Electronic Supplementary Material (ESI) for Green Chemistry.

A promising catalyst for exclusive *para* hydroxylation of substituted aromatic hydrocarbons under UV light

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(1) General Experimental Methods

The chemicals and reagents were purchased from Sigma-Aldrich, Merck, M/S S. D. Fine Chemicals Pvt. Ltd. and Loba chemical, and used without further purification. Melting points were determined in a Büchi 504 apparatus. ^1H and ^{13}C NMR spectra were recorded in a 400 MHz NMR spectrophotometer (JEOL, JNM ECS) using tetramethylsilane (TMS) as the internal standard and coupling constants are expressed in Hertz. Visualization was accomplished with UV lamp or I_2 stain. Reactions were monitored by thin-layer chromatography using aluminium sheets with silica gel 60 F254 (Merck). The products were isolated by column silica gel (60-120 mesh) using EtOAc:Hexane as an eluent. Isophorone diisocyanate (Aldrich, Germany), poly(ethylene glycol) with number average molecular weight (M_n) 600 g/mol (PEG 600, Merck, India), 2,2- bis(hydroxymethyl) propionic acid (Aldrich, Germany), 1,4 butane diol (BD, Merck, India), tannic acid (TA, Sigma-Aldrich, Belgium), triethylamine (Merck, India) and tetrahydrofuran (Merck, India) were used in the polymer preparation. Glycerol based hyperbranched epoxy and vegetable oil based poly(amide amine) were used to cross-linked the fabricated CD/WPU system. Corms of *Colocasia esculenta* (*C. esculenta*) obtained from Tezpur University campus was used to prepare CD. On the other hand**,** ethanol (C_2H_5OH, Merck, India), millipore water (H_2O, obtained from MilliQ® Ultrapure Water Solutions, Type 1, water purifier system) and oxygen (O_2, obtained from Jainex Gas Company, India) were used as starting materials in the photo-catalytic application. Potassium permanganate ($KMnO_4$, Merck, India) was used in the estimation of hydrogen peroxide following a titrimetric method.

(2) Preparation of CD by hydrothermal method

CD was prepared by heating the aqueous extract of corms of *Colocasia esculenta* as reported earlier [1]. Briefly, *C. esculenta* corm (50 g) was cut into small pieces and ground into paste by mixing in distilled water (150 mL). The mixture was filtered through a muslin cloth and collected the filtrate. Then the aqueous extract (25 mL) was mixed with ethanol (25 mL). Mixture was heated at 150 °C for 3.5 h. The soluble fraction of the dark brown colored residue was dissolved in distilled water (25 mL) and separated by filtration. In order to separate the large particles, ethanol (50 mL) was added to the filtrate and centrifuged at 960 × g (3000 rpm) for 15 min. Water was removed under reduced pressure to get the CD.

(3) Preparation of waterborne hyperbranched polyurethane/carbon dot nanocomposite

Carbon dot encapsulated thermosetting hyperbranched waterborne polyurethane was prepared according to the method as described elsewhere [1]. Briefly, isophorone diisocyanate (2.00 mol, Sigma-Aldrich, Germany), poly(ethylene glycol) with number average molecular weight 600 (0.80 mol, Merck, India), bis(hydroxyl methyl) propionic acid (0.53 mol, Sigma-Aldrich, Germany) were reacted in a four necked glass reactor equipped with a condenser, mechanical stirrer and nitrogen inlet. Reaction was carried out for 1.5 h in nitrogen atmosphere with stirring at temperature 85±2 °C. In the next step, 1,4-butane diol (0.50 mol, Merck, India) and tannic acid (0.07 mol, Sigma-Aldrich, Belgium) in limited amount of tetrahydrofuran (Merck, India) were added and the reaction was carried out within a close system at 70±2 °C for 3-3.5 h. In the next step, CD (in 1, 2, 3, 4 and 5 wt%) and 1,4-butane diol were added and the reaction was continued for another 1.5 h at 70±2 °C. Then triethyl amine (0.53 mol, Merck, India) was added slowly at room temperature to create the ionic centers by neutralizing –COOH groups. After this step water was added at a very slow and steady rate with constant mechanical stirring. Finally, tetrahydrofuran was removed carefully under reduced pressure. This polymeric system was cured with 20% glycerol based hyperbranched epoxy and fatty acid based poly(amido amine) using sound and heat energy [1].

(4) Calculation for solid contents in CD

Since the solid content of 1 mL of CD is 6.272 mg

Therefore, the solid content of 5 mL of CD is 31.36 mg.

The solid content of 5 wt% of CD is equal to 11.2 mg (Table 3).

(5) General synthetic procedures for hydroxylation of aromatic hydrocarbons

Aromatic hydrocarbons (1 equivalent) and finely cut thin film of CD@WPU (5 wt%) were stirred in an oven dried round bottomed flask (50 mL) followed by the drop wise addition of H_2O_2 (3 equivalent). The whole contents in the flask were irradiated with UV light under stirring condition for the required period of time and the reaction was monitored by TLC. After the completion of the reaction, ethyl acetate (10 mL) was added to the reaction mixture and filtered to recover the catalyst. The filtrate was washed with water and brine, dried over anhydrous Na_2SO_4, concentrated in a rotary evaporator, and finally the crude product was purified by column chromatography (15% ethyl acetate: hexane as the eluent). The recovered finely cut thin film of catalyst was further washed with ethyl acetate (3x15 mL) and dried at 60 °C for 5 h for reutilizing in the next run of the reaction. All the products were prepared using this method and characterized by ^{1}H NMR, ^{13}C NMR and also by comparing the melting points from the literature.

5.1 Table 2, Entry 1: Procedures for the production of *p*-hydroxy benzoic acid from benzoic acid

Benzoic acid (1 equivalent, 122 mg) was reacted with dropwise addition of H_2O_2 (30% v/v, 3 equivalent, 102 mg) in presence of finely cut thin film of CD@WPU (5 wt%, 11.2 mg) by stirring under ambient conditions and irradiated with UV light (365 nm) for 60 minutes. The product was isolated from the reaction mixture by filtering followed by column chromatography

(15% ethyl acetate: hexane as the eluent). Yield of *p*-hydroxy benzoic acid was 135.24 mg (98%) and selectivity 100%.

5.2 Table 2, Example 2: Process for the production of *p*-hydroxy salicylic acid from salicylic acid

Salicylic acid (1 equivalent, 138 mg) was reacted with dropwise addition of H_2O_2 (30% v/v, 3 equivalent, 102 mg) in presence of finely cut thin film of CD@WPU (5 wt%, 12 mg) by stirring under ambient conditions and irradiated with UV light (365 nm) for a period of 60 minutes. The rest of the procedure was same as example 1. Yield of *p*-hydroxy salicylic acid was 141.68 mg (92%) and selectivity 100%.

5.3 Table 2, Example 3: Process for the production of *p*-hydroxy nitrobenzene from nitrobenzene

Nitrobenzene (1 equivalent, 123 mg) was reacted with dropwise addition of H_2O_2 (30% v/v, 3 equivalent, 102 mg) in presence of finely cut thin film of CD@WPU (5 wt%, 11 mg) by stirring under ambient conditions and irradiated with UV light (365 nm) for 90 minutes. The rest of the procedure was same as example 1. Yield of to *p*-hydroxy nitrobenzene was 122.32 mg (88%) and selectivity 100%.

5.4 Table 2, Example 4: Process for the production of *p*-hydroxy benzaldehyde from benzaldehyde

Benzaldehyde (1 equivalent, 106 mg) was reacted with dropwise addition of H_2O_2 (30% v/v, 3 equivalent, 102 mg) in presence of finely cut thin film of CD@WPU (5 wt%, 10 mg) by stirring under ambient conditions and irradiated with UV light (365 nm) for 70 minutes. The rest of the procedure was same as example 1. Yield of *p*-hydroxy benzaldehyde was 92.72 mg (76%) and selectivity 100%.

5.5 Table 2, Example 5: Process for the production of *p*-hydroxy phenol from phenol

Phenol (1 equivalent, 94 mg) was reacted with dropwise addition of H_2O_2 (30% v/v, 3 equivalent, 102 mg) in presence of finely cut thin film of CD@WPU (5 wt%, 10 mg) by stirring under ambient conditions and irradiated with UV light (365 nm) for 100 minutes. The rest of the procedure was same as example 1. Yield of *p*-hydroxy phenol was 66 mg (60%) and selectivity 100%.

5.6 Table 2, Example 6: Method for the preparation of *p*-hydroxy aniline from aniline

Aniline (1 equivalent, 93 mg) was reacted with dropwise addition of H_2O_2 (30% v/v, 3 equivalent, 102 mg) in presence of finely cut thin film of CD@WPU (5 wt%, 8 mg) by stirring under ambient conditions and irradiated with UV light (365 nm) for 100 minutes. The rest of the procedure was same as example 1. Yield of *p*-hydroxy aniline was 98.10 mg (90%) and selectivity 100%.

5.7 Table 2, Example 7: Process for the production of *p*-hydroxy benzonitrile from benzonitrile

Benzonitrile (1 equivalent, 103 mg) was reacted with dropwise addition of H_2O_2 (30% v/v, 3 equivalent, 102 mg) in presence of finely cut thin film of CD@WPU (5 wt%, 10 mg) by stirring under ambient conditions and irradiated with UV light (365 nm) for 130 minutes. The rest of the procedure was same as example 1. Yield of *p*-hydroxy benzonitrile was 69.02 mg (58%) and selectivity 100%.

5.8 Table 2, Example 8: Process for the production *p*-hydroxy chlorobenzene from chlorobenzene

Chlorobenzene (1 equivalent, 112 mg) was reacted with dropwise addition of H_2O_2 (30% v/v, 3 equivalent, 102 mg) in presence of finely cut thin film of CD@WPU (5 wt%, 11 mg) by stirring under ambient conditions and irradiated with UV light (365 nm) for 150 minutes. The rest of the procedure was same as example 1. Yield of *p*-hydroxy benzonitrile was 102.40 mg (80%) and selectivity 100%.

5.9 Table 2, Example 9: Process for the production *p*-cresol from toluene

Toluene (1 equivalent, 92 mg) was reacted with dropwise addition of H_2O_2 (30% v/v, 3 equivalent, 102 mg) in presence of finely cut thin film of CD@WPU (5 wt%, 10 mg) by stirring under ambient conditions and irradiated with UV light (365 nm) for 150 minutes. The rest of the procedure was same as example 1. Yield of *p*-hydroxy benzonitrile was 75.60 mg (70%) and selectivity 100%.

(6) Recycling potential of the catalyst

CD@WPU is recovered from the reaction mixture through simple filtration, washed several times with hot ethanol and dried at 60 °C prior to use for the next cycle. CD@WPU is recycled up to the 4th run in the hydroxylation reaction of benzoic acid with H_2O_2 for the formation of *para*-hydroxy benzoic acid. Herein, the catalyst showed retention in selectivity (100% in each runs) along with conversion (98% in each runs, 60 minute).

In order to avoid the use of excess amount of catalysts and to clarify the recyclability of CD@WPU, the reaction condition with a low yield as mentioned in entry 15 of Table 1 has been carried out and the results are depicted in Table S1. It can be observed from Table S1 that CD@WPU has been recycled from fresh up to the 4th run without loss in its action. However, slightly longer reaction time was required afterwards to achieve the desired yield of **4** because the finely sliced thin film of CD@WPU slightly stuck amongst each other after 4th run.

Table S1: Recycling chart of CD@WPU using 0.5 wt% CD in WPU[a]

Entry	Run	Time (min)	Yield (%)[b]
1	Fresh	60	54
2	1st	60	54
3	2nd	60	54
4	3rd	60	54
5	4th	60	54
6	5th	90	54
7	6th	120	54

[a]Reaction condition: **3** (5 mmol, 610 mg), H_2O_2 (15 mmol, 510 mg), CD@WPU (5 wt%, 56 mg), UV light. [b] Isolated yield.

(7) UV-visible spectra

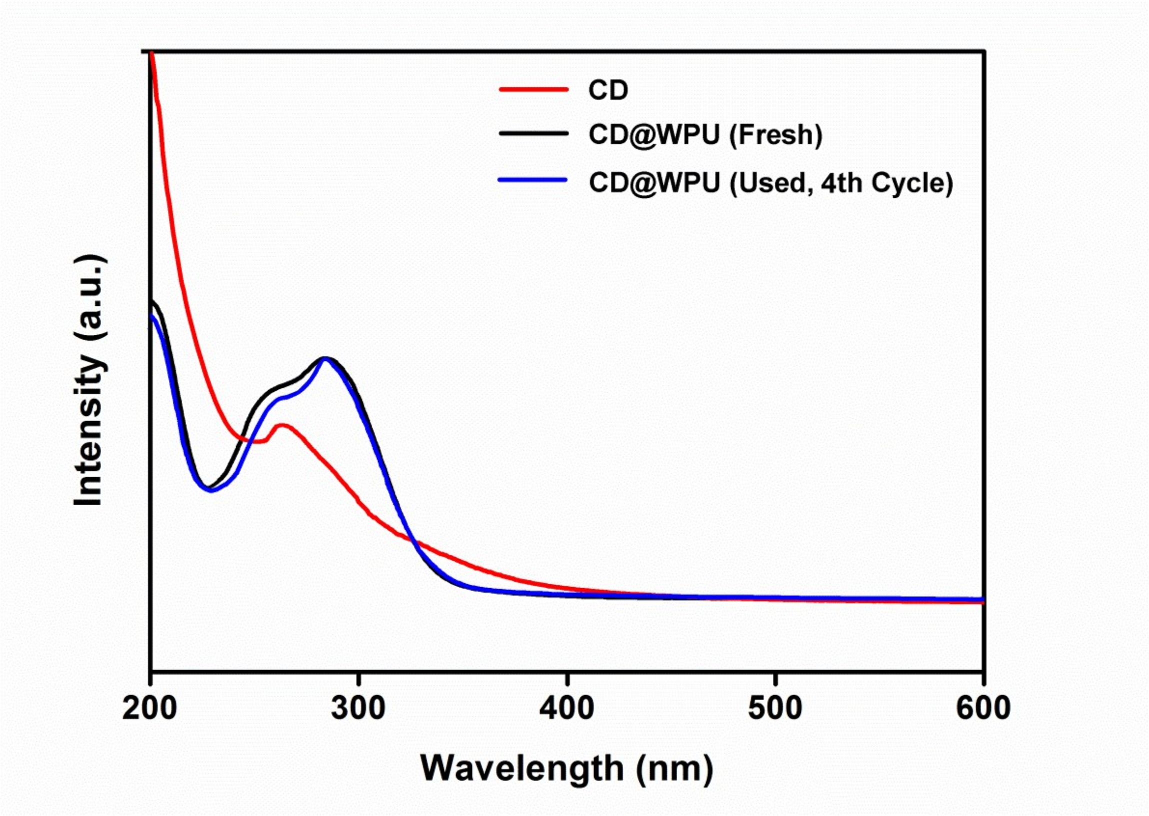


Figure S1. UV-visible spectra of CD, CD@WPU (Fresh and used after 4th consecutive run).

