Supporting Information for

Catalytic C−C Coupling of Diazo Compounds with Arylboronic Acids: Using Surface Modified Sewage Sludge as Greener Catalyst

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1. General considerations

All reactions and manipulations were performed using standard Schlenk techniques. ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra were recorded on a Bruker DRX-400 MHz spectrometer and all chemical shift values refer to $CDCl_3$ ($\delta(^{1}H)$, 7.26 ppm; $\delta(^{13}C)$, 77.16 ppm), $(CD_3)_2SO$ ($\delta(^{1}H)$, 2.50 ppm, $\delta(^{13}C)$, 39.52 ppm). X-ray Crystallographic analysis was achieved by the Analysis Center, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences. The GC analysis was obtained on Agilent 7890/5975C. The HRMS analysis was obtained on a Waters GC-TOF CA156 mass spectrometer. All the melting points were uncorrected. Column chromatographic purifications were performed on SDZF silica gel 160. All the chemical reagents were purchased from commercial sources and used as received unless otherwise indicated. Compounds **3a**,[1-4] **3c**,[3,5] **3d**,[2] **3f**,[3,4] **3g**,[5] **3h**,[6] **3i**,[2,7] **3j**,[7] **3k**,[7] **3l**,[5] **3m**,[4] **3o**,[7] **3z**,[8] **4a**,[9] **4e**,[10] **3z8**,[3,4] **3z8'**,[11] **5**,[12] **6**,[13] were known and their spectroscopic features were in good agreement with that reported in the literatures.

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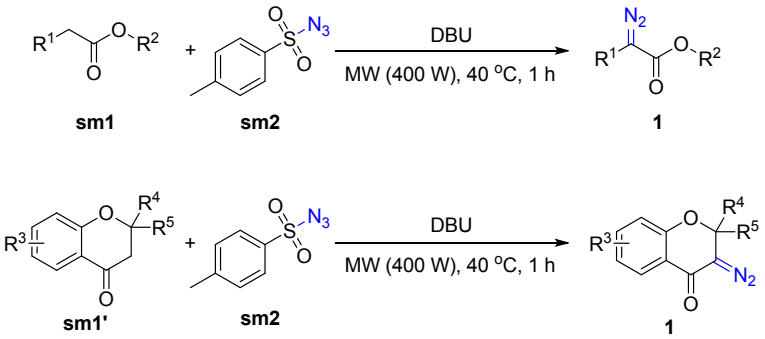
2. Experimental procedures

2.1 Preparation of SWs and Soil-Ⅰ catalysts.[14]

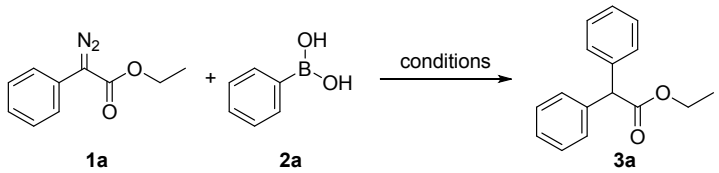
The surface of SW was modified by different kinds of acids or bases to gain SW catalysts. SW was prepared by using municipal sewage sludge from wastewater treatment plant (WWTP) in China. Sewage sludge was dried to constant weight at 105 °C and carbonized at 600 °C for 4 h under a heating rate of 3 °C min^{-1} and a high purity nitrogen (99.999 wt%) flow of 500 mL min^{-1}. After the furnace had cooled to room temperature, SW was obtained. Different kinds of acids or bases were used to treat the SW. In the acid or base treatment process, 50 mL of SW were produced by immersing carbonized SW with the same volume of $HClO_4$ (35.4 wt%), HCl (20.5 wt%), H_2SO_4 (63.4 wt%), H_3PO_4 (45.4 wt%), HNO_3 (40.5 wt%) and NaOH (41.5 wt%) for 24 h, respectively. Then, SW-Ⅰ ($HClO_4$), SW-Ⅱ (HCl), SW-Ⅲ (H_2SO_4), SW-Ⅳ (H_3PO_4), SW-Ⅴ (HNO_3) and SW-Ⅵ (NaOH) were washed with deionized water until the pH of the washing water reached 6-7 and the recovered solids were dried at room temperature. Soil-Ⅰ (common soil treated by $HClO_4$) was prepared by the same method of SW-Ⅰ.

2.2 Preparation for synthesis of α-diazoesters.[15]



A typical procedure for the synthesis of α-diazoesters 1 – *Synthesis of **1a***: DBU (2.24 mL, 15 mmol) was added slowly to a stirred solution of ethyl 2-phenylacetate (**sm1a**, 1.41 mL, 10.0 mmol) and tosylazide (**sm2**, 2.42 mL, 11.0 mmol) in the CH_3CN (20 mL) at 0 °C. After that, it was placed in microwave reactor that was heated to 40 °C (400 W, monitored by IR temperature sensor) and maintained at this temperature for 30 min. After cooling to room temperature, the reaction mixture was quenched with saturated aqueous solution of NH_4Cl (5 mL), extracted with DCM (3 × 30 mL), washed with brine (3 × 30 mL), dried over anhydrous Na_2SO_4, and concentrated under reduced pressure to give the product. The residue was purified by flash chromatography (petroleum ether (60-90 ºC)/AcOEt, 10:1) to afford the corresponding ethyl-2-diazo-2-phenylacetate **1a** as a yellow oil (1.65 g, 87%).

2.3 Screening the optimum reaction conditions for the synthesis of 3a



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

Table S1. Screening the optimum reaction conditions of **3a**[a]

Entry	Catalyst	1a:2a (equiv)	Solvent	Temp. (°C)	Yield[b] (%)
1	—	1:1.5	DCE	70	trace
2	SW[c]	1:1.5	DCE	70	19
3	SW-Ⅱ[c]	1:1.5	DCE	70	23
4	SW-Ⅲ[c]	1:1.5	DCE	70	20
5	SW-Ⅳ[c]	1:1.5	DCE	70	25
6	SW-Ⅴ[c]	1:1.5	DCE	70	22
7	SW-Ⅵ[c]	1:1.5	DCE	70	23
8	$HClO_4$[d]	1:1.5	DCE	70	12
8	SW-Ⅰ[c]	1:1.5	DCE	70	56
9	SW-Ⅰ	1:1.5	DCE	70	59
10	SW-Ⅰ[e]	1:1.5	DCE	70	45
11	SW-Ⅰ[f]	1:1.5	DCE	70	45
12	SW-Ⅰ[g]	1:1.5	DCE	70	47
13	SW-Ⅰ	1:1.5	DMF	70	19
14	SW-Ⅰ	1:1.5	Toluene	70	35
15	SW-Ⅰ	1:1.5	CH_3CN	70	25
16	SW-Ⅰ	1:1.5	DMSO	70	trace

17	SW- Ⅰ	1:1.5	1,4-dioxane	70	28
18	SW- Ⅰ	1:1.5	THF	70	29
19	SW- Ⅰ	1:1.5	DCE:DMF=2:1 (v:v)	70	25
20	SW- Ⅰ	1:1.5	DCE	rt	24
21	SW- Ⅰ	1:1.5	DCE	40	30
22	SW- Ⅰ	1:1.5	DCE	50	35
23	SW- Ⅰ	1:1.5	DCE	60	47
24	SW- Ⅰ	1:1.5	DCE	reflux	45
25	SW- Ⅰ	1:1	DCE	70	35
26	SW- Ⅰ	1:1.1	DCE	70	39
27	SW- Ⅰ	1:1.2	DCE	70	43
28	SW- Ⅰ	1:1.4	DCE	70	51
29	**SW- Ⅰ**	**1:1.6**	**DCE**	**70**	**61**
30	SW- Ⅰ	1:1.7	DCE	70	55
31	SW- Ⅰ	1:2	DCE	70	49
32[h]	SW- Ⅰ	1:1.6	DCE	70	46

[a]Conditions: **1a** (0.5 mmol), **2a**, catalyst (50 mg), solvent (5 mL), 0.1 MPa air, 6 h; symbol "-" means no catalyst. [b]Isolated yield by column chromatography with silica gel. [c]75 mg. [d]$HClO_4$ (2 mg). [e]25 mg. [f]100 mg. [g]150 mg. [h]SW-I reusing one time, 10 h.

2.4 The PH value of SW- Ⅰ

Table S2. The PH value of SW- Ⅰ

Entry	Solvent	SW- Ⅰ (mg)	PH value
1	water	0	7.17
2[a]	water	50	6.70
3	DCE	0	6.14
4[b]	DCE	50	6.01

Conditions: [a]Water (5 mL) + SW- Ⅰ (50 mg); [b]DCE (5 mL) + SW- Ⅰ (50 mg).

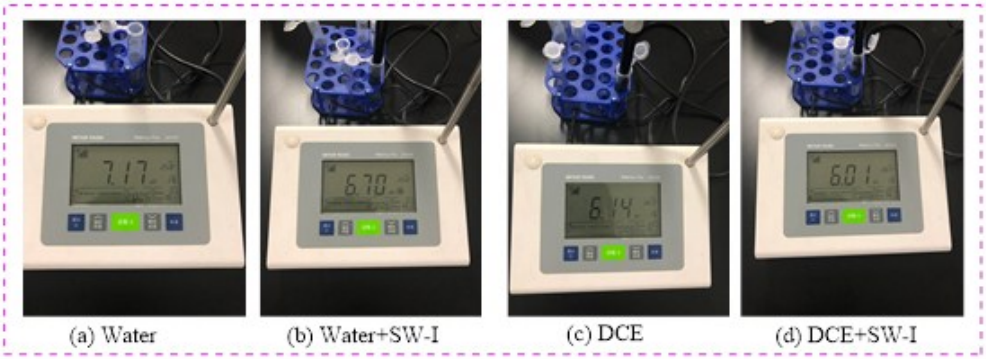
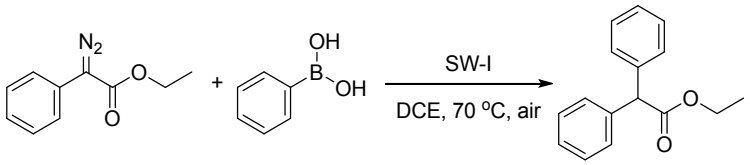


Figure S1. PH value (a) The PH value of water. (b) The PH value of SW- Ⅰ (50 mg)

in water (5 mL). (c) The PH value of DCE. (d) The PH value of SW-Ⅰ (50 mg) in DCE (5 mL).

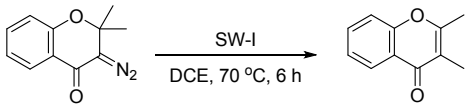
2.5 Typical C-C coupling procedures



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

A typical procedure for the synthesis of C-C coupling products procedures (3) – *Synthesis of **3a***: A mixture of SW-Ⅰ (50 mg), phenylboronic acid (**2a**, 98 mg, 0.8 mmol) and ethyl 2-diazo-2-phenylacetate (**1a**, 95 mg, 0.5 mmol) in DCE (5 mL) was stirred at room temperature under 0.1 Mpa air for 1 h, then the resulting mixture was stirred at 70 °C for 6 h. After filtrating, the filtrate was dissolved in DCM (20 mL). The DCM layer was washed with water (3 × 20 mL) and brine (3 × 20 mL). Then, the organic layer was dried over anhydrous Na_2SO_4 and concentrated to get the crude product. The crude product was purified by flash chromatography with petroleum ether (60-90 °C)/AcOEt (10:1) to afford **3a** as a colorless liquid (73 mg, 61%).

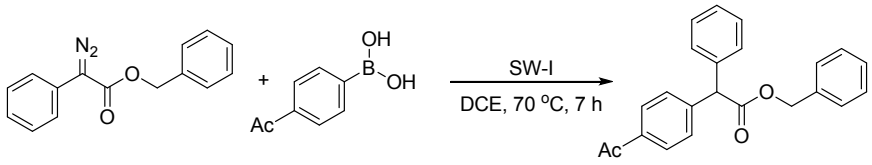
2.6 Typical procedure for 1,2-shift products of chromanone



1 **4a**

A typical procedure for the synthesis of 1,2-shift products of chromanone procedures (4) –*Synthesis of **4a**:* A mixture of SW-Ⅰ (50 mg), 3-diazo-2,2-dimethylchroman-4-one (**1t**, 101 mg, 0.5 mmol) in DCE (5 mL) was stirred at room temperature under 0.1 Mpa air for 1 h, then the resulting mixture was stirred at 70 °C for 6 h. After filtrating and removing solvent in vacuum the product was purified by flash chromatography with petroleum ether (60-90 °C)/AcOEt (10:1) to afford the corresponding **4a** as a white solid (65 mg, 75%).

2.7 A gram-scale experiment



A mixture of SW-Ⅰ (500 mg), (4-acetylphenyl)boronic acid (**2c**, 1.31 g, 8 mmol) and benzyl 2-diazo-2-phenylacetate (**1p**, 1.26 g, 5 mmol) in DCE (50 mL) was stirred at room temperature under 0.1 Mpa air for 1 h, then the resulting mixture was stirred at 70 °C for 7 h. After filtrating, the filtrate was dissolved in DCM (200 mL). The DCM layer was washed with water (3 × 200 mL) and brine (3 × 200 mL). Then, the organic layer was dried over anhydrous Na_2SO_4 and concentrated to get the crude product. The crude product was purified by flash chromatography with petroleum ether (60-90 °C)/AcOEt (10:1) to afford the corresponding **3z6** as a white solid (1.34 g, 78%).

2.8 The synthesis of the core structures of darifenacin and diclofensine

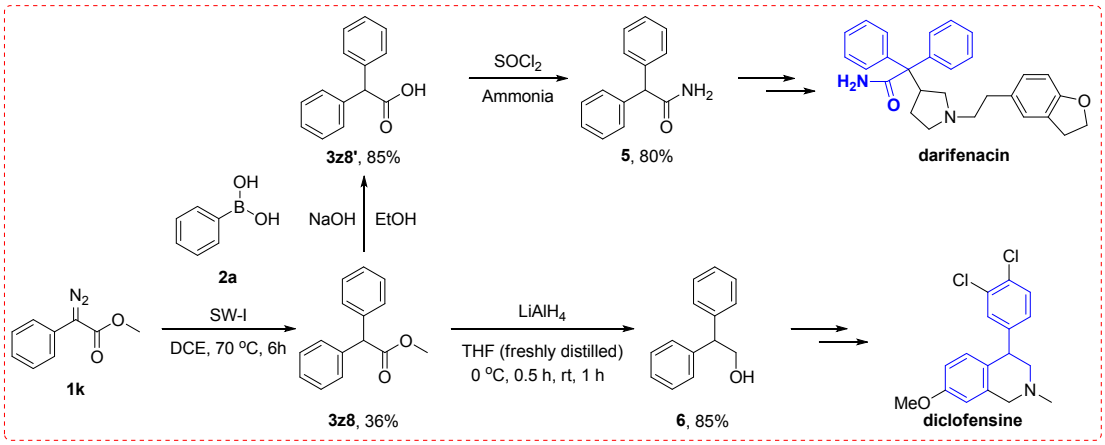


Figure. S2 Synthesis of the core structures of darifenacin and diclofensine.

Synthesis of 3z8. A mixture of SW-Ⅰ (250 mg), phenylboronic acid (**2a**, 490 mg, 4 mmol) and methyl 2-diazo-2-phenylacetate (**1k**, 440 mg, 2.5 mmol) in DCE (25 mL) was stirred at room temperature under 0.1 Mpa air for 1 h, then the resulting mixture was stirred at 70 °C for 6 h. After filtrating, the filtrate was dissolved in DCM (100

mL). The DCM layer was washed with water (3 × 100 mL) and brine (3 × 100 mL). Then, the organic layer was dried over anhydrous Na_2SO_4 and concentrated to get the crude product. The crude product was purified by flash chromatography with petroleum ether (60-90 ^oC)/AcOEt (10:1) to afford the corresponding **3z8** as a colorless liquid (203 mg, 36%).

Synthesis of 3z8'. A mixture of **3z8** (113 mg, 0.5 mmol) and NaOH (32 mg, 0.8 mmol) in EtOH (5 mL) was stirred at 85 ^oC reflux for 6 h. After completion of the reaction (monitored by TLC), adding slowly the 1 M HCl until the PH of reaction mixture reached 3-4. Then, 20 mL H_2O was added to the mixture. The aqueous layer was extracted with ethyl acetate (3 × 20 mL), washed with brine (3 × 20 mL), dried over anhydrous Na_2SO_4, concentrated under reduced pressure to give the crude product. The crude product was purified by flash chromatography with petroleum ether (60-90 ^oC)/AcOEt (3:1) to afford the corresponding **3z8'** as a white solid (90 mg, 85%).

Synthesis of 5. A mixture of **3z8'** (64 mg, 0.3 mmol) in toluene (3 mL, freshly distilled) was stirred at room temperature under 0.1 Mpa air for 1 min, adding slowly the $SOCl_2$ (118 mg, 1 mmol) to the resulting mixture. Then, the resulting mixture was stirred at 55 ^oC for 0.5 h and slowly added the ammonia (60 μL, ca. 4% in Methanol, ca. 2.0 mol/L) to it, stirring at the same temperature for 0.5 h. The resulting mixture was cooled to room temperature and dissolved in 15 mL of ethyl acetate. The ethyl acetate layer was washed with water (3 × 15 mL) and brine (3 × 15 mL). Then, the organic layer was dried over anhydrous Na_2SO_4 and concentrated to get the crude product. The crude product was purified by flash chromatography with petroleum ether (60-90 ^oC)/AcOEt (2:1) to afford the corresponding **5** as a white solid (51 mg, 80%).

Synthesis of 6. THF (3 mL, freshly distilled) was stirred at 0 ^oC under 0.1 Mpa air for 5 min, adding $LiAlH_4$ (19 mg, 0.5 mmol) to it and stirring at the same temperature for 5 min. Then, the compound **3z8** (68 mg, 0.3 mmol) as dissolved in 0.6 mL of freshly distilled THF and slowly added to the reaction mixture, stirring at the same

temperature for 0.5 h. Then, the reaction mixture was stirred at 25 °C for 1 h. After that, the reaction mixture was stirred at 0 °C for 5 min, 6 mL H_2O was added to it and stirred at 0 °C for 0.5 h. Last, extracting with ethyl acetate (3 × 10 mL), washed with brine (3 × 10 mL), dried over anhydrous Na_2SO_4, and concentrated under reduced pressure to give the crude product. The crude product was purified by flash chromatography with petroleum ether (60-90 °C)/AcOEt (3:1) to afford the corresponding **6** as a white solid (50 mg, 85%).

3. X-Ray crystallographic studies

Single crystals of compounds **4b** was grown in petroleum ether (60-90 °C)/CH_2Cl_2 (v/v, 5/1) at 25 °C and their X-ray diffraction studies were carried out on a SMART APEX diffractometer with graphite-monochromated Mo radiation (λ = 0.71073 Å). Cell parameters were obtained by global refinement of the positions of all collected reflections. Intensities were corrected for Lorentz and polarization effects and empirical absorption. The structures were solved by direct methods and refined by full-matrix least squares on F^2. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were placed in calculated positions. Structure solution and refinement were performed by using the SHELXL-97 package. The X-ray crystallographic files, in CIF format, are available from the Cambridge Crystallographic Data Centre on quoting the deposition numbers CCDC 1975819 for **4b**, Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 IEZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www. ccdc.cam.ac.uk).

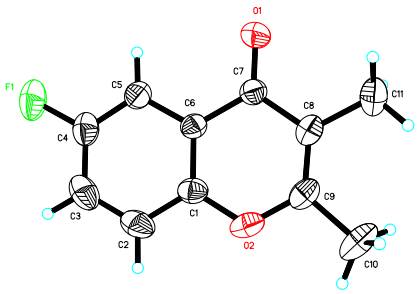


Figure S3. Molecular structure of compound **4b**

Table S3. Crystal data and structure refinement for **4b**

Empirical formula	$C_{11}H_9FO_2$	
Formula weight	192.18	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 8.2958(8) Å	□α = 90°
	b = 7.8128(7) Å	□β = 98.757(3)°
	c = 14.5059(16) Å	□γ = 90°
Volume	929.22(16) $Å^3$	
Z, Calculated density	4, 1.374 Mg/m^3	
Absorption coefficient	0.107 mm^{-1}	
F(000)	400	
Crystal size	0.200 x 0.160 x 0.130 mm^3	
Theta range for data collection	3.479 to 25.499°	
Index ranges	-9<=h<=10, -9<=k<=9, -17<=l<=15	
Reflections collected	9686	
Independent reflections	1716 [R(int) = 0.0321]	
Completeness to theta = 25.242°	99.2 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7456 and 0.6198	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	1716 / 0 / 129	
Goodness-of-fit on F^2	1.031	
Final R indices [I > 2 sigma(I)]	R1 = 0.0487, wR2 = 0.1343	
R indices (all data)	R1 = 0.0586, wR2 = 0.1458	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.180and -0.168 e.$Å^{-3}$	

4. NMR titration experiments

^{1}H-NMR titration experiments.

The preparation of mother liquor: (a) *p*-tolylboronic acid (0.3 mmol), (b) *p*-tolylboronic acid (0.3 mmol) with SW-Ⅰ (20 mg), (c) *p*-tolylboronic acid (0.3 mmol) with SW-Ⅰ (40 mg), (d) *p*-tolylboronic acid (0.3 mmol) with HCl (4 μL), (e) *p*-tolylboronic acid (0.3 mmol) with $HClO_4$ (4 μL). (f) *p*-tolylboronic (0.3 mmol) with Et_3N (4 μL), (g) *p*-tolylboronic (0.3 mmol) with $^iPr_2NH$ (4 μL), (h) *p*-tolylboronic (0.3

mmol) with $^{i}Pr_2NH$ (8 μL) were added in $(CD_3)_2SO$ (2 mL) respectively, stirred at room temperature under 0.1 Mpa air for 1 h. Then, the mother liquor of (a) 30 μL, (b) 50 μL, (c) 50 μL, (d) 50 μL, (e) 50 μL, (f) 50 μL, (g) 50 μL, (h) 50 μL were moved in $(CD_3)_2SO$ (0.5 mL), respectively. Last, conducted a ^1H NMR analysis to show the ^1H of OH in *p*-tolylboronic acid, as shown in Fig. S4-6.

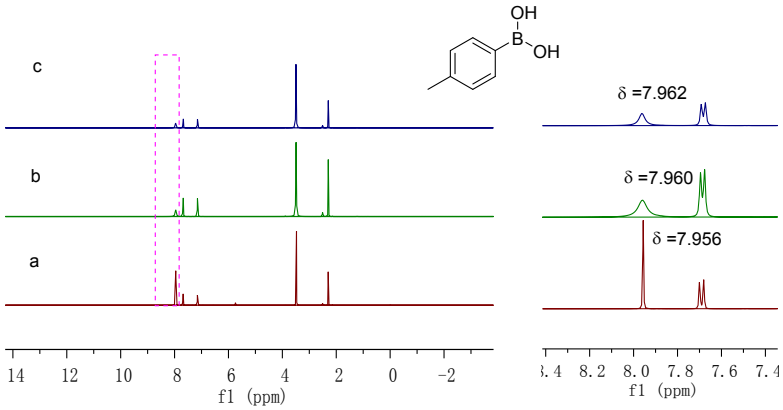


Figure S4. ^1H NMR spectra of the *p*-tolylboronic acid signals in $(CD_3)_2SO$. (a) *p*-tolylboronic acid (0.3 mmol), (b) *p*-tolylboronic acid (0.3 mmol) with SW-Ⅰ (20 mg), (c) *p*-tolylboronic acid (0.3 mmol) with SW-Ⅰ (40 mg).

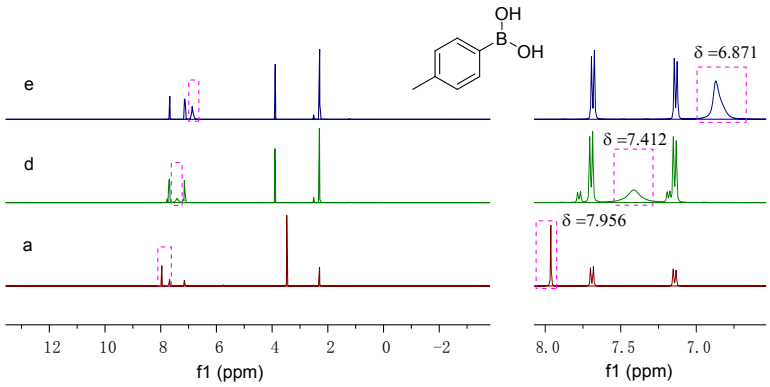


Figure S5. ^1H NMR spectra of the *p*-tolylboronic acid signals in $(CD_3)_2SO$. (d) *p*-tolylboronic acid (0.3 mmol) with HCl (4 μL), (e) *p*-tolylboronic acid (0.3 mmol) with $HClO_4$ (4 μL).

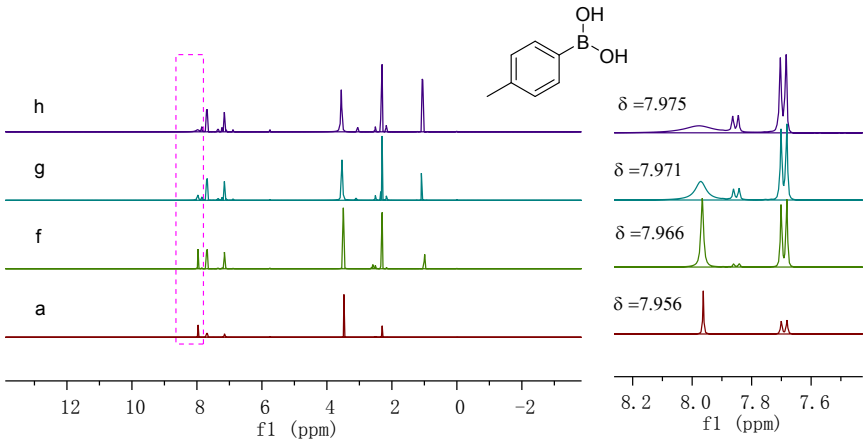


Figure S6. ^{1}H NMR spectra of the *p*-tolylboronic acid signals in $(CD_3)_2SO$. (f) *p*-tolylboronic (0.3 mmol) with Et_3N (4 μL), (g) *p*-tolylboronic (0.3 mmol) with $^iPr_2NH$ (4 μL), (h) *p*-tolylboronic (0.3 mmol) with $^iPr_2NH$ (8 μL)

5. The characterization of SW-Ⅰ, SW and Soil-Ⅰ

Catalyst characterization. X-ray Fluorescence (XRF, Magix 601) and Energy Dispersive Spectrometer (EDS, noran) were used to analyze the element content in the SW catalysts. The morphology and microstructure of SW-Ⅰ catalyst were examined by scanning electron microscope (SEM, HITACHI S4800) and transmission electron microscopy (TEM, JSM7500F).

Table S4. XRF analysis of elements in SW, SW-Ⅰ and Soil-Ⅰ

Chemical composition	SW (wt%)	SW-Ⅰ (wt%)	Soil-Ⅰ (wt%)	Chemical composition	SW (wt%)	SW-Ⅰ (wt%)	Soil-Ⅰ (wt%)
C	13.800	12.180	5.951	TiO_2	0.618	0.720	1.261
Na_2O	1.154	1.016	0.210	Cr_2O_3	0.047	0.040	0.029
MgO	2.490	1.527	0.879	MnO	0.109	0.040	0.090
Al_2O_3	15.207	15.043	19.858	Fe_2O_3	5.050	2.308	6.194
SiO_2	46.269	58.201	62.515	CuO	0.027	0.029	0.006
P_2O_5	2.059	0.458	0.070	ZnO	0.087	0.032	0.009
SO_3	1.377	1.396	0.036	Rb_2O	0.009	0.011	0.009
Cl	0.166	3.815	0.057	SrO	0.029	0.011	0.004
K_2O	1.796	2.123	2.309	Y_2O_3	0.003	0.002	0.001
CaO	9.683	1.018	0.077	ZrO_2	0.021	0.021	0.025

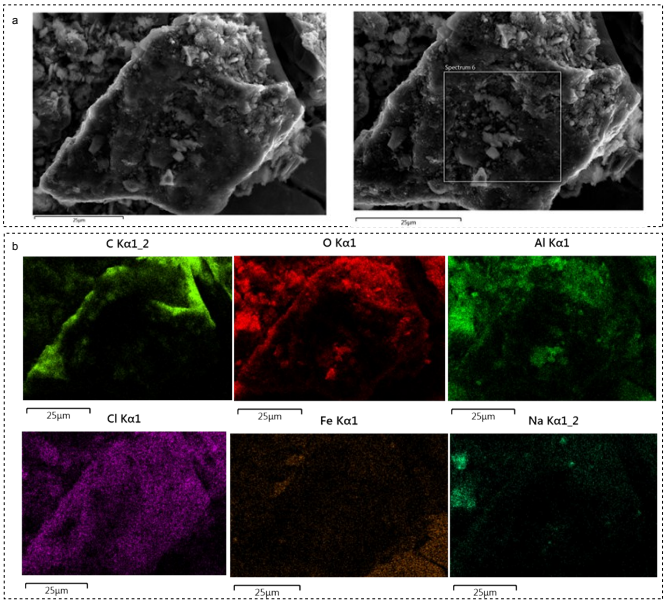


Figure S7. Properties of SW-Ⅰ. (a, b) SEM and EDS elemental mapping images.

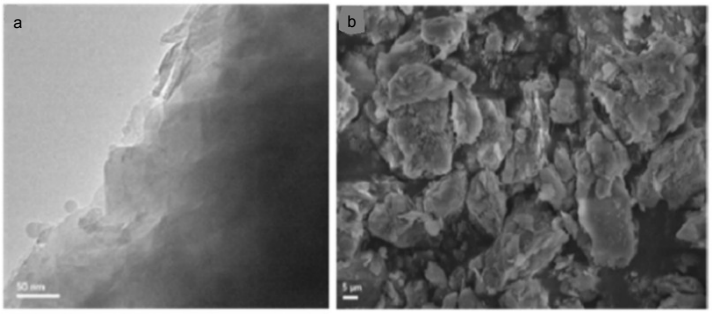
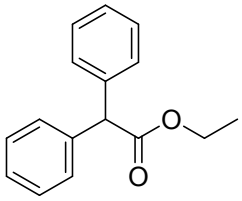
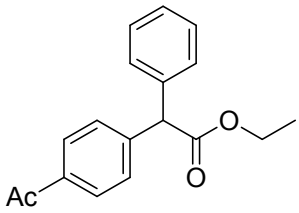


Figure S8. Properties of Soil-Ⅰ. (a, b) TEM and SEM images.

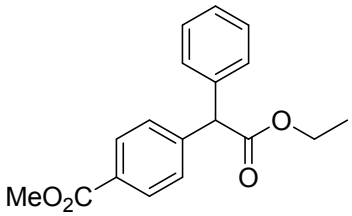
6. Analytical Data for known compounds



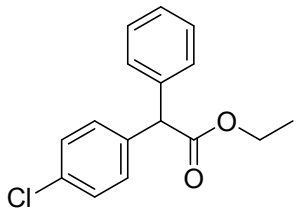
Ethyl 2,2-diphenylacetate (3a): Colorless liquid; 61% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (m, 8H), 7.19 (d, J = 3.7 Hz, 1H), 7.18–7.16 (m, 1H), 4.93 (s, 1H), 4.13 (q, J = 7.1 Hz, 2H), 1.17 (t, J = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 172.63, 138.89, 128.72, 128.70, 127.35, 61.34, 57.24, 14.27.



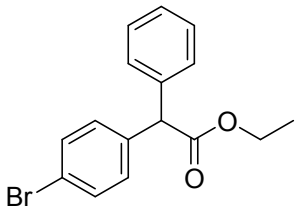
Ethyl 2-(4-acetylphenyl)-2-phenylacetate (3c): Colorless liquid; 83% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96–7.88 (m, 2H), 7.42 (d, J = 8.3 Hz, 2H), 7.36–7.26 (m, 5H), 5.06 (s, 1H), 4.22 (q, J = 7.1 Hz, 2H), 2.58 (s, 3H), 1.26 (t, J = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 197.79, 171.97, 144.18, 138.09, 136.16, 129.01, 128.90, 128.75, 128.65, 127.67, 61.59, 57.15, 26.76, 14.24.



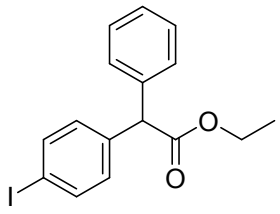
Methyl 4-(2-ethoxy-2-oxo-1-phenylethyl)benzoate (3d): Colorless liquid; 71% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, J = 8.4 Hz, 2H), 7.40 (d, J = 8.3 Hz, 2H), 7.36–7.26 (m, 5H), 5.05 (s, 1H), 4.22 (q, J = 7.1 Hz, 2H), 3.90 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 172.02, 166.95, 143.97, 138.18, 129.99, 129.26, 128.88, 128.83, 128.70, 127.65, 61.56, 57.21, 52.24, 14.25.



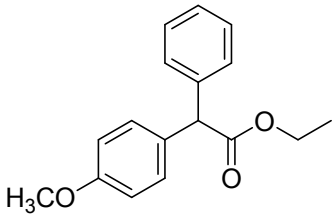
Ethyl 2-(4-chlorophenyl)-2-phenylacetate (3f): Colorless liquid; 56% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42–7.35 (m, 4H), 7.35–7.30 (m, 5H), 5.04 (s, 1H), 4.27 (q, J = 7.1 Hz, 2H), 1.32 (t, J = 7.1 Hz, 3H).^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 172.26, 138.47, 137.42, 133.32, 130.14, 128.84, 128.58, 127.55, 61.49, 56.58, 14.25.



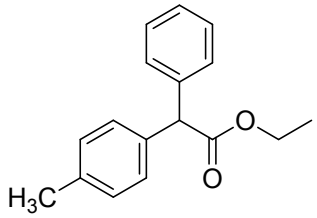
Ethyl 2-(4-bromophenyl)-2-phenylacetate (3g): Colorless liquid; 60% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (dd, *J* = 6.5, 4.6 Hz, 2H), 7.37–7.27 (m, 5H), 7.21 (dd, *J* = 6.3, 4.6 Hz, 2H), 4.97 (s, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 172.16, 138.37, 137.94, 131.78, 130.49, 128.83, 128.56, 127.56, 121.45, 61.48, 56.63, 14.24.



