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Title	TiO2-Supported Re as a General and Chemoselective Heterogeneous Catalyst for Hydrogenation of Carboxylic Acids to Alcohols
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Citation	Chemistry-A European journal, 23(5), 1001-1006 https://doi.org/10.1002/chem.201604762
Issue Date	2017-01-23
Doc URL	https://hdl.handle.net/2115/67661
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Type	journal article
File Information	Supporting Information_Chem Eur J_2017.pdf, Supporting Information



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SUPPORTING INFORMATION

TiO_2-Supported Re as a General and Chemoselective Heterogeneous Catalyst for Hydrogenation of Carboxylic Acids to Alcohols

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1. Experimental and computational methods

1. 1. Materials and Catalyst Preparation

Commercially available organic and inorganic compounds (Tokyo Chemical Industry, Kanto Chemical, Wako Pure Chemical Industries, Nacalai Tesque and Aldrich) were used without further purification. TiO_2 (JRC-TIO-8, 188 $m^2\ g^{-1}$), MgO (JRC-MGO-3, 19 $m^2\ g^{-1}$), SiO_2-Al_2O_3 (JRC-SAL-2, Al_2O_3 = 13.75 wt%, 560 $m^2\ g^{-1}$) and HBEA zeolite (SiO_2/Al_2O_3 = 25±5, JRC-Z-HB25) were supplied by the Catalysis Society of Japan. γ-Al_2O_3 (124 $m^2\ g^{-1}$) was prepared by calcination of γ-AlOOH (Catapal B Alumina from Sasol) at 900 °C for 3 h. SiO_2 (Q-10, 300 $m^2\ g^{-1}$) was supplied by Fuji Silysia Chemical Ltd. Nb_2O_5 (54 $m^2\ g^{-1}$) was prepared by calcination of niobic acid (CBMM) at 500 °C for 3 h. The standard carbon support, Vulcan XC72 (210 $m^2\ g^{-1}$), was commercially supplied. ZrO_2 was prepared by calcination of a hydroxide of Zr at 500 °C for 3 h. SnO_2 was prepared by calcination of H_2SnO_3 (Kojundo Chemical Laboratory Co., Ltd.) at 500 °C for 3 h. NH_4ReO_4 and metallic Re were purchased from Aldrich. ReO_2 and Re_2O_7 were supplied from Hydrus Chemical Inc. and Strem Chemicals Inc., respectively.

Precursors of M/TiO_2 and (M = 5 wt% Re, Pt, Ir, Rh, Ru, Pd, Ag, Cu, Ni and Co) and Re/MOx (5 wt% Re; MOx = metal oxides, zeolites or carbon) were prepared by mixing support material with metal sources, an aqueous solution of NH_4ReO_4, nitrates of Ag, Ni, Cu, Co, $RuCl_3$ or $IrCl_3 \cdot nH_2O$, or aqueous HNO_3 solutions of $Pt(NH_3)_2(NO_3)_2$, $Rh(NO_3)_3$ or $Pd(NH_3)_2(NO_3)_2$. Typically, for the preparation of Re/TiO_2, 0.72 g of NH_4ReO_4 was added to a glass vessel (500 ml) with 100 ml of deionized water (concentration of Re = 0.027 mol/l). After sonication for 1 minute to completely dissolve NH_4ReO_4, TiO_2 (9.5 g) was added to the solution and kept stirring for 15 min with 200 rpm at room temperature. This was followed by evaporation to dryness at 50 °C, and by drying at 90 °C under ambient pressure for 12 h. The

prepared material was calcined at 500 °C in air for 3 h. For each experiment, the active catalysts were prepared by reduction in a Pyrex tube under a flow of H_2 (20 cm^3 min^{-1}) at 500 °C for 0.5 h. The other M/TiO_2 and Re/MOx catalysts were prepared in the same manner described above.

1. 2. Catalyst Characterization

Temperature programmed reduction by H_2 (H_2-TPR) was performed by using BELCAT (MicrotracBEL). A sample was mounted in a quartz tube and then heated at a temperature ramp-rate of 10 °C min^{-1} in a flow of 5% H_2/Ar (20 cm^3 min^{-1}). The effluent gas was passed through a trap containing MS4Å to remove water and then through the thermal conductivity detector, which detected the amount of H_2 consumed during the process.

X-ray diffraction (XRD; Rigaku Miniflex) measurements were conducted using CuKa radiation. Transmission electron microscopy (TEM) was measured using a JEOL JEM-2100F TEM operated at 200 kV. A scanning transmission electron microscope (STEM, Hitachi, HD2000 ultrathin film evaluation system) equipped with energy dispersive X-ray spectroscopy (EDX) system (EDAX Genesis series) was operated at 200 kV. Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) analysis was carried out by a SHIMADZU ICPE-9000 instrument.

1. 3. Catalytic Reactions

Typical procedures for hydrogenation reactions are as follows. After H_2-reduction at 500 °C (See catalysts preparations), the catalyst (2 mol% on the basis of the Re loading amount), and a mixture of 3-phenylpropionic acid (1.0 mmol) and *n*-dodecane (0.29 mmol) were added to a stainless steel autoclave (10 cm^3). The resulting mixture was magnetically stirred at 140 °C

under 5 MPa atmosphere of H_2. The product yields were determined by using GC analysis (Shimadzu GC-14B with Ultra ALLOY capillary column UA$^+$-1 of Frontier Laboratories Ltd., N_2) and *n*-dodecane as an internal standard. The products were isolated by using column chromatography on silica gel 60 (spherical, 63-210 μm, Kanto Chemical Co. Ltd.) with hexane/ethylacetate (90/10) as the eluting solvent and then analyzed by using GC, GCMS (SHIMADZU GCMS-QP2010) and ^{1}H and ^{13}C. ^{1}H and ^{13}C NMR spectra were recorded at ambient temperature using JEOL-ECX 600 operating at 600.17 and 150.92 MHz, respectively with tetramethylsilane as an internal standard. All the yields were calculated based on the starting substrates (carboxylic acid).

Reactions for recycling experiments were performed in a similar manner described above except that 3 ml of octane was used as a solvent and H_2 reduction was conducted at 700 °C. Following the reaction, carried out using 5 MPa H_2 at 140 ºC and 24 h, the catalyst was separated, washed with isopropanol, dried in air and then reused for an ensuing reaction. Note that the recycling reaction was carried out after H_2 reduction as is the case with the 1st run. A leaching test was performed in the same manner for the recycling test. After 3 h period of the reaction, the catalyst was separated by centrifugation. Subsequently, the separated solution was transferred to the reactor and H_2 (5 MPa) was recharged, followed by heating at 140 °C with magnetically stirring for another 21 h.

1. 4. Computational methods

All calculations were performed with the CASTEP program[S1,S2] in the Materials Studio of Accelrys Inc. The Perdew−Burke−Ernzerhof (PBE) generalized gradient functional[S3] was employed for the exchange-correlation energy. The plane-wave basis set with a cutoff energy

of 250 eV was used for the system with periodic boundary conditions. The ultrasoft pseudotentials were used to describe the electron–ion interaction. Brillouin zone was sampled with a (3 × 3 × 1) Monkhorst–Pack[S4] k-points. Re (001), Pd (111) and Rh (111) surfaces were modeled by a supercell slab that consists of a 3 × 3 surface unit cell with 4 atomic layers. Note that the most stable and common planes were used for each metal. The (001) surface has been used for Re metal that has a hexagonal close-packed (hcp) structure and the (111) surface has been used for Pd and Rh metals that have a face-centered cubic (fcc) structure. The slab was separated in vertical direction by a vacuum space with a height of 15 Å. The top 2 layers of the surfaces were fully relaxed, whereas the bottom 2 layers were fixed at the corresponding bulk positions. For a model of partly oxidized Re (ReOx), an oxygen atom adsorbed Re(001) surface was used. The adsorption energy (E_{ads}) was calculated as the difference in the energy of the molecule absorbed on the surface ($E_{molecule/surface}$) and the energy of the individual adsorbate molecule ($E_{molecule}$) and surface ($E_{surface}$) as shown in the following equation:

$$E_{ads} = E_{molecule} + E_{surface} - E_{molecule/surface} \quad (1)$$

To confirm the reliability of the models, we considered the adsorption energy of benzene on Re (001) with extended models ((4 × 4 × 2) k-point, 300 eV cutoff energy, 4 × 4 surface unit cell and 6 surface layers). As summarized in Table S2, the small quantitative differences do not affect the conclusions of the present paper.

2. Supplementary results

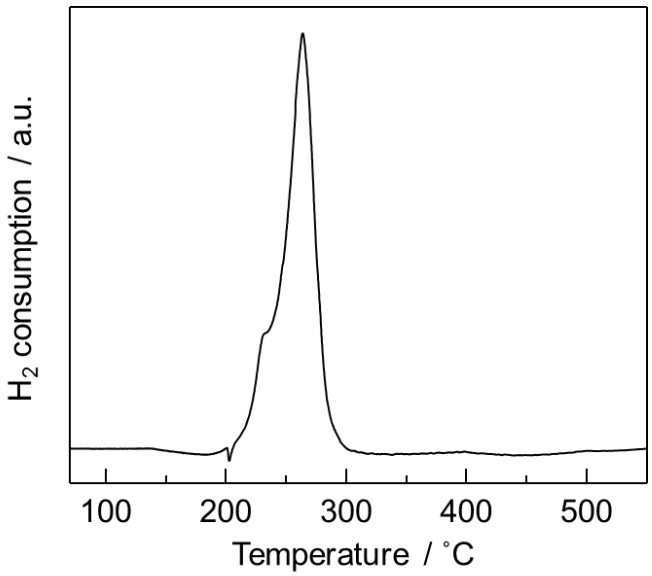


Figure S1. H_2-TPR profile of NH_4ReO_4-impregnated TiO_2 after calcination at 500 °C in air. The sample was heated with a temperature ramp-rate of 10 °C min^{-1} in a flow of 5% H_2/Ar (20 cm^3 min^{-1}).

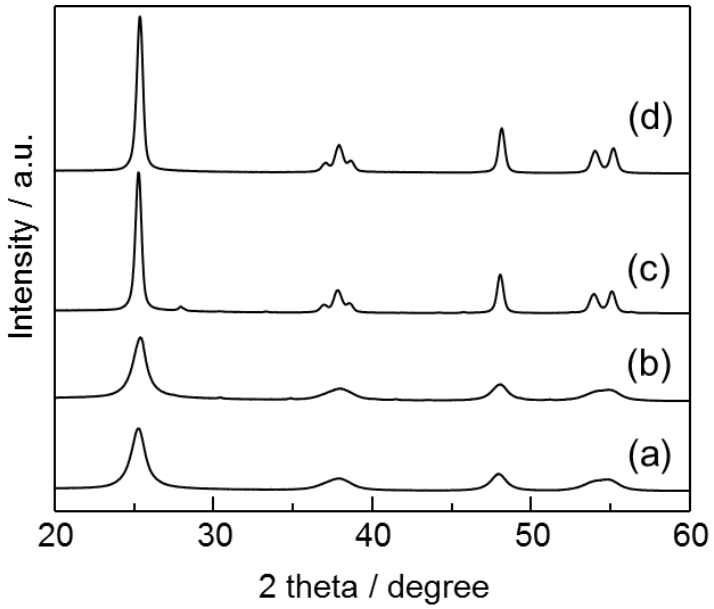


Figure S2. XRD patterns of a) TiO_2; b) NH_4ReO_4-impregnated TiO_2; c) after calcination of (b) at 500 °C in air; d) after reduction of (c) at 500 °C under H_2 flow (Re/TiO_2).

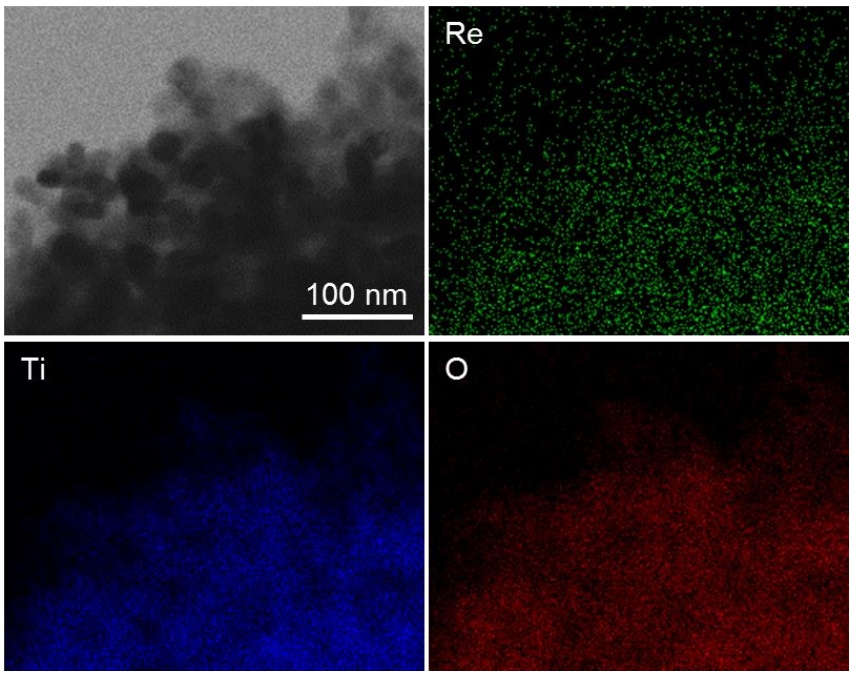


Figure S3. STEM-EDX images of Re/TiO_2.

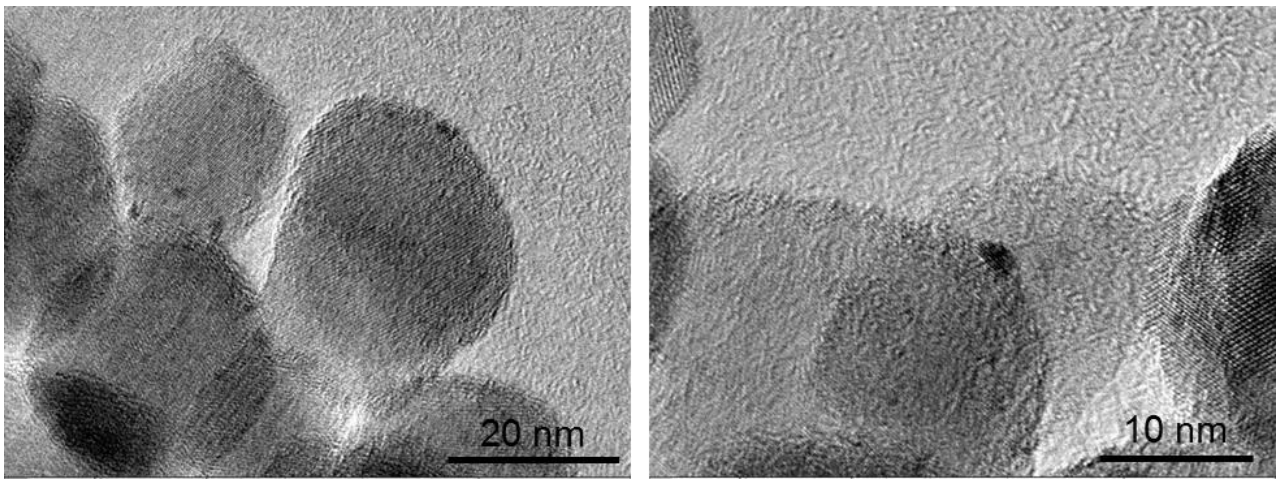
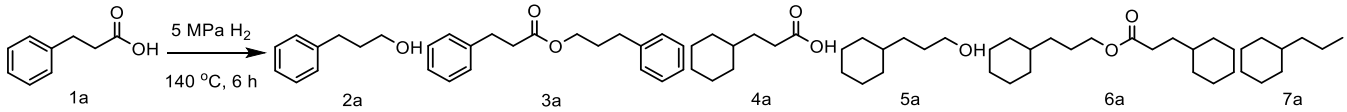


Figure S4. TEM images of Re/TiO_2.

Table S1. Full data for hydrogenation of 3-phenylpropionic acid catalyzed by various catalysts.[a]



Entry	Catalysts	Conv. [%]	Yield [%]					
			2a	3a	4a	5a	6a	7a
1	Re/TiO_2	100	60	40	0	0	0	0
2	Pt/TiO_2	100	0	0	0	64	34	1
3	Ir/TiO_2	96	18	34	14	1	16	7
4	Ru/TiO_2	59	0	6	45	0	6	0
5	Rh/TiO_2	100	0	0	65	0	20	4
6	Pd/TiO_2	91	0	0	90	0	0	0
7	Ag/TiO_2	4	0	0	0	0	0	0
8	Cu/TiO_2	1	0	0	0	0	0	0
9	Ni/TiO_2	0	0	0	0	0	0	0
10	Co/TiO_2	0	0	0	0	0	0	0
11	TiO_2	0	0	0	0	0	0	0
12[b]	Re/TiO_2	21	3	16	0	0	0	0
13	$Re/\gamma\text{-}Al_2O_3$	80	4	30	0	0	0	0
14	$Re/\theta\text{-}Al_2O_3$	46	5	40	0	0	0	0
15	Re/Carbon	41	1	22	0	0	0	0
16	Re/CeO_2	54	4	26	0	0	0	0
17	Re/ZrO_2	22	0	12	0	0	0	0
18	Re/SiO_2	13	1	10	0	0	0	0
19	Re/Nb_2O_5	1	0	2	0	0	0	0
20	Re/SnO_2	0	0	0	0	0	0	0
21	Re/HZSM-5(22)	10	0	8	0	0	0	0
22	Re/HY(5.5)	10	0	10	0	0	0	0
23[c]	NH_4ReO_4	3	0	0	0	0	0	0
24	Metallic Re	0	0	0	0	0	0	0
25[c]	ReO_2	5	0	4	0	0	0	0
26[c]	Re_2O_7	10	0	8	0	0	0	0

[a]Reaction conditions: 2 mol% catalyst, 1 mmol phenylpropionic acid, no solvent, 5 MPa H_2, 140 °C, 6 h. Yields were determined by using GC with n-dodecane as an internal standard. [b]The reaction was performed after calcination at 500 °C in the air. [c]The reaction was performed without pretreatment.

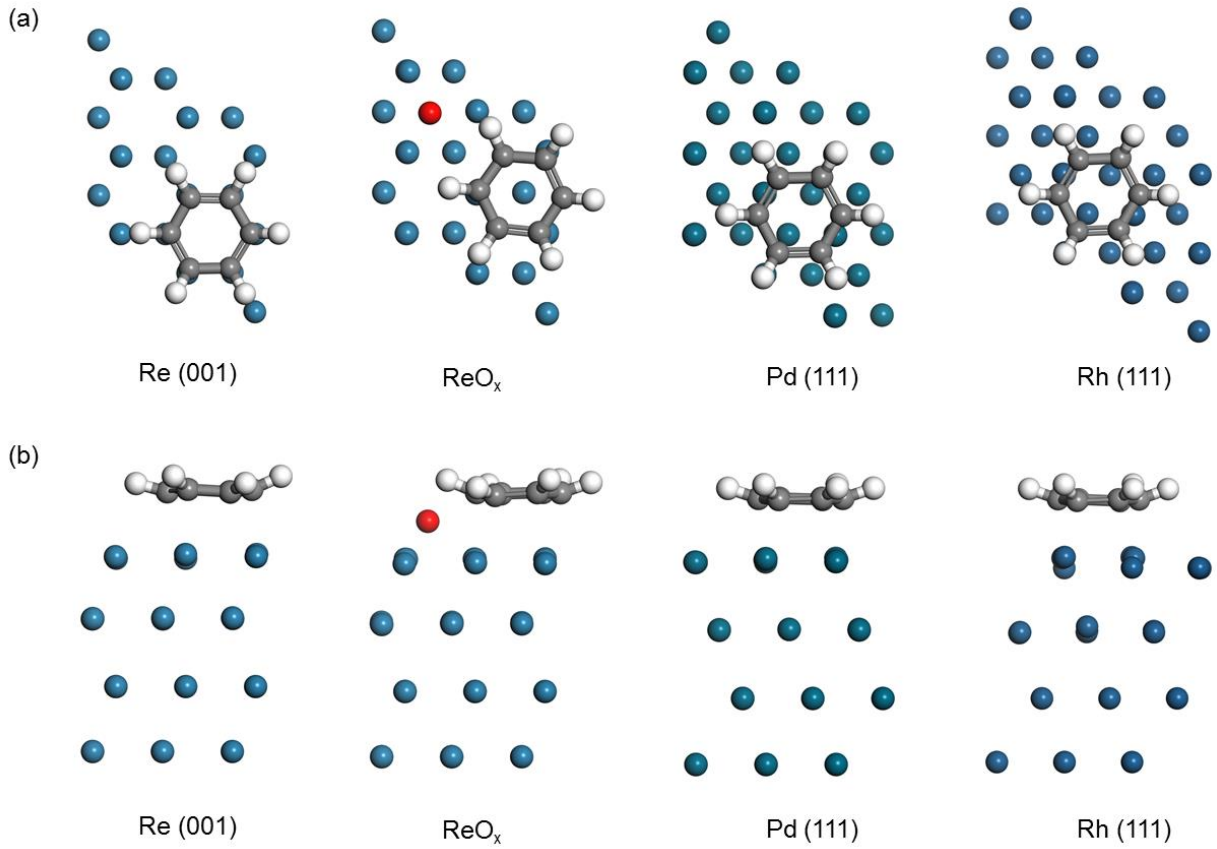


Figure S5. Most stable adsorption structures of benzene adsorbed on Re (001), ReOx, Pd (111) and Rh (111) surfaces. (a) Top view and (b) side view. The C, O, H and metal atoms are colored in gray, red, white and marine blue, respectively

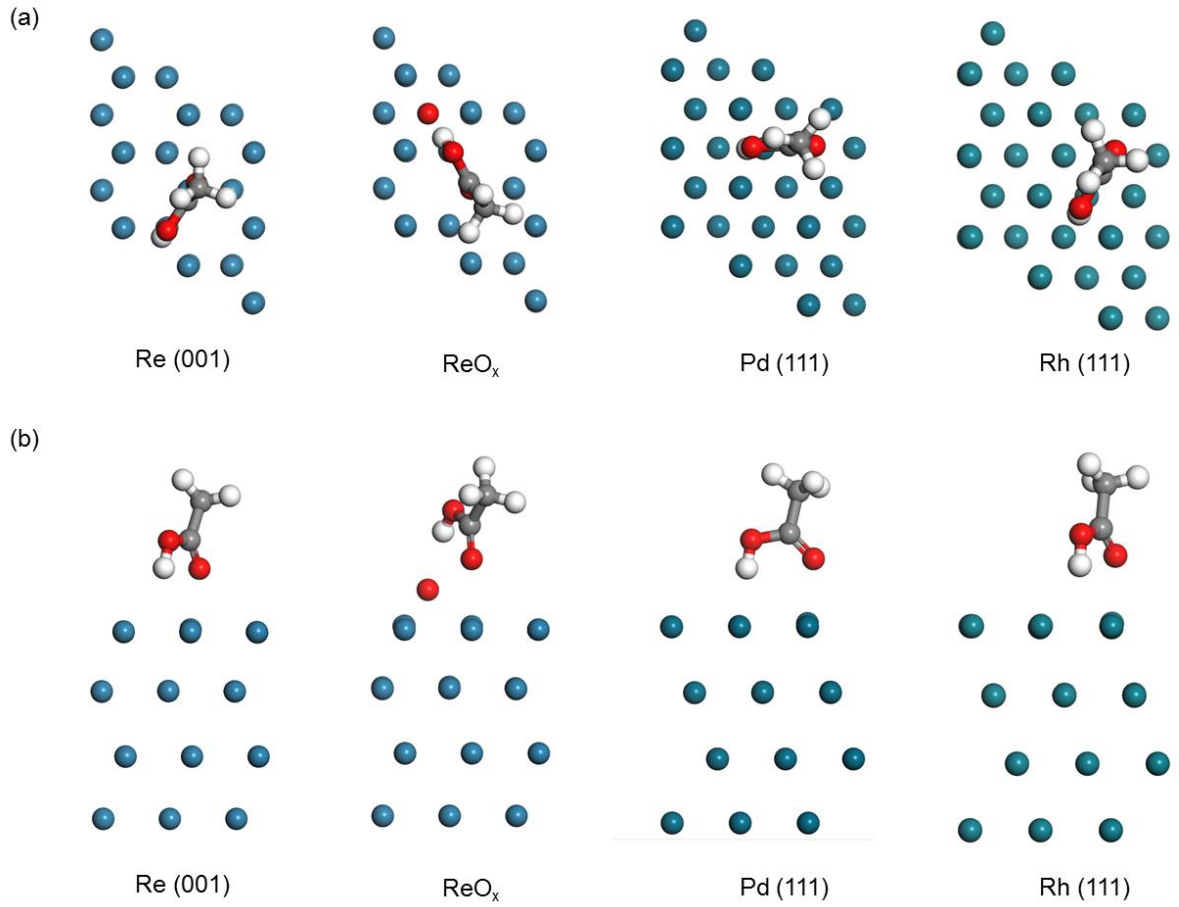
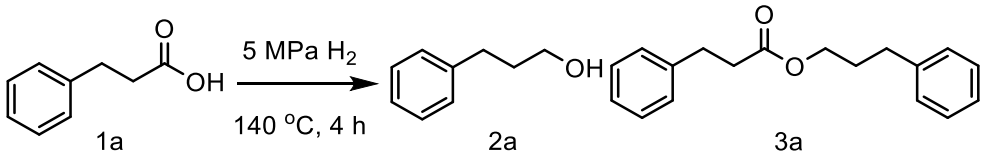


Figure S6. Most stable adsorption structures of acetic acid on Re (001), ReOx, Pd (111) and Rh (111) surfaces. (a) Top view and (b) side view. The C, O, H and metal atoms are colored in gray, red, white and marine blue, respectively

Table S2. Adsorption energies of benzene on Re (001).

Cut off energy [eV]	k-point set	Super cell	Layer number	E_{ads} [kcal/mol]
250	3x3x1	3x3	4	26.6
300	3x3x1	3x3	4	23.4
250	4x4x2	3x3	4	25.9
250	3x3x1	4x4	4	22.1
250	3x3x1	3x3	6	24.4

Table S3. Effect of Re loading on the hydrogenation of 3-phenylpropionic acid catalyzed by Re/TiO_2.[a]

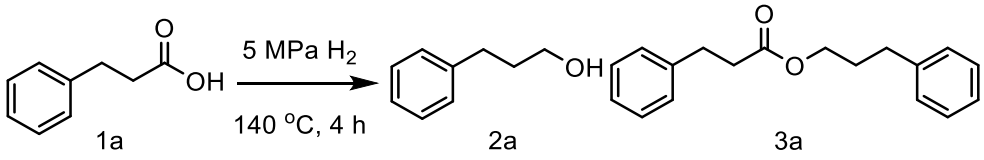


1a 2a 3a

Re loading amount [wt.%]	Conv. [%]	Yield [%]	
		2a	3a
0	0	0	0
1	26	3	22
3	36	9	28
5	61	24	38
10	16	5	12

[a]Reaction conditions: 2 mol% Re, 1 mmol phenylpropionic acid, no solvent, 5 MPa H_2, 140 °C, 4 h. Yields were determined by using GC with n-dodecane as an internal standard.

