

## Electronic Supplementary Information (ESI)

# Continuous-Flow Synthesis of Carboxylic Acids from Alcohols via Platinum and Silicon Dioxide-Catalyzed Hydrogen Peroxide Oxidation

Yoshihiro Kon,<sup>\*,a</sup> Takuya Nakashima,<sup>a</sup> Yoshiki Makino,<sup>b</sup> Shun-ya Onozawa,<sup>a</sup>

Hiroyuki Miyamura,<sup>a</sup> Shū Kobayashi,<sup>\*,a,c</sup> and Kazuhiko Sato,<sup>\*,a</sup>

<sup>a</sup>Interdisciplinary Research Center for Catalytic Chemistry, National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba, Ibaraki 305-8565, Japan

<sup>b</sup>Environmental Management Research Institute, National Institute of Advanced Industrial Science and Technology (AIST), AIST Tsukuba West, 16-1, Onogawa, Tsukuba, Ibaraki 305-8569, Japan.

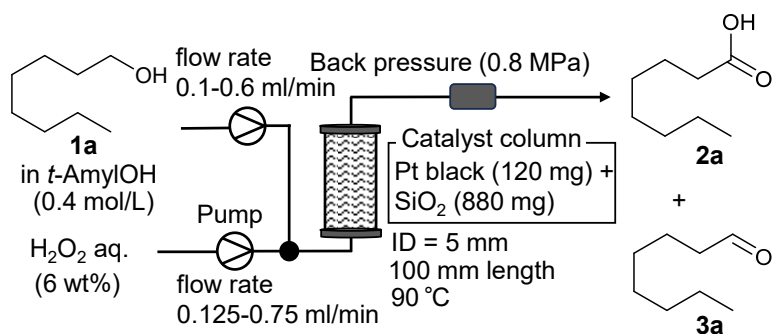
<sup>c</sup>Department of Chemistry, School of Science, The University of Tokyo, Hongo, Bunkyo-ku, Tokyo 113-0033, Japan

### Contents

1. Screening of WHSVs by changing the flow rate	S2
2. Optimization of the reaction conditions	S3
3. H <sub>2</sub> O <sub>2</sub> oxidation of 1a using a batch reactor	S4
4. Continuous flow oxidation of 1a for 210 hours	S5
5. Continuous flow oxidation of furfuryl alcohol	S6
6. XRD and SEM data of the spent Pt + SiO <sub>2</sub> catalyst	S7
7. LA-ICP-MS analyses	S8
8. Optimization of the amounts of H <sub>2</sub> O <sub>2</sub>	S10
9. XPS analyses of Pt black catalyst	S11
10. NMR Spectra	S12

## 1. Screening of WHSVs by changing the flow rate

Table S1. WHSVs and the yields of octanoic acid (**2a**) and octyl aldehyde (**3a**) by screening of flow rate



Flow rate of <b>1a</b> (ml/min)	Flow rate of H <sub>2</sub> O <sub>2</sub> (ml/min)	H <sub>2</sub> O <sub>2</sub> / <b>1a</b>	WHSV	Conv. of <b>1a</b>	Yield of <b>2a</b>	Yield of <b>3a</b>
0.10	0.125	5.5	2.6	>99	98	0
0.20	0.25	5.5	5.2	>99	98	0
0.30	0.375	5.5	7.8	>99	96	3
0.40	0.50	5.5	10	>99	90	9
0.50	0.625	5.5	13	99	83	15
0.60	0.75	5.5	15	98	79	19

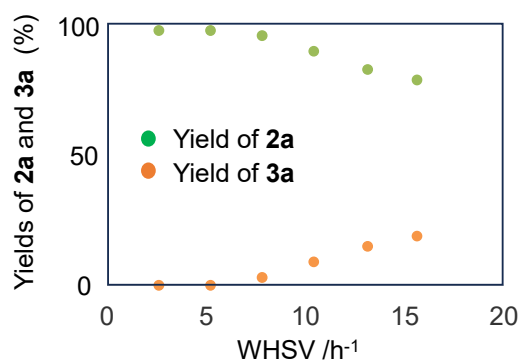


Fig. S1. Plots of WHSVs and the yields of octanoic acid (**2a**) and octyl aldehyde (**3a**) by screening of flow rate

## 2. Optimization of the reaction conditions

Table S2. Screening of 1-octanol (**1a**) concentration based on the oxidation of **1a**

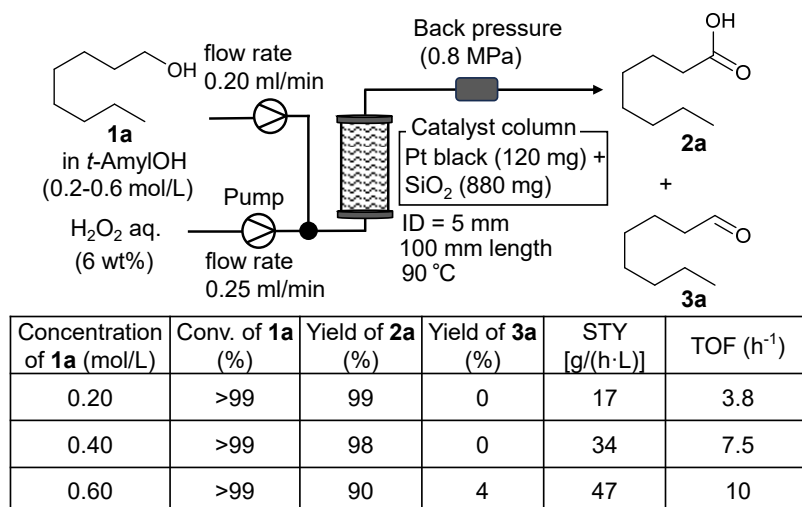


Table S3. Screening of reaction temperature based on the oxidation of **1a**

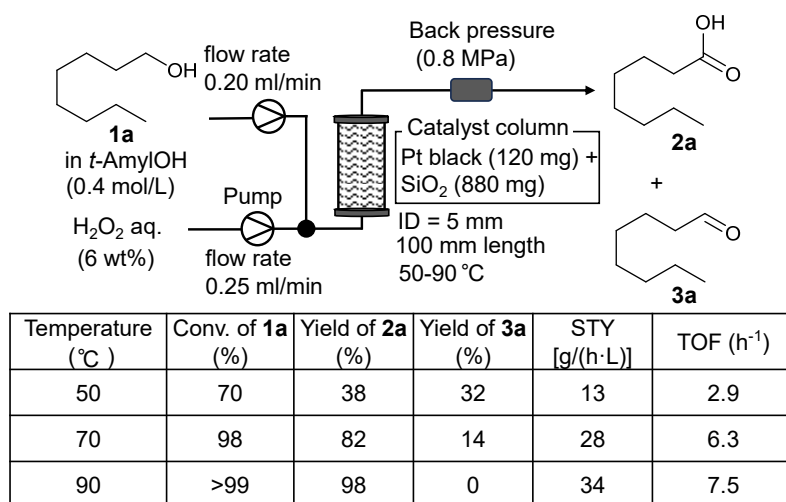
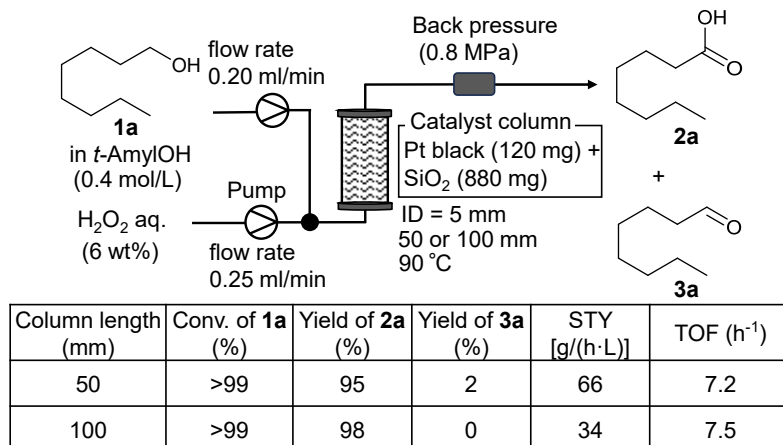
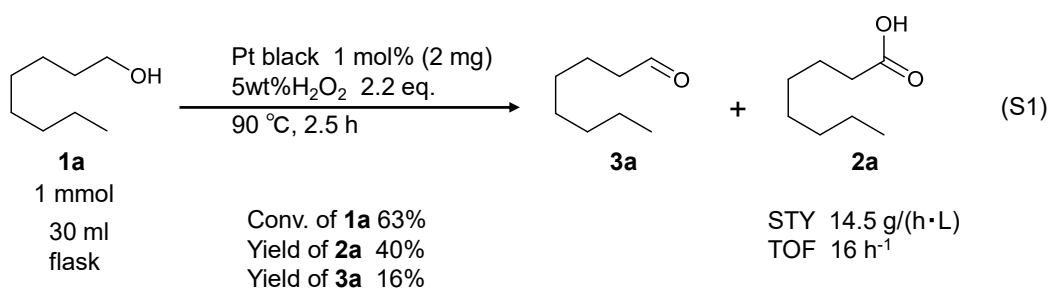


Table S4. Use of 100 mm or 50 mm column length based on the oxidation of **1a**



### 3. H<sub>2</sub>O<sub>2</sub> oxidation of 1a using a batch reactor



#### 4. Continuous flow oxidation of 1a for 210 hours

Table S5. Continuous-flow H<sub>2</sub>O<sub>2</sub> oxidation of 1a for 210 hours

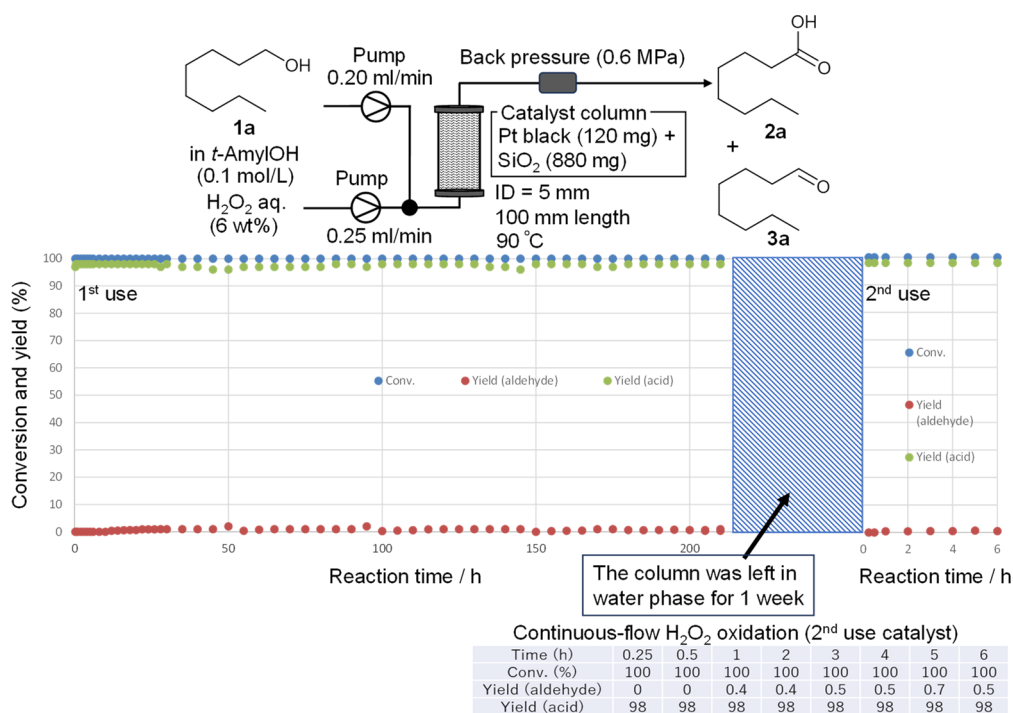
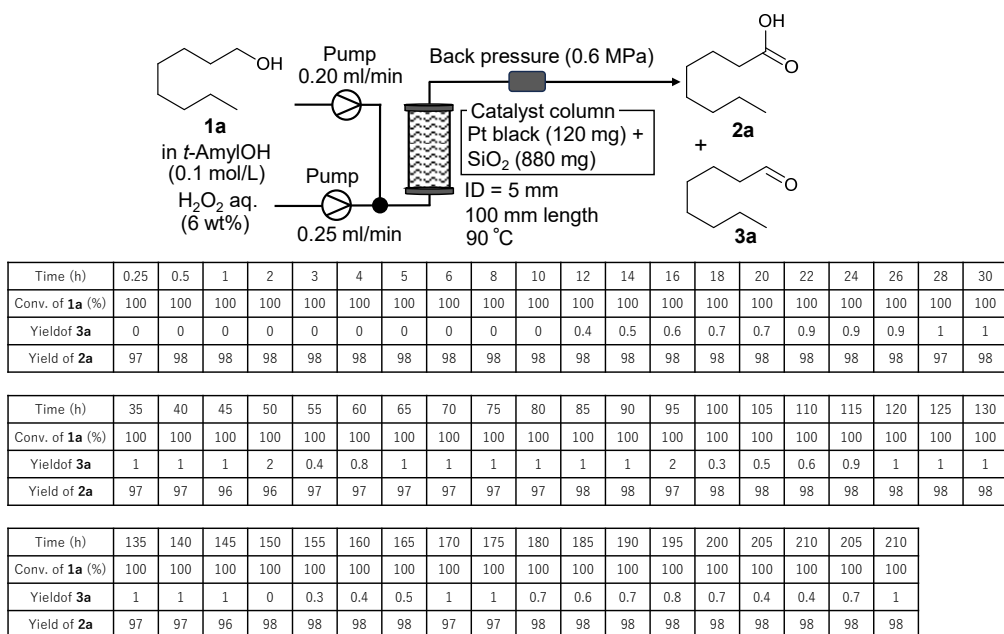
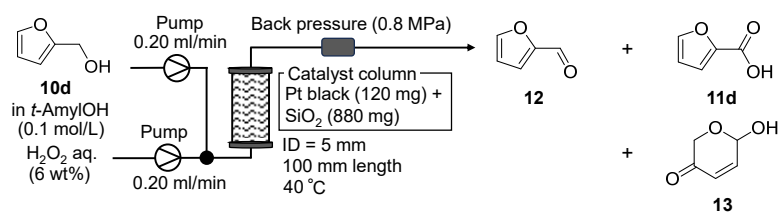


Fig. S2. Long-term recyclability of Pt and SiO<sub>2</sub> catalyst column for the continuous-flow H<sub>2</sub>O<sub>2</sub> oxidation of 1a

## 5. Continuous-flow H<sub>2</sub>O<sub>2</sub> oxidation of furfuryl alcohol

Table S6. Continuous-flow H<sub>2</sub>O<sub>2</sub> oxidation of furfuryl alcohol



Time (h)	Conv. of <b>10d</b> (%)	Yield of <b>12</b> (%)	Yield of <b>11d</b> (%)	Yield of <b>13</b> (%)
0.25	>99	16	0	69
0.5	>99	16	0	78
1	>99	19	0	79
Average	>99	17	0	75

6. XRD and SEM data of the spent Pt + SiO<sub>2</sub> catalyst

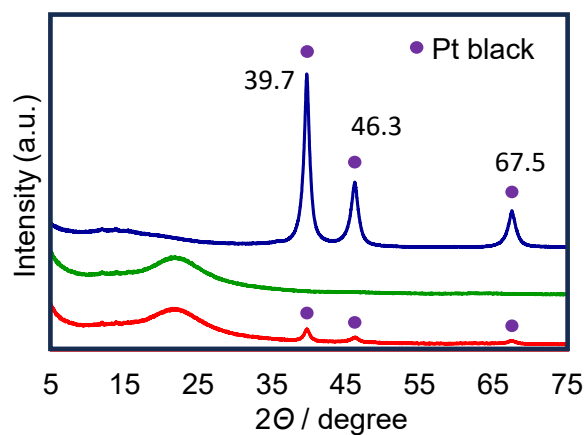


Fig. S3. X-ray diffraction (XRD) data of Pt black (blue line), SiO<sub>2</sub> (green line) and the spent catalyst (red line) after the continuous-flow H<sub>2</sub>O<sub>2</sub> oxidation of **1a** for 1 h

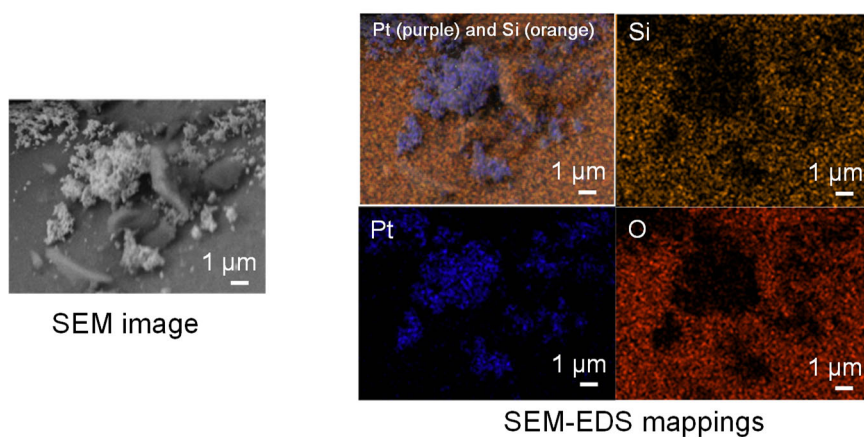


Fig. S4. Scanning electron microscope (SEM) image and the SEM-Energy dispersive X-ray spectroscopy (SEM-EDS) mappings of Pt black with SiO<sub>2</sub> in the catalyst column before the oxidation.

