

Electronic Supplementary Material (ESI) for New Journal of Chemistry.

## **N-heterocyclic carbene - Catalyzed Synthesis of Functionalized 3-hydroxypyrrolidinones via an Aza-Michael/Aldol Domino Reaction**

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### **Table of Contents**

General information	S2
General procedures for the preparation of the products	S2
Spectra data of products	S3
Copies of <sup>1</sup> H and <sup>13</sup> C-NMR	S7
Copies of NOESY <sup>1</sup> HNMR	S18

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## 1 Experimental

### 1.1 General Remarks

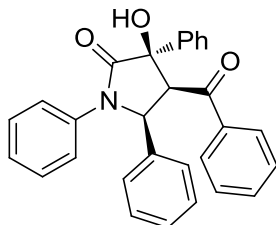
All of the reagents and solvents were commercially available and used without further purification. GC analyses were performed on an Agilent 7890A instrument.  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR were recorded on Bruker DRX 500 and tetramethylsilane (TMS) was used as a reference. The  $^1\text{H}$  NMR spectroscopic data of these precatalysts and products are in agreement with those reported in the literatures.<sup>1-7</sup>

### 1.2 General procedure for the Synthesis of 3-hydroxypyrrolidinones (3).

Under  $\text{N}_2$  atmosphere, to a solution of catalyst precursor A (0.03 mmol), 1 (0.36 mmol) and 2 (0.3 mmol) in ethanol (2 mL) was added the  $\text{KO}^t\text{Bu}$  (0.45 mmol). The reaction was stirred at rt (monitored by TLC) before it was quenched with  $\text{NH}_4\text{Cl}$  (4mL, sat. aq.). The layers were separated and the aqueous layer was extracted with EtOAc. The combined organic layers were washed with brine (5 mL), dried ( $\text{Na}_2\text{SO}_4$ ) and concentrated in vacuo. Flash column chromatography afforded 3-hydroxypyrrolidinones.

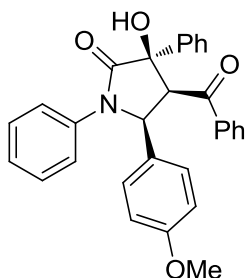
## 2. Characterization Data

### 4-benzoyl-3-hydroxy-1, 3, 5-triphenylpyrrolidin-2-one (**3a**)



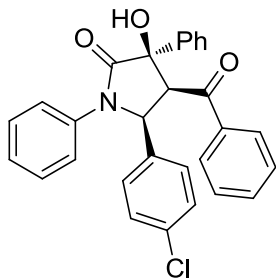
White solid; 75% yield.  $^1\text{H}$  NMR (500 MHz, DMSO)  $\delta$  7.44 (dd,  $J = 16.9, 7.8$  Hz, 4H), 7.39 – 7.33 (m, 3H), 7.32 – 7.25 (m, 5H), 7.23 (t,  $J = 7.7$  Hz, 2H), 7.18 (d,  $J = 7.7$  Hz, 2H), 7.10 (dt,  $J = 22.6, 7.6$  Hz, 4H), 6.83 (s, 1H), 6.16 (d,  $J = 8.2$  Hz, 1H), 4.27 (d,  $J = 8.2$  Hz, 1H);  $^{13}\text{C}$  NMR (126 MHz, DMSO)  $\delta$  193.88, 171.40, 141.22, 138.56, 136.82, 136.20, 132.35, 128.14, 127.91, 127.43, 127.24, 126.90, 125.73, 124.99, 123.60, 79.73, 63.41, 60.17; MS ( $\text{M}^+$ ), found 433; Anal. calcd for  $\text{C}_{29}\text{H}_{23}\text{NO}_3$ : C, 80.15; H, 5.55; N, 3.39; Found: C, 80.35; H, 5.35; N, 3.23.

### 4-benzoyl-3-hydroxy-5-(4-methoxyphenyl)-1, 3-diphenylpyrrolidin-2-one (**3b**)



White solid; 69% yield.  $^1\text{H}$  NMR (500 MHz, DMSO)  $\delta$  7.46 (d,  $J = 8.1$  Hz, 2H), 7.43 – 7.34 (m, 5H), 7.34 – 7.25 (m, 5H), 7.19 (d,  $J = 7.8$  Hz, 2H), 7.11 (dd,  $J = 16.3, 8.5$  Hz, 3H), 6.84 – 6.72 (m, 3H), 6.12 (d,  $J = 8.2$  Hz, 1H), 4.27 (d,  $J = 8.3$  Hz, 1H), 3.63 (s, 3H);  $^{13}\text{C}$  NMR (126 MHz, DMSO)  $\delta$  195.03, 172.39, 159.23, 142.38, 137.94, 137.33, 133.41, 131.28, 129.27, 128.96, 128.50, 128.31, 127.95, 126.82, 126.06, 124.84, 114.60, 80.82, 64.61, 60.84, 55.46; MS ( $\text{M}^+$ ), found 463; Anal. calcd for  $\text{C}_{30}\text{H}_{25}\text{NO}_4$ : C, 77.74; H, 5.44; N, 3.02; Found: C, 77.54; H, 5.59; N, 3.32.

### 4-benzoyl-5-(4-chlorophenyl)-3-hydroxy-1, 3-diphenylpyrrolidin-2-one (**3c**)

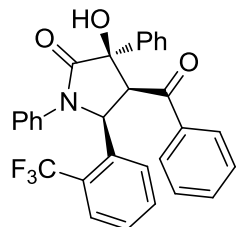


White solid; 75% yield.  $^1\text{H}$  NMR (500 MHz, DMSO)  $\delta$  7.52 – 7.46 (m, 4H), 7.41 – 7.34 (m, 3H), 7.30 (dd,  $J = 17.1, 8.1$  Hz, 7H), 7.22 (d,  $J = 7.8$  Hz, 2H), 7.14 – 7.04 (m, 3H), 6.89 (s, 1H), 6.22 (d,  $J = 8.2$  Hz, 1H), 4.31 (d,  $J = 8.2$  Hz, 1H).  $^{13}\text{C}$  NMR (126 MHz, DMSO)  $\delta$  195.04, 172.49, 142.10, 138.56,

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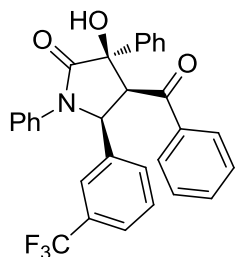
137.74, 137.25, 133.44, 132.98, 130.06, 129.22, 129.08, 128.62, 128.46, 128.29, 128.03, 126.92, 126.20, 124.69, 80.84, 64.30, 60.64; MS ( $M^+$ ), found 467; Anal. calcd for  $C_{29}H_{22}ClNO_3$ : C, 74.44; H, 4.74; N, 2.99; Found: C, 74.14; H, 4.94; N, 2.76.

4-benzoyl-3-hydroxy-1, 3-diphenyl-5-(2-(trifluoromethyl) phenyl) pyrrolidin-2-one  
(3d)



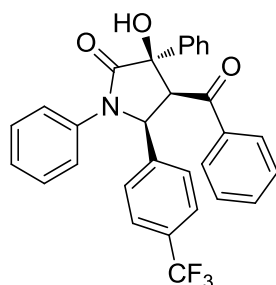
White solid; 52% yield.  $^1H$  NMR (500 MHz, DMSO)  $\delta$  7.63 (d,  $J = 7.8$  Hz, 2H), 7.58 (d,  $J = 7.7$  Hz, 1H), 7.48 (dd,  $J = 17.5, 9.1$  Hz, 5H), 7.37 (d,  $J = 4.0$  Hz, 2H), 7.32 (t,  $J = 7.4$  Hz, 5H), 7.26 (dd,  $J = 14.8, 7.1$  Hz, 3H), 7.12 (t,  $J = 7.4$  Hz, 1H), 6.72 (s, 1H), 6.19 (d,  $J = 3.2$  Hz, 1H), 4.60 (d,  $J = 3.2$  Hz, 1H).  $^{13}C$  NMR (126 MHz, DMSO)  $\delta$  196.27, 173.21, 142.18, 137.98, 137.91, 137.78, 133.67, 133.17, 129.56, 129.13, 129.03, 128.76, 128.69, 127.07, 126.70, 123.98, 80.38, 59.11, 58.26; MS ( $M^+$ ), found 501; Anal. calcd for  $C_{30}H_{22}F_3NO_3$ : C, 71.85; H, 4.42; N, 2.79; Found: C, 71.77; H, 4.62; N, 2.91.

4-benzoyl-3-hydroxy-1, 3-diphenyl-5-(3-(trifluoromethyl)phenyl)pyrrolidin-2-one  
(3e)



White solid; 70% yield.  $^1H$  NMR (500 MHz, DMSO)  $\delta$  7.93 – 7.64 (m, 2H), 7.48 (dt,  $J = 25.2, 8.2$  Hz, 4H), 7.43 – 7.34 (m, 3H), 7.34 – 7.21 (m, 7H), 7.09 (q,  $J = 8.1$  Hz, 3H), 6.89 (d,  $J = 9.6$  Hz, 1H), 6.32 (t,  $J = 8.6$  Hz, 1H), 4.40 (t,  $J = 6.9$  Hz, 1H).  $^{13}C$  NMR (126 MHz, DMSO)  $\delta$  195.39, 172.59, 141.85, 140.81, 137.68, 137.34, 133.39, 132.18, 130.31, 129.12, 128.68, 128.42, 128.24, 128.05, 127.02, 126.27, 125.23, 125.03, 124.66, 80.85, 63.77, 60.85; MS ( $M^+$ ), found 501; Anal. calcd for  $C_{30}H_{22}F_3NO_3$ : C, 71.85; H, 4.42; N, 2.79; Found: C, 71.75; H, 4.59; N, 2.99.

