

N-Fluorobenzenaminium tetrafluoroborate generated in situ by aniline and Selectfluor as a reusable catalyst for ring opening of epoxides with amines under microwave irradiations

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Methods and materials

Aniline and substituted aniline, morpholine and piperidine were purchased from S.D. Fine Chemicals, India. Styrene oxide, 1-hexene oxide, 1-decene oxide, 1-octene oxide, cyclohexene oxide, cyclopentene oxide and epichlorohydrin were used as received from Aldrich, USA. Proton and carbon nuclear magnetic resonance spectra (^1H and ^{13}C NMR, respectively) were recorded on 400 MHz (operating frequencies: ^1H , 400.13 MHz; ^{13}C , 100.61 MHz) FT spectrometers at ambient temperature. In the case of ^1H and ^{13}C NMR spectra, the chemical shifts (δ) for all compounds are listed in parts per million downfield from tetramethylsilane using the NMR solvent as an internal reference. The reference values used for deuterated chloroform (CDCl_3) were 7.26 and 77.00 ppm for ^1H and ^{13}C NMR spectra, respectively. Infrared spectra were recorded on a Perkin Elmer Spectrum BX-2 spectrophotometer. Optical rotation values were measured on a Rudolph Autopol (IV) Research Analytical. Single crystal X-ray diffraction was recorded on Oxford diffraction X-CaliburTM S. Thin layer chromatography was carried out using Merck Kieselgel 60 F254 silica gel plates. Column chromatography separations were performed using Merck Kieselgel 60 (Art. 7734).

Crystal Structure Analysis of (S)-1-(Naphthalen-2-yloxy)-3-piperidin-1-yl-propan-2-ol hydrochloride salt (21.HCl):

$\text{C}_{18}\text{H}_{24}\text{NO}_2\text{Cl}$, Mr = 321.83, monoclinic, space group: $P2_1$, a = 8.117(5), b = 7.669(5), c = 28.305(5) Å, β = 95.859(5)°, V = 1755.6(16) Å³, $\rho_{\text{calcd.}}$ = 1.218 g·cm⁻³, Z = 4, F(000) = 688, crystal dimensions: 0.46×0.18×0.09 mm. The refinement converged at R_1 = 0.0619, wR_2 = 0.1098 for all data; final GOF: 1.003; largest peak/hole in the final difference Fourier map: 0.27/−0.16 e·Å⁻³ and absolute structure parameter -0.07(6). Further details on the crystal structure data can be obtained from the Cambridge Crystallographic Data Centre via www.ccdc.chem.ac.uk/data_request/cif, on quoting the depository numbers CCDC. 895046

A total of 14095 reflections was collected at 273(2) K on an Oxford X calibur Ruby CCD diffractometer (MoK α radiation, λ = 0.71073 Å). Routine Lorentz and polarization corrections were applied, and an absorption correction was performed using the ABSCALE 3 program.¹ Direct methods were used to locate the heavy metal atoms (SHELXS-86). The remaining atoms were located from successive Fourier maps

(SHELXL-97).²The program PLATON was used to check the space group which falls in hexagonal system having chiral space group P 1 21 1 (Flack parameter -0.07(6)).³

General procedure for the ring opening of epoxides with amines catalysed by Selectflour

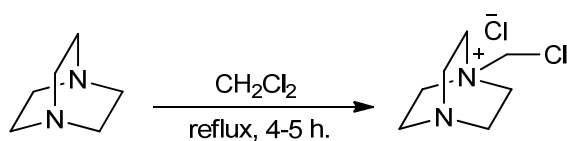
A reaction mixture of epoxide (2 mmol), amine (2 mmol) and Selectflour (2 mol %) was irradiated under microwave for 10 minute at 70 °C temperature and 200W power. The crude product was purified over silica gel by column chromatography to provide pure β -amino alcohol.

Synthesis of 2-(naphthalene-2-yloxy methyl) oxirane

2-naphthol (10 mmol, 1.44 gm) was stirred with KOH (10 mmol, 0.56gm) and methanol (11.33 ml) in a 250ml round bottom flask till the reaction mixture was completely dissolved. The solvent was evaporate by rota-vapour and we obtained dry potassium salt of 2-naphthol. The reaction flask was kept in an oil bath preheated 60 to 90 °C temperature then epichlorohydrin (100mmol, 7.83mL) was added both solvent as reagent. The reaction mixture further stirred at 60 °C for 30 minutes and cooled to room temperature. The brine solution was added to the reaction mixture. Product was extracted from the aq. solution using a sufficient amount of dichloromethane. The organic layer was dried over sodium sulphate and solvent was removed under reduce pressure. The product was purified by silica gel column chromatography using 90:10 (hexane: EtOAc) afforded 92% yield as viscous liquid.

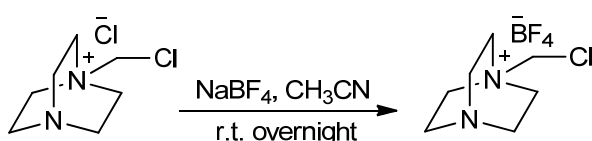
The enantioenriched 2-(naphthalene-2-yloxymethyl) oxirane was obtained using Jacobsen hydrolytic kinetic resolution (HKR) with (*R, R*)-Jacobsen Cobalt (III)OAc and water as a resolving reagent.⁴

Synthesis of 1-(chloromethyl)-1,4-diazabicyclo[2.2.2]octan-1-ium chloride¹⁶



TEDA (2 g, 17.8 mmol) was dissolved in Dry DCM (5.6 mL, 89.03 mmol) and the mixture was allowed to reflux under inert atmosphere for 4 h. The dense white precipitate was formed and filtered under nitrogen atmosphere and washed with dry DCM (9 mL). The crude product was dried under vacuum to obtained 1-(chloromethyl)-1,4-diazabicyclo[2.2.2]octan-1-ium chloride (3.1 g., 90%).

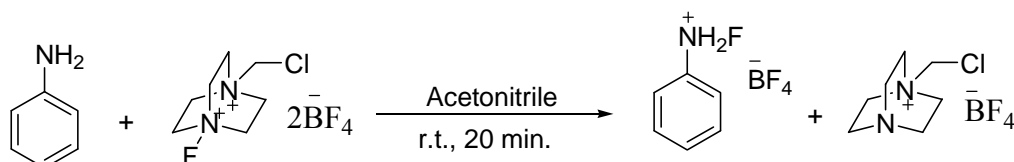
Chloride anion exchange of 1-(chloromethyl)-1,4-diazabicyclo[2.2.2]octan-1-ium chloride with tetrafluoroborate¹⁶



NaBF₄ (1.68 g., 15.5 mmol) in dry acetonitrile (10 mL) was added to a stirred slurry of 1-(chloromethyl)-1,4-diazabicyclo[2.2.2]octan-1-ium chloride (3 g., 15.5 mmol) in acetonitrile (20 mL). The mixture was stirred under inert atmosphere at room temperature for overnight. Precipitate of sodium chloride was formed. It was removed by filtration and washed with dry acetonitrile. The filtrate and washing were combined and evaporate under reduced pressure, leaving a white solid residue which was recrystallized from an ethanol/ethyl.

¹H NMR (D₂O, 400MHz): δ 4.97 (s, 2H), 3.41 (t, *J* = 6.59 Hz, 6H), 3.12 (t, *J* = 6.59 Hz, 6H) ppm. ¹⁹F NMR (D₂O, 376MHz): δ -148.14 ppm.

Preparation of N-fluorobenzenaminium tetrafluoroborate



Aniline (1 mmol, 91 μL) was taken in acetonitrile (1 mL) and Selectfluor (1 mmol, 354 mg, in acetonitrile (1.3 mL)) was slowly added and stirred for 20 min. Acetonitrile was removed under reduced pressure and the resulting white solid was dissolved in the minimum amount of acetone and a solution of 10 mL solution of H₂SO₄ (0.8 mmol, 42 μL) was added drop wise and precipitation of 1-chloromethyl-4-hydro-1,4-diazoniabicyclo [2.2.2] octane hydrogen sulphate tetrafluoroborate was removed by filtration. The filtrate was evaporated under vacuum to give N-fluorobenzenaminium tetrafluoroborate (107 mg, 96%).

¹H NMR (D₂O, 400MHz): δ 7.33-7.28 (m, 3H), 7.19-7.17 (m, 2H). ¹⁹F NMR (D₂O, 376MHz): δ -129.6, -150.6 ppm.

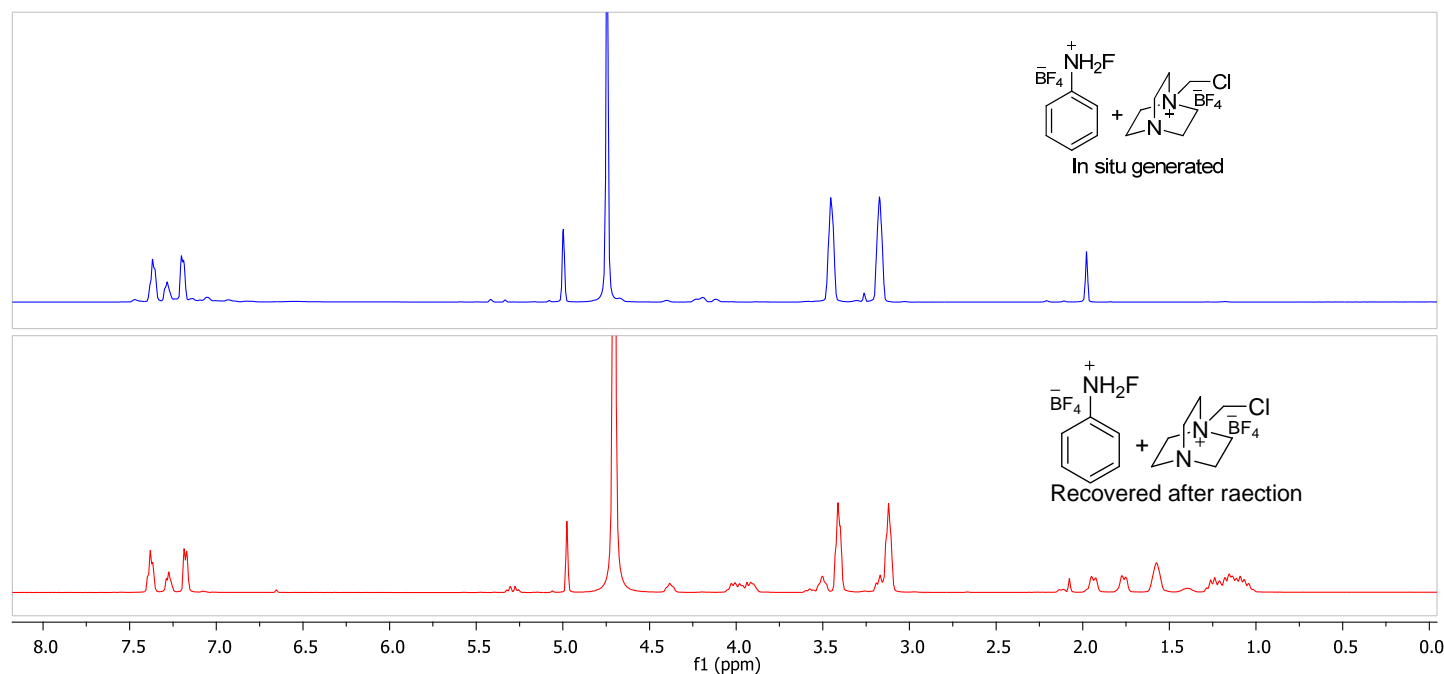


Figure 1. ¹H NMR of fresh in situ generated **1** and recovered catalyst in D₂O.

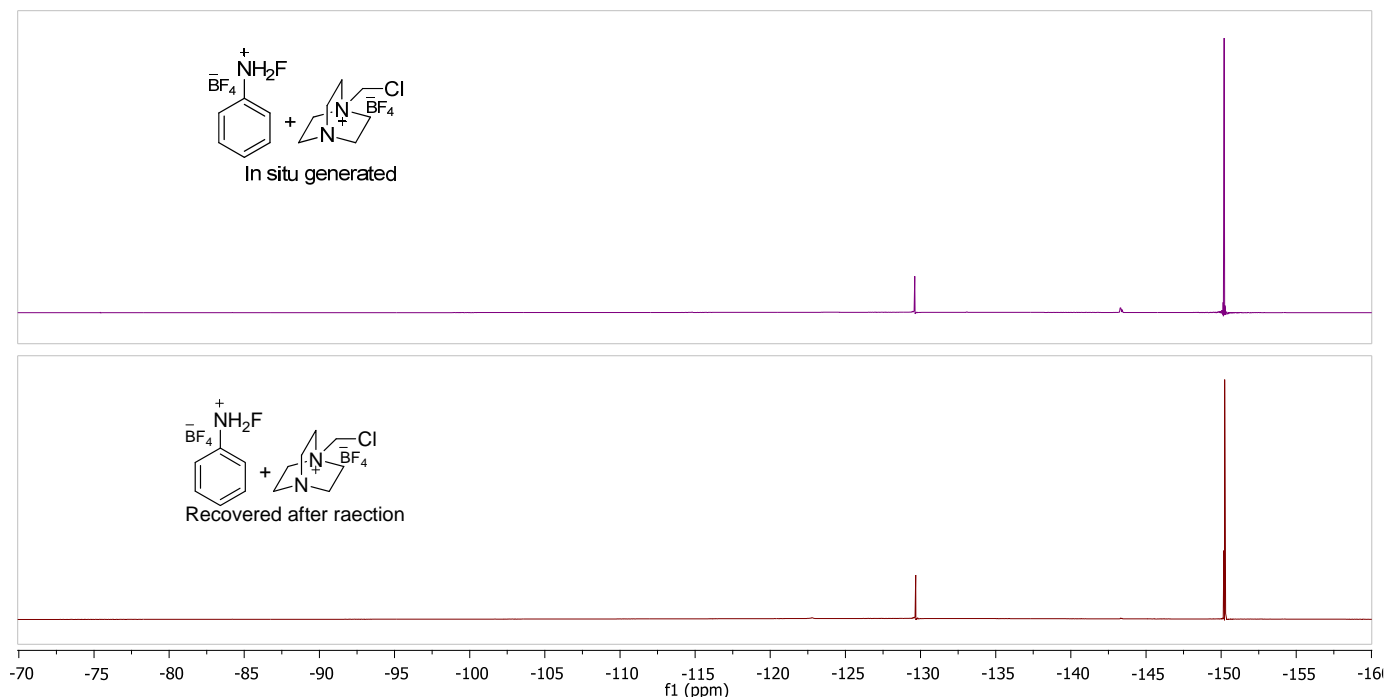
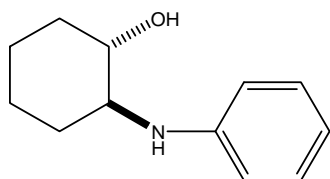


Figure 2. ^{19}F NMR of in situ generate catalyst **1** and recovered catalyst in D_2O .

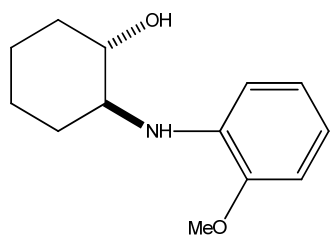
Spectroscopic data for β -aminoalcohols

Trans-2-anilino-1-cyclohexanol (**6a**)^{5-9,11-12}:



^1H NMR (CDCl_3 , 400MHz): δ 7.21 (m, 2H), 6.73-6.80 (m, 3H), 3.37 (ddd, $J = 10.3, 8.8, 4.4$ Hz, 1H), 3.16 (ddd, $J = 11.7, 9.5, 4.4$ Hz, 1H), 3.06 (brs, 2H), 2.15-2.12 (m, 2H), 1.80-1.74 (m, 2H), 1.36-1.30 (m, 3H), 1.11-1.01 (m, 1H) ppm; ^{13}C NMR (CDCl_3 , 100MHz) δ 147.5, 129.0 (2C), 117.9, 114.1 (2C), 73.9, 59.7, 33.0, 31.2, 24.6, 24.0 ppm; IR (liquid in CH_2Cl_2): 3390, 2932, 1602, 1503, 1256, 1066, 748 cm^{-1} .

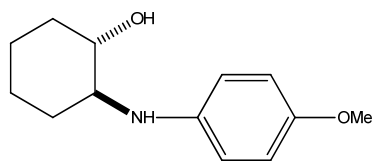
Trans-2-(*o*-methoxyphenylamino)cyclohexanol (**6b**)^{6,9,11}:



^1H NMR (CDCl_3 , 400MHz) δ 6.88-6.84 (m, 1H), 6.79-6.77 (m, 2H), 6.72-6.68 (m, 1H), 3.83 (3H, s), 3.41 (ddd, $J = 10.9, 9.5, 4.4$ Hz, 1H), 3.14 (ddd, $J = 10.9, 8.8, 3.7$ Hz, 1H), 2.10 (t, $J = 13.9$ Hz, 2H), 1.69-1.78 (m, 2H), 1.28-1.36 (m, 3H), 1.11-1.02 (m, 1H) ppm; ^{13}C NMR (CDCl_3 , 100MHz) δ 147.2, 137.4, 121.1,

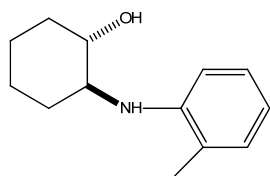
117.0, 111.2, 109.5, 74.3, 59.3, 55.2, 32.9, 31.3, 24.9, 24.1 ppm; IR (liquid in CH₂Cl₂) : 3407, 2931, 1601, 1512, 1222, 1068, 1029, 735 cm⁻¹

***Trans*-2-(*p*-methoxyphenylamino)cyclohexanol (6c)^{5-7,11}:**



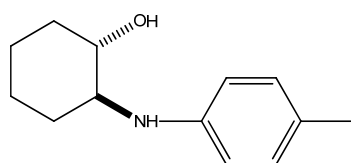
¹H NMR (CDCl₃, 400MHz) δ 6.75 (d, *J* = 9.5 Hz, 2H), 6.65 (d, *J* = 9.5 Hz, 2H), 3.72 (s, 3H), 3.29 (ddd, *J* = 10.9, 10.3, 4.4 Hz, 1H), 2.96 (ddd, *J* = 11.0, 9.5, 4.4 Hz, 1H), 2.07-2.05 (m, 2H), 1.73-1.65 (m, 2H), 1.39-1.20 (m, 3H), 1.01-0.93 (m, 1H) ppm; ¹³C NMR (CDCl₃, 100MHz) δ 152.6, 141.5, 116.3 (2C), 114.6 (2C), 74.0, 61.3, 55.5, 33.0, 31.3, 24.8, 24.1 ppm; IR (liquid in CH₂Cl₂) : 3369, 2931, 1512, 1239, 1065, 1038, 821 cm⁻¹.

***Trans*-2-(*o*-tolylamino)cyclohexanol (6d)^{5,11}:**



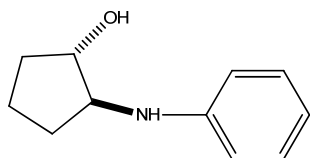
¹H NMR (CDCl₃, 400MHz) δ 7.12-7.07 (m, 2H), 6.79 (d, *J* = 8.1 Hz, 1H), 6.70 (t, *J* = 7.3, 1H), 3.42 (ddd, *J* = 10.9, 10.3, 4.4 Hz, 1H), 3.20 (ddd, *J* = 11.7, 9.5, 4.4 Hz, 1H), 2.15 (s, 3H), 2.15-2.13 (m, 1H), 1.79-1.72 (m, 3H), 1.38-1.30 (m, 3H), 1.1-1.0 (m, 1H) ppm; ¹³C NMR (CDCl₃, 100MHz) δ 145.6, 130.3, 127.0, 122.9, 117.7, 111.4, 74.4, 59.7, 33.1, 31.7, 24.9, 24.2, 17.6 ppm; IR (liquid in CH₃CN) : 3405, 2931, 1605, 1511, 1378, 1258, 1066, 746 cm⁻¹

***Trans*-2-(*p*-tolylamino)cyclohexanol (6e)^{6,9}:**



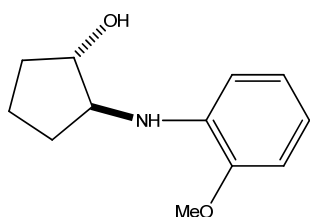
¹H NMR (CDCl₃, 400MHz) δ 7.0 (d, *J* = 8.8 Hz, 2H), 6.64 (d, *J* = 8.8 Hz, 2H), 3.31 (ddd, *J* = 9.9, 8.8, 4.4 Hz, 1H), 3.14 (brs, 2H), 3.08 (ddd, *J* = 11.7, 9.5, 4.4 Hz, 1H), 2.26(s, 3H), 2.13-2.05 (m, 2H), 1.77-1.69 (m, 2H), 1.35-1.27 (m, 3H), 1.05-0.97 (m, 1H) ppm; ¹³C NMR (CDCl₃, 100MHz) δ 145.3, 129.6 (2C), 127.5, 114.6 (2C), 74.2, 60.4, 33.0, 31.3, 24.9, 24.1, 20.2 ppm; IR (liquid in CH₂Cl₂) : 3388, 2930, 1617, 1518, 1375, 1252, 1066, 808 cm⁻¹.

***Trans*-2-(phenylamino)cyclopentanol (7a)⁵⁻⁹:**



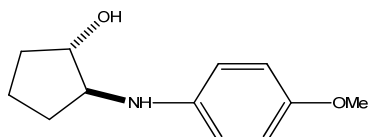
^1H NMR (CDCl_3 , 400MHz) δ 7.17 (t, $J = 7.3$ Hz, 2H), 6.71 (t, $J = 7.3$ Hz, 1H), 6.65 (d, $J = 8.1$ Hz, 2H), 4.02 (dd, $J = 10.3, 4.4$ Hz, 1H), 3.58 (dd, $J = 6.6, 4.4$ Hz, 1H), 2.75 (brs, 2H), 2.29-2.21 (m, 1H), 1.98-1.91 (m, 1H), 1.82-1.68 (m, 2H), 1.66-1.57 (m, 1H), 1.42-1.33 (m, 1H) ppm; ^{13}C NMR (CDCl_3 , 100MHz) δ 147.6, 129.2 (2C), 117.4 (2C), 113.3, 78.0, 61.9, 32.6, 31.0, 20.8 ppm; IR (liquid in CH_2Cl_2) : 3396, 2917, 1602, 1503, 1277, 1044, 749 cm^{-1}

***Trans*-2-(*o*-methoxyphenylamino)cyclopentanol (7b)⁶:**



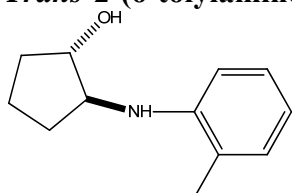
^1H NMR (CDCl_3 , 400MHz) δ 6.86-6.66 (m, 4H), 4.08 (dd, $J = 9.5, 4.4$ Hz, 1H), 3.83 (s, 3H), 3.59 (dd, $J = 6.6, 4.4$ Hz, 1H), 2.29-2.24 (m, 1H), 2.00-1.94 (m, 1H), 1.81-1.74 (m, 2H), 1.65-1.60 (m, 1H), 1.46-1.41 (m, 1H) ppm; ^{13}C NMR (CDCl_3) δ 146.7, 137.5, 120.9, 116.5, 110.3, 109.3, 78.1, 61.7, 55.3, 32.8, 31.1, 21.0 ppm; IR (liquid in CH_2Cl_2) : 3412, 2959, 1602, 1513, 1222, 1028, 738 cm^{-1}

***Trans*-2-(*p*-methoxyphenylamino)cyclopentanol (7c)⁵:**



^1H NMR (CDCl_3) δ 6.75 (d, $J = 9.5$, 2H), 6.64-6.61 (d, $J = 9.5$, 2H), 4.01 (dd, $J = 10.9, 5.1$ Hz, 1H), 3.73 (s, 3H), 3.51 (dd, $J = 7.3, 5.1$ Hz, 1H), 2.75 (brs, 2H), 2.25-2.19 (m, 1H), 1.98-1.93 (m, 1H), 1.80-1.69 (m, 2H), 1.63-1.58 (m, 1H), 1.38-1.33 (m, 1H) ppm; ^{13}C NMR (CDCl_3) δ 152.2, 141.7, 116.4(2C), 114.7(2C), 78.1, 62.9, 55.7, 32.7, 31.0, 20.8 ppm; IR (liquid in CH_2Cl_2): 3378, 2955, 1512, 1238, 1099, 1037, 821 cm^{-1}

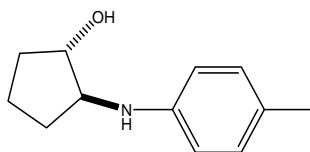
***Trans*-2-(*o*-tolylamino)cyclopentanol (7d)^{5,7,8}:**



^1H NMR (CDCl_3 , 400MHz) δ 7.23 (t, $J = 8.1$ Hz, 1H), 7.16 (d, $J = 7.3$ Hz, 1H), 6.84 (d, $J = 8.1$ Hz, 1H), 6.78 (t, $J = 7.3$ Hz, 1H), 4.17 (dd, $J = 10.3, 4.4$ Hz, 1H), 3.74 (dd, $J = 6.5, 3.7$ Hz, 1H), 2.42-2.36 (m, 1H), 2.22 (s, 3H), 2.09-2.05 (m, 1H), 1.94-1.86 (m, 2H), 1.74-1.72 (m, 1H), 1.55-1.49 (m, 1H) ppm; ^{13}C NMR

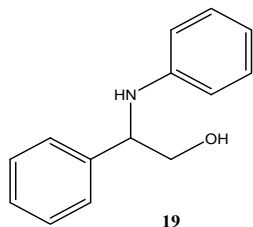
(CDCl₃, 100 MHz) δ 145.5, 130.0, 127.0, 121.9, 117.0, 110.6, 78.0, 61.8, 32.7, 31.3, 21.0, 17.4 ppm; IR (liquid in CH₃CN): 3406, 2960, 1605, 1511, 1378, 1264, 1052, 748 cm⁻¹

Trans-2-(p-tolylamino)cyclopentanol (7e)⁶:



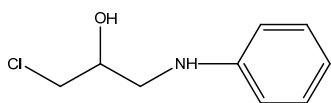
¹H NMR (CDCl₃, 400MHz) δ 7.01 (d, J = 8.1 Hz, 2H), 6.62 (d, J = 8.1 Hz, 2H), 4.02 (dd, J = 10.3, 5.1 Hz, 1H), 3.56 (dd, J = 6.6, 4.4 Hz, 1H), 3.34 (brs, 2H), 2.26 (s, 3H), 2.25-2.22 (m, 1H), 1.97-1.93 (m, 1H), 1.80-1.71 (m, 2H), 1.62-1.61 (m, 1H), 1.41-1.37 (m, 1H) ppm; ¹³C NMR (CDCl₃, 100MHz) δ 144.9, 129.6 (2C), 126.9, 113.8 (2C), 76.6, 62.4, 32.4, 30.7, 20.7, 20.2 ppm; IR (liquid in CH₂Cl₂) : 3369, 2960, 1618, 1519, 1302, 1261, 1041, 808 cm⁻¹

1-phenyl-2-(phenylamino)ethanol (19)^{9,11}:



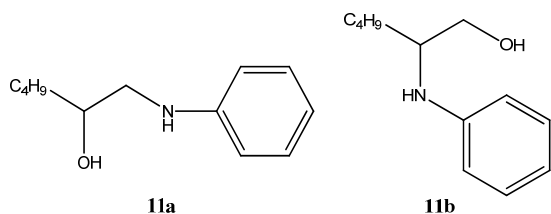
¹H NMR (CDCl₃, 400MHz) δ 7.37-7.25 (m, 5H), 7.12-7.08 (m, 2H), 6.69-6.55 (m, 3H), 4.47 (dd, J = 7.3, 4.4 Hz, 1H), 3.90 (dd, J = 10.9, 3.7 Hz, 1H), 3.71 (dd, J = 10.9, 7.3 Hz, 1H) ppm; ¹³C NMR (CDCl₃, 100MHz) δ 147.1, 140.0, 128.9(2C), 128.5(2C), 127.3 (2C), 126.5 (2C), 117.6, 113.6, 66.9, 59.6 ppm; IR (liquid in CH₂Cl₂) : 3393, 2927, 1602, 1503, 1267, 1066, 751 cm⁻¹

1-chloro-3-(phenylamino)propan-2-ol (17)⁹ :



¹H NMR (CDCl₃, 400MHz) δ 7.16 (t, J = 7.7 Hz, 2H), 6.74 (t, J = 7.3 Hz, 1H), 6.65 (d, J = 8.8 Hz, 2H), 4.07-4.04 (m, 1H), 3.67-3.62 (m, 2H), 3.36 (dd, J = 13.2, 4.4 Hz, 1H), 3.21 (dd, J = 13.2, 8.1 Hz, 1 H) ppm; ¹³C NMR (CDCl₃, 100MHz) δ 147.6, 129.6 (2C), 118.6, 113.2(2C), 69.7, 47.6, 47.0 ppm; IR (liquid in CH₃CN): 3341, 2925, 1601, 1261, 1074, 750, 694 cm⁻¹

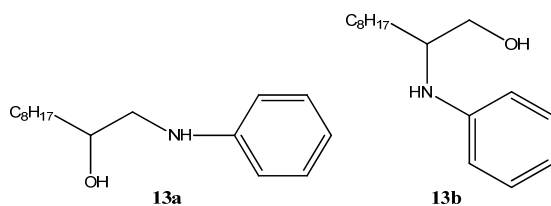
1-(phenylamino)hexan-2-ol (11a) and 2-(phenylamino)hexan-1-ol (11b)¹² :



The compound **11a** was obtained as major regioisomer with ratio of **11a:11b** (77:23), the ratio of regioisomer was determined by ^1H NMR by integrating peaks at δ 3.72 and 3.64 ppm corresponding to compound **11a** and **11b**.

^1H NMR (CDCl_3 , 400MHz) δ 7.21-7.20 (m, 2H), 6.64 (t, $J = 7.3$ Hz, 1H), 6.55 (d, $J = 7.3$ Hz, 2H), 3.72-3.71 (m, 1H), 3.15 (dd, $J = 12.5, 2.9$ Hz, 1H), 2.90 (dd, $J = 13.2, 8.8$ Hz, 1H), 1.43-1.25 (m, 6H), 0.86-0.82 (t, 3H) ppm; ^{13}C NMR (CDCl_3 , 100MHz) (Major) δ 148.2, 129.2 (2C), 117.74, 113.54 (2C), 70.20, 50.16, 34.71, 27.17, 22.66, 13.95 ppm; (Minor) δ 147.73, 129.29(2C), 117.68, 113.58(2C), 64.31, 55.19, 31.79, 28.26, 13.93 ppm; IR (liquid in CH_2Cl_2): 3393, 2930, 1603, 1504, 1257, 1061, 748 cm^{-1}

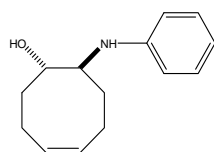
1-(phenylamino)decan-2-ol (**13**):



The compound **13a** was obtained as major regioisomer with ratio of **13a:13b** (79:21), the ratio of regioisomer was determined by ^1H NMR by integrating peaks at δ 3.83 and 3.75 ppm corresponding to compound **13a** and **13b**.

^1H NMR (CDCl_3 , 400MHz) δ 7.21 (t, $J = 7.3$ Hz, 2H), 6.77 (t, $J = 7.3$ Hz, 1H) 6.67 (d, $J = 5.9$ Hz, 2H), 3.81-3.84 (m, 1H), 3.26 (dd, $J = 13.2, 3.7$ Hz, 1H), 2.99 (dd, $J = 13.2, 8.8$ Hz, 1H), 1.33 (brs, 14H), 0.93-0.96 (t, 3H) ppm; ^{13}C NMR (CDCl_3 , 100MHz) (Major) δ 148.24, 129.34(2C), 117.82, 113.27(2C), 70.35, 64.47, 55.36, 50.31, 35.08, 31.84, 29.65, 25.61, 22.64, 14.10 ppm; (Minor) δ 147.74, 129.24(2C), 117.59, 113.55(2C), 64.03, 55.08, 31.97, 30.79, 29.71, 29.41, 29.15, 26.07 ppm; IR (liquid in CH_2Cl_2) : 3393, 2925, 1603, 1505, 1258, 1072, 748 cm^{-1}

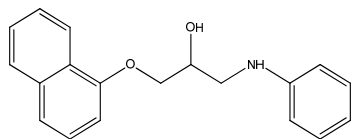
Trans-8-(phenylamino)cyclooct-4-enol (**9**)^{10,11}:



^1H NMR (CDCl_3 , 400MHz) δ 7.14-7.05 (m, 2H), 6.74-6.60 (m, 3H), 5.67-5.63 (m, 1H), 5.52-5.45 (m, 1H), 3.57 (ddd, $J = 11.7, 8.1, 3.7$ Hz, 1H), 3.39 (ddd, $J = 11.7, 8.8, 3.7$ Hz, 1H), 2.40-2.29 (m, 2H), 2.23-2.16 (m, 2H), 2.04-1.91 (m, 2H), 1.69-1.61 (m, 1H), 1.50-1.42 (m, 1H) ppm; ^{13}C NMR (CDCl_3 , 100MHz) δ 147.8,

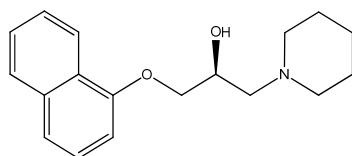
130.4, 129.0 (2C), 127.6, 118.7, 114.7 (2C), 72.7, 58.7, 33.1, 31.8, 23.1, 22.6 ppm; IR (liquid in CH₂Cl₂): 3389, 2930, 1601, 1499, 1252, 1050, 869, 750, 698 cm⁻¹.