(8) TEM images

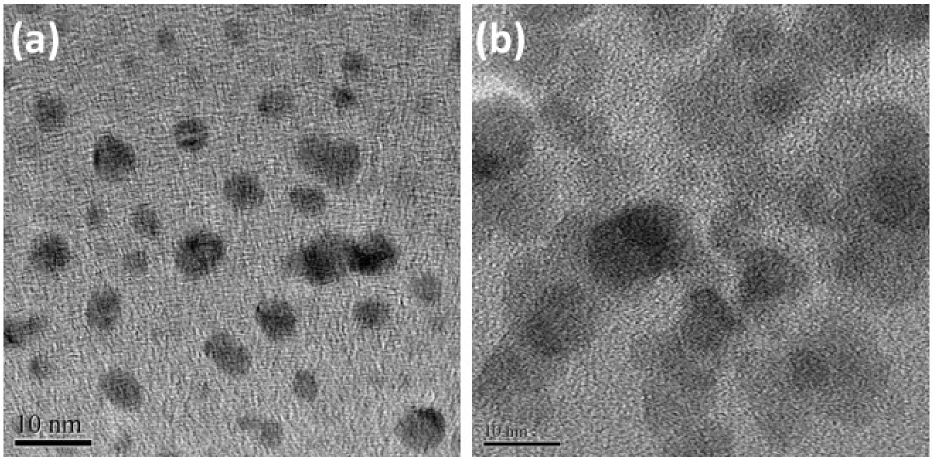


Figure S2. TEM images of CD@WPU (a) Fresh, (b) After 4th consecutive run.

(9) FTIR spectra

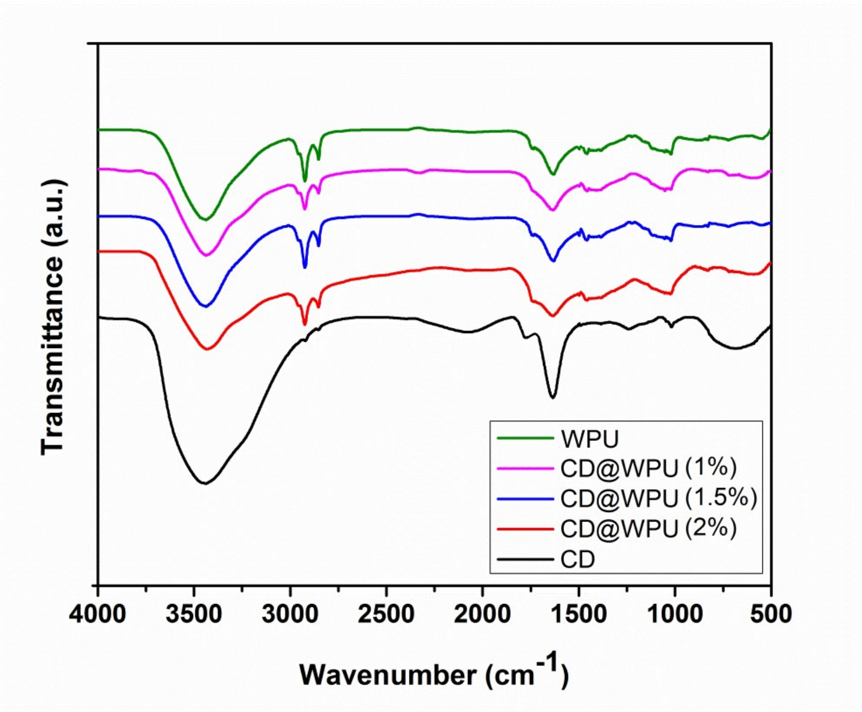


Figure S3. FTIR spectra of CD, WPU and CD@WPU with different CD loading.

(10) Characteristics of CD, WPU and CD@WPU

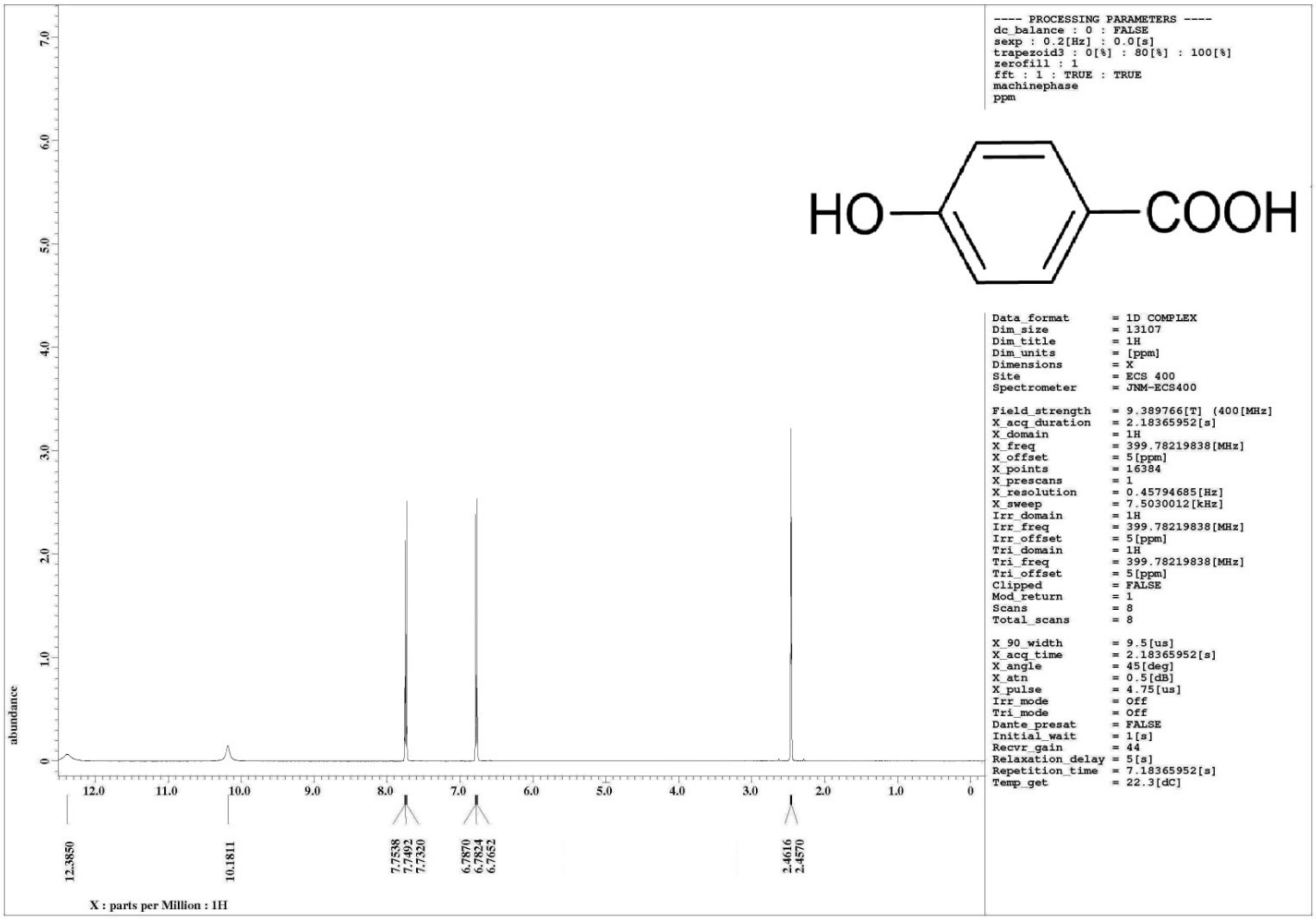
The WPU is characterized by λ_{max}=280 nm and chemical shift (^{1}H NMR, δ ppm) values at 0.86-0.89, 1.5-0.7, 3.4, 6.8-7.2, 7.7-7.9, 10.0, average molecular weight of 2.3x10^{4} g/mol, PDI: 1.23, tensile strength: 6-7 MPa, elongation at break: 450-470 %, and scratch hardness: 5-5.5 kg.

The CD is characterized by λ_{max}=269 nm, spherical shape with size distribution in the range of 1.5-3.5 nm, *d* spacing 0.39 nm, D band at 1362 cm^{-1}, G band at 1582 cm^{-1} with I_D/I_G ratio 0.57 and C:O=61.47:37.53.

The CD@WPU is characterized by tensile strength: 18-25 MPa, elongation at break: 250-300 %, toughness: 36-55 MPa, scratch hardness: >10 kg, *d* spacing: 0.41 nm, D band at 1362 cm^{-1}, G band at 1582 cm^{-1} and I_D/I_G ratio 0.61.

(11) ^{1}H NMR and ^{13}C NMR spectra of compounds

Table 2, Entry 1: ^{1}H NMR of *para*-hydroxy benzoic acid



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ppm
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Table 2, Entry 1: ^{13}C NMR of *para*-hydroxy benzoic acid

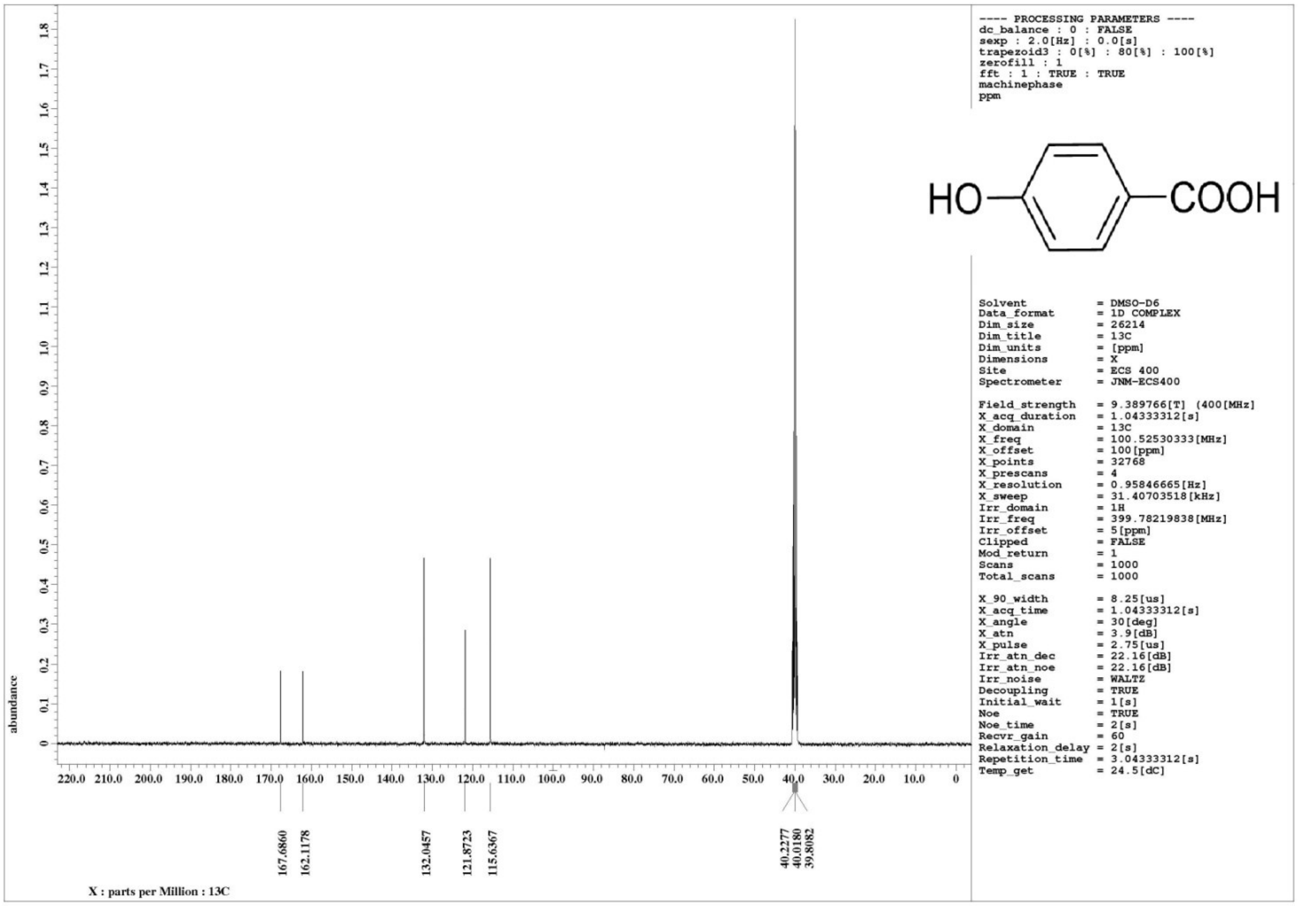

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Table 2, Entry 2: ^{1}H NMR of *para*-hydroxy salicylic acid

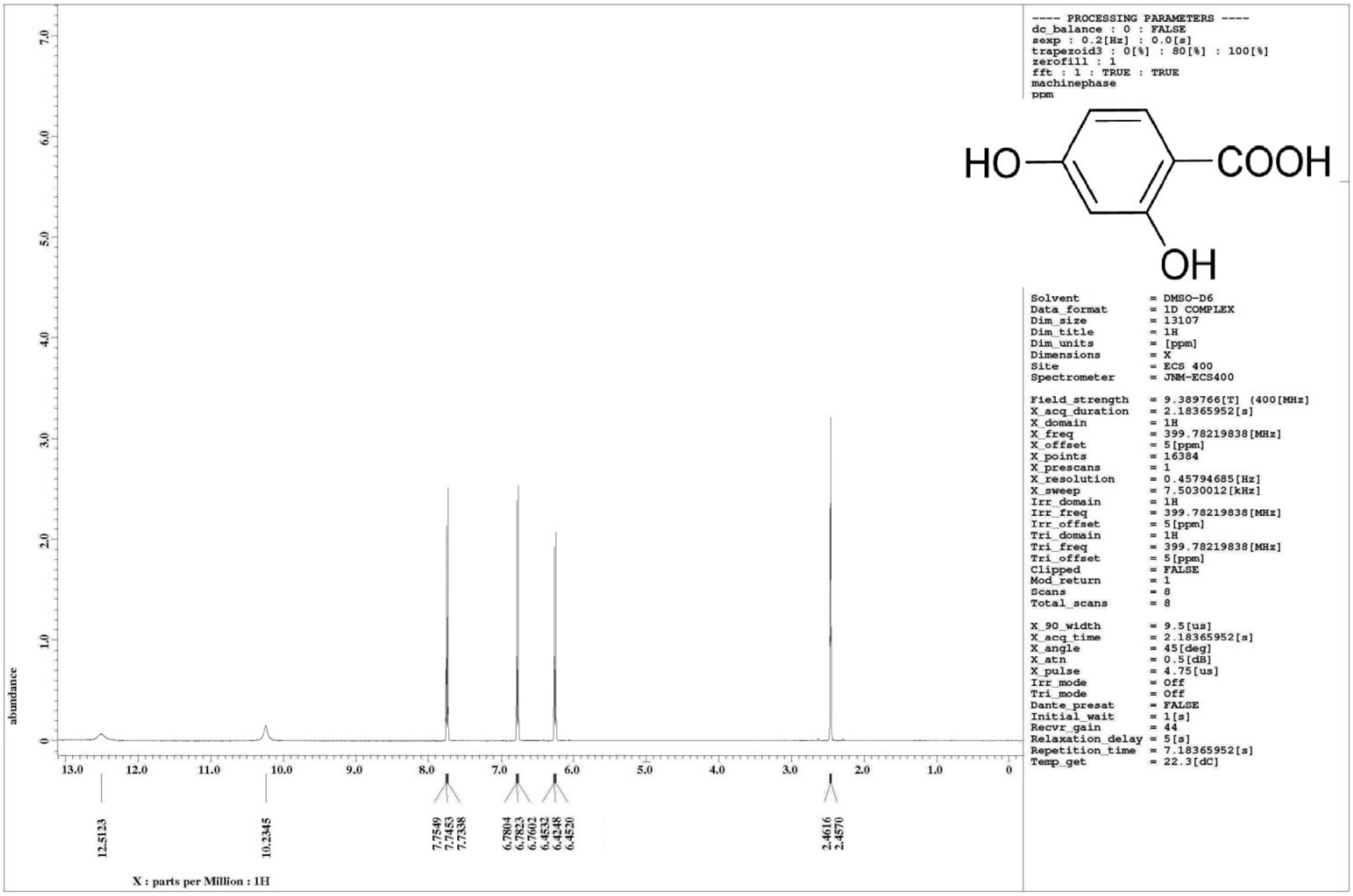

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Table 2, Entry 2: ^{13}C NMR of *para*-hydroxy salicylic acid