Table S4. Effect of reduction temperature on the hydrogenation of 3-phenylpropionic acid catalyzed by Re/TiO_2.[a]

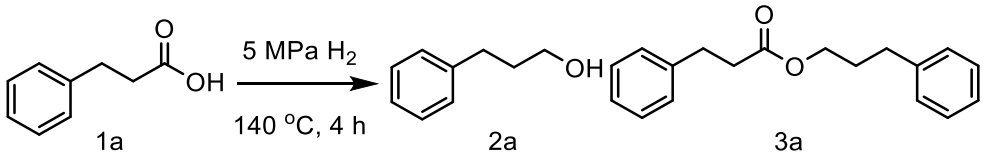


1a 2a 3a

Reduction temperature [°C]	Conv. [%]	Yield [%]	
		2a	3a
No reduction	14	0	14
r.t.	16	0	16
100	8	0	8
200	6	0	6
300	31	6	24
400	59	15	44
500	61	24	38
600	61	23	36
700	47	19	28

[a]Reaction conditions: 2 mol% Re, 1 mmol phenylpropionic acid, no solvent, 5 MPa H_2, 140 °C, 4 h. Yields were determined by using GC with n-dodecane as an internal standard.

Table S5. Effect of H_2 pressure on the hydrogenation of 3-phenylpropionic acid catalyzed by Re/TiO_2.[a]



H_2 pressure [MPa]	Conv. [%]	Yield [%]	
		2a	3a
0.1	0	0	0
1	17	0	16
2	36	3	32
3	51	13	36
4	62	20	41
5	61	24	36
6	75	29	46
7	77	30	48

[a]Reaction conditions: 2 mol% Re, 1 mmol phenylpropionic acid, no solvent, 140 °C, 4 h. Yields were determined by using GC with n-dodecane as an internal standard.

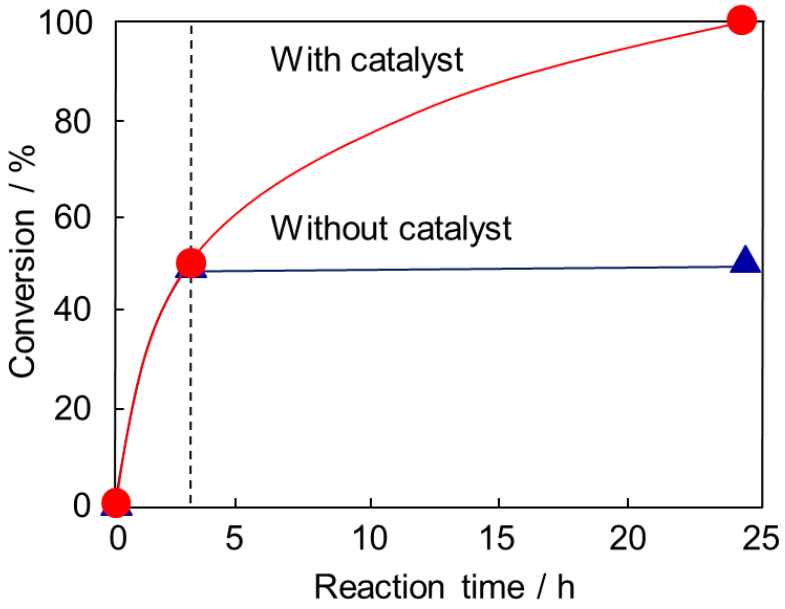
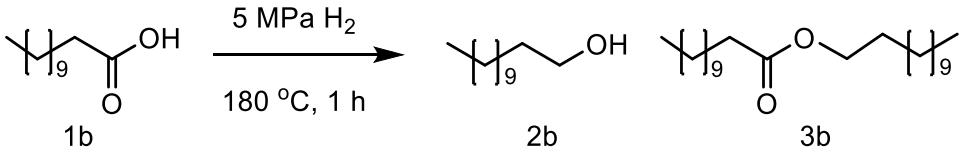


Figure S7. Leaching test for the hydrogenation of 3-phenylpropionic acid over Re/TiO_2 at 140 °C under 5 MPa H_2. After 3 h of the reaction time, the catalyst was filtrated. The reaction solution was further kept at reaction conditions without solid catalyst.

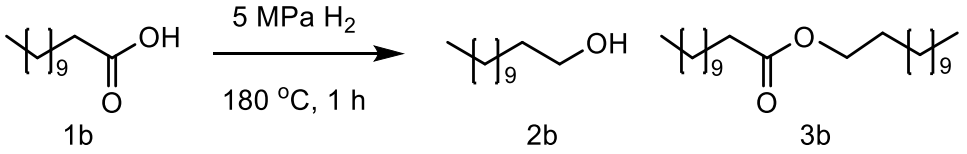
Table S6. Effect of reduction temperature on the hydrogenation of lauric acid catalyzed by Re/TiO_2.[a]



Reduction temperature [°C]	Conv. [%]	Yield [%]	
		2b	3b
r.t.	18	4	10
100	31	7	20
200	10	2	8
300	51	18	30
400	78	39	36
500	89	40	34
500[b]	66	20	44
600	82	41	38
700	54	19	34

[a]Reaction conditions: 2 mol% Re, 1 mmol lauric acid, no solvent, 5 MPa H_2, 180 °C, 1 h. Conversion and yields were determined by using GC with n-hexadecane as an internal standard. [b]The reaction was performed after calcination at 500 °C in the air.

Table S7. Effect of H_2 pressure on the hydrogenation of lauric acid catalyzed by Re/TiO_2.[a]



H_2 pressure [MPa]	Conv. [%]	Yield [%]	
		2b	3b
0.1	18	0	0
1	30	8	20
2	41	12	28
3	61	21	40
4	66	26	34
5	89	40	34
6	82	40	40

[a]Reaction conditions: 2 mol% Re, 1 mmol lauric acid, no solvent, 180 °C, 1 h. Conversion and yields were determined by using GC with n-hexadecane as an internal standard.

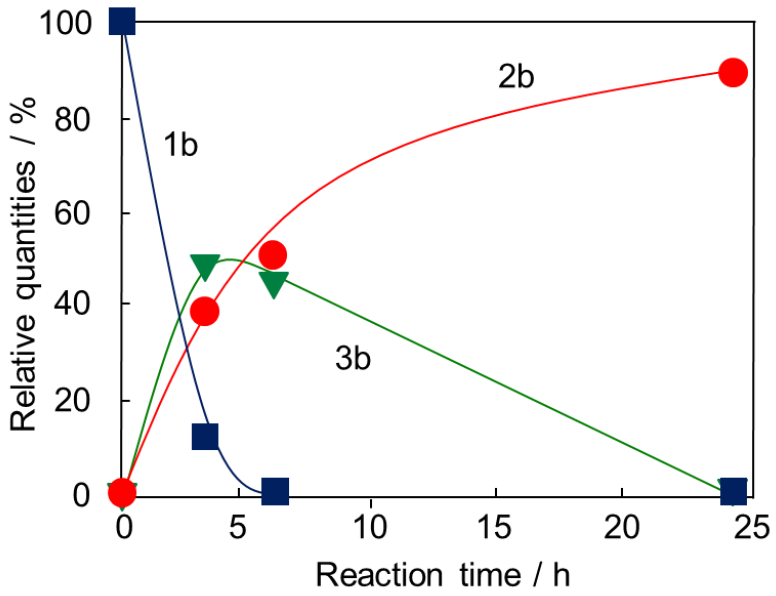
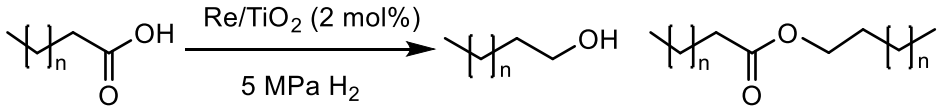


Figure S8. Time course of hydrogenation of lauric acid catalyzed by Re/TiO_2 at 160 °C under 5 MPa H_2. Substances in the plot are lauric acid (1b), lauryl alcohol (2b) and dodecanoic acid dodecyl ester (3b).

Table S8. Hydrogenation of various fatty acids catalyzed by Re/TiO_2.[a]



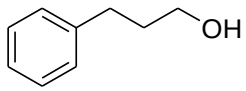
Entry	Substrate	*T* [°C]	Time [h]	Yield [%]	
				Alcohol	Ester
1	n = 2	180	18	90 (83)	0
2	n = 3	180	18	99 (88)	0
3	n = 5	180	12	95 (91)	4
4	n = 7	160	24	88 (84)	8
5	n = 9	160	24	90 (86)	0
6	n = 11	160	24	90 (88)	0
7	n = 13	160	24	92 (90)	0
8	n = 15	180	24	79 (79)	0

[a]Reaction conditions: 2 mol% Re, 1 mmol substrate, no solvent, 5 MPa H_2. Conversion and yields were determined by using GC with n-dodecane as an internal standard. [b]Isolated yields are shown in parentheses.

3. NMR data

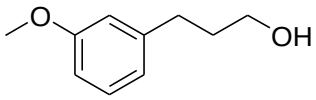
^{1}H and ^{13}C NMR spectra for the obtained alcohols were assigned and reproduced to the corresponding literature. Abbreviations used in the NMR experiments: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. GC-MS spectra were taken by SHIMADZU QP2010.

3-Phenyl-propan-1-ol: [S5]



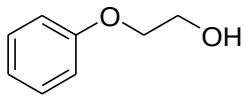
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.27 (t, *J* = 7.56 Hz, 2H), 7.19 (d, *J* = 7.56 Hz, 3H), 3.65 (t, *J* = 6.54 Hz, 2H), 2.69 (t, *J* = 7.89 Hz, 2H), 1.90-1.85 (m, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 141.76, 128.37 (C×2), 128.34, 128.21, 125.79, 62.15, 34.13, 32.00; GC-MS m/e 136.15.

3-(3-Methoxyphenyl)-propane-1-ol:[S6]



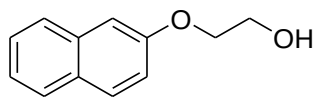
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.20 (t, *J* = 7.56 Hz, 1H), 6.79 (d, *J* = 7.56 Hz, 1H), 6.75-6.72 (m, 2H), 3.79 (s, 3H), 3.66 (t, *J* = 6.18 Hz, 2H), 2.68 (t, *J* = 7.92 Hz, 2H), 1.89-1.87 (m, 2H), 1.66 (br s, 1H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 159.58, 143.43, 129.30, 120.78, 115.15, 111.04, 62.16, 55.07, 34.03, 32.07; GC-MS m/e 166.10.

2-Phenoxy-ethanol:[S7]



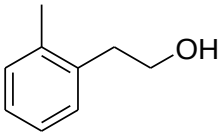
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.27 (t, *J* = 7.14 Hz, 2H), 6.95 (t, *J* = 7.41 Hz, 1H), 6.90 (d, *J* = 7.68 Hz, 2H), 4.05 (t, *J* = 4.65 Hz, 2H), 3.94 (t, *J* = 3.84 Hz, 2H), 2.57 (br s, 1H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 158.49, 129.44 (C×2), 121.00, 114.43 (C×2), 68.99, 61.29; GC-MS m/e 138.07.

2-(Napthalene-2-yloxy)-ethanol:[S8]



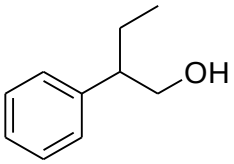
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.75-7.69 (m, 3H), 7.42 (t, *J* = 7.56 Hz, 1H), 7.33 (t, *J* = 7.56 Hz, 1H), 7.16-7.14 (m, 1H), 7.13-7.11 (m, 1H), 4.16-4.15 (m, 2H), 3.99-3.98 (m, 2H), 2.32 (br s, 1H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 156.47, 134.39, 129.56, 129.04, 127.60, 126.72, 126.39, 123.74, 118.65, 106.75, 69.12, 61.37; GC-MS m/e 188.09.

2-*o*-Tolyl-ethanol:[S9]



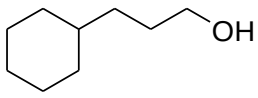
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.14-7.13 (m, 4H), 3.81-3.78 (m, 2H), 2.88-2.86 (m, 2H), 2.32 (s, 3H), 1.80 (br s, 1H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 136.44, 136.42, 130.34, 129.56, 126.51, 125.97, 62.52, 36.30, 19.37; GC-MS m/e 136.15.

2-Phenyl-butan-1-ol:[S10]



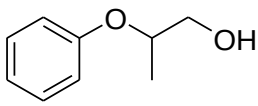
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.31 (d, *J* = 7.56 Hz, 2H), 7.22 (t, *J* = 7.56 Hz, 1H), 7.18 (d, *J* = 7.56 Hz, 2H), 3.74-3.69 (m, 2H), 2.69-2.64 (m, 2H), 1.75-1.72 (m, 1H), 1.58-1.53 (m, 1H) 0.83-0.81 (m, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 142.20, 128.55, 128.06, 128.00, 127.22, 126.63, 67.23, 50.38, 24.92, 11.91; GC-MS m/e 150.15.

3-Cyclohexyl-propane-1-ol:[S11]



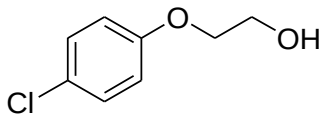
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 3.61 (t, *J* = 6.87 Hz, 2H), 1.87 (br s, 1H), 1.72-1.68 (m, 4H), 1.65-1.63 (m, 1 H), 1.58-1.55 (m, 2H), 1.22-1.15 (m, 6H), 0.90-0.87 (m, 2H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 63.32, 37.44, 33.36 (C×2), 33.31, 30.06, 26.61, 26.32 (C×2); GC-MS m/e 142.15.

2-Phenoxy-propan-1-ol:[S7]



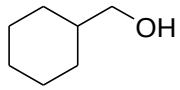
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.29-7.24 (m, 2H), 6.96-6.92 (m, 3H), 4.49-4.48 (m, 1H), 3.74-3.70 (m, 2H), 2.20 (br s, 1H), 1.26-1.25 (m, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 157.64, 129.53 (C×2), 121.14, 116.05 (C×2), 74.63, 66.20, 15.73; GC-MS m/e 152.09.

2-(4-Chloro-phenoxy)-ethanol:[S12]



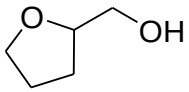
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.26-7.21 (m, 2H), 6.84-6.82 (m, 2H), 4.04-4.02 (m, 2H), 3.95-3.93 (m, 2H), 2.34 (br s, 1H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 157.15, 129.33 (C×2), 125.91, 115.74 (C×2), 69.44, 61.25; GC-MS m/e 172.03.

Cyclohexyl-methanol:[S13]



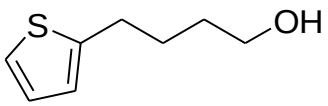
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 3.43 (s, 2H), 1.76-1.72 (m, 4H), 1.69-1.66 (m, 1H), 1.49-1.46 (m, 2H), 1.26-1.21 (m, 2H), 1.19-1.15 (m, 1H), 0.96-0.89 (m, 2H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 68.72, 40.44, 29.52, 26.55 (C×2), 25.80 (C×2); GC-MS m/e 114.15.

(Tetrahydrofuran-2-yl)-methanol:[S14]



^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 4.01-4.00 (m, 1H), 3.87-3.86 (m, 1H), 3.80-3.78 (m, 1H), 3.66-3.64 (m, 1H), 3.52-3.50 (m, 1H), 3.16 (br s, 1H), 1.94-1.87 (m, 3H), 1.65-1.64 (m, 1H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 79.46, 68.03, 64.61, 26.98, 25.74; GC-MS m/e 102.07.

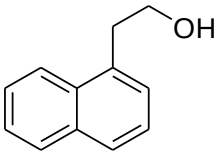
4-Thiophen-2-yl-butane-1-ol:[S15]



^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.10 (d, *J* = 5.52 Hz, 1H), 6.91-6.90 (m, 1H), 6.78 (d, *J* = 3.48 Hz, 1H), 3.65 (t, *J* = 6.51 Hz, 2H), 2.85 (t, *J* = 7.56 Hz, 2H), 1.76-1.72 (m, 2H), 1.68 (br

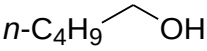
s, 1H), 1.65-1.61 (m, 2H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 145.13, 126.63, 124.07, 122.87, 62.51, 32.01, 29.57, 27.88; GC-MS m/e 156.08.

2-Napthalene-2-yl-ethanol:[S16]



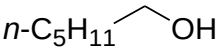
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 8.03 (d, *J* = 6.18 Hz, 1H), 7.84 (d, *J* = 6.84 Hz, 1H), 7.73 (d, *J* = 6.18 Hz, 1H), 7.51-7.48 (m, 2H), 7.39 (t, *J* = 6.54 Hz, 1H), 7.34 (d, *J* = 7.12 Hz, 1H), 3.93 (t, *J* = 5.84 Hz, 2H), 3.31 (t, *J* = 6.84 Hz, 2H), 1.92 (br s, 1H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 134.33, 133.88, 131.98, 128.76, 127.21, 127.07, 125.95, 125.57, 125.43, 123.58, 62.89, 36.12; GC-MS m/e 173.15.

Pentan-1-ol:[S17]



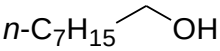
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 3.62 (t, *J* = 6.51 Hz, 2H), 2.13 (br s, 1H), 1.57-1.54 (m, 2H), 1.34-1.32 (m, 4H), 0.90 (t, *J* = 7.23 Hz, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 62.80, 32.35, 27.84 (C×2), 22.42, 13.95; GC-MS m/e 88.10.

Hexan-1-ol:[S18]



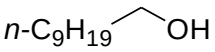
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 3.61 (t, *J* = 6.18 Hz, 2H), 2.09 (br s, 1H), 1.57-1.54 (m, 2H), 1.32-1.30 (m, 6H), 0.89 (t, *J* = 6.87 Hz, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 62.83, 32.64, 31.58, 25.37, 22.56, 13.94; GC-MS m/e 102.15.

Octan-1-ol:[S19]



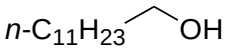
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 3.61 (t, *J* = 6.54 Hz, 2H), 2.20 (br s, 1H), 1.56-1.53 (m, 2H), 1.33-1.28 (m, 10H), 0.88 (t, *J* = 6.83 Hz, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 62.82, 32.68, 31.77, 29.35, 29.23, 25.70, 22.59, 14.00; GC-MS m/e 130.14.

Decan-1-ol:[S20]



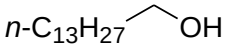
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 3.61 (t, *J* = 6.54 Hz, 2H), 2.18 (br s, 1H), 1.56-1.54 (m, 2H), 1.31-1.26 (m, 14H), 0.88 (t, *J* = 6.87 Hz, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 62.82, 32.69, 31.84, 29.58, 29.51, 29.40, 29.27, 25.70, 22.61, 14.02; GC-MS m/e 158.18.

Dodecan-1-ol:[S21]



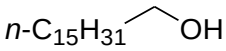
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 3.63 (t, *J* = 6.54 Hz, 2H), 1.58-1.54 (m, 2H), 1.45 (br s, 1H), 1.35-1.25 (m, 18H), 0.88 (t, *J* = 6.87 Hz, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 63.03, 32.77, 31.89, 29.64, 29.61, 29.59, 29.58, 29.42, 29.32, 25.72, 22.66, 14.09; GC-MS m/e 186.20.

Tetradecan-1-ol:[S22]



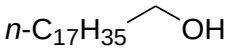
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 3.63 (t, *J* = 6.87 Hz, 2H), 1.57-1.55 (m, 2H), 1.45 (br s, 1H), 1.37-1.25 (m, 22H), 0.88 (t, *J* = 6.87 Hz, 3H), ; ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 63.06, 32.79, 31.90, 29.65 (C×2), 29.60 (C×2), 29.42 (C×2), 29.35 (C×2), 25.72, 22.68, 14.10; GC-MS m/e 214.25.

Hexadecan-1-ol:[S23]

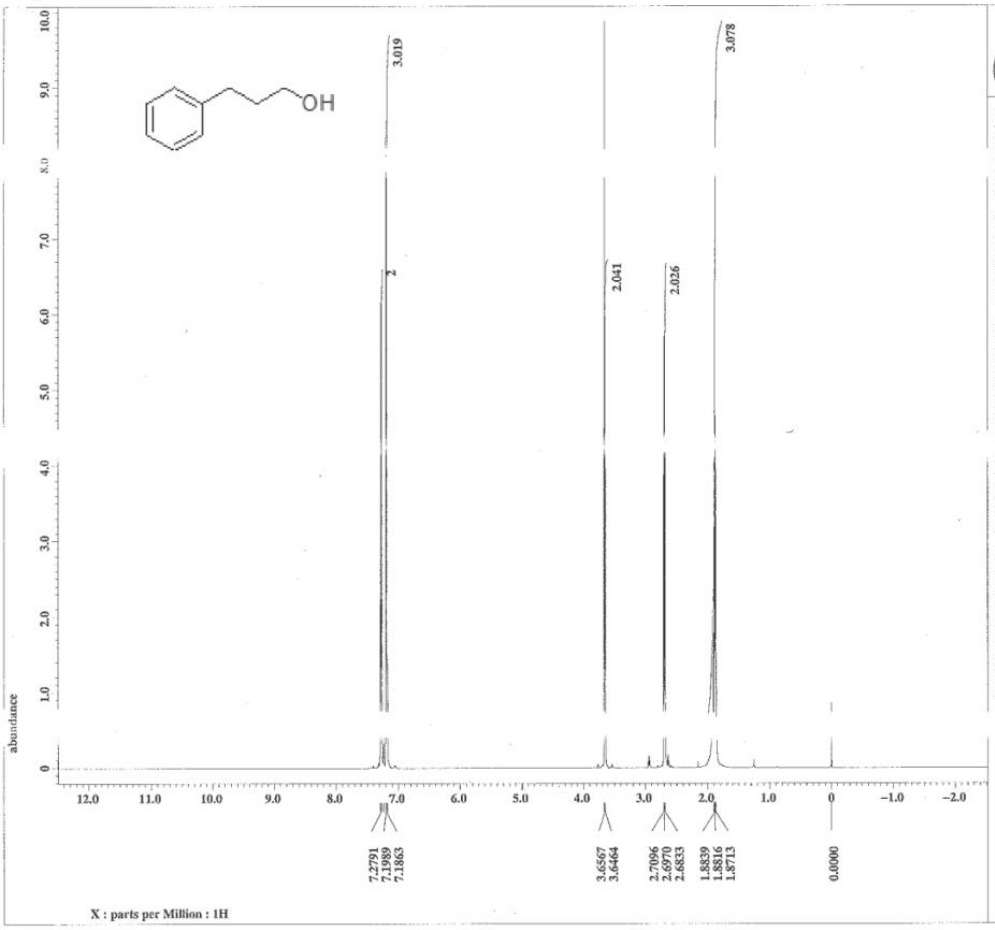


^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 3.63 (t, *J* = 6.87 Hz, 2H), 1.57-1.52 (m, 2H), 1.35-1.25 (m, 27H), 0.88 (t, *J* = 6.87 Hz, 3H), ; ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 63.03, 32.79, 31.91, 29.68 (C×2), 29.65 (C×2), 29.60 (C×2), 29.42 (C×2), 29.35 (C×2), 25.72, 22.68, 14.11; GC-MS m/e 242.28.

Octadecan-1-ol:[S12]



^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 3.63 (t, *J* = 6.18 Hz, 2H), 1.57-1.53 (m, 2H), 1.34-1.31 (m, 4H), 1.30-1.25 (m, 27H), 0.87 (t, *J* = 6.84 Hz, 3H), ; ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 63.07, 32.80, 31.92, 29.69 (C×4), 29.61 (C×2), 29.43 (C×4), 29.35 (C×2), 25.72, 22.68, 14.11; GC-MS m/e 270.30.

