BEILSTEIN JOURNAL OF ORGANIC CHEMISTRY

Supporting Information

for

Rhodium-catalyzed reductive carbonylation of aryl iodides to arylaldehydes with syngas

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MS spectra of isotope-labeling experiments and characterization of products

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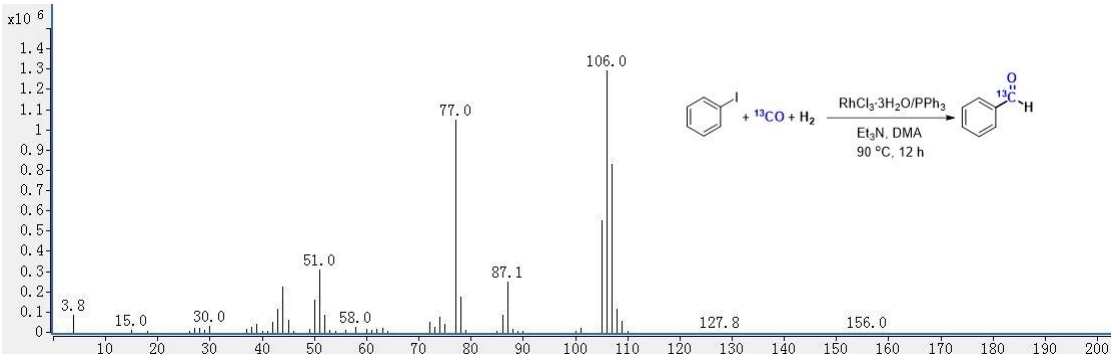
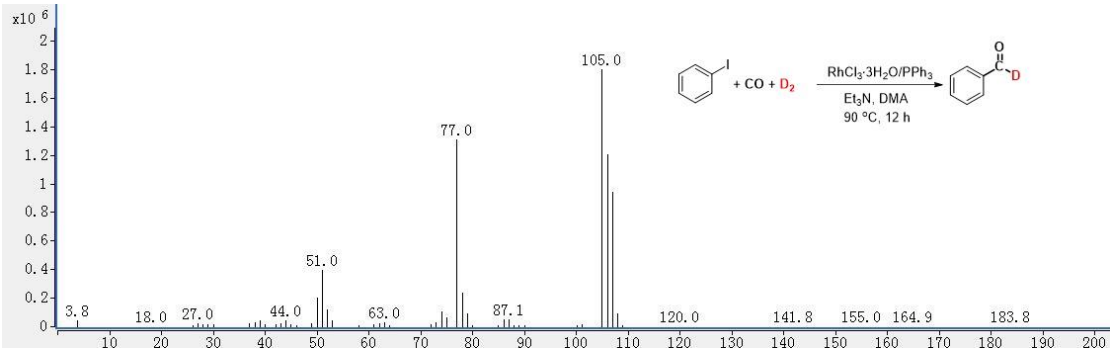
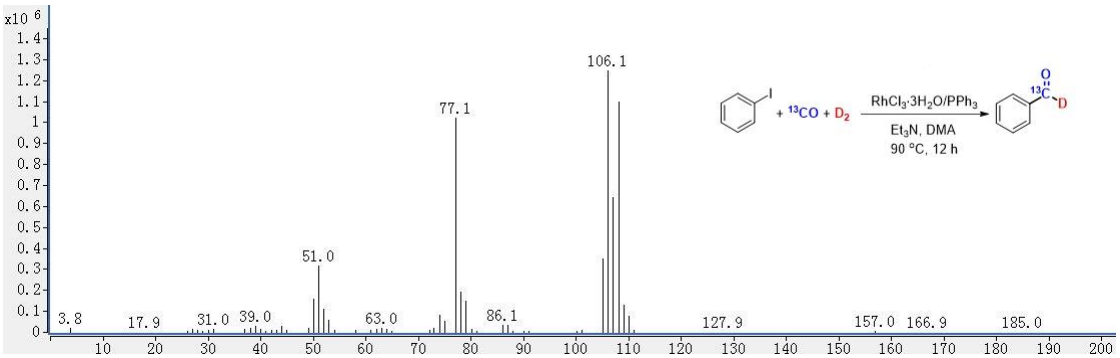
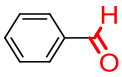
1. MS spectra of ^{13}CO and D_2 in the isotope-labeling experiments

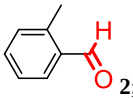


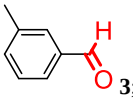


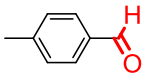


2. NMR data of the aromatic aldehyde products

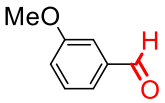
 **1**; Benzaldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.02 (s, 1H), 7.94 – 7.81 (m, 2H), 7.69 – 7.40 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.35, 136.34, 134.41, 129.68, 128.94.

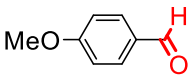
 **2**; 2-Methylbenzaldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.26 (s, 1H), 7.79 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.47 (td, *J* = 7.5, 1.6 Hz, 1H), 7.36 (td, *J* = 7.5, 1.2 Hz, 1H), 7.28 – 7.22 (m, 1H), 2.67 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.73, 140.53, 134.07, 133.58, 131.98, 131.70, 126.25, 19.52.

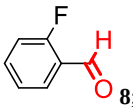
 **3**; 3-Methylbenzaldehyde [2]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.97 (s, 1H), 7.74 – 7.61 (m, 2H), 7.47 – 7.33 (m, 2H), 2.42 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.53, 138.83, 136.39, 135.21, 129.94, 128.80, 127.14, 21.11.

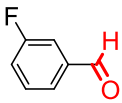
 **4**; 4-Methylbenzaldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (s, 1H), 7.77 (d, *J* = 8.1 Hz, 2H), 7.33 (d, *J* = 7.8 Hz, 2H), 2.44 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.94, 145.49, 134.13, 129.71, 21.83.

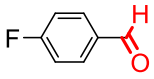
 **5**; 2-Methoxybenzaldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.45 (s, 1H), 7.80 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.52 (ddd, *J* = 8.2, 7.4, 1.8 Hz, 1H), 7.08 – 6.90 (m, 2H), 3.90 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.62, 161.75, 135.83, 128.38, 124.80, 120.55, 111.58, 55.53.

 **6**; 3-Methoxybenzaldehyde [3]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.95 (s, 1H), 7.50 – 7.32 (m, 3H), 7.16 (dt, *J* = 5.9, 2.9 Hz, 1H), 3.84 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.04, 160.05, 137.70, 129.94, 123.43, 121.40, 111.96, 55.36.

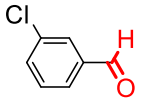
 **7**; 4-Methoxybenzaldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.83 (s, 1H), 8.17 – 7.55 (m, 2H), 6.95 (d, *J* = 8.7 Hz, 2H), 3.83 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.61, 164.44, 131.79, 129.77, 114.15, 55.41.

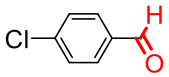
 **8**; 2-Fluorobenzaldehyde [2]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.37 (d, *J* = 0.8 Hz, 1H), 7.88 (td, *J* = 7.4, 1.9 Hz, 1H), 7.61 (dddd, *J* = 8.4, 7.3, 5.4, 1.9 Hz, 1H), 7.33 – 7.09 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 187.12, 166.34, 162.92, 136.29, 128.64, 124.58, 116.45.

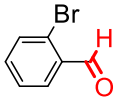
 **9**; 3-Fluorobenzaldehyde [4]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.00 (d, *J* = 1.9 Hz, 1H), 7.69 (dt, *J* = 7.5, 1.3 Hz, 1H), 7.61 – 7.48 (m, 2H), 7.39 – 7.26 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.82, 164.69, 161.38, 138.36, 130.74, 126.00, 121.52, 115.27.

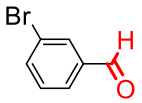
 **10**; 4-Fluorobenzaldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 1H), 7.92 (dd, *J* = 8.7, 5.4 Hz, 2H), 7.22 (t, *J* = 8.5 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.45, 168.16, 164.76, 132.91, 132.18, 116.30.

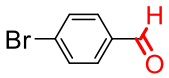
 **11**; 2-Chlorobenzaldehyde [4]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.47 (d, *J* = 0.8 Hz, 1H), 7.91 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.53 (ddd, *J* = 8.0, 7.1, 1.8 Hz, 1H), 7.48 – 7.32 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.67, 137.81, 135.03, 132.32, 130.49, 129.24, 127.18.

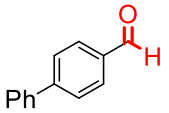
 **12**; 3-Chlorobenzaldehyde [2]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.94 (s, 1H), 7.81 (dd, *J* = 2.1, 1.5 Hz, 1H), 7.73 (dt, *J* = 7.5, 1.4 Hz, 1H), 7.56 (ddd, *J* = 8.0, 2.1, 1.2 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.81, 137.78, 135.40, 134.35, 130.38, 129.22, 127.98.

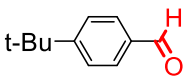
 **13**; 4-Chlorobenzaldehyde [1]; Colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 1H), 7.82 (d, *J* = 8.5 Hz, 2H), 7.51 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.75, 140.81, 134.61, 130.80, 129.34.

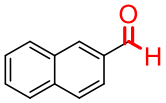
 **14**; 2-Bromobenzaldehyde [4]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.29 (d, *J* = 0.7 Hz, 1H), 7.87 – 7.80 (m, 1H), 7.60 – 7.52 (m, 1H), 7.43 – 7.32 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.55, 135.14, 133.67, 133.25, 129.63, 127.70, 126.88.

 **15**; 3-Bromobenzaldehyde [5]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.92 (s, 1H), 7.95 (d, *J* = 1.8 Hz, 1H), 7.77 (dt, *J* = 7.6, 1.3 Hz, 1H), 7.73 – 7.65 (m, 1H), 7.39 (t, *J* = 7.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.52, 137.78, 137.06, 132.05, 130.46, 128.22, 123.15.

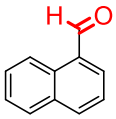
 **16**; 4-Bromobenzaldehyde [3]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (s, 1H), 7.73 (d, *J* = 8.5 Hz, 2H), 7.66 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.98, 135.01, 132.37, 130.90, 129.70.

 **17**; 4-Biphenylcarboxaldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.06 (s, 1H), 7.96 (d, *J* = 8.3 Hz, 2H), 7.76 (d, *J* = 8.3 Hz, 2H), 7.68 – 7.60 (m, 2H), 7.53 – 7.36 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.84, 147.13, 139.65, 135.15, 130.21, 128.96, 128.42, 127.63, 127.31.

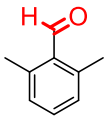
 **18**; 4-*tert*-Butylbenzaldehyde [6]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 1H), 7.82 (d, *J* = 8.4 Hz, 2H), 7.55 (d, *J* = 8.4 Hz, 2H), 1.35 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.02, 158.41, 134.03, 129.66, 125.95, 35.32, 31.03.



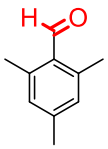
19; 2-Naphthaldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.16 (s, 1H), 8.34 (s, 1H), 8.05 – 7.84 (m, 4H), 7.62 (dddd, *J* = 17.6, 8.2, 6.9, 1.4 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.23, 136.42, 134.5, 134.08, 132.61, 129.50, 129.08, 128.05, 127.07, 122.74.



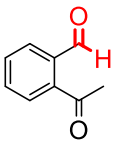
20; 1-Naphthaldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.38 (s, 1H), 9.26 (dd, *J* = 8.6, 1.1 Hz, 1H), 8.08 (dt, *J* = 8.2, 1.0 Hz, 1H), 8.01 – 7.83 (m, 2H), 7.79 – 7.44 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.45, 136.60, 135.19, 133.60, 131.27, 130.41, 128.97, 128.38, 126.86, 124.77.



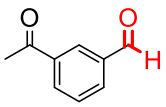
21; 2,6-Dimethylbenzaldehyde [2]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.62 (s, 1H), 7.32 (t, *J* = 7.6 Hz, 1H), 7.09 (d, *J* = 7.6 Hz, 2H), 2.61 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.39, 140.99, 132.85, 132.30, 129.58, 20.34.



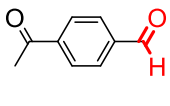
22; Mesitaldehyde [7]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.53 (s, 1H), 6.87 (s, 2H), 2.56 (s, 6H), 2.30 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.76, 143.66, 141.31, 130.38, 129.82, 21.31, 20.33.



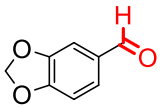
23; 2-Formylacetophenone; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.22 (s, 1H), 7.89 – 7.83 (m, 1H), 7.68 (ddd, J = 22.9, 6.0, 2.9 Hz, 3H), 2.65 (d, J = 1.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.91, 192.12, 140.55, 136.17, 132.95, 131.77, 129.60, 128.40, 28.76.



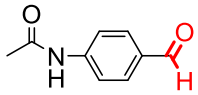
24; 3-Formylacetophenone; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.04 (s, 1H), 8.38 (t, J = 1.8 Hz, 1H), 8.17 (dt, J = 7.8, 1.5 Hz, 1H), 8.04 (dt, J = 7.6, 1.4 Hz, 1H), 7.61 (t, J = 7.7 Hz, 1H), 2.62 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.76, 191.30, 137.68, 136.54, 133.56, 133.43, 129.38, 129.36, 26.53.



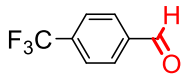
25; 4-Formylacetophenone [6]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.10 (s, 1H), 8.10 (d, *J* = 8.3 Hz, 2H), 7.97 (d, *J* = 8.0 Hz, 2H), 2.66 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.20, 191.42, 140.97, 138.81, 129.57, 128.59, 26.74.



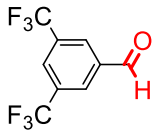
26; Piperonyl aldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.30 (s, 1H), 7.16 (dd, *J* = 8.1, 1.7 Hz, 1H), 7.14 (d, *J* = 1.7 Hz, 1H), 6.62 (d, *J* = 8.1 Hz, 1H), 5.99 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 186.81, 152.20, 151.48, 146.02, 128.14, 107.47, 105.06, 103.89.



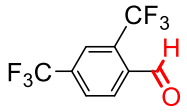
27; 4-Acetamidobenzaldehyde [8]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.91 (s, 1H), 8.00 (s, 1H), 7.89 – 7.79 (m, 2H), 7.71 (d, *J* = 8.4 Hz, 2H), 2.23 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.14, 168.86, 143.62, 132.14, 131.14, 119.22, 24.76.



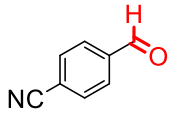
28; 4-(Trifluoromethyl)benzaldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.09 (s, 1H), 8.00 (dt, *J* = 7.9, 0.9 Hz, 2H), 7.80 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.07, 138.63, 135.79, 135.36, 129.89, 126.11, 121.60, 117.99.



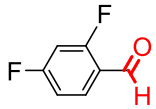
29; 3,5-Bis(trifluoromethyl)benzaldehyde [9]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.13 (s, 1H), 8.34 (d, *J* = 1.8 Hz, 2H), 8.12 (d, *J* = 2.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.06, 137.52, 132.78, 129.38, 127.49, 124.10.



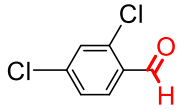
30; 2,4-Bis(trifluoromethyl)benzaldehyde; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.04 (d, J = 0.5 Hz, 1H), 8.06 (d, J = 1.5 Hz, 1H), 7.90 (dd, J = 7.5, 0.5 Hz, 1H), 7.72 (dd, J = 7.5, 1.5 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.42, 134.81, 134.80, 134.74, 133.42, 133.17, 129.59, 129.57, 129.56, 129.54, 126.95, 126.92, 126.88, 126.85, 125.22, 125.19, 125.16, 125.13, 124.91, 123.66, 122.76.



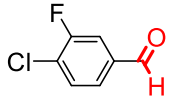
31; 4-Cyanobenzaldehyde [8]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.08 (s, 1H), 7.98 (d, *J* = 8.2 Hz, 2H), 7.84 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.56, 138.65, 132.82, 129.80, 117.57.



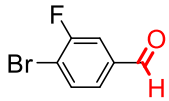
32; 2,4-Difluorobenzaldehyde; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.28 (s, 1H), 7.91 (td, J = 8.3, 6.5 Hz, 1H), 7.02 – 6.97 (m, 1H), 6.91 (ddd, J = 10.8, 8.6, 2.4 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 185.64, 185.59, 168.04, 167.95, 166.55, 166.45, 165.98, 165.88, 164.47, 164.37, 130.74, 130.72, 130.66, 130.63, 121.12, 121.10, 121.06, 112.73, 112.70, 112.55, 112.52, 104.96, 104.76, 104.56.



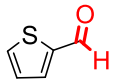
33; 2,4-Dichlorobenzaldehyde; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.23 (d, J = 0.5 Hz, 1H), 7.74 (dd, J = 7.5, 0.5 Hz, 1H), 7.50 (d, J = 1.5 Hz, 1H), 7.41 (dd, J = 7.5, 1.5 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 188.19, 135.58, 134.02, 131.52, 130.26, 130.11, 128.63.



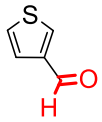
34; 4-Chloro-3-fluorobenzaldehyde; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.92 (t, J = 0.5 Hz, 1H), 7.65 (dddd, J = 9.7, 8.0, 1.5, 0.5 Hz, 2H), 7.46 (dd, J = 7.4, 5.0 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.35, 190.33, 159.21, 157.20, 134.98, 134.91, 129.87, 129.85, 127.87, 127.81, 126.08, 125.92, 116.24, 116.08.



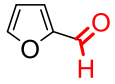
35; 4-Bromo-3-fluorobenzaldehyde; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.92 (d, *J* = 1.8 Hz, 1H), 7.72 (t, *J* = 7.2 Hz, 1H), 7.60 – 7.45 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.77, 160.71, 158.21, 137.20, 134.44, 126.39, 116.71, 116.50, 116.15, 115.92.



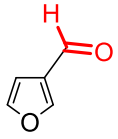
36; 2-Thiophenecarbaldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.95 (d, *J* = 1.2 Hz, 1H), 7.97 – 7.54 (m, 2H), 7.22 (dd, *J* = 4.9, 3.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 182.93, 143.96, 136.27, 135.06, 128.26.



37; 3-Thiophene carboxaldehyde [1]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.90 (d, *J* = 0.8 Hz, 1H), 8.11 (dd, *J* = 2.9, 1.2 Hz, 1H), 7.51 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.35 (ddd, *J* = 5.1, 2.9, 0.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.81, 142.81, 136.65, 127.25, 125.11.

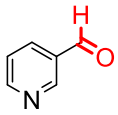


38; 2-Furaldehyde; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.63 (s, 1H), 7.67 (s, 1H), 7.23 (d, J = 4.8 Hz, 1H), 6.58 (dd, J = 4.4, 1.9 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.70, 152.80, 147.96, 112.45.

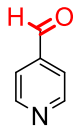


39; 3-Furaldehyde [10]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.89 (s, 1H), 8.06 (s, 1H), 7.44 (s, 1H), 6.72 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.27, 151.28, 144.77, 128.50, 106.70.

40; 2-Pyridinecarboxaldehyde [3]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.09 (s, 1H), 8.81 (dt, *J* = 4.8, 1.3 Hz, 1H), 7.98 (dt, *J* = 7.8, 1.2 Hz, 1H), 7.91 (td, *J* = 7.6, 1.6 Hz, 1H), 7.57 (ddd, *J* = 7.4, 4.8, 1.4 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.20, 152.60, 150.02, 136.89, 127.70, 121.51.

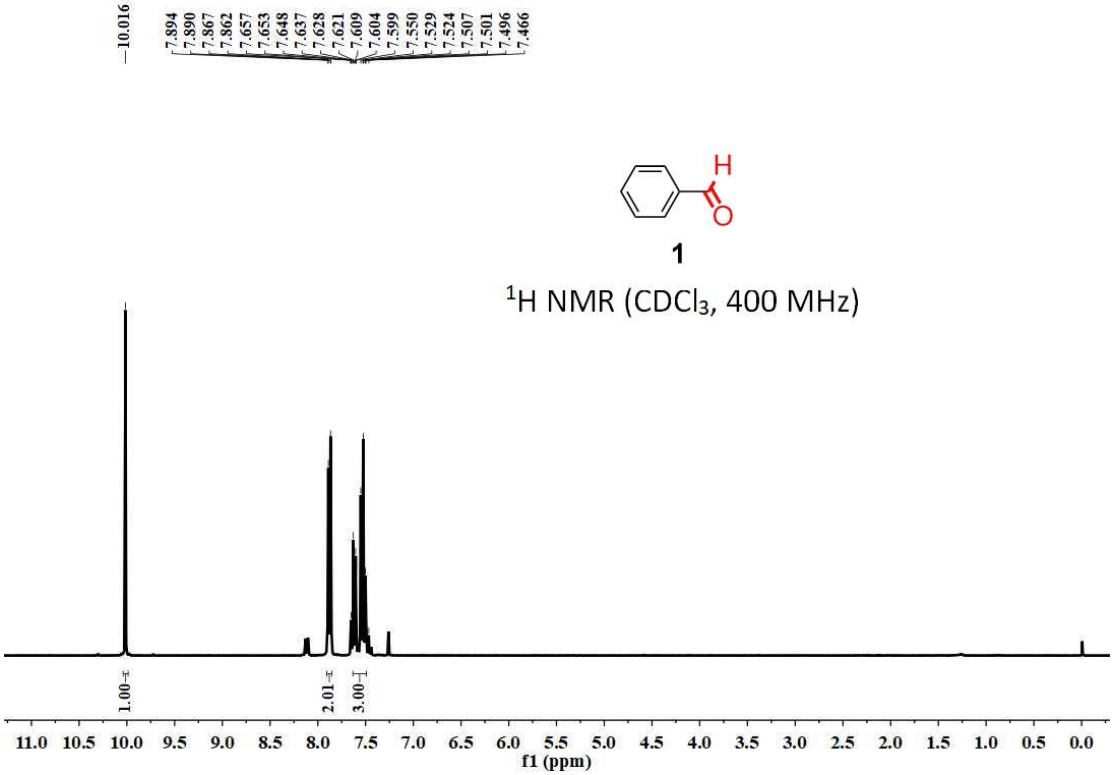


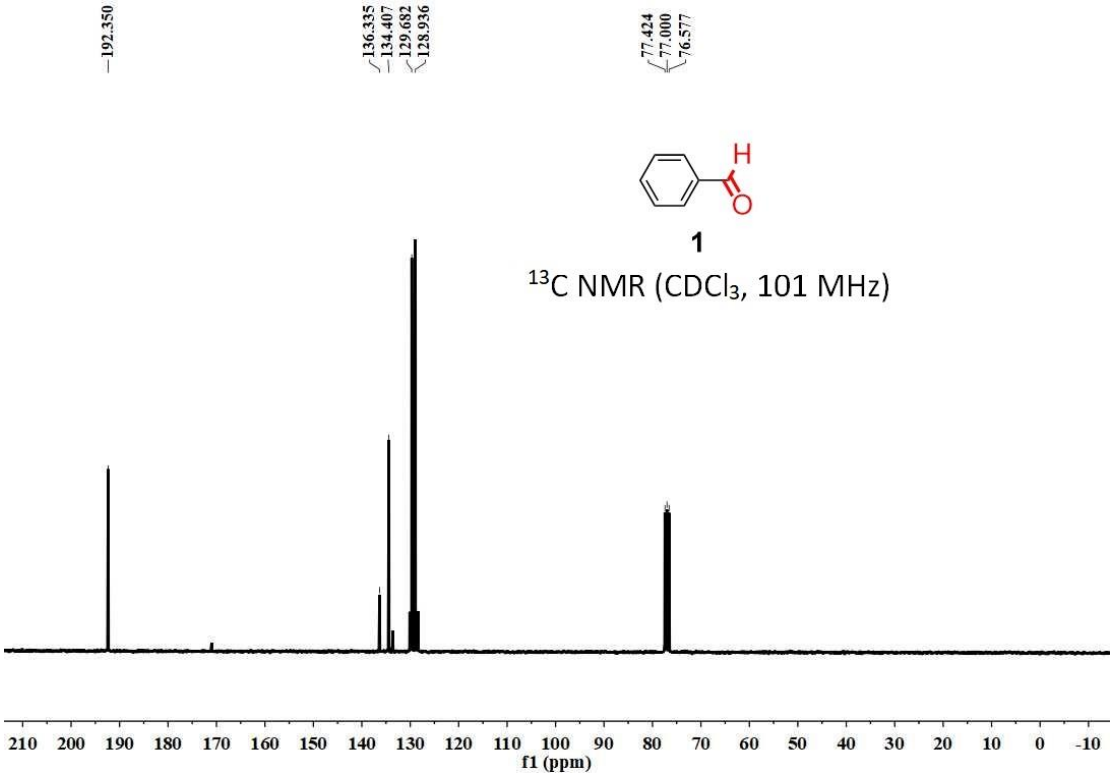
41; 3-Pyridinecarboxaldehyde [11]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.04 (s, 1H), 8.99 (d, *J* = 2.1 Hz, 1H), 8.75 (dd, *J* = 4.9, 1.8 Hz, 1H), 8.08 (dt, *J* = 7.9, 2.0 Hz, 1H), 7.41 (dd, *J* = 7.9, 4.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.60, 154.55, 151.85, 135.58, 131.19, 123.87.

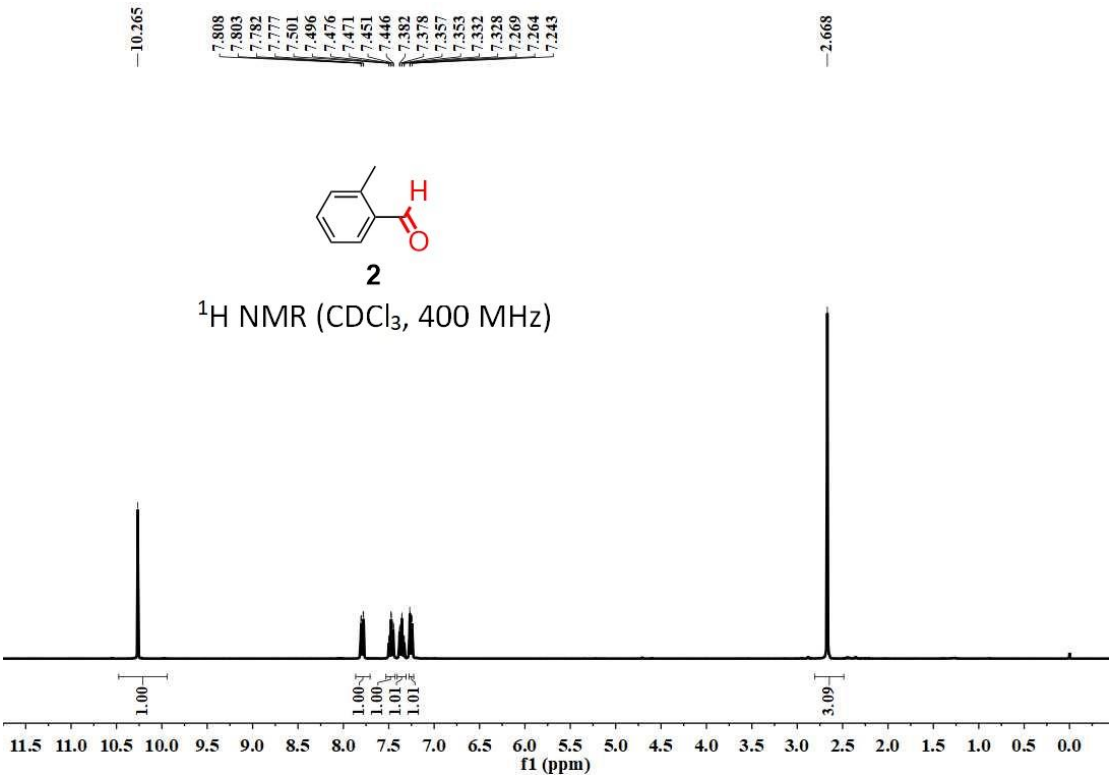


42; 4-Pyridinecarboxaldehyde [12]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 1H), 8.77 (d, *J* = 6.0 Hz, 2H), 7.60 (d, *J* = 6.0 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.29, 150.97, 141.15, 121.85.

