Electronic Supplementary Information

Preparation of optically active cycloalkenes bearing all-carbon quaternary stereogenic centres via lipase–oxovanadium combo-catalysed dynamic kinetic resolution

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Contents

General considerations

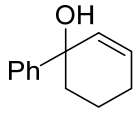
Melting points were determined on a Yanagimoto melting point apparatus and are uncorrected. Infrared (IR) absorption spectra were recorded on a SHIMADZU FTIR-8400S spectrophotometer. ^{1}H and ^{13}C NMR spectra were measured on JEOL JNM-ECA500 (^{1}H: 500 MHz, ^{13}C: 125 MHz), JEOL AL-400 (^{1}H: 400 MHz, ^{13}C: 100 MHz) and JEOL AL-300 (^{1}H: 300 MHz, ^{13}C: 75 MHz) instruments with chemical shifts reported in ppm relative to the residual deuterated solvent. The mass spectra (MS) were measured on JEOL JMS-S3000 (MALDI) and JEOL JMS-700 (FAB) instruments. Yield refers to isolated yields of compounds greater than 95% purity as determined by ^{1}H NMR analysis. ^{1}H NMR and melting points (where applicable) of all known compounds were taken. All new products were further characterized by ^{13}C NMR, IR and high resolution mass spectrum (HRMS). HPLC analyses were carried out using a JASCO LC-2000Plus system (HPLC pump: PU-2080, UV detectors: MD-2018 and MD-4017) equipped with Daicel CHIRALPAK AD-3, IC-3, IE, OD-3 and OJ-H columns. All optically active compounds are detected by 254 nm wavelength absorption unless otherwise noted. Optical rotations were measured on a JASCO P-1030 polarimeter.

Immobilized *Burkholderia cepacia* lipases PS-IM and PS-CII, kindly supplied by Amano Enzyme Inc., Japan, were used as received without further purification. *Candida antarctica* lipase B (CAL-B) immobilized on a support (commercial name: Chirazyme L-2 C4) was purchased from Roche Diagnostics K. K., Japan and was used as received without further purification. V-MPS4 was prepared according to the report[1] (it is now commercially available from Wako Pure Chemical Industries, Ltd. Japan). Kanto silica gel 60N was used for column chromatography. All reagents were of reagent grade unless otherwise stated. In general, the reactions were carried out in anhydrous solvents.

The preparation of racemic tertiary alcohols 5a–5e

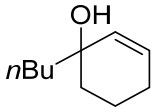
A general procedure: Under an argon atmosphere, an organolithium compound (12.0 mmol) was added to a solution of an enone **1** (10.0 mmol) in anhydrous Et_2O (50 mL) at –78 °C. After being stirred for 10 min at –78 °C, the reaction mixture was warmed to 0 °C over 10 min and quenched with saturated aq. NH_4Cl (50 mL). The aqueous phase was extracted with Et_2O (3 x 25 mL), and the combined organic phases were washed with brine (50 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel) to give (±)-**5**.

1-Phenylcyclohex-2-enol [(±)-5a]



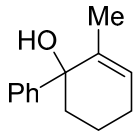
According to the general procedure for the preparation of racemic **5**, (±)-**5a** was obtained in 97% yield as a white solid. MP 44–45 °C (lit.[2] mp 44–45 °C). Its spectroscopic data were in good agreement with data reported previously.[2]

1-Butylcyclohex-2-enol [(±)-5b]



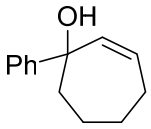
According to the general procedure for the preparation of racemic **5**, (±)-**5b** was obtained in 95% yield as a colorless oil. Its spectroscopic data were in good agreement with data reported previously.[2]

2-Methyl-1-phenylcyclohex-2-enol [(±)-5c]



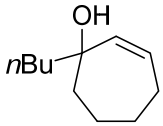
According to the general procedure for the preparation of racemic **5**, (±)-**5c** was obtained in 93% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.51-1.60 (m, 1H), 1.54 (dd, *J* = 2.0, 3.5 Hz, 3H), 1.61-1.71 (m, 1H), 1.87-2.00 (m, 2H), 2.08-2.15 (m, 2H), 5.76-5.78 (m, 1H), 7.24 (tt, *J* = 1.5, 7.5 Hz, 1H), 7.34 (br t, *J* = 7.5 Hz, 2H), 7.44 (br d, *J* = 7.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.6, 19.3, 25.7, 41.3, 75.6, 125.9, 126.7, 127.3, 128.1, 136.5, 146.6; IR (neat) ν 3460 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{13}H_{16}ONa$ $[M+Na]^+$: 211.1093. Found: 211.1096.

1-Phenylcyclohept-2-enol [(±)-5d]



According to the general procedure for the preparation of racemic **5**, (±)-**5d** was obtained in 98% yield as a white solid. MP 42-43 °C (lit.[3] mp 42-43 °C). Its spectroscopic data were in good agreement with data reported previously.[3]

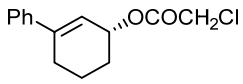
1-Butylcyclohept-2-enol [(±)-5e]



According to the general procedure for the preparation of racemic **5**, (±)-**5e** was obtained in 92% yield as a colorless oil. Its spectroscopic data were in good agreement with data reported previously.[4]

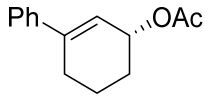
Dynamic kinetic resolution of (±)-5 to provide optically active esters 7

(*R*)-3-Phenylcyclohex-2-enyl chloroacetate [(*R*)-7aD] (Table 1, entry 6): A typical procedure



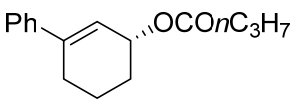
Under an argon atmosphere, *Candida antartica* lipase B (CAL-B) immobilized on a support (commercial name: Chirazyme L-2 C4; 300 mg, 3.0 w/w), V-MPS4 (29 mg, 5.7 μmol of the vanadium component) and vinyl chloroacetate (**3D**) (0.12 mL, 1.5 mmol) were added to a solution of (±)-**5a** (100 mg, 0.57 mmol) in MeCN (7.2 mL, 0.08 M) in this order at room temperature. The reaction mixture was stirred at 35 ºC for 24 h and filtered through a Celite pad. The Celite pad was washed with EtOAc, and the combined filtrate was concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, hexanes/EtOAc = 10:1) to give (*R*)-3-phenylcyclohex-2-enyl chloroacetate [(*R*)-**7aD**] (143 mg, 99% yield, 99% ee) as a colorless oil. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL AD-3 column (hexanes/2-propanol = 99:1, 1.0 mL/min; retention times 9.9 (*S*), 10.9 min (*R*)). $[\alpha]_D^{20}$ = +90 (*c* 1.16, $CHCl_3$). ^{1}H NMR (400 MHz, CD_3OD) δ 1.78-1.97 (m, 4H), 2.35-2.57 (m, 2H), 4.20 (s, 2H), 5.46-5.51 (m, 1H), 6.04-6.09 (m, 1H), 7.25 (tt, *J* = 1.5, 7.0 Hz, 1H), 7.28-7.34 (m, 2H), 7.40 (dt, *J* = 1.5, 7.0 Hz, 2H); ^{13}C NMR (100 MHz, CD_3OD) δ 21.1, 29.3, 29.7, 42.8, 73.0, 123.0, 127.4, 129.7, 130.3, 143.2, 145.5, 169.8; IR (neat) ν 1748 cm^{-1}. HRMS (FAB) *m*/*z* calcd for $C_{14}H_{15}ClO_2Na$ $[M+Na]^+$: 273.0658. Found: 273.0667.

(*R*)-3-Phenylcyclohex-2-enyl acetate [(*R*)-7aA] (Table 1, entry 1)



(*R*)-**7aA** (92% yield, 96% ee) was obtained from (±)-**5a** and **3A**. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL AD-3 column (hexanes/2-propanol = 99:1, 1.0 mL/min; retention times 8.1 (*R*), 8.7 min (*S*)). A colorless oil. $[\alpha]_D^{20}$ = +189 (*c* 0.76, $CHCl_3$) (lit.[5b] $[\alpha]_D^{22}$ = +157.9 (*c* 0.41, $CHCl_3$) for (*R*)-**7aA** (99% ee)). The spectroscopic data (^{1}H NMR, IR) of the obtained product (*R*)-**7aA** were in agreement with those in our previous publications.[5]

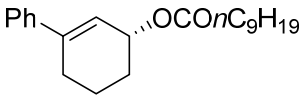
(*R*)-3-Phenylcyclohex-2-enyl butyrate [(*R*)-7aB] (Table 1, entry 3)



(*R*)-**7aB** (95% yield, 99% ee) was obtained from (±)-**5a** and **3B**. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL AD-3 column (hexanes/2-propanol = 99:1, 1.0 mL/min; retention times 5.9 (*R*), 6.6 min (*S*)).

A colorless oil. $[\alpha]_D^{20}$ = +154 (*c* 0.93, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.96 (t, *J* = 7.5 Hz, 3H), 1.68 (sext, *J* = 7.5 Hz, 2H), 1.73-1.85 (m, 2H), 1.86-1.98 (m, 2H), 2.30 (t, *J* = 7.5 Hz, 2H), 2.34-2.43 (m, 1H), 2.49-2.57 (m, 1H), 5.45-5.49 (m, 1H), 6.08 (td, *J* = 1.5, 4.0 Hz, 1H), 7.27 (br t, *J* = 7.5 Hz, 1H), 7.33 (br t, *J* = 7.5 Hz, 2H), 7.41 (br d, *J* = 7.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.7, 18.6, 19.4, 27.3, 28.0, 36.6, 68.6, 122.4, 125.4, 127.6, 128.3, 141.1, 142.0, 173.5; IR (neat) ν 1728 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{16}H_{21}O_2Na$ $[M+Na]^+$: 245.1536. Found: 245.1542.

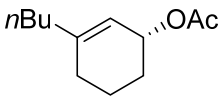
(*R*)- 3-Phenylcyclohex-2-enyl decanoate [(*R*)-7aC] (Table 1, entry 5)



(*R*)-**7aC** (97% yield, 98% ee) was obtained by conducting DKR of (±)-**5a** with **3C**, CAL-B (1.0 w/w) and V-MPS4 (0.5 mol %). Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL AD-3 column (hexanes/2-propanol = 99:1, 0.5 mL/min; retention times 9.6 (*R*), 10.7 min (*S*)).

A colorless oil. $[\alpha]_D^{20}$ = +104 (*c* 1.05, $CHCl_3$) (lit.[2] $[\alpha]_D^{20}$ = +102.1 (*c* 0.31, $CHCl_3$) for (*R*)-**7aC** (99% ee)). The spectroscopic data (^{1}H NMR) of the obtained product **7aC** were in good agreement with those in a previous publication, in which (*R*)-**7aC** (99% ee) was obtained in 72% yield.[2]

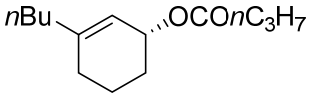
(*R*)- 3-Butylcyclohex-2-enyl acetate [(*R*)-7bA] (Table 1, entry 9)



(*R*)-**7bA** (79% yield, 90% ee) was obtained by conducting DKR of (±)-**5b** with **3A**, CAL-B (1.0 w/w) and V-MPS4 (0.5 mol %) at –10 °C for 8 days. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL AD-3 column (hexanes only, 0.5 mL/min, 208 nm; retention times 12.4 (*R*), 13.3 min (*S*)).

A colorless oil. $[\alpha]_D^{20}$ = +160 (*c* 0.90, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, *J* = 7.5 Hz, 3H), 1.79 (sext, *J* = 7.5 Hz, 2H), 1.34-1.45 (m, 2H), 1.58-1.84 (m, 4H), 1.87-2.03 (m, 4H), 2.04 (s, 3H), 5.23-5.29 (m, 1H), 5.43-5.46 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 19.2, 21.5, 22.4, 28.3, 28.4, 29.6, 37.4, 69.0, 119.2, 145.0, 170.9; IR (neat) ν 1732 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{12}H_{20}O_2Na$ $[M+Na]^+$: 219.1356. Found: 219.1355.

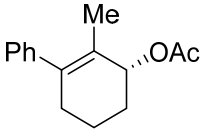
(*R*)- 3-Butylcyclohex-2-enyl butyrate [(*R*)-7bB] (Table 1, entry 10)



(*R*)-**7bB** (93% yield, 96% ee) was obtained from (±)-**5b** and **3B**. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL OD-3 column (hexanes only, 1.0 mL/min, 208 nm; retention times 6.3 (*R*), 7.5 min (*S*)).

A colorless oil. $[\alpha]_D^{20}$ = +145 (*c* 1.09, $CHCl_3$) (lit.[5b] $[\alpha]_D^{27}$ = +147.7 (c 0.47, $CDCl_3$) for (*R*)-**7bB** (99% ee)). The spectroscopic data (^{1}H NMR) of the obtained product (*R*)-**7bB** were in good agreement with those in our previous publication.[5b]

(*R*)- 2-Metyl-3-phenylcyclohex-2-enyl acetate [(*R*)-7cA] (Table 1, entry 12)



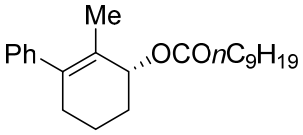
7cA (77% yield, 81% ee) was obtained by conducting DKR of (±)-**5c** with **3A**, lipase PS-IM (3.0 w/w) and V-MPS4 (1.0 mol %) in toluene at 50 °C for 24 h. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL OJ-H column (hexanes/2-propanol = 99:1, 1.0 mL/min; retention times 9.8 (*R*),

10.3 min (*S*)).

A colorless oil. $[\alpha]_D^{20}$ = +73 (*c* 0.89, MeOH). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.56 (t, *J* = 2.0 Hz, 3H), 1.69-1.95 (m, 4H), 2.11 (s, 3H), 2.19-2.38 (m, 2H), 5.34-5.39 (m, 1H), 7.16 (br d, *J* = 7.5 Hz, 2H), 7.24 (br t, *J* = 7.5 Hz, 1H), 7.34 (br t, *J* = 7.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 17.4, 18.9, 21.4, 28.9, 32.1, 71.8, 126.6, 126.9, 128.0, 128.1, 139.0, 142.9, 171.1; IR (neat) ν 1732 cm^{-1}. HRMS (FAB) *m*/*z* calcd for $C_{15}H_{18}O_2Na$ $[M+Na]^+$: 253.1204. Found: 253.1208.

(*R*)- 2-Metyl-3-phenylcyclohex-2-enyl decanoate [(*R*)-7cC] (Table 1, entry 13)

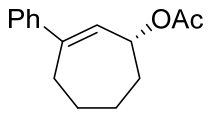
7cC (84% yield, 93% ee) was obtained by conducting DKR of (±)-**5c** with **3C**, lipase PS-IM (3.0 w/w) and V-MPS4 (1.0 mol %) in toluene at 50 °C for 24 h. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL OJ-H column (hexanes/2-propanol = 99.5:0.5, 1.0 mL/min; retention times 4.8 (*S*), 5.5 min (*R*)).



A colorless oil. $[\alpha]_D^{20}$ = +72 (*c* 0.28, MeOH). ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.87 (t, *J* = 7.0 Hz, 3H), 1.16-1.40 (m, 12H), 1.55 (t, *J* = 2.0 Hz, 3H), 1.61-1.92 (m, 6H), 2.17-2.39 (m, 4H), 5.33-5.40 (m, 1H), 7.16 (br d, *J* = 7.5 Hz, 2H), 7.24 (br t, *J* = 7.5 Hz, 1H), 7.33 (br t, *J* = 7.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 17.5, 19.0, 22.7, 25.2, 29.0, 29.16, 29.24, 29.3, 29.4, 31.8, 32.1, 34.8, 71.5, 126.5, 127.0, 128.0, 128.1, 138.9, 143.0, 173.9; IR (neat) ν 1732 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{23}H_{35}O_2$ $[M+H]^+$: 343.2632. Found: 343.2621.

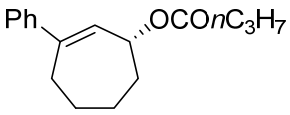
(*R*)-3-Phenylcyclohept-2-enyl acetate [(*R*)-7dA] (Table 1, entry 15)

(*R*)-**7dA** (82% yield, 98% ee) was obtained from (±)-**5d** and **3A**. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL AD-3 column (hexanes/2-propanol = 99:1, 1.0 mL/min; retention times 5.4 (*R*), 6.1 min (*S*)).



A colorless oil. $[\alpha]_D^{20}$ = +66 (*c* 0.98, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.44-1.54 (m, 1H), 1.68-1.93 (m, 4H), 1.99-2.06 (m, 1H), 2.09 (s, 3H), 2.50-2.71 (m, 2H), 5.54-5.62 (m, 1H), 5.87-5.92 (m, 1H), 7.23 (br t, *J* = 7.0 Hz, 1H), 7.27-7.35 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.3, 26.0, 27.6, 32.55, 32.61, 74.3, 125.6, 126.9, 128.2, 131.5, 143.0, 143.7, 170.3; IR (neat) ν 1736 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{15}H_{18}O_2Na$ $[M+Na]^+$: 253.1199. Found: 253.1194.

(*R*)-3-Phenylcyclohept-2-enyl butyrate [(*R*)-7dB] (Table 1, entry 16)



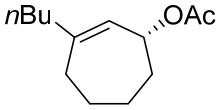
(*R*)-**7dB** (81% yield, 98% ee) was obtained from (±)-**5d** and **3B**. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL AD-3 column (hexanes/2-propanol = 99:1, 0.5 mL/min; retention times 8.8 (*S*), 10.3 min (*R*)).

A colorless oil. $[\alpha]_D^{20}$ = +50 (*c* 0.95, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.97

(t, *J* = 7.5 Hz, 3H), 1.45-1.54 (m, 1H), 1.64-1.92 (m, 6H), 1.99-2.07 (m, 1H), 2.32 (t, *J* = 7.5 Hz, 2H), 2.51-2.59 (m, 1H), 2.64-2.72 (m, 1H), 5.57-5.62 (m, 1H), 5.87-5.91 (m, 1H), 7.23 (dt, *J* = 2.0, 7.0 Hz, 1H), 7.28-7.38 (m, 4H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 13.7, 18.5, 26.0, 27.7, 32.6, 32.7, 36.5, 74.0, 125.8, 126.9, 128.2, 131.6, 143.0, 143.8, 173.0; IR (neat) ν 1732 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{17}H_{23}O_2$ $[M+H]^+$: 259.1692. Found: 259.1690.

(*R*)-3-Butylcyclohept-2-enyl acetate [(*R*)-7eA] (Table 1, entry 17)

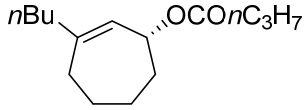
(*R*)-**7eA** (93% yield, 98% ee) was obtained from (±)-**5e** and **3A**. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL AD-3 column (hexanes/2-propanol = 99:1, 0.5 mL/min, 208 nm; retention times 7.6 (*R*), 9.4 min (*S*)).



A colorless oil. $[\alpha]_D^{20}$ = +50 (*c* 0.88, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89 (t, *J* = 7.0 Hz, 3H), 1.23-1.39 (m, 5H), 1.57-1.71 (m, 3H), 1.76-1.82 (m, 1H), 1.86-1.99 (m, 3H), 2.03-2.15 (m, 2H), 2.05 (s, 3H), 5.28-5.45 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 21.4, 22.3, 26.0, 27.2, 29.8, 32.4, 32.9, 39.8, 74.0, 127.1, 143.9, 170.5; IR (neat) ν 1736 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{13}H_{22}O_2Na$ $[M+Na]^+$: 233.1512. Found: 233.1505.

(*R*)-3-Butylcyclohept-2-enyl butyrate [(*R*)-7eB] (Table 1, entry 18)

(*R*)-**7eB** (91% yield, >99% ee) was obtained from (±)-**5e** and **3B**. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL AD-3 column (hexanes/2-propanol = 99.5:0.5, 1.0 mL/min, 208 nm; retention times 5.3 (*R*), 6.5 min (*S*)).



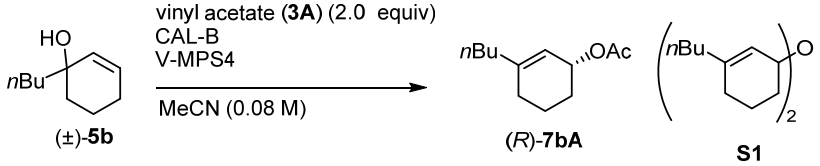
A colorless oil. $[\alpha]_D^{20}$ = +47 (*c* 0.96, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89 (t, *J* = 7.5 Hz, 3H), 0.95 (t, *J* = 7.5 Hz, 3H), 1.24-1.40 (m, 5H), 1.59-1.71 (m, 5H), 1.76-1.82 (m, 1H), 1.87-1.99 (m, 3H), 2.04-2.18 (m, 2H), 2.28 (t, *J* = 7.5 Hz, 2H), 5.34-5.41 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.7, 14.0, 18.5, 22.4, 26.0, 27.2, 29.8, 32.4, 32.9, 36.6, 39.8, 73.7, 127.2, 144.0, 173.1; IR (neat) ν 1732 cm^{-1}.HRMS (MALDI) *m*/*z* calcd for $C_{15}H_{27}O_2$ $[M+H]^+$: 239.2006. Found: 239.1999.

The optimization of the reaction conditions of DKR of (±)-5b using vinyl acetate (3A)

At first, the effect of the amount of V-MPS4 and CAL-B on the optical purity and yield of the ester (*R*)-**7bA** was examined to find the following results: The use of 5 mol % of V-MPS4 at 35 °C resulted in the decrease of the yield of (*R*)-**7bA**, while maintaining the optical purity (80% ee), due to the formation of a significant amount of the dimeric ether **S1** (Table S1, entry 2). The use of a less amount (1 w/w) of CAL-B increased the optical purity to 86% ee although the chemical yield (60%) was decreased (entry 3). Therefore, 3 w/w of CAL-B and 1 mol % of V-MPS4 was found to be a good combination.

Next, we investigated the temperature effect and found that the reaction at either higher temperature (50 °C) or lower temperature (10 °C) produced improvement of the optical purity of (*R*)-**7bA**, among which the reaction at 10 °C was more suitable for affording a higher yield of (*R*)-**7bA** albeit longer reaction time (48 h) (entries 4 and 5). The use of less amounts of V-MPS4 (1 w/w) and CAL-B (0.5 mol %) brought increase in both optical purity (88% ee) and chemical yield (92%), although the reaction was slow and needed longer time (72 h) (entry 6). The optical purity was further improved to 92% ee by performing the reaction at lower temperature (–10 °C). After 72 h, (*R*)-**7bA** was obtained in 65% yield along with racemic 3-butylcyclohex-2-enol (28% yield), which showed that V-MPS4 was active at such low temperature (entry 7). Finally, (*R*)-**7bA** (90% ee) was obtained in 79% yield by the reaction at –10 °C for 192 h (entry 8).

Table S1 The optimization of the reaction conditions of DKR of (±)-**5b** using vinyl acetate (**3A**).



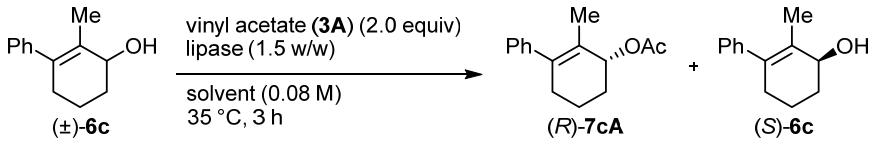
entry	amount of CAL-B	equiv of V-MPS4	temp.	reaction time	(*R*)-**7bA**		yield of **S1**
					isolated yield	optical purity[a]	
1[b]	3 w/w	1 mol %	35 °C	24 h	90%	80% ee	5%
2	3 w/w	5 mol %	35 °C	24 h	33%	80% ee	28%
3	1 w/w	1 mol %	35 °C	24 h	60%	86% ee	7%
4	3 w/w	1 mol %	50 °C	24 h	70%	83% ee	21%
5	3 w/w	1 mol %	10 °C	48 h	85%	85% ee	8%
6	1 w/w	0.5 mol %	10 °C	72 h	92%	88% ee	7%
7[c]	1 w/w	0.5 mol %	–10 °C	72 h	65%	92% ee	3%
8	1 w/w	0.5 mol %	–10 °C	192 h	79%	90% ee	8%

a) Determined by HPLC using a chiral column.
b) Cited from entry 7 of Table 1.
c) Racemic 3-butylcyclohex-2-enol was recovered in 28% yield.

The optimization of the reaction conditions of DKR of (±)-5c using vinyl acetate (3A)

At first, some commercially available immobilized lipases were examined for the kinetic resolution of racemic secondary alcohol **6c** to find that *Candida antarctica* lipase B (CAL-B) and *Burkholderia cepacia* lipases (PS-CII and PS-IM) were effective (Table S2). In particular, the use of PS-IM in a range of organic solvents produced high enantioselectivity (E value: up to 140) (entries 5-8).

Table S2 The screening of lipases and solvents for kinetic resolution of (±)-**6c**

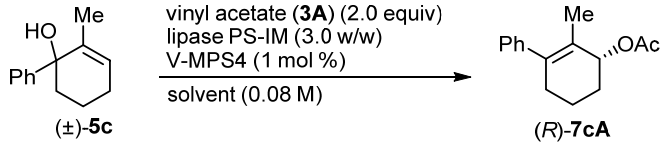


entry	lipase	solvent	conv.[a]	optical purity of (*S*)-**6c**	E value
1	CAL-B	MeCN	43%	48% ee	7.3
2	CAL-B	hexane	68%	91% ee	7.4
3[b]	PS-CII	MeCN	54%	89% ee	24
4[b]	PS-CII	hexane	60%	>99% ee	30
5	PS-IM	MeCN	49%	91% ee	140
6	PS-IM	hexane	61%	>99% ee	23
7	PS-IM	*i*Pr_2O	53%	>99% ee	97
8	PS-IM	toluene	54%	>99% ee	70

a) Determined by ^{1}H NMR. b) Conducted for 1.5 h.

Next, DKR was examined by using PS-IM and V-MPS4 in various organic solvents at 35 °C (Table S3, entries 1-6), in which DKR in toluene afforded the best results in terms of the yield (84%) and optical purity (71% ee) of the ester **7cA** (entry 3). A similar reaction in toluene at 50 °C provided **7cA** with better optical purity (81% ee) albeit in lower yield (77%) (entry 7); however, similar tendency was not observed in CH_2Cl_2 (entry 8). The use of vinyl decanoate (**3C**) instead of vinyl acetate (**3A**) effected improvement of both yield (84%) and optical purity (93% ee) of the corresponding ester (*R*)-**7cC** (entry 9).

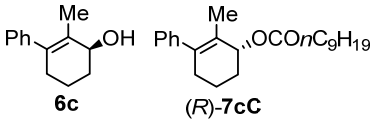
Table S3 The optimization of the reaction conditions of DKR of **5c**



(±)-**5c** (*R*)-**7cA**

entry	solvent	temp.	reaction time	(*R*)-**7cA**	
				isolated yield	optical purity
1	hexane	35 °C	24 h	92%	61% ee
2[a]	hexane	35 °C	48 h	87%	63% ee
3	toluene	35 °C	48 h	84%	71% ee
4	iPr_2O	35 °C	48 h	77%	50% ee
5	c-C_5H_9OMe	35 °C	72 h	58%[b]	73% ee
6	CH_2Cl_2	35 °C	48 h	62%	73% ee
7	toluene	50 °C	24 h	77%	81% ee
8	CH_2Cl_2	50 °C	24 h	49%	60% ee
9[c]	toluene	50 °C	24 h	84%	93% ee

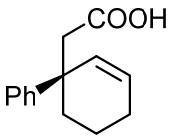
a) PS-IM (1.5 w/w) was used. b) Alcohol **6c** (88% ee) was recovered in 34% yield. c) Vinyl decanoate (**3C**) was used instead of vinyl acetate. The isolated yield and optical purity of (*R*)-**7cC** are shown.



6c (*R*)-**7cC**

The Ireland–Claisen rearrangement of optically active esters 7

(*S*)-(1-Phenylcyclohex-2-enyl)acetic acid (4aA) (Table 2, entry 1): A typical procedure

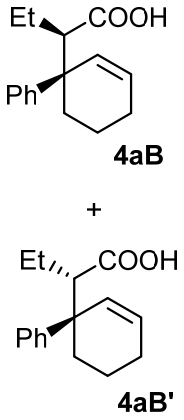


Under an argon atmosphere, HMPA (0.13 mL, 5% of total volume of THF) was added to a solution of LDA (0.55 mmol) in THF (1.0 mL) at –78 °C. Then, a solution of (*R*)-**7aA** (96% ee, 100 mg, 0.46 mmol) in THF (1.0 mL) was added via a cannula. After the reaction mixture was stirred at the same temperature for 15 min, a solution of TBSCl (105 mg, 0.69 mmol) in THF (0.5 mL) was added. After being stirred at –78 °C for 15 min, the reaction mixture was warmed to room temperature over 1 h and then refluxed for 15 h. After cooling to room temperature, aqueous hydrochloric acid (1.0 M, 1.0 mL) was added to the solution, and the mixture was stirred at room temperature for 3 h. Et_2O (5 mL) was added, and the organic phase was separated. The aqueous phase was extracted with Et_2O (2 x 5 mL). The combined organic phases were washed with brine (5 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, hexanes/EtOAc = 3:1) to give (*S*)-**4aA** (79 mg, 79%, 96% ee). Its optical purity was determined by HPLC analysis of the alcohol **S2** obtained by the reduction of (*S*)-**4aA** (vide infra).

(*S*)-**4aA**. A white solid. Mp 87-88 °C. $[\alpha]_D^{20}$ = +39 (*c* 0.86, $CHCl_3$). ^1H NMR (500 MHz, $CDCl_3$) δ 1.30-1.41 (m, 1H), 1.51-1.58 (m, 1H), 1.87 (ddd, *J* = 3.0, 11.5, 13.0 Hz, 1H), 1.95 (ddd, *J* = 2.5, 6.5, 13.0 Hz, 1H), 1.99-2.08 (m, 2H), 2.73 (d, *J* = 14.0 Hz, 1H), 2.83 (d, *J* = 14.0 Hz, 1H), 5.95 (td, *J* = 3.5, 10.0 Hz, 1H), 6.07 (ddd, *J* = 2.5, 3.5, 10.0 Hz, 1H), 7.18 (dt, *J* = 1.5, 7.5 Hz, 1H), 7.30 (br t, *J* = 7.5 Hz, 2H), 7.34 (br d, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 18.6, 25.0, 37.0, 41.7, 46.4, 126.0, 126.7, 128.0, 128.8, 131.8, 146.6,

176.8; IR (neat) ν 1713, 3024 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{14}H_{16}O_2Na$ $[M+Na]^+$: 239.1043. Found: 239.1043.

(*R*)-2-[(*R*)-1-Phenylcyclohex-2-enyl]butanoic acid (4aB) and (*S*)-2-[(*R*)-1-phenylcyclohex-2-enyl]-butanoic acid (4aB') **(Table 2, entry 2)**

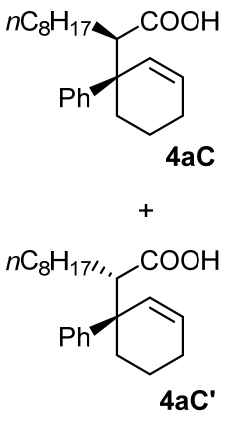


4aB (63% yield, 99% ee) and **4aB'** (6% yield, optical purity was not determined) were obtained from (*R*)-**7aB** (99% ee) by column chromatography (silica gel, hexanes/EtOAc = 7:1).

4aB. A white solid. Mp 145-146 °C. $[\alpha]_D^{20}$ = +28 (*c* 0.96, $CHCl_3$). ^1H NMR (500 MHz, $CDCl_3$) δ 0.82 (t, *J* = 7.0 Hz, 3H), 1.14-1.28 (m, 2H), 1.49-1.57 (m, 1H), 1.63-1.74 (m, 1H), 1.89 (dt, *J* = 2.0, 13.0 Hz, 1H), 1.94-2.06 (m, 2H), 2.20 (dt, *J* = 5.0, 6.5 Hz, 1H), 2.71 (dd, *J* = 3.0, 13.0 Hz, 1H), 5.96 (td, *J* = 3.0, 11.0 Hz, 1H), 6.01 (ddd, *J* = 1.5, 3.5, 11.0 Hz, 1H), 7.19 (t, *J* = 7.0 Hz, 1H), 7.28-7.37 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 12.7, 18.8, 20.8, 25.4, 33.9, 45.6, 58.8, 126.0, 127.3, 128.1, 129.3, 130.0, 146.3, 180.1; IR (neat) ν 1704, 3025 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{16}H_{20}O_2Na$ $[M+Na]^+$: 267.1356. Found: 267.1350. Its optical purity was determined by the HPLC analysis of the alcohol **S3** obtained by the reduction of (*S*)-**4aB** (vide infra) and its relative configuration was determined by the derivatization of **4aB** into the iodo lactone **S5** (vide infra).

4aB'. A colorless oil. $[\alpha]_D^{20}$ = +72 (*c* 1.07, $CHCl_3$). ^1H NMR (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 7.0 Hz, 3H), 1.21-1.34 (m, 2H), 1.49-1.69 (m, 2H), 1.90-2.04 (m, 4H), 2.63 (dd, *J* = 3.5, 11.0 Hz, 1H), 5.89-5.93 (m, 1H), 6.20-6.24 (m, 1H), 7.15 (br t, *J* = 7.5 Hz, 1H), 7.26 (br t, *J* = 7.5 Hz, 2H), 7.33 (br d, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 12.8, 18.7, 21.4, 25.2, 32.4, 45.1, 57.7, 126.0, 127.5, 127.8, 129.0, 130.8, 145.9, 178.9; IR (neat) ν 1703, 3031 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{16}H_{20}O_2Na$ $[M+Na]^+$: 267.1356. Found: 267.1353.

(*R*)-2-[(*R*)-1-Phenylcyclohex-2-enyl]decanoic acid (4aC) and (*S*)-2-[(*R*)-1-phenylcyclohex-2-enyl]-decanoic acid (**4aC'**) **(Table 2, entry 3)**



An 11:1 mixture of **4aC** and **4aC'** was obtained from (*R*)-**7aC** (98% ee), whose ratio was determined by ^1H NMR analysis of a crude product. Pure **4aC** (77% yield) was isolated by column chromatography (silica gel, hexanes/EtOAc = 7:1).

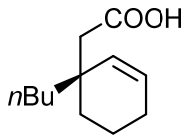
4aC. A colorless oil. $[\alpha]_D^{20}$ = +28 (*c* 1.21, $CHCl_3$). ^1H NMR (500 MHz, $CDCl_3$) δ 0.85 (t, *J* = 7.0 Hz, 3H), 1.04-1.33 (m, 14H), 1.48-1.57 (m, 1H), 1.62-1.74 (m, 1H), 1.82-2.06 (m, 3H), 2.20 (br d, *J* = 14.0 Hz, 1H), 2.78 (br d, *J* = 12.0 Hz, 1H), 5.93-6.03 (m, 2H), 7.19 (br t, *J* = 7.5 Hz, 1H), 7.28-7.36 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 18.8, 22.6, 25.3, 27.4, 28.2, 29.16, 29.22, 29.3, 31.8, 33.7, 45.6, 56.9, 126.0, 127.3, 128.0, 129.2, 130.1, 146.2, 181.1; IR (neat) ν 1701, 3028 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{16}H_{20}O_2Na$ $[M+Na]^+$: 267.1356. Found:

267.1350.

4aC'. Some typical ^{1}H NMR data (500 MHz, $CDCl_3$) δ 5.89-5.93 (m, 1H), 6.24-6.29 (m, 1H).

(*R*)-2-(1-Butylcyclohex-2-enyl)acetic acid (4bA) (Table 2, entry 4)

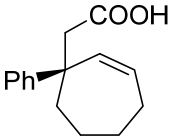
(*R*)-**4bA** (78% yield) was obtained from (*R*)-**7bA** (90% ee).



A colorless oil. $[\alpha]_D^{20}$ = +2.4 (*c* 1.04, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, *J* = 7.0 Hz, 3H), 1.18-1.34 (m, 4H), 1.37-1.71 (m, 6H), 1.87-2.02 (m, 2H), 2.33 (d, *J* = 16.0 Hz, 1H), 2.36 (d, *J* = 16.0 Hz, 1H), 5.53 (td, *J* = 2.0, 10.0 Hz, 1H), 5.70 (td, *J* = 3.5, 10.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 18.8, 23.4, 24.9, 26.0, 32.5, 36.8, 39.6, 44.0, 127.1, 133.9, 178.4; IR (neat) ν 1709, 3020 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{12}H_{20}O_2Na$ $[M+Na]^+$: 219.1356. Found: 219.1360.

(*S*)-2-(1-Phenylcyclohept-2-enyl)acetic acid (4dA) (Table 2, entry 6)

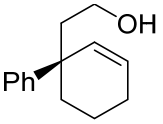
(*S*)-**4dA** (63% yield, 98% ee) was obtained from (*R*)-**7dA** (99% ee) by conducting Ireland-Claisen rearrangement with HMPA, $KN(SiMe_3)_2$ and TBSCl in THF. Its optical purity was determined by the HPLC analysis of the alcohol **S4** obtained by the reduction of (*S*)-**4dA** (vide infra).



A white solid. Mp 110 °C. $[\alpha]_D^{20}$ = +90 (*c* 1.00, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.29-1.53 (m, 3H), 1.58-1.70 (m, 1H), 1.89-2.21 (m, 4H), 2.68 (d, *J* = 14.0 Hz, 1 H), 2.70 (d, *J* = 14.0 Hz, 1 H), 6.00-6.04 (m, 2H), 7.20 (tt, *J* = 1.5, 7.5 Hz, 1H), 7.31 (br t, *J* = 7.5 Hz, 2H), 7.37 (br d, *J* = 7.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.0, 26.3, 27.9, 38.6, 47.5, 49.6, 126.0, 126.9, 128.2, 132.6, 136.2, 144.7, 177.0; IR (neat) ν 1705, 3021 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{15}H_{18}O_2Na$ $[M+Na]^+$: 253.1199. Found: 253.1194.

The determination of the optical purity of (*S*)-4aA, (*R*,*R*)-4aB and (*S*)-4dA

(*S*)-2-(1-Phenylcyclohex-2-enyl)ethanol (S2)



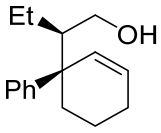
A solution of (*S*)-**4aA** (10 mg, 0.046 mmol) in THF (1.0 mL) was added to a suspension of $LiAlH_4$ (1.8 mg, 0.046 mmol) in THF (1.0 mL) at 0 °C. Then, the reaction mixture was stirred at 60 °C for 2 h, cooled to 0 °C, and quenched with aqueous hydrochloric acid (1.0 M, 1.0 mL). The aqueous phase was extracted with Et_2O (3 x 1.0 mL), and the combined organic phases were washed with brine (1.0 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, hexanes/EtOAc = 3:1) to give **S2** (9.4 mg, quantitative yield). Its optical purity was determined by HPLC

analysis at 20 °C, using a CHIRALCEL IE column (hexanes/2-propanol = 95:5, 1.0 mL/min, 208 nm; retention times 11.0 (*R*), 11.7 min (*S*)).

A colorless oil. $[\alpha]_D^{20}$ = +62 (*c* 1.00, $CHCl_3$). ^1H NMR (400 MHz, $(CD_3)_2SO$) δ 1.14-1.28 (m, 1H), 1.45-1.57 (m, 1H), 1.63-1.73 (m, 1H), 1.79-2.04 (m, 5H), 3.12-3.19 (m, 1H), 3.26-3.30 (m, 1H), 5.81-5.92 (m, 2H), 7.13-7.19 (m, 1H), 7.26-7.34 (m, 4H); ^{13}C NMR (100 MHz, $(CD_3)_2SO$) δ 18.5, 24.9, 36.5, 41.3, 45.1, 57.4, 126.0, 126.7, 127.3, 128.1, 133.2, 147.8; IR (neat) ν 3337 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{14}H_{18}ONa$ $[M+Na]^+$: 225.1250. Found: 225.1251.

(*R*)-2-[(*R*)-1-Phenylcyclohex-2-enyl)butanol (S3)

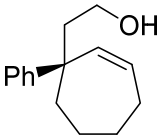
According to the procedure of preparation of **S2**, **S3** was obtained from (*R,R*)-**4aB** in 99% yield. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL IC-3 column (hexanes/2-propanol = 97.5:2.5, 1.0 mL/min, 208 nm; retention times 7.5 (*R,R*), 8.2 min (*S,S*)).



A colorless oil. $[\alpha]_D^{20}$ = +109 (*c* 0.50, $CHCl_3$). ^1H NMR (500 MHz, $CDCl_3$) δ 0.85 (t, *J* = 7.5 Hz, 3H), 1.16-1.37 (m, 3H), 1.51-1.58 (m, 1H), 1.68-1.74 (m, 1H), 1.92-2.09 (m, 4H), 3.745 (dd, *J* = 4.0, 11.5 Hz 1H), 3.754 (dd, *J* = 4.0, 11.5 Hz, 1H), 5.85-5.94 (m, 2H), 7.12 (tt, *J* = 2.5, 6.5 Hz, 1H), 7.28-7.34 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.5, 18.8, 20.2, 25.6, 32.9, 46.5, 52.9, 62.6, 125.6, 127.3, 128.0, 129.6, 132.2, 147.9; IR (neat) ν 3375 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{16}H_{22}ONa$ $[M+Na]^+$: 253.1563. Found: 253.1562.