Ethyl 2-(4-iodophenyl)-2-phenylacetate (3h): Colorless liquid; 41% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.4 Hz, 2H), 7.36–7.26 (m, 5H), 7.07 (d, *J* = 8.2 Hz, 2H), 4.95 (s, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 172.13, 138.64, 138.33, 137.77, 130.76, 128.83, 128.57, 127.56, 93.04, 61.50, 56.74, 14.25.

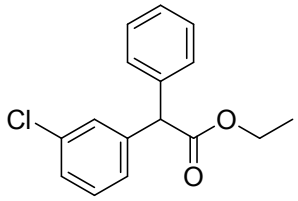


Ethyl 2-(4-methoxyphenyl)-2-phenylacetate (3i): Colorless liquid; 29% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36–7.31 (m, 4H), 7.29–7.25 (m, 3H), 6.88 (d, *J* = 8.8 Hz, 2H), 4.99 (s, 1H), 4.23 (q, *J* = 7.1 Hz, 2H), 3.81 (s, 3H), 1.28 (t, *J* = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 173.08, 159.07, 139.47, 131.23, 130.37, 130.00, 128.87, 128.79, 127.44, 114.29, 61.45, 56.64, 55.58, 14.48.

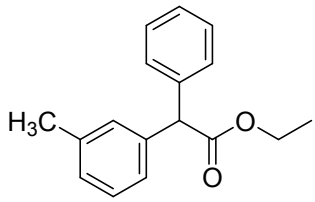


Ethyl 2-phenyl-2-(*p*-tolyl)acetate (3j): Colorless liquid; 43% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (t, *J* = 7.9 Hz, 4H), 7.25–7.17 (m, 3H), 7.11 (d, *J* = 8.0 Hz,

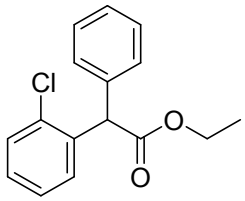
2H), 4.96 (s, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 2.30 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 172.77, 139.14, 137.00, 135.95, 129.40, 128.67, 128.67, 128.59, 127.26, 77.48, 77.16, 76.84, 61.26, 56.91, 21.17, 14.28.



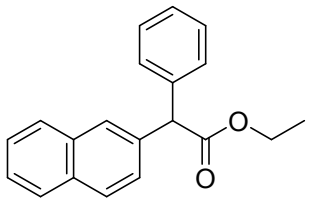
Ethyl 2-(3-chlorophenyl)-2-phenylacetate (3k)： Colorless liquid; 53% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37–7.27 (m, 6H), 7.25–7.20 (m, 3H), 4.97 (s, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 172.02, 140.83, 138.16, 134.50, 129.88, 128.92, 128.86, 128.62, 127.62, 127.58, 126.95, 61.52, 56.82, 14.23.



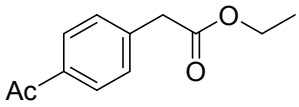
Ethyl 2-phenyl-2-(*m*-tolyl)acetate (3l): Colorless liquid; 41% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, *J* = 4.4 Hz, 4H), 7.25–7.17 (m, 2H), 7.14–7.03 (m, 3H), 4.96 (s, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 2.31 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 172.69, 139.00, 138.78, 138.34, 129.42, 128.72, 128.66, 128.58, 128.13, 127.28, 125.73, 61.27, 57.22, 21.59, 14.27.



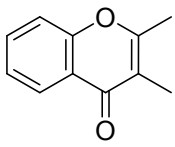
Ethyl 2-(2-chlorophenyl)-2-phenylacetate (3m)： Colorless liquid; 35% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 6.5 Hz, 2H), 7.45–7.34 (m, 4H), 7.15 (td, *J* = 8.2, 1.6 Hz, 1H), 6.94 (td, *J* = 7.7, 1.3 Hz, 1H), 6.87 (dd, *J* = 8.2, 1.2 Hz, 1H), 5.66 (s, 1H), 4.24–4.14 (m, 2H), 1.19 (t, *J* = 7.1 Hz, 3H).$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 169.58, 153.14, 135.23, 130.80, 129.14, 128.88, 127.68, 127.13, 124.23, 122.85, 115.35, 79.64, 61.86, 14.13.



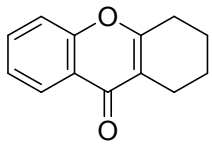
Ethyl 2-(naphthalen-2-yl)-2-phenylacetate (3o): Colorless liquid; 69% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (dd, *J* = 8.8, 3.7 Hz, 4H), 7.49–7.43 (m, 3H), 7.38–7.31 (m, 4H), 7.30–7.26 (m, 1H), 5.19 (s, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 1.27 (t, *J* = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 181.23, 138.83, 136.34, 133.48, 132.65, 128.83, 128.75, 128.42, 128.10, 127.73, 127.42, 127.38, 126.95, 126.30, 126.12, 61.40, 57.37, 14.30. HRMS (ESI) calcd for $C_{20}H_{19}O_2$ $[M+H]^+$: 291.1380; Found: 291.1392.



Ethyl 2-(4-acetylphenyl) acetate (3z): White solid; m.p.: 53-55 °C; 64% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.3 Hz, 2H), 7.37 (d, *J* = 8.3 Hz, 2H), 4.14 (q, *J* = 7.1 Hz, 2H), 3.66 (s, 2H), 2.58 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 197.83, 170.90, 139.58, 136.03, 129.63, 128.69, 61.21, 41.38, 26.71, 14.23.

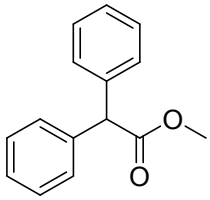


2,3-dimethyl-4H-chromen-4-one (4a): White solid; m.p.: 90-92 °C; 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.55 (ddd, *J* = 8.6, 7.1, 1.7 Hz, 1H), 7.34–7.26 (m, 2H), 2.35 (s, 3H), 2.01 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 177.67, 161.78, 155.69, 132.77, 125.61, 124.30, 122.43, 117.45, 116.71, 18.41, 9.91. HRMS (ESI) calcd for $C_{11}H_{11}O_2$ $[M+H]^+$: 175.0754; Found: 175.0766.

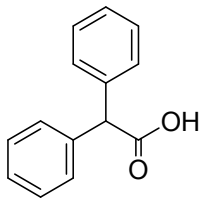


3,4-dihydro-1*H*-xanthen-9(2*H*)-one (4e): White solid; m.p.: 90-92 °C; 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.61–7.54 (m, 1H),

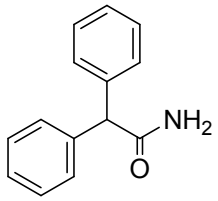
7.37–7.27 (m, 2H), 2.64 (t, J = 6.3 Hz, 2H), 2.56 (t, J = 6.2 Hz, 2H), 1.89–1.81 (m, 2H), 1.77–1.70 (m, 2H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 163.89, 155.95, 132.98, 125.76, 124.41, 123.22, 118.46, 117.66, 28.22, 21.97, 21.72, 21.07.



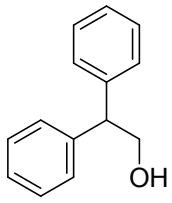
Methyl 2,2-diphenylacetate (3z8): Colorless liquid; 36% yield; ^1H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, J = 4.4 Hz, 8H), 7.33–7.27 (m, 2H), 5.09 (s, 1H), 3.78 (s, 3H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 173.07, 138.72, 128.72, 128.70, 127.40, 57.12, 52.43.



2,2-diphenylacetic acid (3z8'): White solid; m.p.: 146-148 °C; 85% yield; ^1H NMR (400 MHz, $CDCl_3$) δ 10.75 (s, 1H), 7.34 (d, J = 4.4 Hz, 8H), 7.32–7.27 (m, 2H), 5.06 (s, 1H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 178.56, 138.02, 128.81, 127.65, 57.10.

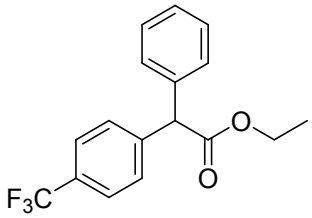


2,2-diphenylacetamide (5): White solid; m.p.: 163-166 °C; 80% yield; ^1H NMR (400 MHz, $(CD_3)_2SO$) δ 7.32 (d, J = 4.3 Hz, 10H), 7.25 (dt, J = 8.8, 4.2 Hz, 2H), 5.06 (s, 1H). $^{13}C\{^1H\}$ NMR (100 MHz, $(CD_3)_2SO$) δ 173.43, 139.62, 128.53, 128.43, 126.87, 56.31. HRMS (ESI) calcd for $C_{14}H_{13}NONa$ $[M+Na]^+$: 234.0889; Found: 234.0888.

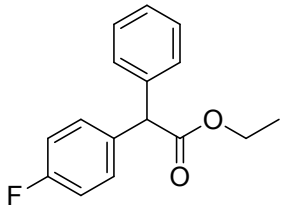


2,2-diphenylethanol (6): White solid; m.p.: 55-57 ^{o}C; 85% yield;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40–7.34 (m, 4H), 7.34–7.27 (m, 6H), 4.28–4.19 (m, 3H), 1.63 (s, 1H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 141.50, 128.84, 128.44, 126.94, 66.25, 53.76.

7. Analytical Data for unknown compounds

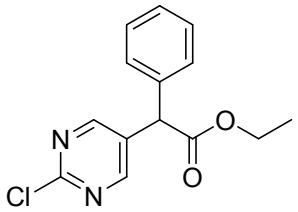


Ethyl 2-phenyl-2-(4-(trifluoromethyl)phenyl)acetate (3b): Colorless liquid; 70% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.2 Hz, 2H), 7.47 (d, *J* = 8.3 Hz, 2H), 7.39–7.29 (m, 5H), 5.08 (s, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 1.28 (t, *J* = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 171.94, 142.89, 138.05, 129.64 (q, *J* = 32.5 Hz), 129.17, 128.93, 128.62, 127.72, 125.62 (q, *J* = 3.7 Hz), 121.52 (q, *J* = 272.0 Hz), 61.61, 57.00, 14.20. HRMS (ESI) calcd for $C_{17}H_{16}F_3O_2$ $[M+H]^+$: 309.1097; Found: 309.1096.

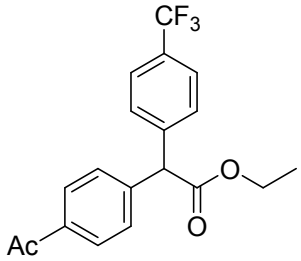


Ethyl 2-(4-fluorophenyl)-2-phenylacetate (3e): Colorless liquid; 59% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (dd, *J* = 7.6, 1.4 Hz, 2H), 7.44–7.34 (m, 3H), 7.22 (d, *J* = 9.0 Hz, 2H), 6.89 (d, *J* = 9.0 Hz, 2H), 5.57 (s, 1H), 4.26–4.12 (m, 2H), 1.21 (t, *J* = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 172.51, 162.14 (d, *J* = 245.9 Hz), 138.76, 134.69 (d, *J* = 3.3 Hz), 130.36 (d, *J* = 8.0 Hz), 128.81, 128.56, 127.48, 115.53

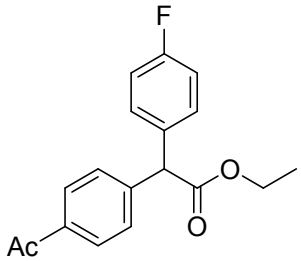
(d, J = 21.4 Hz), 61.42, 56.44, 14.25. HRMS (ESI) calcd for $C_{16}H_{19}FNO_2$ $[M+NH_4]^+$: 276.1394; Found: 276.1391.



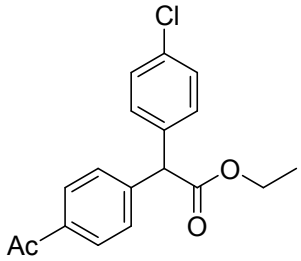
Ethyl 2-(2-chloropyrimidin-5-yl)-2-phenylacetate (3p): Colorless liquid; 73% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (s, 2H), 7.34–7.28 (m, 3H), 7.23 (dd, J = 8.3, 1.4 Hz, 2H), 4.91 (s, 1H), 4.16–4.23 (m, 2H), 1.22 (t, J = 7.1 Hz, 3H) .^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 170.66, 160.41, 159.70, 136.34, 131.33, 129.45, 128.38, 128.18, 62.24, 52.12, 14.15. HRMS (ESI) calcd for $C_{14}H_{14}ClN_2O_2$ $[M+H]^+$: 277.0738; Found: 277.0746.



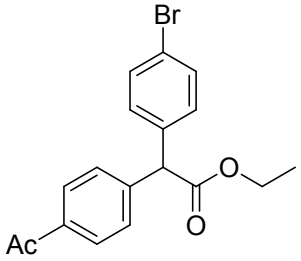
Ethyl 2-(4-acetylphenyl)-2-(4-(trifluoromethyl)phenyl)acetate (3r): Light yellow liquid; 55% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96–7.91 (m, 2H), 7.59 (d, J = 8.2 Hz, 2H), 7.45–7.41 (m, 3H), 7.40 (s, 1H), 5.11 (s, 1H), 4.24 (q, J = 7.1 Hz, 2H), 2.58 (s, 3H), 1.27 (t, J = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 197.65, 171.33, 143.18, 142.02, 136.52, 130.02 (q, J = 32.6 Hz), 129.14, 128.96, 125.84 (q, J = 3.7 Hz), 125.47 (q, J = 272.3 Hz), 61.92, 56.89, 26.75, 14.22. HRMS (ESI) calcd for $C_{19}H_{18}F_3O_3$ $[M+H]^+$: 351.1203; Found: 351.1218.



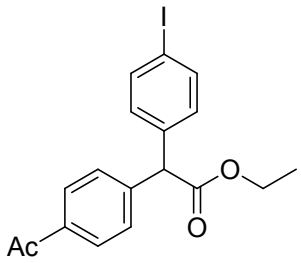
Ethyl 2-(4-acetylphenyl)-2-(4-fluorophenyl)acetate (3s): Light yellow liquid; 78% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.4 Hz, 2H), 7.40 (d, *J* = 8.2 Hz, 2H), 7.32–7.24 (m, 2H), 7.06–6.97 (m, 2H), 5.05 (s, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.58 (s, 3H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 197.63, 171.80, 162.20 (d, *J* = 246.6 Hz), 143.92, 136.22, 133.84 (d, *J* = 3.3 Hz), 130.29 (d, *J* = 8.1 Hz), 128.80, 128.78, 115.70 (d, *J* = 21.5 Hz), 61.63, 56.25, 26.67, 14.16. HRMS (ESI) calcd for $C_{18}H_{18}FO_3$ $[M+H]^+$: 300.1234; Found: 300.1226.



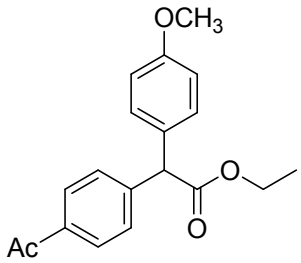
Ethyl 2-(4-acetylphenyl)-2-(4-chlorophenyl)acetate (3t): Colorless liquid; 79% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.2 Hz, 2H), 7.33–7.27 (m, 2H), 7.27–7.21 (m, 2H), 5.02 (s, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.57 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 197.65, 171.60, 143.64, 136.58, 136.33, 133.67, 130.06, 129.02, 128.86, 128.84, 61.73, 56.44, 26.71, 14.20. HRMS (ESI) calcd for $C_{18}H_{18}ClO_3$ $[M+H]^+$: 317.0939; Found: 317.0925.



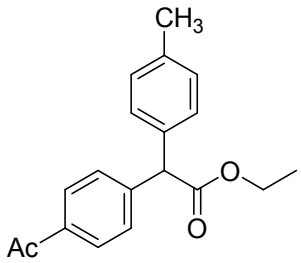
ethyl 2-(4-acetylphenyl)-2-(4-bromophenyl)acetate (3u): Light yellow liquid ; 81% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.48–7.43 (m, 2H), 7.39 (d, *J* = 8.2 Hz, 2H), 7.21–7.15 (m, 2H), 5.01 (s, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.57 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 197.65, 171.52, 143.54, 137.10, 136.34, 131.98, 130.40, 128.86, 128.84, 121.80, 61.75, 56.50, 26.71, 14.20. HRMS (ESI) calcd for $C_{18}H_{18}BrO_3$ $[M+H]^+$: 361.0434; Found: 361.0447.



Ethyl 2-(4-acetylphenyl)-2-(4-iodophenyl)acetate (3v): Colorless liquid; 65% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.65 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.2 Hz, 2H), 7.09–7.02 (m, 2H), 4.99 (s, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 2.57 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 197.63, 171.47, 143.49, 137.94, 137.78, 136.33, 130.64, 128.86, 128.83, 93.38, 61.74, 56.60, 26.72, 14.20. HRMS (ESI) calcd for $C_{18}H_{18}IO_3$ $[M+H]^+$: 409.0295; Found: 409.0307.

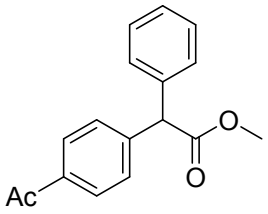


Ethyl 2-(4-acetylphenyl)-2-(4-methoxyphenyl)acetate (3w): Colorless liquid; 72% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.40 (d, *J* = 8.3 Hz, 2H), 7.23 (d, *J* = 8.7 Hz, 2H), 6.89–6.82 (m, 2H), 5.01 (s, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 3.78 (s, 3H), 2.57 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 197.74, 172.19, 159.05, 144.55, 136.07, 130.16, 129.72, 128.84, 128.69, 114.24, 61.47, 56.31, 55.34, 26.68, 14.21. HRMS (ESI) calcd for $C_{19}H_{21}O_4$ $[M+H]^+$: 313.1434; Found: 313.1423.

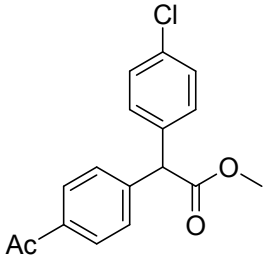


2-(2-Ethoxy-2-oxo-1-phenylethoxy) benzoic acid (3x): Colorless liquid; 76% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95–7.87 (m, 2H), 7.43 (d, *J* = 8.2 Hz, 2H),

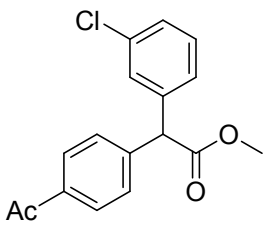
7.21 (d, J = 8.1 Hz, 2H), 7.15 (d, J = 8.0 Hz, 2H), 5.04 (s, 1H), 4.22 (q, J = 7.1 Hz, 2H), 2.57 (s, 3H), 2.33 (s, 3H), 1.26 (t, J = 7.1 Hz, 3H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 197.66, 172.02, 144.36, 137.28, 136.02, 135.07, 129.50, 128.87, 128.63, 128.43, 61.41, 56.70, 26.62, 21.07, 14.16. HRMS (ESI) calcd for $C_{19}H_{21}O_3$ $[M+H]^+$: 297.1485; Found: 297.1473.



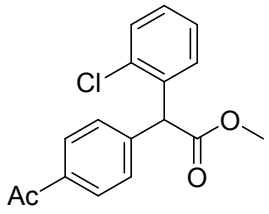
Methyl 2-(4-acetylphenyl)-2-phenylacetate (3z1): Colorless liquid; 80% yield; ^1H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 8.4 Hz, 2H), 7.42 (d, J = 8.3 Hz, 2H), 7.37–7.27 (m, 5H), 5.09 (s, 1H), 3.76 (s, 3H), 2.57 (s, 3H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 197.71, 172.42, 143.97, 137.93, 136.21, 128.99, 128.91, 128.75, 128.63, 127.72, 56.99, 52.60, 26.70. HRMS (ESI) calcd for $C_{17}H_{16}O_3K$ $[M+K]^+$: 307.0731; Found: 307.0746.



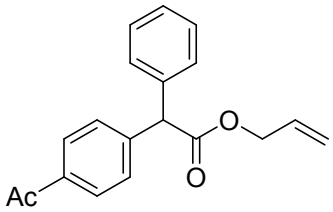
Methyl 2-(4-acetylphenyl)-2-(4-chlorophenyl)acetate (3z2): Colorless liquid; 85% yield; ^1H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 8.3 Hz, 2H), 7.38 (d, J = 8.3 Hz, 2H), 7.33–7.29 (m, 2H), 7.23 (d, J = 8.5 Hz, 2H), 5.05 (s, 1H), 3.76 (s, 3H), 2.58 (s, 3H). $^{13}C\{^1H\}$ (100 MHz, $CDCl_3$) δ 197.67, 172.13, 143.47, 136.42, 136.40, 133.79, 130.07, 129.09, 128.90, 128.89, 56.31, 52.79, 26.77. HRMS (ESI) calcd for $C_{17}H_{19}ClNO_3$ $[M+NH_4]^+$: 320.1048; Found:.320.1059



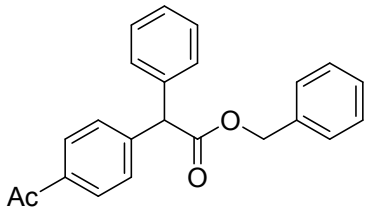
Methyl 2-(4-acetylphenyl)-2-(3-chlorophenyl)acetate (3z3): Colorless liquid; 73% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99–7.89 (m, 2H), 7.43 (d, *J* = 8.2 Hz, 2H), 7.32 (s, 1H), 7.28 (dd, *J* = 3.8, 1.5 Hz, 2H), 7.24–7.17 (m, 1H), 5.07 (s, 1H), 3.79 (s, 3H), 2.60 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 197.59, 171.83, 143.13, 139.81, 136.41, 134.70, 130.09, 128.89, 128.86, 128.81, 127.93, 126.86, 56.49, 52.74, 26.68. HRMS (ESI) calcd for $C_{17}H_{16}ClO_3$ $[M+H]^+$: 303.0782; Found:.303.0797.



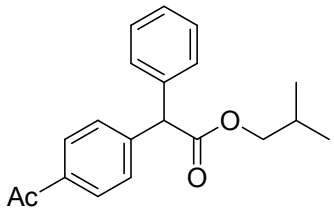
Ethyl 2-(4-allylphenoxy)-2-phenylacetate (3z4): Colorless liquid; 61% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.3 Hz, 1H), 7.32 (dd, *J* = 8.3, 5.9 Hz, 3H), 7.24–7.13 (m, 4H), 5.47 (s, 1H), 3.69 (s, 3H), 2.51 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 197.71, 171.95, 142.50, 136.45, 135.87, 134.37, 129.97, 129.94, 129.35, 129.09, 128.87, 127.26, 53.71, 52.83, 26.75. HRMS (ESI) calcd for $C_{17}H_{19}ClNO_3$ $[M+NH_4]^+$: 320.1048; Found:.320.1059.



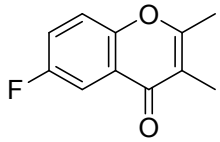
Allyl 2-(4-acetylphenyl)-2-phenylacetate (3z5): Colorless liquid; 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.4 Hz, 2H), 7.43 (d, *J* = 8.3 Hz, 2H), 7.36–7.27 (m, 5H), 5.89 (dd, *J* = 17.2, 10.5 Hz, 1H), 5.30–5.18 (m, 2H), 5.11 (s, 1H), 4.67 (d, *J* = 5.7 Hz, 2H), 2.57 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 197.70, 171.58, 143.95, 137.90, 136.20, 131.74, 128.99, 128.88, 128.73, 128.64, 127.70, 118.84, 66.05, 57.04, 26.69. HRMS (ESI) calcd for $C_{19}H_{19}O_3$ $[M+H]^+$: 295.1329; Found: 295.1342.



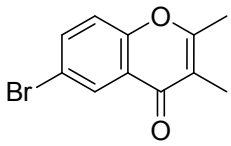
Benzyl 2-(4-acetylphenyl)-2-phenylacetate (3z6): White solid; m.p.: 66-67 °C; 80% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.40 (d, *J* = 8.3 Hz, 2H), 7.36–7.31 (m, 5H), 7.30–7.26 (m, 5H), 5.20 (s, 2H), 5.13 (s, 1H), 2.58 (s, 3H). ^{13}C{^{1}H} (100 MHz, $CDCl_3$) δ 197.77, 171.79, 143.94, 137.89, 136.20, 135.56, 129.03, 128.91, 128.75, 128.70, 128.68, 128.49, 128.37, 127.72, 67.30, 57.06, 26.74. HRMS (ESI) calcd for $C_{23}H_{24}NO_3$ $[M+NH_4]^+$: 362.1751; Found: 362.1764.



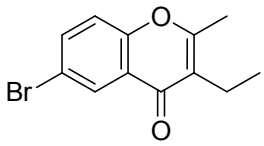
Isobutyl 2-(4-acetylphenyl)-2-phenylacetate (3z7): Light yellow liquid; 72% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94–7.88 (m, 2H), 7.43 (d, *J* = 8.3 Hz, 2H), 7.36–7.27 (m, 5H), 5.09 (s, 1H), 3.95 (d, *J* = 6.6 Hz, 2H), 2.57 (s, 3H), 1.99–1.87 (m, 1H), 0.86 (d, *J* = 6.7 Hz, 6H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 197.66, 171.90, 144.15, 138.09, 136.10, 128.99, 128.79, 128.65, 128.64, 128.55, 127.58, 71.52, 57.24, 27.75, 26.65, 19.05. HRMS (ESI) calcd for $C_{20}H_{26}NO_3$ $[M+NH_4]^+$: 328.1907; Found: 328.1919.



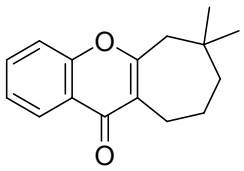
6-fluoro-2,3-dimethyl-4*H*-chromen-4-one (4b): White solid, m.p.: 83-85 °C; 85% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (dd, *J* = 8.4, 3.0 Hz, 1H), 7.41–7.28 (m, 2H), 2.41 (s, 3H), 2.04 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 177.31, 162.36, 159.29 (d, *J* = 245.3 Hz), 152.23, 123.77 (d, *J* = 7.3 Hz), 121.28 (d, *J* = 25.6 Hz), 119.76 (d, *J* = 8.0 Hz), 116.55, 110.64 (d, *J* = 23.5 Hz), 18.69, 10.16. HRMS (ESI) calcd for $C_{11}H_{10}FO_2$ $[M+H]^+$: 193.0659; Found: 193.0670.



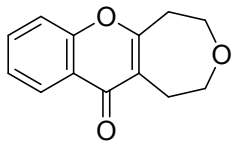
6-bromo-2,3-dimethyl-4*H*-chromen-4-one (4c): White solid, m.p.: 91-92 °C.; 80% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, *J* = 2.4 Hz, 1H), 7.61 (dd, *J* = 8.9, 2.5 Hz, 1H), 7.21 (d, *J* = 8.9 Hz, 1H), 2.37 (s, 3H), 2.00 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 176.54, 162.28, 154.61, 135.88, 128.40, 123.92, 119.67, 117.77, 117.22, 18.64, 10.14. HRMS (ESI) calcd for $C_{11}H_{10}BrO_2$ $[M+H]^+$: 252.9859; Found: 252.9868.



6-bromo-3-ethyl-2-methyl-4*H*-chromen-4-one (4d): White solid, m.p.: 67-68 °C; 74% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (d, *J* = 2.4 Hz, 1H), 7.68 (dd, *J* = 8.9, 2.5 Hz, 1H), 7.30 (d, *J* = 8.9 Hz, 1H), 2.74 (q, *J* = 7.6 Hz, 2H), 2.07 (s, 3H), 1.32 (t, *J* = 7.6 Hz, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 177.03, 166.61, 154.79, 135.96, 128.50, 123.99, 119.76, 117.81, 116.44, 25.75, 11.41, 9.74. HRMS (ESI) calcd for $C_{12}H_{12}BrO_2$ $[M+H]^+$: 267.0015; Found: 267.0023.



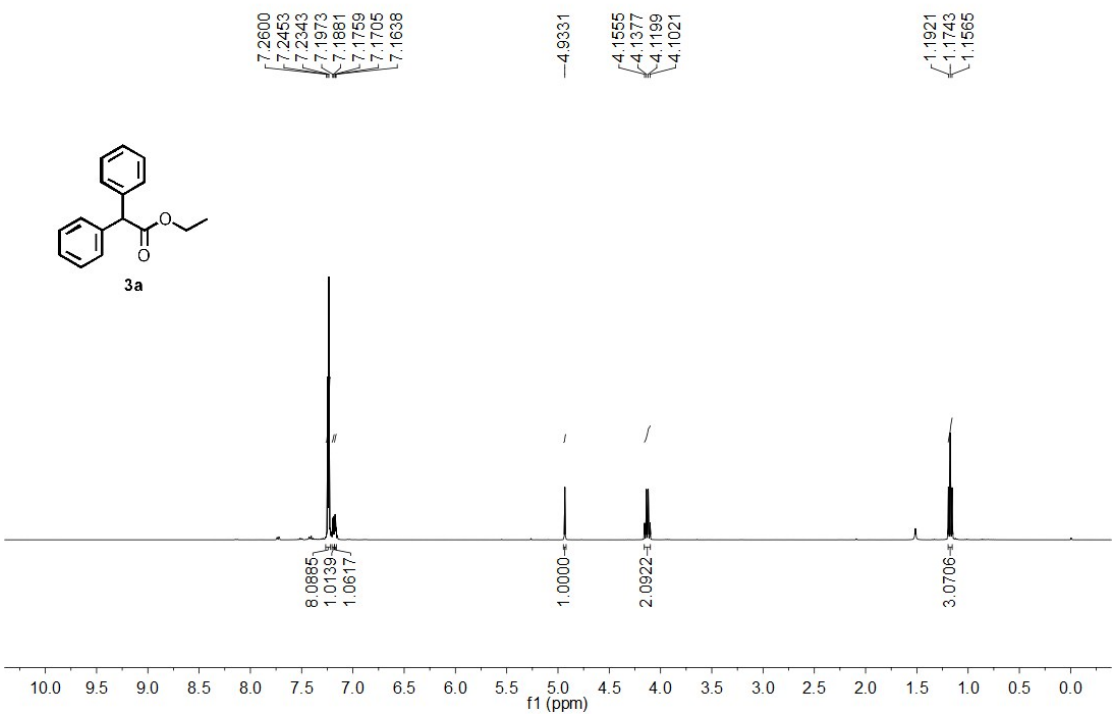
7,7-dimethyl-7,8,9,10-tetrahydrocyclohepta[b]chromen-11(6*H*)-one (4f): White solid; m.p.: 94-96 °C; 69% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.62–7.57 (m, 1H), 7.40–7.32 (m, 2H), 2.85–2.77 (m, 2H), 2.67 (s, 2H), 1.78–1.73 (m, 2H), 1.62 (dd, *J* = 7.6, 4.1 Hz, 2H), 0.94 (s, 6H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 177.50, 168.35, 155.84, 132.91, 126.31, 124.70, 122.86, 119.77, 117.87, 45.34, 34.56, 34.30, 31.13, 28.40, 21.23. HRMS (ESI) calcd for $C_{16}H_{19}O_2$ $[M+H]^+$: 243.1380; Found: 243.1394.



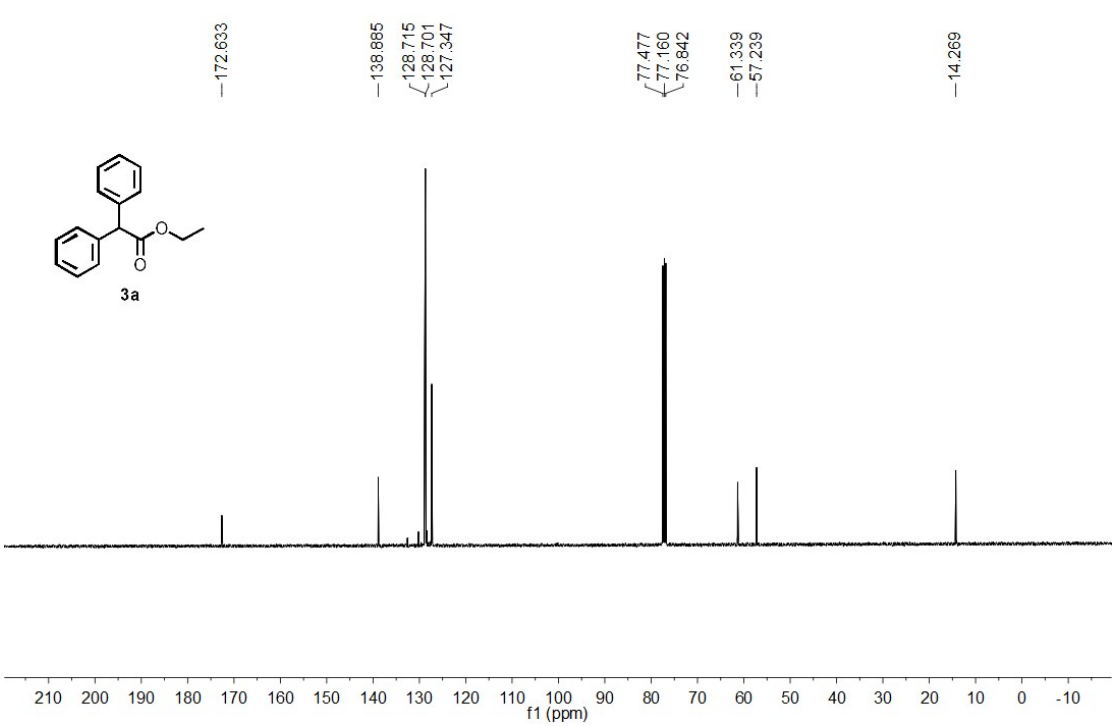
4,5-dihydro-1*H*-oxepino[4,5-*b*]chromen-11(2*H*)-one (4g): White solid, m.p.: 125-127 ^{o}C; 58% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.63 (ddd, *J* = 8.6, 7.1, 1.7 Hz, 1H), 7.41–7.35 (m, 2H), 3.90–3.86 (m, 2H), 3.81–3.77 (m, 2H), 3.10–3.06 (m, 2H), 3.04–3.00 (m, 2H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 177.15, 167.36, 155.96, 133.36, 126.25, 125.05, 122.64, 122.09, 117.90, 70.19, 66.90, 38.36, 25.47. HRMS (ESI) calcd for $C_{13}H_{12}KO_3$ $[M+K]^+$: 255.0418; Found: 255.0426.