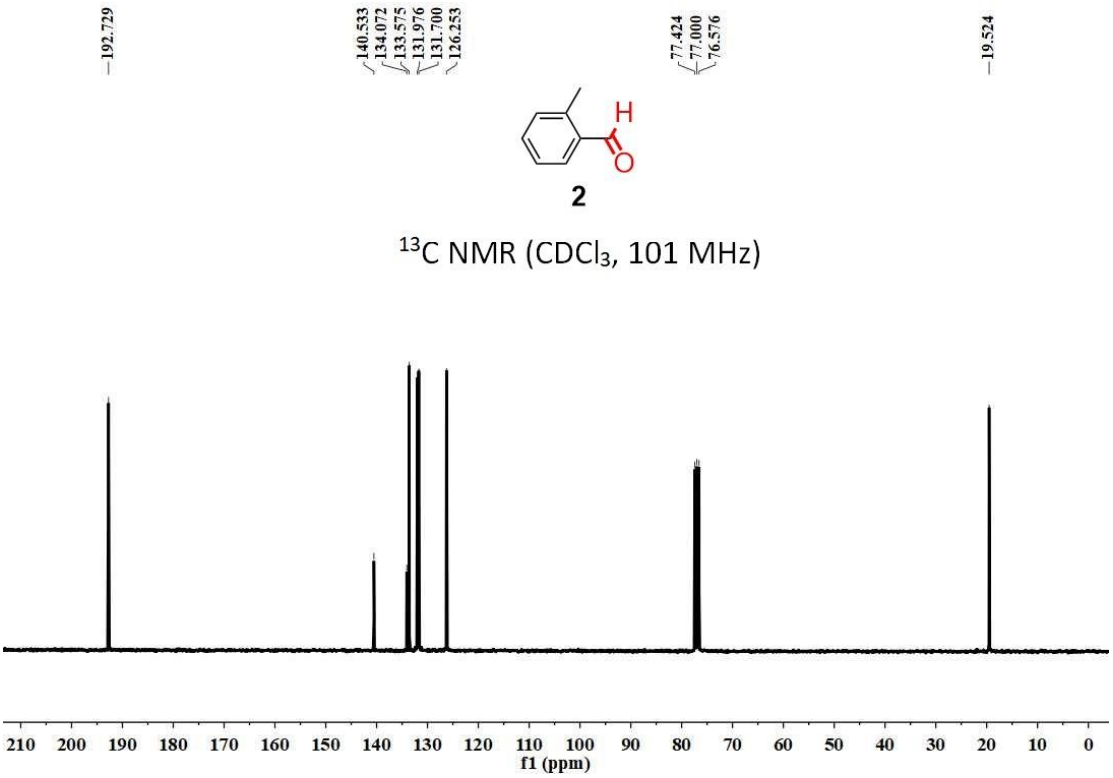
3. NMR spectra of the aromatic aldehyde products



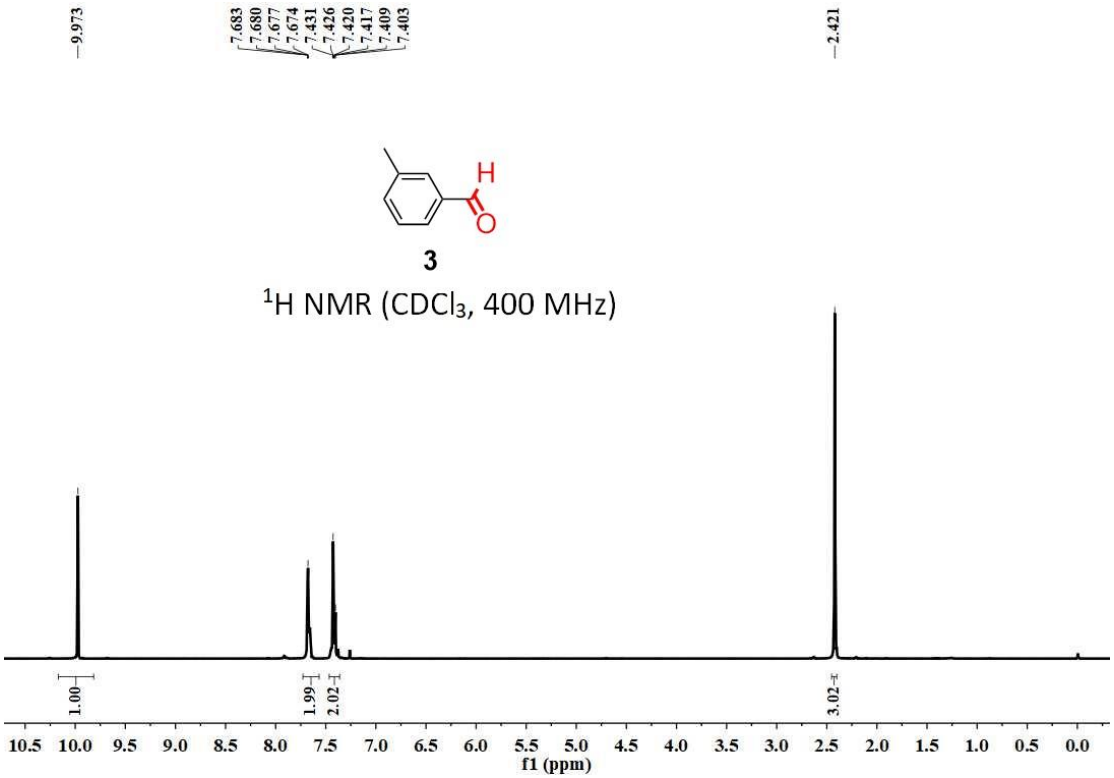




^{1}H NMR ($CDCl_3$, 400 MHz)

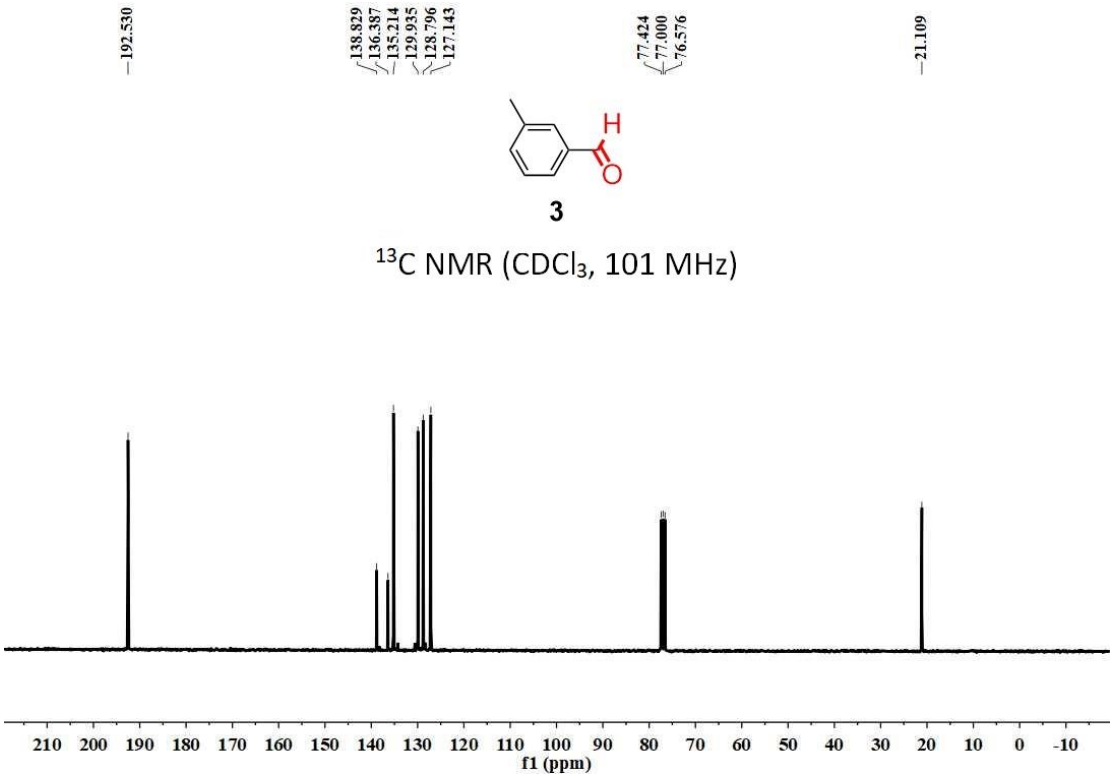


^{13}C NMR ($CDCl_3$, 101 MHz)



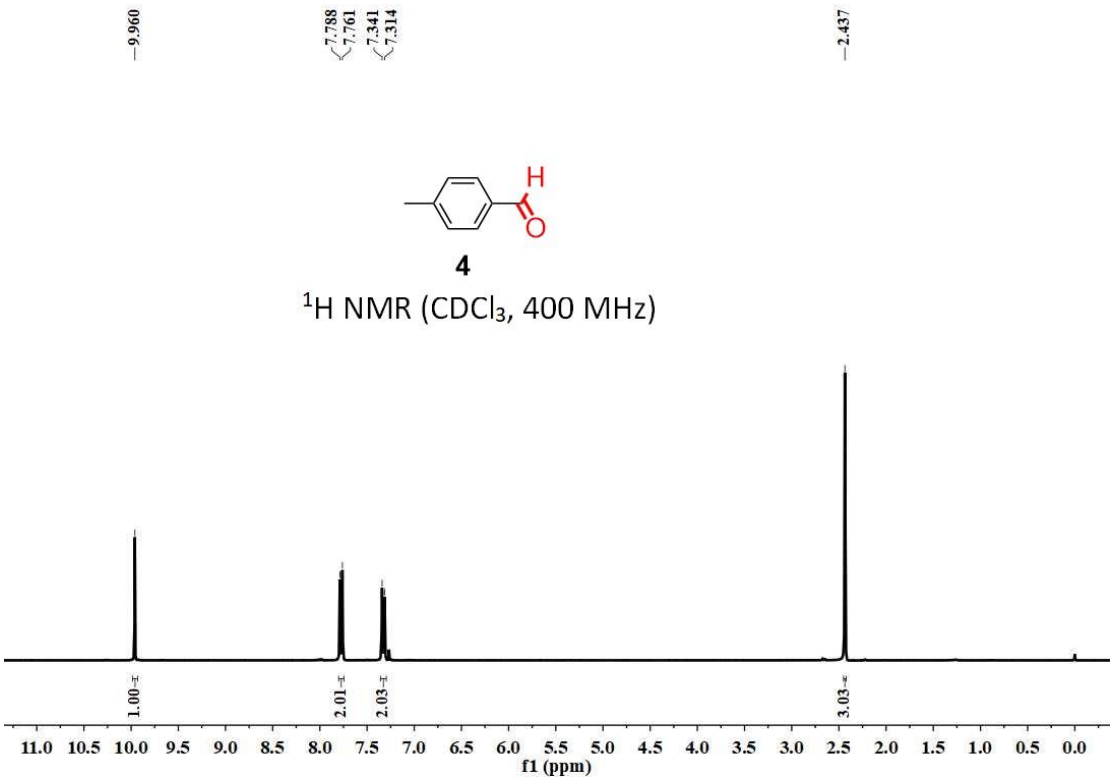
3

^{1}H NMR ($CDCl_3$, 400 MHz)



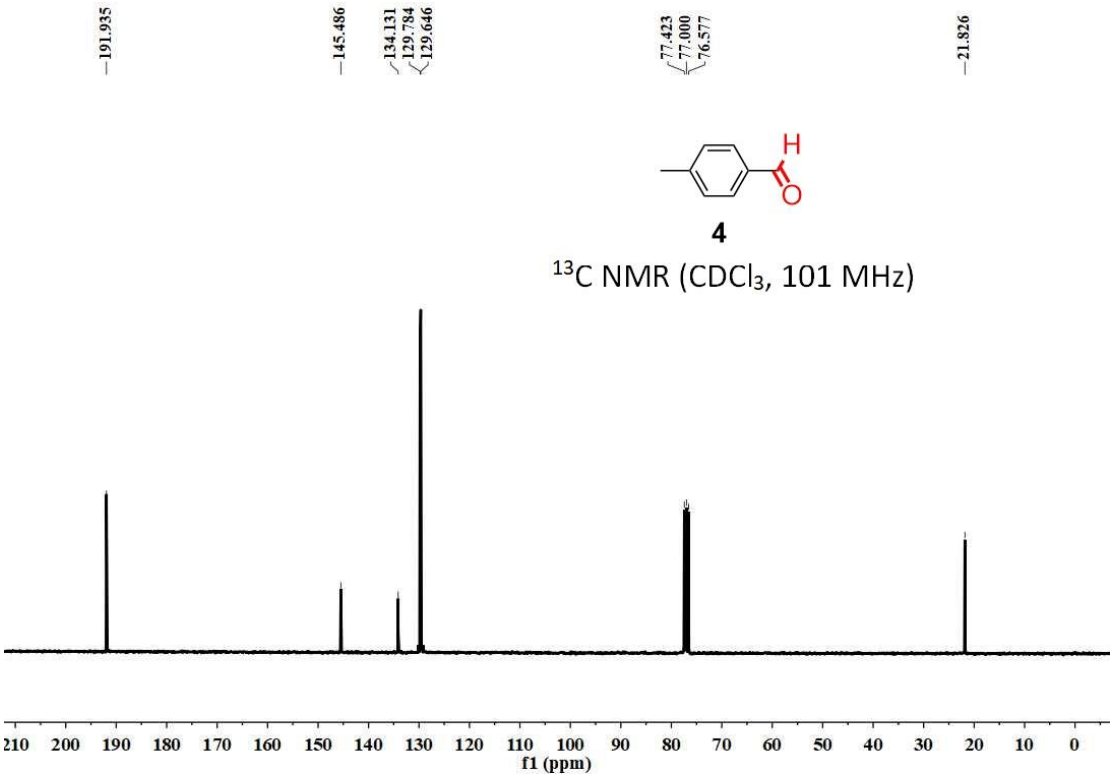
3

^{13}C NMR ($CDCl_3$, 101 MHz)



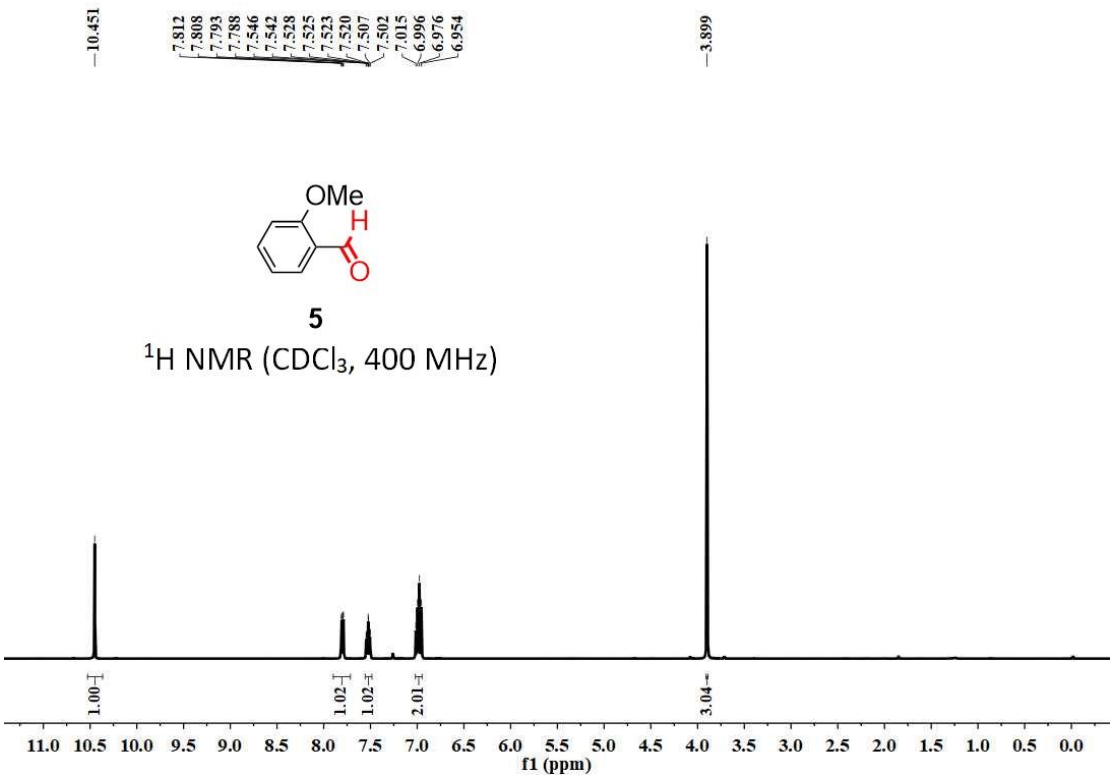
4

^{1}H NMR ($CDCl_3$, 400 MHz)



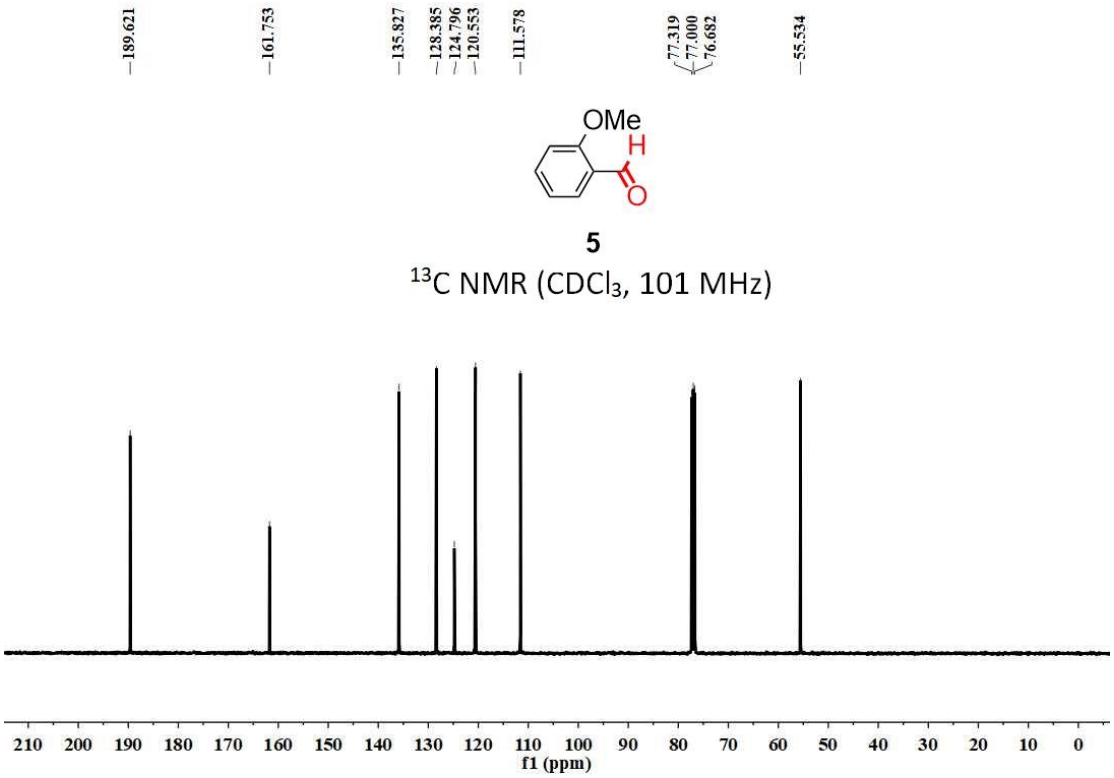
4

^{13}C NMR ($CDCl_3$, 101 MHz)



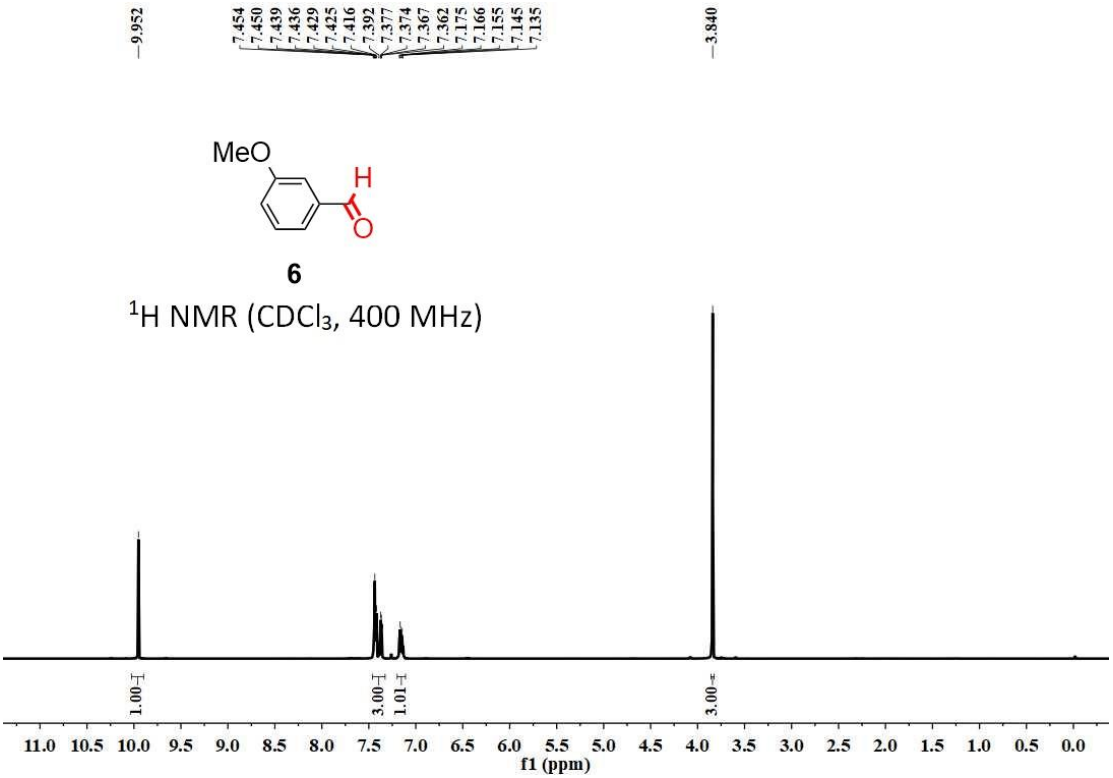
5

^{1}H NMR ($CDCl_3$, 400 MHz)



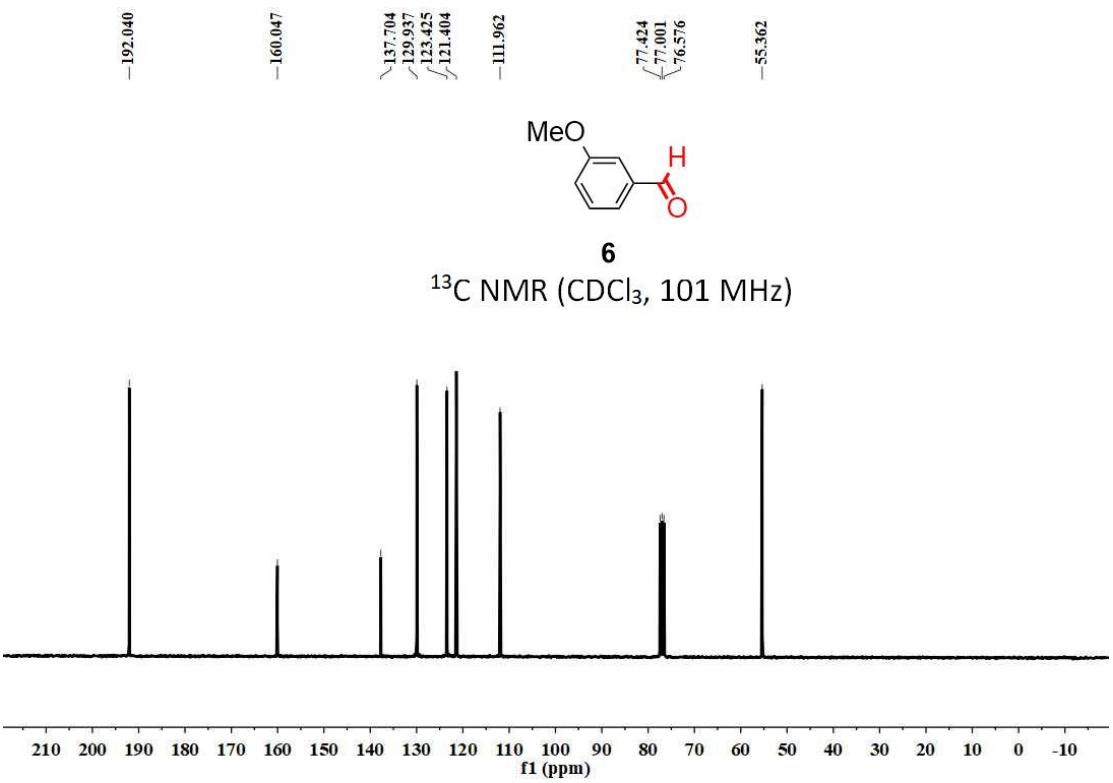
5

^{13}C NMR ($CDCl_3$, 101 MHz)



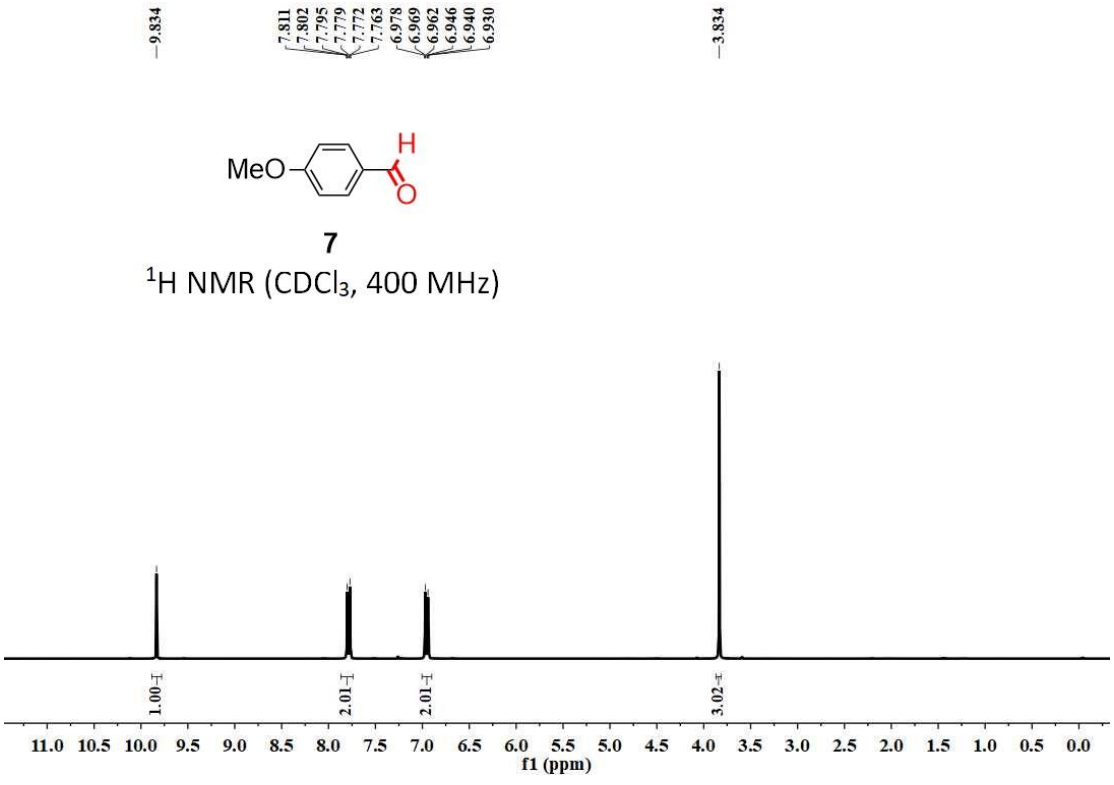
6

^{1}H NMR ($CDCl_3$, 400 MHz)



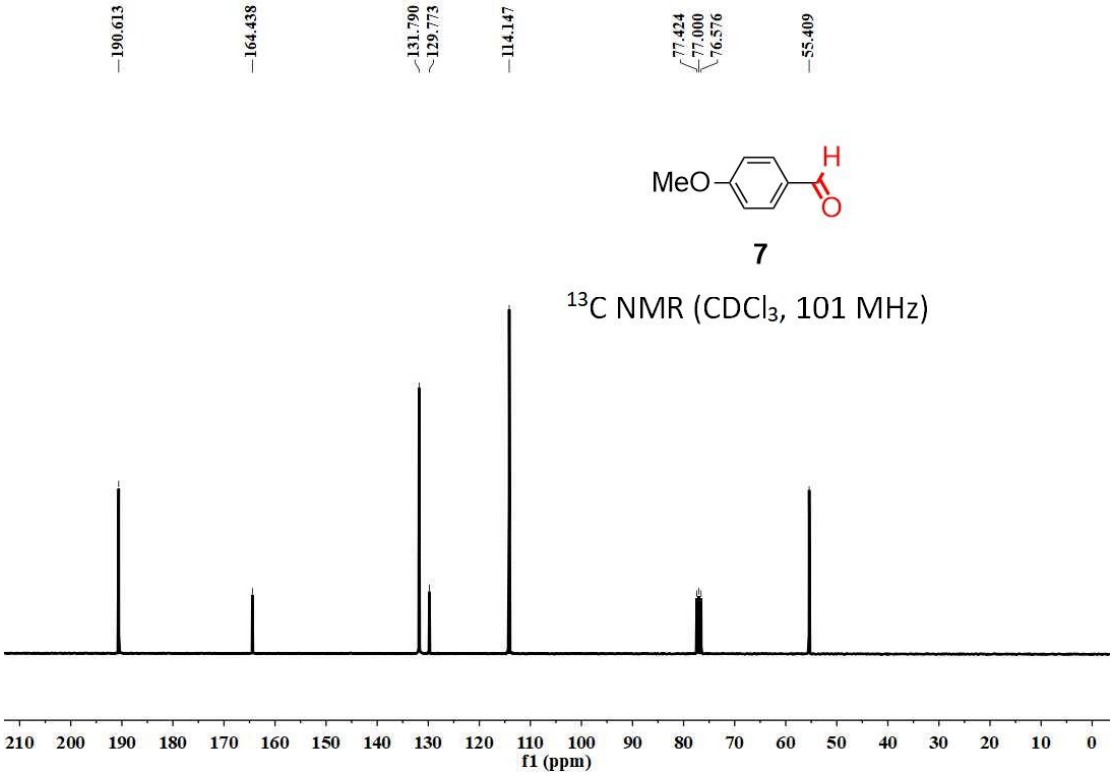
6

^{13}C NMR ($CDCl_3$, 101 MHz)



7

^{1}H NMR ($CDCl_3$, 400 MHz)

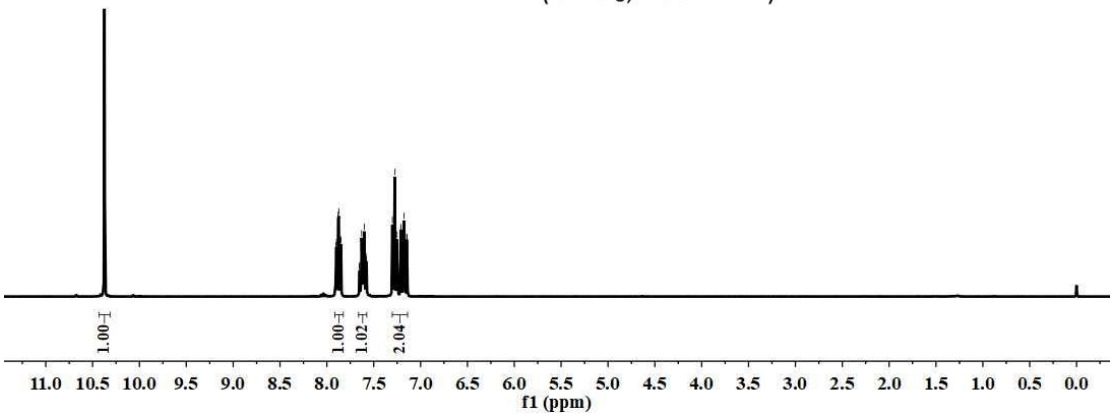


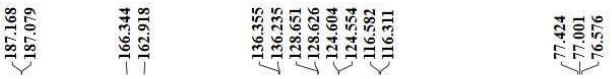
7

^{13}C NMR ($CDCl_3$, 101 MHz)

