Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information

Iodine Catalyzed Oxidation of Alcohols and Aldehydes to Carboxylic Acids in Water: A Metal-Free Route to Synthesis of Furandicarboxylic Acid and Terephthalic Acid

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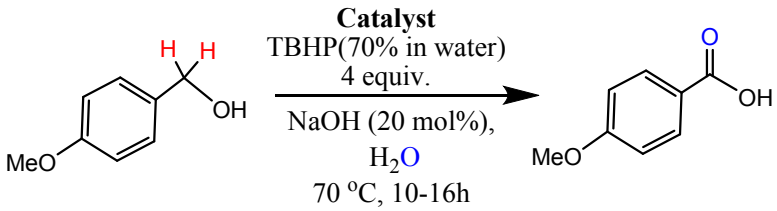
General Information

Unless otherwise stated, all reactions were performed under open atmosphere. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Spectrospin DPX-300 NMR spectrometer at 300 and 75.47 MHz, respectively. All chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. All chemical shifts are related to residual solvent peaks [$CDCl_3$: 7.26 (^{1}H), 77.16 (^{13}C); DMSO-d_6: 2.50 (^{1}H), 39.52 (^{13}C)]. All alcohols and aldehydes were purchased from commercial sources and hydroxymethylfurfural (HMF) was prepared according to literature procedure.[1] Deionised water, TBHP (70% in water), iodine and NaOH were used as received.

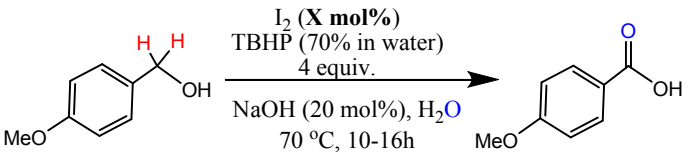
Experimental Section:

Table S1: Effect of catalysts on the C-H oxidation.



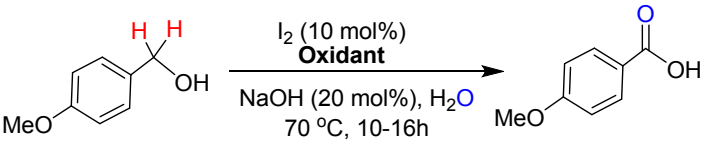
Entry No.	**Catalyst**	% of yield (isolated)
1	-	-
2	I_2	90
3	KI	48
4	NaI	46
5	TBAI	15
6	TBAB	0

Table S2: Effect of amount of catalyst loading for the oxidation reaction.



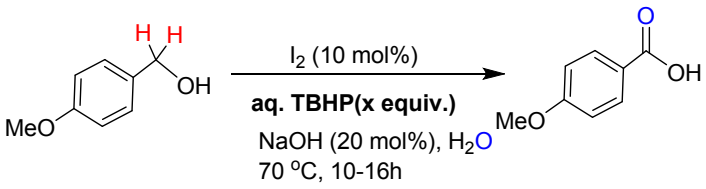
Entry No.	Amount of I_2 (X mol%)	% of yield (isolated)
1	**-**	**-**
2	2	23
3	4	35
4	5	46
5	6	58
6	8	63
7	**10**	**90**
8	12	90
9	15	90
10	20	90

Table S3: Effect of oxidant used for the reaction.



Entry No.	**Oxidant**	% of yield (isolated)
1	**-**	**-**
2	aq. TBHP	90
3	O_2	0
4	H_2O_2	5
5	DTBP	0
6	$K_2S_2O_8$	0
7	NMO	0

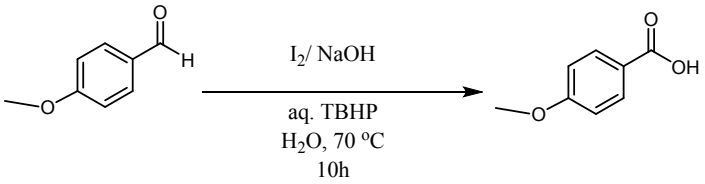
Table S4: Effect of amount of oxidant used for oxidation reaction



Entry No.	**aq. TBHP (x equiv.)**	% of yield (isolated)
1	**-**	**-**
2	1	15
3	2	32
4	3	47
5[a]	**4**	**90**
6	5	90
7	10	90

a. Since the TBHP used was 70% in water, it is equivalent to 2.8 equivalent of pure TBHP.

Table S5: Optimization of reaction conditions for oxidation of aldehyde



Entry No	Iodine (X mol%)	aq. TBHP (Y equiv.)	NaOH (Z mol%)	Reaction temperature/°C	Yield (isolated)
1	-	-	-	70	-
2	5	1	10	70	58
3	5	1.5	10	70	68
4	**5**	**2**	**10**	**70**	**73**
5	10	1.5	20	70	77
6	**10**	**2**	**20**	**70**	**84**
7	10	4	20	70	95

Table S6: Effect of base, solvent and temperature used for the reaction

Entry No.	Base (20mol%)	Solvent	Tempereture /^{o}C	% of yield (isolated)
1	NaOH	H_2O	RT	0
2	NaOH	H_2O	50	42
3	NaOH	H_2O	60	65
4[a]	**NaOH**	**H_2O**	**70**	**90**
5	KOH	H_2O	70	81
6	Na_2CO_3	H_2O	70	37
7	K_2CO_3	H_2O	70	31
8	-	H_2O	70	0
9	NaOAc	H_2O	70	30
10	KOAc	H_2O	70	26
11	NaOH	DMSO	70	0
12	NaOH	DCM	70	0
13	NaOH	1,4 dioxane	70	0
14	NaOH	CH_3CN	70	31
15	NaOH	tert-Butanol	70	38

a. Reaction was also carried out with 1 equiv. of NaOH at 100 ^{o}C with yield of 90%.

General procedure for oxidation of alcohols

A 15 ml screw capped vial was charged with a magnetic bead, 5 mmol of alcohol, 0.5 mmol (10 mol%) of I_2 and 20 mmol (4 equiv.) of aq. TBHP (70% in H_2O). Afterwards, 1 mmol (20 mol%) of NaOH with additional 2 mL of deionized water (pH = 10) were added to the reaction mixture and it was heated at 70 °C for 10-16 h. Afterwards, the reaction mixture was neutralized by aq. HCl and extracted with EtOAc and aq. $Na_2S_2O_3$ solution. The organic layer was dried over anhydrous Na_2SO_4 and after evaporation of the solvent analytically pure carboxylic acids were obtained.[2,3]

General procedure for oxidation of aldehydes

A 15 ml screw capped vial was charged with a magnetic bead, 5 mmol of aldehyde, 0.5 mmol (10 mol%) of I_2 and 20 mmol (4 equiv.) of aq. TBHP. Afterwards, 1 mmol (20 mol%) of NaOH with additional 2 mL of deionized water (pH =10) were added to the reaction mixture and it was heated at 70 °C for 10-16 h. Afterwards, the reaction mixture was neutralized by aq. HCl and extracted with EtOAc and aq. $Na_2S_2O_3$ solution. The organic layer was dried over anhydrous Na_2SO_4 and after evaporation of the solvent analytically pure carboxylic acids were obtained.[2,3]

Gram scale synthesis of FDCA from HMF

A 1000 ml of round bottom flask was charged with a magnetic bead, 79.36 mmol (10 gm) of HMF, 15.87 mmol (20 mol%) of I_2 and 634 mmol (8 equiv.) of aq. TBHP. Afterwards, 39.70 mmol (50 mol%) of NaOH with additional 100 mL of H_2O were added to the reaction mixture and it was heated at 70 °C for 36 h. Afterwards, the reaction mixture was neutralized by aq. HCl and extracted with EtOAc and aq. $Na_2S_2O_3$ solution. The organic layer was dried over anhydrous Na_2SO_4 and after evaporation of the solvent analytically pure FDCA was obtained in 53% yield (6.55 gm).[2,4]

One pot synthesis of FDCA from D-fructose

To a 150 mL flask equipped with stirrer bars, D-fructose (4.5 g, 25 mmol), isopropyl alcohol (50 mL), and hydrochloric acid (12.5m aqueous solution, 0.1 mL) were added. The reaction flask was heated on an oil bath to 120 ^{o}C with stirring. The reaction was stopped after 4 h and the mixture was filtrated to remove insoluble humin by-product. Solvent in the reaction mixture was then evaporated and dried to give the crude HMF product (3.15 gm). Afterwards, I_2 (5 mmol), aq. TBHP (200 mmol, 8 equiv.), NaOH (12.5 mmol) and H_2O (50 mL) were added and the reaction mixture was stirred at 70 ^{o}C for 36h. Afterwards, the reaction mixture was neutralized by aq. HCl and extracted with EtOAc and aq. $Na_2S_2O_3$ solution. The organic layer was dried over anhydrous Na_2SO_4 and after evaporation of the solvent FDCA was obtained in 41% yield (1.61 gm).

Gram scale synthesis of Terephthalic acid

A 100 ml of round bottom flask was charged with a magnetic bead, 14.48 mmol (2 gm) of 1, 4-benzenedimethanol, 2.89 mmol (20 mol%) of I_2 and 115.84 mmol (8 equiv.) of aq. TBHP. Afterwards, 7.24 mmol (50 mol%) of NaOH with additional 10 mL of H_2O were added to the reaction mixture and it was heated at 70 ^{o}C for 36 h. Afterwards, the reaction mixture was neutralized by aq. HCl and extracted with EtOAc and aq. $Na_2S_2O_3$ solution. The organic layer was dried over anhydrous Na_2SO_4 and after evaporation of the solvent analytically pure terephthalic was obtained.[3]

References:

[1] L. Lai, Y. Zhang, *ChemSusChem* **2011**, *4*, 1745-1748.

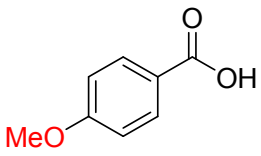
[2] M. S. Ahmed, D.S. mannel, T. W. Root, S. S. Stahl, Org. Process Res. Dev. **2017**, DOI: 10.1021/acs.oprd.7b00223.

[3] A. Sarbajna, I. Dutta, P. Daw, S. Dinda, S. M. W. Rahaman, A. Sarkar, J. K. Bera, *ACS Catal.* **2017**, *7*, 2786-2790.

[4] N. Jiang, B. You, R. Boonstra, I. M. T. Rodriguez, Y. Sun, *ACS Energy Lett.* **2016**, 1, 386-390.

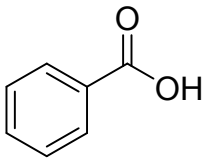
Identification of products:

Compound 1



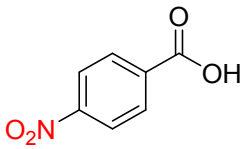
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): 11.62 (br, 1H), 8.05-8.08 (d, J= 9Hz, 2H), 6.93-6.96 (d, J= 9Hz, 2H), 3.88 (s, 3H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): 171.51, 164.06, 132.36, 121.61, 113.76, 55.48

Compound 2



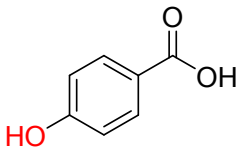
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): 11.13 (br, 1H), 8.16 (m, J= 9Hz, 2H), 7.55 (t, 1H), 7.50 (t, 2H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): 172.33, 133.82, 130.24, 129.35, 128.50.

Compound 3



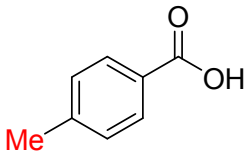
^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 13.62 (br, 1H), 8.30-8.32 (d, J= 6Hz, 2H), 8.16-8.19 (d, 2H); ^{13}C-NMR (DMSO-d_6, 75 MHz, ppm): 166.16, 150.36, 136.81, 131.04, 123.79.

Compound 4



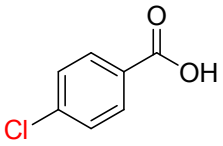
^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 12.43 (br, 1H), 10.24(s, 1H), 7.82-7.85 (d, J= 9Hz, 2H), 6.84-6.87 (d, J= 9Hz, 2H); ^{13}C-NMR (DMSO-d_6, 75 MHz, ppm): 167.65, 162.06, 132.00, 121.82, 115.57.

Compound 5



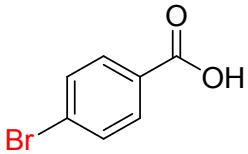
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): 11.57 (br, 1H), 7.91 (d, J= 9Hz, 2H), 7.17-7.20 (d, J= 9Hz, 2H), 2.34 (s, 3H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): 172.55, 144.67, 130.28, 129.22, 126.62, 21.77.

Compound 6



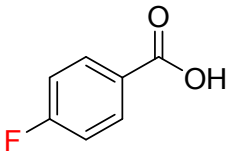
^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 13.20 (br, 1H), 7.85-7.88 (d, J= 9Hz, 2H), 7.68-7.72 (d, J= 12Hz, 2H); ^{13}C-NMR (DMSO-d_6, 75 MHz, ppm): 167.05, 132.14, 131.74, 130.48, 127.32.

Compound 7



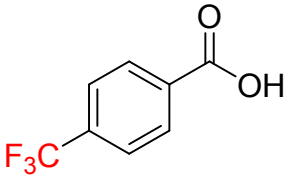
^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 13.19 (br, 1H), 7.84-7.87 (d, J= 9Hz, 2H), 7.67-7.71 (d, J= 12Hz, 2H); ^{13}C-NMR (DMSO-d_6, 75 MHz, ppm): 167.05, 132.14, 131.74, 130.48, 127.32.

Compound 8



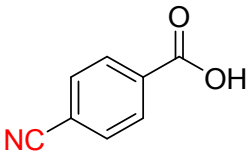
^{1}H-NMR ($CDCl_3$, 300 MHz): 8.13-8.16 (m, 2H), 7.15 (m, 2H); ^{13}C-NMR ($CDCl_3$, 75 MHz): 171.5, 166.7, 133.2, 125.8 (d, J = 2.6 Hz), 116.1; ^{19}F-NMR ($CDCl_3$): -104.1 (s, 1F).

Compound 9



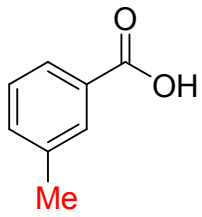
^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 13.46 (br, 1H), 8.12-8.15 (d, J= 9Hz, 2H), 7.86-7.89 (d, J= Hz, 2H); ^{13}C-NMR (DMSO-d_6, 75 MHz, ppm): 167.05, 132.14, 131.74, 130.48, 127.32.

Compound 10



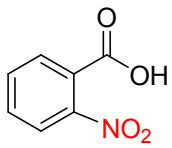
^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 13.42 (br, 1H), 8.11-8.14 (d, J= 9Hz, 2H), 7.83-7.86 (d, J= 9Hz, 2H); ^{13}C-NMR (DMSO-d_6, 75 MHz, ppm): 167.15, 137.24, 132.14, 131.74, 118.48, 115.32.

Compound 11



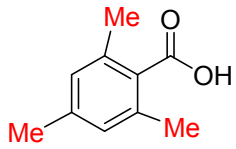
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): 12.76 (br, 1H), 7.80 (s, 1H), 7.17-7.25 (m, 3H), 2.27 (s, 3H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): 172.96, 138.31, 134.65, 130.75, 129.33, 127.44, 21.25.

Compound 12



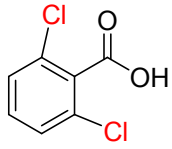
^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 13.87 (br, 1H), 7.73-8.30 (m, 4H); ^{13}C-NMR (DMSO-d_6, 75 MHz, ppm): 166.16, 150.36, 136.81, 131.04, 123.79.

Compound 13



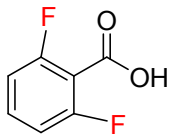
^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 13.06 (br, 1H), 6.90 (s, 2H), 1.98-2.34 (m, 9H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): 171.28, 138.43, 134.33, 128.70, 21.45, 19.50.

Compound 14



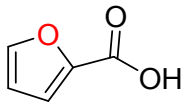
^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 13.82 (br, 1H), 7.64(t, 1H), 7.17-7.25 (m, 2H); ^{13}C-NMR (DMSO-d_6, 75 MHz, ppm): 162.56, 161.40, 158.06, 133.54, 112.76.

Compound 15



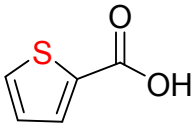
^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 13.92 (br, 1H), 7.63(t, 1H), 7.17-7.23 (m, 2H); ^{13}C-NMR (DMSO-d_6, 75 MHz, ppm): 162.66, 161.30, 158.06, 133.44, 112.86.

Compound 16



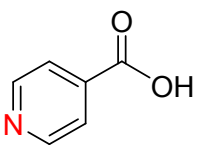
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): 11.95 (br, 1H), 7.57 (s, 1H), 7.27 (s, 1H), 6.48 (s, 1H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): 163.87, 147.49, 143.80, 120.23, 112.30.

Compound 17



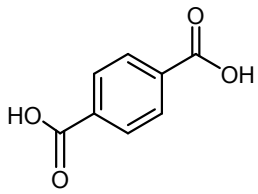
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): 12.16 (br, 1H), 7.82 (d, 1H), 7.56 (d, 1H), 7.06 (s, 1H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): 168.12, 135.11, 134.13, 132.89, 128.12.

Compound 18



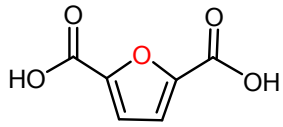
^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 13.68 (br, 1H), 8.76-8.68 (d, J= 9Hz, 2H), 7.80-7.82 (d, 2H); ^{13}C-NMR (DMSO-d_6, 75 MHz, ppm): 166.60, 151.30, 138.54, 123.20.

Compound 19



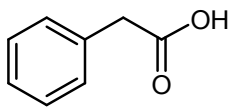
^{1}H-NMR (DMSO–d_6, 300 MHz, ppm): 8.04 (s, 4H), 13.30 (br, s, 2H); ^{13}C-NMR (DMSO–d_6, 75 MHz, ppm): 129.91, 134.90, 167.12.

Compound 20



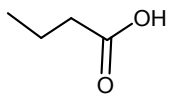
^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 7.30 (s, 2H), ^{13}C-NMR (DMSO-d_6, 75 MHz, ppm): 159.40, 147.53, 118.77.

Compound 21



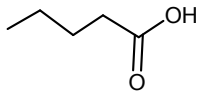
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): 7.38-7.27 (m, 5H), 3.66 (s, 2H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): 178.01, 133.20, 129.30, 128.6, 127.32, 41.05.

Compound 22



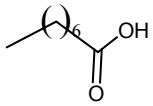
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): 11.70 (s, 1H), 2.35 (t, J = 7.4 Hz, 2H), 1.68 (m, 2H), 1.00 (t, J = 7.5 Hz, 3H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): 180.00, 36.00, 18.72, 13.35.