(*S*)-2-(1-Phenylcyclohept-2-enyl)ethan-1-ol (S4)

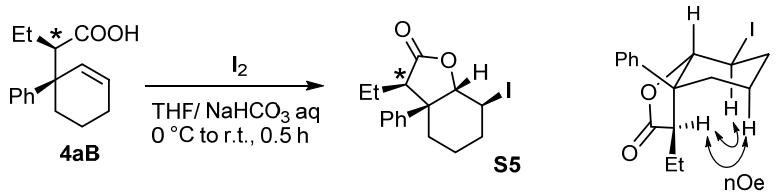
According to the procedure of preparation of **S2**, **S4** was obtained from (*S*)-**4dA** in 99% yield. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL OJ-H column (hexanes/2-propanol = 95:5, 1.0 mL/min, 208 nm; retention times 10.7 (*R*), 19.7 min (*S*)).



A colorless oil. $[\alpha]_D^{20}$ = +115 (*c* 0.88, $CHCl_3$). ^1H NMR (400 MHz, $(CD_3)_2SO$) δ 1.16-1.43 (m, 3H), 1.50-1.63 (m, 1H), 1.70-1.94 (m, 4H), 2.00-2.15 (m, 2H), 3.07-3.17 (m, 1H), 3.30-3.43 (m, 1H), 5.76-5.82 (m, 1H), 5.89-5.97 (m, 1H), 7.12-7.20 (m, 1H), 7.26-7.34 (m, 4H); ^{13}C NMR (100 MHz, $(CD_3)_2SO$) δ 25.6, 27.0, 28.4, 39.2, 48.0, 49.2, 58.6, 126.4, 127.6, 129.0, 132.4, 138.6, 146.9; IR (neat) ν 3345 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{15}H_{20}ONa$ $[M+Na]^+$: 239.1406. Found: 239.1406.

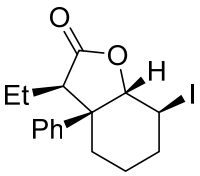
The determination of the relative configuration of the major isomer 4aB

The relative configuration of the major isomer **4aB** was determined based on the nOe experiment of the iodo lactone **S5** obtained by the reaction with iodine under basic reaction conditions (Scheme S1).



Scheme S1. Determination of the relative configuration of **4aB**.

(3*R*,3a*R*,7*S*,7a*S*)-3-Ethyl-7-iodo-3a-phenylhexahydrobenzofuran-2(3*H*)-one (S5)

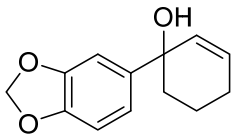


Iodine (20 mg, 0.082 mmol) was added to a solution of **4aB** (10 mg, 0.041 mmol) in THF/aq. $NaHCO_3$ (1:1, 1.0 mL) at 0 °C. Then, the solution was warmed to room temperature over 30 min and quenched with saturated aq. $Na_2S_2O_3$ (1.0 mL). The aqueous phase was extracted with EtOAc (3 x 5.0 mL), and the combined organic phases were washed with brine (1.0 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, hexanes/EtOAc = 5:1) to give **S5** (11.8 mg, 78%).

A white solid. Mp 129-130 °C. $[\alpha]_D^{20}$ = +65 (*c* 0.90, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.95-1.13 (m, 5H), 1.50-1.58 (m, 1H), 1.63-1.72 (m, 1H), 1.83-1.97 (m, 2H), 2.34-2.41 (m, 1H), 2.48-2.54 (m, 1H), 2.60 (dd, *J* = 5.0, 8.0 Hz, 1H), 4.07 (ddd, *J* = 4.5, 9.0, 13.0 Hz, 1H), 4.96 (d, *J* = 9.0 Hz, 1H), 7.21 (br t, *J* = 6.5 Hz, 1H), 7.23-7.32 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 12.5, 19.4, 23.3, 28.1, 31.3, 36.0, 47.2, 50.5, 88.4, 126.2, 127.4, 128.7, 142.0, 176.5; IR (neat) ν 1779 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{16}H_{19}O_2NaI$ $[M]^+$: 393.0322. Found: 393.0321.

Asymmetric total synthesis of (–)-crinane

1-(3,4-methylenedioxyphenyl)cyclohex-2-enol [(±)-5f]



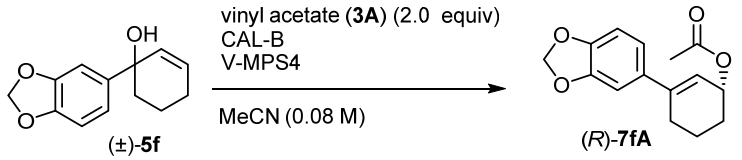
Under an argon atmosphere, *n*BuLi (2.6 M in hexane; 12 mL, 31 mmol) was added to a solution of 4-bromo-1,2-metylenedioxybenzene (6.3 g, 31 mmol) in Et_2O (60 mL) and THF (30 mL) at –78 °C. After 30 min at –78 °C, a solution of cyclohex-2-enone (2.5 g, 26 mmol) in THF (10 mL) was added. The reaction mixture was warmed to room temperature over 15 min and quenched with saturated aq. NH_4Cl (70 mL). The organic phase was separated and the aqueous phase was extracted with EtOAc (3 x 50 mL). The combined organic phases were washed with brine (50 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, hexanes/EtOAc = 5:1) to give **5f** (5.5 g, 96%).

A white solid. Mp 52-53 °C (lit.[6] mp 55-56 °C). The ^{1}H NMR data of the obtained product **5f** was in good agreement with those in a previous publication.[6]

The optimization of the reaction conditions of DKR of (±)-5f using vinyl acetate (3A)

At first, the DKR of (±)-**5f** (50 mg) was conducted under standard conditions at 35 °C for 24 h to give the ester (*R*)-**7fA** with 89% ee in 79% yield (Table S4, entry 1). A similar reaction performed at –5 °C by using CAL-B (1.0 w/w) and V-MPS4 (0.5 mol %) provided the increase of both yield (87%) and optical purity (95% ee) of (*R*)-**7fA** (entry 2). At lower temperature (–10 and –20 °C) the optical purity of (*R*)-**5fA** was further improved to 97–98% ee (entries 3 and 4). A similar reaction of 1 g of (±)-**5f** also afforded the same chemical and optical yields of (*R*)-**7fA** (entry 5).

Table S4 The optimization of the reaction conditions of DKR of (±)-**5f** using vinyl acetate (**3A**)

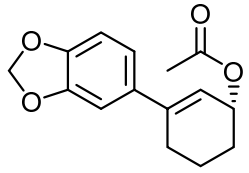


(±)-**5f** (*R*)-**7fA**

entry[a)]	amount of CAL-B	equiv of V-MPS4	temp.	reaction time	(*R*)-**7fA**	
					isolated yield	optical purity
1	3.0 w/w	1.0 mol %	35 °C	24 h	79%	89% ee
2	1.0 w/w	0.5 mol %	–5 °C	65 h	87%	95% ee
3	1.0 w/w	0.5 mol %	–10 °C	72 h	88%	97% ee
4	1.0 w/w	0.5 mol %	–20 °C	78 h	88%	98% ee
5[b]	1.0 w/w	0.5 mol %	–30 °C	120 h	89%	97% ee

a) The reaction was conducted by using 50 mg of (±)-**5f**, otherwise noted.
b) One gram of (±)-**5f** was used.

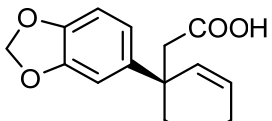
(*R*)-3-(3,4-methylenedioxyphenyl)cyclohex-2-enyl acetate [(*R*)-7fA]



Under an argon atmosphere, CAL-B (100 mg), V-MPS4 (11 mg, 2.3 μmol of the vanadium component) and vinyl acetate (85 μL, 0.92 mmol) were added to a solution of (±)-**5f** (100 mg, 0.46 mmol) in MeCN (5.7 mL, 0.08 M) in this order at –20 °C. The reaction mixture was stirred at –20 ºC for 78 h and then filtered through a Celite pad. The Celite pad was washed with EtOAc, and the combined filtrate was concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, hexanes/EtOAc = 7:1) to give (*R*)-**7fA** (105 mg, 88% yield, 98% ee). Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL AD-3 column (hexanes/2-propanol = 99:1, 1.0 mL/min; retention times 22.9 (*R*), 24.6 min (*S*)).

A colorless oil. $[\alpha]_D^{20}$ = +144 (*c* 0.99, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 1.69-1.98 (m, 4H), 2.07 (s, 3H), 2.27-2.36 (m, 1H), 2.42-2.51 (m, 1H), 5.40-5.46 (m, 1H), 5.93-6.00 (m, 3H), 6.76 (d, *J* = 8.0 Hz, 1H), 6.88 (d, *J* = 2.0 Hz, 1H), 6.91 (dd, *J* = 2.0, 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.4, 21.5, 27.6, 28.0, 68.9, 101.0, 106.0, 108.0, 119.0, 121.2, 135.4, 141.6, 147.1, 147.7, 170.9; IR (neat) ν 1729 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{15}H_{16}O_4$ $[M]^+$: 260.1043. Found: 260.1043.

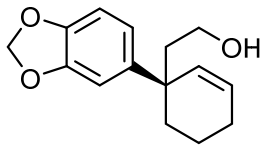
(*S*)-[1-(3,4-methylenedioxyphenyl)cyclohex-2-enyl]acetic acid [(*S*)-4fA]



Similarly to the preparation of (S)-**4aA**, (*S*)-**4fA** was obtained in 70% yield.

A white solid. Mp 118-119 °C. $[\alpha]_D^{20}$ = +43 (*c* 0.91, $CHCl_3$). ^1H NMR (500 MHz, $CDCl_3$) δ1.31-1.41 (m, 1H), 1.50-1.57 (m, 1H), 1.78-1.91 (m, 2H), 1.96-2.04 (m, 2H), 2.67 (d, *J* = 15.0 Hz, 1H), 2.77 (d, *J* = 15.0 Hz, 1H), 5.89-5.95 (m, 3H), 6.03 (dd, *J* = 1.0, 10.0 Hz, 1H), 6.73 (d, *J* = 8.0 Hz, 1H), 6.78 (dd, *J* = 2.0, 8.0 Hz, 1H), 6.84 (d, *J* = 2.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 18.5, 25.0, 37.3, 41.5, 46.5, 100.9, 107.5, 107.7, 120.0, 128.9, 131.7, 140.6, 145.6, 147.4, 177.1; IR (neat) ν 3021, 1705 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{15}H_{16}O_4$ $[M]^+$: 260.1043. Found: 260.1045.

(*S*)-2-[1-(3,4-methylenedioxyphenyl)cyclohex-2-enyl]ethanol [(*S*)-8]

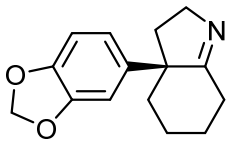


Similarly to the preparation of **S2**, (*S*)-**8** was obtained in quantitative yield with 98% ee. Its optical purity was determined by HPLC analysis at 20 °C, using a CHIRALCEL AD-3 column (hexanes/2-propanol = 90:10, 1.0 mL/min; retention times 11.1 (*R*), 12.0 min (*S*)).

A colorless oil. $[\alpha]_D^{20}$ = +58 (*c* 0.74, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 1.30-1.39 (m, 1H), 1.51-1.59 (m, 1H), 1.71 (ddd, *J* = 3.0, 11.0, 13.0 Hz, 1H), 1.83-1.90 (m, 1H), 1.92-2.06 (m, 4H), 3.51-3.64 (m, 2H), 5.80-5.85 (m, 1H), 5.87-5.91 (m, 1H), 5.92 (s, 2H), 6.73 (d, *J* = 8.5 Hz, 1H), 6.77 (dd, *J* = 2.0, 8.5 Hz,

1H), 6.85 (d, J = 2.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 18.6, 25.3, 37.4, 41.5, 45.2, 59.7, 100.8, 107.5, 107.7, 120.0, 128.4, 132.6, 141.8, 145.4, 147.6; IR (neat) ν 3348 cm^{-1}. HRMS (MALDI) m/z calcd for $C_{15}H_{18}O_3$ $[M]^+$: 246.1250. Found: 246.1248.

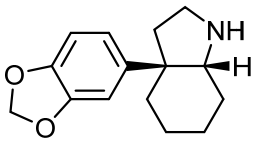
(*S*)-3a-(3,4-methylenedioxyphenyl)-3,3a,4,5,6,7-hexahydro-2*H*-indole [(*S*)-9]



DPPA (53 μL, 0.24 mmol) and DBU (37 μL, 0.24 mmol) were added to a solution of (*S*)-**8** (50 mg, 0.20 mmol) in toluene (0.70 mL) at 30 °C. After the reaction mixture was stirred at 30 °C for 1 h, $TMSN_3$ (27 μL, 0.20 mmol) and DBU (30 μL, 0.20 mmol) were added to the solution. The reaction mixture was refluxed for 24 h and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, CH_2Cl_2/MeOH = 7:1) to give (*S*)-**9** (39 mg, 78% yield).

A colorless oil. $[\alpha]_D^{20}$ = –43 (*c* 1.57, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.33-1.65 (m, 4H), 1.85-1.95 (m, 3H), 2.19-2.30 (m, 1H), 2.57-2.76 (m, 2H), 3.62 (ddt, J = 4.0, 8.5, 15.0 Hz, 1H), 3.86 (dddd, J = 1.5, 3.5, 8.0, 15.0 Hz, 1H), 5.94 (s, 2H), 6.54 (dd, J = 2.0, 8.0 Hz, 1H), 6.65 (d, J = 2.0 Hz, 1H), 6.76 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.4, 27.5, 30.2, 39.2, 41.9, 57.5, 58.5, 101.0, 106.6, 108.4, 119.1, 137.0, 145.8, 148.3, 180.8; IR (neat) ν 1651 cm^{-1}. HRMS (MALDI) m/z calcd for $C_{15}H_{18}NO_2$ $[M+H]^+$: 244.1332. Found: 244.1331.

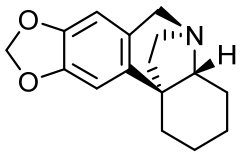
(3a*R*,7a*R*)-3a-(3,4-methylenedioxyphenyl)octahydro-1*H*-indole (10)



$NaBH_3CN$ (11 mg, 0.16 mmol) was added to a solution of (*S*)-**9** (20 mg, 0.082 mmol) in acetic acid (0.33 mL) at room temperature. The reaction mixture was stirred at room temperature for 30 min, and 15% aq. NaOH (2 mL) and Et_2O (10 mL) were added. The organic phase was separated, and the aqueous phase was extracted with Et_2O (3 x 5 mL). The combined organic phases were washed with brine (10 mL), dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, CH_2Cl_2/MeOH = 5:1) to give **10** (17.7 mg, 88% yield).

A colorless oil. $[\alpha]_D^{20}$ = +11 (*c* 0.86, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.15-1.32 (m, 1H), 1.40-1.58 (m, 3H), 1.62-1.94 (m, 5H), 2.00 (ddd, J = 4.0, 8.5, 13.0 Hz, 1H), 2.07 (br s, 1H), 3.00 (dt, J = 4.5, 10.0 Hz, 1H), 3.09-3.17 (m, 1H), 3.42 (t, J = 4.0 Hz, 1H), 5.93 (s, 2H), 6.76 (d, J = 8.0 Hz, 1H), 6.82 (dd, J = 2.0, 8.0 Hz, 1H), 6.88 (d, J = 2.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.1, 22.1, 26.3, 33.9, 41.4, 43.1, 47.9, 61.0, 100.8, 107.6, 107.7, 119.4, 141.0, 145.2, 147.6; IR (neat) ν 3339 cm^{-1}. HRMS (MALDI) m/z calcd for $C_{15}H_{20}NO_2$ $[M+H]^+$: 246.1489. Found: 246.1493.

(–)-Crinane



N,*N*-Dimethylmethyleneammonium iodide (15 mg, 0.082 mmol) was added to a solution of **10** (10 mg, 0.041 mmol) in THF (2 mL) and the mixture was stirred at 50 °C for 36 h. THF was removed under reduced pressure, and Et_2O (5 mL) was added. 15% aq. NaOH was added until the solution became basic. The aqueous phase was extracted with Et_2O (3 x 5 mL). The organic phases were combined and washed with brine (5 mL), dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, CH_2Cl_2/MeOH = 10:1) to give (–)-crinane (10.1 mg, 96% yield).

A colorless oil. $[\alpha]_D^{20}$ = –8.6 (*c* 0.77, $CHCl_3$) [ref.[7] $[\alpha]_D^{20}$ –12.2 (*c* 0.66, $CHCl_3$), ref.[8] $[\alpha]_D^{20}$ +7.0 (c 1.0, $CHCl_3$) for (+)-crinane with 94% ee}]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.12-1.32 (m, 2H), 1.43-1.67(m, 2H), 1.68-1.81 (m, 4H), 2.16-2.26 (m, 1H), 2.34 (d, *J* = 13.0 Hz, 1H), 2.75-2.90 (m, 2H), 3.29-3.41 (m, 1H), 3.75 (d, *J* = 17.0 Hz, 1H), 4.35 (d, *J* = 17.0 Hz, 1H), 5.88 (s, 2H), 6.46 (s, 1H), 6.71 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.8, 24.4, 27.6, 29.0, 37.9, 29.0, 52.0, 62.1, 67.4, 100.7, 103.4, 106.3, 126.0, 142.2, 145.6, 146.3. ; IR (neat) ν 3460 cm^{-1}. HRMS (MALDI) *m*/*z* calcd for $C_{16}H_{20}NO_2$ $[M+H]^+$: 258.1489. Found: 258.1484.

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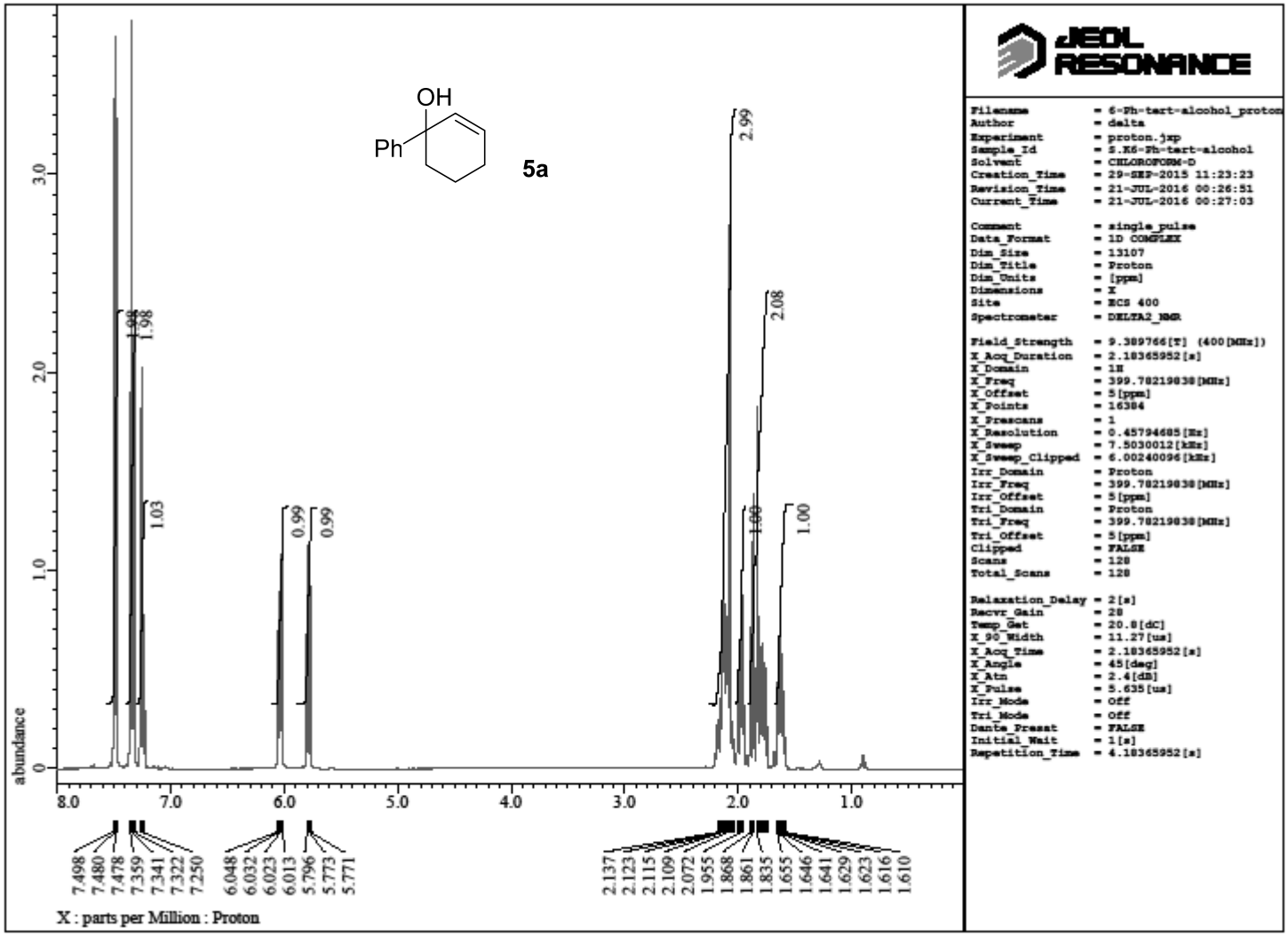
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Ph
5a
abundance
X : parts per Million : Proton
JEOL RESONANCE
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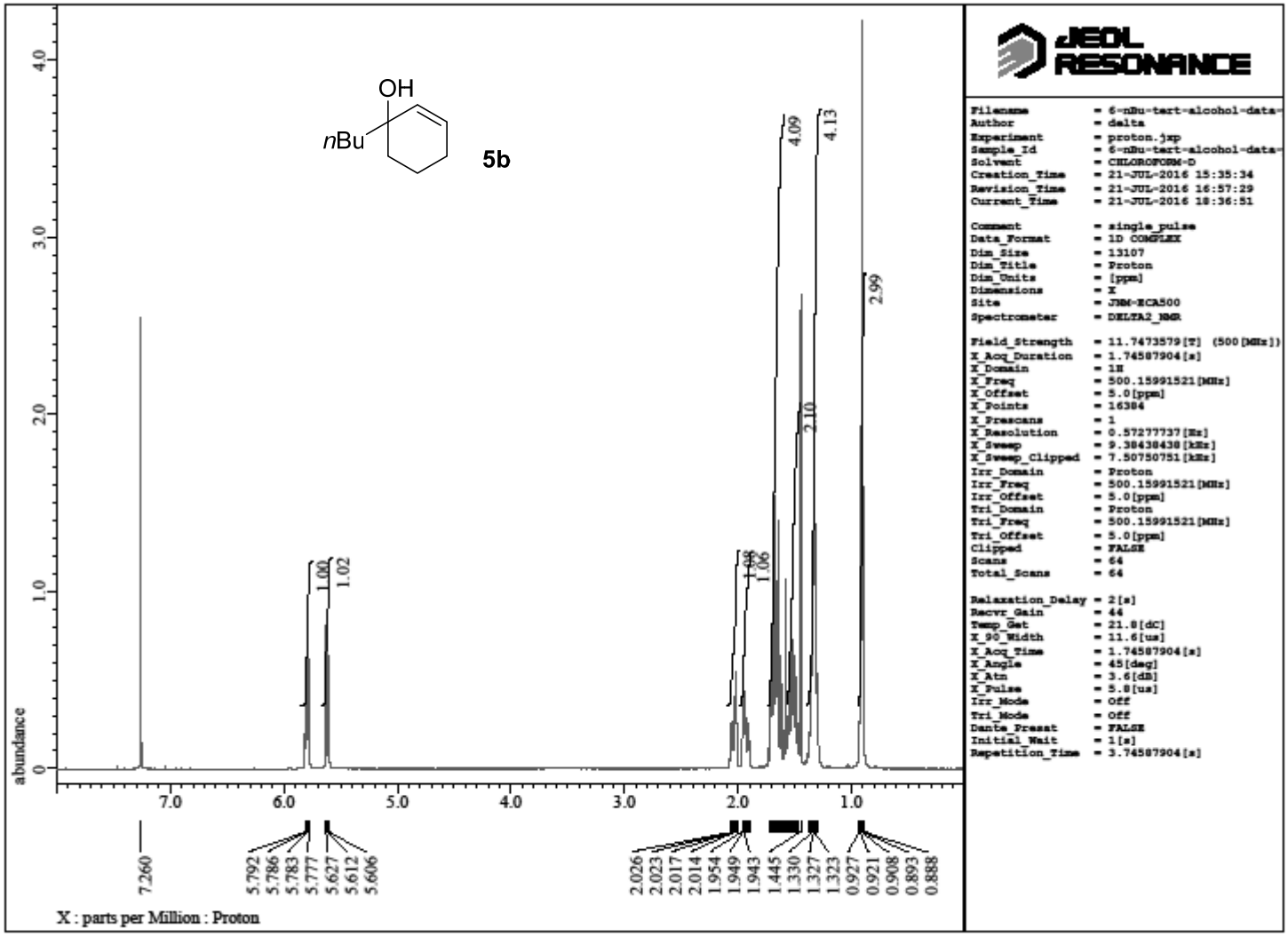
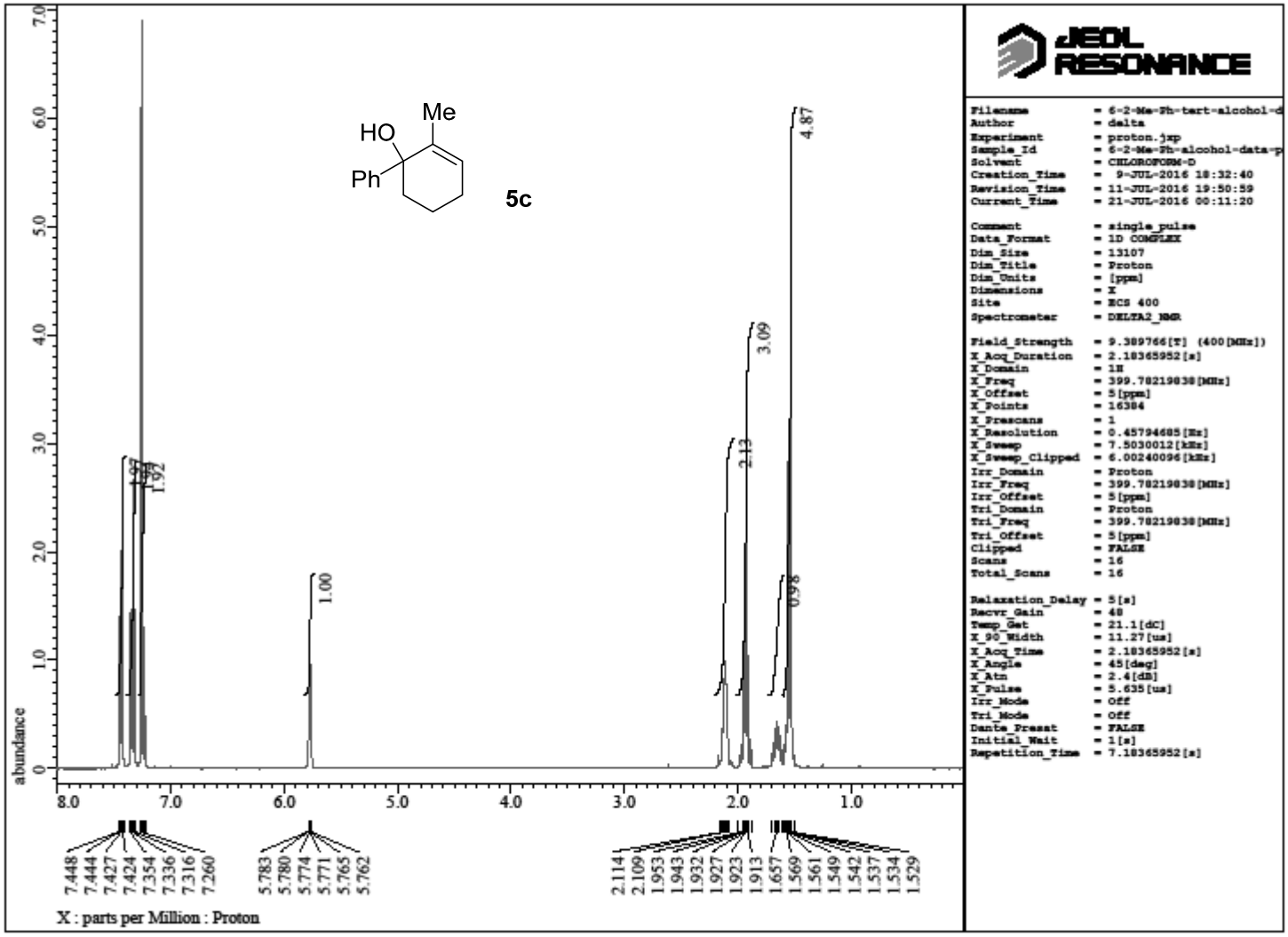
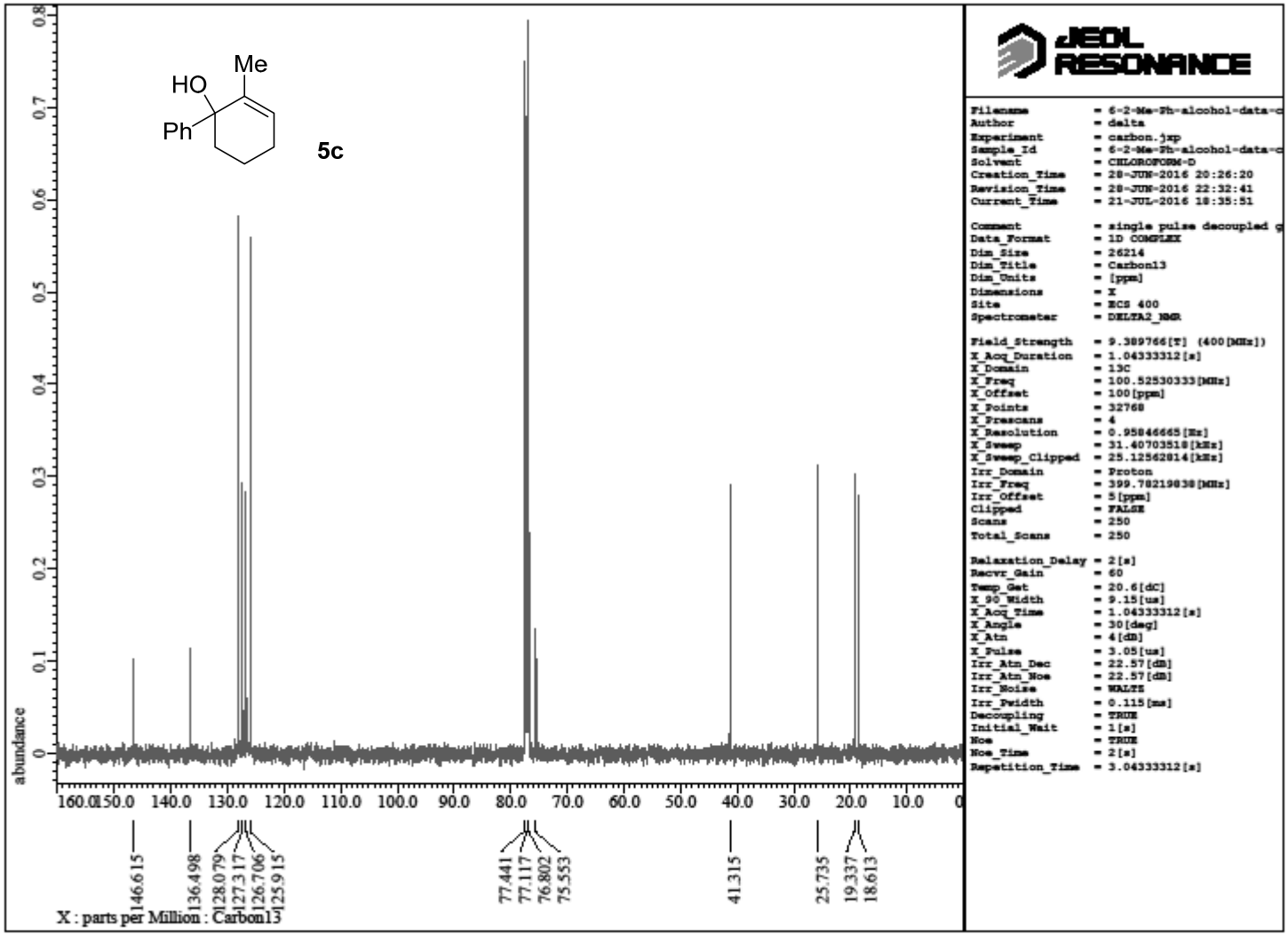
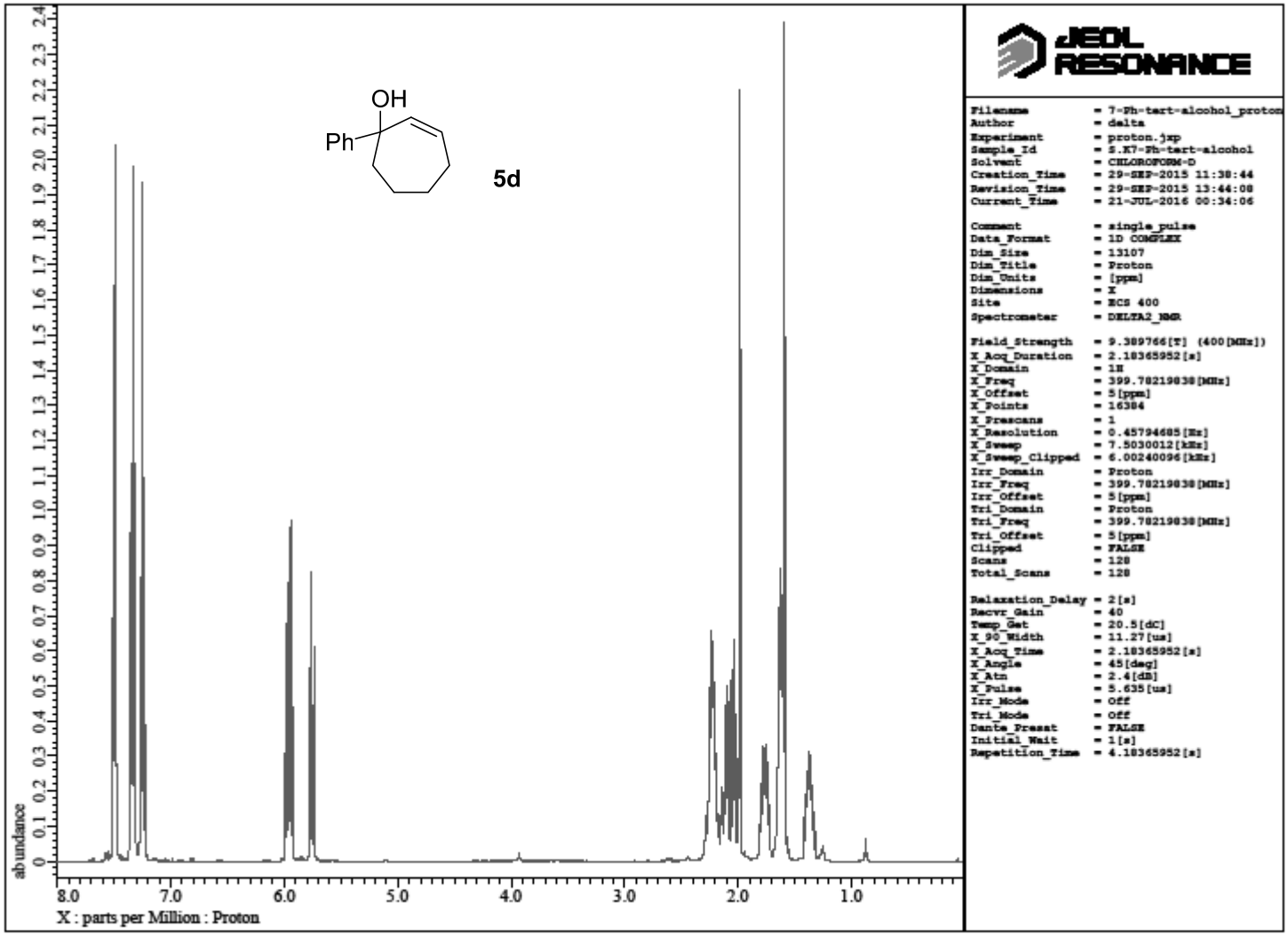
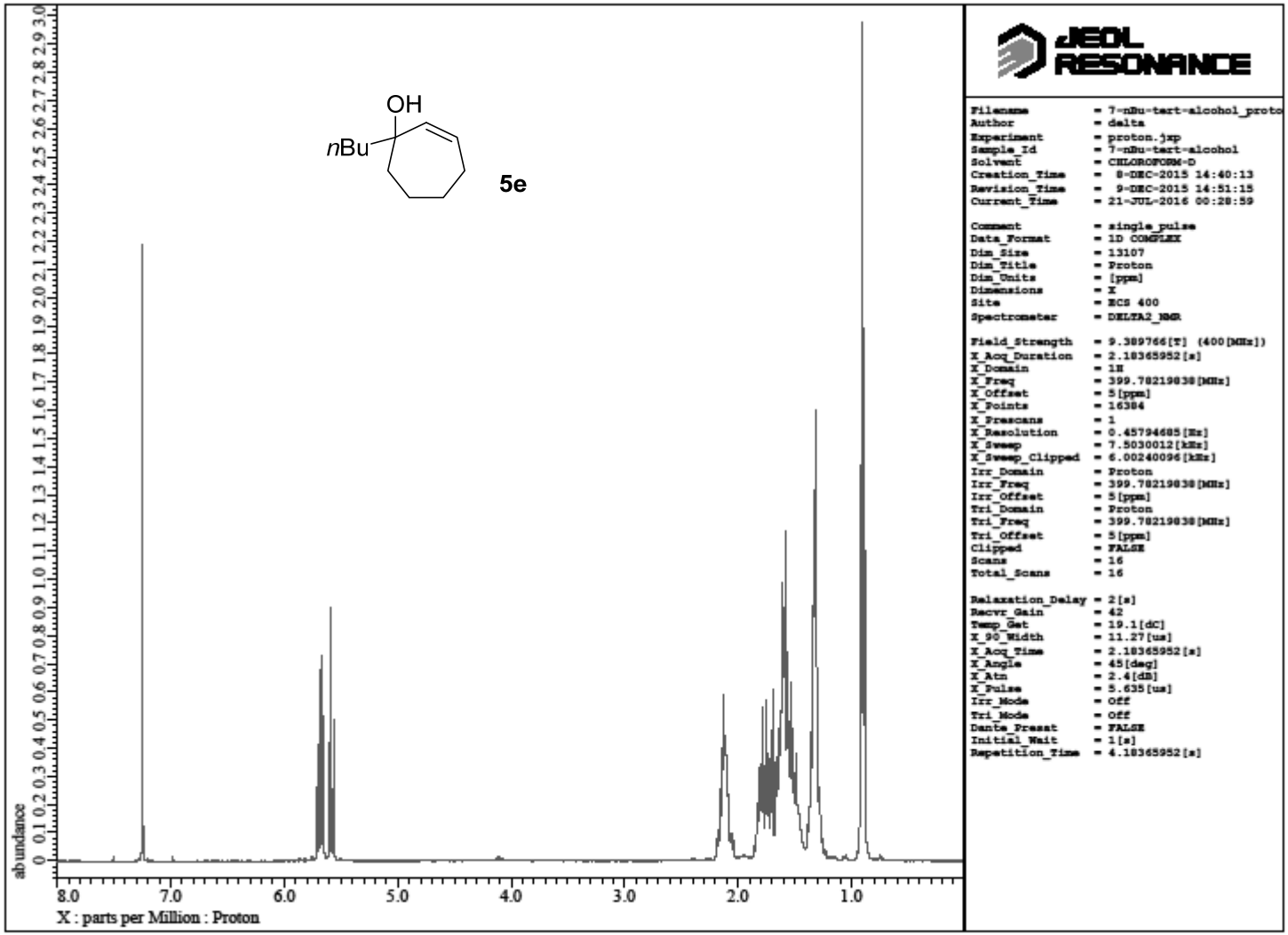
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 128
Total_Scans = 128
Relaxation_Delay = 2[s]
Recvr_Gain = 28
Temp_Get = 20.8[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.6[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]