8

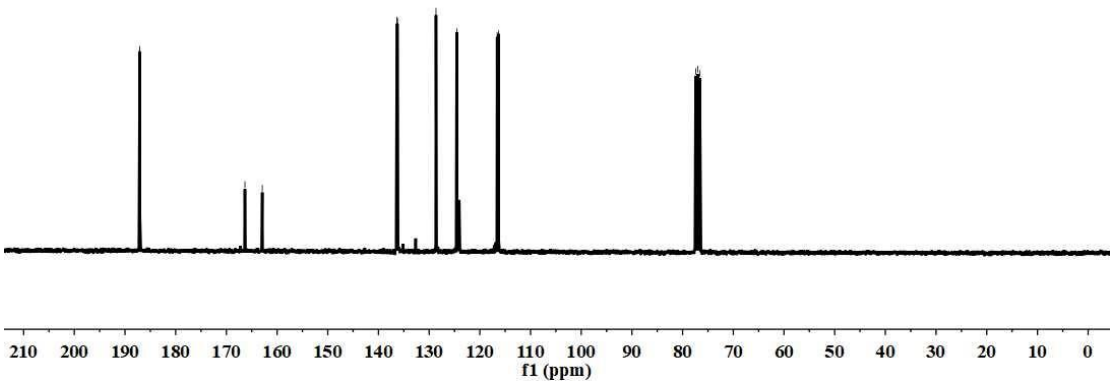
^{1}H NMR ($CDCl_3$, 400 MHz)

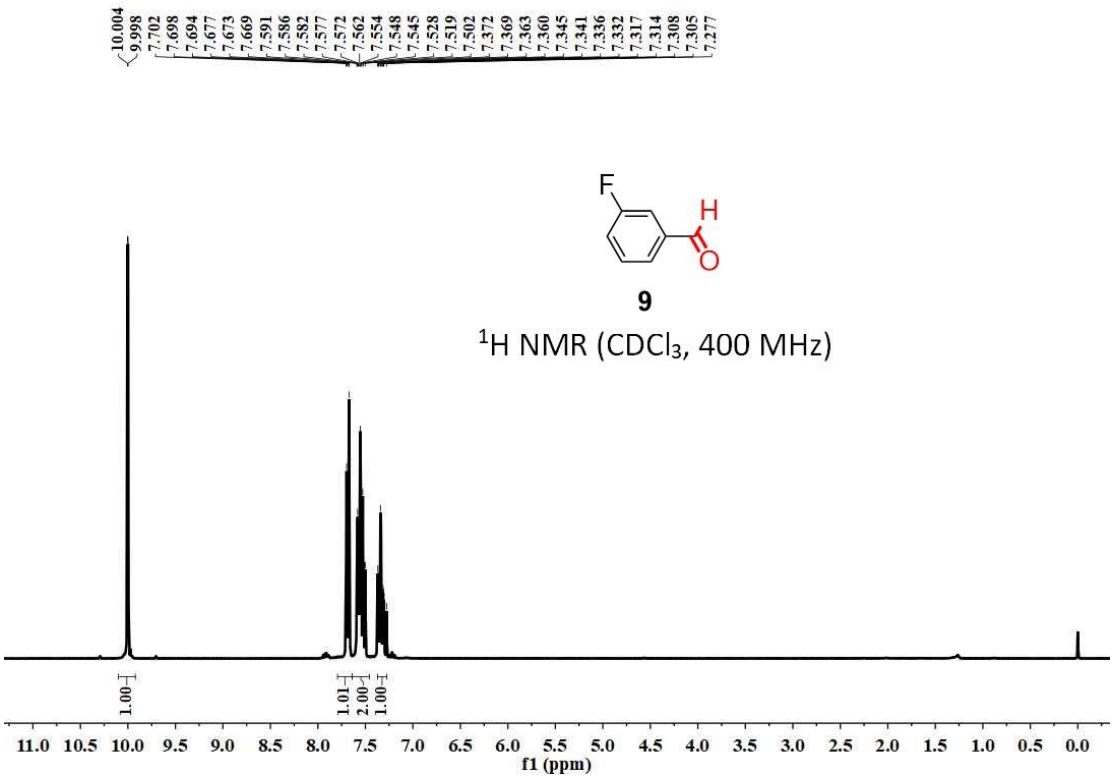




8

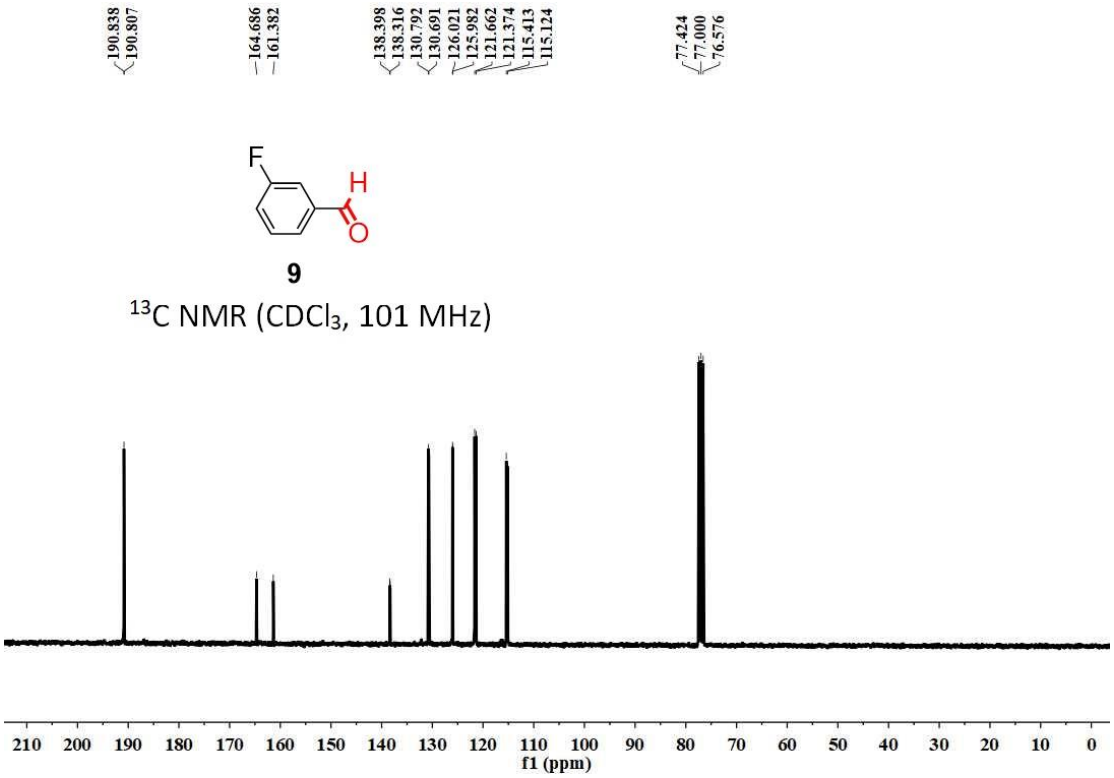
^{13}C NMR ($CDCl_3$, 101 MHz)





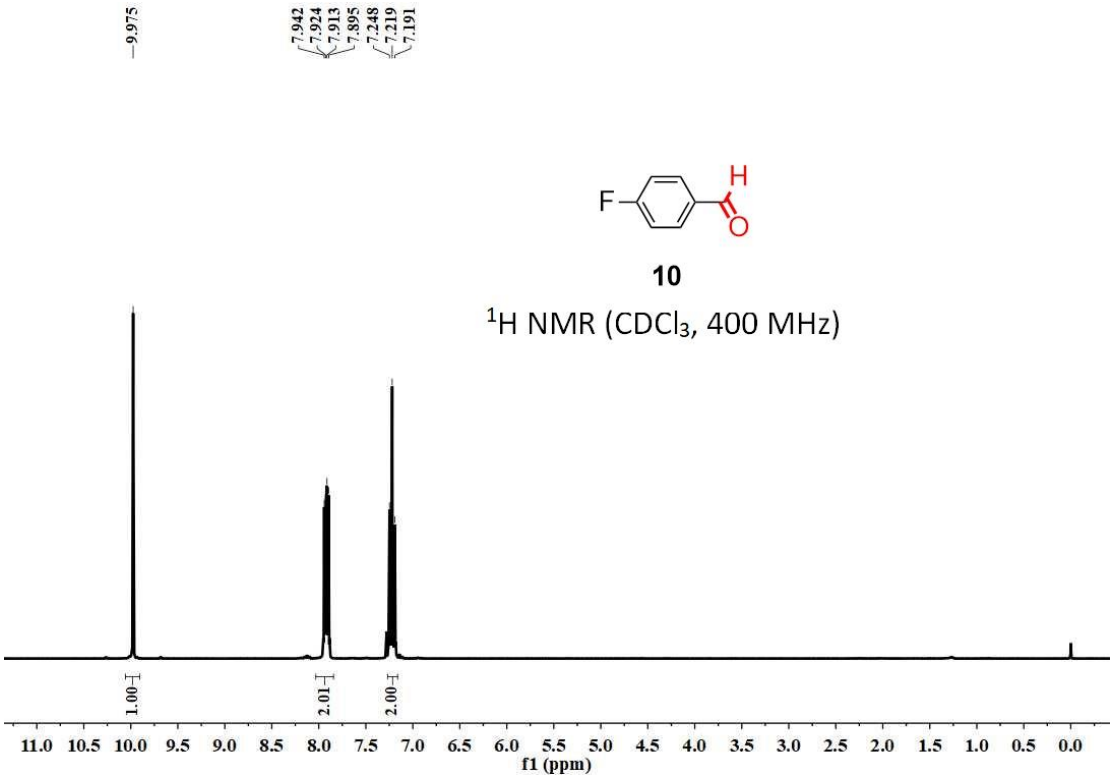
9

^{1}H NMR ($CDCl_3$, 400 MHz)



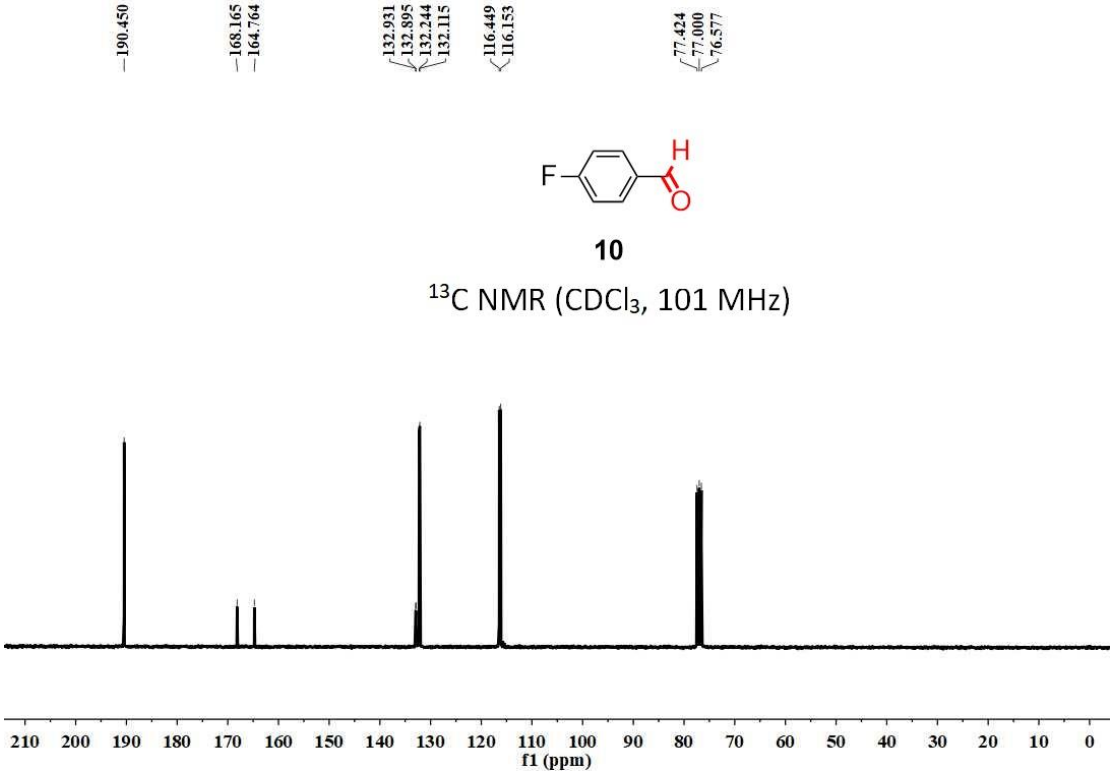
9

^{13}C NMR ($CDCl_3$, 101 MHz)



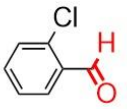
10

^{1}H NMR ($CDCl_3$, 400 MHz)



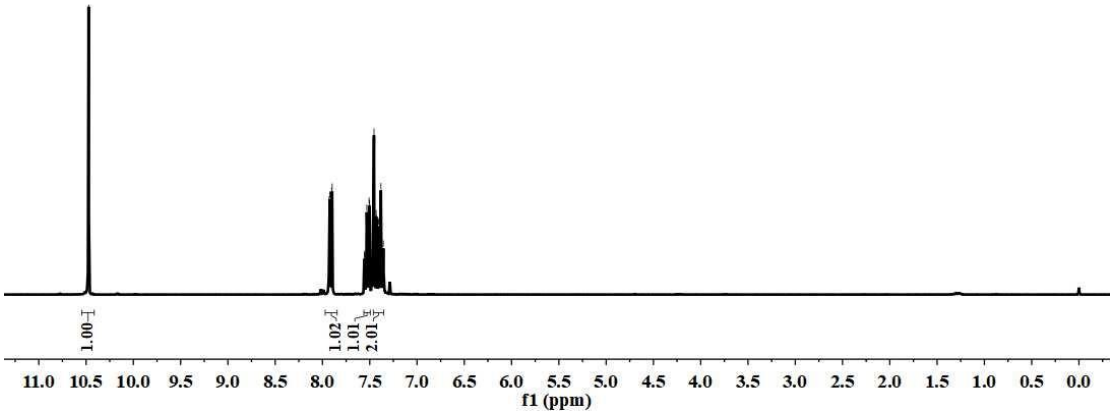
10

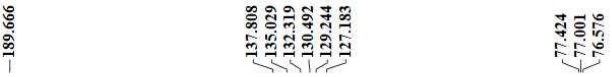
^{13}C NMR ($CDCl_3$, 101 MHz)

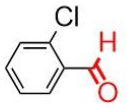


11

^{1}H NMR ($CDCl_3$, 400 MHz)

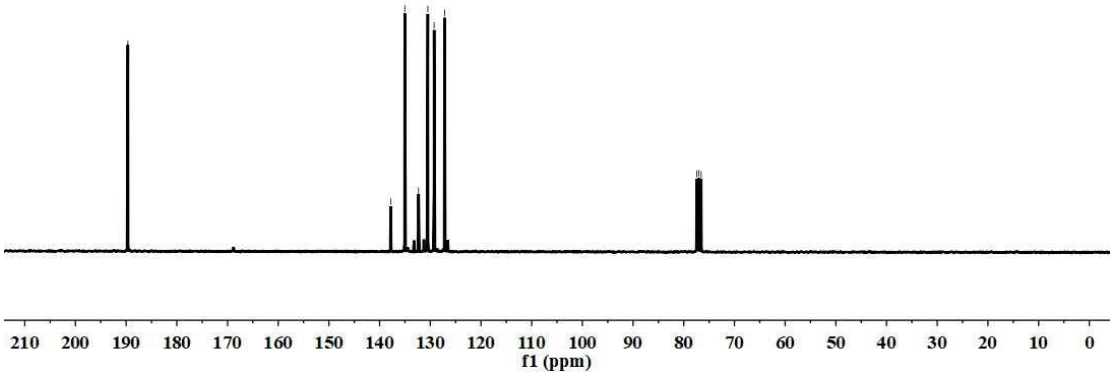


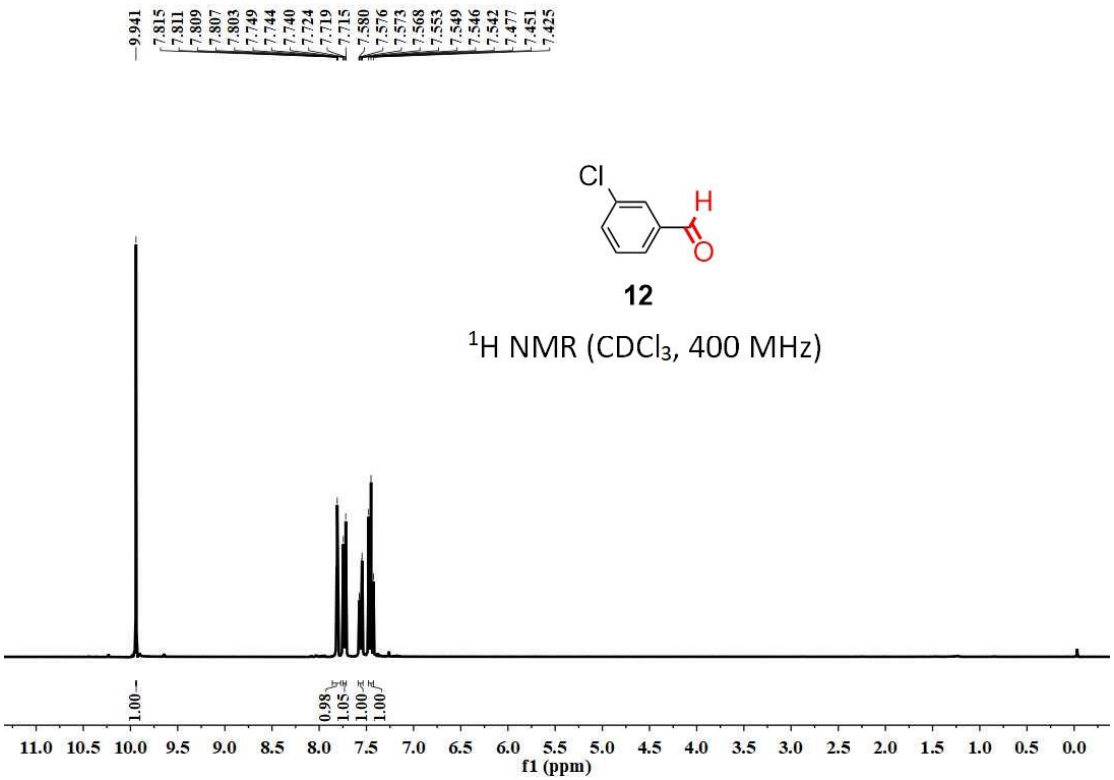




11

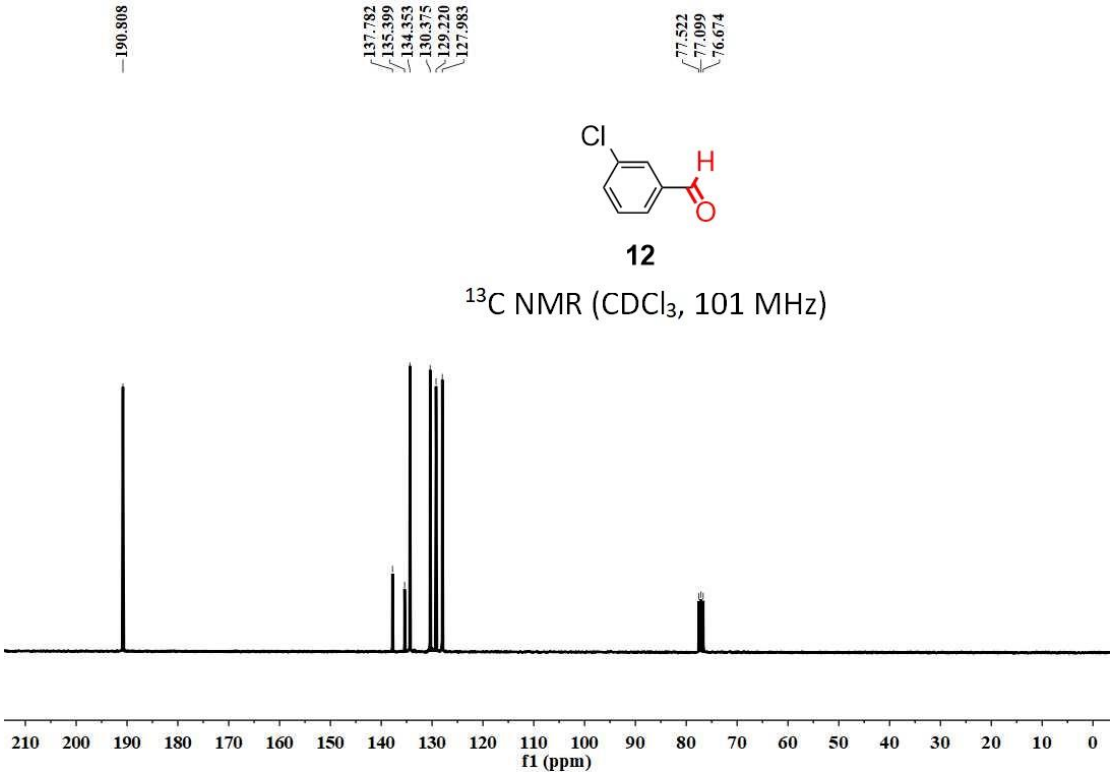
^{13}C NMR ($CDCl_3$, 101 MHz)





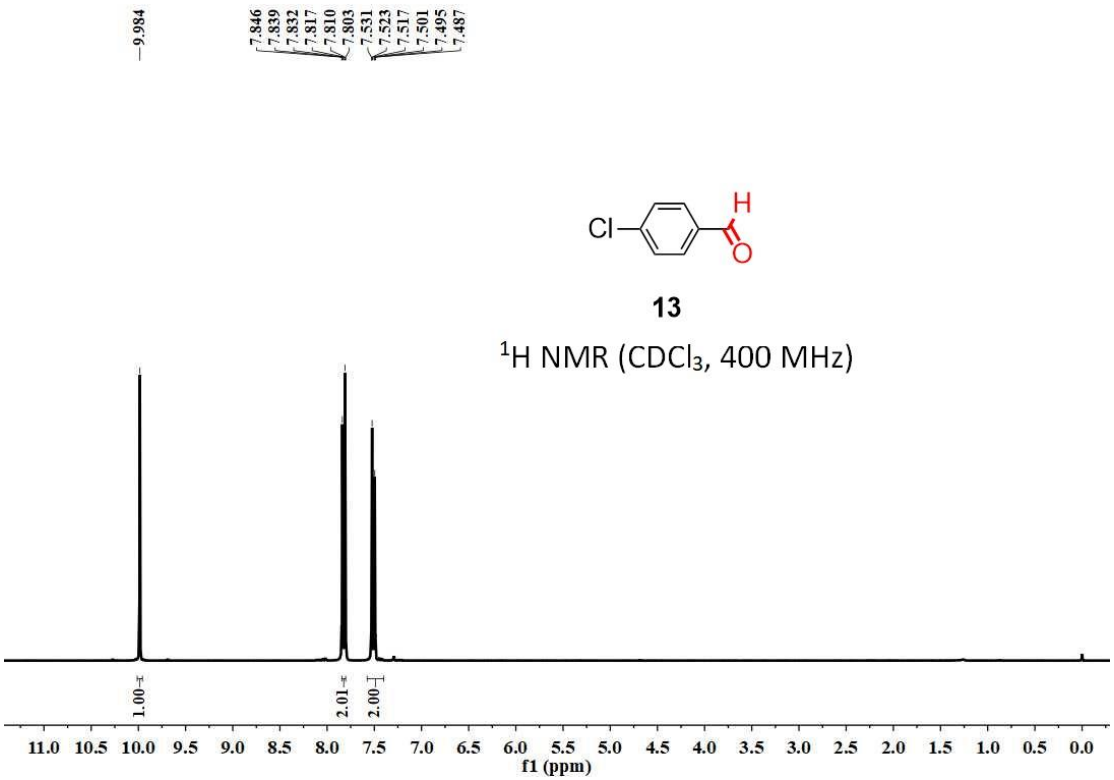
12

^{1}H NMR ($CDCl_3$, 400 MHz)



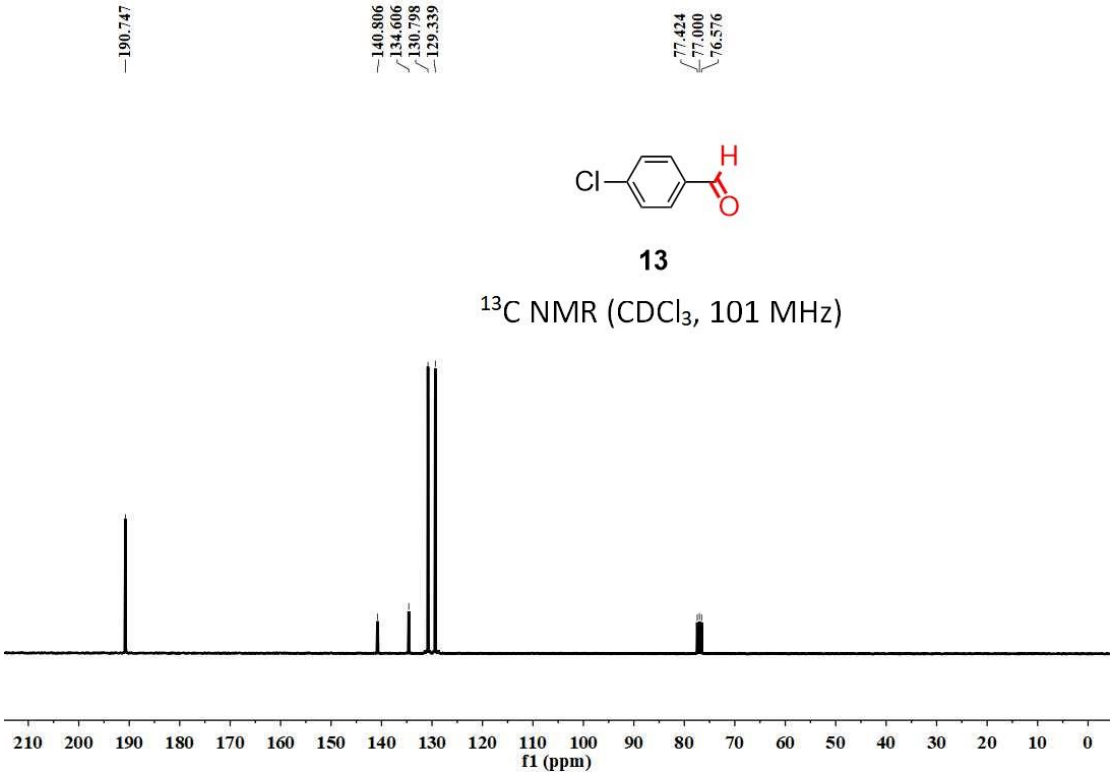
12

^{13}C NMR ($CDCl_3$, 101 MHz)



13

^{1}H NMR ($CDCl_3$, 400 MHz)

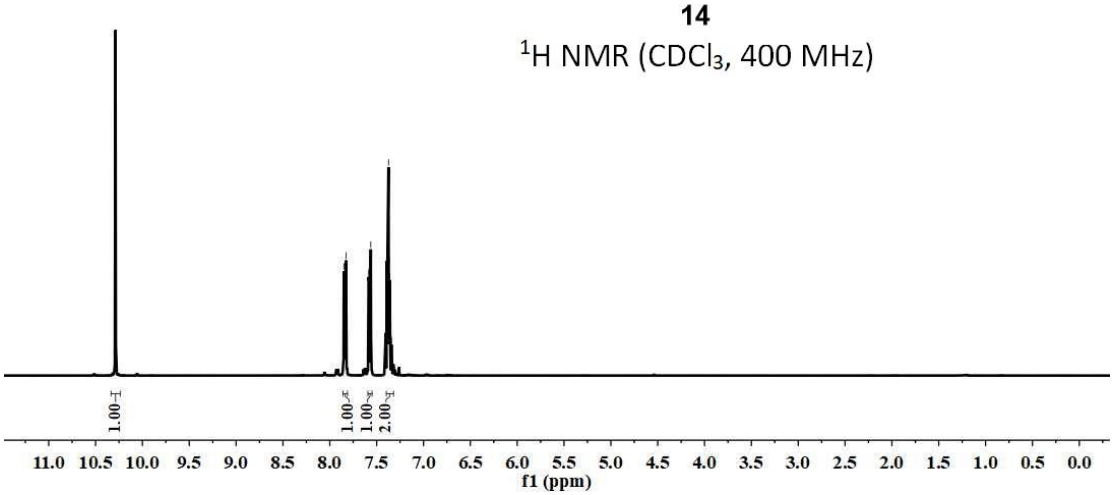


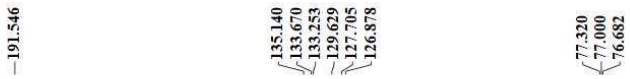
13

^{13}C NMR ($CDCl_3$, 101 MHz)

14

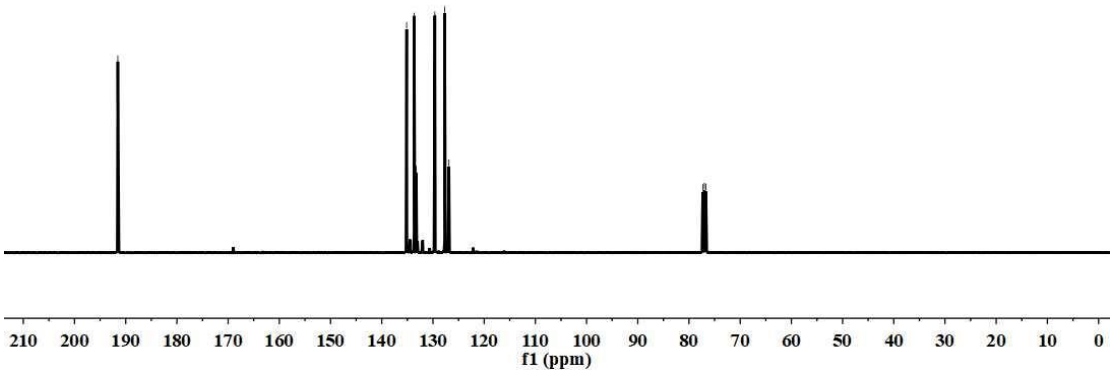
^{1}H NMR ($CDCl_3$, 400 MHz)