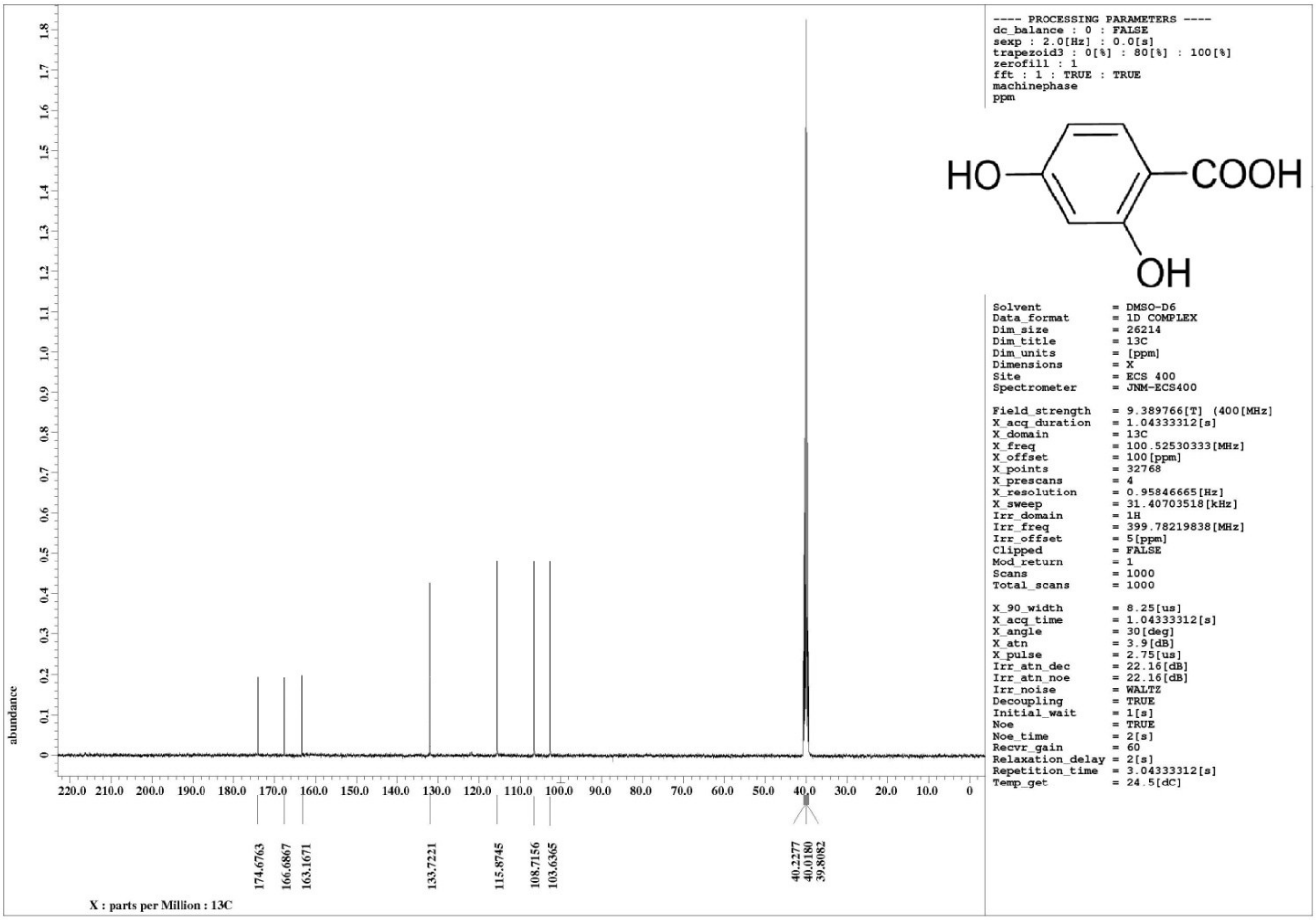

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Table 2, Entry 3: ^{1}H NMR of *para*-nitro phenol

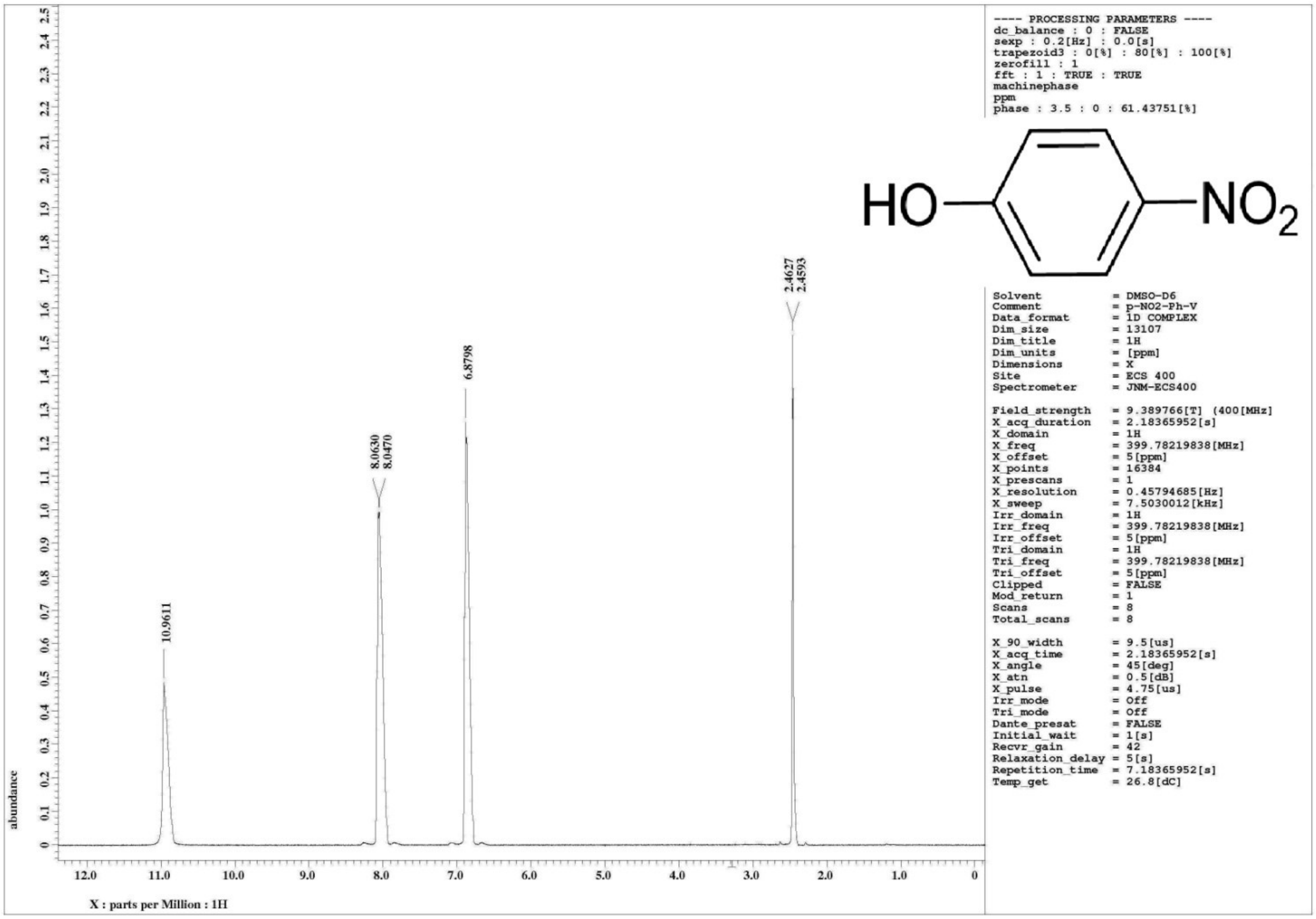

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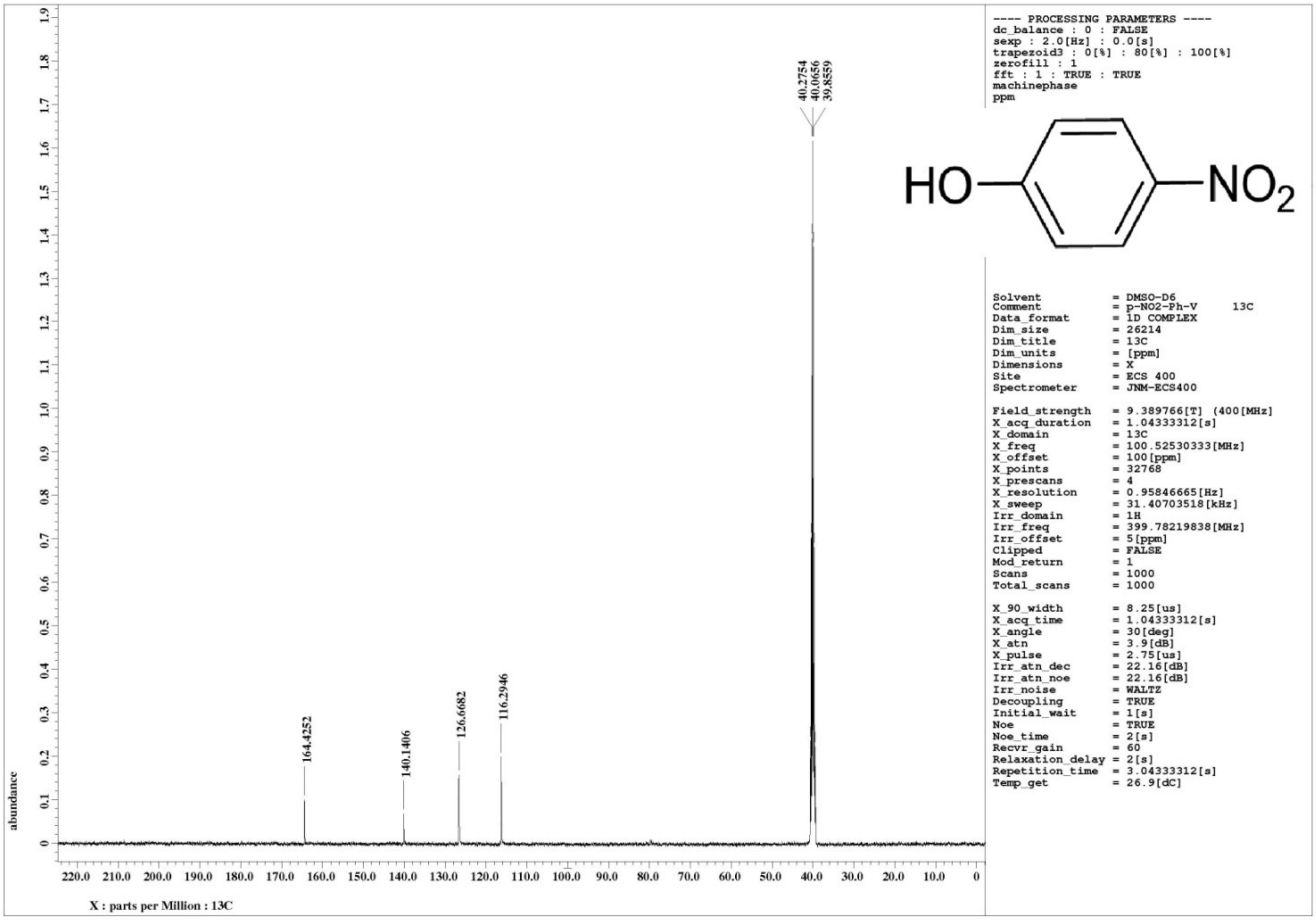
Table 2, Entry 3: ^{13}C NMR of *para*-nitro phenol