## 7. LA-ICP-MS analyses

Table S7. Continuous-flow H<sub>2</sub>O<sub>2</sub> oxidation of **1a** at WHSV 15.6 used for LA-ICP-MS analyses

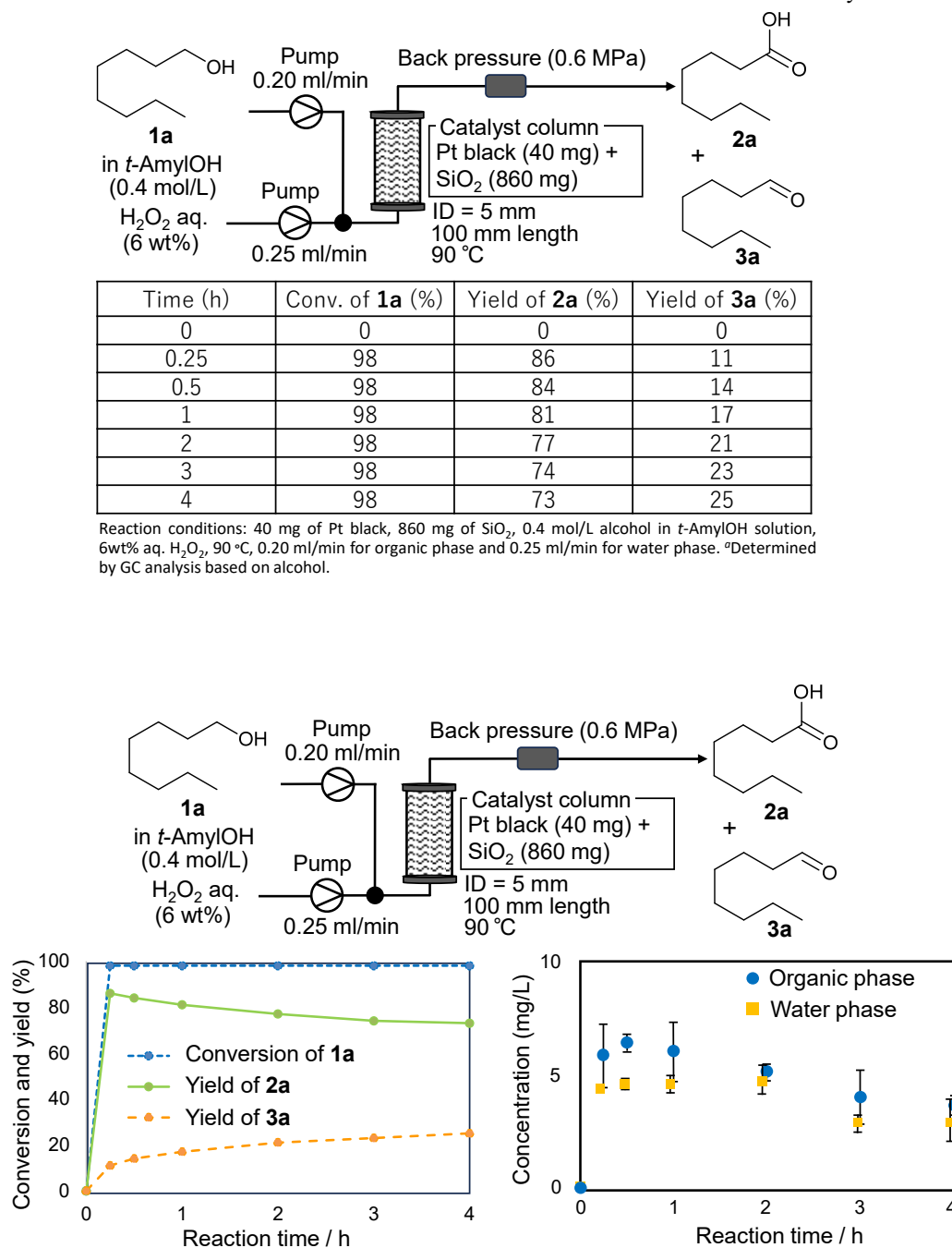


Fig. S5. Correlation between the leaching amounts of Pt and the yields of **2a** and **3a**.

Laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) combined with a dried spot method was used for the determination of leaching amount of platinum from the platinum catalyst. The details of the analytical method were described in our previous paper (ref 16 in the manuscript). The LA system (Jupiter -solid nebulizer-, STJapan, Japan) and ICP-MS (iCAP Q, Thermo Fisher Scientific, Germany) were connected using PFA tubing with an inner diameter of 4 mm. Platinum stock solution (Platinum Standard



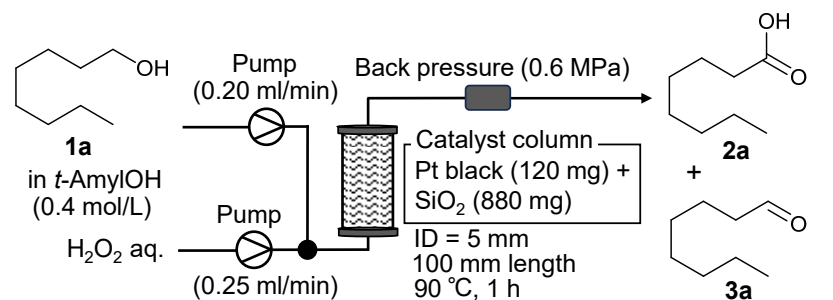
Solution (Pt 1000), FUJIFILM Wako Chemicals, Japan) and guaranteed hydrochloric acid (Ultrapure HCl, Kanto Chemicals, Japan) were used for the preparation of the calibration solutions. The HCl solution was diluted to 1 mol/L by ultra-pure water (18.2 MΩ cm) generated from Milli-Q element module (Merck Millipore, Germany). The 0.2 μl sample and calibration solutions were spotted onto pure cellulose paper (Whatman cellulose chromatography paper 1 Chr, cytiva, USA) by micropipette. The cellulose paper was heated and completely evaporate the solvent by dryer and analyzed by LA-ICP-MS. The operating parameters for LA-ICP-MS are shown in Table S8.

Table S8. Operating parameters for LA-ICP-MS

Laser ablation (Jupiter -solid nebulizer-, STJapan, Japan)	
Wavelength	257 nm
Pulse width	290 fs
Spot size	10 μm
Laser power	100 mW
Repetition rate	60 kHz
Ablation area	5 mm (square)
Pitch	5 μm
Raster speed	80 mm/s
Ablation time	68 s
ICP-MS (iCAP Q, Thermo Fisher Scientific, Germany)	
RF power	1550 W
Cooling gas	14 l/min
Auxiliary gas	0.75 l/min
Sampling depth	5 mm
He carrier gas	0.6 l/min
Ar make up gas	0.6 l/min
Mode	no gas
Data acquisition	TRA
Dwell time	0.1 s/isotope
Integration time	120 s
Isotopes	<sup>13</sup> C (Internal standard), <sup>195</sup> Pt
Cone	Ni

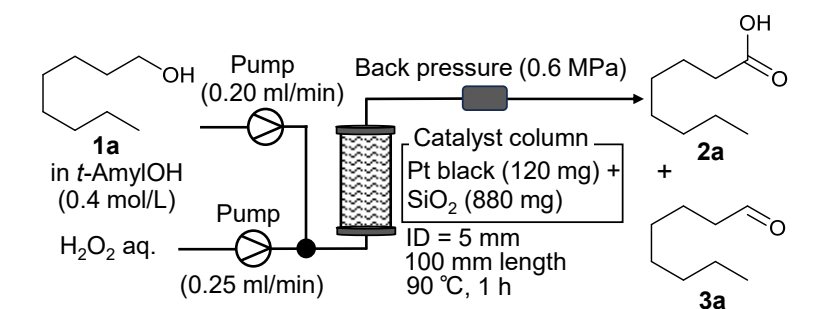
## 8. Optimization of the amounts of H<sub>2</sub>O<sub>2</sub>

Table S9. Screening of the amounts of H<sub>2</sub>O<sub>2</sub> at the continuous-flow H<sub>2</sub>O<sub>2</sub> oxidation of **1a**



Concentration of H <sub>2</sub> O <sub>2</sub> (wt%)	H <sub>2</sub> O <sub>2</sub> eq. vs. <b>1a</b> (eq.)	Conv. of <b>1a</b> (%)	Yield of <b>2a</b> (%)	Yield of <b>3a</b> (%)
2.2	2	93	36	54
3.3	3	>99	83	11
4.4	4	>99	95	0
6.0	5.5	>99	98	0

Table S10. Screening of the amounts of H<sub>2</sub>O<sub>2</sub> at the continuous-flow H<sub>2</sub>O<sub>2</sub> oxidation of **1a**



Time	Conv. of <b>1a</b>	Yield of <b>2a</b>	Yield of <b>3a</b>
0	0	0	0
0.25	93	37	53
0.5	93	36	54
1	93	35	54
Change the amounts of H <sub>2</sub> O <sub>2</sub> from 2 eq. to 4 eq.			
1.25	>99	94	0
1.5	>99	94	0
2	>99	94	0

## 9. XPS analyses of Pt black catalyst

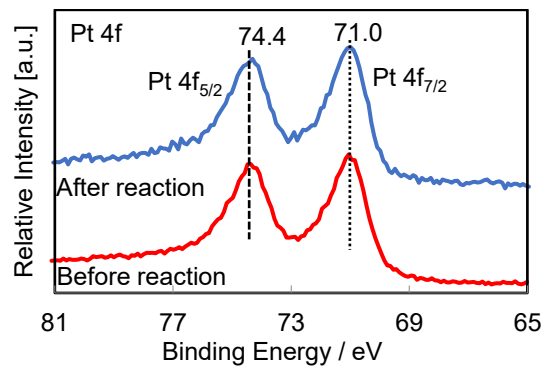


Fig. S6. XPS analyses of Pt black in the catalyst column before (red line) and after (blue line) the reaction.

## 10. NMR Spectra

The NMR spectroscopic data of the synthesized compounds **2a–11c** agreed well with the  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR data reported by the production through another methods.

1-Octanoic acid (**2a**):<sup>(a)</sup> 93% yield (0.63 g, 4.40 mmol, pale yellow oil),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  2.35 (t,  $J = 7.4$  Hz, 2H), 1.67-1.60 (m, 2H), 1.33-1.28 (m, 8H), 0.88 (t,  $J = 6.8$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  180.4, 34.1, 31.1, 29.0, 28.9, 24.7, 22.6, 14.0.

1-Hexanoic acid (**2b**):<sup>(a)</sup> 91% yield (0.50 g, 4.28 mmol, colorless oil),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  2.35 (t,  $J = 7.4$  Hz, 2H), 1.68-1.61 (m, 2H), 1.34-1.31 (m, 4H), 0.90 (t,  $J = 6.9$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  180.5, 34.1, 31.2, 24.3, 22.3, 13.8.

1-Decanoic acid (**2c**):<sup>(b)</sup> 97% yield (0.79 g, 4.57 mmol, colorless oil),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  2.35 (t,  $J = 7.5$  Hz, 2H), 1.67-1.60 (m, 2H), 1.35-1.27 (m, 12H), 0.88 (t,  $J = 6.9$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  180.4, 34.1, 31.8, 29.4, 29.2, 29.0, 24.7, 22.6, 14.1.

4-Methoxybenzoic acid (**5a**):<sup>(a)</sup> 95% yield (0.08 g, 0.55 mmol, colorless solid),  $^1\text{H}$  NMR (400 MHz, DMSO, 25 °C, TMS):  $\delta$  12.61 (s, 1H), 7.92-7.88 (m, 2H), 7.04-7.00 (m, 2H), 3.83 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz, DMSO, 25 °C, TMS):  $\delta$  166.9, 162.8, 131.3, 122.9, 113.8, 55.4.

4-Methylbenzoic acid (**5b**):<sup>(a)</sup> 95% yield (0.08 g, 0.56 mmol, colorless solid),  $^1\text{H}$  NMR (400 MHz, DMSO, 25 °C, TMS):  $\delta$  12.77 (s, 1H), 7.85-7.83 (m, 2H), 7.31-7.29 (m, 2H), 2.37 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz, DMSO, 25 °C, TMS):  $\delta$  167.3, 143.0, 129.3, 129.1, 128.0, 21.1.

Benzoic acid (**5c**):<sup>(a)</sup> 98% yield (0.14 g, 1.18 mmol, colorless solid),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  8.14-8.12 (m, 2H), 7.62 (t,  $J = 7.4$  Hz, 1H), 7.48 (t,  $J = 7.8$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  172.3, 133.8, 130.2, 129.3, 128.5.

4-Chlorobenzoic acid (**5d**):<sup>(a)</sup> 96% yield (0.09 g, 0.57 mmol, colorless solid),  $^1\text{H}$  NMR (400 MHz, DMSO, 25 °C, TMS):  $\delta$  13.16 (s, 1H), 7.96-7.94 (m, 2H), 7.58-7.56 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO, 25 °C, TMS):  $\delta$  166.4, 137.8, 131.1, 129.6, 128.7.

4-Bromobenzoic acid (**5e**):<sup>(a)</sup> 86% yield (0.10 g, 0.51 mmol, colorless solid),  $^1\text{H}$  NMR (400 MHz, DMSO, 25 °C, TMS):  $\delta$  13.17 (s, 1H), 7.88-7.86 (m, 2H), 7.73-7.70 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO, 25 °C, TMS):  $\delta$  166.6, 131.7, 131.2, 130.0, 126.8.

4-(Trifluoromethyl)benzoic acid (**5f**):<sup>(a)</sup> 89% yield (0.10 g, 0.51 mmol, colorless solid),  $^1\text{H}$  NMR (400 MHz, DMSO, 25 °C, TMS):  $\delta$  13.48 (s, 1H), 8.16-8.13 (m, 2H), 7.89-7.87 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz, DMSO, 25 °C, TMS):  $\delta$  166.2, 134.6, 132.6, 132.3, 130.1, 125.6, 125.5, 125.1, 122.4.

3-Methoxybenzoic acid (**5g**):<sup>(a)</sup> 95% yield (0.08 g, 0.55 mmol, colorless solid),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  7.74-7.71 (m, 1H), 7.64-7.63 (m, 1H), 7.38 (t,  $J = 7.9$  Hz, 1H), 7.18-7.15 (m, 1H), 3.87 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  172.1, 159.6, 130.6, 129.5, 122.7, 120.5, 114.4, 55.5.

2-Methoxybenzoic acid (**5h**):<sup>(a)</sup> 44% yield (0.04 g, 0.26 mmol, colorless solid),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  8.18 (dd,  $J = 7.8, 1.8$  Hz, 1H), 7.60-7.56 (m, 1H), 7.16-7.12 (m, 1H), 7.08-7.06 (m, 1H), 4.08 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  165.4, 158.1, 135.0, 133.8, 122.2, 117.6, 111.7, 56.7.

3,4-Dimethoxybenzoic acid (**5i**):<sup>(a)</sup> 95% yield (0.10 g, 0.56 mmol, colorless solid),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  7.78 (dd,  $J = 8.4, 2.0$  Hz, 1H), 7.61 (d,  $J = 2.0$  Hz, 1H), 6.92 (d,  $J = 8.5$  Hz, 1H), 3.96 (s, 3H), 3.95 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  172.9, 153.8, 148.7, 124.6, 121.7, 112.3, 110.3, 56.1, 56.0.

(*E*)-Cinnamic acid (**7a**):<sup>(a)</sup> 89% yield (0.15 g, 1.03 mmol, pale yellow solid),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  7.80 (d,  $J = 16.0$  Hz, 1H), 7.57-7.55 (m, 2H), 7.42-7.40 (m, 3H), 6.47 (d,  $J = 16.0$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  172.4, 147.1, 134.0, 130.7, 129.0, 128.4, 117.3.

2-Methyl-3-phenyl-2-propenoic acid (**7b**):<sup>(a)</sup> 67% yield (0.06 g, 0.38 mmol, colorless solid),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  7.84 (d,  $J = 1.2$  Hz, 1H), 7.45-7.32 (m, 5H), 2.15 (d,  $J = 1.4$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  174.3, 141.1, 135.6, 129.8, 128.7, 128.4, 127.6, 13.7.

(*E*)-2-Octenoic acid (**7c**):<sup>(c)</sup> 60% yield (0.05 g, 0.35 mmol, colorless oil),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  7.13-7.05 (m, 1H), 5.82 (d,  $J = 15.6$  Hz, 1H), 2.26-2.20 (m, 2H), 1.51-1.44 (m, 2H), 1.35-1.28 (m, 4H), 0.90 (t,  $J = 7.0$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  172.1, 152.5, 120.6, 32.3, 31.3, 27.5, 22.4, 13.9.

Phenoxyacetic acid (**9**):<sup>(a)</sup> 91% yield (0.33 g, 2.15 mmol, colorless solid),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  8.28 (br, 1H), 7.33-7.29 (m, 2H), 7.03-7.00 (m, 1H), 6.94-6.91 (m, 1H), 4.69 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ , 25 °C, TMS):  $\delta$  174.2, 157.4, 129.7, 122.1, 114.6, 64.8.

2-Pyridinecarboxylic acid (**11a**):<sup>(a)</sup> 98% yield (0.07 g, 0.57 mmol, colorless solid), <sup>1</sup>H NMR (400 MHz, DMSO, 25 °C, TMS): δ 13.08 (br, 1H), 8.72-8.70 (m, 1H), 8.07-8.04 (m, 1H), 8.01-7.97 (m, 1H), 7.65-7.61 (m, 1H); <sup>13</sup>C NMR (100 MHz, DMSO, 25 °C, TMS): δ 166.1, 149.4, 148.3, 137.5, 127.0, 124.6.