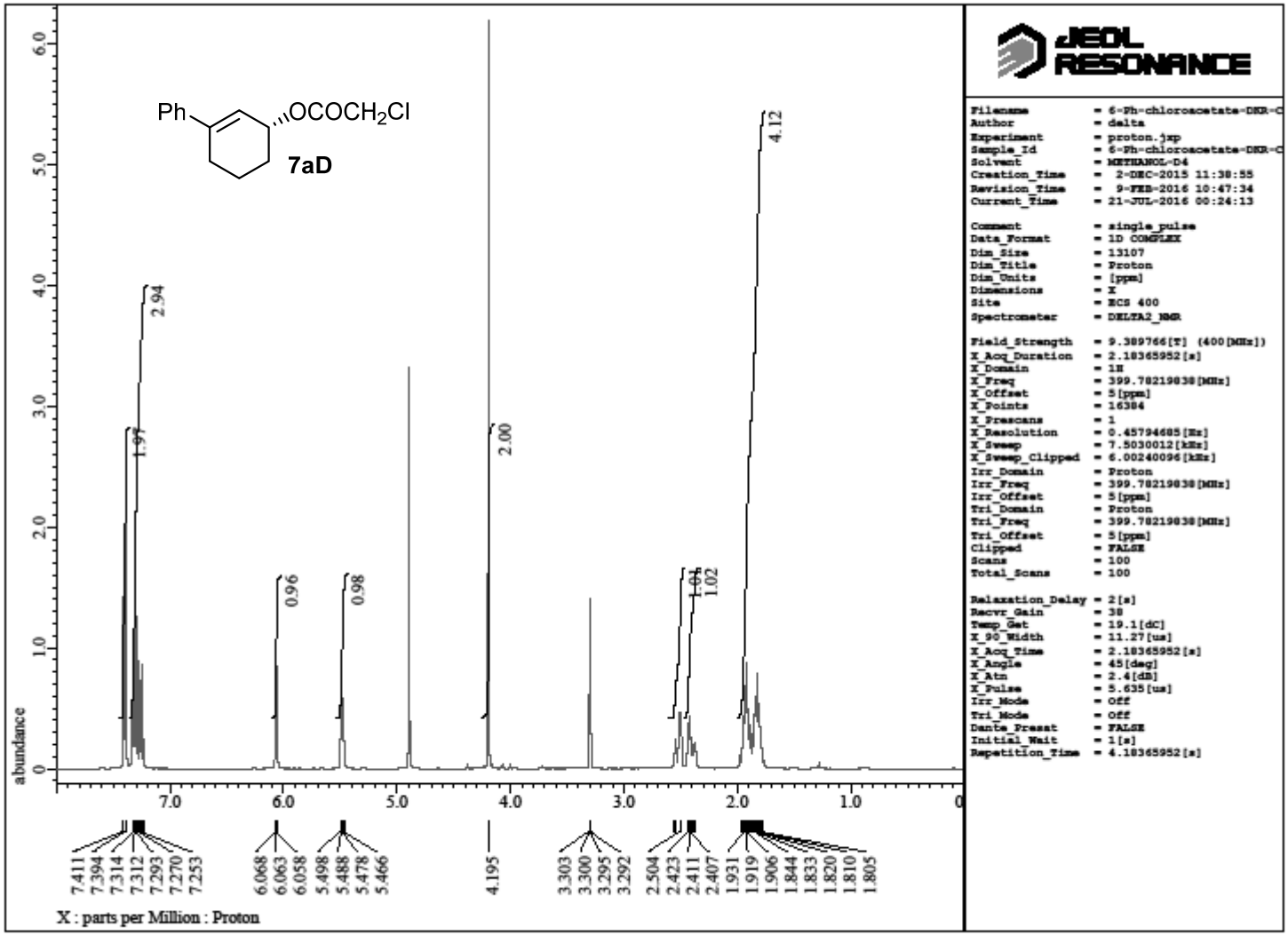

OH
nBu
5b
4.09
4.13
2.99
2.10
1.00
1.02
1.08
1.06
abundance
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.260
5.792
5.786
5.783
5.777
5.627
5.612
5.606
2.026
2.023
2.017
2.014
1.954
1.949
1.943
1.445
1.330
1.327
1.323
0.927
0.921
0.908
0.893
0.888
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-nBu-tert-alcohol-data-
Author = delta
Experiment = proton.jxp
Sample_Id = 6-nBu-tert-alcohol-data-
Solvent = CHLOROFORM-D
Creation_Time = 21-JUL-2016 15:35:34
Revision_Time = 21-JUL-2016 16:57:29
Current_Time = 21-JUL-2016 18:36:51
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 64
Total_Scans = 64
Relaxation_Delay = 2[s]
Recvr_Gain = 44
Temp_Get = 21.8[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 5.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 3.74587904[s]

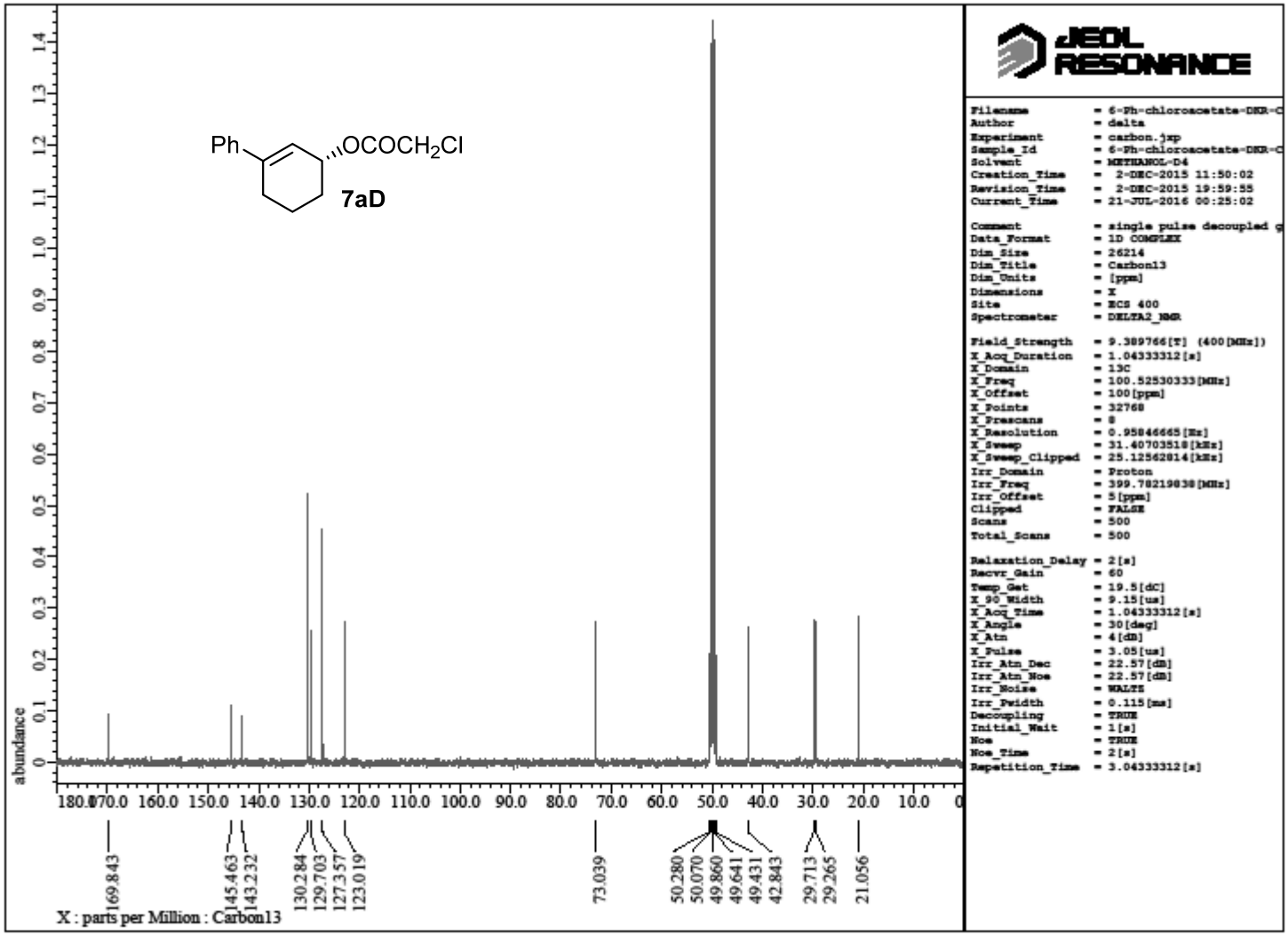

Me
HO
Ph
5c
4.87
3.09
2.13
1.92
1.00
0.98
abundance
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.448
7.444
7.427
7.424
7.354
7.336
7.316
7.260
5.783
5.780
5.774
5.771
5.765
5.762
2.114
2.109
1.953
1.943
1.932
1.927
1.923
1.913
1.657
1.569
1.561
1.549
1.542
1.537
1.534
1.529
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-2-Me-Ph-tert-alcohol-d
Author = delta
Experiment = proton.jxp
Sample_Id = 6-2-Me-Ph-alcohol-data-p
Solvent = CHLOROFORM-D
Creation_Time = 9-JUL-2016 18:32:40
Revision_Time = 11-JUL-2016 19:50:59
Current_Time = 21-JUL-2016 00:11:20
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 48
Temp_Get = 21.1[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.6[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 7.18365952[s]

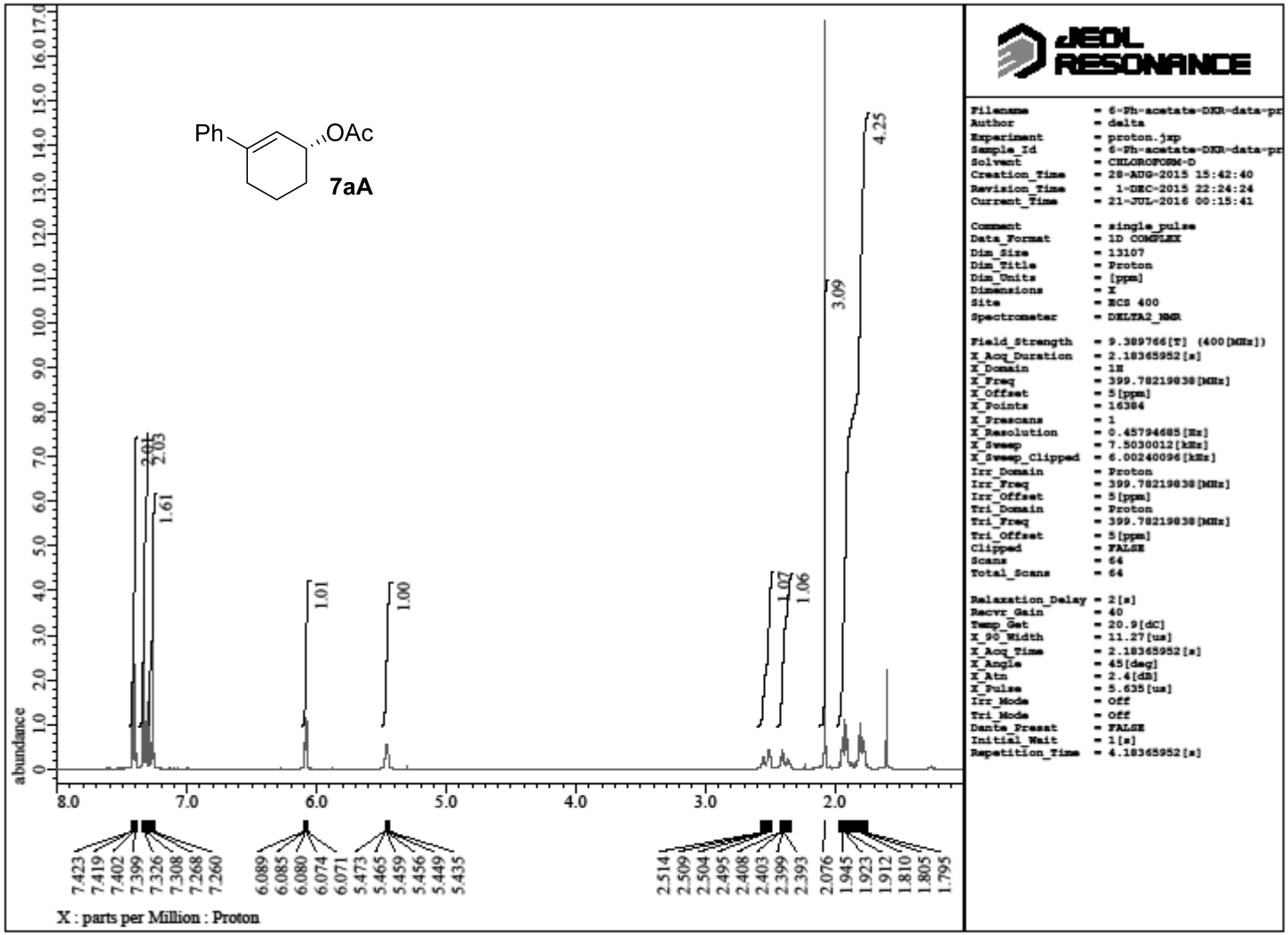

Me
HO
Ph
5c
abundance
X : parts per Million : Carbon13
146.615
136.498
128.079
127.317
126.706
125.915
77.441
77.117
76.802
75.553
41.315
25.735
19.337
18.613
JEOL RESONANCE
Filename = 6-2-Me-Ph-alcohol-data-c
Author = delta
Experiment = carbon.jxp
Sample_Id = 6-2-Me-Ph-alcohol-data-c
Solvent = CHLOROFORM-D
Creation_Time = 28-JUN-2016 20:26:20
Revision_Time = 28-JUN-2016 22:32:41
Current_Time = 21-JUL-2016 18:35:51
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 250
Total_Scans = 250
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 20.6[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

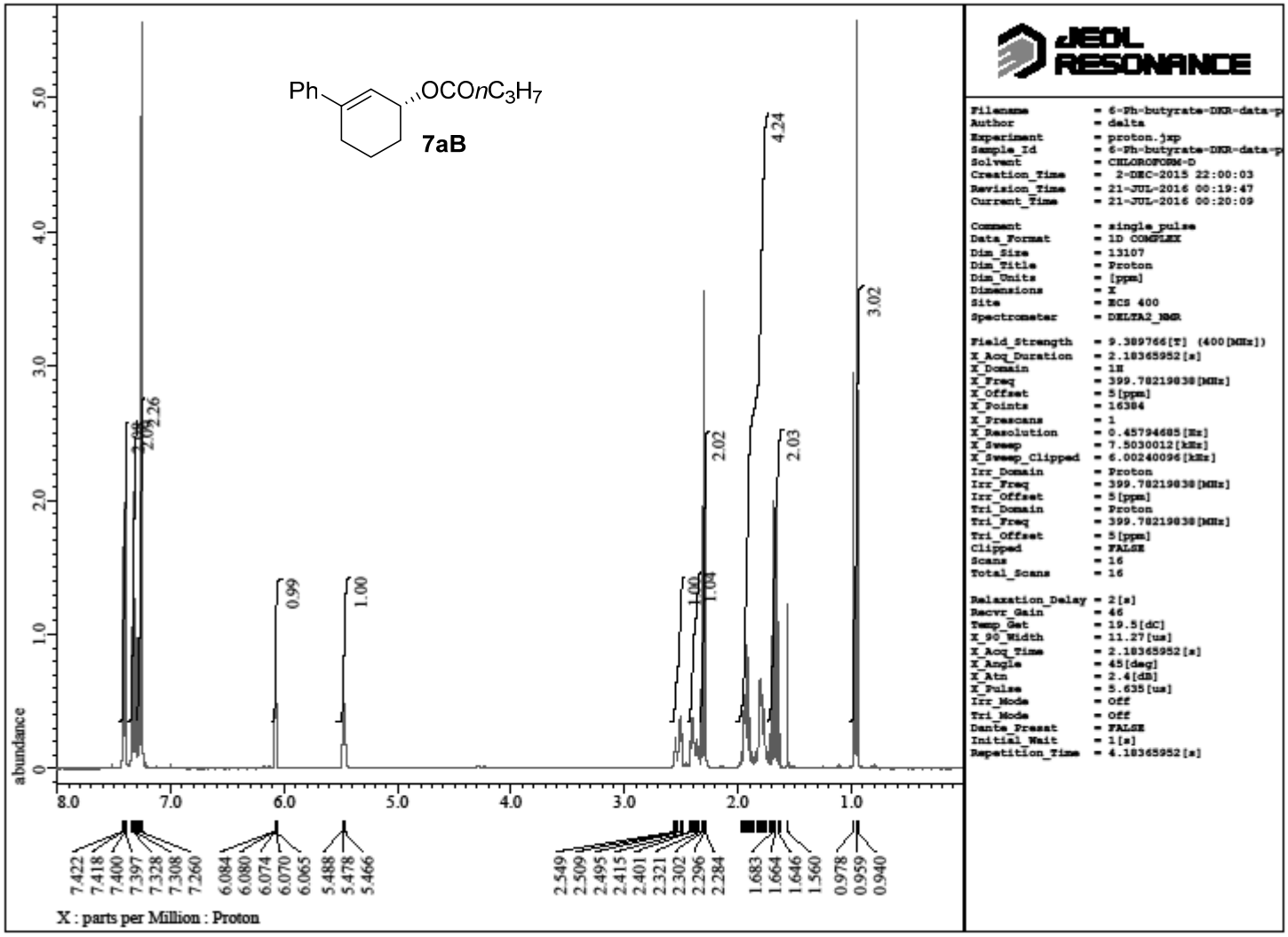

OH
Ph
5d
abundance
X : parts per Million : Proton
JEOL RESONANCE
Filename = 7-Ph-tert-alcohol_proton
Author = delta
Experiment = proton.jxp
Sample_Id = S.K7-Ph-tert-alcohol
Solvent = CHLOROFORM-D
Creation_Time = 29-SEP-2015 11:38:44
Revision_Time = 29-SEP-2015 13:44:08
Current_Time = 21-JUL-2016 00:34:06
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 128
Total_Scans = 128
Relaxation_Delay = 2[s]
Recvr_Gain = 40
Temp_Get = 20.5[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.6[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Preset = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]

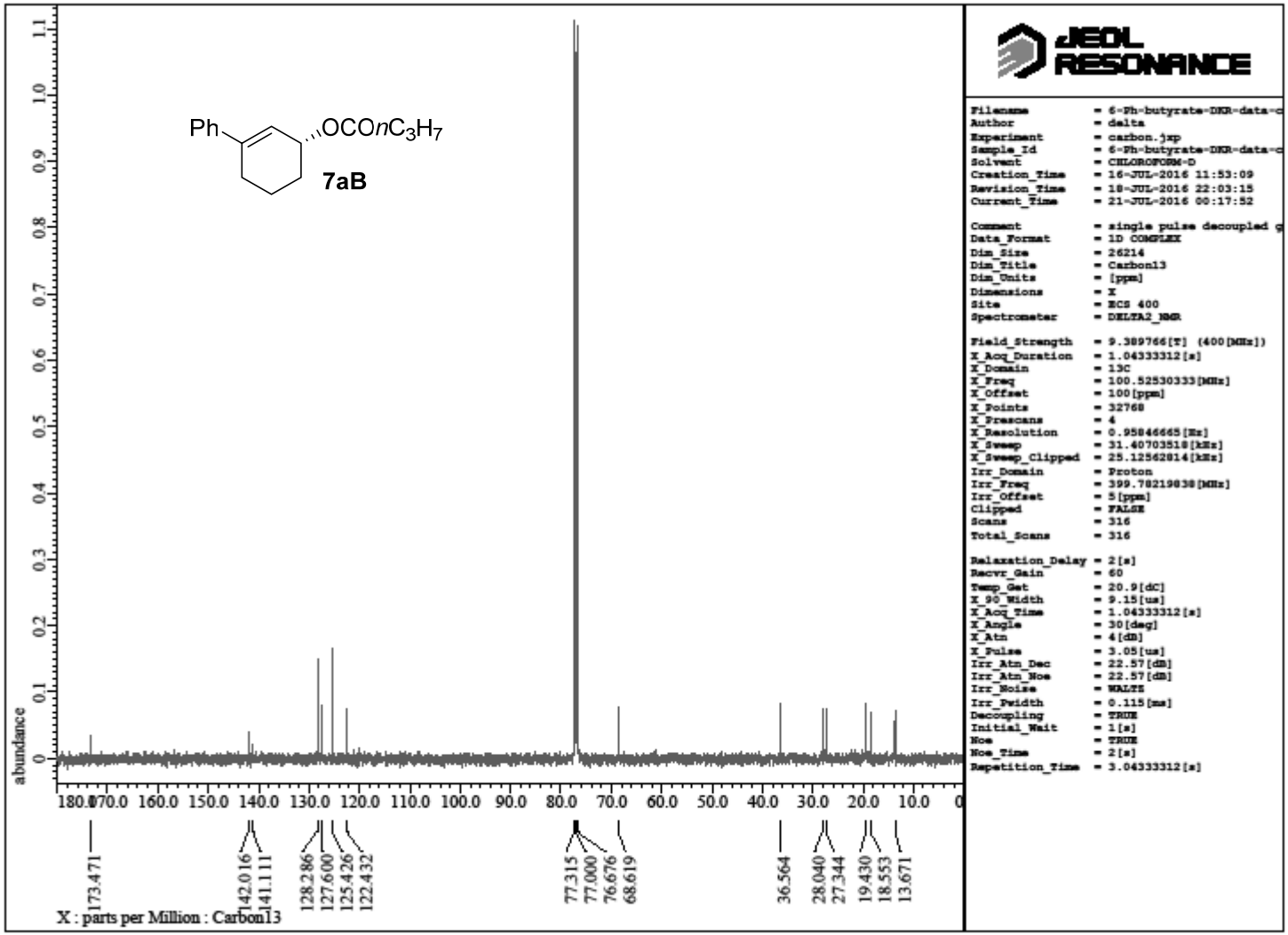

OH
nBu
5e
abundance
X : parts per Million : Proton
JEOL RESONANCE
Filename = 7-nBu-tert-alcohol_proto
Author = delta
Experiment = proton.jxp
Sample_Id = 7-nBu-tert-alcohol
Solvent = CHLOROFORM-D
Creation_Time = 8-DEC-2015 14:40:13
Revision_Time = 9-DEC-2015 14:51:15
Current_Time = 21-JUL-2016 00:28:59
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 2[s]
Recvr_Gain = 42
Temp_Get = 19.1[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Preset = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]

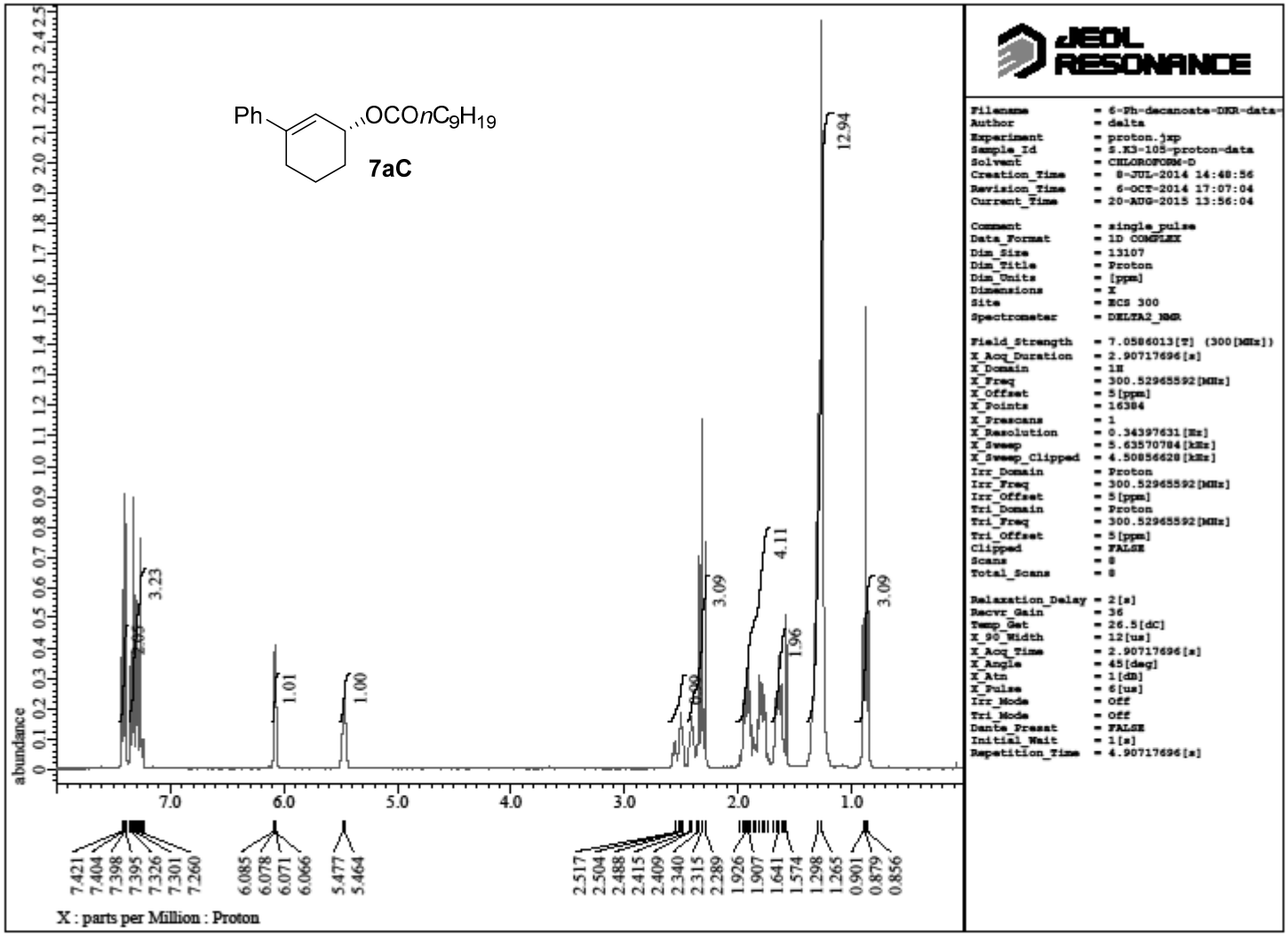

Ph
OCOCH2Cl
7aD
2.94
1.97
0.96
0.98
2.00
1.01
1.02
4.12
abundance
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.411
7.394
7.314
7.312
7.293
7.270
7.253
6.068
6.063
6.058
5.498
5.488
5.478
5.466
4.195
3.303
3.300
3.295
3.292
2.504
2.423
2.411
2.407
1.931
1.919
1.906
1.844
1.833
1.820
1.810
1.805
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-Ph-chloroacetate-DKR-C
Author = delta
Experiment = proton.jxp
Sample_Id = 6-Ph-chloroacetate-DKR-C
Solvent = METHANOL-D4
Creation_Time = 2-DEC-2015 11:38:55
Revision_Time = 9-FEB-2016 10:47:34
Current_Time = 21-JUL-2016 00:24:13
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 100
Total_Scans = 100
Relaxation_Delay = 2[s]
Recvr_Gain = 38
Temp_Get = 19.1[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.6[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]

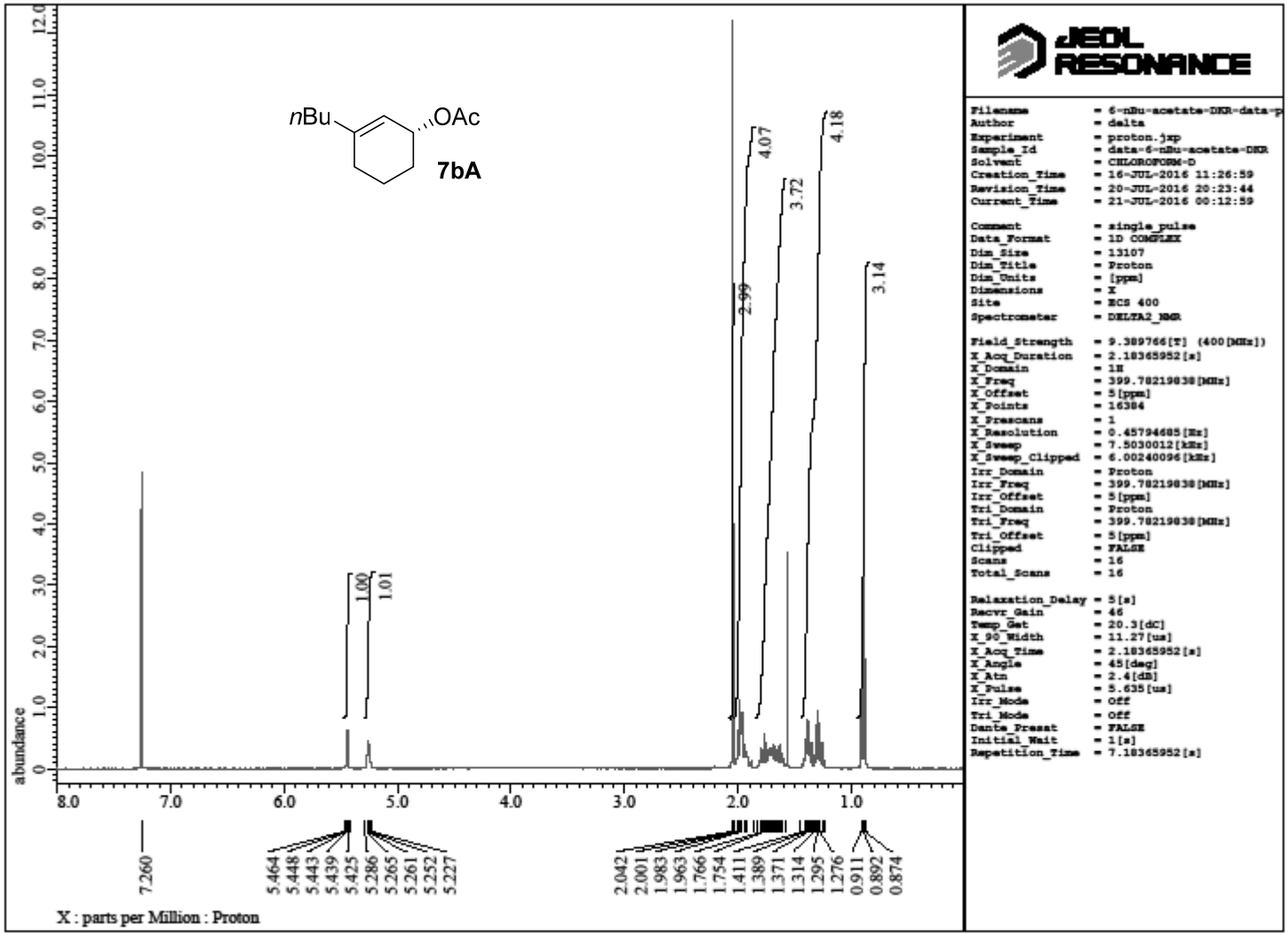

Ph
OCOCH2Cl
7aD
abundance
X : parts per Million : Carbon13
169.843
145.463
143.232
130.284
129.703
127.357
123.019
73.039
50.280
50.070
49.860
49.641
49.431
42.843
29.713
29.265
21.056
Filename = 6-Ph-chloroacetate-DKR-C
Author = delta
Experiment = carbon.jxp
Sample_Id = 6-Ph-chloroacetate-DKR-C
Solvent = METHANOL-D4
Creation_Time = 2-DEC-2015 11:50:02
Revision_Time = 2-DEC-2015 19:59:55
Current_Time = 21-JUL-2016 00:25:02
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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 = 8
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 500
Total_Scans = 500
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.5[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

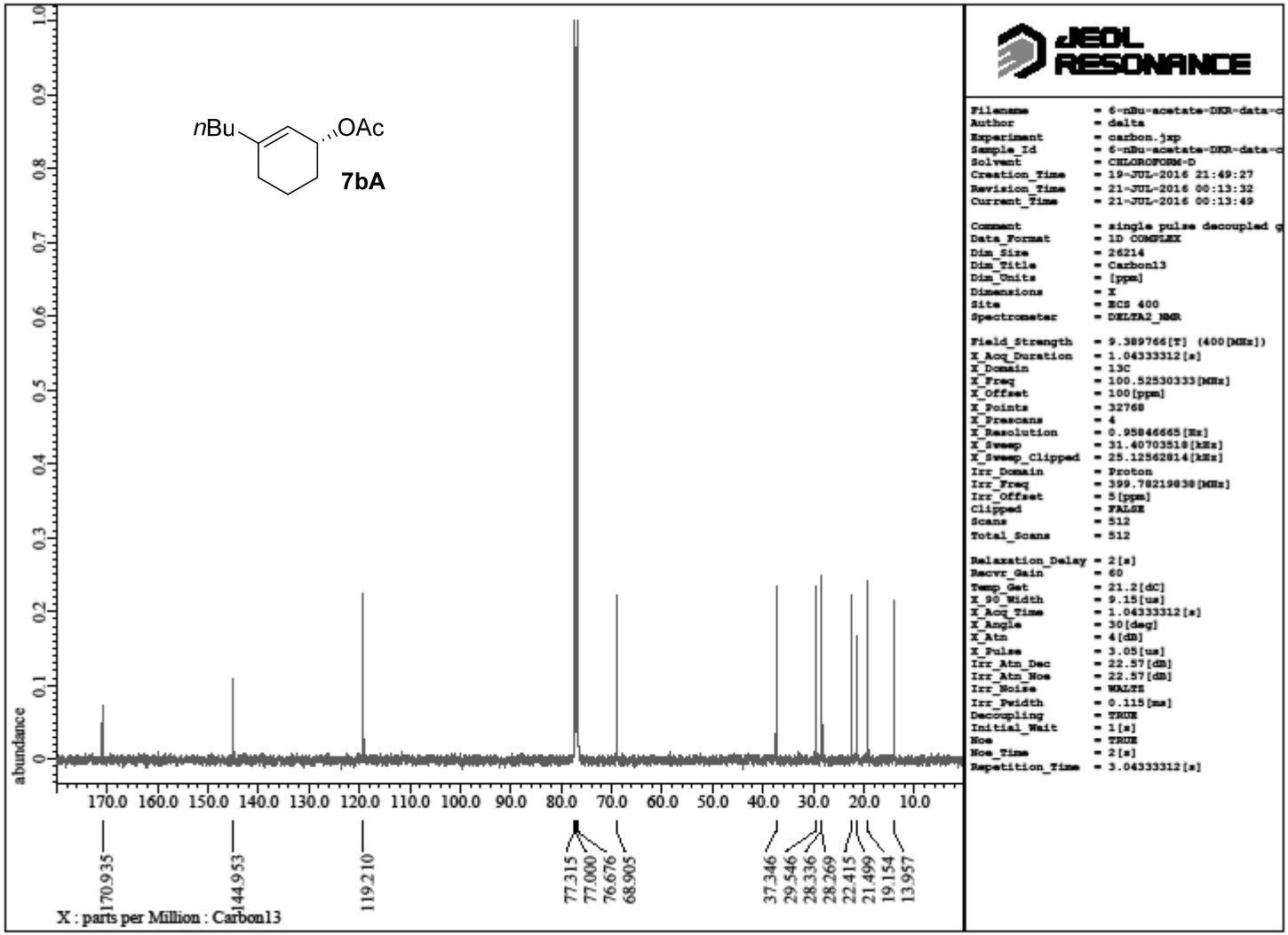

Ph
OAc
7aA
abundance
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-Ph-acetate-DKR-data-pr
Author = delta
Experiment = proton.jxp
Sample_Id = 6-Ph-acetate-DKR-data-pr
Solvent = CHLOROFORM-D
Creation_Time = 28-AUG-2015 15:42:40
Revision_Time = 1-DEC-2015 22:24:24
Current_Time = 21-JUL-2016 00:15:41
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 64
Total_Scans = 64
Relaxation_Delay = 2[s]
Recvr_Gain = 40
Temp_Get = 20.9[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Preset = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]

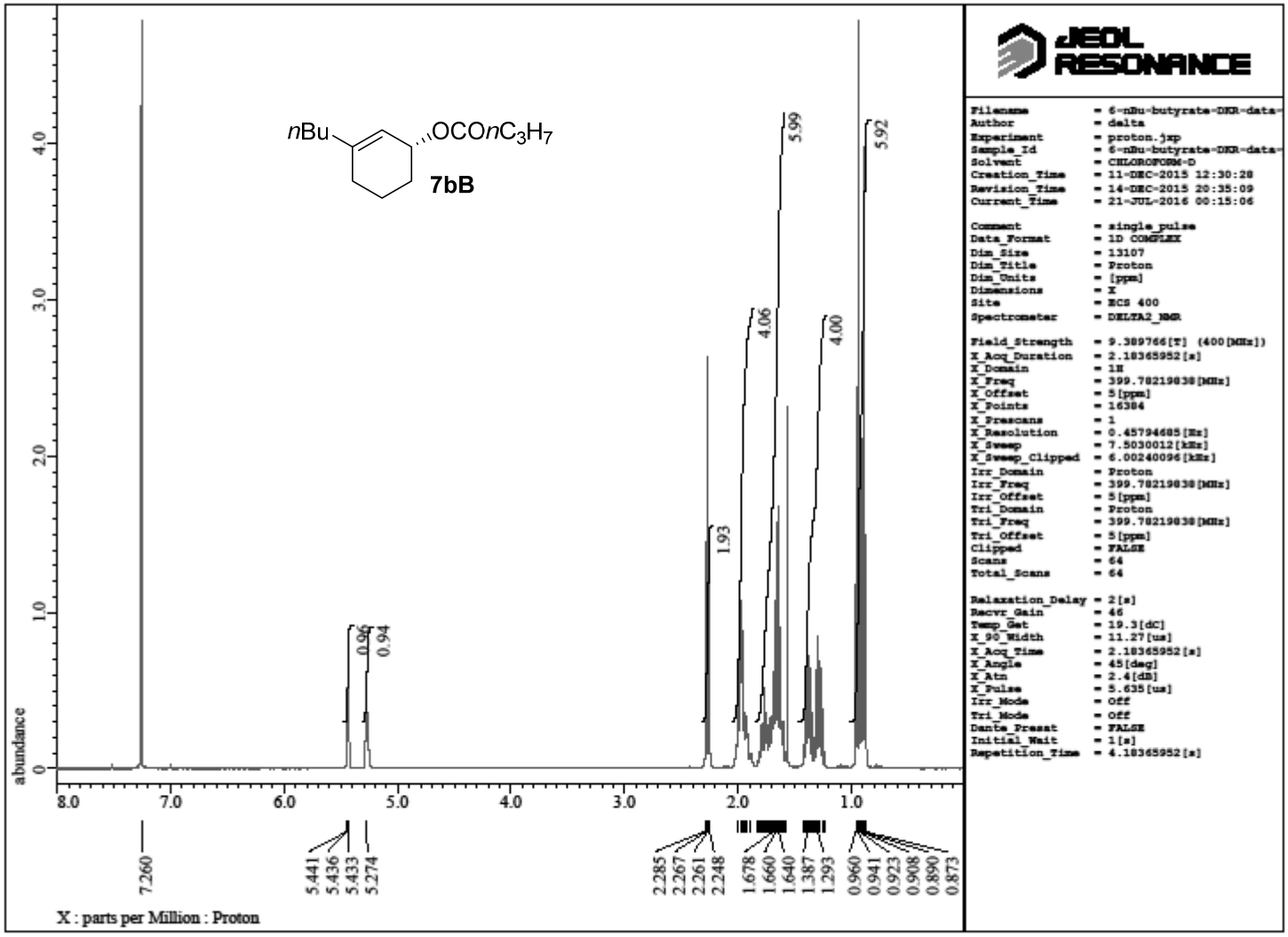

Ph
OCOnC_3H_7
7aB
abundance
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-Ph-butyrate-DKR-data-p
Author = delta
Experiment = proton.jxp
Sample_Id = 6-Ph-butyrate-DKR-data-p
Solvent = CHLOROFORM-D
Creation_Time = 2-DEC-2015 22:00:03
Revision_Time = 21-JUL-2016 00:19:47
Current_Time = 21-JUL-2016 00:20:09
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 2[s]
Recvr_Gain = 46
Temp_Get = 19.5[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]

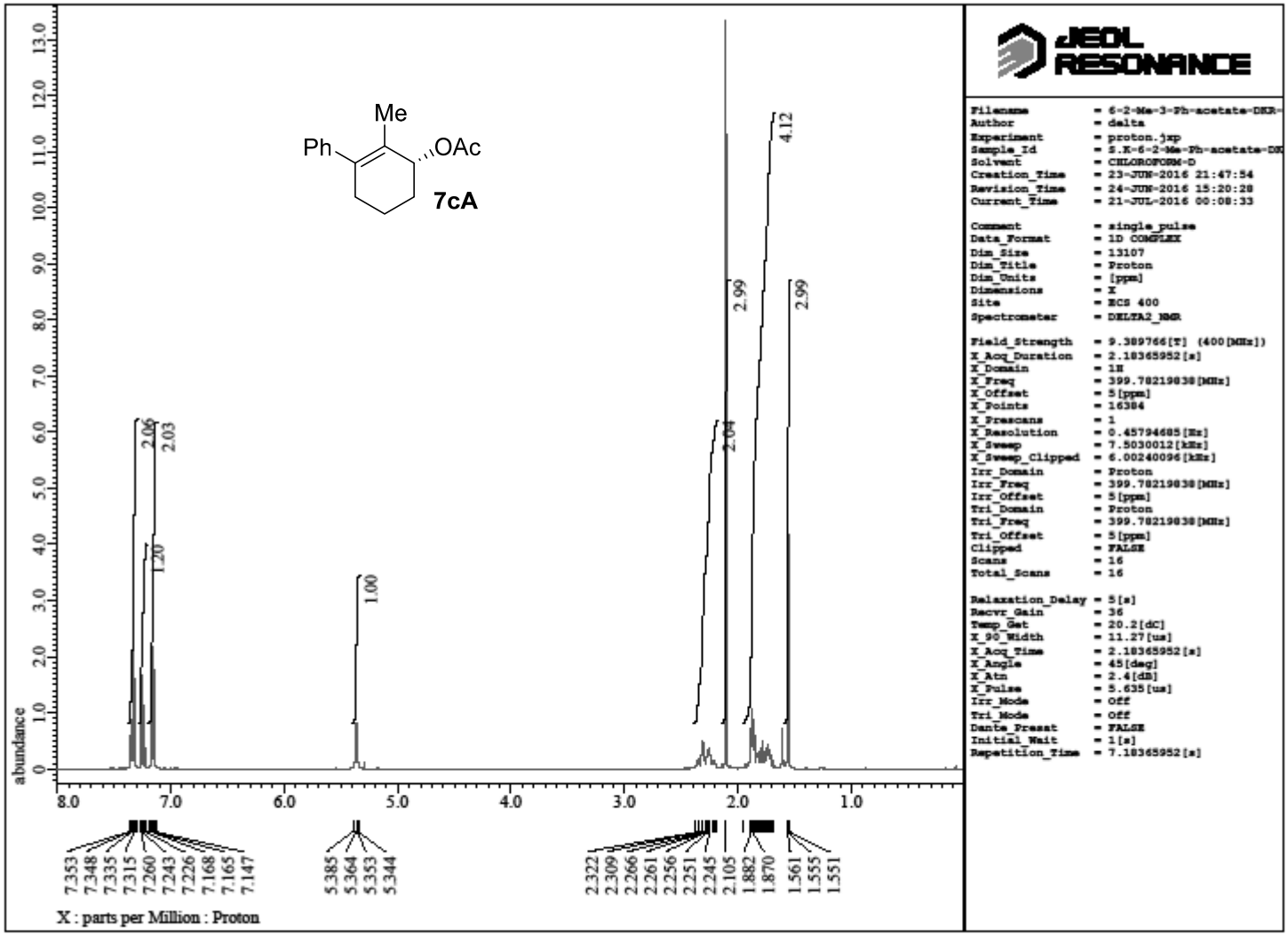

Ph
OCOnC_3H_7
7aB
abundance
X : parts per Million : Carbon13
173.471
142.016
141.111
128.286
127.600
125.426
122.432
77.315
77.000
76.676
68.619
36.564
28.040
27.344
19.430
18.553
13.671
JEOL RESONANCE
Filename = 6-Ph-butyrate-DKR-data-c
Author = delta
Experiment = carbon.jxp
Sample_Id = 6-Ph-butyrate-DKR-data-c
Solvent = CHLOROFORM-D
Creation_Time = 16-JUL-2016 11:53:09
Revision_Time = 18-JUL-2016 22:03:15
Current_Time = 21-JUL-2016 00:17:52
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 316
Total_Scans = 316
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 20.9[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