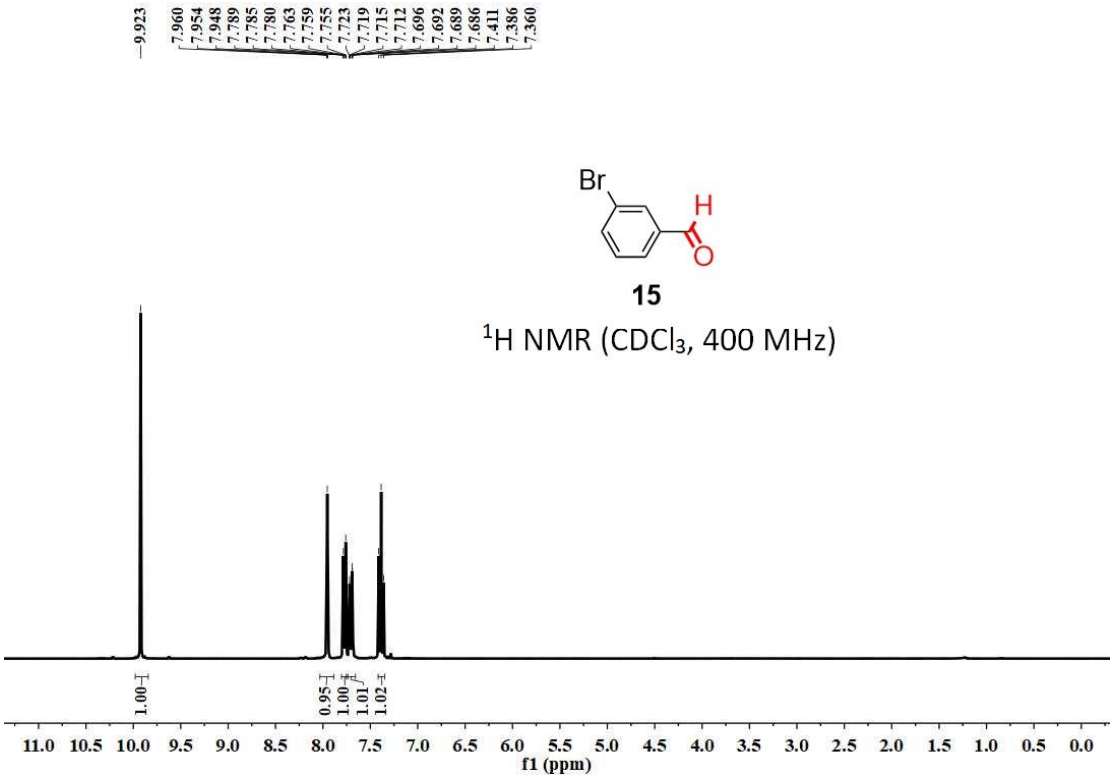


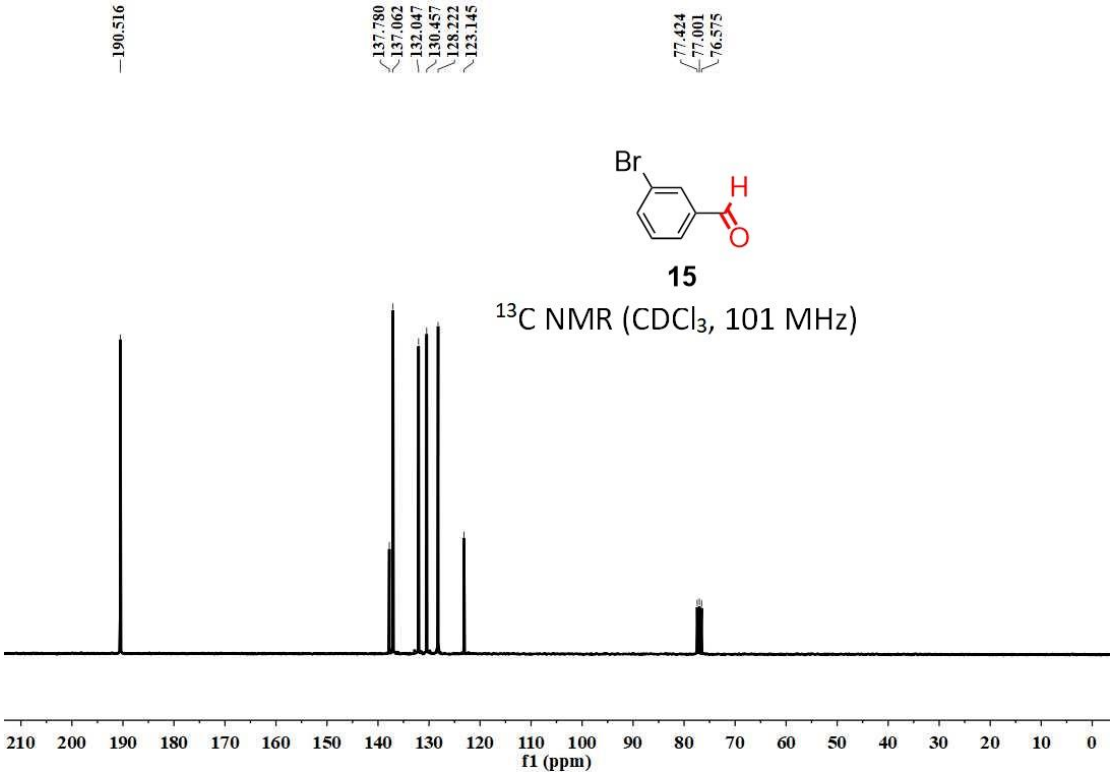


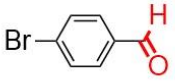
14

^{13}C NMR ($CDCl_3$, 101 MHz)



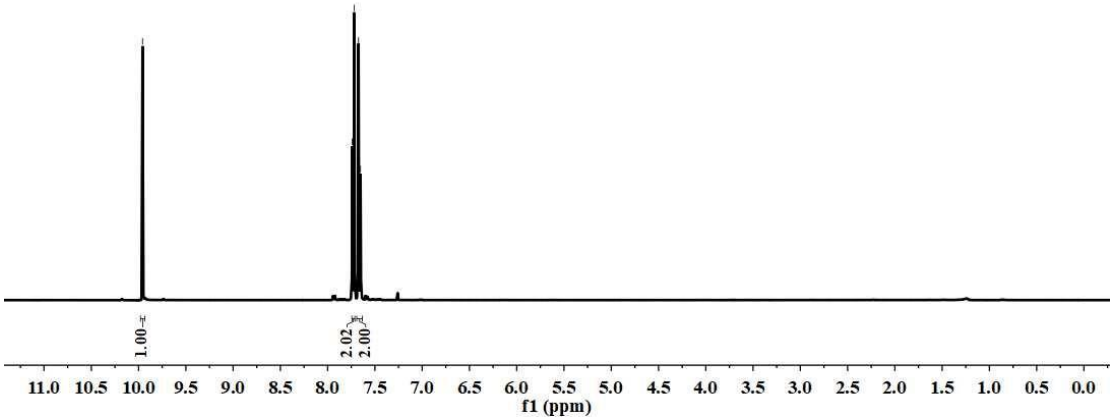

9.923
7.960
7.954
7.948
7.789
7.785
7.780
7.763
7.759
7.755
7.723
7.719
7.715
7.712
7.696
7.692
7.689
7.686
7.411
7.386
7.360
Br
H
O
15
1H NMR (CDCl3, 400 MHz)
1.00
0.95
1.00
1.01
1.02
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

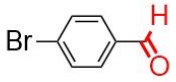

190.516
137.780
137.062
132.047
130.457
128.222
123.145
77.424
77.001
76.575
Br
H
O
15
13C NMR (CDCl3, 101 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



16

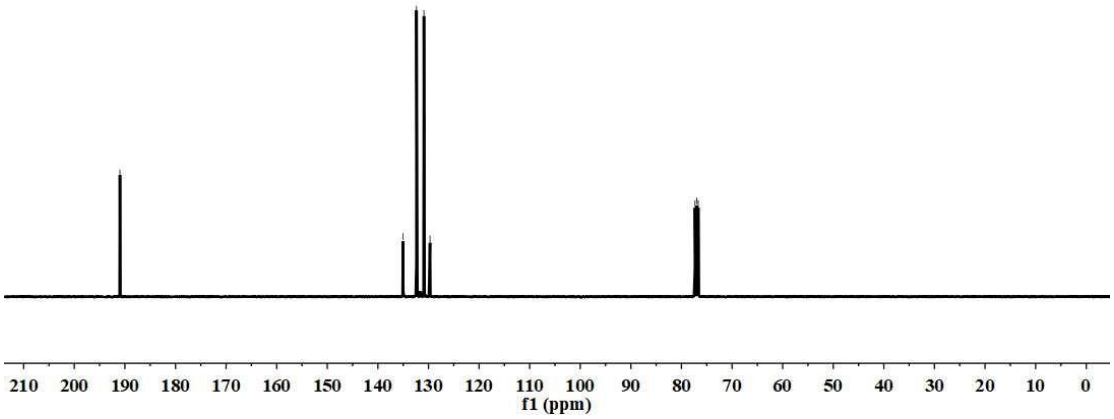
^{1}H NMR ($CDCl_3$, 400 MHz)

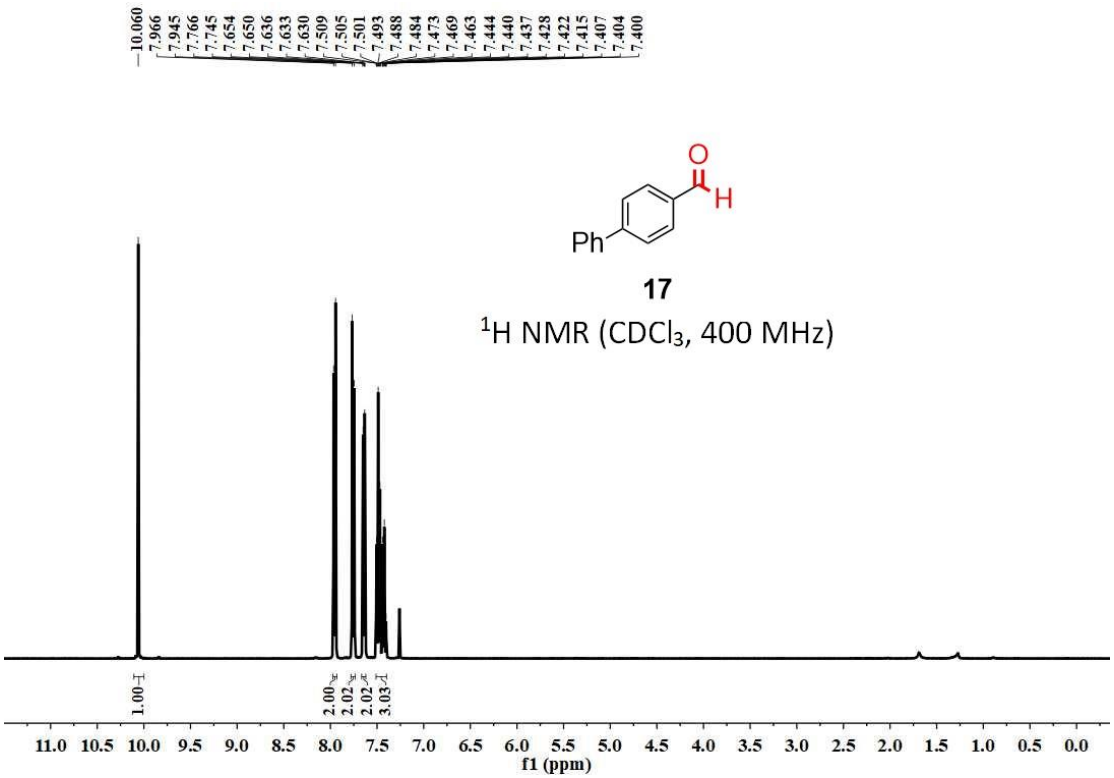




16

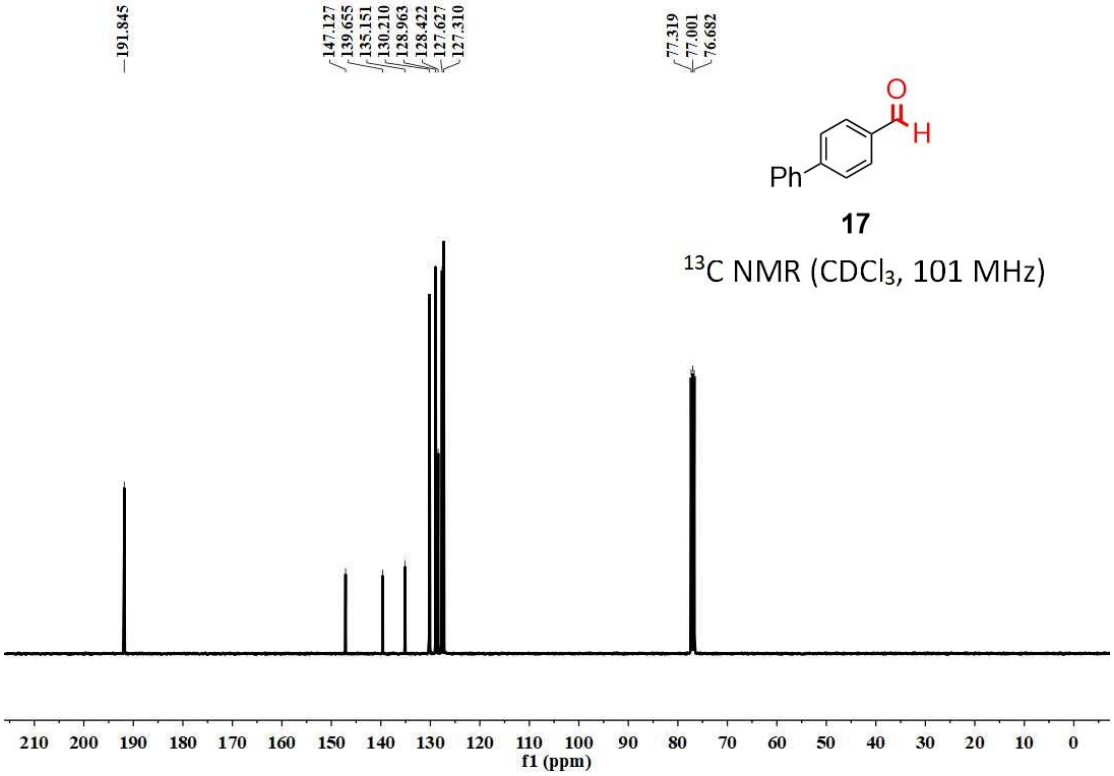
^{13}C NMR ($CDCl_3$, 101 MHz)





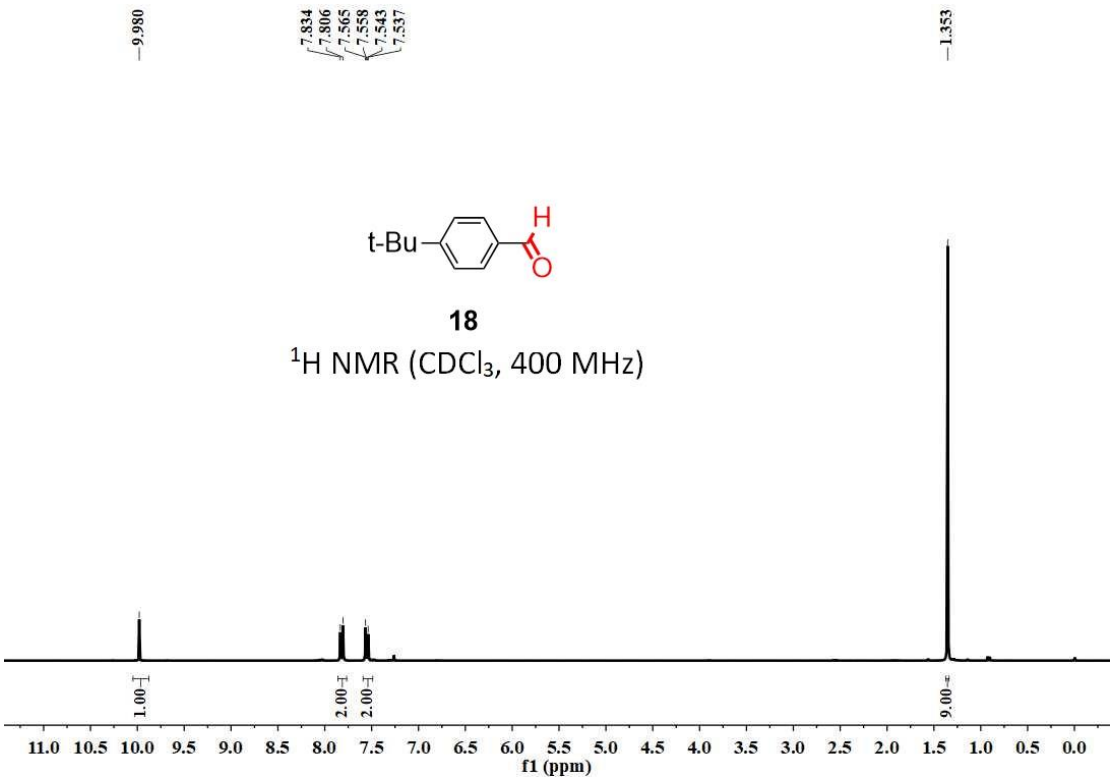
17

^{1}H NMR ($CDCl_3$, 400 MHz)



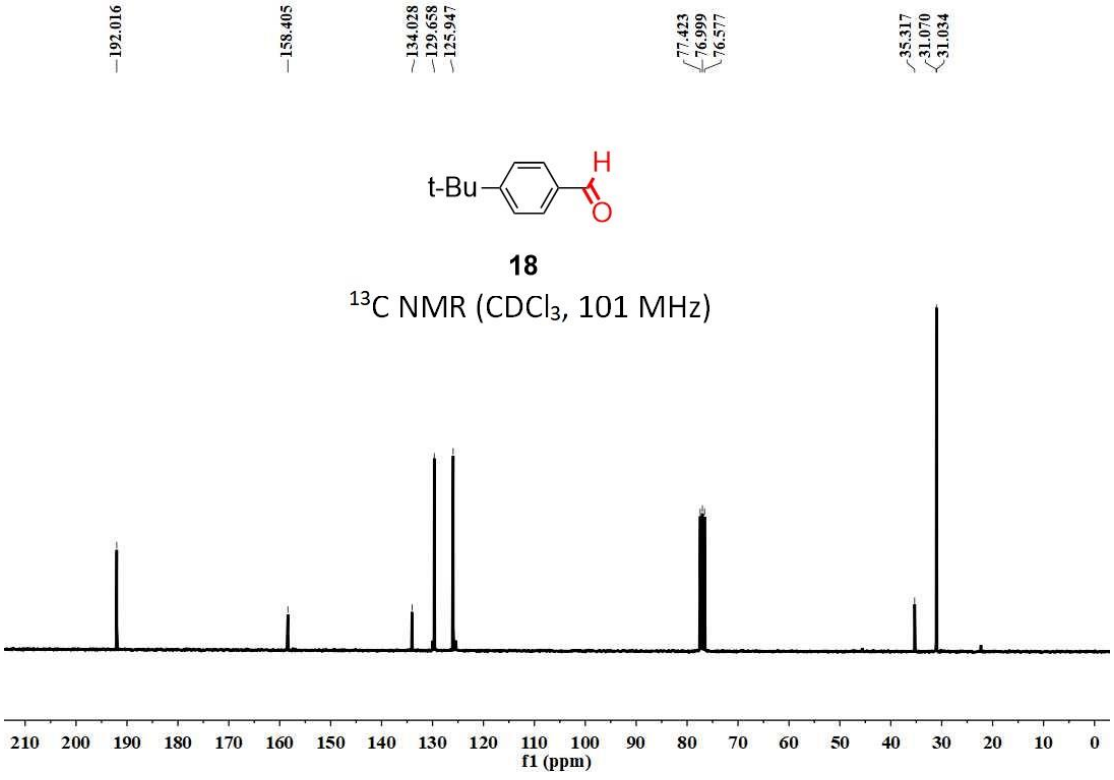
17

^{13}C NMR ($CDCl_3$, 101 MHz)



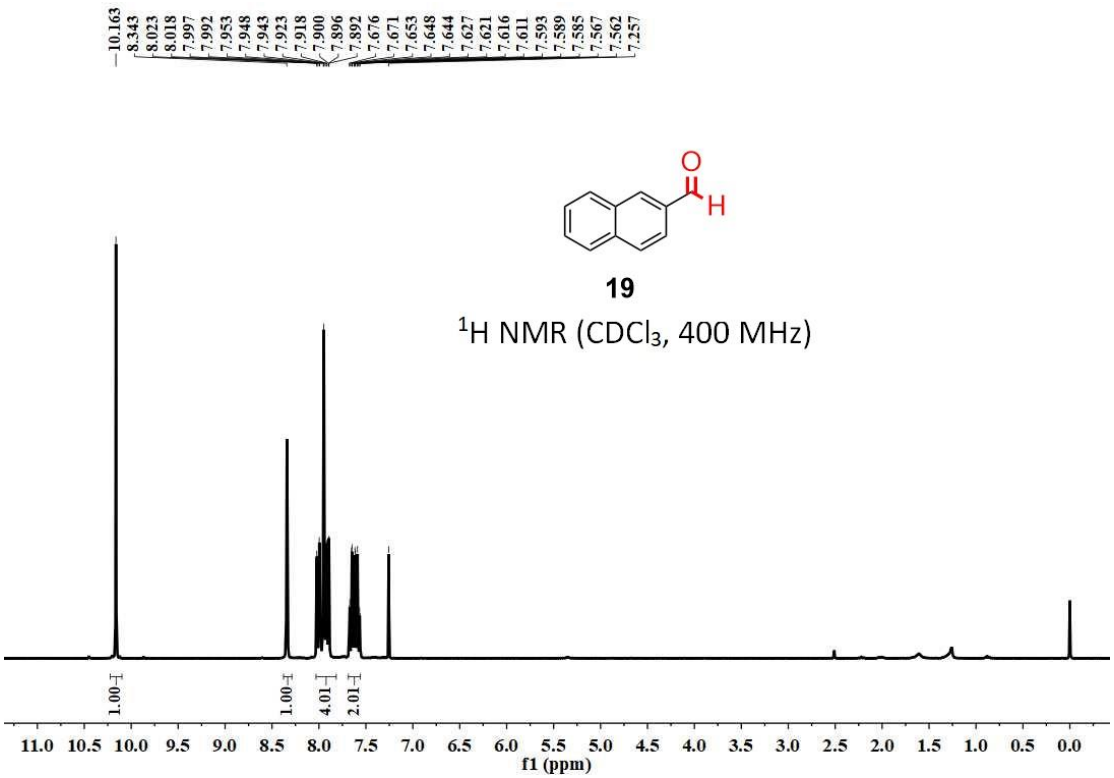
18

^{1}H NMR ($CDCl_3$, 400 MHz)



18

^{13}C NMR ($CDCl_3$, 101 MHz)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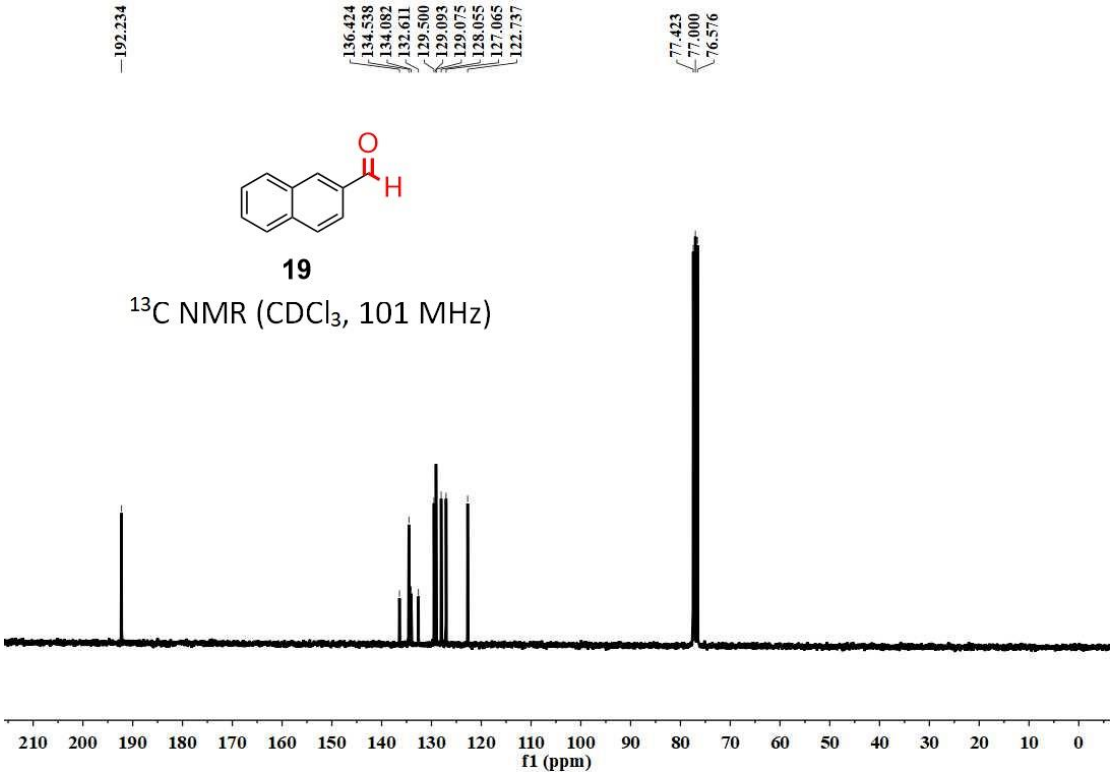
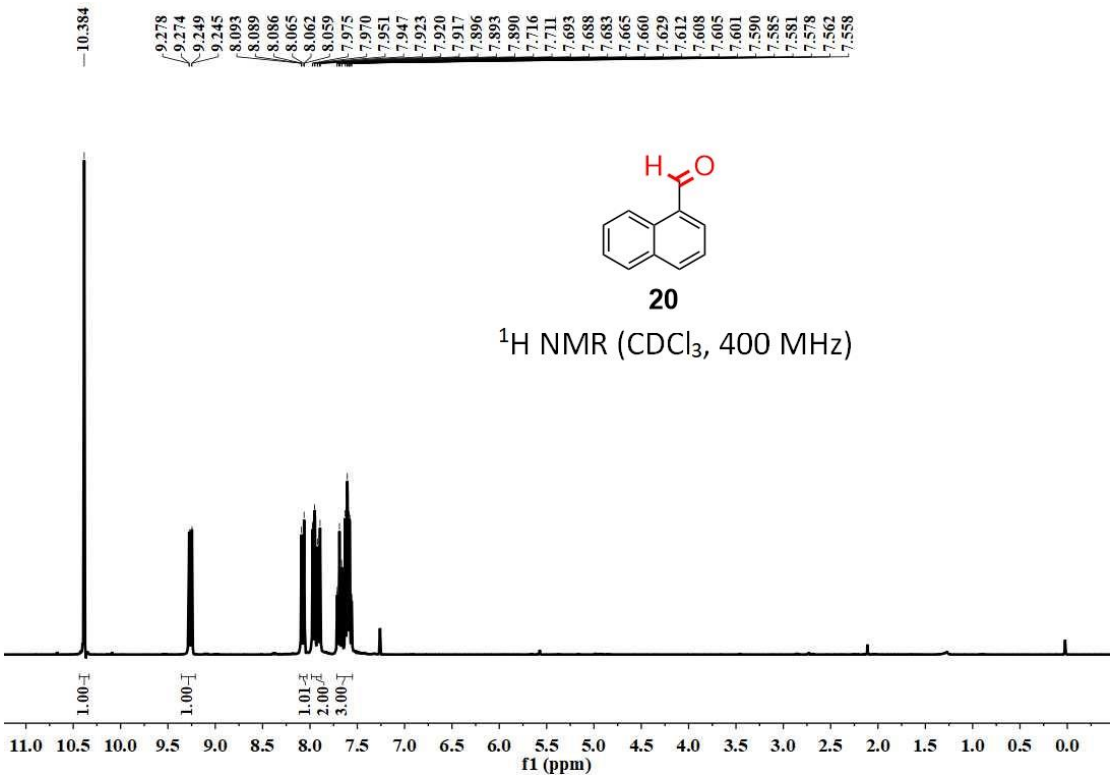
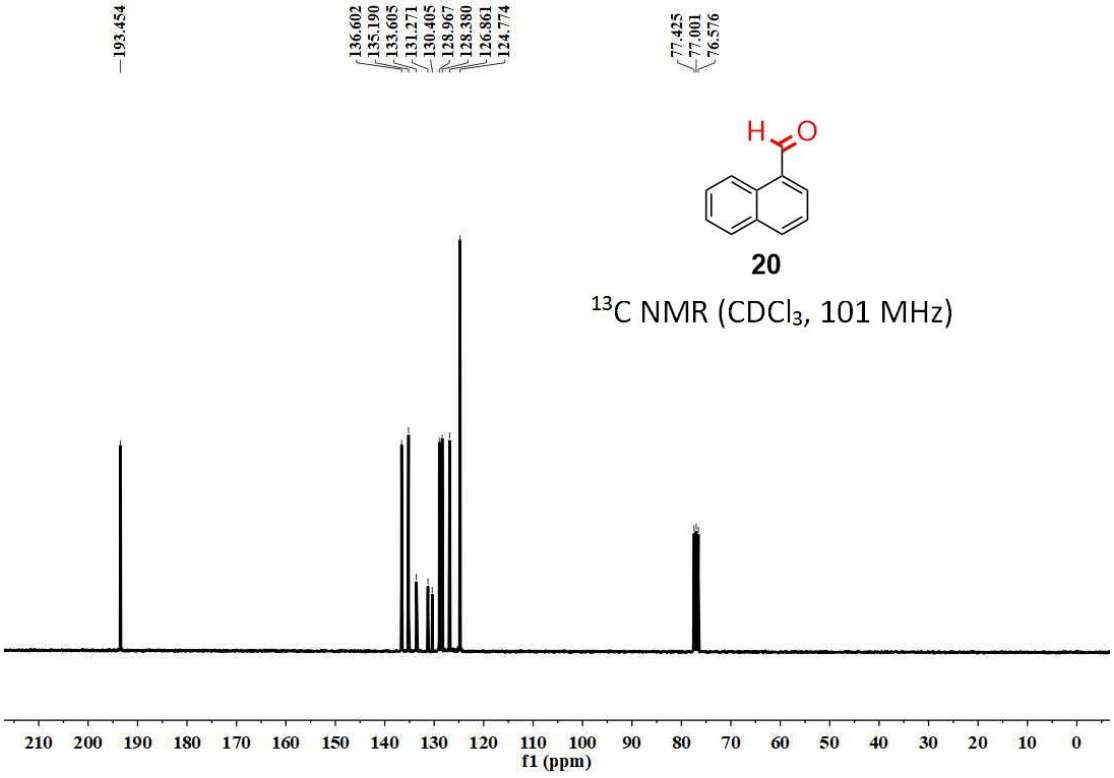
8.343
8.023
8.018
7.997
7.992
7.953
7.948
7.943
7.923
7.918
7.900
7.896
7.892
7.676
7.671
7.653
7.648
7.644
7.627
7.621
7.616
7.611
7.593
7.589
7.585
7.567
7.562
7.257
O
H
19
1H NMR (CDCl3, 400 MHz)
1.00
1.00
4.01
2.01
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


