

Direct Reversible Decarboxylation from Stable Organic Acids in Solution

Duanyang Kong, Patrick Moon, Erica K. J. Lui, Odey Bsharat, **Rylan Lundgren**

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Many classical and emerging methodologies in organic chemistry rely on carbon dioxide extrusion to generate reactive intermediates for subsequent bond-forming events. Synthetic reactions that involve the microscopic reverse, the carboxylation of reactive intermediates such as organometallic nucleophiles, occur under vastly different reaction conditions. We found that under appropriate conditions chemically stable C(sp³) carboxylates undergo rapid, uncatalyzed reversible decarboxylation in solution. The decarboxylation/carboxylation process occurs through the generation and trapping of otherwise undetectable carbanion intermediates that are largely resistant to protodecarboxylation in the presence of Brønsted acids or to trapping by external electrophiles. Isotopically labelled carboxylic acids, including drug molecules and valuable synthetic intermediates, can be prepared in high chemical and isotopic yield by simply supplying an atmosphere of ¹³CO₂ to carboxylate salts in polar aprotic solvents. Our results indicate that the reversibility of decarboxylation from organic acids should be taken into consideration when designing and executing decarboxylative functionalization processes.

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Direct Reversible Decarboxylation from Stable Organic Acids in Solution

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Abstract Many classical and emerging methodologies in organic chemistry rely on carbon dioxide extrusion to generate reactive intermediates for subsequent bond-forming events. Synthetic reactions that involve the microscopic reverse, the carboxylation of reactive intermediates such as organometallic nucleophiles, occur under vastly different reaction conditions. We found that under appropriate conditions chemically stable C(sp³) carboxylates undergo rapid, uncatalyzed reversible decarboxylation in solution. The decarboxylation/carboxylation process occurs through the generation and trapping of otherwise undetectable carbanion intermediates that are largely resistant to protodecarboxylation in the presence of Brønsted acids or to trapping by external electrophiles. Isotopically labelled carboxylic acids, including drug molecules and valuable synthetic intermediates, can be prepared in high chemical and isotopic yield by simply supplying an atmosphere of ¹³CO₂ to carboxylate salts in polar aprotic solvents. Our results indicate that the reversibility of decarboxylation from organic acids should be taken into consideration when designing and executing decarboxylative functionalization processes.

Decarboxylation is a fundamental step in biochemical processes and synthetic organic chemistry. Fermentation, respiration, and the biosynthesis of many secondary metabolites involve the loss of CO₂ from organic acids.¹ Decarboxylases accelerate these reactions by stabilization of developing reactive intermediates, often a carbanion, and encourage CO₂ diffusion from the active site, enabling otherwise unfeasible decarboxylations to occur under physiological condition (Fig 1A).² Acid substrates lacking anion stabilizing groups adjacent to the reactive carbon center are inert towards spontaneous decarboxylation without resorting to pyrolysis conditions (Fig 1B).³ Thus, synthetic reactions driven by decarboxylation often require high reaction temperatures,⁴ the use of oxidizing agents,⁵ or stoichiometric chemical modification of the carboxylate unit.⁶

Carboxylation reactions, the microscopic reverse of decarboxylation, are equally valuable processes in biology and synthetic chemistry. Despite a shared reaction pathway, the biochemical machinery that promote carboxylation in CO₂ fixation operate by a distinct set of substrates and enzymes from those that promote decarboxylation in all but a few rare exceptions.⁷ Similarly, synthetic techniques

that generate carboxylic acid derivatives from CO₂ require strongly nucleophilic organometallics and/or in-situ stoichiometric (electro)chemical substrate reduction.⁸

The potential for the reversibility of decarboxylation/carboxylation mechanistic steps is largely ignored in synthetic methodologies that rely on these reactions. Reports of direct non-enzymatic reversible CO₂-exchange processes of carboxylic acids are restricted to specialized substrate/mediator pairs.⁹ Exchange of carboxylate groups in simple aliphatic acids with CO₂ has been documented, but requires heating of neat substrates at 280–400 °C.¹⁰ Currently, state-of-the-art methods to prepare C(sp³)-^{13/14}CO₂ labeled carboxylic acid-derived targets, sought after in (pre)clinical absorption, distribution, metabolism, and excretion (ADME) studies,¹¹ involve circuitous pathways consisting of chemical activation-decarboxylation-metalation-carboxylation steps mediated by transition metals (Fig 1C).¹² The exchange of C(sp²)-carboxylate groups catalyzed by transition metals has been demonstrated, however reactivity is restricted to stabilized aromatic substrates at high temperature (≥280 °C).¹³ While valuable methods for obtaining labelled targets, these indirect techniques are accompanied by low chemical yields and modest incorporation of isotopic label. Due to these limitations, classical nucleophilic substitution reactions with labelled cyanide followed by hydrolysis or processes that use labelled carbon monoxide remain widely used.¹⁴

In the course of our studies on catalytic decarboxylative cross-coupling reactions,¹⁵ we questioned whether the apparent stability of organic carboxylates could arise from reversible decarboxylation/carboxylation events in solution. Supporting this hypothesis, we observed that simple organic acids that are stable towards protodecarboxylation in solution undergo spontaneous incorporation of ¹³CO₂ when supplied at atmospheric pressure (Fig 1D). This behavior is general to a range of C(sp³) carboxylic acids that feature modest electron-withdrawing groups adjacent to the carboxylate unit (aromatics, carbonyls, imines, nitriles). An understanding of this phenomenon can be exploited to prepare isotopically labelled drug molecules and synthetic precursors in an operationally trivial manner.

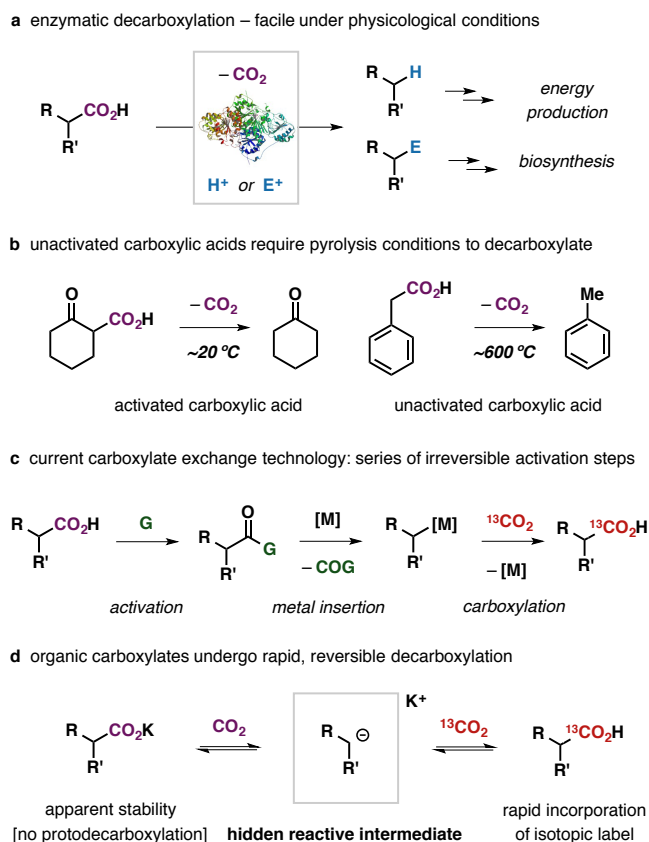


Fig 1. Overview of decarboxylative processes and carboxylate exchange. **(a)** Decarboxylation is catalyzed by enzymes under physiological conditions (E^+ : electrophile). **(b)** The feasibility of protodecarboxylation depends on the stability of the carbanion intermediate generated. **(c)** Current carboxylate exchange technology requires a series of irreversible activation steps. **(d)** This work.

The potassium aryl acetate **1** exemplifies the reversible decarboxylation/carboxylation behavior of otherwise chemically stable carboxylic acids. A 0.1 M solution of **1** in DMF at 20 °C undergoes CO_2 exchange when placed under an atmosphere of $^{13}\text{CO}_2$. In a reaction where approximately seven equivalents of $^{13}\text{CO}_2$ is supplied (13 mL of CO_2 at ~1 atm, dissolved $^{13}\text{CO}_2$ concentration of 0.25 M), equilibrium between ^{12}C and ^{13}C is achieved in 15 hours (Fig 2A, red trace). Quantitative recovery of carboxylate **1** with 83% ^{13}C -enrichment was possible by acid/base extractive workup. Under similar conditions with five equivalents of a weak Brønsted acid (MeOH) no protodecarboxylation of **1** is observed (Fig 2A, black trace). These results demonstrate that re-capture of the putative carbanion intermediate generated by decarboxylation of **1** with dissolved CO_2 is significantly more favorable than protonation. The apparent stability of **1** arises from efficient recapture of CO_2 by an otherwise hidden intermediate.

The counter-cation of the carboxylate salt impacts carboxylate exchange reactivity (Fig 2B). The carboxylic acid of **1** is not reactive, no $^{13}\text{CO}_2$ exchange or protodecarboxylation was observed in DMF at

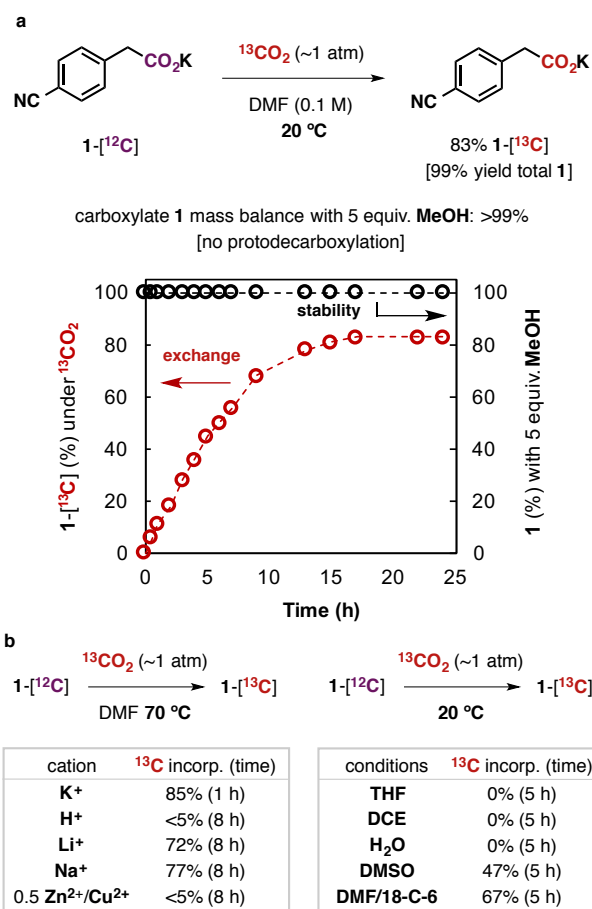


Fig 2. Carboxylate exchange of a stable aryl acetic acid. **(a)** Comparison of CO₂ exchange (red) and protonation with MeOH (black). **(b)** Impact of salt and reaction conditions.

70 °C. Li⁺ and Na⁺ salts of **1** react more slowly, while transition metal salts (Zn²⁺ or Cu²⁺) are inert. The use of polar aprotic solvents is essential for the transformation, reactions conducted in THF, DCE, or water resulted in recovery of unlabeled **1**. The addition of 18-crown-6 (18-C-6) results in an approximate two-fold rate enhancement of carboxylate exchange (see SI Fig S2).¹⁶ The free acid undergoes carboxylate exchange when 1.5 equivalents of K₂CO₃ and 18-C-6 are added (>90% yield and ¹³C incorporation in 19 h). Collectively, these observations suggest that the generation of a solvent-separated ion pair leads to enhanced decarboxylative reactivity. Reversible decarboxylation occurs for an array of carboxylate containing molecules, including valuable synthetic precursors, drug molecules, and amino acid derivatives (Fig 3 and 4). The incorporation of ¹³CO₂ and product recovery remains high (>80%) across several substrate classes. The degree of incorporation is largely a function of the amount of ¹³CO₂ supplied, >95% enrichment can be obtained when a significant excess is provided (see SI Fig S3). Regardless of electronic properties, aryl acetates undergo ¹³CO₂ incorporation by conducting reactions at suitable temperatures. (Hetero)aryl acetates with anion stabilizing groups exchange at moderate temperatures (**1-4**, **9**, **10**, **11-15** at 20–80 °C), while aryl acetates with strongly electron-donating

OMe or NMe₂ groups require higher temperatures (**17-20** at 100–130 °C) and benefit from the addition of 18-C-6. The simplicity of the process enables broad functional group compatibility, including tolerance to boronic esters (**6**), aryl halides (I, Br, Cl, F; **4, 7, 8, 10**), ketones (**11**), aldehydes (**12**) esters (**14**), amides (**13**), sulfonyls (**15**), and potentially reactive heterocycles (chromenone **25**, NH-indole **26**, pyridines **27, 29**, pyrimidines **28**, isoxazole **30**, thiophene **31**). Alkyl and aryl substitution adjacent to the carboxylate is tolerated, including examples of trisubstituted, non-enolizable aryl acetates (**21-24**). Other classes of potassium carboxylates that undergo productive CO₂ exchange include malonate half-esters (**32-35**), β-keto acids (**36**), β-carboxysulfonyls (**37, 38**), cyanoacetates (**39**), and carboxylactams (**40**). Alkene and terminal alkyne functional groups do not interfere with the process (**34, 35**). Potassium malonates undergo exchange at higher temperature (135 °C) to give a mixture of mono- and bis-labelled product along with ¹³C-enriched monoacid (**41** and **42**).

Carboxylate exchange can be used to directly prepare isotopically labelled drug molecules, including aryl acetate and propionate NSAIDs of varying complexity (Fig 4 **43-52**). Pharmaceuticals featuring amide or ester groups are obtained via derivatization of the acid group (Zolpidem **53**, Aprophen **55**) or can be prepared according to established literature protocols (Propiverine **54**, Netupitant **56**, Repaglinide **57**). Consistent with ionization to generate a carbanion, racemization of enantiopure Naproxen (**46**) is observed (see SI Fig S4). Reversible decarboxylation may explain reports of aryl propionate racemization required for kinetic resolution manufacturing processes.¹⁷ Unsubstituted alkyl carboxylic acids do not undergo CO₂ exchange, however isotopically labelled products of this class can be readily obtained by carboxylate exchange/desulfonylation reactions of sulfonyl acids or exchange/decarboxylation sequences of malonic acids in three step processes (**58-60**). The facile generation of ¹³C-diphenylmethylidene glycine at room temperature (**61** 93% incorporation, 76% yield) serves as a starting point for the synthesis of other labelled amino acids.¹⁸

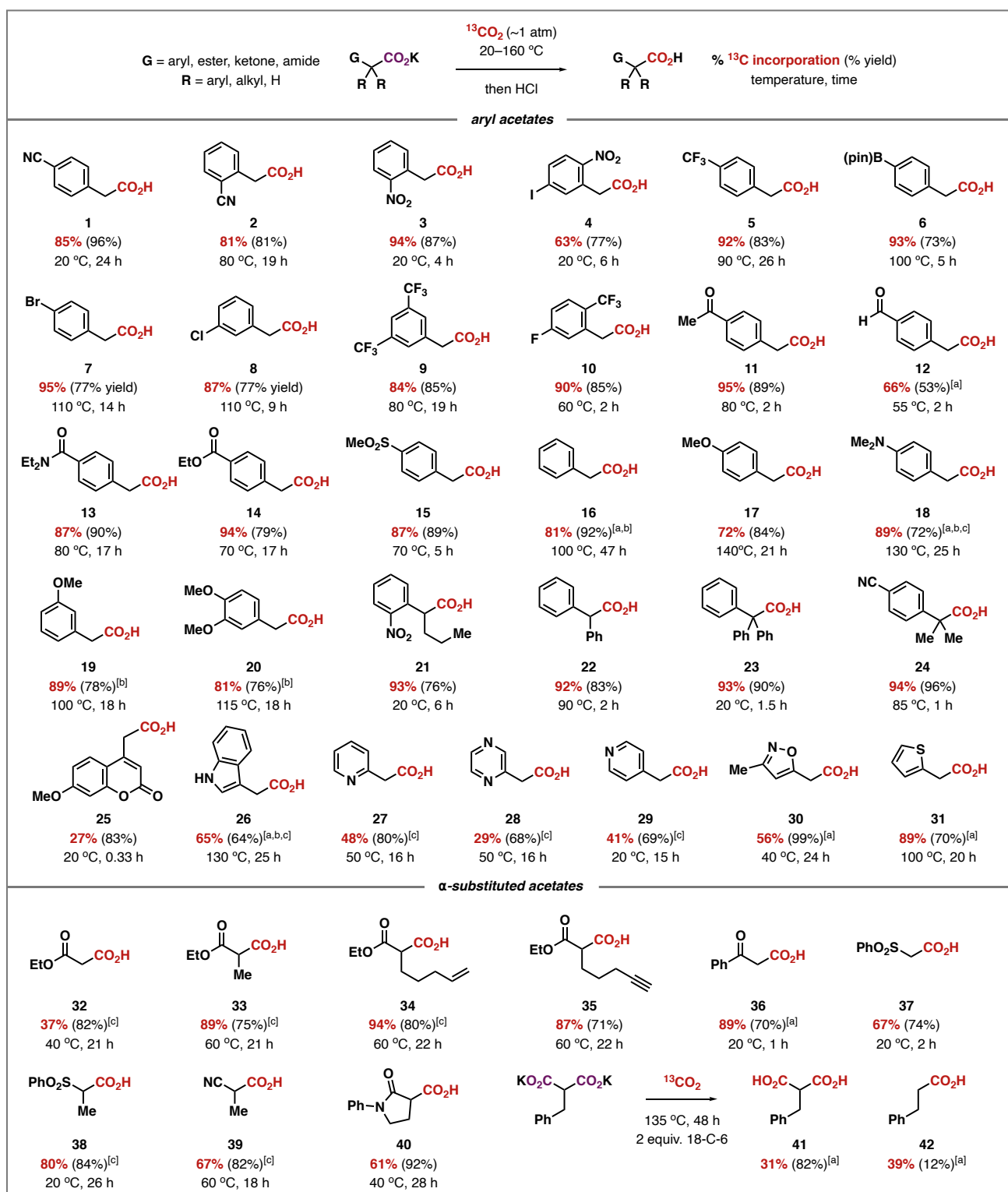


Fig 3. Carboxylate exchange substrate scope. Unless noted yields are of isolated material. [a] calibrated ^1H NMR spectroscopy yield; [b] 18-C-6 added; [c] $\% ^{13}\text{C}$ incorporation and yield determined by analysis of the corresponding ester obtained by reaction with MeI or BnBr. See SI for complete details.

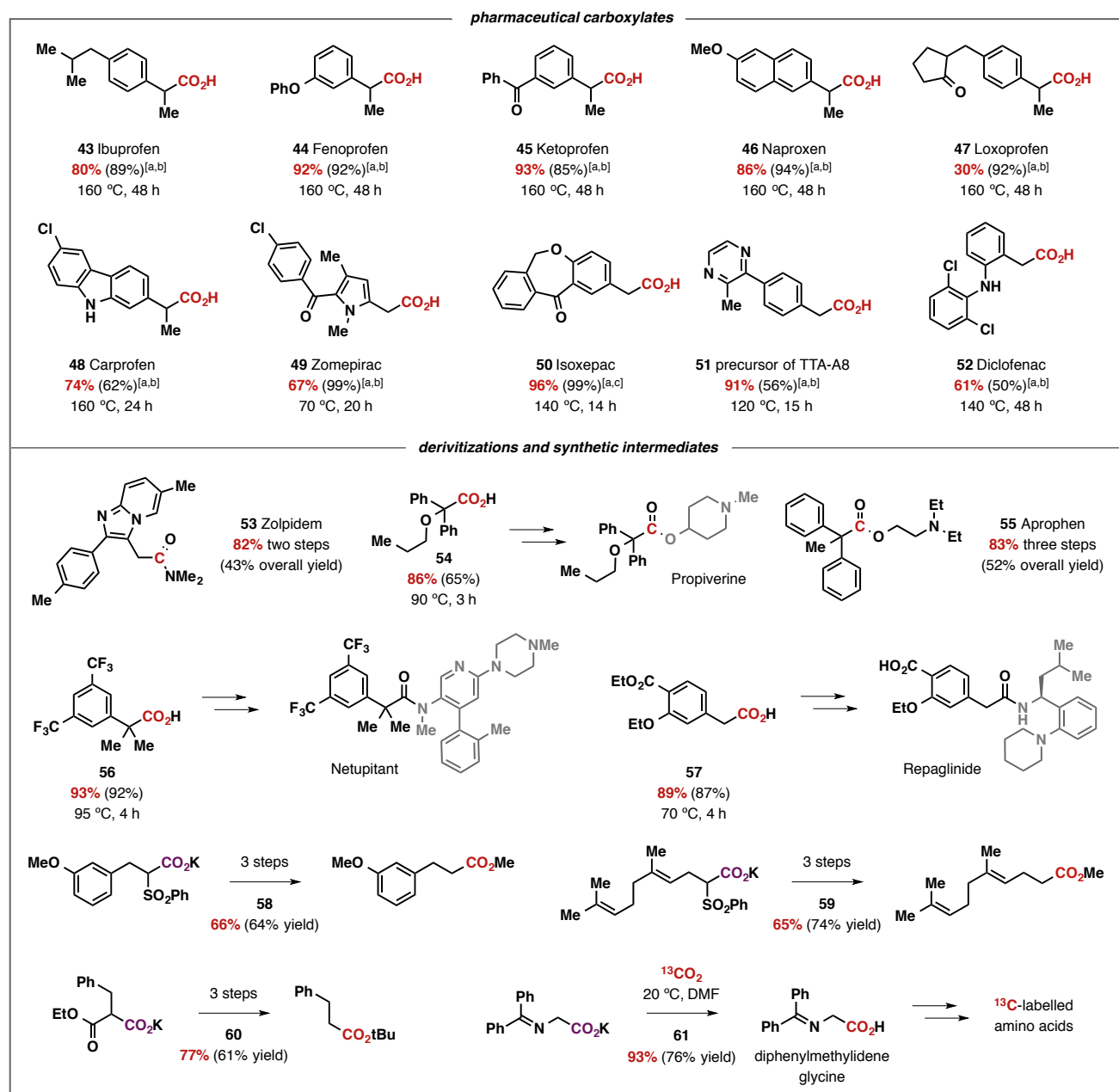


Fig 4. Application of carboxylate exchange, see Fig 3 for experimental details [a] yield determined by ^1H NMR spectroscopy; [b] DMSO instead of DMF; [c] 1.0 equiv 18-C-6 added.

A mechanism for the reversible CO_2 exchange supported by control experiments and structure-reactivity studies involves the direct ionization of the potassium carboxylate to generate a carbanion. The reaction rates and required temperatures for $^{12}\text{CO}_2/^{13}\text{CO}_2$ interconversion correlate with the substrate's ability to stabilize negative charge and not with oxidation potential (compare **1**, **14**, **16**, and **17**). The addition of radical inhibitors (TEMPO, BHT) has no impact on the decarboxylative reactivity of **1** and cyclization of the pendant olefin in **34** is not detected.

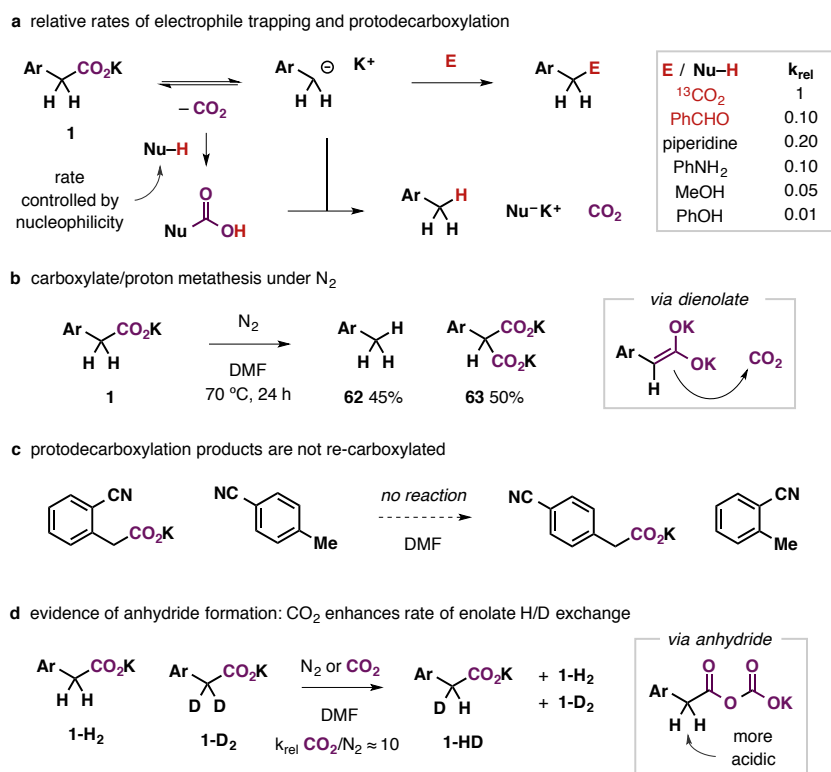


Fig 5. Carbanion trapping studies and mechanistic control experiments. Ar = (4-CN)C₆H₄.

Exchange of CO₂ via a carbanion without competing quenching by other electrophiles (ketones, aldehydes, weak Brønsted acids), stems in part from the relatively high solubility of CO₂ in DMF and the slow kinetics of CO₂ dissolution into the reactor headspace. For example, a 0.25 M solution of CO₂ in DMF retains a concentration of 0.2 M under a headspace of N₂ over one day. For substrate **1** the rate of CO₂ exchange at 70 °C is ~10-fold faster than for reaction with benzaldehyde. Counterintuitively, the rate of protodecarboxylation by weak Brønsted acids is inversely related to acidity (Fig 5a; piperidine, aniline, methanol, phenol see the SI Fig S5 for details). This observation could be attributed to the relative ability of these species to act as nucleophiles to trap the liberated CO₂, rather than directly protonate a carbanion. At 20 °C <10% trapping with these additives is observed after 8 hours. At 70 °C under N₂, **1** undergoes net carboxylate/proton metathesis to generate a half equivalent of the protodecarboxylated product **62** and a half equivalent of the CO₂-trapped malonate **63** (Fig 5b). Product **62** likely arises from deprotonation of a second equivalent of aryl acetate to generate a dienolate nucleophile. The dienolate can be carboxylated by CO₂ released by the initial decarboxylation event. This observation demonstrates the striking efficiency of CO₂ capture by carbon nucleophiles under suitable conditions. Alkyl arene generated by protodecarboxylation does not convert back to carboxylate under the conditions where carboxylate exchange is observed (Fig 5c). Carbonic anhydride intermediates are likely generated under

the reaction conditions on the basis of the observed increase in α -carboxyl H/D exchange rates with **1-H₂** and **1-D₂** under CO₂ (Fig 5d, see SI Fig S7 for details). The generation of a dienolate from the more acidic potassium carbonic anhydride may explain these reactivity differences. Direct detection of anhydride intermediates was not achieved. The ability of non-enolizable carboxylates, such as **23** and **24**, to undergo reversible decarboxylation indicate that a dienolate intermediate is not essential for carboxylate exchange.

The observation that chemically stable carboxylates undergo rapid and reversible decarboxylation/carboxylation processes enable a simple protocol to prepare isotopically labelled small molecules with enriched CO₂. This phenomenon should be taken into consideration when designing and executing decarboxylative functionalization processes.

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Direct Reversible Decarboxylation from Stable Organic Acids in Solution

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I. General Considerations

Unless noted, all reactions were conducted under inert atmosphere employing standard Schlenk technique or by the use of a N₂-filled glovebox. All glassware was oven-dried prior to use. Flash chromatography was performed as described by Still and co-workers¹ (SiliaFlash P60, 40-63µm, 60Å silica gel, Silicycle) or by automated flash chromatography (SiliCycle silica cartridges, Biotage). Analytical thin-layer chromatography was performed using glass plates pre-coated with silica (SiliaPlate G TLC - Glass-Backed, 250µm, Silicycle). HPLC analysis was accomplished on an Agilent 1290 system with Daicel CHIRALPAK IA, IB, IC or IG columns (4.6 × 150 mm, 5 µm particle size). NMR spectra (¹H, ¹³C, ¹⁹F) were obtained on an Agilent VNMRS 700 MHz, Varian VNMRS 600 MHz, Varian VNMRS 500 MHz, or Varian 400 MHz spectrometer. HRMS analyses of carbon-13 labeled compounds were performed on an Agilent Technologies 6220 oaTOF instrument (ESI, APPI and APCI) or Kratos Analytical MS-50G instrument (EI) in positive or negative ionization mode. DMF and ¹³CO₂ (99.0 atom % ¹³C) were purchased from Sigma-Aldrich. The corresponding aryl acetic acids were obtained from commercial vendors. Potassium aryl acetate salts (**18**, **27** and **29**) were synthesized from the corresponding aryl acetic esters as described by Liu and co-workers.² Malonate half ester salts (**34** and **35**) were synthesized according to the literature procedure from the corresponding malonate esters aryl acetic esters.^{3,4} *N*-(diphenylmethylene)glycinate potassium **61** was synthesized from ethyl *N*-diphenylmethylene glycine as described by Alezra and co-workers.⁵ Other aryl acetate salts were synthesized from the corresponding aryl acetic acids as described by Liu and co-workers.⁴

Concentration of ¹³CO₂ in DMF Solutions: In a glovebox filled with N₂, *d*₇-DMF (1 mL) was added to a dried 4-dram vial charged with a stir bar. The vial was sealed with a PTFE-lined cap and removed from the glovebox. The reaction headspace was evacuated (~ 300 mTorr) via a 25-gauge needle. The headspace was next carefully refilled with ~1 atm ¹³CO₂ through the PTFE cap using a 25-gauge needle and stirred at room temperature for 15 minutes. *d*₇-DMF (0.6 mL) was transferred into a *J*-Young NMR tube filled with ¹³CO₂. A concentration of 0.25 M ¹³CO₂ was determined by calibrated ¹³C NMR spectroscopy by comparison with the residual ¹³C in *d*₇-DMF at different time points (15 min, 1 h, 5 h, and 24 h. In a second experiment, *d*₇-DMF (0.6 mL) was transferred into a *J*-Young NMR tube filled with N₂. A concentration of 0.20 M ¹³CO₂ was determined by calibrated ¹³C NMR spectroscopy, which remained static over 24 h. [Note on NMR acquisition: To avoid the need to measure the relaxation time of *d*₇-DMF and CO₂ a single scan (with no steady state scans) with the spectral window set from 170 ppm to 120 ppm was performed when measuring the concentration of CO₂ in DMF.]

II. Graphical Supporting Information for General Procedures

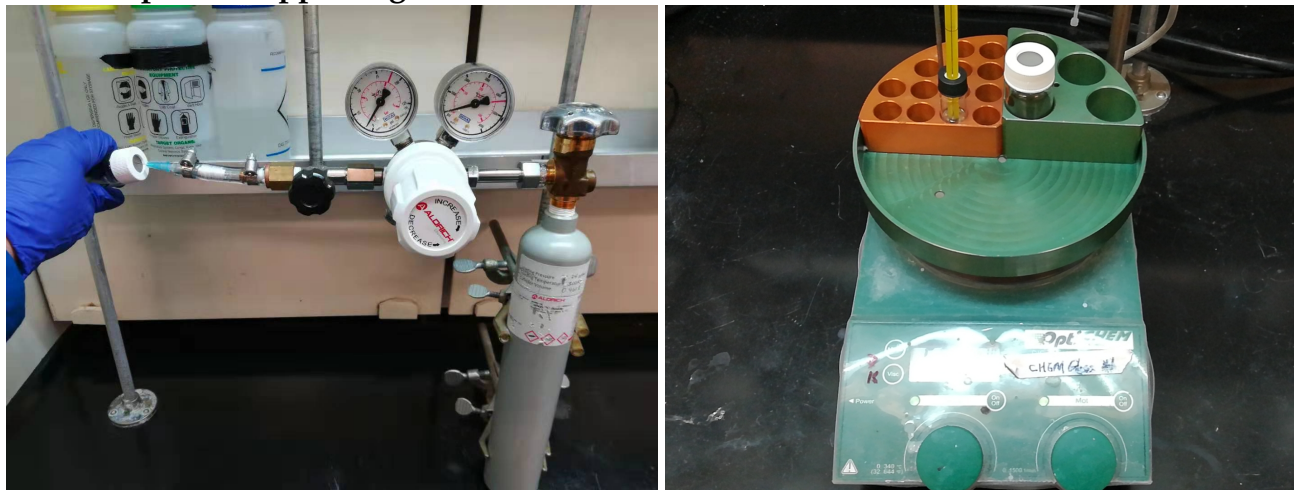


Fig S1. (Left) $^{13}\text{CO}_2$ setup to introduce ~1 atm of gas to a reaction vial. **(Right)** The sealed vial and thermometer setup in aluminum heating blocks.

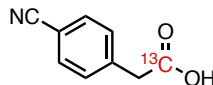
III. General Procedure of $^{13}\text{CO}_2$ Incorporation from Carboxylate Salts

General Procedure A: In a glovebox filled with N_2 , potassium carboxylate salt (0.1 mmol, 1.0 equiv, 1,3,5-trimethoxybenzene (an internal standard) and anhydrous DMF or DMSO (0.1 M) were added sequentially to a dried 4-dram vial charged with a stir bar. The potassium carboxylates are moderately hygroscopic and best stored under inert atmosphere, however a glovebox is not required (see below). The vial was sealed with a PTFE-lined cap and removed from the glovebox. The reaction headspace was evacuated (~ 300 mTorr), via a 25-gauge needle. The headspace was next carefully refilled with ~1 atm $^{13}\text{CO}_2$ through the PTFE cap using a 25-gauge needle and stirred at the corresponding temperature. Reactions at elevated temperatures were heated in an aluminum block. ^1H NMR or LC-MS analysis of small aliquots (~5 μL) was used to follow reactions. Optimal temperatures for carboxylate exchange without re-charging of $^{13}\text{CO}_2$ could be found by adjusting the temperature 10–20 $^\circ\text{C}$ every hour until ^{13}C incorporation was observed. Upon desired incorporation of the ^{13}C -isotope, the mixture was cooled to room temperature. Quantitative ^1H NMR yields were determined from crude reaction mixtures using 1,3,5-trimethoxybenzene as an internal standard prior to the purification. The mixture was diluted with H_2O (10 mL) and washed with EtOAc (3 x 10 mL) to remove the internal standard and protodecarboxylation side-product. Then the aqueous layer was acidified using 1M HCl solution. The aqueous layer was extracted with EtOAc (3 x 10 mL) and the combined organic layers were dried over anhydrous NaSO_4 , filtered, and concentrated *in vacuo* to give the desired product. The $^{13}\text{C}\%$ incorporation was obtained through analysis of high-resolution mass spectrum of the compounds.

For some substrates, the ^{13}C % incorporation was obtained through analysis of the corresponding carboxylic acid ester compound obtained by esterification with MeI or BnBr. Then MeI or BnBr (1 equiv.) was added to this solution at room temperature and stirred for 1–2 hour. (Quantitative ^1H NMR yields were determined from crude reaction mixtures using 1,3,5-trimethoxybenzene as an internal standard prior to the purification.) After purification through a series of extractions and column chromatography, the desired product was obtained.

General Procedure B: Potassium carboxylate salt (0.1 mmol, 1.0 equiv.) was added to a dried 4-dram vial charged with a stir bar. The vial was then refilled with N_2 and evacuated under reduced pressure (3x). Under N_2 flow, using a 1 mL syringe with a 25-gauge needle, DMF or DMSO (1 mL) was added to the reaction vessel. Then the reaction headspace is evacuated (~ 300 mTorr), via a 25-gauge needle. The remainder for the procedure is identical to General Procedure A.

IV. Specific Experimental Details and Product Characterization Data

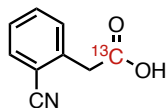


1 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at room temperature for 24 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 96% yield with 85% ^{13}C incorporation. *Procedure using the free acid:* Adapted from General Procedure A, using 4-cyanophenylacetic acid (0.1 mmol, 1 equiv.), K_2CO_3 (1.5 equiv.) and 18-crown-6 (1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at room temperature for 19 hours. The desired product was obtained in >90% NMR yield with >95% ^{13}C incorporation.

^1H NMR (CDCl_3 , 500 MHz) δ 7.63 (d, J = 8.2 Hz, 2H), 7.40 (d, J = 8.2 Hz, 2H), 3.73 (d, J = 8.5 Hz, 2H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 175.9, 138.4, 132.4, 130.3, 118.5, 111.6, 40.9 (d, J = 55.1 Hz);

HRMS (ESI): calcd for $\text{C}_8[^{13}\text{C}]\text{H}_7\text{NO}_2\text{Na}$ $[\text{M}+\text{Na}]^+$: 185.0403. Found 185.0404.

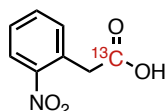


2 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 80 °C for 19 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 81% yield with 81% ^{13}C incorporation.

^1H NMR (CDCl_3 , 400 MHz) δ 7.68 – 7.66 (m, 1H), 7.57 (td, J = 8.2 Hz, 1.3 Hz, 1H), 7.38 – 7.43 (m, 2H), 3.92 (d, J = 7.4 Hz, 2H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 175.2, 137.1, 132.9, 132.8, 130.7, 127.9, 117.4, 113.5, 39.3 (d, J = 62.6 Hz);

HRMS (APPI): calcd for $\text{C}_8[^{13}\text{C}]\text{H}_6\text{NO}_2$ $[\text{M}-\text{H}]^-$: 161.0438. Found 161.0434.

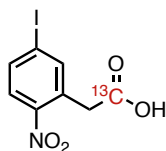


3 Prepared according to General Procedure B from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at room temperature for 4 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 87% yield with 94% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 8.14 (dd, J = 8.2 Hz, 1.2 Hz, 1H), 7.61 (td, J = 7.5 Hz, 1.2 Hz, 1H), 7.51 – 7.48 (m, 1H), 7.37 (d, J = 7.5 Hz, 1H), 4.07 (d, J = 8.3 Hz, 2H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 175.6, 148.7, 133.9, 133.6 (d, J = 2.1 Hz), 129.3 (d, J = 2.6 Hz), 129.0, 125.5, 39.7 (d, J = 57.1 Hz);

HRMS (ESI): calcd for $\text{C}_7[^{13}\text{C}]\text{H}_7\text{NO}_4$ $[\text{M}+\text{NH}_4]^+$: 200.0743. Found 200.0743.



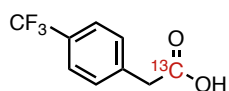
4 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at room

temperature for 6 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 77% yield with 63% ^{13}C incorporation.

^1H NMR (CD_3OD , 400 MHz) δ 7.91 – 7.89 (m, 2H), 7.84 – 7.81 (m, 1H), 3.99 (d, J = 8.1 Hz, 2H);

^{13}C NMR (CD_3OD , 100 MHz) δ 173.3, 150.0, 143.5, 138.9, 133.5, 127.3, 101.6, 39.8 (d, J = 55.3 Hz);

HRMS (APPI): calcd for $\text{C}_7[^{13}\text{C}]\text{H}_6\text{INO}_4$ $[\text{M}]^-$: 307.9381. Found 307.9377.



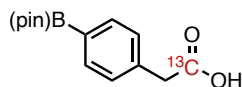
5 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 90 °C for 26 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 83% yield with 92% ^{13}C incorporation.

^1H NMR (CDCl_3 , 600 MHz) δ 7.60 (d, J = 8.0 Hz, 2H), 7.41 (d, J = 8.0 Hz, 2H), 3.72 (d, J = 7.7 Hz, 2H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 176.0, 137.3, 130.3, 129.9 (t, J = 15.4 Hz), 125.7, 124.1 (q, J = 271.8 Hz), 40.7 (d, J = 55.8 Hz);

^{19}F NMR (CDCl_3 , 376 MHz) δ -62.6;

HRMS (APPI): calcd for $\text{C}_8[^{13}\text{C}]\text{H}_6\text{F}_3\text{O}_2$ $[\text{M}-\text{H}]^-$: 204.0359. Found 204.0356.

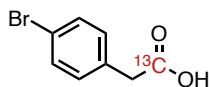


6 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 100 °C for 5 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 73% yield with 93% ^{13}C incorporation.

^1H NMR (CDCl_3 , 500 MHz) δ 7.78 (d, J = 7.9 Hz, 2H), 7.29 (d, J = 7.9 Hz, 2H), 3.66 (d, J = 7.9 Hz, 2H), 1.36 (s, 12H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 176.2, 136.4, 135.2, 128.8, 83.8, 41.1 (d, J = 55.2 Hz), 24.9; (the quaternary carbon attached to boron was not observable)

HRMS (ESI): calcd for $\text{C}_{13}[^{13}\text{C}]\text{H}_{23}[^{11}\text{B}]\text{NO}_4$ $[\text{M}+\text{NH}_4]^+$: 281.1748. Found 281.1748.

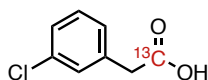


7 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 110 °C for 14 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 77% yield with 95% ^{13}C incorporation.

^1H NMR (CDCl_3 , 500 MHz) δ 7.46 (d, J = 8.3 Hz, 2H), 7.16 (d, J = 8.3 Hz, 2H), 3.61 (d, J = 8.7 Hz, 2H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 177.4, 158.8, 130.4, 125.3, 114.1, 40.1 (d, J = 55.9 Hz);

HRMS (APPI): calcd for $\text{C}_7[^{13}\text{C}]\text{H}_6\text{BrO}_2$ $[\text{M}-\text{H}]^-$: 213.9590. Found 213.9590.

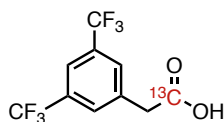


8 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 110 °C for 9 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 77% yield with 87% ^{13}C incorporation.

^1H NMR (CDCl_3 , 400 MHz) δ 7.31 – 7.28 (m, 3H), 7.20 – 7.18 (m, 1H), 3.65 (d, J = 7.8 Hz, 2H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 176.7, 135.1, 134.5, 129.9, 129.8, 129.6, 127.6, 40.6 (d, J = 56.2 Hz);

HRMS (APPI): calcd for $\text{C}_7[^{13}\text{C}]\text{H}_6\text{ClO}_2$ $[\text{M}-\text{H}]^-$: 170.0095. Found 170.0093.



9 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 80 °C for 19

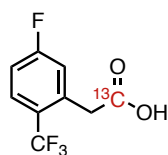
hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 85% yield with 84% ^{13}C incorporation.

^1H NMR (CDCl_3 , 400 MHz) δ 7.82 (s, 1H), 7.75 (s, 2H), 3.81 (d, J = 7.9 Hz, 2H);

^{13}C NMR (CDCl_3 , 100 MHz) δ 175.3, 135.5, 132.1 (q, J = 33.2 Hz), 129.8, 123.2 (q, J = 272.0 Hz), 121.6 (q, J = 3.8 Hz), 40.3 (d, J = 58.8 Hz);

^{19}F NMR (CDCl_3 , 376 MHz) δ -62.9;

HRMS (APPI): calcd for $\text{C}_9[^{13}\text{C}]\text{H}_5\text{F}_6\text{O}_2$ [M-H] $^-$: 272.0233. Found 272.0239.



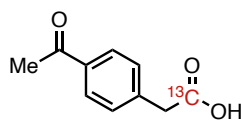
10 Prepared according to General Procedure B from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 60 °C for 2 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 85% yield with 90% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.67 (dd, J = 8.7 Hz, 5.5 Hz, 1H), 7.12 (dd, J = 9.0 Hz, 2.3 Hz, 1H), 7.09 (td, J = 8.2 Hz, 2.2 Hz, 1H), 3.86 (dd, J = 8.3 Hz, 0.8 Hz, 2H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 176.0, 164.4 (d, J = 253.2 Hz), 134.8 (ddq, J = 8.7 Hz, 2.6 Hz, 1.3 Hz), 128.7 (dq, J = 9.8 Hz, 5.1 Hz), 125.5, 124.0 (q, J = 273.1 Hz), 120.0 (dd, J = 22.7 Hz, 1.4 Hz), 114.9 (d, J = 21.7 Hz), 37.9 (d, J = 56.0 Hz);

^{19}F NMR (CDCl_3 , 376 MHz) δ -59.3, -107.5 (q, J = 7.0 Hz);

HRMS (ESI): calcd for $\text{C}_8[^{13}\text{C}]\text{H}_5\text{F}_4\text{O}_2$ [M-H] $^-$: 222.0265. Found 222.0268.

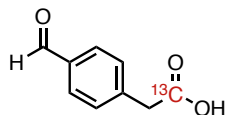


11 Prepared according to General Procedure B from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 80 °C for 2 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 89% yield with 95% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.94 – 7.92 (m, 2H), 7.39 (d, J = 8.0 Hz, 2H), 3.72 (d, J = 8.0 Hz, 2H), 2.60 (s, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 197.9, 178.4, 138.7 (d, $J = 2.9$ Hz), 136.4, 129.8 (d, $J = 1.9$ Hz), 128.9, 41.0 (d, $J = 55.5$ Hz), 26.7;

HRMS (ESI): calcd for $\text{C}_9[^{13}\text{C}]\text{H}_{10}\text{O}_3$ $[\text{M}+\text{H}]^+$: 180.0737. Found 180.0729.

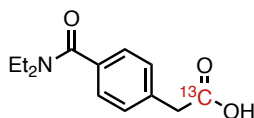


12 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 55 °C for 2 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 22% yield (53% NMR yield) with 66% ^{13}C incorporation.

^1H NMR (CDCl_3 , 500 MHz) δ 10.01 (s, 1H), 7.86 (d, $J = 8.1$ Hz, 2H), 7.47 (d, $J = 8.1$ Hz, 2H), 3.76 (d, $J = 7.5$ Hz, 2H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 191.8, 174.6, 140.1, 135.6, 130.2, 130.1, 40.8 (d, $J = 55.4$ Hz);

HRMS (ESI): calcd for $\text{C}_8[^{13}\text{C}]\text{H}_9\text{O}_3$ $[\text{M}+\text{H}]^+$: 166.0580. Found 166.0576.

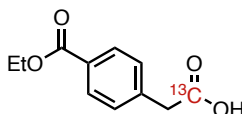


13 Prepared according to General Procedure B from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 80 °C for 17 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 90% yield with 87% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.30 – 7.29 (m, 2H), 7.27 – 7.26 (m, 2H), 3.58 (d, $J = 7.7$ Hz, 2H), 3.55 (s, br, 2H), 3.25 (s, br, 2H), 1.24 (s, br, 3H), 1.10 (s, br, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 174.8, 171.9, 135.5 (d, $J = 2.9$ Hz), 135.2, 129.4 (d, $J = 1.9$ Hz), 126.7, 43.6, 41.3 (d, $J = 55.4$ Hz), 39.7, 14.3, 13.0;

HRMS (ESI): calcd for $\text{C}_{12}[^{13}\text{C}]\text{H}_{17}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 237.1314. Found 237.1303.

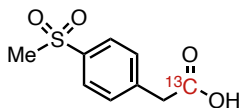


14 Prepared according to General Procedure B from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 70 °C for 17 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 79% yield with 94% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 8.02 – 8.00 (m, 2H), 7.36 – 7.35 (m, 2H), 4.37 (q, J = 7.1 Hz, 2H), 3.71 (d, J = 7.9 Hz, 2H), 1.39 (t, J = 7.1 Hz, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 176.8, 166.5, 138.3 (d, J = 2.7 Hz), 130.0, 129.8, 129.6 (d, J = 1.9 Hz), 61.2, 41.0 (d, J = 55.4 Hz), 14.5;

HRMS (ESI): calcd for $\text{C}_{10}[^{13}\text{C}]\text{H}_{12}\text{O}_4$ $[\text{M}+\text{H}]^+$: 210.0842. Found 210.0836.

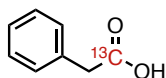


15 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 70 °C for 5 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 89% yield with 87% ^{13}C incorporation.

^1H NMR (CD_3OD , 600 MHz) δ 8.1 (d, J = 8.4 Hz, 2H), 7.75 (d, J = 8.4 Hz, 2H), 3.95 (d, J = 7.9 Hz, 2H), 3.10 (s, 3H);

^{13}C NMR (CD_3OD , 125 MHz) δ 174.1, 142.7, 140.6, 131.6, 128.5, 44.4, 41.8 (d, J = 58.8 Hz);

HRMS (ESI): calcd for $\text{C}_8[^{13}\text{C}]\text{H}_{10}\text{O}_4\text{SNa}$ $[\text{M}+\text{Na}]^+$: 238.0226. Found 238.0225.

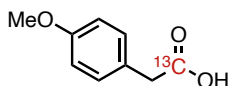


16 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 120 °C for 47 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 83% yield with 94% ^{13}C incorporation. When the reaction mixture was stirred at 100 °C for 47 hours in the presence of 18-crown-6 (1 equiv.), the desired product was obtained in 92% NMR yield with 81% ^{13}C incorporation.

^1H NMR (CDCl_3 , 500 MHz) δ 7.35 – 7.27 (m, 5H), 3.66 (d, J = 7.7 Hz, 2H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 177.4, 133.3, 129.4, 128.7, 127.4, 41.2 (d, J = 54.0 Hz);

HRMS (APPI): calcd for $C_7[^{13}C]H_7O_2$ $[M-H]^-$: 136.0485. Found 136.0485.

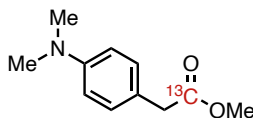


17 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}CO_2$. The reaction mixture was allowed to stir at 140 °C for 21 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 84% yield with 72% ^{13}C incorporation.

1H NMR ($CDCl_3$, 500 MHz) δ 7.20 (d, J = 8.4 Hz, 2H), 6.86 (d, J = 8.4 Hz, 2H), 3.79 (s, 3H) 3.59 (d, J = 7.8 Hz, 2H);

^{13}C NMR ($CDCl_3$, 125 MHz) δ 176.8, 132.3, 131.8, 131.2, 121.6, 55.2, 40.4 (d, J = 55.3 Hz);

HRMS (APPI): calcd for $C_8[^{13}C]H_9O_3$ $[M-H]^-$: 166.0591. Found 166.0590.

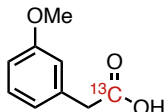


18 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}CO_2$. The reaction mixture was allowed to stir at 140 °C for 25 hours. Then MeI (1 equiv.) was added to this solution at room temperature and stirred for 1 hour. After purification through a series of extractions and column chromatography, the desired product was obtained in 68% yield with 34% ^{13}C incorporation. When the reaction mixture was stirred at 130 °C for 25 hours in the presence of 18-crown-6 (1 equiv.), the desired product was obtained in 72% NMR yield with 89% ^{13}C incorporation.

1H NMR ($CDCl_3$, 600 MHz) δ 7.15 (d, J = 8.7 Hz, 2H), 6.69 (d, J = 8.7 Hz, 2H), 3.67 (t, J = 1.8 Hz, 3H), 3.53 (d, J = 7.9 Hz, 2H), 2.93 (s, 6H);

^{13}C NMR ($CDCl_3$, 125 MHz) δ 172.8, 149.8, 129.9, 121.8, 112.8, 51.9, 40.8, 40.3 (d, J = 60.0 Hz);

HRMS (APPI): calcd for $C_{10}[^{13}C]H_{14}NO_2$ $[M-H]^-$: 193.1064. Found 193.1061.

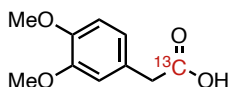


19 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) and 18-crown-6 (1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 100 °C for 18 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 78% yield with 89% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.25 – 7.24 (m, 1H), 6.87 – 6.82 (m, 3H), 3.79 (s, 3H), 3.62 (d, J = 7.7 Hz, 2H);

^{13}C NMR (CDCl_3 , 175 MHz) δ 176.9, 159.7, 134.6, 129.6, 121.7, 115.0, 112.9, 55.2, 40.9 (d, J = 55.1 Hz);

HRMS (ESI): calcd for $\text{C}_8[^{13}\text{C}]\text{H}_9\text{O}_3$ [$\text{M}-\text{H}$] $^-$: 166.0591. Found 166.0586.

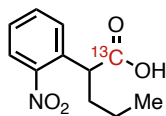


20 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) and 18-crown-6 (1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 115 °C for 18 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 76% yield with 81% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 6.83 (s, 2H), 6.81 (s, 1H), 3.87 (s, 3H), 3.86 (s, 3H), 3.59 (d, J = 7.7 Hz, 2H);

^{13}C NMR (CDCl_3 , 175 MHz) δ 177.7, 149.0, 148.4, 125.7, 121.6, 112.5, 111.3, 55.9 (2 carbon), 40.6 (d, J = 55.1 Hz);

HRMS (ESI): calcd for $\text{C}_9[^{13}\text{C}]\text{H}_{11}\text{O}_4$ [$\text{M}-\text{H}$] $^-$: 196.0696. Found 196.0691.

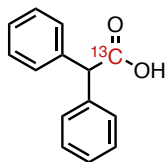


21 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at room temperature for 6 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 76% yield with 93% ^{13}C incorporation.

¹H NMR (CDCl₃, 700 MHz) δ 7.89 (d, *J* = 8.4 Hz, 1H), 7.60 – 7.54 (m, 2H), 7.43 (t, *J* = 7.6 Hz, 1H), 4.24 – 4.20 (m, 1H), 2.17 – 2.13 (m, 1H), 1.86 – 1.83 (m, 1H), 1.40 – 1.25 (m, 2H), 0.92 (t, *J* = 7.5 Hz, 3H);

¹³C NMR (CDCl₃, 125 MHz) δ 177.8, 149.6, 133.1(2), 130.1, 128.3, 124.8, 45.9 (d, *J* = 59.4 Hz), 34.6, 20.9, 13.8;

HRMS (ESI): calcd for C₁₀[¹³C]H₁₃NO₄Na [*M*+Na]⁻: 247.0770. Found 247.0769.

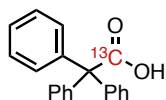


22 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and ¹³CO₂. The reaction mixture was allowed to stir at 90 °C for 2 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 83% yield with 92% ¹³C incorporation.

¹H NMR (CDCl₃, 600 MHz) δ 7.34 – 7.27 (m, 10H), 5.10 (d, *J* = 8.5 Hz, 1H);

¹³C NMR (CDCl₃, 125 MHz) δ 178.1, 138.0, 128.8, 128.7, 127.6, 57.0 (d, *J* = 55.7 Hz);

HRMS (ESI): calcd for C₁₃[¹³C]H₁₂O₂Na [*M*+Na]⁺: 236.0763. Found 236.0762.

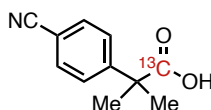


23 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and ¹³CO₂. The reaction mixture was allowed to stir at room temperature for 1.5 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 90% yield with 93% ¹³C incorporation.

¹H NMR (DMSO-d₆, 700 MHz) δ 7.31 – 7.22 (m, 9H), 7.12 – 7.11 (m, 6H);

¹³C NMR (DMSO-d₆, 176 MHz) δ 174.3, 143.2, 129.9, 127.6, 126.6, 66.8 (d, *J* = 54.4 Hz);

HRMS (ESI): calcd for C₁₉[¹³C]H₁₆O₂Na [*M*+Na]⁺: 312.1076. Found 312.1077.

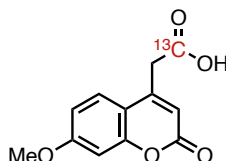


24 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 85 °C for 1 hour. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 96% yield with 94% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.64 (d, J = 8.6 Hz, 2H), 7.51 (d, J = 8.6 Hz, 2H), 1.61 (s, 6H);

^{13}C NMR (CDCl_3 , 175 MHz) δ 181.7, 149.1, 132.3, 126.9, 118.6, 111.1, 46.8 (d, J = 54.0 Hz), 26.1;

HRMS (ESI): calcd for $\text{C}_{10}[^{13}\text{C}]\text{H}_{11}\text{NO}_2\text{Na}$ $[\text{M}+\text{Na}]^+$: 213.0716. Found 213.0712.

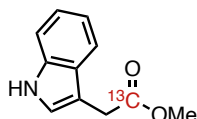


25 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at room temperature for 20 minutes. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 83% yield with 27% ^{13}C incorporation.

^1H NMR (DMSO, 600 MHz) δ 7.62 (d, J = 8.8 Hz, 1H), 7.01 (d, J = 2.6 Hz, 1H), 6.96 (dd, J = 8.8 Hz, 2.6 Hz, 1H), 6.29 (s, 1H), 3.86 (d, J = 7.2 Hz, 2H), 3.85 (s, 3H);

^{13}C NMR (DMSO, 150 MHz) δ 171.0, 162.8, 160.5, 155.4, 150.5, 127.0, 113.3, 112.9, 112.6, 101.3, 56.4, 37.7;

HRMS (ESI): calcd for $\text{C}_{11}[^{13}\text{C}]\text{H}_{10}\text{O}_5\text{Na}$ $[\text{M}+\text{Na}]^+$: 257.0464. Found 257.0462.



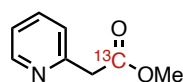
26 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 140 °C for 60 hours. Then MeI (1 equiv.) was added to this solution at room temperature and stirred for 1 hour. After purification through a series of extractions and column chromatography, the

desired product was obtained in 95% yield with 18% ^{13}C incorporation. When the reaction mixture was stirred at 130 °C for 25 hours in the presence of 18-crown-6 (1 equiv.), the desired product was obtained in 64% NMR yield with 65% ^{13}C incorporation.

^1H NMR (CDCl_3 , 600 MHz) δ 8.07 (s, 1H), 7.62 (d, J = 8.1 Hz, 1H), 7.36 (d, J = 8.1 Hz, 1H), 7.13 – 7.22 (m, 3H), 3.80 (d, J = 7.7 Hz, 2H), 3.70 (t, J = 2.1 Hz, 3H);

^{13}C NMR (CDCl_3 , 150 MHz) δ 172.5, 136.2, 127.3, 123.1, 122.3, 119.8, 118.9, 111.2, 108.6, 52.0, 31.2;

HRMS (ESI): calcd for $\text{C}_{10}[^{13}\text{C}]\text{H}_{11}\text{NO}_2\text{Na}$ $[\text{M}+\text{Na}]^-$: 213.0716. Found 213.0716.

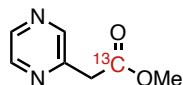


27 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 50 °C for 16 hours. Then the solution was cooled down to room temperature. MeI (1 equiv.) was added to this solution and stirred for 1 hour. After purification through a series of extractions and column chromatography, the desired product was obtained in 80% yield with 48% ^{13}C incorporation.

^1H NMR (CDCl_3 , 500 MHz) δ 8.57 (d, J = 4.8 Hz, 1H), 7.66 (td, J = 7.7 Hz, 1.8 Hz, 1H), 7.29 (d, J = 7.9 Hz, 1H), 7.19 (dd, J = 7.9 Hz, 15.3 Hz, 1H), 3.86 (d, J = 8.2 Hz, 2H), 3.73 (s, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 171.1, 154.4, 149.6, 136.7, 123.9, 122.2, 52.2, 43.8 (d, J = 57.4 Hz);

HRMS (ESI): calcd for $\text{C}_7[^{13}\text{C}]\text{H}_{10}\text{NO}_2$ $[\text{M}+\text{H}]^+$: 153.0740. Found 153.0738.

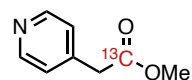


28 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 50 °C for 16 hours. Then the solution was cooled down to room temperature. MeI (1 equiv.) was added to this solution and stirred for 1 hour. After purification through a series of extractions and column chromatography, the desired product was obtained in 68% yield with 29% ^{13}C incorporation.

¹H NMR (CDCl₃, 700 MHz) δ 8.61 (s, 1H), 8.54 – 8.53 (m, 1H), 8.50 (d, *J* = 2.8 Hz, 1H), 3.89 (d, *J* = 8.3 Hz, 2H), 3.75 (t, *J* = 1.8 Hz, 3H);

¹³C NMR (CDCl₃, 175 MHz) δ 170.1, 150.3, 145.3, 144.2, 143.3, 52.4, 41.1 (d, *J* = 57.8 Hz);

HRMS (ESI): calcd for C₆[¹³C]H₉N₂O₂ [M+H]⁺: 154.0692. Found 154.0696.

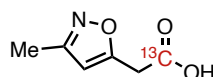


29 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and ¹³CO₂. The reaction mixture was allowed to stir at room temperature for 15 hours. Then MeI (1 equiv.) was added to this solution and stirred for 1 hour. After purification through a series of extractions and column chromatography, the desired product was obtained in 69% yield with 41% ¹³C incorporation.

¹H NMR (CDCl₃, 700 MHz) δ 8.56 (dd, *J* = 4.5 Hz, 1.7 Hz, 2H), 7.22 (dd, *J* = 4.5 Hz, 1.7 Hz, 2H), 3.72 (t, *J* = 1.4 Hz, 3H), 3.63 (d, *J* = 7.7 Hz, 2H);

¹³C NMR (CDCl₃, 125 MHz) δ 170.5, 150.0, 142.7, 124.5, 52.3, 40.5 (d, *J* = 57.8 Hz);

HRMS (ESI): calcd for C₇[¹³C]H₁₀NO₂ [M+H]⁺: 153.0740. Found 153.0740.

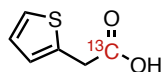


30 Prepared according to General Procedure B from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and ¹³CO₂. The reaction mixture was allowed to stir at 40 °C for 24 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 49% yield (99% NMR yield) with 56% ¹³C incorporation.

¹H NMR (CDCl₃, 700 MHz) δ 6.13 (s, 1H), 3.84 (d, *J* = 7.9 Hz, 2H), 2.29 (s, 3H);

¹³C NMR (CDCl₃, 125 MHz) δ 172.6, 164.3, 160.3, 104.6, 32.5 (d, *J* = 58.8 Hz), 11.6;

HRMS (ESI): calcd for C₅[¹³C]H₇NO₃ [M+H]⁺: 143.0529. Found 143.0530.



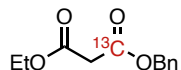
31 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and ¹³CO₂. The reaction mixture was allowed to stir at 100 °C for 20

hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 70% NMR yield with 89% ^{13}C incorporation.

^1H NMR (CDCl_3 , 400 MHz) δ 7.24 (dd, J = 4.7 Hz, 1.9 Hz, 1H), 6.98 – 6.97 (m, 2H), 3.88 (d, J = 7.8 Hz, 2H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 175.9, 134.1, 127.3, 126.9, 125.5, 35.0 (d, J = 55.1 Hz);

HRMS (ESI): calcd for $\text{C}_5[^{13}\text{C}]\text{H}_5\text{SO}_2$ $[\text{M}-\text{H}]^-$: 142.0049. Found 142.0050.

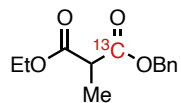


32 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 40 °C for 21 hours. Then the solution was cooled down to room temperature. BnBr (1 equiv.) was added to this solution and stirred for 2 hours. After purification through a series of extractions and column chromatography, the desired product was obtained in 82% yield with 37% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.38 – 7.33 (m, 5H), 5.19 (t, J = 1.8 Hz, 2H), 4.19 (q, J = 7.2 Hz, 2H), 3.42 (d, J = 7.6 Hz, 2H), 1.25 (t, J = 7.2 Hz, 3H);

^{13}C NMR (CDCl_3 , 175 MHz) δ 166.5, 166.4, 135.3, 128.6, 128.4, 128.3, 67.2, 61.6, 41.6 (d, J = 59.2 Hz), 14.0;

HRMS (ESI): calcd for $\text{C}_{11}[^{13}\text{C}]\text{H}_{14}\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$: 246.0818. Found 246.0818.

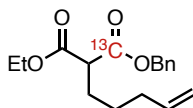


33 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 60 °C for 21 hours. Then the mixture was cooled down to room temperature. BnBr (1 equiv.) was added to this solution and stirred for 2 hours. After purification through a series of extractions and column chromatography, the desired product was obtained in 75% yield with 89% ^{13}C incorporation.

^1H NMR (CDCl_3 , 500 MHz) δ 7.38 – 7.31 (m, 5H), 5.22 – 5.15 (m, 2H), 4.21 – 4.12 (m, 2H), 3.51 – 3.45 (m, 1H), 1.45 – 1.43 (m, 3H), 1.21 (t, J = 7.2 Hz, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 170.2, 170.0, 135.6, 128.6, 128.3, 128.1, 70.0, 61.4, 46.3 (d, J = 58.1 Hz), 14.0, 13.6;

HRMS (ESI): calcd for $\text{C}_{12}[^{13}\text{C}]\text{H}_{16}\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$: 260.0974. Found 260.0974.

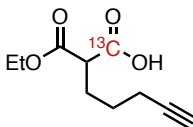


34 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 60 °C for 22 hours. Then the mixture was cooled down to room temperature. BnBr (1 equiv.) was added to this solution and stirred for 2 hours. After purification through a series of extractions and column chromatography, the desired product was obtained in 80% yield with 94% ^{13}C incorporation.

^1H NMR (CDCl_3 , 600 MHz) δ 7.36 – 7.32 (m, 5H), 5.78 – 5.72 (m, 1H), 5.20 – 5.15 (m, 2H), 5.02 – 4.94 (m, 2H), 4.16 (q, J = 7.8 Hz, 2H), 3.39 – 3.36 (m, 1H), 2.06 (q, J = 6.4 Hz, 2H), 1.94 – 1.90 (m, 2H), 1.44 – 1.39 (m, 2H), 1.20 (t, J = 7.2 Hz, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 169.3(2), 137.9, 135.6, 128.6, 128.4, 128.2, 115.1, 67.0, 61.4, 51.9 (d, J = 57.8 Hz), 33.3, 28.2, 26.6, 14.1;

HRMS (ESI): calcd for $\text{C}_{16}[^{13}\text{C}]\text{H}_{22}\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$: 314.1444. Found 314.1442.

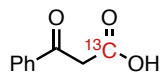


35 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 60 °C for 22 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 71% yield with 87% ^{13}C incorporation.

^1H NMR (CDCl_3 , 600 MHz) δ 4.23 (q, J = 7.2 Hz, 2H), 3.43 – 3.39 (m, 1H), 2.24 (td, J = 6.9, 2.5 Hz, 2H), 2.06 – 2.02 (m, 2H), 1.96 (t, J = 2.7 Hz, 1H), 1.61 (q, J = 8.1 Hz, 2H), 1.29 (t, J = 7.1 Hz, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 174.2, 169.3, 83.3, 69.1, 61.9, 51.2 (d, J = 55.5 Hz), 27.9, 26.1, 18.1, 14.1;

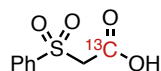
HRMS (ESI): calcd for $\text{C}_9[^{13}\text{C}]\text{H}_{14}\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$: 222.0818. Found 222.0819.



36 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at room temperature for 1 hour. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 70% yield with 89% ^{13}C incorporation. Spectroscopic data agreed with that reported.⁶

^1H NMR (CDCl_3 , 500 MHz) δ 12.4 (brs, 1H, enol form), 7.99 – 7.97 (m, 2H), 7.81 – 7.79 (m, 1H, enol form), 7.67 – 7.64 (m, 1H), 7.54 – 7.50 (m, 2H), 7.44 (m, 2H, enol form), 5.72 (s, 1H, enol form), 4.08 (d, J = 7.0 Hz, 2H);

HRMS (ESI): calcd for $\text{C}_8[^{13}\text{C}]\text{H}_7\text{O}_3$ [$\text{M}-\text{H}$] $^-$: 164.0434. Found 164.0436.

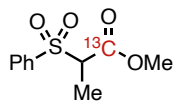


37 Prepared according to General Procedure B from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at room temperature for 2 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 74% yield with 67% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.98 – 7.96 (m, 2H), 7.71 (tt, J = 7.5 Hz, 1.4 Hz, 1H), 7.62 – 7.58 (m, 2H), 4.15 (d, J = 6.3 Hz, 2H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 165.9, 138.6, 134.7, 129.6, 128.7, 60.7 (d, J = 58.2 Hz);

HRMS (ESI): calcd for $\text{C}_7[^{13}\text{C}]\text{H}_8\text{O}_4\text{S}$ [$\text{M}+\text{Na}$] $^+$: 224.0066. Found 224.0070.

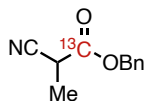


38 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at room temperature for 26 hours. Then MeI (1 equiv.) was added to this solution and stirred for 1 hour. After purification through a series of extractions and column chromatography, the desired product was obtained in 84% yield with 80% ^{13}C incorporation.

¹H NMR (CDCl₃, 600 MHz) δ 7.89 – 7.87 (m, 2H), 7.70 – 7.67 (m, 1H), 7.59 – 7.56 (m, 2H), 4.10 – 4.03 (m, 1H), 3.68 (t, *J* = 1.8 Hz, 3H), 1.58 – 1.55 (m, 3H);

¹³C NMR (CDCl₃, 150 MHz) δ 166.7, 136.9, 134.3, 129.4, 129.1, 65.4 (d, *J* = 60.0 Hz), 53.0, 11.9;

HRMS (ESI): calcd for C₉[¹³C]H₁₂SO₄Na [M+Na]⁺: 252.0382. Found 252.0381.

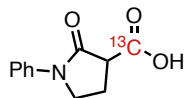


39 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and ¹³CO₂. The reaction mixture was allowed to stir at 60 °C for 18 hours. Then the mixture was cooled down to room temperature. BnBr (1 equiv.) was added to this solution and stirred for 2 hours. After purification through a series of extractions and column chromatography, the desired product was obtained in 82% yield with 67% ¹³C incorporation.

¹H NMR (CDCl₃, 700 MHz) δ 7.39 – 7.36 (m, 5H), 5.26 – 5.22 (m, 2H), 3.61 – 3.56 (m, 1H), 1.61 – 1.60 (m, 3H);

¹³C NMR (CDCl₃, 175 MHz) δ 166.4, 134.5, 128.8, 128.7, 128.3, 117.1, 68.3, 31.6 (d, *J* = 59.8 Hz), 15.2;

HRMS (ESI): calcd for C₁₀[¹³C]H₁₁NO₂Na [M+Na]⁺: 213.0716. Found 213.0717.

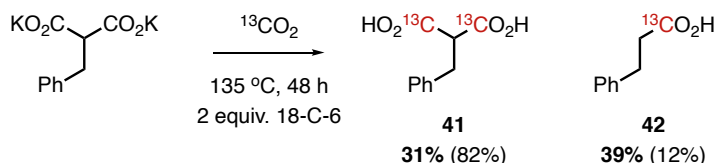


40 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and ¹³CO₂. The reaction mixture was allowed to stir at 40 °C for 28 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 92% yield with 61% ¹³C incorporation.

¹H NMR (CDCl₃, 500 MHz) δ 7.57 (d, *J* = 8.0 Hz, 2H), 7.43-7.40 (m, 2H), 7.25 – 7.23 (m, 1H), 3.97 – 3.90 (m, 2H), 3.68 – 3.63 (m, 1H), 2.60 – 2.48 (m, 2H);

¹³C NMR (CDCl₃, 175 MHz) δ 171.2, 169.4, 137.9, 129.2, 126.1, 120.6, 47.6 (d, *J* = 55.0 Hz), 47.1, 29.7, 20.9;

HRMS (ESI): calcd for C₁₀[¹³C]H₁₁NO₃Na [M+Na]⁺: 229.0665. Found 229.0666.

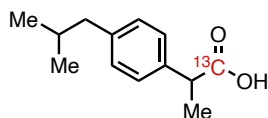


41 and **42** Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) and 18-crown-6 (2 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 135 °C for 48 hours. After purification through a series of extractions as described in General Procedure A, the desired products **41** and **42** were obtained [**41**: 82% NMR yield with 31% ^{13}C incorporation (19% *di*- ^{13}C incorporation and 12% *mono*- ^{13}C incorporation); **42**: 12% NMR yield with 39% ^{13}C incorporation].

^1H NMR (CD_3OD , 500 MHz) δ Product **41**: 7.46 – 7.34 (m, 5H), 3.83 – 3.77 (m, 1H), 3.33 (d, J = 7.7 Hz, 2H); Product **42**: 7.46 – 7.34 (m, 5H), 3.22 – 3.06 (m, 2H), 2.80 – 2.74 (m, 2H);

^{13}C NMR (CD_3OD , 175 MHz) δ Product **41**: 172.7, 139.9, 129.9, 129.5, 127.7, 54.8 (m), 35.3; Product **42**: Only see the incorporated carbon from carboxylic acid due to trace of product **42**, δ : 176.8 (^{13}C);

HRMS (ESI): 41: *di*- ^{13}C incorporation product: calcd for $\text{C}_8[^{13}\text{C}]_2\text{H}_9\text{O}_4$ [M-H] $^-$: 195.0573. Found 195.0570. *mono*- ^{13}C incorporation product: calcd for $\text{C}_9[^{13}\text{C}]\text{H}_9\text{O}_4$ [M-H] $^-$: 194.0540. Found 194.0539.