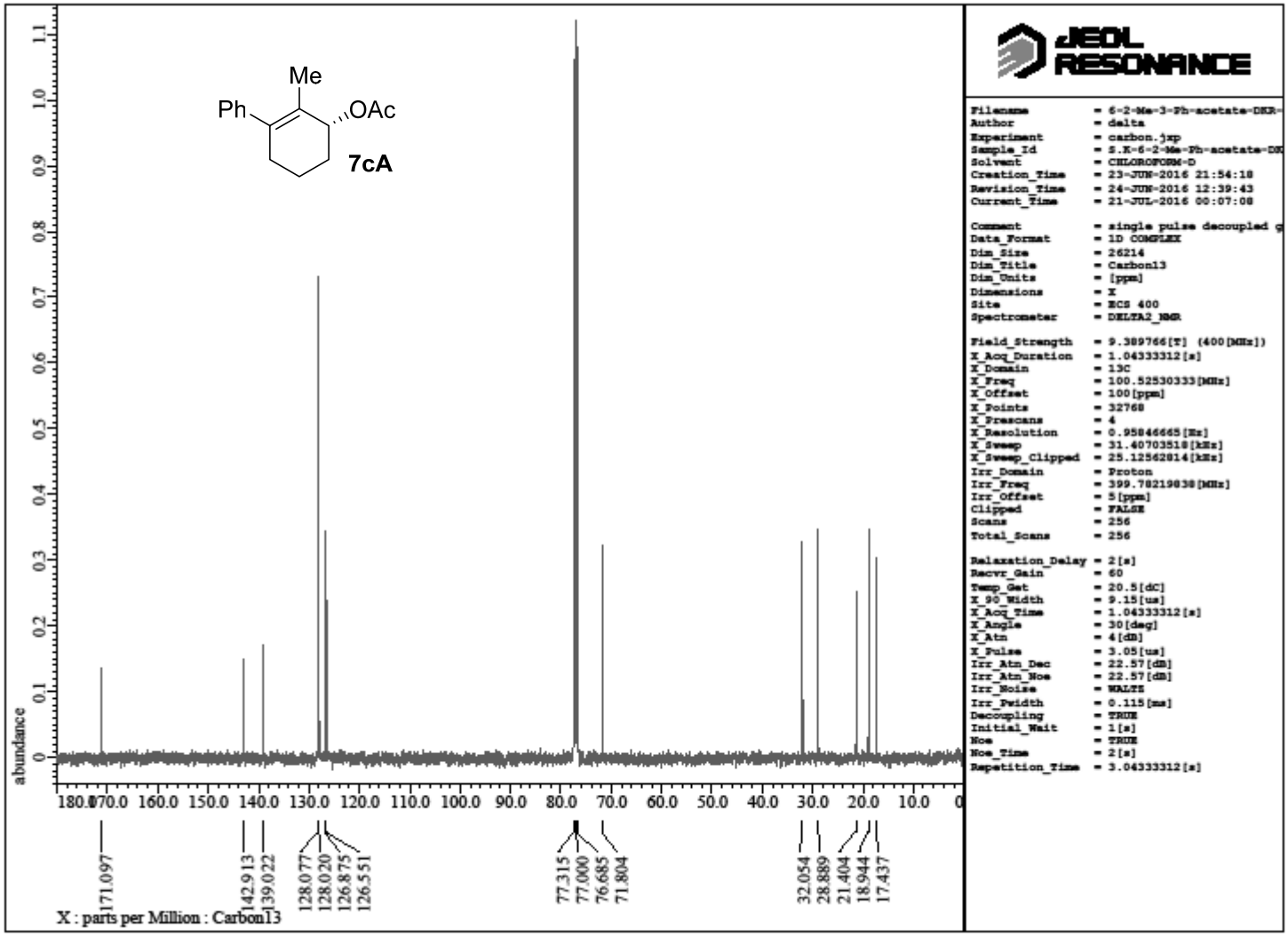

Ph
OCOnC_9H_{19}
7aC
abundance
12.94
4.11
3.23
3.09
3.09
2.03
1.96
1.01
1.00
7.421
7.404
7.398
7.395
7.326
7.301
7.260
6.085
6.078
6.071
6.066
5.477
5.464
2.517
2.504
2.488
2.415
2.409
2.340
2.315
2.289
1.926
1.907
1.641
1.574
1.298
1.265
0.901
0.879
0.856
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-Ph-decanoate-DKR-data-
Author = delta
Experiment = proton.jxp
Sample_Id = S.K3-105-proton-data
Solvent = CHLOROFORM-D
Creation_Time = 8-JUL-2014 14:48:56
Revision_Time = 6-OCT-2014 17:07:04
Current_Time = 20-AUG-2015 13:56:04
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 300
Spectrometer = DELTA2_NMR
Field_Strength = 7.0586013[T] (300[MHz])
X_Acq_Duration = 2.90717696[s]
X_Domain = 1H
X_Freq = 300.52965592[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.34397631[Hz]
X_Sweep = 5.63570784[kHz]
X_Sweep_Clipped = 4.50856628[kHz]
Irr_Domain = Proton
Irr_Freq = 300.52965592[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 300.52965592[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 2[s]
Recvr_Gain = 36
Temp_Get = 26.5[dC]
X_90_Width = 12[us]
X_Acq_Time = 2.90717696[s]
X_Angle = 45[deg]
X_Atn = 1[dB]
X_Pulse = 6[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.90717696[s]

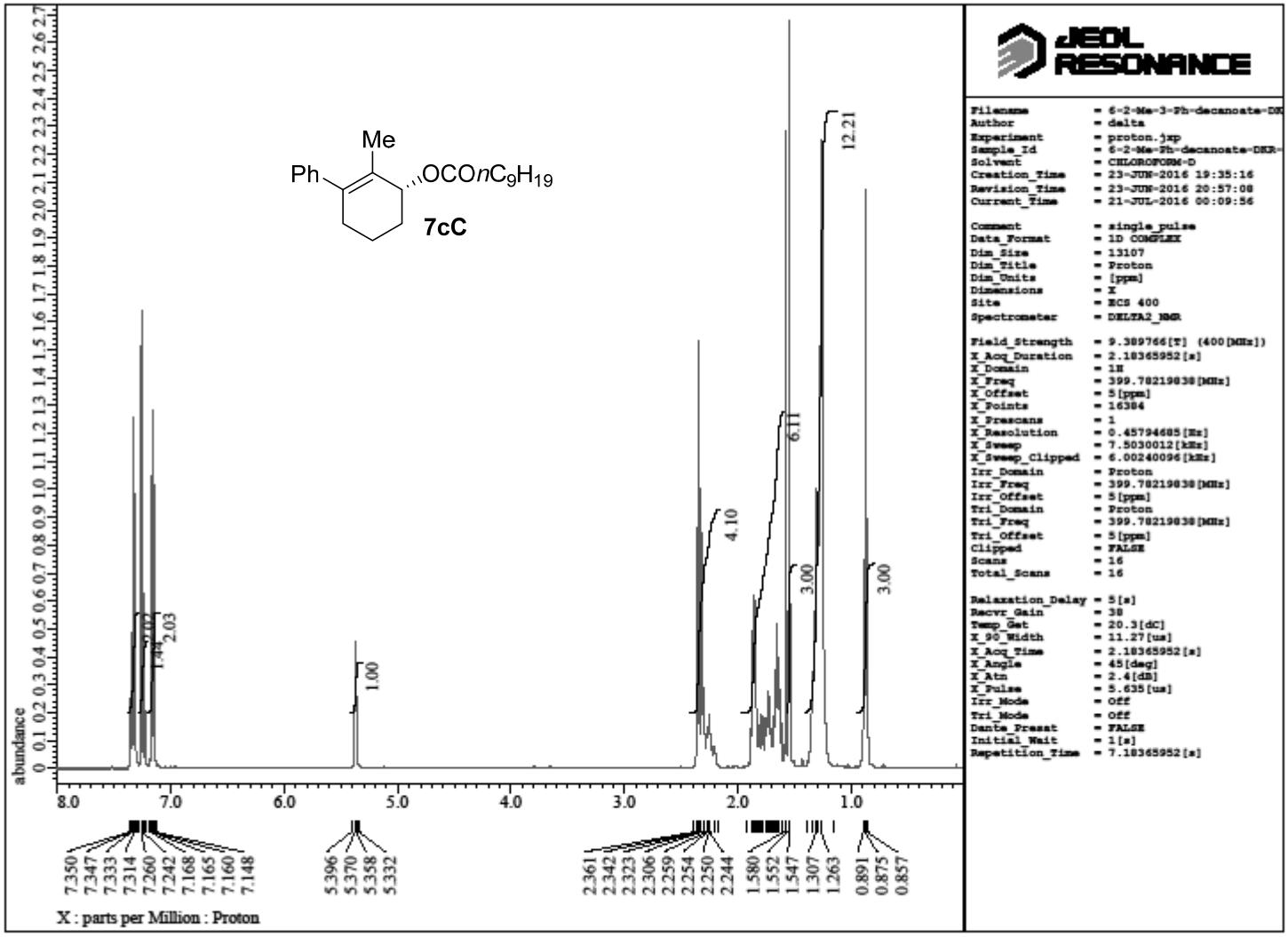

nBu
OAc
7bA
4.07
4.18
3.72
2.99
3.14
1.00
1.01
abundance
X : parts per Million : Proton
7.260
5.464
5.448
5.443
5.439
5.425
5.286
5.265
5.261
5.252
5.227
2.042
2.001
1.983
1.963
1.766
1.754
1.411
1.389
1.371
1.314
1.295
1.276
0.911
0.892
0.874
JEOL RESONANCE
Filename = 6-nBu-acetate-DKR-data-p
Author = delta
Experiment = proton.jxp
Sample_Id = data-6-nBu-acetate-DKR
Solvent = CHLOROFORM-D
Creation_Time = 16-JUL-2016 11:26:59
Revision_Time = 20-JUL-2016 20:23:44
Current_Time = 21-JUL-2016 00:12:59
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 20.3[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 7.18365952[s]

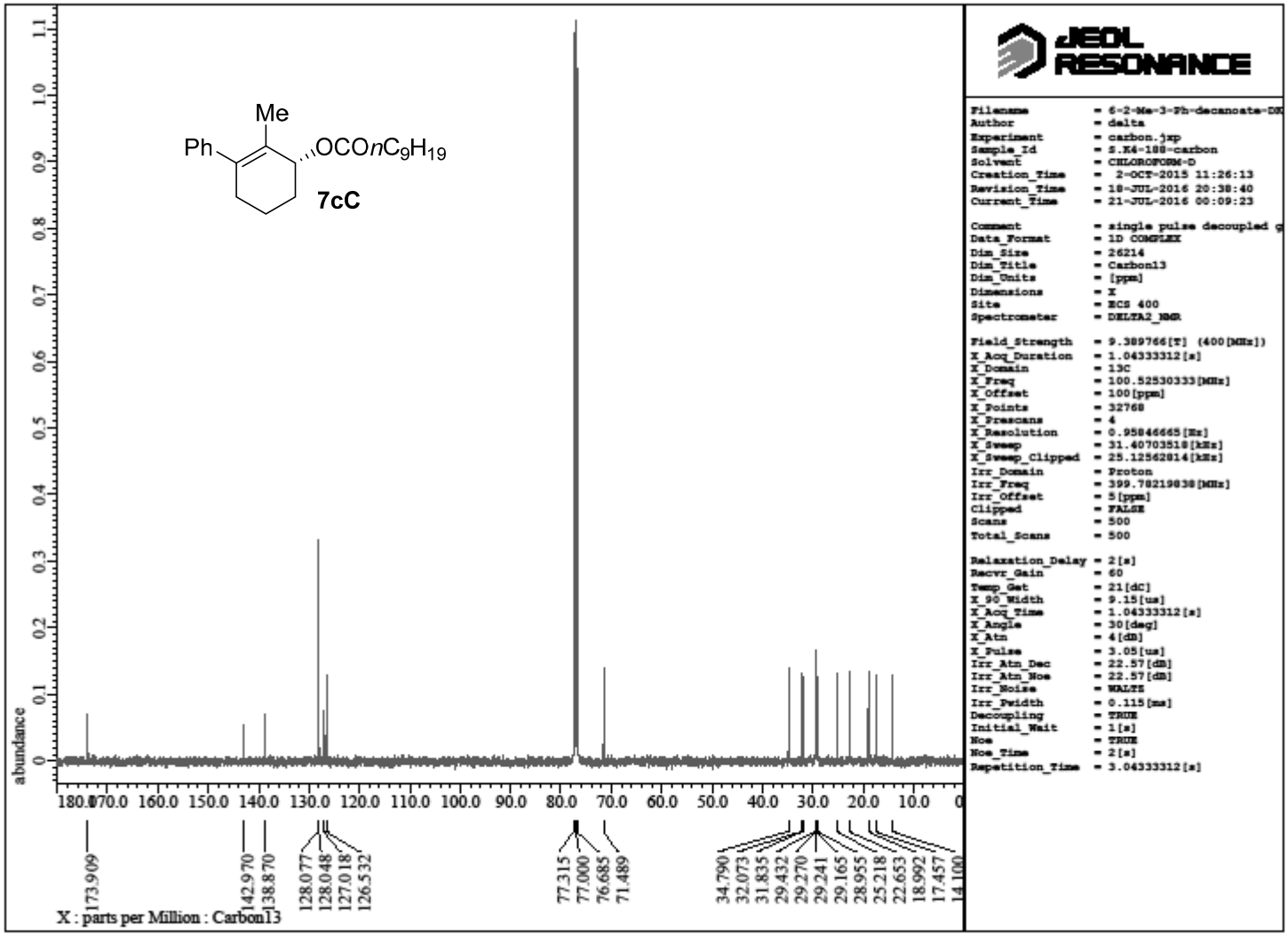

nBu
OAc
7bA
abundance
170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
170.935
144.953
119.210
77.315
77.000
76.676
68.905
37.346
29.546
28.336
28.269
22.415
21.499
19.154
13.957
X : parts per Million : Carbon13
JEOL RESONANCE
Filename = 6-nBu-acetate-DKR-data-c
Author = delta
Experiment = carbon.jxp
Sample_Id = 6-nBu-acetate-DKR-data-c
Solvent = CHLOROFORM-D
Creation_Time = 19-JUL-2016 21:49:27
Revision_Time = 21-JUL-2016 00:13:32
Current_Time = 21-JUL-2016 00:13:49
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 512
Total_Scans = 512
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 21.2[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

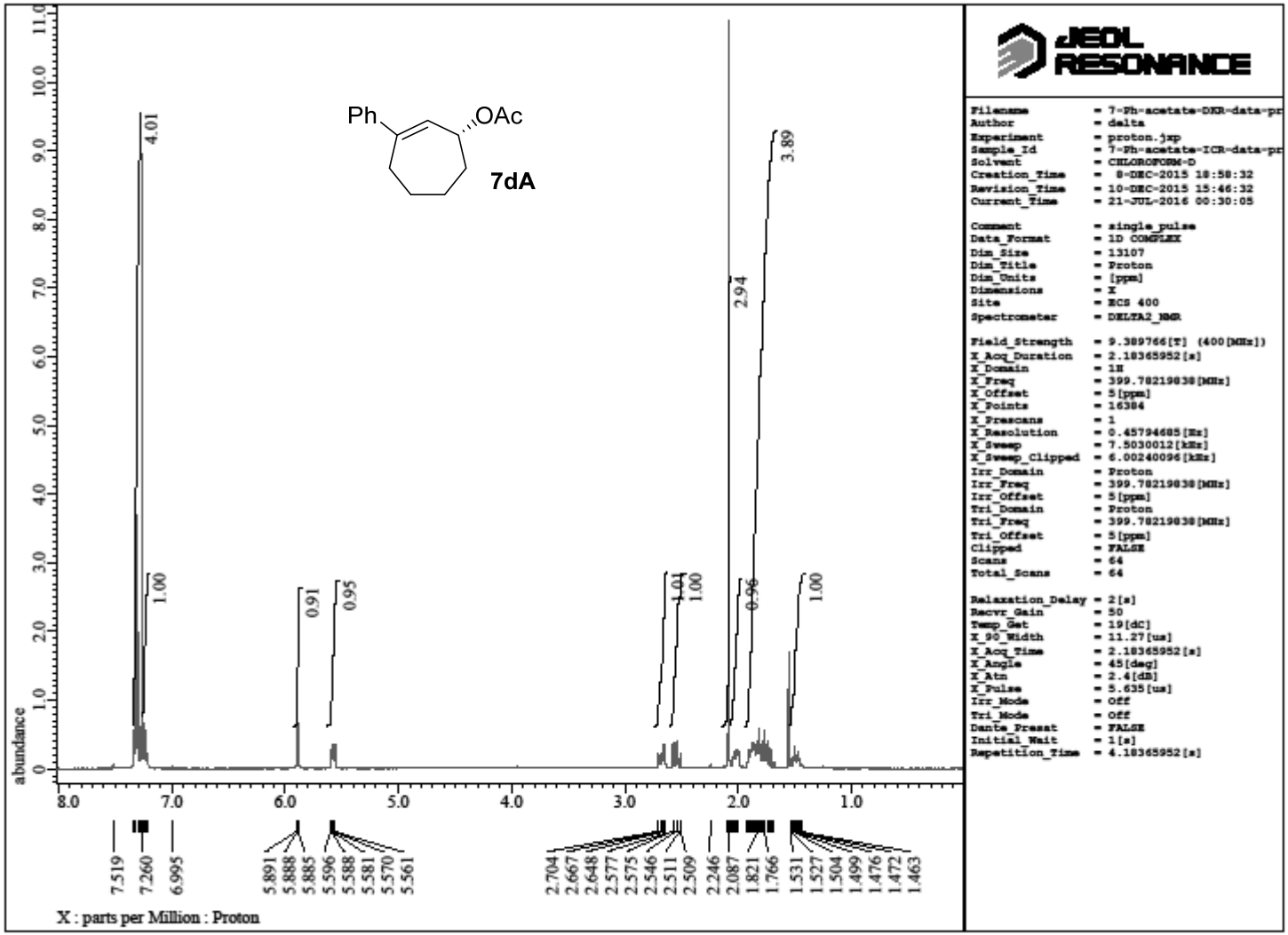

nBu
OCOnC3H7
7bB
5.99
5.92
4.06
4.00
1.93
0.96
0.94
abundance
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.260
5.441
5.436
5.433
5.274
2.285
2.267
2.261
2.248
1.678
1.660
1.640
1.387
1.293
0.960
0.941
0.923
0.908
0.890
0.873
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-nBu-butyrate-DKR-data-
Author = delta
Experiment = proton.jxp
Sample_Id = 6-nBu-butyrate-DKR-data-
Solvent = CHLOROFORM-D
Creation_Time = 11-DEC-2015 12:30:28
Revision_Time = 14-DEC-2015 20:35:09
Current_Time = 21-JUL-2016 00:15:06
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 64
Total_Scans = 64
Relaxation_Delay = 2[s]
Recvr_Gain = 46
Temp_Get = 19.3[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]


Me
Ph
OAc
7cA
2.06
2.03
1.20
1.00
2.99
2.04
4.12
2.99
abundance
X : parts per Million : Proton
7.353
7.348
7.335
7.315
7.260
7.243
7.226
7.168
7.165
7.147
5.385
5.364
5.353
5.344
2.322
2.309
2.266
2.261
2.256
2.251
2.245
2.105
1.882
1.870
1.561
1.555
1.551
JEOL RESONANCE
Filename = 6-2-Me-3-Ph-acetate-DKR-
Author = delta
Experiment = proton.jxp
Sample_Id = S.K-6-2-Me-Ph-acetate-DK
Solvent = CHLOROFORM-D
Creation_Time = 23-JUN-2016 21:47:54
Revision_Time = 24-JUN-2016 15:20:28
Current_Time = 21-JUL-2016 00:08:33
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 20.2[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 7.18365952[s]


Me
Ph
OAc
7cA
abundance
X : parts per Million : Carbon13
171.097
142.913
139.022
128.077
128.020
126.875
126.551
77.315
77.000
76.685
71.804
32.054
28.889
21.404
18.944
17.437
JEOL RESONANCE
Filename = 6-2-Me-3-Ph-acetate-DKR-
Author = delta
Experiment = carbon.jxp
Sample_Id = S.K-6-2-Me-Ph-acetate-DK
Solvent = CHLOROFORM-D
Creation_Time = 23-JUN-2016 21:54:18
Revision_Time = 24-JUN-2016 12:39:43
Current_Time = 21-JUL-2016 00:07:08
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 20.5[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

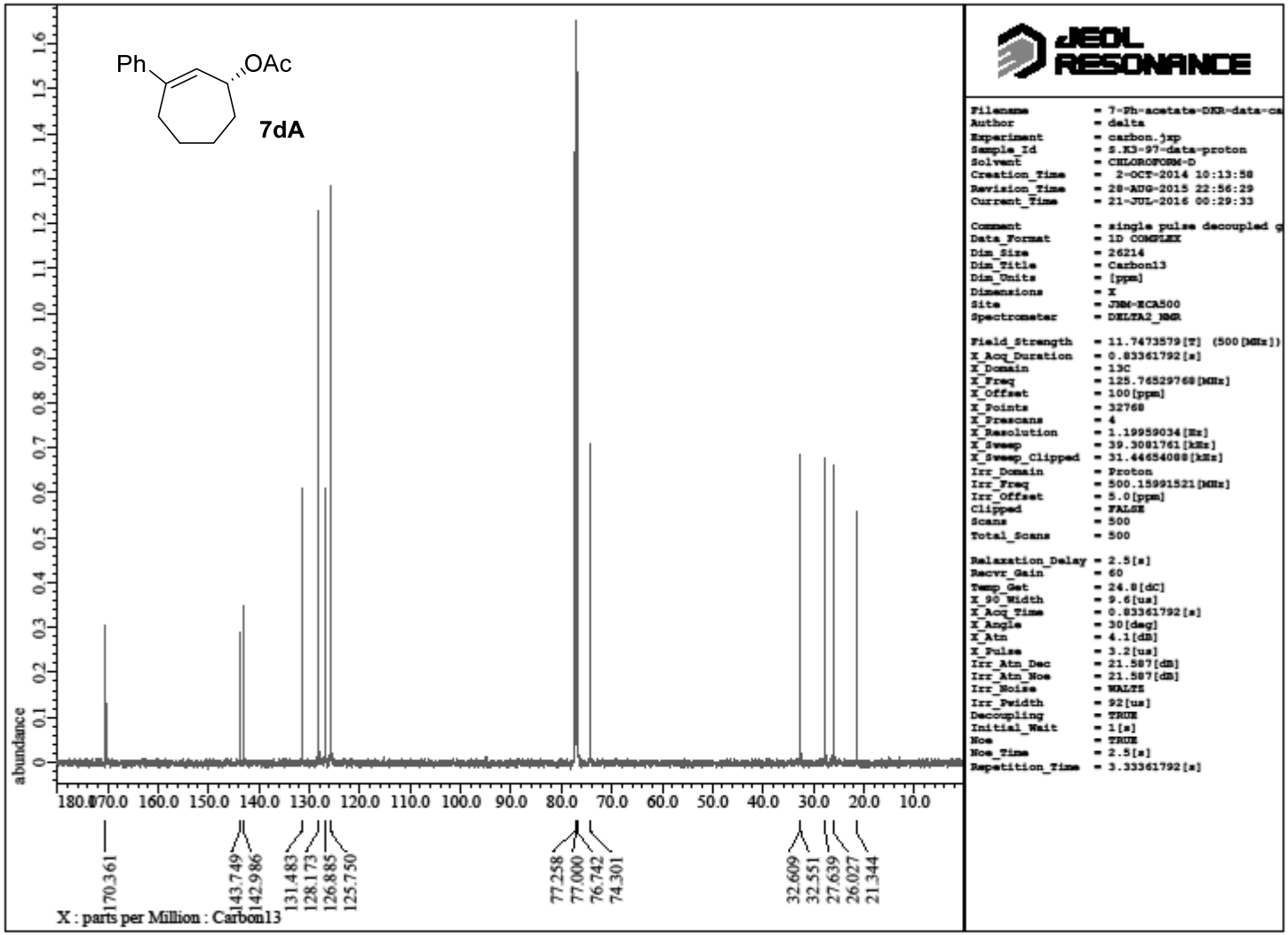

Me
Ph
OCOnC_9H_{19}
7cC
abundance
12.21
6.11
4.10
3.00
3.00
2.03
1.44
1.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.350
7.347
7.333
7.314
7.260
7.242
7.168
7.165
7.160
7.148
5.396
5.370
5.358
5.332
2.361
2.342
2.323
2.306
2.259
2.254
2.250
2.244
1.580
1.552
1.547
1.307
1.263
0.891
0.875
0.857
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-2-Me-3-Ph-decanoate-DX
Author = delta
Experiment = proton.jxp
Sample_Id = 6-2-Me-Ph-decanoate-DNR-
Solvent = CHLOROFORM-D
Creation_Time = 23-JUN-2016 19:35:16
Revision_Time = 23-JUN-2016 20:57:08
Current_Time = 21-JUL-2016 00:09:56
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 38
Temp_Get = 20.3[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 7.18365952[s]


Me
Ph
OCOnC_9H_{19}
7cC
abundance
X : parts per Million : Carbon13
173.909
142.970
138.870
128.077
128.048
127.018
126.532
77.315
77.000
76.685
71.489
34.790
32.073
31.835
29.432
29.270
29.241
29.165
28.955
25.218
22.653
18.992
17.457
14.100
JEOL RESONANCE
Filename = 6-2-Me-3-Ph-decanoate-DX
Author = delta
Experiment = carbon.jxp
Sample_Id = S.K4-188-carbon
Solvent = CHLOROFORM-D
Creation_Time = 2-OCT-2015 11:26:13
Revision_Time = 18-JUL-2016 20:38:40
Current_Time = 21-JUL-2016 00:09:23
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 500
Total_Scans = 500
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 21[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]



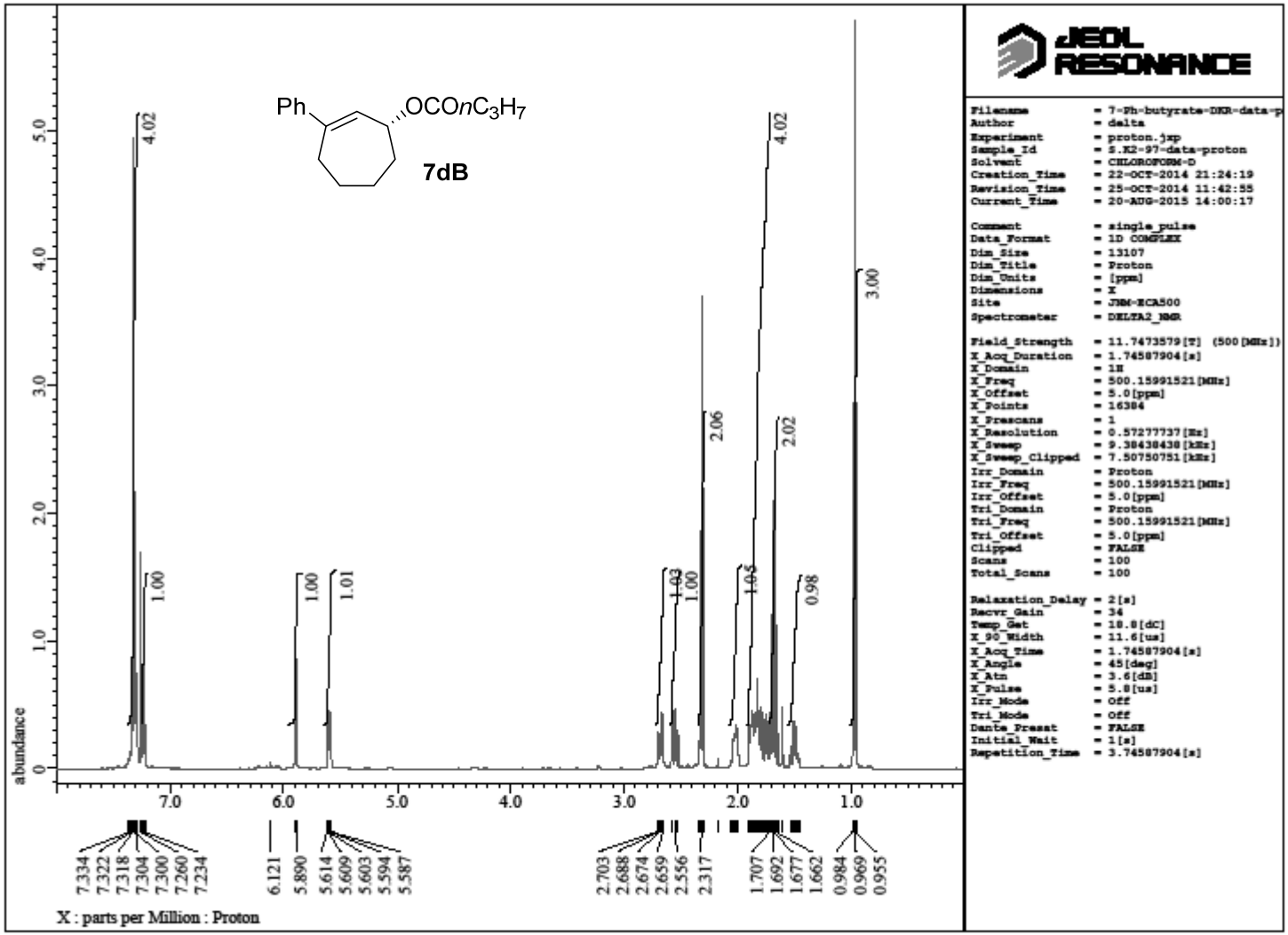
JEOL RESONANCE

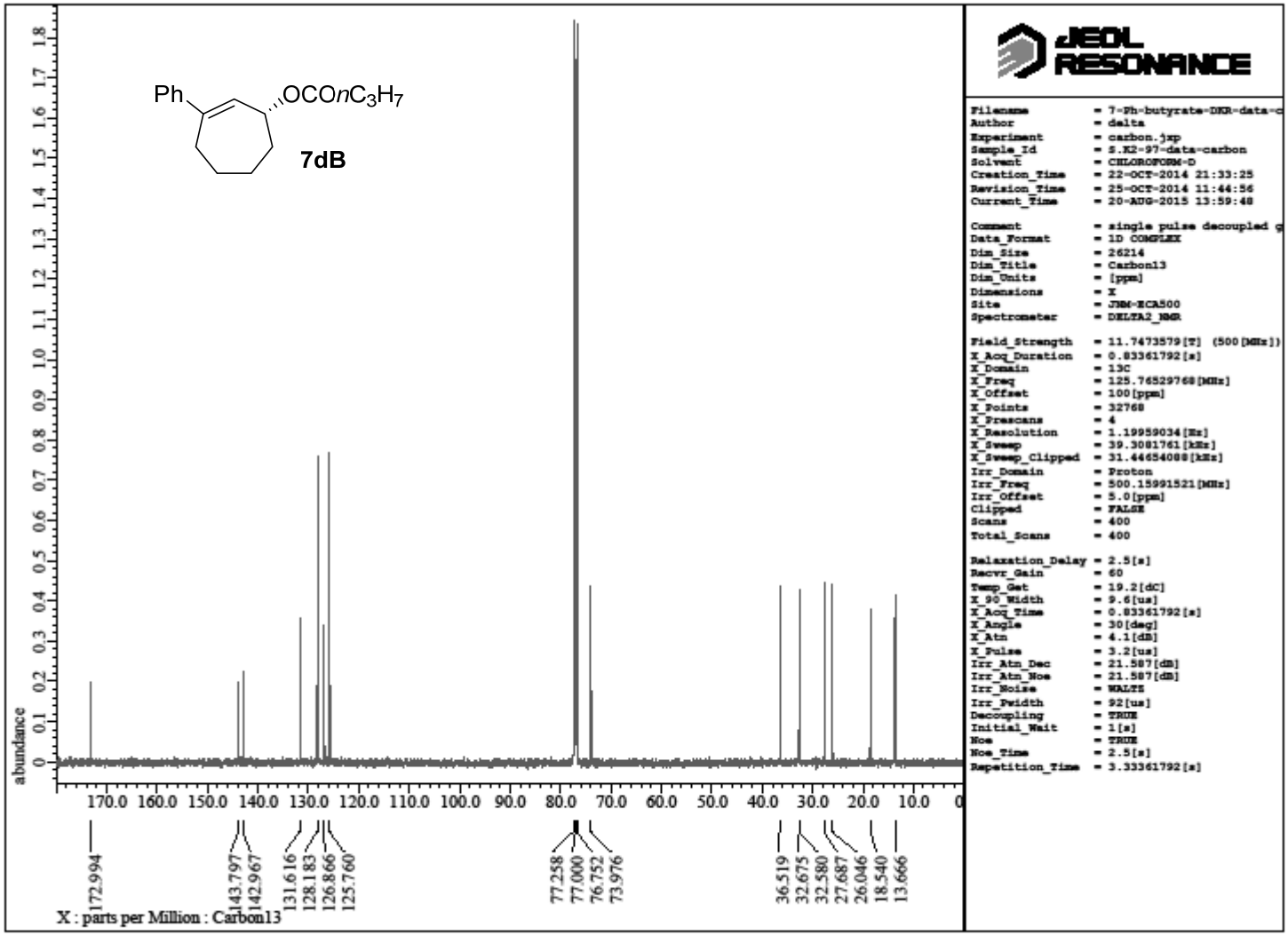
Filename	= 7-Ph-acetate-DKR-data-pr
Author	= delta
Experiment	= proton.jxp
Sample_Id	= 7-Ph-acetate-ICR-data-pr
Solvent	= CHLOROFORM-D
Creation_Time	= 8-DEC-2015 18:58:32
Revision_Time	= 10-DEC-2015 15:46:32
Current_Time	= 21-JUL-2016 00:30:05
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
X_Sweep_Clipped	= 6.00240096[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 64
Total_Scans	= 64
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 19[dC]
X_90_Width	= 11.27[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2.4[dB]
X_Pulse	= 5.635[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Preset	= FALSE
Initial_Wait	= 1[s]
RepetitIon_Time	= 4.18365952[s]

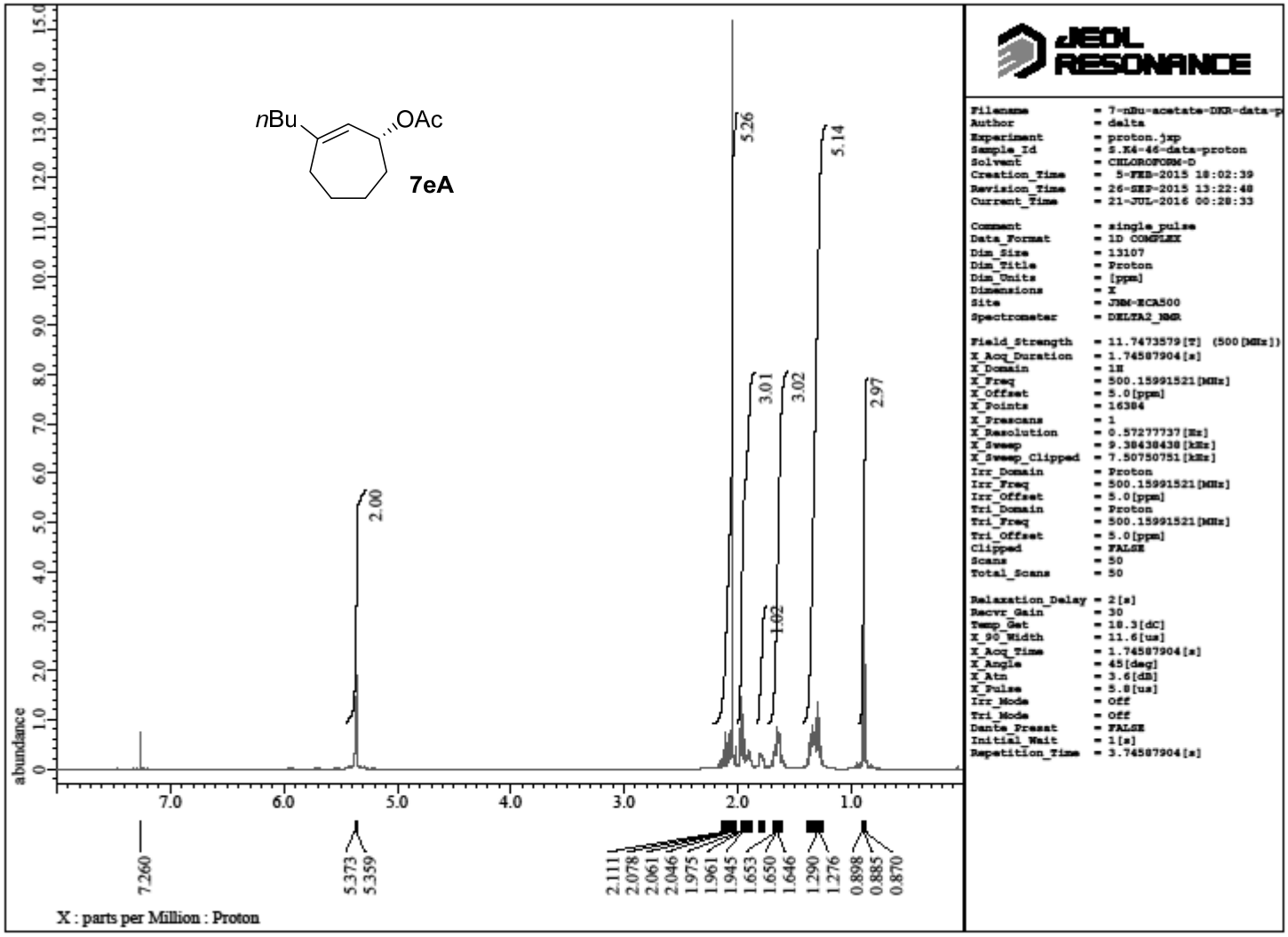


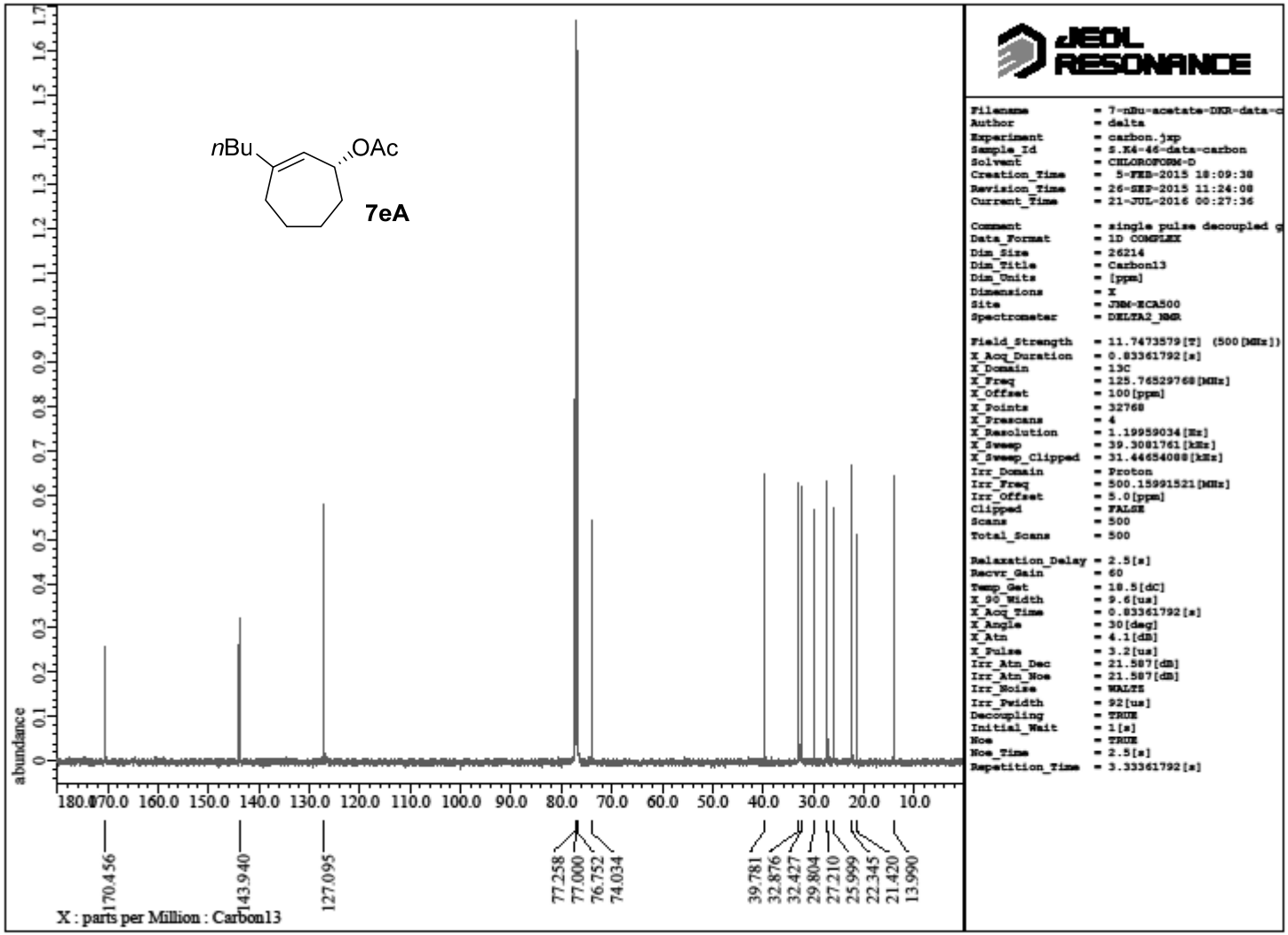
JEOL RESONANCE

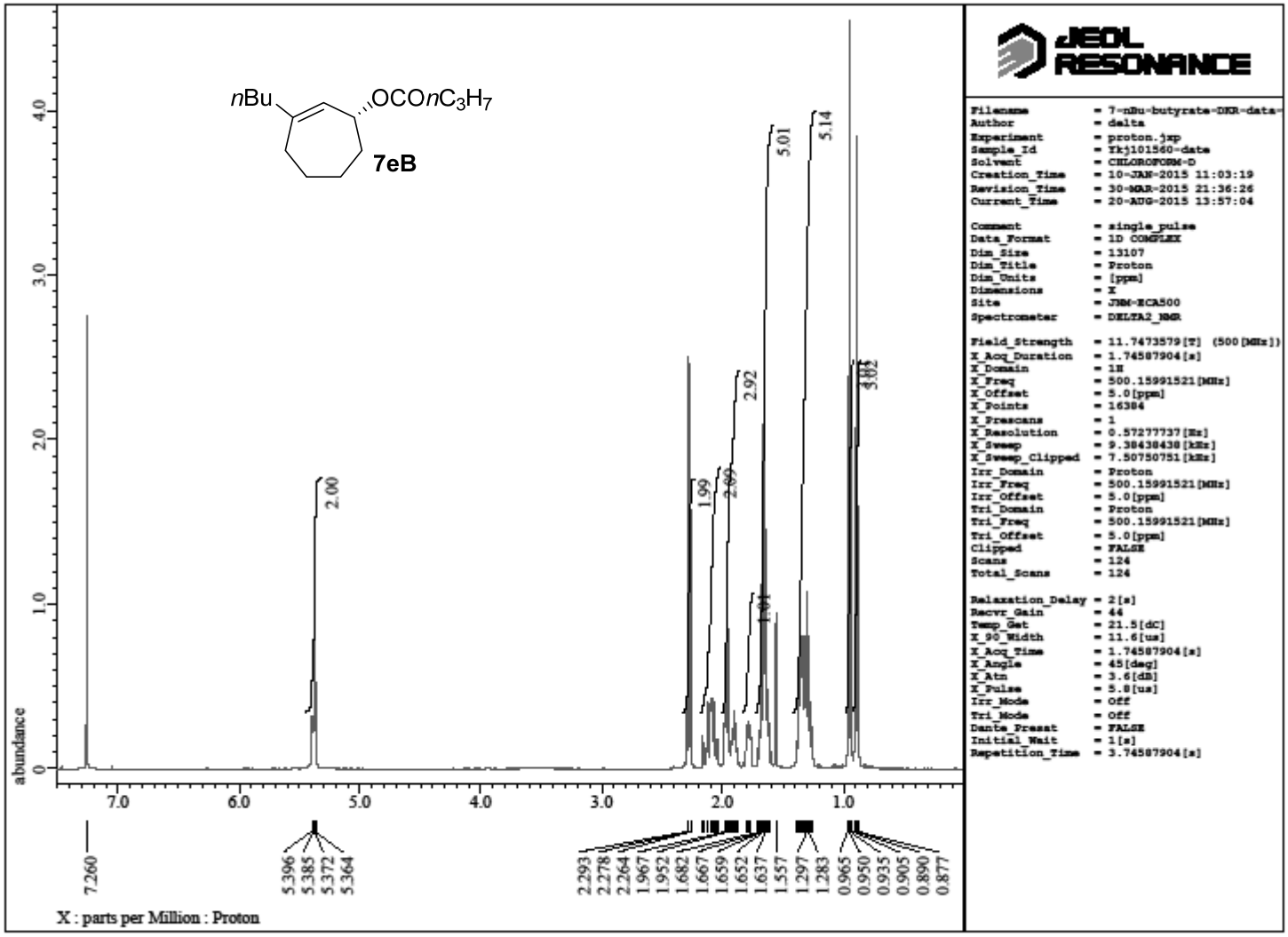
Filename	= 7-Ph-acetate-DKR-data-ca
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= S.K3-97-data-proton
Solvent	= CHLOROFORM-D
Creation_Time	= 2-OCT-2014 10:13:58
Revision_Time	= 28-AUG-2015 22:56:29
Current_Time	= 21-JUL-2016 00:29:33
Comment	= single pulse decoupled g
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECA500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.7473579[T] (500[MHz])
X_Acq_Duration	= 0.83361792[s]
X_Domain	= 13C
X_Freq	= 125.76529768[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.19959034[Hz]
X_Sweep	= 39.3081761[kHz]
X_Sweep_Clipped	= 31.44654088[kHz]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[MHz]
Irr_Offset	= 5.0[ppm]
Clipped	= FALSE
Scans	= 500
Total_Scans	= 500
Relaxation_Delay	= 2.5[s]
Recvr_Gain	= 60
Temp_Get	= 24.8[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 0.83361792[s]
X_Angle	= 30[deg]
X_Atn	= 4.1[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 21.587[dB]
Irr_Atn_Noe	= 21.587[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 92[us]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2.5[s]
Repetition_Time	= 3.33361792[s]