8. Copies of NMR spectra

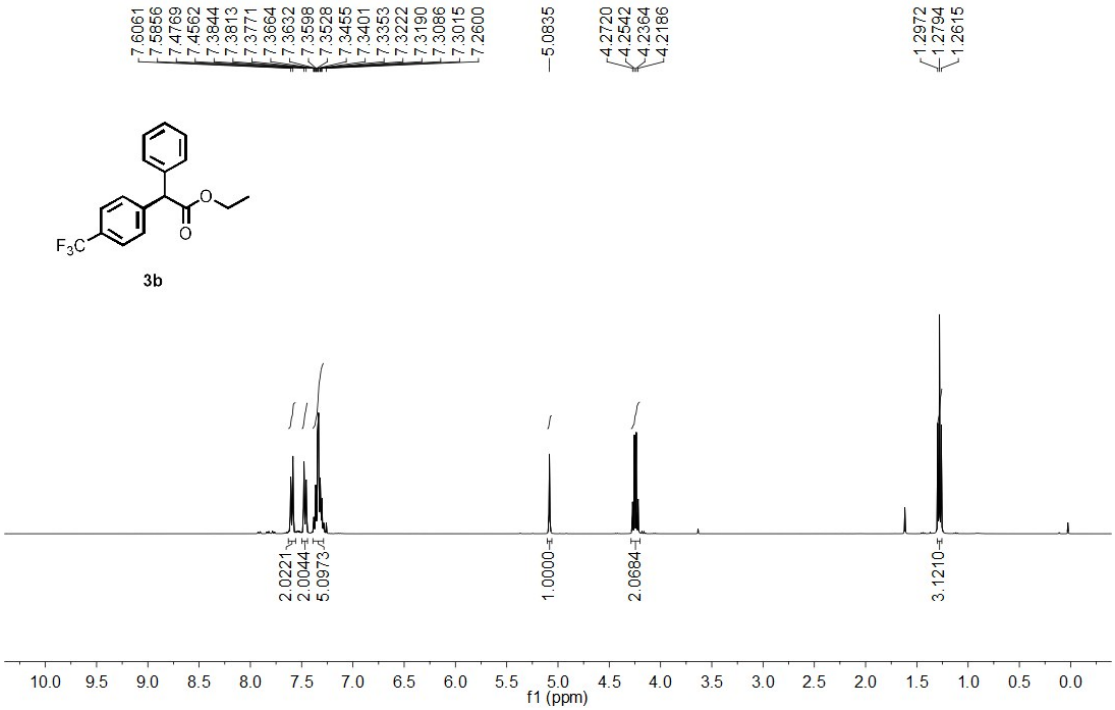
ZZP-74



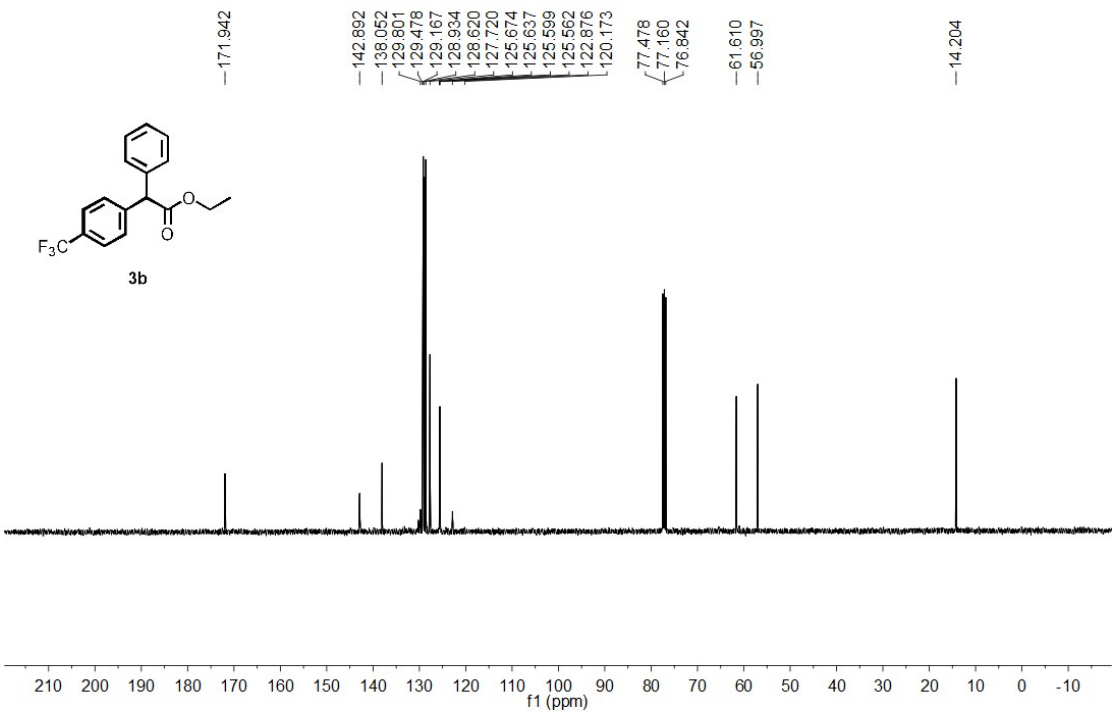
ZZP-74



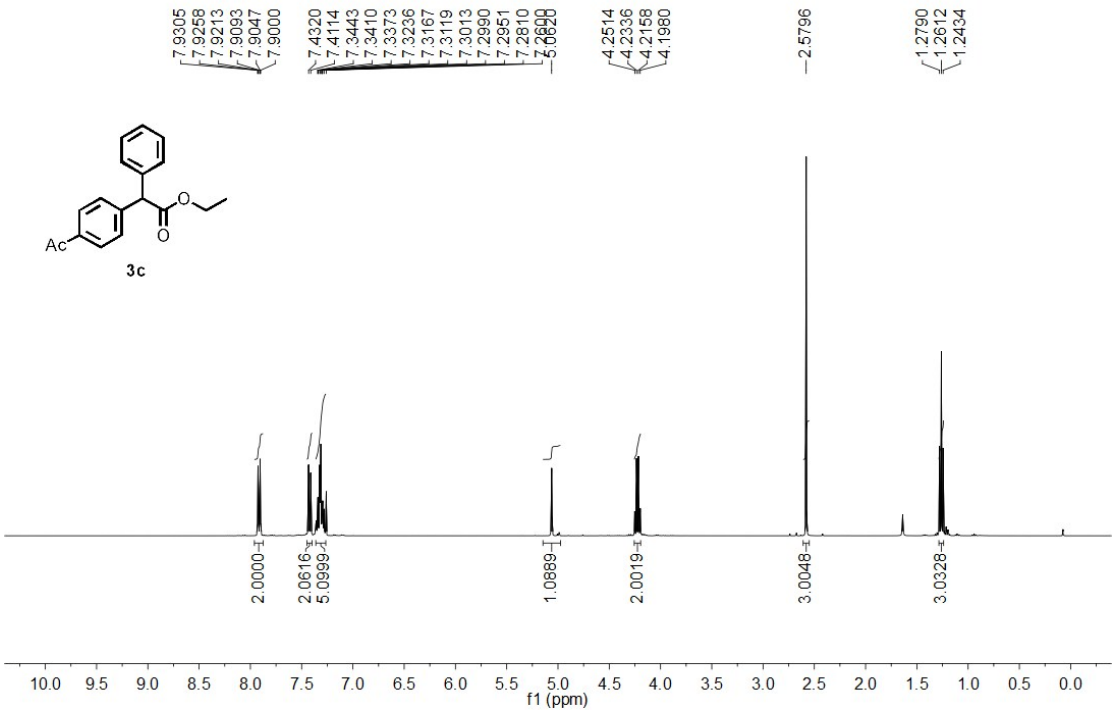
ZZP-99-1



ZZP-99-2



ZZP-62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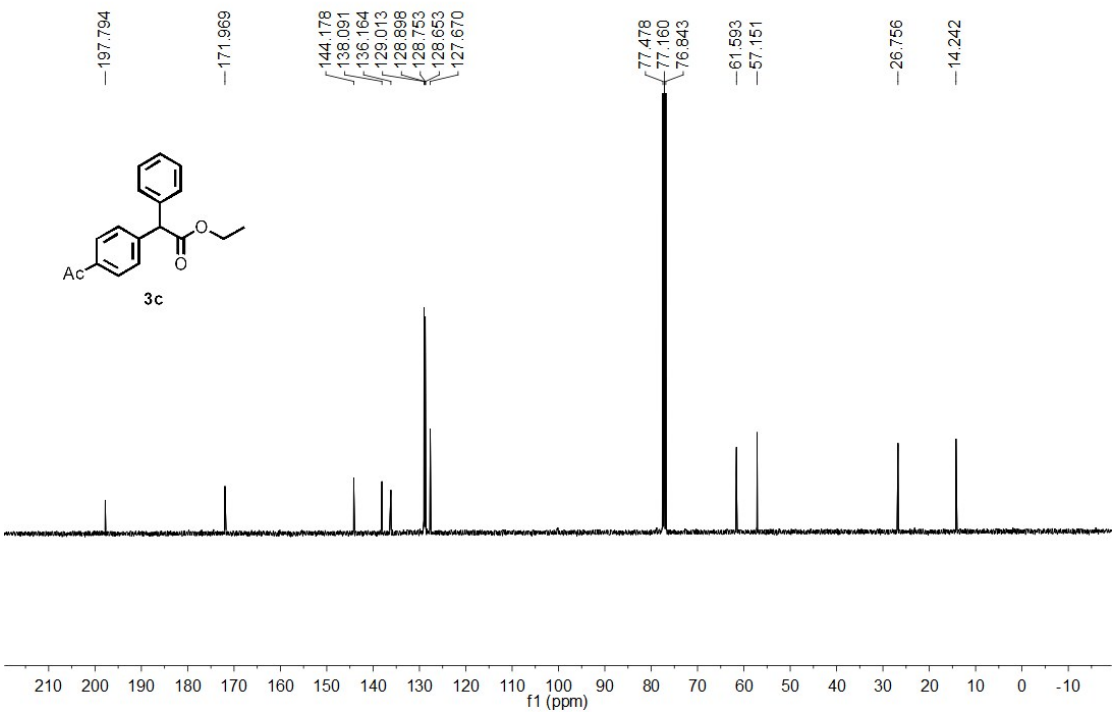
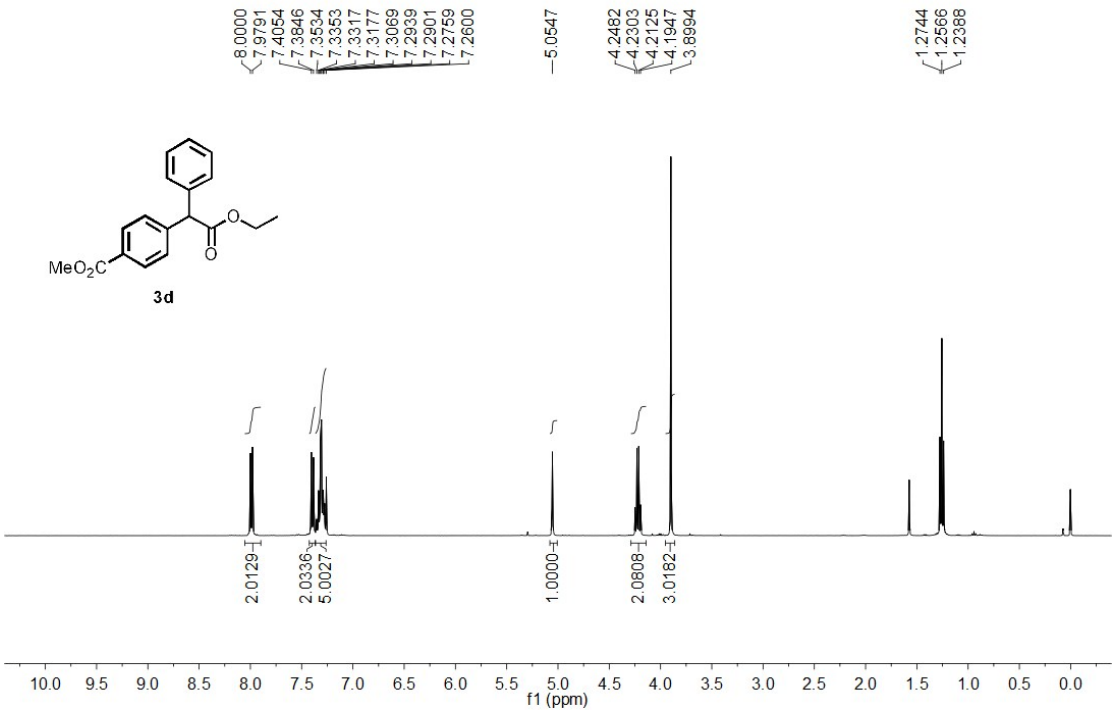
7.9258
7.9213
7.9093
7.9047
7.9000
7.4320
7.4114
7.3443
7.3410
7.3373
7.3236
7.3167
7.3119
7.3013
7.2990
7.2951
7.2810
5.0620
4.2514
4.2336
4.2158
4.1980
2.5796
1.2790
1.2612
1.2434
Ac
O
O
3c
2.0000
2.0616
5.0999
1.0889
2.0019
3.0048
3.0328
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

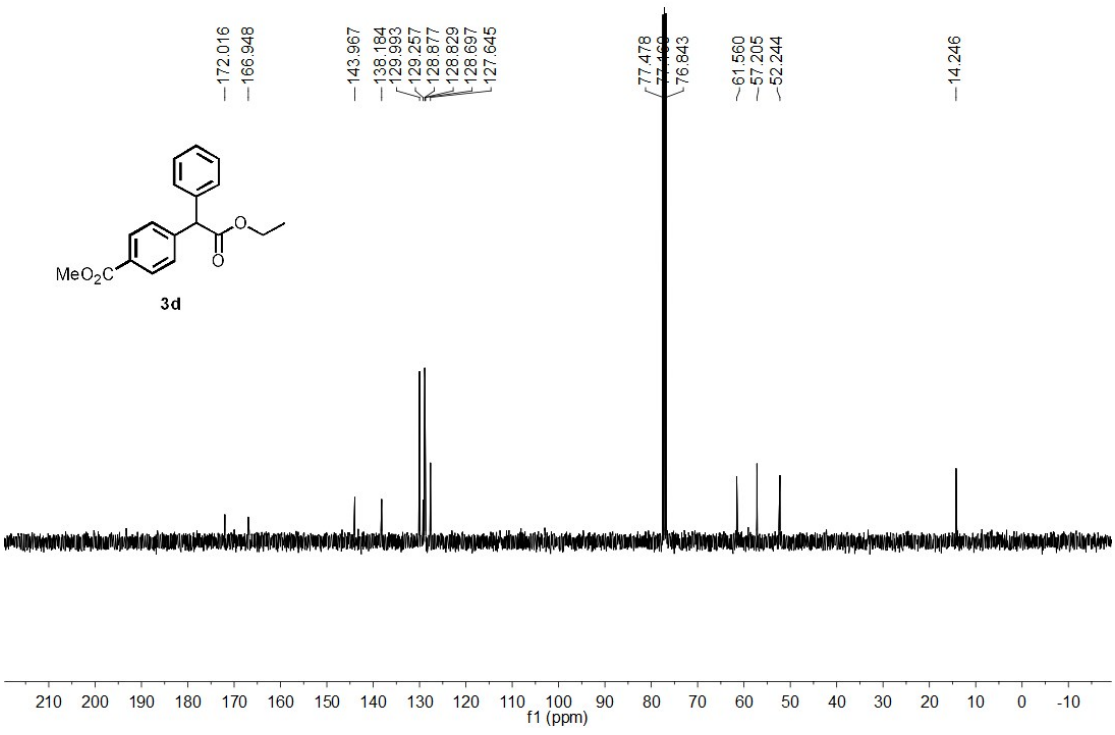
ZZP-62


197.794
171.969
144.178
138.091
136.164
129.013
128.898
128.753
128.653
127.670
77.478
77.160
76.843
61.593
57.151
26.756
14.242
Ac
O
O
3c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

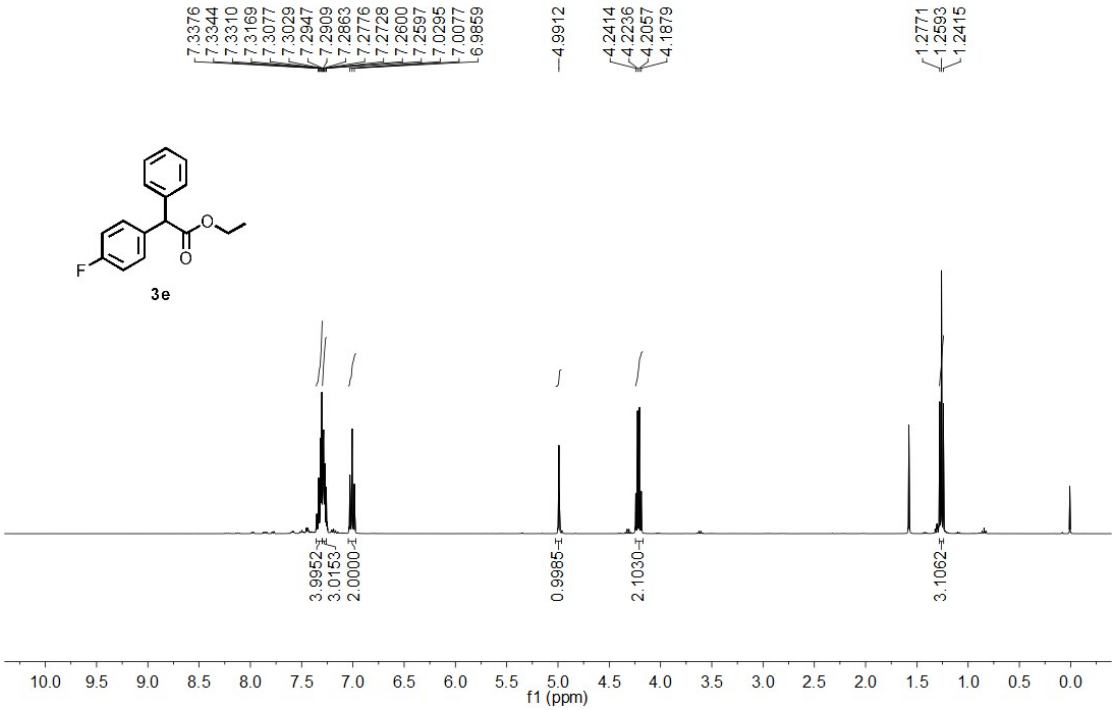
ZZP-271-1



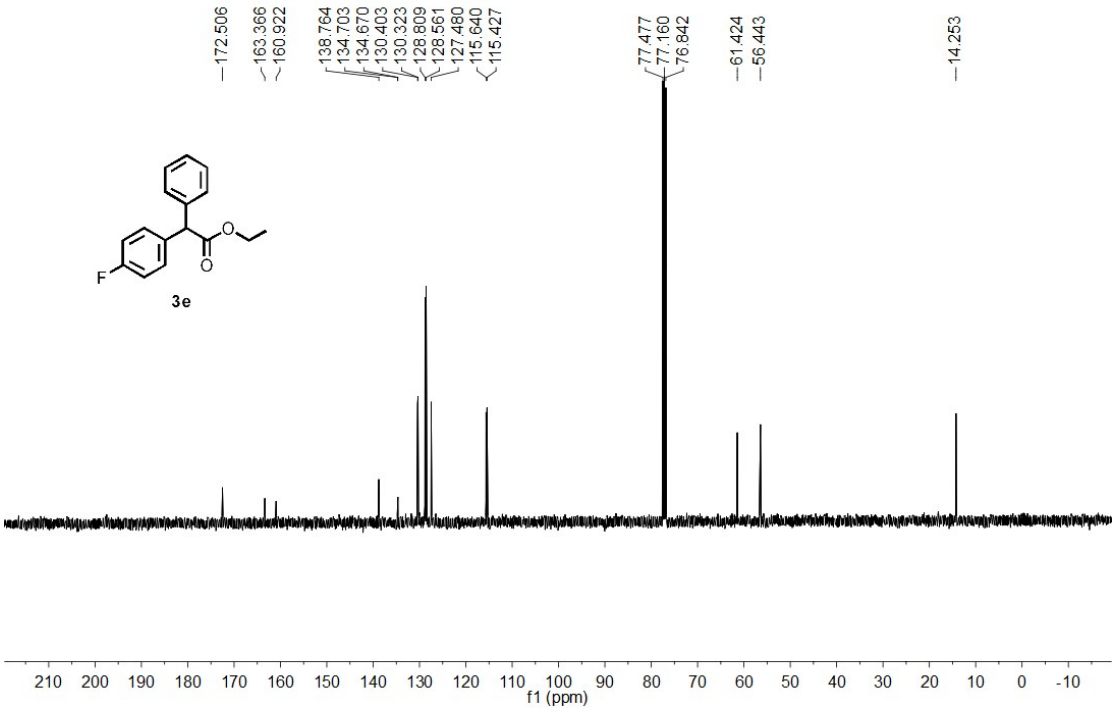
ZZP-271-1



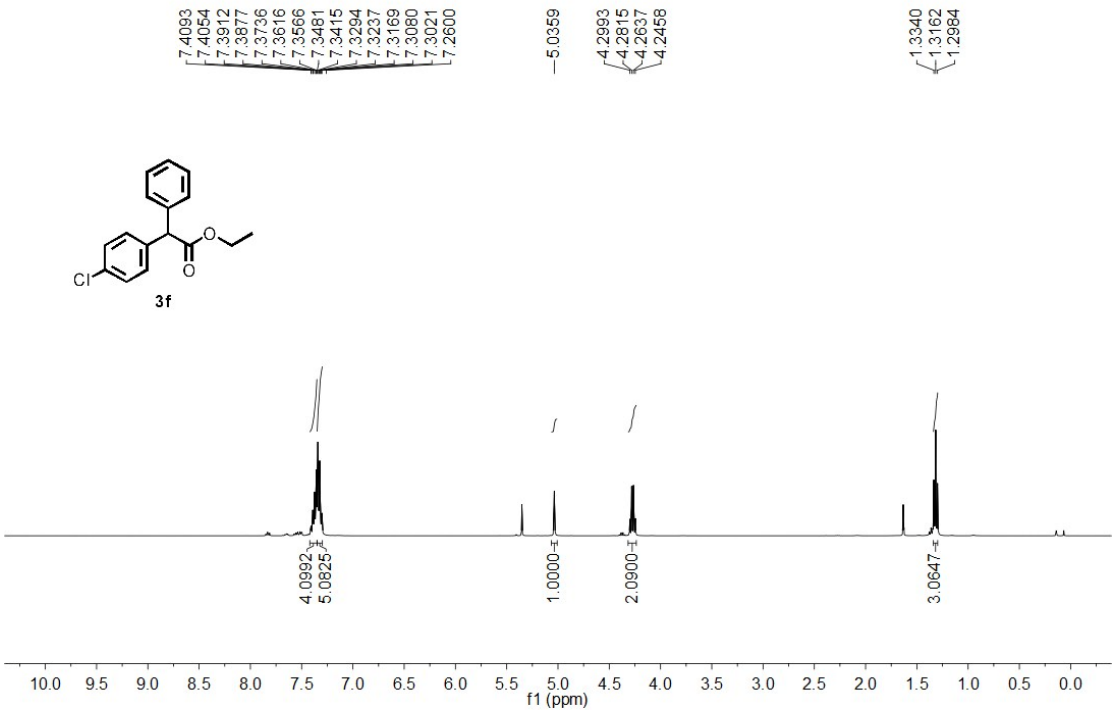
zzp-89-1



ZZP-89-2



ZZP-72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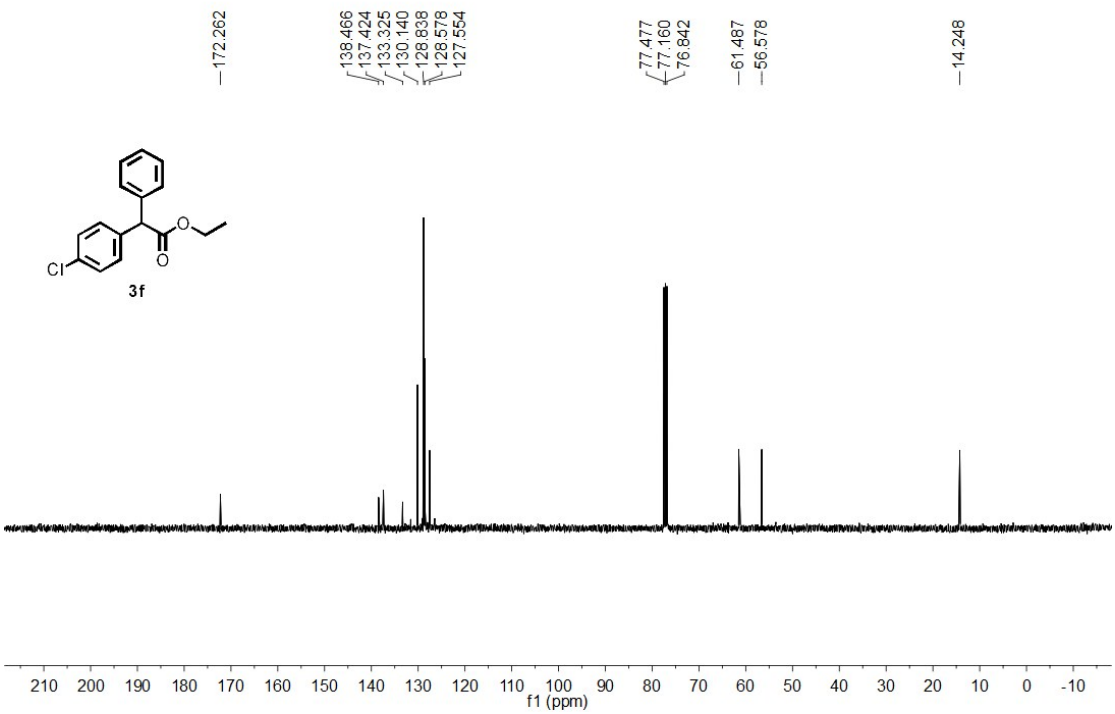
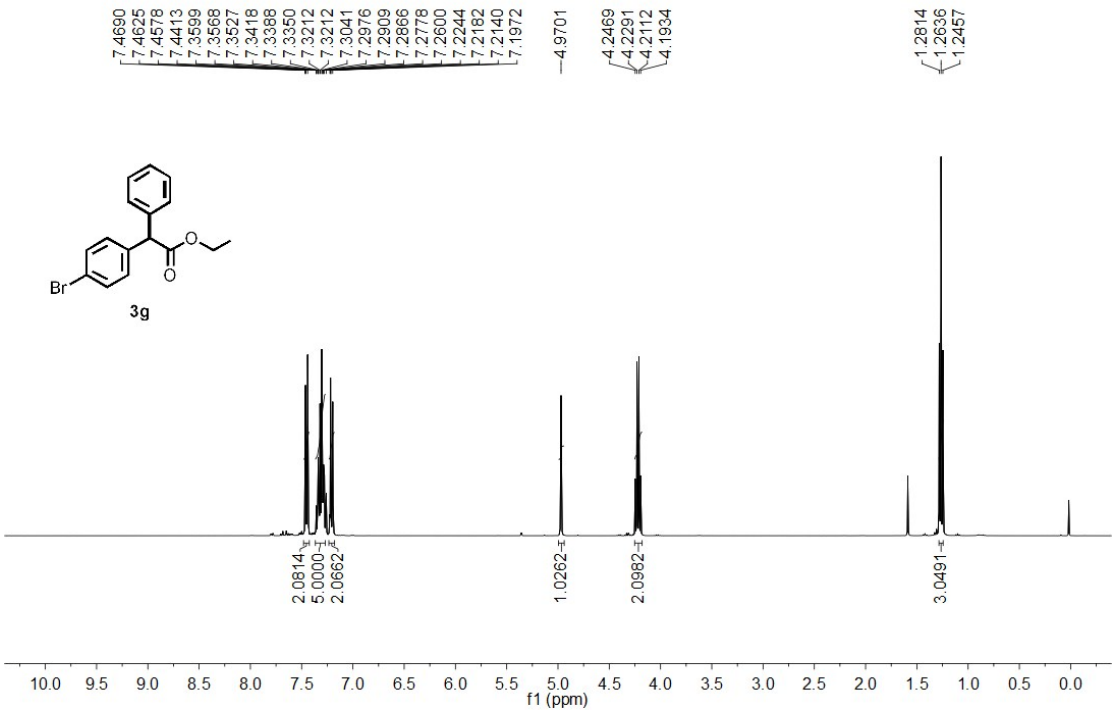
7.4054
7.3912
7.3877
7.3736
7.3616
7.3566
7.3481
7.3415
7.3294
7.3237
7.3169
7.3080
7.3021
7.2600
5.0359
4.2993
4.2815
4.2637
4.2458
1.3340
1.3162
1.2984
Cl
O
O
3f
4.0992
5.0825
1.0000
2.0900
3.0647
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

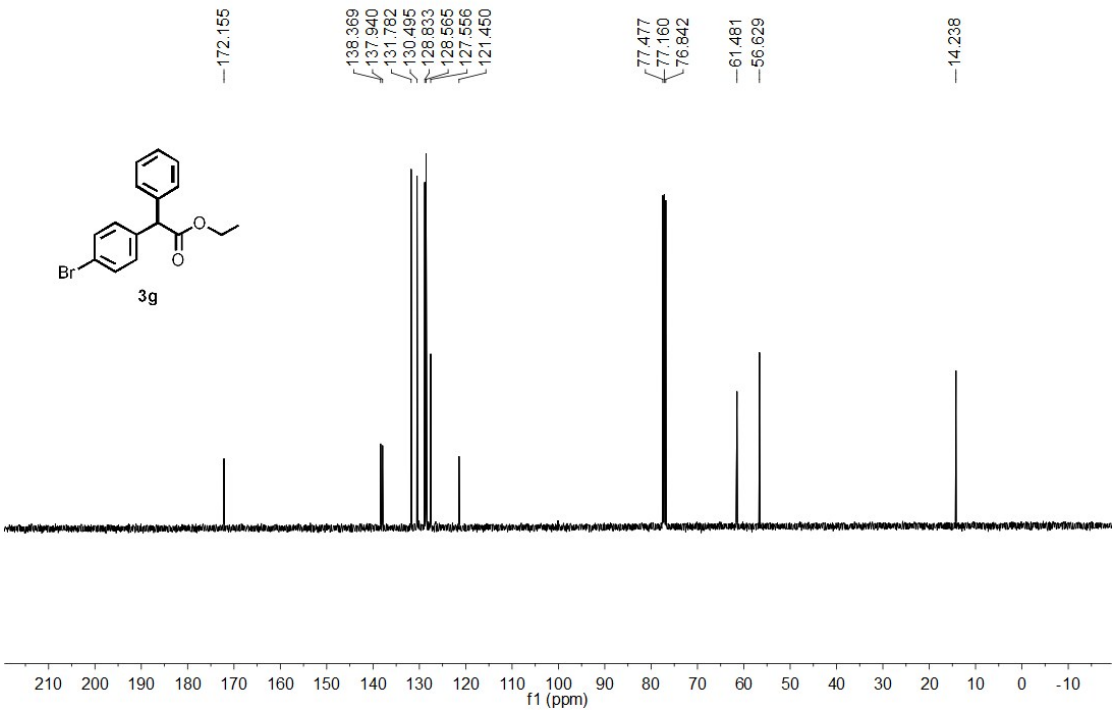
ZZP-72


172.262
138.466
137.424
133.325
130.140
128.838
128.578
127.554
77.477
77.160
76.842
61.487
56.578
14.248
Cl
O
O
3f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