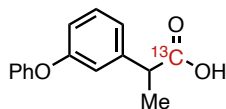


43 Prepared according to General Procedure B from Ibuprofen potassium salt (0.1 mmol, 1 equiv.) in DMSO and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 160 °C for 48 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 71% yield (89% NMR yield) with 80% ^{13}C incorporation. When the reaction mixture was stirred at 130 °C for 36 hours in the presence of 18-crown-6 (1 equiv.), the desired product was obtained in 93% NMR yield with 45% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.23 – 7.21 (m, 2H), 7.11 – 7.09 (m, 2H), 3.71 (quintet, J = 7.3 Hz, 1H), 2.45 (d, J = 7.2 Hz, 2H), 1.84 (dq, J = 13.5, 6.8 Hz, 1H), 1.50 (td, J = 4.9 Hz, 2.3 Hz, 3H), 0.90 (d, J = 6.6 Hz, 6H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 180.4, 141.0, 137.1 (d, J = 2.5 Hz), 129.5, 127.4 (d, J = 1.8 Hz), 45.2, 45.0 (d, J = 54.5 Hz), 30.3, 22.5, 18.3 (d, J = 1.1 Hz);

HRMS (ESI): calcd for $\text{C}_{12}[^{13}\text{C}]\text{H}_{18}\text{O}_2$ [M-H] $^-$: 206.1268. Found 206.1262.

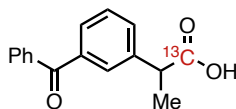


44 Prepared according to General Procedure B from Fenoprofen potassium salt (0.1 mmol, 1 equiv.) in DMSO and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 160 °C for 48 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 80% yield (92% NMR yield) with 92% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.35 – 7.32 (m, 2H), 7.28 (t, J = 7.9 Hz, 1H), 7.12 – 7.10 (m, 1H), 7.06 (d, J = 7.7 Hz, 1H), 7.02 – 7.00 (m, 3H), 6.89 (dd, J = 8.1 Hz, 2.3 Hz, 1H), 3.72 (quintet, J = 7.3 Hz, 1H), 1.52 – 1.50 (m, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 196.6, 179.7, 157.6, 157.1, 130.0, 129.9, 123.5, 122.5 (d, J = 1.8 Hz), 119.1, 118.4 (d, J = 2.0 Hz), 117.6, 45.3 (d, J = 54.6 Hz), 18.2 (d, J = 1.3 Hz);

HRMS (ESI): calcd for $\text{C}_{14}[^{13}\text{C}]\text{H}_{14}\text{O}_3$ $[\text{M}+\text{H}]^+$: 244.1050. Found 244.1044.

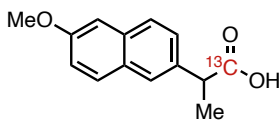


45 Prepared according to General Procedure B from Ketoprofen potassium salt (0.1 mmol, 1 equiv.) in DMSO and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 160 °C for 48 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 75% yield (85% NMR yield) with 93% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.80 – 7.78 (m, 3H), 7.69 (dd, J = 6.5 Hz, 1.2 Hz, 1H), 7.60 – 7.56 (m, 2H), 7.47 (dt, J = 18.7 Hz, 7.7 Hz, 3H), 3.84 (quintet, J = 7.3 Hz, 1H), 1.57 – 1.55 (m, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 196.6, 179.9, 140.3 (d, J = 2.3 Hz), 138.1, 137.6, 132.7, 131.8 (d, J = 1.7 Hz), 130.2, 129.5 (d, J = 2.0 Hz), 129.4, 128.7, 128.4, 45.3 (d, J = 54.6 Hz), 18.3 (d, J = 1.3 Hz);

HRMS (ESI): calcd for $\text{C}_{15}[^{13}\text{C}]\text{H}_{14}\text{O}_3$ $[\text{M}+\text{H}]^+$: 256.1050. Found 256.1040.



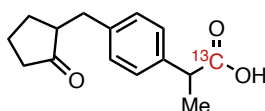
46 Prepared according to General Procedure B from (+)-Naproxen potassium salt (0.1 mmol, 1 equiv.) in DMSO and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 160 °C for 48 hours.

After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 79% yield (94% NMR yield) with 86% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.71 – 7.68 (m, 3H), 7.41 (dd, J = 8.5 Hz, 1.8 Hz, 1H), 7.14 (dd, J = 8.9 Hz, 2.5 Hz, 1H), 7.11 (d, J = 2.4 Hz, 1H), 3.91 (s, 3H), 3.88 (quintet, J = 7.3 Hz, 1H), 1.60 – 1.58 (m, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 180.2, 157.9, 135.0 (d, J = 2.7 Hz), 134.0, 129.4, 129.0, 127.4, 126.32, 126.28 (d, J = 2.2 Hz), 119.2, 105.8, 55.5, 45.3 (d, J = 54.6 Hz), 18.3;

HRMS (ESI): calcd for $\text{C}_{13}[^{13}\text{C}]\text{H}_{14}\text{O}_3$ $[\text{M}+\text{H}]^+$: 232.1050. Found 232.1041.

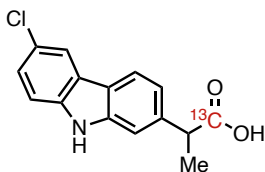


47 Prepared according to General Procedure B from Loxoprofen sodium salt (0.1 mmol, 1 equiv.) in DMSO and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 160 °C for 48 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 75% yield (92% NMR yield) with 30% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.24 – 7.23 (m, 2H), 7.14 – 7.12 (m, 2H), 3.72 (quintet, J = 7.3 Hz, 1H), 3.12 (dd, J = 14.0 Hz, 4.2 Hz, 1H), 2.51 (dd, J = 14.0 Hz, 9.5 Hz, 1H), 2.36 – 2.31 (m, 2H), 2.14 – 2.07 (m, 2H), 1.98 – 1.93 (m, 1H), 1.76 – 1.69 (m, 1H), 1.57 – 1.51 (m, 1H), 1.51 – 1.49 (m, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 220.4, 179.9, 139.3, 137.9, 129.3, 127.8, 51.1, 45.0 (d, J = 54.6 Hz), 38.3, 35.3, 29.4, 20.7, 18.3;

HRMS (ESI): calcd for $\text{C}_{14}[^{13}\text{C}]\text{H}_{18}\text{O}_3$ $[\text{M}+\text{H}]^+$: 265.1627. Found 265.1625.

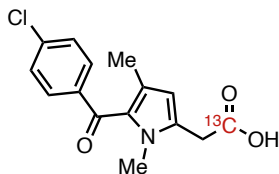


48 Prepared according to General Procedure B from Carprofen potassium salt (0.1 mmol, 1 equiv.) in DMSO and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 160 °C for 24 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 44% yield (62% NMR yield) with 74% ^{13}C incorporation.

¹H NMR (CDCl₃, 700 MHz) δ 8.05 (s, 1H), 7.99 (d, *J* = 1.9 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.39 (s, 1H), 7.36 – 7.32 (m, 2H), 7.20 (dd, *J* = 8.0, 1.3 Hz, 1H), 3.93 – 3.89 (m, 1H), 1.61 – 1.58 (m, 3H);

¹³C NMR (CDCl₃, 125 MHz) δ 178.7, 140.4, 138.7 (d, *J* = 2.5 Hz), 138.2, 126.1, 125.2, 124.5, 122.0, 120.9, 120.2, 119.9 (d, *J* = 0.9 Hz), 111.7, 109.9 (d, *J* = 1.4 Hz), 45.6 (d, *J* = 54.7 Hz), 18.7;

HRMS (ESI): calcd for C₁₄[¹³C]H₁₂NC¹⁸O₂ [M-H]⁻: 273.0516. Found 273.0512.

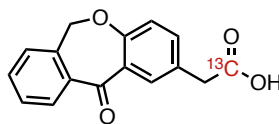


49 Prepared according to General Procedure B from Zomepirac potassium salt (0.1 mmol, 1 equiv.) in DMF and ¹³CO₂. The reaction mixture was allowed to stir at 70 °C for 20 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 76% yield (99% NMR yield) with 67% ¹³C incorporation.

¹H NMR (CDCl₃, 700 MHz) δ 7.66 (d, *J* = 8.4 Hz, 2H), 7.42 (d, *J* = 8.4 Hz, 2H), 5.97 (s, 1H), 3.75 (s, 3H), 3.71 (d, *J* = 7.8 Hz, 2H), 1.75 (s, 3H);

¹³C NMR (CDCl₃, 125 MHz) δ 186.7, 173.4, 139.3, 138.2, 132.4 (d, *J* = 1.9 Hz), 130.7, 130.1, 129.2, 128.8, 112.8, 33.3, 32.3 (d, *J* = 59.4 Hz), 14.6;

HRMS (ESI): calcd for C₁₄[¹³C]H₁₄ClNO₃ [M+H]⁺: 293.0768. Found 293.0753.

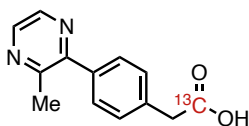


50 Prepared according to General Procedure B from Isoxepac potassium salt (0.1 mmol, 1 equiv.) in DMF and ¹³CO₂. The reaction mixture was allowed to stir at 140 °C for 14 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 89% yield (99% NMR yield) with 91% ¹³C incorporation.

¹H NMR (CDCl₃, 700 MHz) δ 8.13 (d, *J* = 2.4 Hz, 1H), 7.89 (dd, *J* = 7.7 Hz, 1.2 Hz, 1H), 7.56 (td, *J* = 7.4 Hz, 1.3 Hz, 1H), 7.47 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.43 (dd, *J* = 8.4 Hz, 2.4 Hz, 1H), 7.36 (dd, *J* = 7.4 Hz, 0.7 Hz, 1H), 7.04 (d, *J* = 8.4 Hz, 1H), 5.19 (s, 2H), 3.69 (d, *J* = 7.8 Hz, 2H);

¹³C NMR (CDCl₃, 125 MHz) δ 191.0, 177.3, 160.7, 140.5, 136.5, 135.6, 132.93, 132.75 (d, *J* = 2.2 Hz), 129.6, 129.4, 127.9, 127.2 (d, *J* = 2.6 Hz), 125.3, 121.3, 73.8, 40.0 (d, *J* = 55.5 Hz);

HRMS (ESI): calcd for C₁₅[¹³C]H₁₂O₄ [M+H]⁺: 270.0842. Found 270.0838.

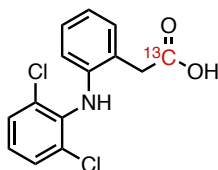


51 Prepared according to General Procedure B from the corresponding carboxylate potassium salt (0.1 mmol, 1 equiv.) in DMF and ¹³CO₂. The reaction mixture was allowed to stir at 120 °C for 15 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 45% yield (56% NMR yield) with 91% ¹³C incorporation.

¹H NMR (CDCl₃, 700 MHz) δ 8.50 (dd, *J* = 2.5 Hz, 0.6 Hz, 1H), 8.47 (d, *J* = 2.5 Hz, 1H), 7.56 – 7.54 (m, 2H), 7.42 (d, *J* = 7.9 Hz, 2H), 3.73 (d, *J* = 7.9 Hz, 2H), 2.65 (s, 3H);

¹³C NMR (CDCl₃, 125 MHz) δ 174.7, 153.7, 152.1, 142.4, 141.5, 137.4, 134.6 (d, *J* = 3.0 Hz), 129.6 (d, *J* = 2.0 Hz), 129.4, 40.9, 23.3 (d, *J* = 55.6 Hz);

HRMS (ESI): calcd for C₁₂[¹³C]H₁₂N₂O₂ [M+H]⁺: 230.1002. Found 230.1000.

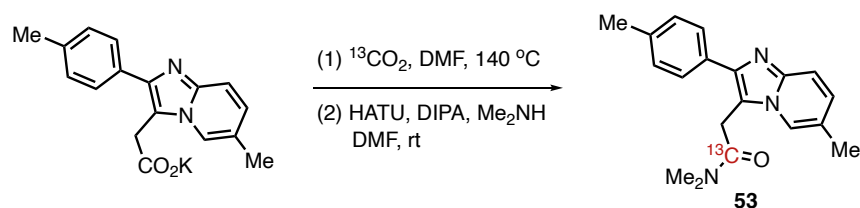


52 Prepared according to General Procedure B from Diclofenac sodium salt (0.1 mmol, 1 equiv.) in DMSO and ¹³CO₂. The reaction mixture was allowed to stir at 140 °C for 48 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 39% yield (50% NMR yield) with 61% ¹³C incorporation.

¹H NMR (CDCl₃, 700 MHz) δ 7.33 (d, *J* = 8.1 Hz, 2H), 7.24 (dd, *J* = 7.5 Hz, 1.1 Hz, 1H), 7.14 (td, *J* = 7.7 Hz, 1.1 Hz, 1H), 6.99 – 6.95 (m, 2H), 6.56 (dd, *J* = 8.1 Hz, 0.9 Hz, 1H), 3.84 (d, *J* = 7.5 Hz, 2H), 2.63 (s, 1H);

¹³C NMR (CDCl₃, 125 MHz) δ 176.1, 142.8, 137.9, 131.1, 129.6, 129.0, 128.3, 124.2, 124.0 (d, *J* = 3.3 Hz), 122.3, 118.6, 38.3 (d, *J* = 55.4 Hz);

HRMS (ESI): calcd for C₁₅[¹³C]H₁₁NCl₂O₂ [M-H]⁻: 295.0126. Found 295.0109.



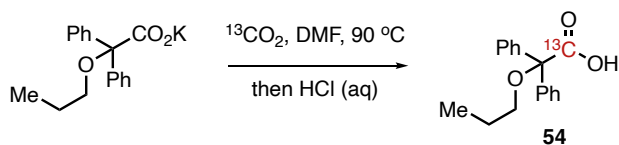
53 Prepared according to General Procedure B from corresponding carboxylate potassium salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 140 °C for 24 hours. Instead of the aqueous work up depicted in General Procedure B, the crude reaction mixture was subjected to a second reaction towards the formation of Zolpidem.

To the reaction solution was added HATU (1.1 equiv.) and the reaction was allowed to stir at a room temperature for 15 minutes. A solution of dimethylamine hydrochloric acid salt (5 equiv.) and *N,N*-diisopropylamine (10 equiv.) in DMF (1 mL) was then added to the reaction mixture, which was allowed to stir for another 3 hours. The reaction was diluted with ethyl acetate (10 mL) and washed with water (10 mL \times 5). The organic layer was dried over anhydrous sodium sulfate and filtered. The volatiles were removed under reduced pressure to afford the crude amide product. Purification by preparative thin-layer chromatography was performed and the desired product was obtained in 43% yield (72% NMR yield) with 82% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 8.01 (dd, J = 1.7 Hz, 0.7 Hz, 1H), 7.55 – 7.52 (m, 3H), 7.26 (dd, J = 8.3 Hz, 0.7 Hz, 2H), 7.04 (dd, J = 9.1 Hz, 1.7 Hz, 1H), 4.09 (d, J = 6.3 Hz, 2H), 2.94 (d, J = 2.8 Hz, 3H), 2.87 (d, J = 3.1 Hz, 3H), 2.40 (s, 3H), 2.34 (d, J = 1.0 Hz, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 168.5, 144.3, 144.0 (d, J = 2.0 Hz), 137.6, 132.0, 129.5, 128.6, 127.6, 122.4, 121.8, 116.8, 113.7 (d, J = 3.2 Hz), 37.7 (d, J = 2.0 Hz), 36.0, 30.5 (d, J = 51.5 Hz), 21.4, 18.6;

HRMS (ESI): calcd for $\text{C}_{18}[^{13}\text{C}]\text{H}_{21}\text{N}_3\text{O}$ $[\text{M}+\text{H}]^+$: 309.1788. Found 309.1784.

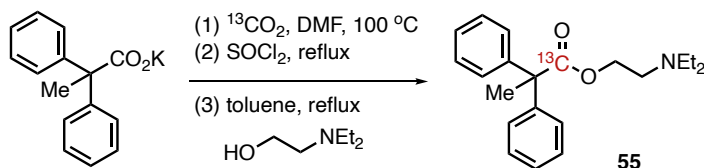


54 Prepared according to General Procedure B from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 90 °C for 3 hours. After purification through a series of extractions as described in General Procedure B, the desired product was obtained in 65% yield with 86% ^{13}C incorporation.

^1H NMR (CDCl_3 , 500 MHz) δ 7.46 – 7.34 (m, 4H), 7.39 – 7.35 (m, 6H), 3.13 (t, J = 6.7 Hz, 2H), 1.65 (sextet, J = 7.2 Hz, 2H), 0.93 (t, J = 7.5 Hz, 3H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 173.0, 138.2, 128.7, 128.6, 128.2, 86.9 (d, J = 57.4 Hz), 67.3, 23.0, 10.6;

HRMS (ESI): calcd for $\text{C}_{16}[^{13}\text{C}]\text{H}_{18}\text{O}_3\text{Na}$ $[\text{M}+\text{Na}]^+$: 294.1182. Found 294.1181.



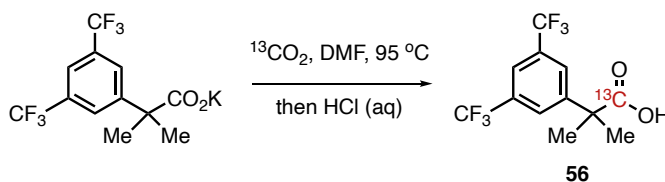
55 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 100 °C for 4 hours. After purification through a series of extractions as described in General Procedure A, the corresponding carboxylic acid was obtained.

A mixture of the corresponding carboxylic acid and SOCl_2 (0.5 mL) was refluxed for 1h. The excess SOCl_2 was removed under reduced pressure, then 2-(diethylamino)ethanol (2 equiv.) and toluene (1 mL) was added and refluxed for 12 hours. After cooling to room temperature, sat. NaHCO_3 solution (5 mL) was added. The aqueous layer was extracted with EtOAc (x3). The combined organic layers were washed with brine, dried over Na_2SO_4 and evaporated under reduced pressure to give the crude mixture. Chromatography on silica gel (10:1 DCM:MeOH) gave Aprophen **55** (the overall yield of 52% with 83% ^{13}C incorporation).

^1H NMR (CDCl_3 , 500 MHz) δ 7.34 – 7.25 (m, 10H), 4.25 (td, J = 6.3 Hz, 2.8 Hz, 2H), 2.68 (t, J = 6.2 Hz, 2H), 2.49 (q, J = 7.2 Hz, 4H), 1.96 (t, J = 2.3 Hz, 3H), 0.97 (t, J = 7.2 Hz, 6H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 175.1, 144.5, 128.1, 128.0, 126.7, 63.7, 56.9 (d, J = 56.5 Hz), 50.9, 47.4, 27.1, 12.0;

HRMS (ESI): calcd for $\text{C}_{20}[^{13}\text{C}]\text{H}_{28}\text{NO}_2$ $[\text{M}+\text{H}]^+$: 327.2148. Found 327.2146.



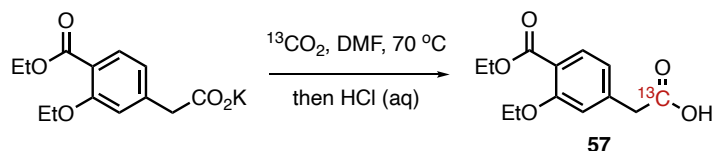
56 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 95 °C for 4 hours.

After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 92% yield with 93% ^{13}C incorporation.

^1H NMR (CDCl_3 , 500 MHz) δ 7.81 (s, 2H), 7.79 (s, 1H), 1.64 (d, J = 4.5 Hz, 6H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 181.4 (d, J = 31.4 Hz), 146.3, 131.7 (q, J = 33.3 Hz), 126.4, 125.4 (q, J = 272.8 Hz), 121.2, 46.6 (d, J = 59.8 Hz), 26.2;

HRMS (ESI): calcd for $\text{C}_{11}[^{13}\text{C}]\text{H}_{10}\text{F}_6\text{O}_2\text{Na}$ $[\text{M}+\text{Na}]^+$: 324.0511. Found 324.0510.

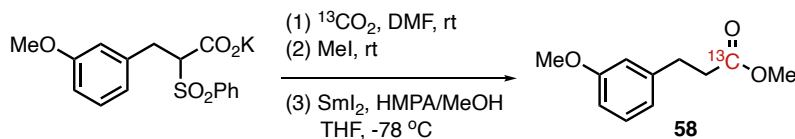


57 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 70 °C for 4 hours. After purification through a series of extractions as described in General Procedure A, the desired product was obtained in 87% yield with 89% ^{13}C incorporation.

^1H NMR (CDCl_3 , 700 MHz) δ 7.72 (d, J = 8.3 Hz, 1H), 6.87 (d, J = 8.3 Hz, 2H), 4.33 (q, J = 7.3 Hz, 2H), 4.08 (q, J = 7.1 Hz, 2H), 3.63 (d, J = 7.7 Hz, 2H), 1.43 (t, J = 7.0 Hz, 3H), 1.35 (t, J = 7.2 Hz, 3H);

^{13}C NMR (CDCl_3 , 175 MHz) δ 176.3, 166.2, 158.6, 138.6, 131.8, 121.1, 120.0, 114.4, 64.7, 60.8, 41.1 (d, J = 55.3 Hz), 14.7, 14.2;

HRMS (ESI): calcd for $\text{C}_{12}[^{13}\text{C}]\text{H}_{17}\text{O}_5$ $[\text{M}+\text{H}]^+$: 254.1104. Found 254.1104.



58 Prepared according to General Procedure A from the corresponding carboxylate salt (0.2 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at room temperature for 19 hours. Then MeI (1 equiv.) was added to this solution and stirred for 1 hour. After purification through a series of extractions and column chromatography, ^{13}C -labeled methyl 2-benzenesulfonyl-3-phenylpropionate was obtained.

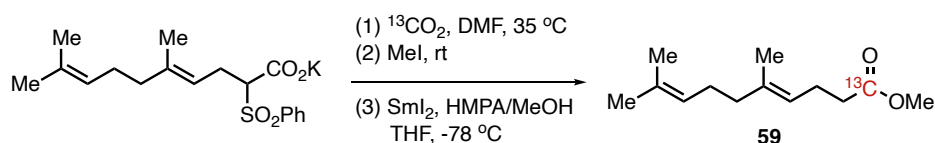
Adapted from the literature,⁷ a solution of SmI_2 (6.25 equiv.; 0.1 M solution in THF) was placed under N_2 atmosphere, and then HMPA (8.0 equiv.) and MeOH (50 equiv.) were added. ^{13}C -labeled methyl 2-benzenesulfonyl-3-phenylpropionate (1.0 equiv.) in THF (0.1 M) was

added at $-78\text{ }^{\circ}\text{C}$. (If the typical deep blue color of SmI_2 faded upon the sulfone solution addition, additional SmI_2 was added until the deep blue color persisted.) The resulting mixture was stirred at $-78\text{ }^{\circ}\text{C}$ for an additional 5 hours. The saturated aq. NH_4Cl was added and the aqueous layer was extracted with EtOAc (x3). The combined organic layers were washed with saturated aq. $\text{Na}_2\text{S}_2\text{O}_3$, brine, dried over MgSO_4 and evaporated under reduced pressure to give the crude mixture. Chromatography on silica gel (10:1 Hexane: EtOAc) gave the title compound **59** (the overall yield of 64% with 66% ^{13}C incorporation).

^1H NMR (CDCl_3 , 400 MHz) δ 7.22 – 7.19 (m, 1H), 6.79 (d, $J = 7.5\text{ Hz}$, 1H), 6.76 – 6.74 (m, 2H), 3.79 (s, 3H), 3.67 (t, $J = 1.9\text{ Hz}$, 3H), 2.94 – 2.91 (m, 2H), 2.65 – 2.61 (m, 2H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 173.3, 159.7, 142.1, 129.5, 120.6, 114.1, 111.6, 55.2, 51.6, 35.6 (d, $J = 57.3\text{ Hz}$), 31.1;

HRMS (ESI): calcd for $\text{C}_{10}[^{13}\text{C}]\text{H}_{14}\text{O}_3$ $[\text{M}]^+$: 195.0977. Found 195.0977.



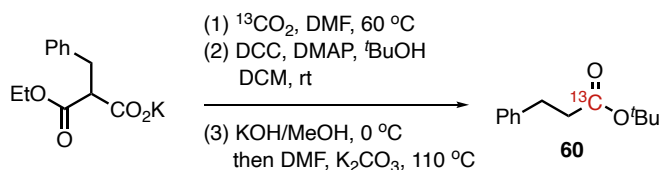
59 Prepared according to General Procedure A from the corresponding carboxylate salt (0.2 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at $35\text{ }^{\circ}\text{C}$ for 25 hours. Then MeI (1 equiv.) was added to this solution and stirred for 1h at rt. After purification through a series of extractions and column chromatography, ^{13}C -labeled sulfone was obtained.

Adapted from the literature,⁷ a solution of SmI_2 (6.25 equiv.; 0.1 M solution in THF) was placed under N_2 atmosphere, then HMPA (8.0 equiv.) and MeOH (50 equiv.) were added. ^{13}C -labeled sulfone (1.0 equiv.) in THF (0.1 M) was added at $-78\text{ }^{\circ}\text{C}$. (If the typical deep blue color of SmI_2 faded upon the sulfone solution addition, additional SmI_2 was added until the deep blue color persisted.) The resulting mixture was stirred at $-78\text{ }^{\circ}\text{C}$ for an additional 5 hours. The saturated aq. NH_4Cl was added and the aqueous layer was extracted with EtOAc (x3). The combined organic layers were washed with saturated aq. $\text{Na}_2\text{S}_2\text{O}_3$, brine, dried over MgSO_4 and evaporated under reduced pressure to give the crude mixture. Chromatography on silica gel (20:1 Hexane: EtOAc) gave the title compound **59** (the overall yield of 74% with 65% ^{13}C incorporation).

^1H NMR (CDCl_3 , 600 MHz) δ 5.11 – 5.05 (m, 2H), 3.66 (t, $J = 1.8\text{ Hz}$, 3H), 2.35 – 2.30 (m, 4H), 2.07 – 2.03 (m, 2H), 1.98 – 1.94 (m, 2H), 1.67 (s, 3H), 1.61 (s, 3H), 1.59 (s, 3H);

^{13}C NMR (CDCl_3 , 150 MHz) δ 173.9, 136.8, 131.5, 124.2, 122.4, 51.5, 39.7, 34.3 (d, $J = 57.6$ Hz), 26.7, 25.7, 23.6, 17.7, 16.1;

HRMS (ESI): calcd for $\text{C}_9[^{13}\text{C}]\text{H}_{15}\text{O}_2$ $[\text{M}]^+$: 168.1106. Found 168.1105.



60 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and $^{13}\text{CO}_2$. The reaction mixture was allowed to stir at 60 °C for 6 hours. After purification through a series of extractions as described in General Procedure A, ^{13}C -labeled 2-benzyl-3-ethoxy-3-oxopropanoic acid was obtained. When the reaction mixture was stirred at 40 °C for 1 hour in the presence of 18-crown-6 (1 equiv.), the desired product was obtained in 95% yield with >95% ^{13}C incorporation.

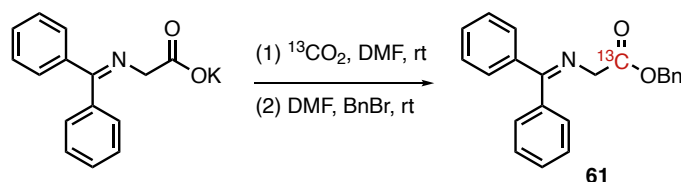
Adapted from the literature,⁸ a solution of the above ^{13}C -labeled carboxylic acid in DCM (0.3 mL) was added to a solution of $t\text{BuOH}$ (1.25 equiv.) and DMAP (0.03 equiv.) in DCM (0.15 mL) at 0 °C. A solution of N,N' -dicyclohexylcarbodiimide (1.3 equiv.) in DCM (0.1 mL) was added. The mixture was stirred for overnight at room temperature and the precipitate formed was filtered off through celite with Et_2O . After removing the solvent under vacuum, the residue was chromatographed on silica gel (10:1 Hexane:EtOAc) to afford ^{13}C -labeled 1-*tert*-butyl 3-ethyl 2-benzylmalonate.

Adapted from the literature,⁹ KOH (1M, 2.0 equiv.) in MeOH solution was added into ^{13}C -labeled 1-*tert*-butyl 3-ethyl 2-benzylmalonate at 0 °C. The reaction was stirred for 3 hours and then the solvent was removed in *vacuo*. The resulting yellowish oils were further dissolved in saturated NaHCO_3 and washed with Et_2O to remove traces of unreacted starting materials. The aqueous layer was then acidified with conc. HCl until approx. $\text{pH}=2$ and extracted with EtOAc (x2). The combined organic phases were washed with brine, dried over Na_2SO_4 , filtered, and concentrated in *vacuo* to give ^{13}C -labeled 2-benzyl-3-(*tert*-butoxy)-3-oxopropanoic acid. Then to a solution of ^{13}C -labeled 2-benzyl-3-(*tert*-butoxy)-3-oxopropanoic acid in DMF (0.5 mL) was added K_2CO_3 (1.0 equiv.) and stirred at 110 °C for 14 hours. The solution was extracted with EtOAc (x3), washed with brine, dried over Na_2SO_4 , filtered, and concentrated. The residue was chromatographed on silica gel (20:1 Hexane:EtOAc) to afford the title compound **60** (the overall yield of 61% with 77% ^{13}C incorporation).

¹H NMR (CDCl₃, 600 MHz) δ 7.29 – 7.27 (m, 2H), 7.21 – 7.18 (m, 3H), 2.92 – 2.89 (tt, *J* = 7.5 Hz, 2.2 Hz, 2H), 2.92 – 2.89 (m, 2H), 1.42 (s, 9H);

¹³C NMR (CDCl₃, 150 MHz) δ 172.4, 140.9, 128.5, 128.4, 126.2, 80.4, 37.2 (d, *J* = 57.1 Hz), 31.2, 28.2;

HRMS (ESI): calcd for C₁₂[¹³C]H₁₈O₂Na [M+Na]⁺: 230.1233. Found 230.1236.

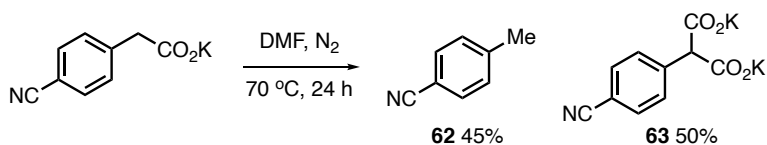


61 Prepared according to General Procedure A from the corresponding carboxylate salt (0.1 mmol, 1 equiv.) in DMF and ¹³CO₂. The reaction mixture was allowed to stir at rt for 24 hours. The ¹³C isotope incorporation was determined to be 93% through analysis of high-resolution mass spectrum of the corresponding carboxylic acid. Then BnBr (1 equiv.) was added to this solution and stirred for 2 hours at room temperature. The mixture was diluted in DCM (10 mL) and sat. NaHCO₃ solution (5 mL). The aqueous layer was extracted with DCM (3 x 10 mL) and the combined organic layers were washed with sat. NaHCO₃ solution (5 mL), dried over anhydrous NaSO₄, filtered, and concentrated *in vacuo* to give the desired product **61** in 76% yield with 93% ¹³C incorporation.

¹H NMR (CDCl₃, 500 MHz) δ 7.66 (d, *J* = 7.6 Hz, 2H), 7.46 – 7.32 (m, 11H), 7.16 (dd, *J* = 7.7 Hz, 3.7 Hz, 2H), 5.19 (d, *J* = 2.7 Hz, 2H), 4.26 (d, *J* = 7.0 Hz, 2H);

¹³C NMR (CDCl₃, 125 MHz) δ 172.1 (q), 170.5, 139.3, 136.0, 135.8, 130.5, 128.8, 128.7 (2), 128.6, 128.3, 128.2, 128.1, 127.7, 65.6, 55.7 (d, *J* = 63.4 Hz);

HRMS (ESI): calcd for C₁₂[¹³C]H₂₀NO₂ [M+H]⁺: 331.1522. Found 331.1520.



In a glovebox filled with N₂, potassium 4-cyanophenylacetate (0.1 mmol, 1.0 equiv.) and anhydrous DMF (0.1 M) were added sequentially to a dried 4-dram vial charged with a stir bar. The vial was sealed with a PTFE-lined cap and removed from the glovebox. The reaction mixture was allowed to stir at 70 °C for 24 hours. The yields were obtained through ¹H NMR spectroscopy.

Compound **62**: ^1H NMR (CDCl_3 , 500 MHz) δ 7.54 (d, J = 8.2 Hz, 2H), 7.27 (d, J = 8.2 Hz, 2H), 2.42 (s, 1H);

^{13}C NMR (CDCl_3 , 125 MHz) δ 143.7, 132.1, 129.9, 119.2, 109.4, 21.9. Spectroscopic data agreed with that reported.¹⁰

Compound **63**: ^1H NMR (D_2O , 500 MHz) δ 7.77 (d, J = 8.2 Hz, 2H), 7.53 (d, J = 8.2 Hz, 2H), 4.57 (s, 1H);

^{13}C NMR (D_2O , 125 MHz) δ 177.9, 145.2, 133.4, 130.8, 121.0, 110.1, 66.2;

HRMS: Mass could not be obtained due to its instability with resulting decarboxylation.

V. Additional Studies and Mechanistic Analysis

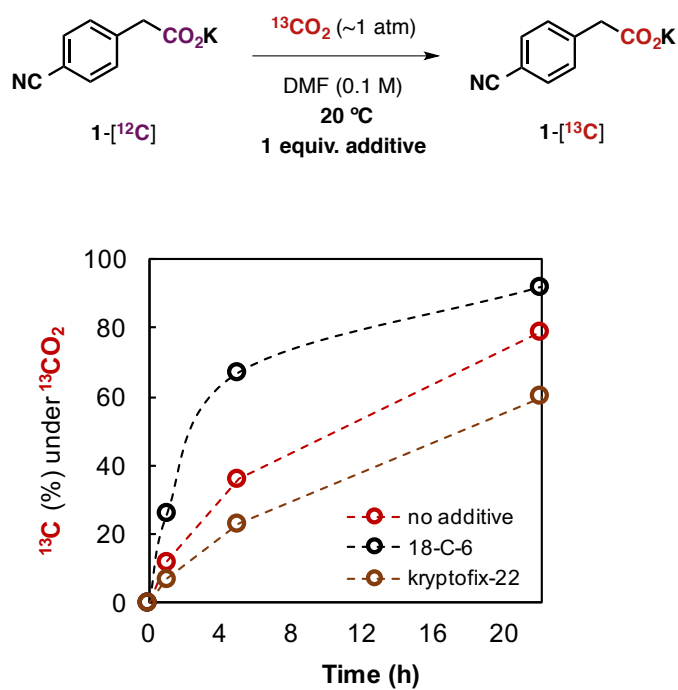


Fig S2. Impact of additives on rate of carboxylate exchange. The addition of 18-C-6 results in an increase in rate while kryptofix-22 slows the rate.

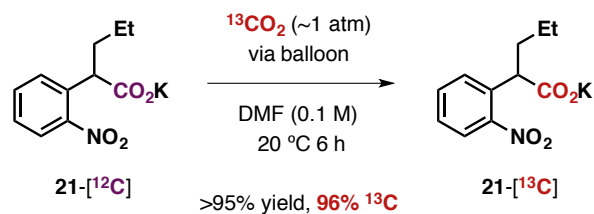


Fig S3. Carboxylate exchange using a large excess of ¹³CO₂ supplied by a balloon results in nearly quantitative ¹³C incorporation.

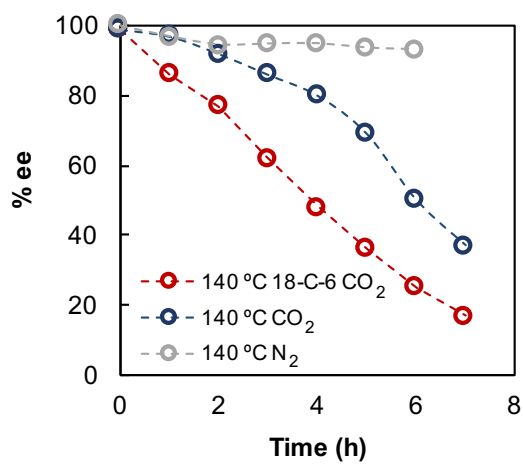
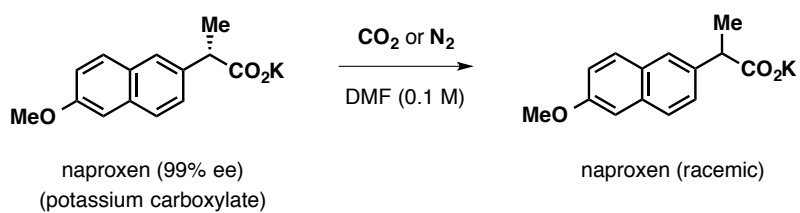


Fig S4. Impact of reaction conditions on the rate of Naproxen racemization. Mass balance is >80% in all cases. The rate of racemization is accelerated under CO₂ and in the presence of 18-C-6.

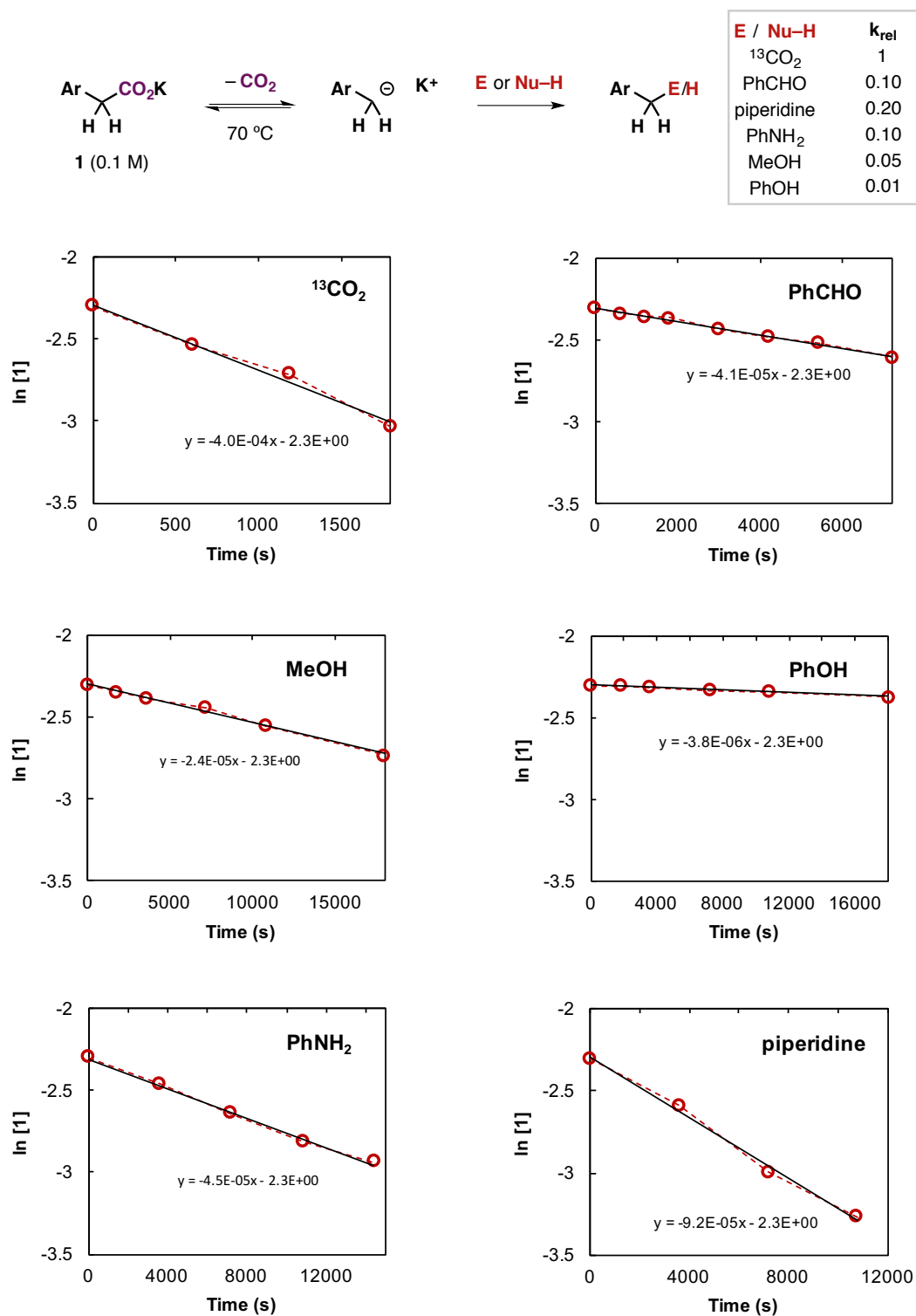


Fig S5. Initial rate comparisons for decarboxylative trapping reactions with electrophiles and Brønsted acids. Initial concentration of ¹³CO₂ ~0.25 M, all other electrophiles or Brønsted acids = 0.5 M. The observation that the weakest acids (or strongest nucleophiles) induce the fastest protodecarboxylation suggest that the rate of nucleophilic trapping of CO₂ by the additive controls the rate of protonation.

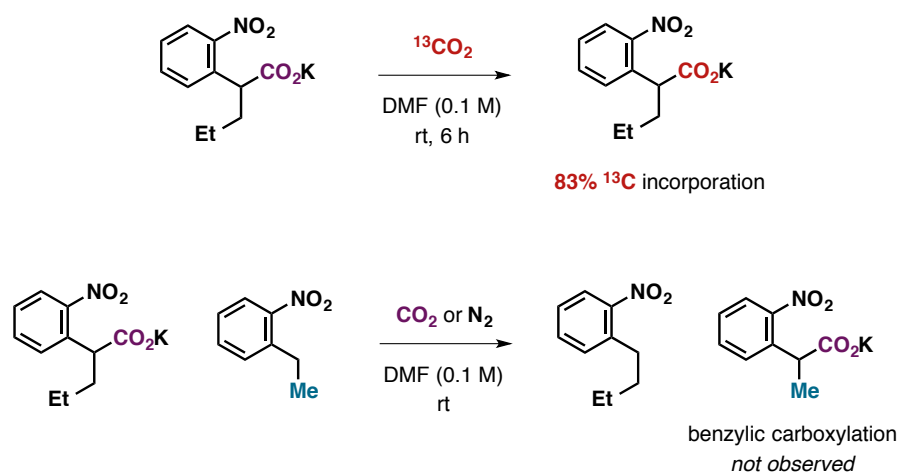
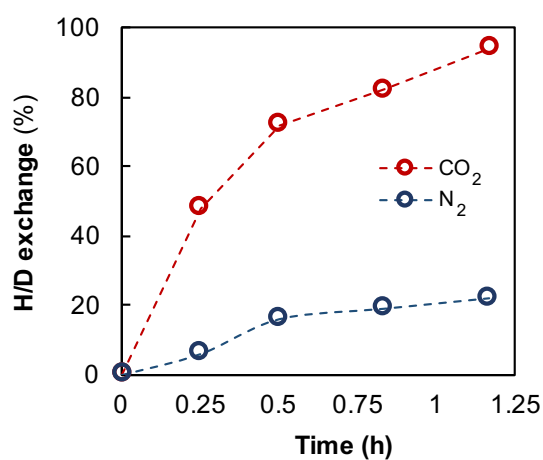
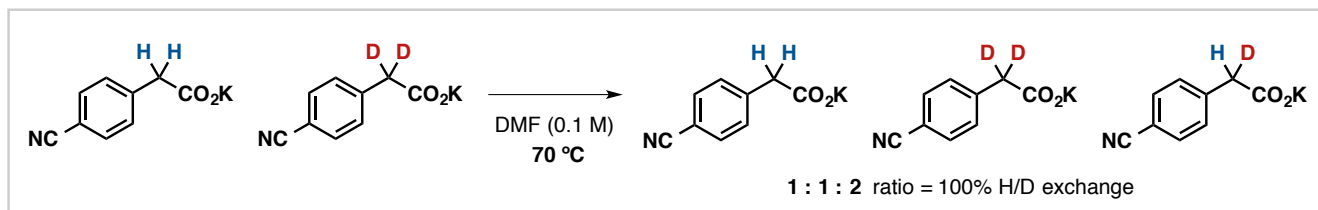


Fig S6. Subjecting alkyl arene to decarboxylation conditions does not result in carboxylation (additional example).



Potential Mechanism

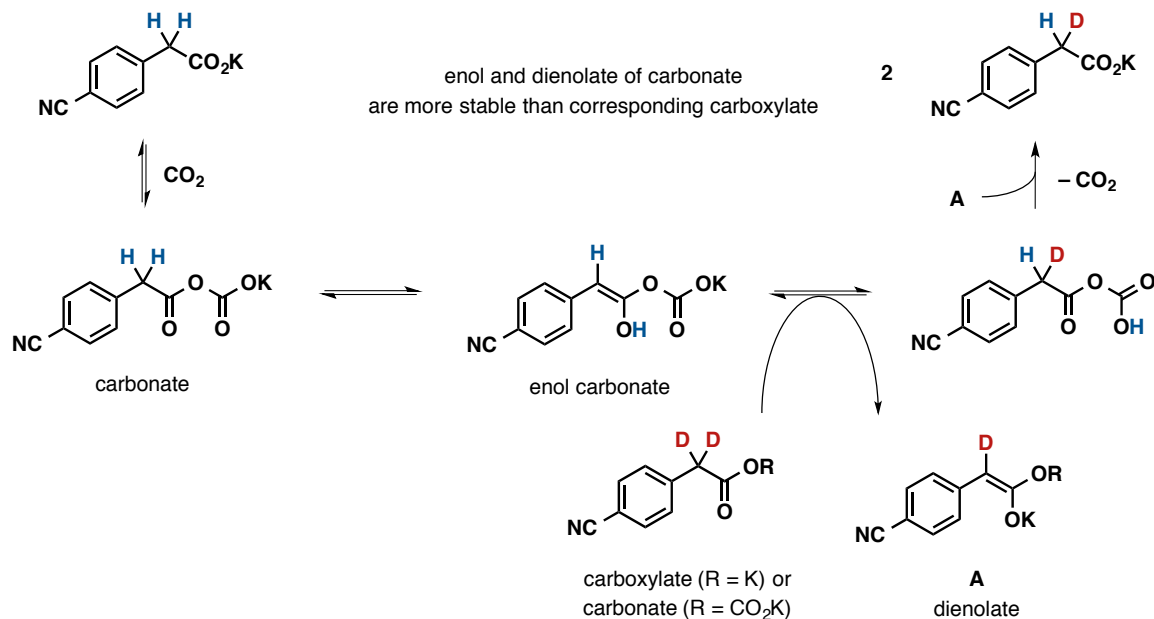
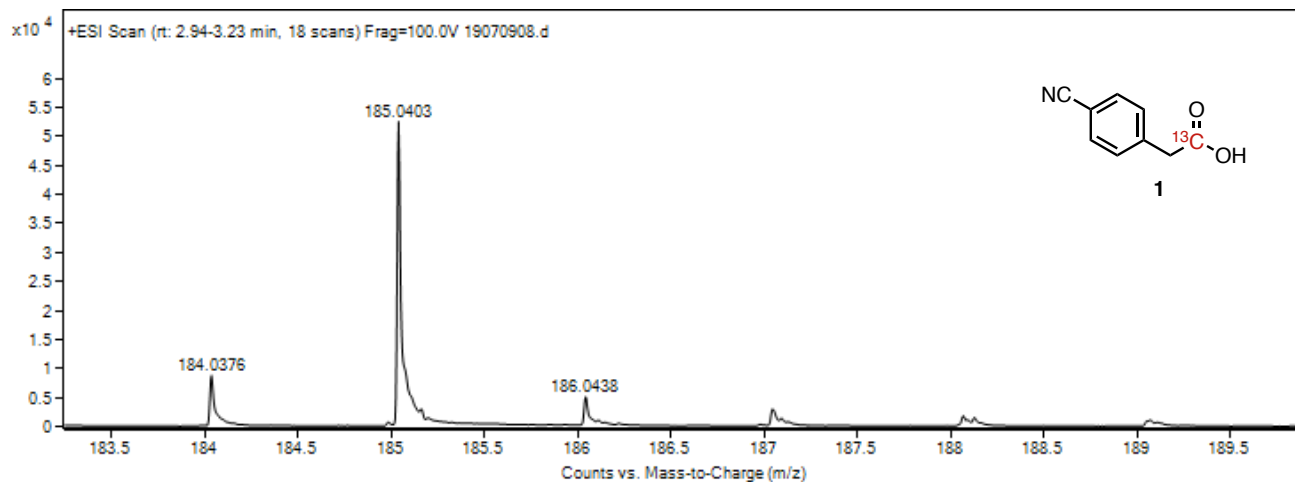


Fig S7. Impact of reaction atmosphere on aryl acetate H/D exchange. Under CO₂ the rate of H/D exchange is increased. The difference in reactivity can be attributed to the generation of a carbonate intermediate capable of intramolecular proton transfer

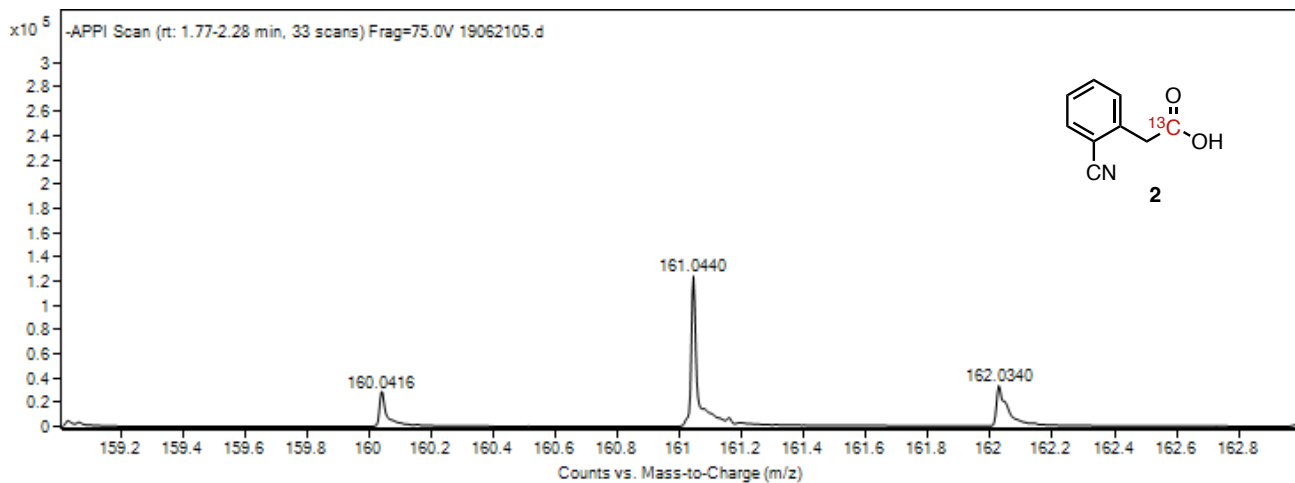
VI. References

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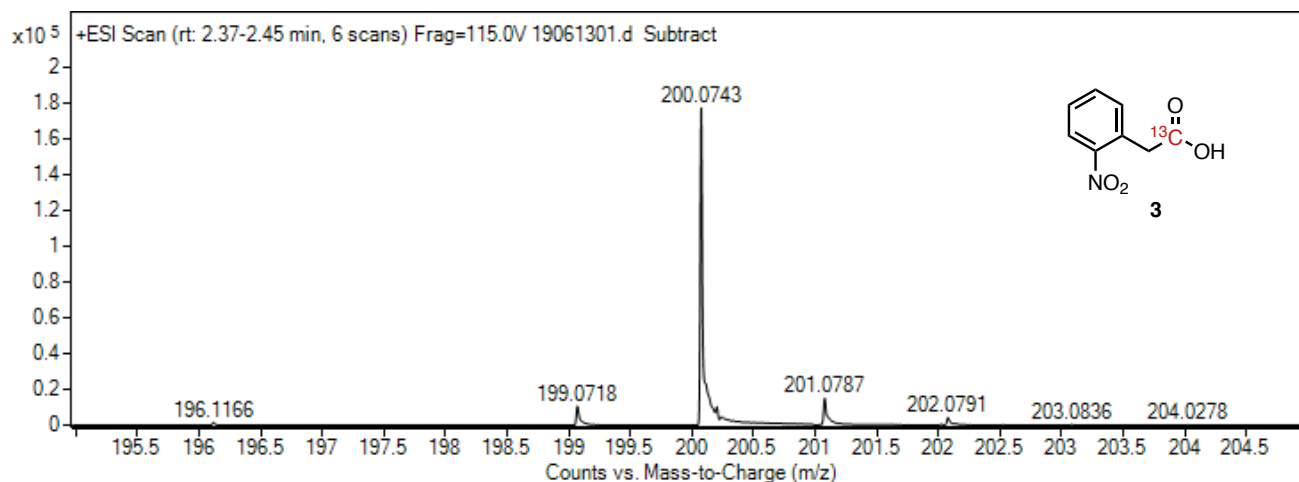
VII. High Resolution Mass Spectrometry Data



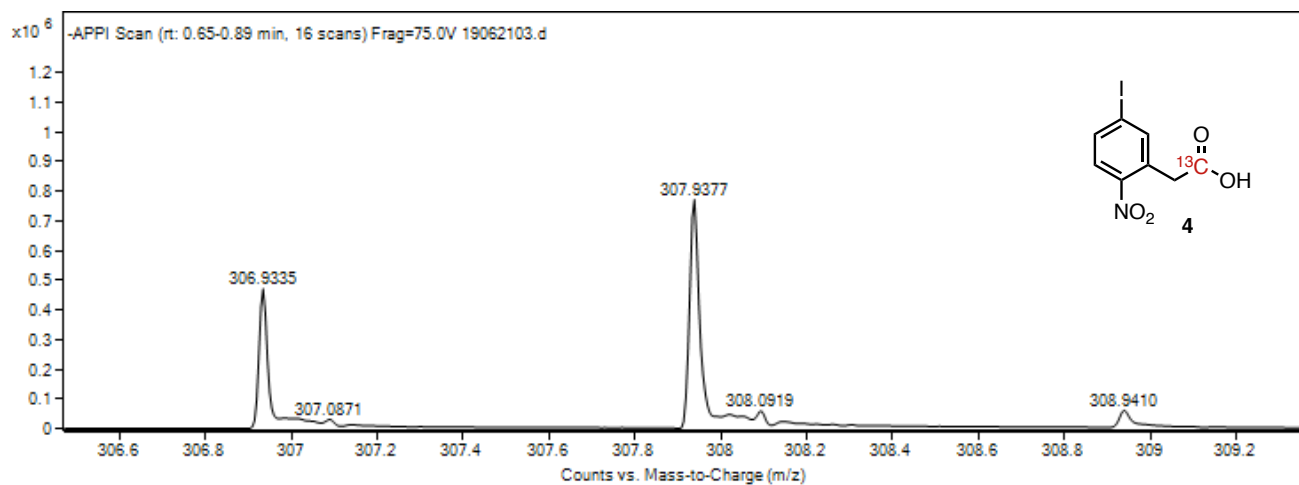
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	8824	8824	15
[M+Na] ⁺ 1	10.26	52675	51770	85



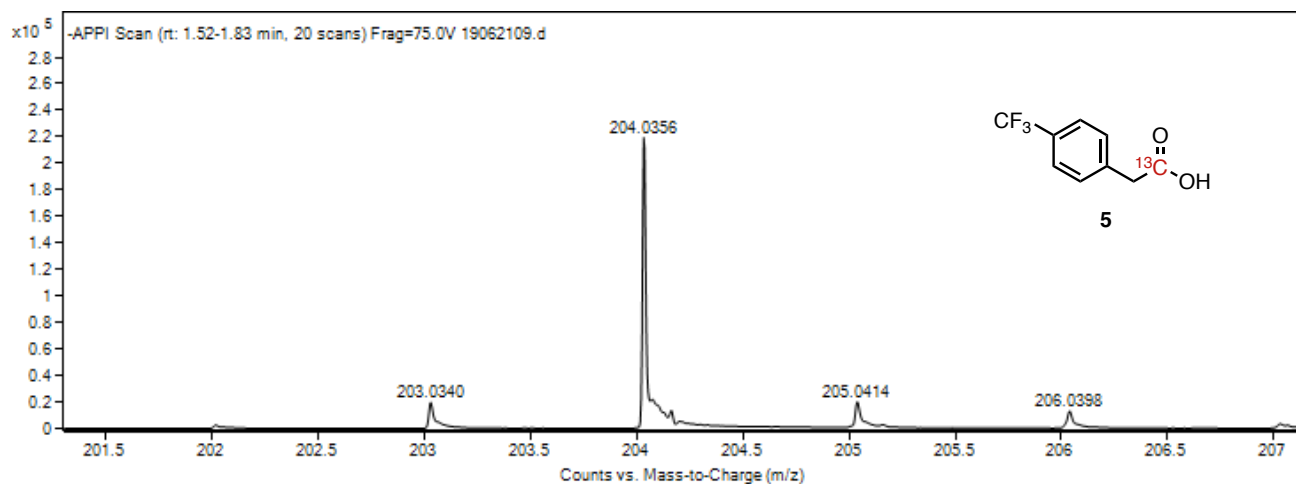
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M-H] ⁺ 0	100	29438	29438	19
[M-H] ⁺ 1	10.24	125243	122229	81



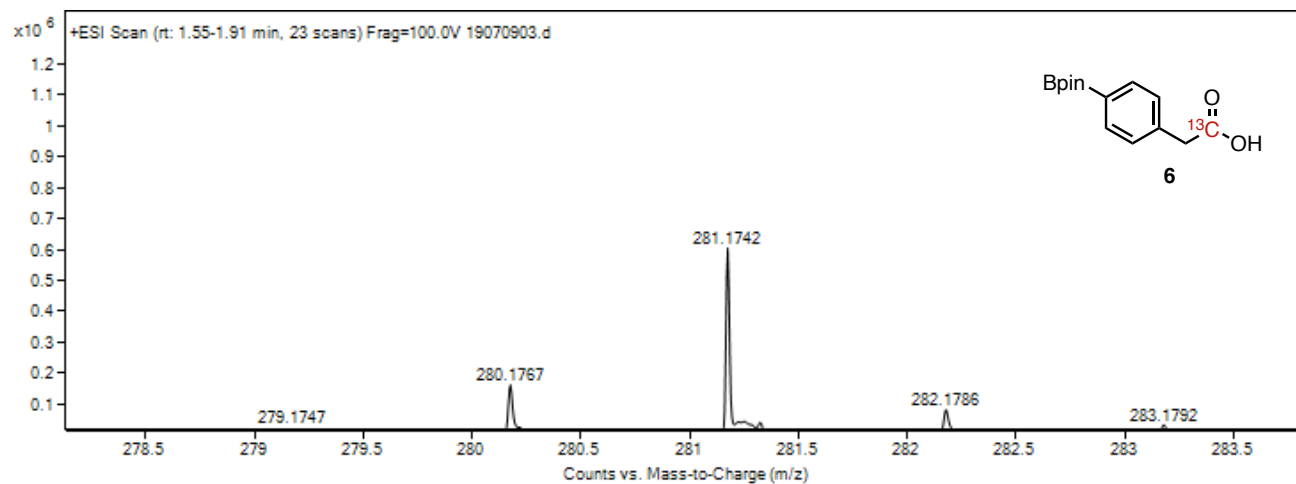
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+NH ₄]+0	100	10924	10924	6
[M+ NH ₄]+1	9.66	182873	181818	94



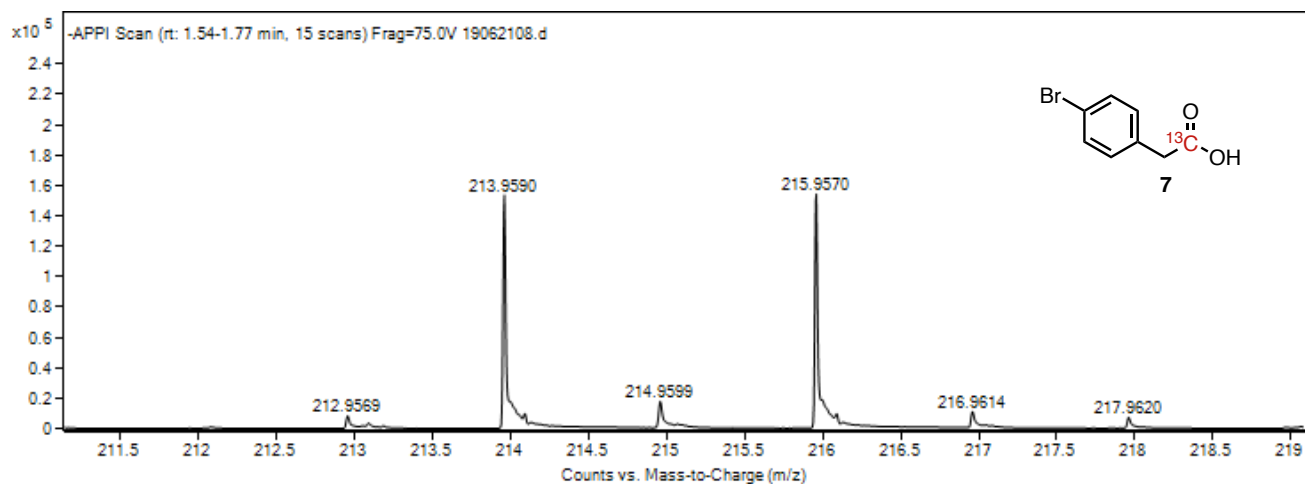
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M-H]+0	100	442051	442051	37
[M-H]+1	9.23	785668	7448672	63



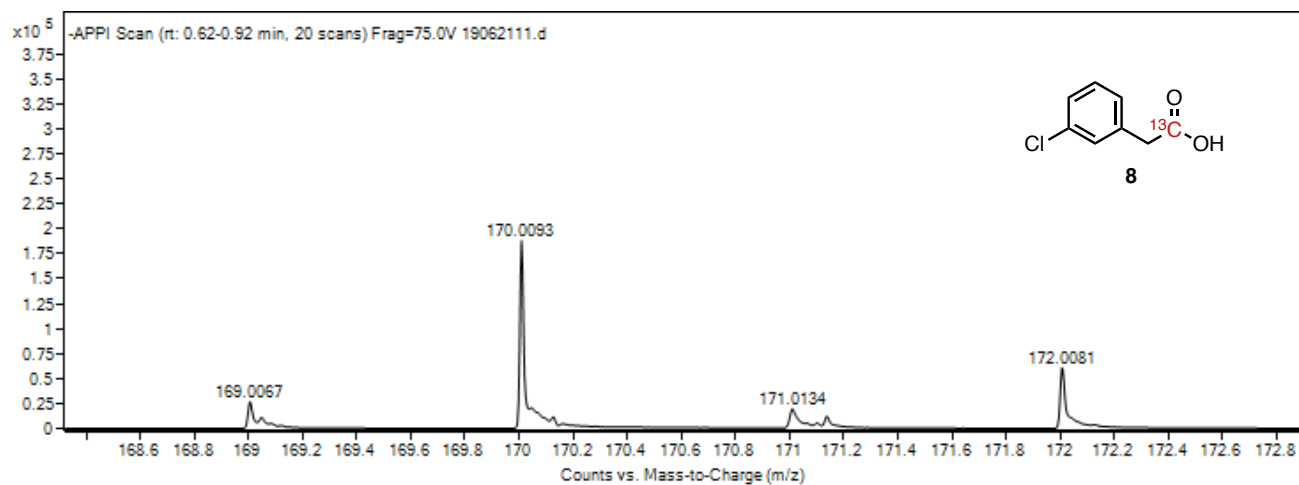
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[M-H] ⁺ 0	100	19909	19909	8
[M-H] ⁺ 1	9.88	224694	222727	92



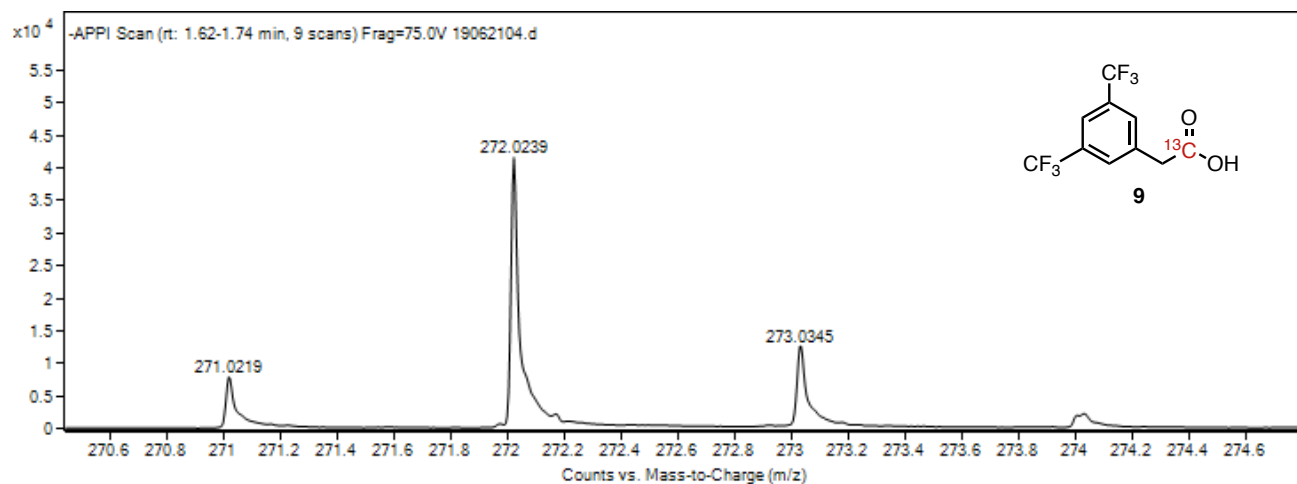
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[M+NH ₄] ⁺ 0	23.9	10310	54521	7
[M+NH ₄] ⁺ 1	100	163130	716067	93
[M+NH ₄] ⁺ 2	15.8	605762	-	-



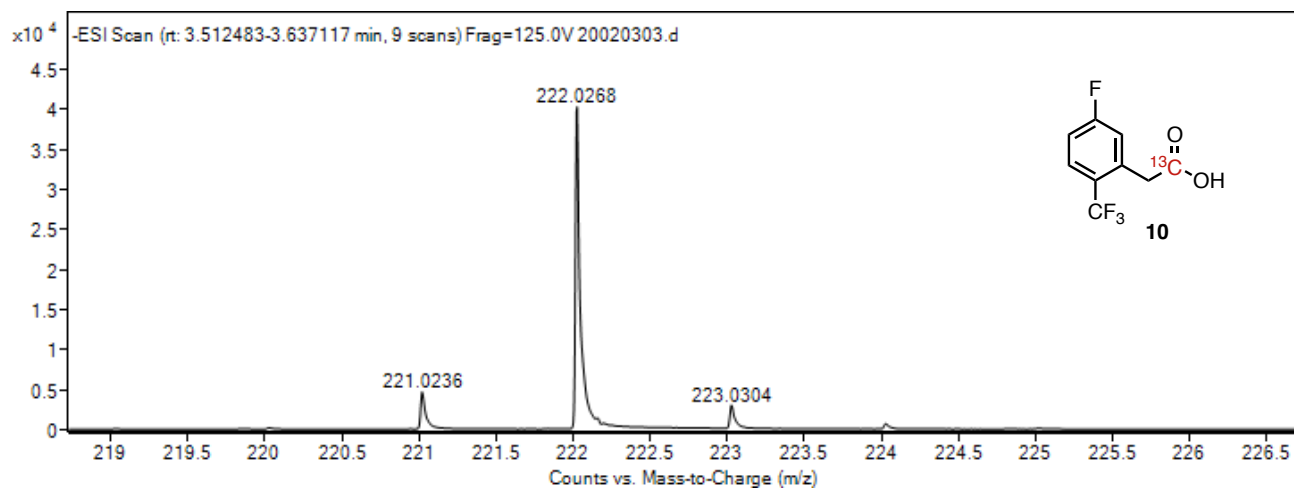
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M-H]+0	100	8426	8426	5
[M-H]+1	8.8	154709	153968	95



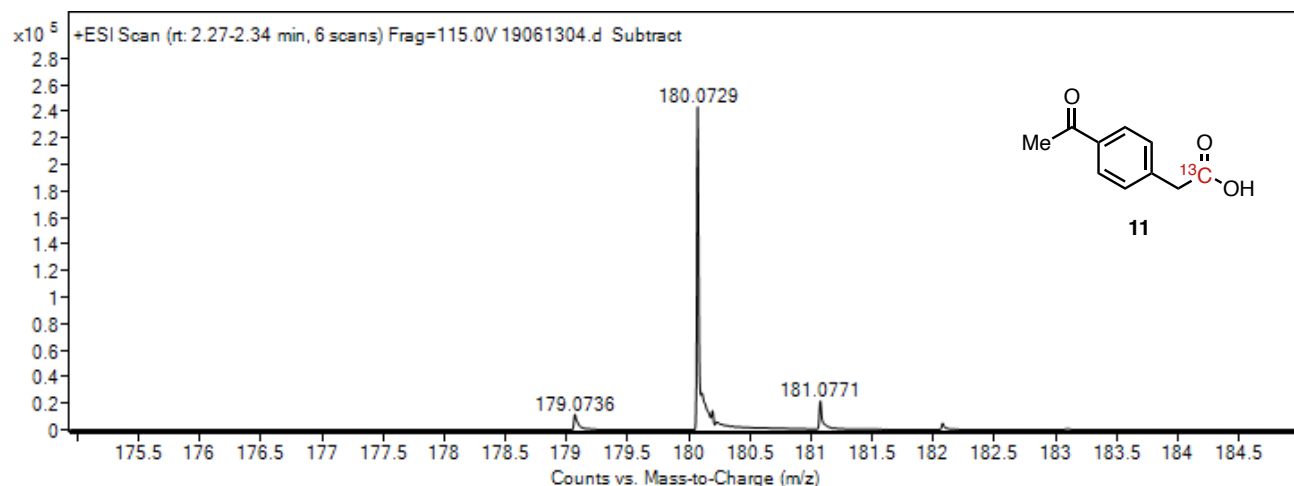
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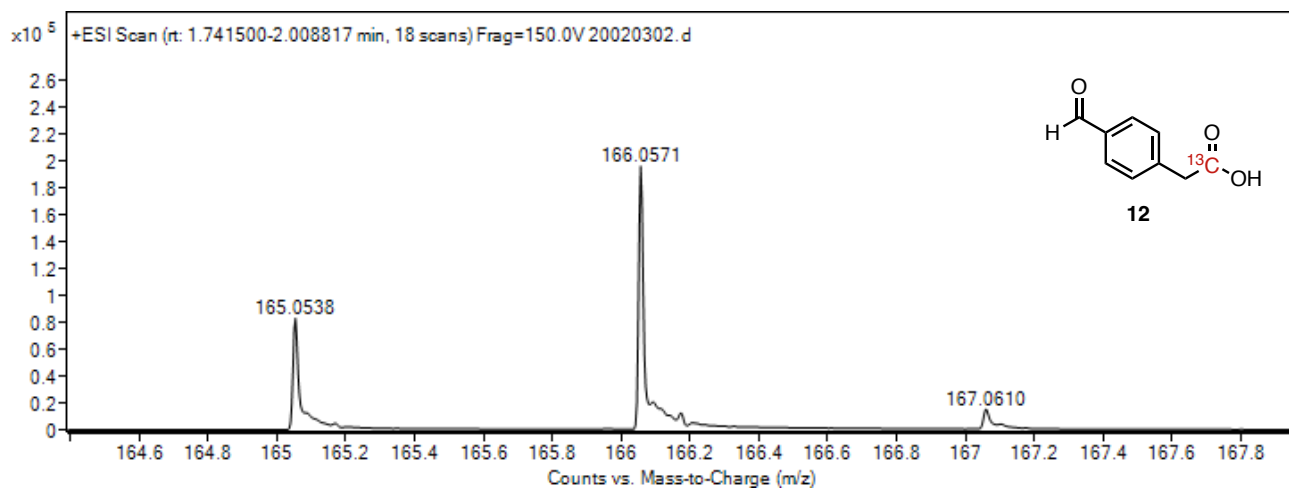
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[M-H] ⁺ 0	100	8023	8023	16
[M-H] ⁺ 1	10.95	41631	40752	84



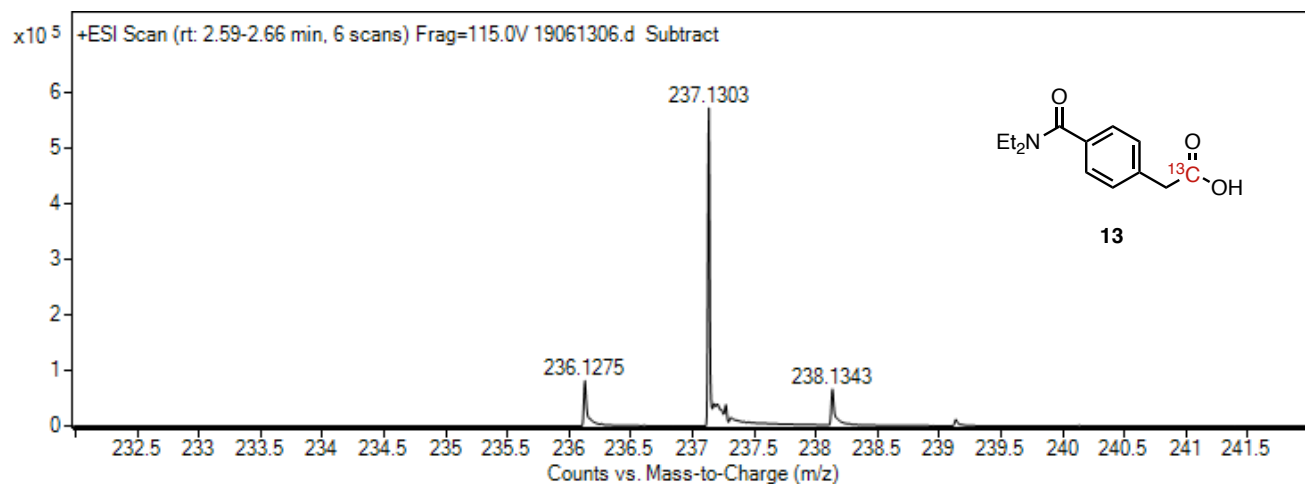
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[M-H] ⁺ 0	100	4296	4296	10
[M-H] ⁺ 1	9.87	38223	37799	90