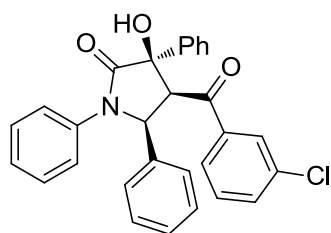
4-benzoyl-3-hydroxy-1, 3-diphenyl-5-(4-(trifluoromethyl) phenyl) pyrrolidin-2-one  
(3f)



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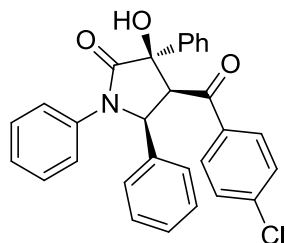
White solid; 72% yield.  $^1\text{H}$  NMR (500 MHz, DMSO)  $\delta$  7.70 (d,  $J = 8.2$  Hz, 2H), 7.60 (d,  $J = 8.4$  Hz, 2H), 7.51 (d,  $J = 8.3$  Hz, 2H), 7.41 – 7.35 (m, 3H), 7.35 – 7.30 (m, 2H), 7.28 (dd,  $J = 6.4, 3.6$  Hz, 3H), 7.23 (d,  $J = 7.7$  Hz, 2H), 7.10 (dt,  $J = 19.9, 7.8$  Hz, 3H), 6.93 (s, 1H), 6.33 (d,  $J = 8.1$  Hz, 1H), 4.35 (d,  $J = 8.2$  Hz, 1H);  $^{13}\text{C}$  NMR (126 MHz, DMSO)  $\delta$  195.04, 172.58, 144.38, 141.98, 137.71, 137.23, 133.44, 129.14, 128.99, 128.67, 128.43, 128.27, 128.04, 126.92, 126.23, 126.08, 124.49, 80.81, 64.06, 60.71; MS ( $\text{M}^+$ ), found 501; Anal. calcd for  $\text{C}_{30}\text{H}_{22}\text{F}_3\text{NO}_3$ : C, 71.85; H, 4.42; N, 2.79 ; Found: C, 71.85; H, 4.58; N, 2.85.

4-(3-chlorobenzoyl)-3-hydroxy-1, 3, 5-triphenylpyrrolidin-2-one (**3g**)



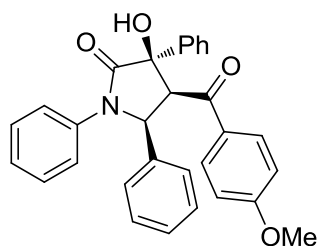
White solid; 64% yield.  $^1\text{H}$  NMR (500 MHz, DMSO)  $\delta$  7.40 (d,  $J = 7.9$  Hz, 5H), 7.30 (d,  $J = 3.9$  Hz, 2H), 7.24 (t,  $J = 7.7$  Hz, 5H), 7.19 (t,  $J = 7.6$  Hz, 3H), 7.14 – 7.00 (m, 4H), 6.86 (s, 1H), 6.10 (d,  $J = 8.2$  Hz, 1H), 4.25 (d,  $J = 8.2$  Hz, 1H).  $^{13}\text{C}$  NMR (126 MHz, DMSO)  $\delta$  193.16, 171.27, 140.90, 138.26, 137.90, 136.74, 132.48, 132.06, 129.37, 128.09, 127.91, 127.58, 127.33, 127.24, 127.14, 125.92, 125.70, 125.05, 123.66, 79.68, 63.63, 59.97; MS ( $\text{M}^+$ ), found 467; Anal. calcd for  $\text{C}_{29}\text{H}_{22}\text{ClNO}_3$ : C, 74.44; H, 4.74; N, 2.99; Found: C, 74.68; H, 4.54; N, 2.79.

4-(4-chlorobenzoyl)-3-hydroxy-1, 3, 5-triphenylpyrrolidin-2-one (**3h**)



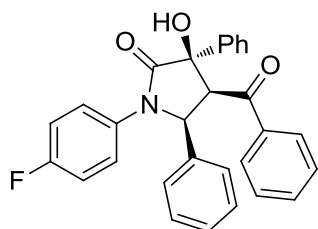
White solid; 67% yield.  $^1\text{H}$  NMR (500 MHz, DMSO)  $\delta$  7.45 (dd,  $J = 13.2, 7.7$  Hz, 4H), 7.35 (dt,  $J = 9.4, 3.4$  Hz, 2H), 7.32 – 7.25 (m, 5H), 7.26 – 7.16 (m, 4H), 7.16 – 7.06 (m, 4H), 6.91 (s, 1H), 6.16 (d,  $J = 8.1$  Hz, 1H), 4.28 (d,  $J = 8.2$  Hz, 1H).  $^{13}\text{C}$  NMR (126 MHz, DMSO)  $\delta$  194.27, 172.39, 142.07, 139.45, 138.34, 137.85, 136.02, 130.39, 129.22, 129.01, 128.56, 128.36, 128.10, 126.85, 126.12, 124.70, 80.77, 64.57, 61.10 ; MS ( $\text{M}^+$ ), found 467 Anal. calcd for  $\text{C}_{29}\text{H}_{22}\text{ClNO}_3$ : C, 74.44; H, 4.74; N, 2.99; Found: C, 74.63; H, 4.58; N, 2.68.

3-hydroxy-4-(4-methoxybenzoyl)-1, 3, 5-triphenylpyrrolidin-2-one (**3i**)



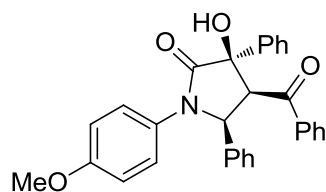
White solid; 71% yield.  $^1\text{H}$  NMR (500 MHz, DMSO)  $\delta$  7.45 (d,  $J$  = 8.1 Hz, 2H), 7.42 – 7.36 (m, 4H), 7.34 – 7.26 (m, 5H), 7.21 (t,  $J$  = 7.4 Hz, 2H), 7.17 – 7.06 (m, 4H), 6.76 (s, 1H), 6.58 (d,  $J$  = 8.1 Hz, 2H), 6.15 (d,  $J$  = 8.2 Hz, 1H), 4.19 (d,  $J$  = 8.1 Hz, 1H), 3.67 (s, 3H);  $^{13}\text{C}$  NMR (126 MHz, DMSO)  $\delta$  193.95, 171.31, 158.15, 141.30, 136.86, 136.25, 132.32, 130.21, 128.19, 127.87, 127.42, 127.22, 126.86, 125.74, 124.97, 123.76, 113.52, 79.74, 63.53, 59.76, 54.38 ; MS ( $\text{M}^+$ ), found 463; Anal. calcd for  $\text{C}_{30}\text{H}_{25}\text{NO}_4$ : C, 77.74; H, 5.44; N, 3.02; Found: C, 77.48; H, 5.69; N, 3.28.

4-benzoyl-1-(4-fluorophenyl)-3-hydroxy-3,5-diphenylpyrrolidin-2-one (**3j**)



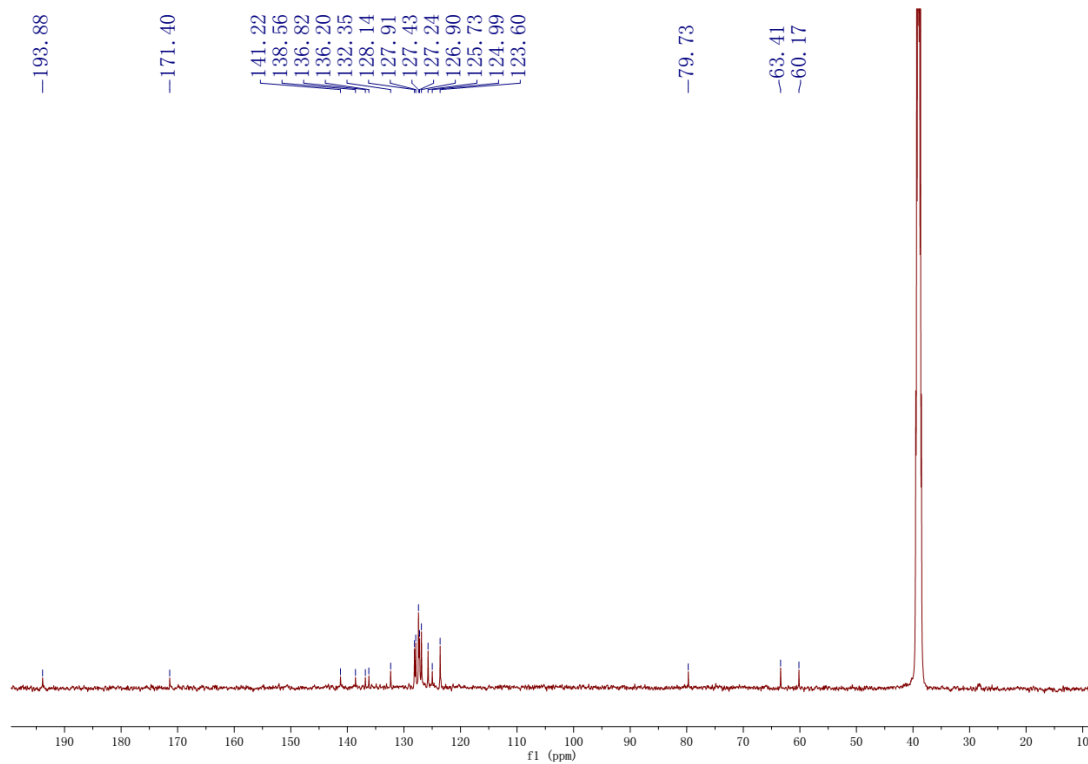
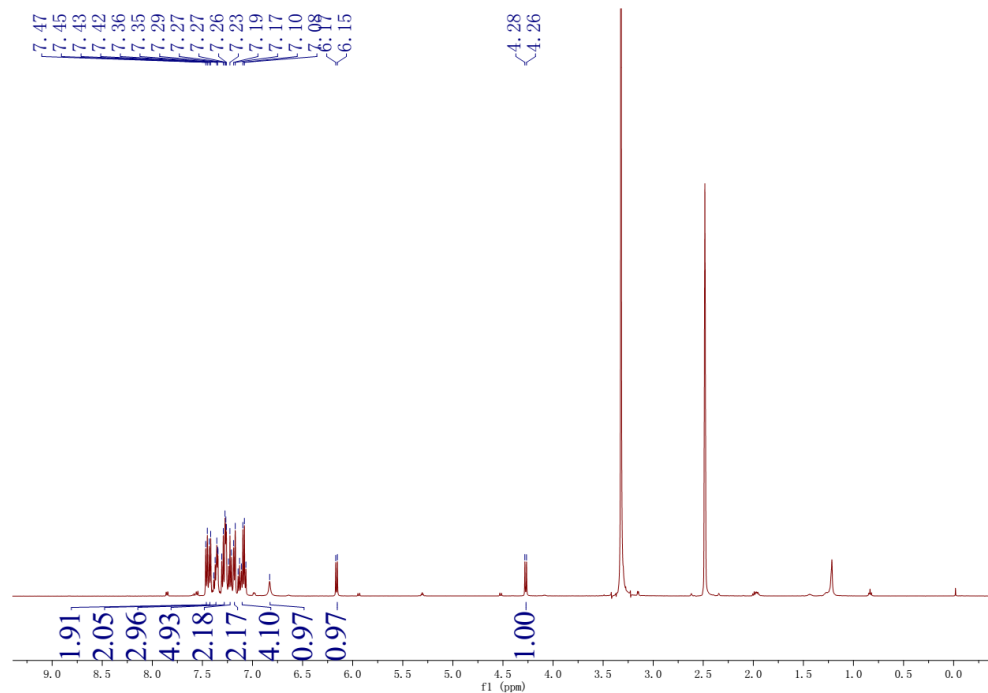
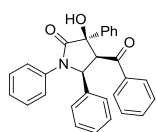
White solid; 65% yield.  $^1\text{H}$  NMR (500 MHz, DMSO)  $\delta$  7.45 (dd,  $J$  = 11.9, 8.1 Hz, 4H), 7.35 (d,  $J$  = 3.2 Hz, 2H), 7.30 (t,  $J$  = 7.5 Hz, 5H), 7.27 – 7.18 (m, 4H), 7.18 – 7.08 (m, 4H), 6.90 (s, 1H), 6.15 (d,  $J$  = 8.1 Hz, 1H), 4.29 (d,  $J$  = 8.1 Hz, 1H).  $^{13}\text{C}$  NMR (126 MHz, DMSO)  $\delta$  193.86, 171.42, 141.11, 138.03, 136.20, 133.13, 132.36, 128.17, 127.43, 127.23, 127.02, 126.91, 125.75, 114.80, 114.62, 79.66, 63.43, 60.44; MS ( $\text{M}^+$ ), found 451; Anal. calcd for  $\text{C}_{29}\text{H}_{22}\text{FNO}_3$ : C, 77.15; H, 4.91; N, 3.10; Found: C, 77.45; H, 4.76; N, 2.89.