1-(naphthalen-2-yloxy)-3-(phenylamino)propan-2-ol (17)^{8,13,14}:



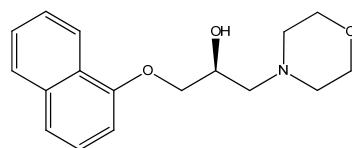
¹H NMR (CDCl₃, 400MHz) δ 7.80-7.72 (m, 3H), 7.48-7.44 (m, 1H), 7.39-7.35 (m, 1H), 7.21-7.19 (m, 4H), 6.77(t, *J* = 7.3, 1H), 6.70 (d, *J* = 7.3 Hz, 2H), 4.35-4.29 (m, 1H), 4.20-4.13 (m, 2H), 3.47 (dd, *J* = 13.2, 4.4 Hz, 1H), 3.34 (dd, *J* = 13.2, 7.3 Hz, 1H); ¹³C NMR (CDCl₃, 100MHz) δ 156.2, 147.9, 134.3, 129.4, 129.2(2C), 129.0, 127.5, 126.7, 126.4, 123.8, 118.4, 117.9, 113.2(2C), 106.8, 70.0, 68.6, 46.5 ppm; IR (liquid in CH₂Cl₂) 3400, 2927, 1629, 1602, 1258, 1034, 750 cm⁻¹

(S)-1-(naphthalen-2-yloxy)-3-(piperidin-1-yl)propan-2-ol (21):



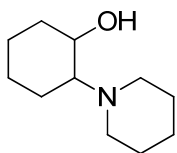
¹H NMR (CDCl₃, 400MHz) δ 7.69-7.63 (m, 3H), 7.37-7.07 (m, 4H), 4.11-4.05 (m, 1H), 4.01-3.97 (m, 2H), 3.22(brs, 1H), 2.60-2.51 (m, 2H), 2.47-2.33 (m, 4H), 1.58-1.50 (m, 4H), 1.40-1.37 (m, 2H) ppm; ¹³C NMR (CDCl₃, 100MHz) : δ 156.7, 134.4, 129.3, 129.0, 127.6, 126.7, 126.3, 123.6, 118.9, 106.6, 70.4, 65.2, 61.2, 54.7 (2C), 25.9 (2C), 24.1 ppm; IR (liquid in CH₂Cl₂) : 3401, 2931, 1629, 1600, 1258, 1218, 1037, 747 cm⁻¹

(S)-1-morpholino-3-(naphthalen-2-yloxy)propan-2-ol (22):



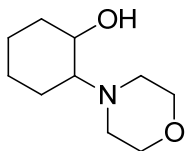
¹H NMR (CDCl₃, 400 MHz) δ 8.17-8.15 (m, 1H), 7.73-7.71 (m, 1H), 7.43-7.18 (m, 4H), 6.74(d, *J* = 7.3 Hz, 1H), 4.29-4.23 (m, 1H), 4.15-4.12 (m, 1H), 4.07-4.03 (m, 1H), 3.75-3.66 (m, 4H), 2.73-2.65 (m, 4H), 2.56-2.54 (m, 2H), ppm; ¹³C NMR (CDCl₃, 100MHz) δ 154.2, 134.4, 127.5, 126.4, 125.8, 125.5, 125.2, 121.8, 120.6, 104.8, 70.3, 66.6(2C), 65.6, 61.4, 53.7 (2C) ppm; IR (liquid in CH₂Cl₂): 3419, 2922, 1628, 1518, 1270, 1241, 1117, 1035, 1070, 772 cm⁻¹

2-(piperidin-1-yl)cyclohexanol :⁹



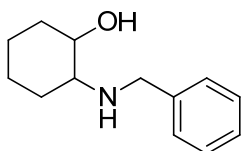
^1H NMR (400 MHz, CDCl_3) δ 3.34-3.28 (m, 1H), 2.65-2.59 (m, 2H), 2.27 (brs, 2H), 2.12-2.04 (m, 2H), 1.75-1.70 (m, 2H), 1.66-1.64 (m, 1H), 1.60-1.44 (m, 4H), 1.40-1.34 (m, 2H), 1.24-1.04 (m, 6H) ppm. ^{13}C NMR (100 MHz, CDCl_3) δ 70.94, 68.49, 49.73 (2C), 33.20, 26.66 (2C), 25.58, 24.79, 24.06, 22.09 ppm.

2-morpholinocyclohexanol :⁹



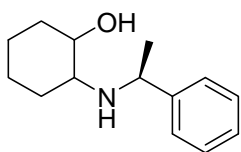
^1H NMR (400 MHz, CDCl_3) δ 4.59 (brs, 1H), 3.71-3.67 (m, 4H), 3.38-3.32 (m, 1H), 2.72-2.69 (m, 2H), 2.44-2.43 (m, 2H), 2.19-2.18 (m, 1H), 2.09-2.06 (m, 1H), 1.80-1.73 (m, 2H), 1.67-1.65 (m, 1H), 1.16-1.11 (m, 4H) ppm. ^{13}C NMR (100 MHz, CDCl_3) δ 70.46, 68.34, 67.37 (2C), 48.65 (2C), 33.11, 25.36, 23.94, 22.19 ppm.

2-(benzylamino)cyclohexanol :⁹



^1H NMR (400 MHz, CDCl_3) δ 7.33-7.22 (m, 5H), 3.93 (d, $J = 13.18$ Hz, 1H), 3.67 (d, $J = 13.18$ Hz, 1H), 3.22-3.18 (m, 2H), 2.34-2.28 (m, 1H), 2.13-2.09 (m, 1H), 1.95-1.93 (m, 1H), 1.70-1.67 (m, 2H), 1.25-1.16 (m, 3H) ppm. ^{13}C NMR (100 MHz, CDCl_3) 139.48, 128.32 (2C), 128.12 (2C), 127, 73.1, 62.8, 50.45, 33.43, 29.85, 24.75, 24.18 ppm.

2-((1-phenylethyl)amino)cyclohexanol :¹⁵



^1H NMR (400 MHz, CDCl_3) δ 7.29-7.16 (m, 5H), 3.89-3.84 (q, $J = 6.59$ Hz, 1H), 3.07-3.02 (m, 1H), 2.30-2.24 (m, 1H), 2.0-1.95 (m, 1H), 1.87-1.83 (m, 1H), 1.63-1.56 (m, 2H), 1.28 (d, 3H), 1.22-1.08 (m, 4H) ppm. ^{13}C NMR (100 MHz, CDCl_3) 146.68, 128.44 (2C), 127.02, 126.38 (2C), 74.02, 61.51, 55.21, 32.94, 31.25, 25.35, 24.20, 23.42 ppm.

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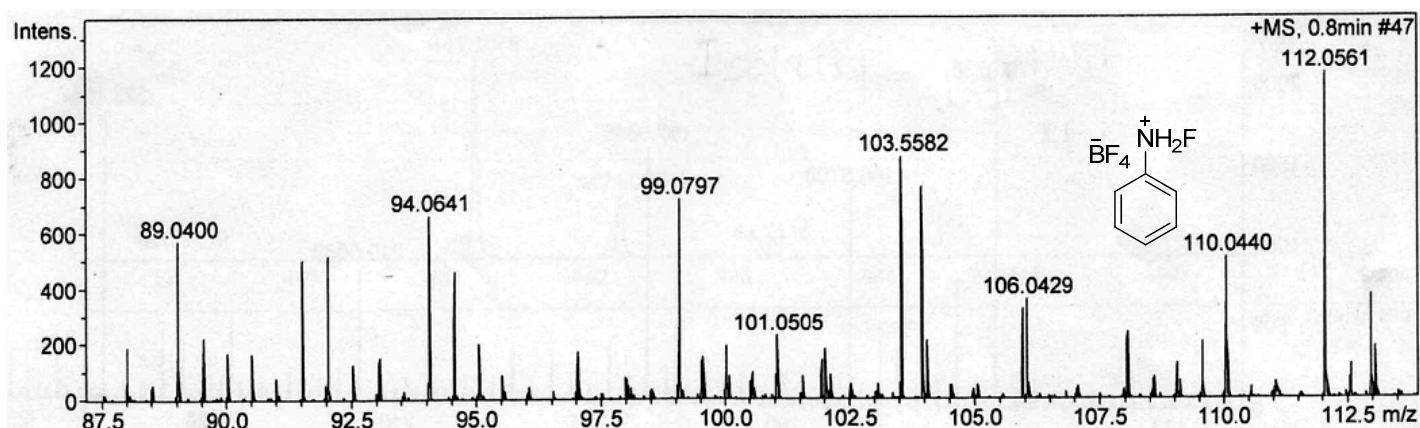


Figure 3. HRMS of N-Fluorobenzenaminium tetrafluoroborate

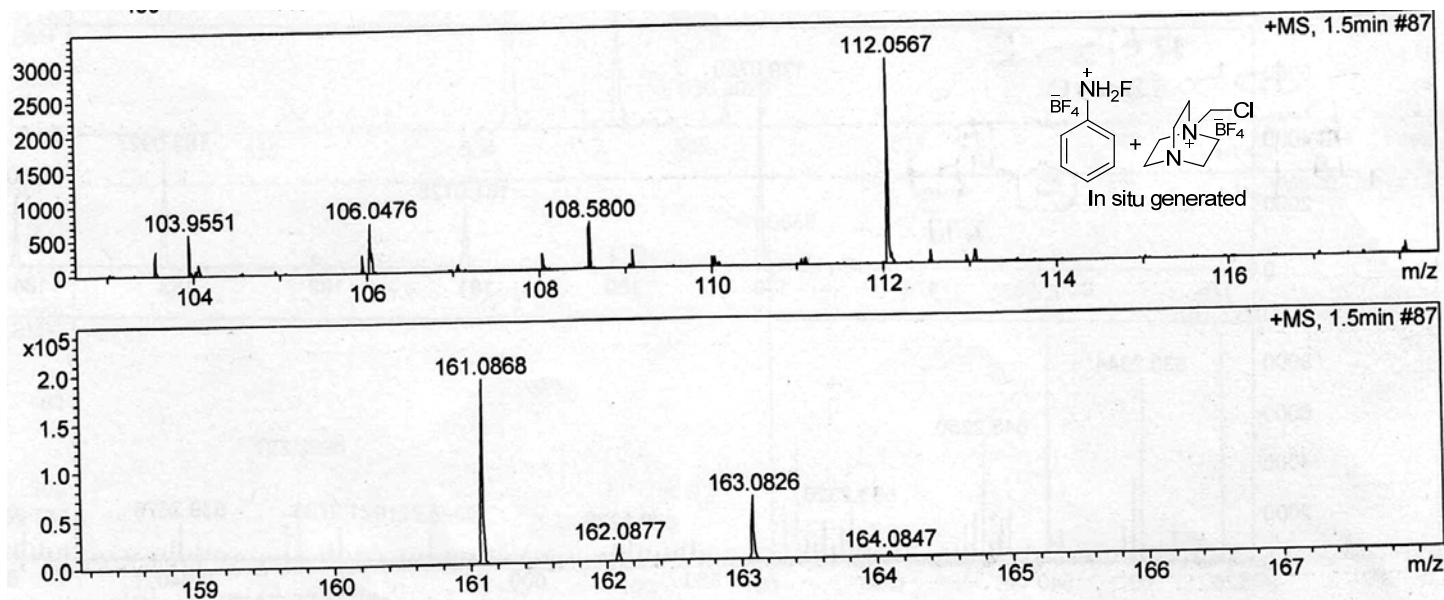


Figure 3. HRMS of in situ generated N-Fluorobenzenaminium tetrafluoroborate

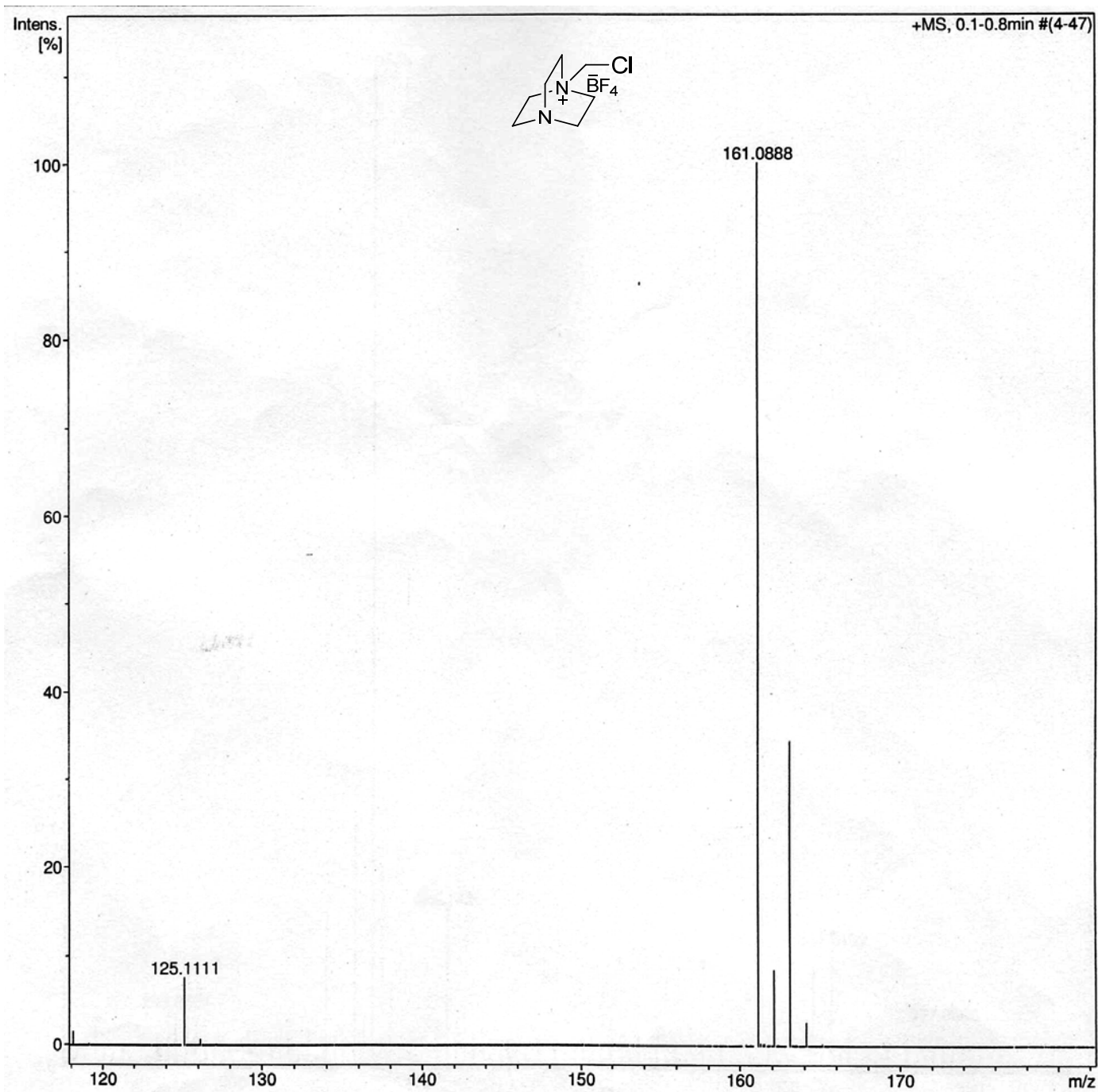


Figure 4. HRMS of 1-(chloromethyl)-1,4-diazabicyclo[2.2.2]octan-1-ium chloride with tetrafluoroborate

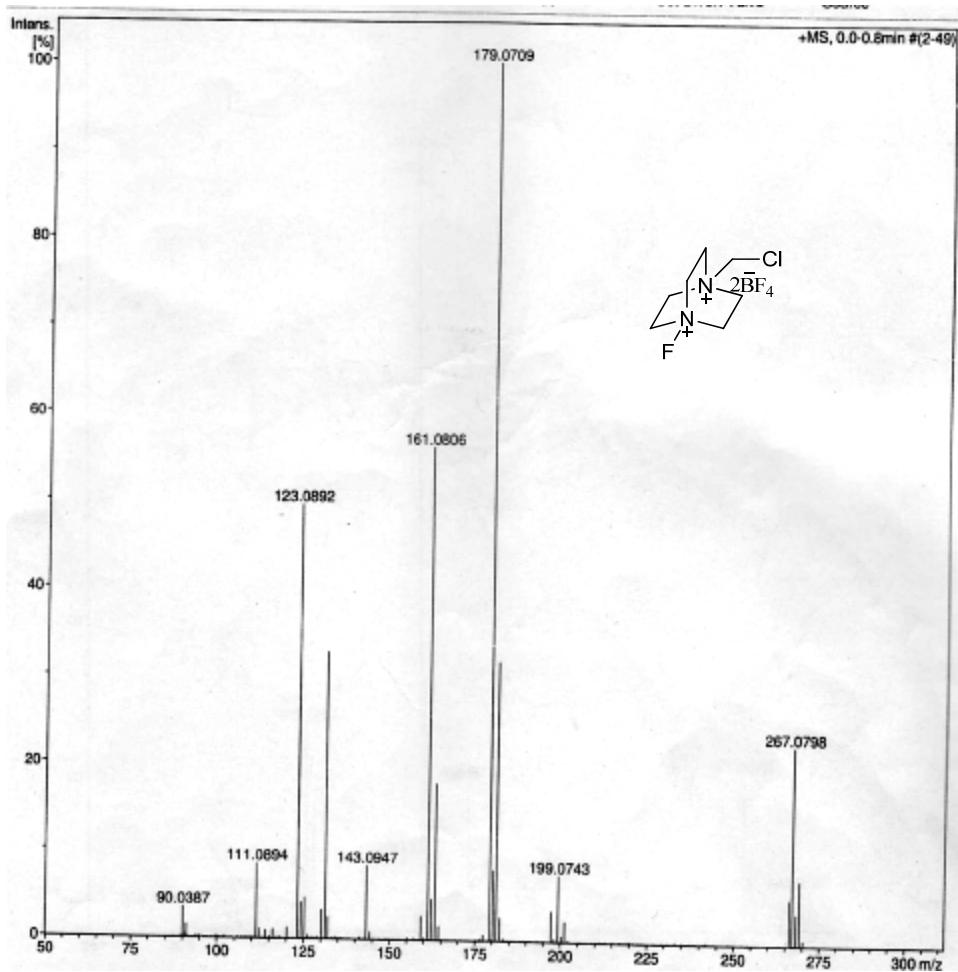
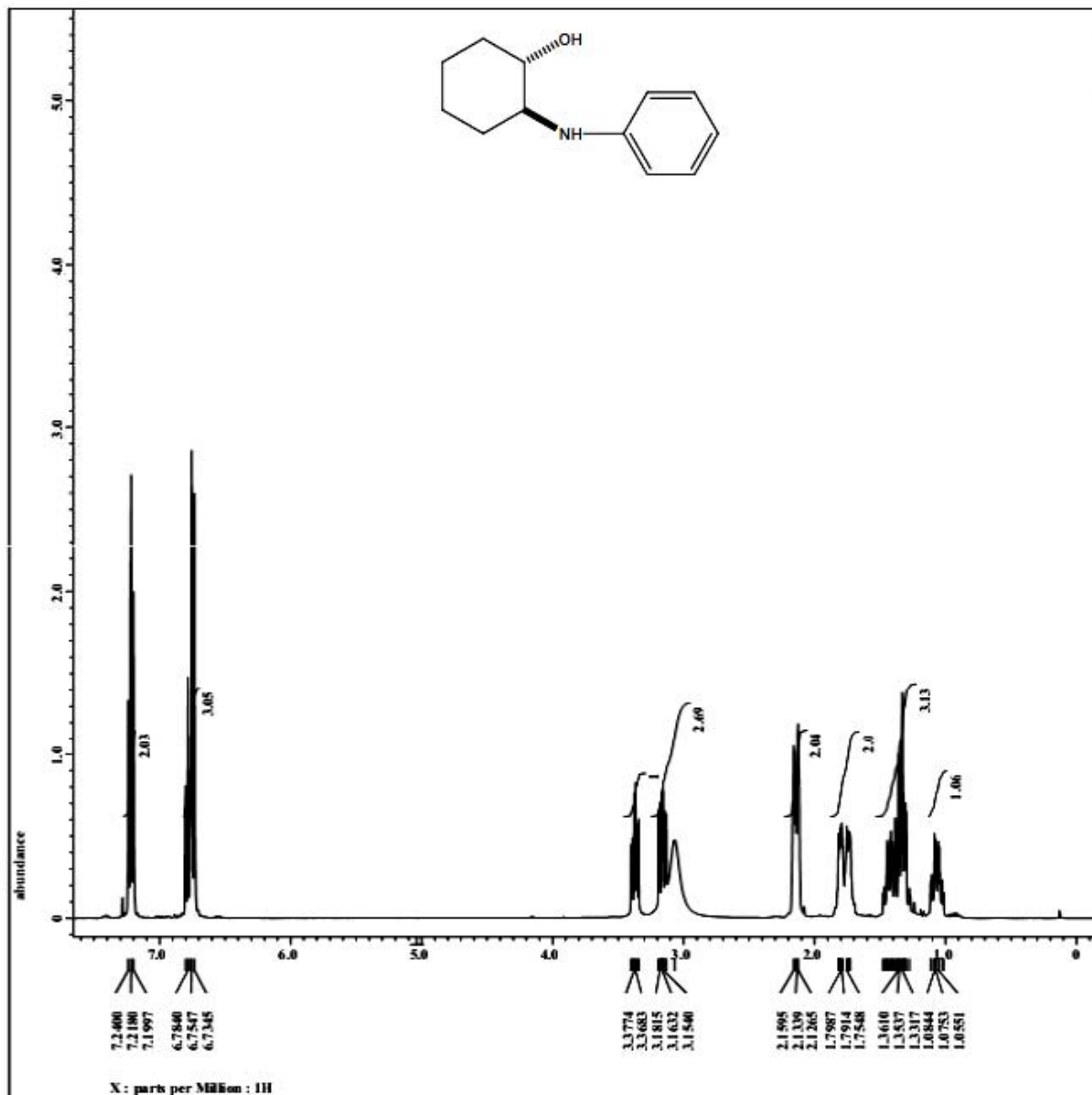


Figure 5. HRMS of Selectfluor

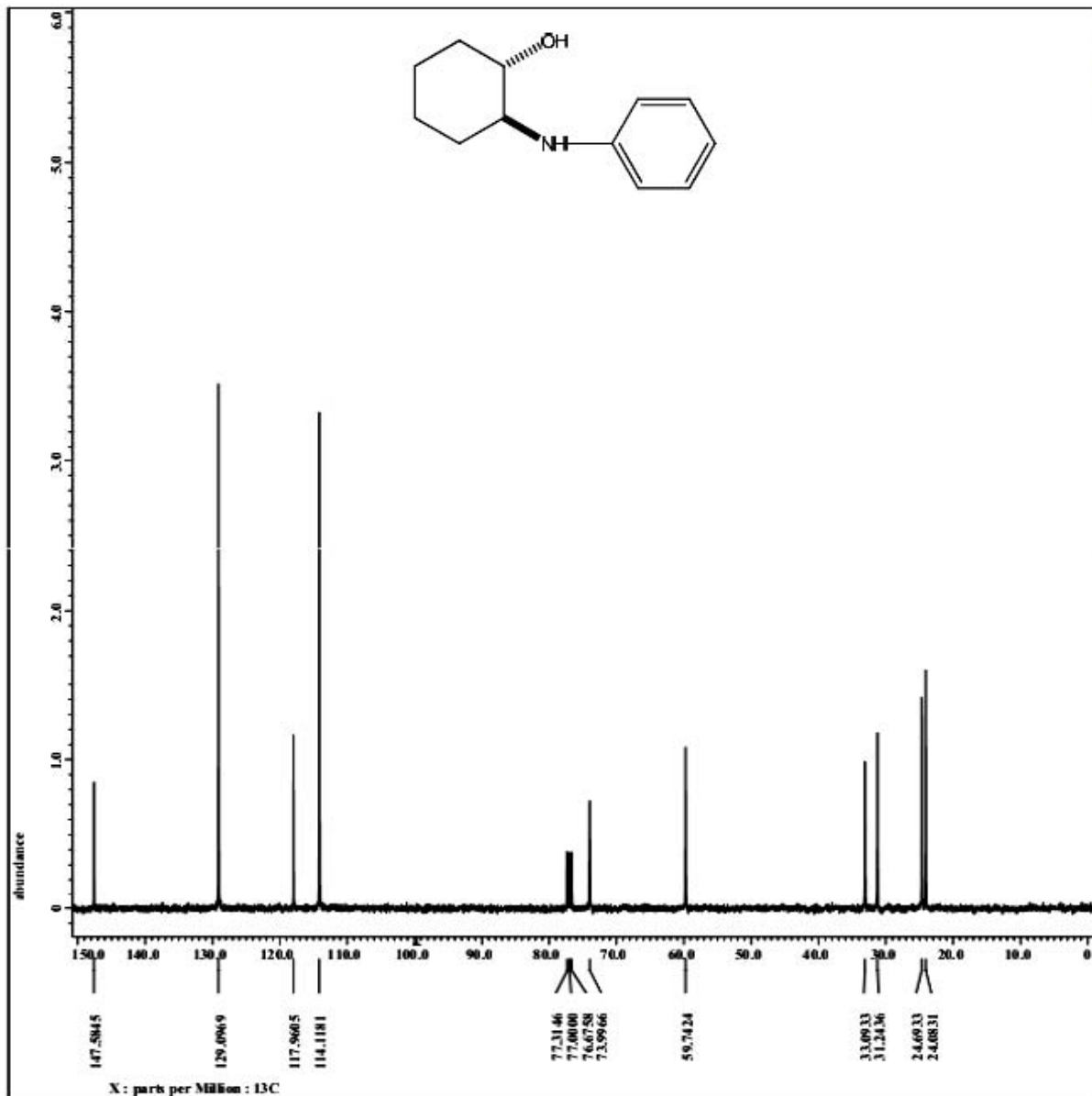


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 Creation time = 28-JUL-2012 13:08:29
 Revision time = 4-AUG-2012 11:05:03
 Current time = 4-AUG-2012 11:05:24

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 Dim title = 1H
 Dim units = [ppm]
 Dimensions = X
 Site = ECK 400P
 Spectrometer = DELTA2 NMR

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 X_domain = 1H
 X_freq = 399.78219838[MHz]
 X_offset = 5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.73218757[Hz]
 X_sweep = 11.99616123[kHz]
 IRR_domain = 1H
 IRR_freq = 399.78219838[MHz]
 IRR_offset = 5[ppm]
 TRI_domain = 1H
 TRI_freq = 399.78219838[MHz]
 TRI_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 16
 Total_scans = 16

X_90_width = 11.57[us]
 X_acq_time = 1.36577024[s]
 X_angle = 45[deg]
 X_atn = 5[dB]
 X_pulse = 5.785[us]
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 TRI_mode = Off
 Delta_preset = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 24
 Relaxation_delay = 4[s]
 Repetition_time = 5.36577024[s]
 Temp_get_time = 20.3[dC]



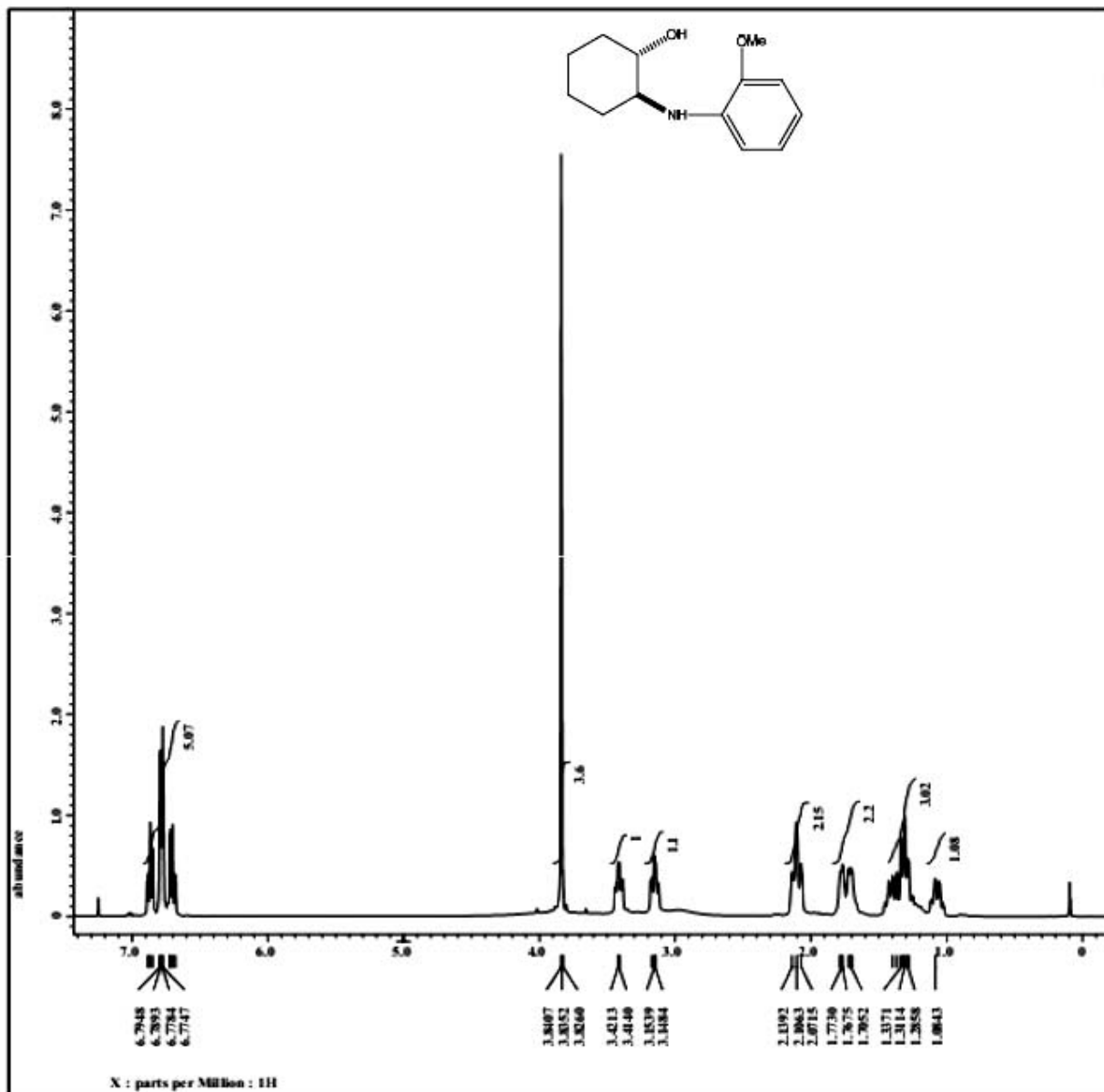
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Author       = delta
Experiment   = single_pulse_dec
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Solvent      = CHLOROFORM-D
Creation_time = 1-AUG-2012 21:41:04
Revision_time = 1-AUG-2012 22:01:33
Current_time  = 4-AUG-2012 11:07:09

Comment      = SS 4-03
Data format  = 1D COMPLEX
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Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECK 400P
Spectrometer = DELTA2 NMR

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X_offset     = 100[ppm]
X_points     = 32768
X_prescans   = 4
X_resolution = 0.95846665[Hz]
X_sweep      = 31.40703518[kHz]
Irr_domain   = 1H
Irr_freq     = 399.78219838[MHz]
Irr_offset   = 5[ppm]
Clipped      = FALSE
Mod_return   = 1
Scafs        = 14
Total_scans  = 14

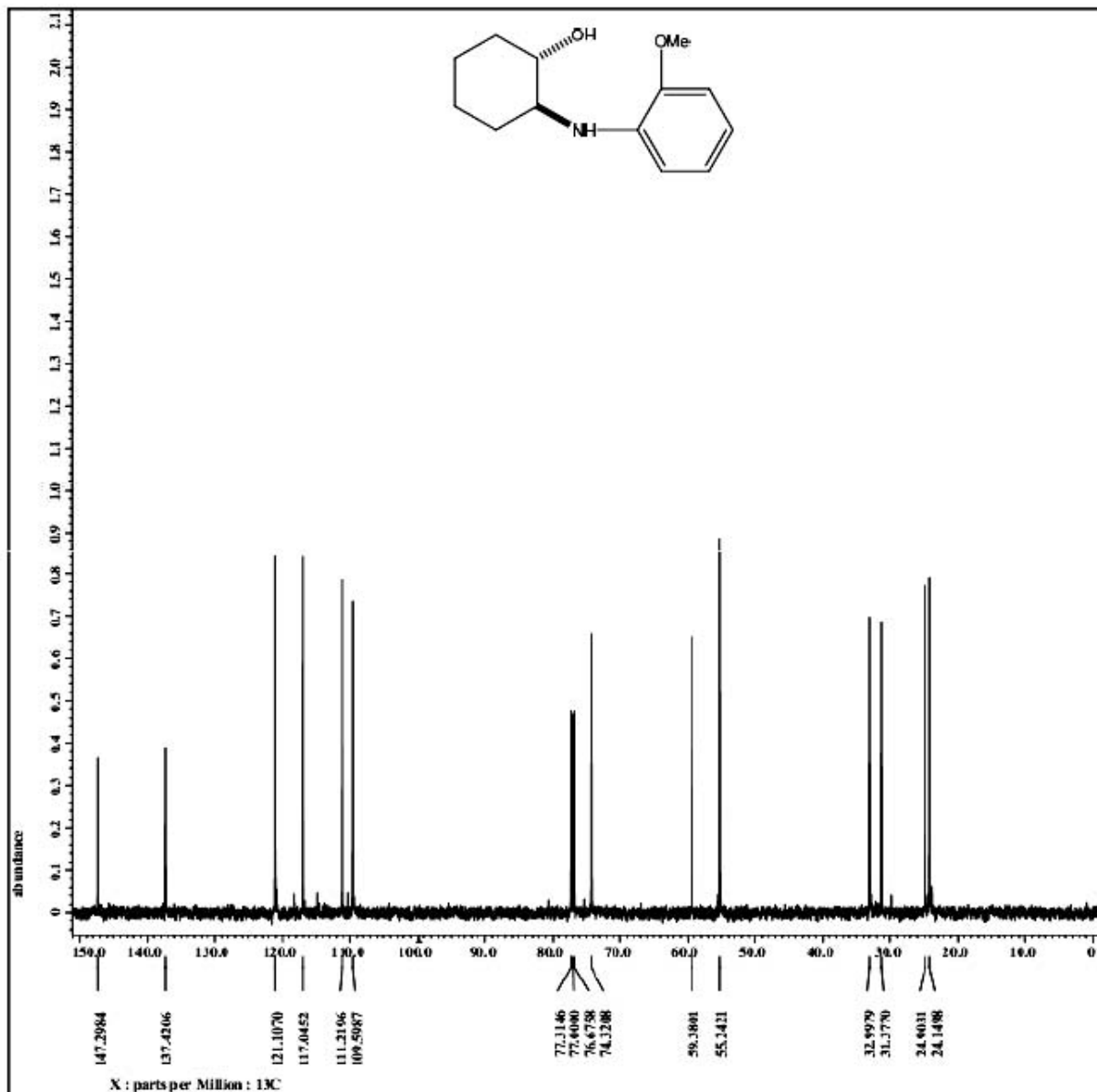
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X_acq_time   = 1.0433312[s]
X_angle      = 30[deg]
X_atn        = 10[dB]
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Irr_atn_dec  = 24.95[dB]
Irr_atn_noe  = 24.95[dB]
Irr_noise    = WALTZ
Decoupling   = TRUE
Initial_wait = 1[s]
Noe          = TRUE
Noe_time     = 2[s]
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Relaxation_delay = 2[s]
Repetition_time = 3.0433312[s]
Temp_get     = 19.6[degC]
  
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 Author = delta
 Experiment = single_pulse.ex2
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 Solvent = CDCl3
 Creation_time = 30-NOV-2011 11:28:42
 Revision_time = 29-JUL-2012 16:16:13
 Current_time = 29-JUL-2012 16:16:35

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 Dir_title = 1H
 Dir_units = [ppm]
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 Site = KCK 400P
 Spectrometer = DELTA2_MMR

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 X_offset = 5[ppm]
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 X_resolution = 0.73218757[Hz]
 X_sweep = 11.99616123[kHz]
 Iir_domain = 1H
 Iir_freq = 399.78219838(MHz)
 Iir_offset = 5[ppm]
 Tri_domain = 1H
 Tri_freq = 399.78219838(MHz)
 Tri_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 16
 Total_scans = 16
 X_90_width = 11.57[us]
 X_acq_time = 1.36577024[s]
 X_angle = 45[deg]
 X_atn = 5[dB]
 X_pulse = 5.785[us]
 Iir_mode = Off
 Tri_mode = Off
 Data_preset = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 24
 Relaxation_delay = 4[s]
 Repetition_time = 5.36577024[s]
 Temp_get = 21.5[degC]