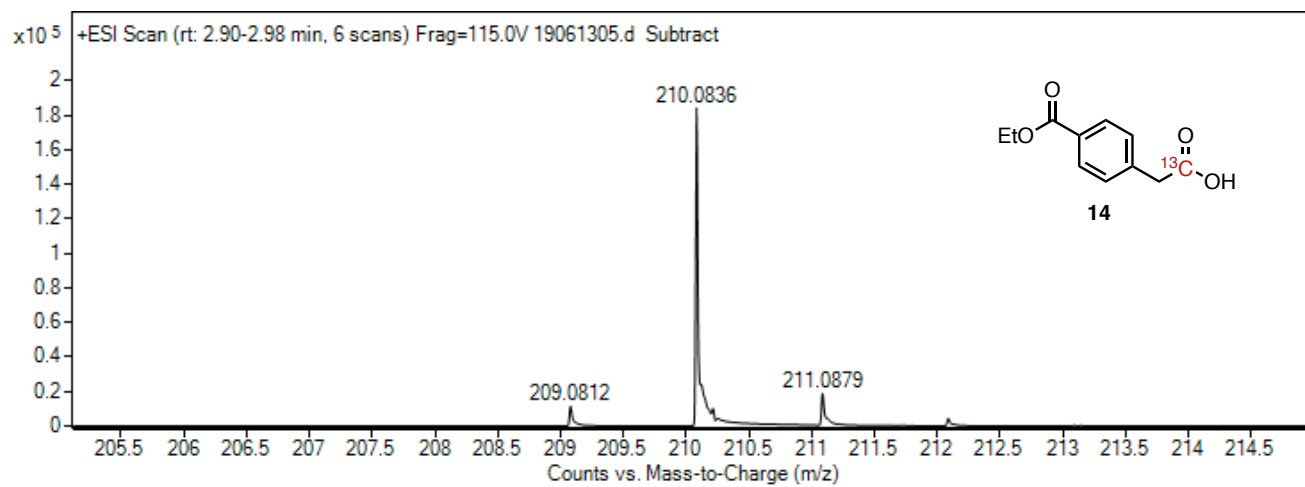
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[M+H] ⁺ 0	100	11964	11964	5
[M+H] ⁺ 1	11.06	243557	242234	95



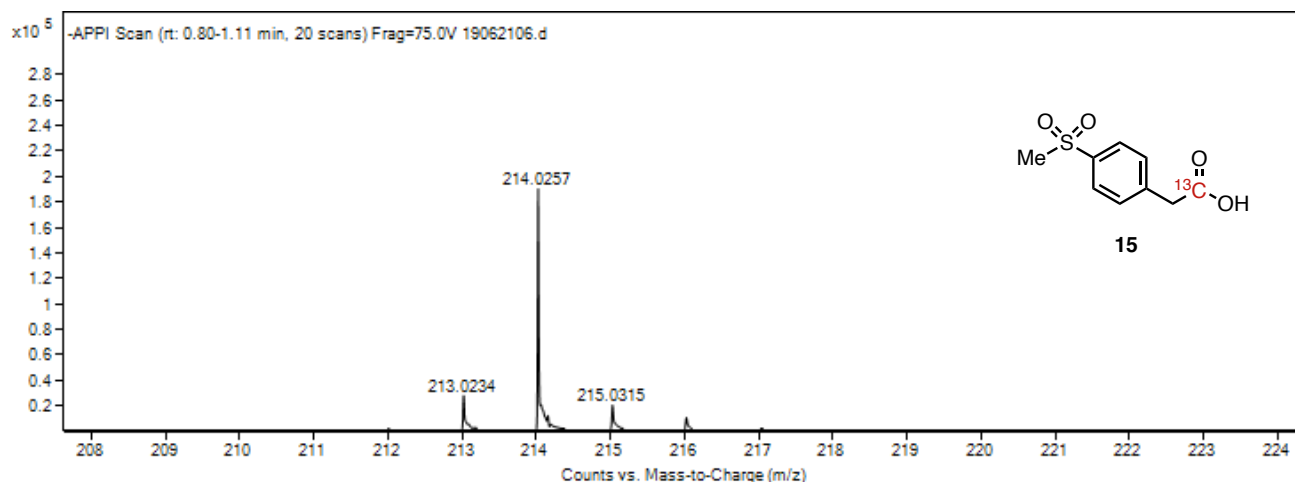
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[M+H] ⁺ 0	100	91507	91507	34
[M+H] ⁺ 1	9.95	184357	175252	66



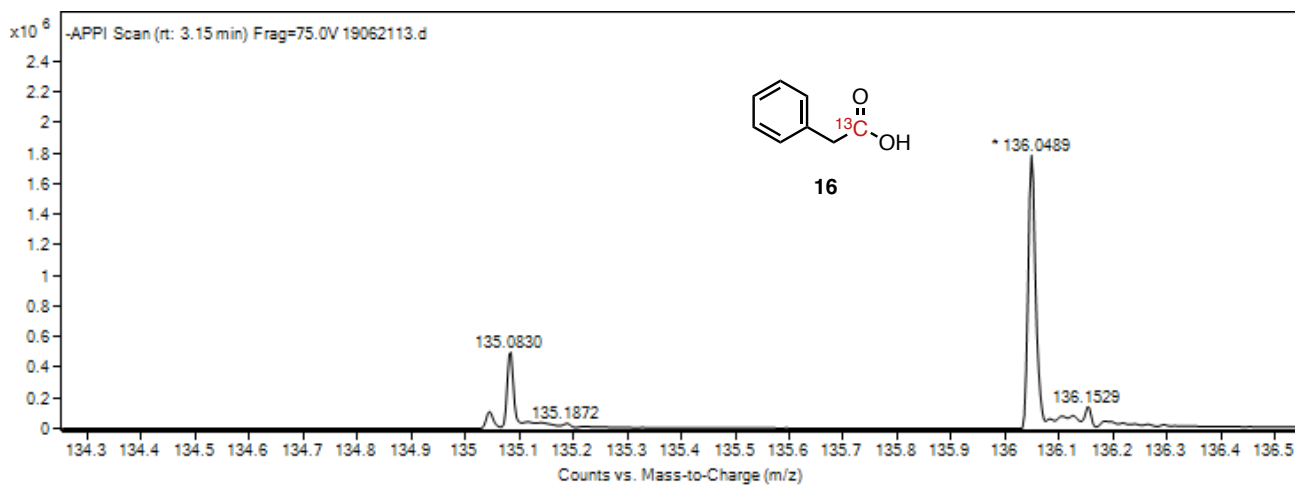
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[M+H] ⁺ 0	100	82172	82172	13
[M+H] ⁺ 1	14.75	578761	566641	87



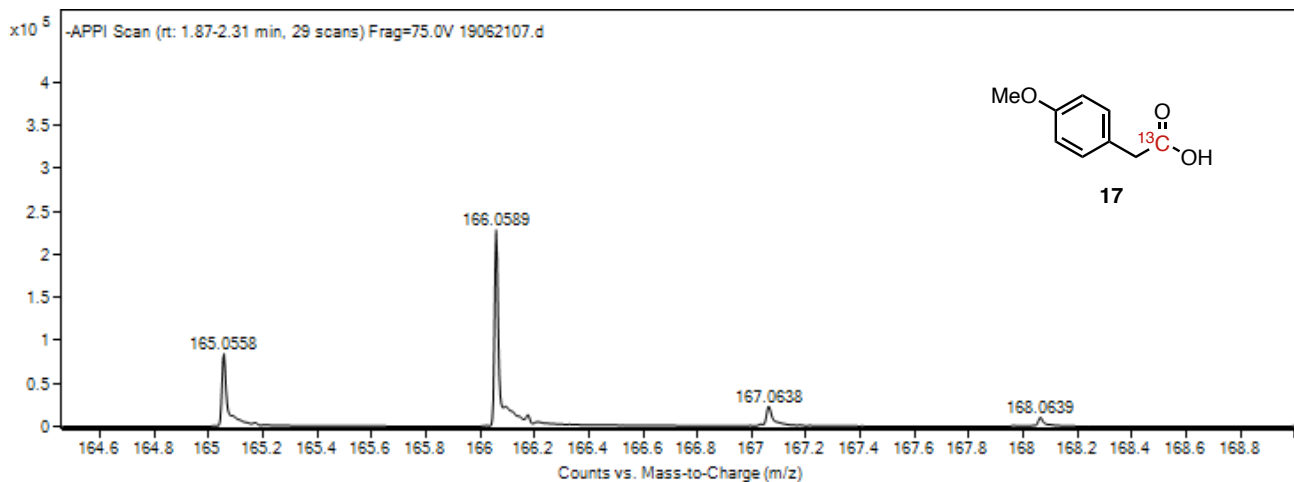
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
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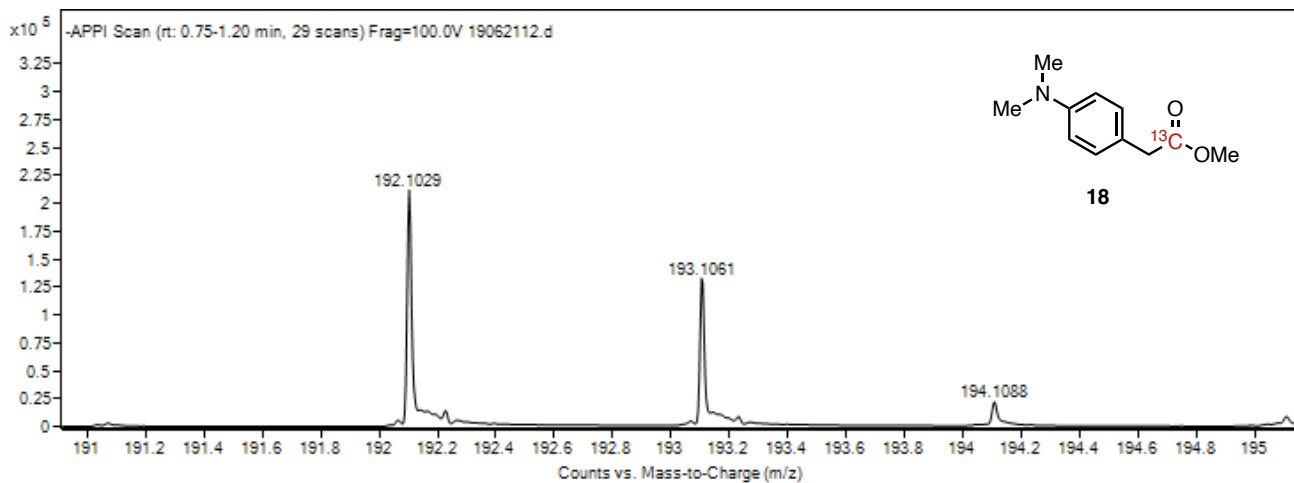
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[M-H] ⁺ 0	100	27905	27905	13
[M-H] ⁺ 1	16.21	191446	186923	87



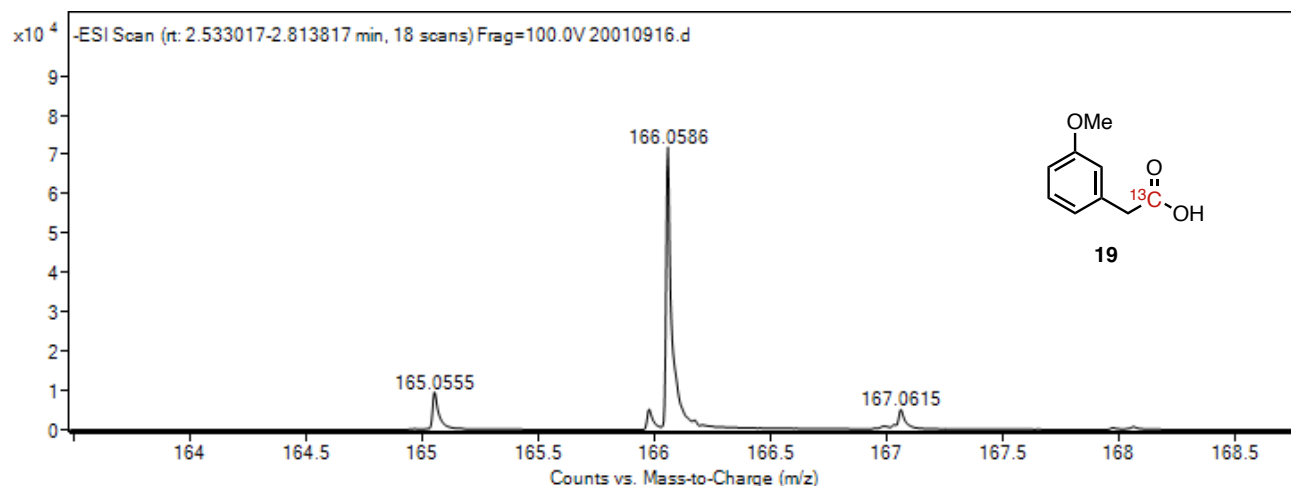
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[M-H] ⁺ 1	8.81	1790204	1780336	94



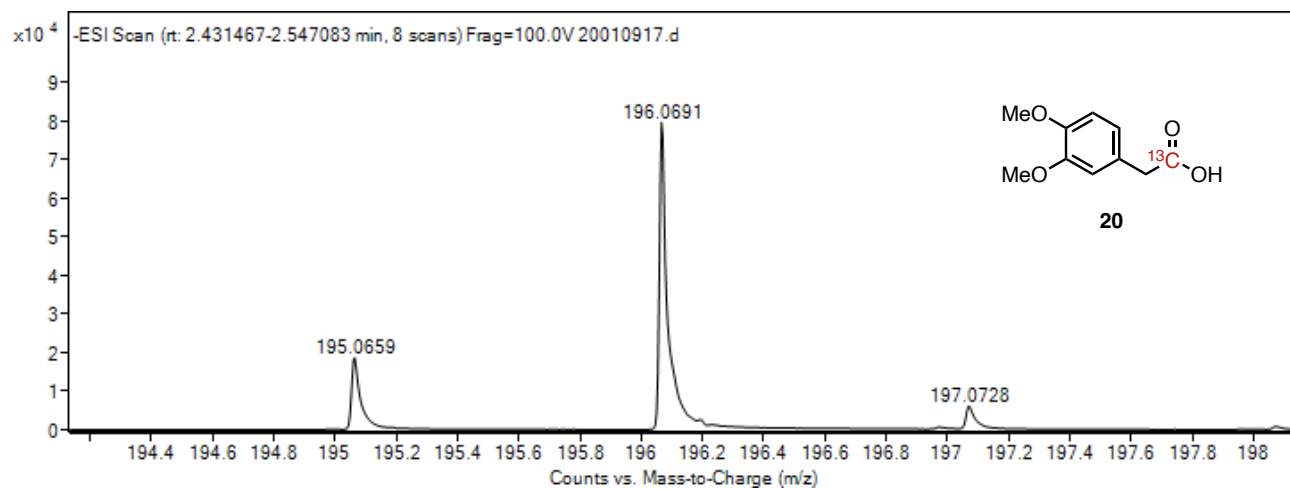
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
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[M-H] ⁺ 1	9.95	228926	220437	72



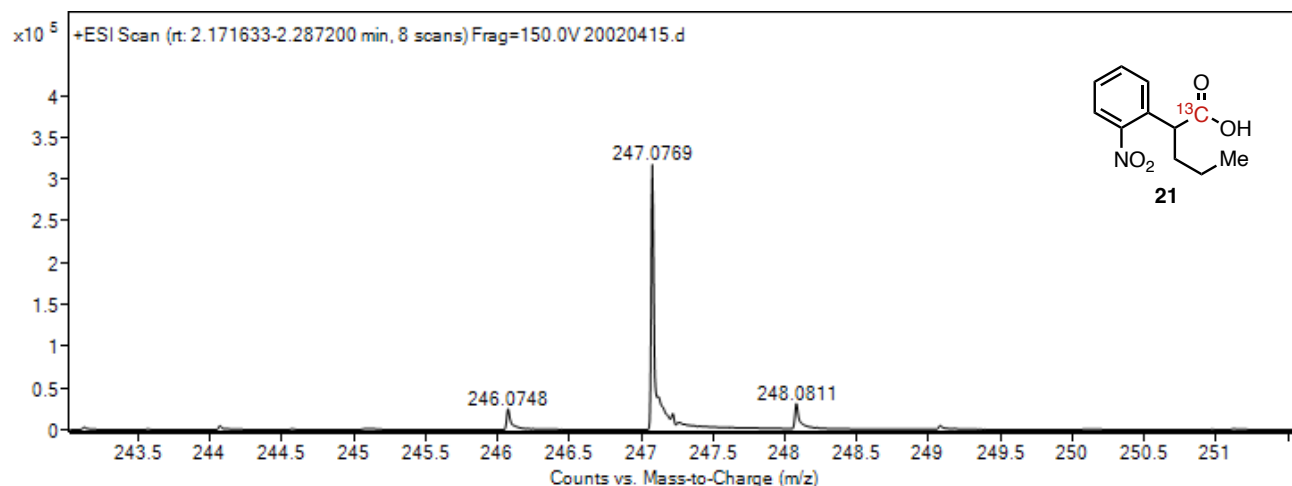
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[M-H] ⁺ 1	12.50	136103	109597	34



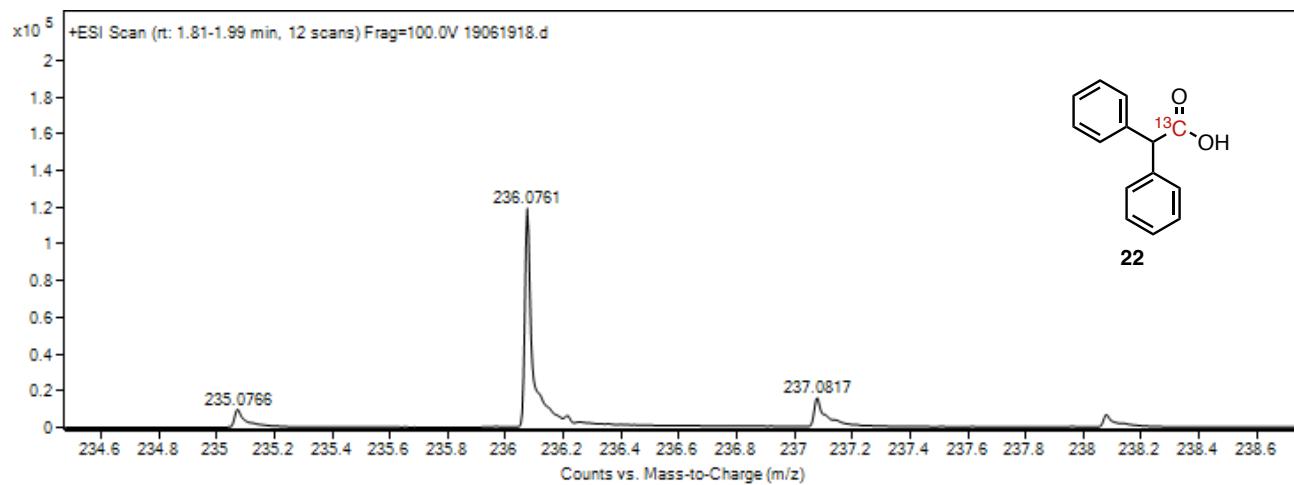
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[M-H] ⁺ 0	100	9903	9903	11
[M-H] ⁺ 1	9.95	78724	77739	89



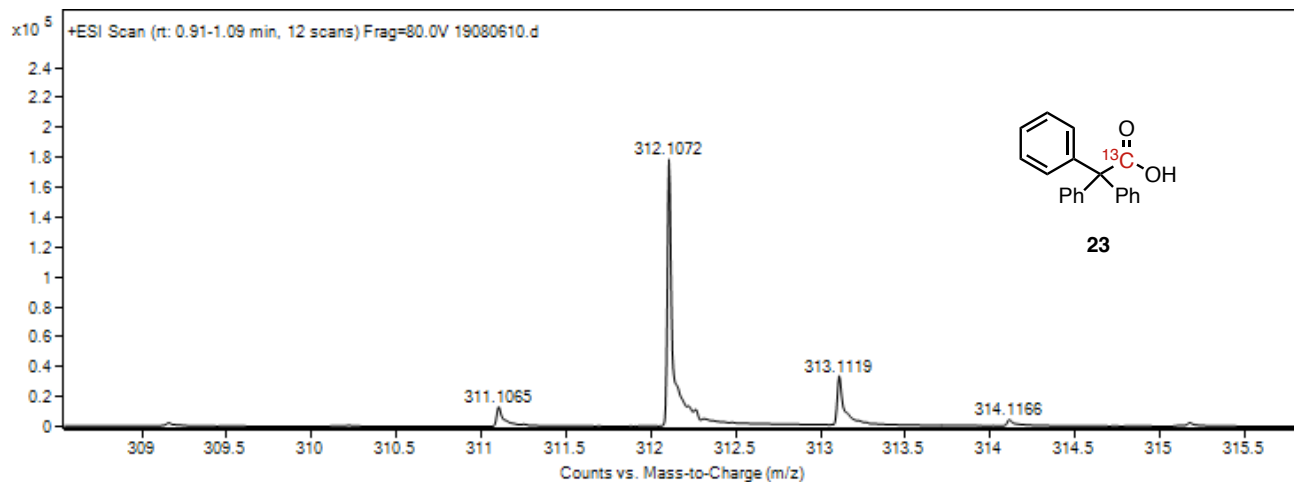
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M-H] ⁺ 0	100	18898	18898	19
[M-H] ⁺ 1	11.09	81796	79931	81



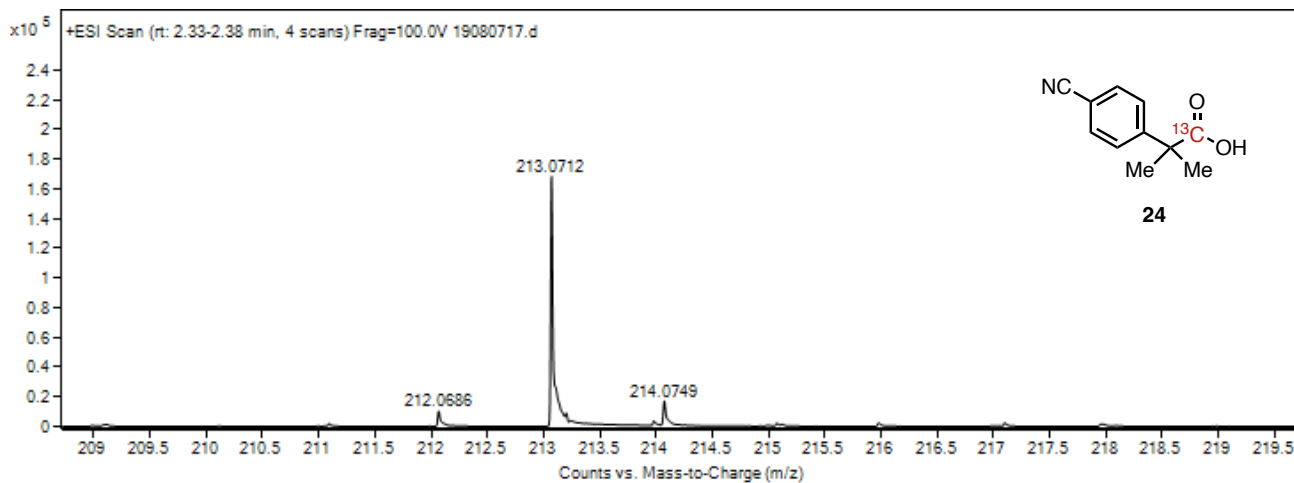
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	25413	25413	7
[M+Na] ⁺ 1	12.56	357646	354454	93



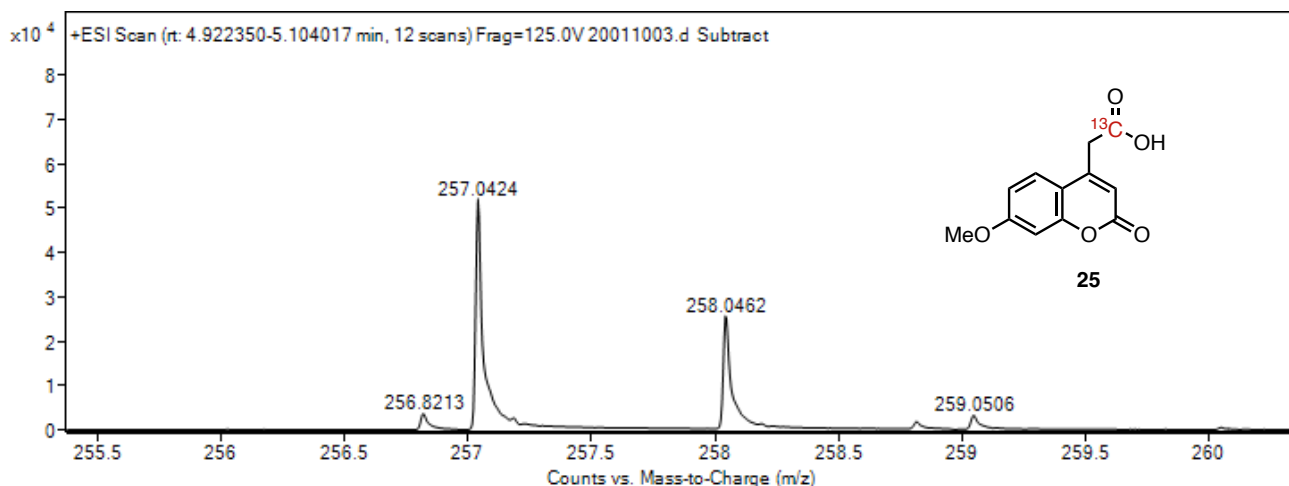
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	10010	10010	8
[M+Na] ⁺ 1	15.36	119539	118001	92



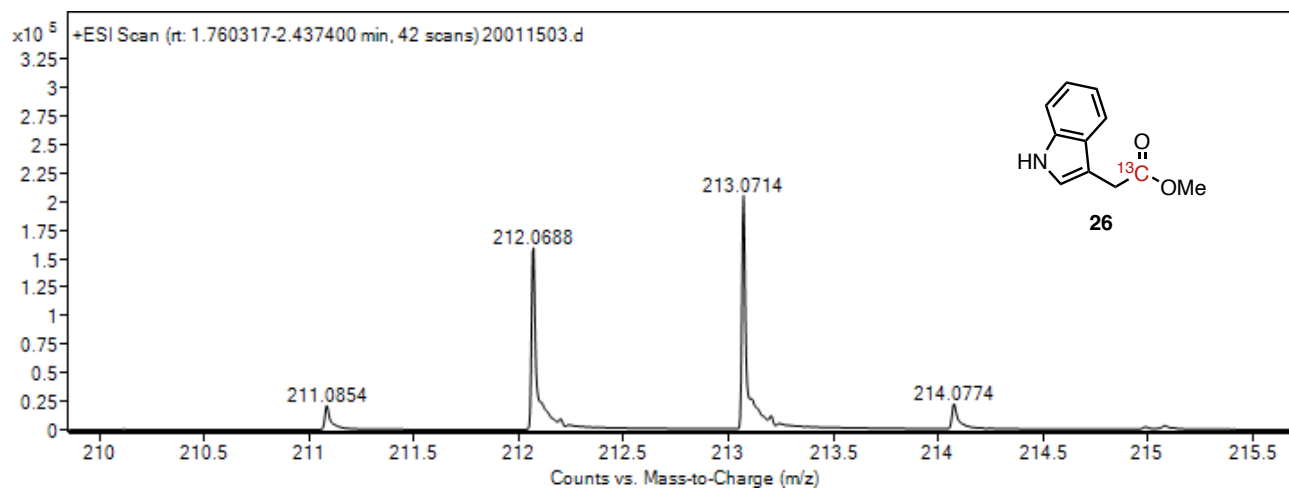
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	9462	9462	7
[M+Na] ⁺ 1	21.89	126712	124641	93



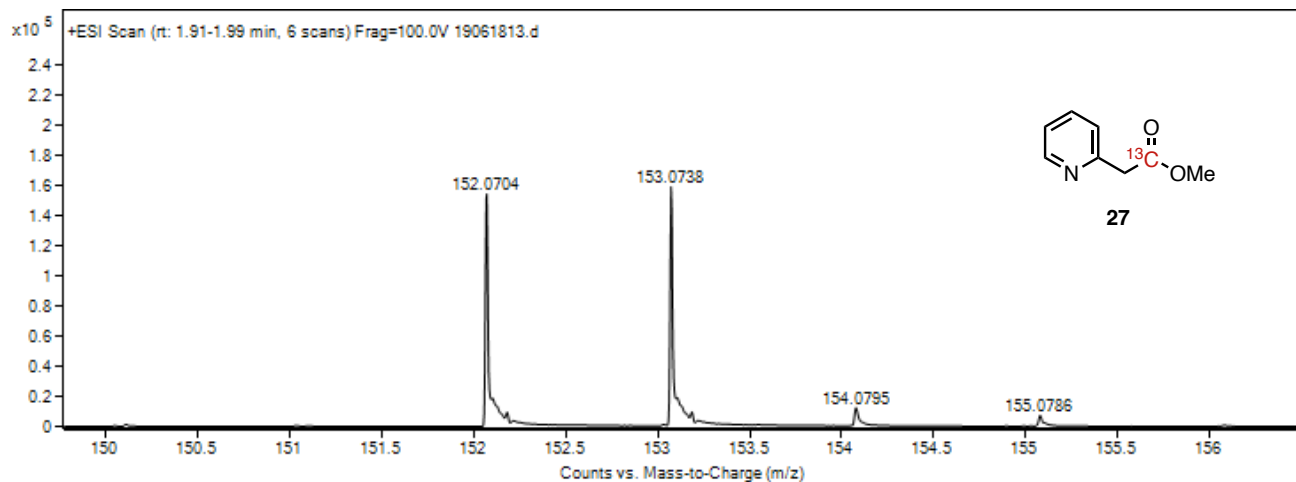
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	10504	10504	6
[M+Na] ⁺ 1	12.47	172181	170871	94



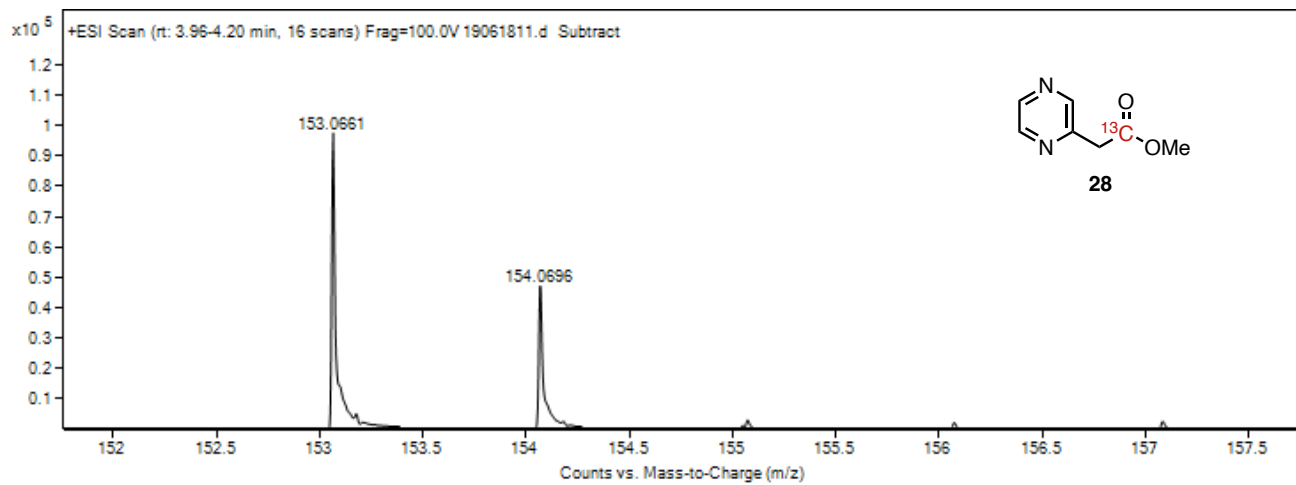
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	54959	54959	73
[M+Na] ⁺ 1	13.28	27951	20652	27



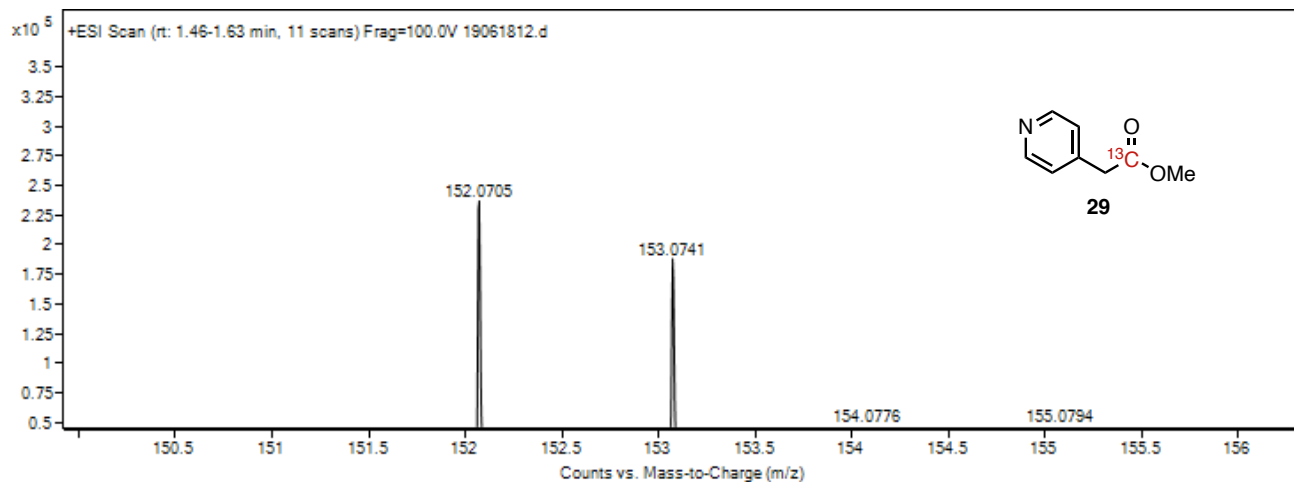
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	88549	88549	35
[M+Na] ⁺ 1	12.47	178617	167575	65



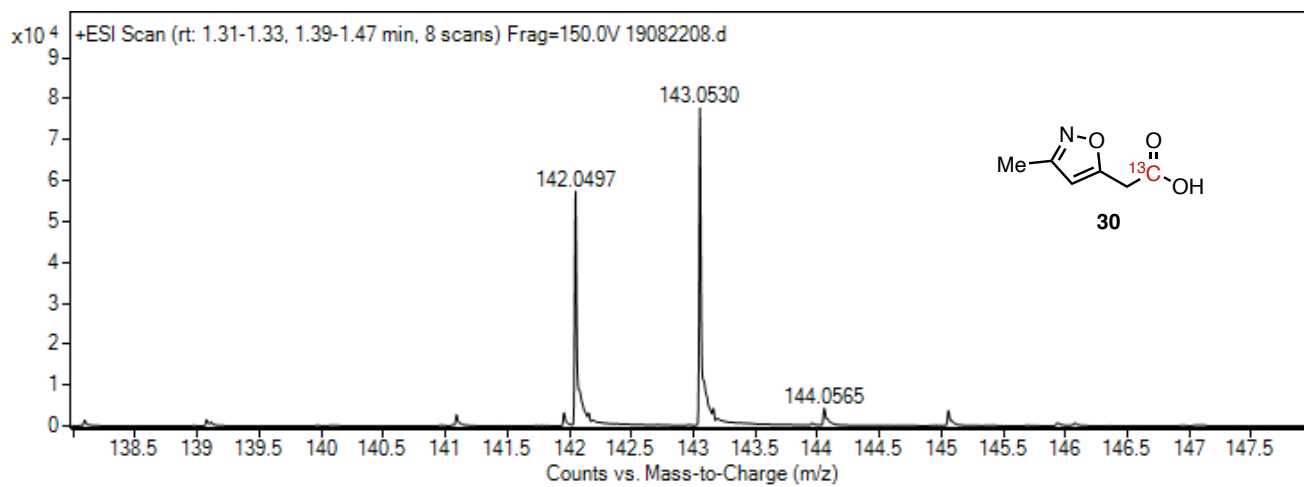
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	158893	158893	52
[M+H] ⁺ 1	9.21	160475	145841	48



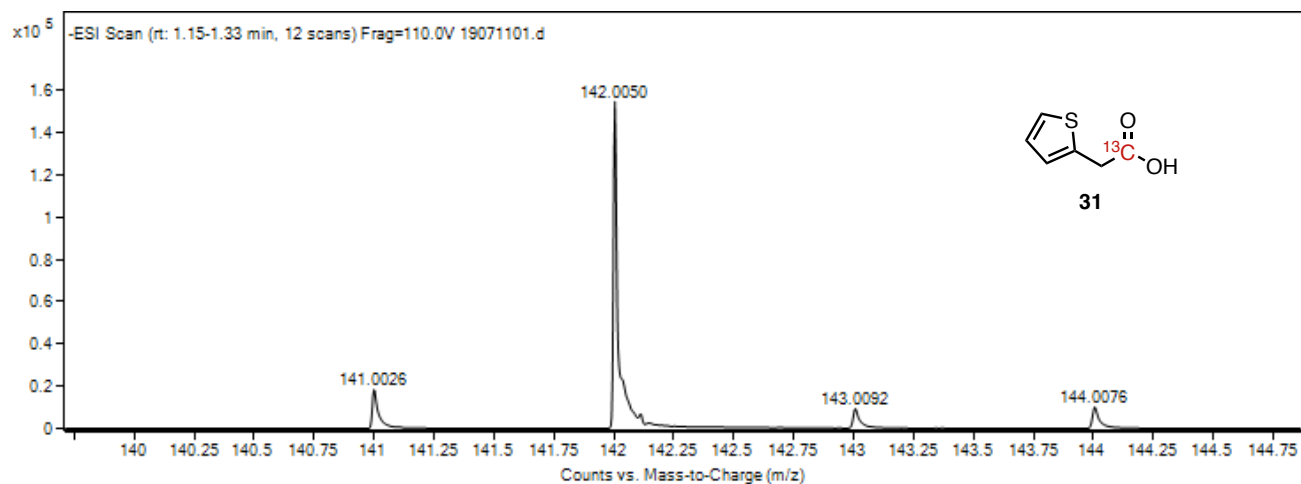
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	97829	97829	71
[M+H] ⁺ 1	8.48	48229	39933	29



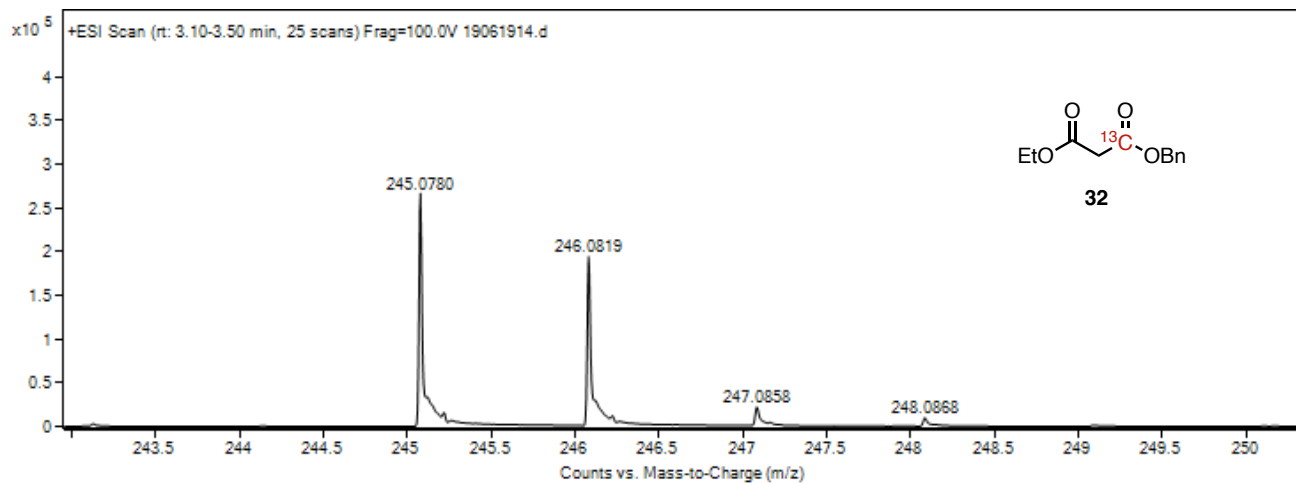
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	242965	242965	59
[M+H] ⁺ 1	9.21	189353	166976	41



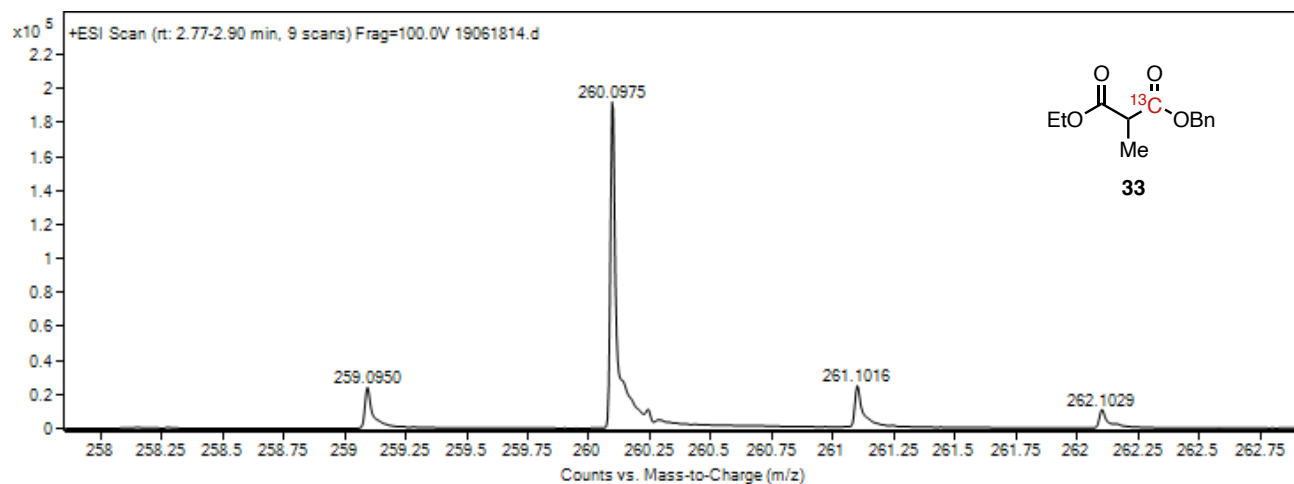
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	58169	58169	44
[M+H] ⁺ 1	7.06	77876	73769	56



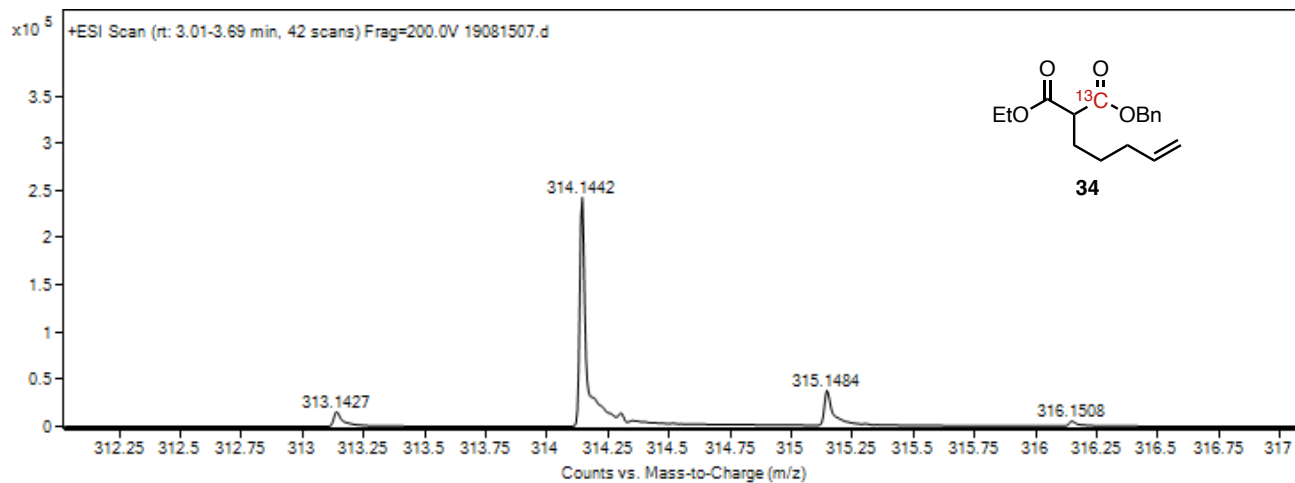
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M-H] ⁺ 0	100	18703	18703	11
[M-H] ⁺ 1	7.41	154836	153450	89



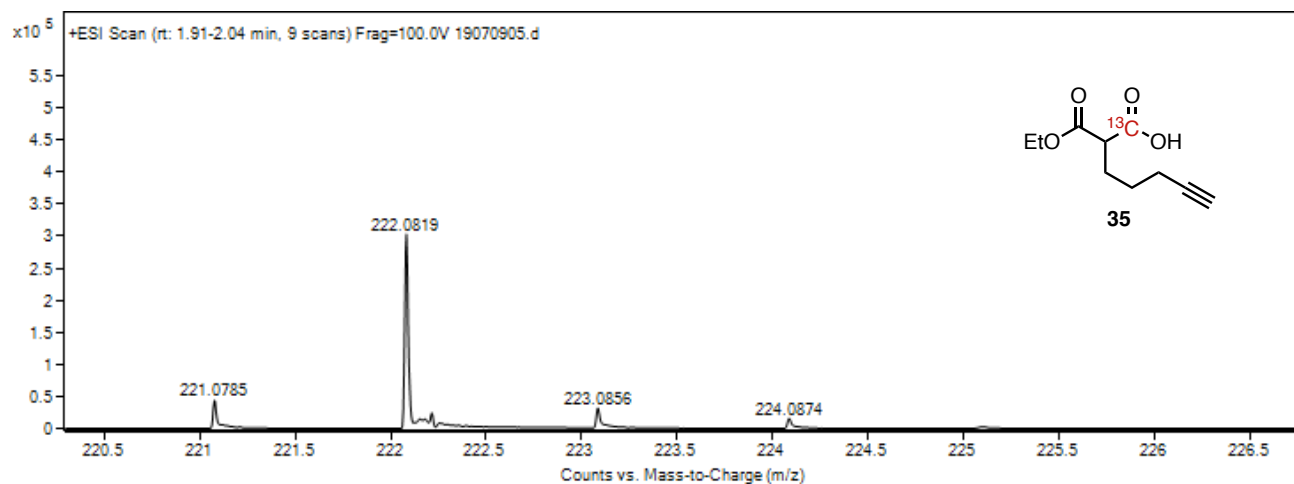
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	269248	269248	63
[M+Na] ⁺ 1	13.29	196707	160924	37



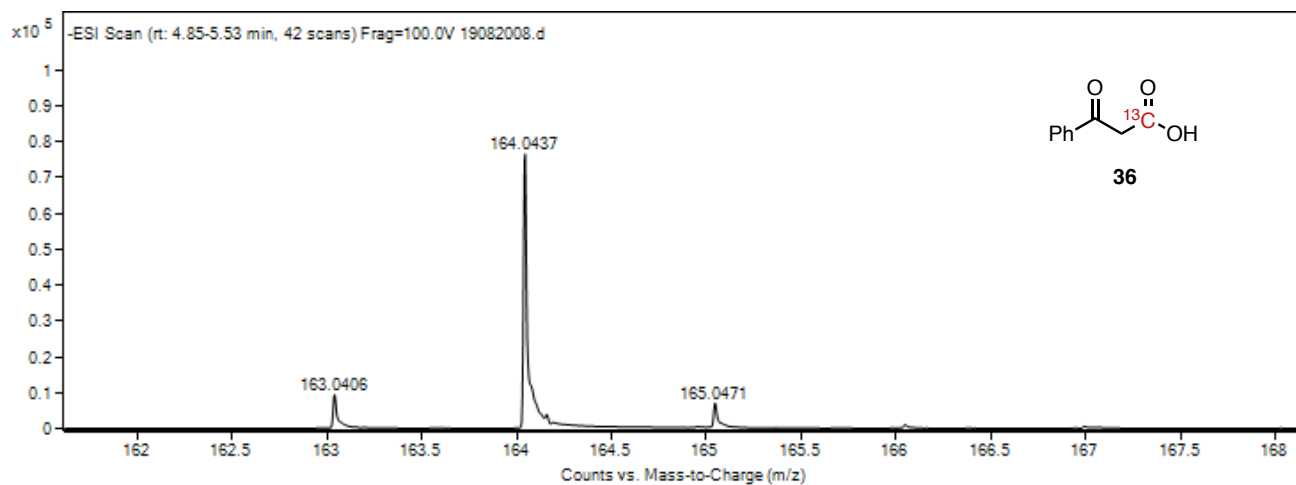
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	24312	24312	11
[M+Na] ⁺ 1	14.40	197034	193533	89



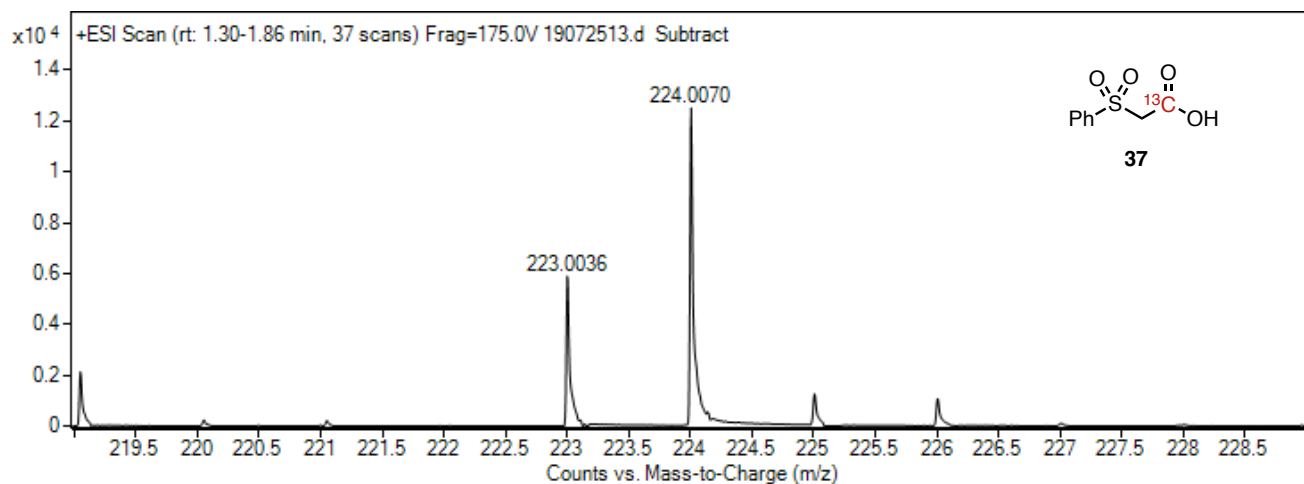
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	15708	15708	6
[M+Na] ⁺ 1	18.79	244067	241115	94



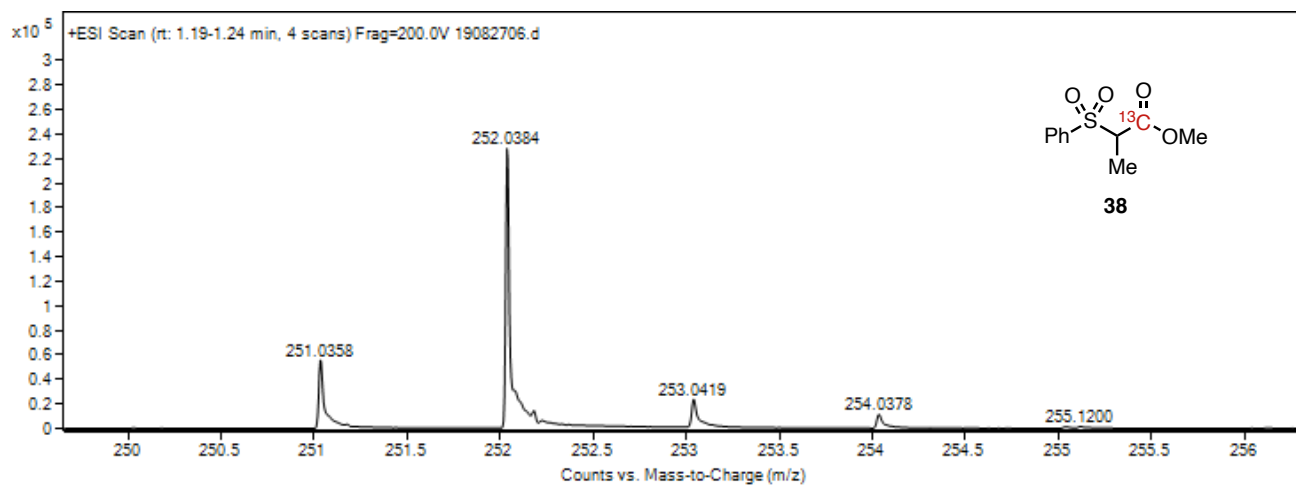
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	44719	44719	13
[M+Na] ⁺ 1	11.13	304377	299400	87



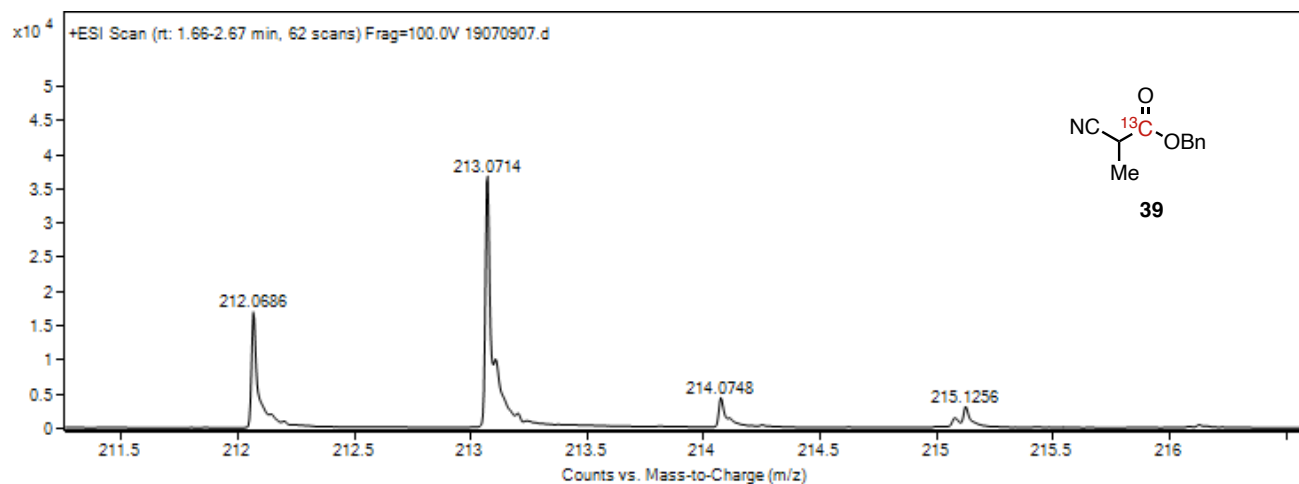
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M-H] ⁻ 0	100	9564	9564	11
[M-H] ⁻ 1	9.93	78112	77162	89



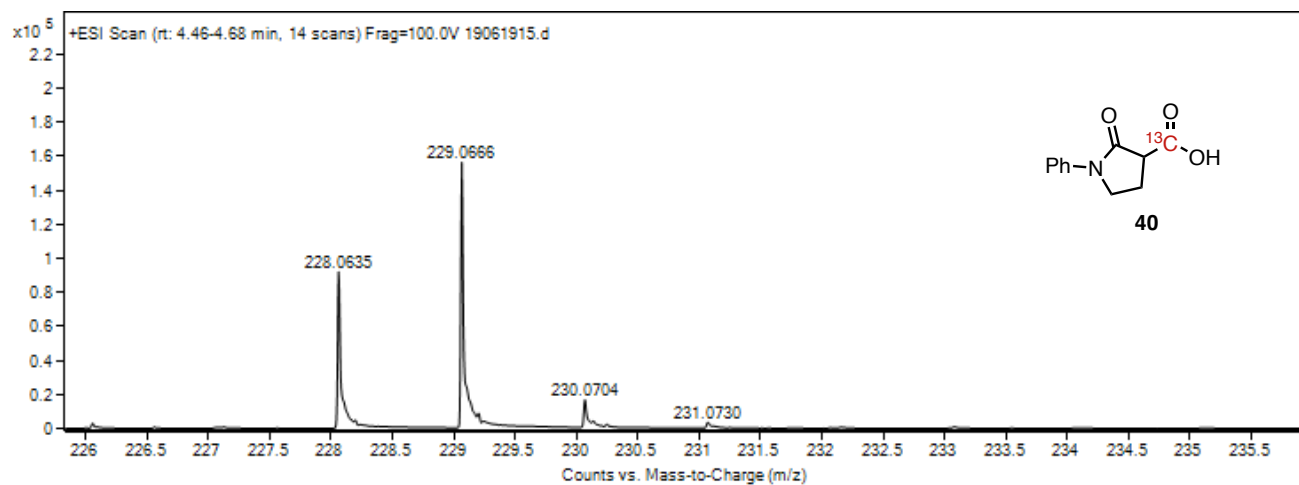
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	10188	10188	33
[M+Na] ⁺ 1	9.69	21330	20343	67



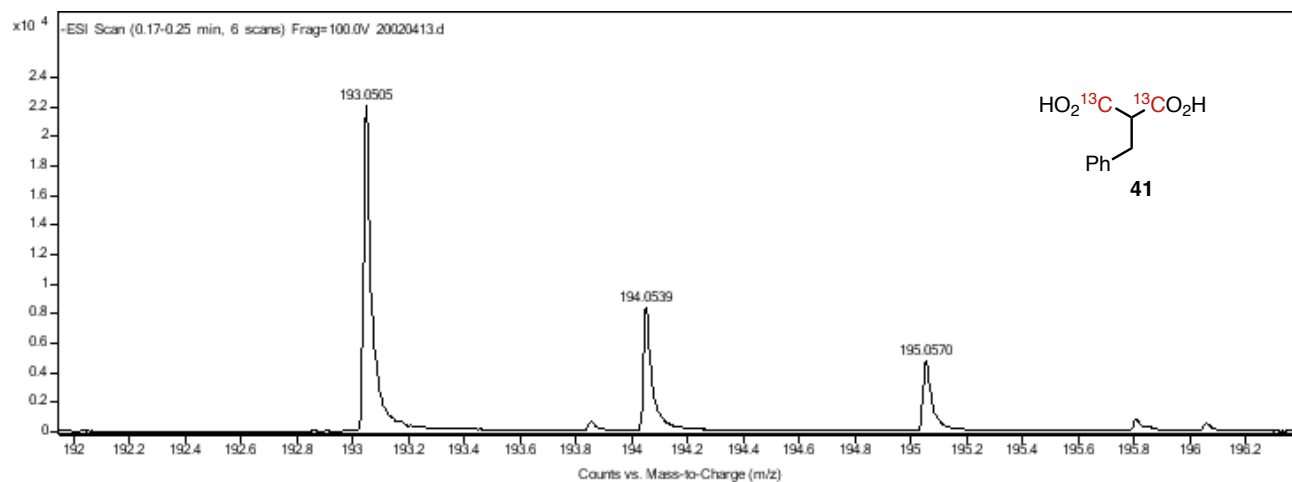
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	56915	56915	20
[M+Na] ⁺ 1	11.90	233176	226403	80



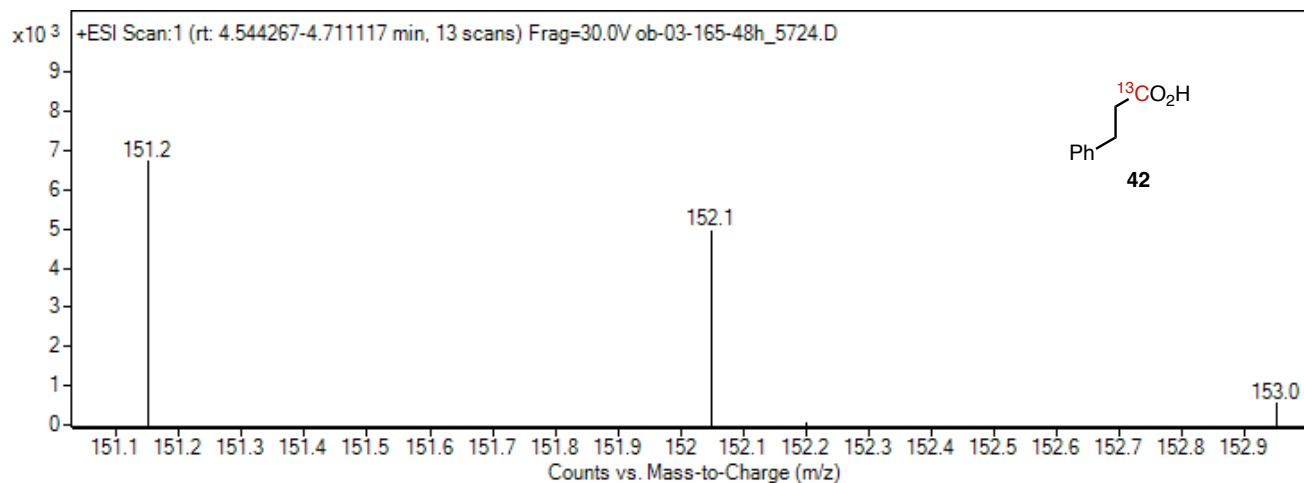
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	17196	17196	33
[M+Na] ⁺ 1	12.47	37528	35384	67



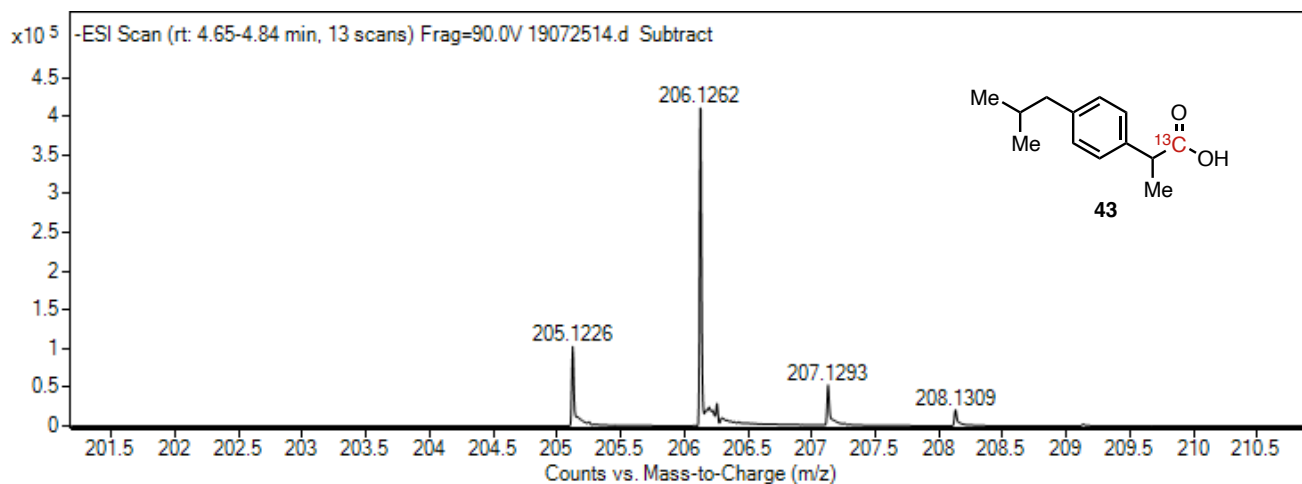
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	93055	93055	39
[M+Na] ⁺ 1	12.50	156561	144929	61



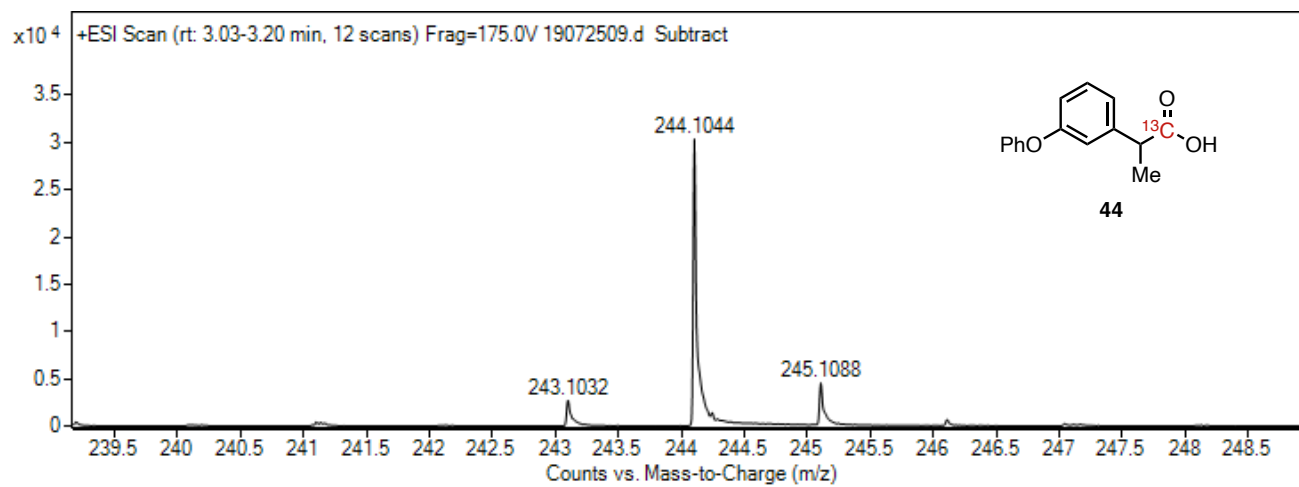
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M-H] ⁺ 0	100	22070	22070	69
[M-H] ⁺ 1	10	8421	5993	19
[M-H] ⁺ 2	8.93	4816	3912	12



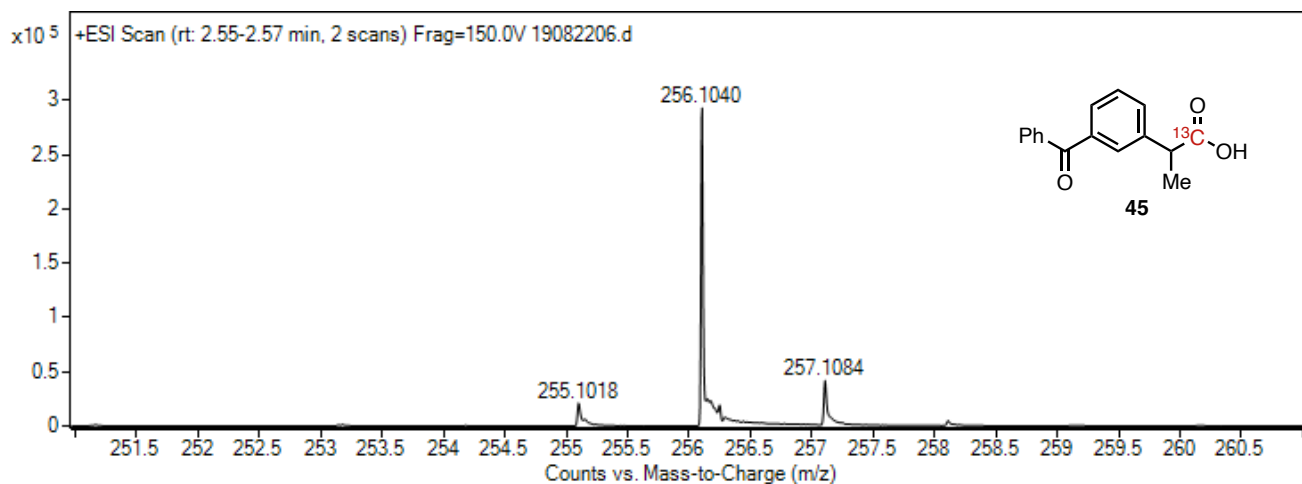
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	6727	6727	61
[M+H] ⁺ 1	8.96	4967	4364	39



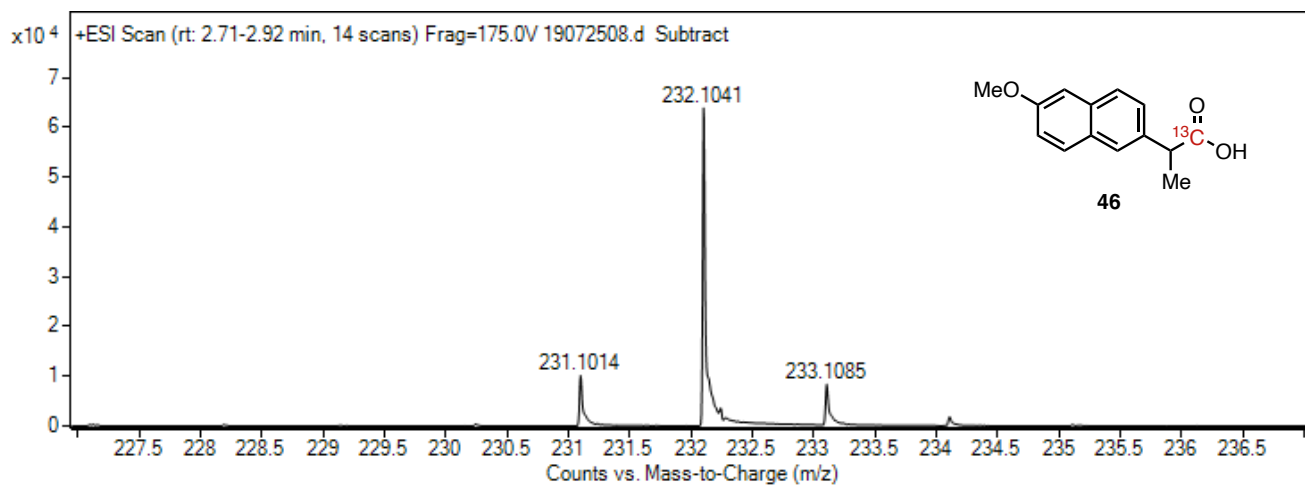
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M-H] ⁺ 0	100	103035	103035	20
[M-H] ⁺ 1	14.33	427379	412615	80



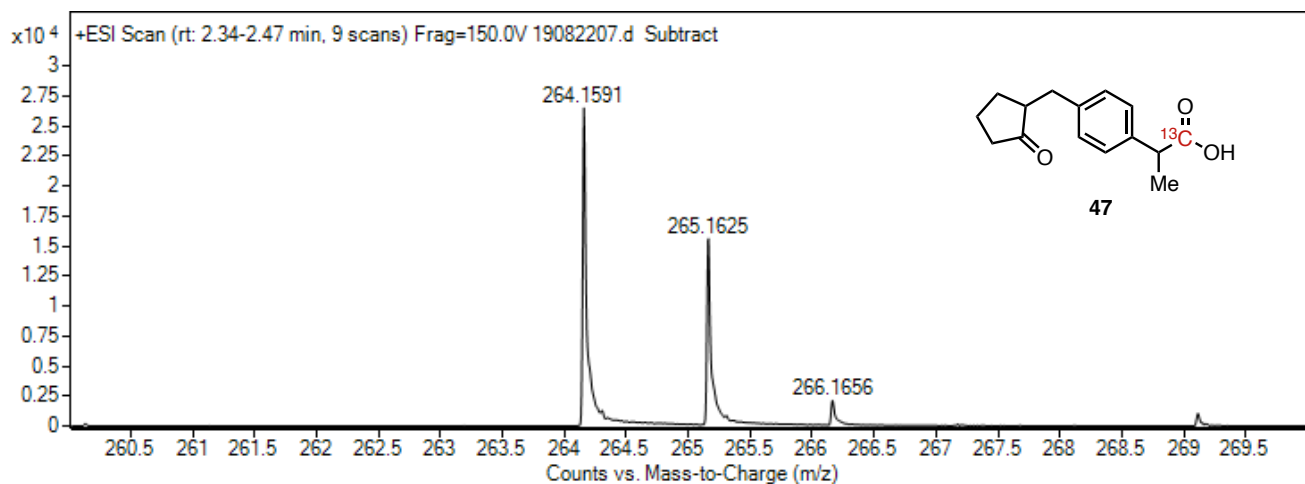
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	2742	2742	8
[M+H] ⁺ 1	16.51	30383	29930	92