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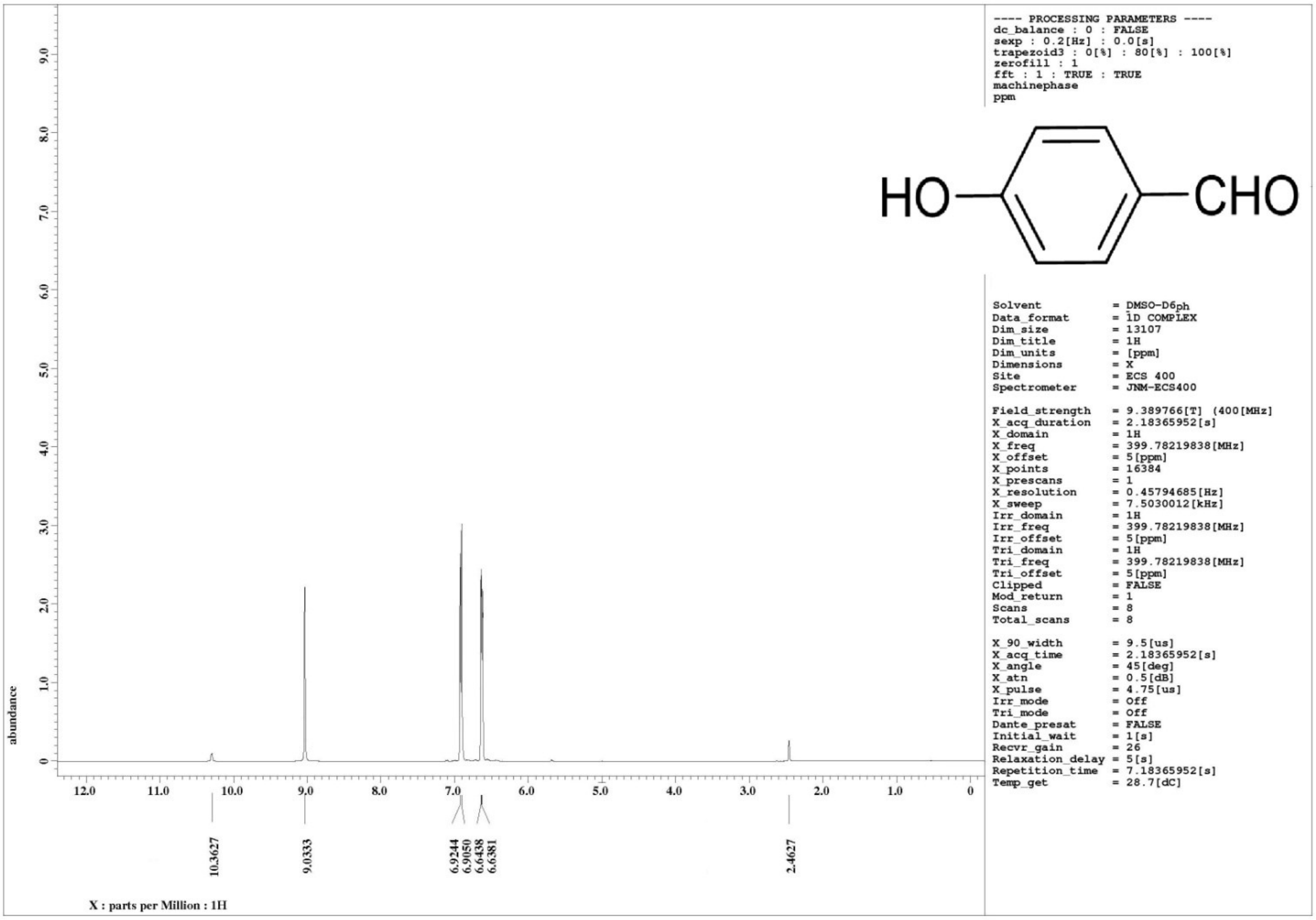

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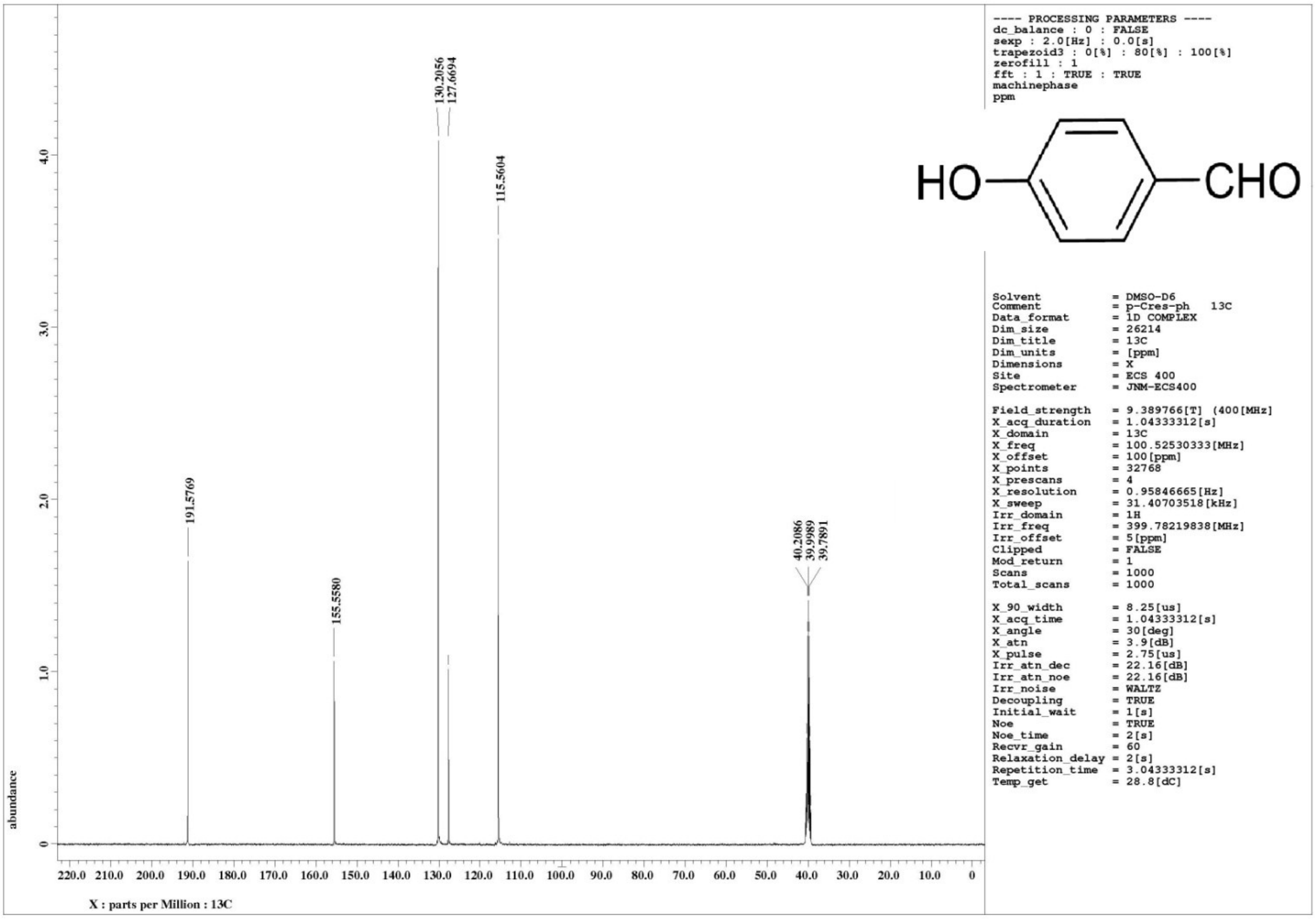
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Solvent           = DMSO-D6ph
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9.5[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 0.5[dB]
X_pulse           = 4.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 26
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 28.7[dC]
```

Table 2, Entry 4: ^{13}C NMR of *para*-hydroxy benzaldehyde



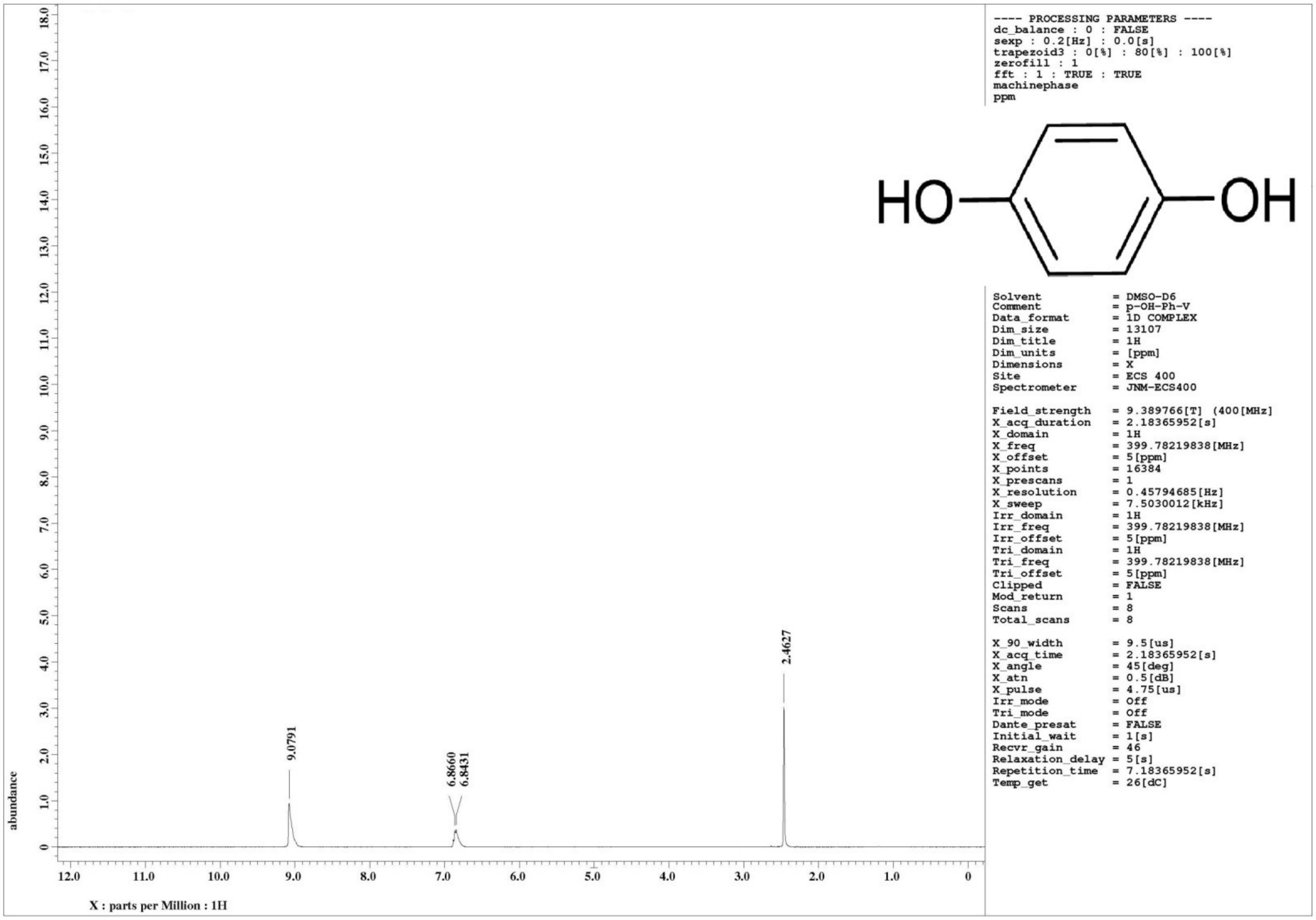
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Solvent          = DMSO-D6
Comment          = p-Cres-ph   13C
Data_format      = 1D COMPLEX
Dim_size         = 26214
Dim_title        = 13C
Dim_units        = [ppm]
Dimensions       = X
Site             = ECS 400
Spectrometer     = JNM-ECS400

Field_strength   = 9.389766[T] (400[MHz]
X_acq_duration   = 1.04333312[s]
X_domain         = 13C
X_freq           = 100.52530333[MHz]
X_offset         = 100[ppm]
X_points         = 32768
X_prescans       = 4
X_resolution     = 0.95846665[Hz]
X_sweep          = 31.40703518[kHz]
Irr_domain       = 1H
Irr_freq         = 399.78219838[MHz]
Irr_offset       = 5[ppm]
Clipped          = FALSE
Mod_return       = 1
Scans            = 1000
Total_scans      = 1000

X_90_width       = 8.25[us]
X_acq_time       = 1.04333312[s]
X_angle          = 30[deg]
X_atn            = 3.9[dB]
X_pulse          = 2.75[us]
Irr_atn_dec      = 22.16[dB]
Irr_atn_noe      = 22.16[dB]
Irr_noise        = WALTZ
Decoupling       = TRUE
Initial_wait     = 1[s]
Noe              = TRUE
Noe_time         = 2[s]
Recvr_gain       = 60
Relaxation_delay = 2[s]
Repetition_time  = 3.04333312[s]
Temp_get         = 28.8[dC]
```