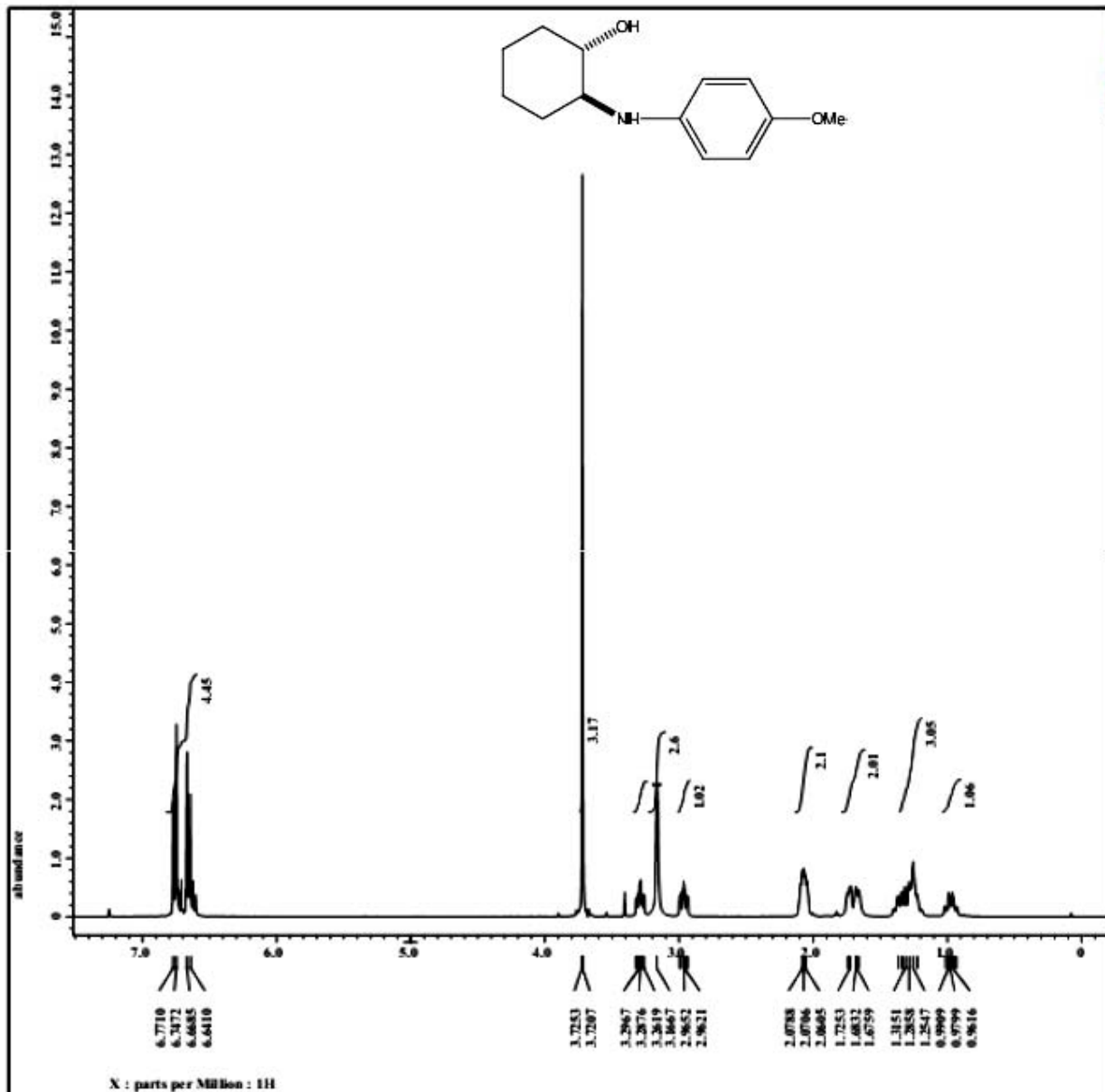


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 Creation time = 9-DEC-2011 17:16:28
 Revision time = 9-DEC-2011 17:19:43
 Current time = 29-JUL-2012 16:17:21

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 File title = 13C
 File units = [ppm]
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 Spectrometer = DELTA2 MMH

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 X_domain = 13C
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 X_offset = 100 (ppm)
 X_points = 32768
 X_prescans = 4
 X_resolution = 0.95846665 (Hz)
 X_sweep = 31.40703518 (kHz)
 Iir_domain = 1H
 Iir_freq = 399.78219838 (MHz)
 Iir_offset = 5 (ppm)
 Clipped = FALSE
 No. scans = 21
 Total scans = 21

X_90 width = 11.75 (us)
 X_acq time = 1.0433312 (s)
 X_angle = 30 (deg)
 X_atn = 10 (dB)
 X_pulse = 3.91666667 (us)
 Iir_atn_dec = 24.95 (dB)
 Iir_atn_noe = 24.95 (dB)
 Iir_noise = WALTZ
 Decoupling = TRUE
 Initial wait = 1 (s)
 Noe = TRUE
 Noe time = 2 (s)
 Recvr gain = 54
 Relaxation delay = 2 (s)
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 Temp_get = 23.2 (deg)



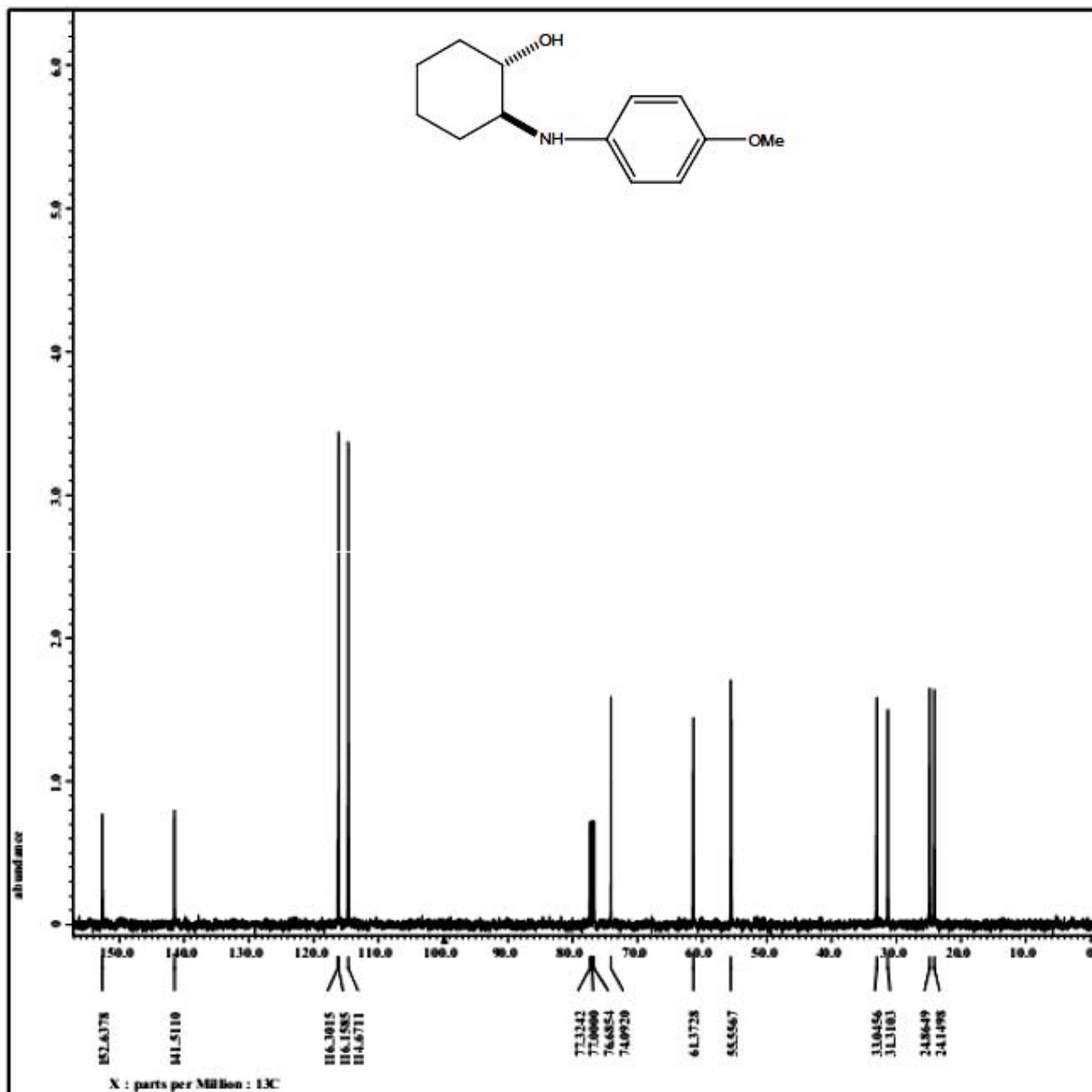
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Creation_time = 29-NOV-2011 09:58:54
Revision_time = 29-JUL-2012 16:22:23
Current_Time = 29-JUL-2012 16:22:41

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Data_size    = 13107
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Dir_units    = [ppm]
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Site         = KCM 400P
Spectrometer = DELTA2_MMR

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X_acq_duration = 1.36577024[s]
X_domain       = 1H
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X_points       = 16384
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X_sweep       = 11.99616123[kHz]
Irr_domain    = 1H
Irr_freq      = 399.78219838[Mhz]
Irr_offset    = 5[ppm]
Tri_domain    = 1H
Tri_freq      = 399.78219838[Mhz]
Tri_offset    = 5[ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 16
Total_scans   = 16

X_90_width    = 11.57[us]
X_acq_time     = 1.36577024[s]
X_angle        = 45[deg]
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X_pulse        = 5.785[us]
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Dante_preset  = FALSE
Init_wait     = 1[s]
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Temp_get       = 22.2[dc]
  
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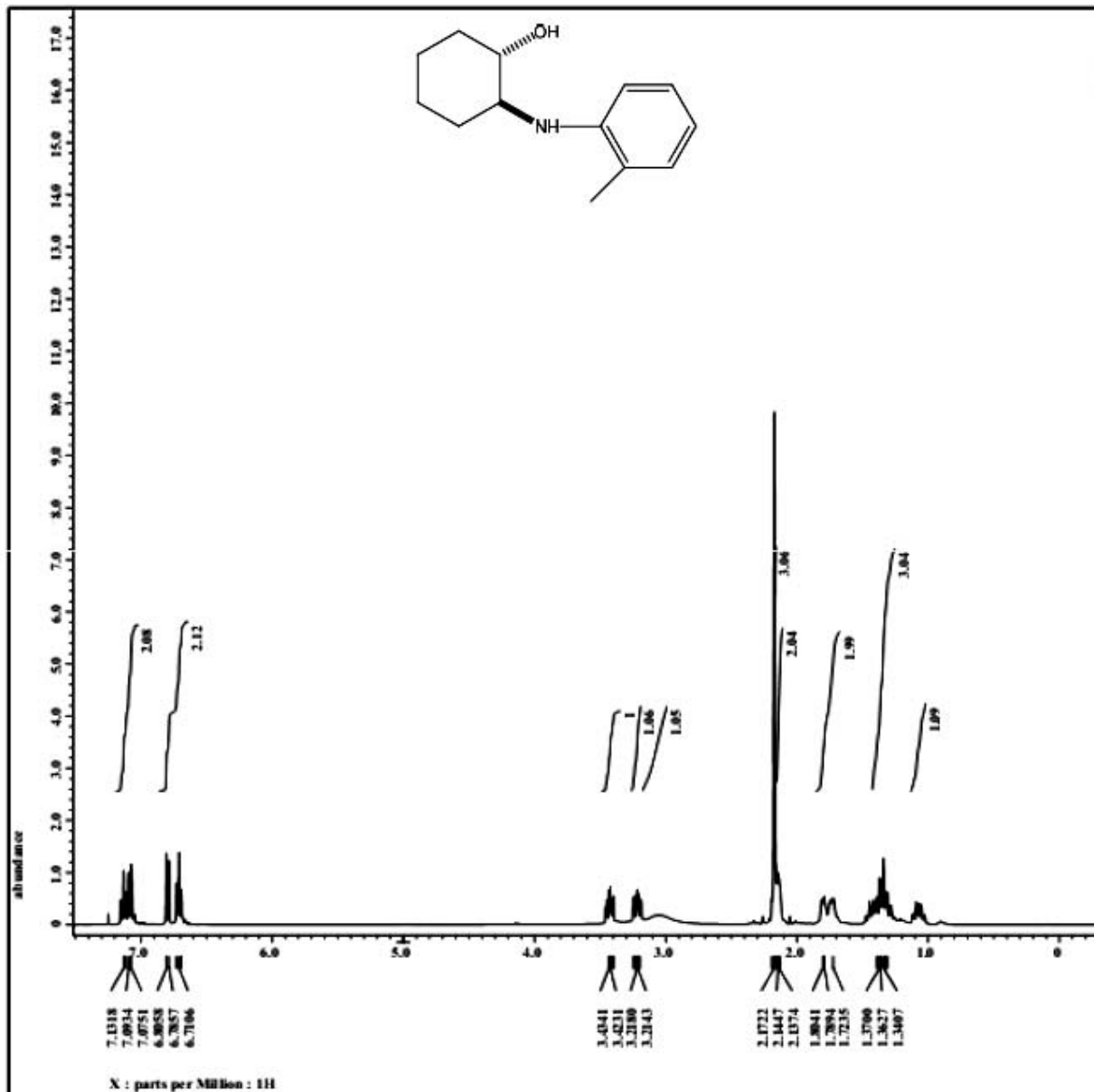
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Solvent       = CHLOROFORM-D
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Revision_time = 30-NOV-2011 20:35:31
Current_Time  = 29-JUL-2012 16:23:21

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Irr_atn_now    = 24.95 [dB]
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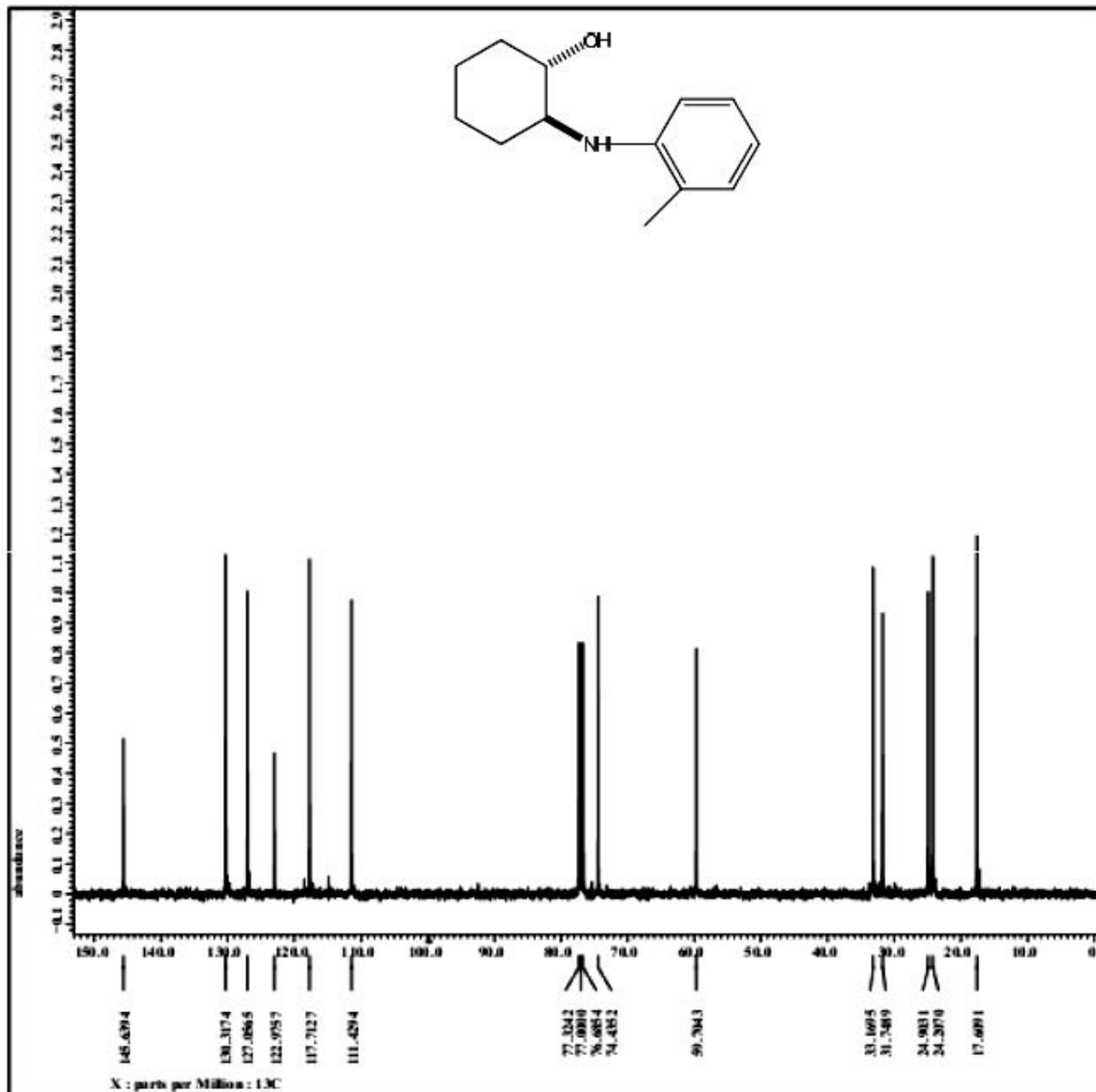
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Spectrometer  = DELTA2_MMR

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X_freq         = 399.78219838[Mhz]
X_offset       = 5[ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.73218757[Hz]
X_sweep        = 11.99616123[kHz]
Irr_domain     = 1H
Irr_freq       = 399.78219838[Mhz]
Irr_offset     = 5[ppm]
Tri_domain     = 1H
Tri_freq       = 399.78219838[Mhz]
Tri_offset     = 5[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 16
Total_scans    = 16

X_90_width     = 11.57[us]
X_acq_time     = 1.36577024[s]
X_angle        = 45[deg]
X_atn          = 5[db]
X_pulse        = 5.785[us]
Irr_mode       = Off
Tri_mode       = Off
Dante_preset   = FALSE
Init_wait      = 1[s]
Recvr_gain     = 26
Relaxation_delay = 4[s]
Repetition_time = 5.36577024[s]
Temp_get       = 22.3[dc]
  
```



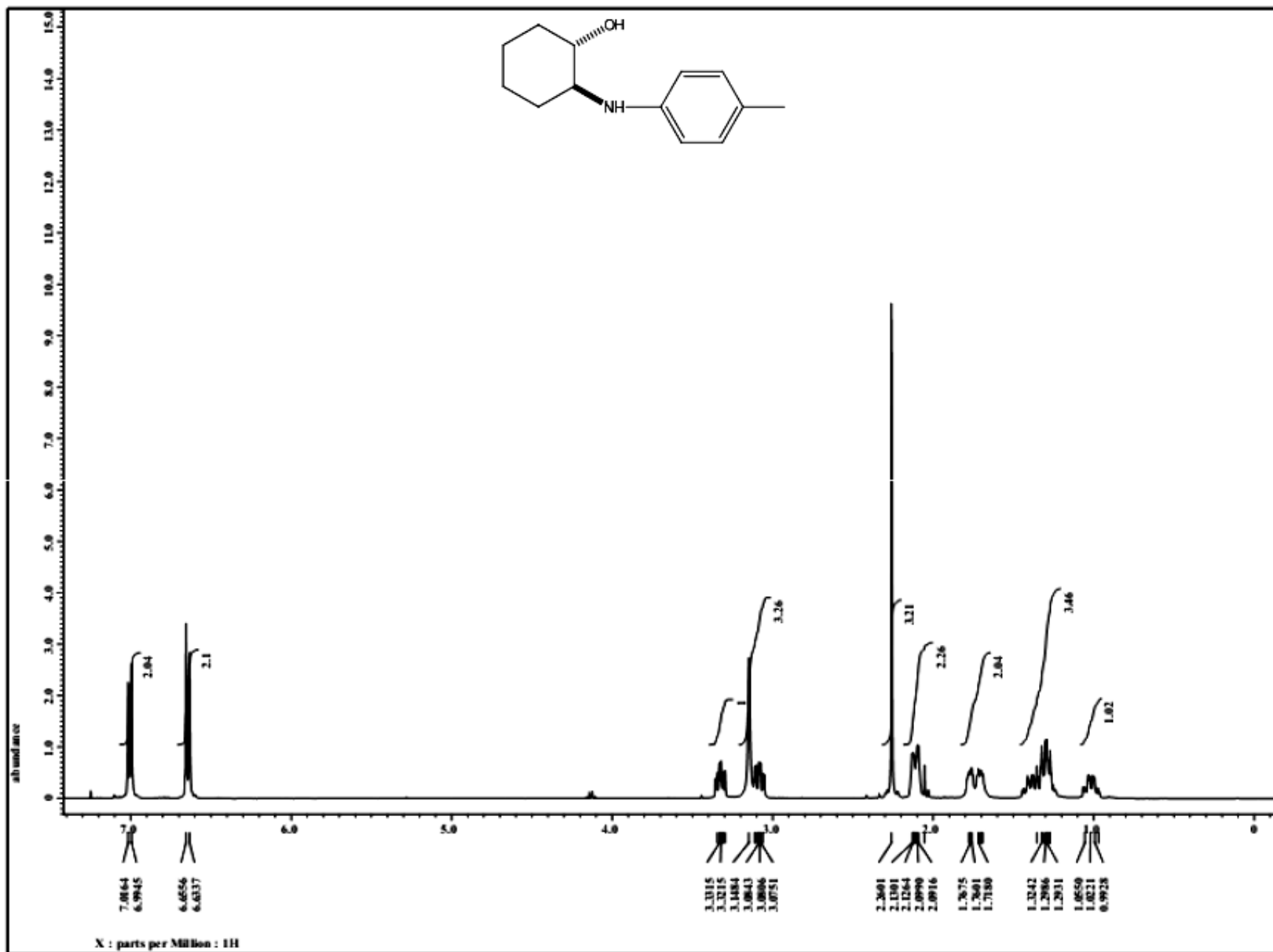
```

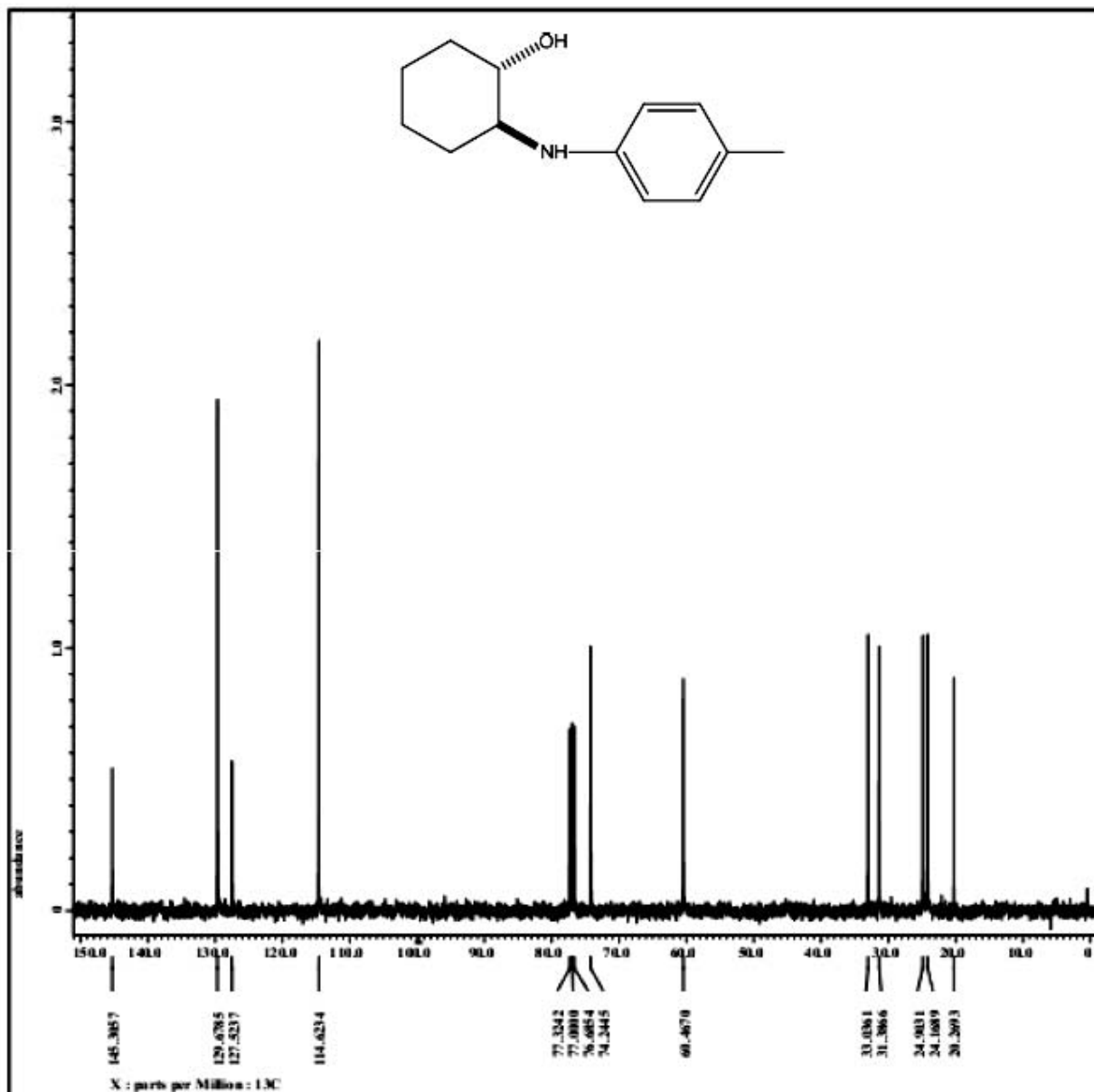
Filename      = SS 3-7307_CARBON-3.jc
Author        = deTia
Experiment    = single_pulse_dec
Sample_id     = SS 3-7307
Solvent       = CHLOROFORM-D
Creation_time  = 7-MAY-2012 20:08:57
Revision_time = 7-MAY-2012 20:23:00
Current_time  = 29-JUL-2012 18:30:34

Comment       = SS 3-7307
Data_format   = ID COMPLEX
Dir_name      = 28214
Dir_title     = 13C
Dir_units     = [ppm]
Dimensions    = X
Site          = NEX 400P
Spectrometer  = DELTA2_MMR

Field_strength = 9.389766[T] (400[MHz])
X_acq_duration = 1.04333312[s]
X_domain       = 13C
X_freq         = 100.62530333[MHz]
X_offset       = 100[ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 0.25846665[Hz]
X_sweep        = 11.40702018[kHz]
Irr_domain     = 1H
Irr_freq       = 399.78219838[MHz]
Irr_offset     = 0[ppm]
Clipped        = ALL32
-----
Scans          = 78
Total_scans    = 78

X_90_width     = 11.75[us]
X_acq_time     = 1.04333312[s]
X_angle        = 30[deg]
X_atn          = 10[dB]
X_pulse        = 3.91666667[us]
Irr_atn_dec    = 24.95[dB]
Irr_atn_noe    = 24.95[dB]
Irr_noise      = 98.172
Decoupling     = THCE
Initial_wait    = 1[s]
Noe            = THCE
Noe_time       = 2[s]
Recvr_gain     = 60
Relaxation_delay = 2[s]
Repetition_time = 1.04333312[s]
Temp_get       = 23.1[degC]
  
```





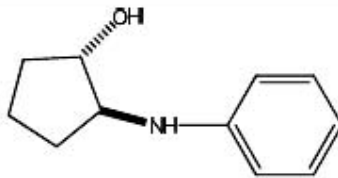
```

Filename      = DE 3-74_PTD_CARBON-1
Author        = deTia
Experiment    = single_pulse_dec
Sample id     = DE 3-74_PTD
Solvent      = CHLOROFORM-D
Creation time = 11-MAY-2012 00:32:03
Revision time = 11-MAY-2012 01:06:03
Current time  = 29-JUL-2012 18:31:40

Comment       = DE 3-74_PTD
Data format   = 1D_COMPLEX
Dir_name      = 28214
Dir_title     = 13C
Dir_units     = (ppm)
Dimensions    = X
Site          = HXK 400P
Spectrometer  = DELTA2_NMR

Field strength = 3.389766(T) (400(MHz))
X_acq_duration = 1.04333312(s)
X_domain       = 13C
X_freq         = 100.52530333(MHz)
X_offset       = 100(ppm)
X_points       = 32768
X_prescans     = 4
X_resolution   = 0.35846665(Hz)
X_sweep        = 11.40702018(kHz)
Irr_domain     = 13C
Irr_freq       = 399.78219838(MHz)
Irr_offset     = 0(ppm)
Clipped        = ALL32
Mod_4turns     = 1
Scale          = 14
Total_scans    = 14

X_90_width     = 11.75(us)
X_acq_time     = 1.04333312(s)
X_angle        = 30(deg)
X_atn          = 10(dB)
X_pulse        = 3.91666667(us)
Irr_atn_dec    = 24.95(dB)
Irr_atn_poc    = 24.95(dB)
Irr_noise      = 98.172
Decoupling     = 1902
Initial_wait   = 1(s)
Noe            = 1902
Noe_time       = 2(s)
RecVr_gain     = 28
Relaxation_delay = 2(s)
Repetition_time = 1.04333312(s)
Temp_get       = 22.9(dC)
  
```

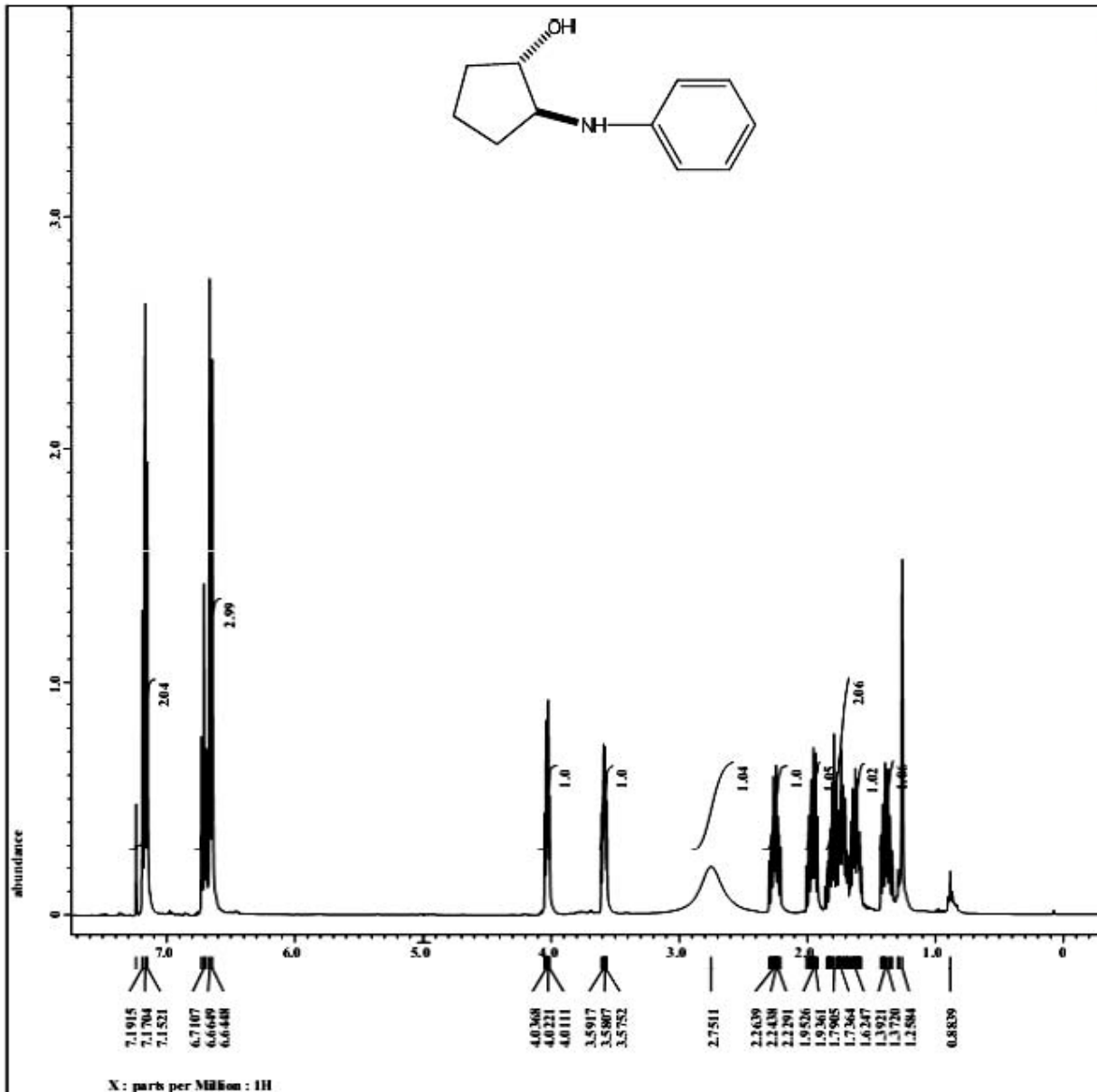



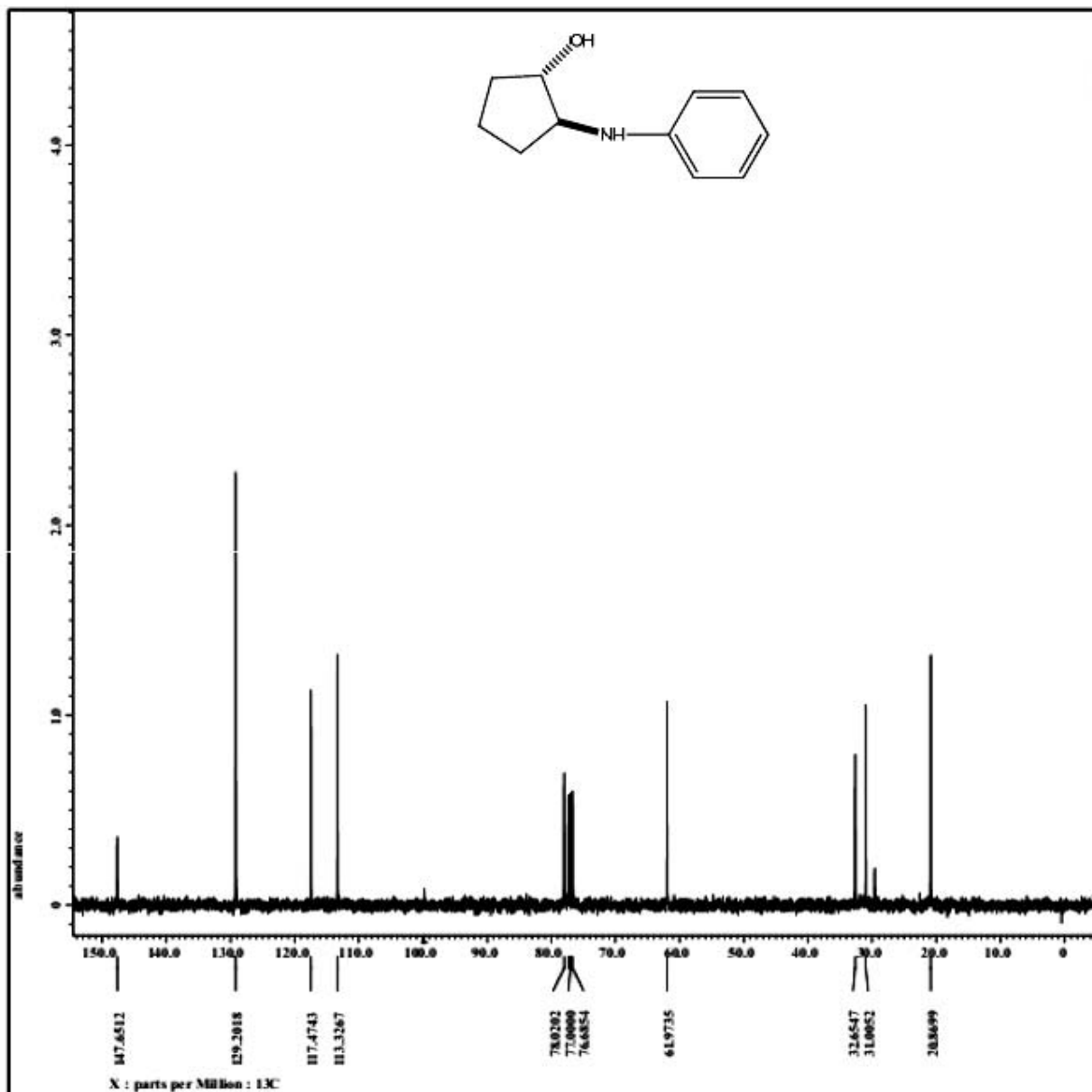
Filename = SS_AMLCPD_PROTON-5.jd
 Author = delta
 Experiment = single pulse.ex2
 Sample_id = SS_AMLCPD
 Solvent = CHLOROFORM-D
 Creation_time = 20-DEC-2011 12:36:45
 Revision_time = 29-JUL-2012 16:48:05
 Current_time = 29-JUL-2012 16:48:19

Comment = SS_AMLCPD
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECK 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 1.36577024[s]
 X_domain = 1H
 X_freq = 399.78219838[MHz]
 X_offset = 5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.73218757[Hz]
 X_sweep = 11.99616123[kHz]
 IR_domain = 1H
 IR_freq = 399.78219838[MHz]
 IR_offset = 5[ppm]
 Tri_domain = 1H
 Tri_freq = 399.78219838[MHz]
 Tri_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 16
 Total_scans = 16

X_90_width = 11.57[us]
 X_acq_time = 1.36577024[s]
 X_angle = 45[deg]
 X_atn = 5[dB]
 X_pulse = 5.785[us]
 IR_mode = Off
 Tri_mode = Off
 Dante_preset = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 30
 Relaxation_delay = 4[s]
 Repetition_time = 5.36577024[s]
 Temp_get = 22.5[dC]





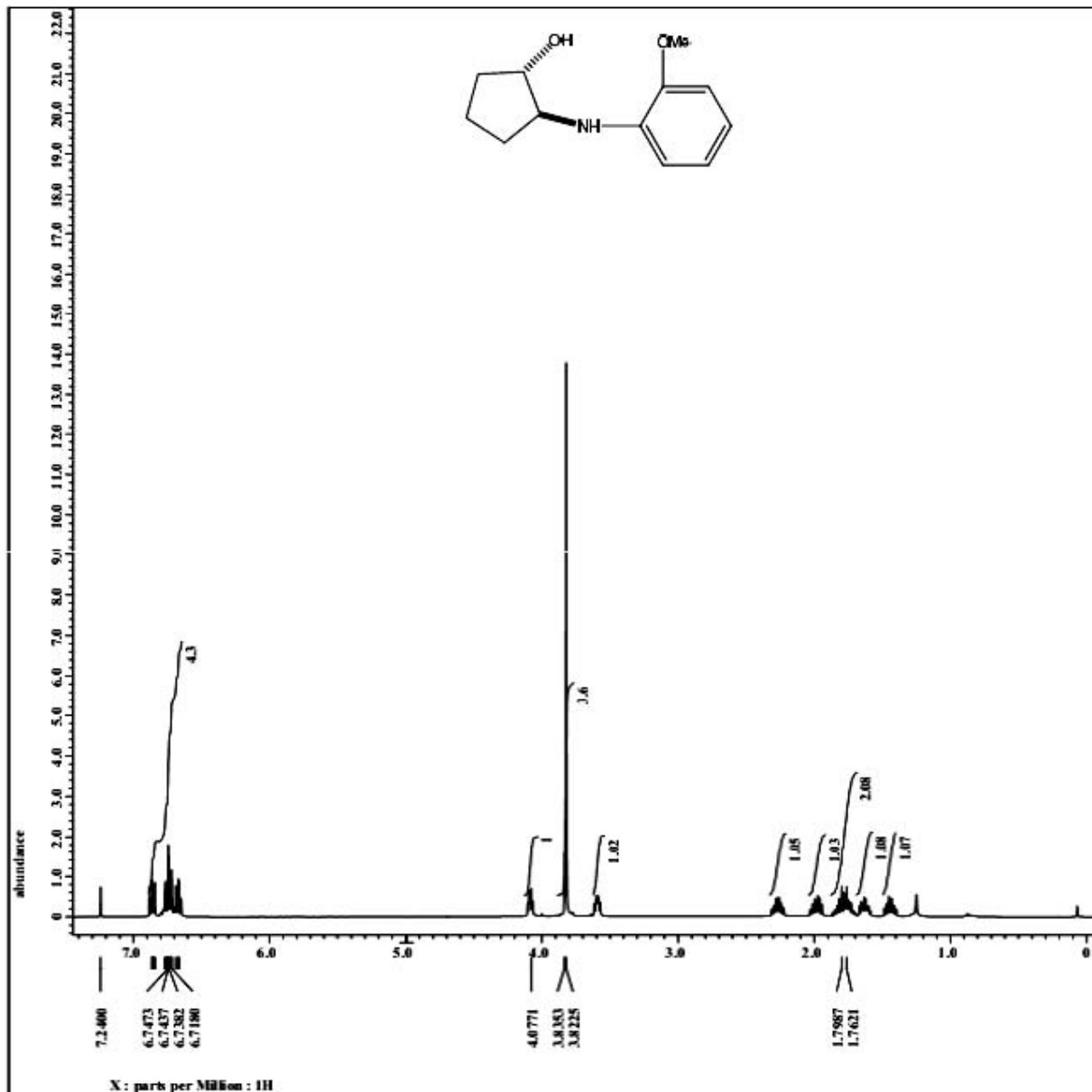
```

