1250, 1216, 1161, 747 cm^{-1} .

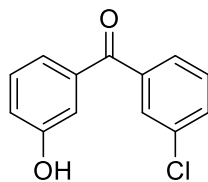


(3-fluorophenyl)(3-hydroxyphenyl)methanone (2k). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a light yellow solid (44.3 mg, 82% yield). m.p. = 110 – 112 °C. Eluant: ethyl acetate/petroleum ether (1:4, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.61 – 7.56 (m, 1H), 7.53 – 7.42 (m, 2H), 7.40 – 7.27 (m, 4H), 7.13 – 7.09 (m, 1H), 5.68 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 195.7 (C-F, $^4J_{\text{C-F}} = 2.0\text{ Hz}$), 162.6 (C-F, $^1J_{\text{C-F}} = 248.1\text{ Hz}$), 156.2, 139.5 (C-F, $^3J_{\text{C-F}} = 6.4\text{ Hz}$), 138.4, 130.2 (C-F, $^3J_{\text{C-F}} = 7.6\text{ Hz}$), 129.8, 126.0 (C-F, $^4J_{\text{C-F}} = 3.0\text{ Hz}$), 123.0, 120.5, 119.8 (C-F, $^2J_{\text{C-F}} = 21.4\text{ Hz}$), 117.0 (C-F, $^2J_{\text{C-F}} = 22.4\text{ Hz}$), 116.6.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_9\text{O}_2\text{FNa}$ 239.0479; Found 239.0484.

IR: 3368, 2925, 1649, 1583, 1480, 1445, 1303, 1254, 1192, 749 cm^{-1} .

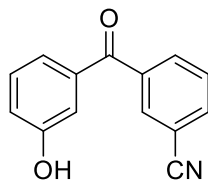


(3-chlorophenyl)(3-hydroxyphenyl)methanone (2l). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (51.0 mg, 88% yield). m.p. = 97 – 99 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.87 (br, 1H), 7.78 – 7.75 (m, 1H), 7.73 – 7.67 (m, 2H), 7.63 – 7.56 (m, 1H), 7.43 – 7.37 (m, 1H), 7.29 – 7.22 (m, 2H), 7.18 – 7.13 (m, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 195.0, 158.4, 140.6, 139.2, 134.8, 132.9, 131.0, 130.5, 130.0, 129.0, 122.1, 120.9, 117.0.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_9\text{O}_2\text{ClNa}$ 255.0183; Found 255.0191.

IR: 3330, 3065, 2926, 1644, 1581, 1567, 1473, 1446, 1418, 1287, 1227, 1165, 1077, 741 cm^{-1} .

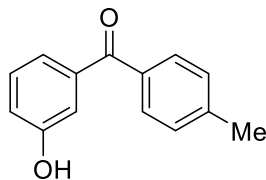


3-(3-hydroxybenzoyl)benzonitrile (2m). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a light yellow solid (42.4 mg, 76% yield). m.p. = 109 – 111 °C. Eluant: ethyl acetate/petroleum ether (1:4, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.88 (br, 1H), 8.15 – 8.11 (m, 1H), 8.10 – 8.03 (m, 2H), 7.84 – 7.76 (m, 1H), 7.45 – 7.38 (m, 1H), 7.31 – 7.22 (m, 2H), 7.20 – 7.14 (m, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 194.6, 158.5, 139.8, 138.8, 136.2, 134.6, 133.8, 130.7, 130.6, 122.2, 121.1, 118.7, 117.0, 113.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_9\text{NO}_2\text{Na}$ 246.0525; Found 246.0519.

IR: 3366, 3069, 2925, 1655, 1595, 1580, 1476, 1448, 1424, 1294, 1224, 1182, 1018, 745 cm^{-1} .

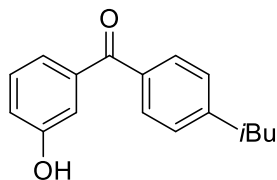


(3-hydroxyphenyl)(p-tolyl)methanone (2n). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (33.4 mg, 63% yield). m.p. = 110 – 113 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.72 (d, J = 7.8 Hz, 2H), 7.41 – 7.34 (m, 1H), 7.33 – 7.25 (m, 4H), 7.12 – 7.05 (m, 1H), 6.05 (br, 1H), 2.44 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.9, 156.1, 143.7, 139.4, 134.8, 130.6 (2C), 129.6, 129.2 (2C), 122.8, 119.8, 116.6, 21.8.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{12}\text{O}_2\text{Na}$ 235.0730; Found 235.0725.

IR: 3320, 2923, 2853, 1638, 1594, 1581, 1446, 1313, 1288, 1225, 1181, 749 cm^{-1} .

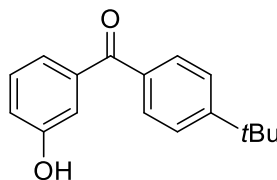


(3-hydroxyphenyl)(4-isobutylphenyl)methanone (2o). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (38.7 mg, 61% yield). m.p. = 70 – 72 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.77 – 7.71 (m, 2H), 7.38 – 7.29 (m, 3H), 7.26 – 7.20 (m, 2H), 7.12 – 7.06 (m, 1H), 5.79 (br, 1H), 2.56 (d, J = 7.2 Hz, 2H), 1.99 – 1.86 (m, 1H), 0.93 (d, J = 6.6 Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.2, 156.2, 147.5, 139.3, 135.0, 130.5 (2C), 129.6, 129.2 (2C), 122.8, 119.9, 116.7, 45.6, 30.3, 22.5 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{18}\text{O}_2\text{Na}$ 277.1199; Found 277.1205.

IR: 3349, 2955, 2925, 1640, 1595, 1582, 1447, 1313, 1288, 1237, 1182, 753 cm^{-1} .

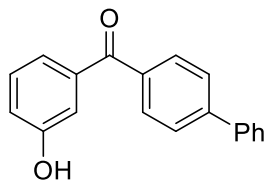


(4-(*tert*-butyl)phenyl)(3-hydroxyphenyl)methanone (2p). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (36.8 mg, 58% yield). m.p. = 98 – 100 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.82 – 7.74 (m, 2H), 7.53 – 7.46 (m, 2H), 7.39 – 7.30 (m, 3H), 7.12 – 7.06 (m, 1H), 5.70 (br, 1H), 1.36 (s, 9H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.0, 156.7, 156.2, 139.3, 134.7, 130.4 (2C), 129.6, 125.4 (2C), 122.9, 119.9, 116.7, 35.3, 31.3 (3C).

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{17}\text{H}_{19}\text{O}_2$ 255.1380; Found 255.1375.

IR: 3322, 2958, 2924, 1638, 1594, 1581, 1446, 1316, 1287, 1105, 973, 766, 716 cm^{-1} .

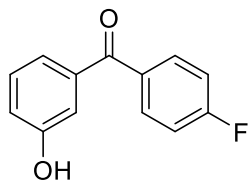


[1,1'-biphenyl]-4-yl(3-hydroxyphenyl)methanone (2q). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (34.9 mg, 51% yield). m.p. = 156 – 158 °C. Eluant: ethyl acetate/petroleum ether (1:4, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.77 (br, 1H), 7.93 – 7.82 (m, 4H), 7.81 – 7.74 (m, 2H), 7.56 – 7.49 (m, 2H), 7.48 – 7.36 (m, 2H), 7.33 – 7.24 (m, 2H), 7.17 – 7.12 (m, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 196.0, 158.3, 145.7, 140.6, 140.1, 137.4, 131.3 (2C), 130.4, 129.9 (2C), 129.1, 128.0 (2C), 127.6 (2C), 122.0, 120.4, 117.0.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{14}\text{O}_2\text{Na}$ 297.0886; Found 297.0874.

IR: 3343, 2954, 2924, 1639, 1595, 1580, 1447, 1403, 1315, 1294, 1236, 746 cm^{-1} .

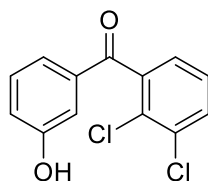


(4-fluorophenyl)(3-hydroxyphenyl)methanone (2r). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a light yellow solid (41.0 mg, 76% yield). m.p. = 102 – 104 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.3).

^1H NMR (400 MHz, CDCl_3) δ 7.90 – 7.81 (m, 2H), 7.39 – 7.31 (m, 2H), 7.30 – 7.26 (m, 1H), 7.20 – 7.13 (m, 2H), 7.12 – 7.07 (m, 1H), 5.88 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.0, 165.7 (C-F, $^1J_{\text{C-F}}$ = 254.9 Hz), 156.3, 138.8, 133.6 (C-F, $^4J_{\text{C-F}}$ = 3.0 Hz), 133.0 (C-F, $^3J_{\text{C-F}}$ = 9.2 Hz, 2C), 129.7, 122.7, 120.3, 116.6, 115.7 (C-F, $^2J_{\text{C-F}}$ = 21.8 Hz, 2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_9\text{O}_2\text{FNa}$ 239.0479; Found 239.0485.

IR: 3350, 2954, 2924, 1643, 1595, 1504, 1446, 1409, 1306, 1286, 1233, 1156, 872, 758 cm^{-1} .

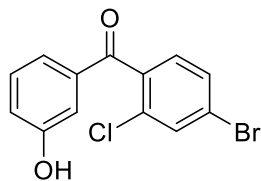


(2,3-dichlorophenyl)(3-hydroxyphenyl)methanone (2s). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (57.0 mg, 86% yield). m.p. = 154 – 156 °C. Eluant: ethyl acetate/petroleum ether (1:4, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.84 (br, 1H), 7.77 (dd, J = 8.0, 1.5 Hz, 1H), 7.57 – 7.51 (m, 1H), 7.43 (dd, J = 7.6, 1.6 Hz, 1H), 7.41 – 7.35 (m, 1H), 7.29 – 7.26 (m, 1H), 7.25 – 7.21 (m, 1H), 7.19 – 7.15 (m, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 194.0, 158.8, 142.2, 138.3, 133.9, 132.5, 131.0, 129.33, 129.30, 128.0, 122.24, 122.22, 116.6.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_8\text{O}_2\text{Cl}_2\text{Na}$ 288.9794; Found 288.9785.

IR: 3364, 2926, 2853, 1655, 1596, 1583, 1448, 1410, 1291, 1227, 1194, 1148, 760, 745 cm^{-1} .

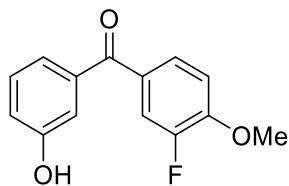


(4-bromo-2-chlorophenyl)(3-hydroxyphenyl)methanone (2t). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (58.7 mg, 76% yield). m.p. = 129 – 131 °C. Eluant: ethyl acetate/petroleum ether (1:4, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.83 (br, 1H), 7.81 (d, J = 1.8 Hz, 1H), 7.72 (dd, J = 8.2, 1.8 Hz, 1H), 7.44 (d, J = 8.1 Hz, 1H), 7.41 – 7.35 (m, 1H), 7.28 – 7.25 (m, 1H), 7.25 – 7.21 (m, 1H), 7.19 – 7.15 (m, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 194.2, 158.7, 139.0, 138.4, 133.2, 132.5, 131.3, 131.2, 131.0, 124.6, 122.2, 122.1, 116.6.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_8\text{O}_2\text{ClBrNa}$ 332.9288; Found 332.9276.

IR: 3356, 2925, 2852, 1655, 1597, 1579, 1449, 1369, 1291, 1225, 1135, 1083, 851, 756 cm^{-1} .

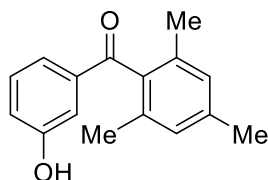


(3-fluoro-4-methoxyphenyl)(3-hydroxyphenyl)methanone (2u). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (44.3 mg, 72% yield). m.p. = 178 – 180 °C. Eluant: ethyl acetate/petroleum ether (1:3, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.75 (br, 1H), 7.66 – 7.56 (m, 2H), 7.42 – 7.34 (m, 1H), 7.33 – 7.25 (m, 1H), 7.24 – 7.16 (m, 2H), 7.15 – 7.07 (m, 1H), 4.01 (s, 3H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 194.2, 158.3, 152.5 (C-F, $^1J_{\text{C-F}}$ = 247.6 Hz), 152.4 (C-F, $^2J_{\text{C-F}}$ = 10.7 Hz), 140.0, 131.2 (C-F, $^3J_{\text{C-F}}$ = 5.1 Hz), 130.4, 128.6 (C-F, $^3J_{\text{C-F}}$ = 3.3 Hz), 121.7, 120.1, 117.7 (C-F, $^2J_{\text{C-F}}$ = 19.2 Hz), 116.8, 113.6 (C-F, $^4J_{\text{C-F}}$ = 1.7 Hz), 56.8.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{11}\text{O}_3\text{FNa}$ 269.0584; Found 269.0575.

IR: 3353, 2923, 2852, 1657, 1608, 1580, 1517, 1443, 1282, 1107, 1023, 754 cm^{-1} .

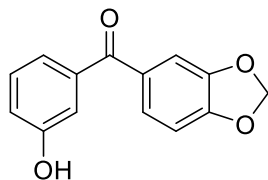


(3-hydroxyphenyl)(mesityl)methanone (2v). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (33.6 mg, 56% yield). m.p. = 122 – 124 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.40 – 7.35 (m, 1H), 7.33 – 7.27 (m, 2H), 7.10 – 7.04 (m, 1H), 6.89 (s, 2H), 5.67 (br, 1H), 2.32 (s, 3H), 2.08 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.0, 156.4, 139.0, 138.7, 136.9, 134.4 (2C), 130.3, 128.5 (2C), 122.5, 121.1, 115.5, 21.3, 19.5 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{16}\text{H}_{16}\text{O}_2\text{Na}$ 263.1043; Found 263.1033.

IR: 3321, 2953, 2923, 1649, 1610, 1595, 1583, 1478, 1448, 1286, 1170, 851, 833, 759 cm^{-1} .

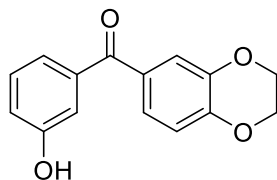


benzo[d][1,3]dioxol-5-yl(3-hydroxyphenyl)methanone (2w). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (47.8 mg, 79% yield). m.p. = 144 – 146 °C. Eluant: ethyl acetate/petroleum ether (1:3, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.70 (br, 1H), 7.40 – 7.33 (m, 2H), 7.29 (d, J = 1.7 Hz, 1H), 7.22 – 7.15 (m, 2H), 7.12 – 7.06 (m, 1H), 6.98 (d, J = 8.1 Hz, 1H), 6.15 (s, 2H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 194.7, 158.2, 152.5, 149.0, 140.5, 132.8, 130.2, 127.3, 121.6, 119.9, 116.8, 109.9, 108.4, 103.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{10}\text{O}_4\text{Na}$ 265.0471; Found 265.0466.

IR: 3300, 2923, 2853, 1637, 1580, 1503, 1485, 1440, 1354, 1293, 1255, 1037, 806, 754 cm^{-1} .

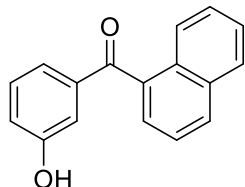


(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)(3-hydroxyphenyl)methanone (2x). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (49.3 mg, 77% yield). m.p. = 145 – 147 °C. Eluant: ethyl acetate/petroleum ether (1:3, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.72 (br, 1H), 7.42 – 7.28 (m, 3H), 7.25 – 7.15 (m, 2H), 7.13 – 7.06 (m, 1H), 6.96 (d, J = 8.1 Hz, 1H), 4.41 – 4.36 (m, 2H), 4.35 – 4.31 (m, 2H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 194.8, 158.1, 148.7, 144.2, 140.4, 131.7, 130.2, 124.8, 121.6, 119.83, 119.81, 117.7, 116.8, 65.6, 65.0.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{15}\text{H}_{12}\text{O}_4\text{Na}$ 279.0628; Found 279.0620.

IR: 3337, 2925, 2853, 1637, 1577, 1504, 1447, 1429, 1287, 1259, 1065, 886, 759 cm^{-1} .

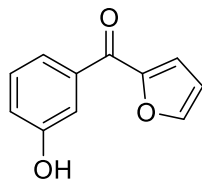


(3-hydroxyphenyl)(naphthalen-1-yl)methanone (2y). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow oil (47.7 mg, 77% yield). Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 8.09 – 8.03 (m, 1H), 8.00 – 7.94 (m, 1H), 7.91 – 7.85 (m, 1H), 7.58 – 7.41 (m, 5H), 7.31 – 7.20 (m, 2H), 7.10 – 7.05 (m, 1H), 6.58 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 198.8, 156.3, 139.6, 136.1, 133.8, 131.7, 131.0, 129.8, 128.6, 128.2, 127.5, 126.6, 125.7, 124.4, 123.5, 121.1, 116.7.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{12}\text{O}_2\text{Na}$ 271.0730; Found 271.0738.

IR: 3351, 3059, 2925, 1643, 1582, 1508, 1481, 1447, 1289, 1233, 1191, 793, 780, 748 cm^{-1} .

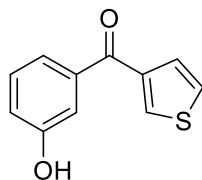


furan-2-yl(3-hydroxyphenyl)methanone (2z). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (33.8 mg, 72% yield). m.p. = 90 – 92 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.81 (br, 1H), 7.95 (d, J = 1.7 Hz, 1H), 7.49 – 7.45 (m, 1H), 7.44 – 7.42 (m, 1H), 7.42 – 7.36 (m, 1H), 7.32 (d, J = 3.6 Hz, 1H), 7.16 – 7.10 (m, 1H), 6.74 (dd, J = 3.6, 1.7 Hz, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 182.2, 158.3, 153.2, 148.4, 139.7, 130.5, 121.3, 121.1, 120.5, 116.4, 113.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{11}\text{H}_8\text{O}_3\text{Na}$ 211.0366; Found 211.0356.

IR: 3325, 2933, 2851, 1629, 1593, 1581, 1560, 1461, 1392, 1316, 1030, 819, 754 cm^{-1} .

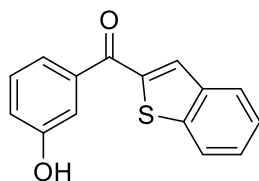


(3-hydroxyphenyl)(thiophen-3-yl)methanone (2aa). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (41.3 mg, 81% yield). m.p. = 158 – 160 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.73 (br, 1H), 8.13 (dd, J = 2.9, 1.3 Hz, 1H), 7.62 (dd, J = 5.1, 2.9 Hz, 1H), 7.56 (dd, J = 5.1, 1.3 Hz, 1H), 7.42 – 7.35 (m, 1H), 7.35 – 7.29 (m, 2H), 7.16 – 7.09 (m, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.7, 158.3, 142.1, 141.0, 134.8, 130.5, 129.1, 127.5, 121.4, 120.2, 116.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{11}\text{H}_8\text{O}_2\text{SNa}$ 227.0137; Found 227.0129.

IR: 3341, 2924, 2852, 1633, 1581, 1509, 1446, 1412, 1390, 1286, 1243, 745 cm^{-1} .

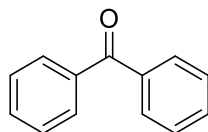


benzo[b]thiophen-2-yl(3-hydroxyphenyl)methanone (2ab). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (27.3 mg, 43% yield). m.p. = 169 – 171 °C. Eluant: ethyl acetate/petroleum ether (1:4, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.83 (br, 1H), 8.12 – 8.03 (m, 3H), 7.60 – 7.54 (m, 1H), 7.53 – 7.47 (m, 1H), 7.46 – 7.42 (m, 2H), 7.41 – 7.37 (m, 1H), 7.22 – 7.13 (m, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.6, 158.5, 143.9, 143.2, 140.3, 140.0, 133.3, 130.7, 128.5, 127.3, 126.1, 123.7, 121.3, 120.5, 116.4.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{15}\text{H}_{10}\text{O}_2\text{SNa}$ 277.0294; Found 277.0303.

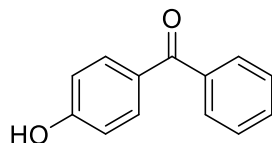
IR: 3350, 2923, 2852, 1626, 1591, 1580, 1510, 1445, 1427, 1298, 1181, 783, 754, 722 cm^{-1} .



benzophenone (5). ^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.76 (m, 4H), 7.64 – 7.56 (m, 2H), 7.54 – 7.44 (m, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.9, 137.7 (2C), 132.6 (2C), 130.2 (4C), 128.4 (4C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{10}\text{ONa}$ 205.0624; Found 205.0615.

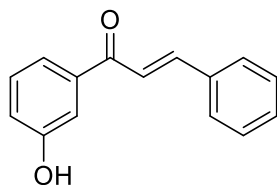
IR: 3060, 2927, 2853, 1657, 1598, 1577, 1447, 1317, 1276, 763, 698 cm^{-1} .



(4-hydroxyphenyl)(phenyl)methanone (6). ^1H NMR (400 MHz, CDCl_3) δ 8.23 (br, 1H), 7.81 – 7.72 (m, 4H), 7.61 – 7.53 (m, 1H), 7.51 – 7.43 (m, 2H), 7.00 – 6.93 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.5, 161.4, 138.0, 133.4 (2C), 132.5, 130.0 (2C), 129.3, 128.4 (2C), 115.6 (2C).

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{13}H_{10}O_2Na$ 221.0573; Found 221.0582.

IR: 3250, 2925, 2853, 1634, 1599, 1585, 1572, 1511, 1445, 1319, 1280, 1170, 743, 699 cm^{-1} .

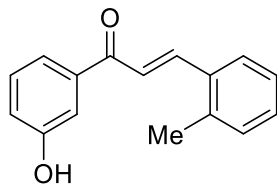


(E)-1-(3-hydroxyphenyl)-3-phenylprop-2-en-1-one (8a). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (29.7 mg, 53% yield). m.p. = 116 – 118 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

1H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 15.7 Hz, 1H), 7.71 – 7.65 (m, 1H), 7.65 – 7.59 (m, 2H), 7.57 (d, J = 7.7 Hz, 1H), 7.51 (d, J = 15.7 Hz, 1H), 7.45 – 7.33 (m, 4H), 7.18 – 7.11 (m, 1H), 6.99 (br, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.2, 156.7, 145.7, 139.6, 134.8, 130.9, 130.1, 129.1 (2C), 128.7 (2C), 122.1, 121.1, 120.8, 115.4.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{15}H_{12}O_2Na$ 247.0730; Found 247.0721.

IR: 3321, 2924, 2853, 1653, 1574, 1494, 1449, 1336, 1304, 1290, 1184, 760 cm^{-1} .

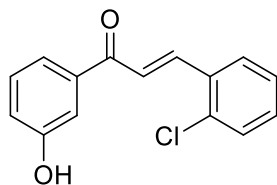


(E)-1-(3-hydroxyphenyl)-3-(o-tolyl)prop-2-en-1-one (8b). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (47.6 mg, 80% yield). m.p. = 95 – 97 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

1H NMR (400 MHz, Acetone- d_6) δ 8.80 (br, 1H), 8.08 (d, J = 15.5 Hz, 1H), 7.94 – 7.89 (m, 1H), 7.73 – 7.63 (m, 2H), 7.59 – 7.56 (m, 1H), 7.43 – 7.37 (m, 1H), 7.36 – 7.25 (m, 3H), 7.16 – 7.10 (m, 1H), 2.48 (s, 3H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.8, 158.7, 142.0, 140.6, 139.0, 134.7, 131.7, 131.1, 130.7, 127.5, 127.3, 123.9, 120.8, 120.7, 115.6, 19.8.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{16}H_{14}O_2Na$ 261.0886; Found 261.0889.

IR: 3285, 3057, 2925, 1649, 1568, 1481, 1445, 1320, 1283, 1180, 975, 757, 731, 698 cm^{-1} .

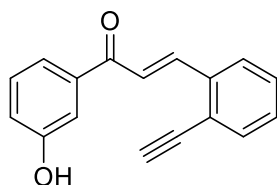


(E)-3-(2-chlorophenyl)-1-(3-hydroxyphenyl)prop-2-en-1-one (8c). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (43.2 mg, 67% yield). m.p. = 111 – 113 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

1H NMR (400 MHz, Acetone- d_6) δ 8.78 (br, 1H), 8.17 (d, J = 15.6 Hz, 1H), 8.11 (dd, J = 7.3, 2.2 Hz, 1H), 7.84 (d, J = 15.6 Hz, 1H), 7.71 – 7.65 (m, 1H), 7.62 – 7.57 (m, 1H), 7.56 – 7.51 (m, 1H), 7.49 – 7.37 (m, 3H), 7.19 – 7.12 (m, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.6, 158.7, 140.3, 139.7, 135.7, 133.9, 132.4, 130.9, 130.7, 129.1, 128.4, 125.6, 121.1, 120.9, 115.7.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{15}H_{11}O_2ClNa$ 281.0340; Found 281.0328.

IR: 3314, 2924, 2852, 1652, 1577, 1468, 1443, 1315, 1277, 1183, 975, 778, 753 cm^{-1} .

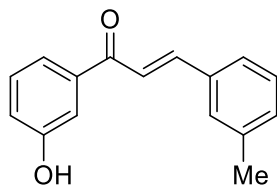


(E)-3-(2-ethynylphenyl)-1-(3-hydroxyphenyl)prop-2-en-1-one (8d). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a brown solid (39.7 mg, 64% yield). m.p. = 117 – 119 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 8.27 (d, J = 15.8 Hz, 1H), 7.73 – 7.68 (m, 1H), 7.66 – 7.62 (m, 1H), 7.61 – 7.51 (m, 3H), 7.41 – 7.30 (m, 3H), 7.16 – 7.11 (m, 1H), 7.06 (br, 1H), 3.43 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 191.4, 156.7, 143.2, 139.4, 136.8, 133.8, 130.2, 130.0, 129.2, 126.7, 124.1, 123.5, 121.2, 120.8, 115.5, 84.0, 81.3.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{12}\text{O}_2\text{Na}$ 271.0730; Found 271.0726.

IR: 3288, 2925, 2853, 1655, 1578, 1475, 1447, 1327, 1277, 1185, 980, 758 cm^{-1} .

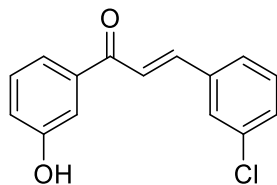


(E)-1-(3-hydroxyphenyl)-3-(*m*-tolyl)prop-2-en-1-one (8e). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (42.2 mg, 71% yield). m.p. = 84 – 86 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.73 (br, 1H), 7.83 – 7.70 (m, 2H), 7.69 – 7.63 (m, 2H), 7.62 – 7.58 (m, 1H), 7.58 – 7.54 (m, 1H), 7.42 – 7.37 (m, 1H), 7.37 – 7.31 (m, 1H), 7.29 – 7.24 (m, 1H), 7.15 – 7.10 (m, 1H), 2.38 (s, 3H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.8, 158.7, 144.9, 140.7, 139.4, 136.0, 132.1, 130.6, 129.9, 129.7, 126.8, 122.8, 120.8, 120.7, 115.6, 21.3.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{16}\text{H}_{14}\text{O}_2\text{Na}$ 261.0886; Found 261.0891.

IR: 3299, 2920, 2851, 1650, 1570, 1481, 1446, 1315, 1267, 1230, 1185, 980, 777, 684 cm^{-1} .

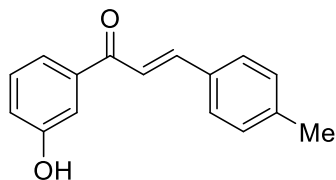


(E)-3-(3-chlorophenyl)-1-(3-hydroxyphenyl)prop-2-en-1-one (8f). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (53.5 mg, 83% yield). m.p. = 112 – 114 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.82 (br, 1H), 7.96 – 7.88 (m, 2H), 7.80 – 7.71 (m, 2H), 7.71 – 7.66 (m, 1H), 7.62 – 7.57 (m, 1H), 7.52 – 7.44 (m, 2H), 7.43 – 7.36 (m, 1H), 7.17 – 7.11 (m, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.6, 158.6, 142.9, 140.3, 138.2, 135.3, 131.4, 130.9, 130.7, 128.7, 128.2, 124.5, 121.0, 120.8, 115.7.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{15}\text{H}_{11}\text{O}_2\text{ClNa}$ 281.0340; Found 281.0332.

IR: 3336, 2926, 1655, 1579, 1473, 1448, 1312, 1185, 979, 779 cm^{-1} .

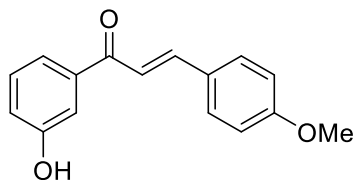


(E)-1-(3-hydroxyphenyl)-3-(p-tolyl)prop-2-en-1-one (8g). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (39.9 mg, 67% yield). m.p. = 110 – 112 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.75 (br, 1H), 7.76 (s, 2H), 7.74 – 7.68 (m, 2H), 7.67 – 7.62 (m, 1H), 7.59 – 7.55 (m, 1H), 7.42 – 7.36 (m, 1H), 7.28 (d, J = 7.9 Hz, 2H), 7.15 – 7.10 (m, 1H), 2.37 (s, 3H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.8, 158.6, 144.8, 141.7, 140.7, 133.3, 130.6, 130.5 (2C), 129.5 (2C), 122.0, 120.73, 120.66, 115.6, 21.4.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{16}\text{H}_{14}\text{O}_2\text{Na}$ 261.0886; Found 261.0894.

IR: 3316, 2923, 2853, 1650, 1577, 1511, 1447, 1332, 1263, 1180, 1030, 982, 812, 791, 737 cm^{-1} .



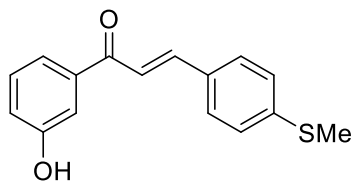
(E)-1-(3-hydroxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one (8h). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (46.4 mg, 73% yield). m.p. = 115 – 117 °C. Eluant: ethyl acetate/petroleum ether (1:4, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.75 (br, 1H), 7.85 – 7.72 (m, 3H), 7.71 – 7.62 (m, 2H), 7.59 – 7.54 (m, 1H), 7.42 – 7.35 (m, 1H), 7.15 – 7.09 (m, 1H), 7.04 – 6.98 (m, 2H), 3.86 (s, 3H).

^{13}C NMR (101 MHz, Acetone- d_6) δ 189.7, 162.7, 158.6, 144.7, 140.9, 131.3 (2C), 130.6, 128.6, 120.60, 120.57, 120.5, 115.6, 115.2 (2C), 55.8.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{16}\text{H}_{14}\text{O}_3\text{Na}$ 277.0835; Found 277.0827.

IR: 3277, 2925, 2839, 1647, 1558, 1509, 1445, 1422, 1250, 1168, 1028, 826, 799, 739 cm^{-1} .

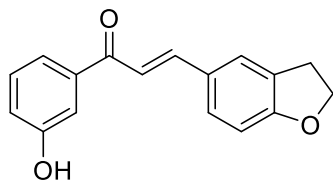


(E)-1-(3-hydroxyphenyl)-3-(4-(methylthio)phenyl)prop-2-en-1-one (8i). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (48.6 mg, 72% yield). m.p. = 114 – 116 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.69 (br, 1H), 7.81 – 7.73 (m, 4H), 7.67 – 7.61 (m, 1H), 7.59 – 7.53 (m, 1H), 7.42 – 7.36 (m, 1H), 7.35 – 7.29 (m, 2H), 7.15 – 7.09 (m, 1H), 2.55 (s, 3H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.7, 158.6, 144.3, 143.3, 140.7, 132.4, 130.6, 129.9 (2C), 126.6 (2C), 121.9, 120.73, 120.67, 115.6, 14.8.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{16}\text{H}_{14}\text{O}_2\text{SNa}$ 293.0607; Found 293.0600.

IR: 3299, 2922, 2852, 1649, 1575, 1549, 1491, 1446, 1406, 1184, 1091, 815, 777 cm^{-1} .

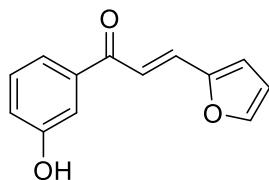


(E)-3-(2,3-dihydrobenzofuran-5-yl)-1-(3-hydroxyphenyl)prop-2-en-1-one (8j). Prepared according to general procedure D and purified by flash column chromatography to afford the product as yellow solid (39.2 mg, 59% yield). m.p. = 179 – 181 °C. Eluant: ethyl acetate/petroleum ether (1:4, R_f = 0.3).

^1H NMR (400 MHz, Acetone- d_6) δ 8.74 (br, 1H), 7.79 – 7.71 (m, 2H), 7.68 – 7.60 (m, 2H), 7.59 – 7.53 (m, 2H), 7.41 – 7.34 (m, 1H), 7.13 – 7.07 (m, 1H), 6.81 (d, J = 8.3 Hz, 1H), 4.63 (t, J = 8.7 Hz, 2H), 3.27 (t, J = 8.7 Hz, 2H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.7, 163.6, 158.6, 145.2, 141.0, 131.2, 130.5, 129.6, 128.8, 126.0, 120.54, 120.51, 119.8, 115.6, 110.2, 72.7, 29.7.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{14}\text{O}_3\text{Na}$ 289.0835; Found 289.0826.

IR: 3349, 2920, 2850, 1658, 1579, 1491, 1445, 1264, 1242, 736 cm^{-1} .

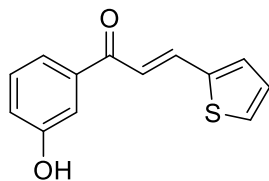


(E)-3-(furan-2-yl)-1-(3-hydroxyphenyl)prop-2-en-1-one (8k). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a brown solid (33.7 mg, 63% yield). m.p. = 128 – 130 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.77 (br, 1H), 7.78 (d, J = 1.8 Hz, 1H), 7.62 – 7.55 (m, 2H), 7.54 – 7.46 (m, 2H), 7.43 – 7.37 (m, 1H), 7.15 – 7.09 (m, 1H), 7.00 (d, J = 3.4 Hz, 1H), 6.65 (dd, J = 3.5, 1.8 Hz, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.2, 158.7, 152.6, 146.4, 140.5, 131.0, 130.7, 120.8, 120.5, 120.0, 117.1, 115.5, 113.7.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{10}\text{O}_3\text{Na}$ 237.0522; Found 237.0517.

IR: 3316, 2923, 2852, 1652, 1577, 1549, 1475, 1447, 1287, 1016, 750 cm^{-1} .

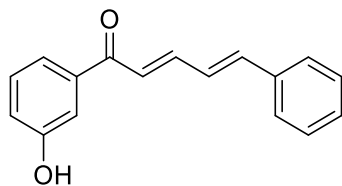


(E)-1-(3-hydroxyphenyl)-3-(thiophen-2-yl)prop-2-en-1-one (8l). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (36.8 mg, 64% yield). m.p. = 109 – 111 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.73 (br, 1H), 7.93 (d, J = 15.3 Hz, 1H), 7.66 (d, J = 5.1 Hz, 1H), 7.62 – 7.56 (m, 2H), 7.55 – 7.52 (m, 1H), 7.49 (d, J = 15.3 Hz, 1H), 7.43 – 7.35 (m, 1H), 7.18 (dd, J = 5.1, 3.6 Hz, 1H), 7.12 (dd, J = 8.0, 2.6 Hz, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.3, 158.6, 141.1, 140.5, 137.3, 133.2, 130.7, 130.2, 129.4, 121.5, 120.8, 120.6, 115.5.

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{13}\text{H}_{11}\text{O}_2\text{S}$ 231.0474; Found 231.0468.

IR: 3281, 2923, 2852, 1644, 1563, 1512, 1486, 1446, 1284, 1200, 966, 705 cm^{-1} .

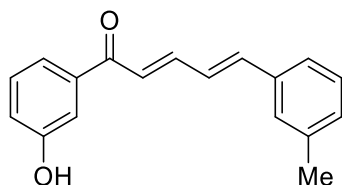


(2E,4E)-1-(3-hydroxyphenyl)-5-phenylpenta-2,4-dien-1-one (8m). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (45.0 mg, 72% yield). m.p. = 115 – 117 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.73 (br, 1H), 7.66 – 7.59 (m, 2H), 7.59 – 7.51 (m, 2H), 7.51 – 7.47 (m, 1H), 7.45 – 7.33 (m, 4H), 7.32 – 7.22 (m, 2H), 7.21 – 7.14 (m, 1H), 7.14 – 7.08 (m, 1H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.8, 158.7, 145.0, 142.3, 140.7, 137.4, 130.6, 129.9, 129.7 (2C), 128.2, 128.1 (2C), 126.5, 120.7, 120.5, 115.5.

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{17}\text{H}_{15}\text{O}_2$ 251.1067; Found 251.1060.

IR: 3322, 2925, 1644, 1566, 1447, 1353, 1290, 1148, 997, 795, 752 cm^{-1} .

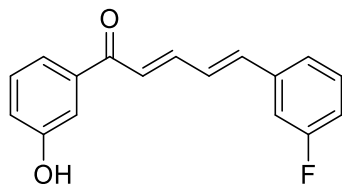


(2E,4E)-1-(3-hydroxyphenyl)-5-(*m*-tolyl)penta-2,4-dien-1-one (8n). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (46.9 mg, 71% yield). m.p. = 65 – 67 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.73 (br, 1H), 7.62 – 7.51 (m, 2H), 7.51 – 7.47 (m, 1H), 7.46 – 7.43 (m, 1H), 7.43 – 7.35 (m, 2H), 7.33 – 7.23 (m, 3H), 7.22 – 7.14 (m, 2H), 7.13 – 7.08 (m, 1H), 2.35 (s, 3H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.8, 158.6, 145.1, 142.5, 140.7, 139.3, 137.3, 130.7, 130.6, 129.6, 128.8, 128.0, 126.4, 125.4, 120.7, 120.5, 115.5, 21.3.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{18}\text{H}_{16}\text{O}_2\text{Na}$ 287.1043; Found 287.1035.

IR: 3282, 2923, 2854, 1643, 1558, 1486, 1446, 1346, 1284, 1148, 996, 793, 723 cm^{-1} .

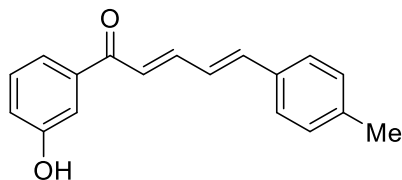


(2E,4E)-5-(3-fluorophenyl)-1-(3-hydroxyphenyl)penta-2,4-dien-1-one (8o). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (54.9 mg, 82% yield). m.p. = 119 – 121 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.73 (br, 1H), 7.63 – 7.49 (m, 3H), 7.48 – 7.36 (m, 4H), 7.35 – 7.25 (m, 2H), 7.20 – 7.06 (m, 3H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.8, 164.0 (C-F, $^1J_{\text{C-F}}$ = 244.1 Hz), 158.6, 144.4, 140.6 (C-F, $^4J_{\text{C-F}}$ = 2.9 Hz), 140.5, 139.9 (C-F, $^3J_{\text{C-F}}$ = 7.7 Hz), 131.5 (C-F, $^3J_{\text{C-F}}$ = 8.4 Hz), 130.7, 129.6, 127.3, 124.4 (C-F, $^4J_{\text{C-F}}$ = 2.8 Hz), 120.8, 120.5, 116.4 (C-F, $^2J_{\text{C-F}}$ = 21.6 Hz), 115.4, 114.0 (C-F, $^2J_{\text{C-F}}$ = 22.0 Hz).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{13}\text{O}_2\text{FNa}$ 291.0792; Found 291.0784.

IR: 3284, 2924, 2853, 1645, 1562, 1445, 1345, 1276, 1147, 995, 784, 722 cm^{-1} .

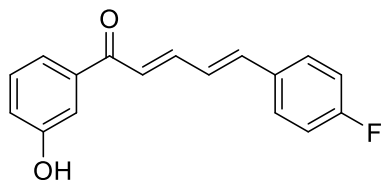


(2E,4E)-1-(3-hydroxyphenyl)-5-(p-tolyl)penta-2,4-dien-1-one (8p). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (50.2 mg, 76% yield). m.p. = 136 – 138 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.75 (br, 1H), 7.61 – 7.51 (m, 3H), 7.50 – 7.46 (m, 2H), 7.42 – 7.34 (m, 1H), 7.30 – 7.16 (m, 4H), 7.15 – 7.08 (m, 2H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.8, 158.6, 145.3, 142.4, 140.7, 140.1, 134.6, 130.6, 130.4 (2C), 128.1 (2C), 127.2, 125.9, 120.6, 120.4, 115.4, 21.3.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{18}\text{H}_{16}\text{O}_2\text{Na}$ 287.1043; Found 287.1030.

IR: 3299, 2923, 2854, 1643, 1561, 1448, 1287, 997, 805, 725 cm^{-1} .

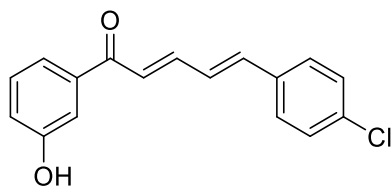


(2E,4E)-5-(4-fluorophenyl)-1-(3-hydroxyphenyl)penta-2,4-dien-1-one (8q). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (52.9 mg, 79% yield). m.p. = 110 – 112 °C. Eluant: ethyl acetate/petroleum ether (1:5, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.74 (br, 1H), 7.70 – 7.61 (m, 2H), 7.61 – 7.48 (m, 3H), 7.42 – 7.35 (m, 1H), 7.27 (d, J = 14.8 Hz, 1H), 7.24 – 7.09 (m, 5H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.8, 163.9 (C-F, $^1J_{\text{C-F}}$ = 247.8 Hz), 158.6, 144.9, 140.9, 140.6, 133.8 (C-F, $^4J_{\text{C-F}}$ = 3.4 Hz), 130.6, 130.1 (C-F, $^3J_{\text{C-F}}$ = 8.3 Hz, 2C), 128.0 (C-F, $^5J_{\text{C-F}}$ = 2.5 Hz), 126.5, 120.7, 120.5, 116.6 (C-F, $^2J_{\text{C-F}}$ = 22.0 Hz, 2C), 115.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{13}\text{O}_2\text{FNa}$ 291.0792; Found 291.0783.

IR: 3283, 2925, 2853, 1644, 1561, 1506, 1447, 1287, 1230, 1155, 996, 844, 794, 781 cm^{-1} .

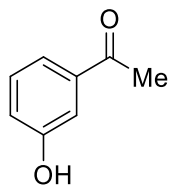


(2E,4E)-5-(4-chlorophenyl)-1-(3-hydroxyphenyl)penta-2,4-dien-1-one (8r). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (55.4 mg, 78% yield). m.p. = 135 – 137 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 8.75 (br, 1H), 7.62 (d, J = 8.2 Hz, 2H), 7.60 – 7.52 (m, 2H), 7.51 – 7.48 (m, 1H), 7.43 (d, J = 8.2 Hz, 2H), 7.41 – 7.35 (m, 1H), 7.34 – 7.20 (m, 2H), 7.19 – 7.08 (m, 2H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 189.8, 158.7, 144.6, 140.64, 140.60, 136.2, 135.0, 130.7, 129.8 (2C), 129.6 (2C), 129.0, 127.0, 120.8, 120.5, 115.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{13}\text{O}_2\text{ClNa}$ 307.0496; Found 307.0490.

IR: 3342, 2925, 2853, 1647, 1568, 1490, 1448, 1283, 1091, 997, 812, 795, 725 cm^{-1} .

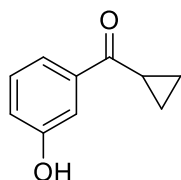


1-(3-hydroxyphenyl)ethan-1-one (10a). Prepared according to procedure for the synthesis of **10a** and purified by flash column chromatography to afford the product as a white solid (21.1 mg, 62% yield). m.p. = 91 – 93 °C. Eluant: ethyl acetate/petroleum ether (1:8, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.58 – 7.54 (m, 1H), 7.54 – 7.49 (m, 1H), 7.38 – 7.31 (m, 1H), 7.15 – 7.10 (m, 1H), 6.84 (br, 1H), 2.61 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 199.6, 156.5, 138.4, 130.1, 121.2, 121.0, 114.8, 26.9.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_8\text{H}_8\text{O}_2\text{Na}$ 159.0417; Found 159.0412.

IR: 3345, 2924, 2852, 1669, 1598, 1585, 1450, 1360, 1288, 1220, 787 cm^{-1} .

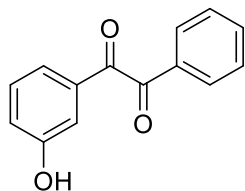


cyclopropyl(3-hydroxyphenyl)methanone (10b). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow oil (22.7 mg, 56% yield). Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.64 – 7.55 (m, 2H), 7.39 – 7.32 (m, 1H), 7.12 – 7.07 (m, 1H), 6.48 (br, 1H), 2.72 – 2.62 (m, 1H), 1.31 – 1.22 (m, 2H), 1.12 – 1.03 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.4, 156.4, 139.5, 130.0, 120.8, 120.4, 114.7, 17.5, 12.2 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{10}\text{H}_{10}\text{O}_2\text{Na}$ 185.0573; Found 185.0578.

IR: 3314, 2925, 2853, 1650, 1582, 1448, 1387, 1276, 1177, 1041, 908, 741 cm^{-1} .

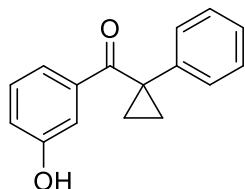


1-(3-hydroxyphenyl)-2-phenylethane-1,2-dione (10c). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (20.3 mg, 36% yield). m.p. = 82 – 84 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 8.00 – 7.92 (m, 2H), 7.70 – 7.62 (m, 1H), 7.55 – 7.44 (m, 4H), 7.40 – 7.33 (m, 1H), 7.18 – 7.12 (m, 1H), 5.83 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 194.8, 194.7, 156.5, 135.2, 134.3, 132.9, 130.6, 130.1 (2C), 129.2 (2C), 123.0, 122.7, 115.8.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{10}\text{O}_3\text{Na}$ 249.0522; Found 249.0513.

IR: 3375, 2924, 2853, 1665, 1595, 1582, 1449, 1261, 1229, 749, 717 cm^{-1} .



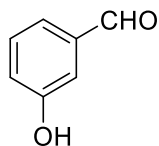
(3-hydroxyphenyl)(1-phenylcyclopropyl)methanone (10d). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow

solid (24.4 mg, 41% yield). m.p. = 62 – 64 °C. Eluant: ethyl acetate/petroleum ether (1:6, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.33 – 7.27 (m, 2H), 7.25 – 7.13 (m, 5H), 7.13 – 7.07 (m, 1H), 6.92 – 6.84 (m, 1H), 5.66 (br, 1H), 1.70 – 1.62 (m, 2H), 1.40 – 1.32 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 200.9, 155.7, 140.9, 138.5, 129.4, 128.8 (2C), 128.0 (2C), 126.8, 122.3, 119.6, 116.0, 35.4, 16.6 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{16}\text{H}_{14}\text{O}_2\text{Na}$ 261.0886; Found 261.0897.

IR: 3358, 2954, 2924, 1656, 1596, 1583, 1446, 1297, 1279, 1172, 1157, 745, 698 cm^{-1} .

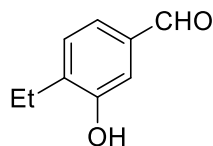


3-hydroxybenzaldehyde (10e). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (26.2 mg, 86% yield). m.p. = 100 – 103 °C. Eluant: ethyl acetate/petroleum ether (1:10, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 9.96 (s, 1H), 7.49 – 7.40 (m, 2H), 7.40 – 7.37 (m, 1H), 7.19 – 7.13 (m, 1H), 5.92 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 192.7, 156.6, 137.9, 130.6, 123.7, 122.3, 114.9.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_7\text{H}_6\text{O}_2\text{Na}$ 145.0260; Found 145.0266.

IR: 3210, 2956, 2924, 1667, 1580, 1493, 1281, 1249, 1172, 783 cm^{-1} .

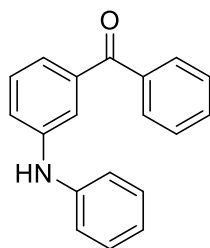


4-ethyl-3-hydroxybenzaldehyde (10f). Prepared according to general procedure D and purified by flash column chromatography to afford the product as a yellow solid (33.4 mg, 89% yield). m.p. = 55 – 57 °C. Eluant: ethyl acetate/petroleum ether (1:10, R_f = 0.30).

^1H NMR (400 MHz, Acetone- d_6) δ 9.90 (s, 1H), 8.83 (br, 1H), 7.40 – 7.32 (m, 3H), 2.72 (q, J = 7.5 Hz, 2H), 1.22 (t, J = 7.5 Hz, 3H). ^{13}C NMR (101 MHz, Acetone- d_6) δ 192.5, 156.4, 139.0, 137.0, 130.6, 123.3, 114.4, 24.2, 14.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_9\text{H}_{10}\text{O}_2\text{Na}$ 173.0573; Found 173.0569.

IR: 3355, 2969, 2934, 1679, 1605, 1584, 1433, 1395, 821, 760 cm^{-1} .

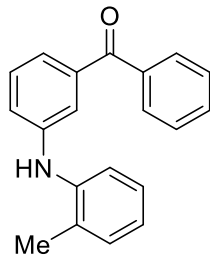


phenyl(3-(phenylamino)phenyl)methanone (12a). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (49.8 mg, 73% yield). m.p. = 72 – 74 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.78 (m, 2H), 7.61 – 7.53 (m, 1H), 7.51 – 7.43 (m, 3H), 7.38 – 7.25 (m, 5H), 7.15 – 7.06 (m, 2H), 7.00 – 6.93 (m, 1H), 5.89 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.9, 143.7, 142.4, 138.9, 137.7, 132.6, 130.2 (2C), 129.6 (2C), 129.3, 128.4 (2C), 122.6, 121.9, 120.9, 118.6 (2C), 118.4.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{19}H_{15}NONa$ 296.1046; Found 296.1052.

IR: 3358, 3056, 2923, 2852, 1648, 1590, 1577, 1494, 1449, 1413, 1318, 1275, 986, 750, 714, 693 cm^{-1} .

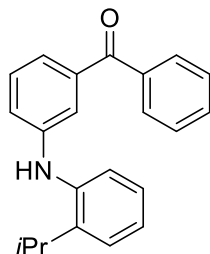


phenyl(3-(*o*-tolylamino)phenyl)methanone (12b). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (43.8 mg, 61% yield). Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

1H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.75 (m, 2H), 7.61 – 7.52 (m, 1H), 7.51 – 7.41 (m, 2H), 7.39 – 7.34 (m, 1H), 7.33 – 7.28 (m, 1H), 7.28 – 7.19 (m, 3H), 7.18 – 7.09 (m, 2H), 7.03 – 6.94 (m, 1H), 5.55 (br, 1H), 2.25 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.9, 144.6, 140.4, 138.9, 137.7, 132.5, 131.2, 130.2 (2C), 129.4, 129.2, 128.3 (2C), 127.0, 123.0, 122.1, 120.4, 119.9, 118.0, 18.1.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{20}H_{17}NONa$ 310.1202; Found 310.1216.

IR: 3368, 3057, 2923, 1649, 1595, 1575, 1514, 1318, 1274, 985, 748, 716, 706 cm^{-1} .

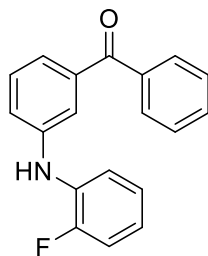


(3-((2-isopropylphenyl)amino)phenyl)(phenyl)methanone (12c). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (55.9 mg, 71% yield). Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

1H NMR (400 MHz, $CDCl_3$) δ 7.86 – 7.78 (m, 2H), 7.61 – 7.53 (m, 1H), 7.50 – 7.42 (m, 2H), 7.37 – 7.23 (m, 4H), 7.22 – 7.14 (m, 2H), 7.13 – 7.08 (m, 1H), 7.07 – 7.02 (m, 1H), 5.56 (br, 1H), 3.23 – 3.09 (m, 1H), 1.23 (d, J = 6.8 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.0, 145.9, 141.7, 138.9, 138.8, 137.8, 132.5, 130.2 (2C), 129.2, 128.3 (2C), 126.7, 126.4, 124.5, 122.8, 121.6, 119.5, 117.0, 27.9, 23.2 (2C).

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{22}H_{21}NONa$ 338.1515; Found 338.1526.

IR: 3370, 3059, 2960, 2924, 1650, 1595, 1576, 1509, 1487, 1447, 1318, 1279, 1082, 754, 722, 711 cm^{-1} .

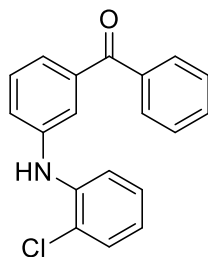


(3-((2-fluorophenyl)amino)phenyl)(phenyl)methanone (12d). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (61.8 mg, 85% yield). Eluant: ethyl acetate/petroleum ether (1:15, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.86 – 7.79 (m, 2H), 7.62 – 7.55 (m, 1H), 7.54 – 7.50 (m, 1H), 7.50 – 7.44 (m, 2H), 7.41 – 7.27 (m, 4H), 7.14 – 6.99 (m, 2H), 6.93 – 6.85 (m, 1H), 5.94 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.7, 153.5 (C-F, $^1J_{\text{C-F}}$ = 241.9 Hz), 142.6, 139.0, 137.6, 132.6, 130.8 (C-F, $^2J_{\text{C-F}}$ = 11.1 Hz), 130.2 (2C), 129.3, 128.4 (2C), 124.5 (C-F, $^3J_{\text{C-F}}$ = 3.7 Hz), 123.4, 121.69 (C-F, $^3J_{\text{C-F}}$ = 7.3 Hz), 121.68, 119.1, 118.2 (C-F, $^4J_{\text{C-F}}$ = 2.1 Hz), 115.8 (C-F, $^2J_{\text{C-F}}$ = 19.3 Hz).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{14}\text{NOFNa}$ 314.0952; Found 314.0960.

IR: 3357, 3059, 2922, 1651, 1618, 1597, 1578, 1320, 1273, 1099, 779, 715 cm^{-1} .

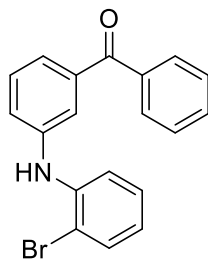


(3-((2-chlorophenyl)amino)phenyl)(phenyl)methanone (12e). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (62.2 mg, 81% yield). Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.98 – 7.78 (m, 2H), 7.64 – 7.53 (m, 2H), 7.52 – 7.44 (m, 2H), 7.43 – 7.28 (m, 5H), 7.19 – 7.09 (m, 1H), 6.88 – 6.80 (m, 1H), 6.20 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.5, 142.1, 139.5, 139.0, 137.5, 132.7, 130.2 (2C), 130.0, 129.4, 128.4 (2C), 127.6, 124.1, 123.2, 122.4, 121.4, 120.5, 116.4.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{14}\text{NOClNa}$ 330.0656; Found 330.0665.

IR: 3358, 3027, 2923, 1652, 1578, 1514, 1446, 1315, 1272, 738, 718, 706 cm^{-1} .



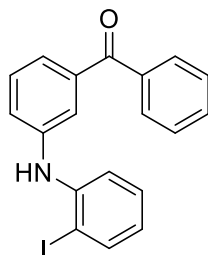
(3-((2-bromophenyl)amino)phenyl)(phenyl)methanone (12f). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (71.9 mg, 82% yield). Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.88 – 7.78 (m, 2H), 7.65 – 7.44 (m, 5H), 7.43 – 7.33 (m, 3H), 7.32 – 7.27 (m, 1H), 7.23 – 7.13 (m, 1H), 6.84 – 6.73 (m, 1H), 6.18 (br, 1H). ^{13}C NMR (101

MHz, CDCl₃) δ 196.5, 142.2, 140.7, 139.1, 137.6, 133.3, 132.7, 130.2 (2C), 129.4, 128.4 (2C), 128.3, 124.2, 123.2, 122.0, 120.6, 116.6, 113.1.

HRMS (ESI) m/z : [M + Na]⁺ Calcd for C₁₉H₁₄NOBrNa 374.0151; Found 374.0167.

IR: 3359, 3060, 2923, 1651, 1577, 1310, 1272, 1022, 745, 716, 703 cm⁻¹.

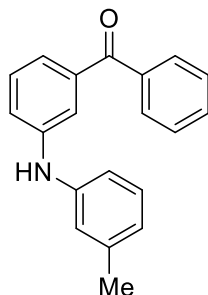


(3-((2-iodophenyl)amino)phenyl)(phenyl)methanone (12g). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (66.8 mg, 67% yield). Eluant: ethyl acetate/petroleum ether (1:25, R_f = 0.30).

¹H NMR (400 MHz, CDCl₃) δ 7.87 – 7.81 (m, 2H), 7.81 – 7.76 (m, 1H), 7.64 – 7.56 (m, 1H), 7.56 – 7.52 (m, 1H), 7.52 – 7.45 (m, 2H), 7.44 – 7.36 (m, 2H), 7.35 – 7.30 (m, 1H), 7.29 – 7.20 (m, 2H), 6.71 – 6.64 (m, 1H), 6.00 (br, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 196.6, 143.2, 142.6, 139.8, 139.1, 137.6, 132.7, 130.2 (2C), 129.4, 129.3, 128.4 (2C), 124.0, 123.0, 122.9, 120.2, 116.8, 89.8.

HRMS (ESI) m/z : [M + Na]⁺ Calcd for C₁₉H₁₄NOINa 422.0012; Found 421.9999.

IR: 3374, 3058, 2920, 2850, 1651, 1574, 1510, 1484, 1456, 1441, 1312, 1277, 1010, 748, 717, 703 cm⁻¹.

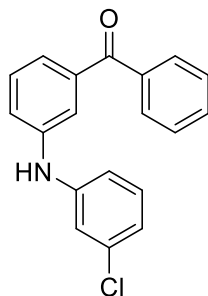


phenyl(3-(*m*-tolylamino)phenyl)methanone (12h). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (48.8 mg, 68% yield). Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

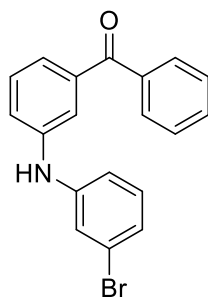
¹H NMR (400 MHz, CDCl₃) δ 7.86 – 7.77 (m, 2H), 7.62 – 7.54 (m, 1H), 7.53 – 7.42 (m, 3H), 7.38 – 7.31 (m, 1H), 7.31 – 7.26 (m, 2H), 7.21 – 7.13 (m, 1H), 6.95 – 6.88 (m, 2H), 6.82 – 6.72 (m, 1H), 5.84 (br, 1H), 2.31 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 196.9, 143.7, 142.3, 139.5, 138.9, 137.7, 132.6, 130.2 (2C), 129.4, 129.3, 128.4 (2C), 122.8, 122.5, 121.0, 119.2, 118.5, 115.7, 21.7.

HRMS (ESI) m/z : [M + Na]⁺ Calcd for C₂₀H₁₇NONa 310.1202; Found 310.1193.

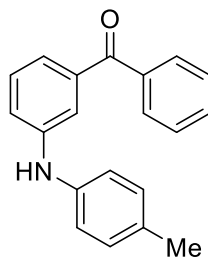
IR: 3362, 3033, 2922, 2853, 1649, 1575, 1484, 1446, 1405, 1319, 1275, 1166, 986, 775, 714, 693 cm⁻¹.



(3-((3-chlorophenyl)amino)phenyl)(phenyl)methanone (12i). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (55.3 mg, 72% yield). m.p. = 68 – 70 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30). ^1H NMR (400 MHz, CDCl_3) δ 7.88 – 7.76 (m, 2H), 7.64 – 7.55 (m, 1H), 7.54 – 7.44 (m, 3H), 7.42 – 7.33 (m, 2H), 7.32 – 7.27 (m, 1H), 7.21 – 7.12 (m, 1H), 7.11 – 7.04 (m, 1H), 6.98 – 6.85 (m, 2H), 5.97 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.7, 144.1, 142.5, 139.0, 137.5, 135.2, 132.7, 130.6, 130.2 (2C), 129.5, 128.5 (2C), 123.6, 122.1, 121.4, 119.6, 117.4, 115.9. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{14}\text{NOClNa}$ 330.0656; Found 330.0649. IR: 3354, 2923, 2852, 1650, 1585, 1519, 1479, 1446, 1319, 1277, 1265, 1075, 991, 769, 720, 703 cm^{-1} .



(3-((3-bromophenyl)amino)phenyl)(phenyl)methanone (12j). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (62.3 mg, 71% yield). m.p. = 65 – 67 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30). ^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.78 (m, 2H), 7.63 – 7.54 (m, 1H), 7.53 – 7.43 (m, 3H), 7.42 – 7.33 (m, 2H), 7.32 – 7.27 (m, 1H), 7.25 – 7.19 (m, 1H), 7.15 – 7.08 (m, 1H), 7.07 – 7.01 (m, 1H), 7.00 – 6.93 (m, 1H), 5.94 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.6, 144.2, 142.5, 139.0, 137.6, 132.7, 130.9, 130.2 (2C), 129.5, 128.5 (2C), 124.3, 123.6, 123.3, 122.1, 120.4, 119.7, 116.4. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{14}\text{NOBrNa}$ 374.0151; Found 374.0133. IR: 3351, 2923, 2853, 1647, 1574, 1515, 1475, 1444, 1317, 1278, 989, 769, 716 cm^{-1} .

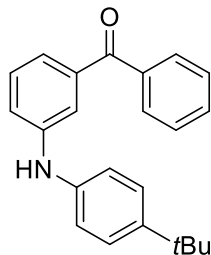


phenyl(3-(p-tolylamino)phenyl)methanone (12k). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (59.6 mg, 83% yield). m.p. = 77 – 79 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.78 (m, 2H), 7.63 – 7.53 (m, 1H), 7.52 – 7.43 (m, 2H), 7.42 – 7.37 (m, 1H), 7.35 – 7.27 (m, 1H), 7.24 – 7.18 (m, 2H), 7.15 – 7.07 (m, 2H), 7.06 – 6.98 (m, 2H), 5.77 (br, 1H), 2.30 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.9, 144.4, 139.6, 138.9, 137.8, 132.5, 131.9, 130.2 (2C), 130.1 (2C), 129.2, 128.3 (2C), 122.0, 120.1, 119.6 (2C), 117.6, 20.9.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{17}\text{NONa}$ 310.1202; Found 310.1214.

IR: 3365, 2918, 2849, 1646, 1595, 1515, 1321, 1275, 811, 716 cm^{-1} .

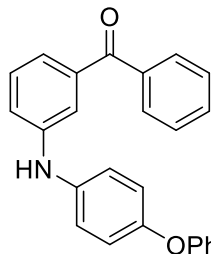


(3-((4-(*tert*-butyl)phenyl)amino)phenyl)(phenyl)methanone (12l). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (65.8 mg, 80% yield). m.p. = 78 – 80 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.77 (m, 2H), 7.60 – 7.52 (m, 1H), 7.50 – 7.39 (m, 3H), 7.36 – 7.27 (m, 3H), 7.26 – 7.18 (m, 2H), 7.11 – 7.01 (m, 2H), 5.83 (br, 1H), 1.30 (s, 9H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.9, 145.1, 144.3, 139.6, 138.9, 137.8, 132.5, 130.2 (2C), 129.2, 128.3 (2C), 126.4 (2C), 122.1, 120.3, 118.9 (2C), 117.8, 34.3, 31.6 (3C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{23}\text{H}_{23}\text{NONa}$ 352.1672; Found 352.1659.

IR: 3361, 3056, 2958, 2864, 1649, 1594, 1577, 1516, 1482, 1445, 1266, 825, 778, 710 cm^{-1} .

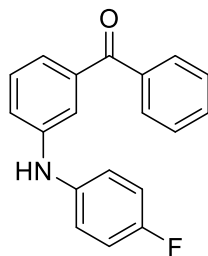


(3-((4-phenoxyphenyl)amino)phenyl)(phenyl)methanone (12m). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (70.3 mg, 77% yield). m.p. = 79 – 81 °C. Eluant: ethyl acetate/petroleum ether (1:15, R_f = 0.30).

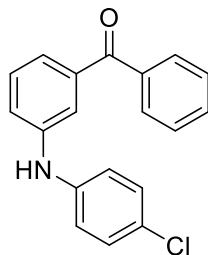
^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.78 (m, 2H), 7.62 – 7.52 (m, 1H), 7.50 – 7.43 (m, 2H), 7.42 – 7.38 (m, 1H), 7.36 – 7.27 (m, 3H), 7.25 – 7.16 (m, 2H), 7.13 – 7.03 (m, 3H), 7.02 – 6.92 (m, 4H), 5.85 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.9, 158.1, 152.1, 144.6, 139.0, 137.9, 137.8, 132.6, 130.2 (2C), 129.8 (2C), 129.3, 128.4 (2C), 123.0, 122.2, 121.4 (2C), 120.6 (2C), 120.0, 118.3 (2C), 117.5.

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{25}\text{H}_{20}\text{NO}_2$ 366.1489; Found 366.1482.

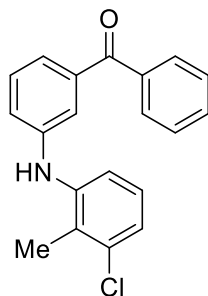
IR: 3360, 2954, 2922, 1649, 1594, 1578, 1503, 1485, 1320, 1217, 778, 715, 689 cm^{-1} .



(3-((4-fluorophenyl)amino)phenyl)(phenyl)methanone (12n). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (59.7 mg, 82% yield). m.p. = 69 – 71 °C. Eluant: ethyl acetate/petroleum ether (1:15, R_f = 0.30). ^1H NMR (400 MHz, CDCl_3) δ 7.84 – 7.77 (m, 2H), 7.62 – 7.53 (m, 1H), 7.51 – 7.41 (m, 2H), 7.41 – 7.35 (m, 1H), 7.34 – 7.27 (m, 1H), 7.26 – 7.20 (m, 1H), 7.19 – 7.13 (m, 1H), 7.12 – 7.03 (m, 2H), 7.02 – 6.92 (m, 2H), 5.83 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.9, 158.6 (C-F, $^1J_{\text{C-F}}$ = 241.1 Hz), 144.5, 139.0, 138.2 (C-F, $^4J_{\text{C-F}}$ = 2.7 Hz), 137.7, 132.6, 130.2 (2C), 129.3, 128.4 (2C), 122.3, 121.5 (C-F, $^3J_{\text{C-F}}$ = 7.9 Hz, 2C), 120.0, 117.4, 116.2 (C-F, $^2J_{\text{C-F}}$ = 22.4 Hz, 2C). HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{14}\text{NOFNa}$ 314.0952; Found 314.0940. IR: 3355, 3058, 2923, 2853, 1648, 1595, 1578, 1503, 1445, 1320, 1274, 822, 778, 713 cm^{-1} .



(3-((4-chlorophenyl)amino)phenyl)(phenyl)methanone (12o). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (64.5 mg, 84% yield). m.p. = 97 – 99 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30). ^1H NMR (400 MHz, CDCl_3) δ 7.86 – 7.77 (m, 2H), 7.63 – 7.55 (m, 1H), 7.51 – 7.42 (m, 3H), 7.38 – 7.32 (m, 1H), 7.31 – 7.27 (m, 1H), 7.26 – 7.18 (m, 3H), 7.07 – 6.97 (m, 2H), 5.88 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.7, 143.3, 141.1, 139.1, 137.6, 132.7, 130.2 (2C), 129.6 (2C), 129.4, 128.4 (2C), 126.5, 123.1, 121.2, 119.7 (2C), 118.6. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{14}\text{NOCINa}$ 330.0656; Found 330.0643. IR: 3351, 2923, 2852, 1647, 1587, 1576, 1512, 1487, 1445, 1318, 1273, 810, 779, 716 cm^{-1} .

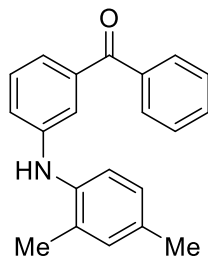


(3-((3-chloro-2-methylphenyl)amino)phenyl)(phenyl)methanone (12p). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (60.2 mg, 75% yield). m.p. = 79 – 81 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.86 – 7.76 (m, 2H), 7.63 – 7.53 (m, 1H), 7.52 – 7.42 (m, 2H), 7.38 – 7.30 (m, 2H), 7.29 – 7.22 (m, 1H), 7.20 – 7.01 (m, 4H), 5.62 (br, 1H), 2.31 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.8, 144.3, 142.0, 139.0, 137.6, 135.6, 132.6, 130.2 (2C), 129.3, 128.4 (2C), 127.9, 127.1, 124.0, 122.6, 120.8, 118.7, 118.3, 14.8.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{16}\text{NOClNa}$ 344.0813; Found 344.0807.

IR: 3361, 3058, 2923, 2853, 1650, 1594, 1570, 1446, 1317, 1265, 1013, 774, 718 cm^{-1} .

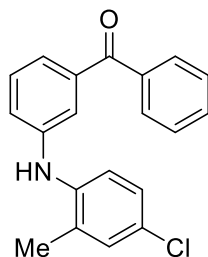


(3-((2,4-dimethylphenyl)amino)phenyl)(phenyl)methanone (12q). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (63.2 mg, 84% yield). Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.77 (m, 2H), 7.61 – 7.53 (m, 1H), 7.51 – 7.42 (m, 2H), 7.31 – 7.25 (m, 2H), 7.21 – 7.11 (m, 2H), 7.08 – 7.00 (m, 2H), 7.00 – 6.94 (m, 1H), 5.45 (br, 1H), 2.30 (s, 3H), 2.22 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.0, 145.5, 138.8, 137.8, 137.5, 133.3, 132.5, 131.9, 130.7, 130.2 (2C), 129.1, 128.3 (2C), 127.5, 121.6, 121.4, 119.4, 116.9, 20.9, 18.0.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{21}\text{H}_{19}\text{NONa}$ 324.1359; Found 324.1347.

IR: 3368, 3023, 2920, 2854, 1649, 1595, 1577, 1504, 1318, 1272, 778, 716 cm^{-1} .

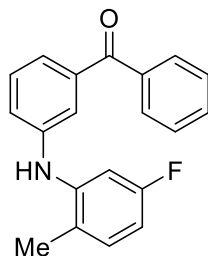


(3-((4-chloro-2-methylphenyl)amino)phenyl)(phenyl)methanone (12r). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (66.6 mg, 83% yield). Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.86 – 7.76 (m, 2H), 7.62 – 7.53 (m, 1H), 7.51 – 7.42 (m, 2H), 7.36 – 7.28 (m, 2H), 7.27 – 7.21 (m, 1H), 7.20 – 7.14 (m, 2H), 7.13 – 7.05 (m, 2H), 5.52 (br, 1H), 2.22 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.8, 144.2, 139.1, 139.0, 137.6, 132.6, 131.2, 130.9, 130.1 (2C), 129.3, 128.4 (2C), 127.6, 126.9, 122.5, 121.0, 120.6, 118.0, 17.9.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{16}\text{NOClNa}$ 344.0813; Found 344.0799.

IR: 3371, 3059, 2924, 2853, 1650, 1595, 1579, 1513, 1485, 1447, 1274, 779, 717, 703 cm^{-1} .

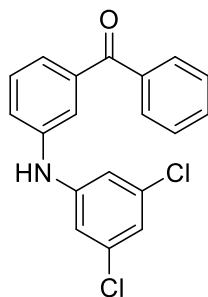


(3-((5-fluoro-2-methylphenyl)amino)phenyl)(phenyl)methanone (12s). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (54.1 mg, 71% yield). Eluant: ethyl acetate/petroleum ether (1:15, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.88 – 7.78 (m, 2H), 7.63 – 7.55 (m, 1H), 7.54 – 7.46 (m, 2H), 7.45 – 7.42 (m, 1H), 7.41 – 7.31 (m, 2H), 7.28 – 7.22 (m, 1H), 7.15 – 7.08 (m, 1H), 7.00 – 6.92 (m, 1H), 6.65 – 6.56 (m, 1H), 5.58 (br, 1H), 2.21 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.7, 162.0 (C-F, $^1J_{\text{C-F}}$ = 242.5 Hz), 143.1, 142.2 (C-F, $^3J_{\text{C-F}}$ = 10.0 Hz), 139.0, 137.6, 132.7, 131.9 (C-F, $^3J_{\text{C-F}}$ = 9.3 Hz), 130.2 (2C), 129.4, 128.4 (2C), 123.4, 122.7 (C-F, $^4J_{\text{C-F}}$ = 2.9 Hz), 122.0, 119.7, 108.3 (C-F, $^2J_{\text{C-F}}$ = 21.0 Hz), 104.4 (C-F, $^2J_{\text{C-F}}$ = 24.9 Hz), 17.4.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{16}\text{NOFNa}$ 328.1108; Found 328.1101.

IR: 3370, 3060, 2923, 2853, 1650, 1595, 1579, 1519, 1414, 1319, 1275, 1156, 779, 715 cm^{-1} .

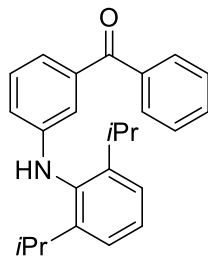


(3-((3,5-dichlorophenyl)amino)phenyl)(phenyl)methanone (12t). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (54.6 mg, 64% yield). m.p. = 76 – 78 °C. Eluant: ethyl acetate/petroleum ether (1:15, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.78 (m, 2H), 7.64 – 7.56 (m, 1H), 7.54 – 7.47 (m, 3H), 7.46 – 7.39 (m, 2H), 7.36 – 7.29 (m, 1H), 6.95 – 6.89 (m, 2H), 6.89 – 6.85 (m, 1H), 6.04 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.6, 145.2, 141.5, 139.1, 137.4, 135.8 (2C), 132.8, 130.2 (2C), 129.7, 128.5 (2C), 124.5, 123.2, 120.84, 120.82, 115.1 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{19}\text{H}_{14}\text{NOCl}_2$ 342.0447; Found 342.0432.

IR: 3350, 3061, 2922, 2852, 1648, 1571, 1444, 1319, 1274, 1111, 942, 799, 781, 718 cm^{-1} .



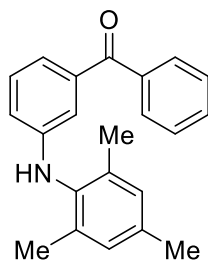
(3-((2,6-diisopropylphenyl)amino)phenyl)(phenyl)methanone (12u). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a

yellow solid (70.5 mg, 79% yield). m.p. = 101 – 103 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.76 (m, 2H), 7.59 – 7.51 (m, 1H), 7.49 – 7.40 (m, 2H), 7.33 – 7.26 (m, 1H), 7.24 – 7.15 (m, 3H), 7.09 – 7.00 (m, 2H), 6.64 – 6.55 (m, 1H), 5.31 (br, 1H), 3.28 – 3.11 (m, 2H), 1.15 (d, J = 6.8 Hz, 12H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.2, 148.4, 147.7 (2C), 138.8, 137.9, 134.6, 132.4, 130.2 (2C), 129.1, 128.3 (2C), 127.8, 124.1 (2C), 120.0, 116.5, 114.1, 28.4 (2C), 24.0 (4C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{25}\text{H}_{27}\text{NONa}$ 380.1985; Found 380.1976.

IR: 3369, 3062, 2961, 2867, 1650, 1600, 1578, 1502, 1469, 1446, 1321, 799, 724, 710 cm^{-1} .

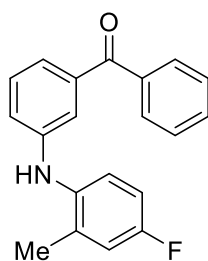


(3-(mesitylamino)phenyl)(phenyl)methanone (12v). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (70.1 mg, 89% yield). m.p. = 103 – 105 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.76 (m, 2H), 7.60 – 7.51 (m, 1H), 7.49 – 7.40 (m, 2H), 7.22 – 7.15 (m, 1H), 7.09 – 7.04 (m, 1H), 7.03 – 6.99 (m, 1H), 6.93 (s, 2H), 6.63 – 6.57 (m, 1H), 5.27 (br, 1H), 2.29 (s, 3H), 2.18 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.2, 147.0, 138.8, 138.0, 136.2 (2C), 136.0, 134.9, 132.3, 130.2 (2C), 129.5 (2C), 129.1, 128.2 (2C), 120.2, 116.7, 114.4, 21.0, 18.4 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{22}\text{H}_{21}\text{NONa}$ 338.1515; Found 338.1507.

IR: 3368, 2917, 2853, 1649, 1595, 1577, 1500, 1480, 1446, 1320, 1272, 1218, 984, 778, 719, 705 cm^{-1} .

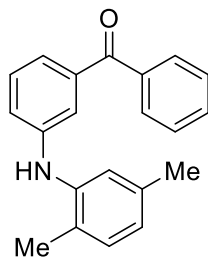


(3-((4-fluoro-2-methylphenyl)amino)phenyl)(phenyl)methanone (12w). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (59.5 mg, 78% yield). m.p. = 58 – 60 °C. Eluant: ethyl acetate/petroleum ether (1:15, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.88 – 7.76 (m, 2H), 7.62 – 7.53 (m, 1H), 7.52 – 7.42 (m, 2H), 7.32 – 7.26 (m, 1H), 7.24 – 7.21 (m, 1H), 7.21 – 7.15 (m, 2H), 7.00 – 6.92 (m, 2H), 6.91 – 6.83 (m, 1H), 5.41 (br, 1H), 2.24 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.0, 159.5 (C-F, $^1J_{\text{C-F}}$ = 242.7 Hz), 145.7, 138.9, 137.7, 136.0 (C-F, $^4J_{\text{C-F}}$ = 2.8 Hz), 134.0 (C-F, $^3J_{\text{C-F}}$ = 7.8 Hz), 132.5, 130.2 (2C), 129.2, 128.3 (2C), 124.2 (C-F, $^3J_{\text{C-F}}$ = 8.4 Hz), 121.6, 119.1, 117.7 (C-F, $^2J_{\text{C-F}}$ = 22.2 Hz), 116.5, 113.6 (C-F, $^2J_{\text{C-F}}$ = 22.1 Hz), 18.2 (C-F, $^4J_{\text{C-F}}$ = 1.4 Hz).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{16}\text{NOFNa}$ 328.1108; Found 328.1096.

IR: 3361, 3058, 2922, 2853, 1650, 1596, 1579, 1491, 1447, 1320, 1268, 1148, 779, 716 cm^{-1} .

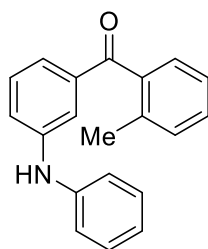


(3-((2,5-dimethylphenyl)amino)phenyl)(phenyl)methanone (12x). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (47.4 mg, 63% yield). Eluant: ethyl acetate/petroleum ether (1:20, $R_f = 0.30$).

^1H NMR (400 MHz, CDCl_3) δ 7.88 – 7.77 (m, 2H), 7.61 – 7.53 (m, 1H), 7.52 – 7.43 (m, 2H), 7.38 – 7.29 (m, 2H), 7.27 – 7.22 (m, 1H), 7.16 – 7.03 (m, 3H), 6.84 – 6.75 (m, 1H), 5.49 (br, 1H), 2.28 (s, 3H), 2.21 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.9, 144.7, 140.2, 138.9, 137.8, 136.7, 132.5, 131.1, 130.2 (2C), 129.3, 128.3 (2C), 126.3, 123.9, 121.9, 120.6, 120.5, 118.0, 21.3, 17.6.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{21}\text{H}_{19}\text{NONa}$ 324.1359; Found 324.1366.

IR: 3369, 3023, 2921, 2854, 1650, 1596, 1575, 1523, 1496, 1473, 1446, 1319, 1275, 1001, 983, 805, 779, 717, 701 cm^{-1} .

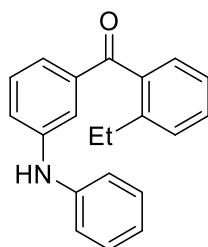


(3-(phenylamino)phenyl)(o-tolyl)methanone (13b). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (52.4 mg, 73% yield). Eluant: ethyl acetate/petroleum ether (1:20, $R_f = 0.30$).

^1H NMR (400 MHz, CDCl_3) δ 7.52 – 7.47 (m, 1H), 7.42 – 7.21 (m, 9H), 7.13 – 7.04 (m, 2H), 7.01 – 6.92 (m, 1H), 5.86 (br, 1H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 198.7, 143.8, 142.3, 139.1, 138.8, 136.9, 131.1, 130.4, 129.6 (2C), 129.5, 128.6, 125.2, 122.9, 121.9, 121.5, 118.5 (2C), 118.2, 20.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{17}\text{NONa}$ 310.1202; Found 310.1196.

IR: 3361, 3056, 2954, 2923, 2853, 1654, 1589, 1515, 1494, 1484, 1451, 1307, 1263, 985, 738, 703 cm^{-1} .



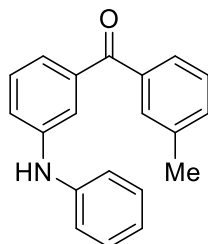
(2-ethylphenyl)(3-(phenylamino)phenyl)methanone (13c). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (53.4 mg, 71% yield). Eluant: ethyl acetate/petroleum ether (1:20, $R_f = 0.30$).

^1H NMR (400 MHz, CDCl_3) δ 7.54 – 7.49 (m, 1H), 7.45 – 7.37 (m, 1H), 7.36 – 7.20 (m, 8H), 7.13 – 7.04 (m, 2H), 7.01 – 6.93 (m, 1H), 5.85 (br, 1H), 2.68 (q, $J = 7.6$ Hz, 2H), 1.17 (t, $J = 7.5$

Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 198.8, 143.8, 143.1, 142.3, 139.2, 138.5, 130.4, 129.6 (2C), 129.5, 129.4, 128.4, 125.2, 123.0, 121.9, 121.6, 118.5 (2C), 118.2, 26.5, 16.1.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{21}\text{H}_{19}\text{NONa}$ 324.1359; Found 324.1347.

IR: 3363, 3059, 2961, 2871, 1654, 1589, 1515, 1484, 1452, 1307, 1265, 986, 747, 704 cm^{-1} .

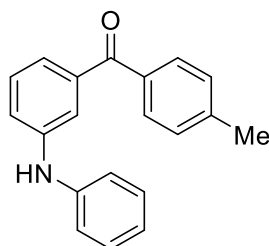


(3-(phenylamino)phenyl)(*m*-tolyl)methanone (13h). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (42.3 mg, 59% yield). m.p. = 51 – 53 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.64 (s, 1H), 7.62 – 7.56 (m, 1H), 7.49 – 7.44 (m, 1H), 7.42 – 7.32 (m, 3H), 7.31 – 7.23 (m, 4H), 7.15 – 7.06 (m, 2H), 7.01 – 6.92 (m, 1H), 5.90 (br, 1H), 2.42 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.1, 143.5, 142.4, 139.0, 138.2, 137.7, 133.4, 130.6, 129.6 (2C), 129.3, 128.2, 127.5, 122.6, 121.8, 120.9, 118.49, 118.46 (2C), 21.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{17}\text{NONa}$ 310.1202; Found 310.1211.

IR: 3356, 3037, 2922, 2853, 1647, 1590, 1578, 1517, 1494, 1309, 1277, 992, 787, 739 cm^{-1} .

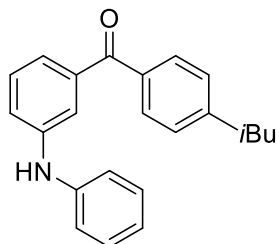


(3-(phenylamino)phenyl)(*p*-tolyl)methanone (13n). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (40.9 mg, 57% yield). m.p. = 78 – 80 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.79 – 7.70 (m, 2H), 7.48 – 7.41 (m, 1H), 7.38 – 7.22 (m, 7H), 7.15 – 7.06 (m, 2H), 7.01 – 6.92 (m, 1H), 5.88 (br, 1H), 2.43 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.6, 143.5, 143.4, 142.4, 139.3, 134.9, 130.4 (2C), 129.6 (2C), 129.2, 129.1 (2C), 122.5, 121.8, 120.7, 118.5 (2C), 118.4, 21.8.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{17}\text{NONa}$ 310.1202; Found 310.1207.

IR: 3352, 3032, 2922, 2852, 1645, 1590, 1579, 1494, 1313, 1275, 1179, 834, 791, 744 cm^{-1} .



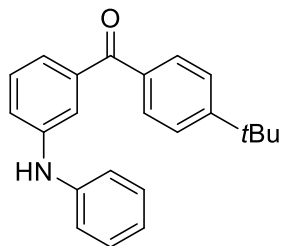
(4-isobutylphenyl)(3-(phenylamino)phenyl)methanone (13o). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (50.2 mg, 61% yield). m.p. = 76 – 78 °C. Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.82 – 7.71 (m, 2H), 7.47 – 7.43 (m, 1H), 7.37 – 7.21 (m, 7H),

7.15 – 7.06 (m, 2H), 7.00 – 6.92 (m, 1H), 5.88 (br, 1H), 2.55 (d, $J = 7.2$ Hz, 2H), 2.00 – 1.83 (m, 1H), 0.93 (d, $J = 6.6$ Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.7, 147.1, 143.6, 142.4, 139.3, 135.2, 130.3 (2C), 129.6 (2C), 129.2, 129.1 (2C), 122.5, 121.8, 120.7, 118.5 (2C), 118.4, 45.5, 30.3, 22.5 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{23}\text{H}_{23}\text{NONa}$ 352.1672; Found 352.1684.

IR: 3357, 3049, 2954, 2867, 1647, 1591, 1580, 1495, 1313, 1264, 1180, 986, 790, 735 cm^{-1} .

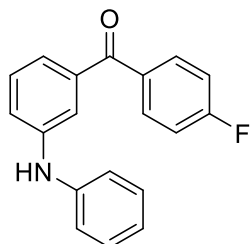


(4-(*tert*-butyl)phenyl)(3-(phenylamino)phenyl)methanone (13p). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (51.0 mg, 62% yield). m.p. = 68 – 70 °C. Eluant: ethyl acetate/petroleum ether (1:20, $R_f = 0.30$).

^1H NMR (400 MHz, CDCl_3) δ 7.82 – 7.74 (m, 2H), 7.52 – 7.47 (m, 2H), 7.47 – 7.43 (m, 1H), 7.36 – 7.25 (m, 5H), 7.14 – 7.07 (m, 2H), 7.00 – 6.92 (m, 1H), 5.91 (br, 1H), 1.36 (s, 9H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.6, 156.3, 143.5, 142.4, 139.3, 134.9, 130.3 (2C), 129.6 (2C), 129.2, 125.3 (2C), 122.6, 121.8, 120.7, 118.5, 118.4 (2C), 35.2, 31.3 (3C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{23}\text{H}_{23}\text{NONa}$ 352.1672; Found 352.1664.

IR: 3355, 3037, 2959, 2867, 1645, 1591, 1518, 1495, 1451, 1315, 1266, 798, 739, 716 cm^{-1} .

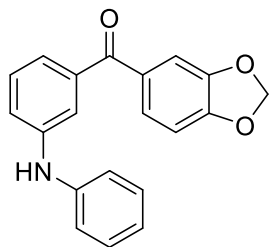


(4-fluorophenyl)(3-(phenylamino)phenyl)methanone (13r). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow solid (45.1 mg, 62% yield). m.p. = 53 – 55 °C. Eluant: ethyl acetate/petroleum ether (1:15, $R_f = 0.30$).

^1H NMR (400 MHz, CDCl_3) δ 7.91 – 7.82 (m, 2H), 7.45 – 7.40 (m, 1H), 7.38 – 7.20 (m, 5H), 7.19 – 7.07 (m, 4H), 7.01 – 6.94 (m, 1H), 5.89 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 195.4, 165.5 (C-F, $^1J_{\text{C-F}} = 254.3$ Hz), 143.8, 142.2, 138.8, 133.9 (C-F, $^4J_{\text{C-F}} = 3.3$ Hz), 132.8 (C-F, $^3J_{\text{C-F}} = 9.2$ Hz, 2C), 129.6 (2C), 129.4, 122.3, 122.0, 120.9, 118.7 (2C), 118.1, 115.5 (C-F, $^2J_{\text{C-F}} = 21.8$ Hz, 2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{14}\text{NOFNa}$ 314.0952; Found 314.0941.

IR: 3360, 3059, 2953, 2923, 2853, 1649, 1590, 1579, 1494, 1451, 1410, 1310, 1274, 1226, 1154, 987, 750, 699 cm^{-1} .

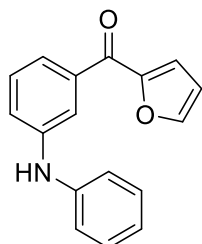


benzo[d][1,3]dioxol-5-yl(3-(phenylamino)phenyl)methanone (13w). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (45.2 mg, 57% yield). Eluant: ethyl acetate/petroleum ether (1:10, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.45 – 7.36 (m, 3H), 7.36 – 7.20 (m, 5H), 7.16 – 7.07 (m, 2H), 7.01 – 6.93 (m, 1H), 6.86 (d, J = 8.1 Hz, 1H), 6.07 (s, 2H), 5.86 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 195.3, 151.7, 148.0, 143.5, 142.4, 139.5, 132.0, 129.6 (2C), 129.2, 127.0, 122.2, 121.9, 120.6, 118.6 (2C), 118.2, 110.0, 107.8, 102.0.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{15}\text{NO}_3\text{Na}$ 340.0944; Found 340.0936.

IR: 3351, 3055, 2922, 2852, 1644, 1590, 1579, 1495, 1483, 1437, 1257, 1035, 748, 721 cm^{-1} .

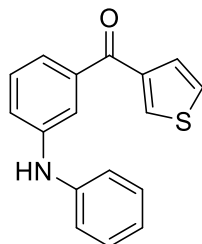


furan-2-yl(3-(phenylamino)phenyl)methanone (13z). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (38.1 mg, 58% yield). Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.72 – 7.67 (m, 1H), 7.63 – 7.58 (m, 1H), 7.51 – 7.45 (m, 1H), 7.40 – 7.33 (m, 1H), 7.33 – 7.26 (m, 3H), 7.24 (d, J = 3.5 Hz, 1H), 7.15 – 7.08 (m, 2H), 7.02 – 6.95 (m, 1H), 6.61 – 6.56 (m, 1H), 5.88 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 182.7, 152.4, 147.3, 143.8, 142.4, 138.6, 129.6 (2C), 129.5, 122.0, 121.7, 121.2, 120.7, 118.7 (2C), 117.8, 112.3.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{13}\text{NO}_2\text{Na}$ 286.0838; Found 286.0845.

IR: 3346, 3054, 2954, 2922, 2852, 1634, 1591, 1578, 1494, 1460, 1307, 1171, 1026, 744 cm^{-1} .



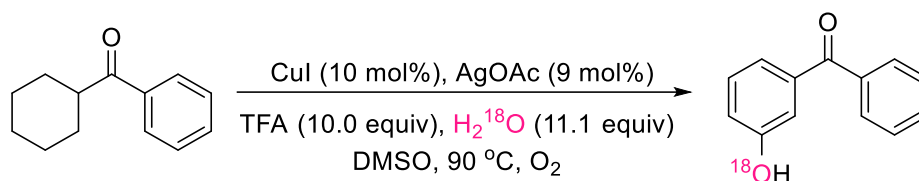
(3-(phenylamino)phenyl)(thiophen-3-yl)methanone (13aa). Prepared according to general procedure E and purified by flash column chromatography to afford the product as a yellow oil (37.0 mg, 53% yield). Eluant: ethyl acetate/petroleum ether (1:20, R_f = 0.30).

^1H NMR (400 MHz, CDCl_3) δ 7.99 – 7.93 (m, 1H), 7.60 (d, J = 5.1 Hz, 1H), 7.53 – 7.46 (m, 1H), 7.43 – 7.33 (m, 3H), 7.32 – 7.27 (m, 3H), 7.17 – 7.07 (m, 2H), 7.03 – 6.94 (m, 1H), 5.89 (br, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 190.1, 143.8, 142.3, 141.4, 140.0, 134.2, 129.6 (2C), 129.4, 128.7, 126.3, 122.0, 121.8, 120.8, 118.7 (2C), 117.7.

HRMS (ESI) m/z : $[M + Na]^+$ Calcd for $C_{17}H_{13}NOSNa$ 302.0610; Found 302.0619.
IR: 3352, 3054, 2921, 2851, 1635, 1590, 1578, 1509, 1494, 1451, 1412, 1265, 790, 740 cm^{-1} .

5. Preliminary Mechanistic Studies

5.1 ^{18}O -labeled water experiment



A 15 mL sealed tube containing a magnetic stir bar was charged with ketone **1a** (47.0 mg, 0.25 mmol), CuI (4.8 mg, 10 mol%), AgOAc (3.7 mg, 9 mol%) and DMSO (1 mL) sequentially. The tube was evacuated and backfilled with O_2 three times. Subsequently, H_2^{18}O (50 μL) and TFA (186 μL , 10.0 equiv) was added. The tube was sealed and the mixture was stirred at $90\text{ }^\circ\text{C}$ for 60 h. After cooling to room temperature, the reaction mixture was diluted with EtOAc (50 mL) and washed with H_2O ($3 \times 1\text{ mL}$) and brine ($3 \times 1\text{ mL}$). The combined organic layer was dried over anhydrous Na_2SO_4 , and concentrated to give the residue, which was further purified by silica gel column chromatography to afford the desired product. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{10}\text{O}^{18}\text{ONa}$ 223.0615; Found 223.0610.

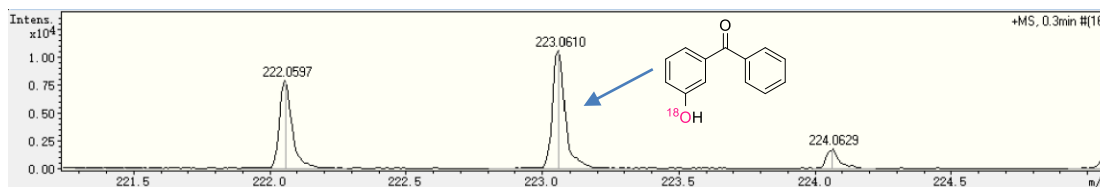
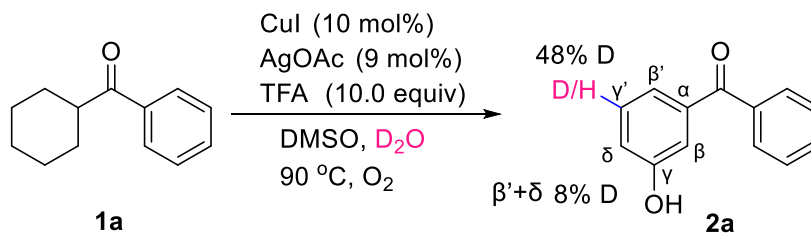


Figure S1. HRMS spectrum of the ^{18}O Compound

5.2 Deuterium-labeling experiments

5.2.1 H/D exchange experiments—investigations of γ -C–H activation



A 15 mL sealed tube containing a magnetic stir bar was charged with ketone **1a** (47.0 mg, 0.25 mmol), CuI (4.8 mg, 10 mol%), AgOAc (3.7 mg, 9 mol%) and DMSO (1 mL) sequentially. The tube was evacuated and backfilled with O_2 three times. Subsequently, D_2O (50 μL) and TFA (186 μL , 10.0 equiv) was added. The tube was sealed and the mixture was stirred at $90\text{ }^\circ\text{C}$ for 60 h. After cooling to room temperature, the reaction mixture was diluted with EtOAc (50 mL) and washed with H_2O ($3 \times 1\text{ mL}$) and brine ($3 \times 1\text{ mL}$). The combined organic layer was dried over anhydrous Na_2SO_4 , and concentrated to give the residue, which was further purified by silica gel column chromatography to afford the deuterium-labeling product which was characterized by

^1H NMR spectroscopy. The deuterium-labeling product was found to contain 48% D incorporation at the γ' position.

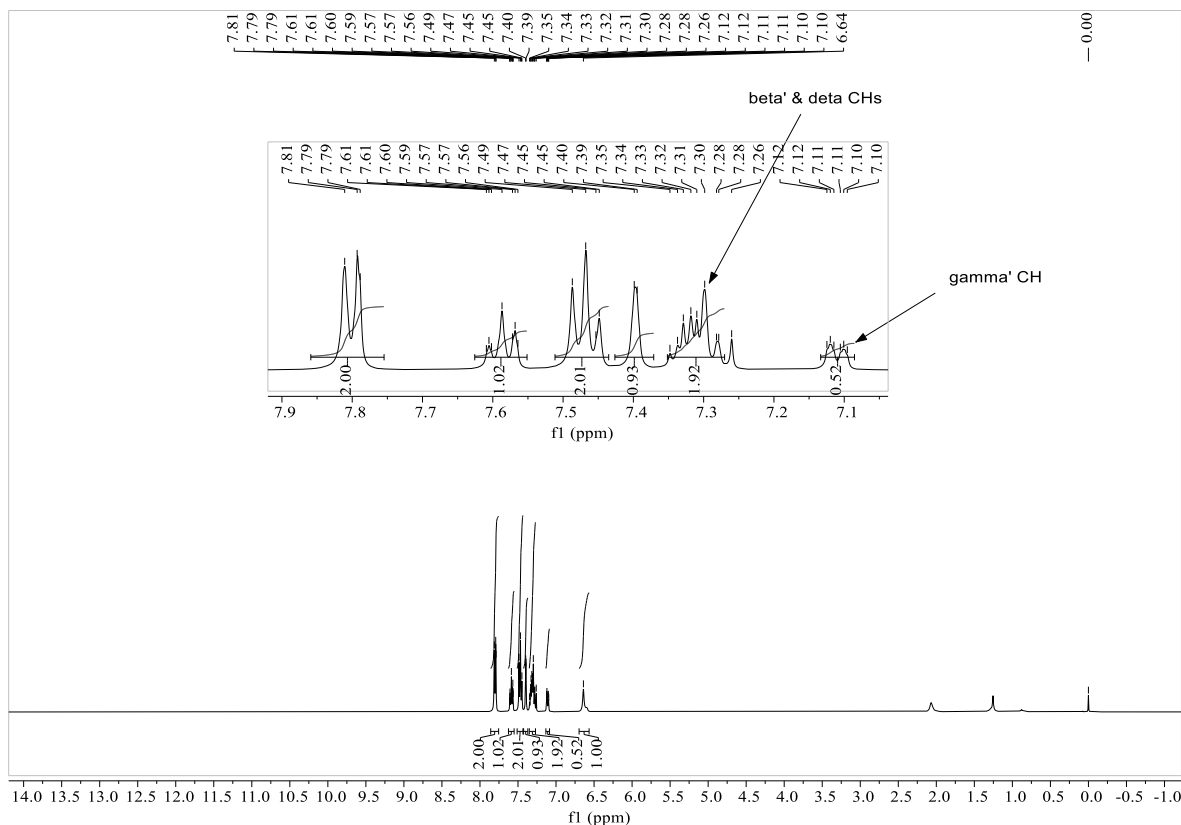
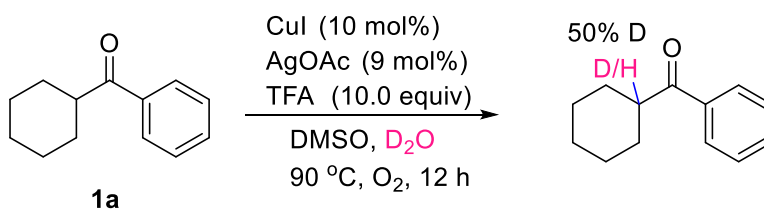


Figure S2. ^1H NMR spectrum of compound deuterium-labeling **2a** (400 MHz, CDCl_3)

5.2.2 H/D exchange experiments – investigations of α -C–H activation



A 15 mL sealed tube containing a magnetic stir bar was charged with ketone **1a** (47.0 mg, 0.25 mmol), CuI (4.8 mg, 10 mol%), AgOAc (3.7 mg, 9 mol%) and DMSO (1 mL) sequentially. The tube was evacuated and backfilled with O_2 three times. Subsequently, D_2O (50 μL) and TFA (186 μL , 10.0 equiv) was added. The tube was sealed and the mixture was stirred at 90 $^\circ\text{C}$ for 12 h. After cooling to room temperature, the reaction mixture was diluted with EtOAc (50 mL) and washed with H_2O ($3 \times 1 \text{ mL}$) and brine ($3 \times 1 \text{ mL}$). The combined organic layer was dried over anhydrous Na_2SO_4 , and concentrated to give the residue, which was further purified by silica gel column chromatography to afford the recovered **1a** which was characterized by ^1H NMR spectroscopy. The recovered **1a** was found to contain 50% D at the α position.

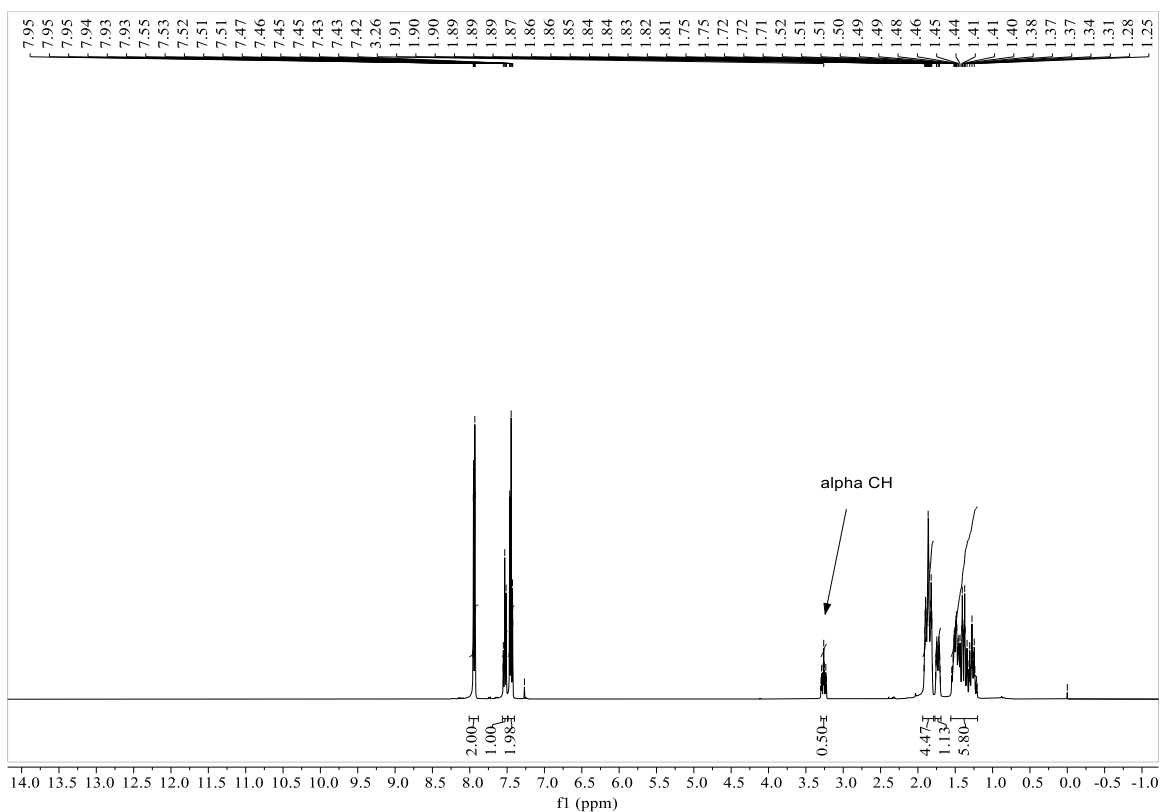
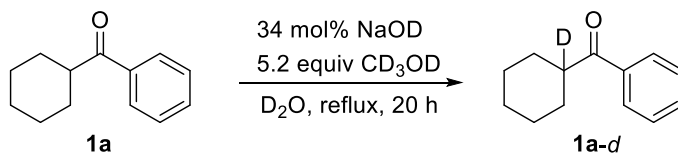


Figure S3. ^1H NMR spectrum of the recovered **1a** (400 MHz, CDCl_3)

5.3 KIE experiments

Deuterated-substrate preparation: Synthesis of **(cyclohexyl-1-*d*)(phenyl)methanone (1a-*d*)**.²²



(cyclohexyl-1-*d*)(phenyl)methanone (1a-*d*). An oven-dried 50 mL round bottom flask was placed 8.5 mL D_2O , cyclohexyl(phenyl)methanone (1.88 g, 10.0 mmol), CD_3OD (1.82 g, 52.0 mmol) and a solution of 40% NaOD in D_2O (0.3 mL, 3.4 mmol) under argon atmosphere. The reaction mixture was stirred at reflux for 20 h. After cooling to room temperature, 8 mL diethyl ether was added via syringe. After stirring for 1 h, the layers were separated and the aqueous layer was washed with ether (2×20 mL). The combined organic layers were washed with water (2×20 mL) and brine (2×20 mL), dried over Na_2SO_4 and concentrated by evaporation. The crude product was shown to be 98% deuterated material.

^1H NMR (400 MHz, CDCl_3) δ 8.00 – 7.89 (m, 2H), 7.58 – 7.51 (m, 1H), 7.50 – 7.41 (m, 2H), 1.96 – 1.80 (m, 4H), 1.79 – 1.66 (m, 1H), 1.58 – 1.19 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 204.1, 136.4, 132.9, 128.7 (2C), 128.4 (2C), 45.3 (t, J = 19.3 Hz), 29.4 (2C), 26.1, 26.0 (2C).

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{15}\text{DONa}$ 212.1156; Found 212.1148.

IR: 2928, 2856, 1666, 1595, 1578, 1444, 1286, 1251, 1180, 980, 763, 699 cm^{-1} .

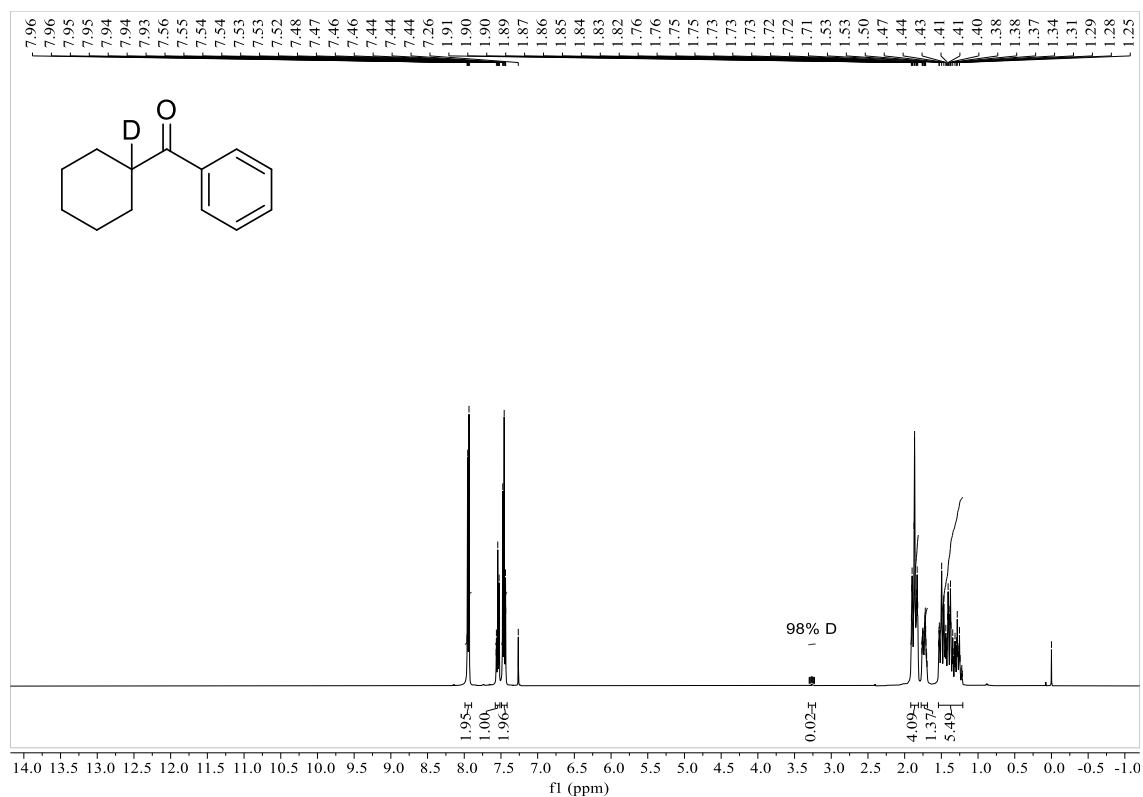


Figure S4. ¹H NMR spectrum of compound **1a-d** (400 MHz, CDCl₃)

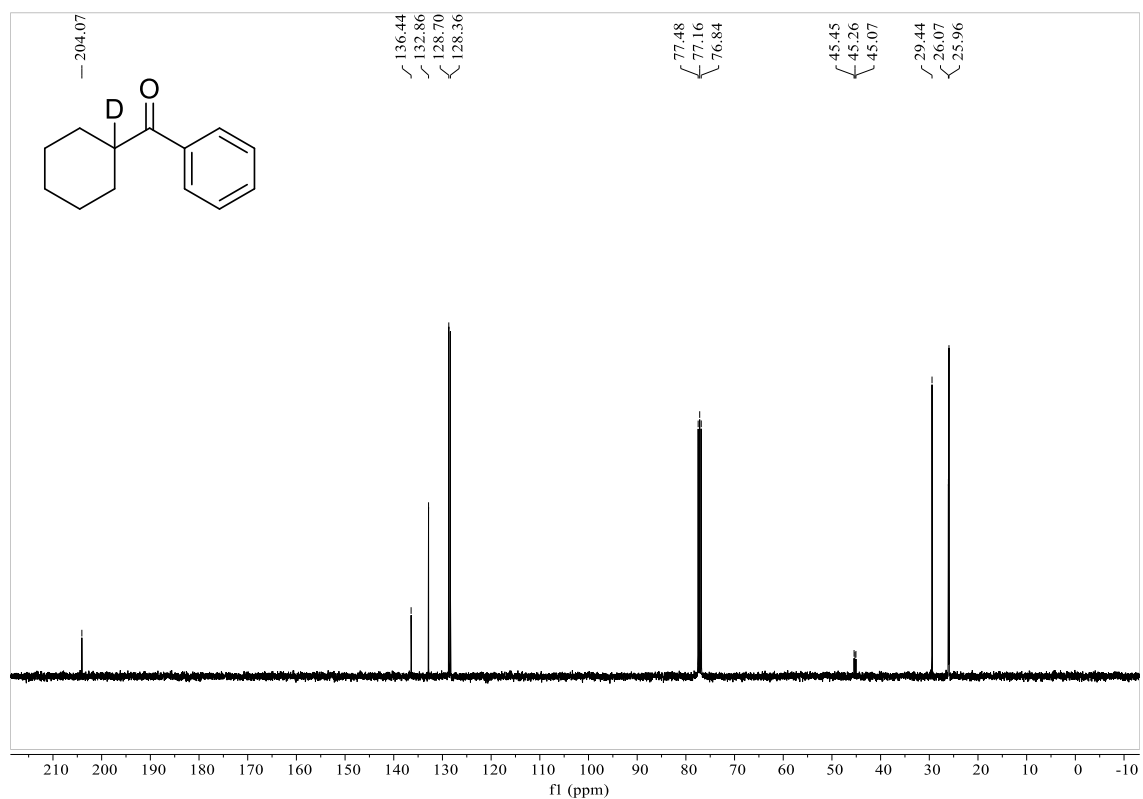
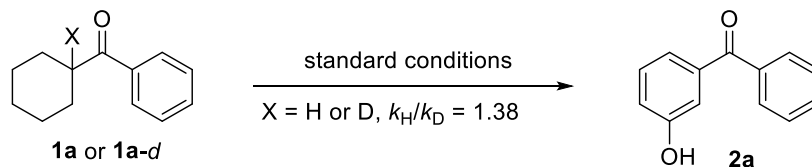


Figure S5. ¹³C NMR spectrum of compound **1a-d** (101 MHz, CDCl₃)



KIE experiment: We use **1a** and deuterated **1a-d** as starting materials and two parallel reactions were carried out. A 15 mL sealed tube containing a magnetic stir bar was charged with **1a** (0.25 mmol) or **1a-d** (0.25 mmol), CuI (4.8 mg, 10 mol%), AgOAc (3.7 mg, 9 mol%) and DMSO (1 mL) sequentially. The tubes were evacuated and backfilled with O₂ three times. Subsequently, H₂O (50 uL, 11.1 equiv) and TFA (186 uL, 10.0 equiv) was added. The tubes were sealed and the mixture was stirred at 90 °C. A 200 uL reaction mixture was taken at 5 h, 7 h, 8 h, 9 h, 12 h. ¹H NMR was taken to determine the amount of **2a** using dibromomethane as the internal standard. The obtained yields were plotted as concentration vs. time (Figure S6) and the following initial rates were calculated.