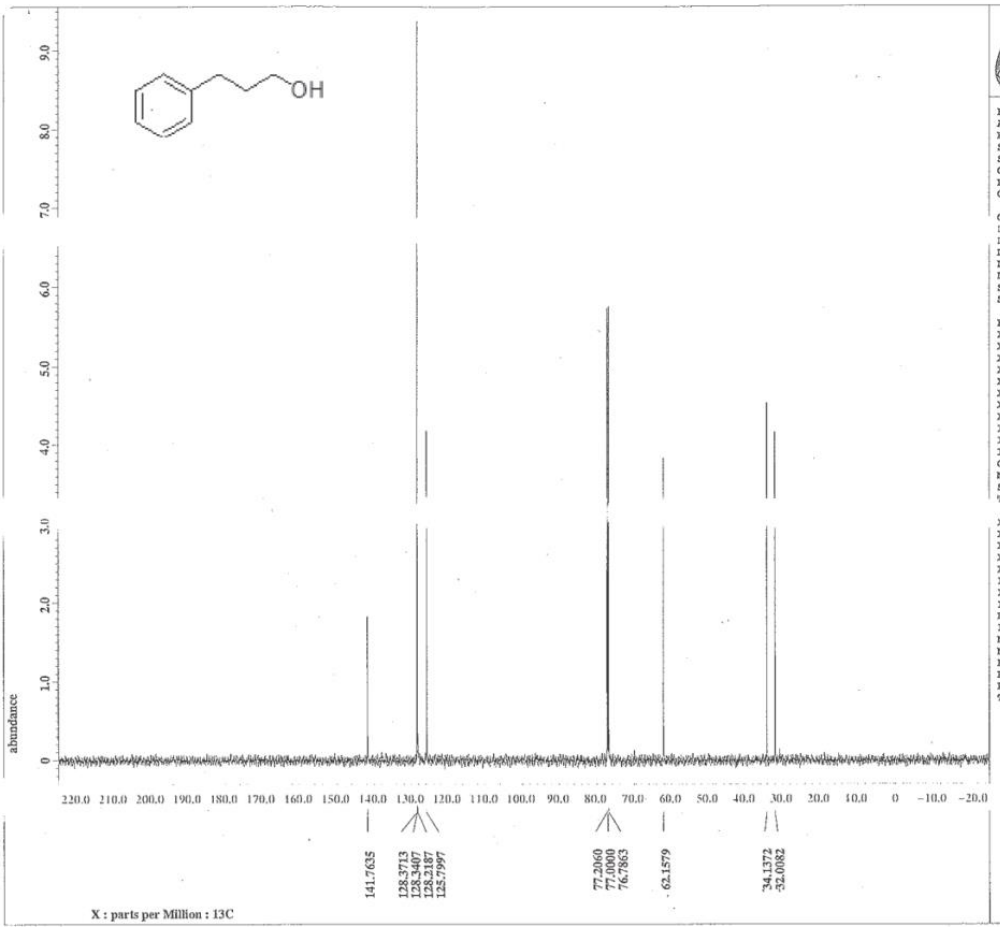



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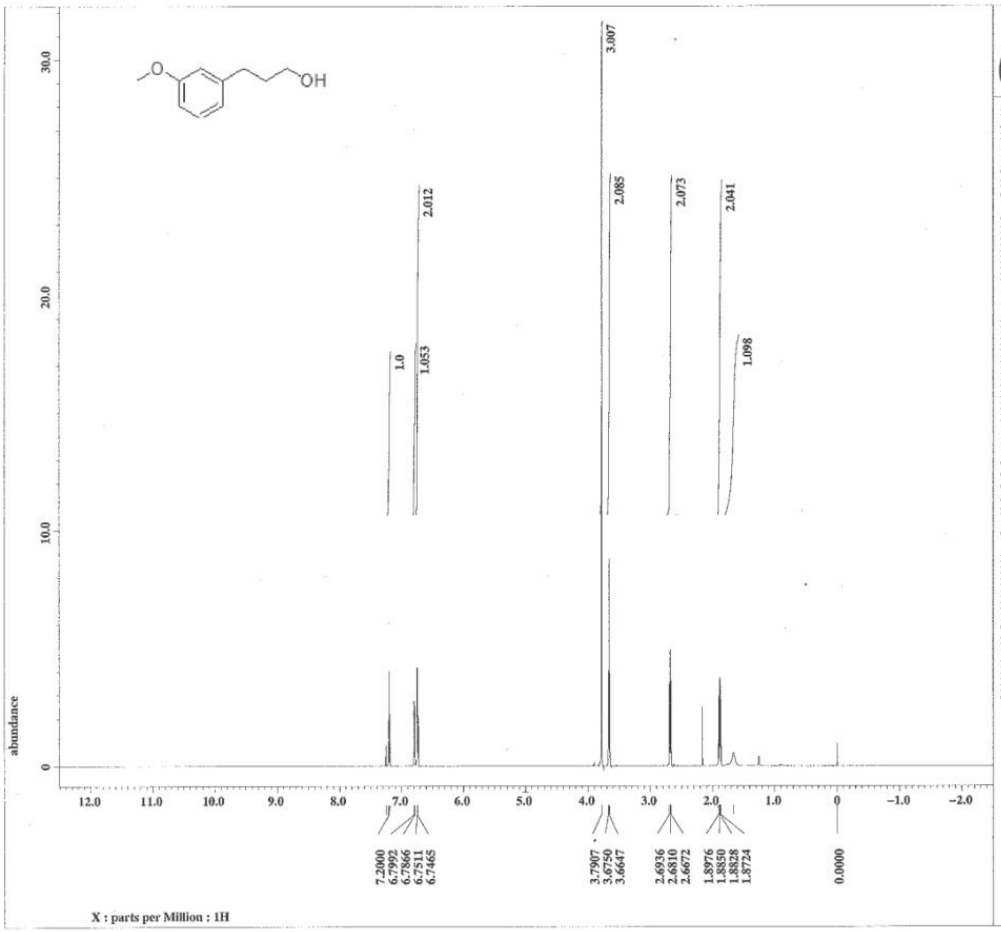



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Revision_time     =  1-APR-2016 16:58:22
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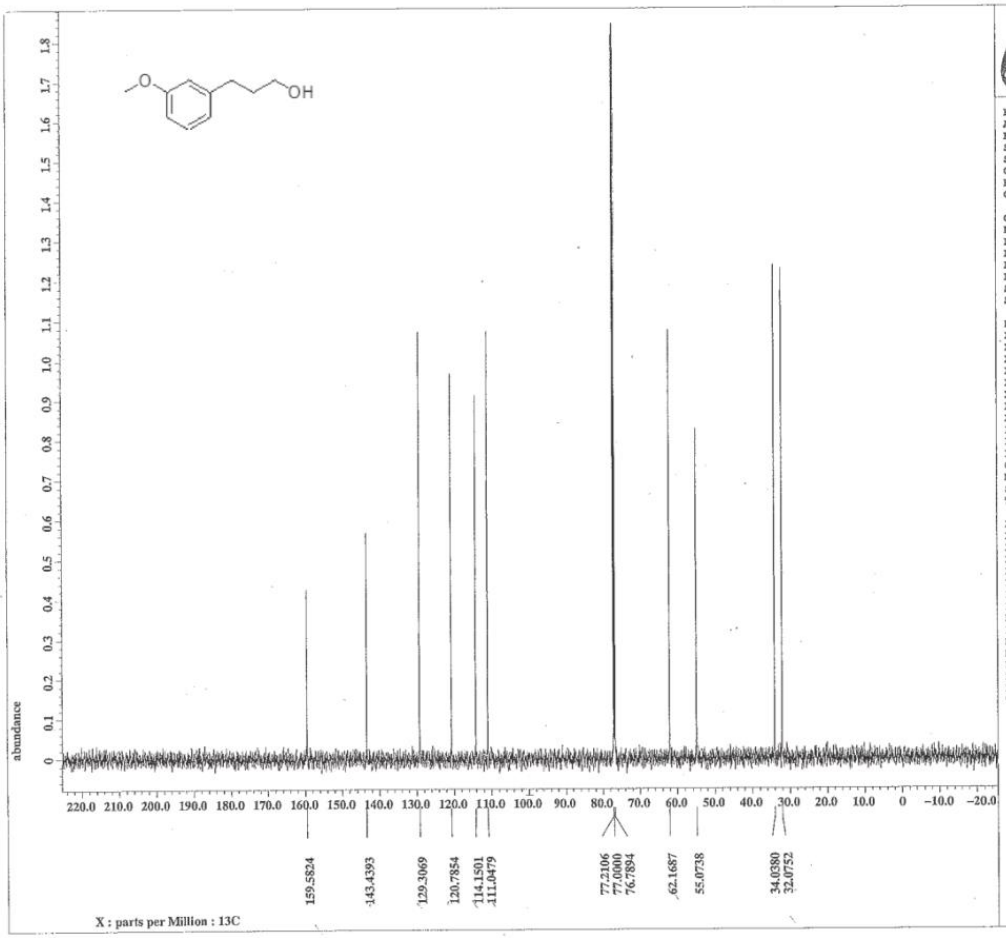
JEOL

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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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 20.9[dC]
```



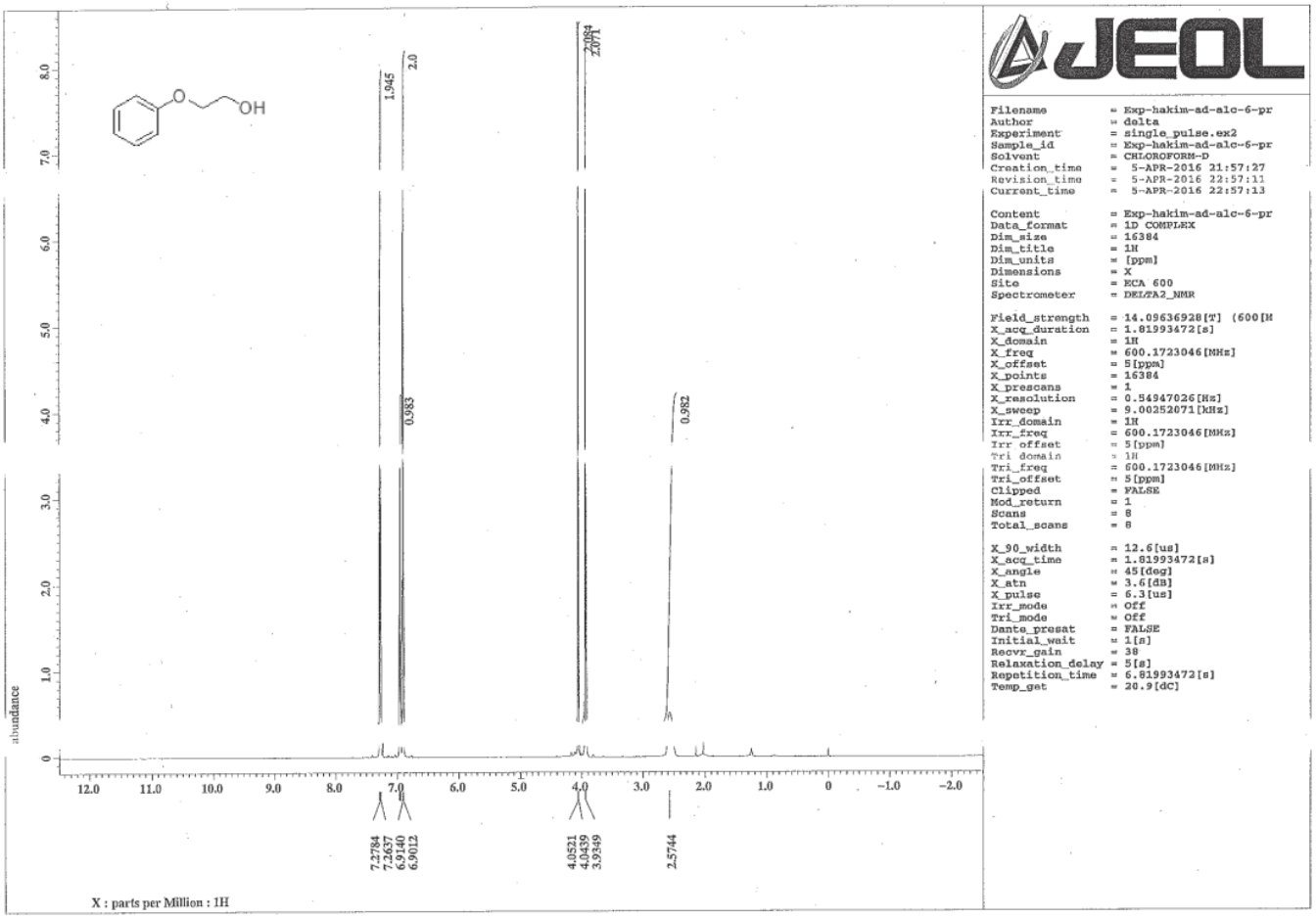
JEOL

```
Filename          = Exp-hakim-ad-alc-n-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-n-ca
Solvent           = CHLOROFORM-D
Creation_time     = 11-JUL-2016 15:55:49
Revision_time     = 11-JUL-2016 16:32:30
Current_time      = 11-JUL-2016 16:32:32

Content           = Exp-hakim-ad-alc-n-ca
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             = 60
Total_scans       = 60

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          = 21.3[dC]
```

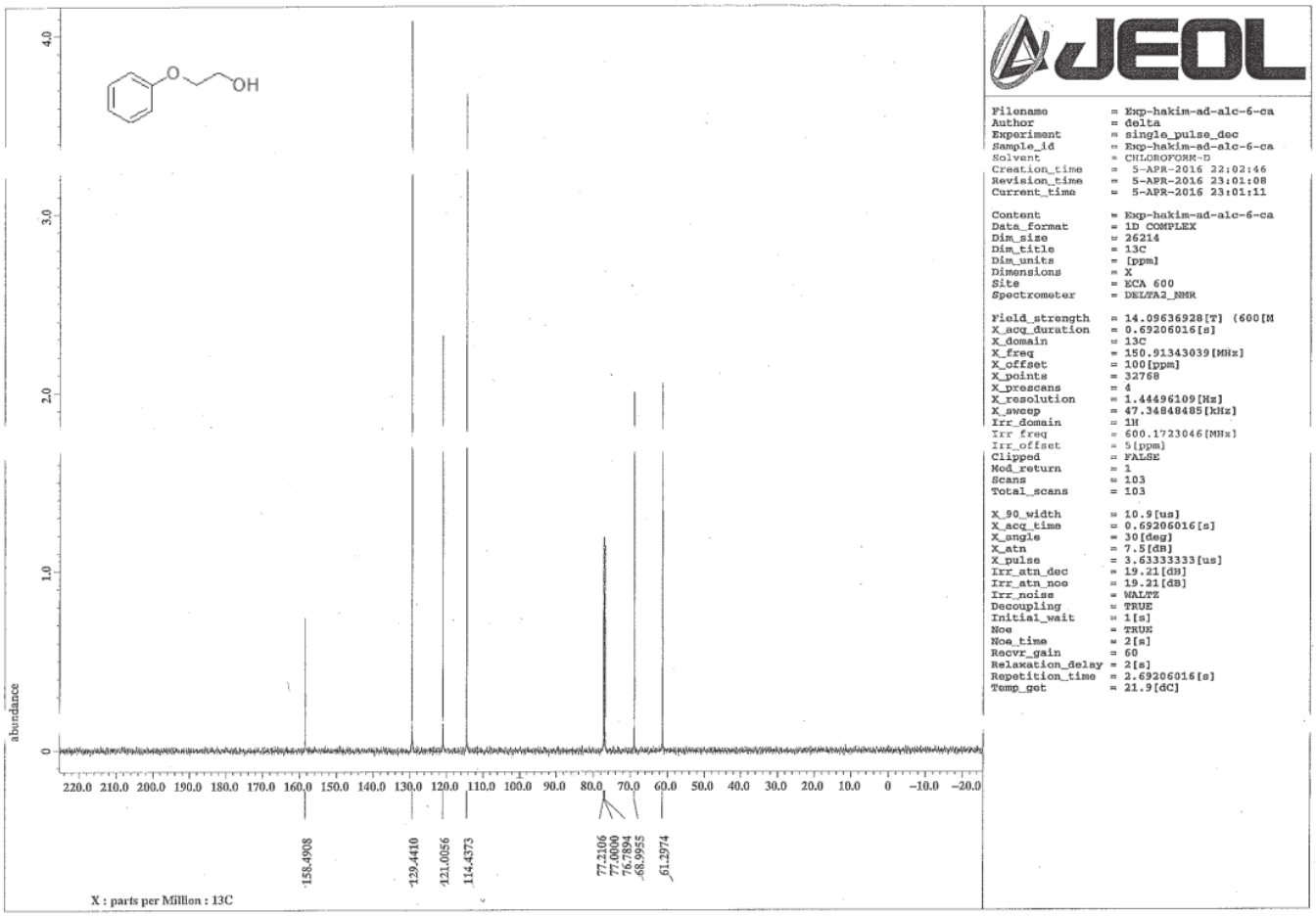



```
Filename          = Exp-hakim-ad-alc-6-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-6-pr
Solvent           = CHLOROFORM-D
Creation_time     =  5-APR-2016 21:57:27
Revision_time     =  5-APR-2016 22:57:11
Current_time      =  5-APR-2016 22:57:13

Content           = Exp-hakim-ad-alc-6-pr
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.81993472[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.54947026[Hz]
X_sweep           = 9.00252071[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        = 1.81993472[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]
Recvr_gain        = 38
Relaxation_delay  = 5[s]
Repetition_time   = 6.81993472[s]
Temp_get          = 20.9[dC]
```

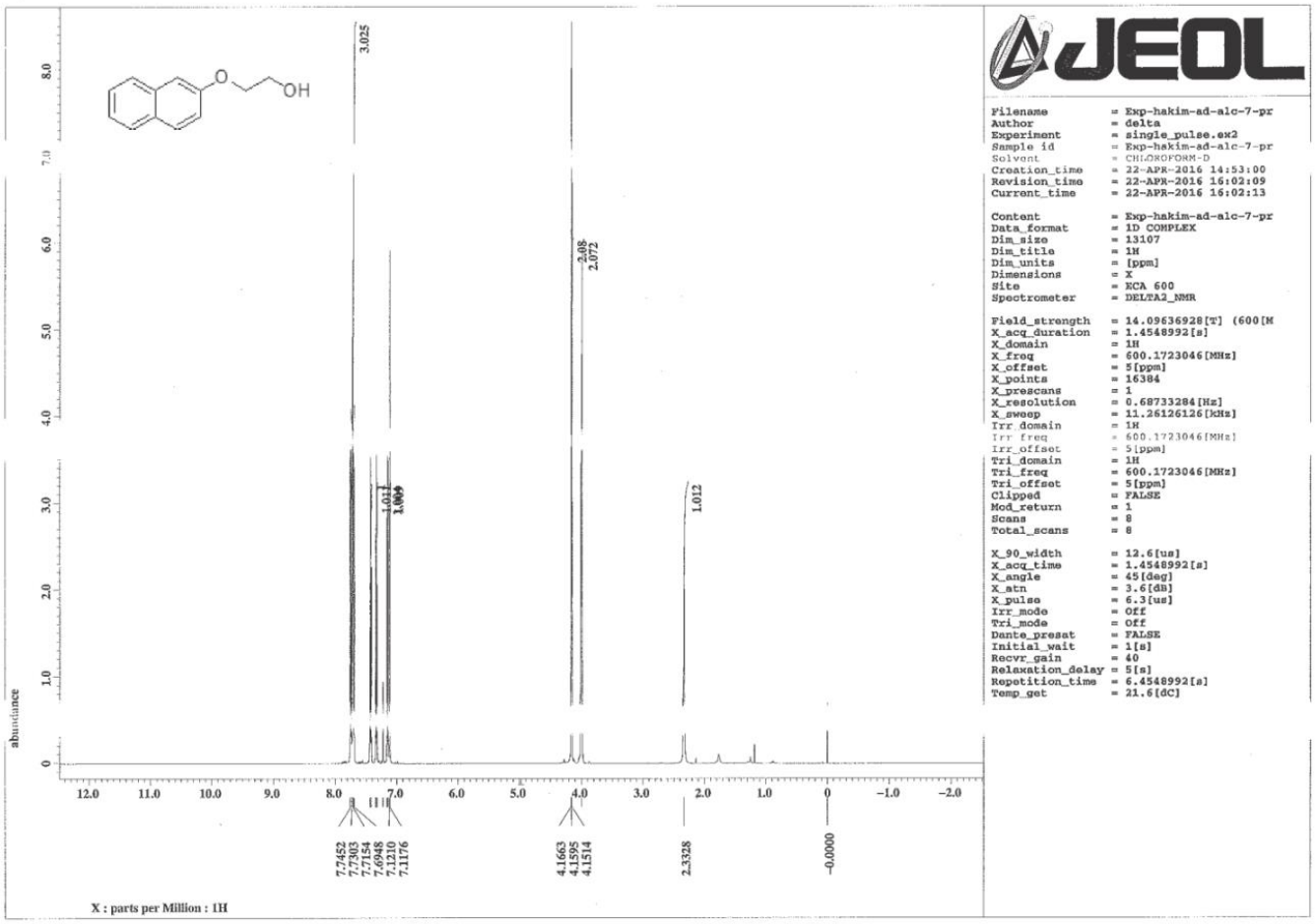



```
Filename          = Exp-hakim-ad-alc-6-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-6-ca
Solvent           = CHLOROFORM-D
Creation_time     =  5-APR-2016 22:02:46
Revision_time     =  5-APR-2016 23:01:08
Current_time      =  5-APR-2016 23:01:11

Content           = Exp-hakim-ad-alc-6-ca
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

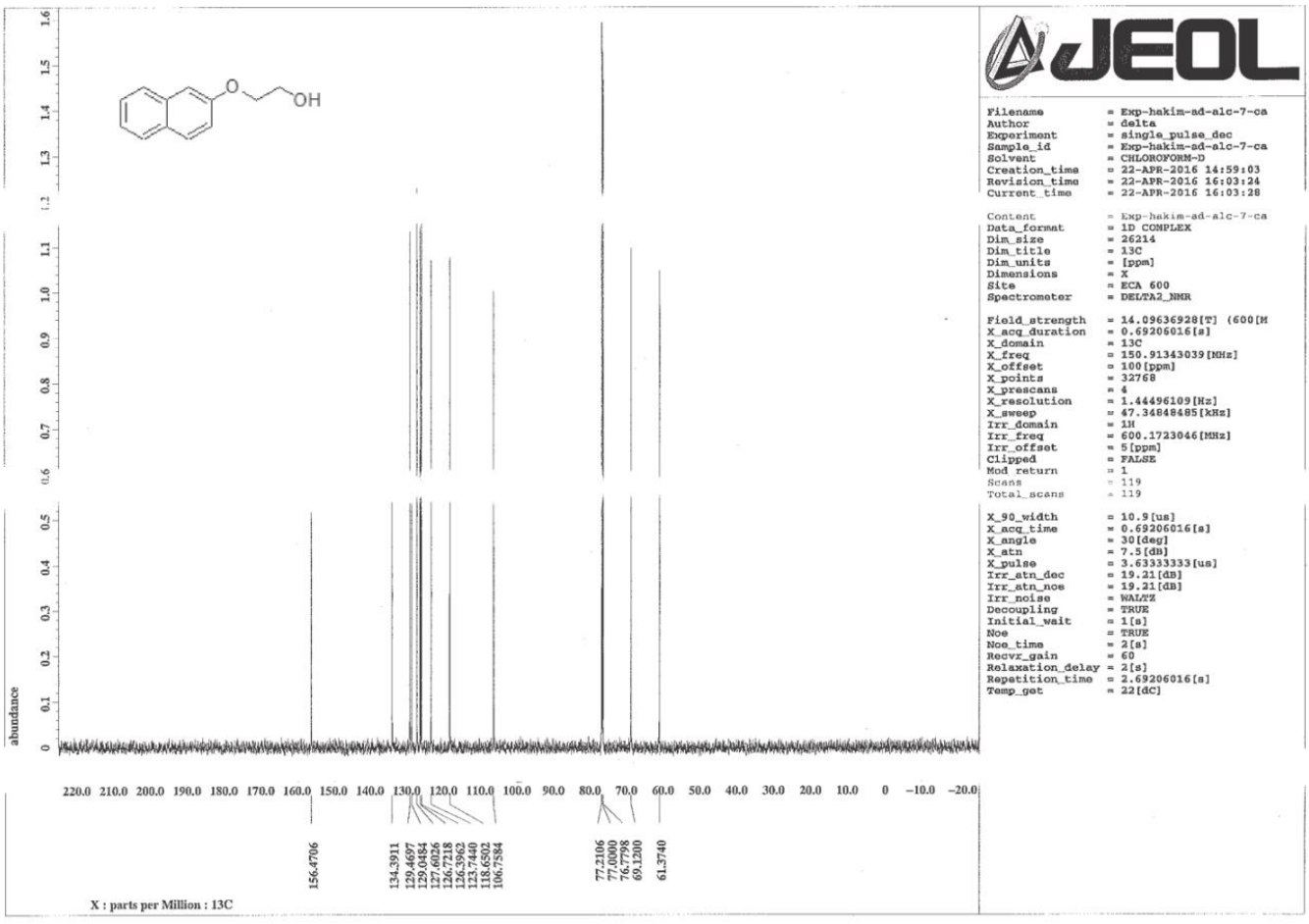
Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.69206016[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.44496109[Hz]
X_sweep           = 47.34848485[kHz]
Irr_domain        = 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_noe       = 19.21[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   = 2.69206016[s]
Temp_get          = 21.9[dC]
```



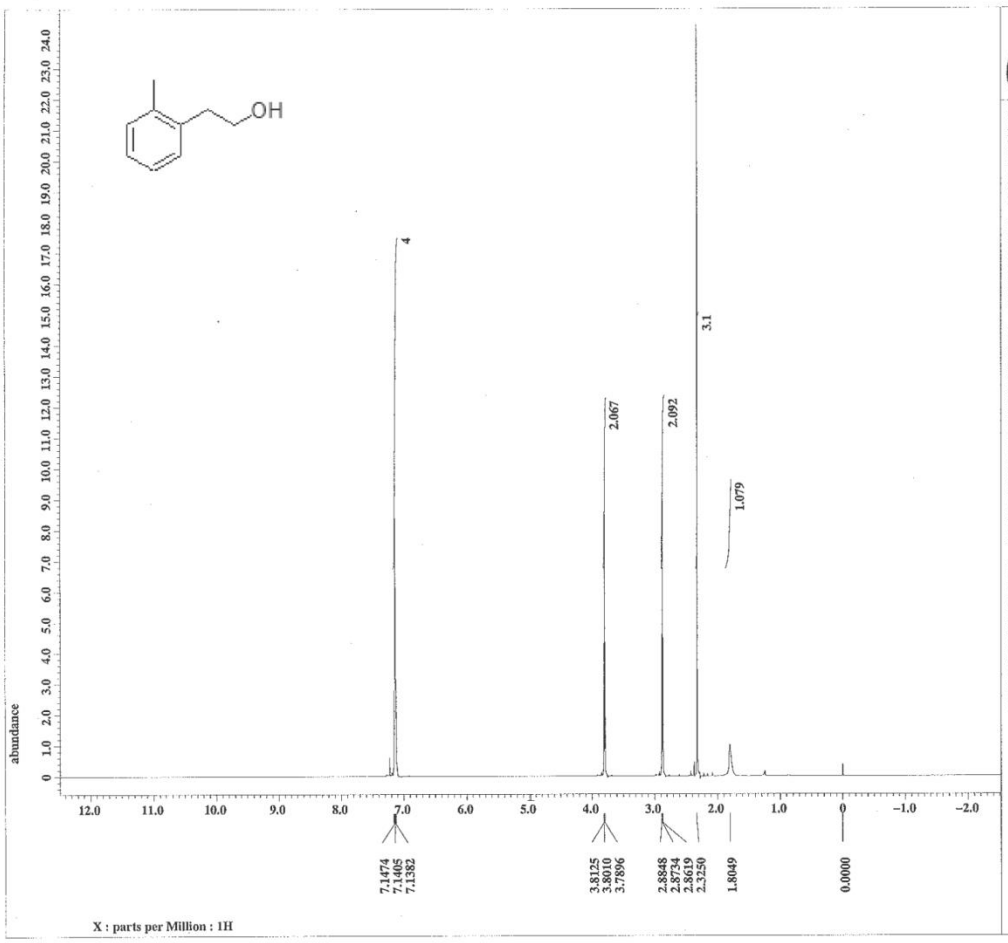
JEOL

Filename	= Exp-hakim-ad-alc-7-pr
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= Exp-hakim-ad-alc-7-pr
Solvent	= CHLOROFORM-D
Creation_time	= 22-APR-2016 14:53:00
Revision_time	= 22-APR-2016 16:02:09
Current_time	= 22-APR-2016 16:02:13
Content	= Exp-hakim-ad-alc-7-pr
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[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_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 6.4548992[s]
Temp_get	= 21.6[dC]



JEOL

Filename	= Exp-hakim-ad-alc-7-ca
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= Exp-hakim-ad-alc-7-ca
Solvent	= CHLOROFORM-D
Creation_time	= 22-APR-2016 14:59:03
Revision_time	= 22-APR-2016 16:03:24
Current_time	= 22-APR-2016 16:03:28
Content	= Exp-hakim-ad-alc-7-ca
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	= 119
Total_scans	= 119
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[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 2.69206016[s]
Temp_get	= 22[dC]

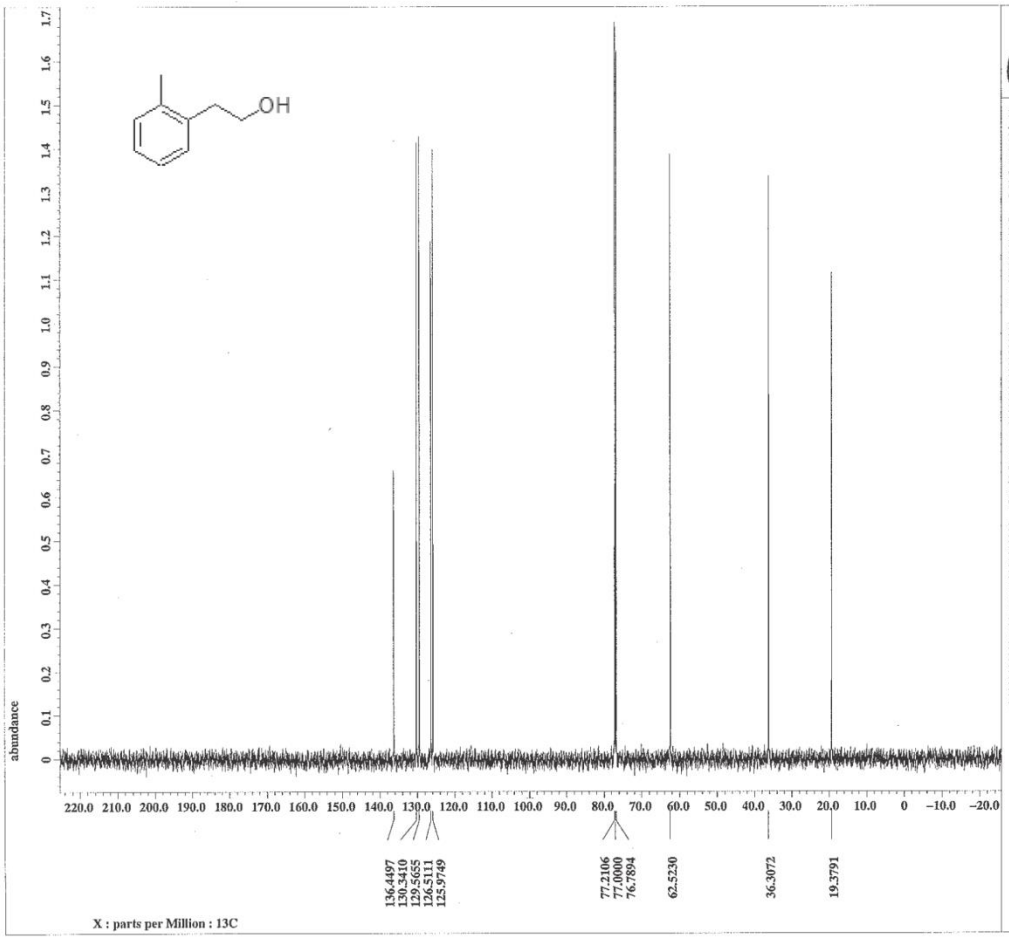