Filename      = SS ANLCHO_CARBON-3.jd
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = SS ANLCHO
Solvent      = CHLOROFORM-D
Creation_time = 26-DEC-2011 23:43:19
Revision_time = 26-DEC-2011 23:46:07
Current_Time = 29-JUL-2012 16:49:12

Comment      = SS ANLCHO
Data_format  = 1D COMPLEX
Data_size    = 26214
Dir_title    = 13C
Dir_units    = [ppm]
Dimensions   = X
Site         = KXC 400P
Spectrometer = DELTA2_MMR

Field_strength = 9.389766[T] [400[Mhz]]
X_acq_duration = 1.0433332[s]
X_domain      = 13C
X_freq        = 100.62530333 [MHz]
X_offset      = 100[ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 0.95846665 [Hz]
X_sweep       = 31.40703518 [kHz]
Irr_domain    = 1H
Irr_freq      = 399.78219838 [MHz]
Irr_offset    = 5[ppm]
Clipped       = FALSE
Modulation    = -
Scale         = 14
Total_scans   = 14

X_90_width    = 11.75[us]
X_acq_time    = 1.0433332[s]
X_angle       = 30[deg]
X_atn         = 10[db]
X_pulse       = 3.91666667[us]
Irr_atn_dec   = 24.95[db]
Irr_atn_pow   = 24.95[db]
Irr_noise     = WALTZ
Decoupling    = FMR
Initial_wait  = 1[s]
New_time      = 786
New_time      = 2[s]
Recvr_gain    = 60
Relaxation_delay = 2[s]
Repetition_time = 3.0433332[s]
Temp_get      = 22.9[dc]
  
```

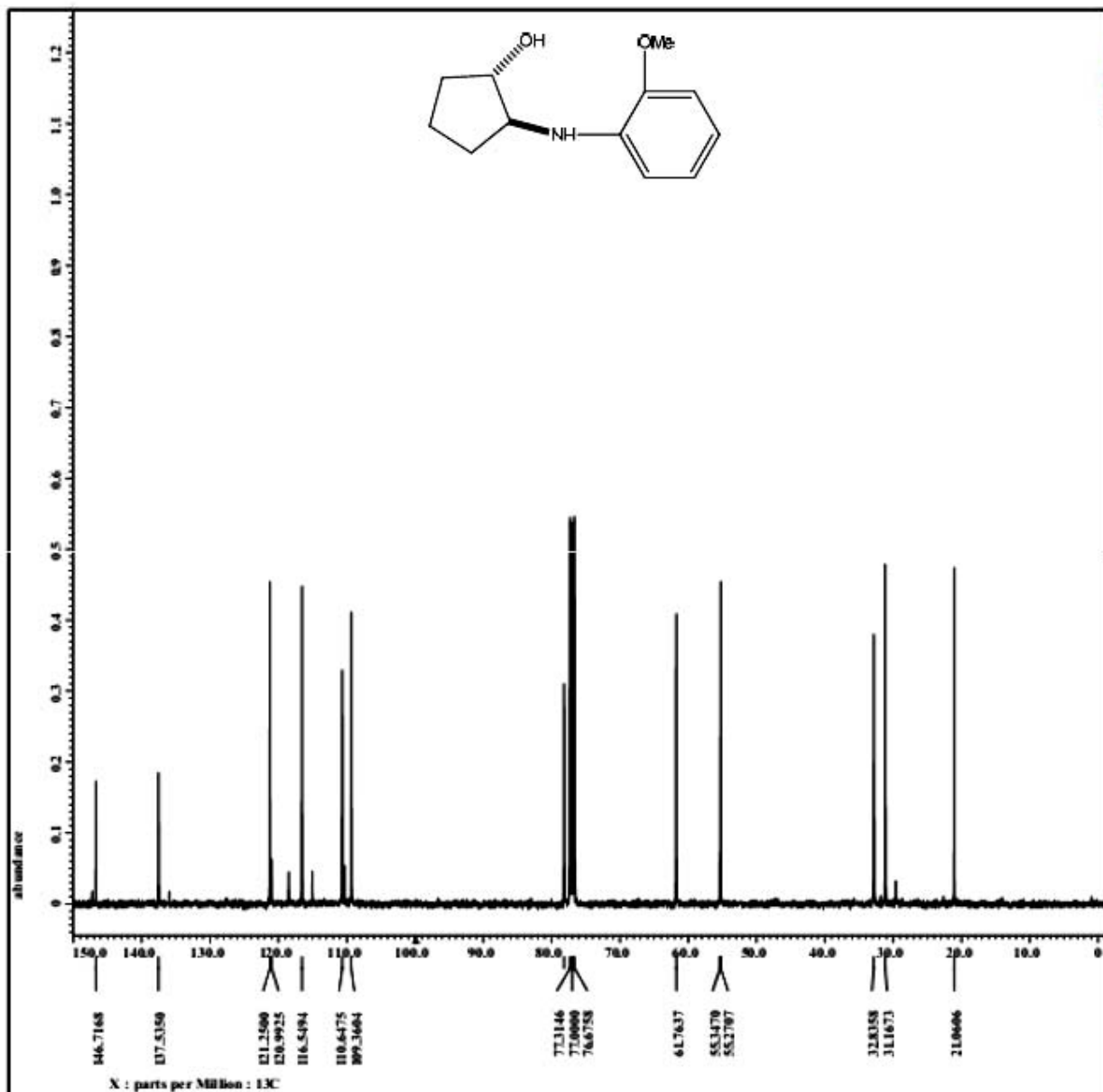


Filename = SS CPOOM_PROTON-4.jdf
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = SS CPOOM
 Solvent = CHLOROFORM-D
 Creation_time = 20-DEC-2011 12:42:53
 Revision_time = 29-JUL-2012 16:51:22
 Current_time = 29-JUL-2012 16:51:51

Comment = SS CPOOM
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECK 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 1.36577024[s]
 X_domain = 1H
 X_freq = 399.78219838[MHz]
 X_offset = 5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.73218757[Hz]
 X_sweep = 11.99616123[kHz]
 IR_domain = 1H
 IR_freq = 399.78219838[MHz]
 IR_offset = 5[ppm]
 Tri_domain = 1H
 Tri_freq = 399.78219838[MHz]
 Tri_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 16
 Total_scans = 16

X_90_width = 11.57[us]
 X_acq_time = 1.36577024[s]
 X_angle = 45[deg]
 X_atn = 5[dB]
 X_pulse = 5.795[us]
 IR_mode = Off
 Tri_mode = Off
 Dante_preset = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 32
 Relaxation_delay = 4[s]
 Repetition_time = 5.36577024[s]
 Temp_get = 22.1[dC]



```

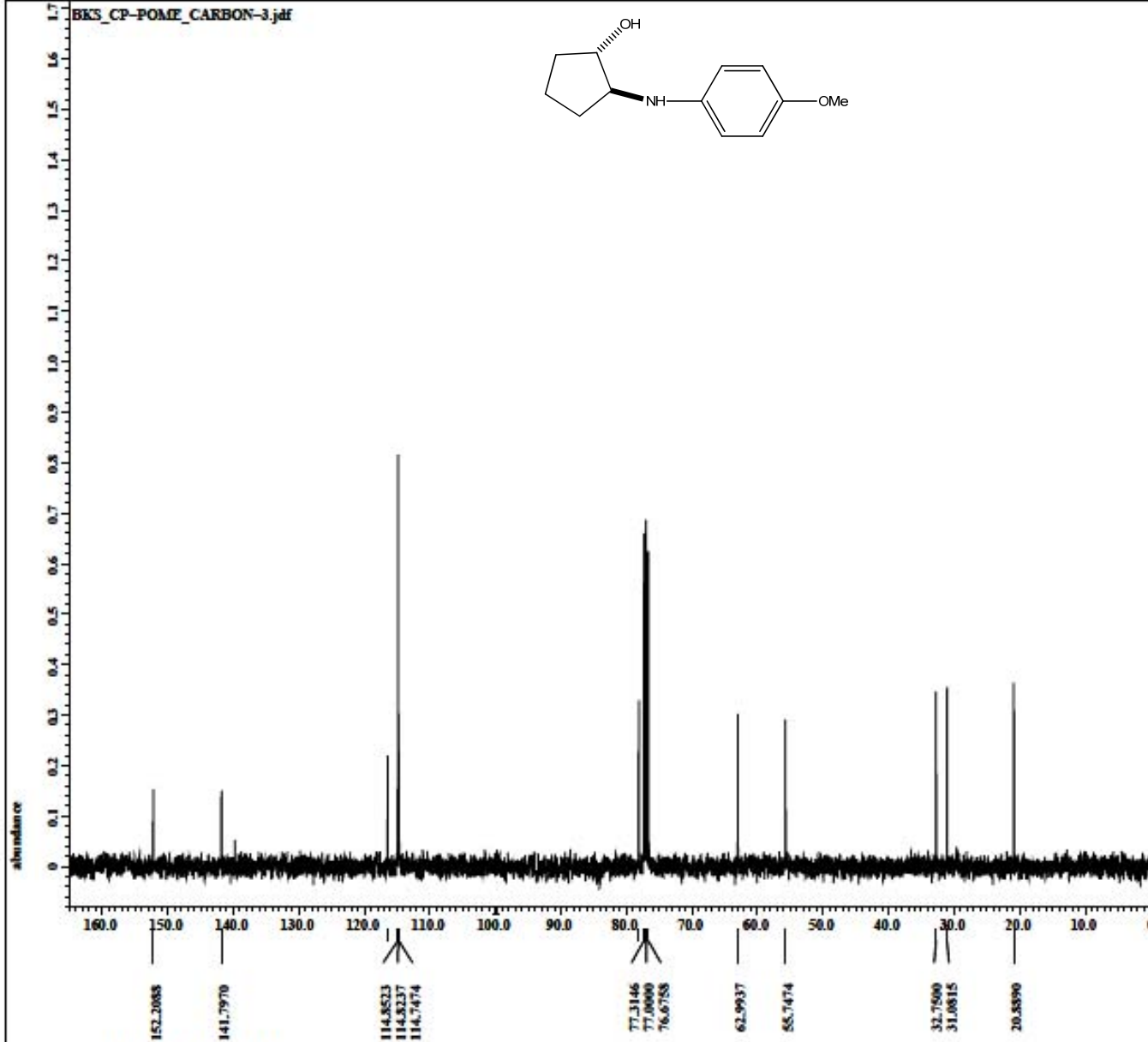
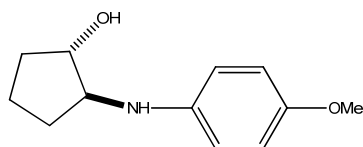
Filename      = SS CPOON_CARBON-4.jdf
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = SS CPOON
Solvent      = CHLOROFORM-D
Creation_time = 24-DEC-2011 00:19:43
Revision_time = 29-JUL-2012 16:53:00
Current_Time = 29-JUL-2012 16:53:10

Comment      = SS CPOON
Data_format  = 1D COMPLEX
Data_size    = 26214
Data_title   = 13C
Data_units   = [ppm]
Dimensions   = X
Site         = KCM 400P
Spectrometer = DELTA2_MMR

Field_strength = 9.389766[T] [400[MHz]]
X_acq_duration = 1.0433332[s]
X_domain       = 13C
X_freq         = 100.62530333[MHz]
X_offset       = 100[ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 0.95846665[Hz]
X_sweep        = 31.40703518[kHz]
Irr_domain     = 1H
Irr_freq       = 399.78219838[MHz]
Irr_offset     = 5[ppm]
Clipped       = FALSE
Proc_method    =
Scale         = 393
Total_scans    = 393

X_90_width    = 11.75[us]
X_acq_time     = 1.0433332[s]
X_angle        = 30[deg]
X_atn          = 10[db]
X_pulse        = 3.91666667[us]
Irr_atn_dec    = 24.95[db]
Irr_atn_now    = 24.95[db]
Irr_noise      = WALTZ
Decoupling     = FMR
Initial_wait   = 1[s]
New_time       = 766
New_time       = 2[s]
RecVr_gain     = 58
Relaxation_delay = 2[s]
Repetition_time = 3.0433332[s]
Temp_get       = 22.1[dc]
  
```


BKS_CP-POME_CARBON-3.jdf



X : part per Million : 13C

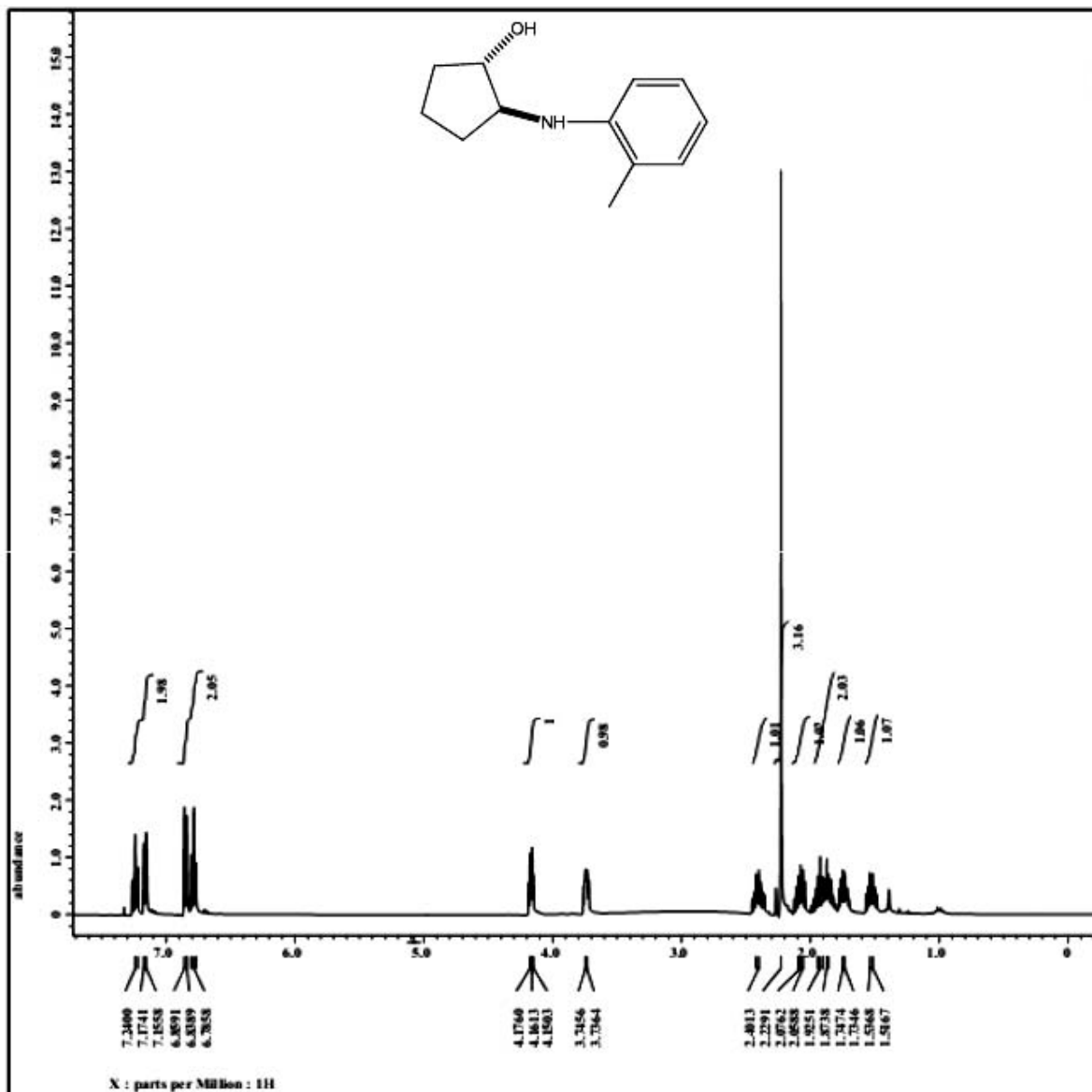


Filename = BKS_CP-POME_CARBON-3.
Author = delta
Experiment = single_pulse_dec
Sample id = BKS_CP-POME
Solvent = CHLOROFORM-D
Creation time = 26-DEC-2011 23:17:34
Revision time = 26-DEC-2011 23:20:22
Current time = 15-JAN-2012 16:55:26

Comment = BKS_CP-POME
Data format = 1D COMPLEX
Dim size = 26214
Dim title = 13C
Dim units = [ppm]
Dimensions = X
Site = RCI 400P
Spectrometer = DELTA2_NMR

Field strength = 9.389766 [T] (400 [MHz])
F1 acq duration = 1.04333312 [s]
F1 domain = 13C
F1 freq = 100.52530333 [MHz]
F1 offset = 100 [ppm]
F1 points = 32768
F1 prescans = 4
F1 resolution = 0.95846665 [Hz]
F1 sweep = 31.40703518 [kHz]
Irr domain = 1H
Irr freq = 399.78219838 [MHz]
Irr offset = 5 [ppm]
Clipped = FALSE
Mod return = 1
Scans = 20
Total scans = 20

F2 90 width = 11.75 [us]
F2 acq time = 1.04333312 [s]
F2 angle = 30 [deg]
F2 atm = 10 [dB]
F2 pulse = 3.91666667 [us]
Irr atm_dec = 24.95 [dB]
Irr atm_noe = 24.95 [dB]
Irr noise = WALTZ
Decoupling = TRUE
Initial wait = 1 [s]
Noe = TRUE
Noe time = 2 [s]
Recvr gain = 58
Relaxation delay = 2 [s]
Repetition time = 3.04333312 [s]
Temp_get = 22.6 [dC]



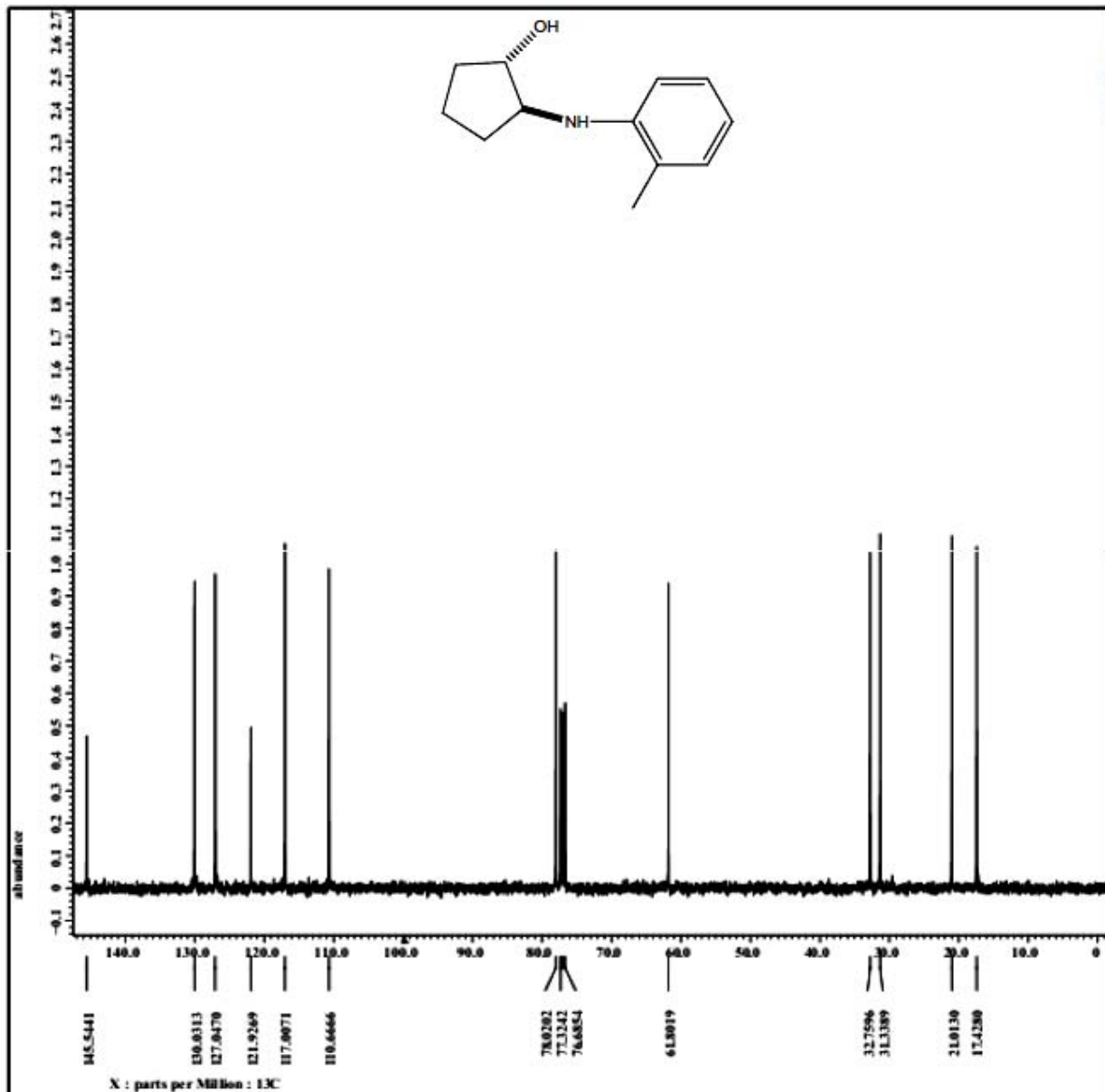
```

Filename      = SS 3-75 OTD PHOTON-4.
Author        = delta
Experiment    = single_pulse_ax2
Sample_id     = SS 3-75 OTD
Solvent       = CHLOROFORM-D
Creation_time = 8-MAY-2012 09:32:01
Revision_time = 29-JUL-2012 16:34:07
Current_Time  = 29-JUL-2012 16:34:13

Comment       = SS 3-75 OTD
Data_format   = 1D COMPLEX
Dir_size      = 13107
Dir_title     = 1H
Dir_units     = [ppm]
Dimensions    = X
Site          = KCM 400P
Spectrometer  = DELTA2_MMR

Field_strength = 9.389766[T] [400[MHz]]
X_acq_duration = 1.36577024[s]
X_domain       = 1H
X_freq         = 399.78219838[MHz]
X_offset       = 5[ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.73218757[Hz]
X_sweep        = 11.99616123[kHz]
Irr_domain     = 1H
Irr_freq       = 399.78219838[MHz]
Irr_offset     = 5[ppm]
Tri_domain     = 1H
Tri_freq       = 399.78219838[MHz]
Tri_offset     = 5[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 16
Total_scans    = 16

X_90_width    = 11.57[us]
X_acq_time     = 1.36577024[s]
X_angle        = 45[deg]
X_atn          = 5[db]
X_pulse        = 5.785[us]
Irr_mode       = Off
Tri_mode       = Off
Dnfs_preset   = FALSE
Init_wait     = 1[s]
Recvr_gain     = 24
Relaxation_delay = 4[s]
Repetition_time = 5.36577024[s]
Temp_get       = 22.5[dc]
  
```



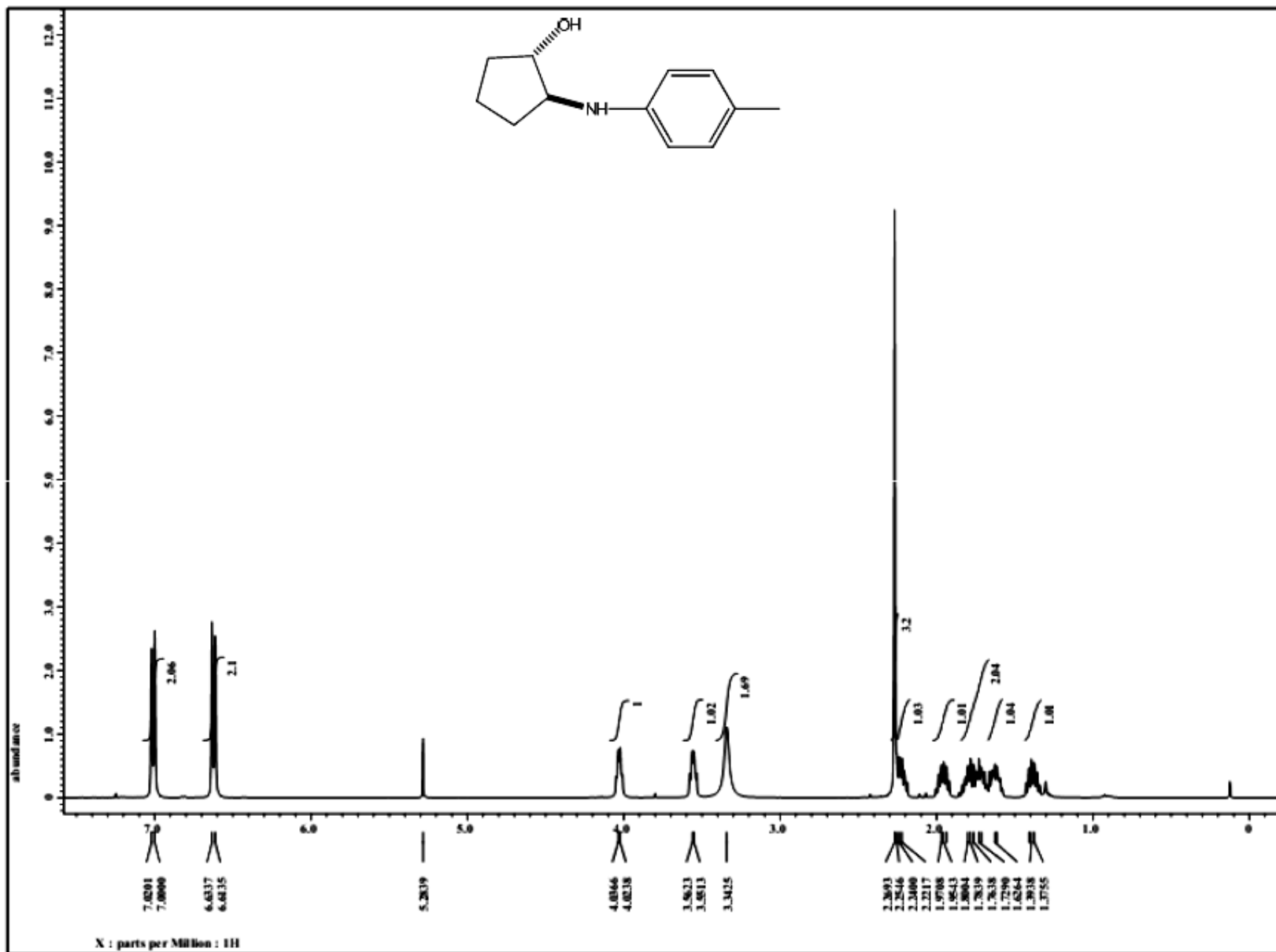
```

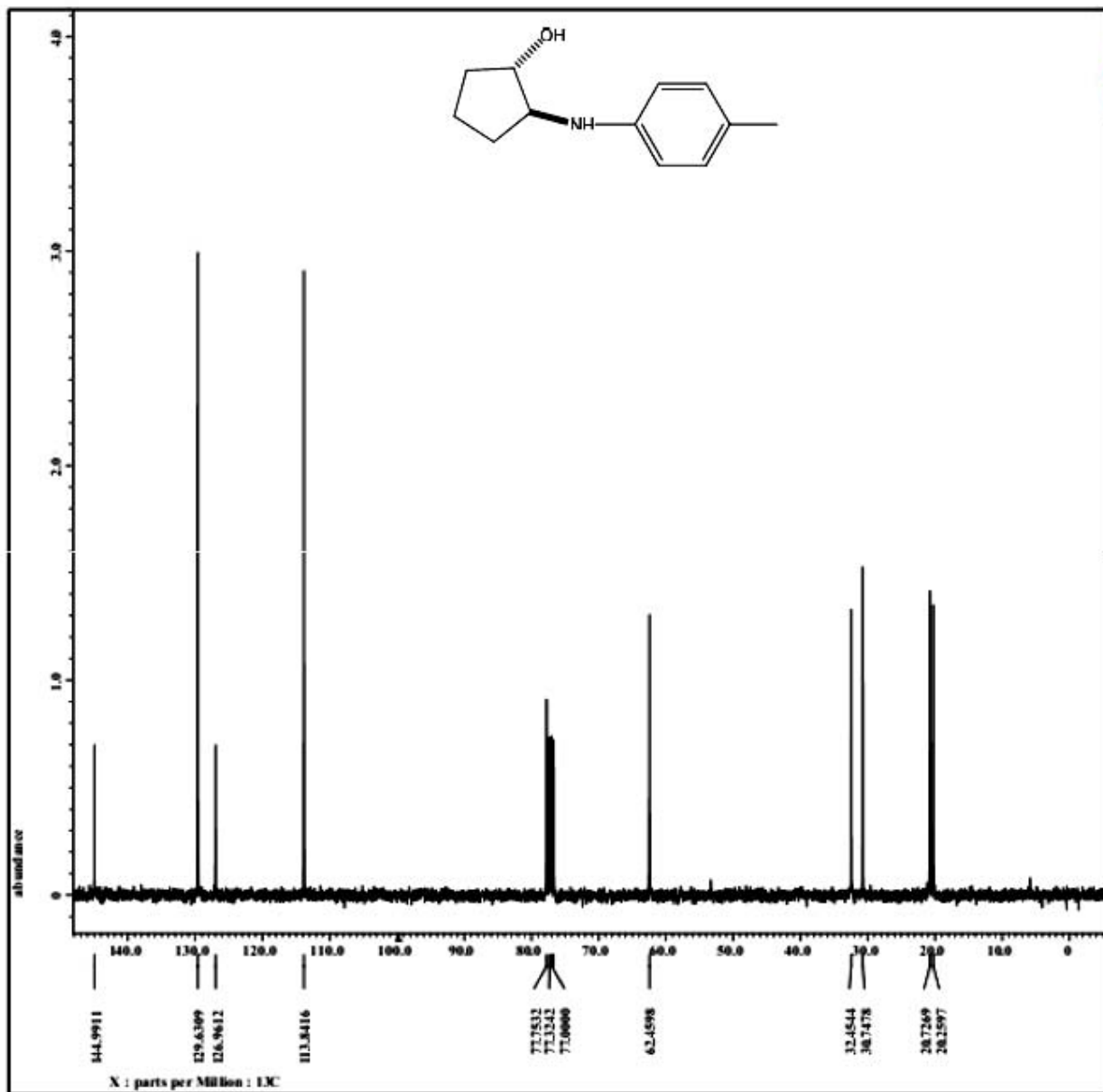
Filename      = DR_3-75_OTD_CARBON-3.
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = DR_3-75_OTD
Solvent      = CHLOROFORM-D
Creation_time = 11-MAY-2012 00:46:44
Revision_time = 11-MAY-2012 01:00:44
Current_Time = 29-JUL-2012 16:36:32