Table S13. Conversion of the reaction of **1a** and **1a-d**

Reaction time (h)	H-Concentration (M)	D-Concentration (M)
5	0.0015	0.0015
7	0.00375	0.00375
8	0.0055	0.00475
9	0.00625	0.00525
12	0.00925	0.00725

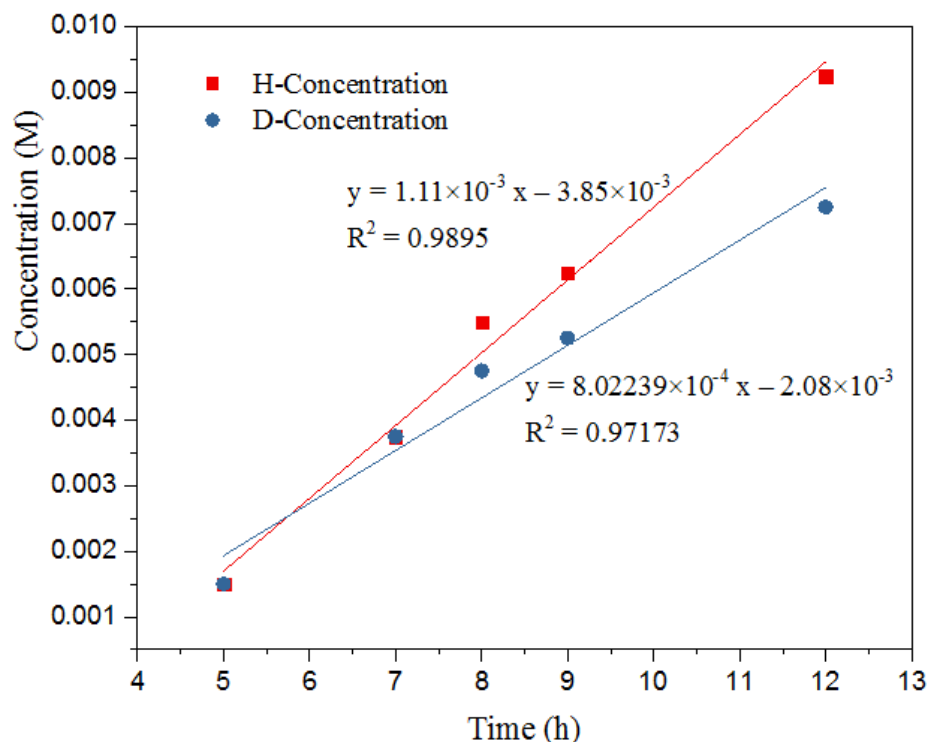
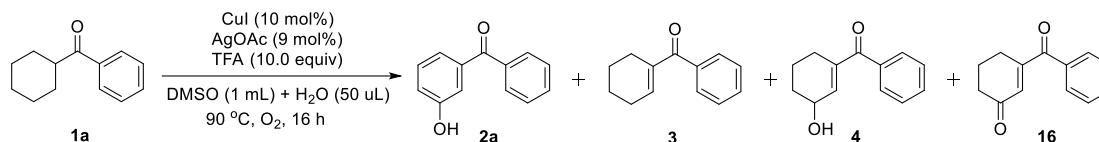


Figure S6. Concentration versus time

5.4 Intermediate trapping experiments and kinetic profiles

5.4.1 Intermediate trapping experiments



A 15 mL sealed tube containing a magnetic stir bar was charged with ketone **1a** (47.0 mg, 0.25 mmol), CuI (4.8 mg, 10 mol%), AgOAc (3.7 mg, 9 mol%) and DMSO (1 mL) sequentially. The tube was evacuated and backfilled with O₂ three times. Subsequently, H₂O (50 uL, 11.1 equiv) and TFA (186 uL, 10.0 equiv) was added. The tube was sealed and the mixture was stirred at 90 °C for 16 h. A 200 uL reaction mixture was taken and immediately diluted with internal standard CH₂Br₂ (1 uL) in DMSO-*d*₆ (400 uL). The reaction mixture was found to contain compounds **2a**, **3**, **4** and **16**. The desired product **2a**, compounds **3**, **4**, and **16** were also isolated.

3: ¹H NMR (400 MHz, CDCl₃) δ 7.79 – 7.72 (m, 2H), 7.65 – 7.58 (m, 1H), 7.57 – 7.50 (m, 2H), 6.73 – 6.68 (m, 1H), 2.59 – 2.51 (m, 2H), 2.43 – 2.35 (m, 2H), 1.91 – 1.76 (m, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 198.4, 144.3, 138.8 (2C), 131.4, 129.3 (2C), 128.1 (2C), 26.2, 24.1, 22.1, 21.8.

HRMS (ESI) *m/z*: [M + Na]⁺ Calcd for C₁₃H₁₄ONa 209.0937; Found 209.0945.

IR: 3058, 2929, 2858, 1643, 1598, 1577, 1446, 1276, 1255, 700 cm⁻¹.

4: ¹H NMR (400 MHz, CDCl₃) δ 7.71 – 7.63 (m, 2H), 7.55 – 7.47 (m, 1H), 7.46 – 7.37 (m, 2H), 6.45 – 6.40 (m, 1H), 4.47 – 4.38 (m, 1H), 2.48 – 2.24 (m, 3H), 2.06 – 1.82 (m, 2H), 1.75 – 1.56 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 198.4, 142.8, 140.0, 137.8, 132.1, 129.4 (2C), 128.3 (2C), 66.3, 31.4, 24.3, 19.2.

HRMS (ESI) *m/z*: [M + H]⁺ Calcd for C₁₃H₁₅O₂ 203.1067; Found 203.1076.

IR: 3392, 3059, 2931, 2861, 1633, 1597, 1576, 1446, 1303, 1265, 1243, 1123, 958, 750 cm⁻¹.

16: ¹H NMR (400 MHz, CDCl₃) δ 7.83 – 7.76 (m, 2H), 7.65 – 7.58 (m, 1H), 7.53 – 7.45 (m, 2H), 6.27 (t, *J* = 1.8 Hz, 1H), 2.71 (td, *J* = 6.0, 1.8 Hz, 2H), 2.59 – 2.51 (m, 2H), 2.22 – 2.13 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 200.3, 197.2, 156.0, 135.7, 133.6, 132.6, 129.8 (2C), 128.8 (2C), 38.0, 25.7, 22.4.

HRMS (ESI) *m/z*: [M + Na]⁺ Calcd for C₁₃H₁₂O₂Na 223.0730; Found 223.0735.

IR: 3057, 2922, 2850, 1677, 1655, 1597, 1448, 1254, 1233, 964, 733, 704 cm⁻¹.

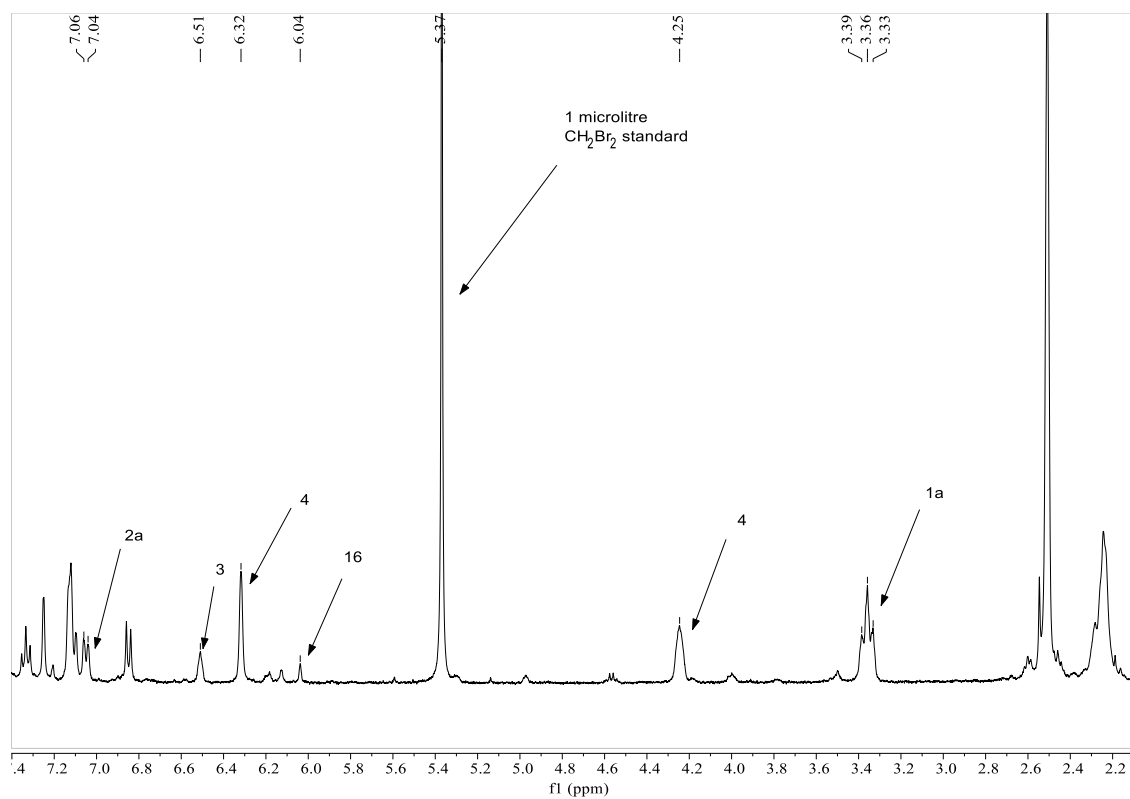


Figure S7. Intermediate trapping experiments

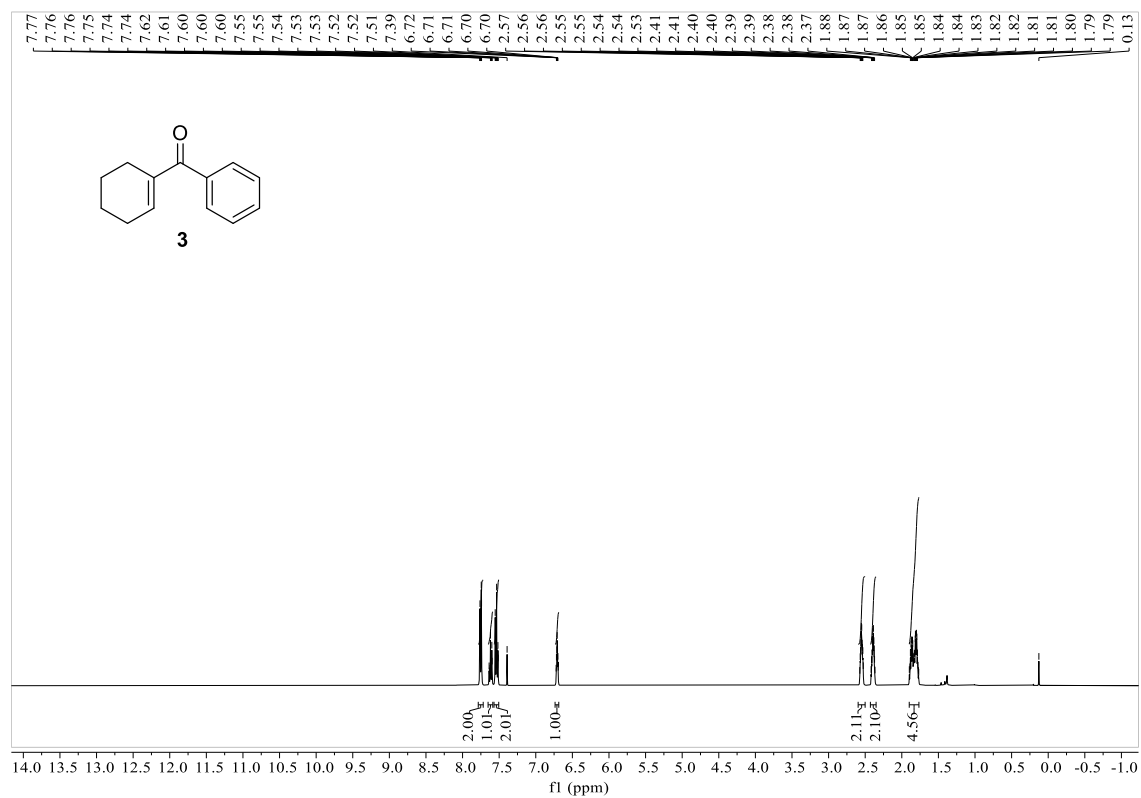


Figure S8. ^1H NMR spectrum of compound **3** (400 MHz, CDCl_3)

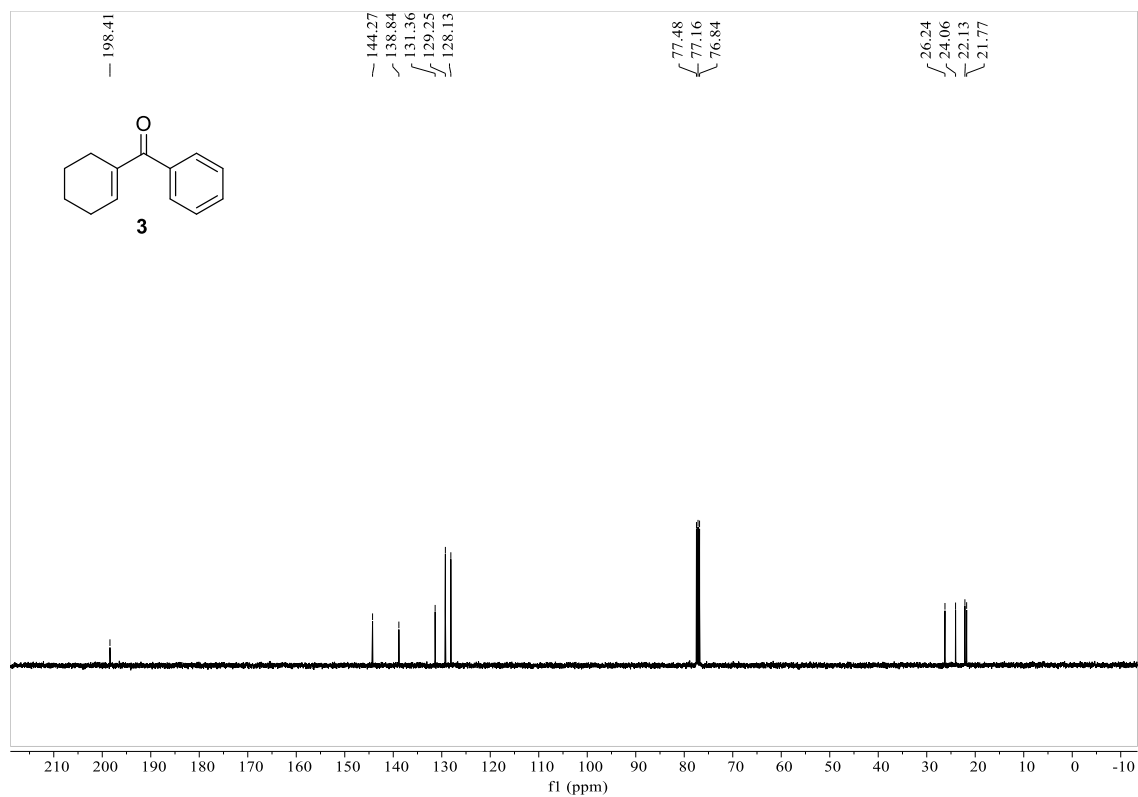


Figure S9. ¹³C NMR spectrum of compound **3** (101 MHz, CDCl₃)

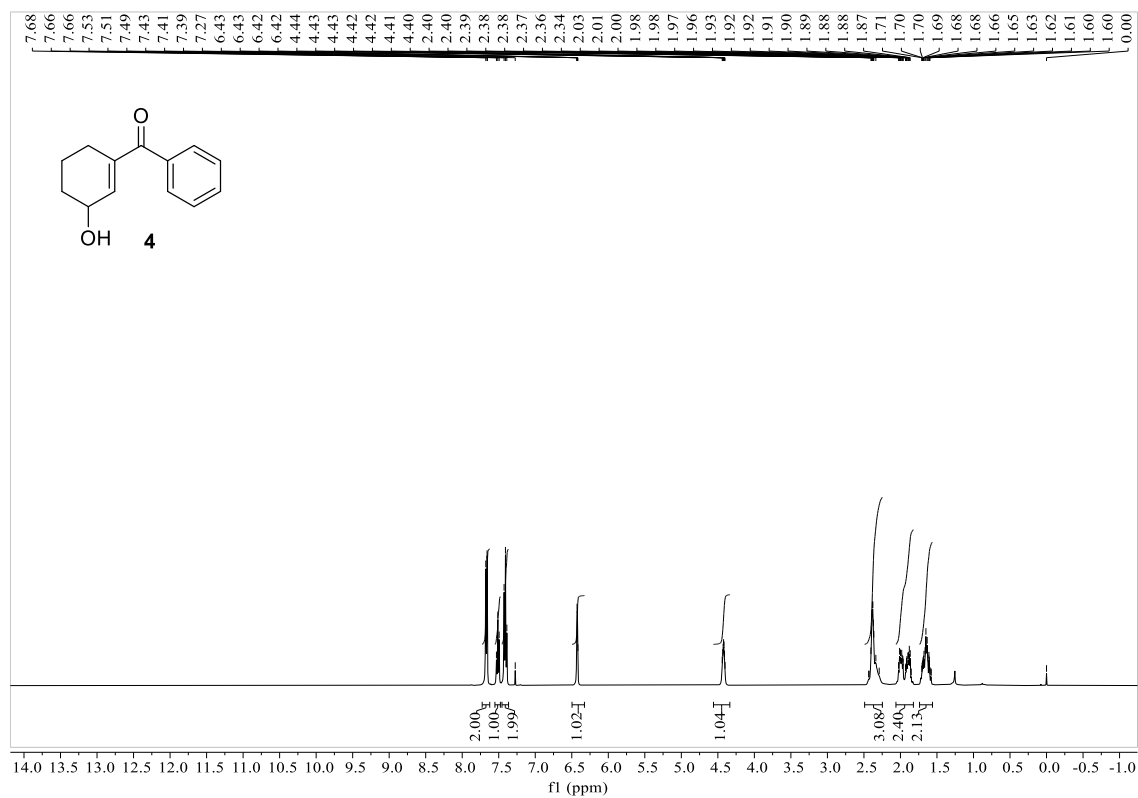


Figure S10. ¹H NMR spectrum of compound **4** (400 MHz, CDCl₃)

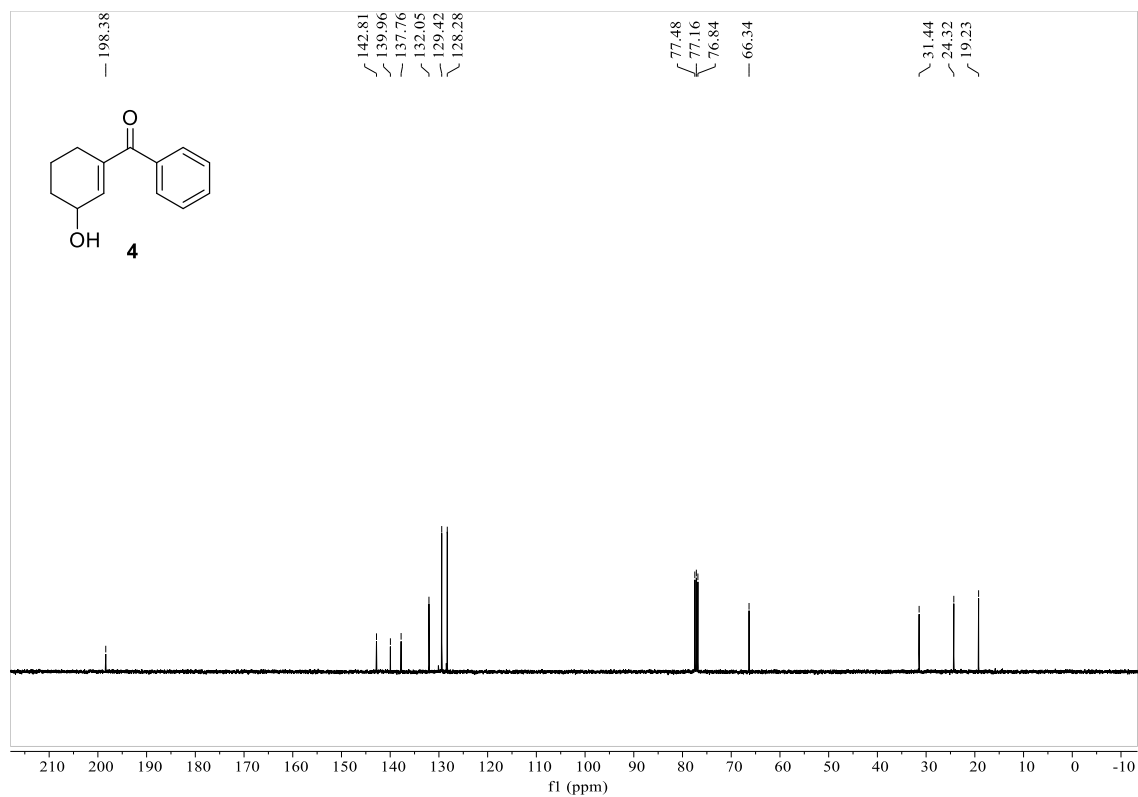


Figure S11. ¹³C NMR spectrum of compound **4** (101 MHz, CDCl₃)

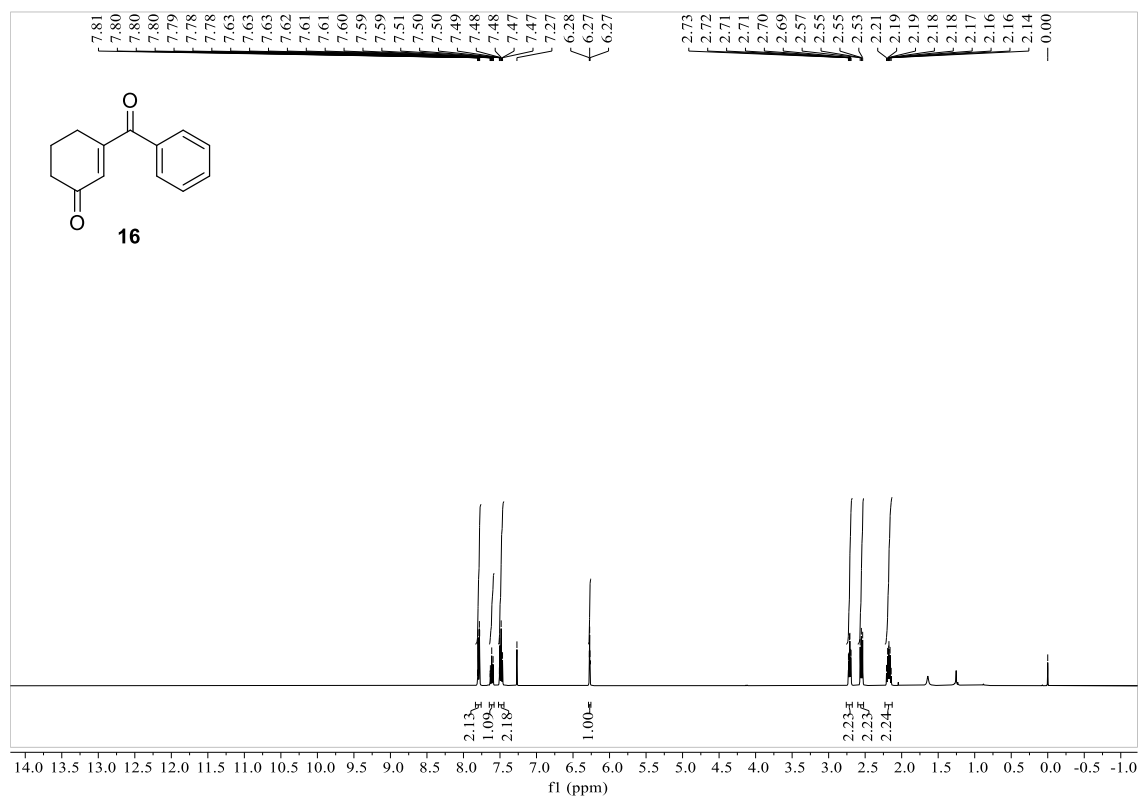


Figure S12. ¹H NMR spectrum of compound **16** (400 MHz, CDCl₃)

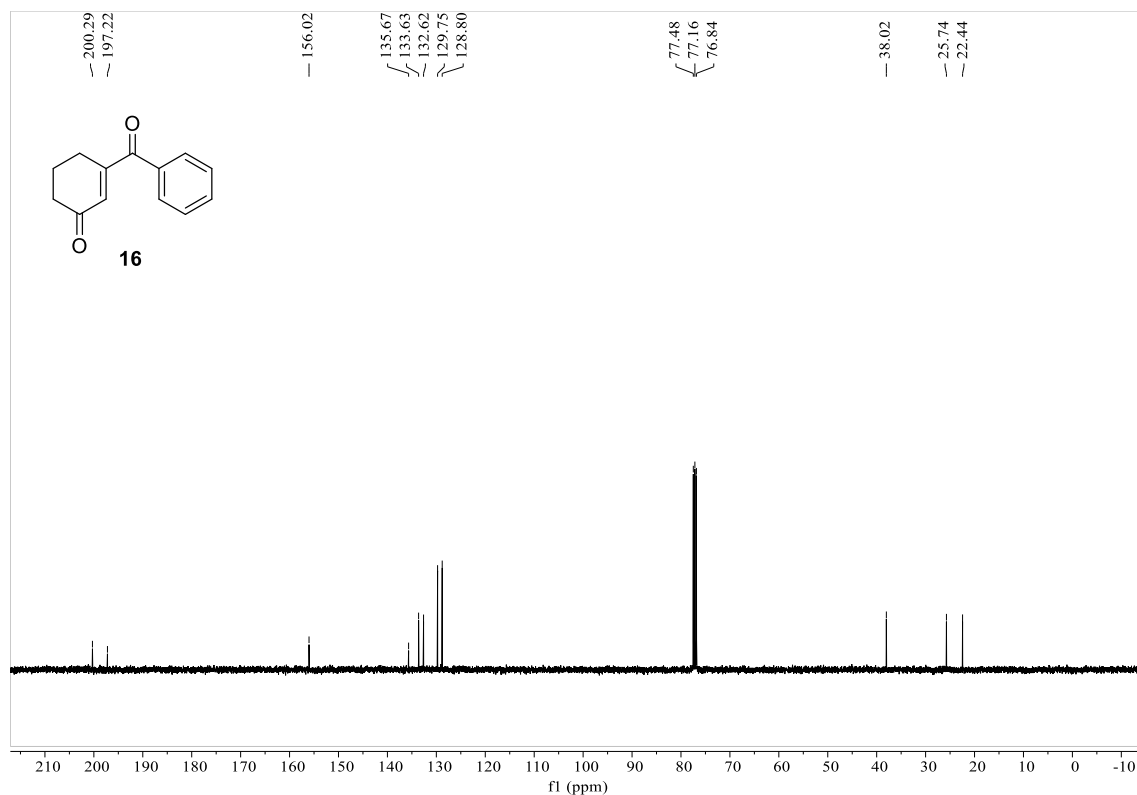
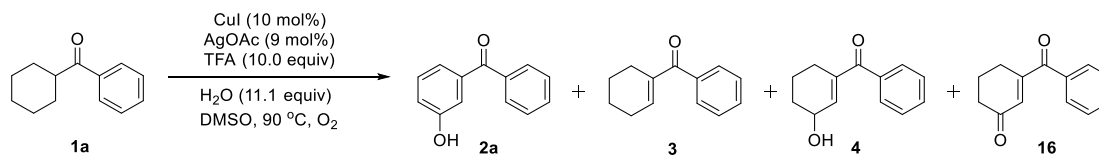


Figure S13. ^{13}C NMR spectrum of compound **16** (101 MHz, CDCl_3)

5.4.2 Kinetic profiles



A 15 mL sealed tube containing a magnetic stir bar was charged with ketones (47.0 mg, 0.25 mmol), CuI (4.8 mg, 10 mol%), AgOAc (3.7 mg, 9 mol%) and DMSO (1 mL) sequentially. The tube was evacuated and backfilled with O_2 three times. Subsequently, H_2O (50 μL , 11.1 equiv) and TFA (186 μL , 10.0 equiv) was added. The tube was sealed and the mixture was stirred at $90\text{ }^\circ\text{C}$. The reaction aliquot was periodically sampled and analyzed by ^1H NMR.

Table S14. Concentration of **1a**, **2a**, **3**, **4** and **16**.

Time (h)	Concentration (M)				
	1a	2a	3	4	16
0	0.25	0	0	0	0
1	0.213		0.01175		
2			0.01675	0.00625	
3	0.176		0.02025	0.01275	
4	0.167		0.02225		
5			0.0215		

6	0.14	0.003	0.01975	0.02575	0.00325
12	0.095	0.01225	0.016	0.035	0.0045
16	0.076	0.026	0.01425	0.0415	0.00525
20	0.062	0.0365	0.012	0.0395	0.005
60	0.0025	0.19	0.00525	0.00375	0

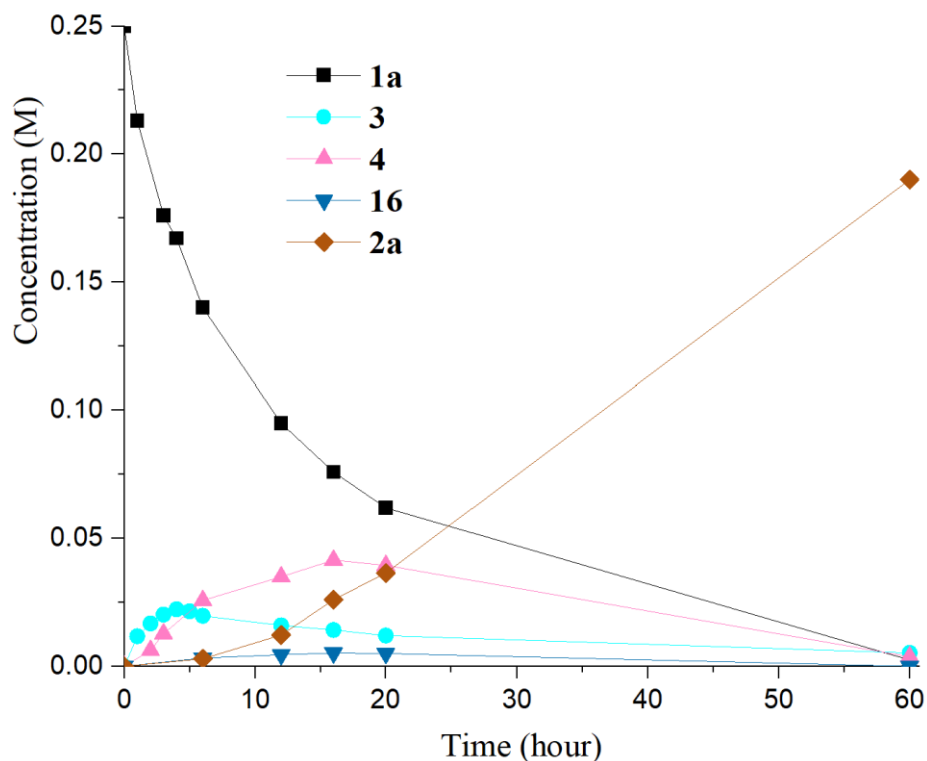
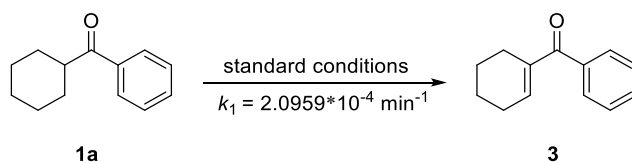


Figure S14. Analysis of the reaction profile.

5.5 Kinetic studies

5.5.1 Determination of kinetic rate constants of the first step: k_1



A 15 mL sealed tube containing a magnetic stir bar was charged with **1a** (47.0 mg, 0.25 mmol), CuI (4.8 mg, 10 mol%), AgOAc (3.7 mg, 9 mol%) and DMSO (1 mL) sequentially. The tube was evacuated and backfilled with O₂ three times. Subsequently, H₂O (50 uL, 11.1 equiv) and TFA (186 uL, 10.0 equiv) was added. The tube was sealed and the mixture was stirred at 90 °C. A 200 uL reaction mixture was taken at 20 min, 30 min, 40 min, 60 min, 90 min, 110 min, 130 min, 150 min. ¹H NMR was taken to determine the amount of **3** using dibromomethane as the internal standard. The obtained yields were plotted as concentration vs. time (Figure S15) and the following initial rates were calculated.

Table S15. Conversion of the reaction of **1a**.

Reaction time (min)	Concentration (M)
20	0.00425
30	0.006
40	0.0085
60	0.012
90	0.01875
110	0.02225
130	0.027
150	0.03175

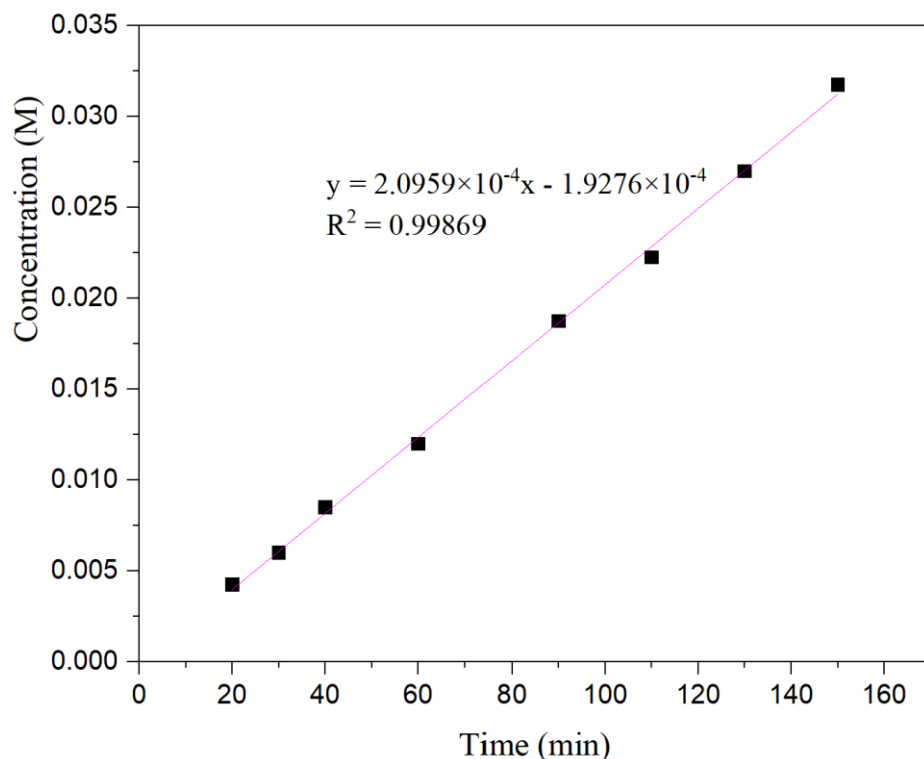
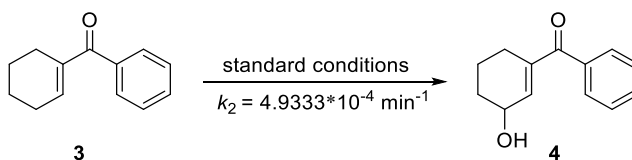


Figure S15. Concentration versus time

5.5.2 Determination of kinetic rate constants of the second step: k_2



A 15 mL sealed tube containing a magnetic stir bar was charged with **3** (46.5 mg, 0.25 mmol), CuI (4.8 mg, 10 mol%), AgOAc (3.7 mg, 9 mol%) and DMSO (1 mL) sequentially. The tube was evacuated and backfilled with O₂ three times. Subsequently, H₂O (50 uL, 11.1 equiv) and TFA (186 uL, 10.0 equiv) was added. The tube was sealed and the mixture was stirred at 90 °C. A 200 uL reaction mixture was taken at 15 min, 30 min, 45 min, 60 min, 75 min. ¹H NMR was

taken to determine the amount of **4** using dibromomethane as the internal standard. The obtained yields were plotted as concentration vs. time (Figure S16) and the following initial rates were calculated.

Table S16. Conversion of the reaction of **3**.

Reaction time (min)	Concentration (M)
15	0.01425
30	0.02275
45	0.03025
60	0.03575
75	0.04475

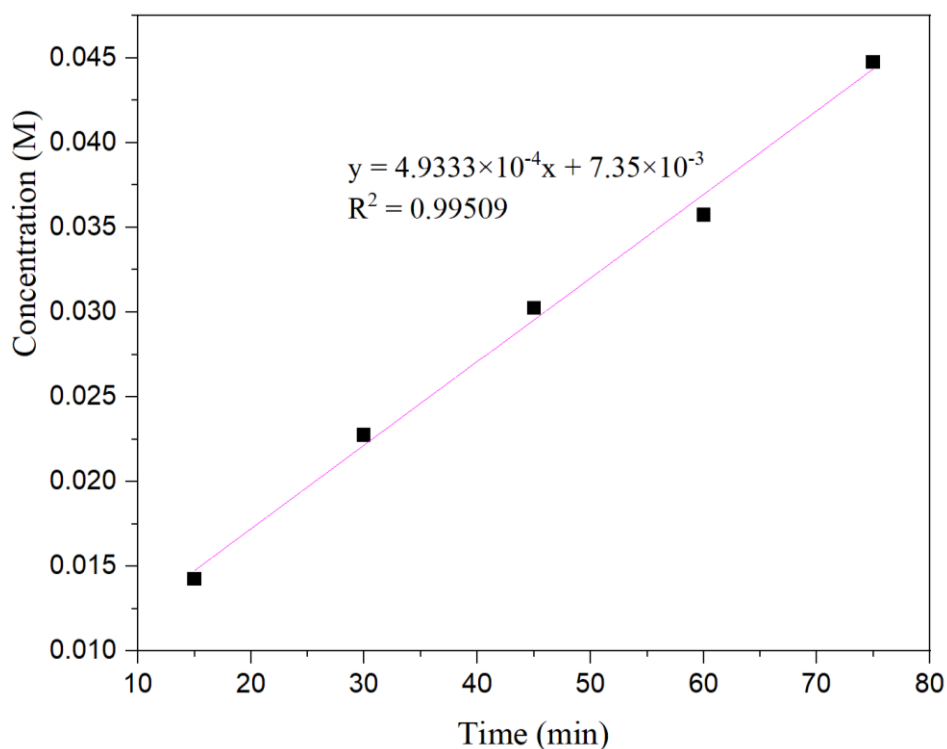
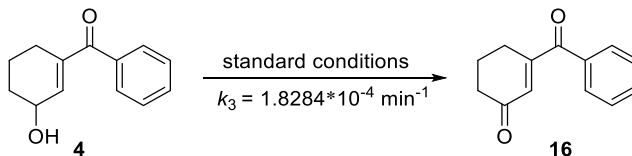


Figure S16. Concentration versus time

5.5.3 Determination of kinetic rate constants of the third step: k_3



A 15 mL sealed tube containing a magnetic stir bar was charged with **4** (50.5 mg, 0.25 mmol), CuI (4.8 mg, 10 mol%), AgOAc (3.7 mg, 9 mol%) and DMSO (1 mL) sequentially. The tube was evacuated and backfilled with O₂ three times. Subsequently, H₂O (50 uL, 11.1 equiv) and

TFA (186 μ L, 10.0 equiv) was added. The tube was sealed and the mixture was stirred at 90 $^{\circ}$ C. A 200 μ L reaction mixture was taken at 30 min, 60 min, 90 min, 120 min, 150 min, 180 min, 210 min, 240 min. 1 H NMR was taken to determine the amount of **16** using dibromomethane as the internal standard. The obtained yields were plotted as concentration vs. time (Figure S17) and the following initial rates were calculated.

Table S17. Conversion of the reaction of **4**.

Reaction time (min)	Concentration (M)
30	0.00475
60	0.009
90	0.01375
120	0.0215
150	0.0265
180	0.032
210	0.03775
240	0.0415

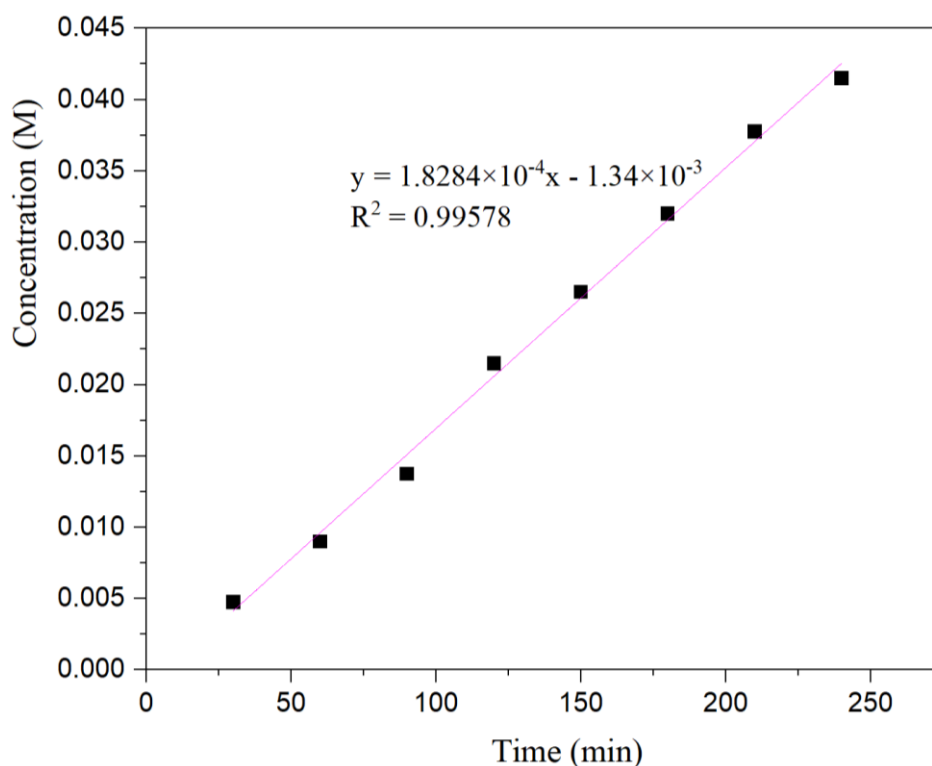
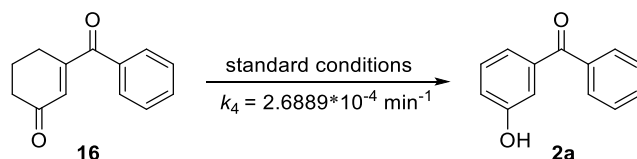


Figure S17. Concentration versus time

5.5.4 Determination of kinetic rate constants of the fourth step: k_4



A 15 mL sealed tube containing a magnetic stir bar was charged with **16** (50 mg, 0.25 mmol), CuI (4.8 mg, 10 mol%), AgOAc (3.7 mg, 9 mol%) and DMSO (1 mL) sequentially. The tube was evacuated and backfilled with O₂ three times. Subsequently, H₂O (50 uL, 11.1 equiv) and TFA (186 uL, 10.0 equiv) was added. The tube was sealed and the mixture was stirred at 90 °C. A 200 uL reaction mixture was taken at 30 min, 45 min, 60 min, 75 min, 90 min, 105 min, 120 min, 135 min, 150min. ¹H NMR was taken to determine the amount of **2a** using dibromomethane as the internal standard. The obtained yields were plotted as concentration vs. time (Figure S18) and the following initial rates were calculated.

Table S18. Conversion of the reaction of **16**.

Reaction time (min)	Concentration (M)
30	0.00725
45	0.01175
60	0.01575
75	0.0185
90	0.02275
105	0.02675
120	0.031
135	0.0355
150	0.04025

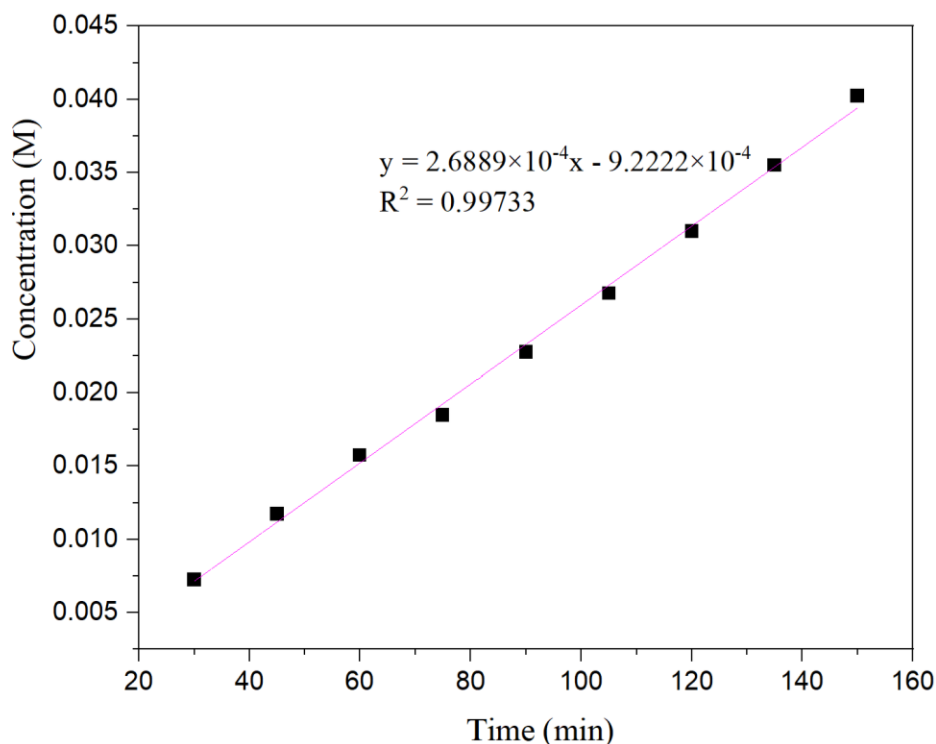
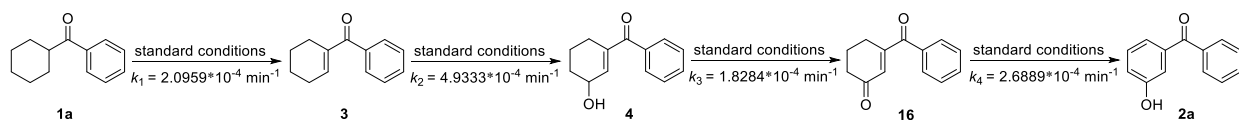


Figure S18. Concentration versus time

The rate constants for each step were determined as follows: $k_1 = 2.0959 \times 10^{-4} \text{ min}^{-1}$, $k_2 = 4.9333 \times 10^{-4} \text{ min}^{-1}$, $k_3 = 1.8284 \times 10^{-4} \text{ min}^{-1}$, $k_4 = 2.6889 \times 10^{-4} \text{ min}^{-1}$, giving a ratio of $k_1/k_2/k_3/k_4 = 1.15 : 2.70 : 1 : 1.47$.



5.6 Proposed mechanism

Based on the experimental results and the related literature, a plausible reaction mechanism for synthesis of *meta*-functionalized phenols or anilines was proposed in Figure S19. First, Cu^{I} species is oxidized in situ by AgOAc in the presence of TFA under oxygen atmosphere, generating $\text{Cu}^{\text{II}}(\text{O}_2\text{CCF}_3)_2$; meanwhile, the ketone suffers from the enolization. The formation of copper(II) enolate followed by the oxidation or the disproportionation gives copper(III) enolate that undergoes β -hydride elimination to deliver the α,β -unsaturated ketone **3** along with a Cu^{III} -hydride intermediate. The Cu^{III} -hydride species eliminates a TFA, resulting in $\text{Cu}^{\text{I}}\text{O}_2\text{CCF}_3$ that is reoxidized to $\text{Cu}^{\text{II}}(\text{O}_2\text{CCF}_3)_2$ by AgOAc and O_2 in the presence of TFA. Subsequently, α,β -unsaturated ketone **3** isomerizes into diene **E**. The terminal $\text{C}=\text{C}$ double bond of diene is activated by $\text{Cu}^{\text{II}}(\text{O}_2\text{CCF}_3)_2$ and then attacked by water generating hydroxycupration intermediate **F** that proceeds the same oxidation or disproportionation and β -hydride elimination to furnish product **4**, simultaneously releasing Cu^{III} -hydride species which proceeds the same process as above to regenerate the active $\text{Cu}^{\text{II}}(\text{O}_2\text{CCF}_3)_2$. **4** is oxidized producing 1,4-enedione **16** which then undergoes the similar procedure like **1a** to **3**, affording the targeted product **2a**.

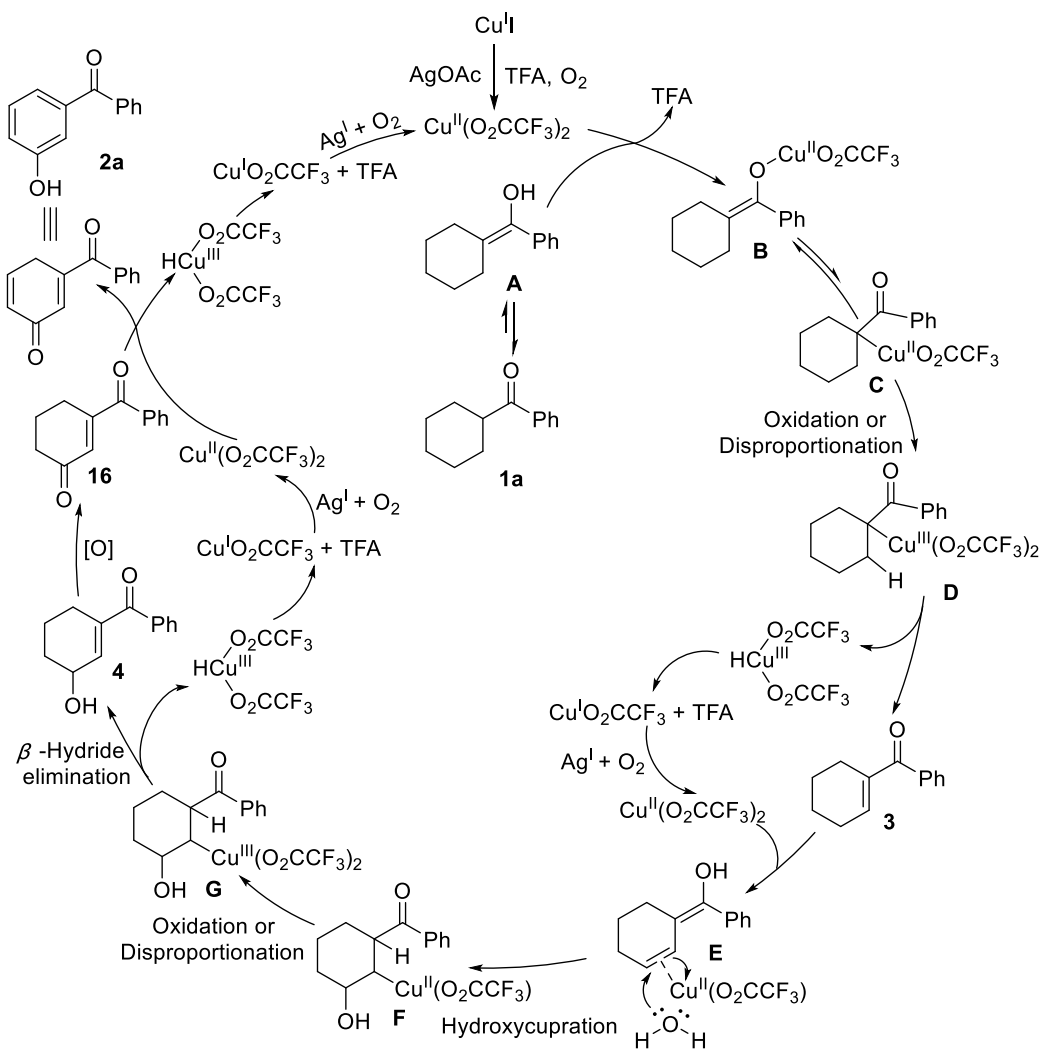


Figure S19. Proposed mechanism.

6. X-Ray Crystallographic Data

The single crystal for compound **2k**, **8a**, **12i** and **12k** were prepared from a mixture solvent of dichloromethane and Petroleum ether (v/v = 1:4). The data were collected on a Bruker Smart APEXIIICCD instrument using Mo-K α radiation ($\lambda = 0.71073$ Å) at 296 K. The crystal structures were solved and refined using the SHELXTL software package. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added in the riding model and refined with isotropic thermal parameters. The crystallographic data have already been deposited at the Cambridge Crystallographic Data Centre. CCDC numbers: 2264711 (**2k**), 2264714 (**8a**), 2264715 (**12i**) and 2264716 (**12k**).

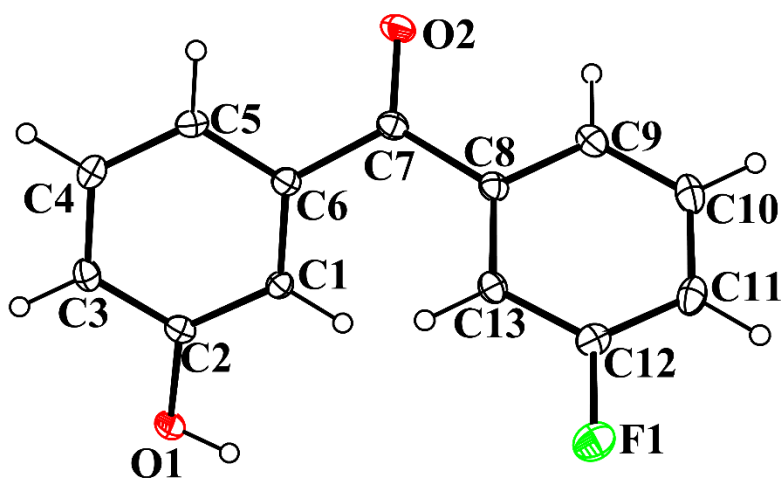


Figure S20. X-ray derived ORTEP of **2k** with thermal ellipsoids shown at the 30% probability level

Table S19. Crystal data and structure refinement for **2k**

Identification code	2k
Empirical formula	C ₁₃ H ₉ F O ₂
Formula weight	216.20
Temperature	150.0 K
Wavelength	1.34139 Å
Crystal system	Orthorhombic
Space group	Pbca
Unit cell dimensions	$a = 7.8303(9)$ Å $\alpha = 90^\circ$ $b = 12.3029(13)$ Å $\beta = 90^\circ$ $c = 20.903(2)$ Å $\gamma = 90^\circ$
Volume	$2013.7(4)$ Å ³
Z	8
Calculated density	1.426 Mg/m ³
Absorption coefficient	0.581 mm ⁻¹

F(000)	896
Crystal size	0.32 x 0.24 x 0.22 mm ³
Theta range for data collection	3.679 to 54.944 °
Index ranges	-8<=h<=9, -14<=k<=14, -25<=l<=25
Reflections collected	19376
Independent reflections	1915 [R(int) = 0.0619]
Completeness to theta = 53.594 °	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7508 and 0.6351
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	1915 / 0 / 146
Goodness-of-fit on F ²	1.110
Final R indices [I>2sigma(I)]	R1 = 0.0454, wR2 = 0.1348
R indices (all data)	R1 = 0.0483, wR2 = 0.1374
Largest diff. peak and hole	0.749 and -0.316 e.Å ⁻³

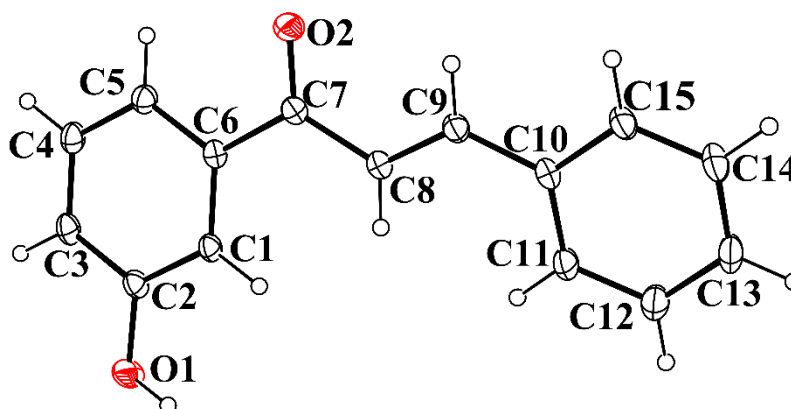


Figure S21. X-ray derived ORTEP of **8a** with thermal ellipsoids shown at the 30% probability level

Table S20. Crystal data and structure refinement for **8a**

Type or paste caption here. Create a page break and paste in the table above the caption.

Identification code	8a
Empirical formula	C15 H12 O2
Formula weight	224.25
Temperature	150.0 K
Wavelength	1.34138 Å
Crystal system	Monoclinic
Space group	P 1 21/c 1
Unit cell dimensions	a = 12.6299(9) Å α = 90 °

	$b = 12.0299(7) \text{ \AA}$ $\beta = 102.285(4)^\circ$
	$c = 7.6700(4) \text{ \AA}$ $\gamma = 90^\circ$
Volume	$1138.67(12) \text{ \AA}^3$
Z	4
Calculated density	1.308 Mg/m^3
Absorption coefficient	0.443 mm^{-1}
F(000)	472
Crystal size	$0.18 \times 0.14 \times 0.12 \text{ mm}^3$
Theta range for data collection	4.465 to 56.949°
Index ranges	$-15 \leq h \leq 15$, $-15 \leq k \leq 14$, $-9 \leq l \leq 8$
Reflections collected	9018
Independent reflections	2286 [R(int) = 0.0635]
Completeness to theta = 53.594°	98.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7512 and 0.3608
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2286 / 0 / 158
Goodness-of-fit on F^2	1.087
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0703$, $wR2 = 0.1805$
R indices (all data)	$R1 = 0.0725$, $wR2 = 0.1846$
Largest diff. peak and hole	0.393 and $-0.421 \text{ e.\AA}^{-3}$

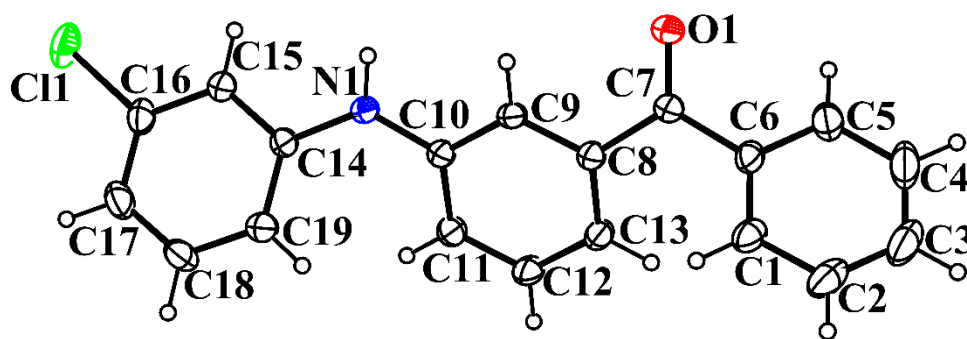


Figure S22. X-ray derived ORTEP of **12i** with thermal ellipsoids shown at the 30% probability level

Table S21. Crystal data and structure refinement for **12i**

Identification code	12i
Empirical formula	$\text{C}_{19} \text{H}_{14} \text{Cl} \text{N} \text{O}$
Formula weight	307.76
Temperature	260.0 K
Wavelength	$1.34139 \text{ \AA}$

Crystal system	Monoclinic
Space group	P 1 21/n 1
Unit cell dimensions	a = 7.4040(6) Å α = 90 ° b = 14.9600(13) Å β = 92.581(4) ° c = 13.7178(12) Å γ = 90 °
Volume	1517.9(2) Å ³
Z	4
Calculated density	1.347 Mg/m ³
Absorption coefficient	1.448 mm ⁻¹
F(000)	640
Crystal size	0.20 x 0.15 x 0.12 mm ³
Theta range for data collection	3.806 to 57.270 °
Index ranges	-9<=h<=9, -18<=k<=18, -16<=l<=17
Reflections collected	12632
Independent reflections	3089 [R(int) = 0.0802]
Completeness to theta = 53.594 °	99.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7512 and 0.4088
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3089 / 0 / 199
Goodness-of-fit on F ²	1.149
Final R indices [I>2sigma(I)]	R1 = 0.0582, wR2 = 0.1464
R indices (all data)	R1 = 0.0695, wR2 = 0.1593
Largest diff. peak and hole	0.333 and -0.353 e.Å ⁻³

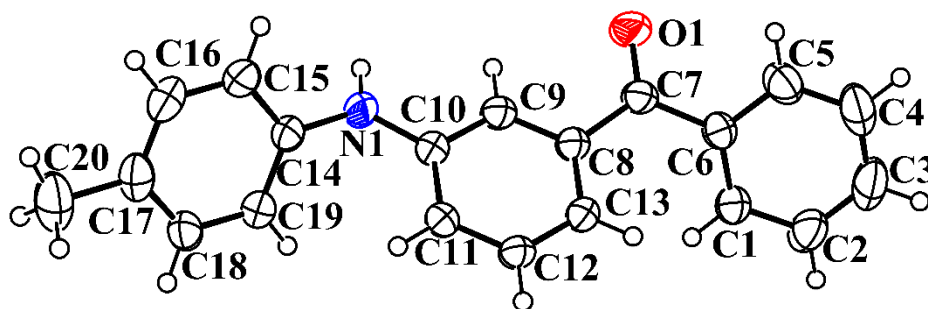


Figure S23. X-ray derived ORTEP of **12k** with thermal ellipsoids shown at the 30% probability level

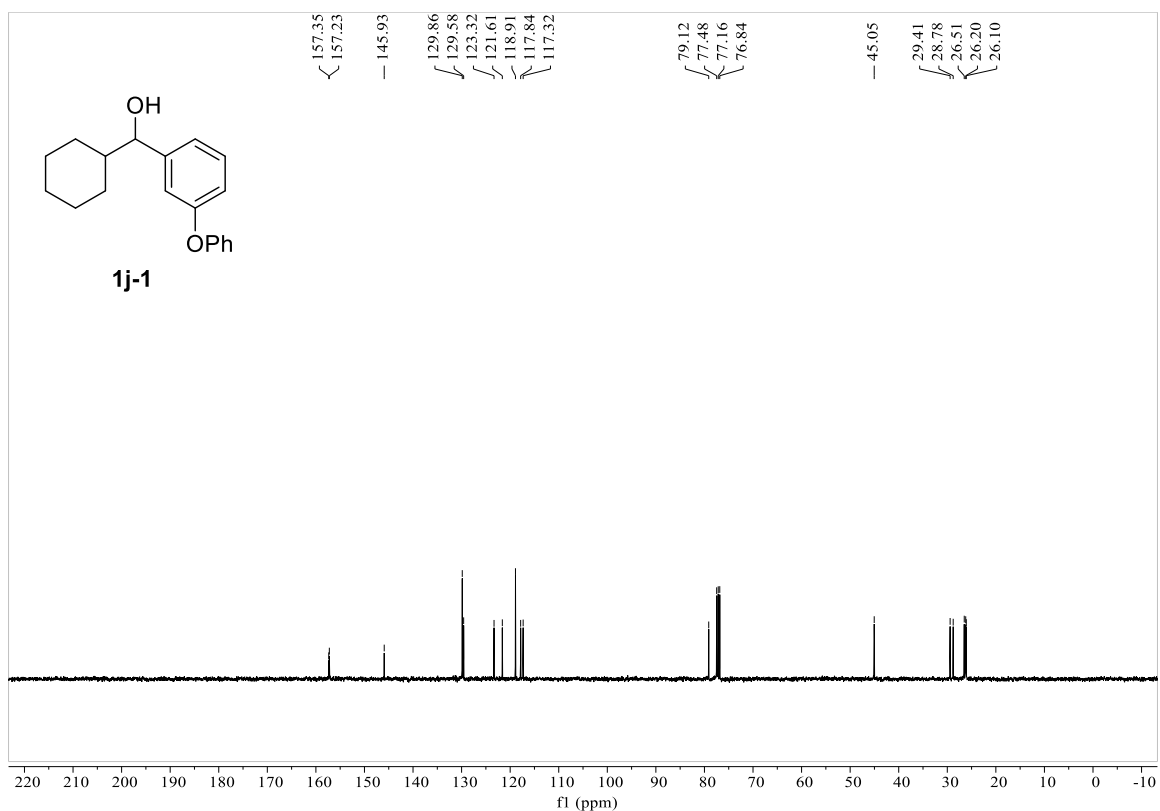
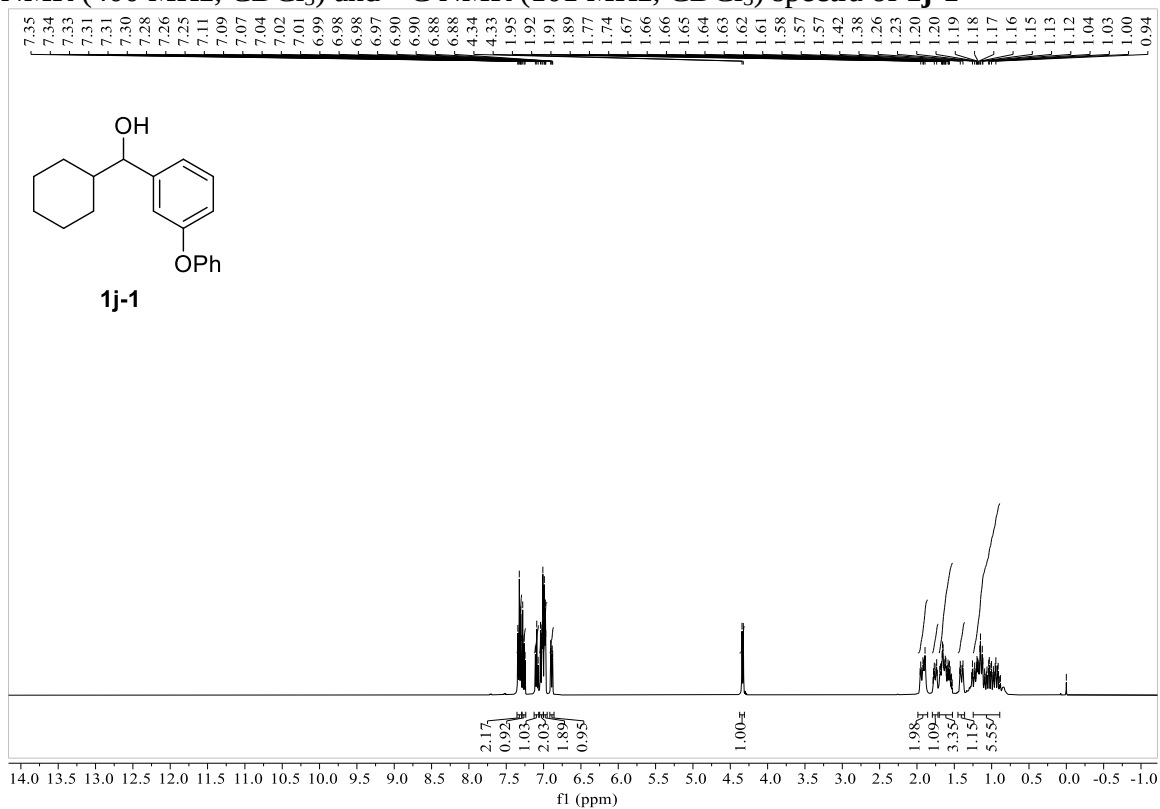
Table S22. Crystal data and structure refinement for **12k**

Identification code	12k
Empirical formula	C ₂₀ H ₁₇ N O

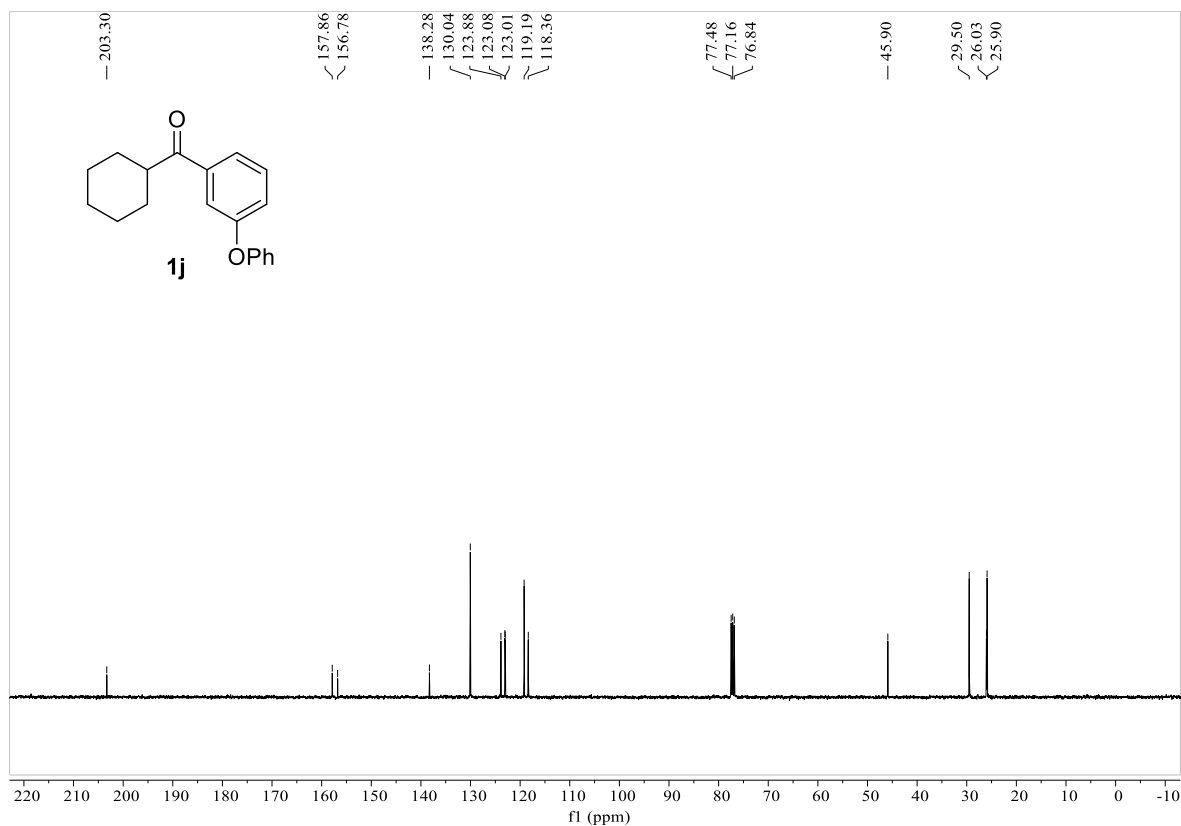
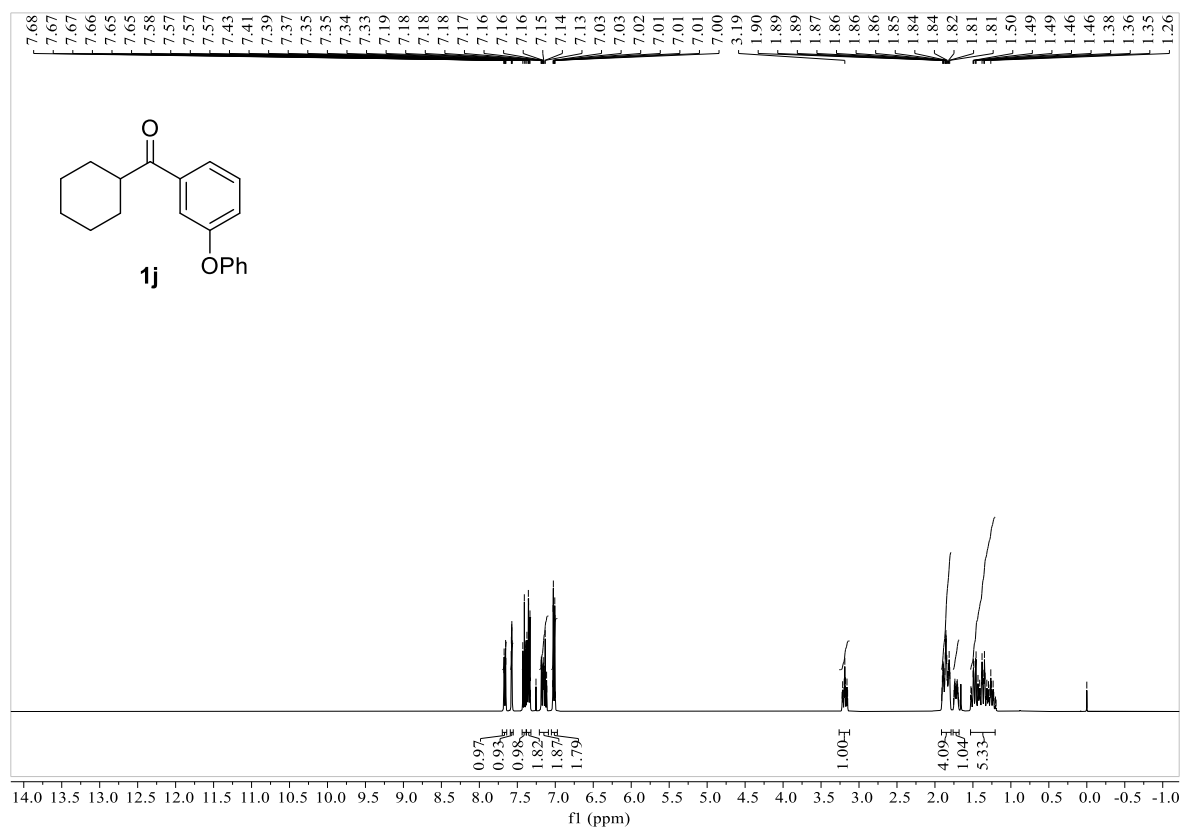
Formula weight	287.36
Temperature	260.0 K
Wavelength	1.34139 Å
Crystal system	Monoclinic
Space group	P 1 21/n 1
Unit cell dimensions	a = 15.8980(8) Å α = 90 ° b = 7.6272(4) Å β = 94.789(2) ° c = 25.4492(12) Å γ = 90 °
Volume	3075.1(3) Å ³
Z	4
Calculated density	1.241 Mg/m ³
Absorption coefficient	0.382 mm ⁻¹
F(000)	1216
Crystal size	0.21 x 0.20 x 0.19 mm ³
Theta range for data collection	3.723 to 57.098 °
Index ranges	-19<= <i>h</i> <=19, -7<= <i>k</i> <=9, -31<= <i>l</i> <=30
Reflections collected	34502
Independent reflections	6283 [R(int) = 0.0773]
Completeness to theta = 53.594 °	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7512 and 0.5284
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	6283 / 0 / 399
Goodness-of-fit on F ²	1.042
Final R indices [I>2sigma(I)]	R1 = 0.0544, wR2 = 0.1429
R indices (all data)	R1 = 0.0901, wR2 = 0.1645
Largest diff. peak and hole	0.314 and -0.288 e.Å ⁻³

7. NMR Spectra

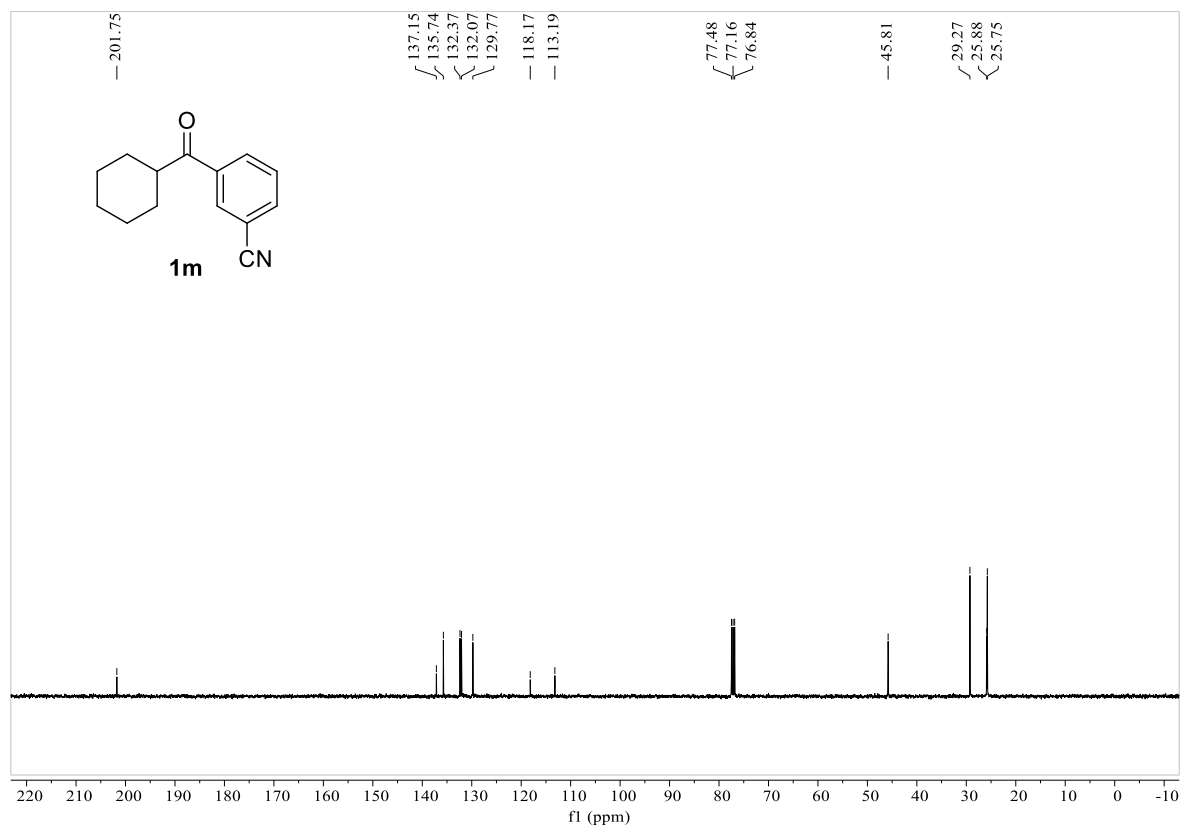
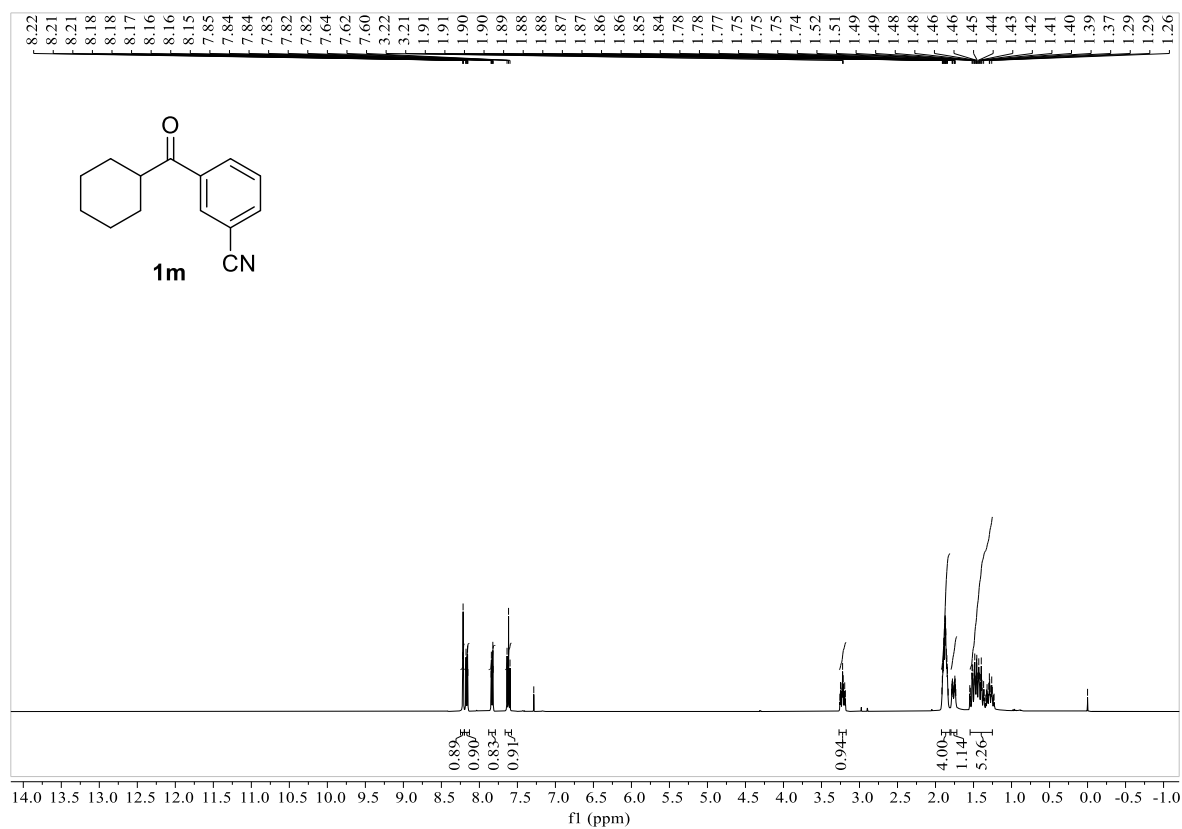
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1j-1**



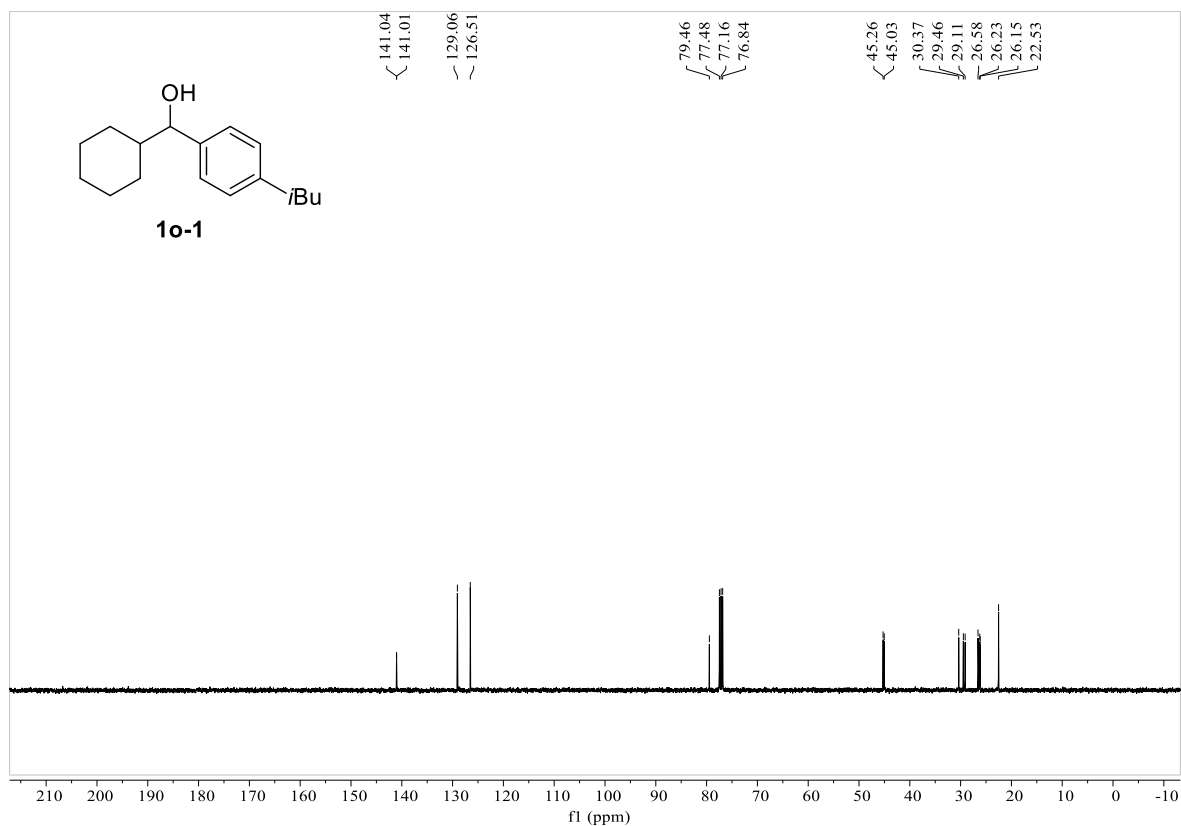
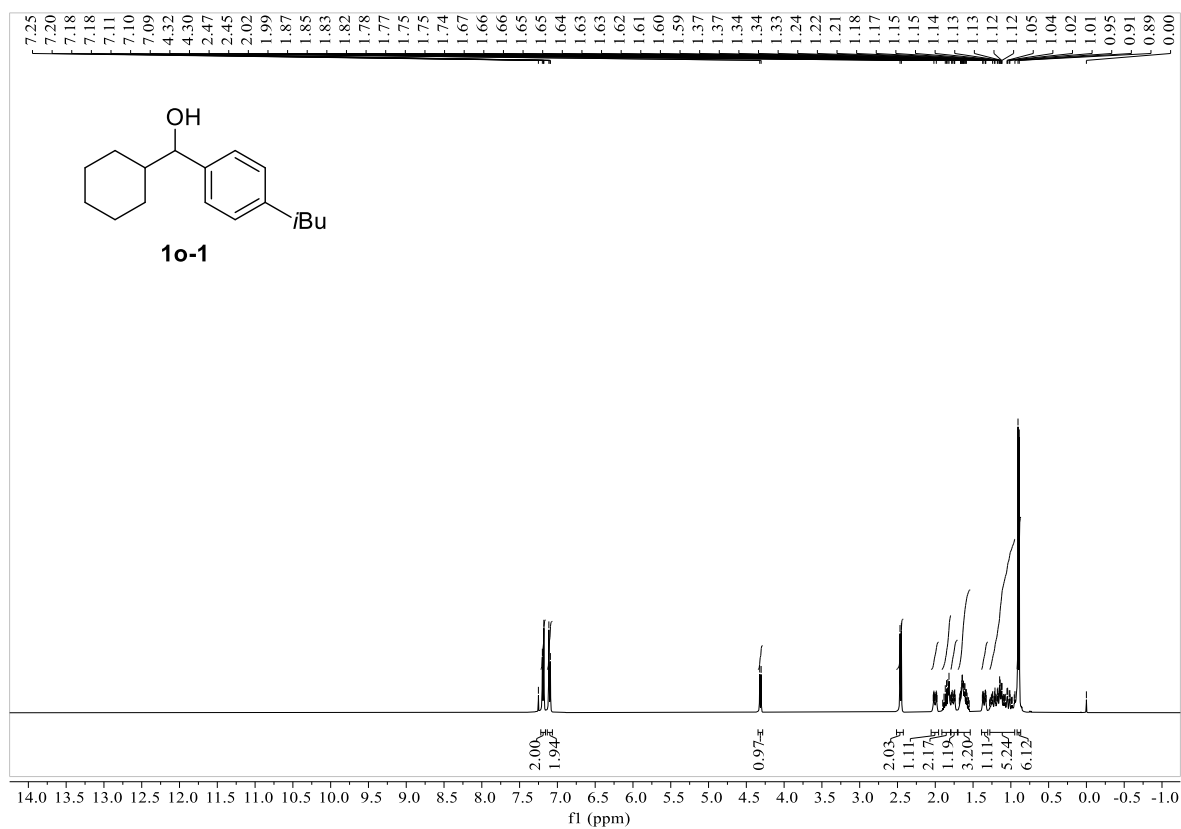
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1j**



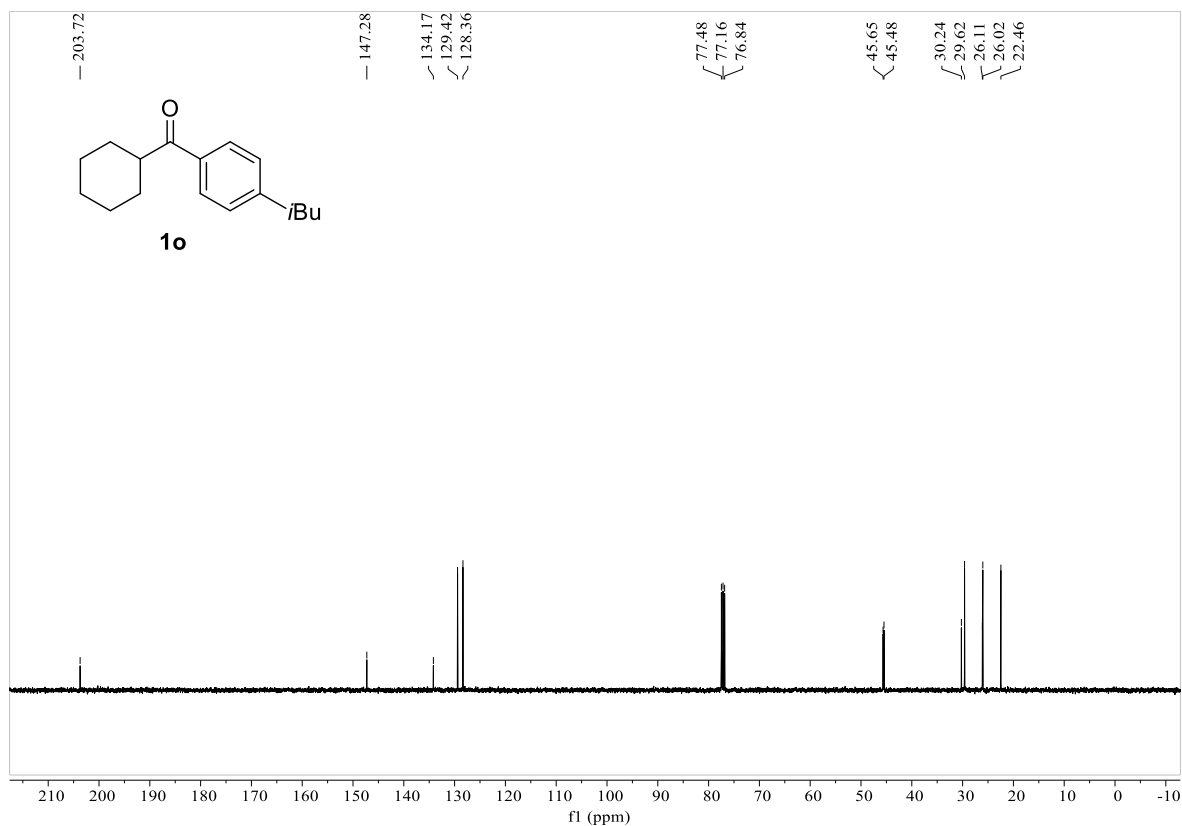
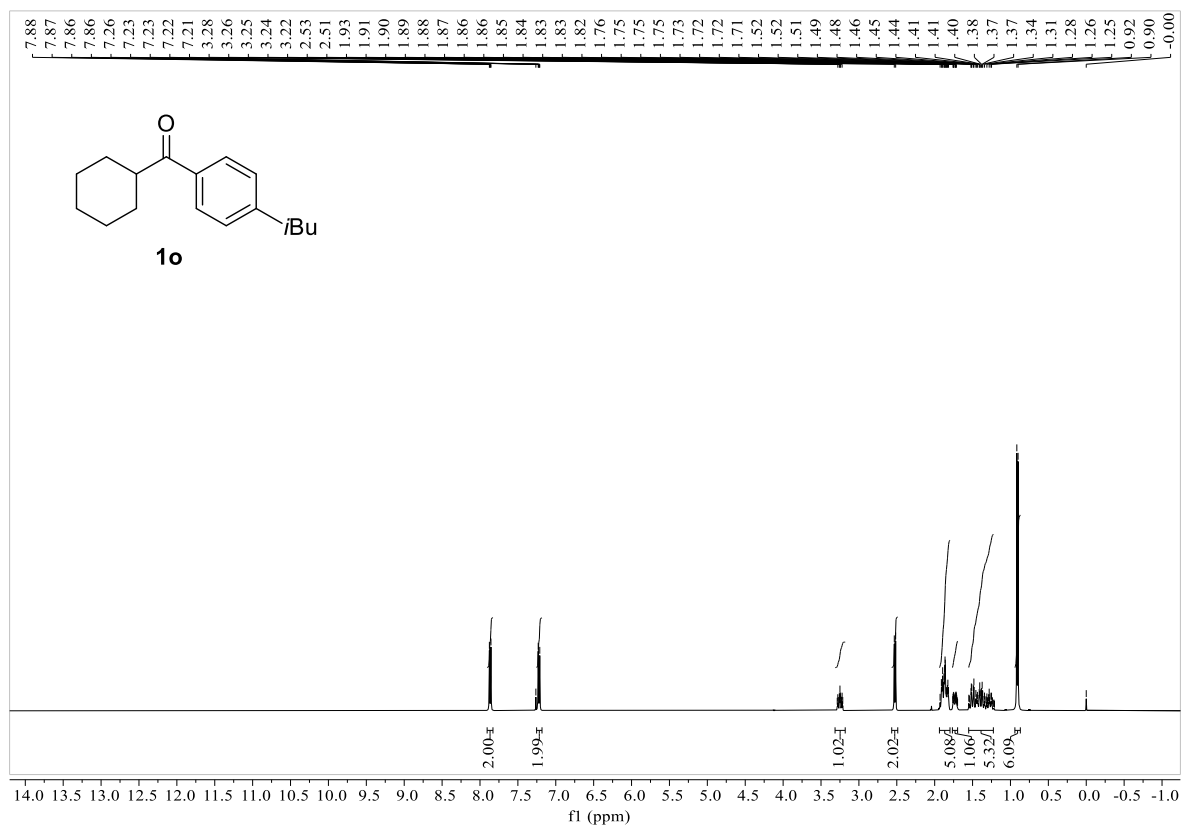
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1m**



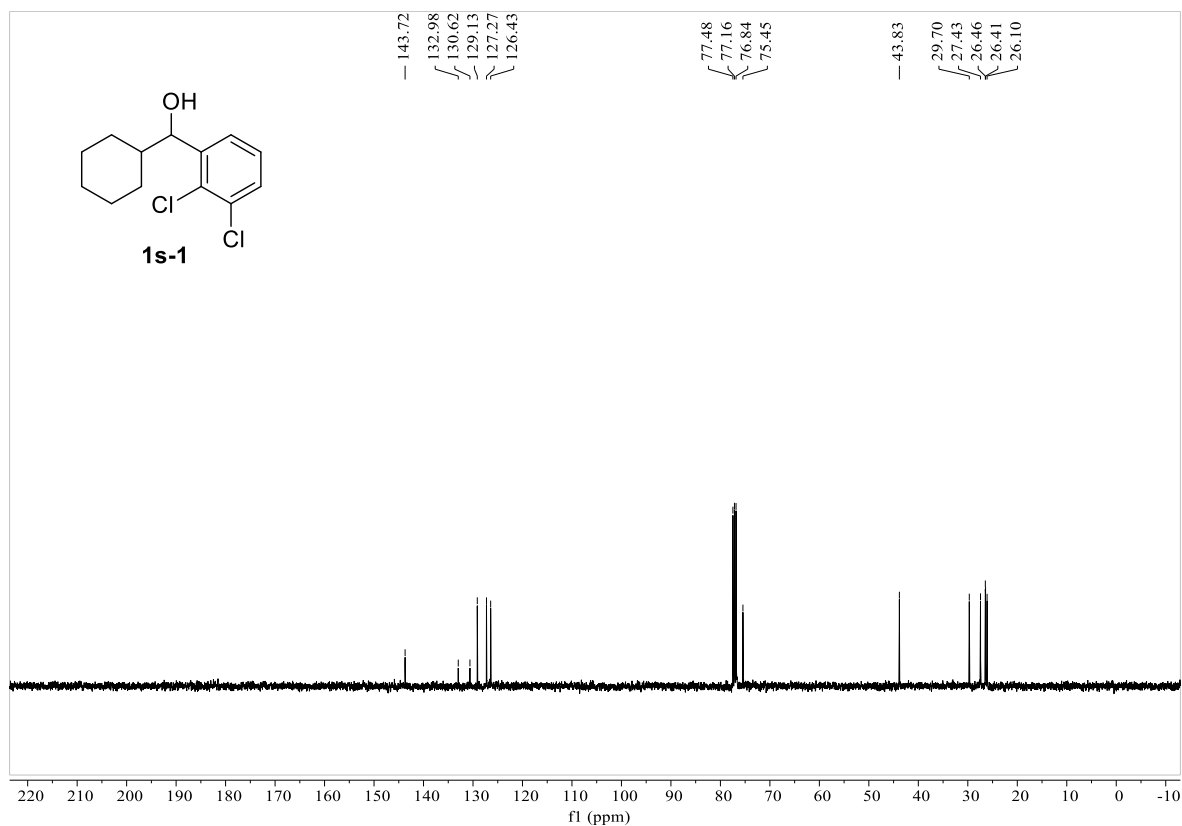
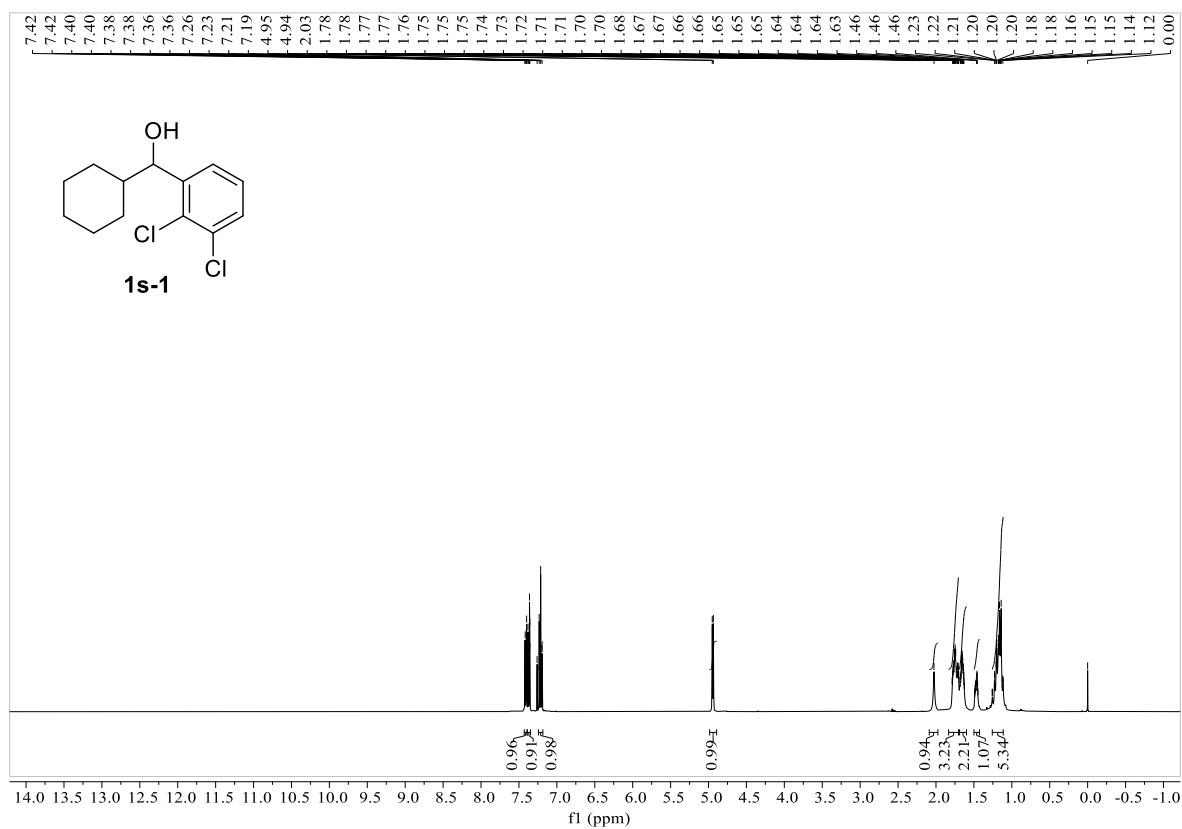
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1o-1**



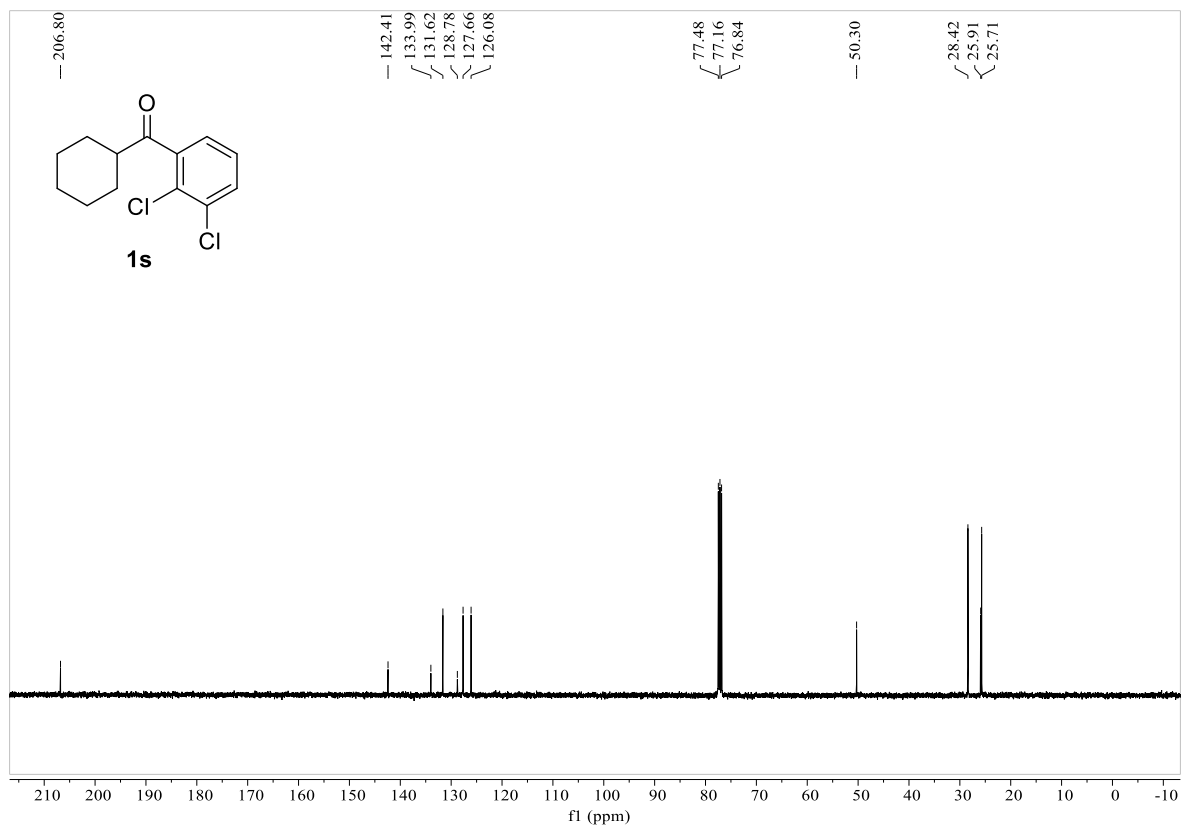
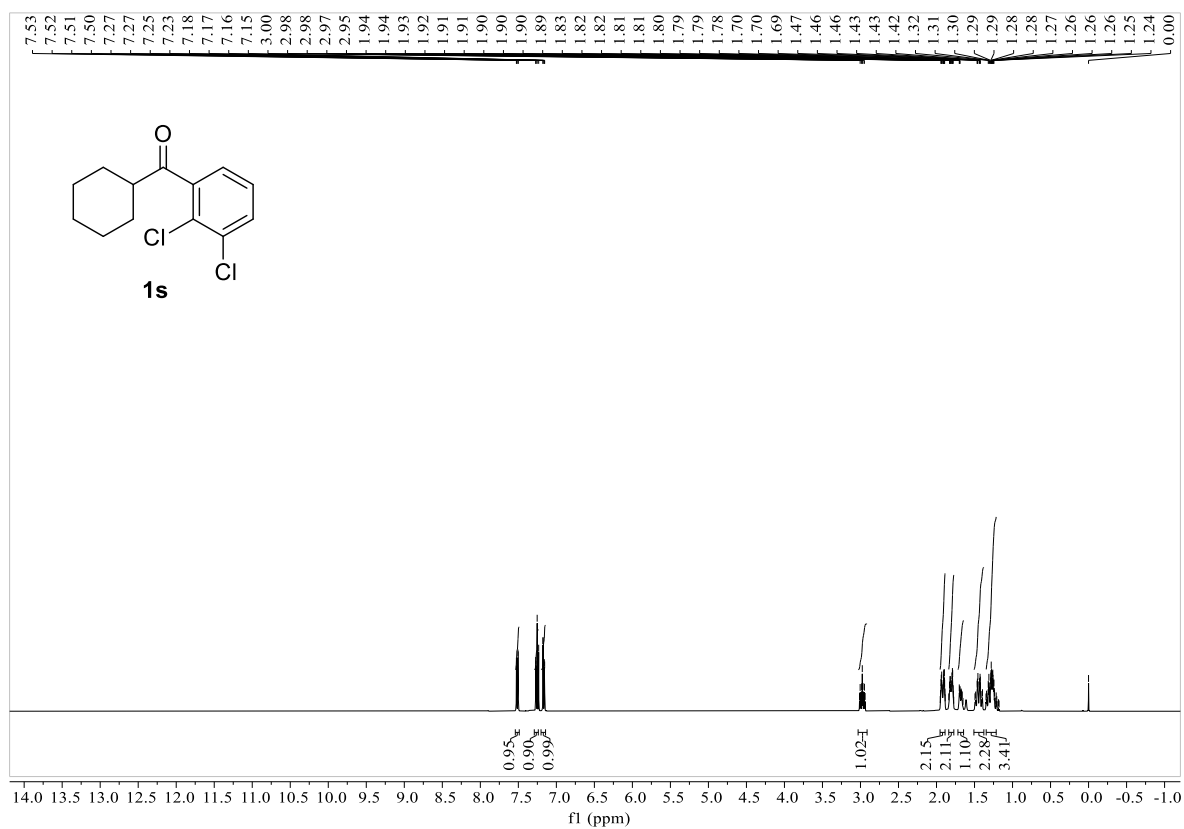
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1o**