```
Filename          = Exp-hakim-ad-alc-r-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-r-pr
Solvent           = CHLOROFORM-D
Creation_time     =  4-JUL-2016 11:14:51
Revision_time     =  4-JUL-2016 11:53:38
Current_time      =  4-JUL-2016 11:53:46

Content           = Exp-hakim-ad-alc-r-pr
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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.4[dC]
```

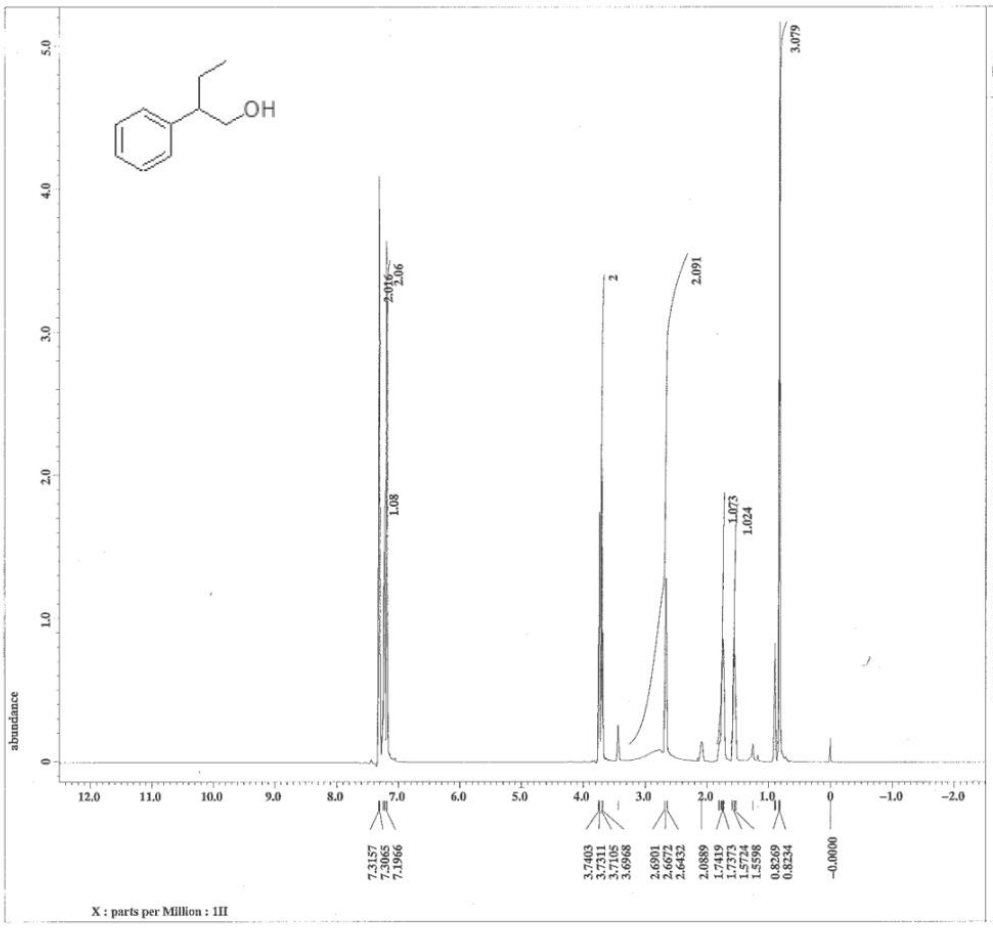



```
Filename          = Exp-hakim-ad-alc-r-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-r-ca
Solvent           = CHLOROFORM-D
Creation_time     =  4-JUL-2016 11:18:58
Revision_time     =  4-JUL-2016 11:56:57
Current_time      =  4-JUL-2016 11:57:17

Content           = Exp-hakim-ad-alc-r-ca
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

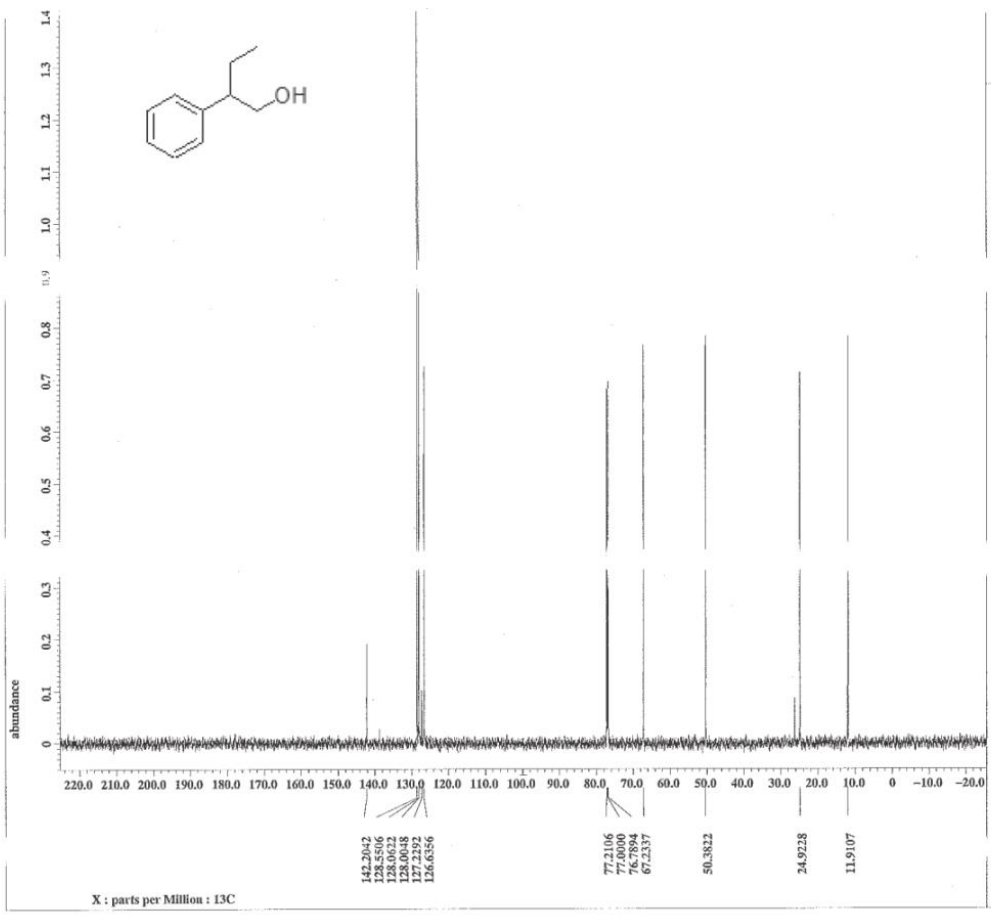
Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.69206016[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.44496109[Hz]
X_sweep           = 47.34848485[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 62
Total_scans       = 62

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          = 22[dC]
```



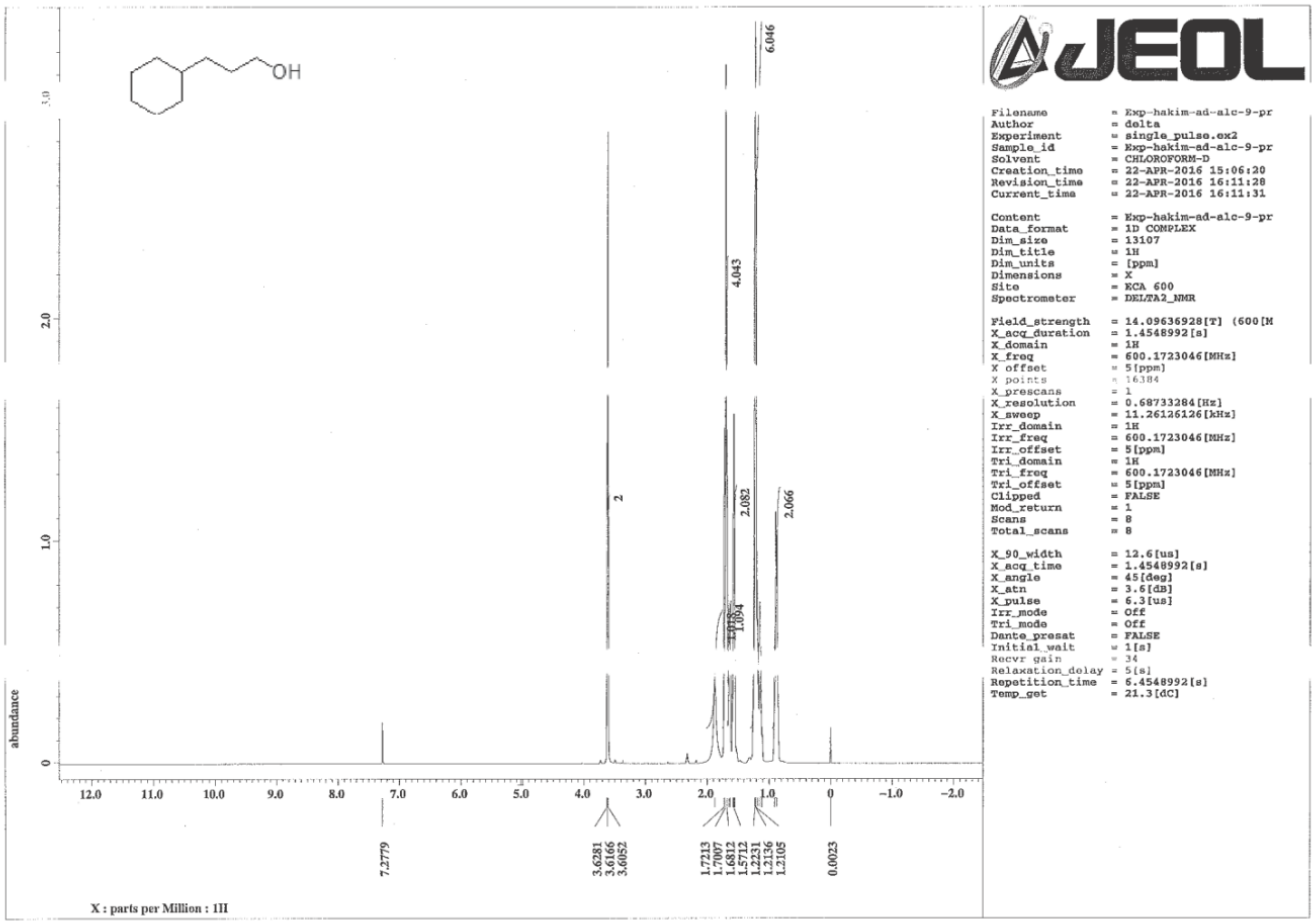
JEOL

Filename	= Exp-hakim-ad-alc-8-pr
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= Exp-hakim-ad-alc-8-pr
Solvent	= CHLOROFORM-D
Creation_time	= 22-APR-2016 14:40:08
Revision_time	= 22-APR-2016 15:50:38
Current_time	= 22-APR-2016 15:50:50
Content	= Exp-hakim-ad-alc-8-pr
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_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 30
Relaxation_delay	= 5[s]
Repetition_time	= 6.4548992[s]
Temp_get	= 21.3[dC]



JEOL

Filename	= Exp-hakim-ad-alc-8-ca
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= Exp-hakim-ad-alc-8-ca
Solvent	= CHLOROFORM-D
Creation_time	= 22-APR-2016 14:44:42
Revision_time	= 22-APR-2016 15:56:57
Current_time	= 22-APR-2016 15:57:02
Content	= Exp-hakim-ad-alc-8-ca
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	= 88
Total_scans	= 88
X_90_width	= 10.9[us]
X_acq_time	= 0.69206016[s]
X_angle	= 30[deg]
X_atn	= 7.5[dB]
X_pulse	= 3.633333333[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[s]
Recvr_gain	= 56
Relaxation_delay	= 2[s]
Repetition_time	= 2.69206016[s]
Temp_get	= 21.7[dC]



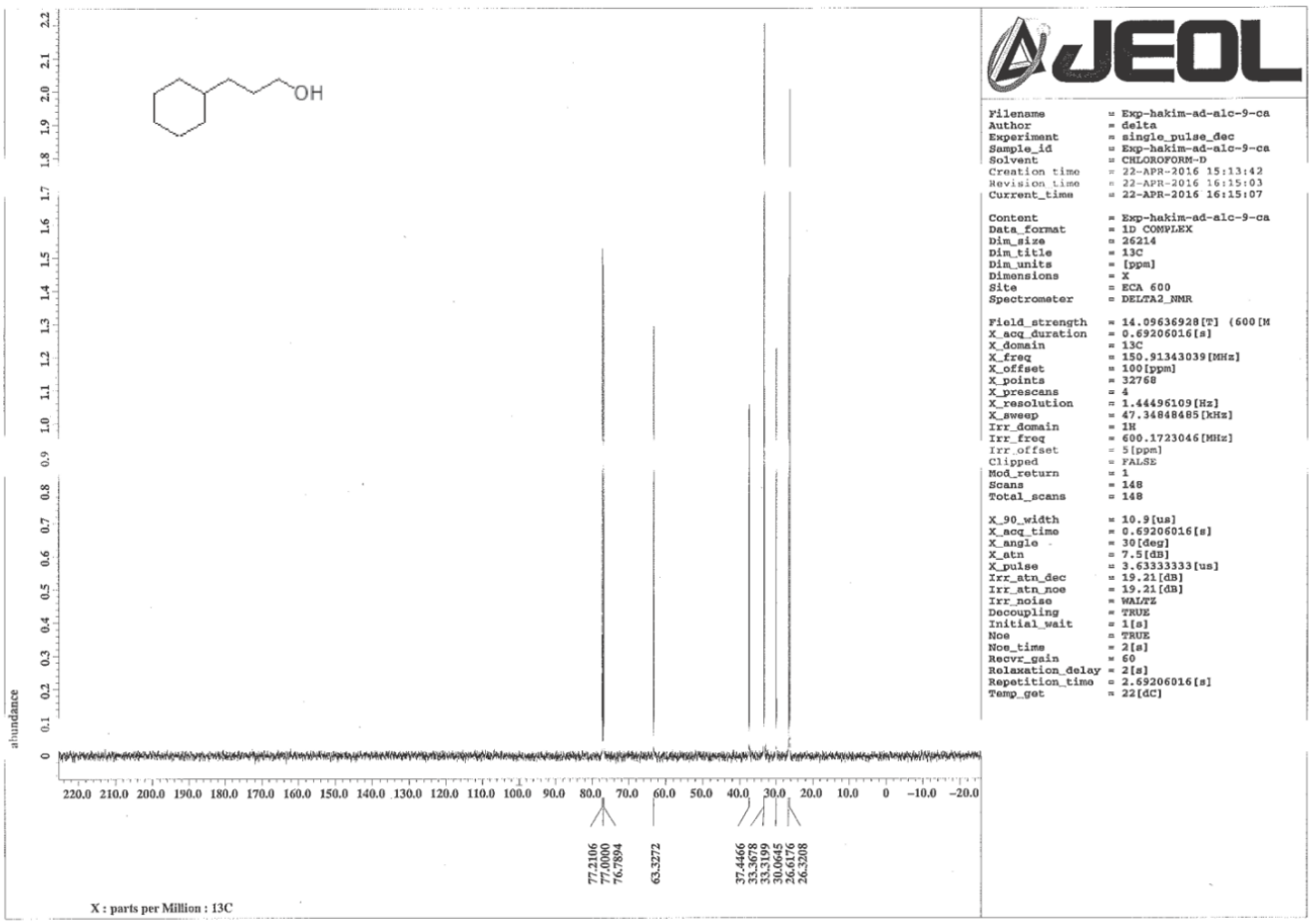
JEOL

```
Filename          = Exp-hakim-ad-alc-9-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-9-pr
Solvent           = CHLOROFORM-D
Creation_time     = 22-APR-2016 15:06:20
Revision_time     = 22-APR-2016 16:11:28
Current_time      = 22-APR-2016 16:11:31

Content           = Exp-hakim-ad-alc-9-pr
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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.3[dC]
```



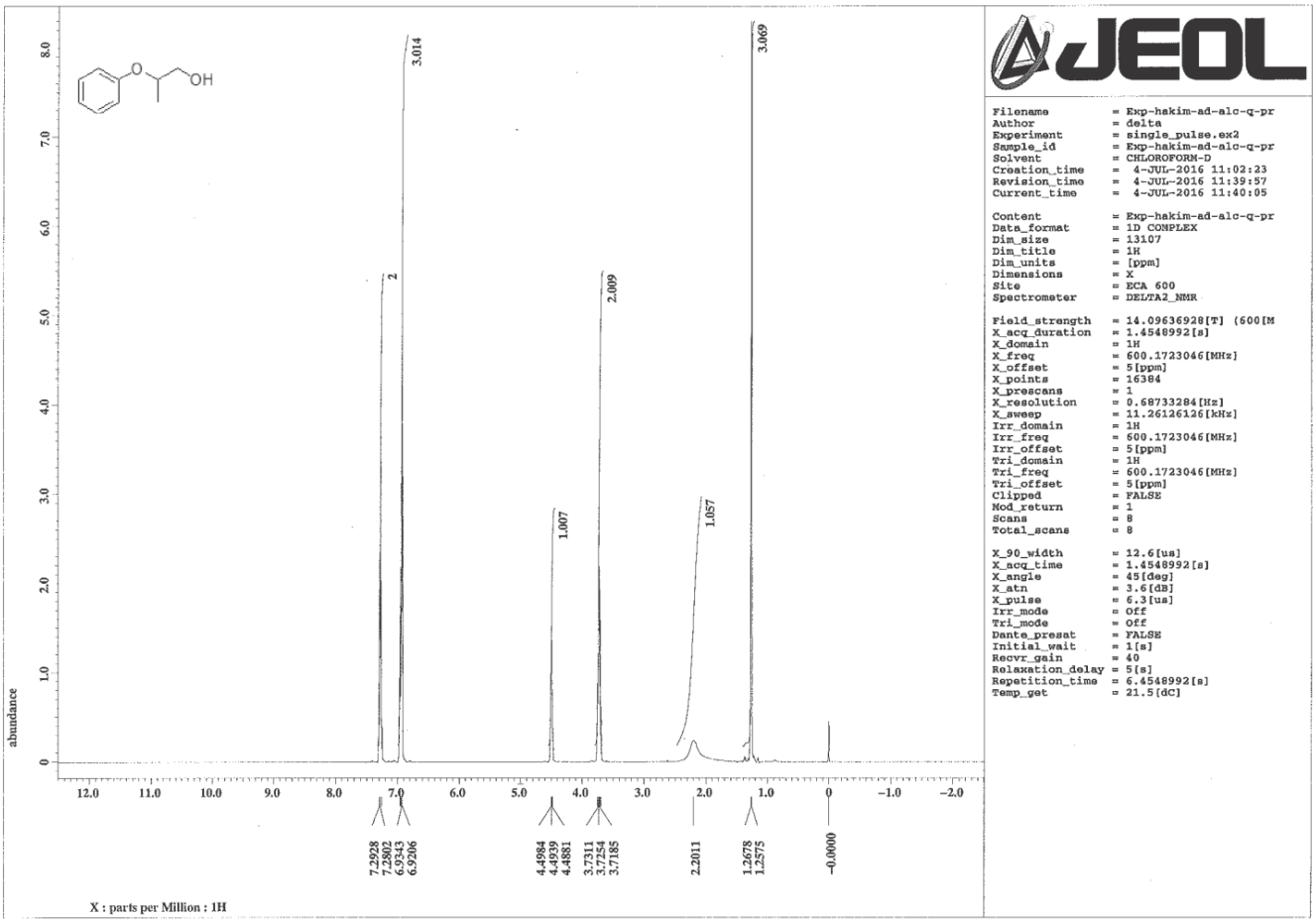
JEOL

```
Filename          = Exp-hakim-ad-alc-9-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-9-ca
Solvent           = CHLOROFORM-D
Creation_time     = 22-APR-2016 15:13:42
Revision_time     = 22-APR-2016 16:15:03
Current_time      = 22-APR-2016 16:15:07

Content           = Exp-hakim-ad-alc-9-ca
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             = 148
Total_scans       = 148

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[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.69206016[s]
Temp_get          = 22[dC]
```



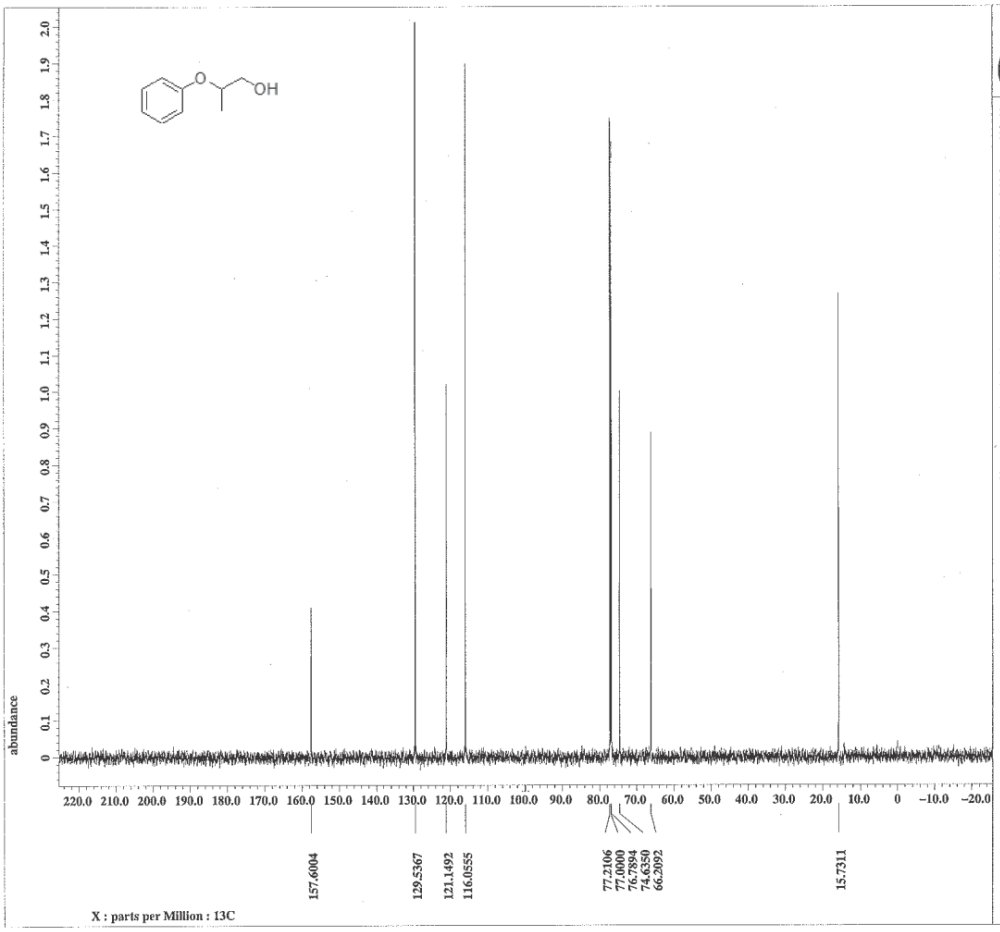
JEOL

```
Filename          = Exp-hakim-ad-alc-q-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-q-pr
Solvent           = CHLOROFORM-D
Creation_time     =  4-JUL-2016 11:02:23
Revision_time     =  4-JUL-2016 11:39:57
Current_time      =  4-JUL-2016 11:40:05

Content           = Exp-hakim-ad-alc-q-pr
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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.5[dC]
```



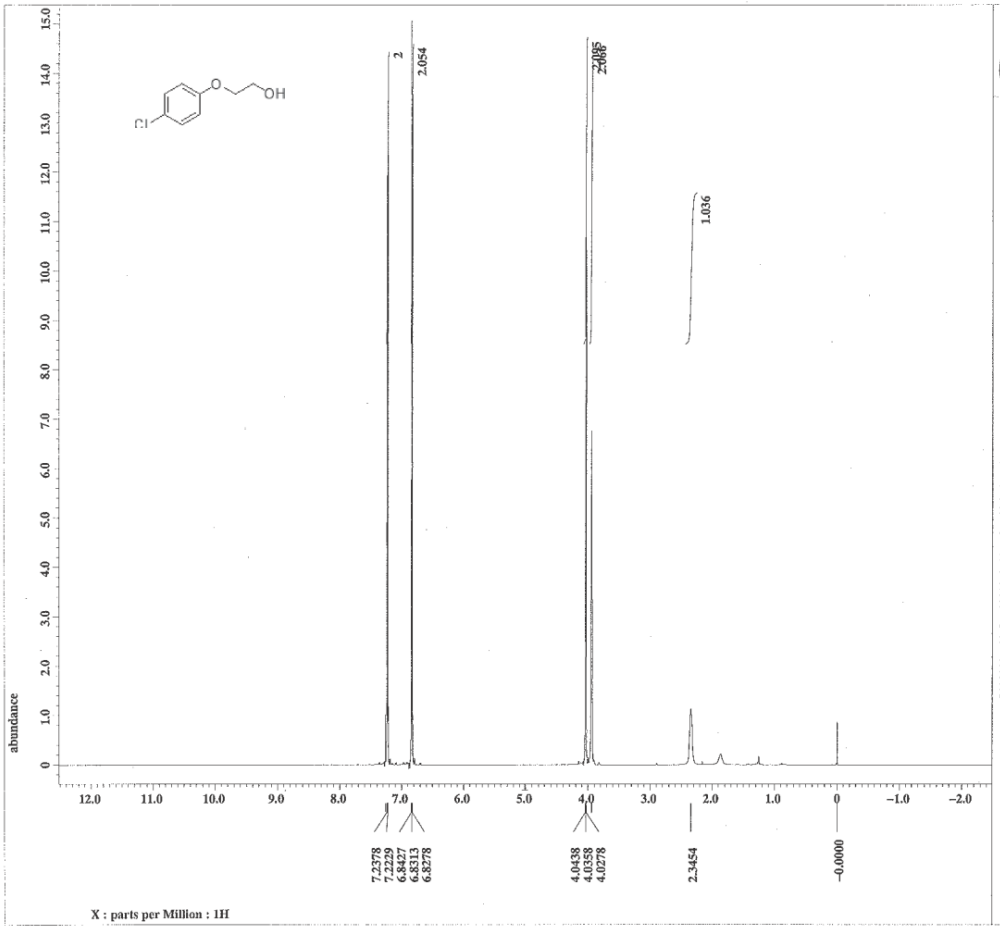
JEOL