Table 2, Entry 5: ^{1}H NMR of *para*-hydroxy phenol



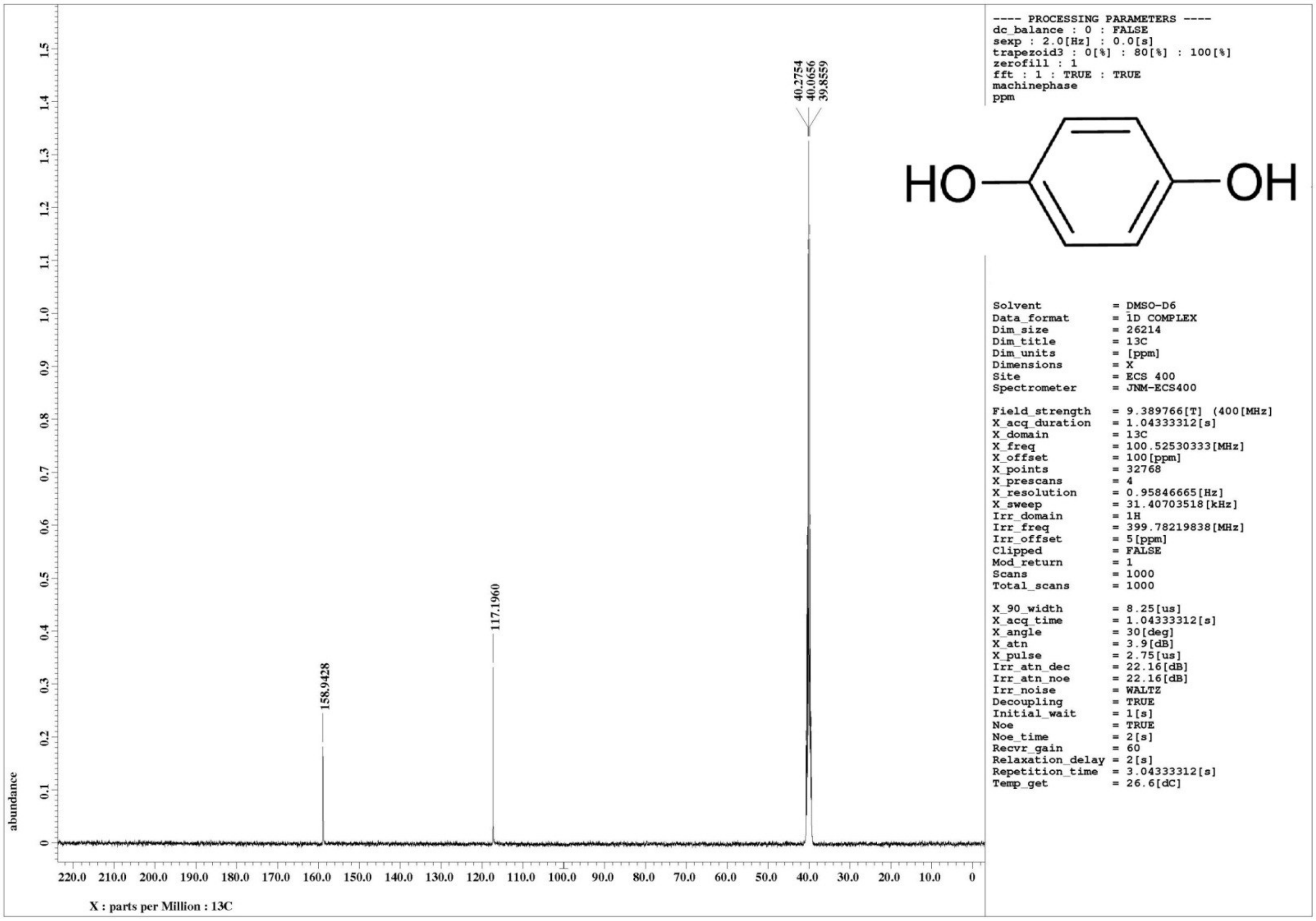
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Solvent           = DMSO-D6
Comment           = p-OH-Ph-V
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9.5[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 0.5[dB]
X_pulse           = 4.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 26[dC]
```

Table 2, Entry 5: ^{13}C NMR of *para*-hydroxy phenol



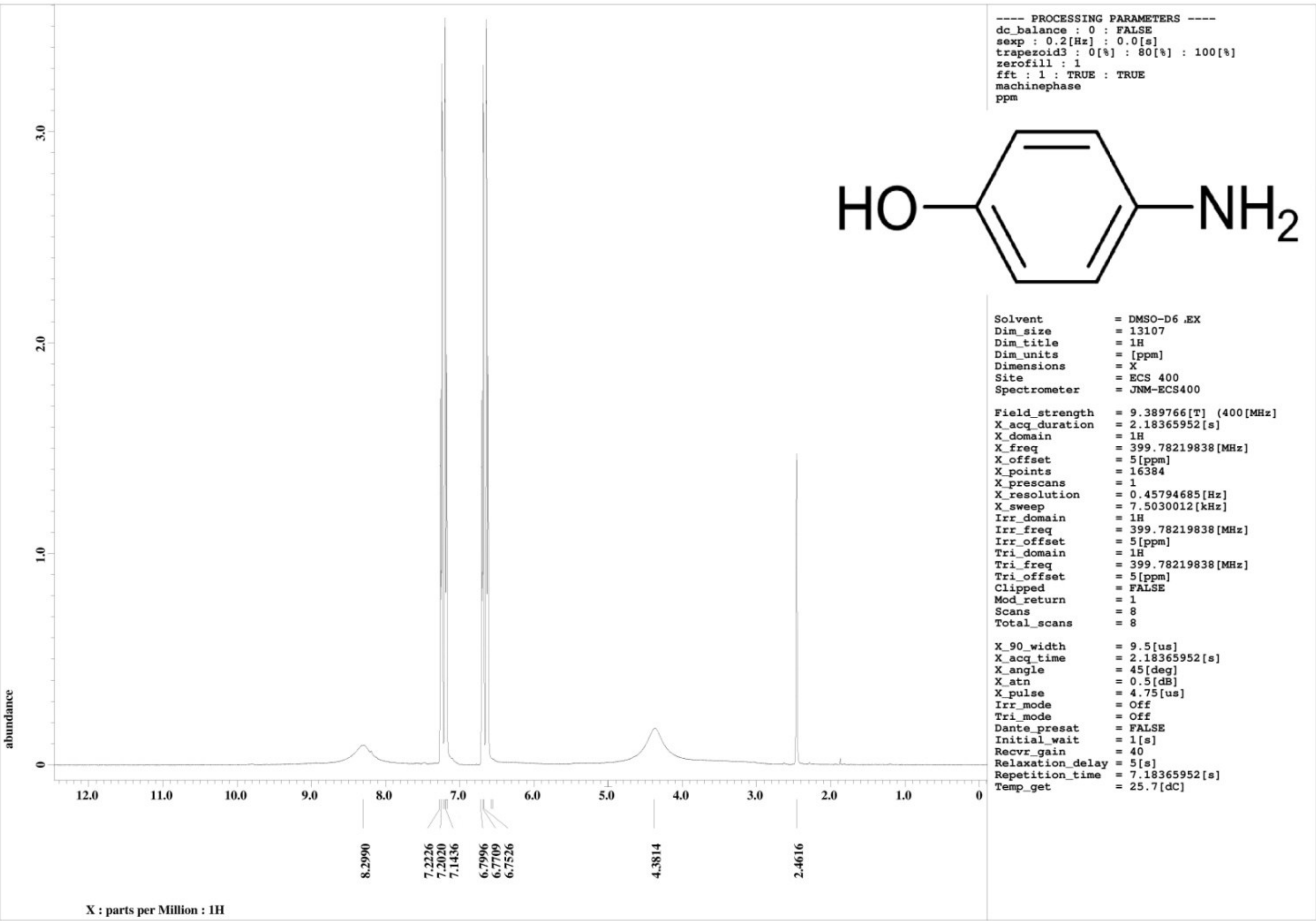
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Solvent           = DMSO-D6
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 8.25[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 3.9[dB]
X_pulse           = 2.75[us]
Irr_atn_dec       = 22.16[dB]
Irr_atn_noe       = 22.16[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.04333312[s]
Temp_get          = 26.6[dC]
```

Table 2, Entry 6: ^{1}H NMR of *para*-amino phenol



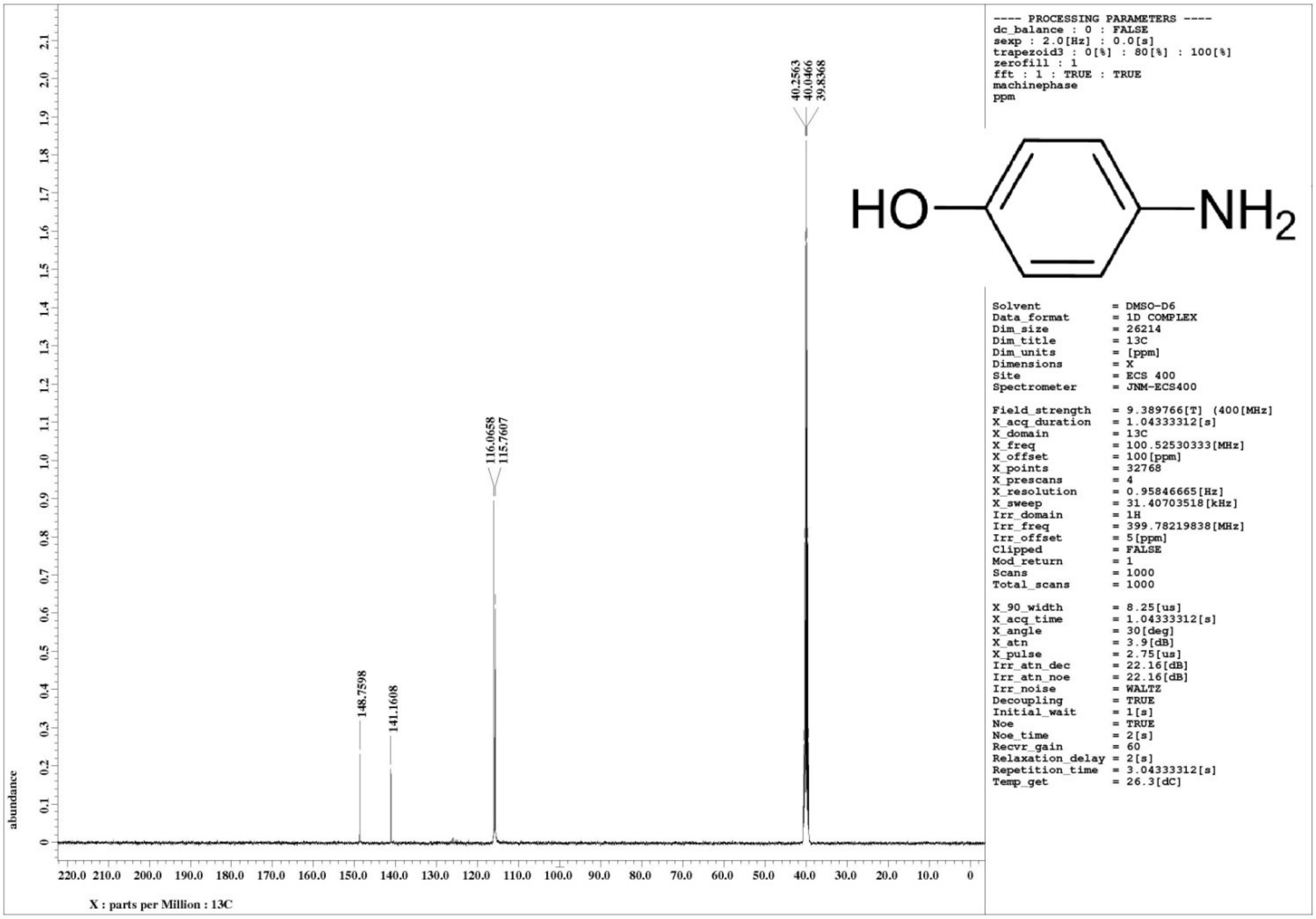
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Solvent          = DMSO-D6 .EX
Dim_size         = 13107
Dim_title        = 1H
Dim_units        = [ppm]
Dimensions       = X
Site             = ECS 400
Spectrometer     = JNM-ECS400

Field_strength   = 9.389766[T] (400[MHz]
X_acq_duration   = 2.18365952[s]
X_domain         = 1H
X_freq           = 399.78219838[MHz]
X_offset         = 5[ppm]
X_points         = 16384
X_prescans       = 1
X_resolution     = 0.45794685[Hz]
X_sweep          = 7.5030012[kHz]
Irr_domain       = 1H
Irr_freq         = 399.78219838[MHz]
Irr_offset       = 5[ppm]
Tri_domain       = 1H
Tri_freq         = 399.78219838[MHz]
Tri_offset       = 5[ppm]
Clipped          = FALSE
Mod_return       = 1
Scans            = 8
Total_scans      = 8

X_90_width       = 9.5[us]
X_acq_time       = 2.18365952[s]
X_angle          = 45[deg]
X_atn            = 0.5[dB]
X_pulse          = 4.75[us]
Irr_mode         = Off
Tri_mode         = Off
Dante_presat     = FALSE
Initial_wait     = 1[s]
Recvr_gain       = 40
Relaxation_delay = 5[s]
Repetition_time  = 7.18365952[s]
Temp_get         = 25.7[dC]
```

Table 2, Entry 6: ^{13}C NMR of *para*-amino phenol



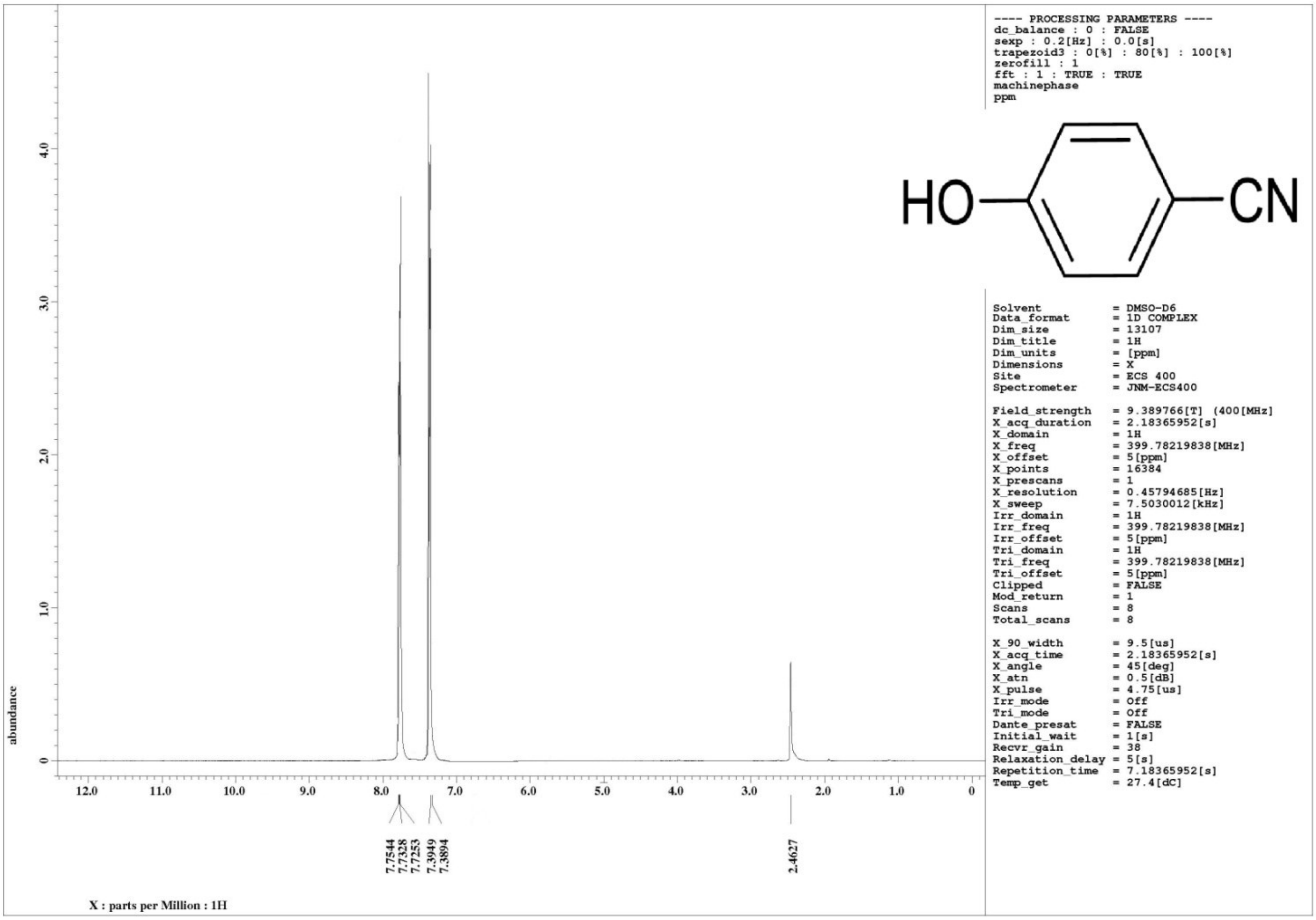
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Solvent            = DMSO-D6
Data_format        = 1D COMPLEX
Dim_size           = 26214
Dim_title          = 13C
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.389766[T] (400[MHz]
X_acq_duration     = 1.04333312[s]
X_domain           = 13C
X_freq             = 100.52530333[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 0.95846665[Hz]
X_sweep            = 31.40703518[kHz]
Irr_domain         = 1H
Irr_freq           = 399.78219838[MHz]
Irr_offset         = 5[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 1000
Total_scans        = 1000

X_90_width         = 8.25[us]
X_acq_time         = 1.04333312[s]
X_angle            = 30[deg]
X_atn              = 3.9[dB]
X_pulse            = 2.75[us]
Irr_atn_dec        = 22.16[dB]
Irr_atn_noe        = 22.16[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 2[s]
Recvr_gain         = 60
Relaxation_delay = 2[s]
Repetition_time    = 3.04333312[s]
Temp_get           = 26.3[dC]
```

