

Appendix

APPENDIX

Figure A.1 TEM image of ZO-5 (a), particle size distribution of ZO-5 (b), TEM image of ZO-15 (c) and particle size distribution of ZO-15 (d).

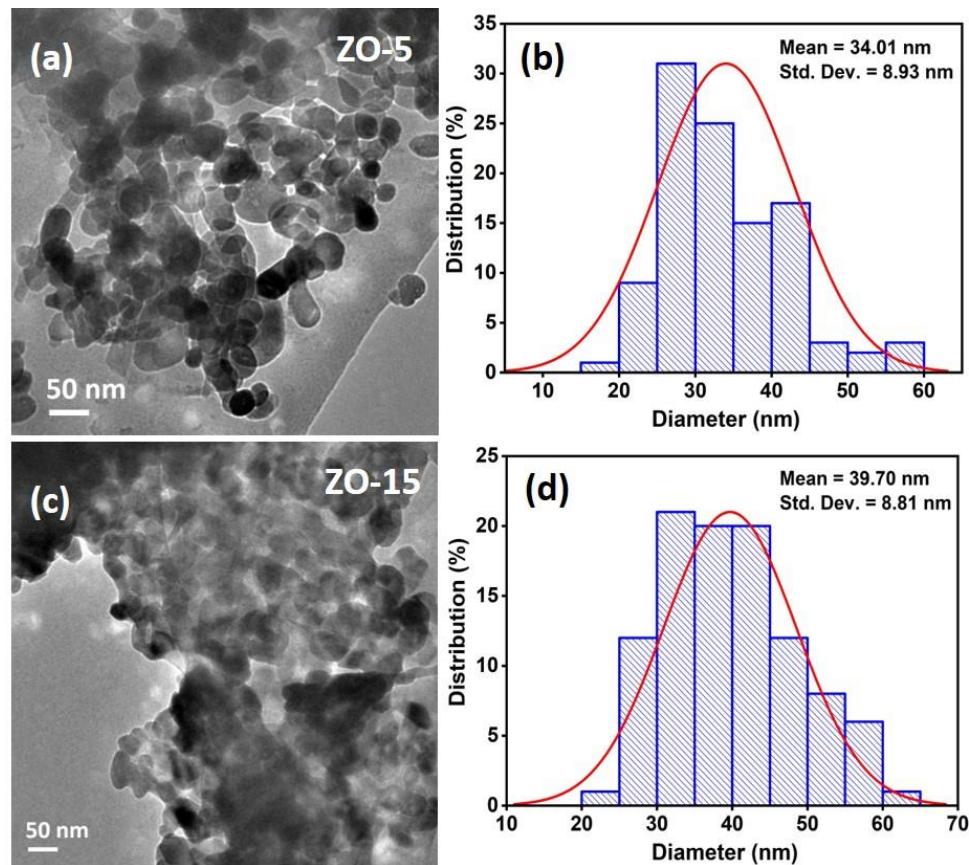


Figure A.2 Mid-FTIR spectrum (a), far-FTIR spectrum (b) and SEM images (c,d) of SnO₂ nanosheets assembly.

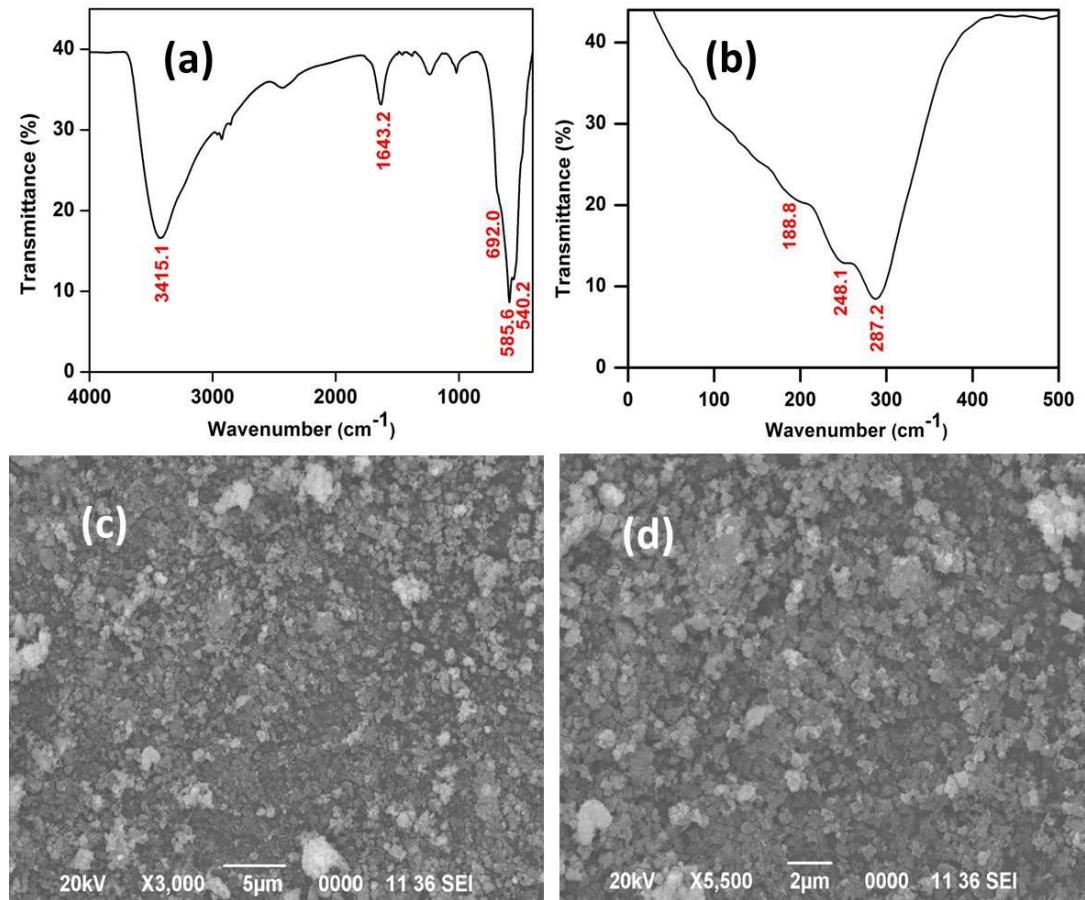


Table A.1 Lattice and Structure Parameters of Scrutinyite-SnO₂

Lattice parameters (Rietveld refinement data)						
a (Å)	b (Å)	c (Å)	Cell volume, V (Å³)		Correlation unit	
4.8106	5.7551	16.0239	433.60		0.998	
Structure parameters (Theoretical model data)						
Atom	Site/Wyck	Occupancy	x	y	z	B (Å²)
Position						
Sn1	8d	1	0.0030	0.3437	0.0840	1
Sn2	4c	1	0.0000	0.0208	0.2500	1
O3	8d	1	0.2063	0.3762	0.5295	1
O4	8d	1	0.2109	0.0659	0.3633	1
O5	8d	1	0.2271	0.2948	0.1947	1

Figure A.3 Raman spectrum of SnO₂ nanosheets assembly.

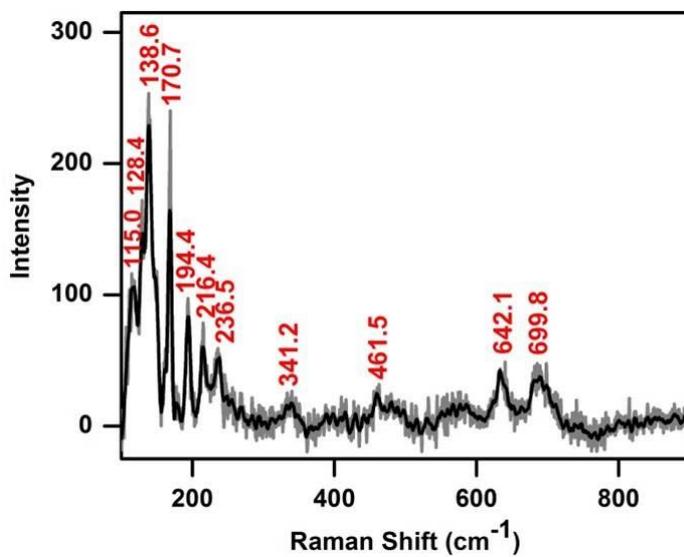


Figure A.4 NH₃-TPD profile of SnO₂ nanosheets assembly.

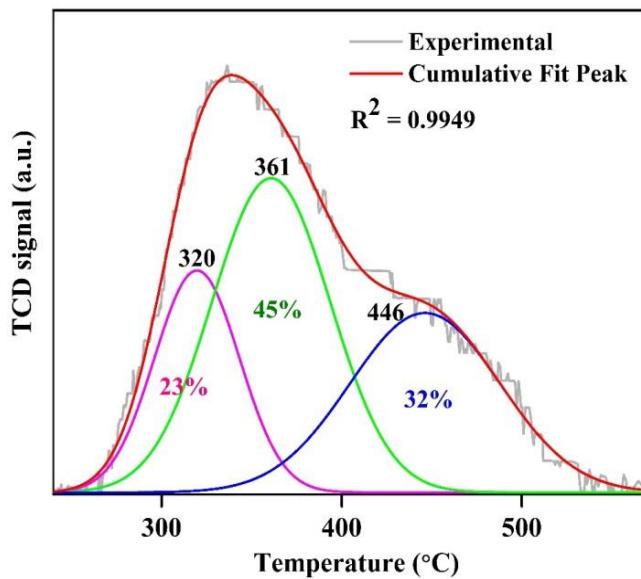


Figure A.5 Effect of temperature on the yield of 4-methoxybenzophenone; Reaction conditions: anisole (4 mmol), benzoyl chloride (2 mmol) and catalyst (10 mol %).