```
Filename          = Exp-hakim-ad-alc-q-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-q-ca
Solvent           = CHLOROFORM-D
Creation_time     =  4-JUL-2016 11:07:30
Revision_time     =  4-JUL-2016 11:45:23
Current_time      =  4-JUL-2016 11:45:36

Content           = Exp-hakim-ad-alc-q-ca
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             = 81
Total_scans       = 81

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          = 22[dC]
```



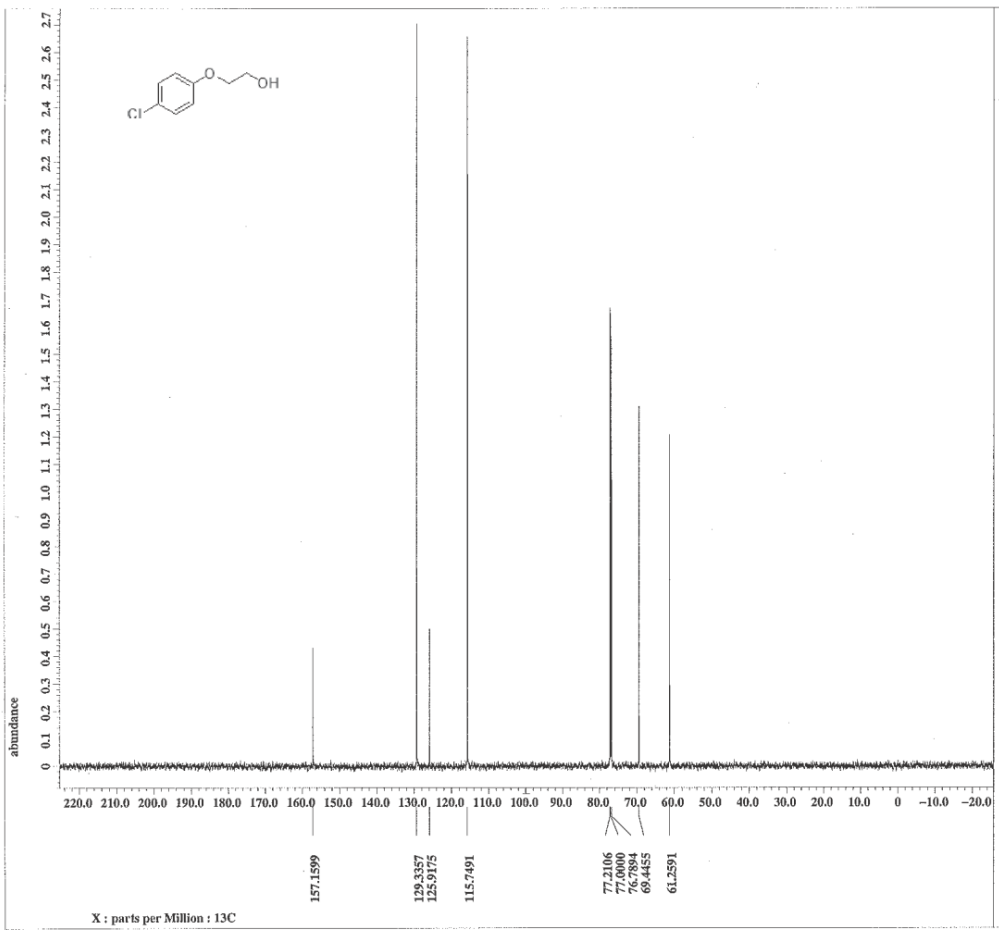
JEOL

```
Filename          = Exp-hakim-ad-alc-p-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-p-pr
Solvent           = CHLOROFORM-D
Creation_time     =  4-JUL-2016 10:27:51
Revision_time     =  4-JUL-2016 11:06:48
Current_time      =  4-JUL-2016 11:06:50

Content           = Exp-hakim-ad-alc-p-pr
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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 42
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.5[dC]
```



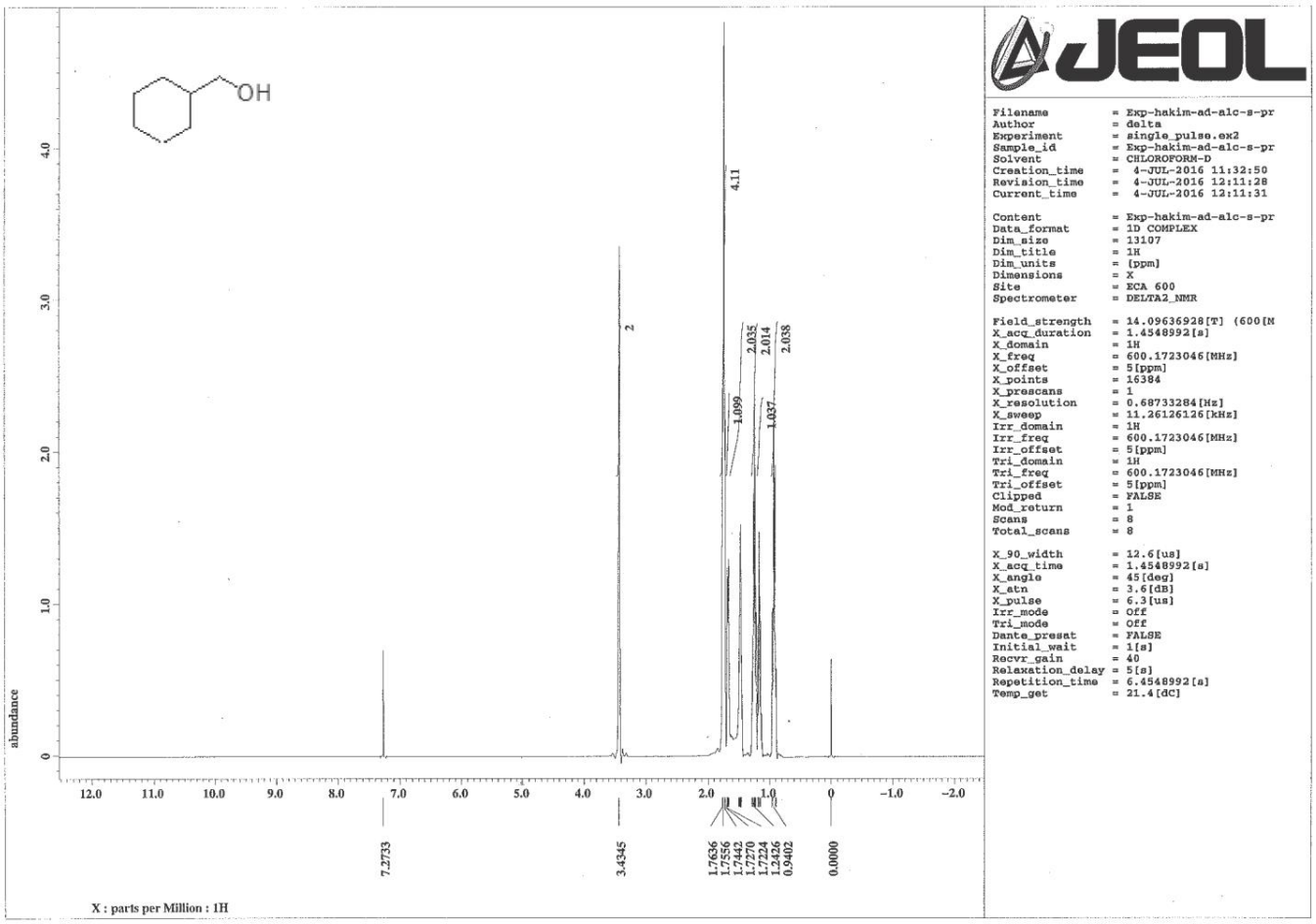
JEOL

```
Filename          = Exp-hakim-ad-alc-p-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-p-ca
Solvent           = CHLOROFORM-D
Creation_time     =  4-JUL-2016 10:36:58
Revision_time     =  4-JUL-2016 11:36:54
Current_time      =  4-JUL-2016 11:37:19

Content           = Exp-hakim-ad-alc-p-ca
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

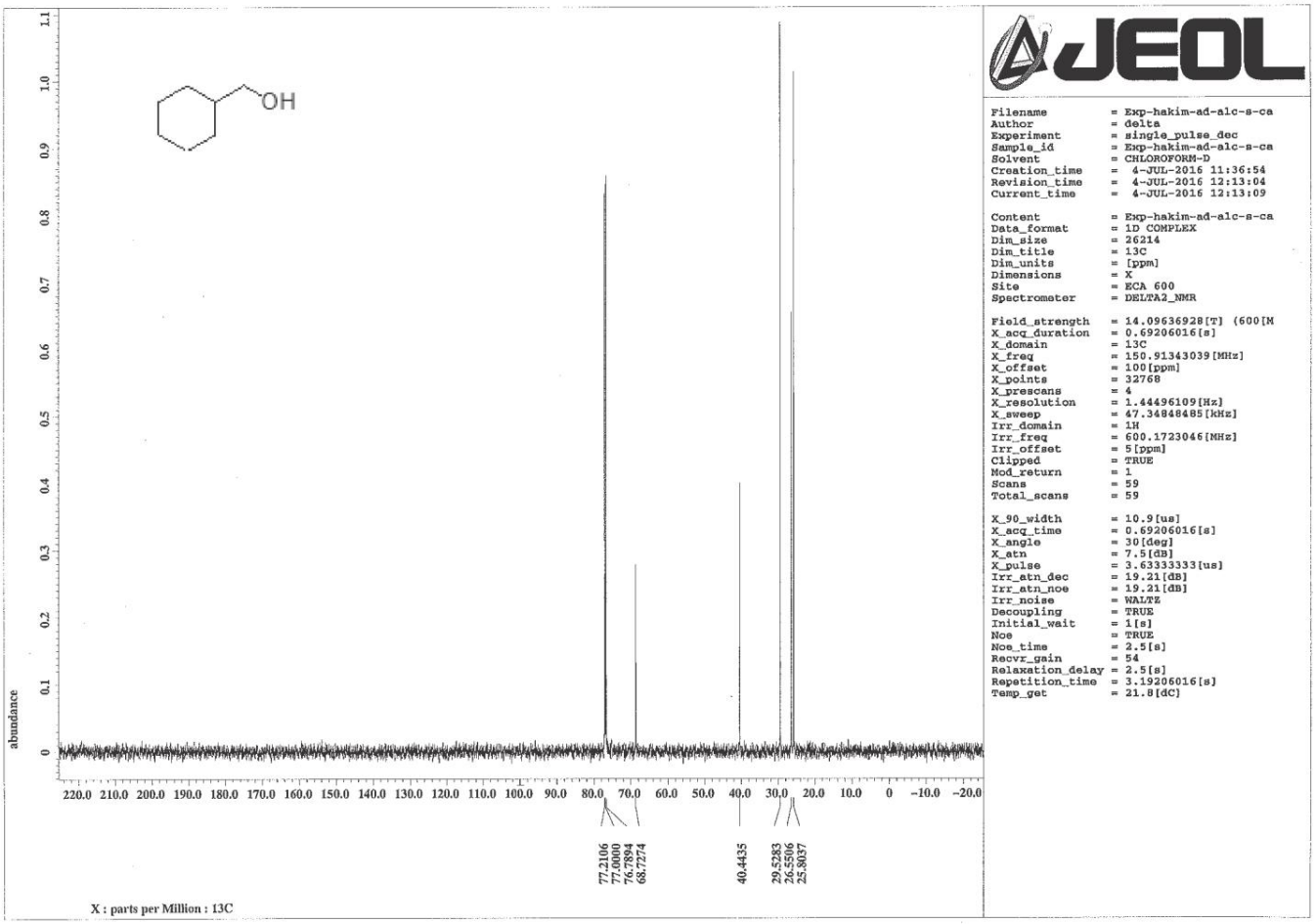
Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.69206016[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.44496109[Hz]
X_sweep           = 47.34848485[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 156
Total_scans       = 156

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          = 22.2[dC]
```



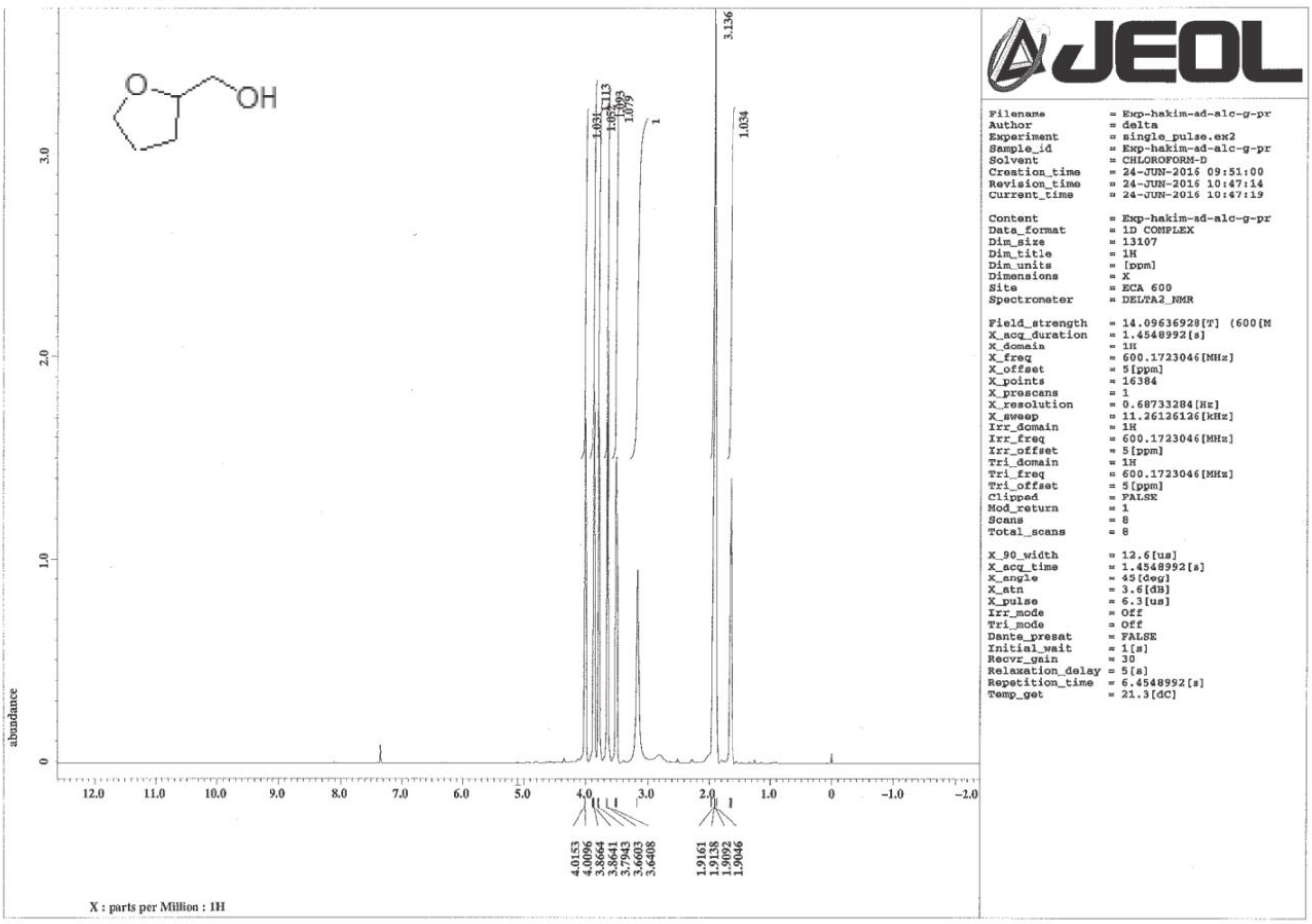
JEOL

Filename	= Exp-hakim-ad-alc-s-pr
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= Exp-hakim-ad-alc-s-pr
Solvent	= CHLOROFORM-D
Creation_time	= 4-JUL-2016 11:32:50
Revision_time	= 4-JUL-2016 12:11:28
Current_time	= 4-JUL-2016 12:11:31
Content	= Exp-hakim-ad-alc-s-pr
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_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 6.4548992[s]
Temp_get	= 21.4[dC]



JEOL

Filename	= Exp-hakim-ad-alc-s-ca
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= Exp-hakim-ad-alc-s-ca
Solvent	= CHLOROFORM-D
Creation_time	= 4-JUL-2016 11:36:54
Revision_time	= 4-JUL-2016 12:13:04
Current_time	= 4-JUL-2016 12:13:09
Content	= Exp-hakim-ad-alc-s-ca
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	= 59
Total_scans	= 59
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	= 54
Relaxation_delay	= 2.5[s]
Repetition_time	= 3.19206016[s]
Temp_get	= 21.8[dC]



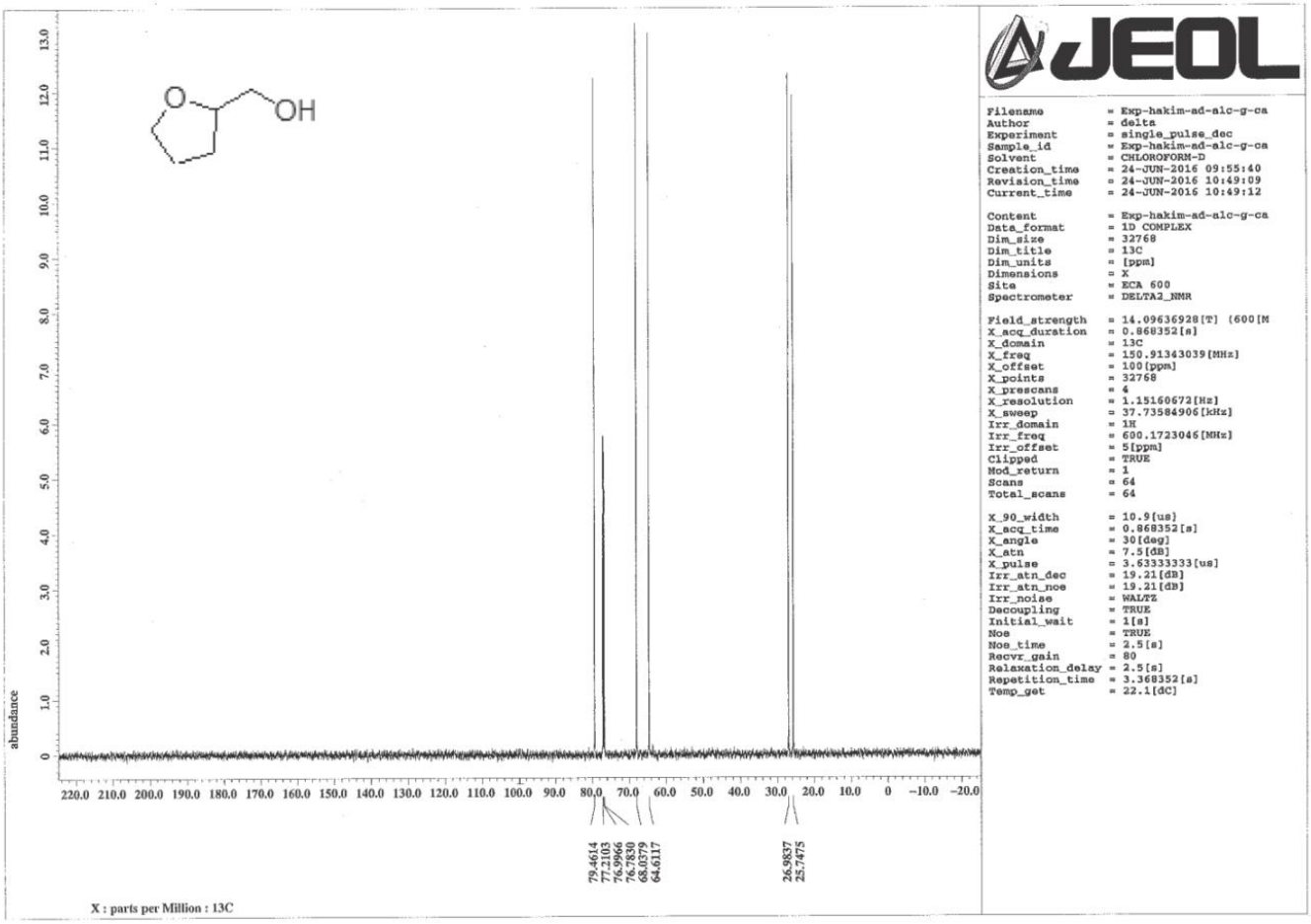
JEOL

```
Filename          = Exp-hakim-ad-alc-g-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-g-pr
Solvent           = CHLOROFORM-D
Creation_time     = 24-JUN-2016 09:51:00
Revision_time     = 24-JUN-2016 10:47:14
Current_time      = 24-JUN-2016 10:47:19

Content           = Exp-hakim-ad-alc-g-pr
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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 30
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.3[dC]
```



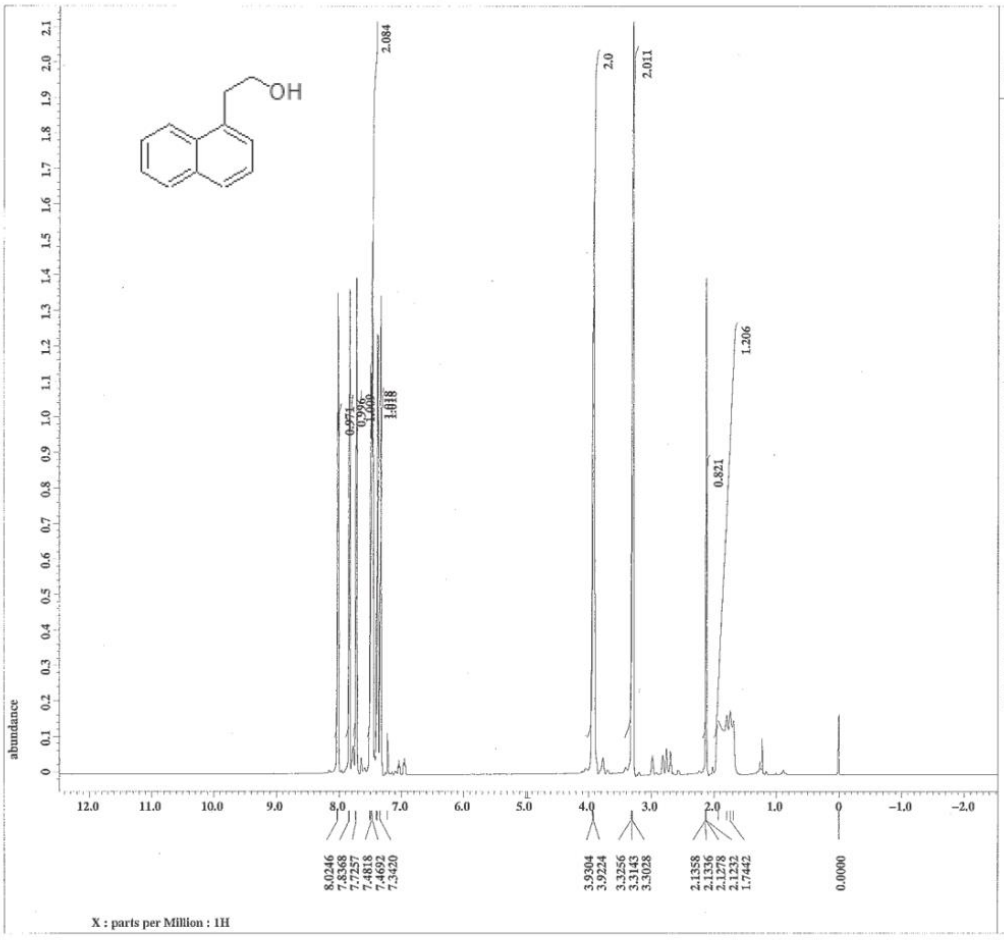
JEOL

```
Filename          = Exp-hakim-ad-alc-g-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-g-ca
Solvent           = CHLOROFORM-D
Creation_time     = 24-JUN-2016 09:55:40
Revision_time     = 24-JUN-2016 10:49:09
Current_time      = 24-JUN-2016 10:49:12

