Electronic Supplementary Material (ESI) for RSC Advances.

Copper-catalyzed one-pot oxidative amidation of alcohol to amide *via* C-H activation

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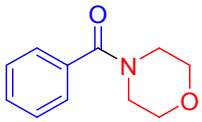
1. General details

Reaction solvents were obtained commercially, and used without further purification. Commercial reagents were used as received. Reaction were monitored by thin-layer chromatography (TLC) on 0.25mm precoated Merck Silica Gel 60 F254, visualizing with ultraviolet light. $^{1}H/^{13}C$ NMR spectra were recorded on 400'54 ascend purchased from Bruker Biospin AG, operating at 400/100 MHz, respectively. Chemical shifts (δ) are reported in parts per million (ppm) downfield from tetramethylsilane (TMS), which was used as internal standard. High-resolution mass spectra (HRMS) were obtained from Agilent 6520 LC-MS. Flash column chromatography was performed on Merck Silica Gel 60 (200-300mesh) using petroleum ether and ethyl acetate.

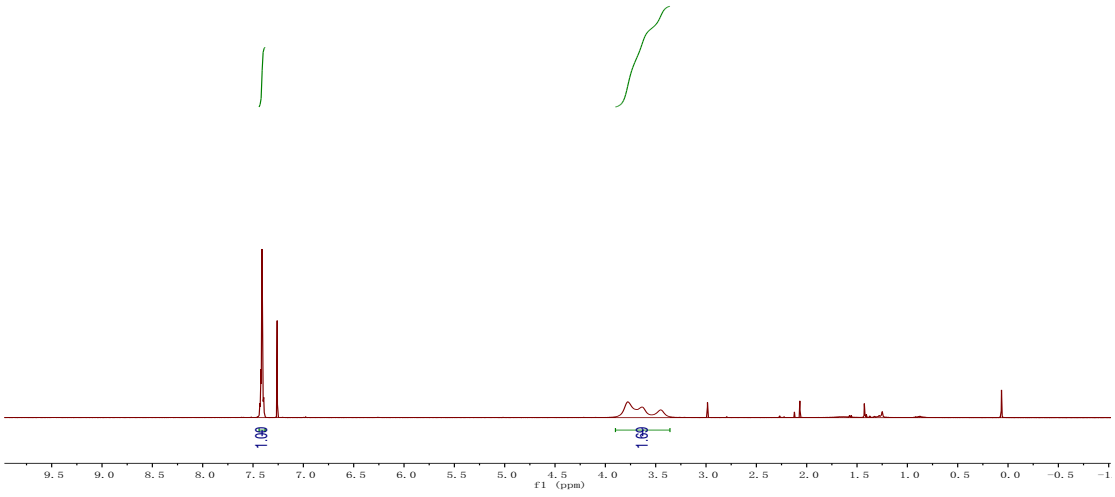
2. Experimental procedure

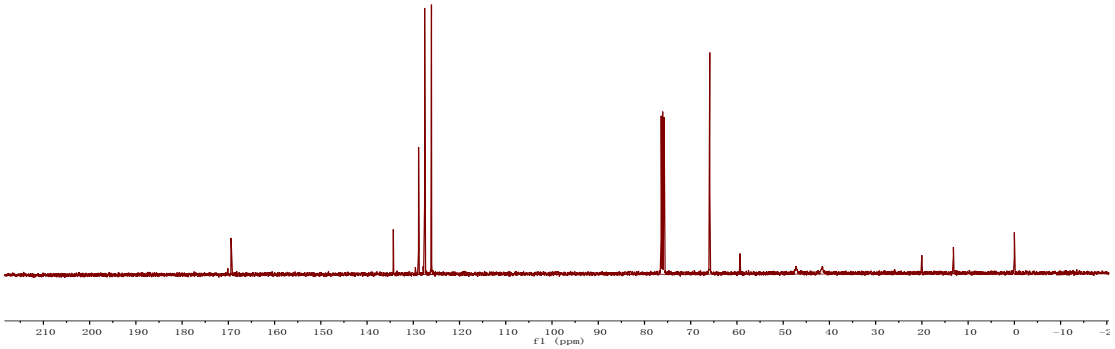
General procedure for synthesis of compound 3: 1.5mmol of amine (**1**) and 1.65mmol (1.1 equiv.) of NCS were dissolved in 4mL acetonitrile, stirring at room temperature for 3 h. Then alcohol (**2**) (1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (20 mol%) and TBHP (70 wt% in water, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. Then the reaction mixture was extracted with ethyl acetate, washed with NH_4Cl solution. The organic layer was dried over anhydrous sodium sulfate and solvent was removed under vacuum. And the crude product was purified by flash chromatography on silica gel by gradient elution with ethyl acetate in petroleum ether to obtain the amide product **3**.

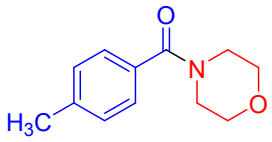
3. NMR Spectra



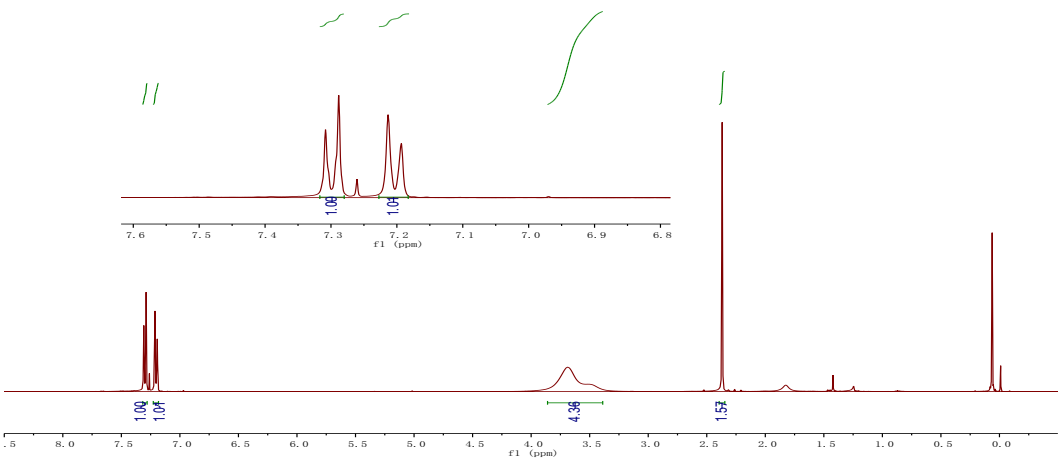
Benzoyl morpholine (**3a**). Morpholine (130mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification as white solid (263mg, 92% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44–7.38 (m, 5H), 3.90–3.36 (m, 8H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 169.4, 134.3, 128.9, 127.5, 126.1, 65.9, 59.4, 20.0, 13.2; HRMS (ESI) m/z calcd for $C_{11}H_{13}NO_2$ $[M+H]^+$ 192.1019, found 192.1040.

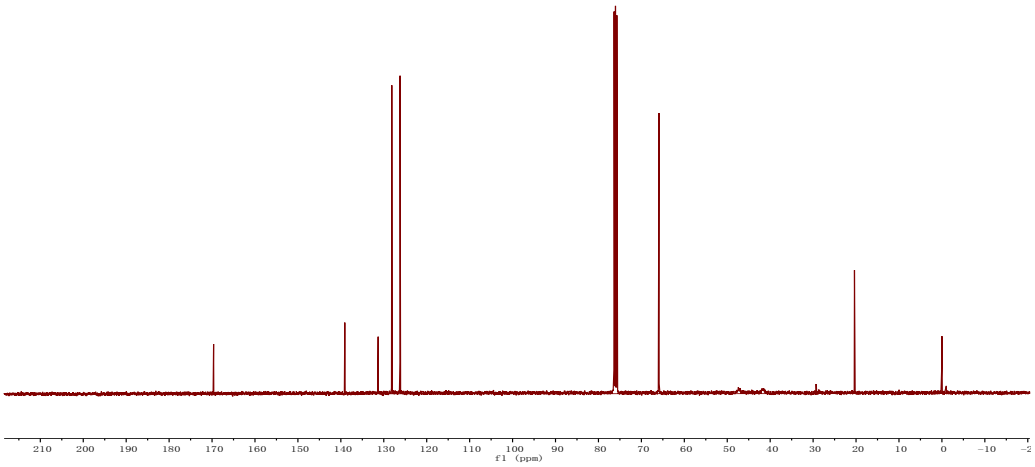


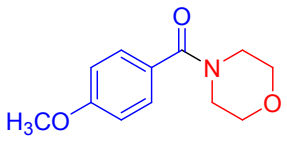




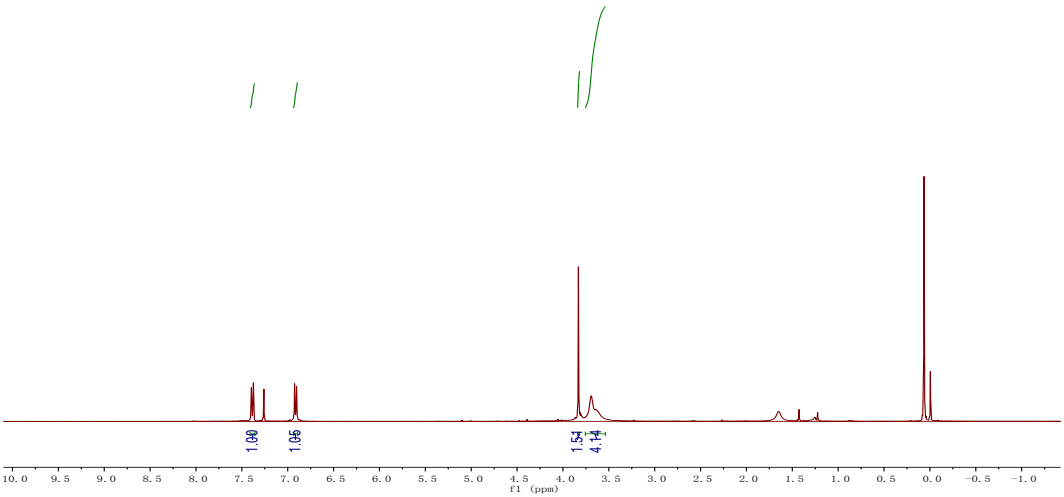
N-(4-Methybenzoyl)morpholine (**3b**). Morpholine (130mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then 4-methylbenzyl alcohol (220mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification as light yellow oil (252mg, 82% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.1 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 3.69 (m, 8H), 2.37 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.6, 139.1, 131.3, 128.1, 126.2, 65.9, 29.3, 20.4; HRMS (ESI) m/z calcd for $C_{12}H_{15}NO_2$ $[M+H]^+$ 206.1136, found 206.1147.

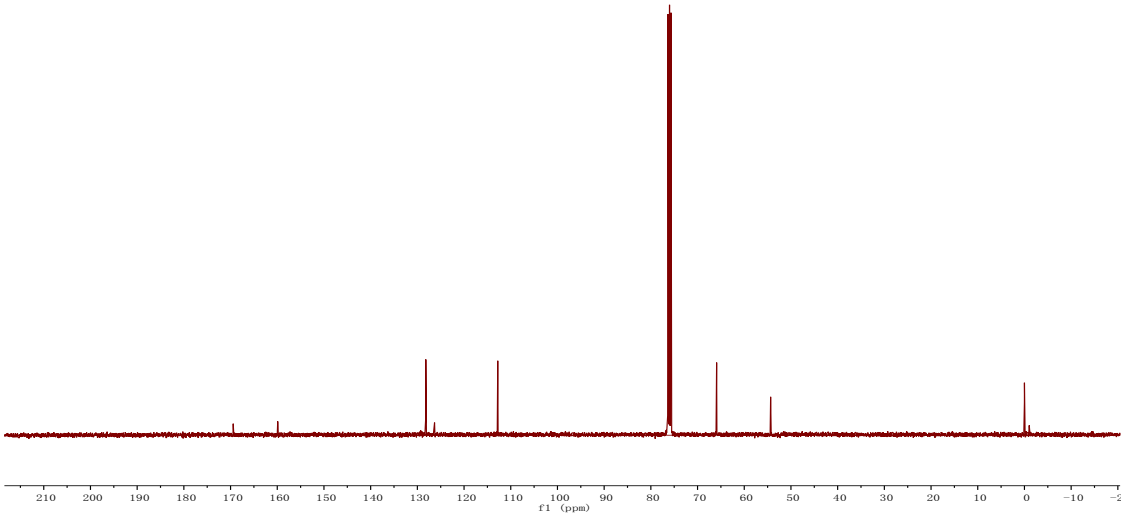


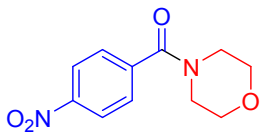




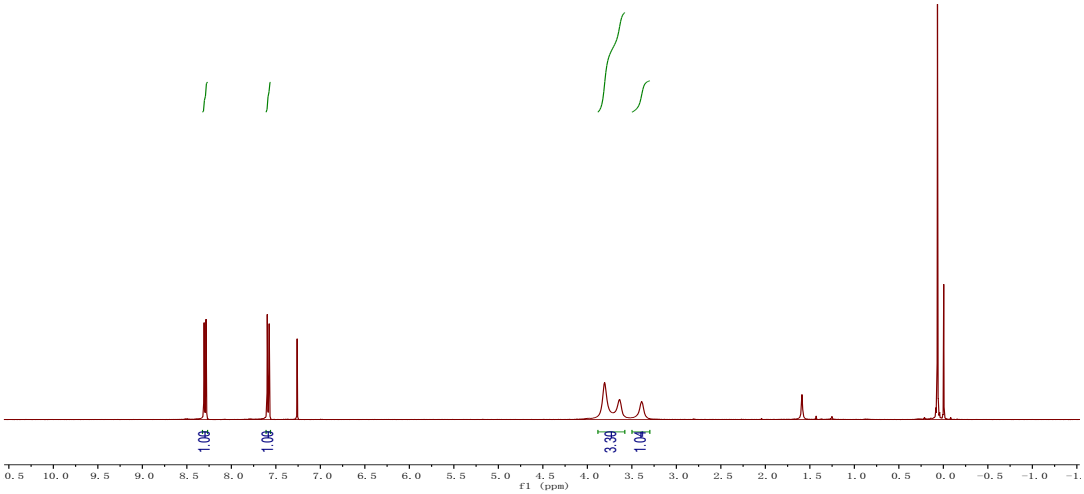
N-(4-Methoxybenzoyl)morpholine (**3c**). Morpholine (130mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then 4-methoxybenzyl alcohol (250mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 20h. And the product was obtained after purification as yellow oil (262mg, 79% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41–7.36 (m, 2H), 6.94–6.89 (m, 2H), 3.84 (s, 3H), 3.76–3.54 (m, 8H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.4, 159.9, 128.2, 126.3, 112.8, 65.9, 54.4; HRMS (ESI) m/z calcd for $C_{12}H_{15}NO_3$ $[M+H]^+$ 222.1085, found 222.1094.