4-benzoyl-3-hydroxy-1-(4-methoxyphenyl)-3,5-diphenylpyrrolidin-2-one (**3k**)

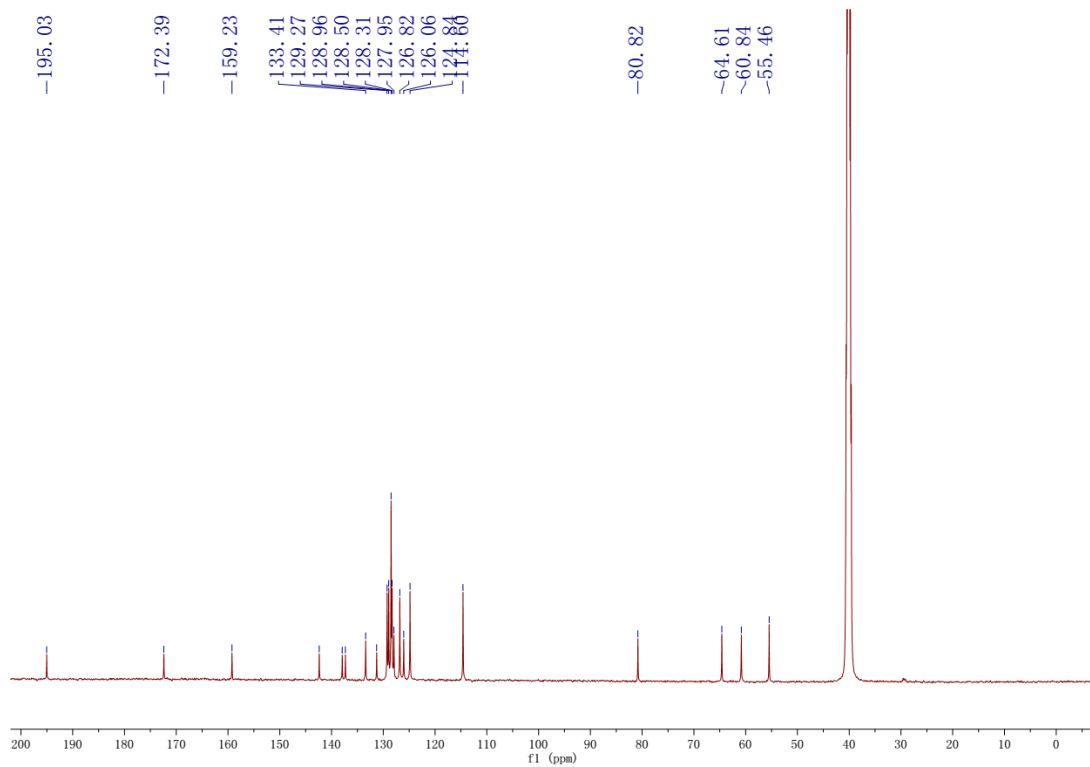
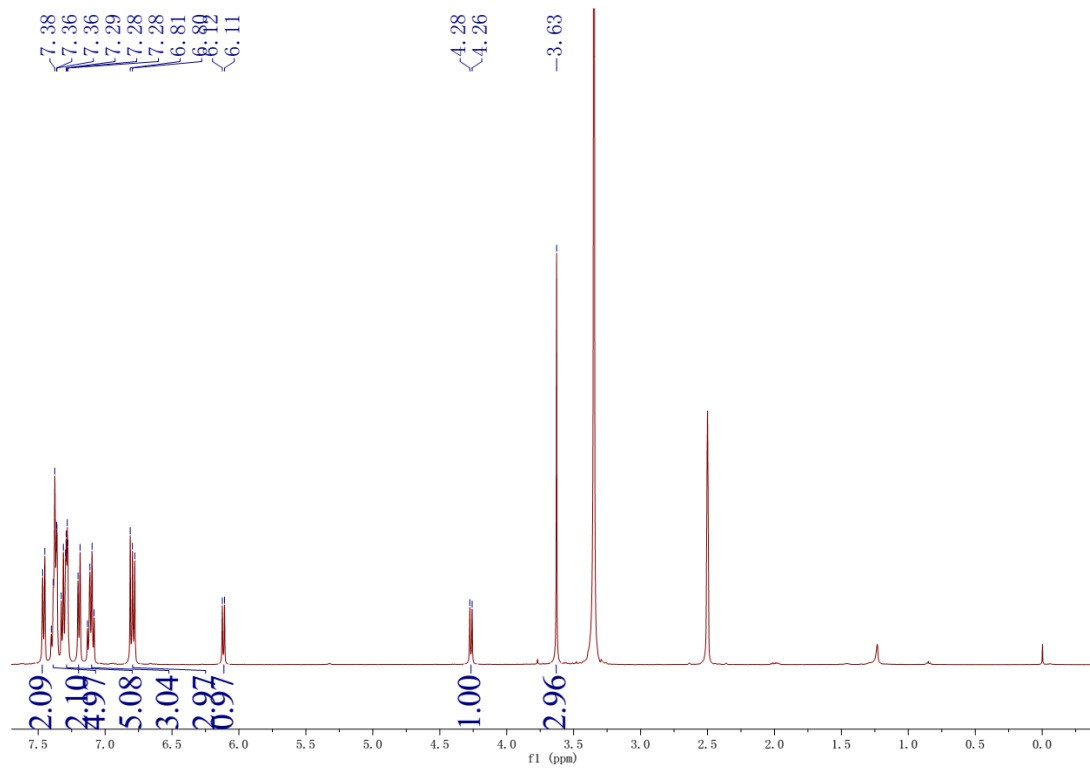
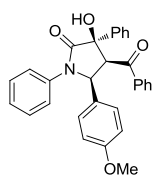


White solid; 78% yield.  $^1\text{H}$  NMR (500 MHz, DMSO)  $\delta$  7.43 (d,  $J$  = 7.6 Hz, 2H), 7.37 (dd,  $J$  = 15.5, 6.6 Hz, 5H), 7.31 – 7.21 (m, 5H), 7.21 – 7.13 (m, 3H), 7.09 (t,  $J$  = 7.7 Hz, 2H), 6.86 (d,  $J$  = 8.9 Hz, 2H), 6.81 (s, 1H), 6.09 (d,  $J$  = 8.2 Hz, 1H), 4.26 (d,  $J$  = 8.2 Hz, 1H), 3.69 (s, 3H).  $^{13}\text{C}$  NMR (126 MHz, DMSO)  $\delta$  195.05, 172.43, 157.45, 142.46, 139.71, 137.31, 133.42, 130.79, 129.22, 128.51, 128.32, 128.07, 127.94, 126.78, 126.38, 114.26, 80.73, 64.57, 61.64, 55.69; MS ( $\text{M}^+$ ), found 463; Anal. calcd for  $\text{C}_{30}\text{H}_{25}\text{NO}_4$ : C, 77.74; H, 5.44; N, 3.02; Found: C, 77.52; H, 5.59; N, 3.24.