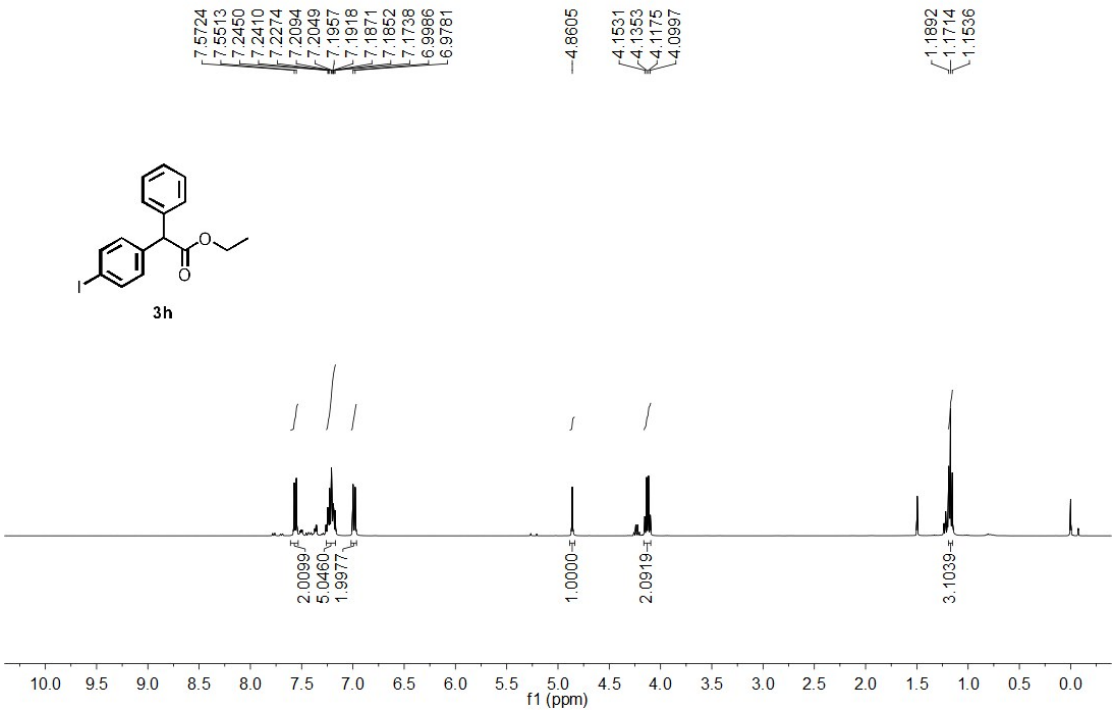
zzp-88-1



ZZP-88-2



ZZP-70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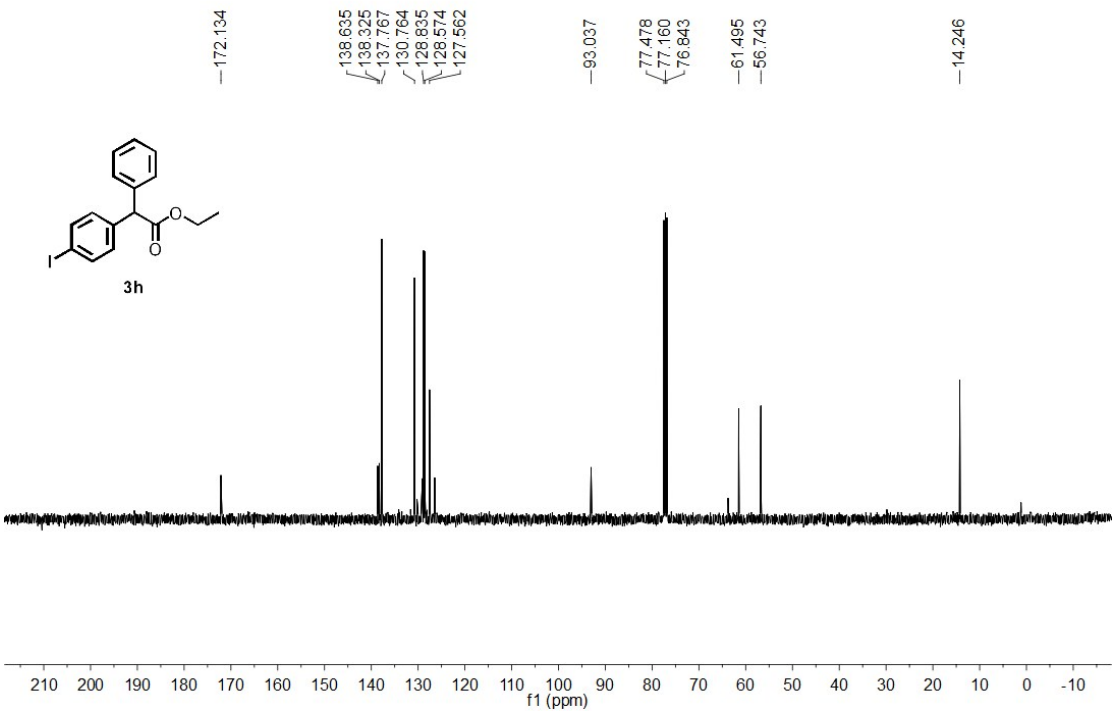
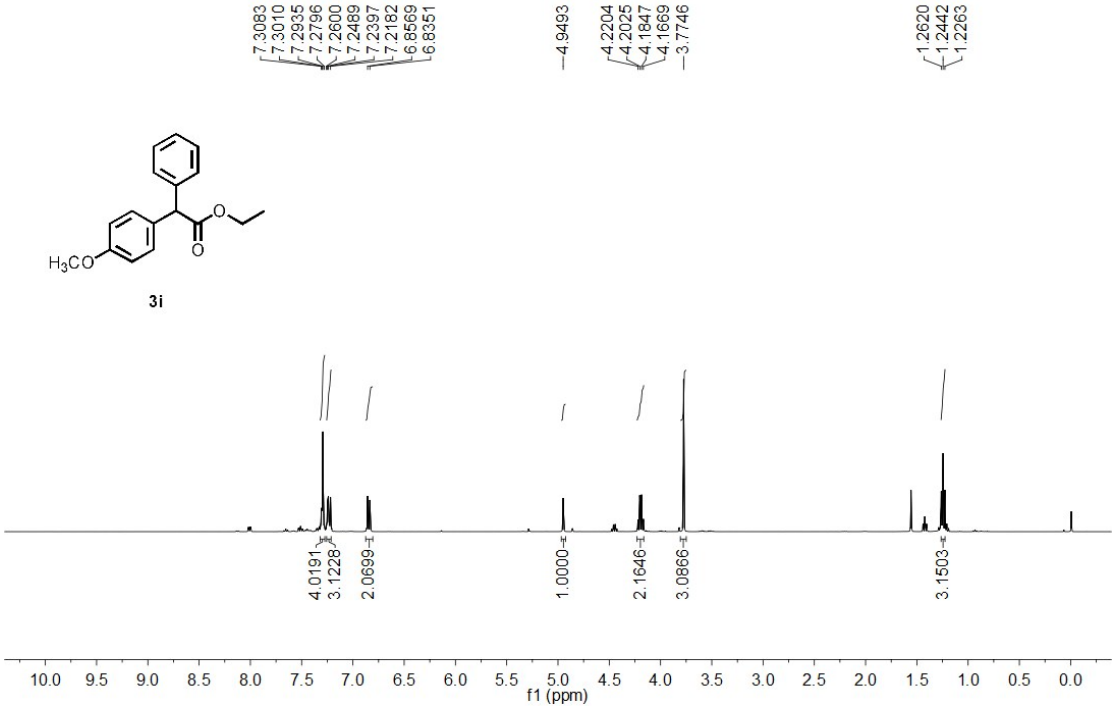
7.5513
7.2450
7.2410
7.2274
7.2094
7.2049
7.1957
7.1918
7.1871
7.1852
7.1738
6.9986
6.9781
4.8605
4.1531
4.1353
4.1175
4.0997
1.1892
1.1714
1.1536
3h
2.0099
5.0460
1.9977
1.0000
2.0919
3.1039
f1 (ppm)

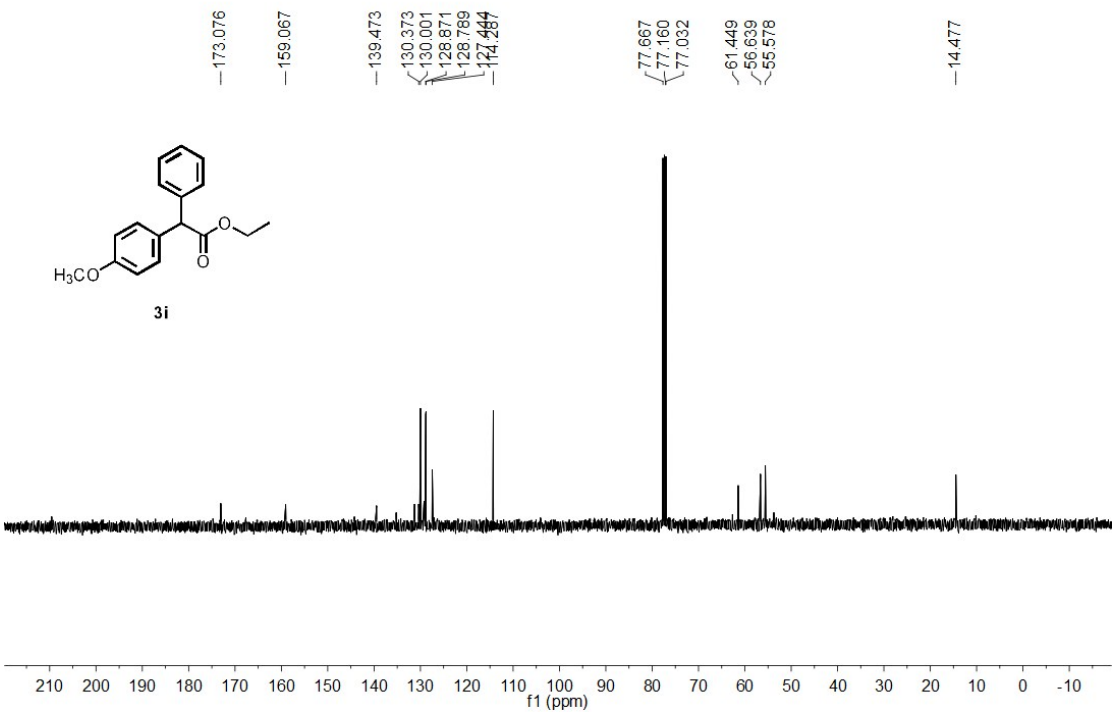
ZZP-70


172.134
138.635
138.325
137.767
130.764
128.835
128.574
127.562
93.037
77.478
77.160
76.843
61.495
56.743
14.246
3h
f1 (ppm)

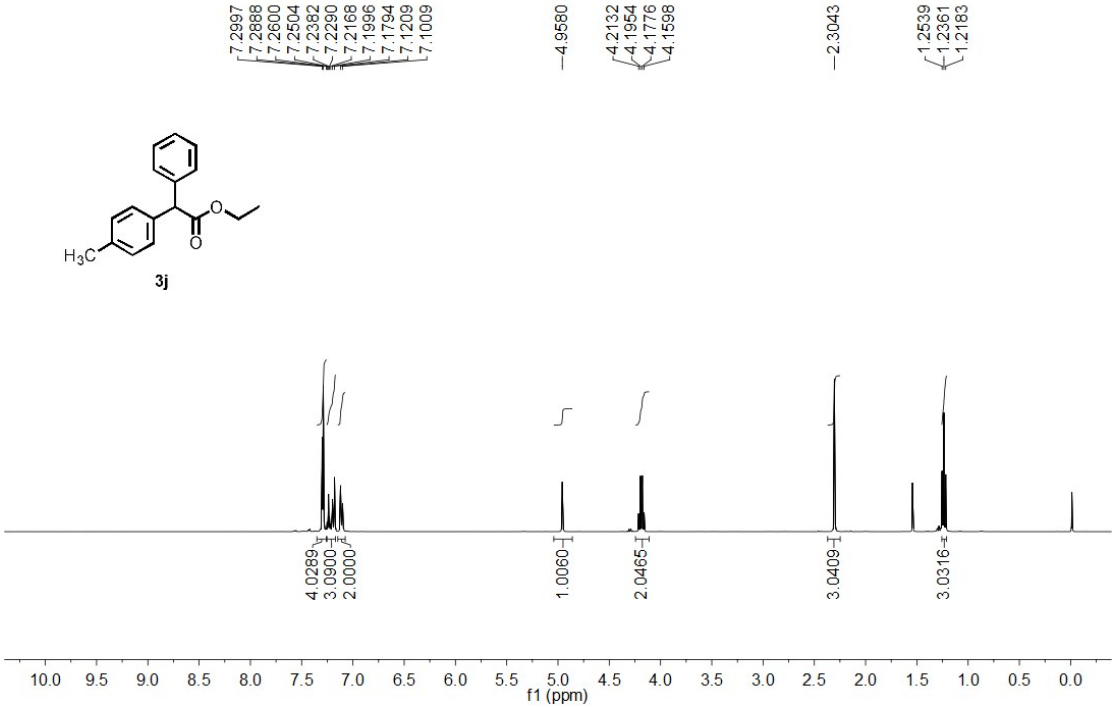
ZZP-292-1



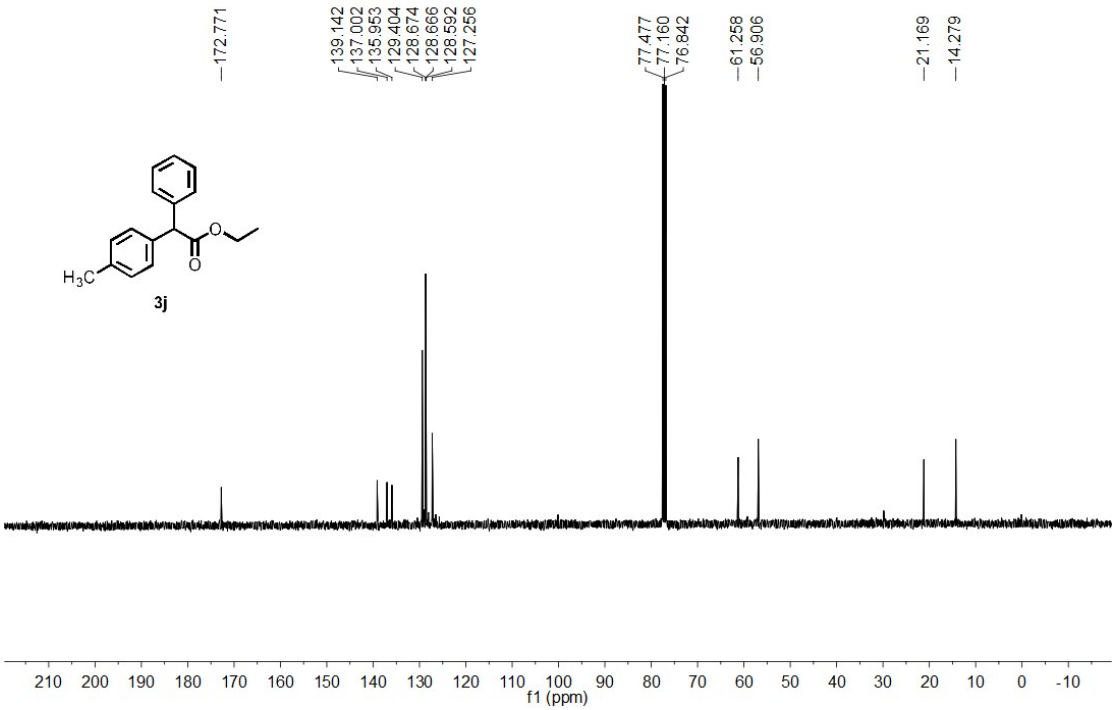
ZZP-292-3



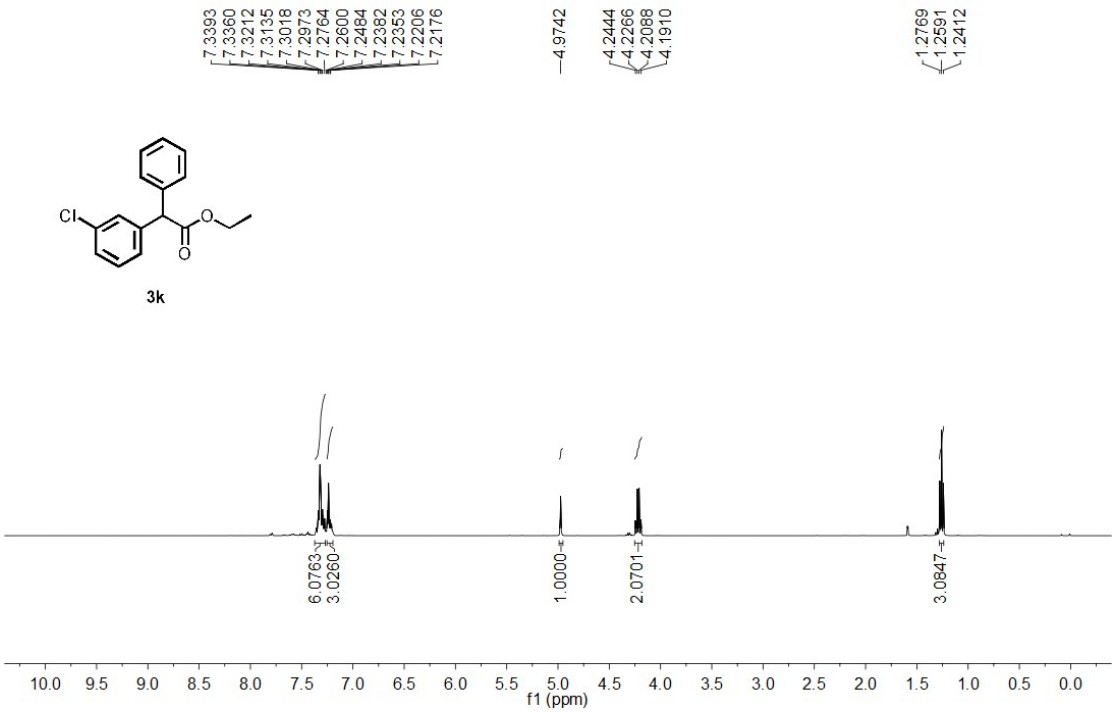
ZZP-90-1



ZZP-90-3



ZZP-76-1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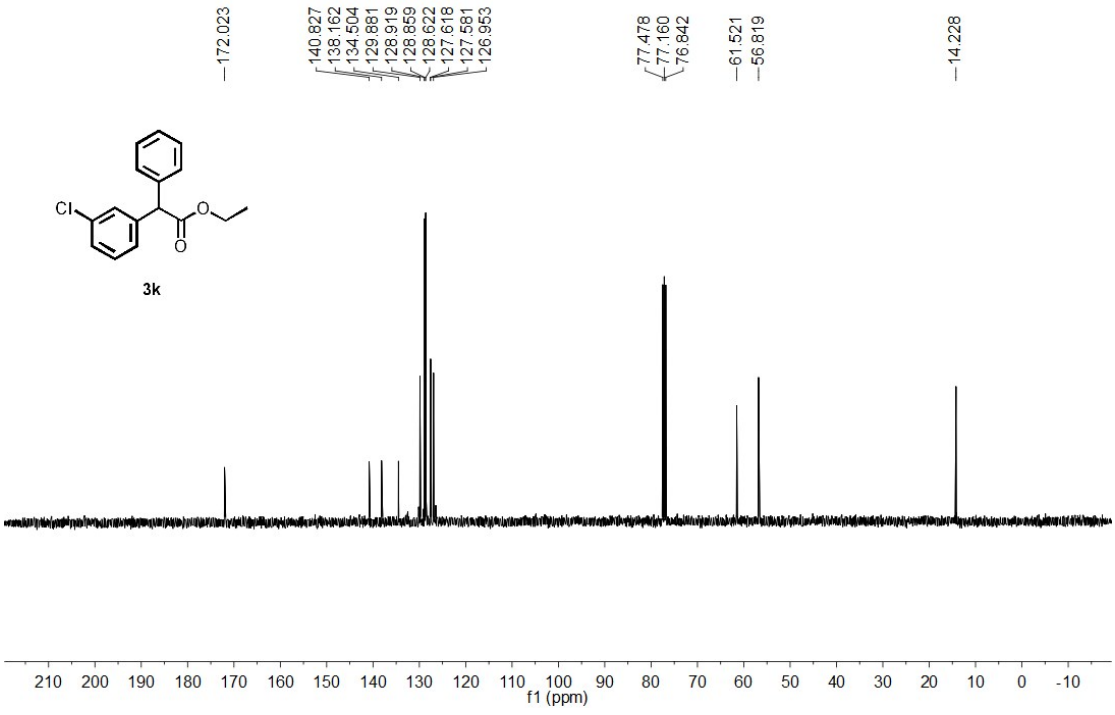
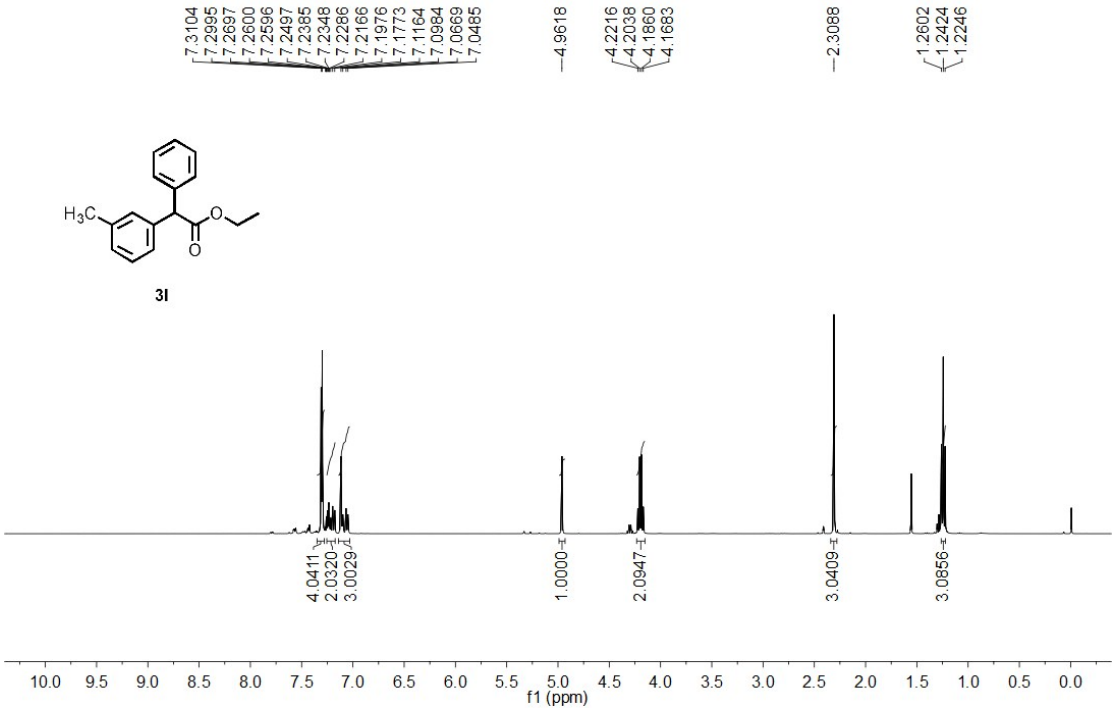
7.3212
7.3135
7.3018
7.2973
7.2764
7.2600
7.2484
7.2382
7.2353
7.2206
7.2176
4.9742
4.2444
4.2266
4.2088
4.1910
1.2769
1.2591
1.2412
Cl
O
O
3k
6.0763
3.0260
1.0000
2.0701
3.0847
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

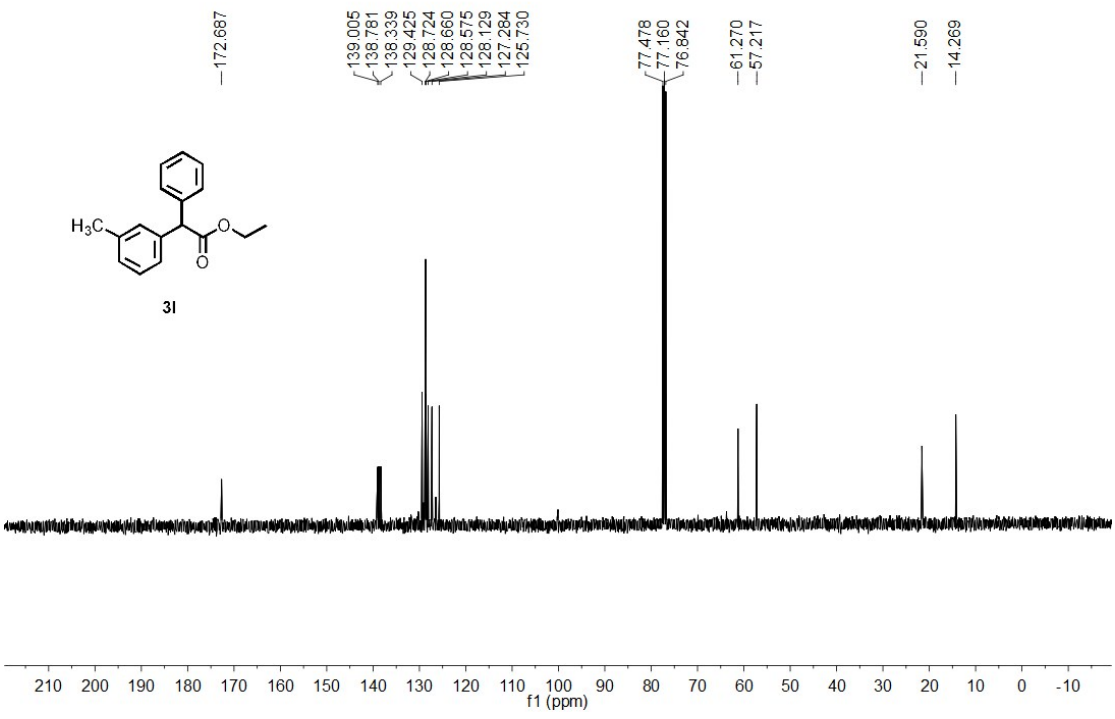
ZP-76


172.023
140.827
138.162
134.504
129.881
128.919
128.859
128.622
127.618
127.581
126.953
77.478
77.160
76.842
61.521
56.819
14.228
Cl
O
O
3k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