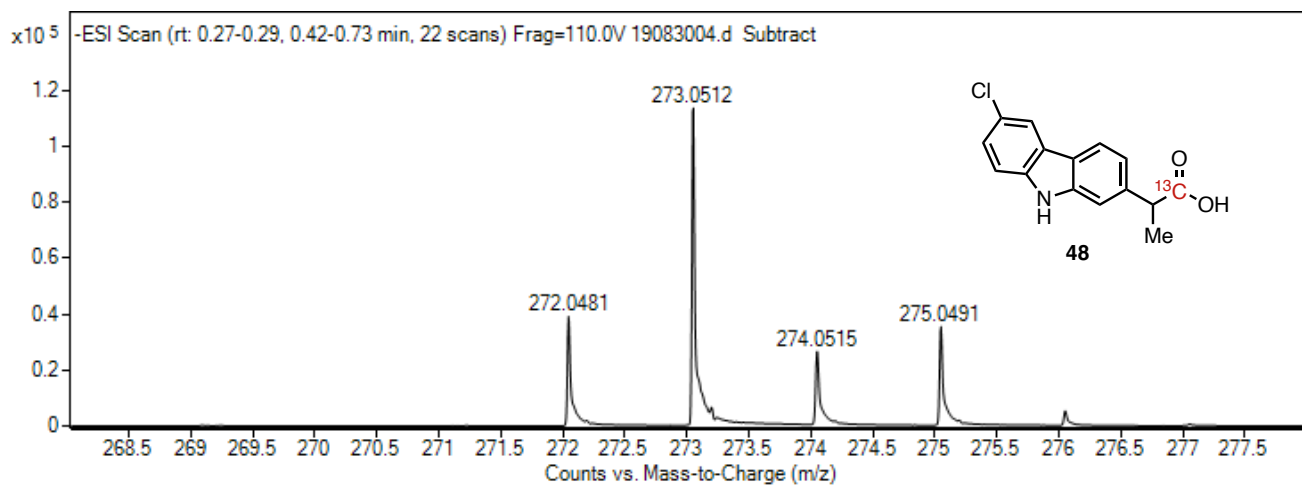
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	21534	21534	7
[M+H] ⁺ 1	17.59	300814	297026	93



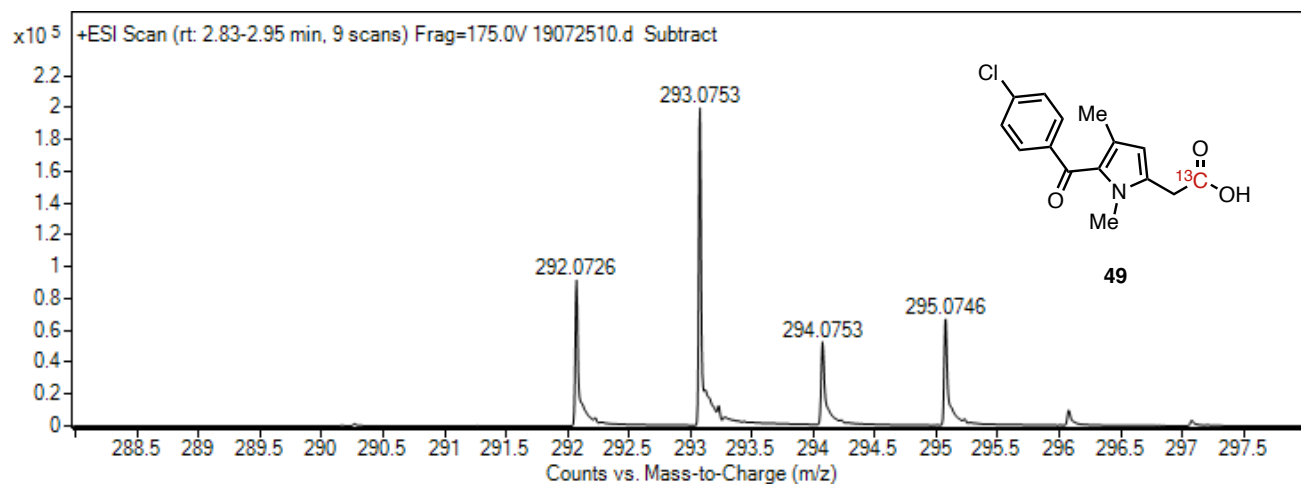
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	10208	10208	14
[M+H] ⁺ 1	15.43	65384	63809	86



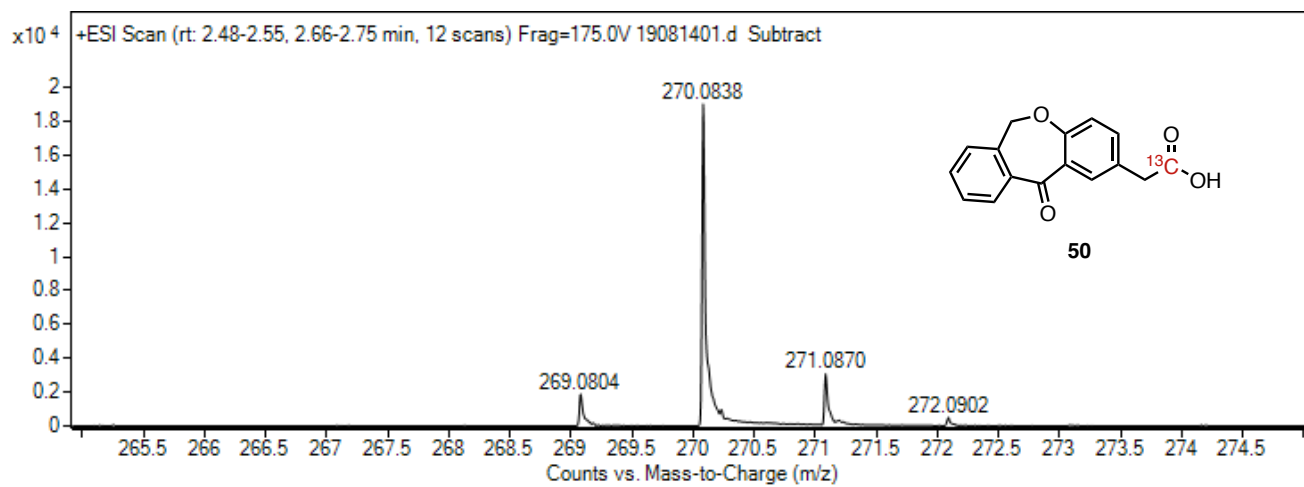
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	27004	27004	70
[M+H] ⁺ 1	16.96	16324	11744	30



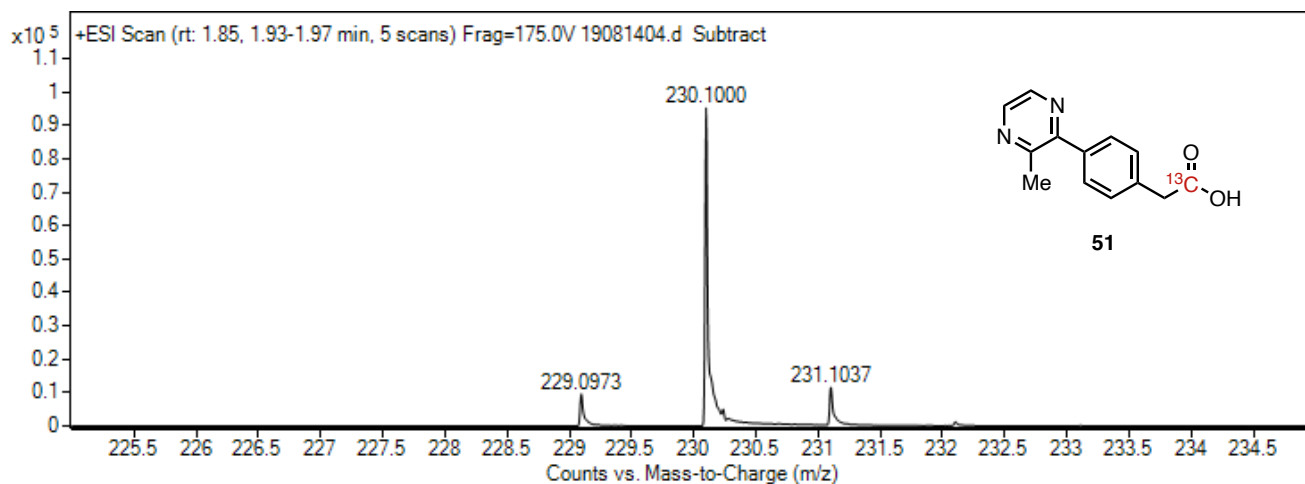
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M-H] ⁻ 0	100	81171	81171	26
[M-H] ⁻ 1	16.79	249387	235758	74



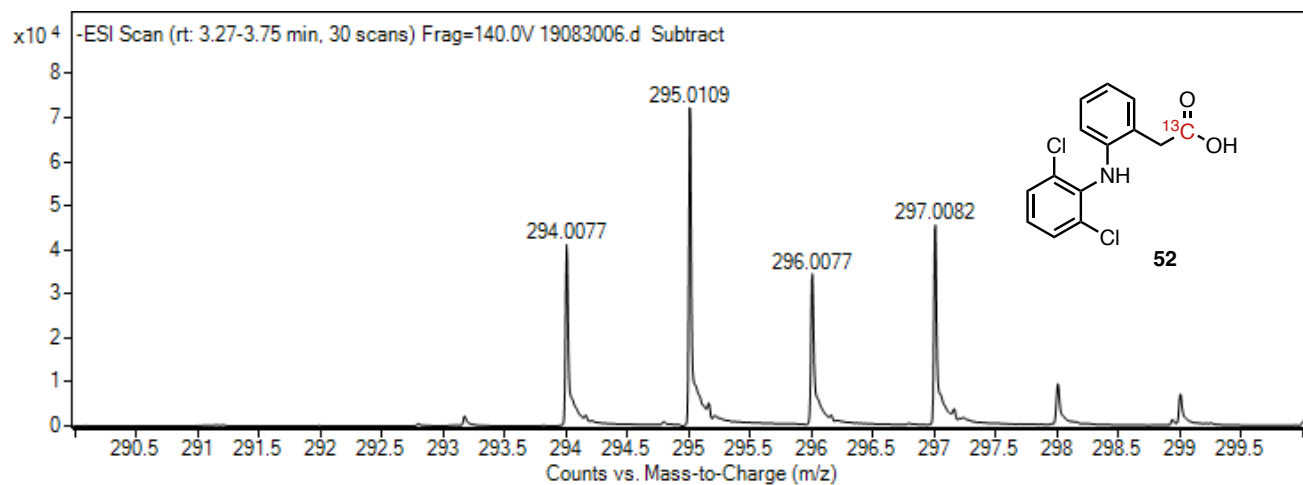
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	91794	91794	33
[M+H] ⁺ 1	16.88	201751	186256	67



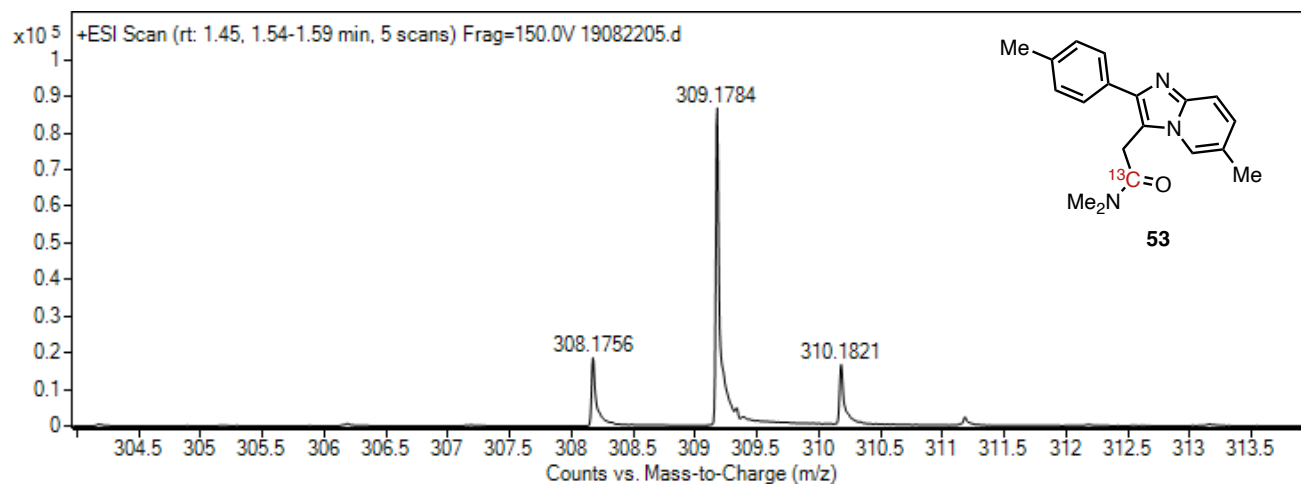
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	1909	1909	9
[M+H] ⁺ 1	17.61	19208	18872	91



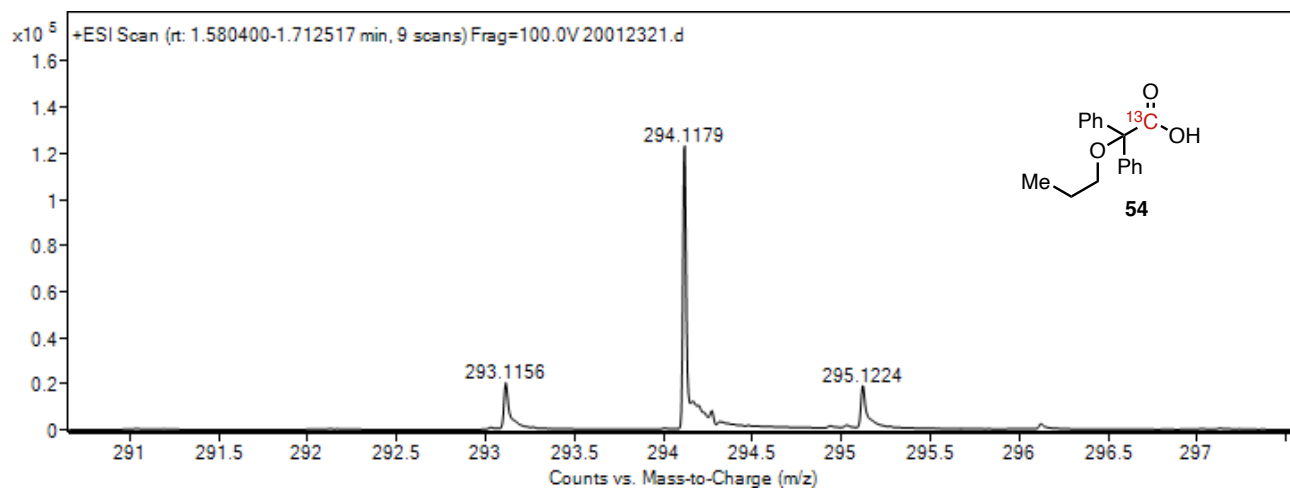
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	9656	9656	9
[M+H] ⁺ 1	15.02	95419	93969	91



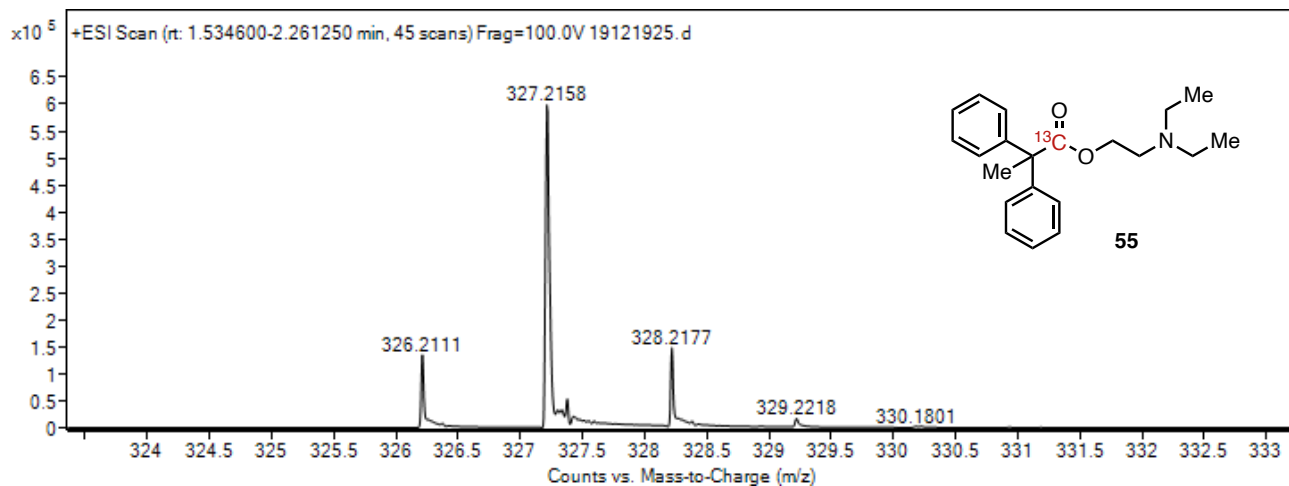
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M-H] ⁻ 0	100	28933	28933	42
[M-H] ⁻ 1	15.70	49367	44825	58



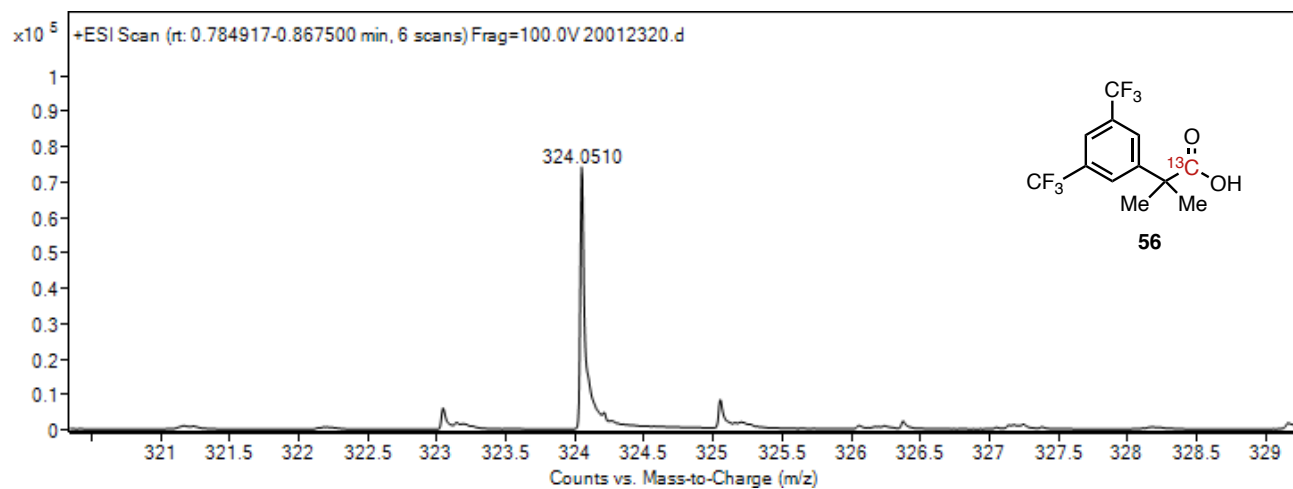
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M-H] ⁺ 0	100	18786	18786	18
[M-H] ⁺ 1	21.94	87604	83482	82



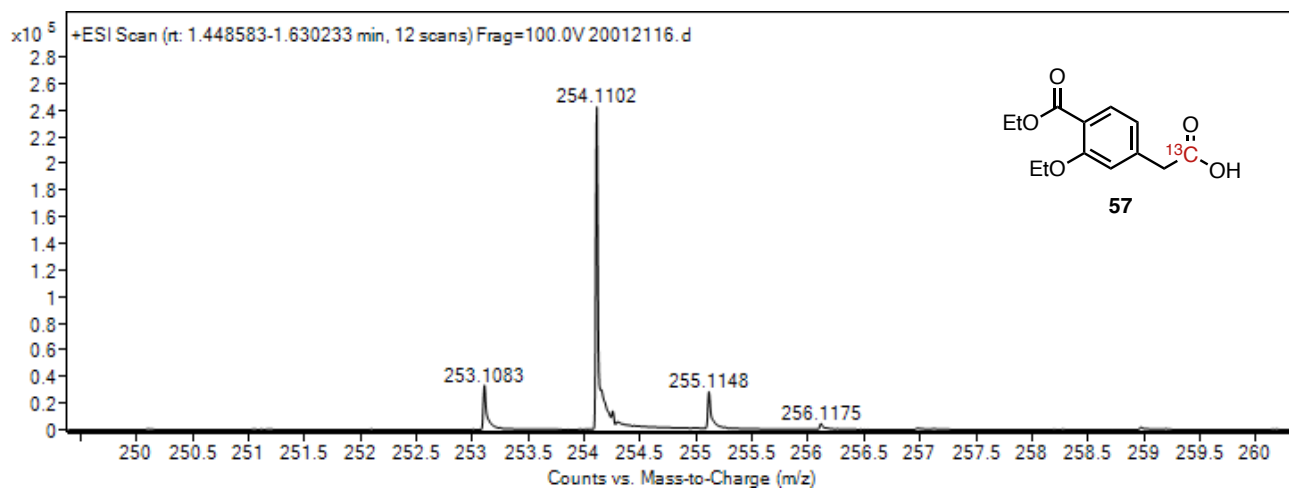
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	20576	20576	14
[M+Na] ⁺ 1	18.71	131668	127818	86



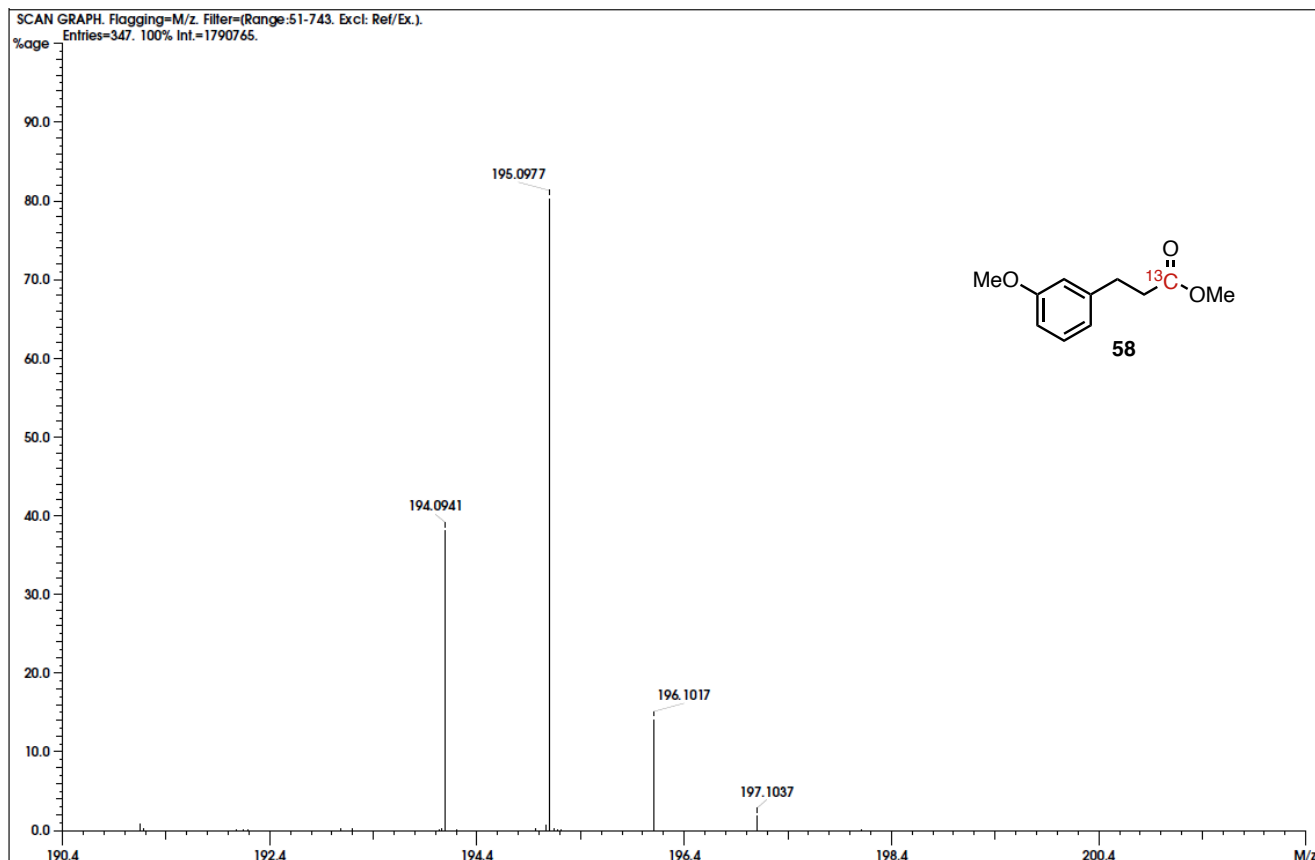
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	41426	41426	17
[M+H] ⁺ 1	23.48	213148	203421	83



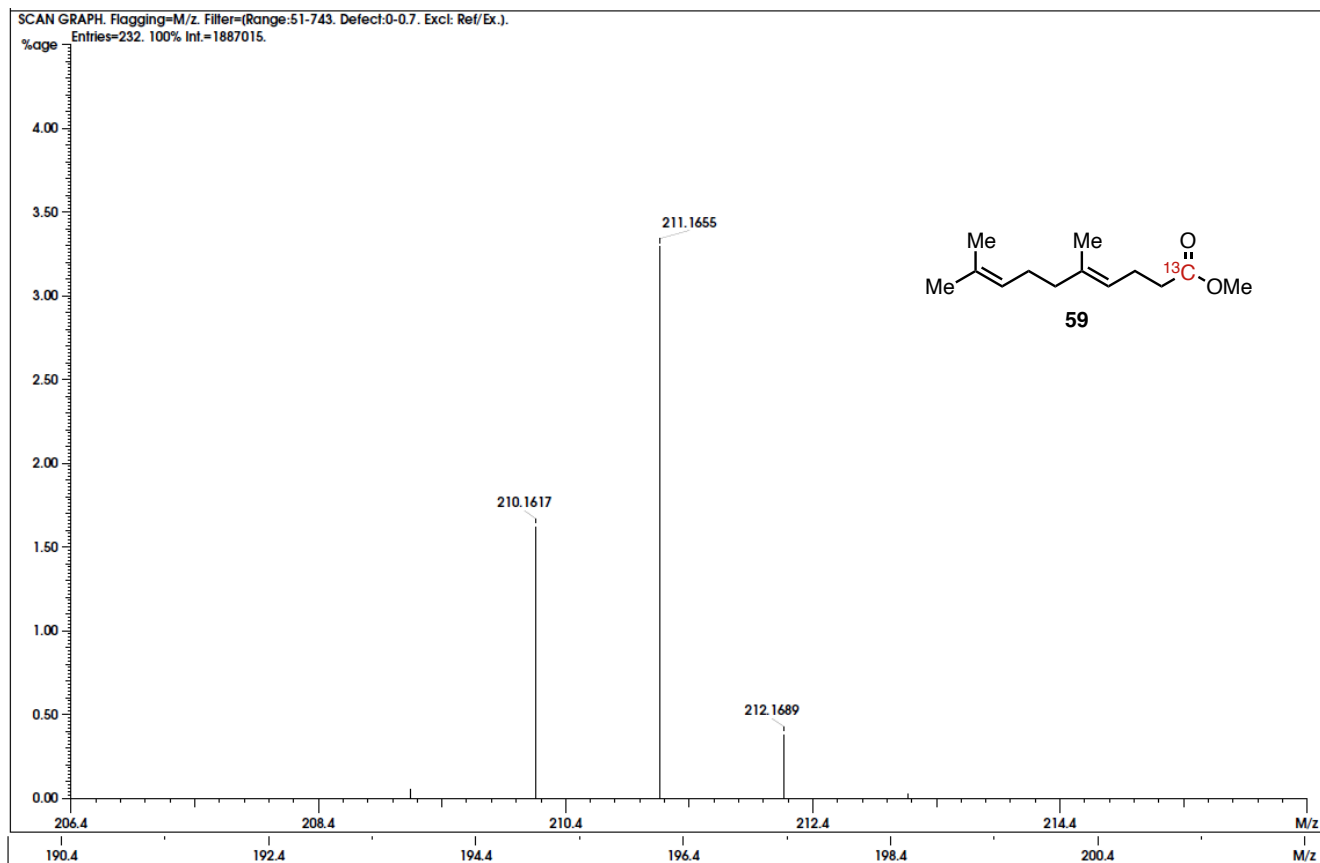
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	3923	3923	7
[M+Na] ⁺ 1	13.17	54759	54242	93



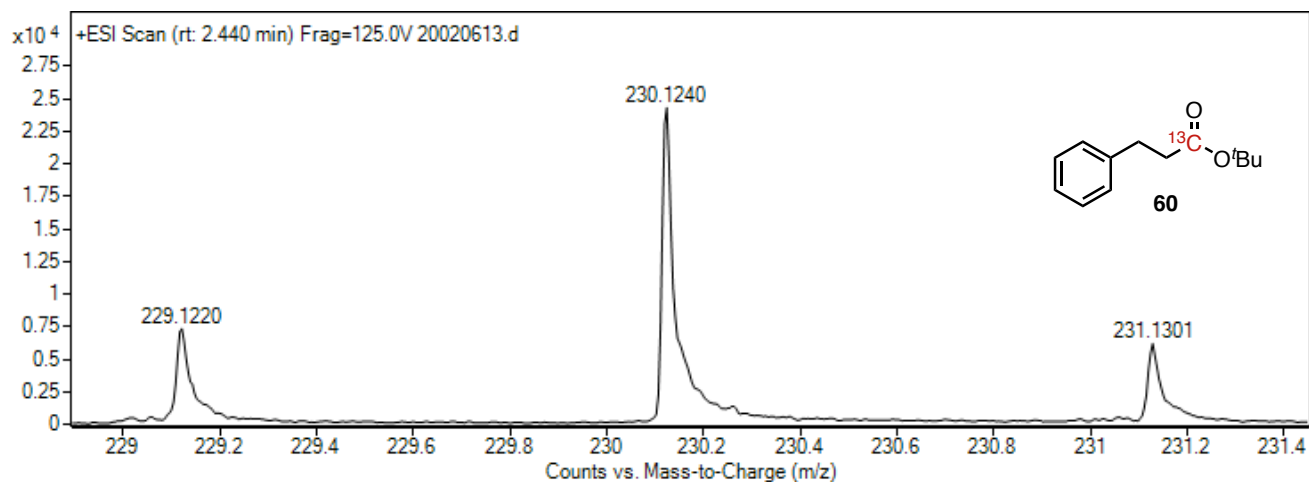
m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	34022	34022	11
[M+H] ⁺ 1	14.45	275587	270671	89



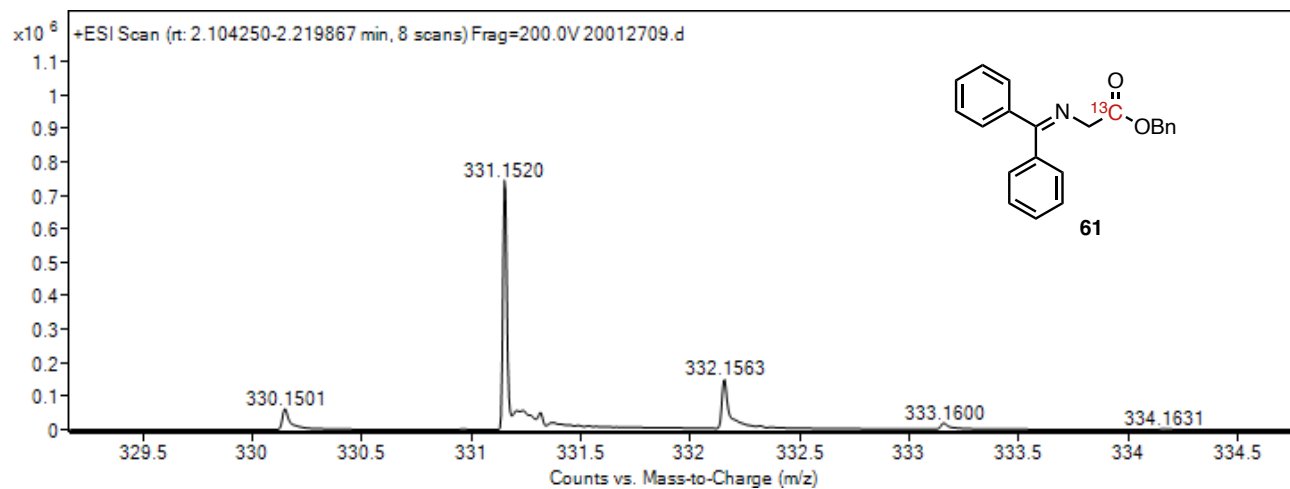
m/z	Relative Natural Abundance (%)	Observed Area (%)	Corrected Area (%)	Isotopic Enrichment Relative (%)
[M]+0	100	38	38	34
[M]+1	12.17	80	75	66



m/z	Relative Natural Abundance (%)	Observed Area (%)	Corrected Area (%)	Isotopic Enrichment Relative (%)
[M]+0	100	1.62	1.62	35
[M]+1	14.39	3.29	3	65



m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+Na] ⁺ 0	100	175276	175276	23
[M+Na] ⁺ 1	14.34	616876	591741	77



m/z	Relative Natural Abundance (%)	Observed Abundance (%)	Corrected Abundance (%)	Isotopic Enrichment Relative (%)
[M+H] ⁺ 0	100	62772	62772	7
[M+H] ⁺ 1	24.47	795359	779999	93

XII ^1H and ^{13}C Spectra



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Recorded on: **u500, Jun 26 2019**
Pulse Sequence: **PRESAT**

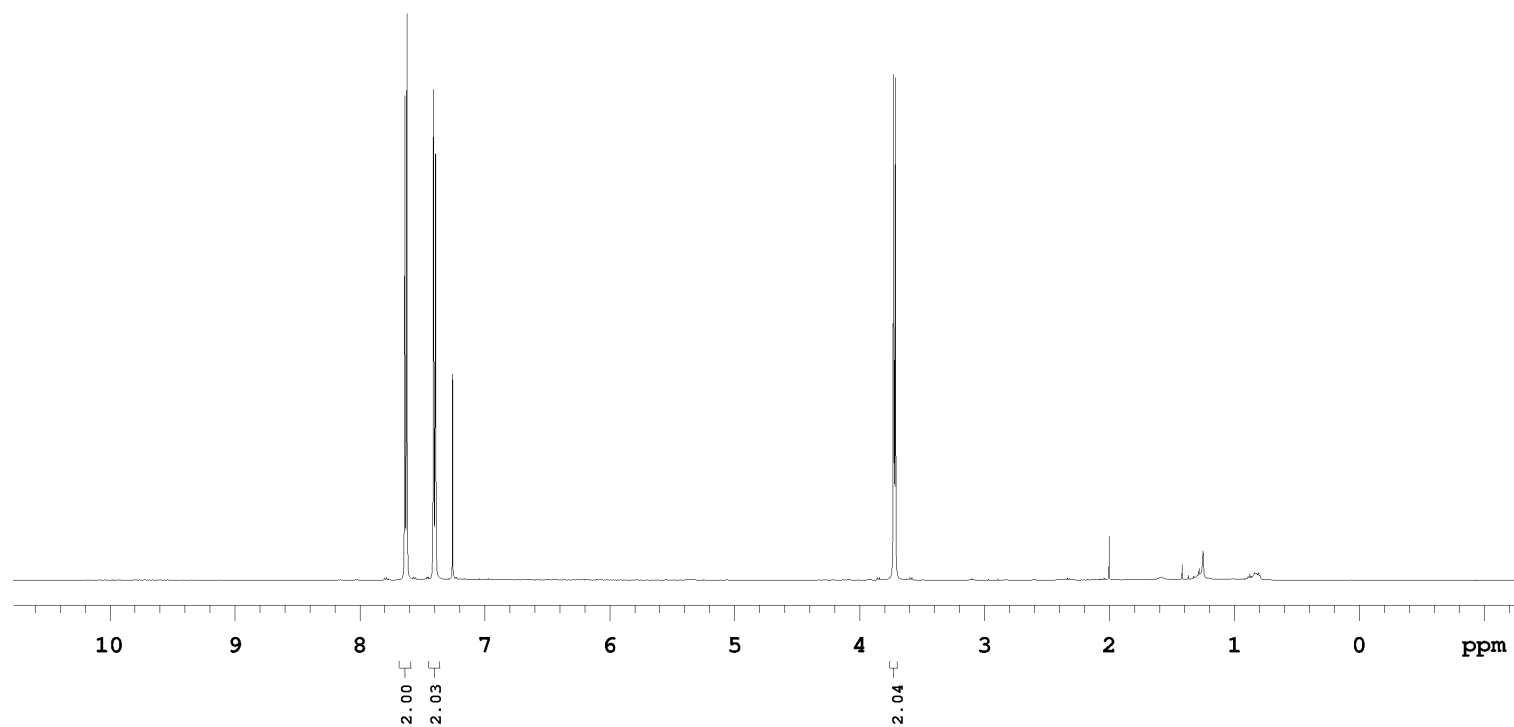
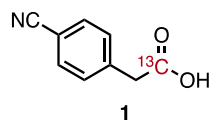
Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **25.04**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-11-31-A

499.787 MHz ^1H 1D in CDCl_3 (ref. to CDCl_3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jun 26 2019**
Pulse Sequence: **s2pul**

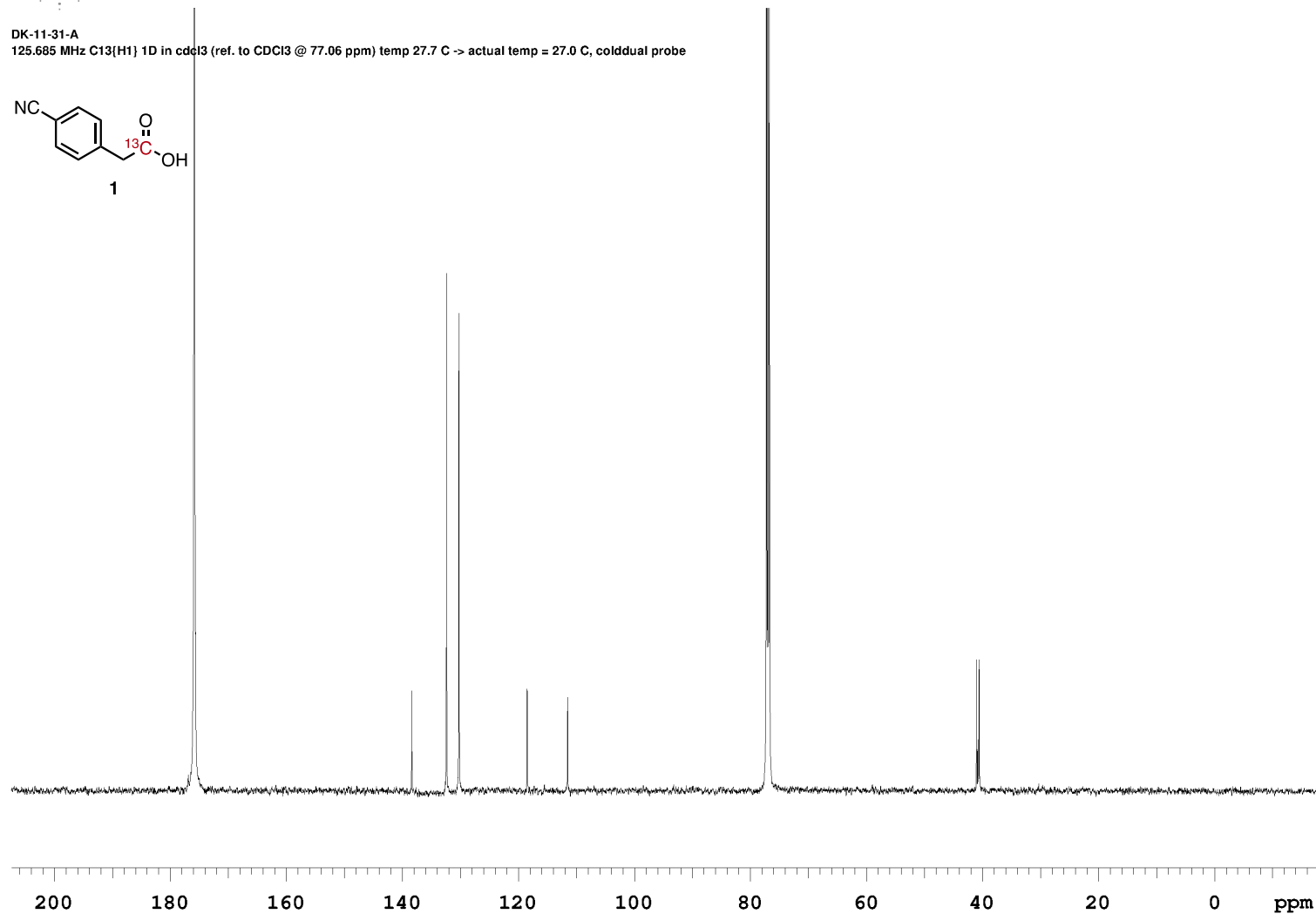
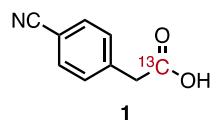
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **118.24**

Relaxation Delay(s): **1**
Completed Scans: **184**

DK-11-31-A

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, May 30 2019**
Pulse Sequence: **s2pul**

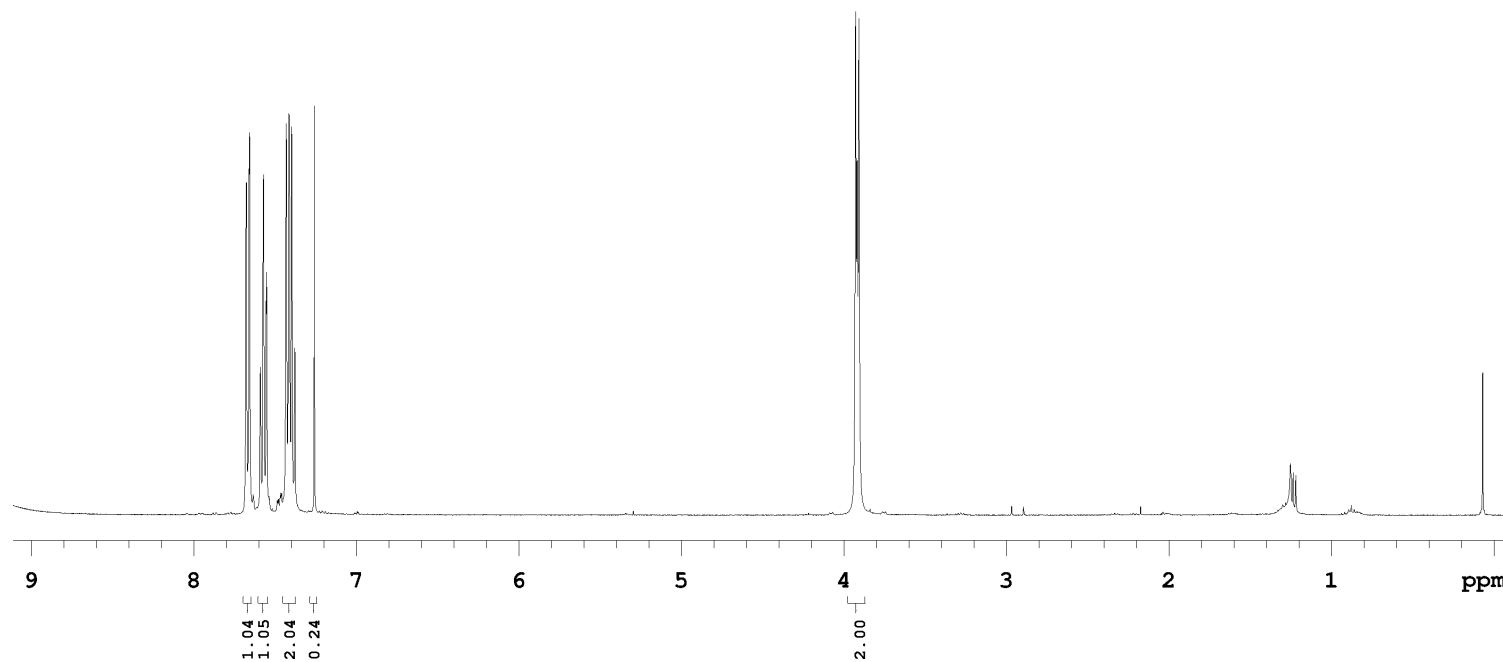
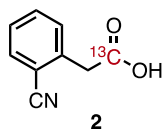
Sweep Width(Hz): **4607.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **15.41**

Relaxation Delay(s): **0.1**
Completed Scans **8**

OB-02-71-B

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe





Agilent Technologies

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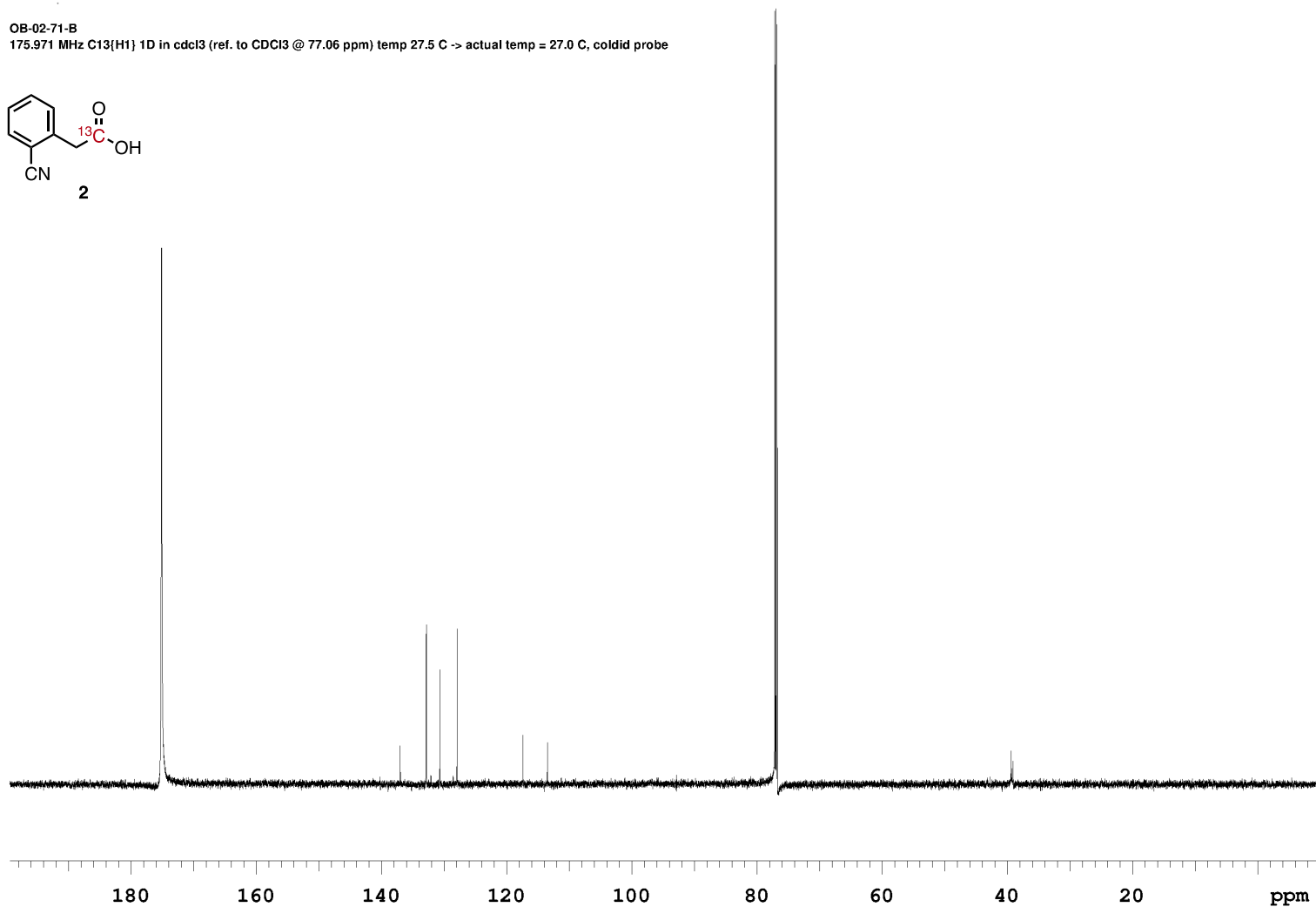
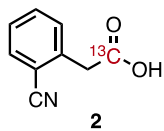
Recorded on: **v700, May 30 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **52**

OB-02-71-B
175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Jun 8 2019**
Pulse Sequence: **PRESAT**

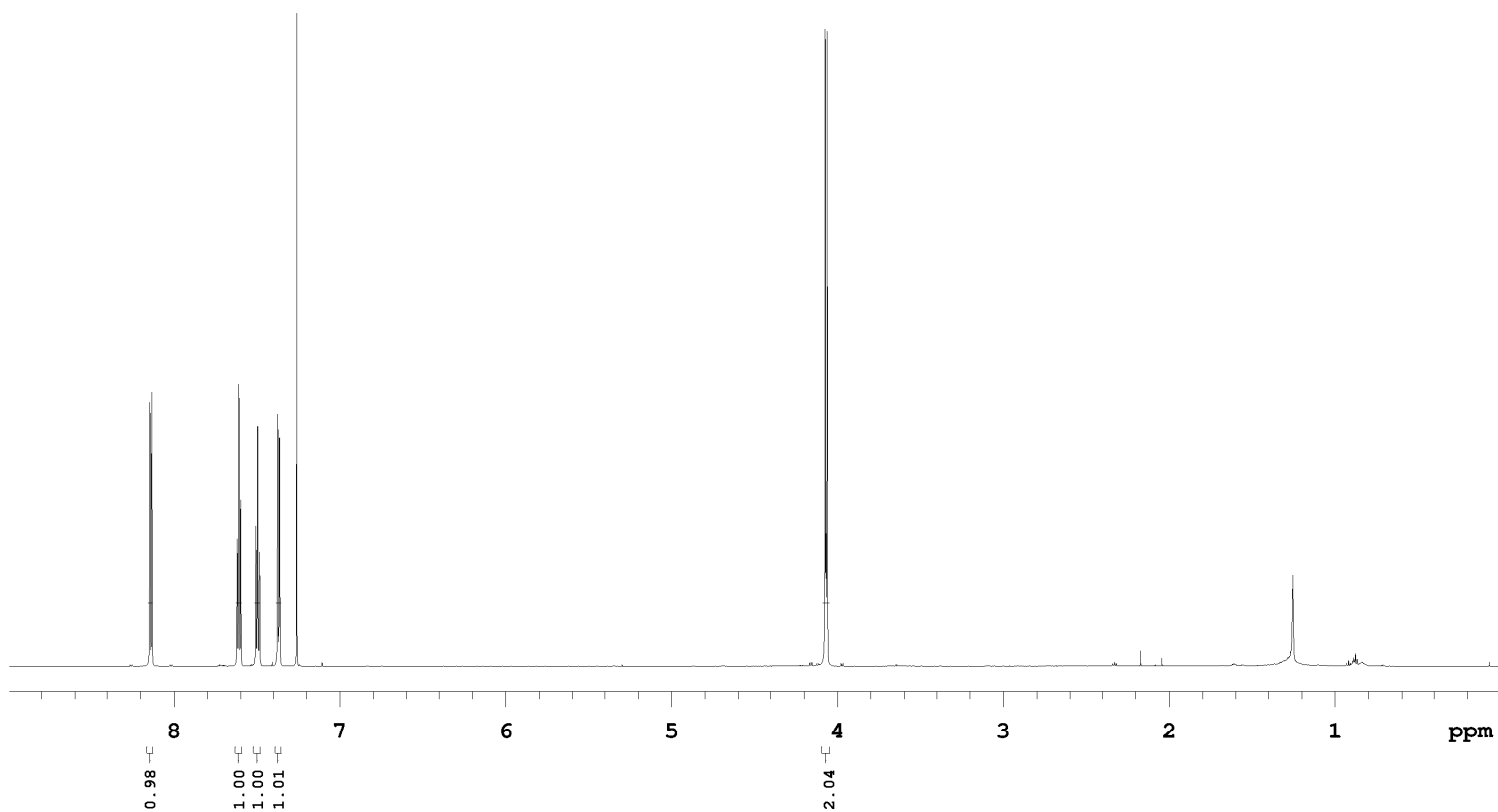
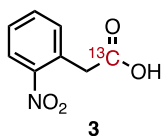
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.25**

Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL01-55

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jun 11 2019**
Pulse Sequence: **s2pul**

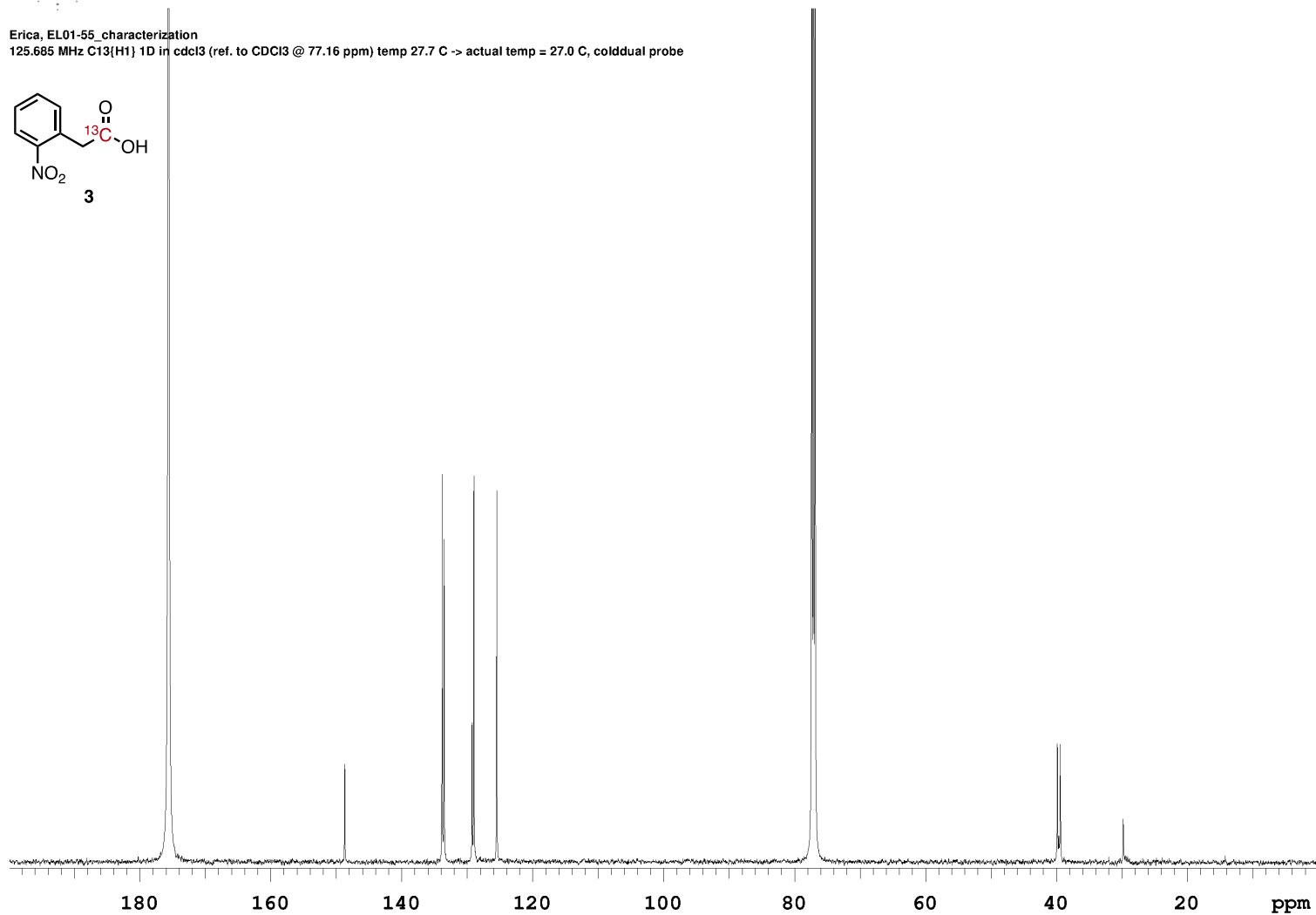
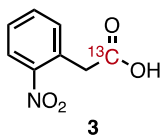
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.7**

Relaxation Delay(s): **1**
Completed Scans **680**

Erica, EL01-55_characterization

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **i400, May 28 2019**
Pulse Sequence: **presat**

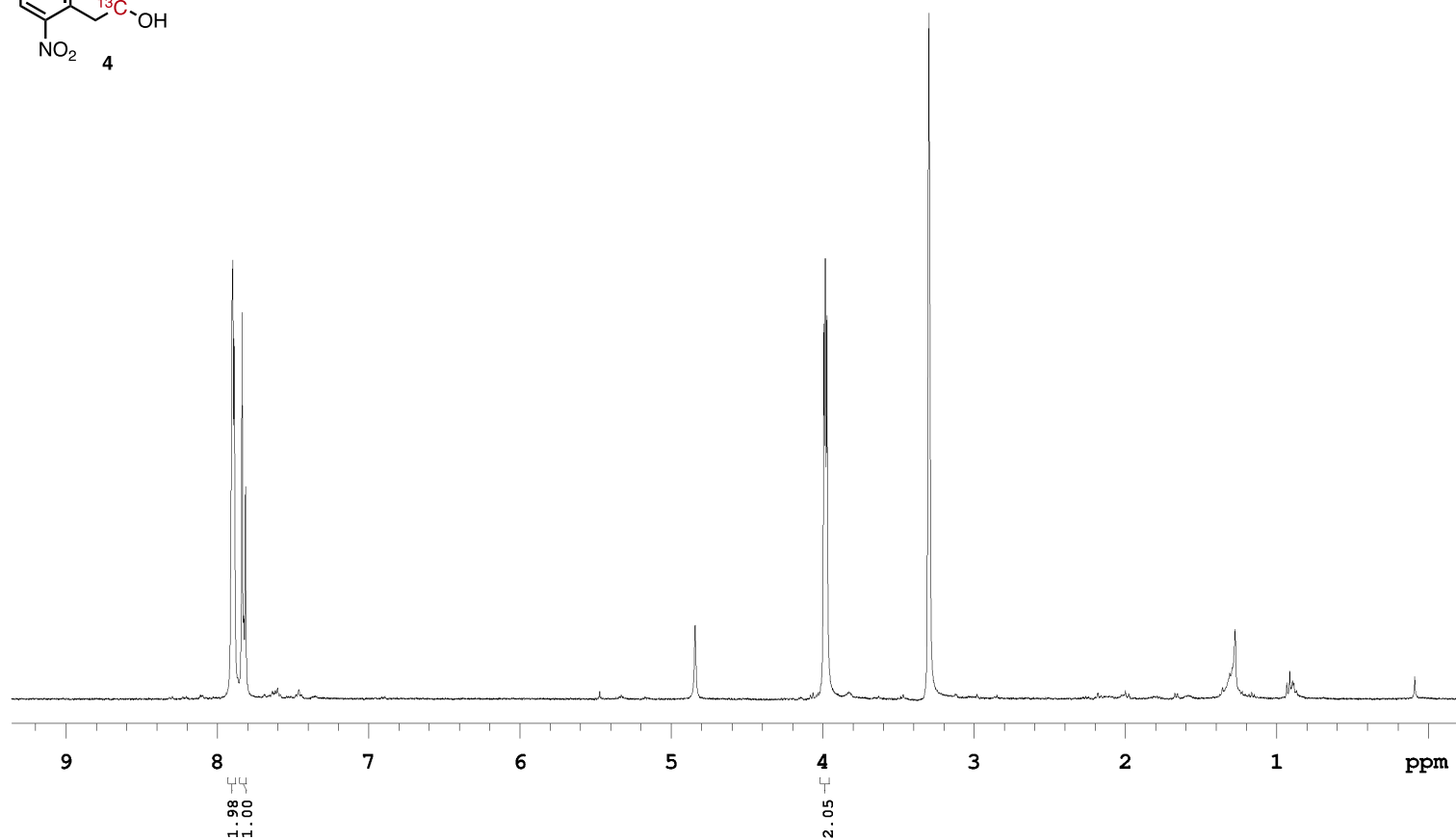
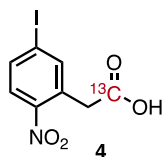
Sweep Width(Hz): **4801.92**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **2.999**
Hz per mm(Hz/mm): **15.91**

Relaxation Delay(s): **2**
Completed Scans **8**

OB-02-69-B

399.796 MHz H1 1D in cd3od (ref. to CD3OD @ 3.30 ppm) temp 26.5 C -> actual temp = 27.0 C, autoxdb probe





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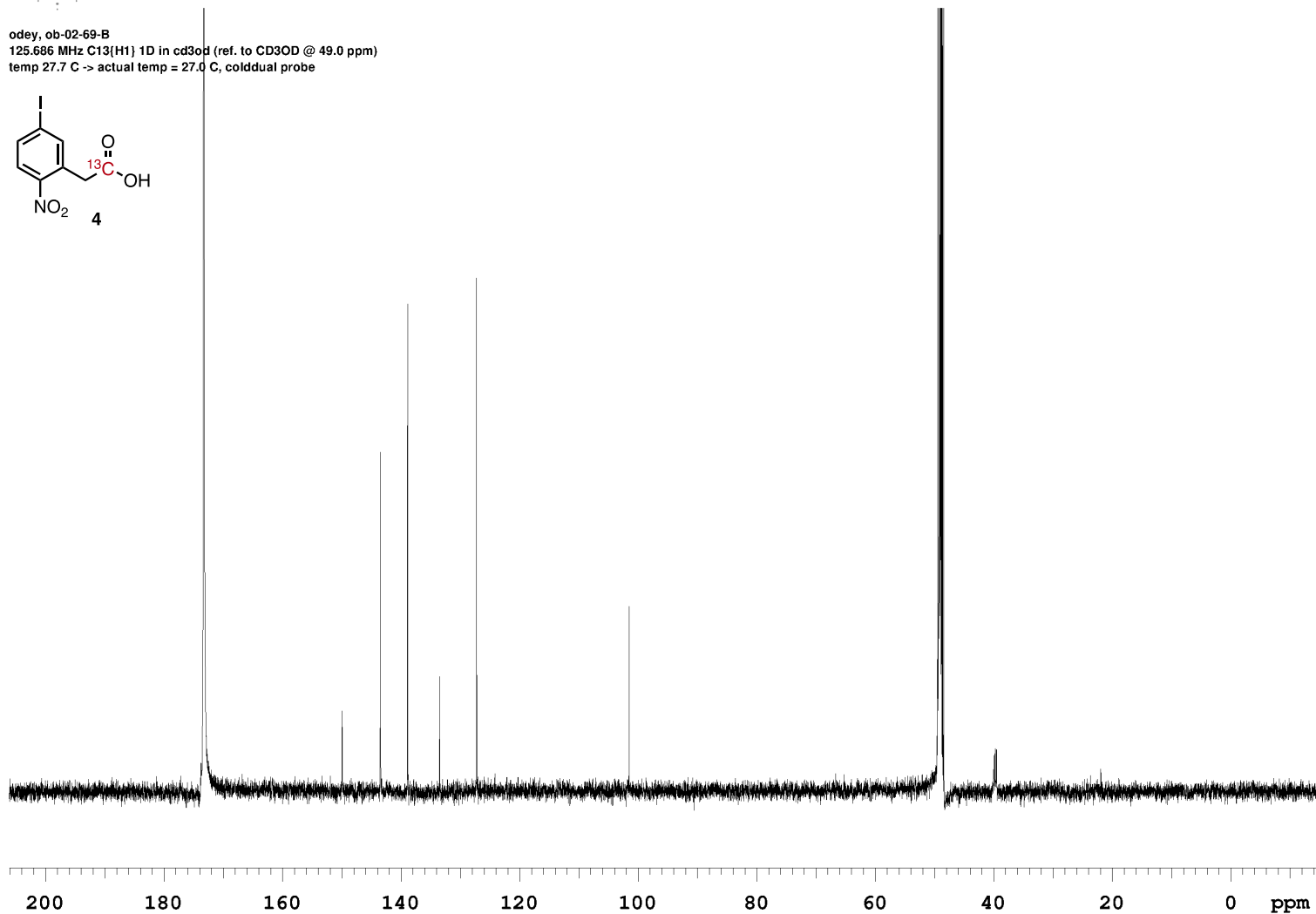
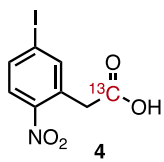
Recorded on: **u500, Jun 4 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **115.62**

Relaxation Delay(s): **1**
Completed Scans **128**

odey, ob-02-69-B
125.686 MHz C13{H1} 1D in cd3od (ref. to CD3OD @ 49.0 ppm)
temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Agilent Technologies

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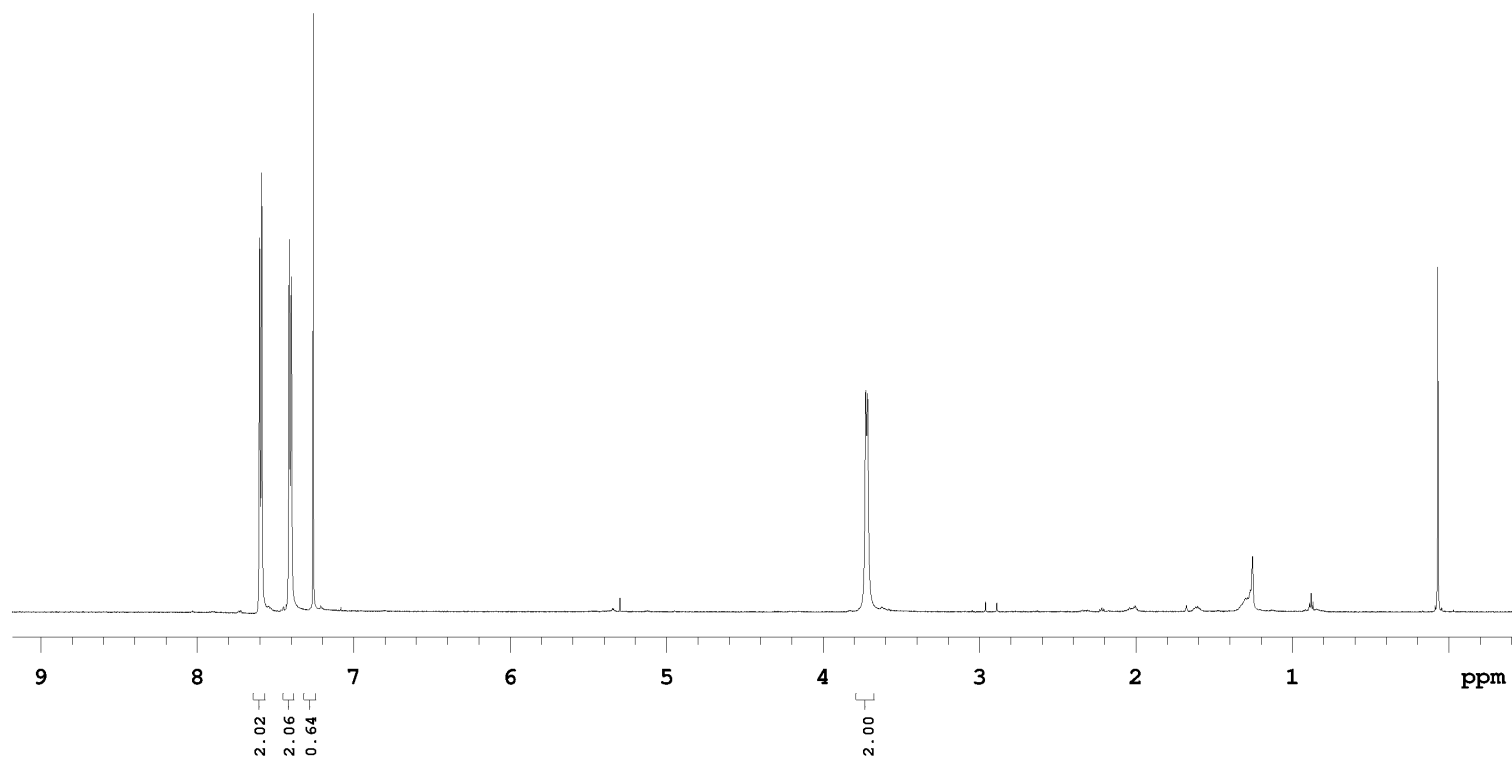
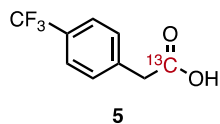
Recorded on: **i600, Jun 13 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **7183.91**
Digital Res.(Hz/pt): **0.11**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **24.01**

Relaxation Delay(s): **0.1**
Completed Scans **4**

OB-02-83-A 599.926 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.2 C -> actual temp = 27.0 C, autoxid probe





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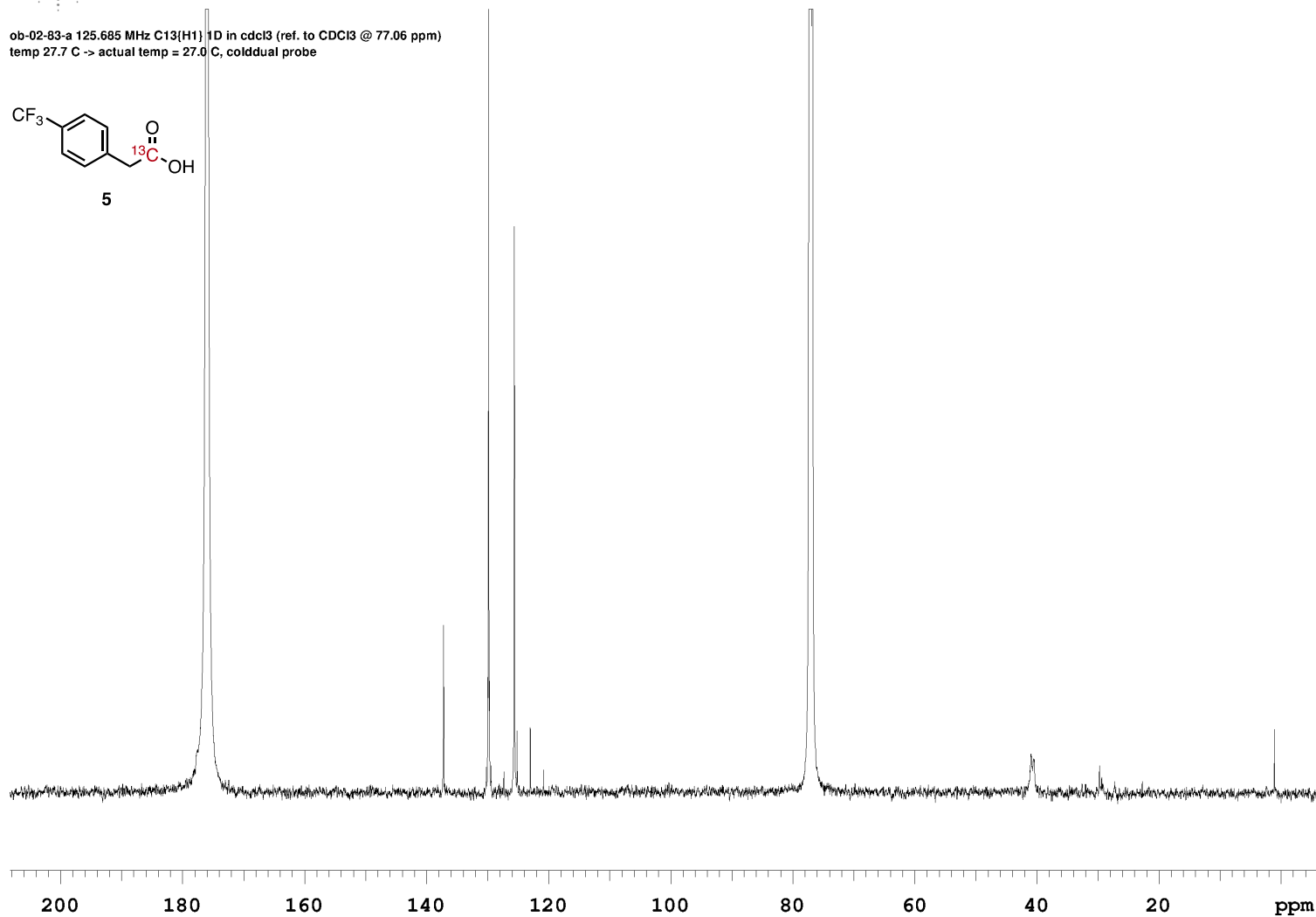
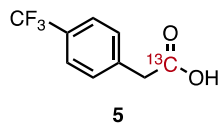
Recorded on: **u500, Jun 14 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **112.59**

Relaxation Delay(s): **1**
Completed Scans **1660**

ob-02-83-a 125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Department of Chemistry, University of Alberta