192.234
136.424
134.538
134.082
132.611
129.500
129.093
129.075
128.055
127.065
122.737
77.423
77.000
76.576
O
H
19
13C NMR (CDCl3, 101 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



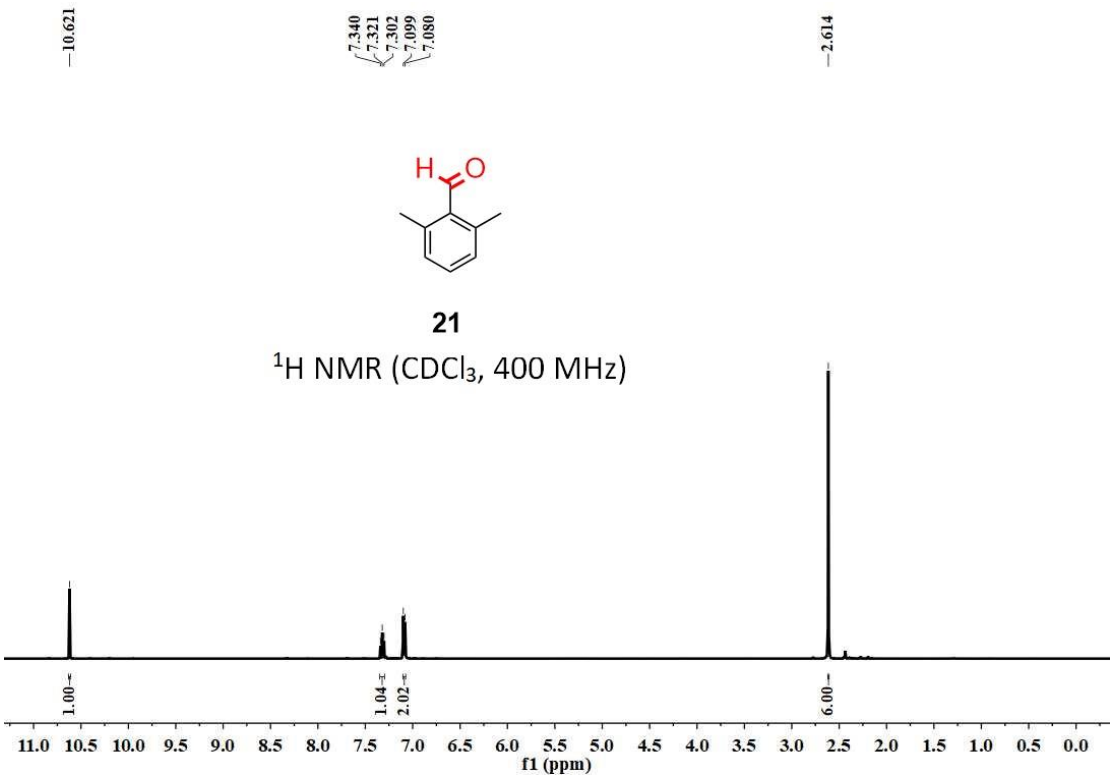
20

^{1}H NMR ($CDCl_3$, 400 MHz)



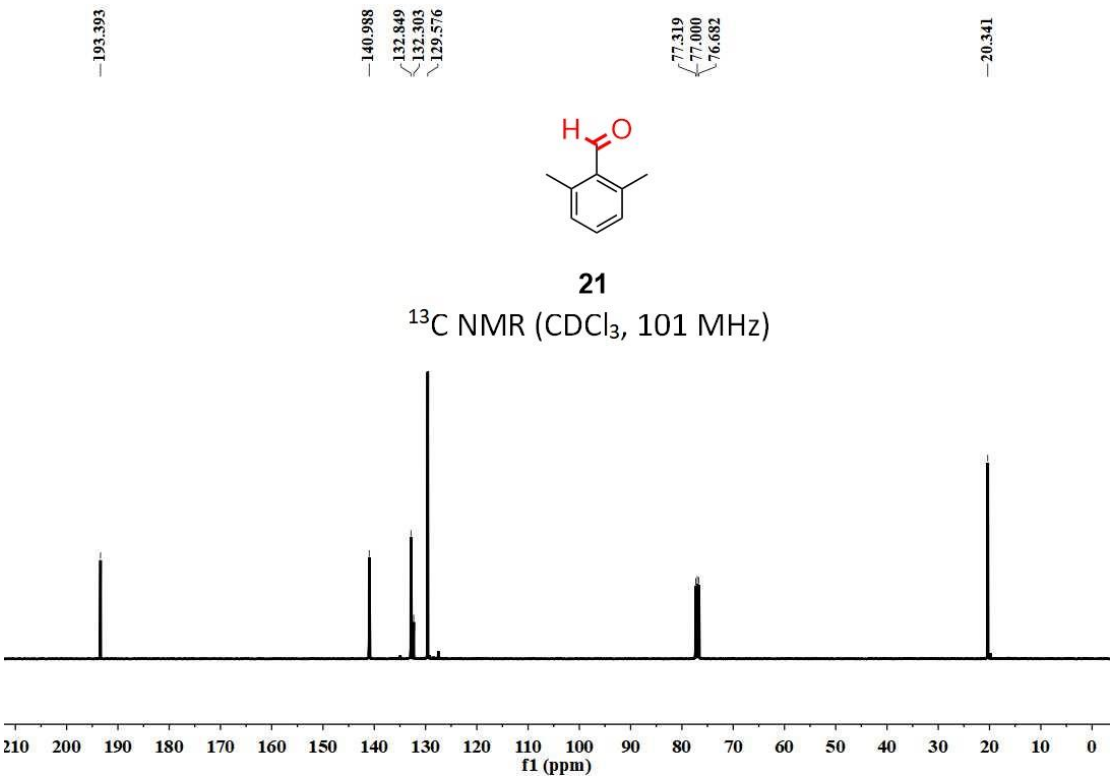
20

^{13}C NMR ($CDCl_3$, 101 MHz)



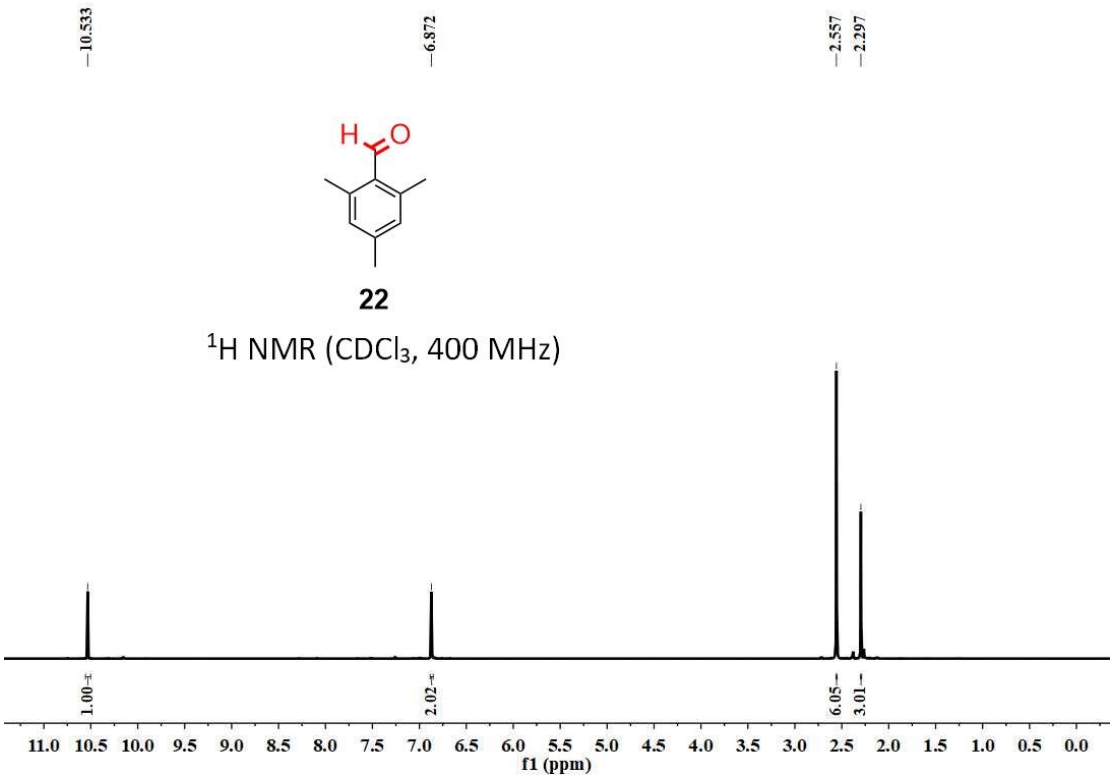
21

^{1}H NMR ($CDCl_3$, 400 MHz)



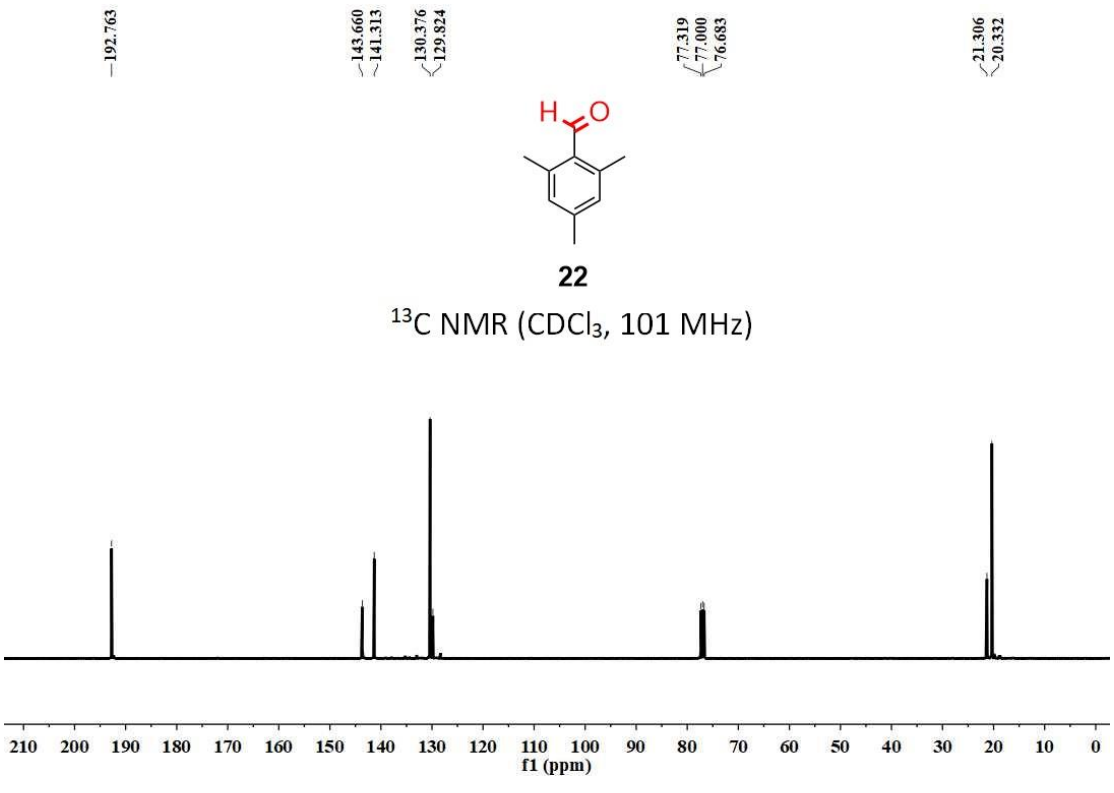
21

^{13}C NMR ($CDCl_3$, 101 MHz)



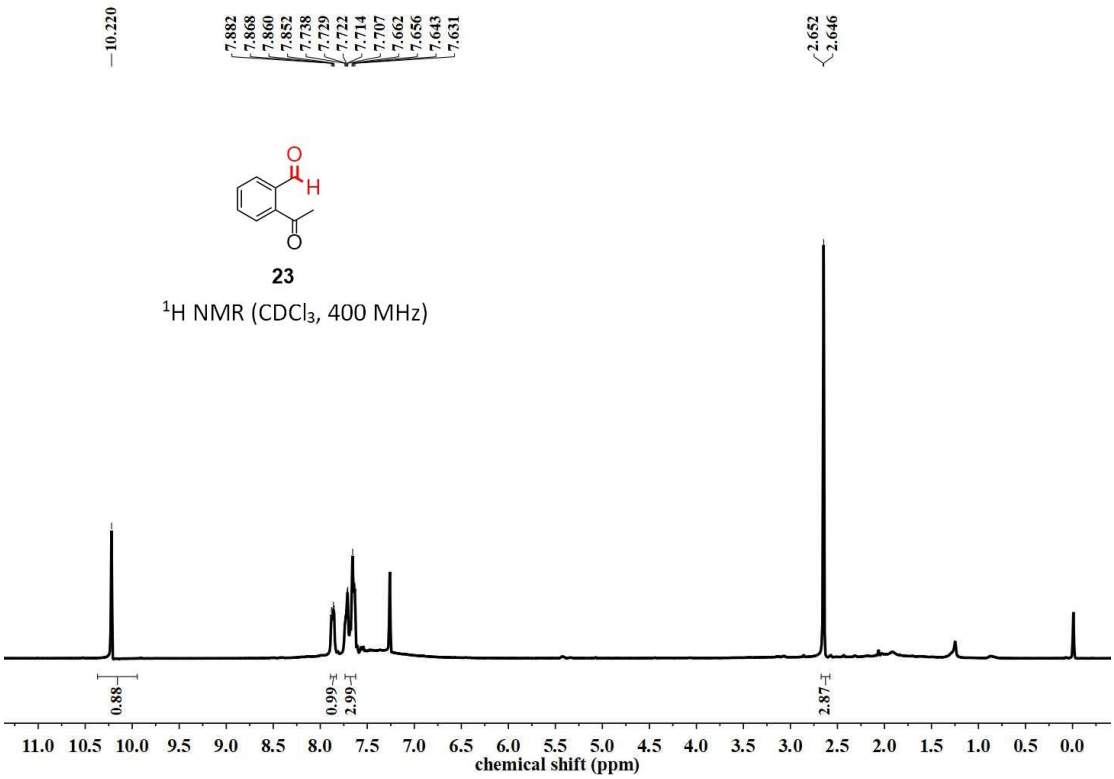
22

^{1}H NMR ($CDCl_3$, 400 MHz)



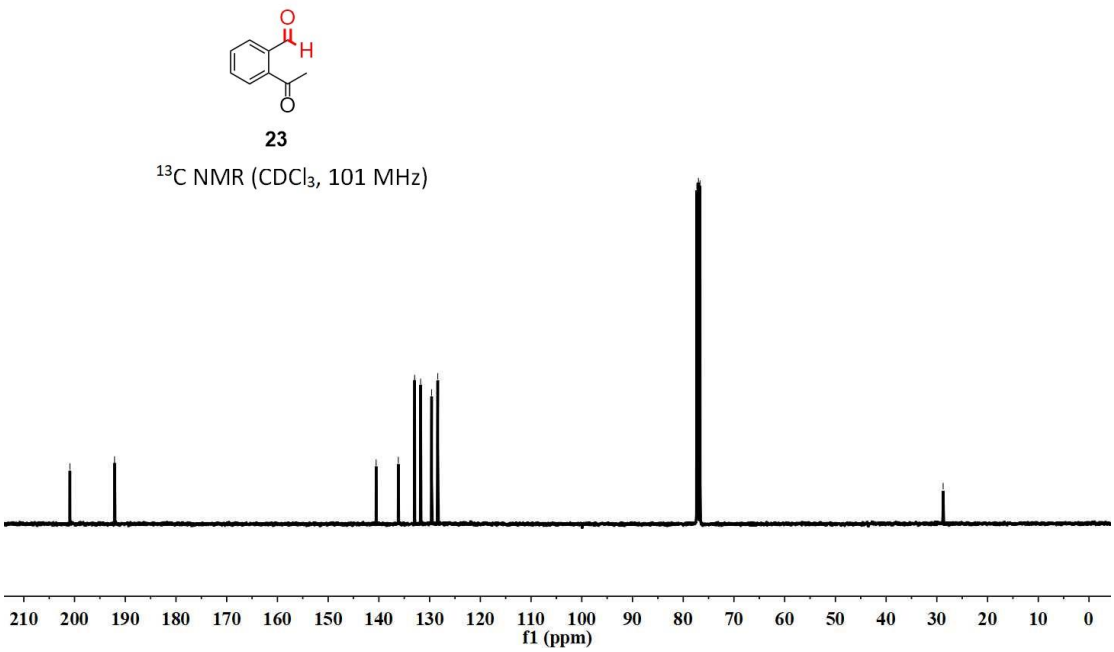
22

^{13}C NMR ($CDCl_3$, 101 MHz)



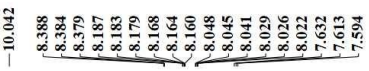
23

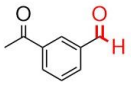
^{1}H NMR ($CDCl_3$, 400 MHz)



23

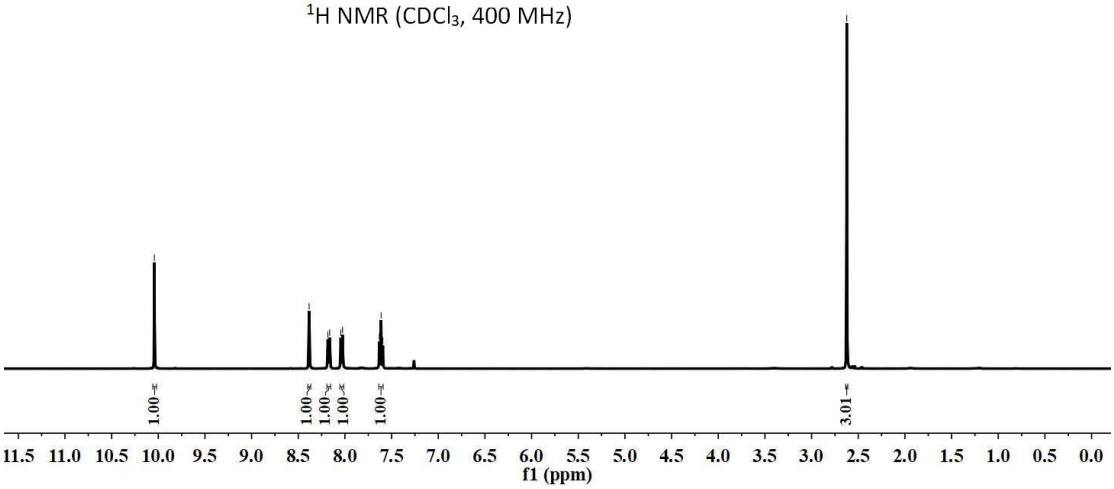
^{13}C NMR ($CDCl_3$, 101 MHz)

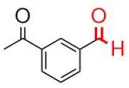




24

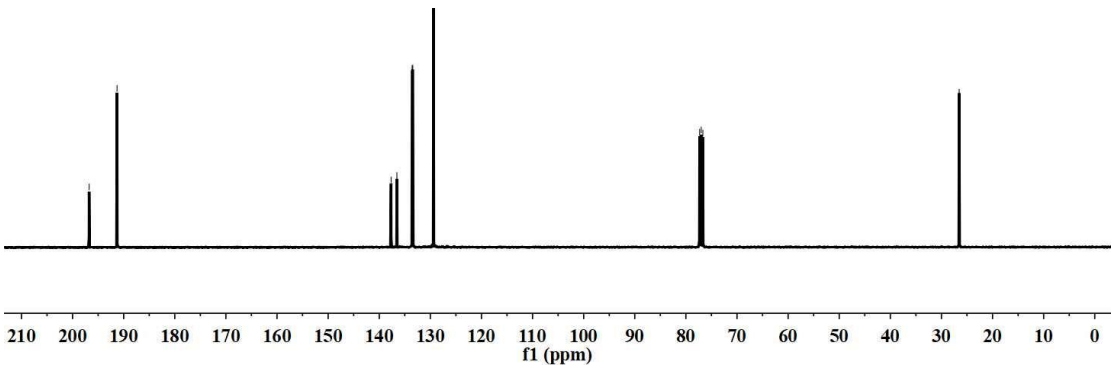
^{1}H NMR ($CDCl_3$, 400 MHz)

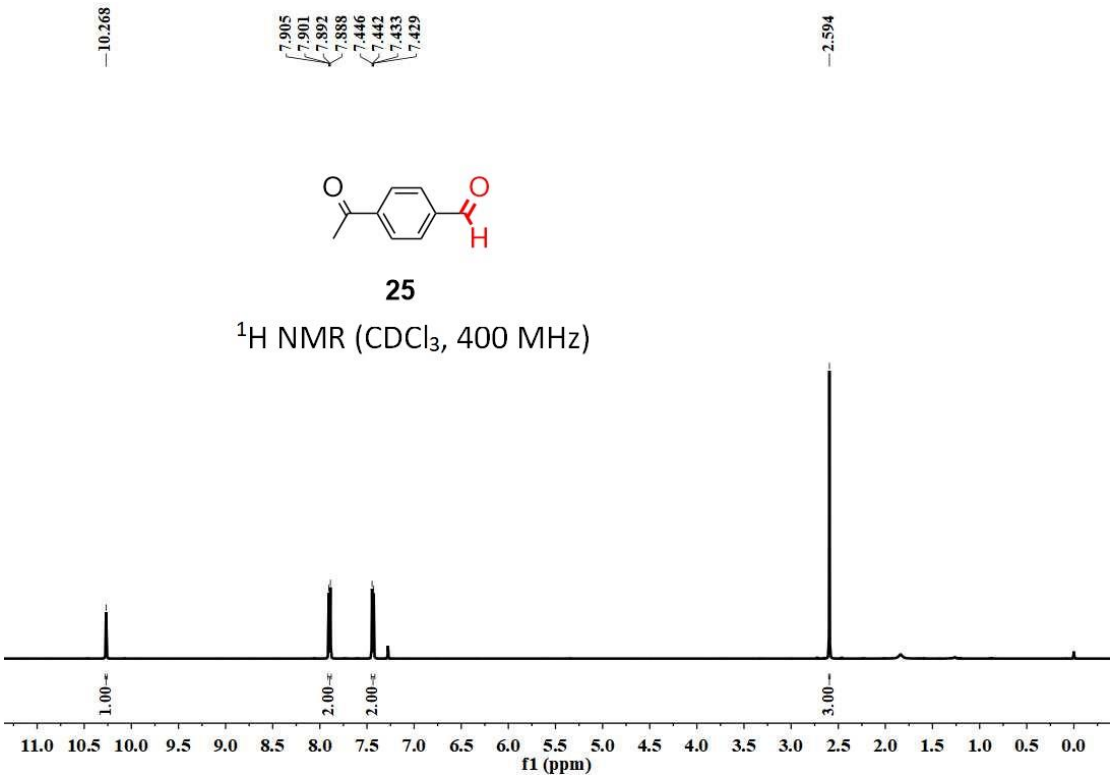




24

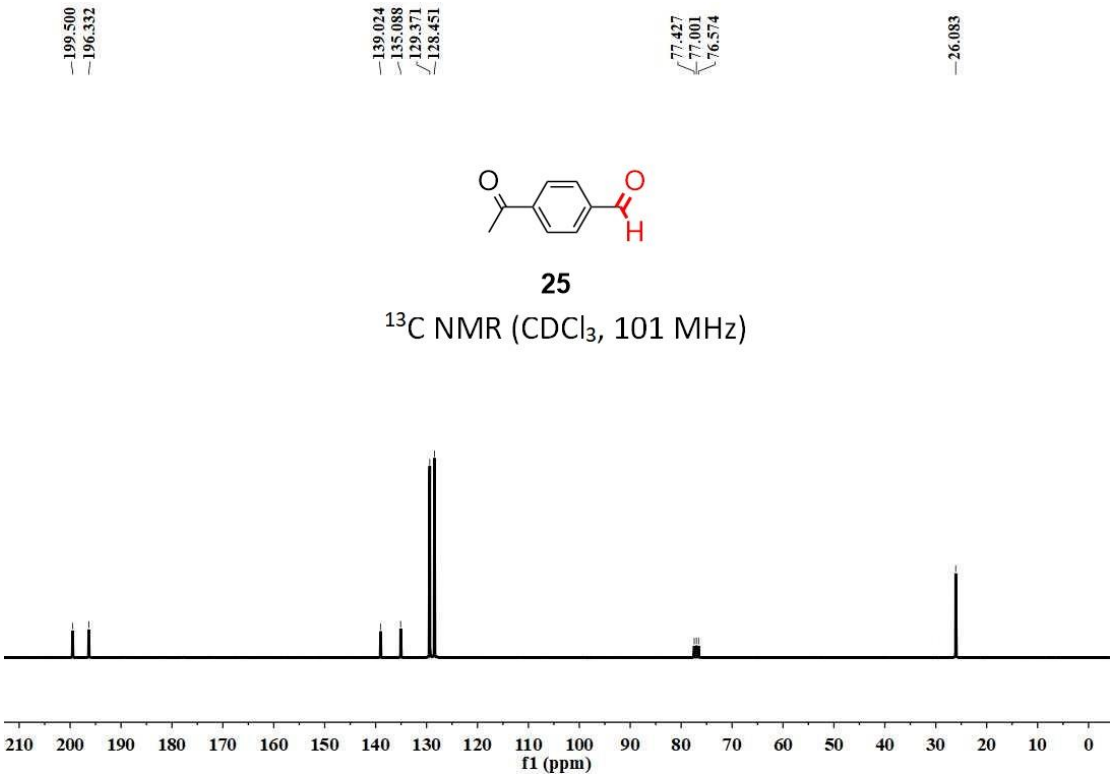
^{13}C NMR ($CDCl_3$, 101 MHz)





25

^{1}H NMR ($CDCl_3$, 400 MHz)



25

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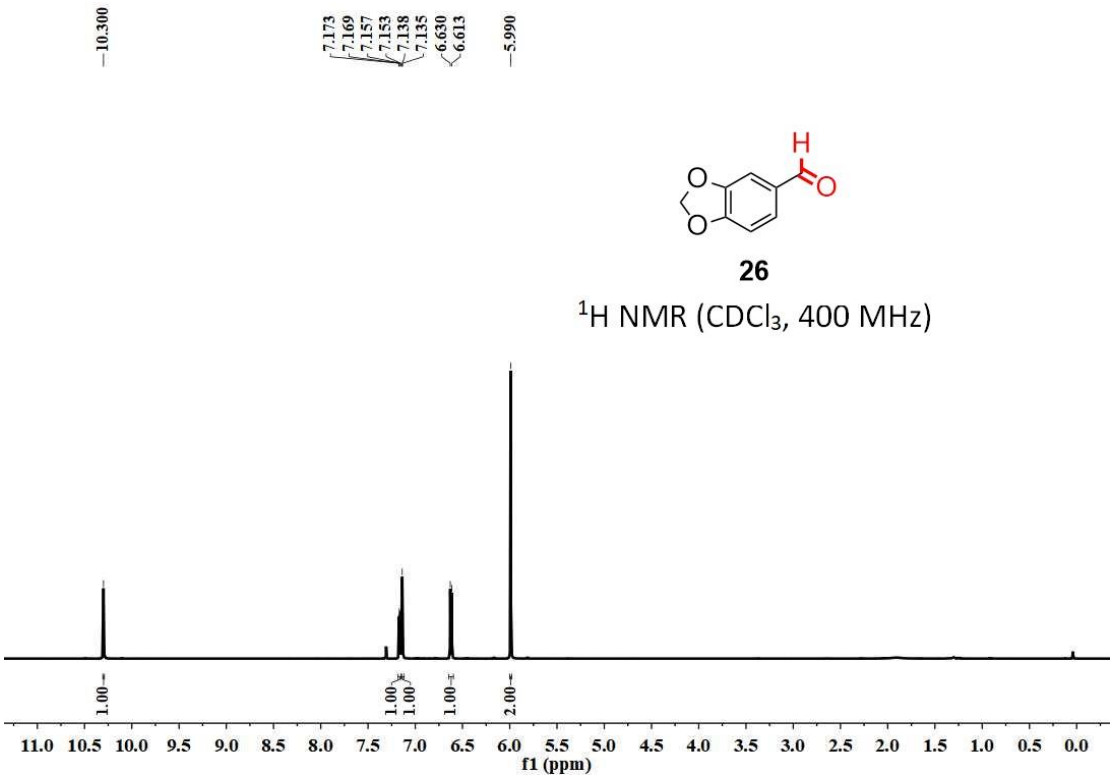
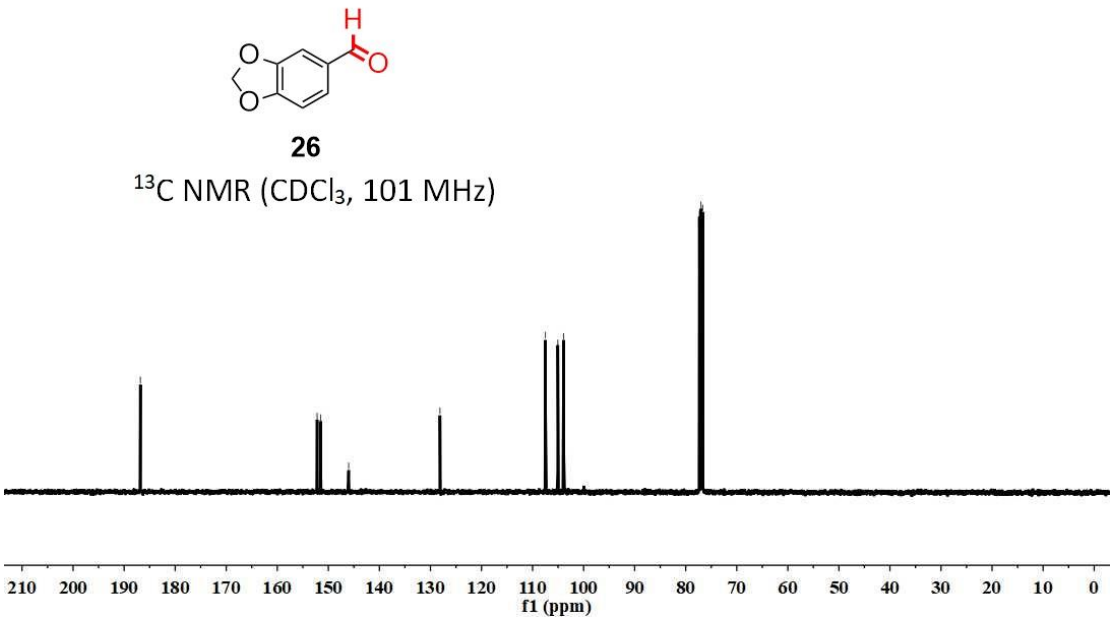