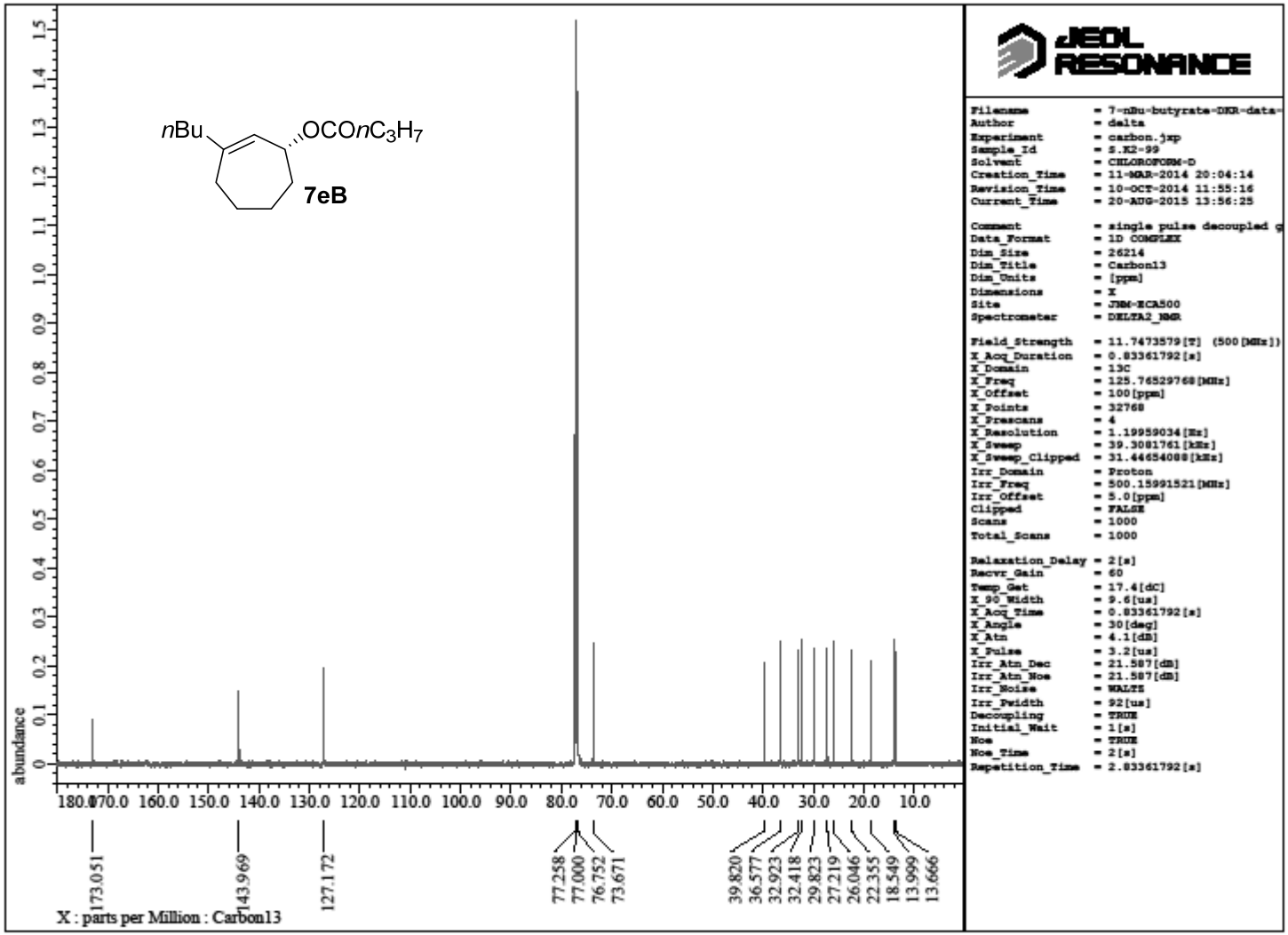

Ph
OCOnC_3H_7
7dB
4.02
1.00
1.00
1.01
1.03
1.00
2.06
1.04
4.02
2.02
0.98
3.00
abundance
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.334
7.322
7.318
7.304
7.300
7.260
7.234
6.121
5.890
5.614
5.609
5.603
5.594
5.587
2.703
2.688
2.674
2.659
2.556
2.317
1.707
1.692
1.677
1.662
0.984
0.969
0.955
X : parts per Million : Proton
JEOL RESONANCE
Filename = 7-Ph-butyrate-DKR-data-p
Author = delta
Experiment = proton.jxp
Sample_Id = S.K2-97-data-proton
Solvent = CHLOROFORM-D
Creation_Time = 22-OCT-2014 21:24:19
Revision_Time = 25-OCT-2014 11:42:55
Current_Time = 20-AUG-2015 14:00:17
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 100
Total_Scans = 100
Relaxation_Delay = 2[s]
Recvr_Gain = 34
Temp_Get = 18.8[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 5.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 3.74587904[s]

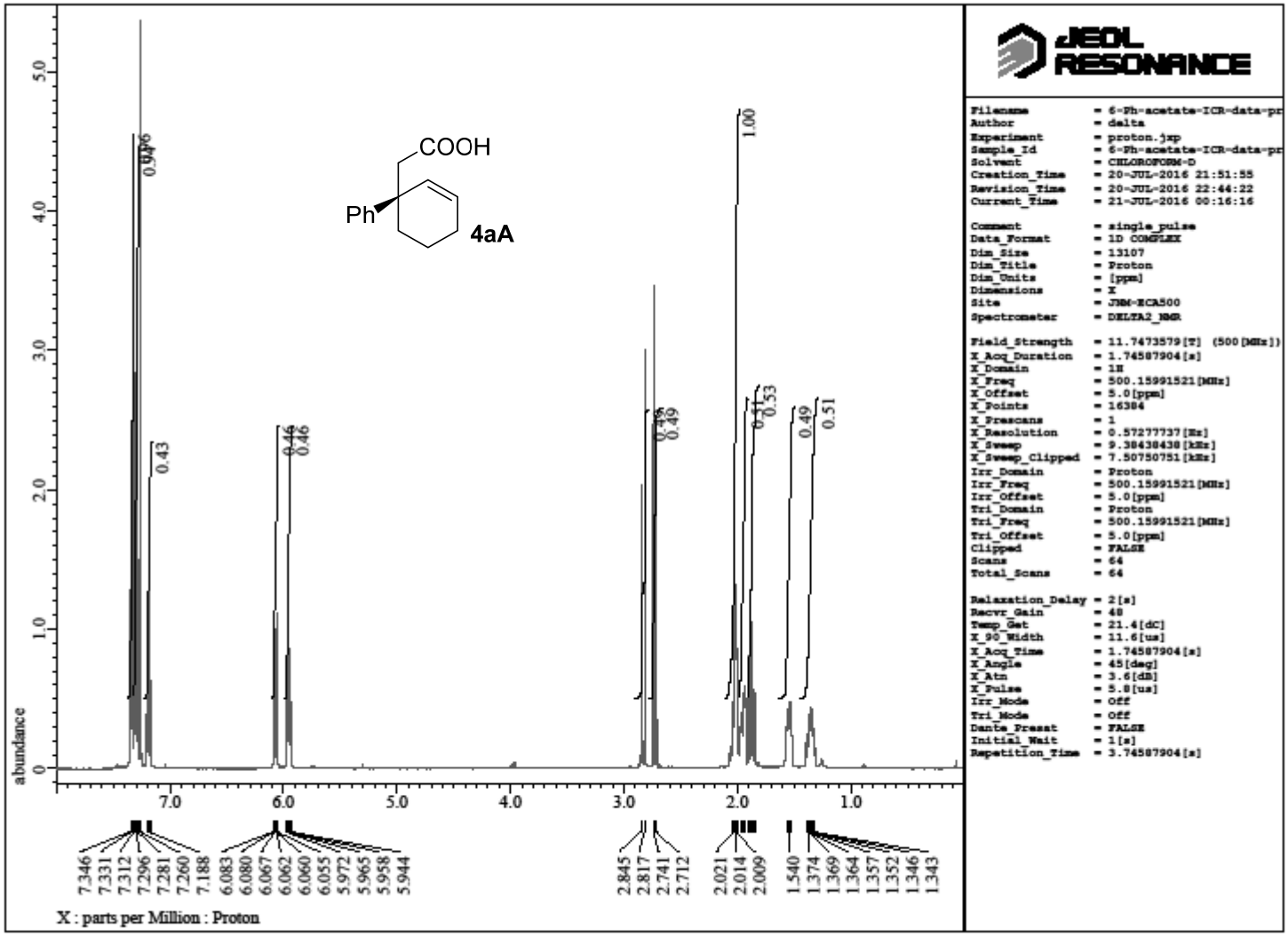

Ph
OCOnC_3H_7
7dB
abundance
170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
172.994
143.797
142.967
131.616
128.183
126.866
125.760
77.258
77.000
76.752
73.976
36.519
32.675
32.580
27.687
26.046
18.540
13.666
X : parts per Million : Carbon13
JEOL RESONANCE
Filename = 7-Ph-butyrate-DKR-data-c
Author = delta
Experiment = carbon.jxp
Sample_Id = S.K2-97-data-carbon
Solvent = CHLOROFORM-D
Creation_Time = 22-OCT-2014 21:33:25
Revision_Time = 25-OCT-2014 11:44:56
Current_Time = 20-AUG-2015 13:59:48
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 400
Total_Scans = 400
Relaxation_Delay = 2.5[s]
Recvr_Gain = 60
Temp_Get = 19.2[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 4.1[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.587[dB]
Irr_Atn_Noe = 21.587[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2.5[s]
Repetition_Time = 3.33361792[s]

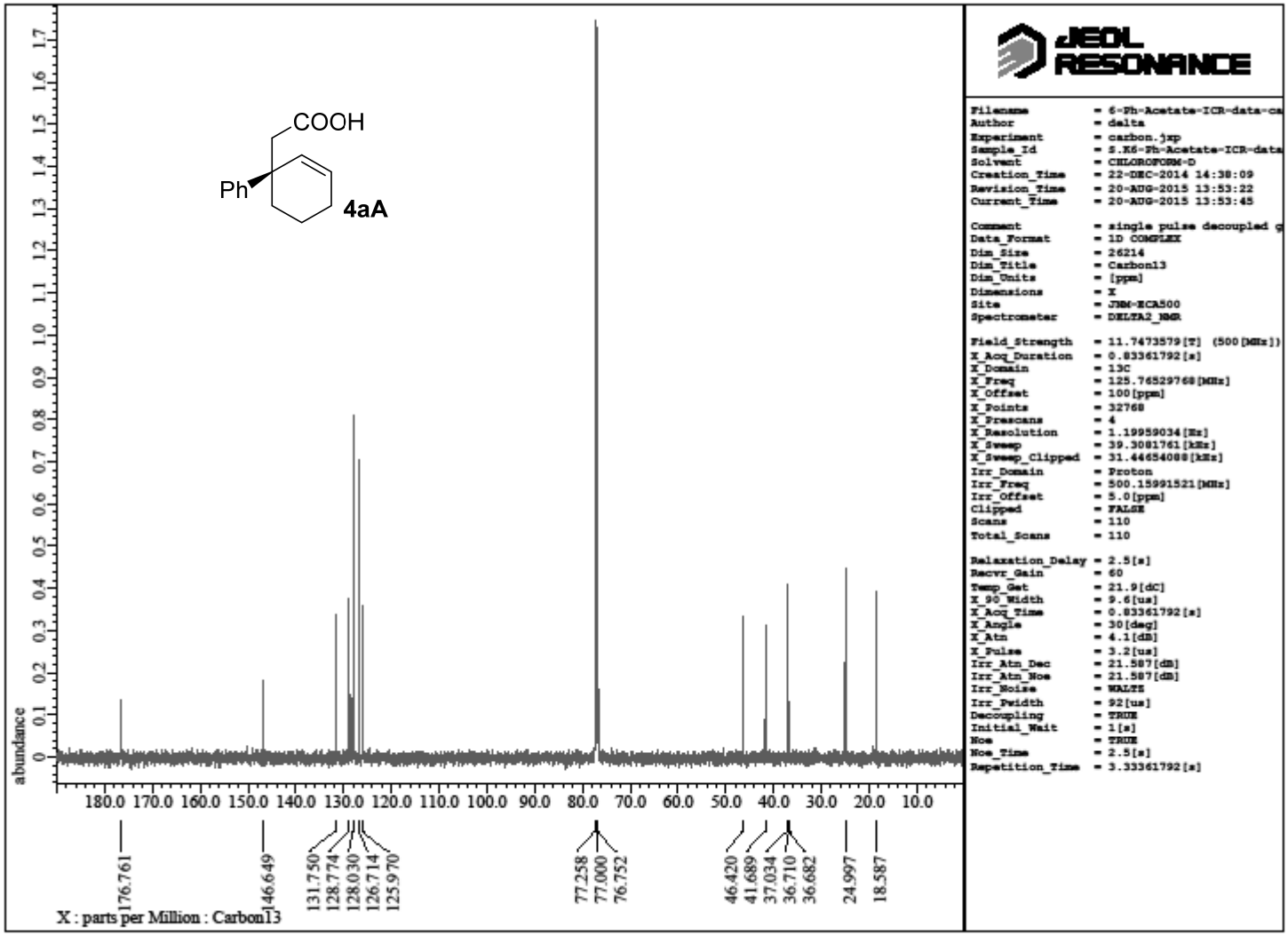

nBu
OAc
7eA
5.26
5.14
3.01
3.02
2.97
2.00
1.02
abundance
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.260
5.373
5.359
2.111
2.078
2.061
2.046
1.975
1.961
1.945
1.653
1.650
1.646
1.290
1.276
0.898
0.885
0.870
X : parts per Million : Proton
JEOL RESONANCE
Filename = 7-nBu-acetate-DKR-data-p
Author = delta
Experiment = proton.jxp
Sample_Id = S.K4-46-data-proton
Solvent = CHLOROFORM-D
Creation_Time = 5-FEB-2015 18:02:39
Revision_Time = 26-SEP-2015 13:22:48
Current_Time = 21-JUL-2016 00:28:33
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 50
Total_Scans = 50
Relaxation_Delay = 2[s]
Recvr_Gain = 30
Temp_Get = 18.3[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 5.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 3.74587904[s]

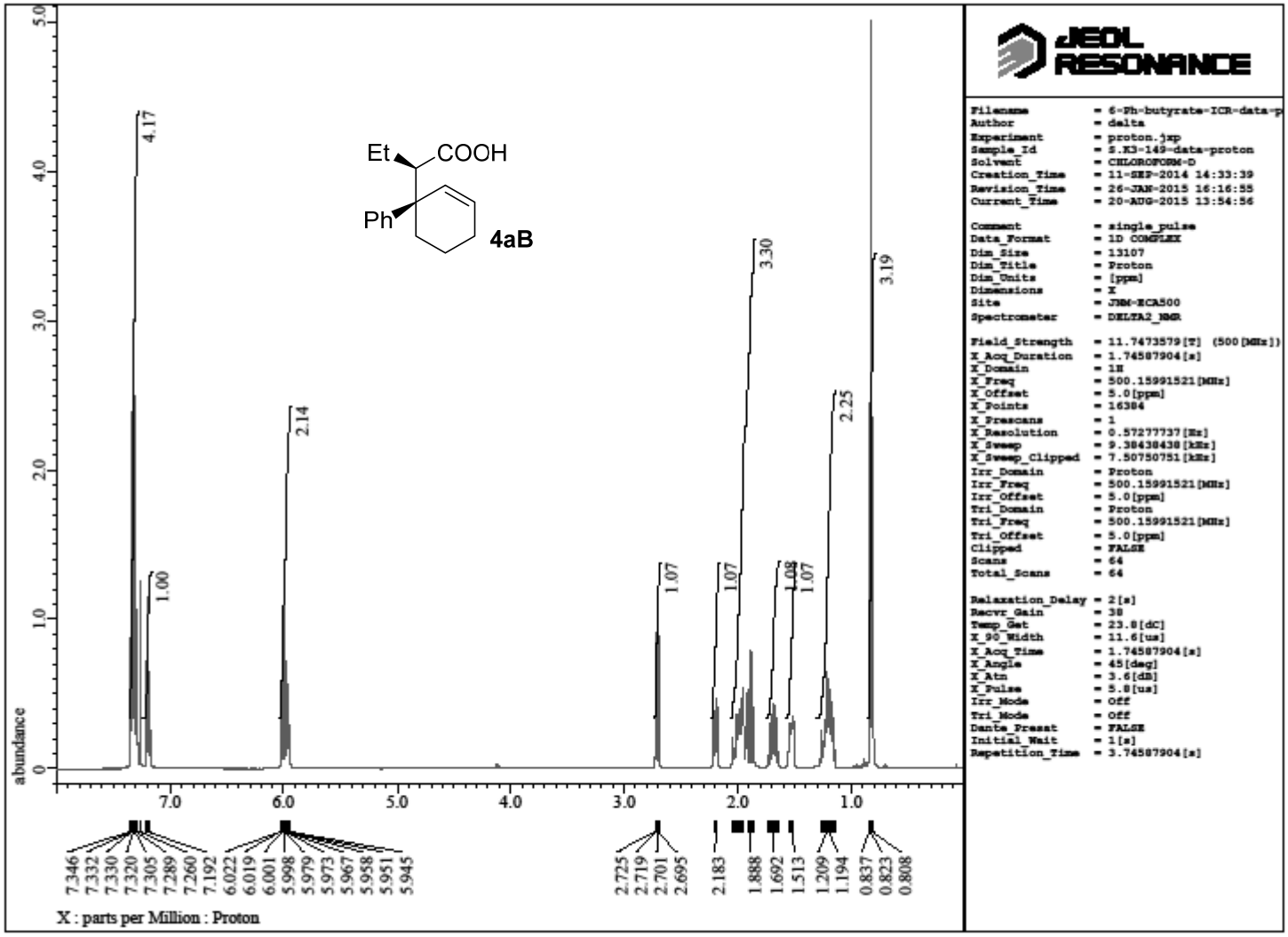

nBu
OAc
7eA
abundance
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
170.456
143.940
127.095
77.258
77.000
76.752
74.034
39.781
32.876
32.427
29.804
27.210
25.999
22.345
21.420
13.990
X : parts per Million : Carbon13
JEOL RESONANCE
Filename = 7-nBu-acetate-DKR-data-c
Author = delta
Experiment = carbon.jxp
Sample_Id = S.K4-46-data-carbon
Solvent = CHLOROFORM-D
Creation_Time = 5-FEB-2015 18:09:38
Revision_Time = 26-SEP-2015 11:24:08
Current_Time = 21-JUL-2016 00:27:36
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 500
Total_Scans = 500
Relaxation_Delay = 2.5[s]
Recvr_Gain = 60
Temp_Get = 18.5[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 4.1[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.587[dB]
Irr_Atn_Noe = 21.587[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2.5[s]
Repetition_Time = 3.33361792[s]

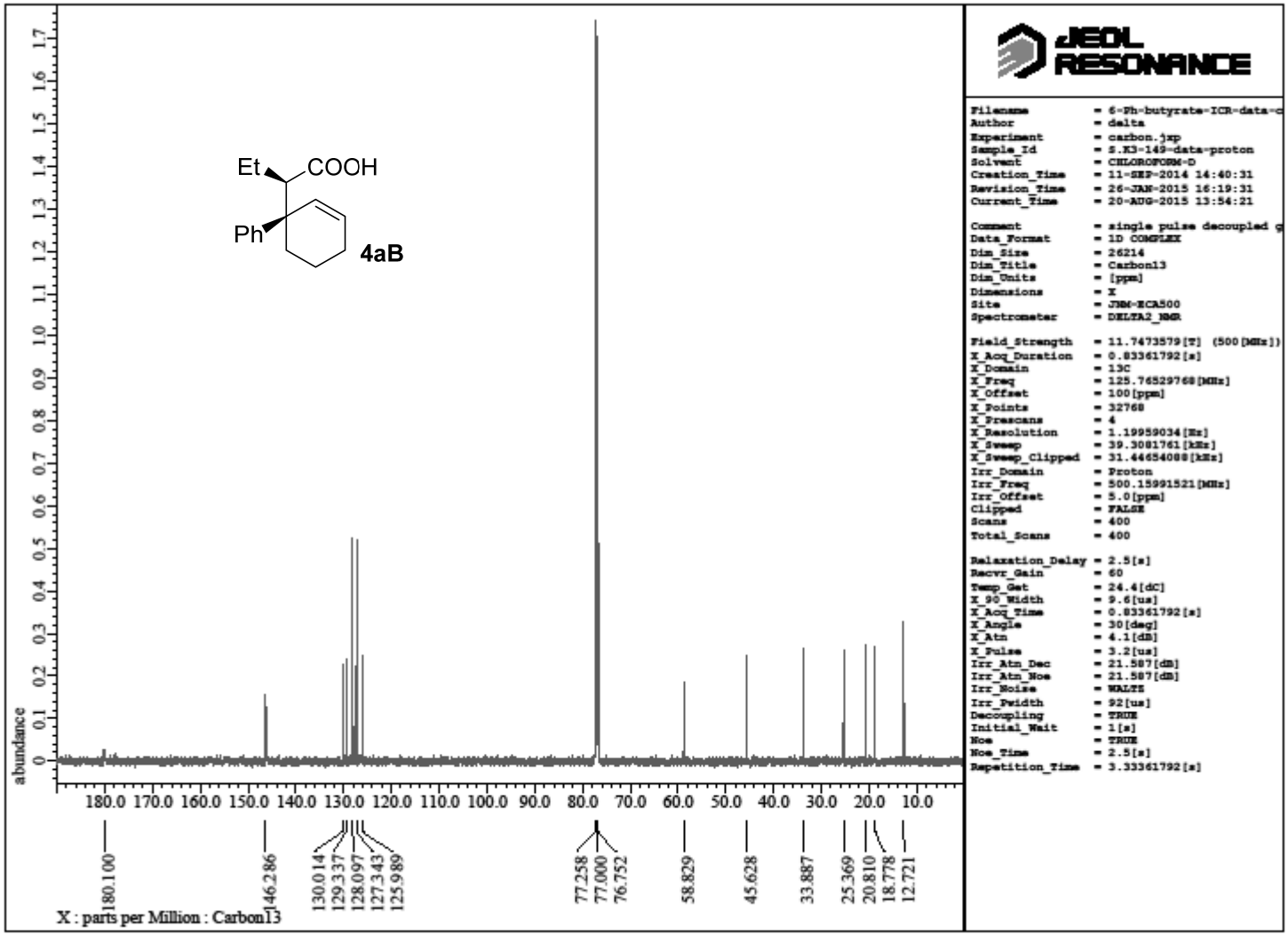

nBu
OCOnC3H7
7eB
abundance
X : parts per Million : Proton
JEOL RESONANCE
Filename = 7-nBu-butyrate-DKR-data-
Author = delta
Experiment = proton.jxp
Sample_Id = Tkj101560-data
Solvent = CHLOROFORM-D
Creation_Time = 10-JAN-2015 11:03:19
Revision_Time = 30-MAR-2015 21:36:26
Current_Time = 20-AUG-2015 13:57:04
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 124
Total_Scans = 124
Relaxation_Delay = 2[s]
Recvr_Gain = 44
Temp_Get = 21.5[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 5.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 3.74587904[s]

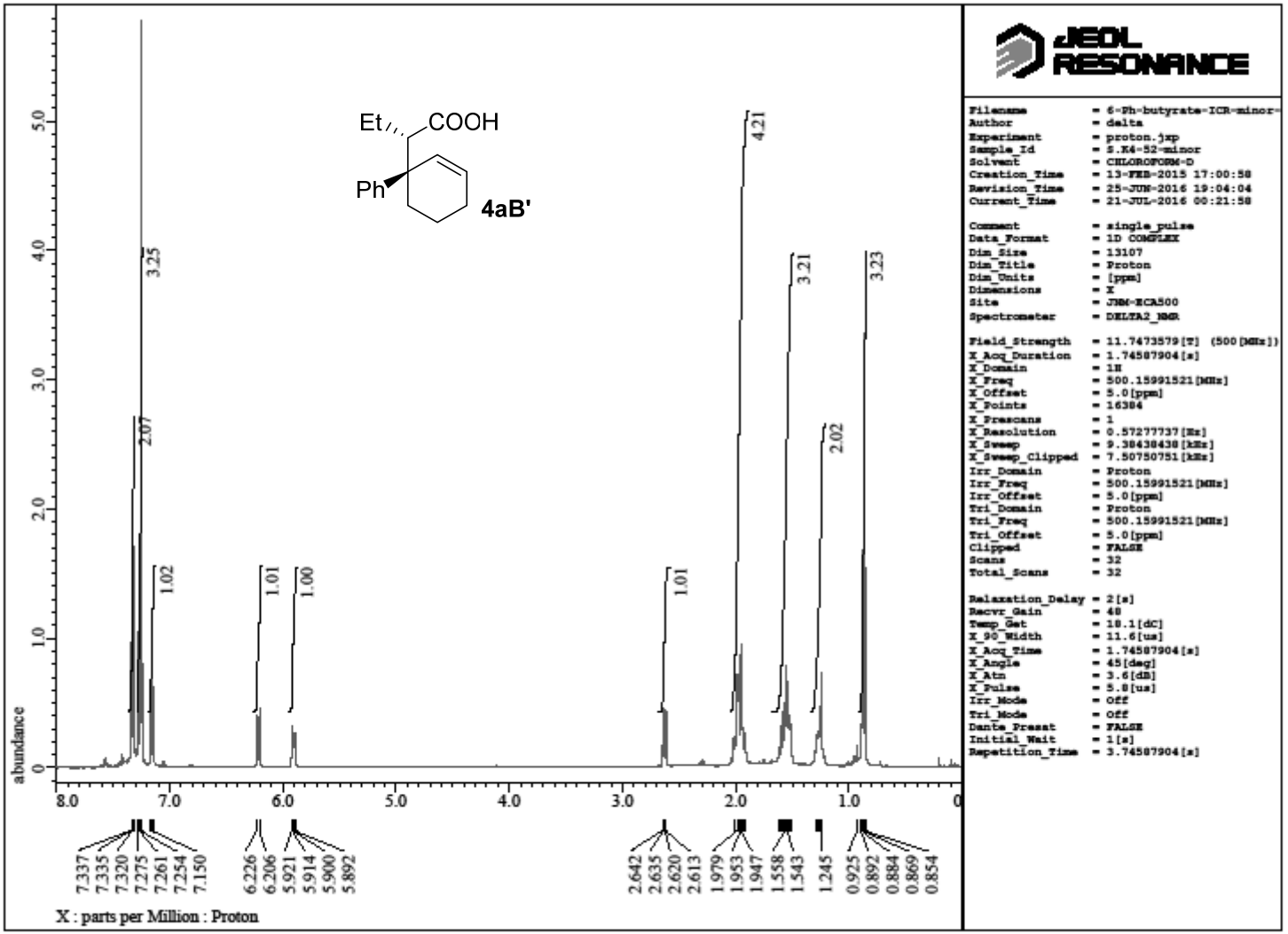

nBu
OCOnC3H7
7eB
abundance
X : parts per Million : Carbon13
173.051
143.969
127.172
77.258
77.000
76.752
73.671
39.820
36.577
32.923
32.418
29.823
27.219
26.046
22.355
18.549
13.999
13.666
JEOL RESONANCE
Filename = 7-nBu-butyrate-DKR-data-
Author = delta
Experiment = carbon.jxp
Sample_Id = S.K2-99
Solvent = CHLOROFORM-D
Creation_Time = 11-MAR-2014 20:04:14
Revision_Time = 10-OCT-2014 11:55:16
Current_Time = 20-AUG-2015 13:56:25
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 1000
Total_Scans = 1000
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 17.4[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 4.1[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.587[dB]
Irr_Atn_Noe = 21.587[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.83361792[s]

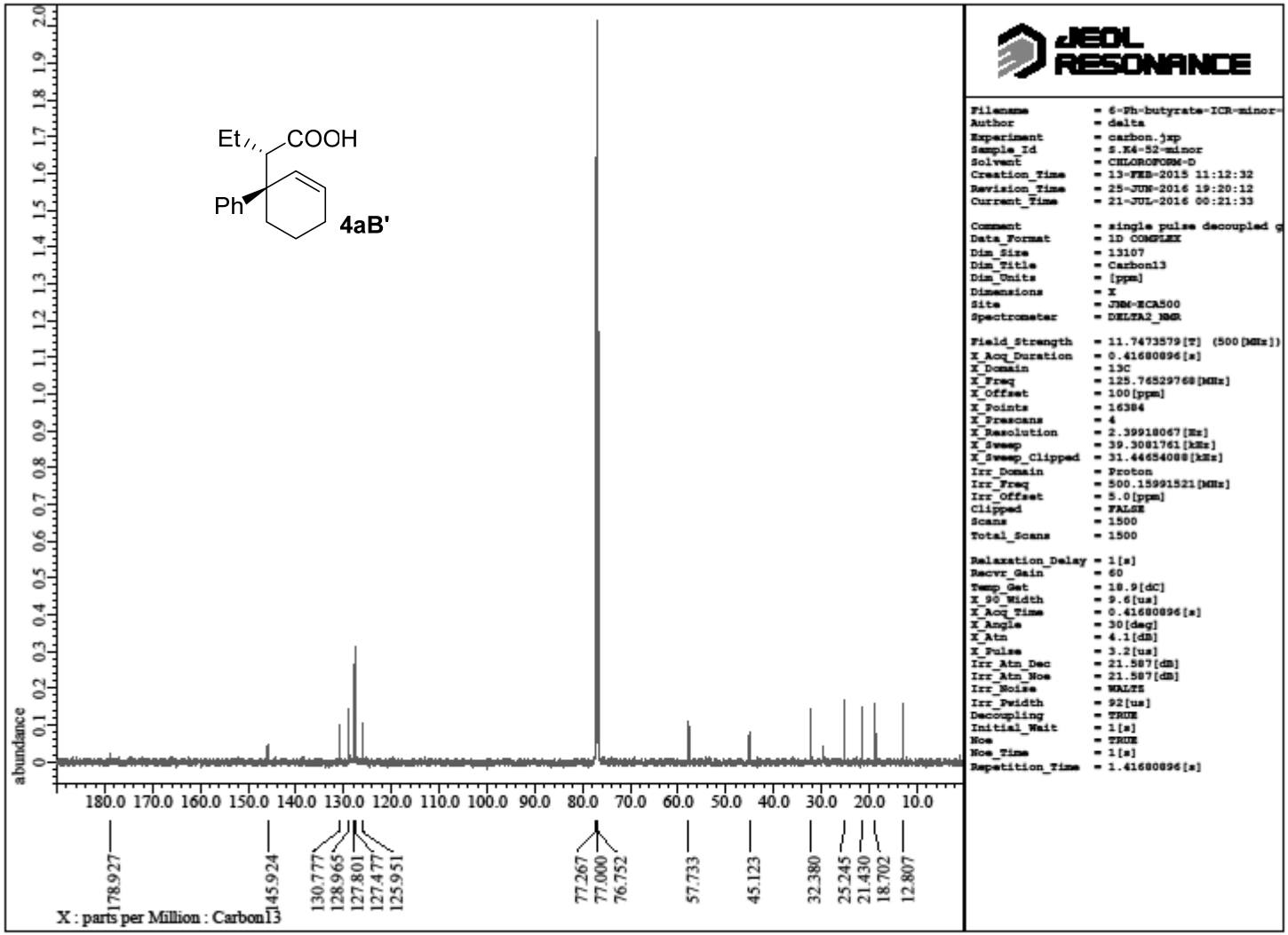

COOH
Ph
4aA
abundance
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-Ph-acetate-ICR-data-pr
Author = delta
Experiment = proton.jxp
Sample_Id = 6-Ph-acetate-ICR-data-pr
Solvent = CHLOROFORM-D
Creation_Time = 20-JUL-2016 21:51:55
Revision_Time = 20-JUL-2016 22:44:22
Current_Time = 21-JUL-2016 00:16:16
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 64
Total_Scans = 64
Relaxation_Delay = 2[s]
Recvr_Gain = 48
Temp_Get = 21.4[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 5.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 3.74587904[s]

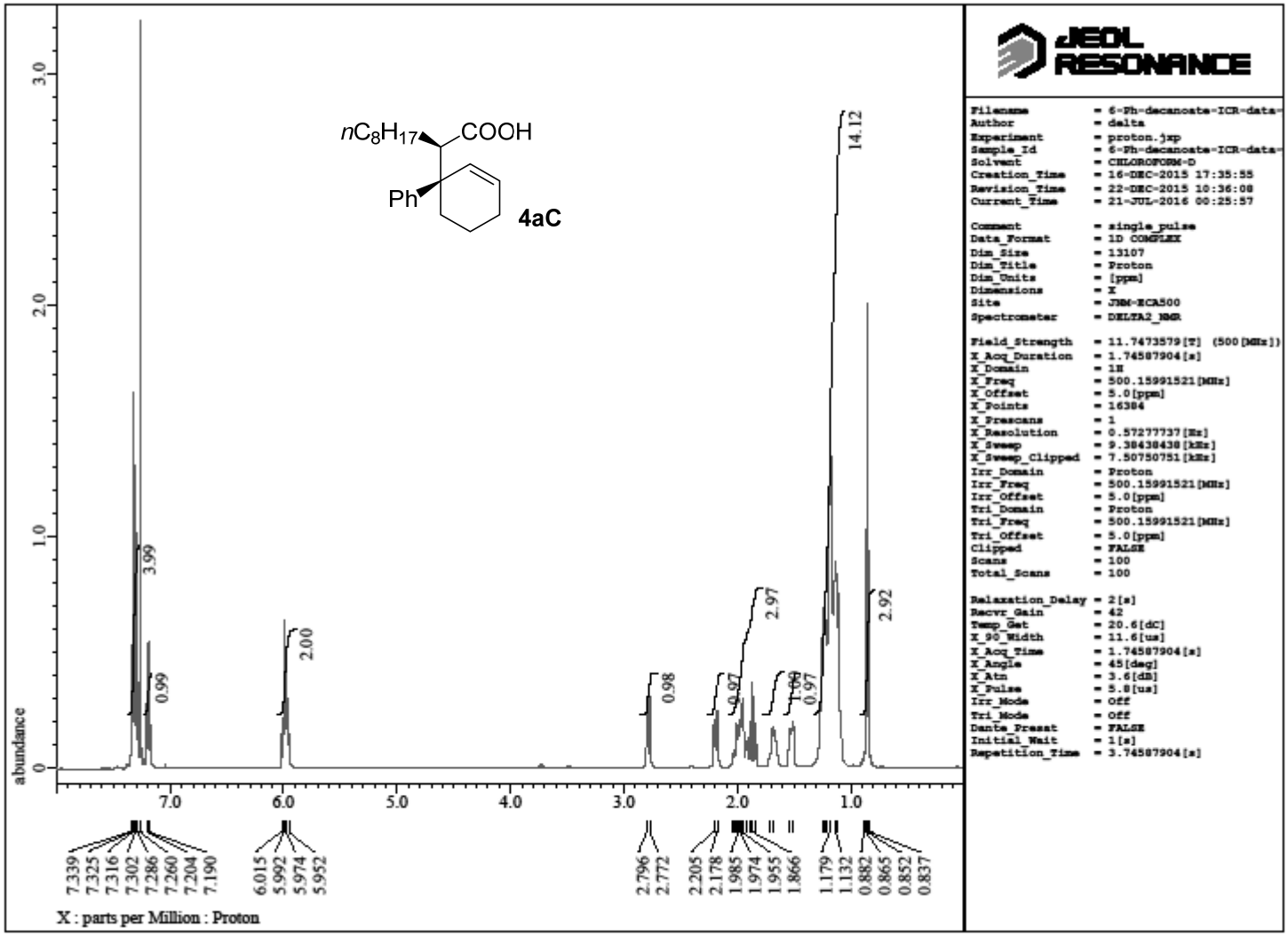

COOH
Ph
4aA
abundance
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
176.761
146.649
131.750
128.774
128.030
126.714
125.970
77.258
77.000
76.752
46.420
41.689
37.034
36.710
36.682
24.997
18.587
X : parts per Million : Carbon13
JEOL RESONANCE
Filename = 6-Ph-Acetate-ICR-data-ca
Author = delta
Experiment = carbon.jxp
Sample_Id = S.K6-Ph-Acetate-ICR-data
Solvent = CHLOROFORM-D
Creation_Time = 22-DEC-2014 14:38:09
Revision_Time = 20-AUG-2015 13:53:22
Current_Time = 20-AUG-2015 13:53:45
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 110
Total_Scans = 110
Relaxation_Delay = 2.5[s]
Recvr_Gain = 60
Temp_Get = 21.9[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 4.1[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.587[dB]
Irr_Atn_Noe = 21.587[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2.5[s]
Repetition_Time = 3.33361792[s]