Content           = Exp-hakim-ad-alc-g-ca
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.868352[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.15160672[Hz]
X_sweep           = 37.73584906[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 10.9[us]
X_acq_time        = 0.868352[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        = 80
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 22.1[dC]
```

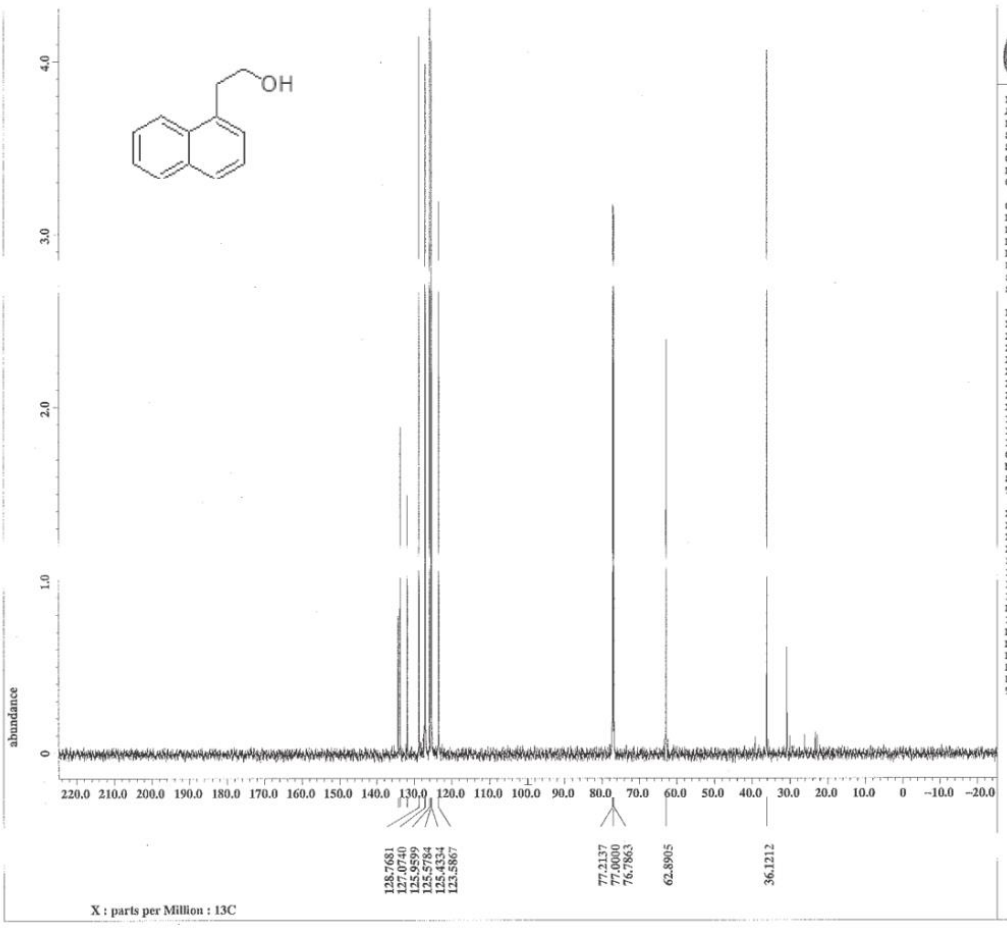


Filename = Exp-hakim-ad-alc-5-pr
Author = delta
Experiment = single_pulse.ex2
Sample_id = Exp-hakim-ad-alc-5-pr
Solvent = CHLOROFORM-D
Creation_time = 1-APR-2016 16:46:57
Revision_time = 5-APR-2016 22:35:26
Current_time = 5-APR-2016 22:35:53

Content = Exp-hakim-ad-alc-5-pr
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_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 36
Relaxation_delay = 5[s]
Repetition_time = 6.4548992[s]
Temp_get = 21.3[dC]

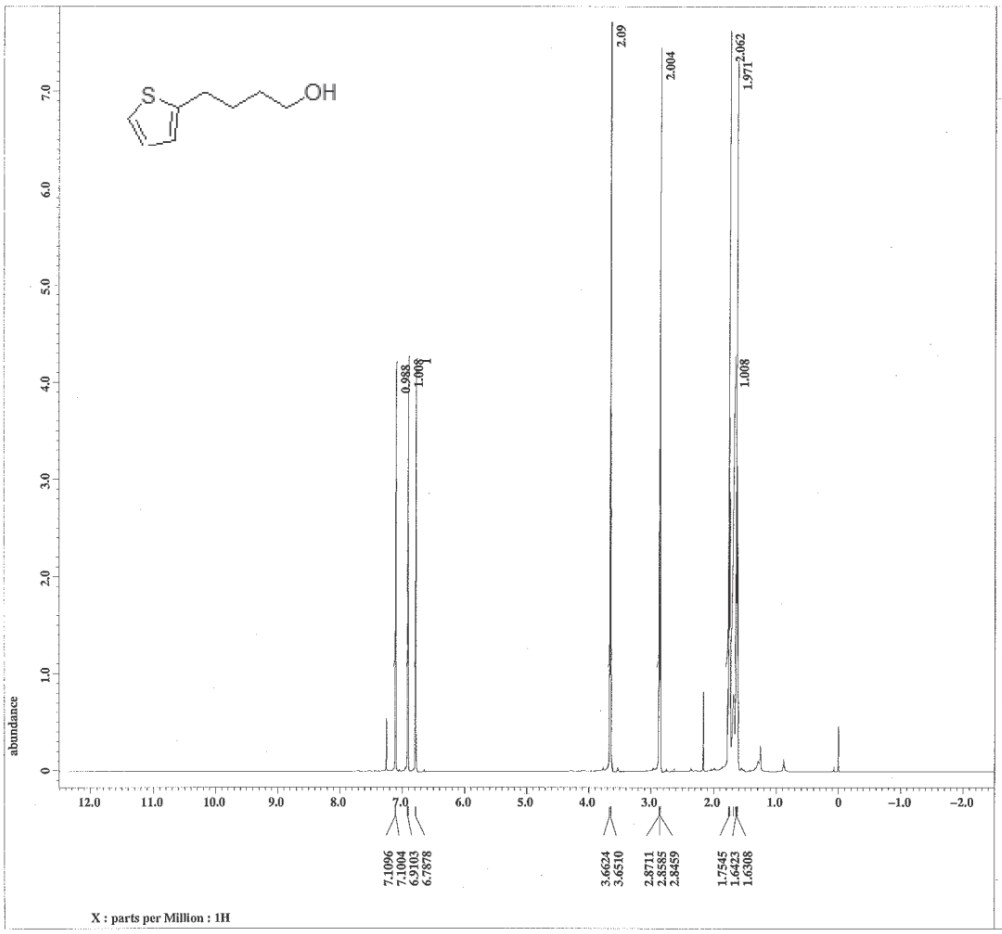


Filename = Exp-hakim-ad-alc-5-ca
Author = delta
Experiment = single_pulse_dec
Sample_id = Exp-hakim-ad-alc-5-ca
Solvent = CHLOROFORM-D
Creation_time = 1-APR-2016 17:06:06
Revision_time = 5-APR-2016 22:37:27
Current_time = 5-APR-2016 22:37:31

Content = Exp-hakim-ad-alc-5-ca
Data_format = 1D COMPLEX
Dim_size = 32768
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 600
Spectrometer = DELTA2_NMR

Field_strength = 14.09636928[T] (600[M
X_acq_duration = 0.868352[s]
X_domain = 13C
X_freq = 150.91343039[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.15160672[Hz]
X_sweep = 37.73584906[kHz]
Irr_domain = 1H
Irr_freq = 600.1723046[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 326
Total_scans = 326

X_90_width = 10.9[us]
X_acq_time = 0.868352[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 = 80
Relaxation_delay = 2.5[s]
Repetition_time = 3.368352[s]
Temp_get = 21.6[dC]



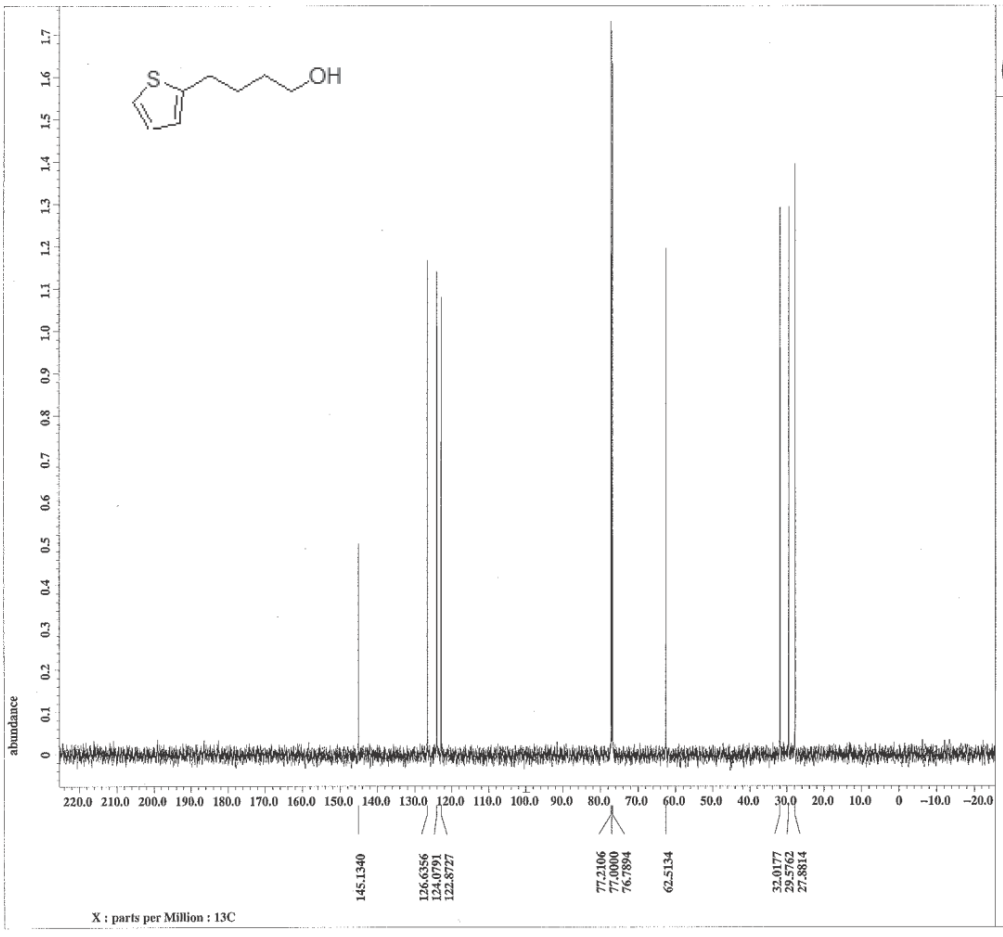
JEOL

```
Filename          = Exp-hakim-ad-alc-m-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-m-pr
Solvent           = CHLOROFORM-D
Creation_time     = 11-JUL-2016 15:40:45
Revision_time     = 11-JUL-2016 16:18:54
Current_time      = 11-JUL-2016 16:19:10

Content           = Exp-hakim-ad-alc-m-pr
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
Nod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 12.5[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]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21[dC]
```



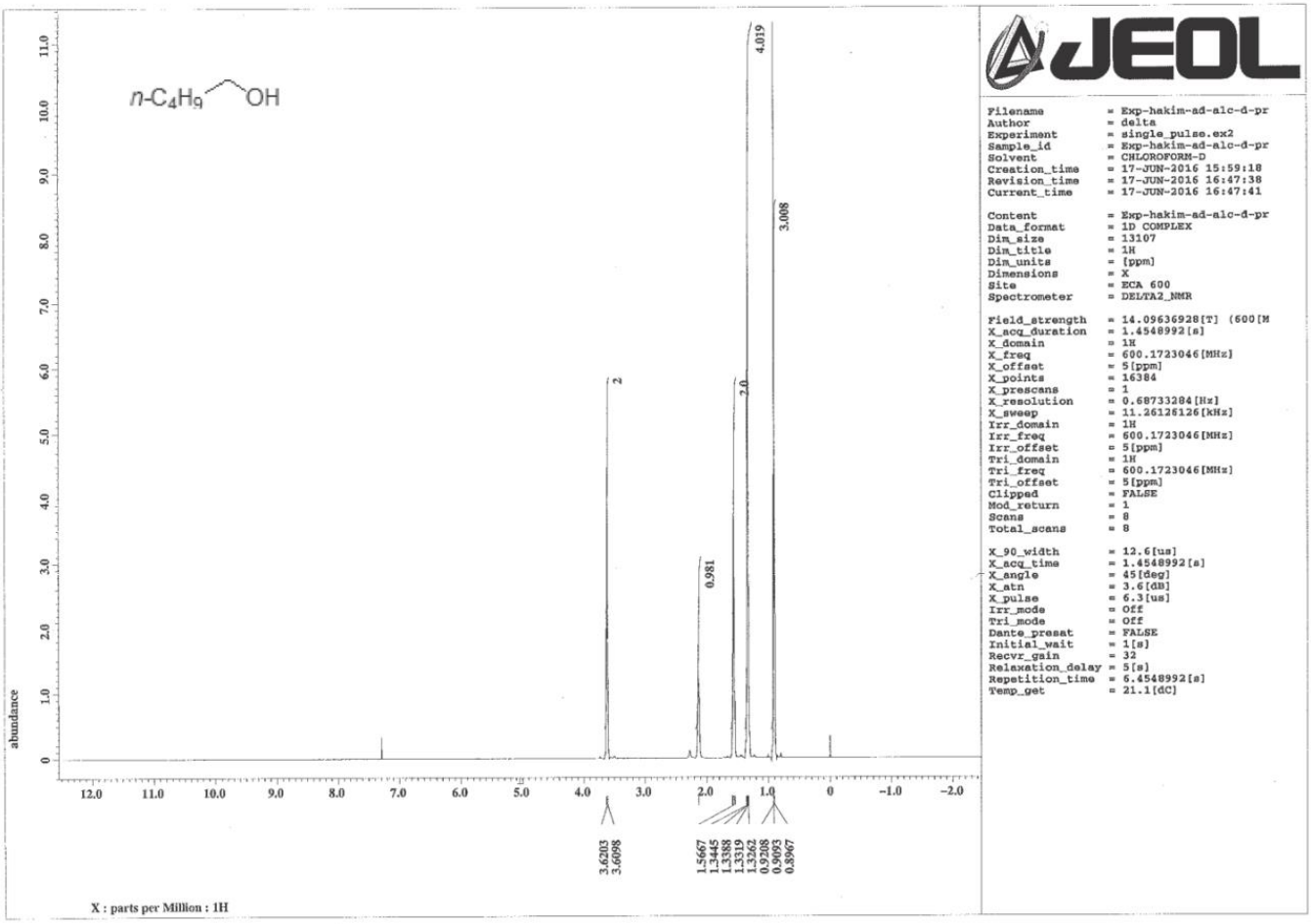
JEOL

```
Filename          = Exp-hakim-ad-alc-m-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-m-ca
Solvent           = CHLOROFORM-D
Creation_time     = 11-JUL-2016 15:45:11
Revision_time     = 11-JUL-2016 16:23:25
Current_time      = 11-JUL-2016 16:23:36

Content           = Exp-hakim-ad-alc-m-ca
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
Nod_return        = 1
Scans             = 68
Total_scans       = 68

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          = 21.2[dC]
```



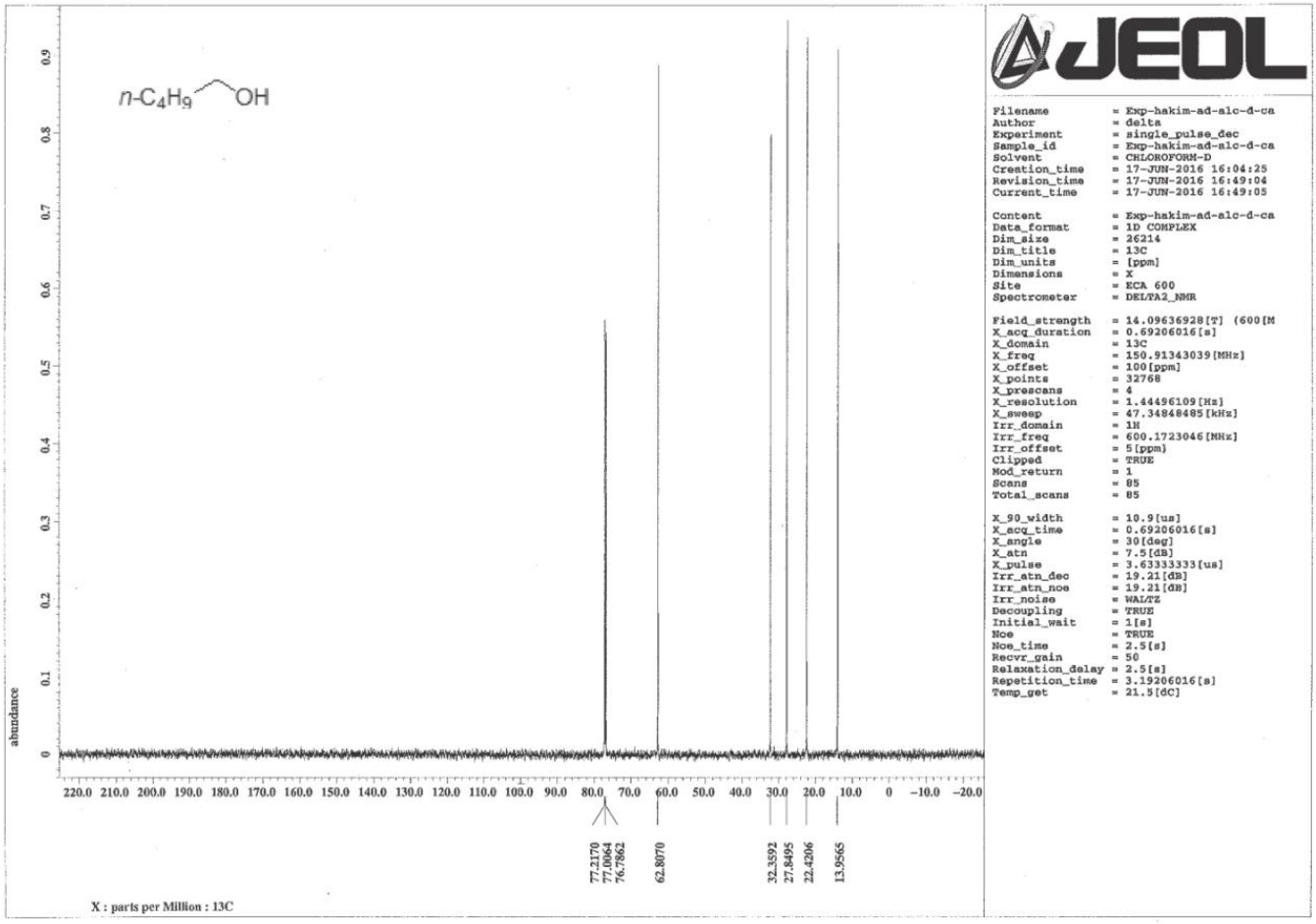
JEOL

```
Filename          = Exp-hakim-ad-alc-d-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-d-pr
Solvent           = CHLOROFORM-D
Creation_time     = 17-JUN-2016 15:59:18
Revision_time     = 17-JUN-2016 16:47:38
Current_time      = 17-JUN-2016 16:47:41

Content           = Exp-hakim-ad-alc-d-pr
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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 32
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.1[dC]
```



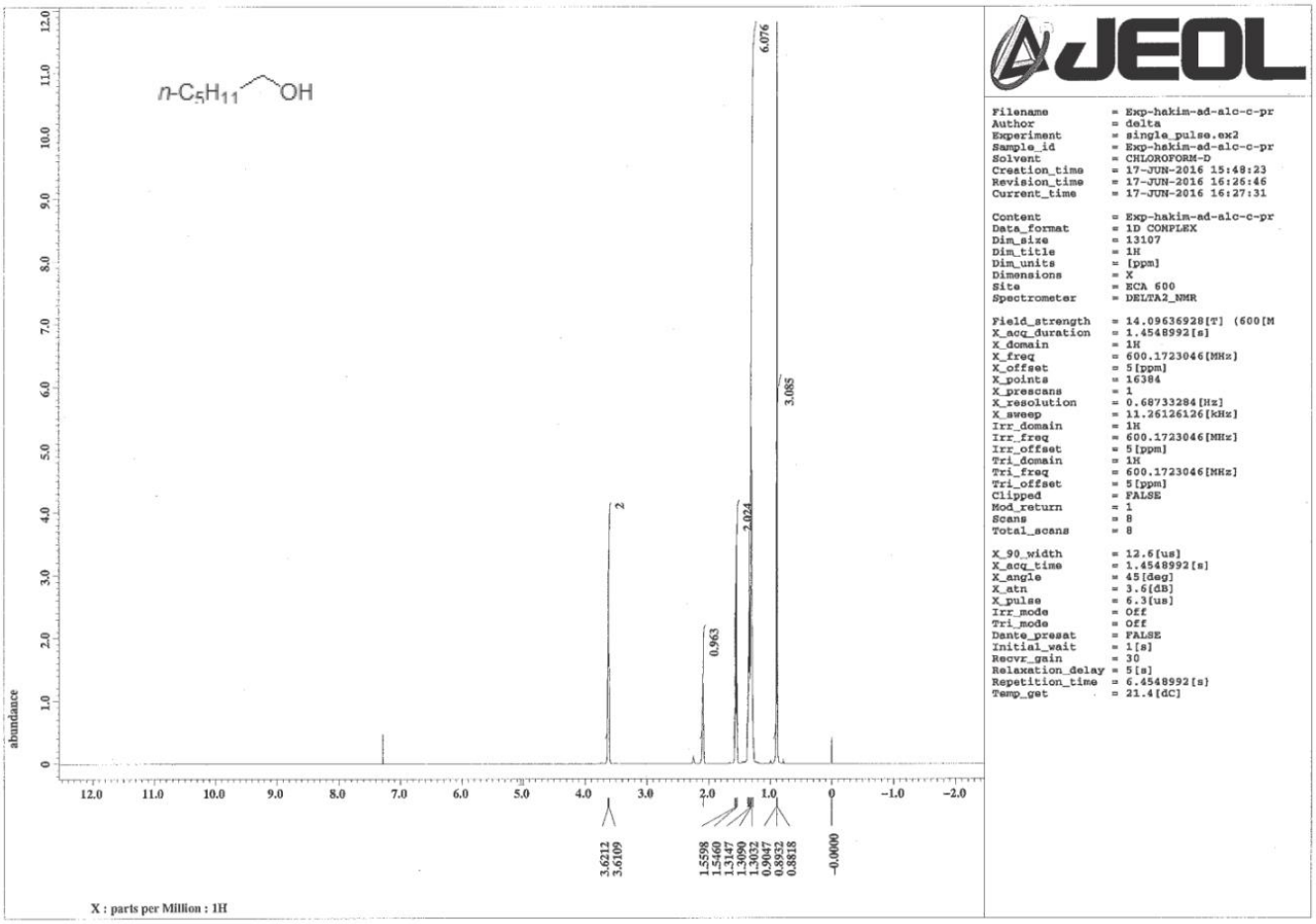
JEOL

```
Filename          = Exp-hakim-ad-alc-d-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-d-ca
Solvent           = CHLOROFORM-D
Creation_time     = 17-JUN-2016 16:04:25
Revision_time     = 17-JUN-2016 16:49:04
Current_time      = 17-JUN-2016 16:49:05

Content           = Exp-hakim-ad-alc-d-ca
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        = 50
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.19206016[s]
Temp_get          = 21.5[dC]
```



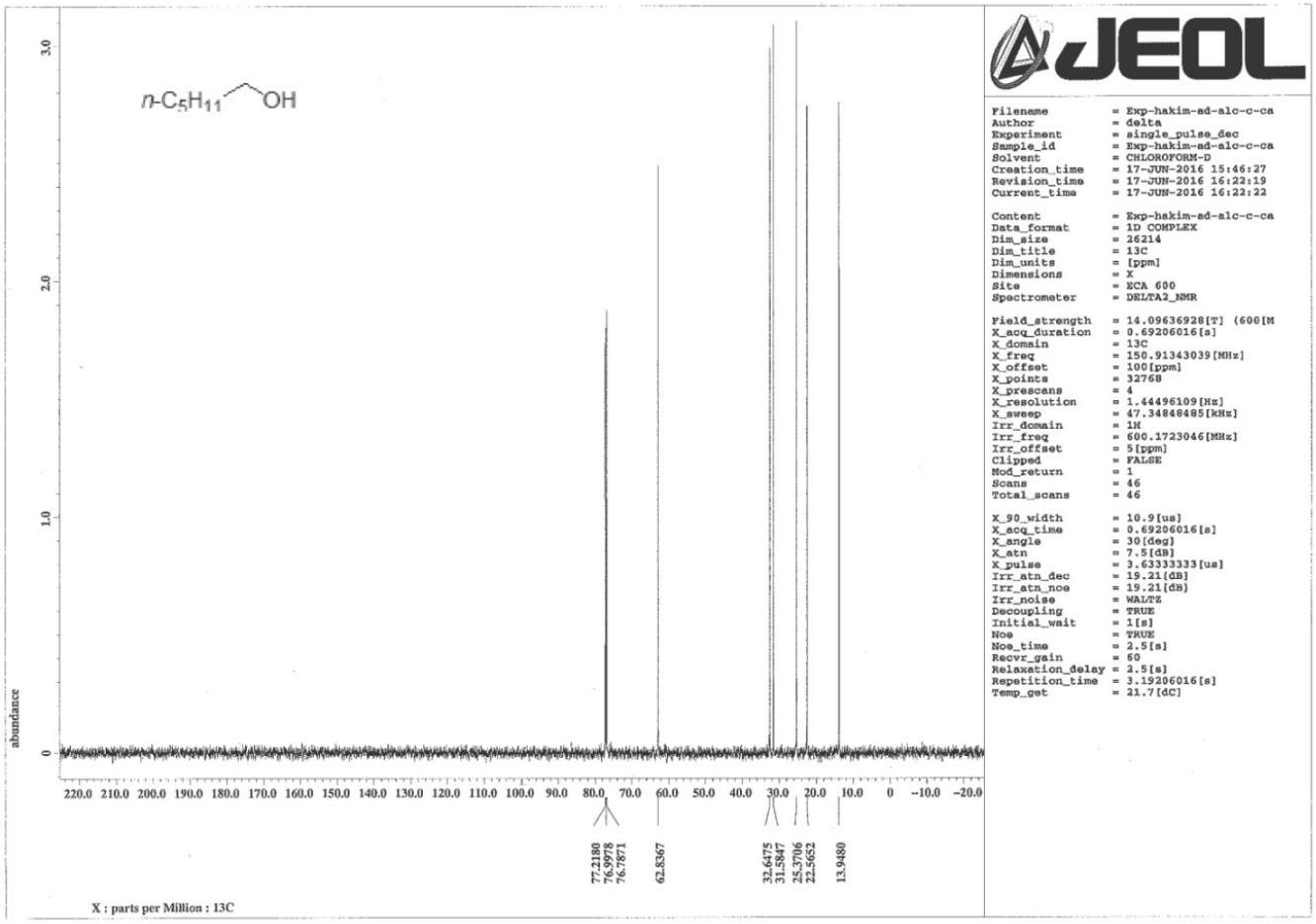
JEOL

```
Filename          = Exp-hakim-ad-alc-c-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-c-pr
Solvent           = CHLOROFORM-D
Creation_time     = 17-JUN-2016 15:48:23
Revision_time     = 17-JUN-2016 16:26:46
Current_time      = 17-JUN-2016 16:27:31