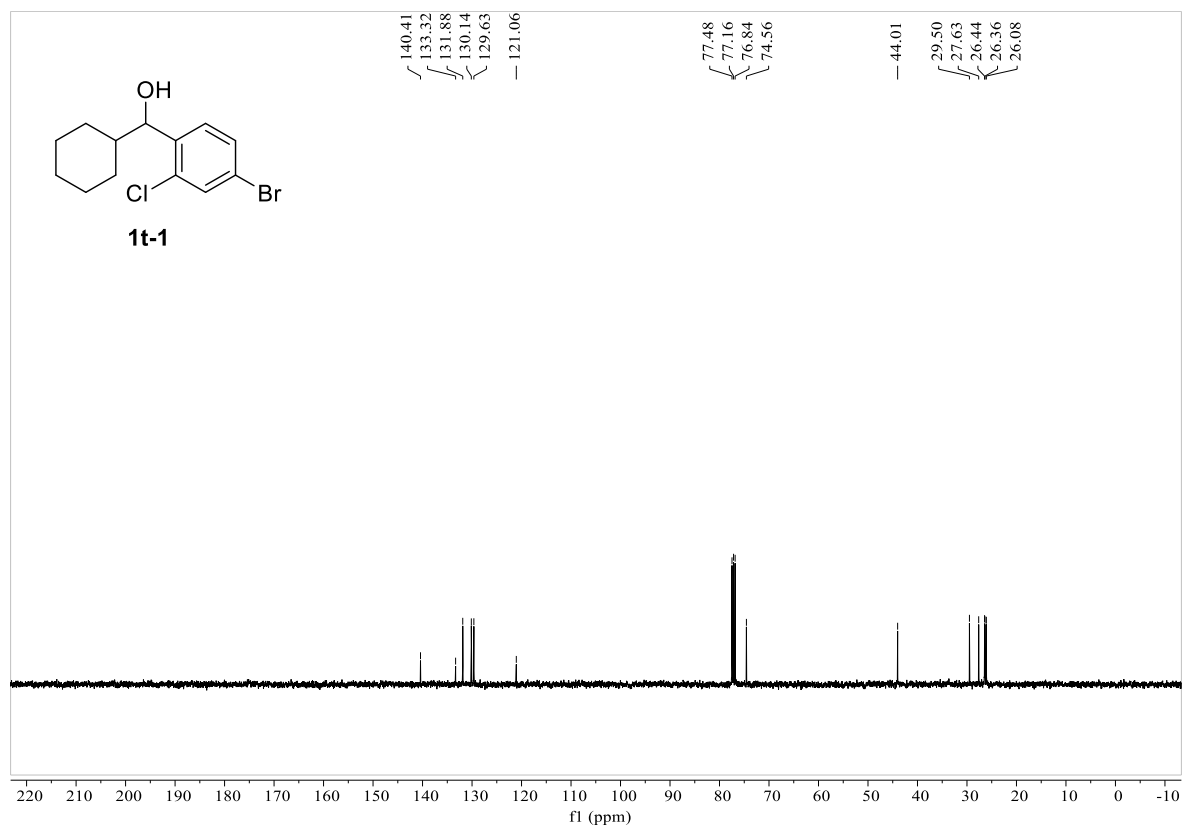
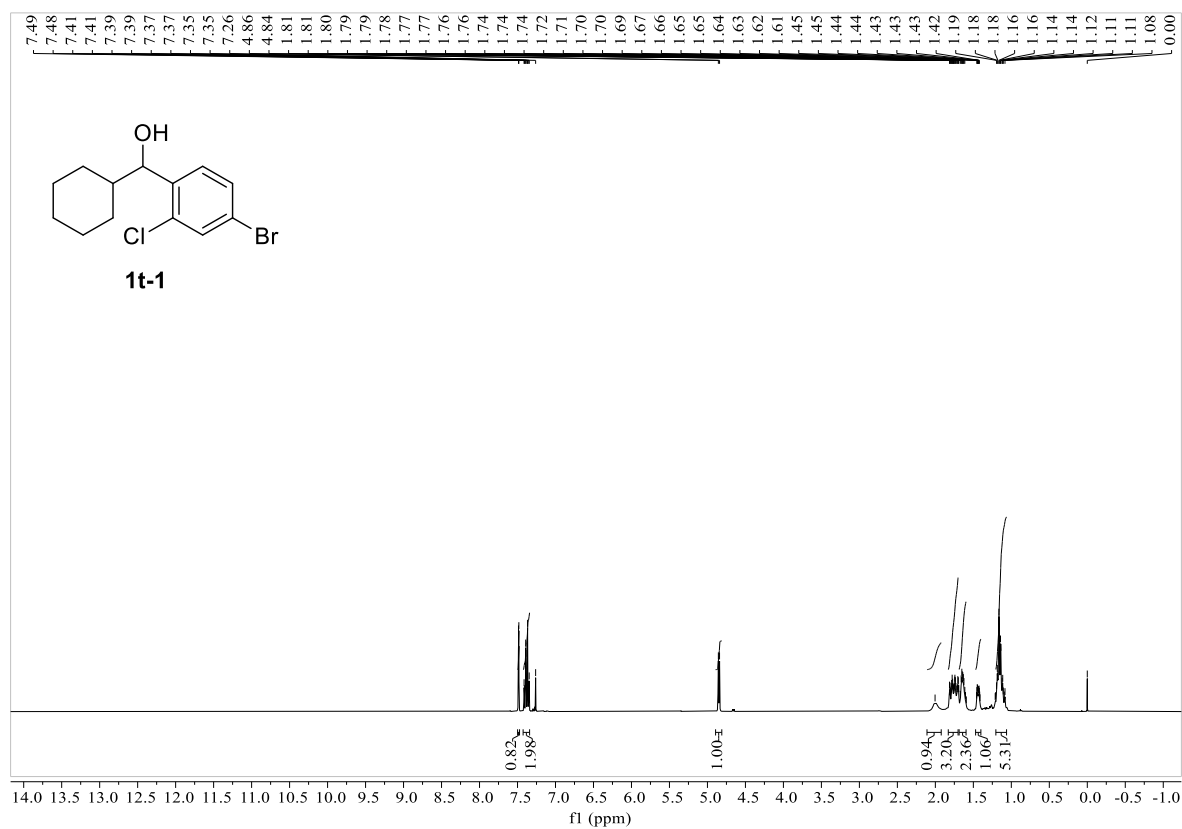
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1s-1**



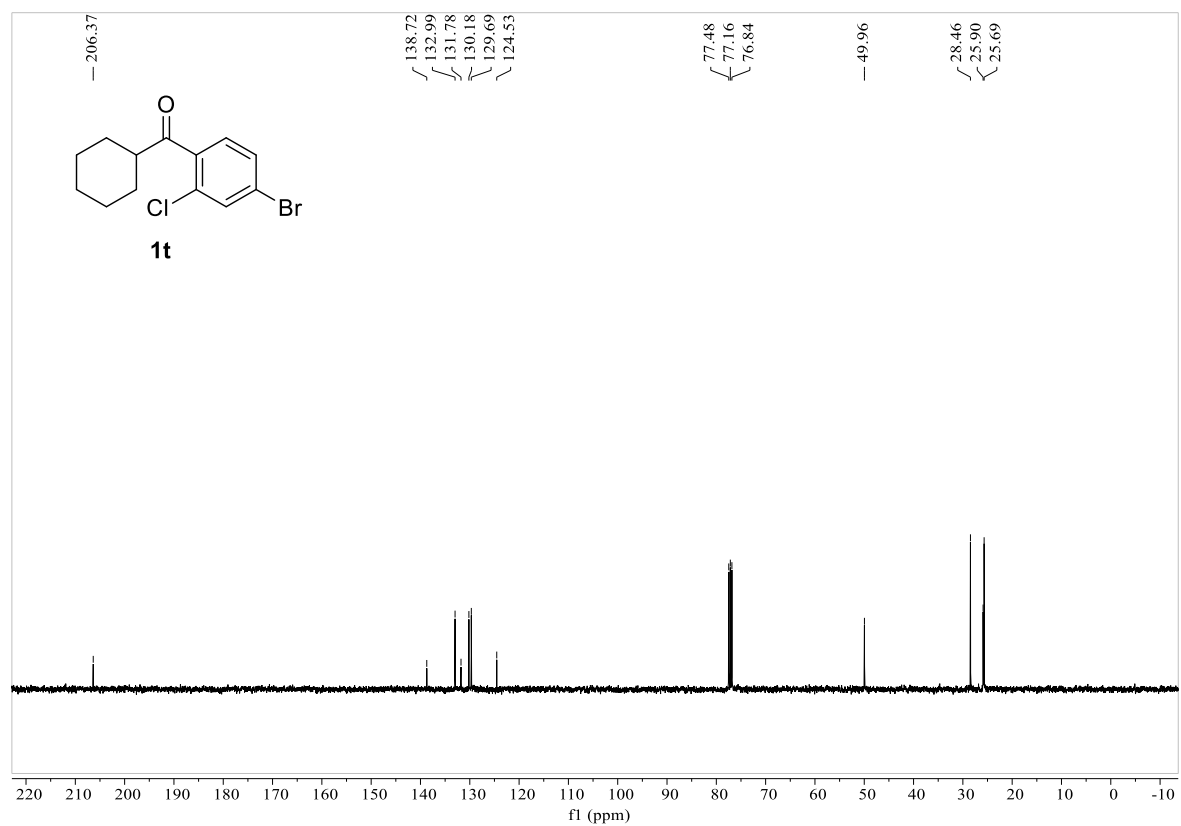
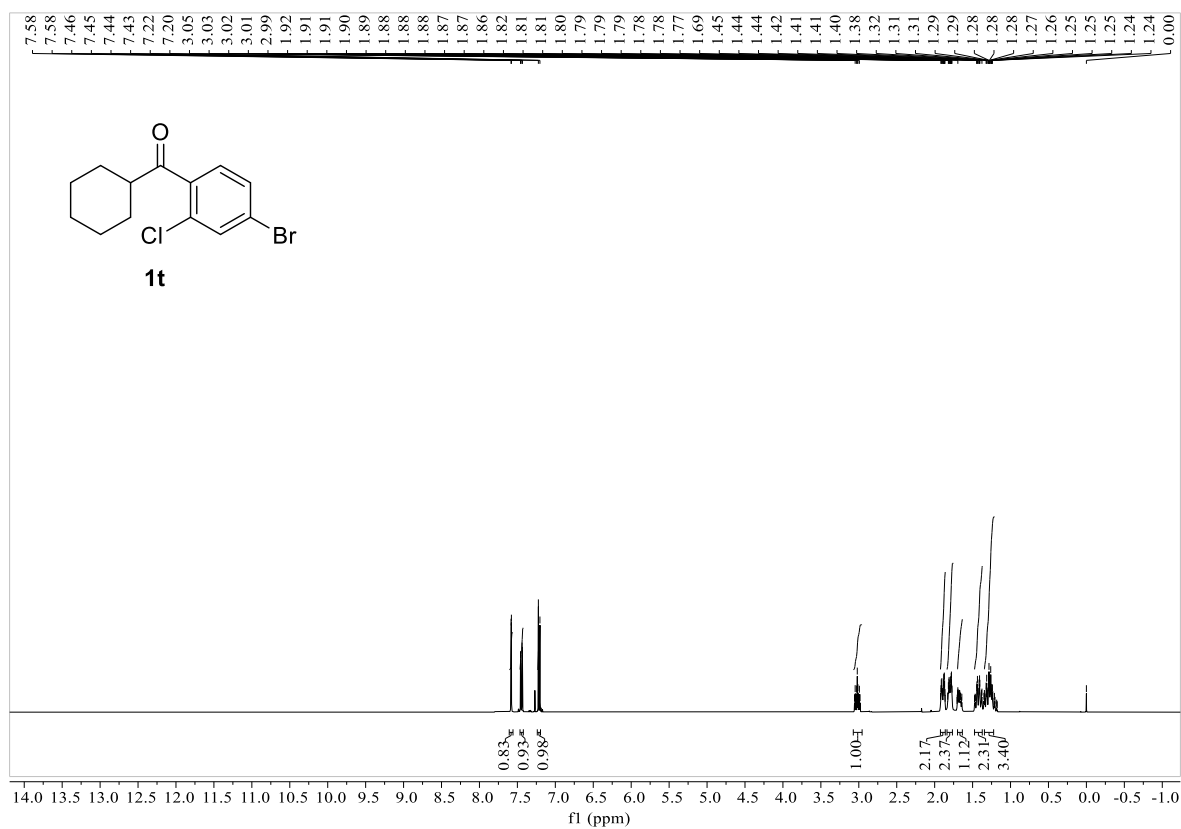
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1s**



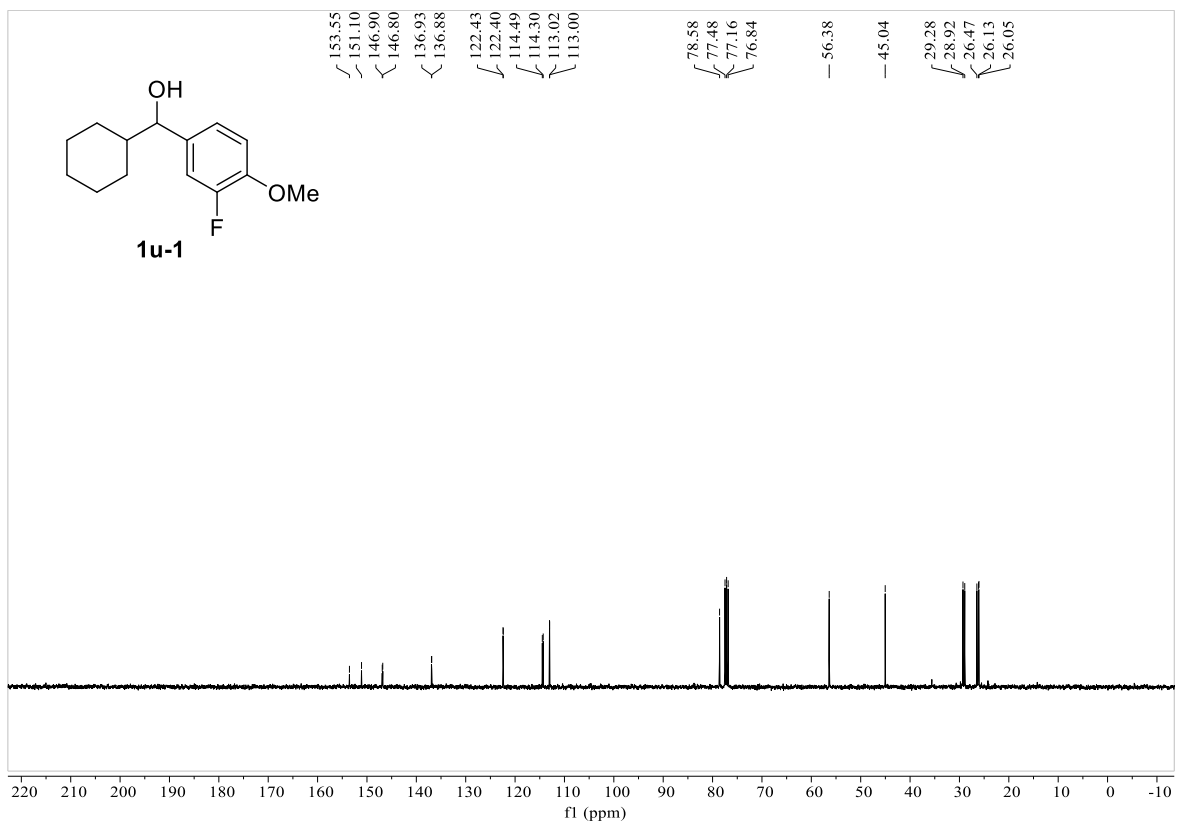
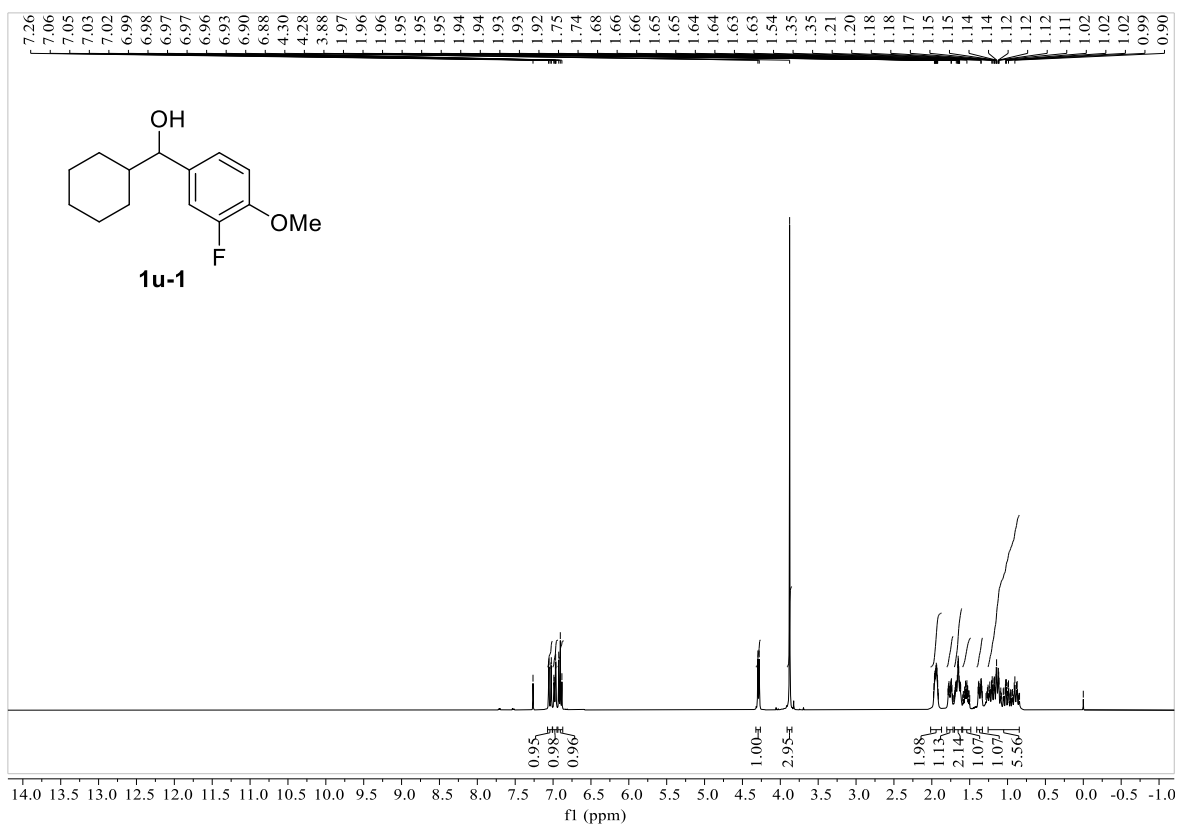
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1t-1**



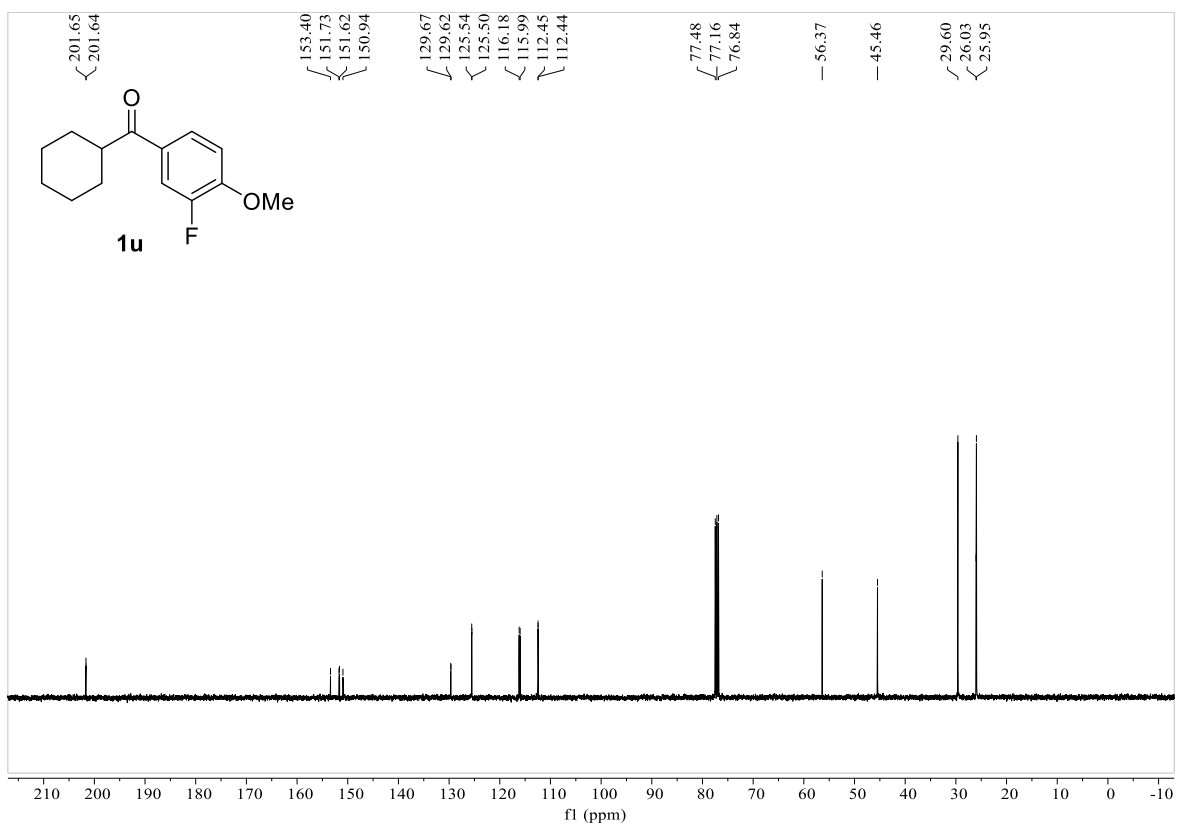
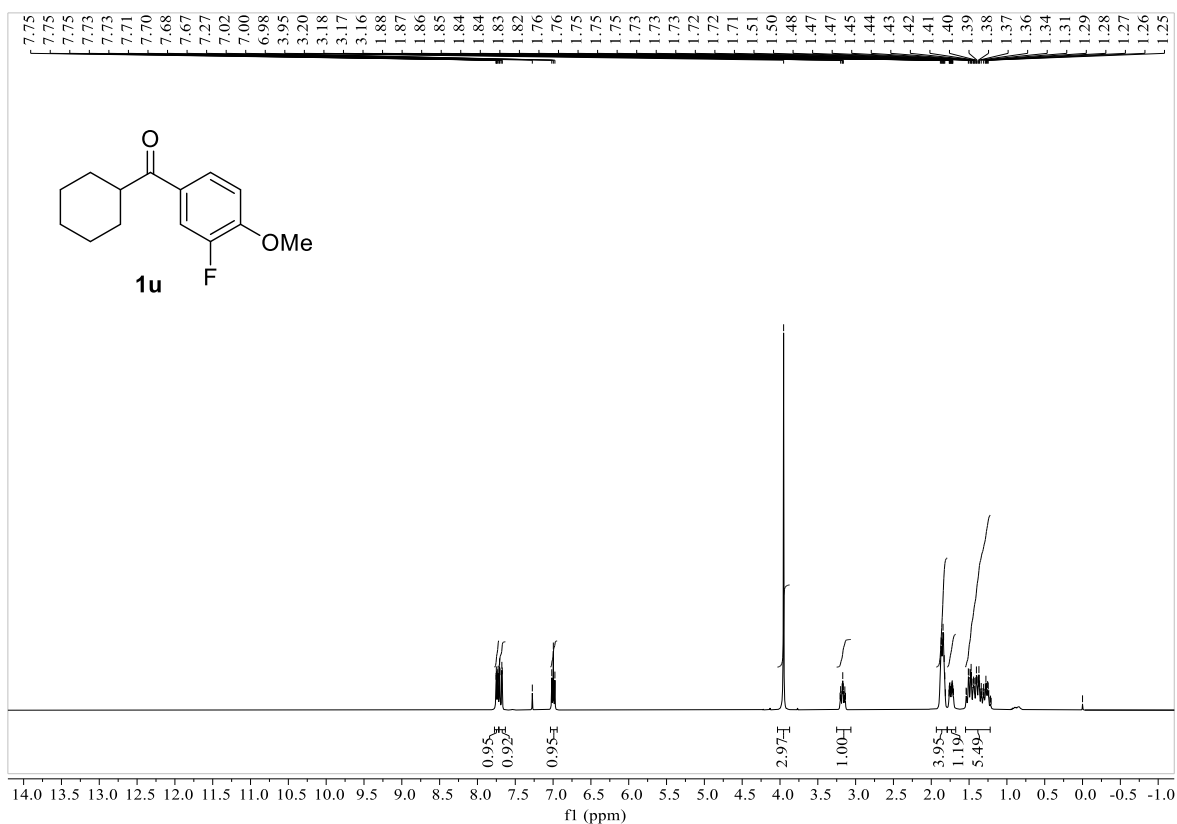
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1t**



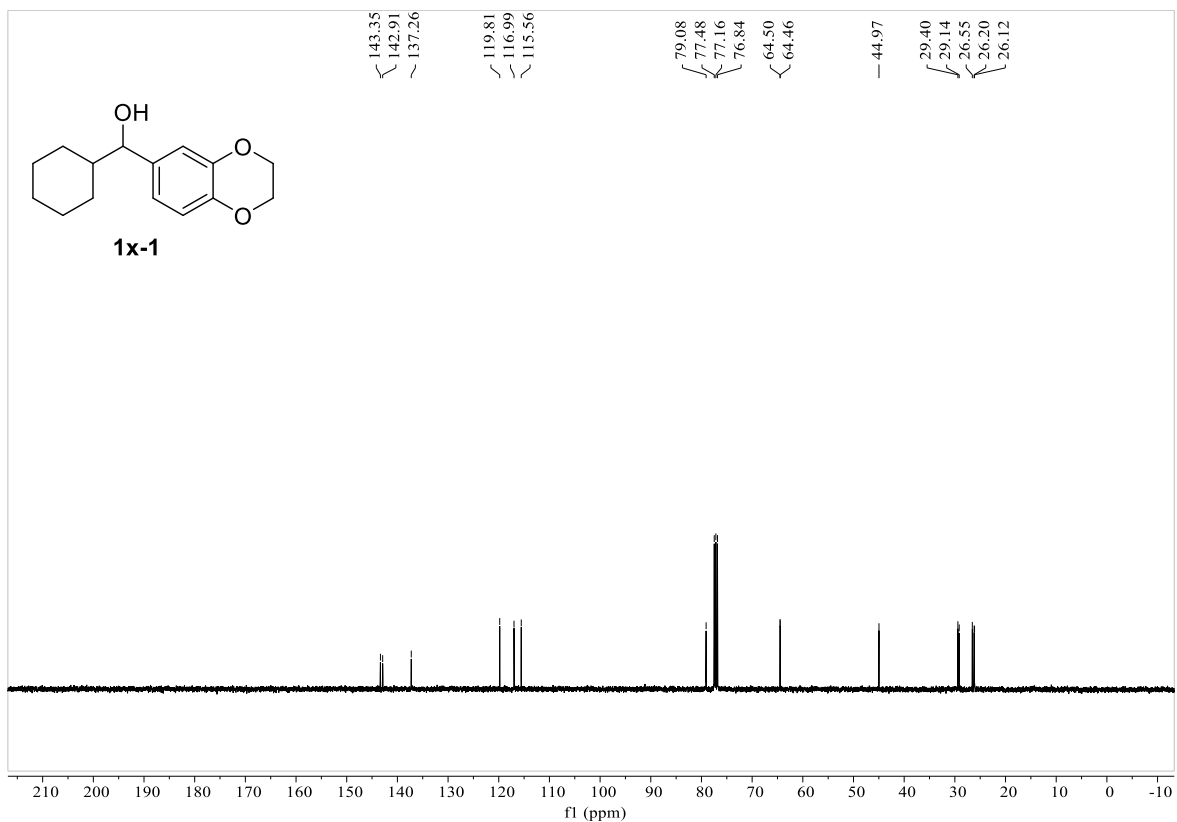
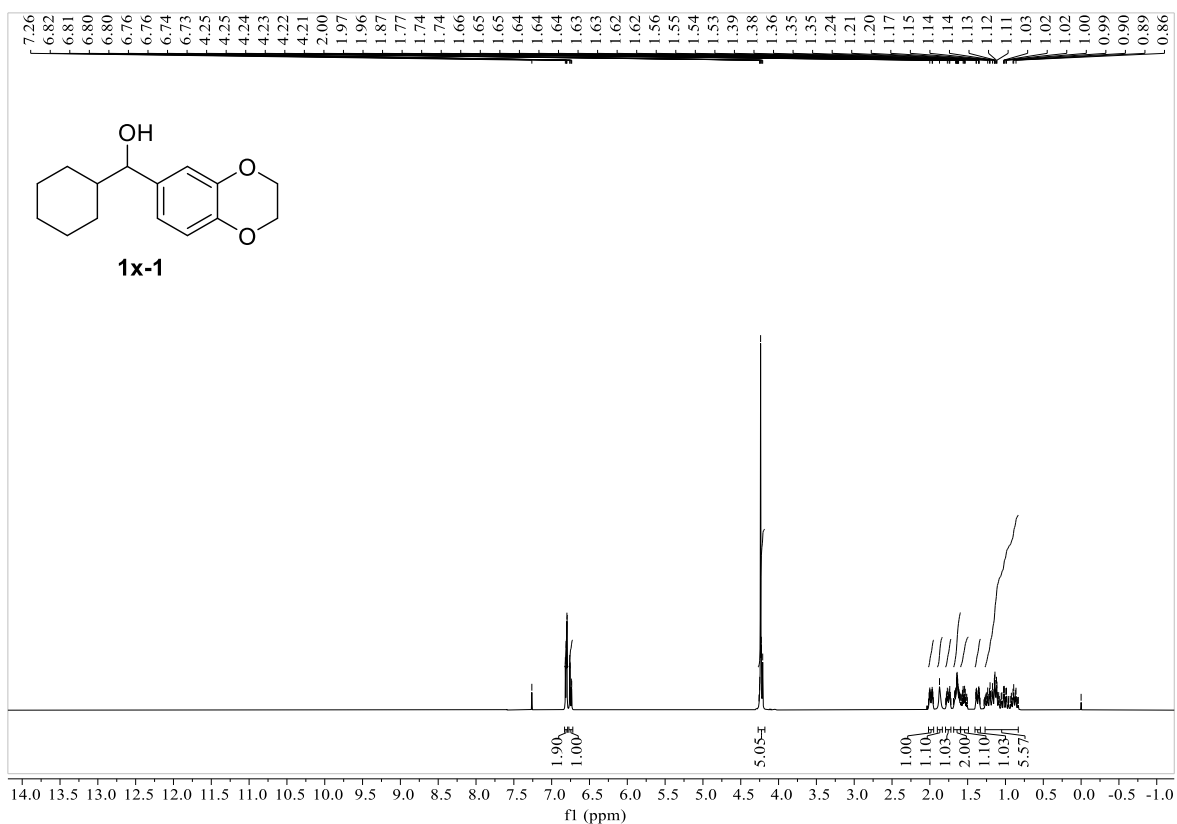
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1u-1**



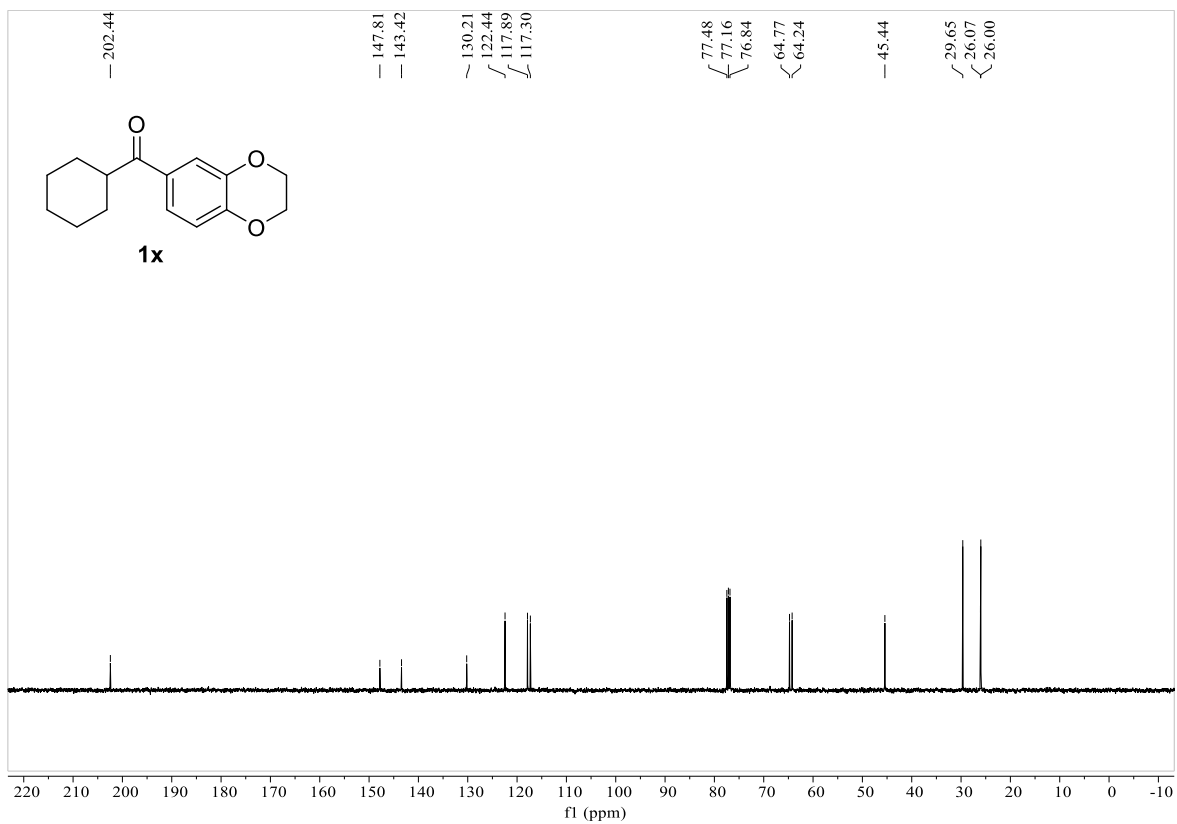
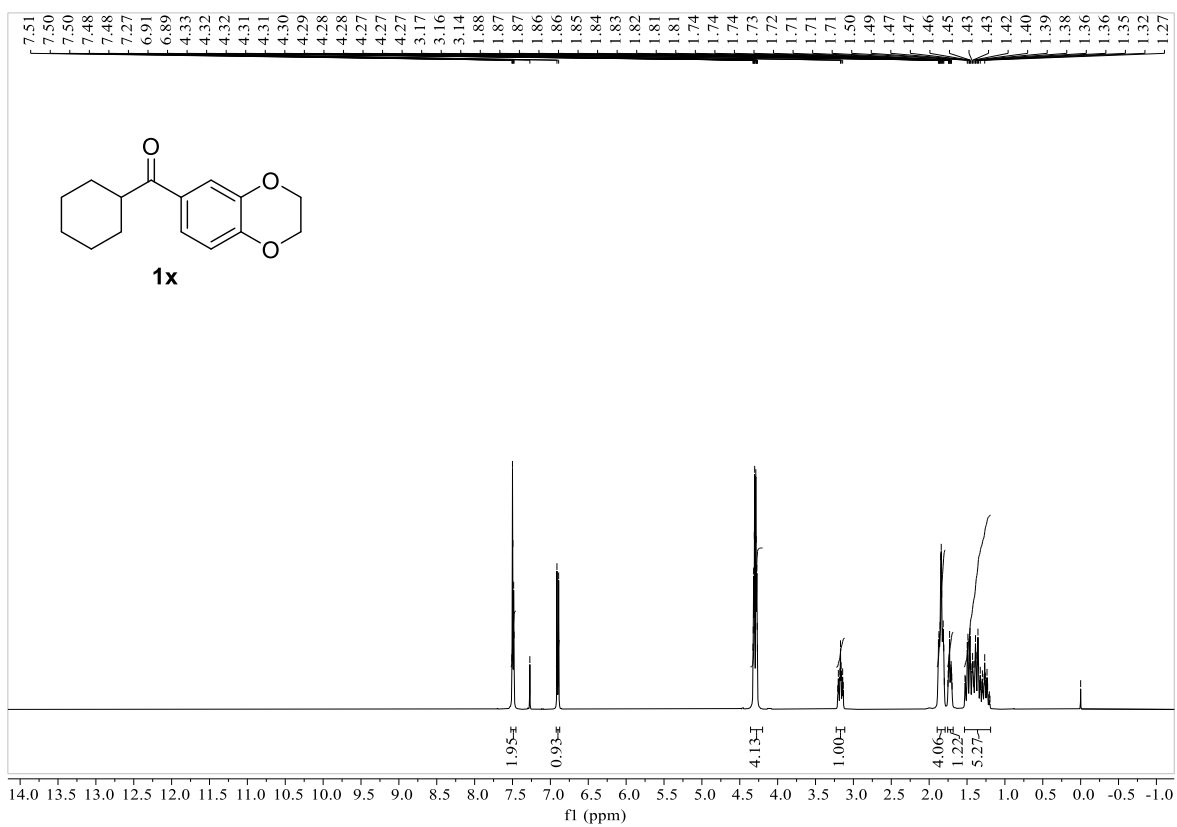
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1u**



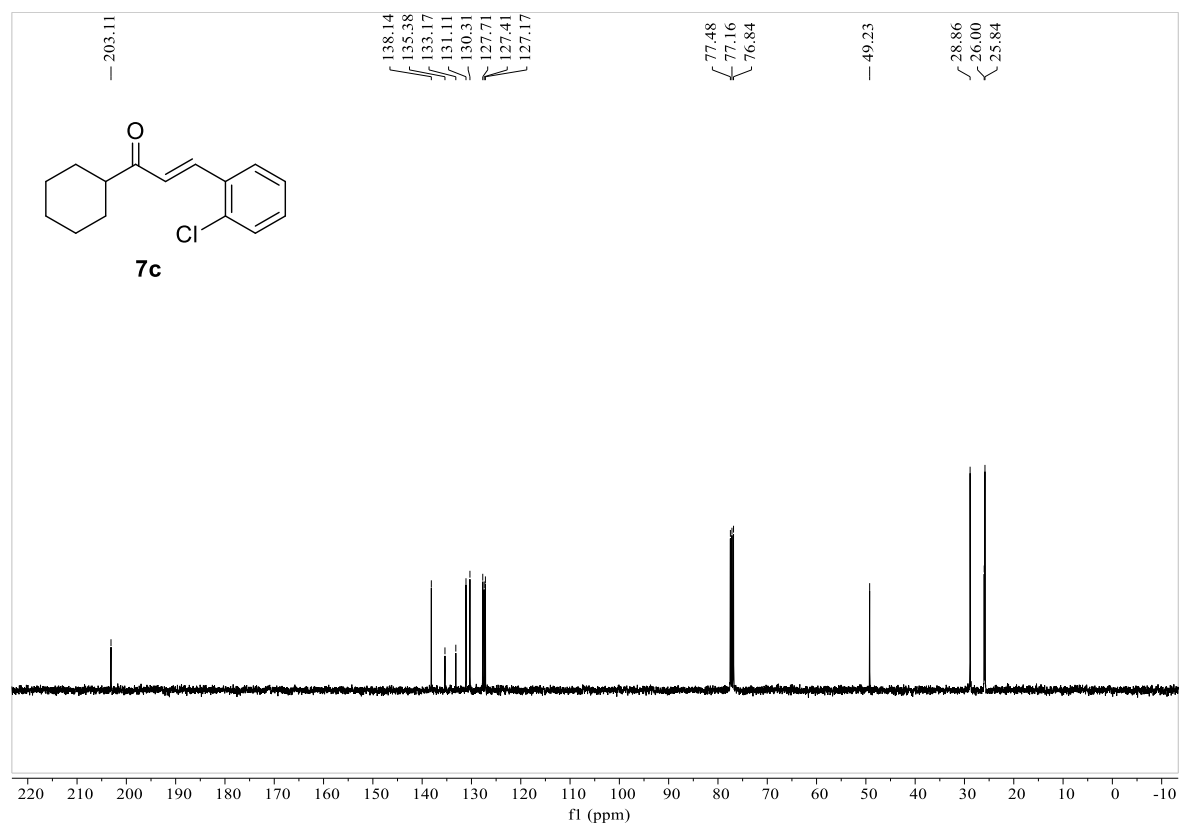
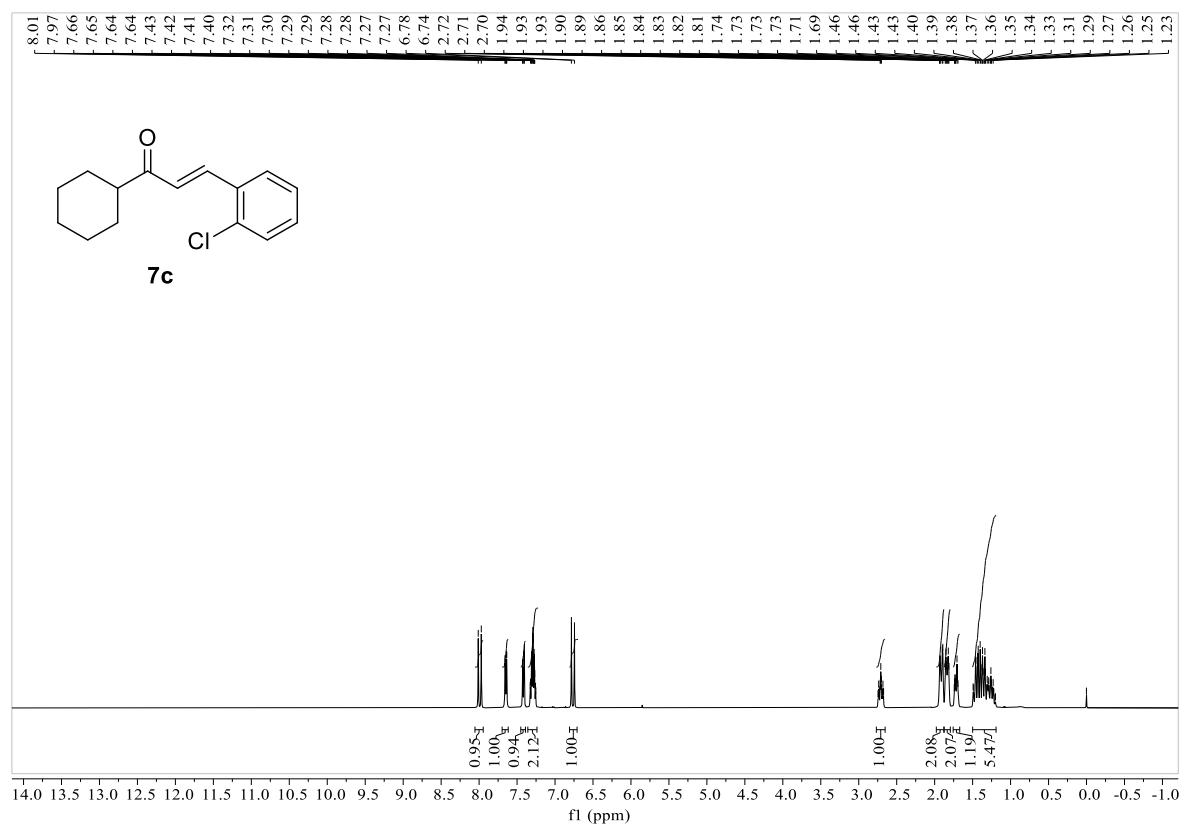
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1x-1**



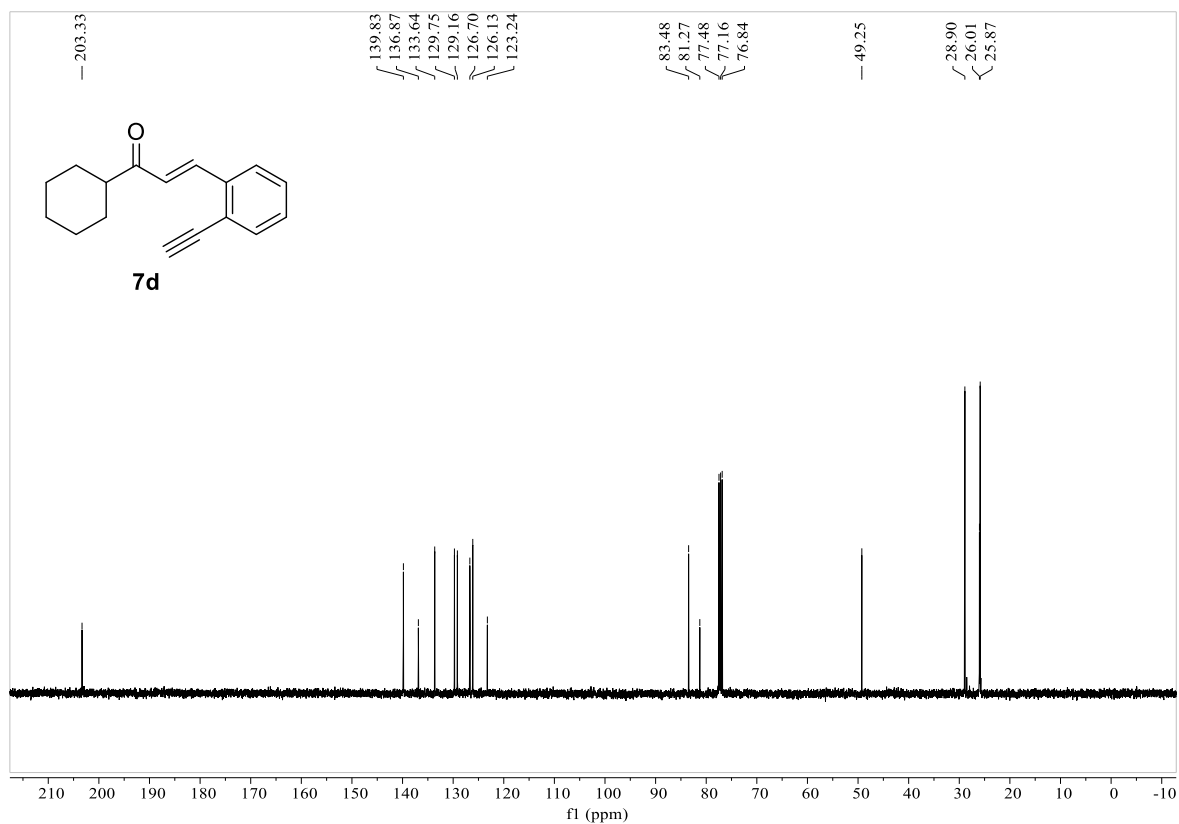
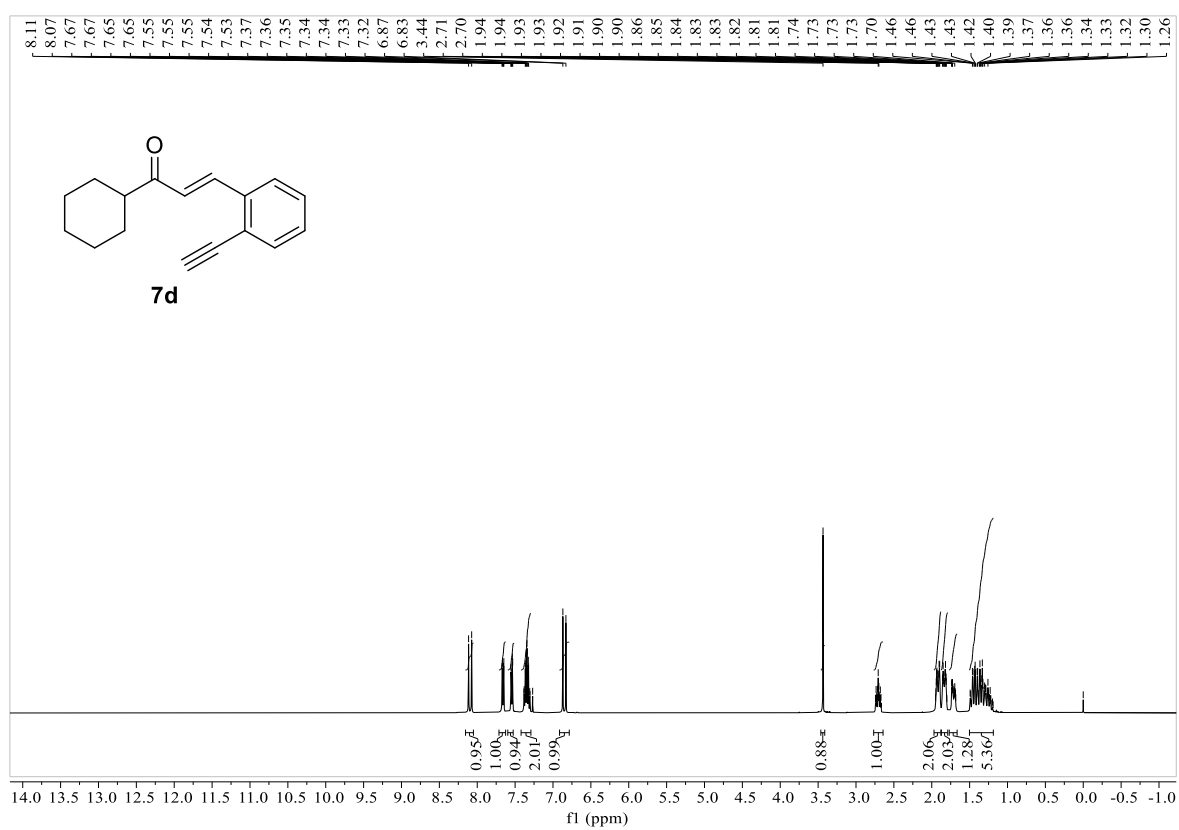
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **1x**



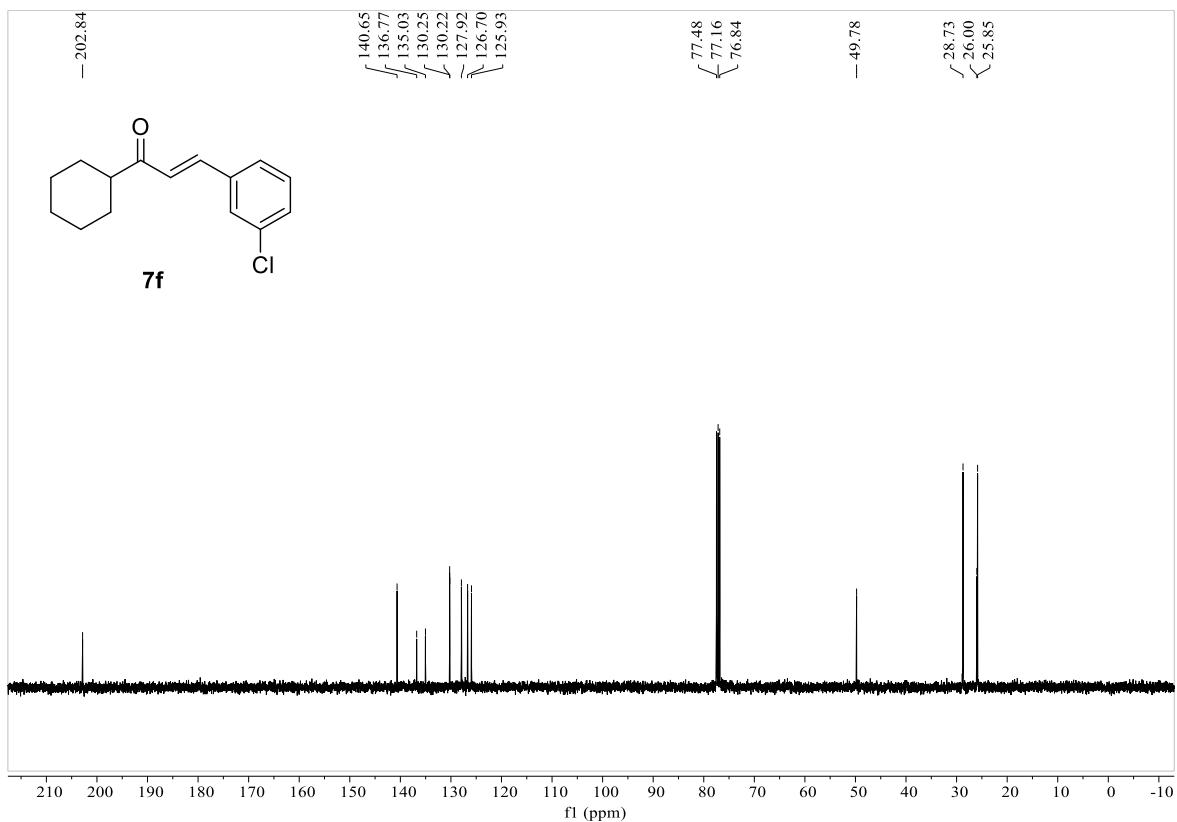
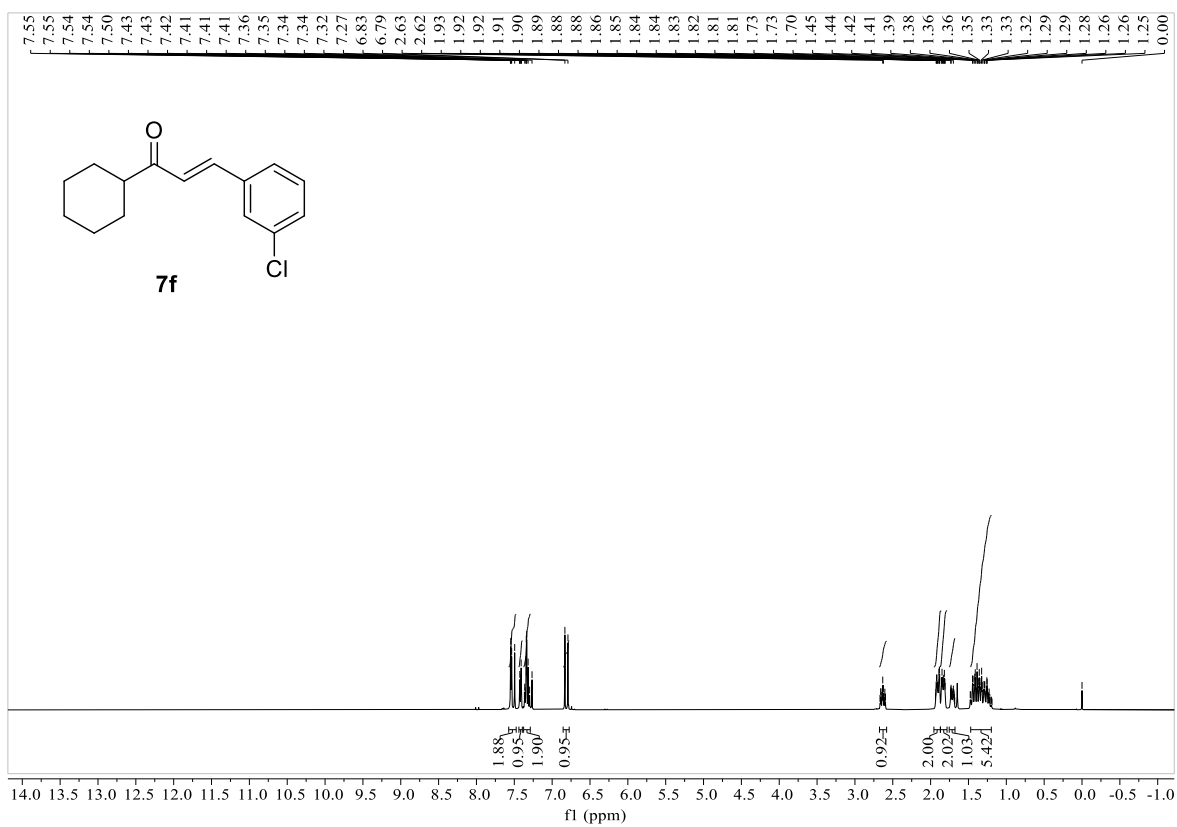
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **7c**



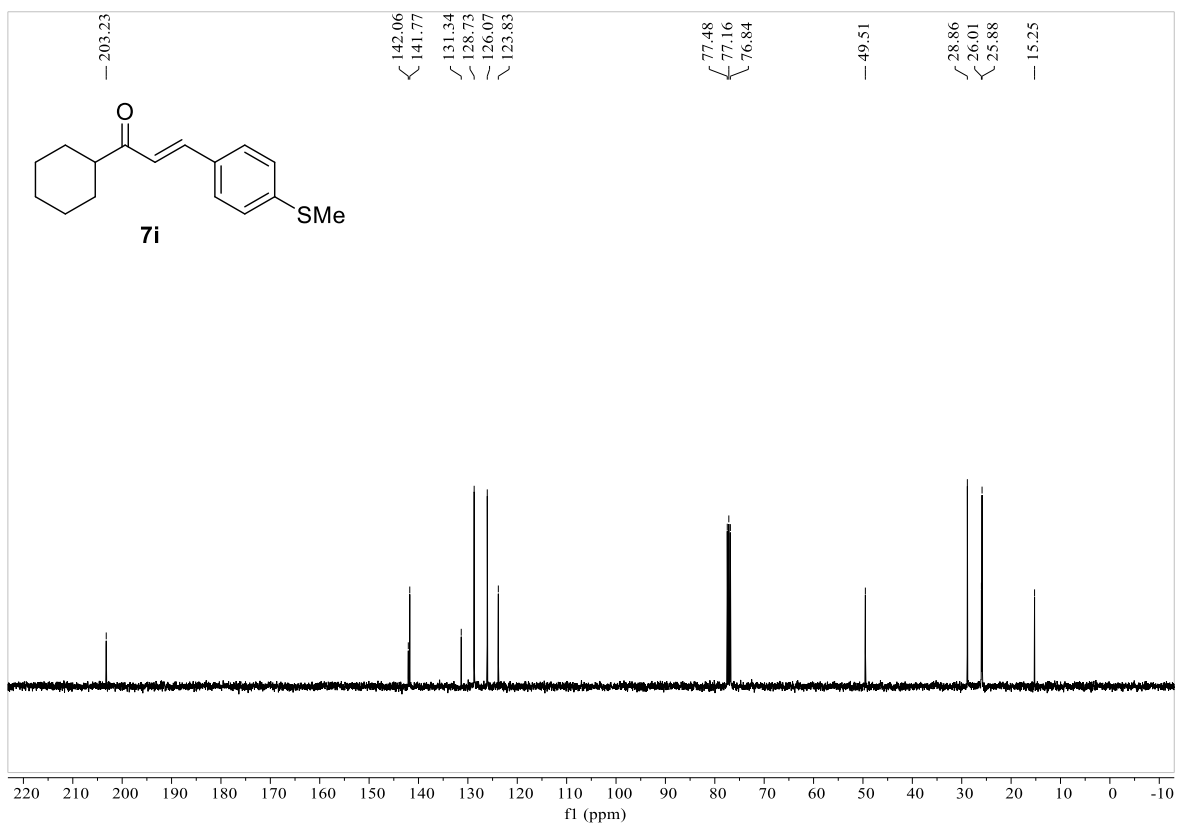
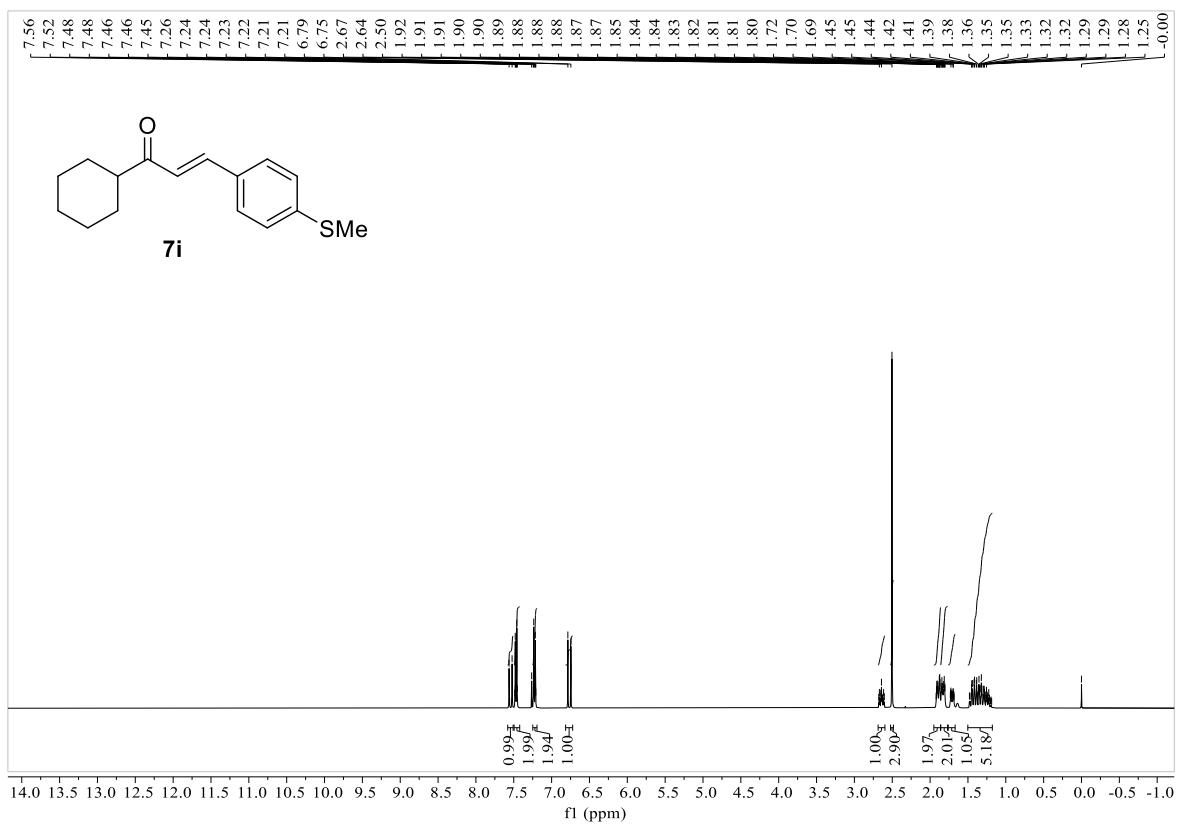
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **7d**



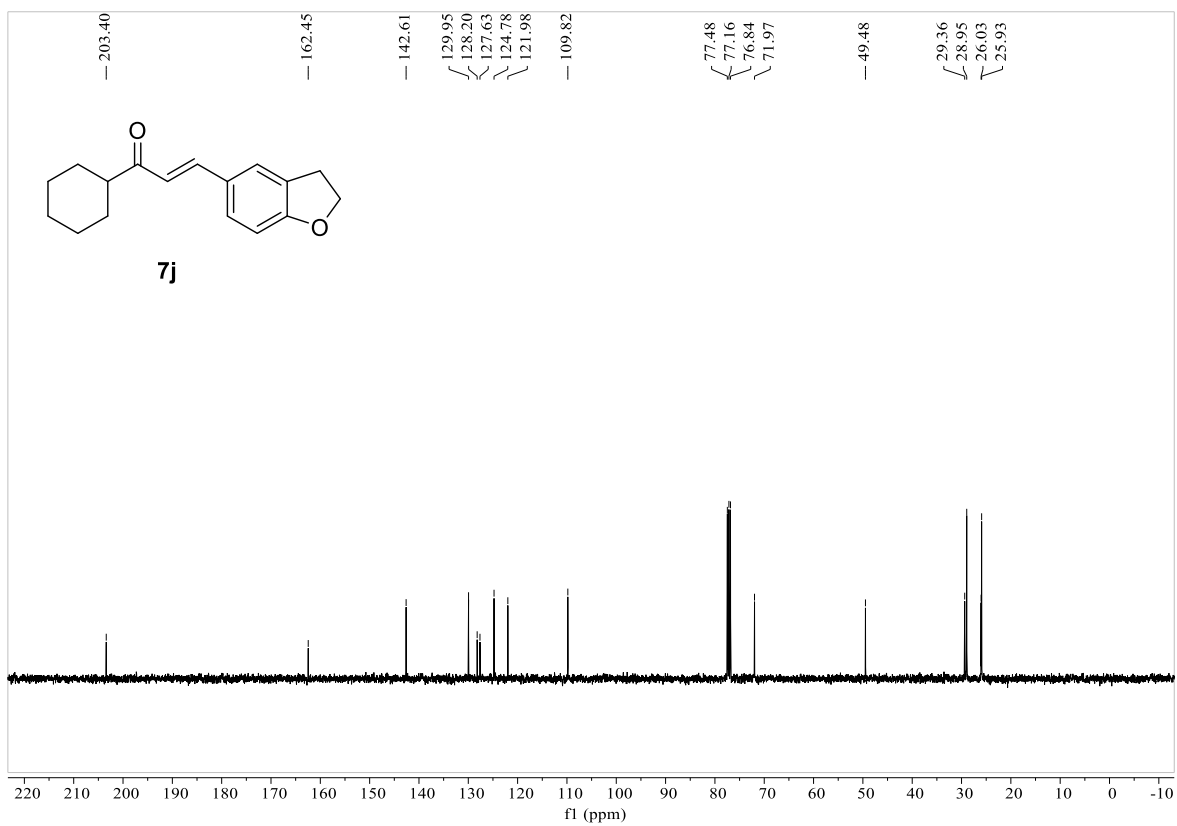
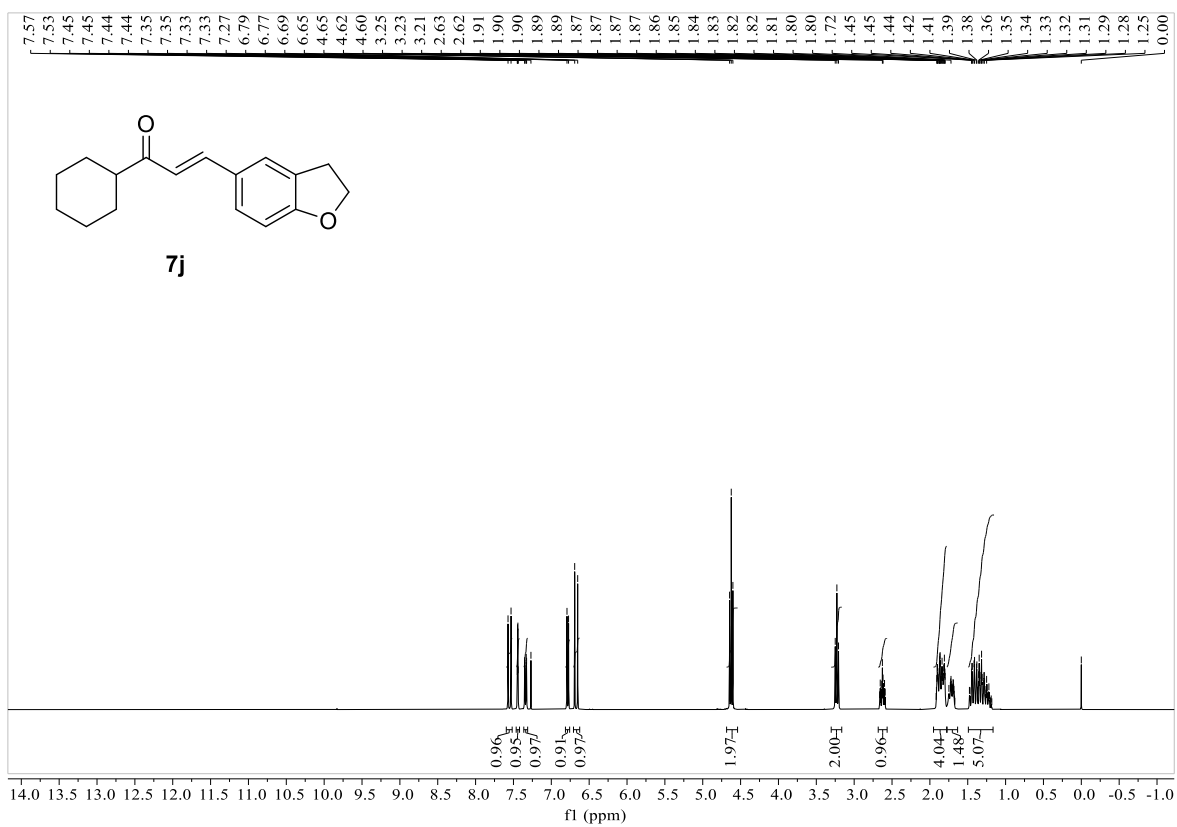
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **7f**



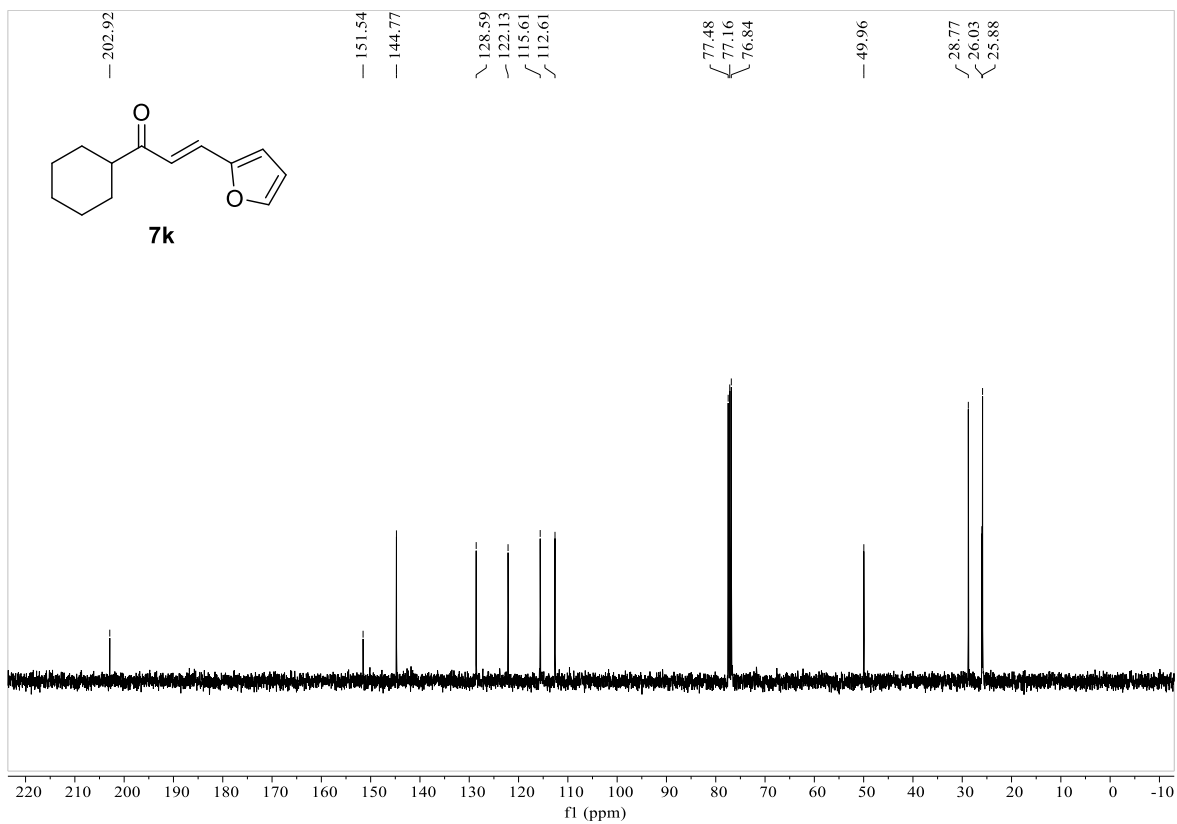
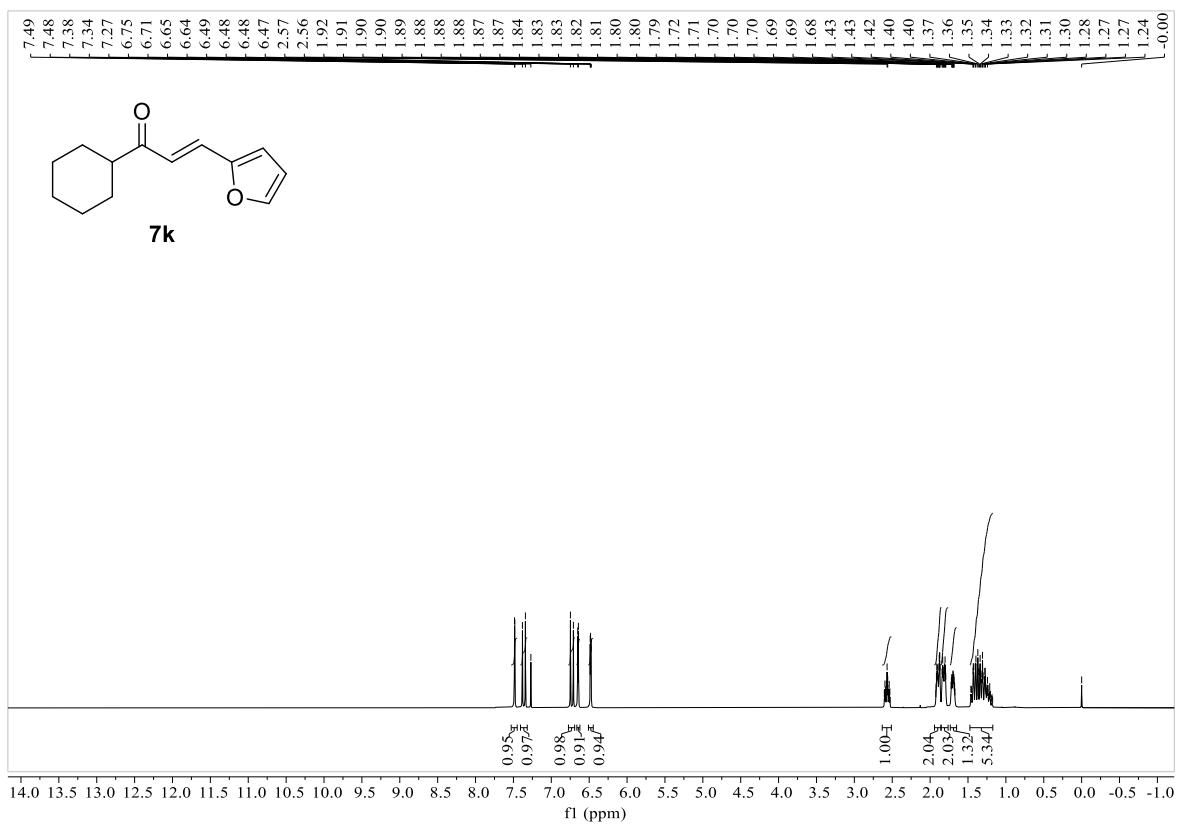
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **7i**



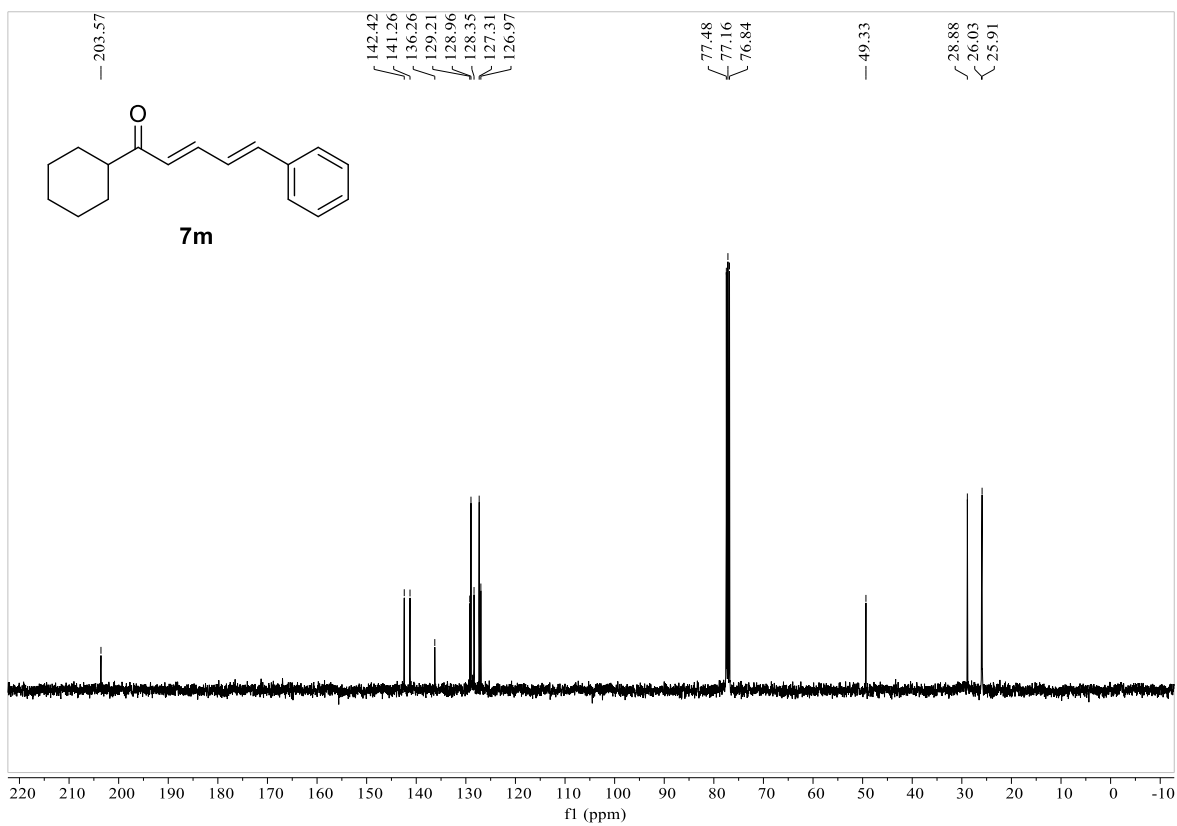
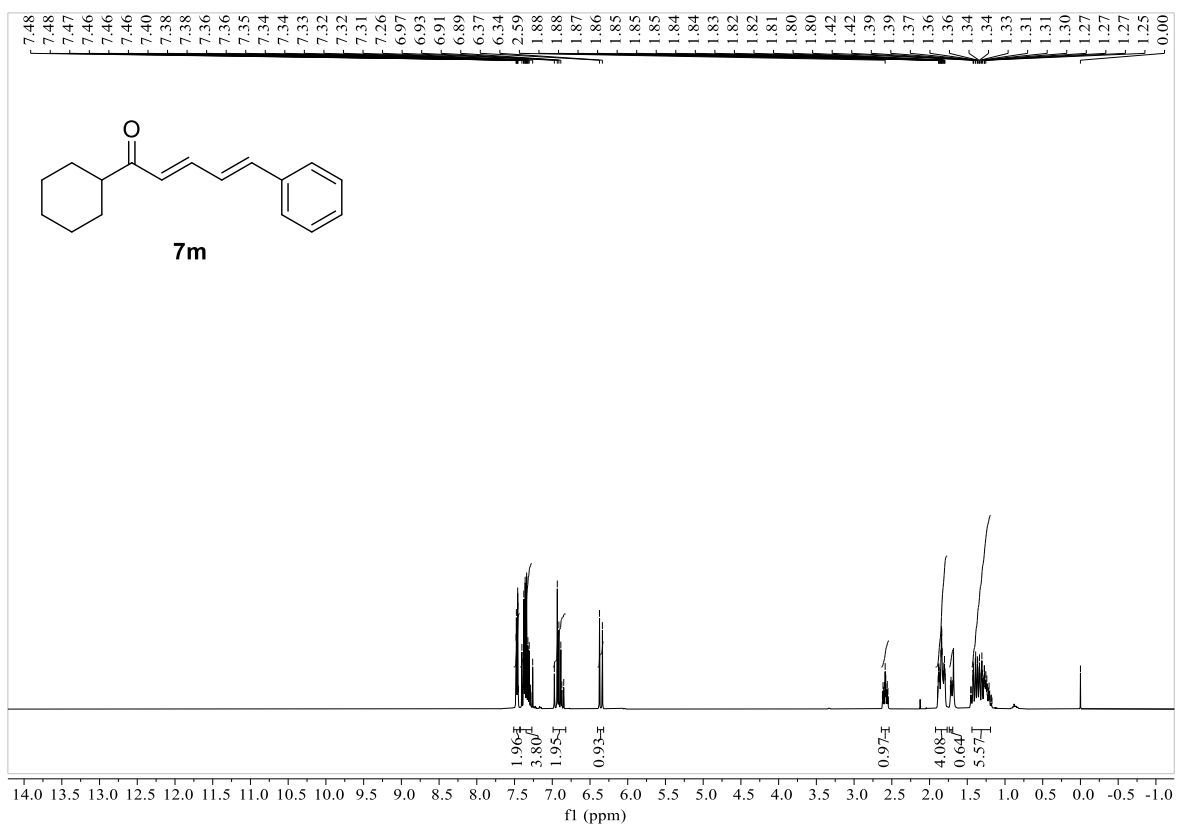
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **7j**



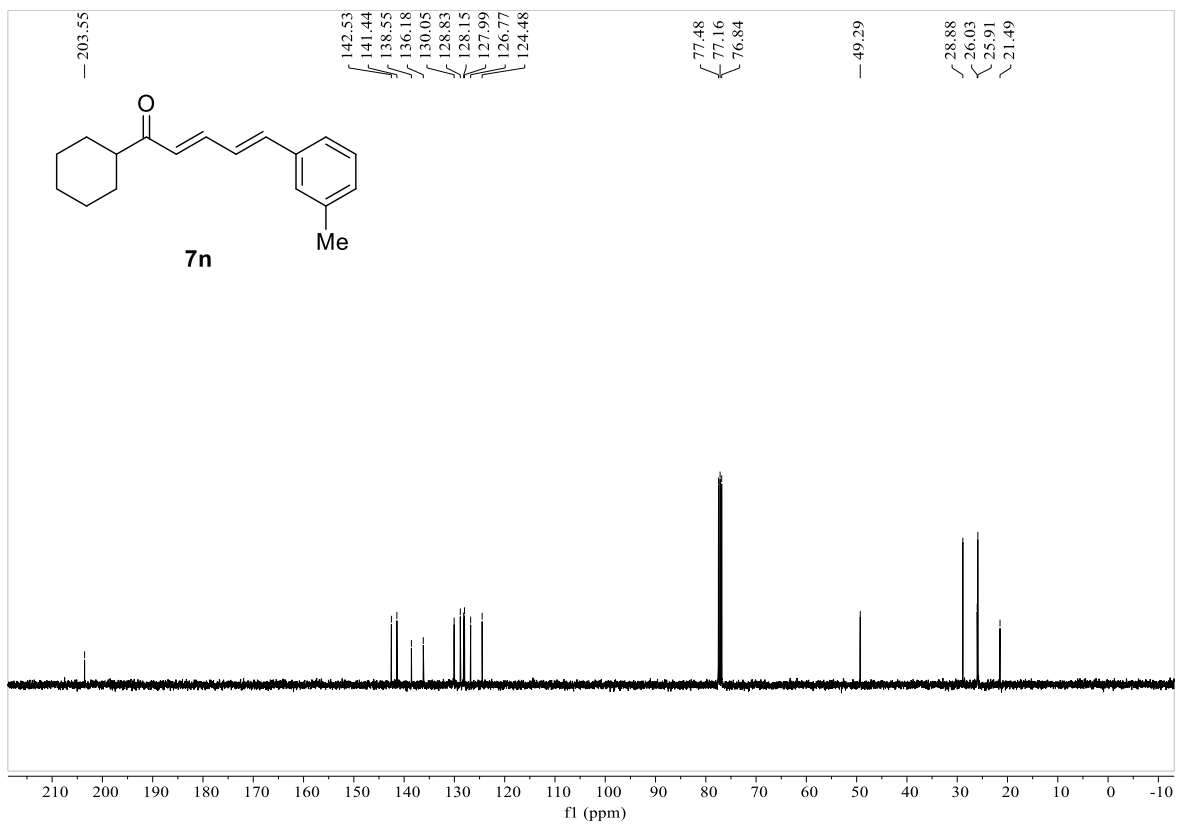
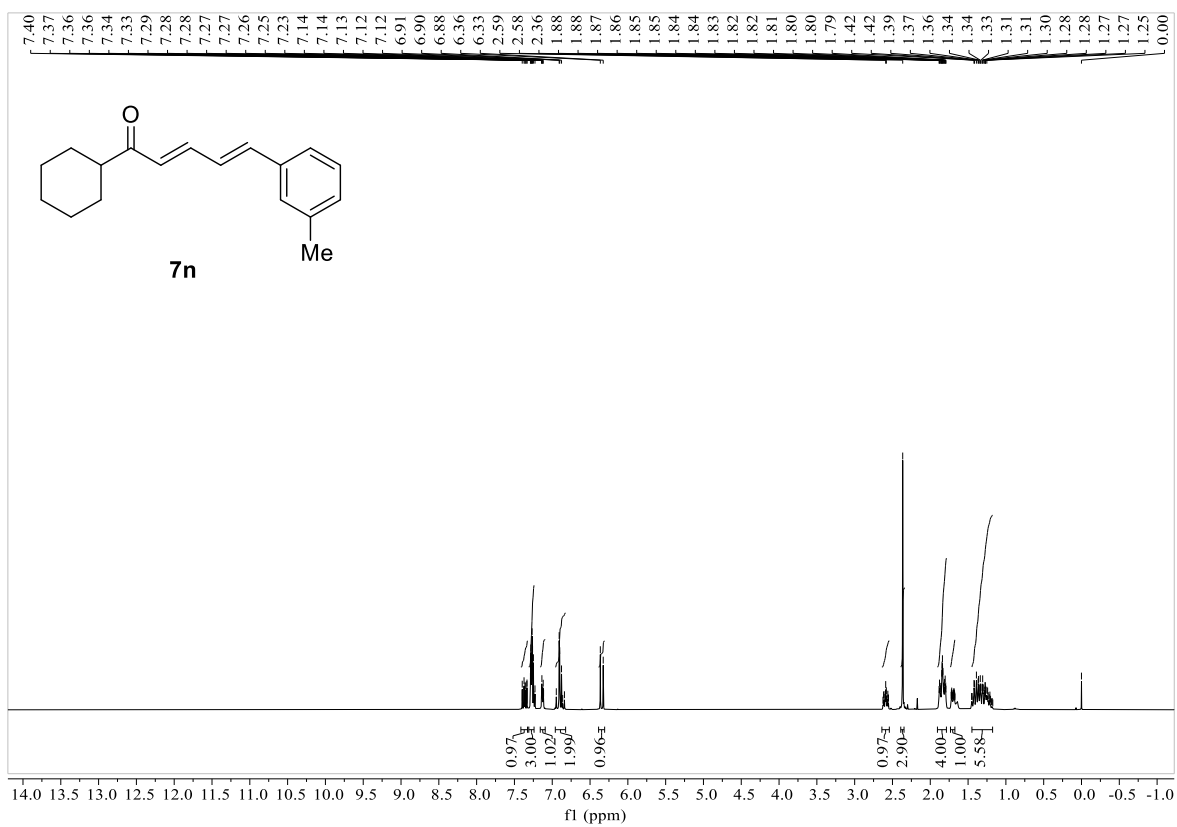
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **7k**



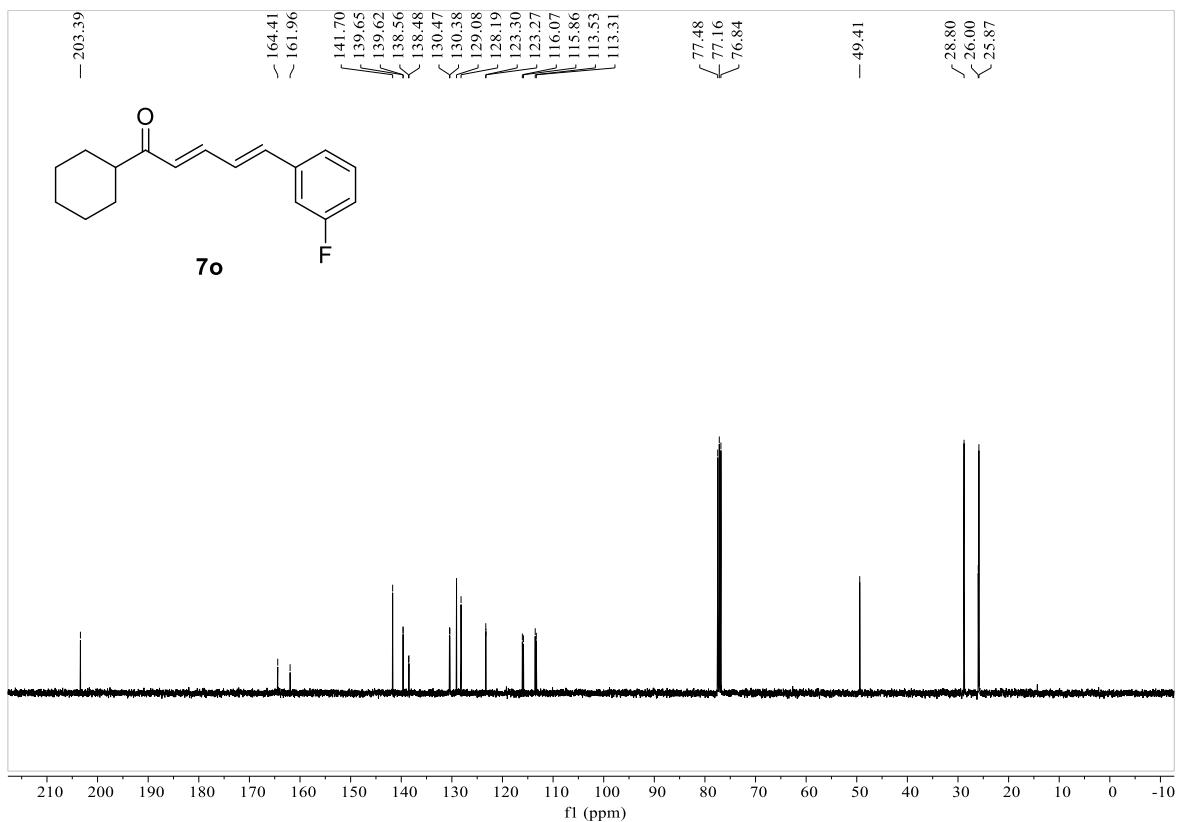
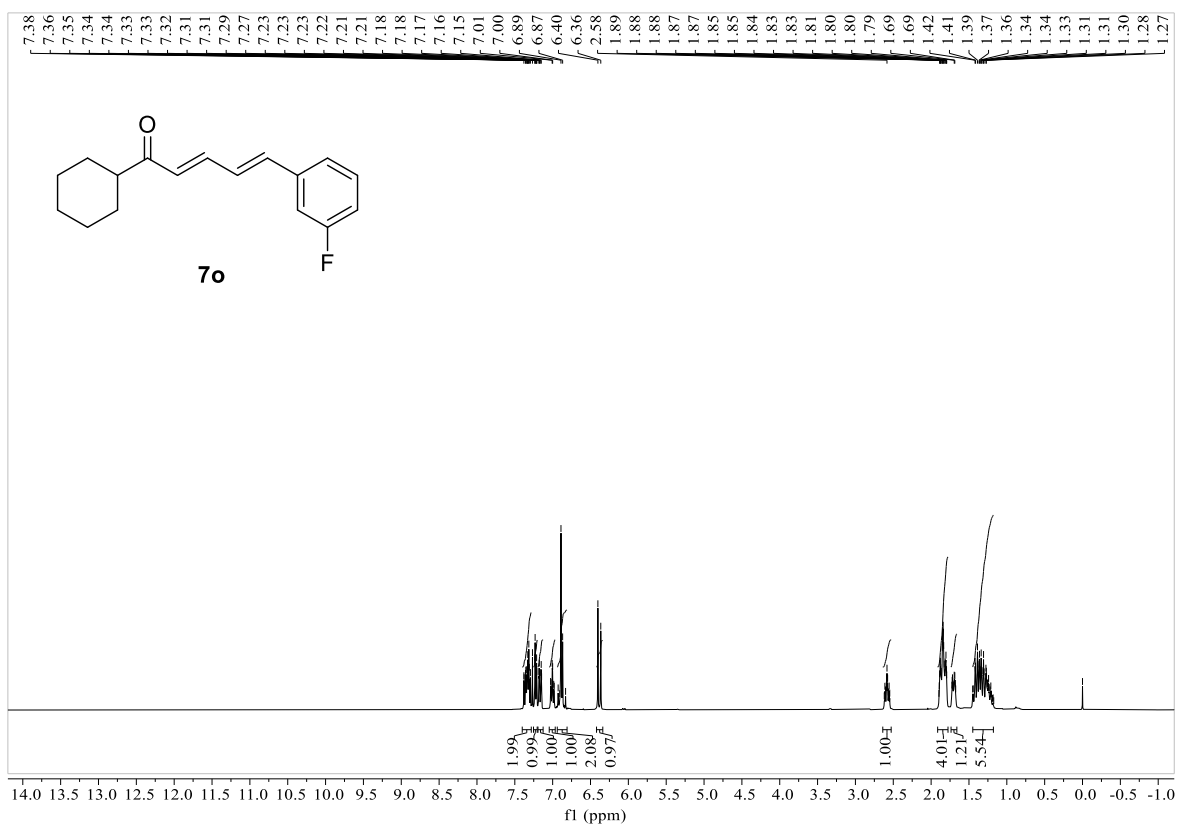
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **7m**



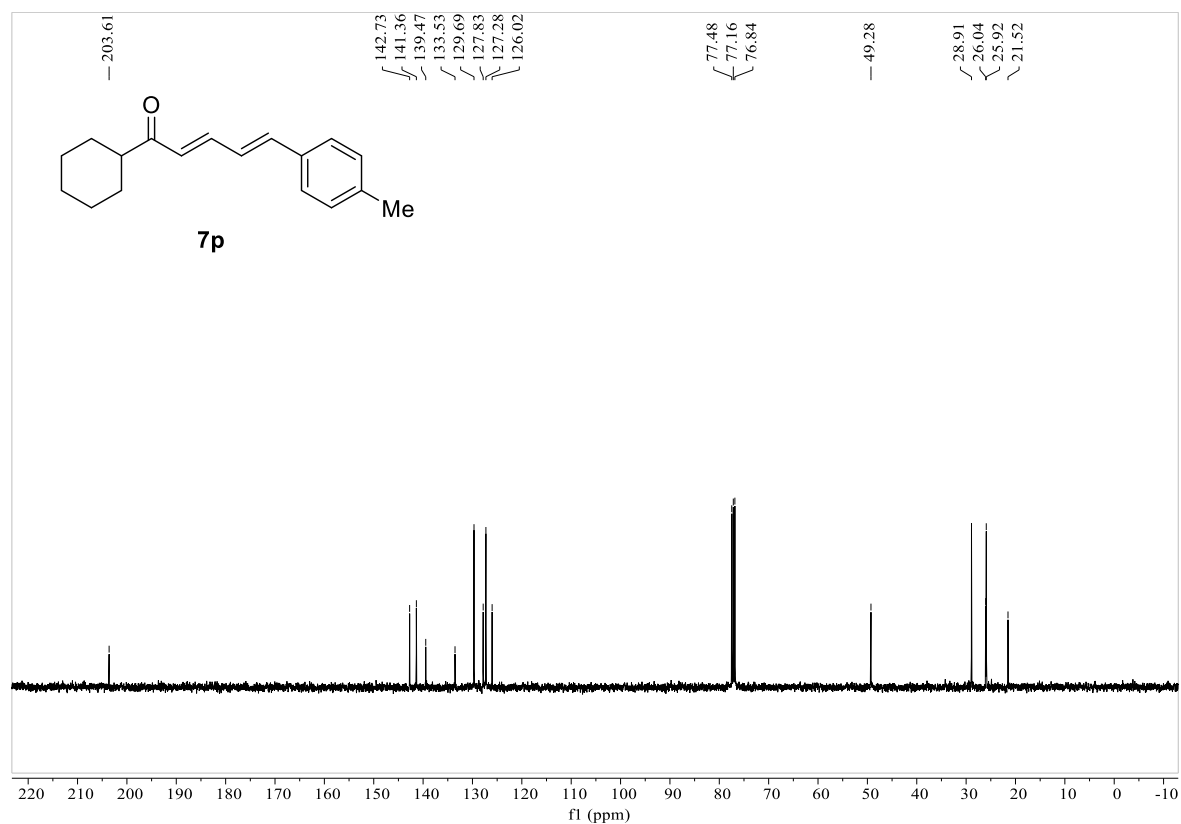
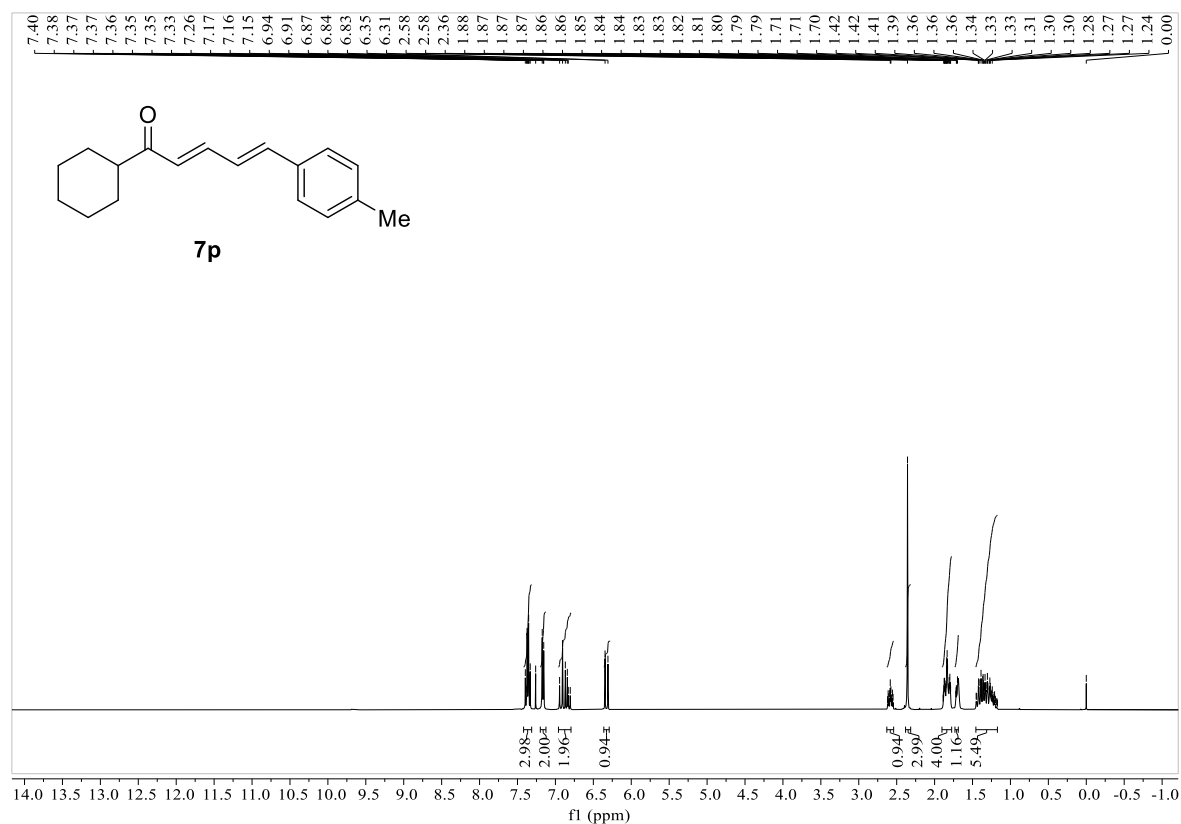
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **7n**



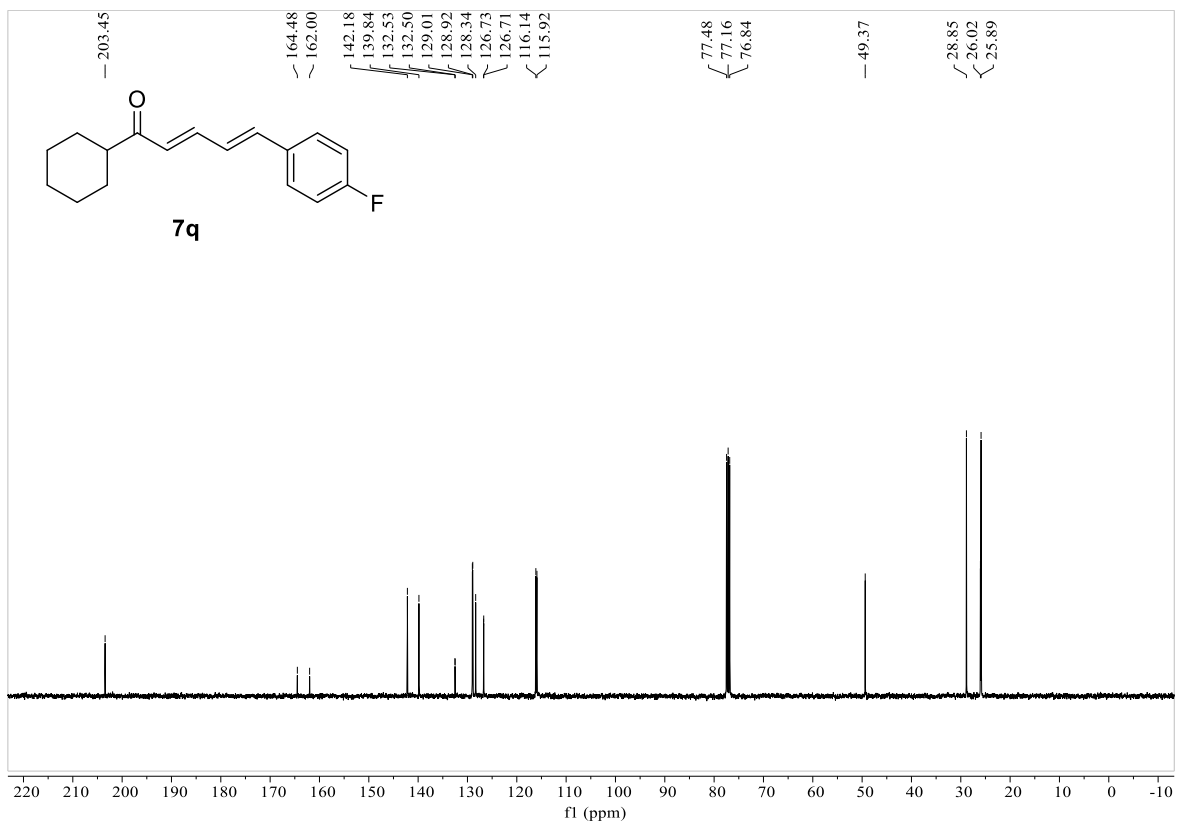
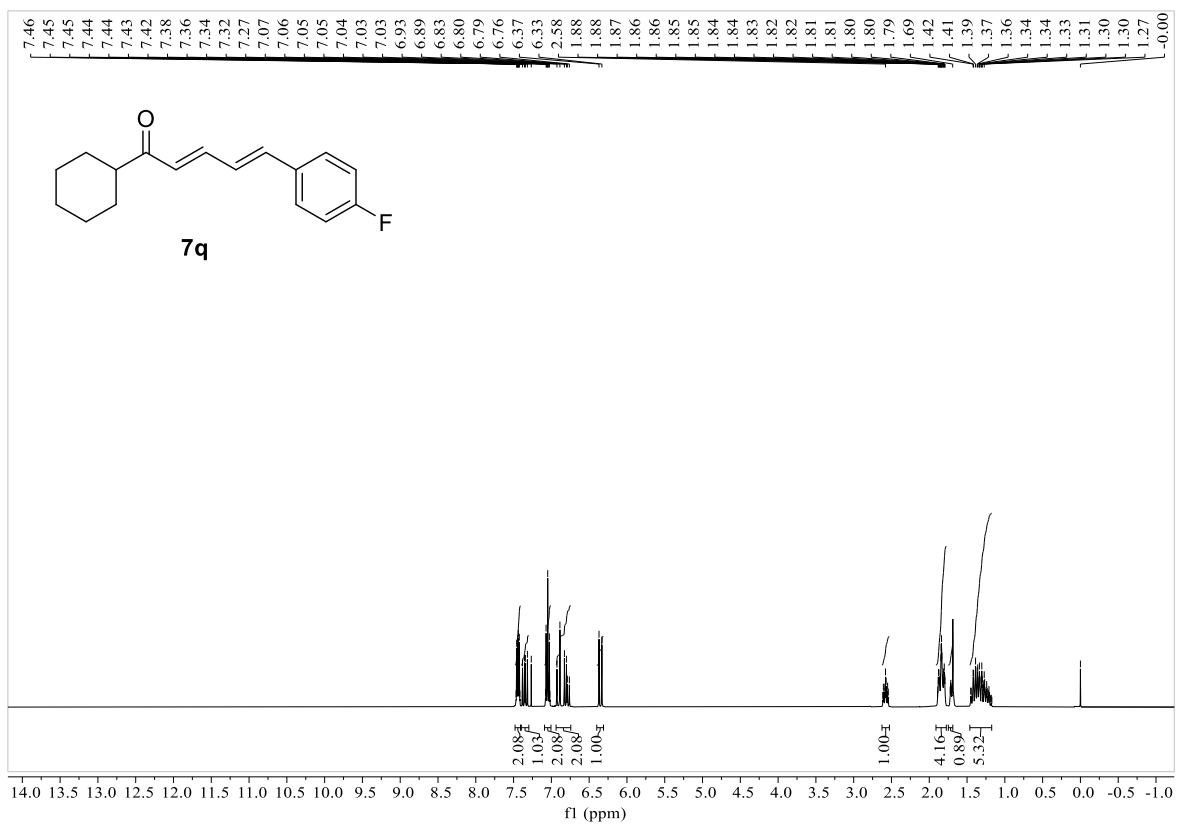
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **7o**



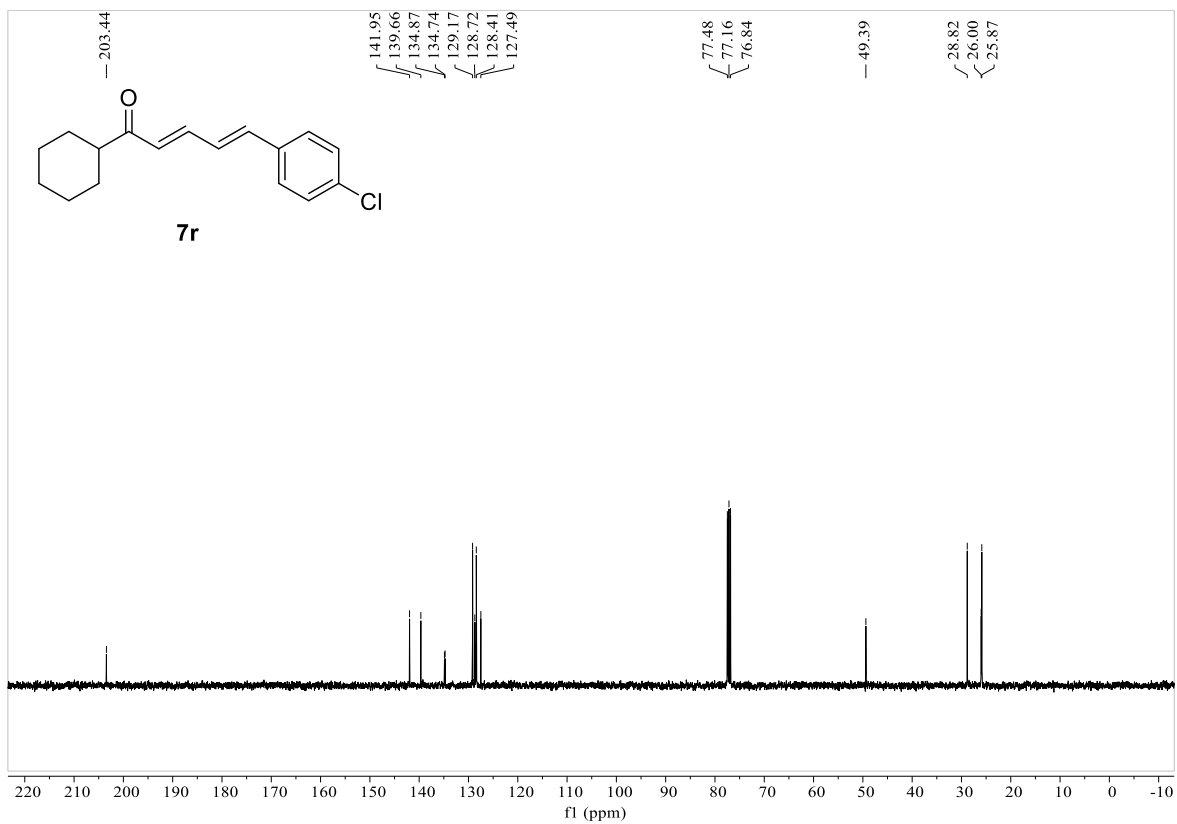
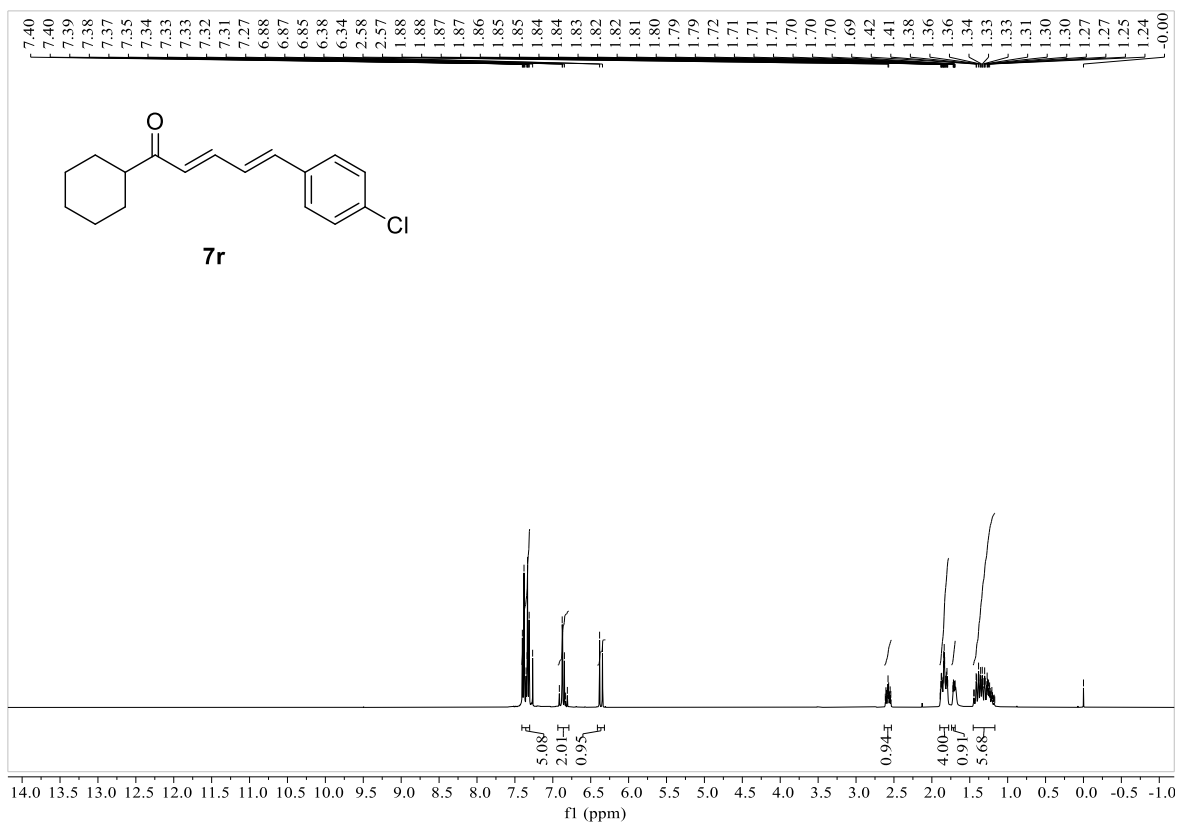
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **7p**