Comment      = DR_3-75_OTD
Data_format  = 1D_COMPLEX
Data_size    = 26214
Dir_title    = 13C
Dir_units    = [ppm]
Dimensions   = X
Site         = KXC 400P
Spectrometer = DELTA2_MMR

Field_strength = 9.389766[T] [400[MHz]]
X_acq_duration = 1.04333312[s]
X_domain      = 13C
X_freq        = 100.62530333 [MHz]
X_offset      = 100[ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 0.95846665 [Hz]
X_sweep       = 31.40703518 [kHz]
Irr_domain    = 1H
Irr_freq      = 399.78219838 [MHz]
Irr_offset    = 5[ppm]
Clipped      = FALSE
Proc_method   = -
Scale        = 24
Total_scans   = 24

X_90_width    = 11.75[us]
X_acq_time     = 1.04333312[s]
X_angle       = 30[deg]
X_atn         = 10[db]
X_pulse       = 3.91666667[us]
Irr_atn_dec   = 24.95[db]
Irr_atn_now   = 24.95[db]
Irr_noise     = WALTZ
Decoupling    = FMR
Initial_wait  = 1[s]
Nox_time      = FMR
Nox_time      = 2[s]
Recvr_gain    = 56
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get      = 22.9[dc]
  
```



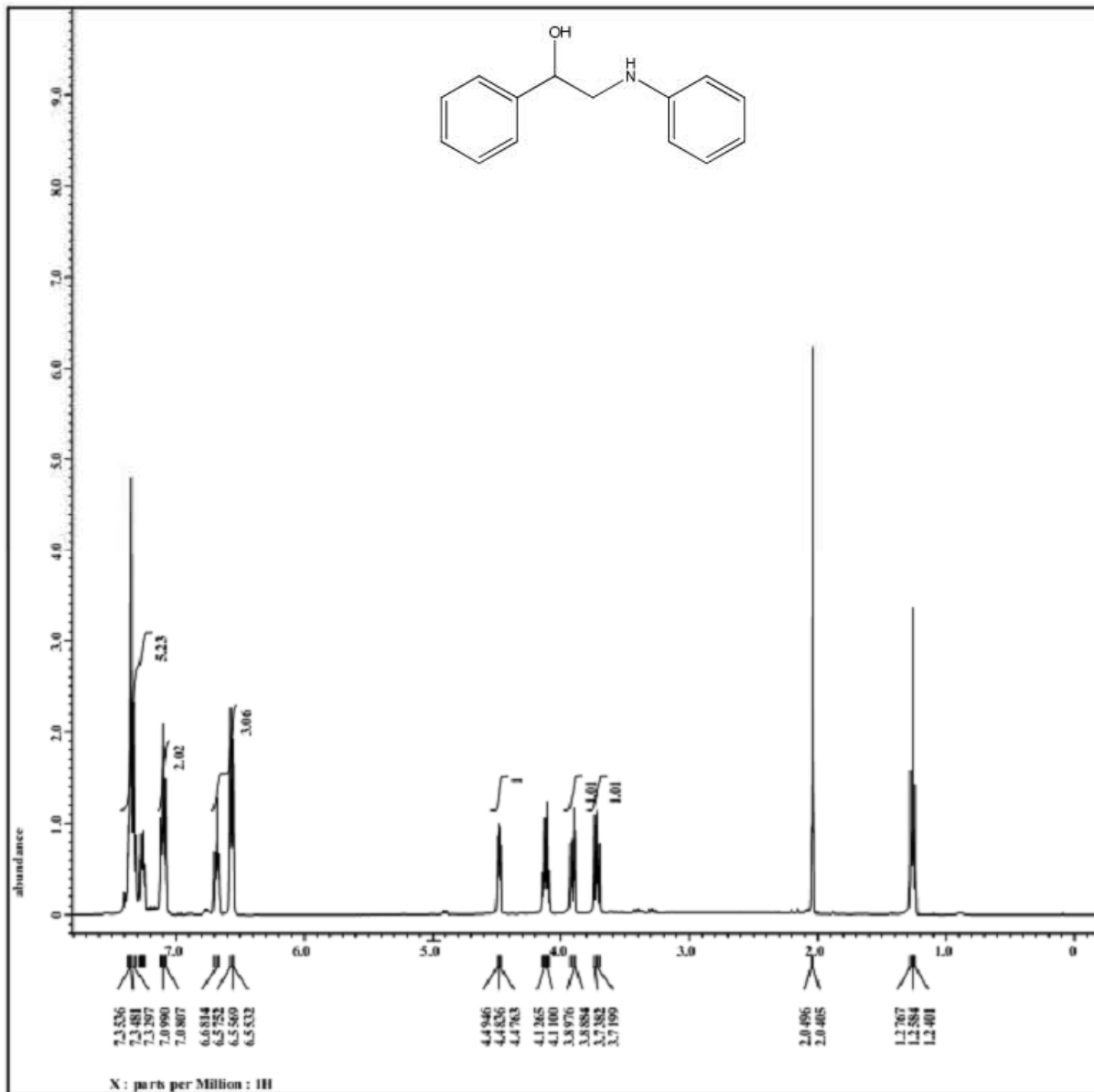
```

Filename      = SS 3-75_PT_CARDON-3.)
Author        = delta
Experiment    = single_pulse_dec
Sample_id     = SS 3-75 PT
Solvent       = CHLOROFORM-D
Creation_time  = 12-MAY-2012 16:39:52
Revision_time = 12-MAY-2012 16:53:46
Current_time  = 29-JUL-2012 16:35:01

Comment       = SS 3-75 PT
Data_format   = 1D_COMPLEX
Dir_size      = 26214
Dir_title     = 13C
Dir_units     = [ppm]
Dimensions    = X
Site          = RCK 400P
Spectrometer  = DELTA2_MMR

Field_strength = 9.389766[T] [400[MHz]]
X_acq_duration = 1.04333312[s]
X_domain       = 13C
X_freq         = 100.52530333 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 0.95846665 [Hz]
X_sweep        = 31.40703518 [kHz]
Irr_domain     = 1H
Irr_freq       = 399.78219838 [MHz]
Irr_offset     = 5 [ppm]
Clipped        = FALSE
wdw            = EM
ScaNs         = 14
Total_scans    = 14

X_90_width     = 11.75 [us]
X_acq_time     = 1.04333312 [s]
X_angle        = 30 [deg]
X_atn          = 10 [dB]
X_pulse        = 3.91666667 [us]
Irr_atn_dec    = 24.95 [dB]
Irr_atn_pow    = 24.95 [dB]
Irr_noise      = WALTZ
Decoupling     = TWHH
Initial_wait   = 1 [s]
Nox            = TWHH
Nox_time       = 2 [s]
RecVr_gain     = 58
Relaxation_delay = 2 [s]
Repetition_time = 3.04333312 [s]
Temp_get       = 22.9 [dC]
  
```



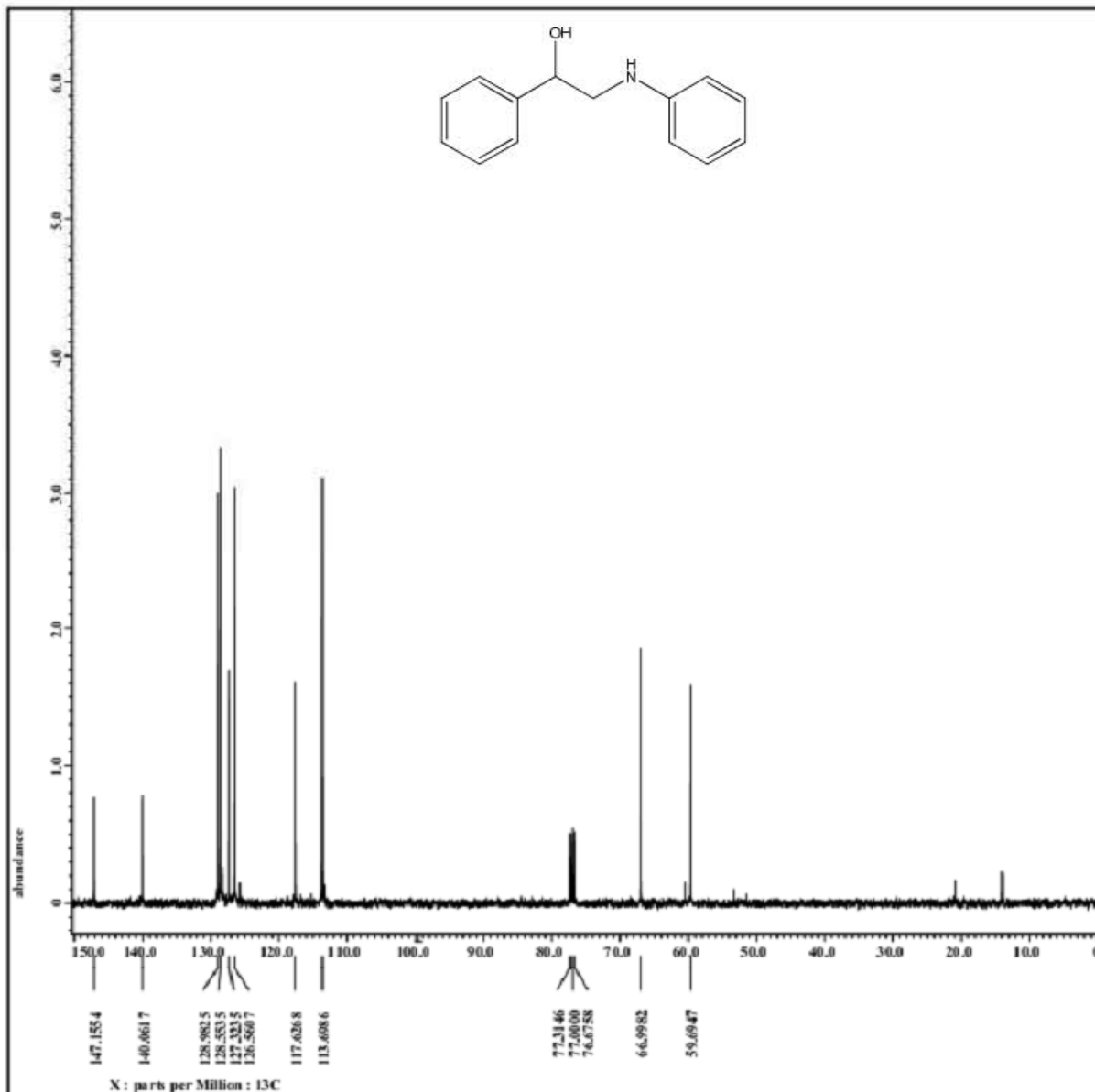
```

Filename      = SS 2-9ANL_PROTON-4.)d
Author       = delta
Experiment   = single pulse.ex2
Sample id    = SS 2-9ANL
Solvent      = CHLOROFORM-d
Creation time = 5-OCT-2011 12:04:30
Revision time = 6-AUG-2012 00:31:34
Current time  = 6-AUG-2012 00:34:18

Comment      = SS 2-9ANL
Data format  = 1D_COMPLEX
Bin_size     = 13107
Bin_title    = 1R
Bin_units    = [ppm]
Dimensions   = X
Site         = ECK 400P
Spectrometer = DELTA2 NMR

Field strength = 5.389766 [T] (400 [MHz])
X_acq_duration = 1.36577024 [s]
X_domain       = 1R
X_freq         = 399.78219838 [MHz]
X_offset       = 5 [ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.73218757 [Hz]
X_sweep        = 11.99616123 [kHz]
Irr_domain     = 1R
Irr_freq       = 399.78219838 [MHz]
Irr_offset     = 5 [ppm]
Tri_domain     = 1R
Tri_freq       = 399.78219838 [MHz]
Tri_offset     = 5 [ppm]
Clipped        = FALSE
Mod_return     = 1
SCANS         = 16
Total_scans    = 16

X_90_width    = 11.57 [us]
X_acq_time     = 1.36577024 [s]
X_angle        = 45 [deg]
X_atn          = 5 [dB]
X_pulse        = 5.785 [us]
Irr_mode       = OFF
Tri_mode       = OFF
Dante_presat   = FALSE
Initial_wait   = 1 [s]
Recvs_gain     = 18
Relaxation_delay = 4 [s]
Repetition_time = 5.36577024 [s]
Temp_get       = 22.4 [C]
  
```



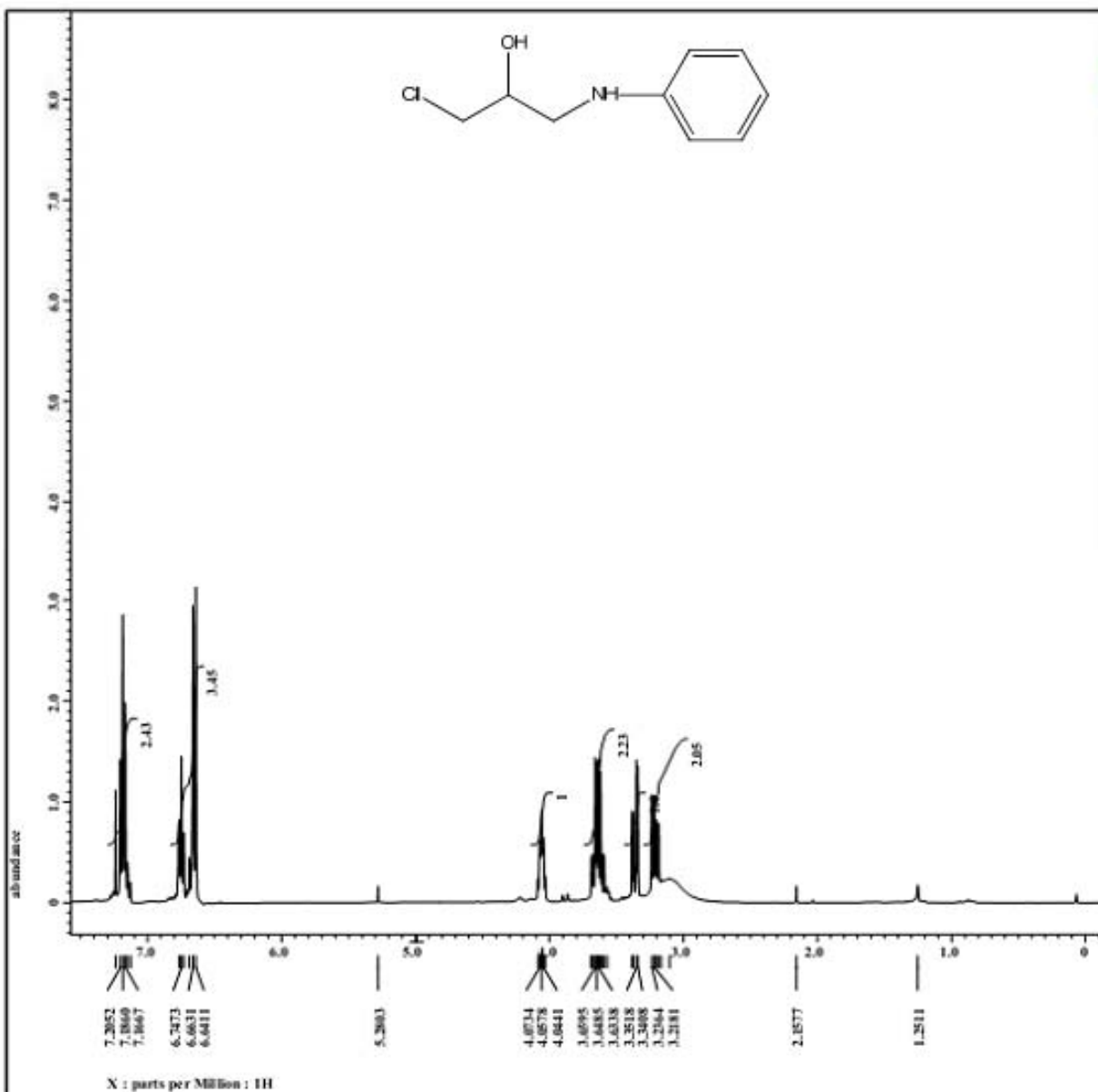
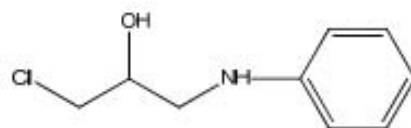
```

Filename      = FH-2-9ANL_CARBON-4.jd
Author       = delta
Experiment   = single pulse_dec
Sample_id    = FH-2-9ANL
Solvent      = CHLOROFORM-d
Creation_time = 15-OCT-2011 00:24:02
Revision_time = 6-AUG-2012 00:35:07
Current_time  = 6-AUG-2012 00:35:23

Comment      = FH-2-9ANL
Data_format  = 1D COMPLEX
Bin_size     = 26214
Bin_title    = 13C
Bin_units    = [ppm]
Dimensions   = X
Site         = ECK 400P
Spectrometer = DELTA2 NMR

Field_strength = 9.399766[T] (400[MHz])
X_acq_duration = 1.04333312[s]
X_domain       = 13C
X_freq         = 100.52533333[MHz]
X_offset      = 100[ppm]
X_points       = 32768
X_prescans    = 4
X_resolution  = 0.93846665[Hz]
X_sweep       = 31.40703518[kHz]
Irr_domain    = 1H
Irr_freq      = 399.78219838[MHz]
Irr_offset    = 5[ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 14
Total_scans   = 14

X_90_width    = 11.75[us]
X_acq_time    = 1.04333312[s]
X_angle       = 30[deg]
X_atn         = 10[db]
X_pulse       = 3.91666667[us]
Irr_atn_dec   = 24.95[db]
Irr_atn_noe   = 24.95[db]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1[s]
Noe           = TRUE
Noe_time      = 2[s]
Recvr_gain    = 58
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get      = 22.4[degC]
  
```



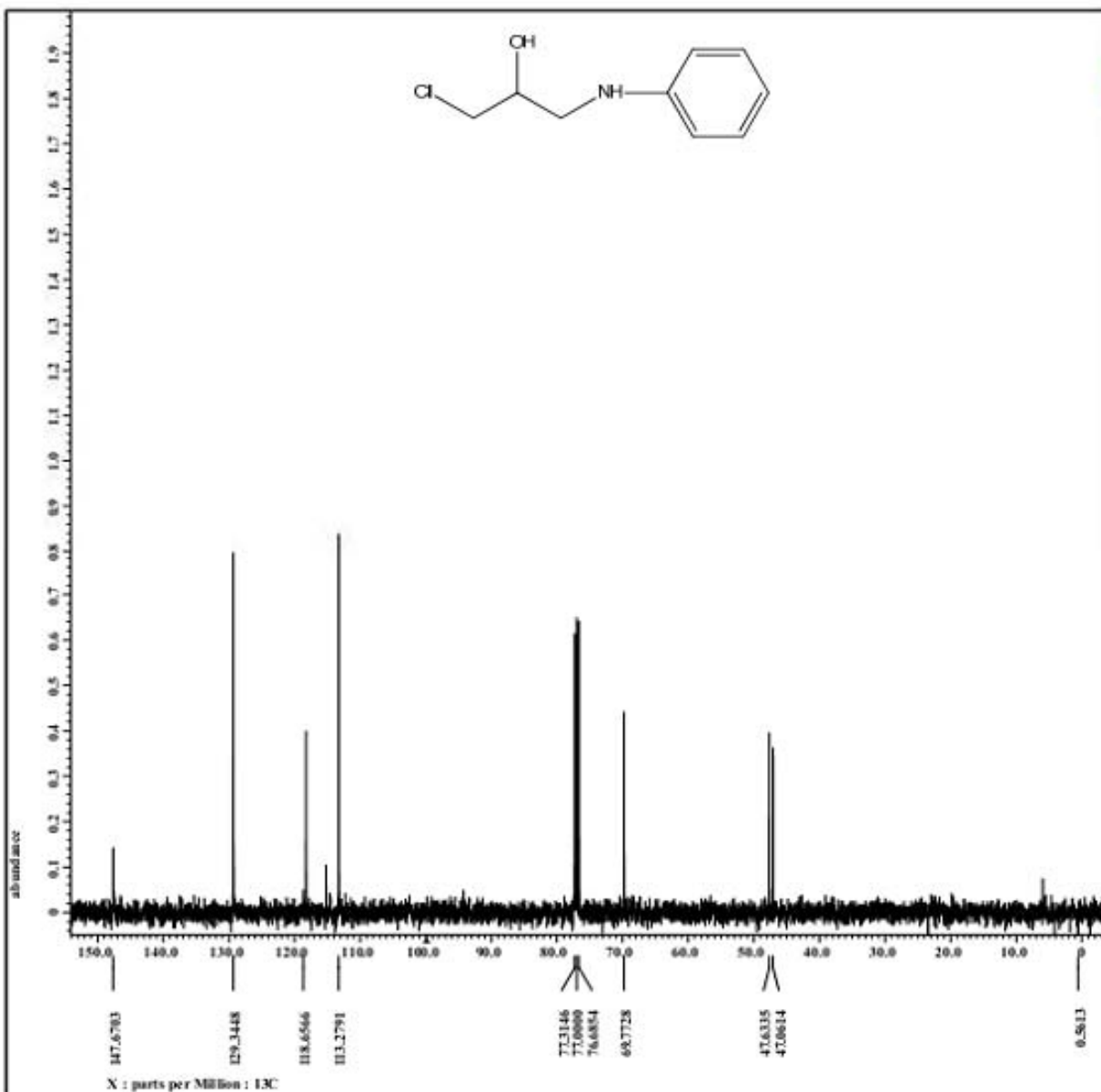
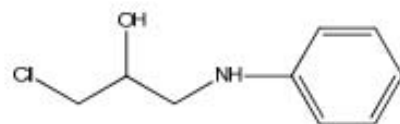
```

Filename      = SS 3-SEPI_PROTON-4.jd
Author        = delta
Experiment    = single pulse.ex2
Sample id     = SS 3-SEPI
Solvent       = CDCl3/CF3COOH-D
Creation time  = 26-OCT-2011 14:42:42
Revision time = 29-JUL-2012 16:04:45
Current Time  = 29-JUL-2012 16:05:05

Comment       = SS 3-SEPI
Data format   = 1D COMPLEX
Dir_size      = 13107
Dir_title     = 1H
Dir_units     = [ppm]
Dimensions    = X
Site          = HXK 400P
Spectrometer  = DELTA2 300B

Field strength = 9.389766[T] (400[Mhz])
X_acq_duration = 1.36577024[s]
X_domain       = 1H
X_freq         = 399.78219838 [Mhz]
X_offset       = 5 [ppm]
X_points       = 14384
X_resolution   = 0.73218757 [Hz]
X_sweep        = 11.99616123 [kHz]
irf_domain     = 1H
irf_freq       = 399.78219838 [Mhz]
irf_offset     = 5 [ppm]
Tri_domain     = 1H
Tri_freq       = 299.78219838 [Mhz]
Tri_offset     = 5 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scale          = 16
Total_scans    = 16

X_90_width     = 11.57[us]
X_acq_time     = 1.36577024[s]
X_angle        = 45[deg]
X_atn          = 5[dB]
X_pulse        = 5.785[us]
irf_mode       = off
Tri_mode       = off
Date_preset    = FALSE
Initial_wait   = 1[s]
Recvr_gain     = 35
Relaxation_delay = 4[s]
Repetition_time = 5.36577024[s]
Temp_get       = 21.4 [dC]
  
```



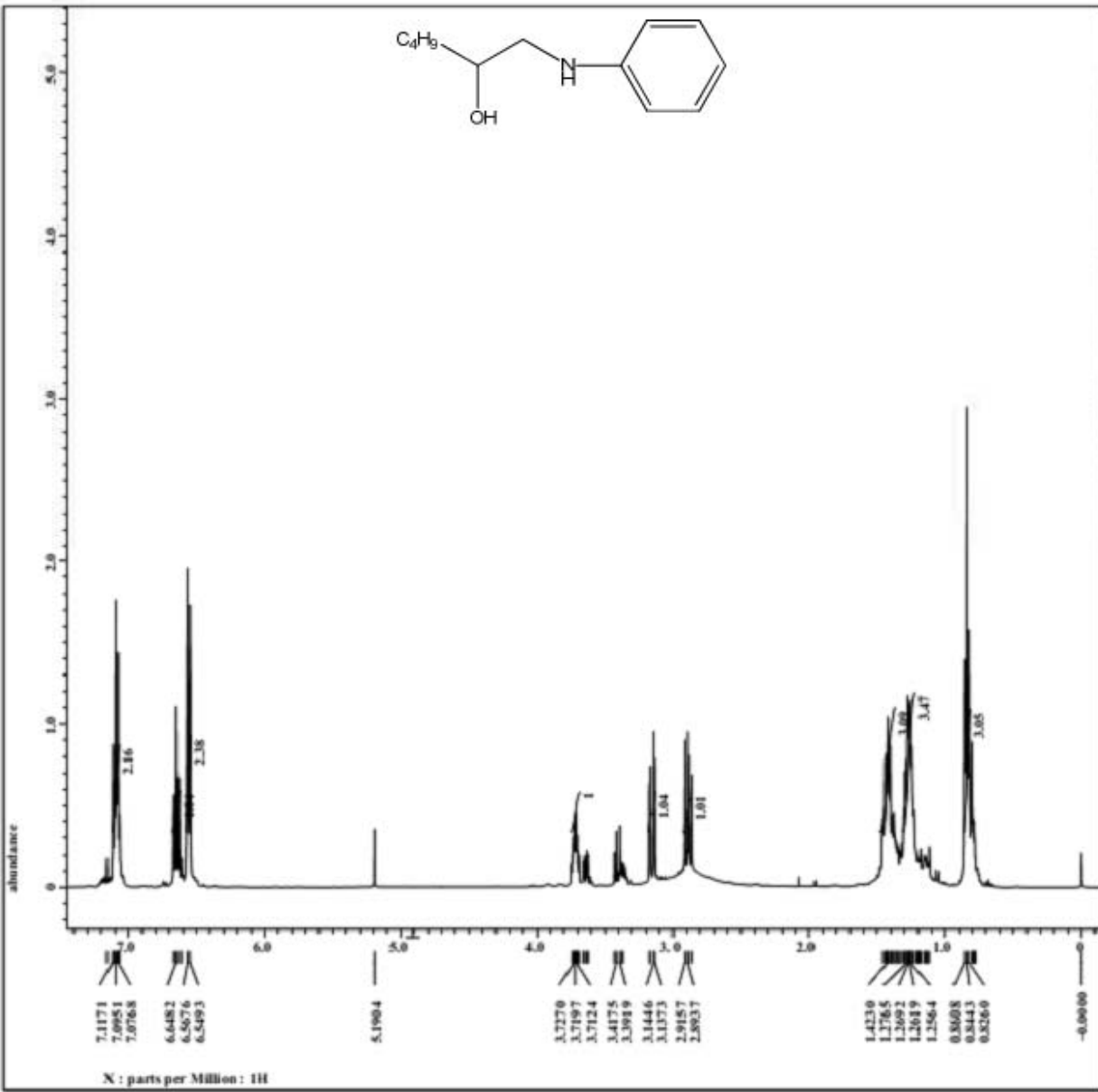
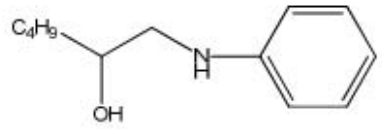
```

Filename      = 55_3-8-EP1_CANDON-4.j
Author        = delta
Experiment    = single_pulse_dec
Sample id     = 55_3-8-EP1
Solvent       = CHLOROFORM-D
Creation time  = 27-OCT-2011 18:16:11
Revision time = 29-JUL-2012 16:07:19
Current Time  = 29-JUL-2012 16:07:40

Comment       = 55_3-8-EP1
Data format   = 1D COMPLEX
Dir_size      = 26214
Dir_title     = 13C
Dir_units     = [ppm]
Dimensions    = X
Site          = KCX 400P
Spectrometer  = DELTA2_MK2

Field strength = 9.389766[?] (400[Mhz])
X_acq_duration = 1.0433312[s]
X_delay        = 13C
X_delay_time   = 100.52530333 [Mhz]
X_offset       = 100[ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 0.95846665[Hz]
X_sweep        = 31.40703518 [kHz]
f1r_domain     = 1H
f1r_freq       = 399.78219838 [Mhz]
f1r_offset     = 5[ppm]
Clipped        = FALSE
Max_sweep     = 1
Solve         = 14
Total_scans    = 14

X_90_width     = 11.75[us]
X_acq_time      = 1.0433312[s]
X_angle         = 30[deg]
X_atn           = 10[db]
X_pulse         = 3.91666667[us]
f1r_atn_dec     = 24.95[db]
f1r_atn_noise   = 24.95[db]
f1r_noise       = NONE
Decoupling      = NONE
Initial_wait    = 1[s]
Noise           = NONE
Noise time     = 2[s]
Recvr_gain      = 58
Relaxation_delay = 2[s]
Repetition_time = 1.0433312[s]
Temp_get        = 22.6 [dC]
  
```



```

Filename      = SS EHAM_PROTON-4.jdf
Author       = delta
Experiment   = single_pulse.ex2
Sample_id    = SS EHAM
Solvent      = CHLOROFORM-D
Creation_time = 9-AUG-2012 14:39:36
Revision_time = 11-AUG-2012 09:14:12
Current_time  = 11-AUG-2012 09:37:38

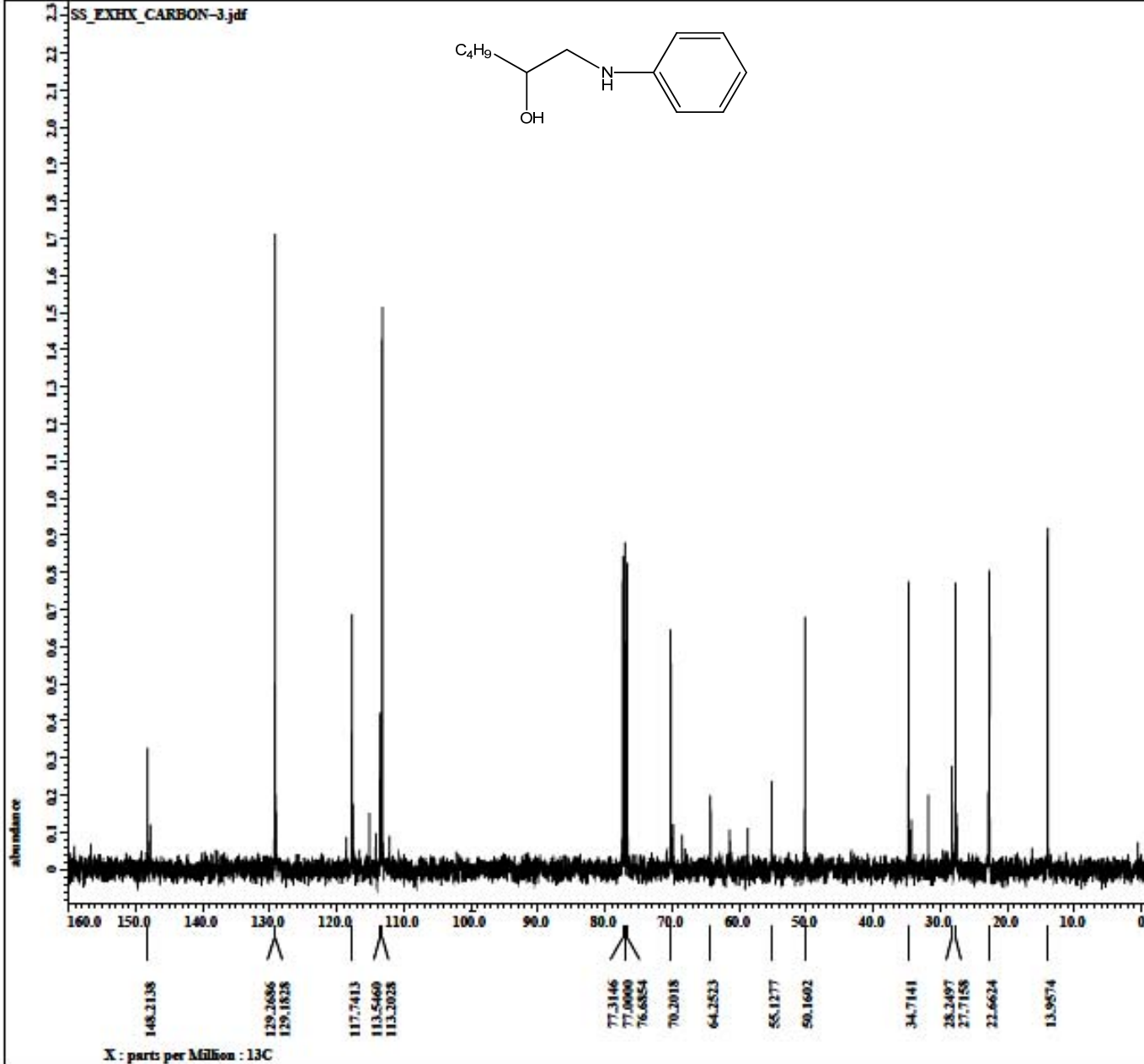
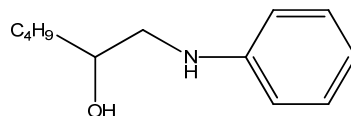
Comment      = SS EHAM
Data_format  = 1D COMPLEX
Dim_size     = 13107
Dim_title    = 1H
Dim_units    = [ppm]
Dimensions   = K
Site         = MZX 400P
Spectrometer = DELTA2_800

Field_strength = 9.389766[T] (400 [MHz])
X_acq_duration = 1.36577024[s]
X_domain       = 1H
X_freq         = 399.78219838 [MHz]
X_offset       = 5 [ppm]
X_points       = 16384
X_procscans   = 1
X_resolution   = 0.73218757 [Hz]
X_sweep        = 11.99616123 [kHz]
IR_domain     = 1H
IR_freq        = 399.78219838 [MHz]
IR_offset      = 5 [ppm]
TR1_domain     = 1H
TR1_freq       = 399.78219838 [MHz]
TR1_offset     = 5 [ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 16
Total_scans    = 16