Compound 23



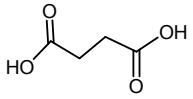
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): 11.73 (s, 1H), 2.35 (t, J = 7.4 Hz, 2H), 1.68 (m, 2H), 1.00 (m, J = 7.5 Hz, 2H), 0.98 (t, 3H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): 180.05, 26.00, 23.78, 18.75, 13.15

Compound 24



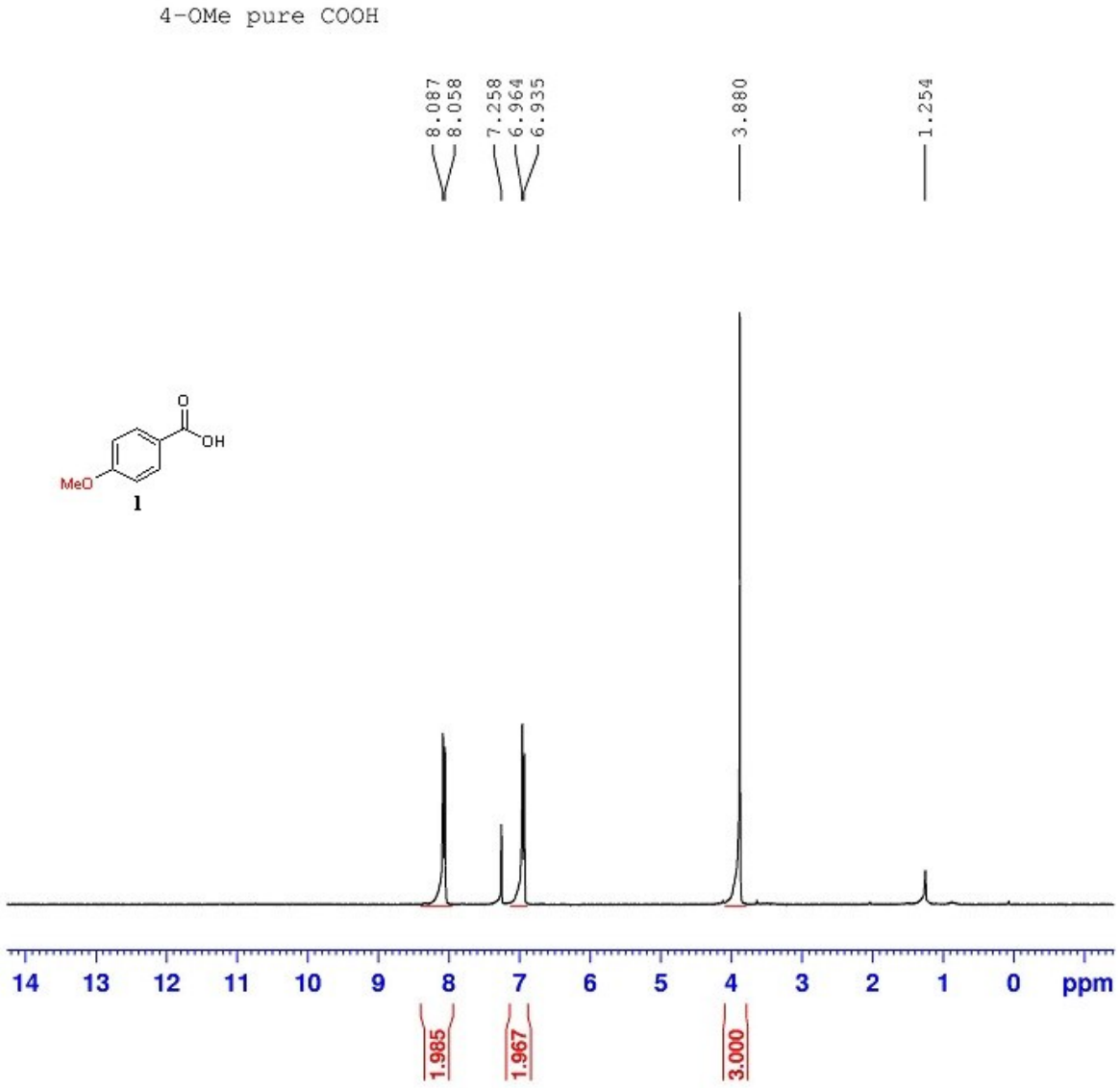
^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): 11.80 (br, s, 1H), 2.34 (t, J = 7.7 Hz, 2H), 1.61 (m, 2H), 1.29 (m, 8H), 0.88 (m, 3H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): 176.87, 34.3, 31.9, 29.2, 29.1, 25.9, 22.30, 13.45.

Compound 25

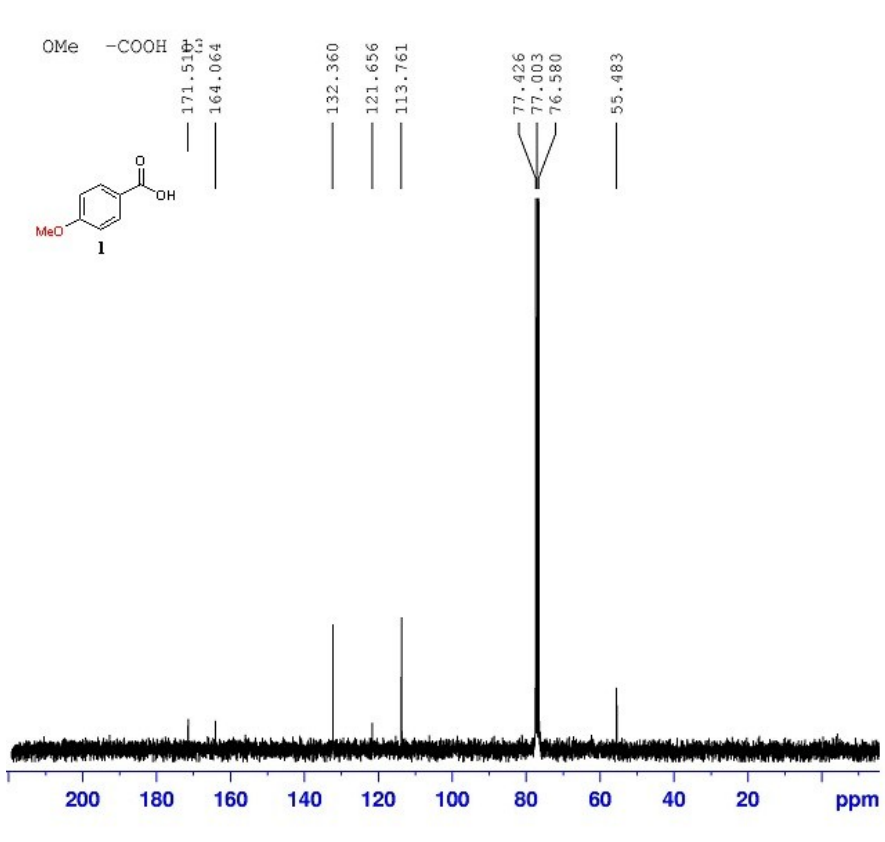


^{1}H-NMR (DMSO-d_6, 300 MHz, ppm): 11.30 (b, 2H), 2.43 (m, 4H); ^{13}C-NMR (DMSO-d_6, 75 MHz, ppm): 174.09, 29.18

^{1}H- NMR (in $CDCl_3$) spectra of compound **1**

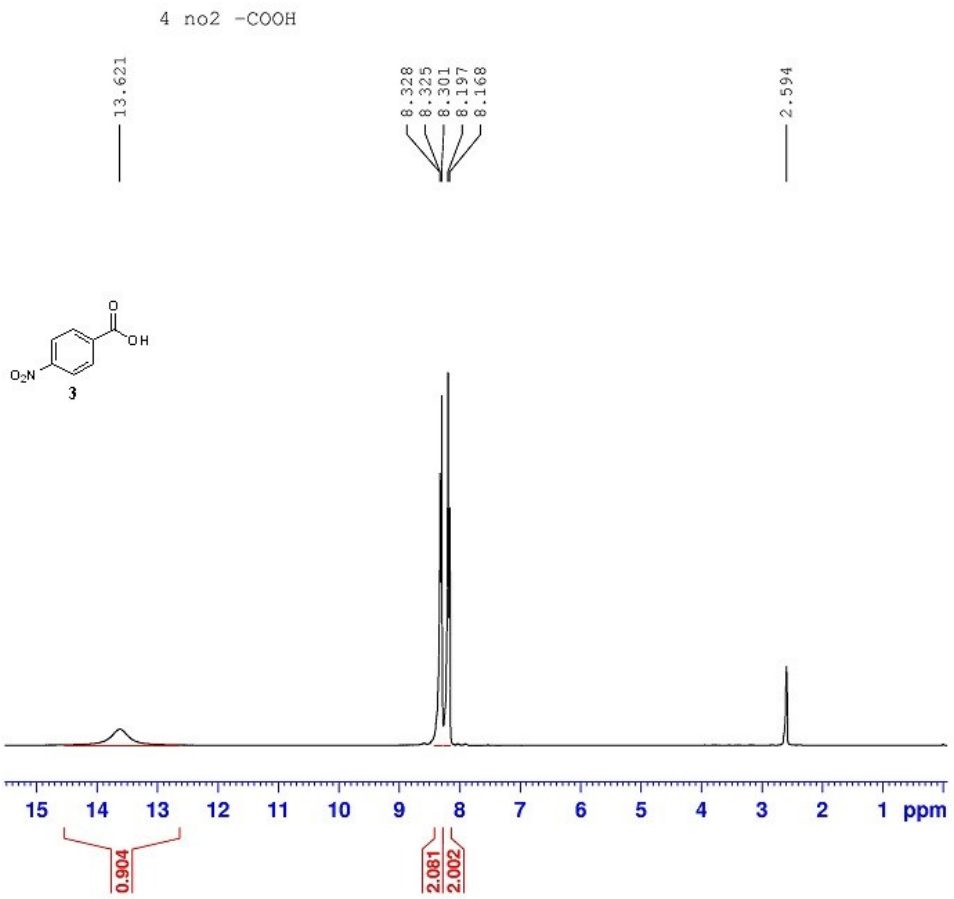


$^{13}C\{H^1\}$- NMR (in $CDCl_3$) spectra of compound **1**



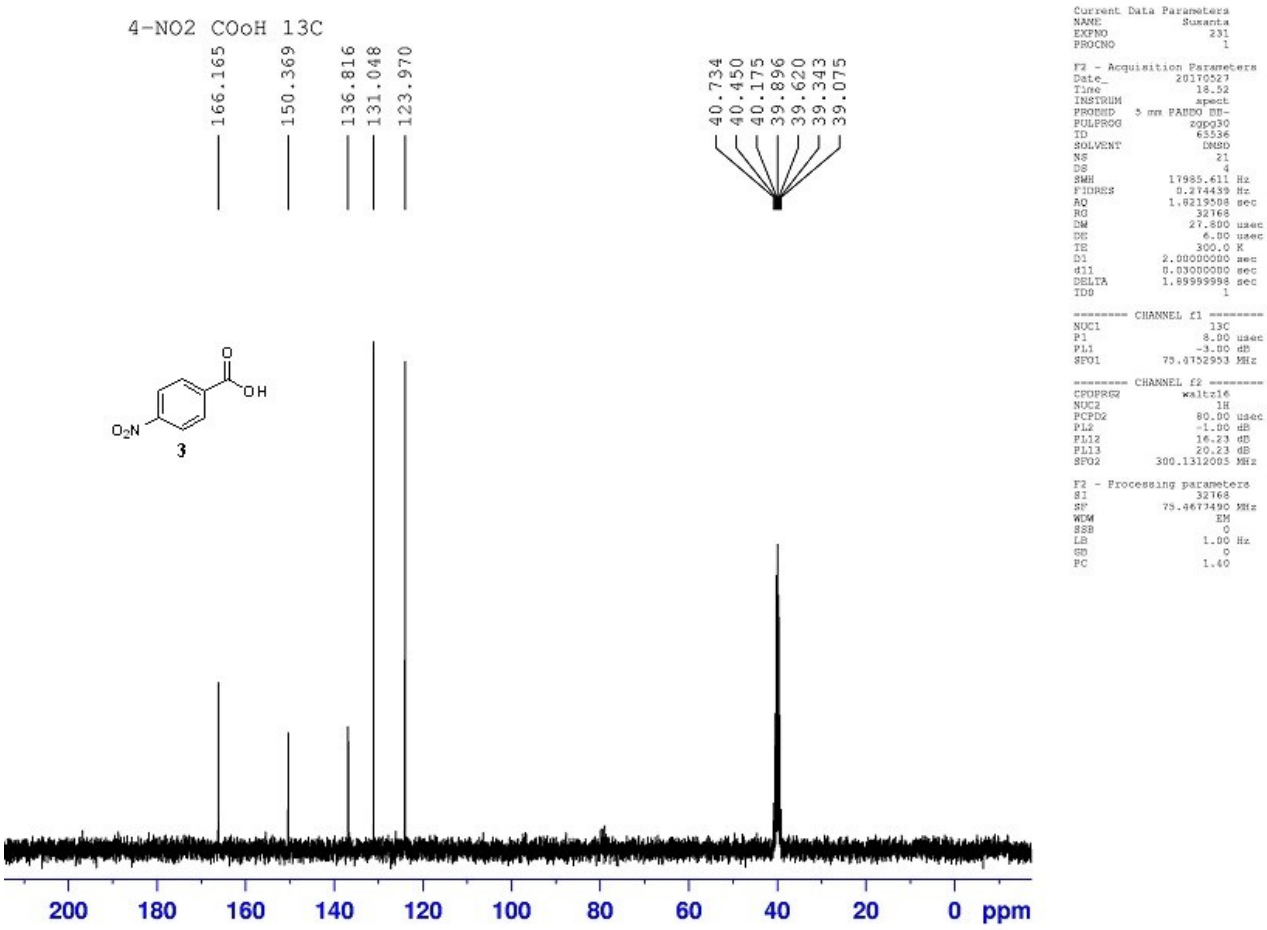

```
NAME                Susanta
EXPNO                   217
PROCNO                    1

F2 - Acquisition Paramet
Date_              20170527
Time                  15.00
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDC13
NS                     478
DS                       4
SWH              17985.611
FIDRES           0.274439
AQ               1.8219508
RG                   32768
DW                  27.800
DE                    6.00
TE                   300.0
D1             2.00000000
d11            0.03000000
DELTA          1.89999998
TD0                       1

======== CHANNEL f1 ====
NUC1                   13C
P1                    8.00
PL1                  -3.00
SFO1           75.4752953

======== CHANNEL f2 ====
CPDPRG2            waltz16
NUC2                   1H
PCPD2                80.00
PL2                  -1.00
PL12                 16.23
PL13                 20.23
SFO2          300.1312005
```