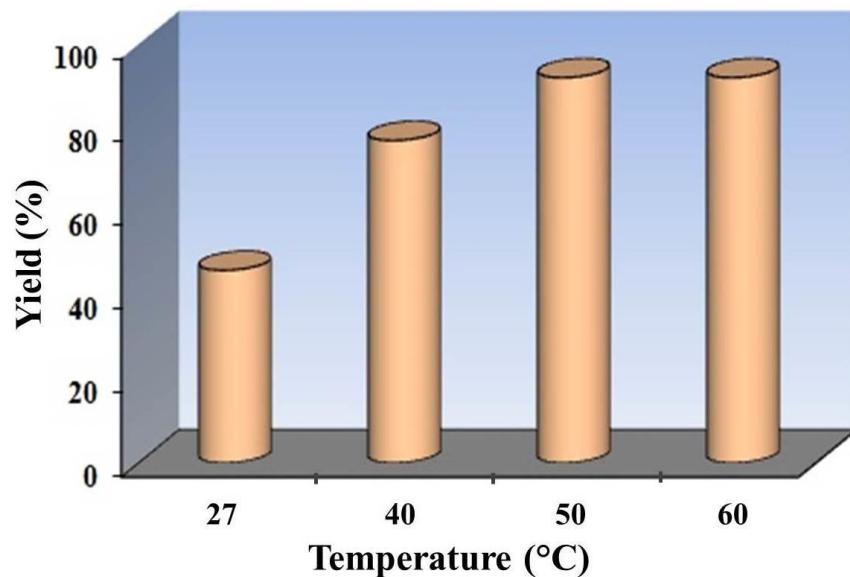


Figure A.6 Effect of catalyst dosage on the yield of 4-methoxybenzophenone; Reaction conditions: anisole (4 mmol), benzoyl chloride (2 mmol) and temperature (50 °C).

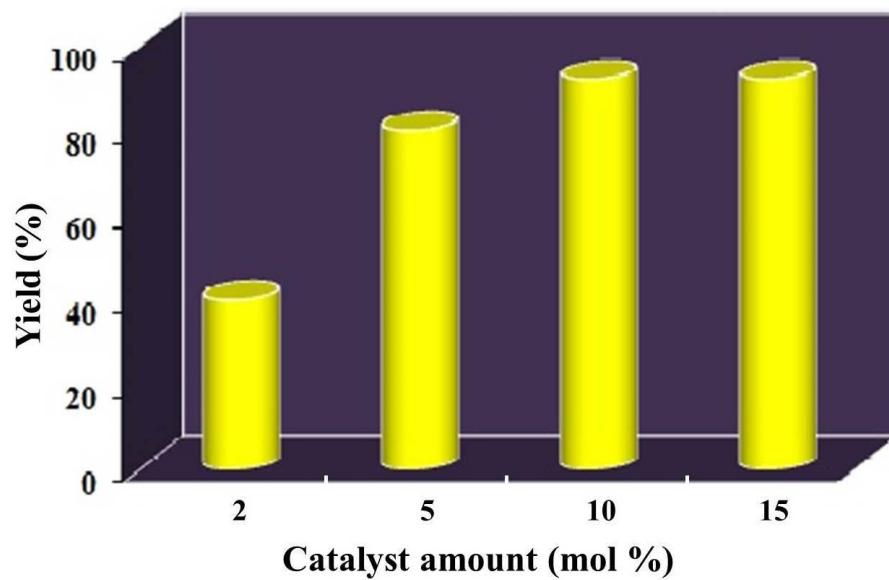


Figure A.7 FTIR spectrum of recovered SnO₂ nanosheets after six catalytic runs of Friedel-Crafts acylation reaction.

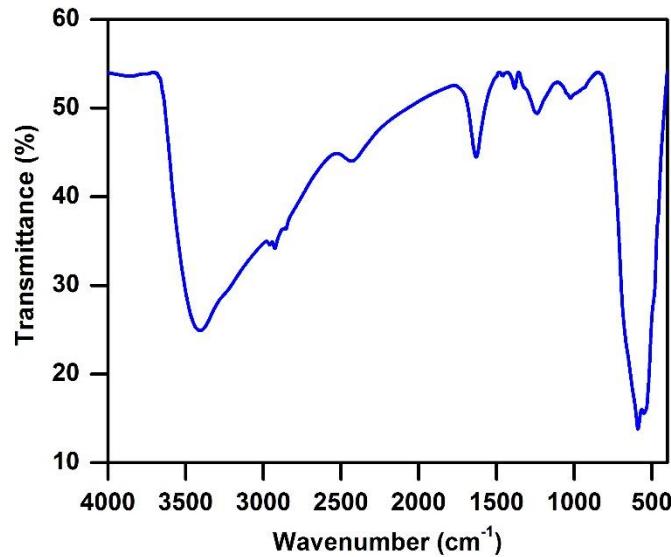


Figure A.8 Powder XRD (a) and TEM image (b) of recovered SnO₂ nanosheets after six catalytic runs of Friedel-Crafts acylation reaction.

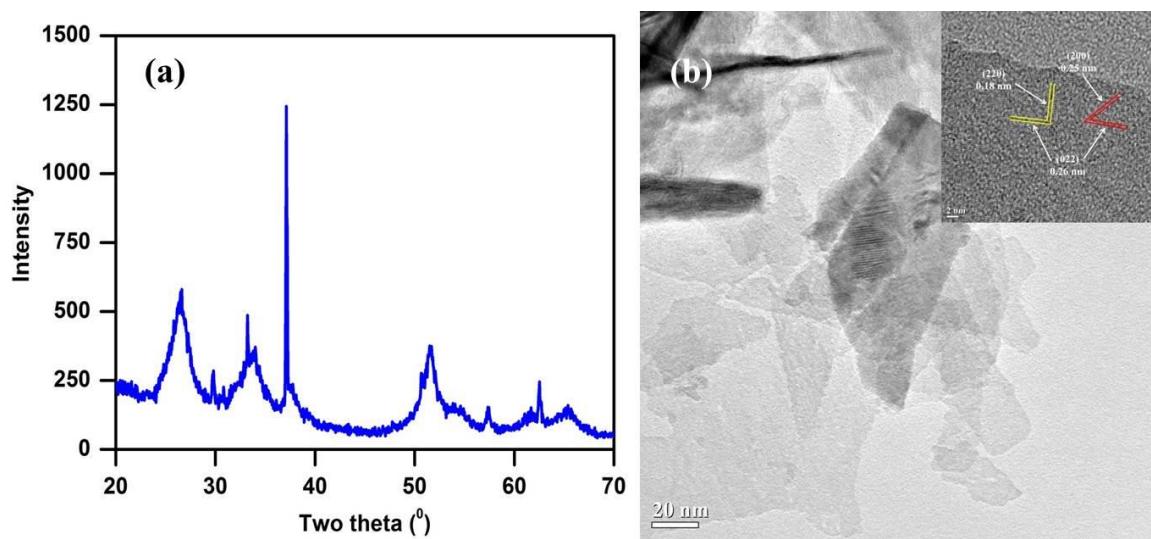
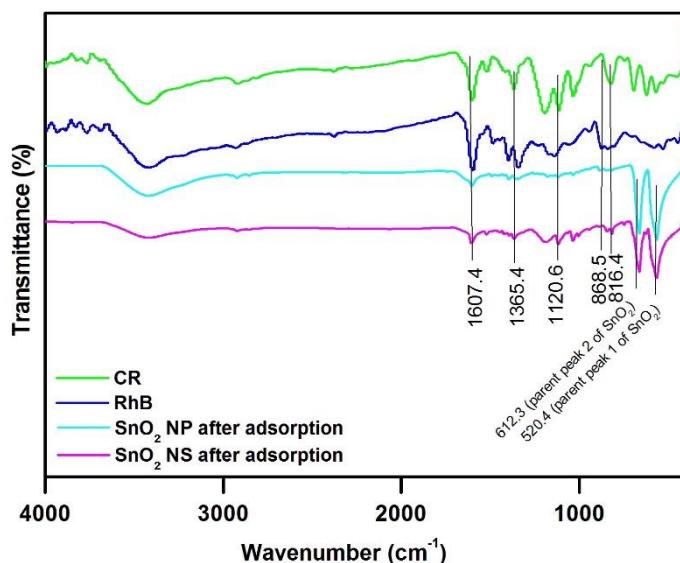
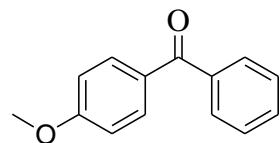


Figure A.9 FTIR spectra of pure dye pollutants, Rhodamine B (RhB) & Congo red (CR) and SnO_2 samples after adsorption of dye pollutants.



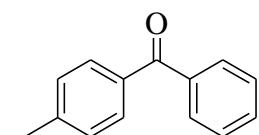
ANALYTICAL DATA OF PRODUCTS

1. 4-Methoxybenzophenone



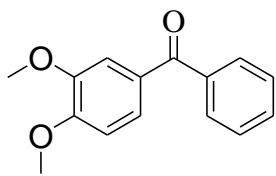
White solid, **M.p.** 59–60 °C. **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 7.84 (d, $J = 8.7$ Hz, 2H), 7.77 (d, $J = 8.2$ Hz, 2H), 7.58 (t, $J = 7.3$ Hz, 1H), 7.48 (t, $J = 7.8$ Hz, 2H), 6.97 (d, $J = 8.7$ Hz, 2H), 3.90 (s, 3H). **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 195.5, 163.2, 138.2, 132.5, 131.8, 130.1, 129.7, 128.14, 113.5, 55.5. **HRMS** (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{13}\text{O}_2^+$, 213.0910; found, 213.0921. **CHN analysis** Calcd for $\text{C}_{14}\text{H}_{12}\text{O}_2$: C, 78.22; H, 5.70. Found: C, 78.5; H, 5.68.

2. 4-Methylbenzophenone



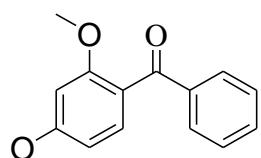
White solid, **M.p.** 56–57 °C. **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ 7.80 (d, $J = 8.2$ Hz, 2H), 7.74 (d, $J = 8.2$ Hz, 2H), 7.59 (t, $J = 6.9$ Hz, 1H), 7.49 (t, $J = 7.3$ Hz, 2H), 7.30 (d, $J = 7.8$ Hz, 2H), 2.46 (s, 3H). **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ 196.5, 143.3, 137.9, 134.8, 132.2, 130.3, 129.9, 129.0, 128.2, 21.7. **HRMS** (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{13}\text{O}^+$, 197.0961; found, 197.0960. **CHN analysis** Calcd for $\text{C}_{14}\text{H}_{12}\text{O}$: C, 85.68; H, 6.16. Found: C, 85.65; H, 6.12.