Content           = Exp-hakim-ad-alc-c-pr
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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 30
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.4[dC]
```



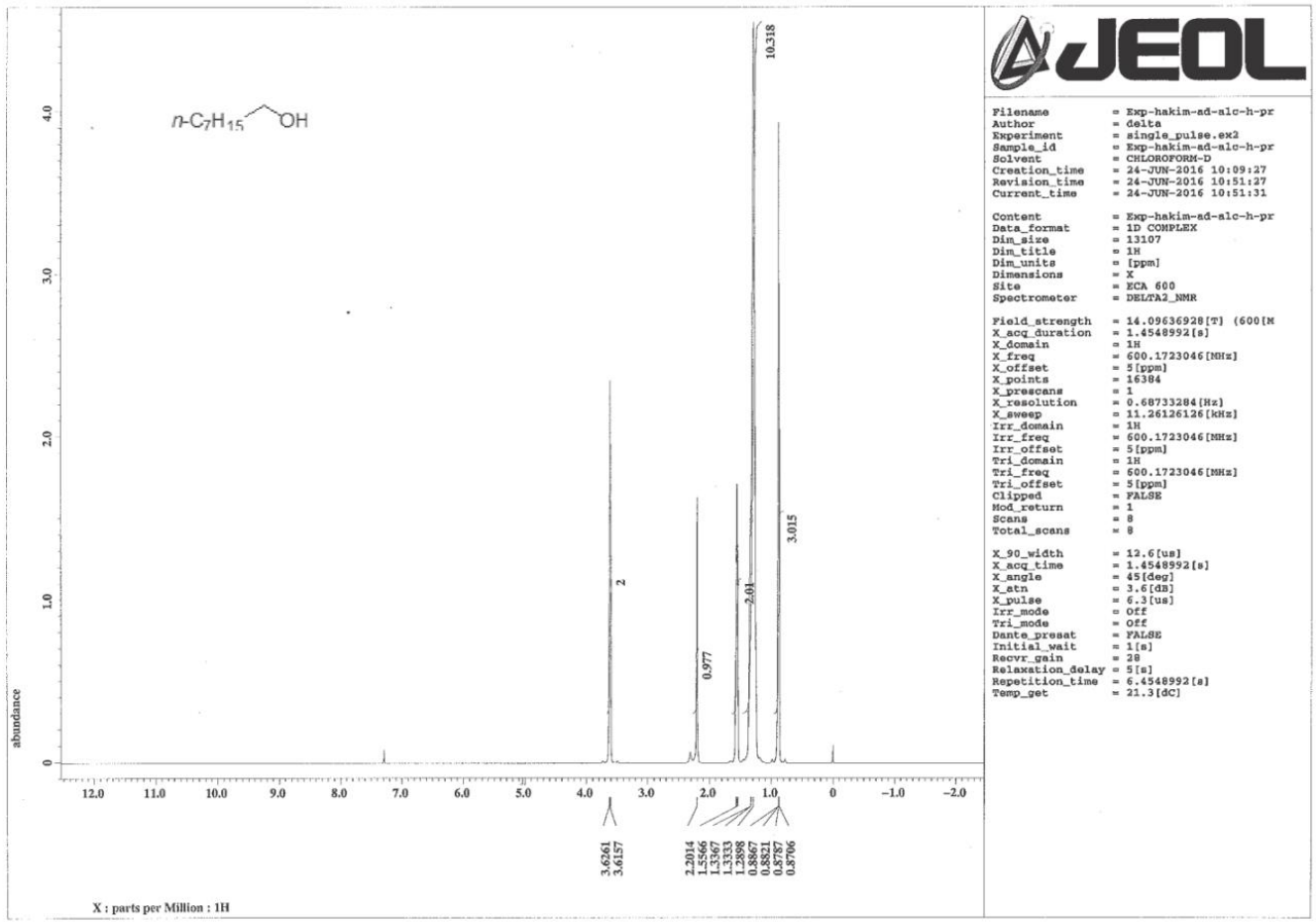
JEOL

```
Filename          = Exp-hakim-ad-alc-c-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-c-ca
Solvent           = CHLOROFORM-D
Creation_time     = 17-JUN-2016 15:46:27
Revision_time     = 17-JUN-2016 16:22:19
Current_time      = 17-JUN-2016 16:22:22

Content           = Exp-hakim-ad-alc-c-ca
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             = 46
Total_scans       = 46

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          = 21.7[dC]
```



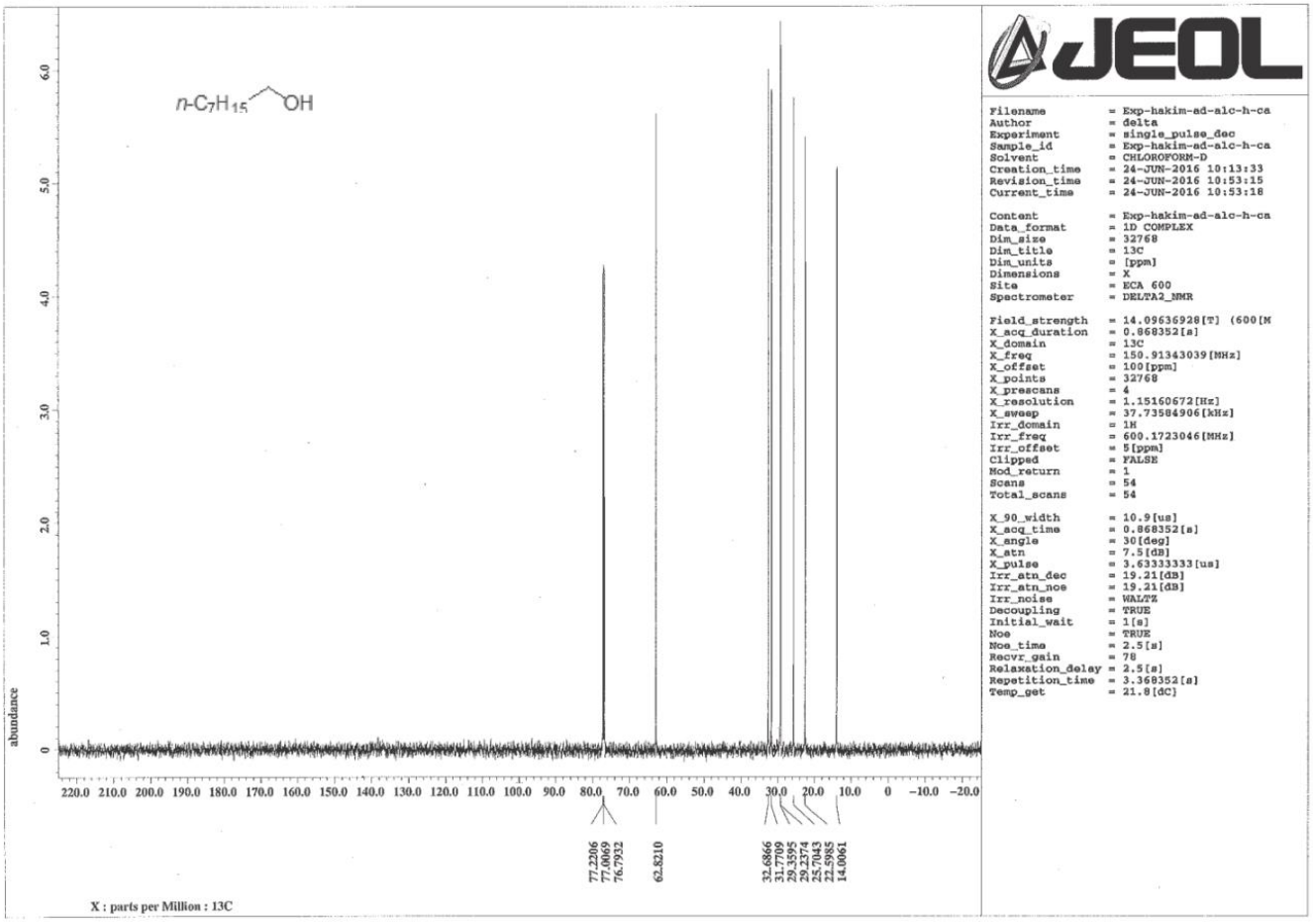
JEOL

```
Filename          = Exp-hakim-ad-alc-h-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-h-pr
Solvent           = CHLOROFORM-D
Creation_time     = 24-JUN-2016 10:09:27
Revision_time     = 24-JUN-2016 10:51:27
Current_time      = 24-JUN-2016 10:51:31

Content           = Exp-hakim-ad-alc-h-pr
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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 28
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.3[dC]
```



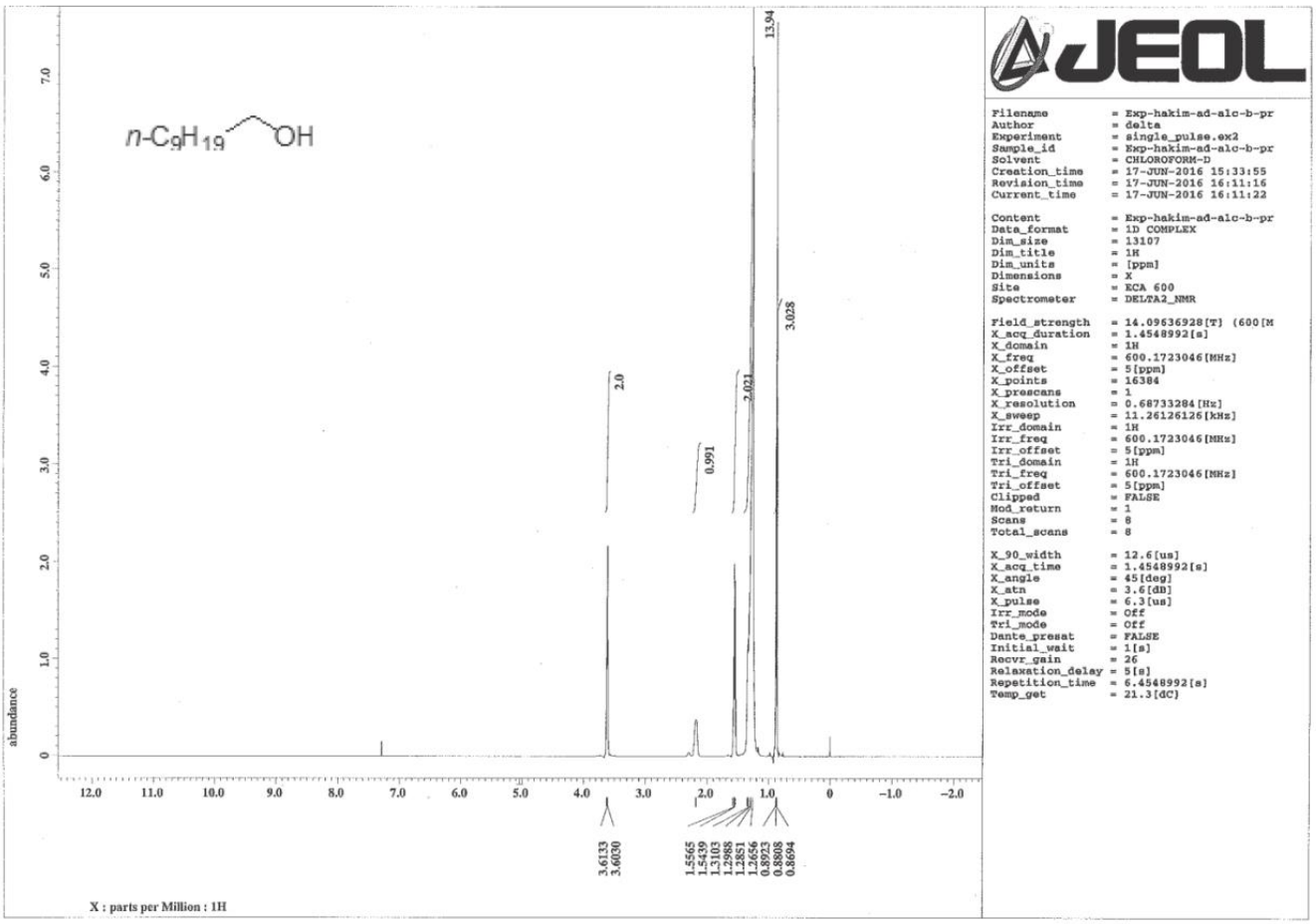
JEOL

```
Filename          = Exp-hakim-ad-alc-h-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-h-ca
Solvent           = CHLOROFORM-D
Creation_time     = 24-JUN-2016 10:13:33
Revision_time     = 24-JUN-2016 10:53:15
Current_time      = 24-JUN-2016 10:53:18

Content           = Exp-hakim-ad-alc-h-ca
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.868352[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.15160672[Hz]
X_sweep           = 37.73584906[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 54
Total_scans       = 54

X_90_width        = 10.9[us]
X_acq_time        = 0.868352[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        = 78
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 21.8[dC]
```



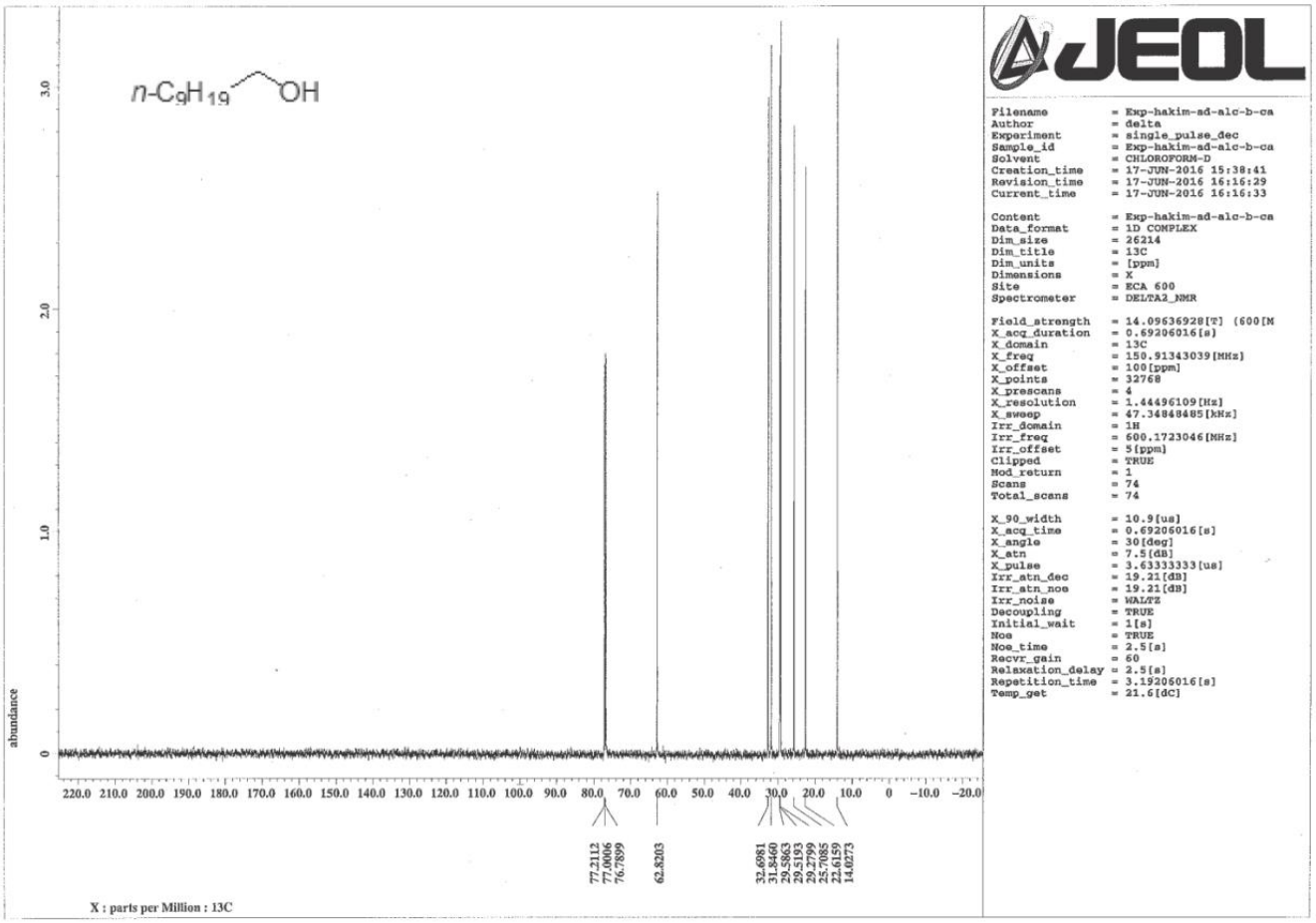
JEOL

```
Filename          = Exp-hakim-ad-alc-b-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-b-pr
Solvent           = CHLOROFORM-D
Creation_time     = 17-JUN-2016 15:33:55
Revision_time     = 17-JUN-2016 16:11:16
Current_time      = 17-JUN-2016 16:11:22

Content           = Exp-hakim-ad-alc-b-pr
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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 26
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.3[dC]
```



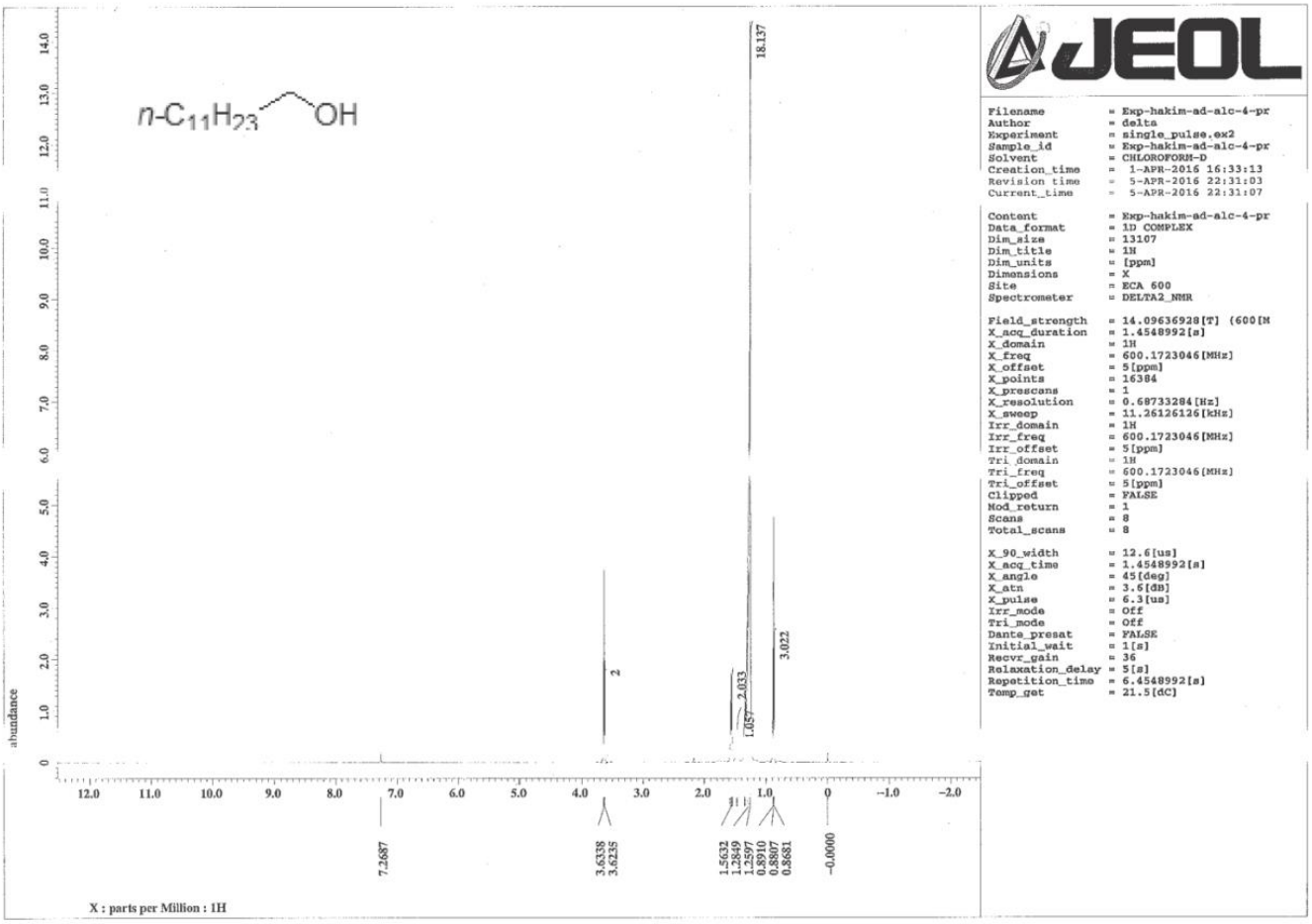
JEOL

```
Filename          = Exp-hakim-ad-alc-b-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-b-ca
Solvent           = CHLOROFORM-D
Creation_time     = 17-JUN-2016 15:38:41
Revision_time     = 17-JUN-2016 16:16:29
Current_time      = 17-JUN-2016 16:16:33

Content           = Exp-hakim-ad-alc-b-ca
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             = 74
Total_scans       = 74

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          = 21.6[dC]
```

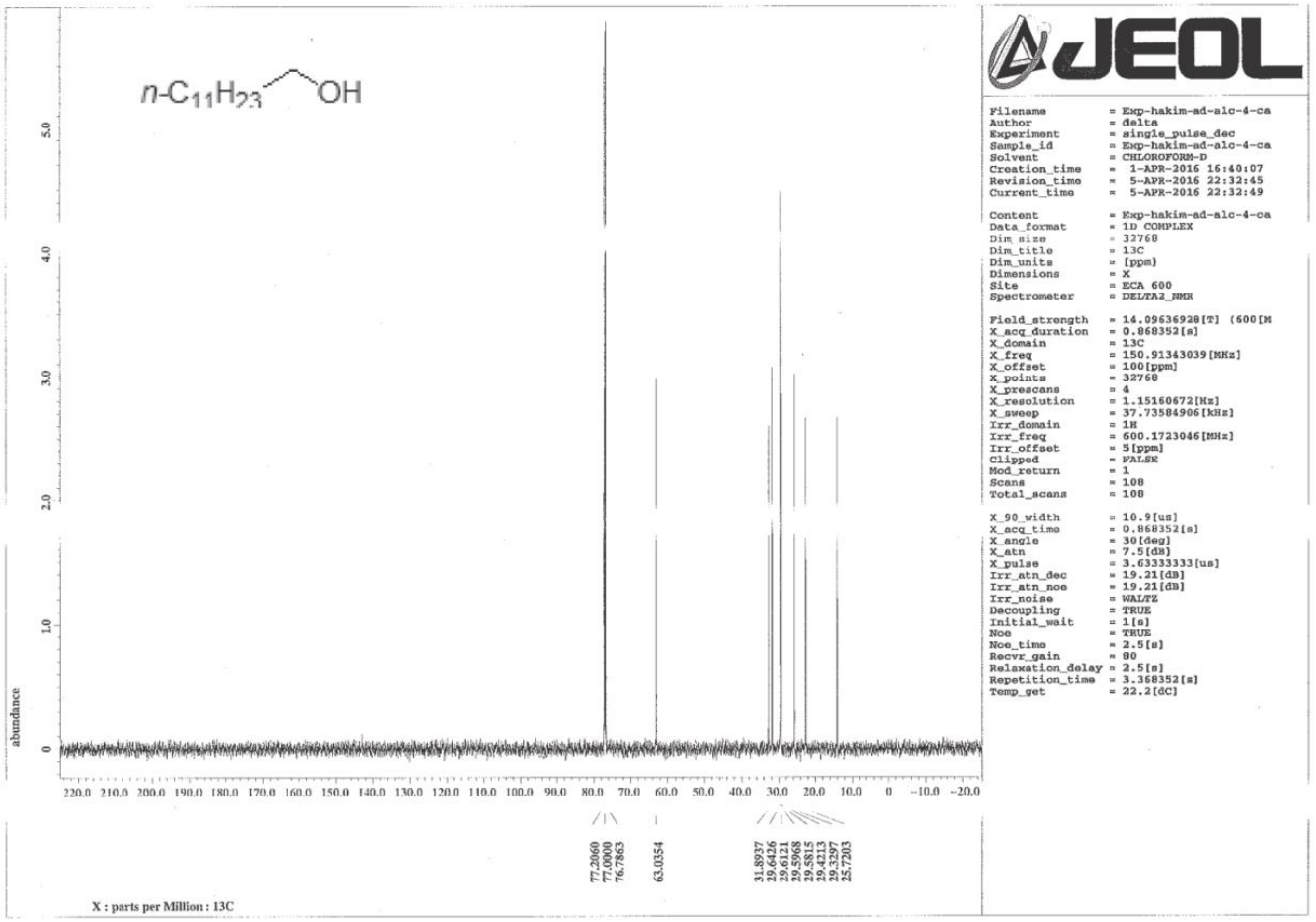



```
Filename          = Exp-hakim-ad-alc-4-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-4-pr
Solvent           = CHLOROFORM-D
Creation_time     =  1-APR-2016 16:33:13
Revision_time     =  5-APR-2016 22:31:03
Current_time      =  5-APR-2016 22:31:07

Content           = Exp-hakim-ad-alc-4-pr
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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.5[dC]
```

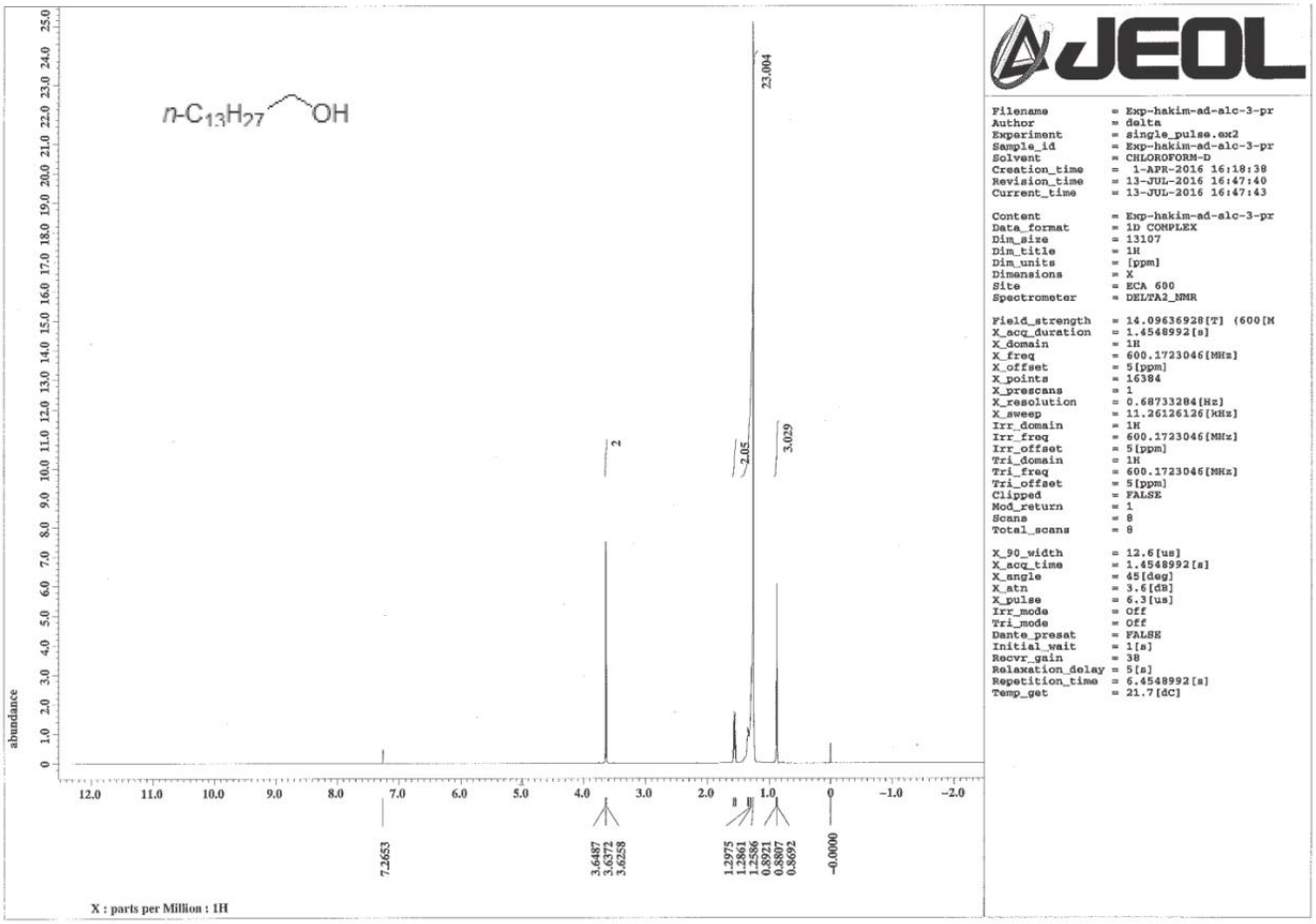