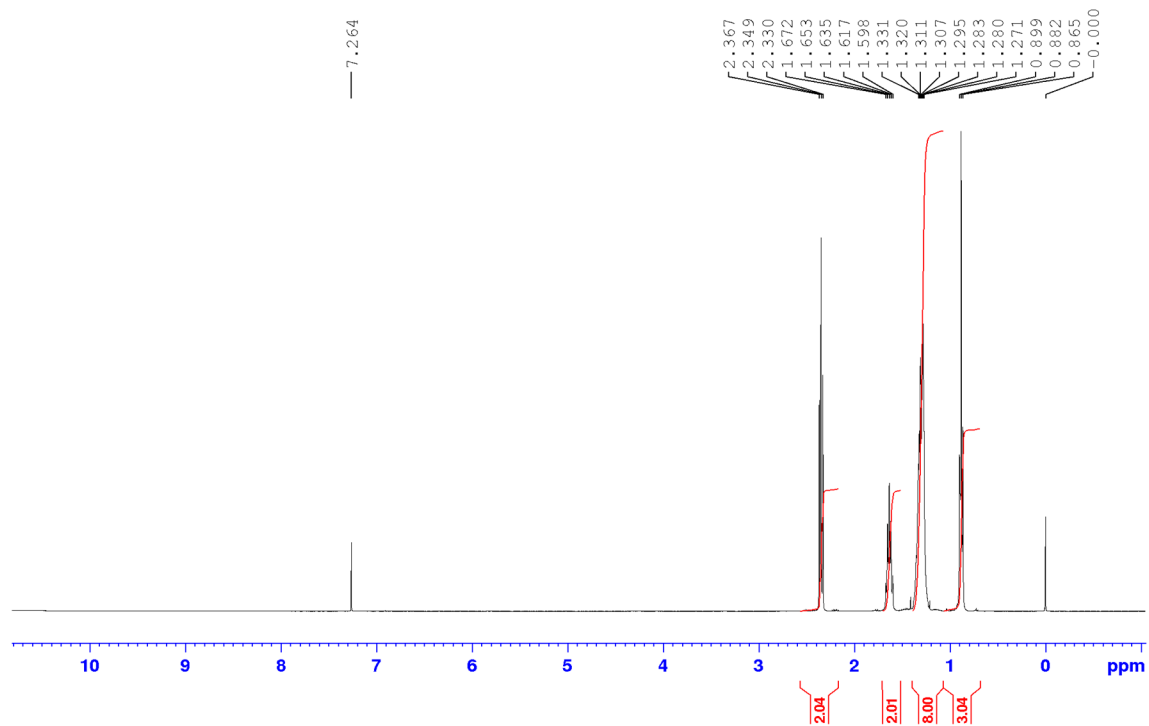
4-Pyridinecarboxylic acid (**11b**):<sup>(a)</sup> 98% yield (0.07 g, 0.56 mmol, colorless solid), <sup>1</sup>H NMR (400 MHz, DMSO, 25 °C, TMS): δ 13.63 (br, 1H), 8.78 (dd, *J* = 4.4, 2.0 Hz, 2H), 7.82 (dd, *J* = 4.4, 1.6 Hz, 2H); <sup>13</sup>C NMR (100 MHz, DMSO, 25 °C, TMS): δ 166.2, 150.6, 138.1, 122.7.

2-Thiophenecarboxylic Acid (**11c**):<sup>(d)</sup> 18% yield (0.03 g, 0.22 mmol, colorless solid), <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, 25 °C, TMS): δ 7.91-7.89 (m, 1H), 7.66-7.64 (m, 1H), 7.14 (dd, *J* = 5.0, 3.8 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>, 25 °C, TMS): δ 167.5, 135.0, 134.0, 132.8, 128.1.

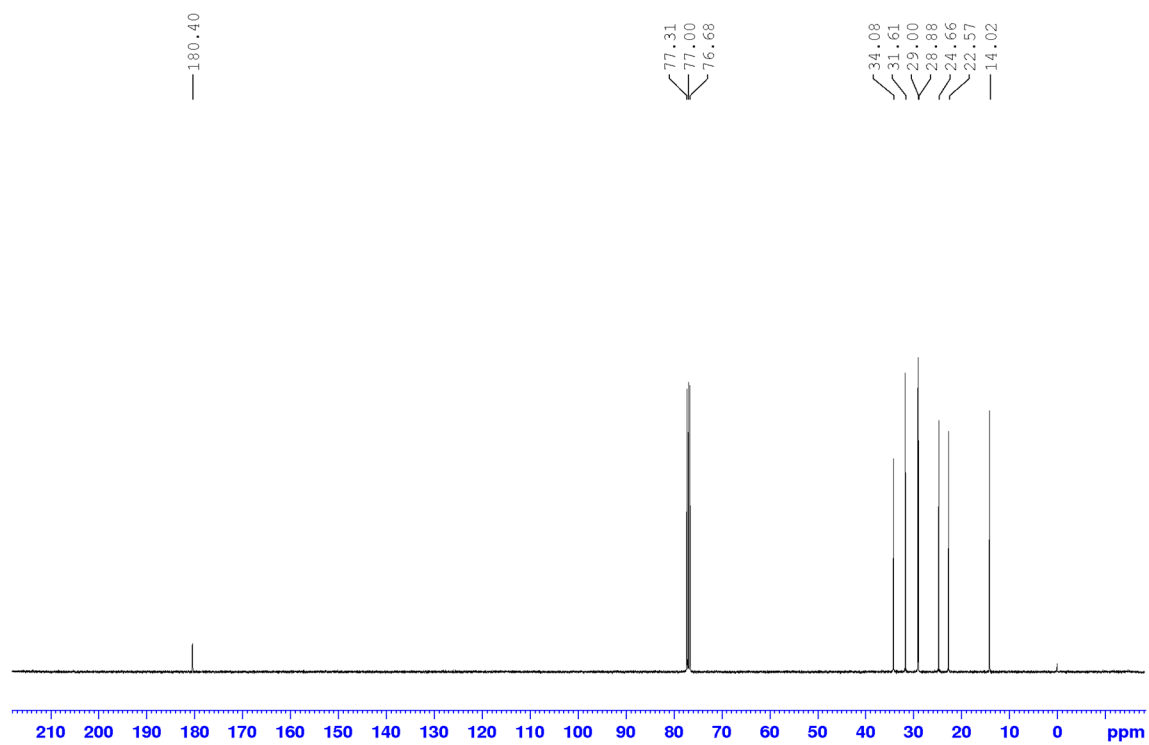
Ref). (a) SDBSWeb: <https://sdbs.db.aist.go.jp> (National Institute of Advanced Industrial Science and Technology, Dec. 14, 2021); (b) C. J. Pouchert and J. Behnke, *The Aldrich Library of <sup>13</sup>C and <sup>1</sup>H FT NMR Spectra, 1st ed.* Vol. 1, Aldrich Chemical, Milwaukee, 1993, 754-B; (c) C. J. Pouchert and J. Behnke, *The Aldrich Library of <sup>13</sup>C and <sup>1</sup>H FT NMR Spectra, 1st ed.* Vol. 1, Aldrich Chemical, Milwaukee, 1993, 780-B; (d) C. J. Pouchert and J. Behnke, *The Aldrich Library of <sup>13</sup>C and <sup>1</sup>H FT NMR Spectra, 1st ed.* Vol. 3, Aldrich Chemical, Milwaukee, 1993, 59-A.

1-Octanoic acid (2a)

$^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ , 25 °C)

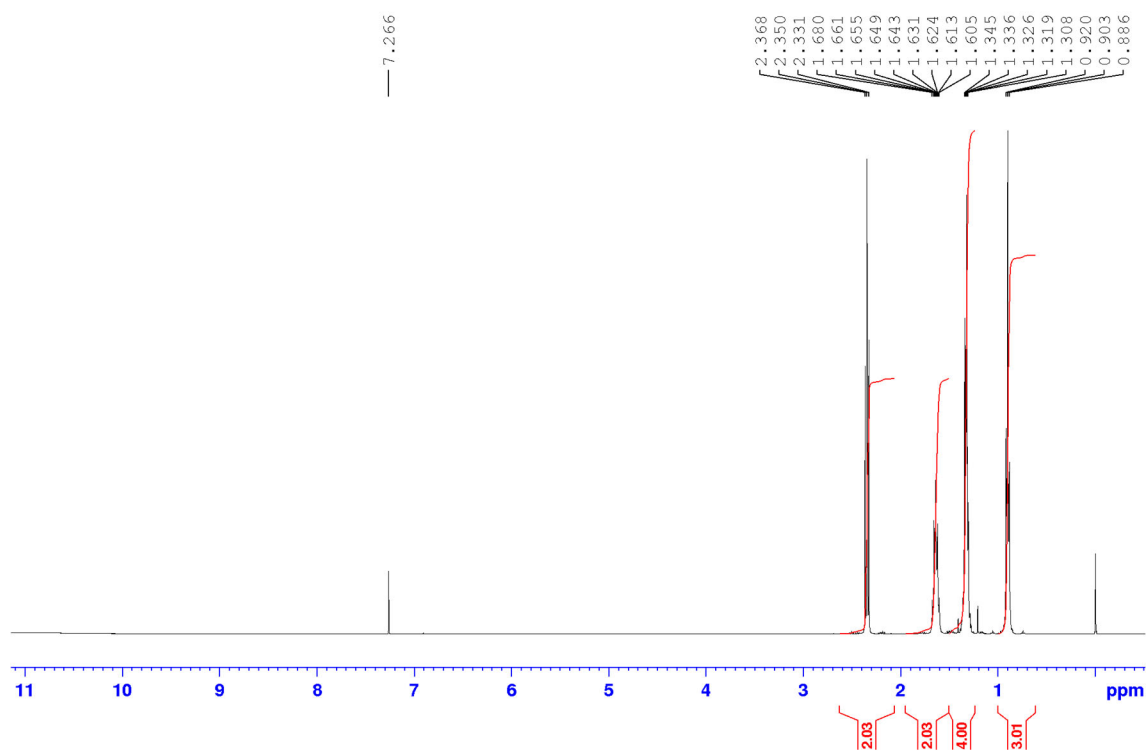


$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ , 25 °C)