3. 3,4-Dimethoxybenzophenone



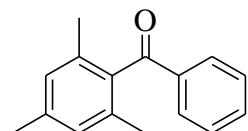
White crystalline solid; **M.p.** 89–93 °C. **¹H NMR** (400 MHz, CDCl₃): δ = 7.76 (d, *J* = 7.3 Hz, 2H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.45 – 7.50 (m, 3H), 7.38 (d, *J* = 8.7 Hz, 1H), 6.89 (d, *J* = 8.3 Hz, 1H), 3.96 (s, 3H), 3.94 (s, 3H). **¹³C NMR** (100 MHz, CDCl₃) δ = 195.5, 153.0, 149.0, 138.2, 131.8, 130.2, 129.7, 128.1, 125.5, 112.1, 109.7, 56.1, 56.0. **HRMS** (m/z): [M+H]⁺ calcd for C₁₅H₁₅O₃⁺, 243.1016; found, 243.1018. **CHN analysis** Calcd for C₁₅H₁₄O₃: C, 74.36; H, 5.82. Found: C, 74.41; H, 5.82.

4. 2,4-Dimethoxybenzophenone



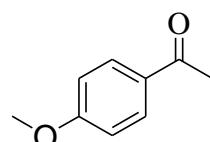
Colorless liquid. **¹H NMR** (400 MHz, CDCl₃): δ = 7.81 (m, 2H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.42 (m, 3H), 6.59 (m, 2H), 3.87 (s, 3H), 3.70 (s, 3H). **¹³C NMR** (100 MHz, CDCl₃) δ = 195.6, 163.3, 159.6, 138.8, 132.3, 129.6, 129.4, 127.9, 121.5, 104.5, 98.7, 55.5, 55.4. **HRMS** (m/z): [M+H]⁺ calcd for C₁₅H₁₅O₃⁺, 243.1016; found, 243.1016. **CHN analysis** Calcd for C₁₅H₁₄O₃: C, 74.36; H, 5.82. Found: C, 74.35; H, 5.82.

5. 2,4,6-Trimethylbenzophenone



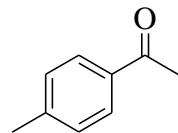
Colorless liquid. **¹H NMR** (400 MHz, CDCl₃): δ = 7.82 (d, *J* = 7.3 Hz, 2H), 7.59 (t, *J* = 7.3 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 2H), 6.91 (s, 2H), 2.35 (s, 3H), 2.10 (s, 6H). **¹³C NMR** (100 MHz, CDCl₃) δ = 200.8, 138.5, 137.3, 136.8, 134.2, 133.5, 129.4, 128.8, 128.3, 21.1, 19.3. **HRMS** (m/z): [M+H]⁺ calcd for C₁₆H₁₇O⁺, 225.1274; found 225.1270. **CHN analysis** Calcd for C₁₅H₁₄O₃: C, 85.76; H, 7.19. Found: C, 85.76; H, 7.19.

6. 4-Methoxyacetophenone



White solid, **M.p.** 36–37 °C. **¹H NMR** (400 MHz, CDCl₃): δ 7.93 (d, *J* = 8.7 Hz, 2H), 6.93 (d, *J* = 8.7 Hz, 2H), 3.86 (s, 3H), 2.55 (s, 3H). **¹³C NMR** (100 MHz, CDCl₃): δ 197.1, 163.5, 130.6, 130.2, 113.6, 55.4, 26.2. **HRMS** (m/z): [M+H]⁺ calcd for C₉H₁₁O₂⁺, 151.0754; found, 151.0752. **CHN analysis** Calcd for C₉H₁₀O: C, 71.98; H, 6.71. Found: C, 71.97; H, 6.70.

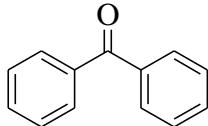
7. 4-Methylacetophenone



Colorless oil. **¹H NMR** (400 MHz, CDCl₃): δ 7.87 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 2.59 (s, 3H), 2.43 (s, 3H). **¹³C NMR** (100 MHz, CDCl₃): δ 197.7, 143.8, 134.7, 129.2, 128.4, 26.5, 21.6. **HRMS** (m/z):

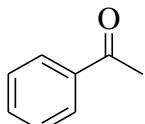
$[M+H]^+$ calcd for $C_9H_{11}O^+$, 135.0804; found, 135.0801. **CHN analysis** Calcd for $C_9H_{10}O$: C, 80.56; H, 7.51. Found: C, 80.59; H, 7.48.

8. Benzophenone



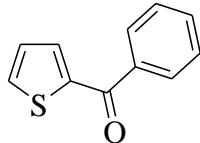
White solid. **M.p.** 47–48 °C. **1H NMR** (400 MHz, $CDCl_3$): δ 7.82 (d, J = 7.3 Hz, 4H), 7.60 (t, J = 7.8 Hz, 2H), 7.50 (t, J = 7.3 Hz, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 196.9, 137.7, 132.5, 130.1, 128.3. **HRMS** (m/z): $[M+H]^+$ calcd for $C_{13}H_{11}O^+$, 183.0804; found, 183.0804. **CHN analysis** Calcd for $C_{13}H_{10}O$: C, 85.69; H, 5.53. Found: C, 85.67; H, 5.53.

9. Acetophenone



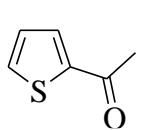
Colorless liquid. **1H NMR** (400 MHz, $CDCl_3$): δ 7.92 (d, J = 6.9 Hz, 2H), 7.52 (t, J = 7.3 Hz, 1H), 7.42 (t, J = 7.8 Hz, 2H), 2.56 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.9, 136.9, 132.9, 128.4, 128.1, 26.4. **HRMS** (m/z): $[M+H]^+$ calcd for $C_8H_9O^+$, 121.0648; found, 121.0635. **CHN analysis** Calcd for C_8H_8O : C, 79.97; H, 6.71. Found: C, 79.95; H, 6.70.

10. 2-Benzoylthiophene



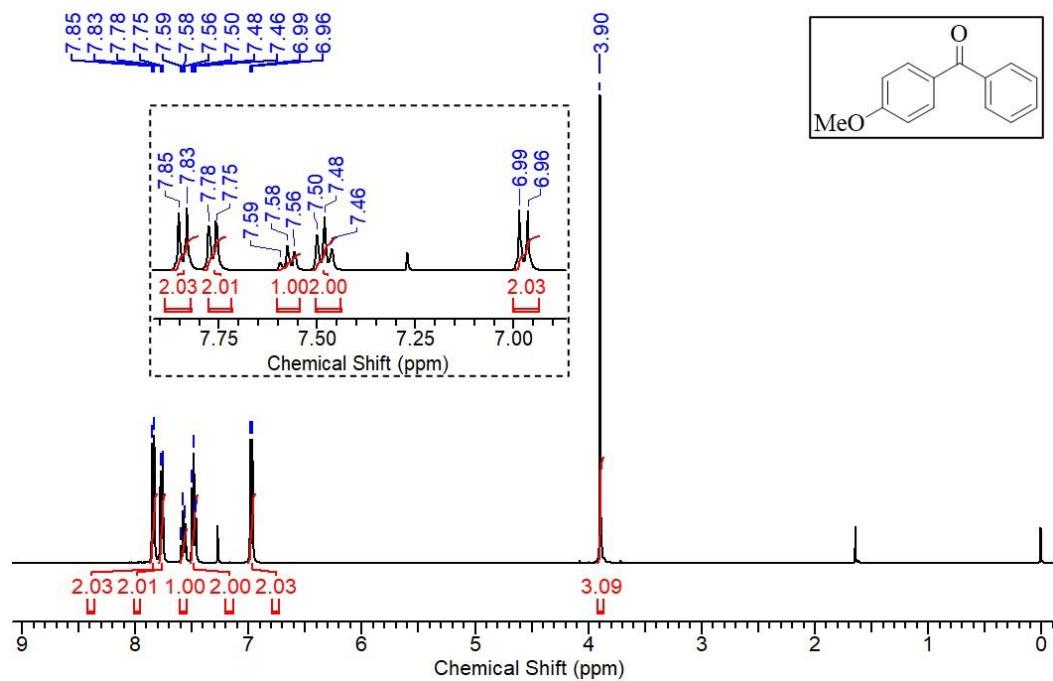
White solid. **M.p.** 54–55 °C. **1H NMR** (400 MHz, $CDCl_3$): δ 7.88 (d, J = 6.9 Hz, 2H), 7.73 (d, J = 4.6 Hz, 1H), 7.66–7.65 (m, 1H), 7.60 (t, J = 7.3 Hz, 1H), 7.50 (t, J = 7.8 Hz, 2H), 7.18–7.16 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 188.2, 143.6, 138.1, 134.7, 134.1, 132.2, 129.1, 128.3, 127.9. **HRMS** (m/z): $[M+H]^+$ calcd for $C_{11}H_9OS^+$, 189.0369; found, 189.0371. **CHN analysis** Calcd for $C_{11}H_8OS$: C, 70.18; H, 4.28. Found: C, 70.20; H, 4.13.