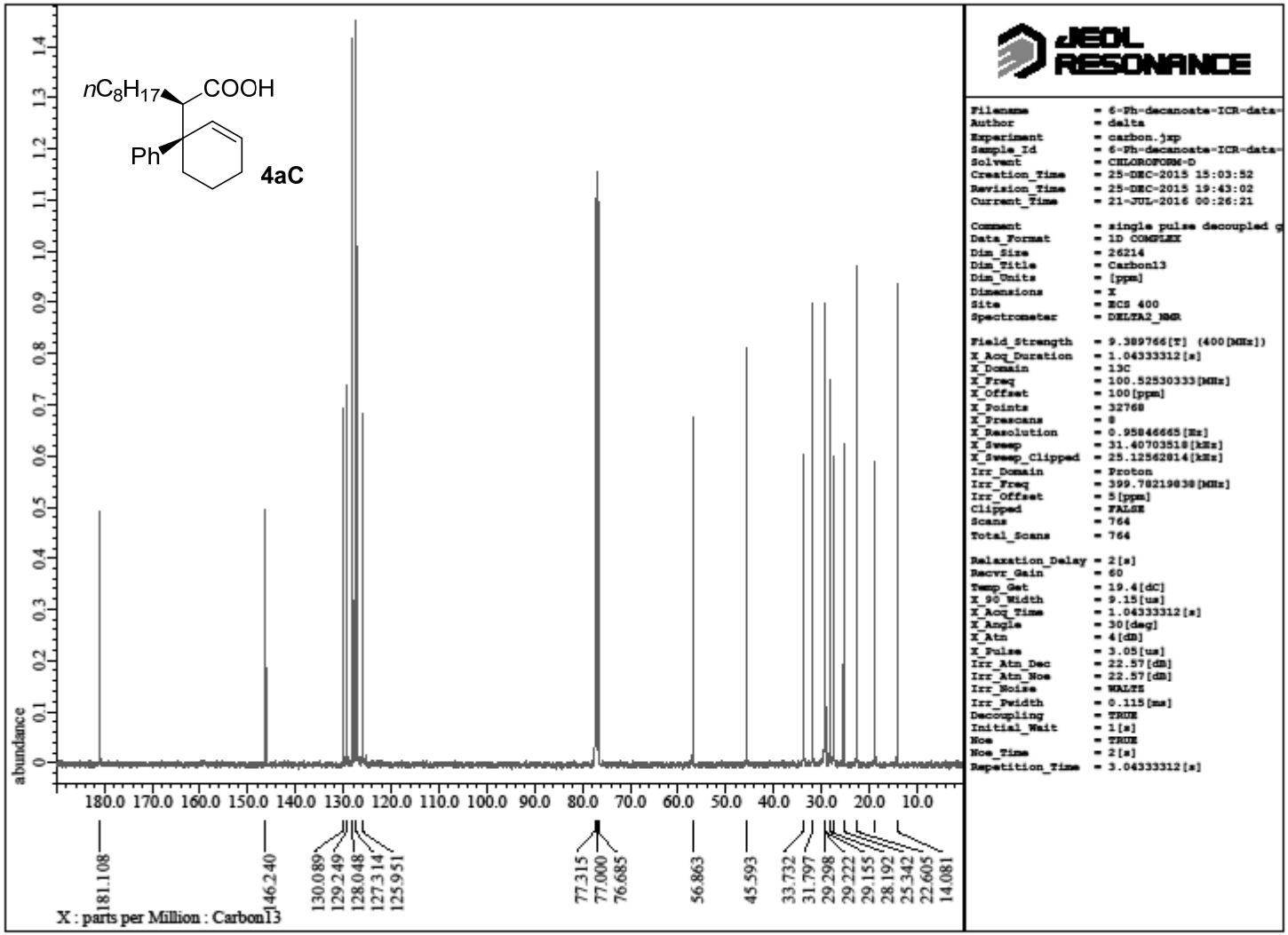

Et
COOH
Ph
4aB
4.17
1.00
2.14
1.07
1.07
1.08
1.07
3.30
2.25
3.19
abundance
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-Ph-butyrate-ICR-data-p
Author = delta
Experiment = proton.jxp
Sample_Id = S.K3-149-data-proton
Solvent = CHLOROFORM-D
Creation_Time = 11-SEP-2014 14:33:39
Revision_Time = 26-JAN-2015 16:16:55
Current_Time = 20-AUG-2015 13:54:56
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 64
Total_Scans = 64
Relaxation_Delay = 2[s]
Recvr_Gain = 38
Temp_Get = 23.8[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 5.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Preset = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 3.74587904[s]

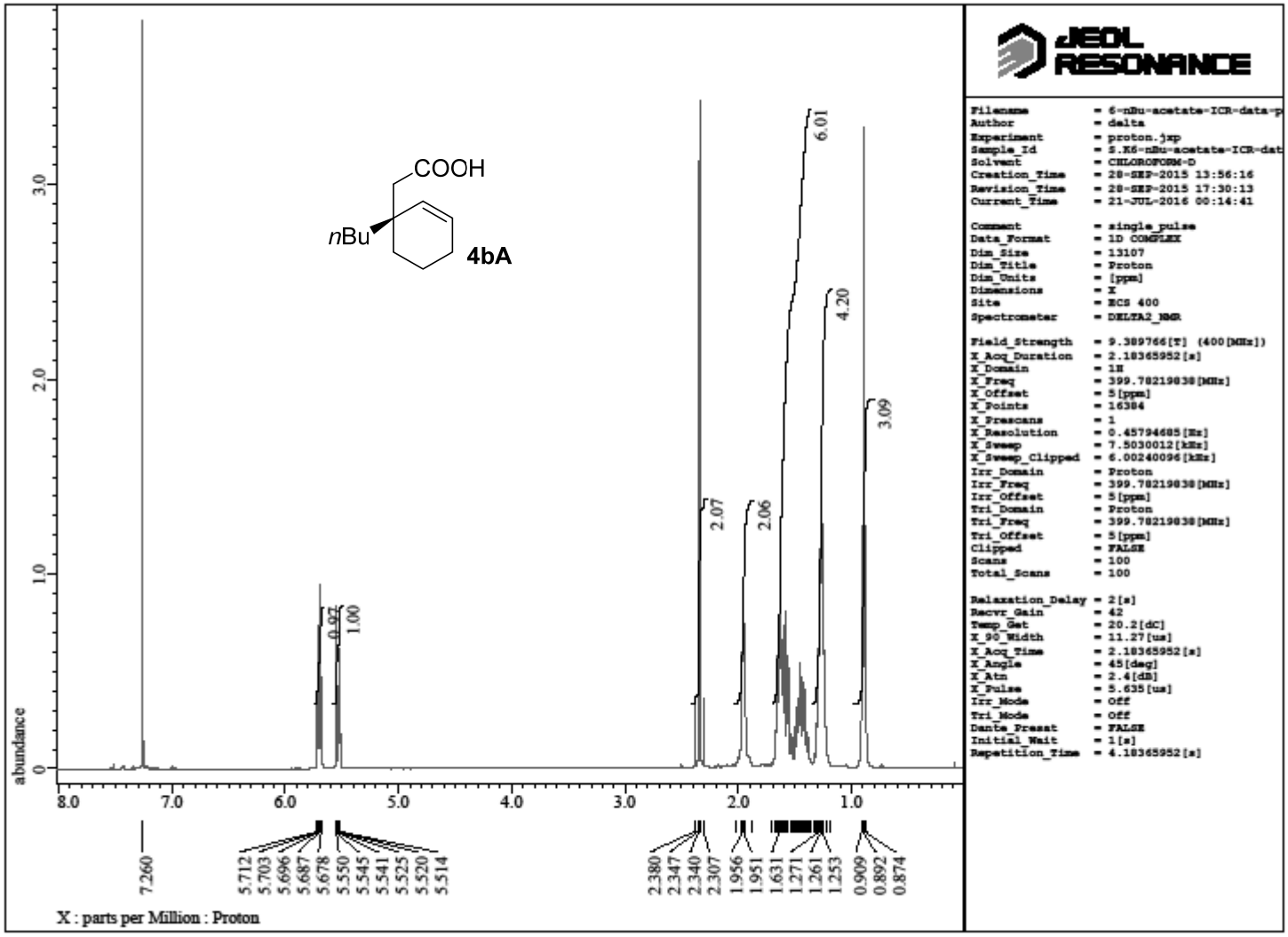

Et
COOH
Ph
4aB
abundance
180.100
146.286
130.014
129.337
128.097
127.343
125.989
77.258
77.000
76.752
58.829
45.628
33.887
25.369
20.810
18.778
12.721
X : parts per Million : Carbon13
JEOL RESONANCE
Filename = 6-Ph-butyrate-ICR-data-c
Author = delta
Experiment = carbon.jxp
Sample_Id = S.K3-149-data-proton
Solvent = CHLOROFORM-D
Creation_Time = 11-SEP-2014 14:40:31
Revision_Time = 26-JAN-2015 16:19:31
Current_Time = 20-AUG-2015 13:54:21
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 400
Total_Scans = 400
Relaxation_Delay = 2.5[s]
Recvr_Gain = 60
Temp_Get = 24.4[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 4.1[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.587[dB]
Irr_Atn_Noe = 21.587[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2.5[s]
Repetition_Time = 3.33361792[s]

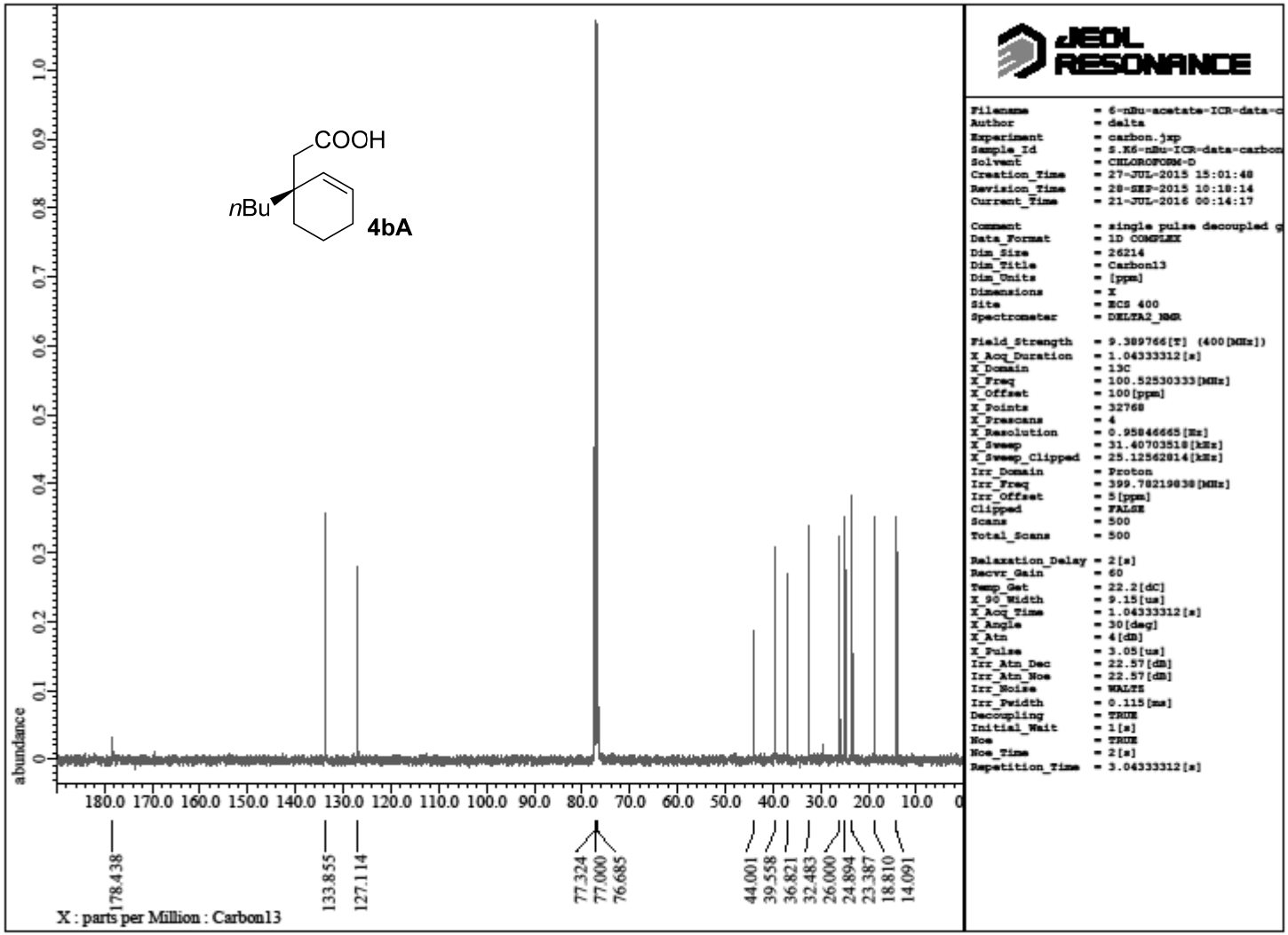

Et
COOH
Ph
4aB'
abundance
3.25
2.07
1.02
1.01
1.00
1.01
4.21
3.21
2.02
3.23
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.337
7.335
7.320
7.275
7.261
7.254
7.150
6.226
6.206
5.921
5.914
5.900
5.892
2.642
2.635
2.620
2.613
1.979
1.953
1.947
1.558
1.543
1.245
0.925
0.892
0.884
0.869
0.854
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-Ph-butyrate-ICR-minor-
Author = delta
Experiment = proton.jxp
Sample_Id = S.K4-52-minor
Solvent = CHLOROFORM-D
Creation_Time = 13-FEB-2015 17:00:58
Revision_Time = 25-JUN-2016 19:04:04
Current_Time = 21-JUL-2016 00:21:58
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 32
Total_Scans = 32
Relaxation_Delay = 2[s]
Recvr_Gain = 48
Temp_Get = 18.1[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 5.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 3.74587904[s]

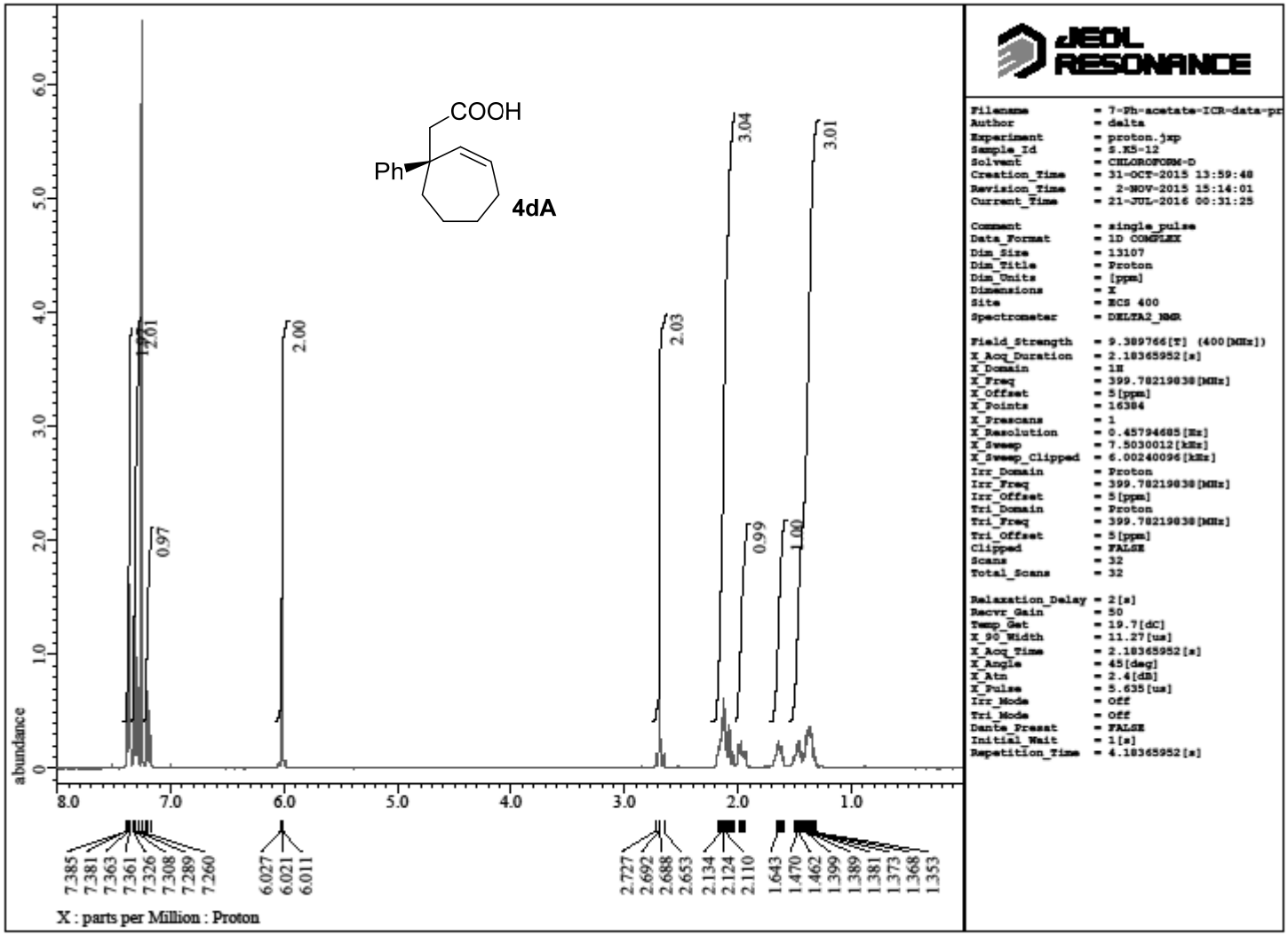

Et
COOH
Ph
4aB'
abundance
0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5 1.6 1.7 1.8 1.9 2.0
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
178.927
145.924
130.777
128.965
127.801
127.477
125.951
77.267
77.000
76.752
57.733
45.123
32.380
25.245
21.430
18.702
12.807
X : parts per Million : Carbon13
JEOL RESONANCE
Filename = 6-Ph-butyrate-ICR-minor-
Author = delta
Experiment = carbon.jxp
Sample_Id = S.K4-52-minor
Solvent = CHLOROFORM-D
Creation_Time = 13-FEB-2015 11:12:32
Revision_Time = 25-JUN-2016 19:20:12
Current_Time = 21-JUL-2016 00:21:33
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.41680896[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 16384
X_Prescans = 4
X_Resolution = 2.39918067[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 1500
Total_Scans = 1500
Relaxation_Delay = 1[s]
Recvr_Gain = 60
Temp_Get = 18.9[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 0.41680896[s]
X_Angle = 30[deg]
X_Atn = 4.1[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.587[dB]
Irr_Atn_Noe = 21.587[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 1[s]
Repetition_Time = 1.41680896[s]

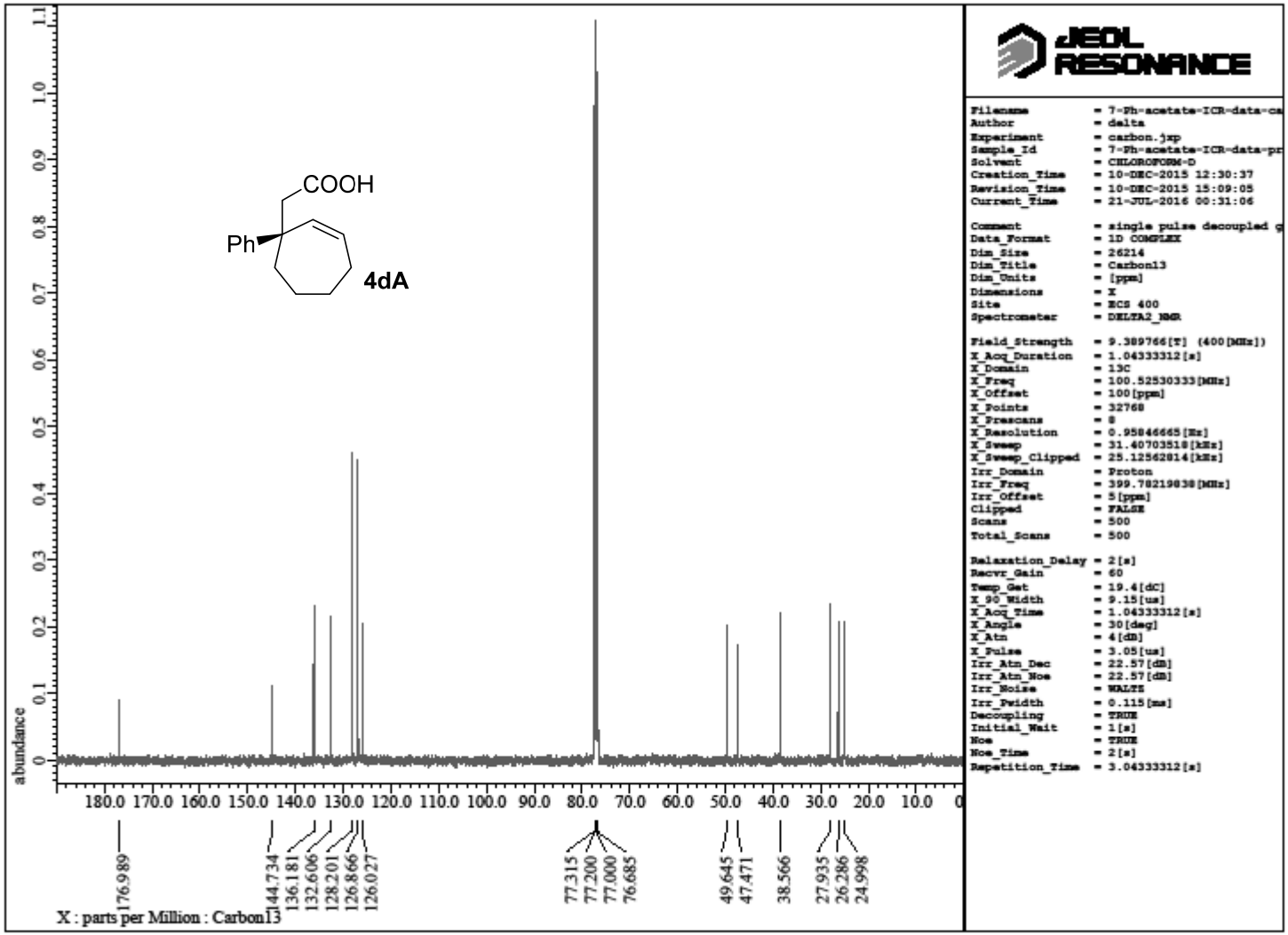

nC8H17
COOH
Ph
4aC
abundance
3.99
0.99
2.00
0.98
0.97
2.97
1.00
0.97
14.12
2.92
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.339
7.325
7.316
7.302
7.286
7.260
7.204
7.190
6.015
5.992
5.974
5.952
2.796
2.772
2.205
2.178
1.985
1.974
1.955
1.866
1.179
1.132
0.882
0.865
0.852
0.837
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-Ph-decanoate-ICR-data-
Author = delta
Experiment = proton.jxp
Sample_Id = 6-Ph-decanoate-ICR-data-
Solvent = CHLOROFORM-D
Creation_Time = 16-DEC-2015 17:35:55
Revision_Time = 22-DEC-2015 10:36:08
Current_Time = 21-JUL-2016 00:25:57
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 100
Total_Scans = 100
Relaxation_Delay = 2[s]
Recvr_Gain = 42
Temp_Get = 20.6[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 5.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 3.74587904[s]

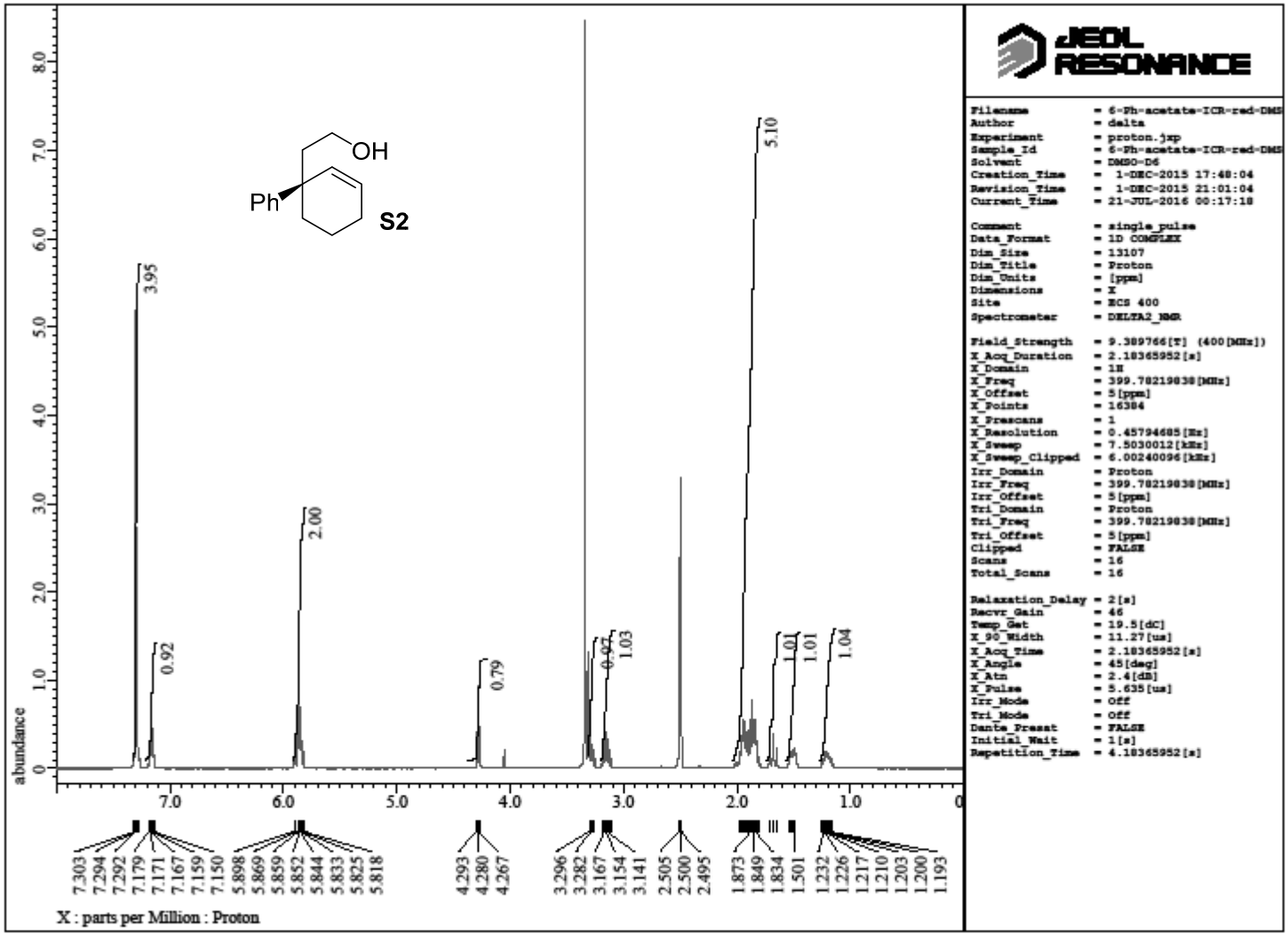

nC8H17
COOH
Ph
4aC
abundance
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
181.108
146.240
130.089
129.249
128.048
127.314
125.951
77.315
77.000
76.685
56.863
45.593
33.732
31.797
29.298
29.222
29.155
28.192
25.342
22.605
14.081
X : parts per Million : Carbon13
JEOL RESONANCE
Filename = 6-Ph-decanoate-ICR-data-
Author = delta
Experiment = carbon.jxp
Sample_Id = 6-Ph-decanoate-ICR-data-
Solvent = CHLOROFORM-D
Creation_Time = 25-DEC-2015 15:03:52
Revision_Time = 25-DEC-2015 19:43:02
Current_Time = 21-JUL-2016 00:26:21
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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 = 8
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 764
Total_Scans = 764
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.4[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

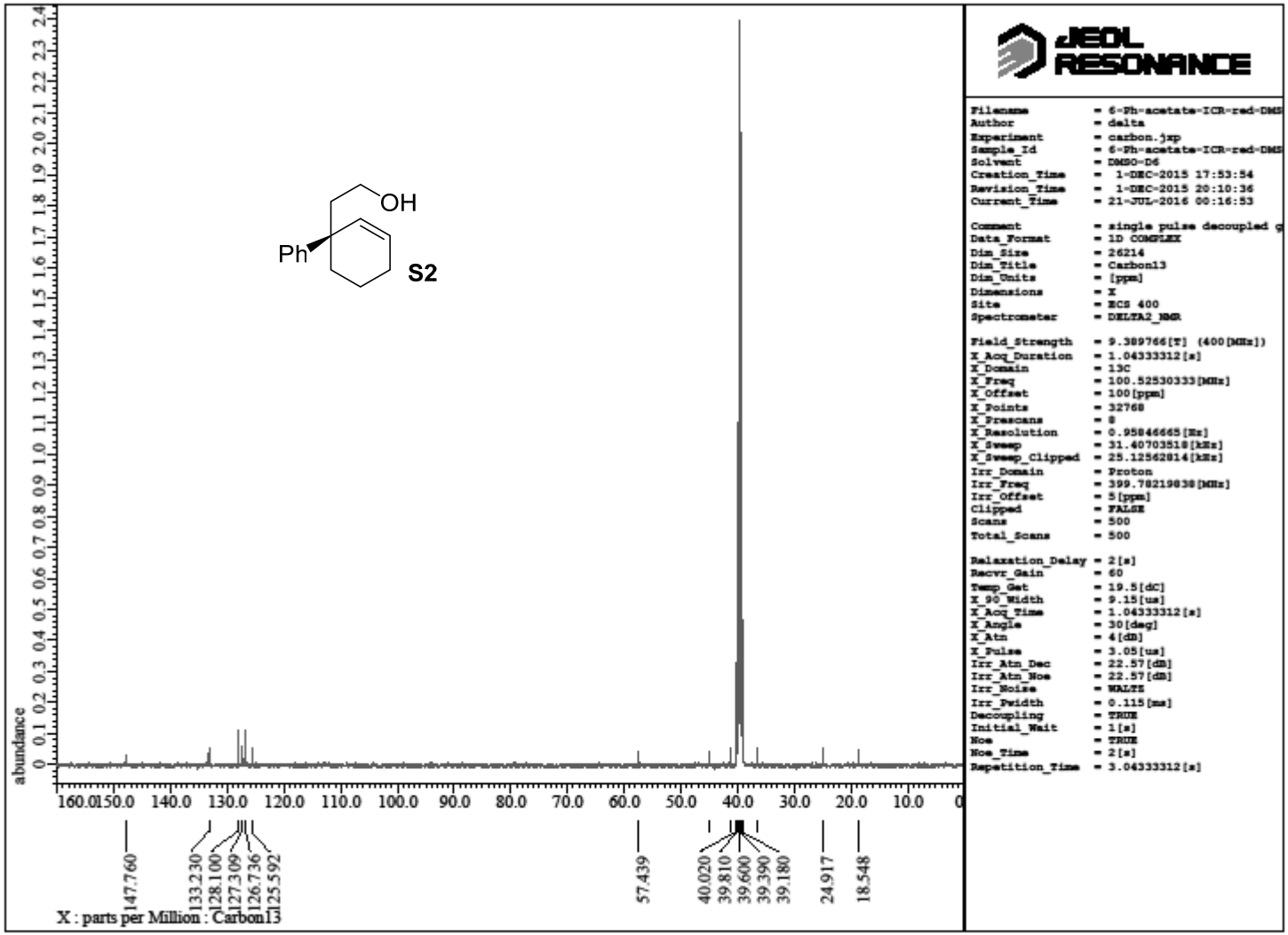

COOH
nBu
4bA
6.01
4.20
3.09
2.07
2.06
0.92
1.00
abundance
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.260
5.712
5.703
5.696
5.687
5.678
5.550
5.545
5.541
5.525
5.520
5.514
2.380
2.347
2.340
2.307
1.956
1.951
1.631
1.271
1.261
1.253
0.909
0.892
0.874
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-nBu-acetate-ICR-data-p
Author = delta
Experiment = proton.jxp
Sample_Id = S.K6-nBu-acetate-ICR-dat
Solvent = CHLOROFORM-D
Creation_Time = 28-SEP-2015 13:56:16
Revision_Time = 28-SEP-2015 17:30:13
Current_Time = 21-JUL-2016 00:14:41
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 100
Total_Scans = 100
Relaxation_Delay = 2[s]
Recvr_Gain = 42
Temp_Get = 20.2[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]

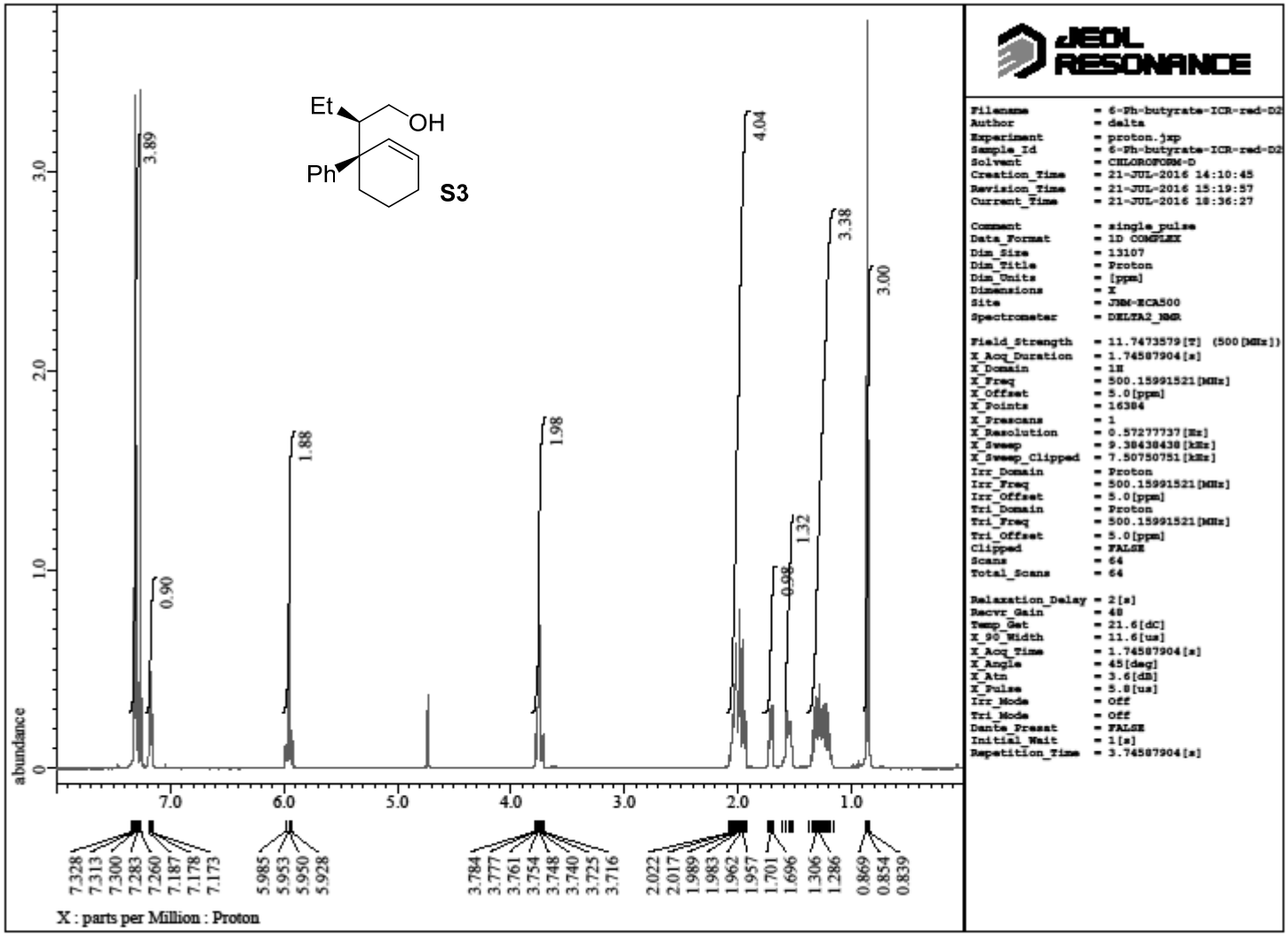

COOH
nBu
4bA
abundance
X : parts per Million : Carbon13
178.438
133.855
127.114
77.324
77.000
76.685
44.001
39.558
36.821
32.483
26.000
24.894
23.387
18.810
14.091
JEOL RESONANCE
Filename = 6-nBu-acetate-ICR-data-c
Author = delta
Experiment = carbon.jxp
Sample_Id = S.K6-nBu-ICR-data-carbon
Solvent = CHLOROFORM-D
Creation_Time = 27-JUL-2015 15:01:48
Revision_Time = 28-SEP-2015 10:18:14
Current_Time = 21-JUL-2016 00:14:17
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 500
Total_Scans = 500
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 22.2[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

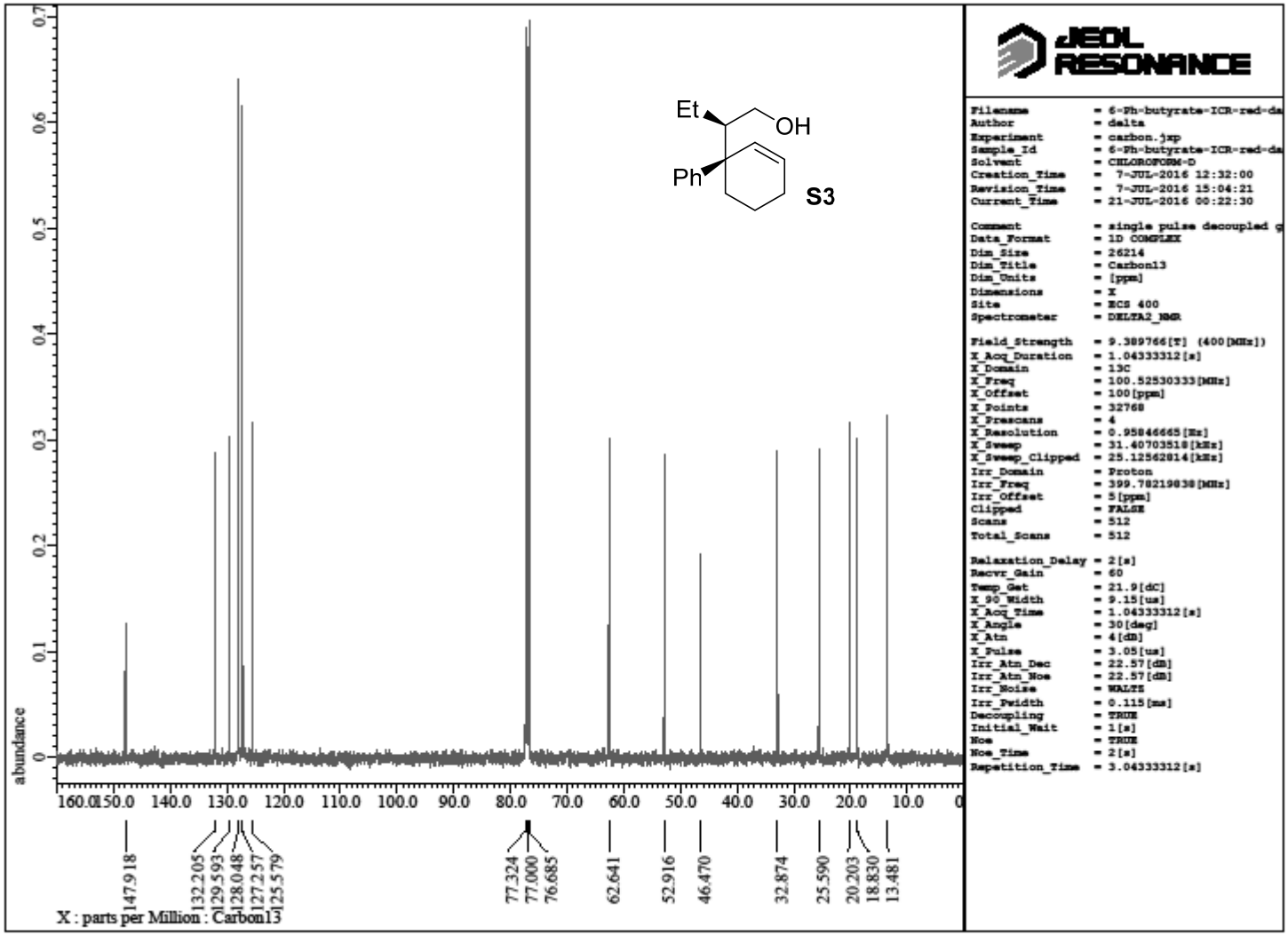

COOH
Ph
4dA
3.04
3.01
2.03
2.00
2.01
0.99
1.00
0.97
abundance
X : parts per Million : Proton
7.385
7.381
7.363
7.361
7.326
7.308
7.289
7.260
6.027
6.021
6.011
2.727
2.692
2.688
2.653
2.134
2.124
2.110
1.643
1.470
1.462
1.399
1.389
1.381
1.373
1.368
1.353
JEOL RESONANCE
Filename = 7-Ph-acetate-ICR-data-pr
Author = delta
Experiment = proton.jxp
Sample_Id = S.K5-12
Solvent = CHLOROFORM-D
Creation_Time = 31-OCT-2015 13:59:48
Revision_Time = 2-NOV-2015 15:14:01
Current_Time = 21-JUL-2016 00:31:25
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 32
Total_Scans = 32
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 19.7[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]