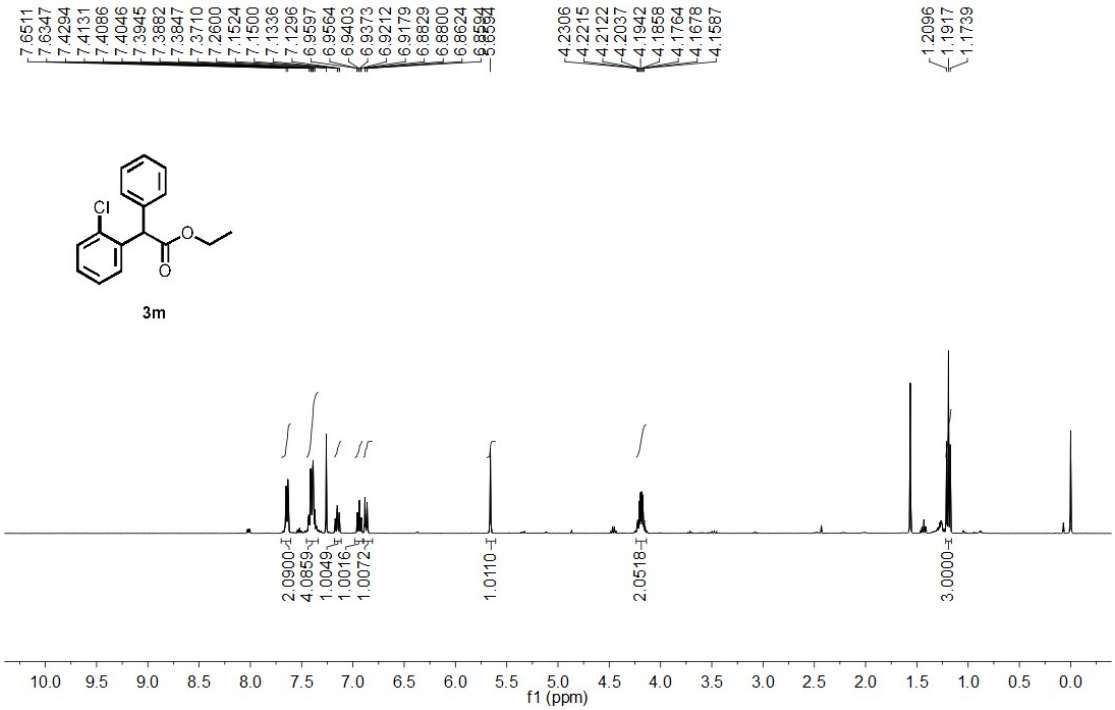
ZZP-94-1



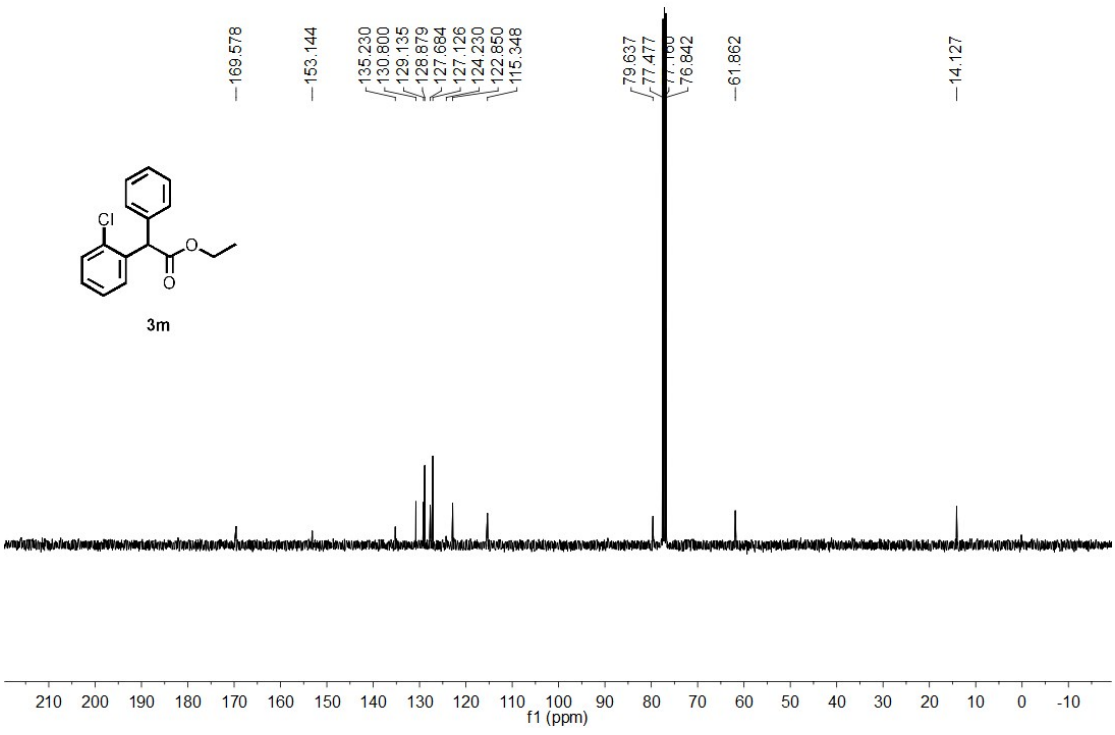
ZZP-94-2



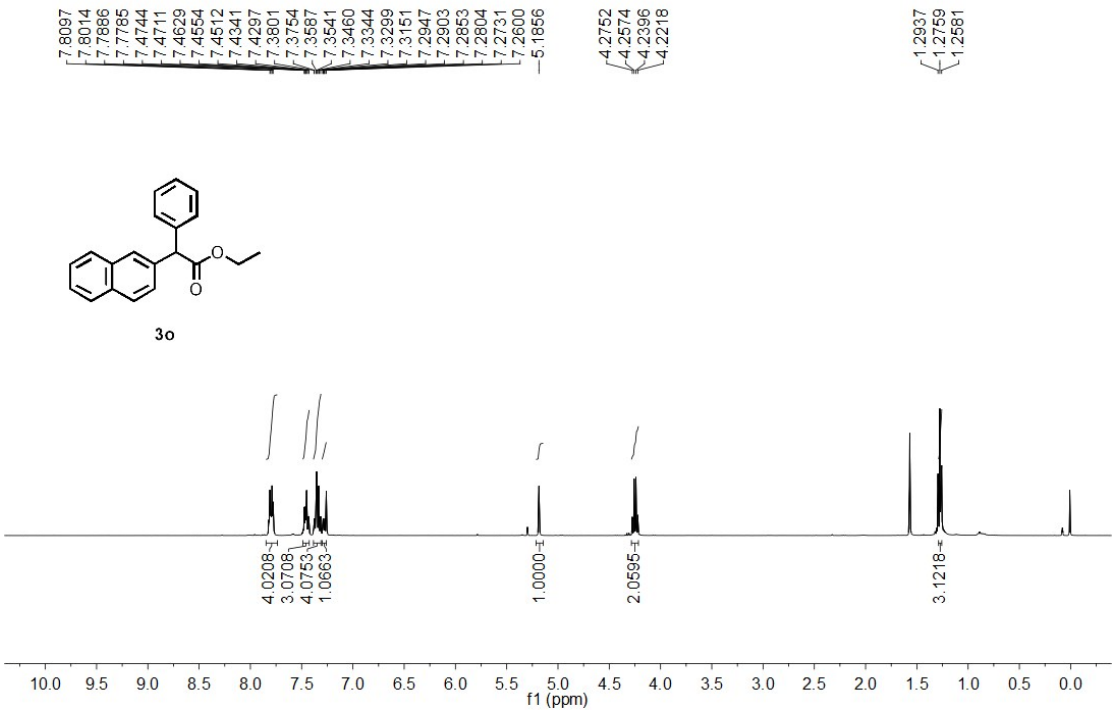
ZZP-354-2



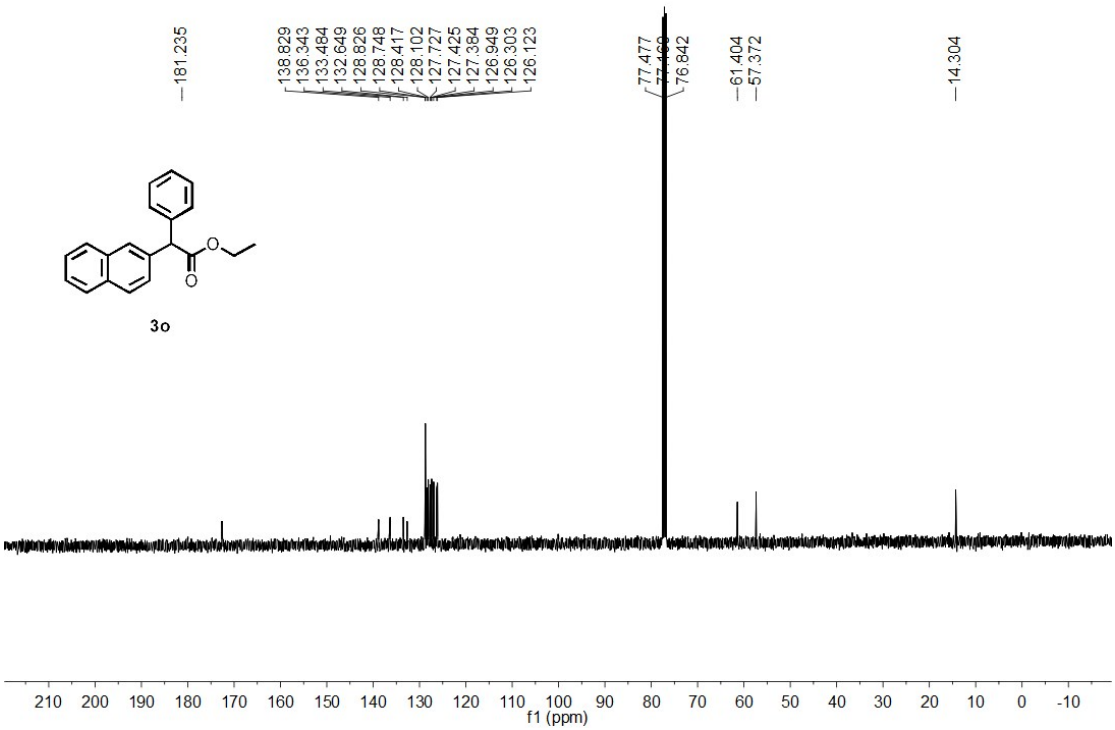
ZZP-354-7



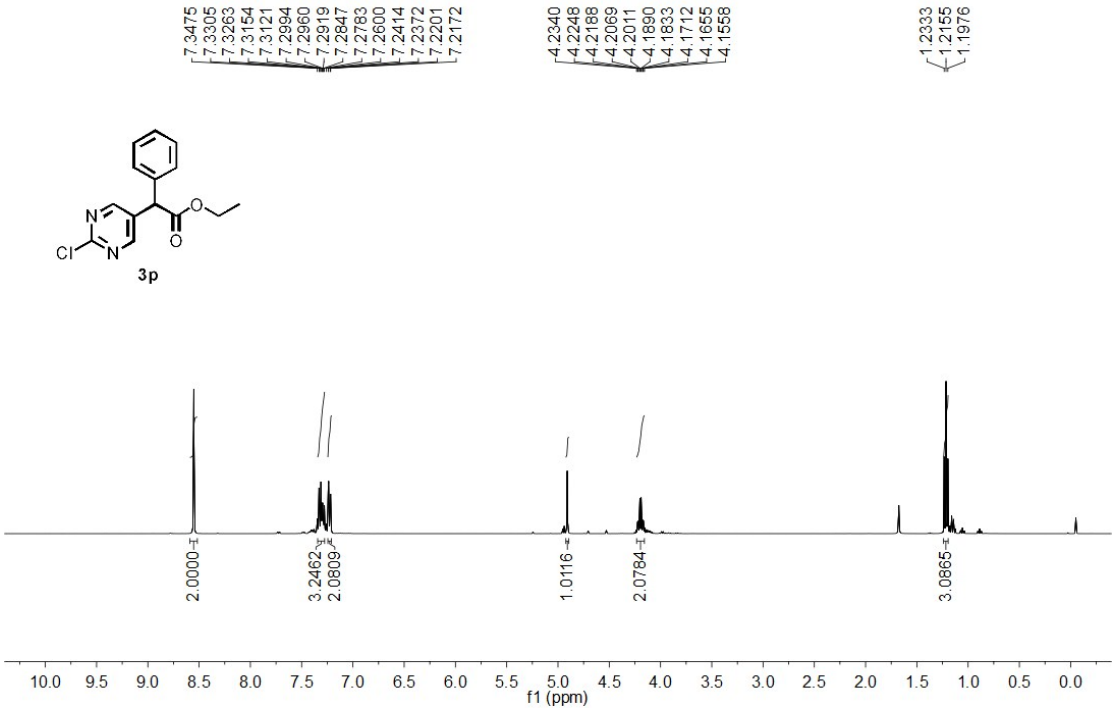
ZZP-96-1



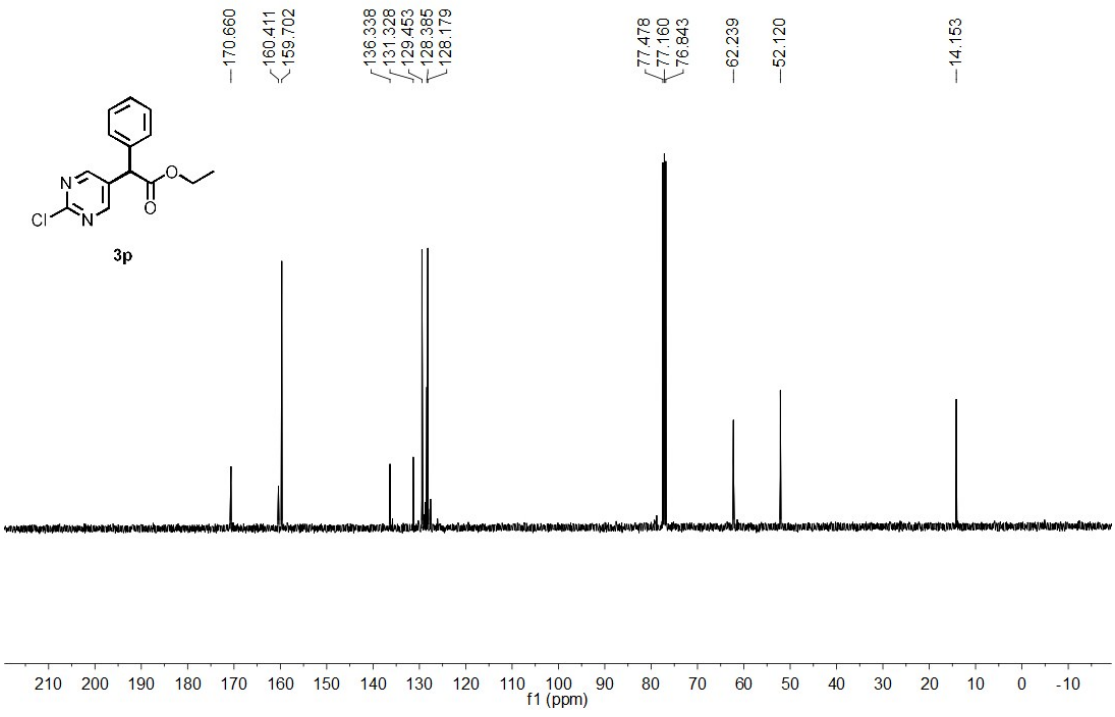
ZZP-96-2



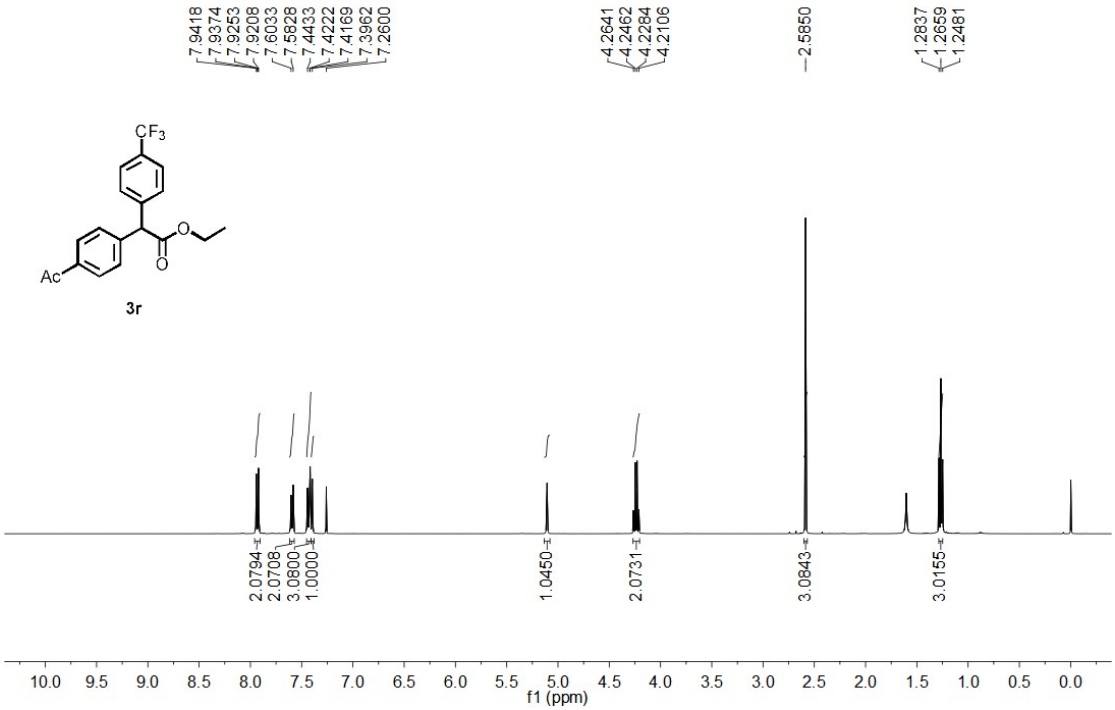
ZZP-97-1



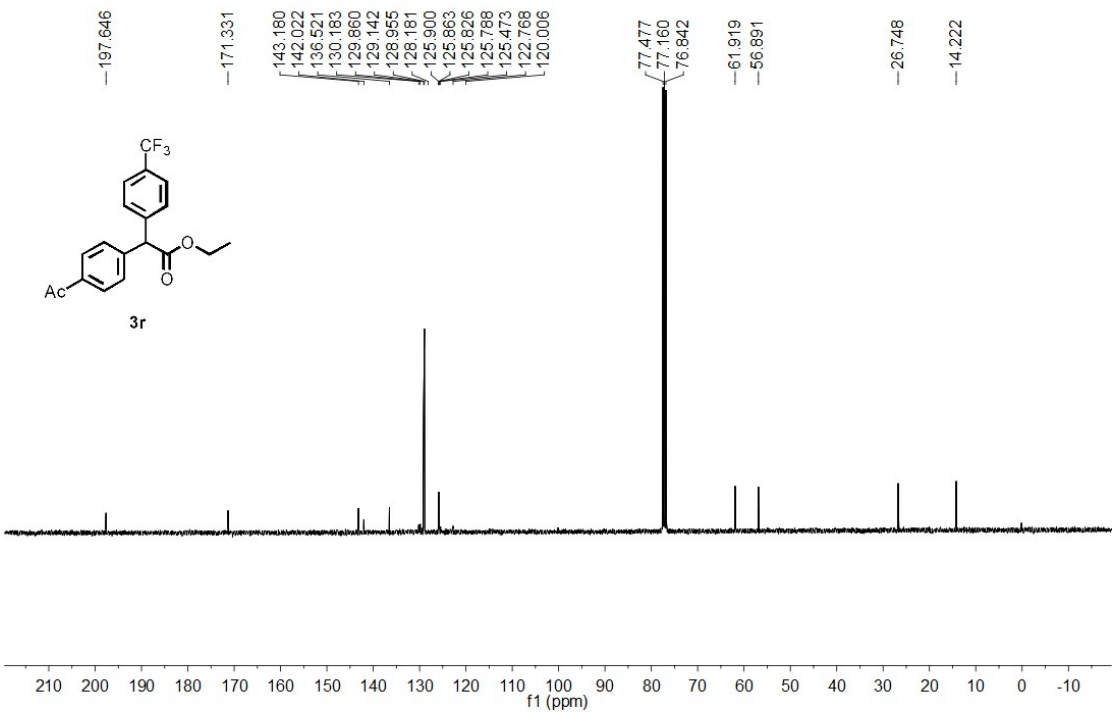
ZZP-97-2



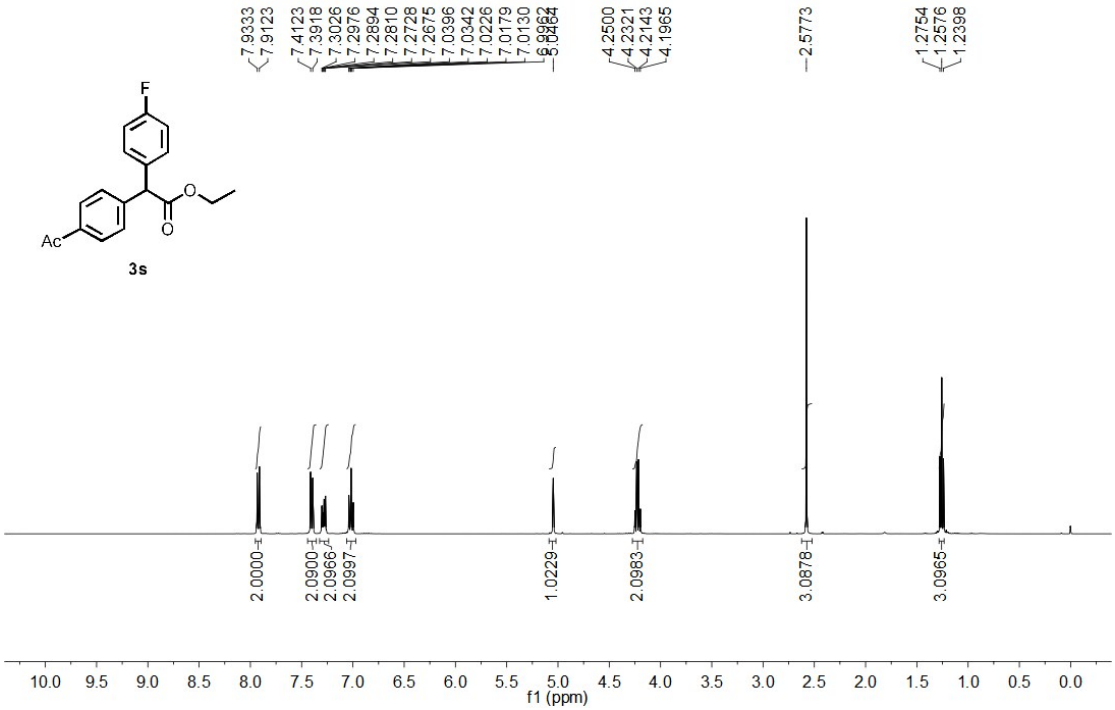
ZZP-115-2



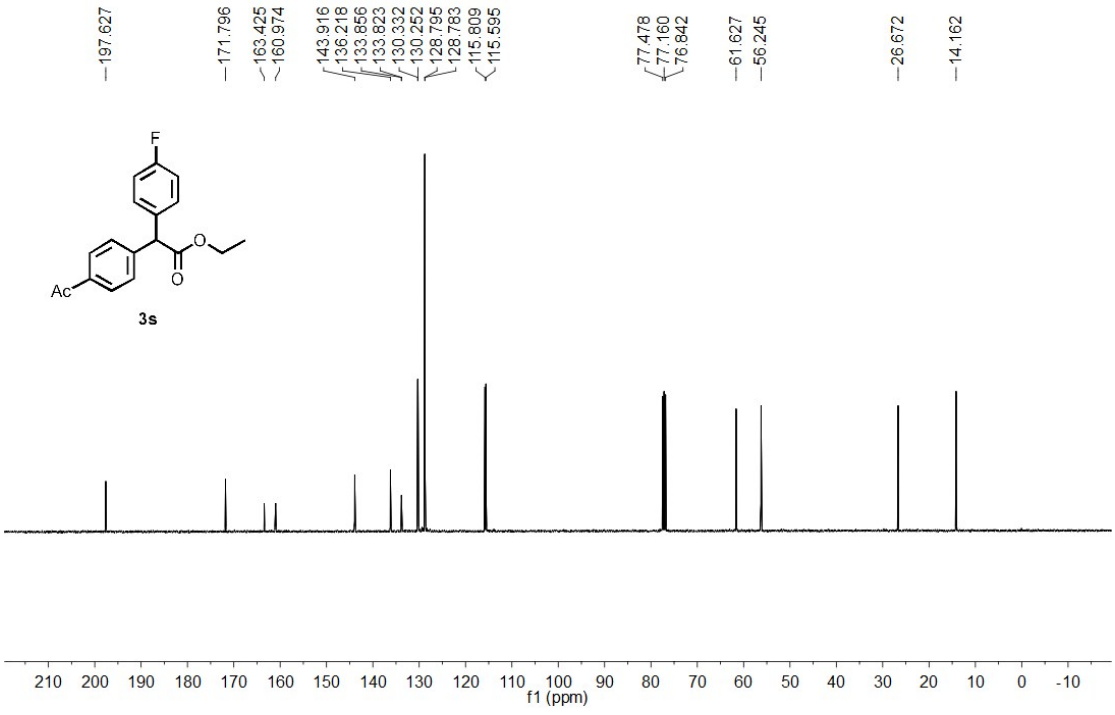
ZZP-115-2



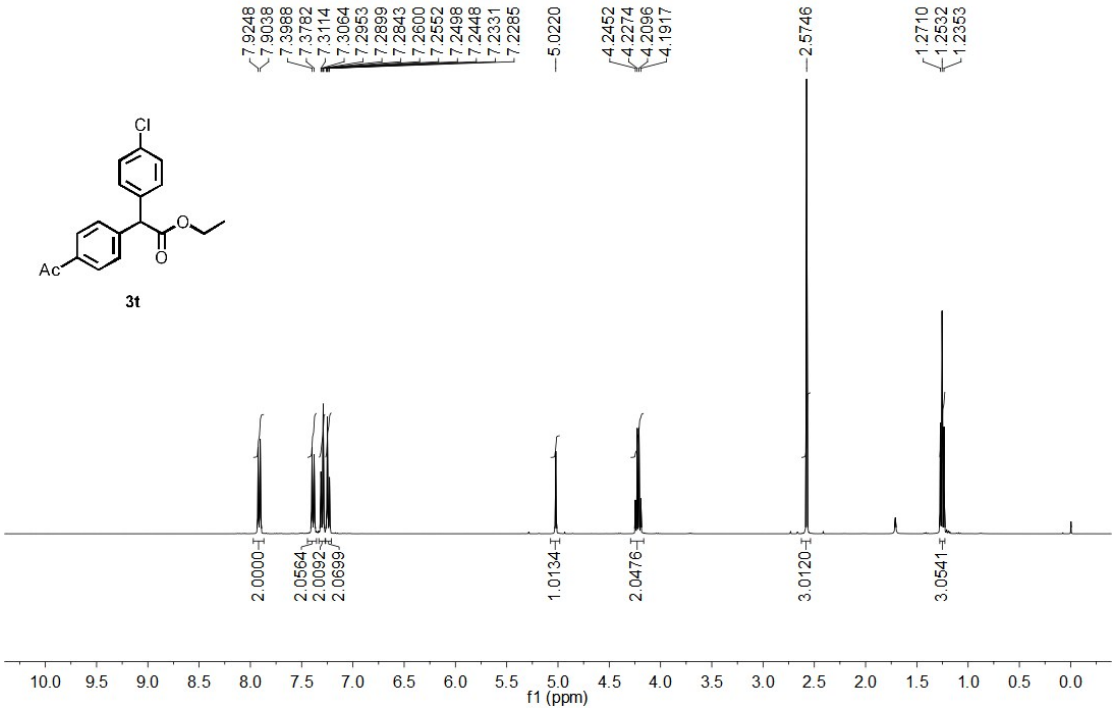
ZZP-218-1



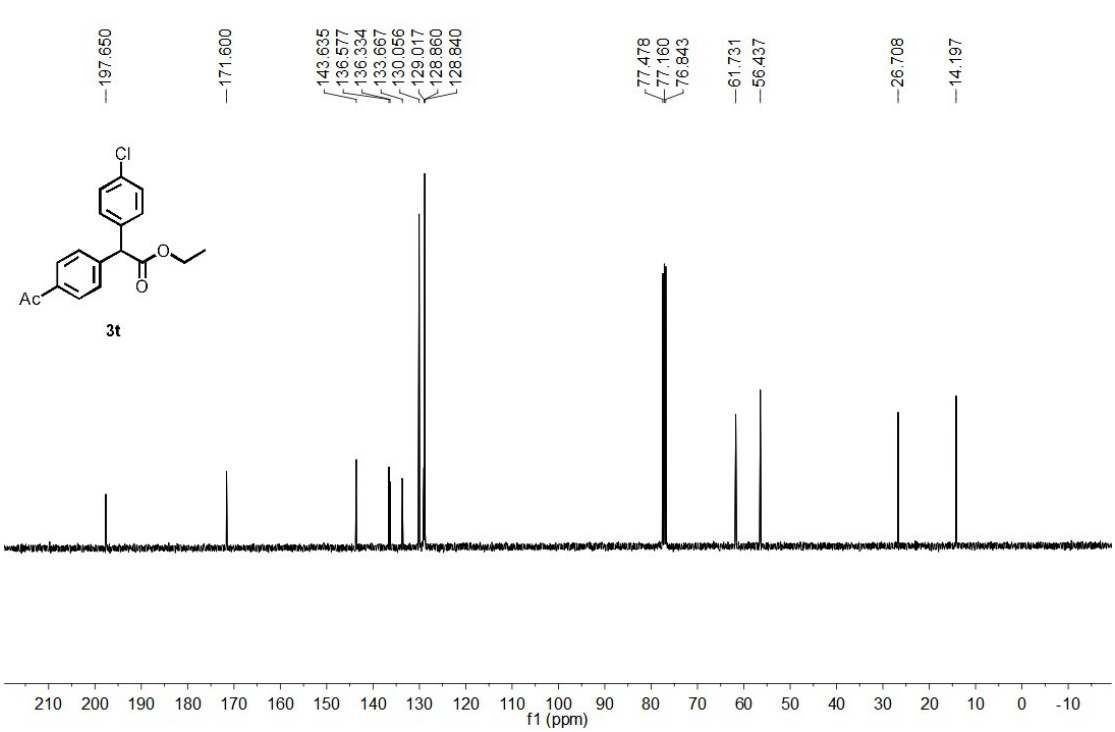
ZZP-218-1



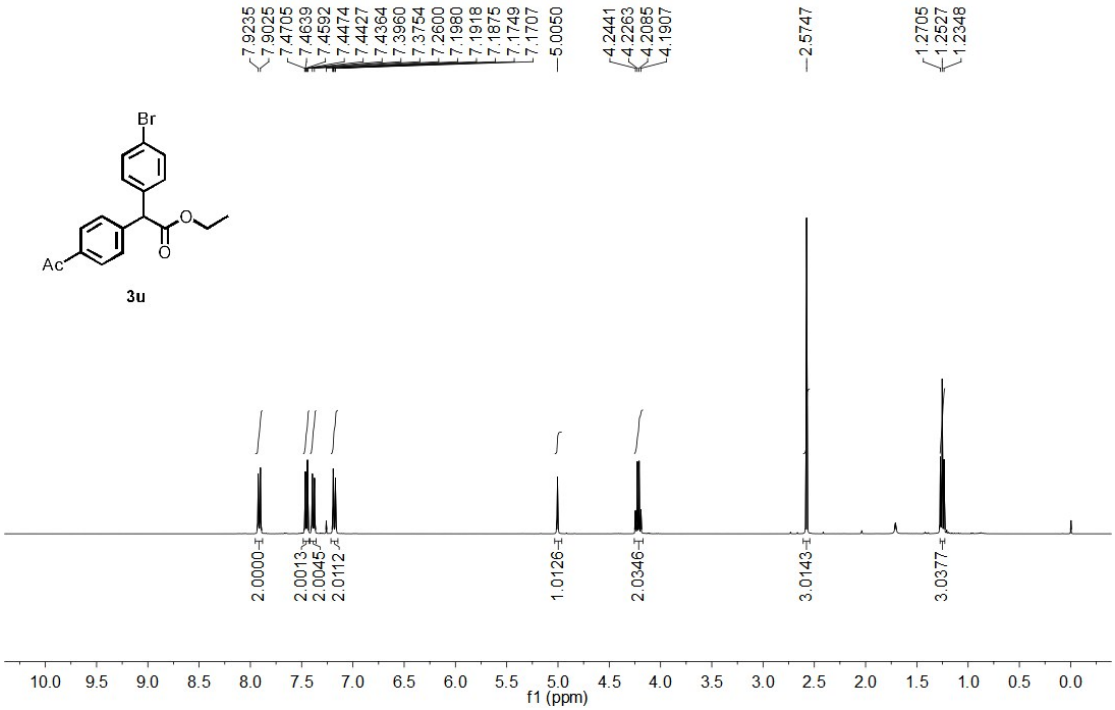
ZZP-110-2



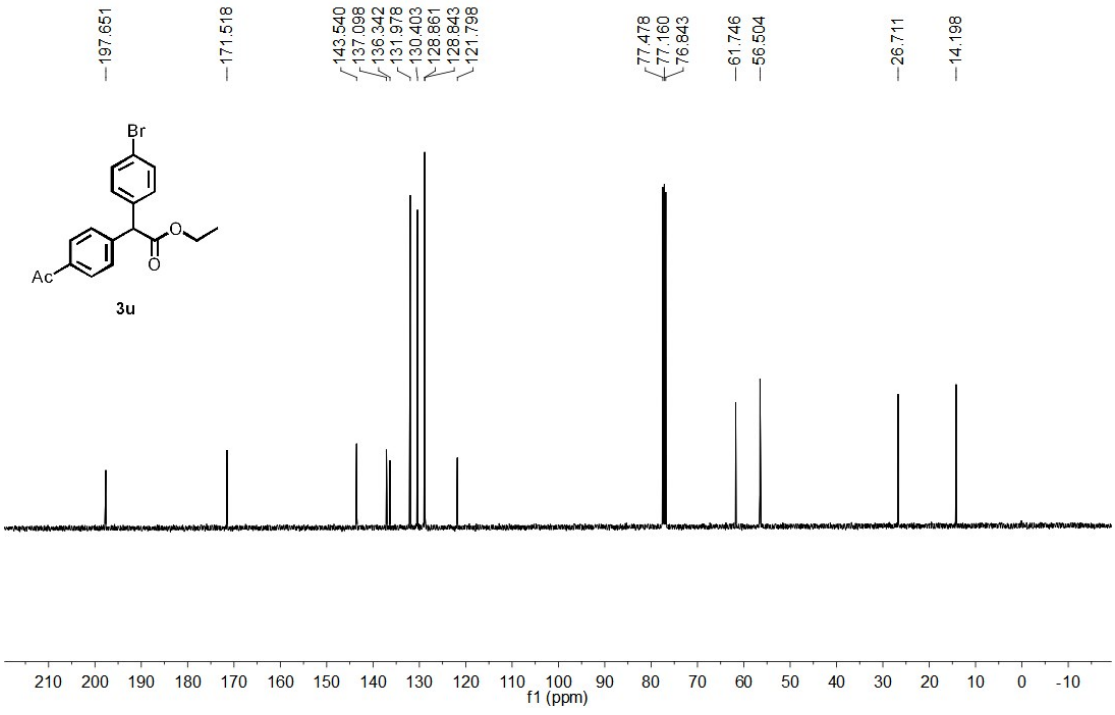
ZZP-110-2



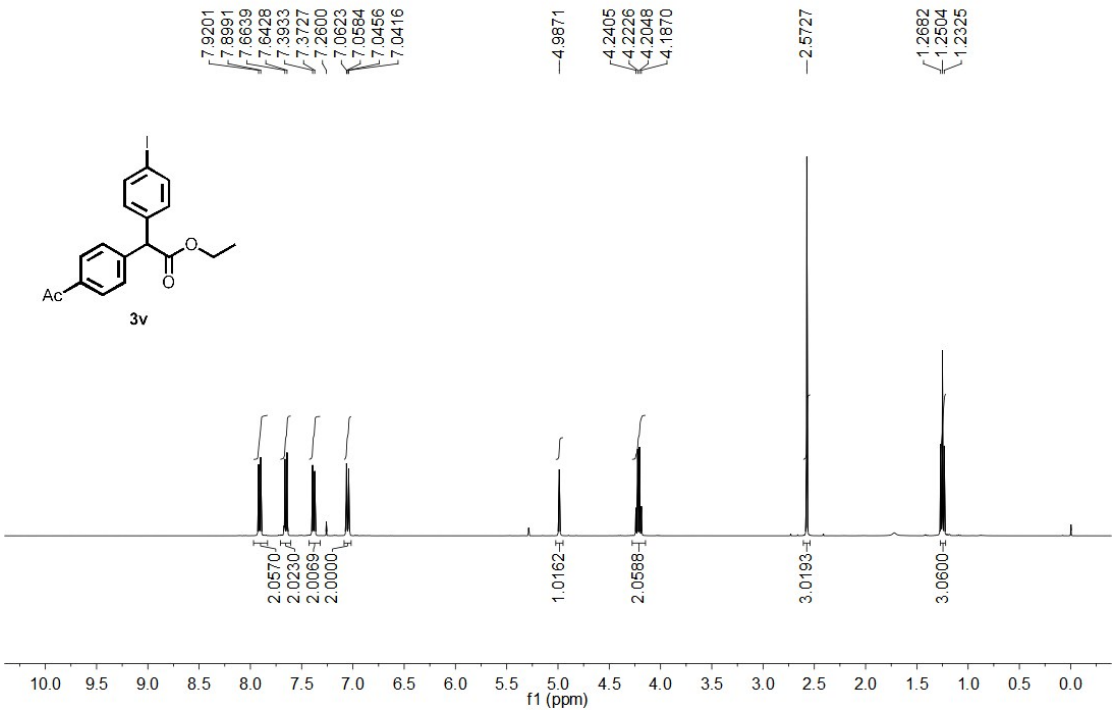
ZZP-113-2