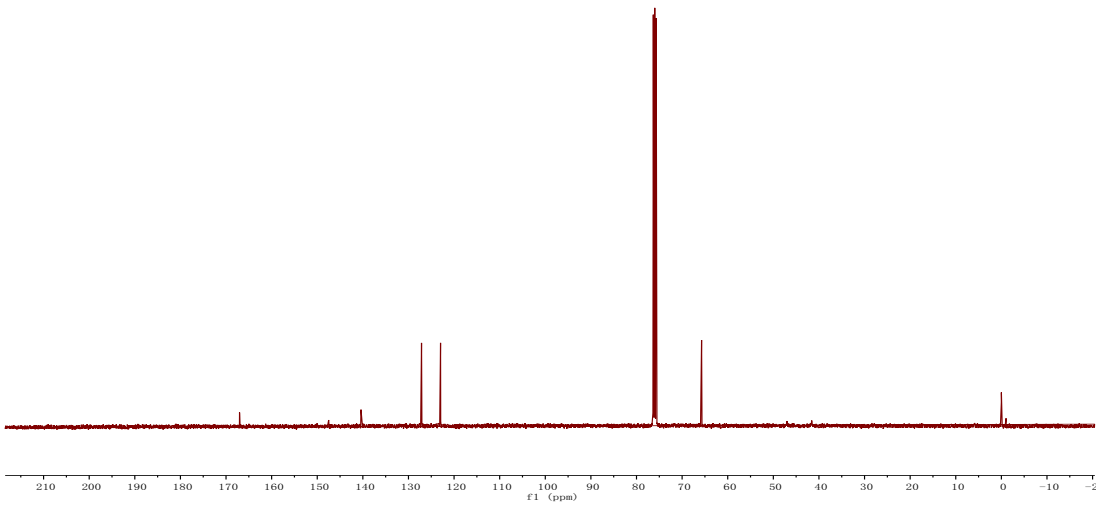


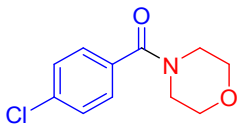




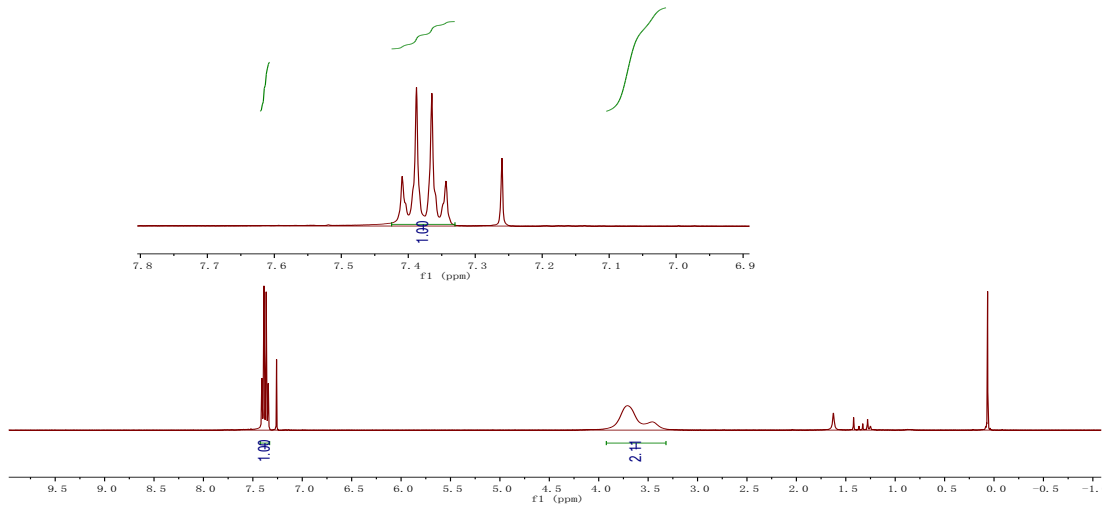
N-(4-Nitrobenzoyl)morpholine (**3d**). Morpholine (130mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then 4-nitrobenzyl alcohol (275mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification as light yellow solid (297mg, 84% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32–8.27 (m, 2H), 7.61–7.56 (m, 2H), 3.72 (d, *J* = 67.2 Hz, 6H), 3.39 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.0, 147.5, 140.4, 127.1, 123.0, 65.7; HRMS (ESI) m/z calcd for $C_{11}H_{11}N_2O_4$ $[M+H]^+$ 237.0831, found 237.0857.

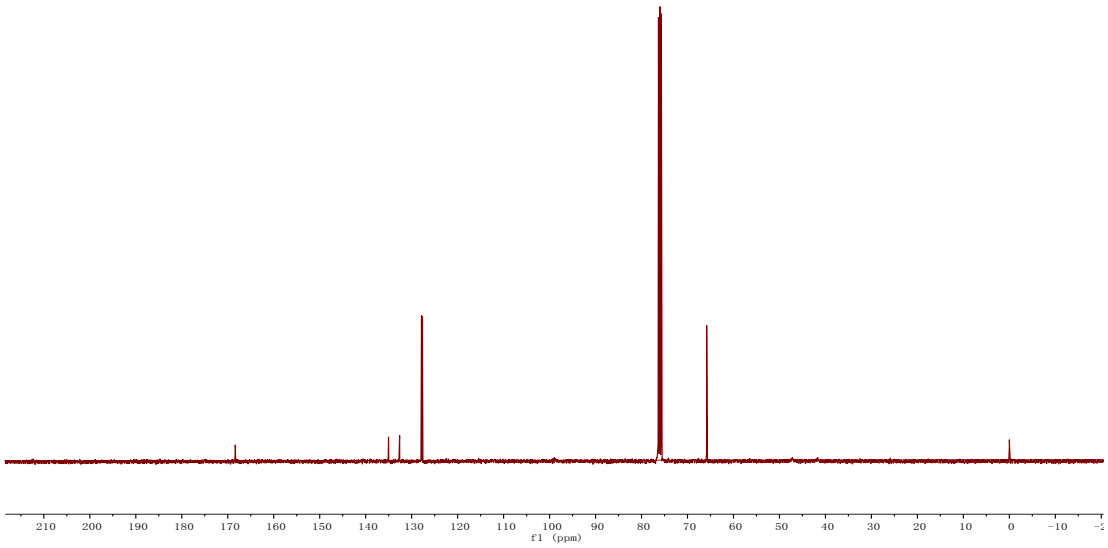


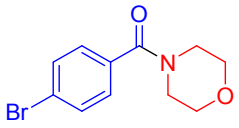




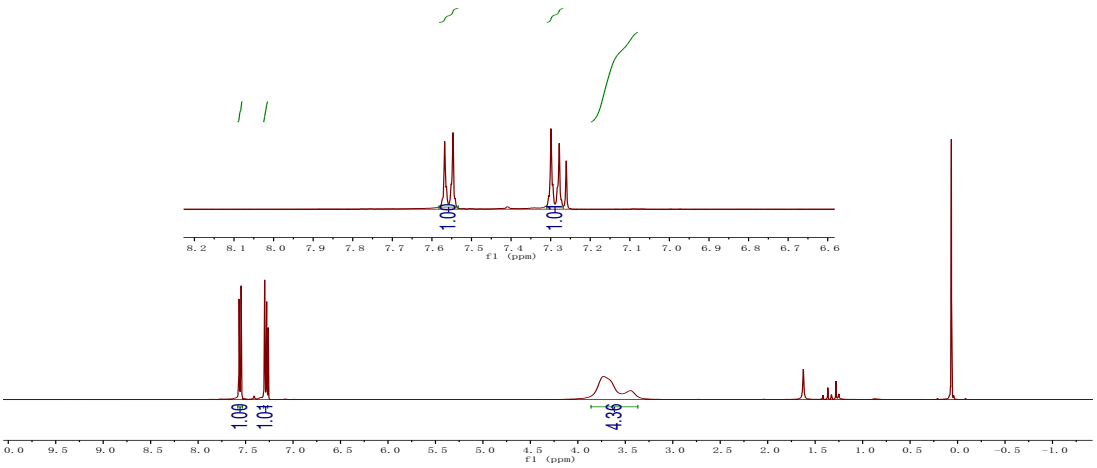
N-(4-Chlorobenzoyl)morpholine (**3e**). Morpholine (130mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then 4-chlorobenzyl alcohol (256mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 18h. And the product was obtained after purification as white solid (274mg, 81% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (q, *J* = 8.5 Hz, 4H), 3.92–3.32 (m, 8H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.4, 135.0, 132.6, 127.9, 127.6, 99.9, 65.8; HRMS (ESI) m/z calcd for $C_{11}H_{12}ClNO_2$ $[M+H]^+$ 226.6720, found 226.6724.

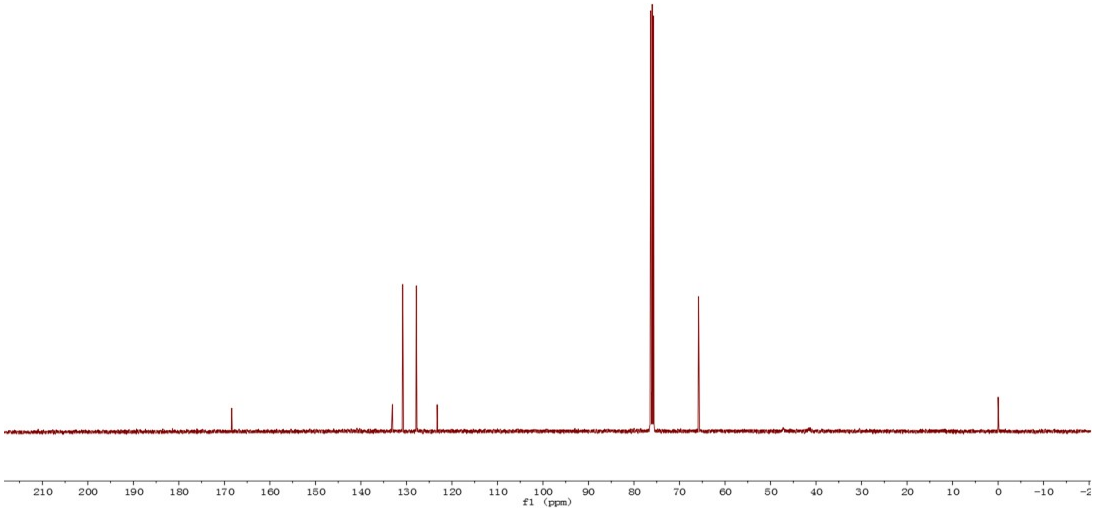


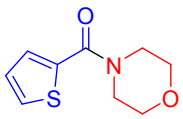




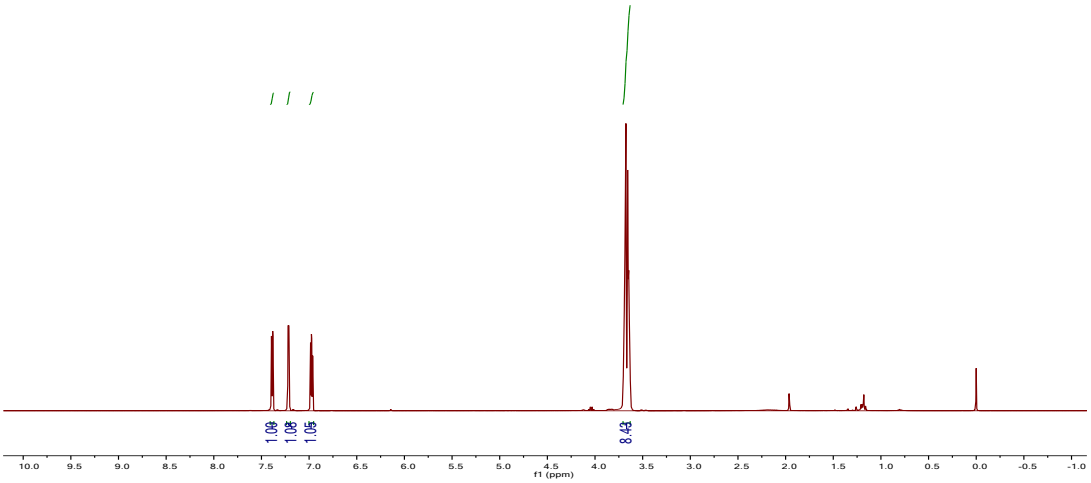
N-(4-Bromobenzoyl)morpholine (**3f**). Morpholine (130mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then 4-bromobenzyl alcohol (336mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 20h. And the product was obtained after purification as light yellow solid (340mg, 84% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58–7.54 (m, 2H), 7.31–7.27 (m, 2H), 3.86–3.31 (m, 8H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.4, 133.1, 130.8, 127.8, 123.2, 65.8; HRMS (ESI) m/z calcd for $C_{11}H_{12}BrNO_2$ $[M+H]^+$ 271.1260, found 271.1284.