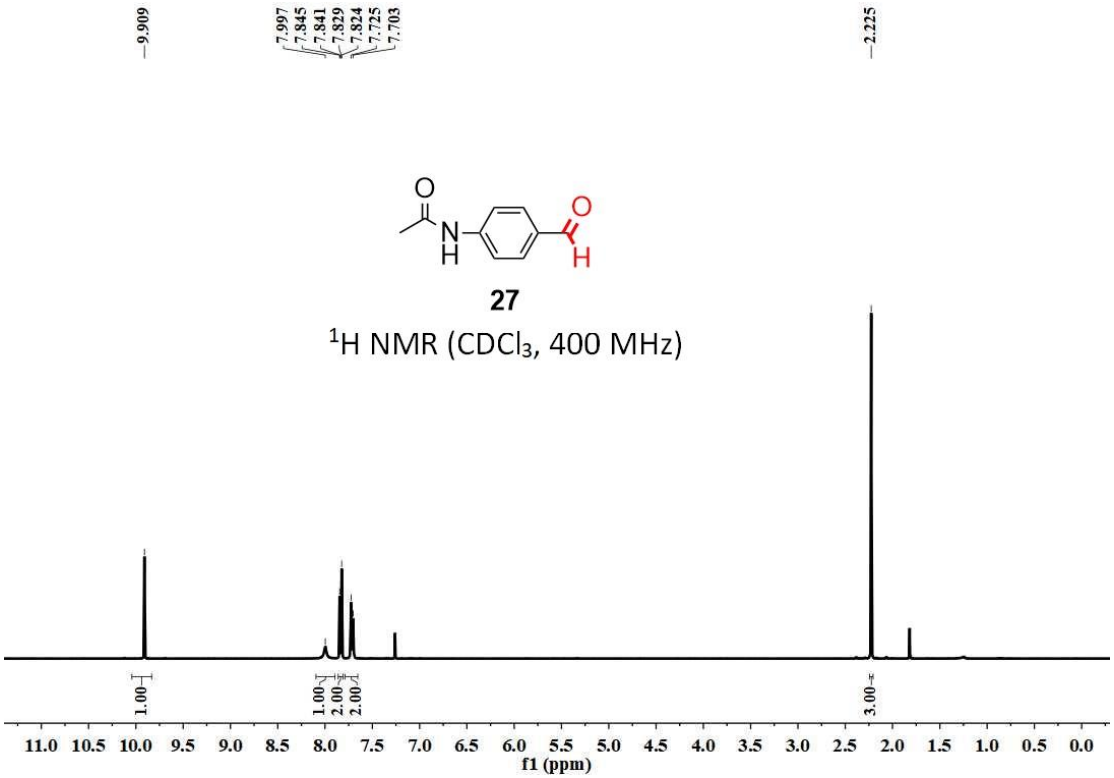
26

^{1}H NMR ($CDCl_3$, 400 MHz)



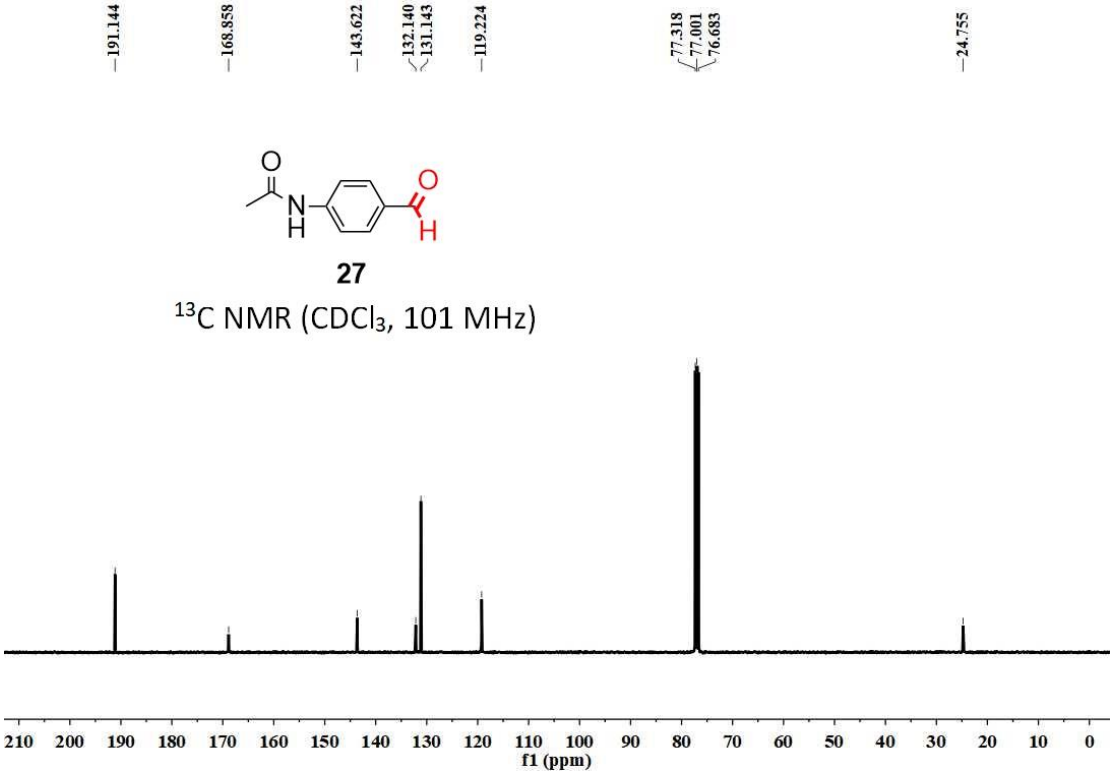
26

^{13}C NMR ($CDCl_3$, 101 MHz)



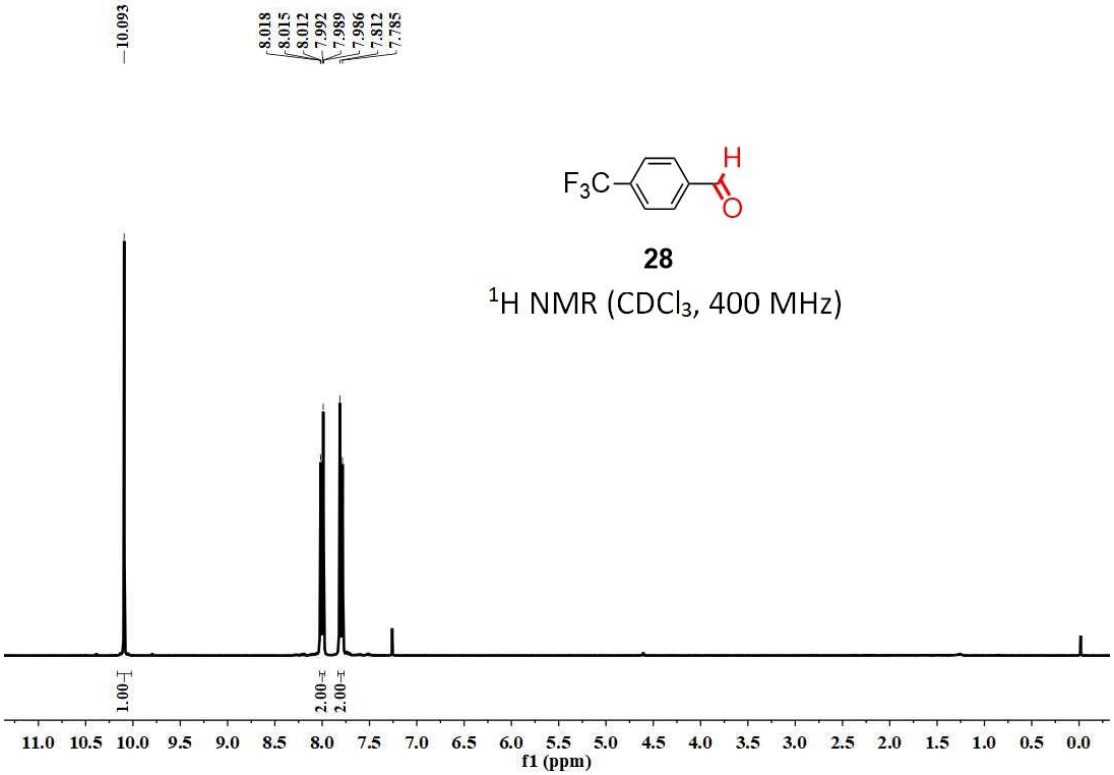
27

^{1}H NMR ($CDCl_3$, 400 MHz)



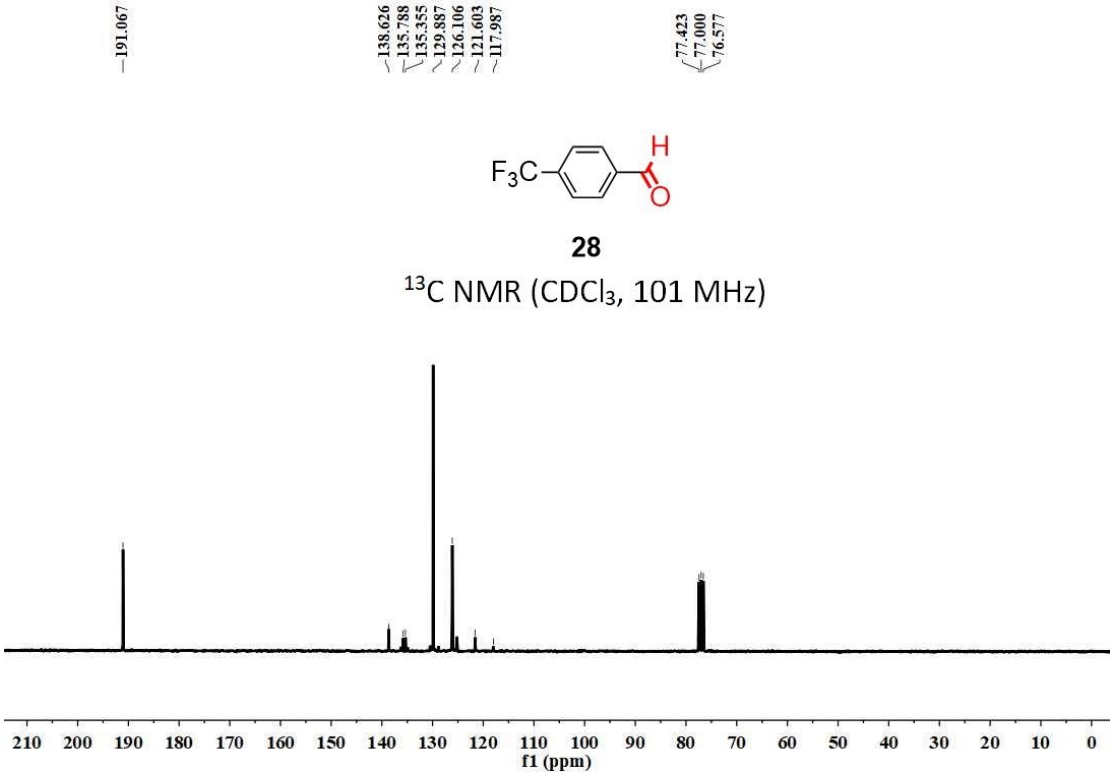
27

^{13}C NMR ($CDCl_3$, 101 MHz)



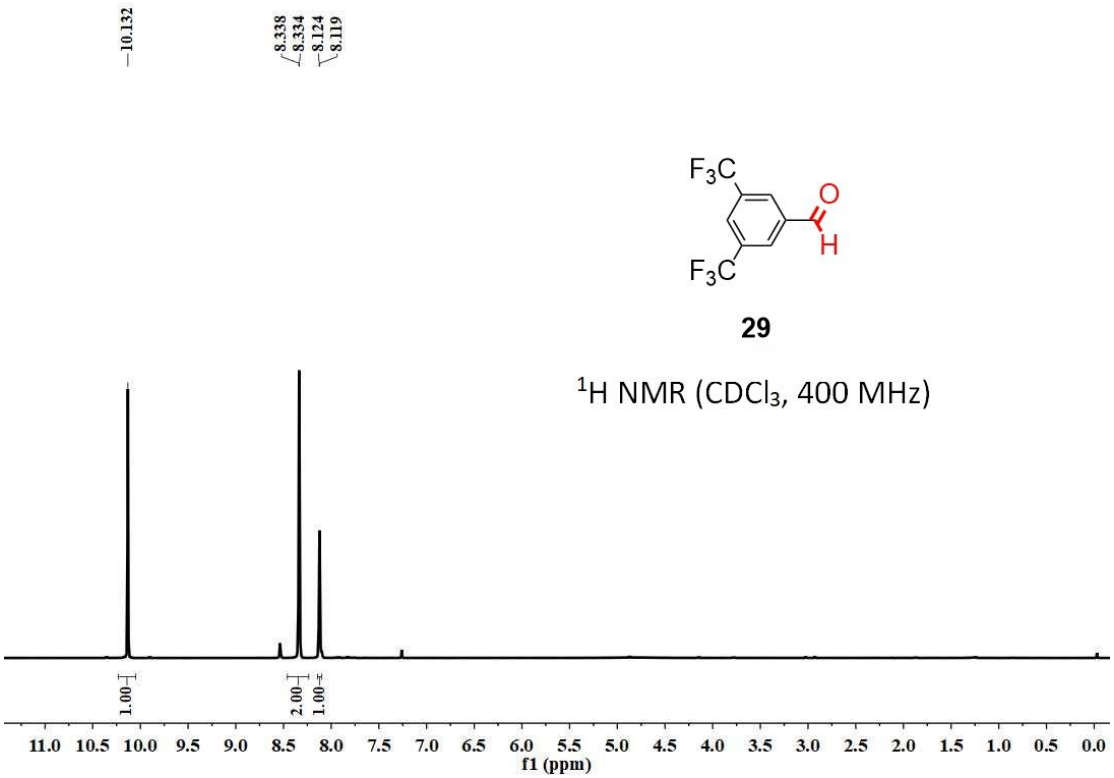
28

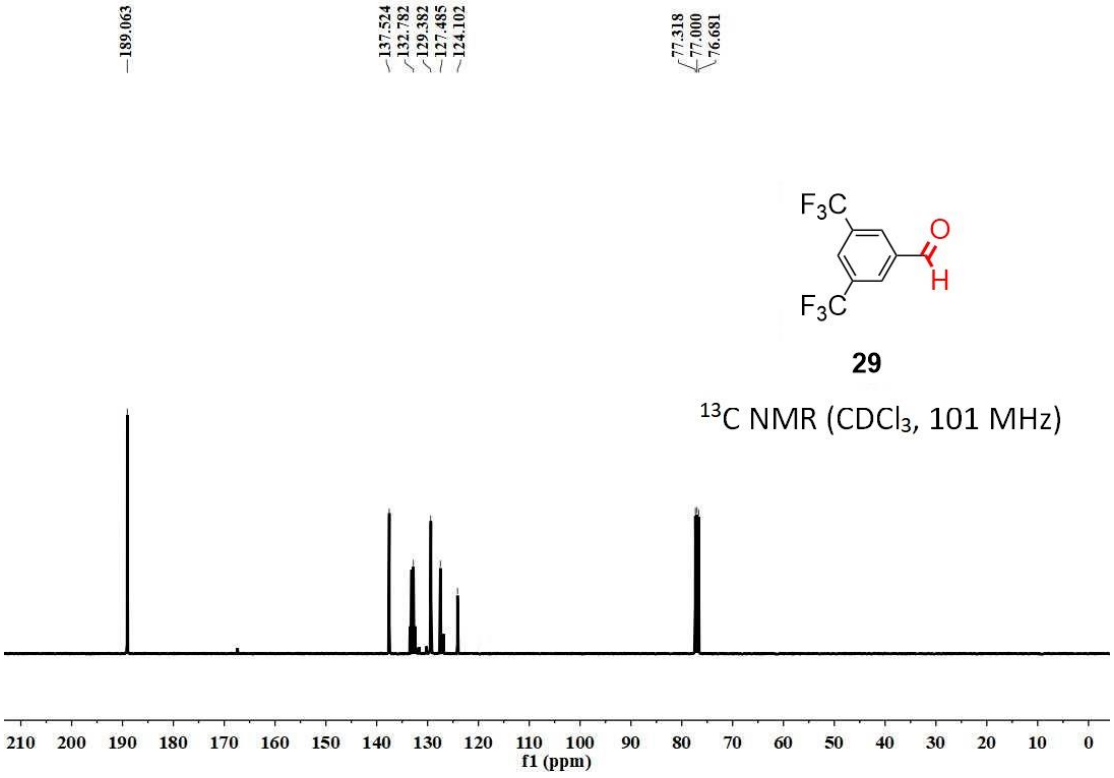
^{1}H NMR ($CDCl_3$, 400 MHz)

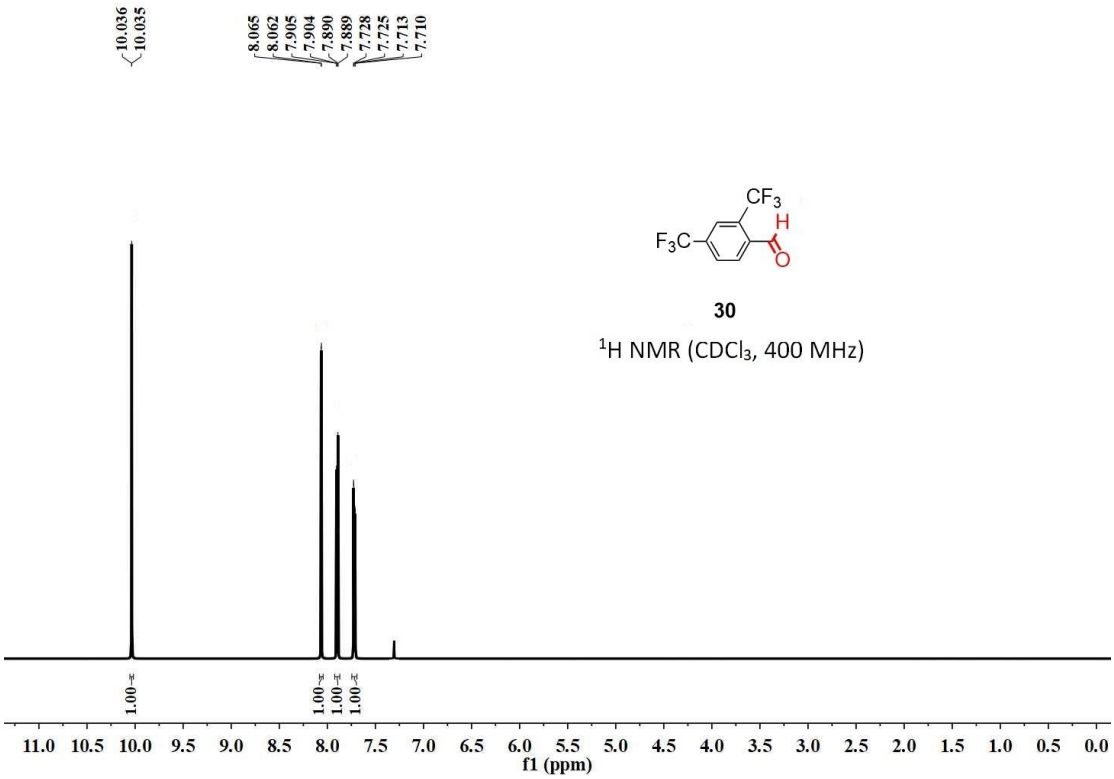


28

^{13}C NMR ($CDCl_3$, 101 MHz)

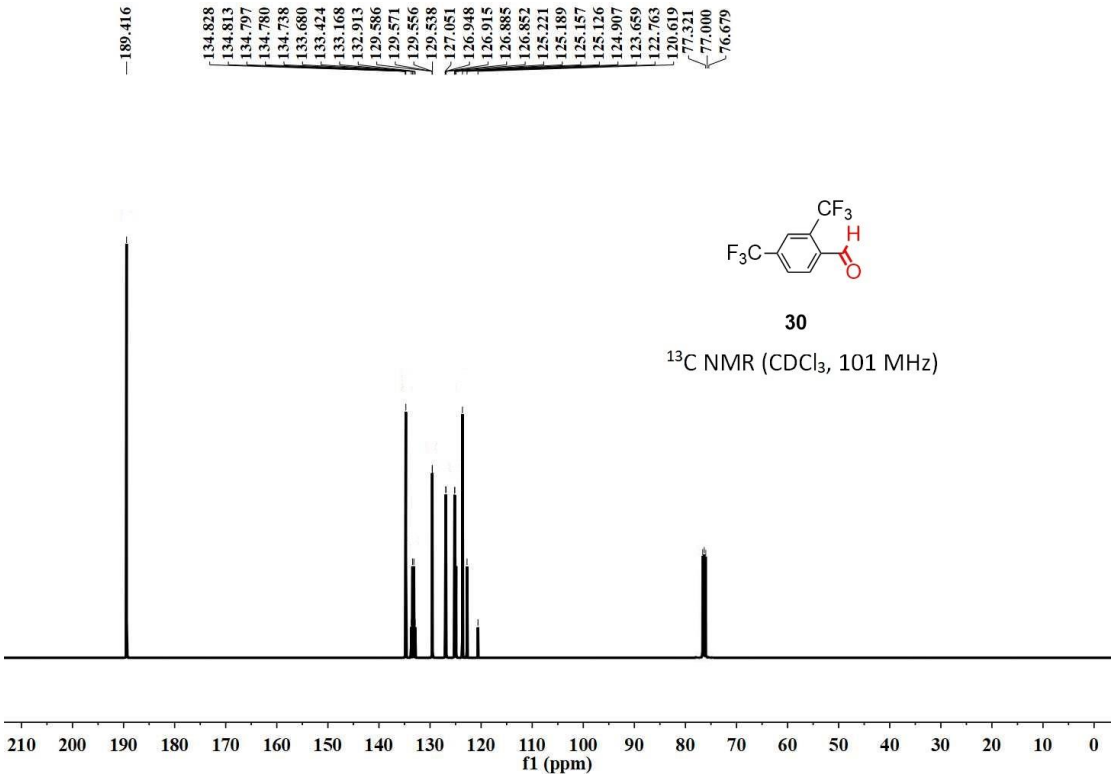

10.132
8.338
8.334
8.124
8.119
F3C
O
H
F3C
29
1H NMR (CDCl3, 400 MHz)
1.00
2.00
1.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


189.063
137.524
132.782
129.382
127.485
124.102
77.318
77.000
76.681
F3C
O
H
F3C
29
13C NMR (CDCl3, 101 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



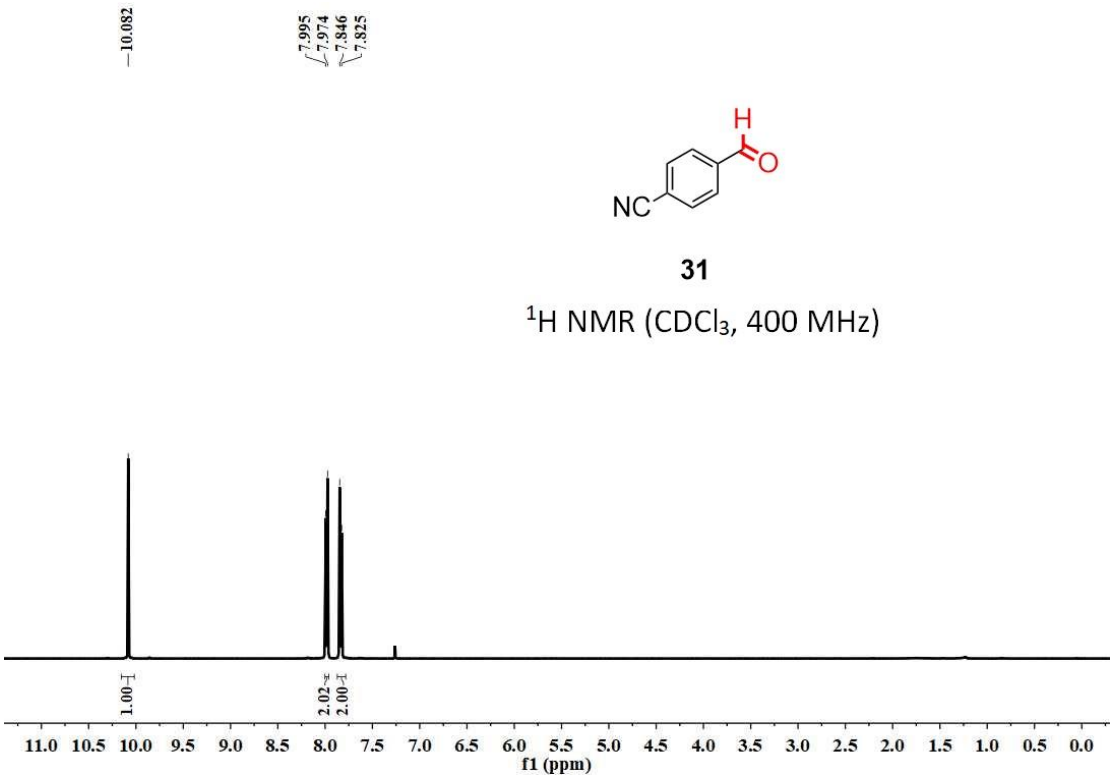
30

^{1}H NMR ($CDCl_3$, 400 MHz)



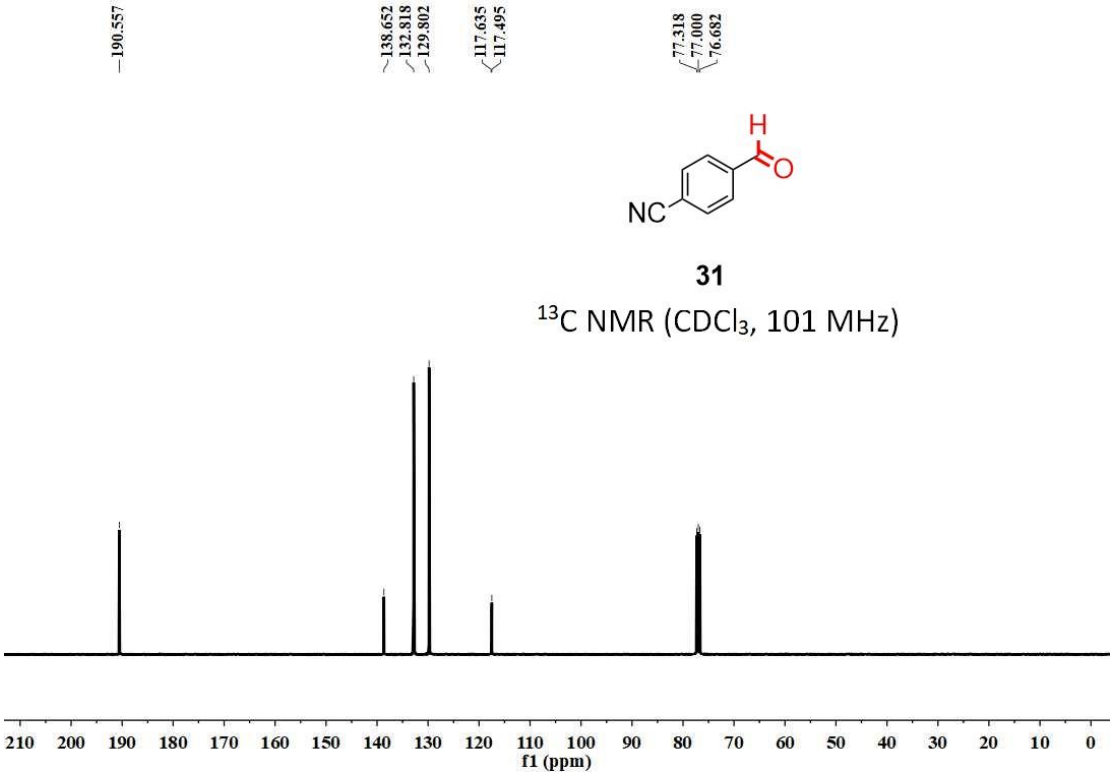
30

^{13}C NMR ($CDCl_3$, 101 MHz)