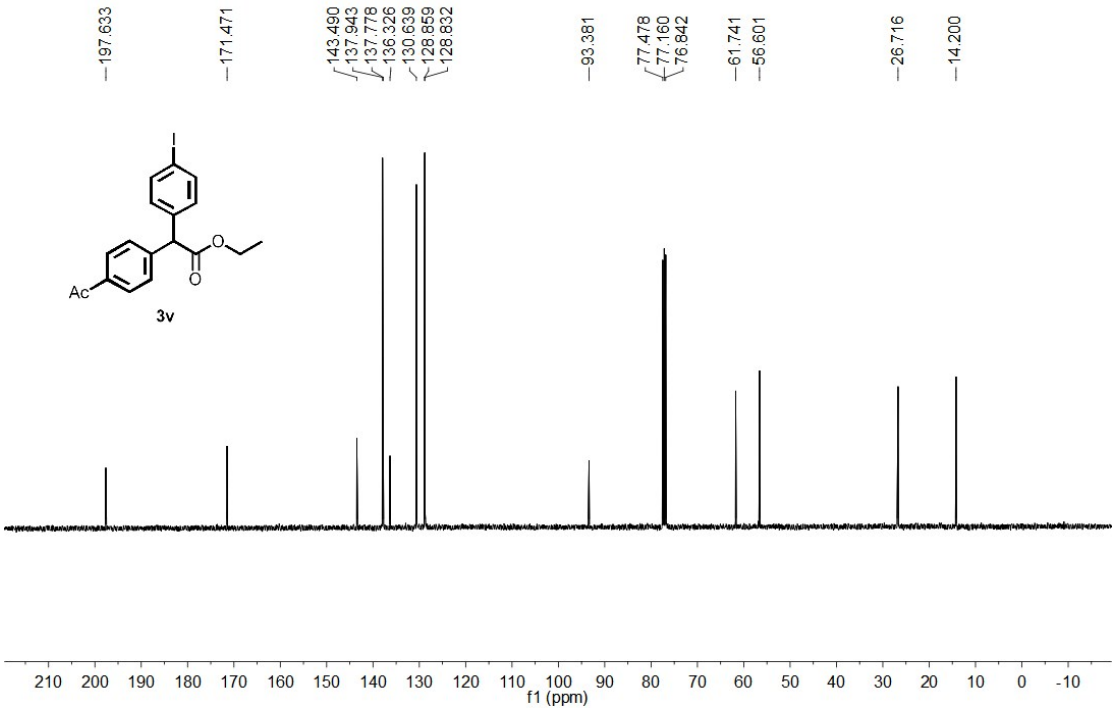
ZZP-113-2



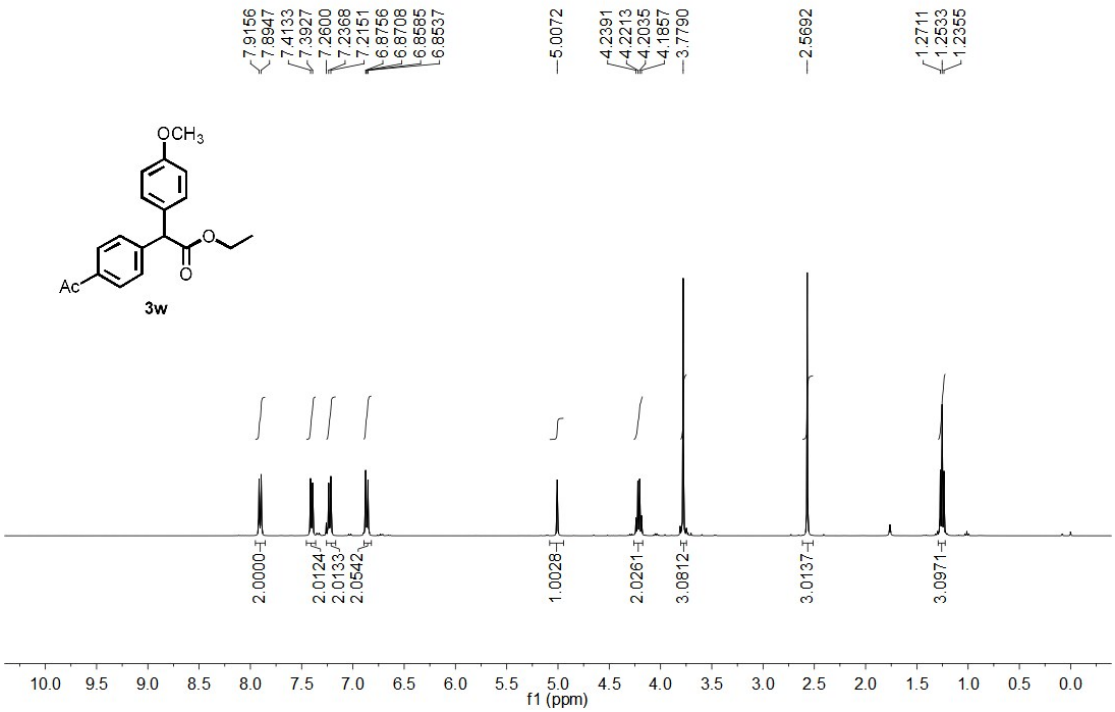
ZZP-143-2



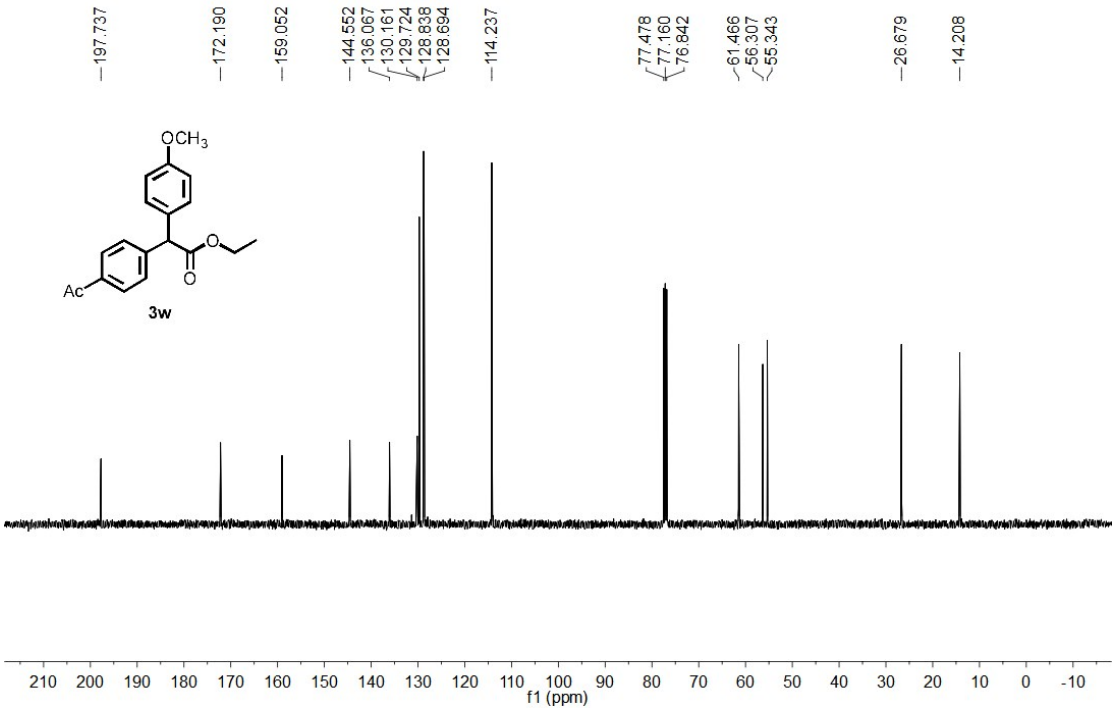
ZZP-143-2



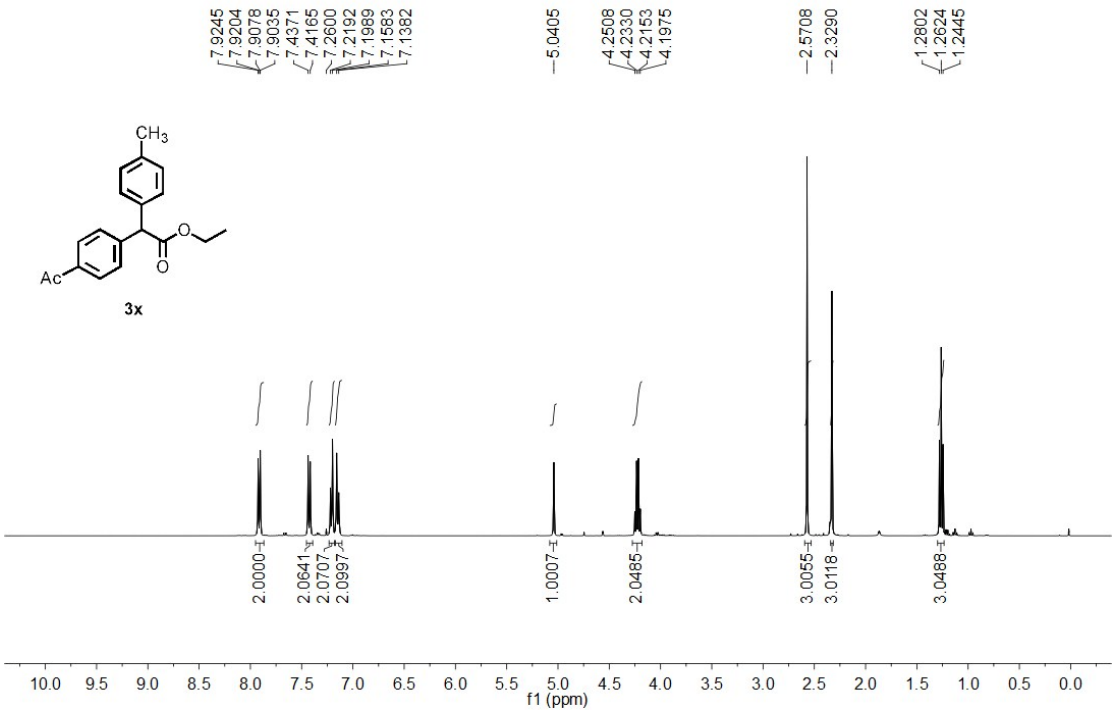
ZZP-73-1



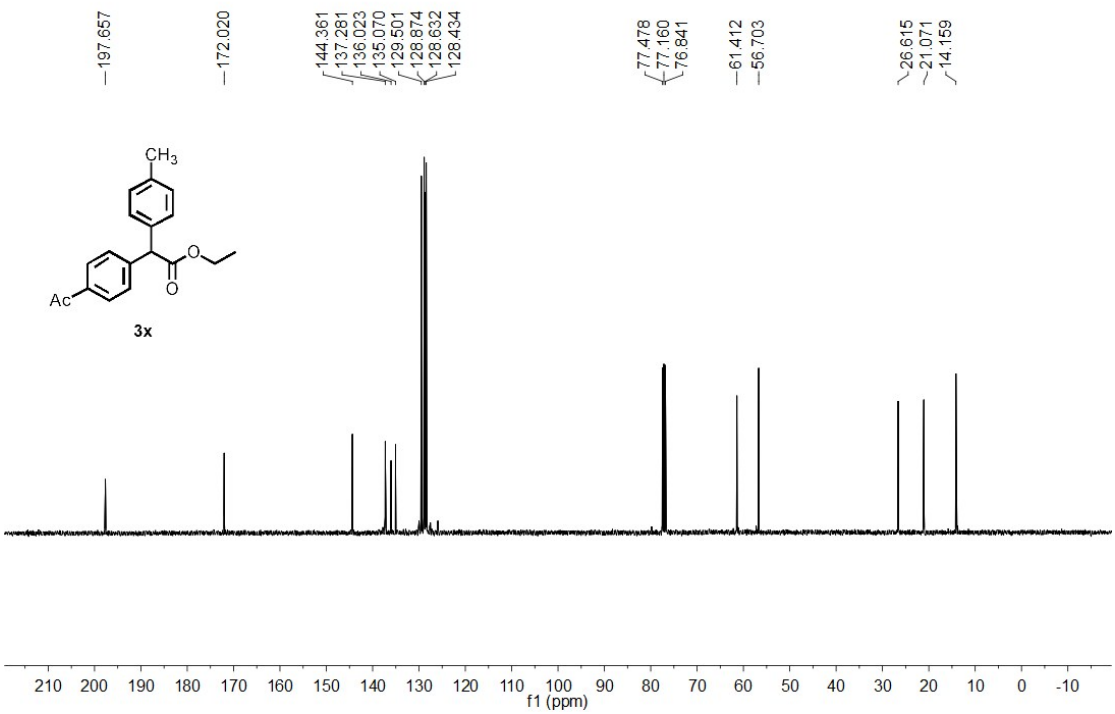
ZZP-73



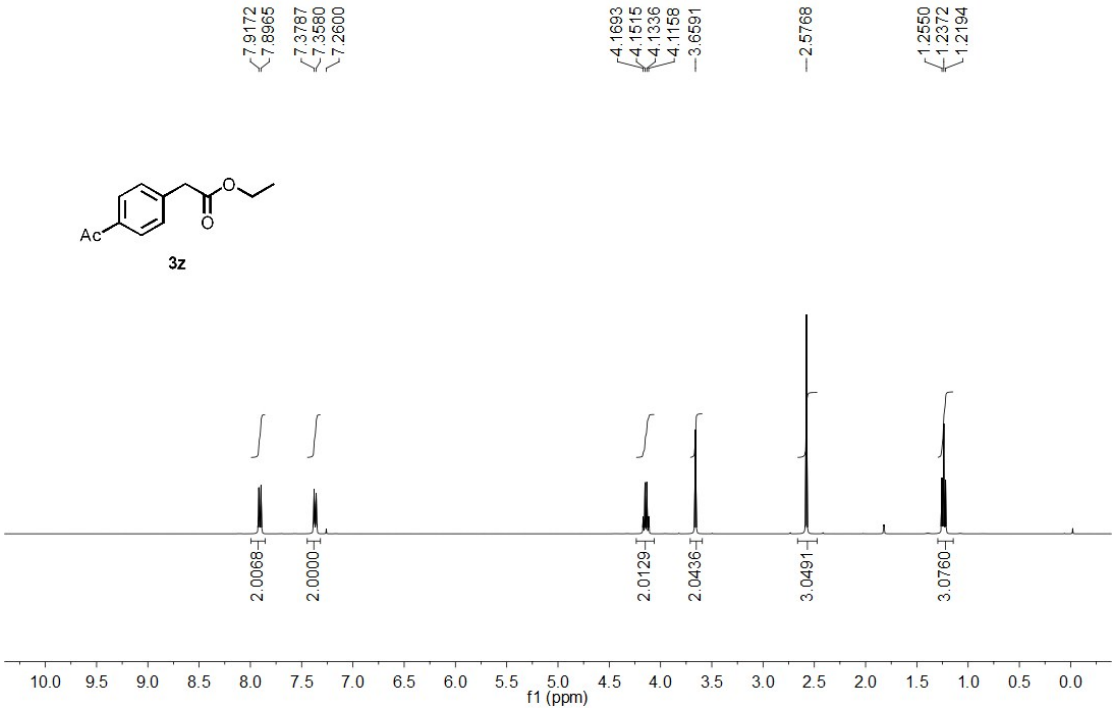
ZZP-92-1



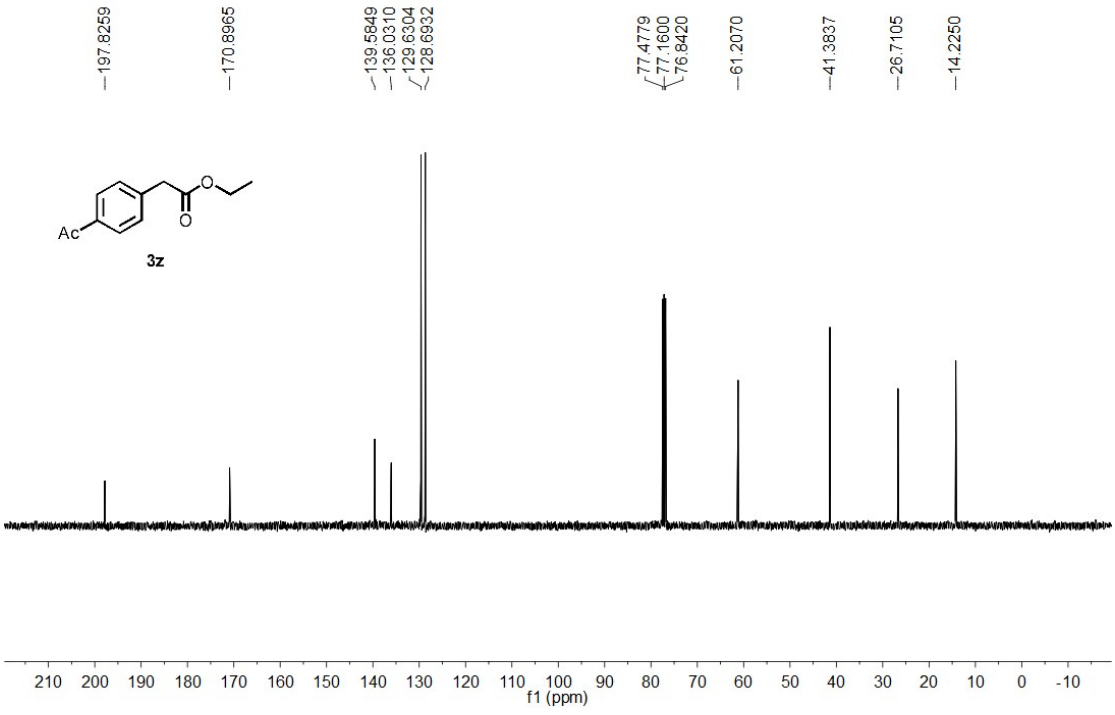
ZZP-92-1



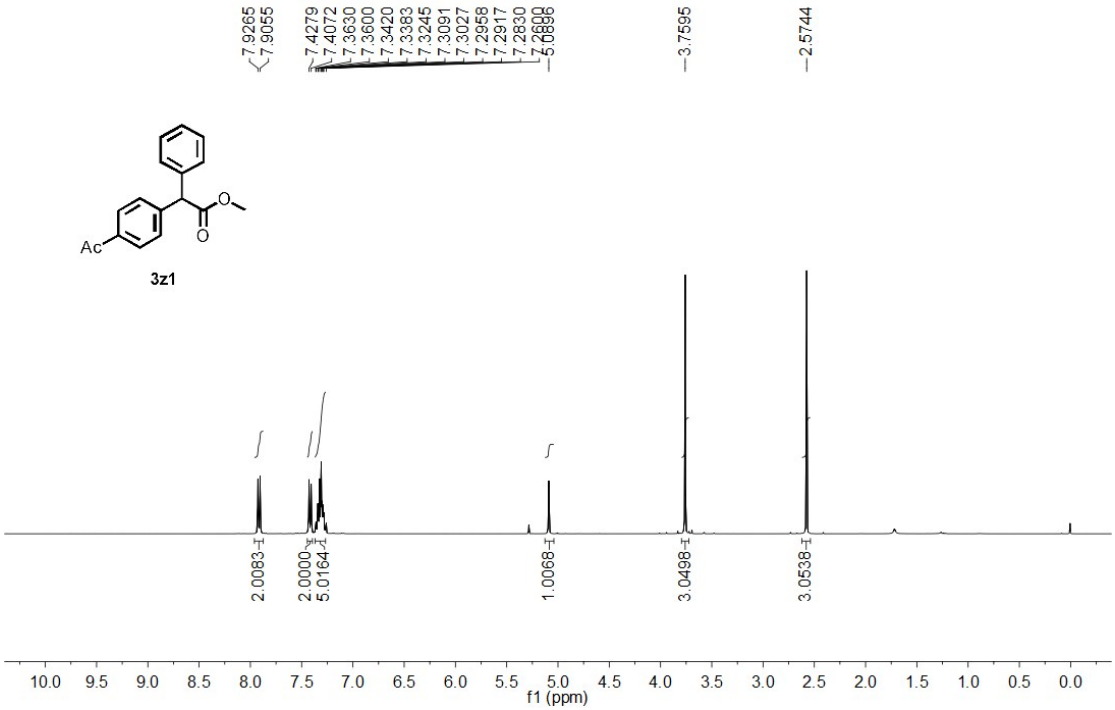
ZZP-425-1



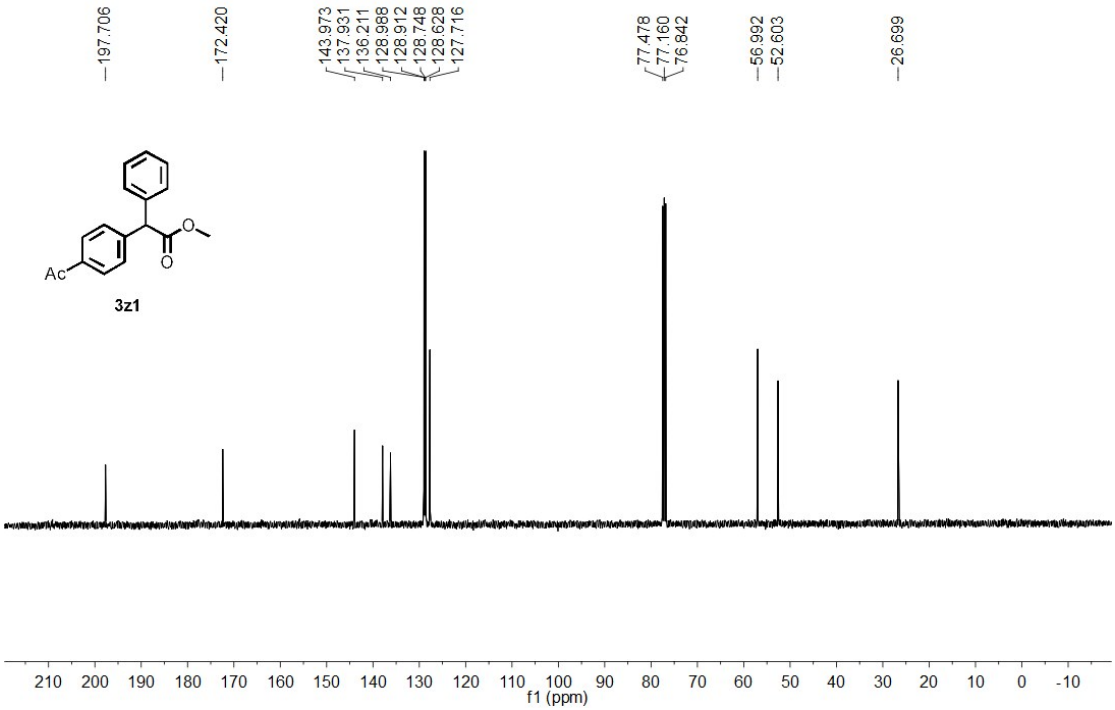
ZZP-425-1



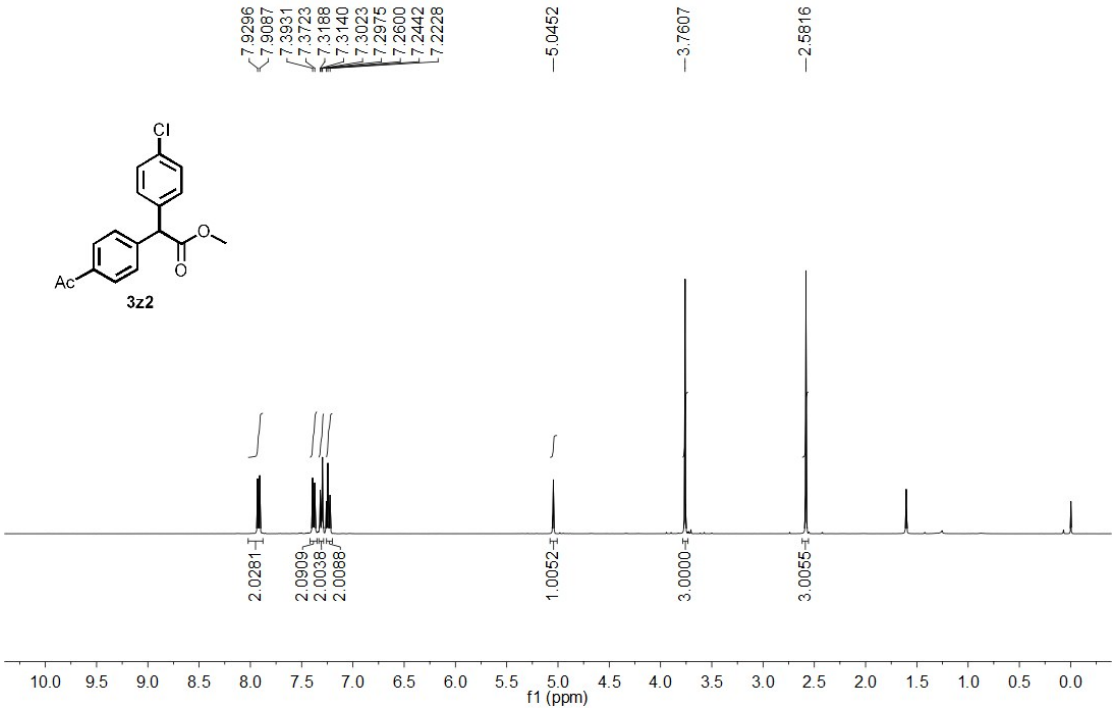
ZZP-108-1



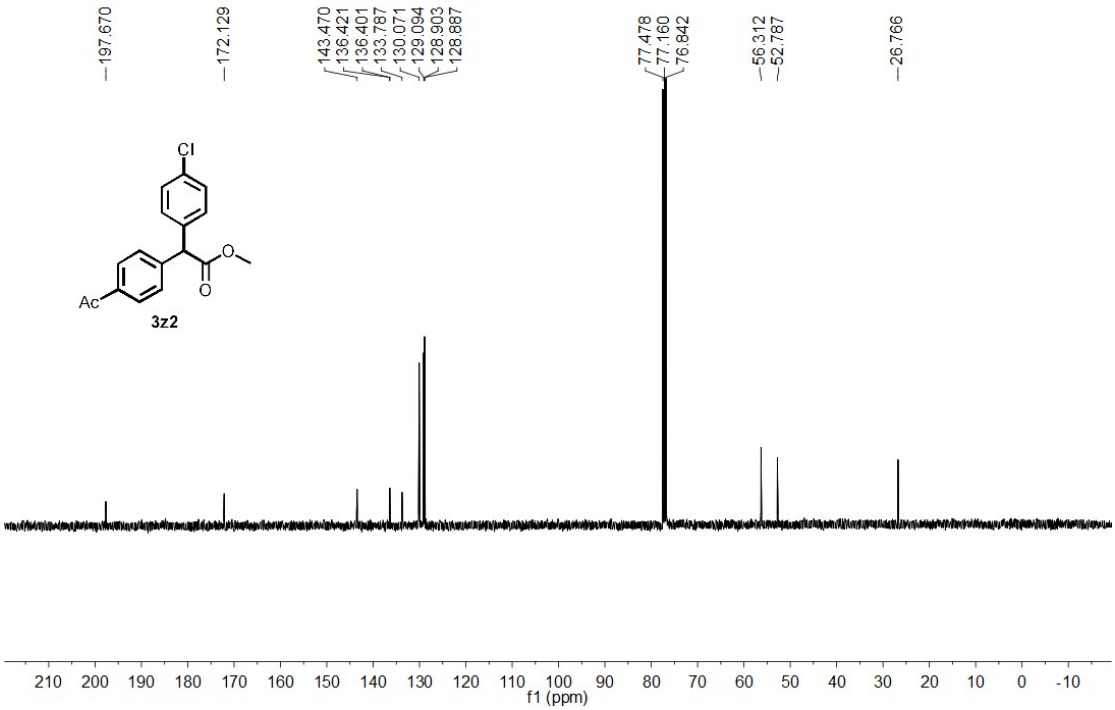
ZZP-108-1



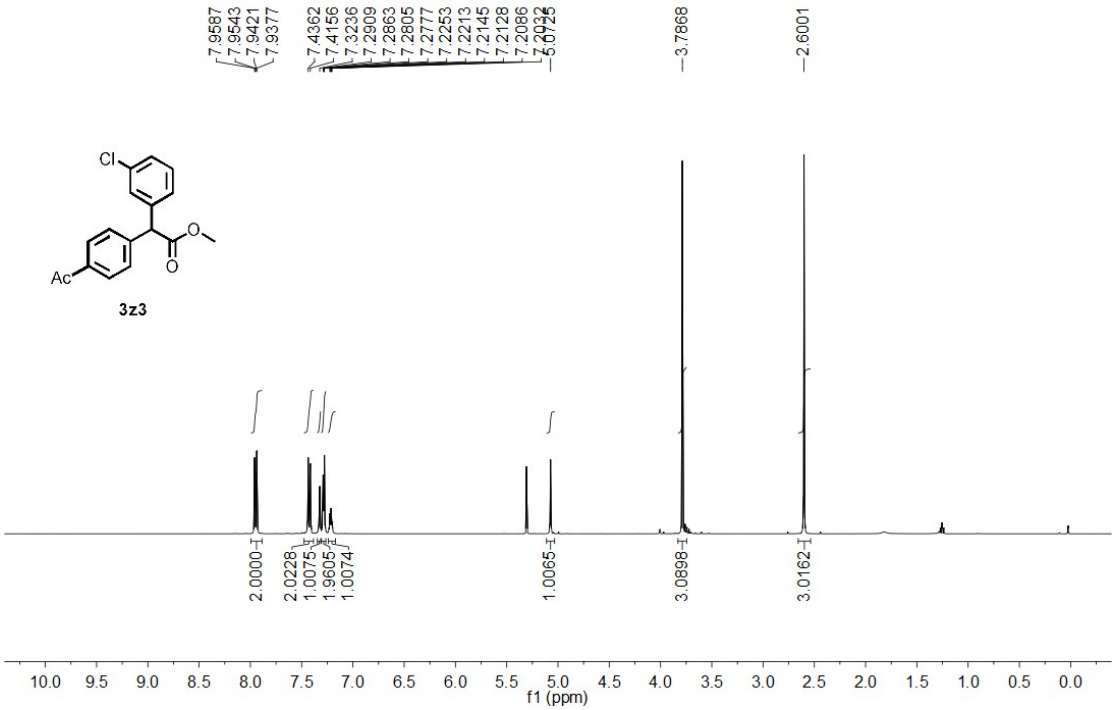
ZZP-400-1



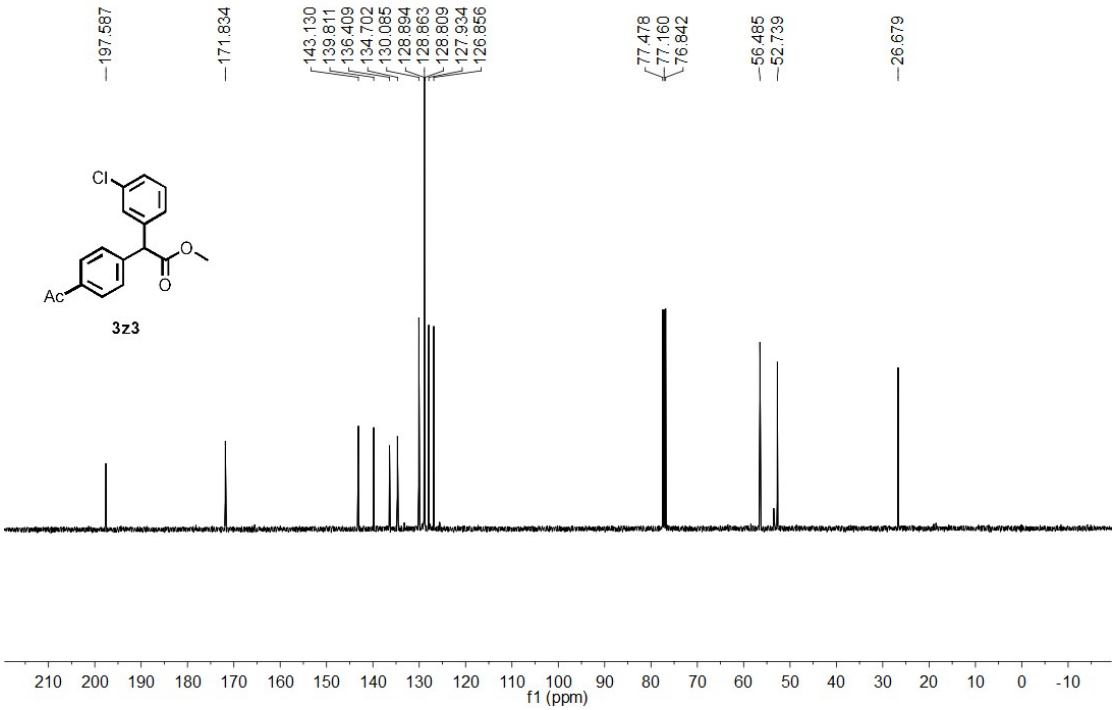
ZZP-400-2



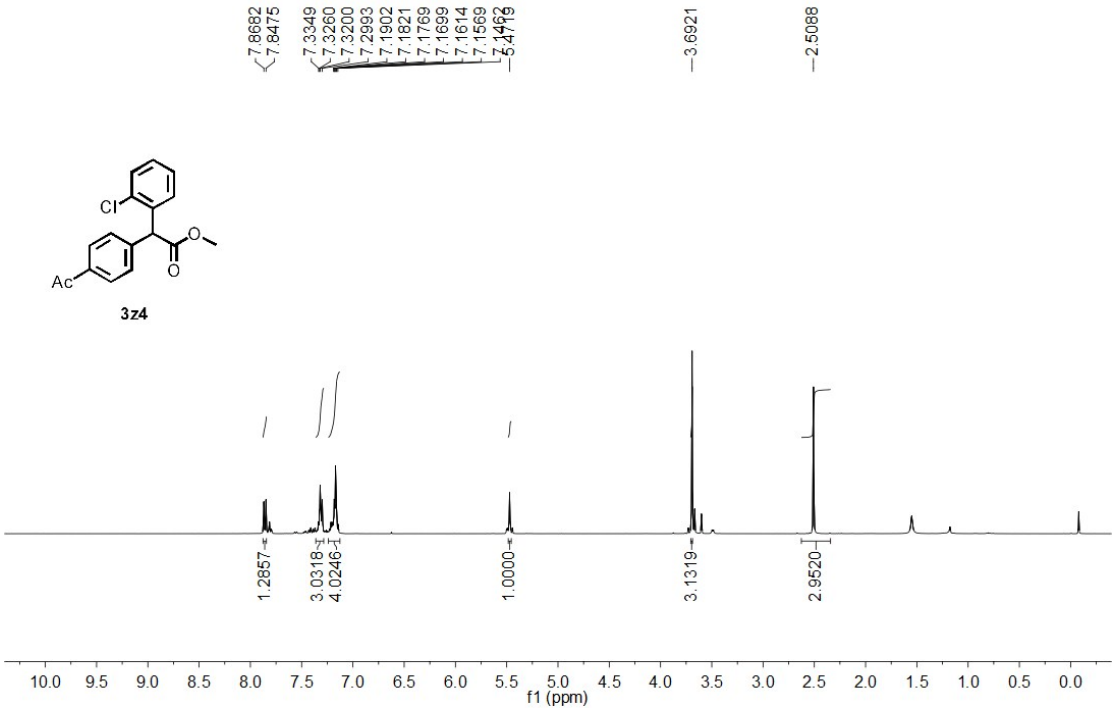
ZZP-109-2



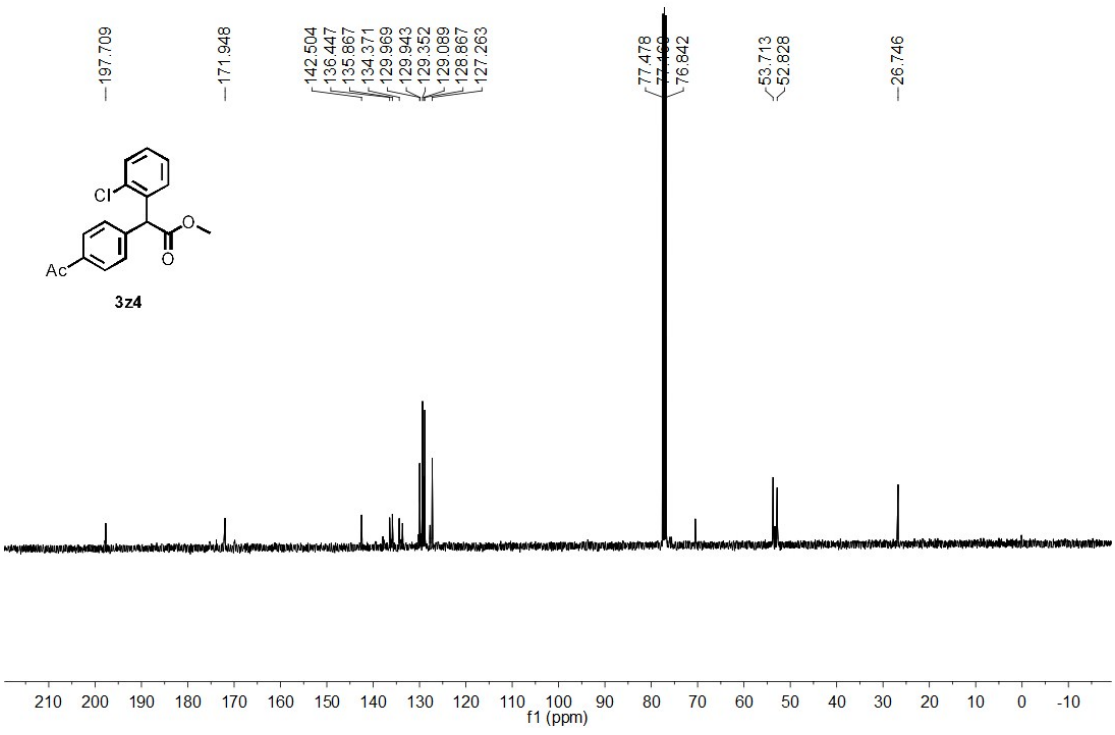
ZZP-109-2



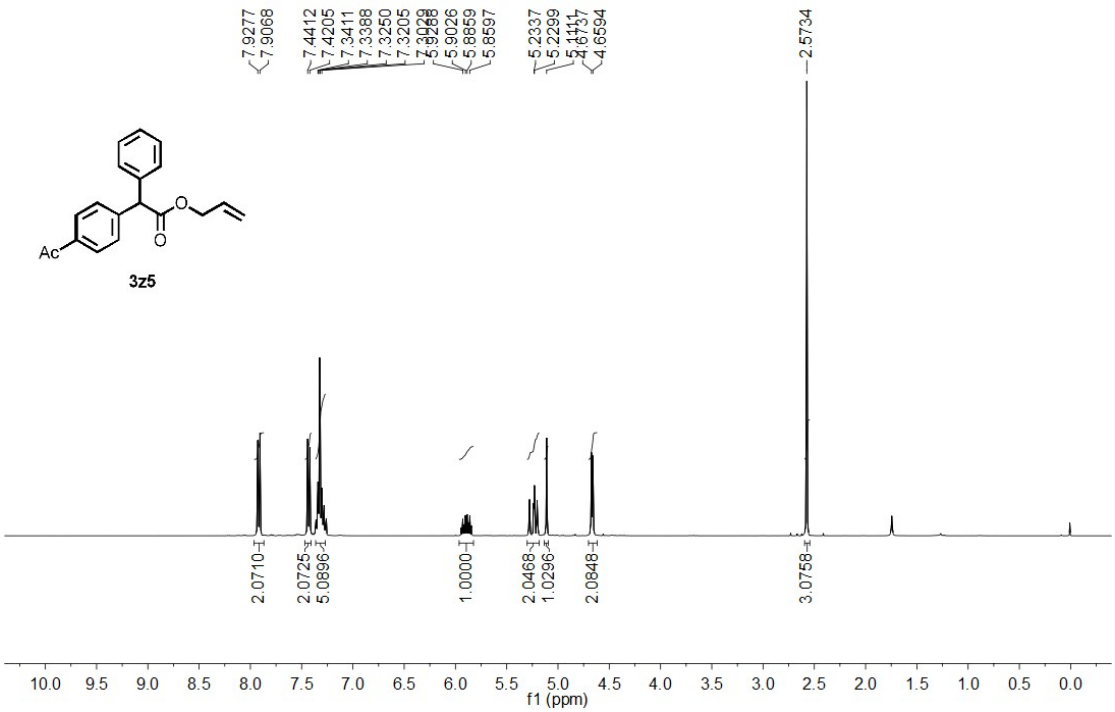