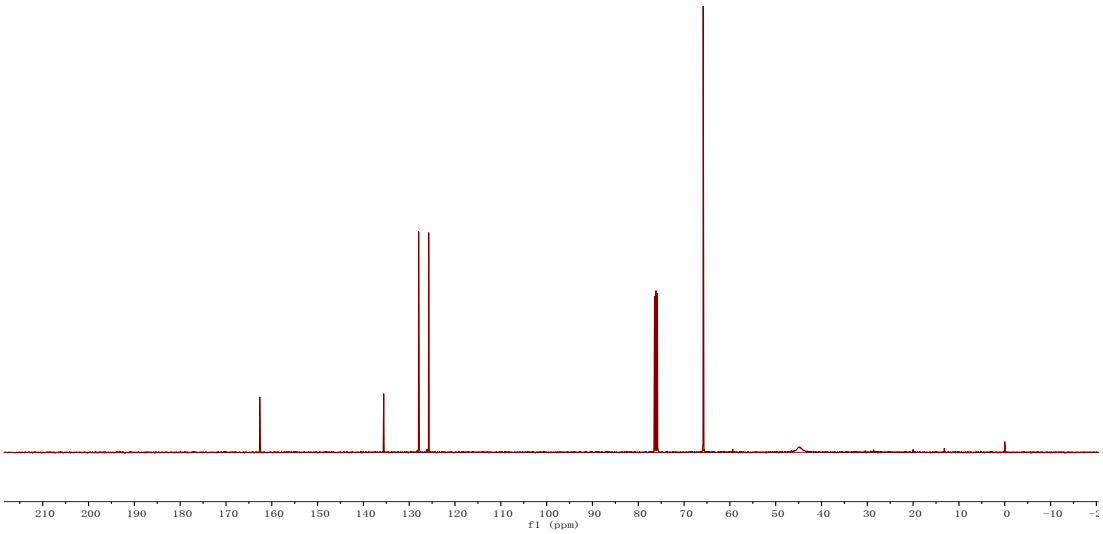


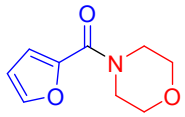




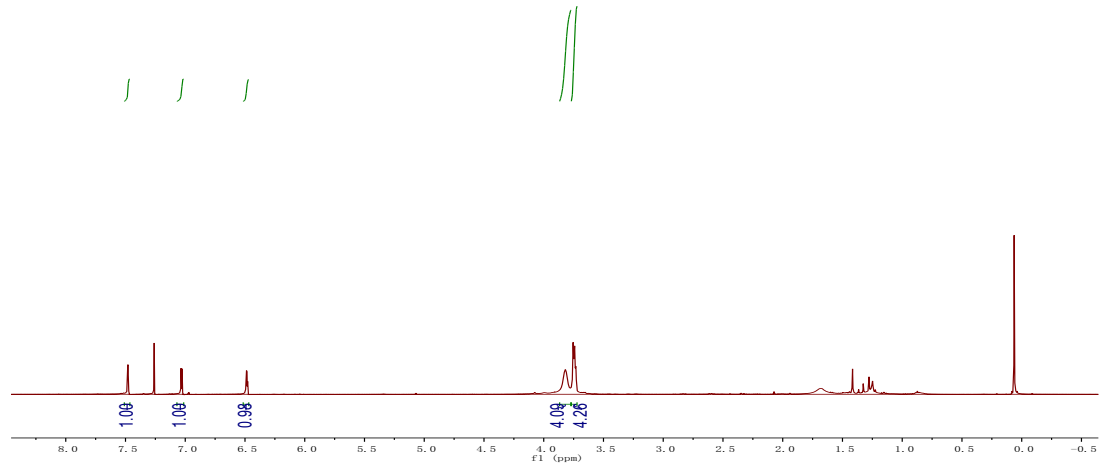
4-Morpholinyl-2-thienylmethanone (**3g**). Morpholine (130mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then 2-thiophenemethanol (205mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification as light yellow oil (269mg, 91% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (dd, *J* = 5.0, 1.0 Hz, 1H), 7.22 (dd, *J* = 3.6, 1.0 Hz, 1H), 6.97 (dd, *J* = 5.0, 3.7 Hz, 1H), 3.71–3.63 (m, 8H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.63, 135.57, 127.9, 127.8, 125.7, 65.8, 59.4, 20.0, 13.2; HRMS (ESI) m/z calcd for $C_9H_{11}NO_2S$ $[M+H]^+$ 198.0544, found 198.0568.

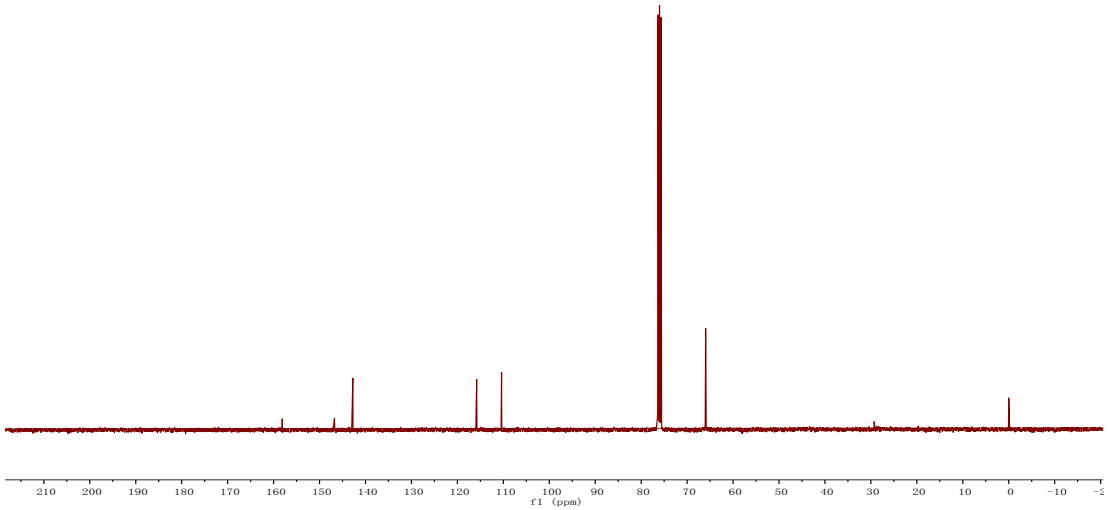


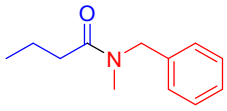




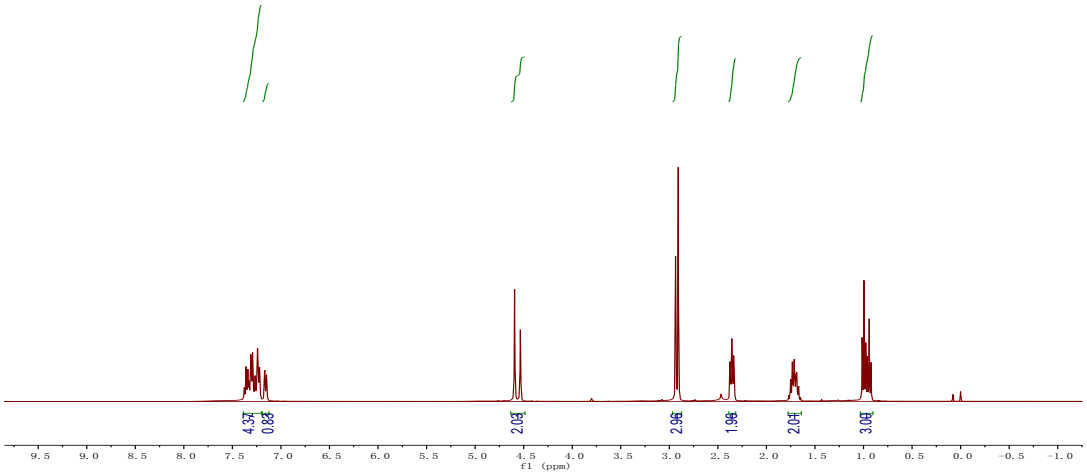
2-Furanyl-4-morpholinylmethanone (**3h**). Morpholine (130mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then 2-furanmethanol (176mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 20h. And the product was obtained after purification as yellow oil (238mg, 93% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (dd, *J* = 1.7, 0.8 Hz, 1H), 7.03 (dd, *J* = 3.5, 0.7 Hz, 1H), 6.49 (dd, *J* = 3.5, 1.8 Hz, 1H), 3.82 (s, 4H), 3.77–3.72 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.1, 146.8, 142.8, 115.8, 110.4, 65.9, 29.3; HRMS (ESI) m/z calcd for $C_9H_{11}NO_3$ $[M+H]^+$ 182.0772, found 182.0783.

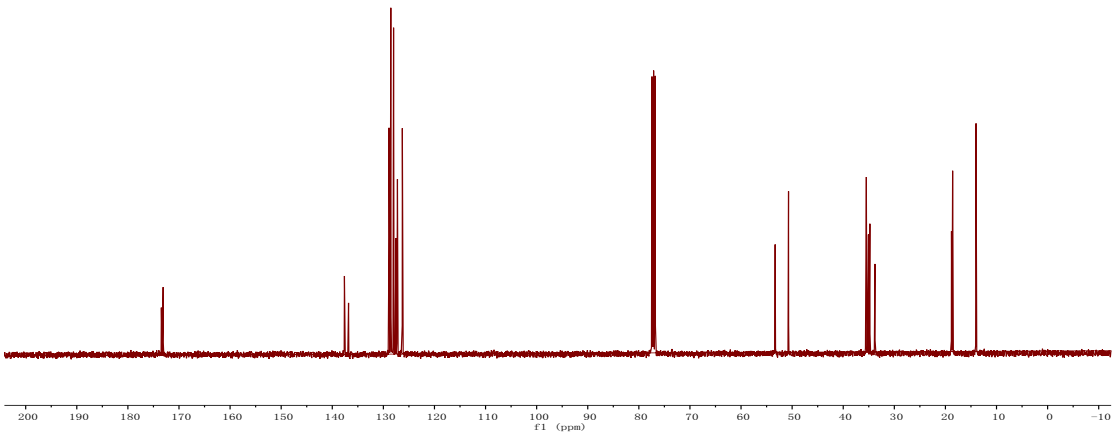


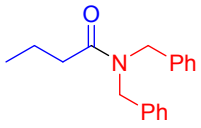




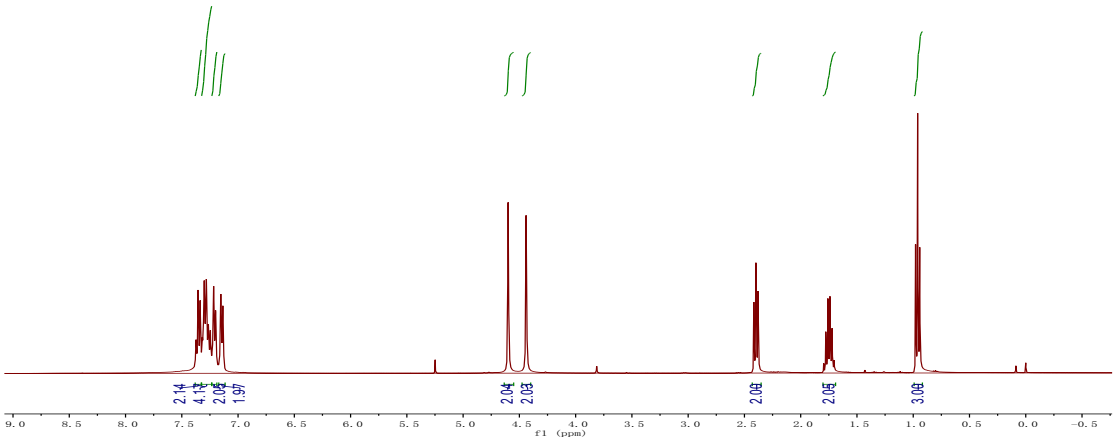
N-Benzyl-N-methyl-butyramide (**3i**). N-Methylbenzylamine (182mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then 1-butanol (133mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification as colorless oil (132mg, 46% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39–7.20 (m, 4H), 7.16 (d, *J* = 7.3 Hz, 1H), 4.56 (d, *J* = 24.0 Hz, 2H), 2.92 (d, *J* = 11.1 Hz, 3H), 2.35 (td, *J* = 7.5, 2.5 Hz, 2H), 1.78–1.64 (m, 2H), 0.97 (dt, *J* = 21.8, 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.4, 173.1, 137.6, 136.8, 128.9, 128.5, 128.0, 127.5, 127.2, 126.3, 53.3, 50.7, 35.5, 35.1, 34.8, 33.8, 18.8, 18.6, 14.0; HRMS (ESI) m/z calcd for $C_{12}H_{17}NO$ $[M+H]^+$ 192.1344, found 192.1363.