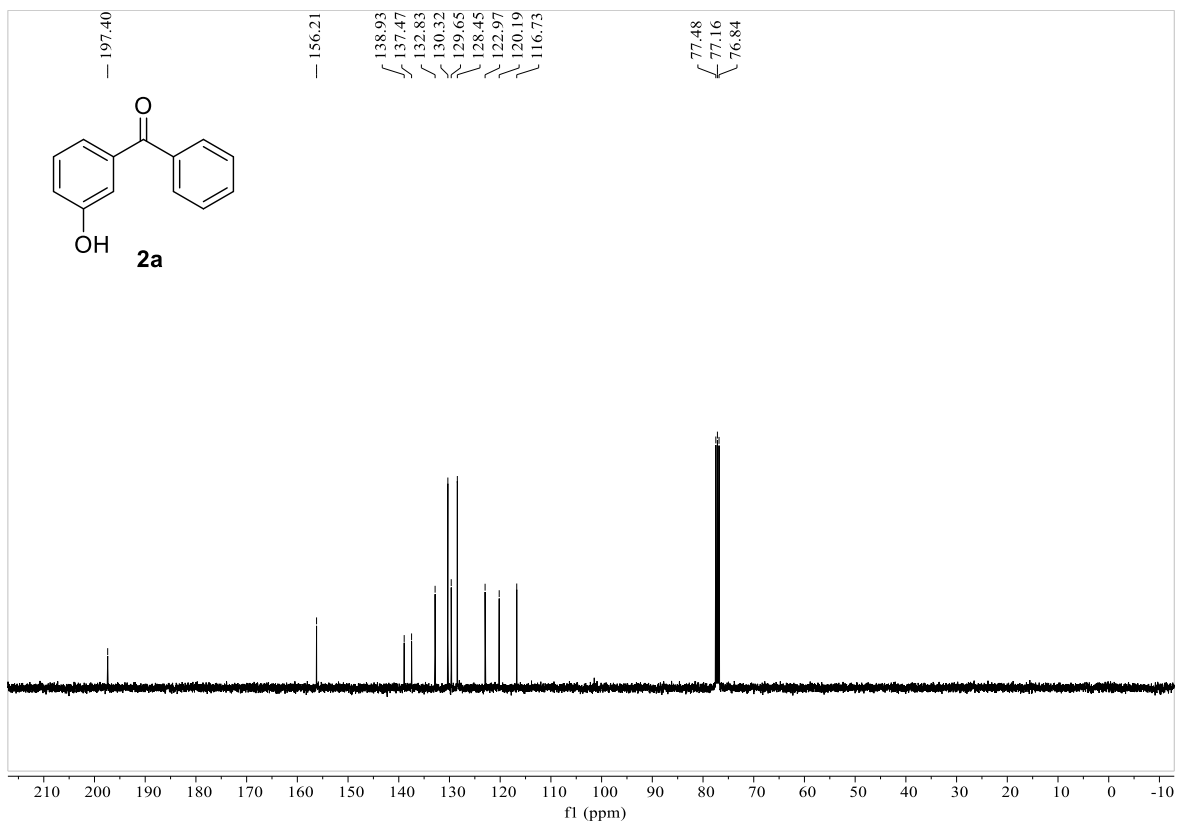
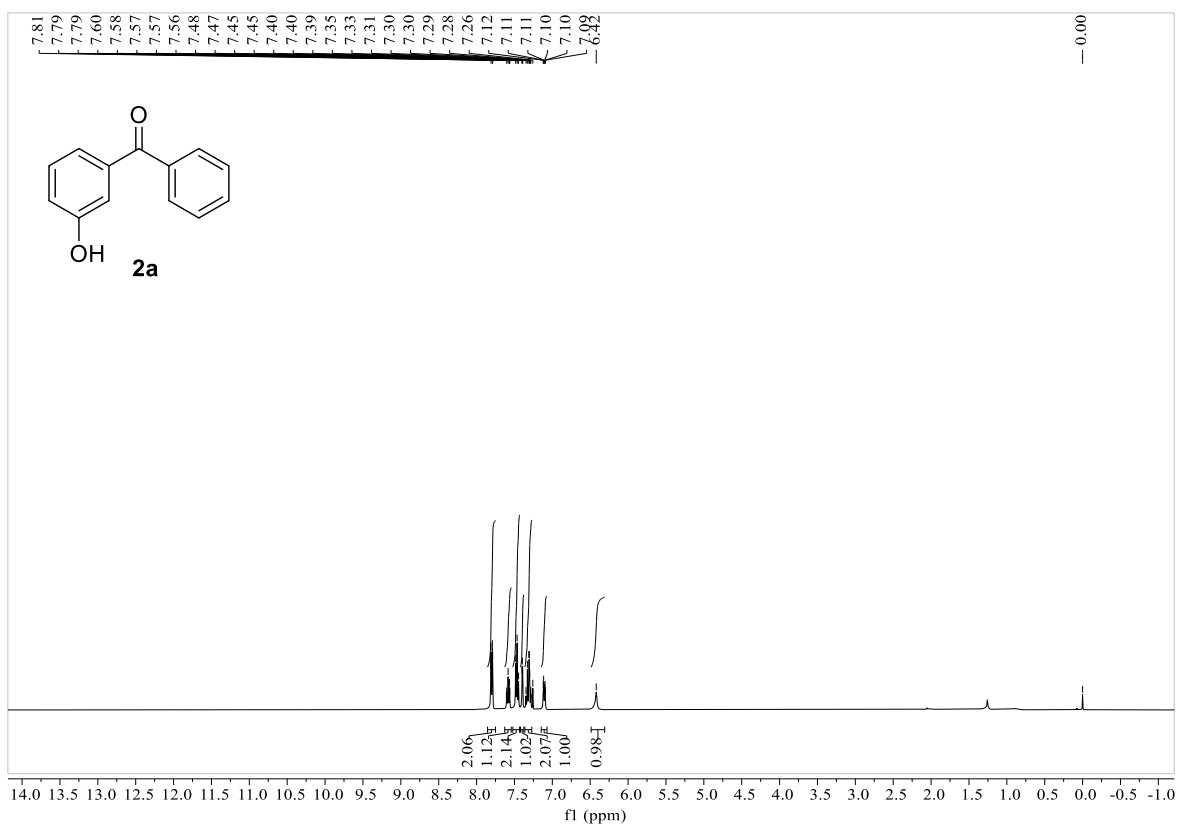
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **7q**



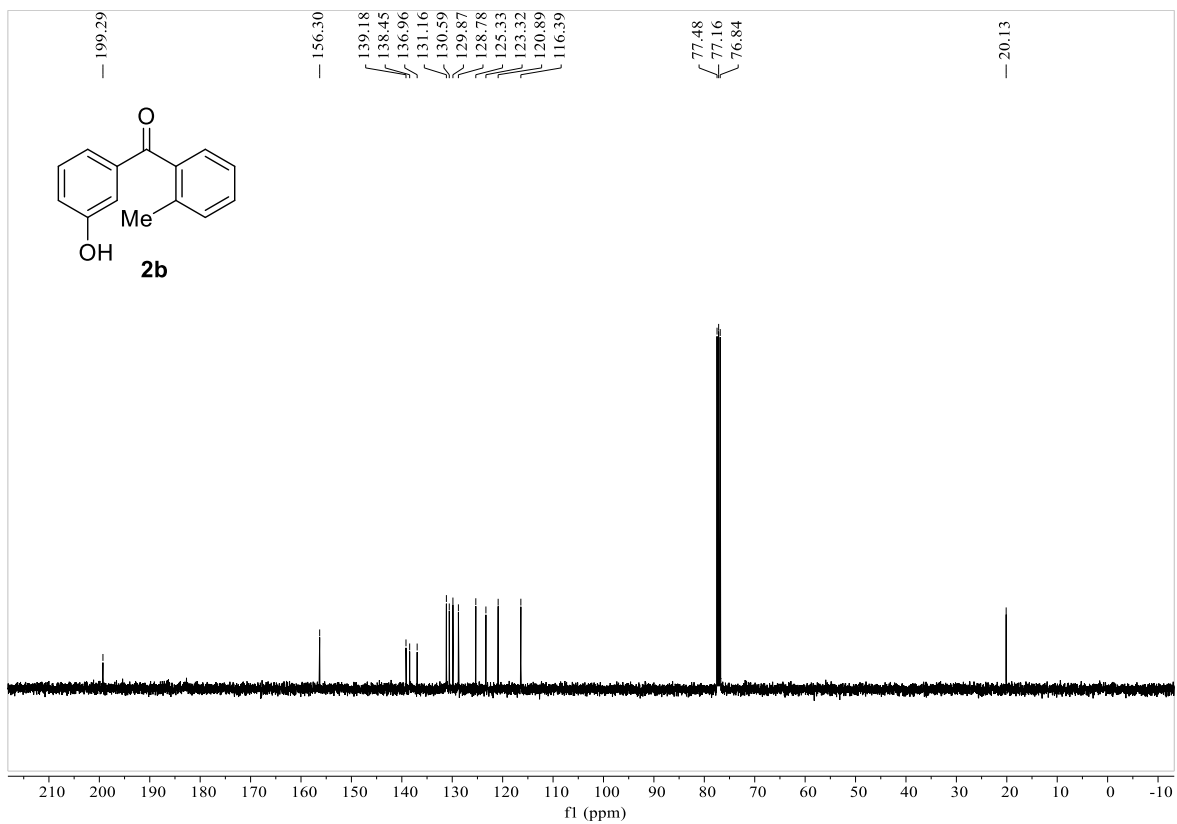
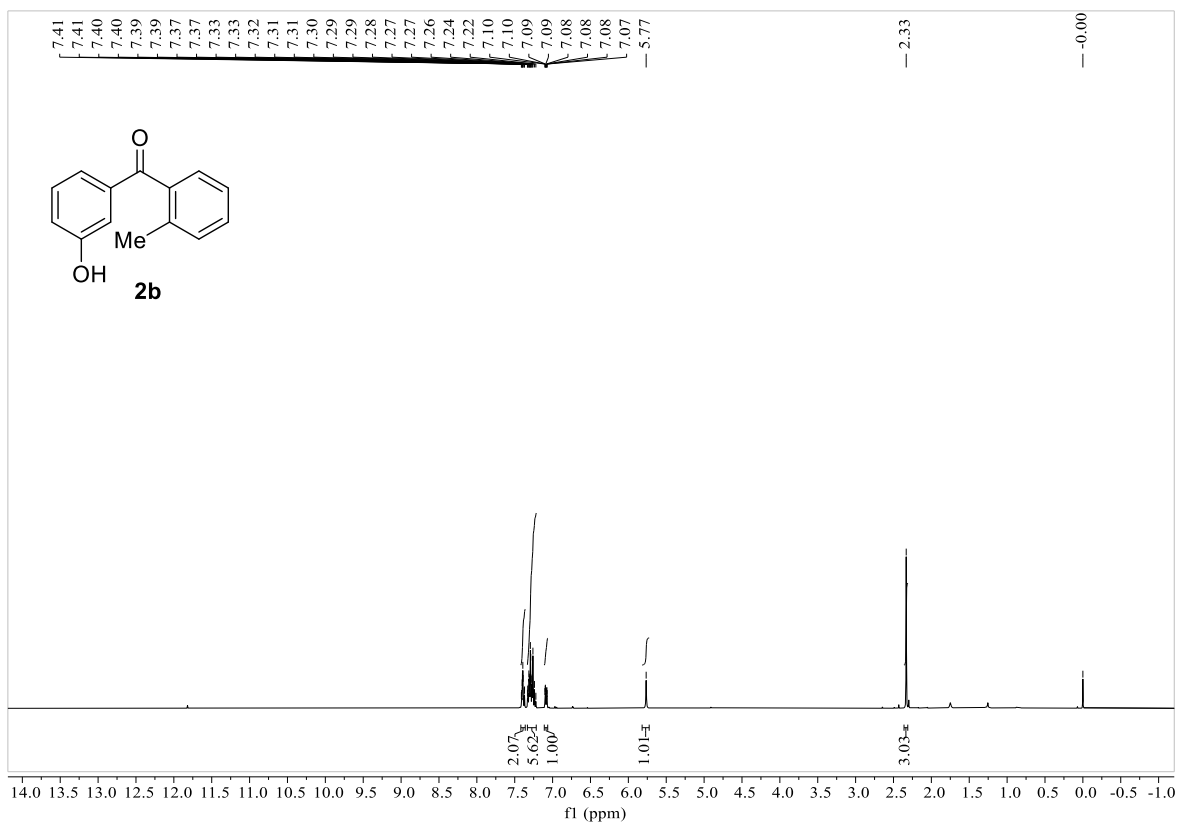
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **7r**



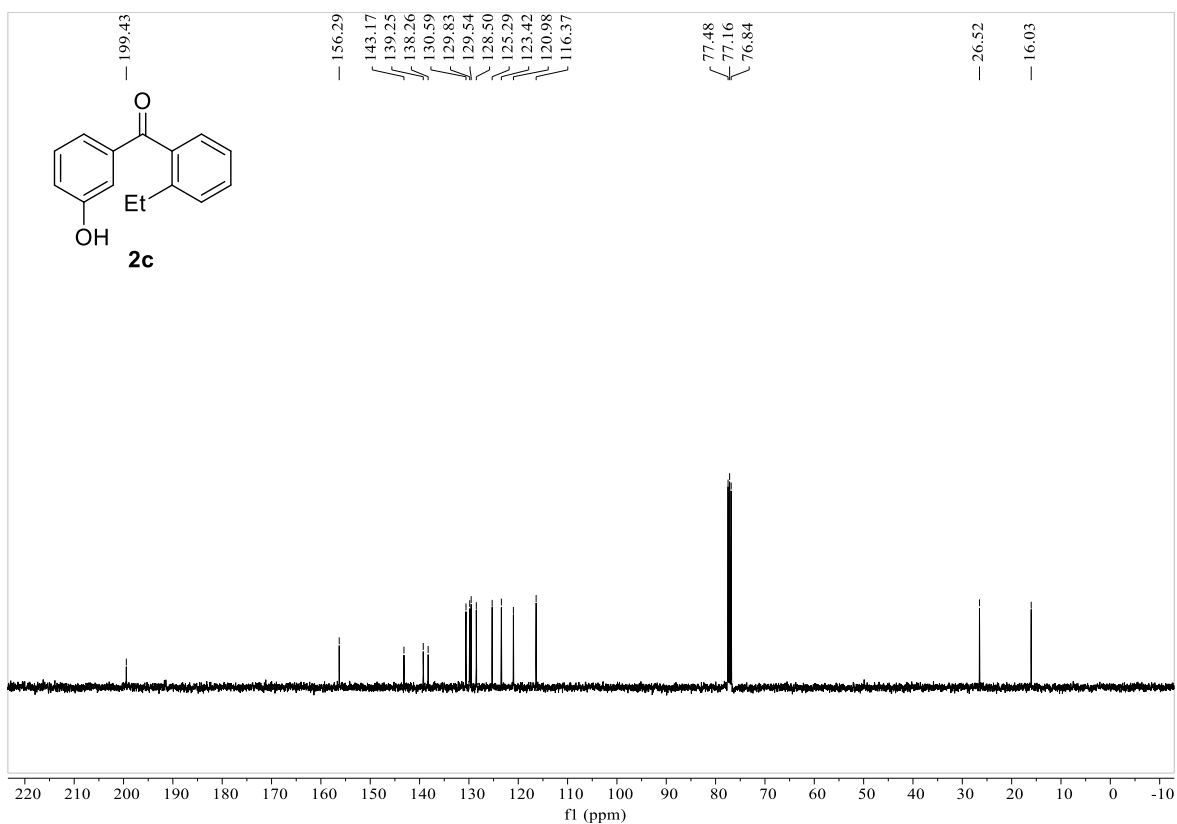
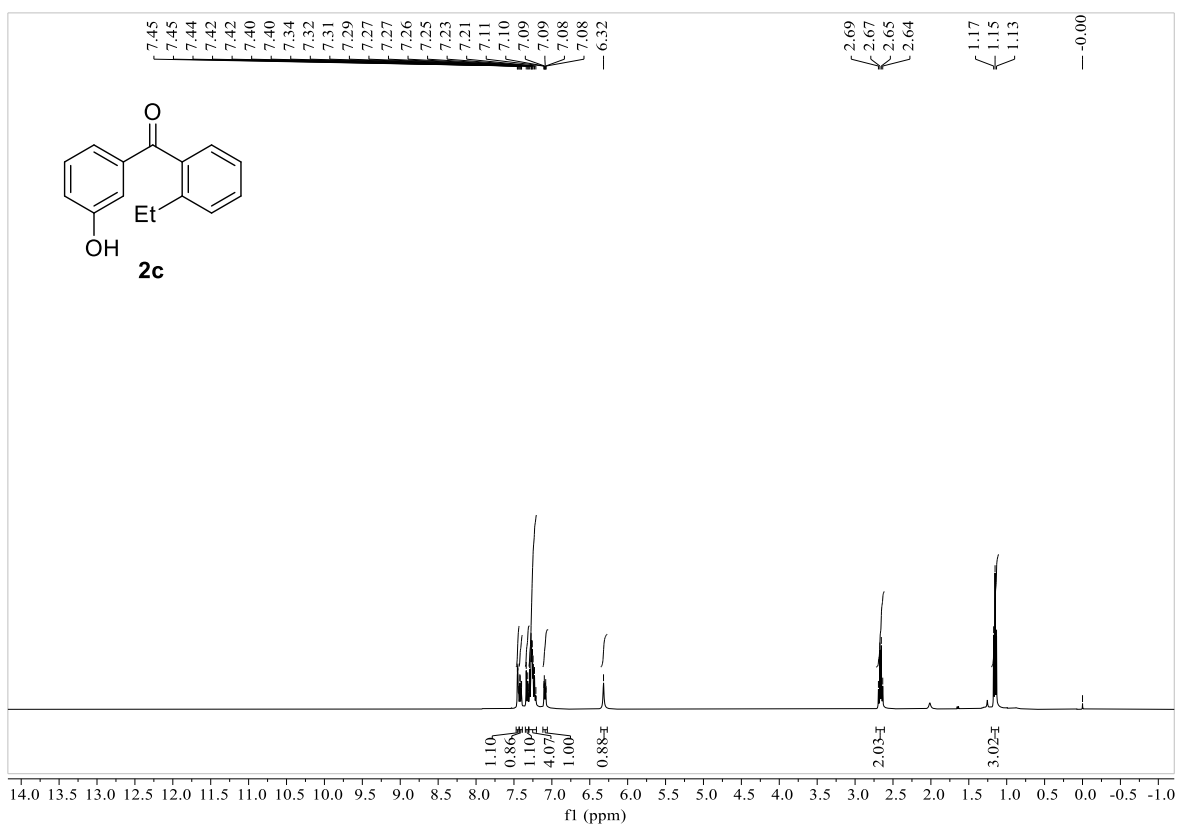
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2a**



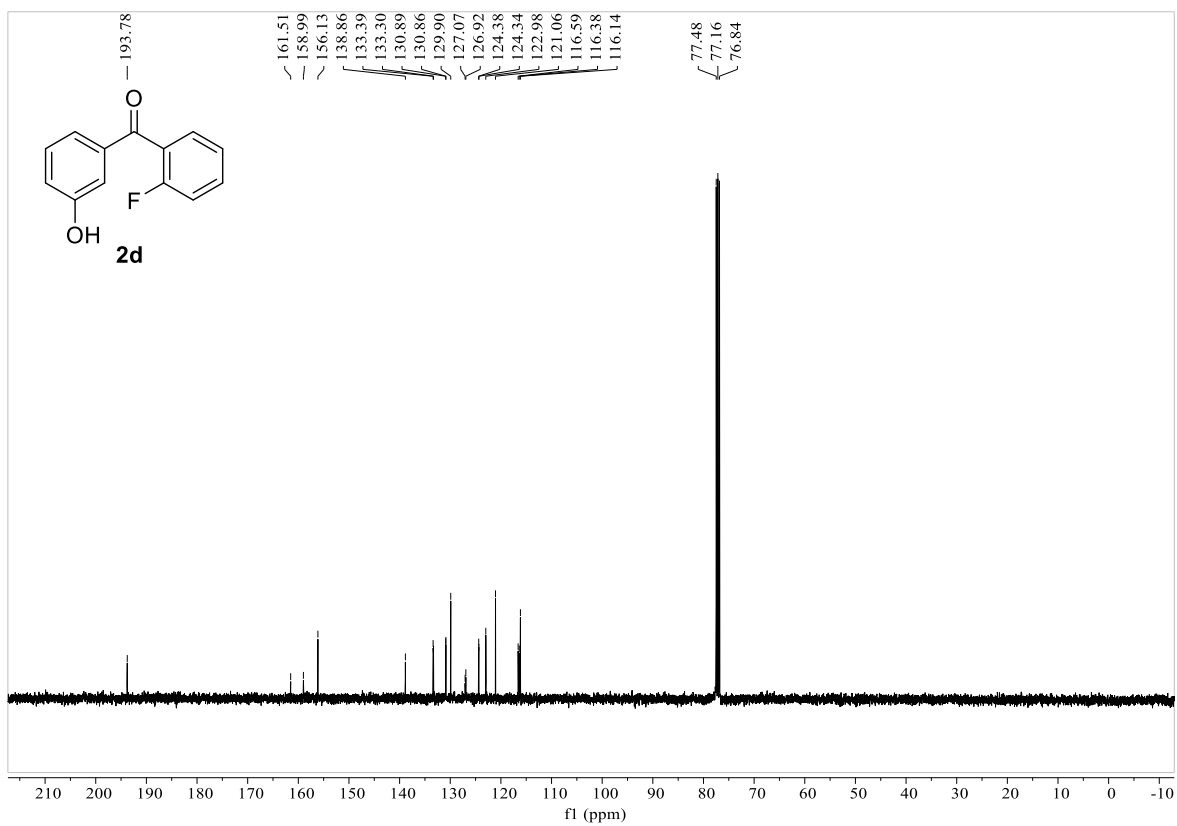
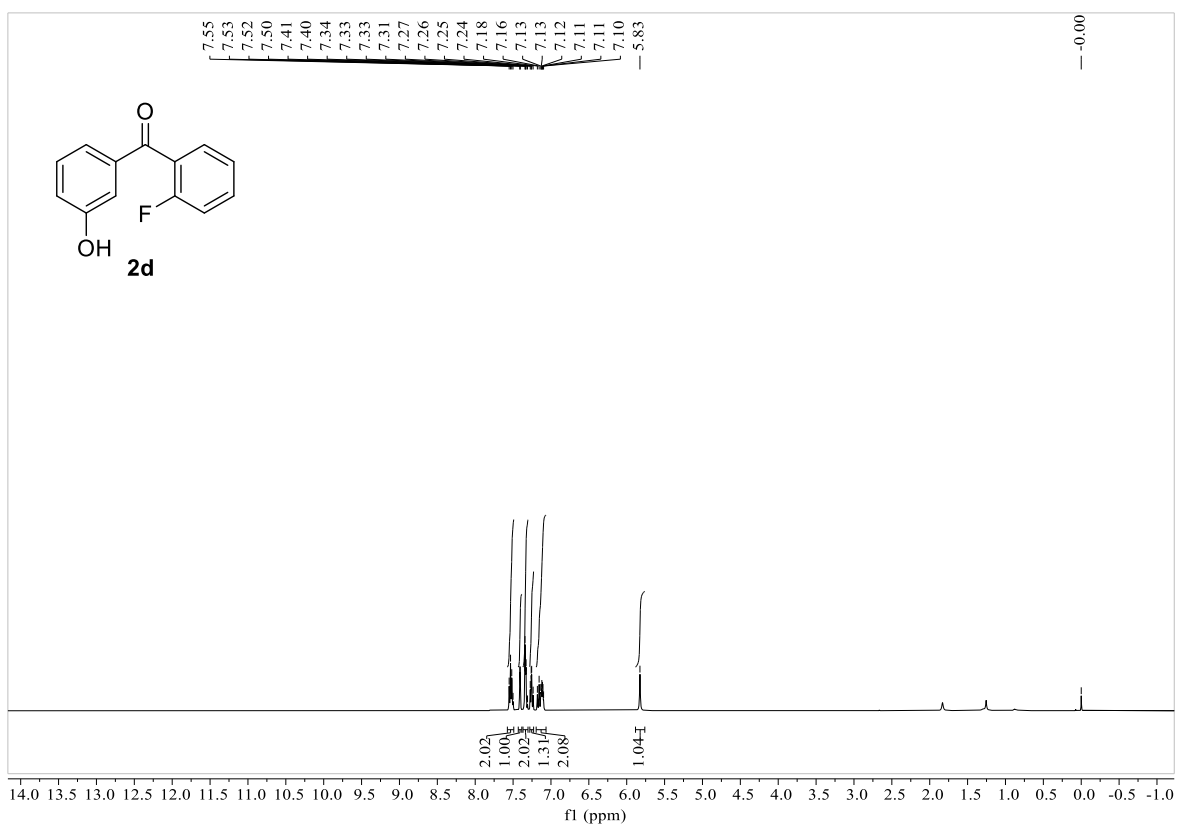
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2b**



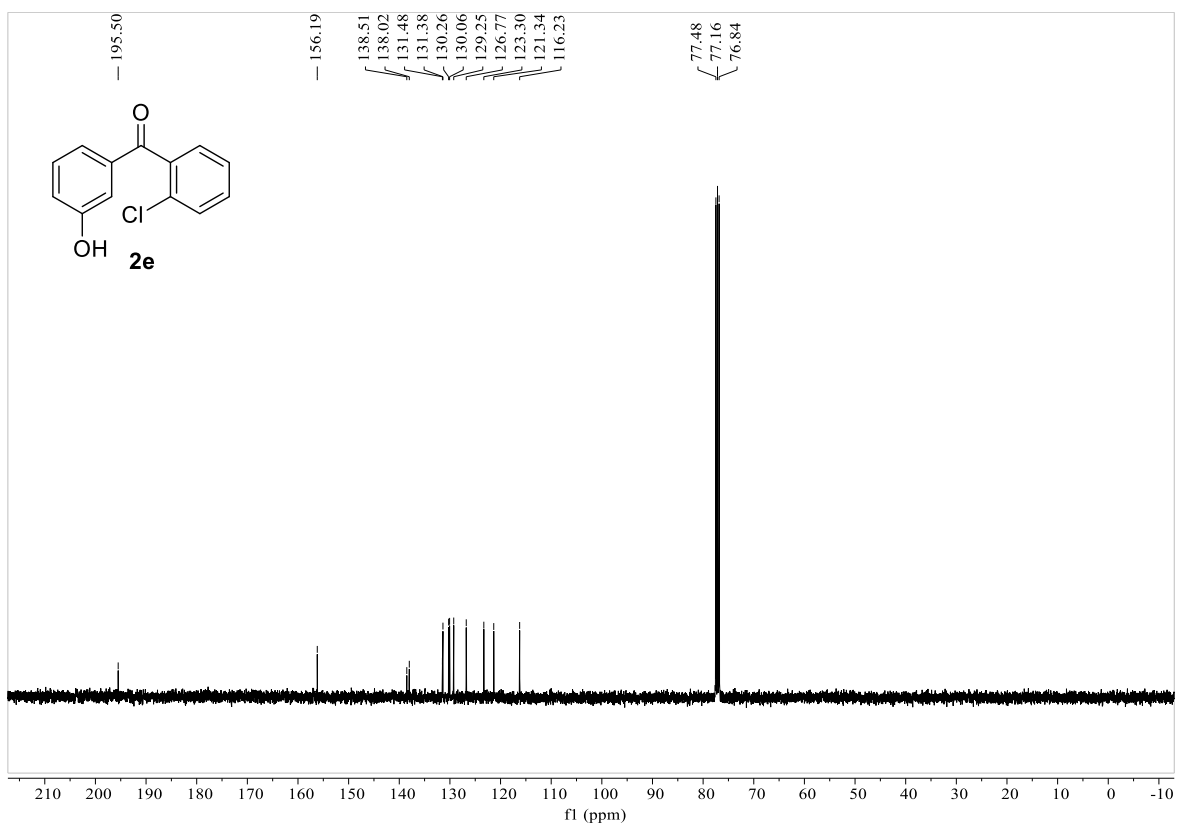
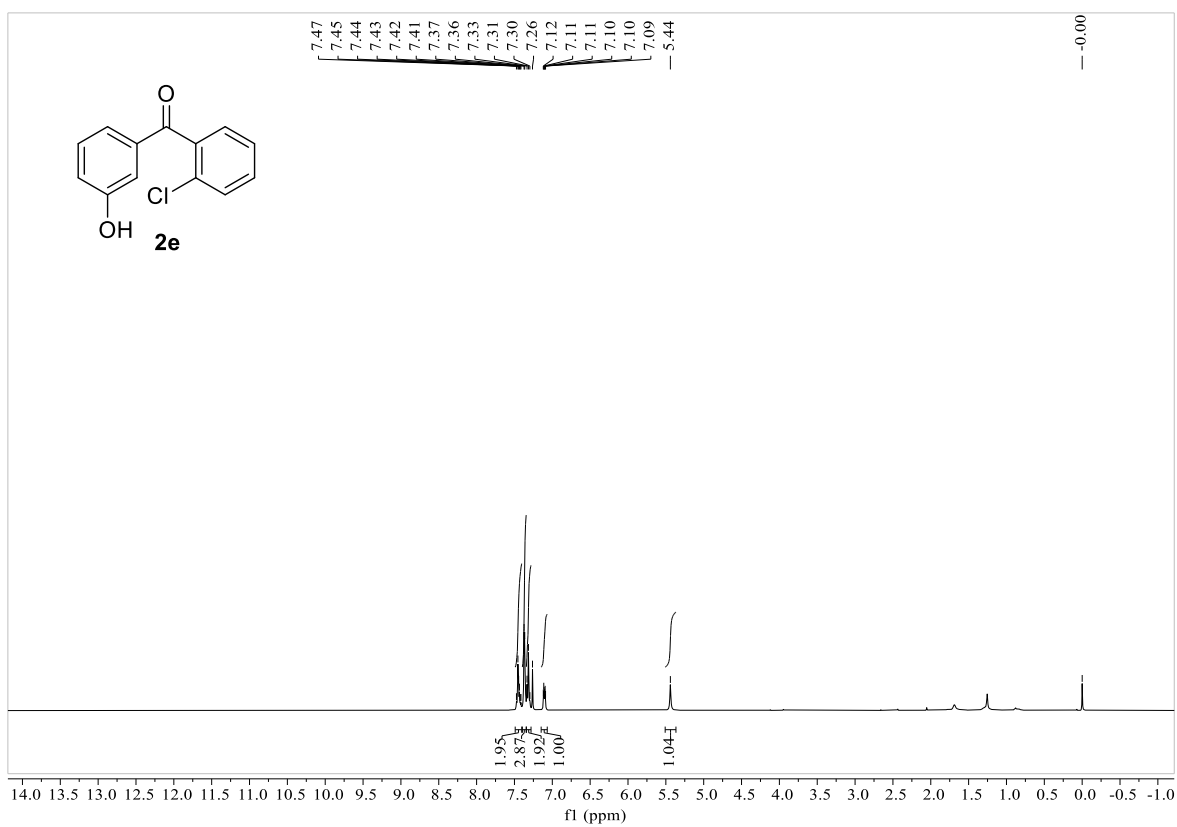
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2c**



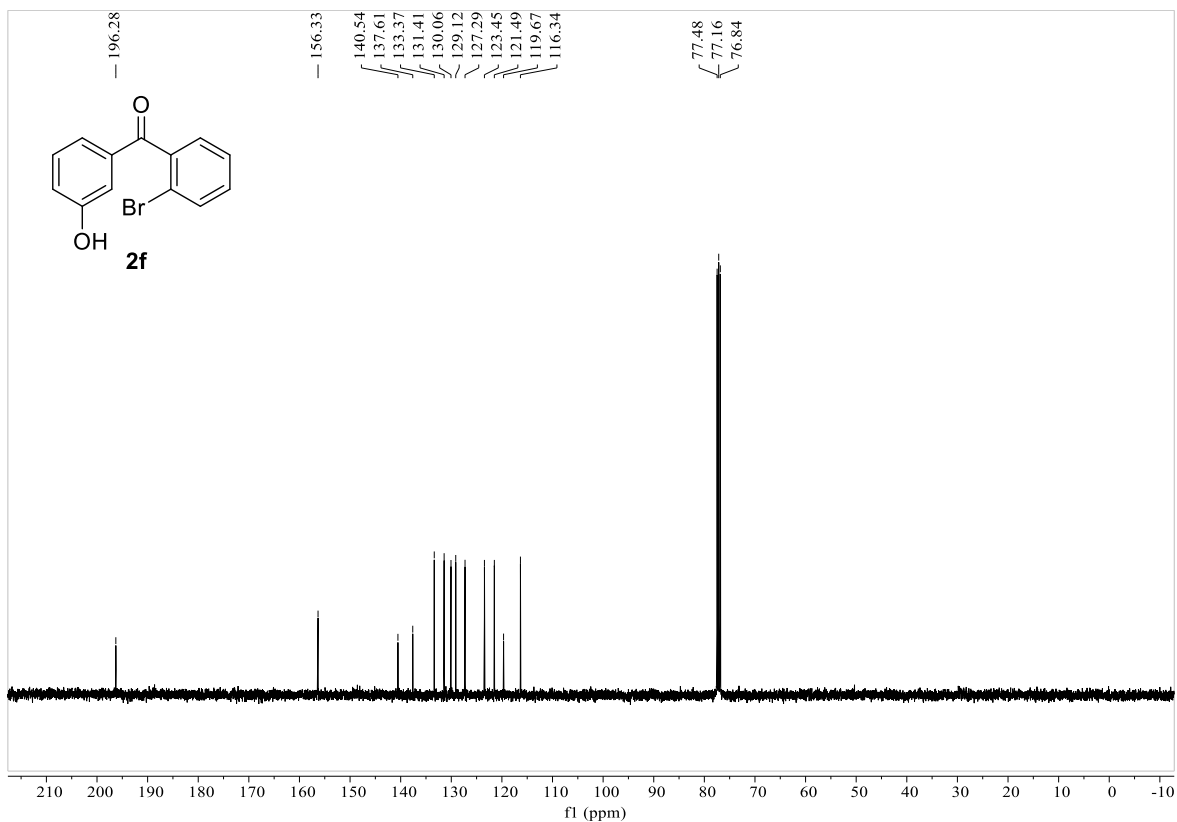
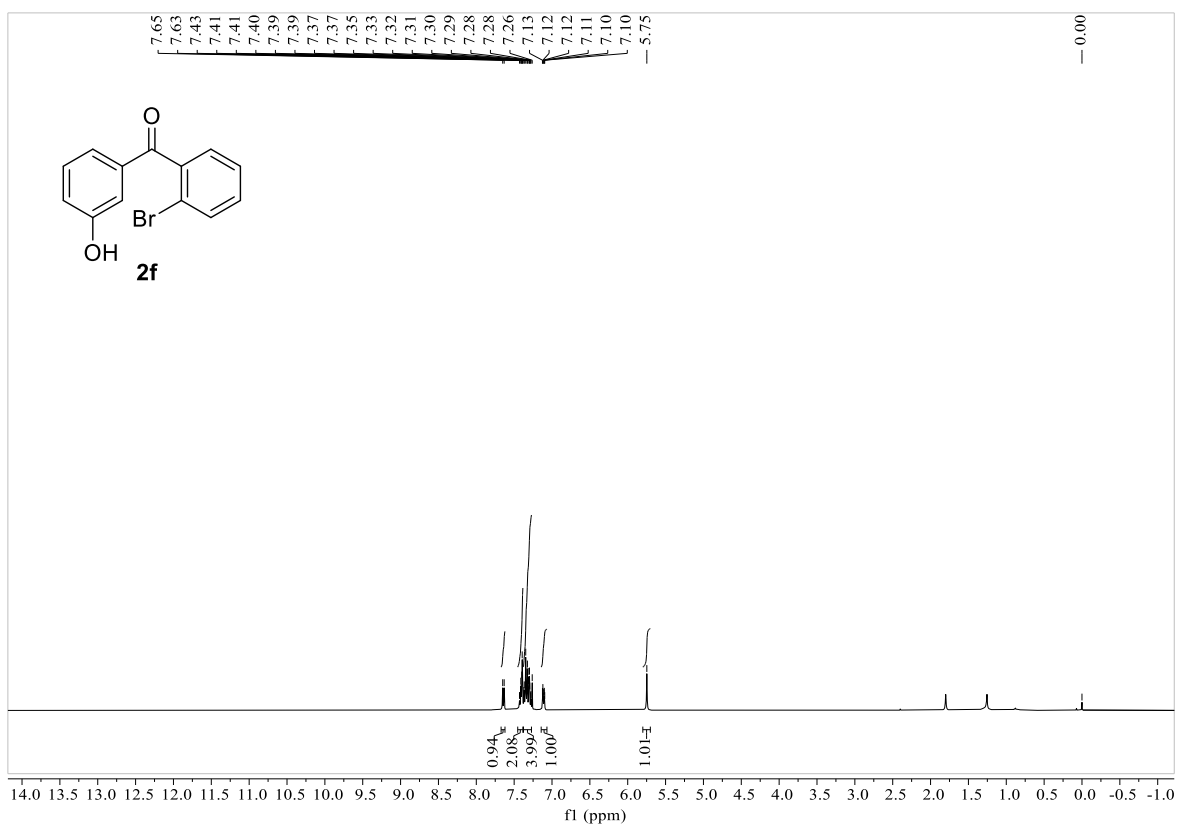
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2d**



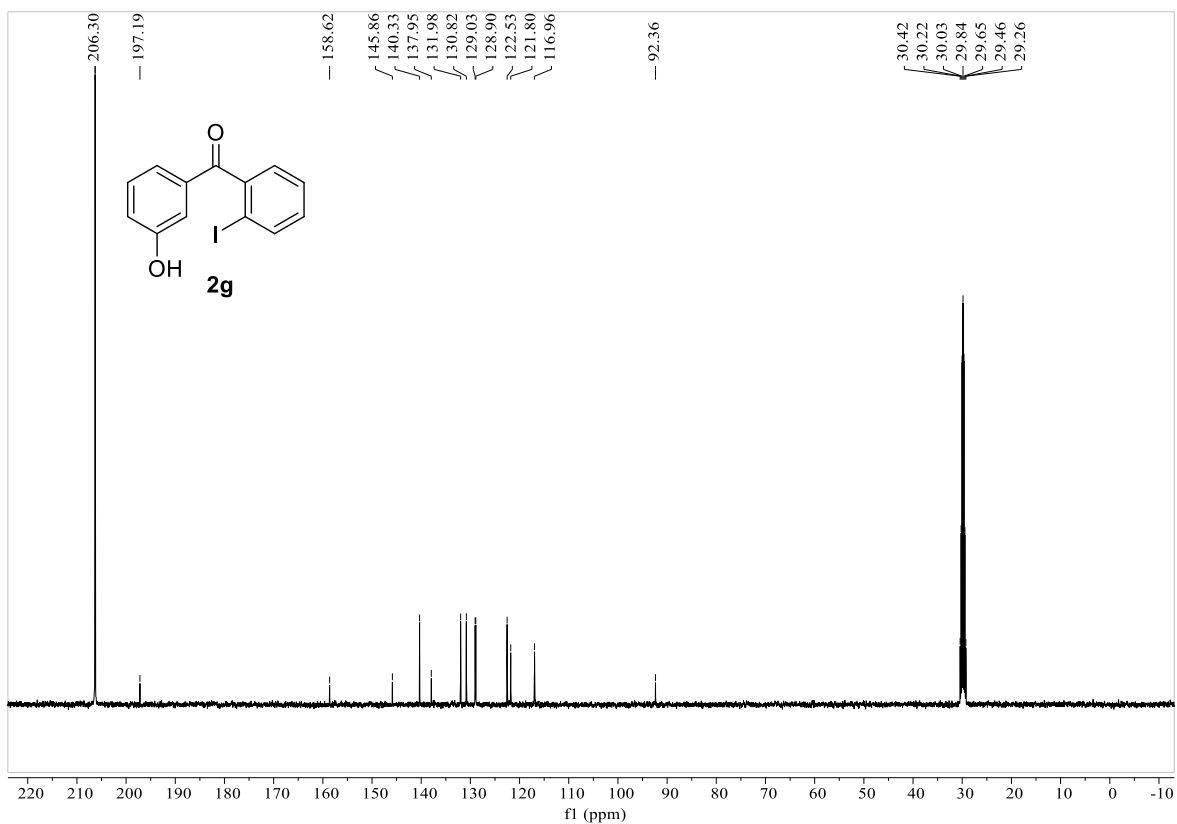
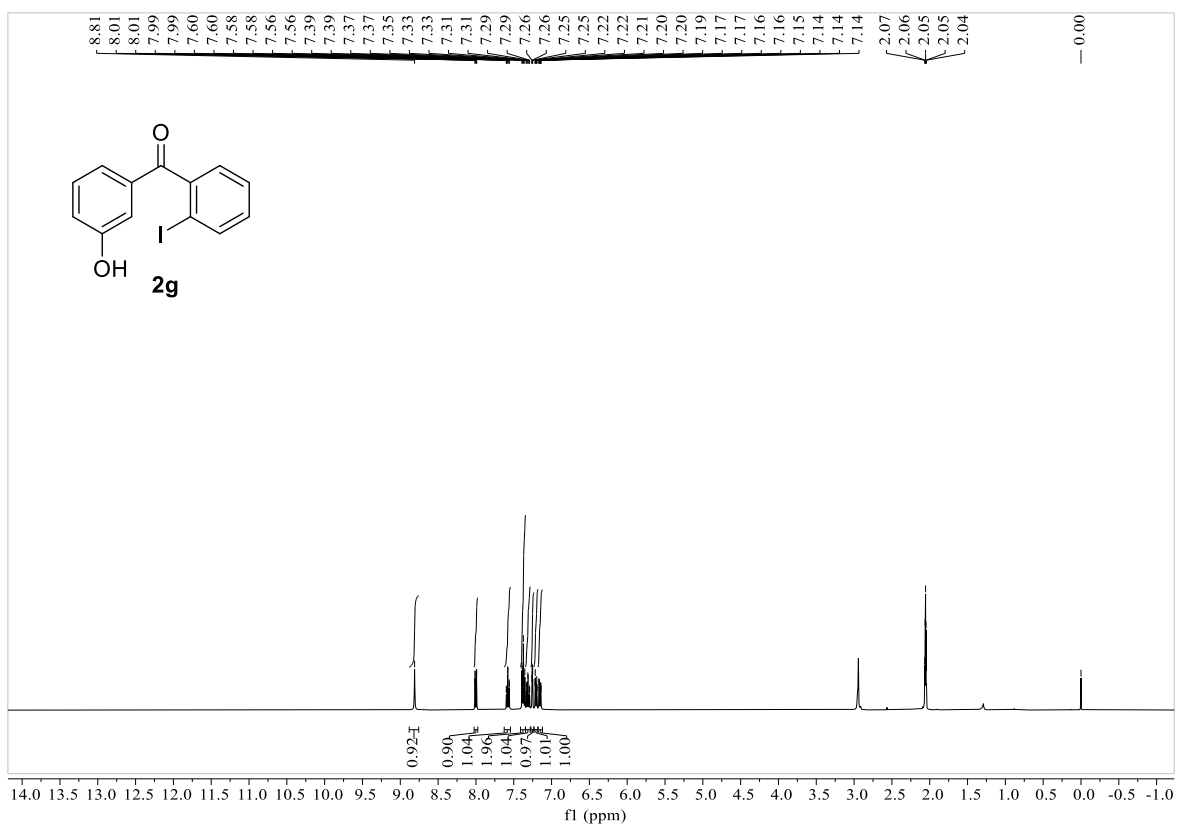
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2e**



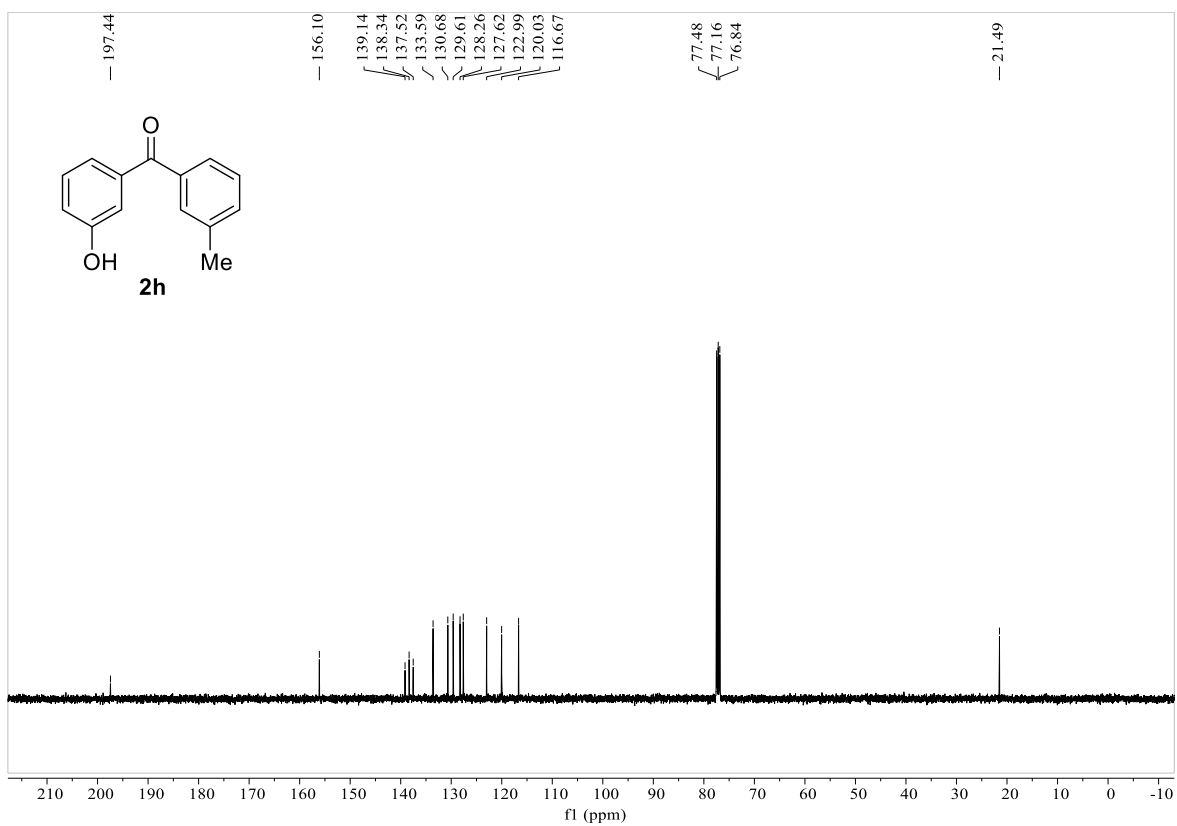
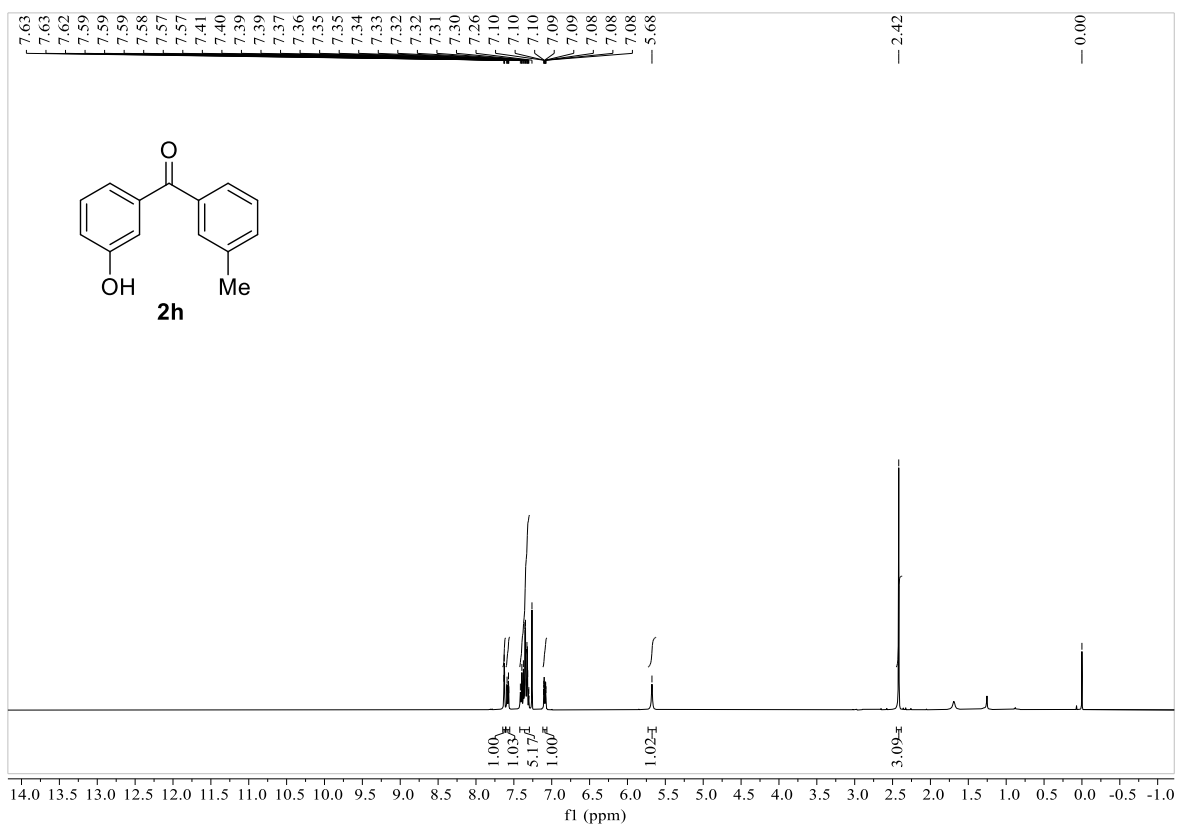
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2f**



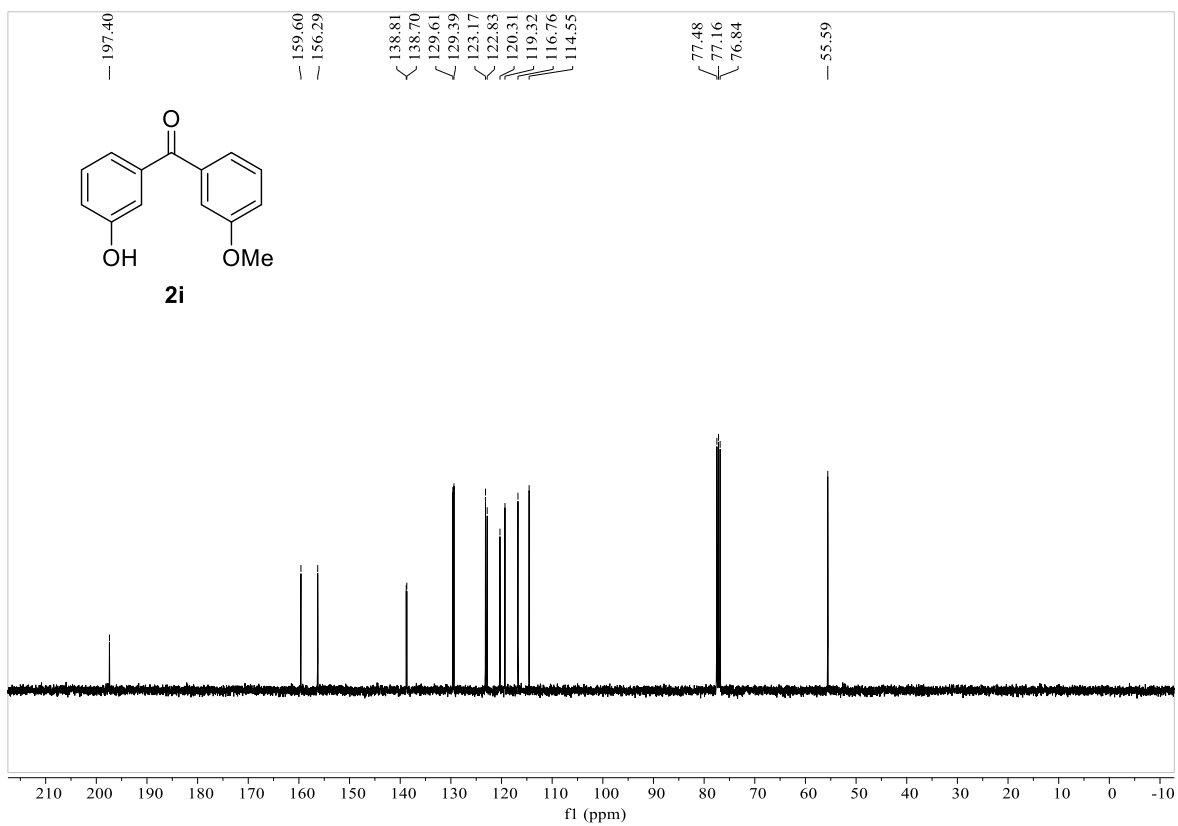
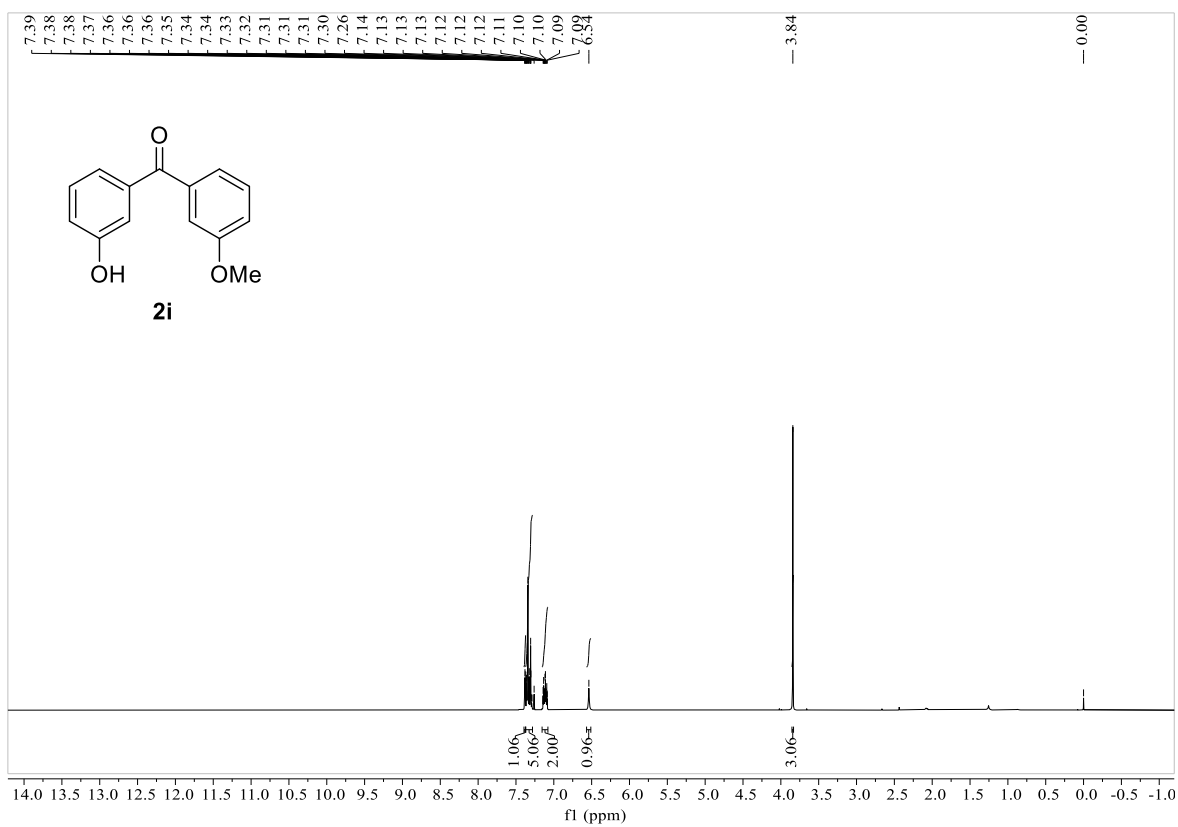
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2g**



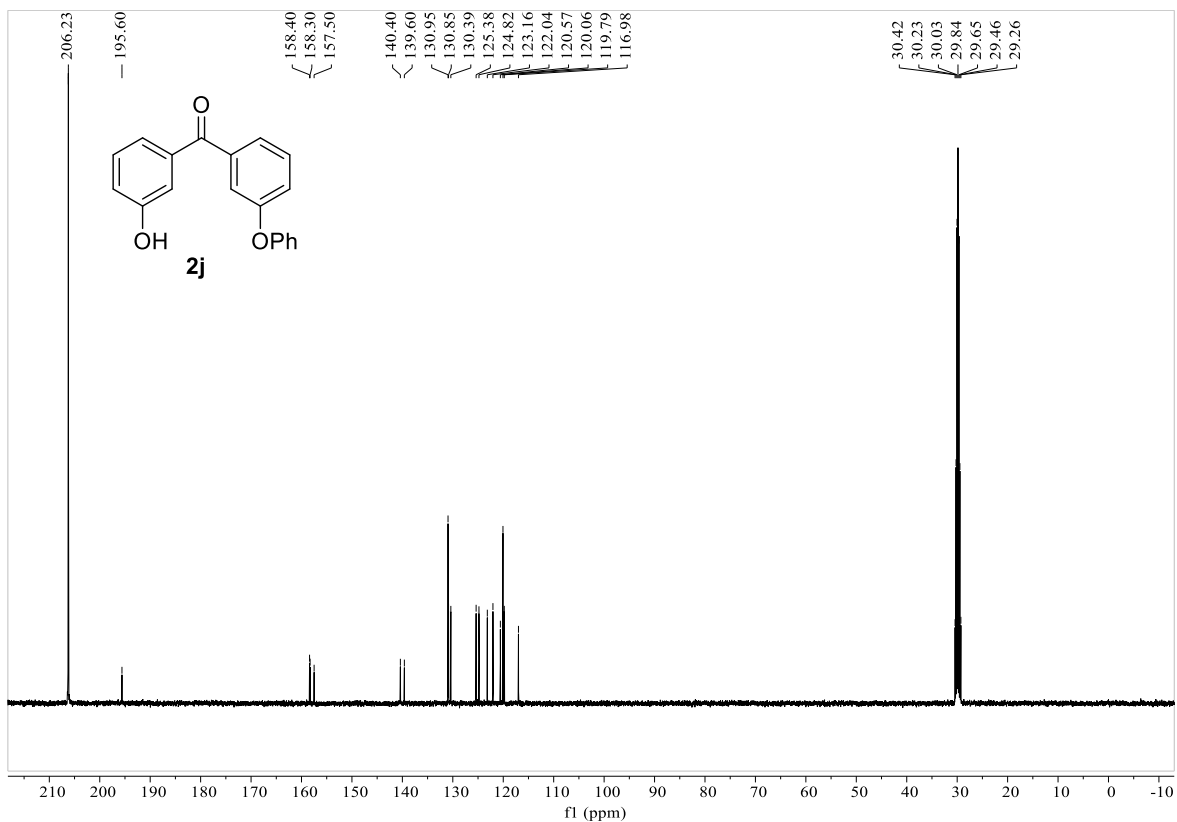
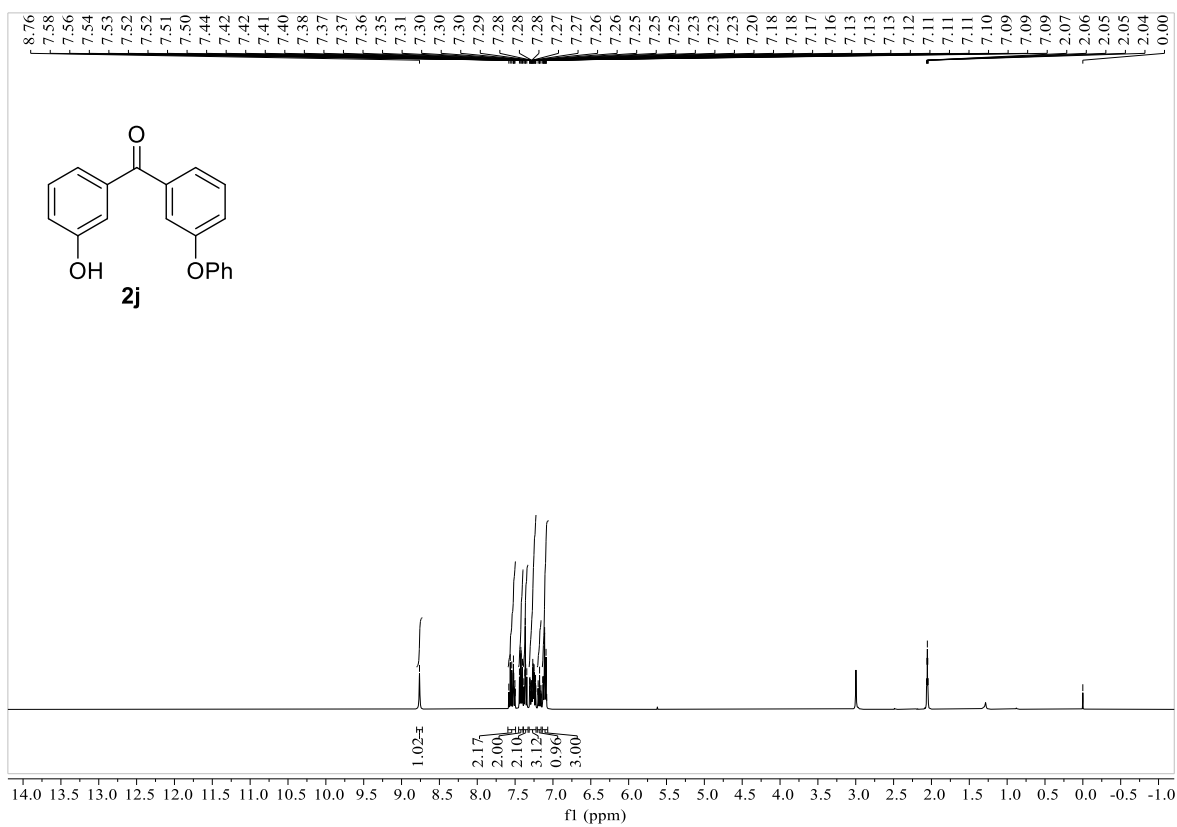
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2h**



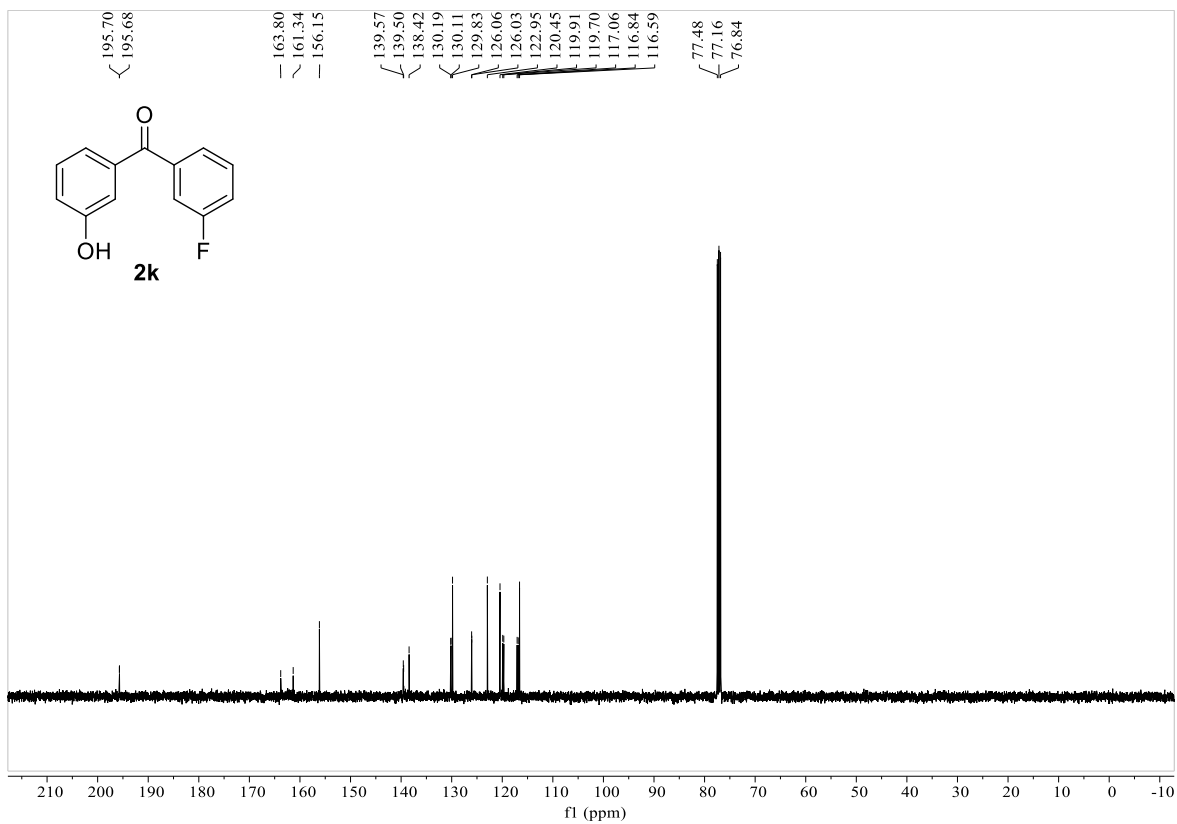
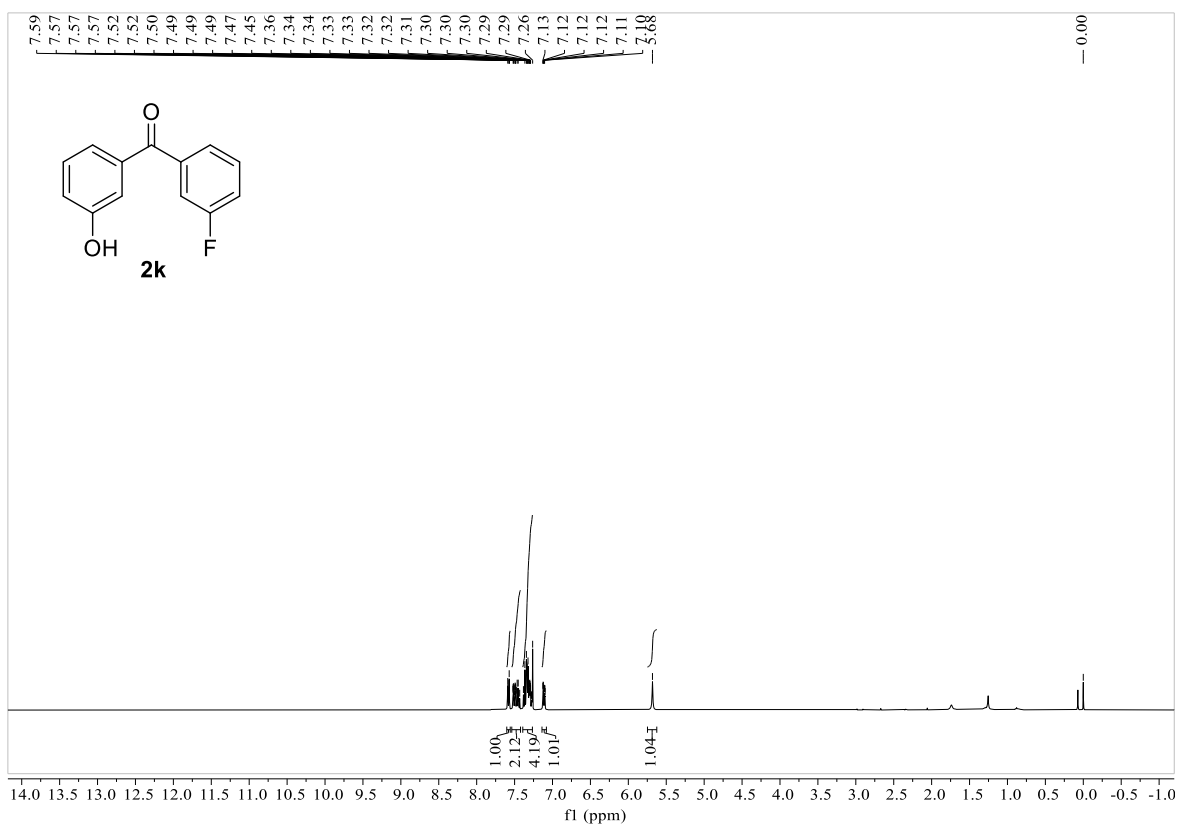
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2i**



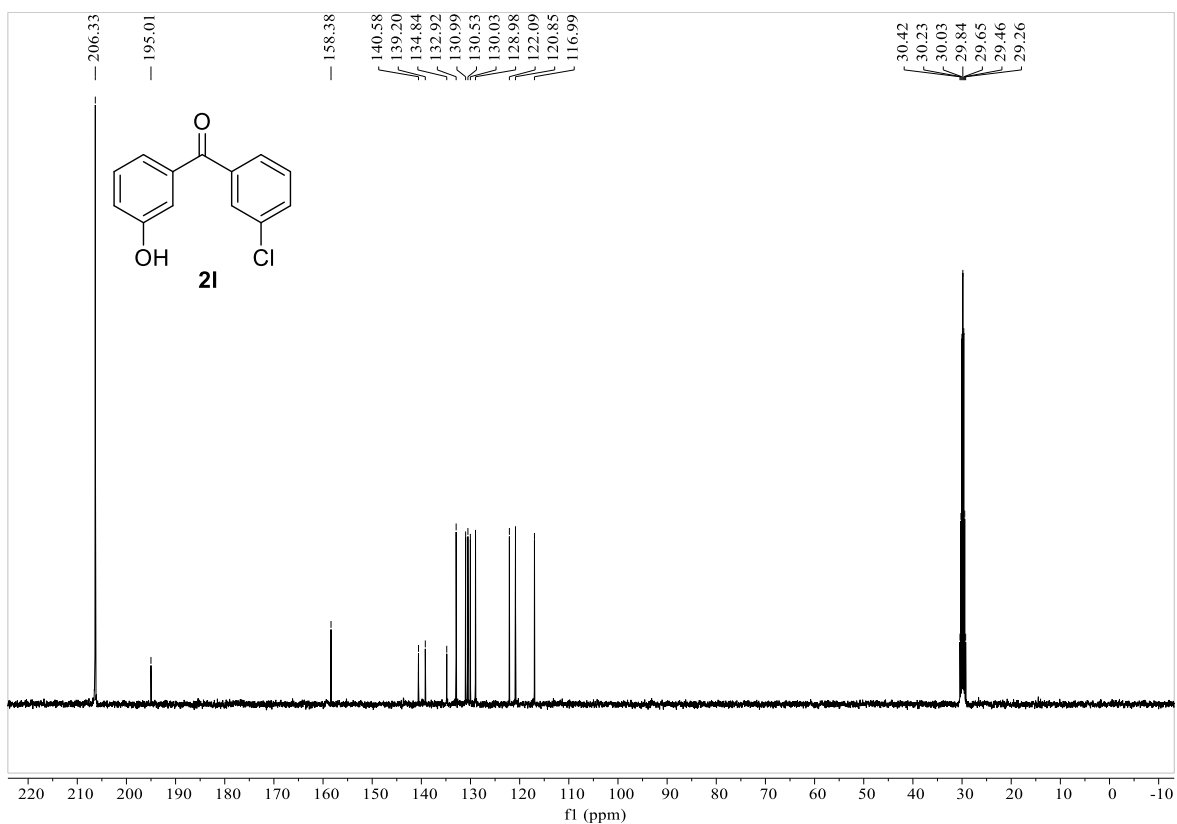
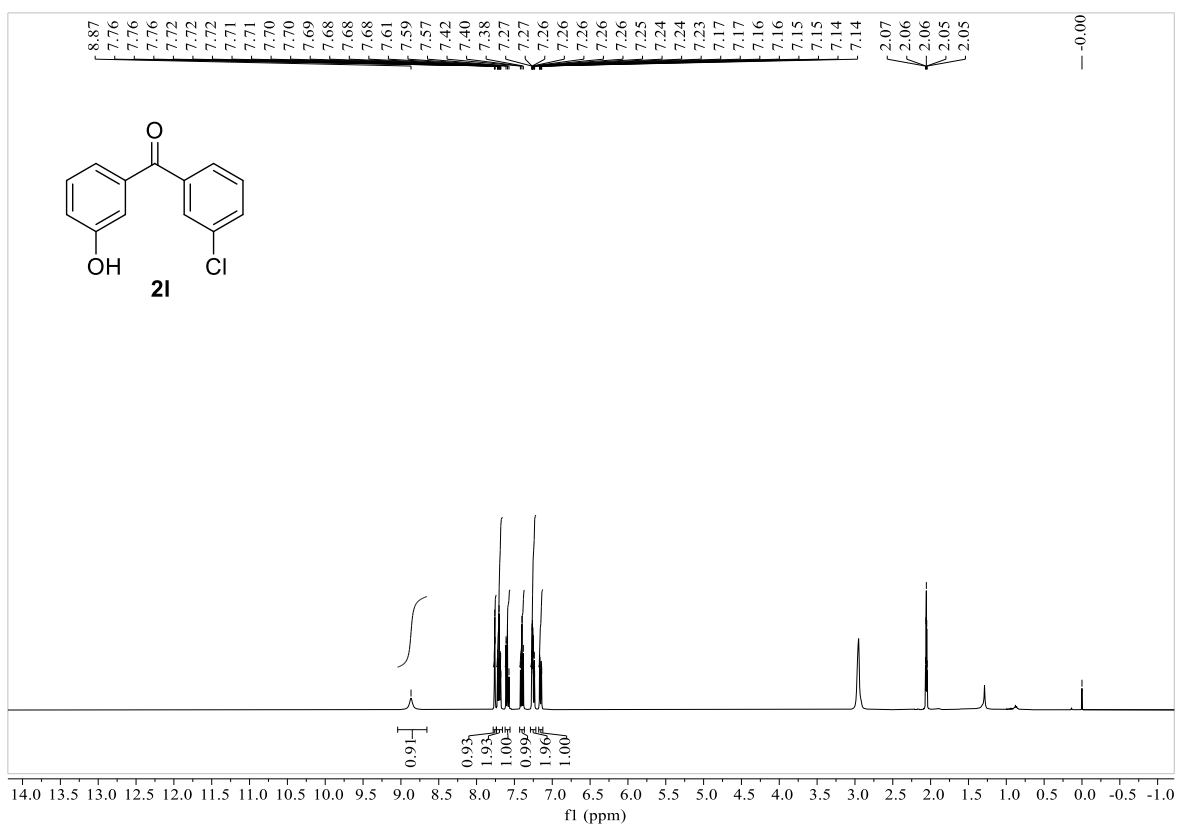
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2j**



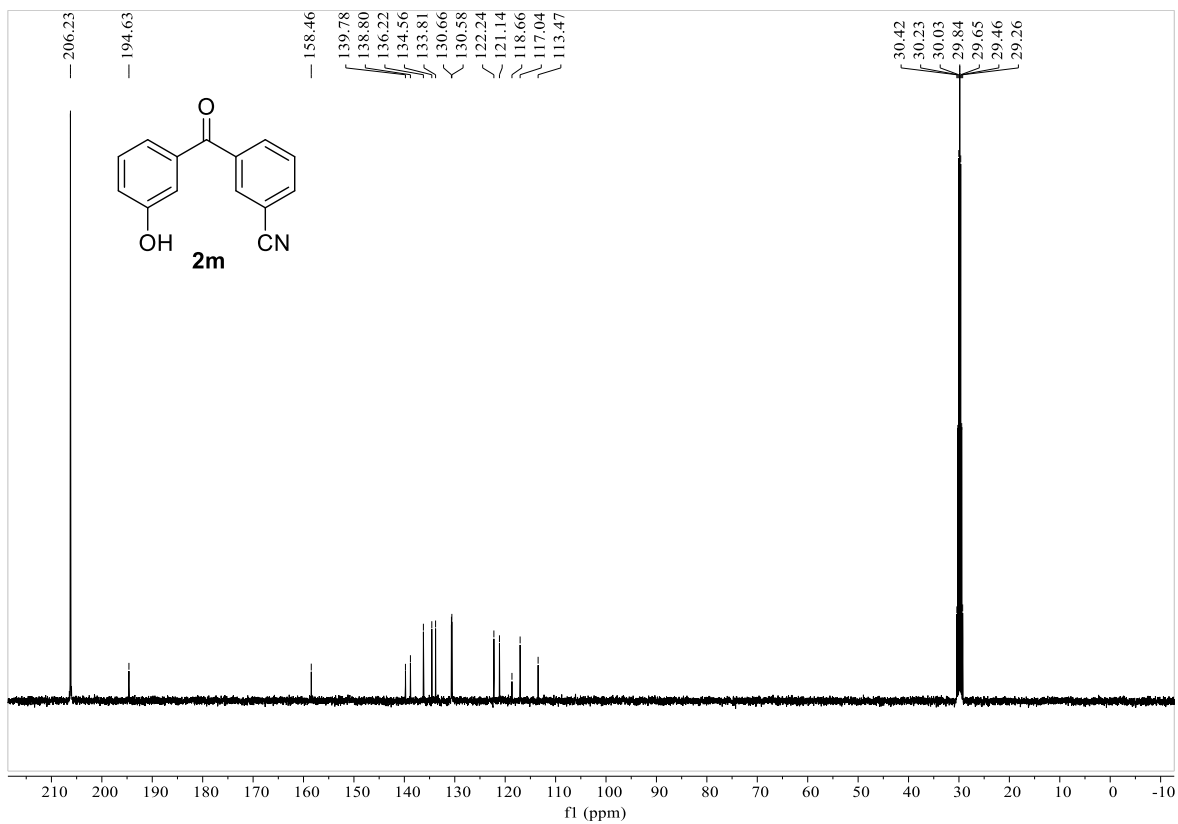
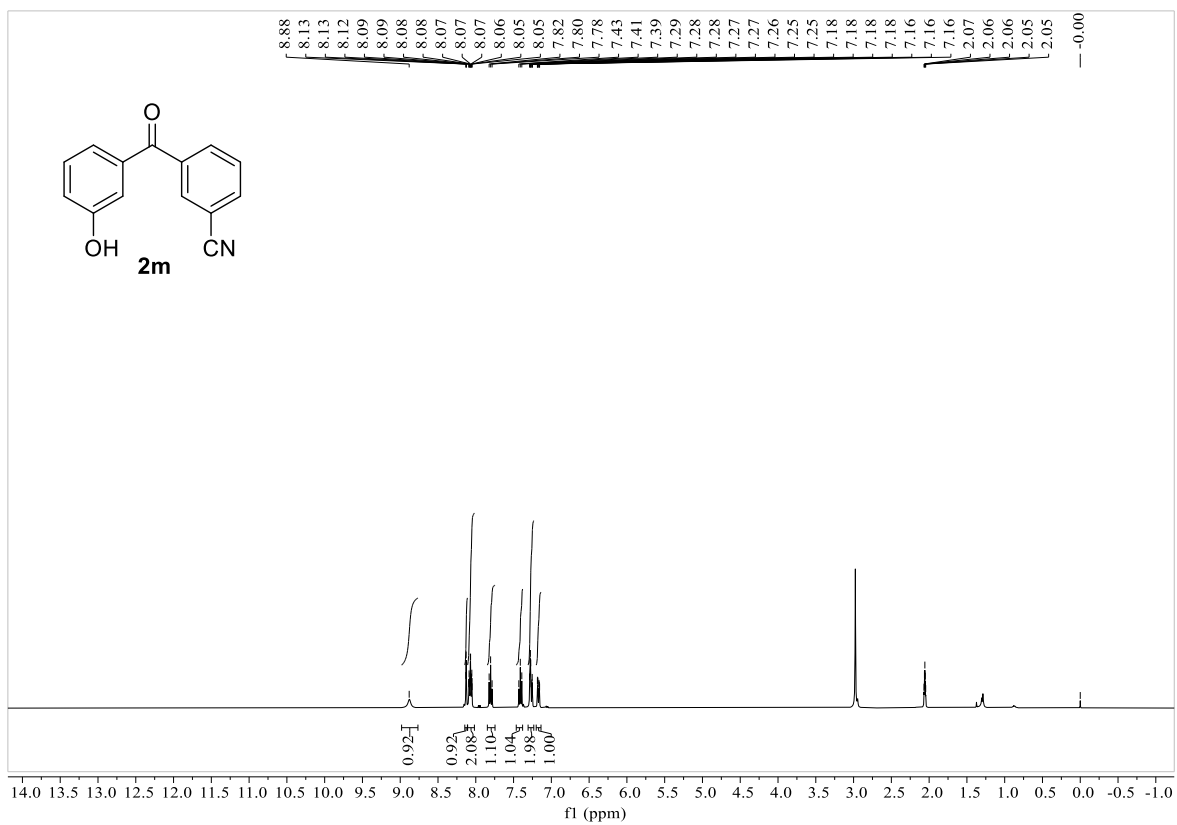
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2k**



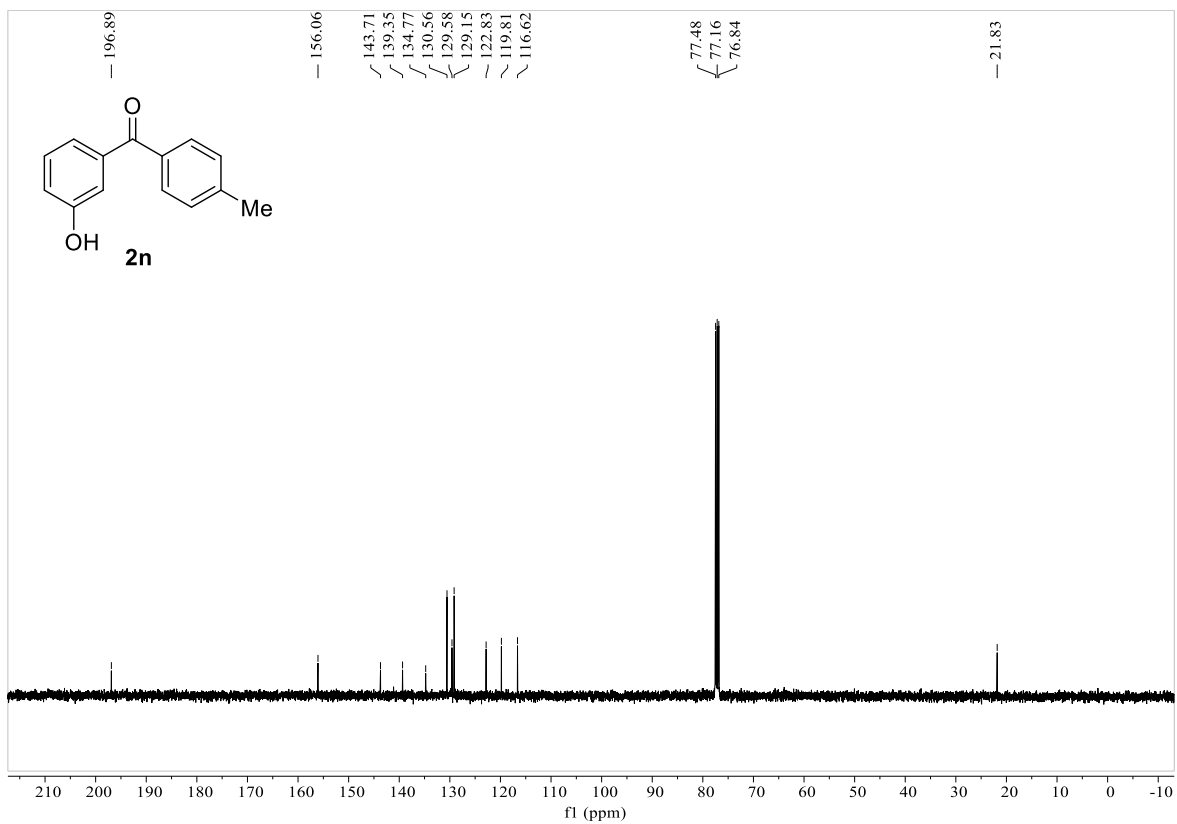
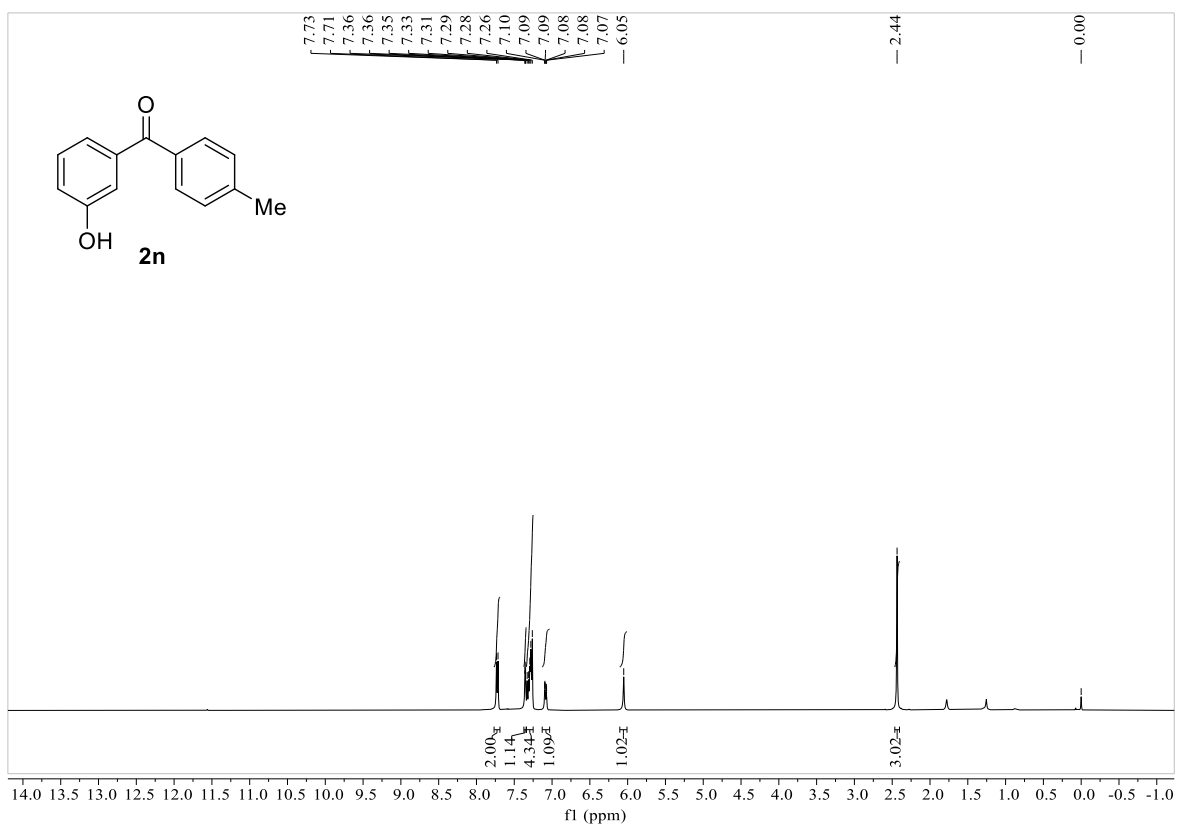
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2l**



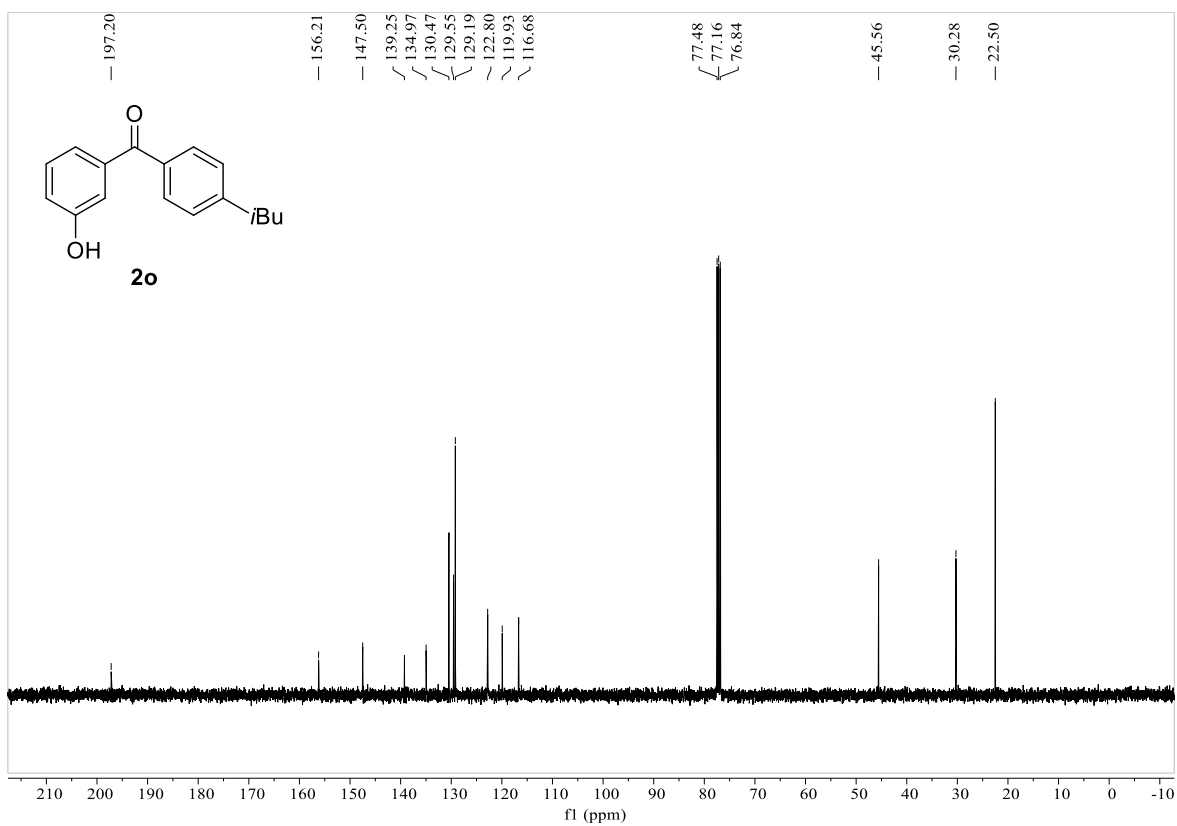
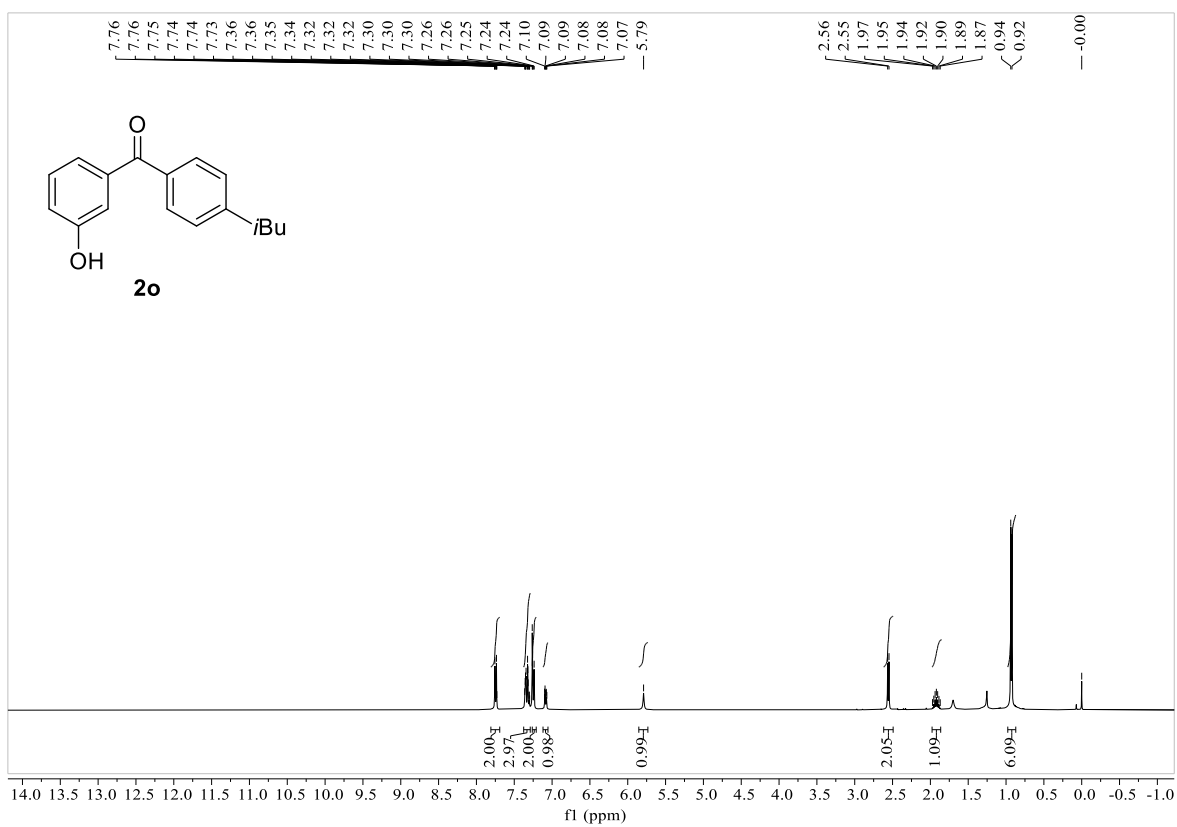
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2m**



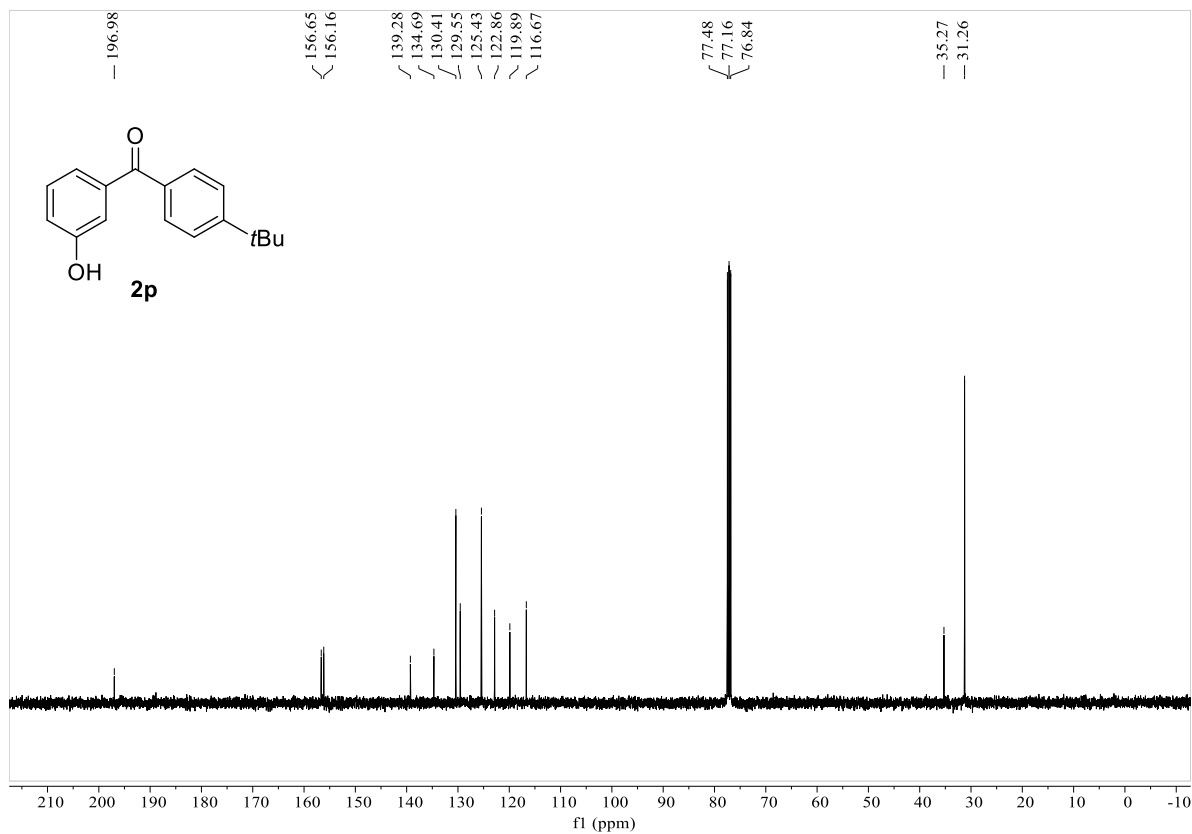
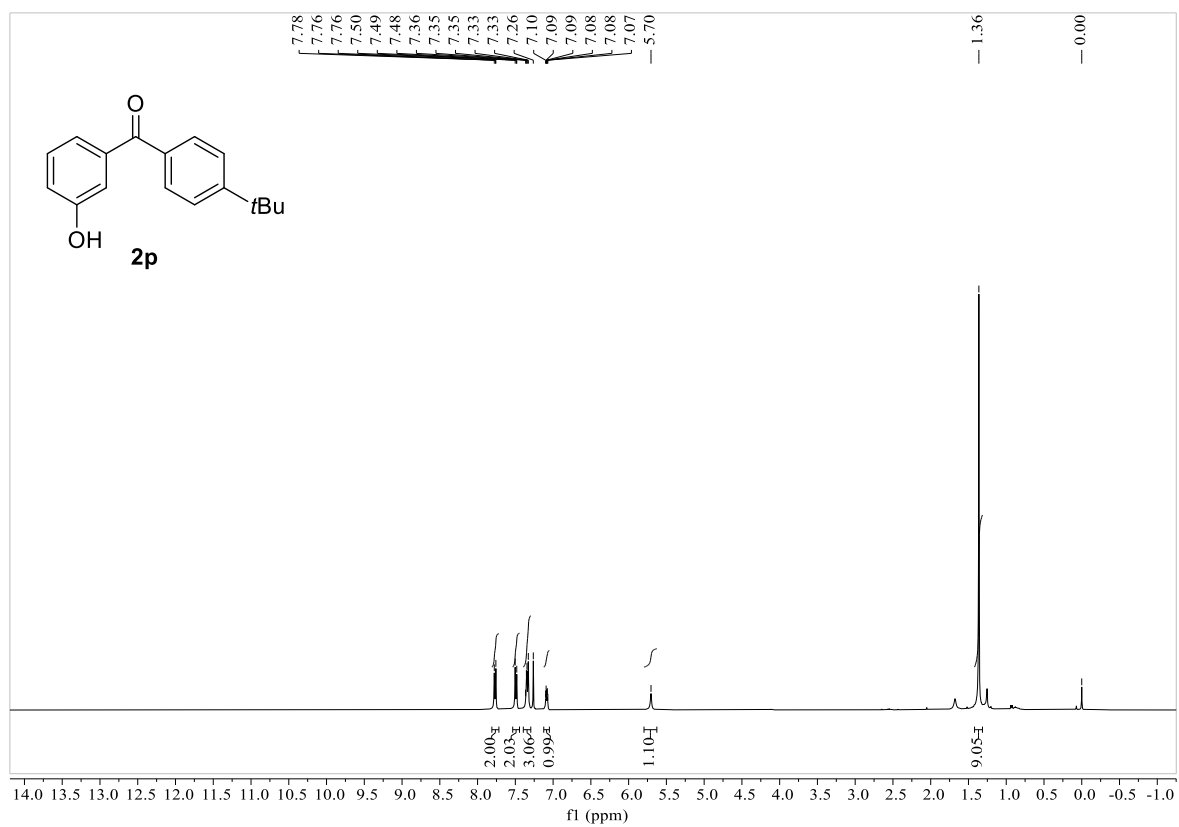
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2n**



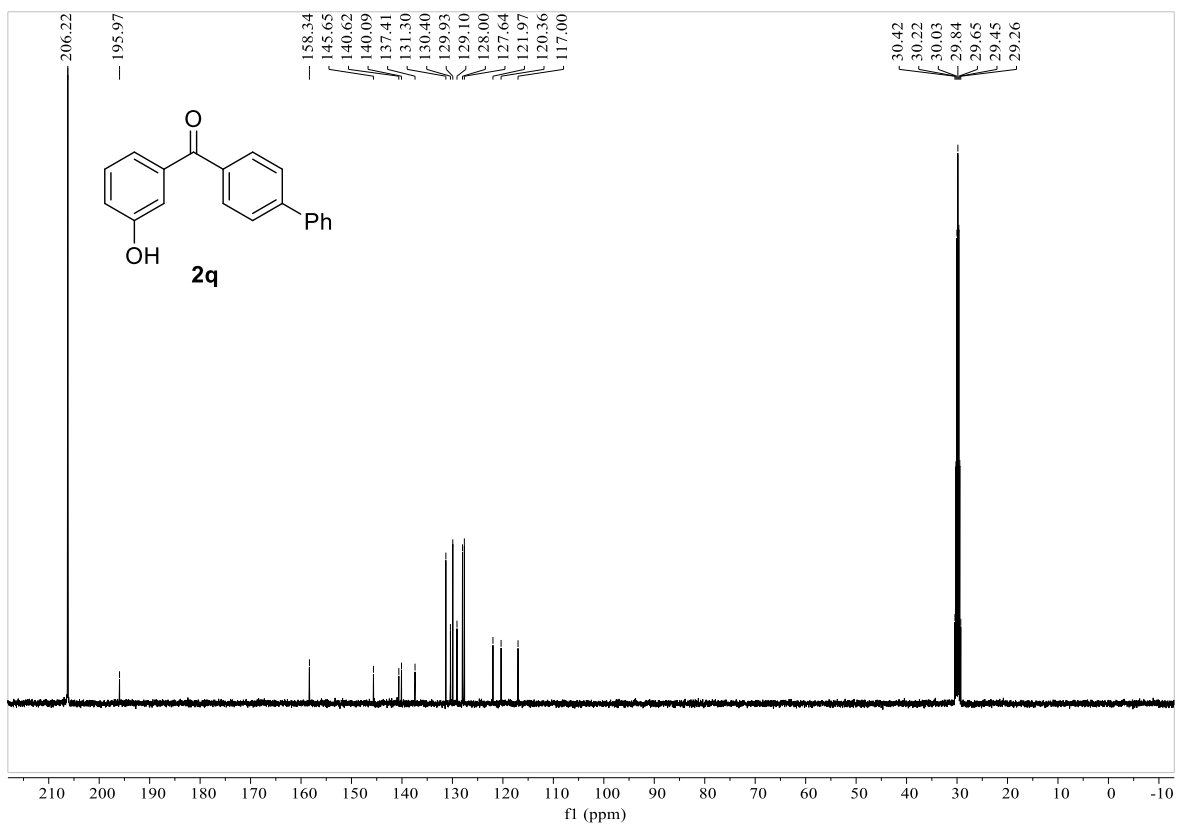
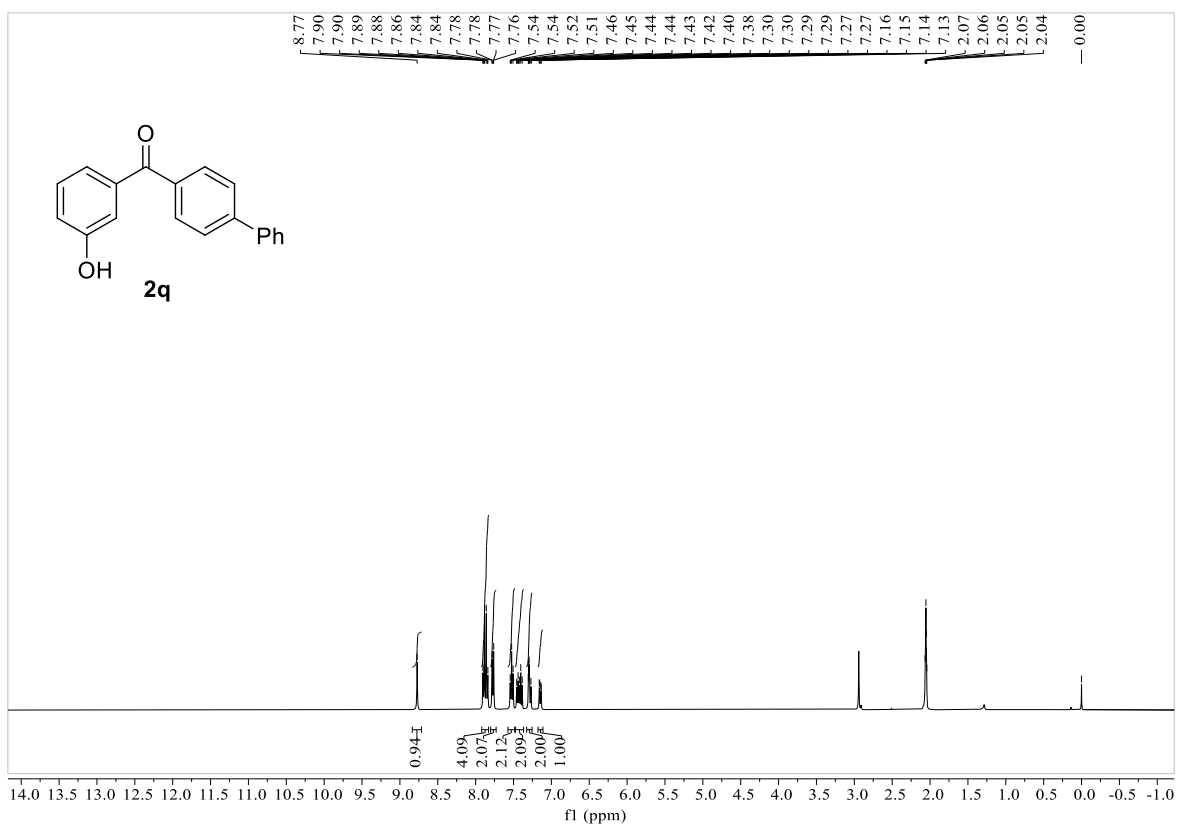
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2o**



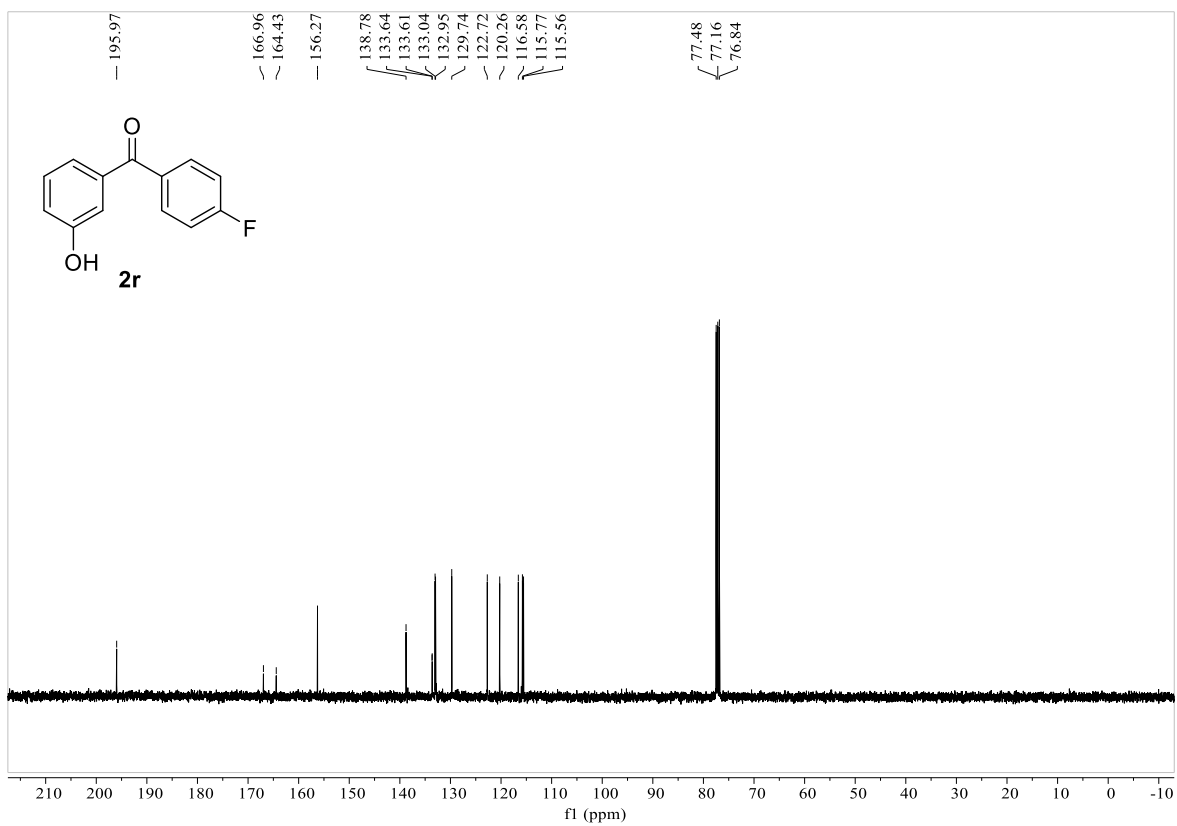
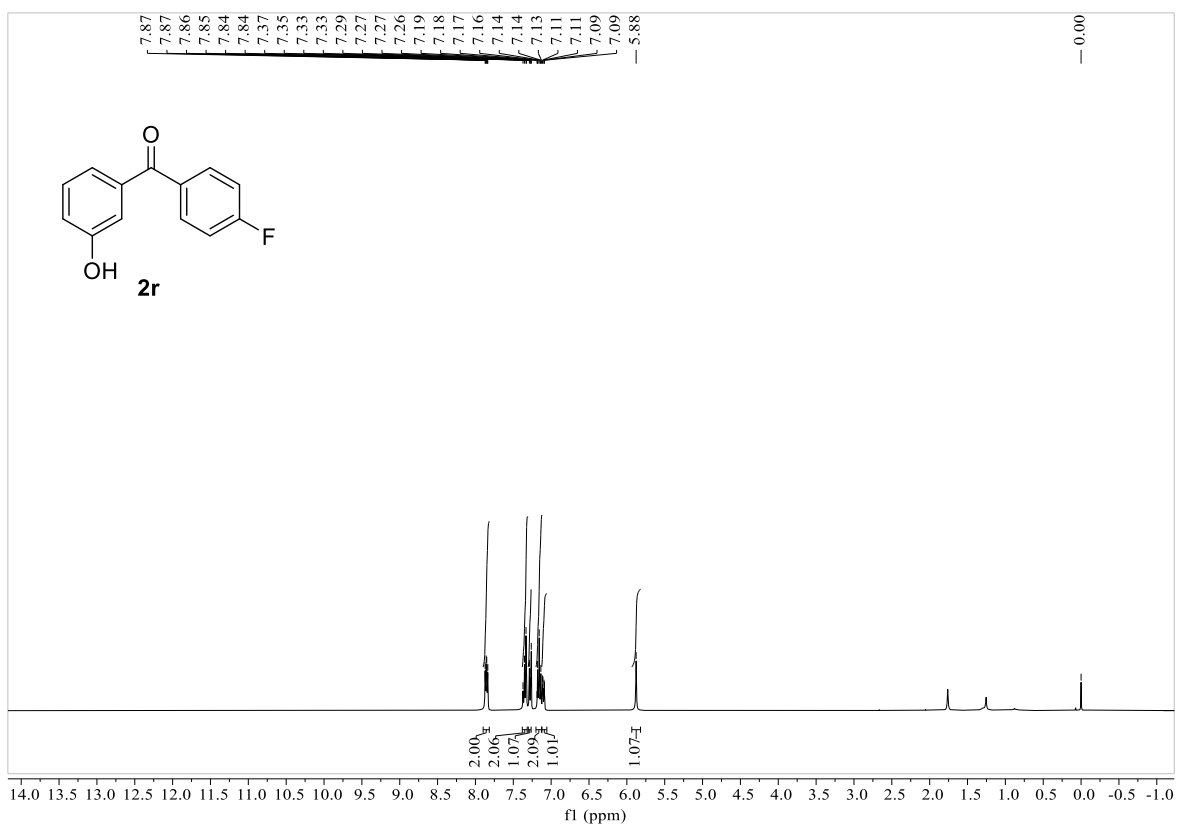
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2p**