11. 2-Acetylthiophene

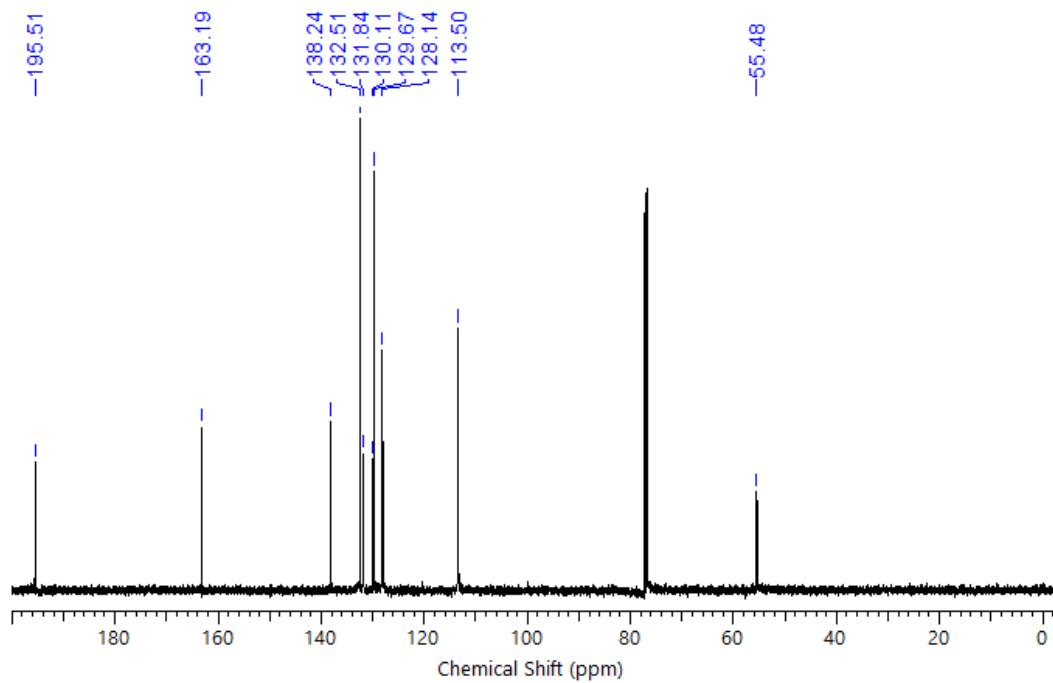


Colorless oil. **1H NMR** (400 MHz, $CDCl_3$): δ 7.69 (d, J = 3.2 Hz, 1H), 7.62 (d, J = 5.0 Hz, 1H), 7.12 (t, J = 4.1 Hz, 1H), 2.55 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 190.6, 144.5, 133.7, 132.4, 128.0, 26.8. **HRMS** (m/z): $[M+H]^+$ calcd for $C_6H_7OS^+$, 127.0212; found, 127.0218. **CHN analysis** Calcd for C_6H_6OS : C, 57.11; H, 4.79. Found: C, 57.01; H, 4.62.

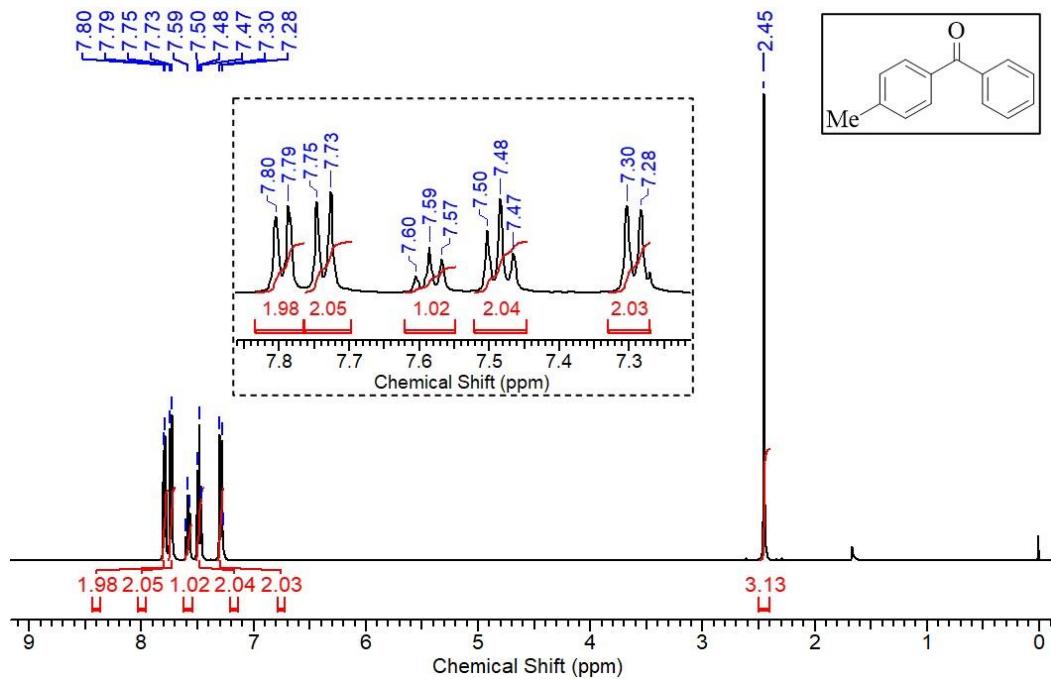
SPECTRAL COPIES OF ^1H AND ^{13}C NMR OF PRODUCTS



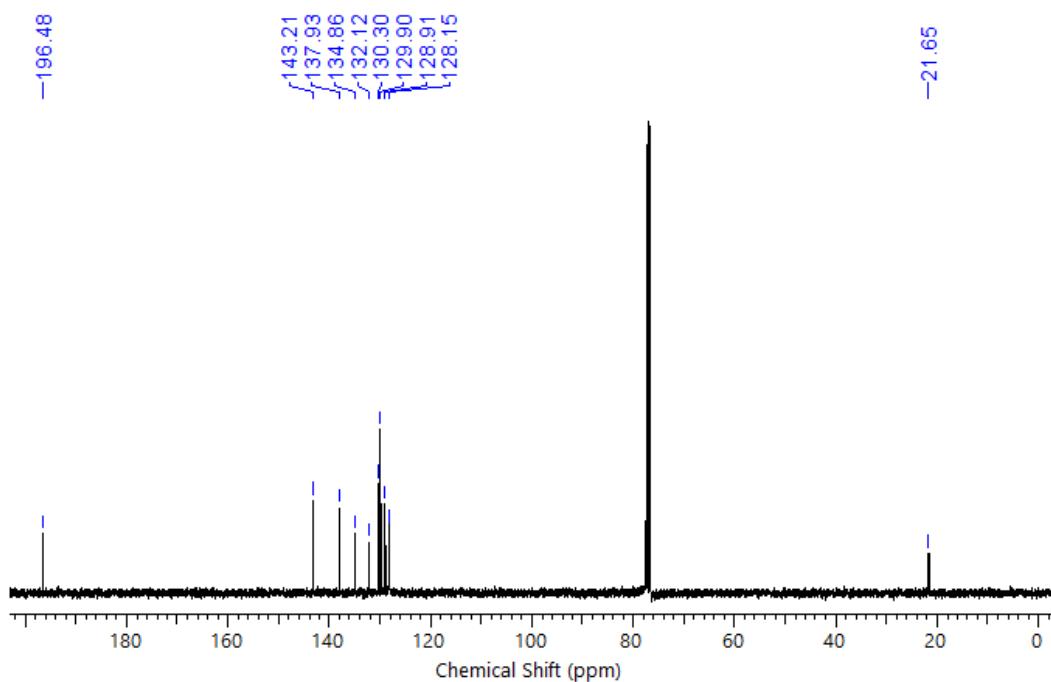
^1H NMR spectrum of 4-Methoxybenzophenone



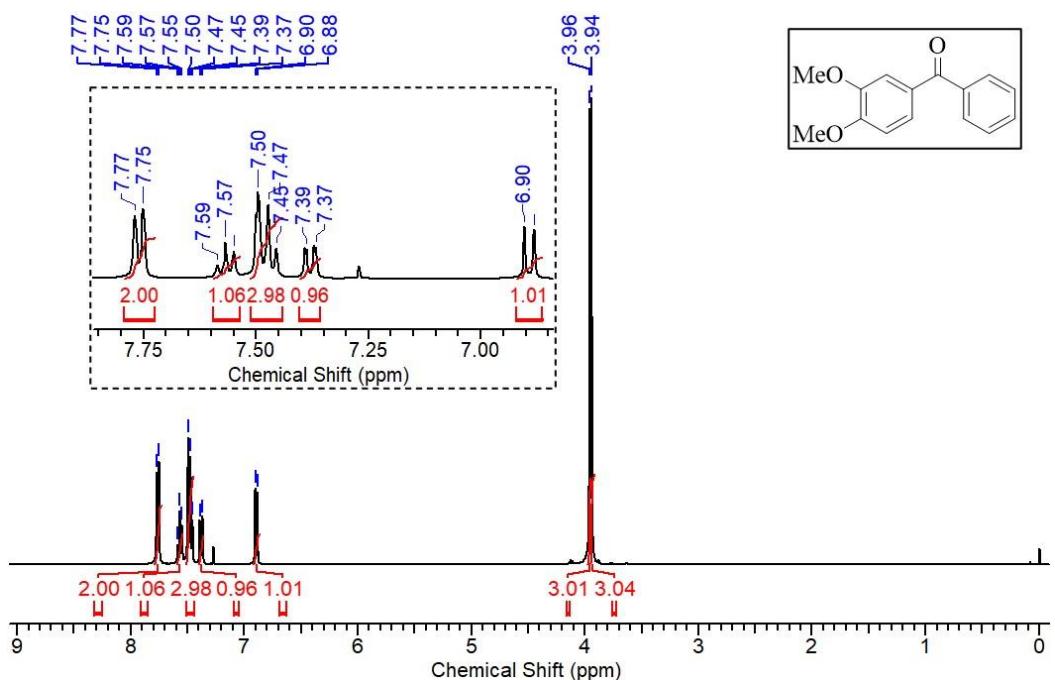
^{13}C NMR spectrum of 4-Methoxybenzophenone