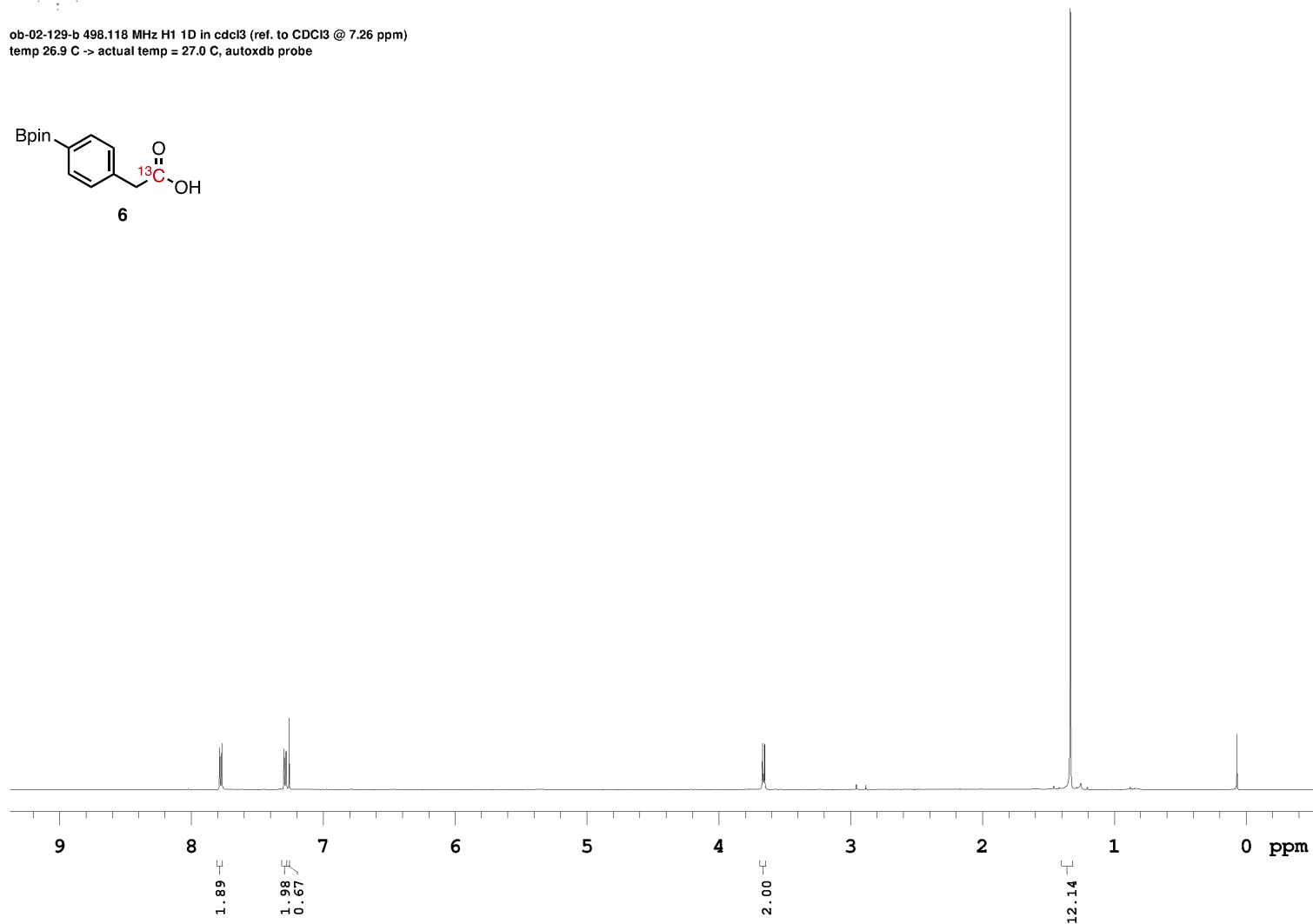
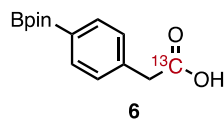
Recorded on: **ibd5, Jul 9 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6000.6**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.56**

Relaxation Delay(s): **0.1**
Completed Scans **16**

ob-02-129-b 498.118 MHz ¹H 1D in cdcl₃ (ref. to CDC13 @ 7.26 ppm)
temp 26.9 C -> actual temp = 27.0 C, autotdb probe





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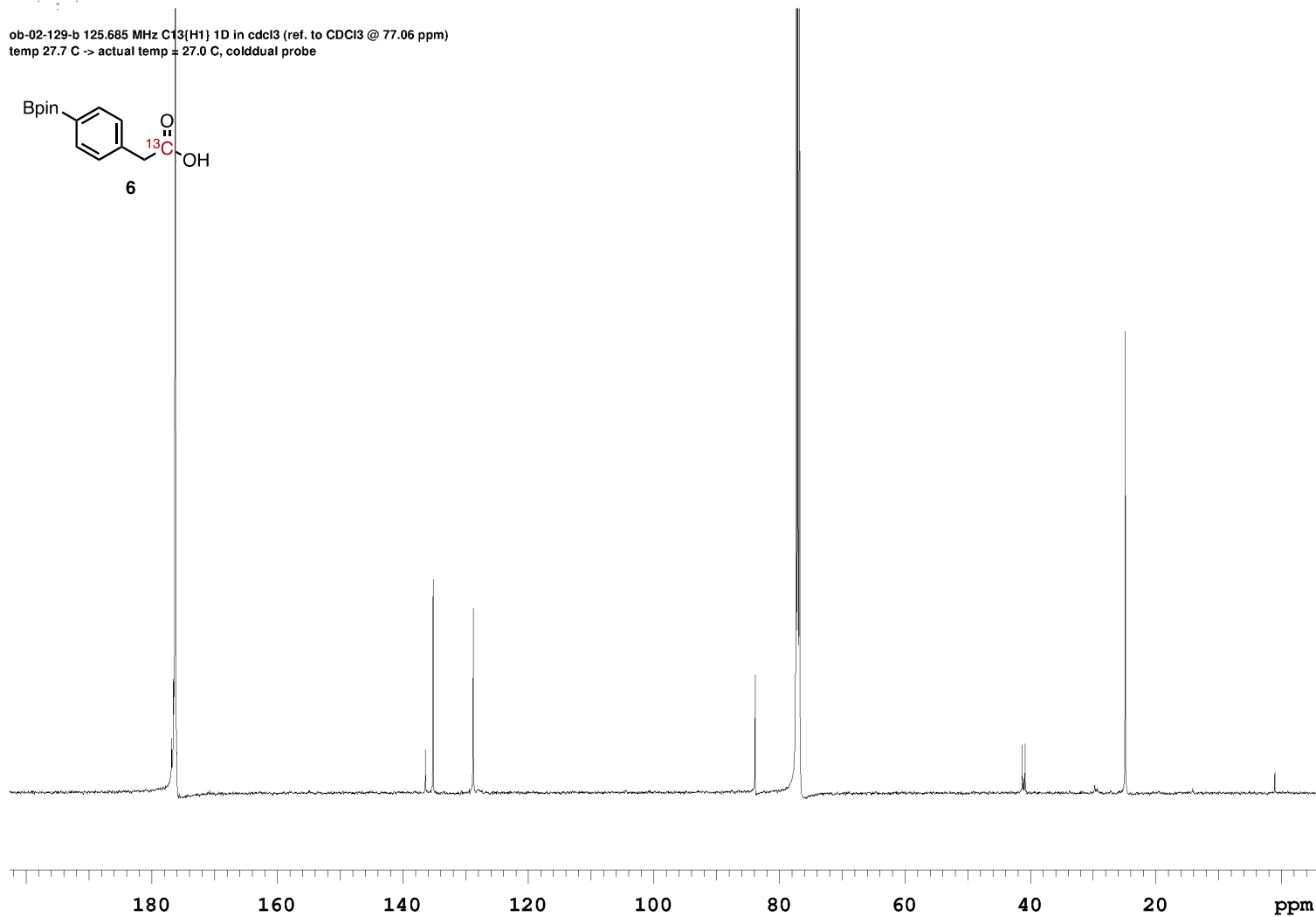
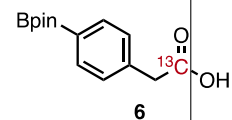
Recorded on: **u500, Jul 10 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **109.47**

Relaxation Delay(s): **1**
Completed Scans **1272**

ob-02-129-b 125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp ± 27.0 C, cold dual probe





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Recorded on: **ibd5, Jun 12 2019**

Sweep Width(Hz): **6000.6**

Acquisition Time(s): **5**

Relaxation Delay(s): **0.1**

Pulse Sequence: **s2pul**

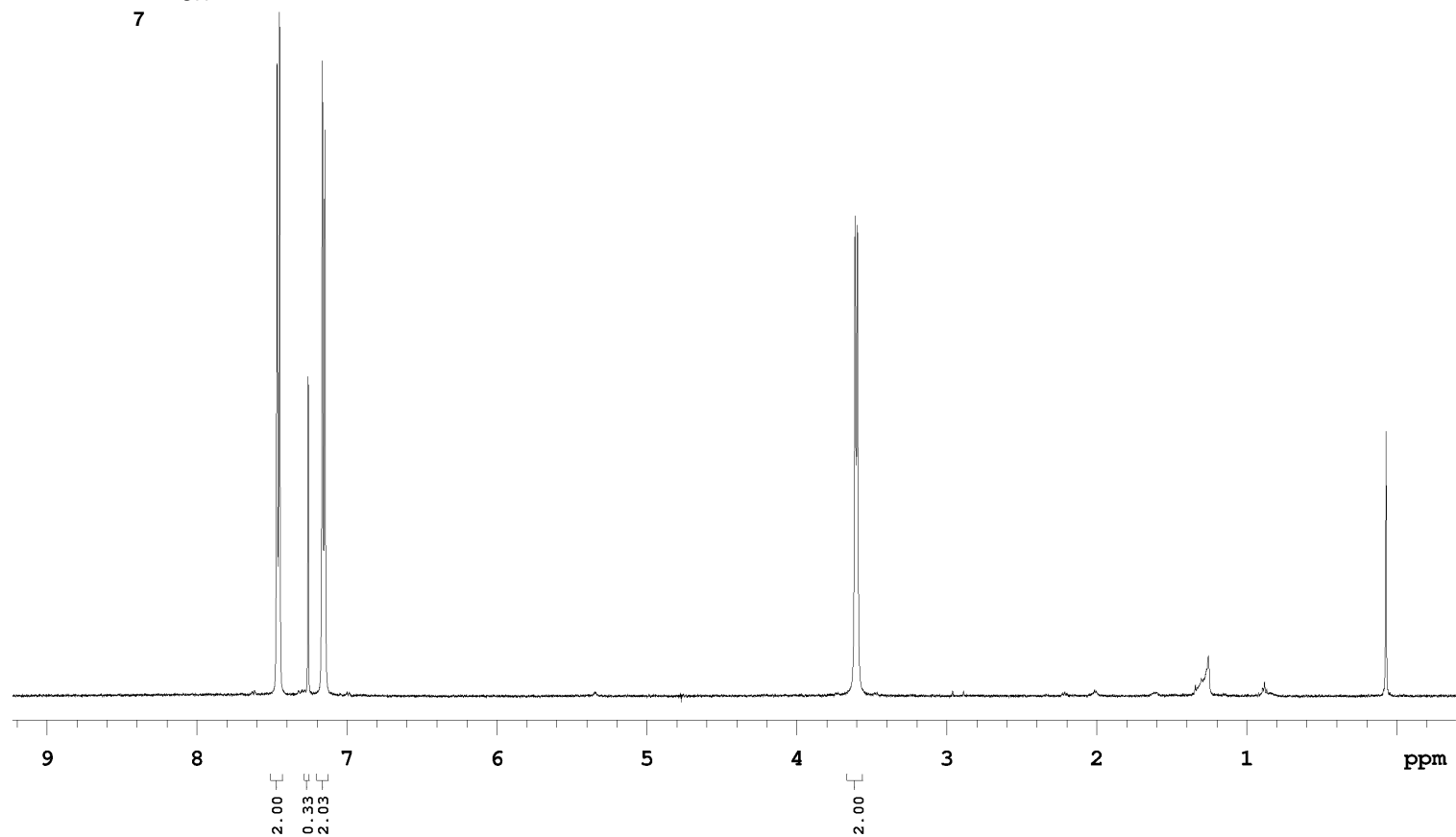
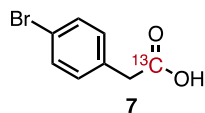
Digital Res.(Hz/pt): **0.09**

Hz per mm(Hz/mm): **19.98**

Completed Scans **4**

OB-02-81-A

498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe





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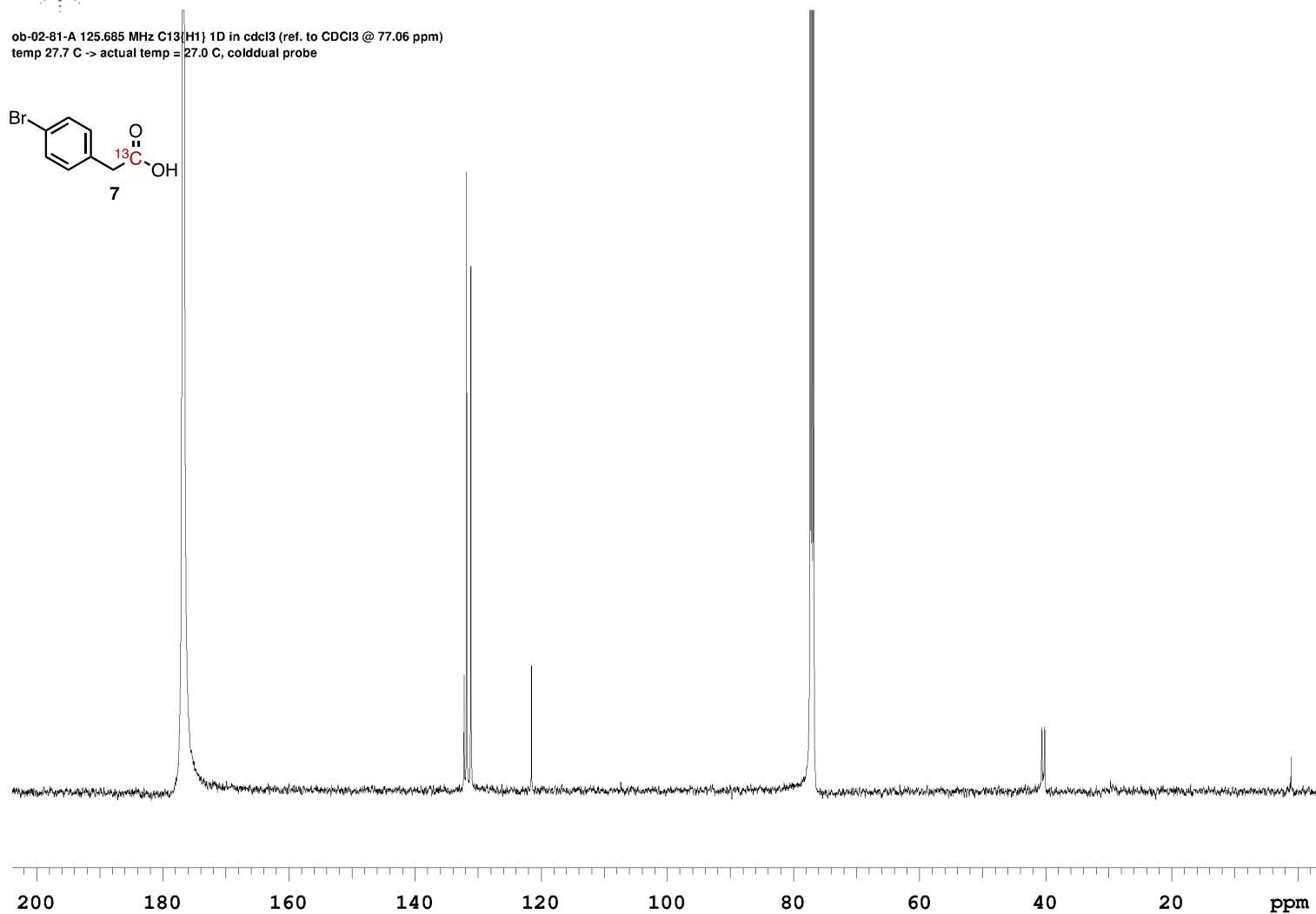
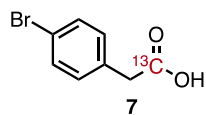
Recorded on: **u500, Jun 12 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **108.37**

Relaxation Delay(s): **1**
Completed Scans **292**

ob-02-81-A 125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **i400, Jun 15 2019**

Pulse Sequence: **s2pul**

Sweep Width(Hz): **4801.92**

Digital Res.(Hz/pt): **0.07**

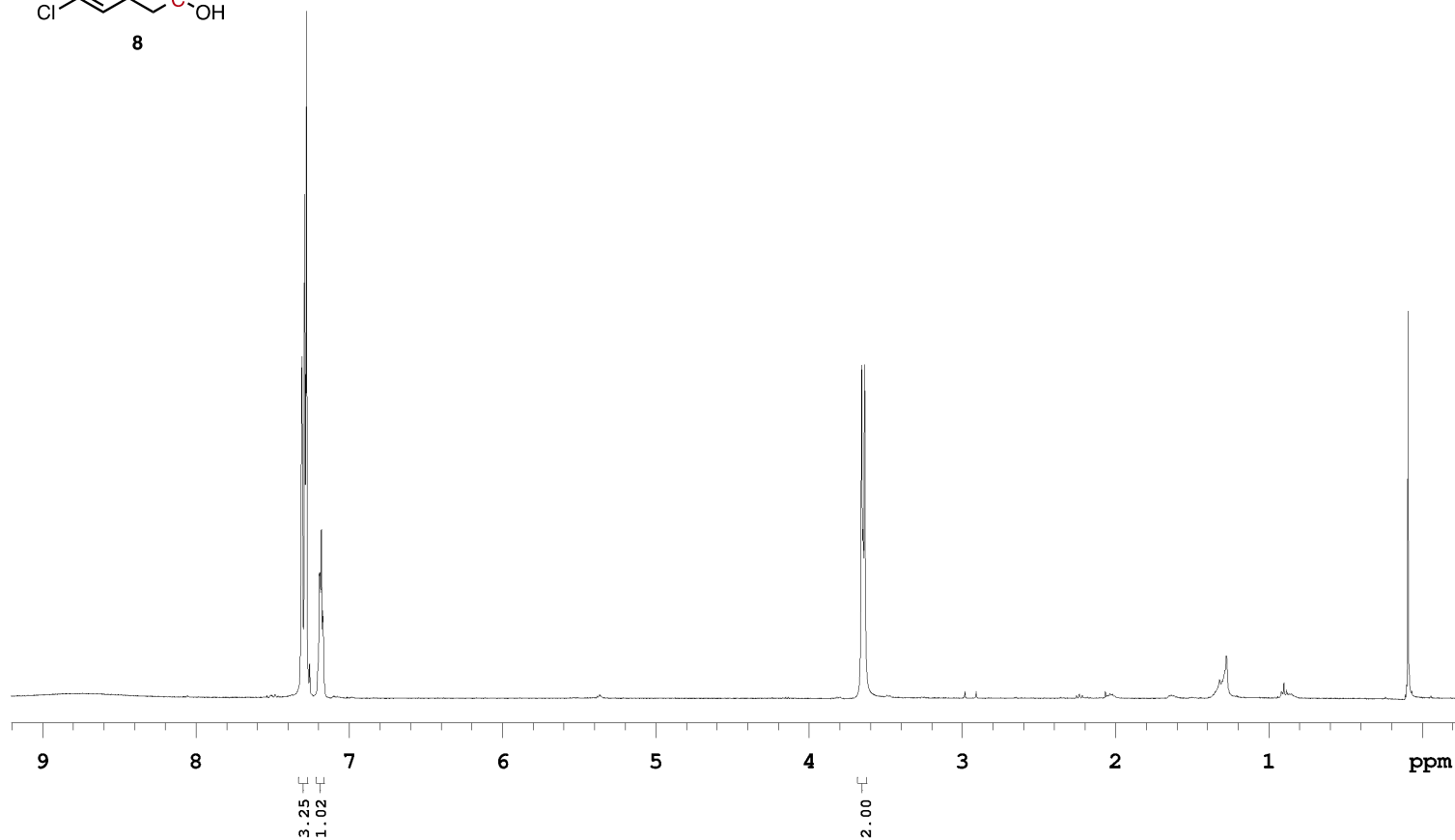
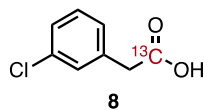
Acquisition Time(s): **4.998**

Hz per mm(Hz/mm): **15.76**

Relaxation Delay(s): **0.1**

Completed Scans **16**

ob-02-85-B 399.794 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.5 C -> actual temp = 27.0 C, autoxdb probe





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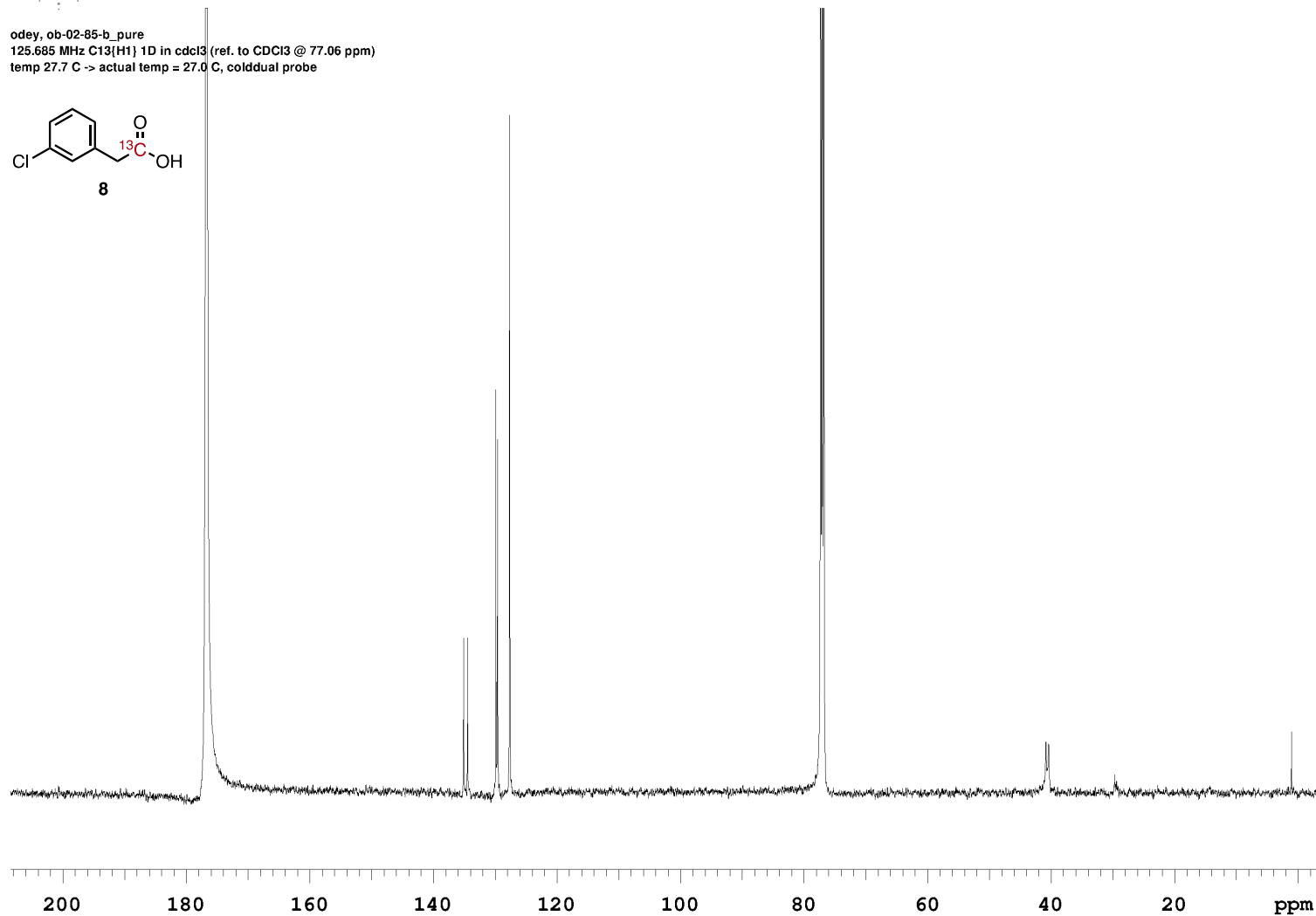
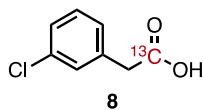
Recorded on: **u500, Jun 15 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **111.12**

Relaxation Delay(s): **1**
Completed Scans **308**

odey, ob-02-85-b_pure
125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **i400, May 30 2019**
Pulse Sequence: **s2pul**

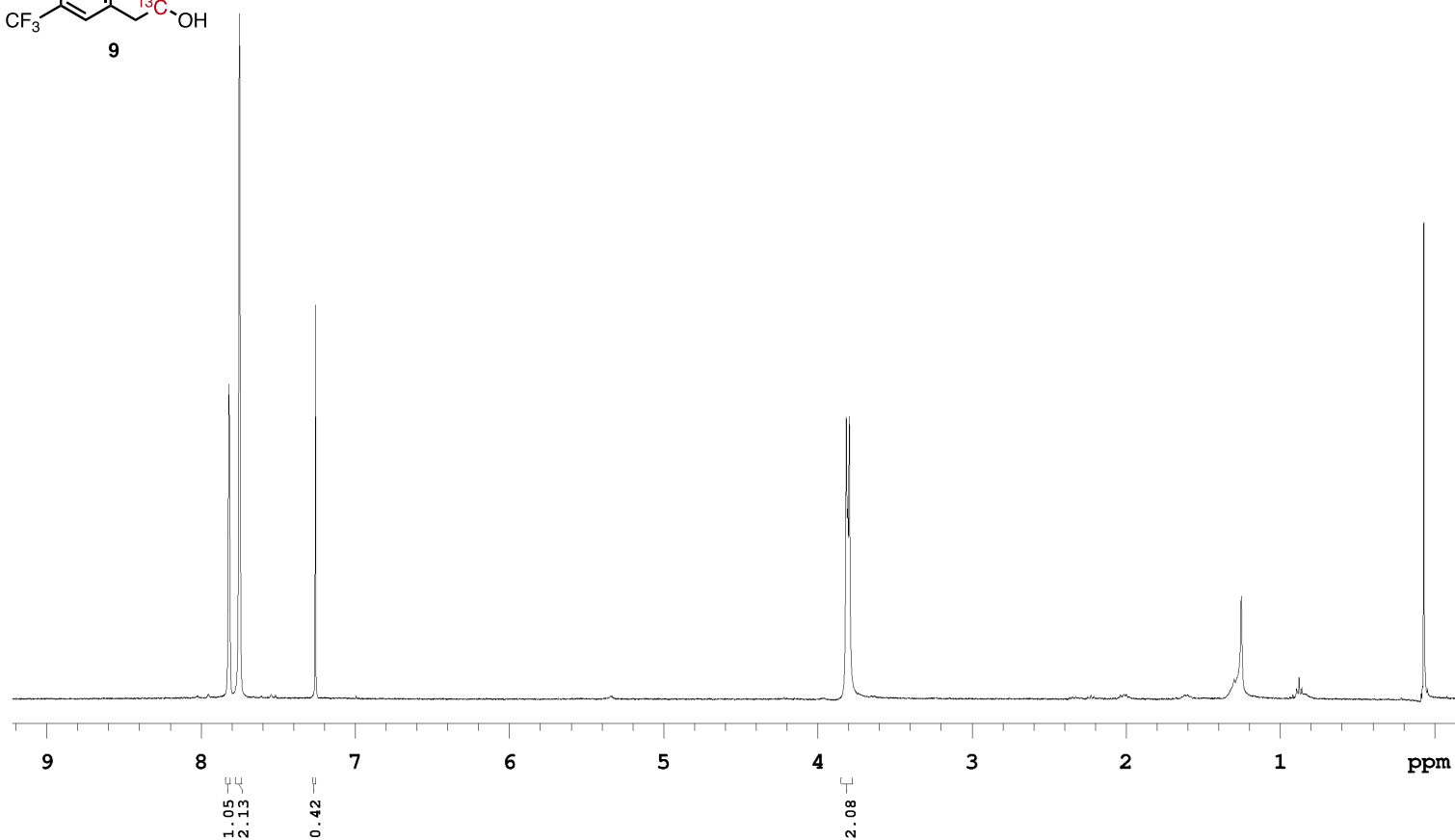
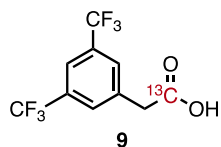
Sweep Width(Hz): **4801.92**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **4.998**
Hz per mm(Hz/mm): **15.64**

Relaxation Delay(s): **0.1**
Completed Scans **8**

OB-02-71-A

399.794 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.5 C -> actual temp = 27.0 C, autotdb probe





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Recorded on: **u500, May 31 2019**

Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**

Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**

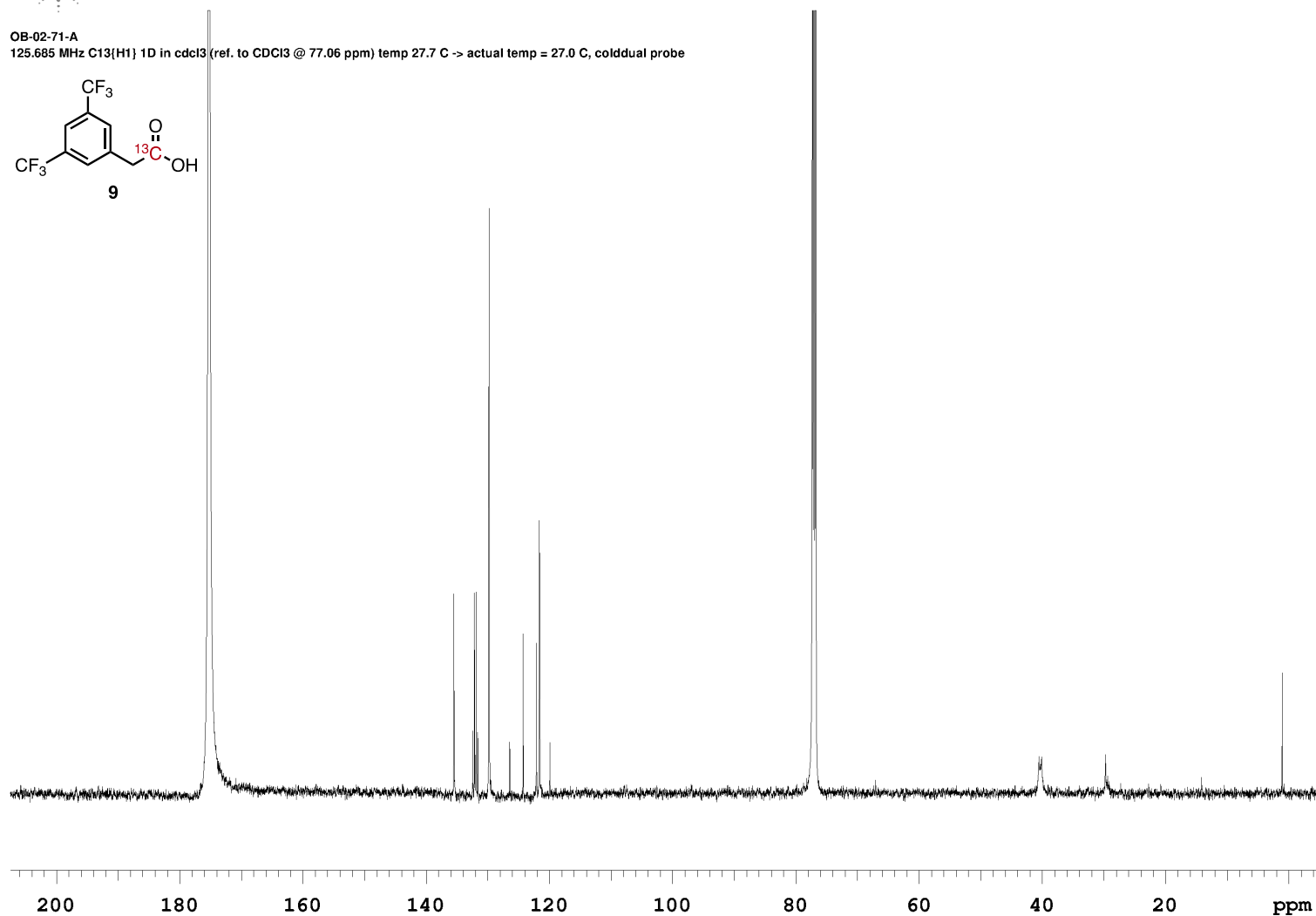
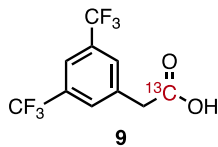
Hz per mm(Hz/mm): **111.3**

Relaxation Delay(s): **1**

Completed Scans **756**

OB-02-71-A

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Jun 7 2019**
Pulse Sequence: **PRESAT**

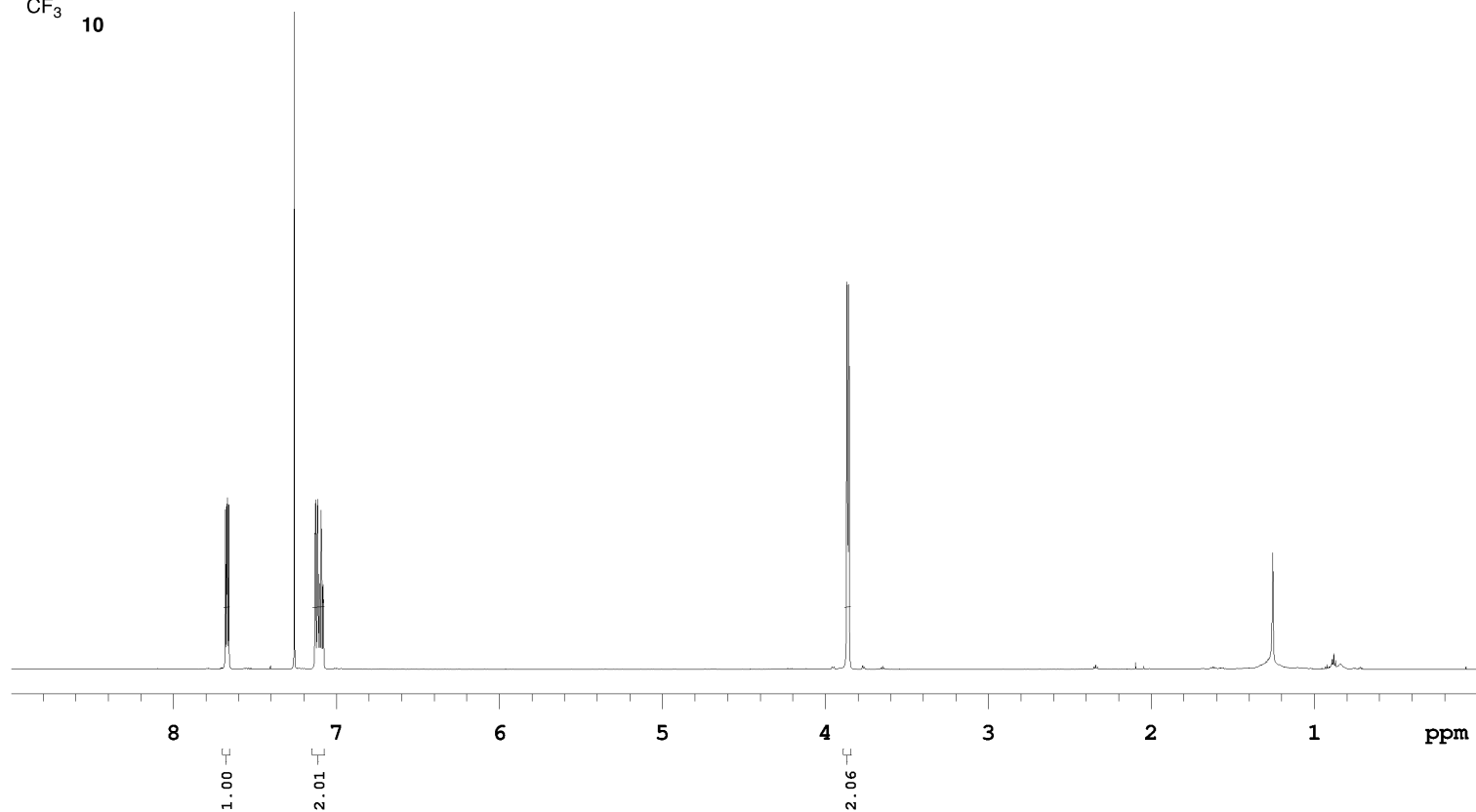
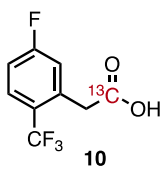
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.24**

Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL01-56_characterization

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **u500, Jun 11 2019**
Pulse Sequence: **s2pul**

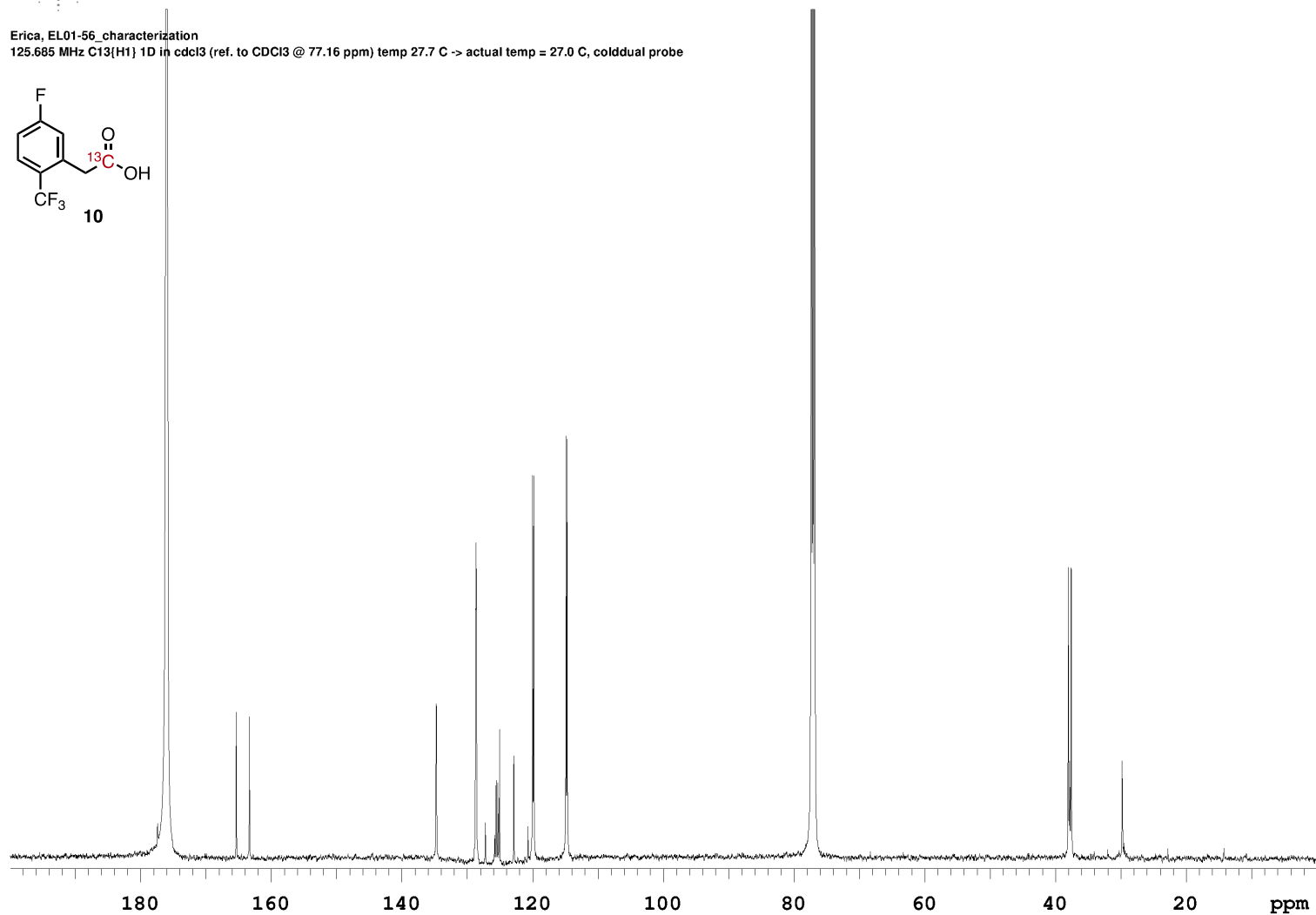
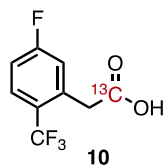
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.7**

Relaxation Delay(s): **1**
Completed Scans **1068**

Erica, EL01-56_characterization

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Jun 7 2019**

Sweep Width(Hz): **8389.26**

Acquisition Time(s): **5**

Relaxation Delay(s): **0.1**

Pulse Sequence: **PRESAT**

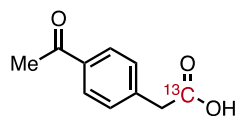
Digital Res.(Hz/pt): **0.13**

Hz per mm(Hz/mm): **26.26**

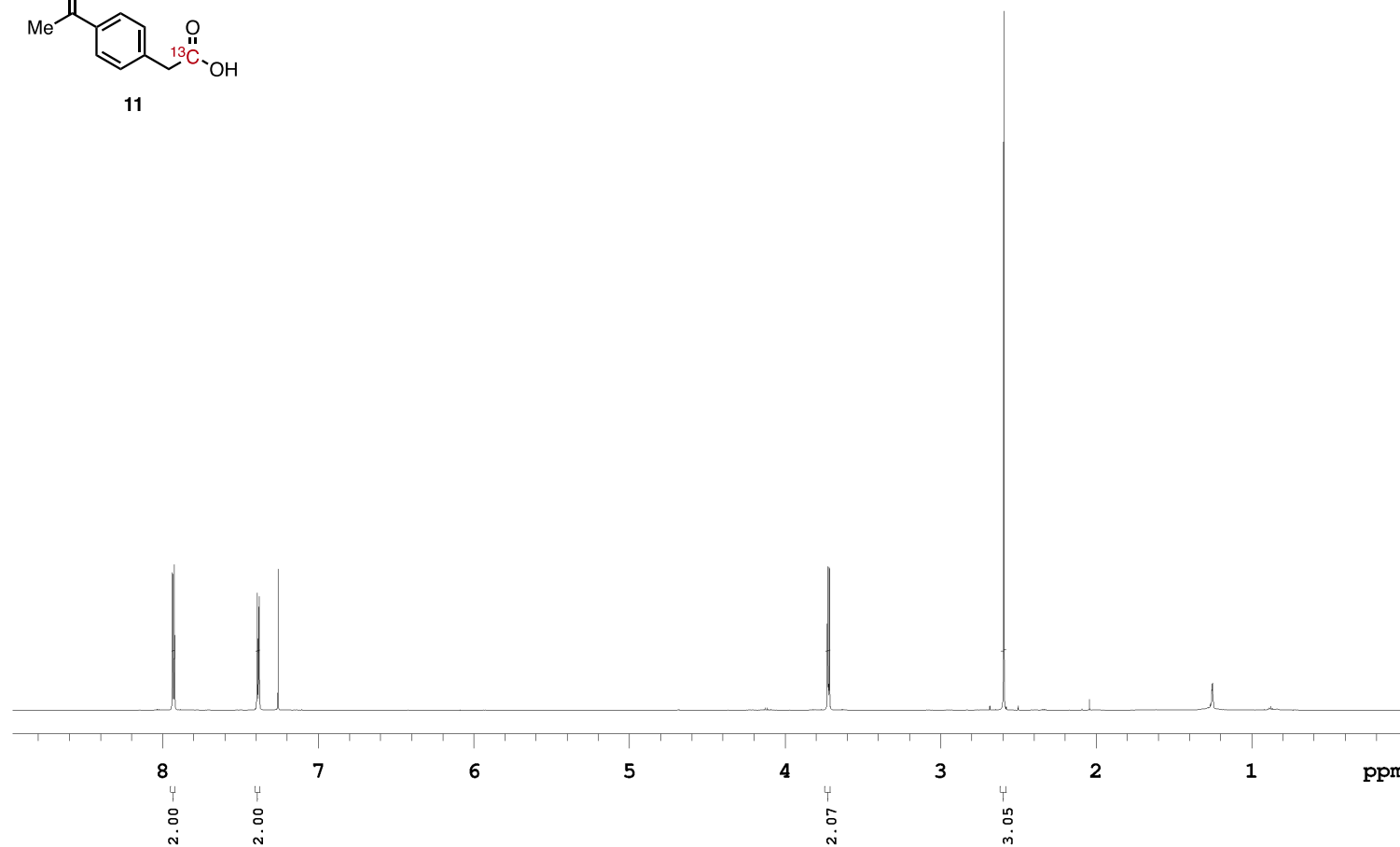
Completed Scans **8**

Erica, EL01-57A_characterization

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe



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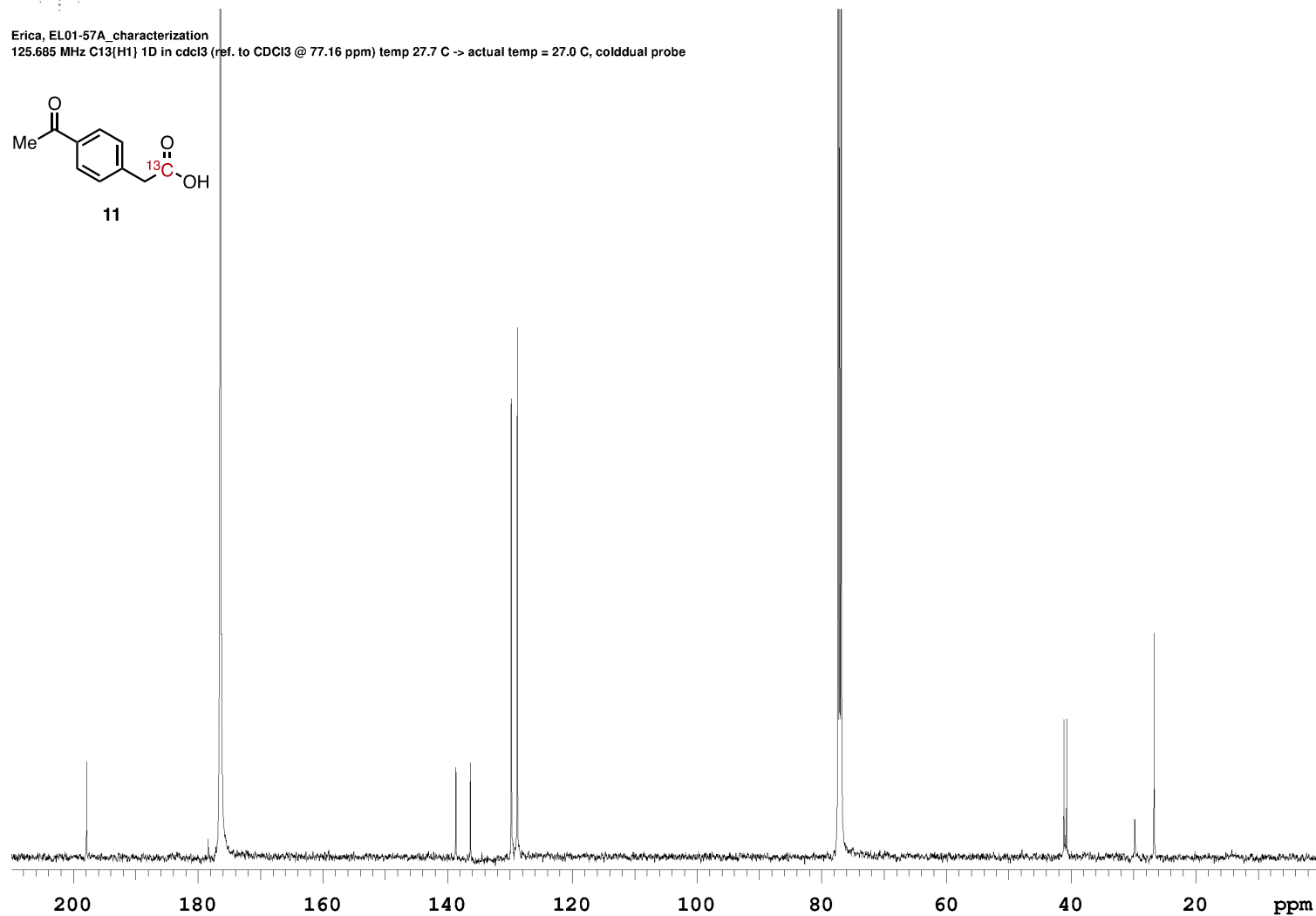
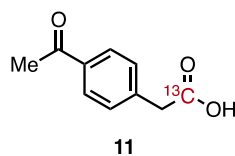
Recorded on: **u500, Jun 12 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **109.95**

Relaxation Delay(s): **1**
Completed Scans: **108**

Erica, EL01-57A_characterization
125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Jun 7 2019**
Pulse Sequence: **PRESAT**

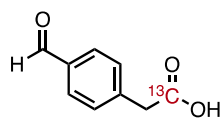
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.24**

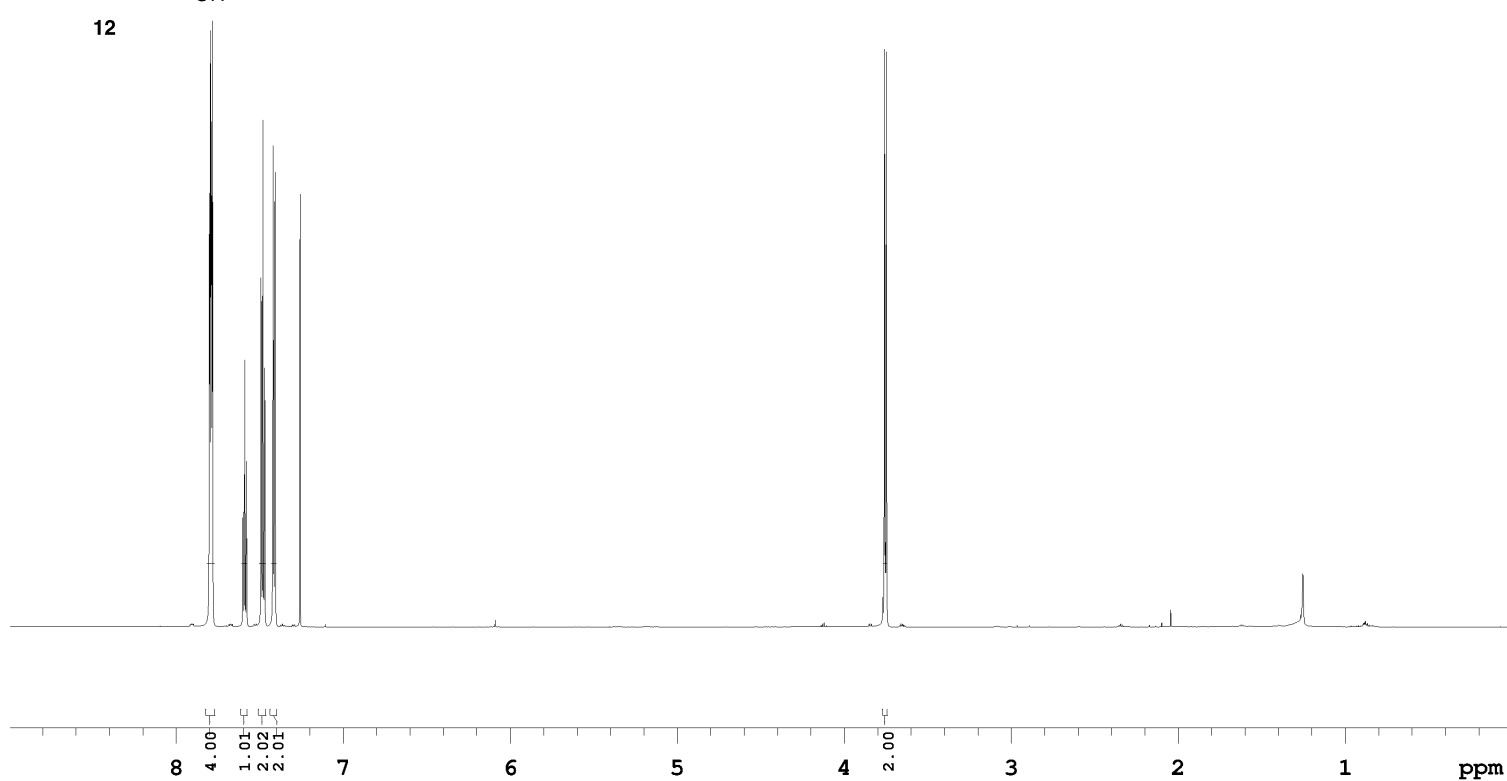
Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL01-57B_characterization

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe



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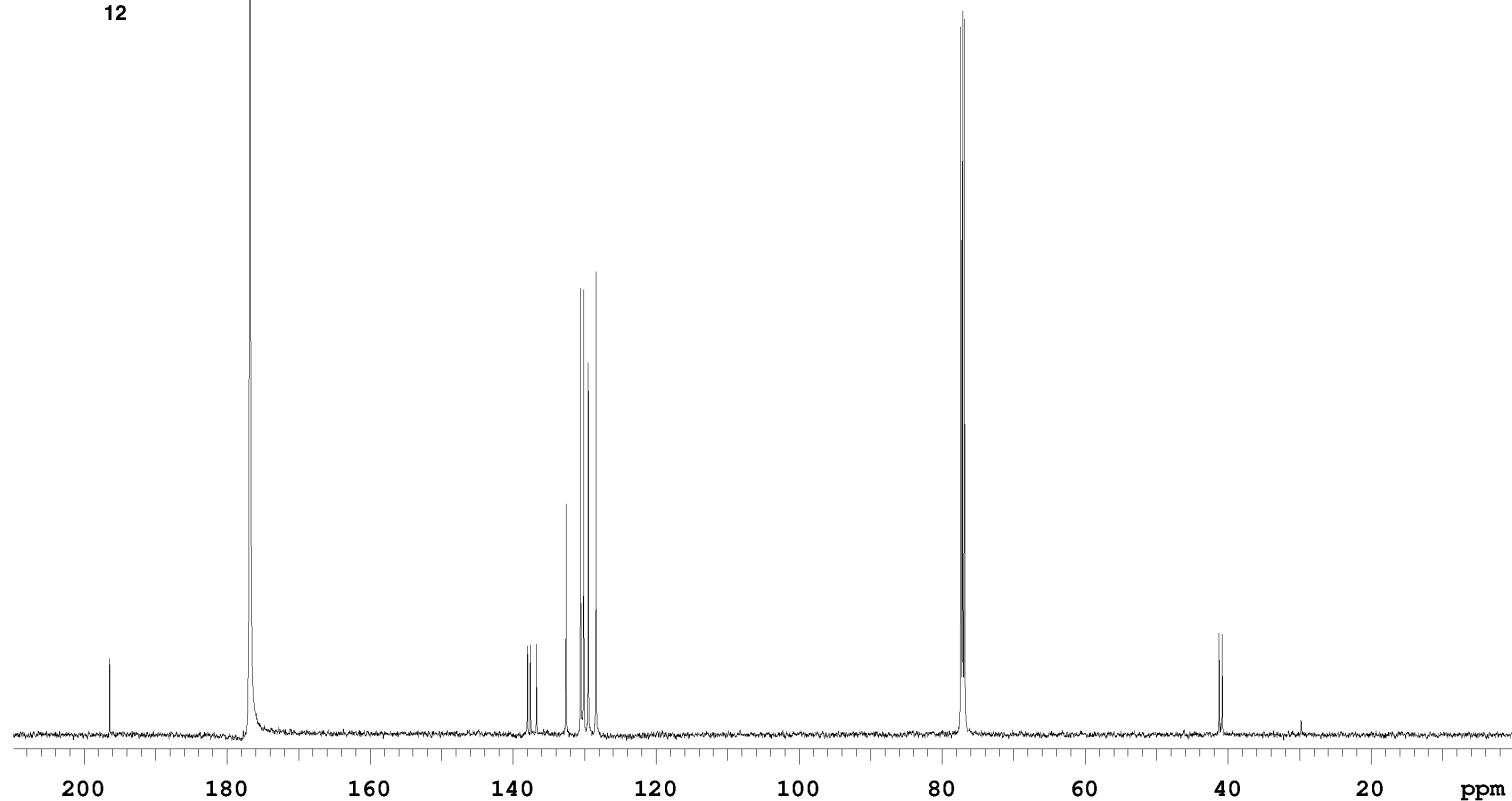
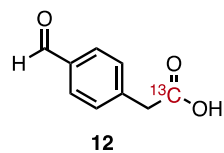
Recorded on: **u500, Jun 12 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **109.95**

Relaxation Delay(s): **1**
Completed Scans **36**

Erica, EL01-57B_characterization
125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Jun 7 2019**
Pulse Sequence: **PRESAT**

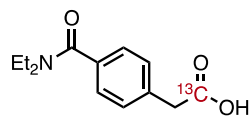
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.26**

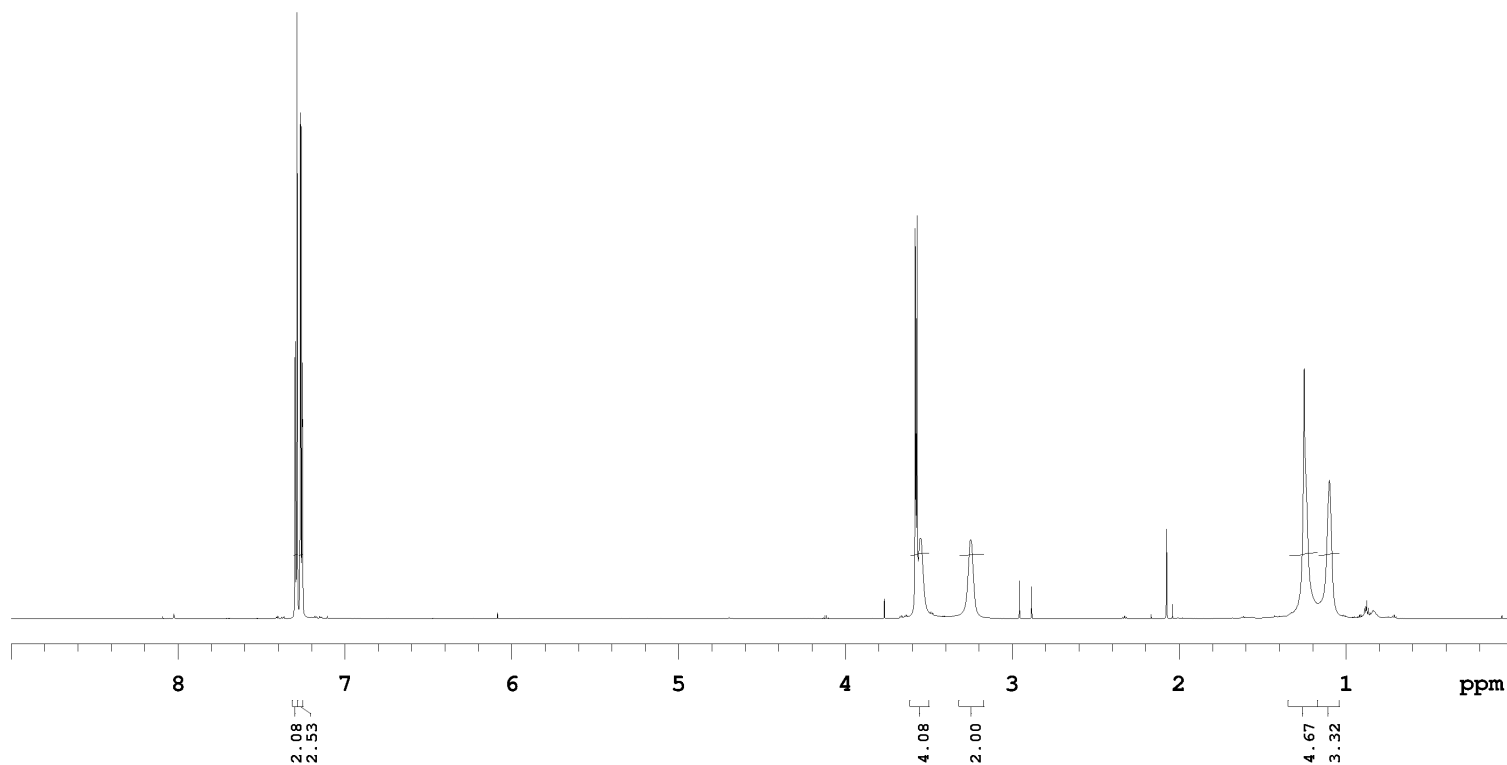
Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL01-58B_characterization

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe



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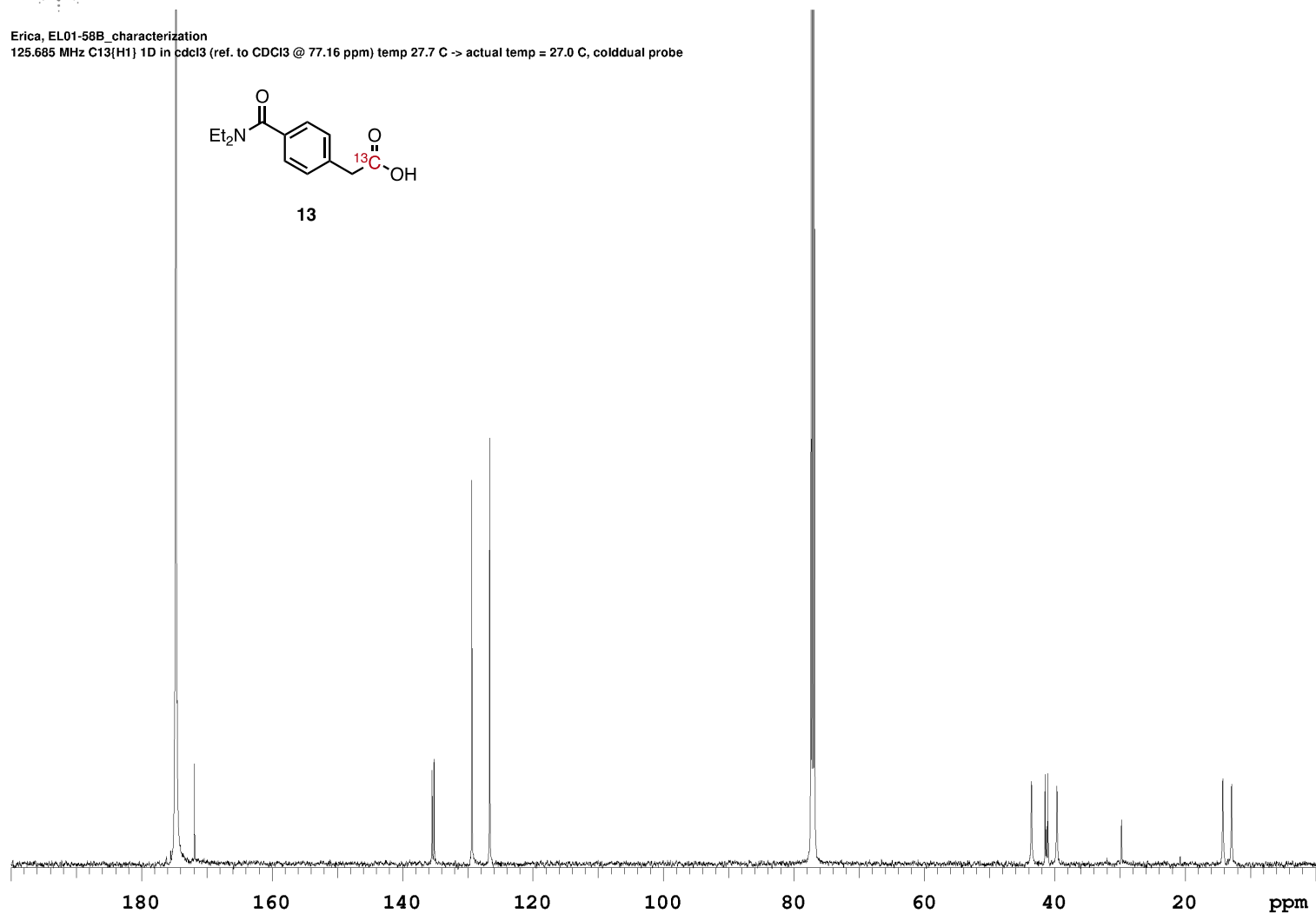
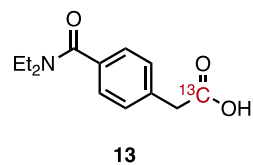
Recorded on: **u500, Jun 13 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.78**

Relaxation Delay(s): **1**
Completed Scans: **104**

Erica, EL01-58B_characterization
125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Jun 7 2019**
Pulse Sequence: **PRESAT**

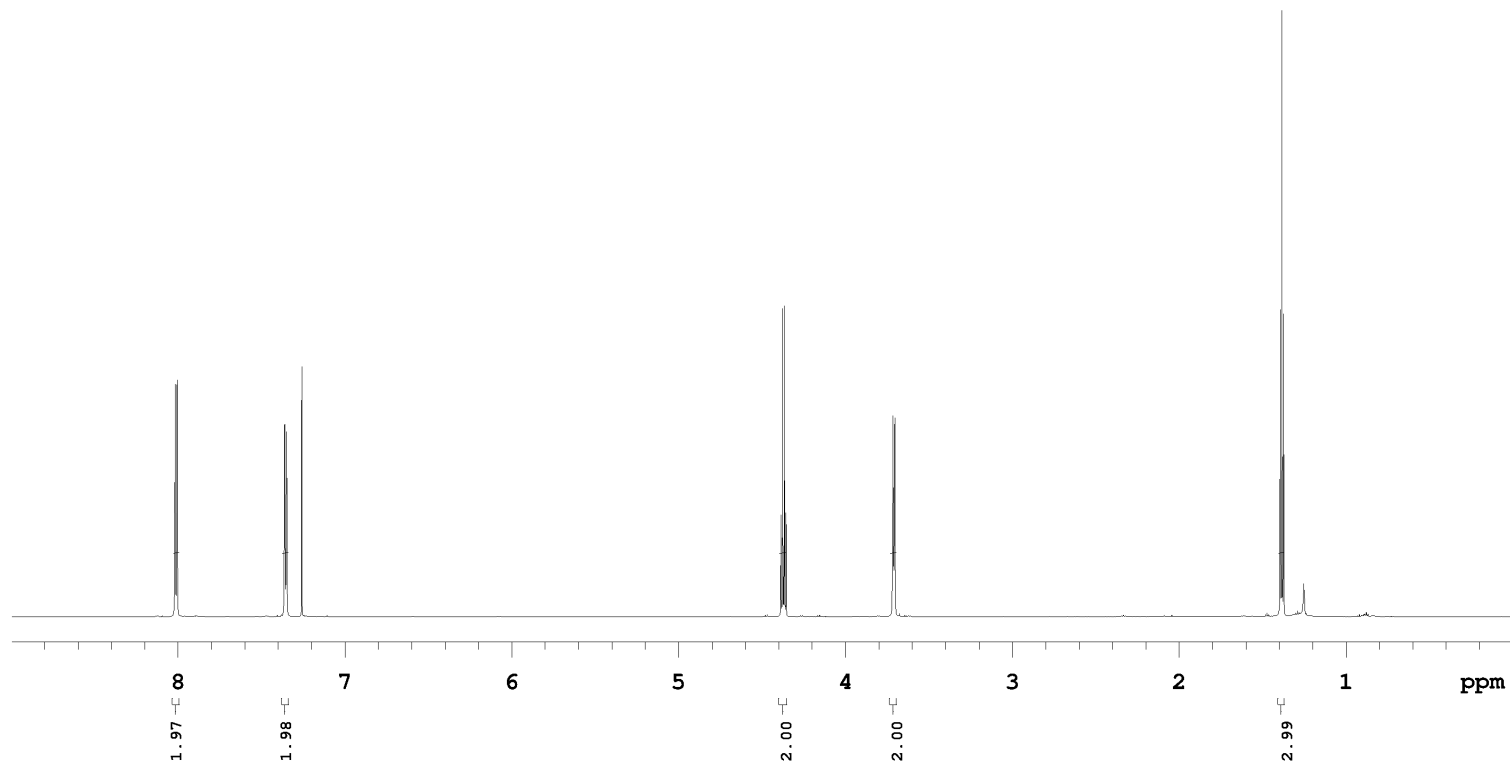
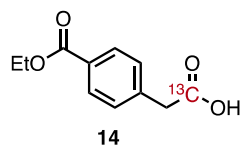
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.26**

Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL01-58A_characterization

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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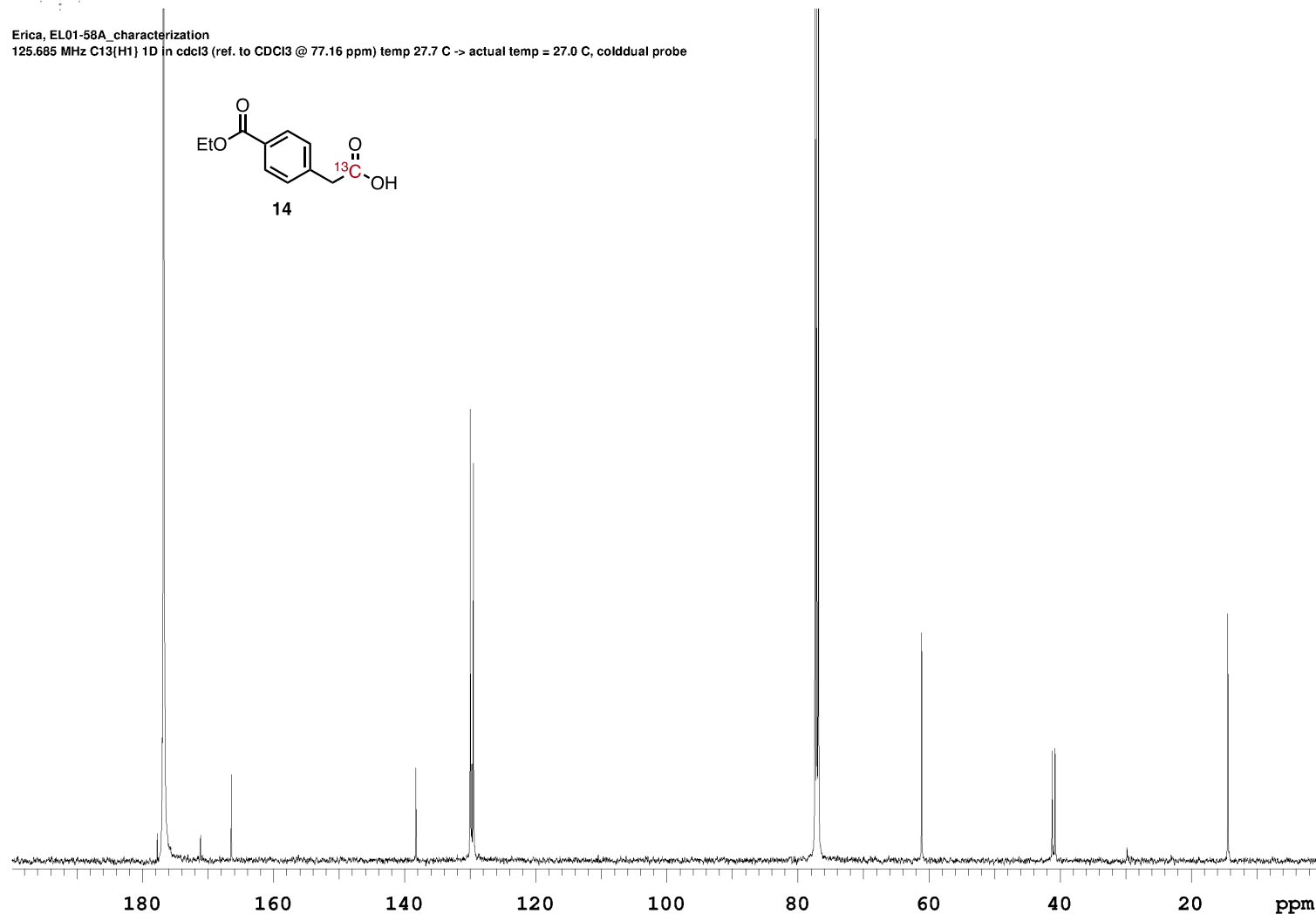
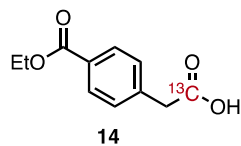
Recorded on: **u500, Jun 13 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.7**

Relaxation Delay(s): **1**
Completed Scans **60**

Erica, EL01-58A_characterization
125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **1600, May 31 2019**
Pulse Sequence: **presat**

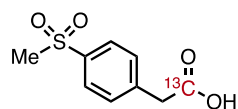
Sweep Width(Hz): **7183.91**
Digital Res.(Hz/pt): **0.11**

