



Title	Rhenium-Loaded TiO ₂ : A Highly Versatile and Chemoselective Catalyst for the Hydrogenation of Carboxylic Acid Derivatives and the N-Methylation of Amines Using H ₂ and CO ₂
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SUPPORTING INFORMATION

Rhenium-Loaded TiO₂: A Highly Versatile and Chemoselective Catalyst for the Hydrogenation of Carboxylic Acid Derivatives and the *N*-Methylation of Amines Using H₂ and CO₂

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1. Experimental (Detailed procedure for catalytic reactions)

Typical procedure for the hydrogenation of esters:

After the reduction with H_2 at $T = 700\text{ }^\circ\text{C}$ (cf. Catalyst Preparation), the catalyst (2 mol% with respect to the Re loading) and a mixture of 3-phenylpropionic acid methyl ester (1.0 mmol), octane (3 mL), and *n*-dodecane (0.29 mmol) were added to a stainless steel autoclave (10 cm^3). The resulting mixture was magnetically stirred ($T = 180\text{ }^\circ\text{C}$, $p_{\text{H}_2} = 5\text{ MPa}$) for 24 h. The products were analyzed by GCMS (SHIMADZU GCMS-QP2010 with an Ultra ALLOY capillary column UA+-1; Frontier Laboratories Ltd.) and GC (Shimadzu GC-14B with an Ultra ALLOY capillary column UA+-1; Frontier Laboratories Ltd.). Yields were determined by GC analysis using *n*-dodecane as the internal standard. All products were subjected to column chromatography on silica gel 60 (spherical, 63-210 μm , Kanto Chemical Co. Ltd.; eluent: hexane/ethyl acetate = 90/10, v/v), and subsequently analyzed by GC and GCMS. ^1H and ^{13}C NMR analysis was carried out for those products that were isolated successfully. ^1H and ^{13}C NMR spectra were recorded at ambient temperature on a JEOL-ECX 600 spectrometer (^1H : 600.17 MHz, ^{13}C : 150.92 MHz), using tetramethylsilane as the internal standard. Isolated yields were determined relative to the esters.

Typical procedure for the hydrogenation of amides:

After the reduction with H_2 at $T = 700\text{ }^\circ\text{C}$, the catalyst (2 mol% with respect to the Re loading) and a mixture of *N*-benzylacetamide (1.0 mmol), octane (3 mL), and *n*-dodecane (0.29 mmol) were added to a stainless steel autoclave (10 cm^3). The resulting mixture was magnetically stirred ($T = 180\text{ }^\circ\text{C}$, $p_{\text{H}_2} = 5\text{ MPa}$) for 24 h. The products were analyzed by GCMS and GC, and the product yields were determined by GC analysis using *n*-dodecane as the internal standard. *N*-ethylbenzylamine as a product was isolated by column chromatography on silica gel 60, and subsequently analyzed by GC, GCMS, as well as by ^1H and ^{13}C NMR spectroscopy. Isolated yields were determined relative to the starting amide.

One-pot *N*-alkylation of amines with carboxylic acids or esters:

After the reduction with H_2 at $T = 700\text{ }^\circ\text{C}$, the catalyst (2 mol% with respect to the Re loading) and a mixture of 3-phenylpropionic acid (1.0 mmol) or 3-phenylpropionic acid methyl ester (1.0 mmol), dimethylamine (3 mmol), octane (3 mL), and *n*-dodecane (0.29 mmol) were added to a stainless steel autoclave (10 cm^3). The resulting mixture was magnetically stirred ($T = 200\text{ }^\circ\text{C}$, $p_{\text{H}_2} = 5\text{ MPa}$) for 48 h. 3-phenylpropyl-dimethylamine as the product was isolated by column chromatography on silica gel 60, and subsequently analyzed by GC, GCMS, as well as by ^1H and ^{13}C NMR spectroscopy. Isolated yields were determined relative to the starting carboxylic acid or ester.

Typical procedure for the *N*-methylation of amines with CO_2 and H_2 :

After the reduction with H_2 at $T = 700\text{ }^\circ\text{C}$, the catalyst (2 mol% with respect to the Re loading) and a mixture of *N*-methylaniline (1.0 mmol), octane (3 mL), and *n*-dodecane (0.29 mmol) were added to a stainless steel autoclave (10 cm^3). The resulting mixture was magnetically stirred ($T = 200\text{ }^\circ\text{C}$, $p_{\text{CO}_2} = 1\text{ MPa}$, $p_{\text{H}_2} = 5\text{ MPa}$) for 24 h. Products were analyzed by GCMS and GC, and the product yields were determined by GC analysis using *n*-dodecane as the internal standard. *N,N*-dimethylaniline as a product was isolated by column chromatography on silica

gel 60, and subsequently analyzed by GC, GCMS, as well as by ^1H and ^{13}C NMR spectroscopy. Isolated yields were determined relative to the starting amide.

2. Supplementary results

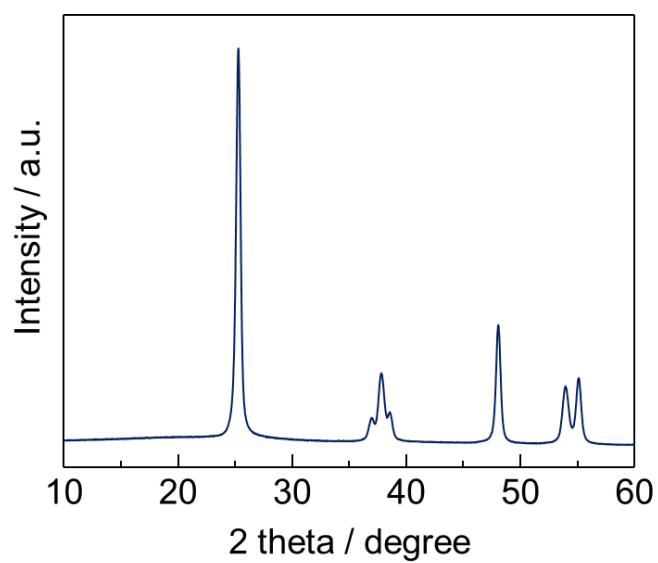


Figure S1. Powder XRD pattern for Re/TiO₂.

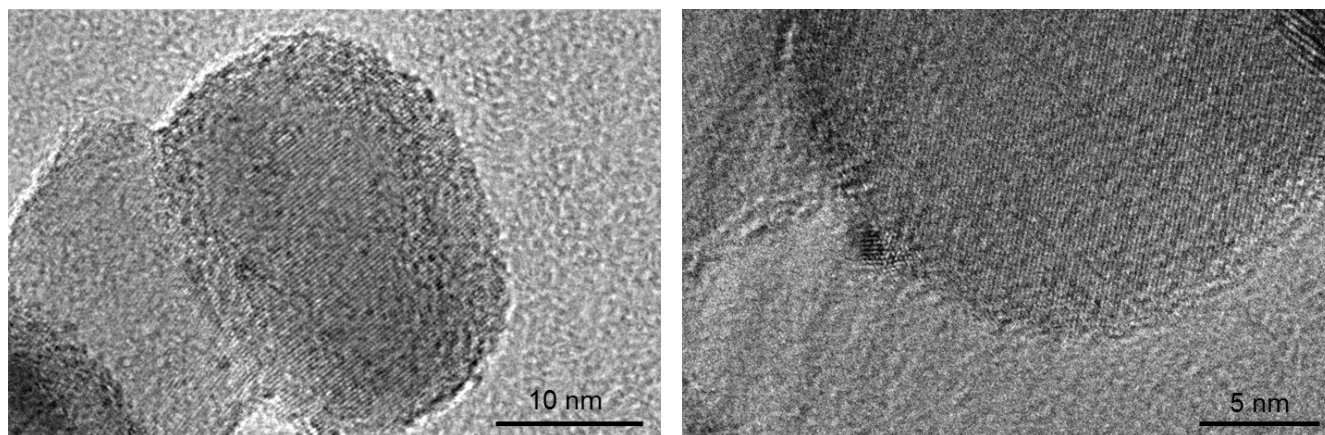


Figure S2. TEM images of Re/TiO₂.

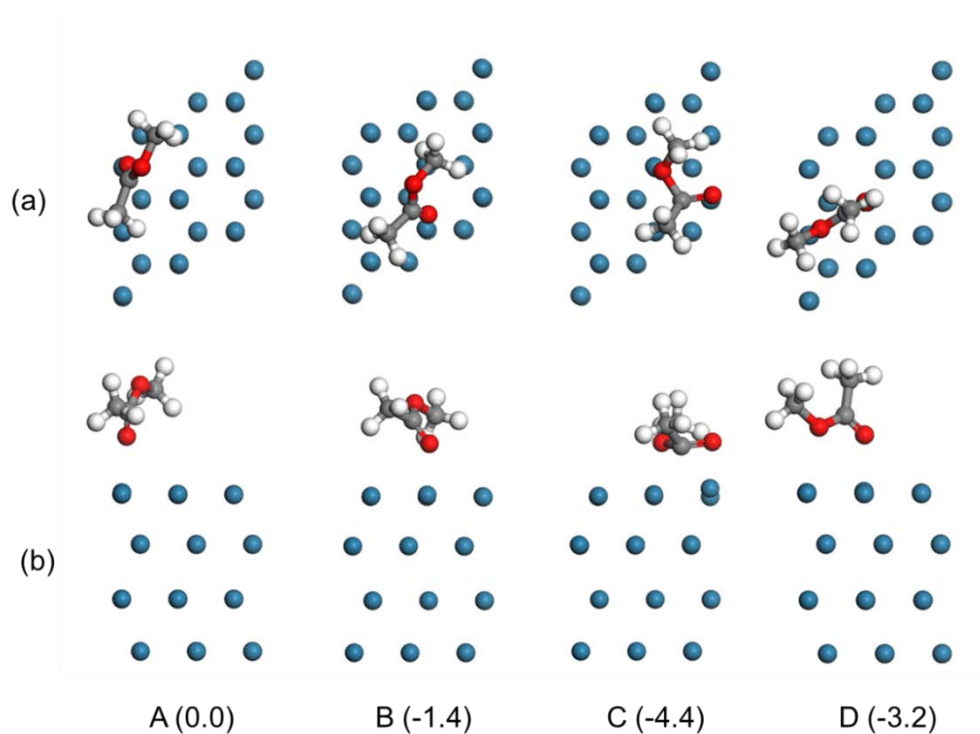


Figure S3. Possible adsorption modes for ethyl acetate on the Re(0001) surface (A-D): (a) top view and (b) side view. Color code: gray = C; red = O; white = H; blue = metal atoms. Relative energies in kcal/mol are given in parenthesis. Structure A represents the most stable adsorption mode.

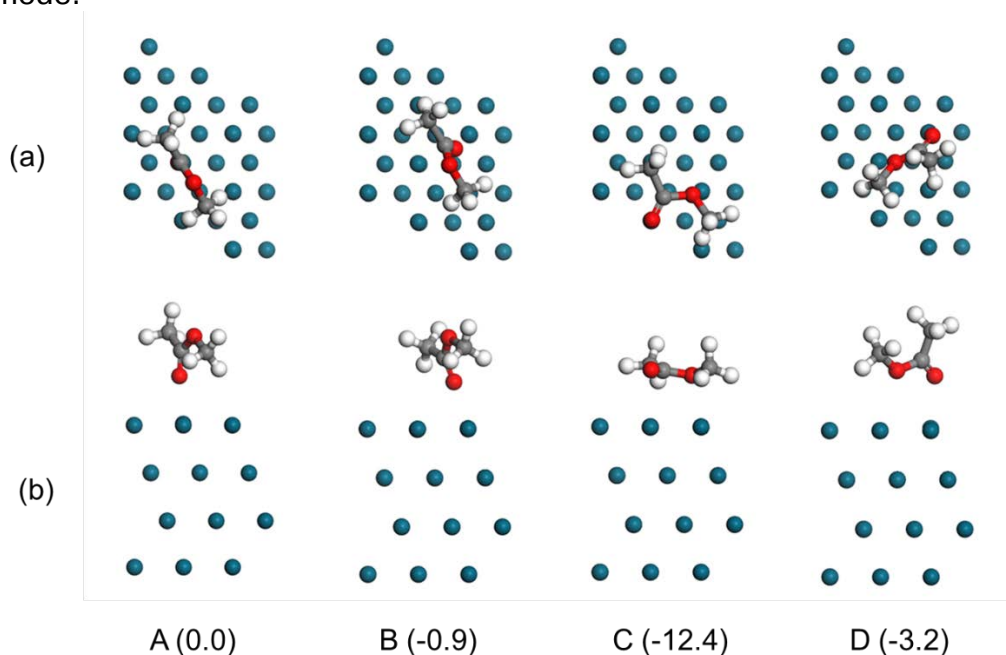


Figure S4. Possible adsorption modes of ethyl acetate on the Pd(111) surface (A-D): (a) top view and (b) side view. Color code: gray = C; red = O; white = H; blue = metal atoms. Relative energies in kcal/mol are given in parenthesis. Structure A represents the most stable adsorption mode.

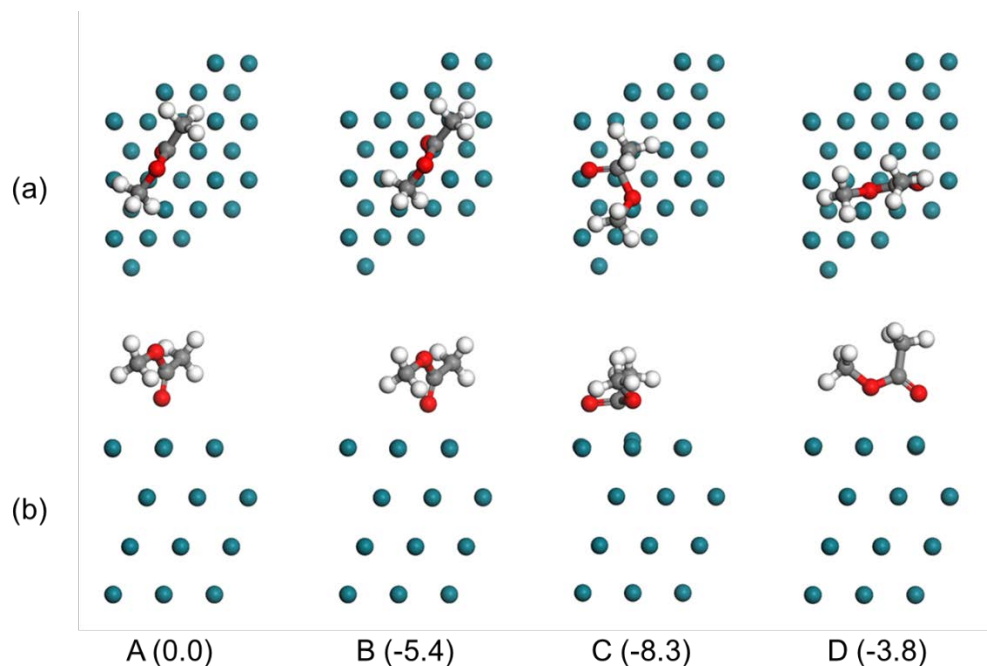
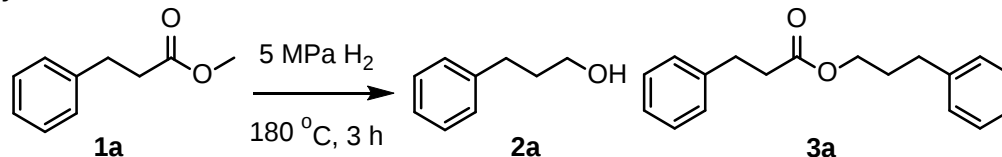


Figure S5. Possible adsorption modes of ethyl acetate on the Rh(111) surface (A-D): (a) top view and (b) side view. Color code: gray = C; red = O; white = H; blue = metal atoms. Relative energies in kcal/mol are given in parenthesis. Structure A represents the most stable adsorption mode.

Table S1. Total Mulliken charges of methyl acetate and benzene on metal surfaces

Metal surface	Methyl acetate	Benzene
Re(0001)	-0.03	-1.22
Pd(111)	-0.55	-1.35
Rh(111)	-0.20	-1.46

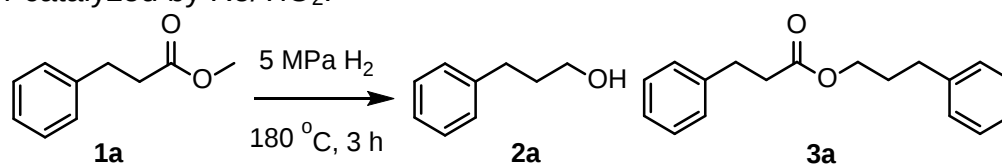
Table S2. Effect of Re loading on the hydrogenation of 3-phenylpropionic acid methyl ester catalyzed by Re/TiO₂.^a



Re loading [wt%]	Conv. [%]	Yield [%]	
		2a	3a
0	0	0	0
1	26	12	14
3	43	32	9
5	50	43	7
10	19	18	1

^aReaction conditions: 2 mol% Re, 1 mmol phenylpropionic acid methyl ester, 3 mL octane, p_{H_2} = 5 MPa, T = 180 °C, t = 3 h. Yields were determined by GC using *n*-dodecane as the internal standard.

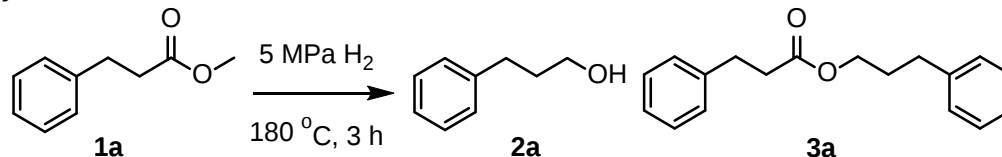
Table S3. Effect of the reduction temperature on the hydrogenation of 3-phenylpropionic acid methyl ester catalyzed by Re/TiO₂.^a



Reduction temperature [°C]	Conv. [%]	Yield [%]	
		2a	3a
r.t.	20	17	1
100	25	21	3
200	31	25	6
300	41	35	5
400	55	46	8
500	58	49	9
600	55	46	8
700	50	43	7

^aReaction conditions: 2 mol% Re, 1 mmol phenylpropionic acid methyl ester, 3 mL octane, p_{H_2} = 5 MPa, T = 180 °C, t = 3 h. Yields were determined by GC using *n*-dodecane as the internal standard.

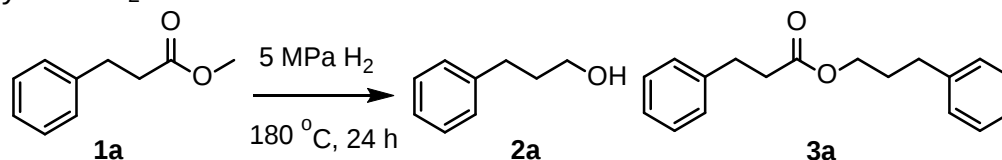
Table S4. Effect of H₂ pressure on the hydrogenation of 3-phenylpropionic acid methyl ester catalyzed by Re/TiO₂.^a



H ₂ pressure [MPa]	Conv. [%]	Yield [%]	
		2a	3a
0.1	1	1	0
1	12	0	1
2	20	18	1
3	32	25	6
4	34	31	2
5	50	43	7
6	47	45	1
7	45	44	1

^aReaction conditions: 2 mol% Re, 1 mmol phenylpropionic acid methyl ester, 3 mL octane, $T = 180\text{ }^{\circ}\text{C}$, $t = 3\text{ h}$. Yields were determined by GC using *n*-dodecane as the internal standard.

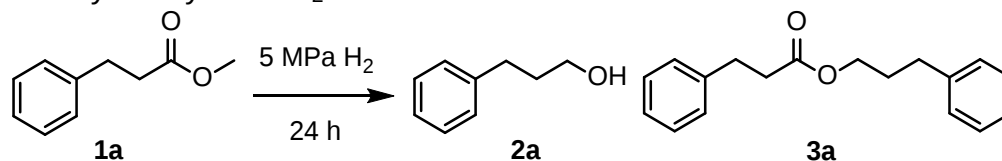
Table S5. Effect of the solvent on the hydrogenation of 3-phenylpropionic acid methyl ester catalyzed by Re/TiO₂.^a



Solvent	Conv. [%]	Yield [%]	
		2a	3a
None	100	96	4
Octane	99	96	3
Hexane	96	90	6
p-Xylene	99	97	2
o-Xylene	100	95	4
1,4-Dioxane	100	94	5
THF	99	96	2
DME	99	90	9
Ethanol	88	58	0
Diethylene glycol dimethyl ether	40	37	2

^aReaction conditions: 2 mol% Re, 1 mmol phenylpropionic acid methyl ester, 3 mL solvent, $p_{\text{H}_2} = 5\text{ MPa}$, $T = 180\text{ }^{\circ}\text{C}$, $t = 24\text{ h}$. Yields were determined by GC using *n*-dodecane as the internal standard.

Table S6. Effect of the reaction temperature on the hydrogenation of 3-phenylpropionic acid methyl ester catalyzed by Re/TiO₂.^a



Temperature [°C]	Conv. [%]	Yield [%]	
		2a	3a
180	99	96	3
170	93	84	7
160	88	68	18
140	41	34	7

^aReaction conditions: 2 mol% Re, 1 mmol phenylpropionic acid methyl ester, 3 mL octane, p_{H_2} = 5 MPa, t = 24 h. Yields were determined by GC using *n*-dodecane as the internal standard.

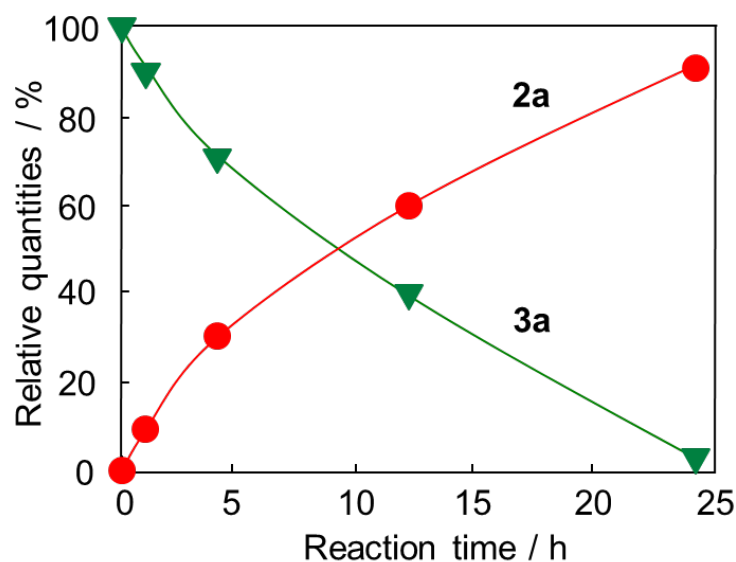


Figure S6. Time-course plot for the hydrogenation of 3-phenylpropyl 3-phenyl-propionate (**3a**) over Re/TiO₂ at T = 180 °C and p_{H_2} = 5 MPa. **2a**: 3-phenylpropanol; **3a**: 3-phenylpropyl 3-phenyl-propionate.

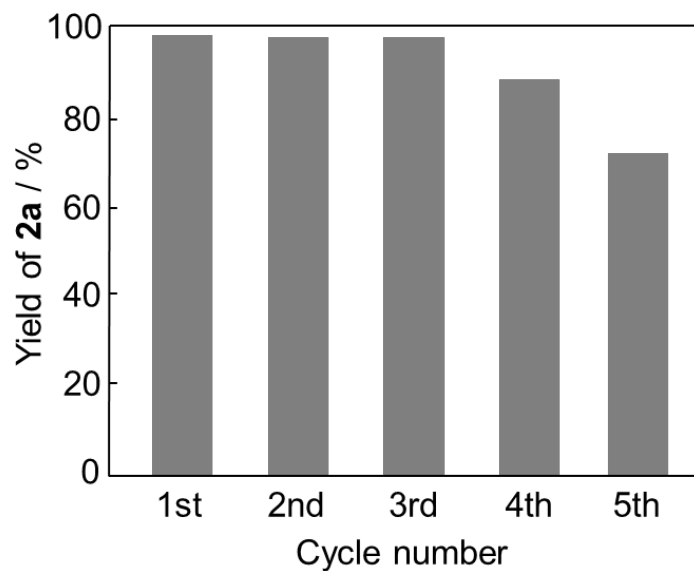


Figure S7. Recycling study for hydrogenation of 3-phenylpropionic acid methyl ester catalyzed by Re/TiO₂ at $T = 180\text{ }^{\circ}\text{C}$ and $p_{\text{H}_2} = 5\text{ MPa}$.