```
Filename          = Exp-hakim-ad-alc-4-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-4-ca
Solvent           = CHLOROFORM-D
Creation_time     =  1-APR-2016 16:40:07
Revision_time     =  5-APR-2016 22:32:45
Current_time      =  5-APR-2016 22:32:49

Content           = Exp-hakim-ad-alc-4-ca
Data_format       = 1D COMPLEX
Dim_size          = 32760
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.868352[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32760
X_prescans        = 4
X_resolution      = 1.15160672[Hz]
X_sweep           = 37.73584906[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 108
Total_scans       = 108

X_90_width        = 10.9[us]
X_acq_time        = 0.868352[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        = 80
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 22.2[dC]
```

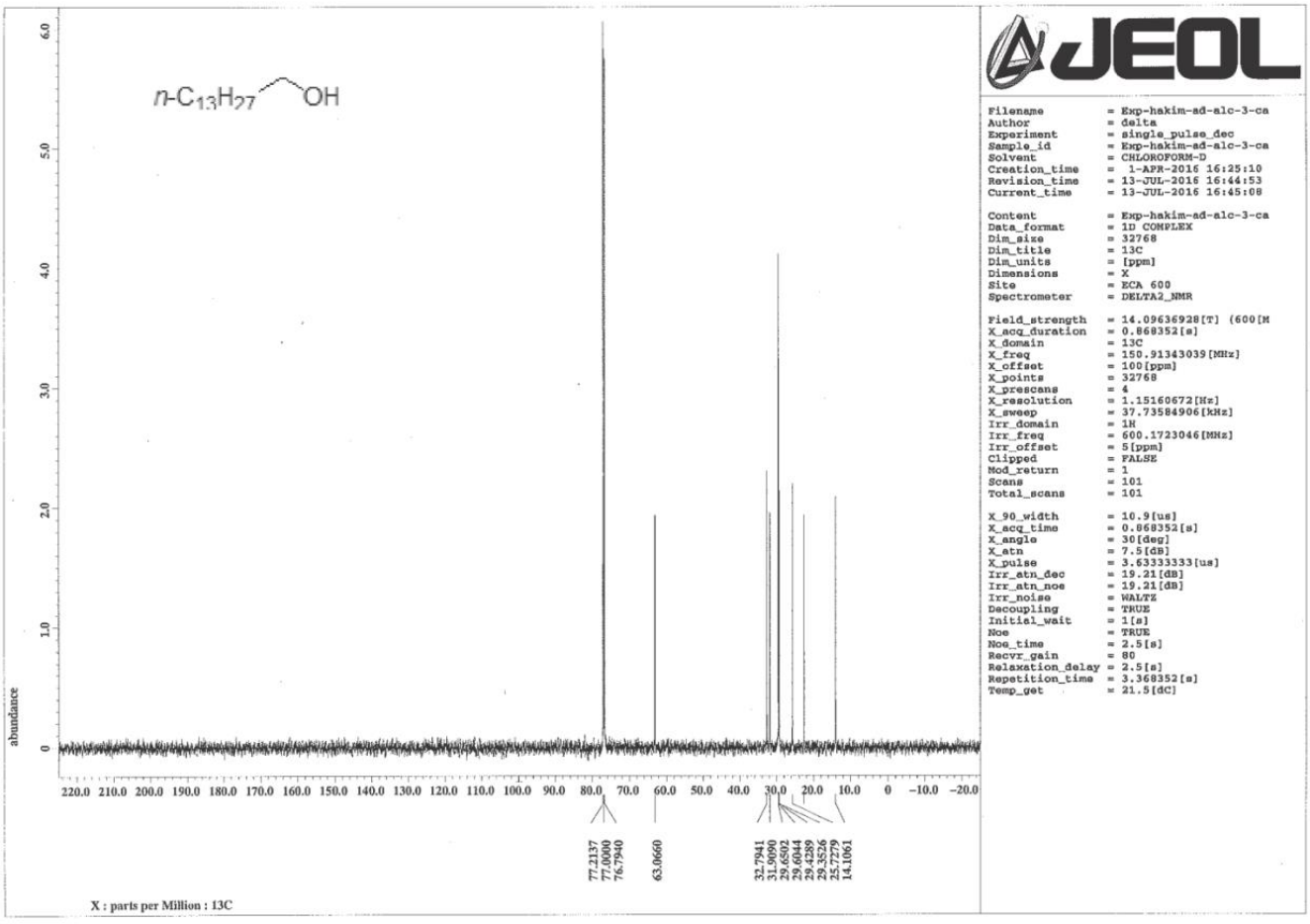



```
Filename          = Exp-hakim-ad-alc-3-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-3-pr
Solvent           = CHLOROFORM-D
Creation_time     =  1-APR-2016 16:18:38
Revision_time     = 13-JUL-2016 16:47:40
Current_time      = 13-JUL-2016 16:47:43

Content           = Exp-hakim-ad-alc-3-pr
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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.7[dC]
```

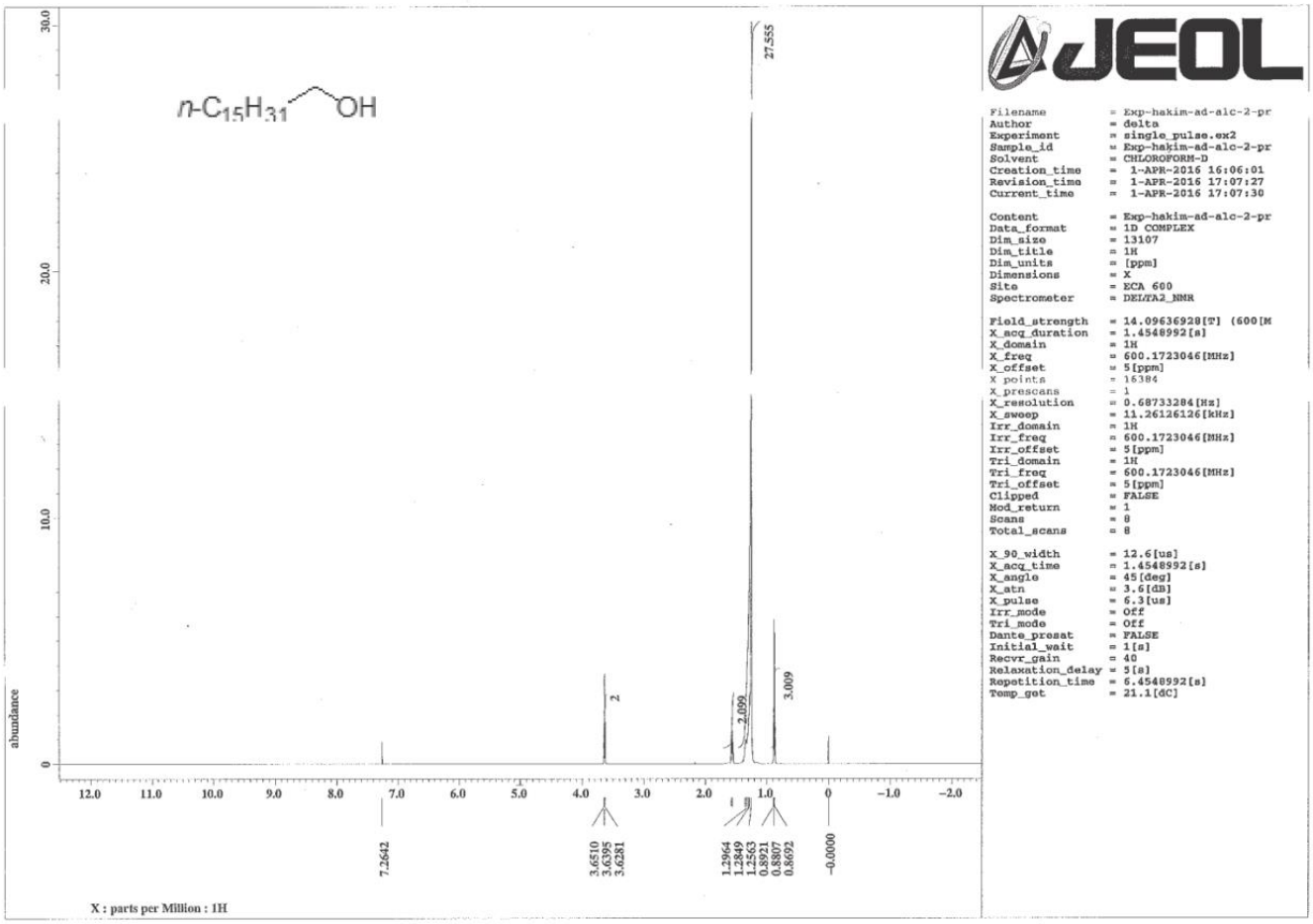



```
Filename          = Exp-hakim-ad-alc-3-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-3-ca
Solvent           = CHLOROFORM-D
Creation_time     =  1-APR-2016 16:25:10
Revision_time     = 13-JUL-2016 16:44:53
Current_time      = 13-JUL-2016 16:45:08

Content           = Exp-hakim-ad-alc-3-ca
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.868352[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.15160672[Hz]
X_sweep           = 37.73584906[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 101
Total_scans       = 101

X_90_width        = 10.9[us]
X_acq_time        = 0.868352[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.633333333[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        = 80
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 21.5[dC]
```

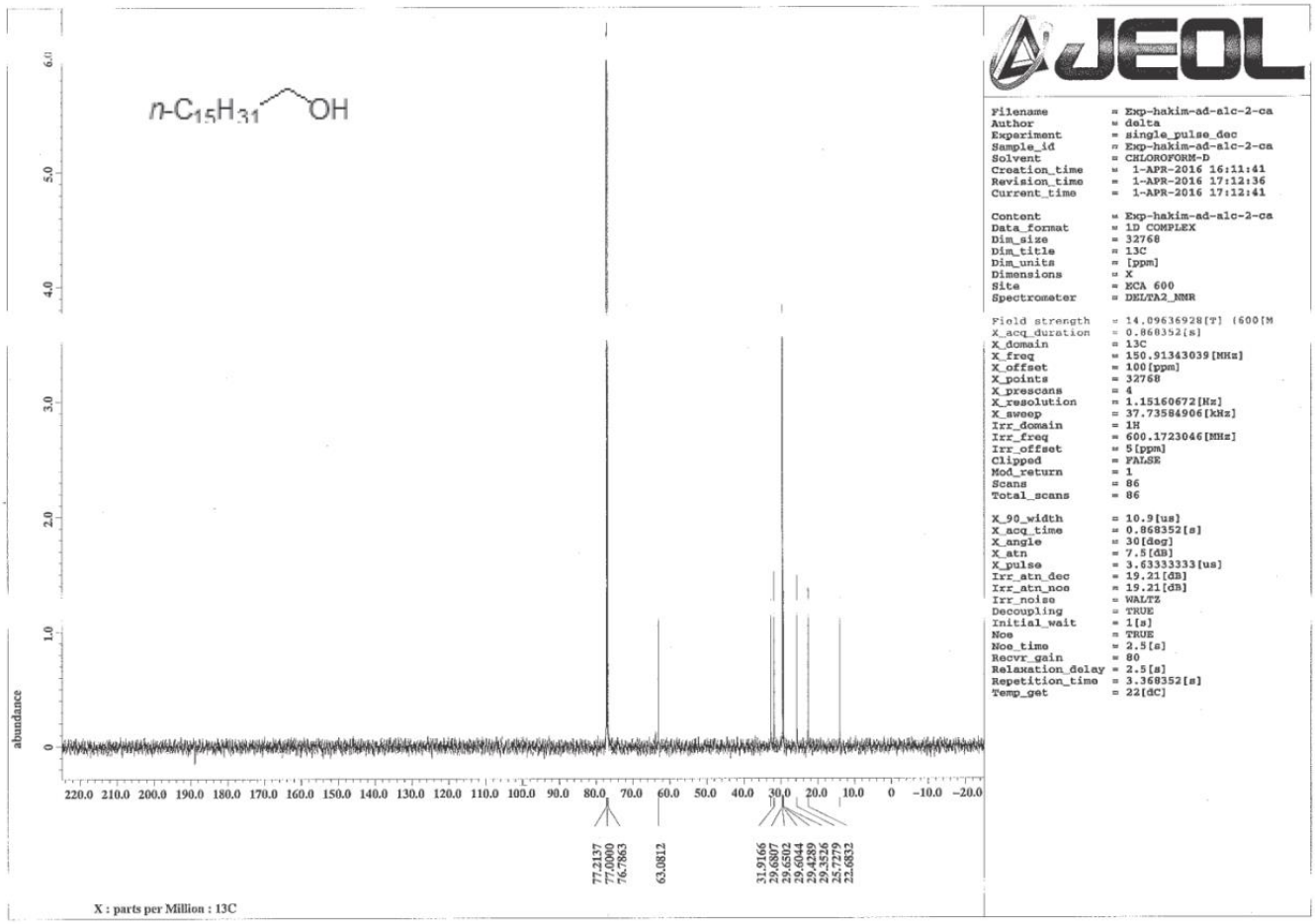



```
Filename        = Exp-hakim-ad-alc-2-pr
Author          = delta
Experiment      = single_pulse.ex2
Sample_id       = Exp-hakim-ad-alc-2-pr
Solvent         = CHLOROFORM-D
Creation_time   =  1-APR-2016 16:06:01
Revision_time   =  1-APR-2016 17:07:27
Current_time    =  1-APR-2016 17:07:30

Content         = Exp-hakim-ad-alc-2-pr
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_presat    = FALSE
Initial_wait    = 1[s]
Recvr_gain      = 40
Relaxation_delay = 5[s]
Repetition_time = 6.4548992[s]
Temp_get        = 21.1[dC]
```

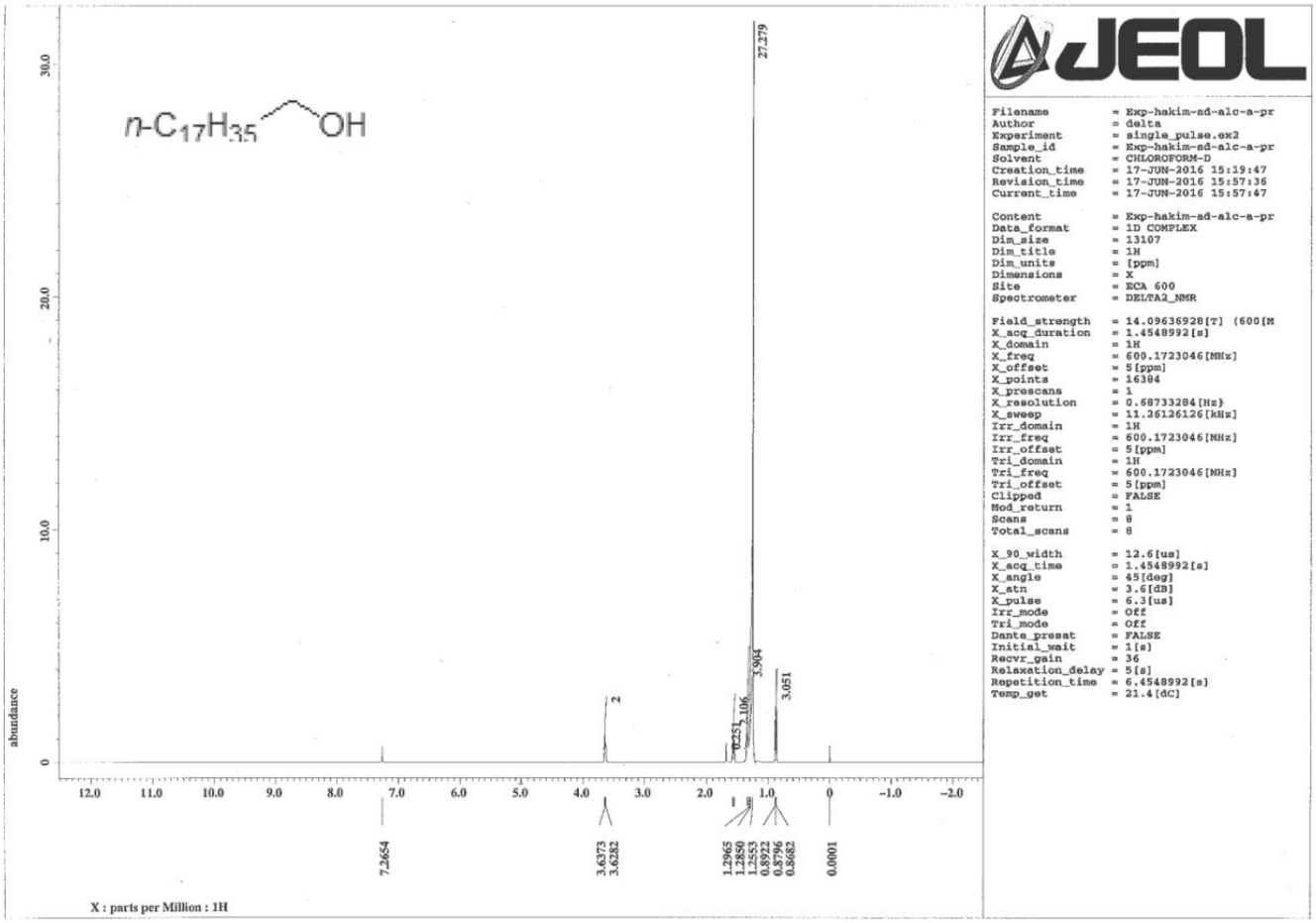



```
Filename        = Exp-hakim-ad-alc-2-ca
Author          = delta
Experiment      = single_pulse_dec
Sample_id       = Exp-hakim-ad-alc-2-ca
Solvent         = CHLOROFORM-D
Creation_time   =  1-APR-2016 16:11:41
Revision_time   =  1-APR-2016 17:12:36
Current_time    =  1-APR-2016 17:12:41

Content         = Exp-hakim-ad-alc-2-ca
Data_format     = 1D COMPLEX
Dim_size        = 32768
Dim_title       = 13C
Dim_units       = [ppm]
Dimensions      = X
Site            = ECA 600
Spectrometer    = DELTA2_NMR

Field_strength  = 14.09636928[T] (600[M
X_acq_duration  = 0.868352[s]
X_domain        = 13C
X_freq          = 150.91343039[MHz]
X_offset        = 100[ppm]
X_points        = 32768
X_prescans      = 4
X_resolution    = 1.15160672[Hz]
X_sweep         = 37.73584906[kHz]
Irr_domain      = 1H
Irr_freq        = 600.1723046[MHz]
Irr_offset      = 5[ppm]
Clipped         = FALSE
Mod_return      = 1
Scans           = 86
Total_scans     = 86

X_90_width      = 10.9[us]
X_acq_time      = 0.868352[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      = 80
Relaxation_delay = 2.5[s]
Repetition_time = 3.360352[s]
Temp_get        = 22[dC]
```



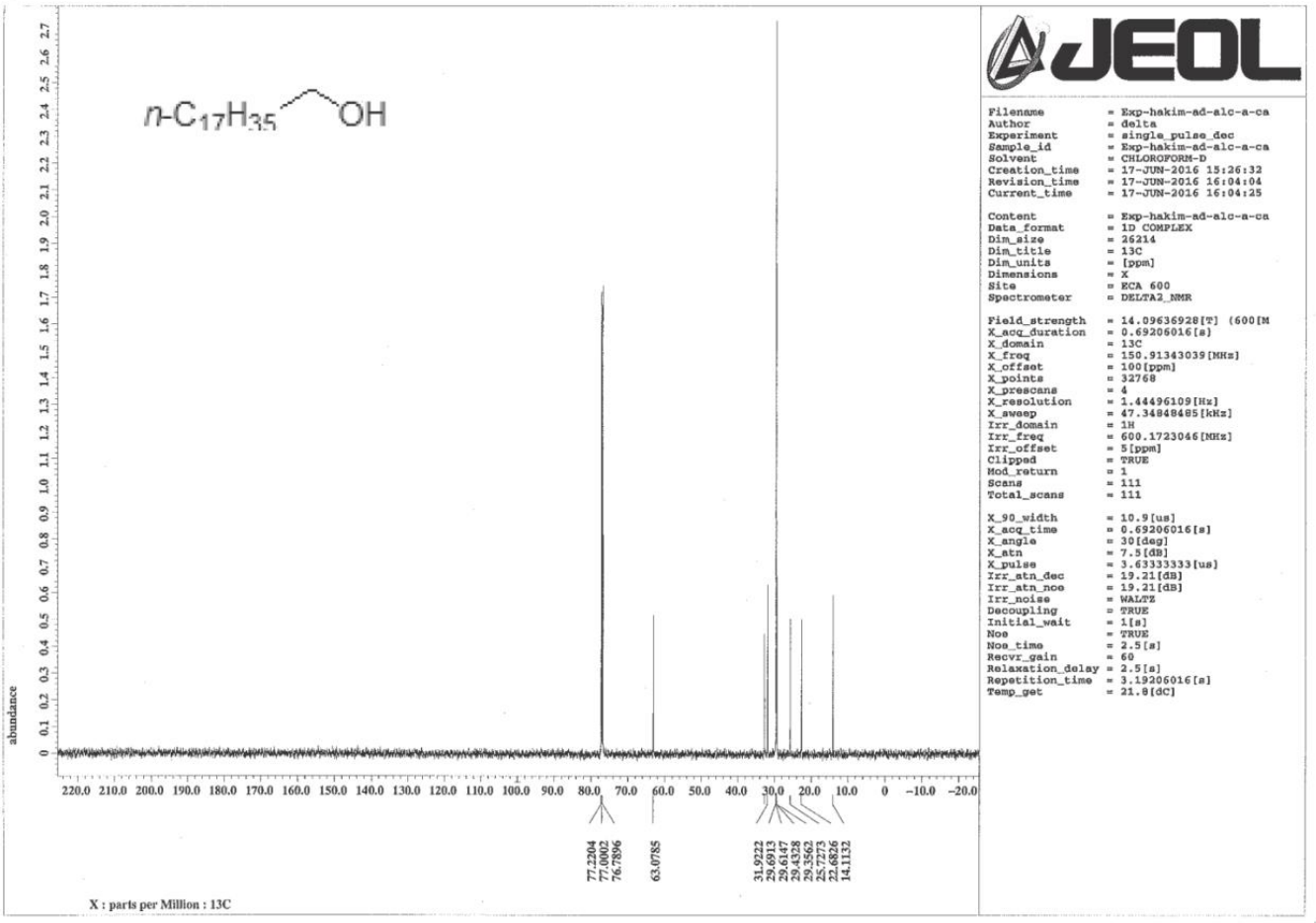
JEOL

```
Filename          = Exp-hakim-ad-alc-a-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-ad-alc-a-pr
Solvent           = CHLOROFORM-D
Creation_time     = 17-JUN-2016 15:19:47
Revision_time     = 17-JUN-2016 15:57:36
Current_time      = 17-JUN-2016 15:57:47

Content           = Exp-hakim-ad-alc-a-pr
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_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 21.4[dC]
```



JEOL

```
Filename          = Exp-hakim-ad-alc-a-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-ad-alc-a-ca
Solvent           = CHLOROFORM-D
Creation_time     = 17-JUN-2016 15:26:32
Revision_time     = 17-JUN-2016 16:04:04
Current_time      = 17-JUN-2016 16:04:25

Content           = Exp-hakim-ad-alc-a-ca
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             = 111
Total_scans       = 111

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          = 21.9[dC]
```

4. References

S1) M. D. Segall, P. J. D. Lindan, M. J. Probert, C. J. Pickard, P. J. Hasnip, S. J. Clark, M. C. Payne, *J. Phys.: Condens. Matter* **2002**, *14*, 2717–2744.

S2) S. J. J. Clark, M. D. Segall, C. J. J. Pickard, P. J. J. Hasnip, M. I. J. Probert, K. Refson, M. C. Payne, *Zeitschrift für Krist.* **2005**, *220*, 567–570.

S3) J. P. Perdew, K. Burke, M. Ernzerhof, *Phys. Rev. Lett.* **1996**, 77, 3865−3868.

S4) H. J. Monkhorst, J. D. Pack, *Phys. Rev. B* **1976**, *13*, 5188−5192.

S5) M. Szastak, M. Spain, D. J. Procter, *Chem. A. Eur. J.* **2014**, 15, 4222–4226.

S6) D. B. Konopacki, K. C. Shortsleeves, M. M. Turnbull, J. L. Wikaira, A. D. Hobson, *Eur. J. Org. Chem*. **2015**, *24*, 5453–5463.

S7) K. B. Ling, A. D. Smith, *Chem. Commun.* **2011**, *47*, 373–375.

S8) M. Sutter, R. Lafon, Y. Raoul, E. Metay, M. Lemaire, *Eur. J. Org. Chem.* **2013**, *26*, 5902–5916.

S9) S. Demay, F. Volant, P. Knochel, *Angew. Chem. Int. Ed.* **2001**, *40*, 1235–1238.

S10) S. Bruckner, U. Bilitewski, R. Schobert, *Org. Lett.* **2016**, *18*, 1136–1139.

S11) J. hang, Y. Li, F. Zhang, Y. Chen, *Angew. Chem. Int. Ed.* **2016**, *128*, 1904–1907.

S12) Y. J. Zhang, W. Dayoub, G. R. Chen, M. Lemaire, *Tetrahedron*, **2012**, *68*, 7400–7407.

S13) G. Bastug, S. Dierick, F. Lebreux, I.E. Markó, *Org. Lett.* **2012**, *3*, 341–344.

S14) R. Fang, H. Liu, R. Luque, Y. Li, *Green Chem*. **2015**, *17*, 4183–4188.

S15) A. R. Morrin, C. Martin, *J. Chem. Soc., Perkin Trans.1* **1980**, 1185–1193.

S16) V. H. Jadhav, J. G. Kim, H. J. Jeong, D. W. Kim, *J. Org. Chem.* **2015**, *80*, 7275–7280.

S17) T. Mizugaki, Y. Nagatsu, K. Togo, Z. Maeno, T. Mitsudome, K. Jitsukawa, K. Kaneda, *Green Chem.*, **2015**, *17*, 5136–5139.

S18) G. A. Filonenko, M. J. B. Aguila, E. N. Schulpen, R. V. Putten, J. Wiecko, C. Müller, L. Lefort, E. J. M. Hensen, E. A. Pidko, *J. Am. Chem. Soc.* **2015**, *137*, 7620–7623.

S19) X. Cui, Y. Li, C. Topf, K. Junge, M. Beller, *Angew. Chem. Int. Ed.* **2015**, *54*, 10596–10599.

S20) J. H. Babler, D. W. Ziemke, R. M. Hamer, *Tetrahedron Lett*. **2013**, *54*, 1754–1757.

S21) K. Tanemura, T. Suzuki, *Tetrahedron Lett*. **2013**, *54*, 6740–6743.

S22) X. Tan, Y. Wang, Y. Liu, F. Wang, L. Shi, K. H. Lee, Z. Lin, H. Lv, X. Zhang, *Org. Lett.* **2015**, *17*, 454–457.

S23) M. C. Pischl, C. F. Weise, S. Haseloff, M. A. Müller, A. Pfaltz, C. Schneider, C*hem. A. Eur. J.* **2014**, *20*, 17360–17374.