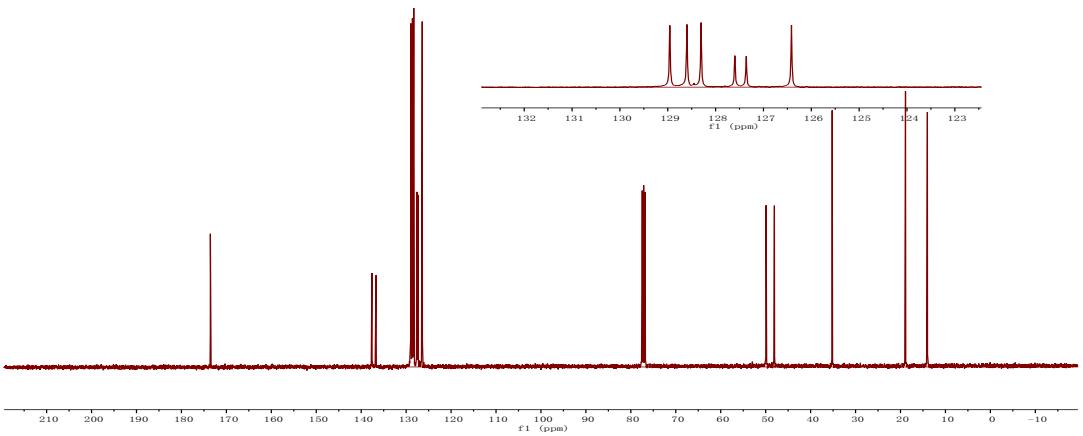


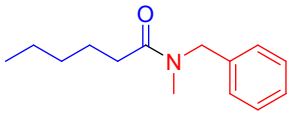




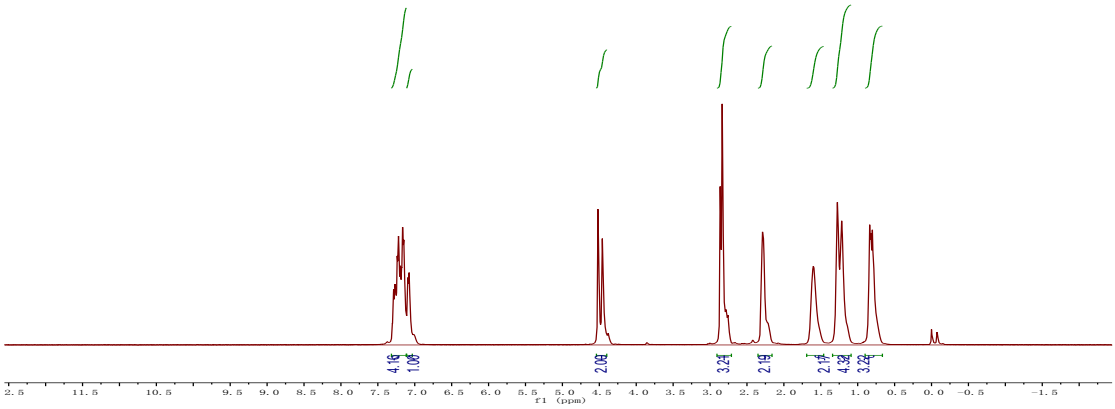
N,N-Dibenzylbutyramide (**3j**). Dibenzylamine (296mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then 1-butanol (133mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification as colorless oil (221mg, 55% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (t, *J* = 7.3 Hz, 2H), 7.32–7.23 (m, 4H), 7.21 (d, *J* = 6.8 Hz, 2H), 7.14 (d, *J* = 7.2 Hz, 2H), 4.60 (s, 2H), 4.44 (s, 2H), 2.43–2.35 (m, 2H), 1.80–1.69 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.6, 137.6, 136.7, 128.9, 128.6, 128.3, 127.6, 127.4, 126.4, 49.9, 48.1, 35.2, 18.9, 14.0; HRMS (ESI) m/z calcd for $C_{18}H_{21}NO$ $[M+H]^+$ 268.1657, found 268.1667.

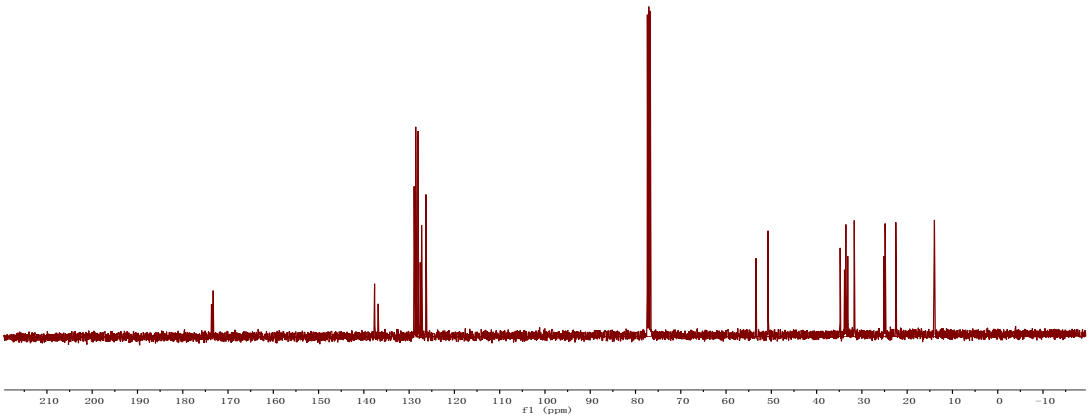


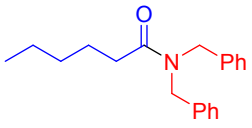




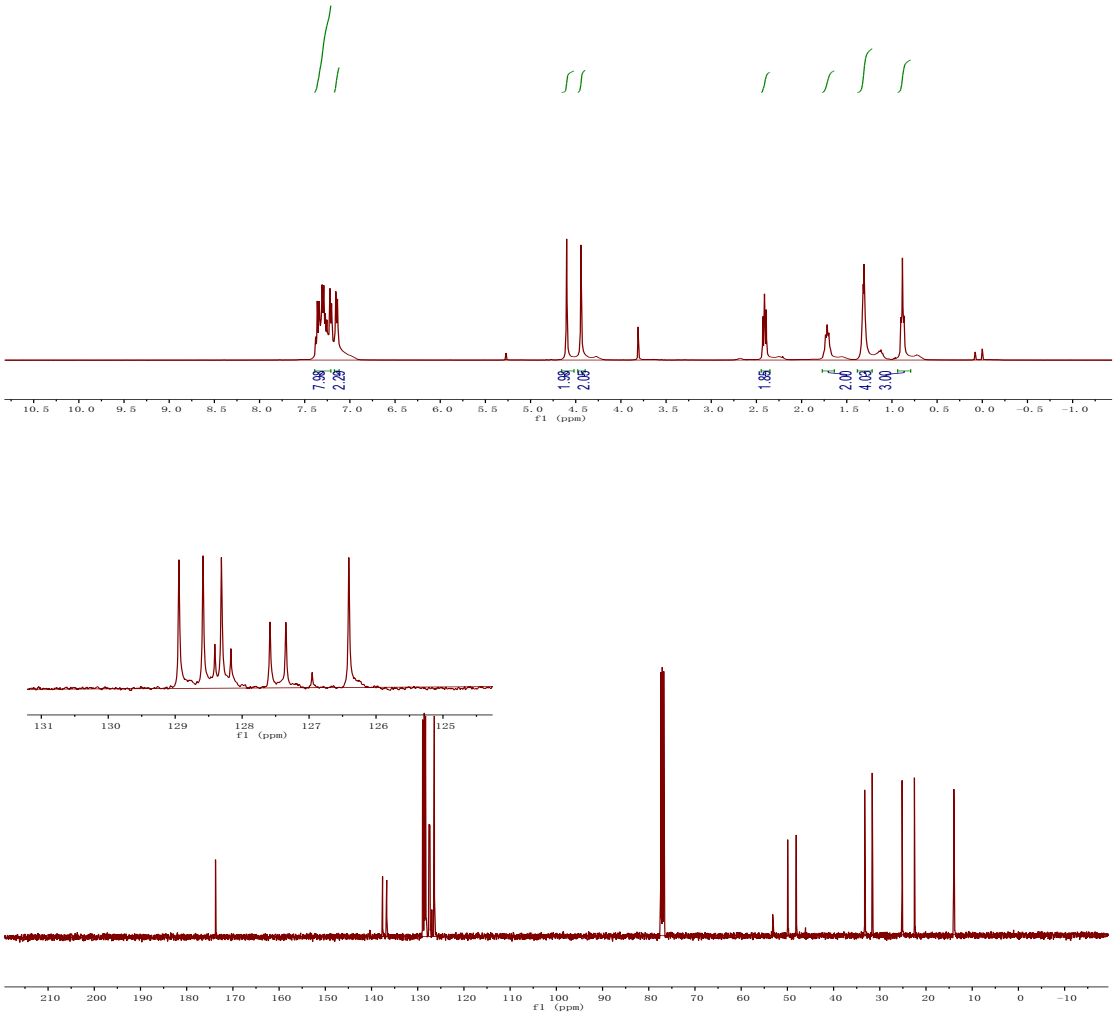
N-Benzyl-N-methyl-hexanamide (**3k**). N-Methylbenzylamine (182mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then hexyl alcohol (184mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification as colorless oil (155mg, 47% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32–7.12 (m, 4H), 7.08 (d, *J* = 6.6 Hz, 1H), 4.49 (d, *J* = 23.7 Hz, 2H), 2.91–2.71 (m, 3H), 2.28 (d, *J* = 5.1 Hz, 2H), 1.60 (s, 2H), 1.25 (d, *J* = 23.0 Hz, 4H), 0.82 (dd, *J* = 8.9, 5.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.6, 173.3, 137.6, 136.8, 128.9, 128.5, 128.0, 127.6, 127.2, 126.3, 53.4, 50.7, 34.8, 33.8, 33.5, 33.1, 31.7, 31.6, 25.1, 24.9, 22.5, 22.48, 14.0, 13.9; HRMS (ESI) m/z calcd for $C_{14}H_{21}NO$ $[M+H]^+$ 220.1657, found 220.1682.

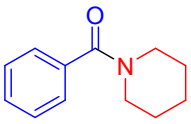




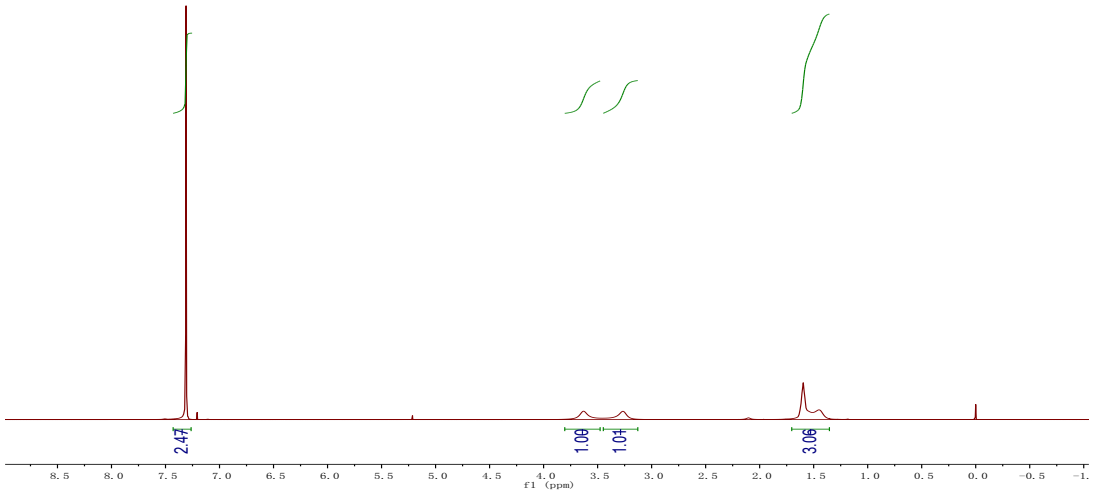


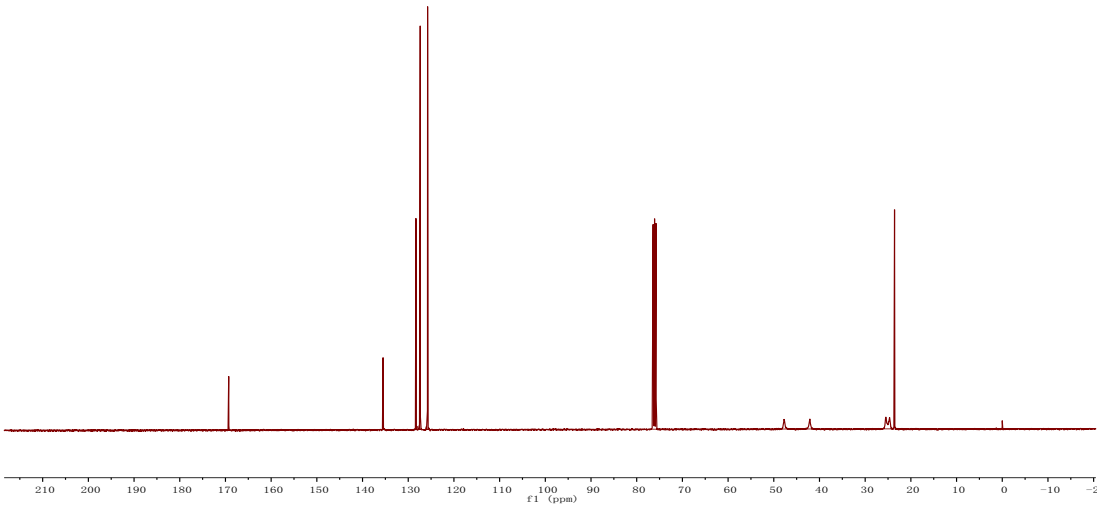
N,N-Dibenzylhexanamide (**3l**). Dibenzylamine (296mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then hexyl alcohol (184mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification as colorless oil (230mg, 52% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39–7.21 (m, 8H), 7.15 (d, *J* = 7.3 Hz, 2H), 4.60 (s, 2H), 4.44 (s, 2H), 2.45–2.35 (m, 2H), 1.72 (dt, *J* = 14.5, 7.4 Hz, 2H), 1.31 (dd, *J* = 8.9, 5.3 Hz, 4H), 0.89 (dd, *J* = 9.1, 4.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.8, 137.6, 136.7, 128.9, 128.4, 128.3, 128.2, 127.6, 127.4, 127.0, 126.4, 53.2, 49.9, 48.1, 33.3, 31.6, 25.2, 22.5, 14.0; HRMS (ESI) m/z calcd for $C_{20}H_{25}NO$ $[M+H]^+$ 296.1970, found 296.1988.