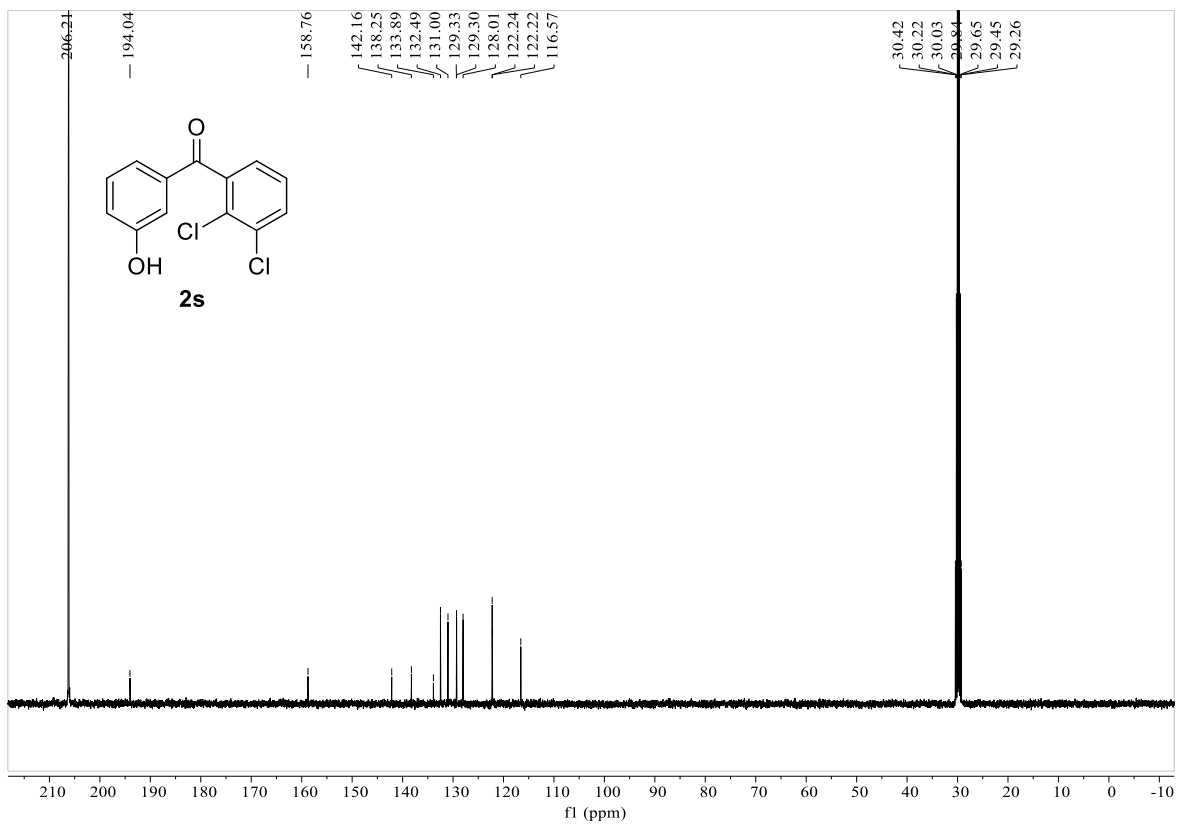
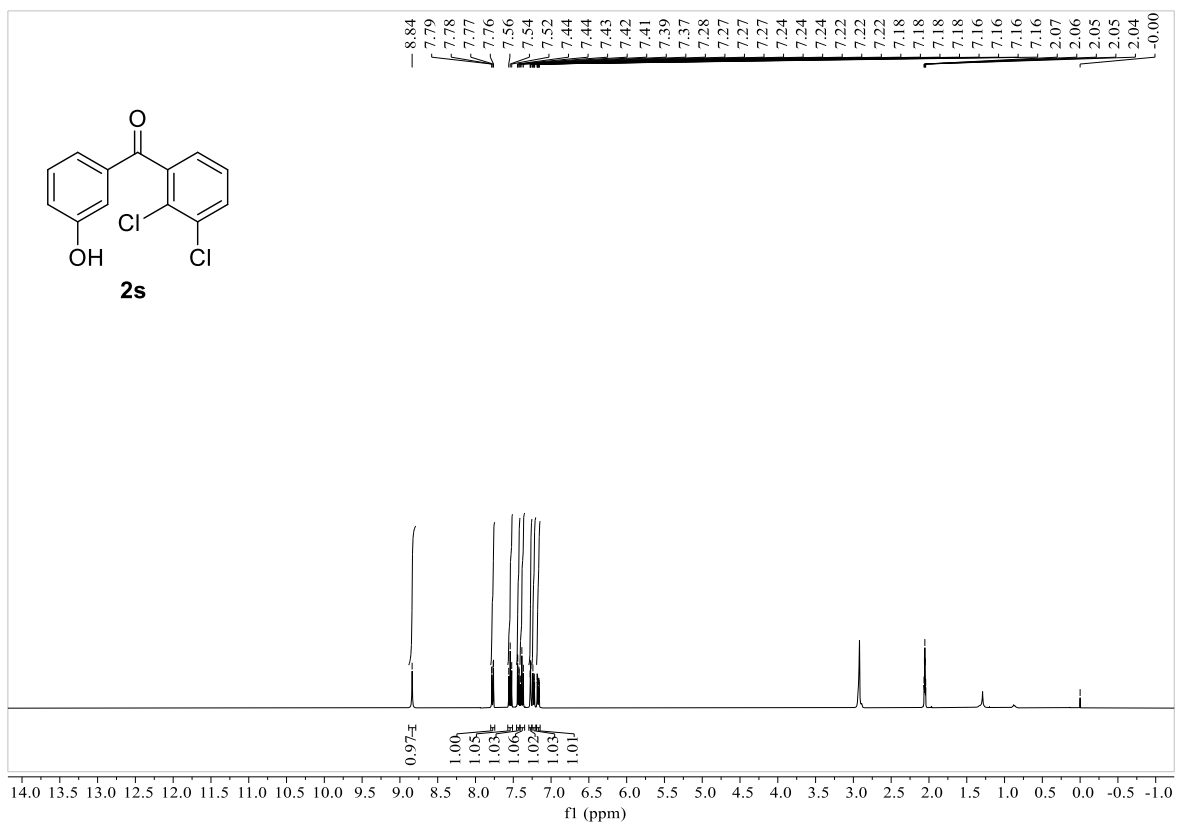
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2q**



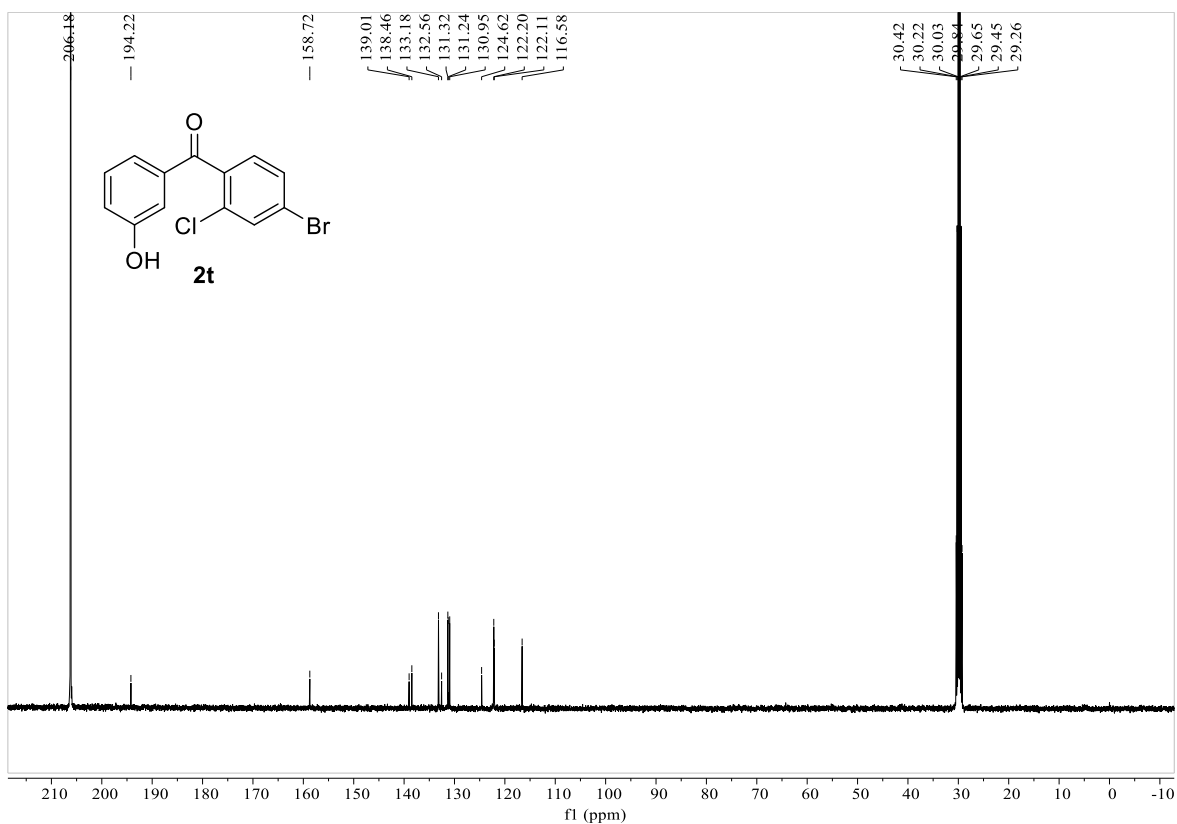
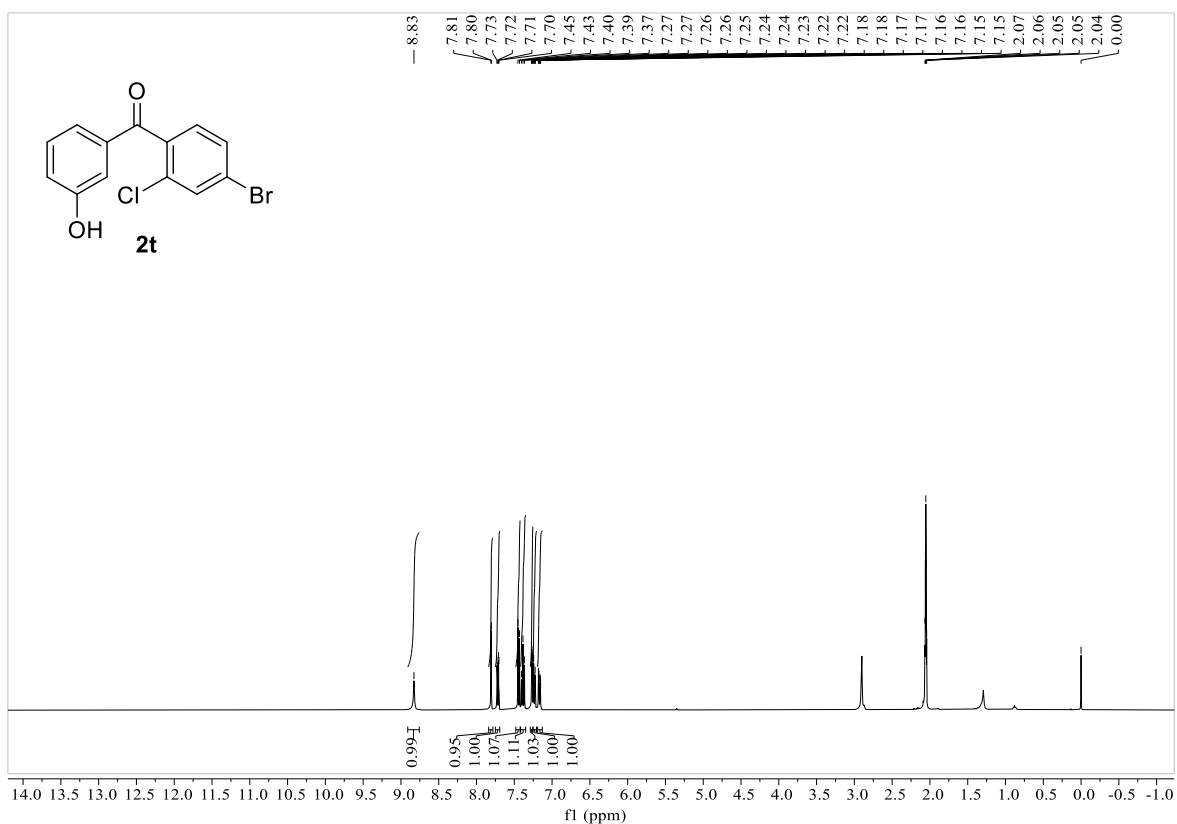
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2r**



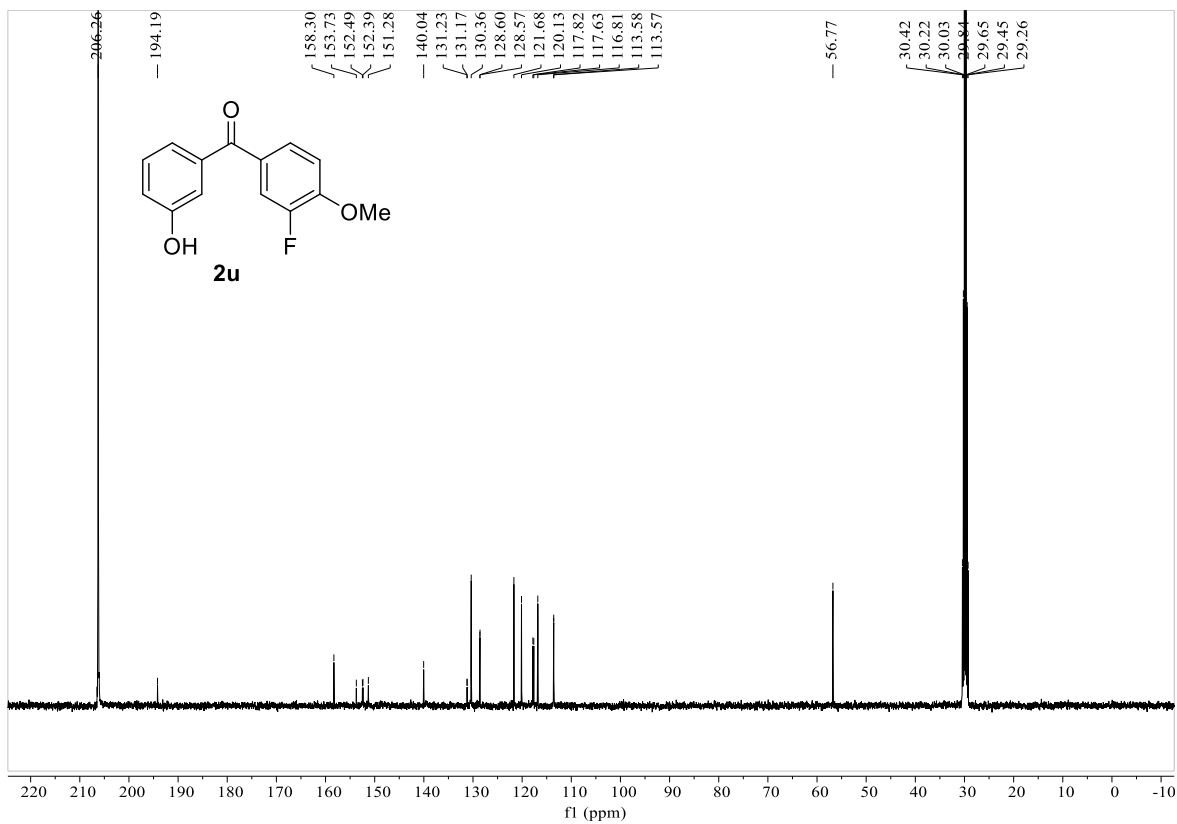
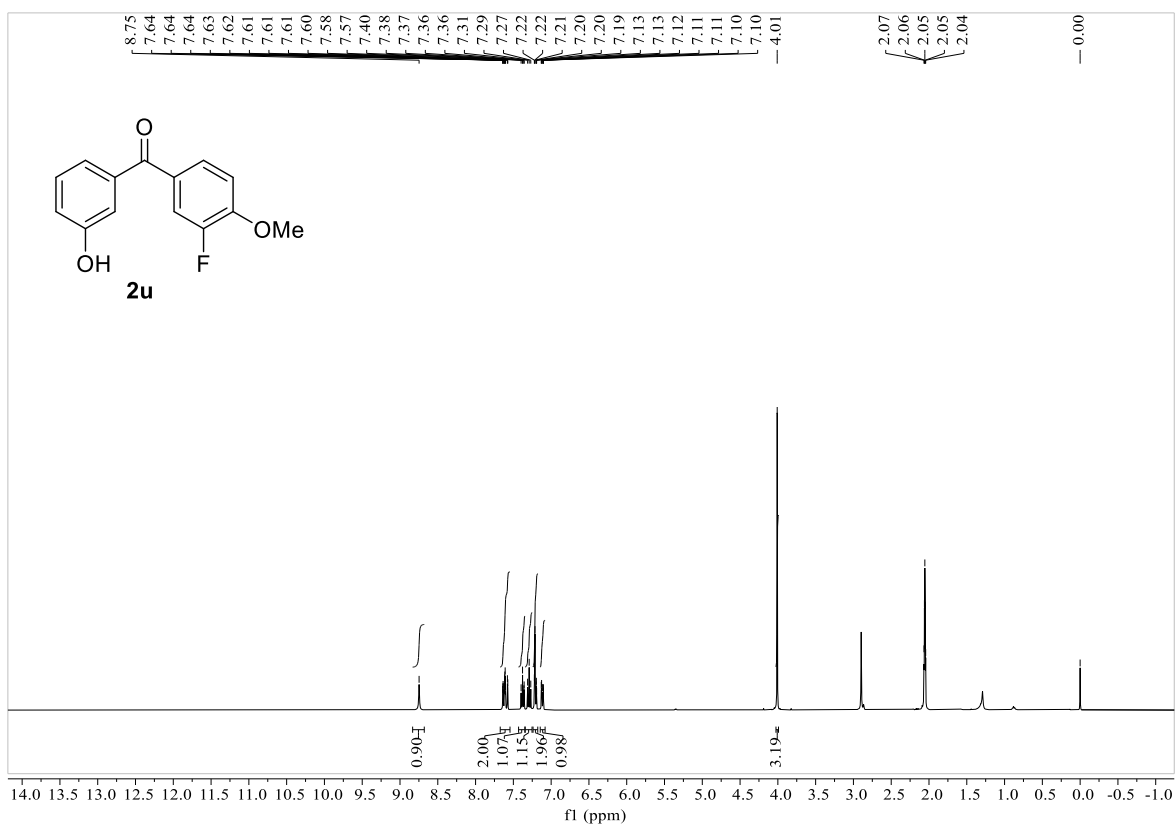
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2s**



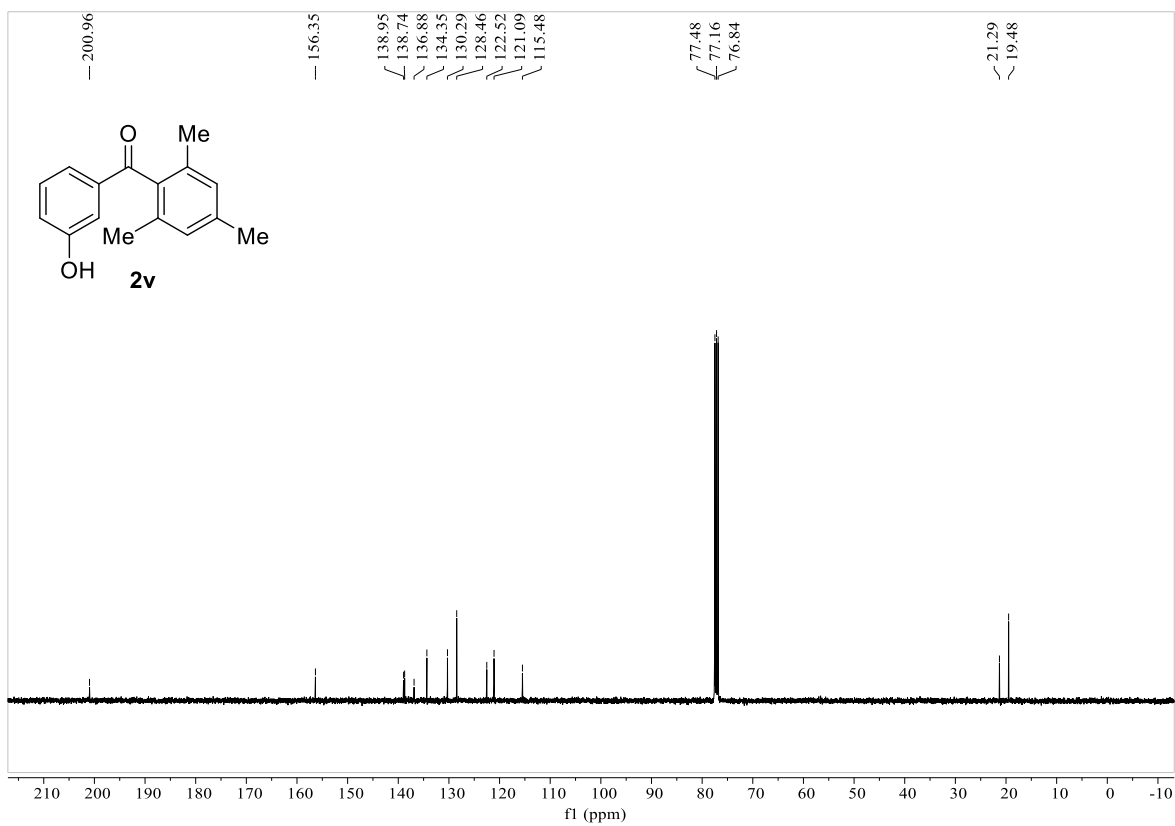
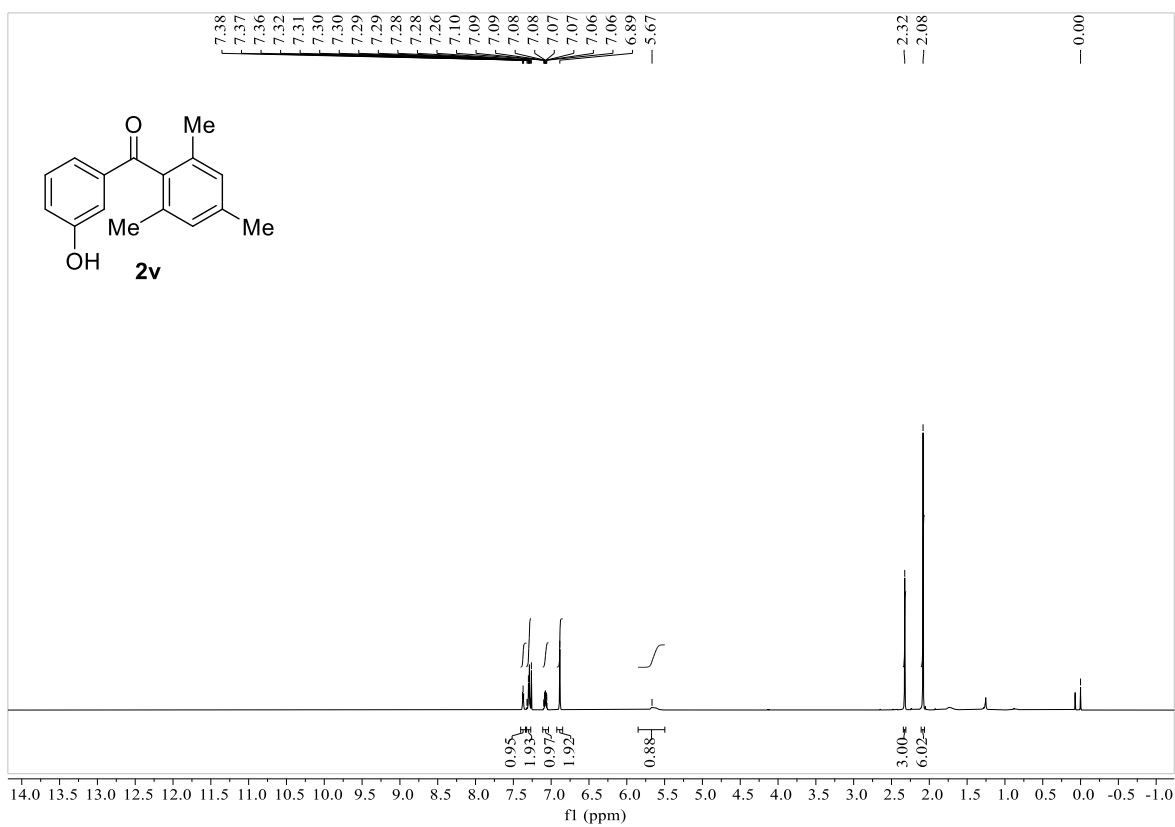
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2t**



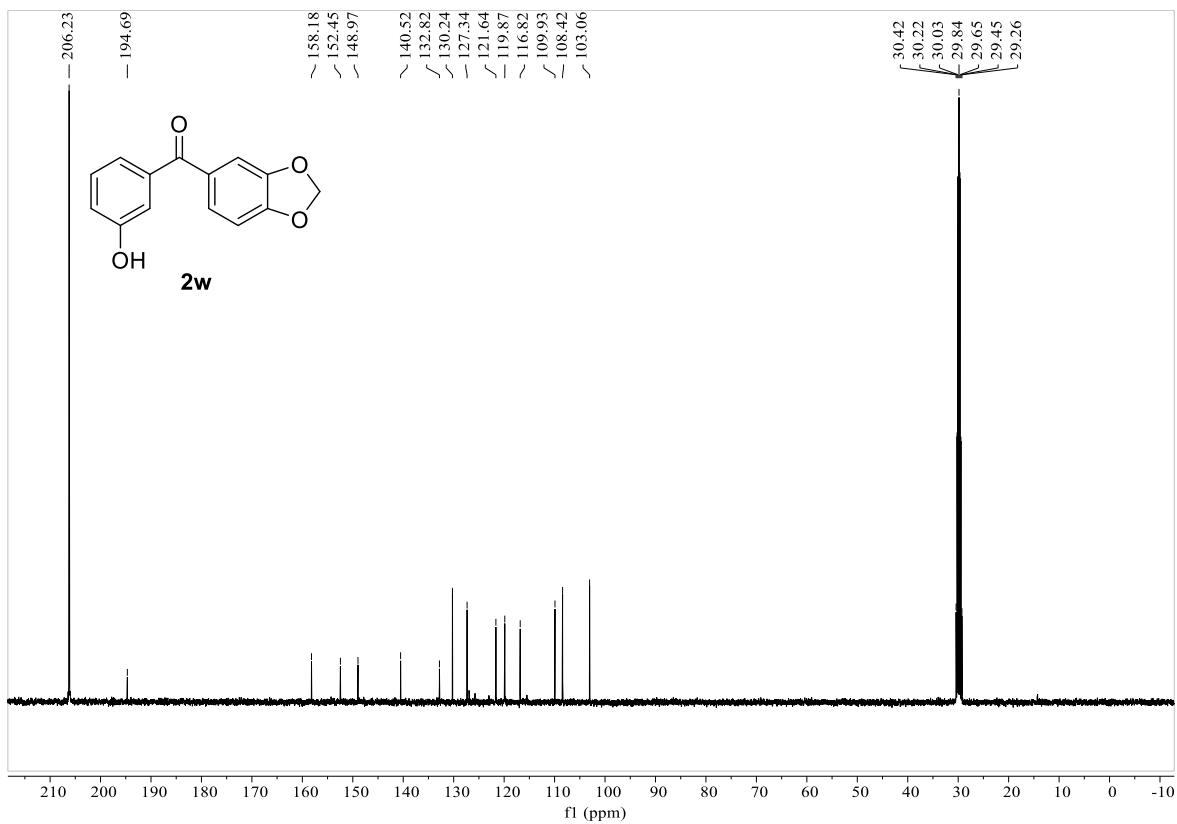
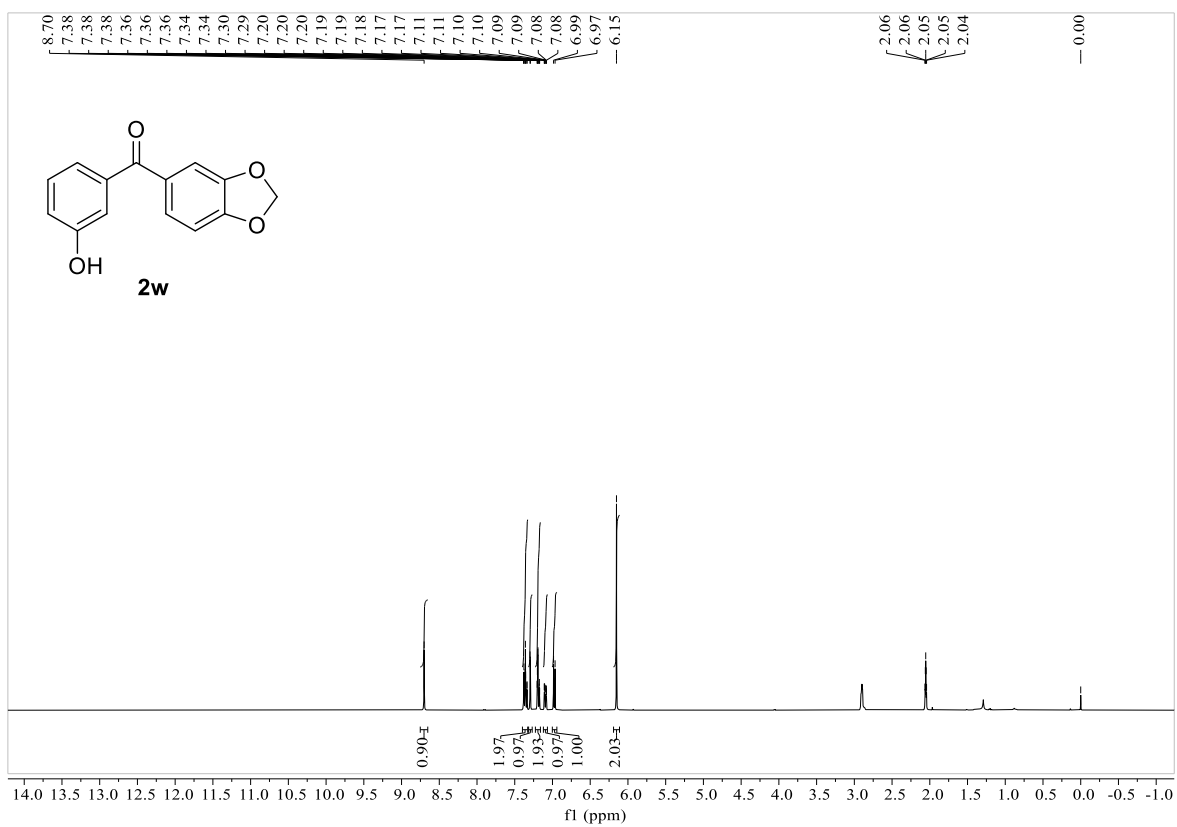
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2u**



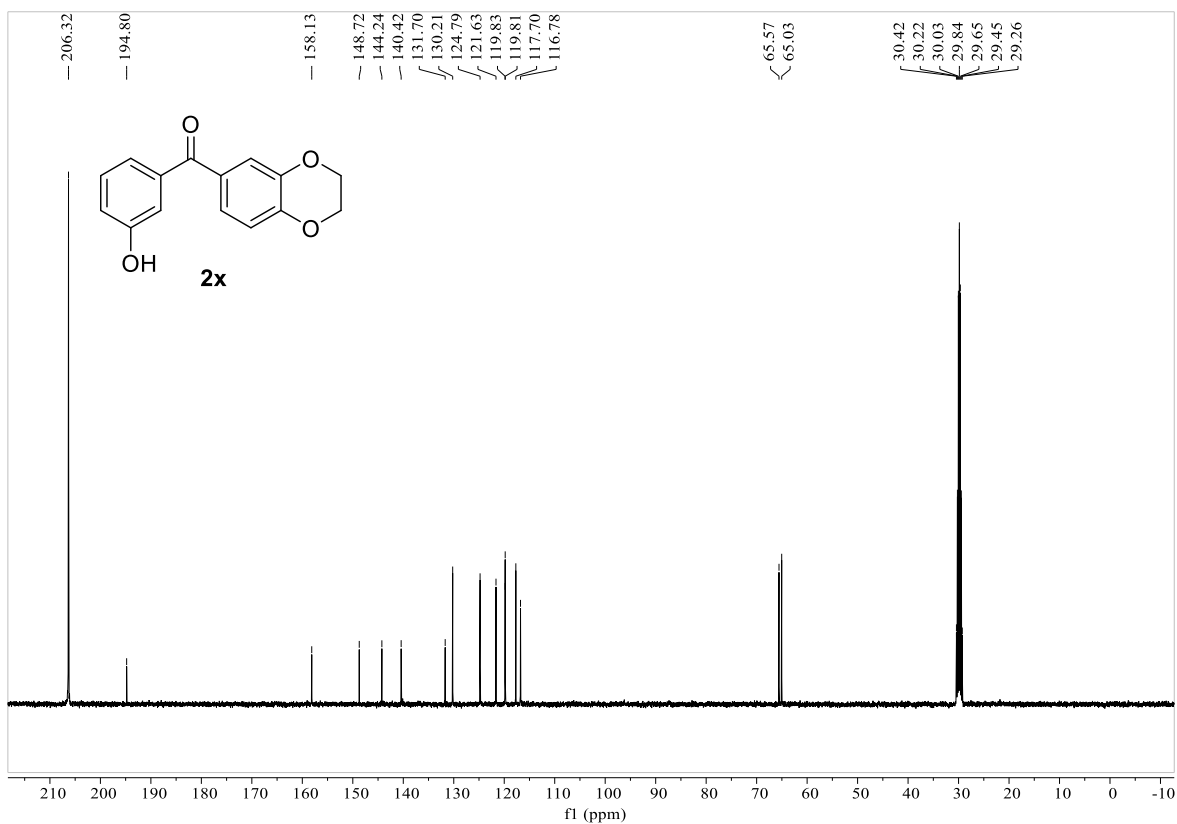
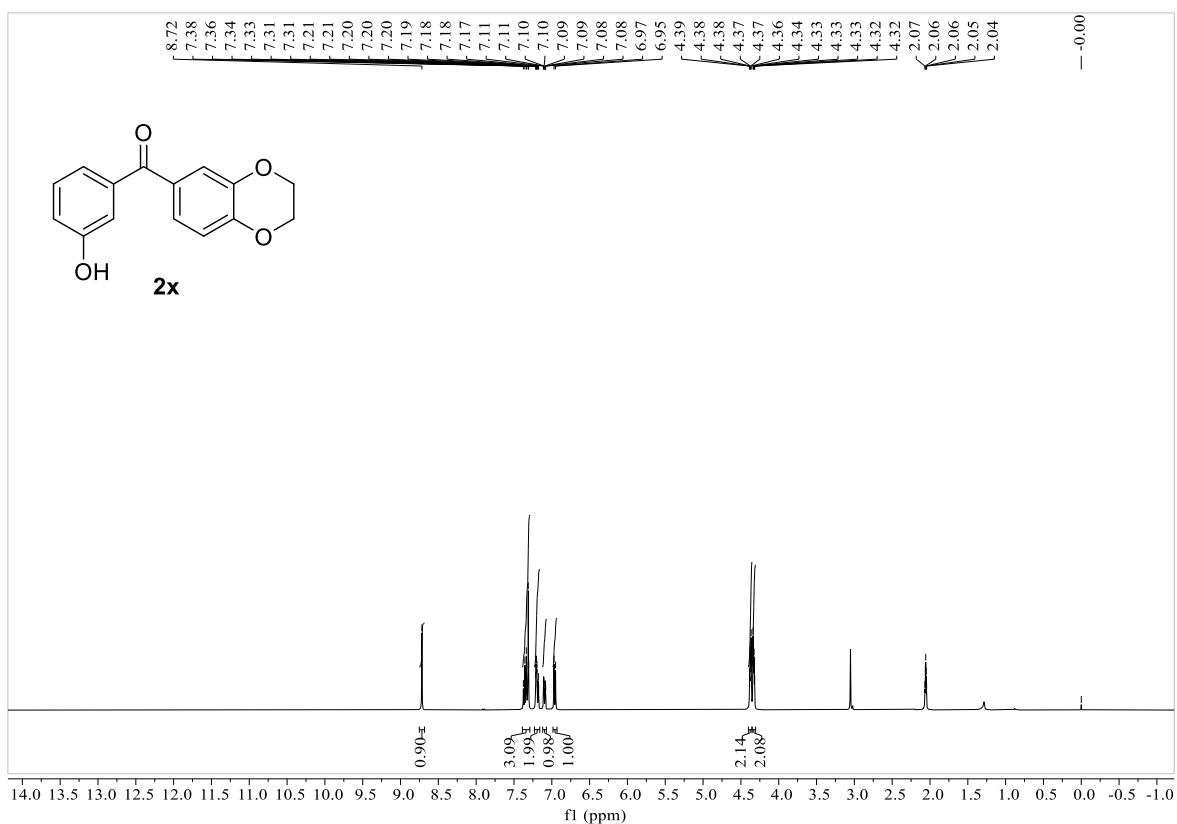
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2v**



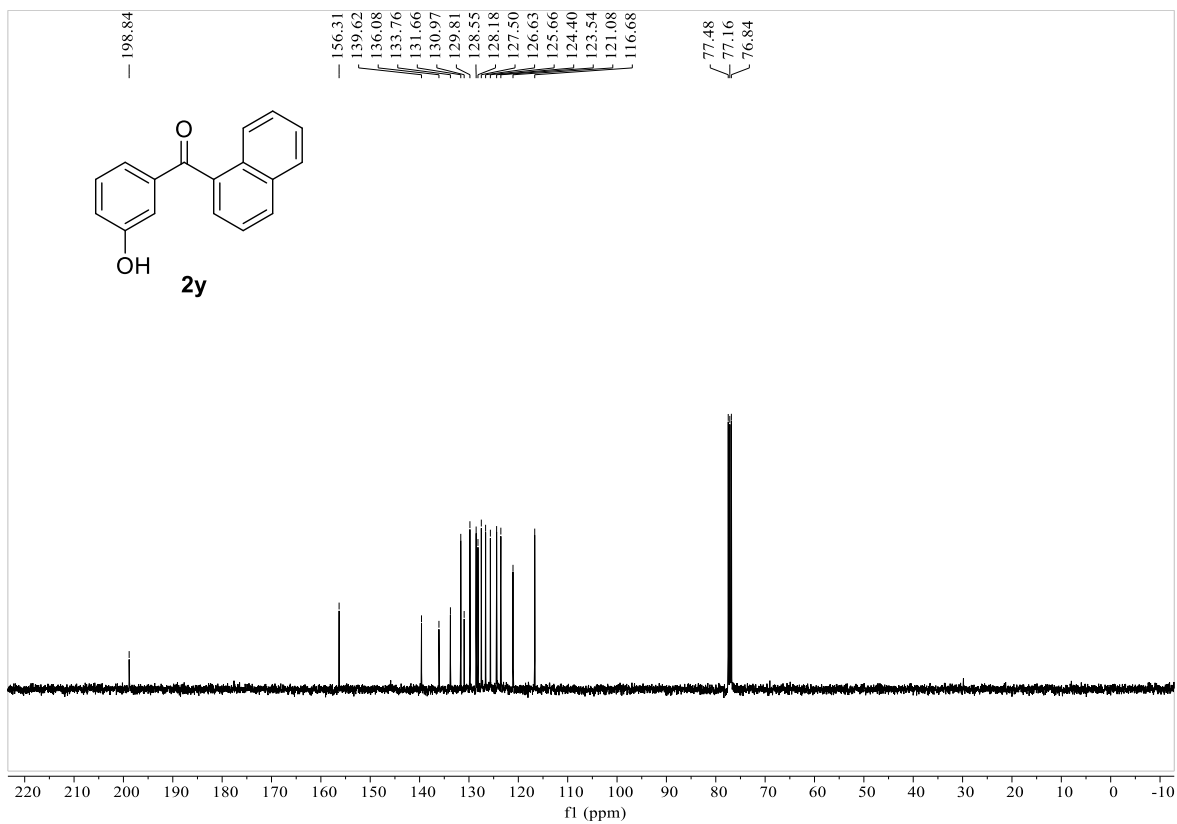
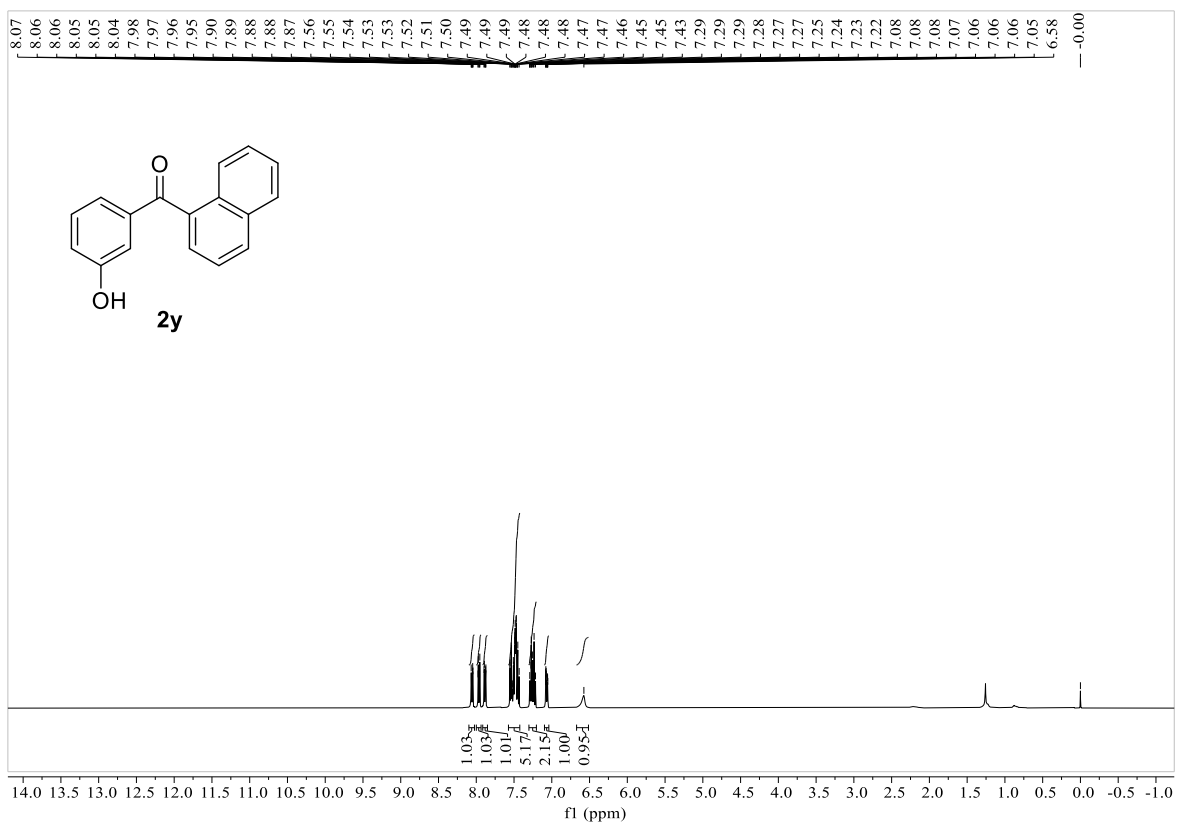
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2w**



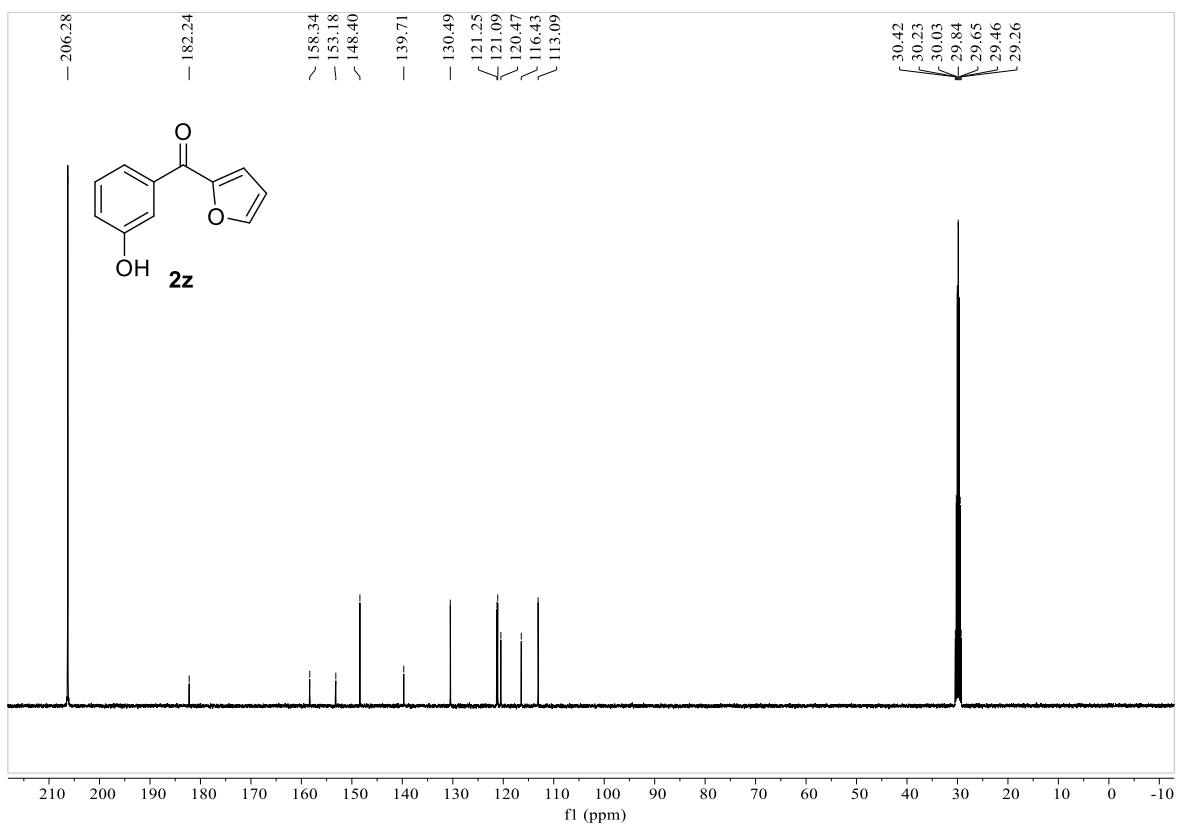
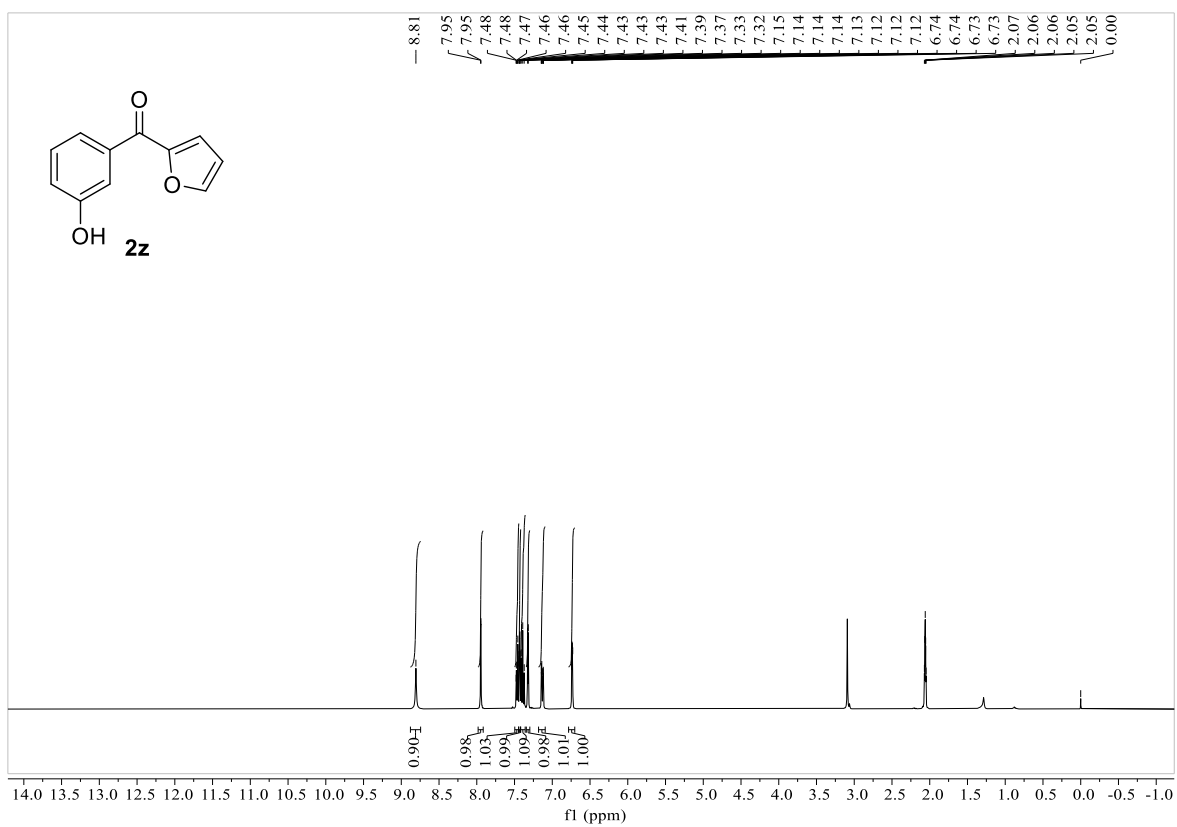
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2x**



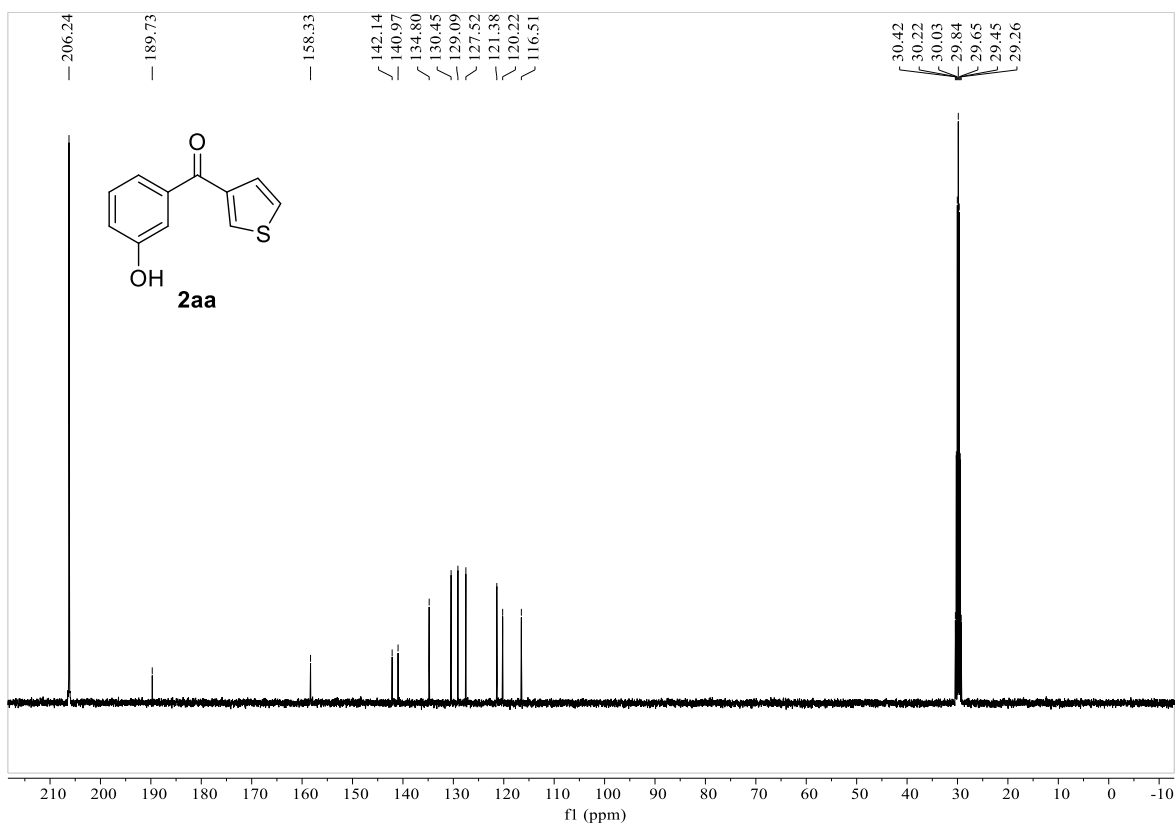
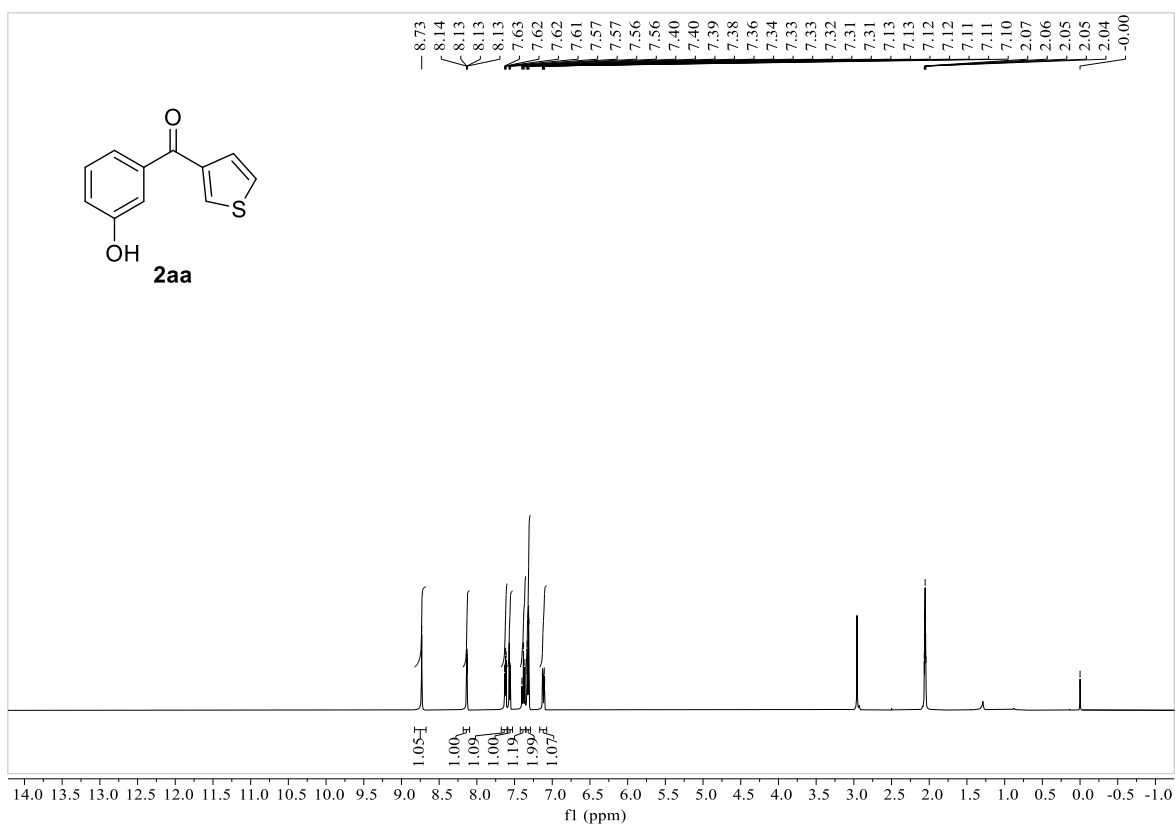
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **2y**



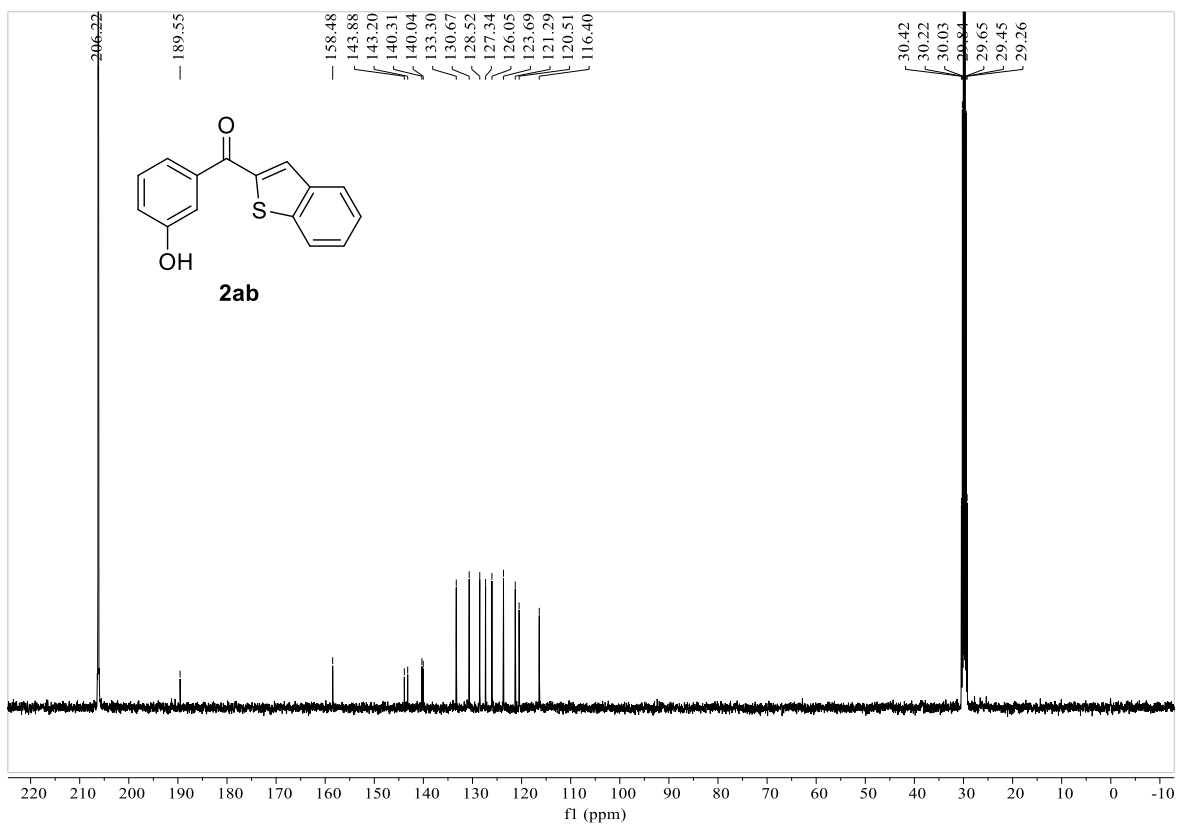
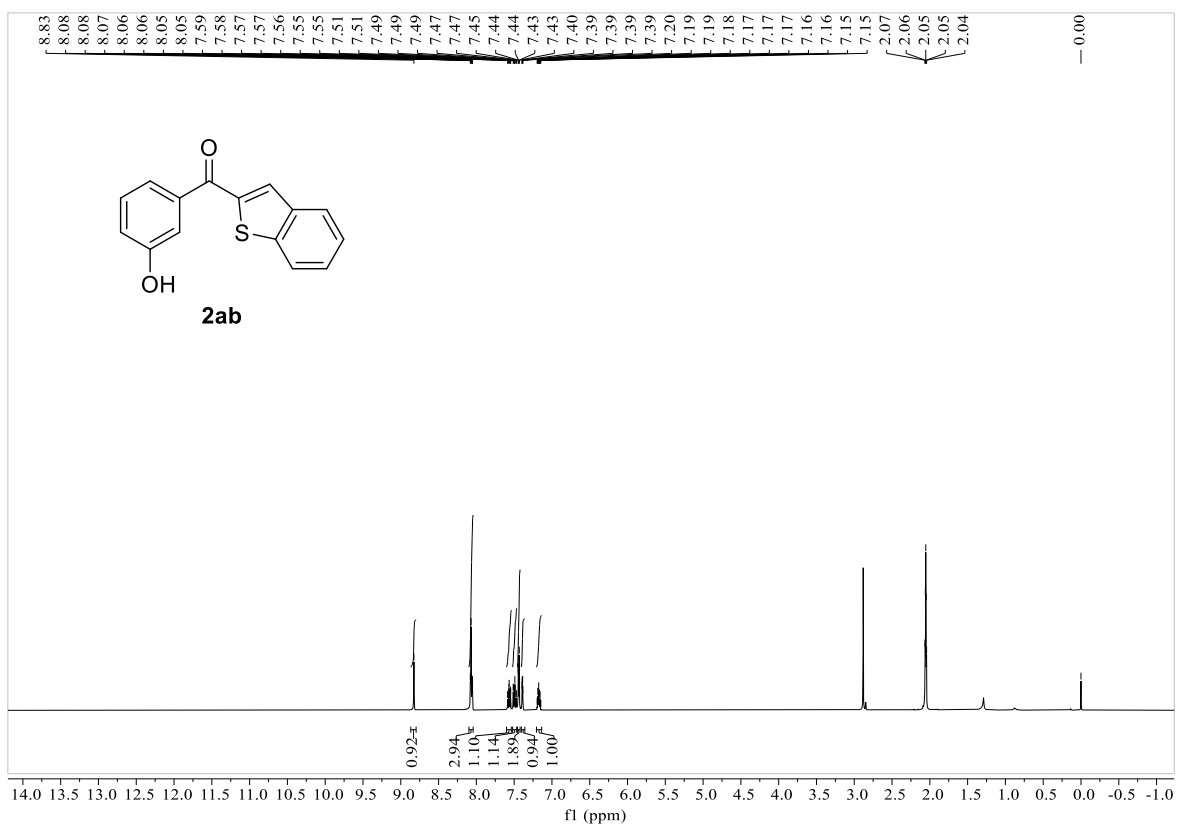
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2z**



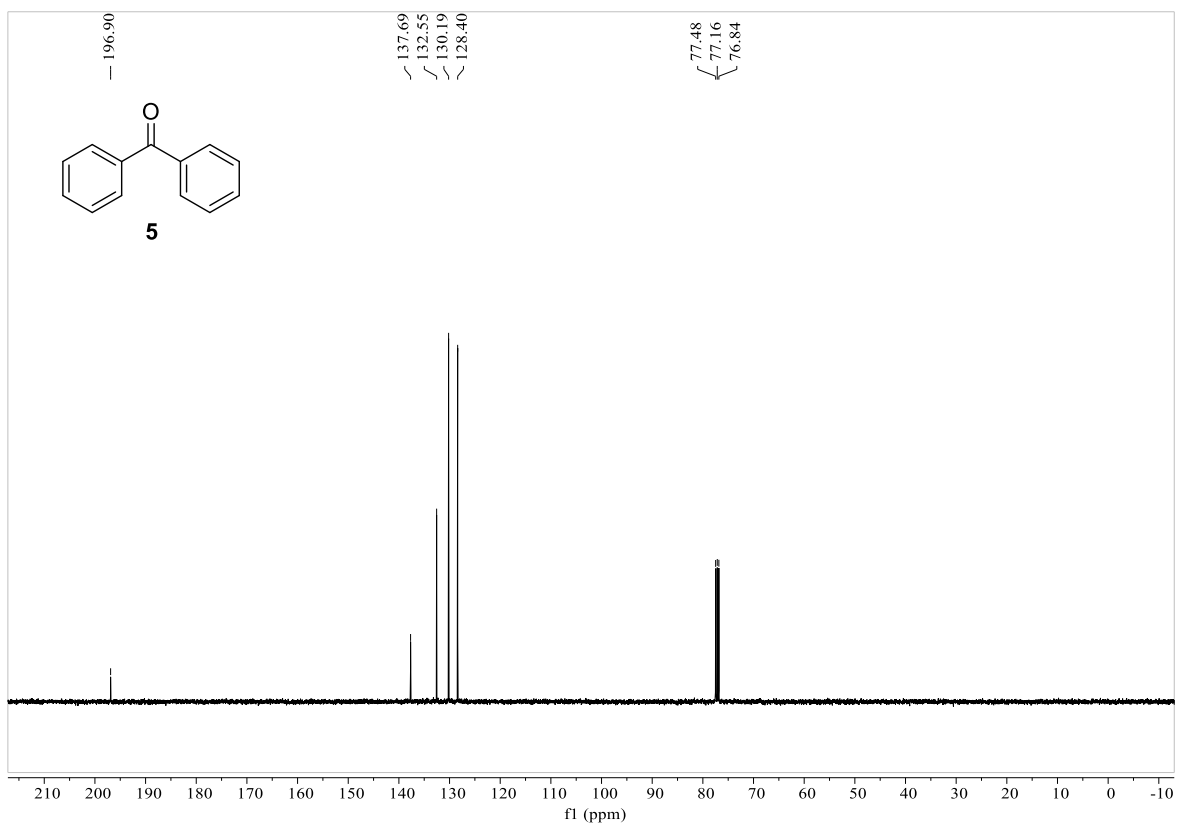
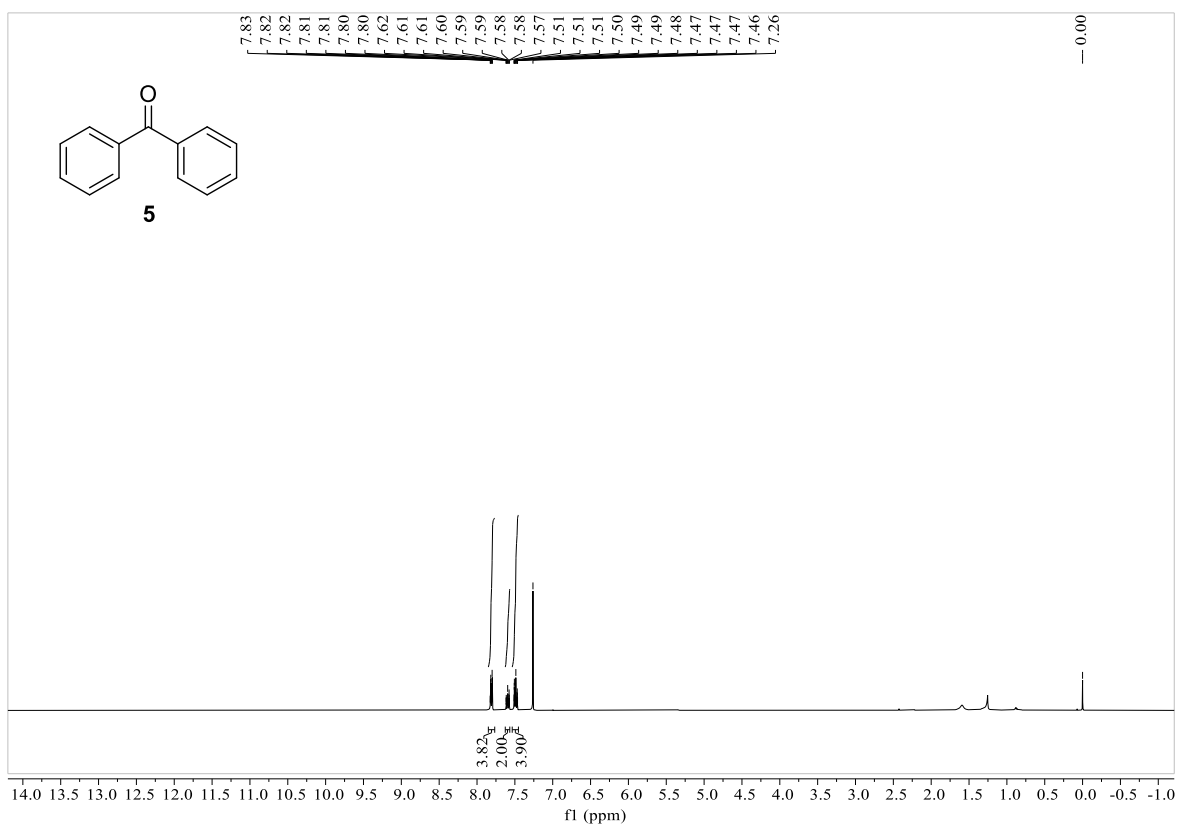
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2aa**



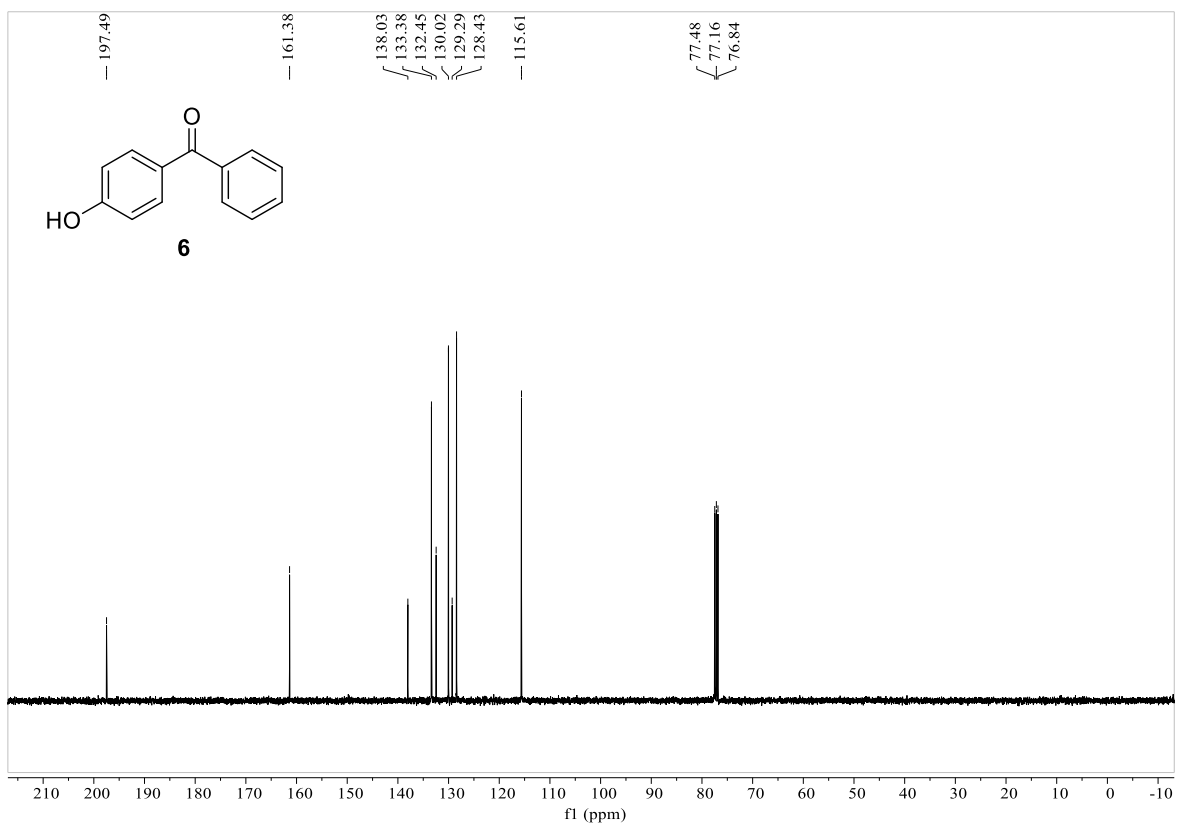
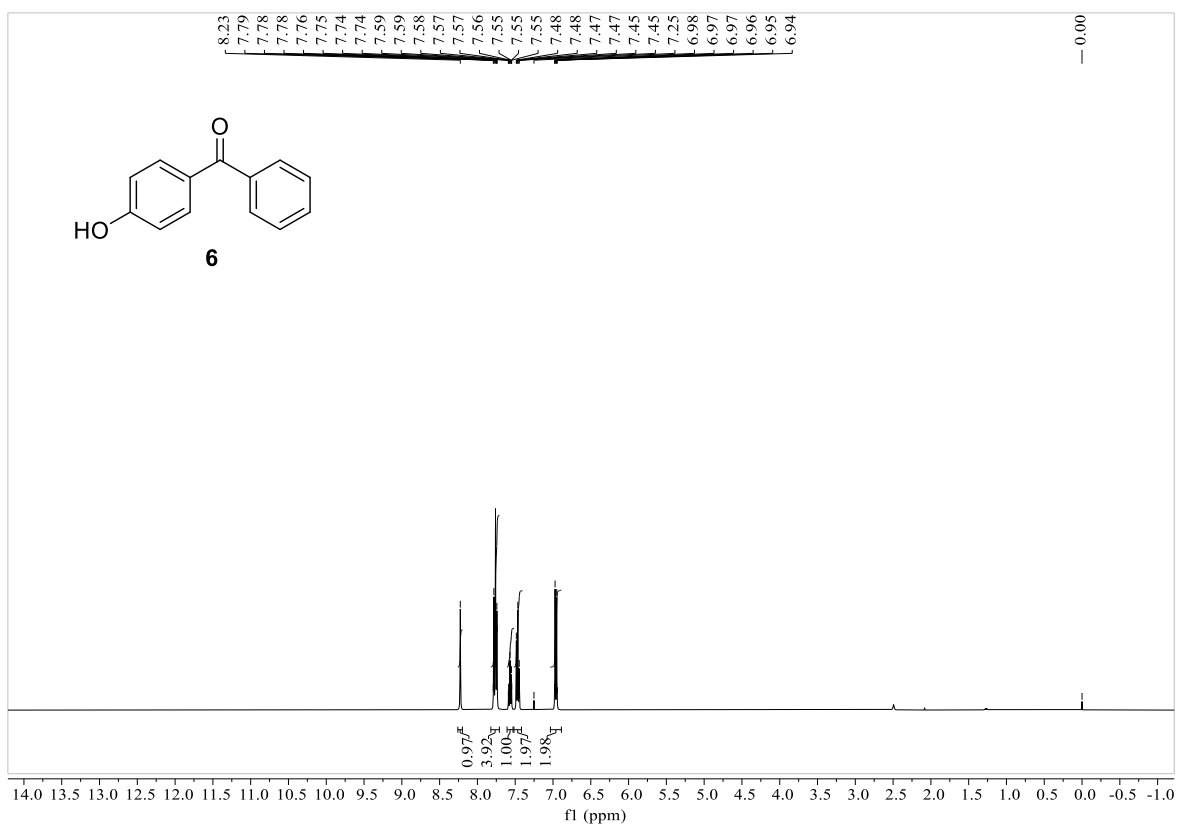
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **2ab**



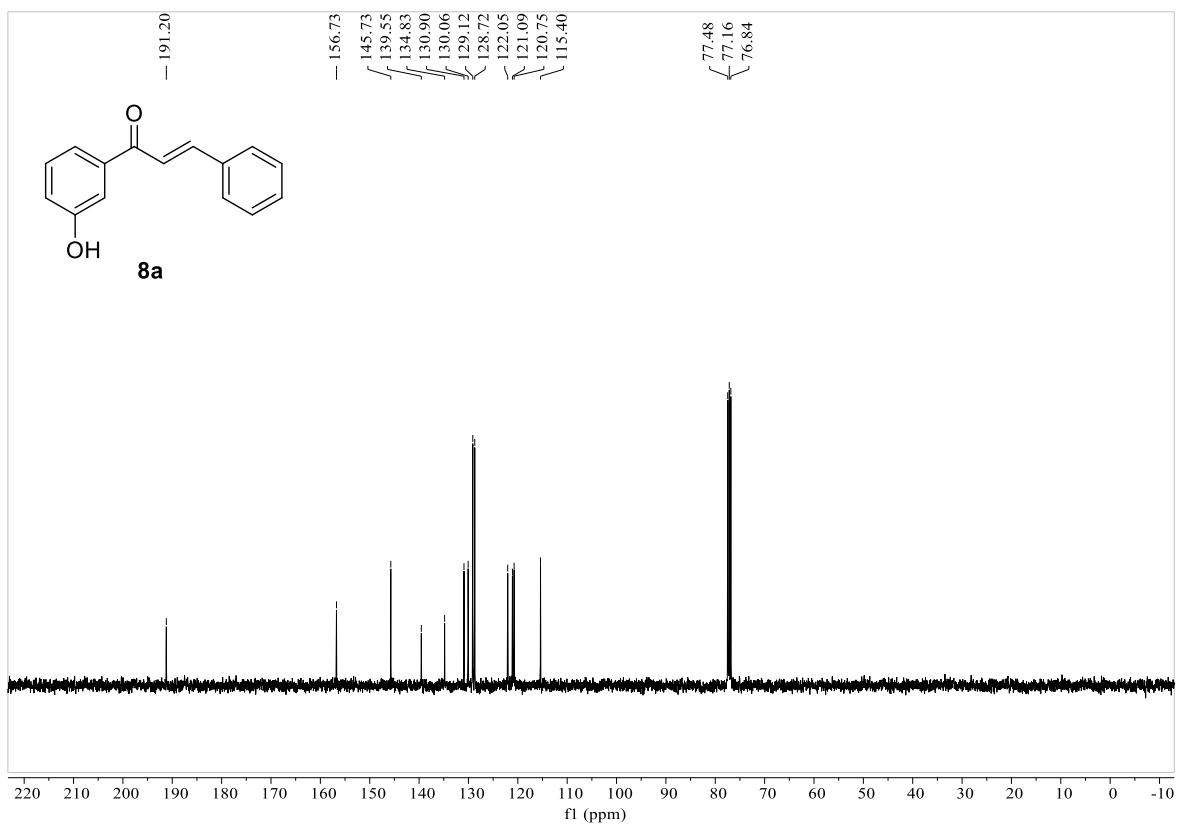
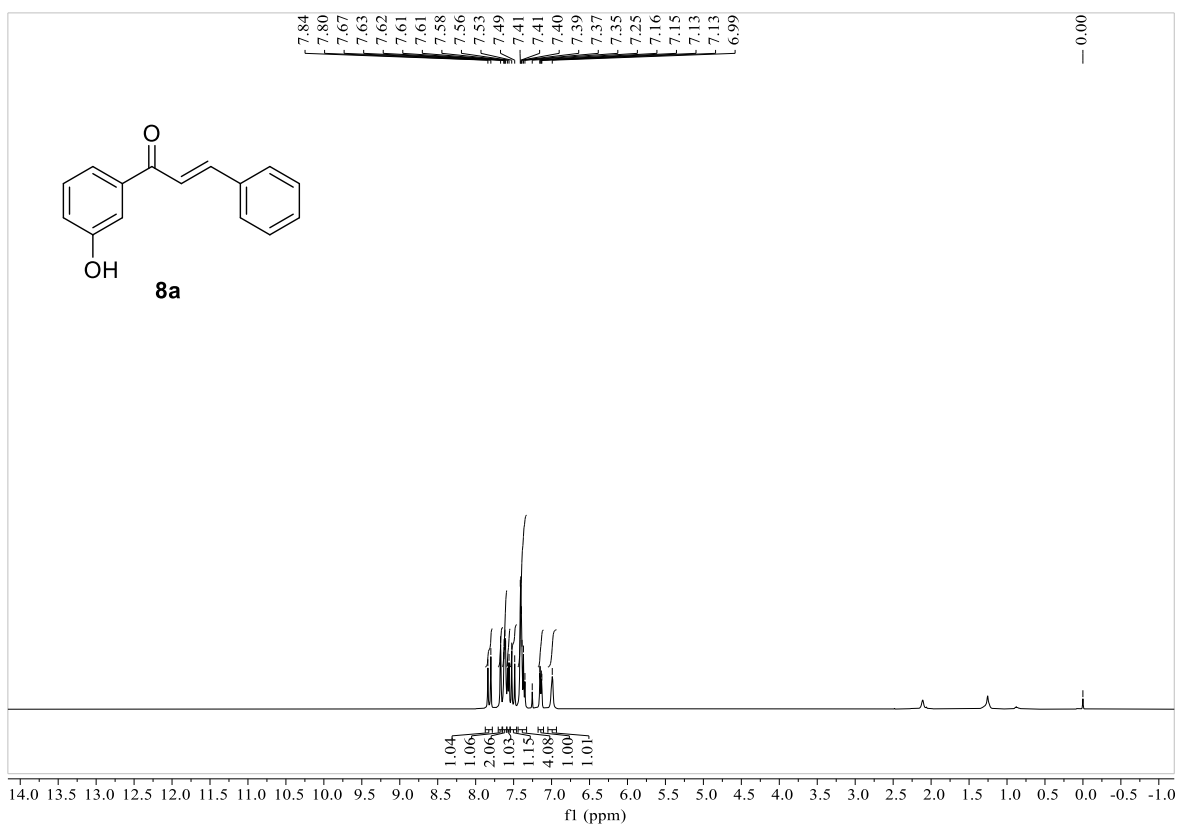
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **5**



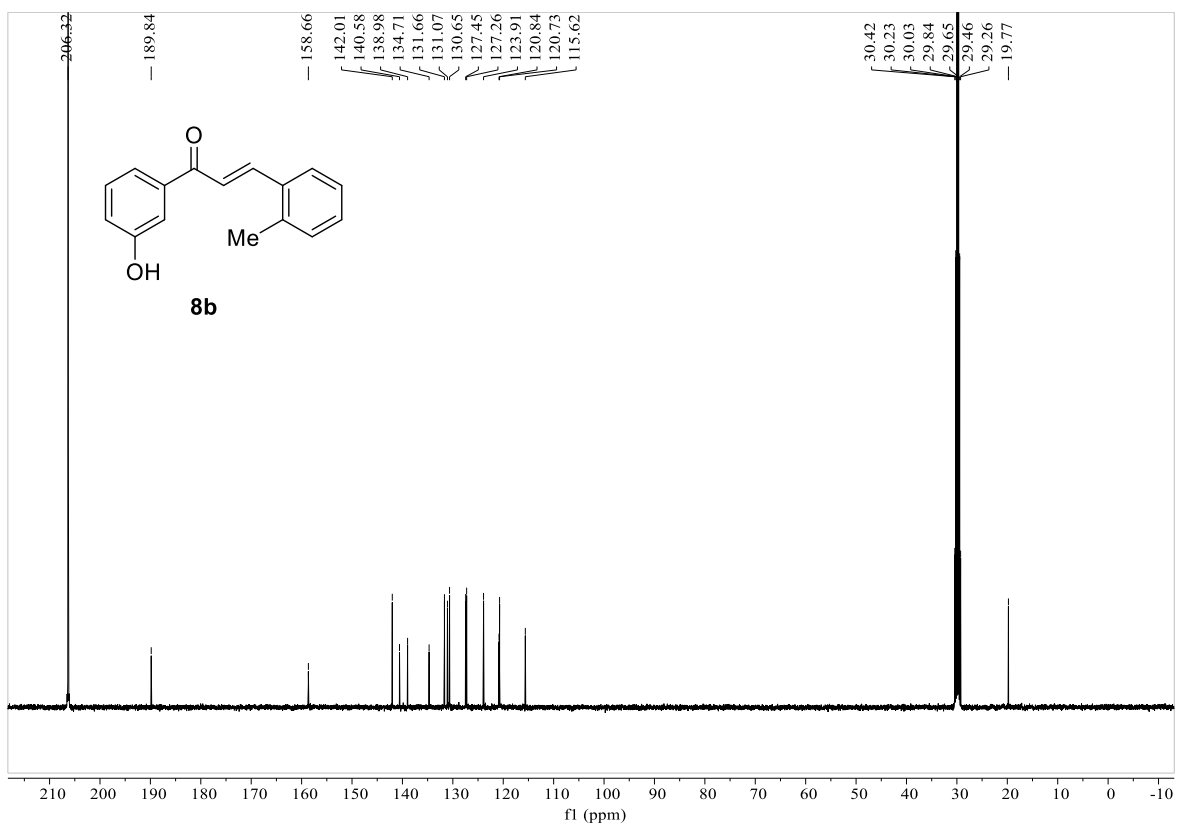
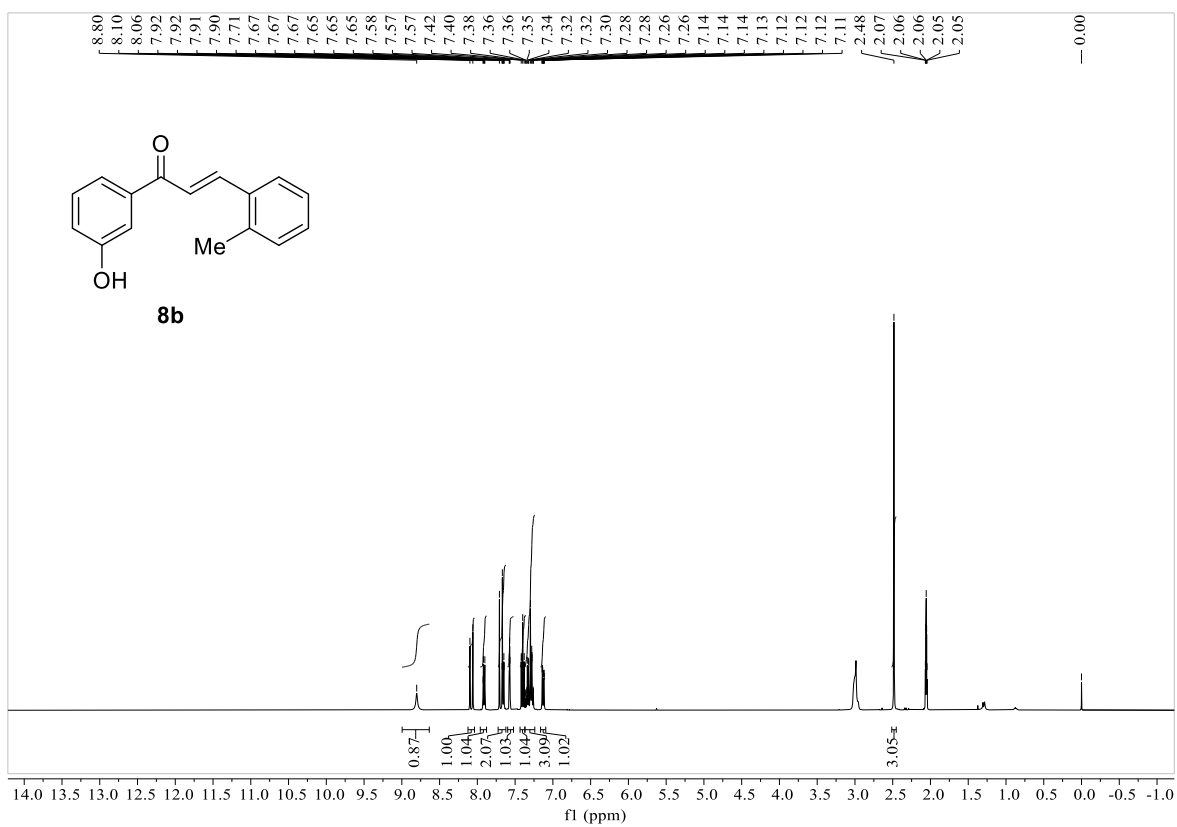
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **6**



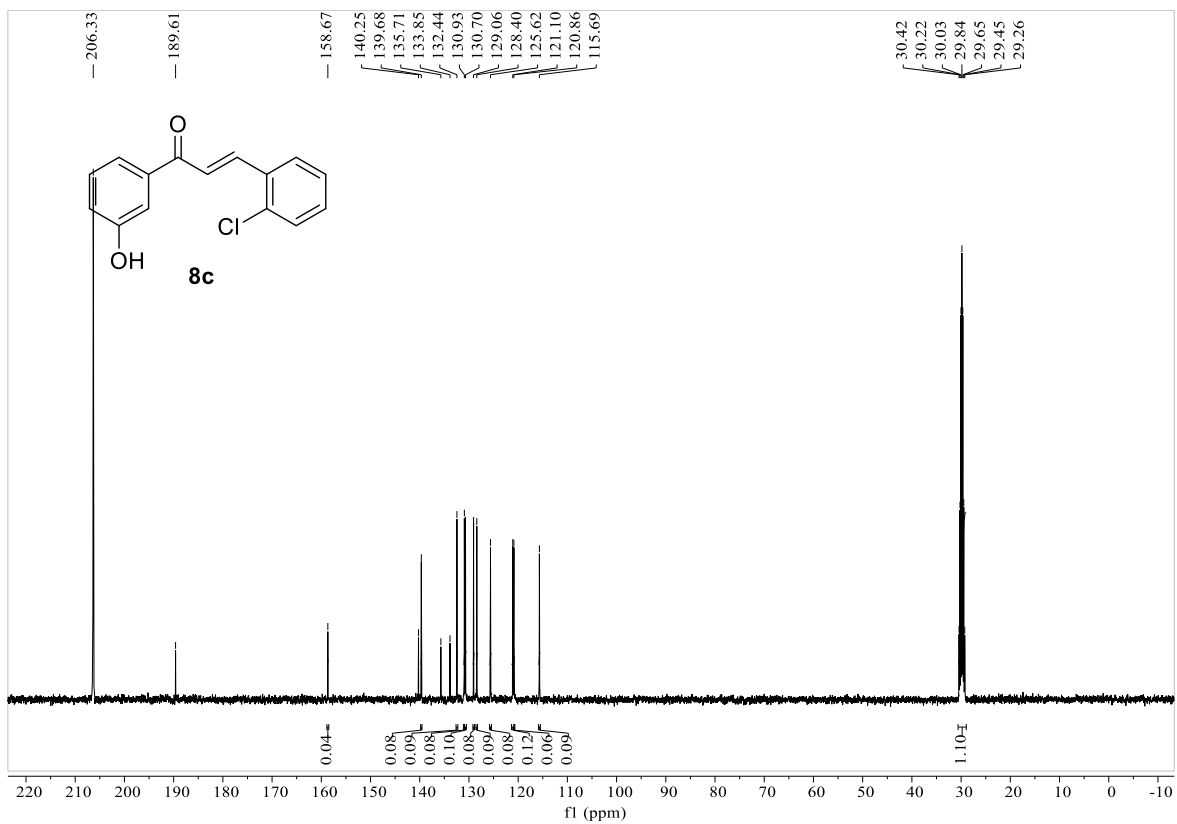
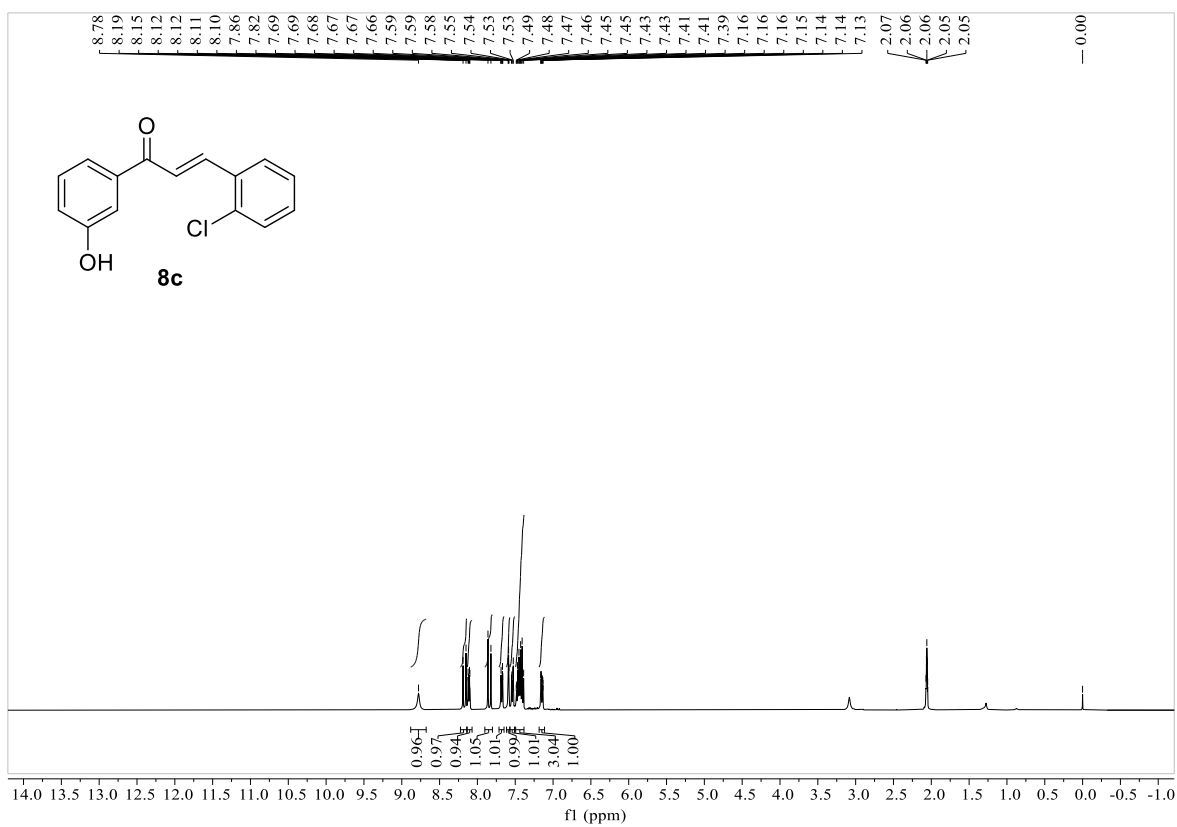
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **8a**



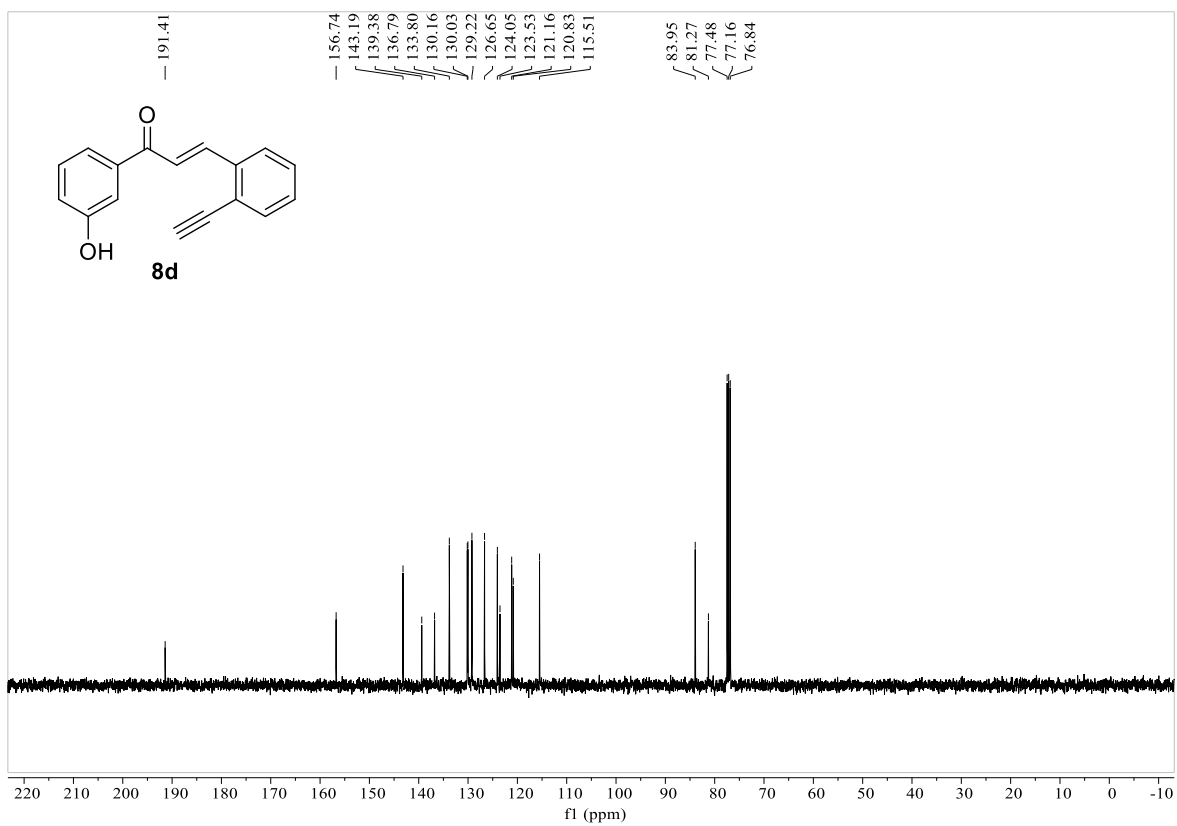
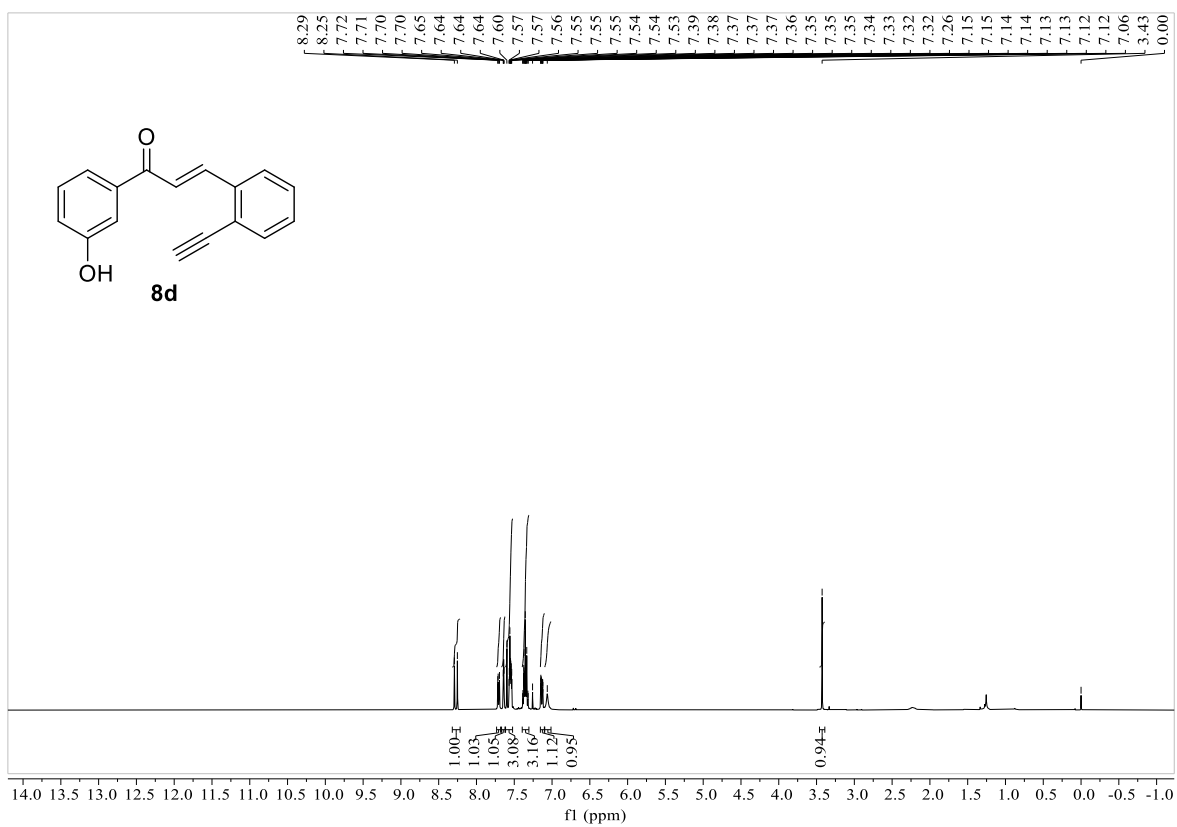
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8b**



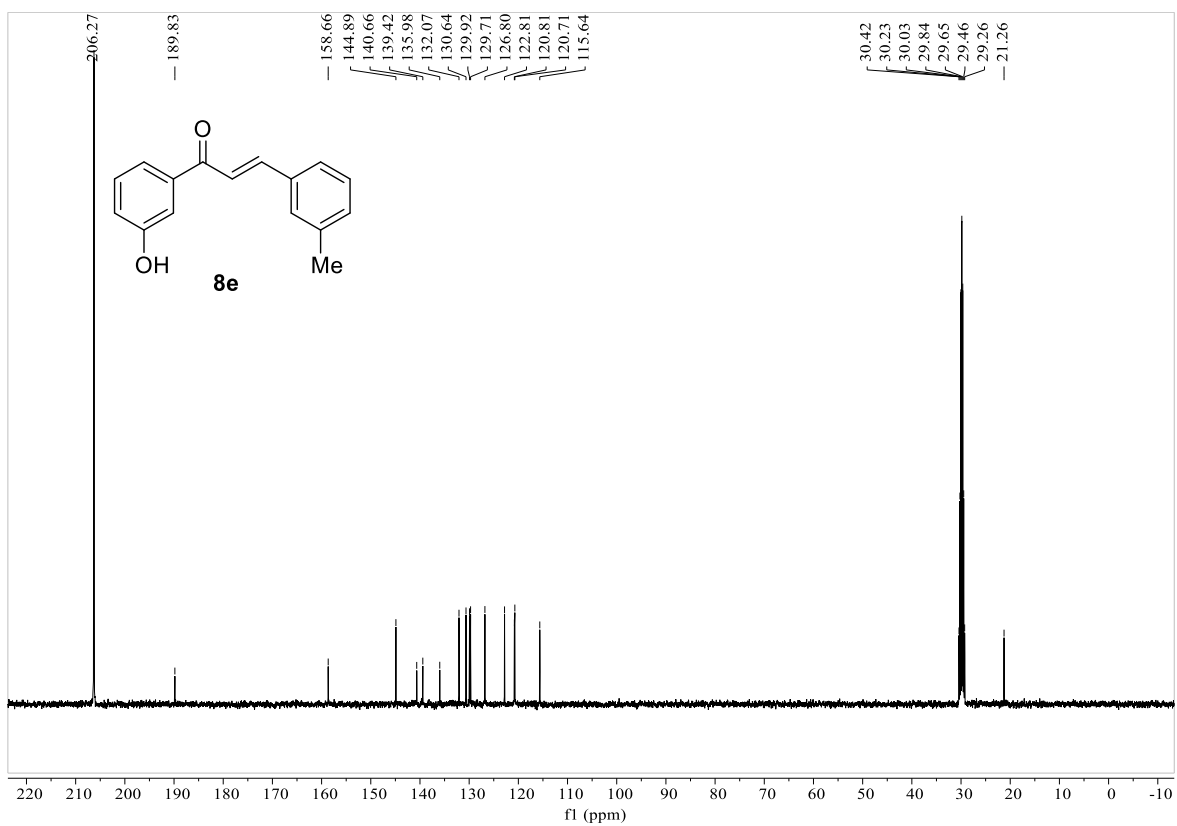
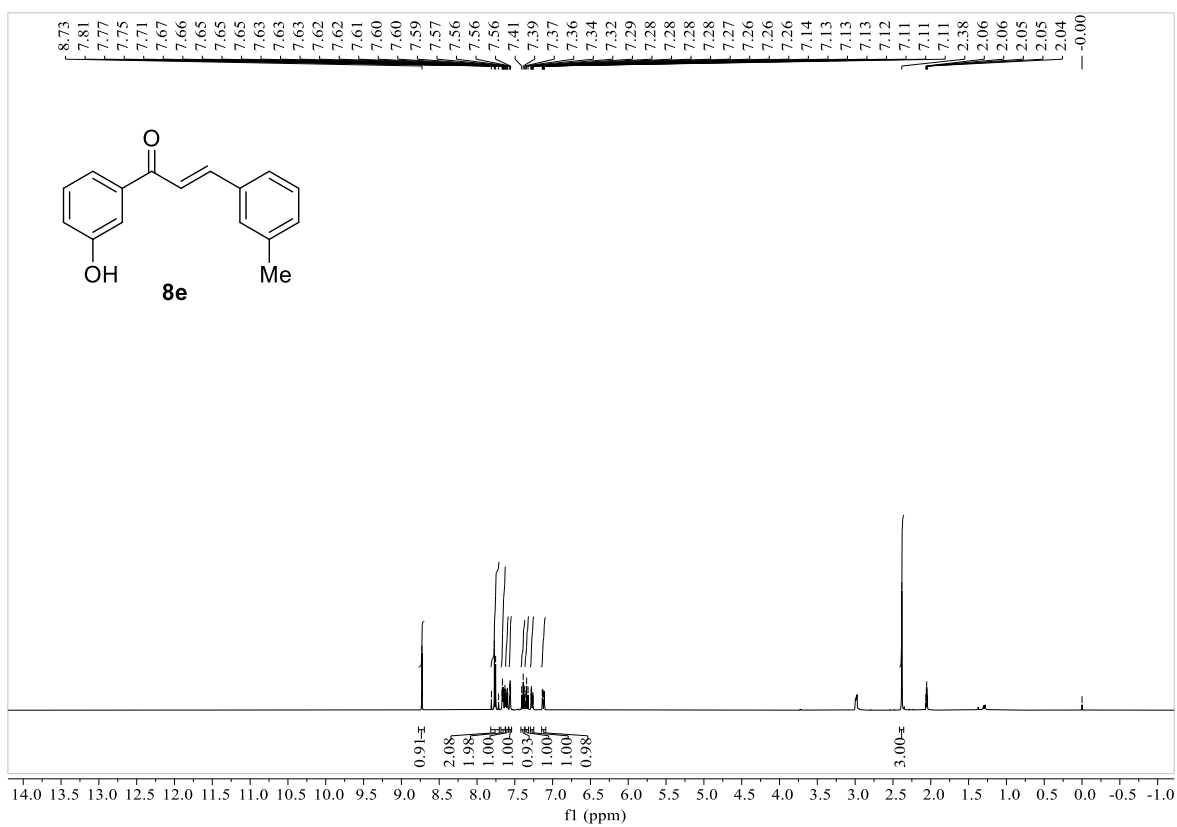
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8c**



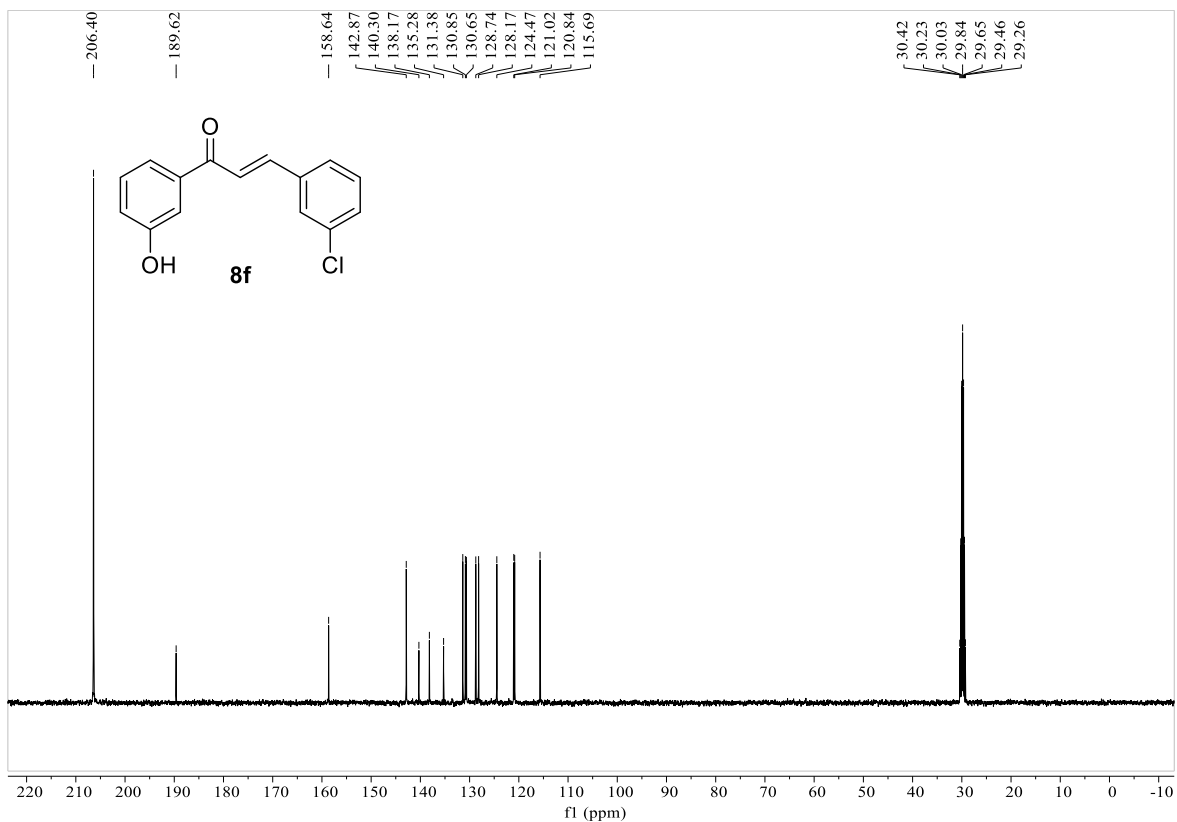
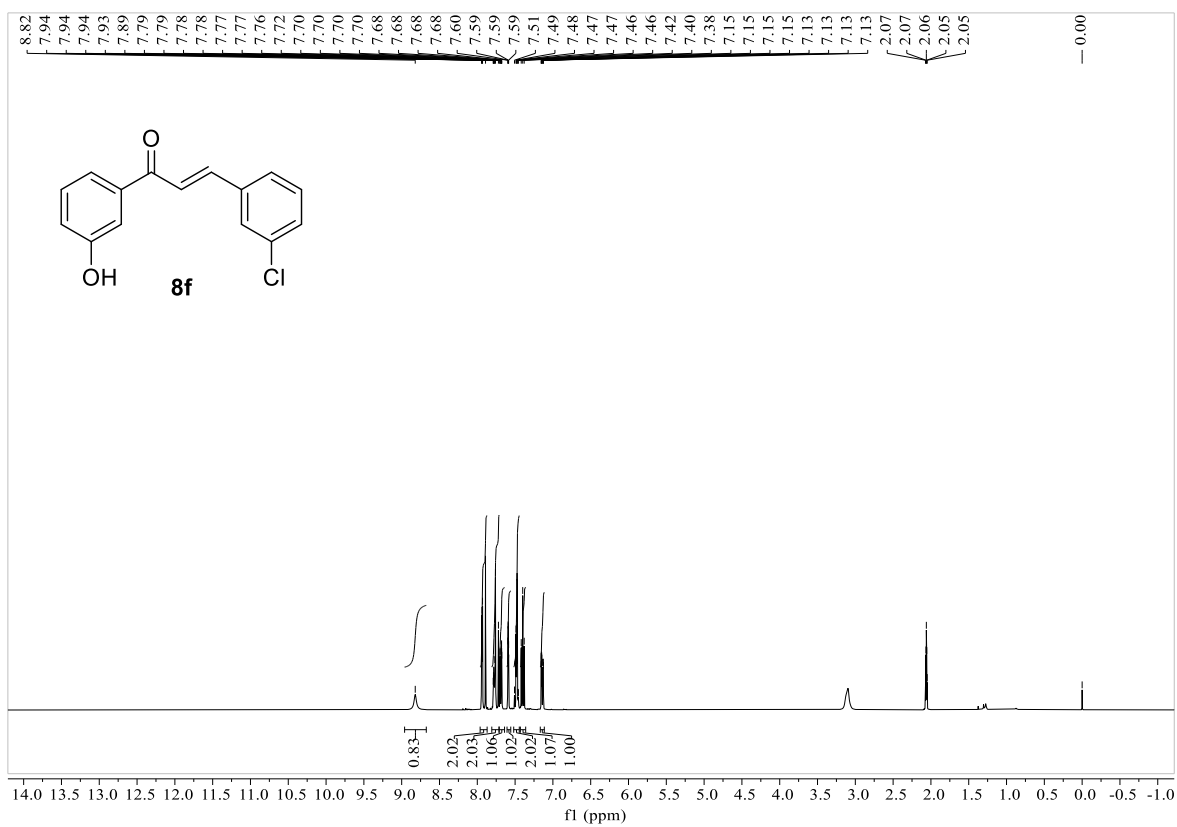
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **8d**