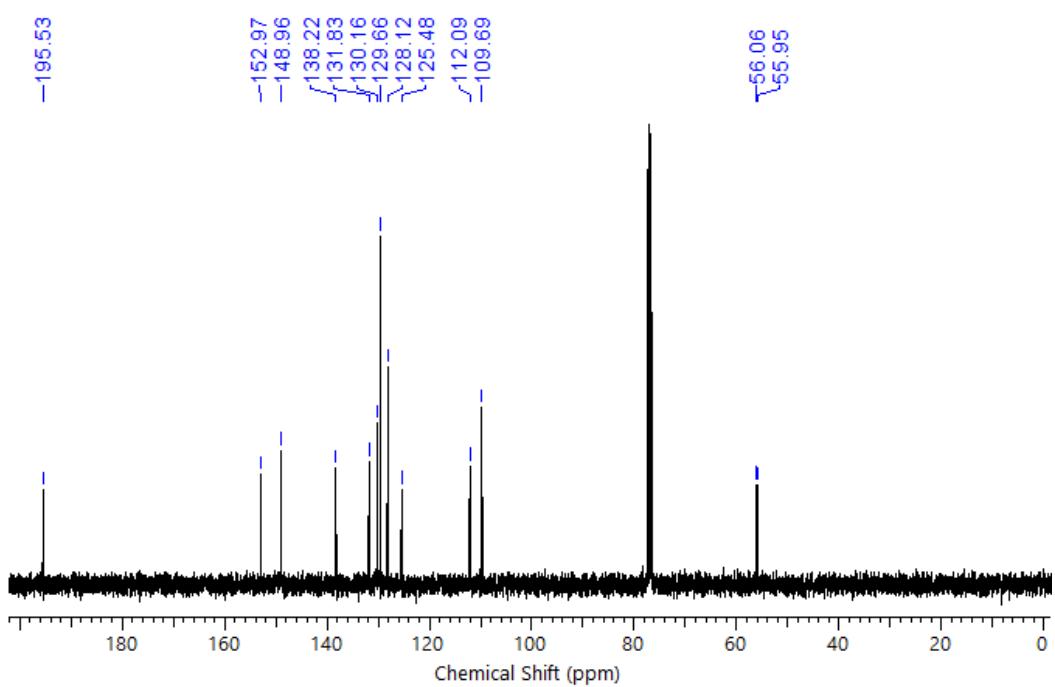
¹H NMR spectrum of 4-Methylbenzophenone



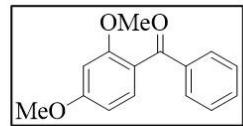
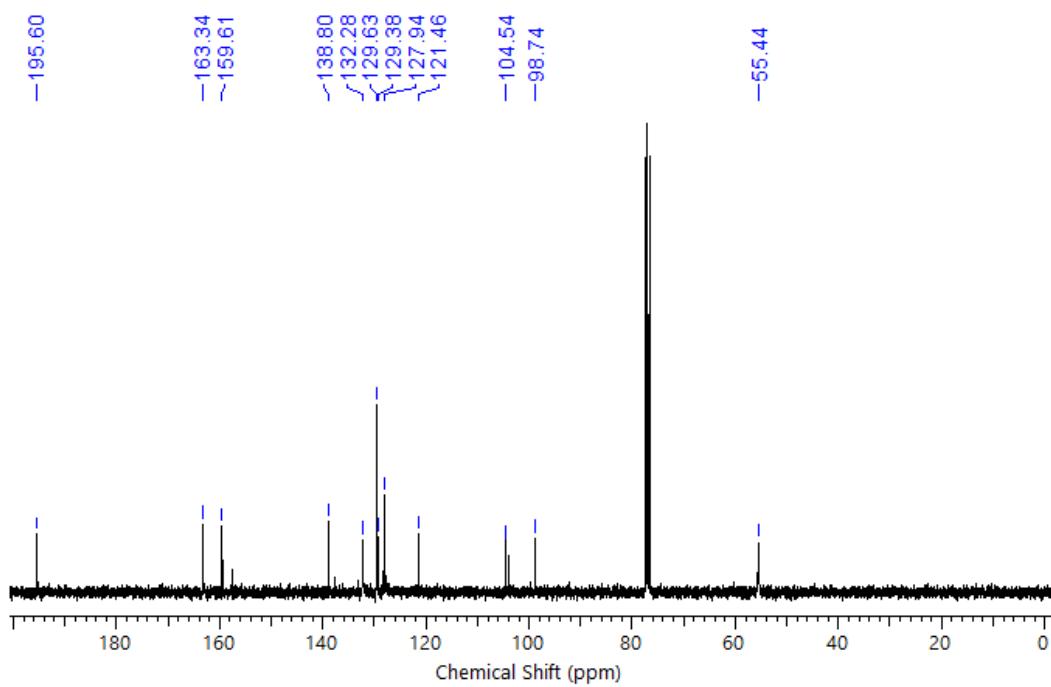
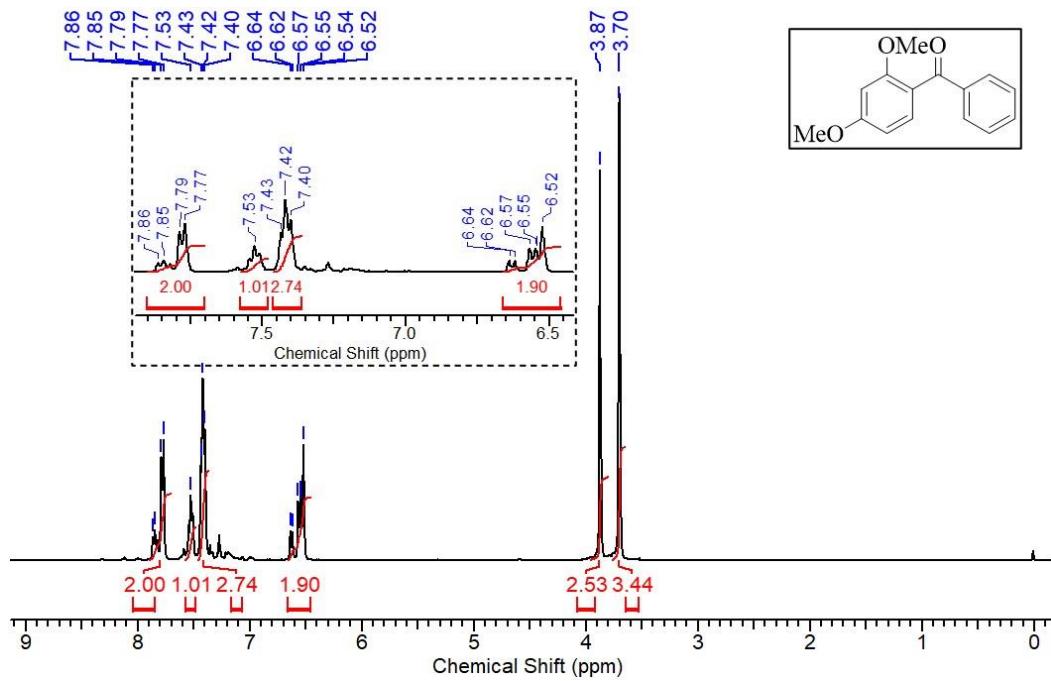
¹³C NMR spectrum of 4-Methylbenzophenone

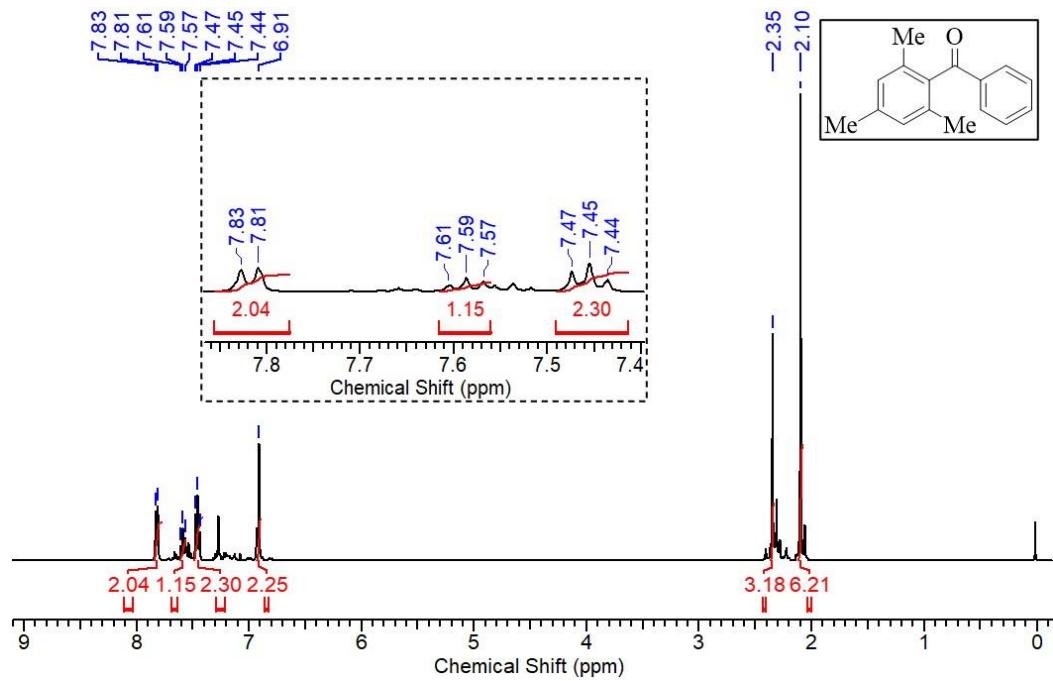
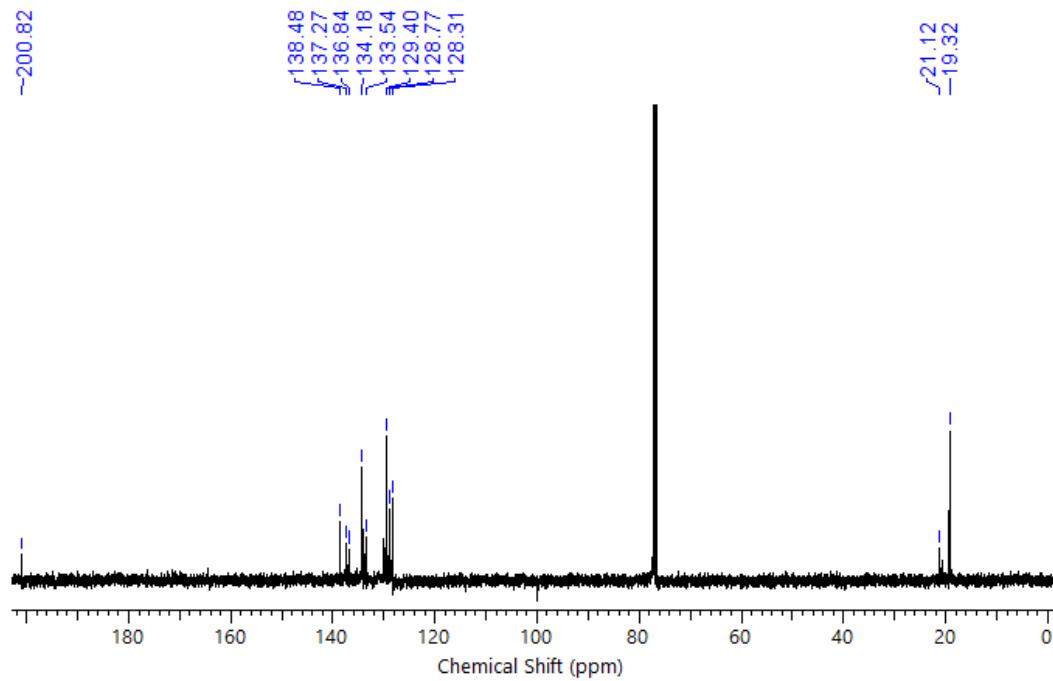