### 3. NMR Spectra of Products

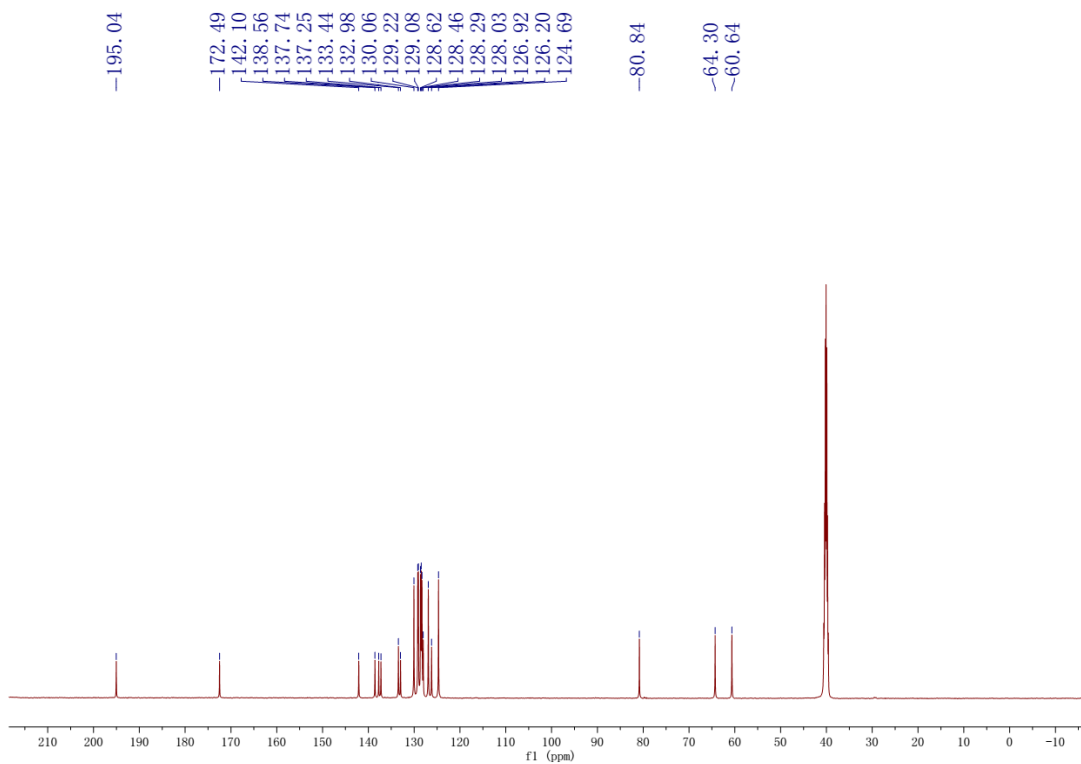
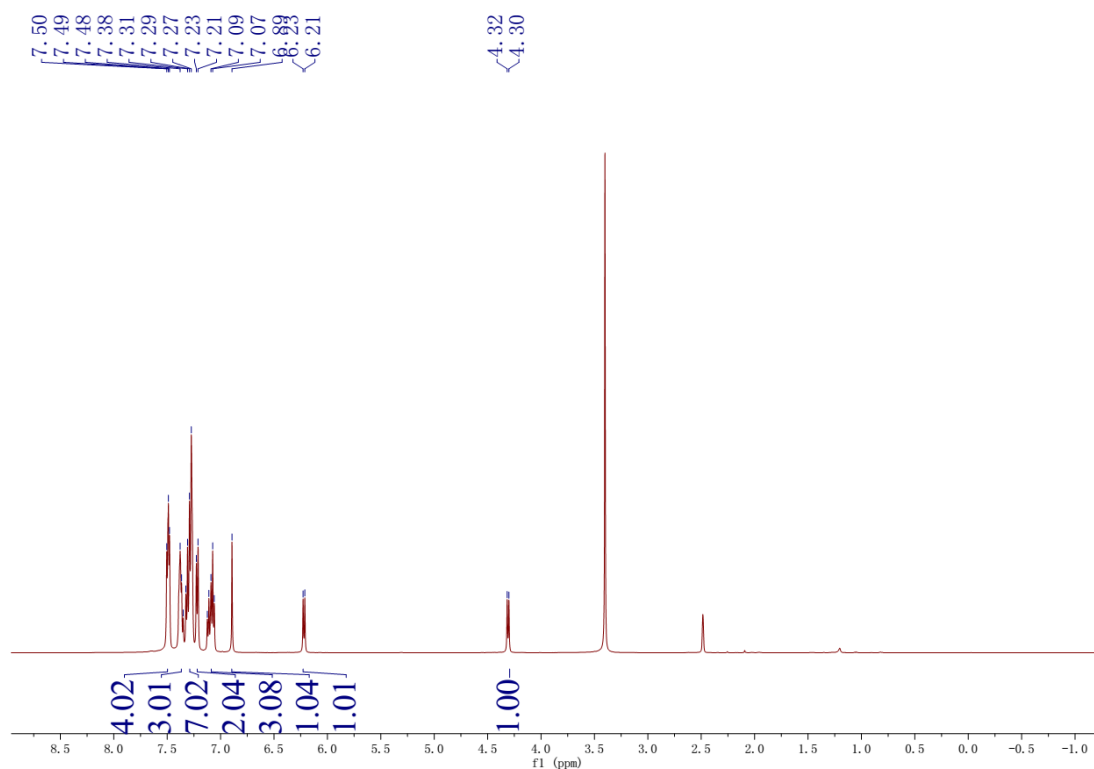
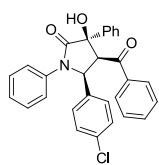


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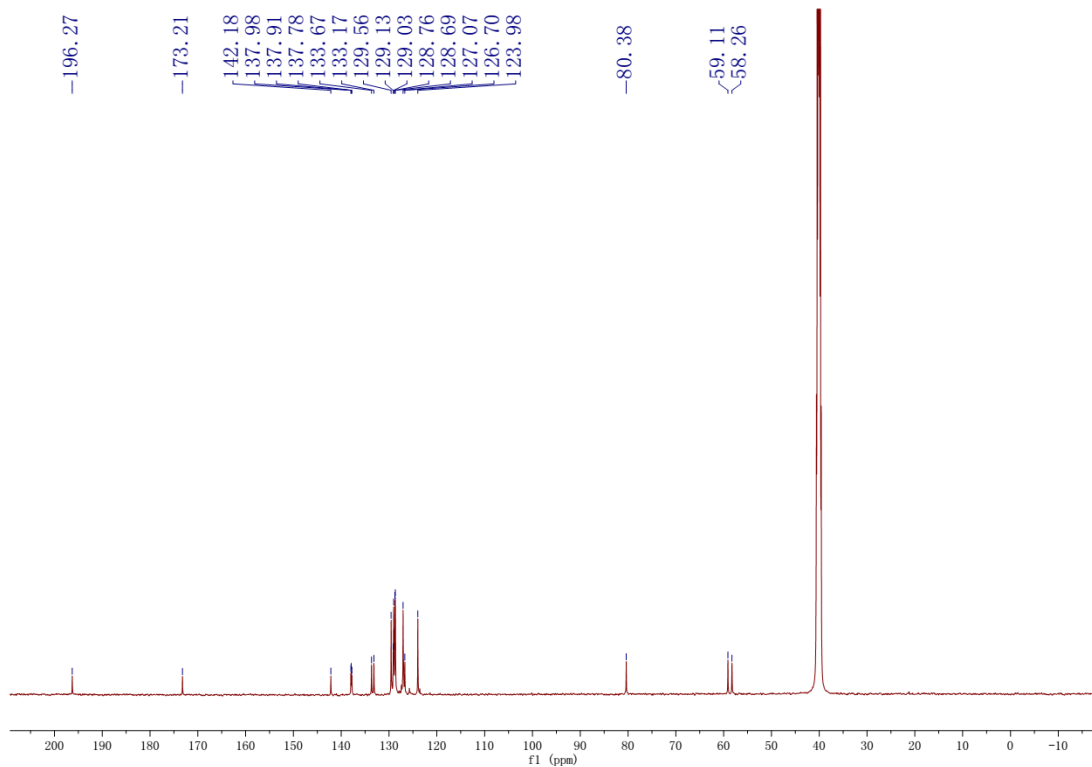
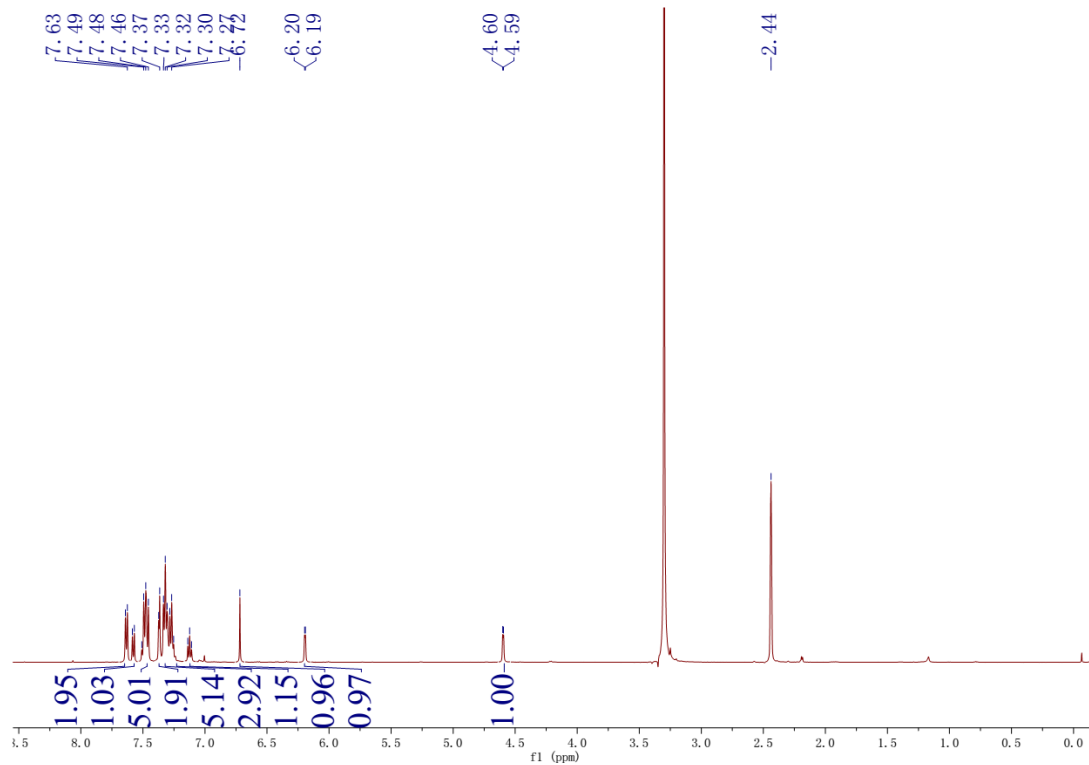
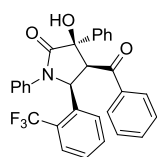