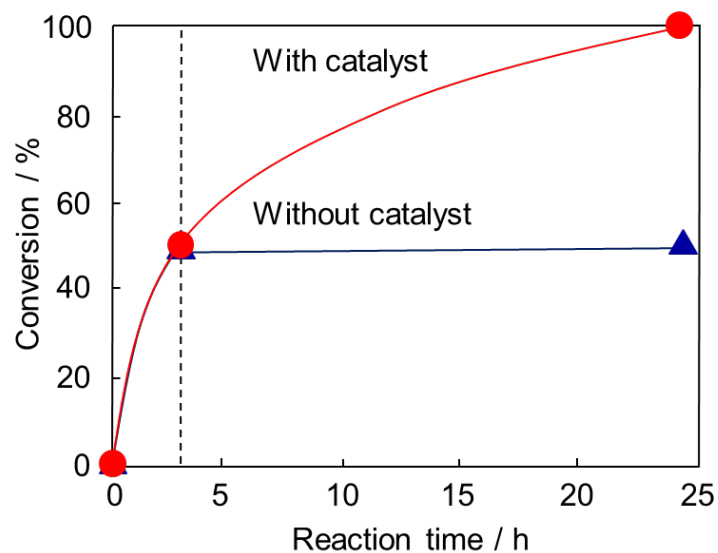
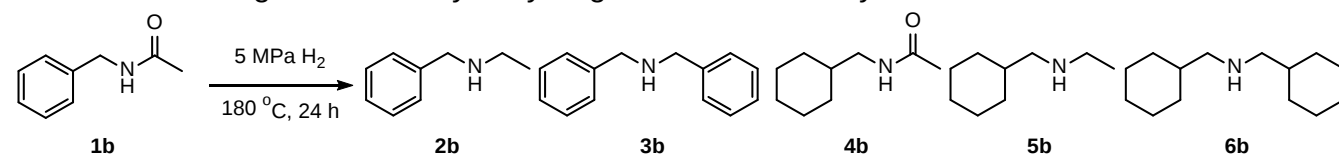


Figure S8. Leaching test for the hydrogenation of 3-phenylpropionic acid methyl ester over Re/TiO₂ at $T = 180\text{ }^{\circ}\text{C}$ and $p_{\text{H}_2} = 5\text{ MPa}$. After $t = 3\text{ h}$, the catalyst was isolated by filtration. Subsequently, the reaction mixture was kept under reaction conditions in the absence of a solid catalyst.

Table S7. Heterogeneous catalytic hydrogenation of *N*-benzylacetamide.



Entry	Catalysts	Conv. [%]	Yield [%]				
			2b	3b	4b	5b	6b
1	Re/TiO ₂	99	86	3	0	0	0
2	Ru/TiO ₂	100	0	0	2	51	35
3	Pt/TiO ₂	98	0	0	2	42	31
4	Ir/TiO ₂	100	0	0	17	35	12
5	Rh/TiO ₂	100	0	0	15	64	12
6	Pd/TiO ₂	100	0	0	92	0	0
7	Re/Carbon	76	60	3	0	0	0
8	Re/ θ -Al ₂ O ₃	31	27	0	0	0	0
9	Re/ γ -Al ₂ O ₃	25	23	0	0	0	0

^aReaction conditions: 2 mol% catalyst, 1 mmol phenylpropionic acid methyl ester, 3 mL octane, p_{H_2} = 5 MPa, T = 180 °C, t = 24 h. Yields were determined by GC using *n*-dodecane as the internal standard.

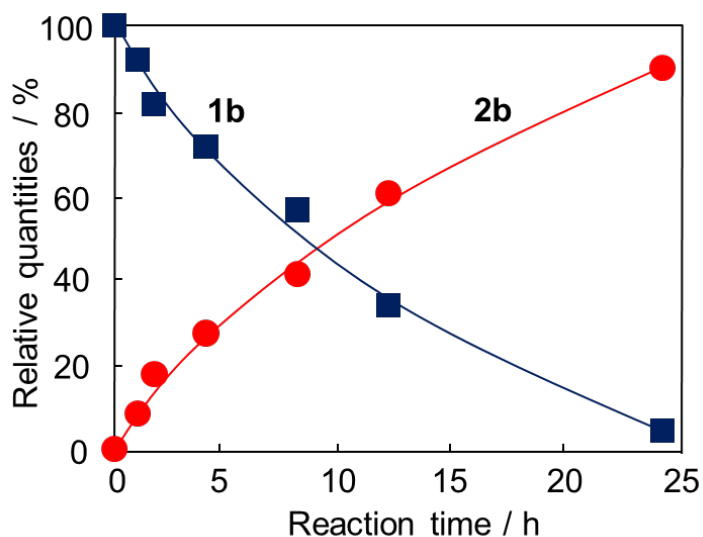
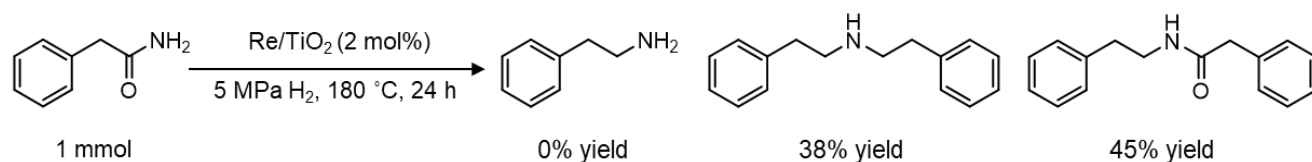


Figure S9. Time-course plot for the hydrogenation of *N*-benzylacetamide catalyzed by Re/TiO₂; **1b**: *N*-benzylacetamide; **2b**: *N*-ethylbenzylamine.



Scheme S1. Hydrogenation of 2-phenylacetamide ($p_{\text{H}_2} = 5\text{ MPa}$ and $T = 180\text{ }^\circ\text{C}$) catalyzed by Re/TiO₂.

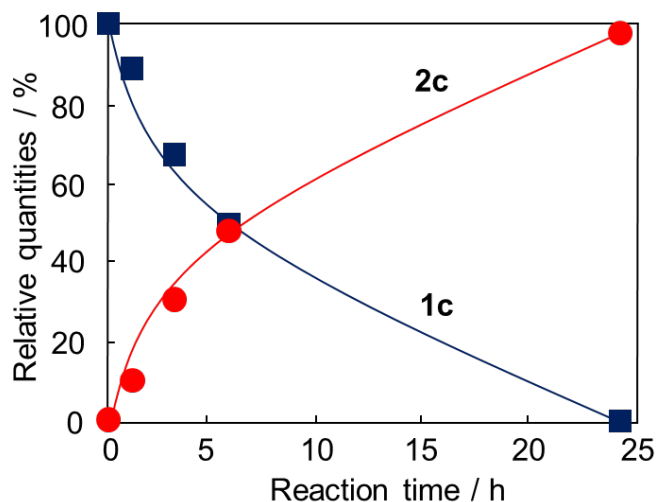
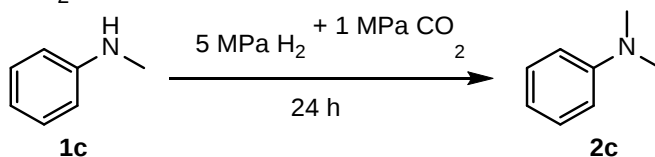


Figure S10. Time-course plot for the *N*-methylation of *N*-methylaniline with CO₂ and H₂ catalyzed by Re/TiO₂; **1c**: *N*-methylaniline; **2c**: *N,N*-dimethylaniline.

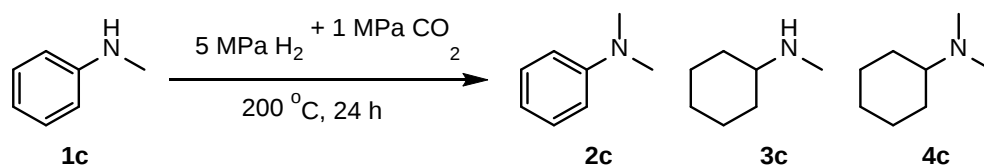
Table S8. Effect of the reaction temperature on the *N*-methylation of *N*-methylaniline with CO₂ and H₂ catalyzed by Re/TiO₂.^a



Temperature [°C]	Conv. [%]	Yield [%]
200	98	97
180	85	80
160	64	58
140	42	35

^aReaction conditions: 2 mol% Re, 1 mmol *N*-methylaniline, 3 mL octane, $p_{\text{CO}_2} = 1\text{ MPa}$ and $p_{\text{H}_2} = 5\text{ MPa}$, $t = 24\text{ h}$. Yields were determined by GC using *n*-dodecane as the internal standard.

Table S9. Heterogeneous catalytic *N*-methylation of *N*-methylaniline with CO₂ and H₂.



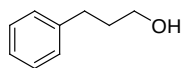
Entry	Catalysts	Conv. [%]	Yield [%]		
			2c	3c	4c
1	Re/TiO ₂	98	97	0	0
2	Ru/TiO ₂	94	0	22	31
3	Pt/TiO ₂	38	15	0	0
4	Ir/TiO ₂	14	8	3	0
5	Rh/TiO ₂	10	5	3	0
6	Pd/TiO ₂	19	3	1	0
7	Ag/TiO ₂	5	5	0	0
8	Cu/TiO ₂	6	4	0	0
9	Ni/TiO ₂	4	2	0	0
10	Co/TiO ₂	4	1	0	0
11	TiO ₂	1	1	0	0
12	Re/Carbon	8	7	0	0
13	Re/ θ -Al ₂ O ₃	32	31	0	0
14	Re/ γ -Al ₂ O ₃	32	32	0	0
15	Re/ZrO ₂	23	20	0	0
16	Re/HZSM-5(22)	15	6	0	0
17	Re/SiO ₂	7	3	0	0
18	Re/Nb ₂ O ₅	25	18	0	0
19	Re/CeO ₂	12	8	0	0
20	Re/MgO	8	5	0	0

^aReaction conditions: 2 mol% catalyst, 1 mmol *N*-methylaniline, 3 mL octane, p_{CO_2} = 1 MPa and p_{H_2} = 5 MPa, T = 200 °C, t = 24 h. Yields were determined by GC using *n*-dodecane as the internal standard.

3. NMR data

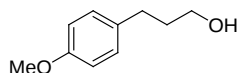
^1H and ^{13}C NMR spectra for the obtained products were assigned according to previous reports in the literature. Abbreviations used in the NMR experiments: s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet. GC-MS spectra were recorded on a SHIMADZU QP2010.

3-Phenyl-propan-1-ol: ^[1]



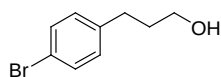
^1H NMR (600.17 MHz, CDCl_3 , TMS): δ 7.29-7.27 (m, 2H), 7.19 (d, J = 6.84 Hz, 3H), 3.66 (t, J = 6.18 Hz, 2H), 2.70 (t, J = 7.56 Hz, 2H), 1.90-1.88 (m, 2H), 1.45 (s, 1H); ^{13}C NMR (150.92 MHz, CDCl_3): δ 141.78, 128.38 (C $\times$ 2), 128.36 (C $\times$ 2), 125.83, 62.22, 34.18, 32.03; GC-MS m/e 136.15.

3-(3-Methoxyphenyl)-propan-1-ol: ^[1]



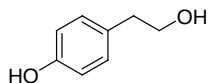
^1H NMR (600.17 MHz, CDCl_3 , TMS): δ 7.18 (t, J = 7.92 Hz, 1H), 6.77 (d, J = 7.56 Hz, 1H), 6.74-6.71 (m, 2H), 3.77 (s, 3H), 3.64 (t, J = 6.54 Hz, 2H), 2.66 (t, J = 7.56 Hz, 2H), 2.00 (br s, 1H), 1.89-1.84 (m, 2H); ^{13}C NMR (150.92 MHz, CDCl_3): δ 159.54, 143.42, 129.25, 120.75, 114.13, 111.01, 62.04, 55.02, 33.99, 32.02; GC-MS m/e 166.15.

3-(3-bromophenyl)-propan-1-ol: ^[2]



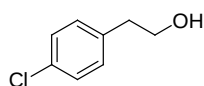
^1H NMR (600.17 MHz, CDCl_3 , TMS): δ 7.40 (t, J = 6.12 Hz, 2H), 7.06 (d, J = 6.9 Hz, 2H), 3.65 (s, 2H), 2.66 (t, J = 6.87 Hz, 2H), 1.85 (t, J = 6.87 Hz, 2H), 1.40 (s, 1H); ^{13}C NMR (150.92 MHz, CDCl_3): δ 140.73, 131.04 (C $\times$ 2), 130.17 (C $\times$ 2), 119.55, 61.95, 33.97, 31.41; GC-MS m/e 214.15.

4-(2-hydroxy-ethyl)-phenol: ^[3]



¹H NMR (600.17 MHz, CDCl₃, TMS): δ 8.07 (br, s, 1H), 7.04 (d, *J* = 8.28 Hz, 2H), 6.73 (d, *J* = 8.22 Hz, 2H), 3.68 (td, *J* = 8.00, 4.82 Hz, 2H), 3.61 (br, t, *J* = 5.49 Hz, 1H), 2.70 (t, *J* = 7.02 Hz, 2H); ¹³C NMR (150.92 MHz, CDCl₃): δ 156.11, 130.60, 130.29 (C×2), 115.47 (C×2), 63.86, 39.07; GC-MS *m/e* 138.10.

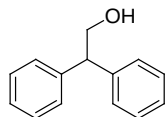
4-(2-chloro-ethyl)-phenol: ^[4]



¹H NMR (600.17 MHz, CDCl₃, TMS): δ 7.28 (d, *J* = 8.22 Hz, 2H), 7.15 (d, *J* = 8.22 Hz, 2H), 3.82 (t, *J* = 6.18 Hz, 2H), 2.82 (t, *J* = 6.18 Hz, 2H), 1.60 (br s, 1H); ¹³C NMR (150.92 MHz, CDCl₃): δ 137.02, 132.23, 130.32 (C×2), 128.61 (C×2), 63.39, 38.44; GC-MS *m/e* 156.10.

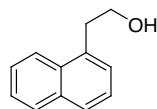
Note: 2-phenyl-ethanol was observed in 16% yield by NMR and GCMS analysis.

3,3-diphenyl-propan-1-ol: ^[5]

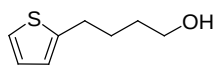


¹H NMR (600.17 MHz, CDCl₃, TMS): δ 7.32-7.29 (m, 4H), 7.25-7.22 (m, 4H), 7.21-7.20 (m, 2H), 4.19 (t, *J* = 7.23 Hz, 1H), 4.15 (t, *J* = 6.15 Hz, 2H), 1.60 (br s, 1H); ¹³C NMR (150.92 MHz, CDCl₃): δ 141.36 (C×2), 128.67 (C×4), 128.28 (C×4), 126.77 (C×2), 66.08, 53.59; GC-MS *m/e* 198.15.

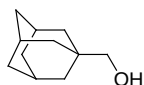
2-Napthalene-1-yl-ethanol:^[5]



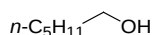
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 8.00 (d, *J* = 8.22 Hz, 1H), 7.81 (d, *J* = 7.56 Hz, 1H), 7.70 (d, *J* = 7.56 Hz, 1H), 7.49-7.43 (m, 2H), 7.37 (t, *J* = 7.56 Hz, 1H), 7.31 (d, *J* = 6.90 Hz, 1H), 3.89 (t, *J* = 6.54 Hz, 2H), 3.27 (t, *J* = 6.51 Hz, 2H), 1.83 (br s, 1H); ¹³C NMR (150.92 MHz, CDCl₃): δ 134.34, 133.86, 131.96, 128.74, 127.18, 127.03, 125.93, 125.54, 125.40, 123.56, 62.85, 36.07; GC-MS *m/e* 172.10.

4-thiophen-2-yl-butane-1-ol: ^[6]

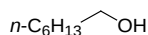
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 7.09 (d, *J* = 6.18 Hz, 1H), 6.91-6.89 (m, 1H), 6.77 (d, *J* = 3.84 Hz, 1H), 3.64 (t, *J* = 6.54 Hz, 2H), 2.85 (t, *J* = 7.56 Hz, 2H), 1.77-1.72 (m, 2H), 1.65-1.60 (m, 3H); ¹³C NMR (150.92 MHz, CDCl₃): δ 145.13, 126.63, 124.06, 122.86, 62.52, 32.03, 29.57, 27.88; GC-MS *m/e* 156.10.

Adamantan-1-yl-methanol: ^[7]

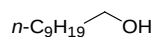
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 3.19 (s, 2H), 1.99 (s, 3H), 1.73 (pseudo-d, *J* = 12.36 Hz, 3H), 1.65 (pseudo-d, *J* = 12.36 Hz, 3H), 1.50 (s, 6H), 1.27 (br, s, 1H); ¹³C NMR (150.92 MHz, CDCl₃): δ 73.86, 39.02 (C×3), 37.16 (C×3), 34.46 (C×2), 28.16 (C×2); GC-MS *m/e* 166.15.

Hexan-1-ol: ^[6]

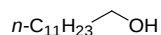
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 3.64 (t, *J* = 6.09 Hz, 2H), 1.58-1.53 (m, 2H), 1.38-1.27 (m, 6H), 1.19 (br s, 1H), 0.89 (t, *J* = 6.75 Hz, 3H); ¹³C NMR (150.92 MHz, CDCl₃): δ 63.10, 32.70, 31.52, 25.36, 22.54, 14.01; GC-MS *m/e* 102.15.

Heptan-1-ol: ^[6]

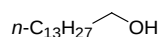
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 3.58 (t, *J* = 6.87 Hz, 2H), 3.44 (br s, 1H), 1.54 (t, *J* = 6.90 Hz, 2H), 1.32-1.28 (m, 8H), 0.88 (t, *J* = 6.51 Hz, 3H); ¹³C NMR (150.92 MHz, CDCl₃): δ 62.38, 32.53, 31.72, 29.03, 25.63, 22.47, 13.85; GC-MS *m/e* 116.15.

Decan-1-ol:^[6]

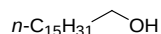
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 3.60 (t, *J* = 6.51 Hz, 2H), 2.46 (br s, 1H), 1.56-1.54 (m, 2H), 1.33-1.26 (m, 14H), 0.88 (t, *J* = 6.87 Hz, 3H); ¹³C NMR (150.92 MHz, CDCl₃): δ 62.72, 32.66, 31.83, 29.57, 29.51, 29.40, 29.26, 25.71, 22.60, 13.98; GC-MS *m/e* 158.20.

Dodecan-1-ol:^[6]

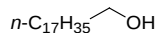
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 3.63 (pseudo-t, *J* = 6.54 Hz, 2H), 1.56-1.54 (m, 2H), 1.33-1.26 (m, 19H), 0.88 (t, *J* = 6.18 Hz, 3H); ¹³C NMR (150.92 MHz, CDCl₃): δ 63.06, 32.79, 31.90, 29.64, 29.60, 29.59, 29.58, 29.42, 29.32, 25.72, 22.67, 14.09; GC-MS *m/e* 186.25.

Tetradecan-1-ol:^[6]

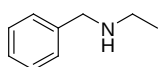
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 3.61 (t, *J* = 6.84 Hz, 2H), 1.96 (br s, 1H), 1.56-1.53 (m, 2H), 1.34-1.26 (m, 22H), 0.87 (t, *J* = 6.56 Hz, 3H); ¹³C NMR (150.92 MHz, CDCl₃): δ 62.88, 32.73, 31.88, 29.65 (C×2), 29.59 (C×2), 29.42 (C×2), 29.32 (C×2), 25.72, 22.64, 14.04; GC-MS *m/e* 214.25.

Hexadecan-1-ol:^[6]

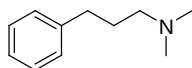
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 3.63 (t, *J* = 6.54 Hz, 2H), 1.57-1.55 (m, 2H), 1.34-1.25 (m, 27H), 0.88 (t, *J* = 7.20 Hz, 3H); ¹³C NMR (150.92 MHz, CDCl₃): δ 63.07, 32.80, 31.91, 29.68 (C×2), 29.65 (C×2), 29.60 (C×2), 29.42 (C×2), 29.35 (C×2), 25.72, 22.68, 14.10; GC-MS *m/e* 242.30.

Octadecan-1-ol:^[6]

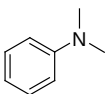
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 3.63 (t, *J* = 6.54 Hz, 2H), 1.57-1.55 (m, 2H), 1.35-1.25 (m, 31H), 0.88 (t, *J* = 6.87 Hz, 3H); ¹³C NMR (150.92 MHz, CDCl₃): δ 63.06, 32.79, 31.91, 29.69 (C×4), 29.65 (C×2), 29.59 (C×2), 29.43 (C×2), 29.35 (C×2), 25.72, 22.68, 14.10; GC-MS *m/e* 270.30.

Benzyl-ethyl-amine:^[8]

¹H NMR (600.17 MHz, CDCl₃, TMS): δ 7.33-7.29 (m, 4H), 7.22 (d, *J* = 8.28 Hz, 1H), 3.77 (s, 2H), 2.68-2.65 (m, 2 H), 1.15-1.11 (m, 4H); ¹³C NMR (150.92 MHz, CDCl₃): δ 140.43, 128.25 (C×2), 128.00 (C×2), 126.73, 53.87, 43.55, 15.20; GC-MS *m/e* 136.10.

Dimethyl (3- phenyl-propyl)-amine:^[9]

¹H NMR (600.17 MHz, CDCl₃, TMS): δ 7.27 (d, *J* = 6.84 Hz, 2H), 7.18 (t, *J* = 7.56 Hz, 3H), 2.63 (t, *J* = 7.82 Hz, 2H), 2.29 (t, *J* = 7.56 Hz, 2H), 2.22 (s, 6H), 1.80-1.77 (m, 2H); ¹³C NMR (150.92 MHz, CDCl₃): δ 142.26, 128.33 (C×2), 128.26 (C×2), 125.67, 59.27, 45.47 (C×2), 33.63, 29.44; GC-MS *m/e* 163.25.

Dimethyl-phenyl-amine:^[10]

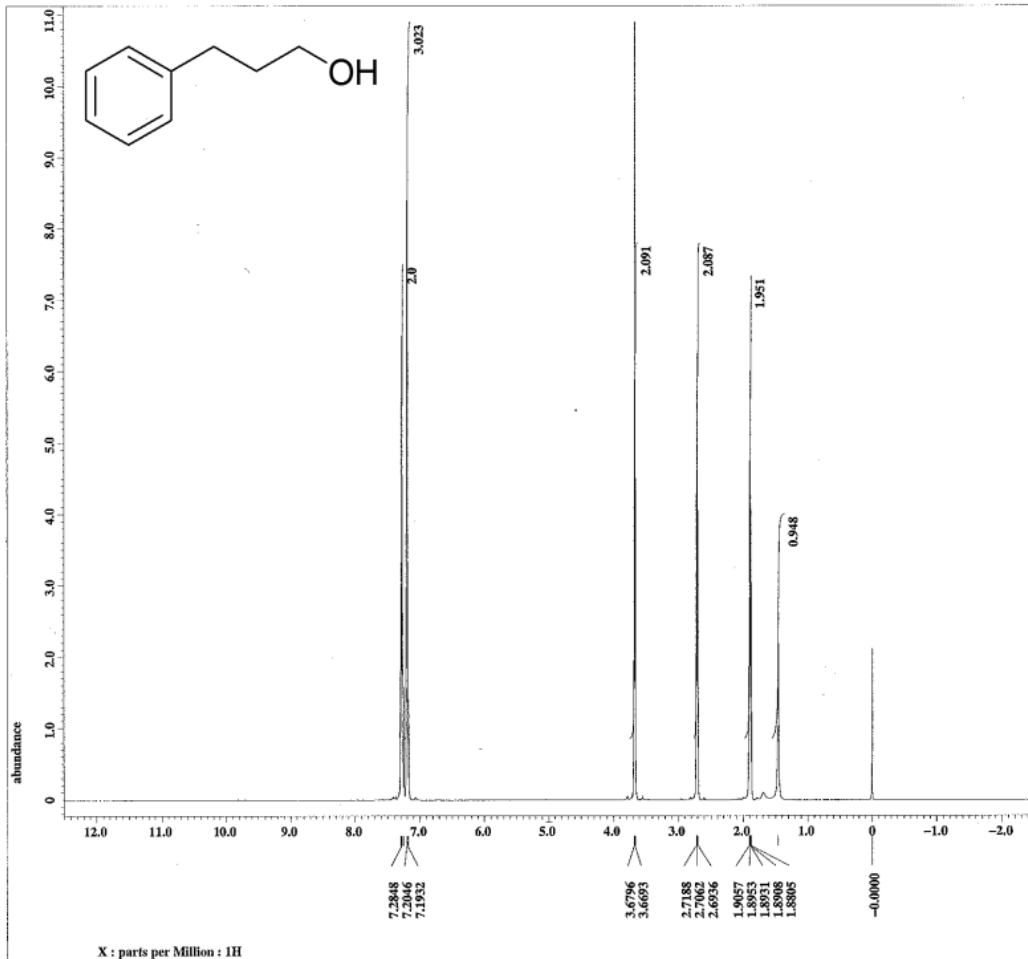
¹H NMR (600.17 MHz, CDCl₃, TMS): δ 7.24 (t, *J* = 7.56 Hz, 2H), 6.75-6.70 (m, 3H), 2.93 (s, 6H); ¹³C NMR (150.92 MHz, CDCl₃): δ 150.61, 129.02 (C×2), 116.58, 112.60 (C×2), 40.58 (C×2); GC-MS *m/e* 122.10.