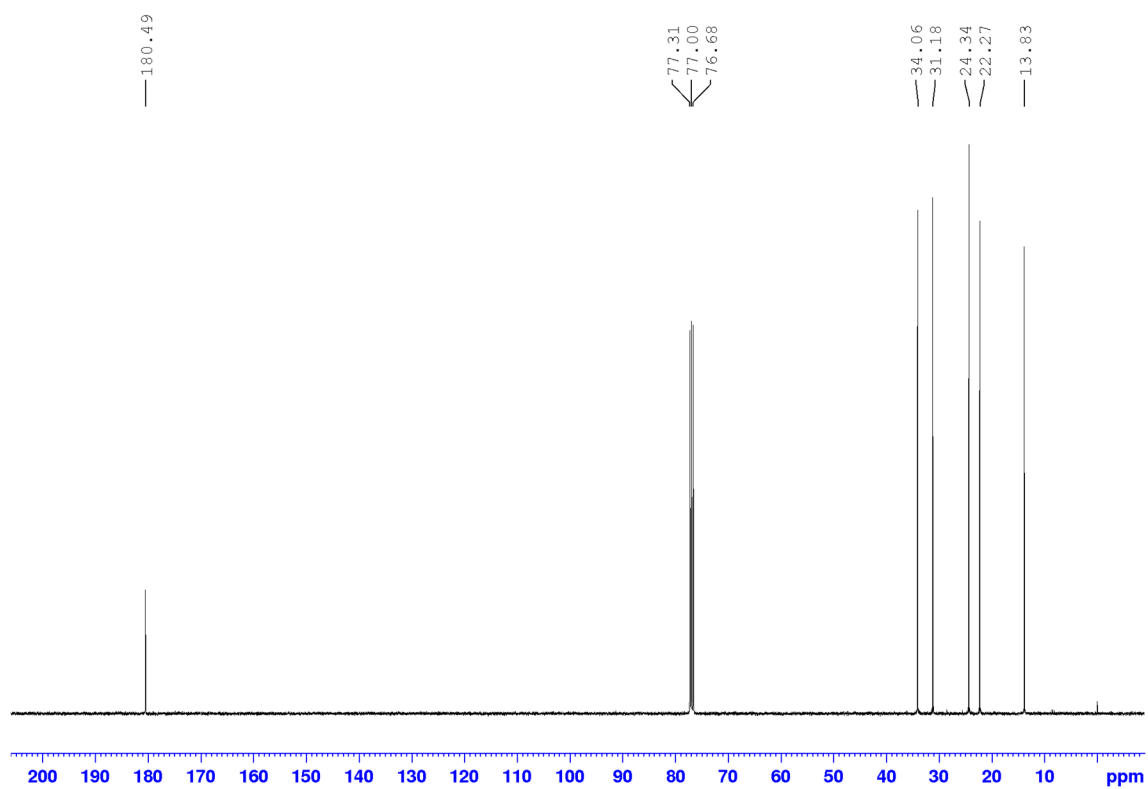


**1-Hexanoic acid (2b)**

**<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>, 25 °C)**

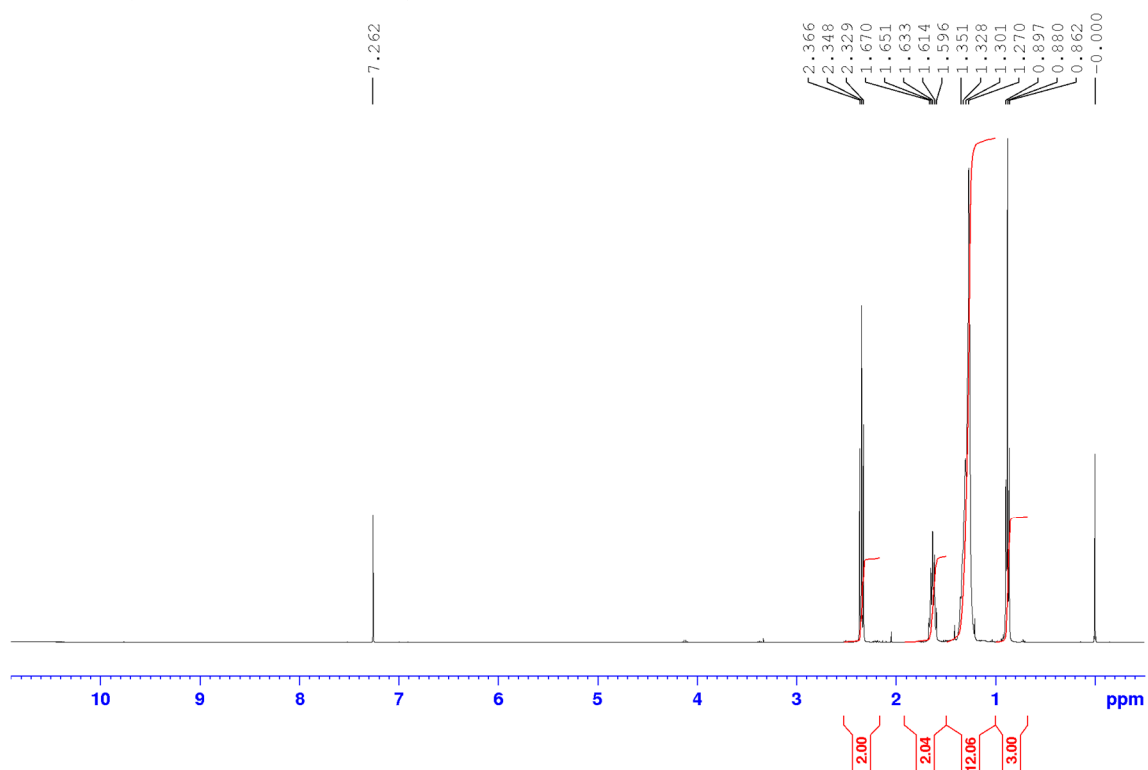


**<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>, 25 °C)**

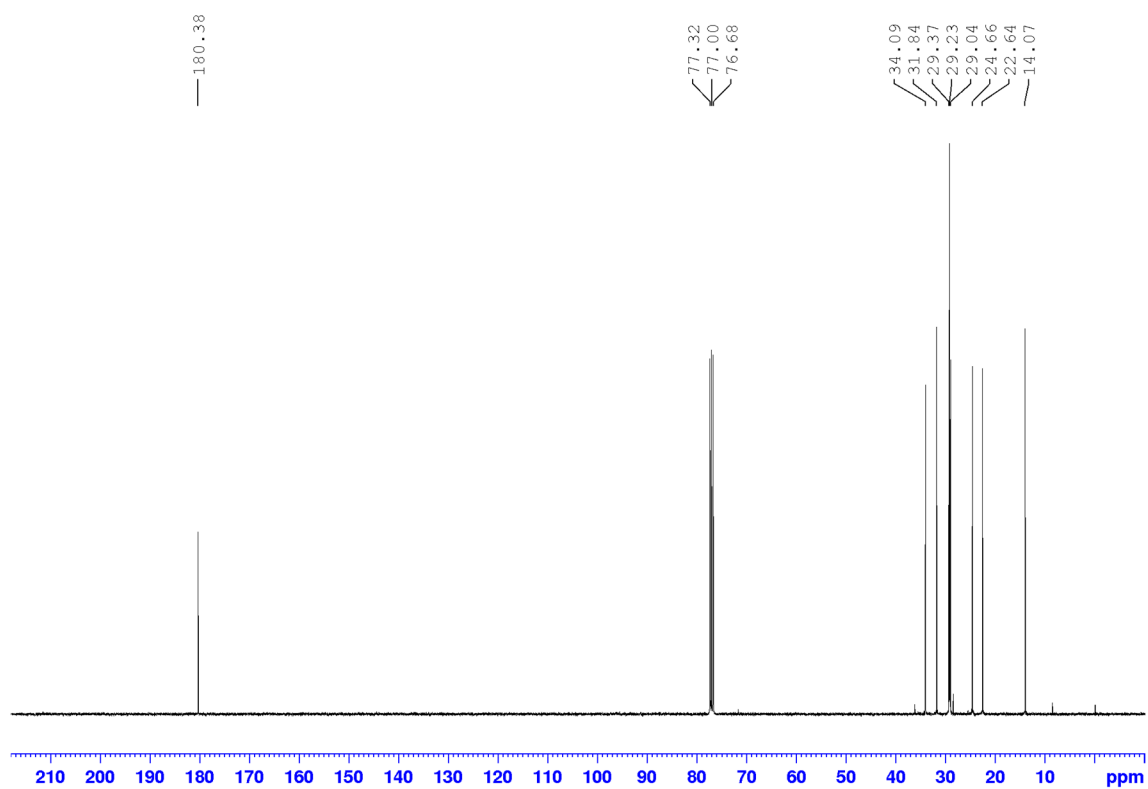


**1-Decanoic acid (2c)**

**$^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ , 25 °C)**



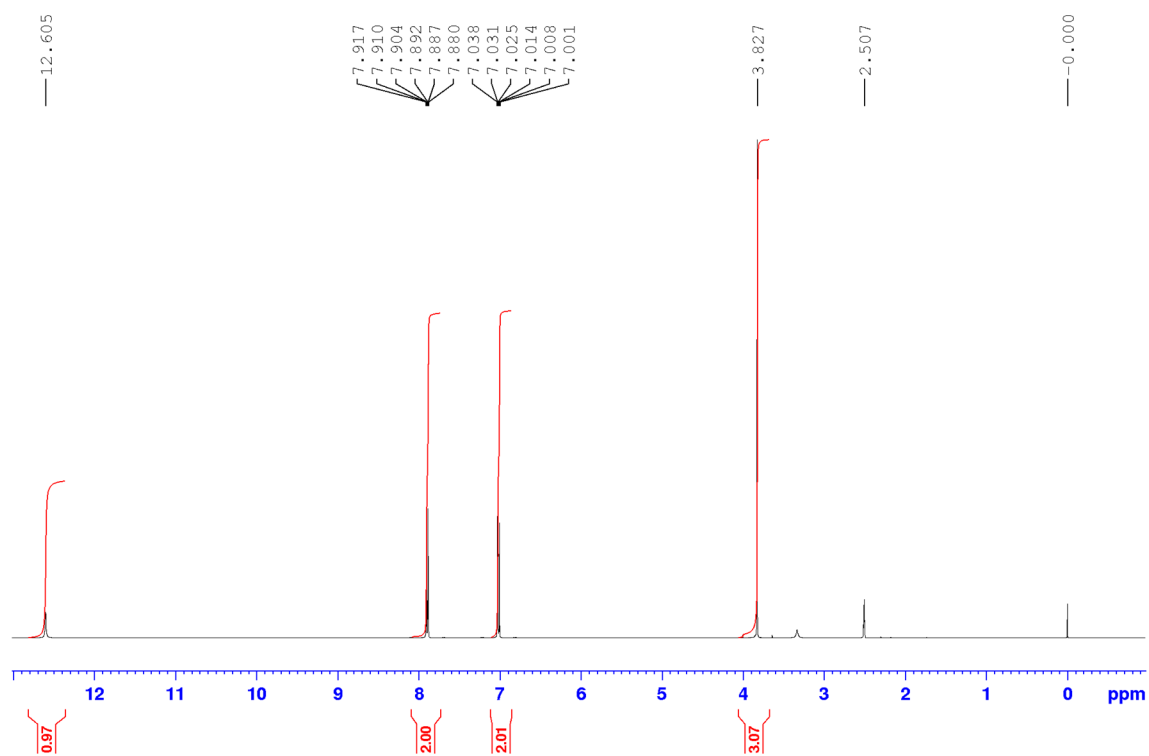
**$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ , 25 °C)**



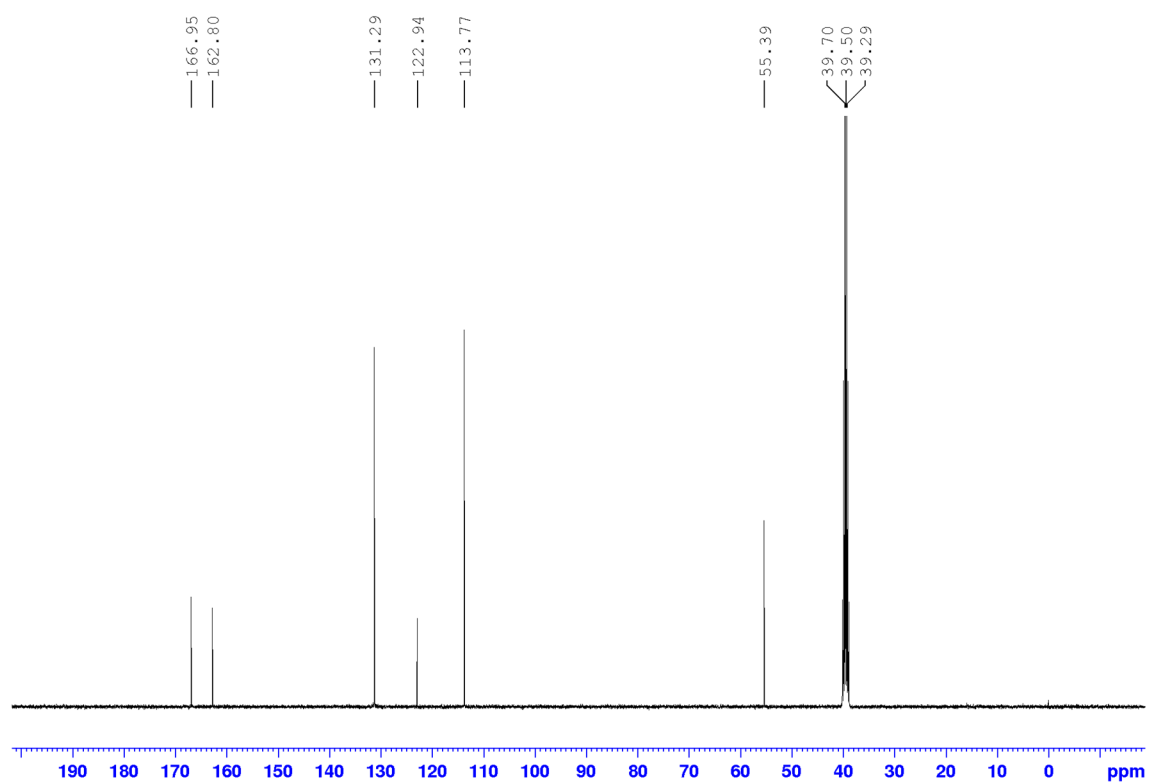


**4-Methoxybenzoic acid (5a)**

**<sup>1</sup>H NMR (400MHz, DMSO, 25 °C)**

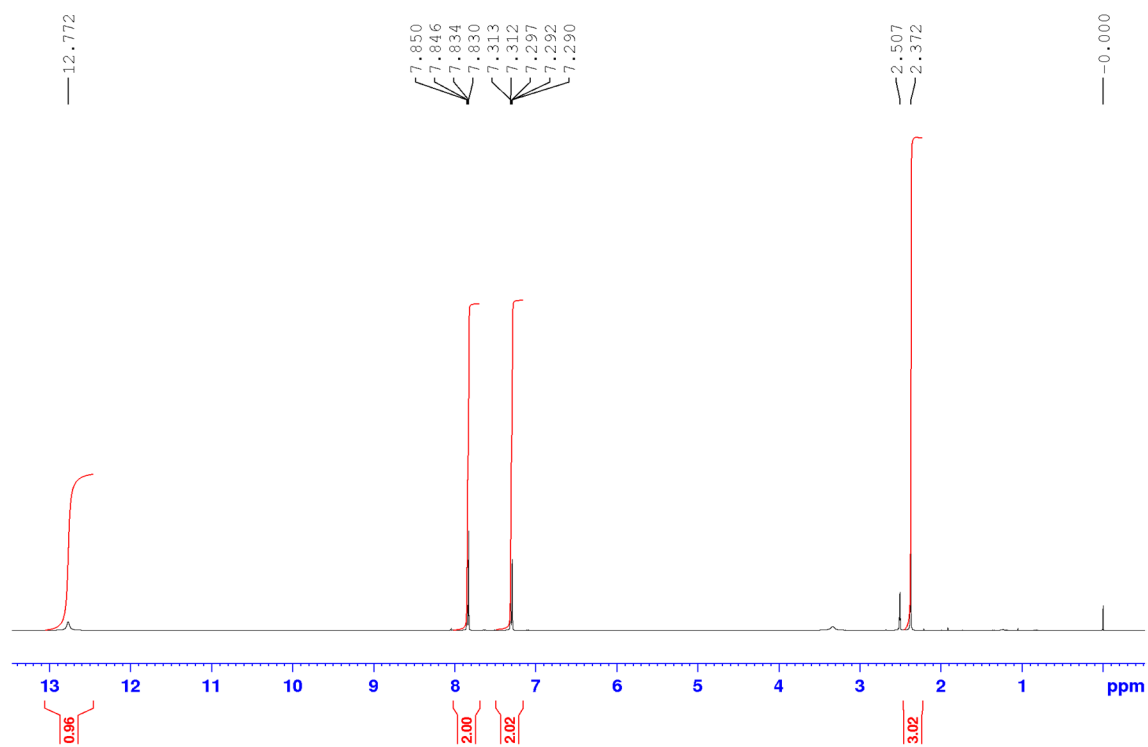


**<sup>13</sup>C NMR (100MHz, DMSO, 25 °C)**

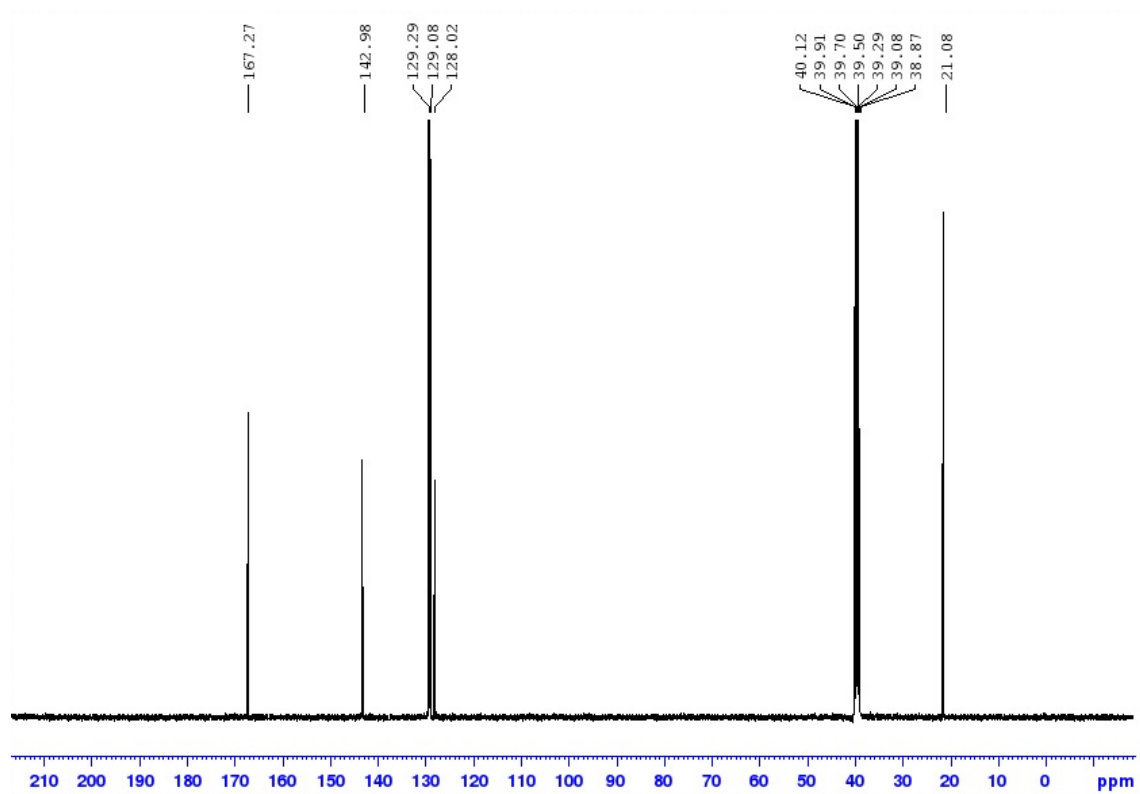


### 4-Methylbenzoic acid (5b)

$^1\text{H}$  NMR (400MHz, DMSO, 25 °C)

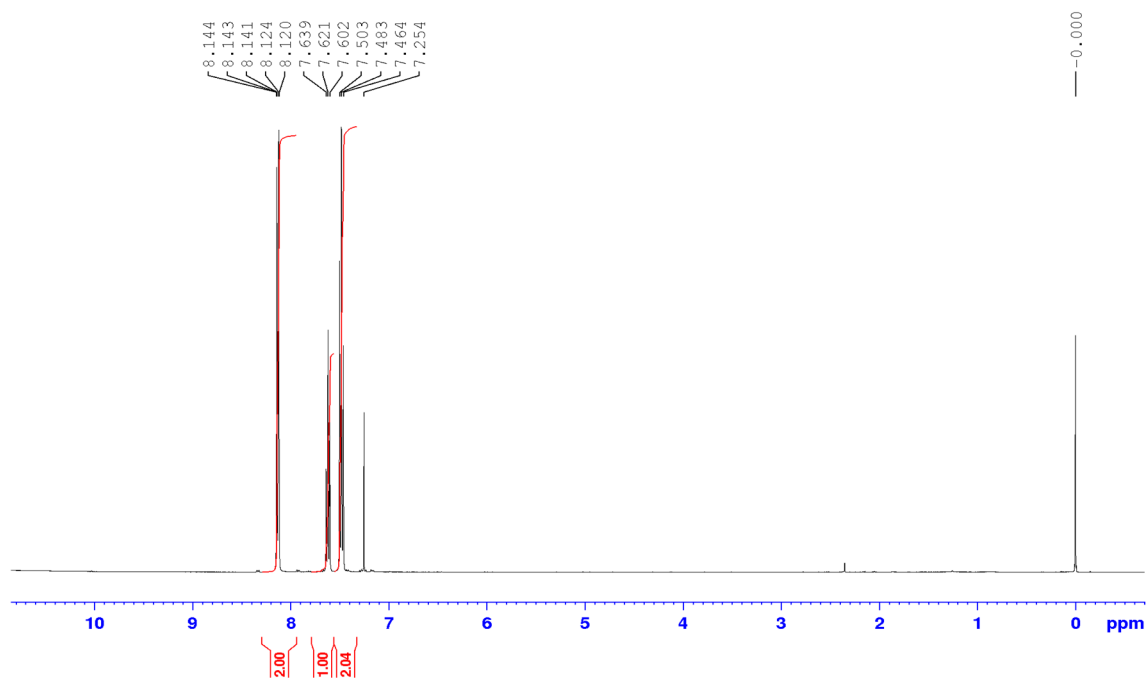


$^{13}\text{C}$  NMR (100MHz, DMSO, 25 °C)

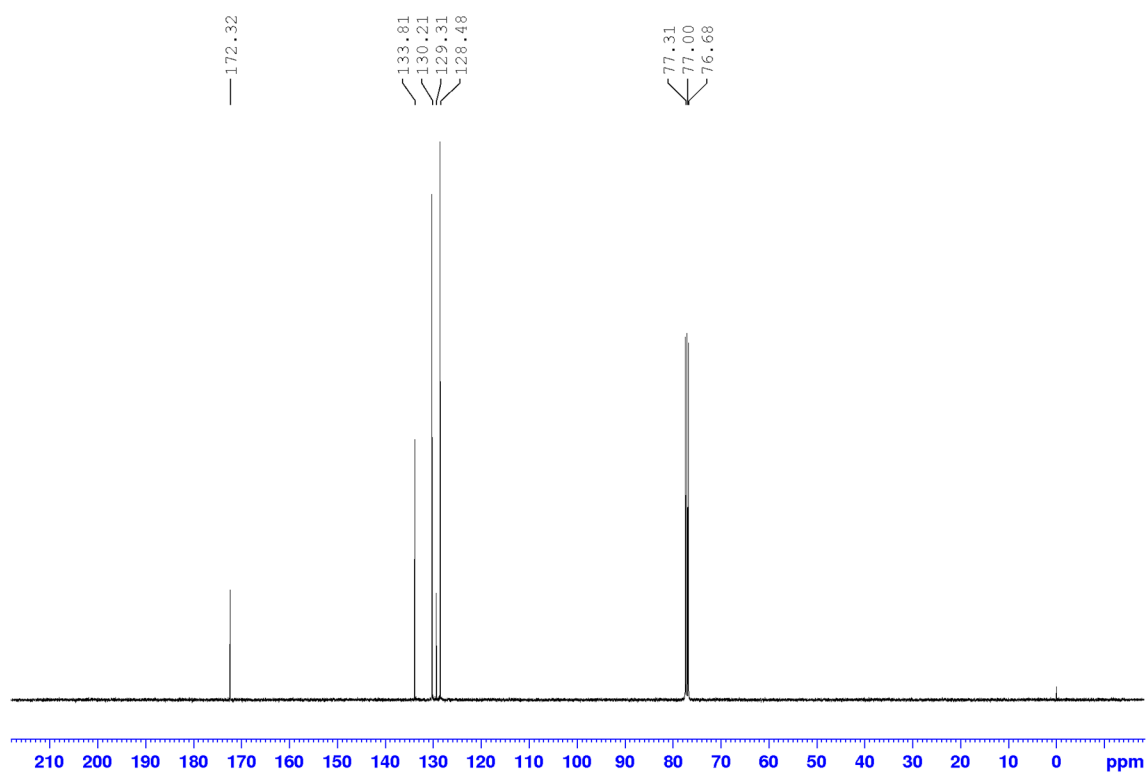


Benzoic acid (5c)

$^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ , 25 °C)