X_90_width    = 11.57 [us]
X_acq_time    = 1.36577024 [s]
X_angle       = 45 [deg]
X_atn         = 5 [dB]
X_pulse       = 5.785 [us]
IRF_mode      = Off
TR1_mode      = Off
Data_preset   = FALSE
Initial_wait  = 1 [s]
Recvr_gain    = 26
Relaxation_delay = 4 [s]
Repetition_time = 5.36577024 [s]
Temp_get      = 22.8 [dC]
  
```

X : parts per Million : 1H

SS_EXHX_CARBON-3.jdr

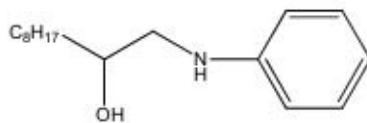


Filename = SS_EXHX_CARBON-3.jdr
Author = delta
Experiment = single pulse dec
Sample id = SS_EXHX
Solvent = CHLOROFORM-D
Creation time = 3-JAN-2012 23:03:00
Revision time = 3-JAN-2012 23:05:34
Current time = 11-JAN-2012 16:56:40

Comment = SS_EXHX
Data format = 1D COMPLEX
Dim size = 26214
Dim title = 13C
Dim units = [ppm]
Dimensions = 1
Site = RCX 400P
Spectrometer = DELTA2_NMR

Field strength = 9.389766 [T] (400 [MHz])
X acq duration = 1.04333312 [s]
X domain = 13C
X freq = 100.52530333 [MHz]
X offset = 100 [ppm]
X points = 32768
X prescans = 4
X resolution = 0.95846665 [Hz]
X sweep = 31.40703518 [kHz]
Irr domain = 1H
Irr freq = 399.78219838 [MHz]
Irr offset = 5 [ppm]
Clipped = FALSE
Mod return = 1
Scans = 14
Total scans = 14

X 90 width = 11.75 [us]
X acq time = 1.04333312 [s]
X angle = 30 [deg]
X atn = 10 [dB]
X pulse = 3.91666667 [us]
Irr atn dec = 24.95 [dB]
Irr atn noe = 24.95 [dB]
Irr noise = WALTZ
Decoupling = TRUR
Initial wait = 1 [s]
Noe = TRUE
Noe time = 2 [s]
Recvr gain = 60
Relaxation delay = 2 [s]
Repetition time = 3.04333312 [s]
Temp get = 22.2 [dC]

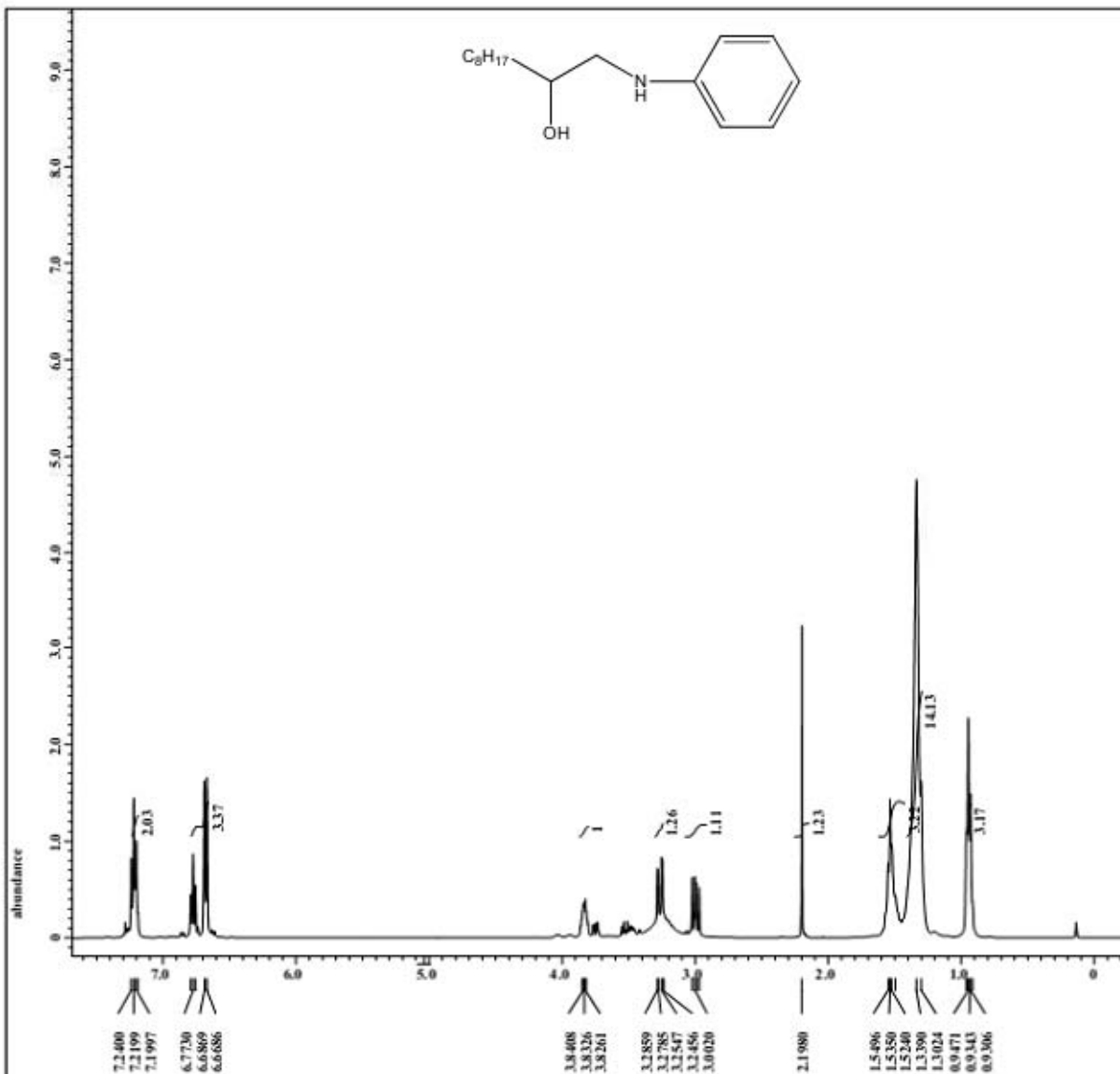


Filename = SS_ED-H_PROTON-4.jdf
 Author = delta
 Experiment = single_pulse.ex2
 Sample_id = SS_ED-H
 Solvent = CHLOROFORM-D
 Creation_time = 3-AUG-2012 17:13:05
 Revision_time = 11-AUG-2012 10:03:08
 Current_time = 11-AUG-2012 10:03:24

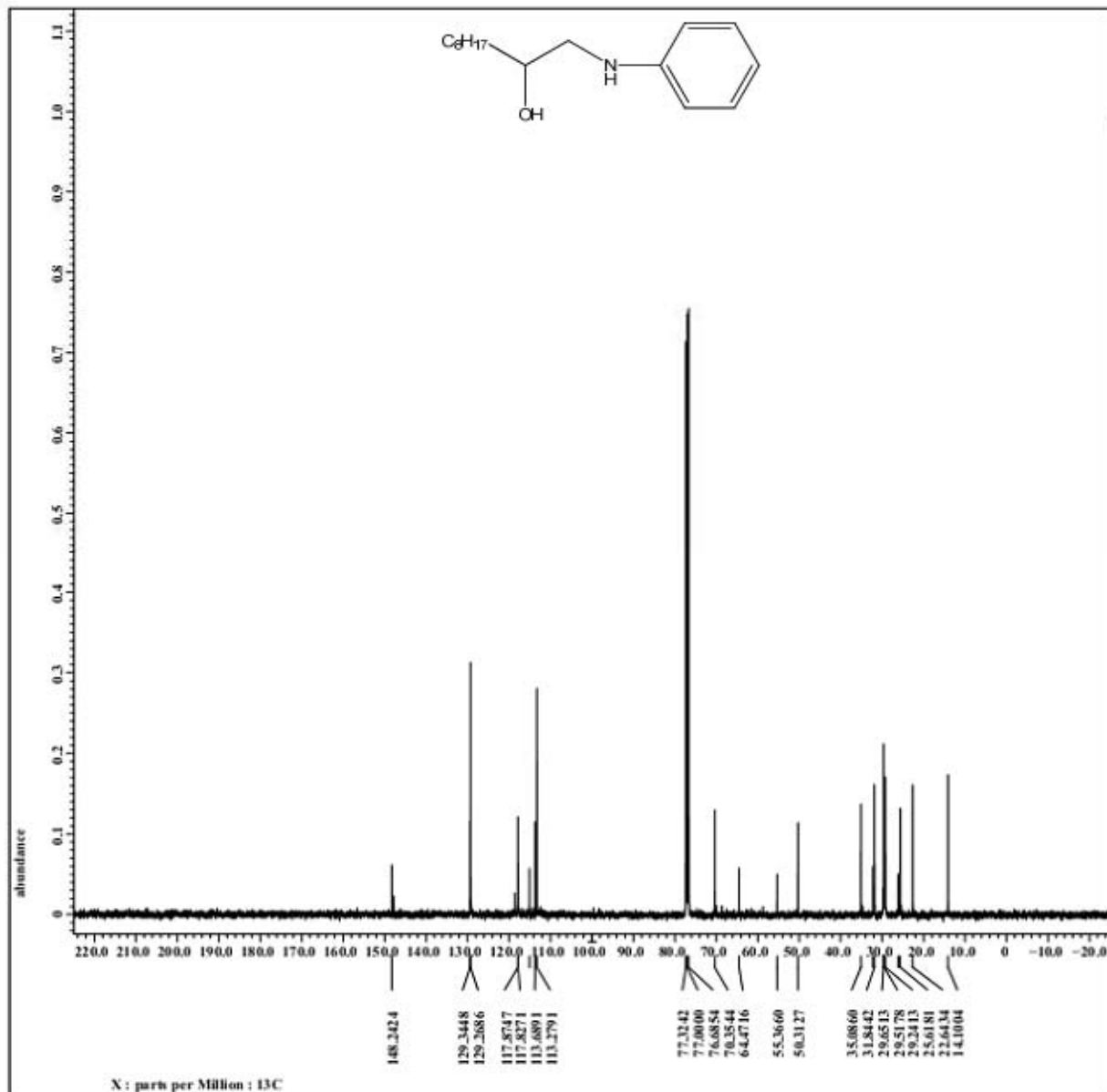
Comment = SS ED-H
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECX 400P
 Spectrometer = DELTA2_SMR

Field_strength = 9.389766[T] (400[MHz])
 X_acq_duration = 1.36577024[s]
 X_domain = 1H
 X_freq = 399.78219838[MHz]
 X_offset = 5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.73218757[Hz]
 X_sweep = 11.99616123[kHz]
 Iir_domain = 1H
 Iir_freq = 399.78219838[MHz]
 Iir_offset = 5[ppm]
 Tri_domain = 1H
 Tri_freq = 399.78219838[MHz]
 Tri_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 16
 Total_scans = 16

X_90_width = 11.57[us]
 X_acq_time = 1.36577024[s]
 X_angle = 45[deg]
 X_atn = 5[dB]
 X_pulse = 5.785[us]
 Iir_mode = Off
 Tri_mode = Off
 Dante_preset = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 22
 Relaxation_delay = 4[s]
 Repetition_time = 5.36577024[s]
 Temp_get = 19.2[C]



X : parts per Million : 1H

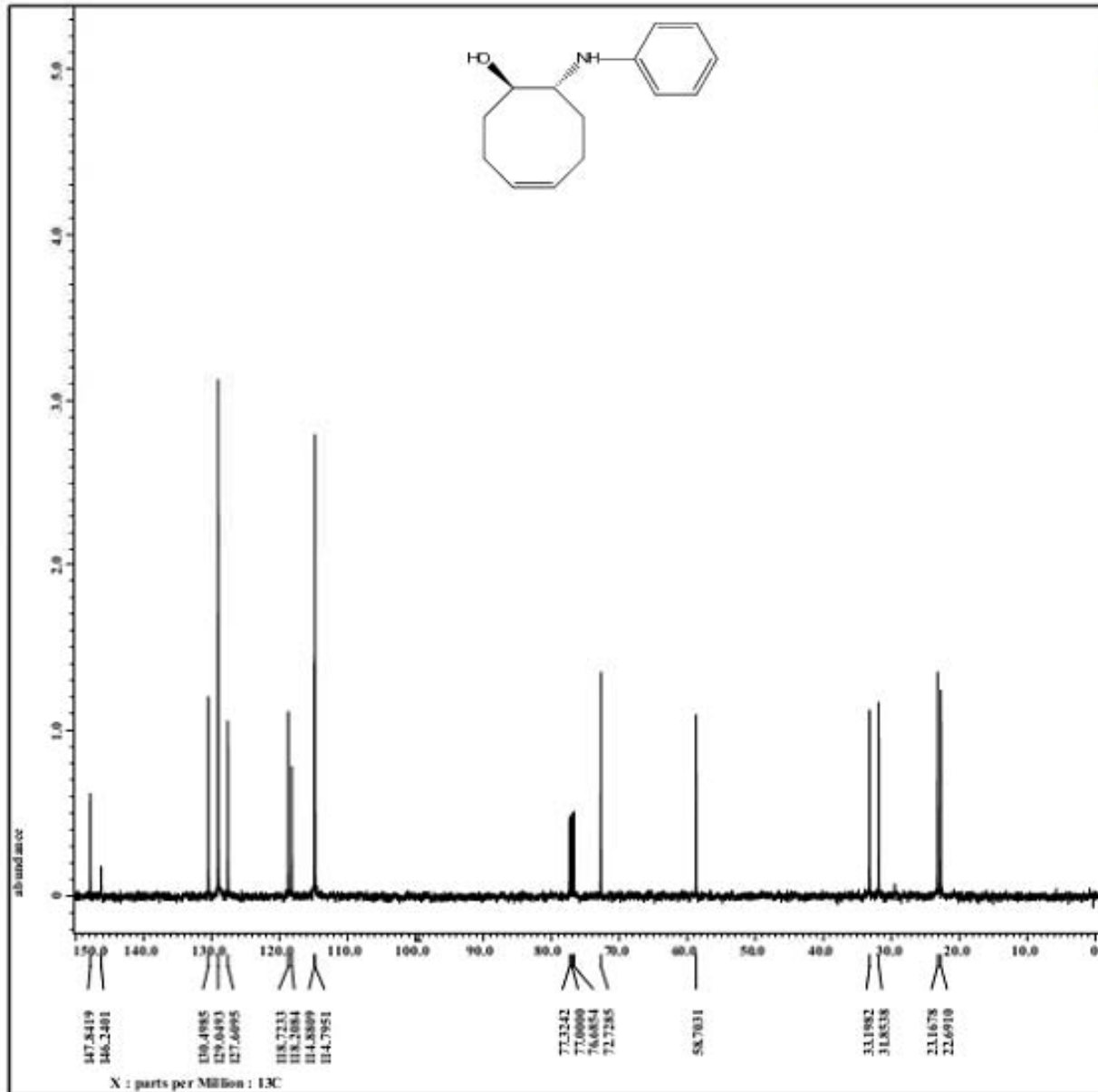
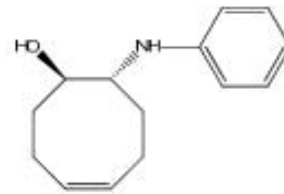


Filename = SS_EXDC_CARBON-3.jdf
 Author = delta
 Experiment = single_pulse_dec
 Sample_id = SS_EXDC
 Solvent = CHLOROFORM-D
 Creation_time = 3-JAN-2012 22:57:24
 Revision_time = 3-JAN-2012 22:59:58
 Current_time = 11-AGO-2012 10:54:12

Comment = SS_EXDC
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = X
 Site = ECK 400P
 Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[Mhz])
 X_acq_duration = 1.04333312[s]
 X_domain = 13C
 X_freq = 100.52530333[Mhz]
 X_offset = 100[ppm]
 X_points = 32768
 X_prescans = 4
 X_resolution = 0.95846665[Hz]
 X_sweep = 31.40703518[kHz]
 Irr_domain = 1H
 Irr_freq = 399.78219838[Mhz]
 Irr_offset = 5[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 750
 Total_scans = 750

X_90_width = 11.75[us]
 X_acq_time = 1.04333312[s]
 X_angle = 30[deg]
 X_atn = 10[db]
 X_pulse = 3.91666667[us]
 Irr_atn_dec = 24.95[db]
 Irr_atn_noe = 24.95[db]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 1[s]
 Noe = TRUE
 Noe_time = 2[s]
 Recvr_gain = 60
 Relaxation_delay = 2[s]
 Repetition_time = 3.04333312[s]
 Temp_get = 21.9[dc]

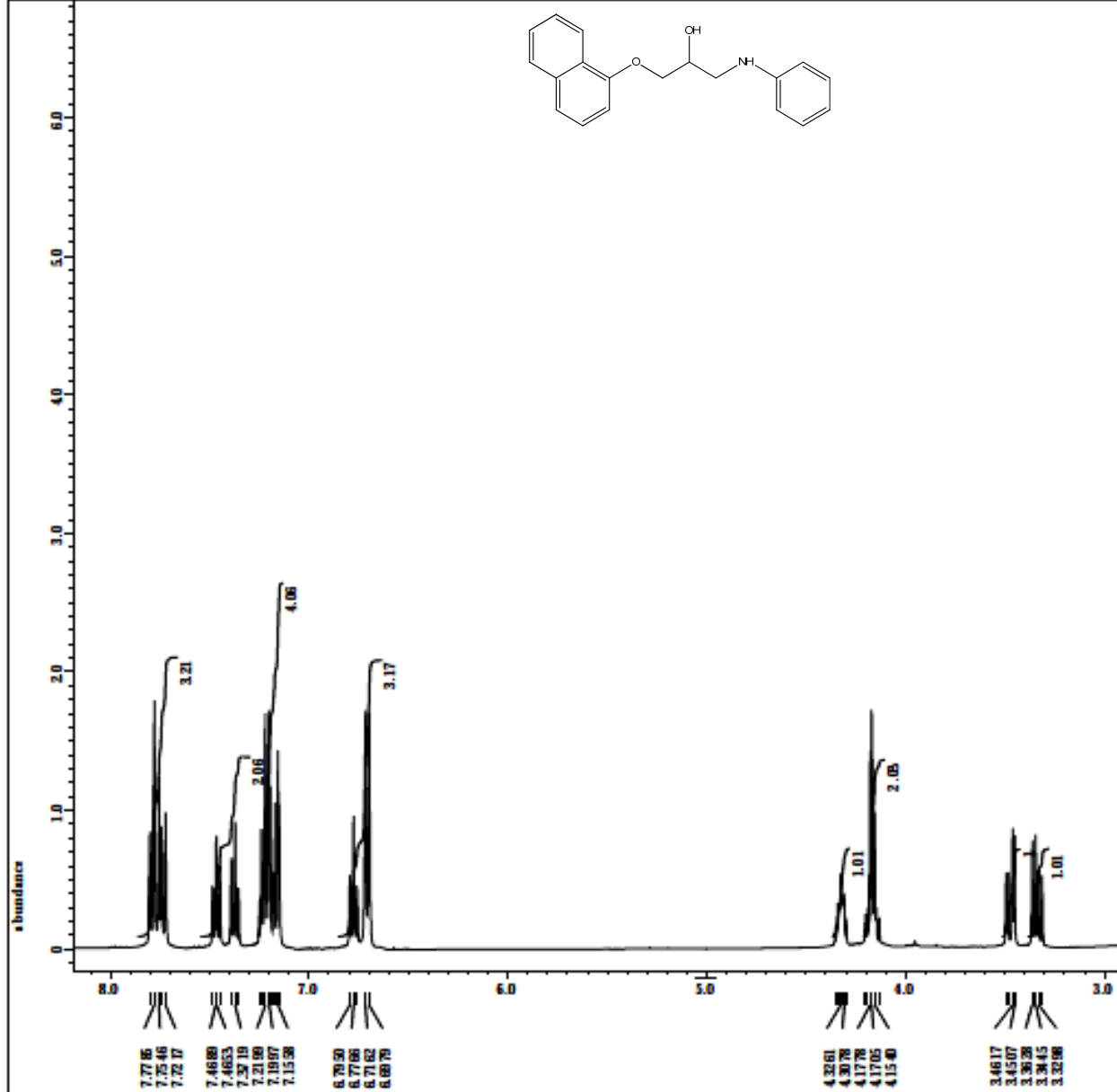
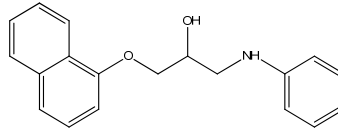


```
Filename      = FR_3-12C_CARRION-3.jdf
Author       = delta
Experiment   = single_pulse_dec
Sample id    = FR_3-12C
Solvent      = CHLOROFORM-D
Creation_time = 5-JAN-2012 15:24:03
Revision_time = 5-JAN-2012 15:26:36
Current_time = 29-JUL-2012 17:04:35

Comment      = FR_3-12C
Data_format  = 1D_COMPLEX
Dir_size     = 26214
Dir_title    = 12C
Dir_units    = [ppm]
Dimensions   = X
Site         = SUX 400P
Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[Mhz])
X_acq_duration = 1.04333312[s]
X_domain       = 12C
X_freq         = 100.52530333[Mhz]
X_offset      = 100[ppm]
X_points       = 32768
X_prescans    = 4
X_resolution  = 0.95846665[Hz]
X_sweep        = 31.40703518[kHz]
IFR_domain     = 1H
IFR_freq       = 399.78219838[Mhz]
IFR_offset     = 5[ppm]
Clipped        = FALSE
Mod_ex_time    = 1
Scans          = 14
Total_scans    = 14

X_90_width    = 11.75[us]
X_acq_time     = 1.04333312[s]
X_angle        = 30[deg]
X_atn          = 10[db]
X_pulse        = 3.91666667[us]
IFR_atn_dec    = 24.95[db]
IFR_atn_pow    = 24.95[db]
IFR_noise      = WALTZ
Decoupling     = THM
Initial_wait   = 1[s]
Noe            = THM
Noe_time       = 2[s]
RecVr_gain     = 58
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get       = 22.2[degC]
```



X : parts per Million : 1H

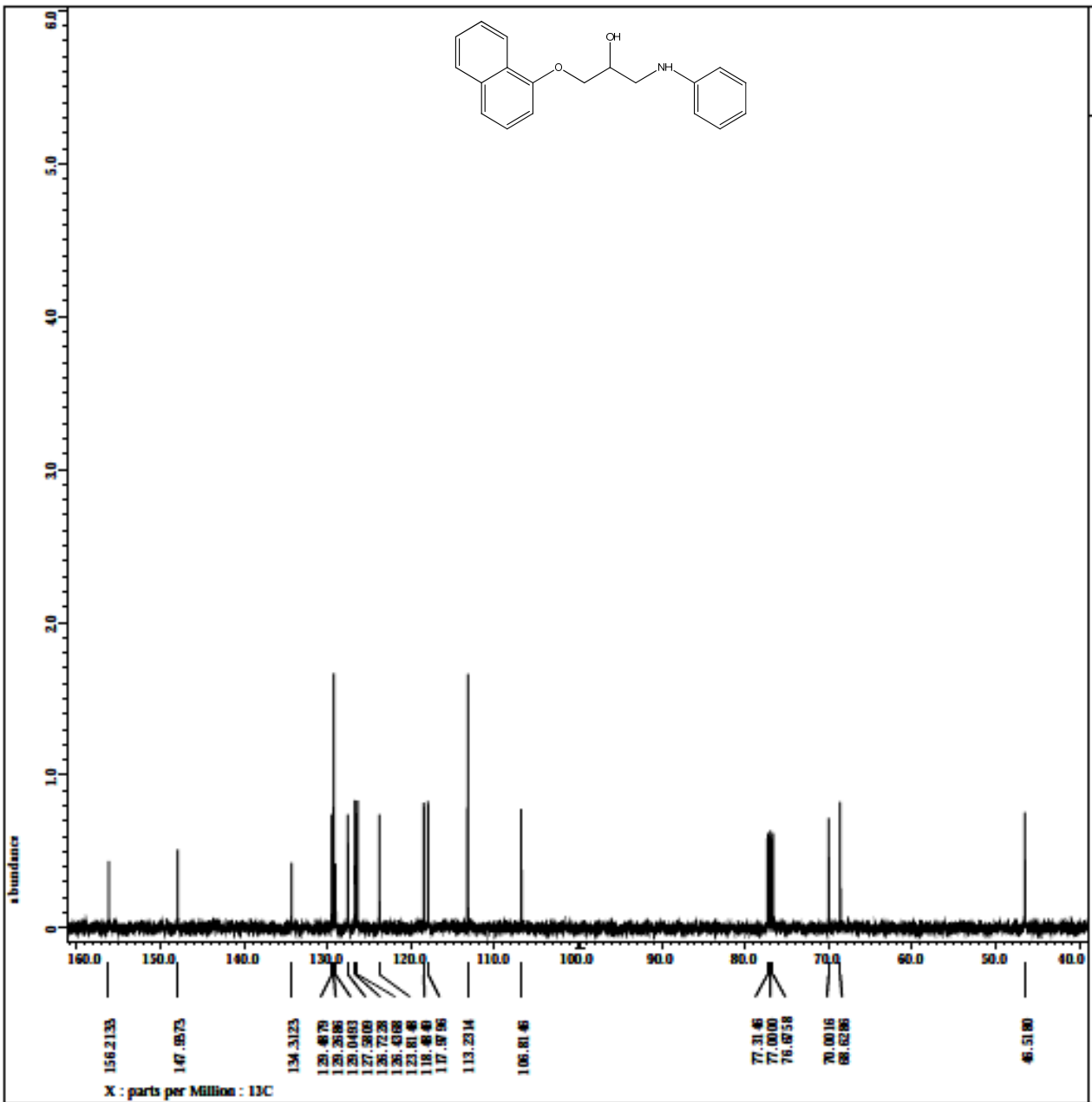
```

File name      = FK 4-16A_PROTON-4.jdt
Author         = Delta
Experiment     = single pulse.scf
Sample id      = FK 4-16A
Solvent        = CHLOROFORM-D
Creation time   = 25-AUG-2012 16:36:29
Revision time  = 17-APR-2014 12:47:32
Current time   = 17-APR-2014 12:47:59

Comment       = FK 4-16A
Data format    = 1D COMPLEX
Data size      = 13107
Data title     = 1H
Data units     = [ppm]
Dimensions     = 1
Site           = KCI 400P
Spectrometer   = DELTA2_NMR

Field strength = 9.389766 [T] (400 [MHz])
F1 acq duration = 1.36377024 [s]
F1 domain      = 1H
F1 freq         = 399.78219838 [MHz]
F1 offset      = 5 [ppm]
F1 points      = 16384
F1 prescans    = 1
F1 resolution  = 0.73218757 [Hz]
F1 sweep       = 11.99416123 [kHz]
Irr domain     = 1H
Irr freq       = 399.78219838 [MHz]
Irr offset     = 5 [ppm]
Tri domain     = 1H
Tri freq       = 399.78219838 [MHz]
Tri offset     = 5 [ppm]
Clipped        = FALSE
Mod return     = 1
Scale          = 16
Total scans    = 16

F2 90 width    = 11.57 [us]
F2 acq time    = 1.36377024 [s]
F2 angle       = 45 [deg]
F2 atm         = 5 [dB]
F2 pulse       = 5.785 [us]
Irr mode       = Off
Tri mode       = Off
Delta preset   = FALSE
Initial wait   = 1 [s]
Recvr gain     = 30
Relaxation delay = 4 [s]
Repetition time = 5.36377024 [s]
Temp get       = 21.1 [dC]
  
```



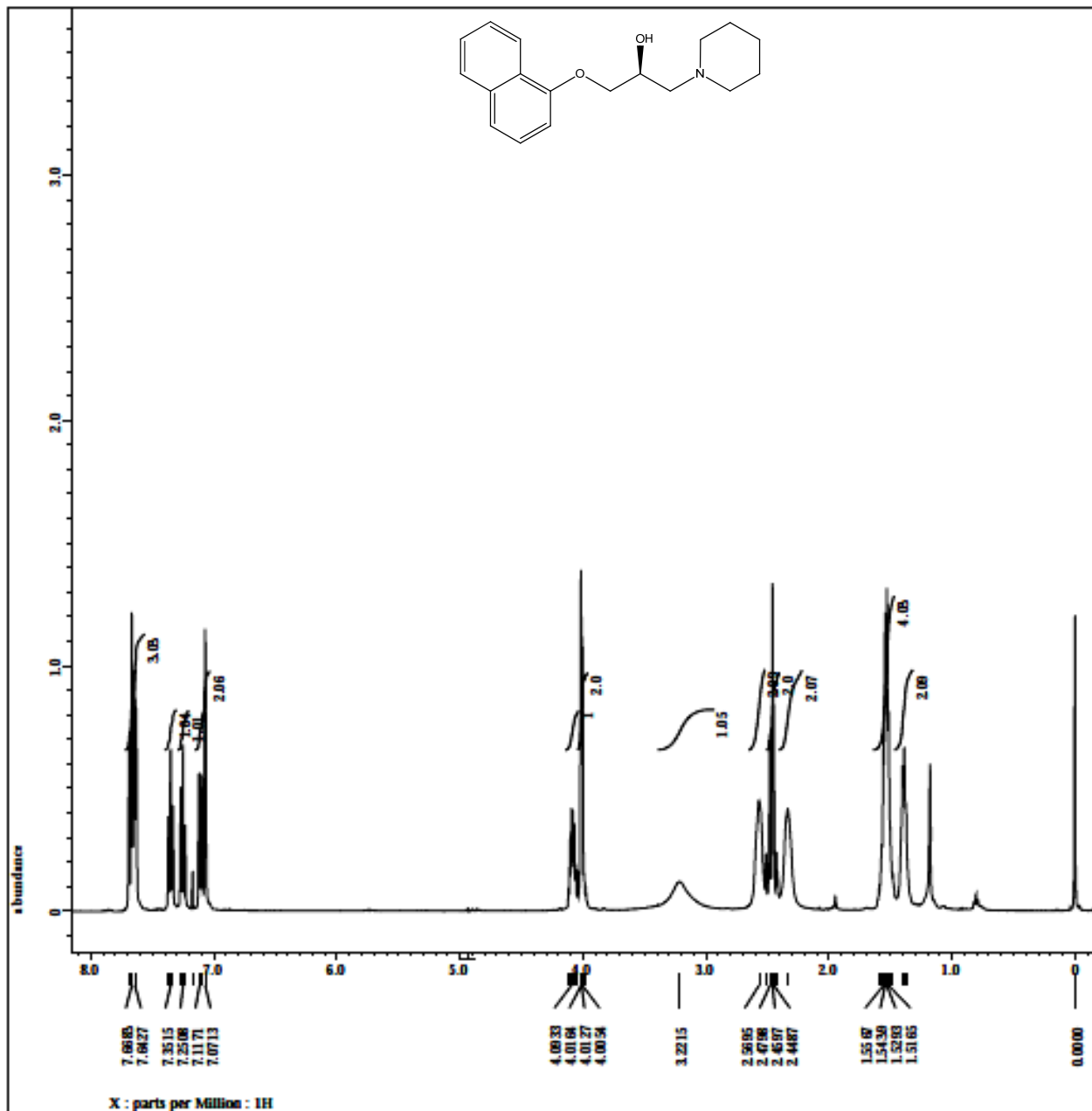
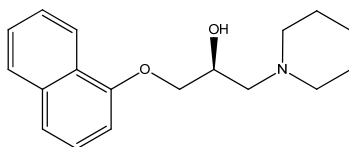
```

File Name      = FX 4-16C_CARBON-3.jdf
Author        = delta
Experiment    = single pulse dec
Sample id     = FX 4-16C
Solvent       = CDCl3/DMSO-D
Creation time = 26-AUG-2012 01:43:01
Revision time = 26-AUG-2012 02:02:54
Current time  = 17-APR-2014 12:48:41

Comment       = FX 4-16C
Data format   = 1D COMPLEX
Dir size      = 26214
Dir title     = 13C
Dir units     = [ppm]
Dimensions    = 2
Site         = ECI 400P
Spectrometer  = DELTA2_HMR

Field strength = 9.389766 [T] (400 [MHz])
F1 acq duration = 1.04333312 [s]
F1 domain      = 13C
F1 freq        = 100.62530333 [MHz]
F1 offset      = 100 [ppm]
F1 points      = 32763
F1 prescans    = 4
F1 resolution  = 0.95846665 [Hz]
F1 sweep       = 31.40703518 [kHz]
Irr domain     = 1H
Irr freq       = 399.78219838 [MHz]
Irr offset     = 5 [ppm]
Clipped       = FALSE
Mod return     = 1
Scale         = 14
Total scans    = 14

F2 90 width    = 11.75 [us]
F2 acq time    = 1.04333312 [s]
F2 angle       = 30 [deg]
F2 atm         = 10 [dB]
F2 pulse       = 3.91666667 [us]
Irr atm dec    = 24.93 [dB]
Irr atm noc   = 24.93 [dB]
Irr noise      = WALTZ
Decoupling     = TRUE
Initial wait   = 1 [s]
Noe            = TRUE
Noe time       = 2 [s]
Recvr gain     = 58
Relaxation delay = 2 [s]
Repetition time = 3.04333312 [s]
Temp get       = 21.2 [degC]
  
```



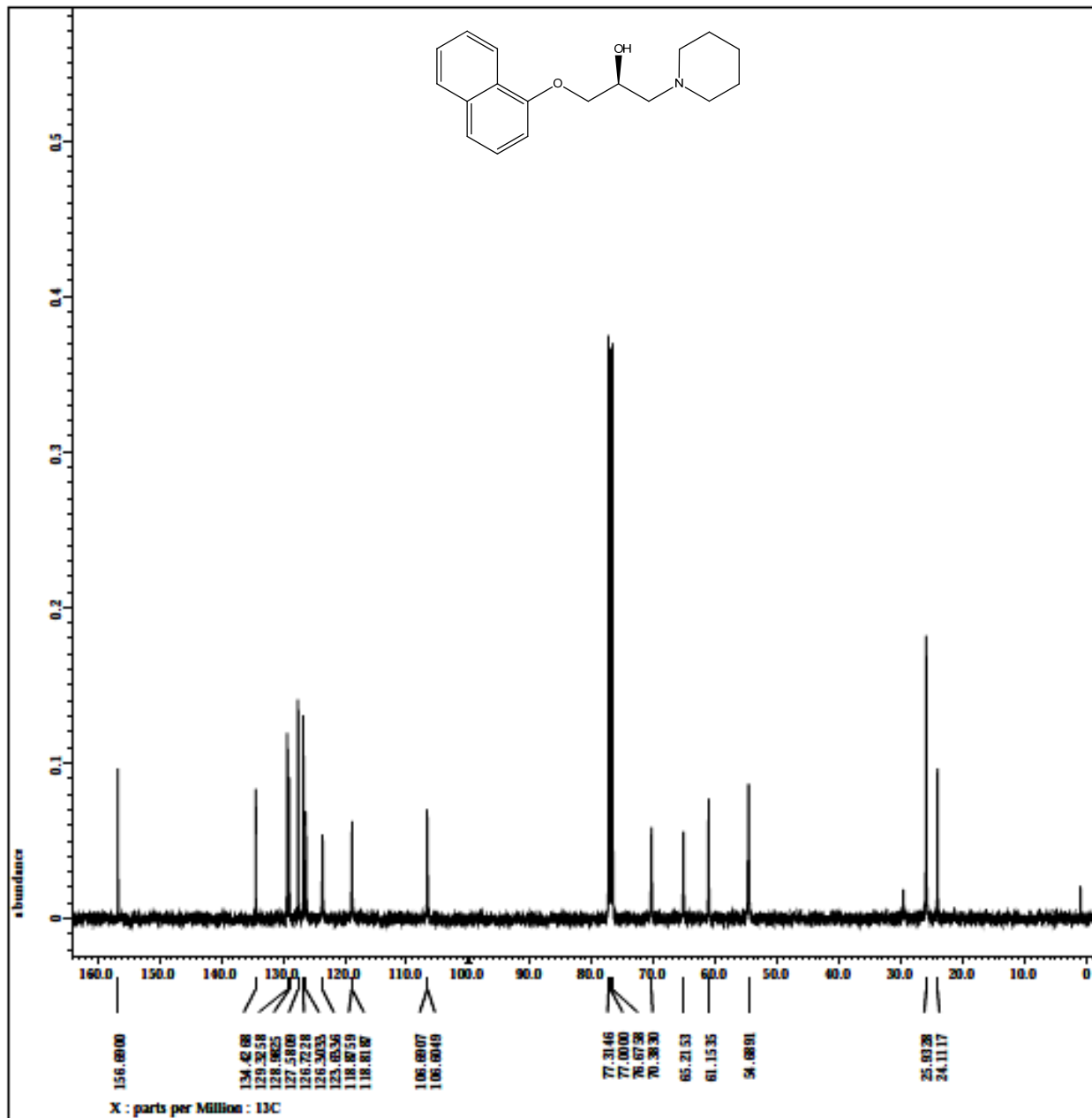
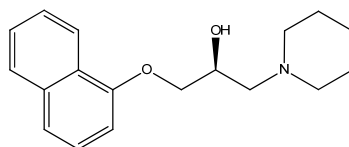
```