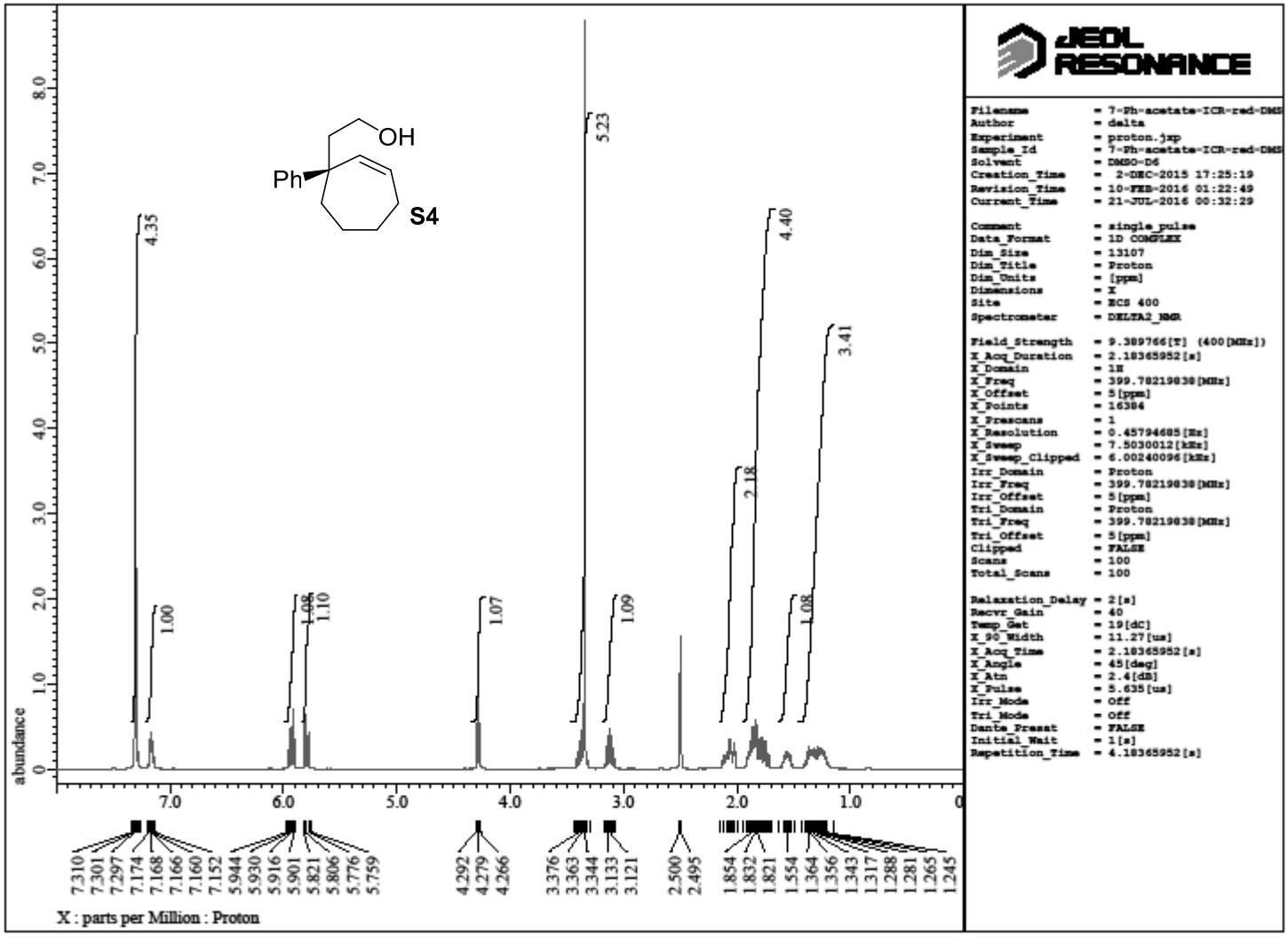

COOH
Ph
4dA
abundance
X : parts per Million : Carbon13
176.989
144.734
136.181
132.606
128.201
126.866
126.027
77.315
77.200
77.000
76.685
49.645
47.471
38.566
27.935
26.286
24.998
JEOL RESONANCE
Filename = 7-Ph-acetate-ICR-data-ca
Author = delta
Experiment = carbon.jxp
Sample_Id = 7-Ph-acetate-ICR-data-pr
Solvent = CHLOROFORM-D
Creation_Time = 10-DEC-2015 12:30:37
Revision_Time = 10-DEC-2015 15:09:05
Current_Time = 21-JUL-2016 00:31:06
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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 = 8
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 500
Total_Scans = 500
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.4[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

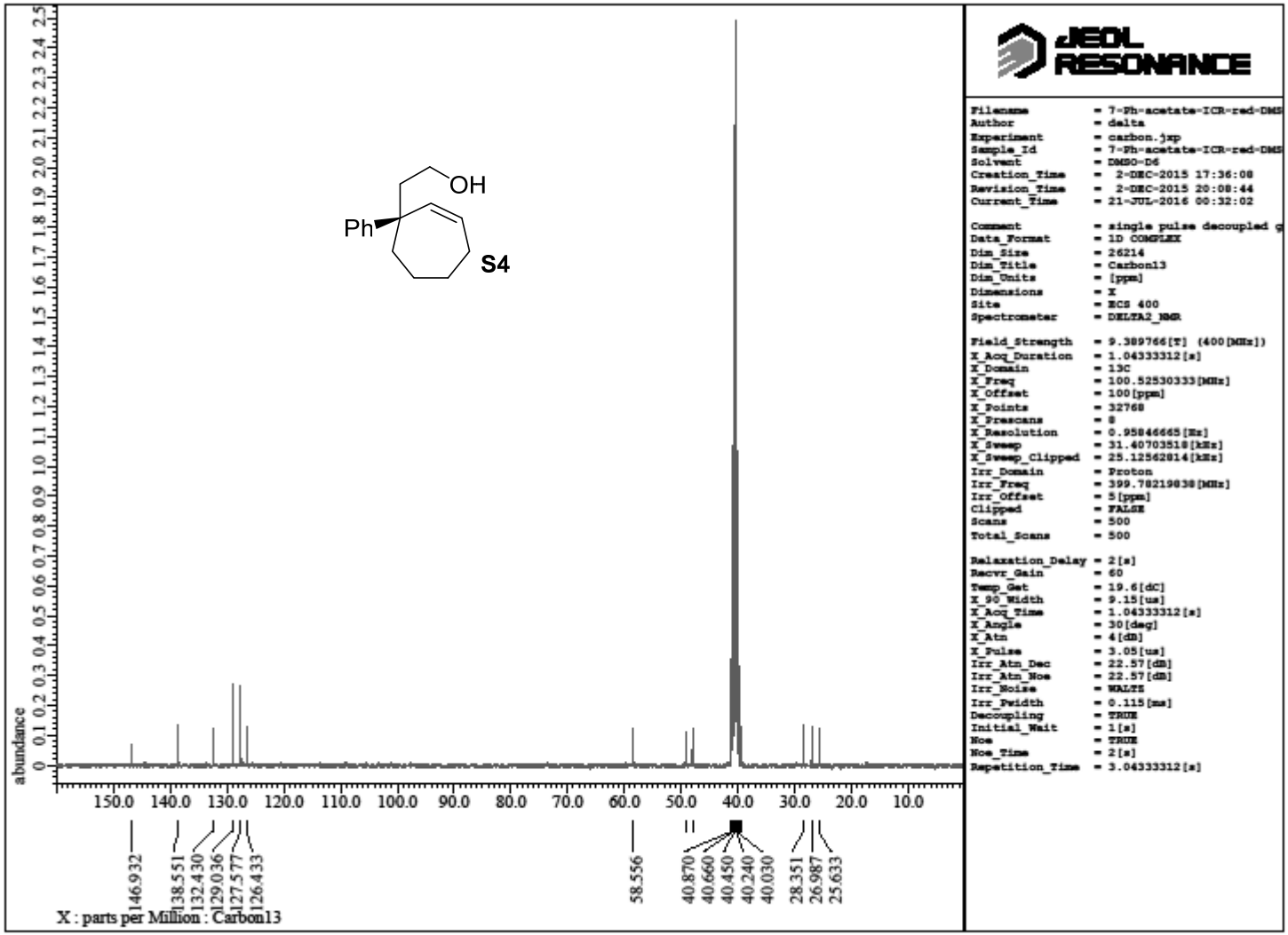

OH
Ph
S2
abundance
3.95
0.92
2.00
0.79
0.97
1.03
5.10
1.01
1.01
1.04
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.303
7.294
7.292
7.179
7.171
7.167
7.159
7.150
5.898
5.869
5.859
5.852
5.844
5.833
5.825
5.818
4.293
4.280
4.267
3.296
3.282
3.167
3.154
3.141
2.505
2.500
2.495
1.873
1.849
1.834
1.501
1.232
1.226
1.217
1.210
1.203
1.200
1.193
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-Ph-acetate-ICR-red-DMS
Author = delta
Experiment = proton.jxp
Sample_Id = 6-Ph-acetate-ICR-red-DMS
Solvent = DMSO-D6
Creation_Time = 1-DEC-2015 17:48:04
Revision_Time = 1-DEC-2015 21:01:04
Current_Time = 21-JUL-2016 00:17:18
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 2[s]
Recvr_Gain = 46
Temp_Get = 19.5[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.6[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]


OH
Ph
S2
abundance
X : parts per Million : Carbon13
147.760
133.230
128.100
127.309
126.736
125.592
57.439
40.020
39.810
39.600
39.390
39.180
24.917
18.548
JEOL RESONANCE
Filename = 6-Ph-acetate-ICR-red-DMS
Author = delta
Experiment = carbon.jxp
Sample_Id = 6-Ph-acetate-ICR-red-DMS
Solvent = DMSO-D6
Creation_Time = 1-DEC-2015 17:53:54
Revision_Time = 1-DEC-2015 20:10:36
Current_Time = 21-JUL-2016 00:16:53
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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 = 8
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 500
Total_Scans = 500
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.5[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]


Et
OH
Ph
S3
3.89
0.90
1.88
1.98
4.04
0.98
1.32
3.38
3.00
abundance
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.328
7.313
7.300
7.283
7.260
7.187
7.178
7.173
5.985
5.953
5.950
5.928
3.784
3.777
3.761
3.754
3.748
3.740
3.725
3.716
2.022
2.017
1.989
1.983
1.962
1.957
1.701
1.696
1.306
1.286
0.869
0.854
0.839
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-Ph-butyrate-ICR-rad-D2
Author = delta
Experiment = proton.jxp
Sample_Id = 6-Ph-butyrate-ICR-rad-D2
Solvent = CHLOROFORM-D
Creation_Time = 21-JUL-2016 14:10:45
Revision_Time = 21-JUL-2016 15:19:57
Current_Time = 21-JUL-2016 18:36:27
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 64
Total_Scans = 64
Relaxation_Delay = 2[s]
Recvr_Gain = 48
Temp_Get = 21.6[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 5.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 3.74587904[s]

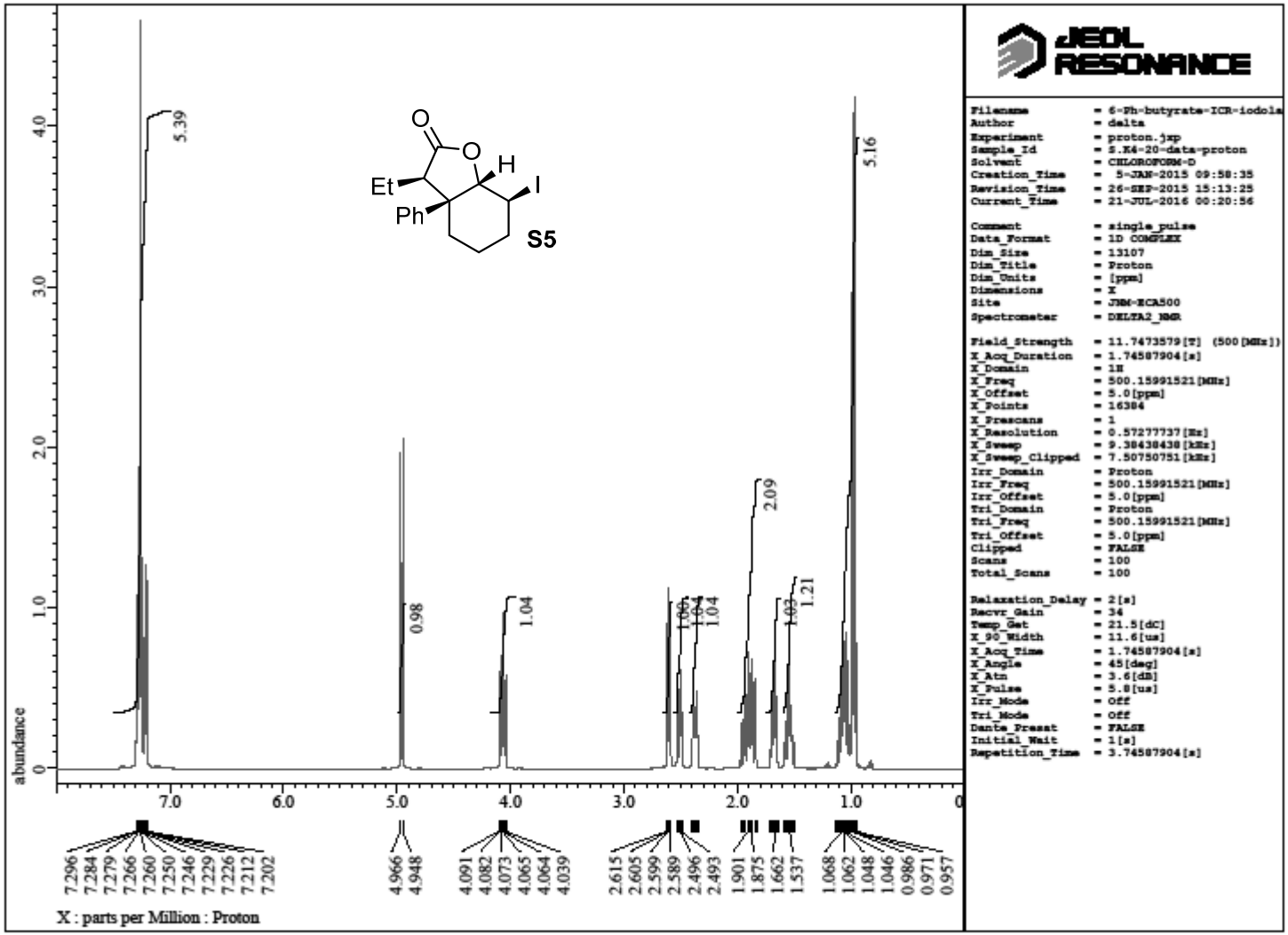

Et
OH
Ph
S3
abundance
X : parts per Million : Carbon13
147.918
132.205
129.593
128.048
127.257
125.579
77.324
77.000
76.685
62.641
52.916
46.470
32.874
25.590
20.203
18.830
13.481
Filename = 6-Ph-butyrate-ICR-rad-da
Author = delta
Experiment = carbon.jxp
Sample_Id = 6-Ph-butyrate-ICR-rad-da
Solvent = CHLOROFORM-D
Creation_Time = 7-JUL-2016 12:32:00
Revision_Time = 7-JUL-2016 15:04:21
Current_Time = 21-JUL-2016 00:22:30
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 512
Total_Scans = 512
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 21.9[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

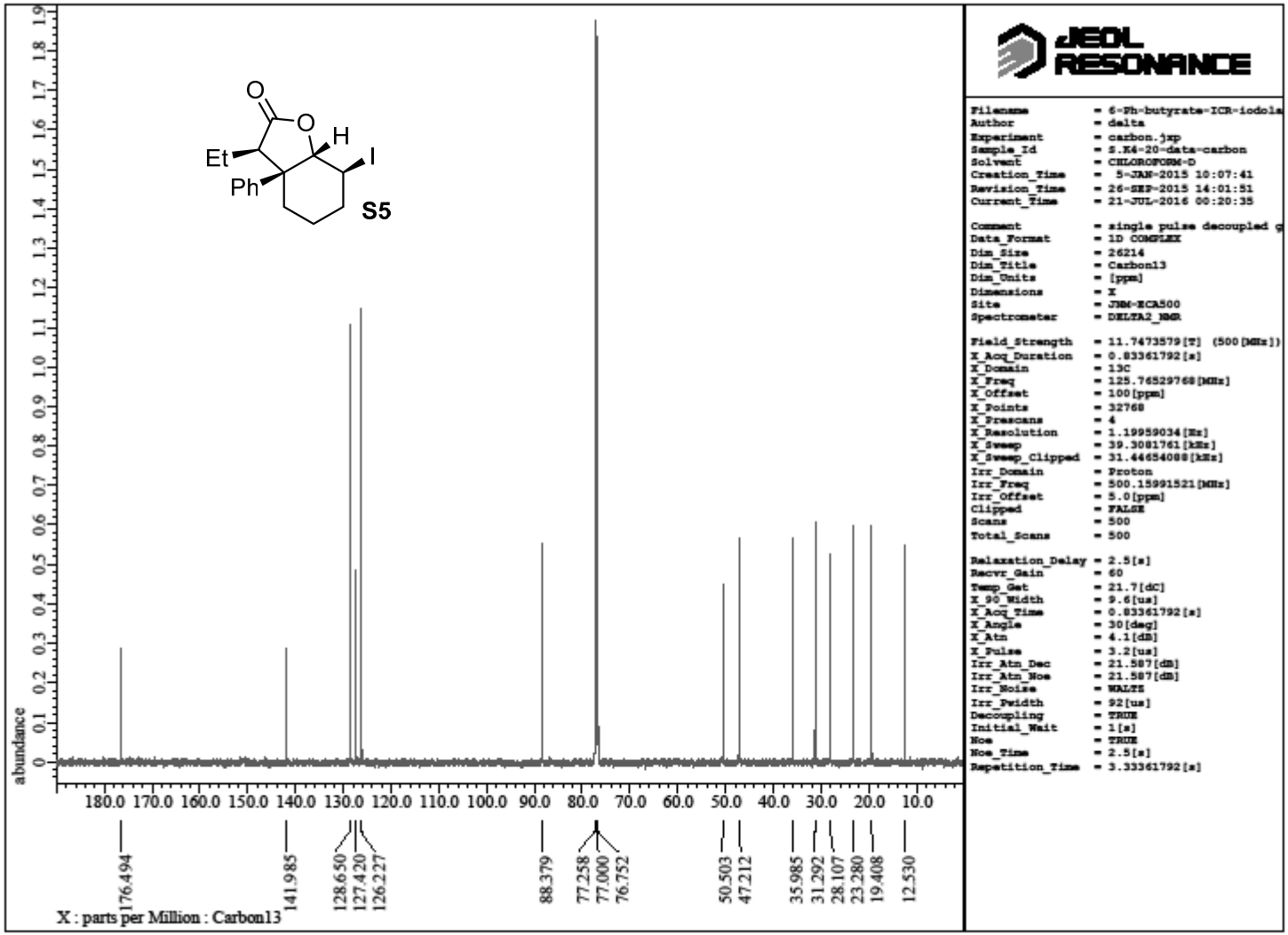

OH
Ph
S4
4.35
1.00
1.08
1.10
1.07
5.23
1.09
2.18
4.40
1.08
3.41
abundance
X : parts per Million : Proton
7.310
7.301
7.297
7.174
7.168
7.166
7.160
7.152
5.944
5.930
5.916
5.901
5.821
5.806
5.776
5.759
4.292
4.279
4.266
3.376
3.363
3.344
3.133
3.121
2.500
2.495
1.854
1.832
1.821
1.554
1.364
1.356
1.343
1.317
1.288
1.281
1.265
1.245
JEOL RESONANCE
Filename = 7-Ph-acetate-ICR-red-DMS
Author = delta
Experiment = proton.jxp
Sample_Id = 7-Ph-acetate-ICR-red-DMS
Solvent = DMSO-D6
Creation_Time = 2-DEC-2015 17:25:19
Revision_Time = 10-FEB-2016 01:22:49
Current_Time = 21-JUL-2016 00:32:29
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 100
Total_Scans = 100
Relaxation_Delay = 2[s]
Recvr_Gain = 40
Temp_Get = 19[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Preset = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]

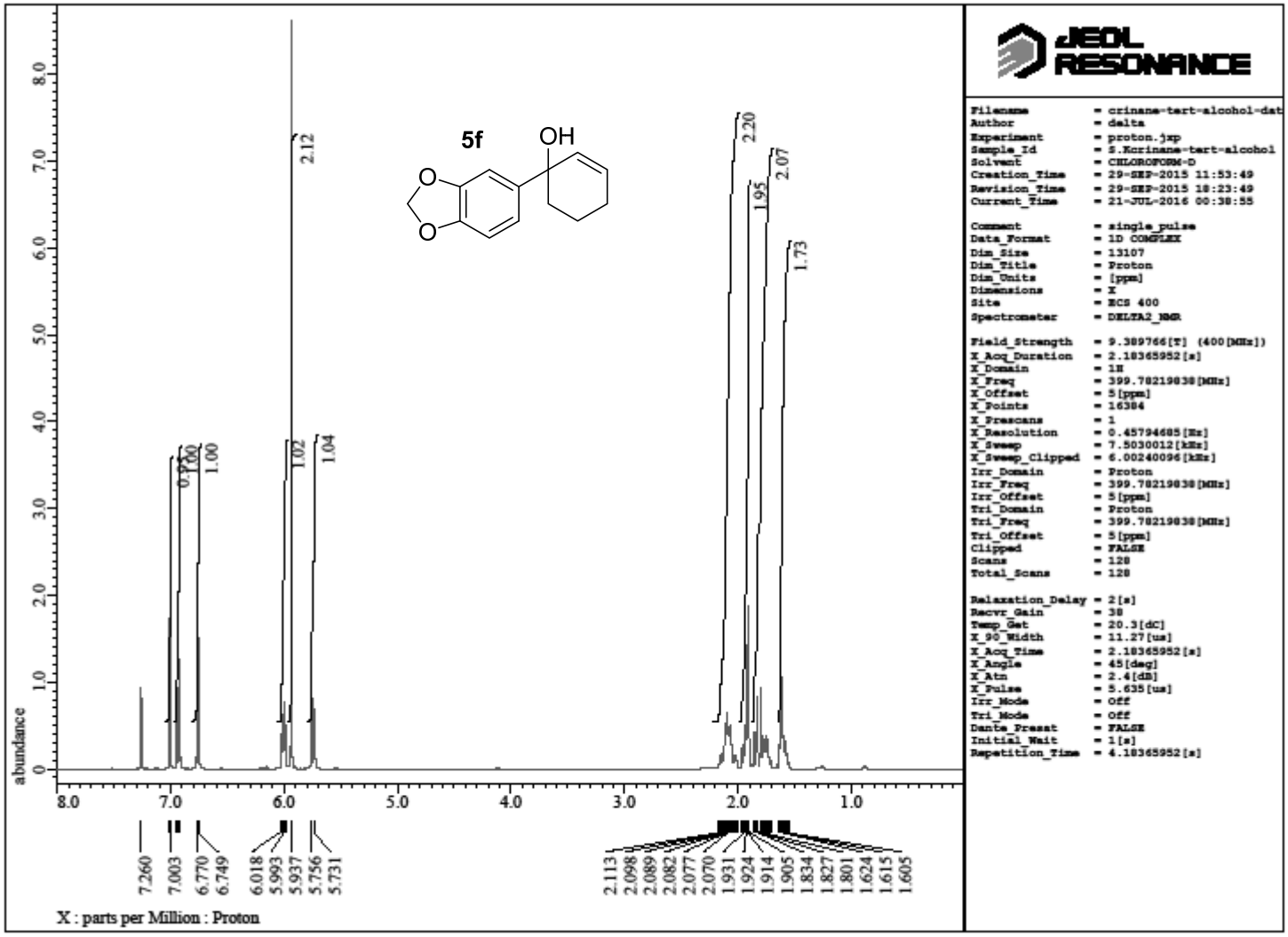


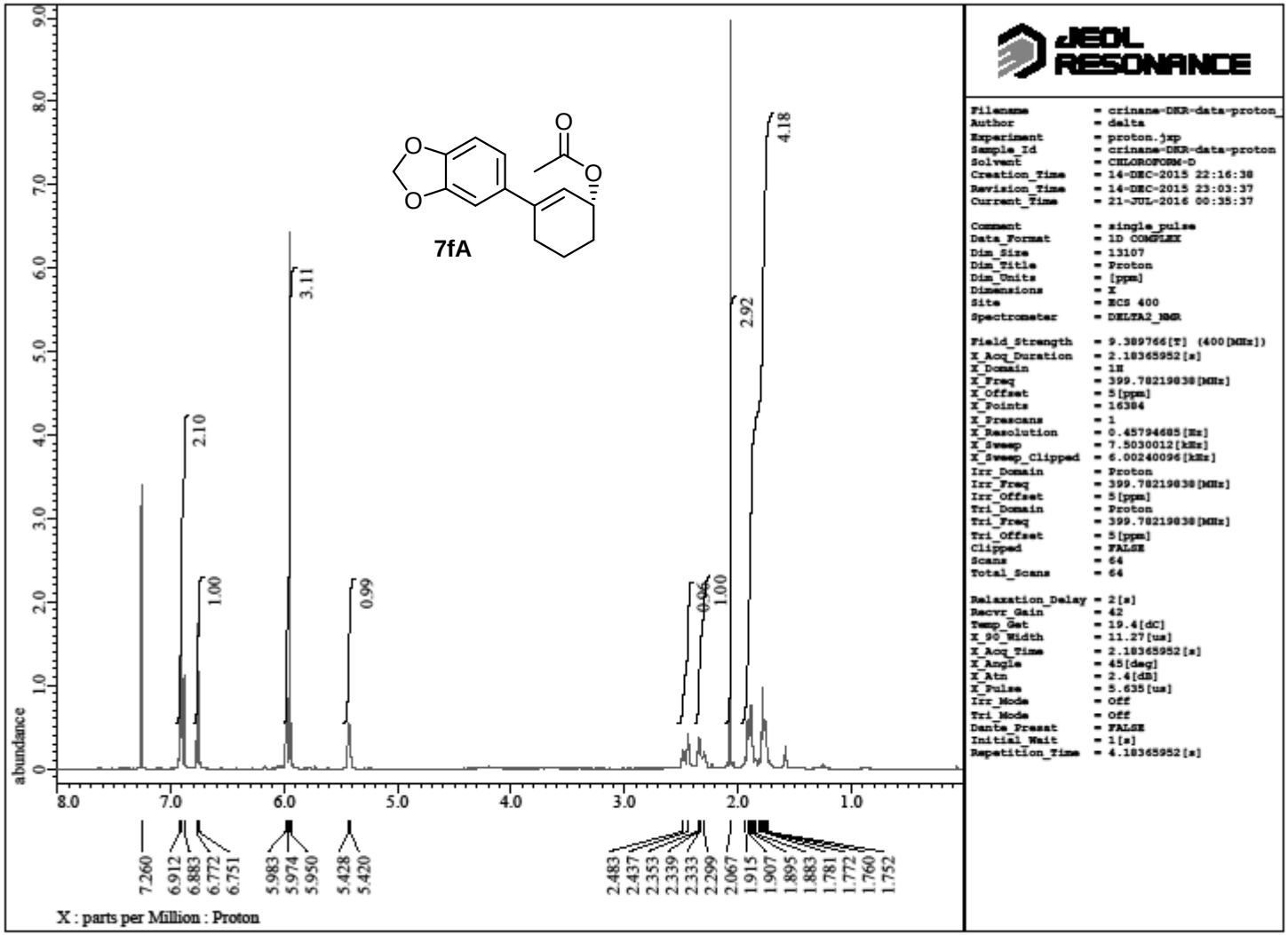
JEOL RESONANCE

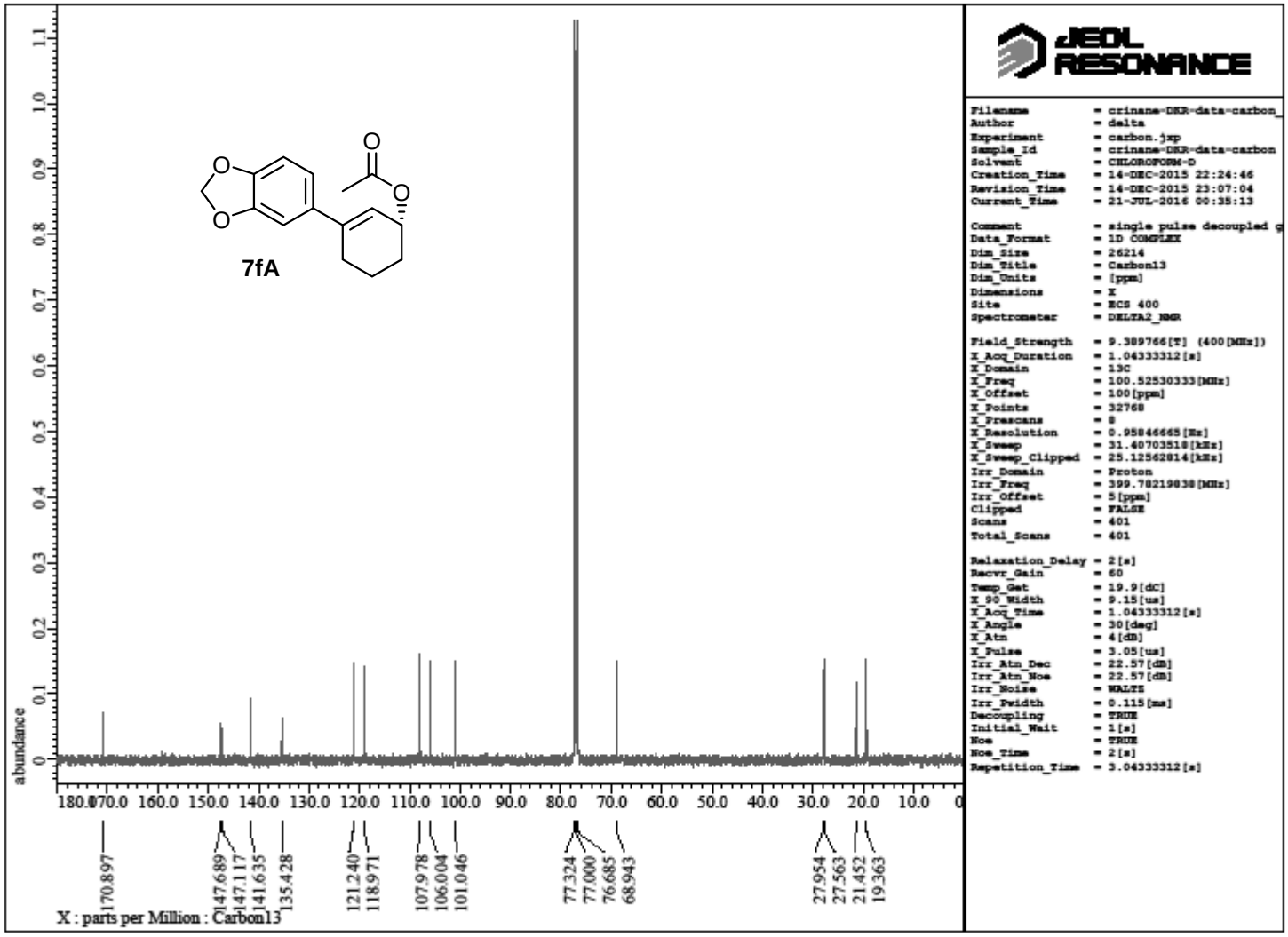
Filename	= 7-Ph-acetate-ICR-red-DMS
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= 7-Ph-acetate-ICR-red-DMS
Solvent	= DMSO-D6
Creation_Time	= 2-DEC-2015 17:36:08
Revision_Time	= 2-DEC-2015 20:08:44
Current_Time	= 21-JUL-2016 00:32:02
Comment	= single pulse decoupled g
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
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	= 8
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 500
Total_Scans	= 500
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 19.6[dC]
X_90_Width	= 9.15[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 4[dB]
X_Pulse	= 3.05[us]
Irr_Atn_Dec	= 22.57[dB]
Irr_Atn_Noe	= 22.57[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]

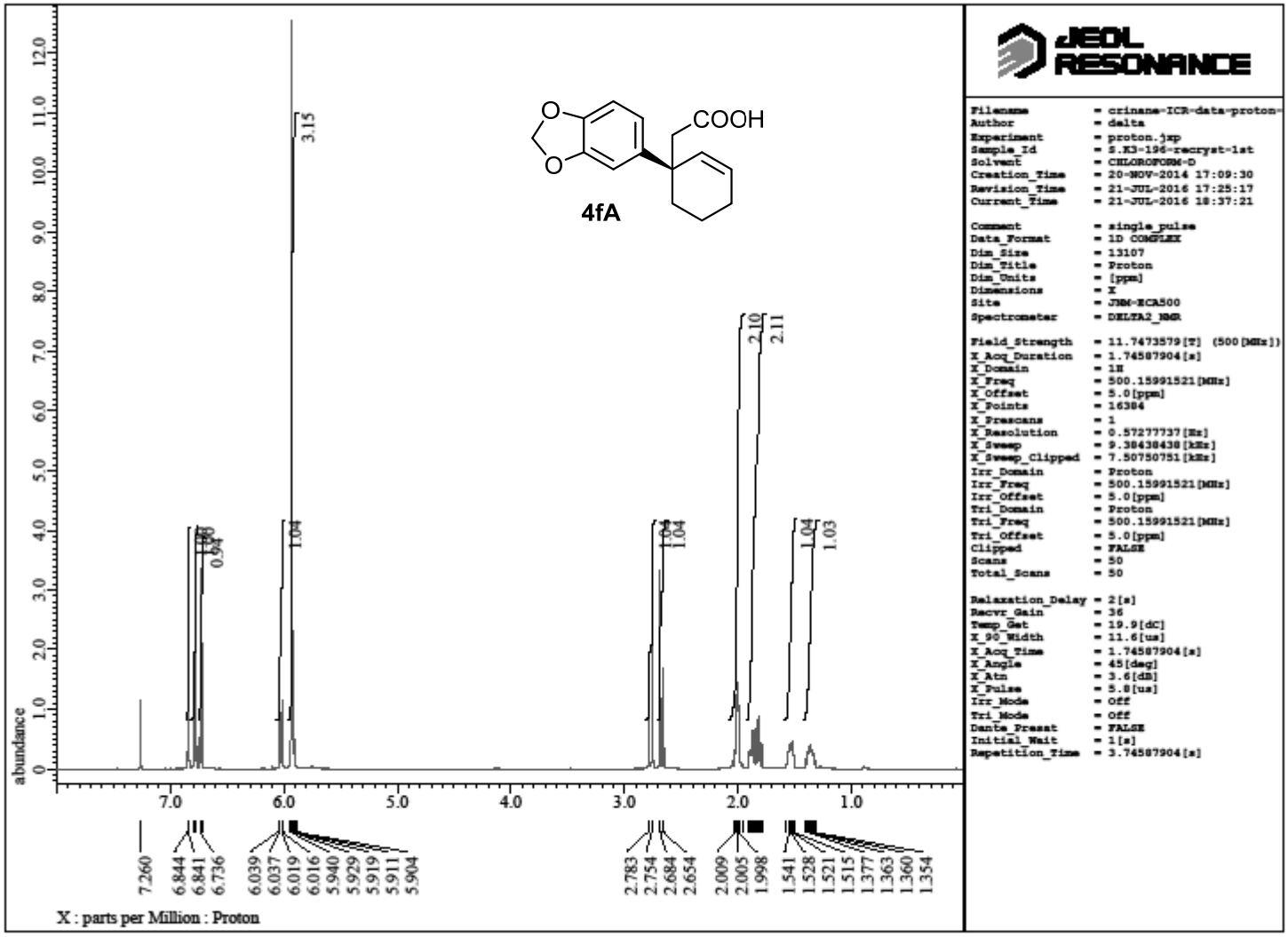

O
O
H
I
Et
Ph
S5
5.39
5.16
2.09
1.21
1.04
0.98
abundance
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.296
7.284
7.279
7.266
7.260
7.250
7.246
7.229
7.226
7.212
7.202
4.966
4.948
4.091
4.082
4.073
4.065
4.064
4.039
2.615
2.605
2.599
2.589
2.496
2.493
1.901
1.875
1.662
1.537
1.068
1.062
1.048
1.046
0.986
0.971
0.957
X : parts per Million : Proton
JEOL RESONANCE
Filename = 6-Ph-butyrate-ICR-iodola
Author = delta
Experiment = proton.jxp
Sample_Id = S.K4-20-data-proton
Solvent = CHLOROFORM-D
Creation_Time = 5-JAN-2015 09:58:35
Revision_Time = 26-SEP-2015 15:13:25
Current_Time = 21-JUL-2016 00:20:56
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 100
Total_Scans = 100
Relaxation_Delay = 2[s]
Recvr_Gain = 34
Temp_Get = 21.5[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 5.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 3.74587904[s]

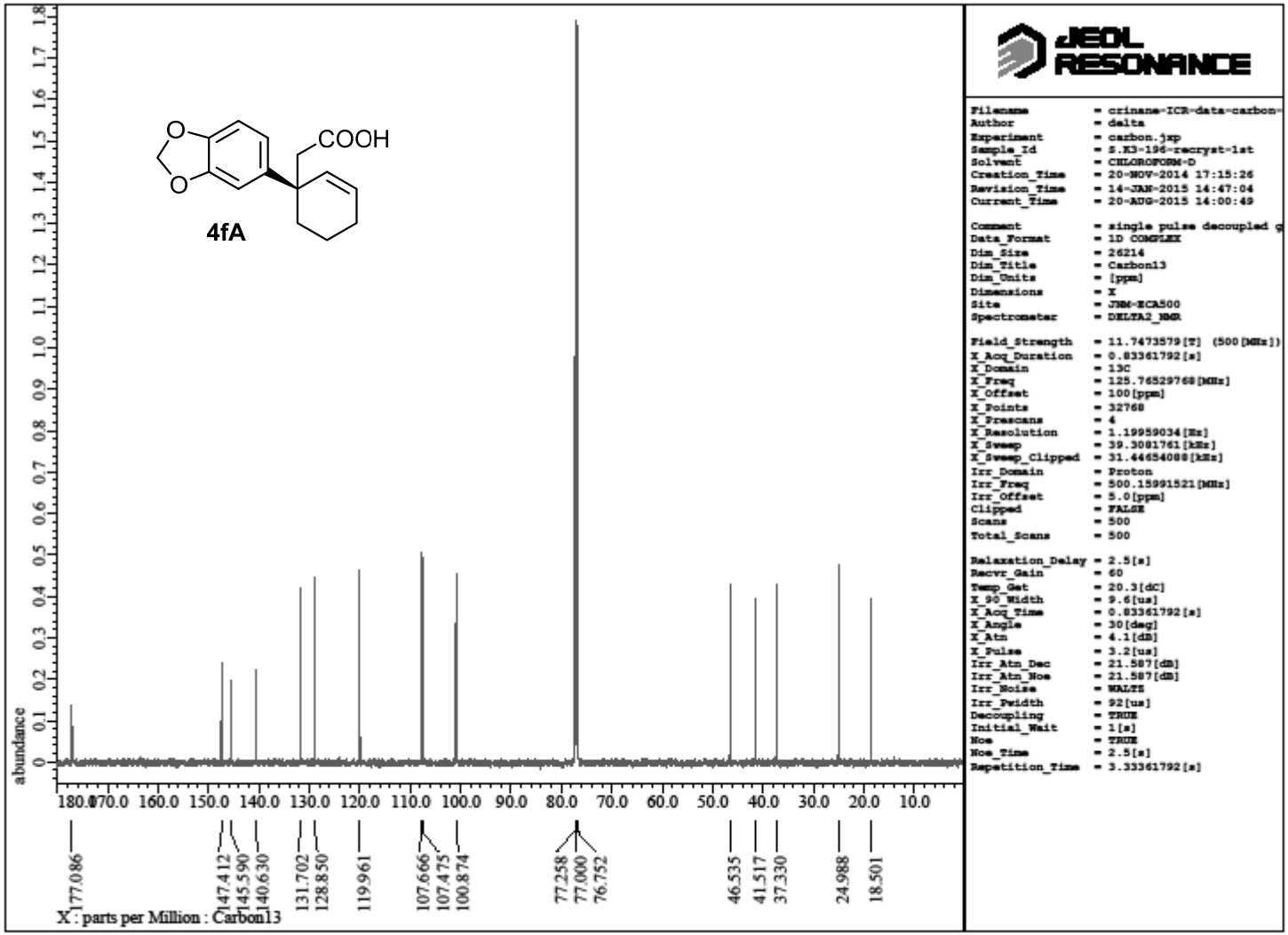

O
O
H
I
Et
Ph
S5
abundance
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
176.494
141.985
128.650
127.420
126.227
88.379
77.258
77.000
76.752
50.503
47.212
35.985
31.292
28.107
23.280
19.408
12.530
X : parts per Million : Carbon13
JEOL RESONANCE
Filename = 6-Ph-butyrate-ICR-iodola
Author = delta
Experiment = carbon.jxp
Sample_Id = S.K4-20-data-carbon
Solvent = CHLOROFORM-D
Creation_Time = 5-JAN-2015 10:07:41
Revision_Time = 26-SEP-2015 14:01:51
Current_Time = 21-JUL-2016 00:20:35
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 500
Total_Scans = 500
Relaxation_Delay = 2.5[s]
Recvr_Gain = 60
Temp_Get = 21.7[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 4.1[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.587[dB]
Irr_Atn_Noe = 21.587[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2.5[s]
Repetition_Time = 3.33361792[s]