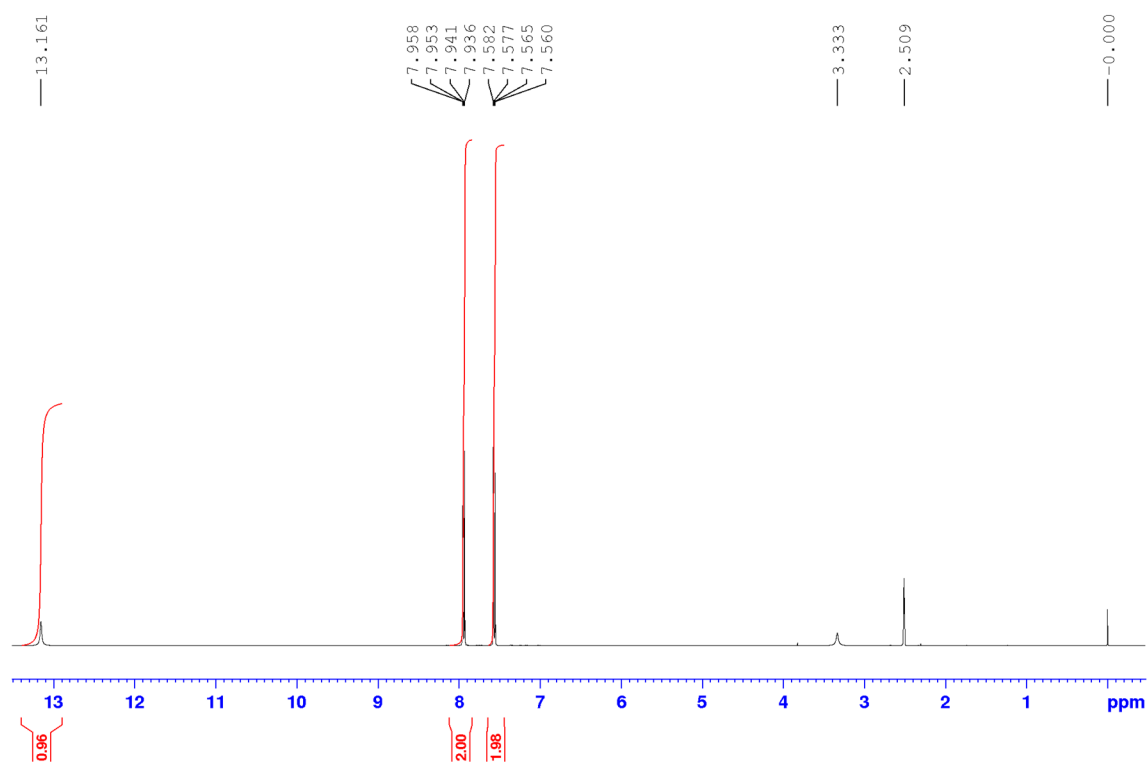


$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ , 25 °C)

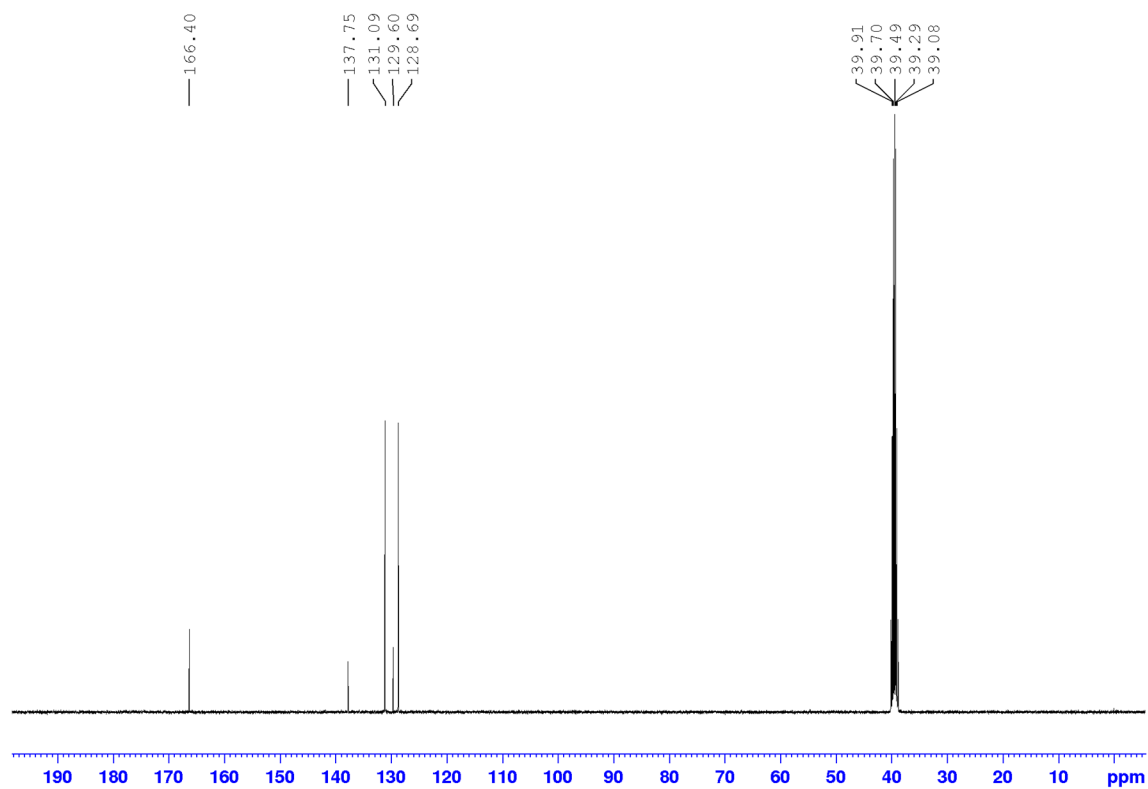


**4-Chlorobenzoic acid (5d)**

**<sup>1</sup>H NMR (400MHz, DMSO, 25 °C)**

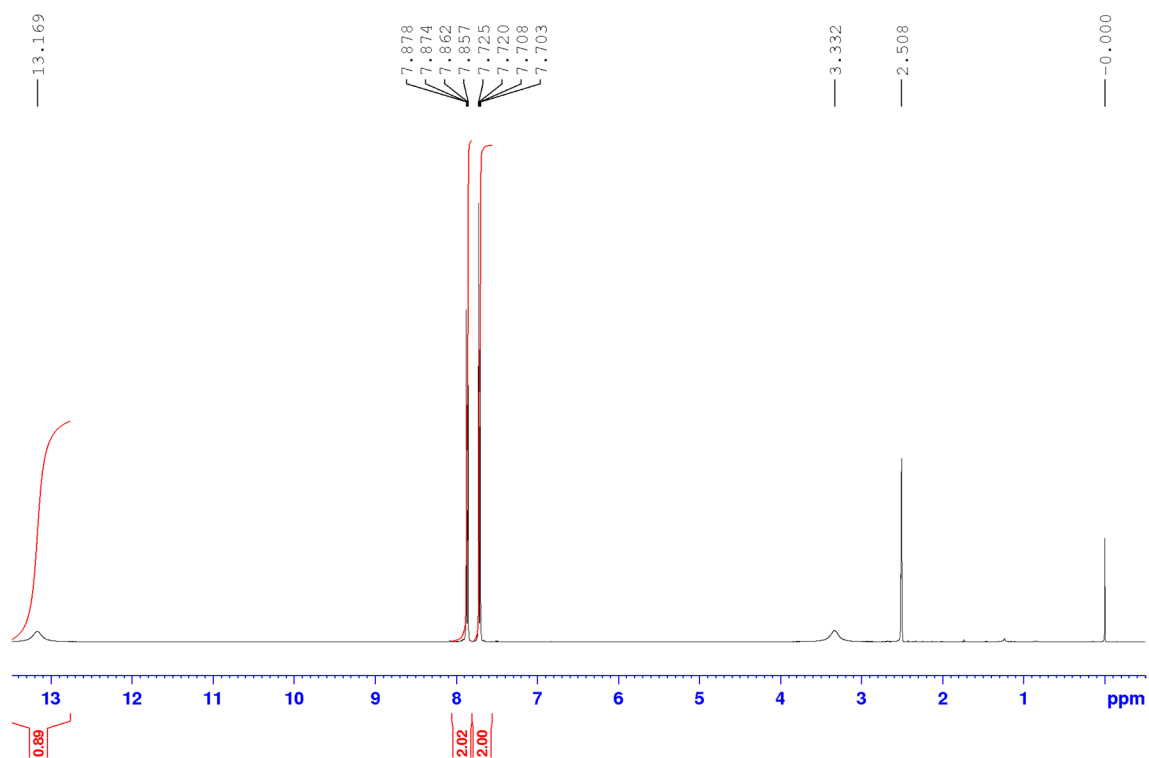


**<sup>13</sup>C NMR (100MHz, DMSO, 25 °C)**

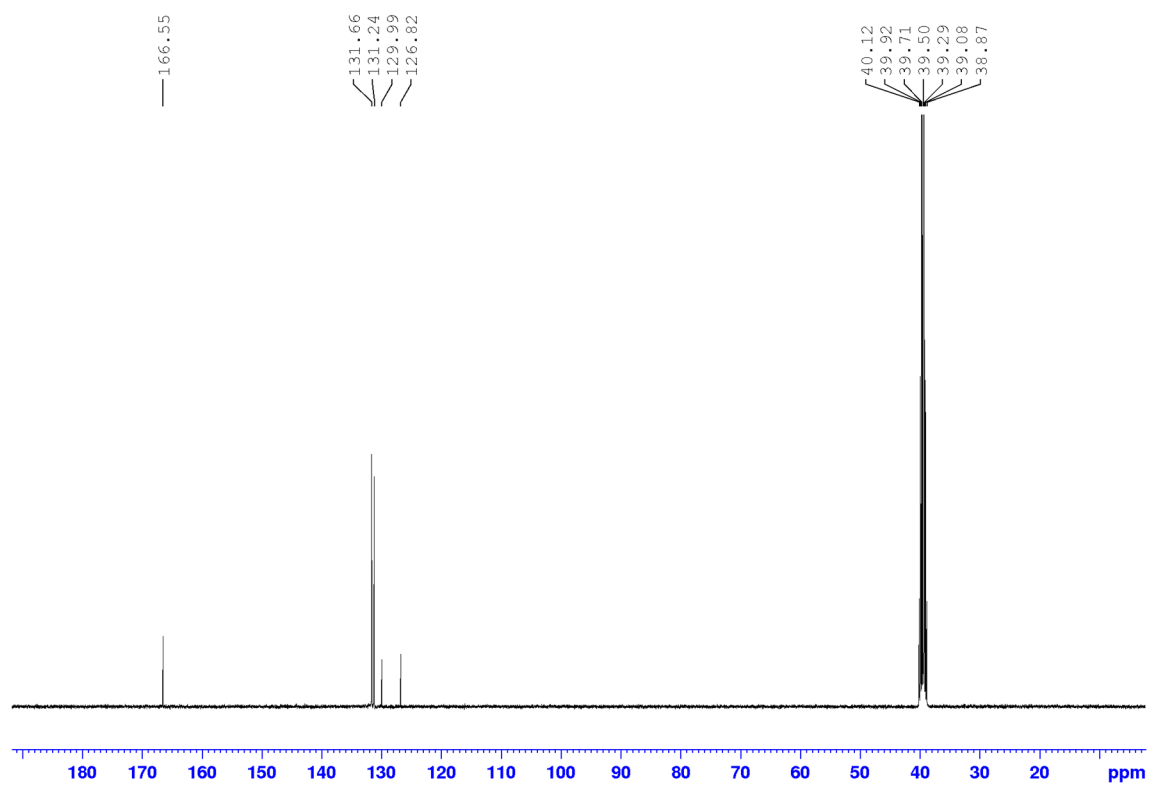


**4-Bromobenzoic acid (5e)**

**$^1\text{H}$  NMR (400MHz, DMSO, 25 °C)**

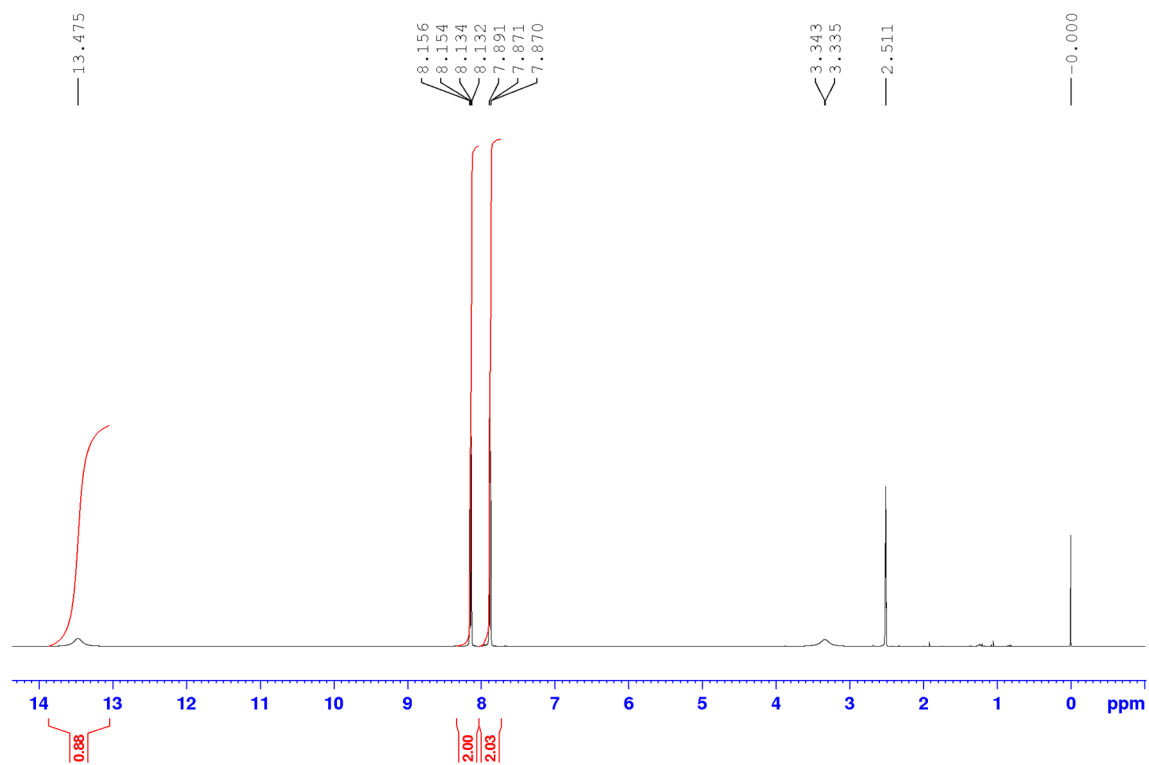


**$^{13}\text{C}$  NMR (100MHz, DMSO, 25 °C)**

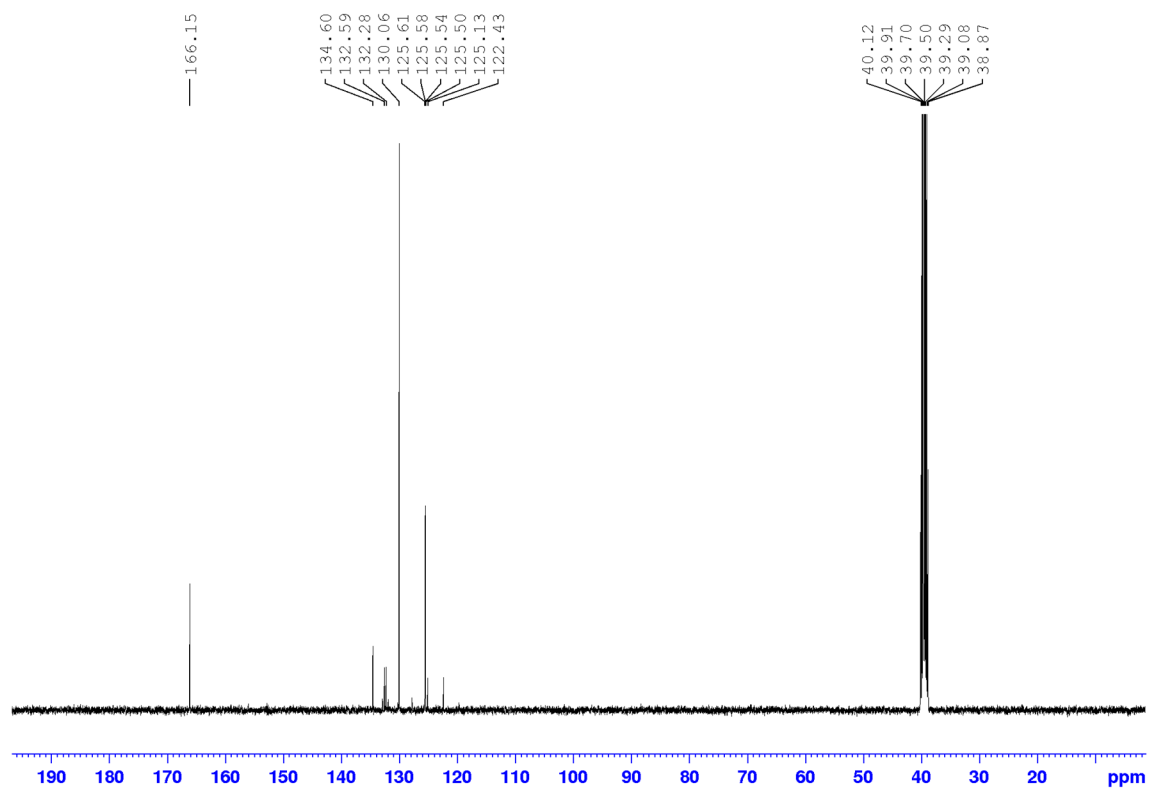


### 4-(Trifluoromethyl)benzoic acid (5f)

$^1\text{H}$  NMR (400MHz, DMSO, 25 °C)