Filename = Exp-AB-Est-red-Ph-C3-
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = Exp-AB-Est-red-Ph-C3-
 Solvent = CHLOROFORM-D
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 Revision_time = 18-JAN-2017 16:21:02
 Current_time = 18-JAN-2017 16:21:07

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 Spectrometer = DELTA2_NMR

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 X_points = 16384
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 X_resolution = 0.68733284[Hz]
 X_sweep = 11.26126126[KHz]
 Irr_domain = 1H
 Irr_freq = 600.1723046[MHz]
 Irr_offset = 5[ppm]
 Tri_domain = 1H
 Tri_freq = 600.1723046[MHz]
 Tri_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 8
 Total_scans = 8

X_90_width = 12.6[us]
 X_acq_time = 1.4548992[s]
 X_angle = 45[deg]
 X_atn = 3.6[db]
 X_pulse = 6.3[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_preset = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 46
 Relaxation_delay = 5[s]
 Repetition_time = 6.4548992[s]
 Temp_get = 22.6[degC]

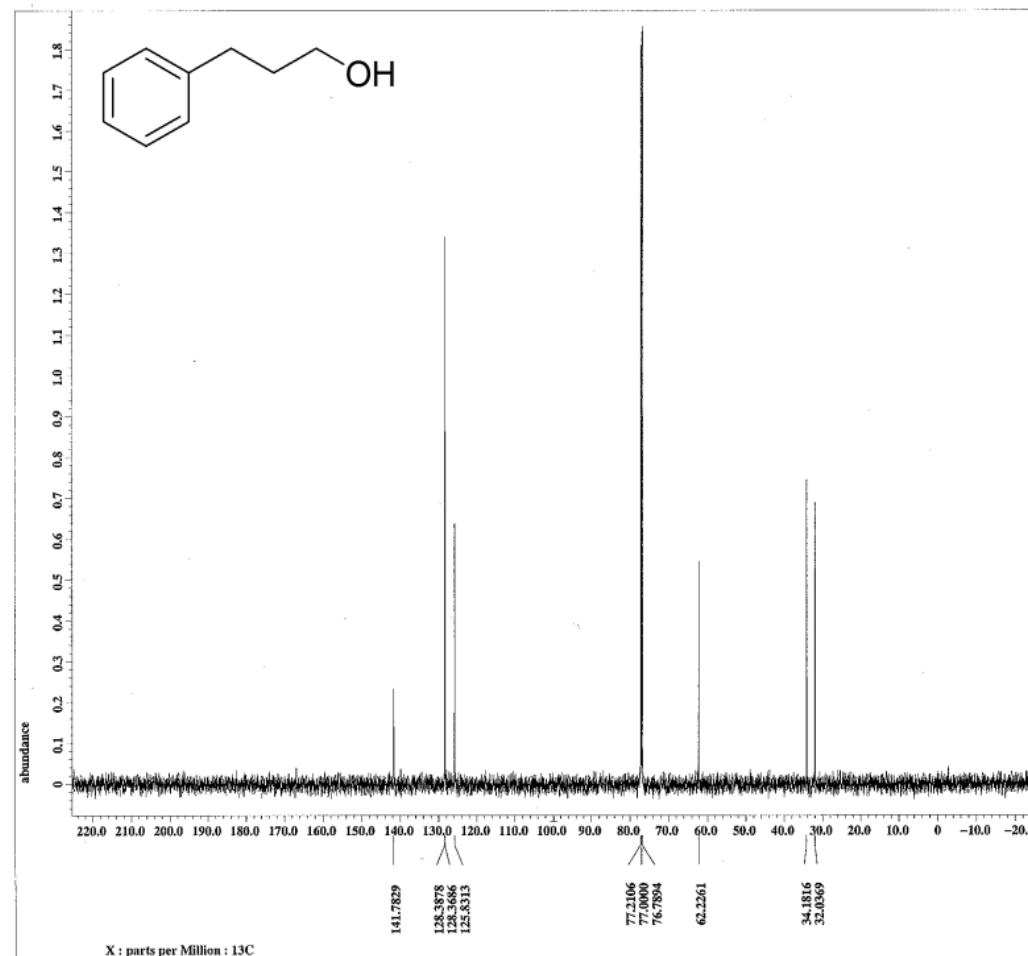


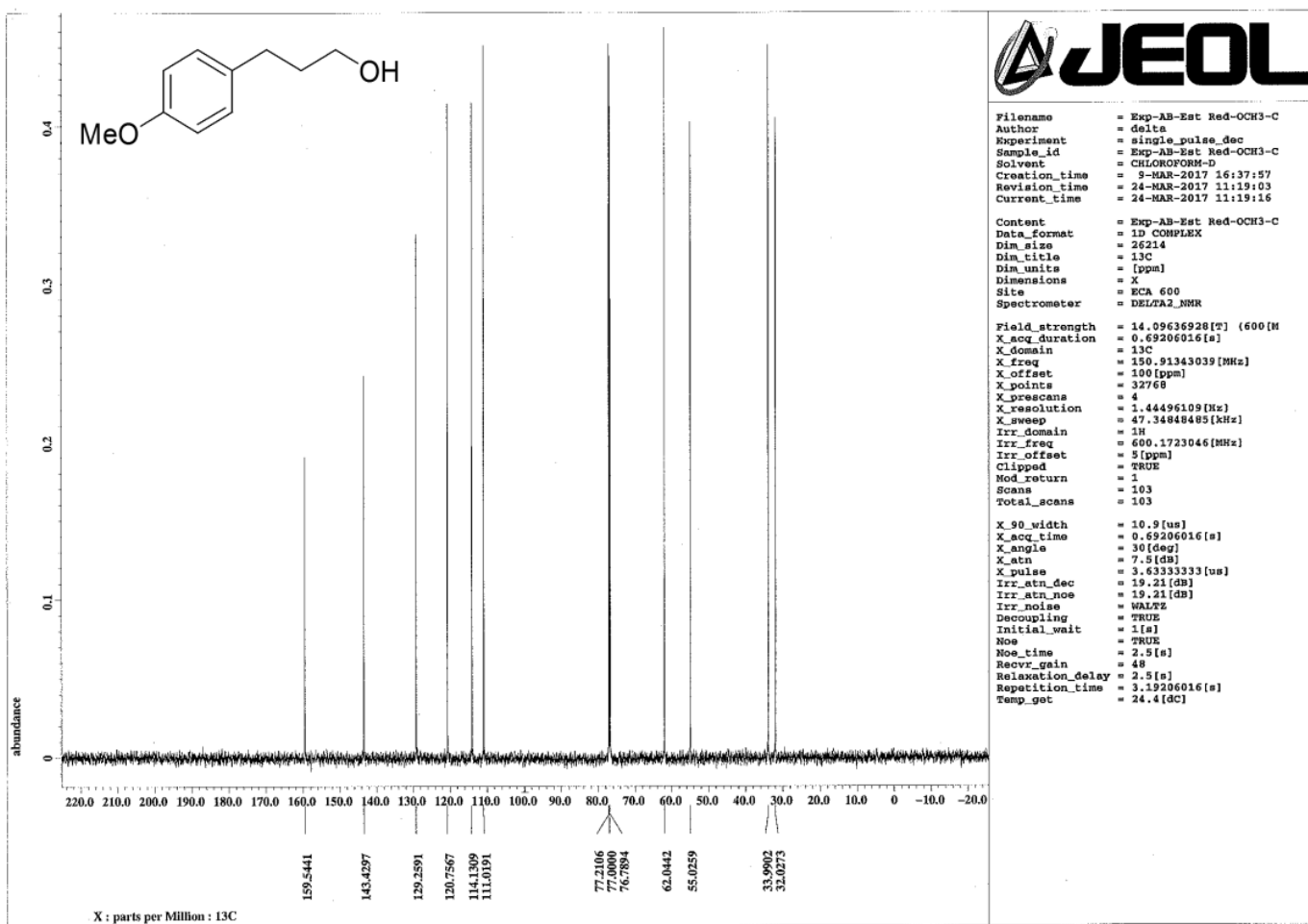
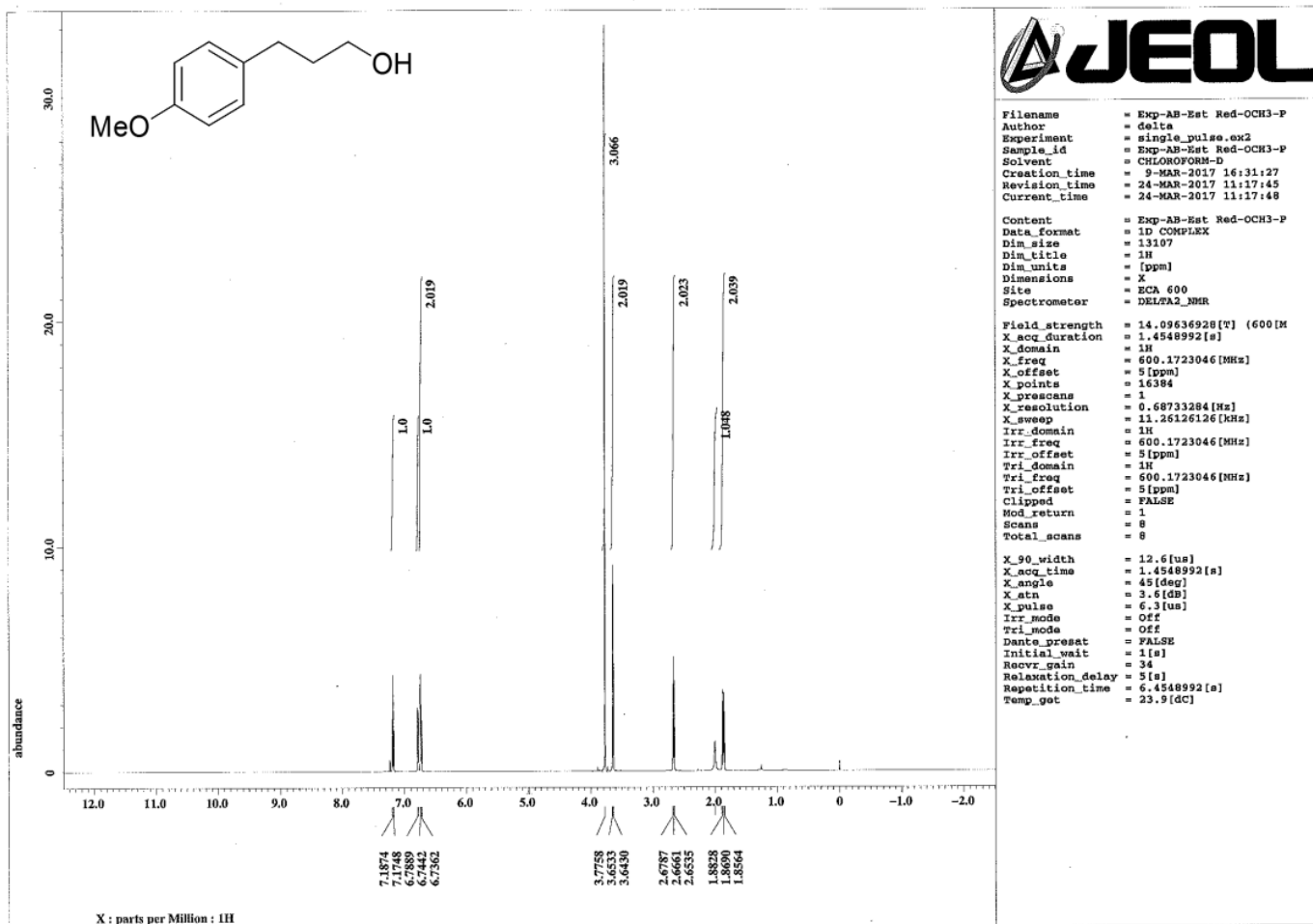
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 Solvent = CHLOROFORM-D
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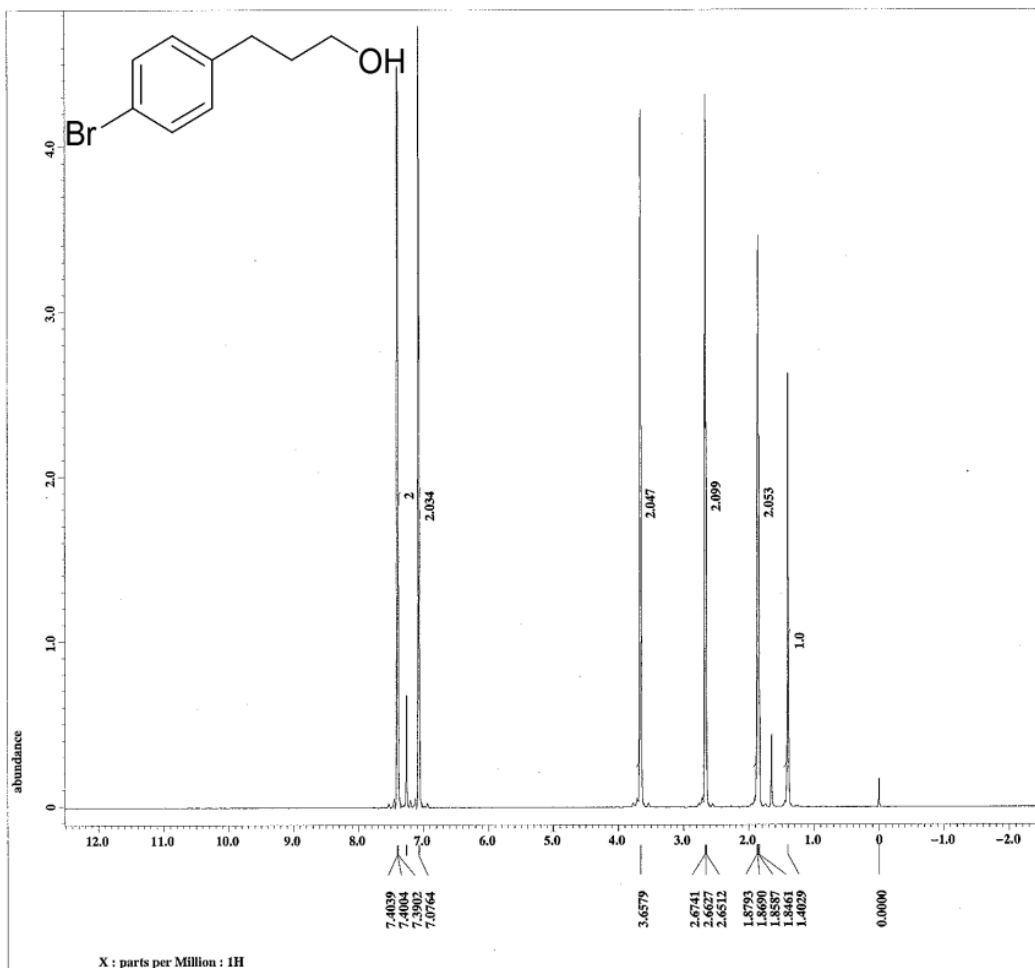
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 Site = ECA 600
 Spectrometer = DELTA2_NMR

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 Irr_domain = 1H
 Irr_freq = 600.1723046[MHz]
 Irr_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 53
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X_90_width = 10.9[us]
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 X_atn = 7.5[db]
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 Decoupling = TRUE
 Initial_wait = 1[s]
 Noe = TRUE
 Noe_time = 2.5[s]
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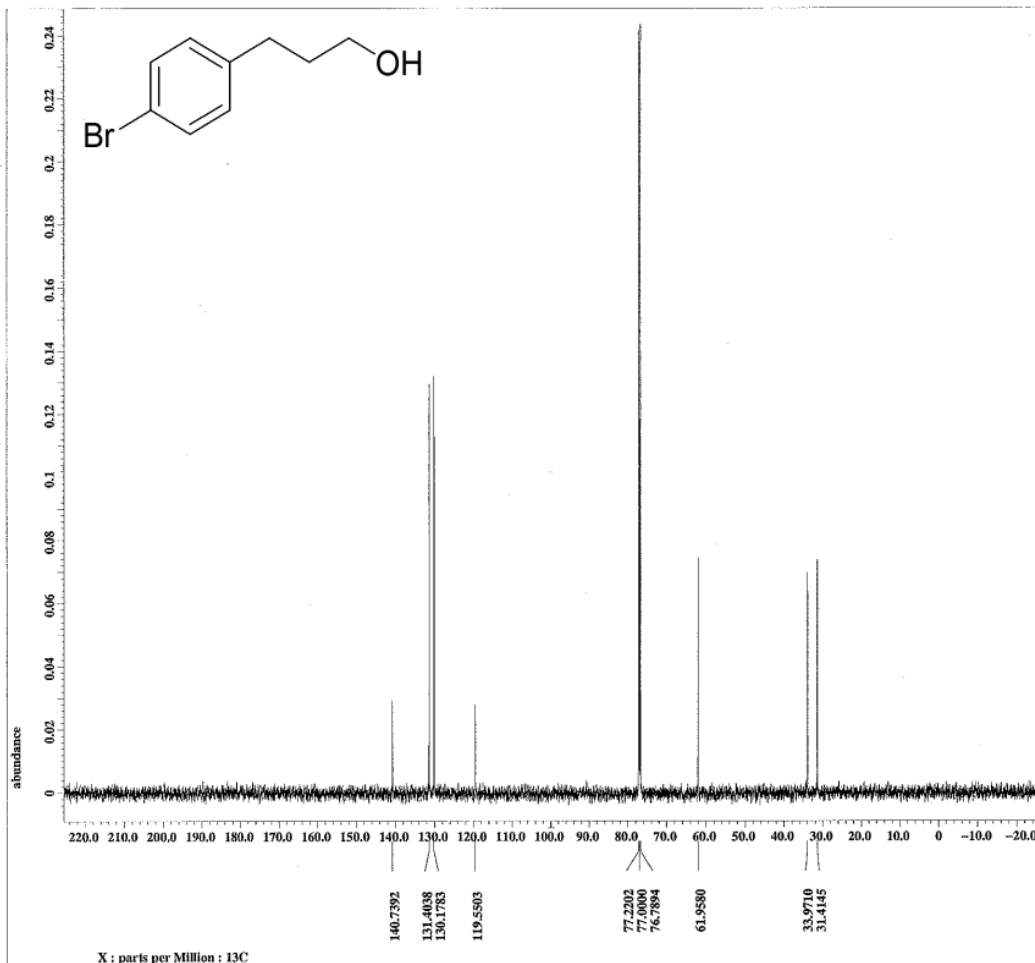


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 Solvent = CHLOROFORM-D
 Creation_time = 18-JAN-2017 15:49:03
 Revision_time = 18-JAN-2017 16:53:22
 Current_time = 18-JAN-2017 16:53:29

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 Data_format = 1D COMPLEX
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 Dim_units = [ppm]
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 Site = ECA 600
 Spectrometer = DELTA2_NMR

Field_strength = 14.09636928 [T] (600 [M]
 X_acq_duration = 1.4548992 [s]
 X_domain = 1H
 X_freq = 600.1723046 [MHz]
 X_offset = 5 [ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.68733284 [Hz]
 X_sweep = 11.26126126 [kHz]
 Irr_domain = 1H
 Irr_freq = 600.1723046 [MHz]
 Irr_offset = 5 [ppm]
 Tri_domain = 1H
 Tri_freq = 600.1723046 [MHz]
 Tri_offset = 5 [ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 8
 Total_scans = 8

X_90_width = 12.6 [us]
 X_acq_time = 1.4548992 [s]
 X_angle = 45 [deg]
 X_atn = 3.6 [dB]
 X_pulse = 6.3 [us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_preset = FALSE
 Initial_wait = 1 [s]
 Recvr_gain = 48
 Relaxation_delay = 5 [s]
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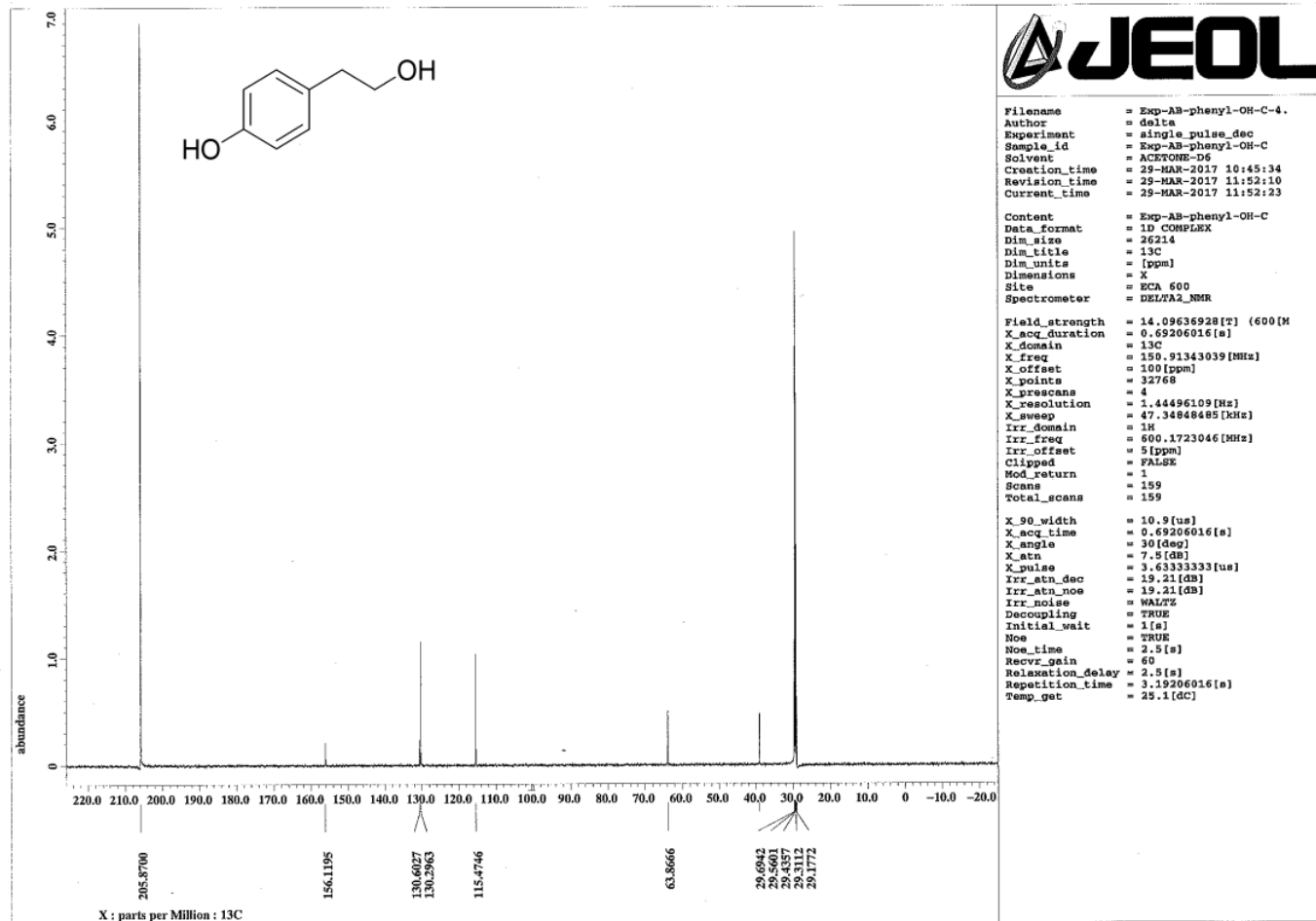
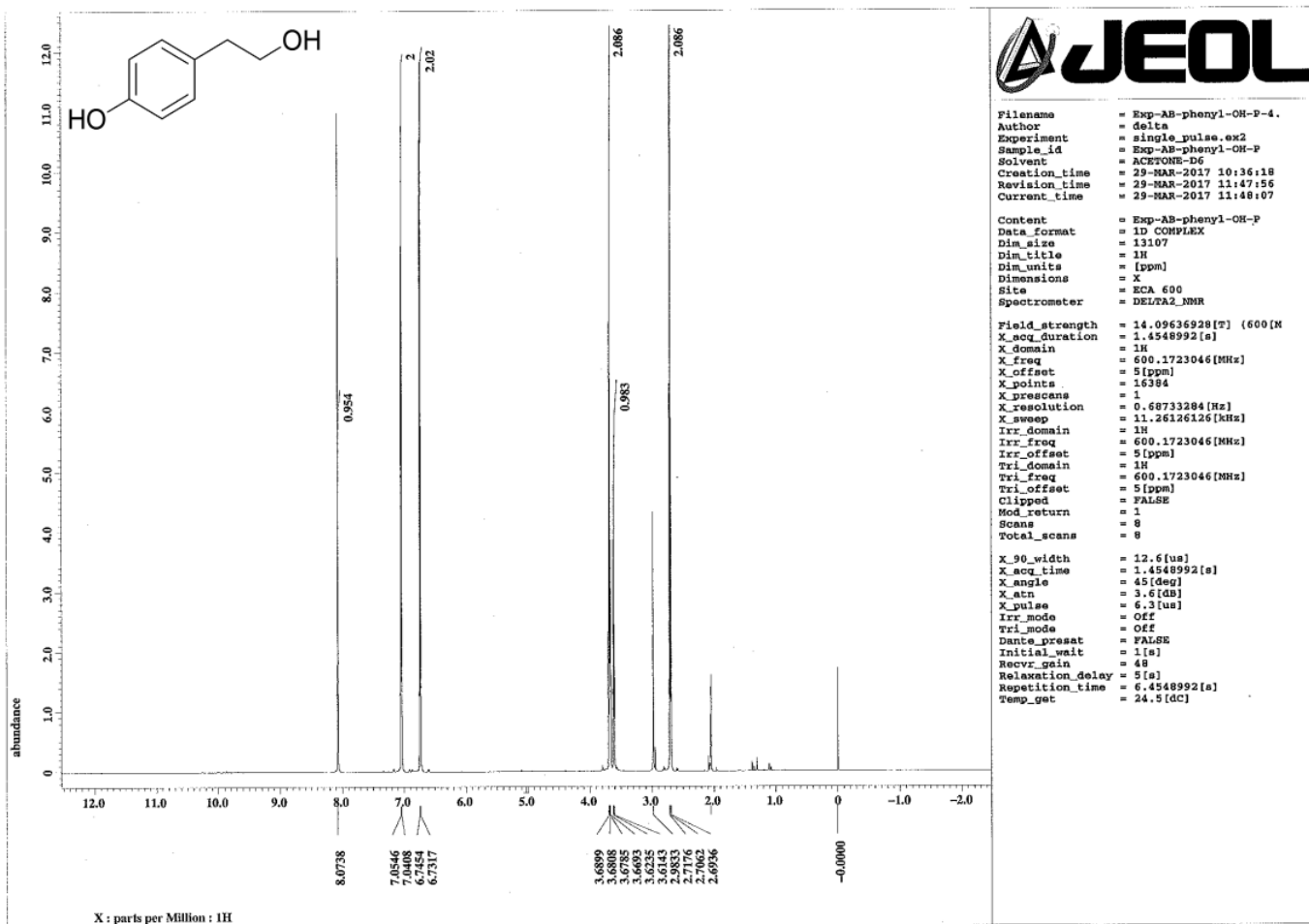