^{1}H- NMR (in DMSO-d_6) spectra of compound **3**

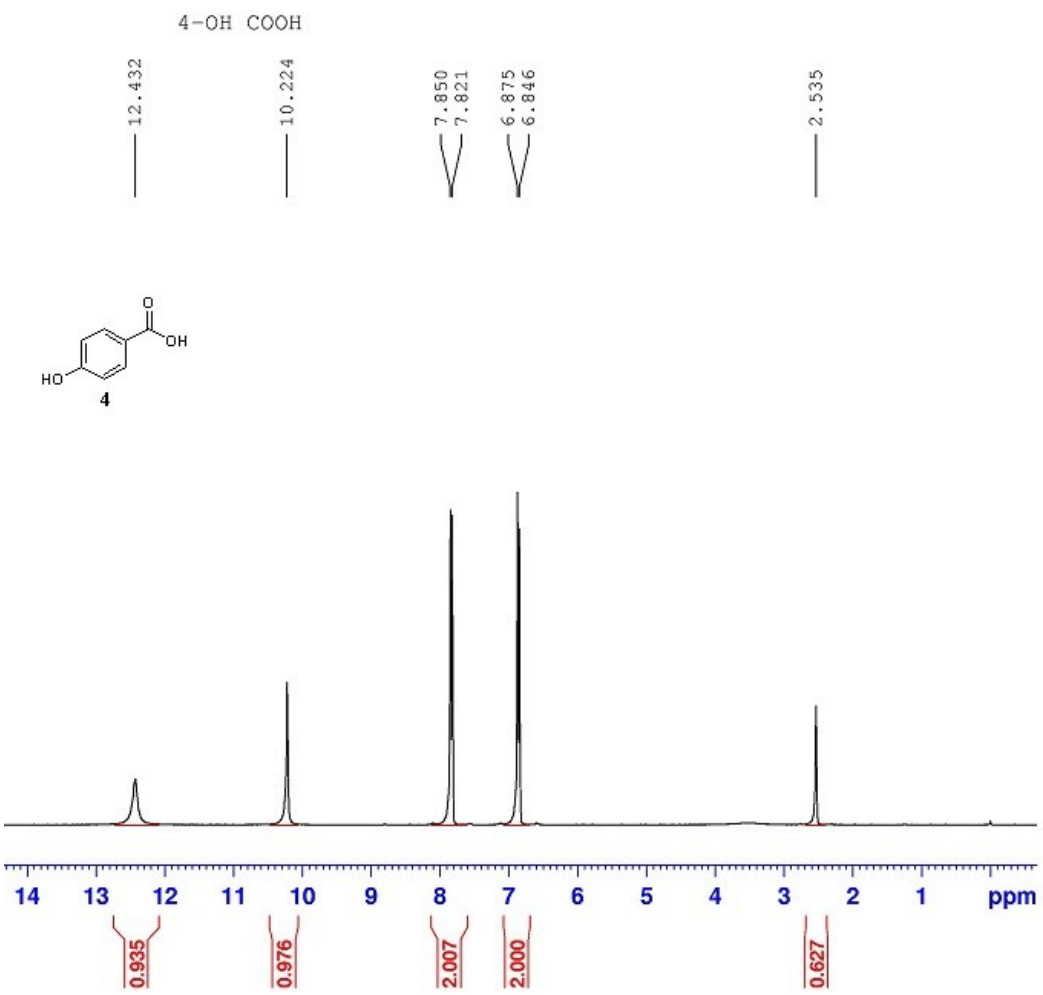



```
Current Data Parameters
NAME                  Susanta
EXPNO                     213
PROCNO                      1

F2 - Acquisition Parameters
Date_              20170527
Time                  14.38
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       10
DS                        2
SWH                6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                     90.5
DW                   81.000 usec
DE                    6.00 usec
TE                   300.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.00 usec
PL1                    -1.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF            300.1299746 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

$^{13}C\{H^1\}$- NMR (in DMSO-d_6) spectra of compound **3**

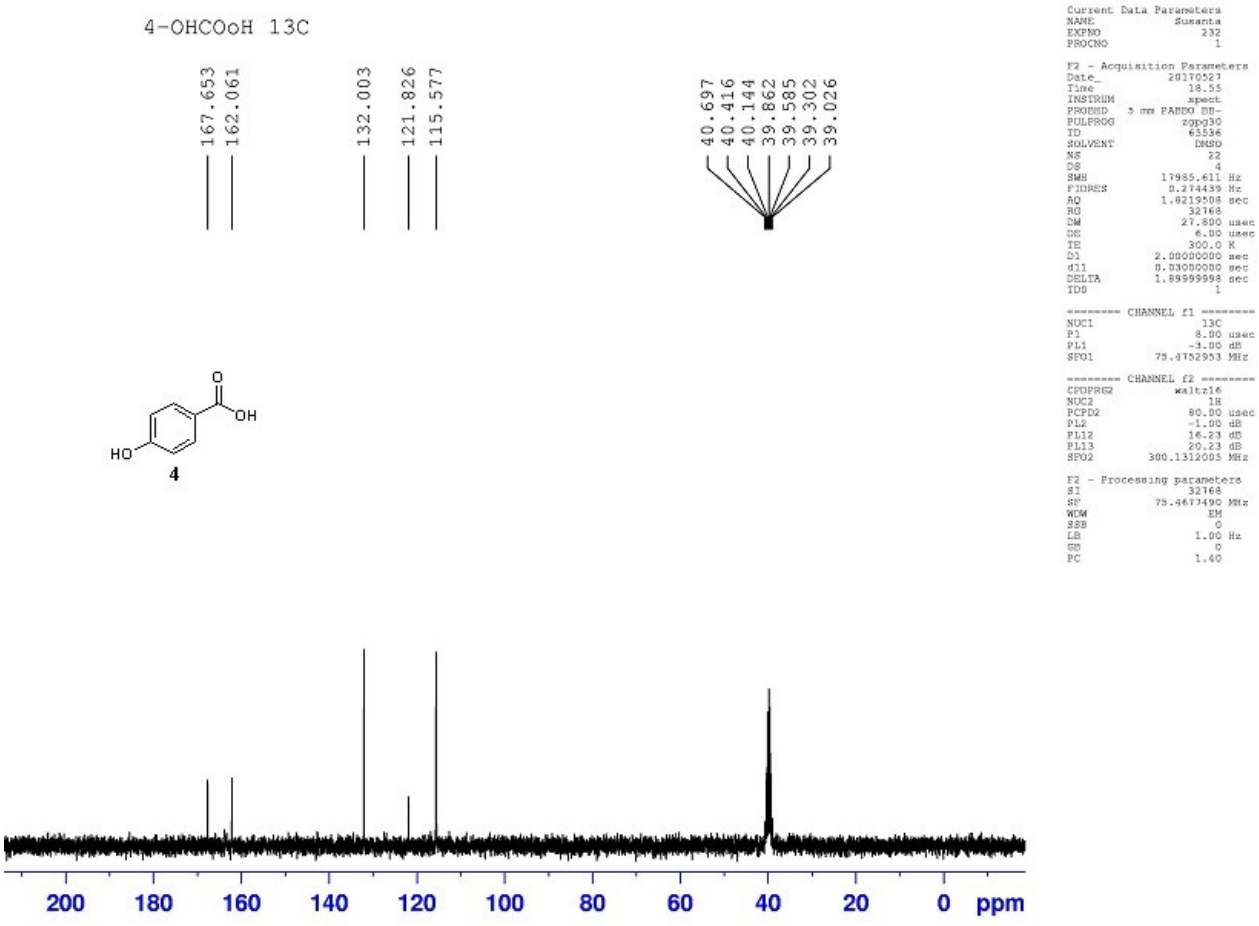


^{1}H- NMR (in DMSO-d_6) spectra of compound **4**



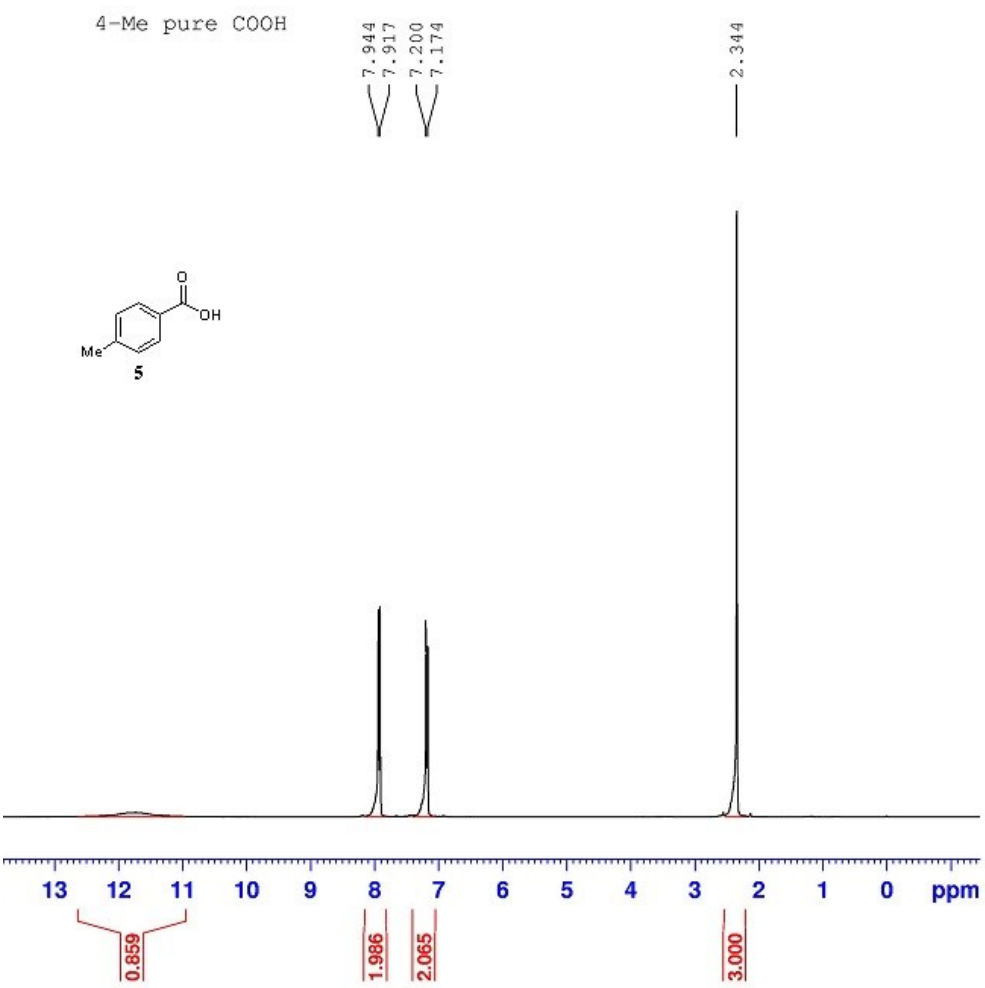

```
Current Data Parameters
NAME                  Susanta
EXPNO                     220
PROCNO                      1

F2 - Acquisition Parameters
Date_              20170527
Time                  17.36
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                        15
DS                         2
SWH                6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                      114
DW                 81.000 usec
DE                  6.00 usec
TE                  300.0 K
D1             1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                 11.00 usec
PL1                -1.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                     32768
SF            300.1299918 MHz
WDW                       EM
SSB                        0
LB                  0.30 Hz
GB                         0
PC                      1.00
```

$^{13}C\{H^1\}$- NMR (in DMSO-d_6) spectra of compound **4**



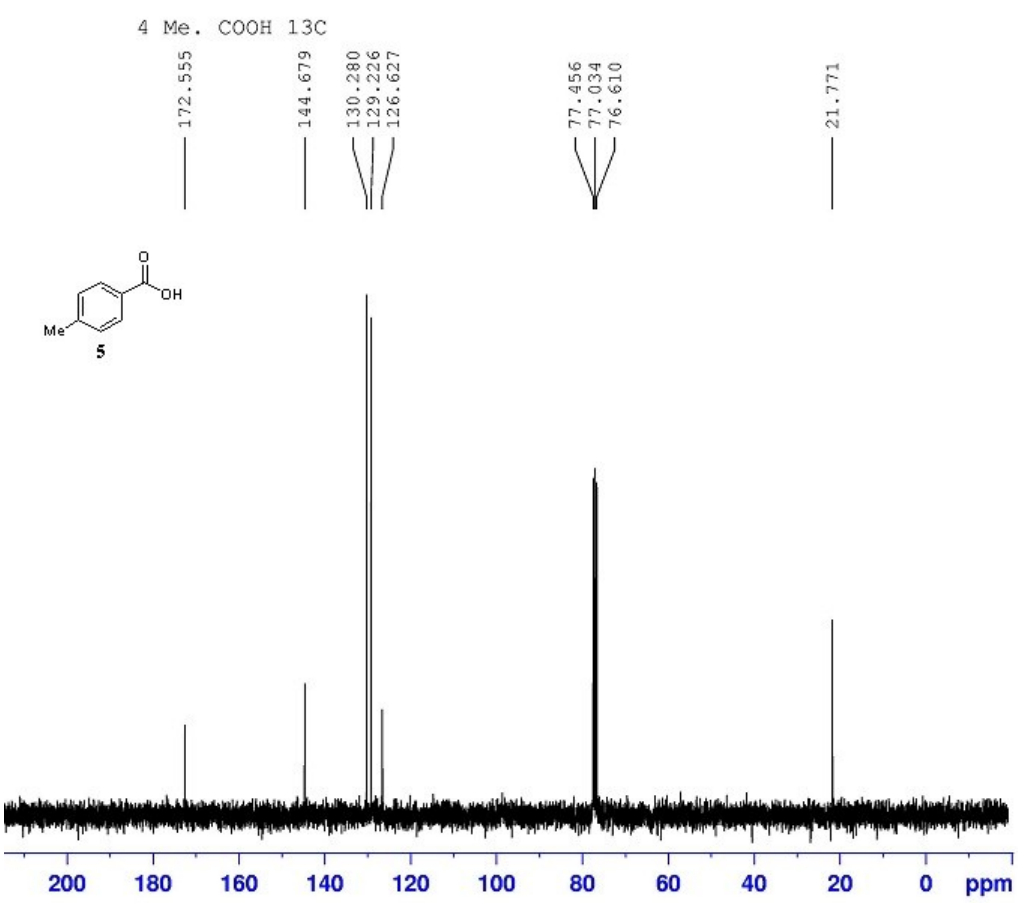

```
Current Data Parameters
NAME                  Susanta
EXPNO                     232
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170523
Time                   18.55
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                      65536
SOLVENT                 DMSO
NS                         22
DS                          4
SWH                17985.611 Hz
FIDRES                0.274439 Hz
AQ                    1.8219508 sec
RG                        32768
DW                       27.800 usec
DE                        6.00 usec
TE                       300.0 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                        8.00 usec
PL1                      -3.00 dB
SFO1                79.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     80.00 usec
PL2                      -1.00 dB
PL12                     16.23 dB
PL13                     20.23 dB
SFO2                300.1312005 MHz

F2 - Processing parameters
SI                         32768
SF                  79.4677490 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```

^{1}H- NMR (in $CDCl_3$) spectra of compound **5**



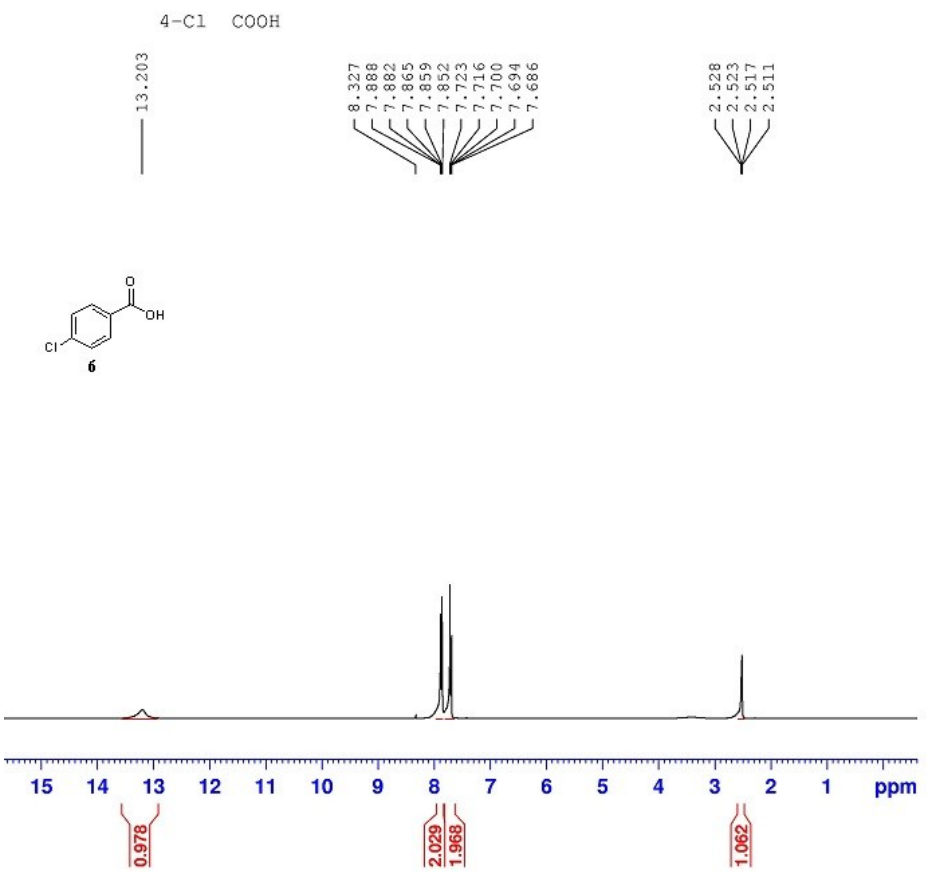

```
Current Data Parameters
NAME                  Susanta
EXPNO                     199
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170526
Time                   16.32
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 6172.839 Hz
FIDRES              0.094190 Hz
AQ                  5.3084660 sec
RG                      143.7
DW                     81.000 usec
DE                      6.00 usec
TE                     300.0 K
D1                  1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                     11.00 usec
PL1                    -1.00 dB
SFO1             300.1318534 MHz

F2 - Processing parameters
SI                      32768
SF               300.1300348 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

$^{13}C\{H^1\}$- NMR (in $CDCl_3$) spectra of compound **5**



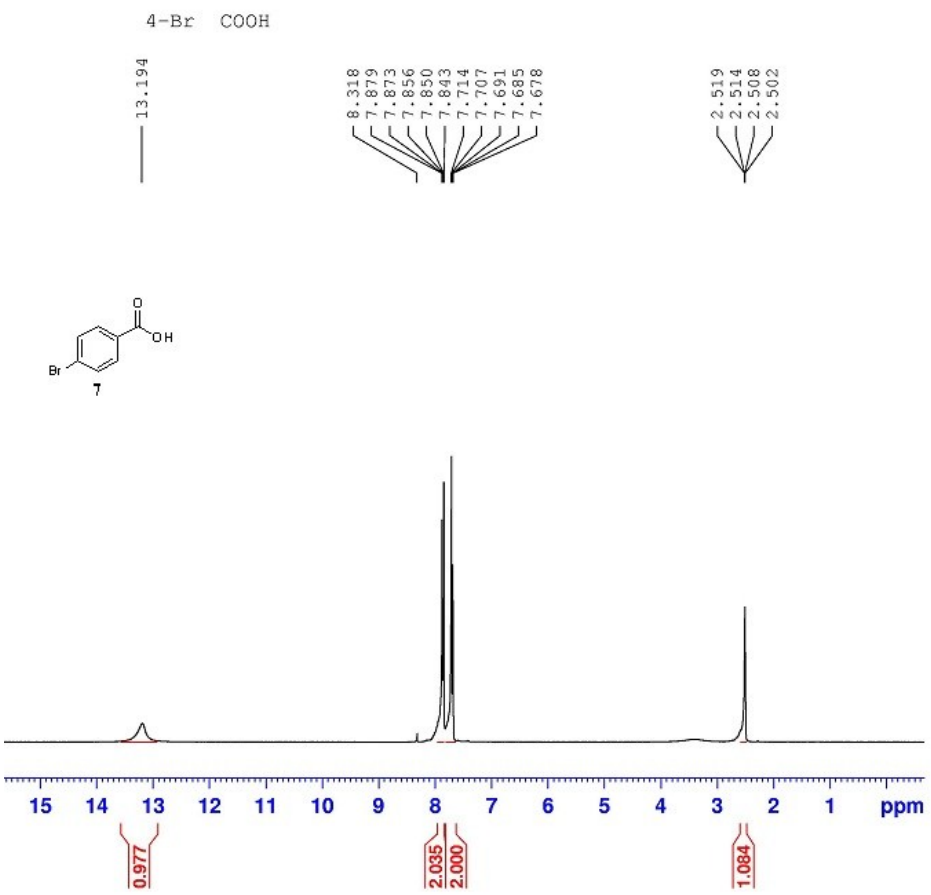

```
Current Data Parameters
NAME                  Susanta
EXPNO                     206
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170527
Time                   12.05
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        52
DS                         4
SWH                17985.611 Hz
FIDRES                0.274439 Hz
AQ                 1.8219508 sec
RG                     32168
DW                    27.800 usec
DE                     6.00 usec
TE                     300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                       8.00 usec
PL1                     -3.00 dB
SFO1              75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                     -1.00 dB
PL12                    16.23 dB
PL13                    20.23 dB
SFO2             300.1312005 MHz

F2 - Processing parameters
SI                        32768
SF                75.4677490 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

^{1}H- NMR (in DMSO-d_6) spectra of compound **6**



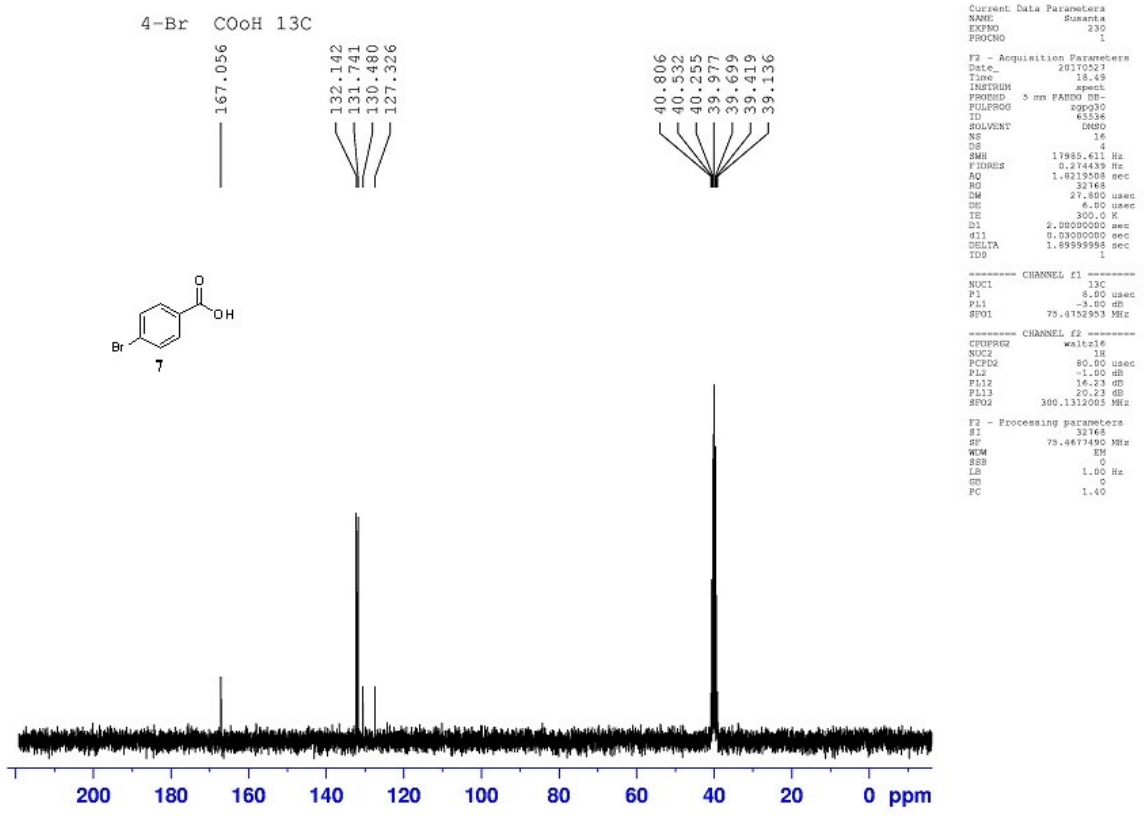

```
Current Data Parameters
NAME                  Susanta
EXPNO                     222
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170527
Time                   17.49
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                        16
DS                         2
SWH                6172.839 Hz
FIDRES             0.094190 Hz
AQ                 5.3084660 sec
RG                     228.1
DW                    81.000 usec
DE                     6.00 usec
TE                    300.0 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.00 usec
PL1                    -1.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF             300.1299964 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

^{1}H- NMR (in DMSO-d_6) spectra of compound **7**