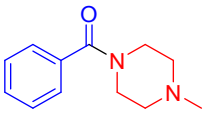




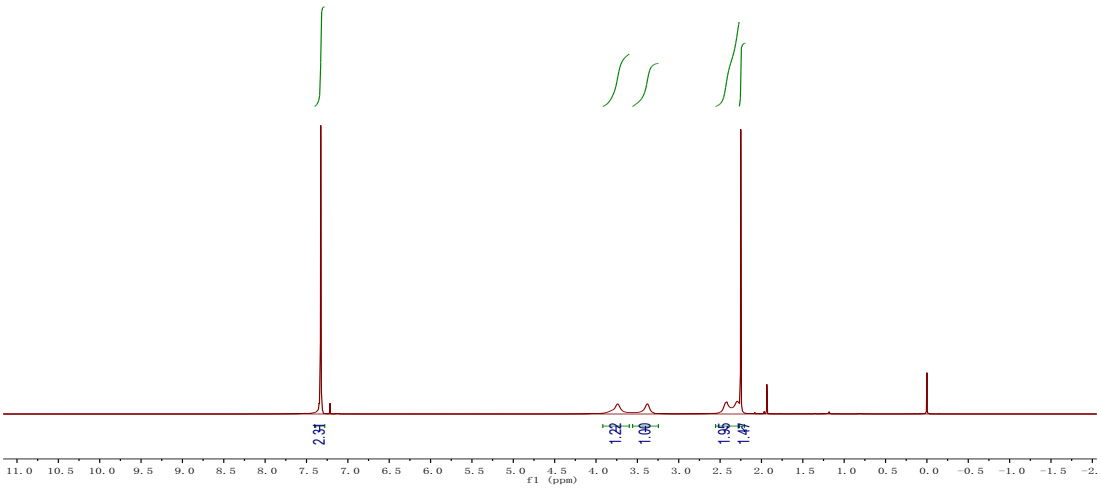
Benzoylpiperidine (**3m**). Piperidine (128mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 22h. And the product was obtained after purification as colourless oil (252mg, 89% yield); ^{1}H NMR(400 MHz, $CDCl_3$) δ 7.31(s, 5H), 3.63 (s, 2H), 3.27 (s, 2H), 1.70–1.35 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.3, 135.5, 128.3, 127.4, 125.8, 47.8, 42.1, 25.4, 24.6, 23.6; HRMS (ESI) m/z calcd for $C_{12}H_{15}NO$ $[M+H]^+$ 190.1187, found 190.1192.

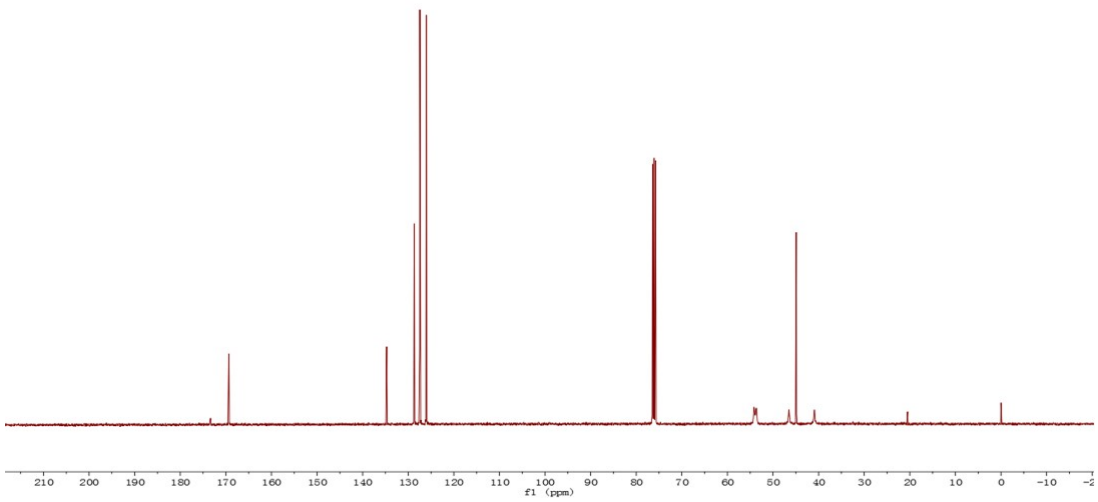


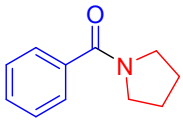




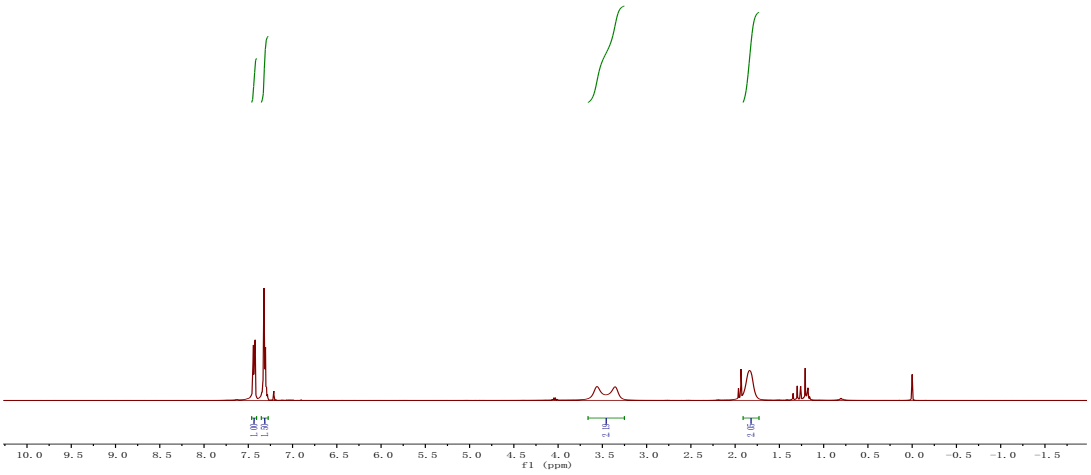
1-Benzoyl-4-methylpiperazine (**3n**). 1-Methylpiperazine (150mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 20h. And the product was obtained after purification as yellow oil (257mg, 84% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40–7.28 (m, 5H), 3.74 (s, 2H), 3.34 (s, 2H), 2.55–2.27 (m, 4H), 2.25 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.4, 169.3, 134.7, 128.7, 127.5, 126.0, 54.2, 53.6, 46.5, 44.9, 40.9, 20.5; HRMS (ESI) m/z calcd for $C_{12}H_{16}N_2O$ $[M+H]^+$ 205.1296, found 205.1305.

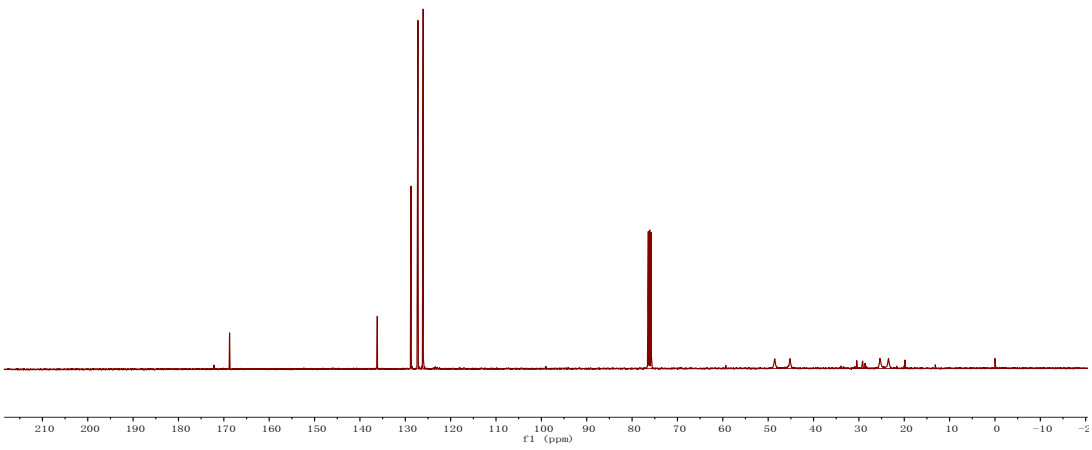


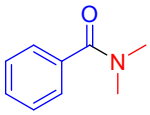




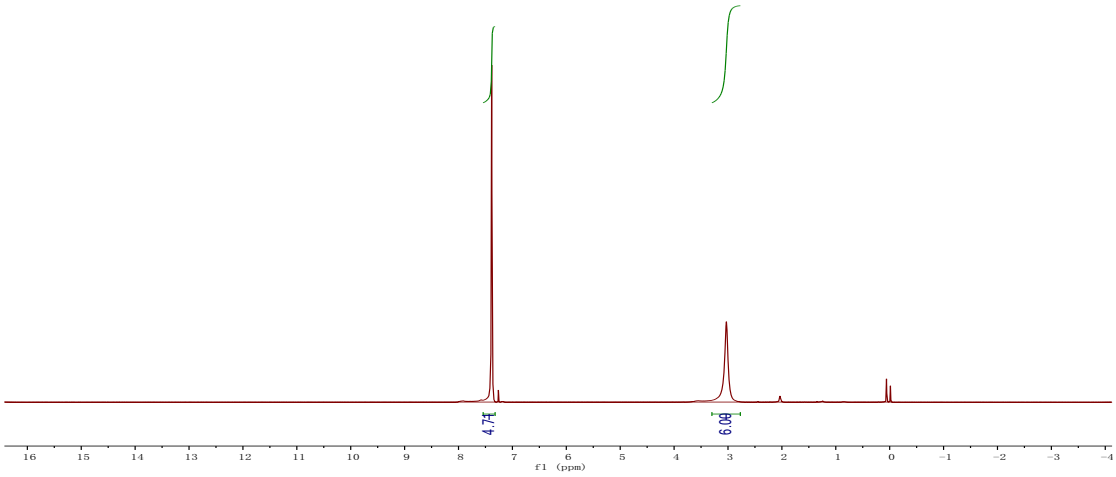
Benzoylpyrrolidine (**3o**). Pyrrolidin (106mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification as colourless oil (228mg, 87% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (dt, *J* = 8.5, 3.7 Hz, 2H), 7.35–7.28 (m, 3H), 3.46 (d, *J* = 81.7 Hz, 4H), 1.84 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 136.2, 128.8, 127.2, 126.0, 48.6, 45.2, 25.4, 23.5; HRMS (ESI) m/z calcd for $C_{11}H_{13}NO$ $[M+H]^+$ 176.1031, found 176.1045.