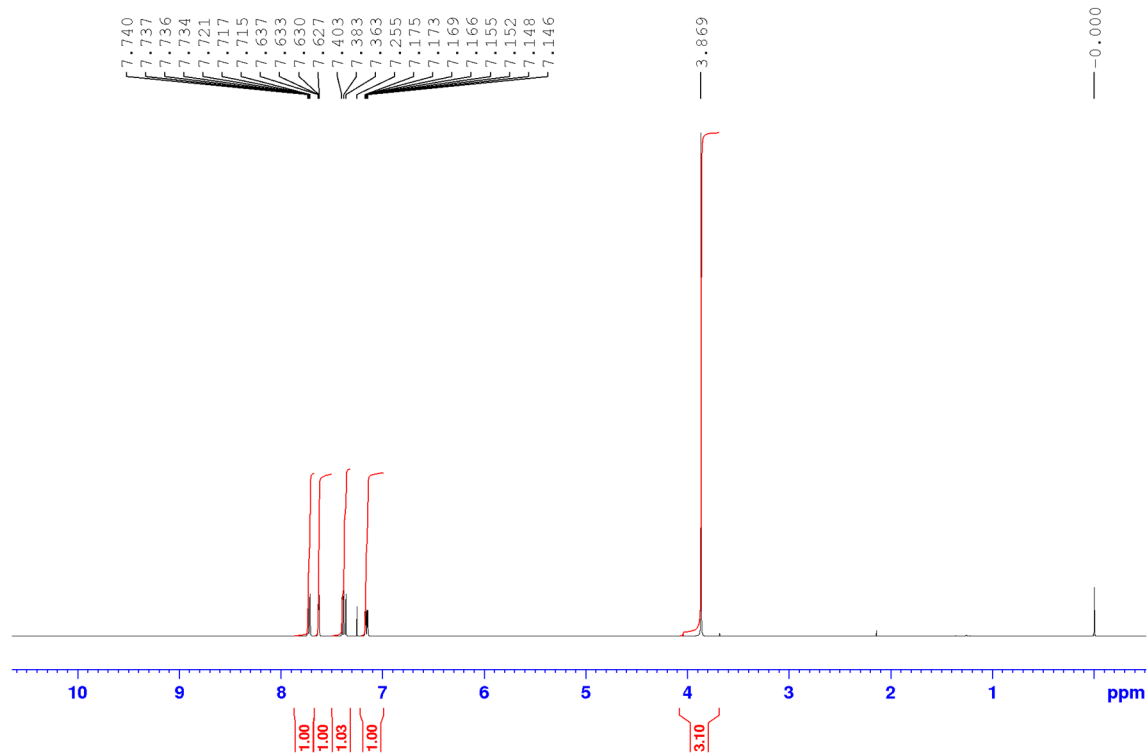


$^{13}\text{C}$  NMR (100MHz, DMSO, 25 °C)

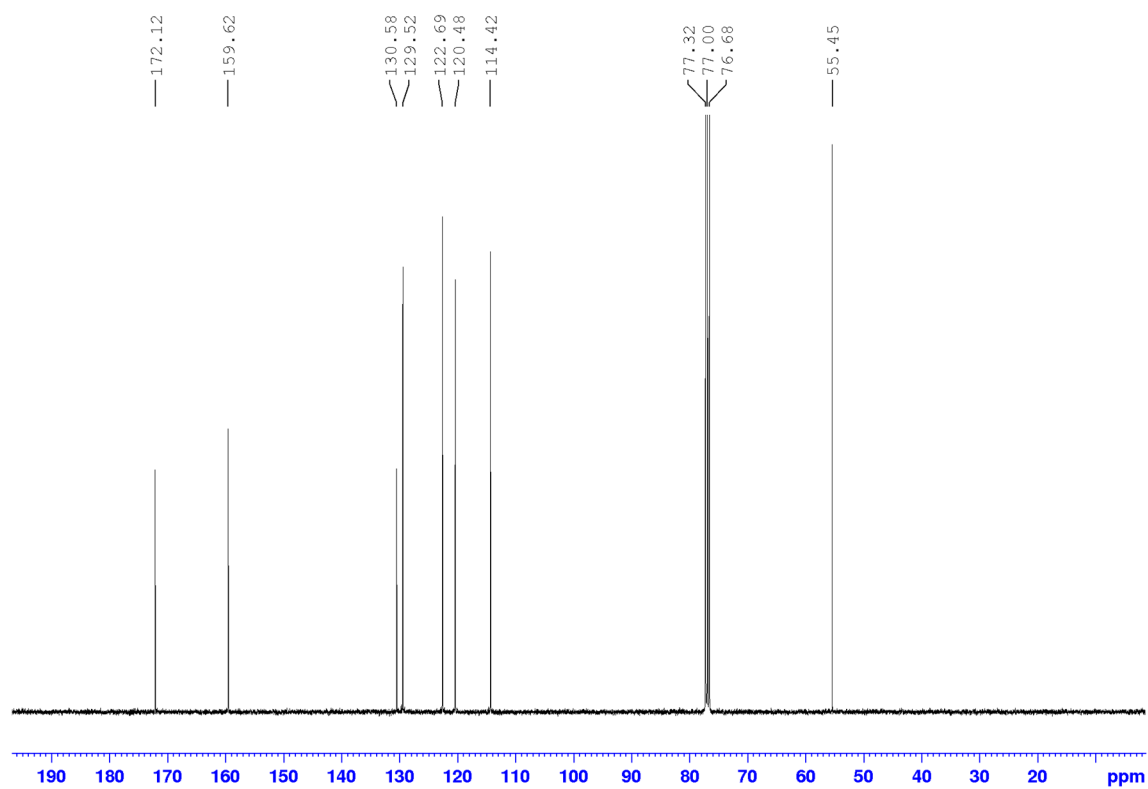


### 3-Methoxybenzoic acid (5g)

$^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ , 25 °C)

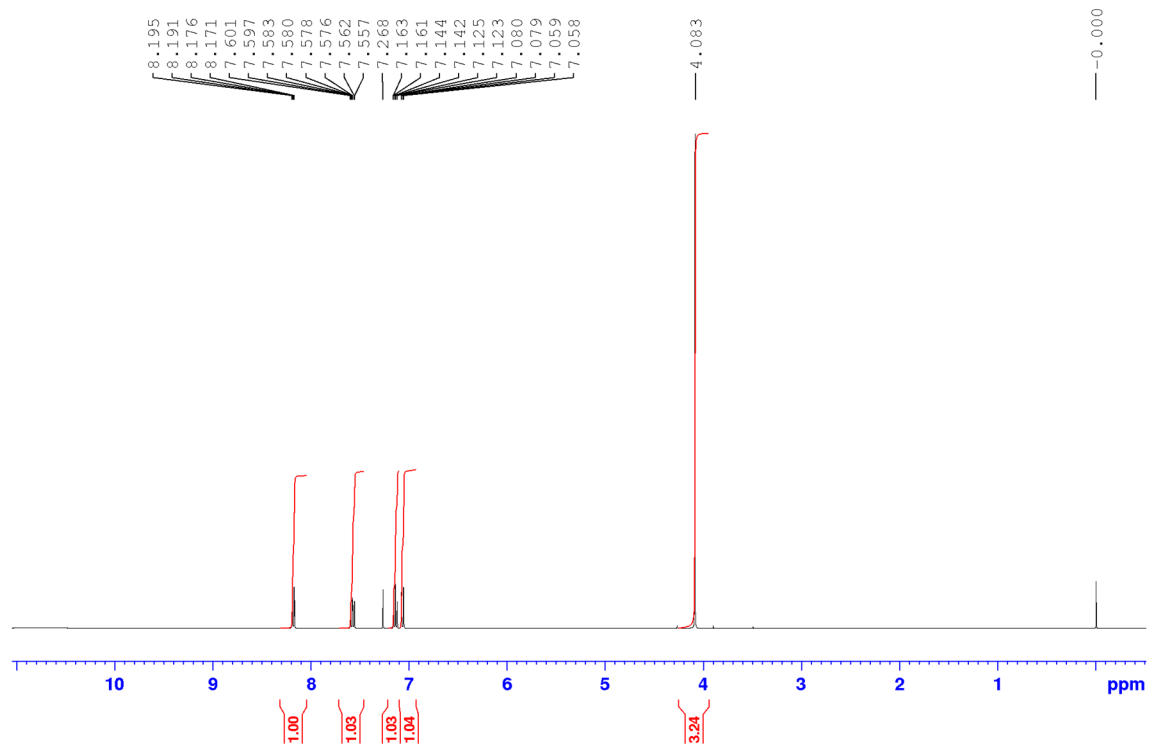


$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ , 25 °C)

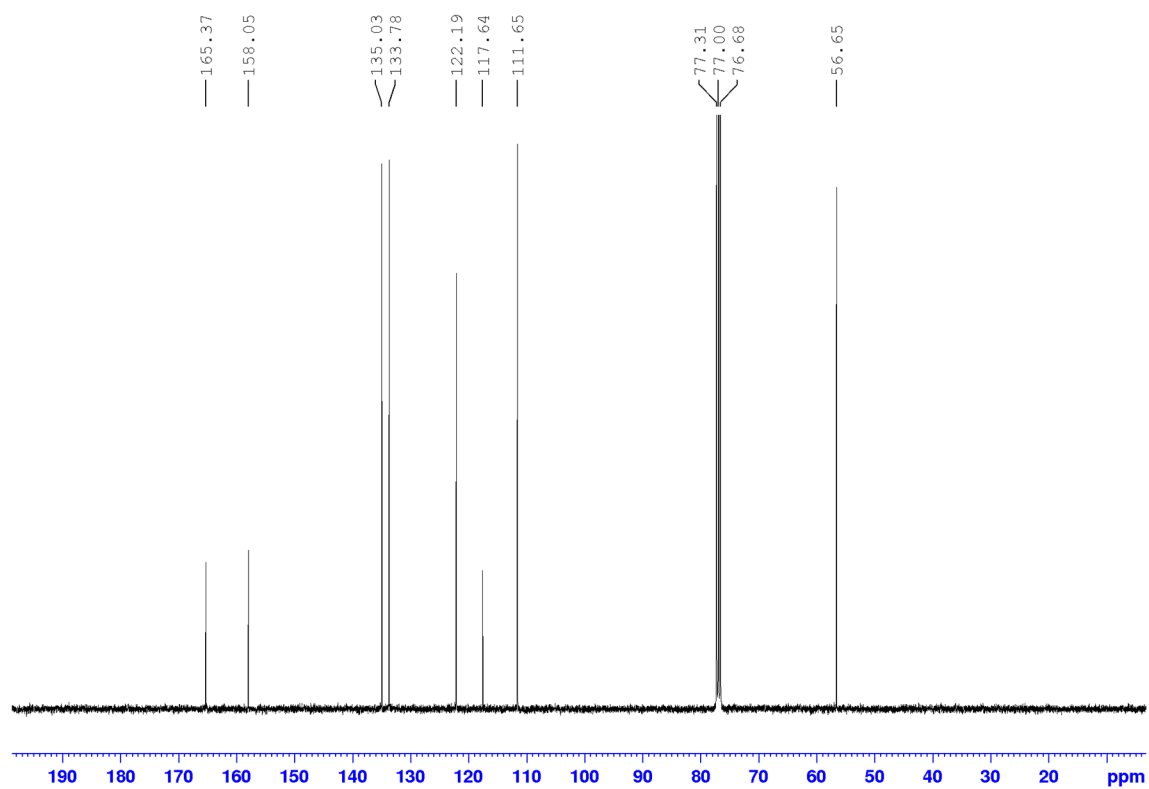


## 2-Methoxybenzoic acid (5h)

$^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ , 25 °C)



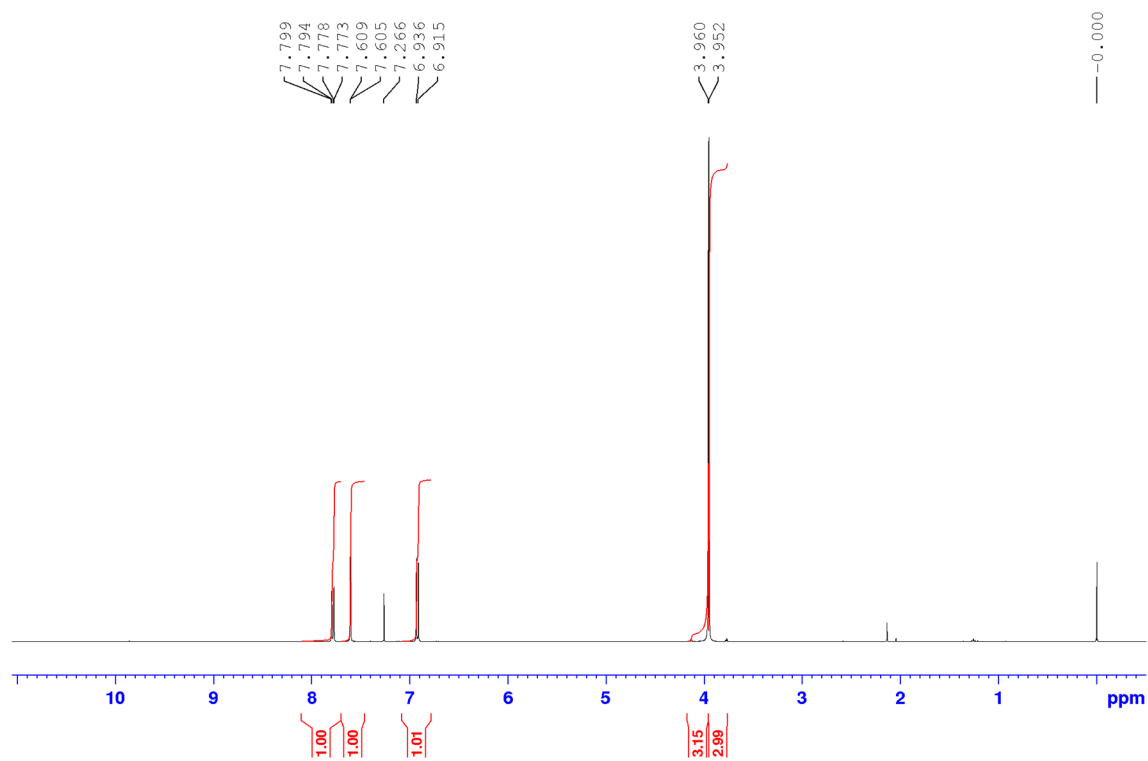
$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ , 25 °C)