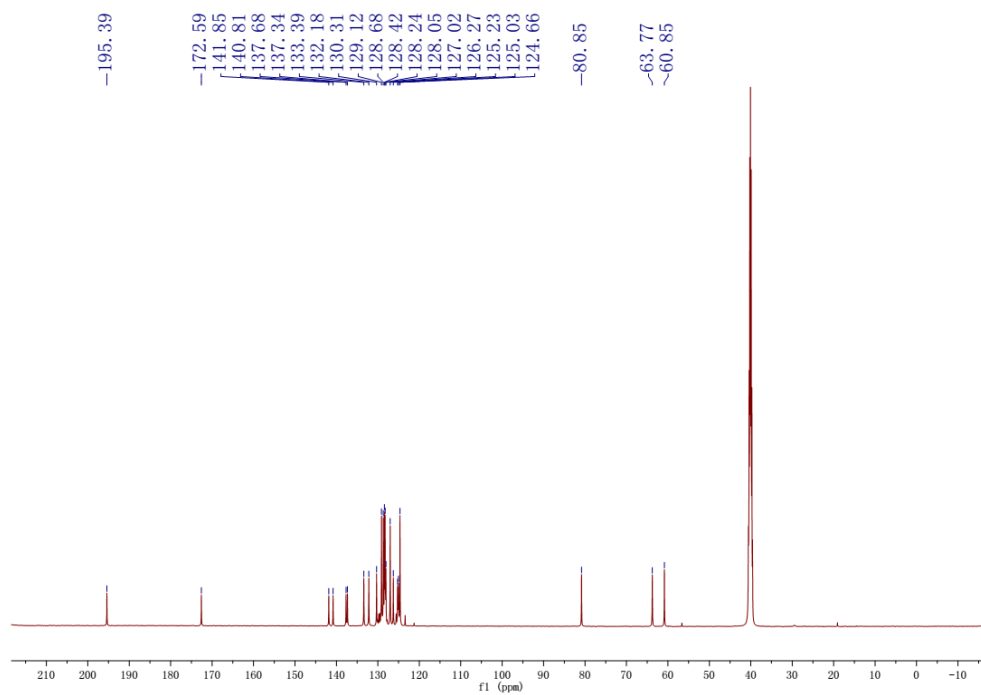
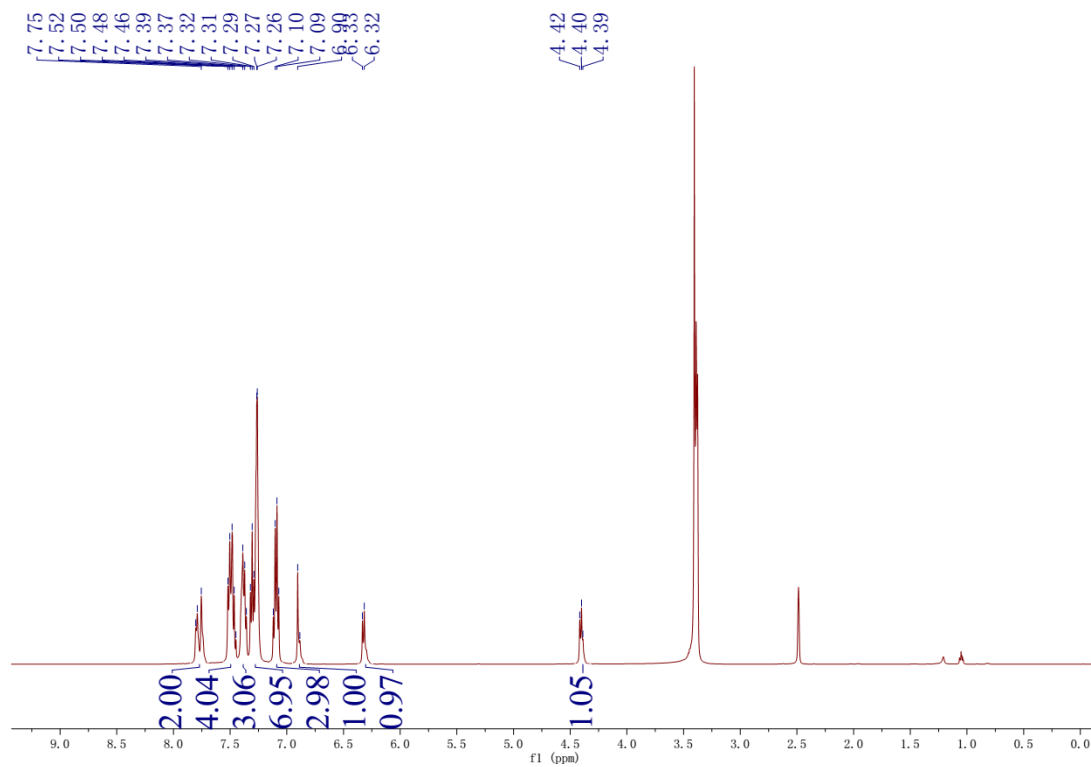
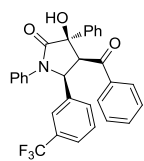
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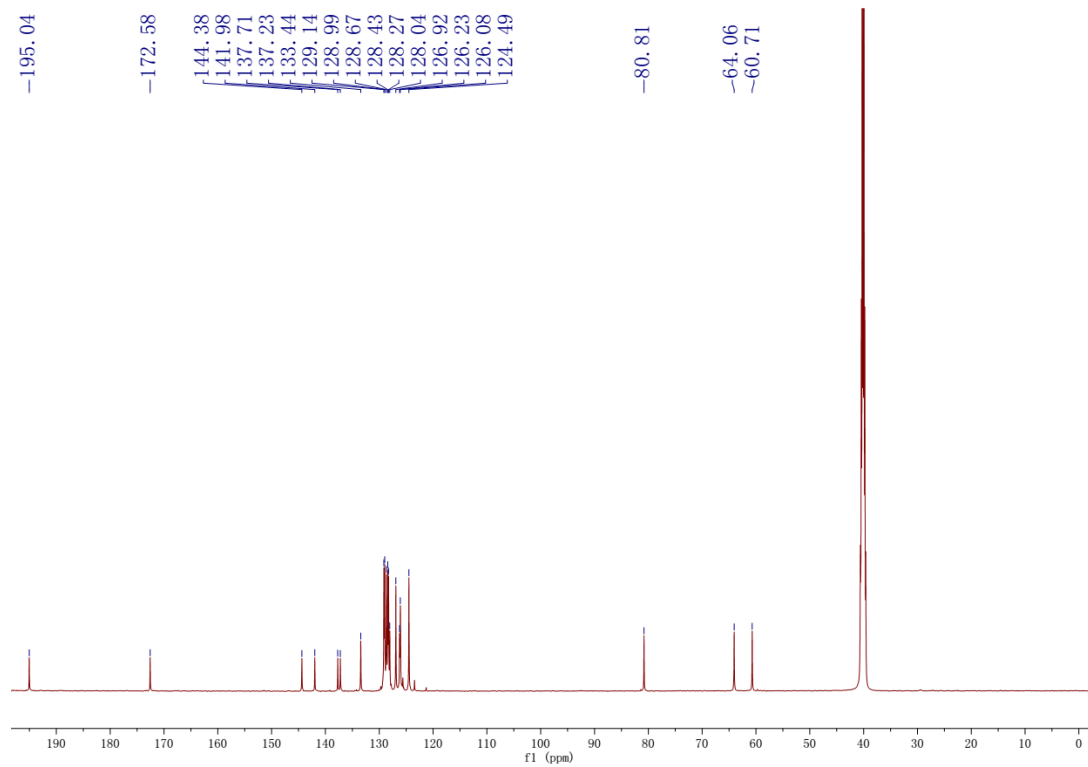
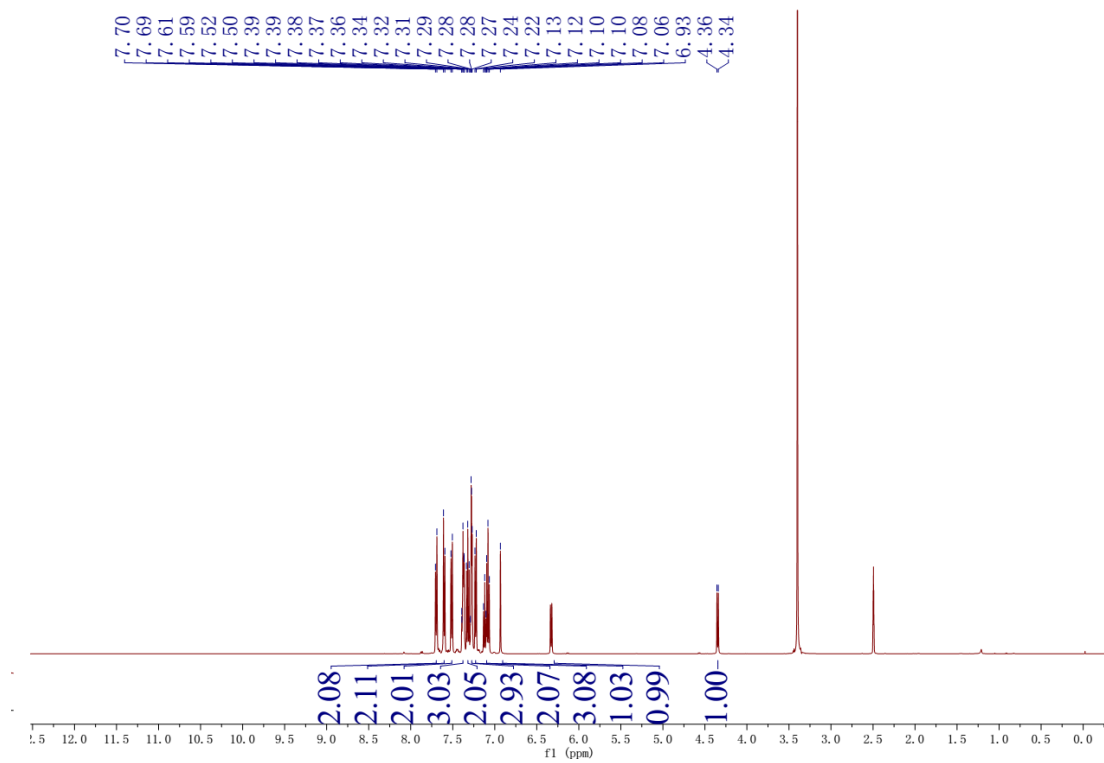
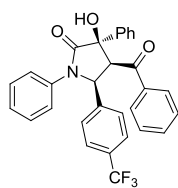
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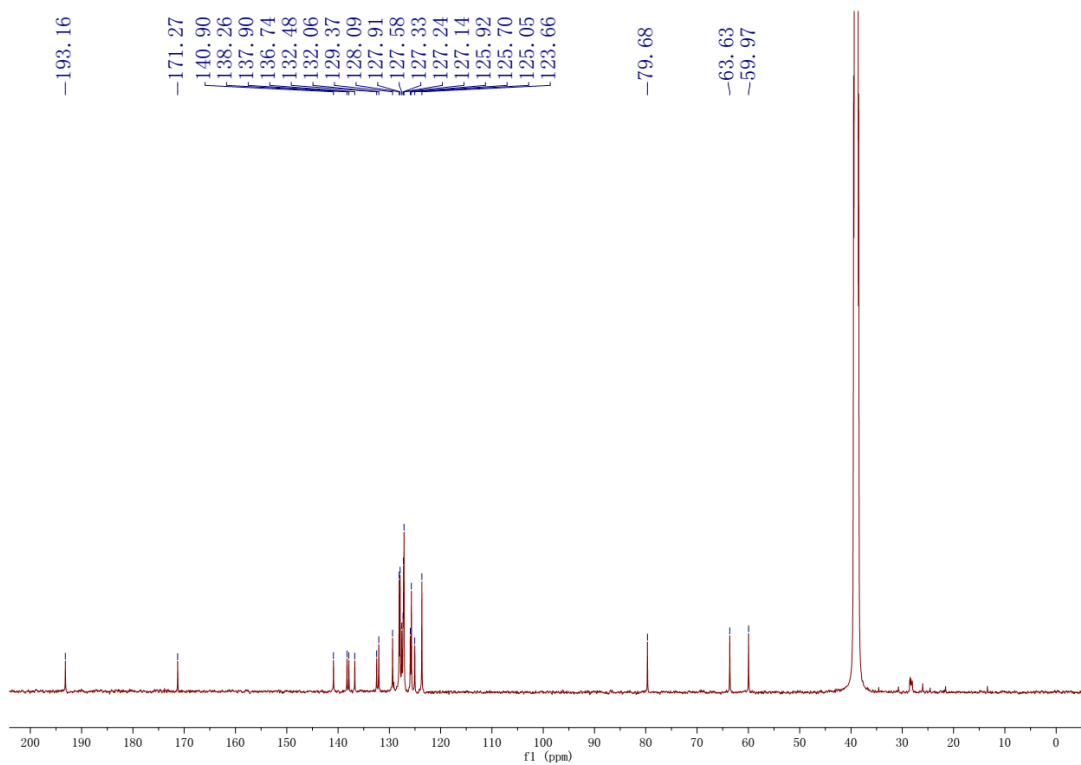
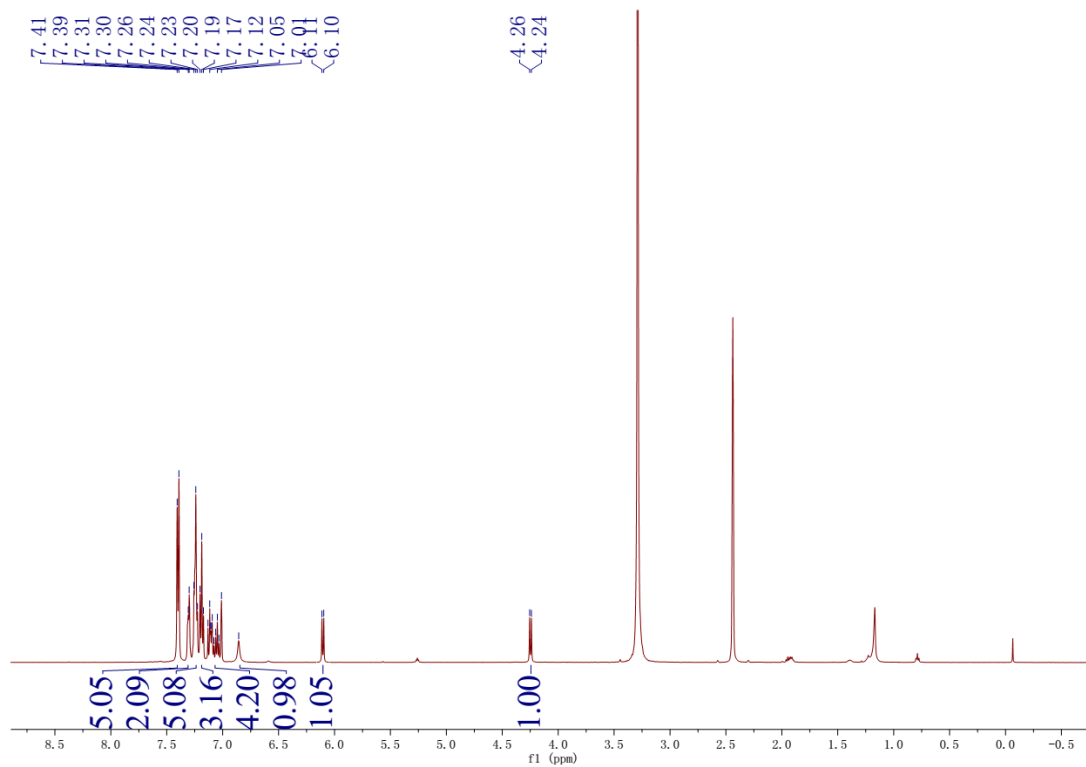
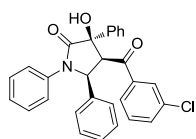
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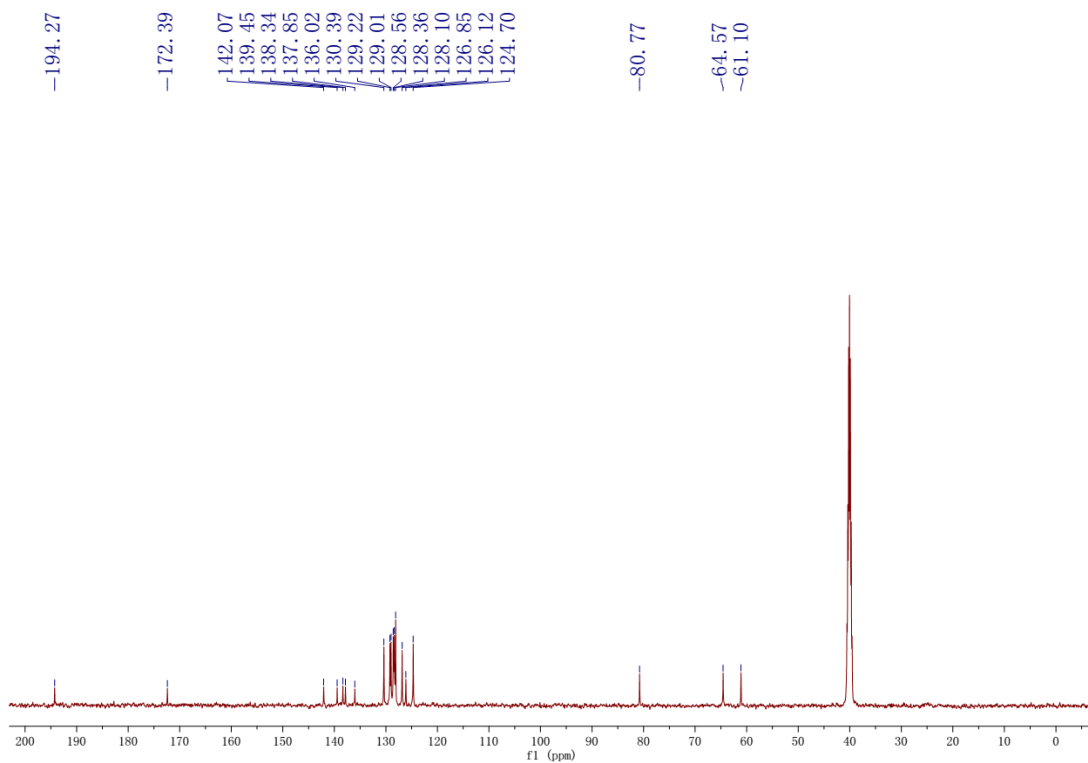
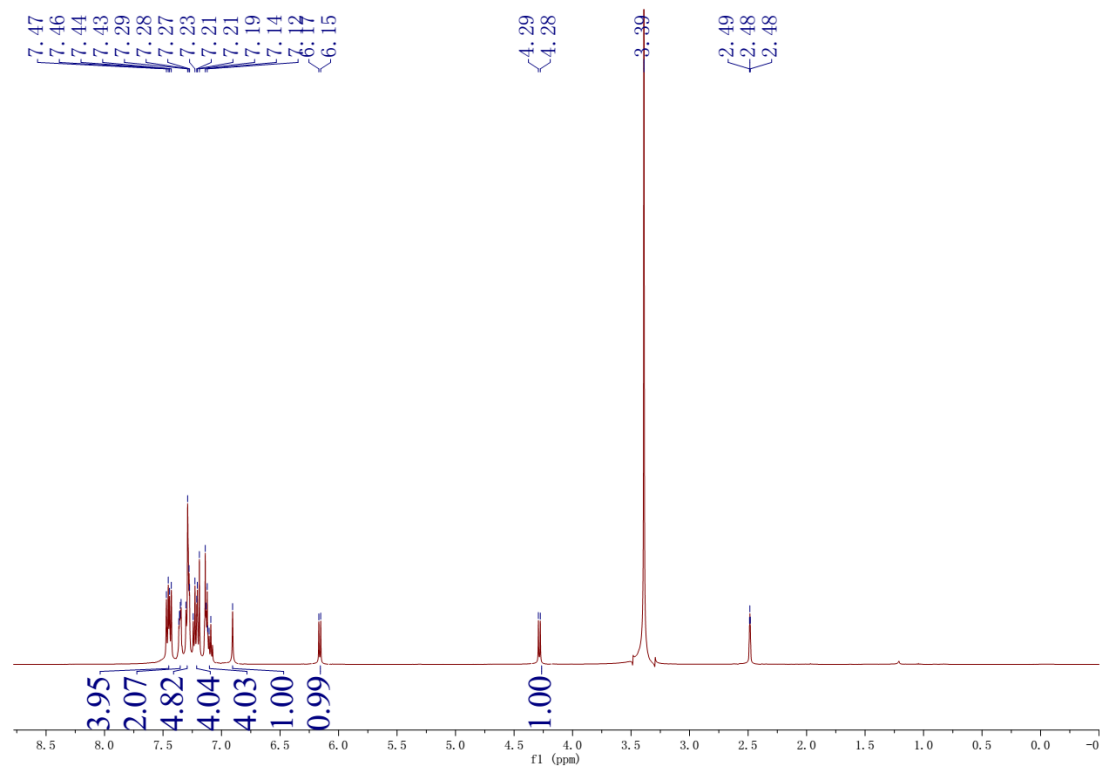
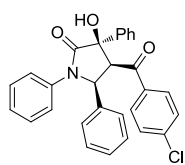
Electronic Supplementary Material (ESI) for New Journal of Chemistry.