File Name      = SS 3-15PR_PROTON-4.jd
Author        = Delta
Experiment    = single pulse.scf
Sample id     = SS 3-15PR
Solvent       = CHLOROFORM-D
Creation time  = 11-JUN-2012 09:46:38
Revision time = 15-APR-2014 16:04:50
Current time  = 15-APR-2014 16:05:15

Comment       = SS 3-15PR
Data format   = 1D COMPLEX
Data size     = 13107
Data title    = 1H
Data units    = [ppm]
Dimensions    = 1
Site          = KCI 400P
Spectrometer  = DELTA2_NMR

Field strength = 9.389766 [T] (400 [MHz])
F1 acq duration = 1.36577024 [s]
F1 domain      = 1H
F1 freq        = 399.78219838 [MHz]
F1 offset      = 5 [ppm]
F1 points      = 16384
F1 prescans    = 1
F1 resolution  = 0.73218757 [Hz]
F1 sweep       = 11.99416123 [kHz]
Irr domain     = 1H
Irr freq       = 399.78219838 [MHz]
Irr offset     = 5 [ppm]
Tri domain     = 1H
Tri freq       = 399.78219838 [MHz]
Tri offset     = 5 [ppm]
Clipped       = FALSE
Mod return     = 1
Scans         = 16
Total scans    = 16

X 90 width     = 11.57 [us]
X acq time     = 1.36577024 [s]
X angle        = 45 [deg]
X atm         = 5 [dB]
X pulse       = 5.785 [us]
Irr mode       = Off
Tri mode       = Off
Delta preset   = FALSE
Initial wait   = 1 [s]
Recvr gain     = 30
Relaxation delay = 4 [s]
Repetition time = 5.36577024 [s]
Temp get       = 20.3 [dC]
  
```

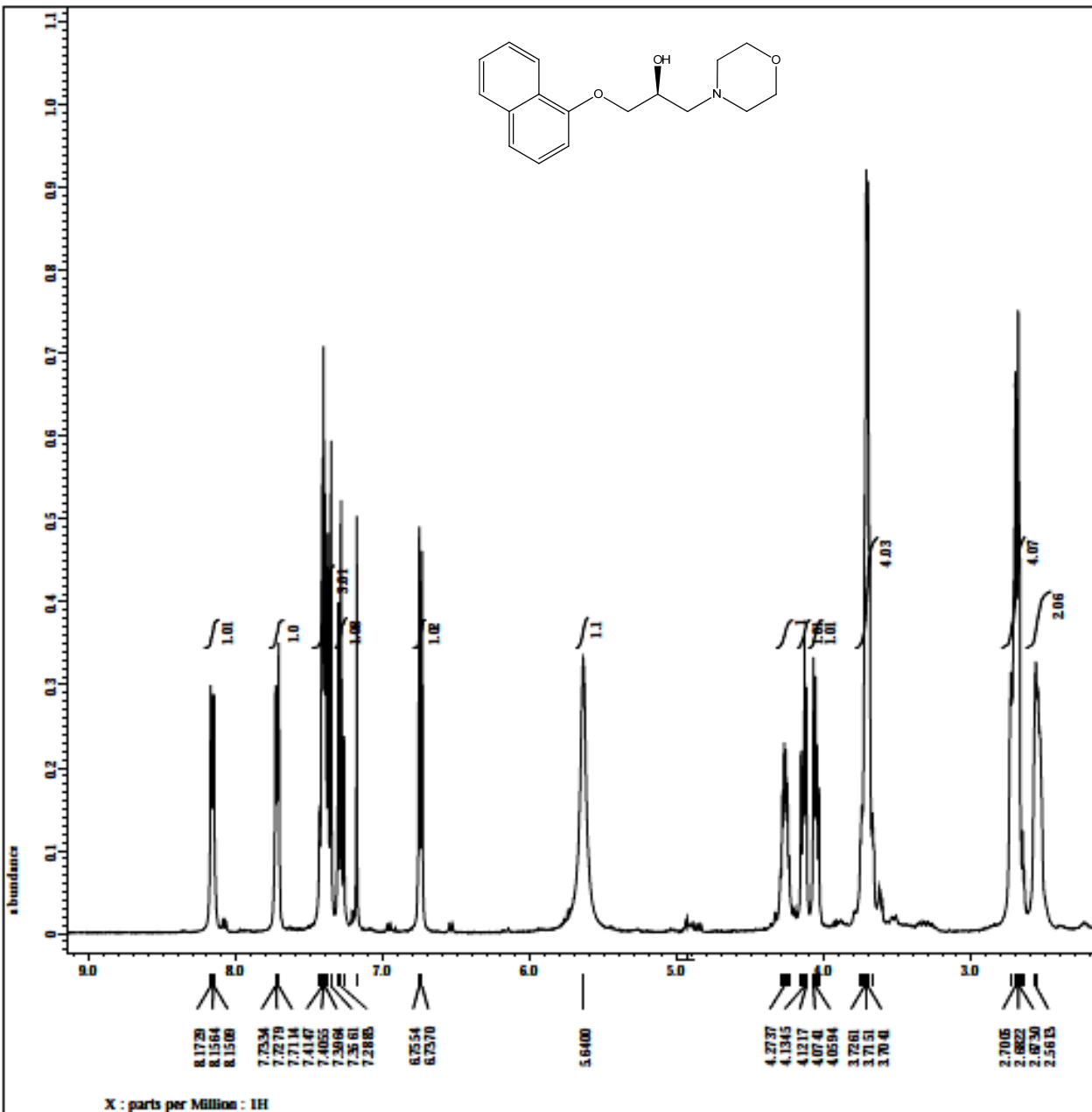
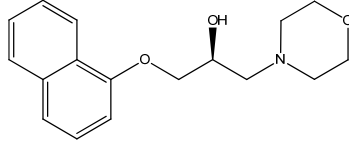


Filename = FN_3-15PR_CARBON-4.
 Author = delta
 Experiment = single pulse_dec
 Sample_id = FN_3-15PR
 Solvent = CHLOROFORM-D
 Creation_time = 12-JUL-2012 22:46:30
 Revision_time = 15-APR-2014 22:10:24
 Current_time = 15-APR-2014 23:00:09

Comment = FN_3-15PR
 Data_format = 1D_COMPLEX
 Dim_size = 26214
 Dim_title = 13C
 Dim_units = [ppm]
 Dimensions = 1
 Site = NCI 400P
 Spectrometer = DELTA2 NMR

Field_strength = 9.389766 [T] (400 [MHz])
 X_acq_duration = 1.04333312 [s]
 X_domain = 13C
 X_freq = 100.52530333 [MHz]
 X_offset = 100 [ppm]
 X_points = 32768
 X_prescans = 4
 X_resolution = 0.25846665 [Hz]
 X_sweep = 31.40703518 [kHz]
 Irr_domain = 1H
 Irr_freq = 399.78219838 [MHz]
 Irr_offset = 5 [ppm]
 Clipped = FALSE
 Mod_return = 1
 Scale = 750
 Total_scans = 750

X_90_width = 11.75 [ns]
 X_acq_time = 1.04333312 [s]
 X_angle = 30 [deg]
 X_atn = 10 [dB]
 X_pulse = 3.91666667 [ns]
 Irr_atn_dec = 24.95 [dB]
 Irr_atn_noc = 24.95 [dB]
 Irr_noise = WALTZ
 Decoupling = TRUE
 Initial_wait = 1 [s]
 Noe = TRUE
 Noe_time = 2 [s]
 Recvr_gain = 54
 Relaxation_delay = 2 [s]
 Repetition_time = 3.04333312 [s]
 Temp_get = 21.7 [dC]



```

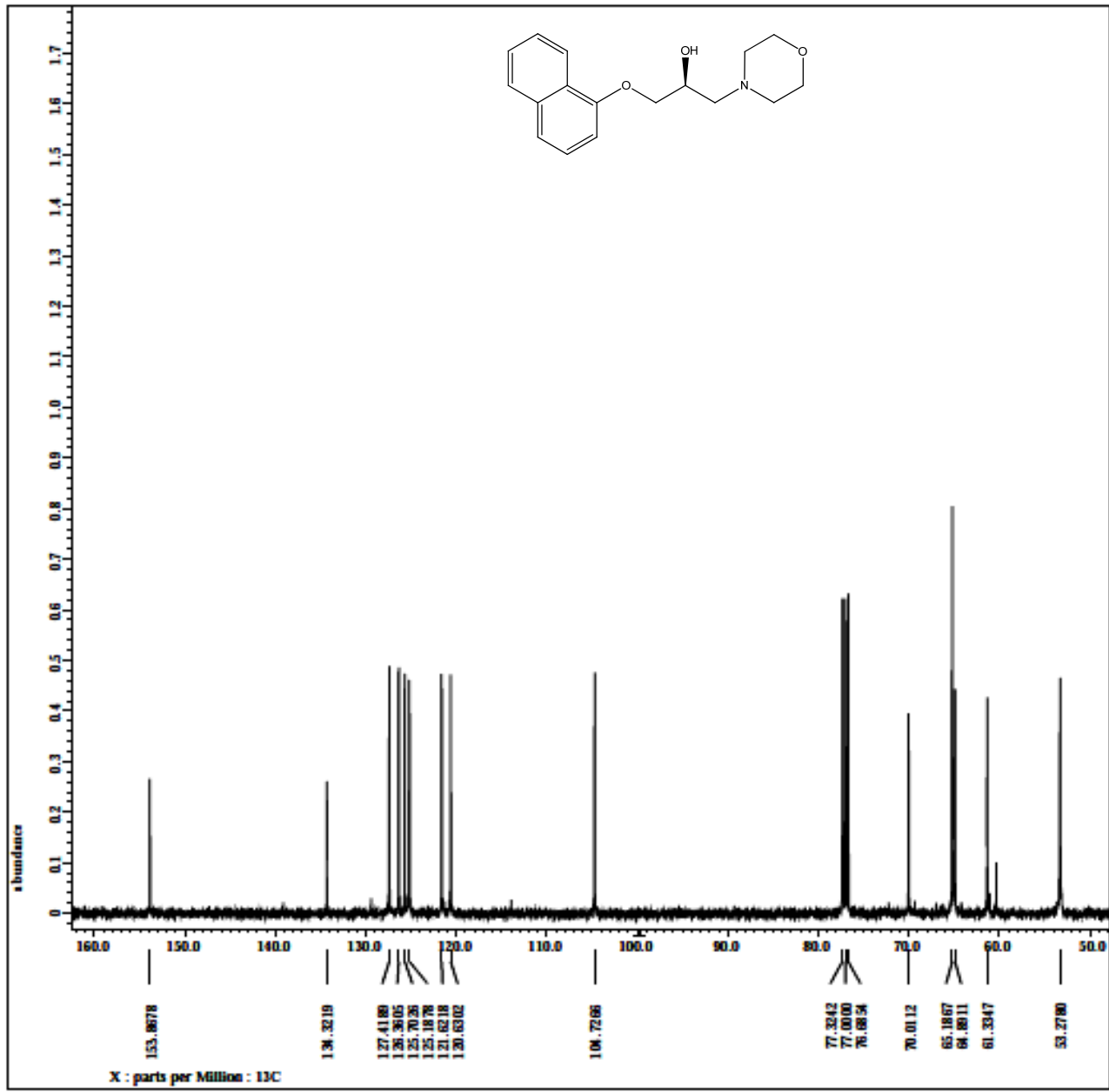
File name      = SS 3-15MF_PROTON-6.jd
Author         = delta
Experiment     = single pulse.sc2
Sample id      = SS 3-15MF
Solvent        = CHLOROFORM-D
Creation time   = 11-MAY-2012 11:02:54
Revision time  = 17-APR-2014 11:36:12
Current time   = 17-APR-2014 11:36:23

Comment       = SS 3-15MF
Data format    = 1D COMPLEX
Data size      = 13107
Data title     = 1H
Data units     = [ppm]
Dimensions    = 1
Site           = KCI 400P
Spectrometer   = DELTA2 NMR

Field strength = 9.389766 [T] (400 [MHz])
F1 acq duration = 1.36577024 [s]
F1 domain       = 1H
F1 freq         = 399.78219838 [MHz]
F1 offset       = 5 [ppm]
F1 points       = 16354
F1 prescans     = 1
F1 resolution   = 0.73218757 [Hz]
F1 sweep        = 11.99616123 [kHz]
Irr domain      = 1H
Irr freq        = 399.78219838 [MHz]
Irr offset       = 5 [ppm]
Tri domain      = 1H
Tri freq        = 399.78219838 [MHz]
Tri offset       = 5 [ppm]
Clipped         = FALSE
Mod return      = 1
Scans           = 16
Total scans     = 16

X 90 width      = 11.57 [us]
X acq time      = 1.36577024 [s]
X angle         = 45 [deg]
X atm           = 5 [dB]
X pulse         = 5.785 [us]
Irr mode        = Off
Tri mode        = Off
Delta preset    = FALSE
Initial wait    = 1 [s]
Recvr gain      = 30
Relaxation delay = 4 [s]
Repetition time = 5.36577024 [s]
Temp get        = 22.4 [dC]
  
```

X : parts per Million : 1H



```

File name      = SS 3-15MF_CASRON-4.jd
Author        = delta
Experiment    = single pulse_dec
Sample id     = SS 3-15MF
Solvent       = CHLOROFORM-D
Creation time  = 15-MAY-2012 17:13:46
Revision time = 17-APR-2014 11:39:23
Current time  = 17-APR-2014 11:41:40

Comment       = SS 3-15MF
Data format   = 1D COMPLEX
Dim size      = 26214
Dim title     = 13C
Dim units     = [ppm]
Dimensions    = X
Site          = ECI 400P
Spectrometer  = DELTA2 400P

Field strength = 9.389766 [T] (400 [MHz])
X_acq duration = 1.04333312 [s]
X_domain       = 13C
X_freq         = 100.52530333 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 0.95846665 [Hz]
X_sweep        = 31.40703518 [kHz]
Irr_domain     = 1H
Irr_freq       = 399.78219838 [MHz]
Irr_offset     = 5 [ppm]
Clipped        = FALSE
Mod return     = 1
Scans          = 750
Total scans    = 750

X_90 width     = 11.75 [us]
X_acq time     = 1.04333312 [s]
X_angle        = 30 [deg]
X_atn          = 10 [dB]
X_pulse        = 3.91666667 [us]
Irr_atn_dec    = 24.95 [dB]
Irr_atn_noe    = 24.95 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe time       = 2 [s]
Recvr gain     = 58
Relaxation delay = 2 [s]
Repetition time = 3.04333312 [s]
Temp_get       = 19 [C]
  
```

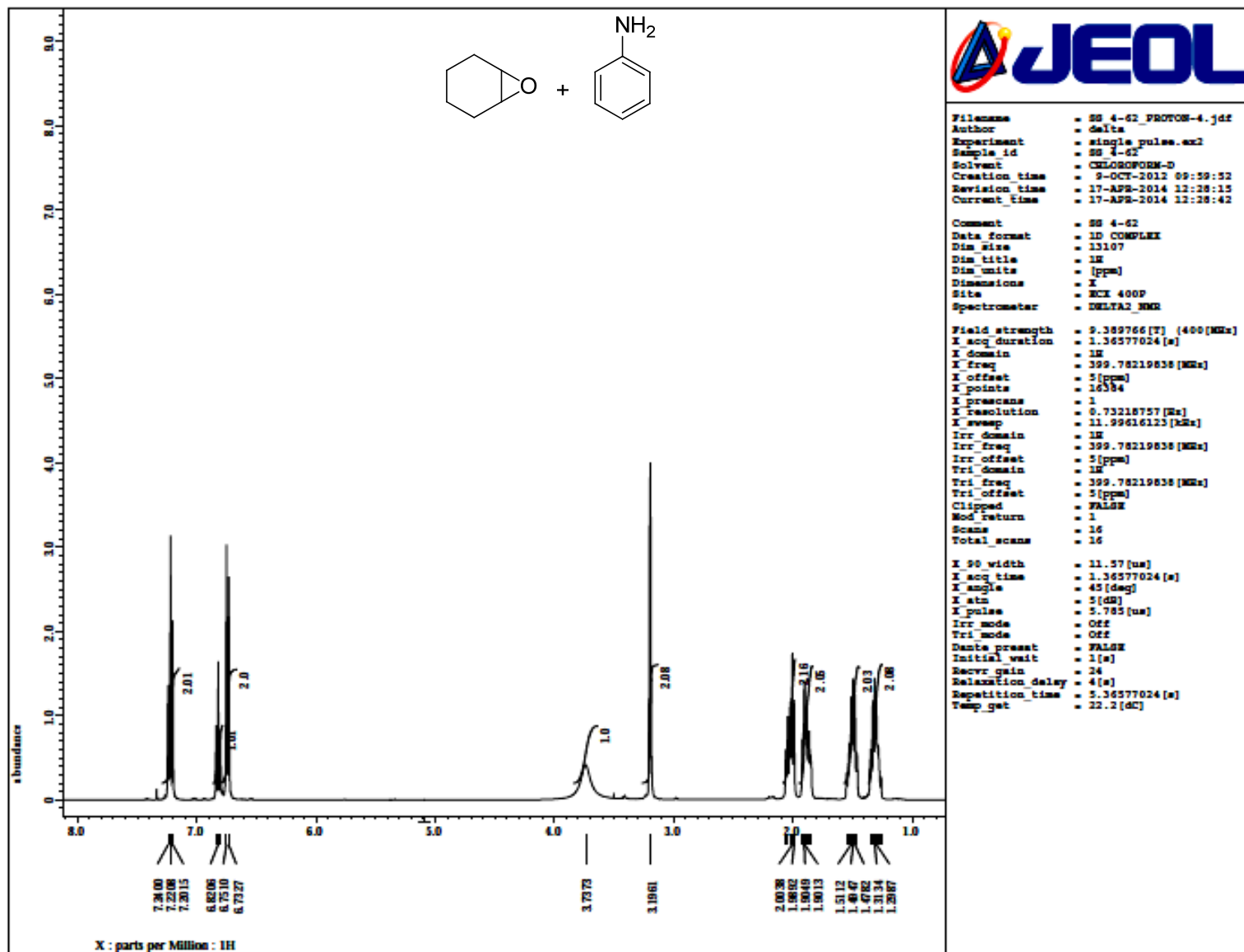
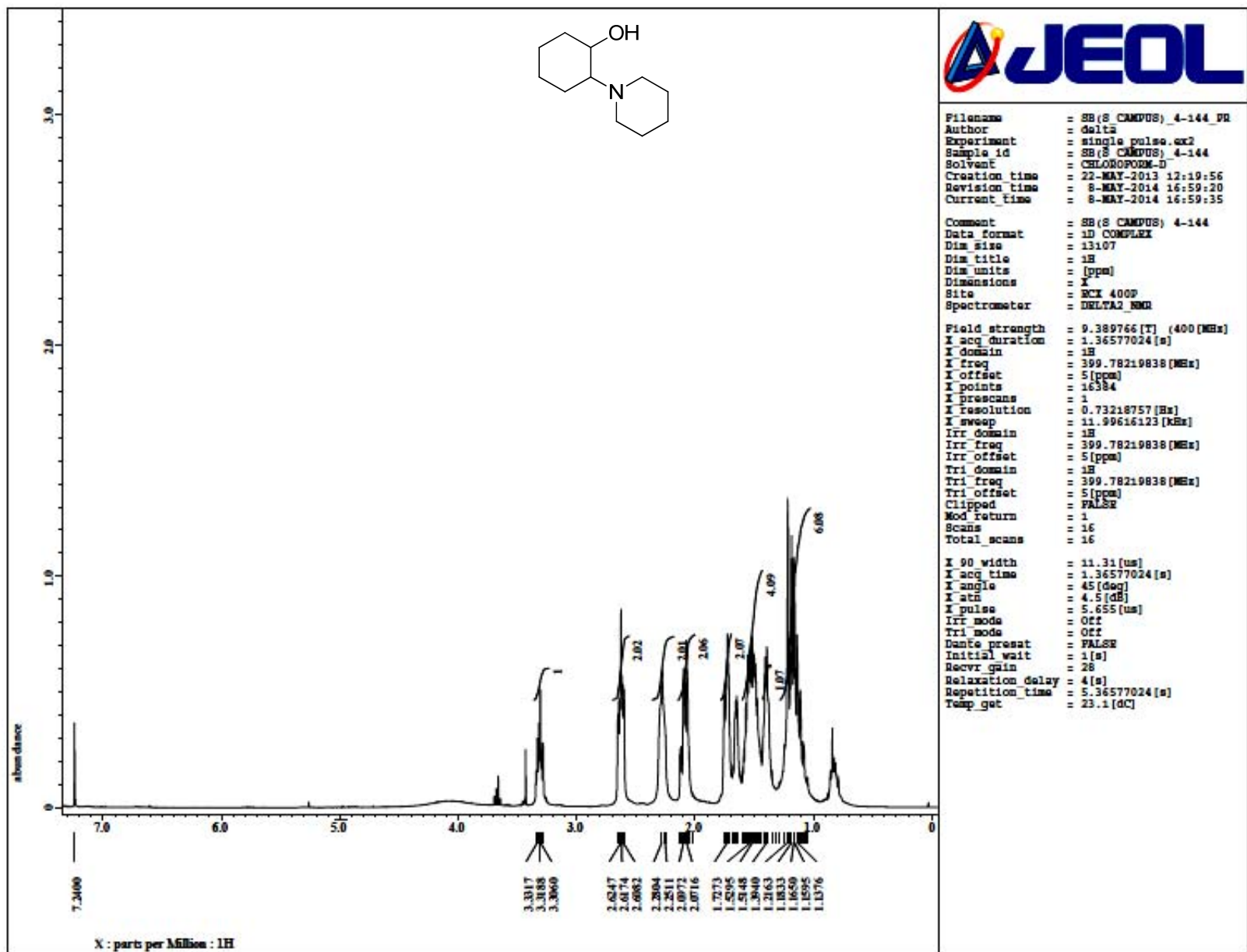
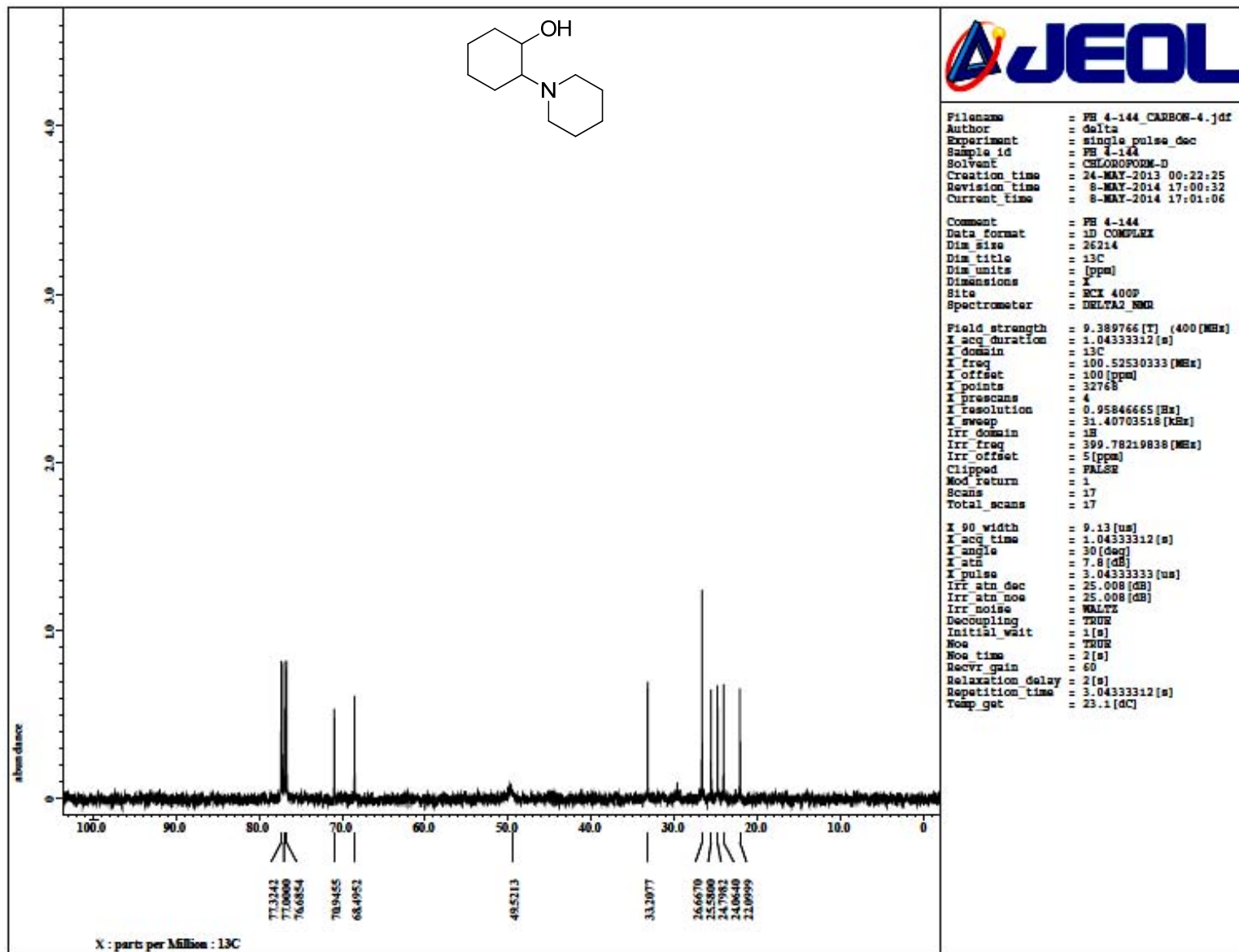
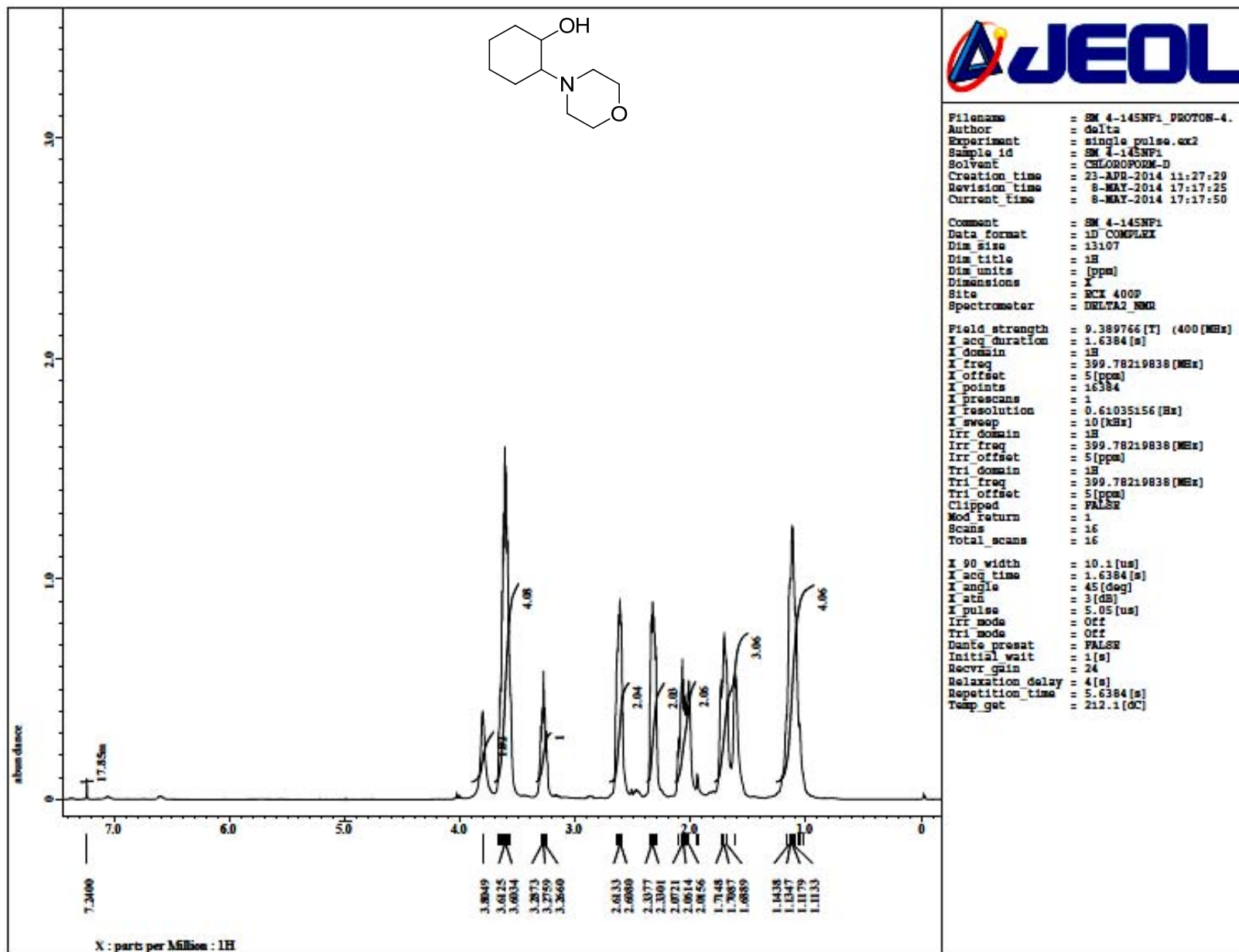
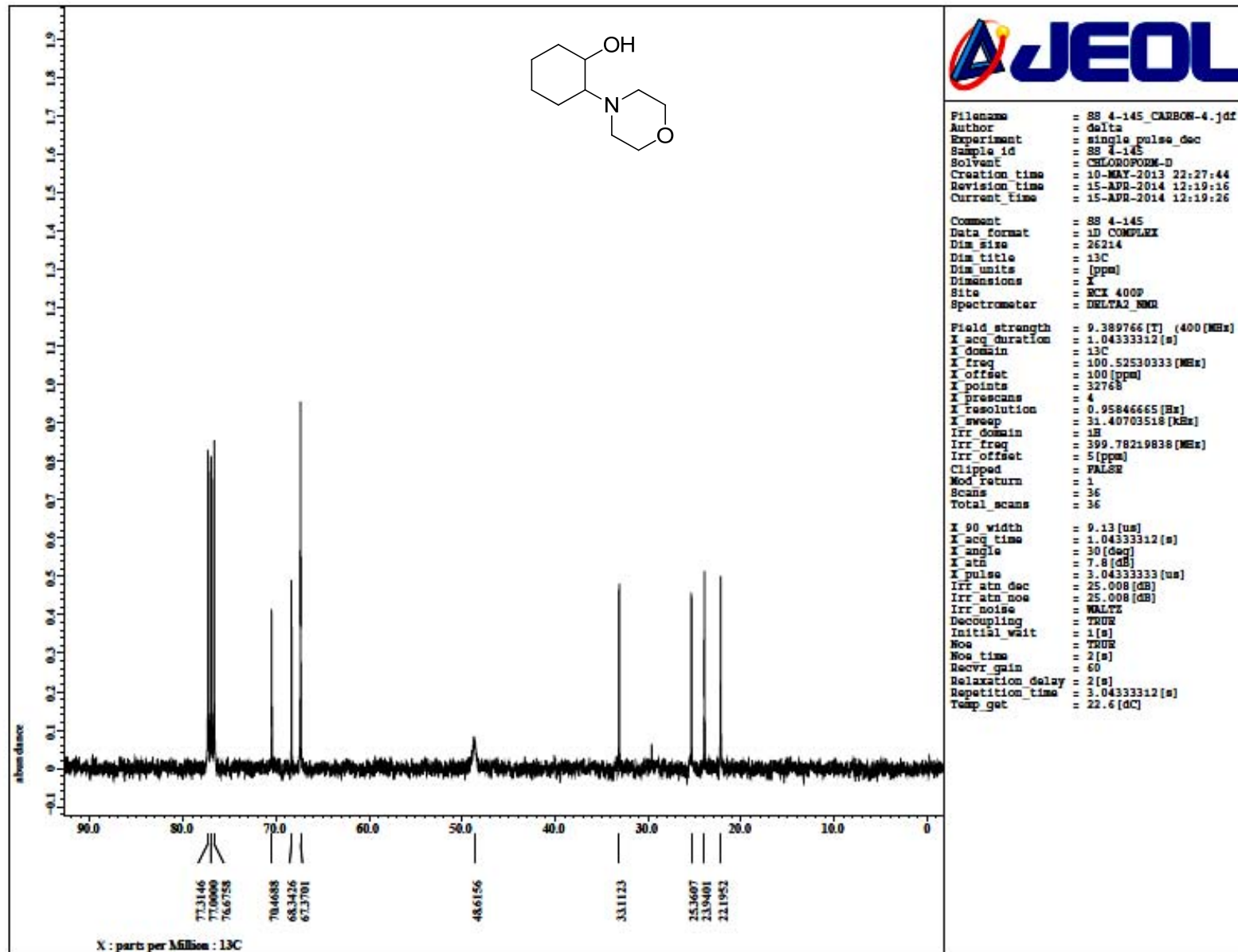


Table 1: entry (without catalyst)