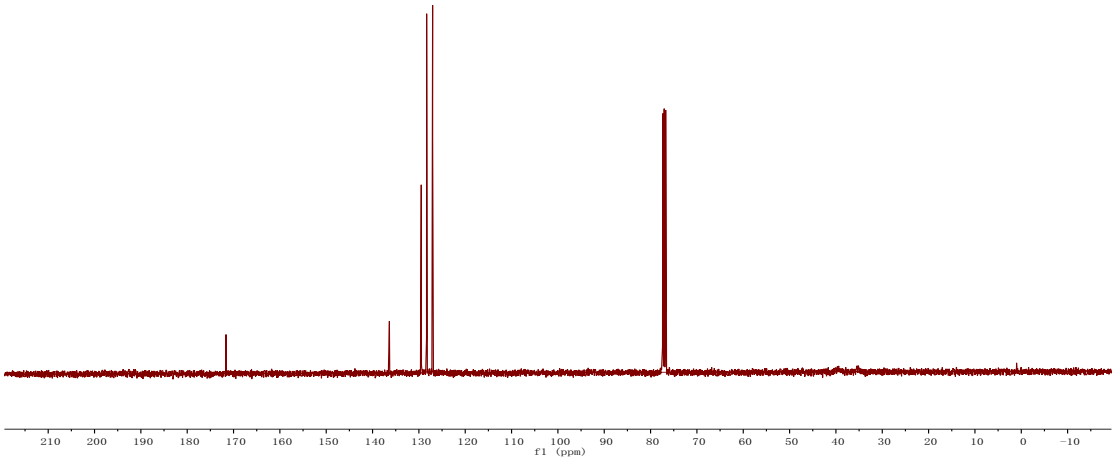


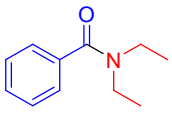




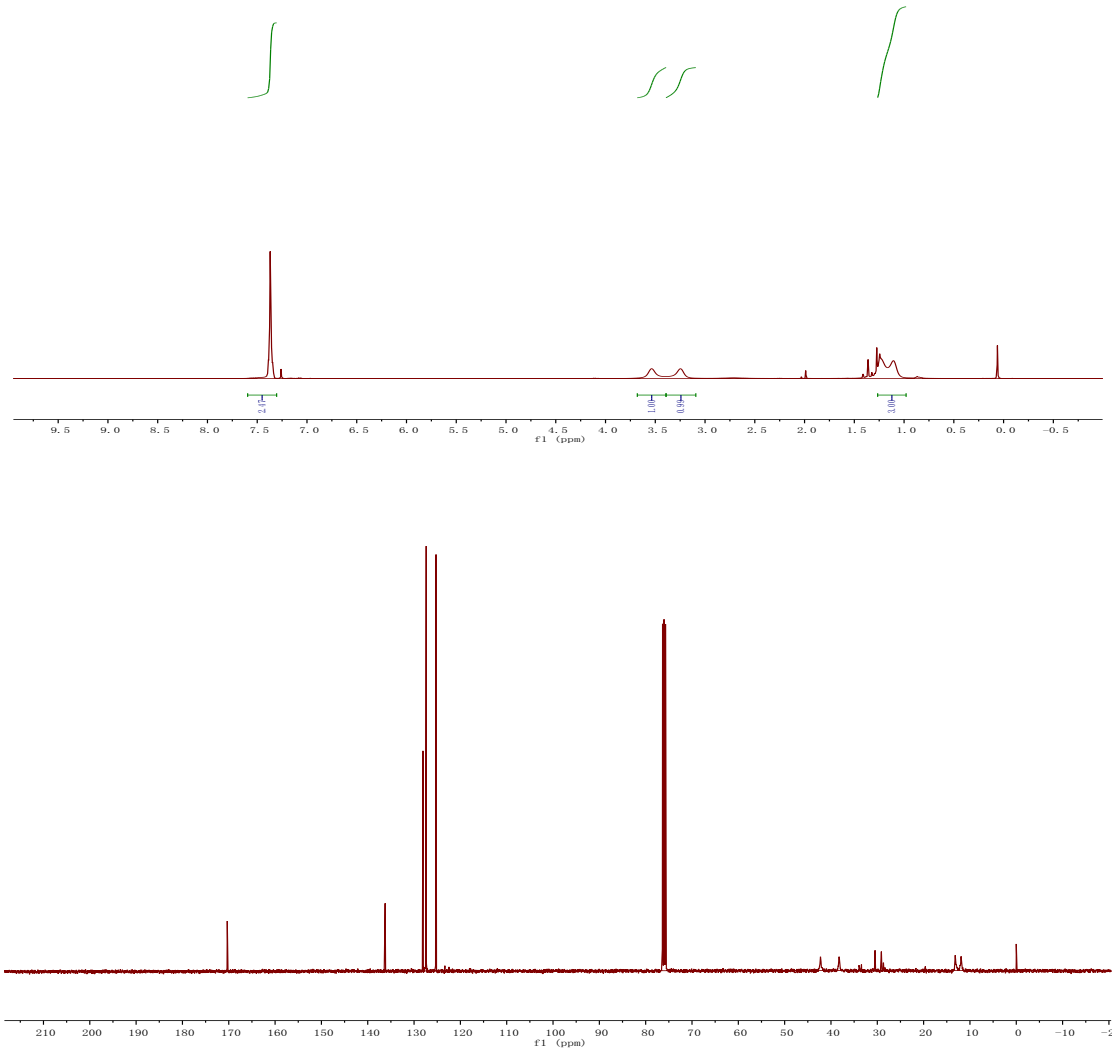
N,N-Dimethylbenzamide (**3p**). Dimethylamine (40 wt% in water, 169mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 20h. And the product was obtained after purification as white solid (163mg, 73% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54–7.32 (m, 5H), 3.03 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.6, 136.4, 129.5, 128.3, 127.0; HRMS (ESI) m/z calcd for $C_9H_{11}NO$ $[M+H]^+$ 150.0874, found 150.0889.

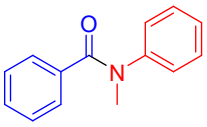




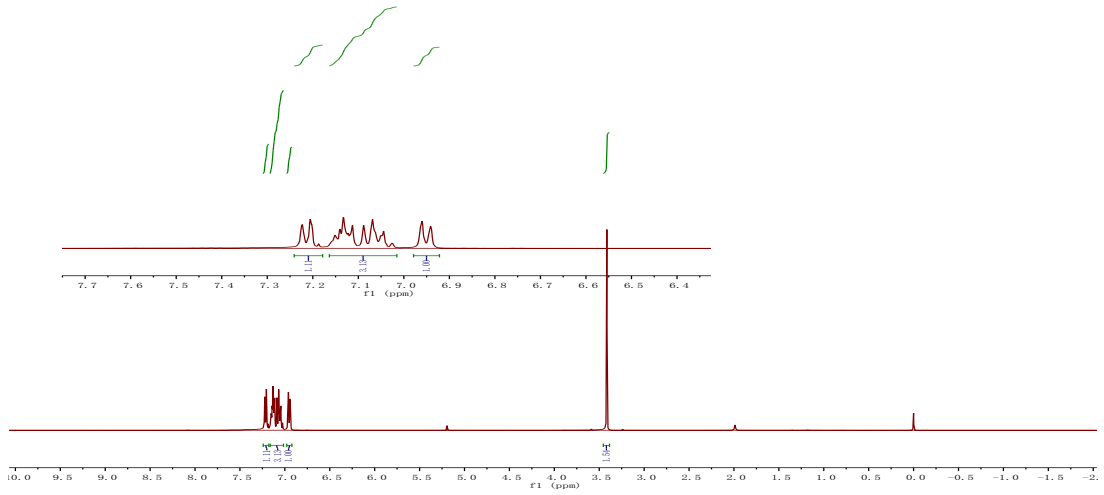


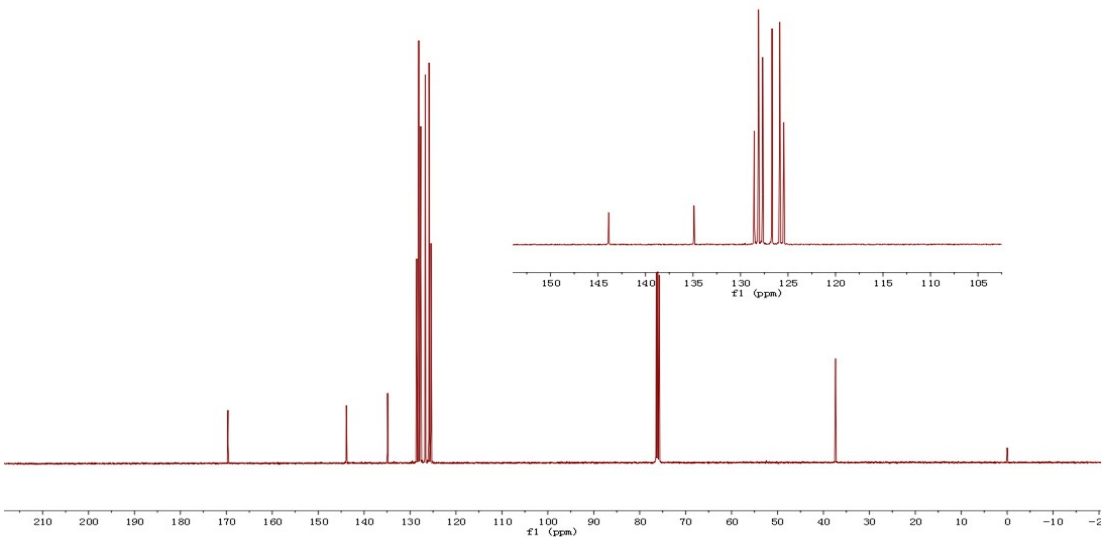
N,N-Diethylbenzamide (**3q**). Diethylamine (110mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 22h. And the product was obtained after purification as colourless oil (196mg, 74% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60–7.30 (m, 5H), 3.54 (s, 2H), 3.25 (s, 2H), 1.27–0.98 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.3, 136.2, 128.1, 127.4, 125.3, 42.3, 38.2, 30.5, 29.1, 13.2, 11.9; HRMS (ESI) m/z calcd for $C_{11}H_{15}NO$ $[M+H]^+$ 178.1187, found 178.1183.

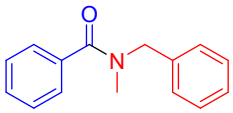




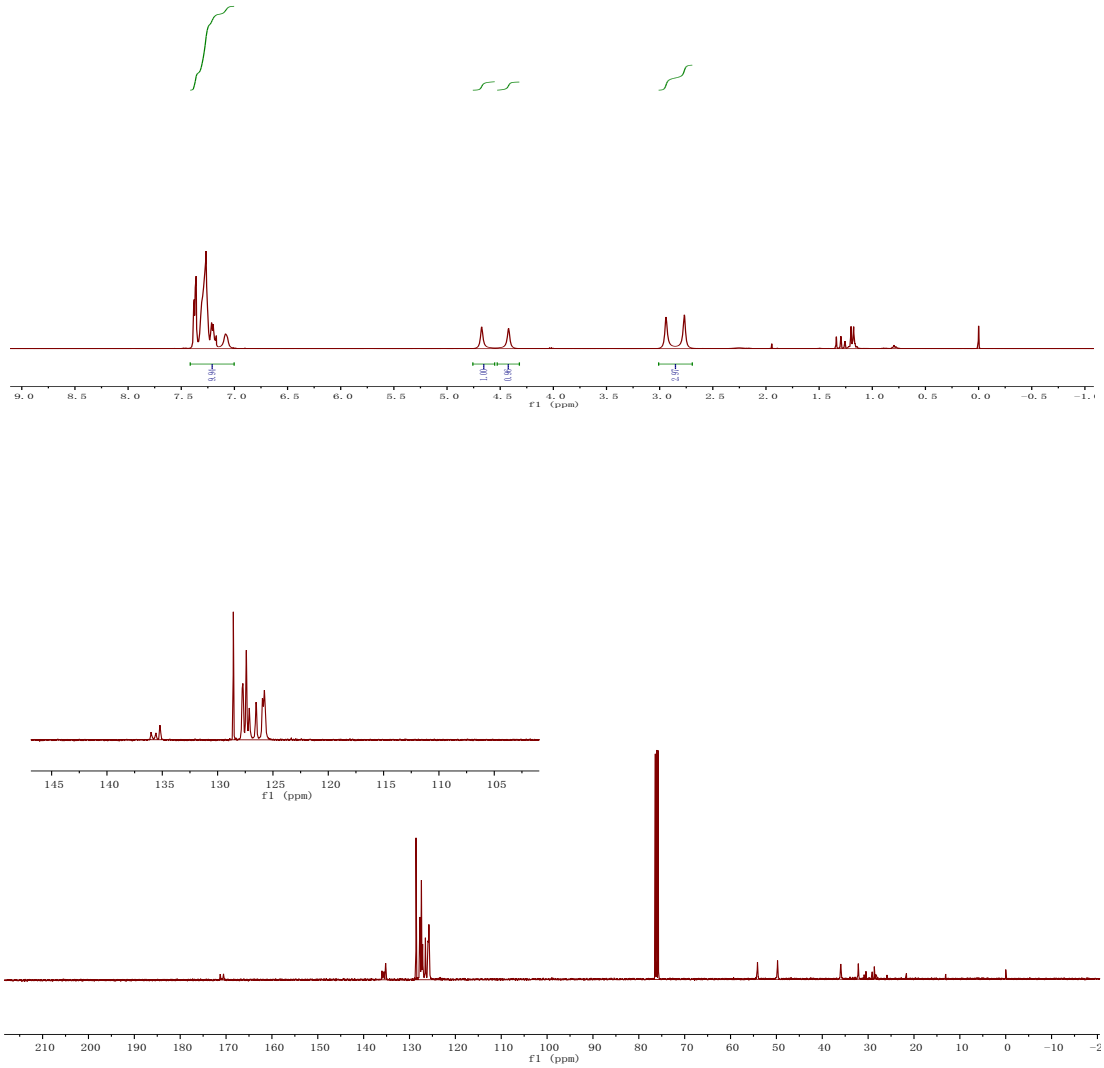
N-Methyl-N-phenylbenzamide (**3r**). N-Methylaniline (160mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification as colourless oil (250mg, 79% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (dd, J = 7.9, 6.6 Hz, 2H), 7.16–7.02 (m, 6H), 6.95 (d, J = 7.6 Hz, 2H), 3.42 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.6, 143.9, 134.9, 128.6, 128.1, 127.7, 126.7, 125.9, 125.4, 37.4; HRMS (ESI) m/z calcd for $C_{14}H_{13}NO$ $[M+H]^+$ 212.1031, found 212.1048.