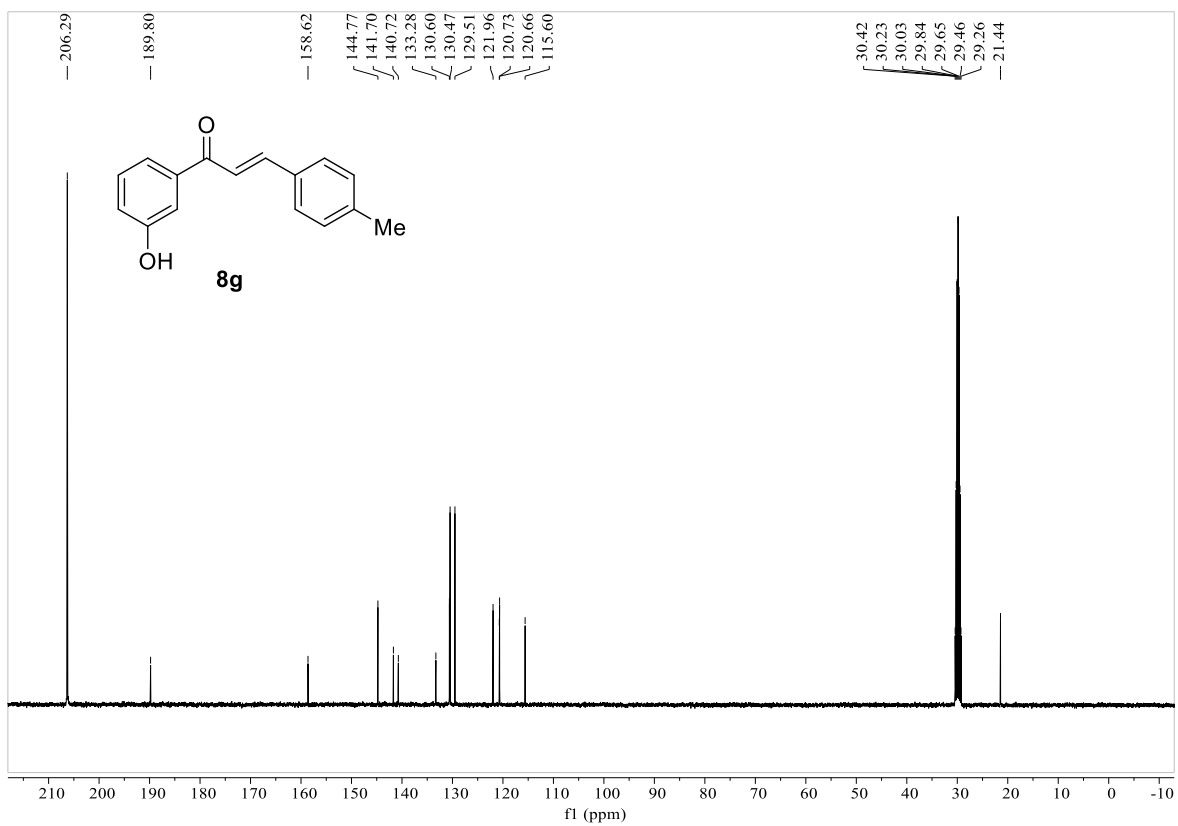
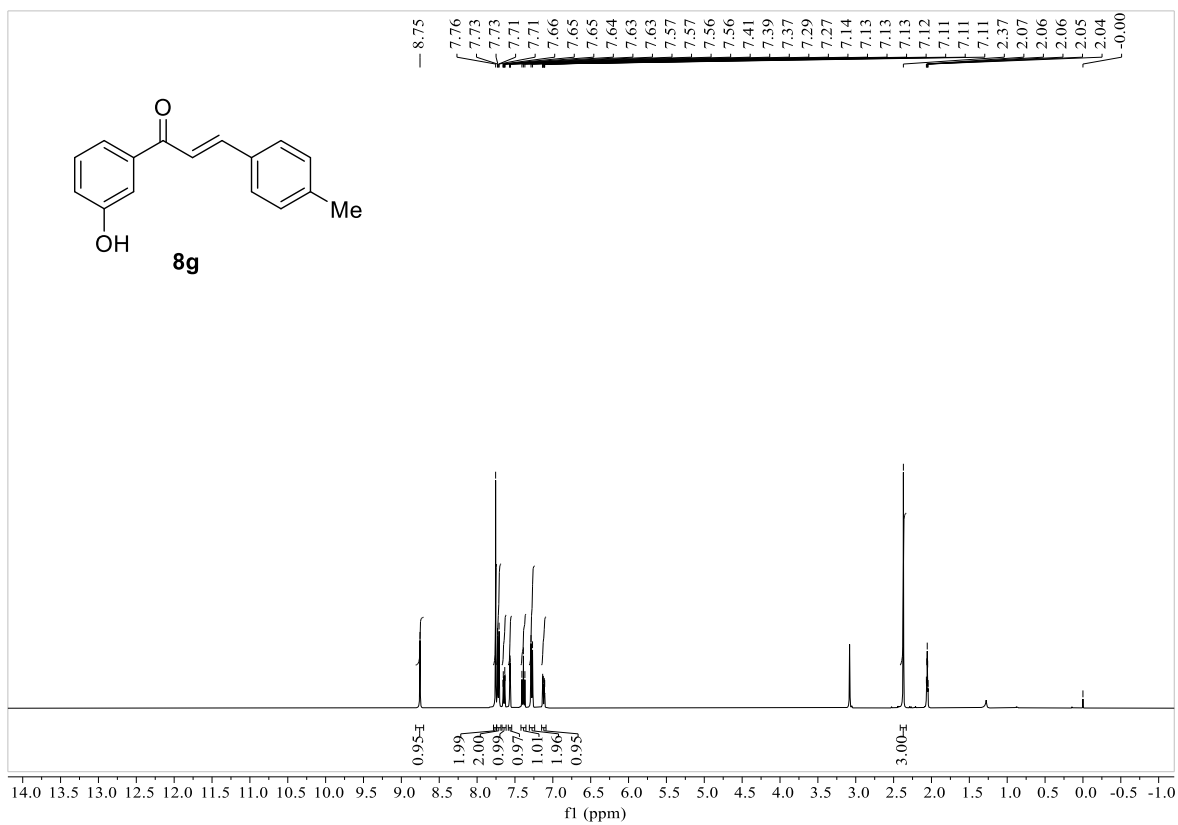
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8e**



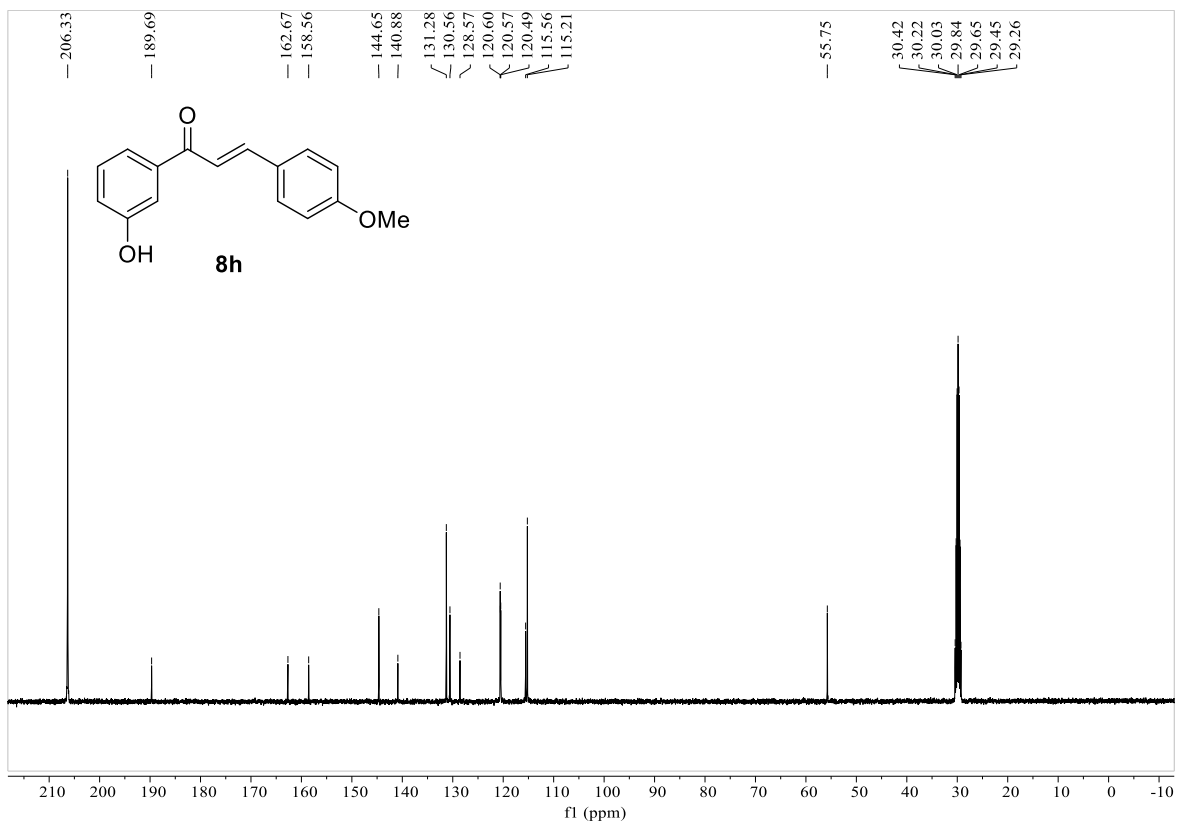
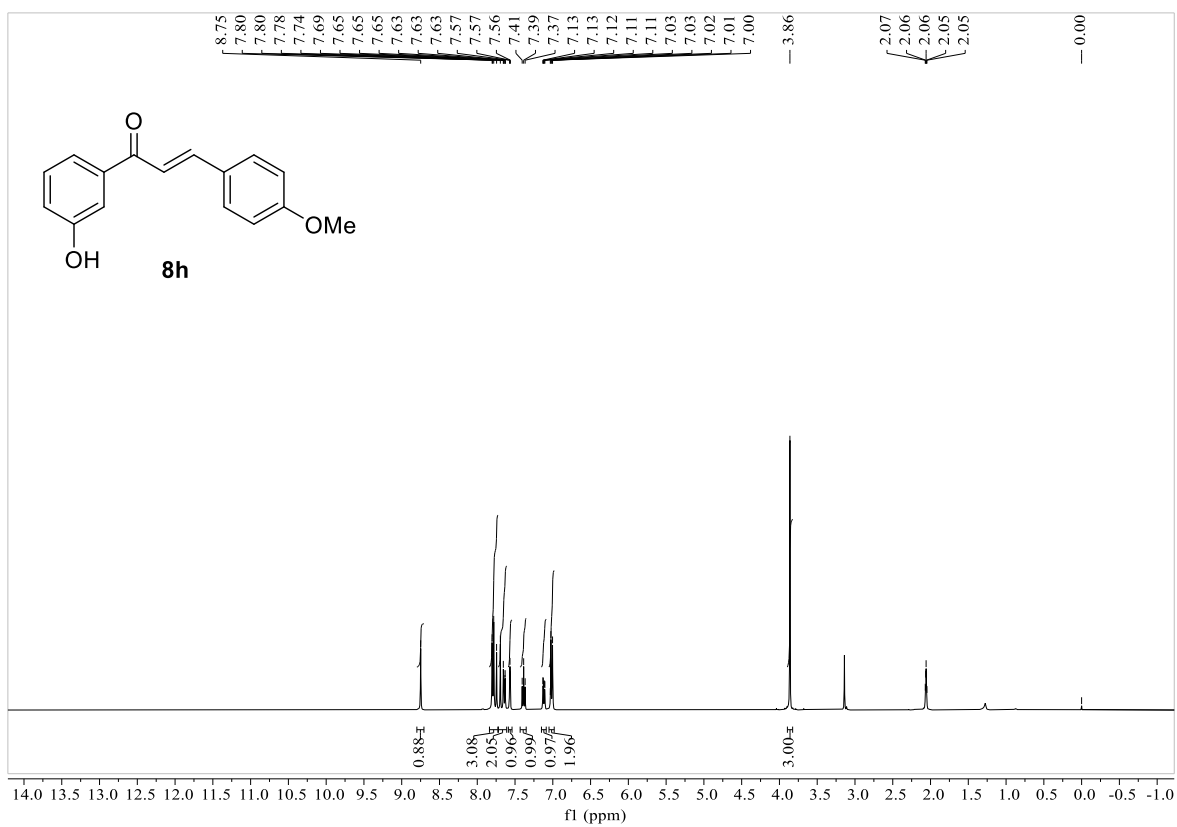
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8f**



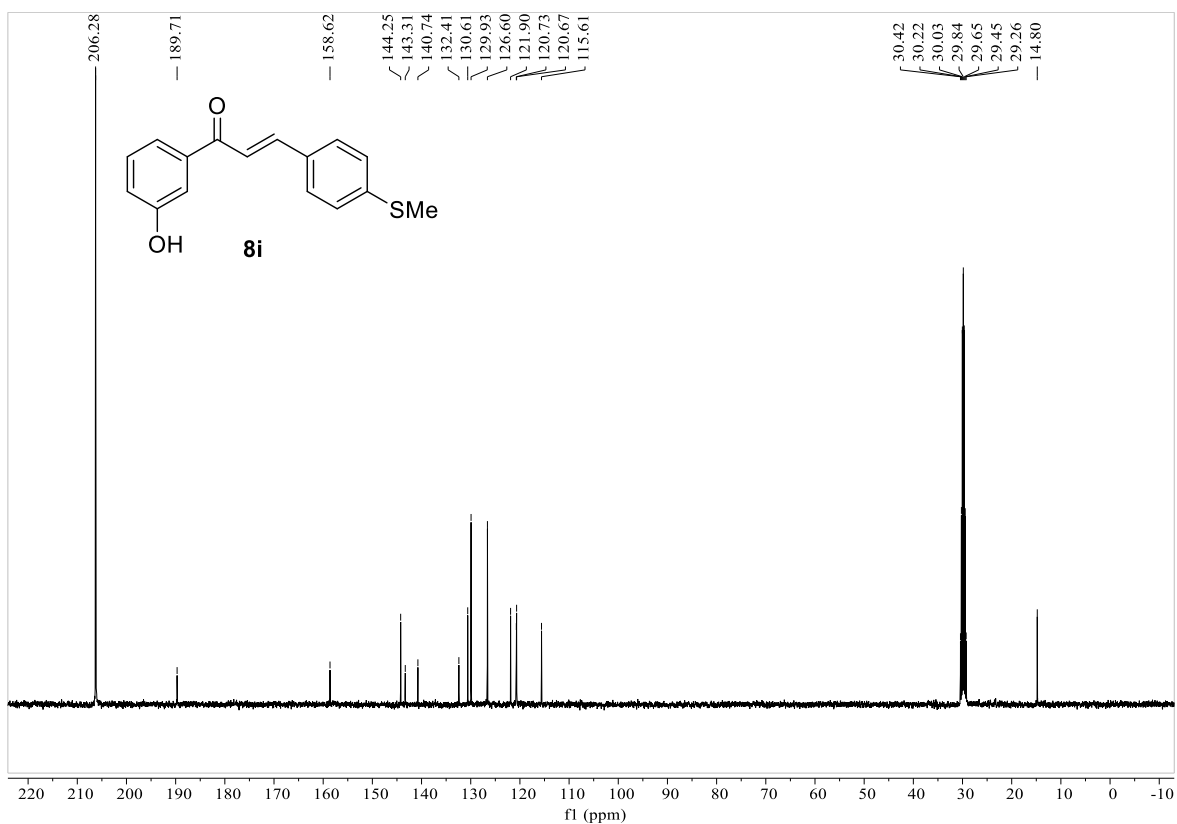
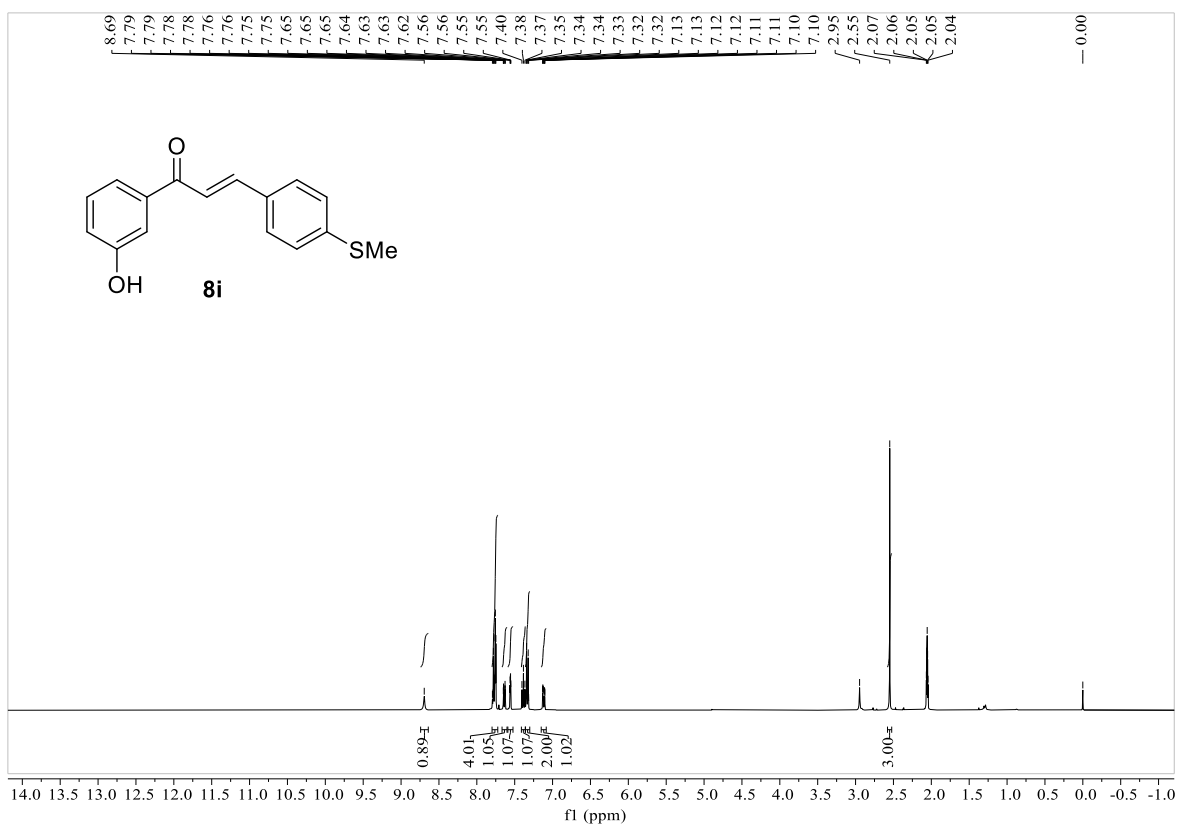
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8g**



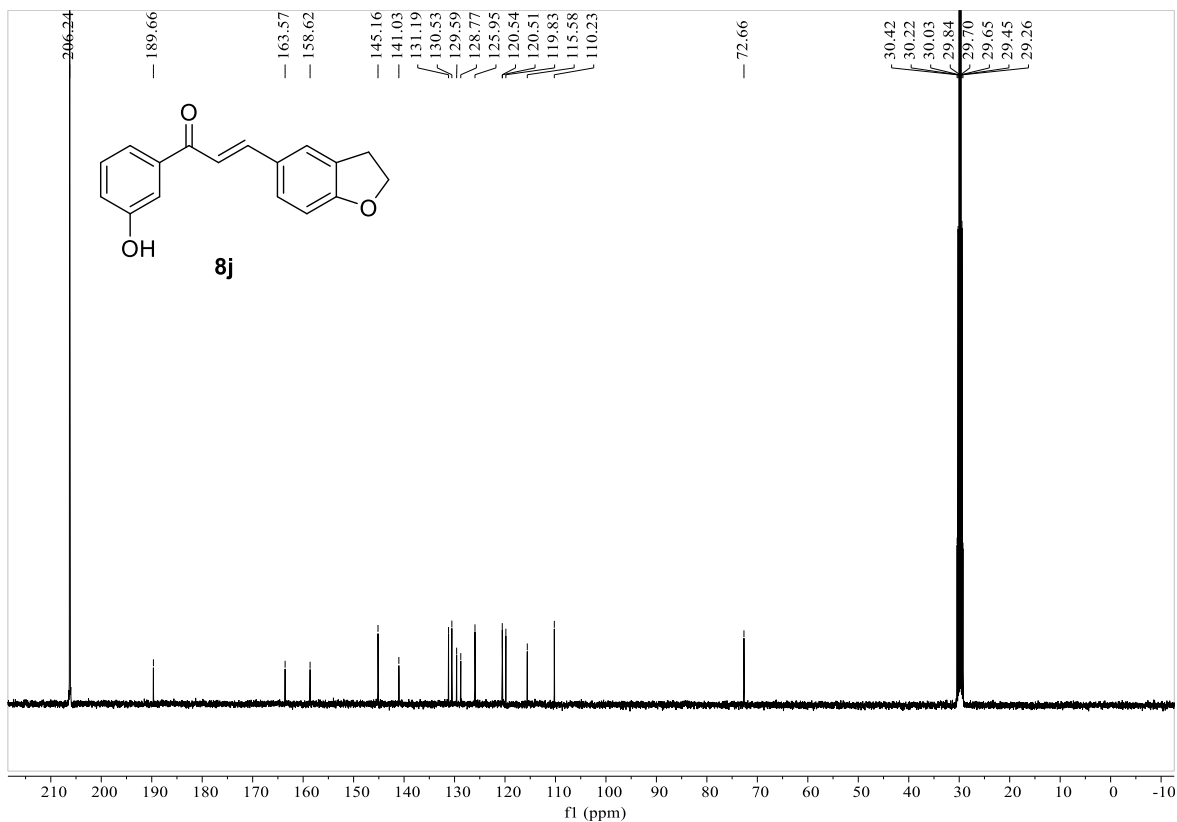
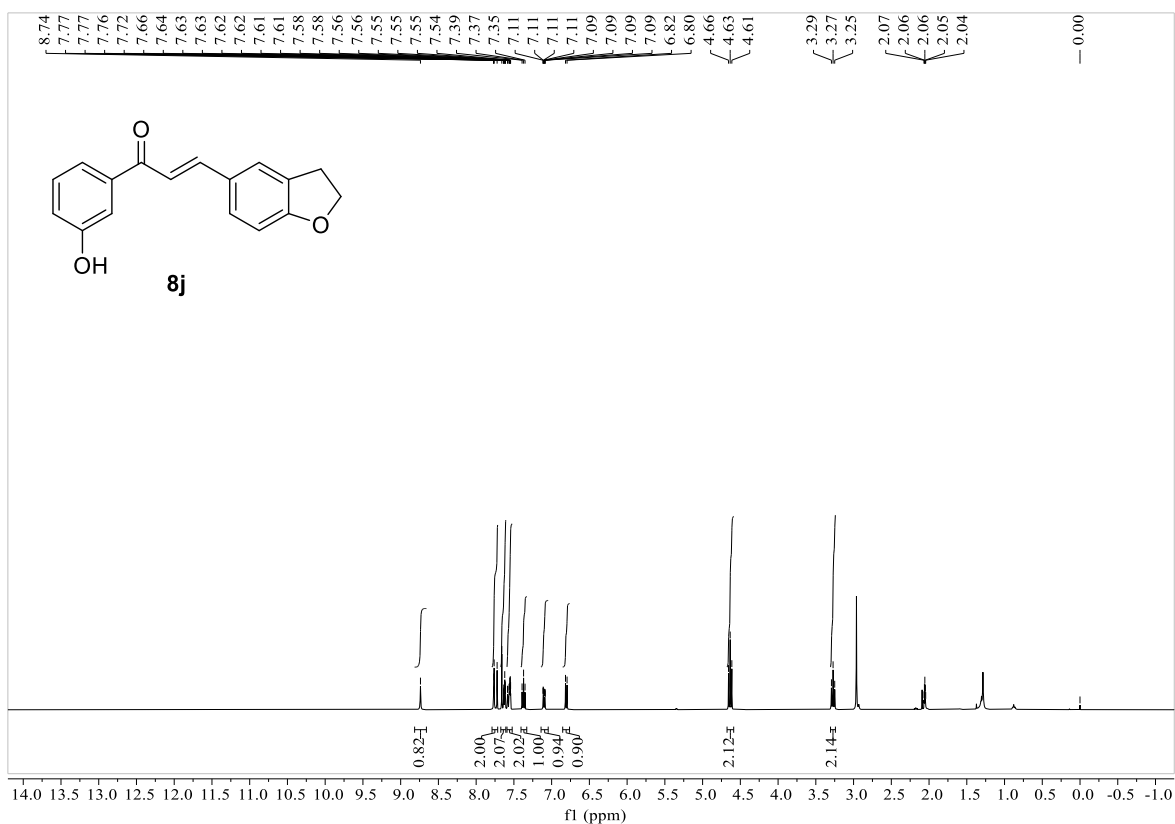
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8h**



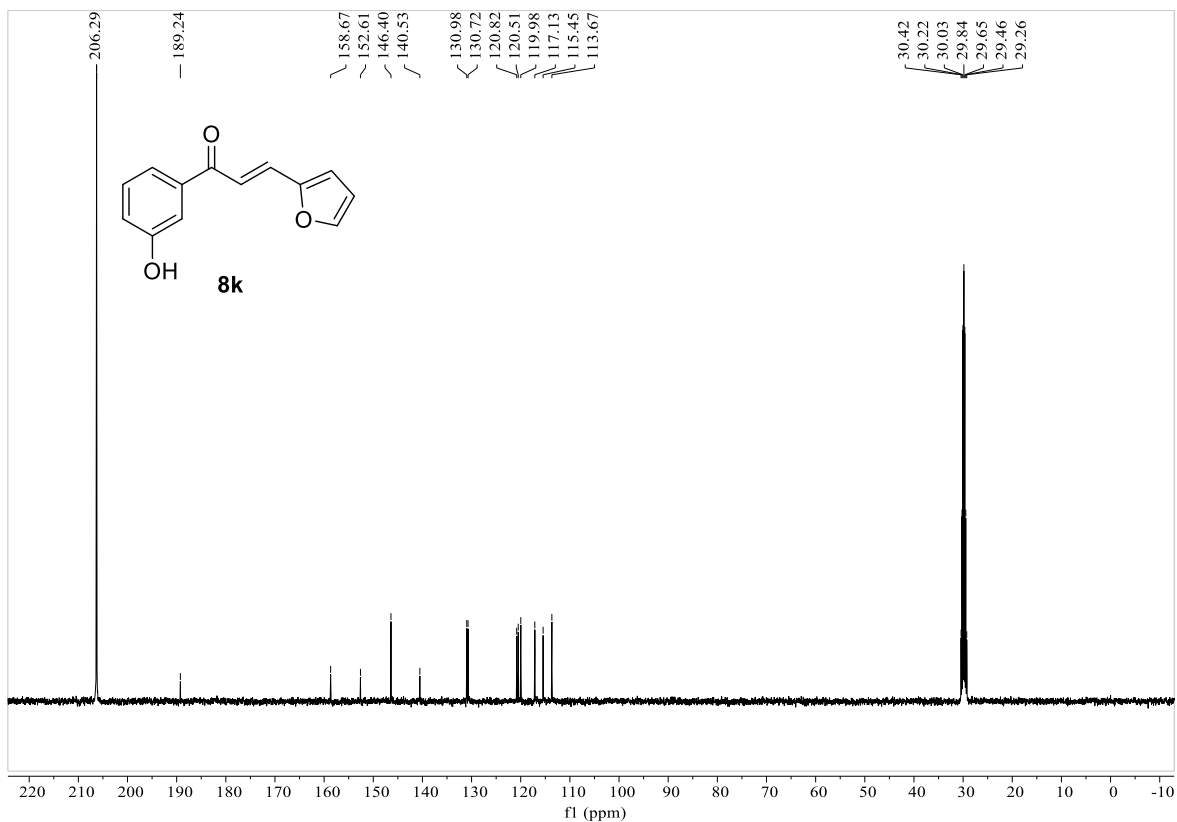
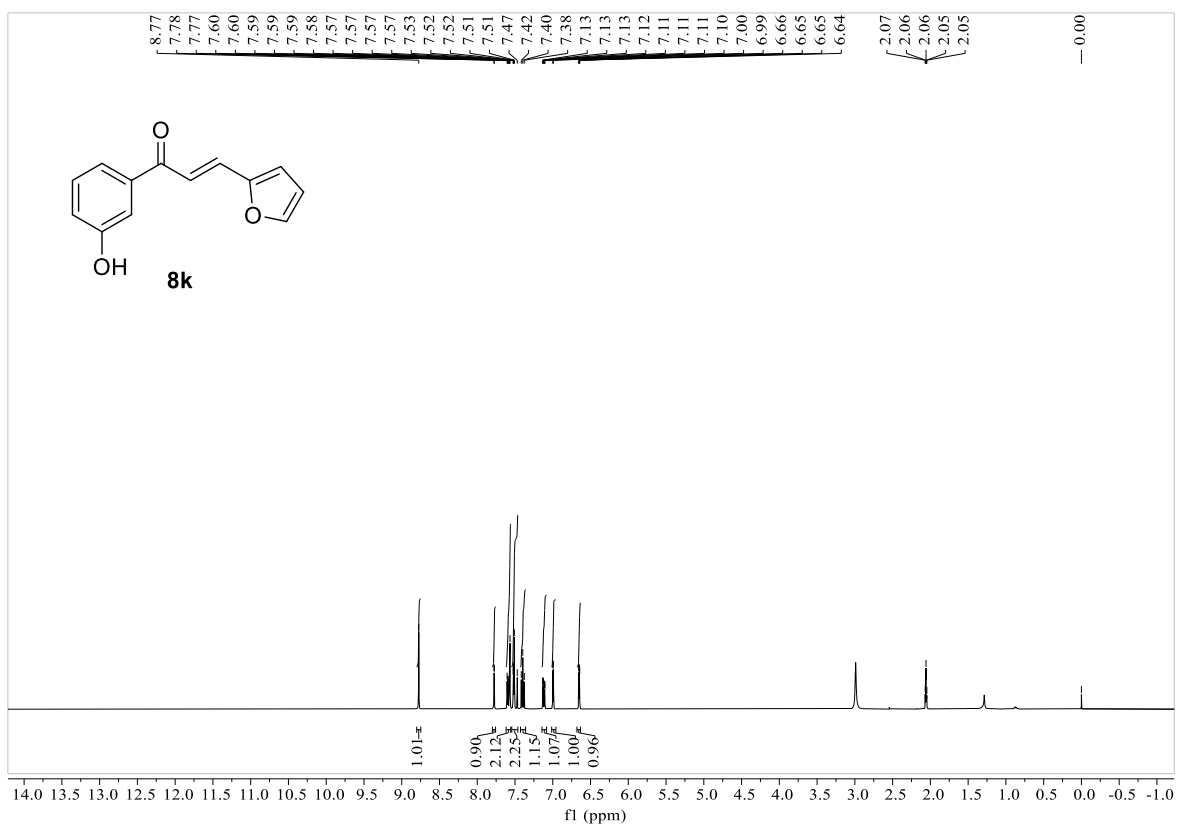
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8i**



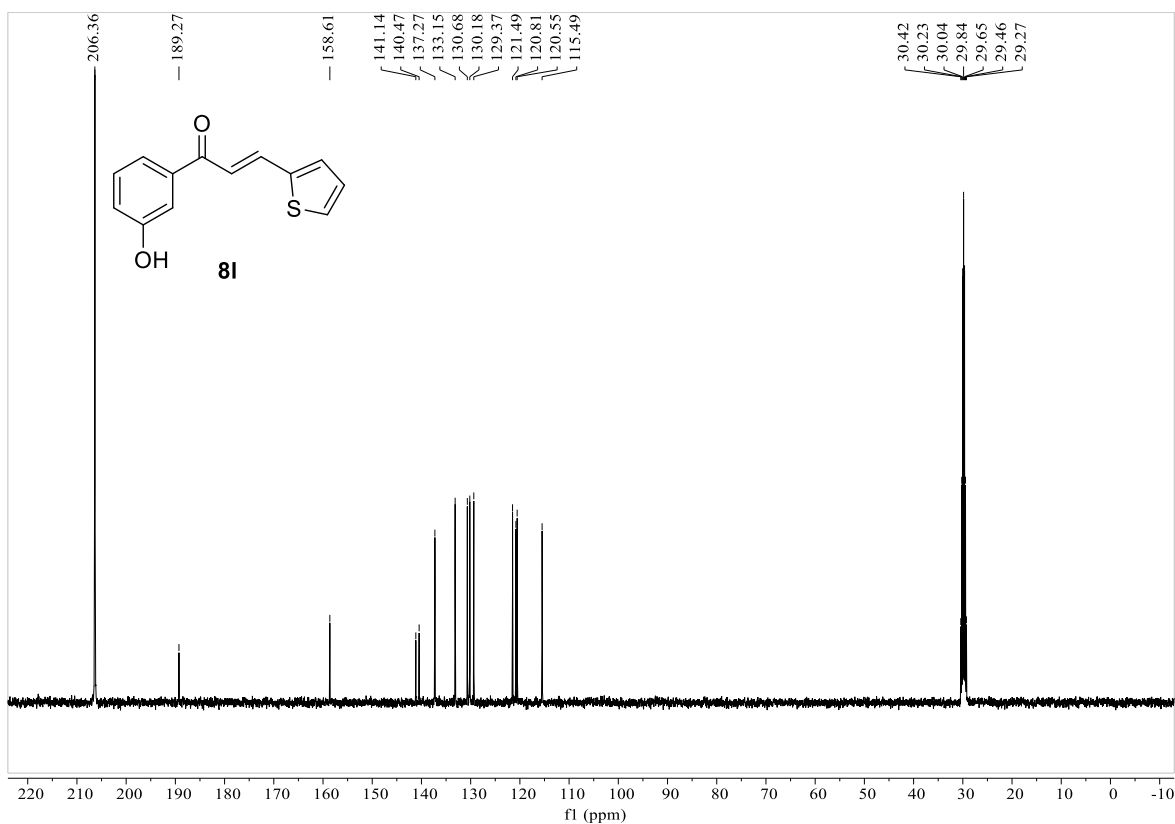
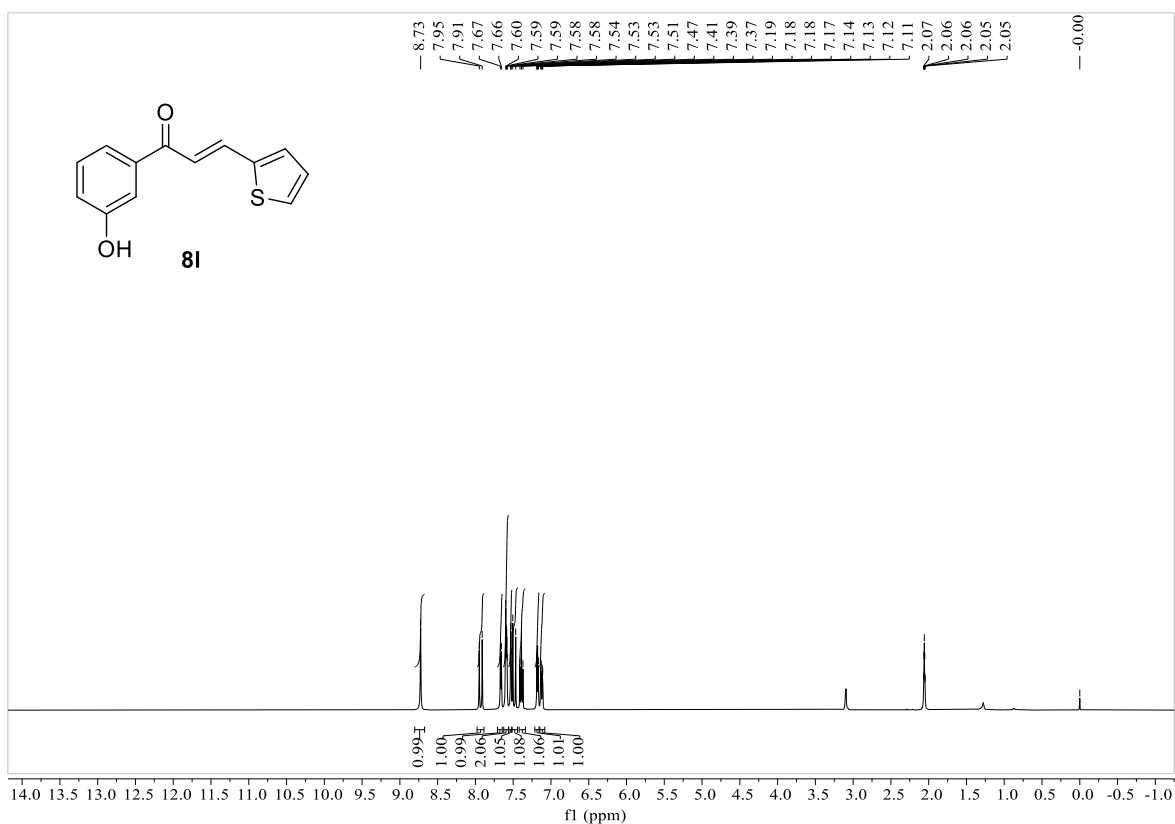
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8j**



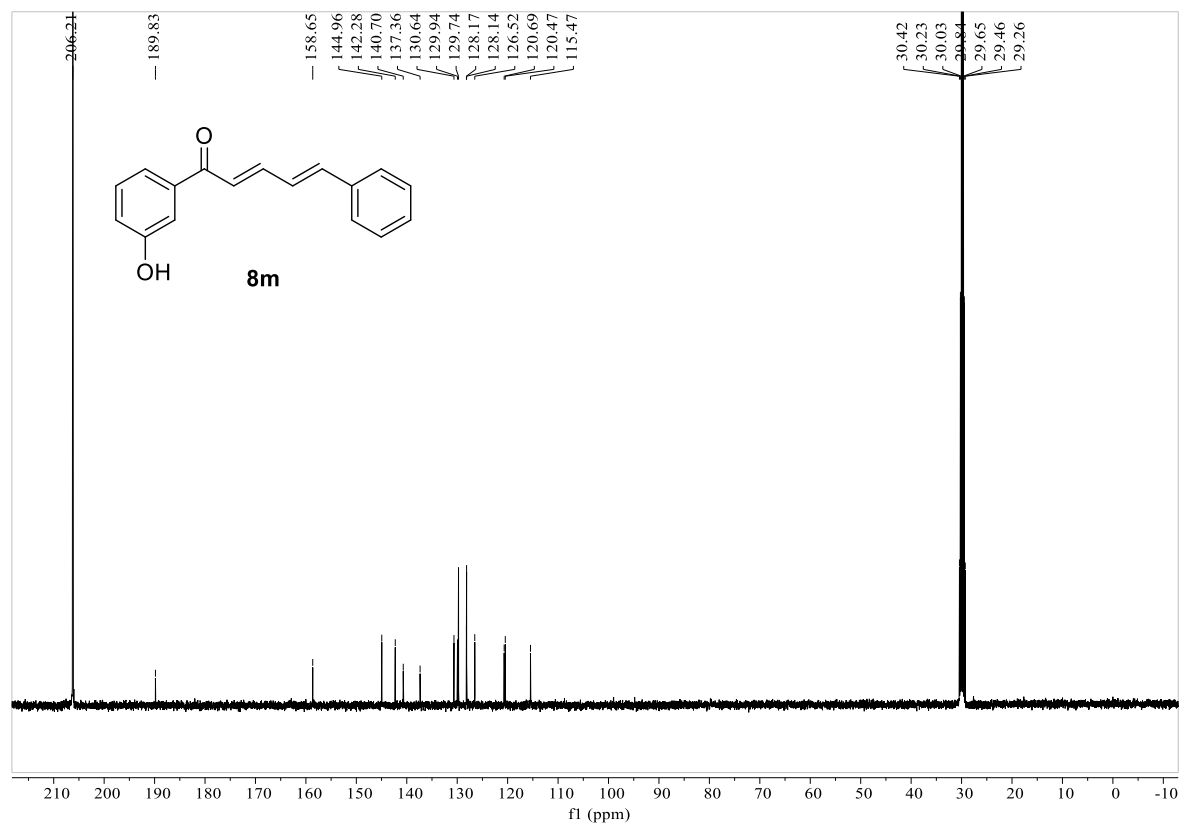
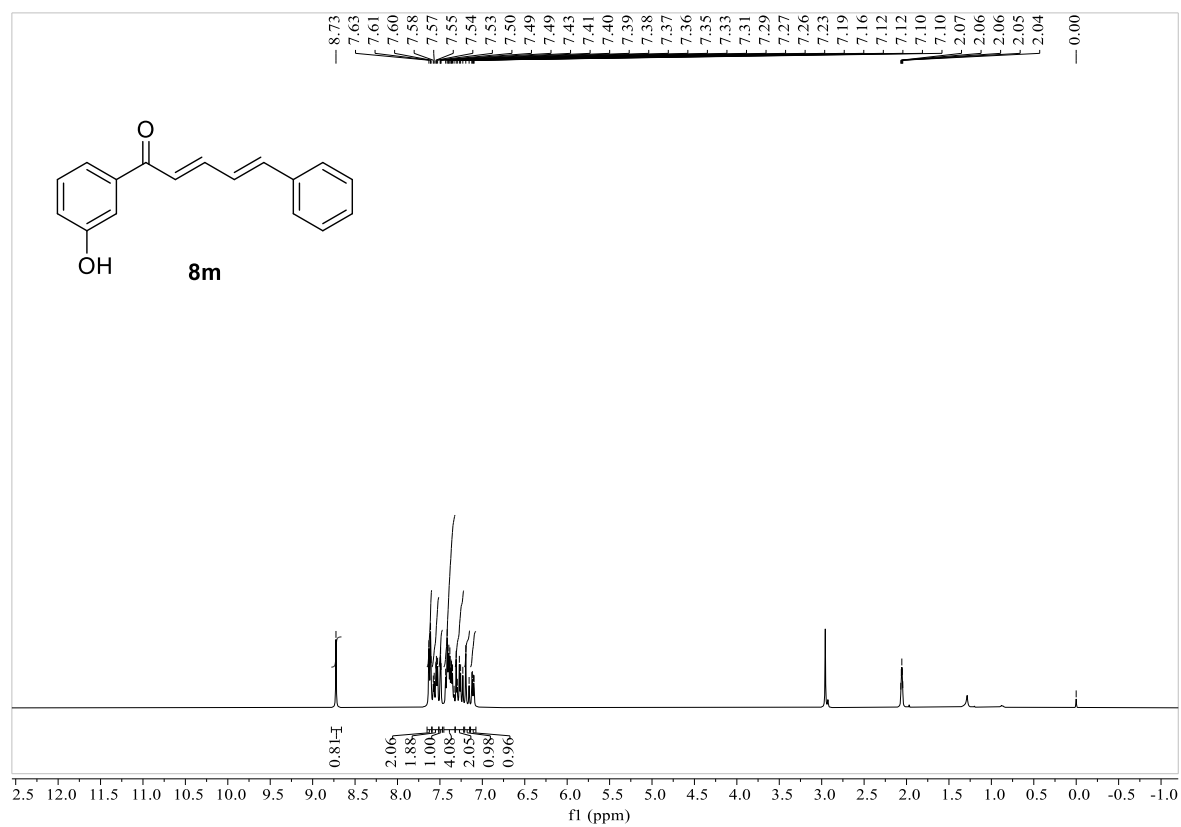
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8k**



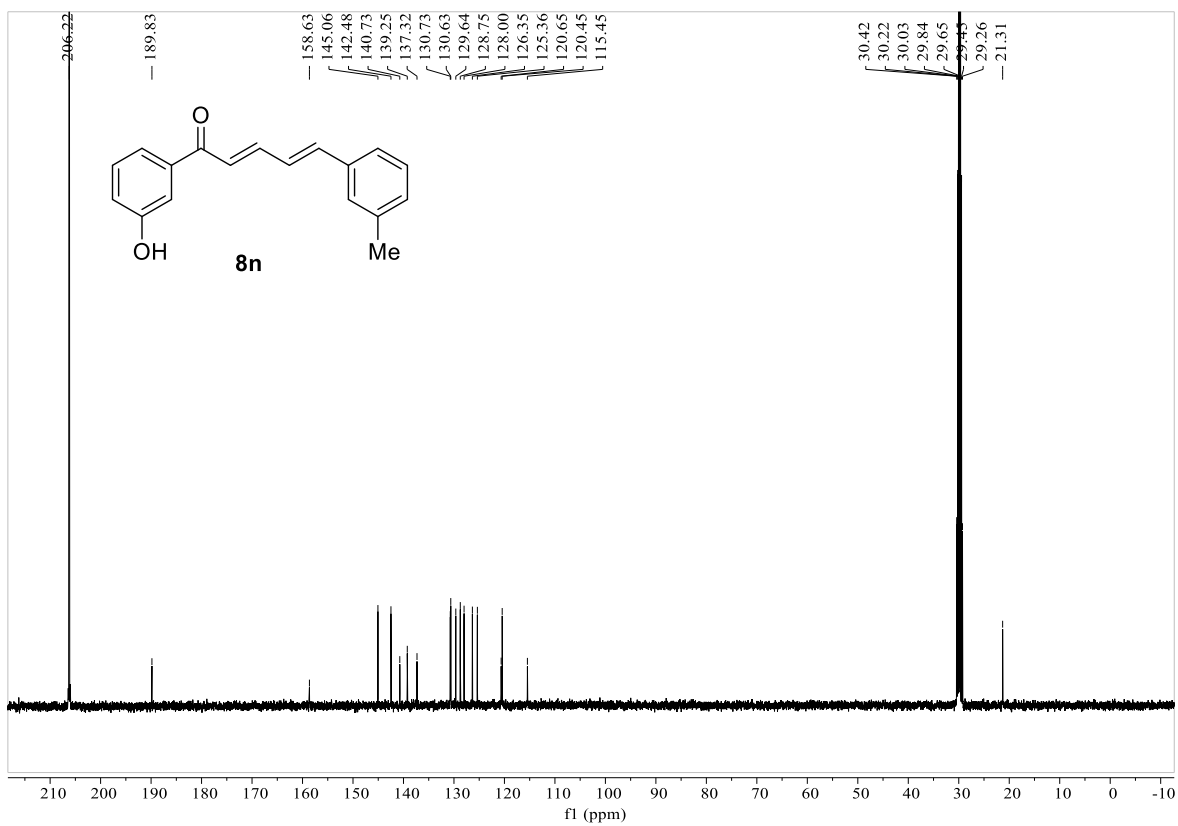
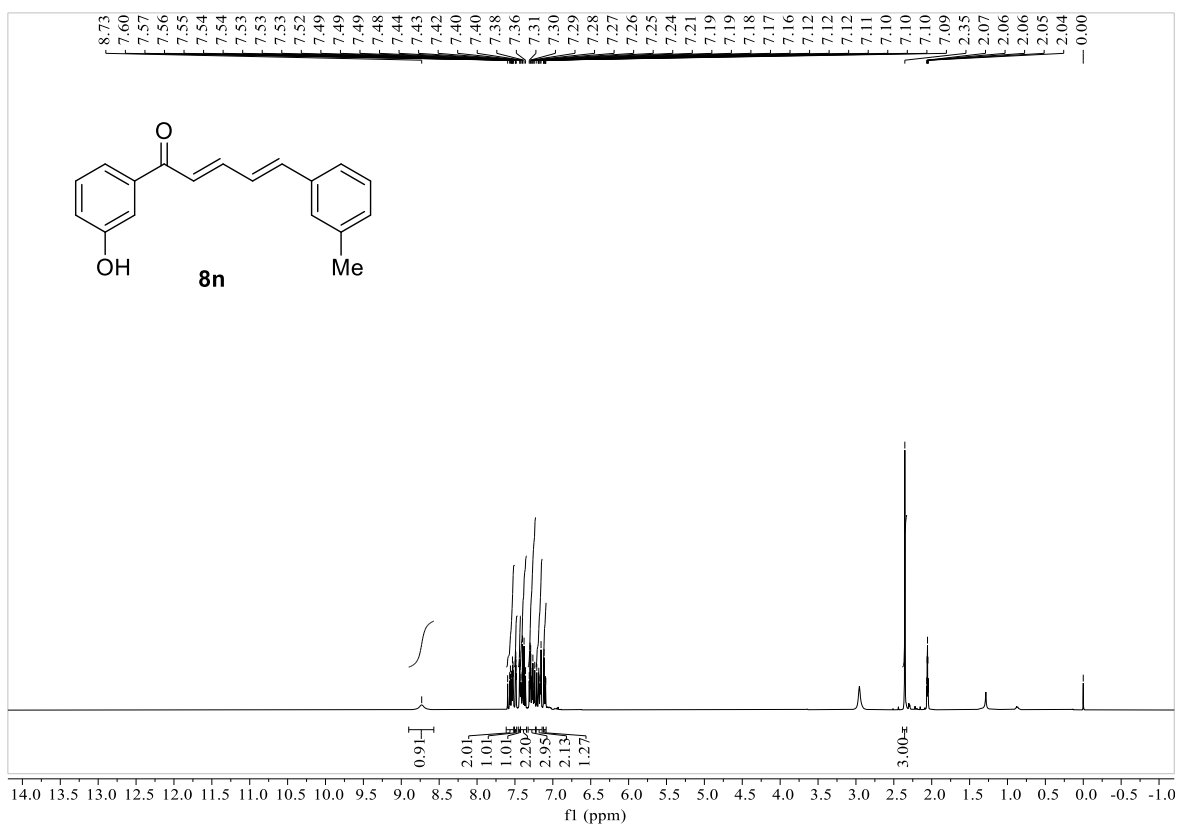
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8l**



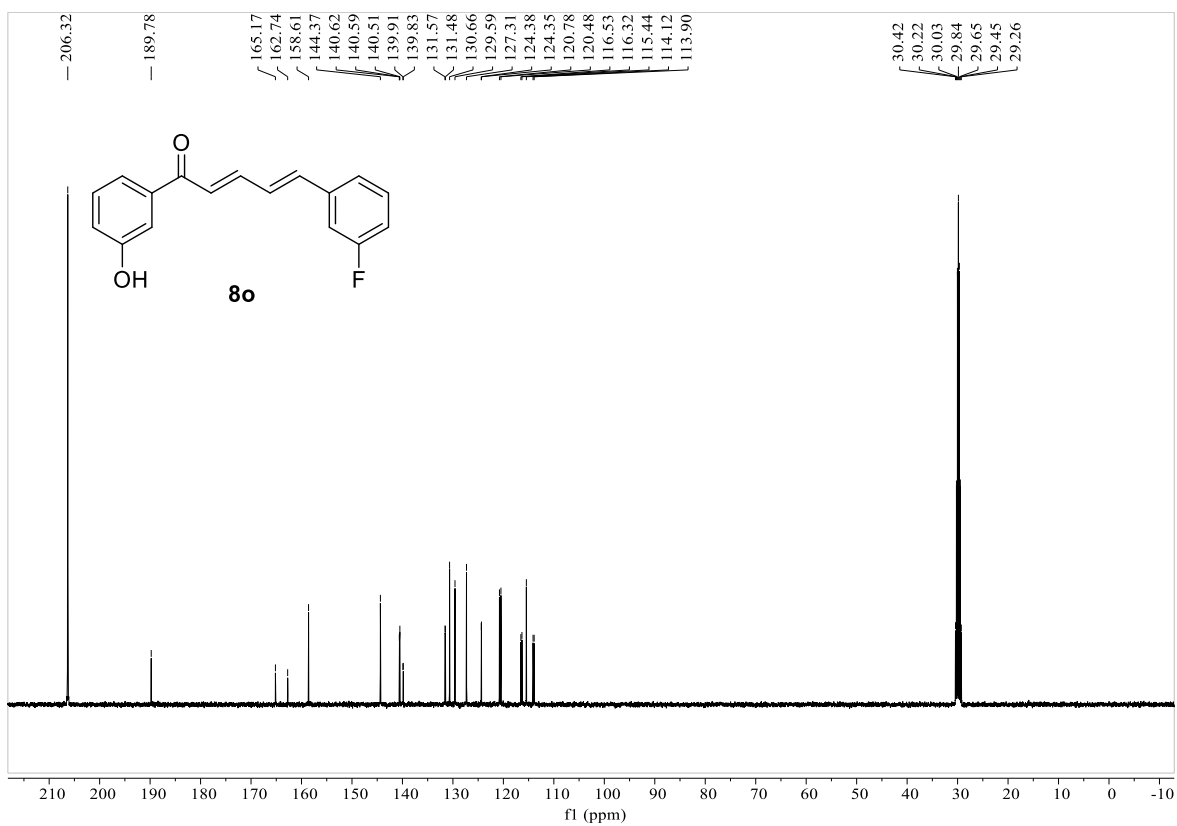
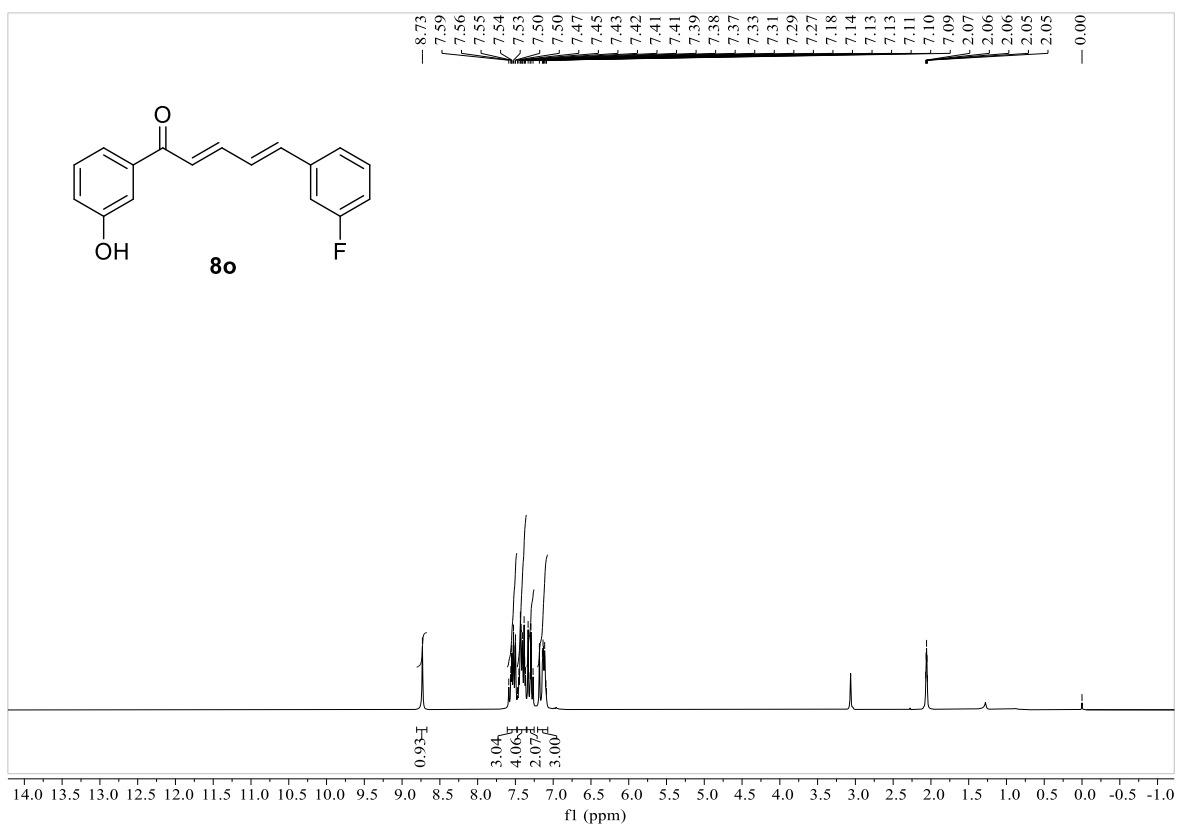
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8m**



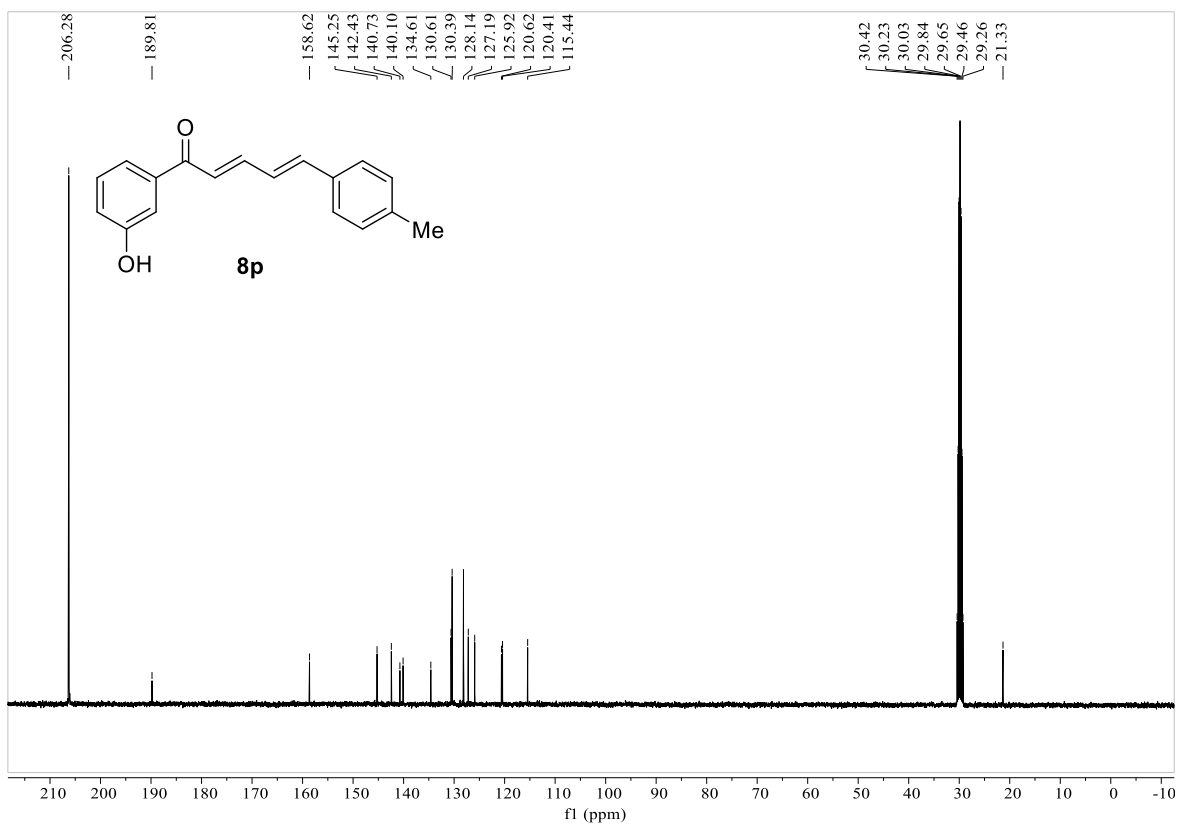
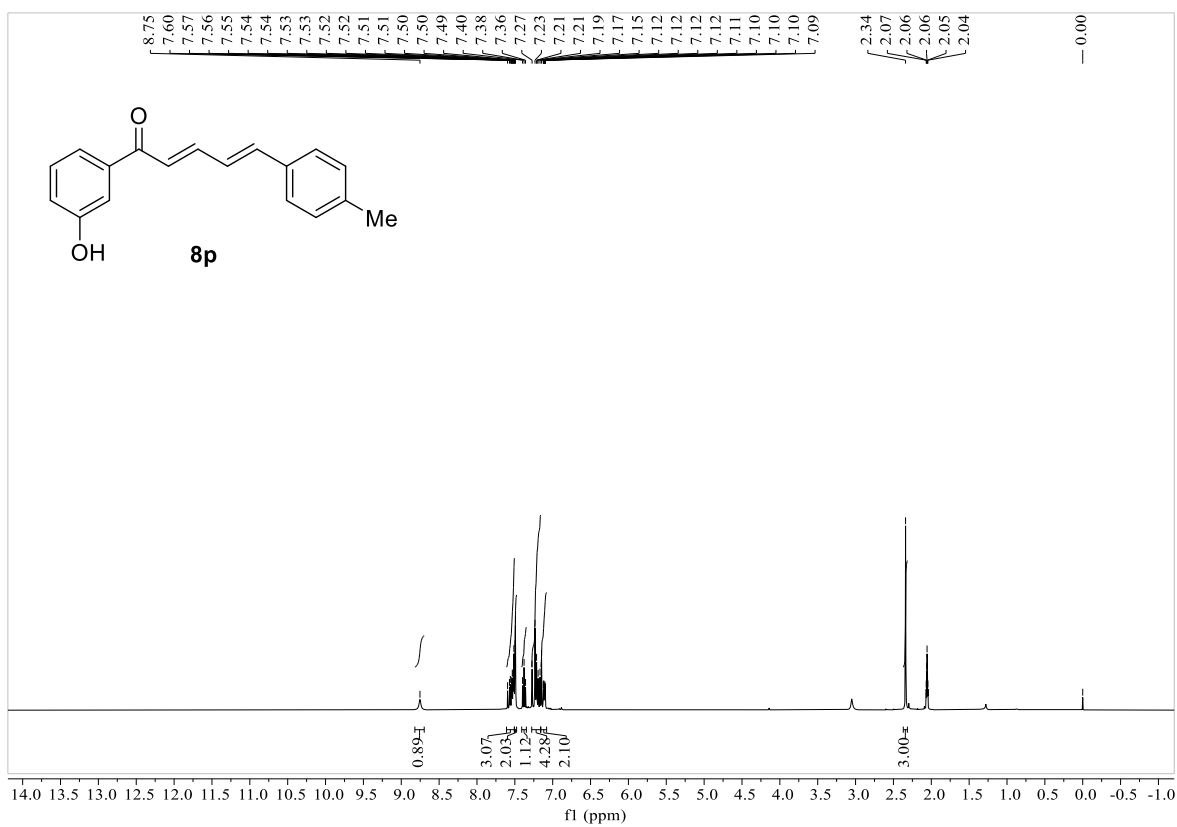
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8n**



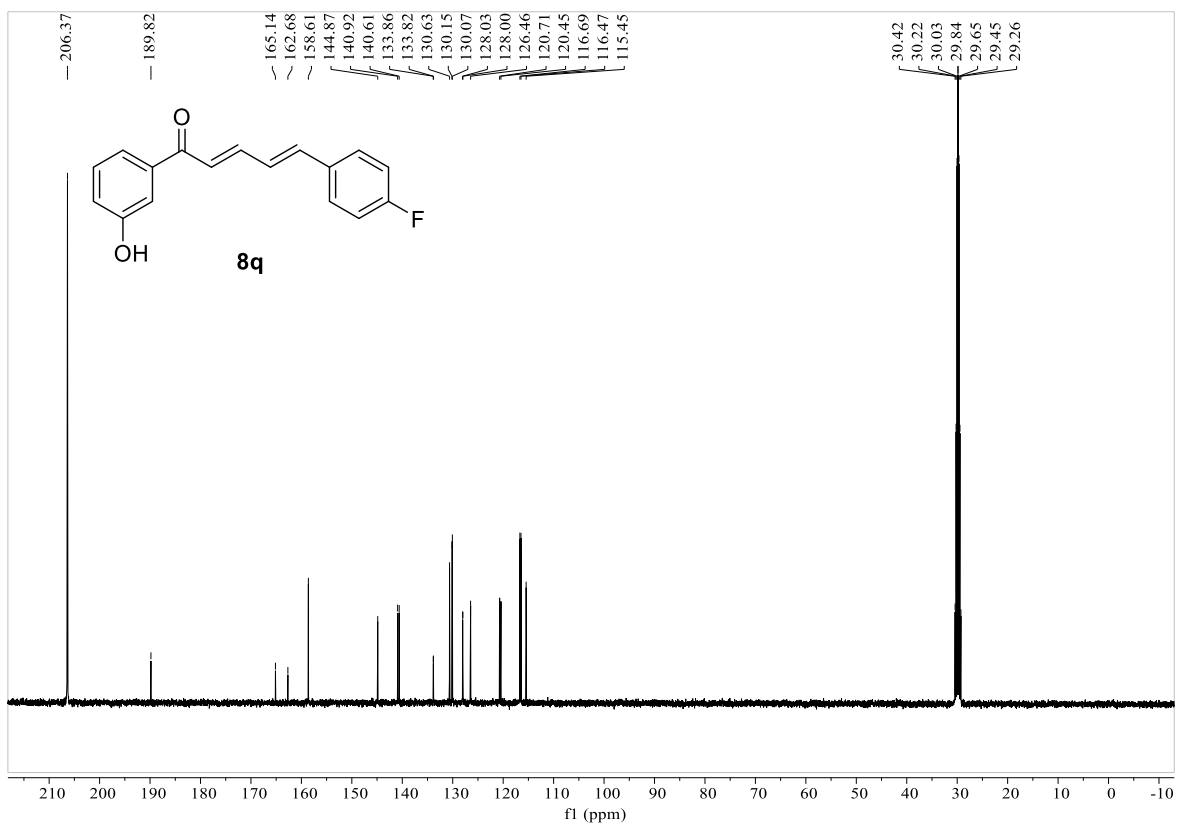
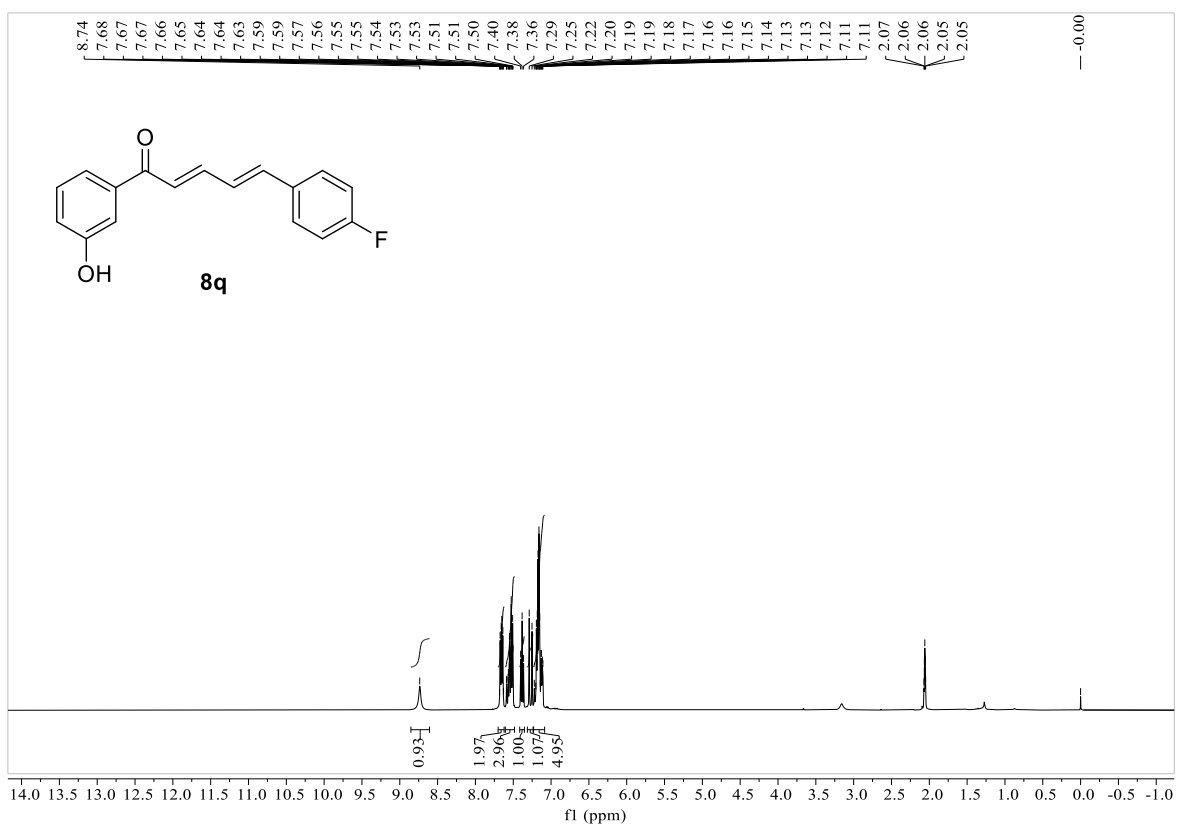
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8o**



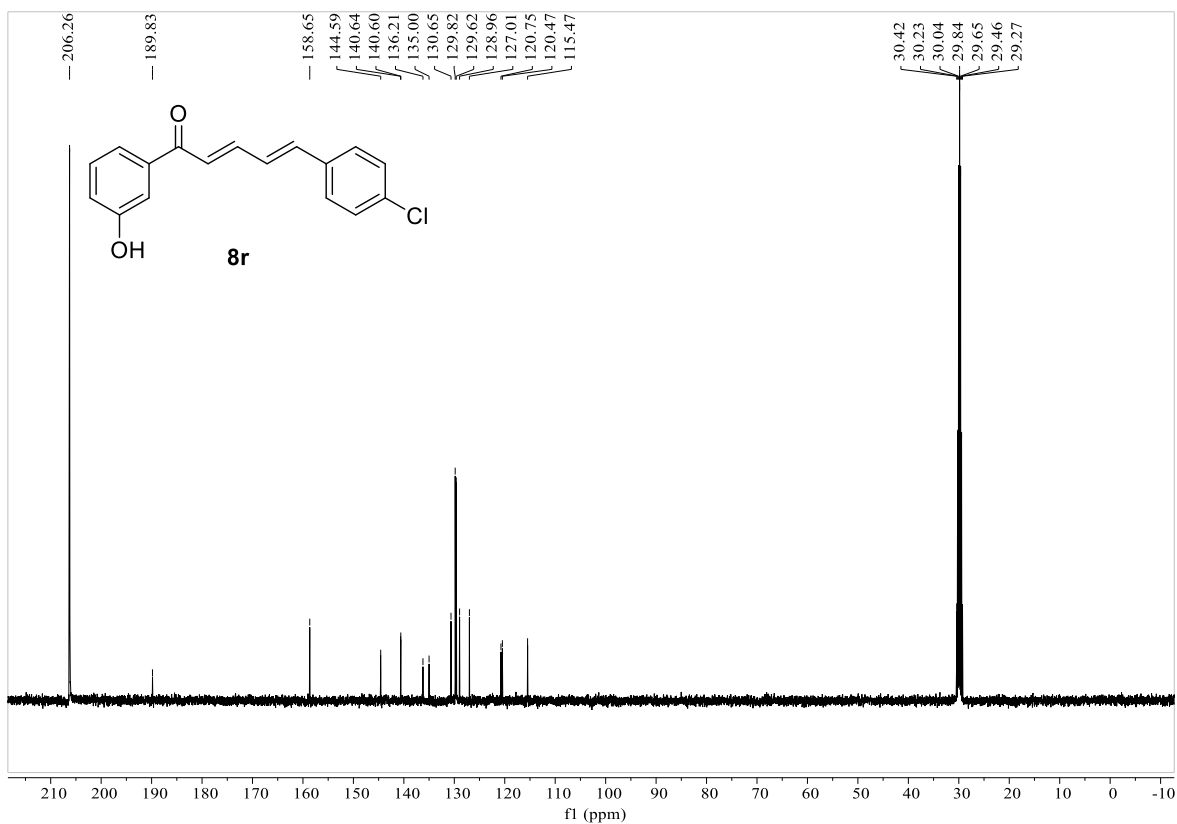
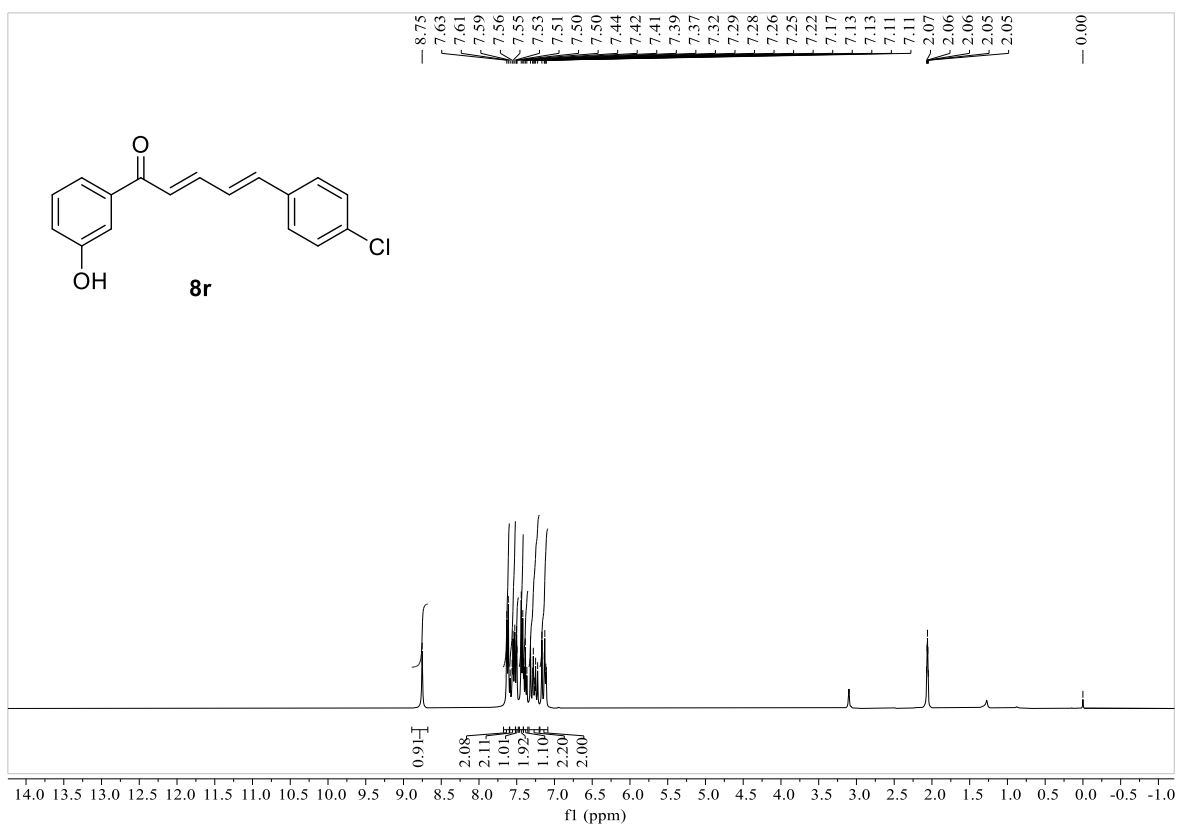
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8p**



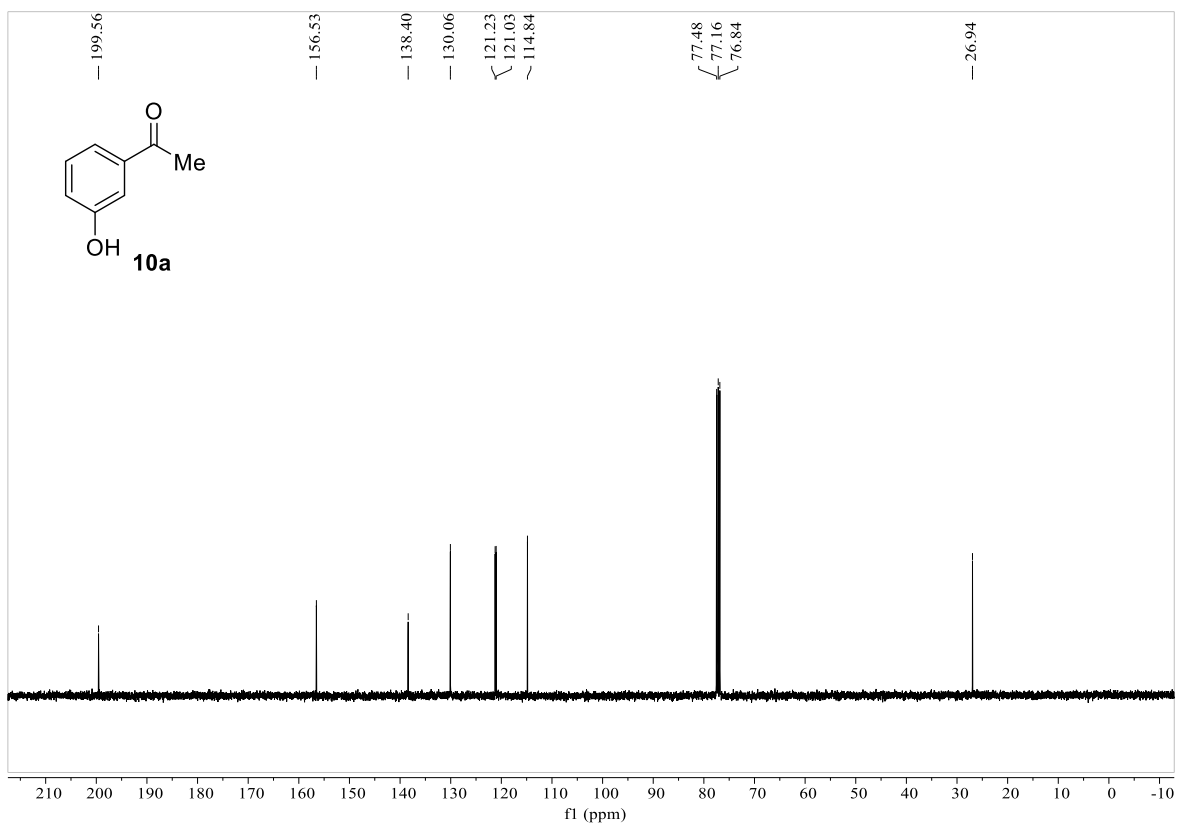
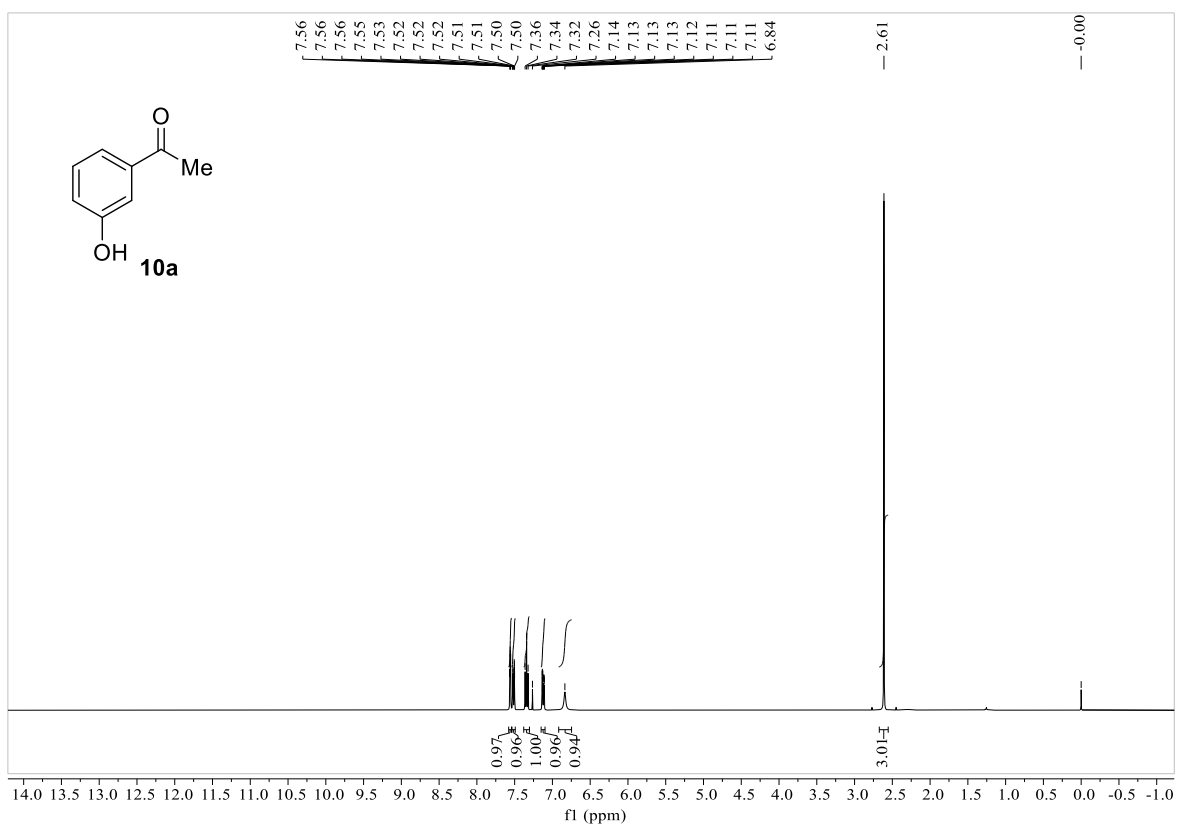
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8q**



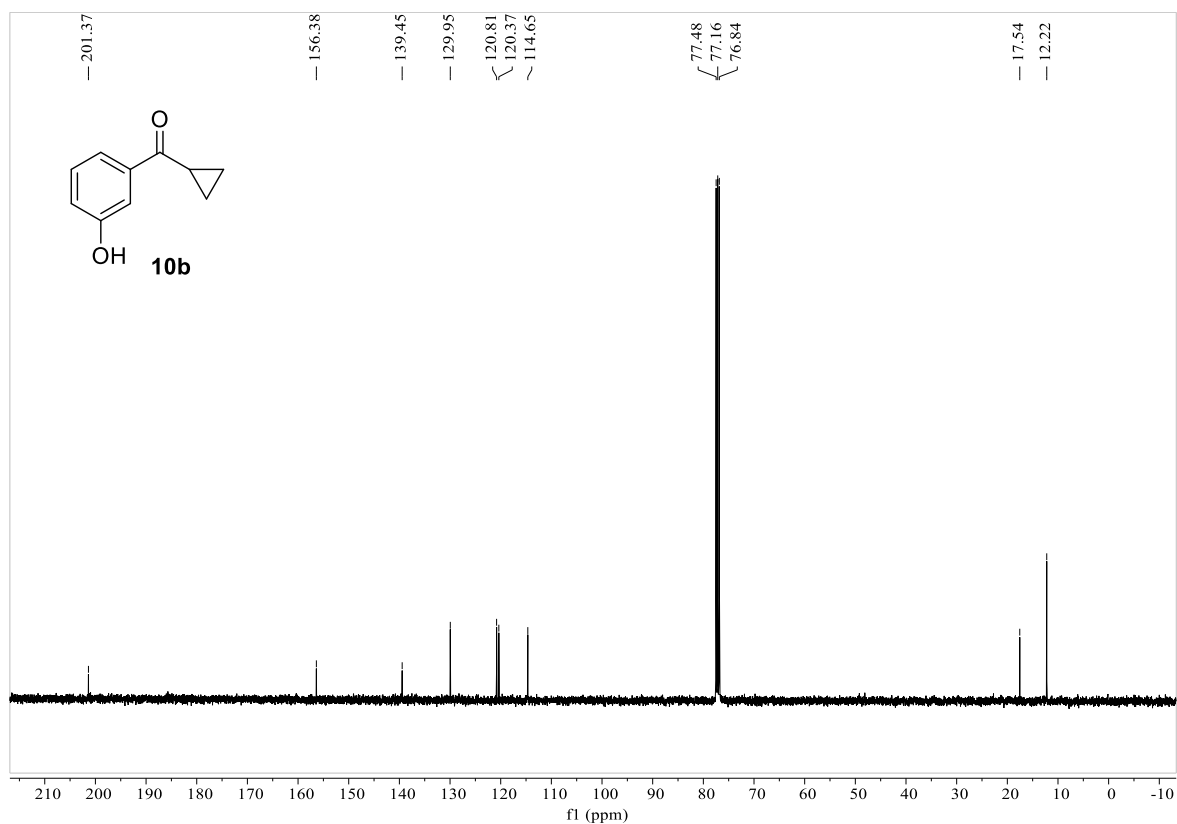
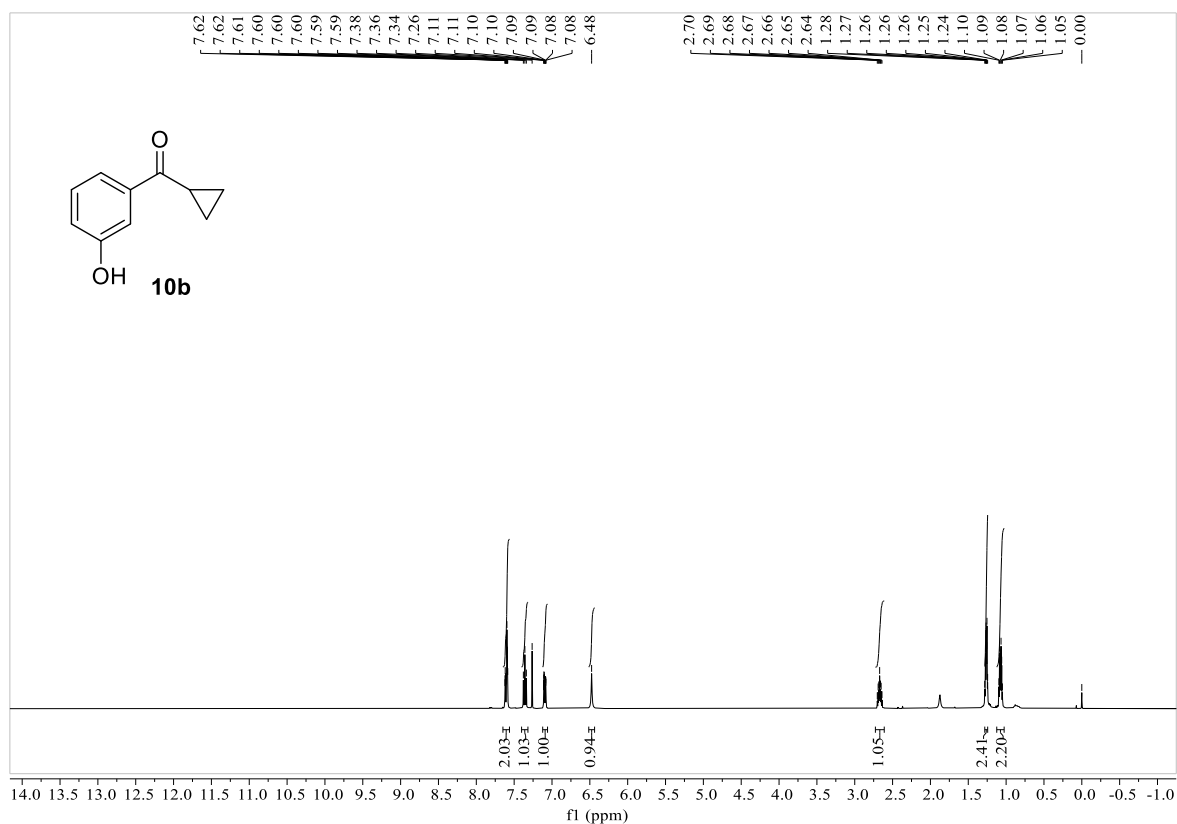
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **8r**



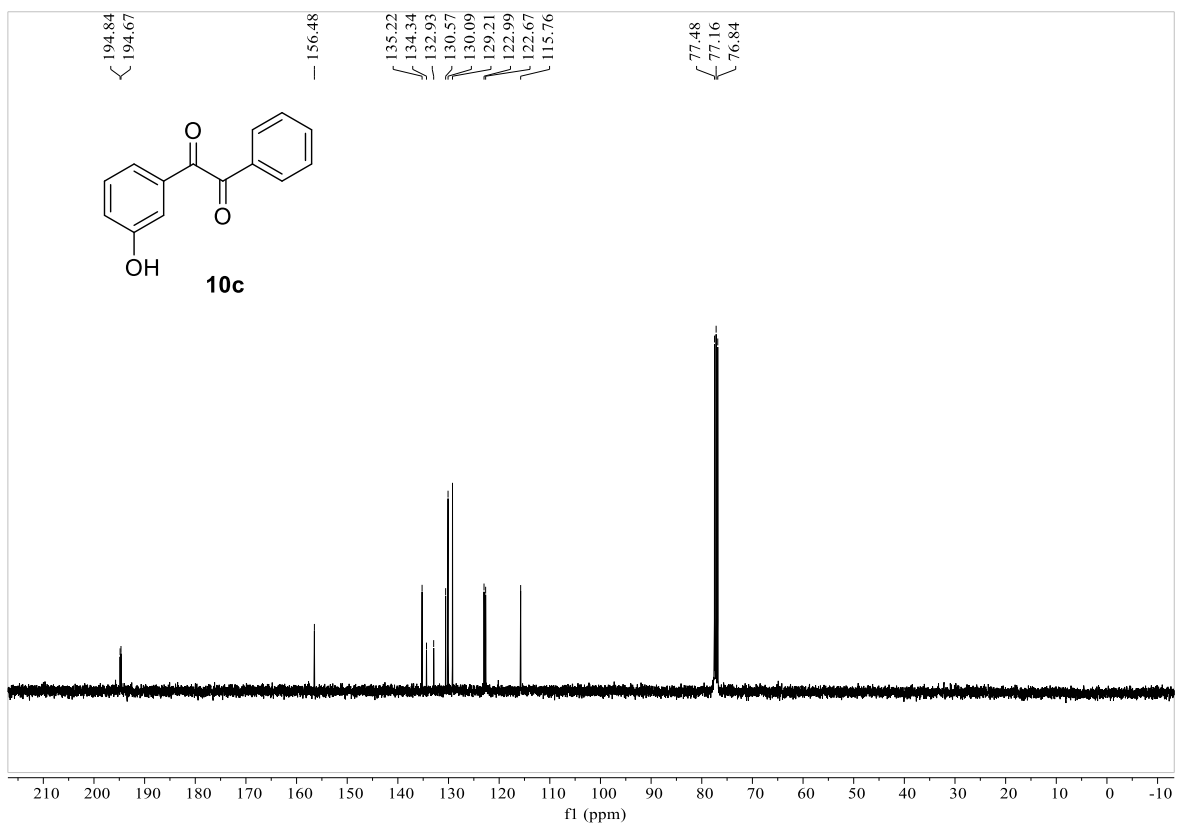
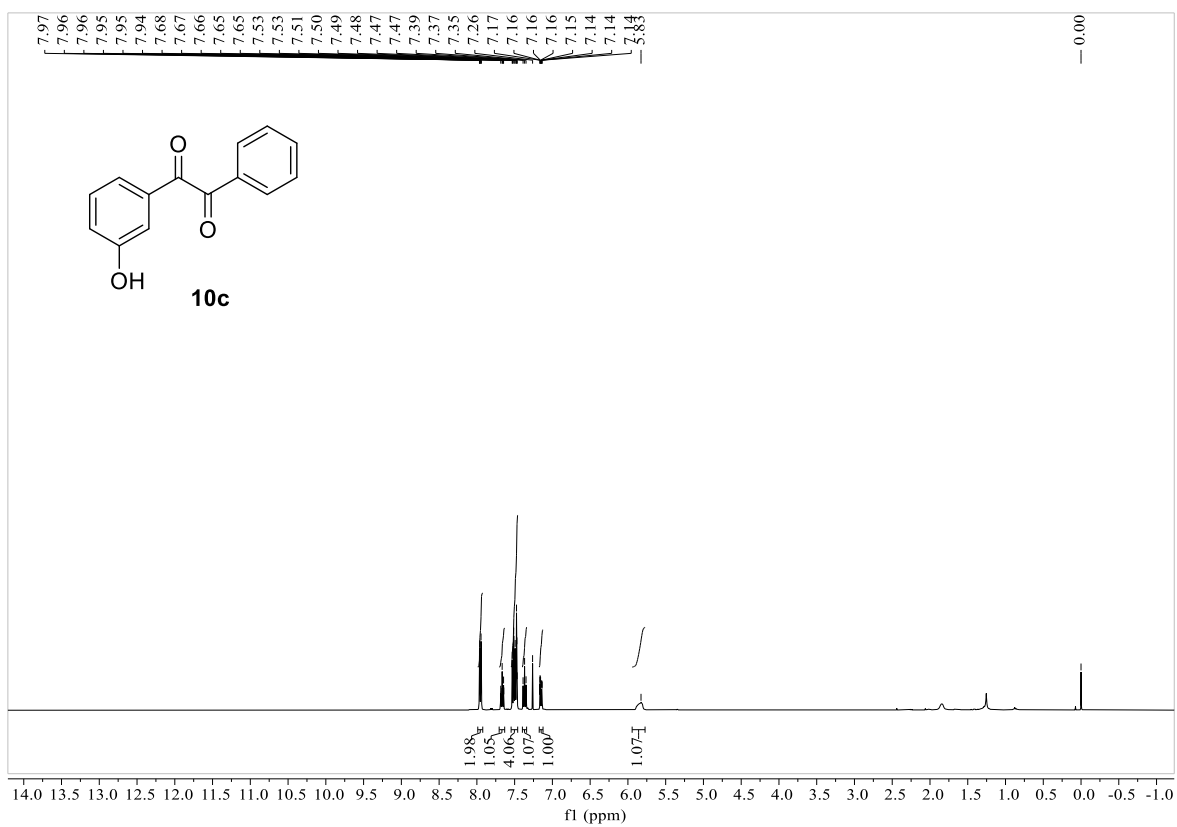
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **10a**



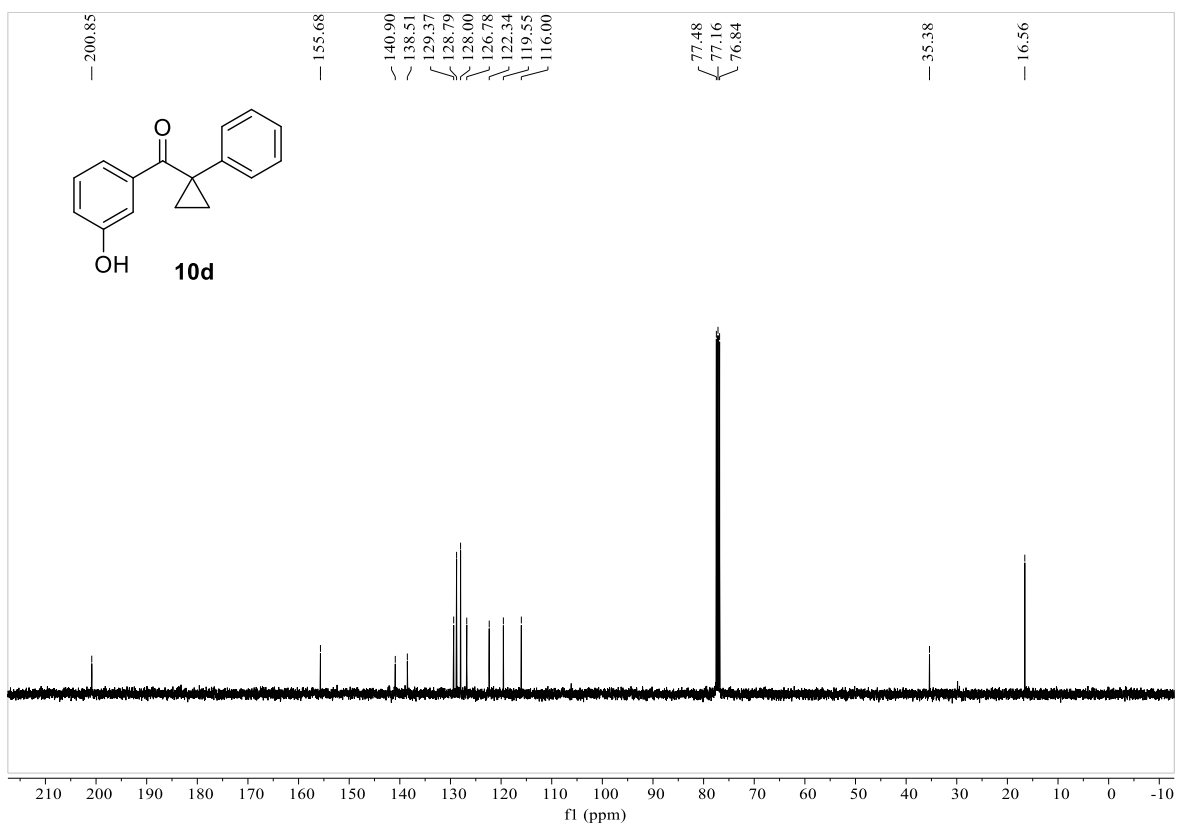
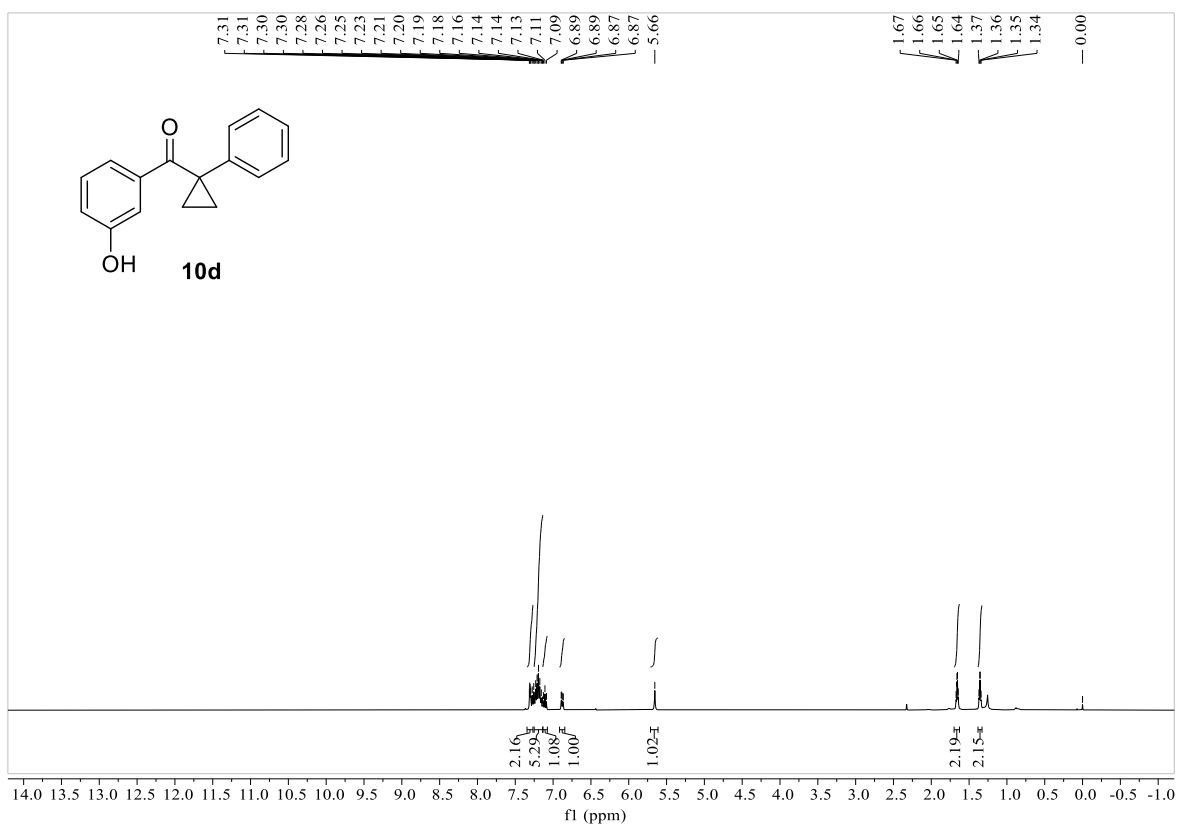
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **10b**



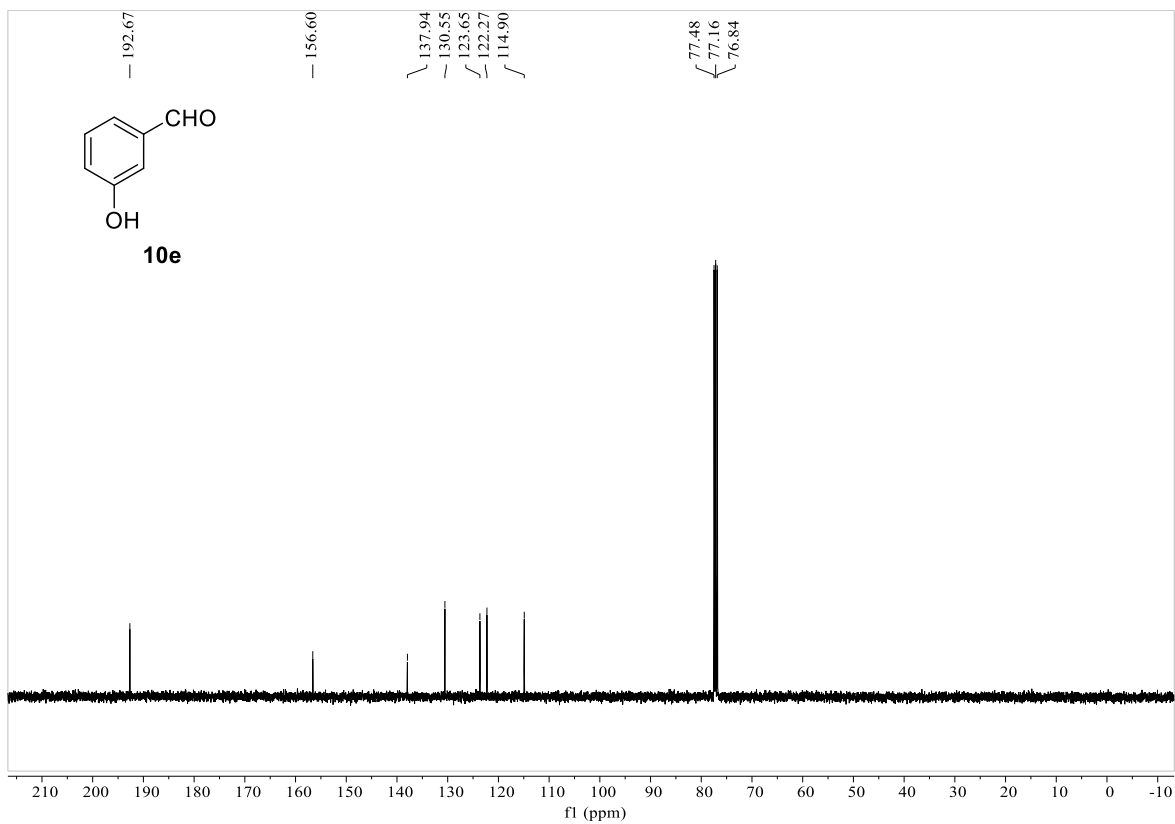
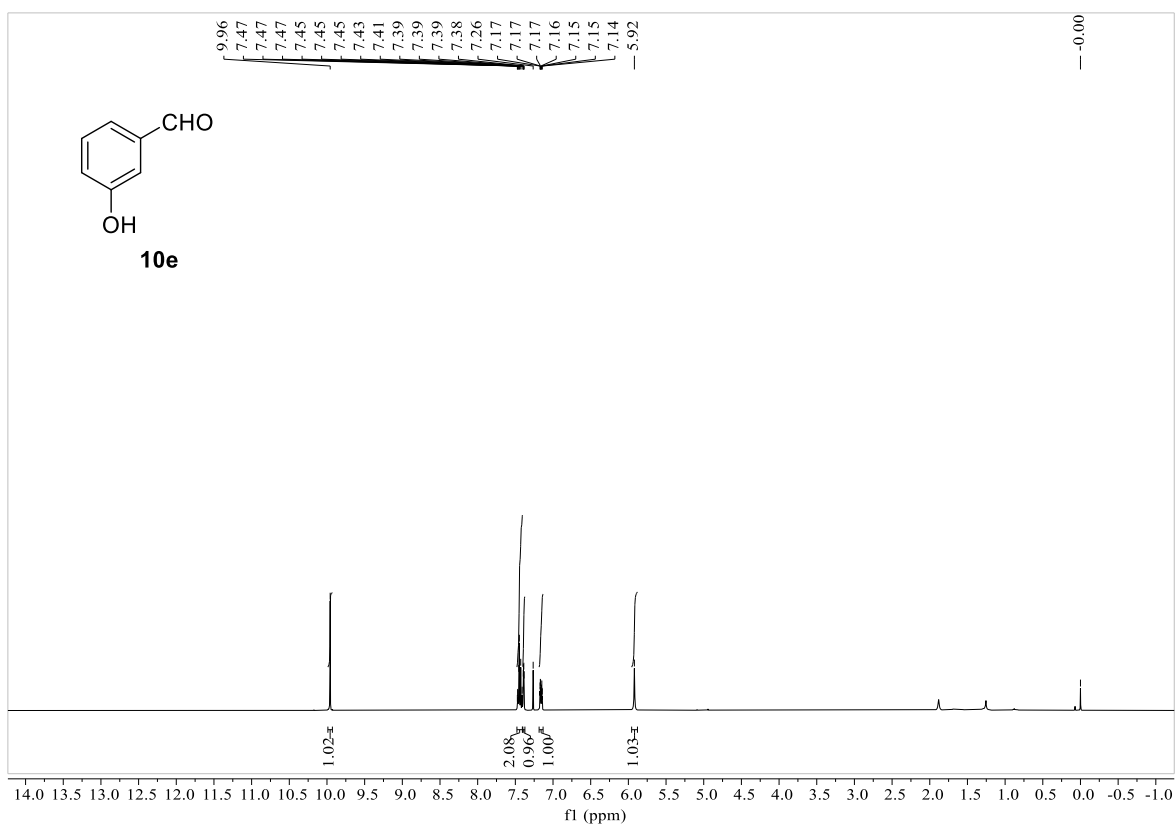
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **10c**