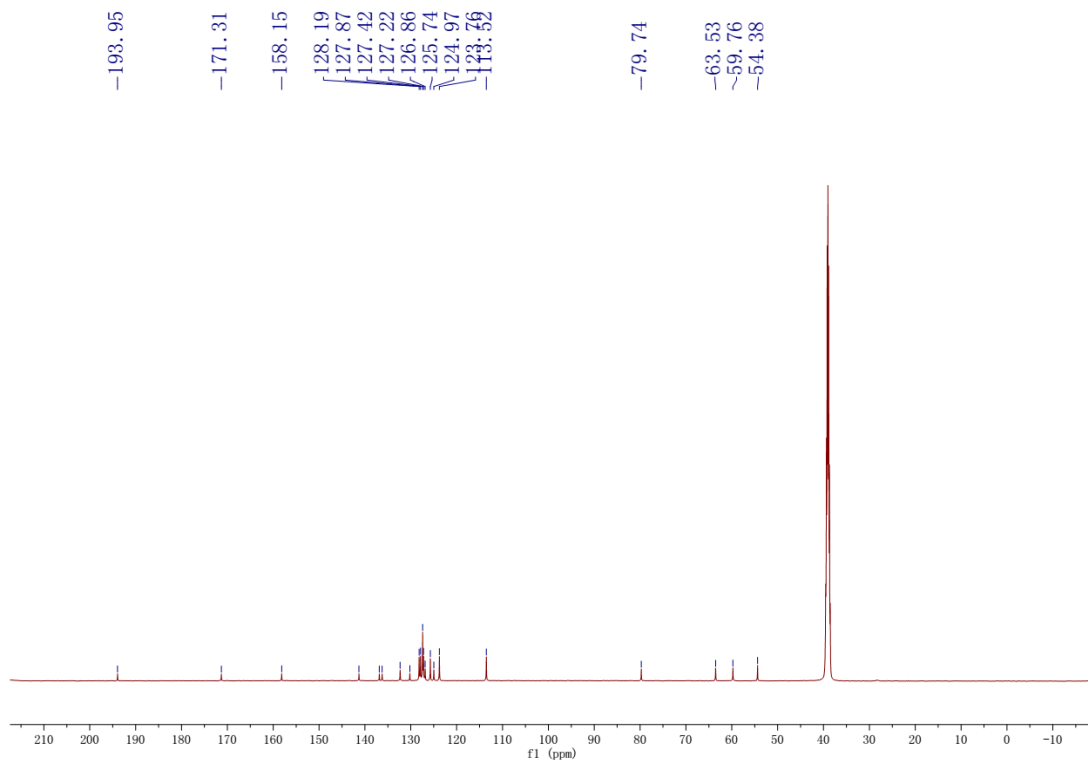
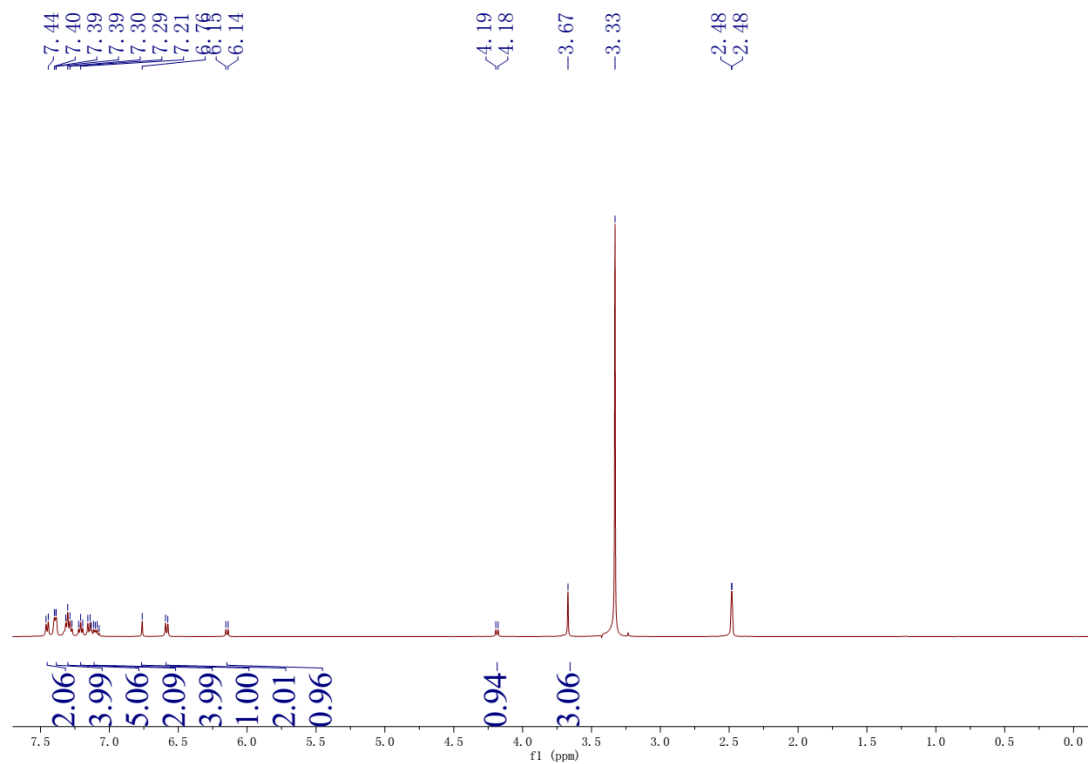
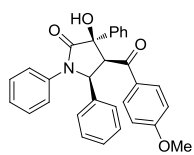
Electronic Supplementary Material (ESI) for New Journal of Chemistry.