Filename = Exp-AB-Est-red-Br-Ph-
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 Solvent = CHLOROFORM-D
 Creation_time = 18-JAN-2017 15:56:44
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 Site = ECA 600
 Spectrometer = DELTA2_NMR

Field_strength = 14.09636928 [T] (600 [M]
 X_acq_duration = 0.69206016 [s]
 X_domain = 13C
 X_freq = 150.91343039 [MHz]
 X_offset = 100 [ppm]
 X_points = 32768
 X_prescans = 4
 X_resolution = 1.44496109 [Hz]
 X_sweep = 47.34848485 [kHz]
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 Clipped = TRUE
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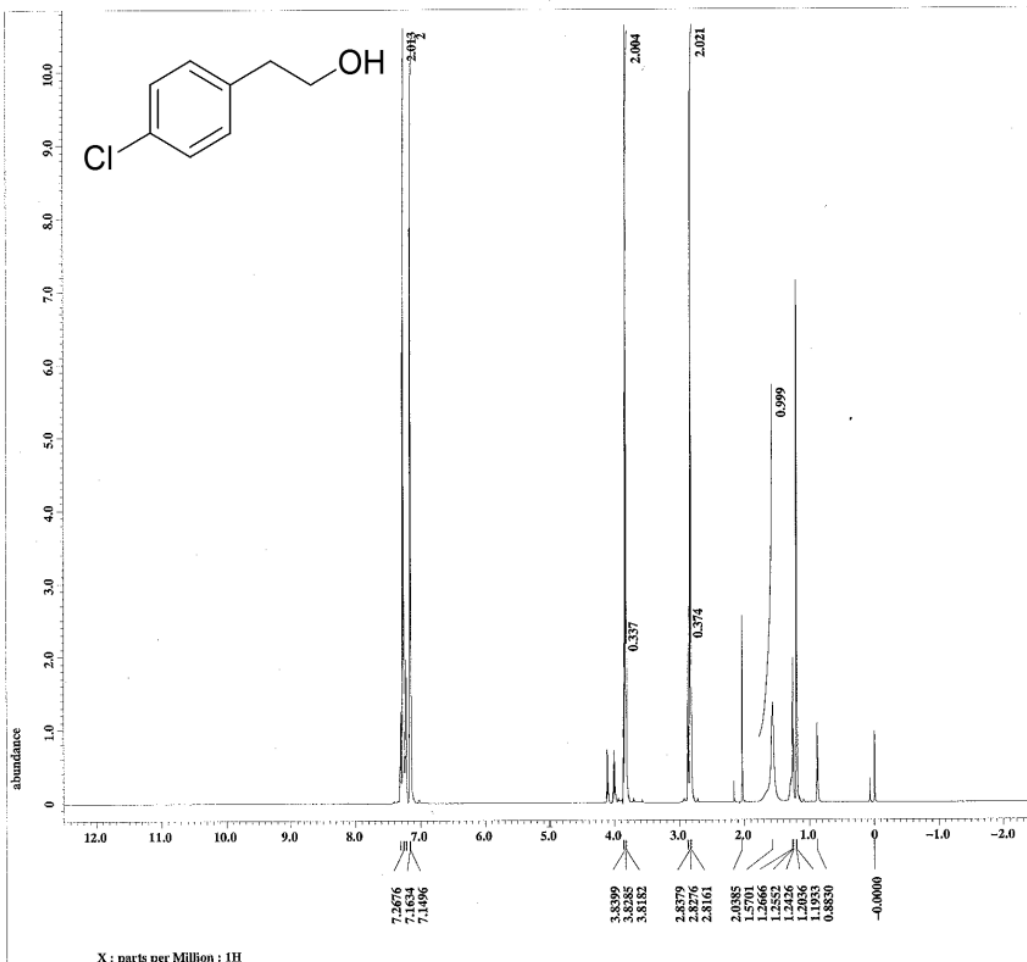
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 Irr_atn_dec = 19.21 [dB]
 Irr_atn_noe = 19.21 [dB]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 1 [s]
 Noe = TRUE
 Noe_time = 2.5 [s]
 Recvr_gain = 44
 Relaxation_delay = 2.5 [s]
 Repetition_time = 3.19206016 [s]
 Temp_get = 23.2 [dc]



Filename = Exp-AB-Chloro-P-4.jdf
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 Experiment = single_pulse.ex2
 Sample_id = Exp-AB-Chloro-P
 Solvent = CHLOROFORM-D
 Creation_time = 28-MAR-2017 16:30:10
 Revision_time = 29-MAR-2017 11:01:09
 Current_time = 29-MAR-2017 11:01:11

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 Data_format = 1D COMPLEX
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 Dim_units = [ppm]
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 Site = ECA 600
 Spectrometer = DELTA2_NMR

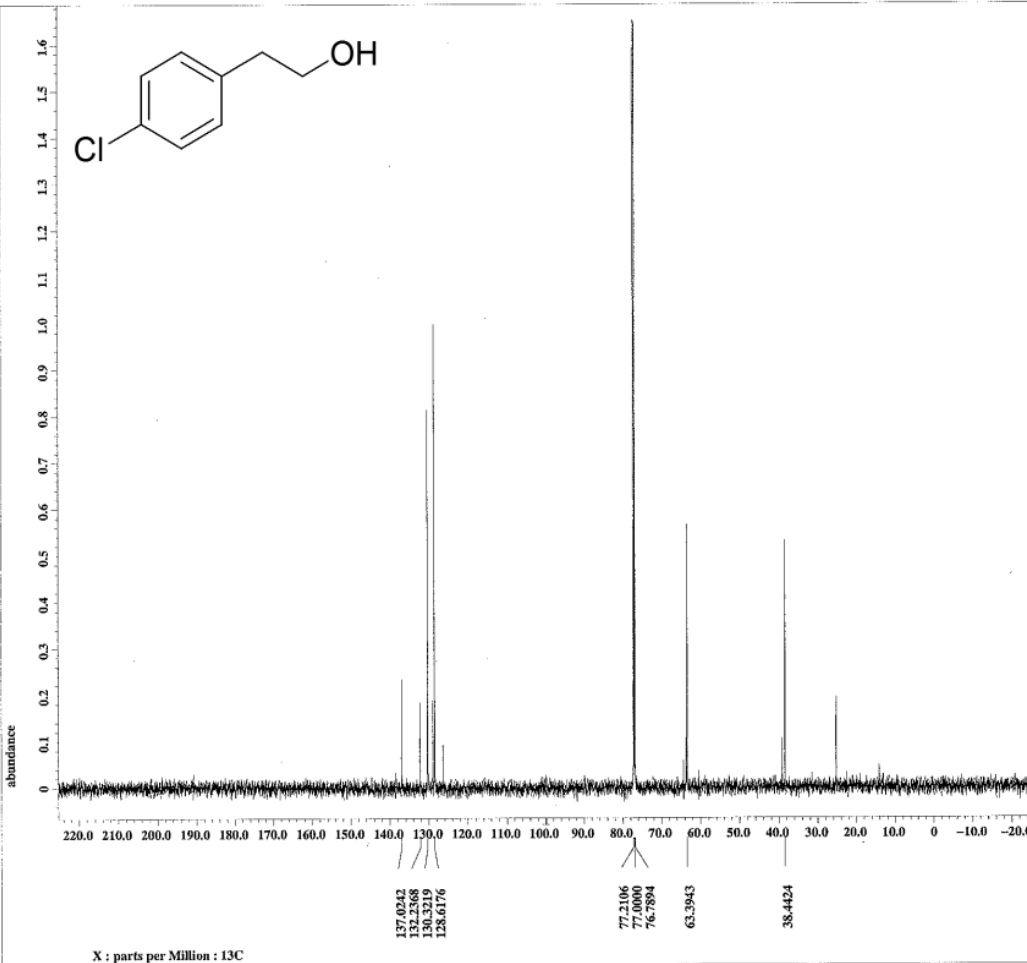
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 Irr_domain = 1H
 Irr_freq = 600.1723046[MHz]
 Irr_offset = 5[ppm]
 Tri_domain = 1H
 Tri_freq = 600.1723046[MHz]
 Tri_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 8
 Total_scans = 8
 X_90_width = 12.6[us]
 X_acq_time = 1.4548992[s]
 X_angle = 45[deg]
 X_atn = 3.6[db]
 X_pulse = 6.3[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
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 Relaxation_delay = 5[s]
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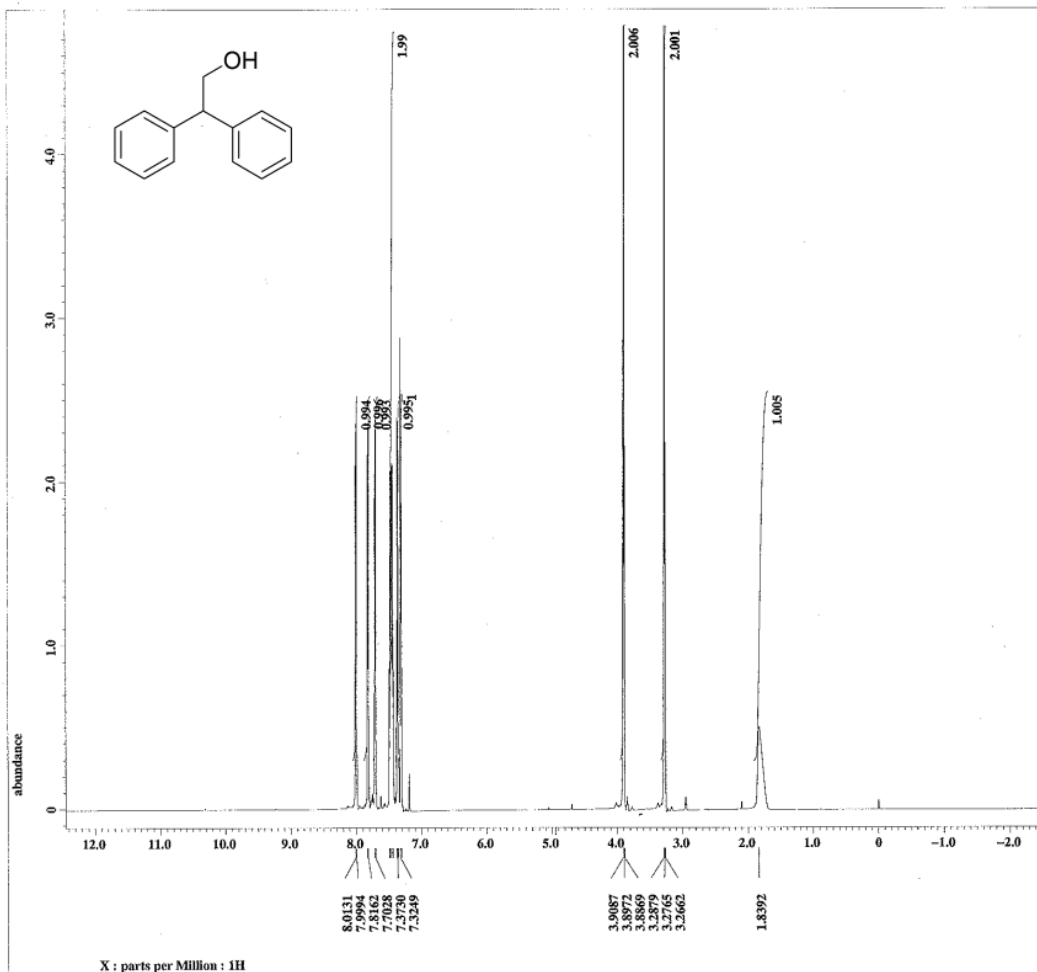


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 Current_time = 29-MAR-2017 11:04:40

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 Spectrometer = DELTA2_NMR

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 X_freq = 150.91343039[MHz]
 X_offset = 100[ppm]
 X_points = 32768
 X_prescans = 4
 X_resolution = 1.44496109[Hz]
 X_sweep = 47.34848485[kHz]
 Irr_domain = 1H
 Irr_freq = 600.1723046[MHz]
 Irr_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 104
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 X_acq_time = 0.69206016[s]
 X_angle = 30[deg]
 X_atn = 7.5[db]
 X_pulse = 3.63333333[us]
 Irr_atn_dec = 19.21[db]
 Irr_atn_noe = 19.21[db]
 Irr_noise = WALTZ
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 Initial_wait = 1[s]
 Noe = TRUE
 Noe_time = 2.5[s]
 Recvr_gain = 60
 Relaxation_delay = 2.5[s]
 Repetition_time = 3.19206016[s]
 Temp_get = 24.8[dc]





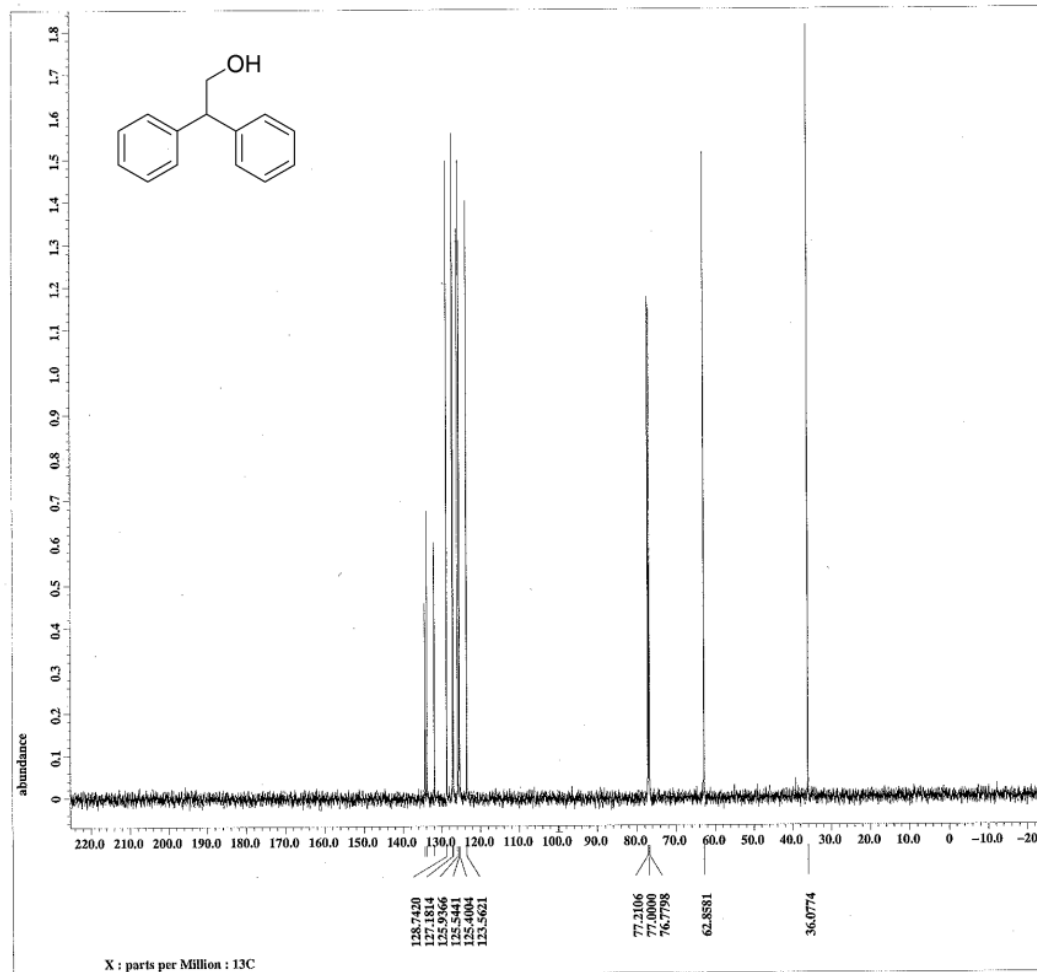
```

Filename      = Exp-AB-Est Red-1-napt
Author        = delta
Experiment    = single_pulse.ex2
Sample_id     = Exp-AB-Est Red-1-napt
Solvent       = CHLOROFORM-D
Creation_time  = 9-MAR-2017 16:45:37
Revision_time  = 24-MAR-2017 11:13:32
Current_time  = 24-MAR-2017 11:13:38

Content       = Exp-AB-Est Red-1-napt
Data_format   = 1D COMPLEX
Dim_size      = 13107
Dim_title     = 1H
Dim_units     = [ppm]
Dimensions    = X
Site          = ECA 600
Spectrometer  = DELTA2_NMR

Field_strength = 14.09636928[T] (600[M]
X_acq_duration = 1.4548992[s]
X_domain      = 1H
X_freq        = 600.1723046[MHz]
X_offset      = 5[ppm]
X_points      = 16384
X_prescans    = 1
X_resolution  = 0.68733284[Hz]
X_sweep       = 11.26126126[kHz]
Irr_domain    = 1H
Irr_freq      = 600.1723046[MHz]
Irr_offset    = 5[ppm]
Tri_domain    = 1H
Tri_freq      = 600.1723046[MHz]
Tri_offset    = 5[ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 8
Total_scans   = 8

X_90_width    = 12.6[us]
X_acq_time    = 1.4548992[s]
X_angle       = 45[deg]
X_atn         = 3.6[db]
X_pulse       = 6.3[us]
Irr_mode      = Off
Tri_mode      = Off
Dante_preset  = FALSE
Initial_wait  = 1[s]
Recvr_gain    = 36
Relaxation_delay = 5[s]
Repetition_time = 6.4548992[s]
Temp_get      = 23.9[degC]
  
```



```

Filename      = Exp-AB-Est Red-1-napt
Author        = delta
Experiment    = single_pulse_dec
Sample_id     = Exp-AB-Est Red-1-napt
Solvent       = CHLOROFORM-D
Creation_time  = 9-MAR-2017 16:51:55
Revision_time  = 24-MAR-2017 11:14:26
Current_time  = 24-MAR-2017 11:14:46

Content       = Exp-AB-Est Red-1-napt
Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECA 600
Spectrometer  = DELTA2_NMR

Field_strength = 14.09636928[T] (600[M]
X_acq_duration = 0.69206016[s]
X_domain      = 13C
X_freq        = 150.91343039[MHz]
X_offset      = 100[ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 1.44496109[Hz]
X_sweep       = 47.34848485[kHz]
Irr_domain    = 1H
Irr_freq      = 600.1723046[MHz]
Irr_offset    = 5[ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 103
Total_scans   = 103

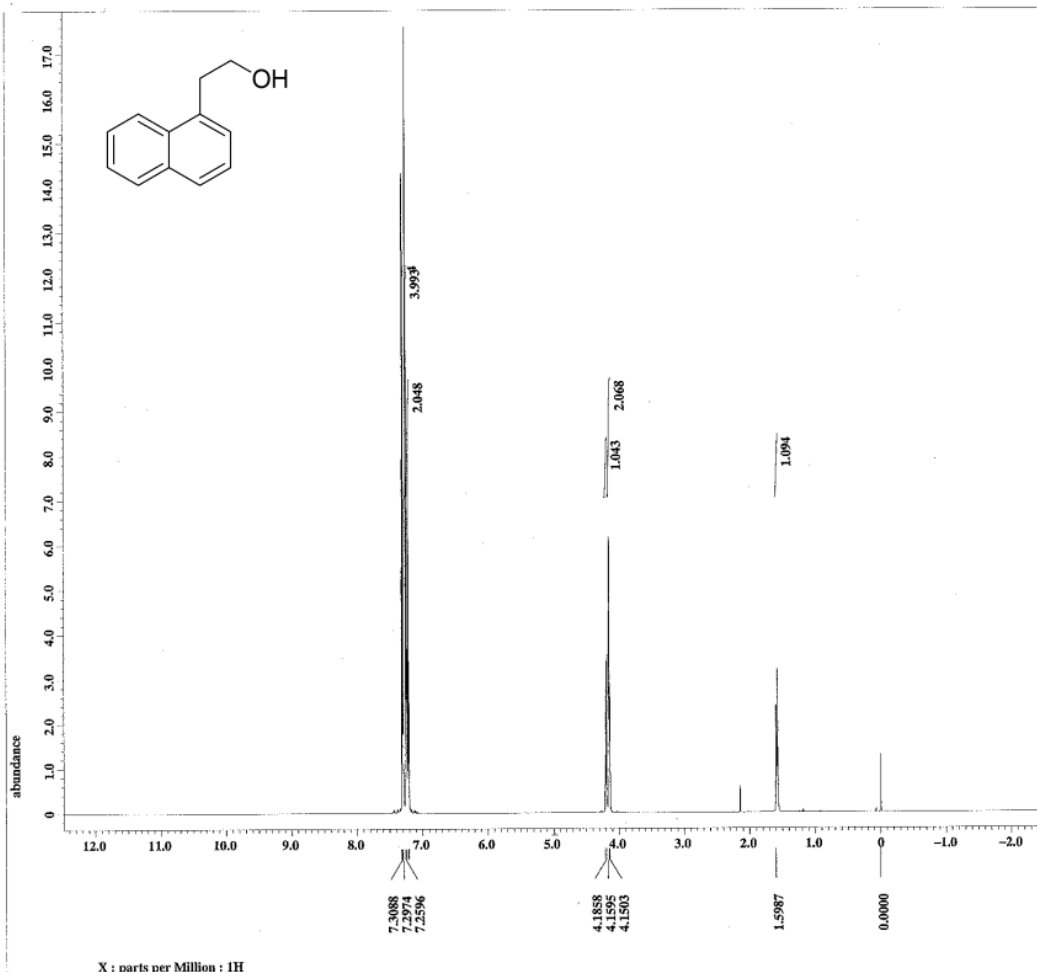
X_90_width    = 10.9[us]
X_acq_time    = 0.69206016[s]
X_angle       = 30[deg]
X_atn         = 7.5[db]
X_pulse       = 3.63333333[us]
Irr_atn_dec   = 19.21[db]
Irr_atn_dec   = 19.21[db]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1[s]
Noe           = TRUE
Noe_time      = 2.5[s]
Recvr_gain    = 60
Relaxation_delay = 2.5[s]
Repetition_time = 3.19206016[s]
Temp_get      = 24.4[degC]
  