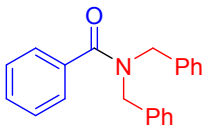




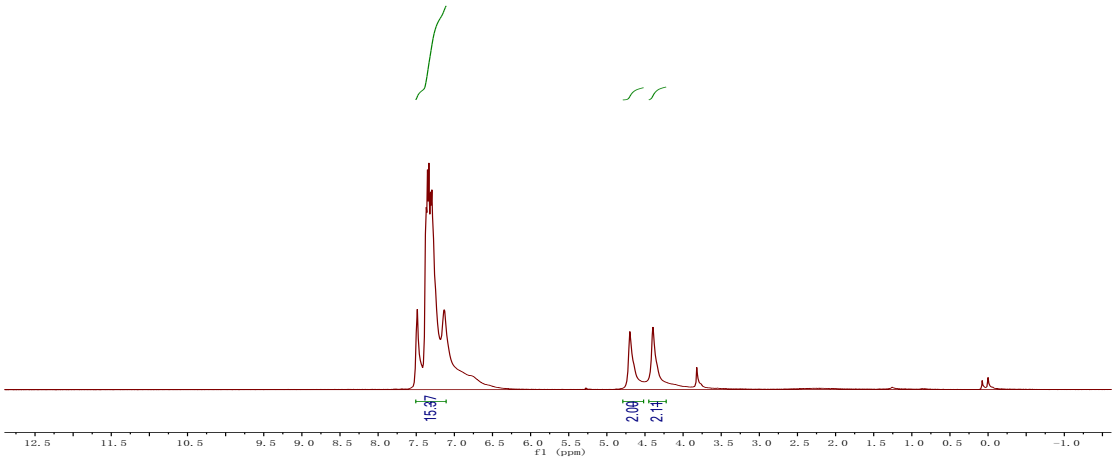


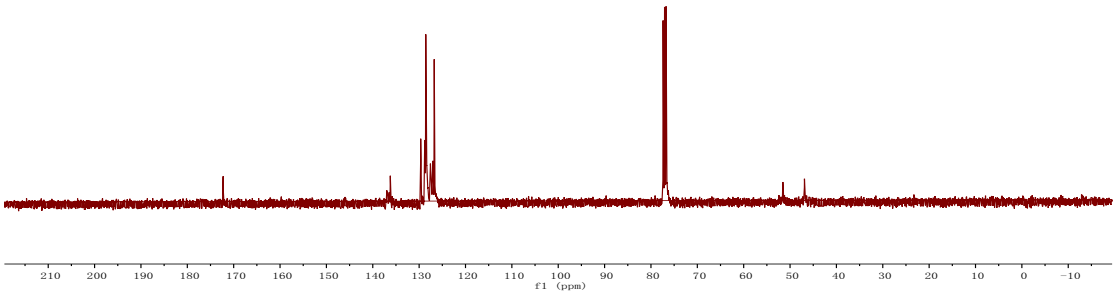
N-Benzyl-N-methylbenzamide (**3s**). N-Methylbenzylamine (182mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification as colourless oil (294mg, 87% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42–7.00 (m, 10H), 4.67 (s, 1H), 4.42 (s, 1H), 2.85 (d, *J* = 69.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 136.0, 135.6, 135.2, 128.6, 127.7, 127.4, 127.1, 126.5, 125.9, 54.1, 49.8, 36.0, 32.1, 28.6; HRMS (ESI) m/z calcd for $C_{15}H_{15}NO$ $[M+H]^+$ 226.1187, found 226.1189.

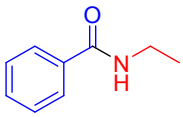




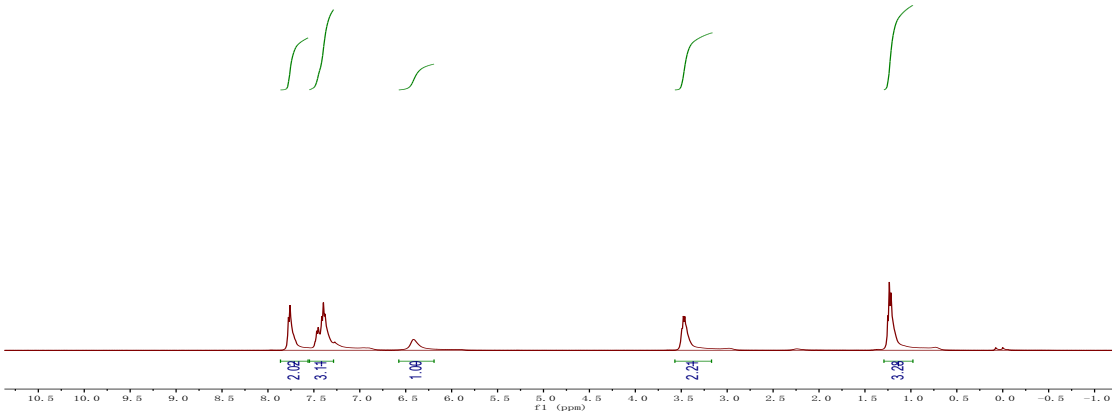
N,N-Dibenzylbenzamide (**3t**). Dibenzylamine (296mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification as white solid (410mg, 91% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.11 (m, 15H), 4.70 (s, 2H), 4.40 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.3, 137.0, 136.4, 136.2, 129.7, 128.8, 128.6, 128.4, 127.6, 127.0, 126.7, 51.6, 46.9; HRMS (ESI) m/z calcd for $C_{21}H_{19}NO$ $[M+H]^+$ 302.1500, found 302.1509.

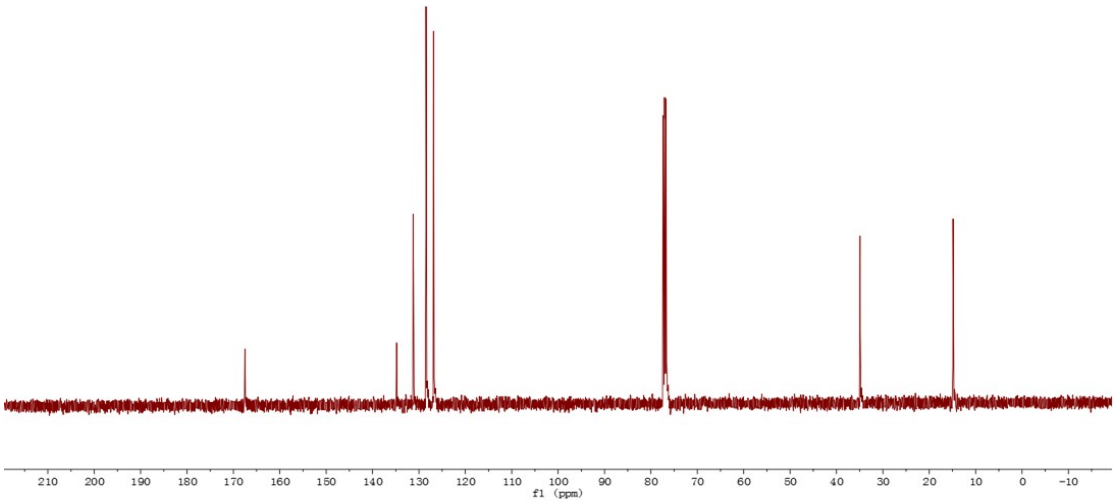


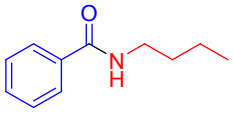




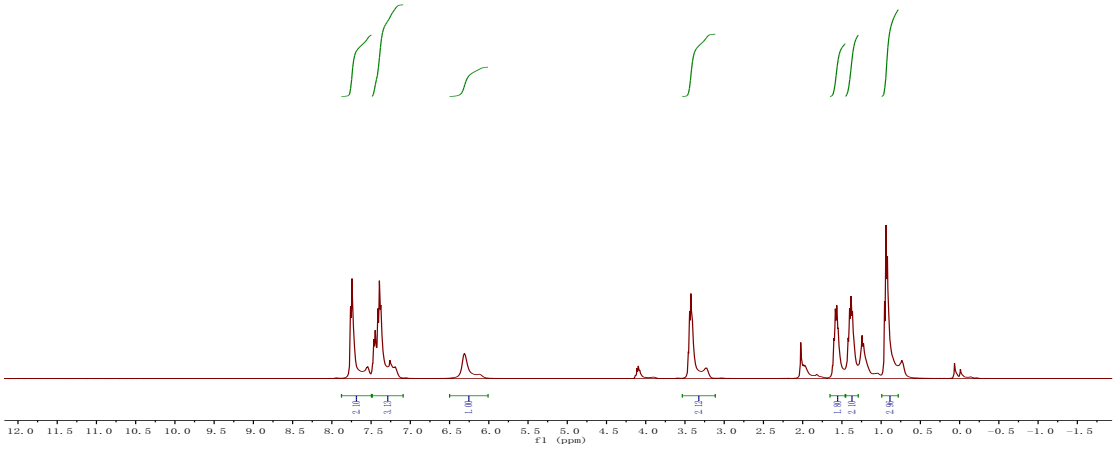
N-Ethylbenzamide (**3u**). Ethylamine (68mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 20h. And the product was obtained after purification as white solid (181mg, 81% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.1 Hz, 2H), 7.55–7.29 (m, 3H), 6.41 (s, 1H), 3.57–3.17 (m, 2H), 1.29–0.98 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.5, 134.8, 131.3, 128.5, 126.9, 34.9, 14.9; HRMS (ESI) m/z calcd for $C_9H_{11}NO$ $[M+H]^+$ 150.0874, found 150.0887.

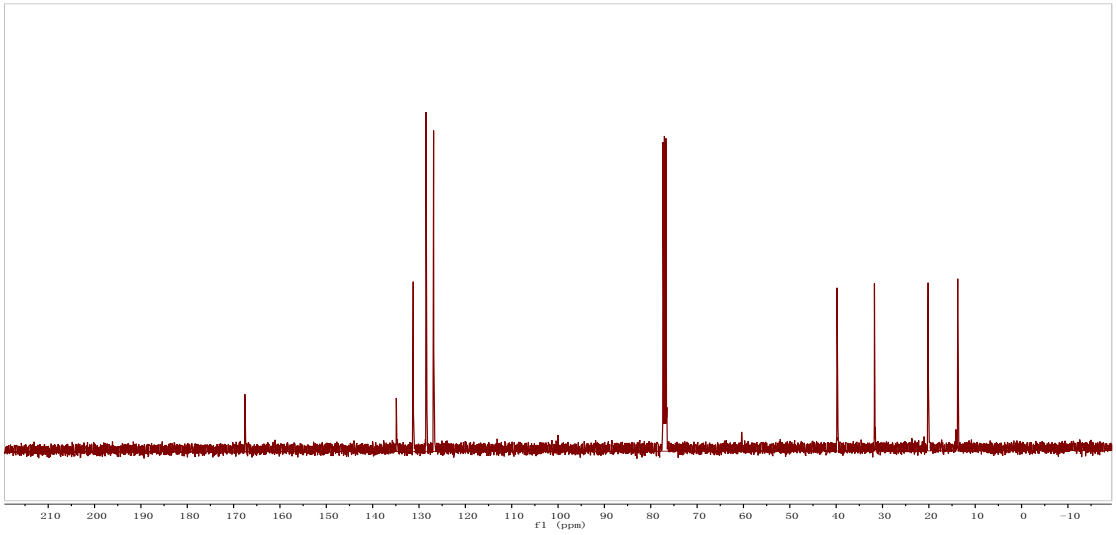


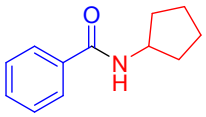




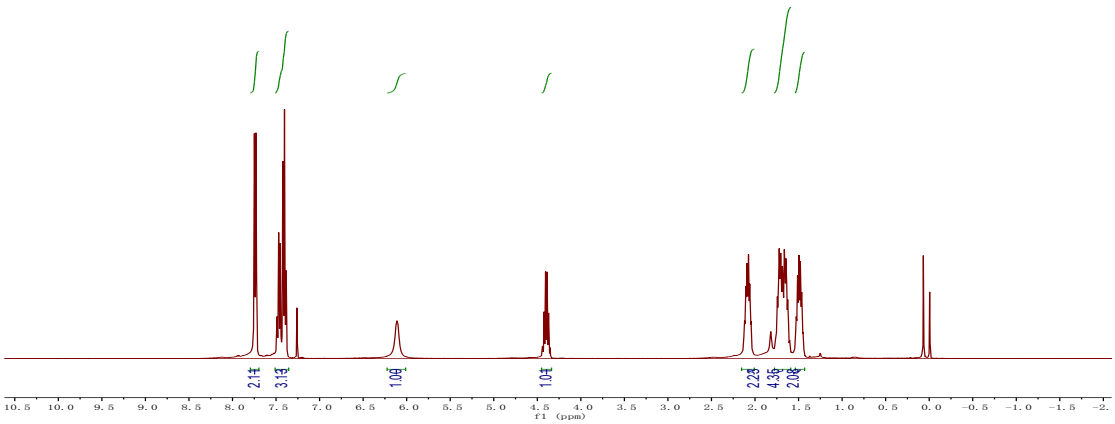
N-Butylbenzamide (**3v**). Butylamine (110mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 20h. And the product was obtained after purification as white solid (239mg, 90% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (dd, *J* = 93.1, 13.0 Hz, 2H), 7.48–7.09 (m, 3H), 6.22 (d, *J* = 75.4 Hz, 1H), 3.53–3.11 (m, 2H), 1.58 (dt, *J* = 14.0, 7.0 Hz, 2H), 1.39 (dd, *J* = 14.5, 7.2 Hz, 2H), 0.94 (dd, *J* = 9.2, 5.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.6, 134.9, 131.2, 128.5, 126.8, 39.8, 31.7, 20.2, 13.8; HRMS (ESI) m/z calcd for $C_{11}H_{15}NO$ $[M+H]^+$ 178.1187, found 178.1189.