```
Current Data Parameters
NAME                  Susanta
EXPNO                     222
PROCNO                      1

F2 - Acquisition Parameters
Date_              20170527
Time                  17.49
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       16
DS                        2
SWH                6172.839 Hz
FIDRES            0.094190 Hz
AQ                5.3084660 sec
RG                    228.1
DW                   81.000 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    11.00 usec
PL1                   -1.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF            300.1299990 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

$^{13}C\{H^1\}$- NMR (in DMSO-d_6) spectra of compound **7**



Current Data Parameters
NAME Susanta
EXPNO 230
PROCNO 1

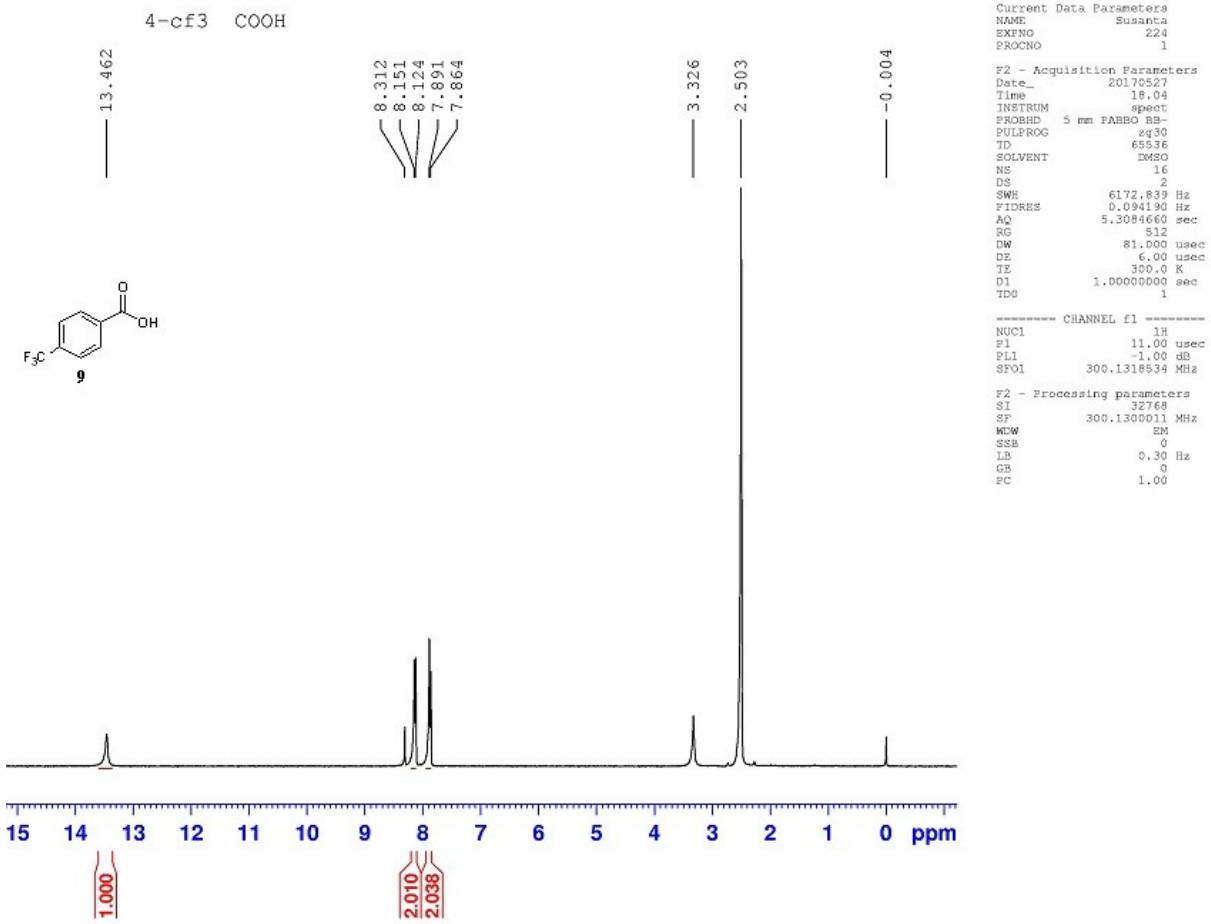
F2 - Acquisition Parameters
Date_ 20170527
Time 18.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 16
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz

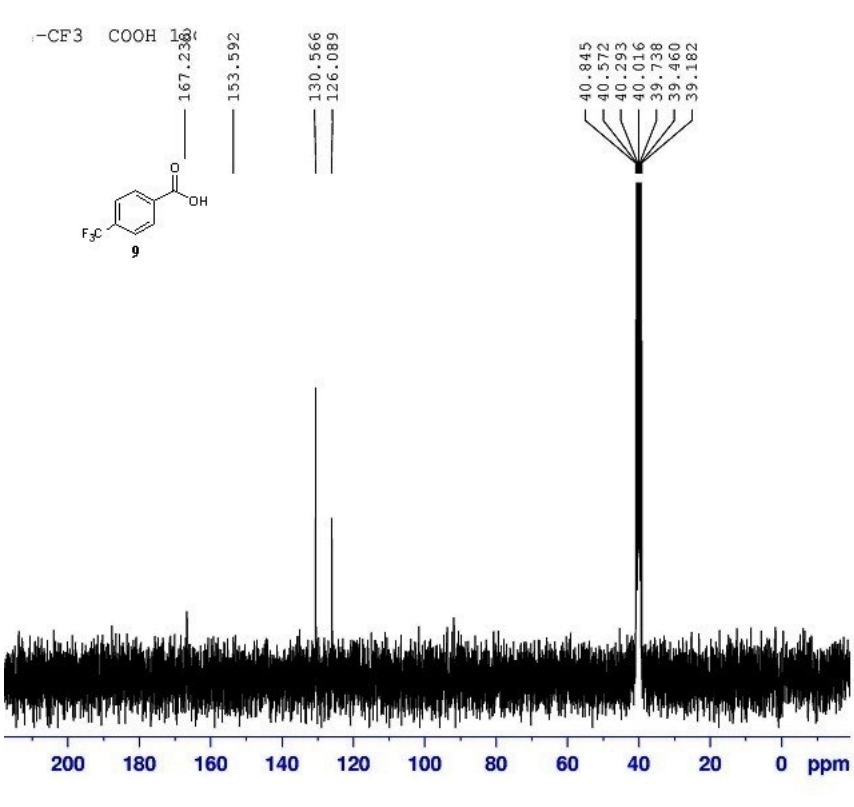
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 16.23 dB
PL13 20.23 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{1}H- NMR (in DMSO-d_6) spectra of compound **9**

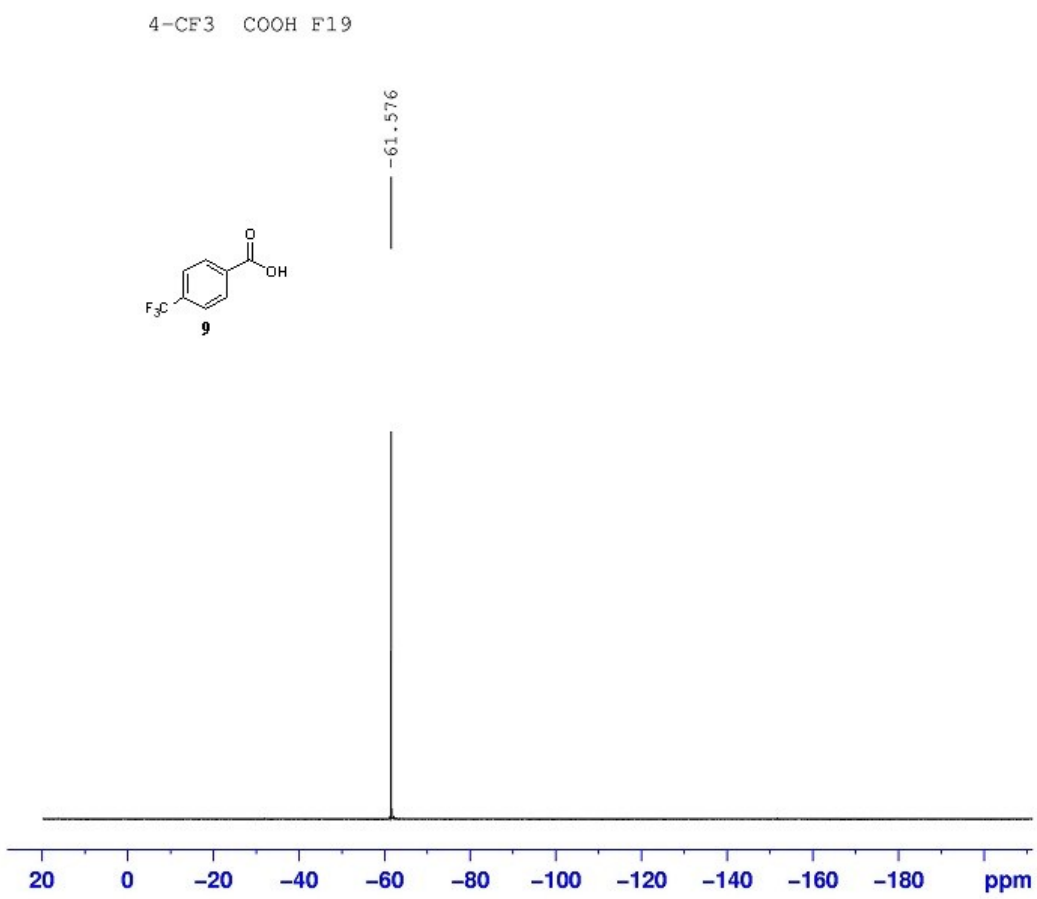


^{13}C{H^{1}}- NMR (in DMSO-d_6) spectra of compound **9**



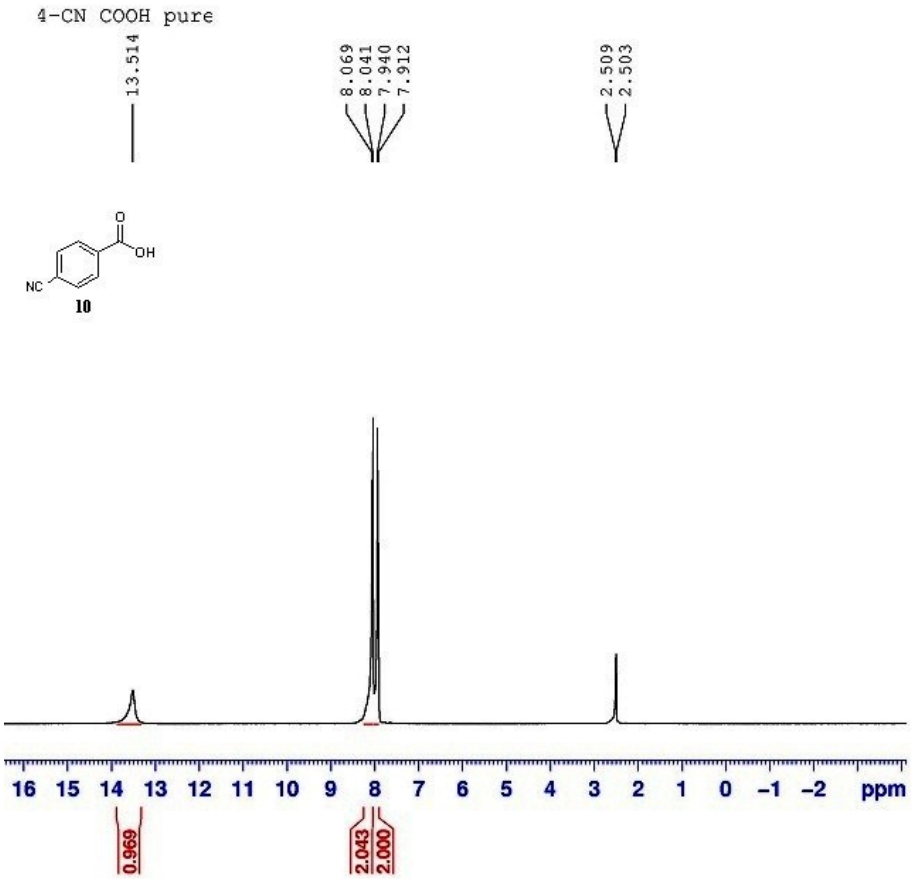

```
NAME                  Sudarica
EXPNO                      229
PROCNO                       1

F2 - Acquisition Paramet
Date_                 20170527
Time                     18.38
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                   DMSO
NS                         140
DS                           4
SWH                  17985.611
FIDRES                0.274439
AQ                    1.8219508
RG                        32768
DW                       27.800
DE                        6.00
TE                       300.0
D1                   2.00000000
d11                  0.03000000
DELTA                1.89999998
TD0                          1

======== CHANNEL f1 ====
NUC1                       13C
P1                        8.00
PL1                      -3.00
SFO1                75.4752953

======== CHANNEL f2 ====
CPDPRG2                waltz16
NUC2                       1H
PCPD2                     80.00
PL2                       -1.00
PL12                      16.23
PL13                      20.23
SFO2                300.1312005
```

^{19}F- NMR (in DMSO-d_6) spectra of compound **9**



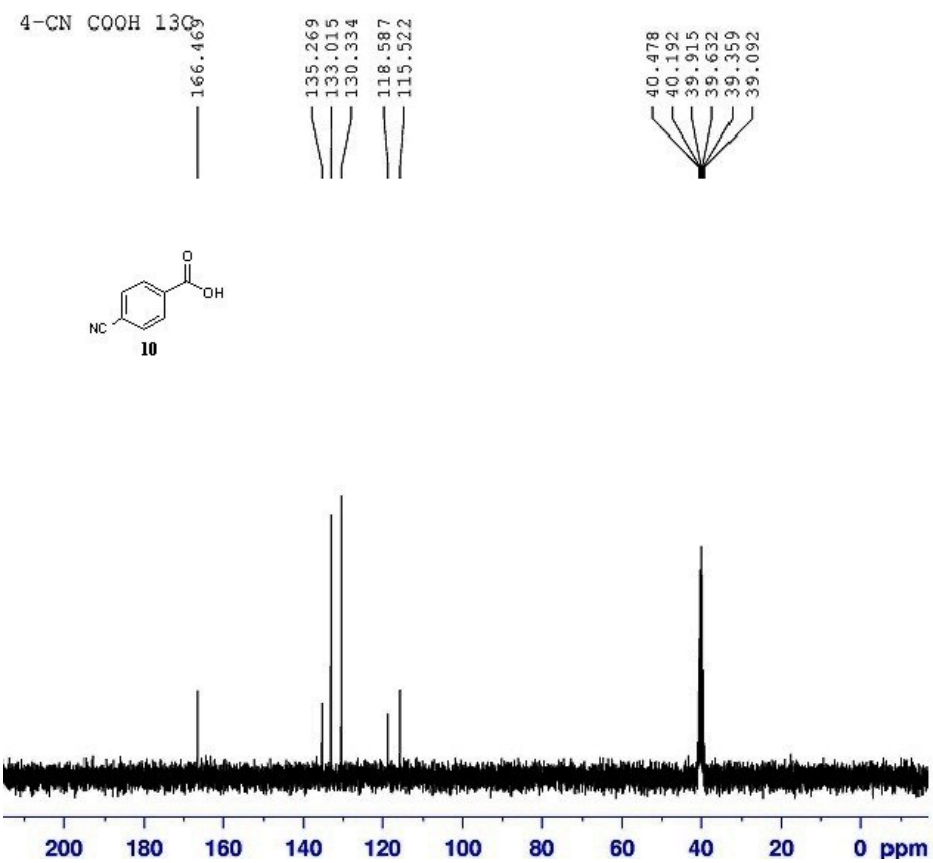

```
Current Data Parameters
NAME                  Susanta
EXPNO                     228
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170523
Time                   18.34
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG             zgfhigqn
TD                    131072
SOLVENT                 DMSO
NS                        16
DS                         4
SWH                 67567.570 Hz
FIDRES              0.515500 Hz
AQ                  0.9699828 sec
RG                       4096
DW                     7.400 usec
DE                     6.00 usec
TE                     300.0 K
D1                1.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      19F
P1                      11.70 usec
PL1                     -1.00 dB
SFO1             282.3761148 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                   80.00 usec
PL2                     -1.00 dB
PL12                    16.23 dB
SFO2             300.1312005 MHz

F2 - Processing parameters
SI                        65536
SF               282.4043550 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```

^{1}H- NMR (in DMSO-d_6) spectra of compound **9**



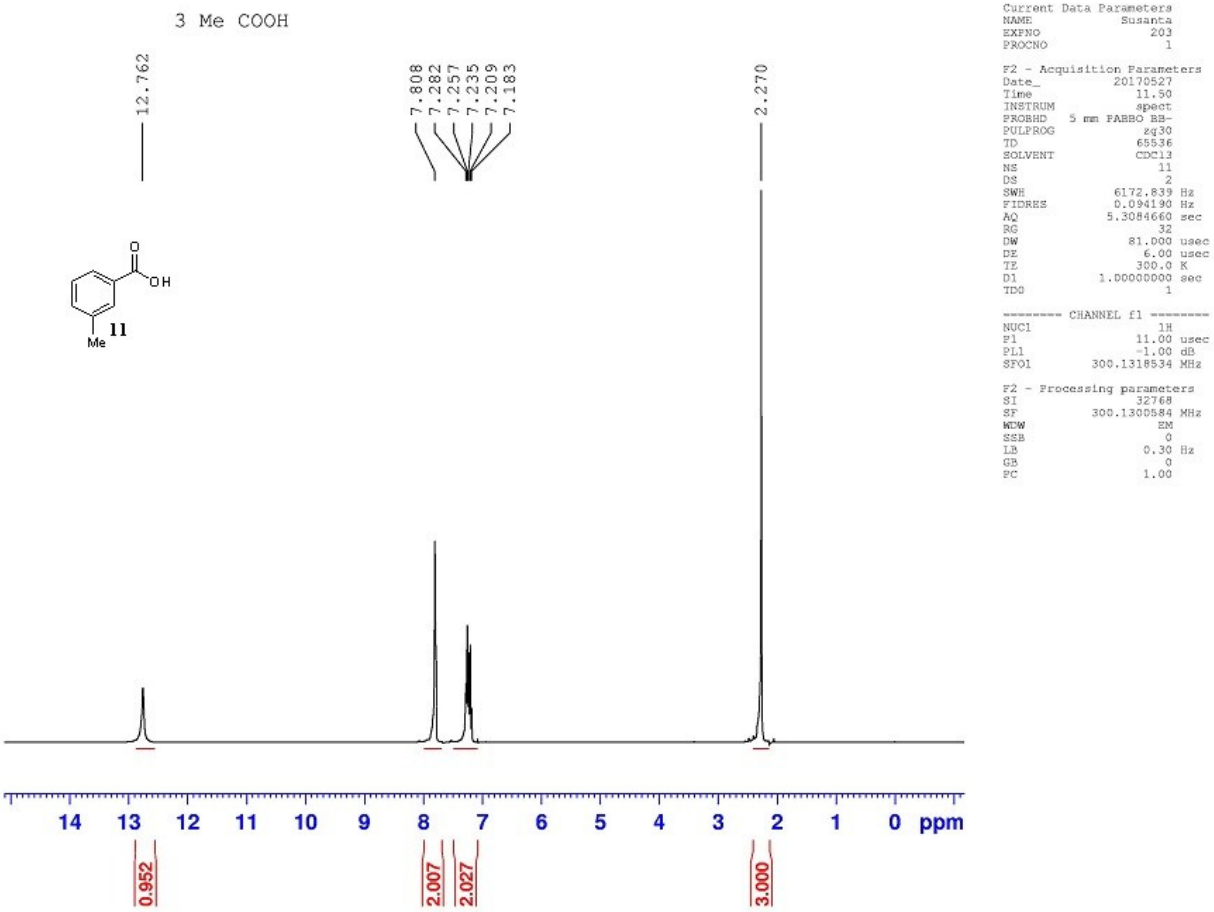