```

Filename = Exp-AB-diphenylethyl-
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = Exp-AB-diphenylethyl-
 Solvent = CHLOROFORM-D
 Creation_time = 29-MAR-2017 10:22:26
 Revision_time = 29-MAR-2017 11:26:58
 Current_time = 29-MAR-2017 11:27:02

Content = Exp-AB-diphenylethyl-
 Data_format = 1D COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECA 600
 Spectrometer = DELTA2_NMR

Field_strength = 14.09636928[T] (600[M]
 X_acq_duration = 1.4548992[s]
 X_domain = 1H
 X_freq = 600.1723046[MHz]
 X_offset = 5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.08733284[Hz]
 X_sweep = 11.26126126[MHz]
 Irr_domain = 1H
 Irr_freq = 600.1723046[MHz]
 Irr_offset = 5[ppm]
 Irr_mode = 1H
 Tri_freq = 600.1723046[MHz]
 Tri_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 8
 Total_scans = 8

X_90_width = 12.6[us]
 X_acq_time = 1.4548992[s]
 X_angle = 45[deg]
 X_atn = 3.6[db]
 X_pulse = 6.3[us]
 Irr_mode = OFF
 Irr_offset = OFF
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 44
 Relaxation_delay = 5[s]
 Repetition_time = 6.4548992[s]
 Temp_get = 24.4[dc]

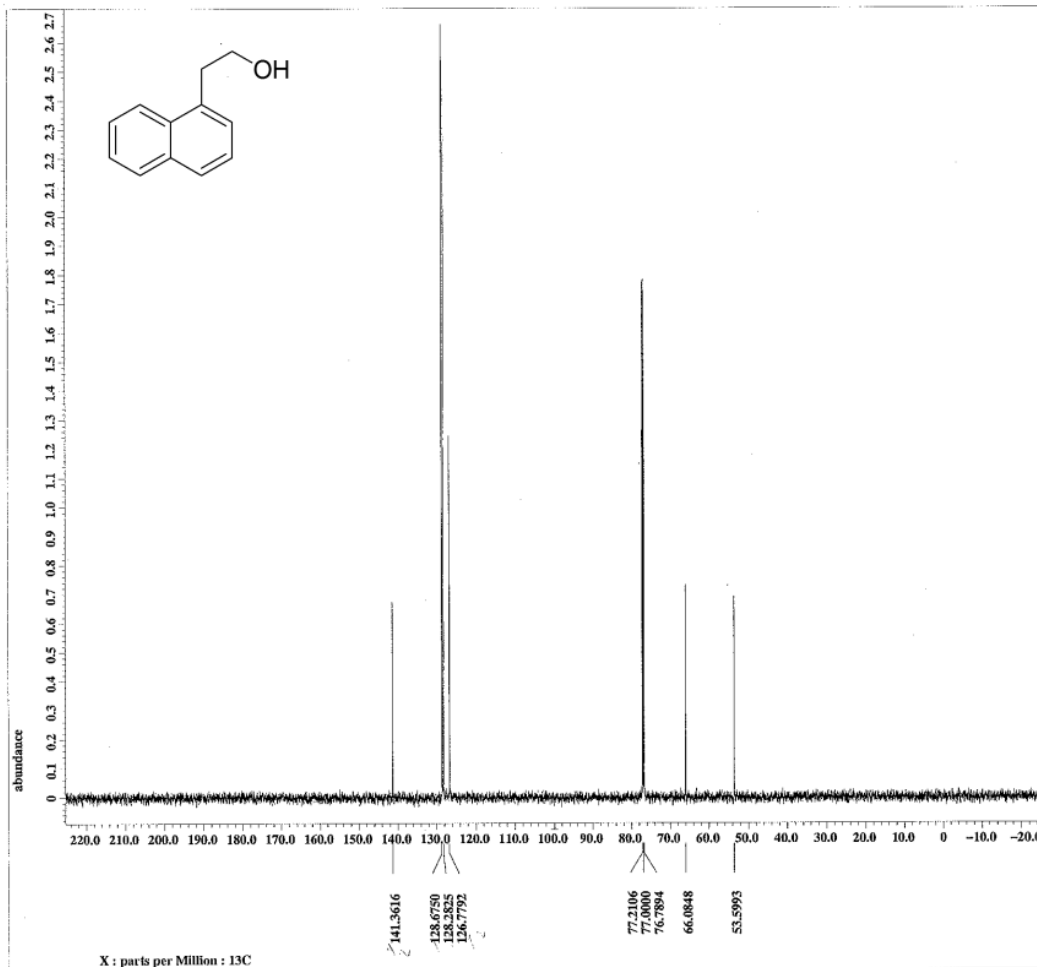


Filename = Exp-AB-diphenylethyl-
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = Exp-AB-diphenylethyl-
 Solvent = CHLOROFORM-D
 Creation_time = 29-MAR-2017 10:27:37
 Revision_time = 29-MAR-2017 11:29:20
 Current_time = 29-MAR-2017 11:29:24

Content = Exp-AB-diphenylethyl-
 Data_format = 1D COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECA 600
 Spectrometer = DELTA2_NMR

Field_strength = 14.09636928[T] (600[M]
 X_acq_duration = 0.69206016[s]
 X_domain = 13C
 X_freq = 150.91343039[MHz]
 X_offset = 100[ppm]
 X_points = 32768
 X_prescans = 4
 X_resolution = 1.44496109[Hz]
 X_sweep = 47.34848485[MHz]
 Irr_domain = 1H
 Irr_freq = 600.1723046[MHz]
 Irr_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 82
 Total_scans = 82

X_90_width = 10.9[us]
 X_acq_time = 0.69206016[s]
 X_angle = 30[deg]
 X_atn = 7.5[db]
 X_pulse = 3.63333333[us]
 Irr_atn_dec = 19.21[db]
 Irr_atn_noe = 19.21[db]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 1[s]
 Noe = TRUE
 Noe_time = 2.5[s]
 Recvr_gain = 60
 Relaxation_delay = 2.5[s]
 Repetition_time = 3.19206016[s]
 Temp_get = 25[dc]

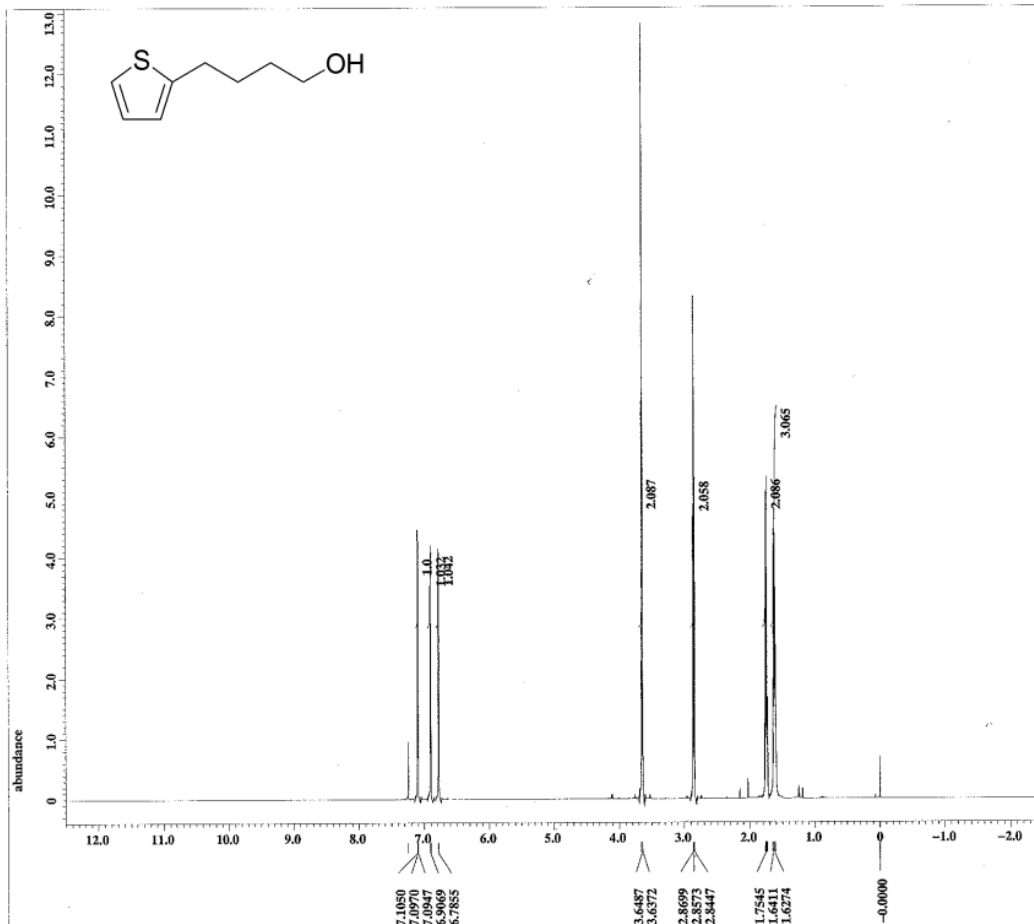


Filename = Exp-AB-OND-Sulpur-Pro
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = Exp-AB-OND-Sulpur-Pro
 Solvent = CHLOROFORM-D
 Creation_time = 24-MAR-2017 16:33:58
 Revision_time = 24-MAR-2017 17:36:49
 Current_time = 24-MAR-2017 17:36:53

Content = Exp-AB-OND-Sulpur-Pro
 Data_format = 1D COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECA 600
 Spectrometer = DELTA2_NMR

Field_strength = 14.09636928[T] (600[M]
 X_acq_duration = 1.4548992[s]
 X_domain = 1H
 X_freq = 600.1723046[MHz]
 X_offset = 5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.69733284[Hz]
 X_sweep = 11.26126126[kHz]
 Irr_domain = 1H
 Irr_freq = 600.1723046[MHz]
 Irr_offset = 5[ppm]
 Tri_domain = 1H
 Tri_freq = 600.1723046[MHz]
 Tri_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 8
 Total_scans = 8

X_90_width = 12.6[us]
 X_acq_time = 1.4548992[s]
 X_angle = 45[deg]
 X_atn = 3.6[db]
 X_pulse = 6.3[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_preset = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 40
 Relaxation_delay = 5[s]
 Repetition_time = 6.4548992[s]
 Temp_get = 25.9[deg]



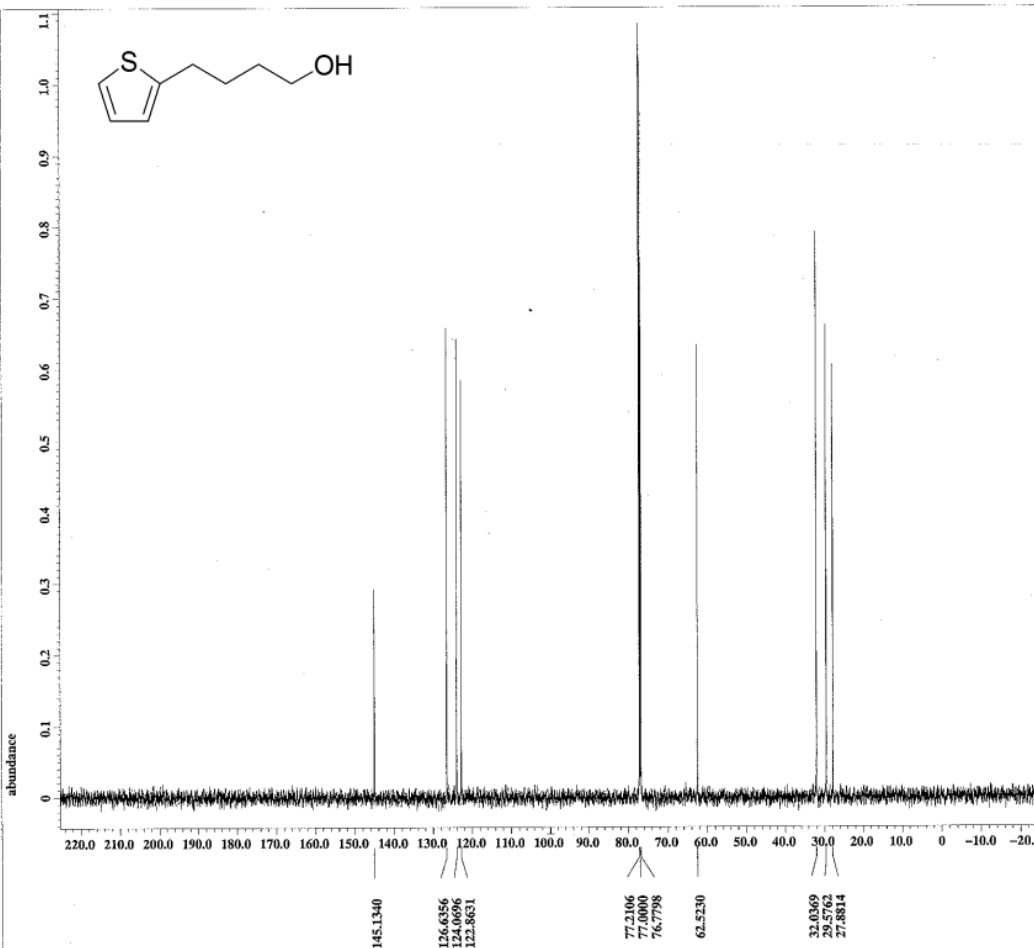
X : parts per Million : 1H

Filename = Exp-AB-OND-Sulpur-car
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = Exp-AB-OND-Sulpur-car
 Solvent = CHLOROFORM-D
 Creation_time = 24-MAR-2017 16:39:12
 Revision_time = 24-MAR-2017 17:40:03
 Current_time = 24-MAR-2017 17:40:07

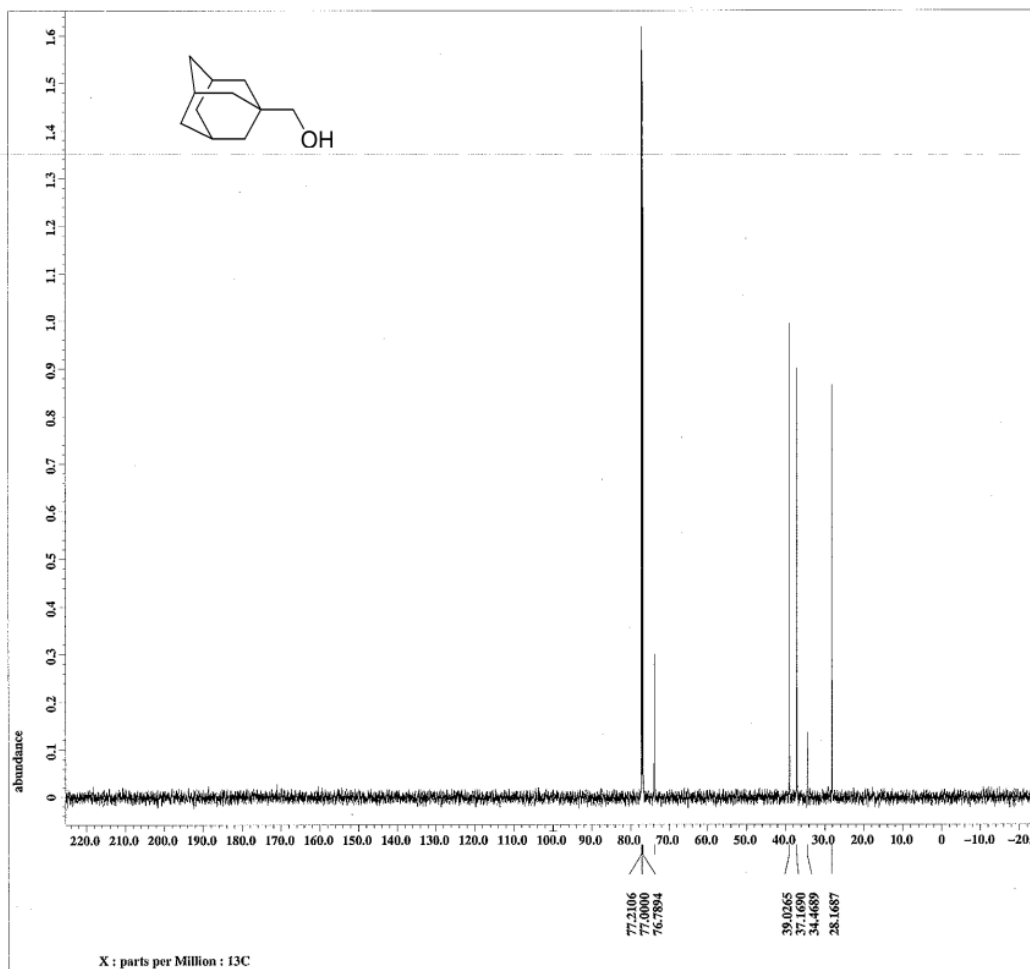
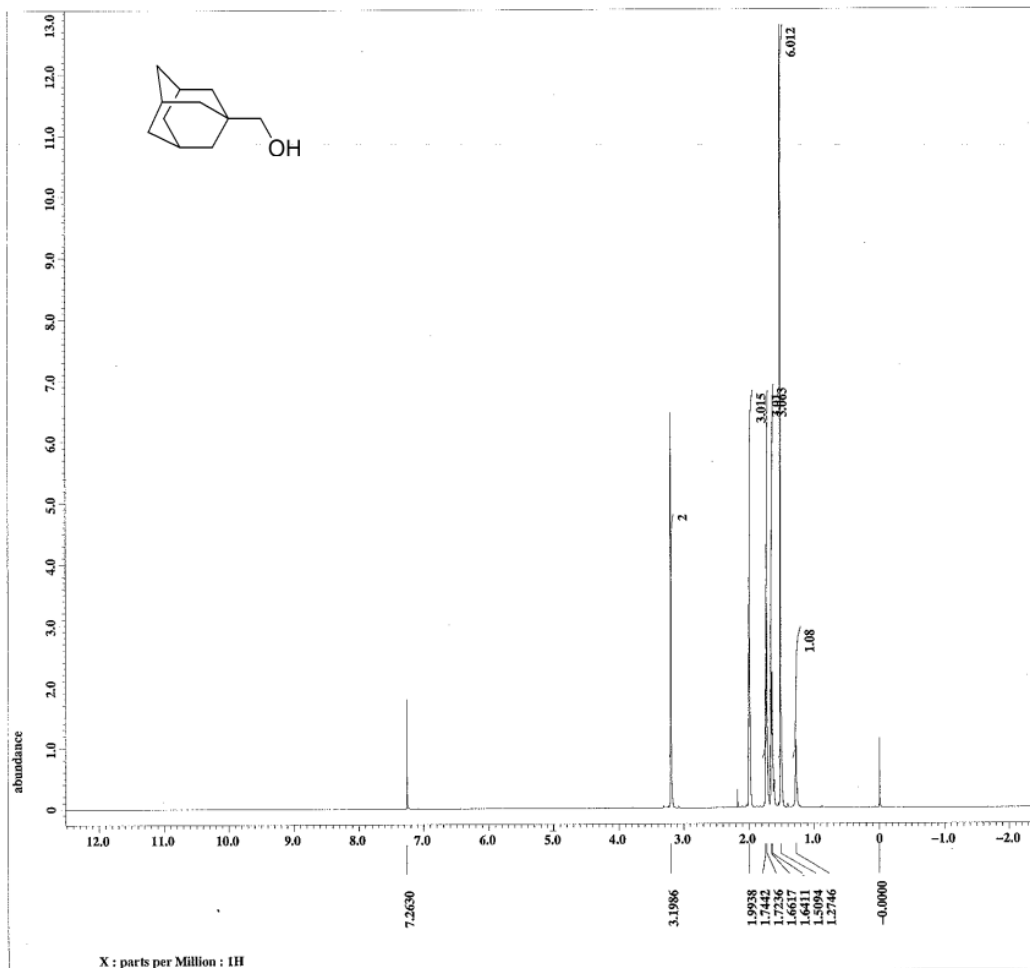
Content = Exp-AB-OND-Sulpur-car
 Data_format = 1D COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECA 600
 Spectrometer = DELTA2_NMR

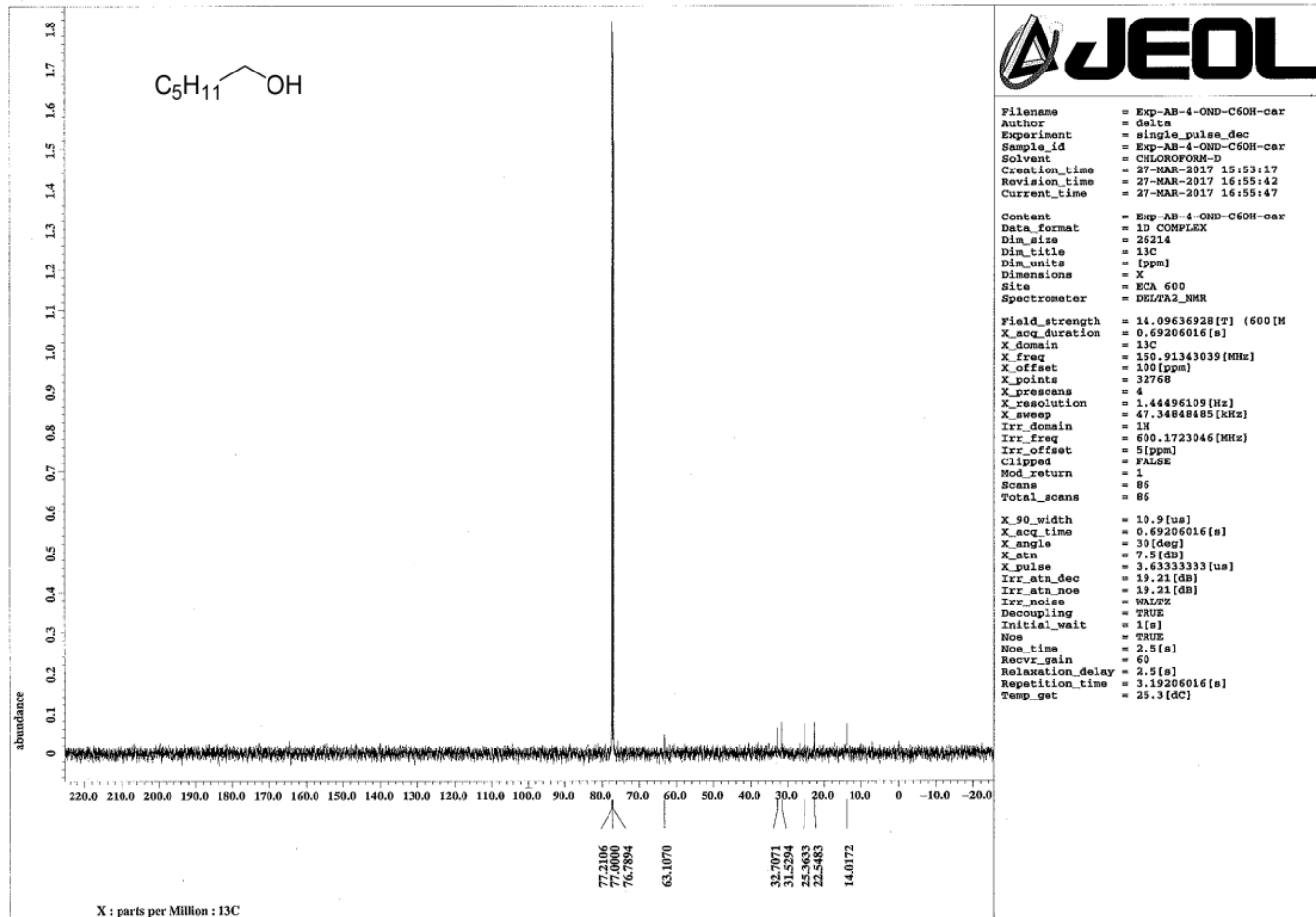
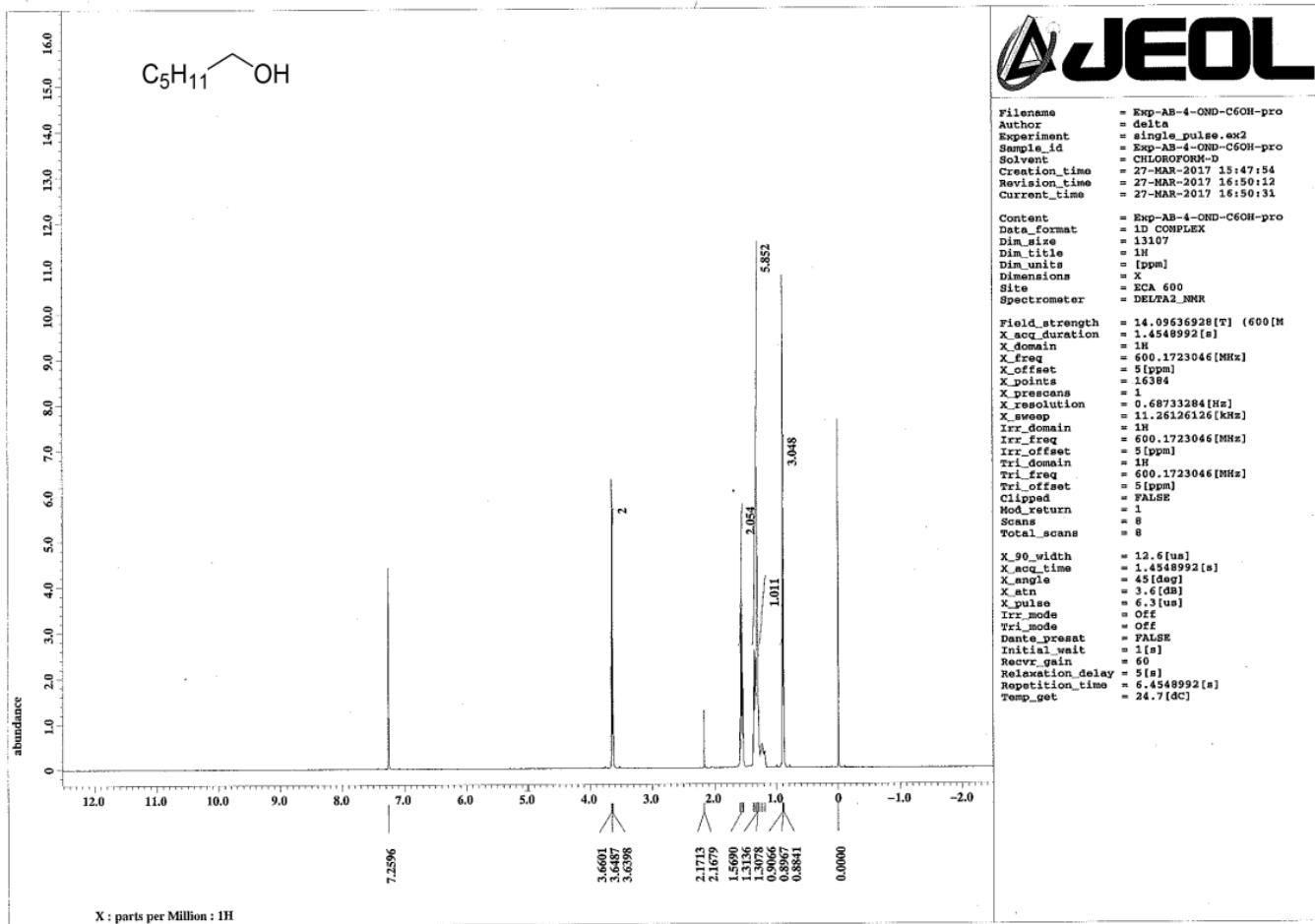
Field_strength = 14.09636928[T] (600[M]
 X_acq_duration = 0.69206016[s]
 X_domain = 13C
 X_freq = 150.91343039[MHz]
 X_offset = 100[ppm]
 X_points = 32768
 X_prescans = 4
 X_resolution = 1.44496109[Hz]
 X_sweep = 47.34848485[kHz]
 Irr_domain = 1H
 Irr_freq = 600.1723046[MHz]
 Irr_offset = 5[ppm]
 Clipped = TRUE
 Mod_return = 1
 Scans = 85
 Total_scans = 85