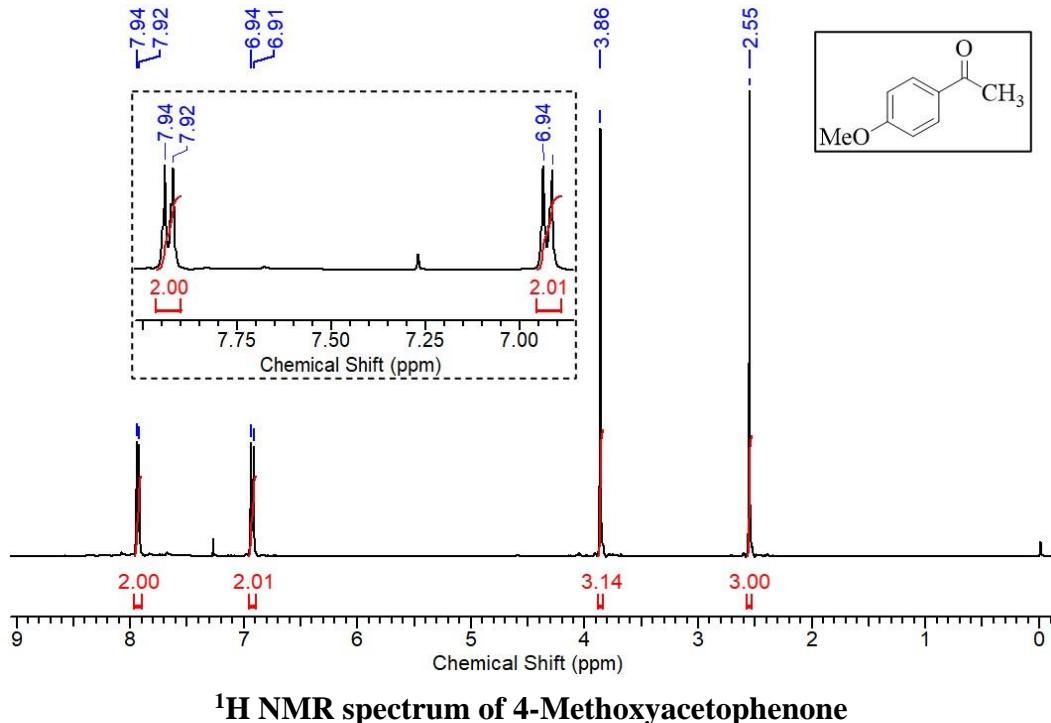
¹H NMR spectrum of 3,4-Dimethoxybenzophenone



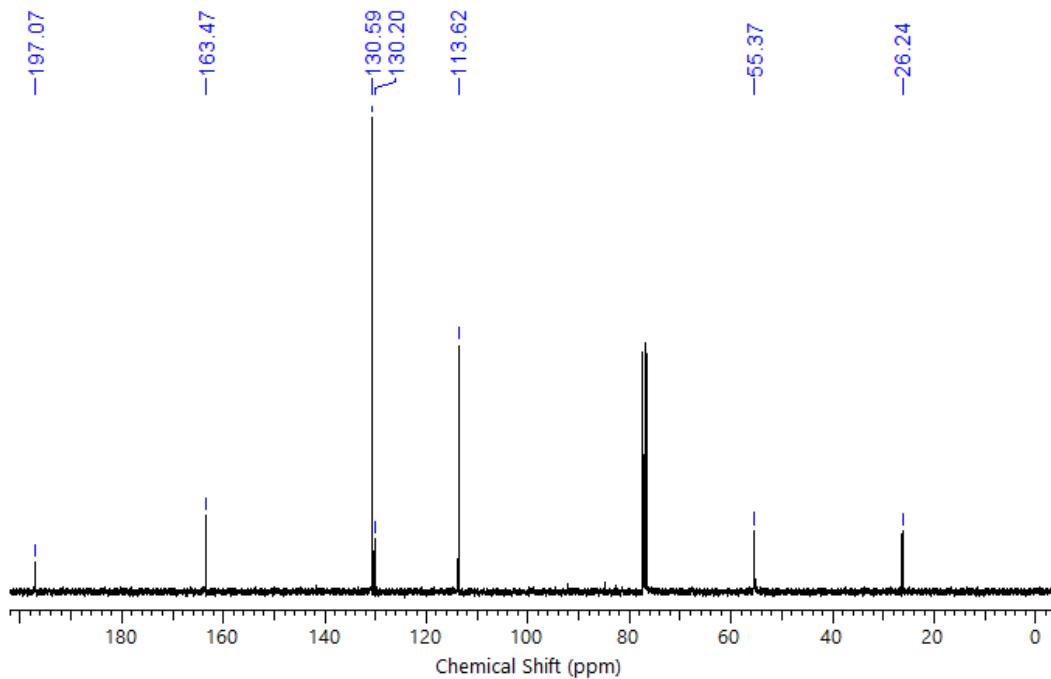
¹³C NMR spectrum of 3,4-Dimethoxybenzophenone



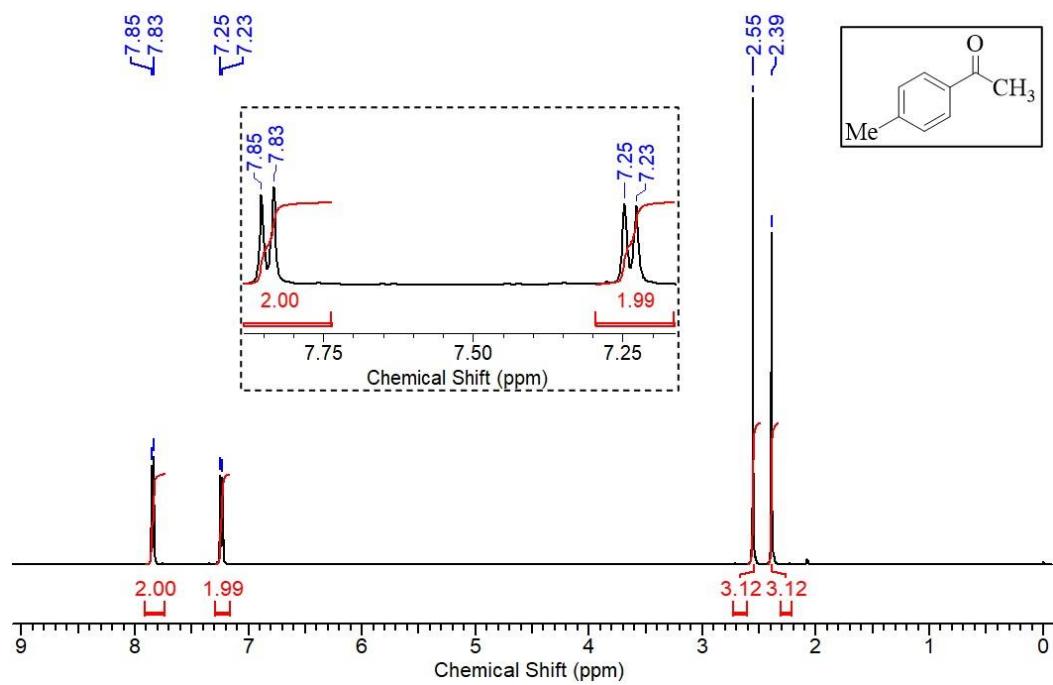
 ^1H NMR spectrum of 2,4,6-Trimethylbenzophenone ^{13}C NMR spectrum of 2,4,6-Trimethylbenzophenone



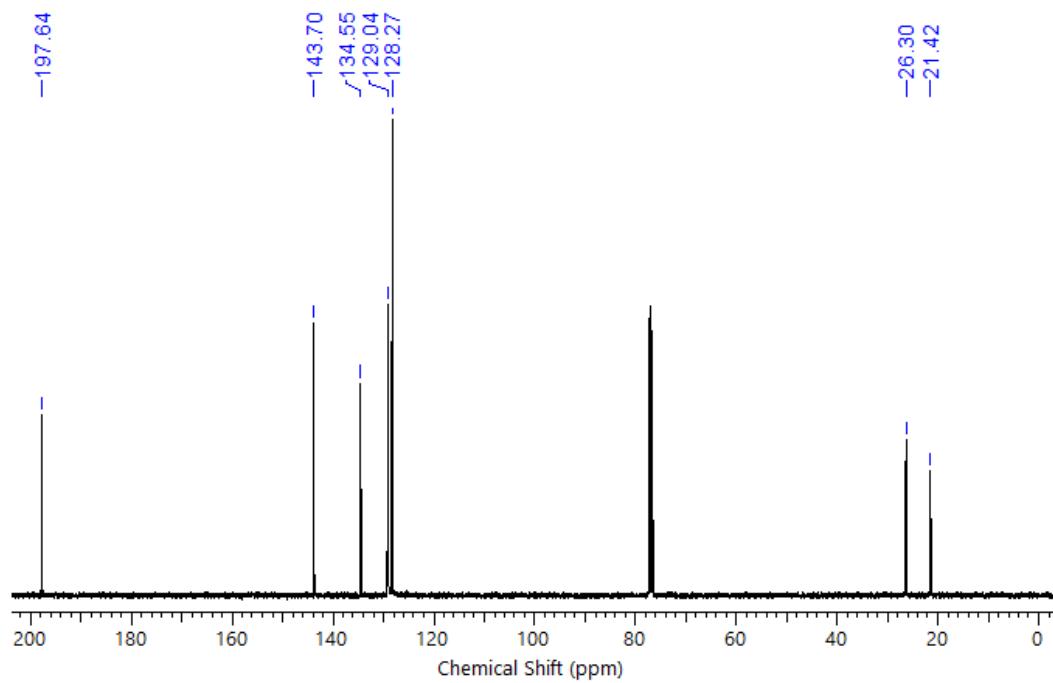
¹H NMR spectrum of 4-Methoxyacetophenone