```
Current Data Parameters
NAME                  Susanta
EXPNO                     265
PROCNO                      1

F2 - Acquisition Paramet
Date_                20170805
Time                     9.46
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                 DMSO
NS                        10
DS                         2
SWH                 6172.839
FIDRES              0.094190
AQ                  5.3084660
RG                      101.6
DW                     81.000
DE                       6.00
TE                      300.0
D1                1.00000000
TD0                         1

======== CHANNEL f1 ====
NUC1                       1H
P1                      11.00
PL1                     -1.00
SFO1             300.1318534

F2 - Processing paramete
SI                      32768
SF               300.1300000
WDW                        EM
SSB                         0
LB                       0.30
GB                          0
PC                       1.00
```

$^{13}C\{H^1\}$- NMR (in DMSO-d_6) spectra of compound **10**

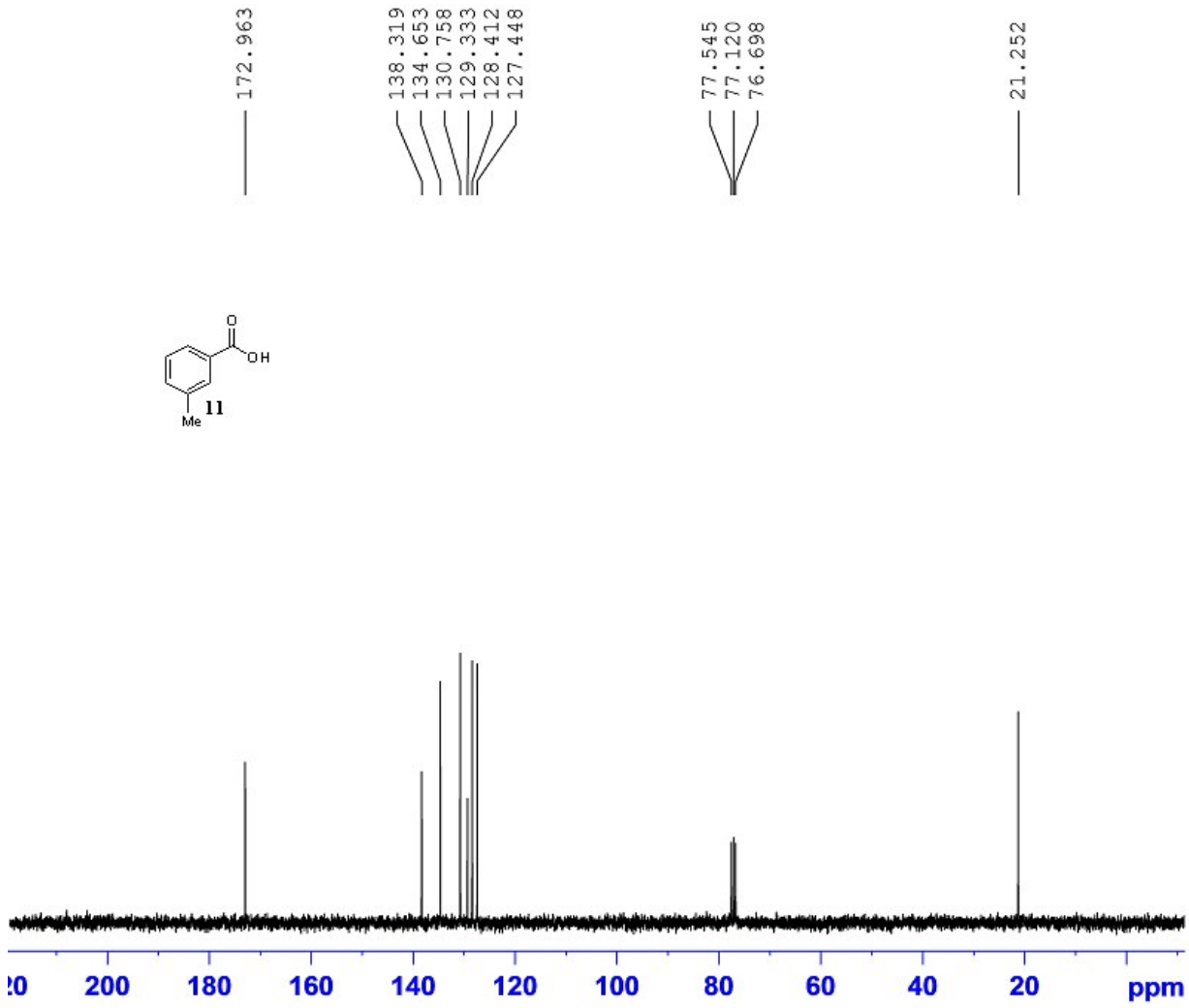


EXPNO	266
PROCNO	1
F2 - Acquisition Paramet	
Date_	20170805
Time	9.50
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	62
DS	4
SWH	17985.611
FIDRES	0.274439
AQ	1.8219508
RG	32768
DW	27.800
DE	6.00
TE	300.0
D1	2.00000000
d11	0.03000000
DELTA	1.89999998
TD0	1
======== CHANNEL f1 ====	
NUC1	13C
P1	8.00
PL1	-3.00
SFO1	75.4752953
======== CHANNEL f2 ====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00
PL2	-1.00
PL12	16.23
PL13	20.23

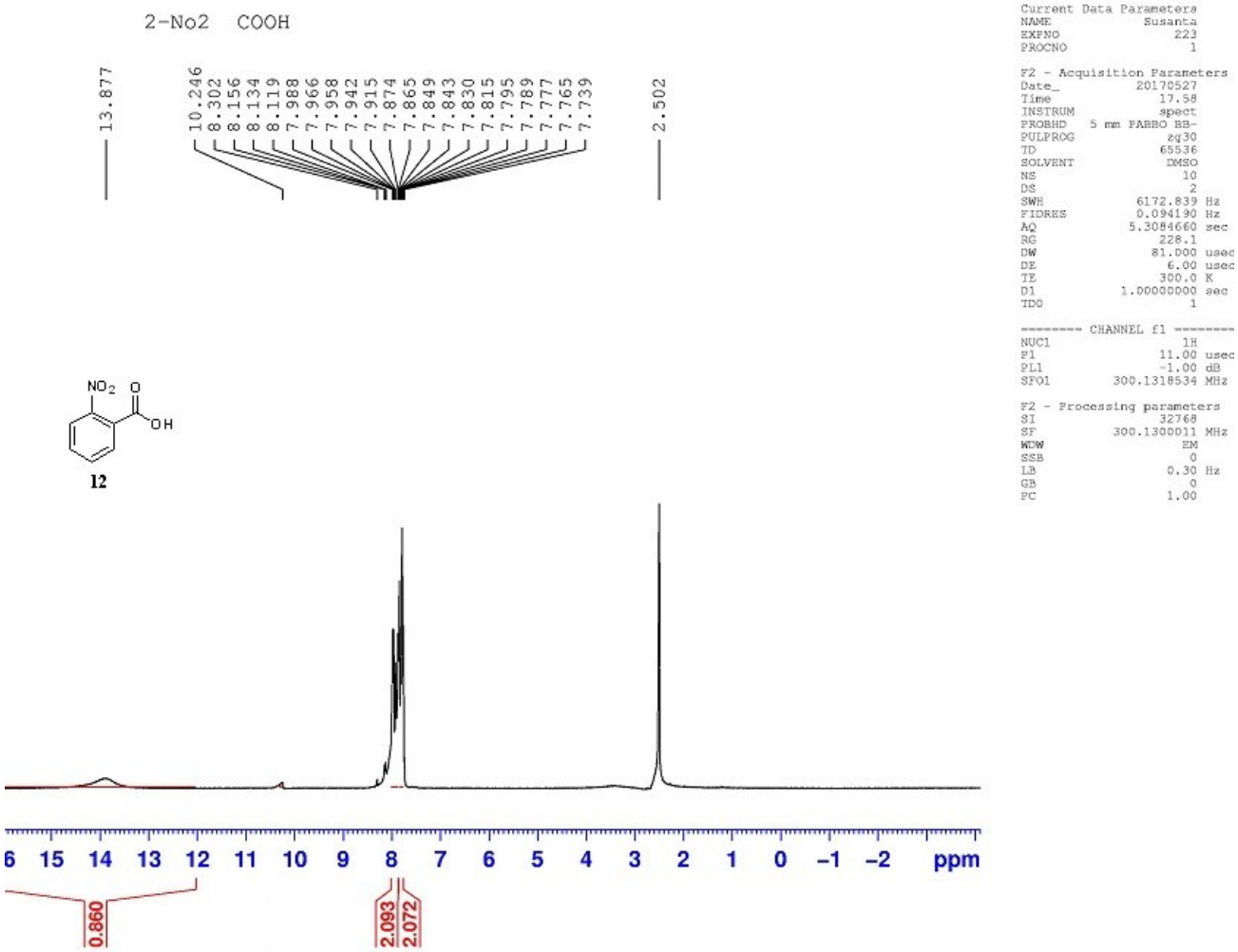
^{1}H- NMR (in $CDCl_3$) spectra of compound **11**



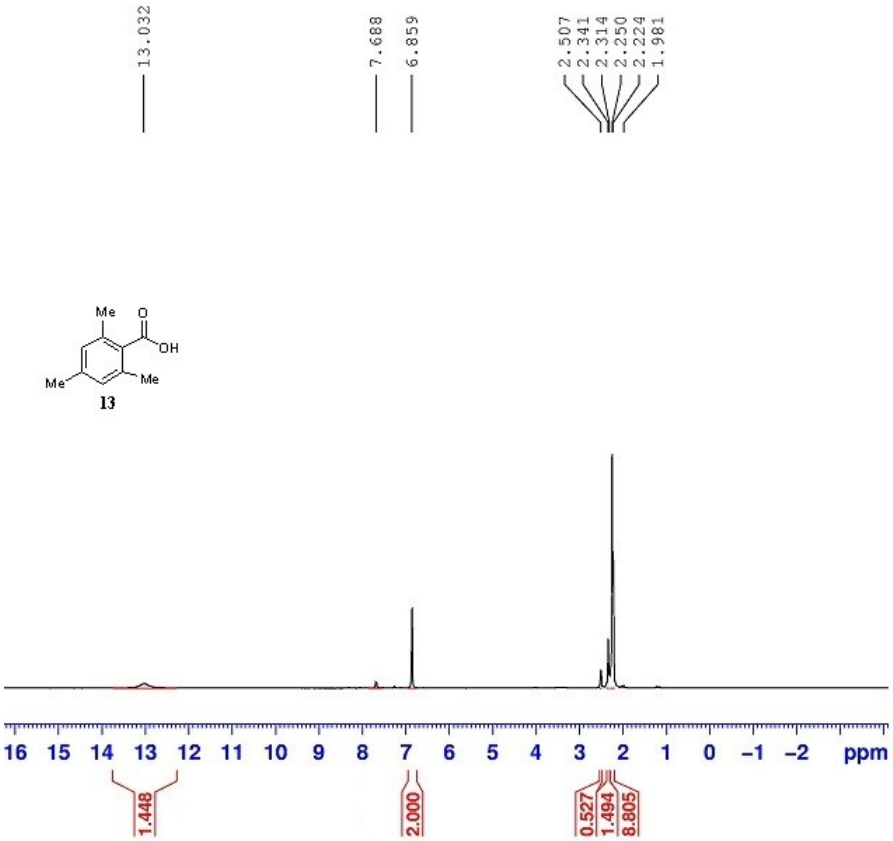
$^{13}C\{H^1\}$- NMR (in $CDCl_3$) spectra of compound **11**



^1H- NMR (in DMSO-d_6) spectra of compound **12**

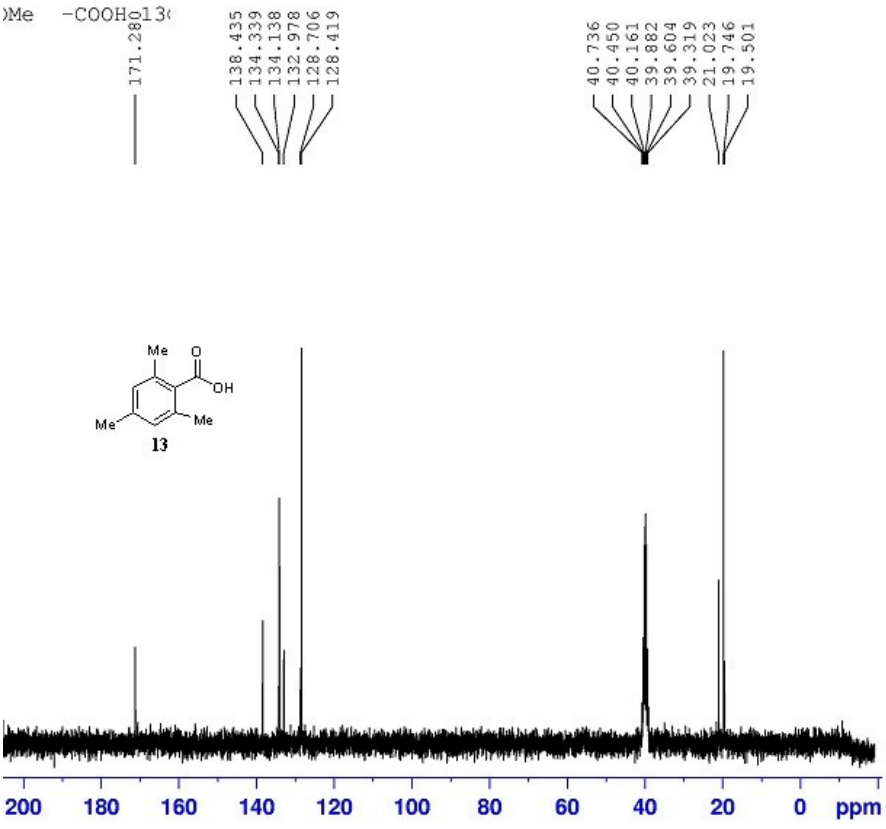


^{1}H- NMR (in DMSO-d_6) spectra of compound **13**



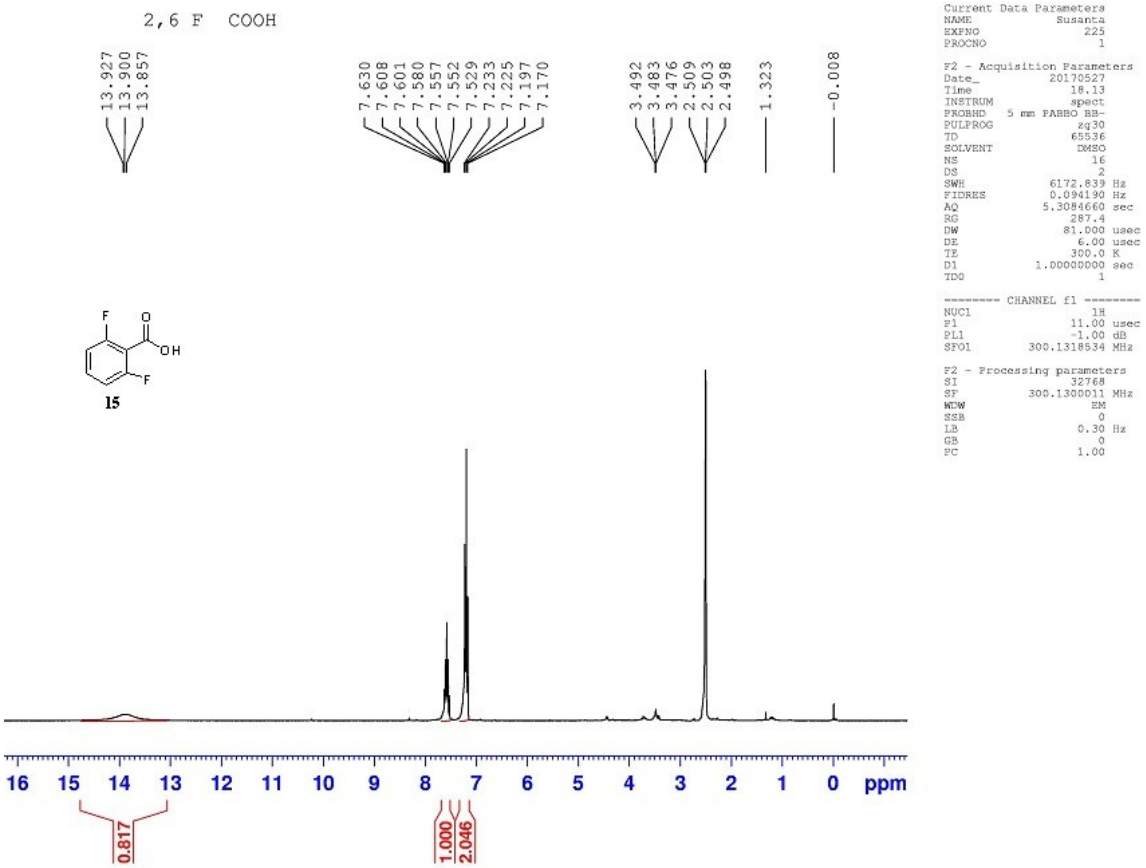

```
F2 - Acquisition Paramet
Date_              20170803
Time                 19.40
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH               6172.839
FIDRES            0.094190
AQ                5.3084660
RG                     50.8
DW                   81.000
DE                     6.00
TE                    300.0
D1              1.00000000
TD0                     1

======== CHANNEL f1 ====
NUC1                   1H
P1                  11.00
PL1                 -1.00
SFO1           300.1318534