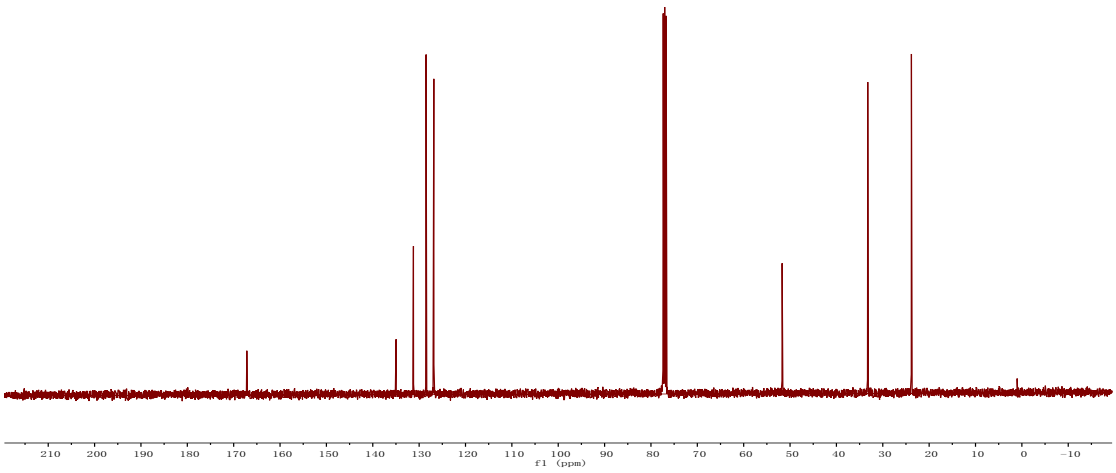


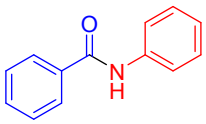




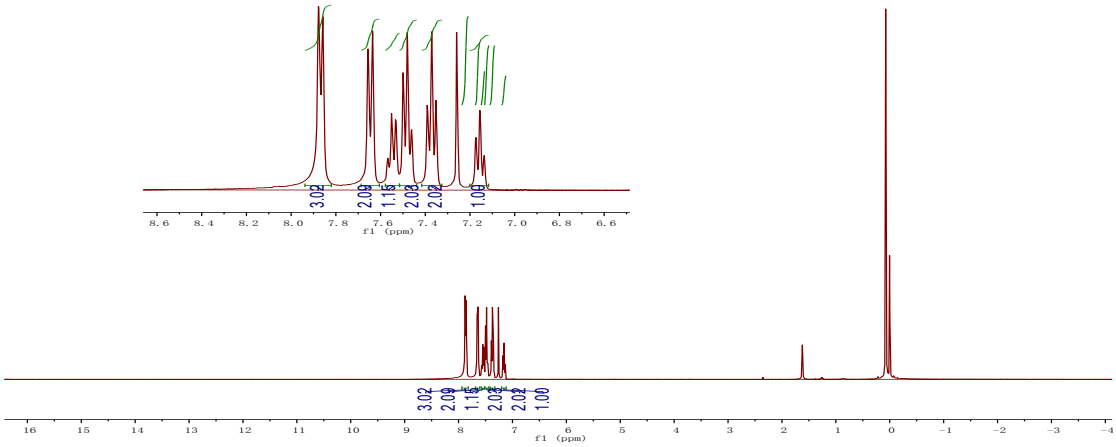
N-Cyclopentylbenzamide (**3x**). Cyclopentylamine (128mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 22h. And the product was obtained after purification as white solid (226mg, 80% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80–7.70 (m, 2H), 7.51–7.35 (m, 3H), 6.11 (s, 1H), 4.45–4.34 (m, 1H), 2.08 (td, *J* = 11.4, 6.1 Hz, 2H), 1.78–1.59 (m, 4H), 1.49 (td, *J* = 12.5, 6.1 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.2, 135.0, 131.2, 128.5, 126.8, 51.7, 33.2, 23.8; HRMS (ESI) m/z calcd for $C_{12}H_{15}NO$ $[M+H]^+$ 190.1187, found 190.1192.

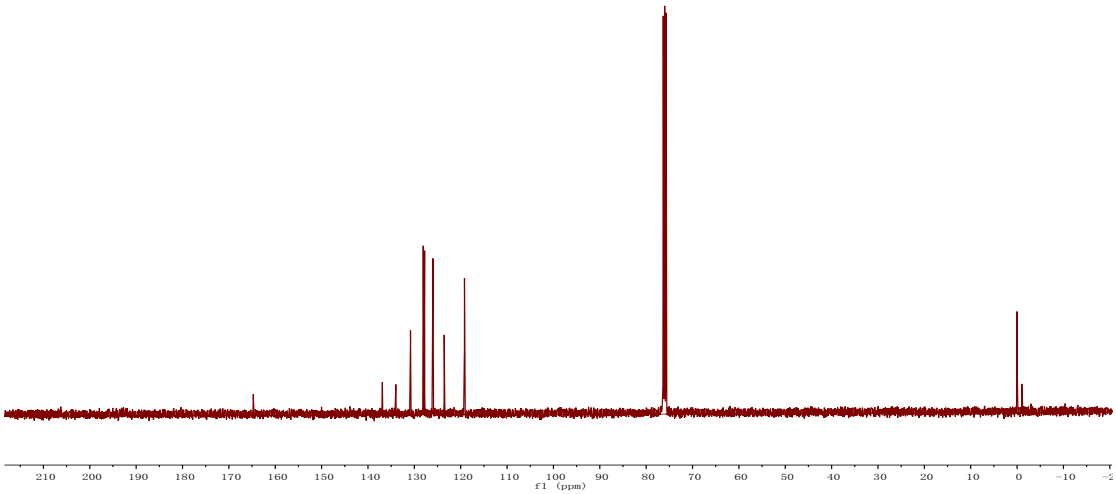


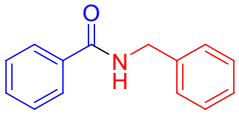




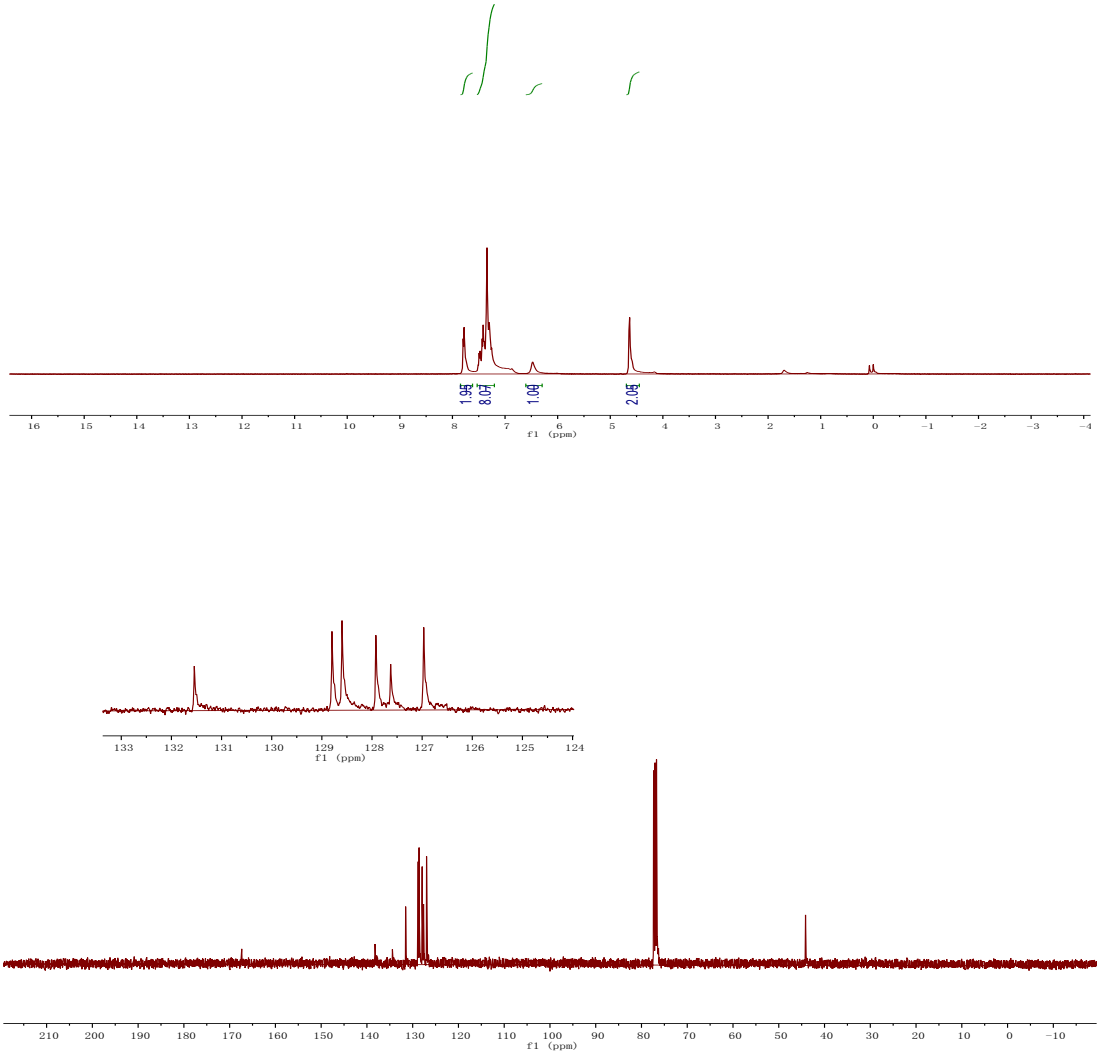
N-Phenylbenzamide (**3y**). Aniline (140mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification aswhite solid (200mg, 68% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.9 Hz, 3H), 7.65 (d, *J* = 8.1 Hz, 2H), 7.58–7.52 (m, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.37 (t, *J* = 7.8 Hz, 2H), 7.16 (t, *J* = 7.4 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.7, 136.9, 134.0, 130.8, 128.1, 127.8, 126.0, 123.6, 119.2; HRMS (ESI) m/z calcd for $C_{13}H_{11}NO$ $[M+H]^+$ 198.0874, found 198.0886.

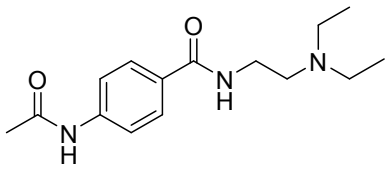






N-Benzylbenzamide (**3z**). Benzylamine (160mg, 1.5mmol, 1 equiv.) was treated with *N*-chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then benzyl alcohol (194mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 22h. And the product was obtained after purification as white solid (272mg, 86% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.2 Hz, 2H), 7.53–7.20 (m, 8H), 6.48 (s, 1H), 4.64 (d, *J* = 5.5 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.4, 138.2, 134.4, 131.5, 128.8, 128.6, 127.9, 127.6, 127.0, 44.2; HRMS (ESI) m/z calcd for $C_{14}H_{13}NO$ $[M+H]^+$ 212.1031, found 212.1084.





4-Acetamido-N-[(2-diethylamino)ethyl]benzamide (**5**). N,N-Diethyl-1,2-ethanediamine (174mg, 1.5mmol, 1 equiv.) was treated with *N*- chlorosuccinimide (NCS) (220mg, 1.65mmol, 1.1 equiv.) in 4mL acetonitrile at room temperature for 3h, and then 4-acetamidobenzyl alcohol (297mg, 1.8mmol, 1.2 equiv.), $CuSO_4 \cdot 5H_2O$ (75mg, 0.3mmol, 20 mol%) and TBHP (70 wt% in water, 965mg, 7.5mmol, 5 equiv.) were added to the reaction mixture, stirring at 80°C for 24h. And the product was obtained after purification aswhite solid (282mg, 68% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.6 Hz, 2H), 7.64 – 7.49 (m, 3H), 6.92 (s, 1H), 3.47 (dd, *J* = 11.3, 5.3 Hz, 2H), 2.64 (t, *J* = 5.9 Hz, 2H), 2.56 (q, *J* = 7.1 Hz, 4H), 2.20 (s, 3H), 1.03 (t, *J* = 7.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.4, 140.7, 130.2, 127.9, 119.2, 51.3, 46.8, 37.2, 12.0.