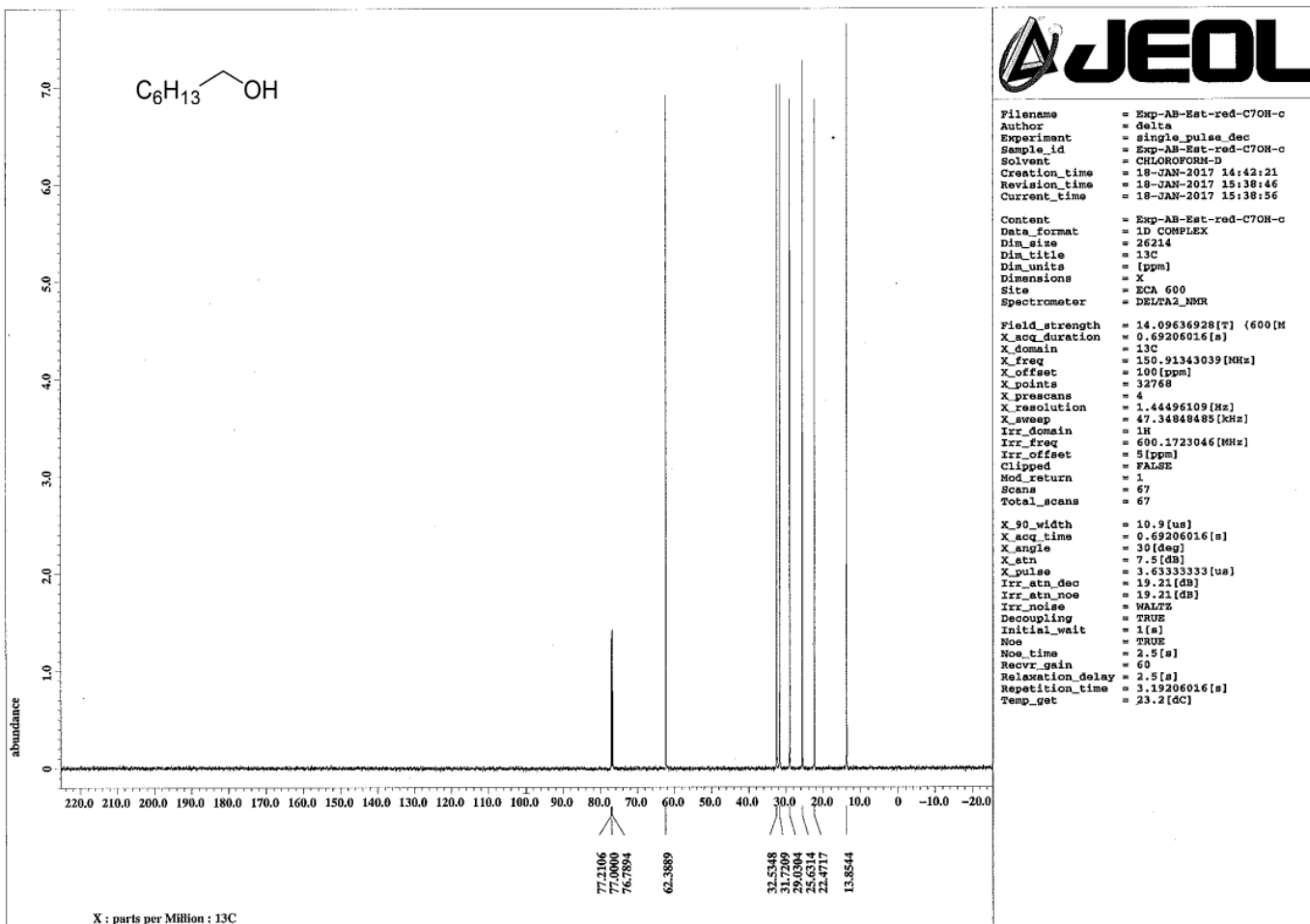
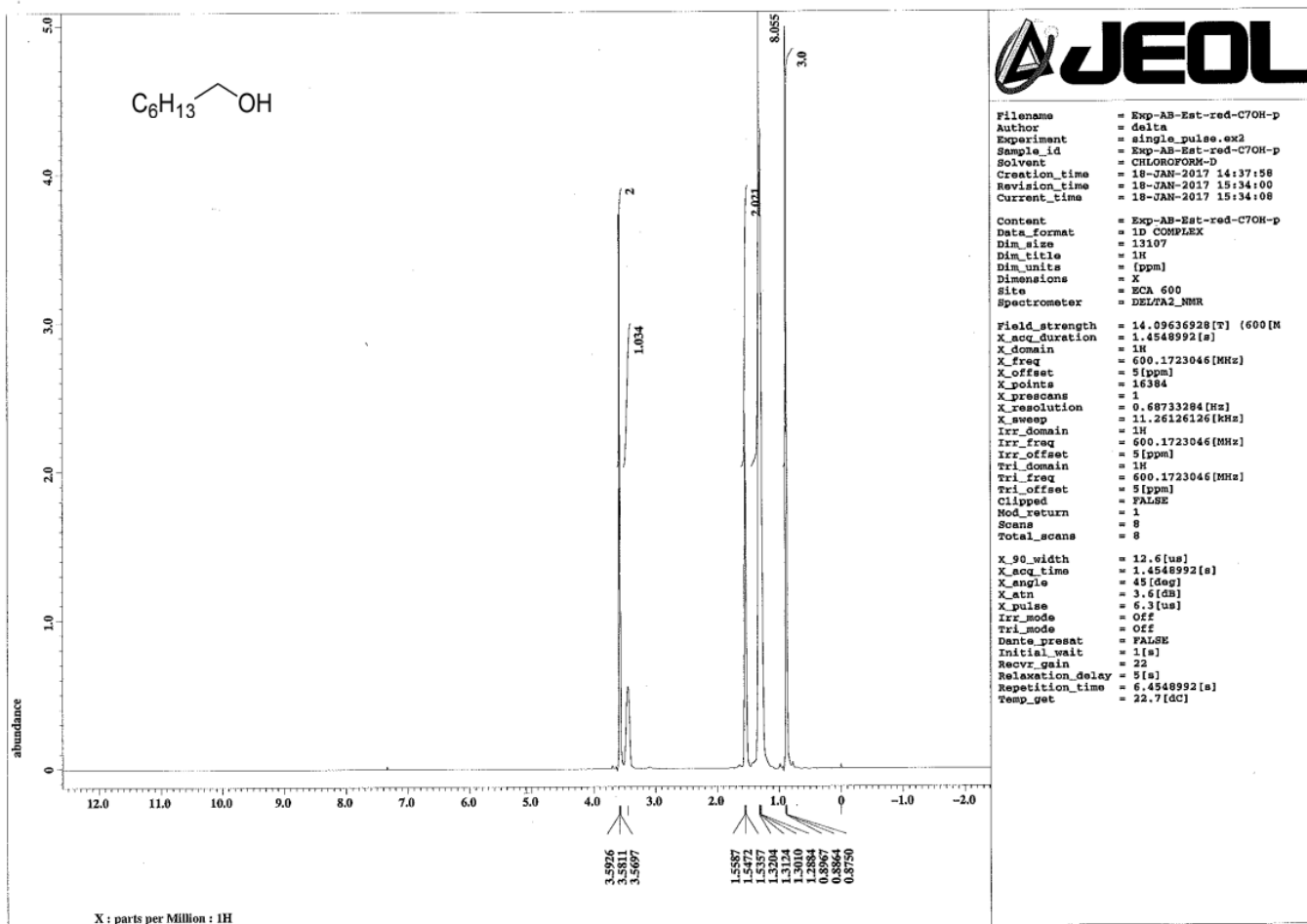
X_90_width = 10.9[us]
 X_acq_time = 0.69206016[s]
 X_angle = 30[deg]
 X_atn = 7.5[db]
 X_pulse = 3.63333333[us]
 Irr_atn_dec = 19.21[db]
 Irr_atn_noe = 19.21[db]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 1[s]
 Noe = TRUE
 Noe_time = 2.5[s]
 Recvr_gain = 56
 Relaxation_delay = 2.5[s]
 Repetition_time = 3.19206016[s]
 Temp_get = 25.7[deg]

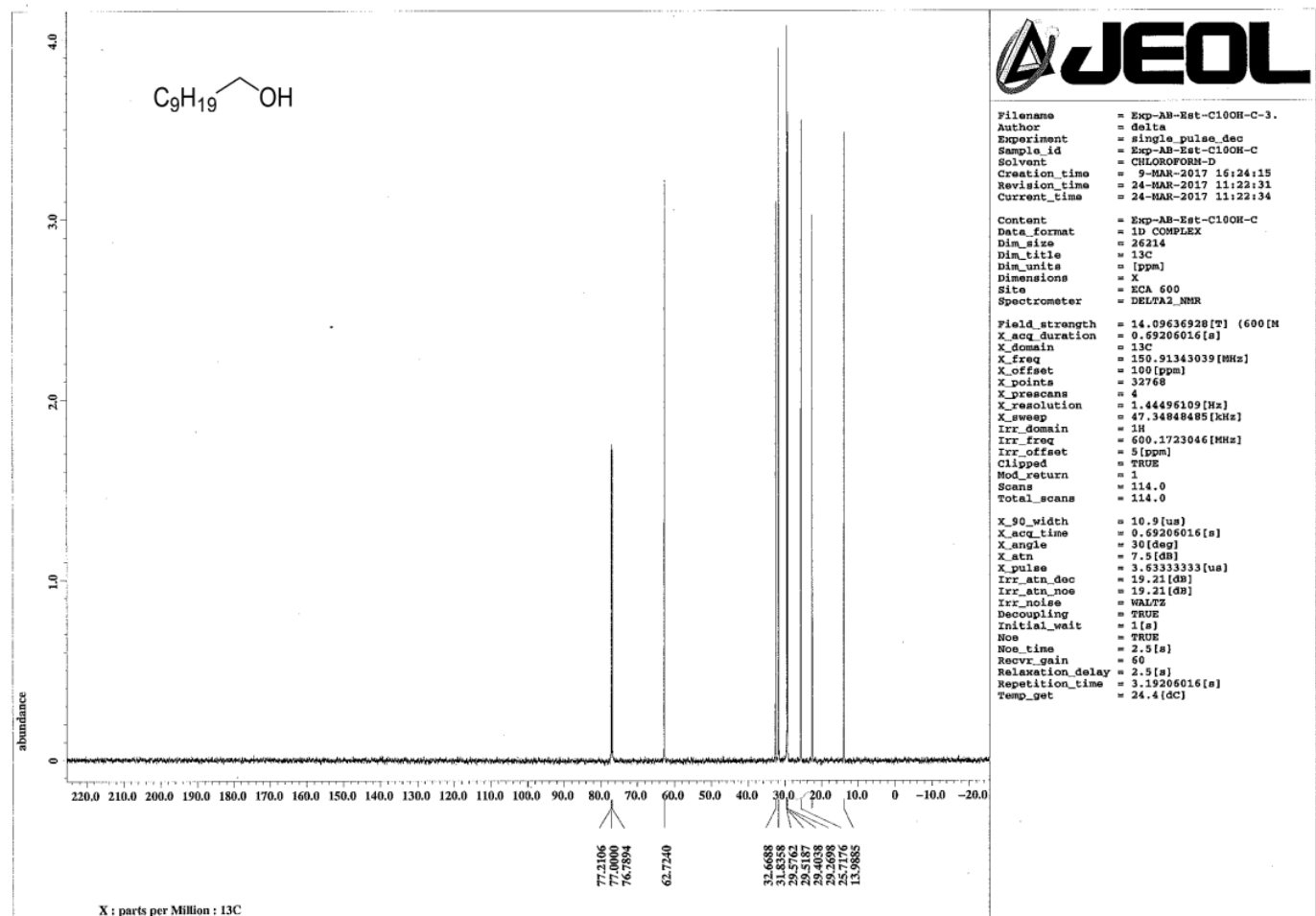
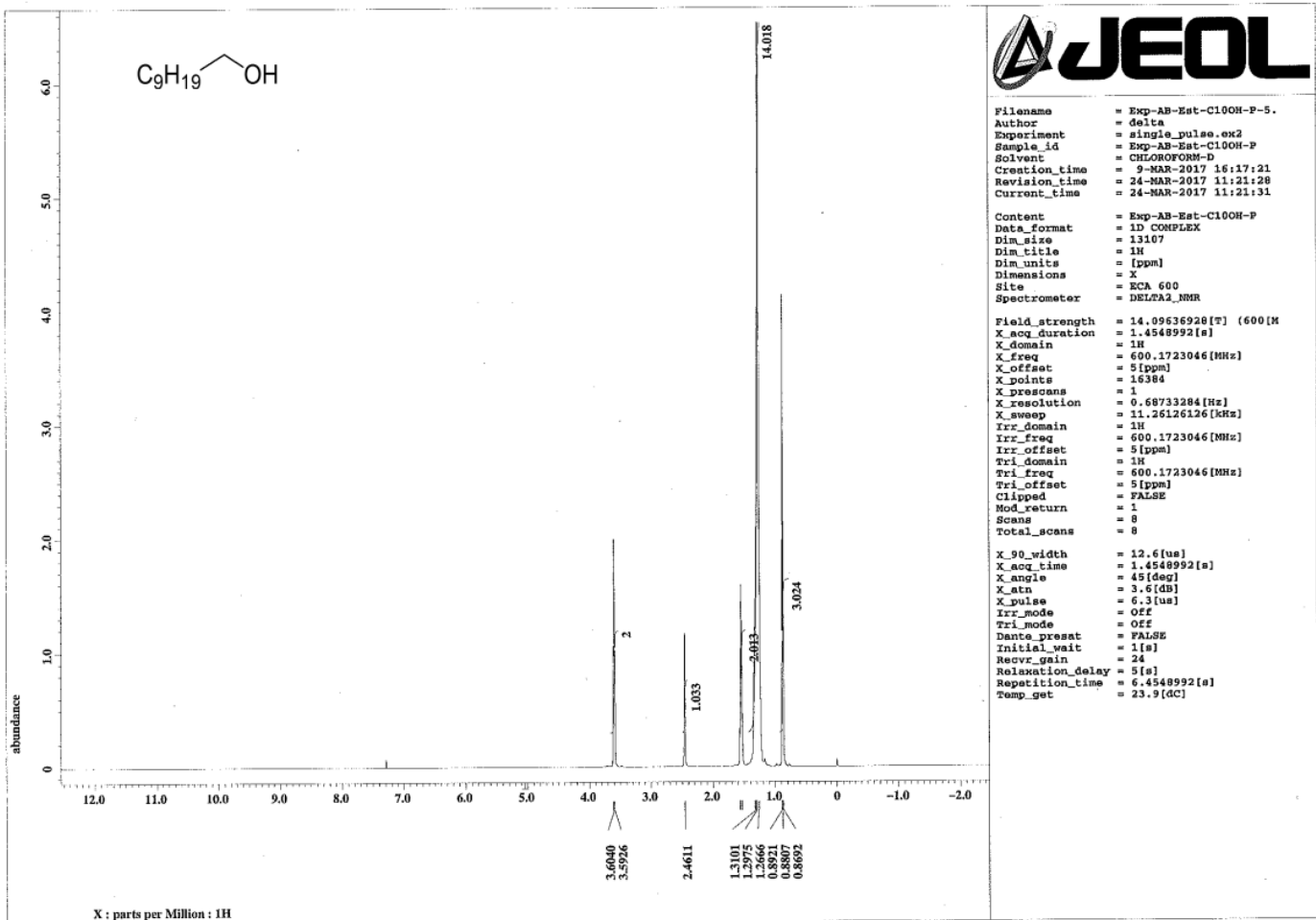


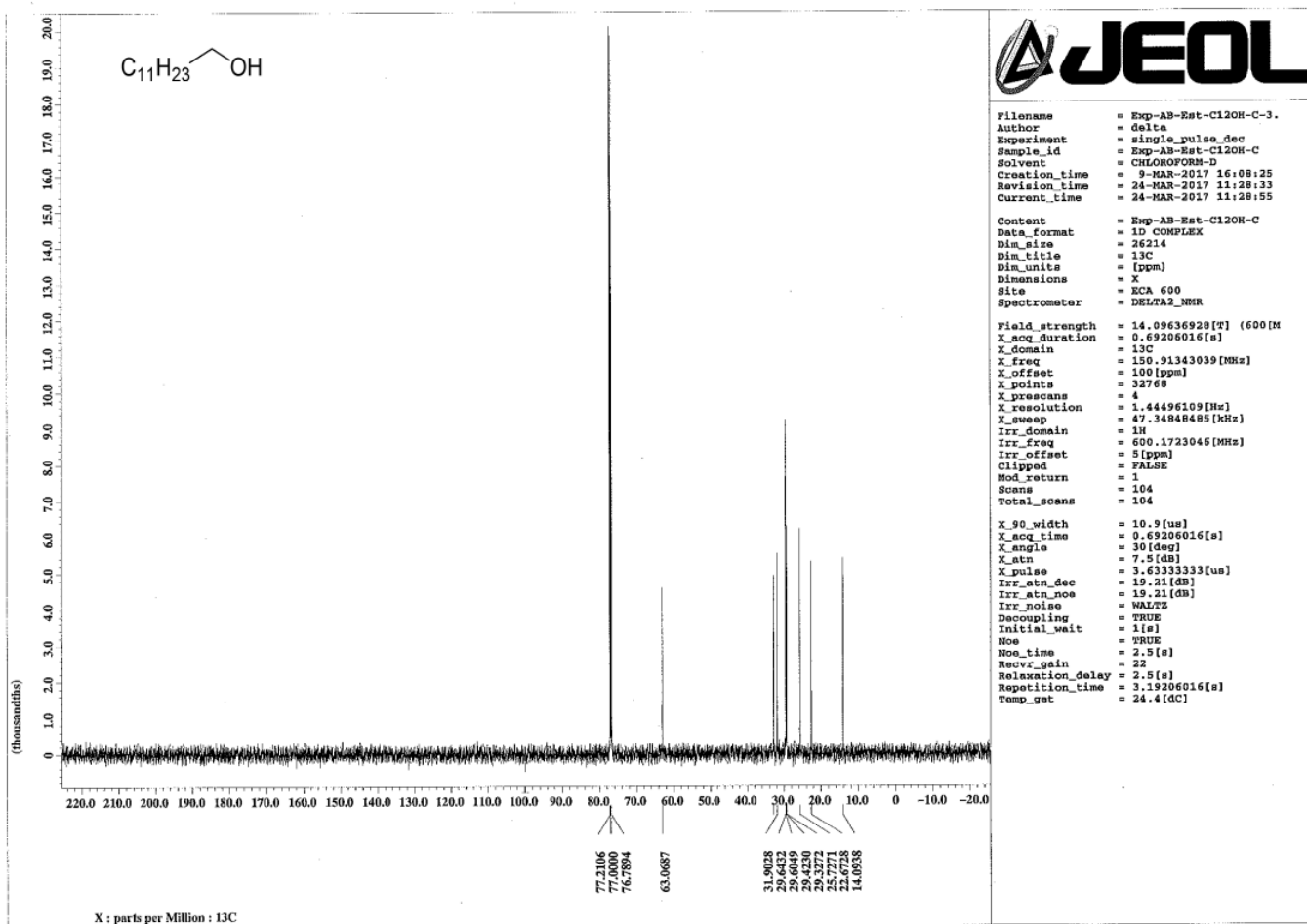
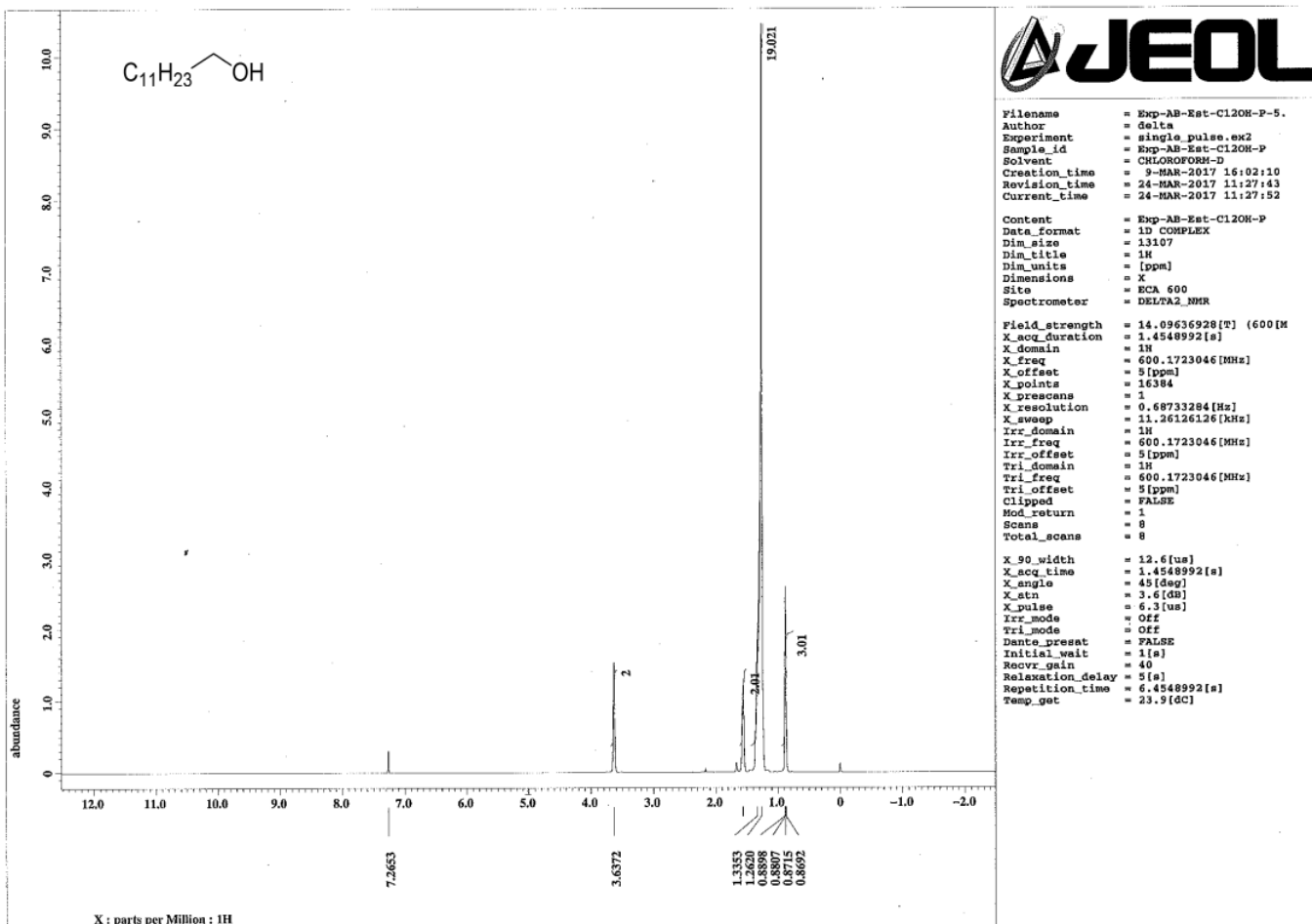
X : parts per Million : 13C