F2 - Processing paramete
SI                    32768
SF             300.1300000
WDW                      EM
SSB                       0
LB                     0.30
GB                        0
PC                     1.00
```

$^{13}C\{H^1\}$- NMR (in DMSO-d_6) spectra of compound **13**

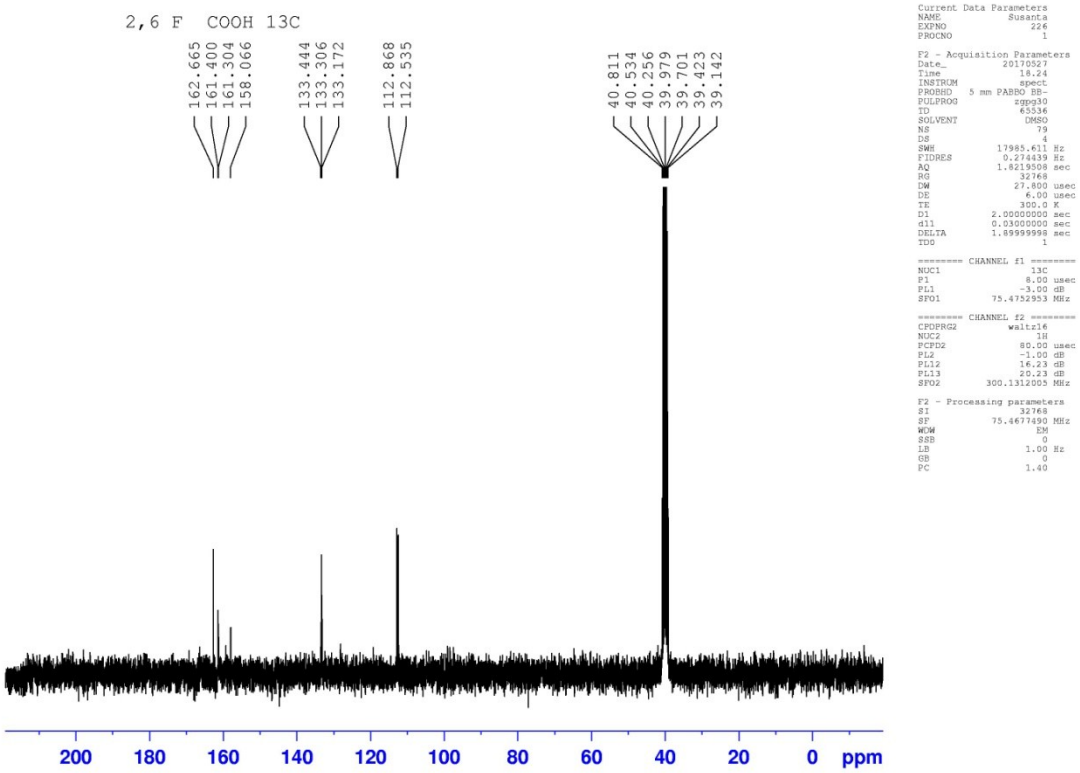


PROCNO	1
F2 - Acquisition Paramet	
Date_	20170803
Time	19.54
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	115
DS	4
SWH	17985.611
FIDRES	0.274439
AQ	1.8219508
RG	32768
DW	27.800
DE	6.00
TE	300.0
D1	2.00000000
d11	0.03000000
DELTA	1.89999998
TD0	1
======== CHANNEL f1 ====	
NUC1	13C
P1	8.00
PL1	-3.00
SFO1	75.4752953
======== CHANNEL f2 ====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00
PL2	-1.00
PL12	16.23
PL13	20.23
SFO2	300.1312005

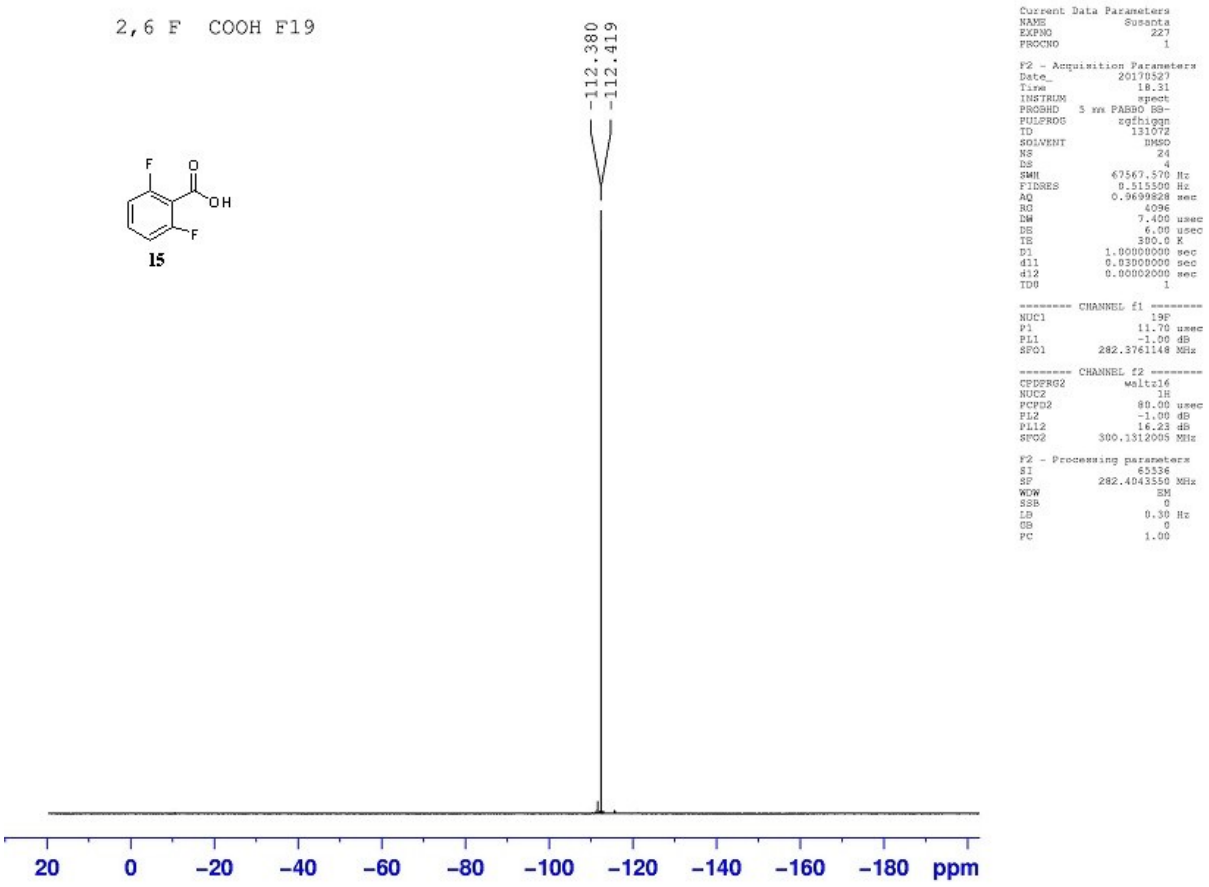
^{1}H- NMR (in DMSO-d_6) spectra of compound **15**



$^{13}C\{H^1\}$- NMR (in DMSO-d_6) spectra of compound **15**

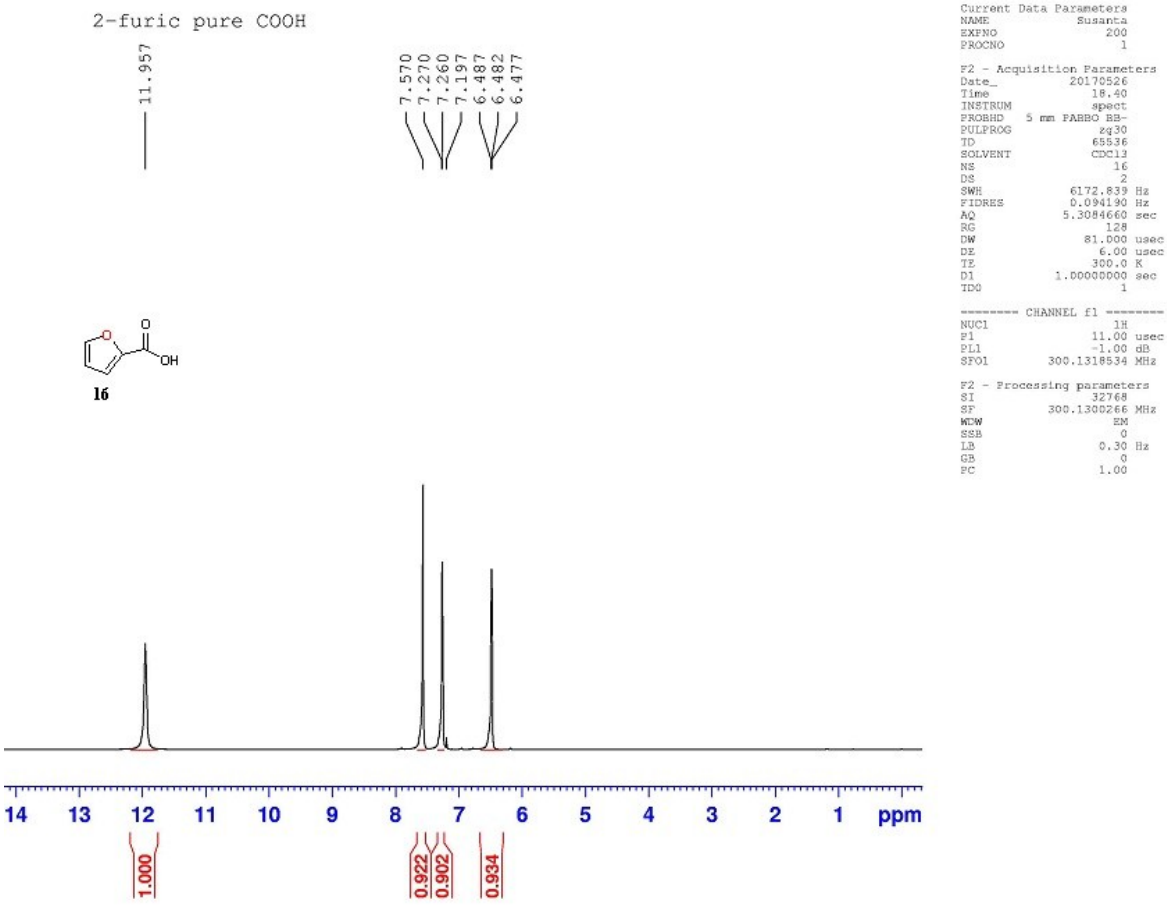


^{19}F- NMR (in DMSO-d_6) spectra of compound **15**



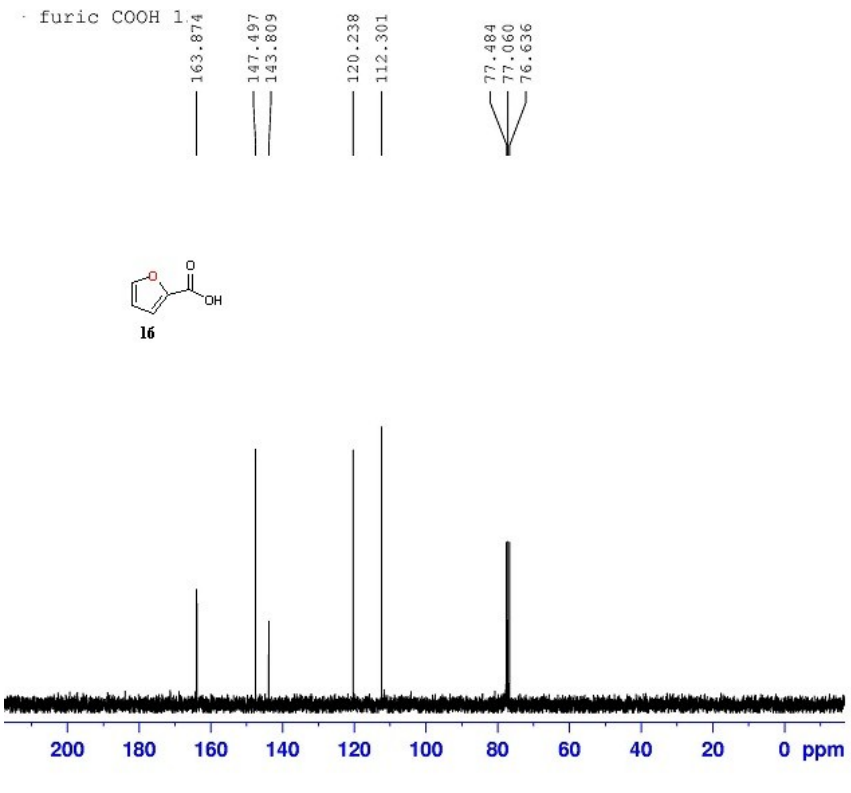

```
Current Data Parameters
NAME                  Susanta
EXPNO                     227
PROCNO                      1

F2 - Acquisition Parameters
Date_              20170527
Time                   18.31
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG          zgfhigqn
TD                     131072
SOLVENT                DMSO
NS                        24
DS                         4
SWH                67567.570 Hz
FIDRES                 0.515300 Hz
AQ                     0.9699828 sec
RG                        4096
DW                        7.400 usec
DE                        6.00 usec
TE                      300.0 K
D1                 1.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      19F
P1                       11.70 usec
PL1                      -1.00 dB
SFO1               282.3761148 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 usec
PL2                      -1.00 dB
PL12                     16.23 dB
SFO2               300.1312005 MHz

F2 - Processing parameters
SI                        65536
SF                 282.4043550 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

^{1}H- NMR (in $CDCl_3$) spectra of compound **16**



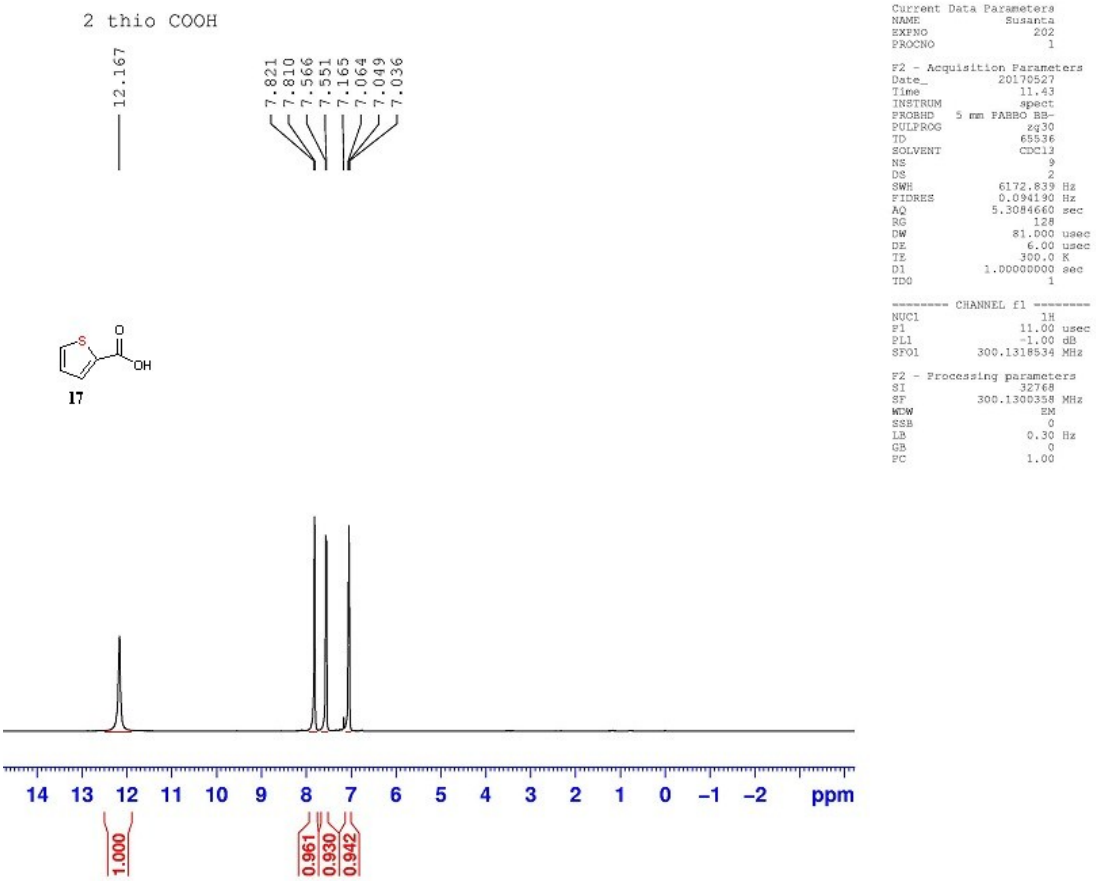

```
Current Data Parameters
NAME                  Susanta
EXPNO                     200
PROCNO                      1

F2 - Acquisition Parameters
Date_                20170526
Time                    18.40
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 6172.839 Hz
FIDRES              0.094190 Hz
AQ                  5.3084660 sec
RG                       128
DW                    81.000 usec
DE                     6.00 usec
TE                    300.0 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.00 usec
PL1                    -1.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                       32768
SF                 300.1300266 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

$^{13}C\{H^1\}$- NMR (in $CDCl_3$) spectra of compound **16**



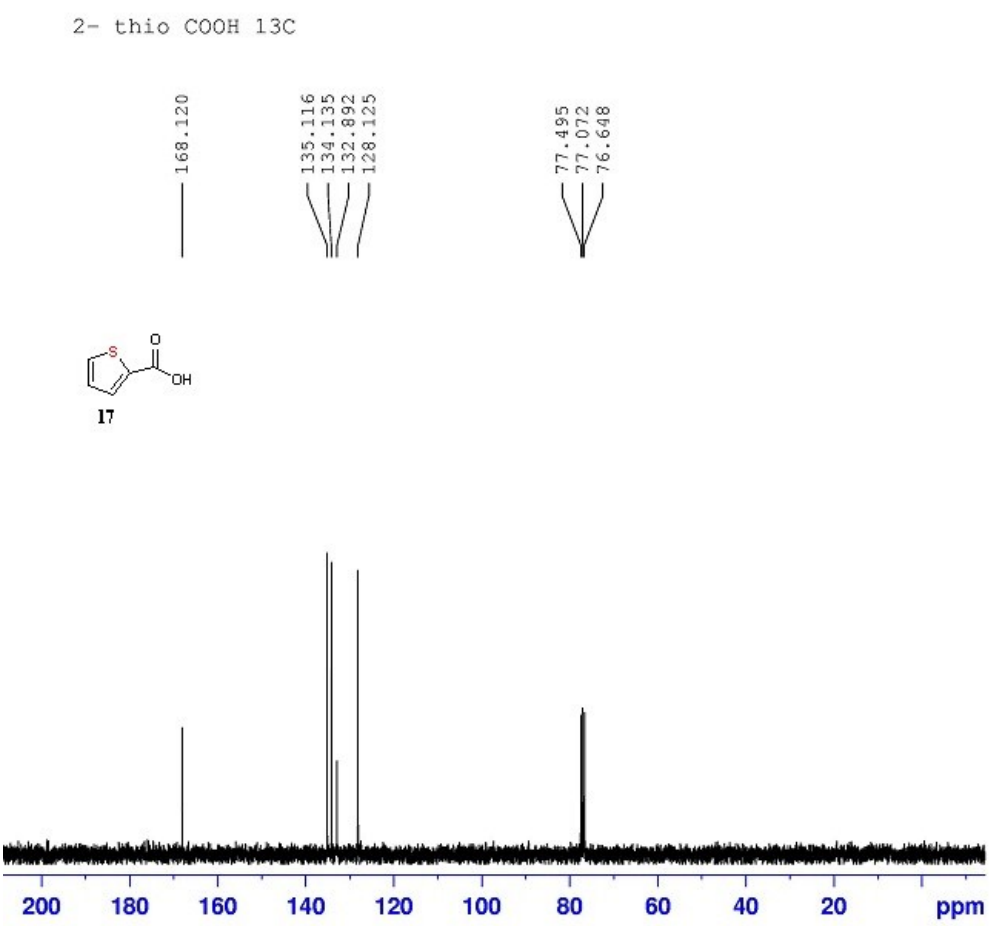

```
NAME              Susanta
EXPNO                 208
PROCNO                  1

F2 - Acquisition Paramet
Date_              20170527
Time                 12.16
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      31
DS                       4
SWH              17985.611
FIDRES           0.274439
AQ               1.8219508
RG                   32768
DW                  27.800
DE                    6.00
TE                   300.0
D1             2.00000000
d11            0.03000000
DELTA          1.89999998
TD0                     1