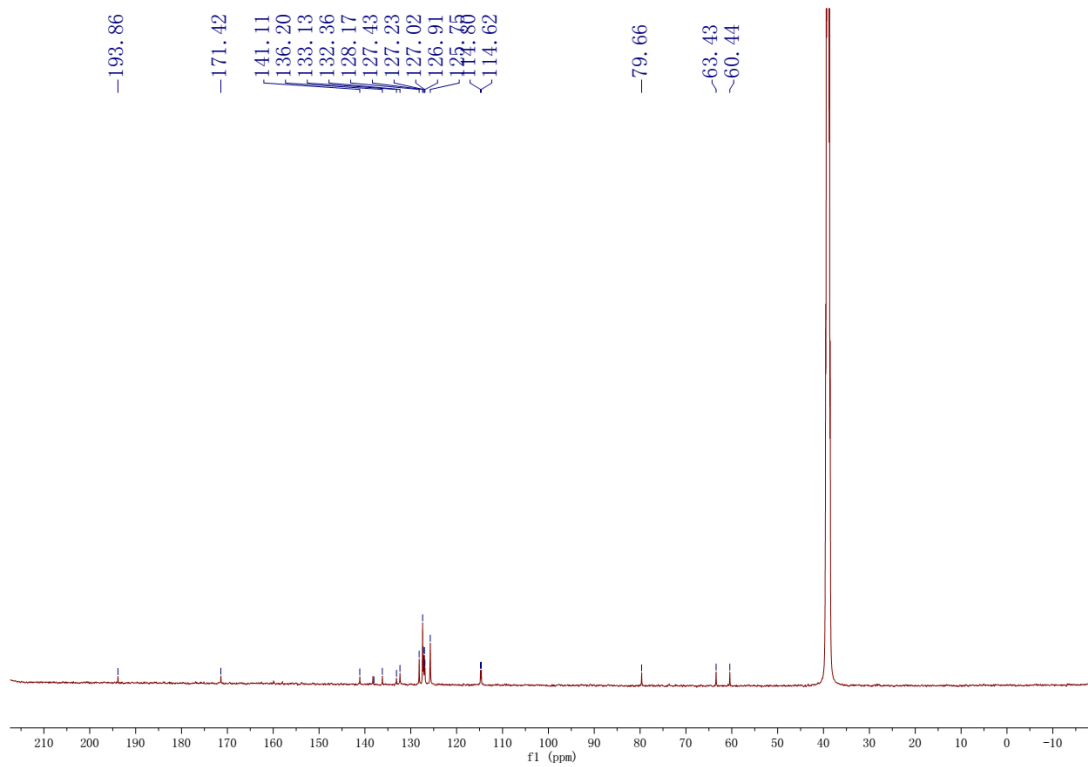
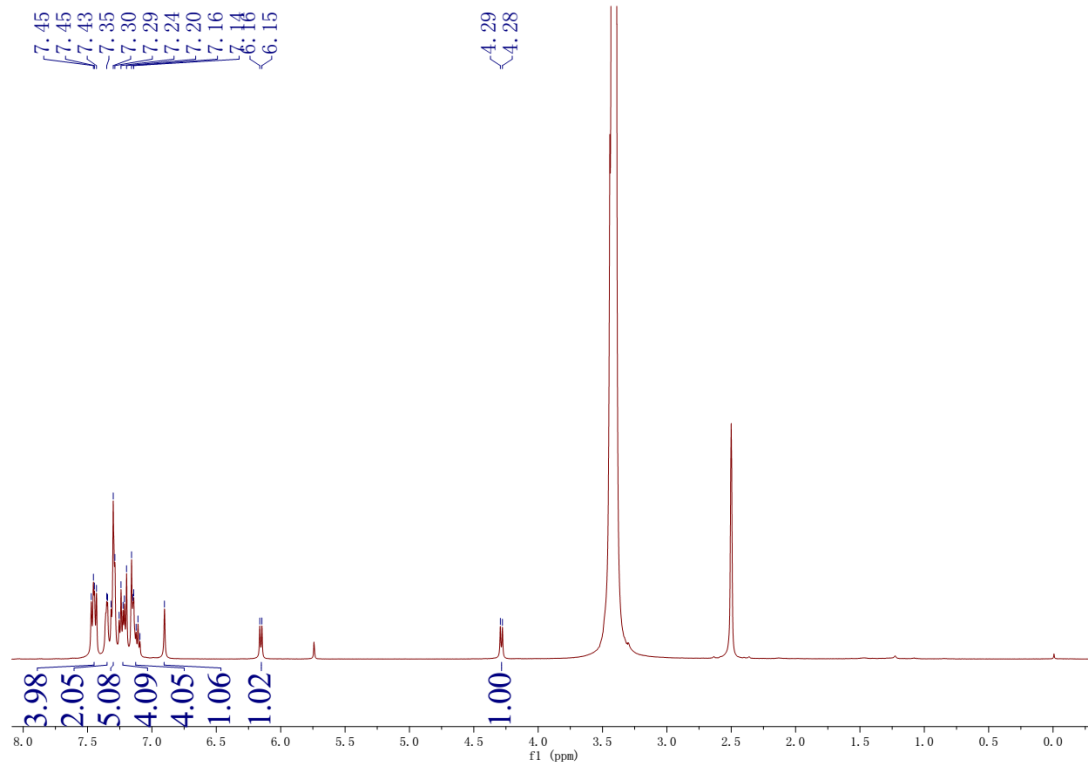
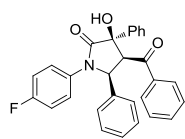
Electronic Supplementary Material (ESI) for New Journal of Chemistry.