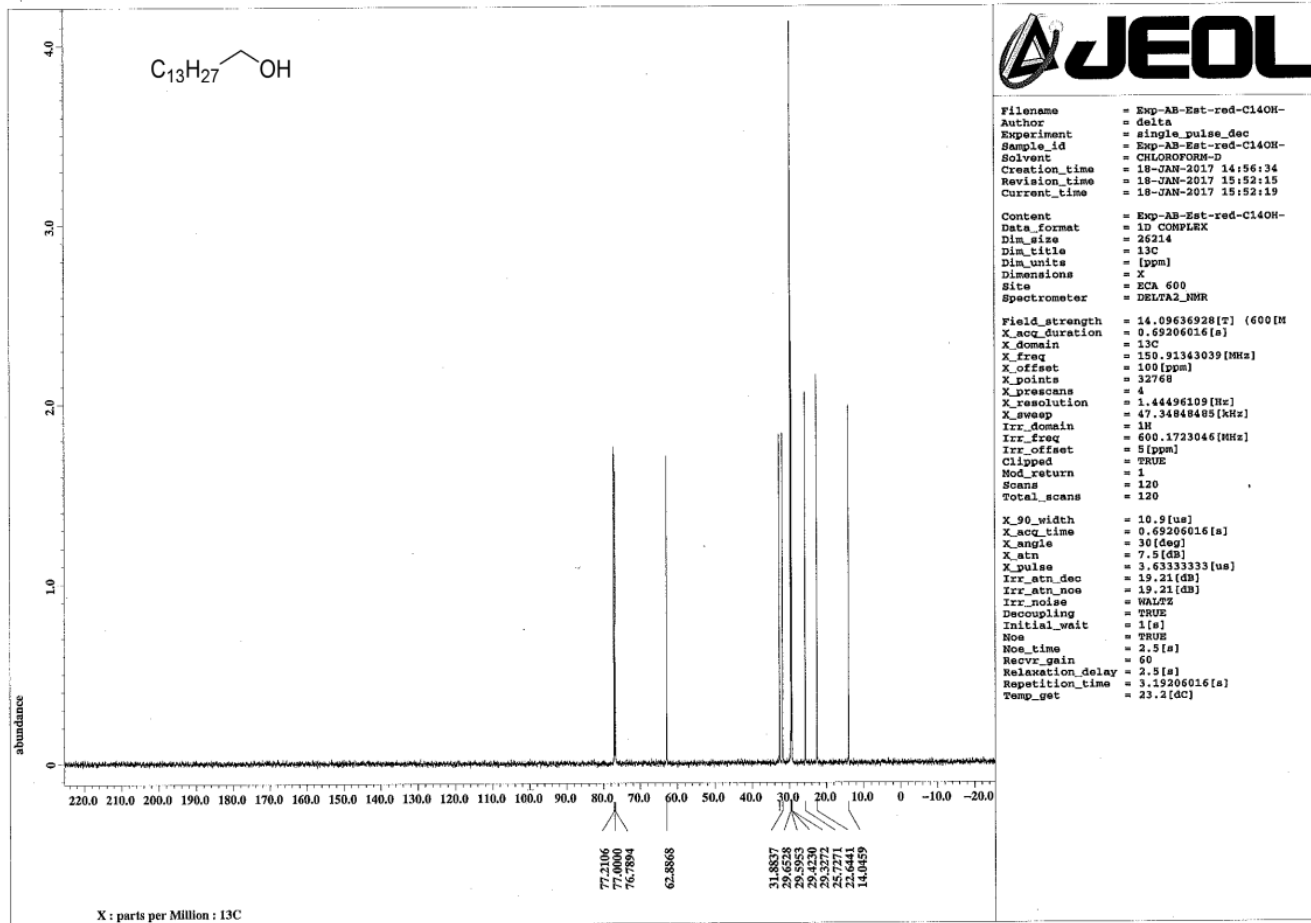
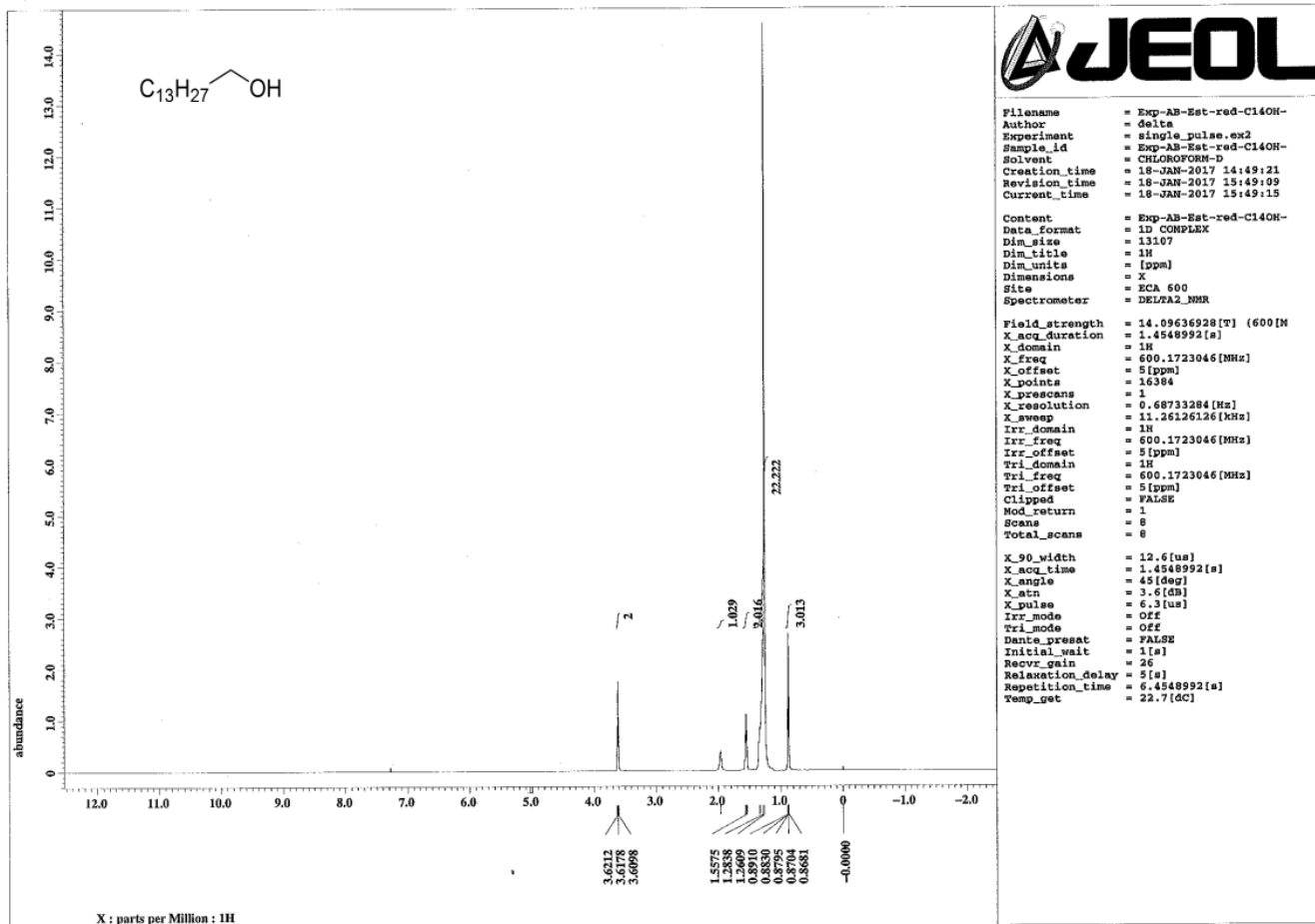


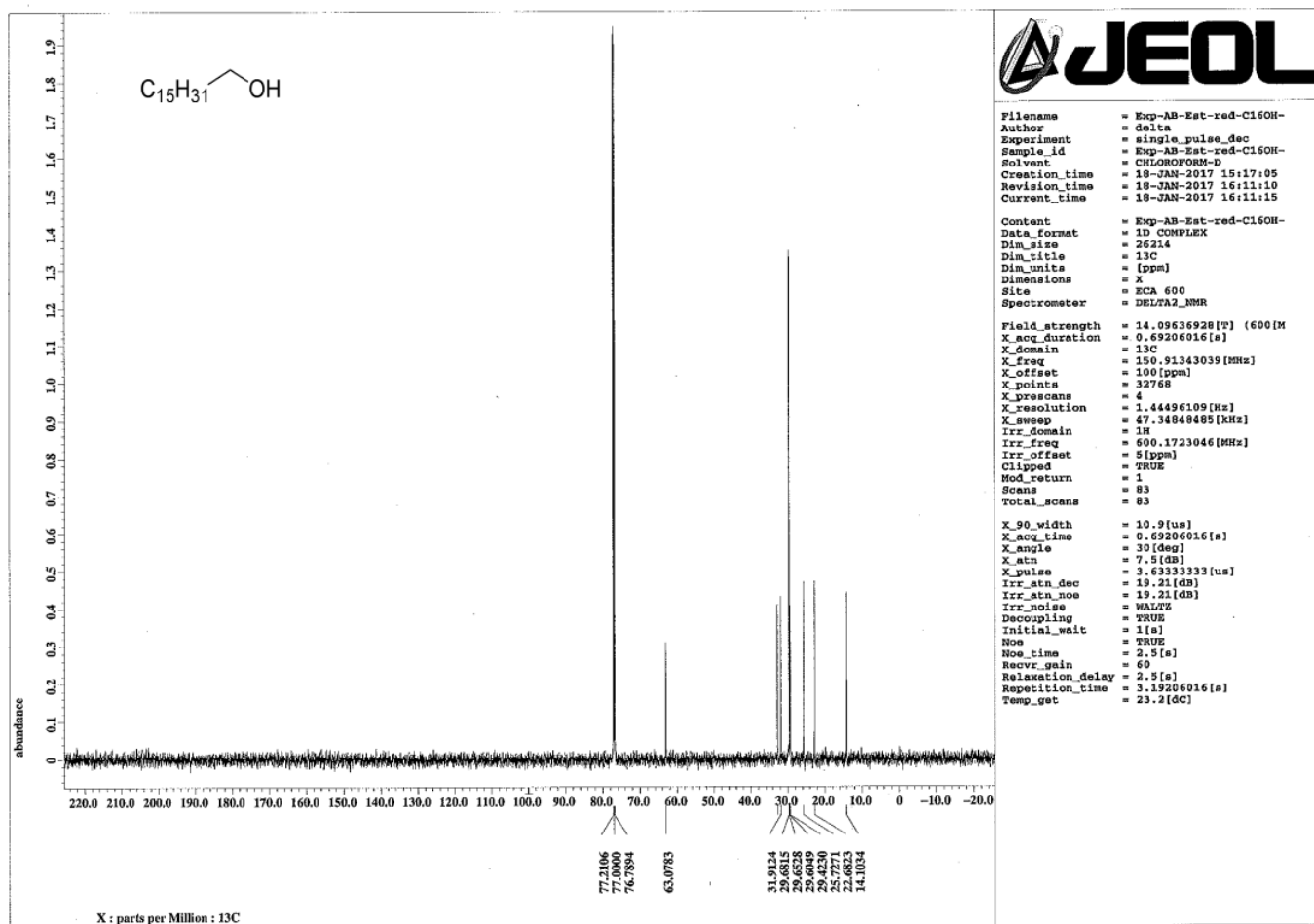
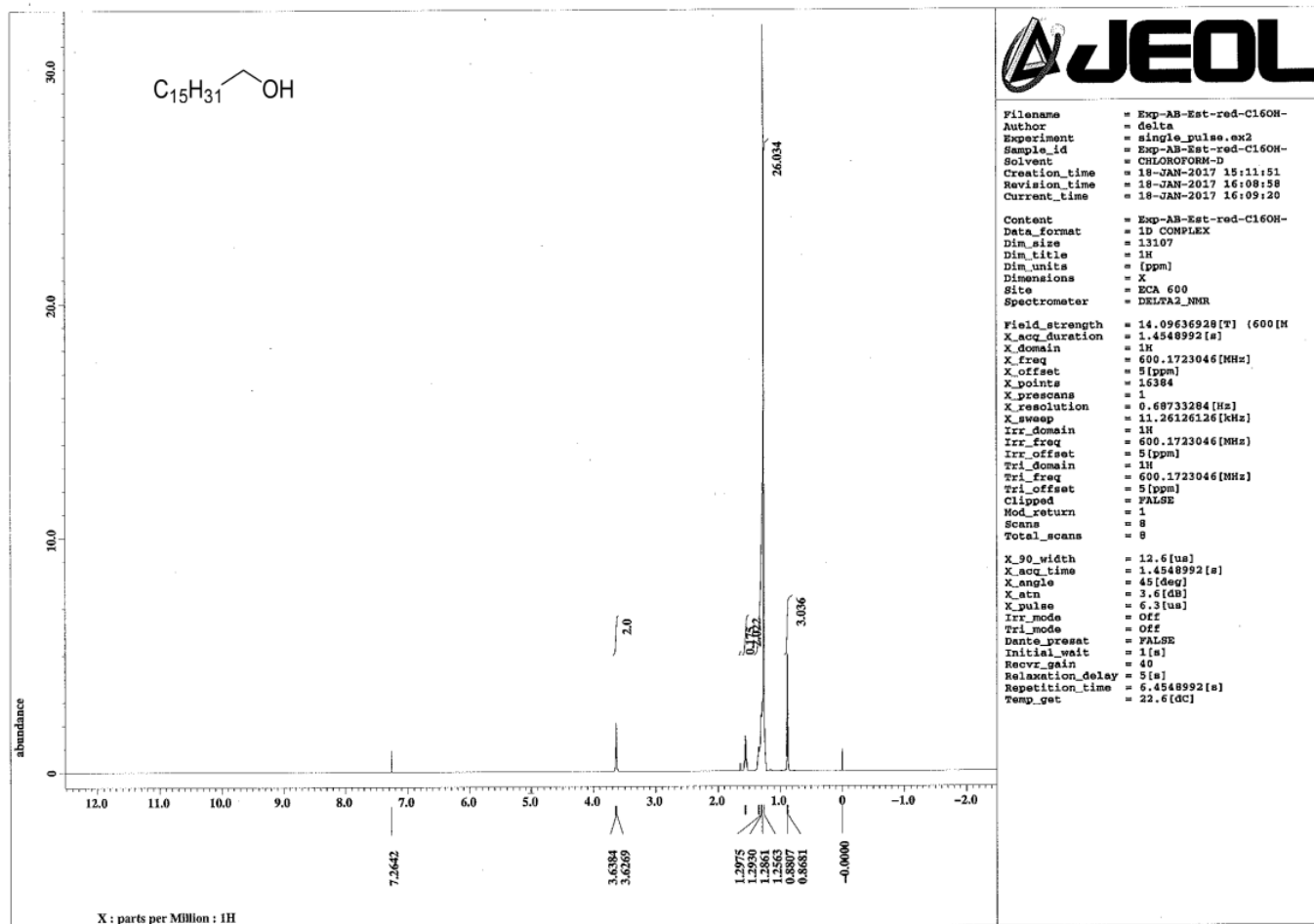


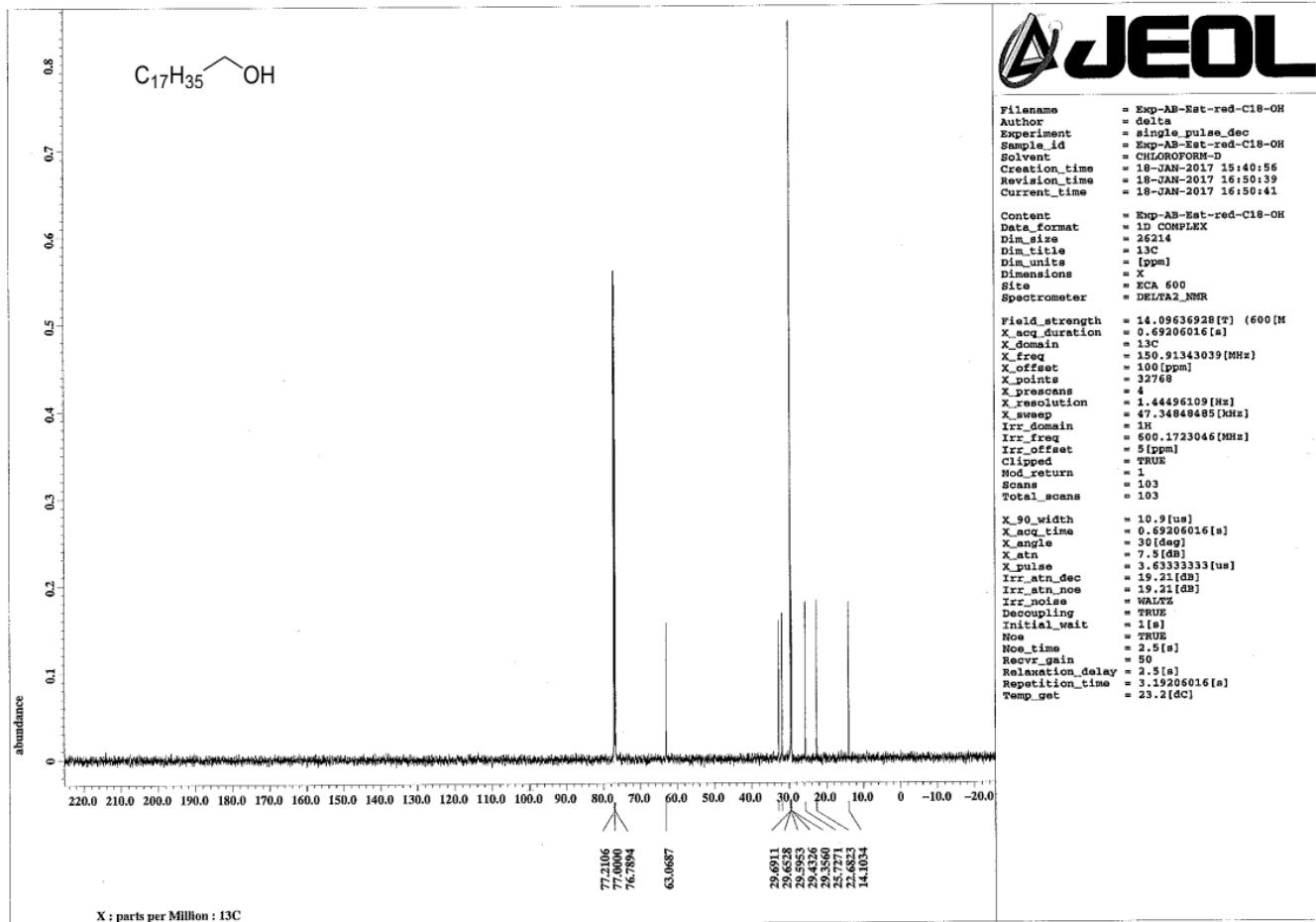
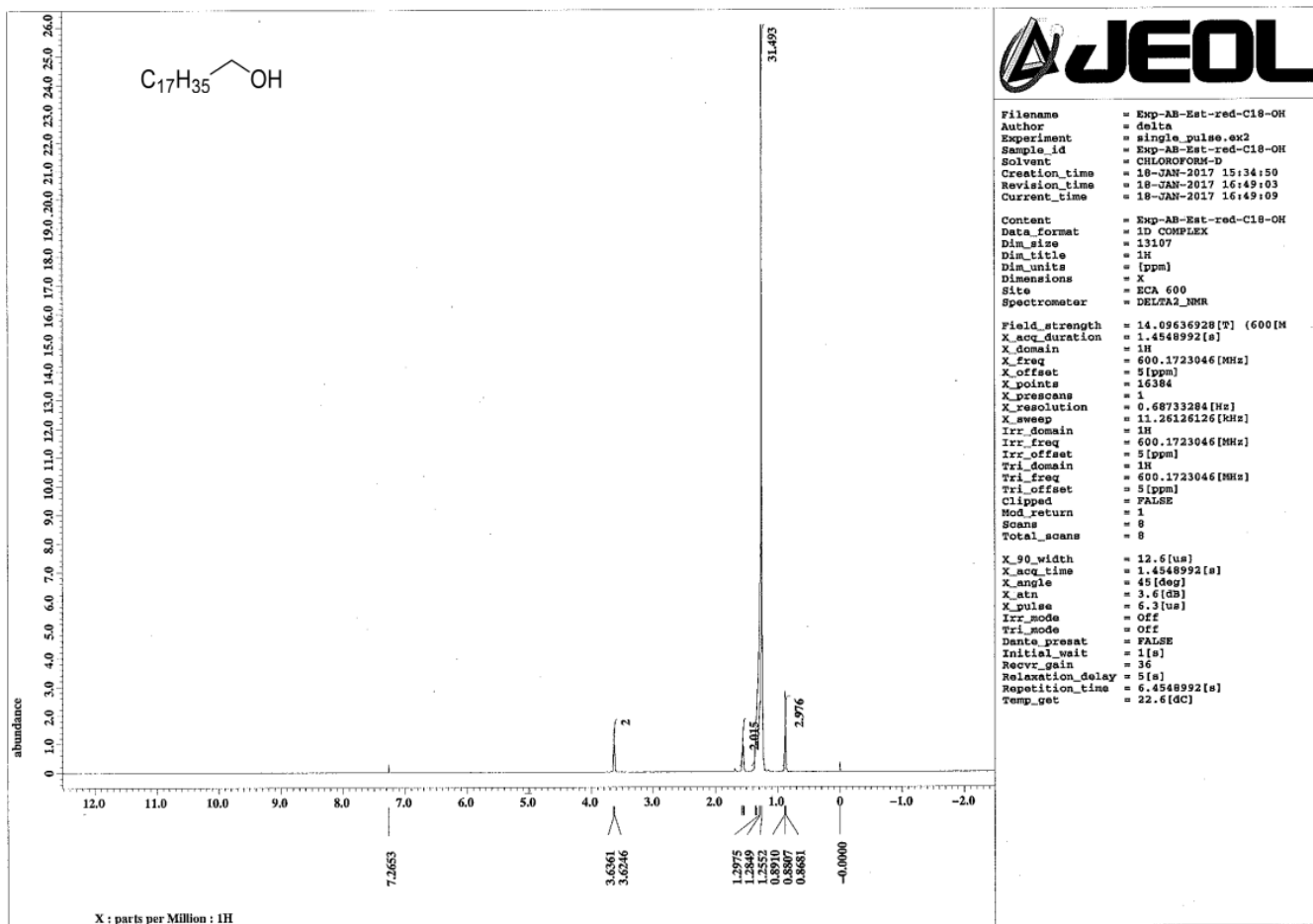