======== CHANNEL f1 ====
NUC1                  13C
P1                   8.00
PL1                 -3.00
SFO1           75.4752953

======== CHANNEL f2 ====
CPDPRG2           waltz16
NUC2                   1H
PCPD2                80.00
PL2                  -1.00
PL12                 16.23
PL13                 20.23
SFO2          300.1312005
```

^{1}H- NMR (in $CDCl_3$) spectra of compound **17**



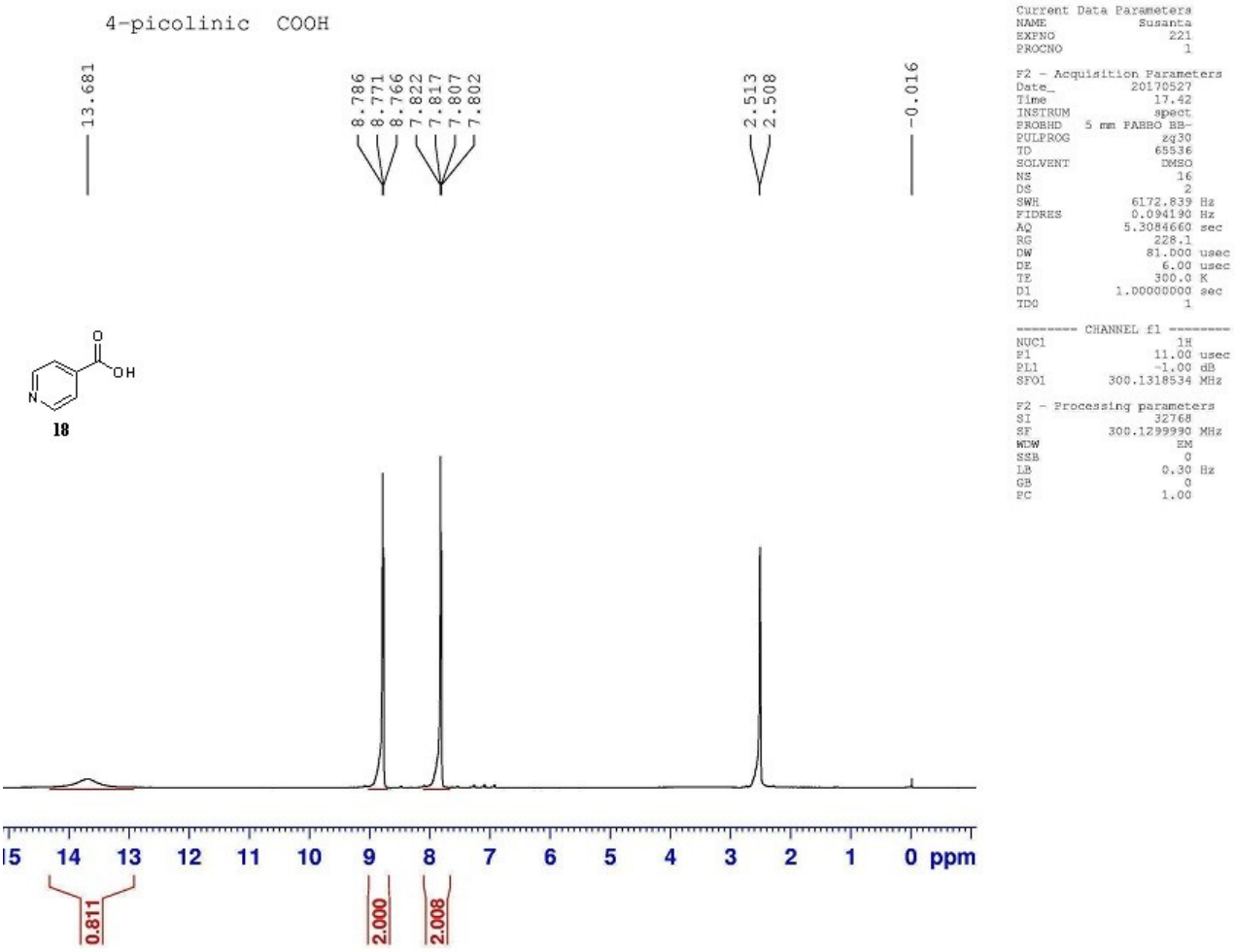

```
Current Data Parameters
NAME                  Susanta
EXPNO                     202
PROCNO                      1

F2 - Acquisition Parameters
Date_              20170527
Time                  11.43
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        9
DS                        2
SWH                6172.839 Hz
FIDRES             0.094190 Hz
AQ                 5.3084660 sec
RG                      128
DW                   81.000 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.00 usec
PL1                    -1.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF             300.1300358 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

^{13}C {H^1}- NMR (in $CDCl_3$) spectra of compound **17**

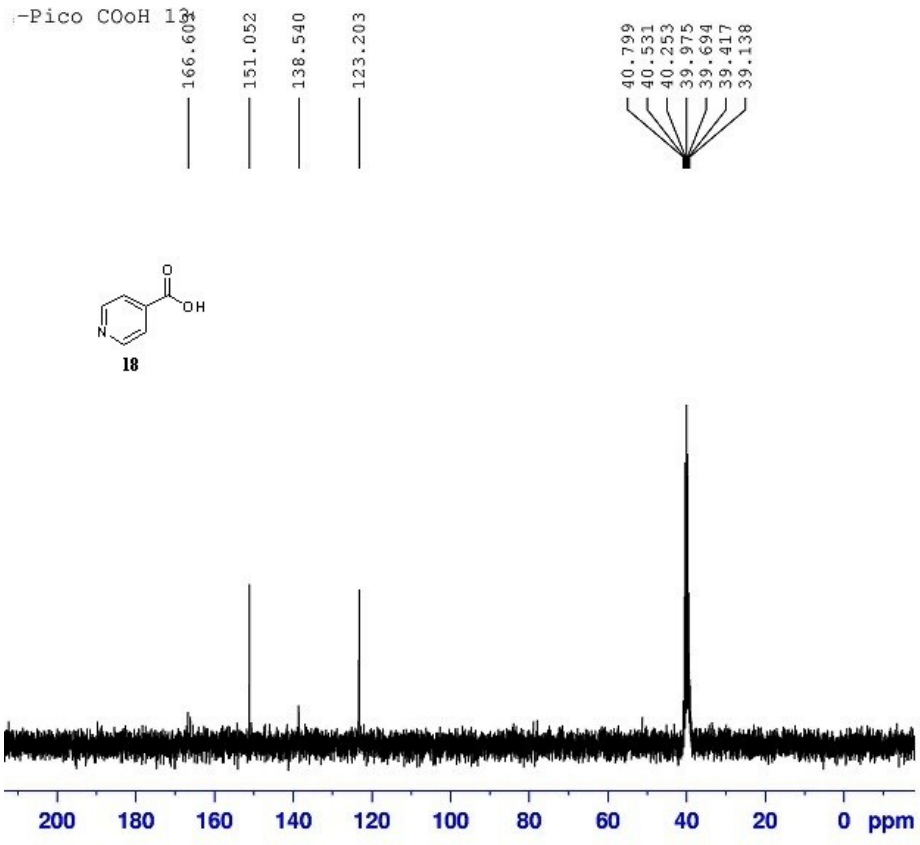



```
Current Data Parameters
NAME                  Sumanta
EXPNO                     207
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170527
Time                   12.12
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         24
DS                          4
SWH                 17985.611 Hz
FIDRES              0.274439 Hz
AQ                  1.8219508 sec
RG                      32768
DW                     27.800 usec
DE                      6.00 usec
TE                     300.0 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                       8.00 usec
PL1                     -3.00 dB
SFO1                75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                     -1.00 dB
PL12                    16.23 dB
PL13                    20.23 dB
SFO2               300.1312005 MHz

F2 - Processing parameters
SI                         32768
SF                  75.4677490 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

^{1}H- NMR (in DMSO-d_6) spectra of compound **18**

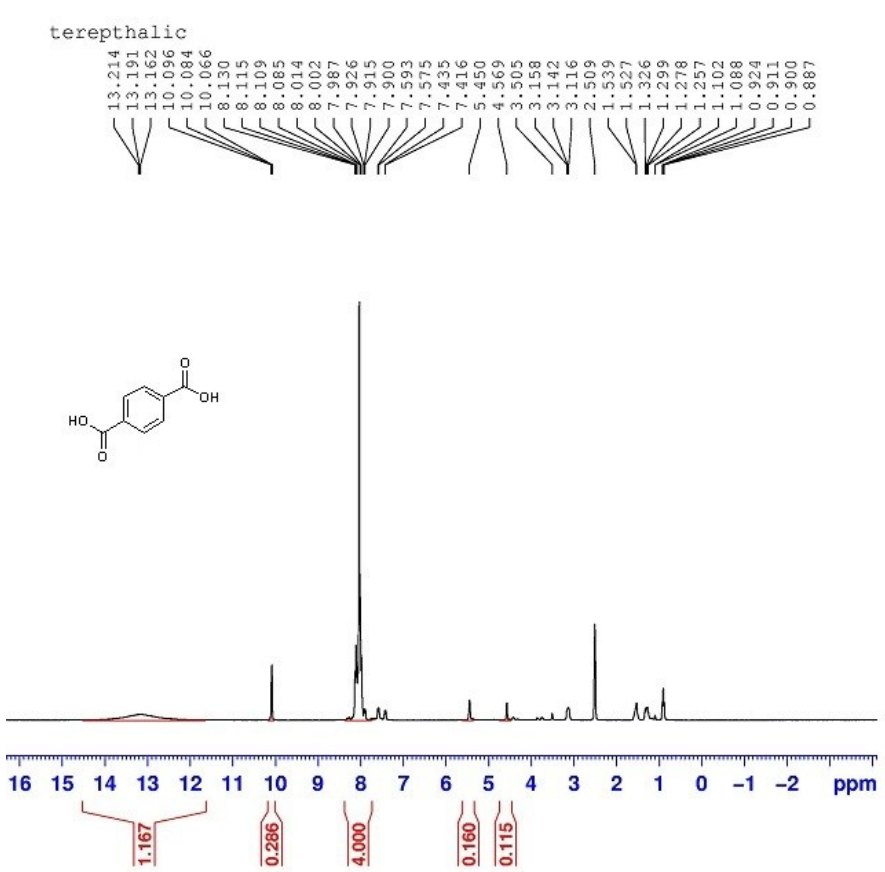


$^{13}C\{H^1\}$- NMR (in DMSO-d_6) spectra of compound **18**



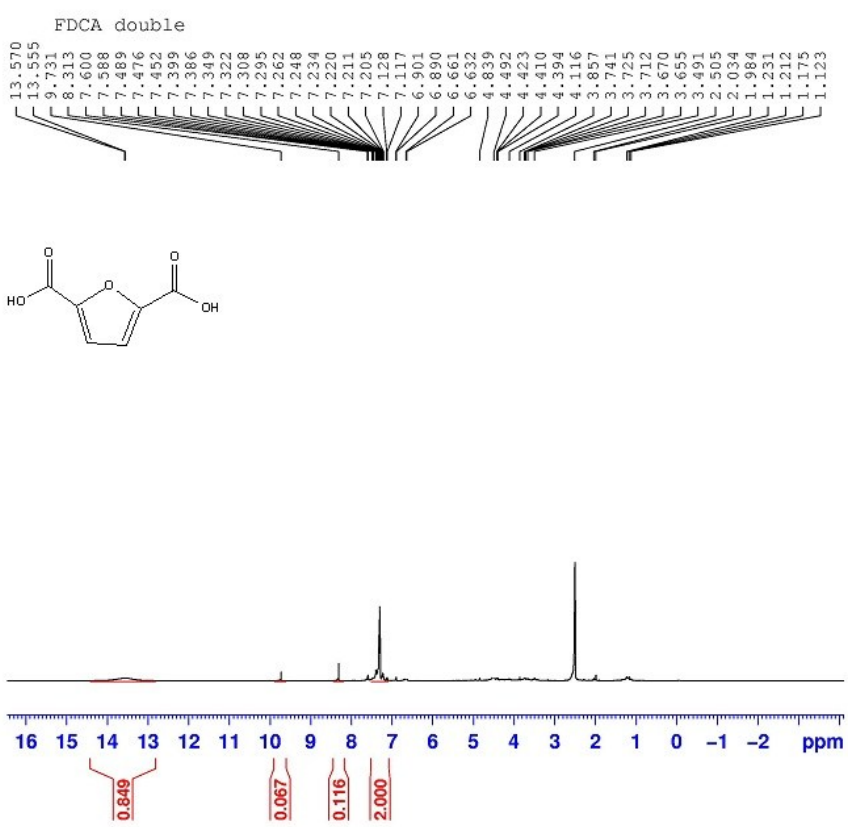

```
EXPNO                  233
PROCNO                   1

F2 - Acquisition Paramet
Date_             20170527
Time                 18.58
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                     33
DS                      4
SWH             17985.611
FIDRES          0.274439
AQ              1.8219508
RG                  32768
DW                 27.800
DE                  6.00
TE                 300.0
D1             2.00000000
d11            0.03000000
DELTA          1.89999998
TD0                     1

======== CHANNEL f1 ====
NUC1                  13C
P1                   8.00
PL1                 -3.00
SFO1          75.4752953

======== CHANNEL f2 ====
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00
PL2                 -1.00
PL12                16.23
PL13                20.23
SFO2         300.1312005
```

^{1}H- NMR (in DMSO-d_6) spectra of compound **19**



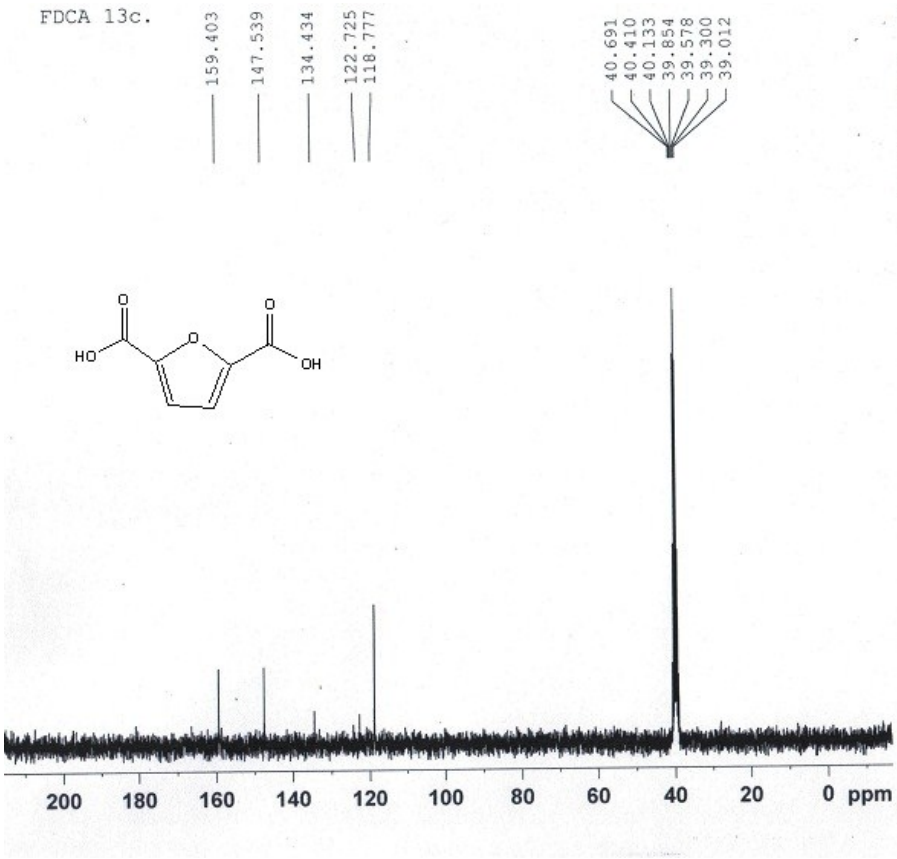

```
EXPNO                  259
PROCNO                   1

F2 - Acquisition Paramet
Date_             20170803
Time                 20.20
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH               6172.839
FIDRES            0.094190
AQ                5.3084660
RG                     90.5
DW                   81.000
DE                    6.00
TE                   300.0
D1              1.00000000
TD0                      1

======== CHANNEL f1 ====
NUC1                   1H
P1                  11.00
PL1                 -1.00
SFO1          300.1318534

F2 - Processing paramete
SI                   32768
SF            300.1300000
WDW                    EM
SSB                     0
LB                   0.30
GB                      0
PC                   1.00
```

^{1}H- NMR (in DMSO-d_6) spectra of compound **20**



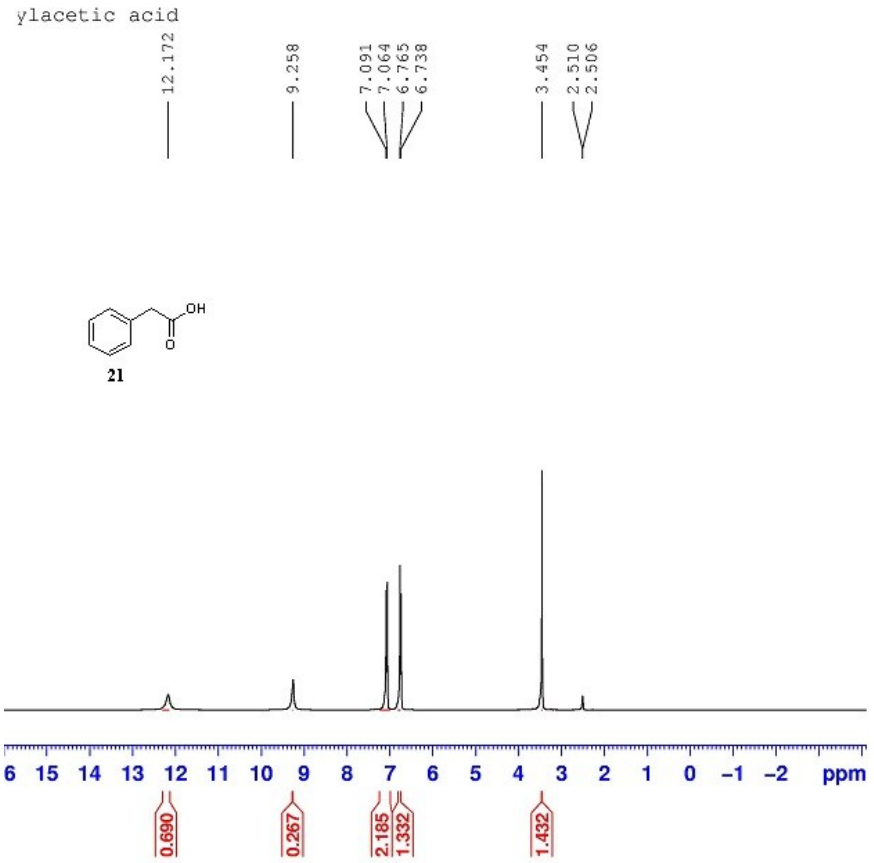

```
EXPNO                  303
PROCNO                   1

F2 - Acquisition Paramet
Date_             20170822
Time                 13.25
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT               DMSO
NS                      16
DS                       2
SWH               6172.839
FIDRES            0.094190
AQ                5.3084660
RG                    287.4
DW                   81.000
DE                     6.00
TE                    300.0
D1              1.00000000
TD0                      1

======== CHANNEL f1 ====
NUC1                    1H
P1                   11.00
PL1                  -1.00
SFO1           300.1318534