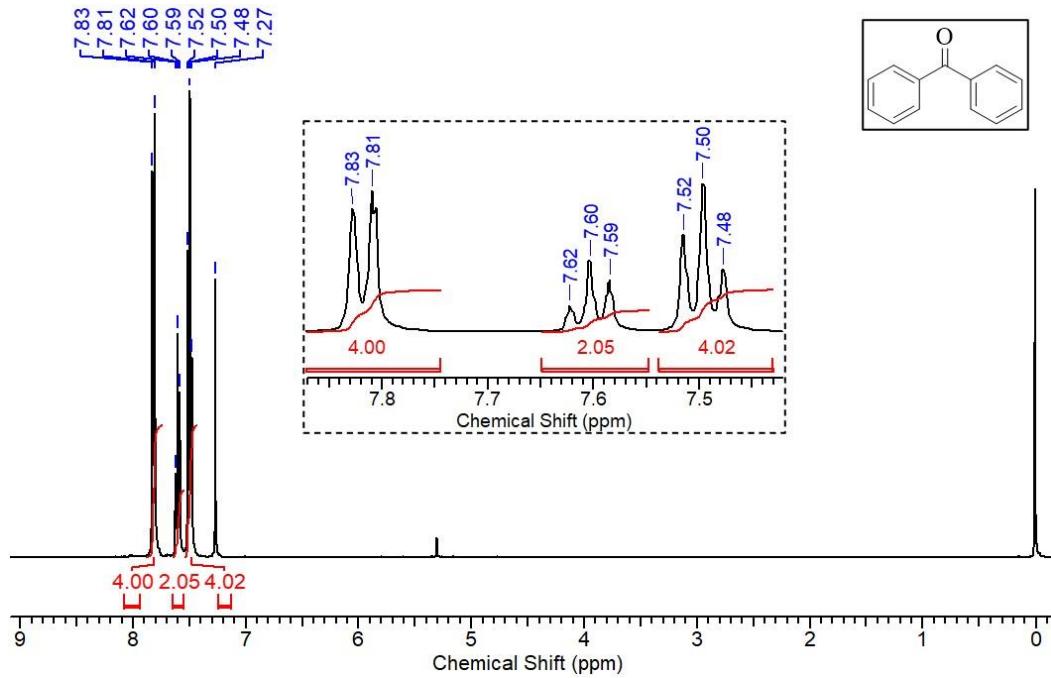
¹³C NMR spectrum of 4-Methoxyacetophenone



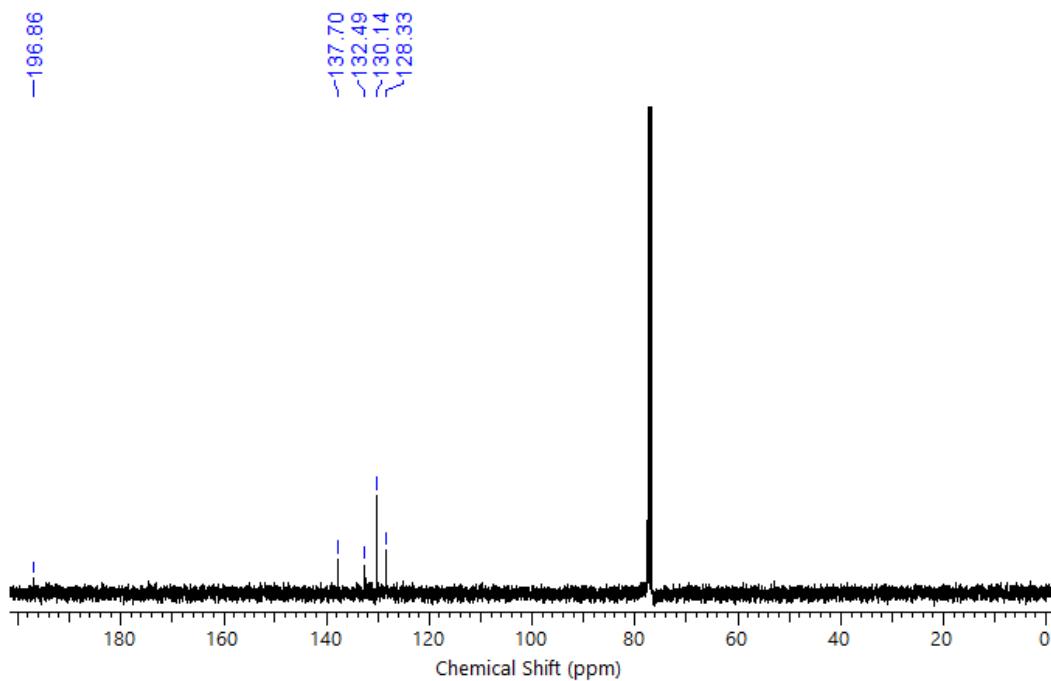
¹H NMR spectrum of 4-Methylacetophenone



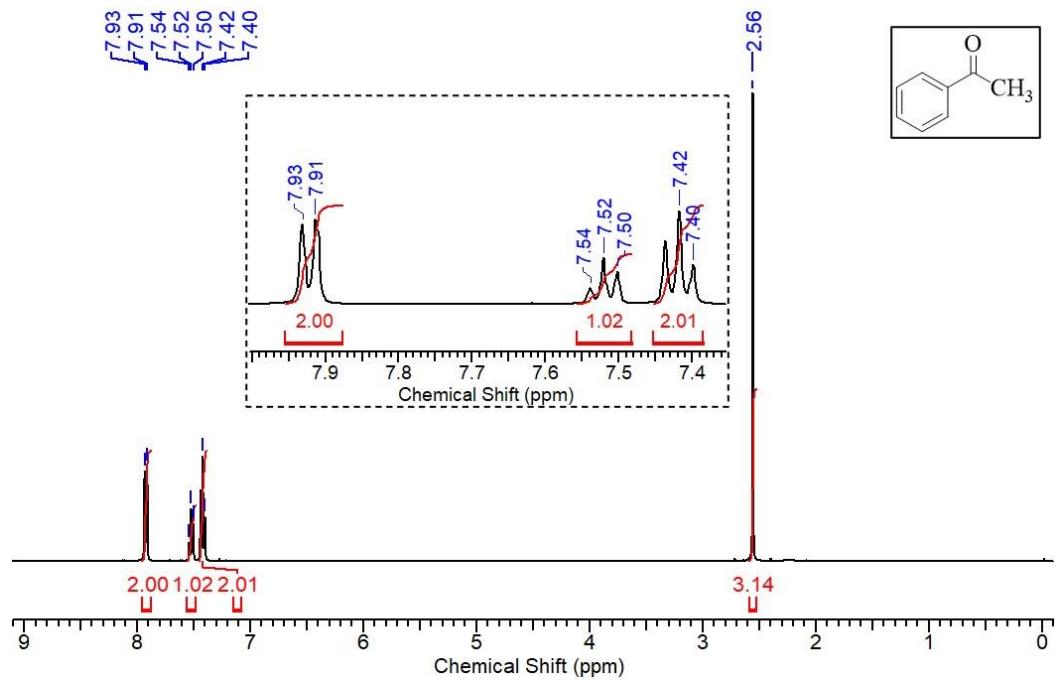
¹³C NMR spectrum of 4-Methylacetophenone