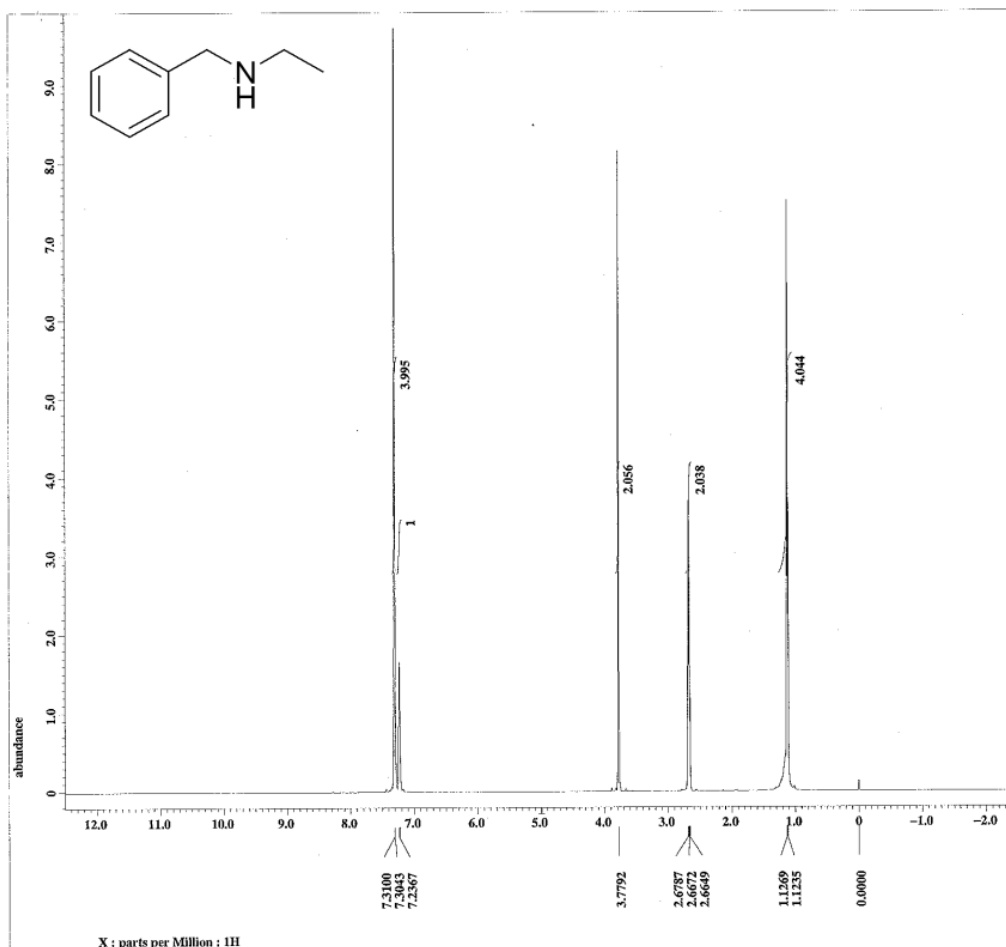












JEOL

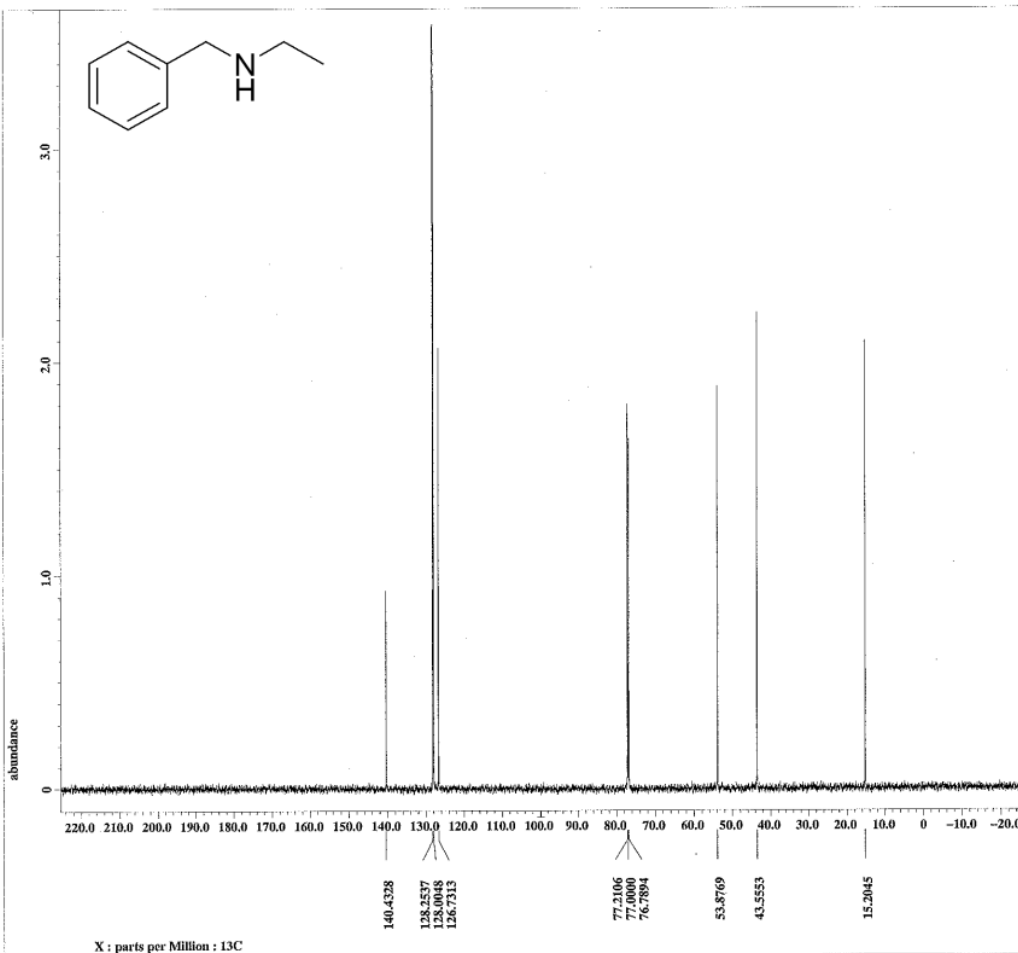
```

Filename      = Exp-AB-amide-amine-P-
Author        = delta
Experiment     = single_pulse.ex2
Sample_id     = Exp-AB-amide-amine-P
Solvent       = CHLOROFORM-D
Creation_time  = 11-APR-2017 12:11:30
Revision_time = 11-APR-2017 13:10:16
Current_time  = 11-APR-2017 13:10:22

Content       = Exp-AB-amide-amine-P
Data_format   = 1D COMPLEX
Dim_size      = 13107
Dim_title     = 1H
Dim_units     = [ppm]
Dimensions    = X
Site          = KCA 600
Spectrometer  = DELTA2_NMR

Field_strength = 14.09636928[T] (600[M]
X_acq_duration = 1.4548992[s]
X_domain       = 1H
X_freq         = 600.1723046[MHz]
X_offset       = 5[ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.68733284[Mz]
X_sweep        = 11.26126126[kHz]
Irr_domain     = 1H
Irr_freq       = 600.1723046[MHz]
Irr_offset     = 5[ppm]
Tri_domain     = 1H
Tri_freq       = 600.1723046[MHz]
Tri_offset     = 5[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 8
Total_scans    = 8

X_90_width     = 12.6[us]
X_acq_time      = 1.4548992[s]
X_angle         = 45[deg]
X_atn           = 3.6[db]
X_pulse         = 6.3[us]
Irr_mode        = Off
Tri_mode        = Off
Dante_preset    = FALSE
Initial_wait    = 1[s]
Recvr_gain      = 14
Relaxation_delay = 5[s]
Repetition_time = 6.4548992[s]
Temp_get        = 20.6[dc]
  
```



JEOL

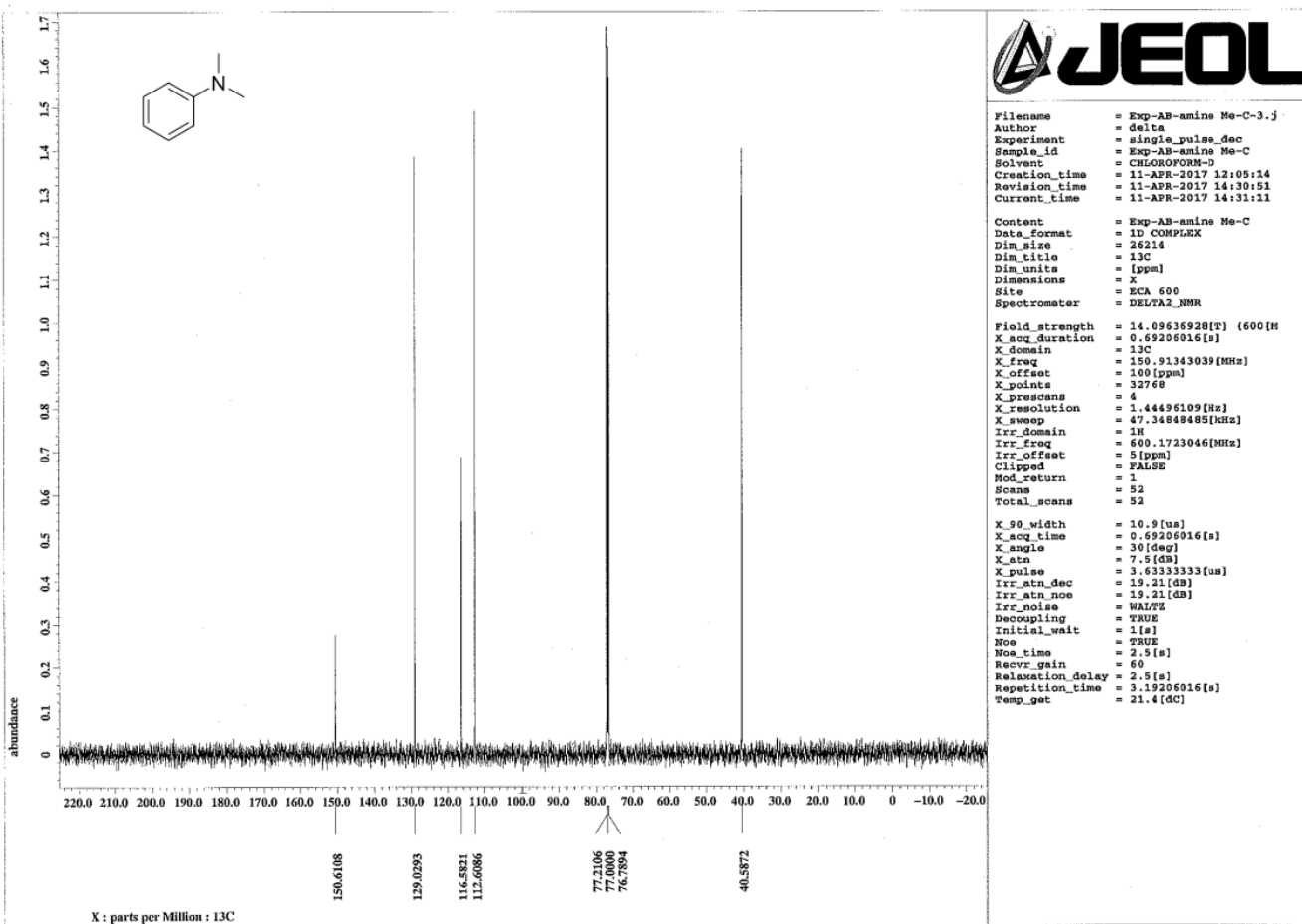
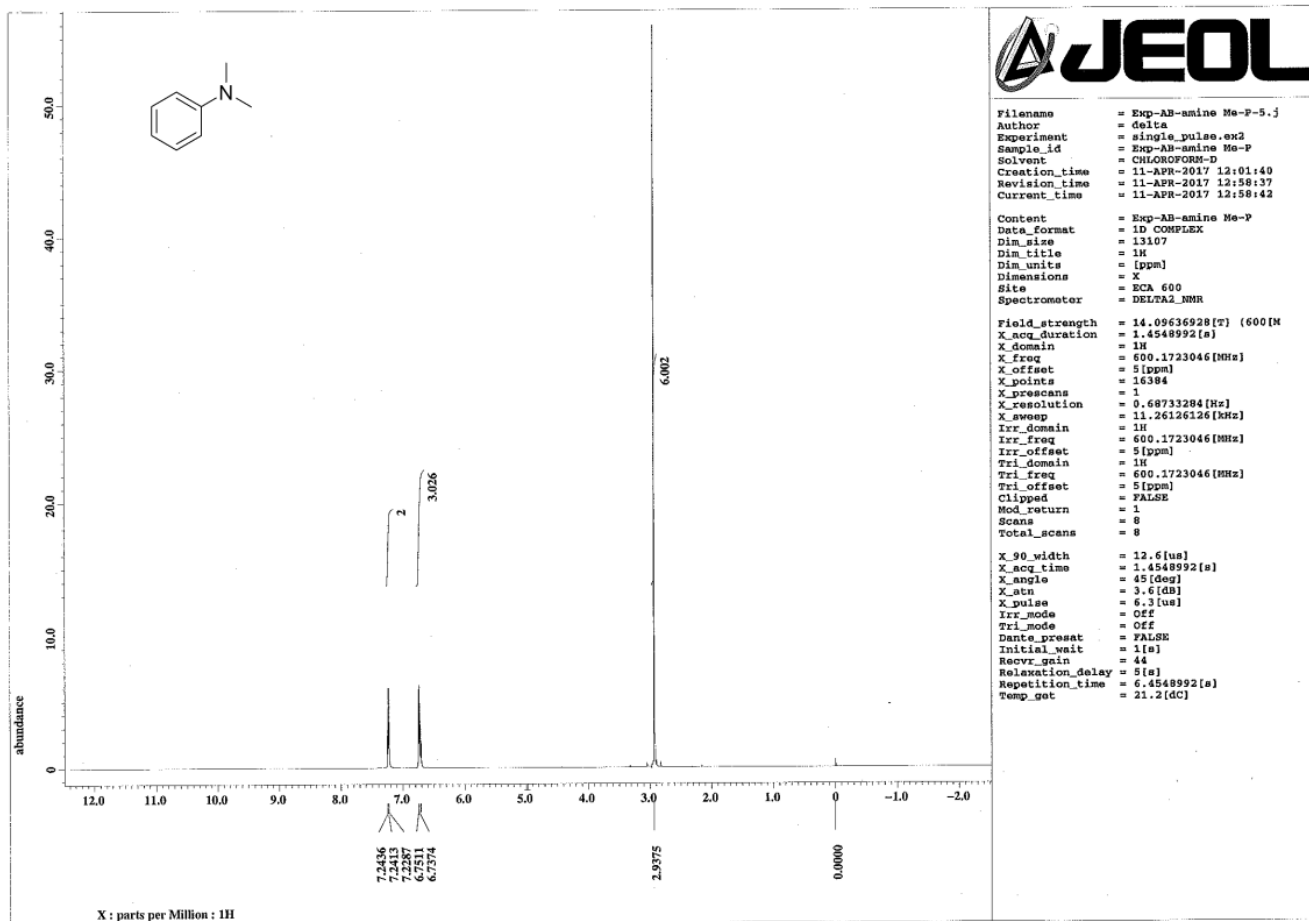
```

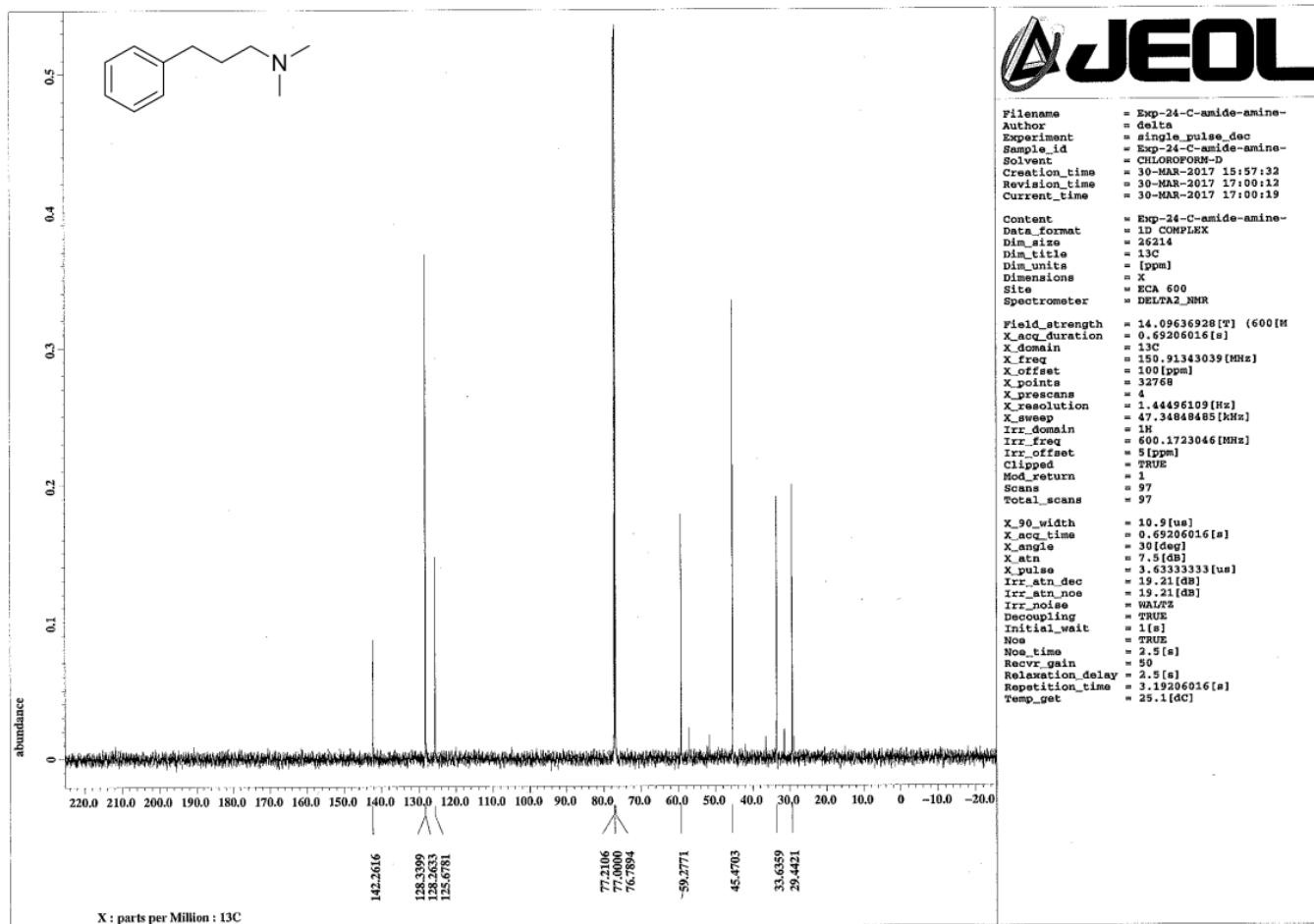
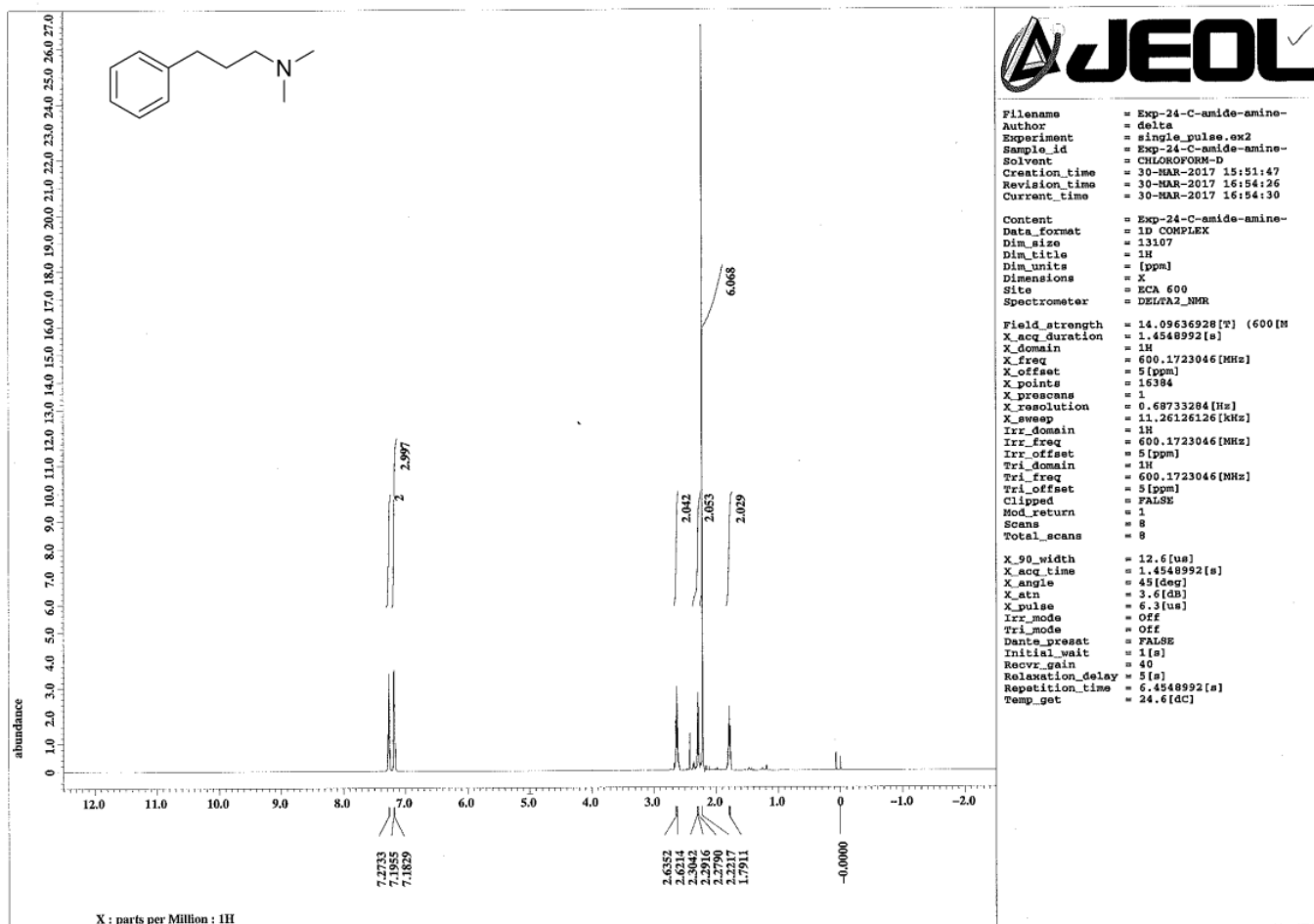
Filename      = Exp-AB-amide-amine-C-
Author        = delta
Experiment     = single_pulse_dec
Sample_id     = Exp-AB-amide-amine-C
Solvent       = CHLOROFORM-D
Creation_time  = 11-APR-2017 12:16:35
Revision_time = 11-APR-2017 13:11:48
Current_time  = 11-APR-2017 13:11:52

Content       = Exp-AB-amide-amine-C
Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = KCA 600
Spectrometer  = DELTA2_NMR

Field_strength = 14.09636928[T] (600[M]
X_acq_duration = 0.69206016[s]
X_domain       = 13C
X_freq         = 150.91343039[MHz]
X_offset       = 100[ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.44496109[Mz]
X_sweep        = 47.34848485[kHz]
Irr_domain     = 1H
Irr_freq       = 600.1723046[MHz]
Irr_offset     = 5[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 80
Total_scans    = 80

X_90_width     = 10.9[us]
X_acq_time      = 0.69206016[s]
X_angle         = 30[deg]
X_atn           = 7.5[db]
X_pulse         = 3.63333333[us]
Irr_atn_dec     = 19.21[db]
Irr_atn_noe     = 19.21[db]
Irr_noise       = WALFE
Decoupling      = TRUE
Initial_wait    = 1[s]
Noe             = TRUE
Noe_time        = 2.5[s]
Recvr_gain      = 60
Relaxation_delay = 2.5[s]
Repetition_time = 3.19206016[s]
Temp_get        = 21.7[dc]
  
```





4. References

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