5f
OH
O
O
2.12
2.20
2.07
1.95
1.73
1.04
1.02
1.00
abundance
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.260
7.003
6.770
6.749
6.018
5.993
5.937
5.756
5.731
2.113
2.098
2.089
2.082
2.077
2.070
1.931
1.924
1.914
1.905
1.834
1.827
1.801
1.624
1.615
1.605
X : parts per Million : Proton
JEOL RESONANCE
Filename = crinane-tert-alcohol-dat
Author = delta
Experiment = proton.jxp
Sample_Id = S.Ecrinane-tert-alcohol
Solvent = CHLOROFORM-D
Creation_Time = 29-SEP-2015 11:53:49
Revision_Time = 29-SEP-2015 18:23:49
Current_Time = 21-JUL-2016 00:38:55
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 128
Total_Scans = 128
Relaxation_Delay = 2[s]
Recvr_Gain = 38
Temp_Get = 20.3[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.6[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]


7fA
abundance
X : parts per Million : Proton
JEOL RESONANCE
Filename = crinane-DKR-data-proton_
Author = delta
Experiment = proton.jxp
Sample_Id = crinane-DKR-data-proton
Solvent = CHLOROFORM-D
Creation_Time = 14-DEC-2015 22:16:38
Revision_Time = 14-DEC-2015 23:03:37
Current_Time = 21-JUL-2016 00:35:37
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 64
Total_Scans = 64
Relaxation_Delay = 2[s]
Recvr_Gain = 42
Temp_Get = 19.4[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]

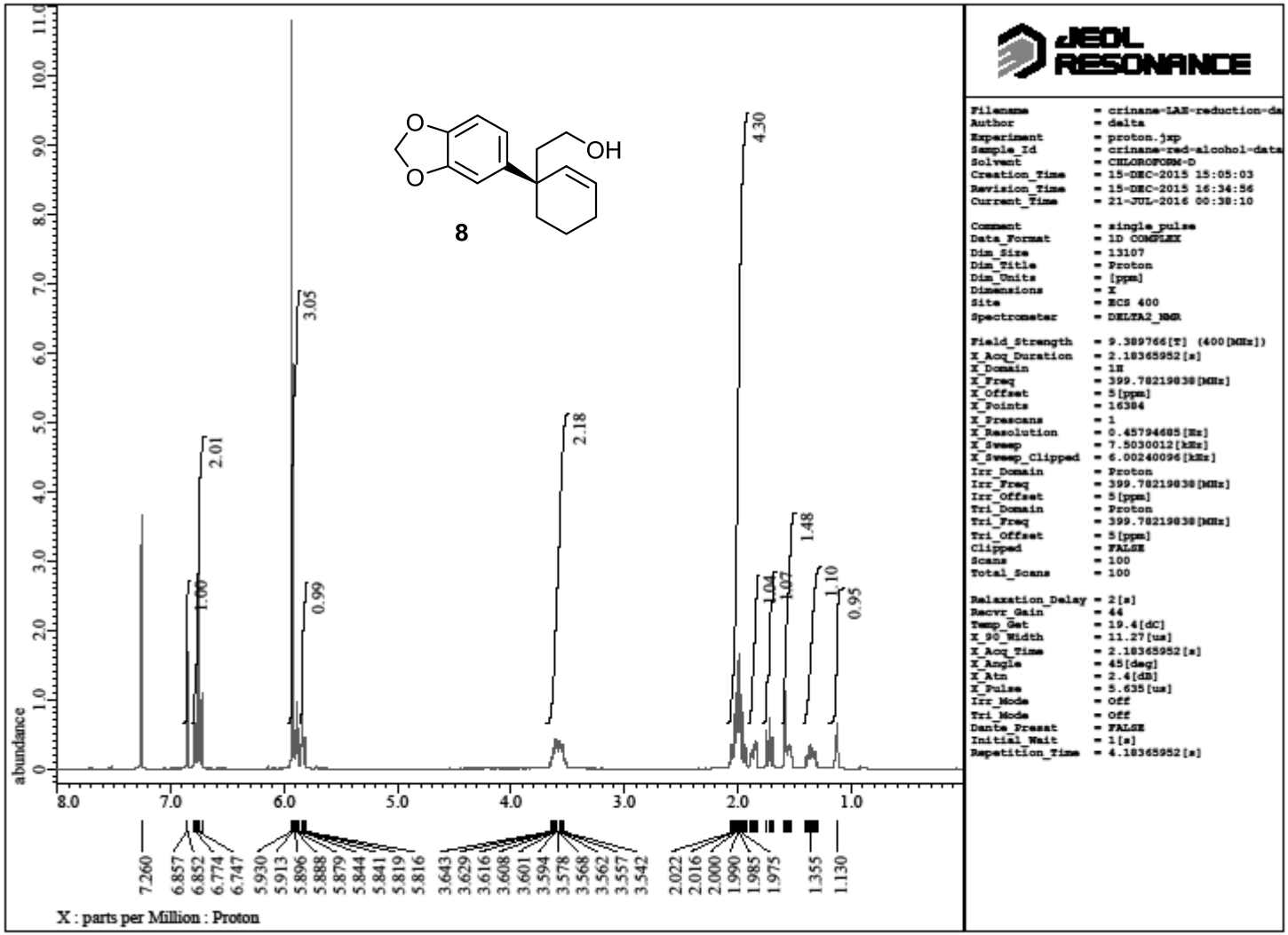

7fA
abundance
X : parts per Million : Carbon13
170.897
147.689
147.117
141.635
135.428
121.240
118.971
107.978
106.004
101.046
77.324
77.000
76.685
68.943
27.954
27.563
21.452
19.363
JEOL RESONANCE
Filename = crinane-DKR-data-carbon_
Author = delta
Experiment = carbon.jxp
Sample_Id = crinane-DKR-data-carbon
Solvent = CHLOROFORM-D
Creation_Time = 14-DEC-2015 22:24:46
Revision_Time = 14-DEC-2015 23:07:04
Current_Time = 21-JUL-2016 00:35:13
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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 = 8
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 401
Total_Scans = 401
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.9[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

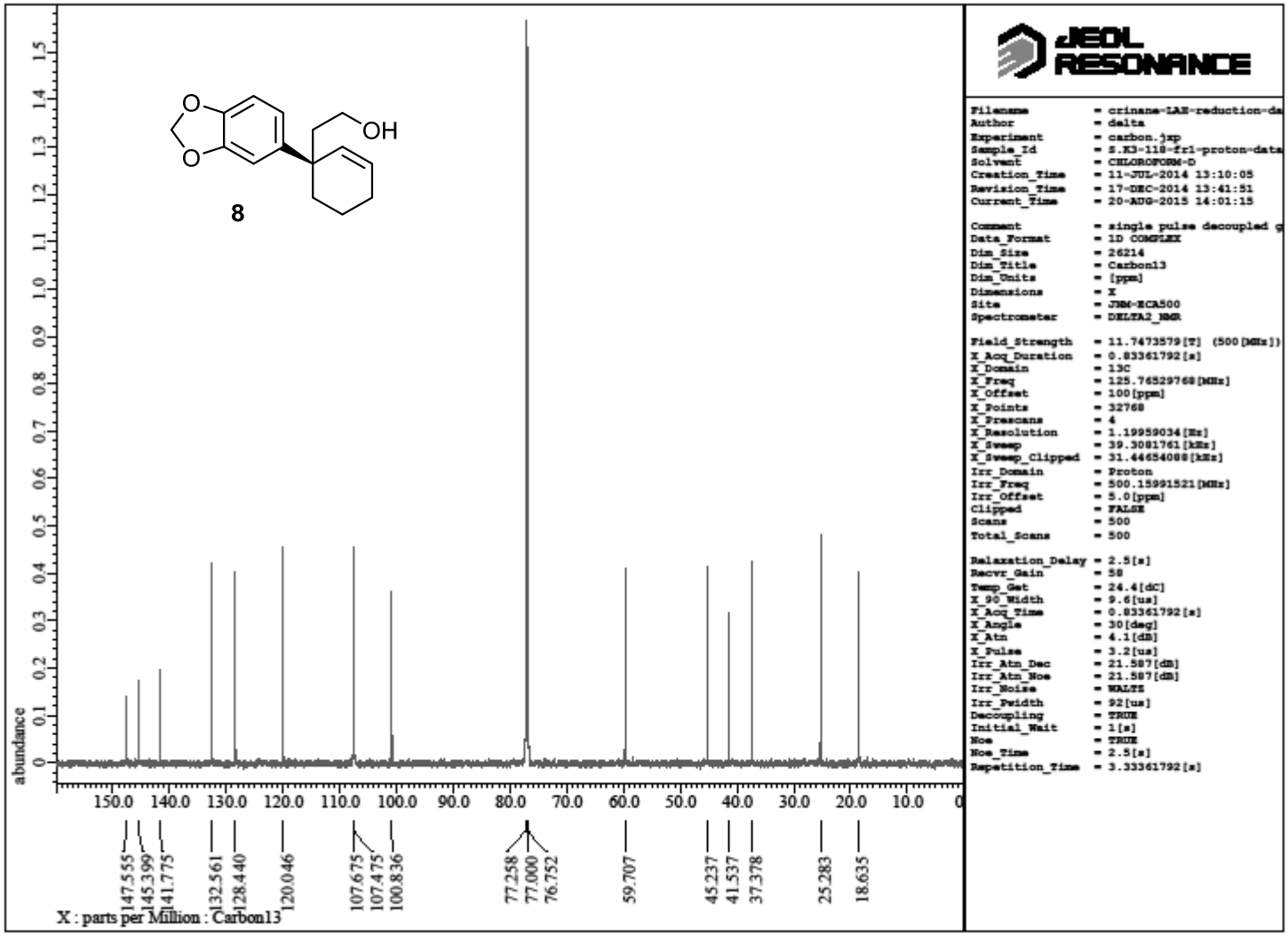

COOH
4fA
3.15
1.04
0.94
2.10
2.11
1.04
1.04
1.04
1.03
abundance
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.260
6.844
6.841
6.736
6.039
6.037
6.019
6.016
5.940
5.929
5.919
5.911
5.904
2.783
2.754
2.684
2.654
2.009
2.005
1.998
1.541
1.528
1.521
1.515
1.377
1.363
1.360
1.354
X : parts per Million : Proton
JEOL RESONANCE
Filename = crinane-ICR-data-proton-
Author = delta
Experiment = proton.jxp
Sample_Id = S.K3-196-recryst-1st
Solvent = CHLOROFORM-D
Creation_Time = 20-NOV-2014 17:09:30
Revision_Time = 21-JUL-2016 17:25:17
Current_Time = 21-JUL-2016 18:37:21
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 50
Total_Scans = 50
Relaxation_Delay = 2[s]
Recvr_Gain = 36
Temp_Get = 19.9[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 5.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 3.74587904[s]

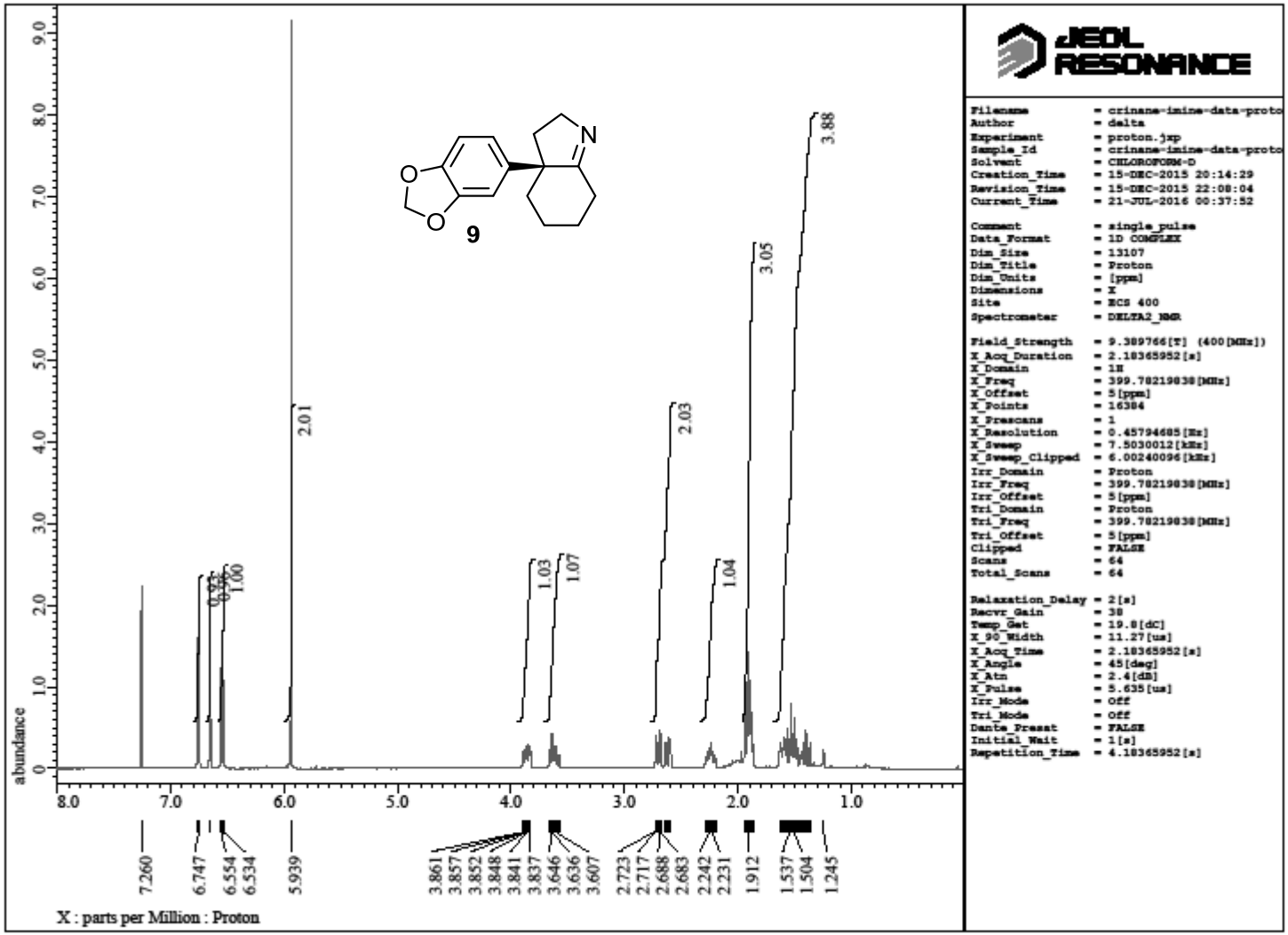


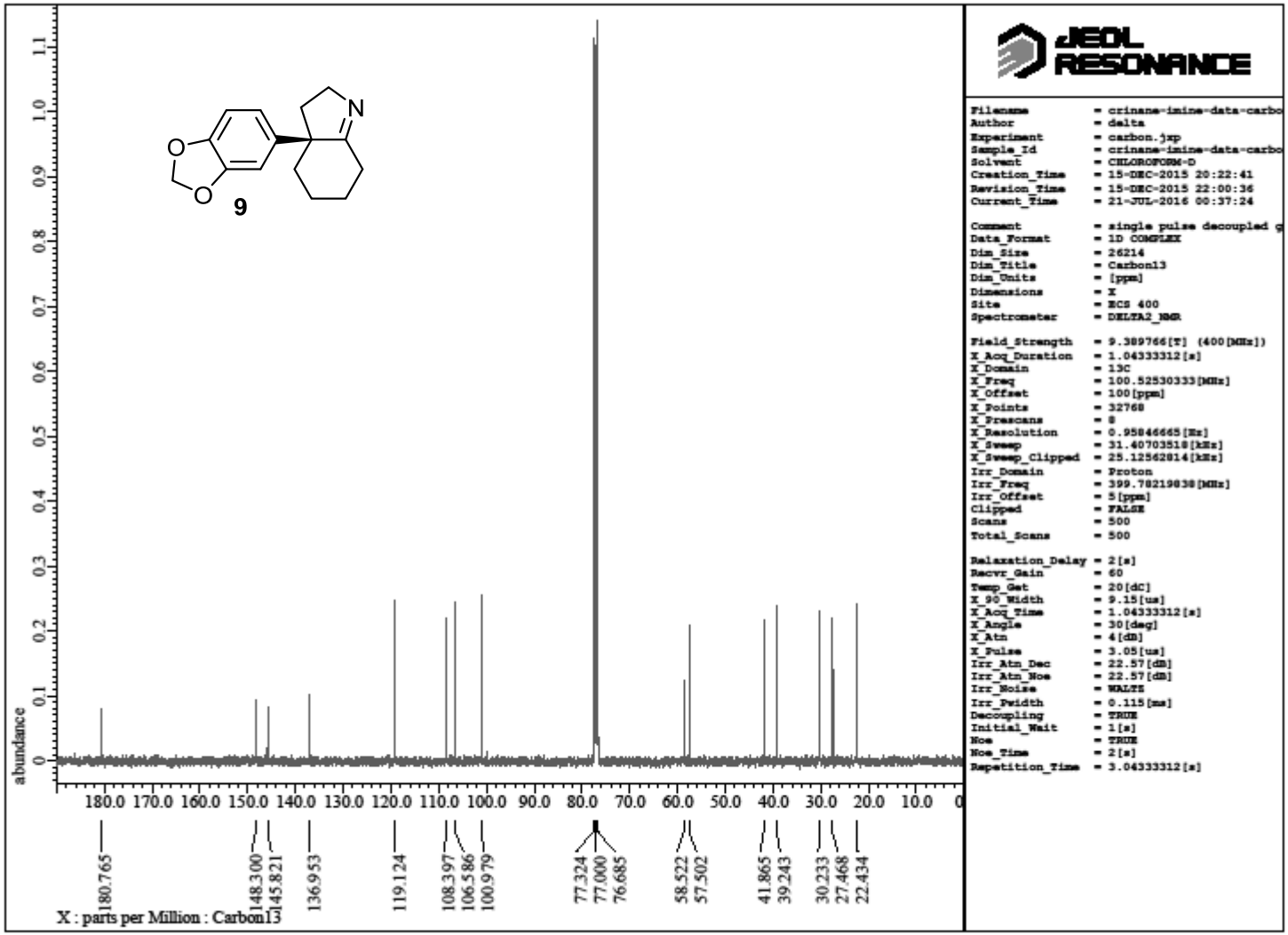
JEOL RESONANCE

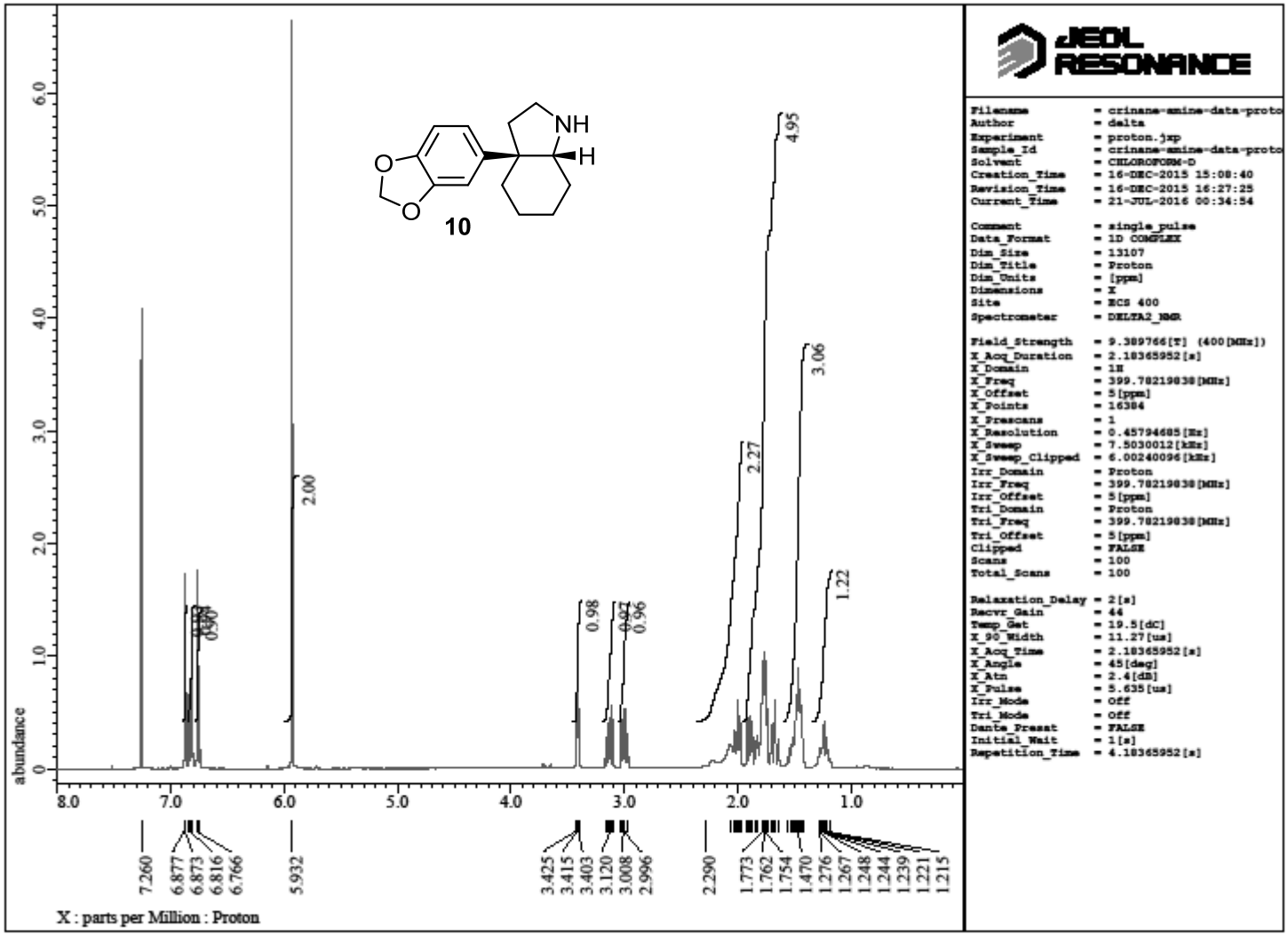
Filename	= crinane-ICR-data-carbon-
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= S.K3-196-recryst-1st
Solvent	= CHLOROFORM-D
Creation_Time	= 20-NOV-2014 17:15:26
Revision_Time	= 14-JAN-2015 14:47:04
Current_Time	= 20-AUG-2015 14:00:49
Comment	= single pulse decoupled g
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECA500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.7473579[T] (500[MHz])
X_Acq_Duration	= 0.83361792[s]
X_Domain	= 13C
X_Freq	= 125.76529768[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.19959034[Hz]
X_Sweep	= 39.3081761[kHz]
X_Sweep_Clipped	= 31.44654088[kHz]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[MHz]
Irr_Offset	= 5.0[ppm]
Clipped	= FALSE
Scans	= 500
Total_Scans	= 500
Relaxation_Delay	= 2.5[s]
Recvr_Gain	= 60
Temp_Get	= 20.3[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 0.83361792[s]
X_Angle	= 30[deg]
X_Atn	= 4.1[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 21.587[dB]
Irr_Atn_Noe	= 21.587[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 92[us]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2.5[s]
Repetition_Time	= 3.33361792[s]

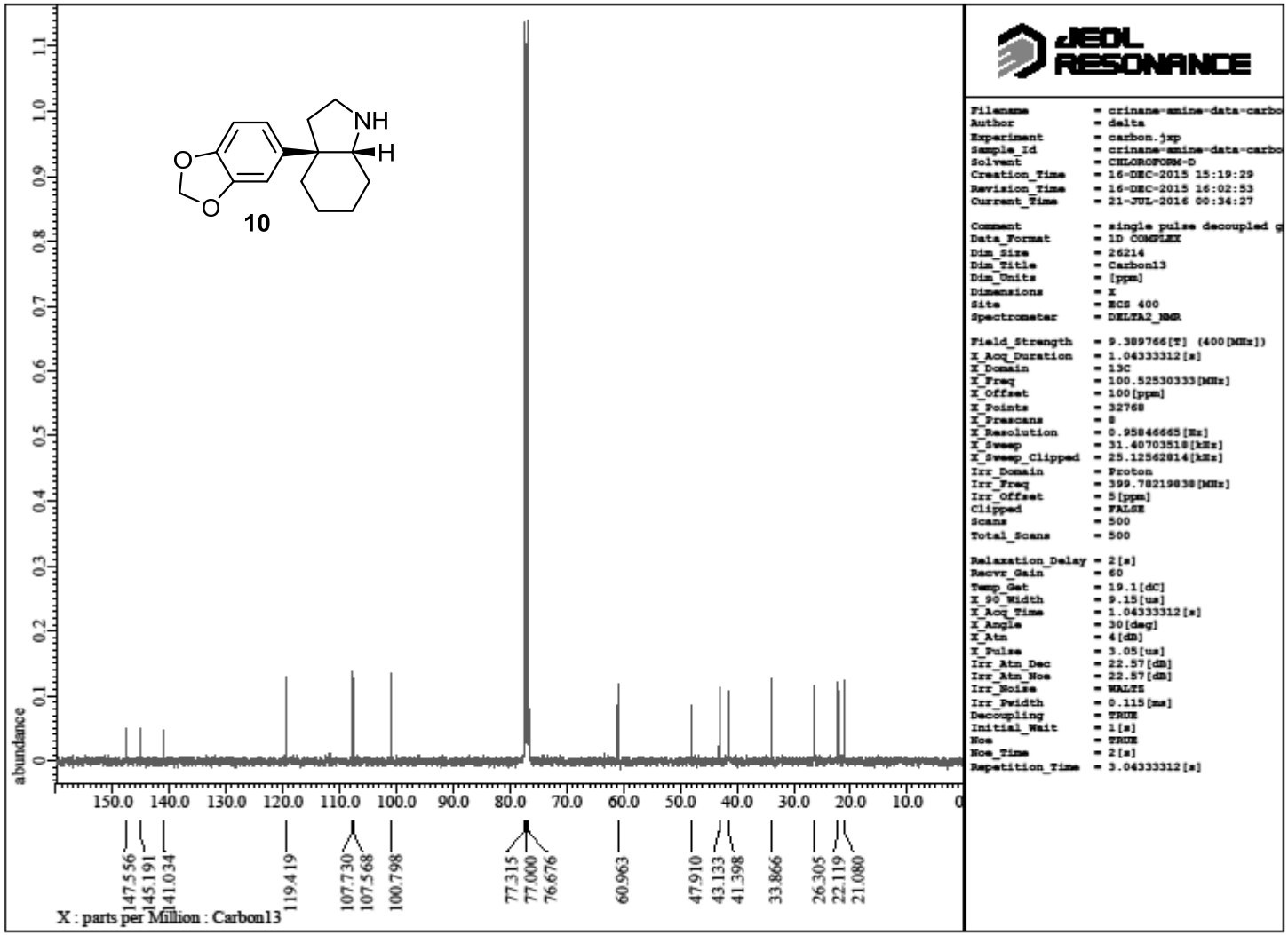

8
OH
abundance
X : parts per Million : Proton
7.260
6.857
6.852
6.774
6.747
5.930
5.913
5.896
5.888
5.879
5.844
5.841
5.819
5.816
3.643
3.629
3.616
3.608
3.601
3.594
3.578
3.568
3.562
3.557
3.542
2.022
2.016
2.000
1.990
1.985
1.975
1.355
1.130
2.01
1.00
3.05
0.99
2.18
4.30
1.48
1.04
1.07
1.10
0.95
Filename = crinane-LAH-reduction-da
Author = delta
Experiment = proton.jxp
Sample_Id = crinane-red-alcohol-data
Solvent = CHLOROFORM-D
Creation_Time = 15-DEC-2015 15:05:03
Revision_Time = 15-DEC-2015 16:34:56
Current_Time = 21-JUL-2016 00:38:10
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 100
Total_Scans = 100
Relaxation_Delay = 2[s]
Recvr_Gain = 44
Temp_Get = 19.4[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]

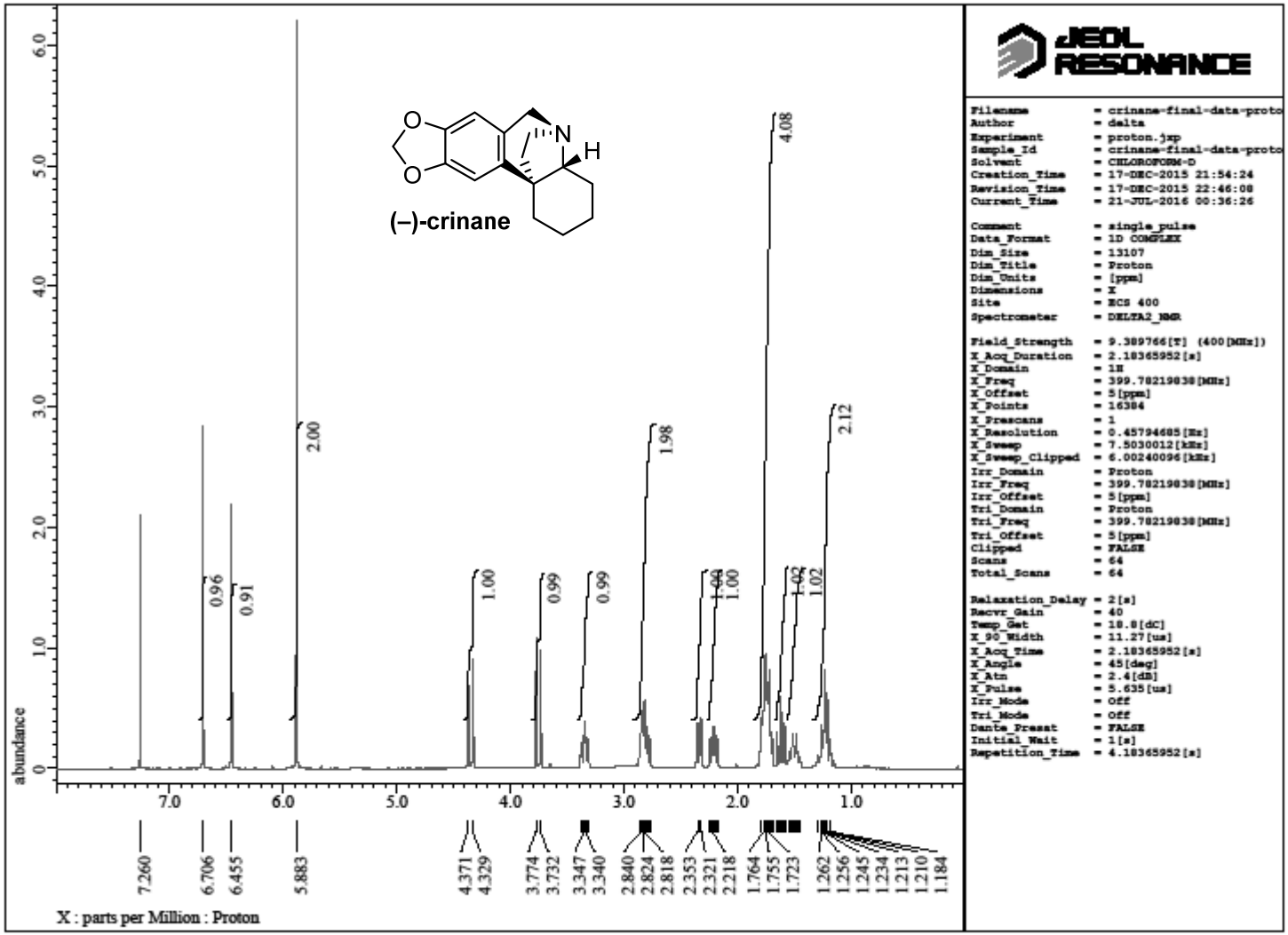

8
OH
O
O
abundance
0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5
150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
147.555
145.399
141.775
132.561
128.440
120.046
107.675
107.475
100.836
77.258
77.000
76.752
59.707
45.237
41.537
37.378
25.283
18.635
X : parts per Million : Carbon13
JEOL RESONANCE
Filename = crinane-LAH-reduction-da
Author = delta
Experiment = carbon.jxp
Sample_Id = S.K3-118-fr1-proton-data
Solvent = CHLOROFORM-D
Creation_Time = 11-JUL-2014 13:10:05
Revision_Time = 17-DEC-2014 13:41:51
Current_Time = 20-AUG-2015 14:01:15
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 500
Total_Scans = 500
Relaxation_Delay = 2.5[s]
Recvr_Gain = 58
Temp_Get = 24.4[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 4.1[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.587[dB]
Irr_Atn_Noe = 21.587[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2.5[s]
Repetition_Time = 3.33361792[s]

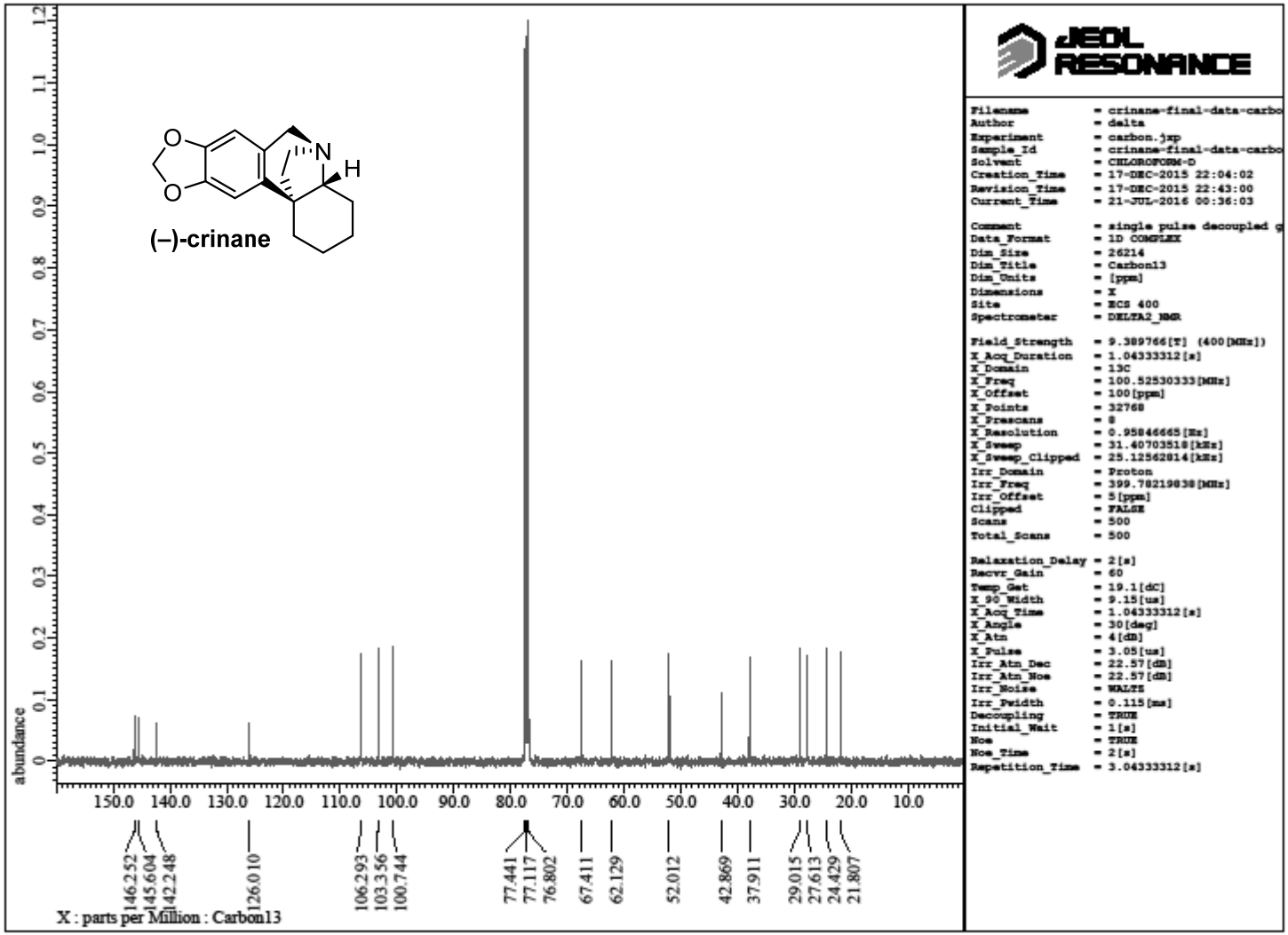

9
abundance
X : parts per Million : Proton
2.01
1.03
1.07
2.03
1.04
3.05
3.88
1.00
7.260
6.747
6.554
6.534
5.939
3.861
3.857
3.852
3.848
3.841
3.837
3.646
3.636
3.607
2.723
2.717
2.688
2.683
2.242
2.231
1.912
1.537
1.504
1.245
Filename = crinane-imine-data-proto
Author = delta
Experiment = proton.jxp
Sample_Id = crinane-imine-data-proto
Solvent = CHLOROFORM-D
Creation_Time = 15-DEC-2015 20:14:29
Revision_Time = 15-DEC-2015 22:08:04
Current_Time = 21-JUL-2016 00:37:52
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 64
Total_Scans = 64
Relaxation_Delay = 2[s]
Recvr_Gain = 38
Temp_Get = 19.8[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.6[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Preset = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]


9
abundance
180.765
148.300
145.821
136.953
119.124
108.397
106.586
100.979
77.324
77.000
76.685
58.522
57.502
41.865
39.243
30.233
27.468
22.434
X : parts per Million : Carbon13
JEOL RESONANCE
Filename = crinane-imine-data-carbo
Author = delta
Experiment = carbon.jxp
Sample_Id = crinane-imine-data-carbo
Solvent = CHLOROFORM-D
Creation_Time = 15-DEC-2015 20:22:41
Revision_Time = 15-DEC-2015 22:00:36
Current_Time = 21-JUL-2016 00:37:24
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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 = 8
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 500
Total_Scans = 500
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 20[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]


NH
H
O
O
O
10
abundance
X : parts per Million : Proton
4.95
3.06
2.27
2.00
1.22
0.98
0.97
0.96
7.260
6.877
6.873
6.816
6.766
5.932
3.425
3.415
3.403
3.120
3.008
2.996
2.290
1.773
1.762
1.754
1.470
1.276
1.267
1.248
1.244
1.239
1.221
1.215
JEOL RESONANCE
Filename = crinane-amine-data-proto
Author = delta
Experiment = proton.jxp
Sample_Id = crinane-amine-data-proto
Solvent = CHLOROFORM-D
Creation_Time = 16-DEC-2015 15:08:40
Revision_Time = 16-DEC-2015 16:27:25
Current_Time = 21-JUL-2016 00:34:54
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 100
Total_Scans = 100
Relaxation_Delay = 2[s]
Recvr_Gain = 44
Temp_Get = 19.5[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Preset = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]


NH
H
O
O
10
abundance
150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
147.556
145.191
141.034
119.419
107.730
107.568
100.798
77.315
77.000
76.676
60.963
47.910
43.133
41.398
33.866
26.305
22.119
21.080
X : parts per Million : Carbon13
JEOL RESONANCE
Filename = crinane-amine-data-carbo
Author = delta
Experiment = carbon.jxp
Sample_Id = crinane-amine-data-carbo
Solvent = CHLOROFORM-D
Creation_Time = 16-DEC-2015 15:19:29
Revision_Time = 16-DEC-2015 16:02:53
Current_Time = 21-JUL-2016 00:34:27
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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 = 8
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 500
Total_Scans = 500
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.1[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]


(–)-crinane
abundance
X : parts per Million : Proton
7.260
6.706
6.455
5.883
4.371
4.329
3.774
3.732
3.347
3.340
2.840
2.824
2.818
2.353
2.321
2.218
1.764
1.755
1.723
1.262
1.256
1.245
1.234
1.213
1.210
1.184
0.96
0.91
2.00
1.00
0.99
0.99
1.98
1.00
1.00
4.08
1.02
2.12
JEOL RESONANCE
Filename = crinane-final-data-proto
Author = delta
Experiment = proton.jxp
Sample_Id = crinane-final-data-proto
Solvent = CHLOROFORM-D
Creation_Time = 17-DEC-2015 21:54:24
Revision_Time = 17-DEC-2015 22:46:08
Current_Time = 21-JUL-2016 00:36:26
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 64
Total_Scans = 64
Relaxation_Delay = 2[s]
Recvr_Gain = 40
Temp_Get = 18.8[dC]
X_90_Width = 11.27[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 5.635[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Preset = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 4.18365952[s]


(–)-crinane
abundance
X : parts per Million : Carbon13
146.252
145.604
142.248
126.010
106.293
103.356
100.744
77.441
77.117
76.802
67.411
62.129
52.012
42.869
37.911
29.015
27.613
24.429
21.807
JEOL RESONANCE
Filename = crinane-final-data-carbo
Author = delta
Experiment = carbon.jxp
Sample_Id = crinane-final-data-carbo
Solvent = CHLOROFORM-D
Creation_Time = 17-DEC-2015 22:04:02
Revision_Time = 17-DEC-2015 22:43:00
Current_Time = 21-JUL-2016 00:36:03
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
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 = 8
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 500
Total_Scans = 500
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 19.1[dC]
X_90_Width = 9.15[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 3.05[us]
Irr_Atn_Dec = 22.57[dB]
Irr_Atn_Noe = 22.57[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]