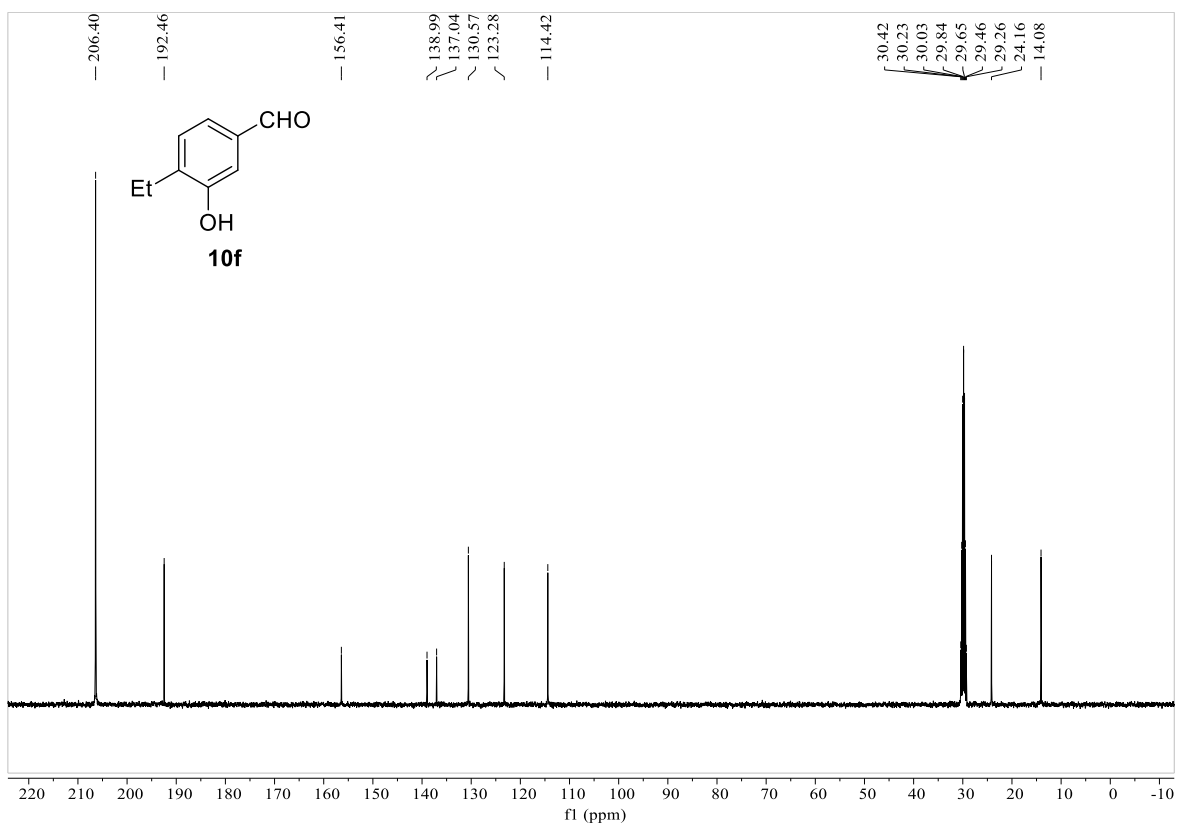
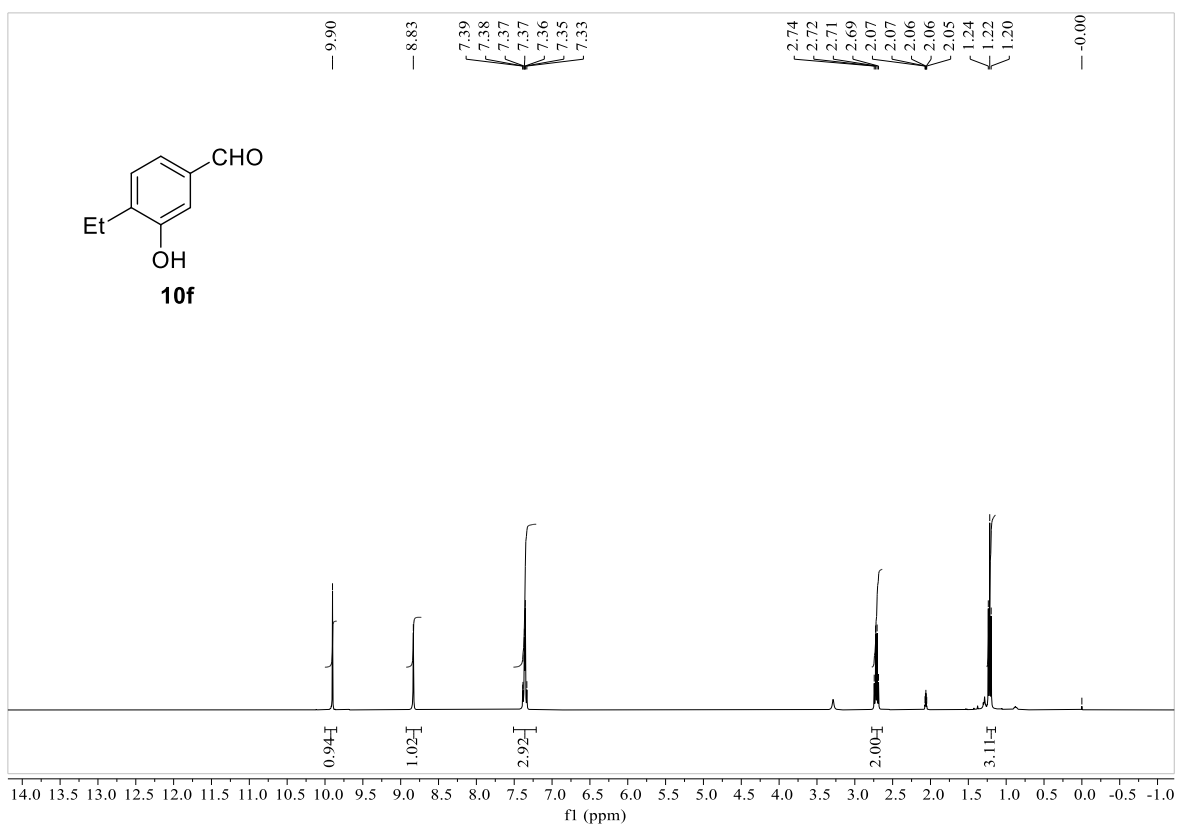
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **10d**



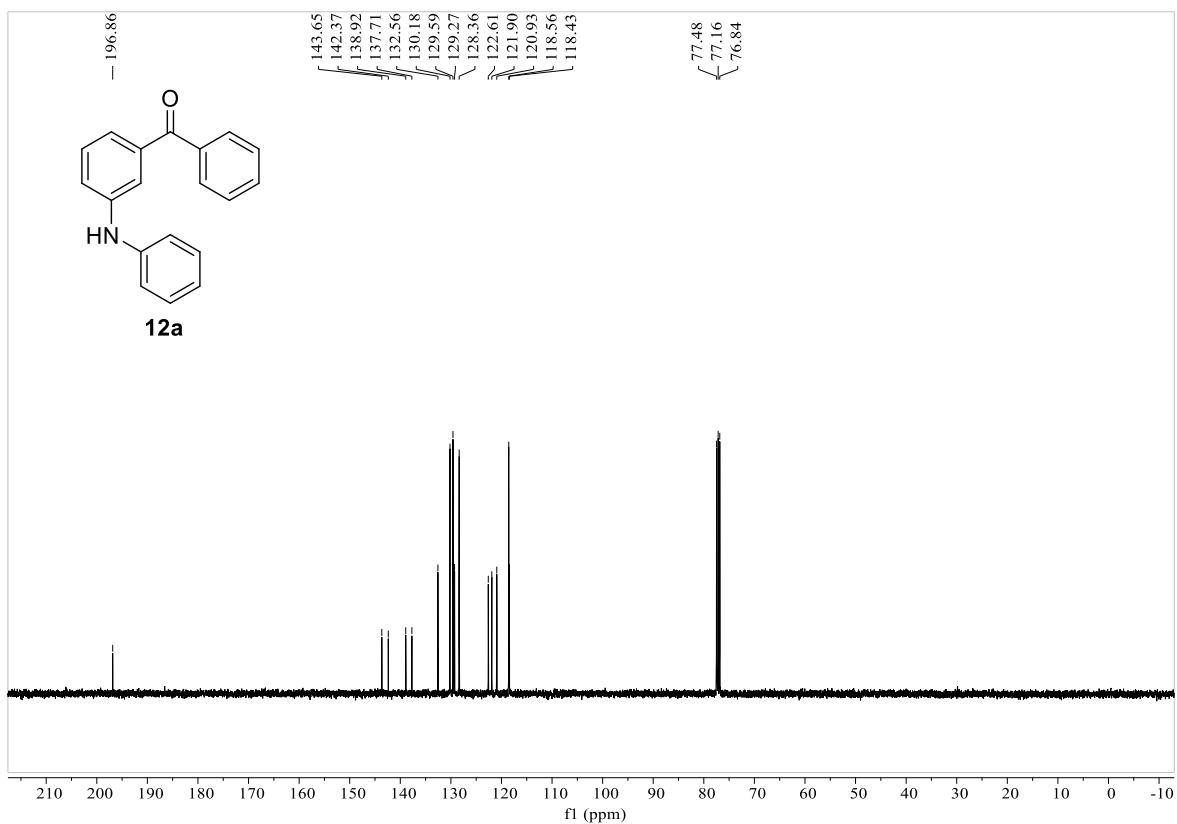
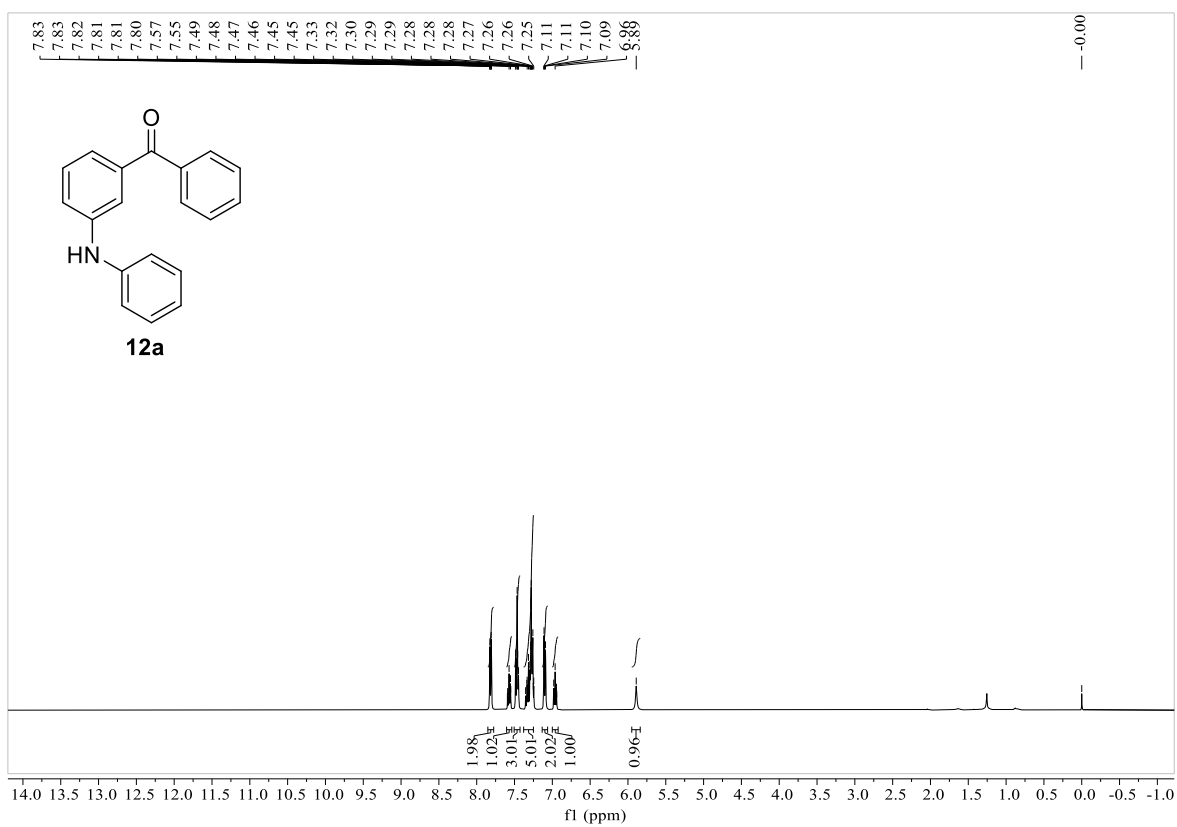
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **10e**



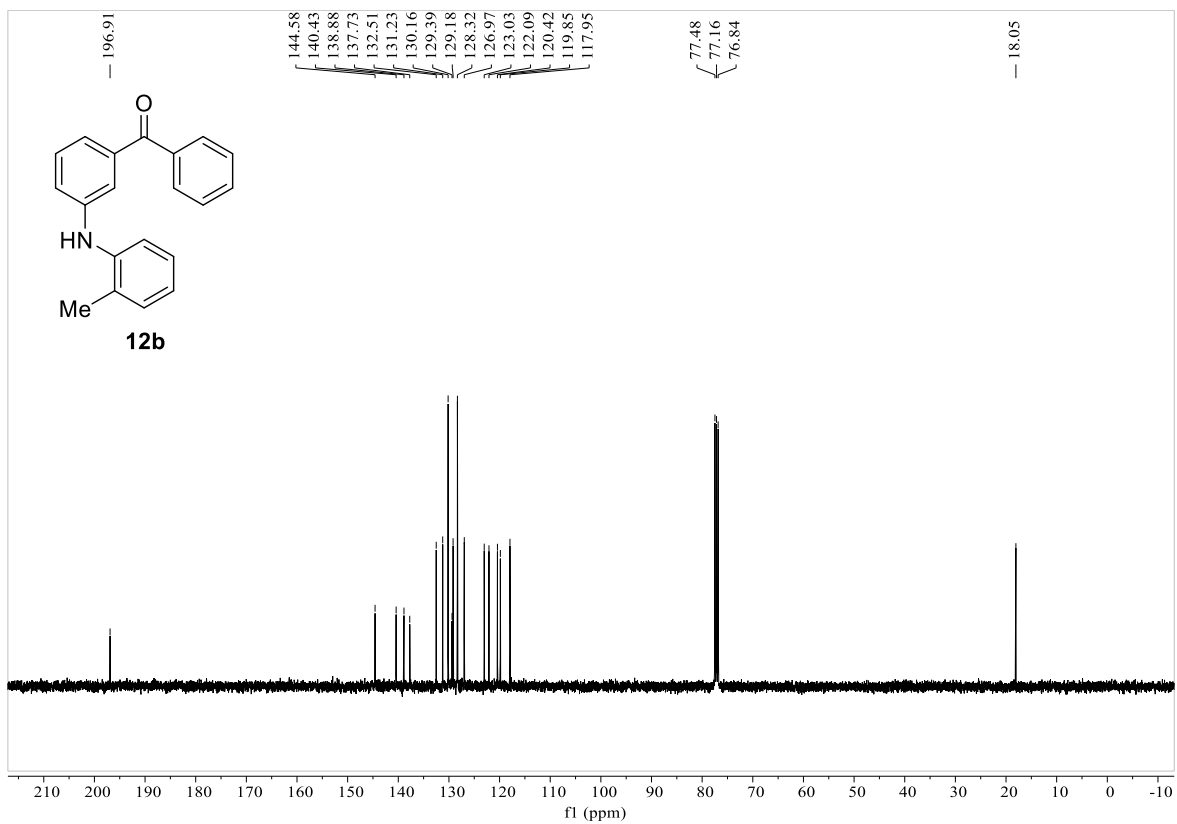
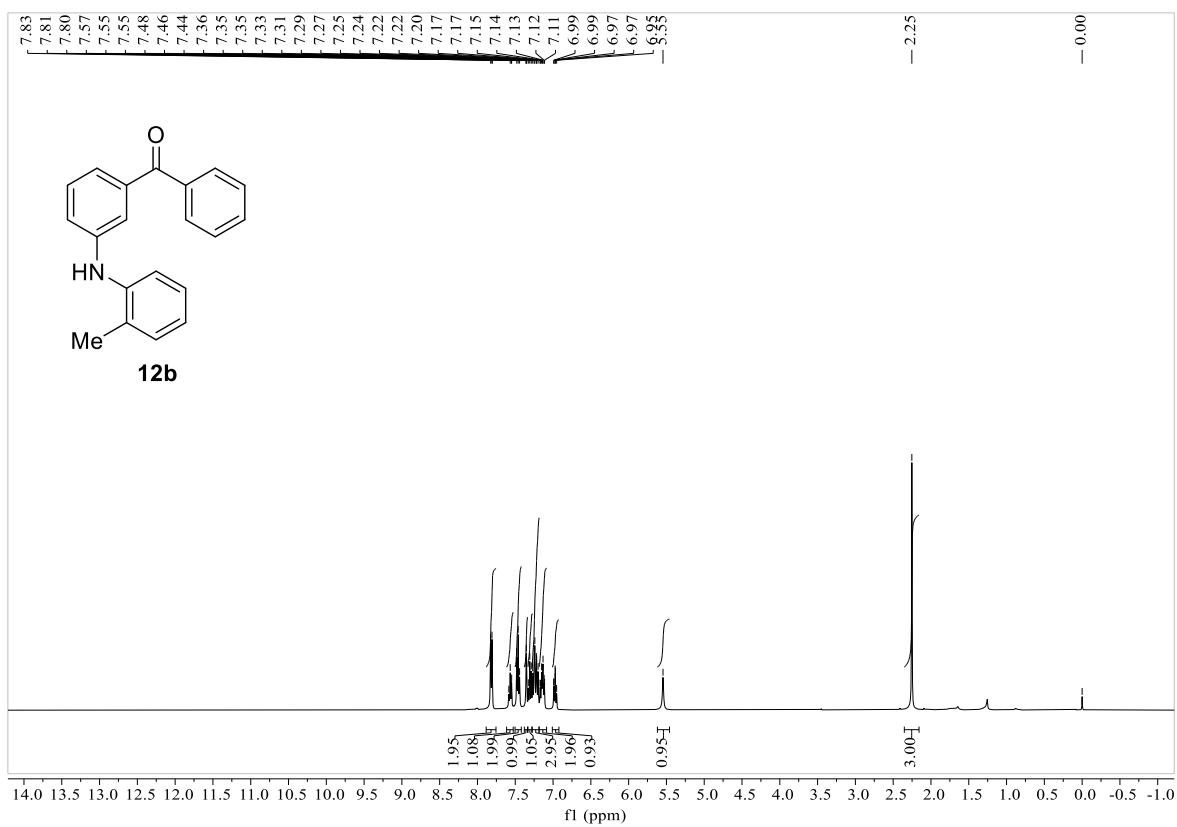
^1H NMR (400 MHz, Acetone- d_6) and ^{13}C NMR (101 MHz, Acetone- d_6) spectra of **10f**



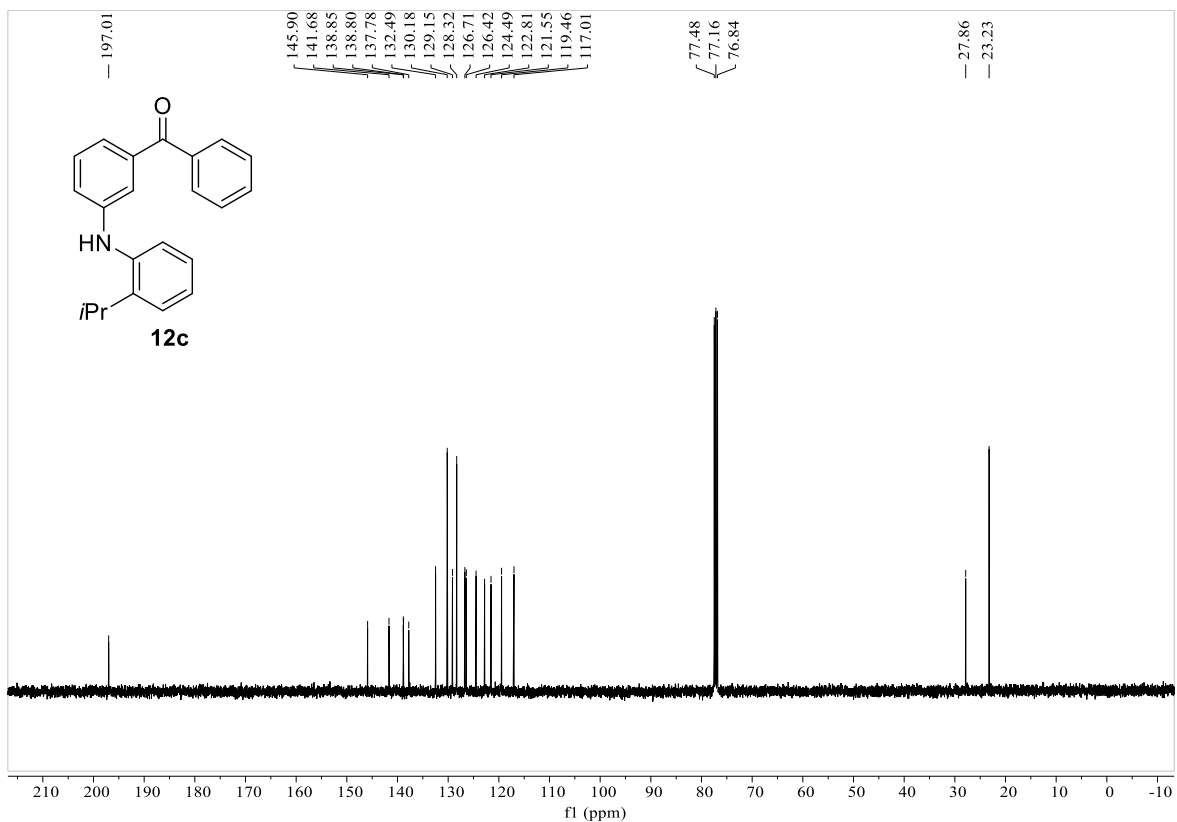
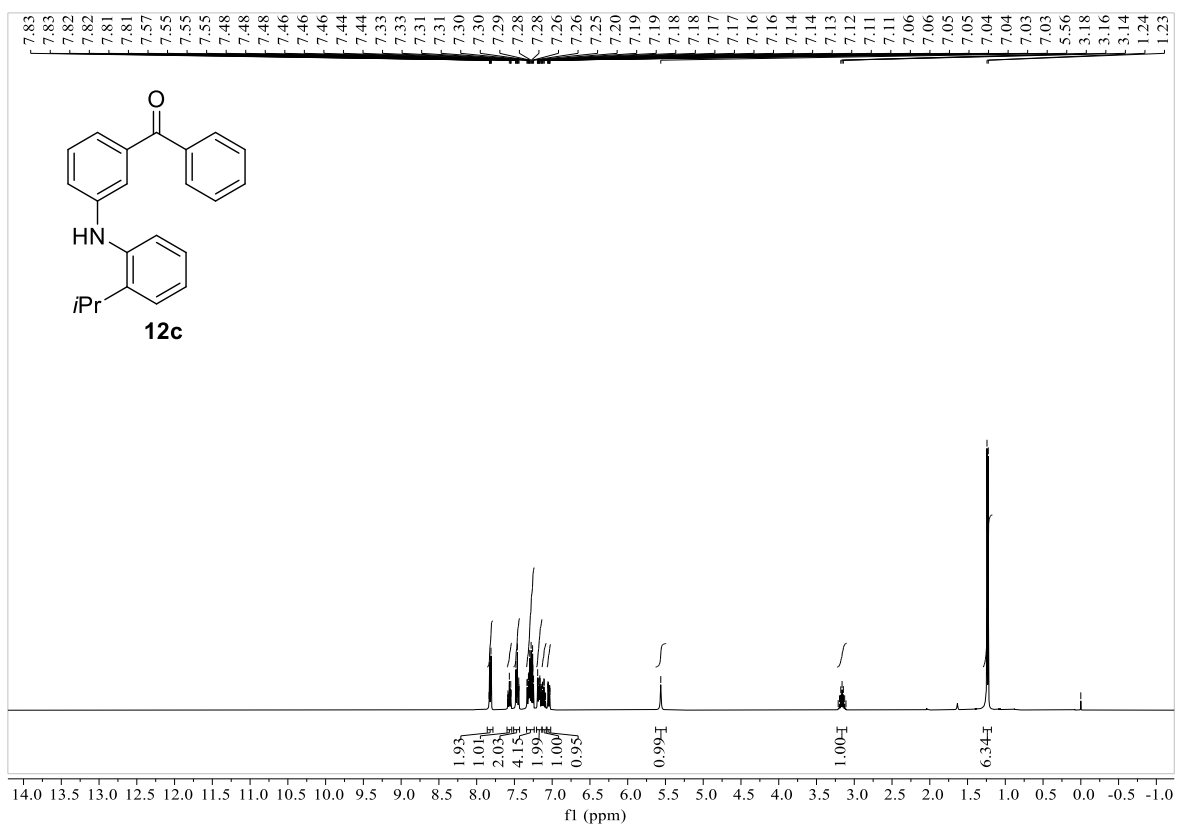
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12a**



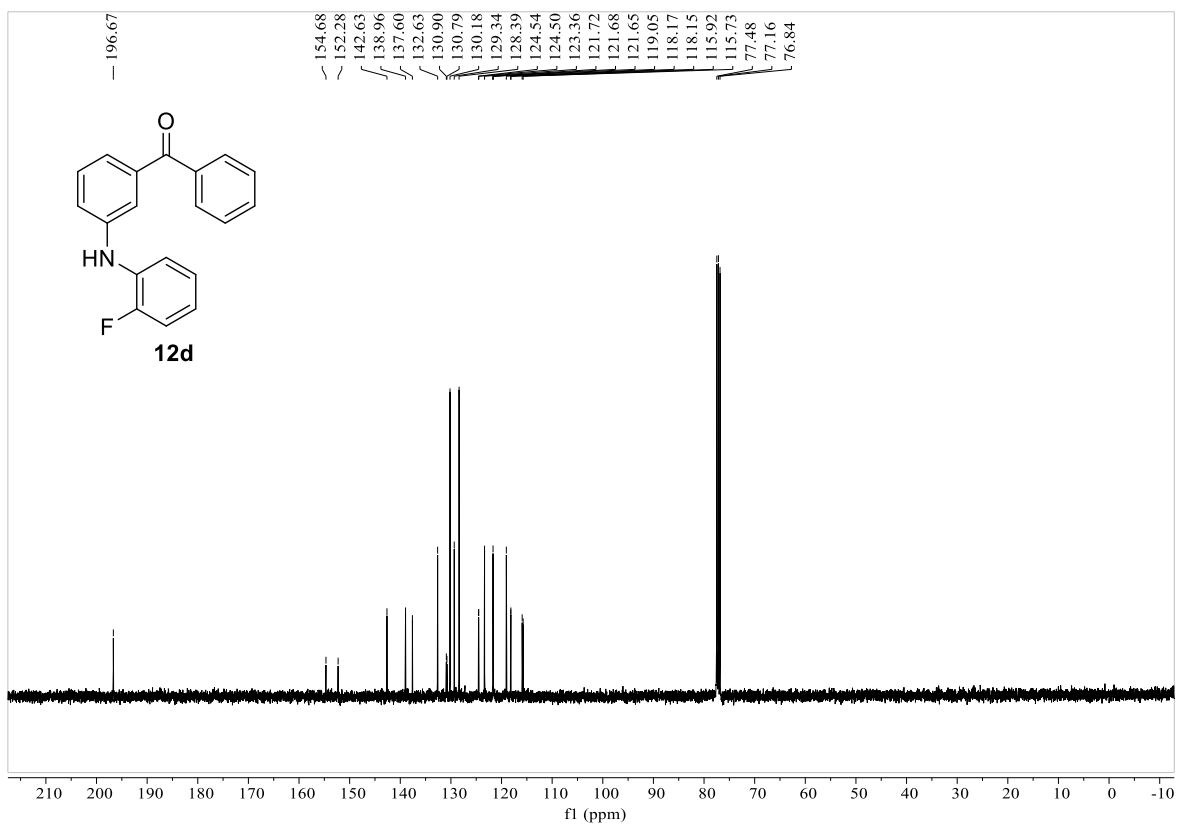
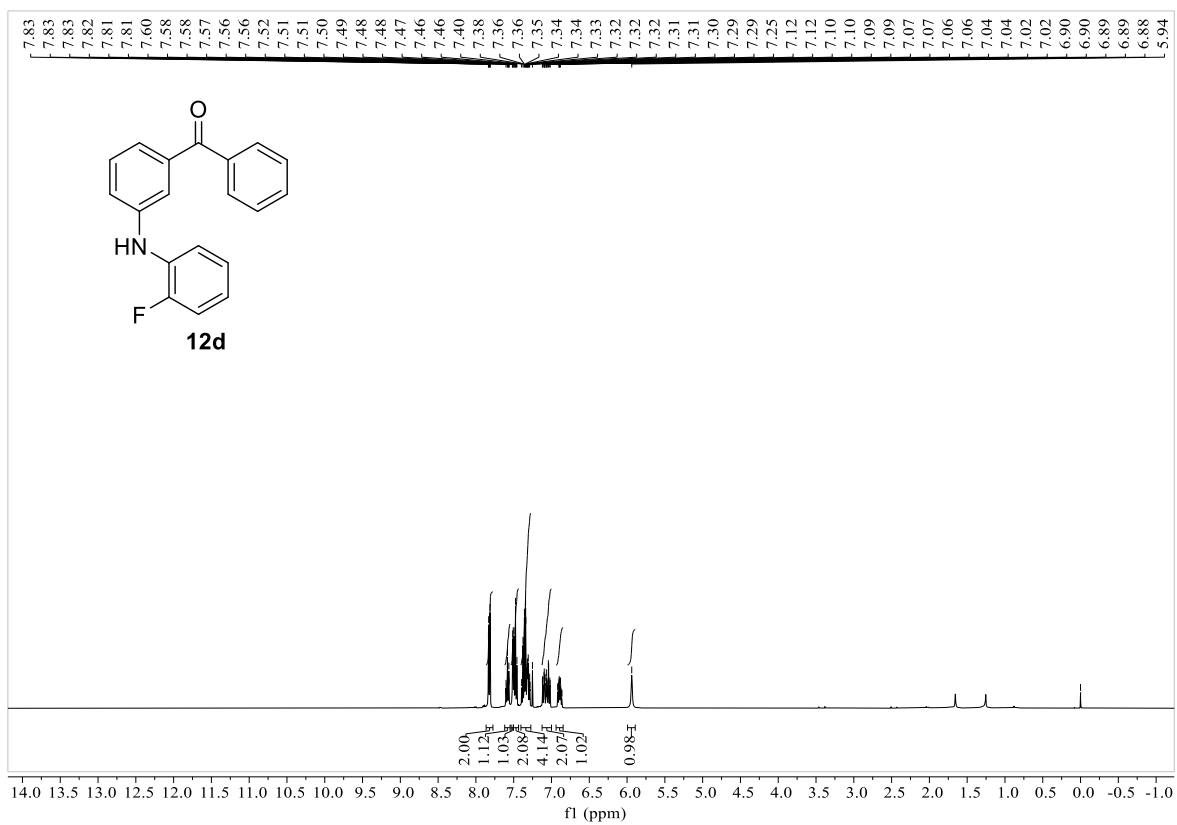
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12b**



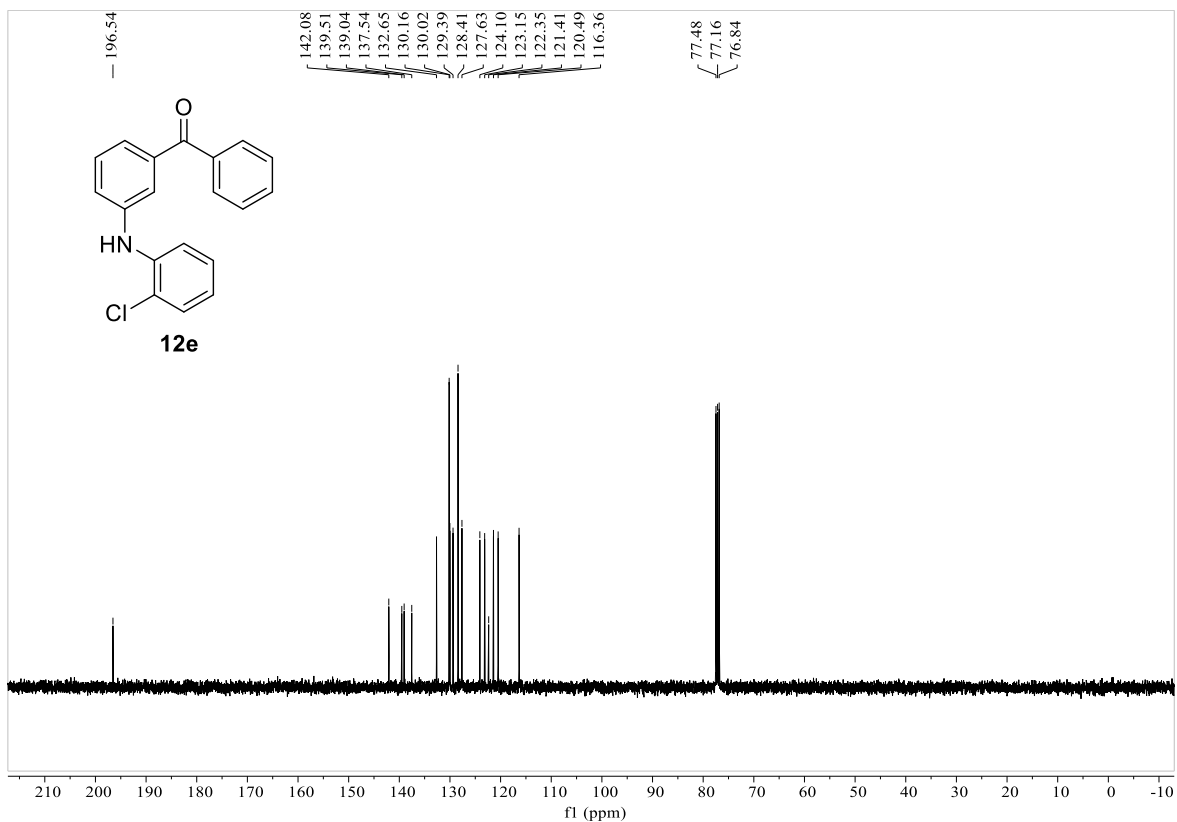
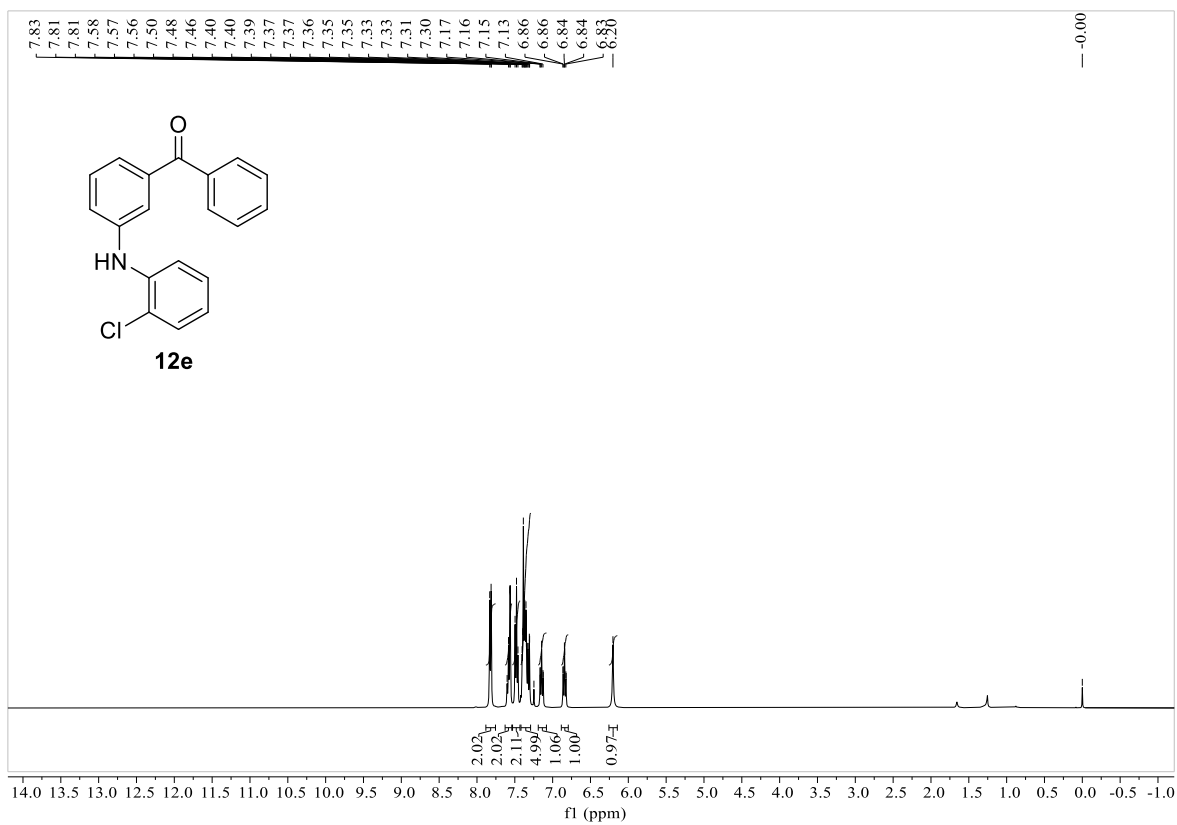
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12c**



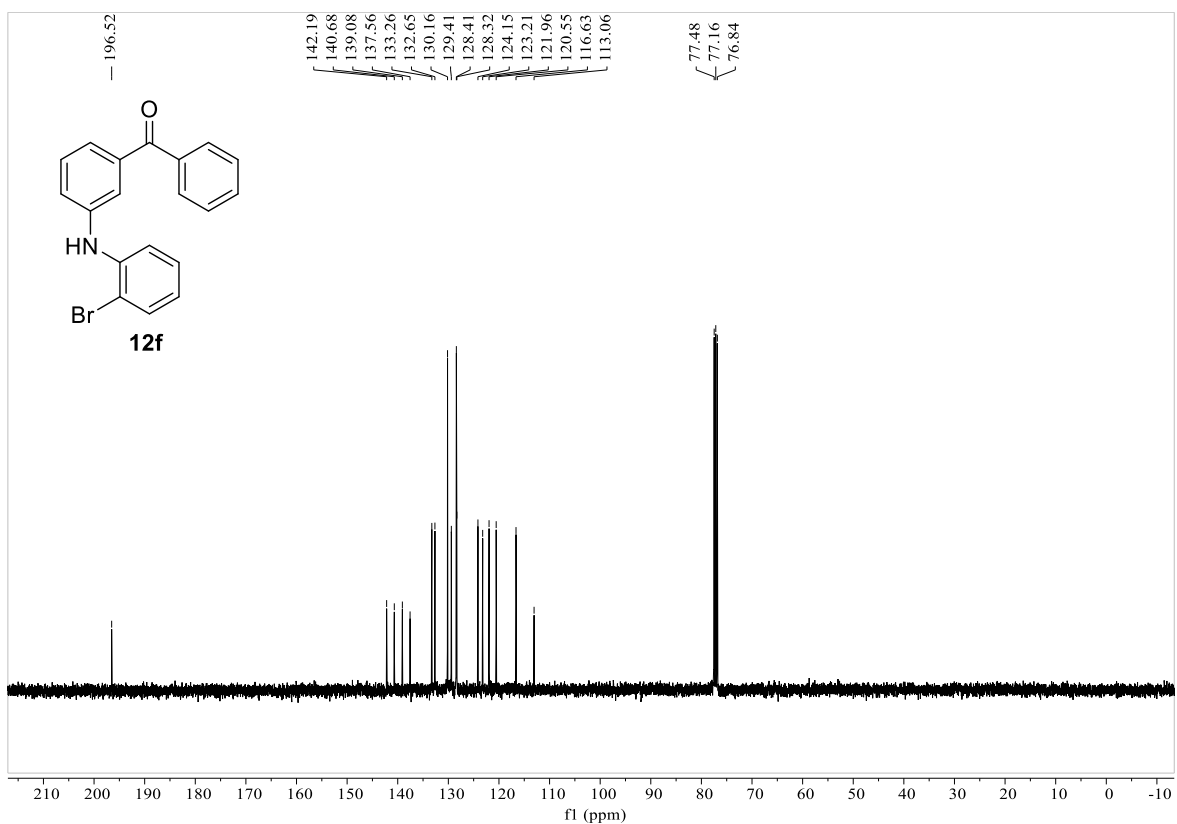
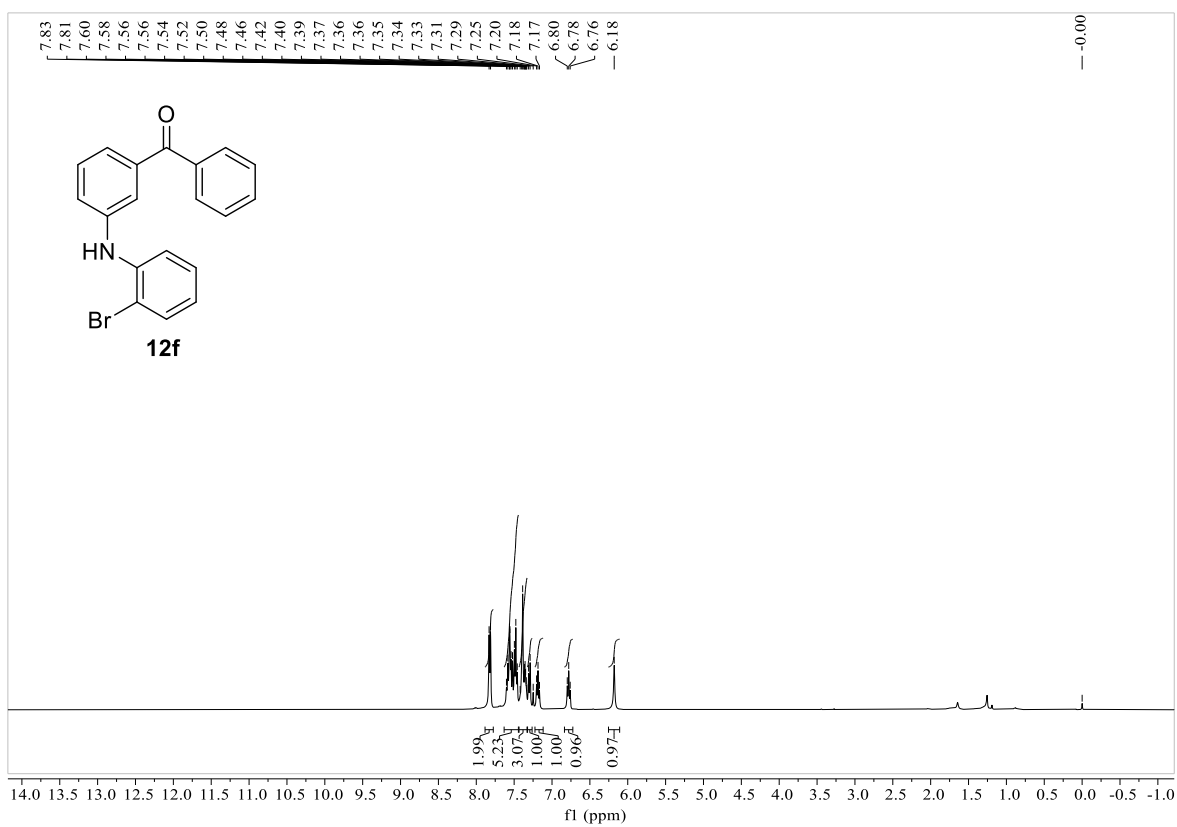
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12d**



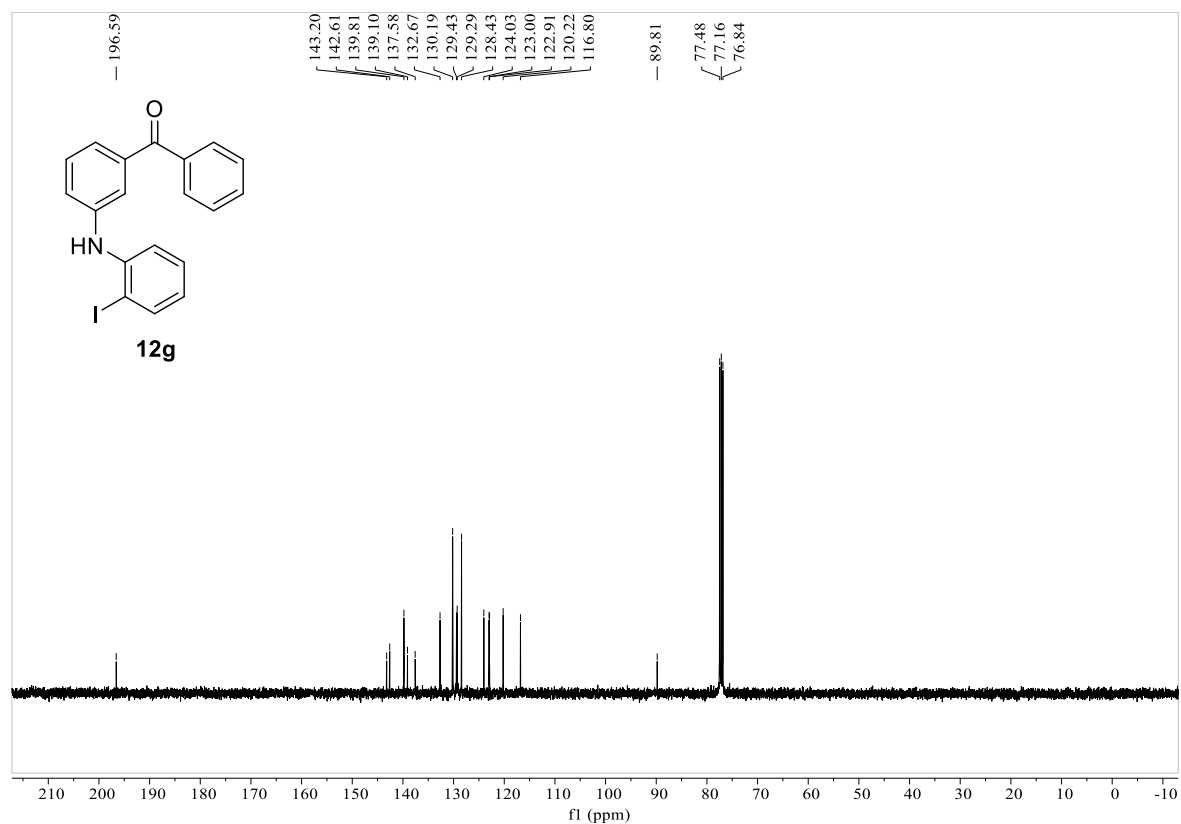
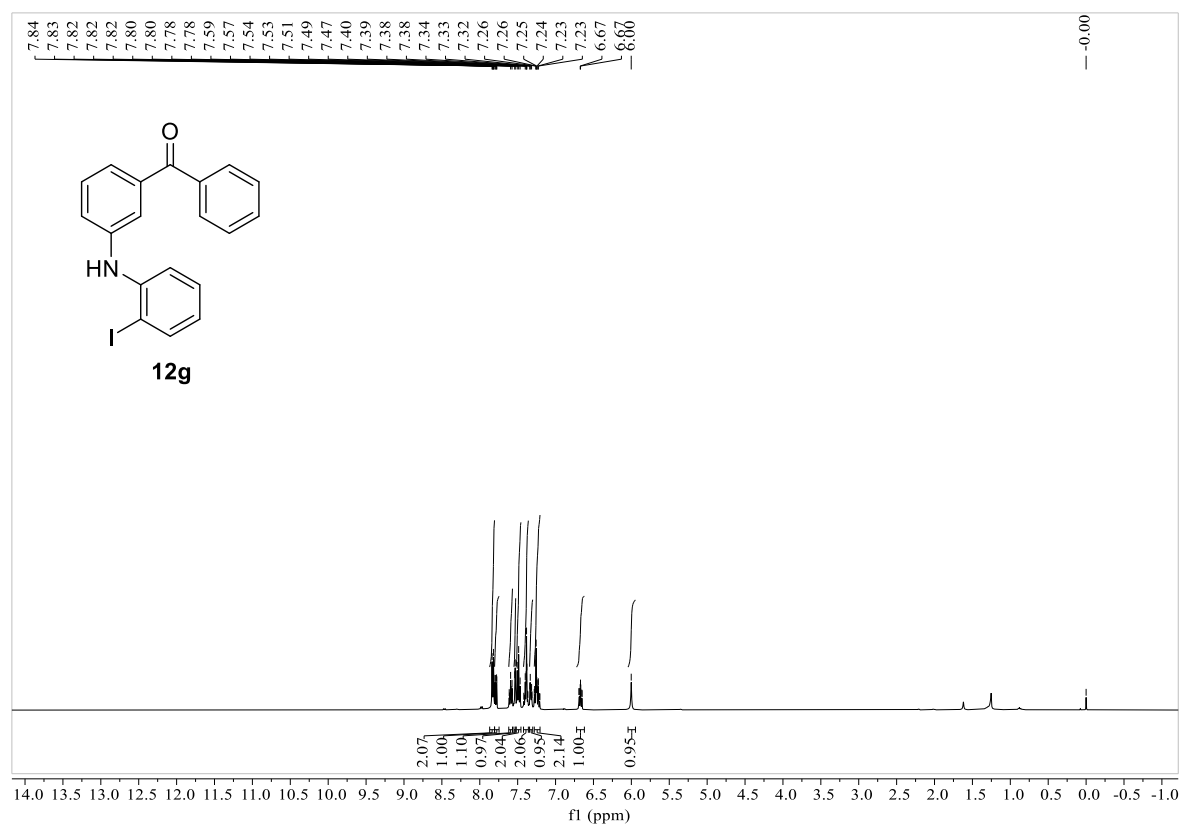
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12e**



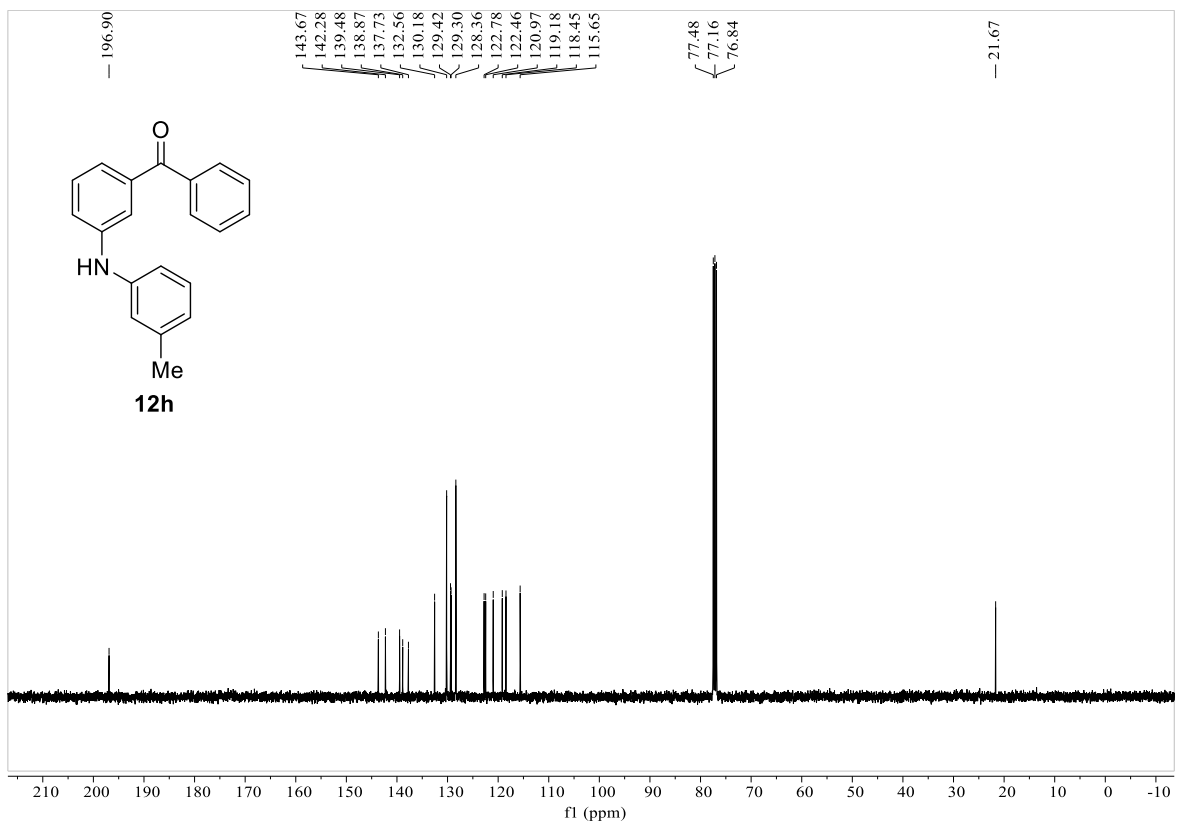
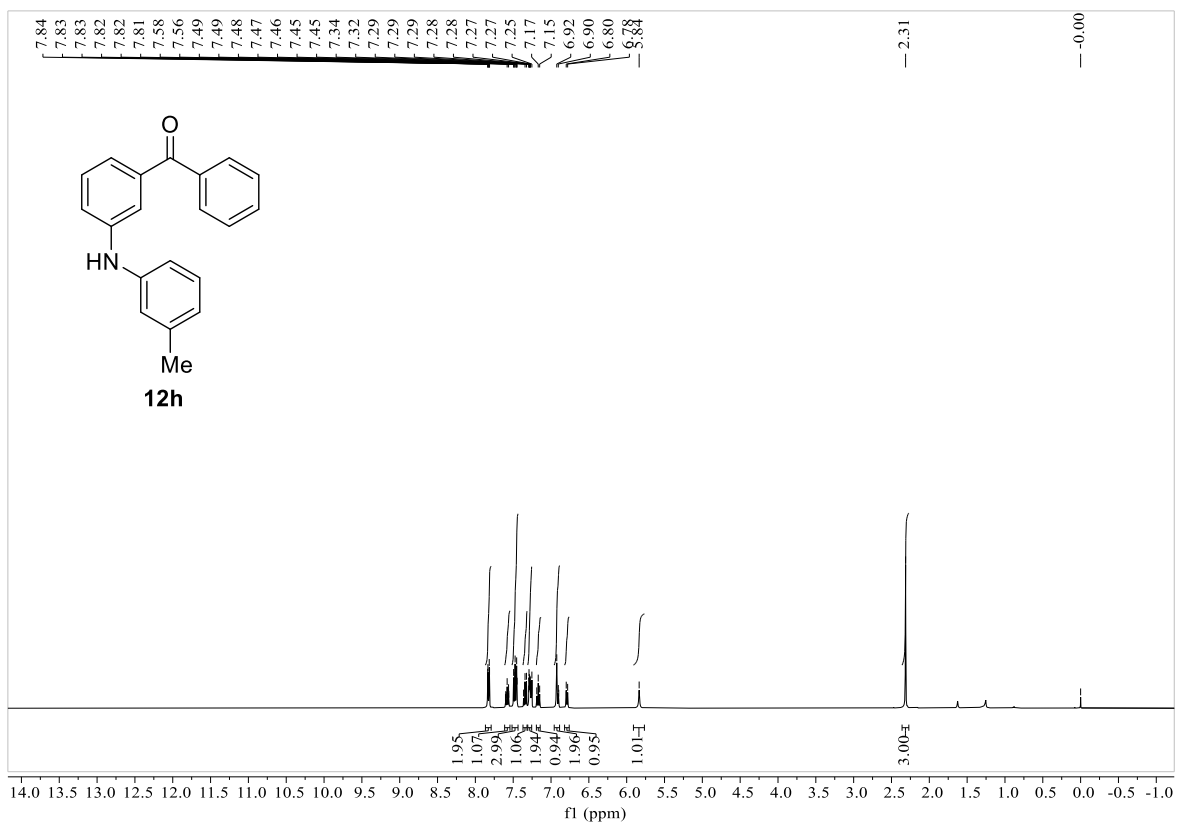
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12f**



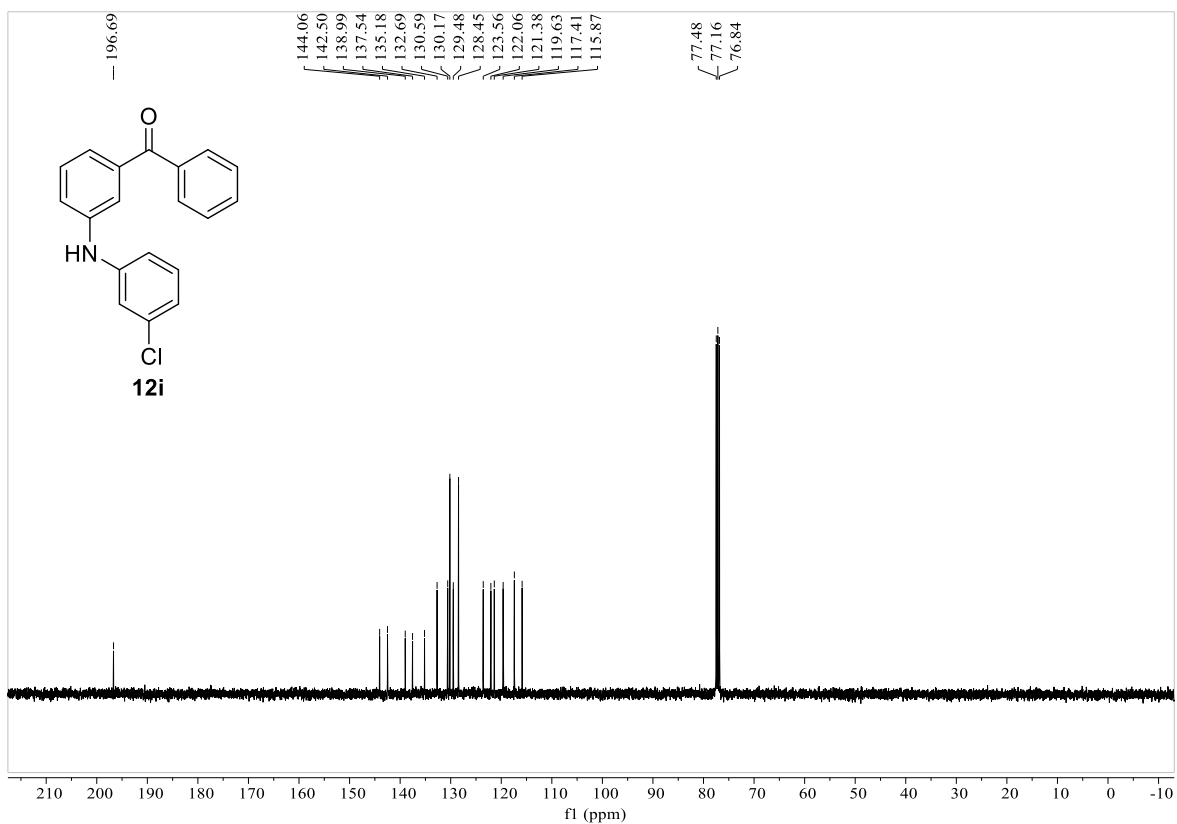
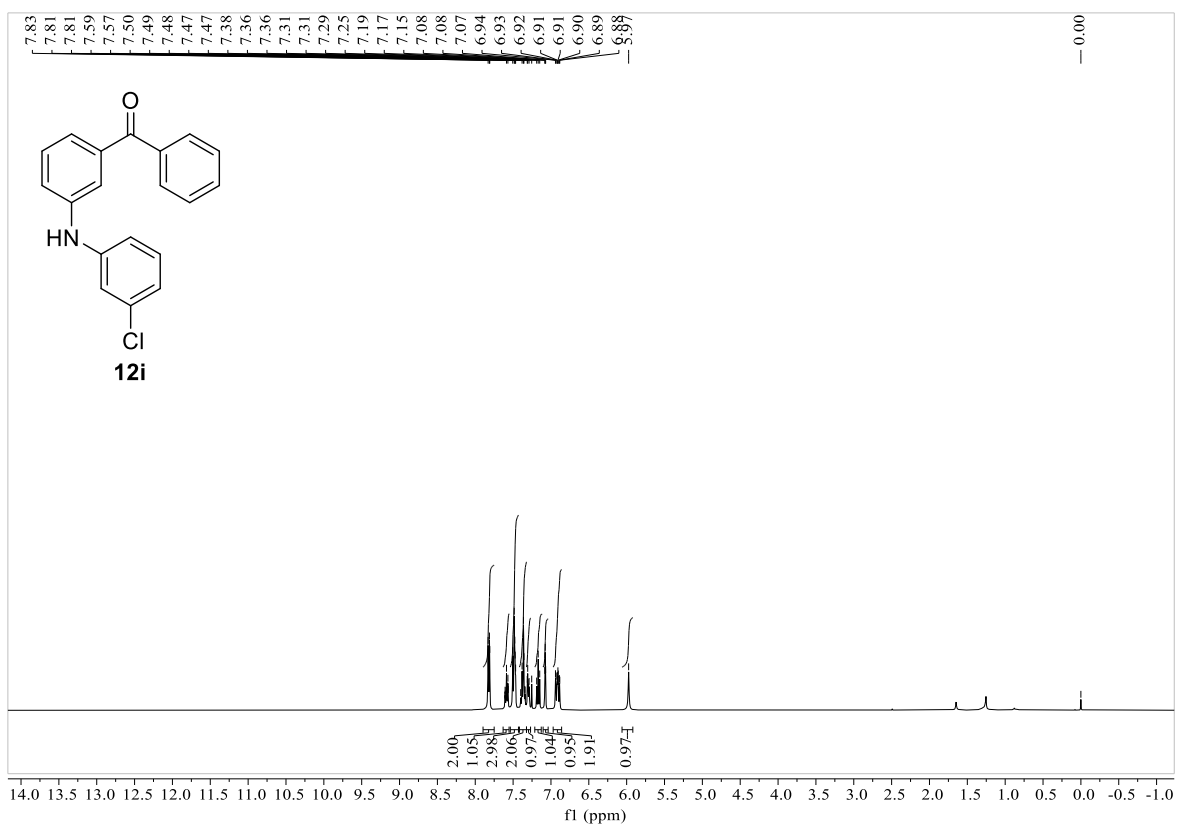
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12g**



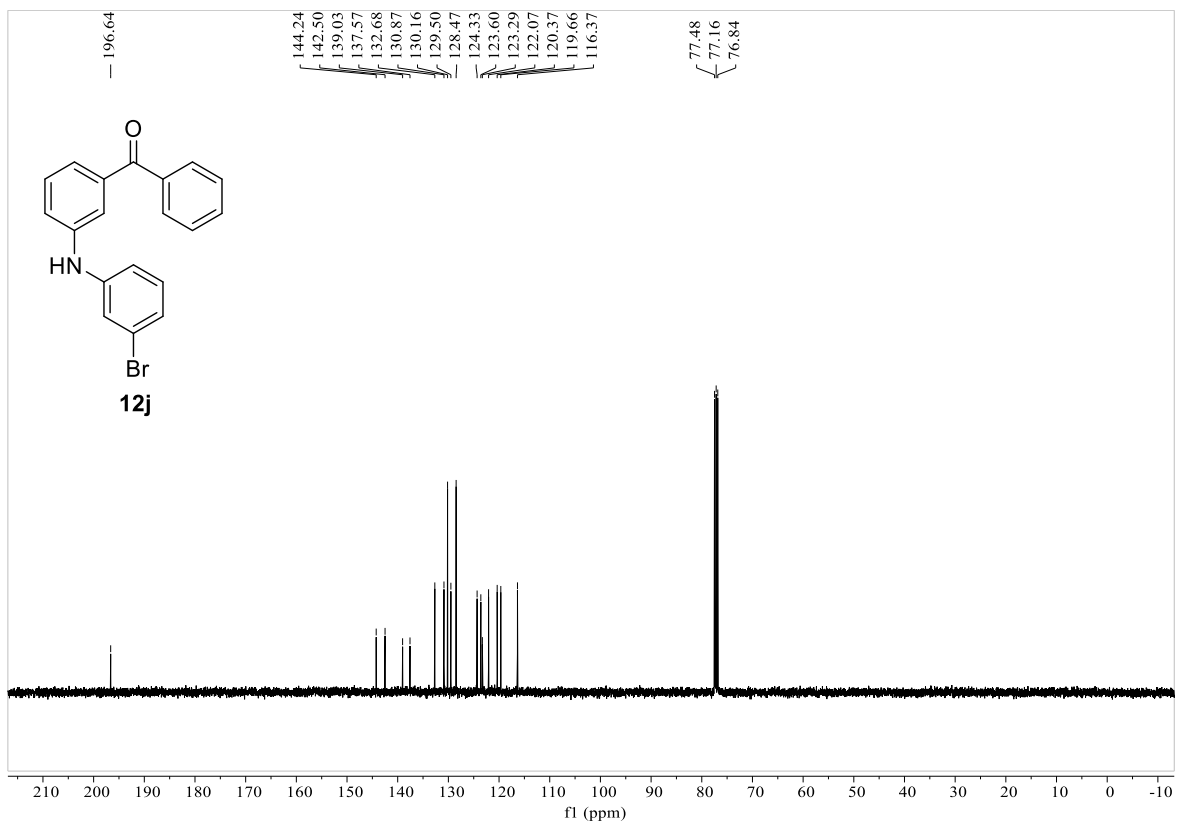
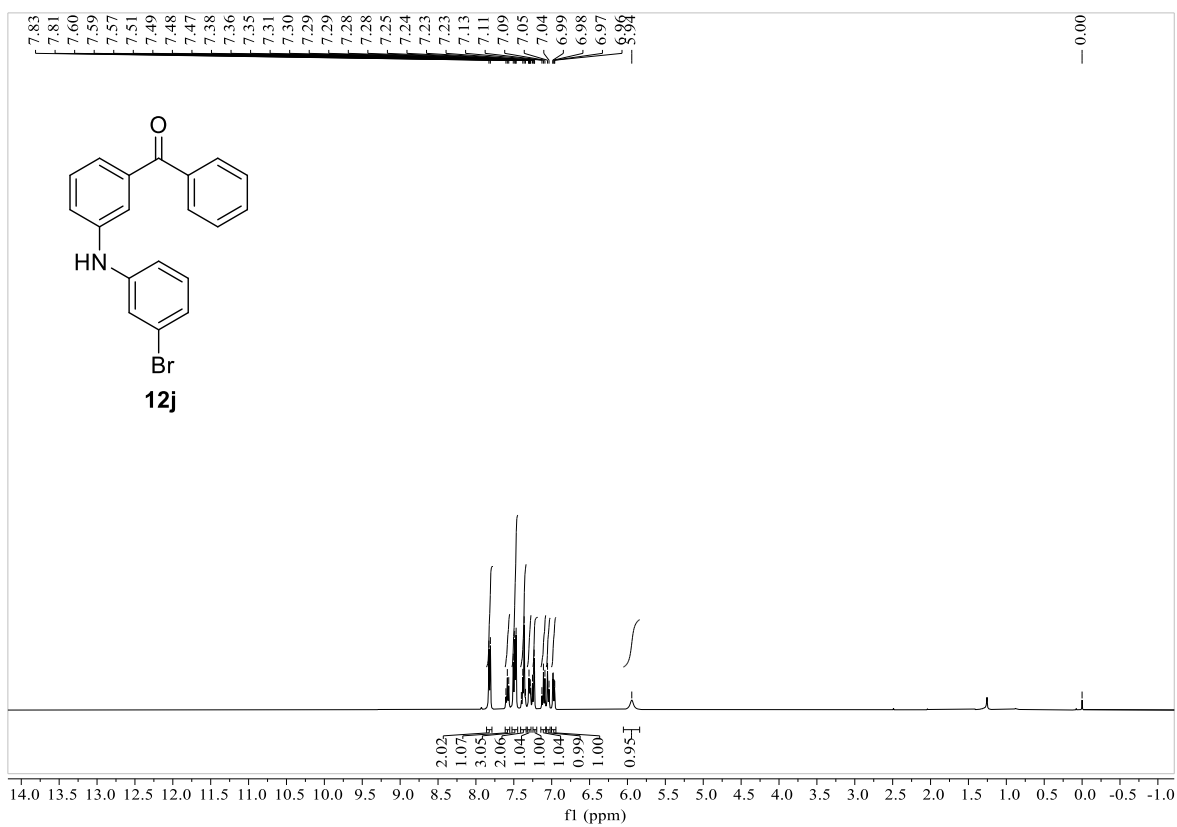
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12h**



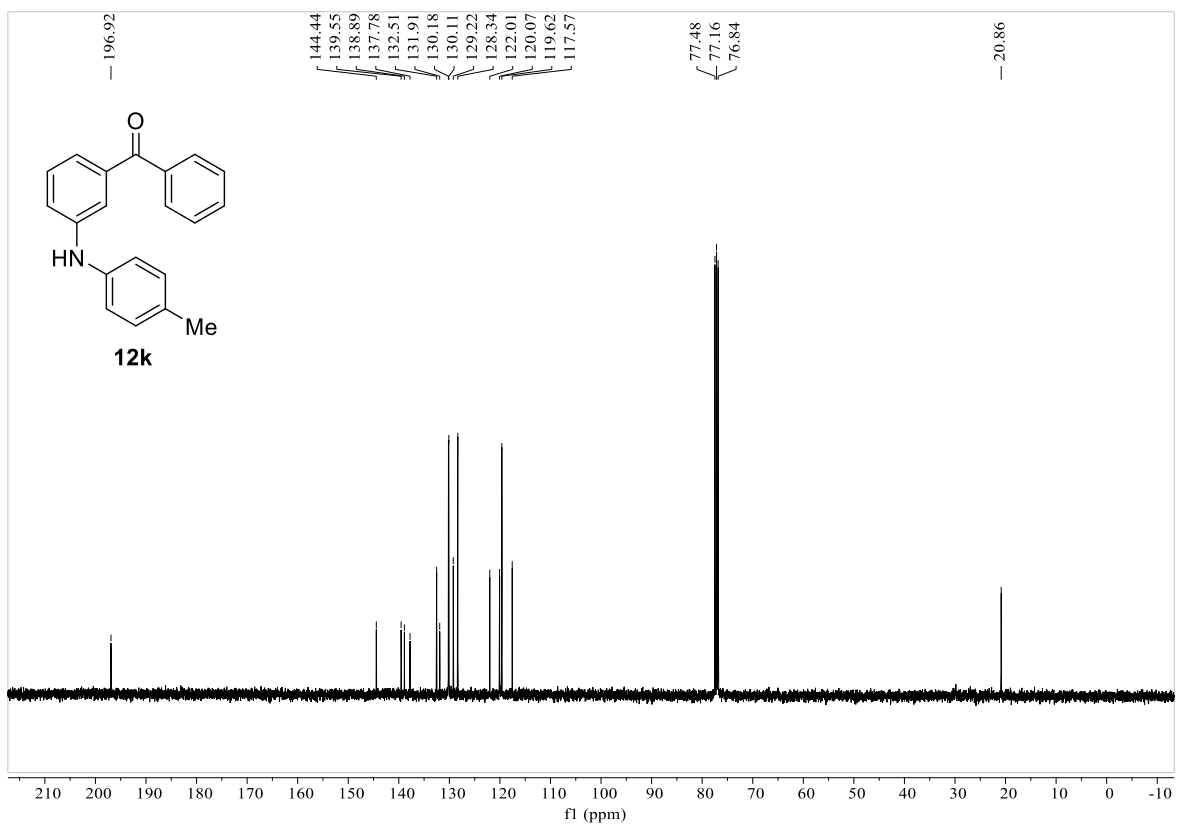
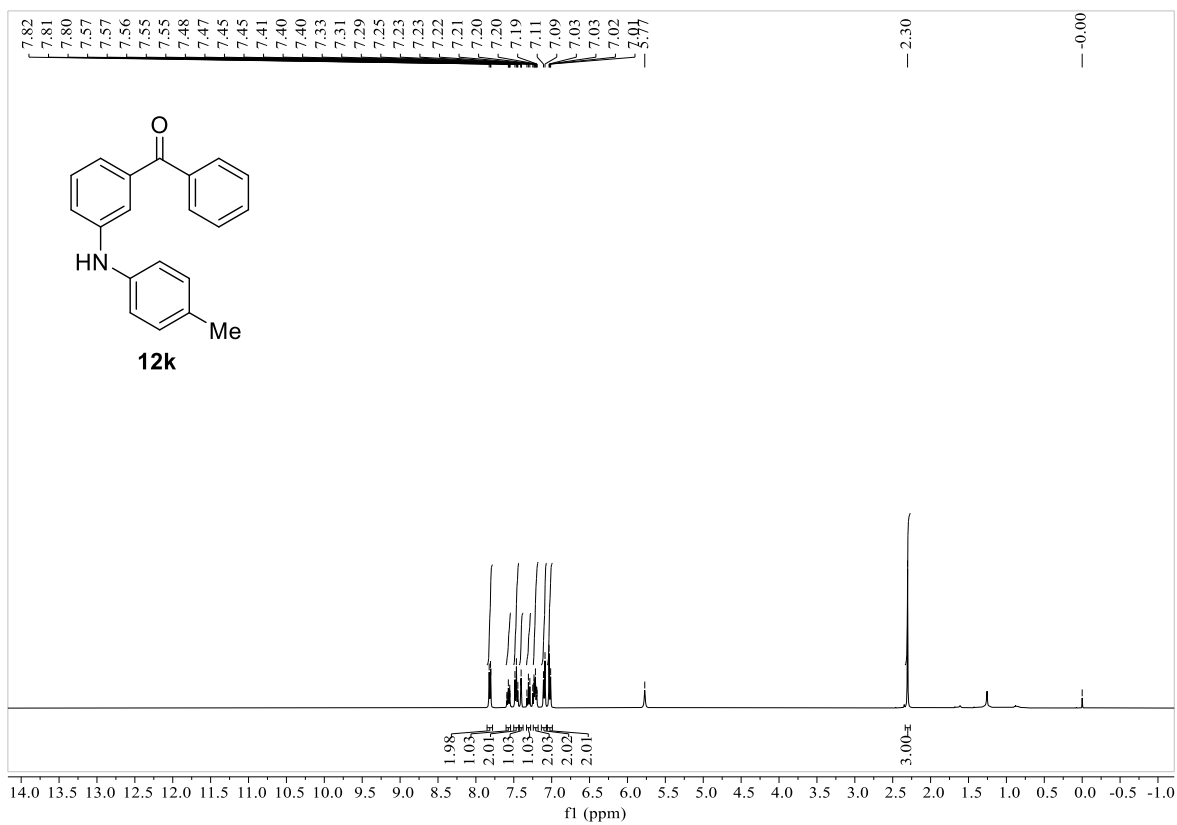
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12i**



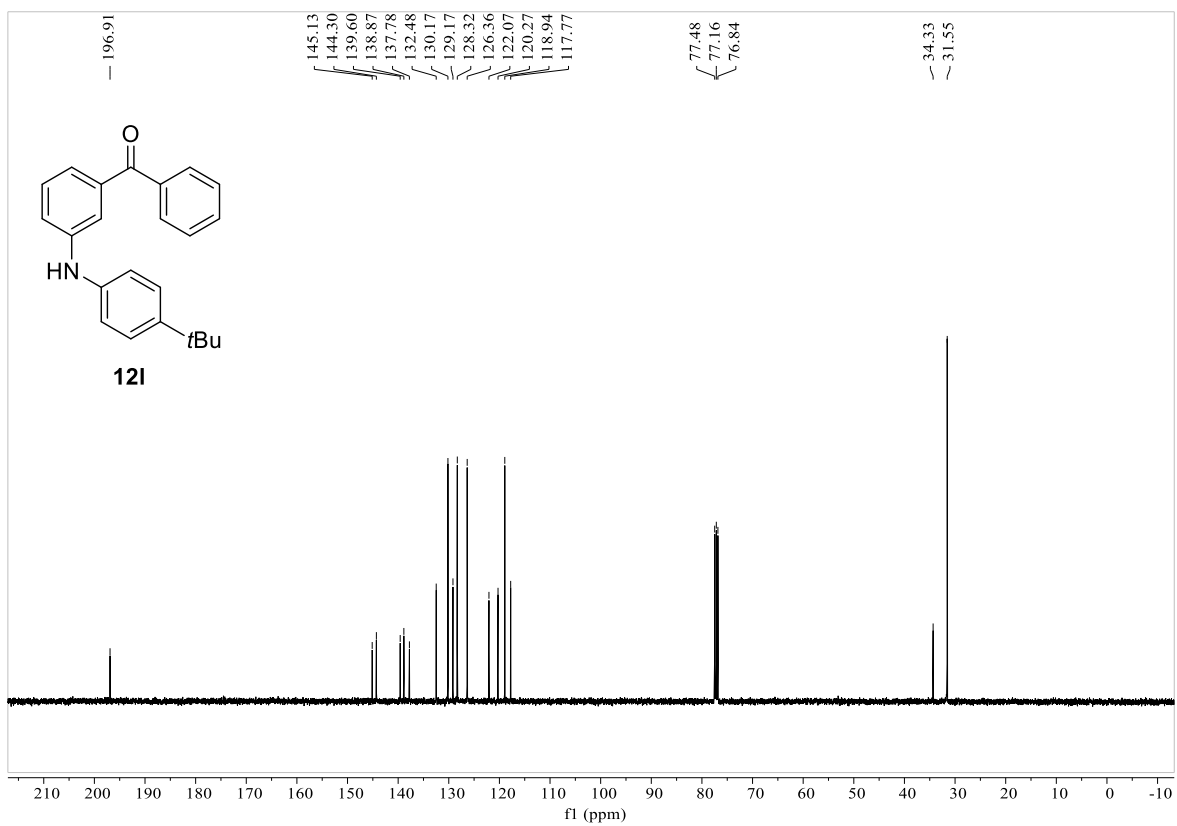
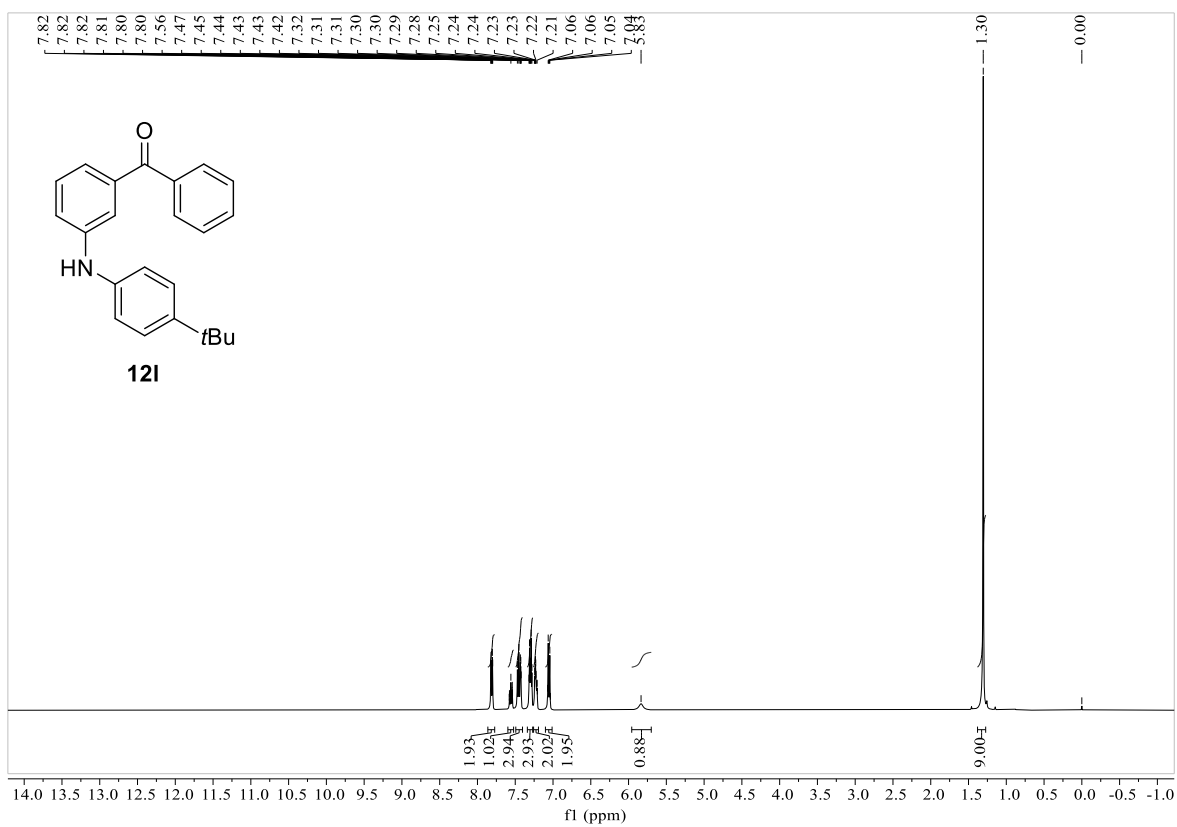
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12j**



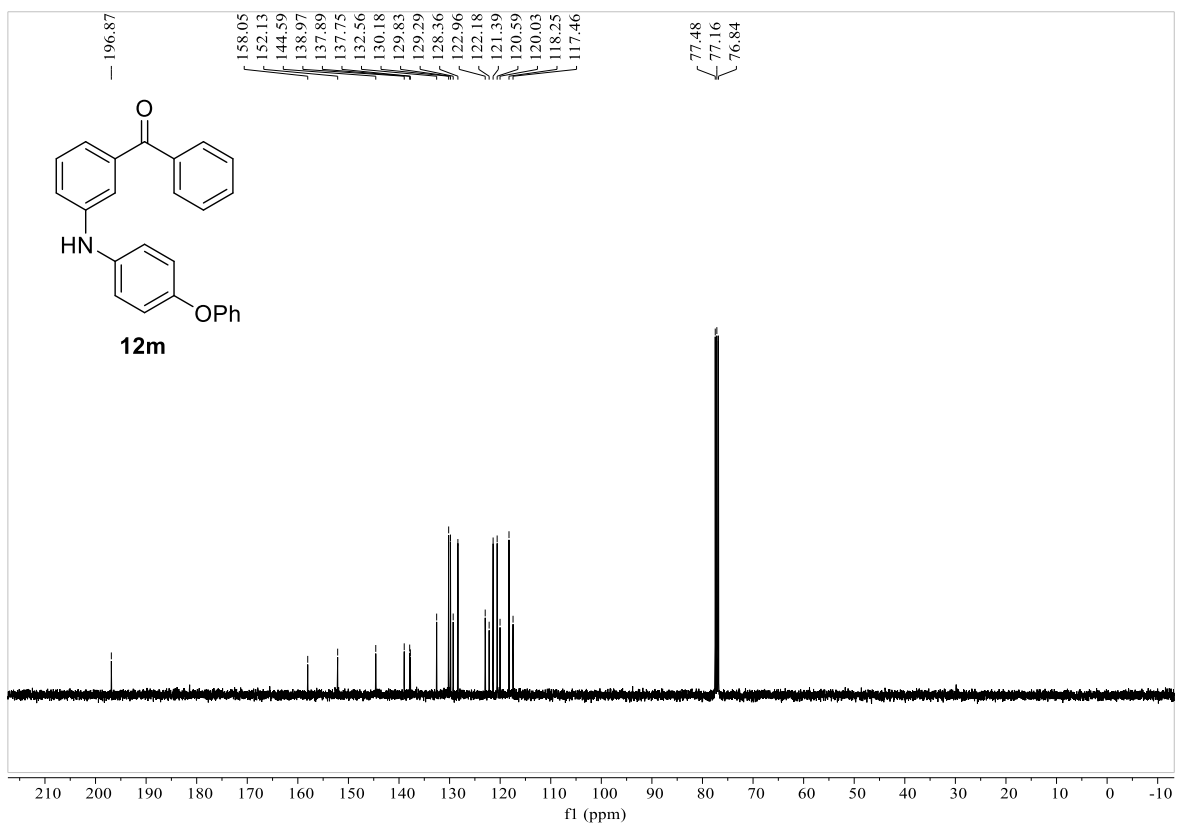
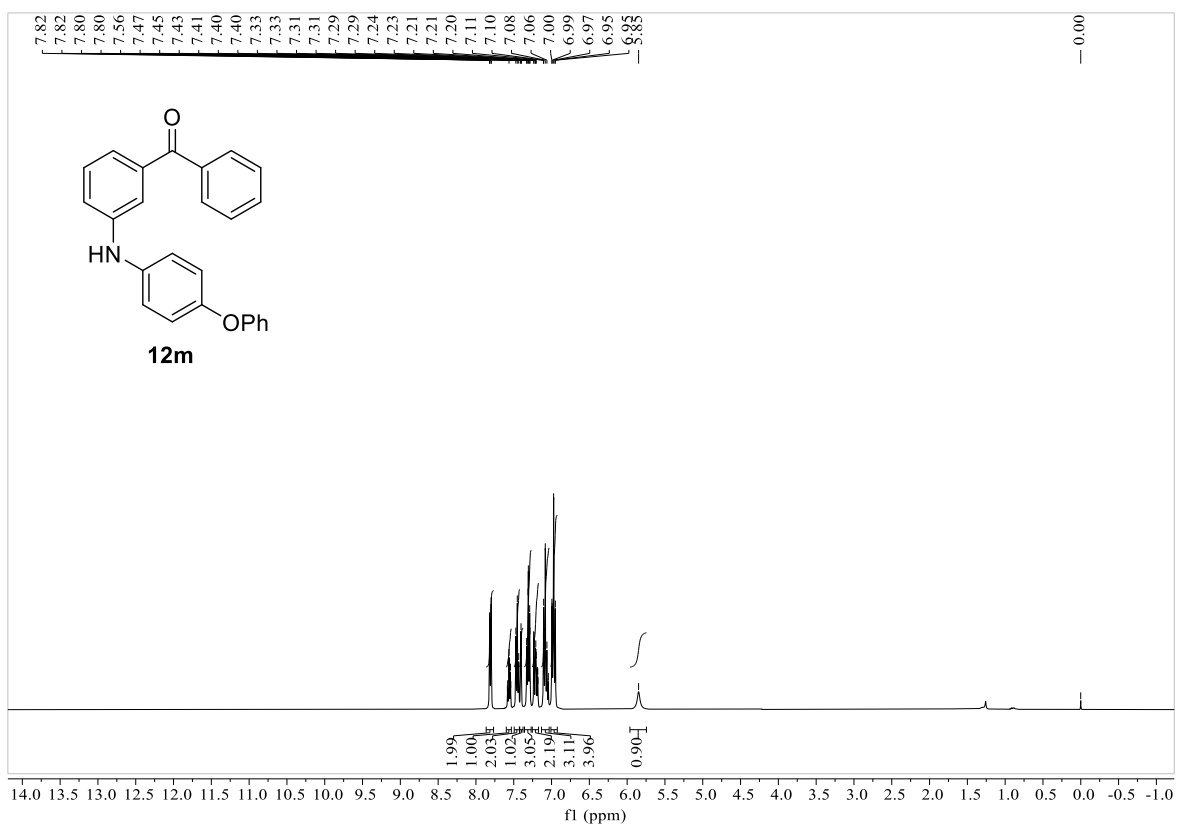
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12k**



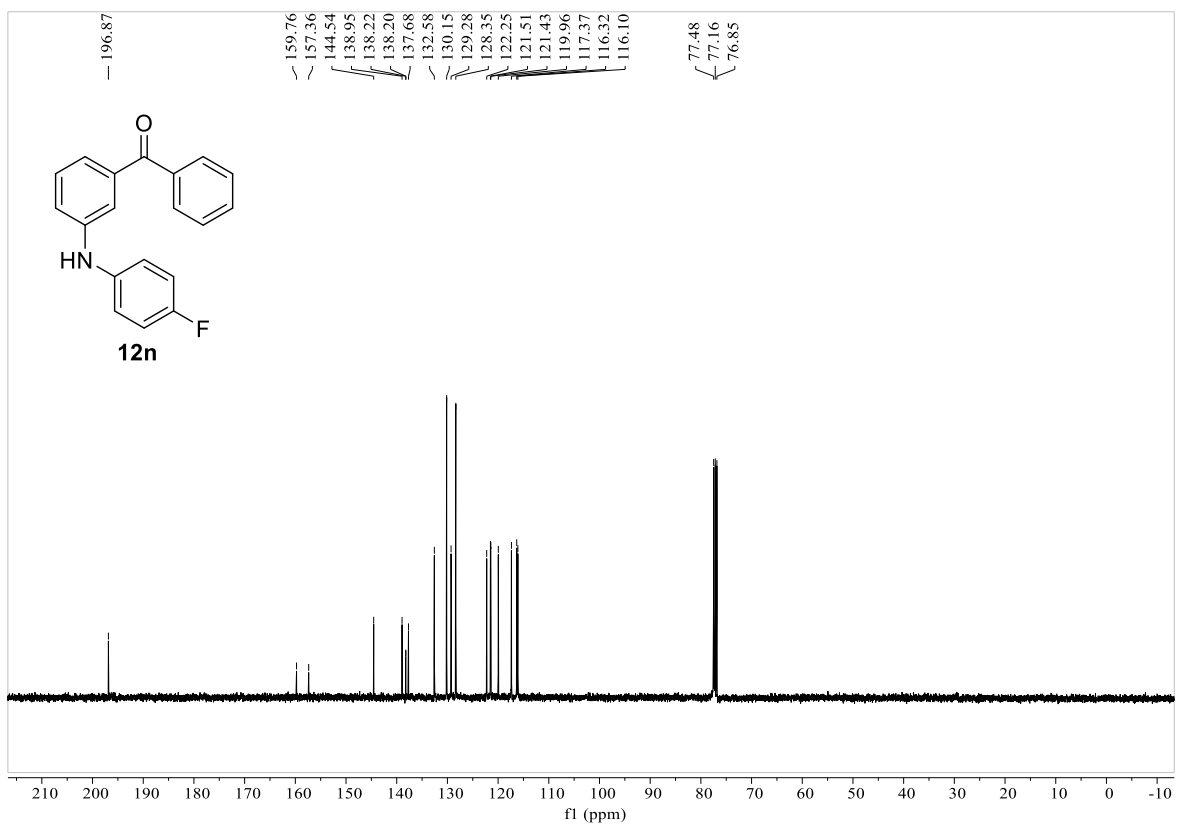
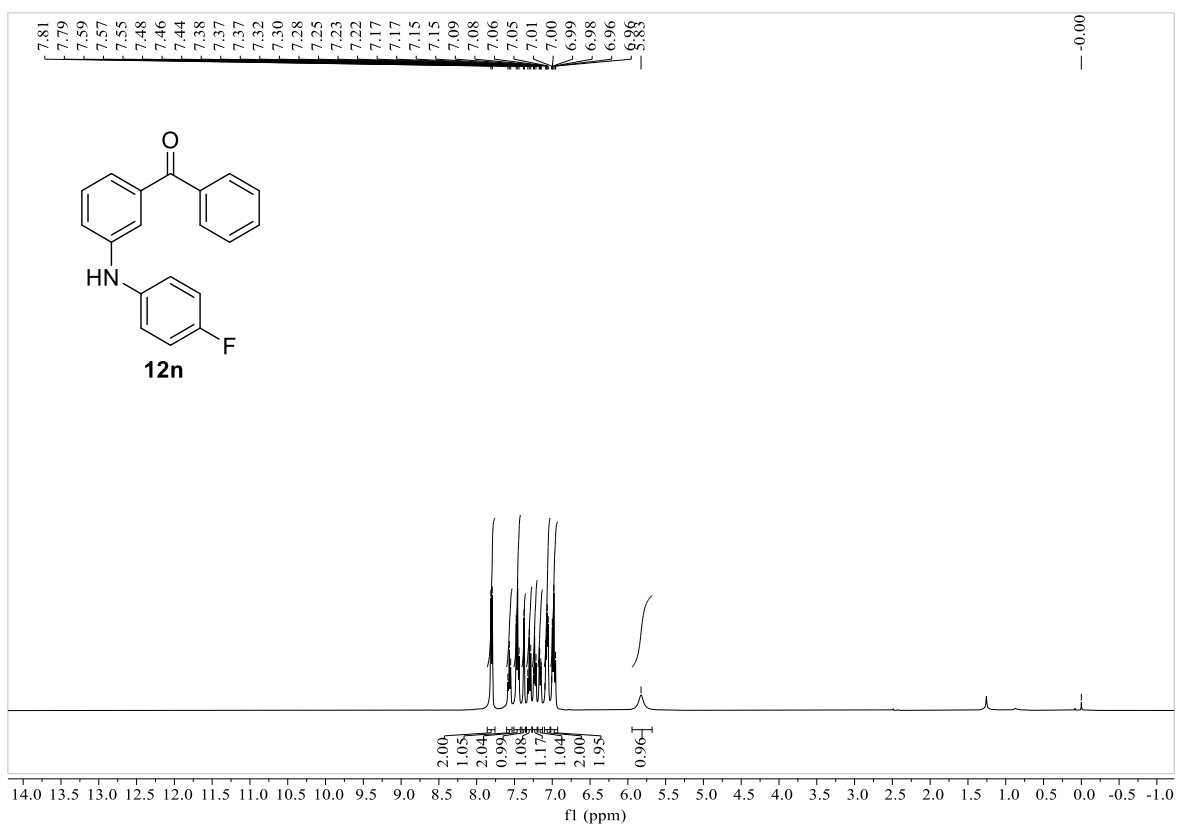
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12l**



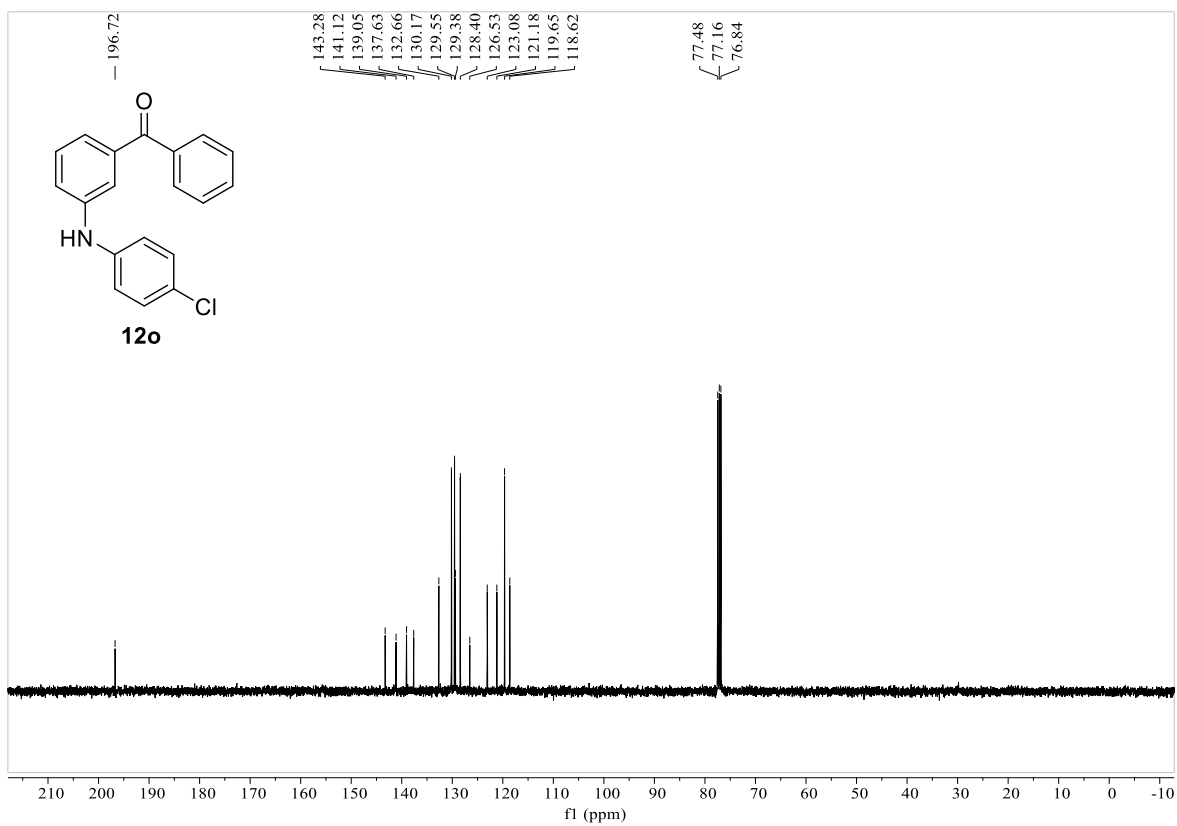
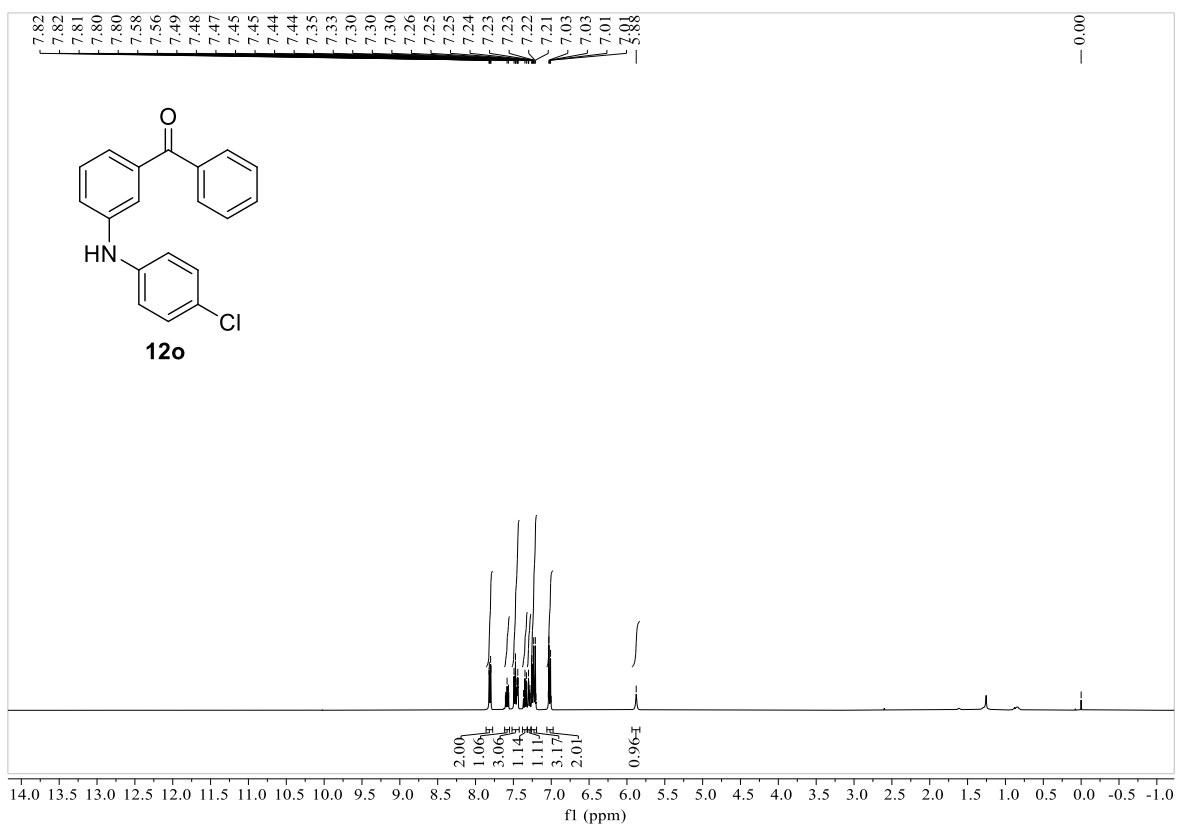
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12m**



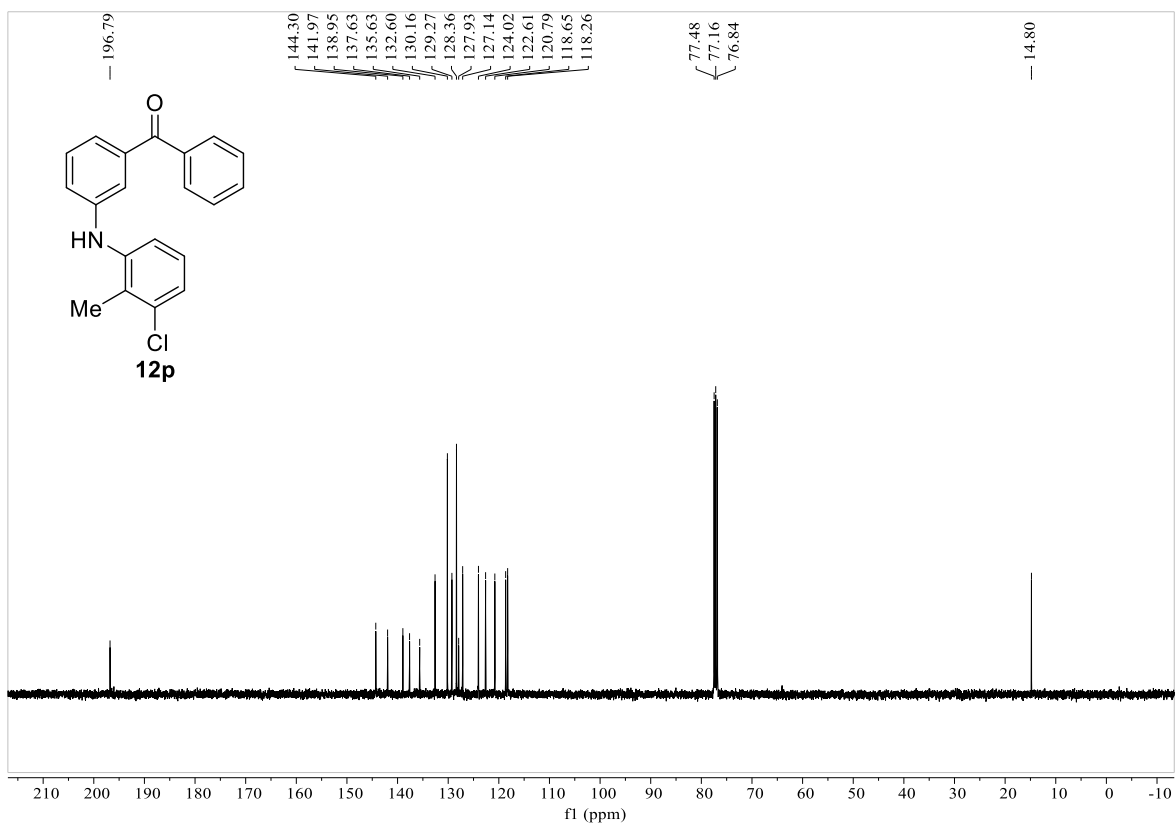
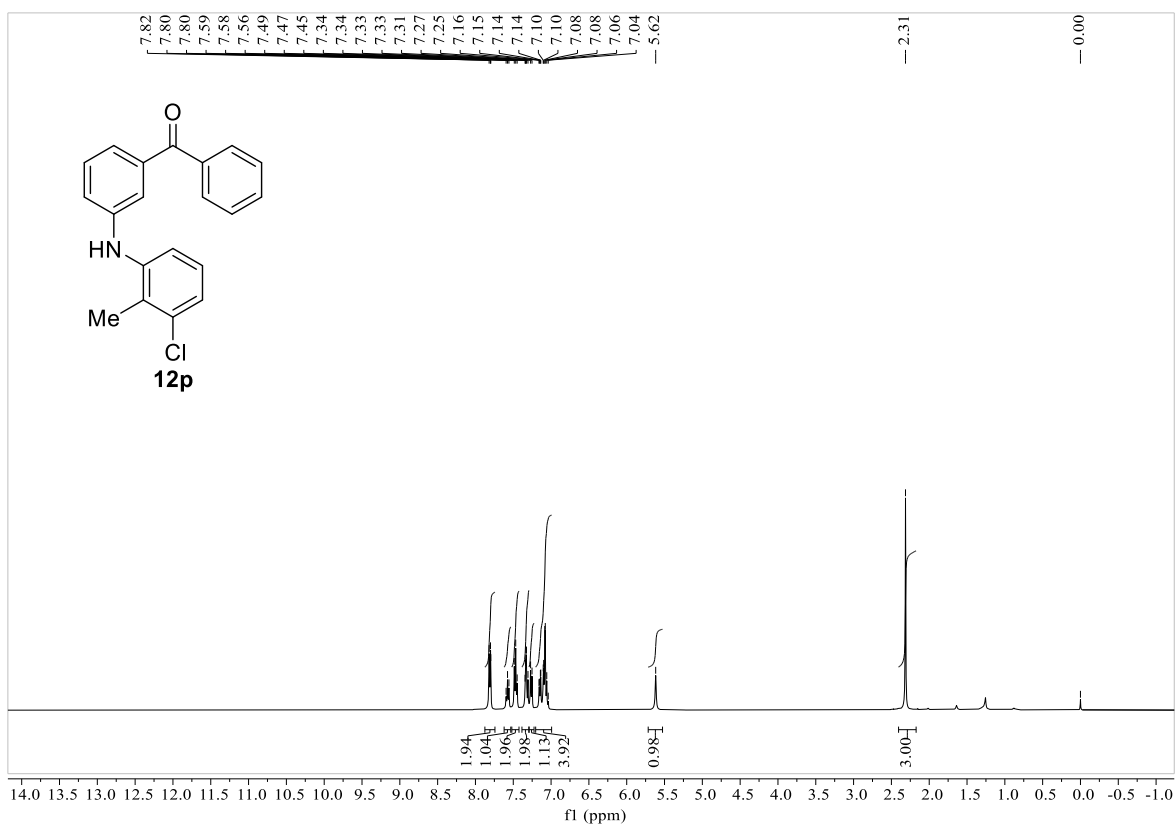
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12n**



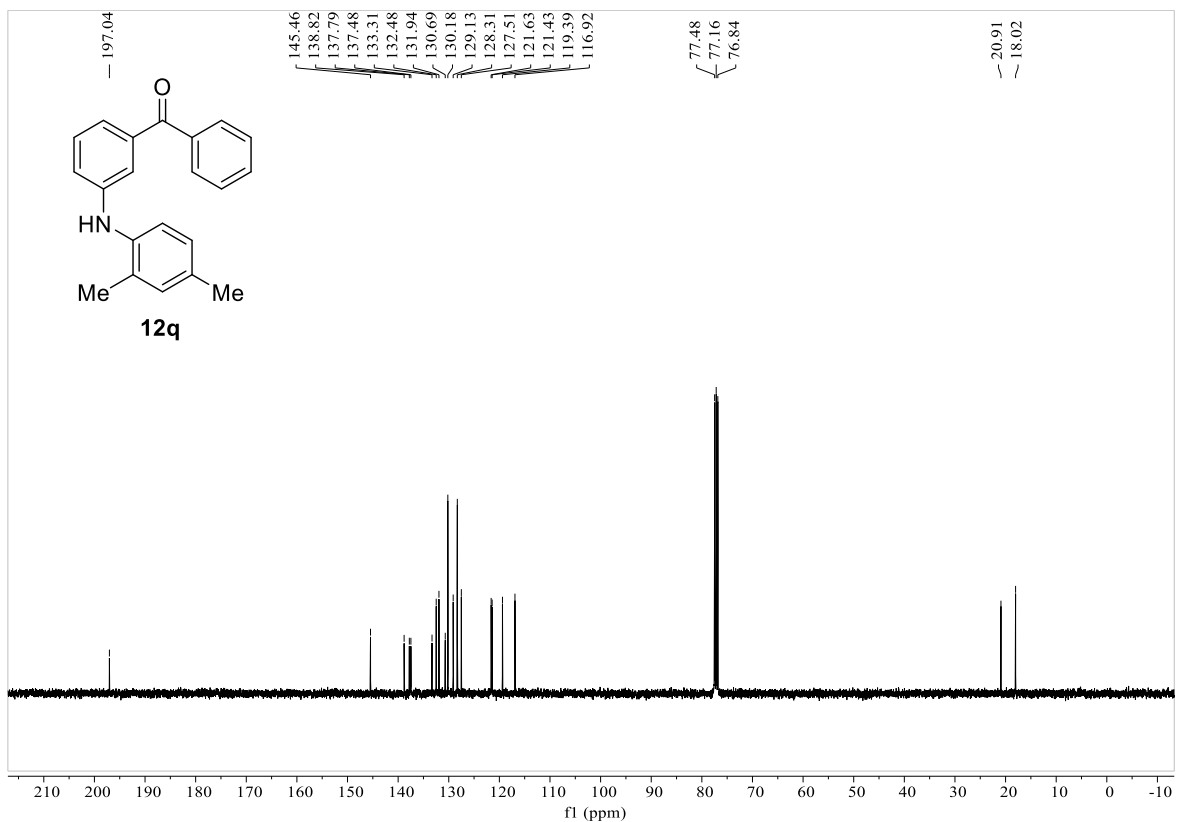
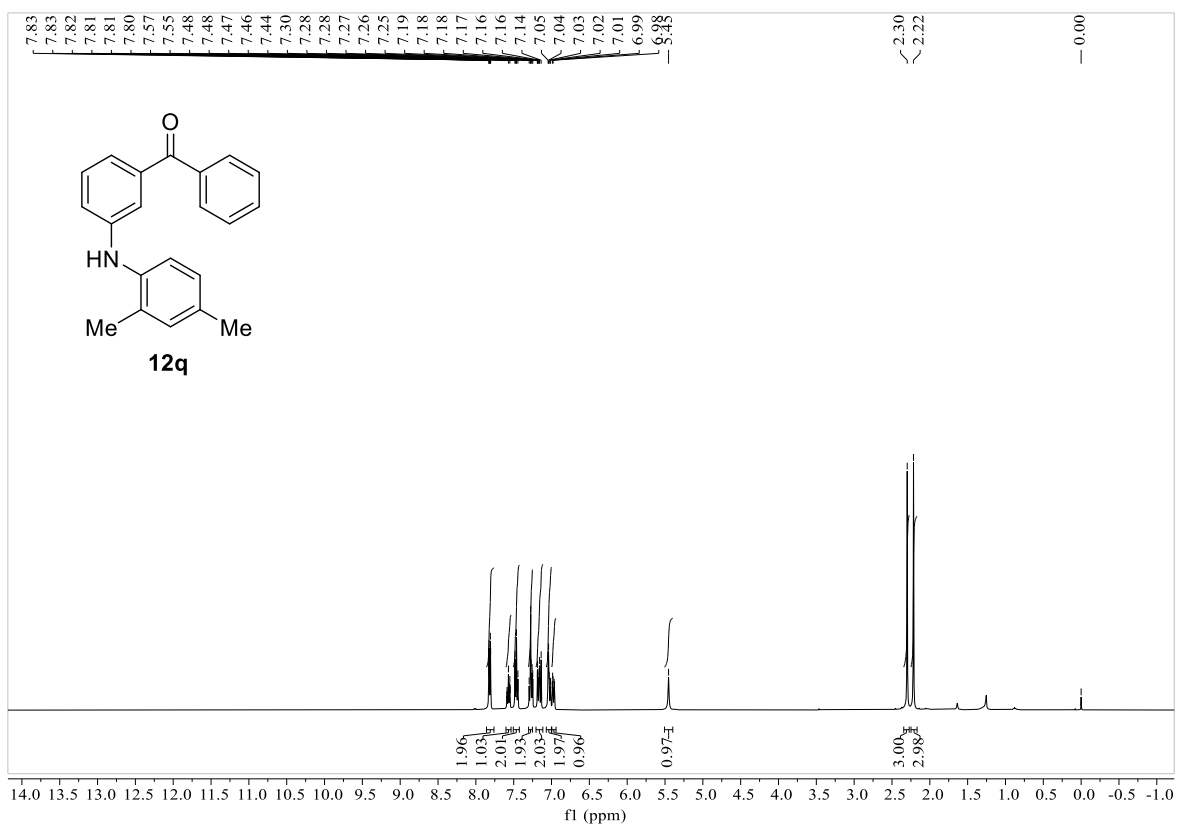
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12o**