Acquisition Time(s): **3**
Hz per mm(Hz/mm): **23.44**

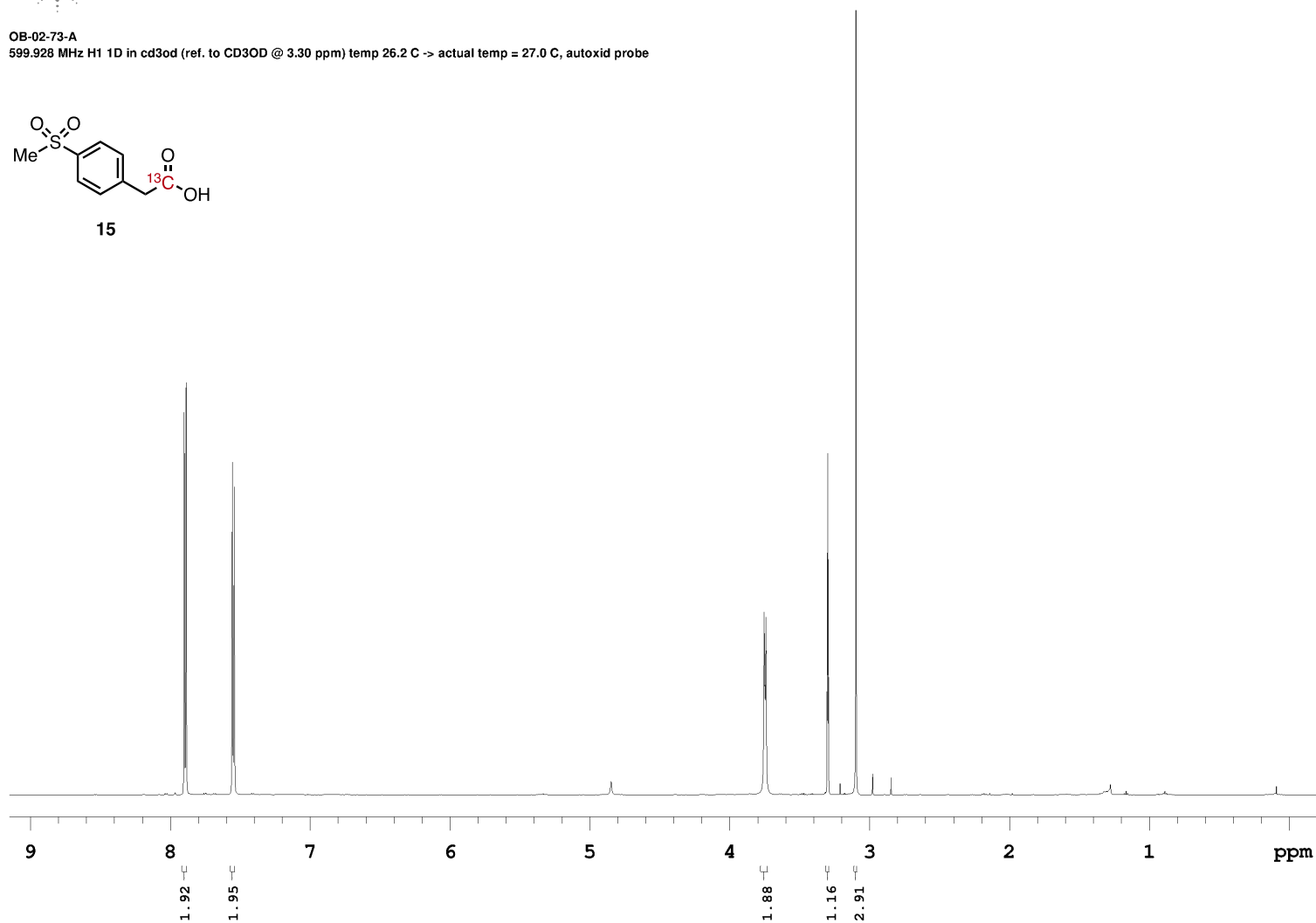
Relaxation Delay(s): **2**
Completed Scans **16**

OB-02-73-A

599.928 MHz H1 1D in cd3od (ref. to CD3OD @ 3.30 ppm) temp 26.2 C -> actual temp = 27.0 C, autoxid probe



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Recorded on: **u500, May 31 2019**
Pulse Sequence: **s2pul**

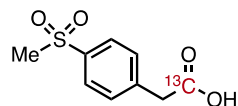
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **110.11**

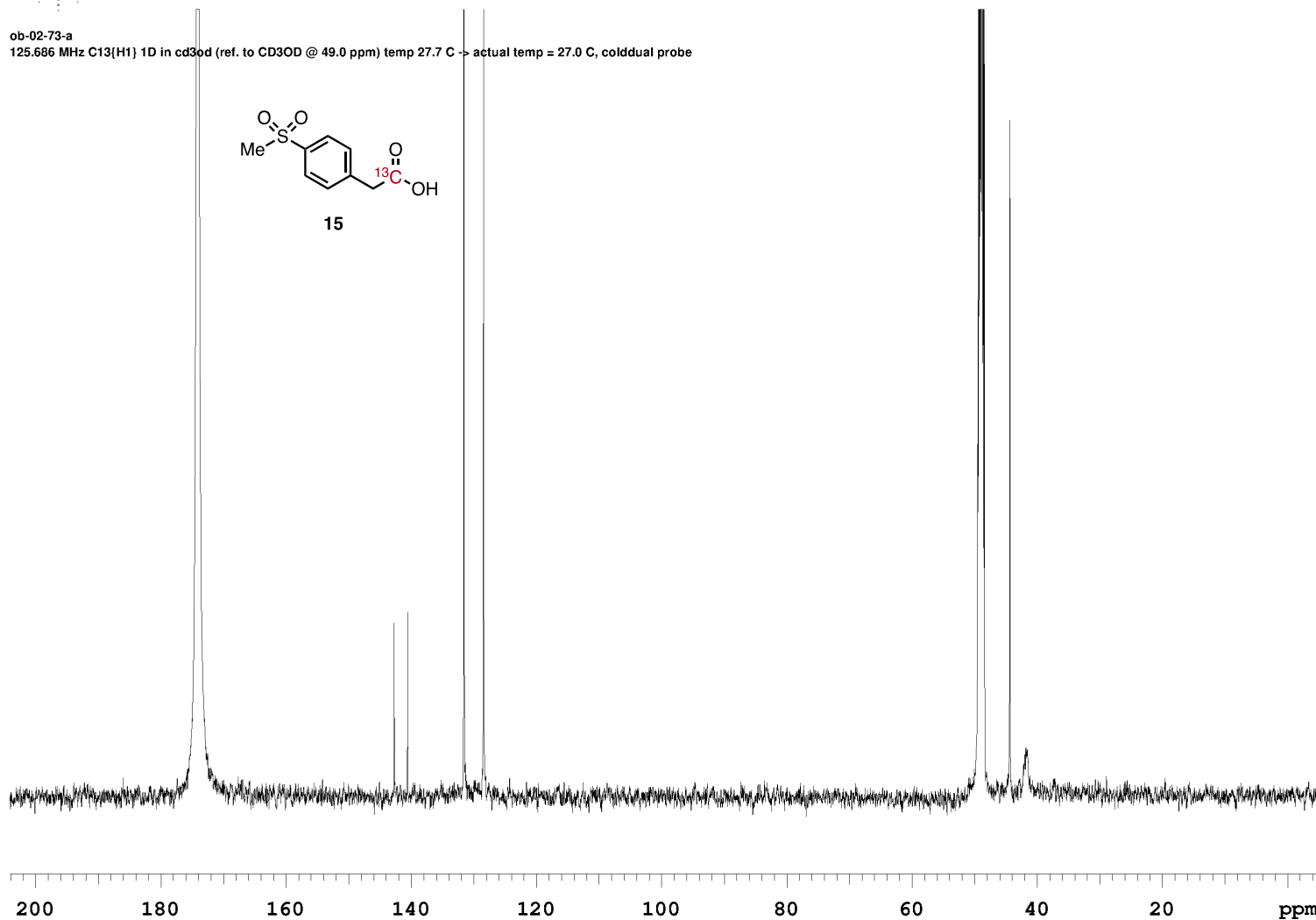
Relaxation Delay(s): **1**
Completed Scans **80**

ob-02-73-a

125.686 MHz C13{H1} 1D in cd3od (ref. to CD3OD @ 49.0 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe



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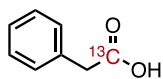
Recorded on: **ibd5, Jun 18 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6000.6**
Digital Res.(Hz/pt): **0.09**

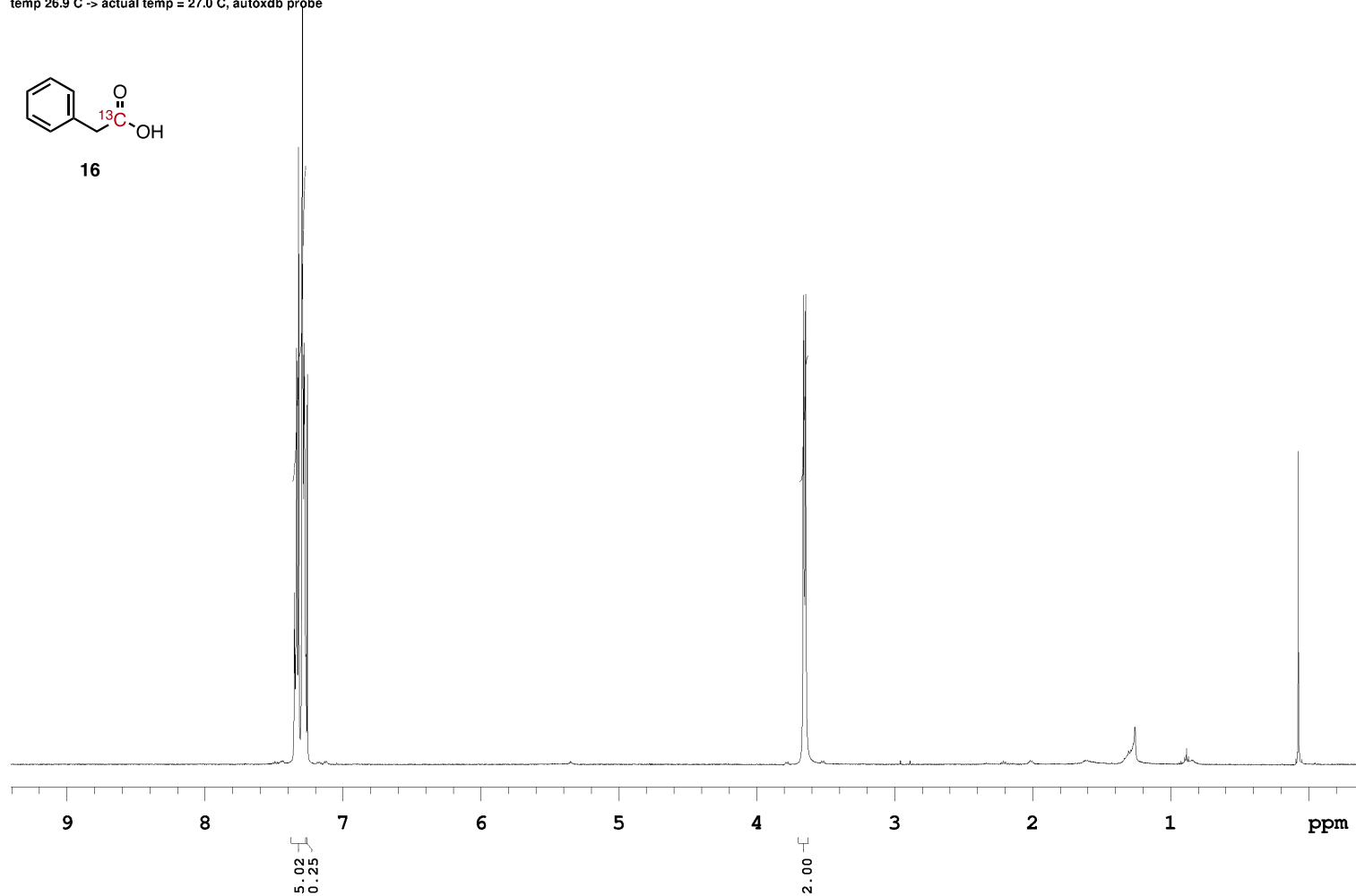
Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.25**

Relaxation Delay(s): **0.1**
Completed Scans **16**

OB-02-91 498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.9 C -> actual temp = 27.0 C, autotdb probe



16





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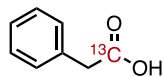
Recorded on: **u500, Jun 18 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

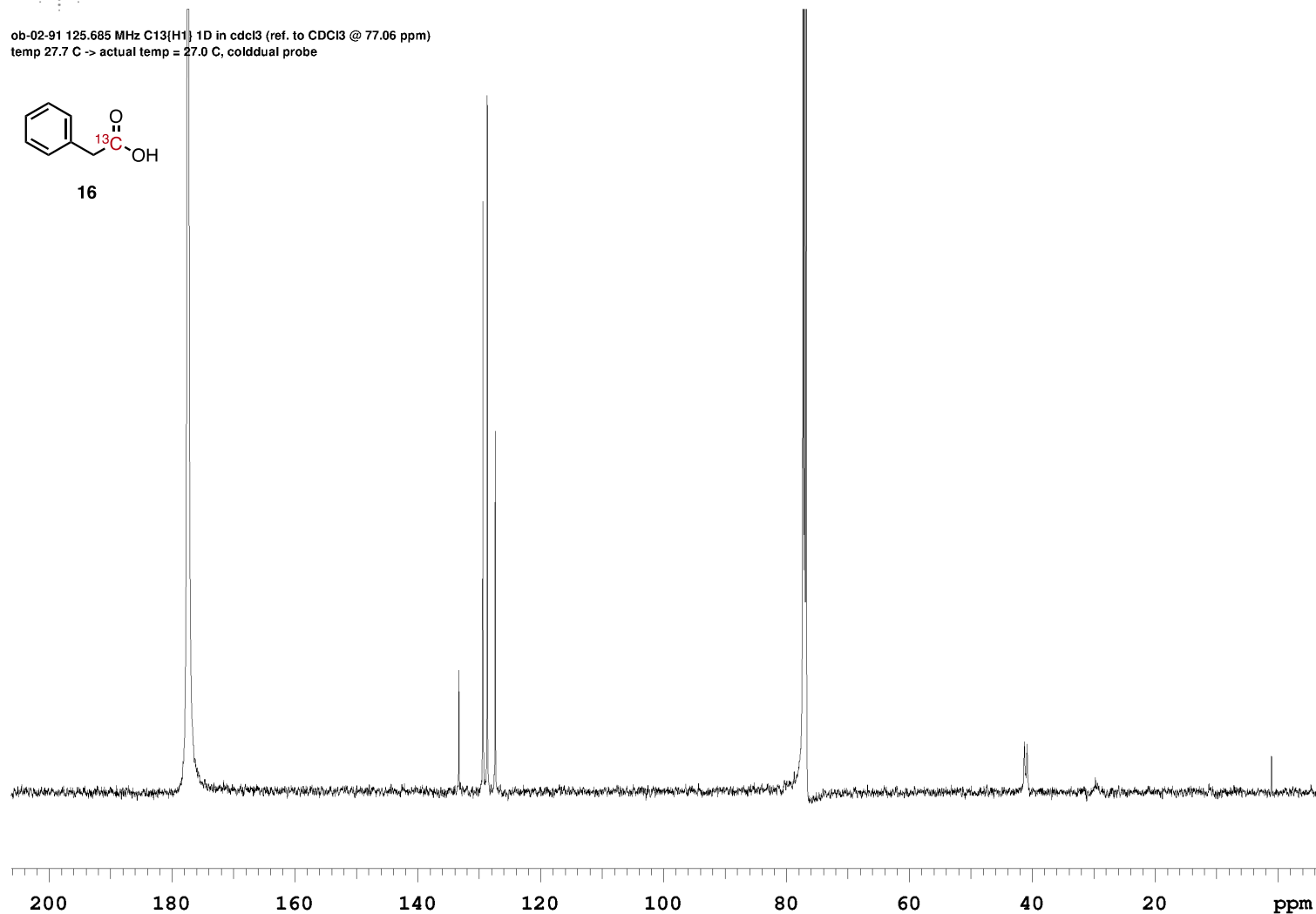
Acquisition Time(s): **1**
Hz per mm(Hz/mm): **111.58**

Relaxation Delay(s): **1**
Completed Scans **208**

ob-02-91 125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, cold dual probe



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Recorded on: **ibd5, Jun 10 2019**
Pulse Sequence: **s2pul**

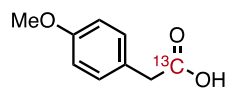
Sweep Width(Hz): **6000.6**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.91**

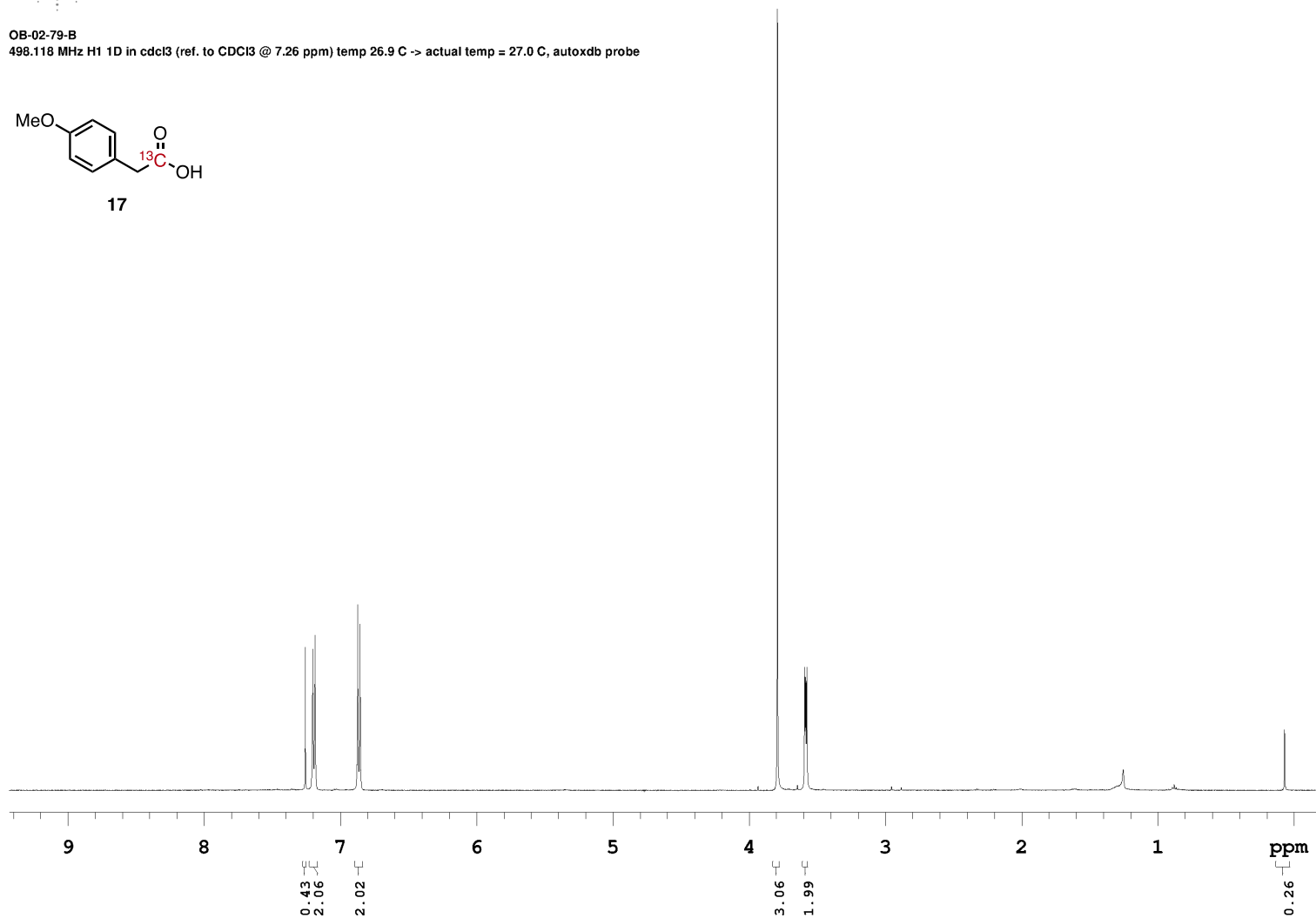
Relaxation Delay(s): **0.1**
Completed Scans **4**

OB-02-79-B

498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdo probe



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Recorded on: **u500, Jun 10 2019**
Pulse Sequence: **s2pul**

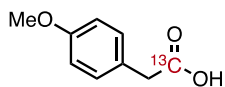
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **113.14**

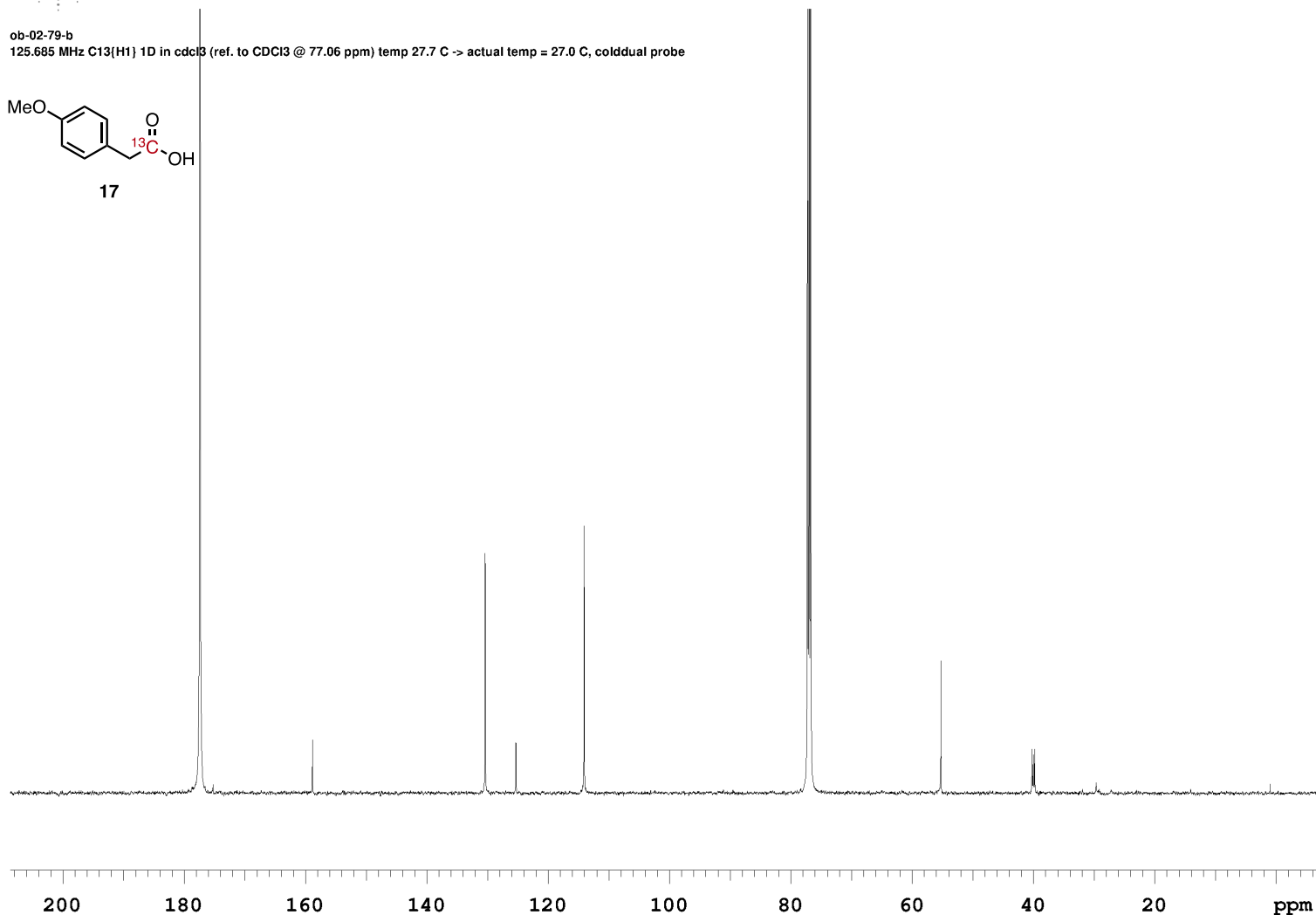
Relaxation Delay(s): **1**
Completed Scans **528**

ob-02-79-b

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe



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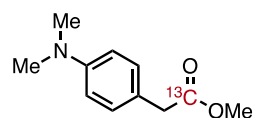
Recorded on: **i600, Jun 19 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **7183.91**
Digital Res.(Hz/pt): **0.11**

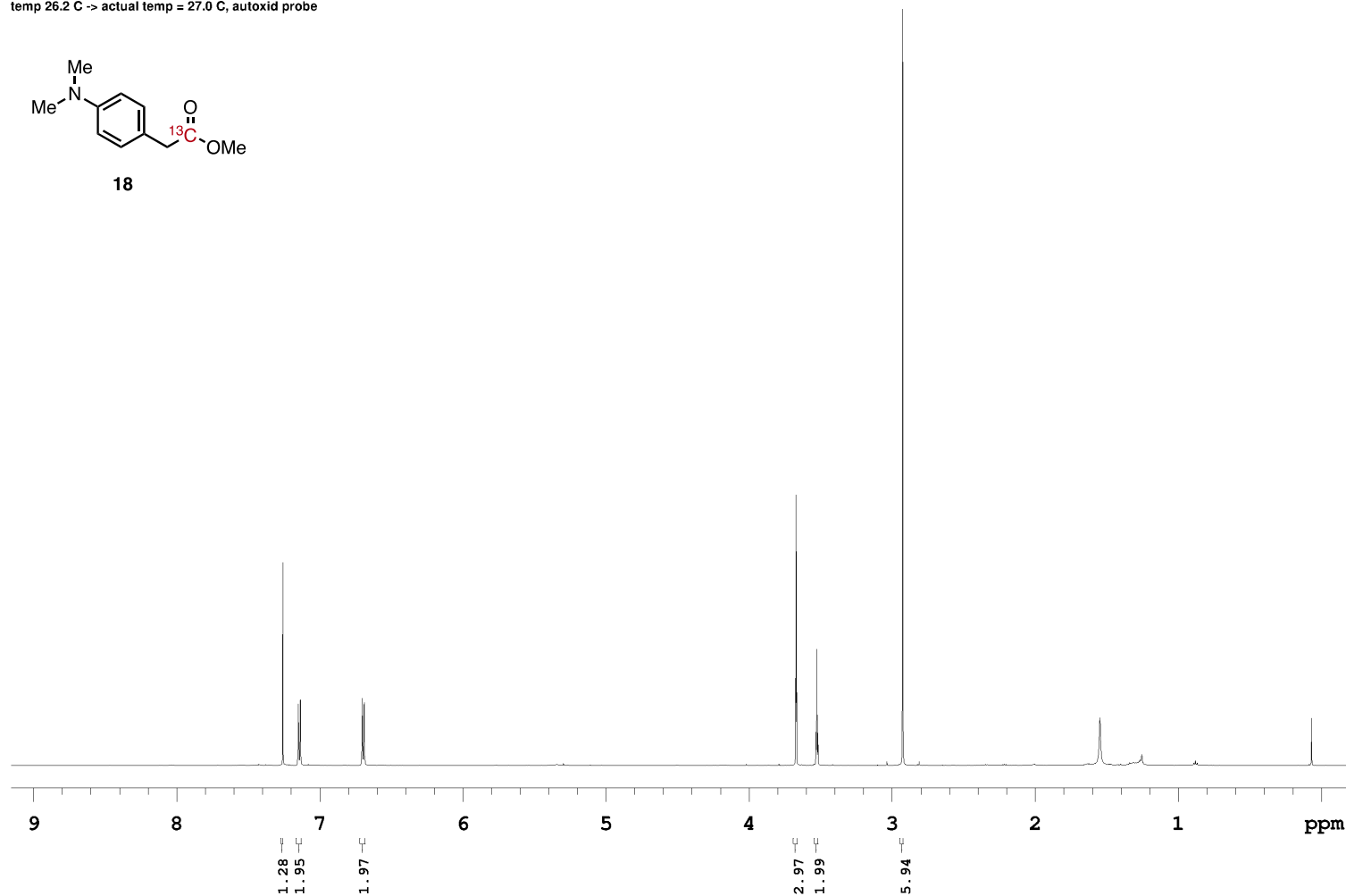
Acquisition Time(s): **5**
Hz per mm(Hz/mm): **23.44**

Relaxation Delay(s): **0.1**
Completed Scans **16**

ob-02-93 599.926 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.2 C -> actual temp = 27.0 C, autoxid probe



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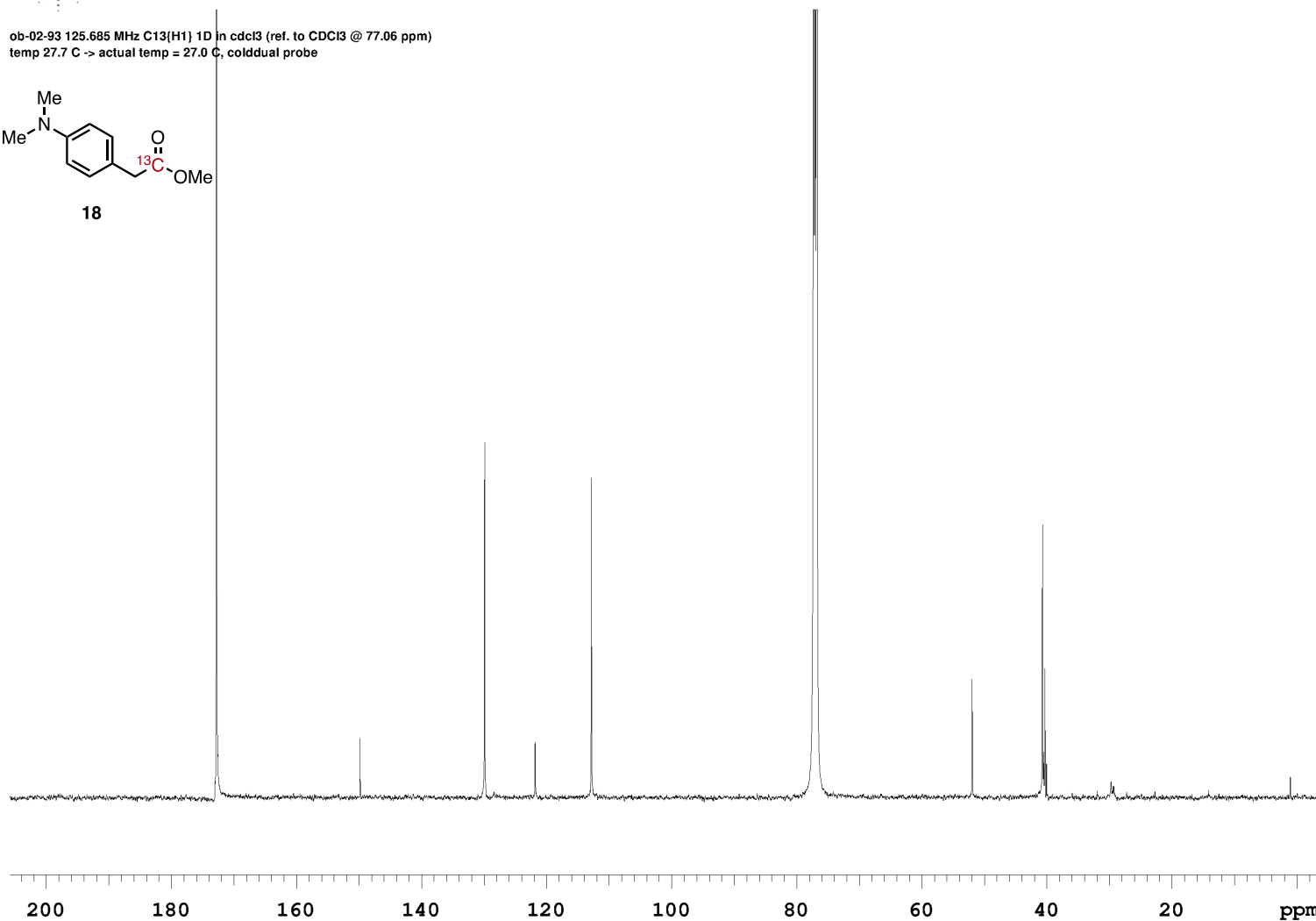
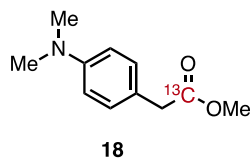
Recorded on: **u500, Jun 19 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **110.3**

Relaxation Delay(s): **1**
Completed Scans: **4804**

ob-02-93 125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Jan 10 2020**
Pulse Sequence: **PRESAT**

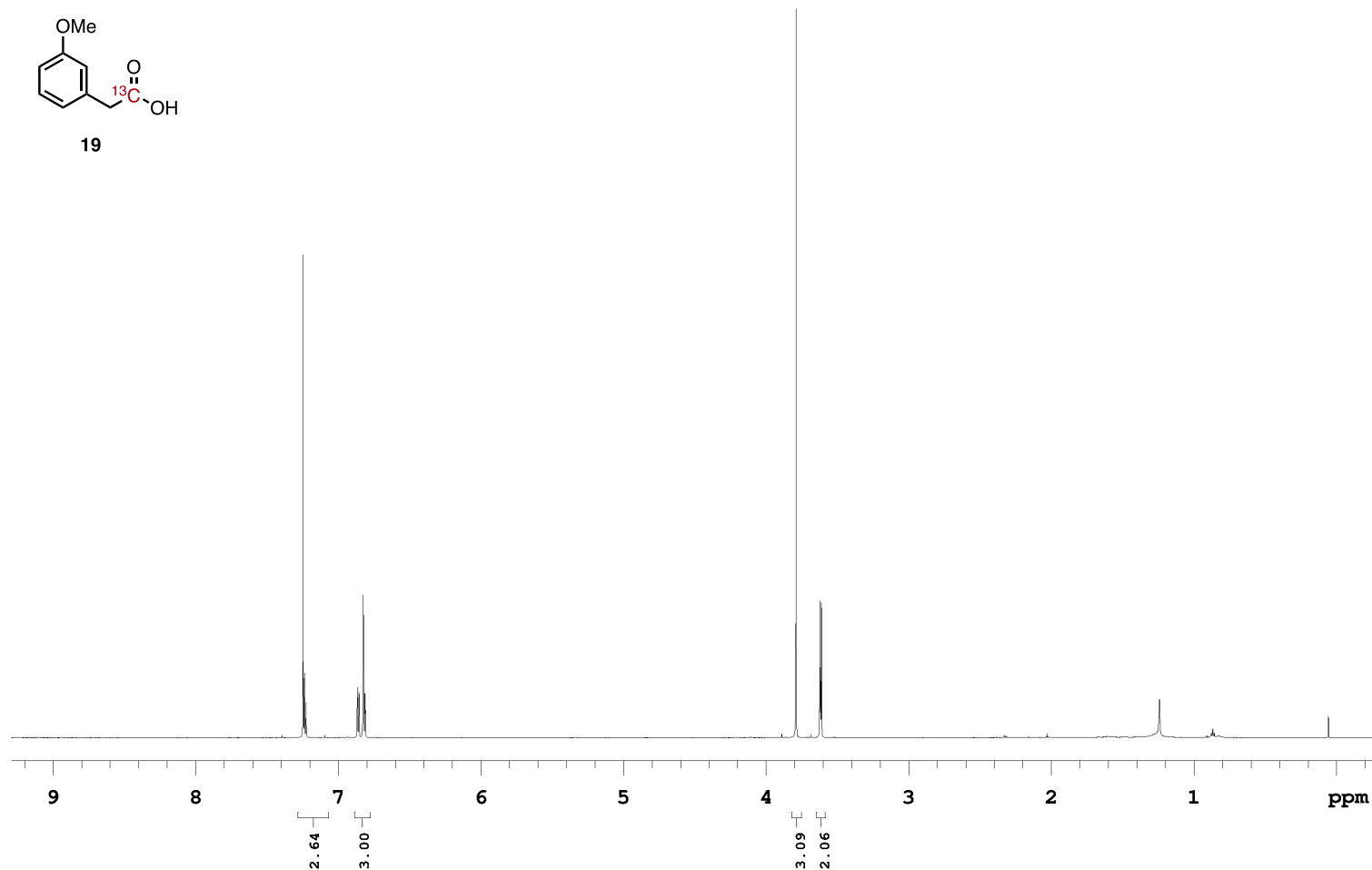
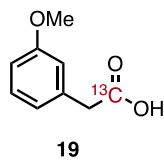
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.92**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-13-17-A

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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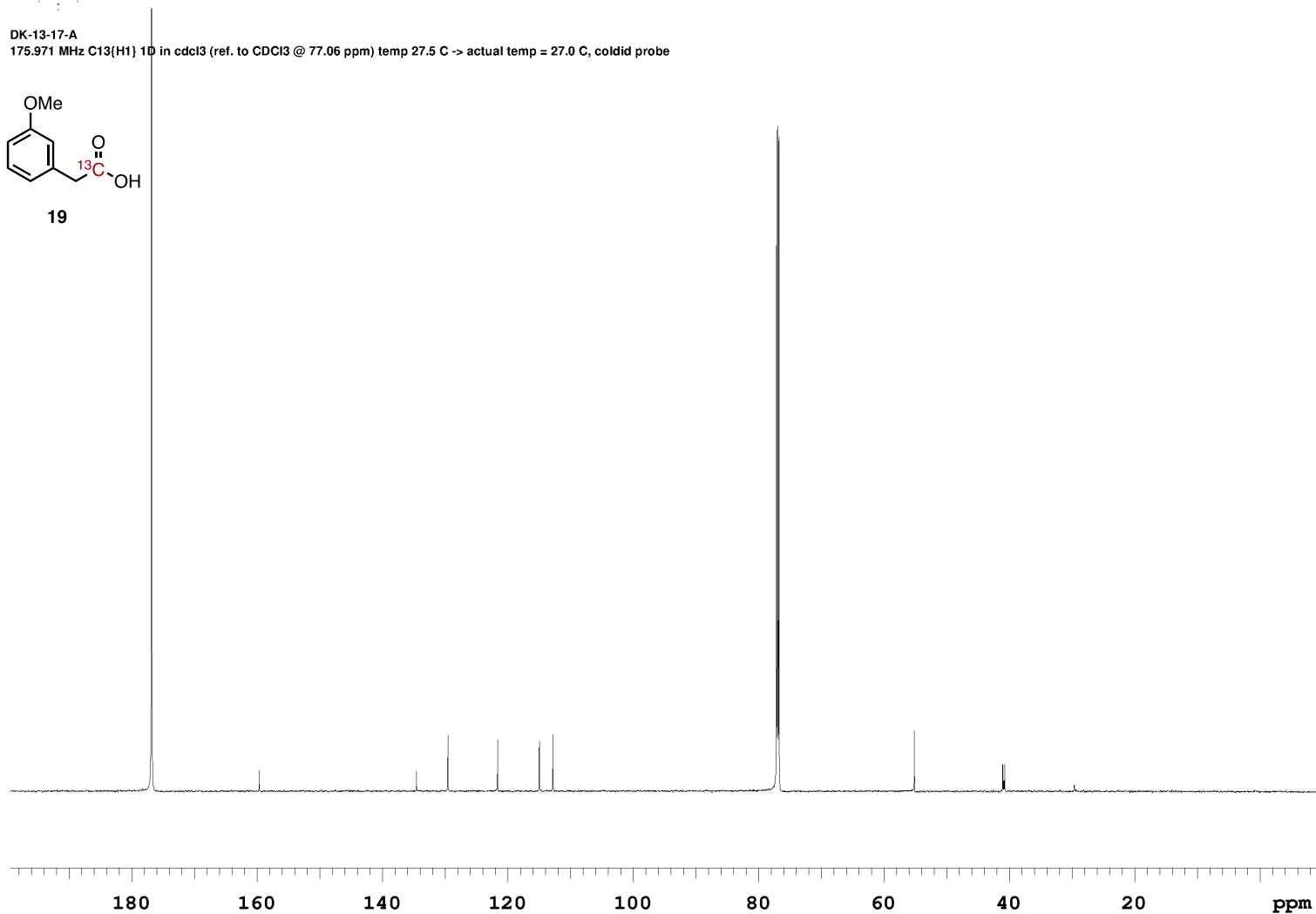
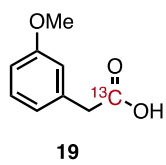
Recorded on: **v700, Jan 9 2020**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **1000**

DK-13-17-A
175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coidid probe





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Recorded on: **v700, Jan 9 2020**
Pulse Sequence: **PRESAT**

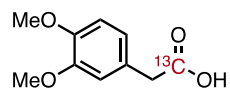
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.13**

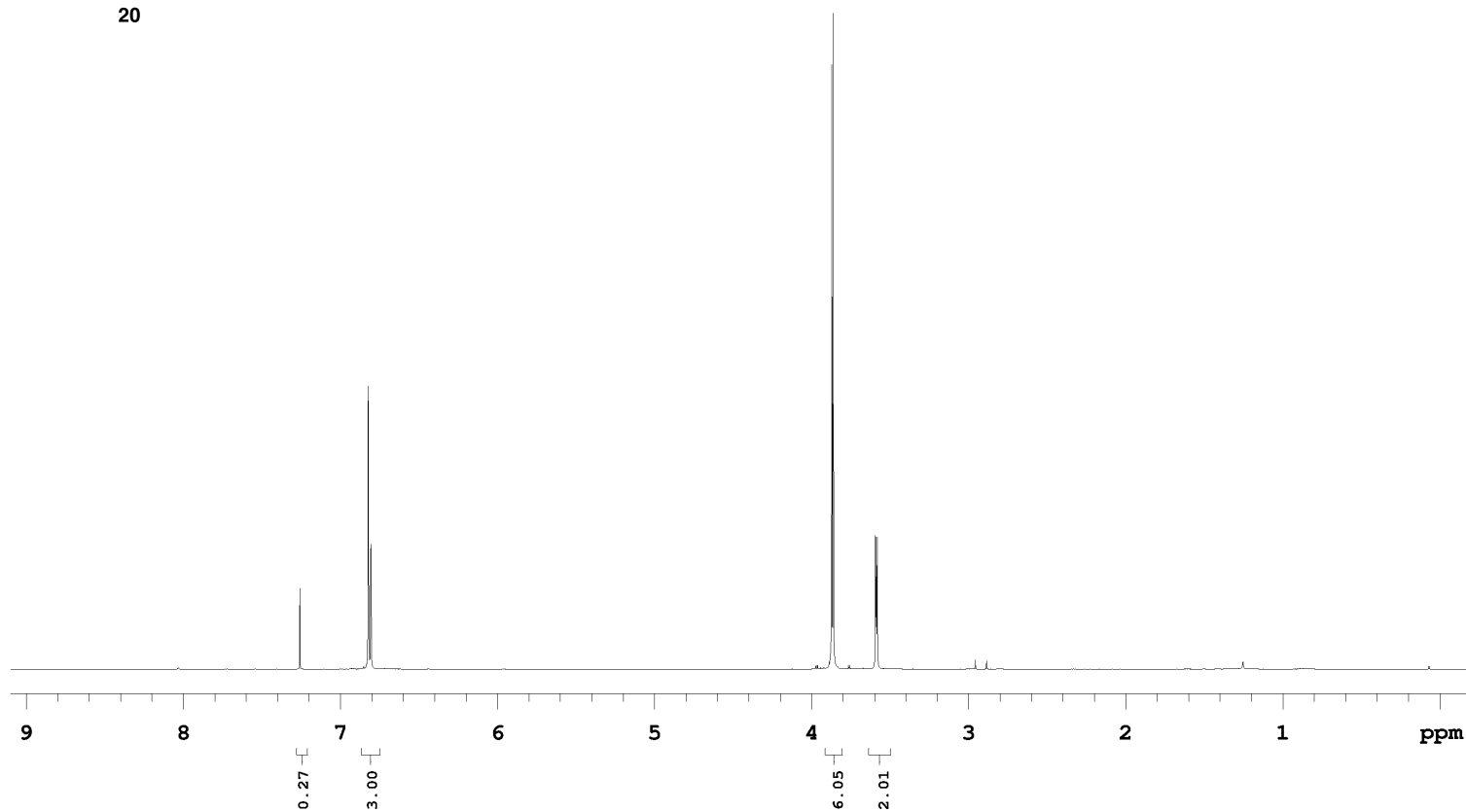
Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-13-17-B

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe



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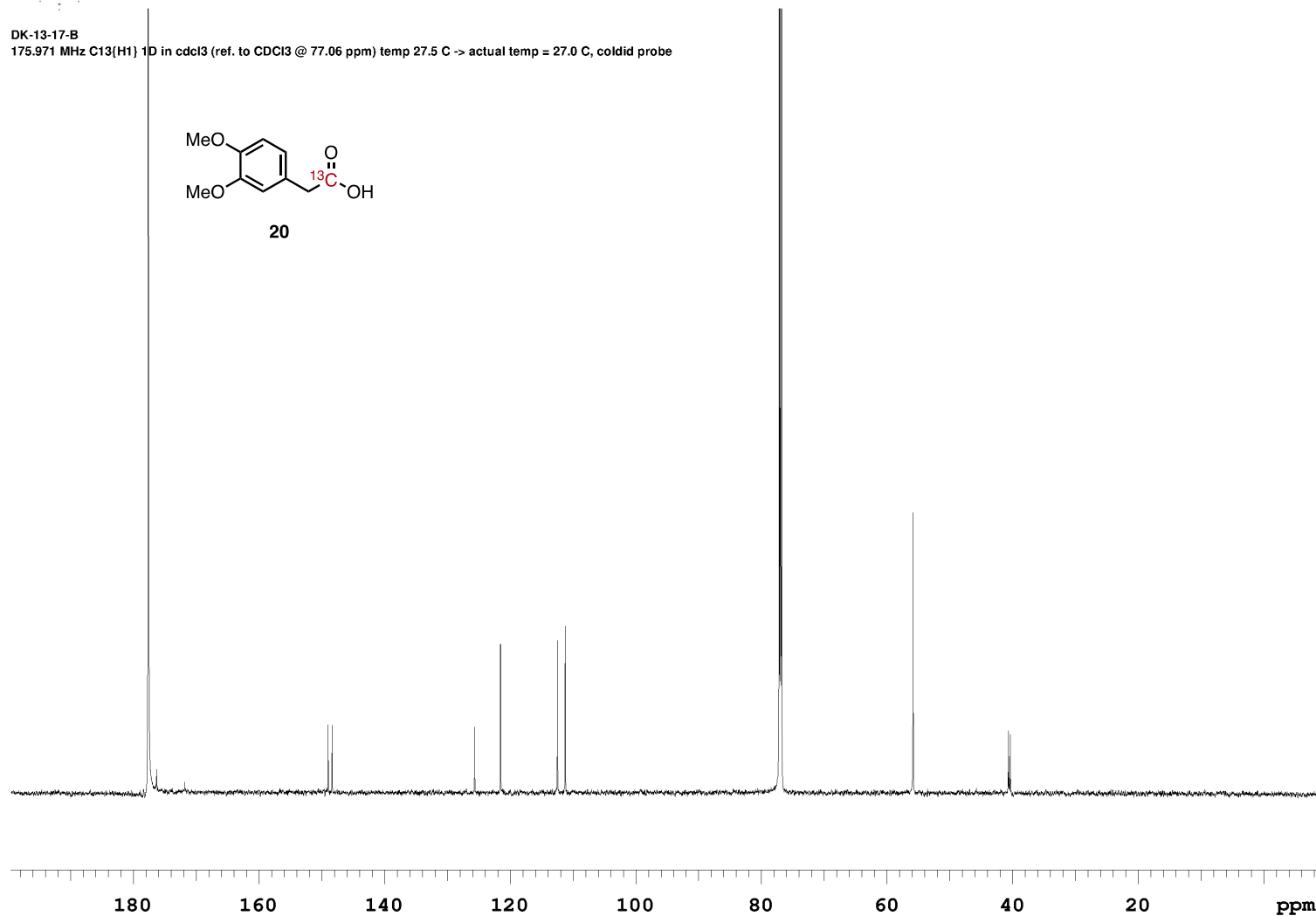
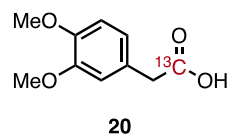
Recorded on: **v700, Jan 9 2020**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **252**

DK-13-17-B
175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **v700, Feb 1 2020**
Pulse Sequence: **PRESAT**

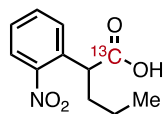
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **28.27**

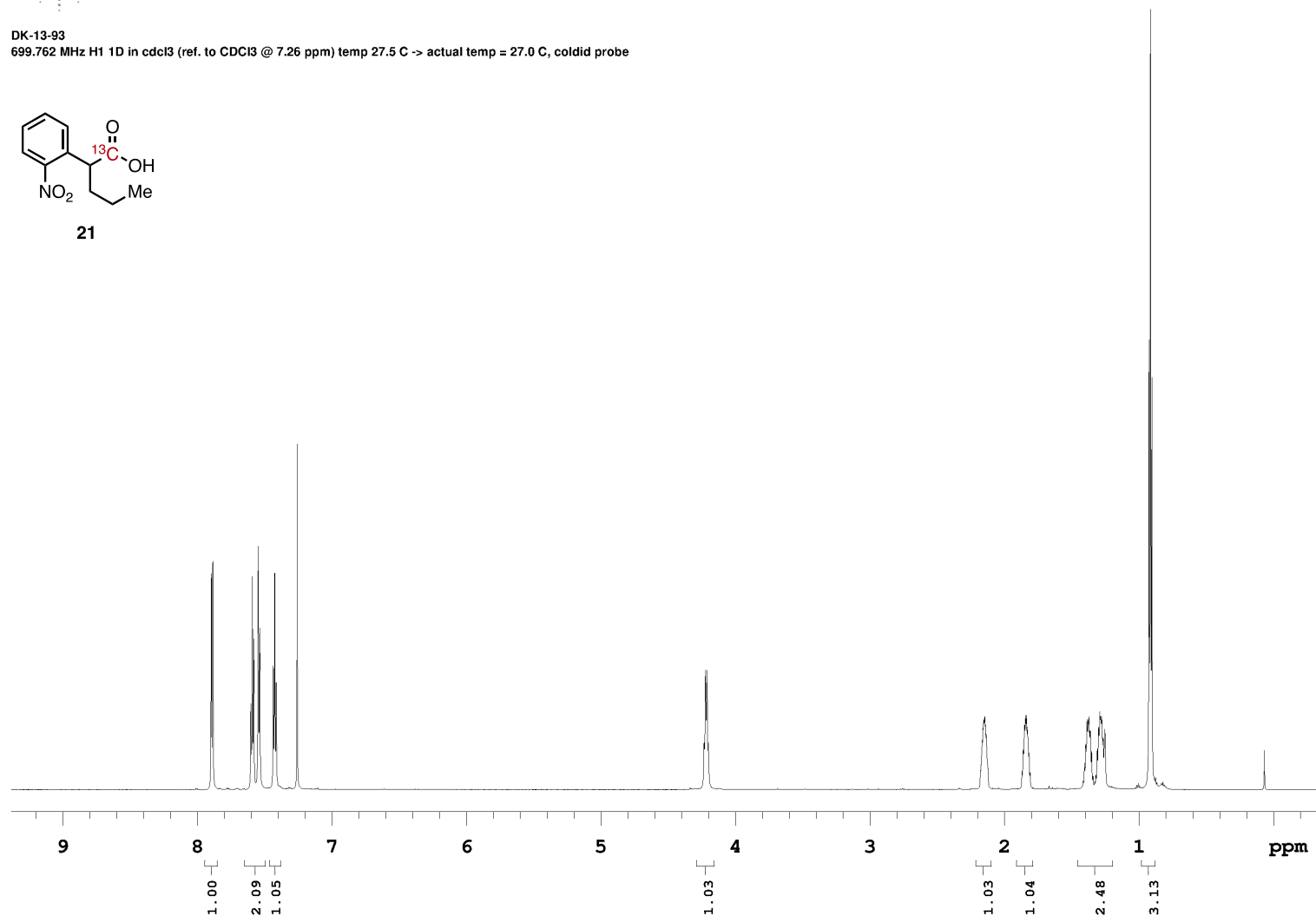
Relaxation Delay(s): **0.1**
Completed Scans **4**

DK-13-93

699.762 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe



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Recorded on: **u500, Feb 1 2020**
Pulse Sequence: **s2pul**

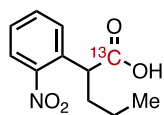
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **115.06**

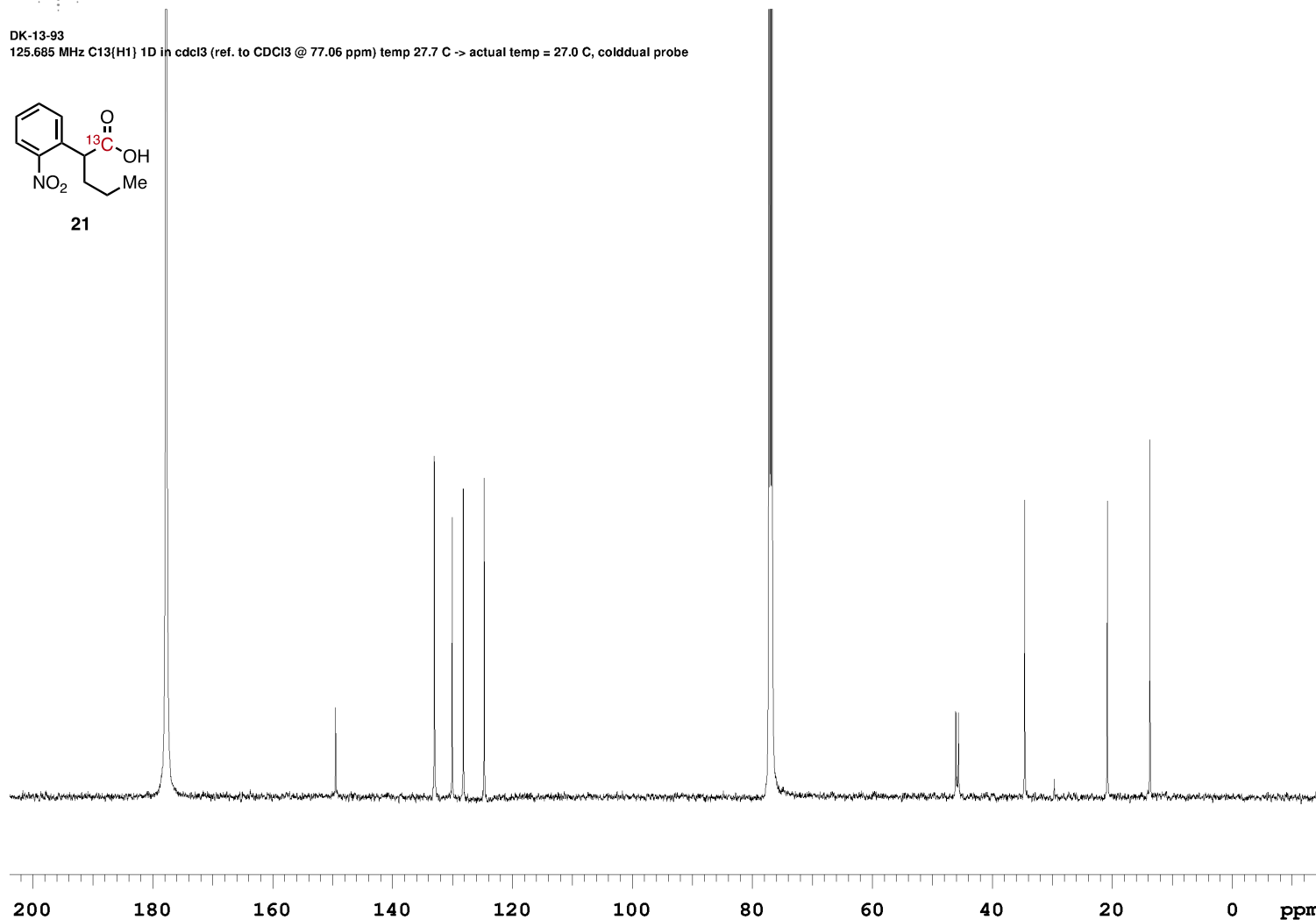
Relaxation Delay(s): **1**
Completed Scans **684**

DK-13-93

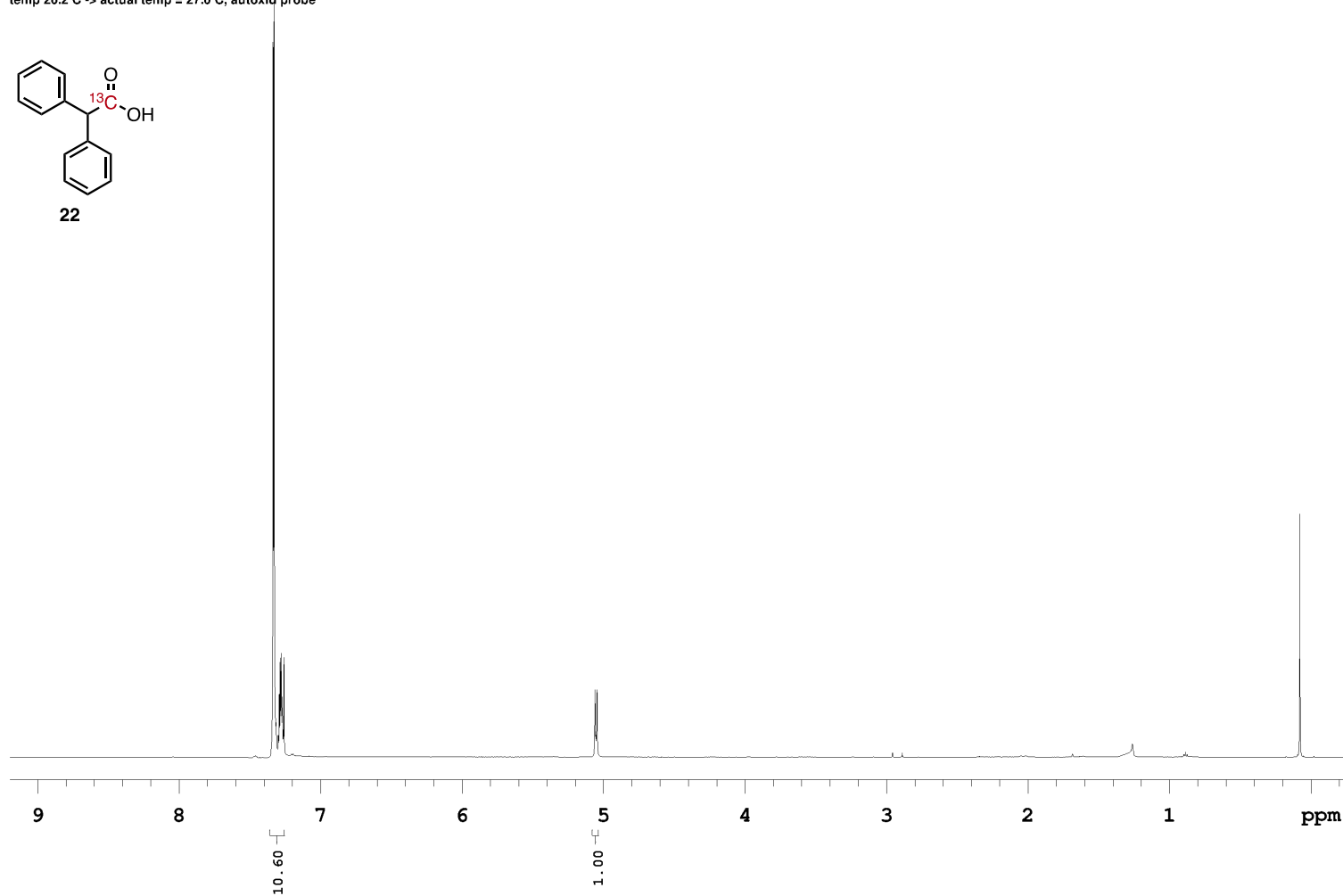
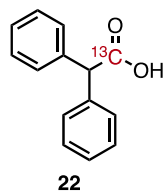
125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe



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ob-02-83-B 599.926 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.2 C -> actual temp = 27.0 C, autoxid probe





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Recorded on: **u500, Jun 12 2019**

Sweep Width(Hz): **33783.8**

Acquisition Time(s): **1**

Relaxation Delay(s): **1**

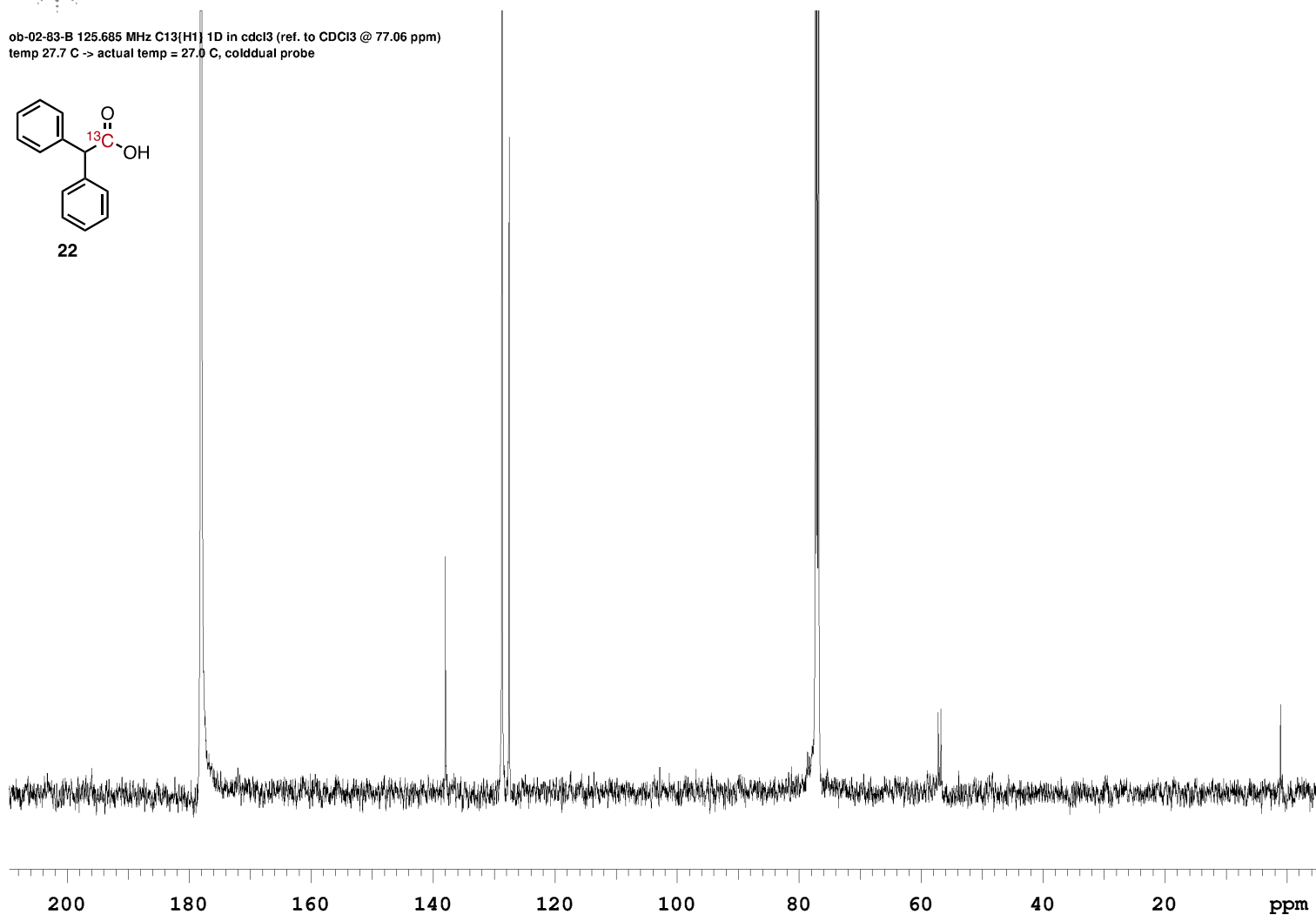
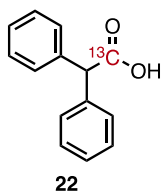
Pulse Sequence: **s2pul**

Digital Res.(Hz/pt): **0.26**

Hz per mm(Hz/mm): **112.23**

Completed Scans **24**

ob-02-83-B 125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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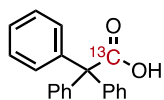
Recorded on: **v700, Aug 6 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

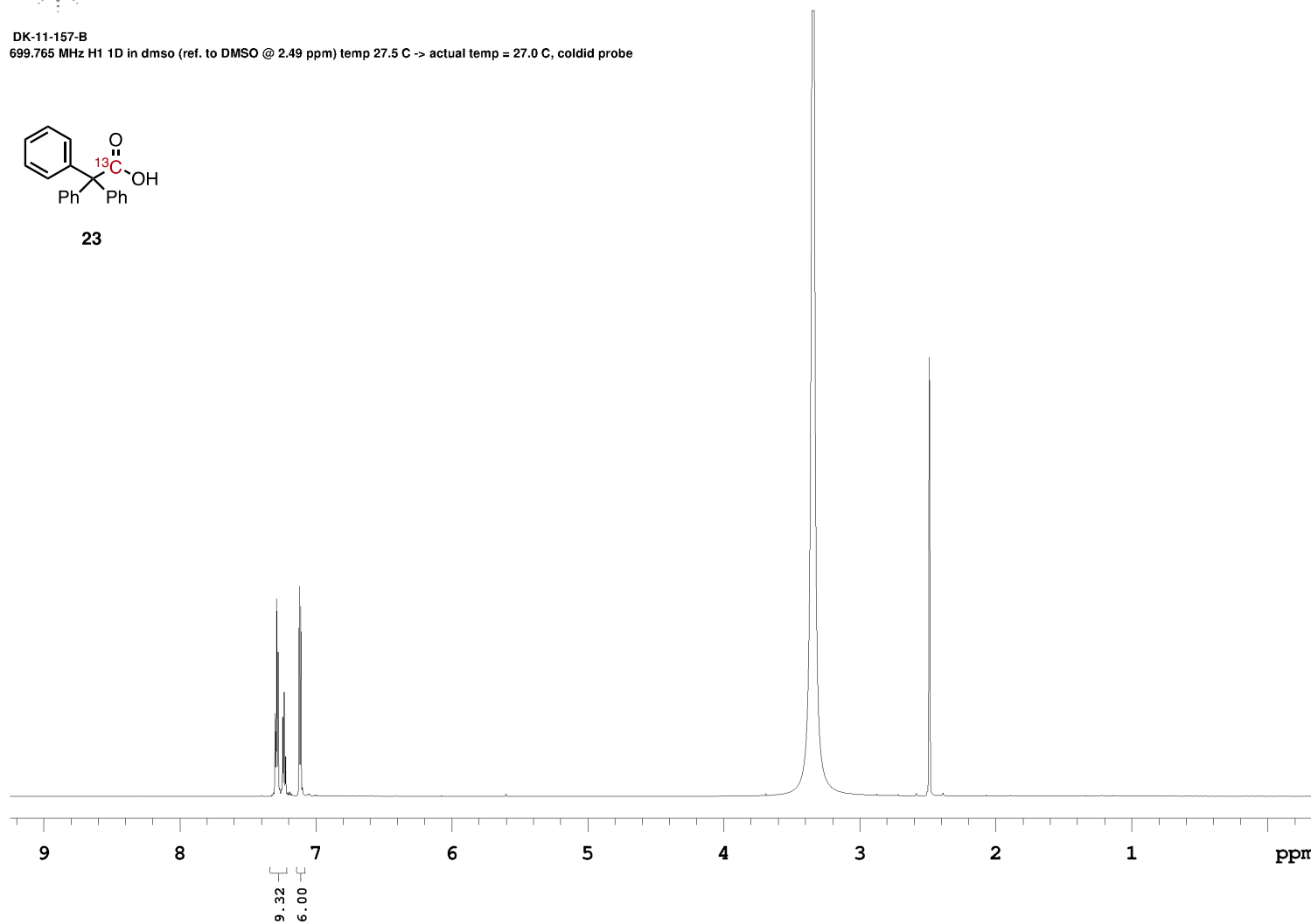
Acquisition Time(s): **5**
Hz per mm(Hz/mm): **28.15**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-11-157-B
699.765 MHz H1 1D in dms0 (ref. to DMSO @ 2.49 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe



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Recorded on: **v700, Aug 5 2019**
Pulse Sequence: **s2pul**

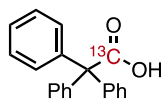
Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

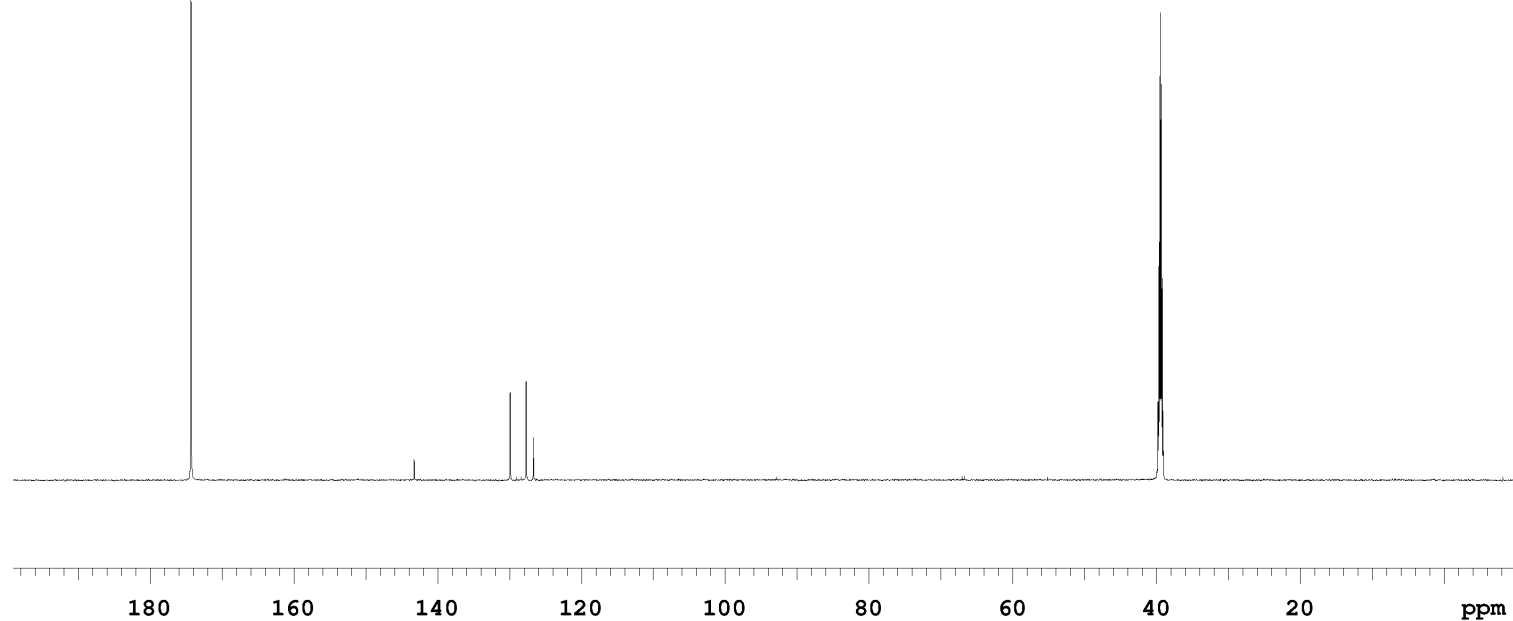
Relaxation Delay(s): **1**
Completed Scans **164**

DK-11-157-B

175.972 MHz C13{H1} 1D in dmso (ref. to DMSO @ 39.5 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe



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Recorded on: **v700, Aug 7 2019**
Pulse Sequence: **PRESAT**

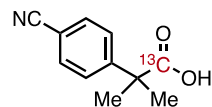
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **28.58**

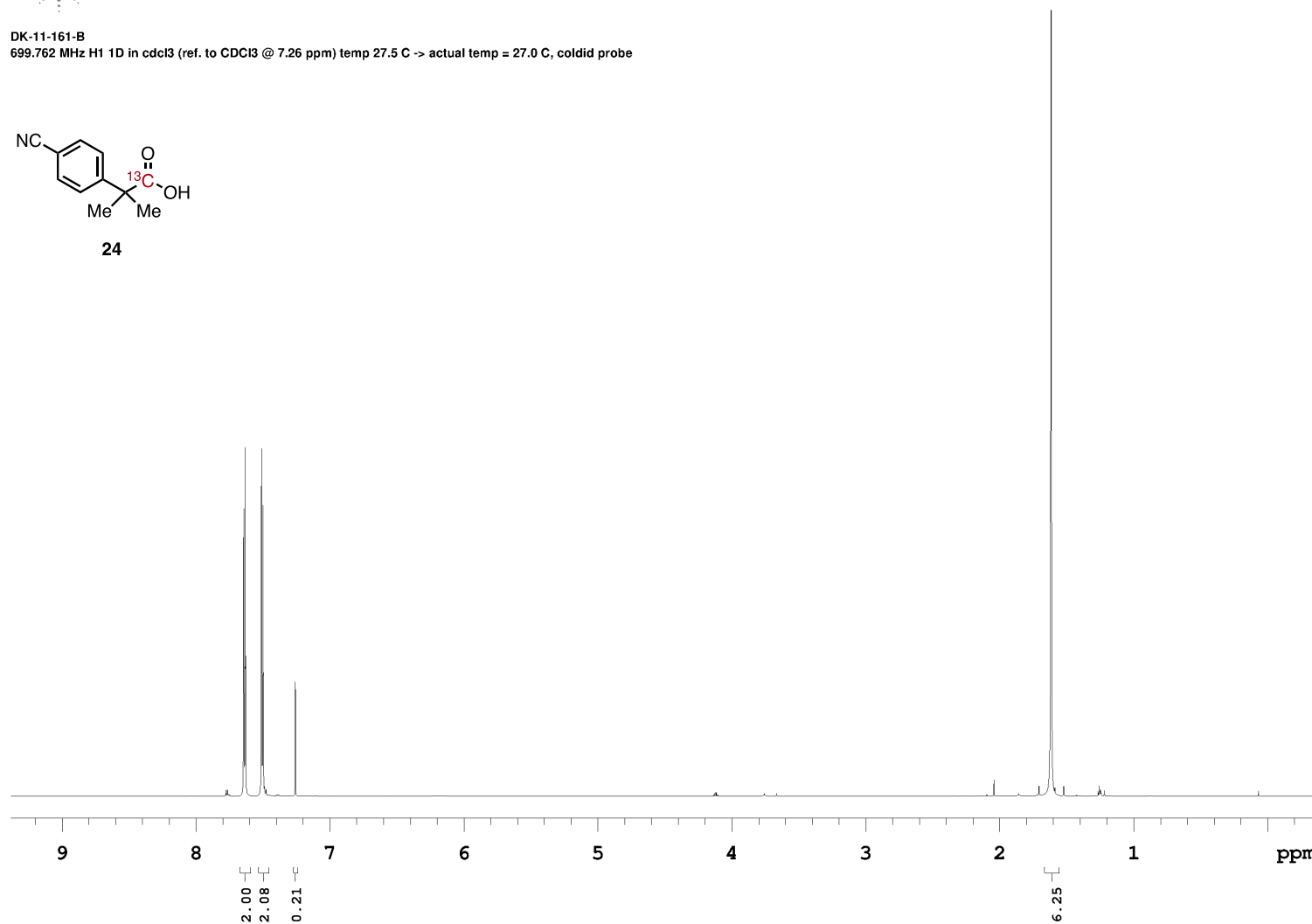
Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-11-161-B