F2 - Processing paramete
SI                   32768
SF              300.1300000
WDW                     EM
SSB                      0
LB                    0.30
GB                       0
PC                    1.00
```

$^{13}C\{H^1\}$- NMR (in DMSO-d_6) spectra of compound **20**



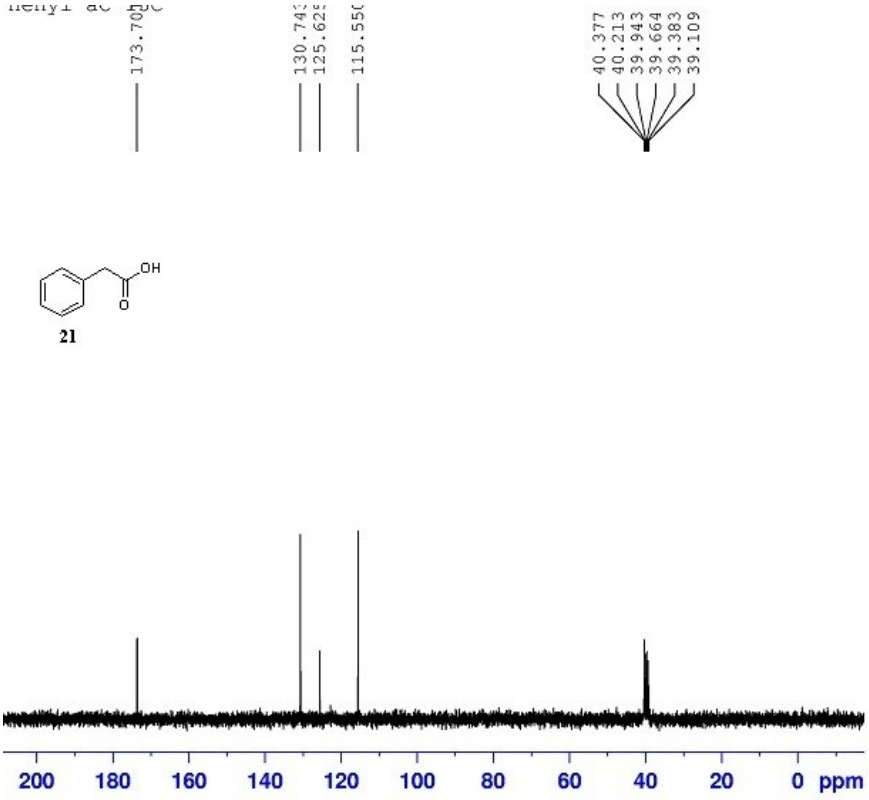

```
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170914
Time                11.12
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                      21
DS                       4
SWH             17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                   32768
DW                27.800 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                  8.00 usec
PL1                -3.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               16.23 dB
PL13               20.23 dB
SFO2        300.1312005 MHz
```

^{1}H- NMR (in DMSO-d_6) spectra of compound **21**



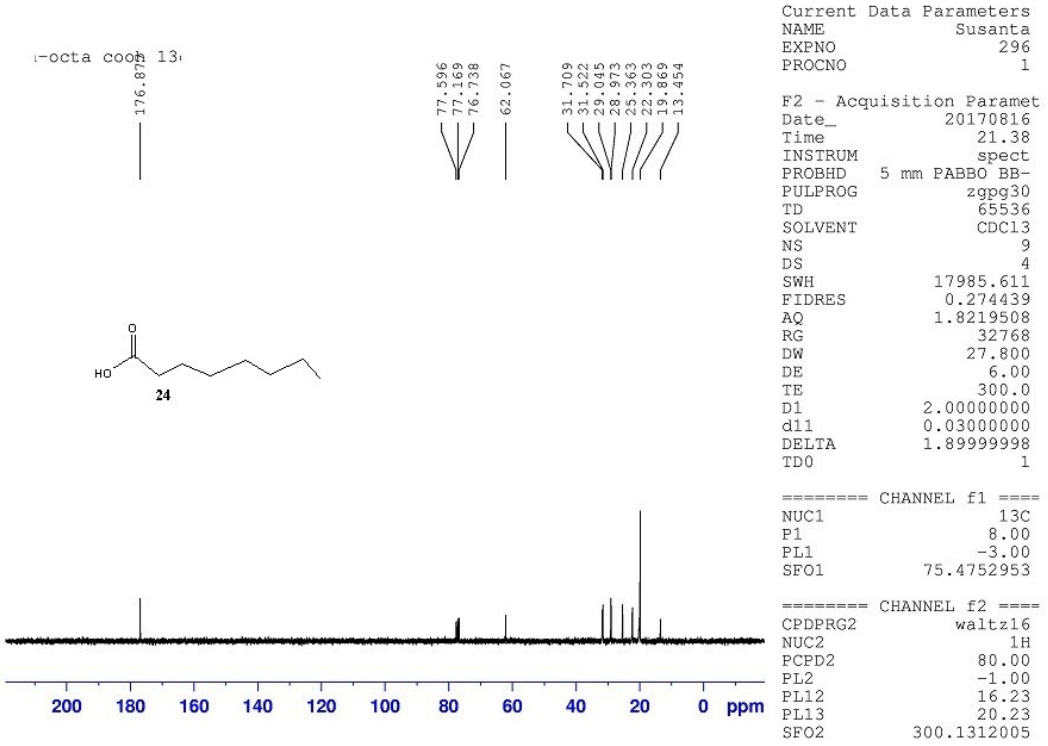

```
PROCNO                    1

F2 - Acquisition Paramet
Date_              20170805
Time                   9.57
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT               DMSO
NS                      16
DS                       2
SWH                6172.839
FIDRES             0.094190
AQ                 5.3084660
RG                      40.3
DW                    81.000
DE                      6.00
TE                     300.0
D1              1.00000000
TD0                      1

======== CHANNEL f1 ====
NUC1                    1H
P1                   11.00
PL1                  -1.00
SFO1           300.1318534

F2 - Processing paramete
SI                   32768
SF             300.1300000
WDW                     EM
SSB                      0
LB                    0.30
GB                       0
PC                    1.00
```

$^{13}C\{H^1\}$- NMR (in DMSO-d_6) spectra of compound **21**



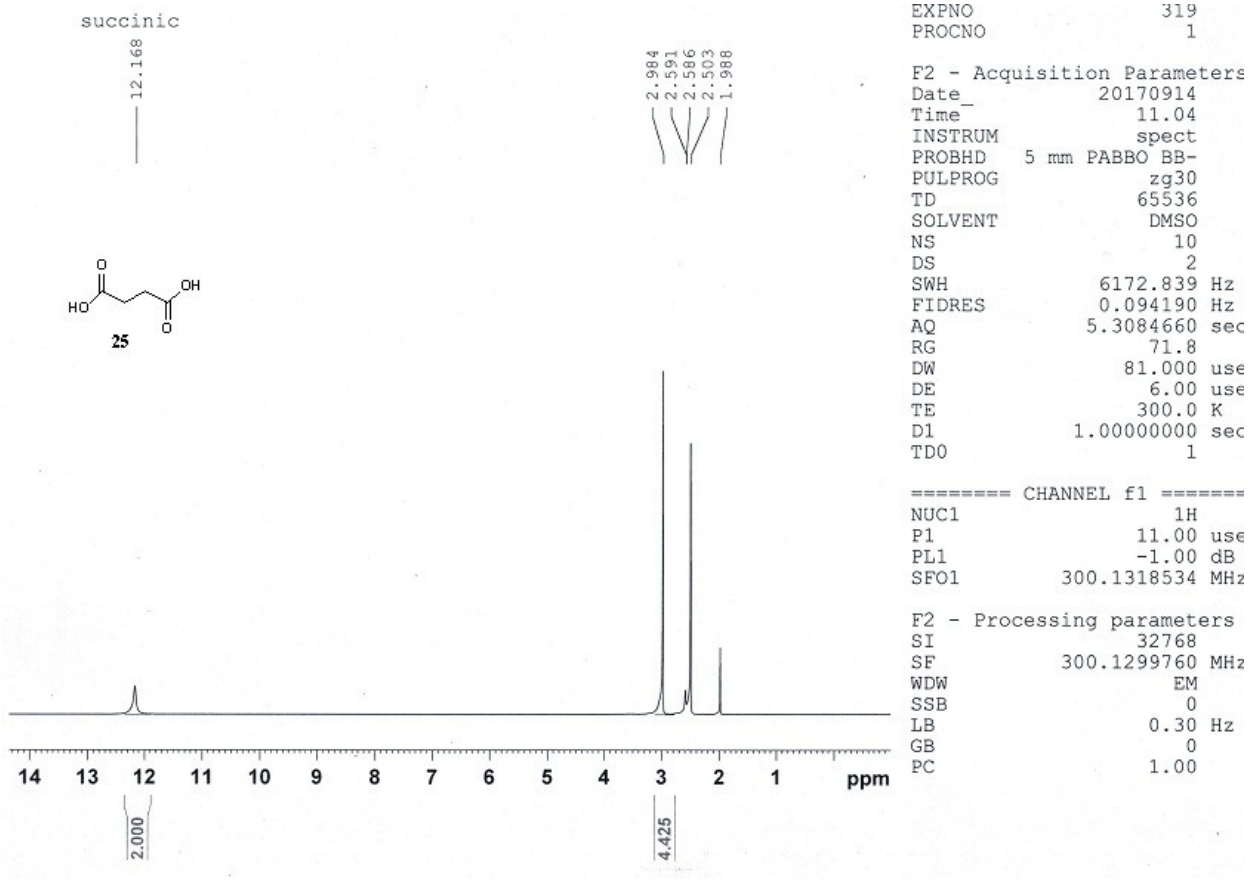
PROCNO	1
F2 - Acquisition Paramet	
Date_	20170805
Time	10.03
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	18
DS	4
SWH	17985.611
FIDRES	0.274439
AQ	1.8219508
RG	32768
DW	27.800
DE	6.00
TE	300.0
D1	2.00000000
d11	0.03000000
DELTA	1.89999998
TD0	1
======== CHANNEL f1 ====	
NUC1	13C
P1	8.00
PL1	-3.00
SFO1	75.4752953
======== CHANNEL f2 ====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00
PL2	-1.00
PL12	16.23
PL13	20.23
SFO2	300.1312005

$^{13}C\{H^1\}$- NMR (in CDCl3) spectra of compound **24**

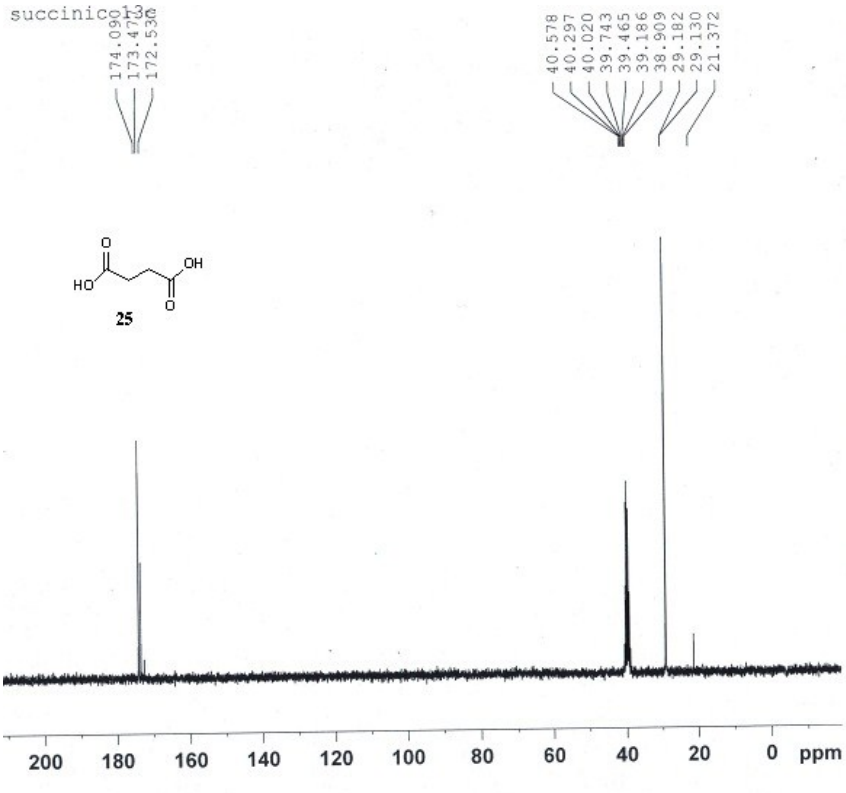



```
Current Data Parameters
NAME                  Susanta
EXPNO                     296
PROCNO                      1

F2 - Acquisition Paramet
Date_                20170816
Time                    21.38
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                          9
DS                          4
SWH                 17985.611
FIDRES               0.274439
AQ                   1.8219508
RG                       32768
DW                      27.800
DE                       6.00
TE                      300.0
D1                 2.00000000
d11                0.03000000
DELTA              1.89999998
TD0                         1

======== CHANNEL f1 ====
NUC1                      13C
P1                       8.00
PL1                     -3.00
SFO1               75.4752953

======== CHANNEL f2 ====
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00
PL2                     -1.00
PL12                    16.23
PL13                    20.23
SFO2              300.1312005
```

^{1}H- NMR (in DMSO-d_6) spectra of compound **25**

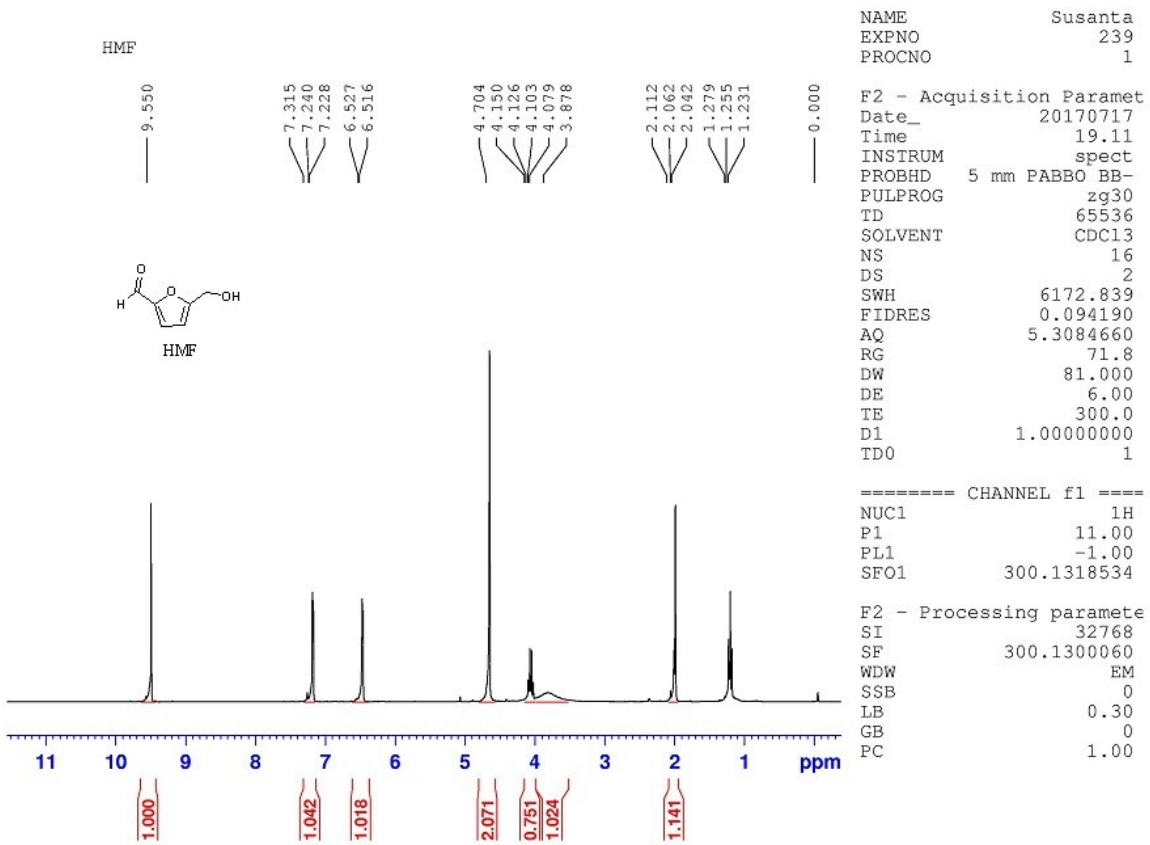


$^{13}C\{H^1\}$- NMR (in CDCl3) spectra of compound **25**

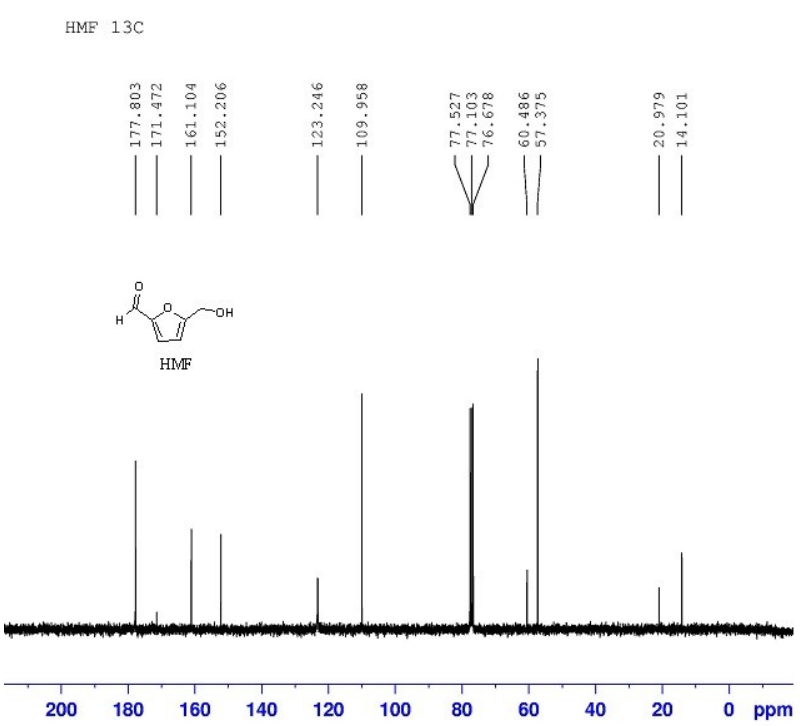


PROCNO	1
F2 - Acquisition Parameters	
Date_	20170914
Time	11.09
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	35
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	32768
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	-3.00 dB
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	16.23 dB
PL13	20.23 dB
SFO2	300.1312005 MHz

^{1}H- NMR (in $CDCl_3$) spectra of HMF



$^{13}C\{H^1\}$- NMR (in CDCl3) spectra of HMF




```
NAME                 Susan
EXPNO                   2
PROCNO

F2 - Acquisition Parar
Date_             201707
Time                19.
INSTRUM              spe
PROBHD   5 mm PABBO BI
PULPROG             zgpg
TD                   655
SOLVENT              CDC
NS                     
DS
SWH              17985.6
FIDRES            0.2744
AQ               1.82195
RG                   327
DW                  27.8
DE                   6.
TE                  300.
D1               2.000000
d11              0.030000
DELTA            1.899999
TD0

======== CHANNEL f1 ==
NUC1                   1
P1                   8.
PL1                 -3.
SFO1           75.475295

======== CHANNEL f2 ==
CPDPRG2            waltz
NUC2
PCPD2                80.
PL2                  -1.
PL12                 16.
PL13                 20.
SFO2          300.13120
```