Table 2, Entry 7: ^{1}H NMR of *para*-hydroxy benzonitrile



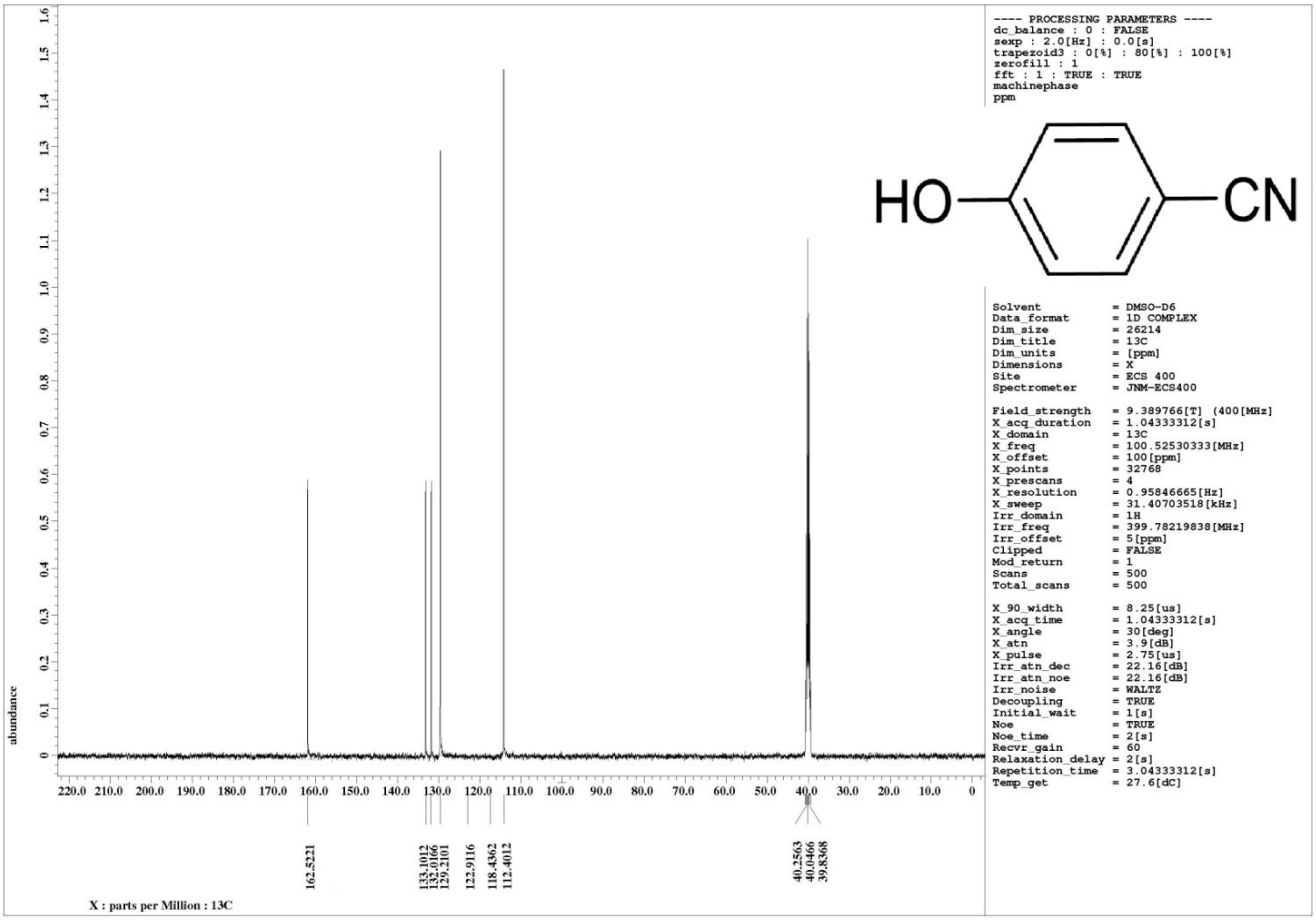
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Solvent            = DMSO-D6
Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.389766[T] (400[MHz]
X_acq_duration     = 2.18365952[s]
X_domain           = 1H
X_freq             = 399.78219838[MHz]
X_offset           = 5[ppm]
X_points           = 16384
X_prescans         = 1
X_resolution       = 0.45794685[Hz]
X_sweep            = 7.5030012[kHz]
Irr_domain         = 1H
Irr_freq           = 399.78219838[MHz]
Irr_offset         = 5[ppm]
Tri_domain         = 1H
Tri_freq           = 399.78219838[MHz]
Tri_offset         = 5[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 8
Total_scans        = 8

X_90_width         = 9.5[us]
X_acq_time         = 2.18365952[s]
X_angle            = 45[deg]
X_atn              = 0.5[dB]
X_pulse            = 4.75[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 38
Relaxation_delay = 5[s]
Repetition_time    = 7.18365952[s]
Temp_get           = 27.4[dC]
```

Table 2, Entry 7: ^{13}C NMR of *para*-hydroxy benzonitrile



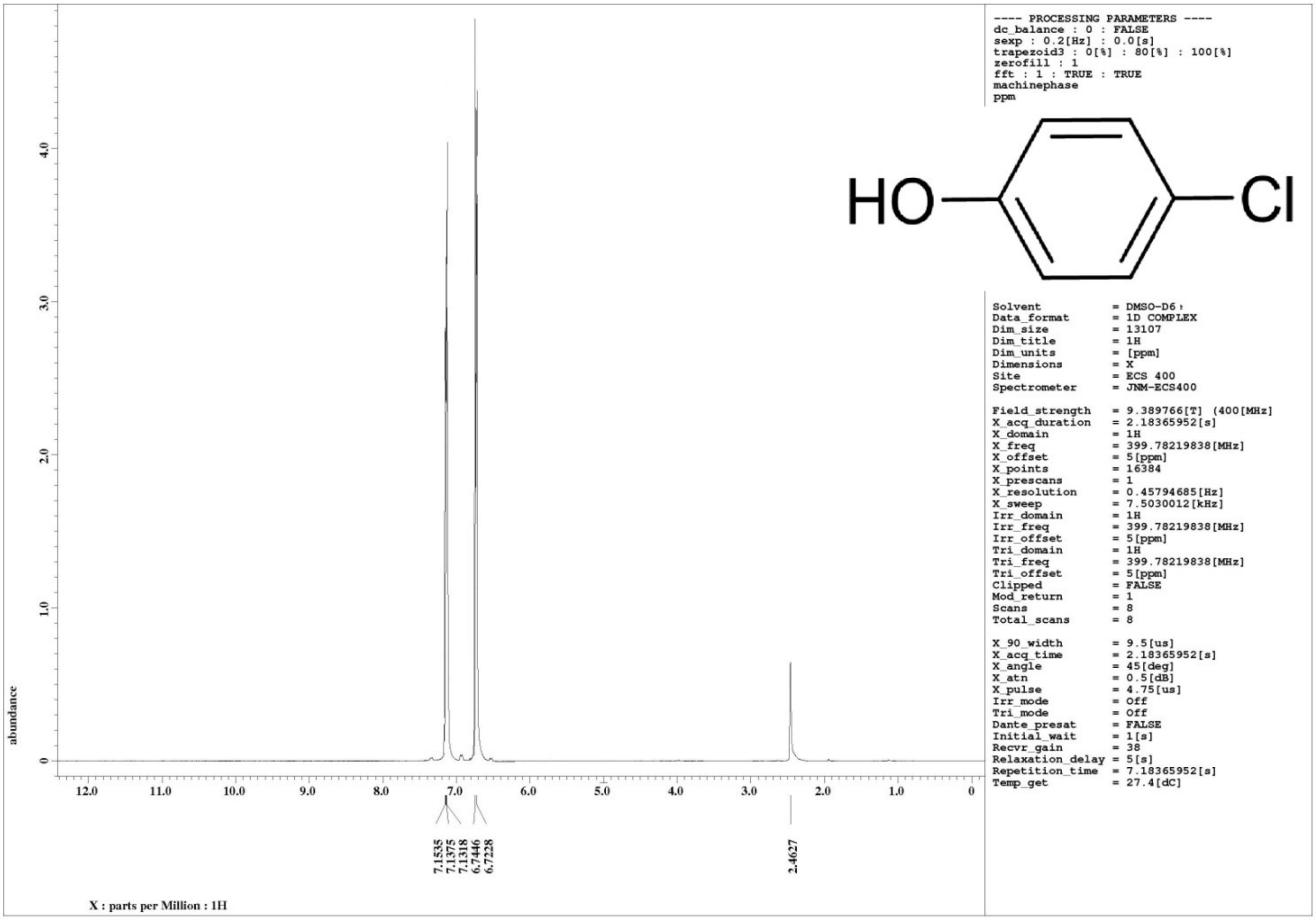
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Solvent            = DMSO-D6
Data_format        = 1D COMPLEX
Dim_size           = 26214
Dim_title          = 13C
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.389766[T] (400[MHz]
X_acq_duration     = 1.04333312[s]
X_domain           = 13C
X_freq             = 100.52530333[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 0.95846665[Hz]
X_sweep            = 31.40703518[kHz]
Irr_domain         = 1H
Irr_freq           = 399.78219838[MHz]
Irr_offset         = 5[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 500
Total_scans        = 500

X_90_width         = 8.25[us]
X_acq_time         = 1.04333312[s]
X_angle            = 30[deg]
X_atn              = 3.9[dB]
X_pulse            = 2.75[us]
Irr_atn_dec        = 22.16[dB]
Irr_atn_noe        = 22.16[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 2[s]
Recvr_gain         = 60
Relaxation_delay = 2[s]
Repetition_time    = 3.04333312[s]
Temp_get           = 27.6[dC]
```

Table 2, Entry 8: ^{1}H NMR of *para*-chloro phenol



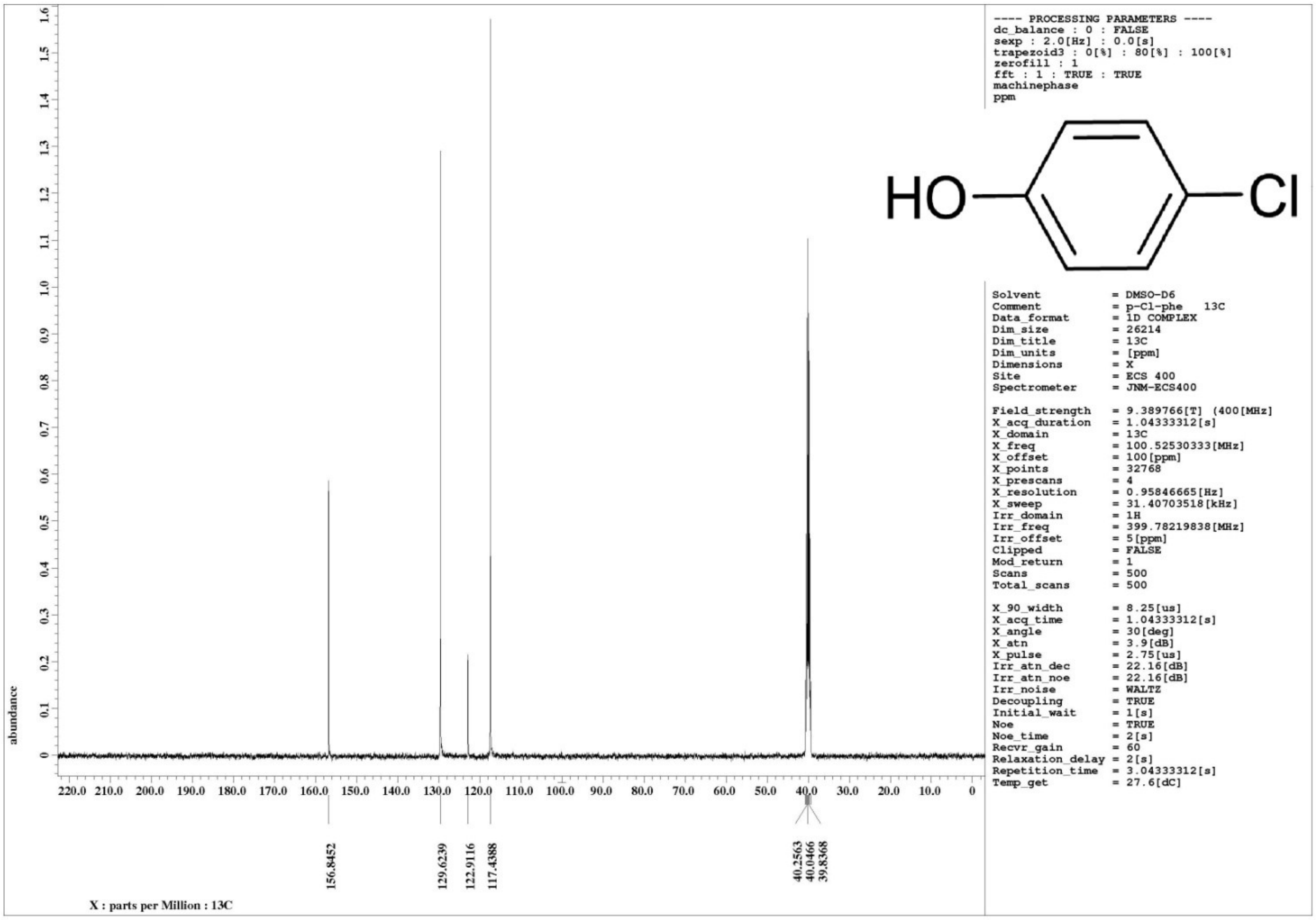
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Solvent            = DMSO-D6
Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.389766[T] (400[MHz]
X_acq_duration     = 2.18365952[s]
X_domain           = 1H
X_freq             = 399.78219838[MHz]
X_offset           = 5[ppm]
X_points           = 16384
X_prescans         = 1
X_resolution       = 0.45794685[Hz]
X_sweep            = 7.5030012[kHz]
Irr_domain         = 1H
Irr_freq           = 399.78219838[MHz]
Irr_offset         = 5[ppm]
Tri_domain         = 1H
Tri_freq           = 399.78219838[MHz]
Tri_offset         = 5[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 8
Total_scans        = 8