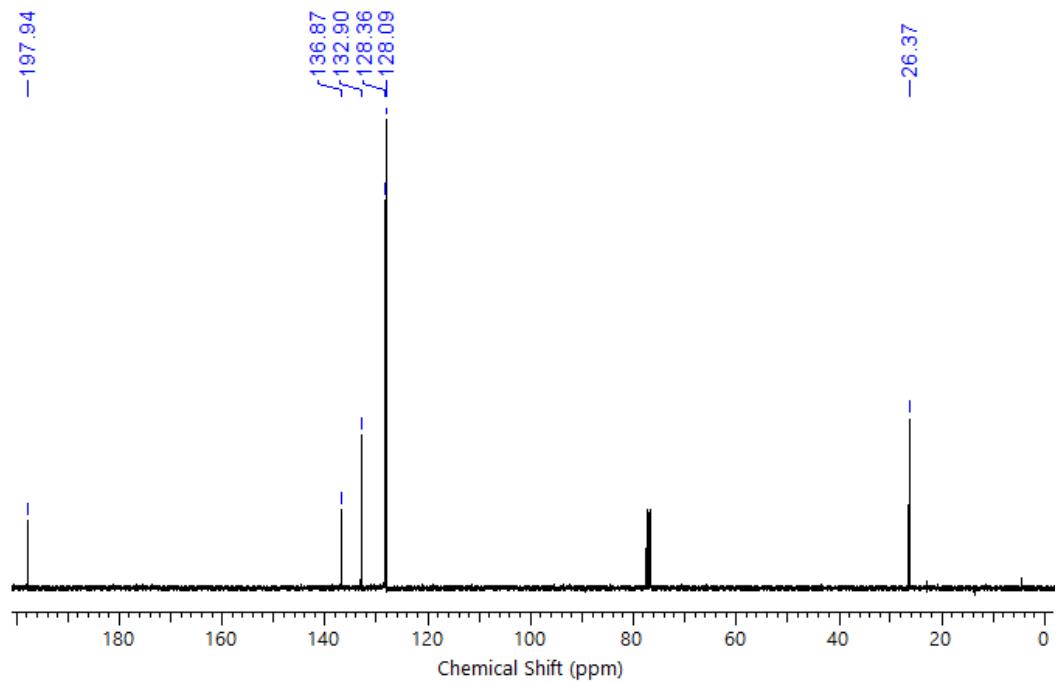
¹H NMR spectrum of Benzophenone



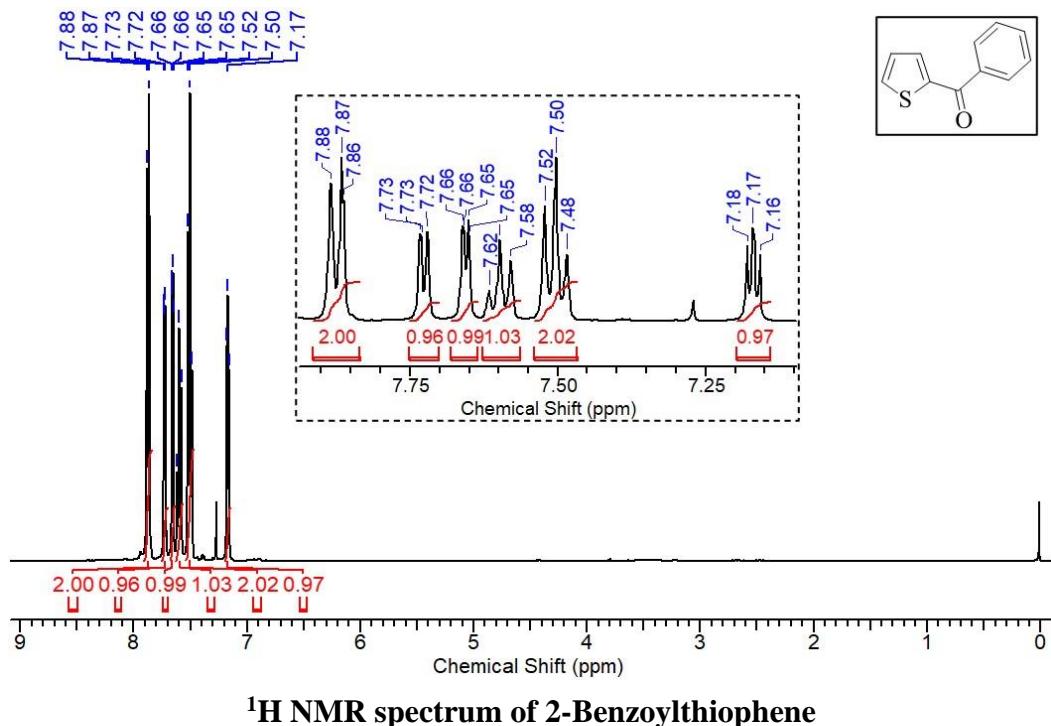
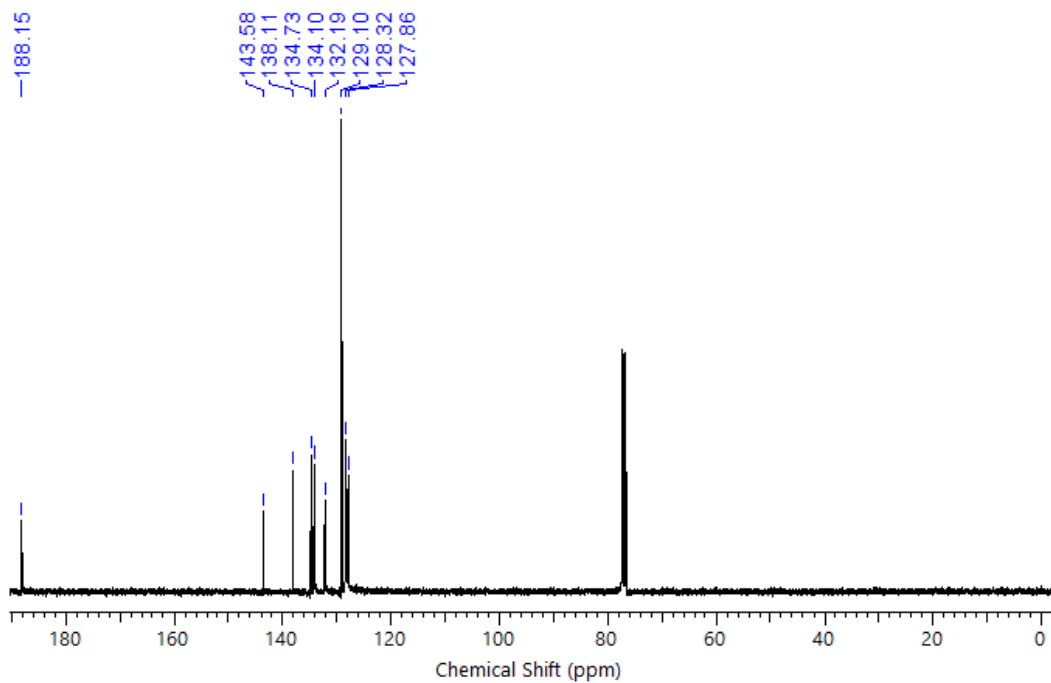
¹³C NMR spectrum of Benzophenone

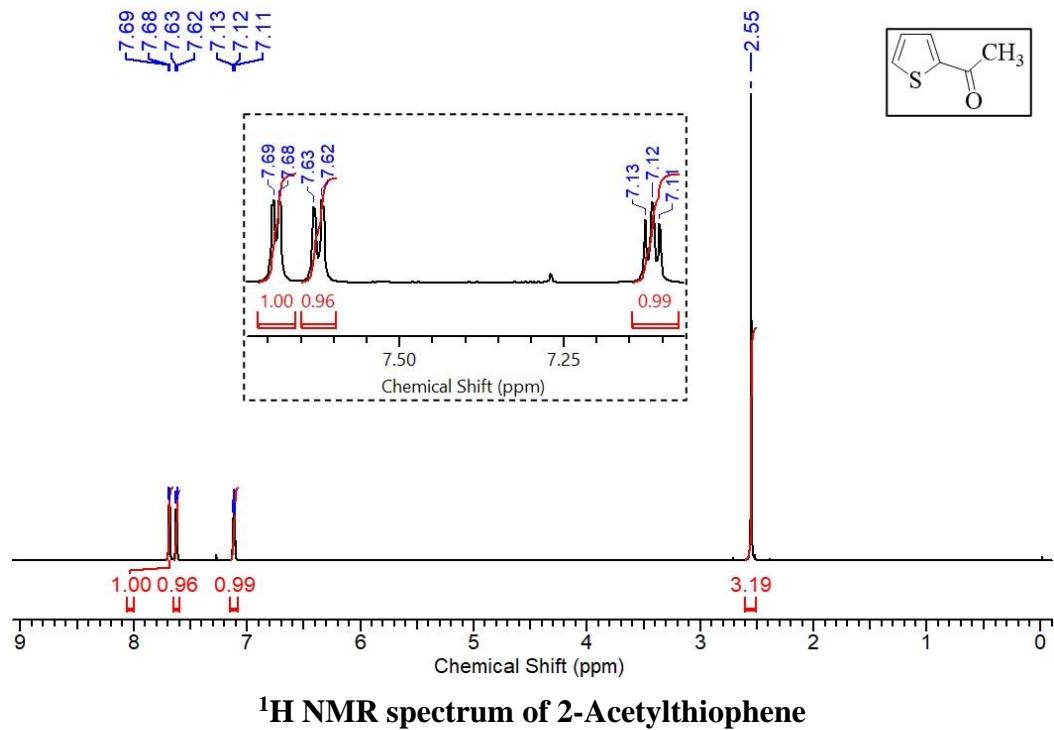


¹H NMR spectrum of Acetophenone

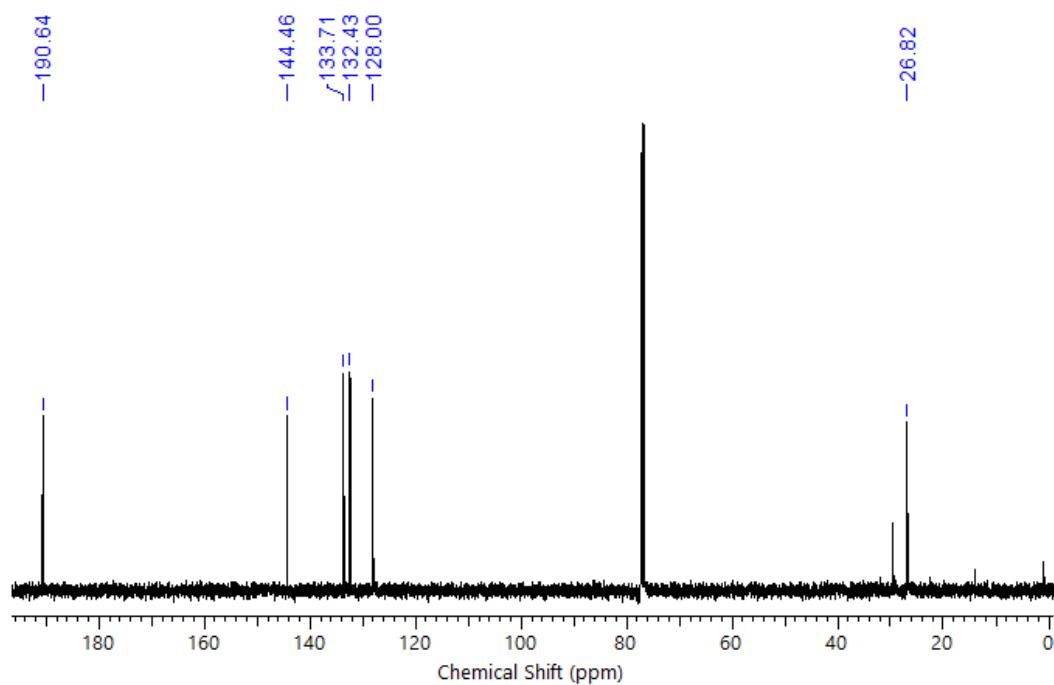


¹³C NMR spectrum of Acetophenone

¹H NMR spectrum of 2-Benzoylthiophene¹³C NMR spectrum of 2-Benzoylthiophene



¹H NMR spectrum of 2-Acetylthiophene



¹³C NMR spectrum of 2-Acetylthiophene