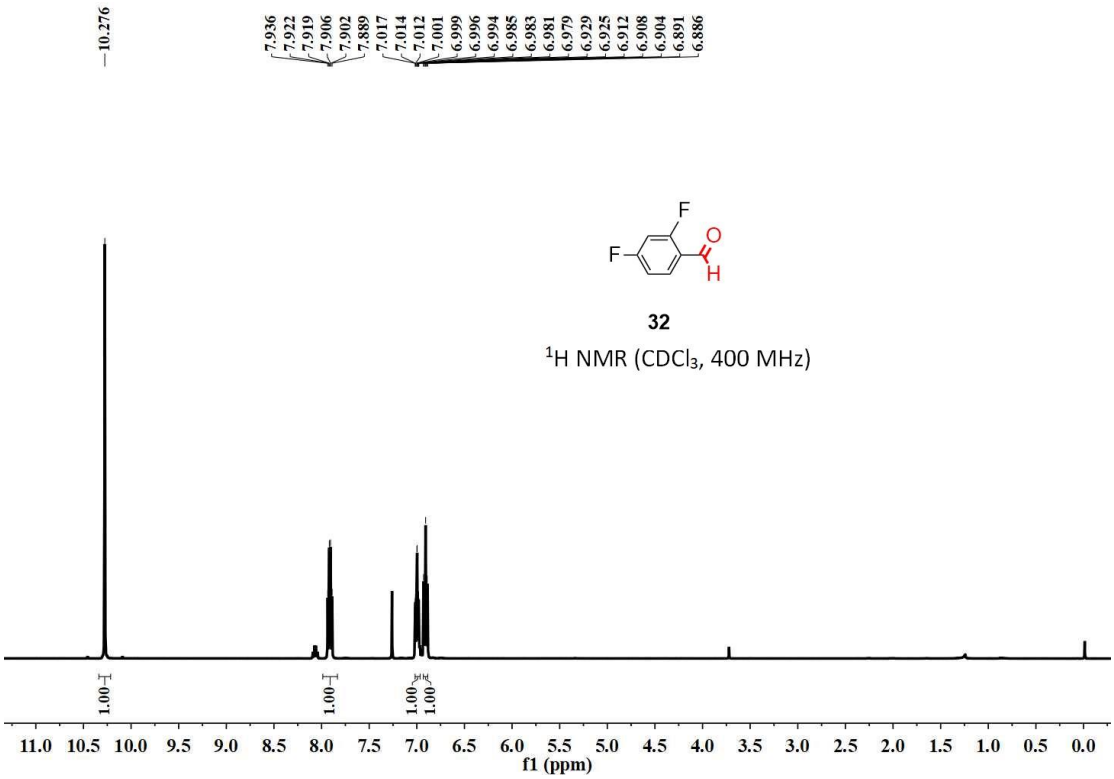
31

^{1}H NMR ($CDCl_3$, 400 MHz)



31

^{13}C NMR ($CDCl_3$, 101 MHz)



32

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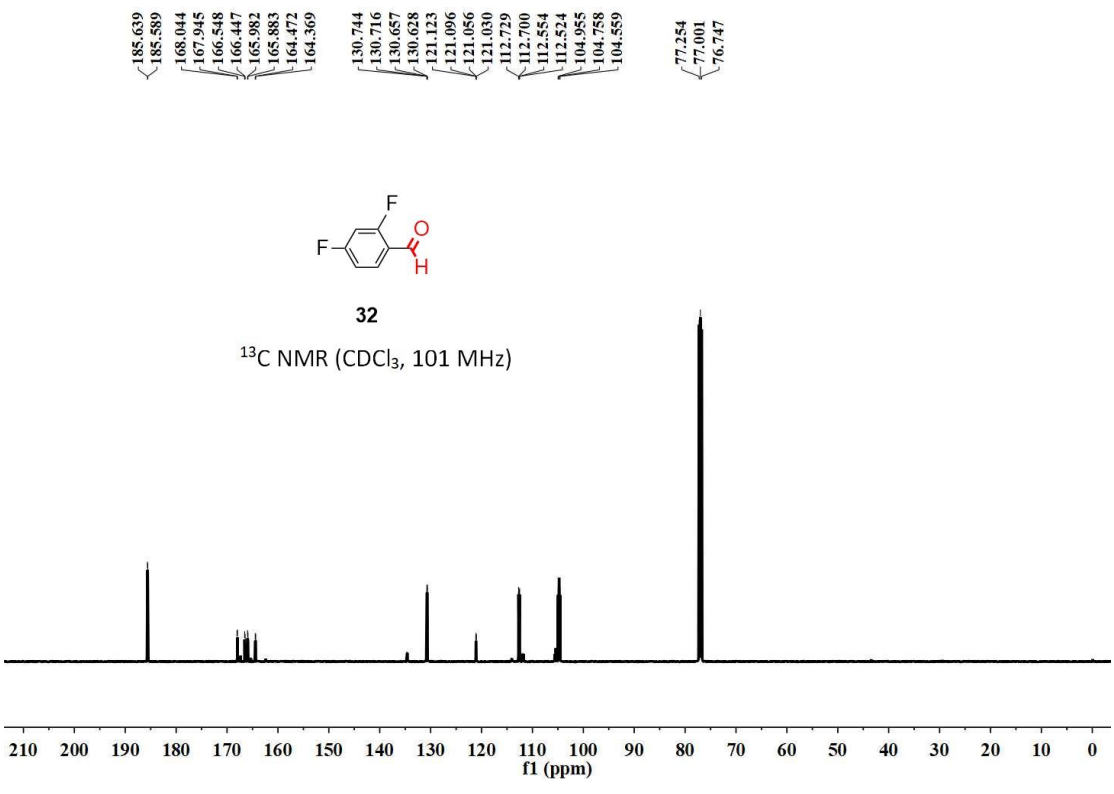
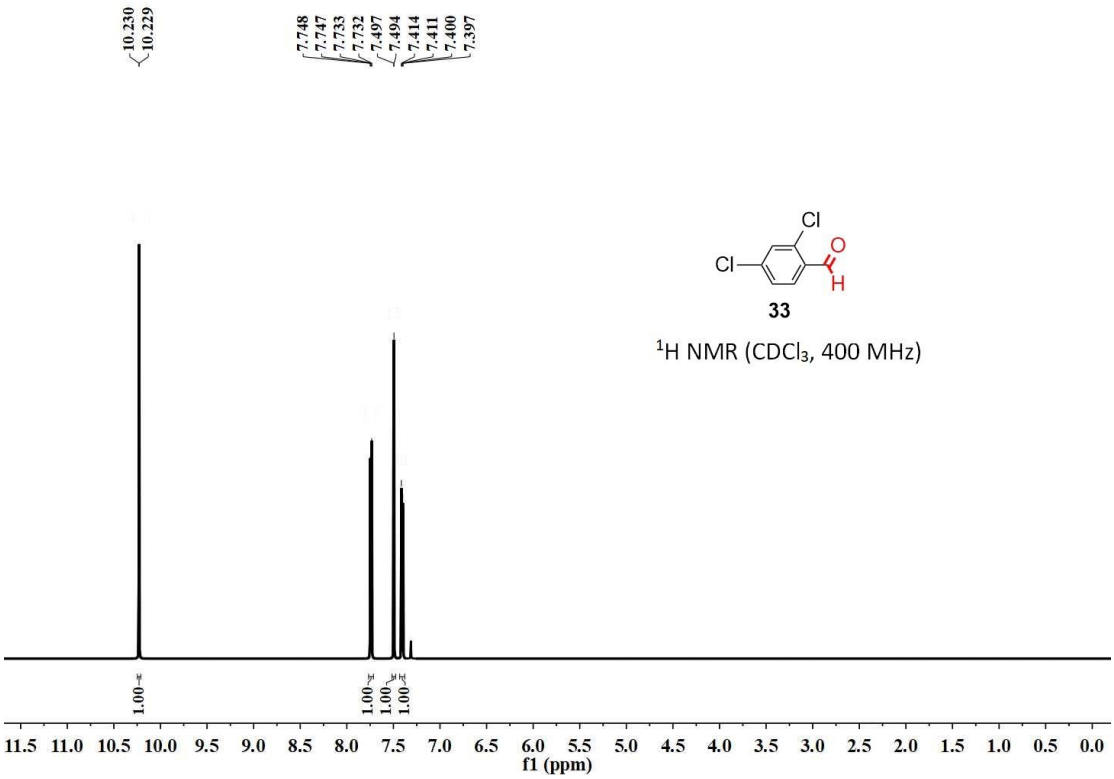


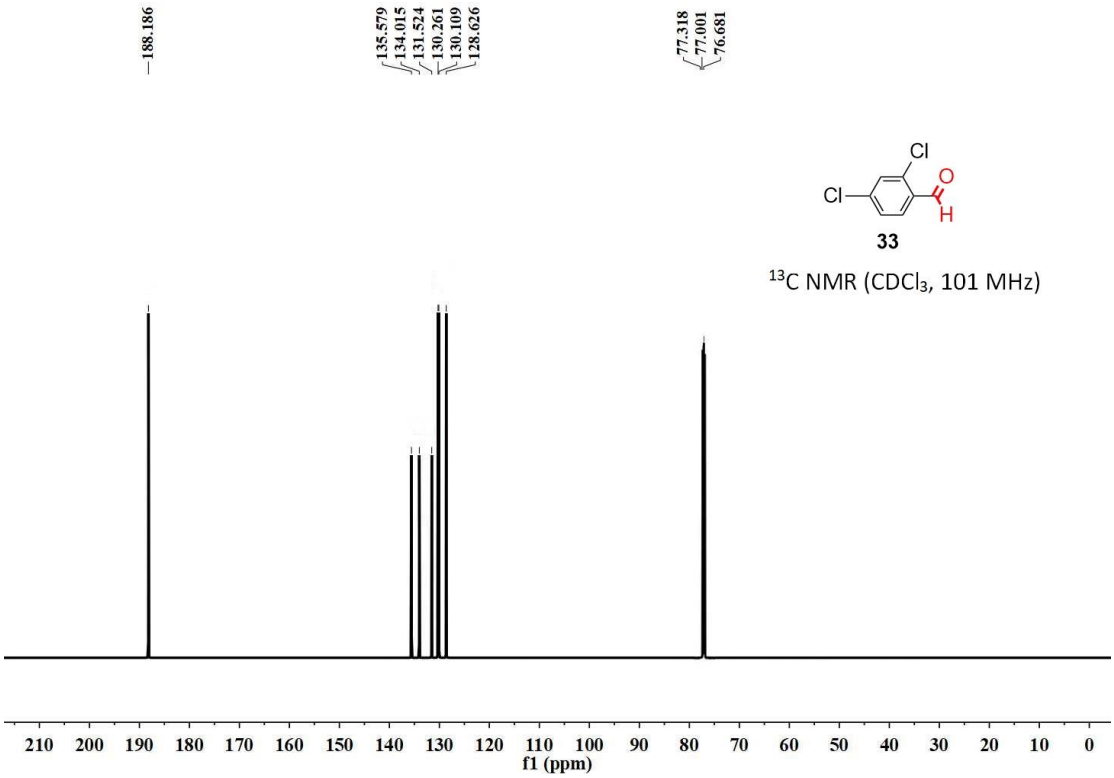
32

^{13}C NMR ($CDCl_3$, 101 MHz)



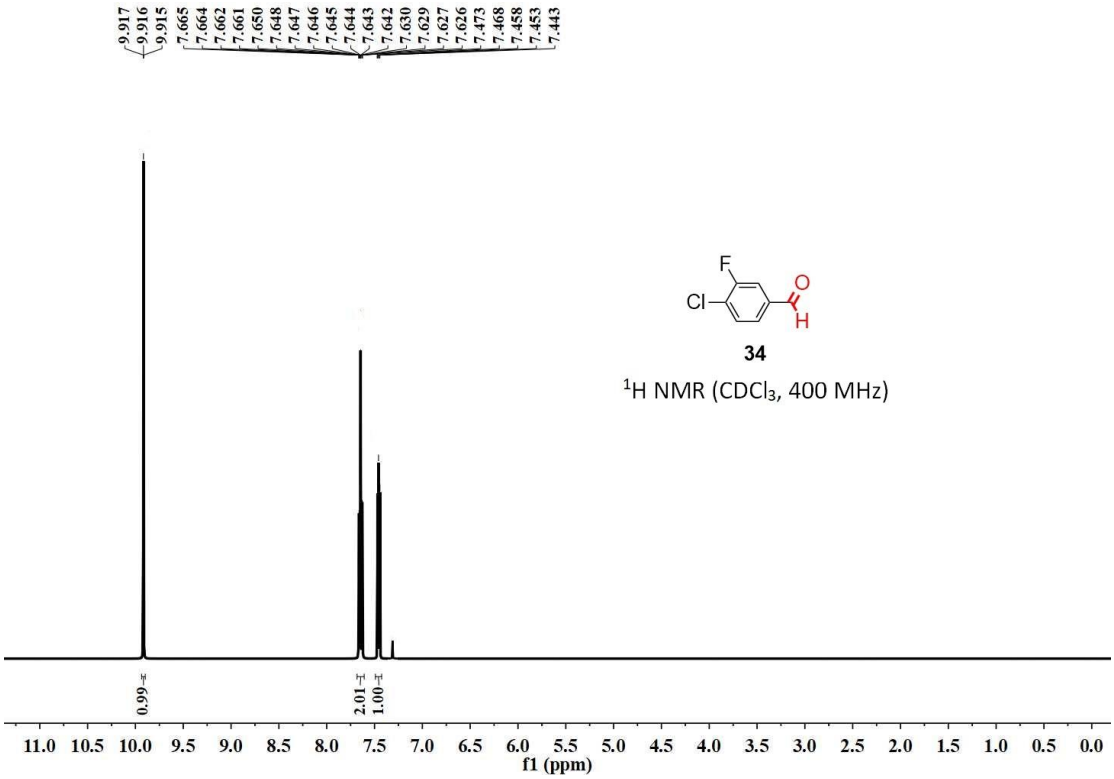
33

^{1}H NMR ($CDCl_3$, 400 MHz)



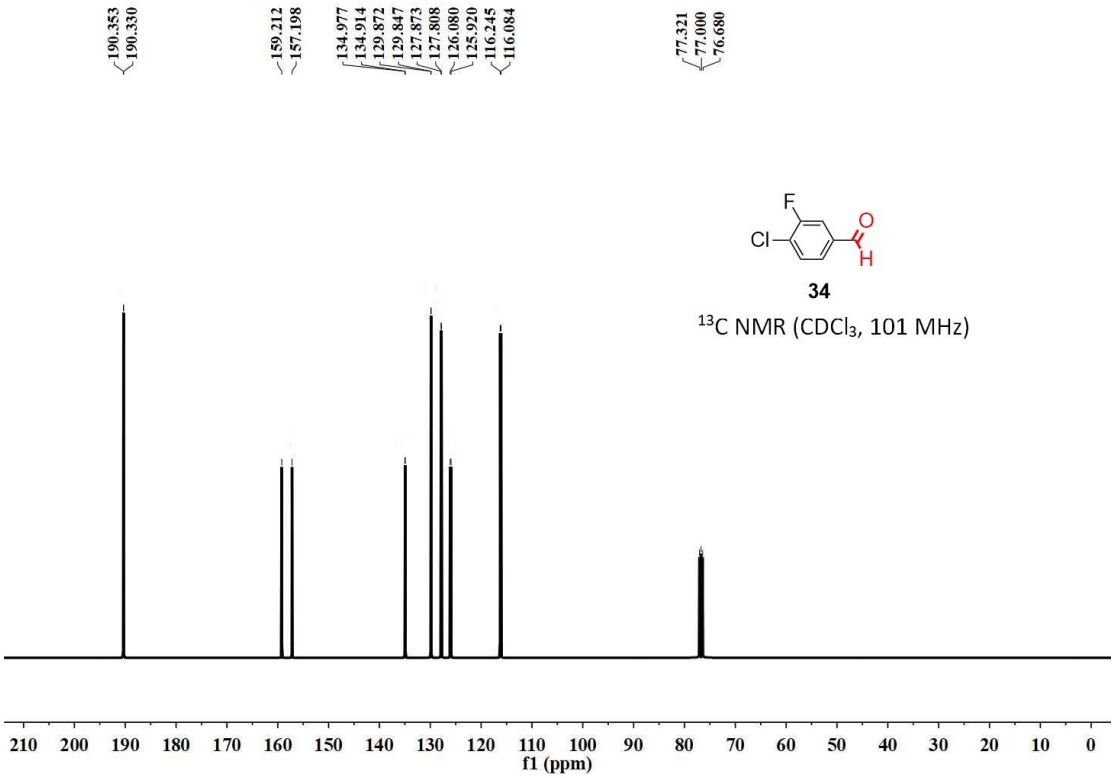
33

^{13}C NMR ($CDCl_3$, 101 MHz)



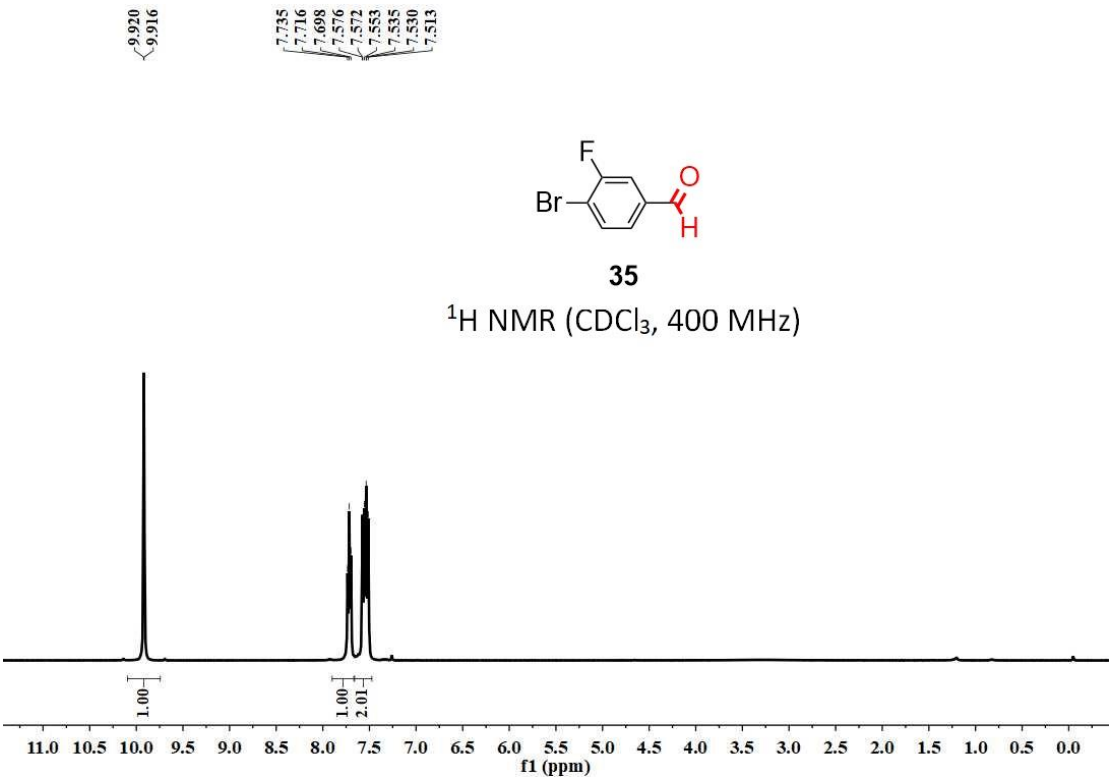
34

^{1}H NMR ($CDCl_3$, 400 MHz)



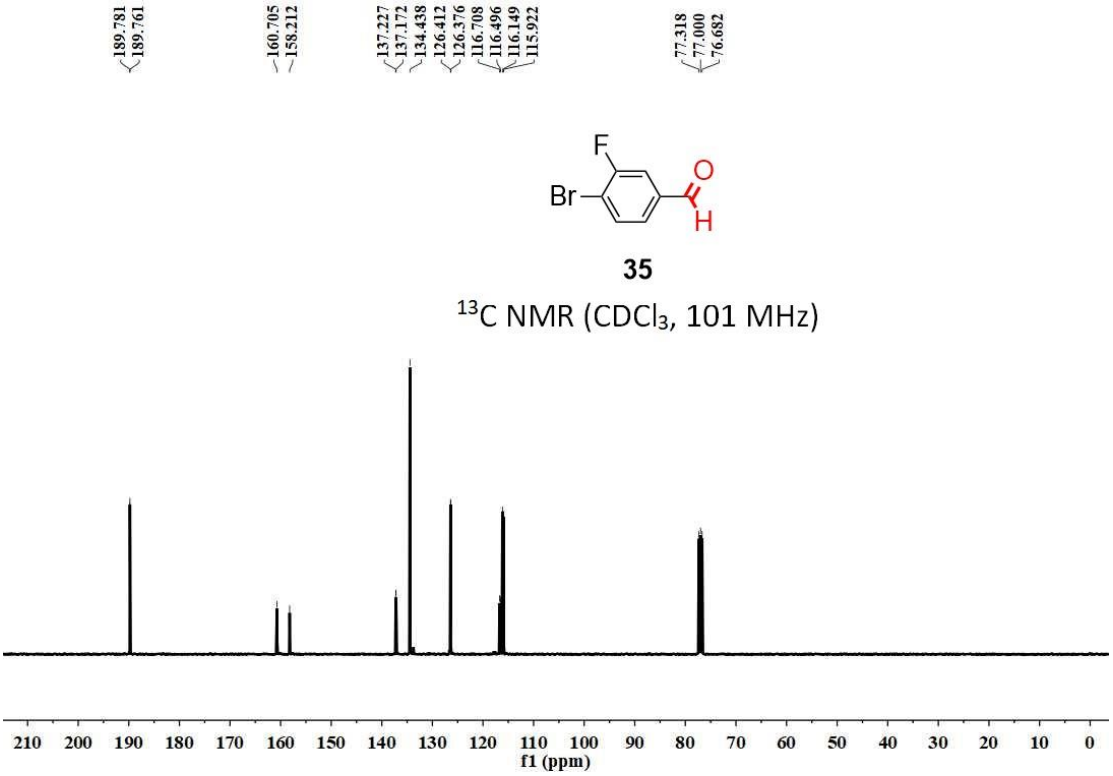
34

^{13}C NMR ($CDCl_3$, 101 MHz)



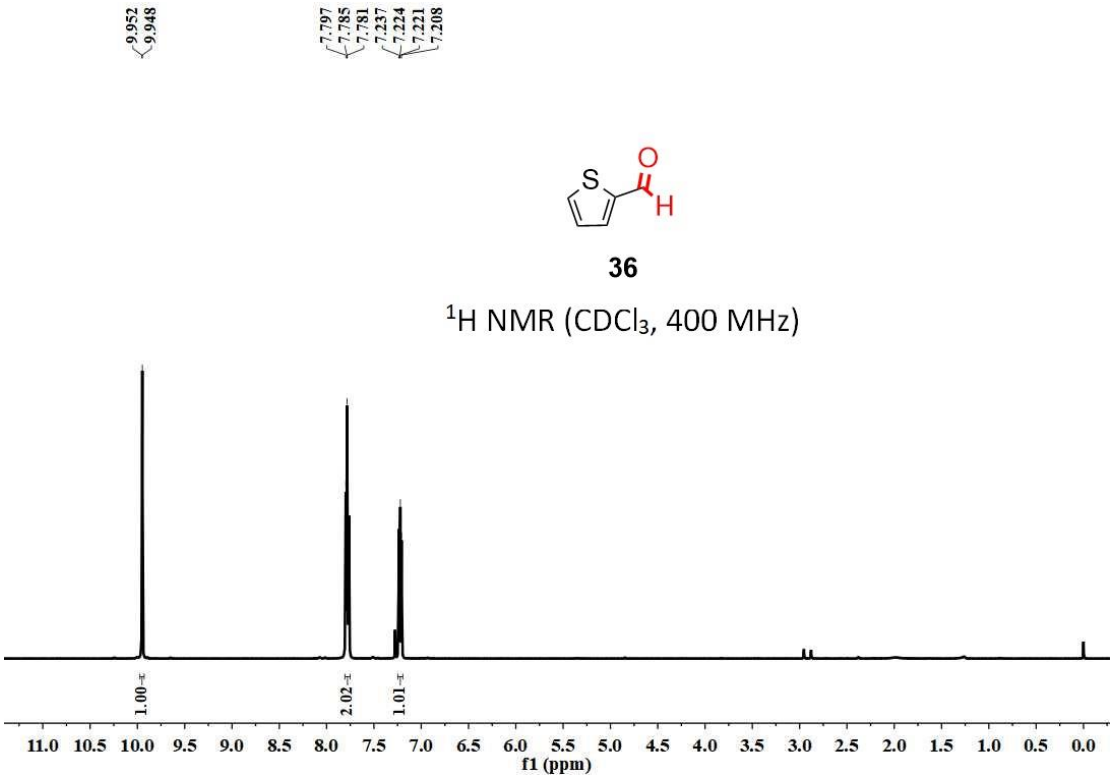
35

^{1}H NMR ($CDCl_3$, 400 MHz)



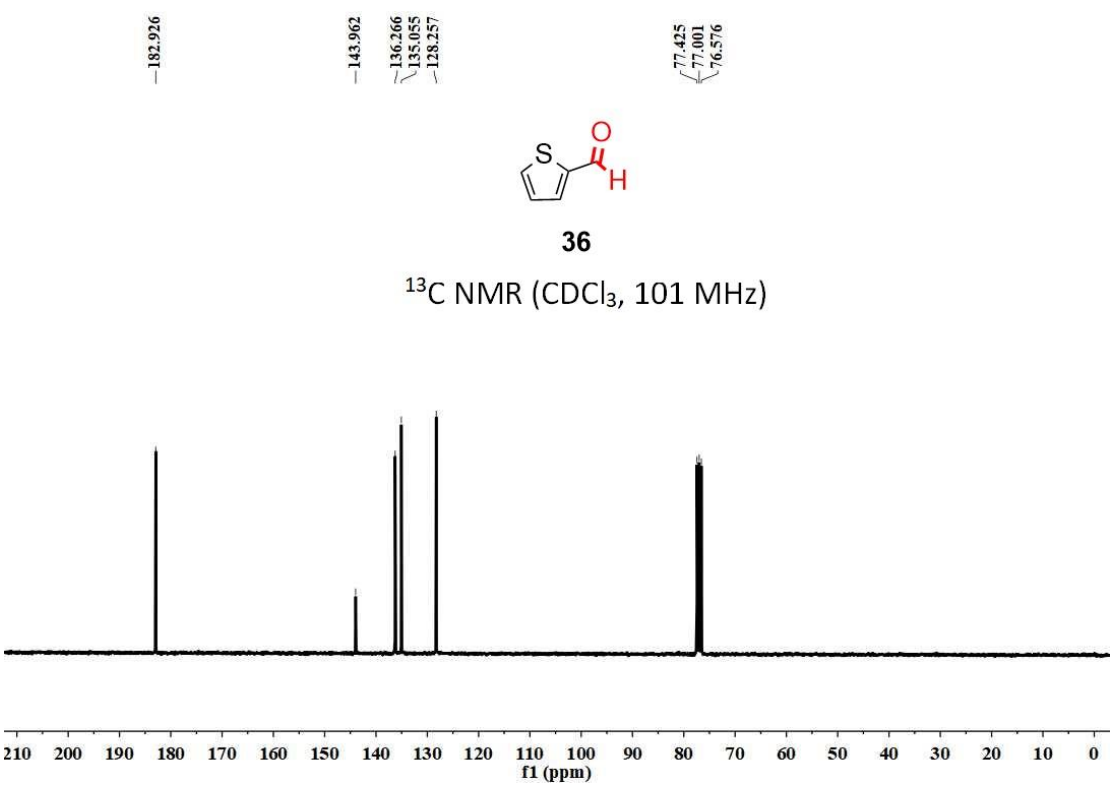
35

^{13}C NMR ($CDCl_3$, 101 MHz)



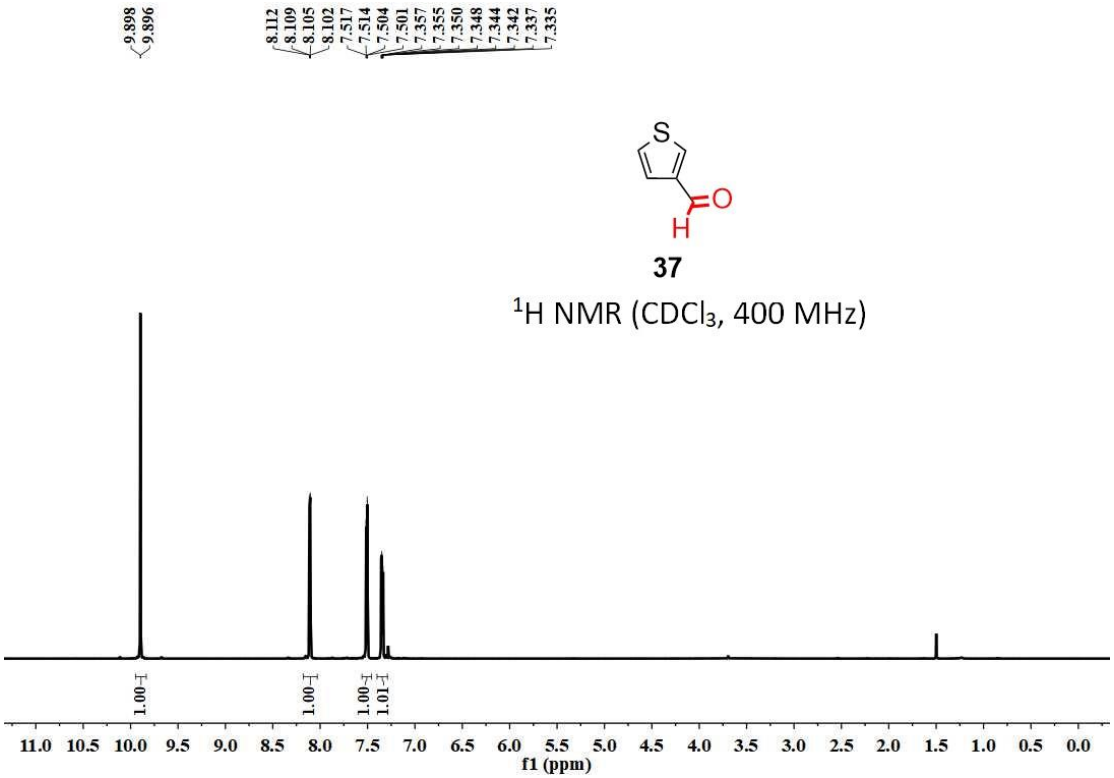
36

^{1}H NMR ($CDCl_3$, 400 MHz)