X_90_width         = 9.5[us]
X_acq_time         = 2.18365952[s]
X_angle            = 45[deg]
X_atn              = 0.5[dB]
X_pulse            = 4.75[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 38
Relaxation_delay   = 5[s]
Repetition_time    = 7.18365952[s]
Temp_get           = 27.4[dC]
```

Table 2, Entry 8: ^{13}C NMR of *para*-chloro phenol



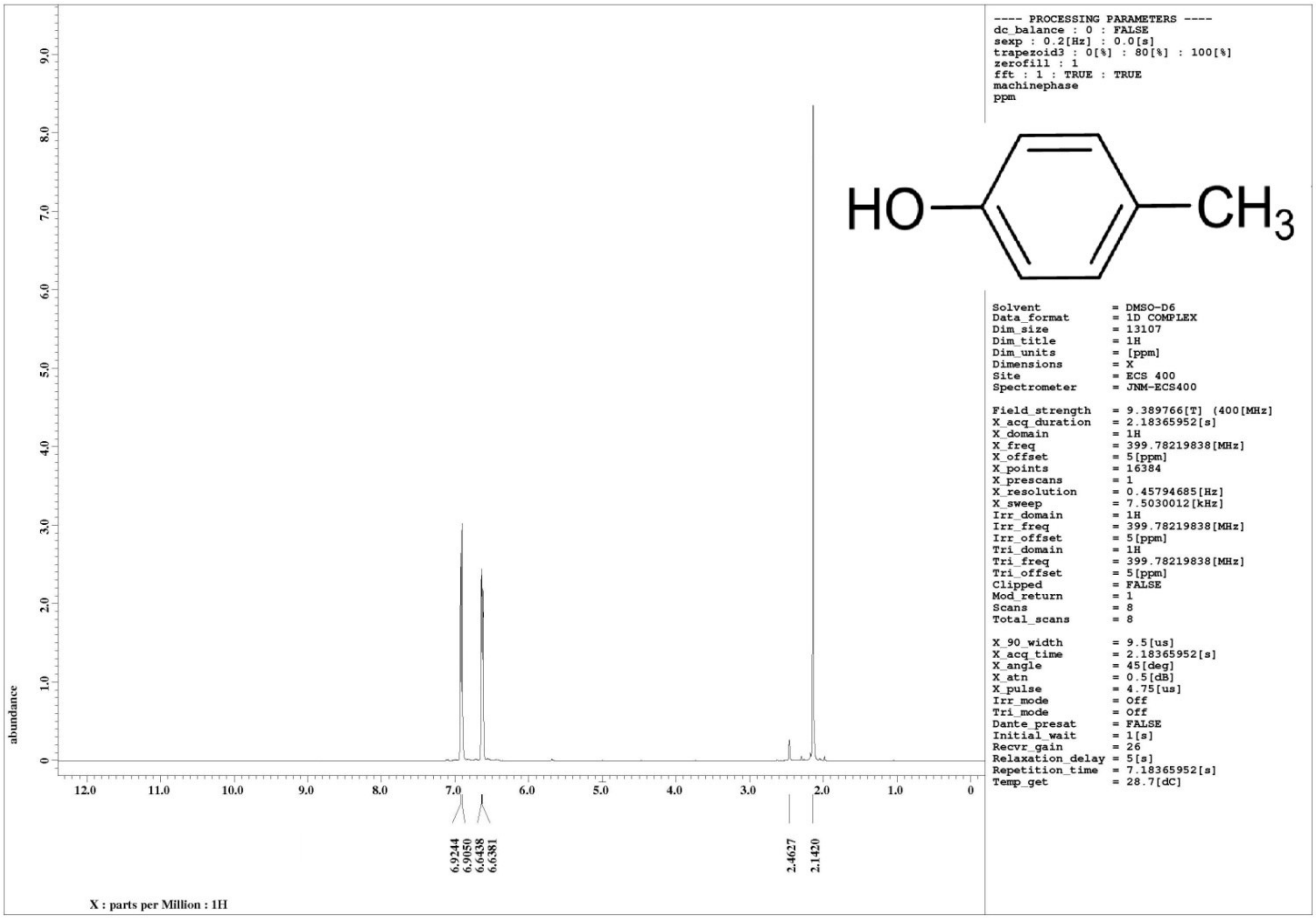
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Solvent            = DMSO-D6
Comment            = p-Cl-phe   13C
Data_format        = 1D COMPLEX
Dim_size           = 26214
Dim_title          = 13C
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.389766[T] (400[MHz]
X_acq_duration     = 1.04333312[s]
X_domain           = 13C
X_freq             = 100.52530333[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 0.95846665[Hz]
X_sweep            = 31.40703518[kHz]
Irr_domain         = 1H
Irr_freq           = 399.78219838[MHz]
Irr_offset         = 5[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 500
Total_scans        = 500

X_90_width         = 8.25[us]
X_acq_time         = 1.04333312[s]
X_angle            = 30[deg]
X_atn              = 3.9[dB]
X_pulse            = 2.75[us]
Irr_atn_dec        = 22.16[dB]
Irr_atn_noe        = 22.16[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 2[s]
Recvr_gain         = 60
Relaxation_delay   = 2[s]
Repetition_time    = 3.04333312[s]
Temp_get           = 27.6[dC]
```

Table 2, Entry 9: ^{1}H NMR of *para*-cresol



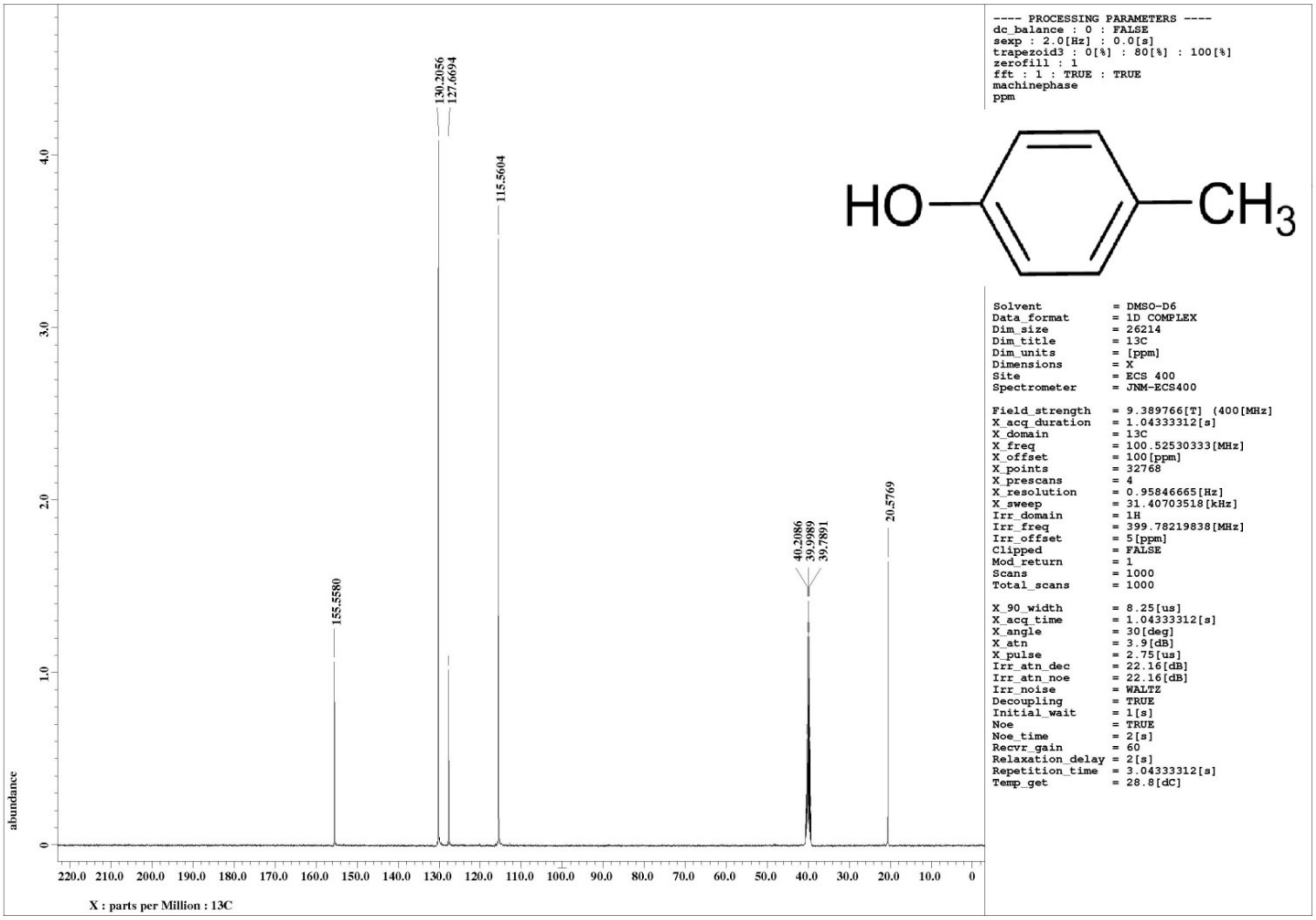
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Solvent            = DMSO-D6
Data_format        = 1D COMPLEX
Dim_size           = 13107
Dim_title          = 1H
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.389766[T] (400[MHz]
X_acq_duration     = 2.18365952[s]
X_domain           = 1H
X_freq             = 399.78219838[MHz]
X_offset           = 5[ppm]
X_points           = 16384
X_prescans         = 1
X_resolution       = 0.45794685[Hz]
X_sweep            = 7.5030012[kHz]
Irr_domain         = 1H
Irr_freq           = 399.78219838[MHz]
Irr_offset         = 5[ppm]
Tri_domain         = 1H
Tri_freq           = 399.78219838[MHz]
Tri_offset         = 5[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 8
Total_scans        = 8

X_90_width         = 9.5[us]
X_acq_time         = 2.18365952[s]
X_angle            = 45[deg]
X_atn              = 0.5[dB]
X_pulse            = 4.75[us]
Irr_mode           = Off
Tri_mode           = Off
Dante_presat       = FALSE
Initial_wait       = 1[s]
Recvr_gain         = 26
Relaxation_delay = 5[s]
Repetition_time    = 7.18365952[s]
Temp_get           = 28.7[dC]
```

Table 2, Entry 9: ^{13}C NMR of *para*-cresol



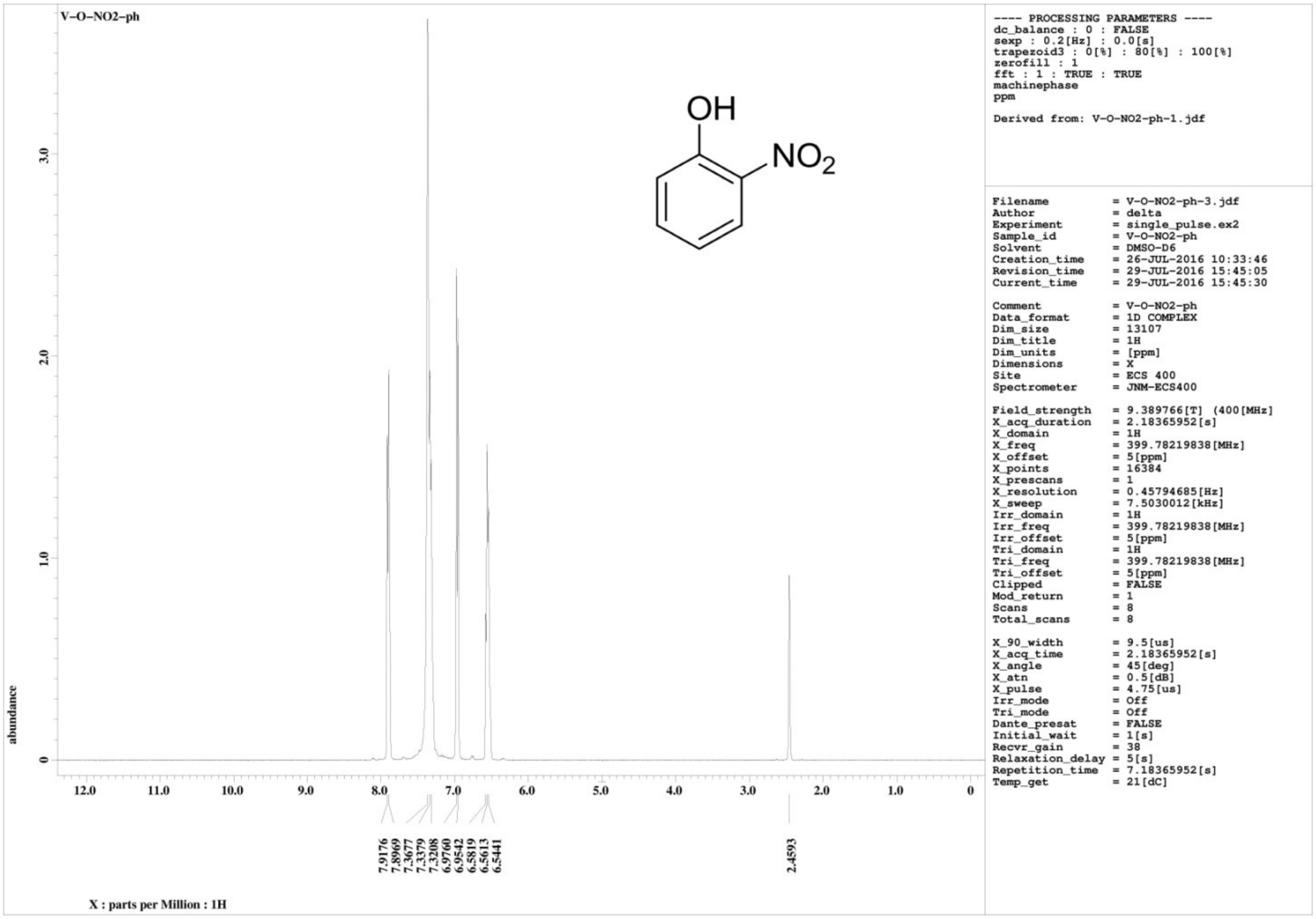
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Solvent            = DMSO-D6
Data_format        = 1D COMPLEX
Dim_size           = 26214
Dim_title          = 13C
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.389766[T] (400[MHz]
X_acq_duration     = 1.04333312[s]
X_domain           = 13C
X_freq             = 100.52530333[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 0.95846665[Hz]
X_sweep            = 31.40703518[kHz]
Irr_domain         = 1H
Irr_freq           = 399.78219838[MHz]
Irr_offset         = 5[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 1000
Total_scans        = 1000

X_90_width         = 8.25[us]
X_acq_time         = 1.04333312[s]
X_angle            = 30[deg]
X_atn              = 3.9[dB]
X_pulse            = 2.75[us]
Irr_atn_dec        = 22.16[dB]
Irr_atn_noe        = 22.16[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 2[s]
Recvr_gain         = 60
Relaxation_delay   = 2[s]
Repetition_time    = 3.04333312[s]
Temp_get           = 28.8[dC]
```