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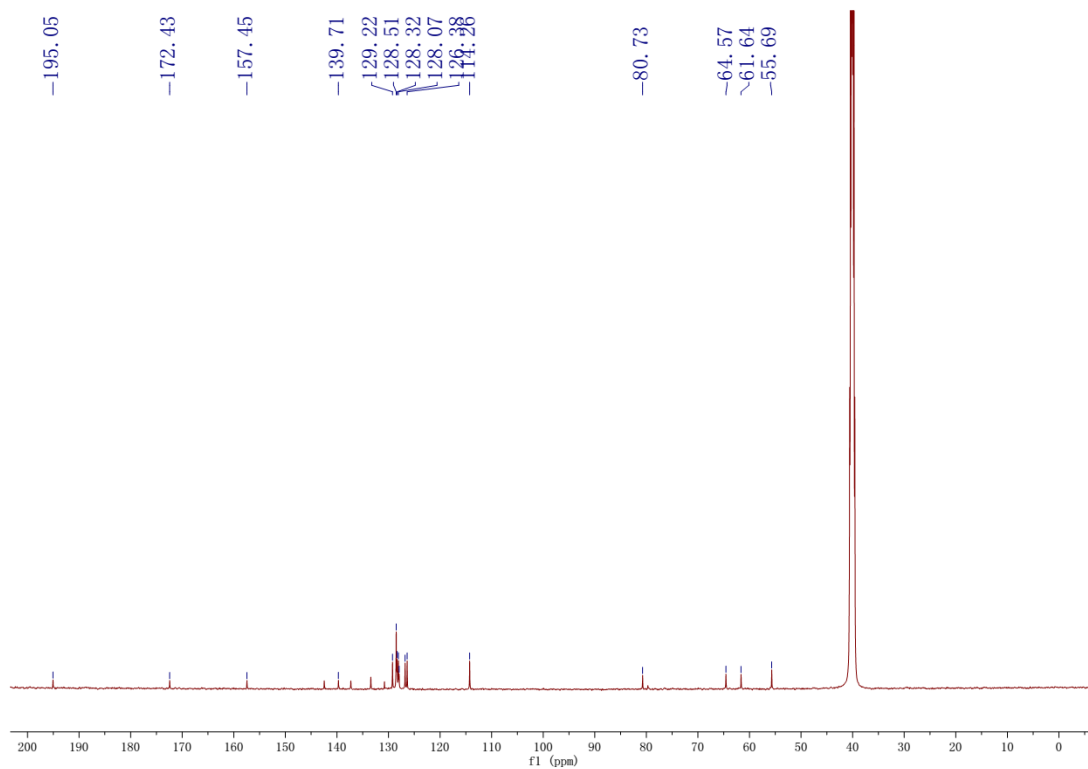
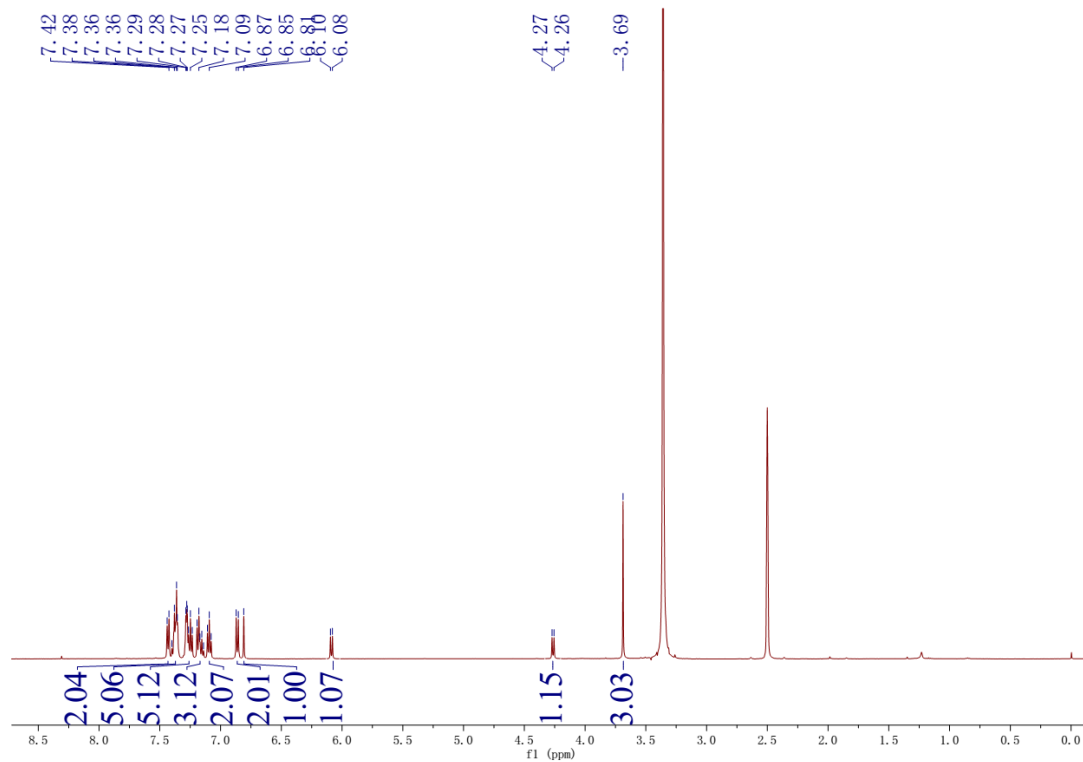
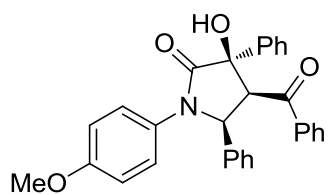


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## 4. NOESY $^1\text{H}$ NMR Spectra of 3f