```

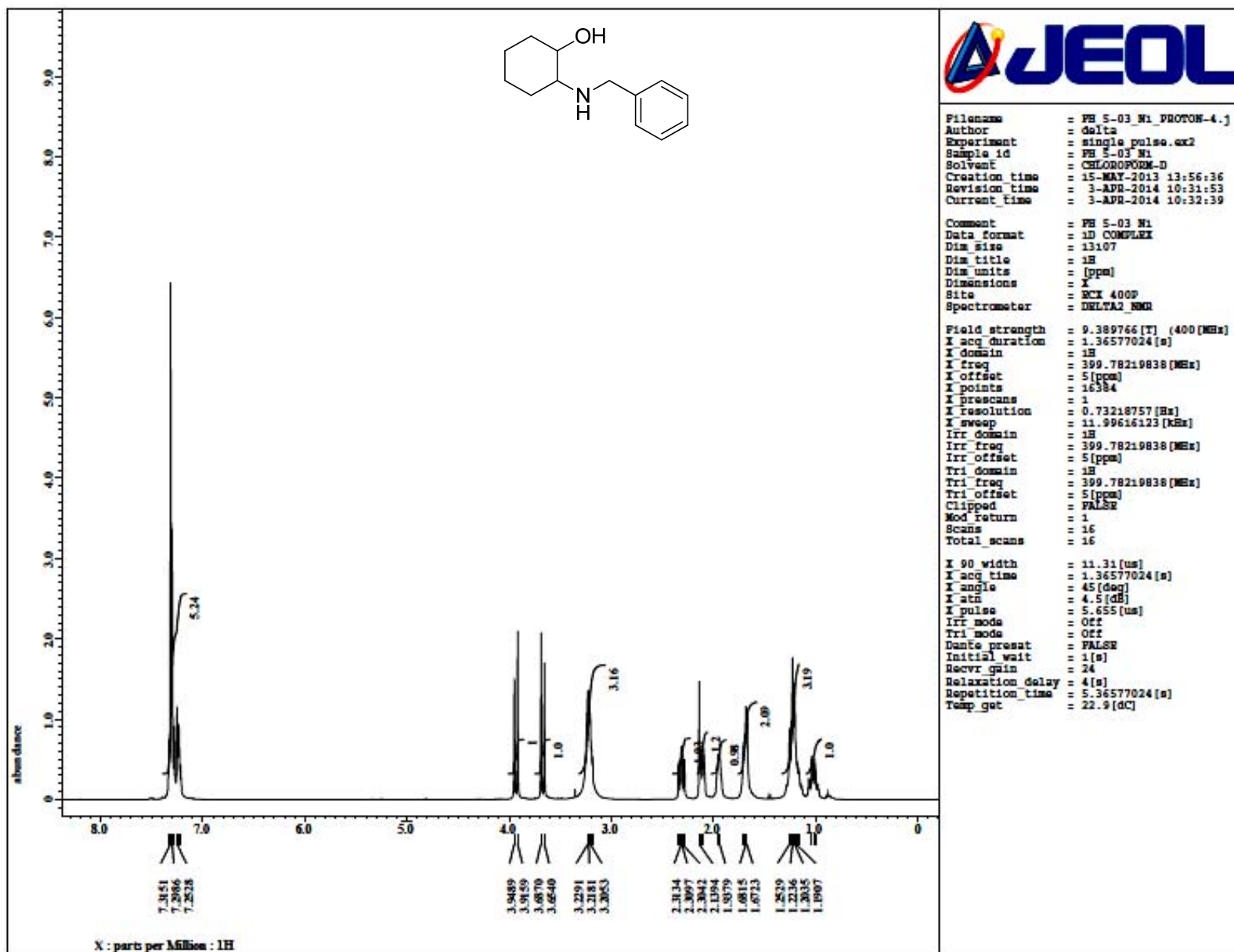
Filename      = 88 4-145 CARBON-4.jdf
Author       = delta
Experiment   = single pulse_dec
Sample id    = 88 4-145
Solvent      = CHLOROFORM-D
Creation time = 10-MAY-2013 22:27:44
Revision time = 15-APR-2014 12:19:16
Current time  = 15-APR-2014 12:19:26

Comment      = 88 4-145
Data format  = 1D COMPLEX
Dim size     = 26214
Dim title    = 13C
Dim units    = [ppm]
Dimensions   = 1
Site         = ECI 400P
Spectrometer = DELTA2 NMR

Field strength = 9.389766 [T] (400 [MHz])
F1 acq duration = 1.04333312 [s]
F1 domain      = 13C
F1 freq        = 100.52530333 [MHz]
F1 offset      = 100 [ppm]
F1 points      = 32768
F1 prescans    = 4
F1 resolution  = 0.95846665 [Hz]
F1 sweep       = 31.40703518 [kHz]
F1 domain      = 1H
F1 freq        = 399.78219838 [MHz]
F1 offset      = 5 [ppm]
Clipped       = FALSE
Mod Return    = 1
Scans         = 36
Total scans   = 36

F2 90 width   = 9.13 [us]
F2 acq time   = 1.04333312 [s]
F2 angle      = 30 [deg]
F2 atn        = 7.8 [dB]
F2 pulse      = 3.04333333 [us]
F2 atn dec    = 25.008 [dB]
F2 atn noe    = 25.008 [dB]
F2 noise      = WALTZ
Decoupling    = TRUE
Initial wait  = 1 [s]
Noe           = TRUE
Noe time      = 2 [s]
Recvr gain    = 60
Relaxation delay = 2 [s]
Repetition time = 3.04333312 [s]
Temp get      = 22.6 [dC]

```



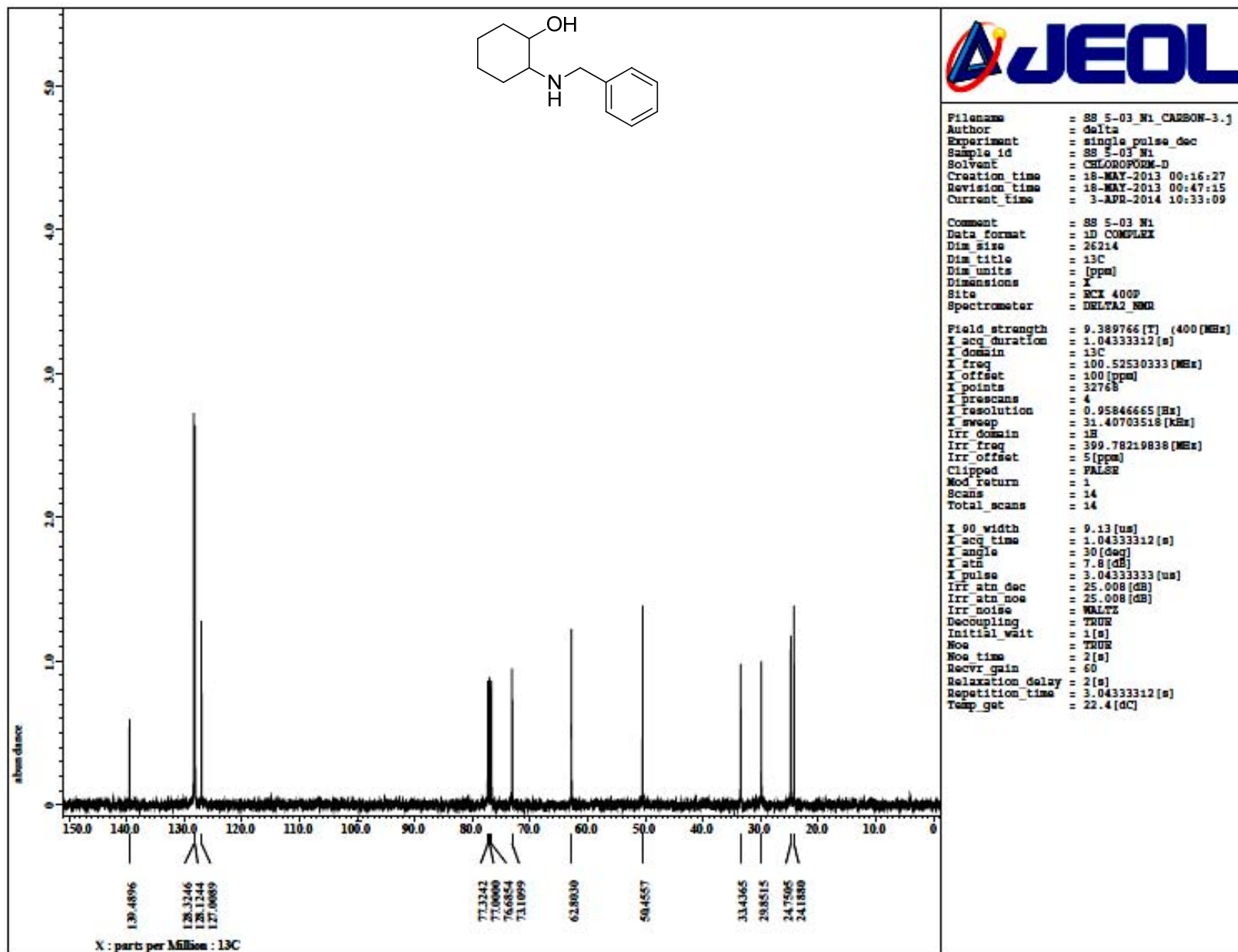
```

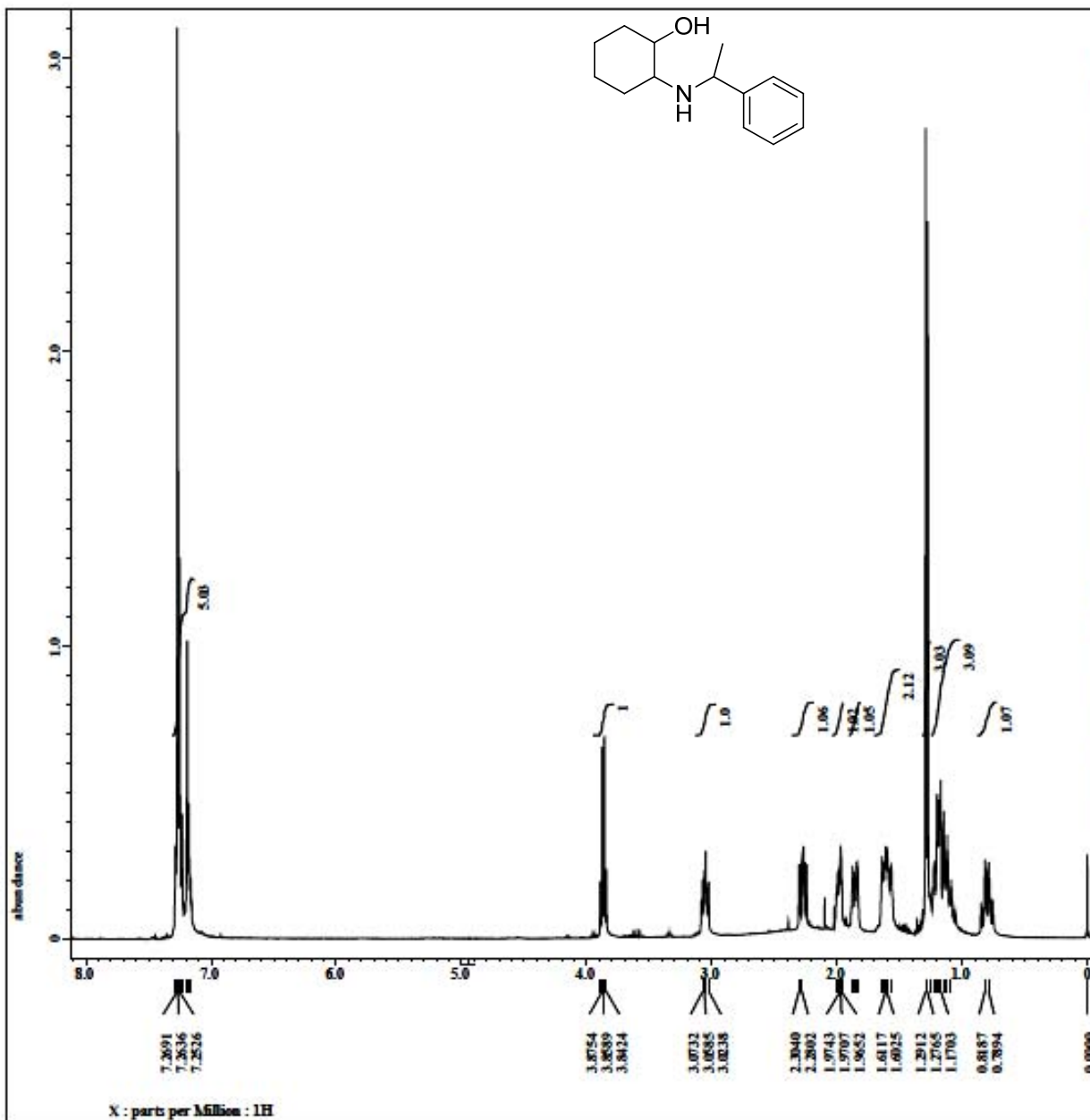
Filename      = PB 5-03 N1 PHOTON-4.]
Author       = delta
Experiment   = single pulse.ex2
Sample id    = PB 5-03 N1
Solvent      = CHLOROFORM-D
Creation time = 15-MAY-2013 13:56:36
Revision time = 3-APR-2014 10:31:53
Current_time = 3-APR-2014 10:32:39

Comment      = PB 5-03 N1
Data format  = 1D COMPLEX
Dim size     = 13107
Dim title    = 1H
Dim units    = [ppm]
Dimensions   = X
Site         = ECI 400P
Spectrometer = DELTA2 NMR

Field strength = 9.389766 [T] (400 [MHz])
F1 acq duration = 1.36577024 [s]
F1 domain      = 1H
F1 freq       = 399.78219838 [MHz]
F1 offset     = 5 [ppm]
F1 points     = 16384
F1 prescans   = 1
F1 resolution = 0.73218757 [Hz]
F1 sweep      = 11.99616123 [kHz]
F1r domain    = 1H
F1r freq      = 399.78219838 [MHz]
F1r offset    = 5 [ppm]
F1r domain    = 1H
F1r freq      = 399.78219838 [MHz]
F1r offset    = 5 [ppm]
Clipped      = FALSE
Mod return   = 1
Scans        = 16
Total scans  = 16

F2 90 width   = 11.31 [us]
F2 acq time   = 1.36577024 [s]
F2 angle      = 45 [deg]
F2 attn       = 4.5 [dB]
F2 pulse     = 5.655 [us]
F2r mode     = Off
F2r mode     = Off
Dante preset = FALSE
Initial wait  = 1 [s]
Recvr gain   = 24
Relaxation delay = 4 [s]
Repetition time = 5.36577024 [s]
Temp_gat     = 22.9 [dC]
  
```

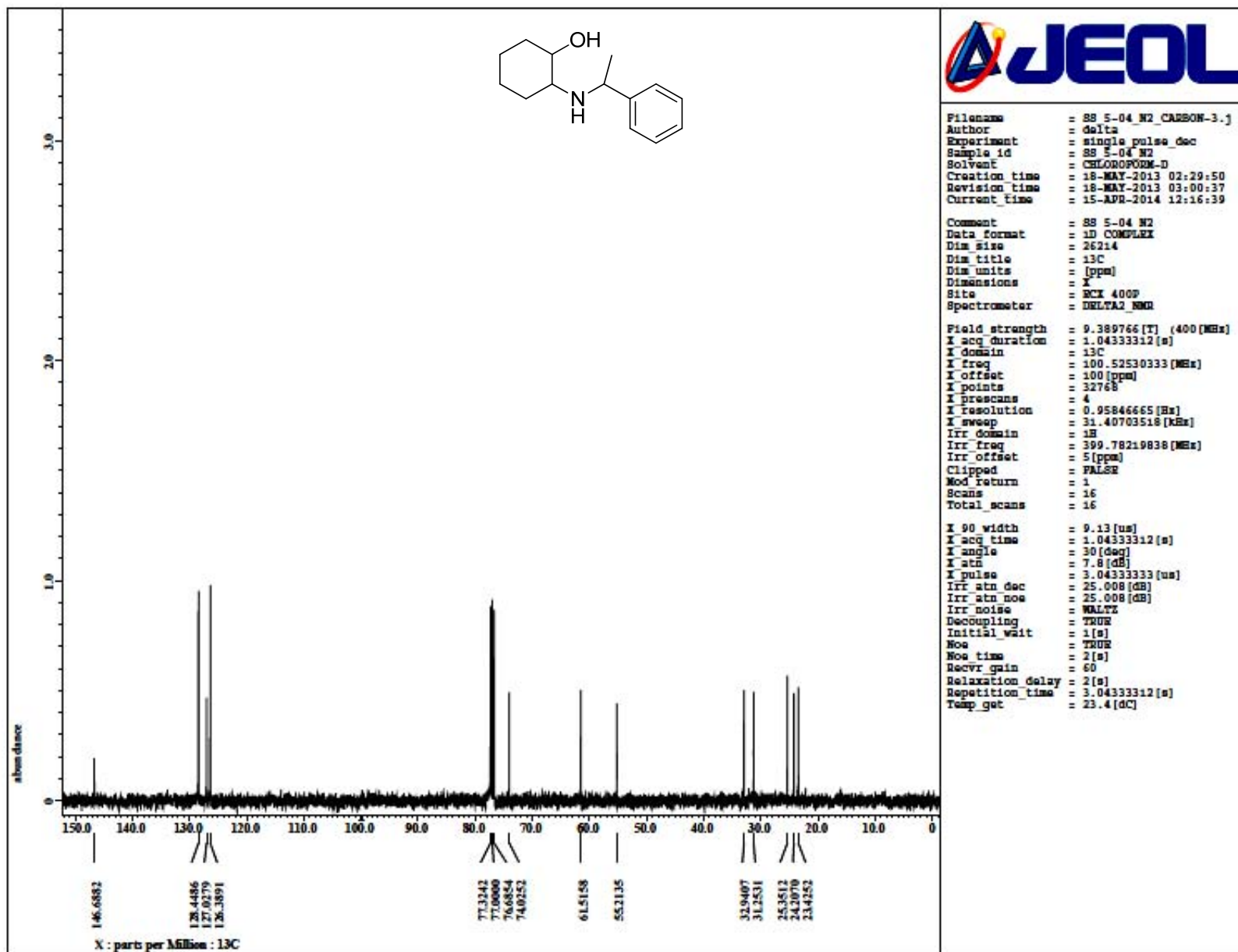


Filename = PH 5-04_M2_PROTON-6.j
 Author = delta
 Experiment = single pulse.ex2
 Sample id = PH 5-04_M2
 Solvent = CHLOROFORM-D
 Creation time = 4-JUN-2013 12:54:47
 Revision time = 15-APR-2014 12:14:58
 Current time = 15-APR-2014 12:15:24

Comment = PH 5-04_M2
 Data format = 1D COMPLEX
 Data size = 13107
 Data title = 1H
 Data units = [ppm]
 Dimensions = 1
 Site = ECK 400P
 Spectrometer = DELTA2_NMR

Field strength = 9.389766 [T] (400 [MHz])
 X acq duration = 1.36577024 [s]
 X domain = 1H
 X freq = 399.78219838 [MHz]
 X offset = 5 [ppm]
 X points = 16384
 X prescans = 1
 X resolution = 0.73218757 [Hz]
 X sweep = 11.99616123 [kHz]
 Irr domain = 1H
 Irr freq = 399.78219838 [MHz]
 Irr offset = 5 [ppm]
 Tri domain = 1H
 Tri freq = 399.78219838 [MHz]
 Tri offset = 5 [ppm]
 Clipped = FALSE
 Mod return = 1
 Scans = 16
 Total scans = 16

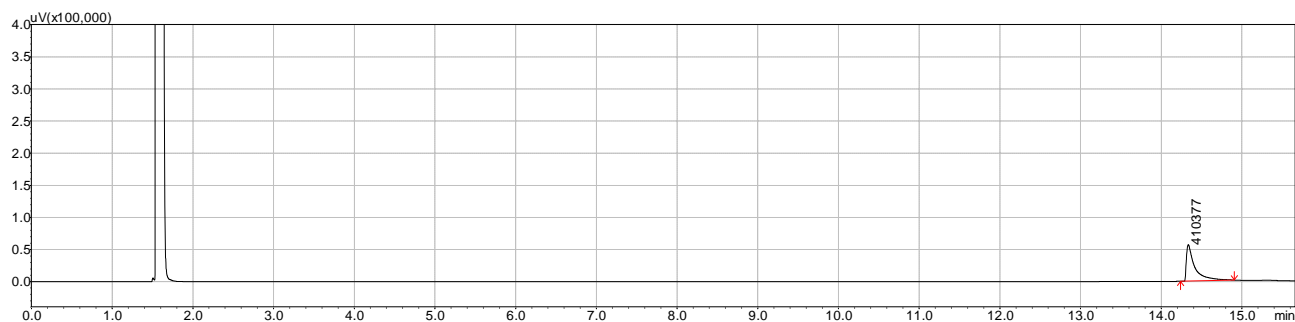
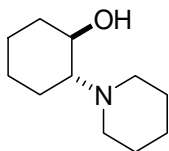
X 90 width = 11.31 [us]
 X acq time = 1.36577024 [s]
 X angle = 45 [deg]
 X atn = 4.5 [dB]
 X pulse = 5.655 [us]
 Irr mode = Off
 Tri mode = Off
 Dante presat = FALSE
 Initial wait = 1 [s]
 Recvr gain = 30
 Relaxation delay = 4 [s]
 Repetition time = 5.36577024 [s]
 Temp set = 23 [dC]



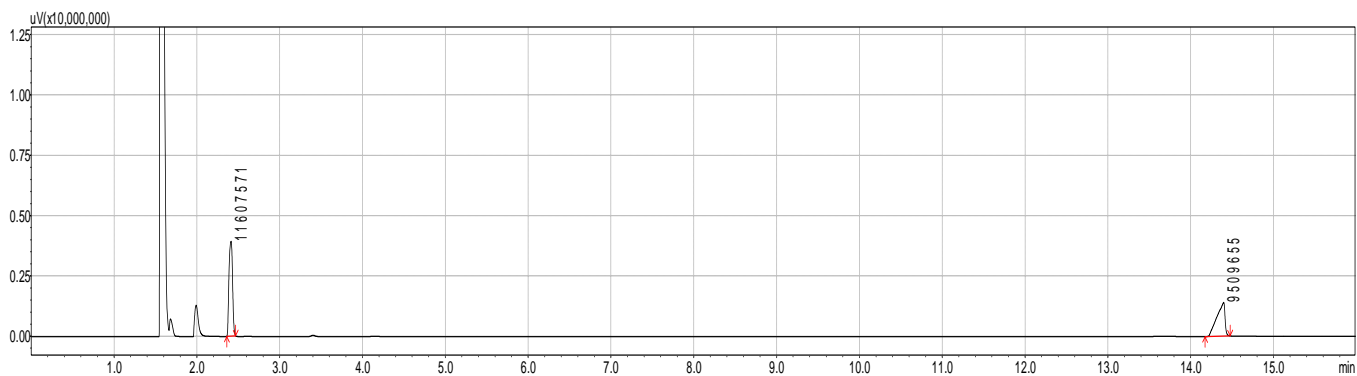
GC chromatogram for the table 2

Conditions for GC: GC parameters for capillary columns (30 m × 0.25 mm): injector 250 °C; detector 250 °C; oven 100 °C for 3 min then 5 °C min⁻¹ until 180 °C for 15 min; column pressure 118.3 kPa, column flow 1.36 mL min⁻¹; linear velocity 35 cm s⁻¹; total flow 45.1 mL min⁻¹.

Trans-2-(piperidin-1-yl)cyclohexanol

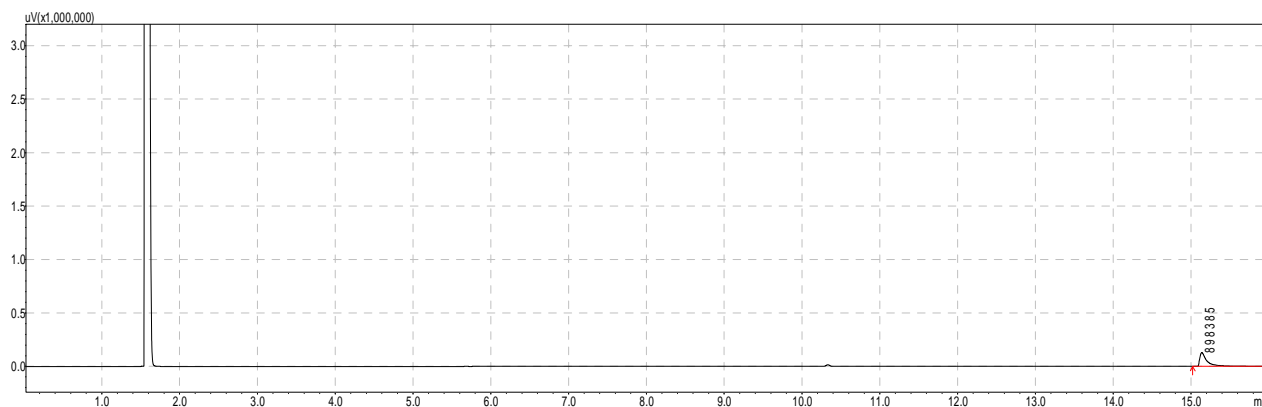
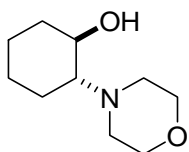


Peak#	Ret.Time	Area	Height	Conc.	Area%
1	14.336	410377.3	56495.7	100.0000	100.0000

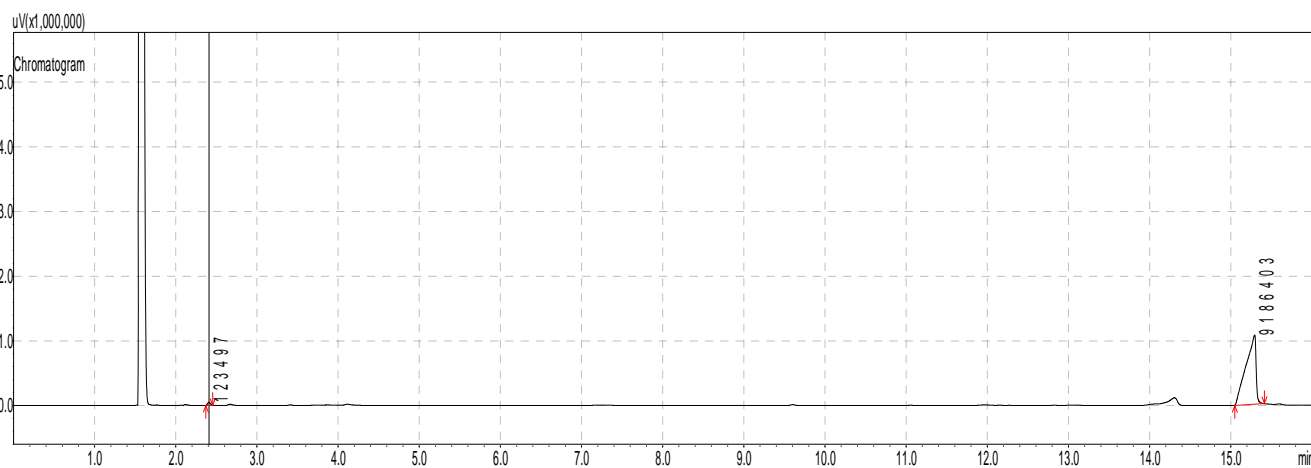


Peak	Ret.Time	Area	Height	Conc.	Area%
1	2.412	11607571.0	3885690.0	54.96731	54.9673
2	14.398	9509654.7	1388792.0	45.03269	45.0327

Trans-2-morpholinocyclohexanol

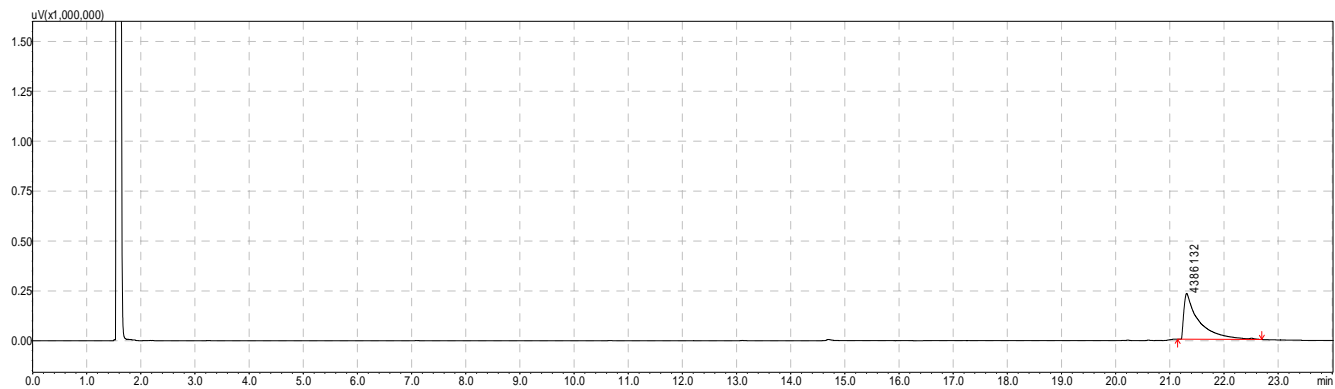
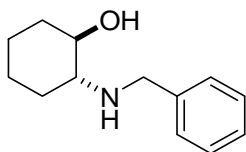


Peak#	Ret.Time	Area	Height	Conc.	Area%
1	15.140	898384.7	129738.9	100.0000	100.0000

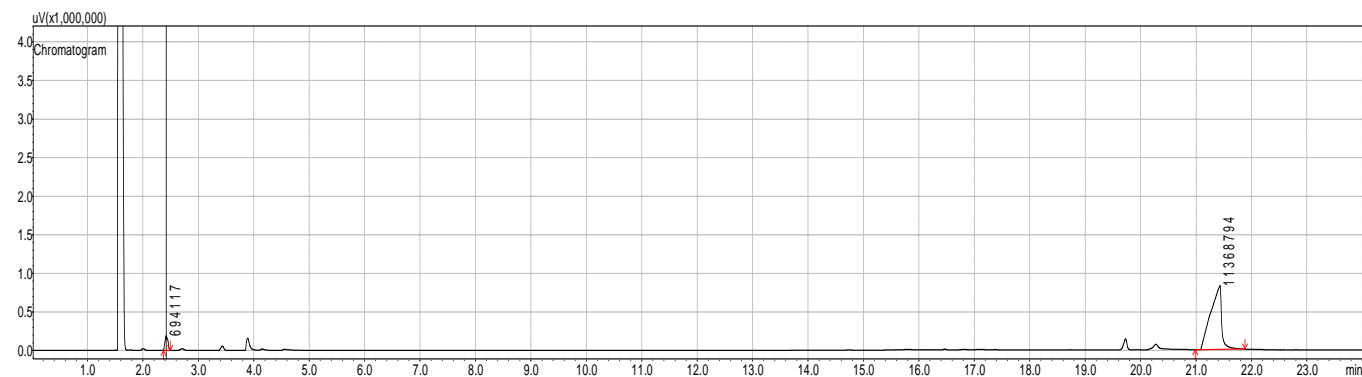


Peak	Ret.Time	Area	Height	Conc.	Area%
1	2.409	123497.0	48783.9	1.32651	1.3265
2	15.294	9186403.0	1069509.8	98.67349	98.6735

Trans-2-(benzylamino)cyclohexanol :

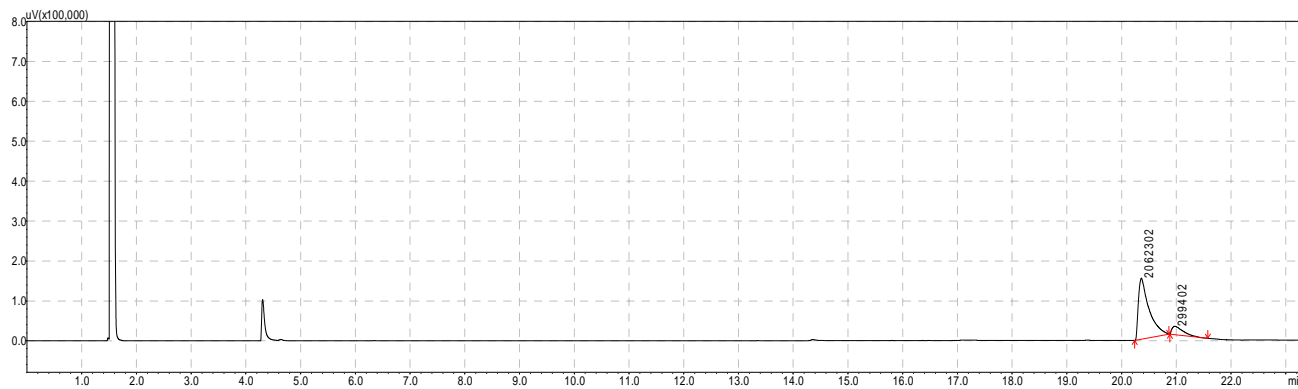
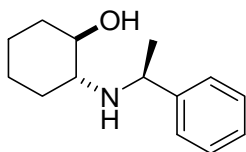


Peak#	Ret.Time	Area	Height	Conc.	Area%
1	21.312	4386132.2	229637.6	100.0000	100.0000

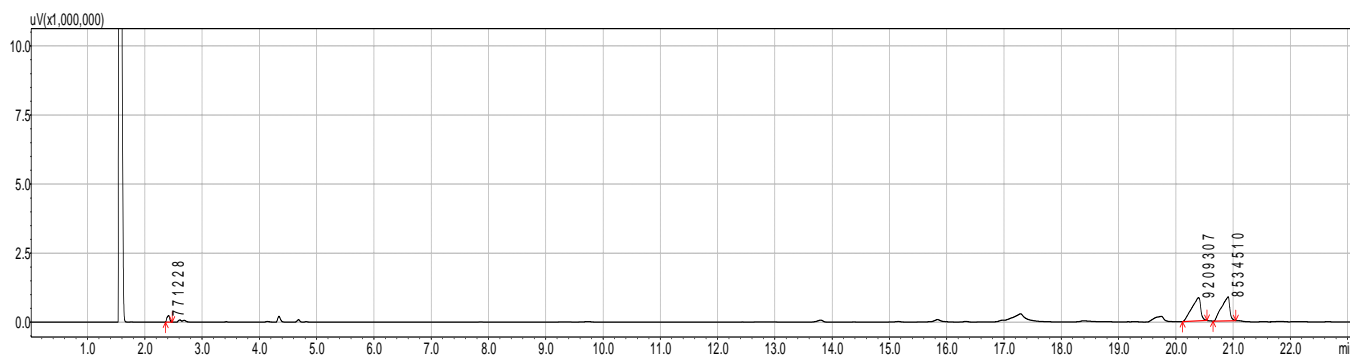


Peak	Ret.Time	Area	Height	Conc.	Area%
1	2.421	694117.5	185040.6	5.75415	5.7541
2	21.431	11368794.5	826008.3	94.24585	94.2459

Trans-2-((S)-1-phenylethylamino)cyclohexanol :



Peak#	Ret.Time	Area	Height	Conc.	Area%
1	20.363	2062301.8	152902.8	87.32261	87.3226
2	20.974	299402.4	21296.3	12.67739	12.6774



Peak	Ret.Time	Area	Height	Conc.	Area%
1	2.415	771228.4	239607.3	4.16541	4.1654
2	20.399	9209306.9	839048.6	49.73959	49.7396
3	20.915	8534510.1	861032.9	46.09500	46.0950