ZZP-114-2



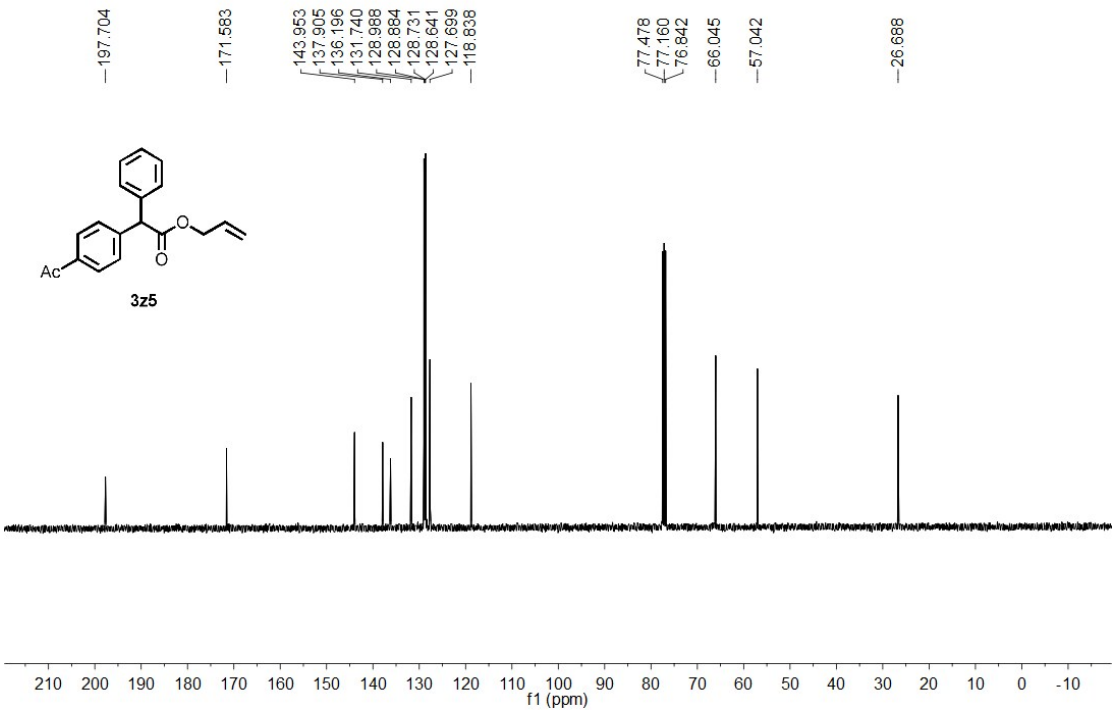
ZZP-114-2



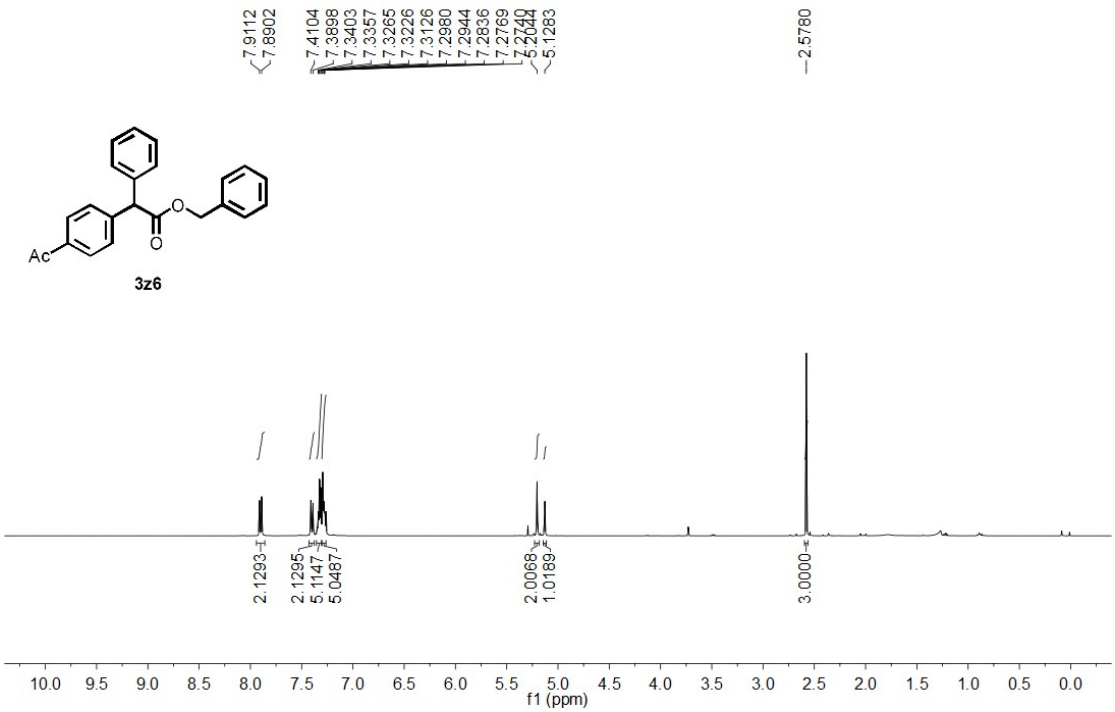
ZZP-107-2



ZZP-107-2



ZZP-17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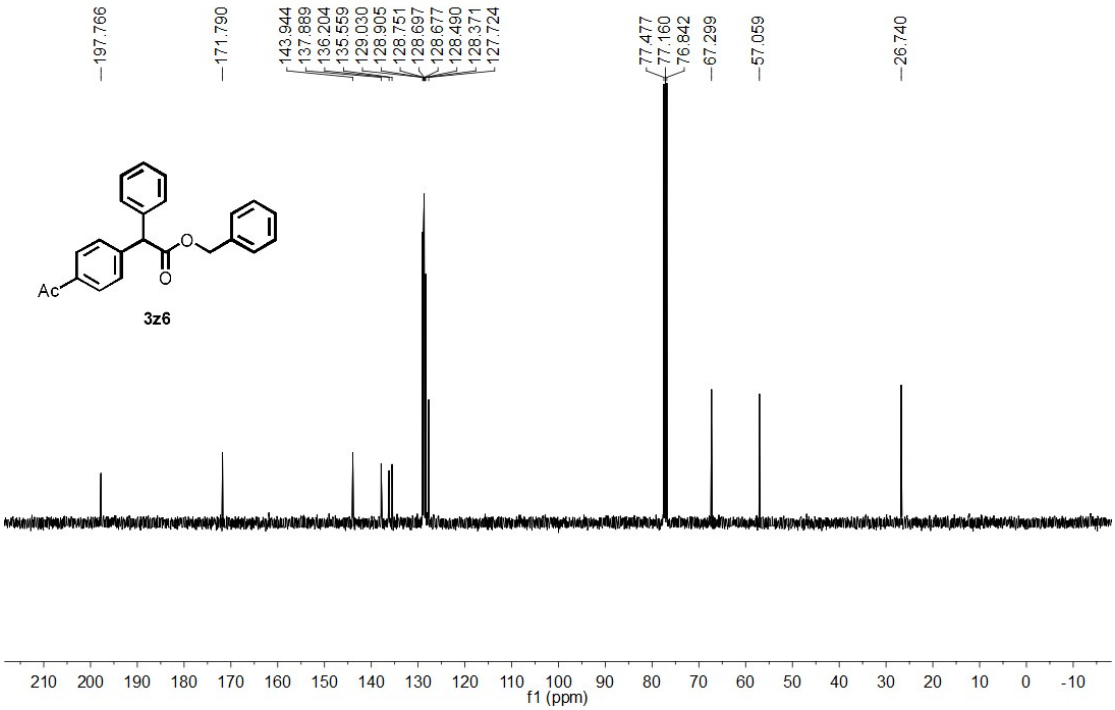
7.8902
7.4104
7.3898
7.3403
7.3357
7.3265
7.3226
7.3126
7.2980
7.2944
7.2836
7.2769
5.1283
2.5780
Ac
O
O
3z6
2.1293
2.1295
5.1147
5.0487
2.0068
1.0189
3.0000
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

ZZP-17


197.766
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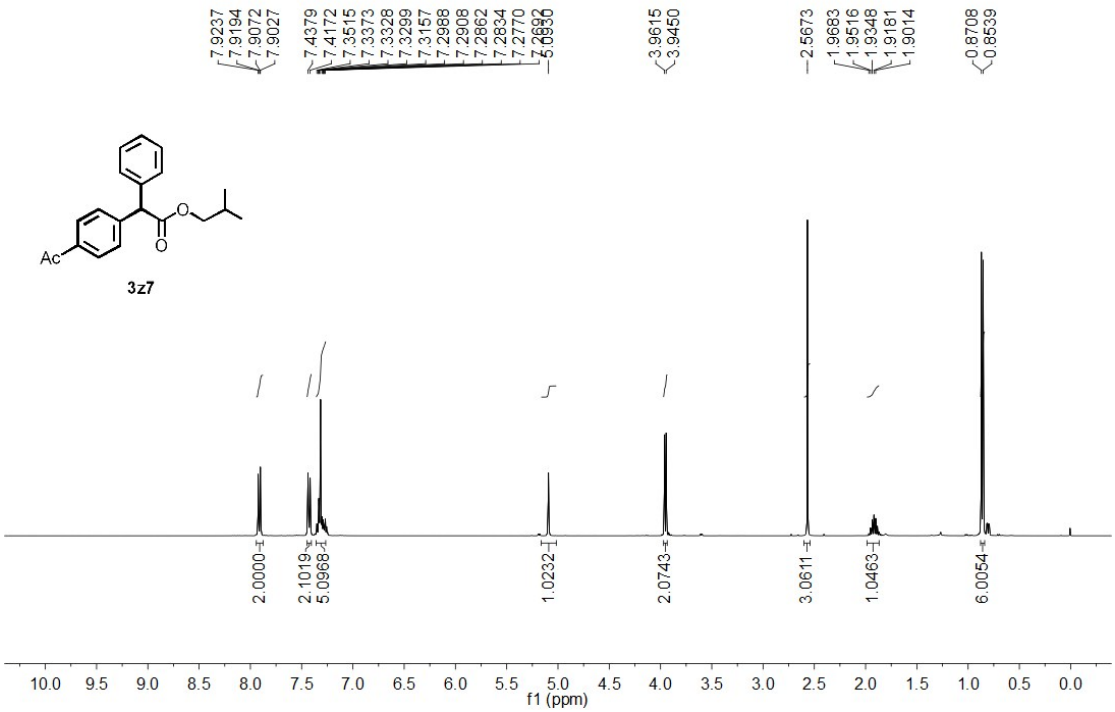
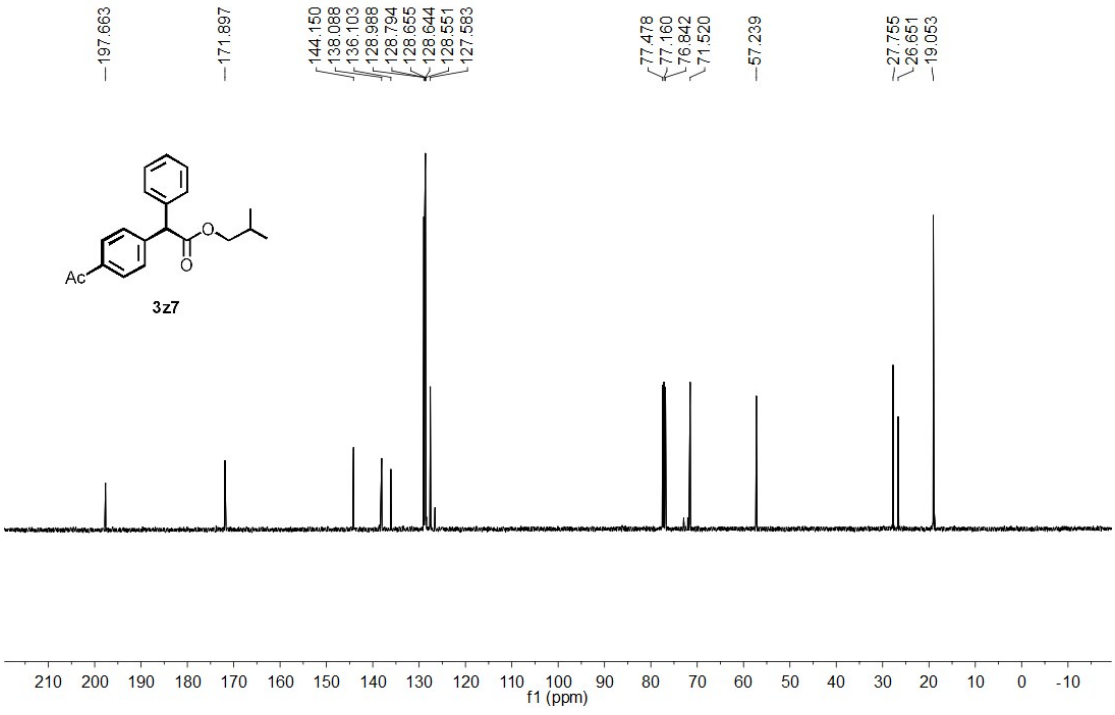
137.889
136.204
135.559
129.030
128.905
128.751
128.697
128.677
128.490
128.371
127.724
77.477
77.160
76.842
67.299
57.059
26.740
Ac
O
O
3z6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