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe



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Recorded on: **v700, Aug 6 2019**
Pulse Sequence: **s2pul**

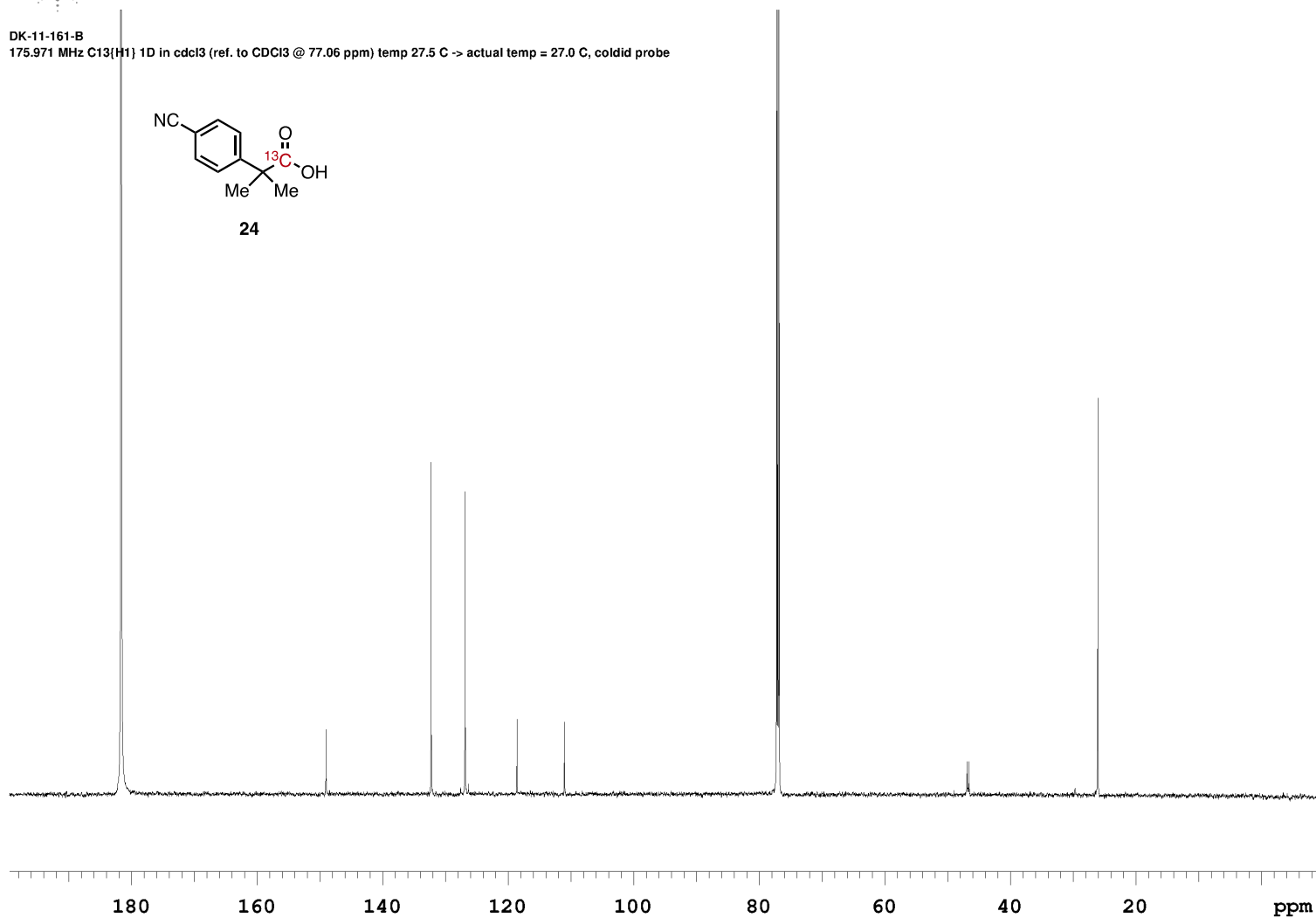
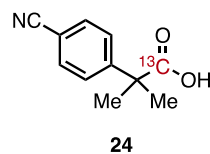
Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **228**

DK-11-161-B

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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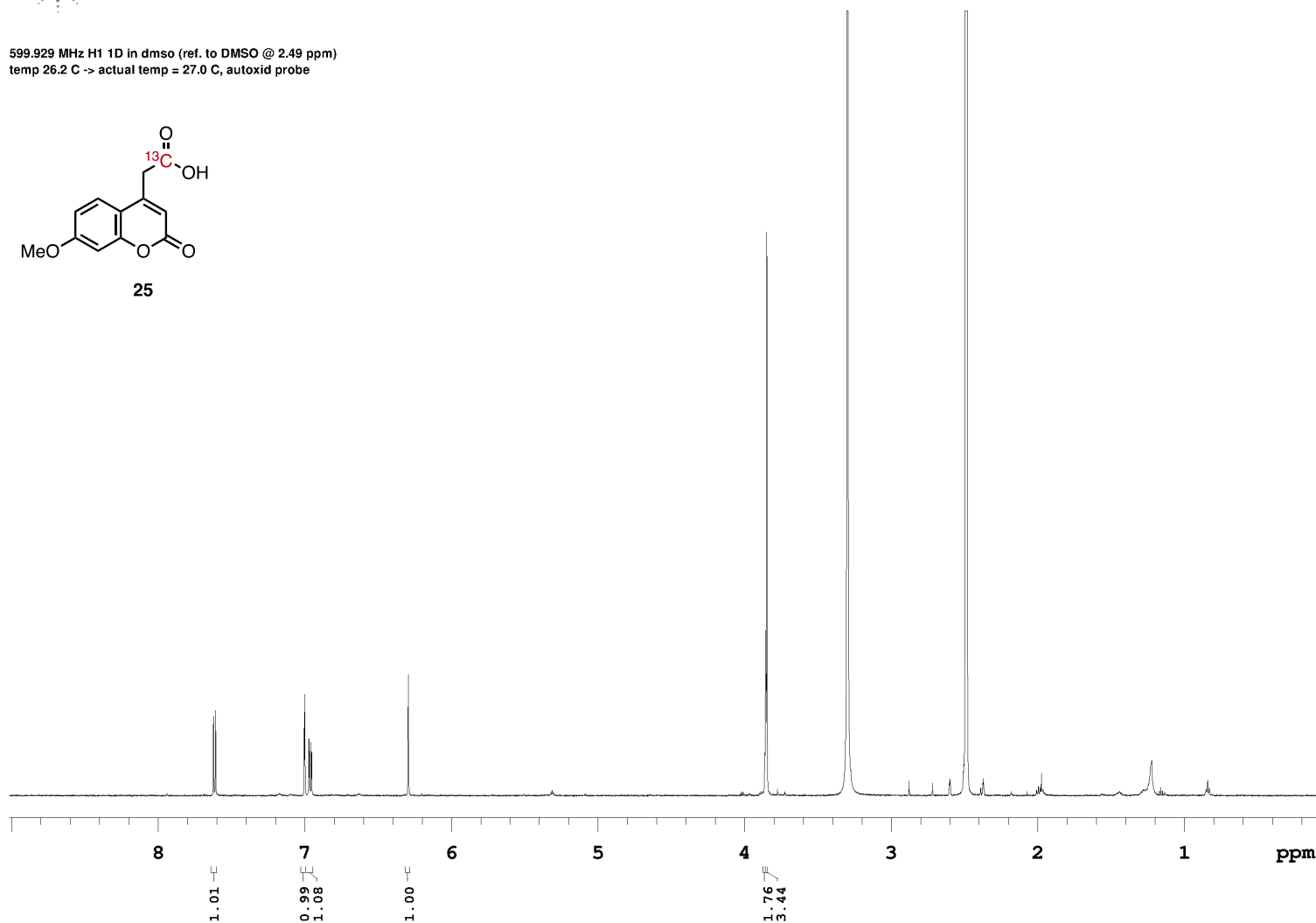
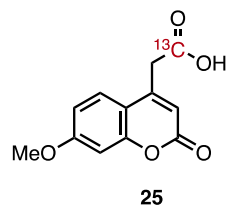
Recorded on: **1600, Jan 10 2020**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **7183.91**
Digital Res.(Hz/pt): **0.11**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **22.4**

Relaxation Delay(s): **0.1**
Completed Scans **16**

599.929 MHz H1 1D in dmso (ref. to DMSO @ 2.49 ppm)
temp 26.2 C -> actual temp = 27.0 C, autotxid probe





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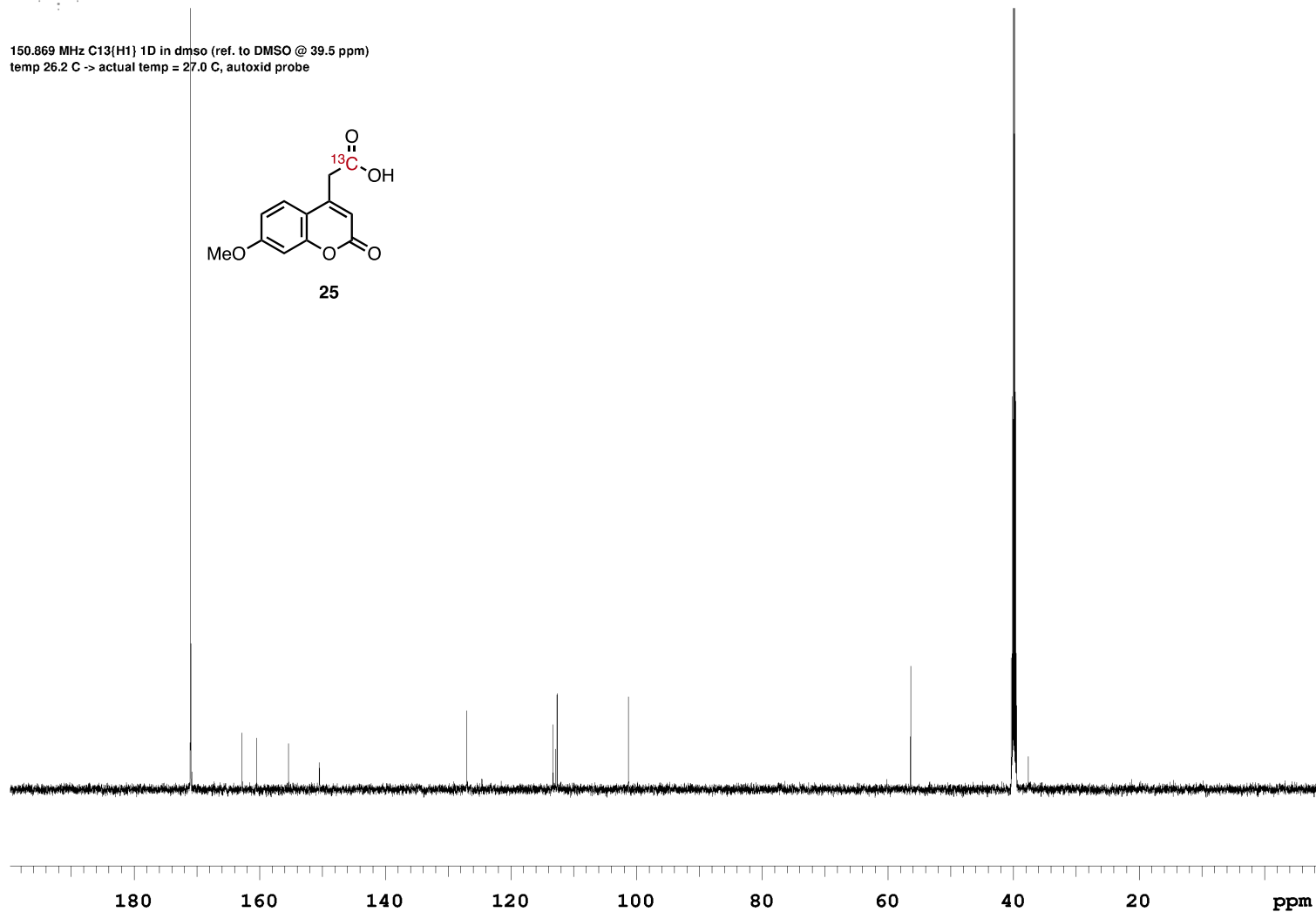
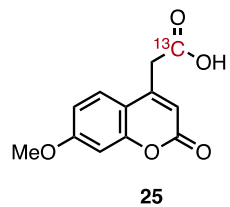
Recorded on: **i600, Jan 9 2020**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **40322.6**
Digital Res.(Hz/pt): **0.31**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **131.05**

Relaxation Delay(s): **1**
Completed Scans **424**

150.869 MHz C13{H1} 1D in dms0 (ref. to DMSO @ 39.5 ppm)
temp 26.2 C -> actual temp = 27.0 C, autotxid probe





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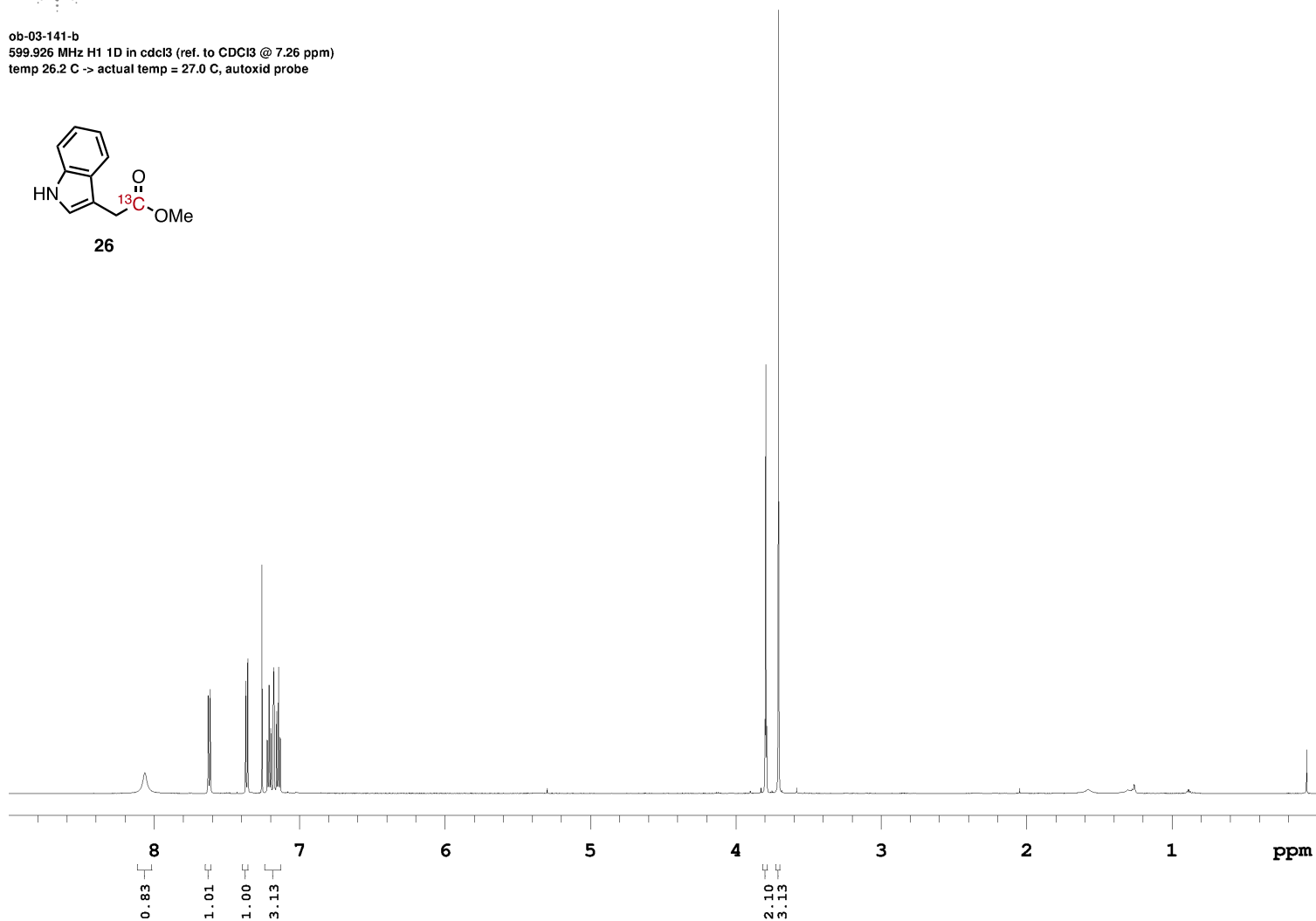
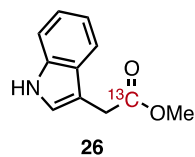
Recorded on: **i600, Jan 15 2020**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **7183.91**
Digital Res.(Hz/pt): **0.11**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **22.54**

Relaxation Delay(s): **0.1**
Completed Scans **8**

ob-03-141-b
599.926 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.2 C -> actual temp = 27.0 C, autoxid probe





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Recorded on: **1600, Jan 15 2020**

Sweep Width(Hz): **40322.6**

Acquisition Time(s): **1**

Relaxation Delay(s): **1**

Pulse Sequence: **s2pul**

Digital Res.(Hz/pt): **0.31**

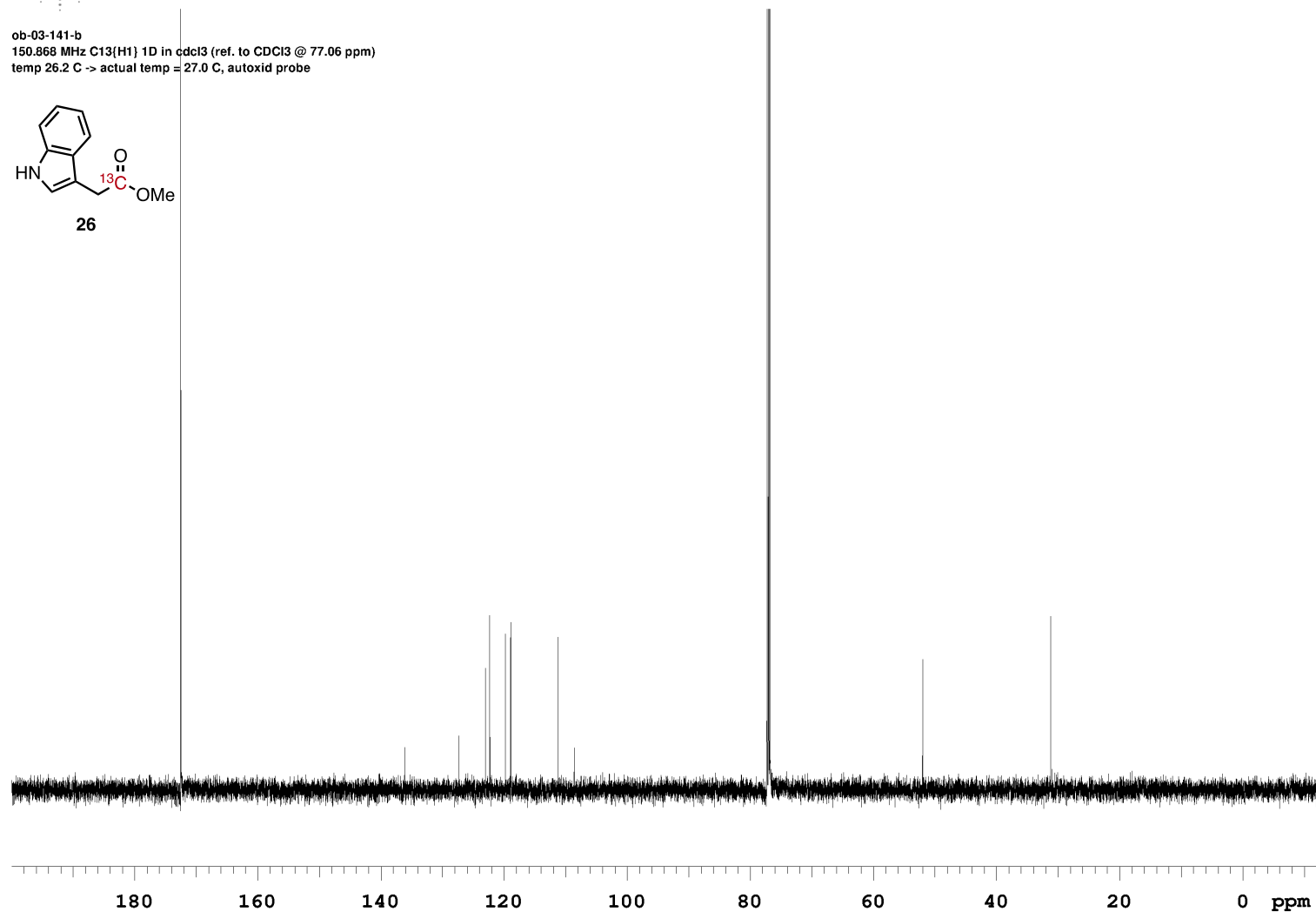
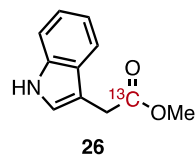
Hz per mm(Hz/mm): **133.4**

Completed Scans **628**

ob-03-141-b

150.868 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)

temp 26.2 C -> actual temp = 27.0 C, autotxid probe





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Recorded on: **u500, Jun 17 2019**
Pulse Sequence: **PRESAT**

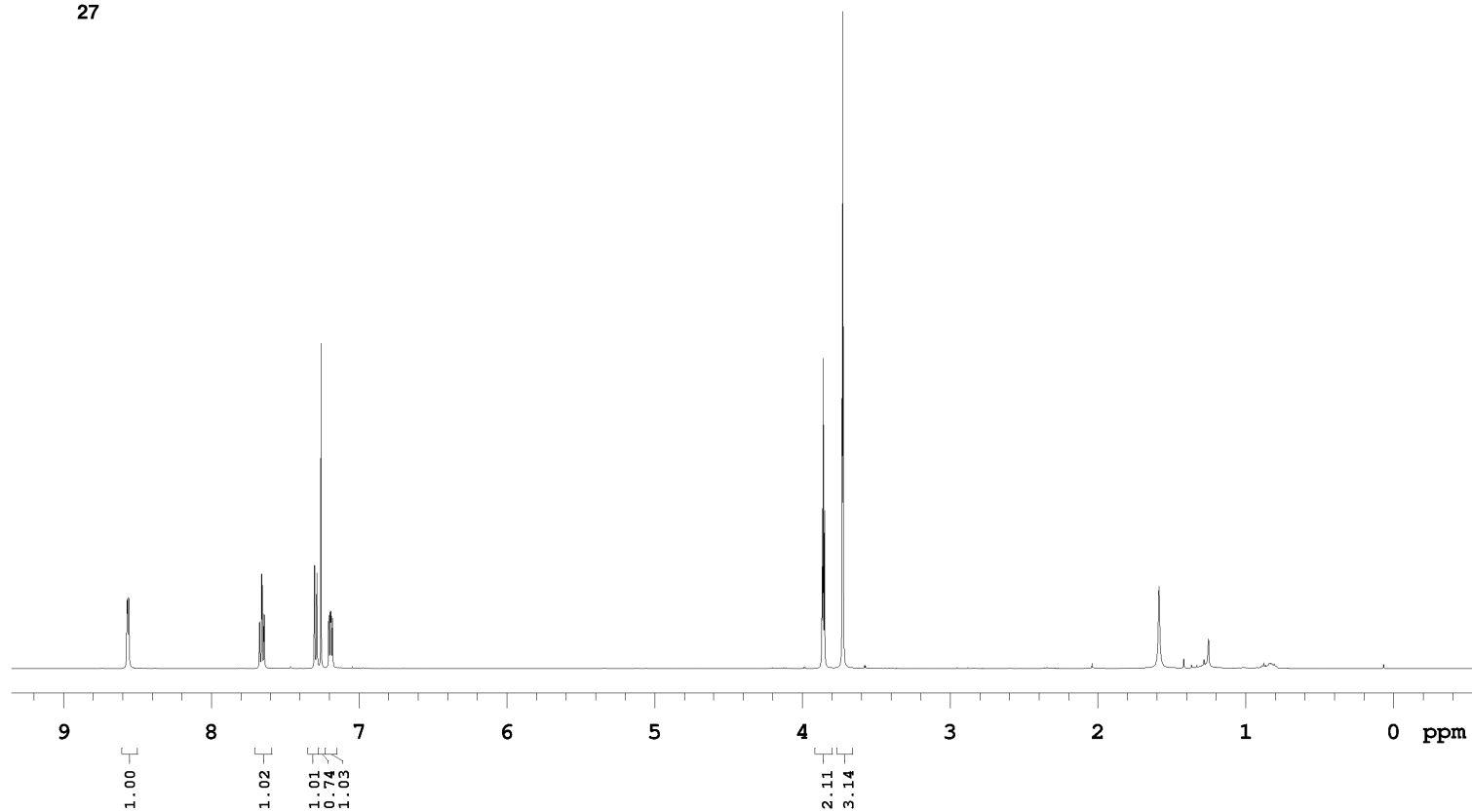
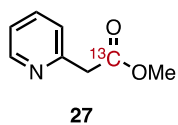
Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.63**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-205

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **u500, Jun 17 2019**
Pulse Sequence: **s2pul**

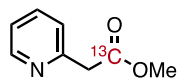
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **118.42**

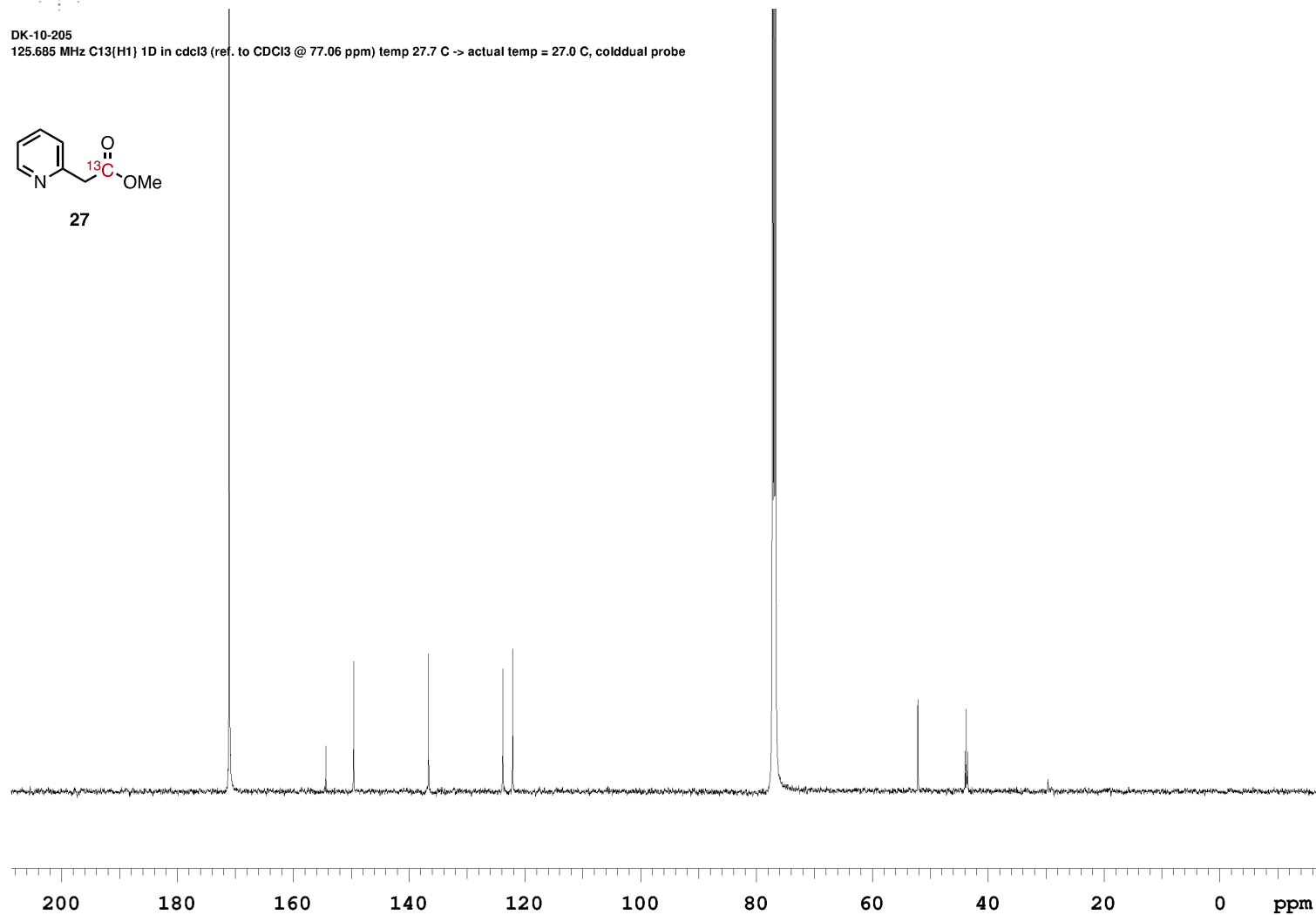
Relaxation Delay(s): **1**
Completed Scans: **708**

DK-10-205

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe



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Recorded on: **v700, Jun 14 2019**
Pulse Sequence: **PRESAT**

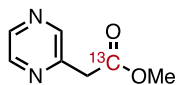
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **30.15**

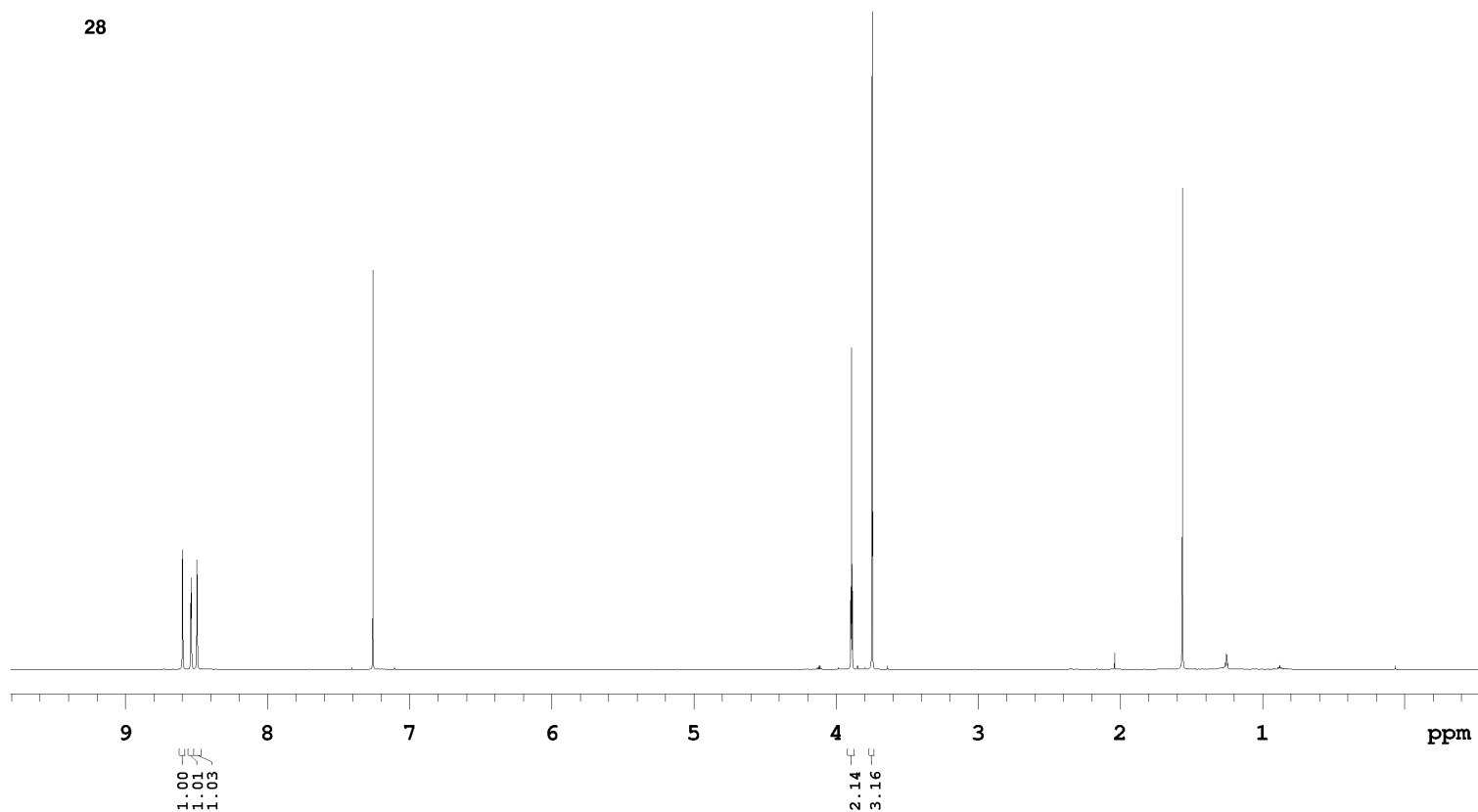
Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-199

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe



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Recorded on: **v700, Jun 14 2019**
Pulse Sequence: **s2pul**

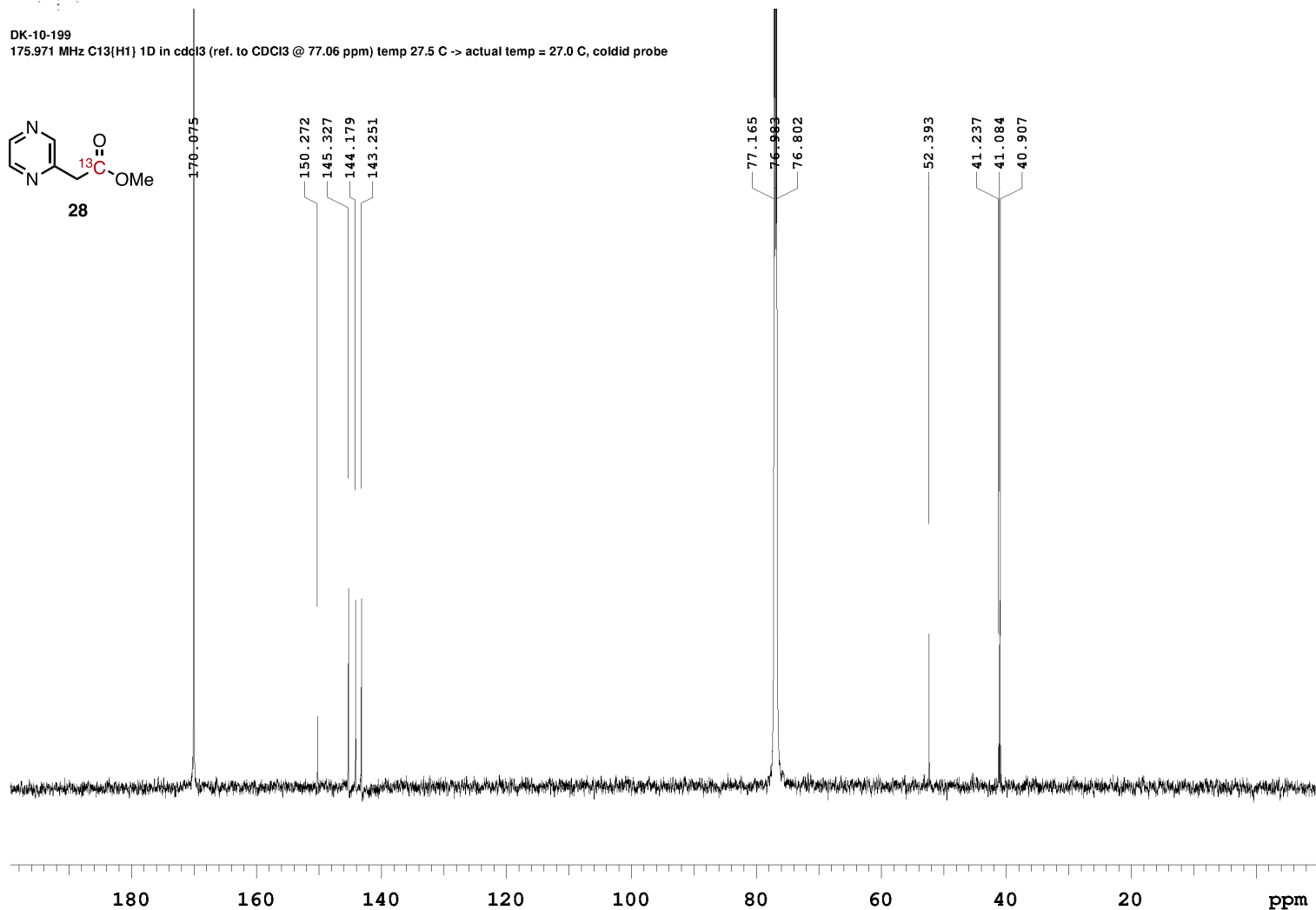
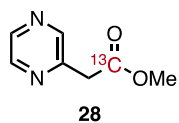
Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **1000**

DK-10-199

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **v700, Jun 12 2019**
Pulse Sequence: **PRESAT**

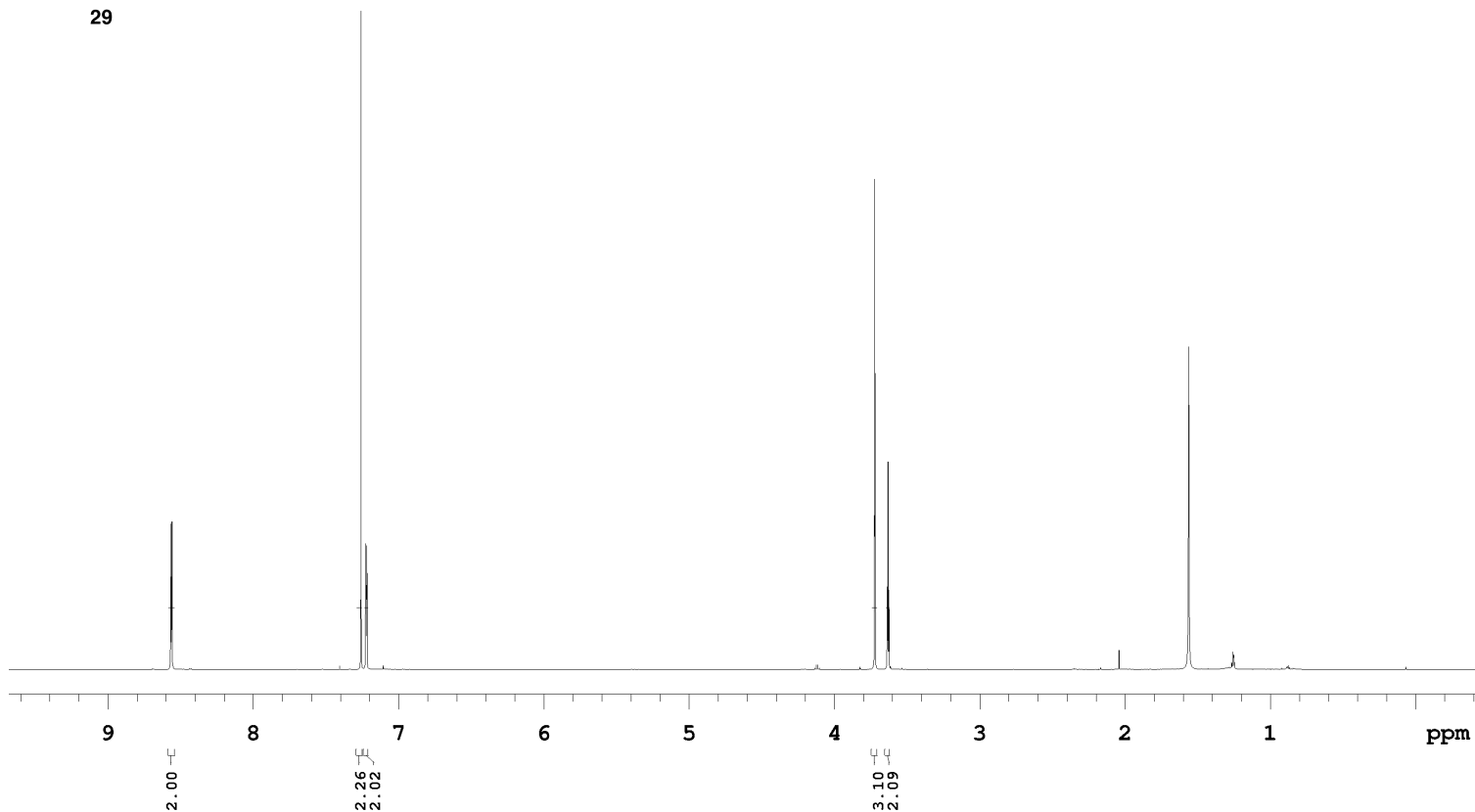
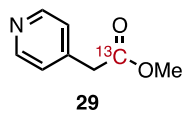
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **29.51**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-197-A

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **u500, Jun 12 2019**
Pulse Sequence: **s2pul**

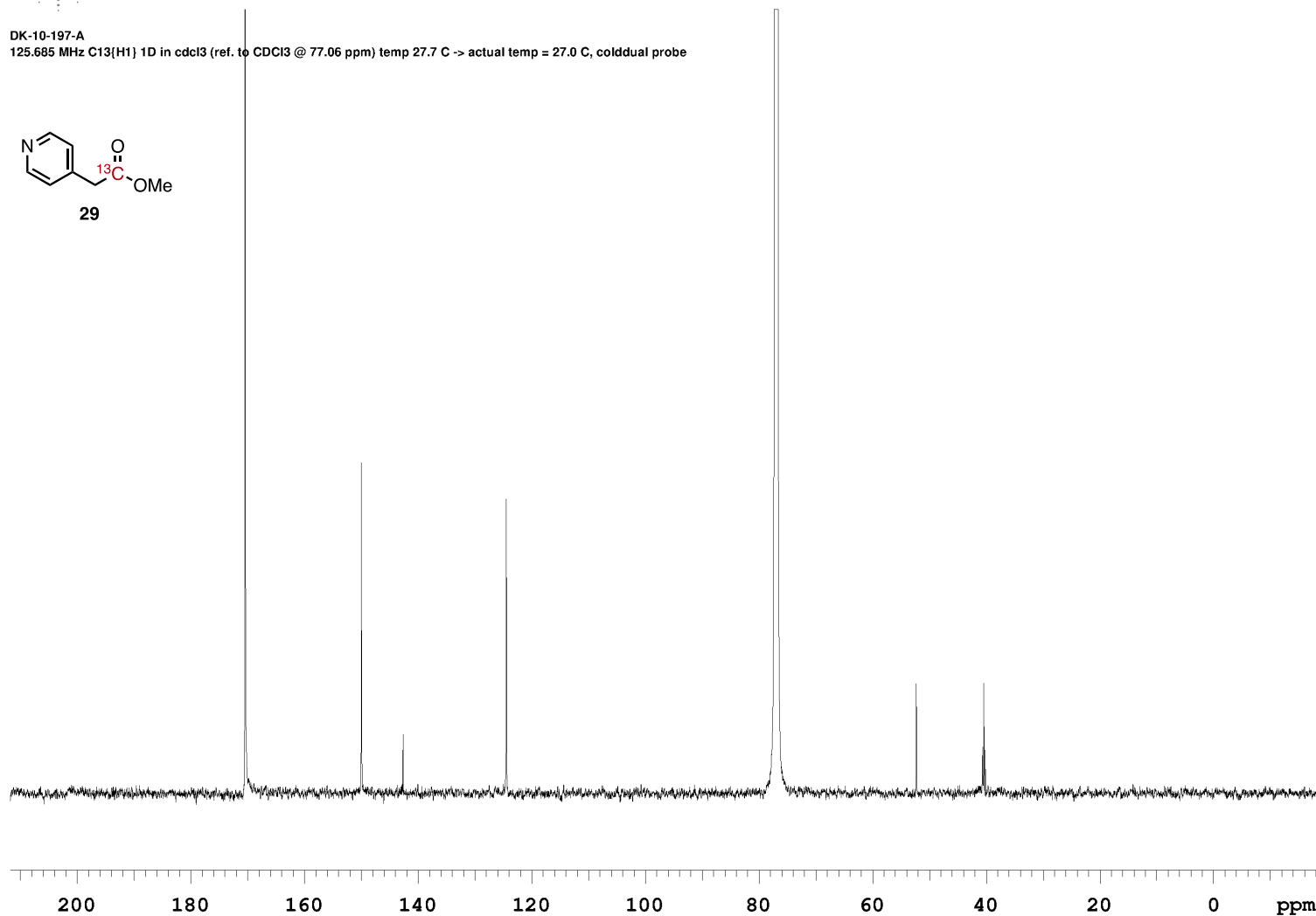
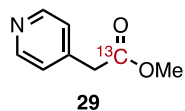
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **121.1**

Relaxation Delay(s): **1**
Completed Scans **2500**

DK-10-197-A

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, coldddual probe





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Recorded on: **v700, Jul 3 2019**
Pulse Sequence: **PRESAT**

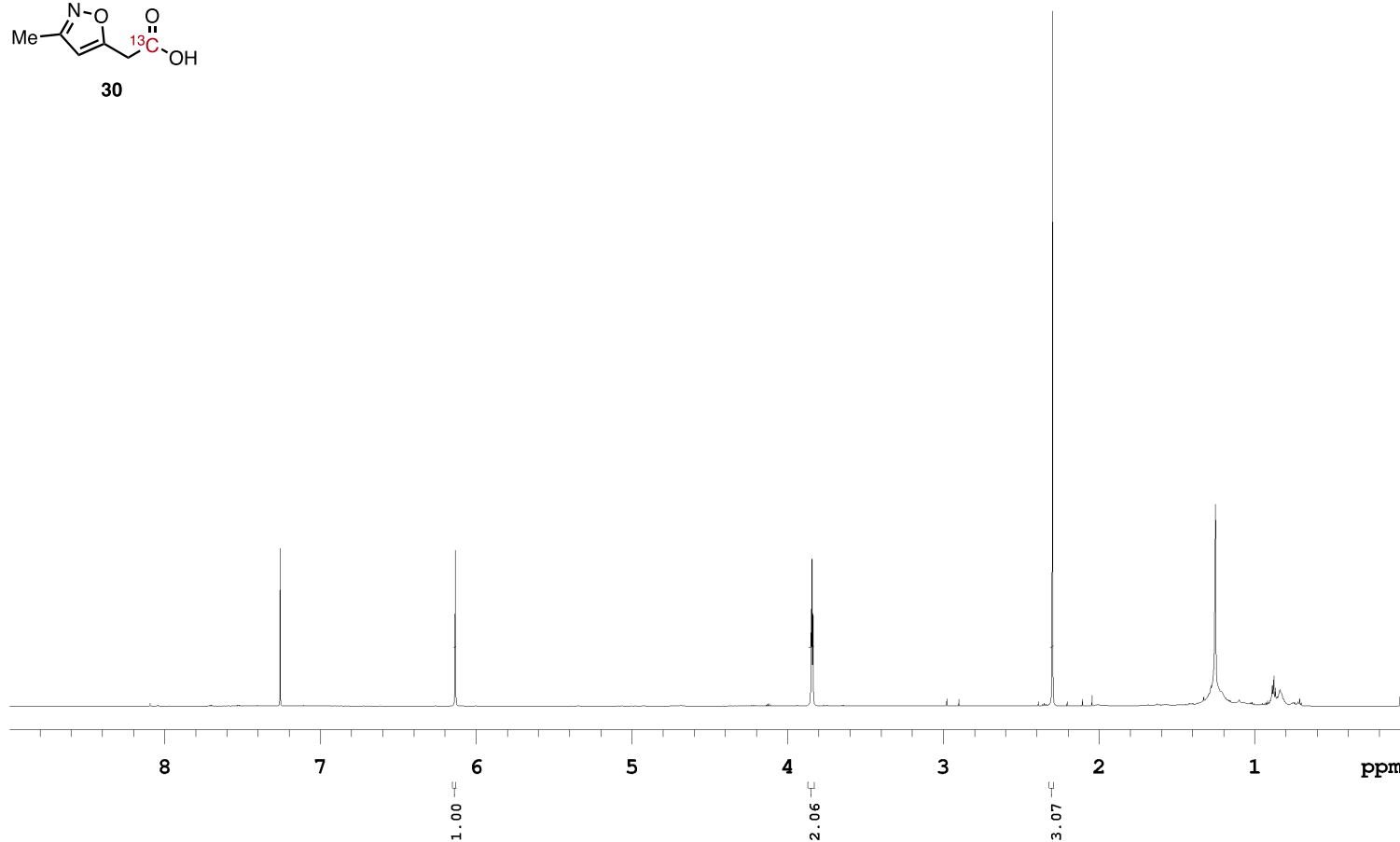
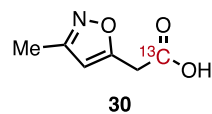
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.24**

Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL01-79_characterization

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **u500, Aug 22 2019**
Pulse Sequence: **s2pul**

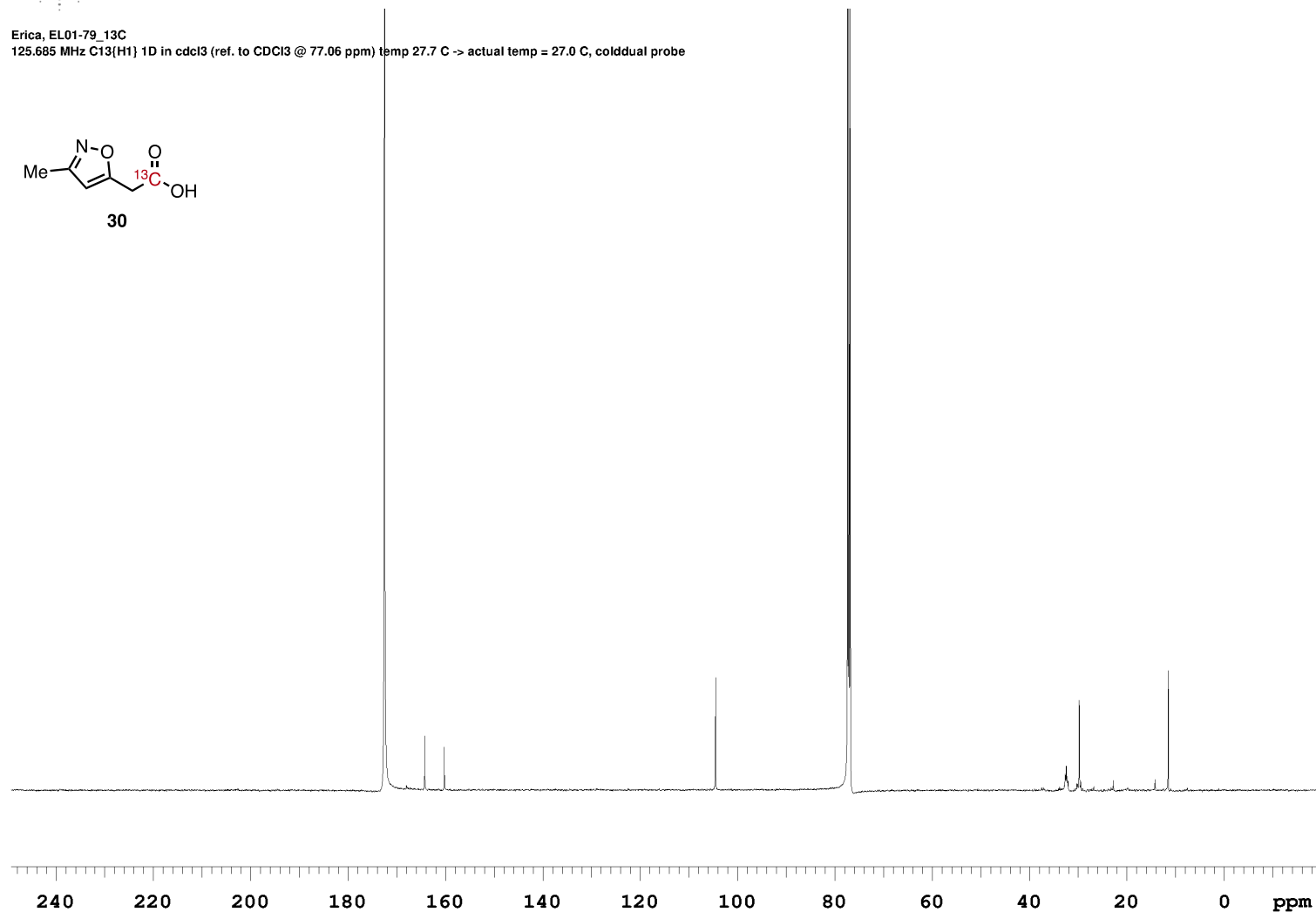
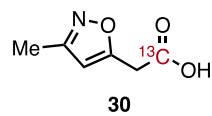
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **140.76**

Relaxation Delay(s): **1**
Completed Scans **1736**

Erica, EL01-79_13C

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **1400, Jul 2 2019**
Pulse Sequence: **s2pul**

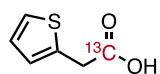
Sweep Width(Hz): **4801.92**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **4.998**
Hz per mm(Hz/mm): **15.97**

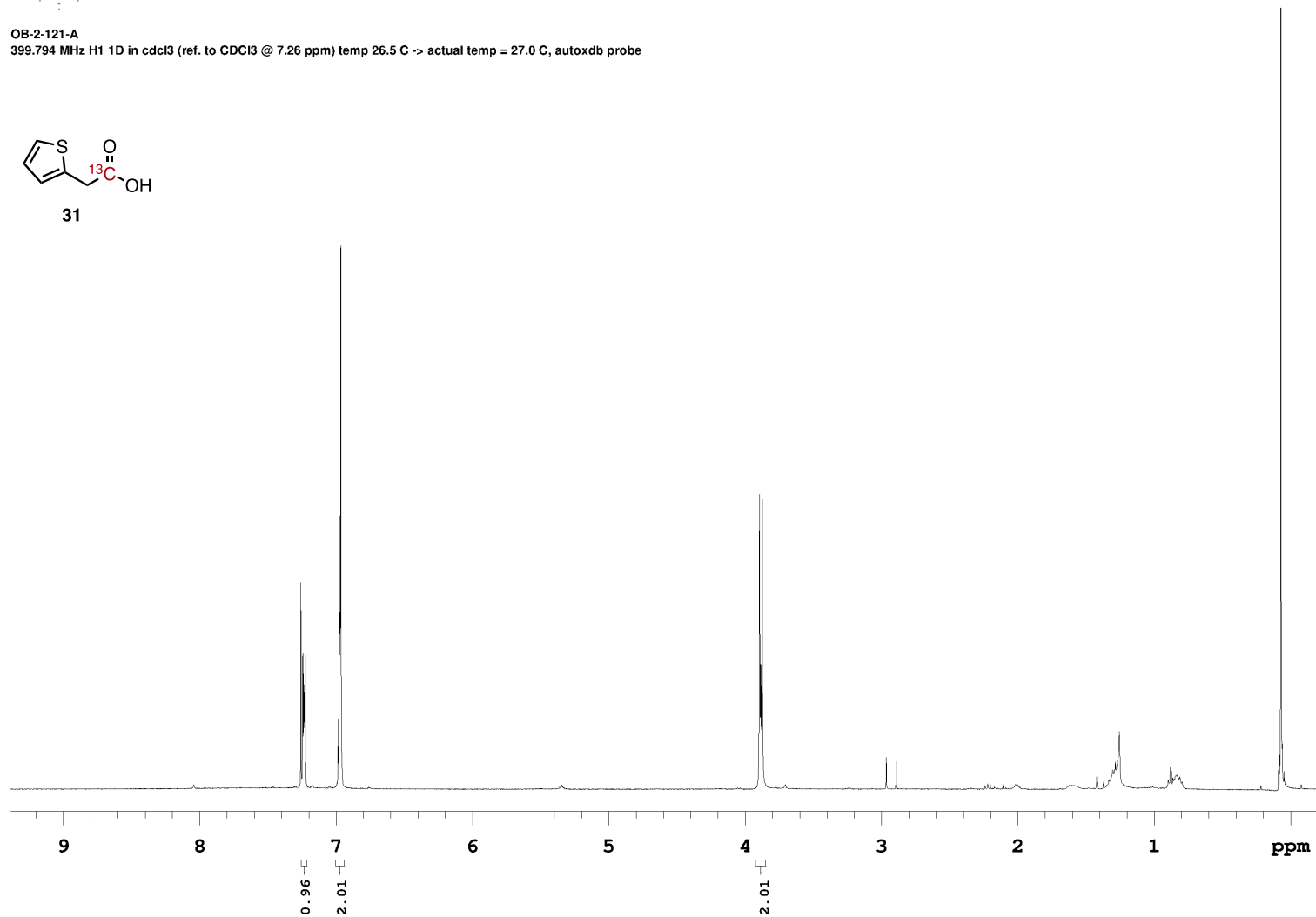
Relaxation Delay(s): **0.1**
Completed Scans **16**

OB-2-121-A

399.794 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.5 C -> actual temp = 27.0 C, autotdb probe



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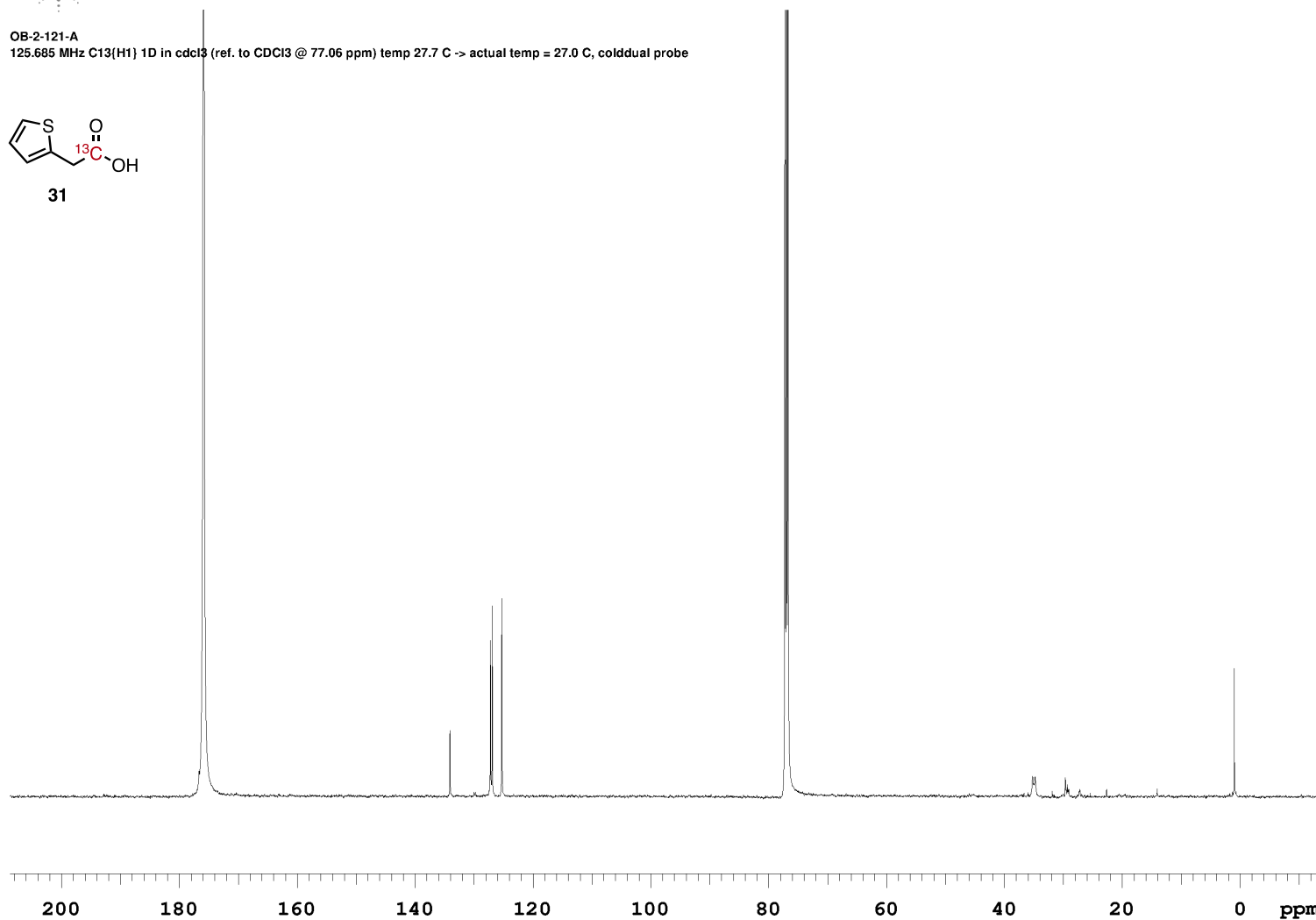
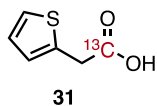
Recorded on: **u500, Jul 2 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **117.03**

Relaxation Delay(s): **1**
Completed Scans: **1644**

OB-2-121-A
125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Jun 12 2019**
Pulse Sequence: **PRESAT**

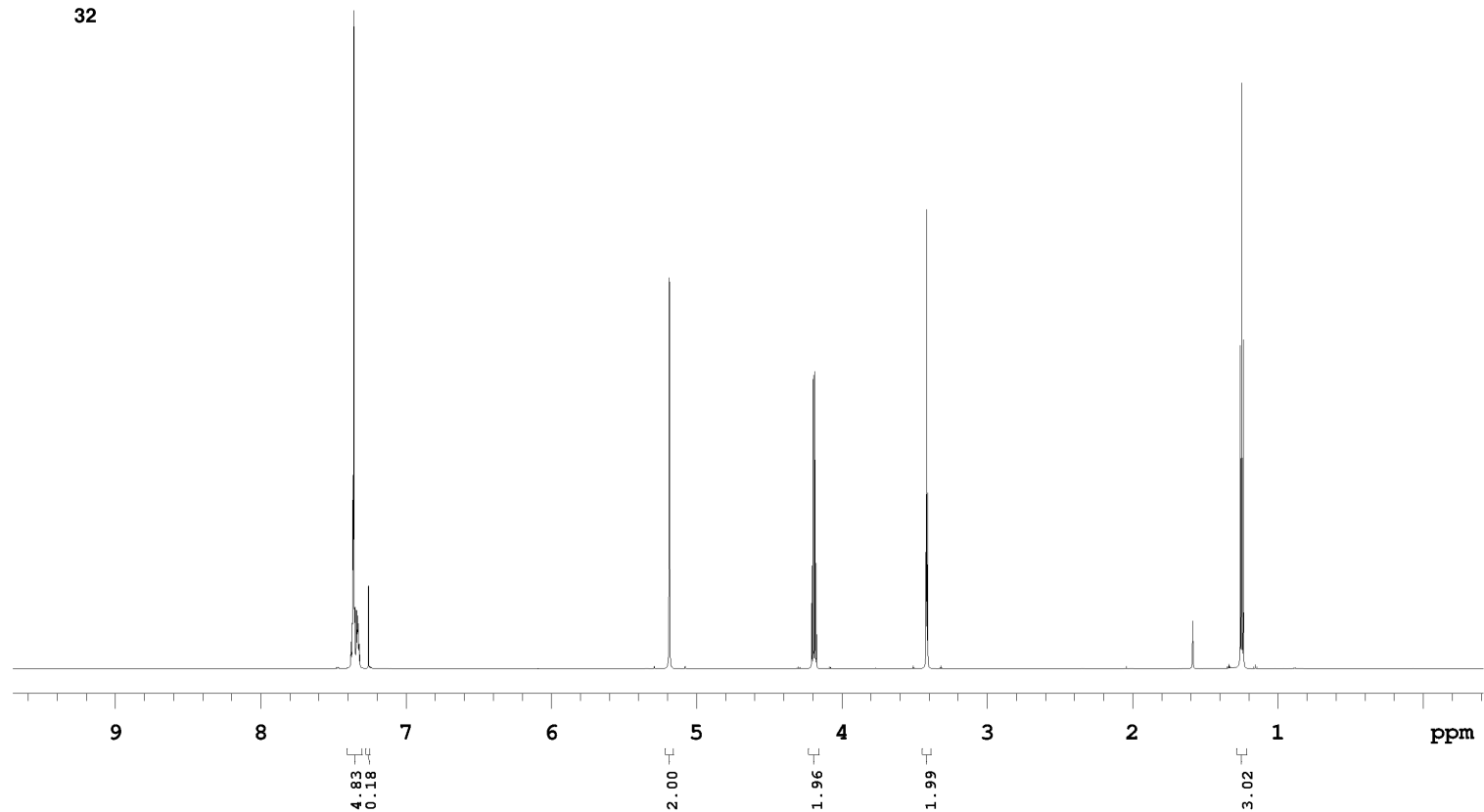
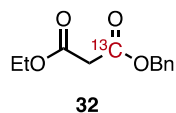
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **29.51**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-191-A

699.762 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **v700, Jun 12 2019**
Pulse Sequence: **s2pul**

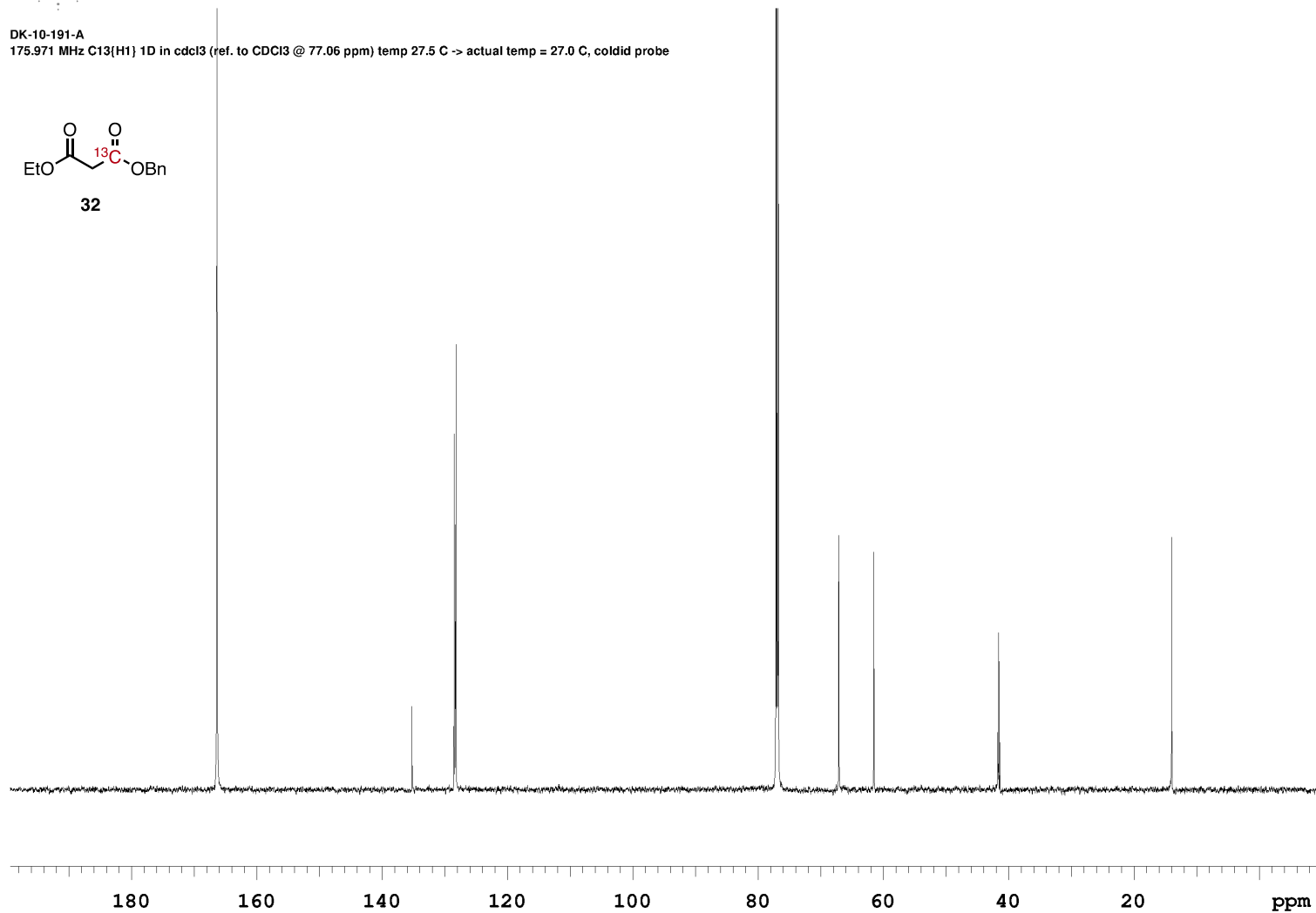
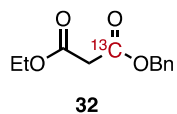
Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **176**

DK-10-191-A

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **u500, Jun 11 2019**
Pulse Sequence: **PRESAT**

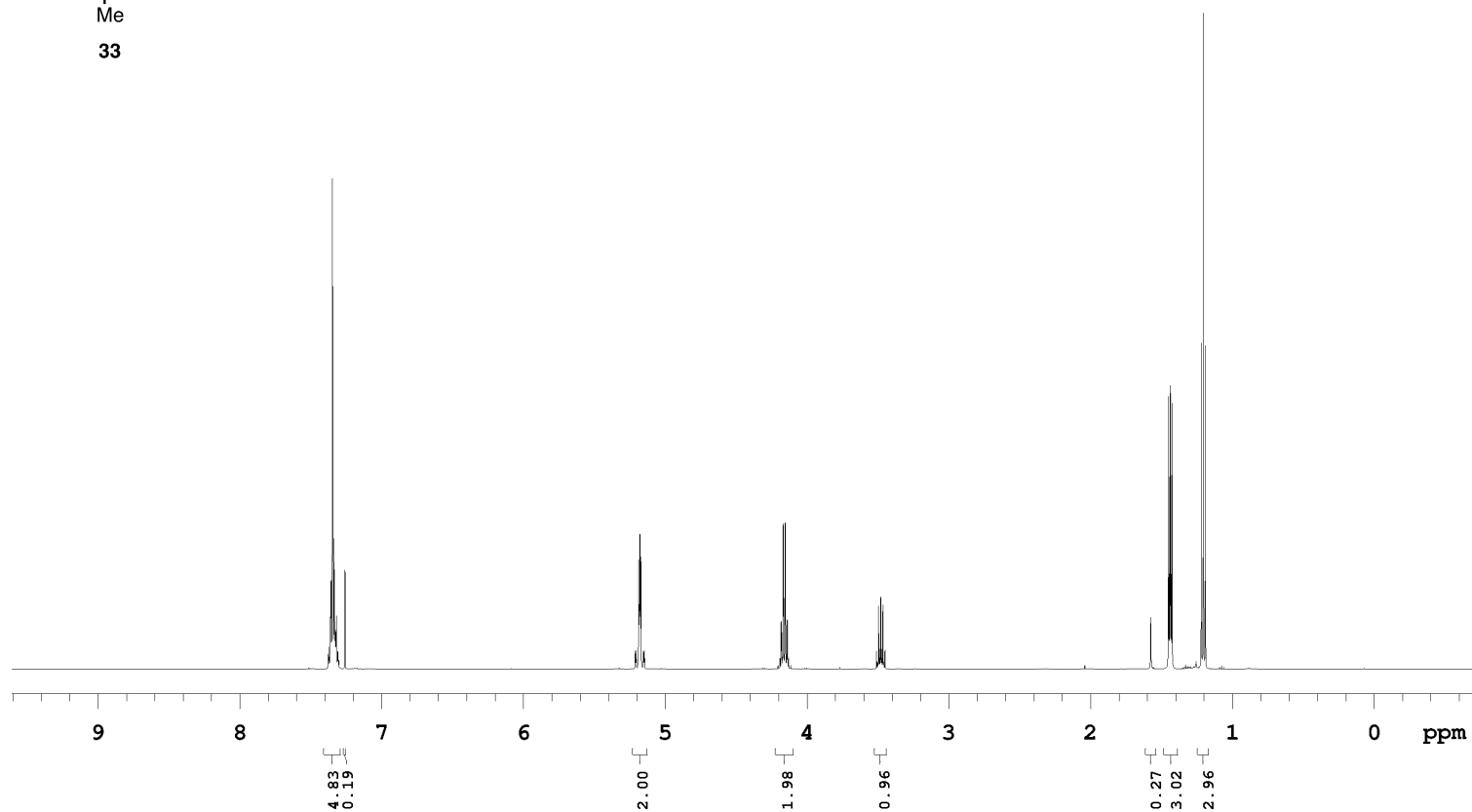
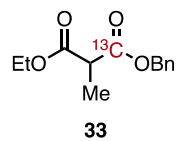
Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **21.5**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-191-D