Table 3, Entry 3: ^{1}H NMR of *ortho*-nitro phenol



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: V-O-NO2-ph-1.jdf
```

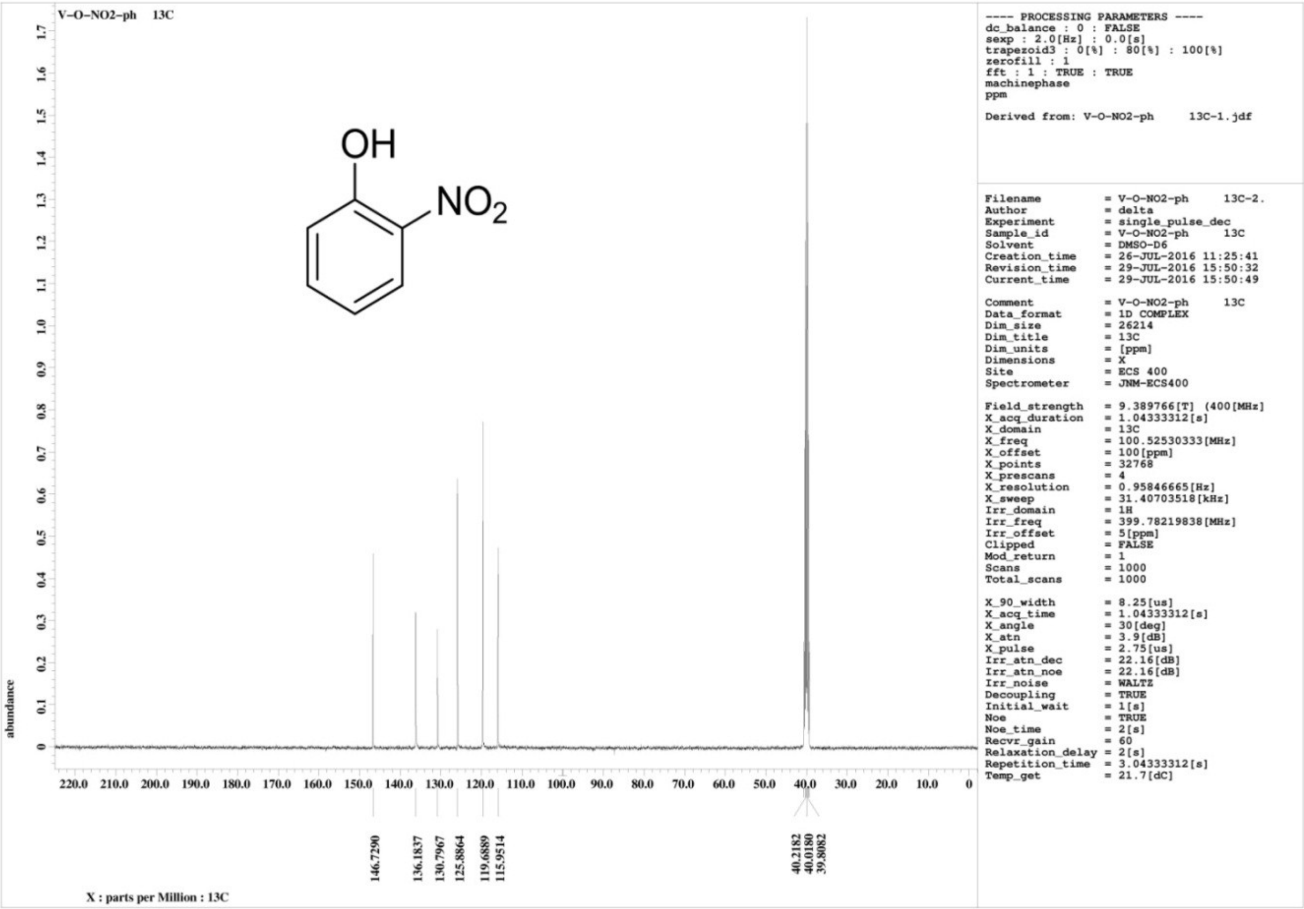
```
Filename          = V-O-NO2-ph-3.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = V-O-NO2-ph
Solvent           = DMSO-D6
Creation_time     = 26-JUL-2016 10:33:46
Revision_time     = 29-JUL-2016 15:45:05
Current_time      = 29-JUL-2016 15:45:30

Comment           = V-O-NO2-ph
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9.5[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 0.5[dB]
X_pulse           = 4.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 21[dC]
```

Table 3, Entry 3: ^{13}C NMR of *ortho*-nitro phenol



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: V-O-NO2-ph     13C-1.jdf

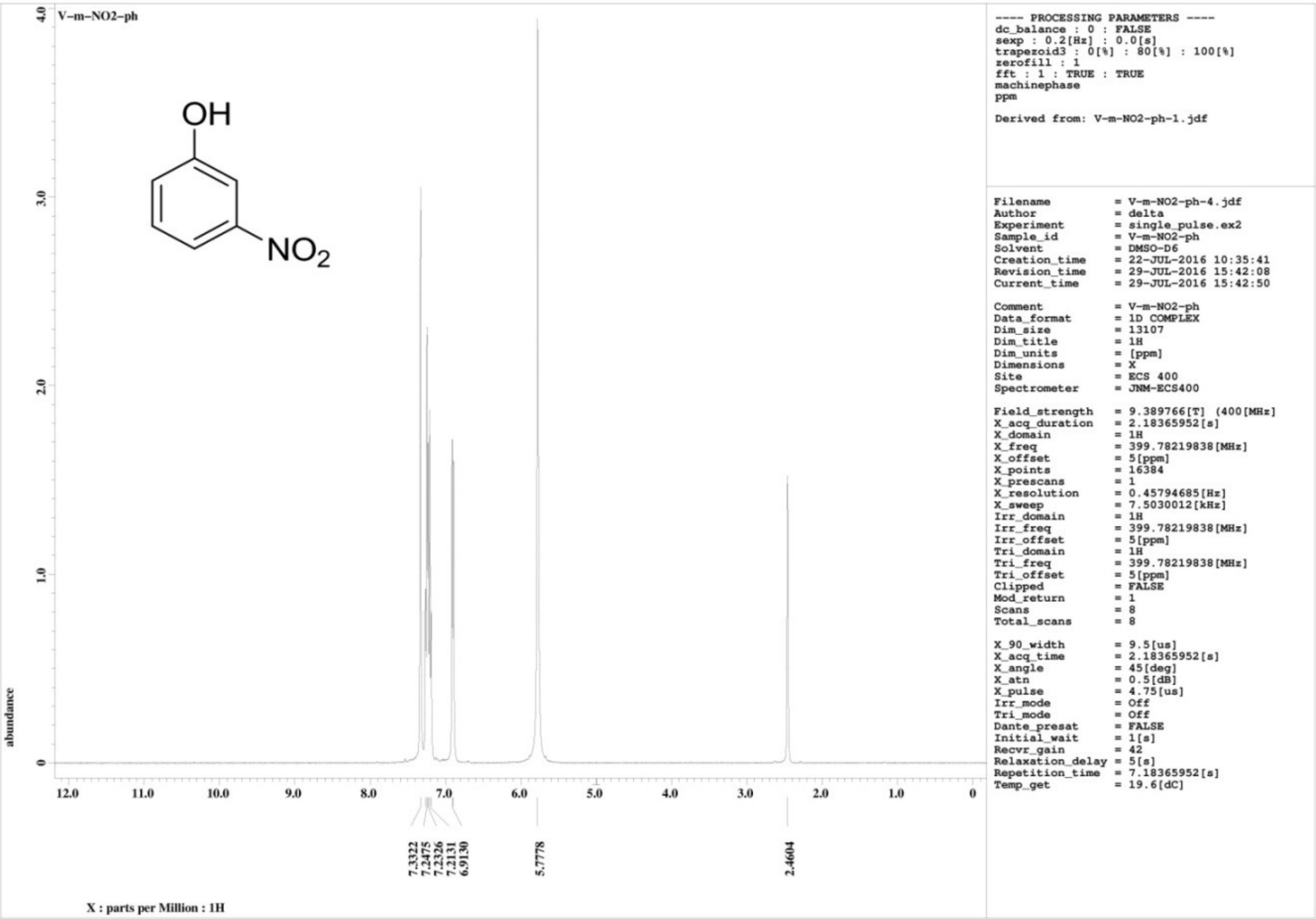
Filename           = V-O-NO2-ph     13C-2.
Author             = delta
Experiment         = single_pulse_dec
Sample_id          = V-O-NO2-ph     13C
Solvent            = DMSO-D6
Creation_time      = 26-JUL-2016 11:25:41
Revision_time      = 29-JUL-2016 15:50:32
Current_time       = 29-JUL-2016 15:50:49

Comment            = V-O-NO2-ph     13C
Data_format        = 1D COMPLEX
Dim_size           = 26214
Dim_title          = 13C
Dim_units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = JNM-ECS400

Field_strength     = 9.389766[T] (400[MHz]
X_acq_duration     = 1.04333312[s]
X_domain           = 13C
X_freq             = 100.52530333[MHz]
X_offset           = 100[ppm]
X_points           = 32768
X_prescans         = 4
X_resolution       = 0.95846665[Hz]
X_sweep            = 31.40703518[kHz]
Irr_domain         = 1H
Irr_freq           = 399.78219838[MHz]
Irr_offset         = 5[ppm]
Clipped            = FALSE
Mod_return         = 1
Scans              = 1000
Total_scans        = 1000

X_90_width         = 8.25[us]
X_acq_time         = 1.04333312[s]
X_angle            = 30[deg]
X_atn              = 3.9[dB]
X_pulse            = 2.75[us]
Irr_atn_dec        = 22.16[dB]
Irr_atn_noe        = 22.16[dB]
Irr_noise          = WALTZ
Decoupling         = TRUE
Initial_wait       = 1[s]
Noe                = TRUE
Noe_time           = 2[s]
Recvr_gain         = 60
Relaxation_delay   = 2[s]
Repetition_time    = 3.04333312[s]
Temp_get           = 21.7[dC]
```

Table 3, Entry 3: ^{1}H NMR of *meta*-nitro phenol



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: V-m-NO2-ph-1.jdf
```

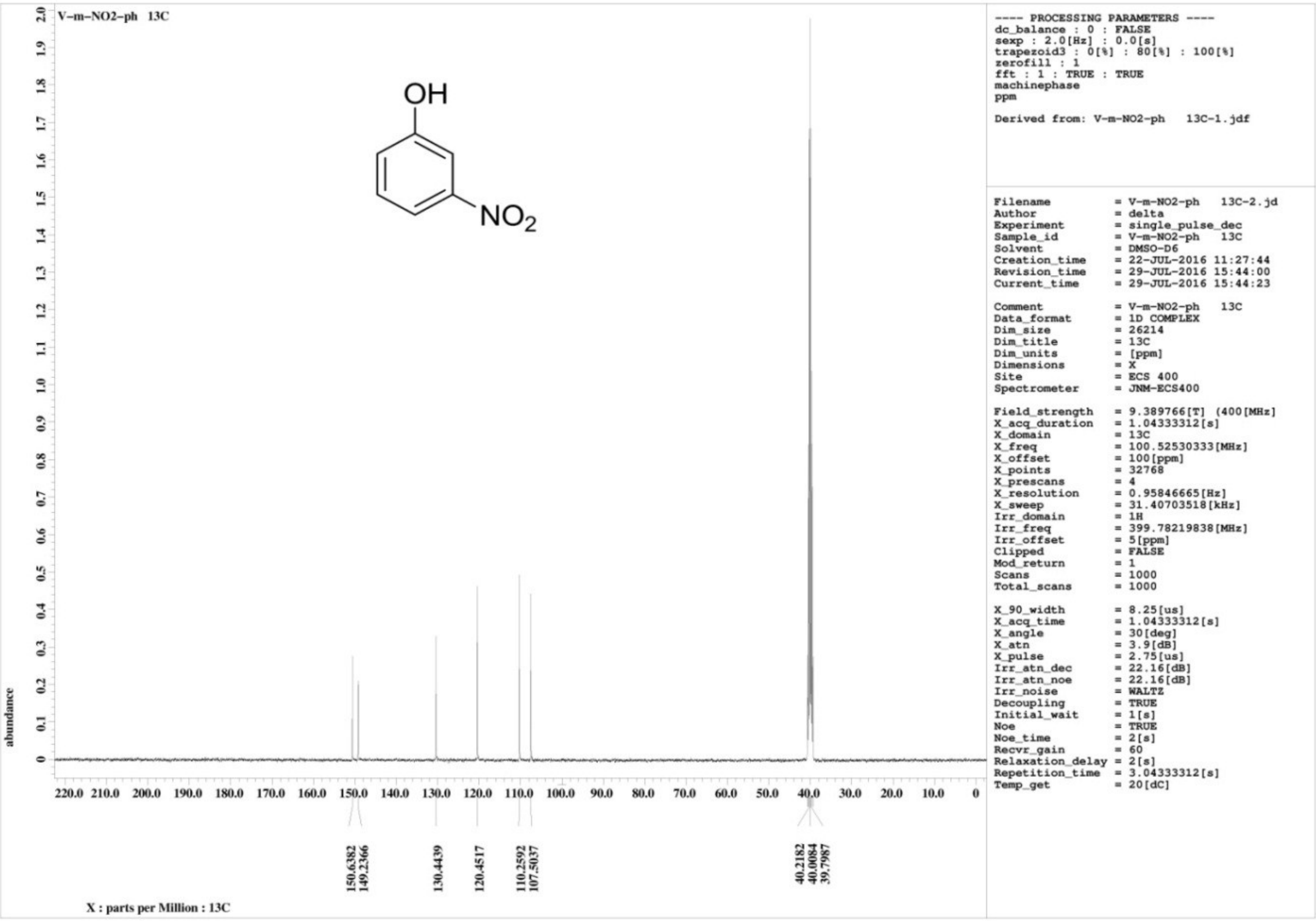
```
Filename          = V-m-NO2-ph-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = V-m-NO2-ph
Solvent           = DMSO-D6
Creation_time     = 22-JUL-2016 10:35:41
Revision_time     = 29-JUL-2016 15:42:08
Current_time      = 29-JUL-2016 15:42:50

Comment           = V-m-NO2-ph
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9.5[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 0.5[dB]
X_pulse           = 4.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 42
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 19.6[dC]
```

Table 3, Entry 3: ^{13}C NMR of *meta*-nitro phenol



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: V-m-NO2-ph   13C-1.jdf

Filename          = V-m-NO2-ph   13C-2.jd
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = V-m-NO2-ph   13C
Solvent           = DMSO-D6
Creation_time     = 22-JUL-2016 11:27:44
Revision_time     = 29-JUL-2016 15:44:00
Current_time      = 29-JUL-2016 15:44:23

Comment           = V-m-NO2-ph   13C
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 8.25[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 3.9[dB]
X_pulse           = 2.75[us]
Irr_atn_dec       = 22.16[dB]
Irr_atn_noe       = 22.16[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.04333312[s]
Temp_get          = 20[dC]
```

(9) Reference

1. Gogoi, S.; Kumar, M.; Mandal, B. B.; Karak, N. *Comp. Sci. Technol.* **2015**, *118*, 39.