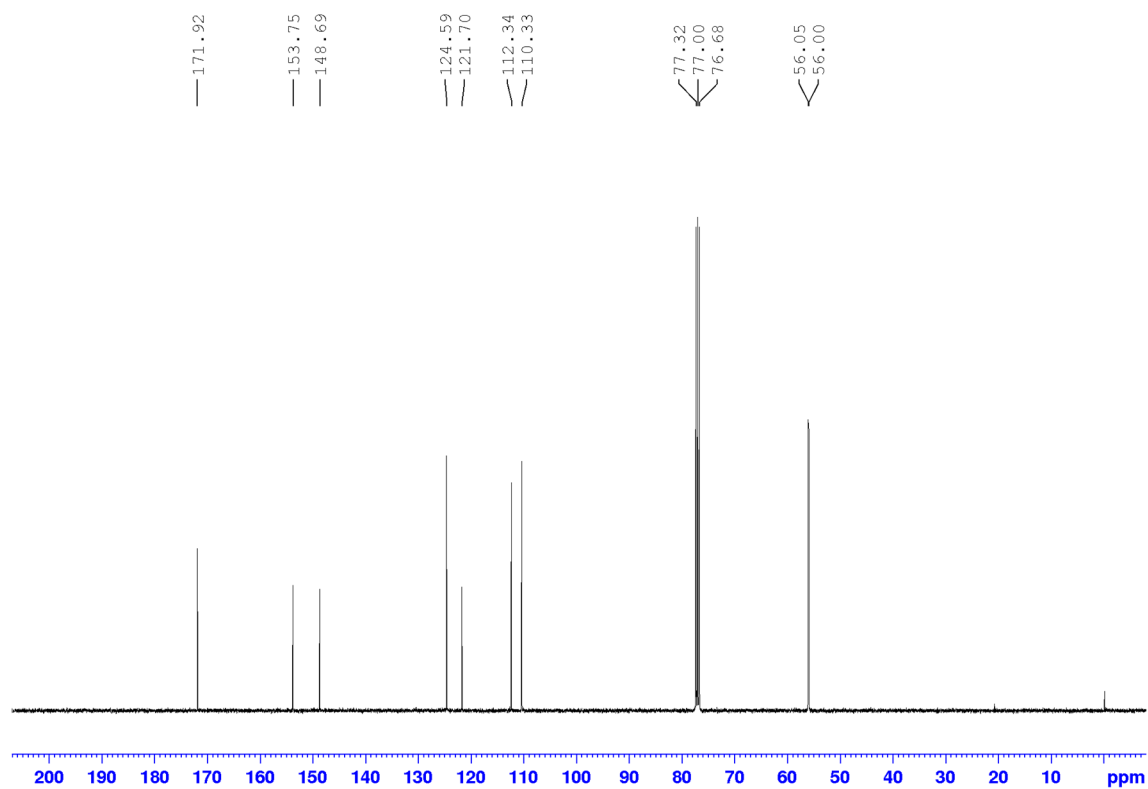


**3,4-Dimethoxybenzoic acid (5i)**

**<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>, 25 °C)**

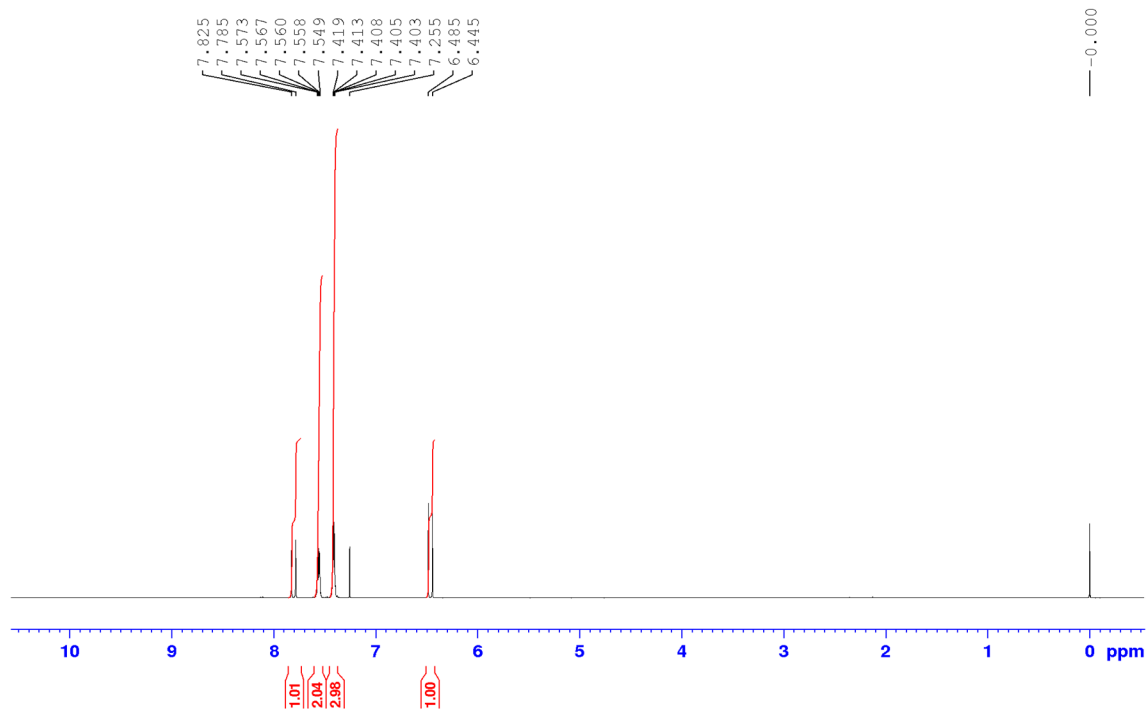


**<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>, 25 °C)**

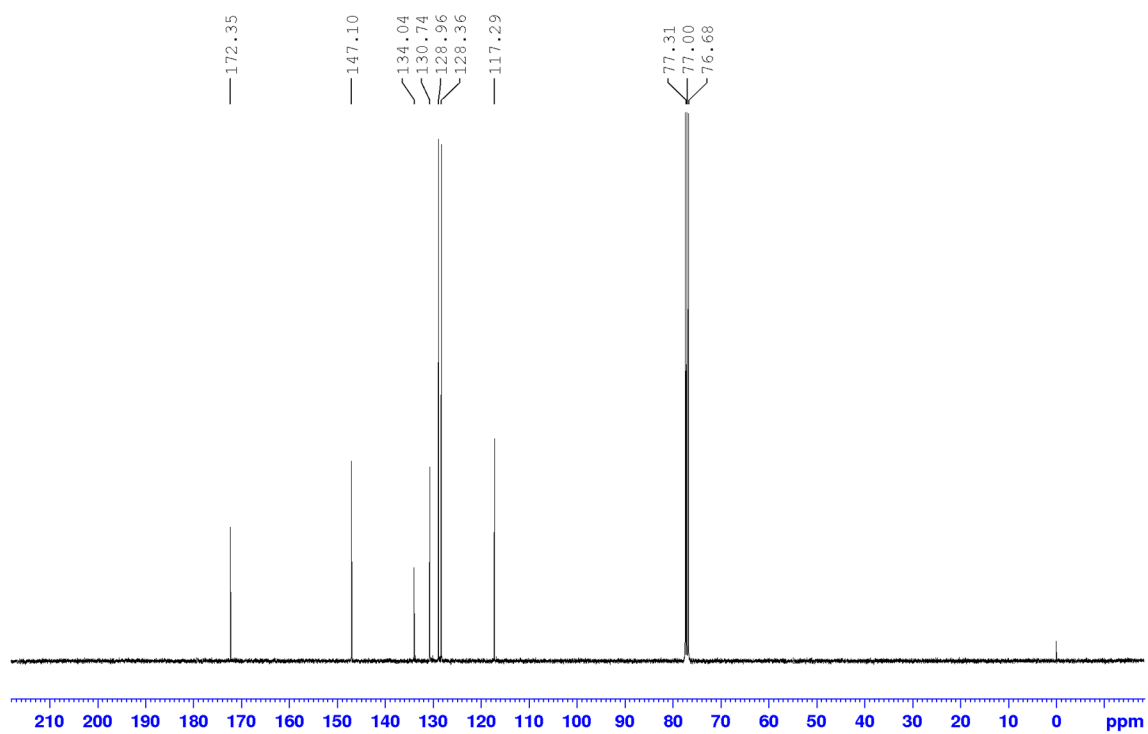


**(E)-cinnamic acid (7a)**

**<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>, 25 °C)**

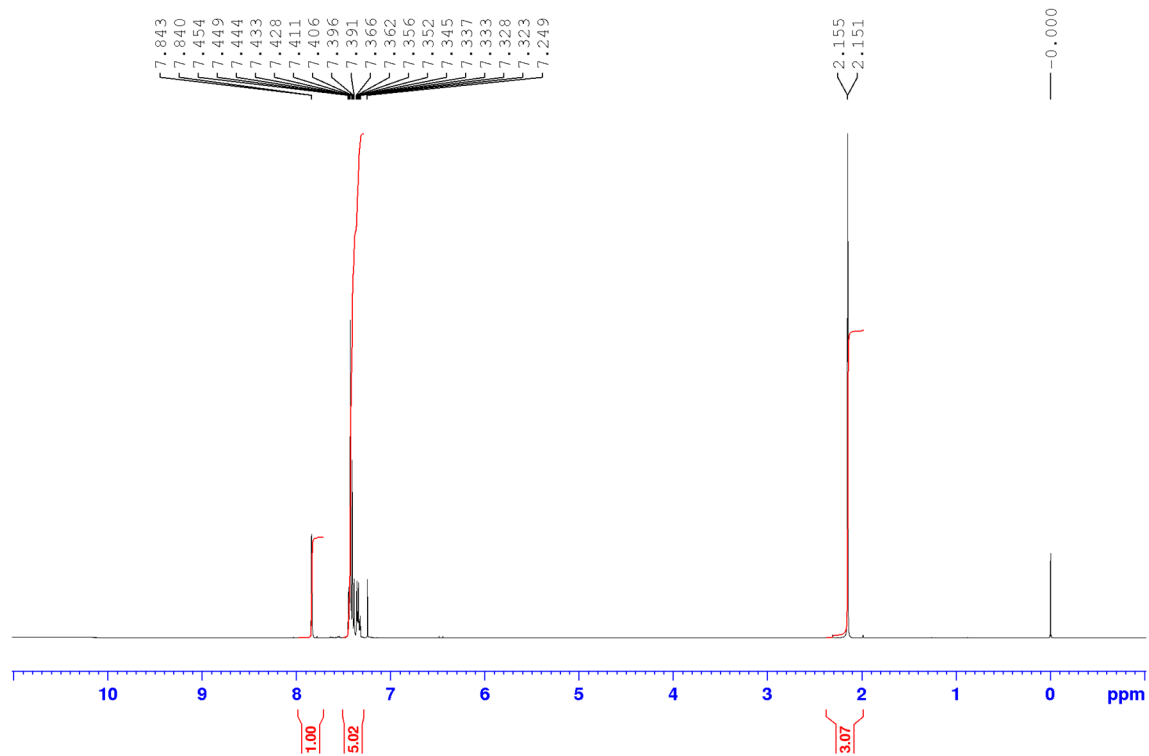


**<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>, 25 °C)**

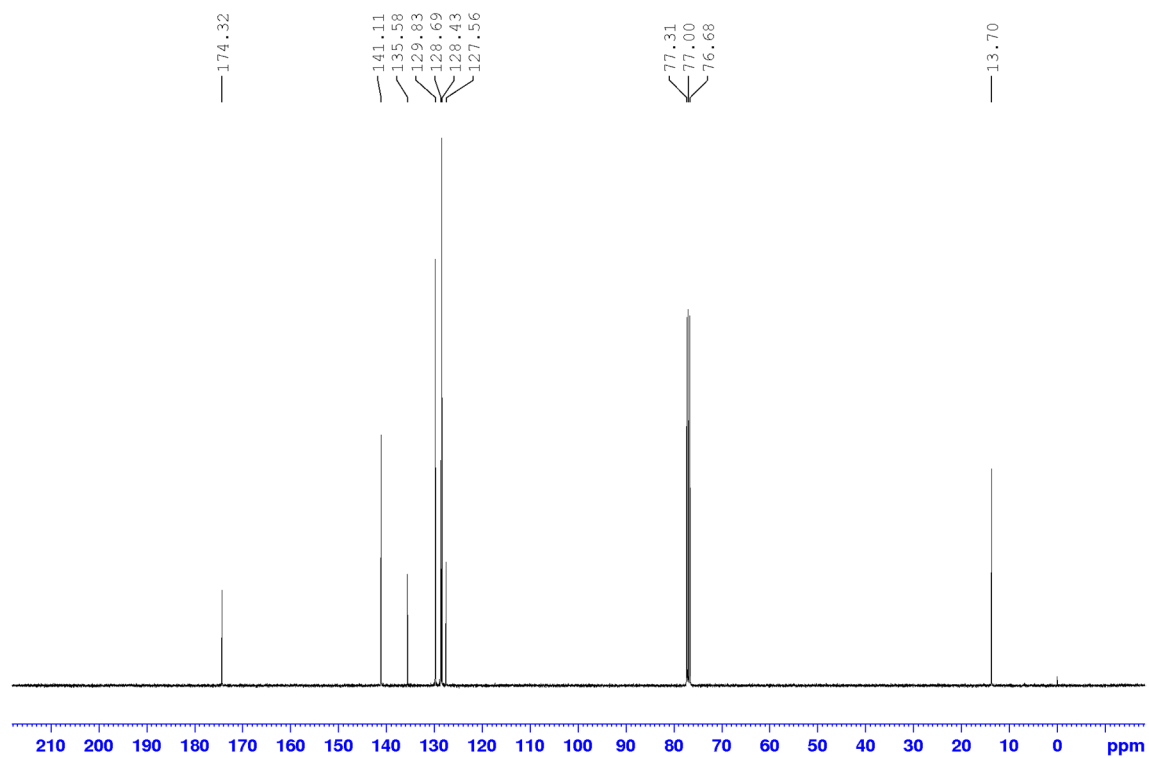


2-Methyl-3-phenyl-2-propenoic acid (7b)

$^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ , 25 °C)

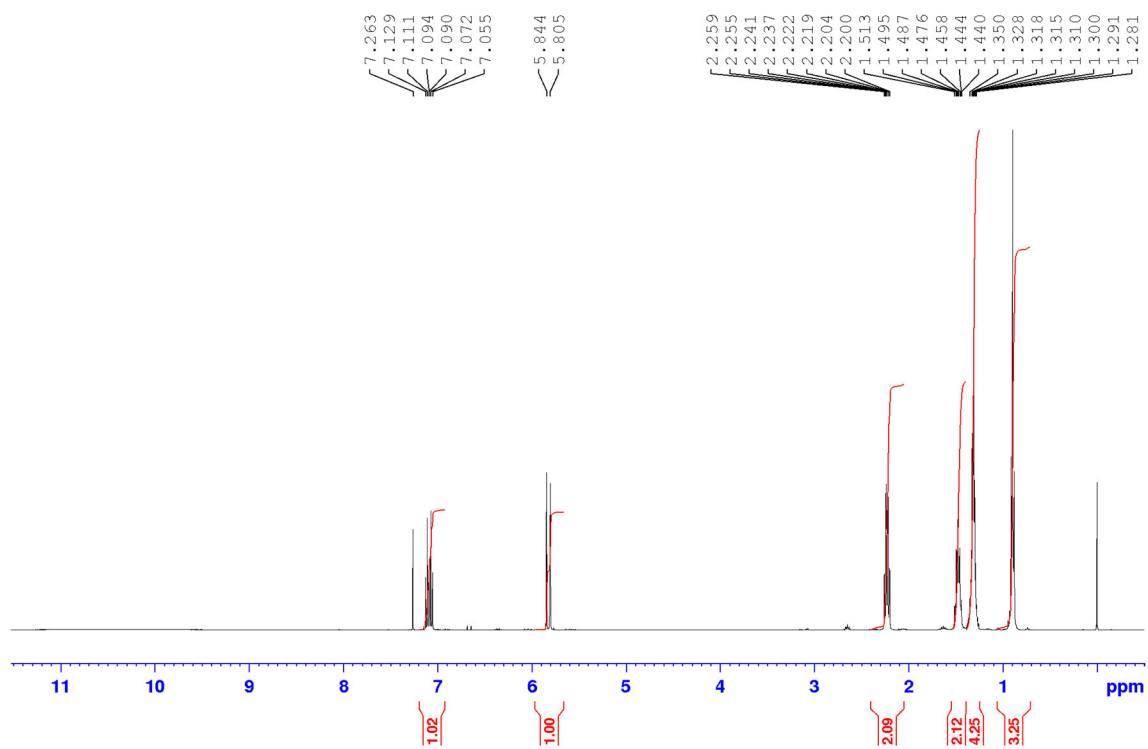


$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ , 25 °C)