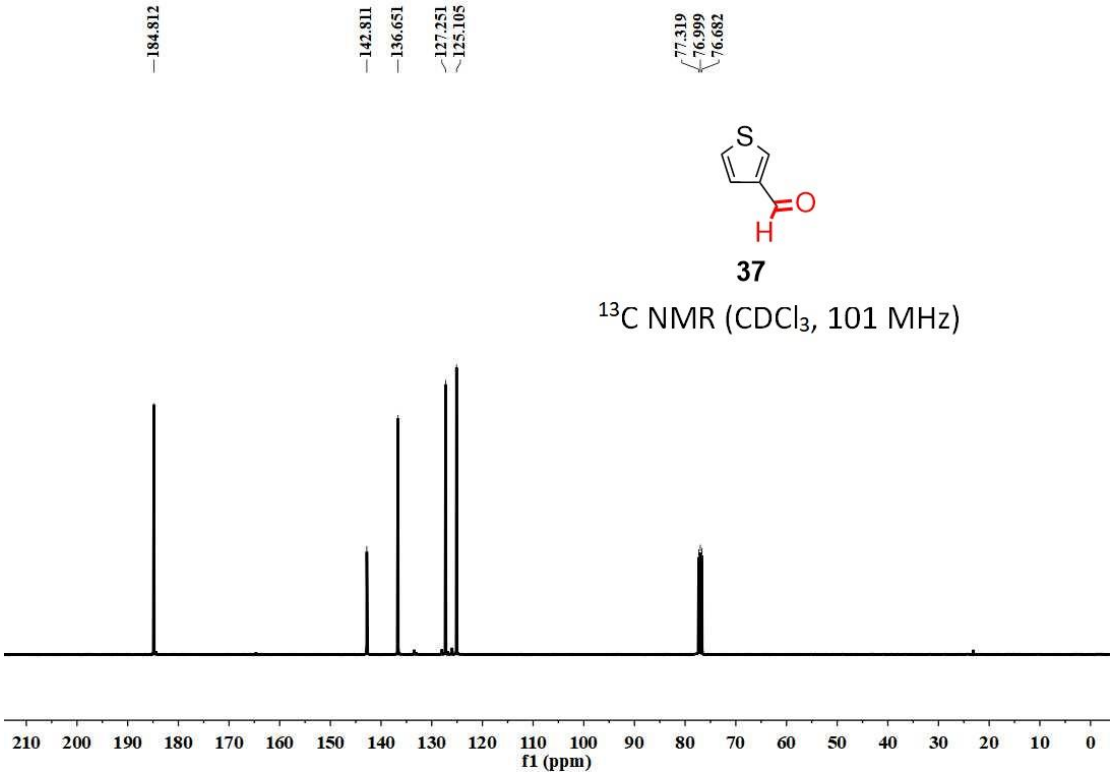
36

^{13}C NMR ($CDCl_3$, 101 MHz)



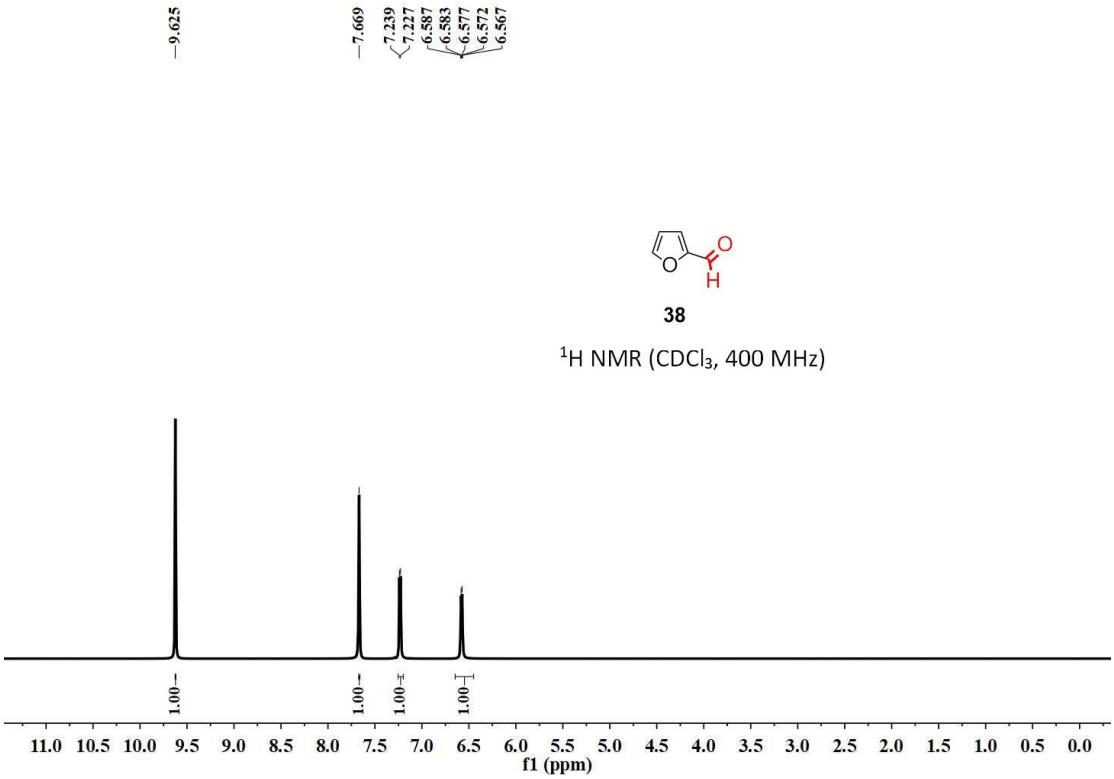
37

^{1}H NMR ($CDCl_3$, 400 MHz)



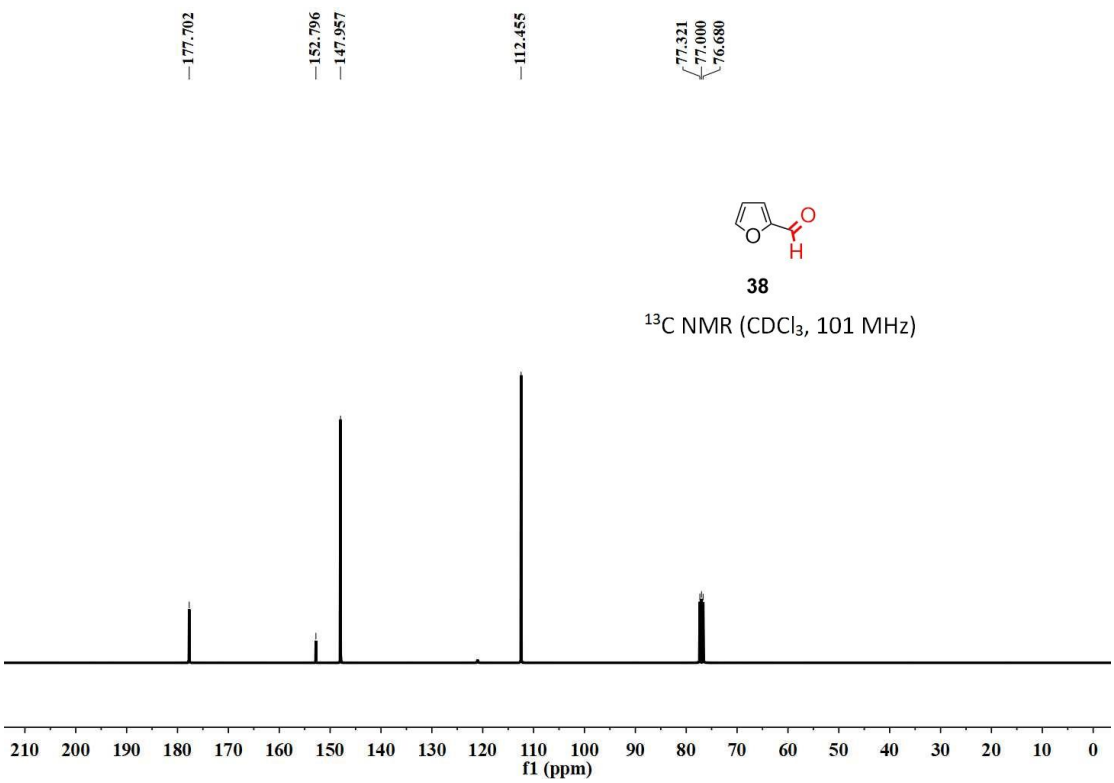
37

^{13}C NMR ($CDCl_3$, 101 MHz)



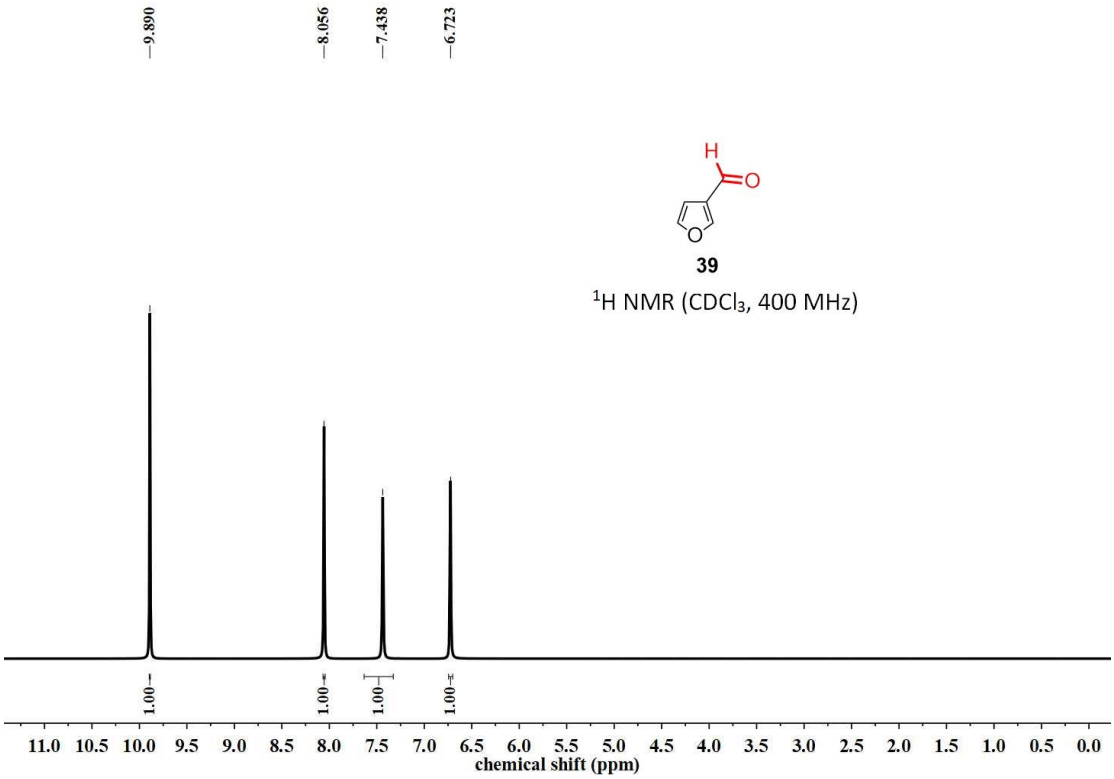
38

^{1}H NMR ($CDCl_3$, 400 MHz)



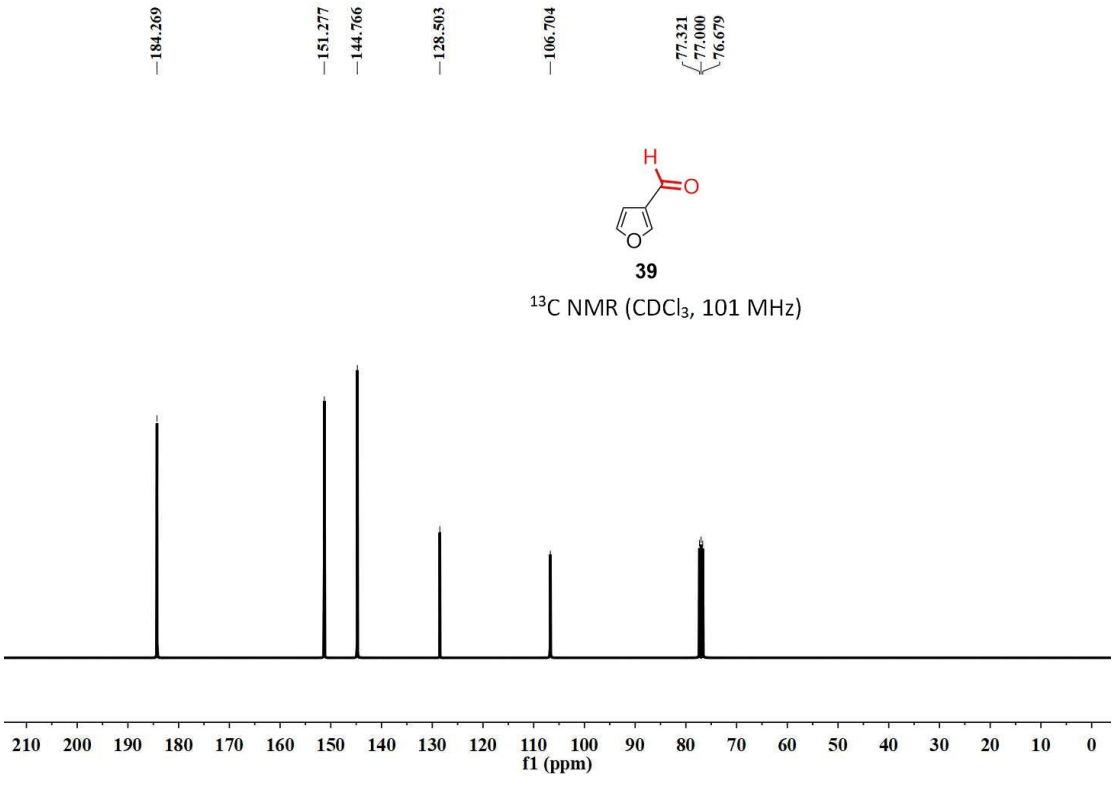
38

^{13}C NMR ($CDCl_3$, 101 MHz)



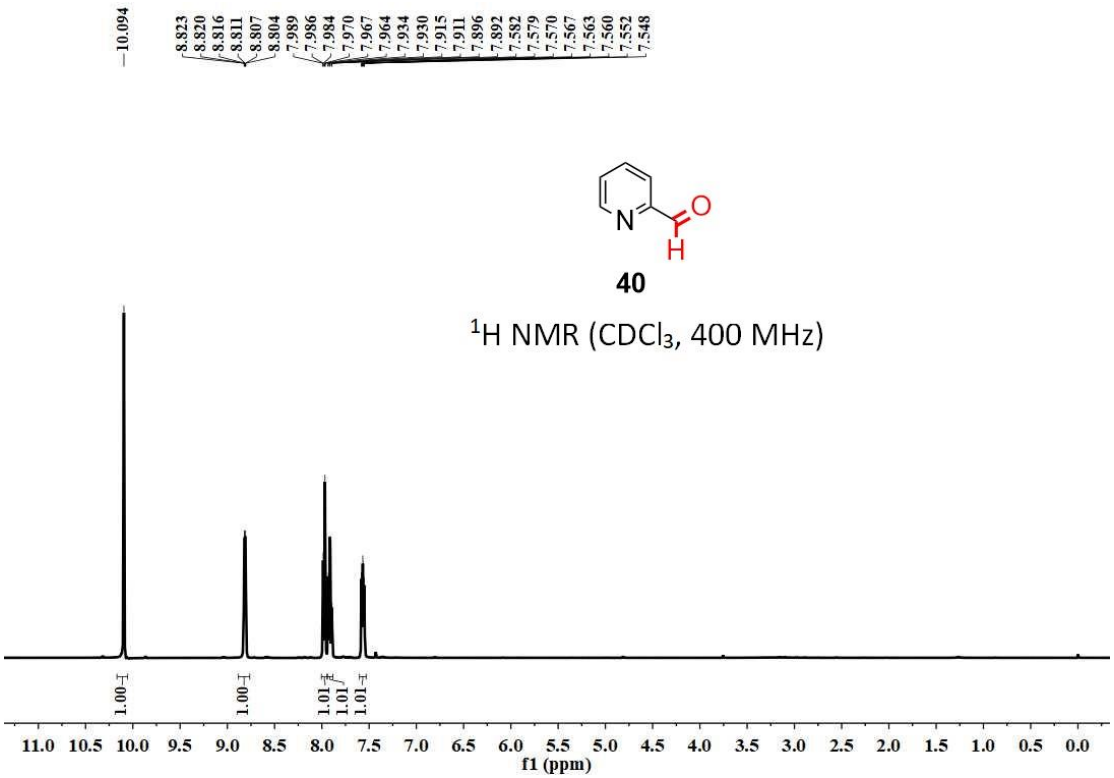
39

^{1}H NMR ($CDCl_3$, 400 MHz)



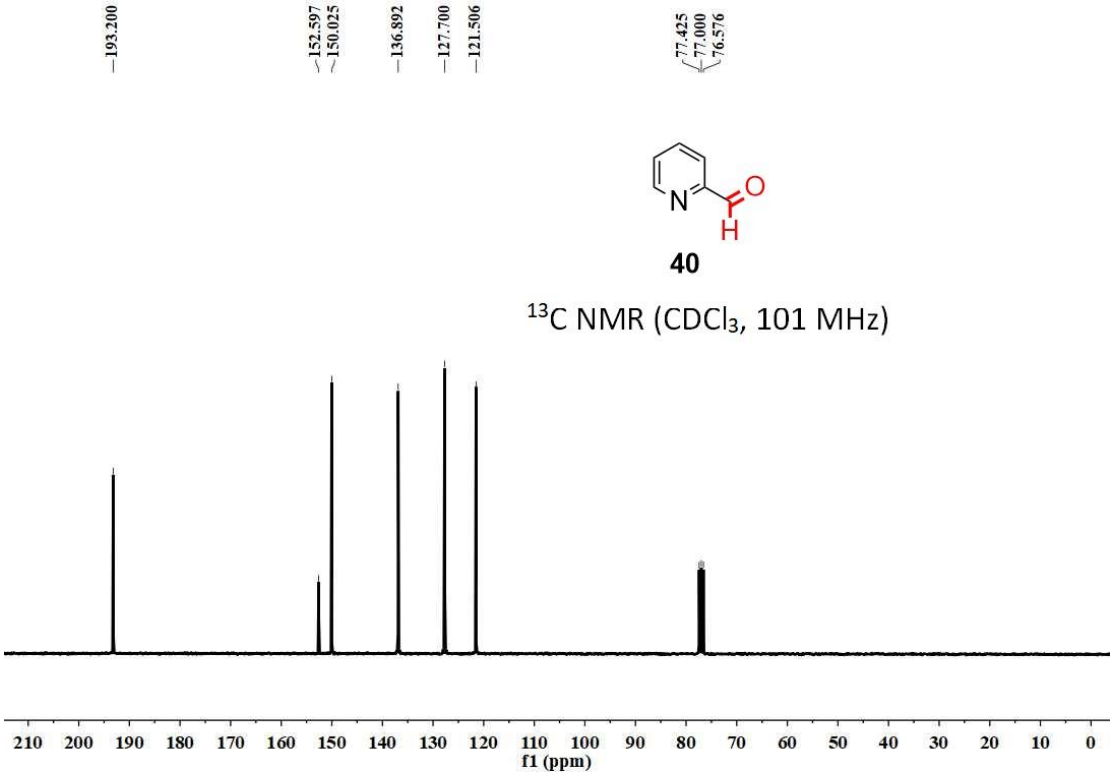
39

^{13}C NMR ($CDCl_3$, 101 MHz)



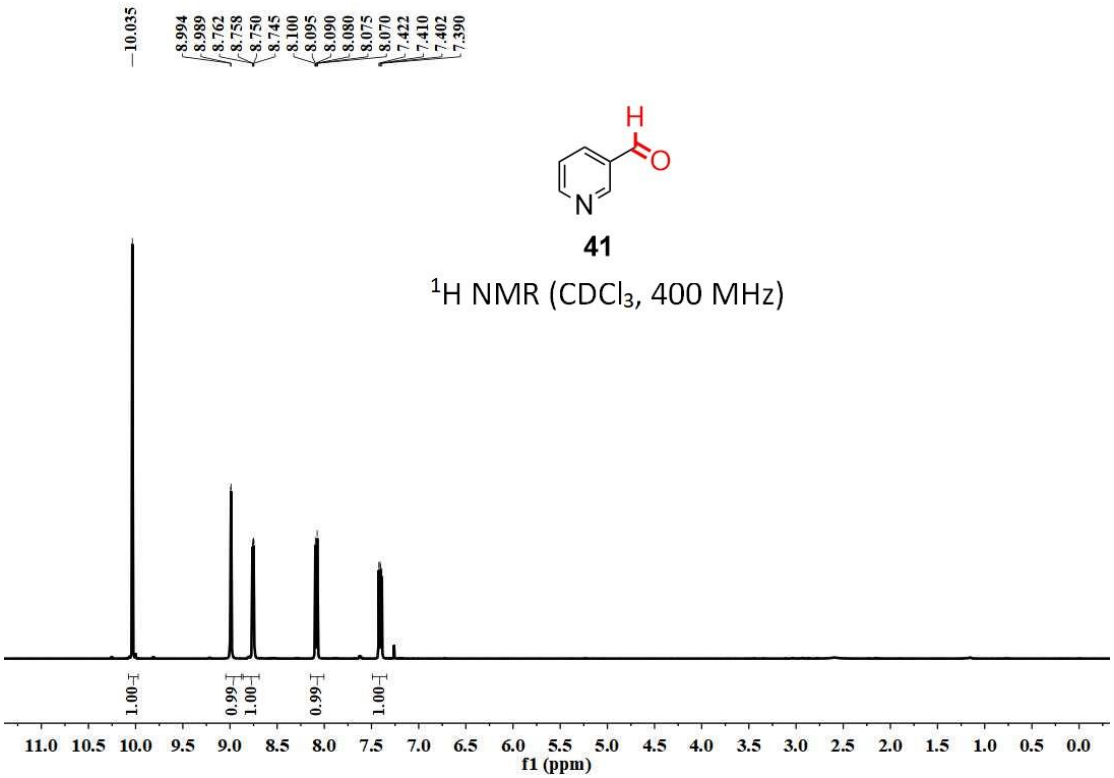
40

^{1}H NMR ($CDCl_3$, 400 MHz)



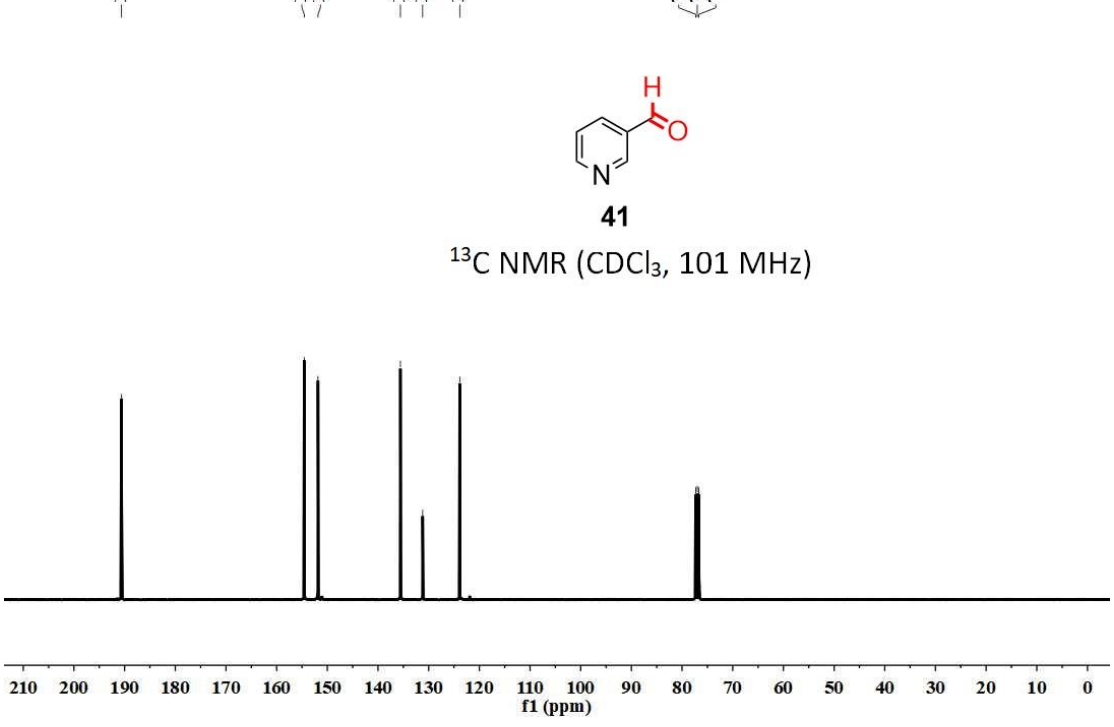
40

^{13}C NMR ($CDCl_3$, 101 MHz)



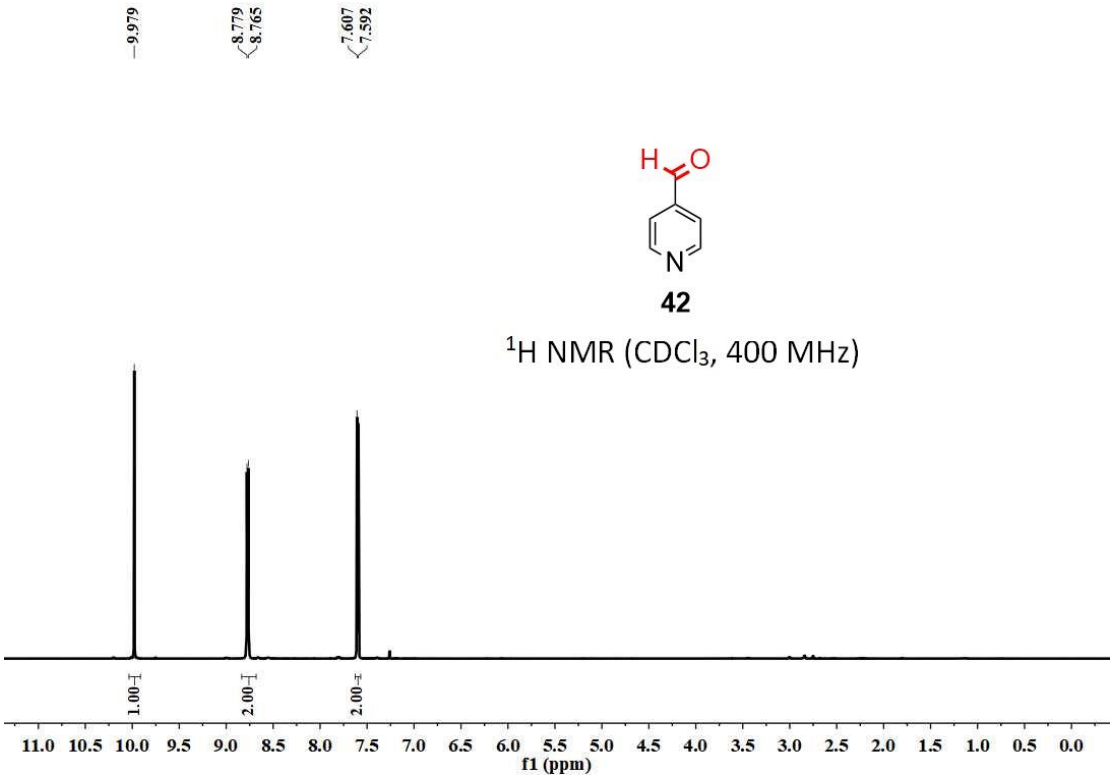
41

^{1}H NMR ($CDCl_3$, 400 MHz)



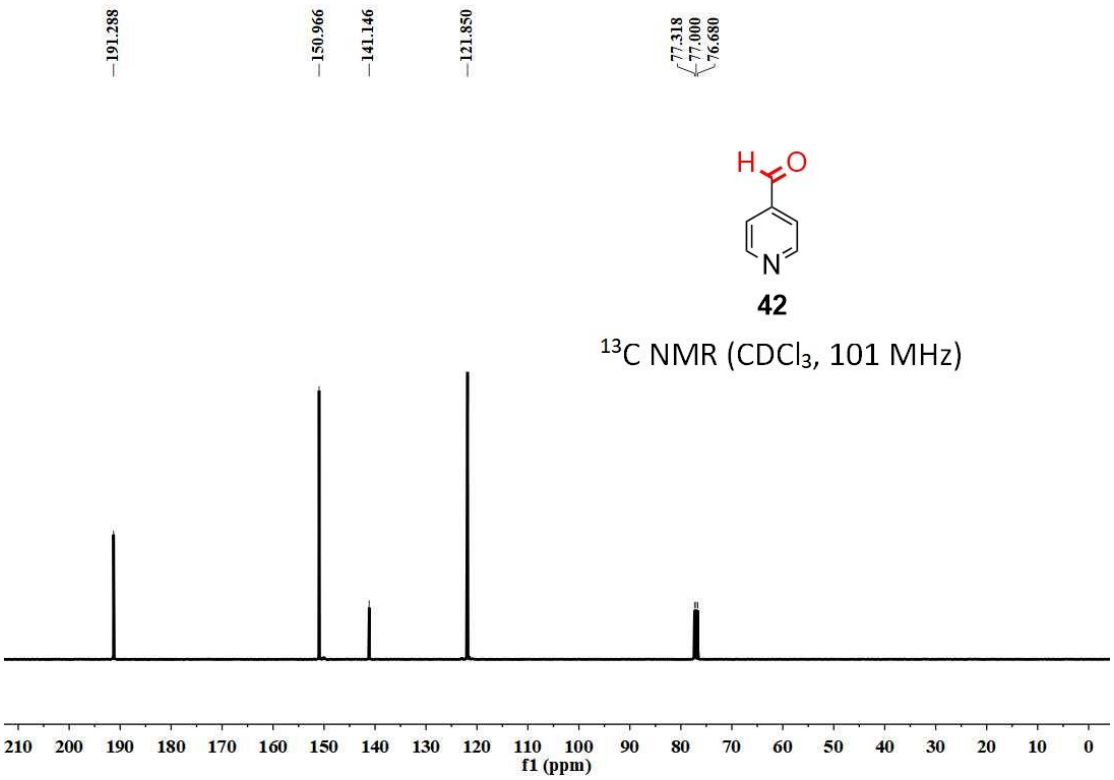
41

^{13}C NMR ($CDCl_3$, 101 MHz)



42

^{1}H NMR ($CDCl_3$, 400 MHz)



42

^{13}C NMR ($CDCl_3$, 101 MHz)

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