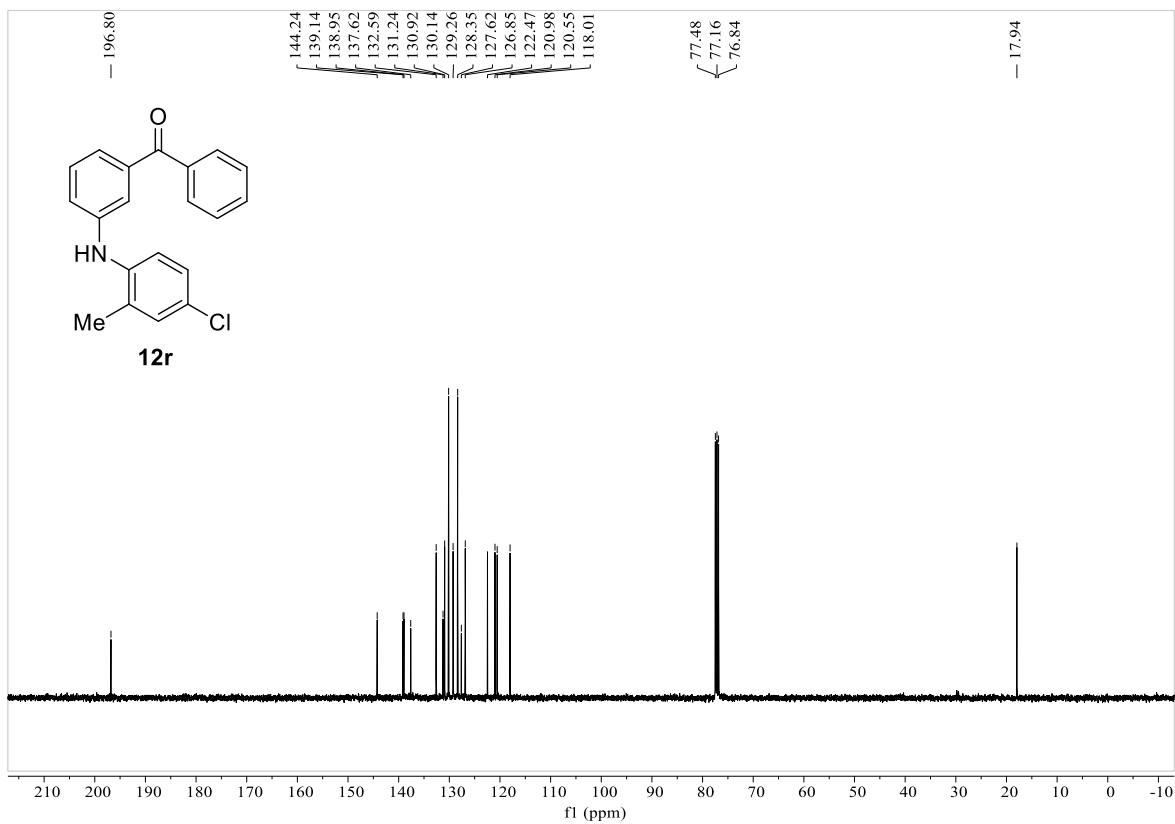
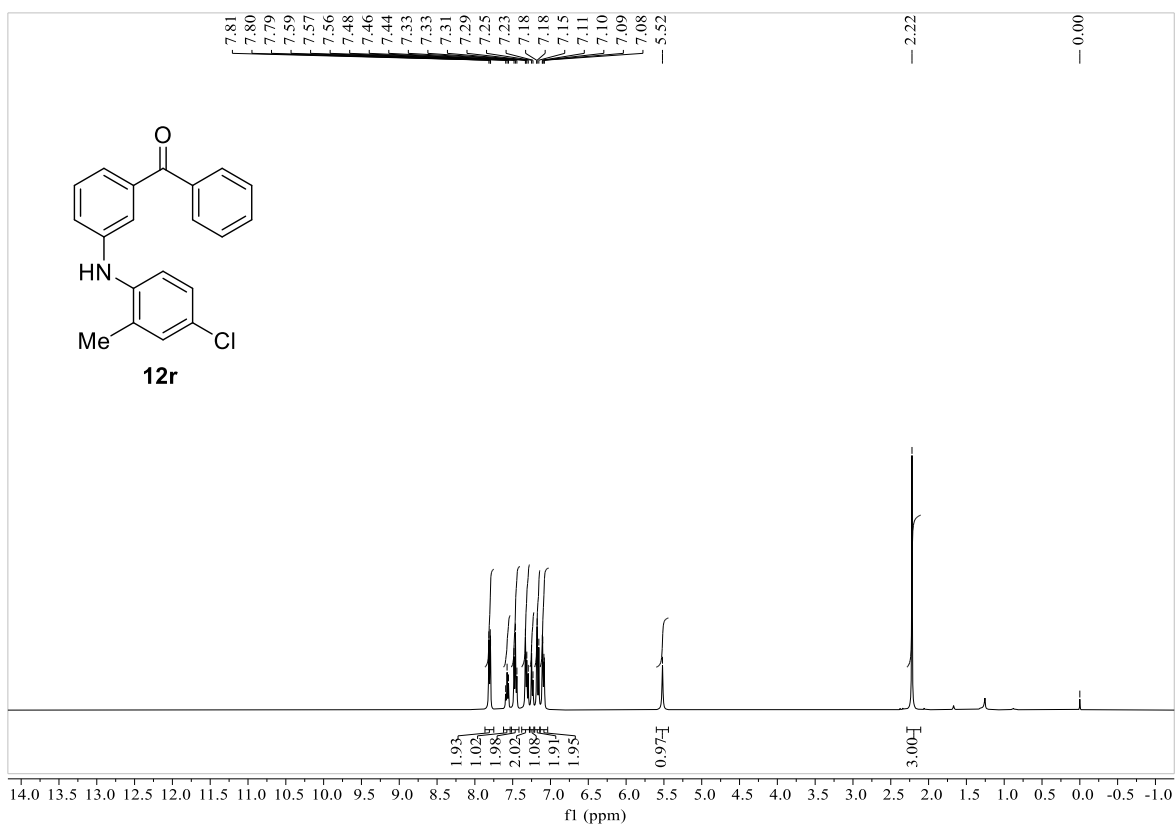
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12p**



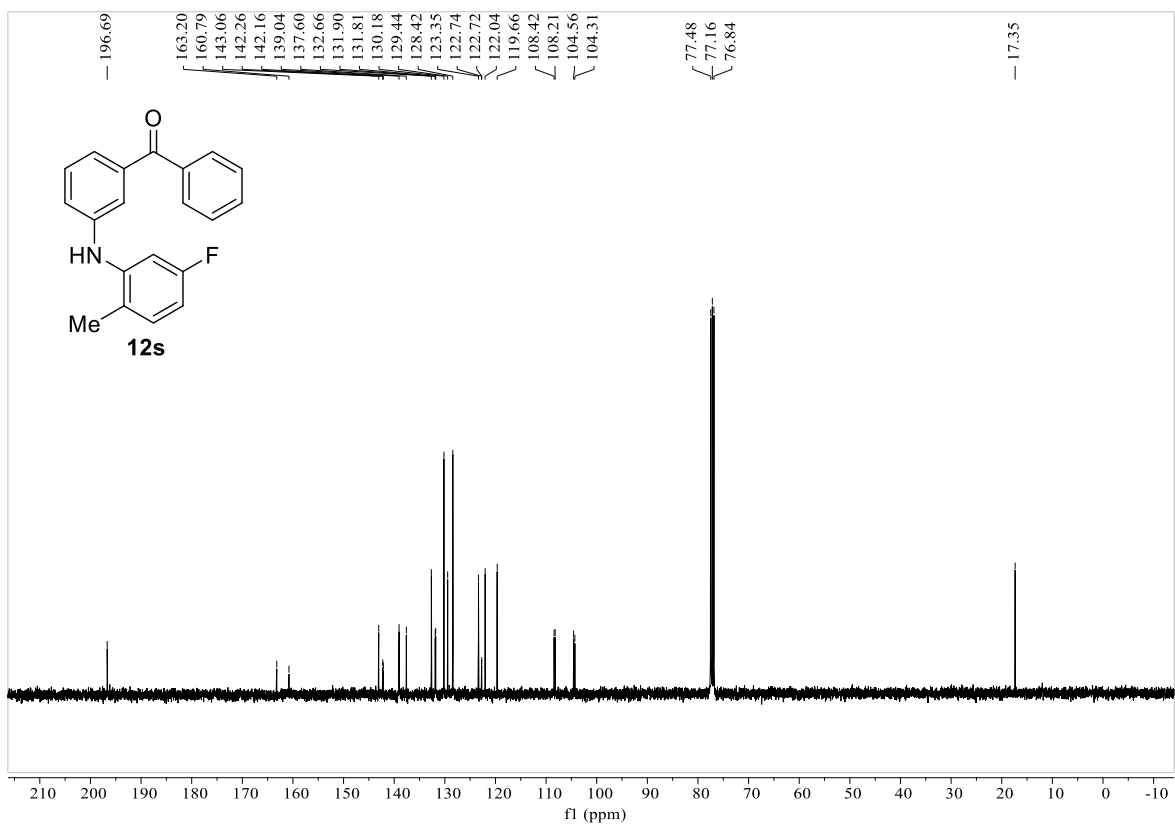
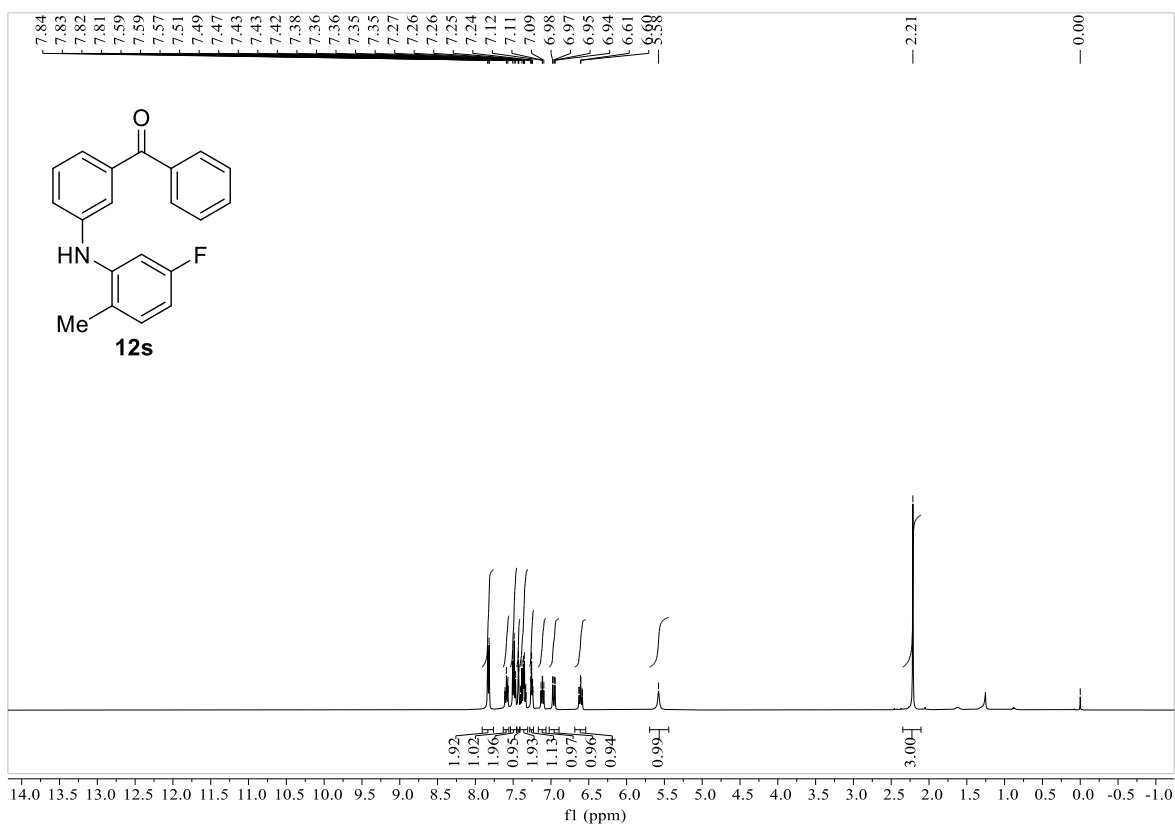
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12q**



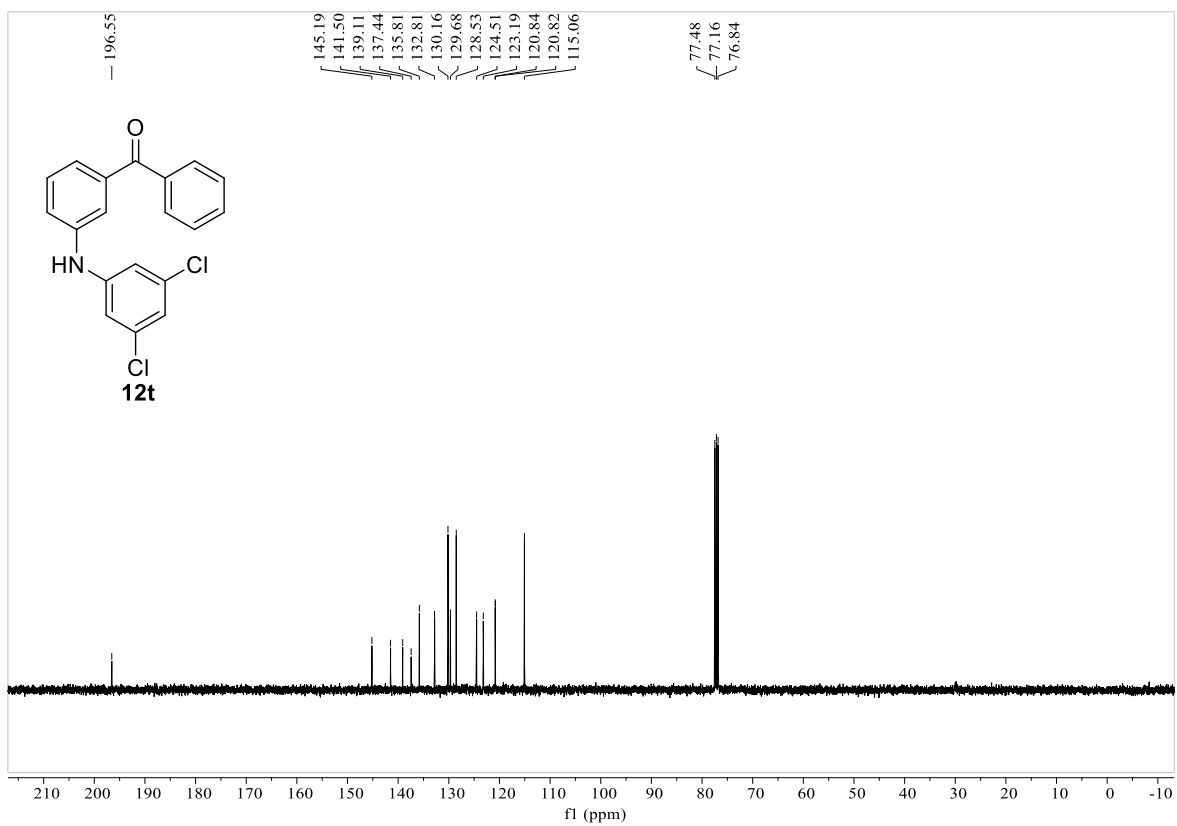
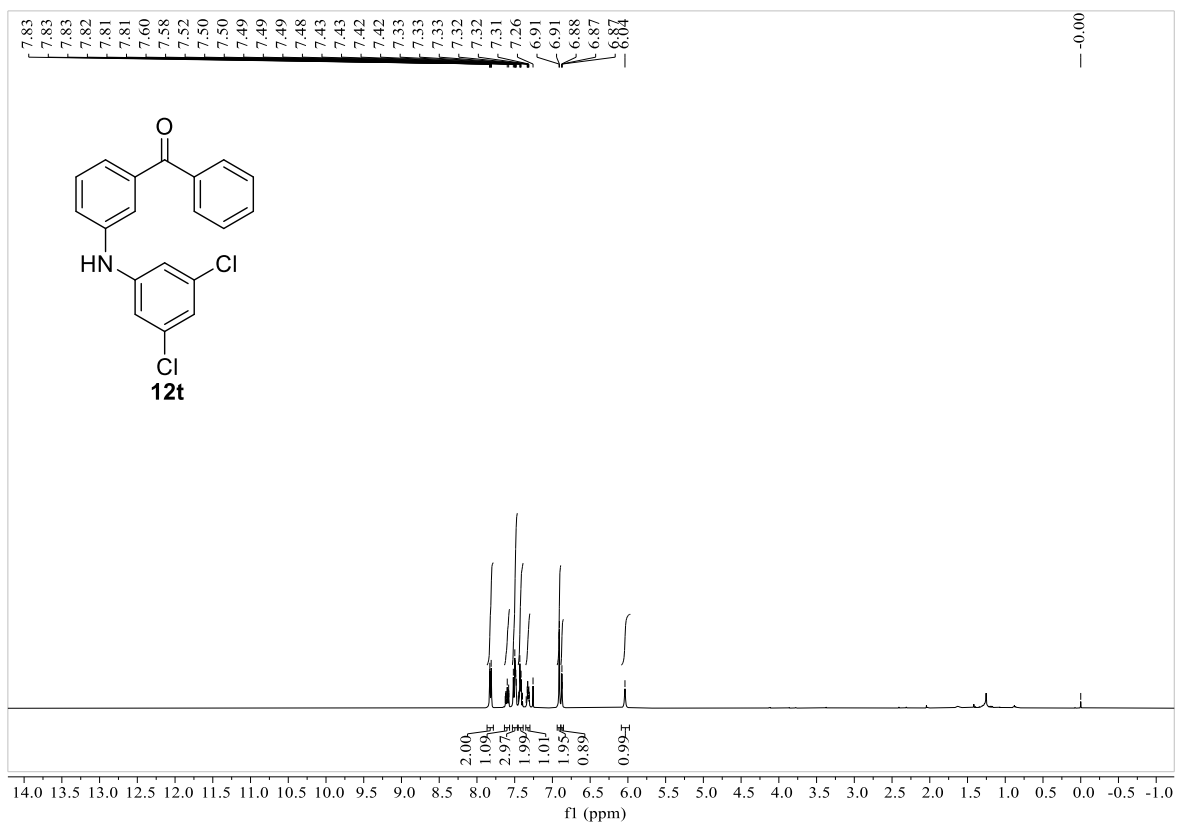
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12r**



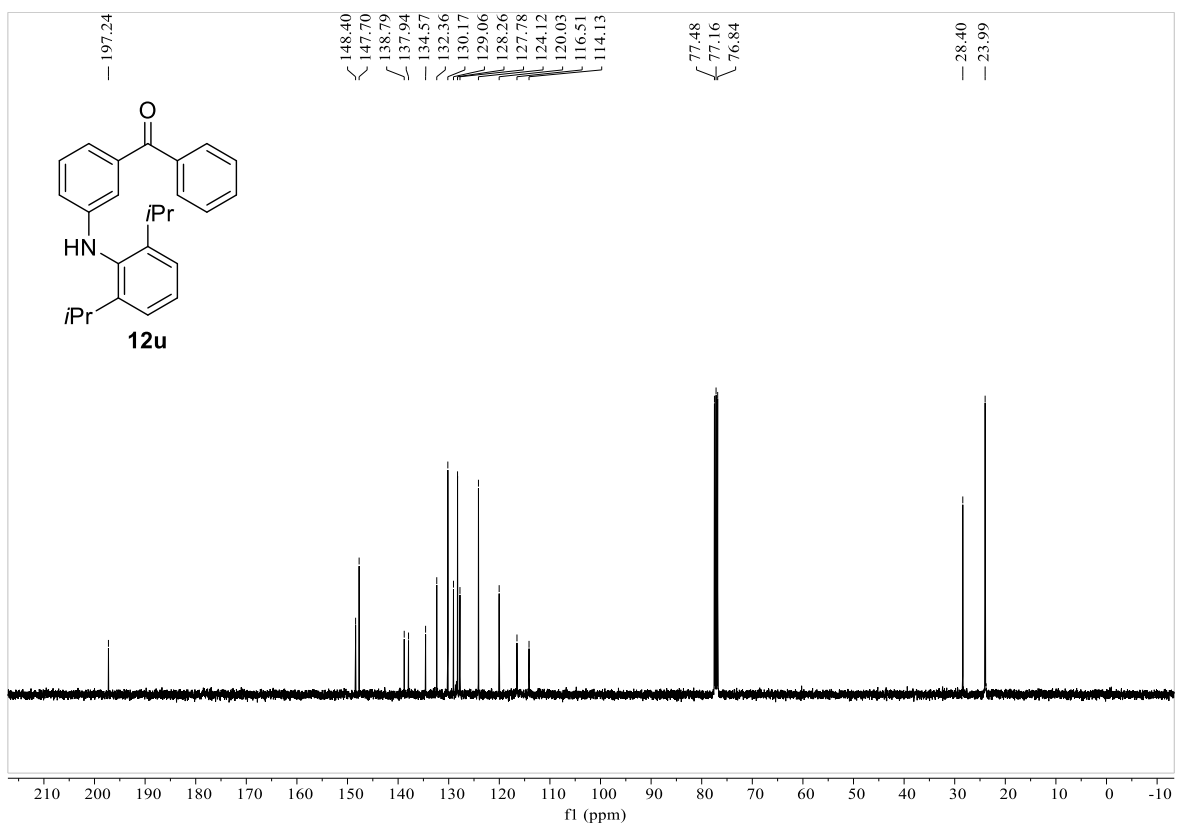
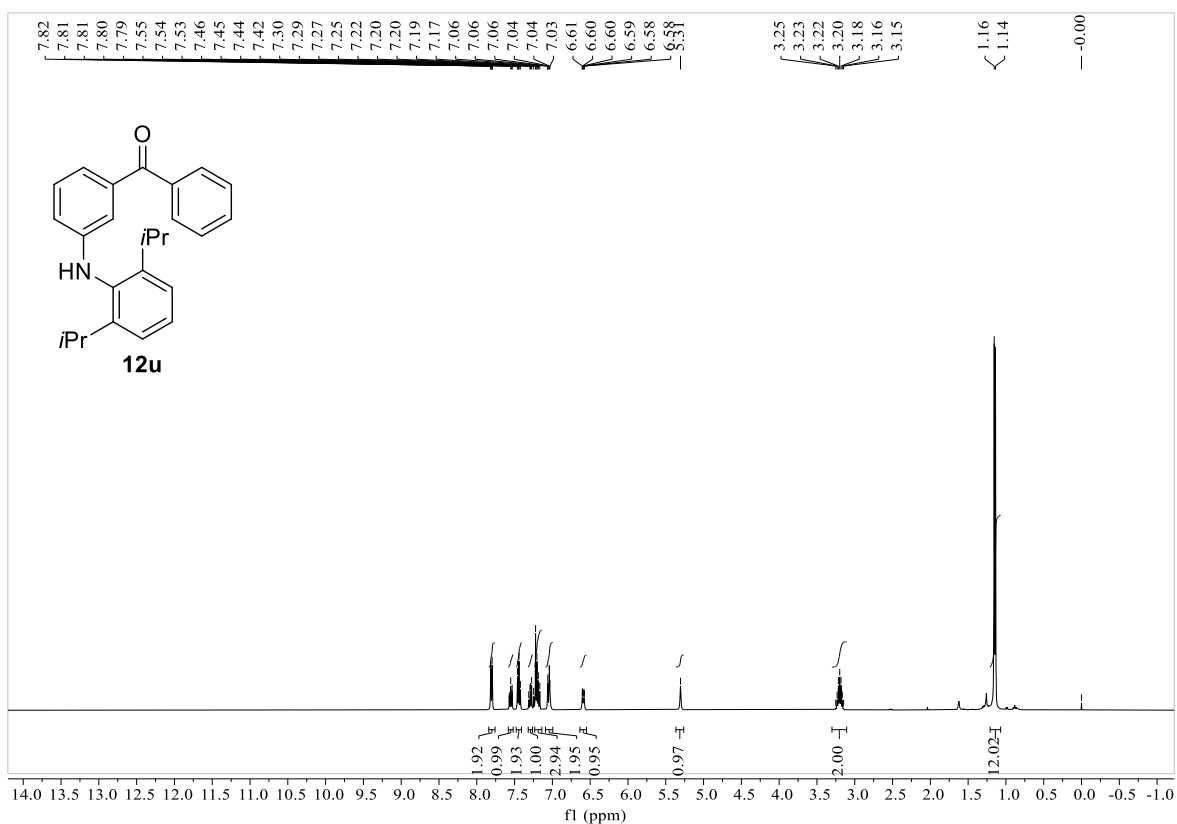
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12s**



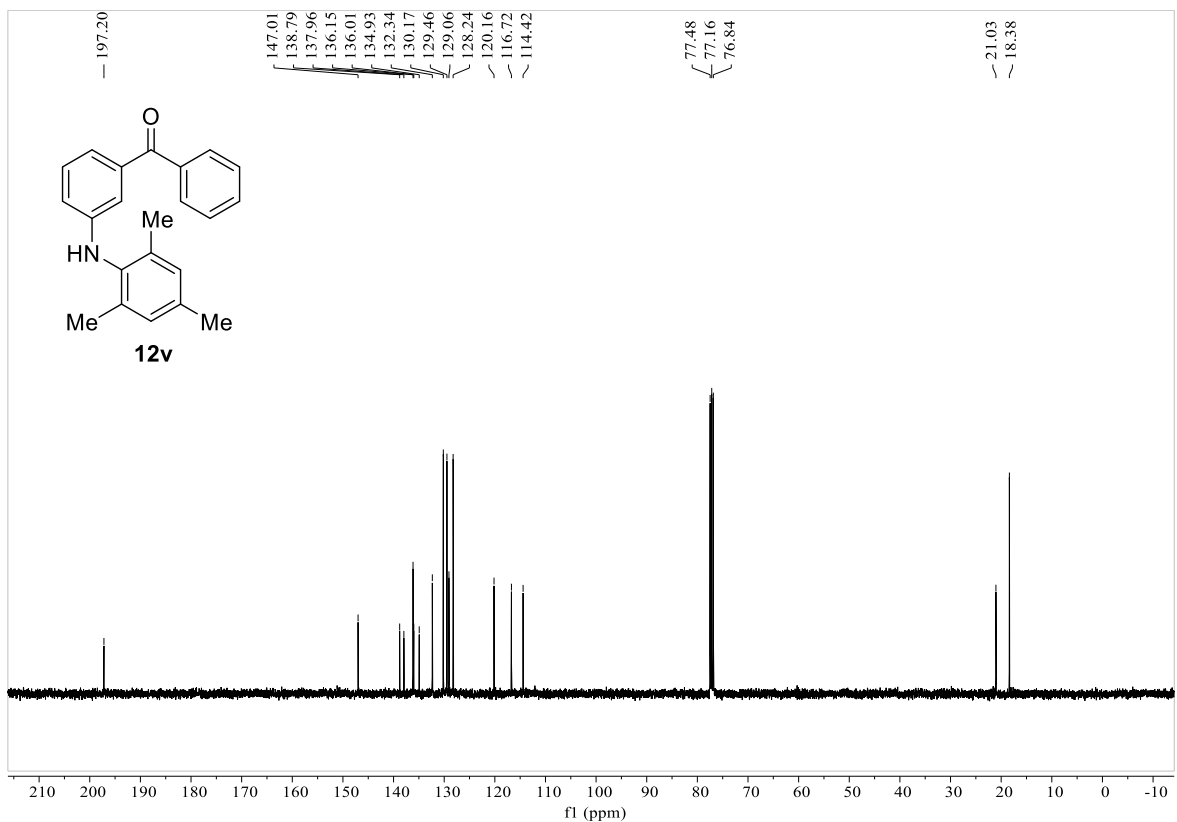
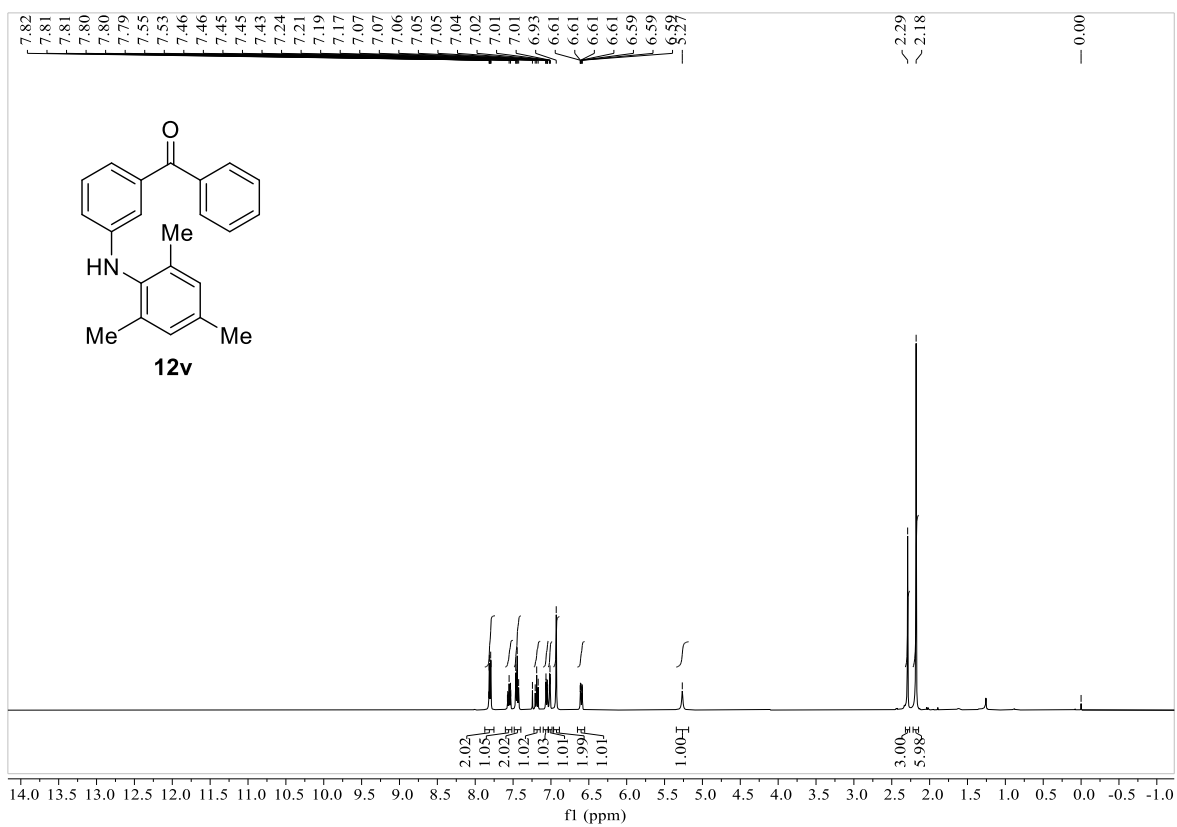
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12t**



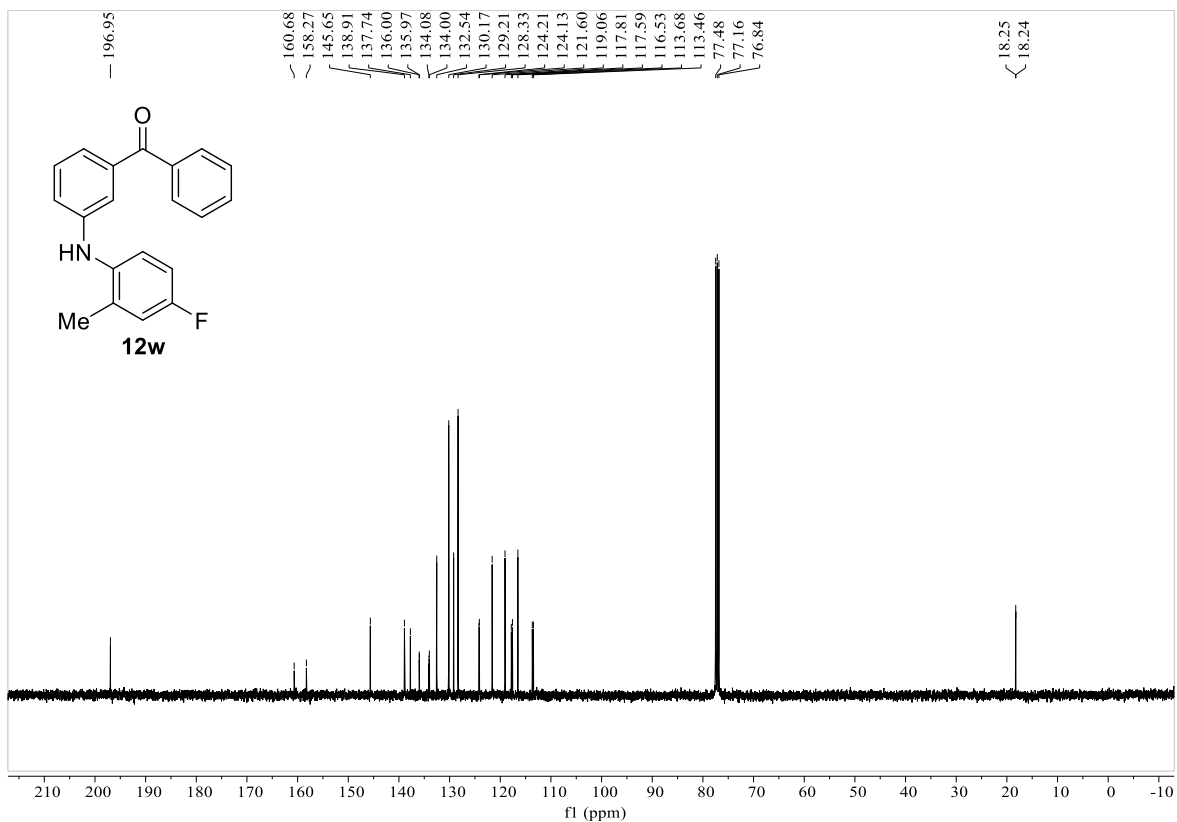
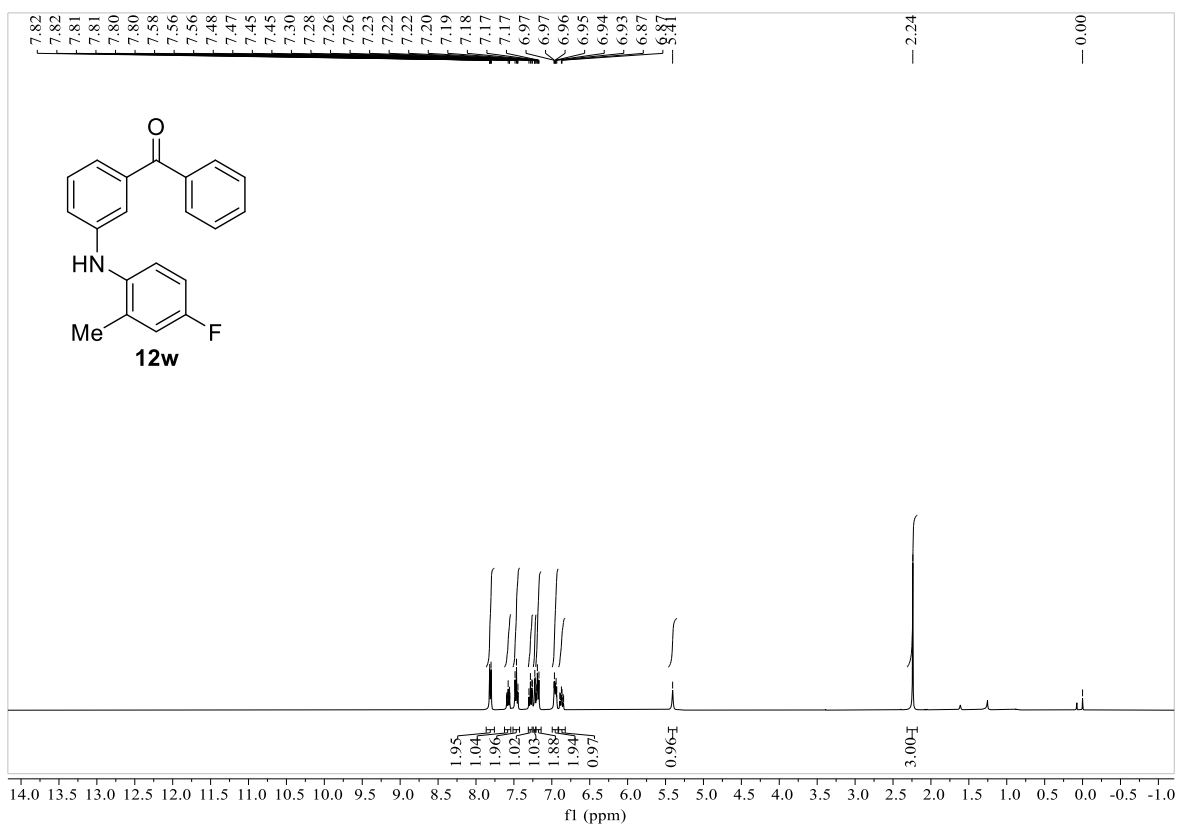
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12u**



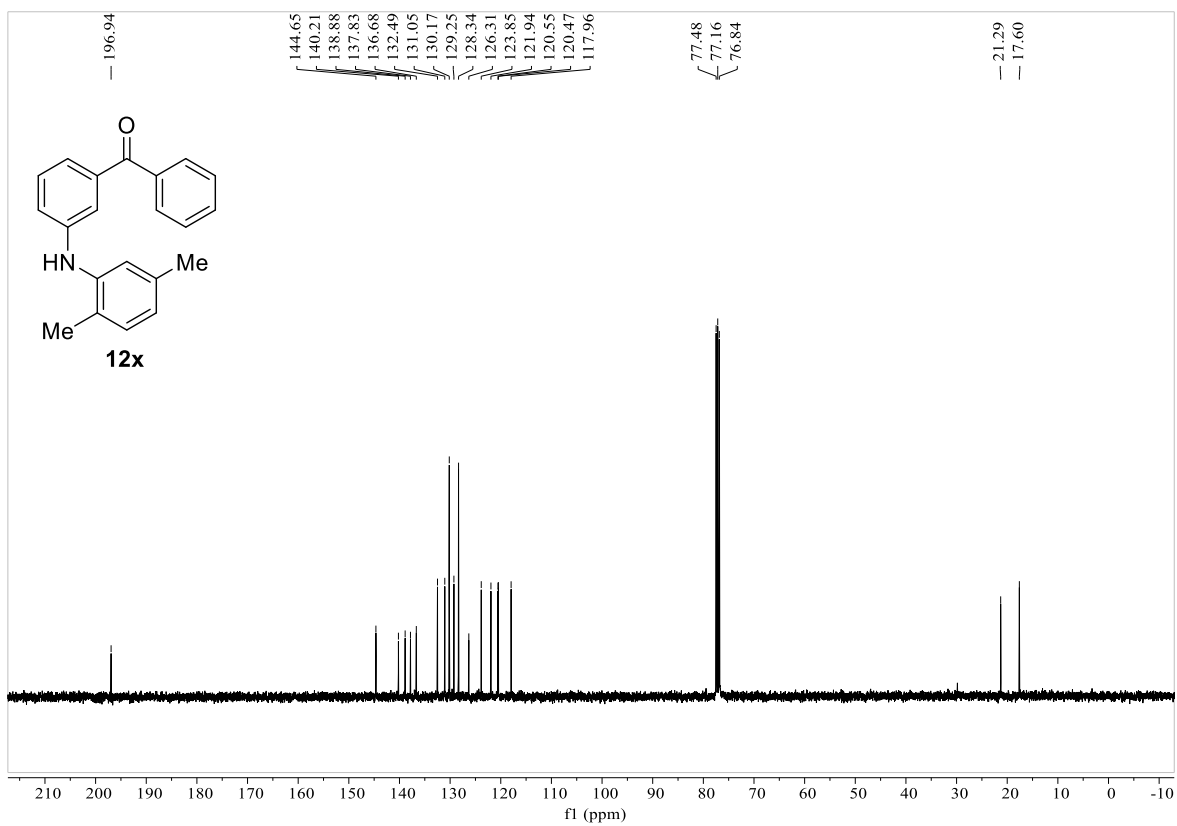
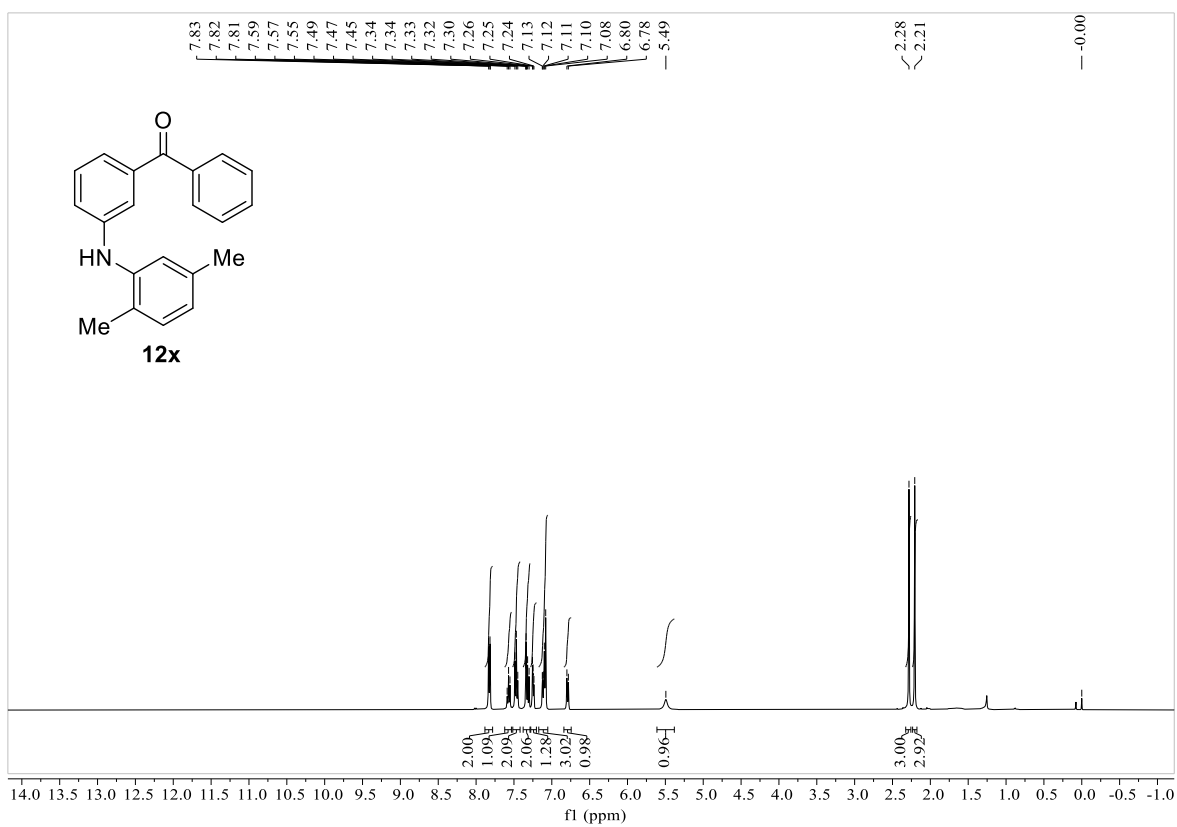
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12v**



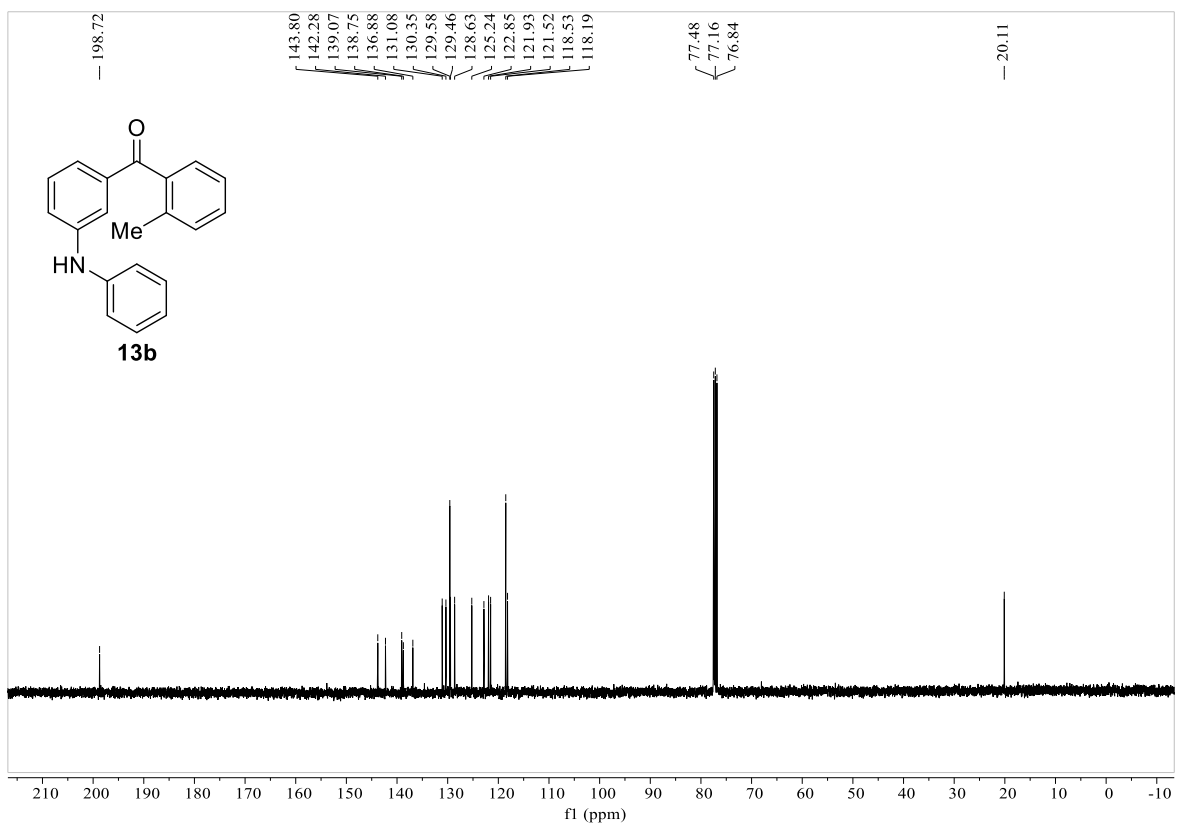
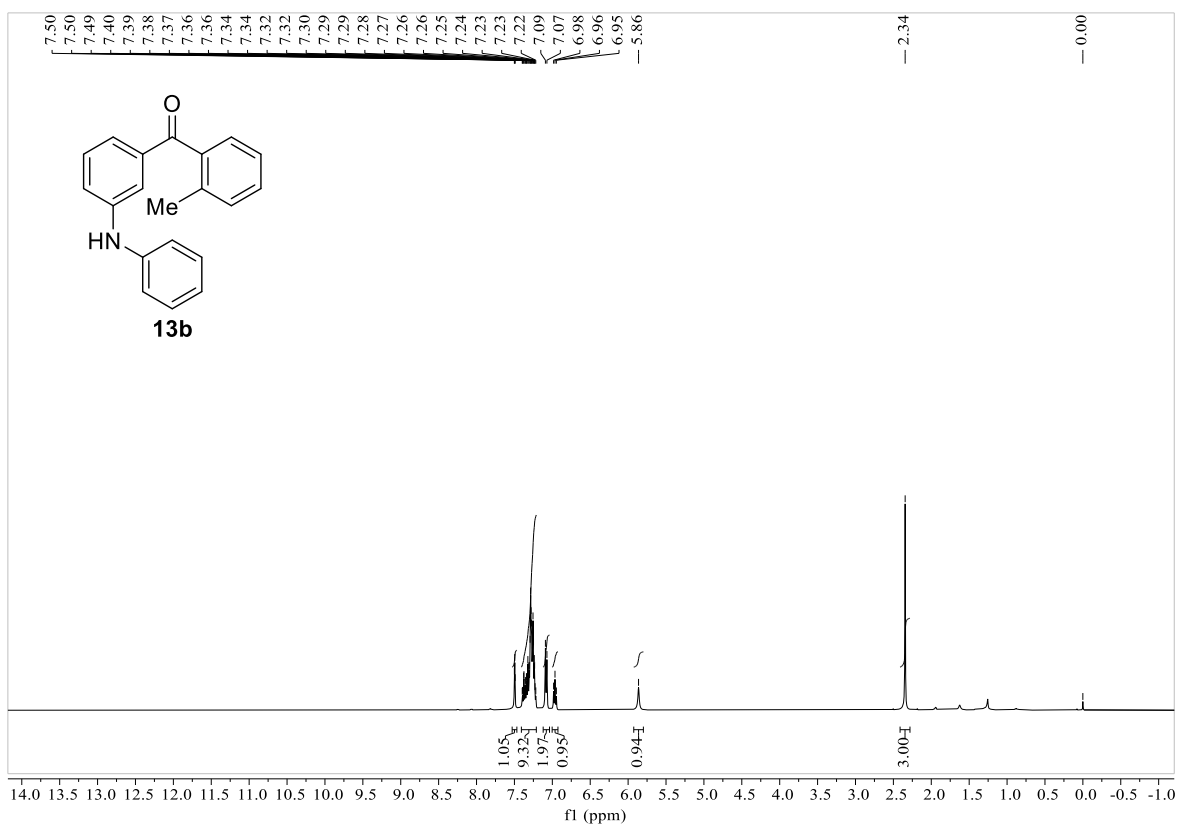
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12w**



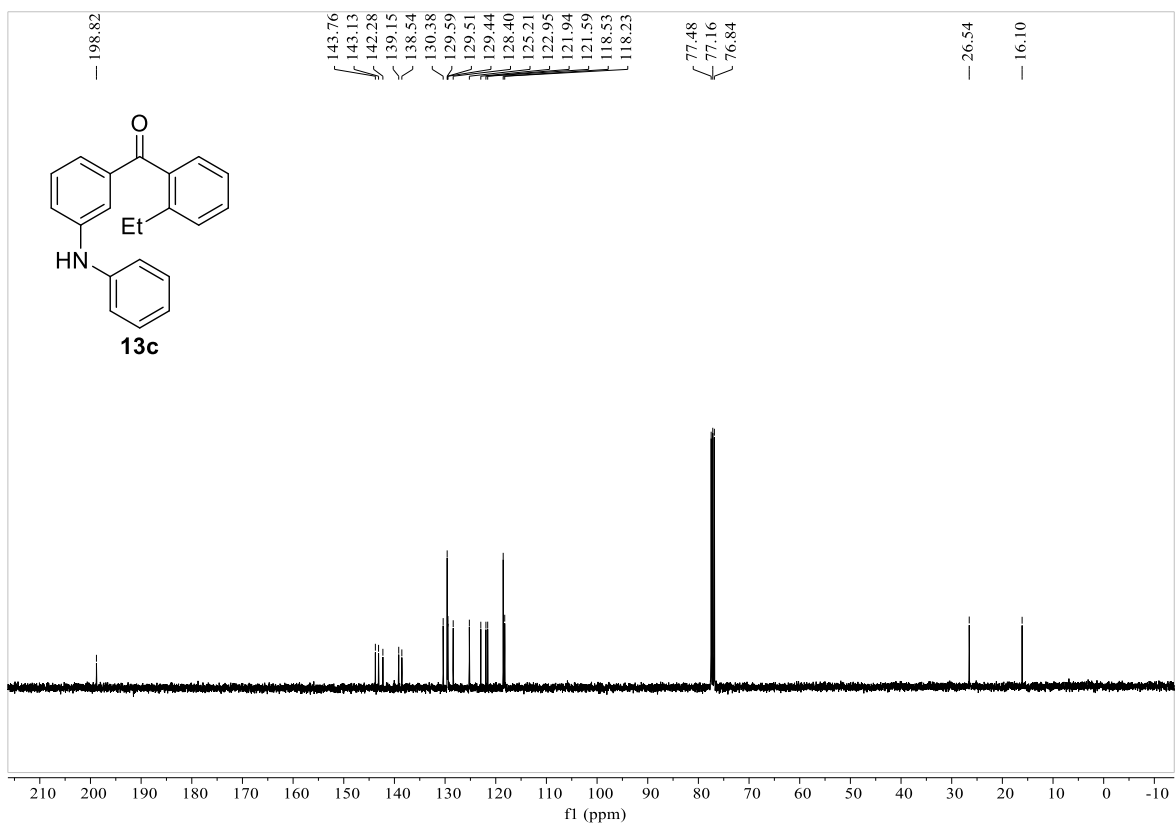
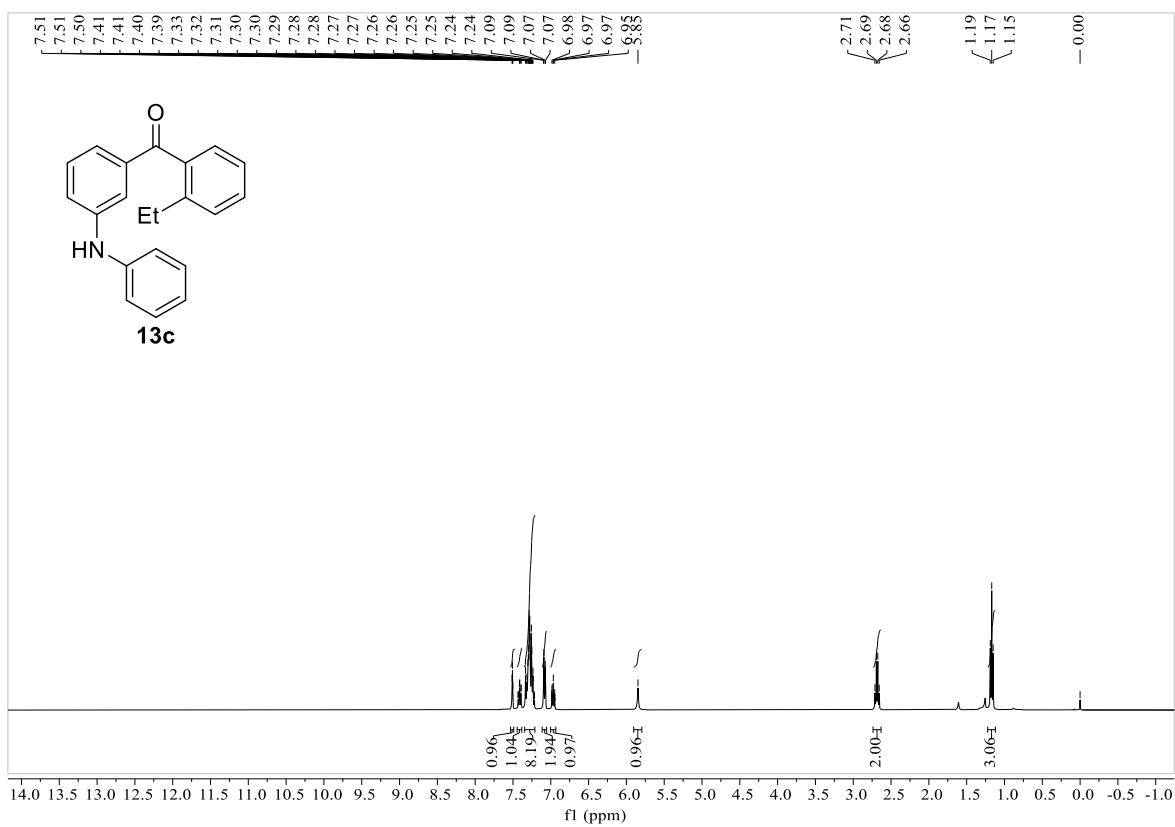
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **12x**



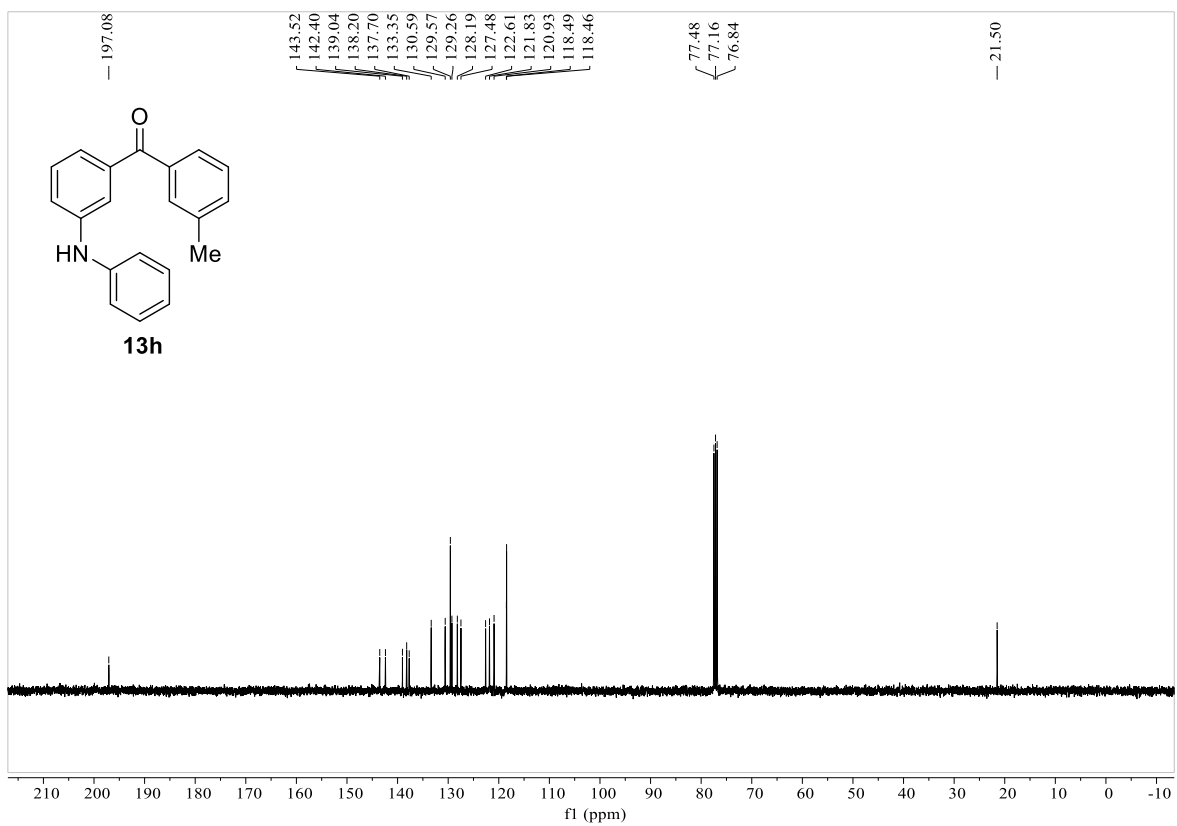
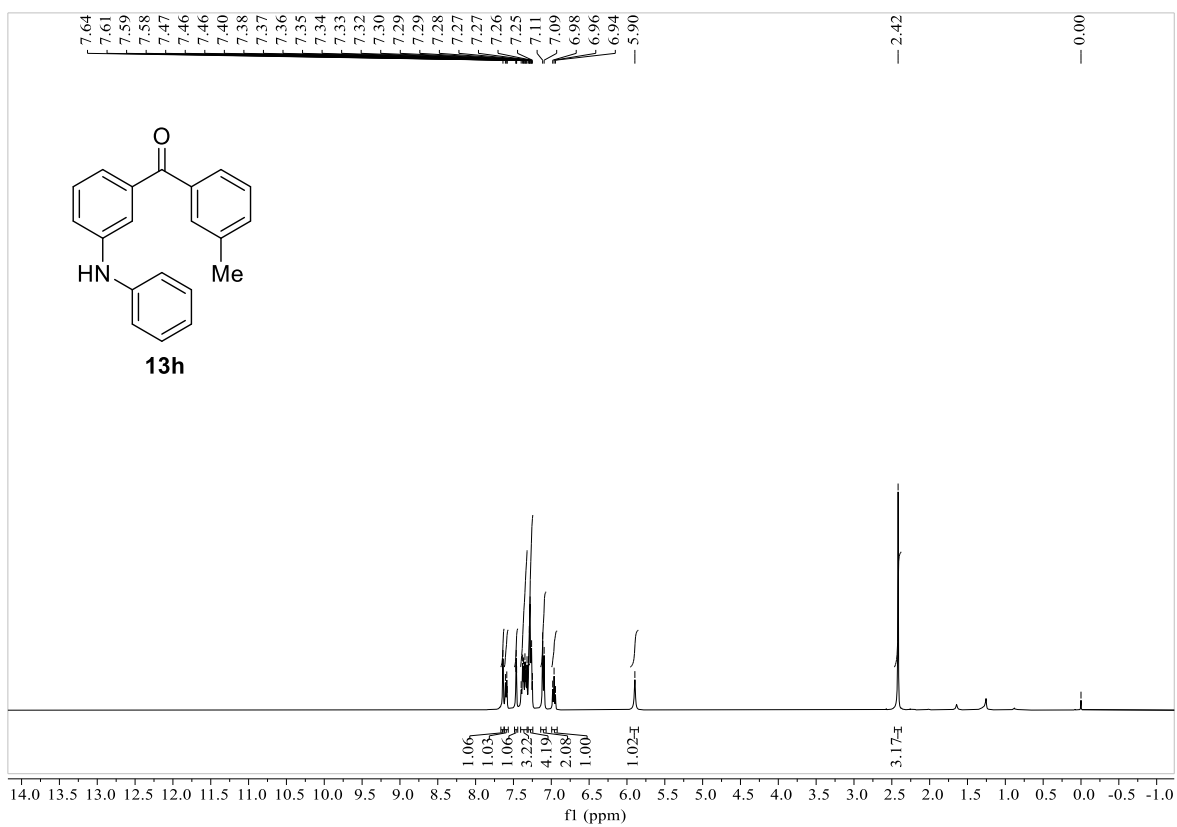
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **13b**



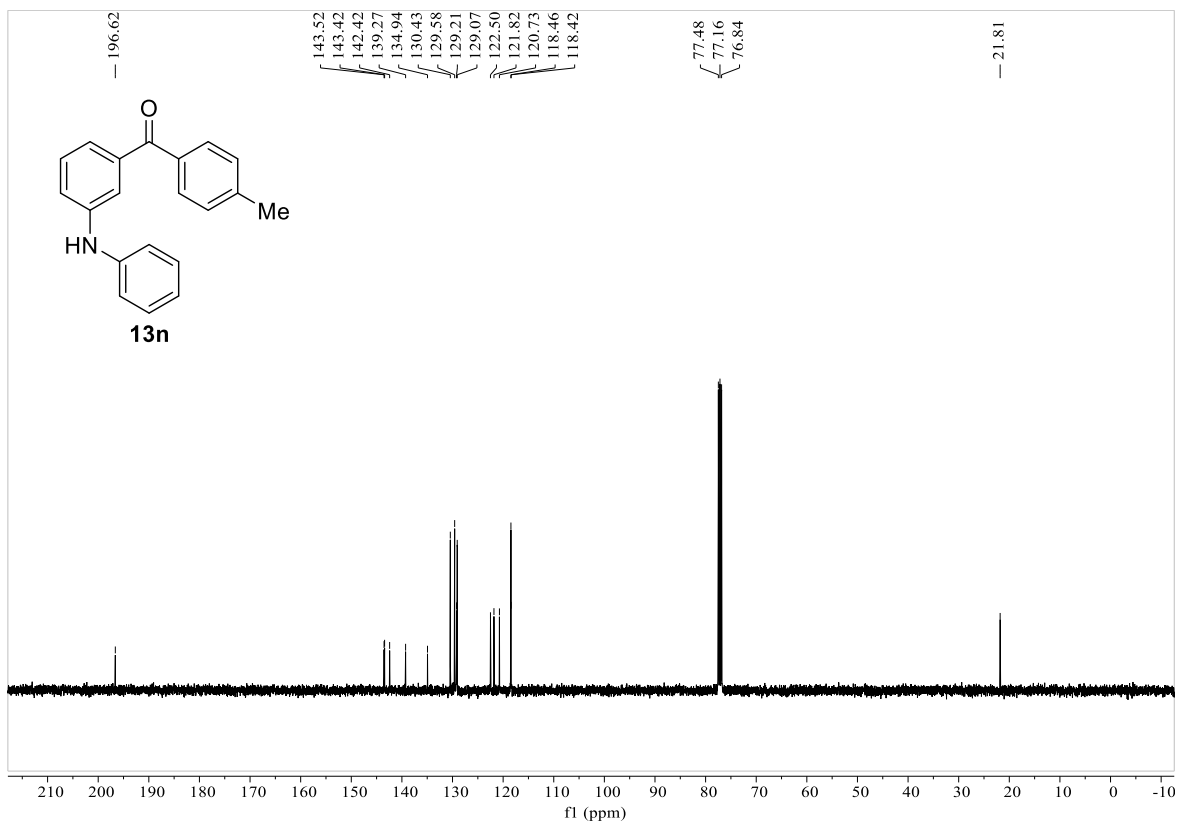
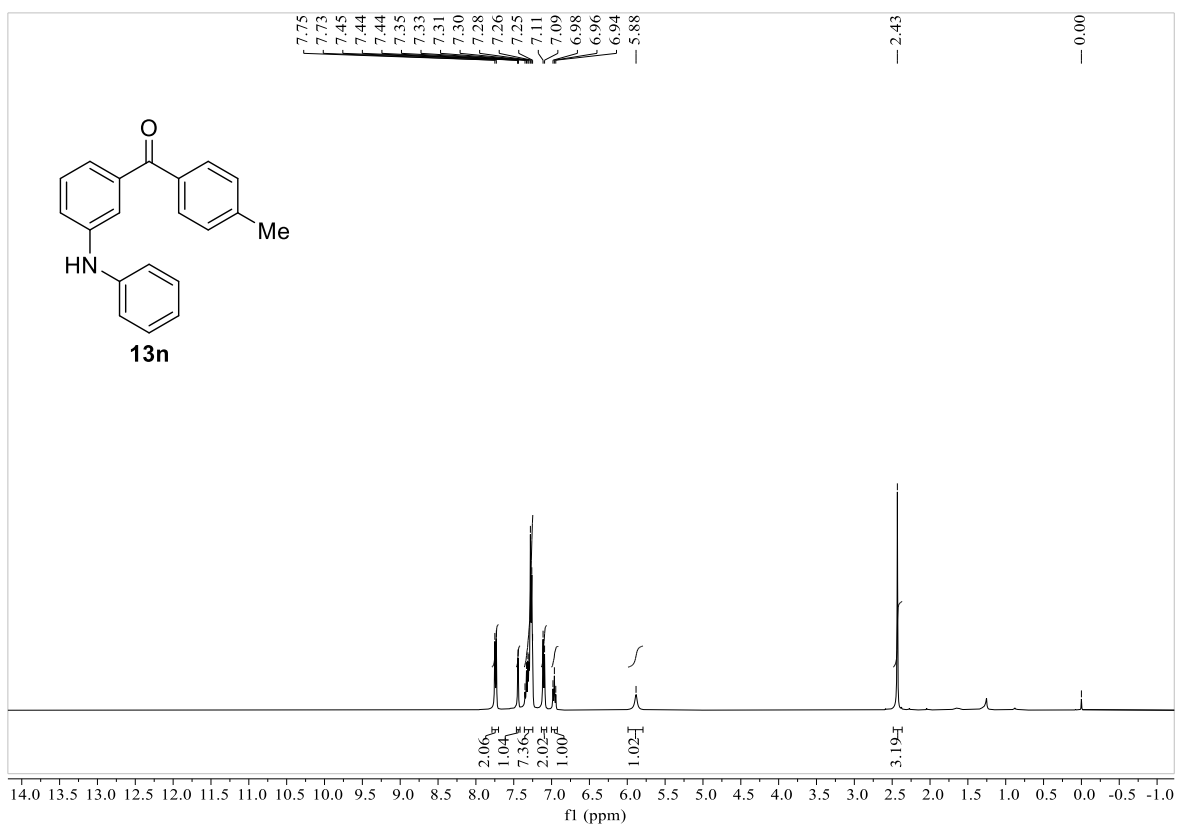
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **13c**



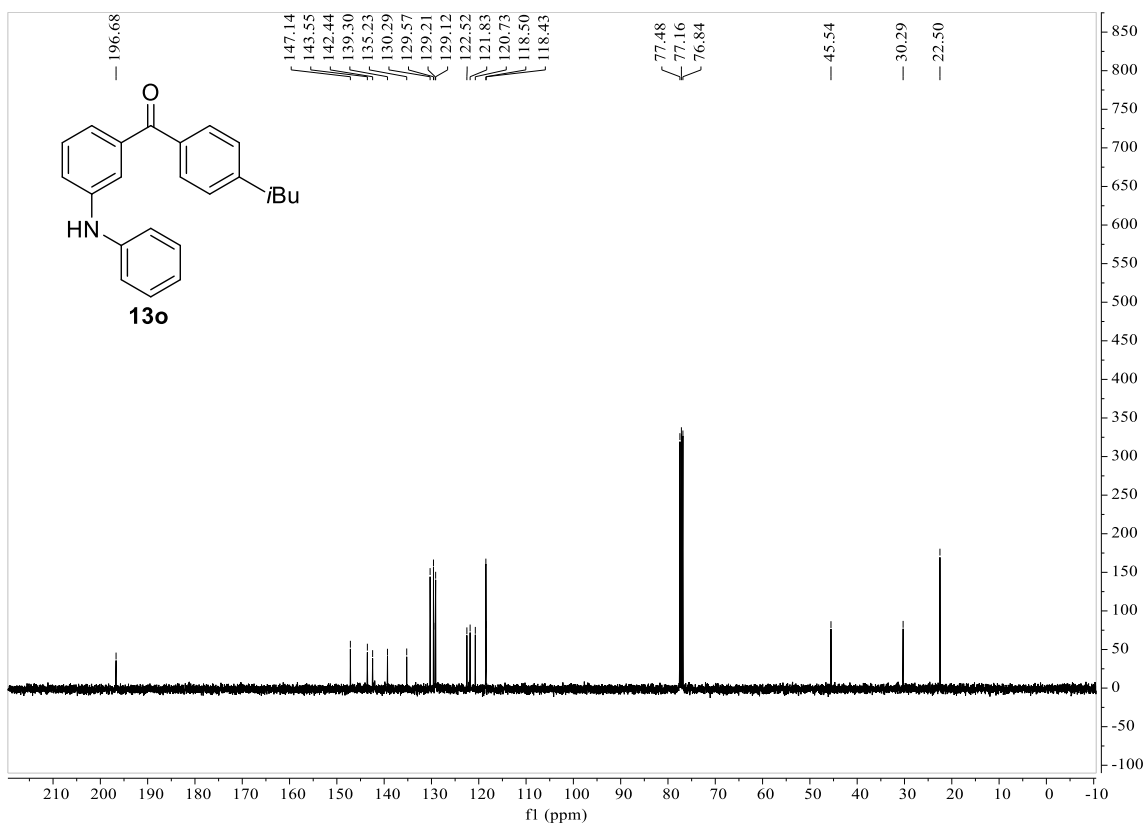
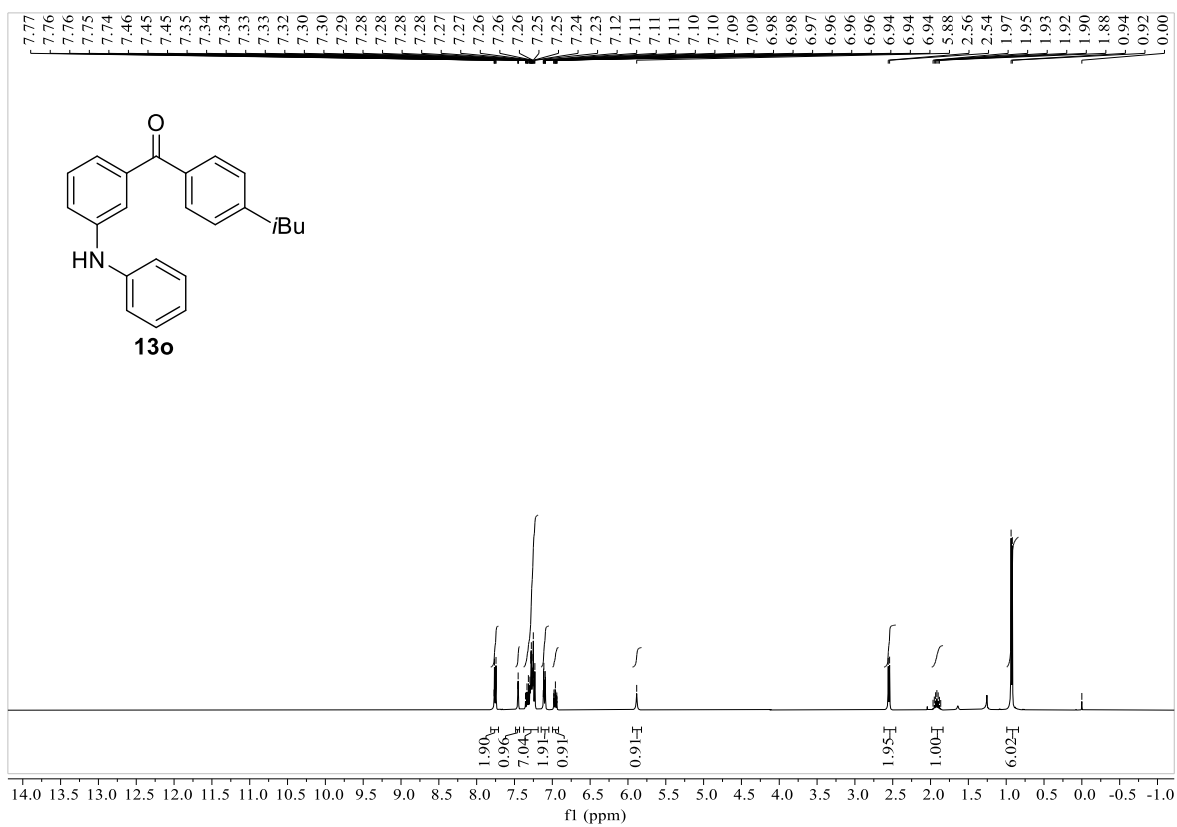
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **13h**



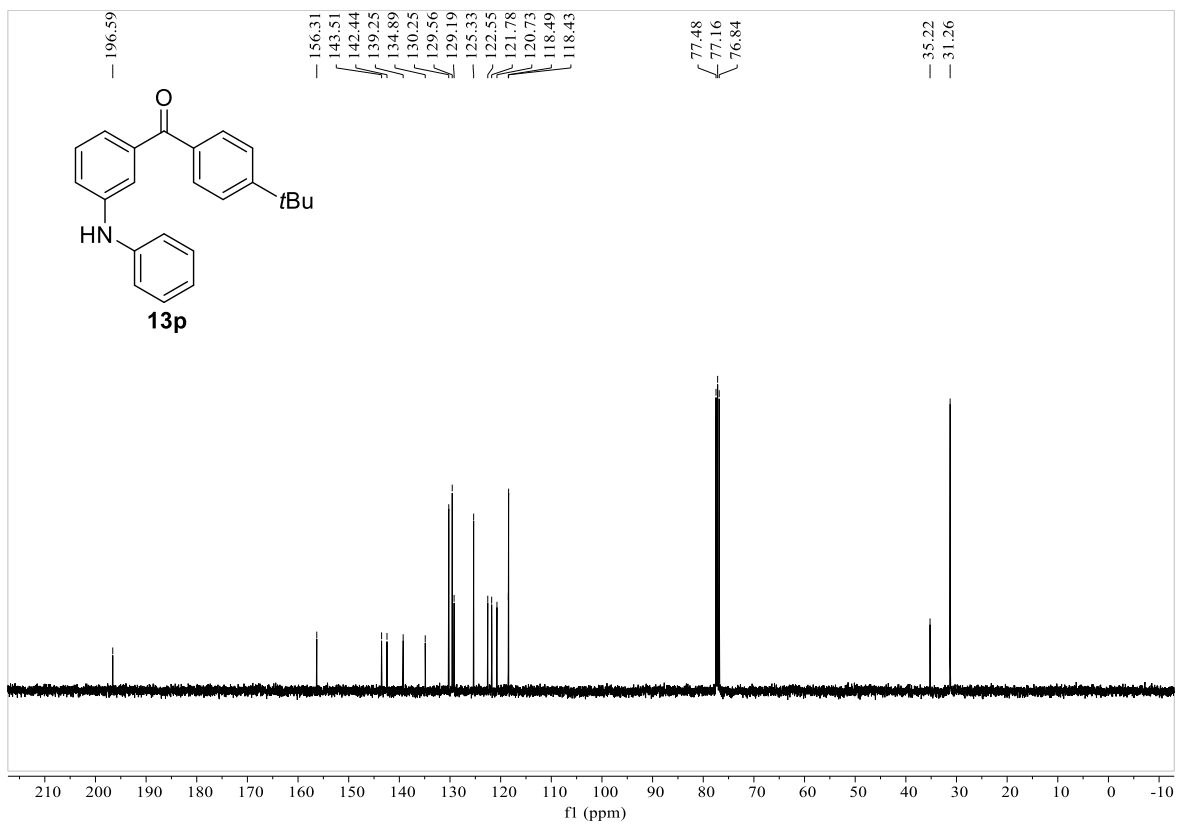
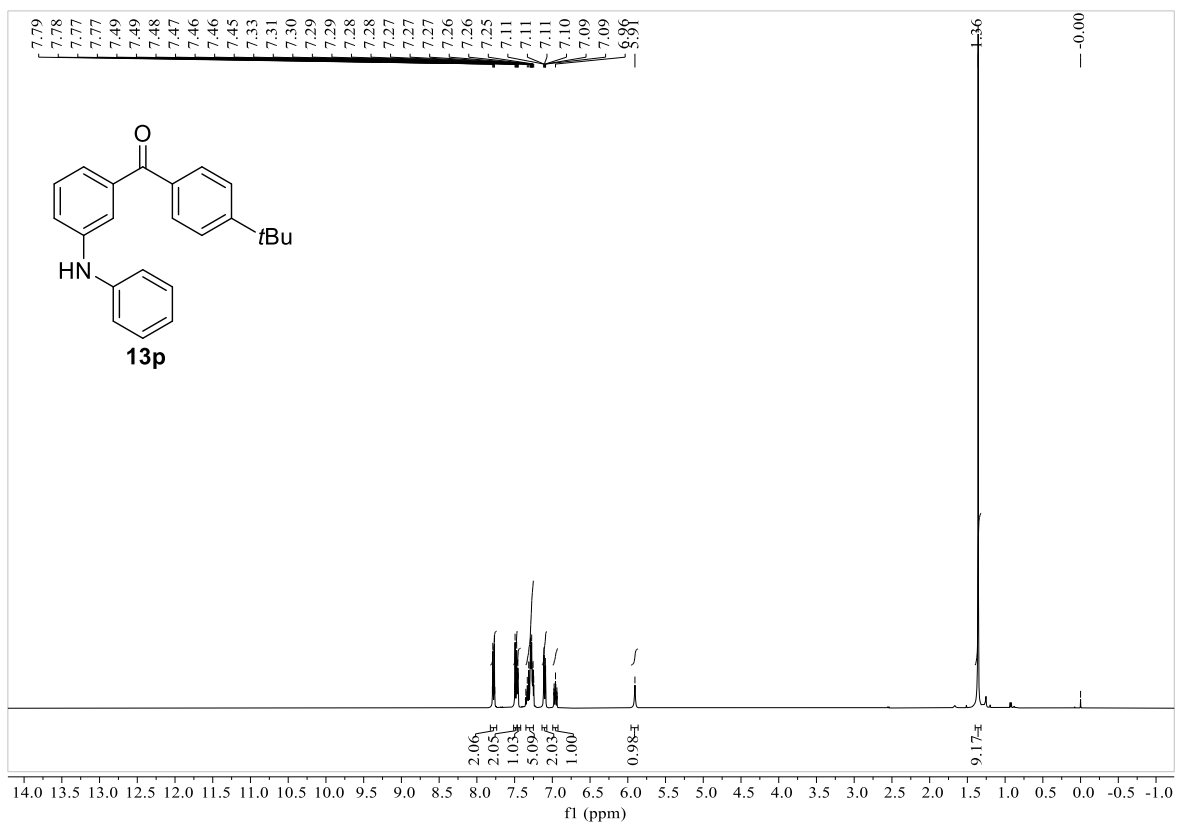
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **13n**



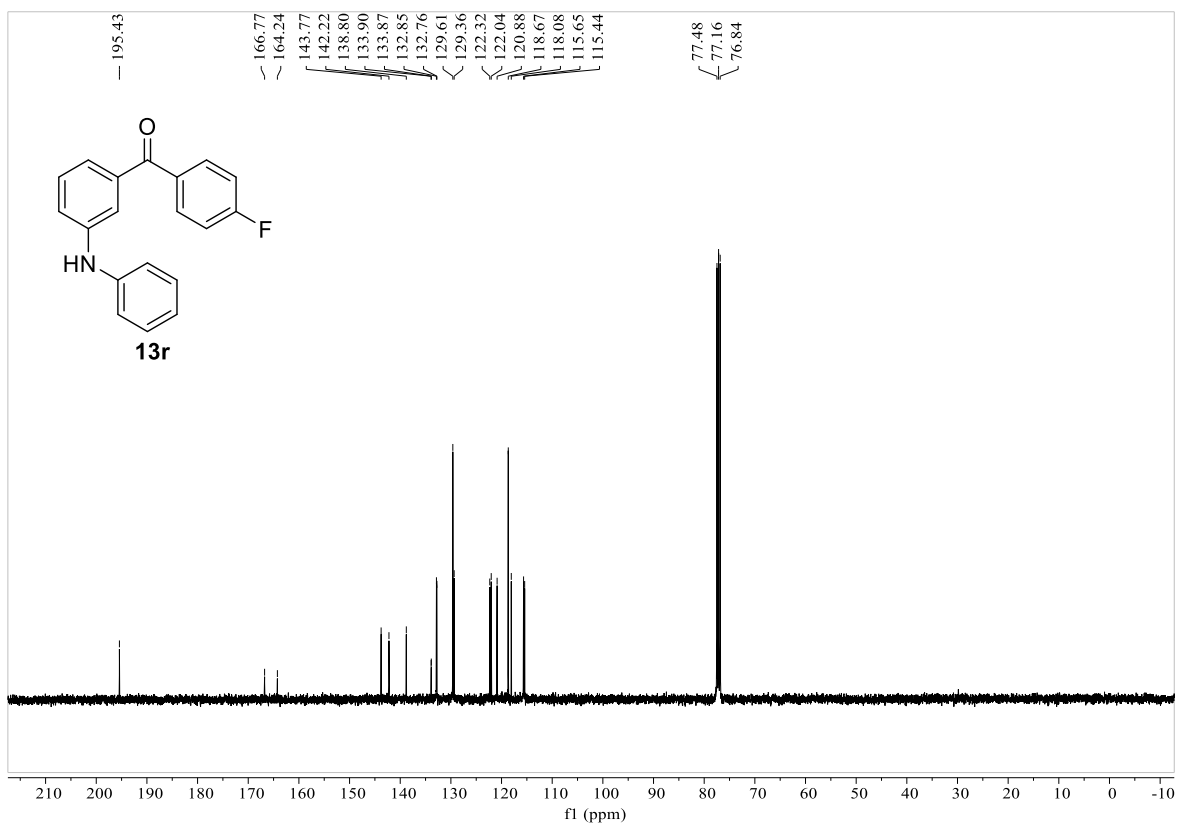
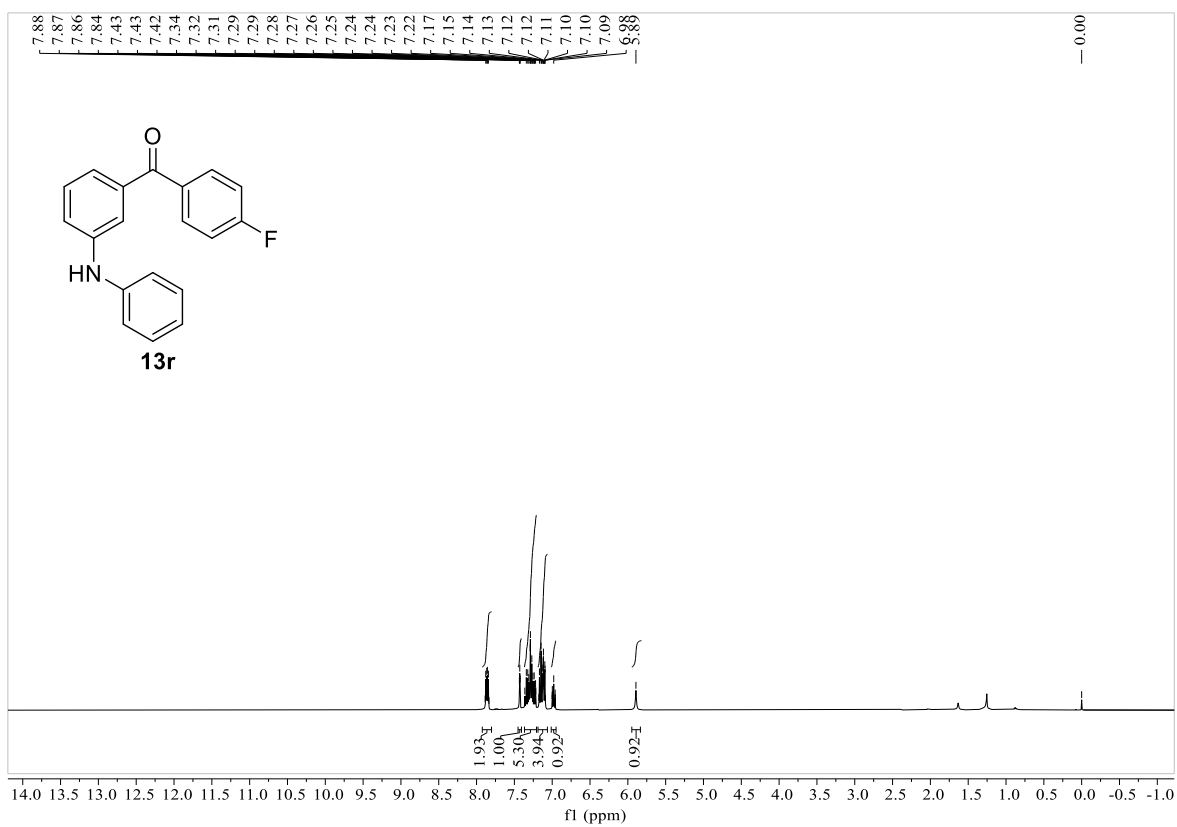
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **13o**



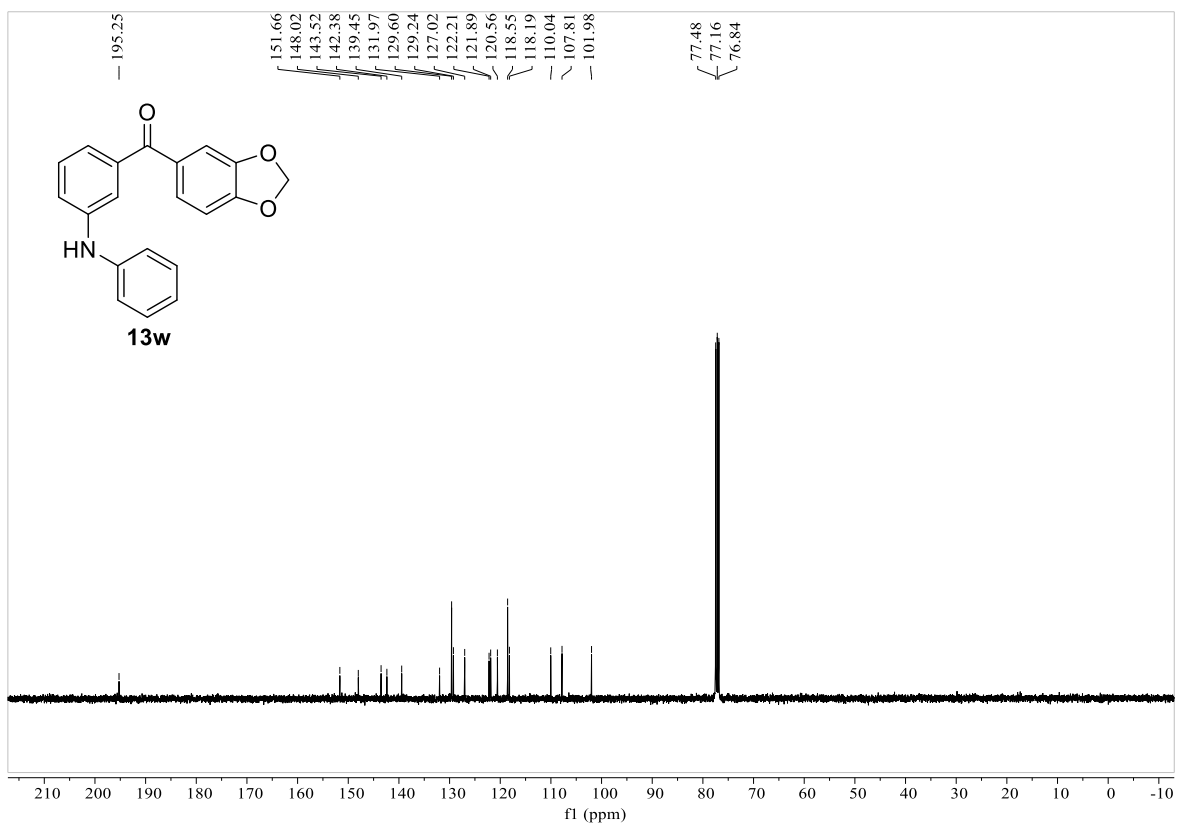
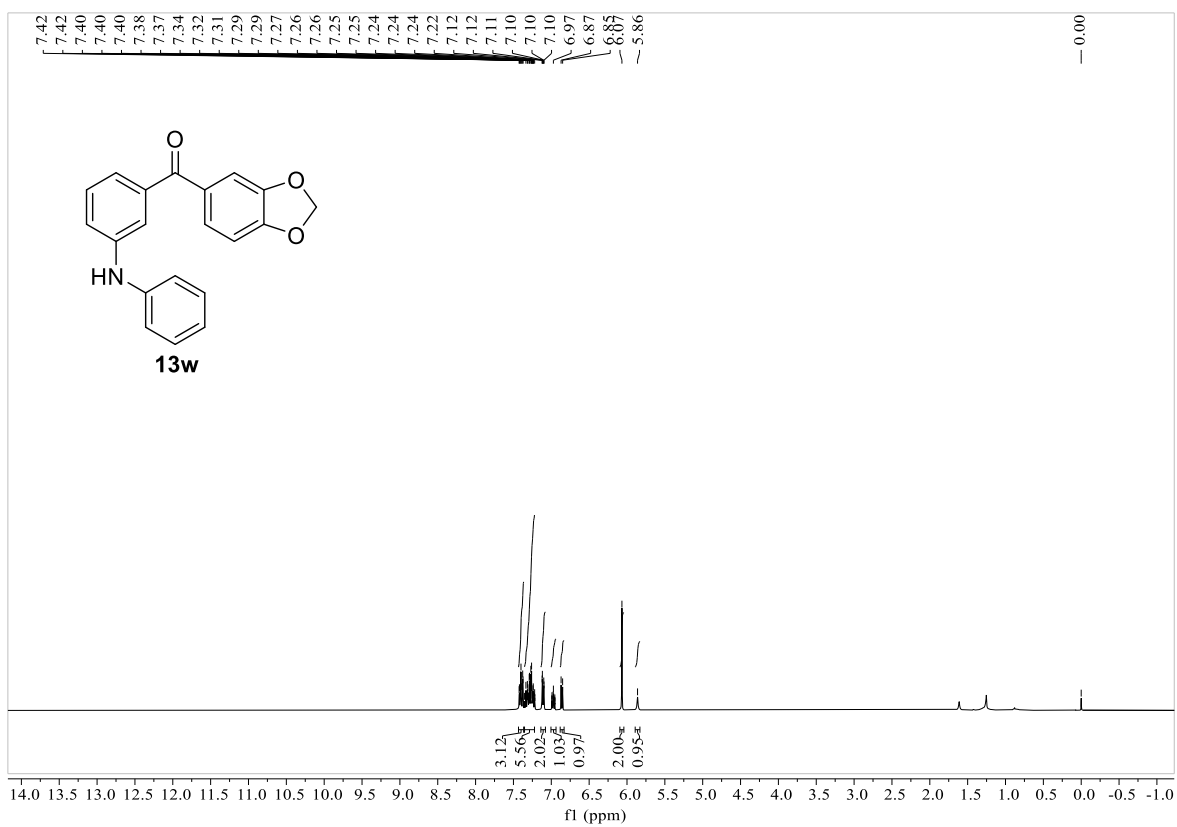
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **13p**



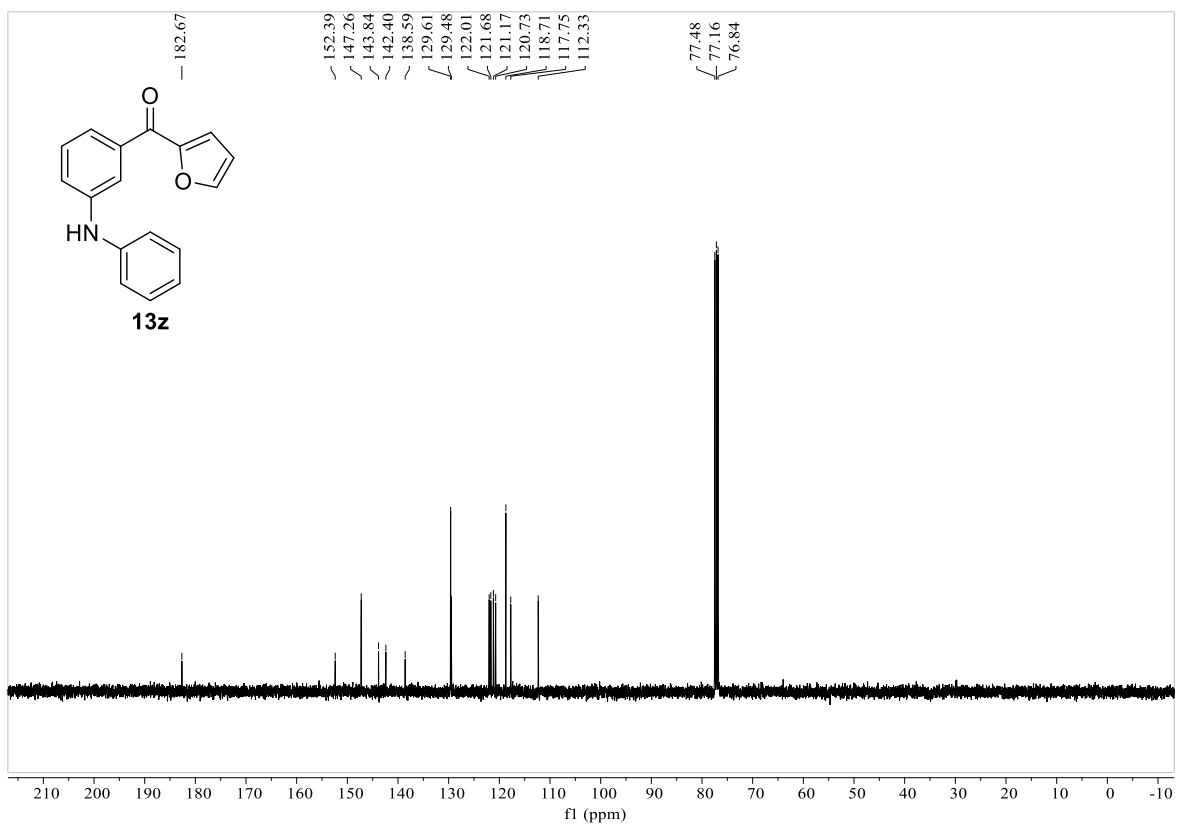
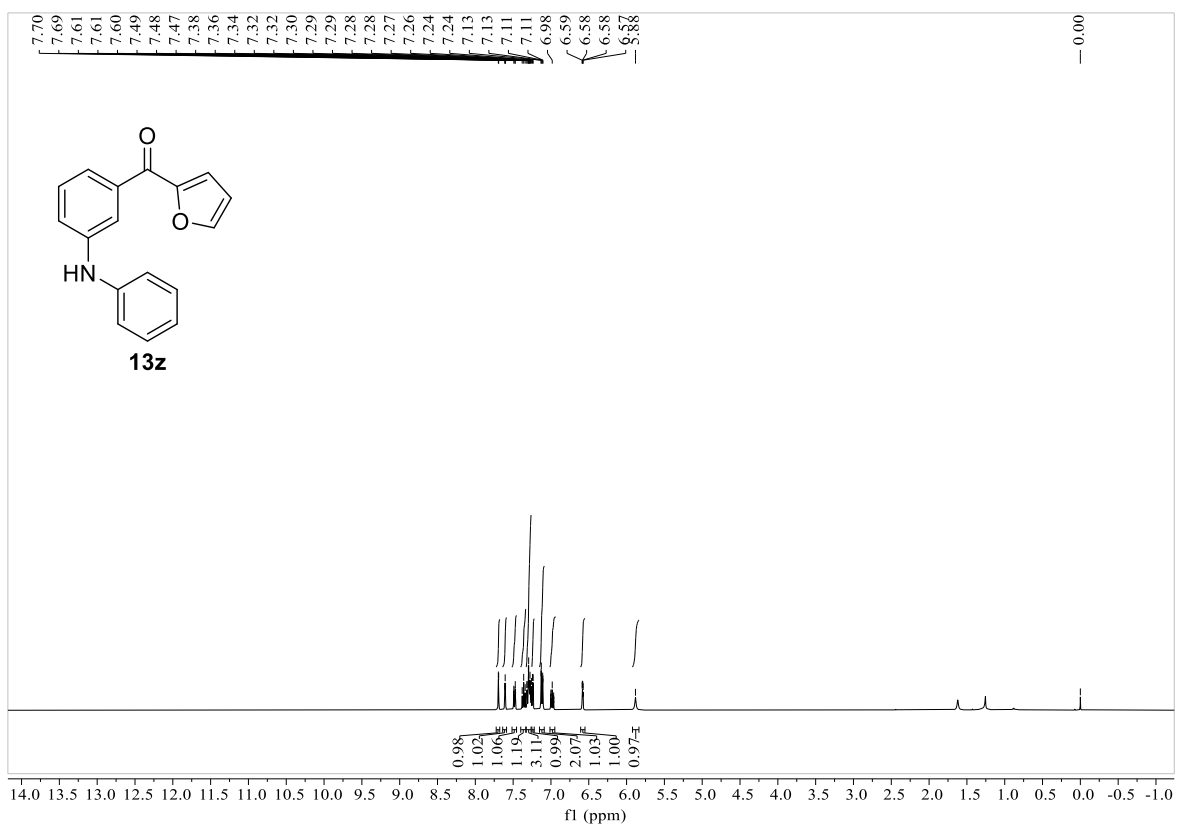
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **13r**



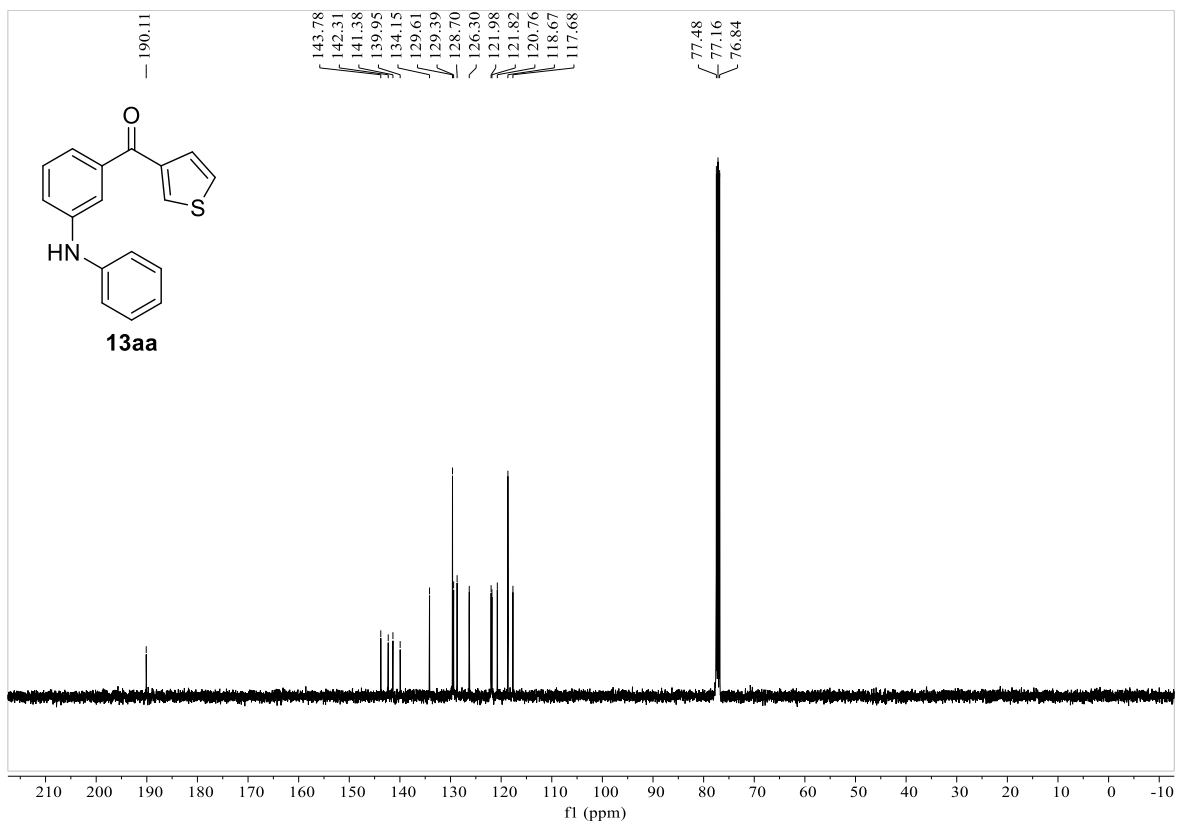
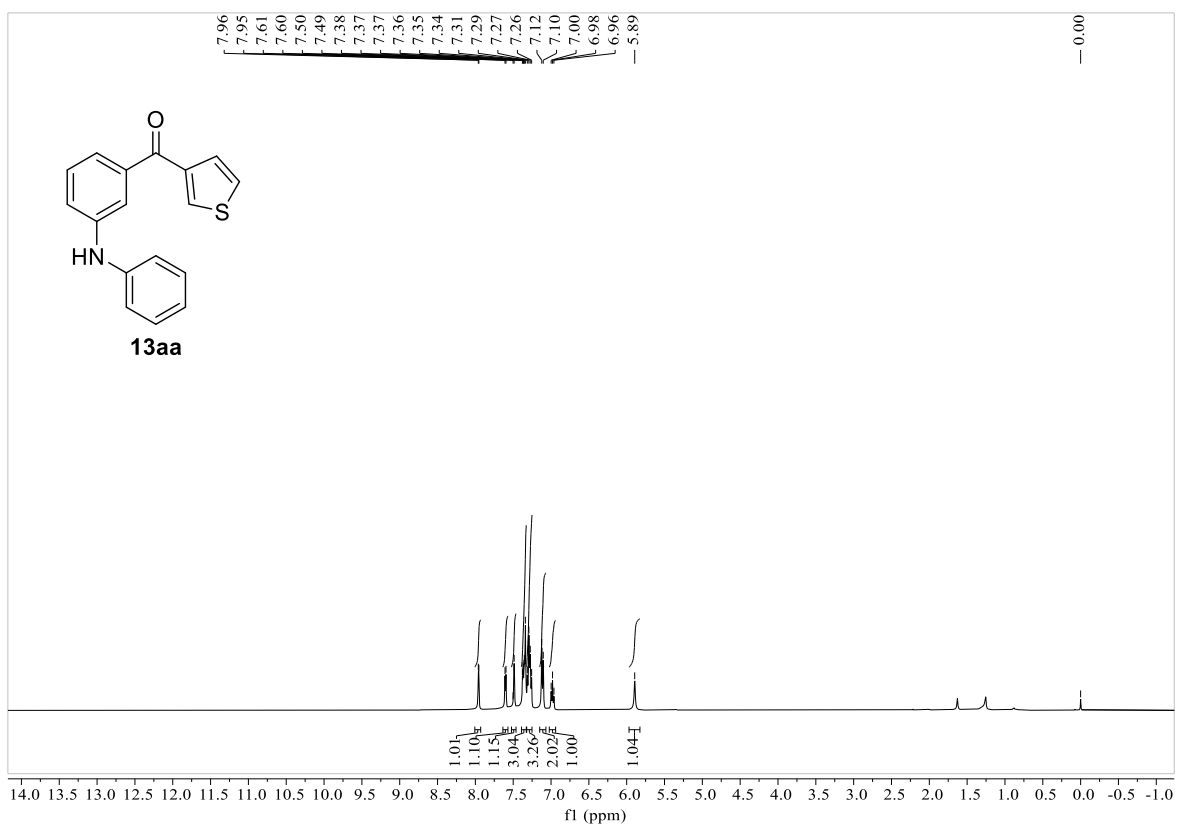
^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **13w**



^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **13z**



^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra of **13aa**



8. References

1. Zhao, B.-Y. et al. Aerobic oxidative dehydrogenation of ketones to 1,4-enediones. *Org. Lett.* **23**, 1216–1221 (2021).
2. Gutiérrez-Bonet, Á., Flores-Gaspar, A. & Martin, R. Fe-catalyzed regiodivergent [1,2]-shift of α -aryl aldehydes. *J. Am. Chem. Soc.* **135**, 12576–12579 (2013).
3. Li, X. & Zou, G. Palladium-catalyzed acylative cross-coupling of amides with diarylborinic acids and sodium tetraarylborates. *J. Organomet. Chem.* **794**, 136–145 (2015).
4. Wu, Q. et al. Pd-catalysed direct C(sp²)-H fluorination of aromatic ketones: concise access to anacetrapib. *Chem. Commun.* **57**, 4544–4547 (2021).
5. Wang, L. et al. Direct C-H arylation of aldehydes by merging photocatalyzed hydrogen atom transfer with palladium catalysis. *ACS Catal.* **10**, 7543–7551 (2020).
6. Wang, C.-A. et al. Palladium-NHC (NHC = N-heterocyclic carbene)-catalyzed Suzuki-Miyaura cross-coupling of alkyl amides. *ACS Catal.* **12**, 2426–2433 (2022).
7. Amani, J. & Molander, G. A. Synergistic photoredox/nickel coupling of acyl chlorides with secondary alkyltrifluoroborates: dialkyl ketone synthesis. *J. Org. Chem.* **82**, 1856–1863 (2017).
8. Lee, G. S., Won, J., Choi, S., Baik, M.-H. & Hong, S. H. Synergistic activation of amides and hydrocarbons for direct C(sp³)-H acylation enabled by metallaphotoredox catalysis. *Angew. Chem. Int. Ed.* **59**, 16933–16942 (2020).
9. Jana, A. et al. A phosphine-free manganese catalyst enables stereoselective synthesis of (1 + *n*)-membered cycloalkanes from methyl ketones and 1,*n*-Diols. *ACS Catal.* **10**, 2615–2626 (2020).
10. Ardhean, R., Mortimore, M., Paton, R. S. & Fletcher, S. P. Formation of quaternary centres by copper catalysed asymmetric conjugate addition to β -substituted cyclopentenones with the aid of a quantitative structure–selectivity relationship. *Chem. Sci.* **9**, 2628–2632 (2018).
11. Mazzarella, D., Pulcinella, A., Bovy, L., Broersma, R. & Nođ, T. Rapid and direct photocatalytic C(sp³)-H acylation and arylation in flow. *Angew. Chem. Int. Ed.* **60**, 21277–21282 (2021).
12. Liu, M. T., Na, M., Li, Y., Biscoe, M. R. & Ryan, K. Conformational sensing by a mammalian olfactory receptor. *Chem. Eur. J.* **26**, 11462–11469 (2020).
13. Michailidou, F. et al. Pac13 is a small, monomeric dehydratase that mediates the formation of the 3'-deoxy nucleoside of pacidamycins. *Angew. Chem. Int. Ed.* **56**, 12492–12497 (2017).
14. Zhang, X., Wang, Z., Fan, X. & Wang, J. Synthesis of ketones through microwave irradiation promoted metal-free alkylation of aldehydes by activation of C(sp³)-H bond. *J. Org. Chem.* **80**, 10660–10667 (2015).
15. Zheng, Z. et al. Copper-catalyzed synthesis of 2-acylbenzo[*b*]thiophenes from 3-(2-iodophenyl)-1-arylpropan-1-ones and potassium sulfide under aerobic conditions. *Org. Biomol. Chem.* **16**, 8020–8024 (2018).
16. Liu, Q. et al. Decatungstate catalyzed photochemical acetylation of C(sp³)-H bonds. *Org. Lett.* **24**, 7983–7987 (2022).
17. Huang, B., Li, Y., Yang, C. & Xia, W. Electrochemical 1,4-reduction of α,β -unsaturated ketones with methanol and ammonium chloride as hydrogen sources. *Chem. Commun.* **55**, 6731–6734 (2019).
18. Sumino, S., Ui, T., Hamada, Y., Fukuyama, T. & Ryu, I. Carbonylative Mizoroki–Heck reaction of alkyl iodides with arylalkenes using a Pd/photoirradiation system. *Org. Lett.* **17**, 4952–4955 (2015).

19. Abdel-Halim, M. et al. From celecoxib to a novel class of phosphodiesterase 5 inhibitors: trisubstituted pyrazolines as novel phosphodiesterase 5 inhibitors with extremely high potency and phosphodiesterase isozyme selectivity. *J. Med. Chem.* **64**, 4462–4477 (2021).
20. Chaturvedi, J., Haldar, C., Bisht, R., Pandey, G. & Chattopadhyay, B. Meta selective C–H borylation of sterically biased and unbiased substrates directed by electrostatic interaction. *J. Am. Chem. Soc.* **143**, 7604–7611 (2021).
21. Sun, X., Wang, M., Li, P., Zhang, X. & Wang, L. H₂O₂-mediated oxidative formation of amides from aromatic amines and 1,3-diketones as novel acylation agents via C–C bond cleavage at room temperature in water under metal-free conditions. *Green Chem.* **15**, 3289–3294 (2013).
22. Diao, T., Wadzinski, T. J. & Stahl, S. S. Direct aerobic α,β -dehydrogenation of aldehydes and ketones with a Pd(TFA)₂/4,5-diazafluorenone catalyst. *Chem. Sci.* **3**, 887–891 (2012).