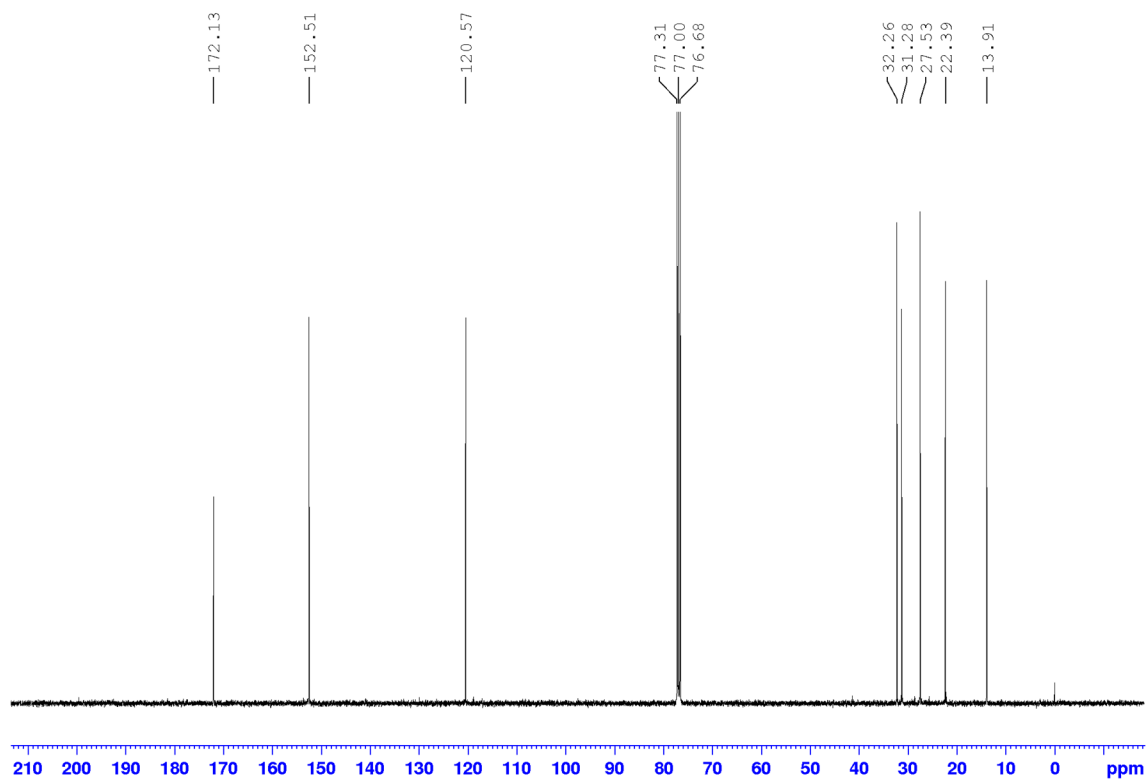


**(E)-2-Octenoic acid (7c)**

**<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>, 25 °C)**

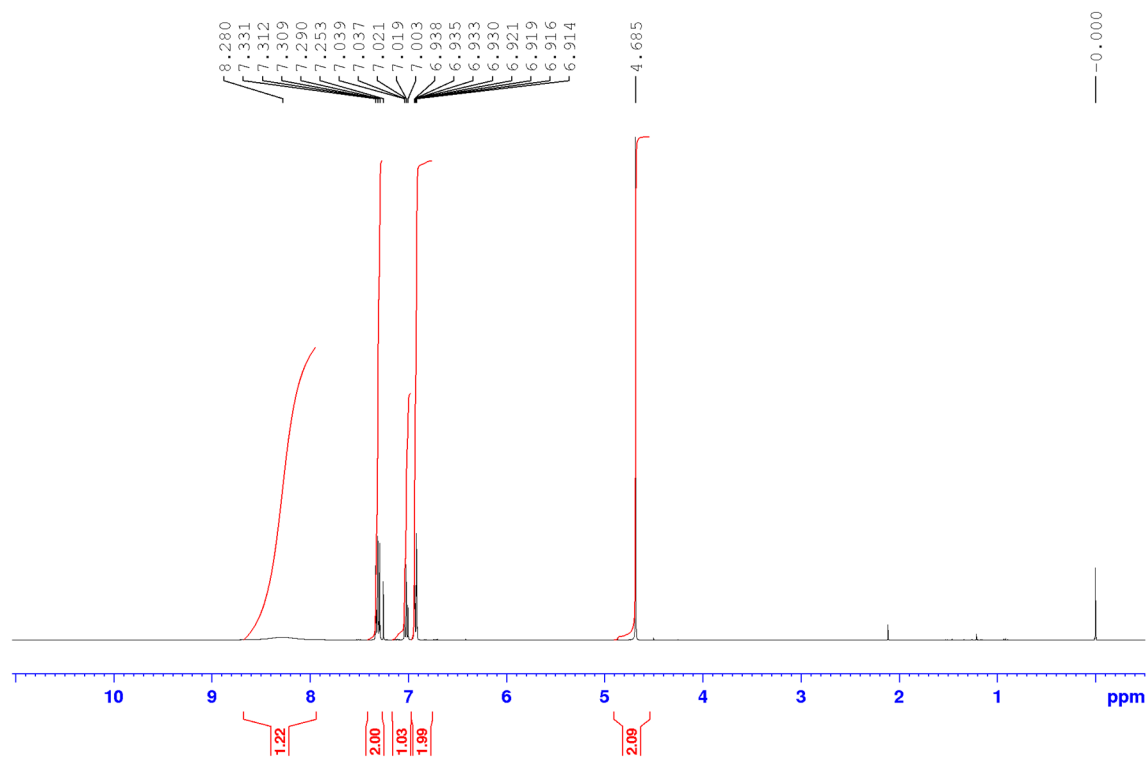


**<sup>13</sup>C NMR (100MHz, CDCl<sub>3</sub>, 25 °C)**

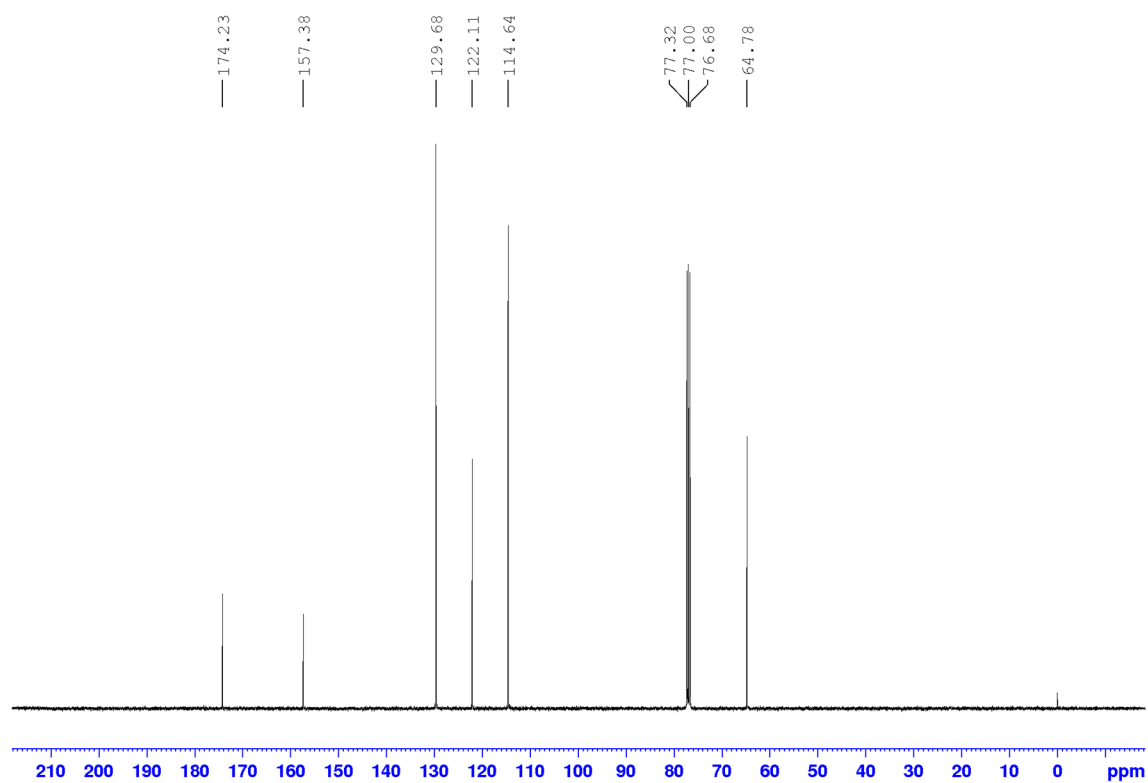


Phenoxyacetic acid (9)

$^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ , 25 °C)

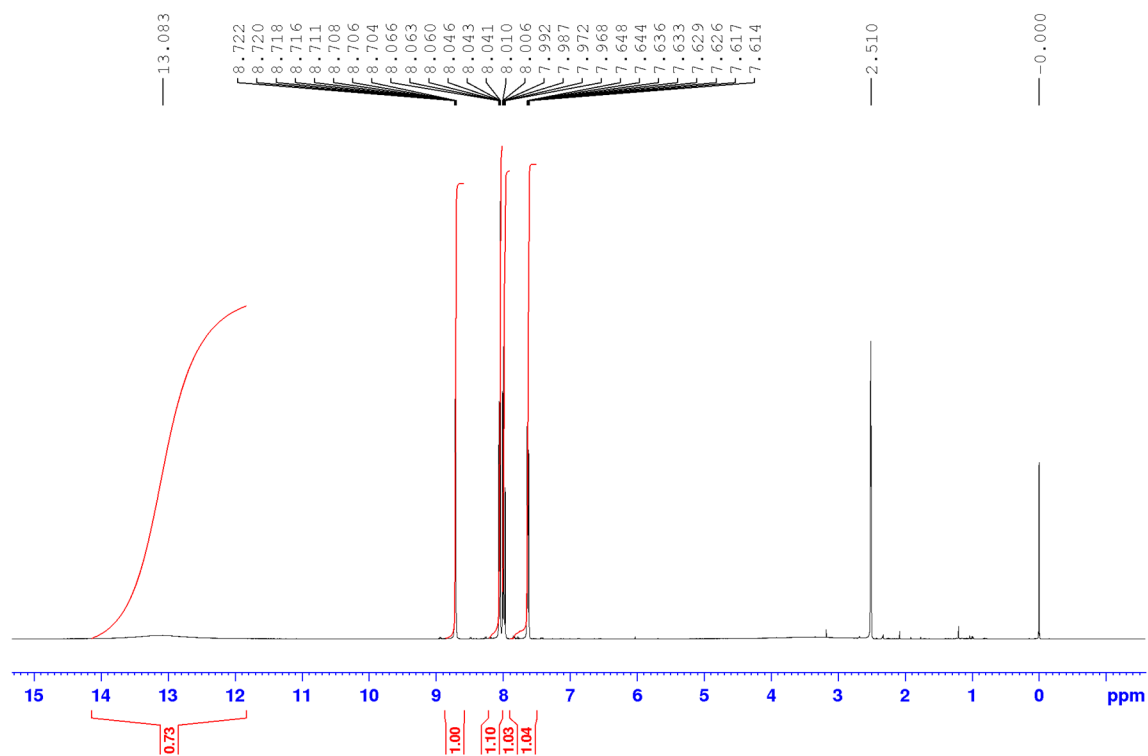


$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ , 25 °C)

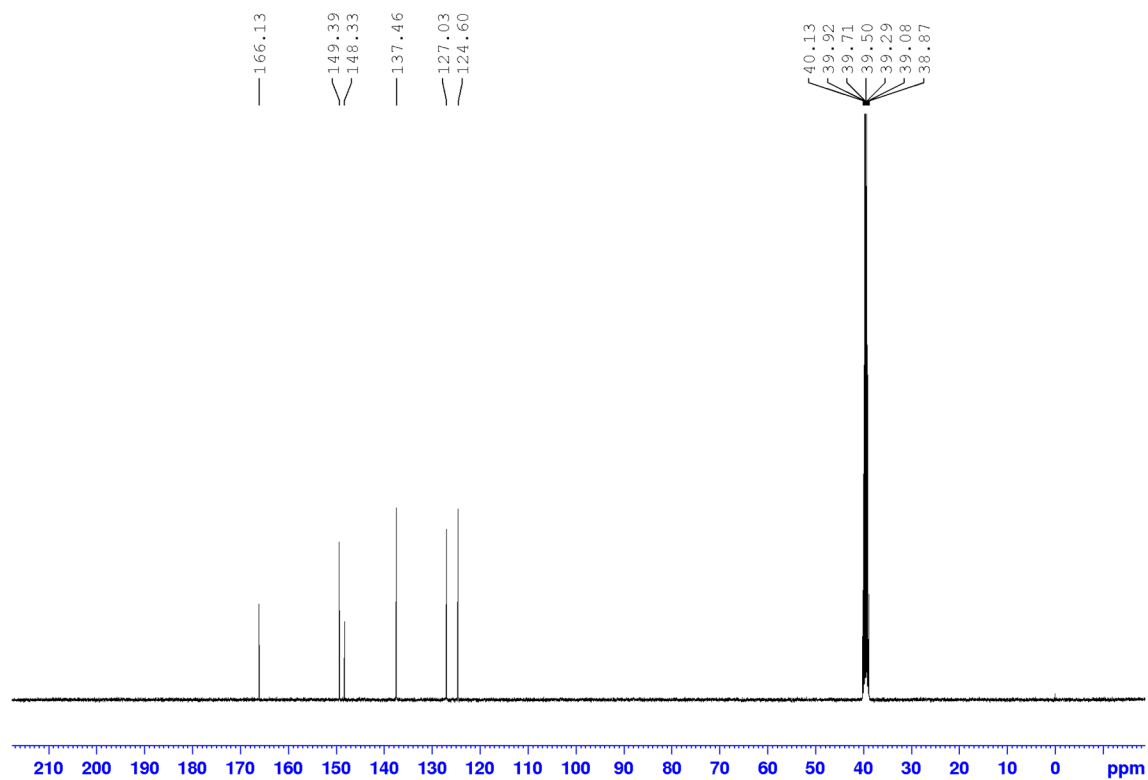


**2-Pyridinecarboxylic acid (11a)**

**<sup>1</sup>H NMR (400MHz, DMSO, 25 °C)**

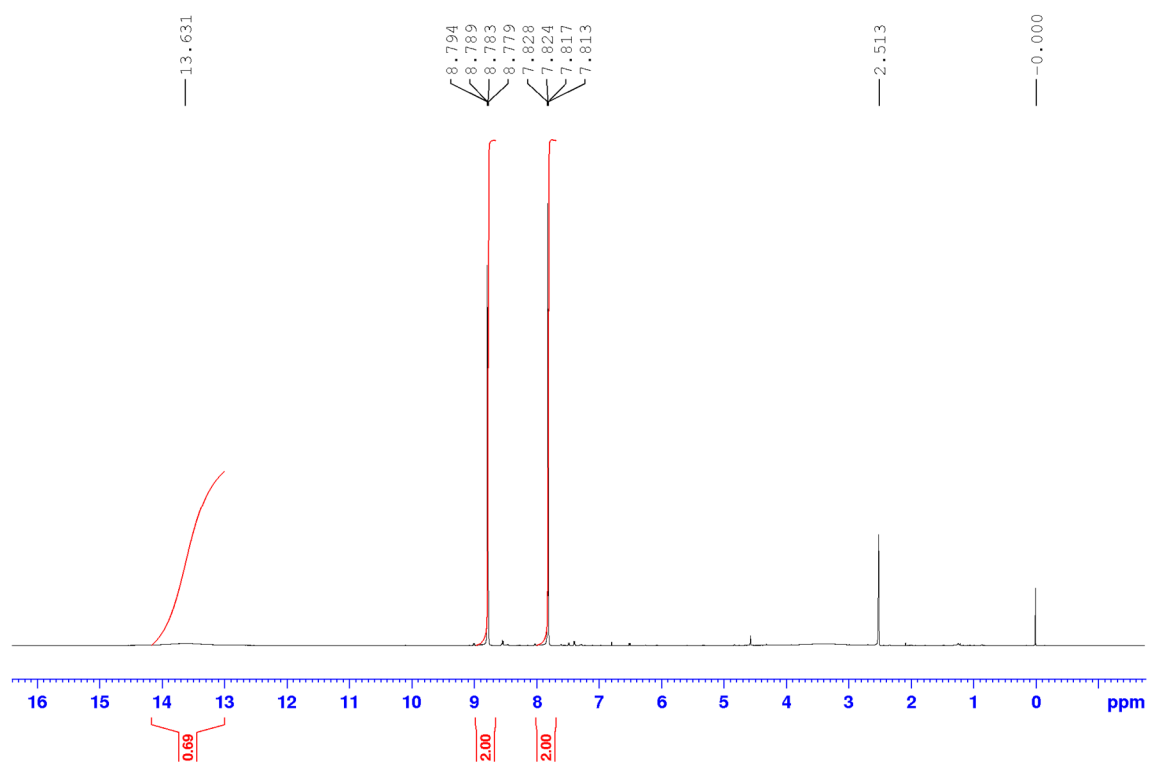


**<sup>13</sup>C NMR (100MHz, DMSO, 25 °C)**

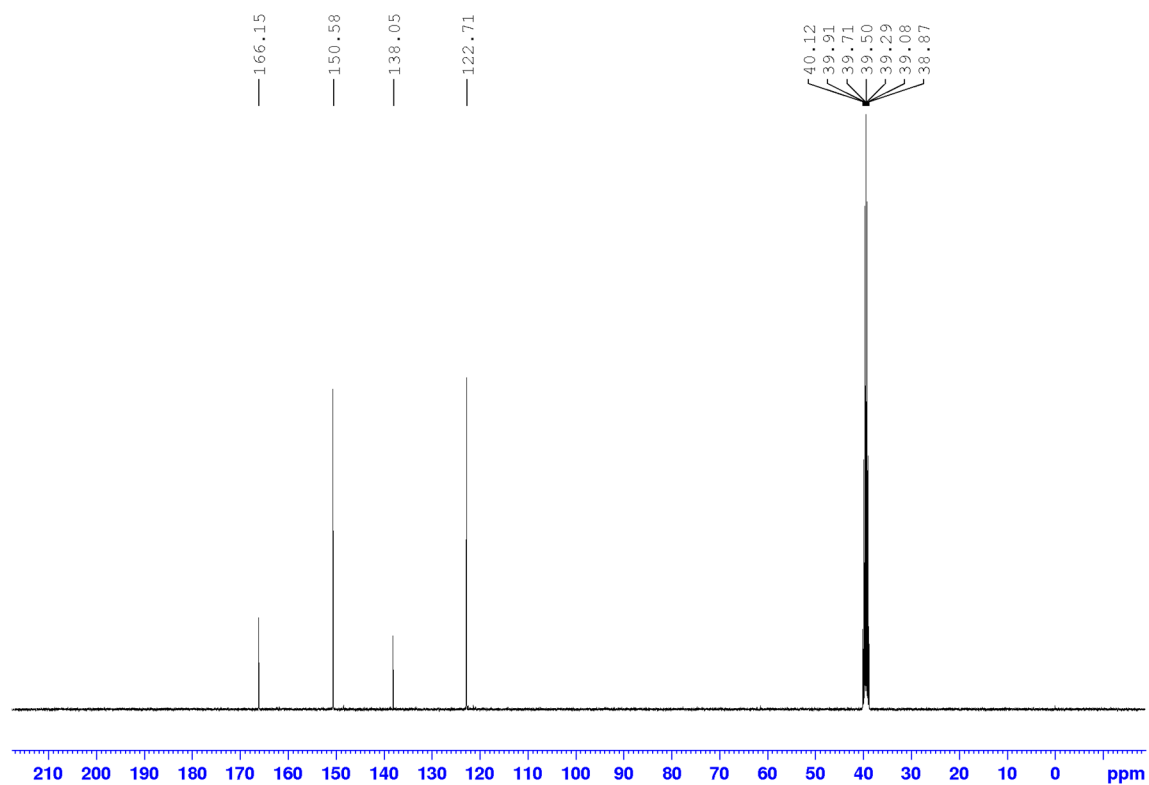


**4-Pyridinecarboxylic acid (11b)**

**<sup>1</sup>H NMR (400MHz, DMSO, 25 °C)**

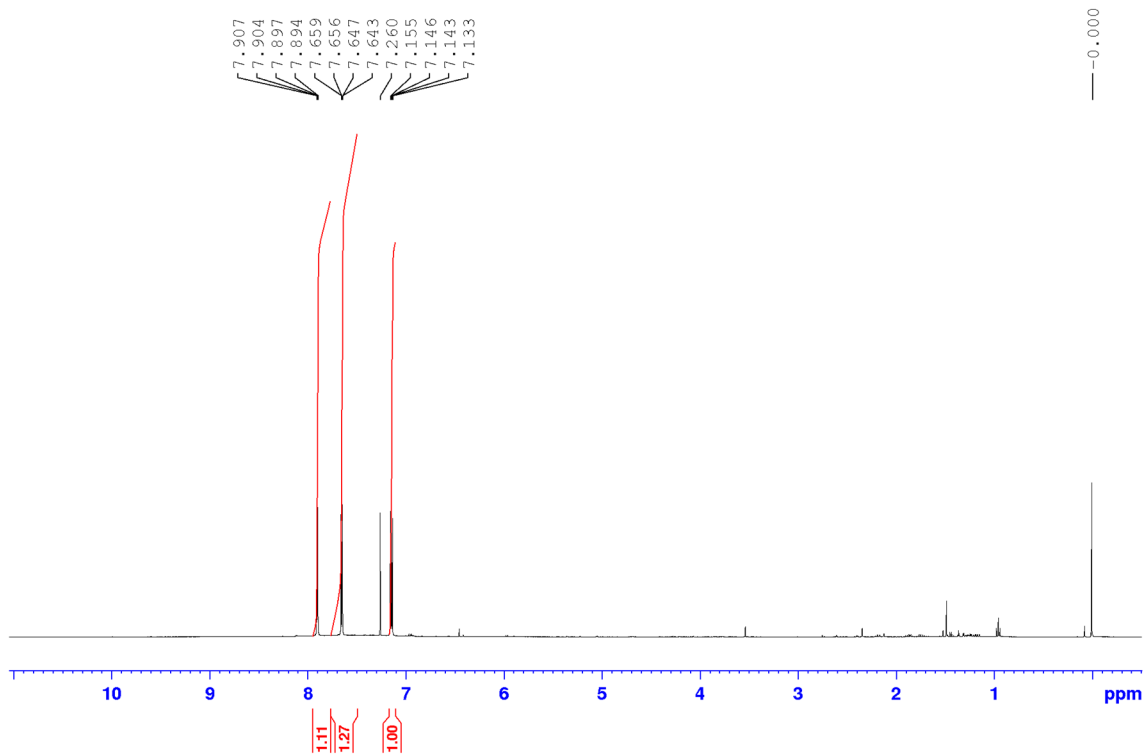


**<sup>13</sup>C NMR (100MHz, DMSO, 25 °C)**



**2-Thiophenecarboxylic Acid (11c)**

**$^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ , 25 °C)**



**$^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ , 25 °C)**