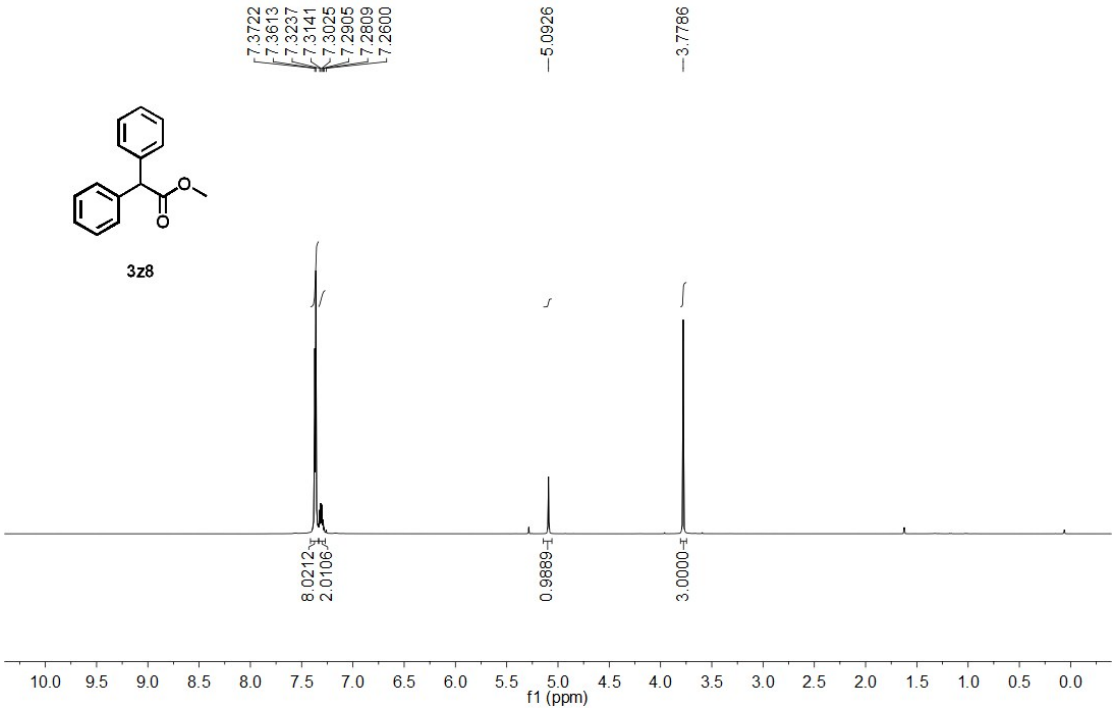
ZZP-93-1



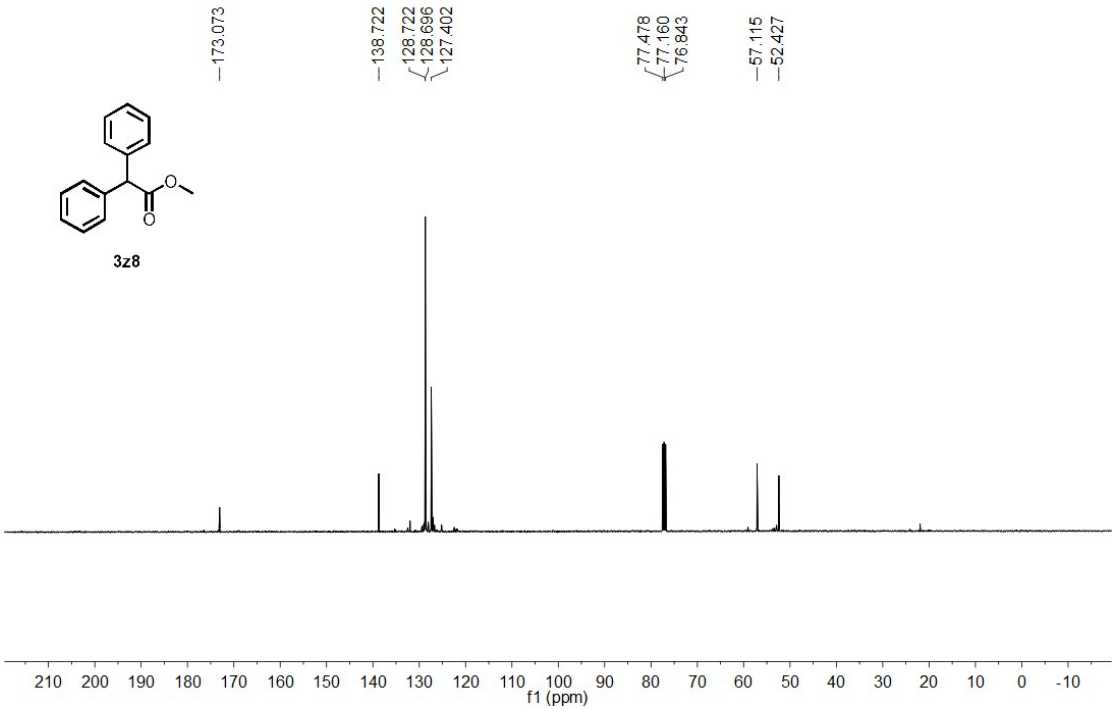
ZZP-93-1



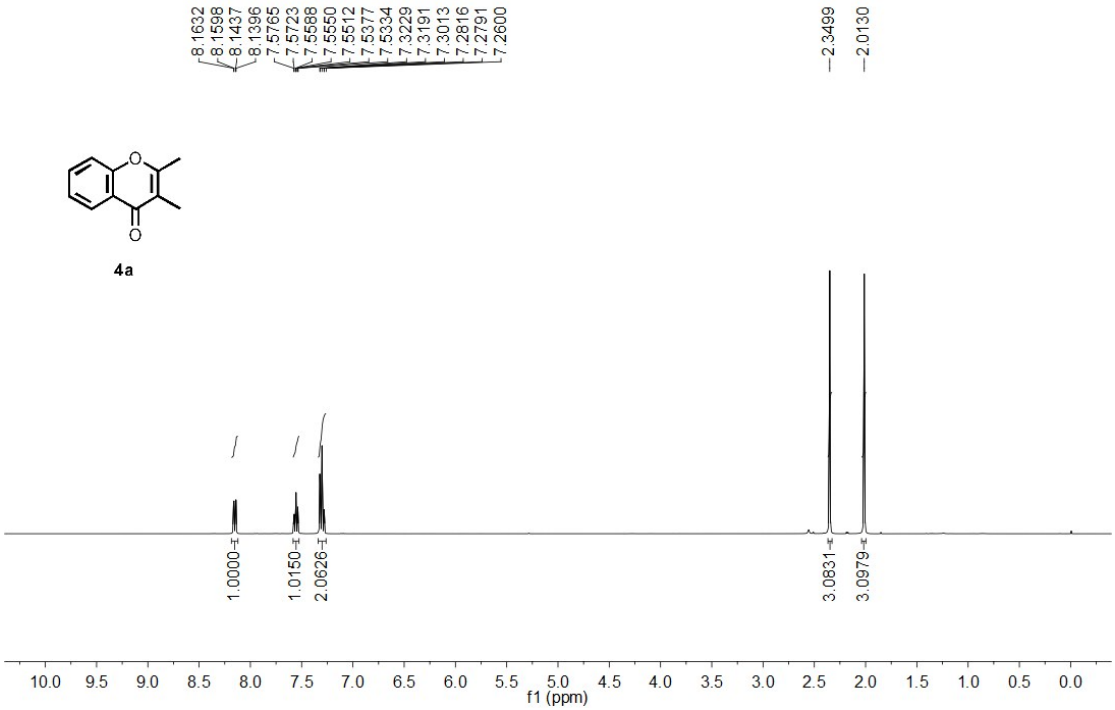
ZZP-358-1



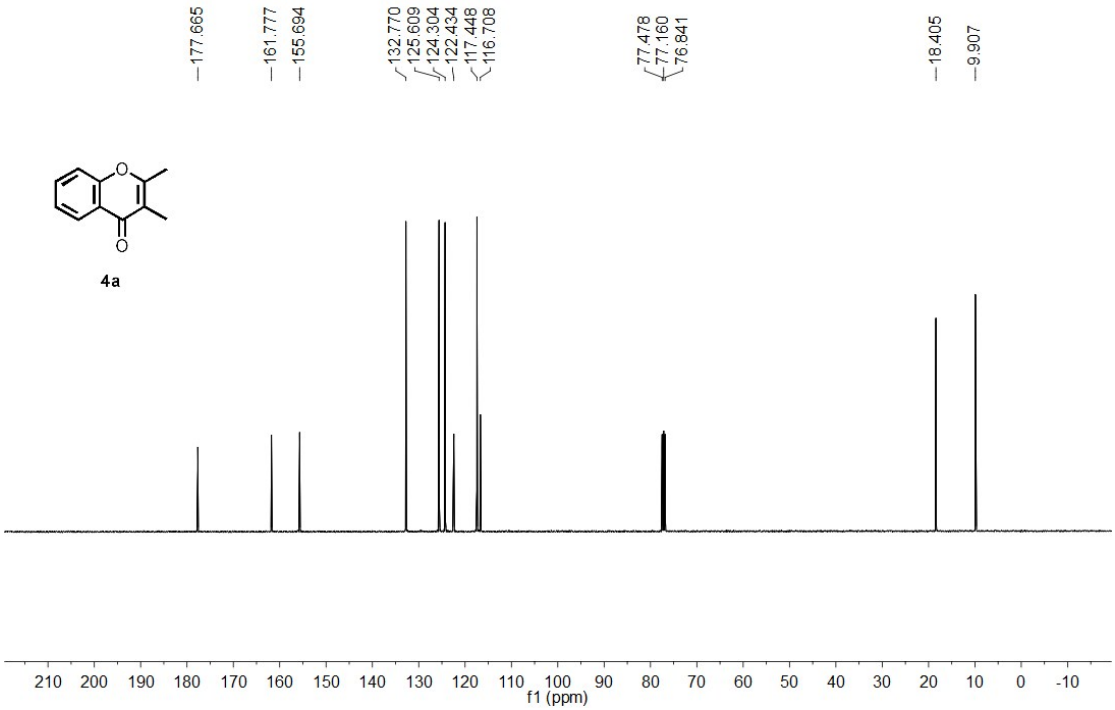
ZZP-252-3



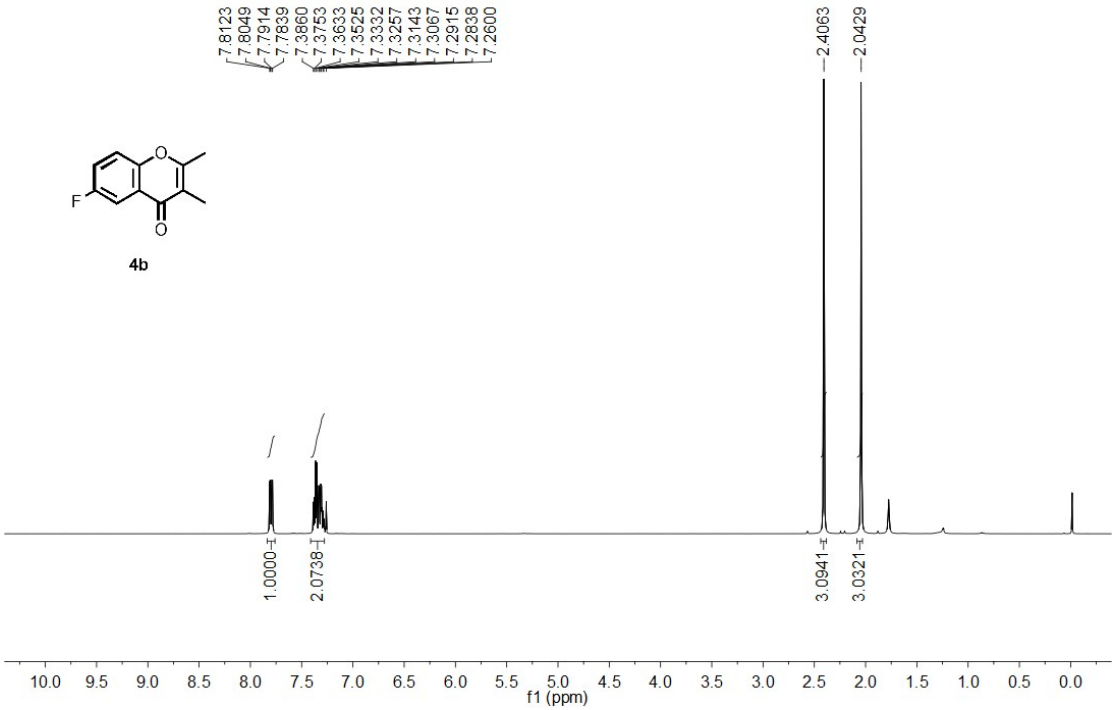
ZZP-137-1



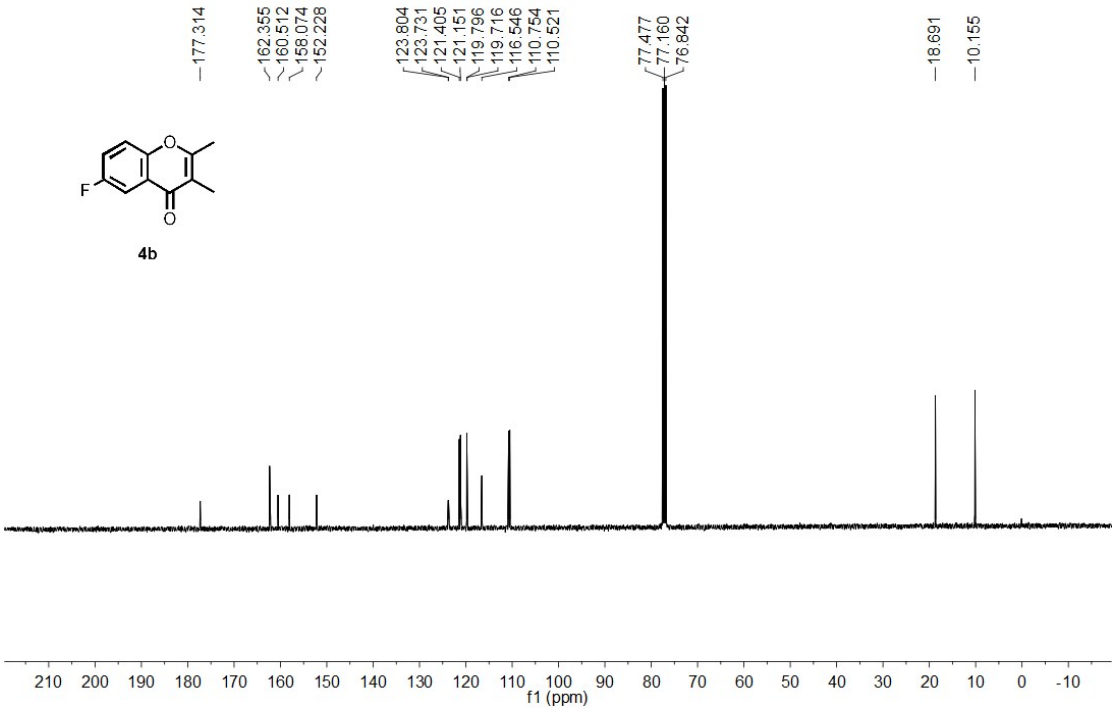
ZZP-137-1



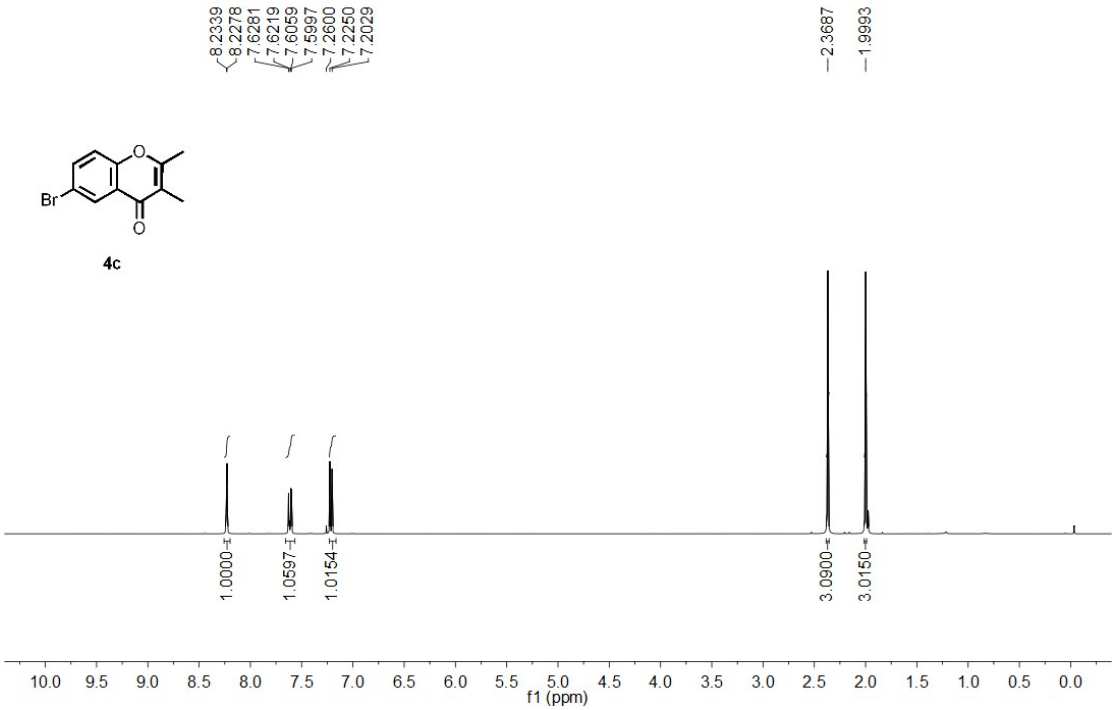
ZZP-131-1



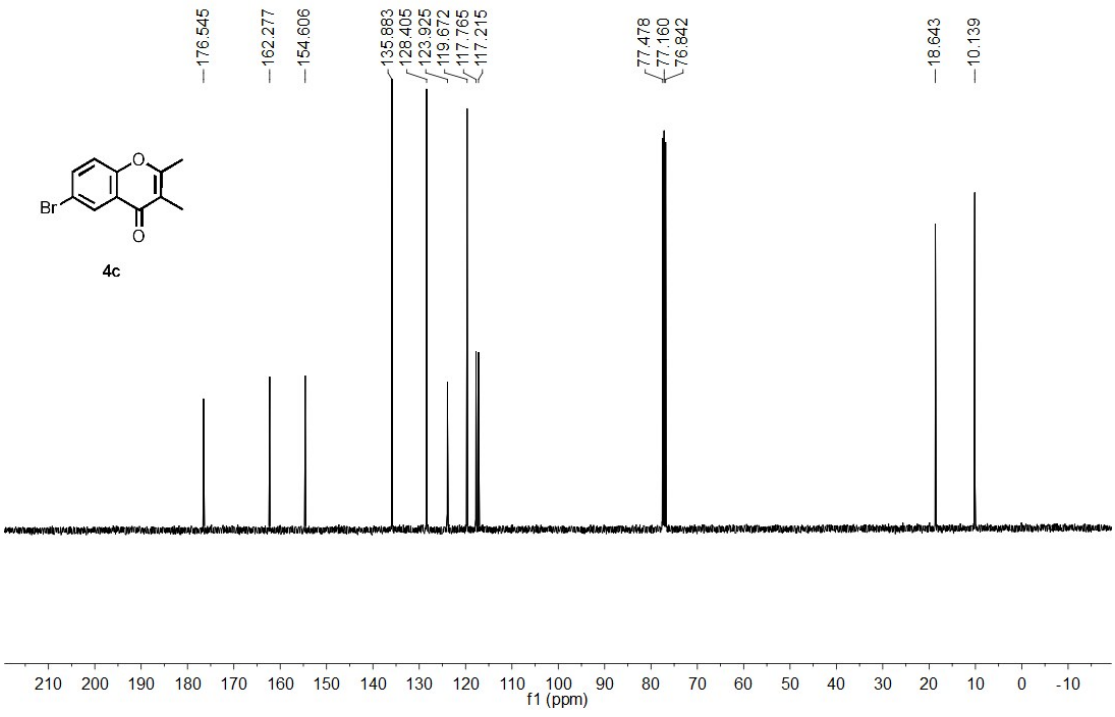
ZZP-131-1



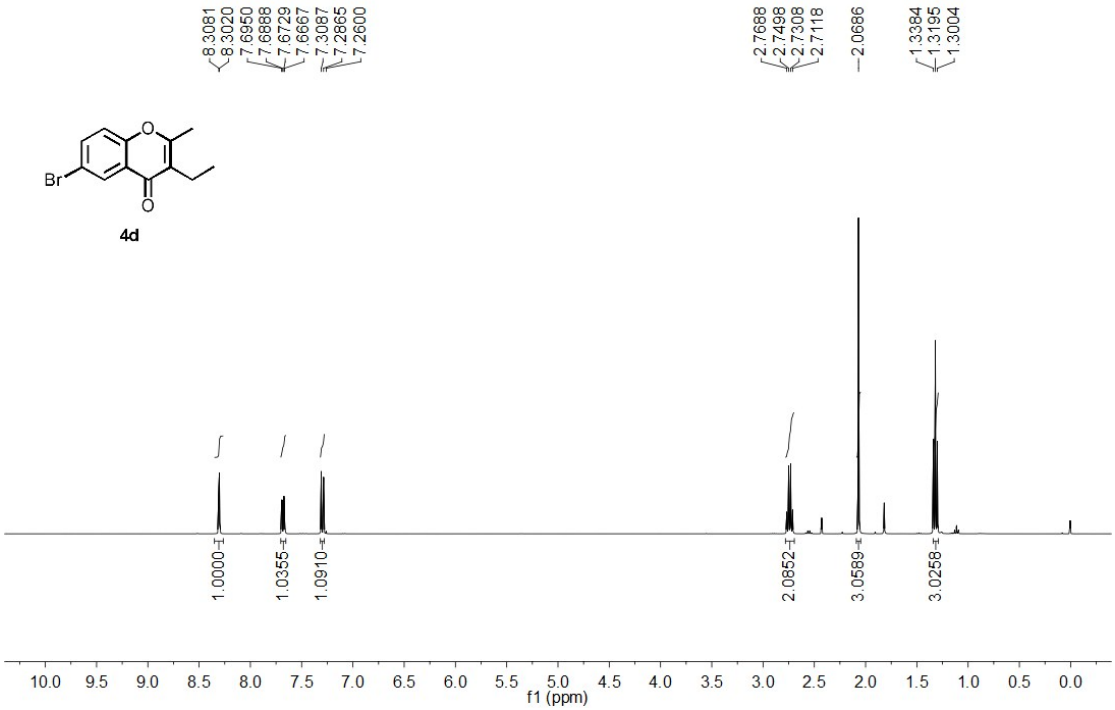
ZZP-133-1



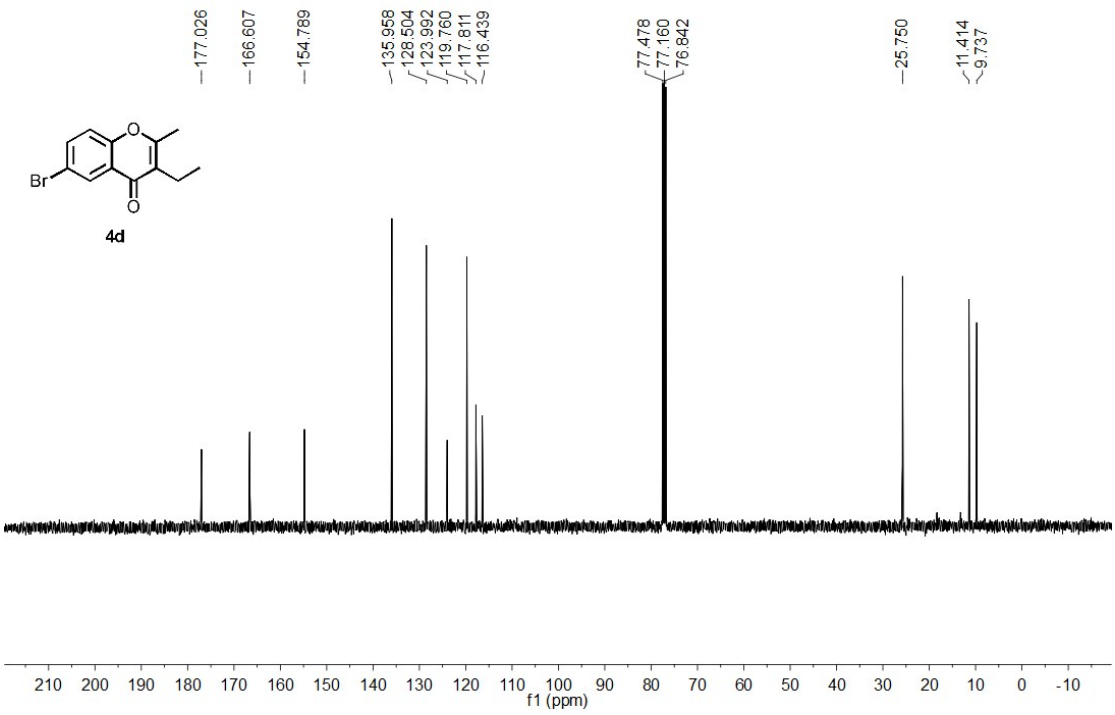
ZZP-133-1



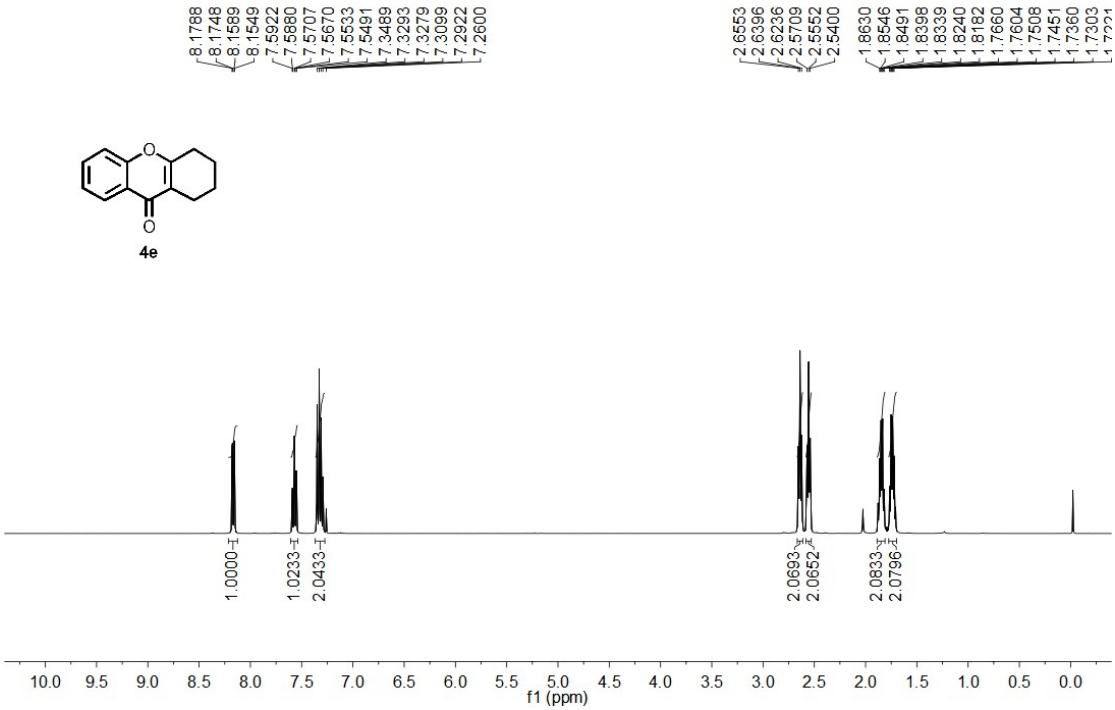
ZZP-213-2



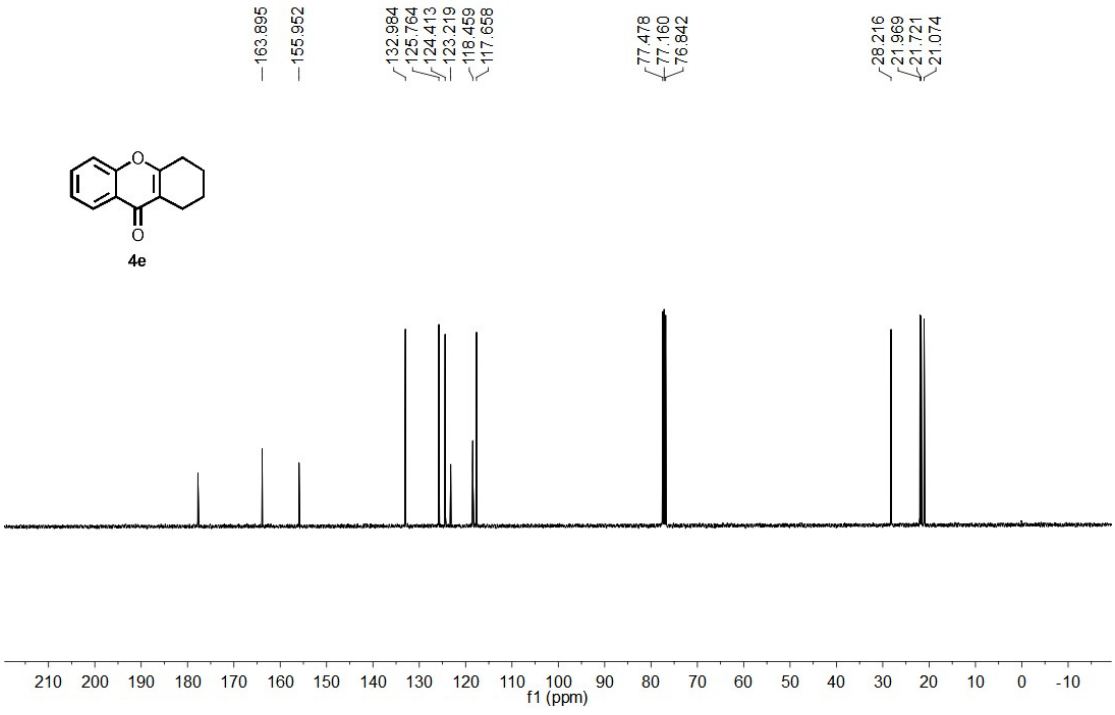
ZZP-213-2



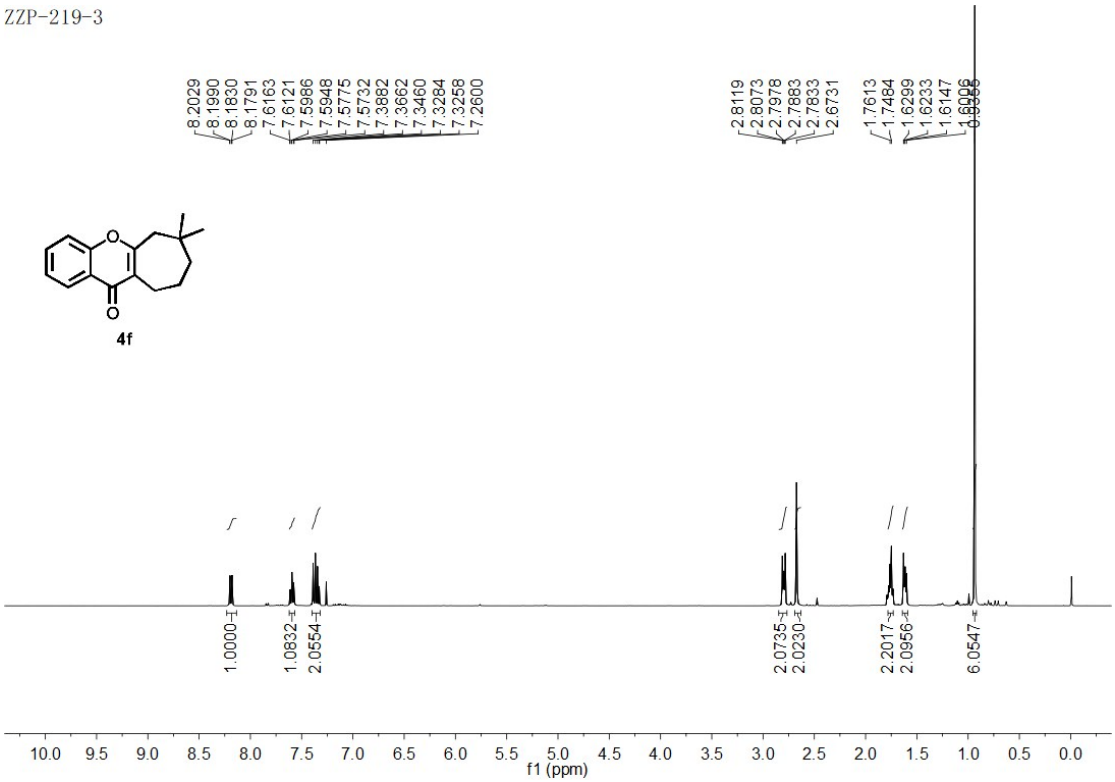
ZZP-223-1



ZZP-223-1

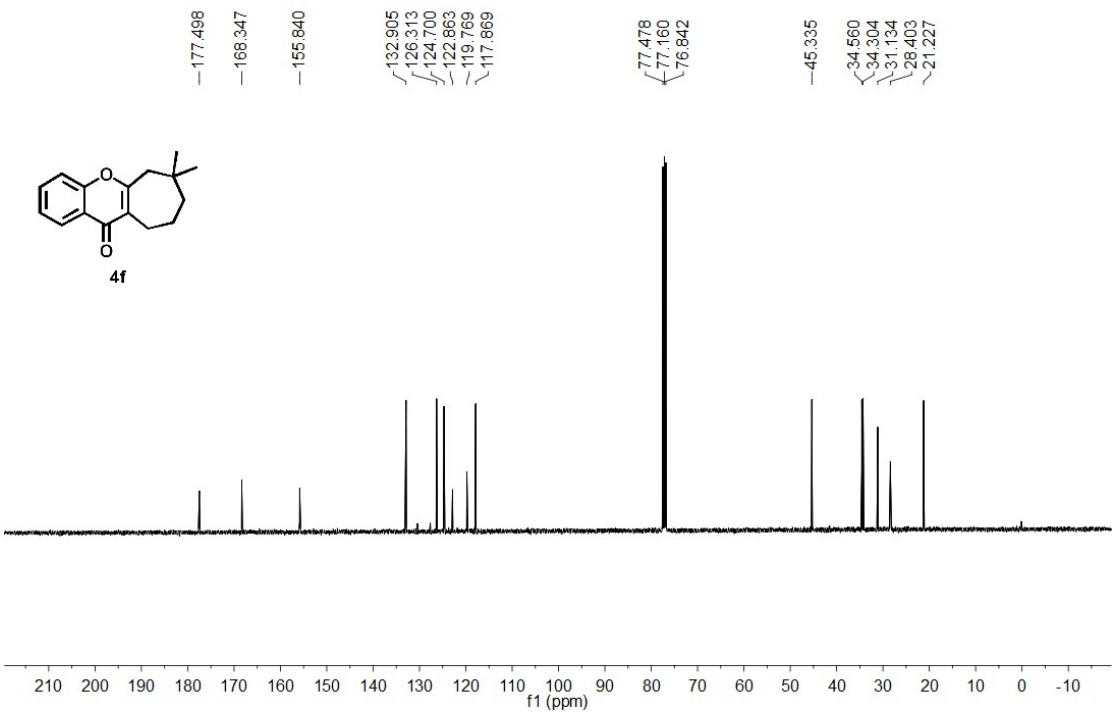


ZZP-219-3



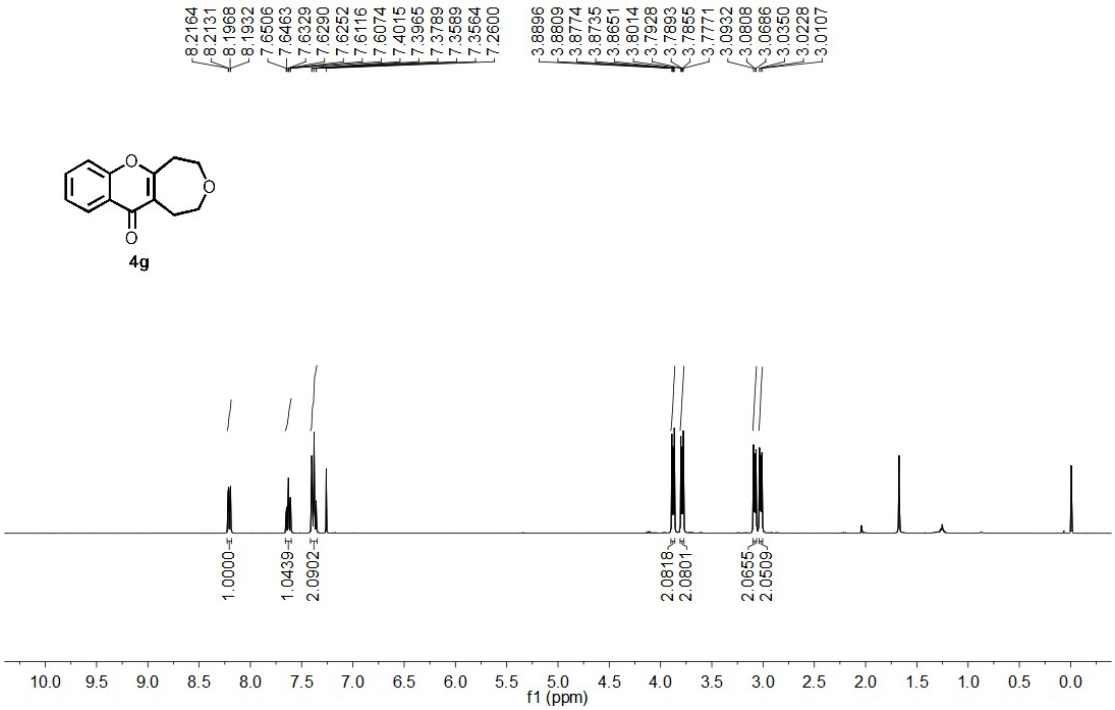
4f

ZZP-219-3

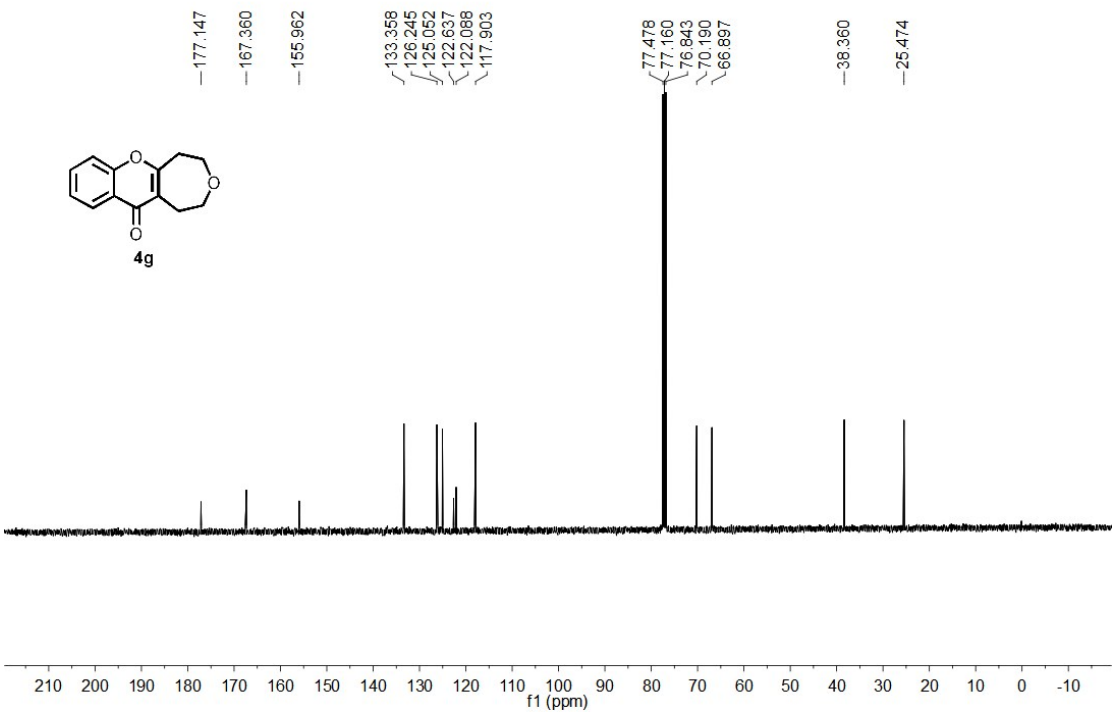


4f

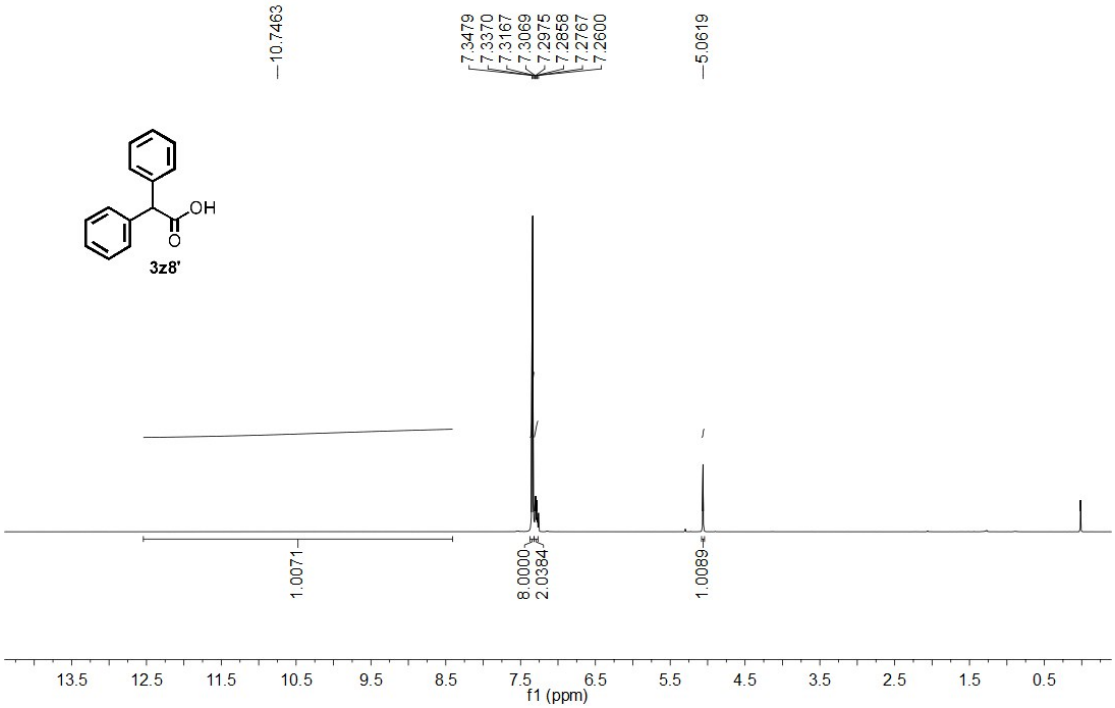
ZZP-222-1



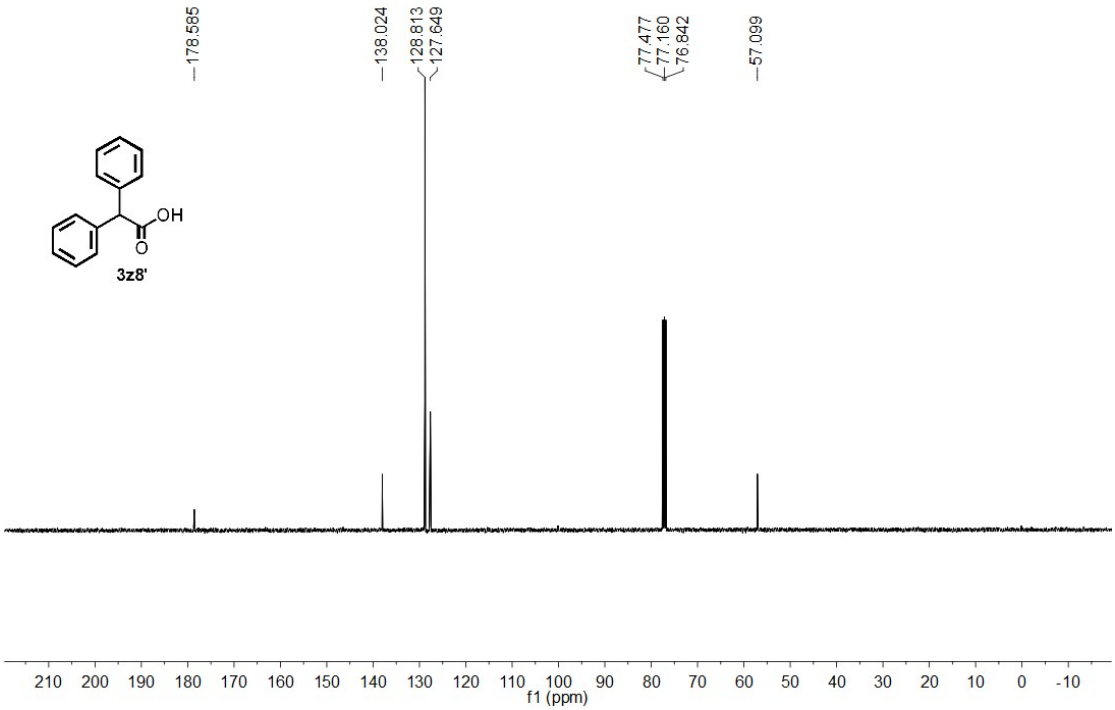
ZZP-222-1



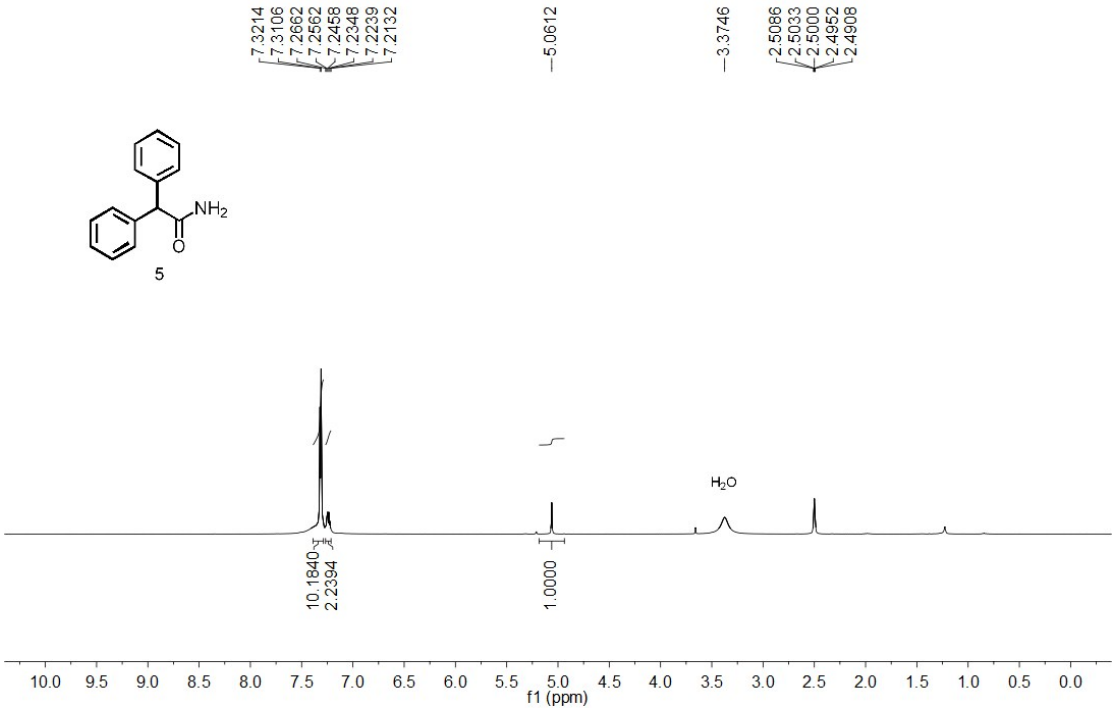
ZZP-402-1



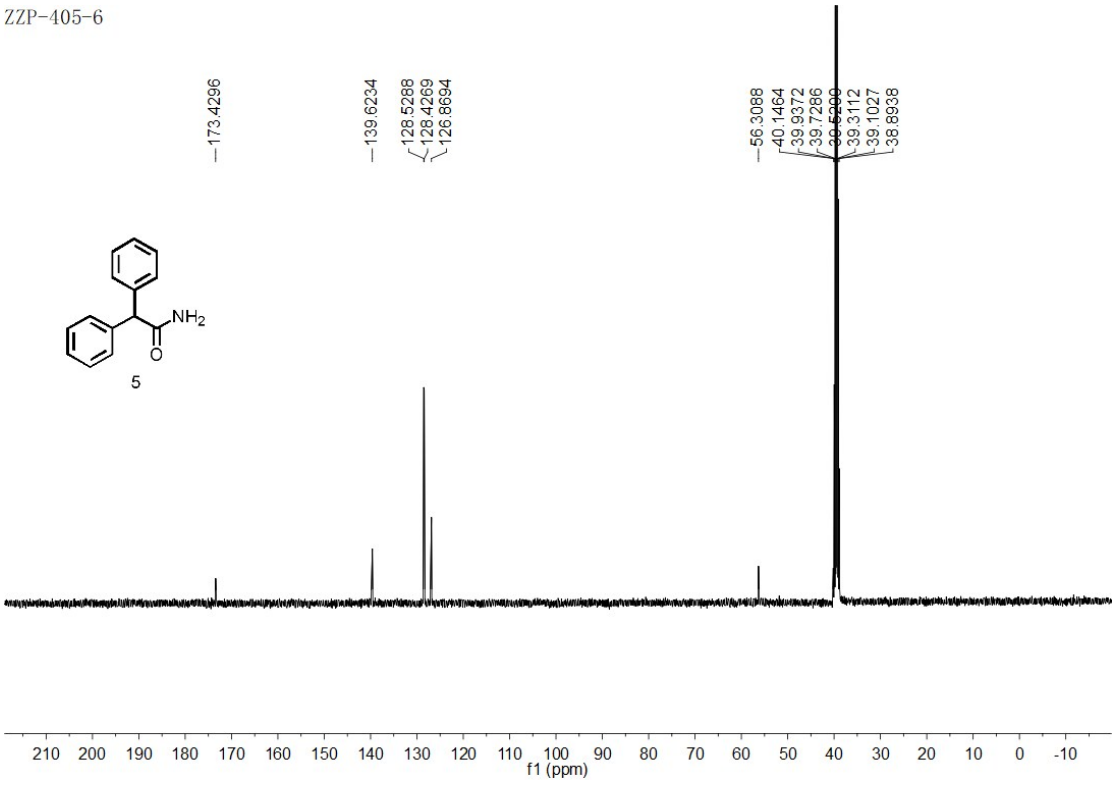
ZZP-402-1



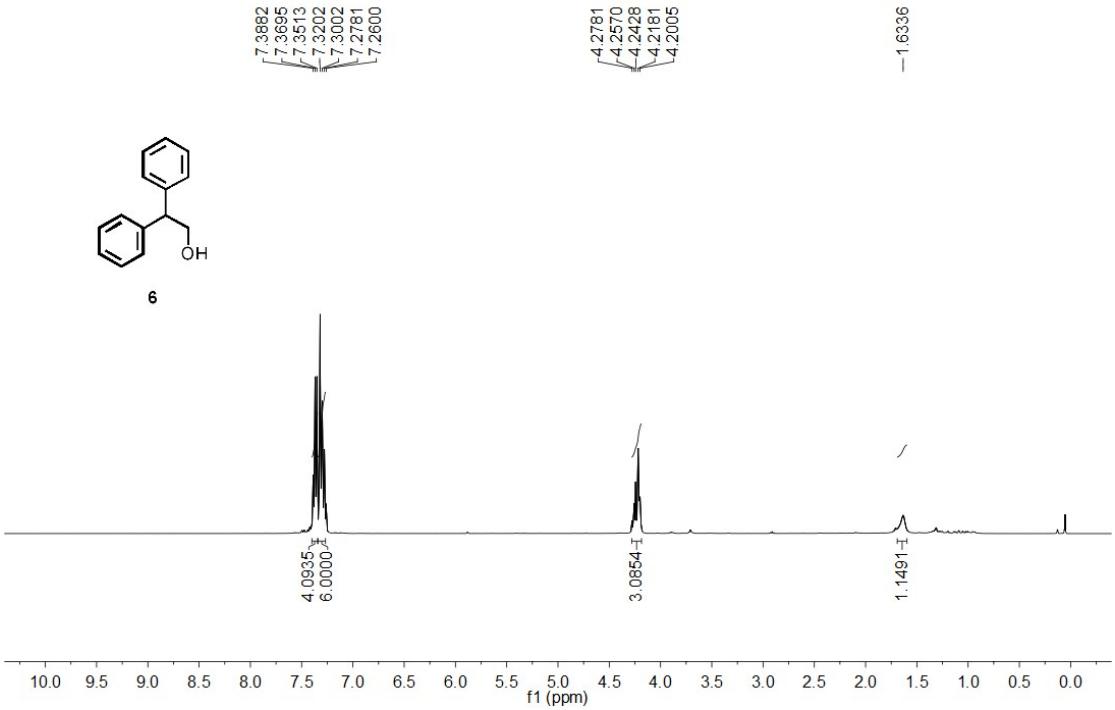
ZZP-405-5



ZZP-405-6



ZZP-404-1



ZZP-404-1