499.787 MHz H1 1D in cdcl3 (ref. to CDCI3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **u500, Jun 12 2019**

Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**

Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**

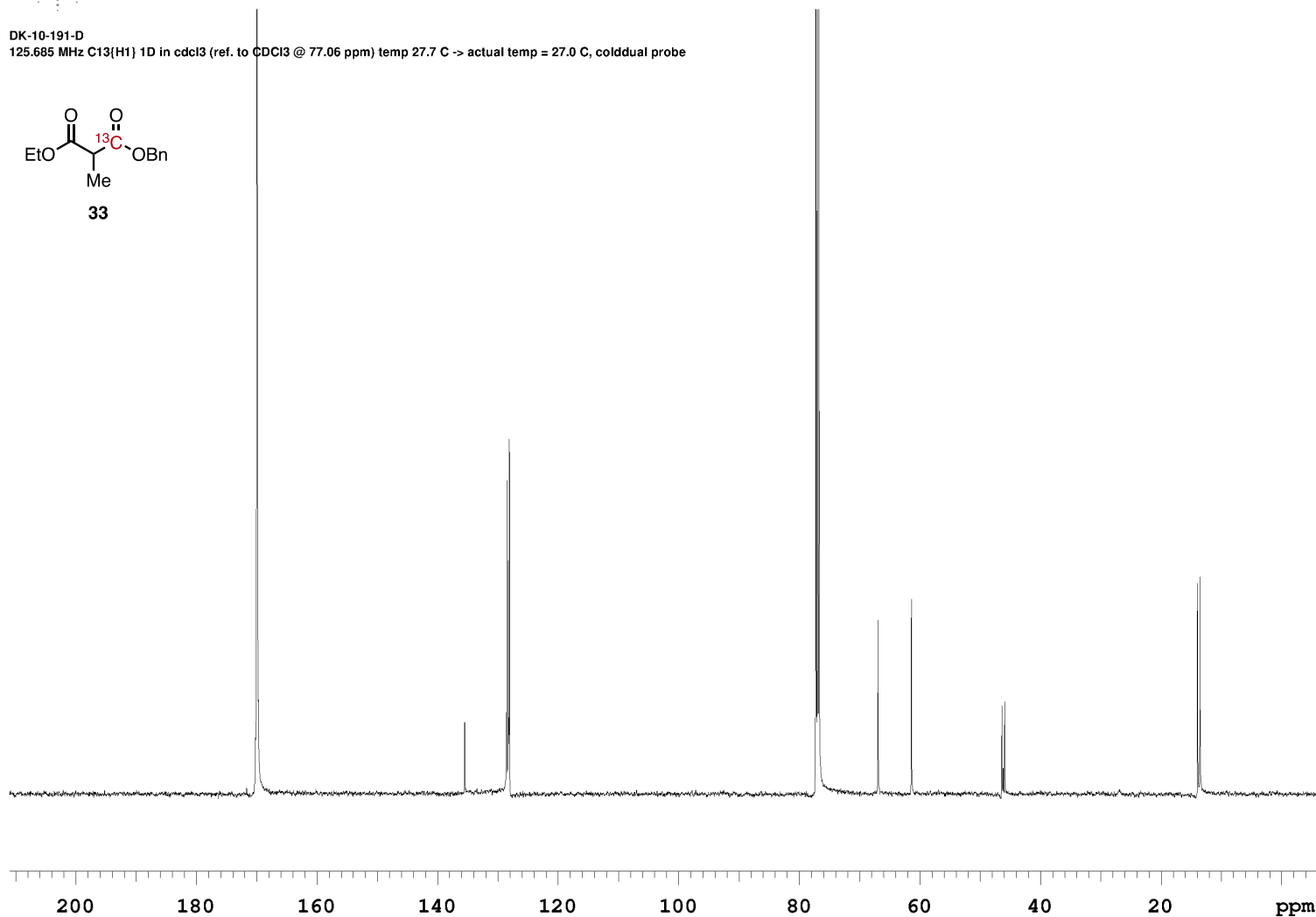
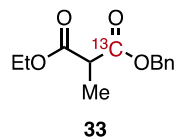
Hz per mm(Hz/mm): **114.11**

Relaxation Delay(s): **1**

Completed Scans **104**

DK-10-191-D

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **i600, Aug 7 2019**

Sweep Width(Hz): **7183.91**

Acquisition Time(s): **5**

Relaxation Delay(s): **0.1**

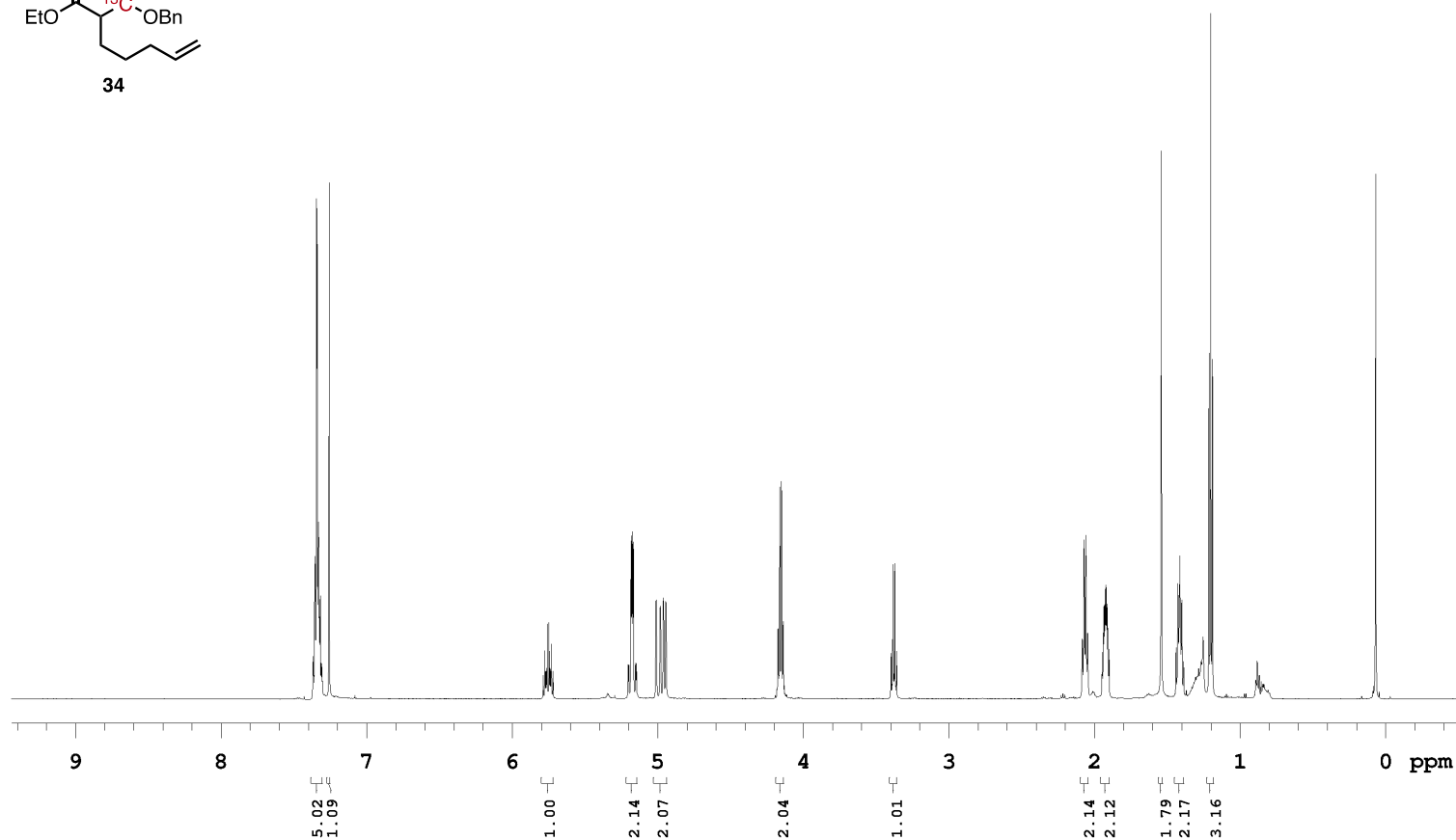
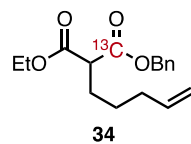
Pulse Sequence: **s2pul**

Digital Res.(Hz/pt): **0.11**

Hz per mm(Hz/mm): **24.91**

Completed Scans **32**

ob-02-177 599.926 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.2 C -> actual temp = 27.0 C, autoxid probe





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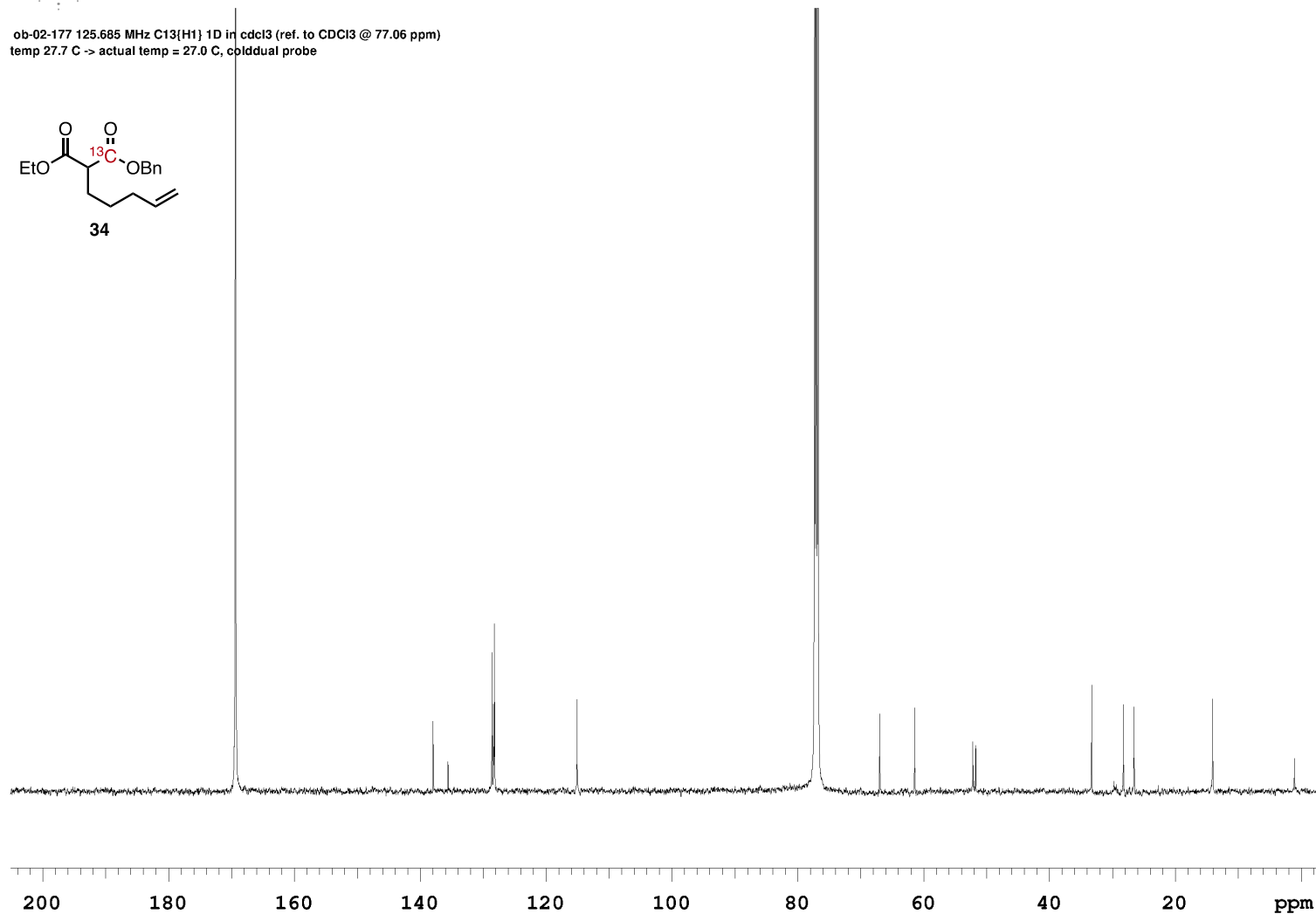
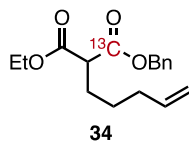
Recorded on: **u500, Aug 7 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **109.11**

Relaxation Delay(s): **1**
Completed Scans **512**

ob-02-177 125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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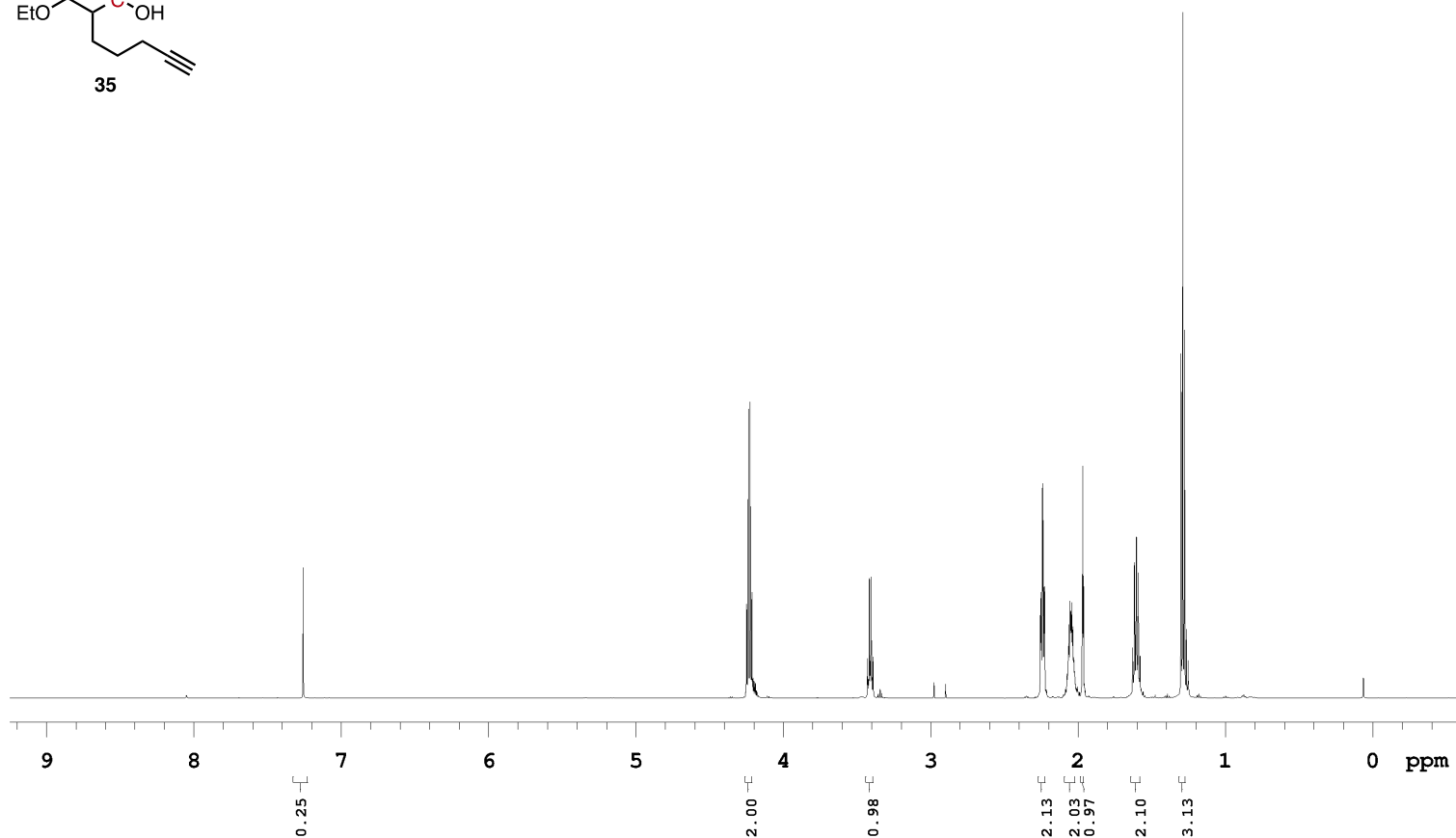
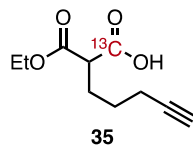
Recorded on: **i600, Jul 4 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **7183.91**
Digital Res.(Hz/pt): **0.11**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **24.56**

Relaxation Delay(s): **0.1**
Completed Scans **16**

ob-02-127-b 599.926 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.2 C -> actual temp = 27.0 C, autoxid probe





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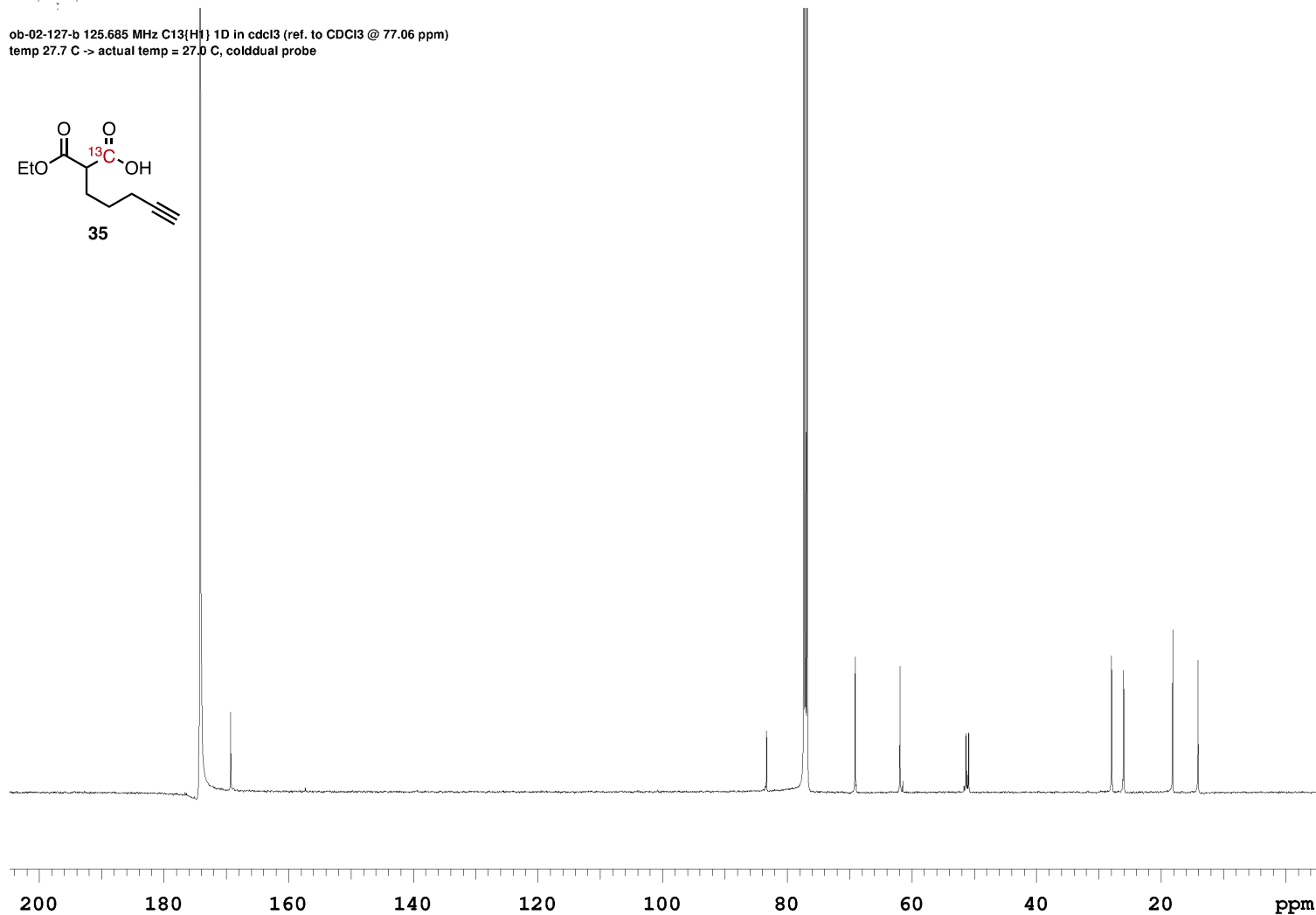
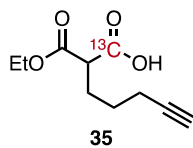
Recorded on: **u500, Jul 4 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

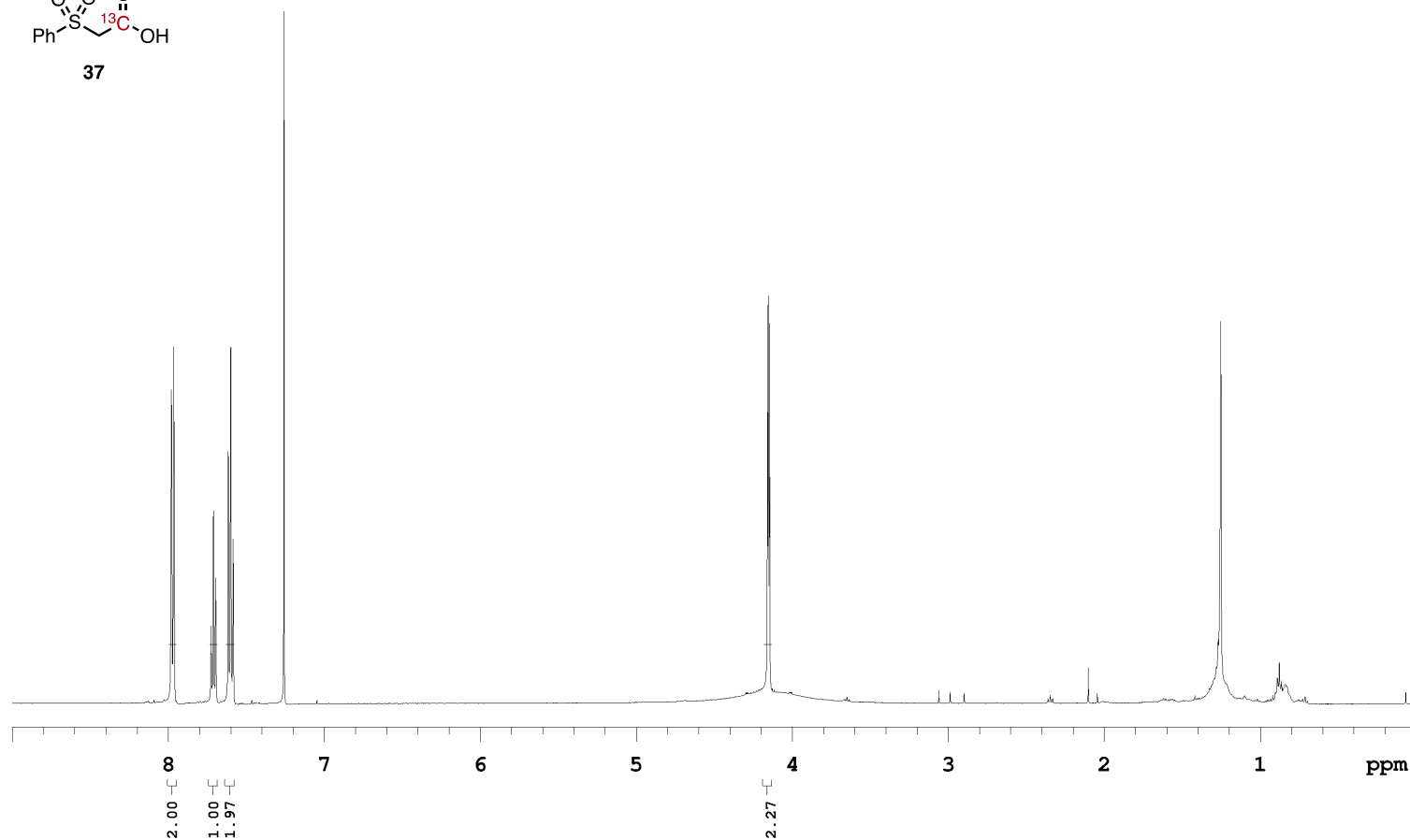
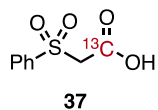
Acquisition Time(s): **1**
Hz per mm(Hz/mm): **110.3**

Relaxation Delay(s): **1**
Completed Scans **720**

ob-02-127-b 125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, cold dual probe



Erica, EL01-71_characterization
 499.787 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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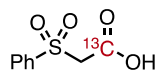
Recorded on: **u500, Jul 23 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

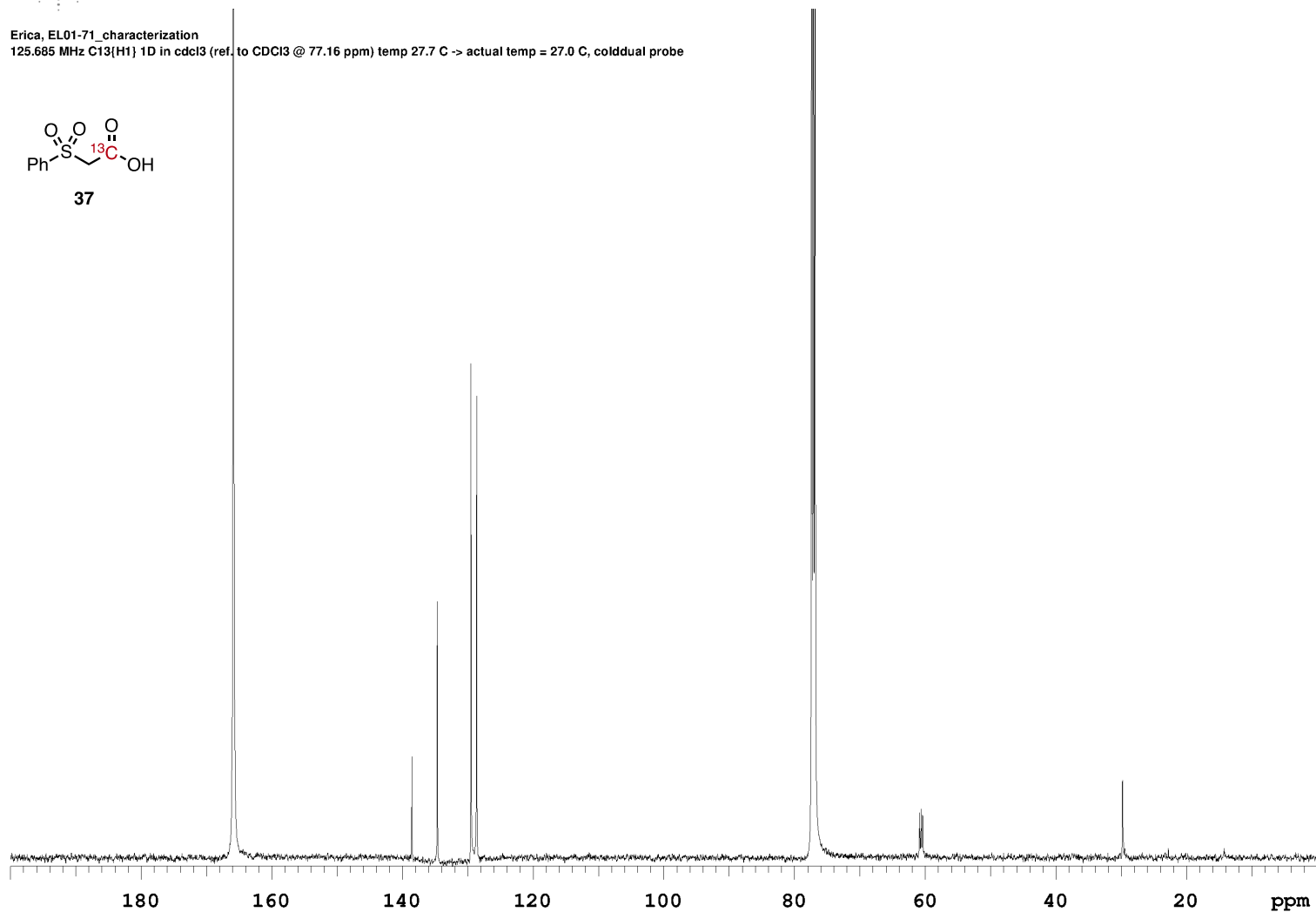
Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.77**

Relaxation Delay(s): **1**
Completed Scans **644**

Erica, EL01-71_characterization
125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe



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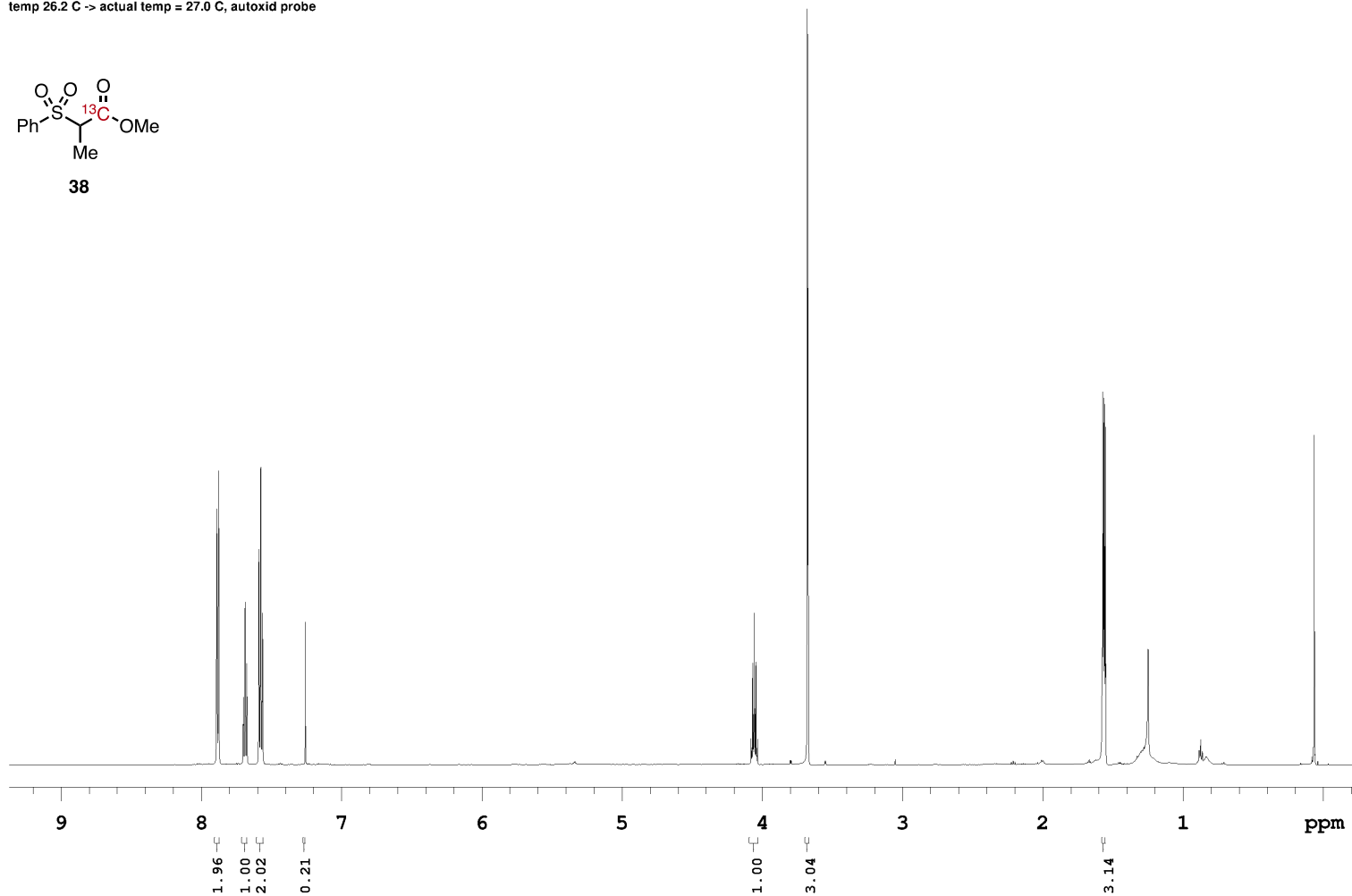
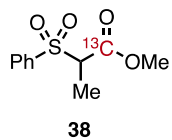
Recorded on: **i600, Aug 26 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **7183.91**
Digital Res.(Hz/pt): **0.11**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **23.95**

Relaxation Delay(s): **0.1**
Completed Scans **16**

ob-02-193 599.926 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.2 C -> actual temp = 27.0 C, autoxid probe





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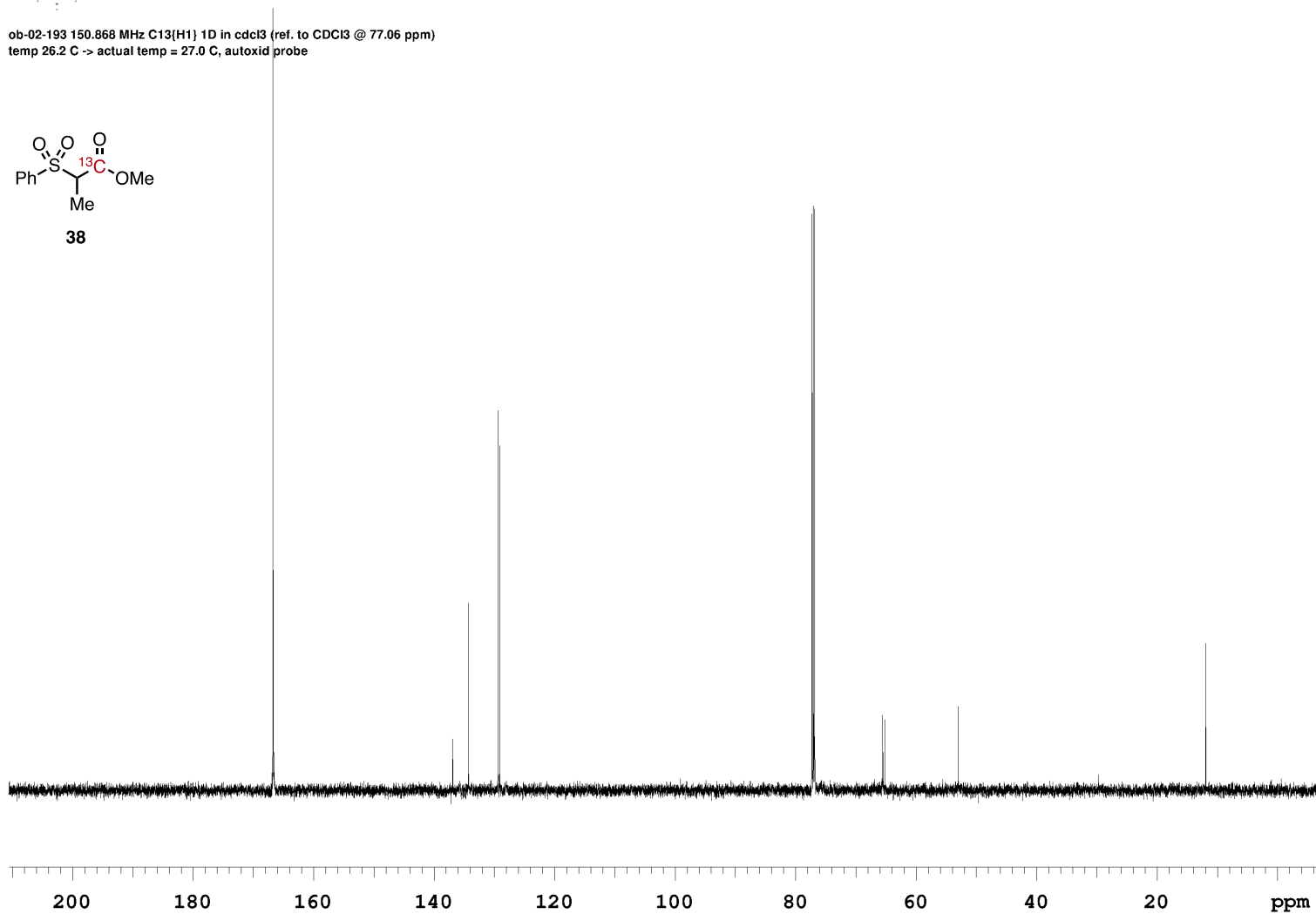
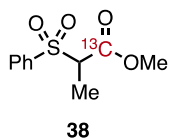
Recorded on: **i600, Aug 26 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **40322.6**
Digital Res.(Hz/pt): **0.31**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **136.59**

Relaxation Delay(s): **1**
Completed Scans **432**

ob-02-193 150.868 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 26.2 C -> actual temp = 27.0 C, autotxid probe





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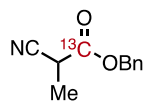
Recorded on: **v700, Jun 11 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

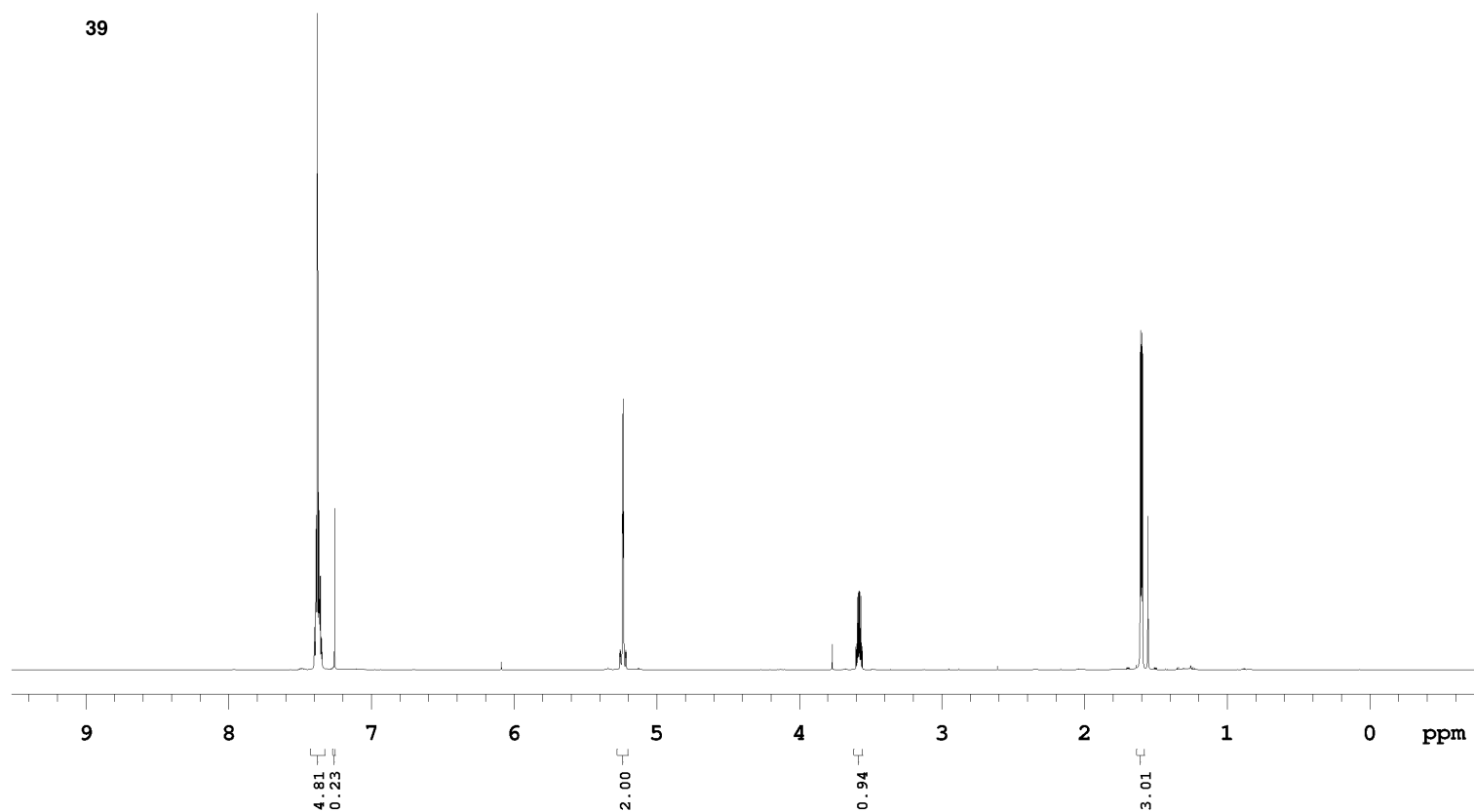
Acquisition Time(s): **5**
Hz per mm(Hz/mm): **29.92**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-185-A-prod
699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe



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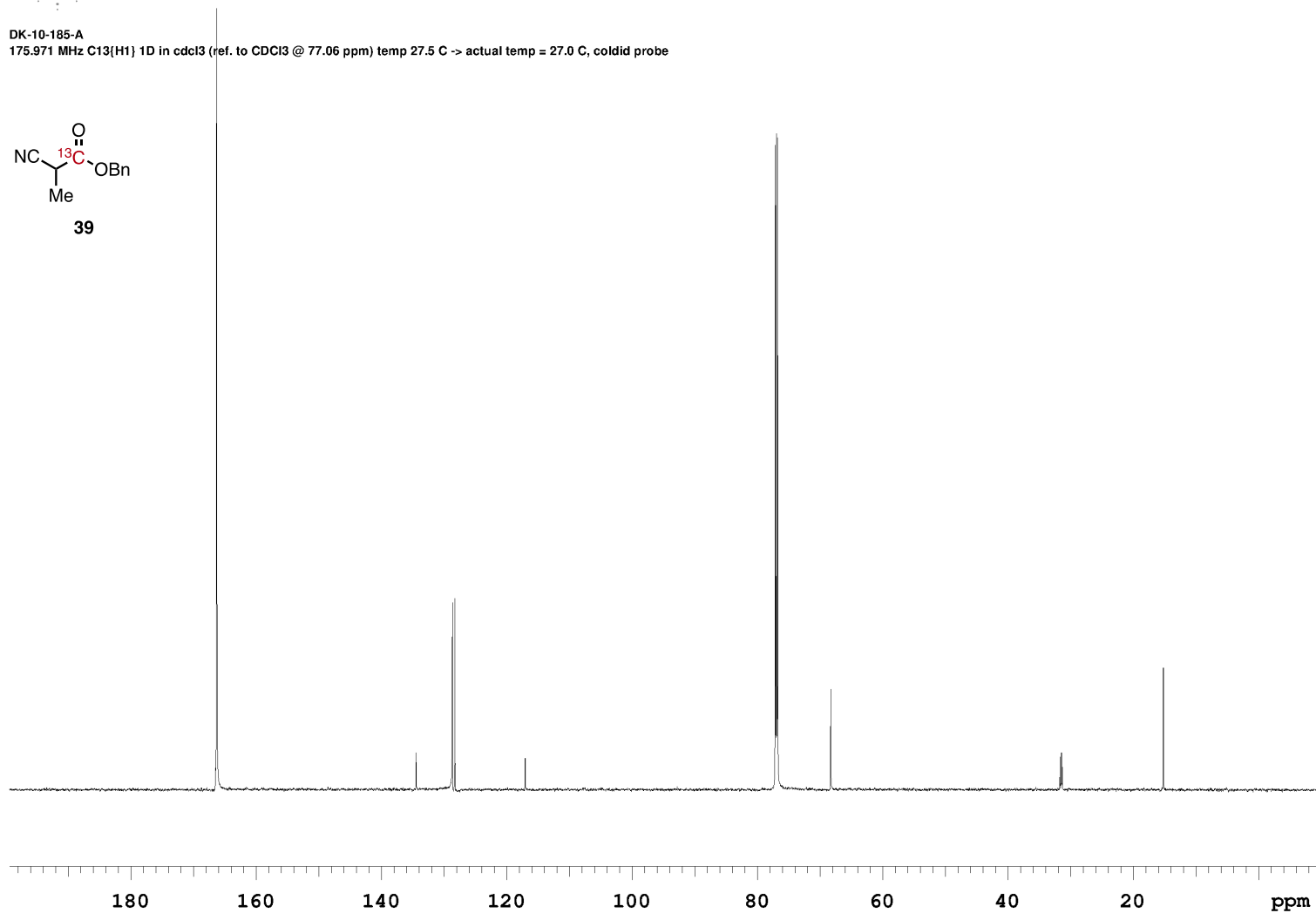
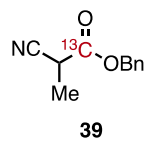
Recorded on: **v700, Jun 11 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **336**

DK-10-185-A
175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **u500, May 28 2019**
Pulse Sequence: **PRESAT**

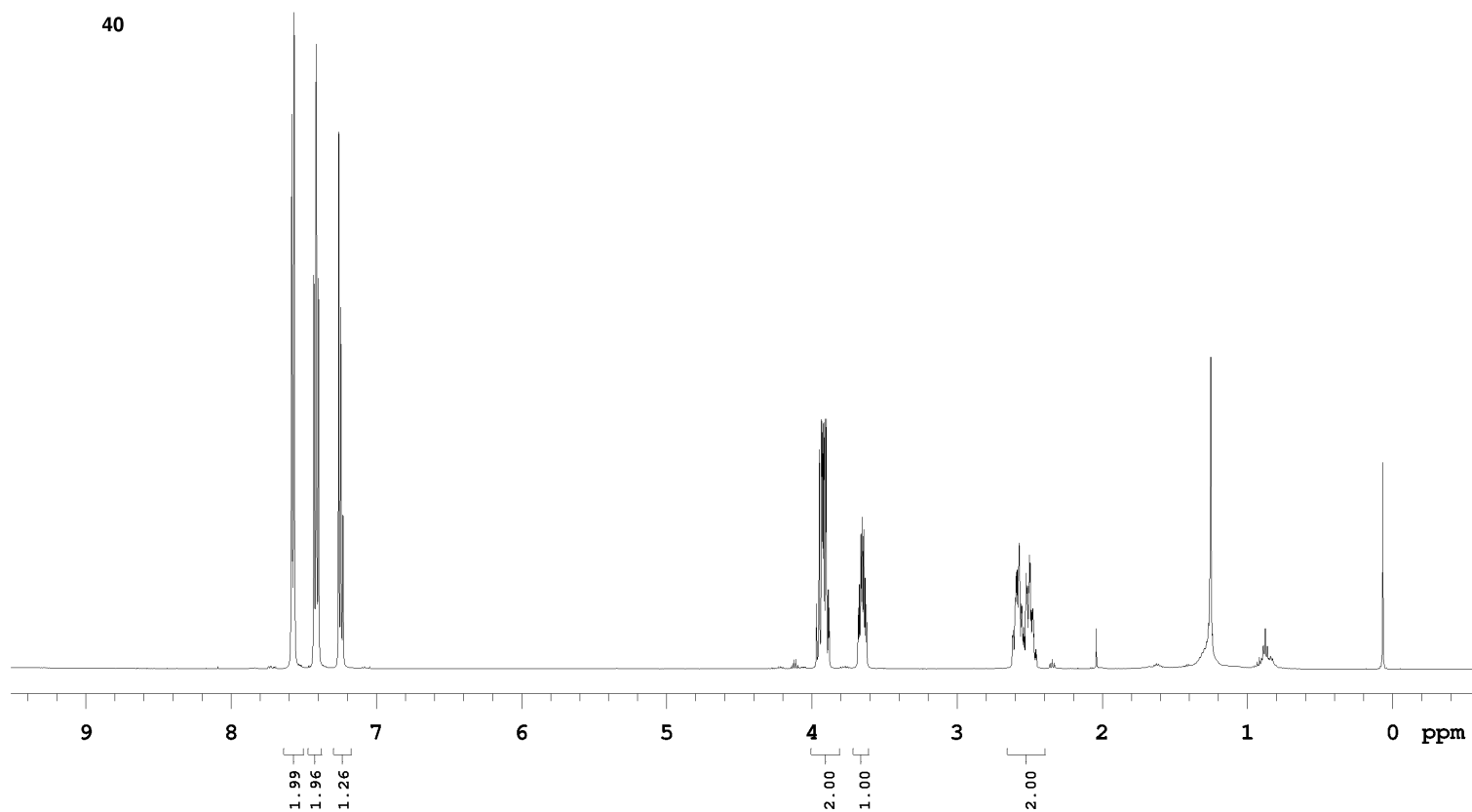
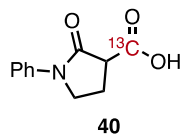
Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.99**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-159-B

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Jun 8 2019**
Pulse Sequence: **s2pul**

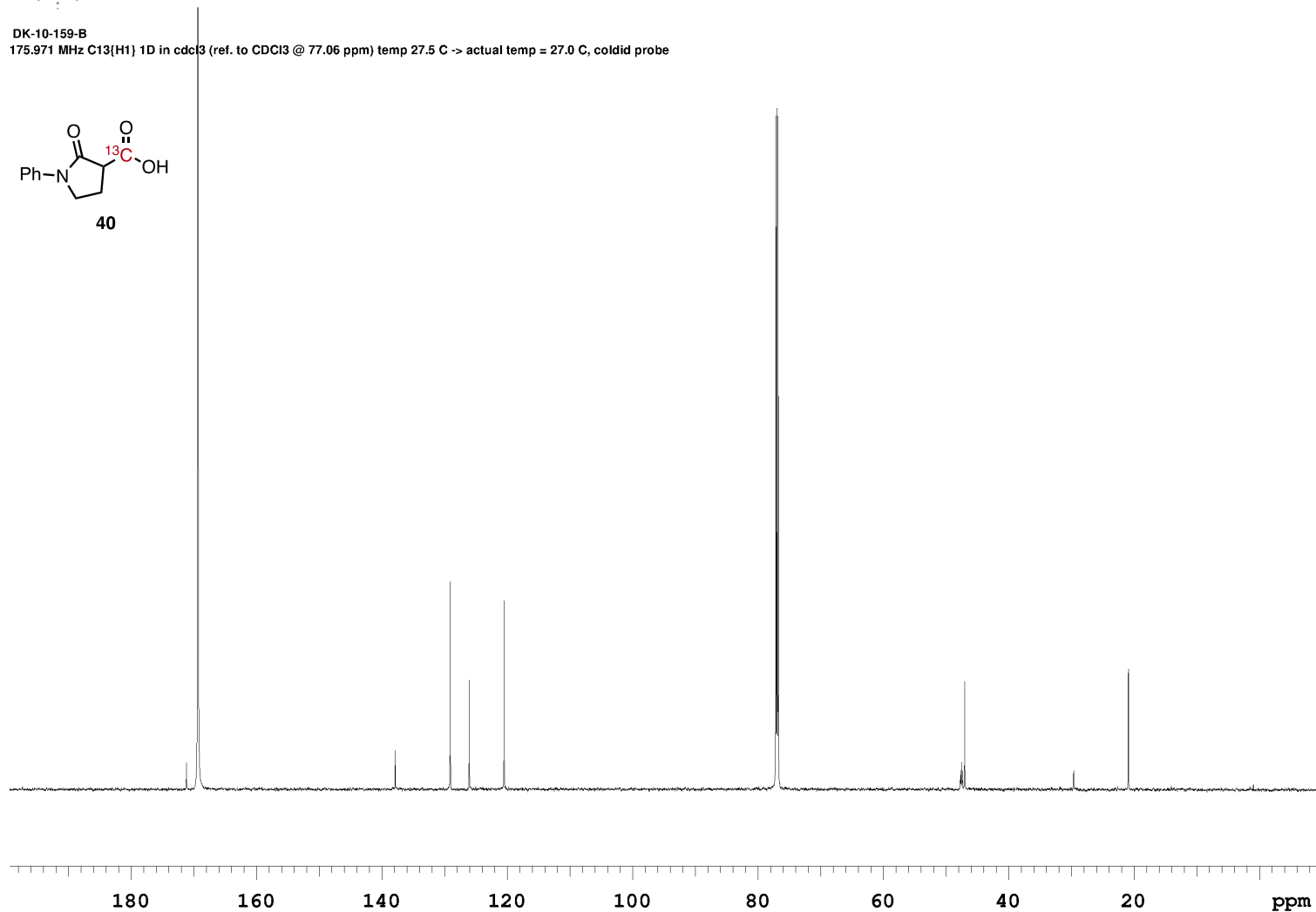
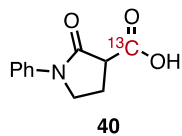
Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **280**

DK-10-159-B

175.971 MHz C13{H1} 1D in cdc13 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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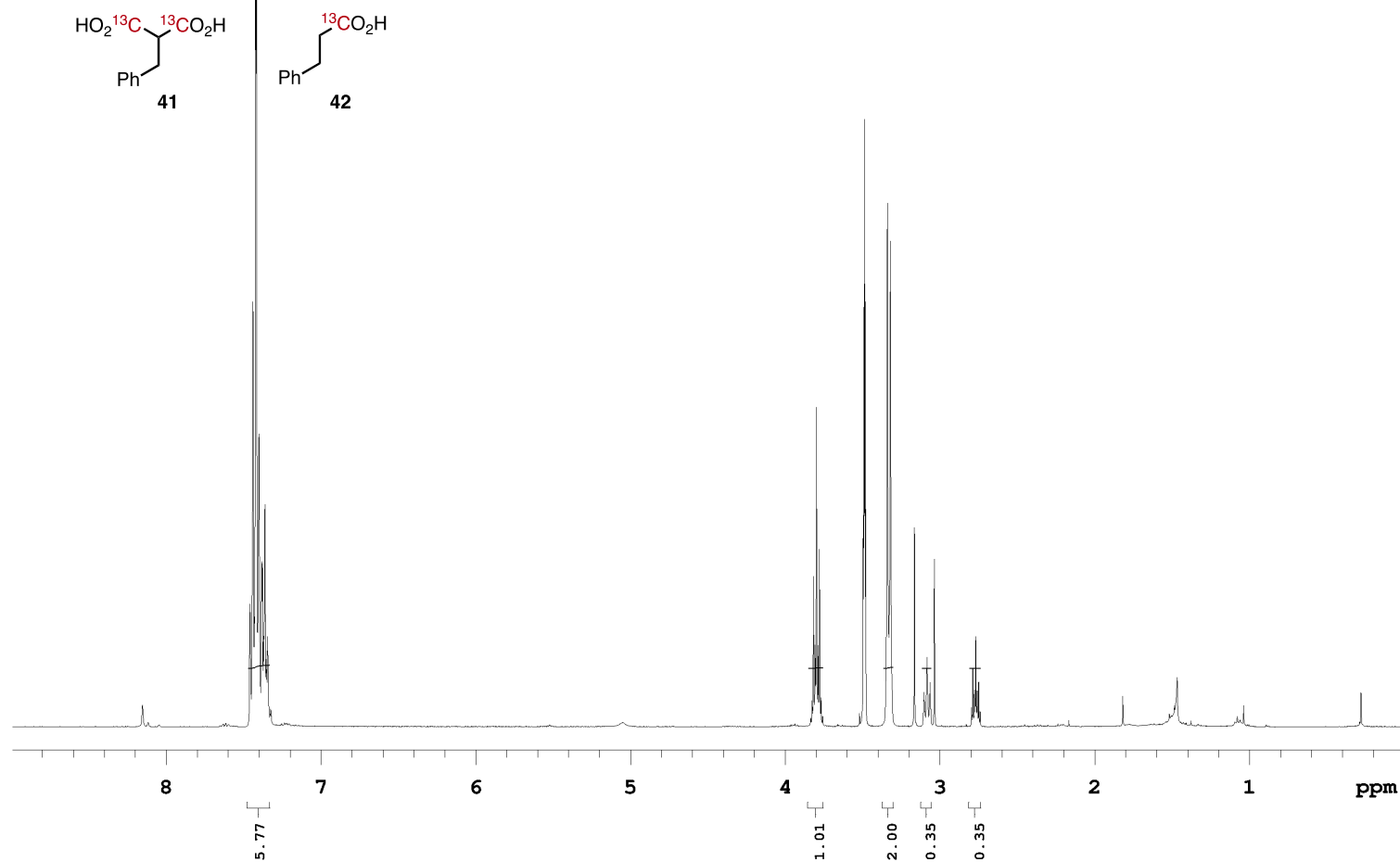
Recorded on: **mr400, Jan 23 2020**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **3**
Hz per mm(Hz/mm): **15**

Relaxation Delay(s): **2**
Completed Scans **16**

399.980 MHz H1 1D in cd3od (ref. to CD3OD @ 3.30 ppm)
temp 25.9 C -> actual temp = 27.0 C, orienmr probe





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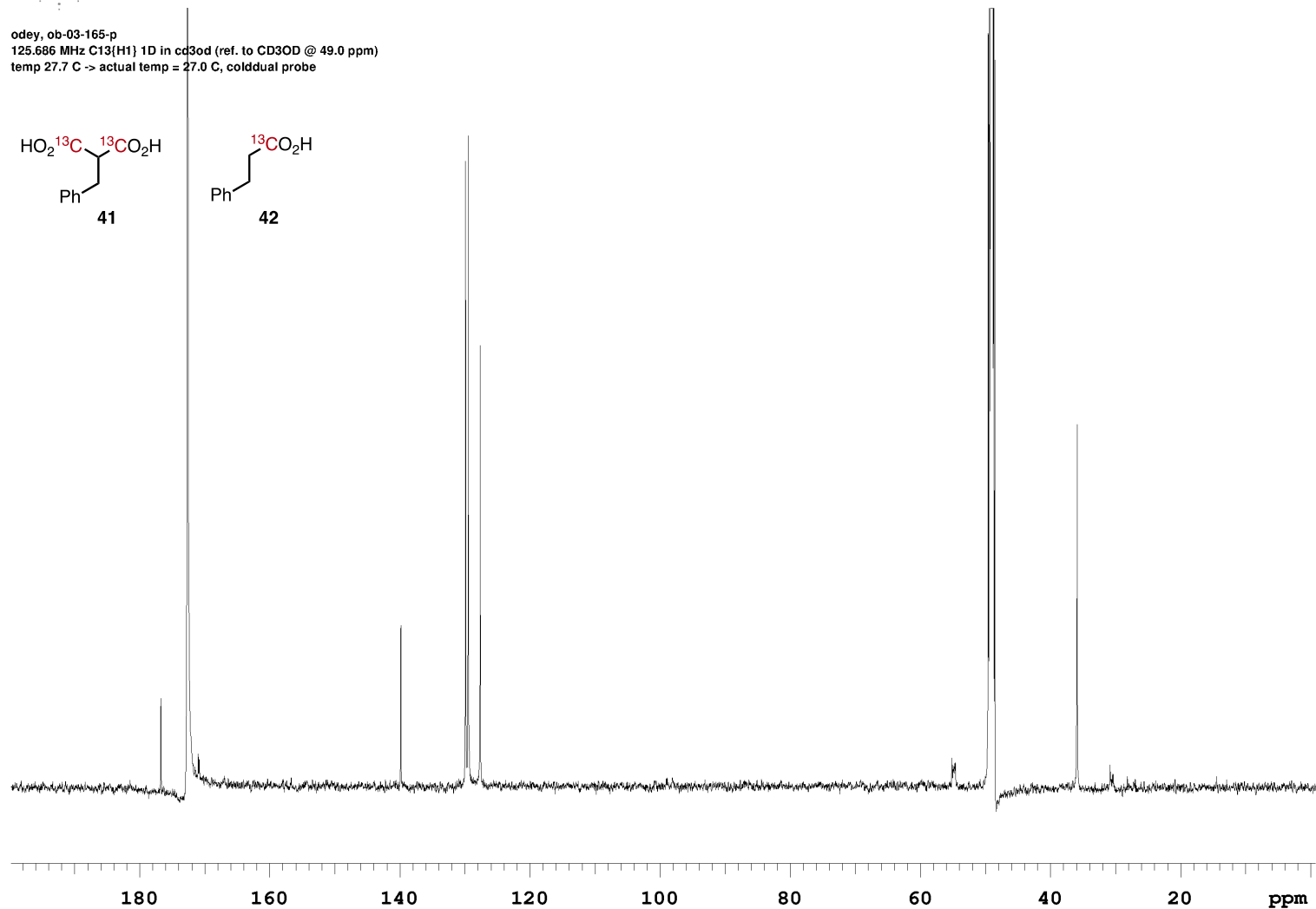
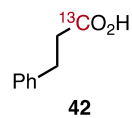
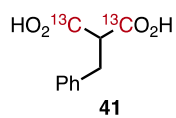
Recorded on: **u500, Jan 24 2020**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.98**

Relaxation Delay(s): **1**
Completed Scans **592**

odey, ob-03-165-p
125.686 MHz C13{H1} 1D in cd3od (ref. to CD3OD @ 49.0 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe





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Recorded on: **v700, Jul 18 2019**
Pulse Sequence: **PRESAT**

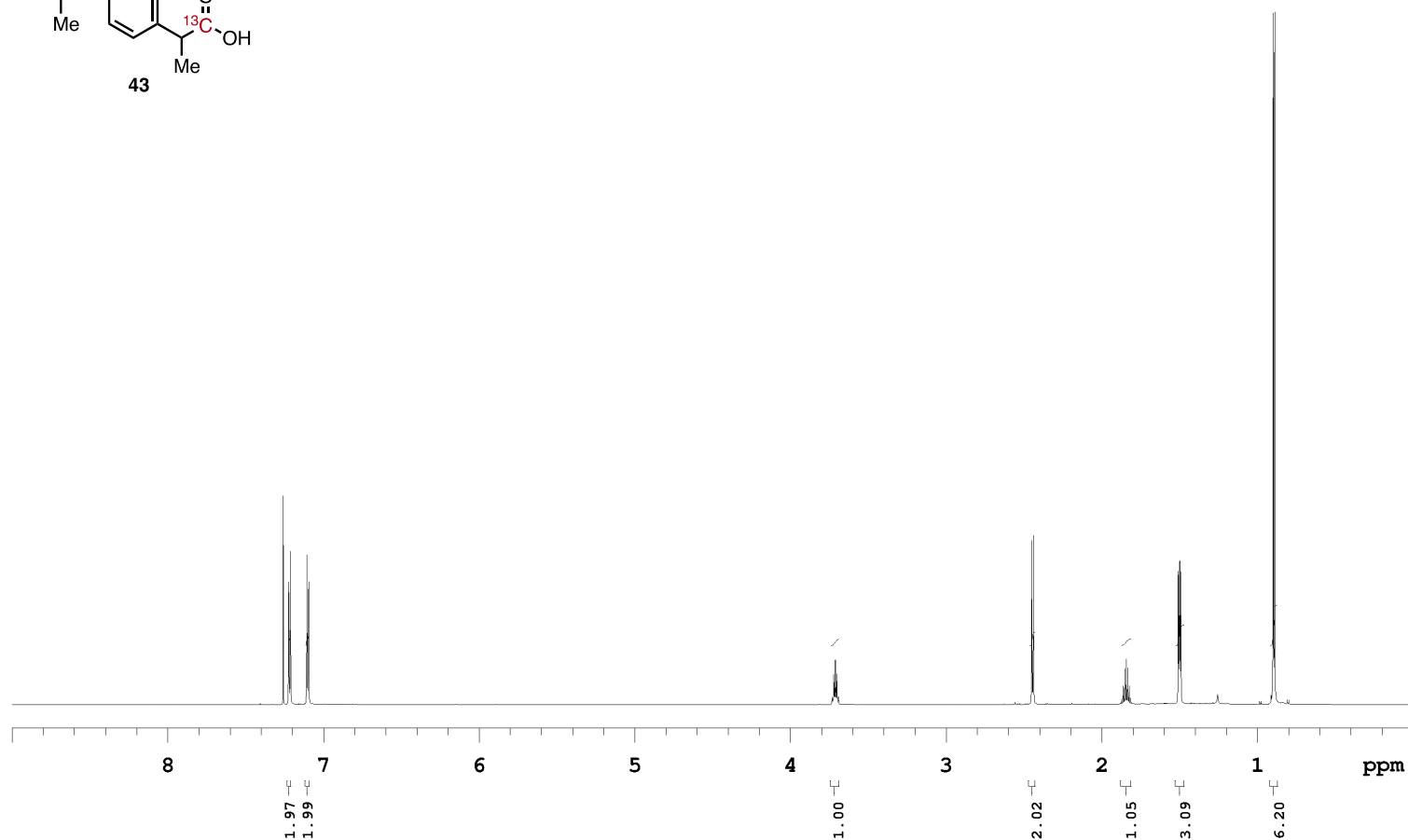
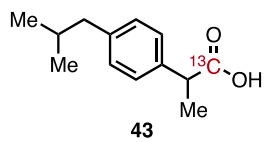
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.24**

Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL01-67_characterization

699.762 MHz ¹H 1D in cdcl₃ (ref. to CDCl₃ @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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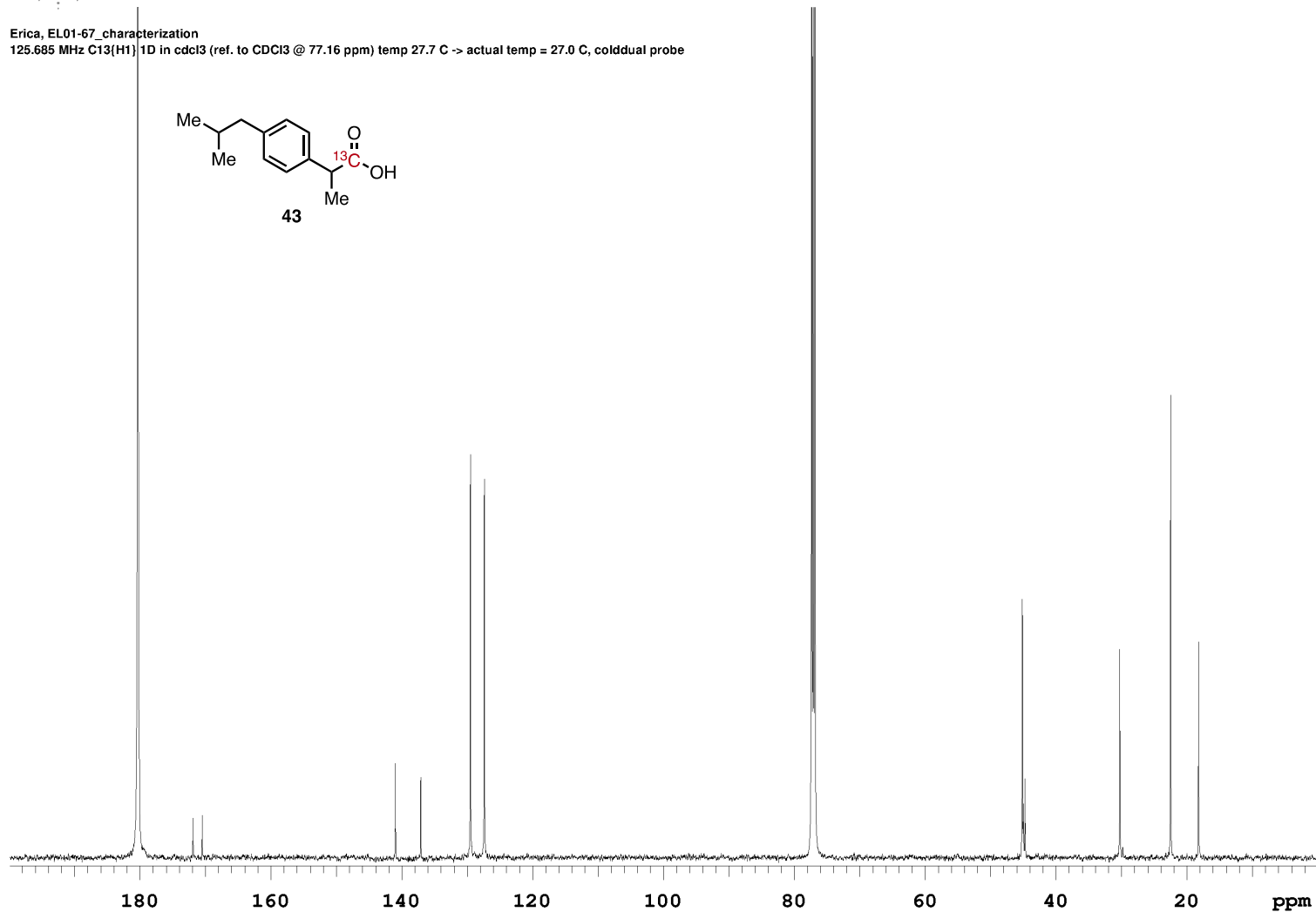
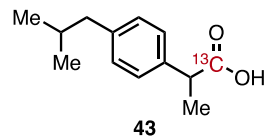
Recorded on: **u500, Sep 7 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.77**

Relaxation Delay(s): **1**
Completed Scans **276**

Erica, EL01-67_characterization
125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Department of Chemistry, University of Alberta

Recorded on: **v700, Jul 17 2019**
Pulse Sequence: **PRESAT**

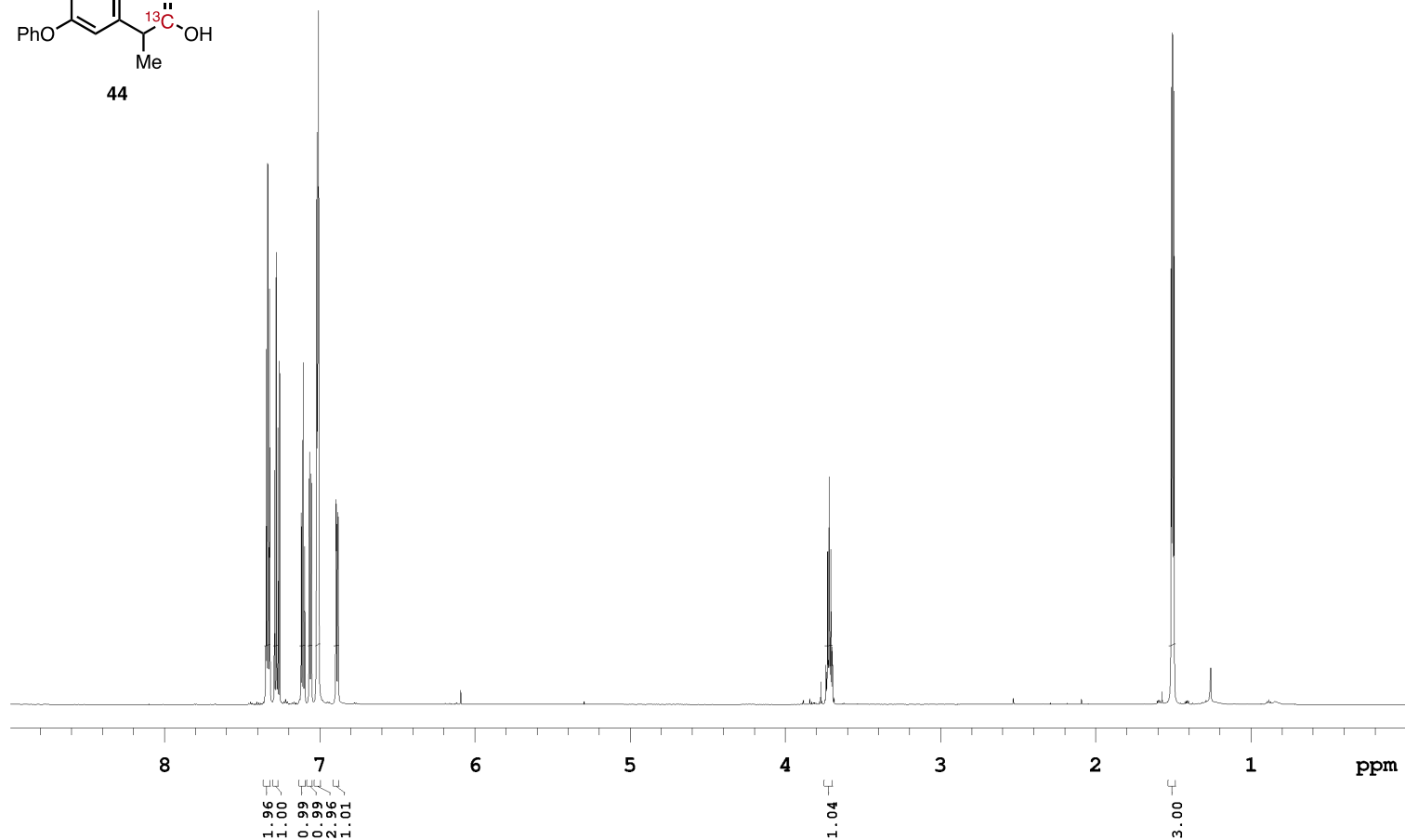
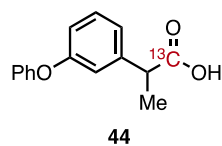
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.22**

Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL01-69B_characterization

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **u500, Sep 7 2019**
Pulse Sequence: **s2pul**

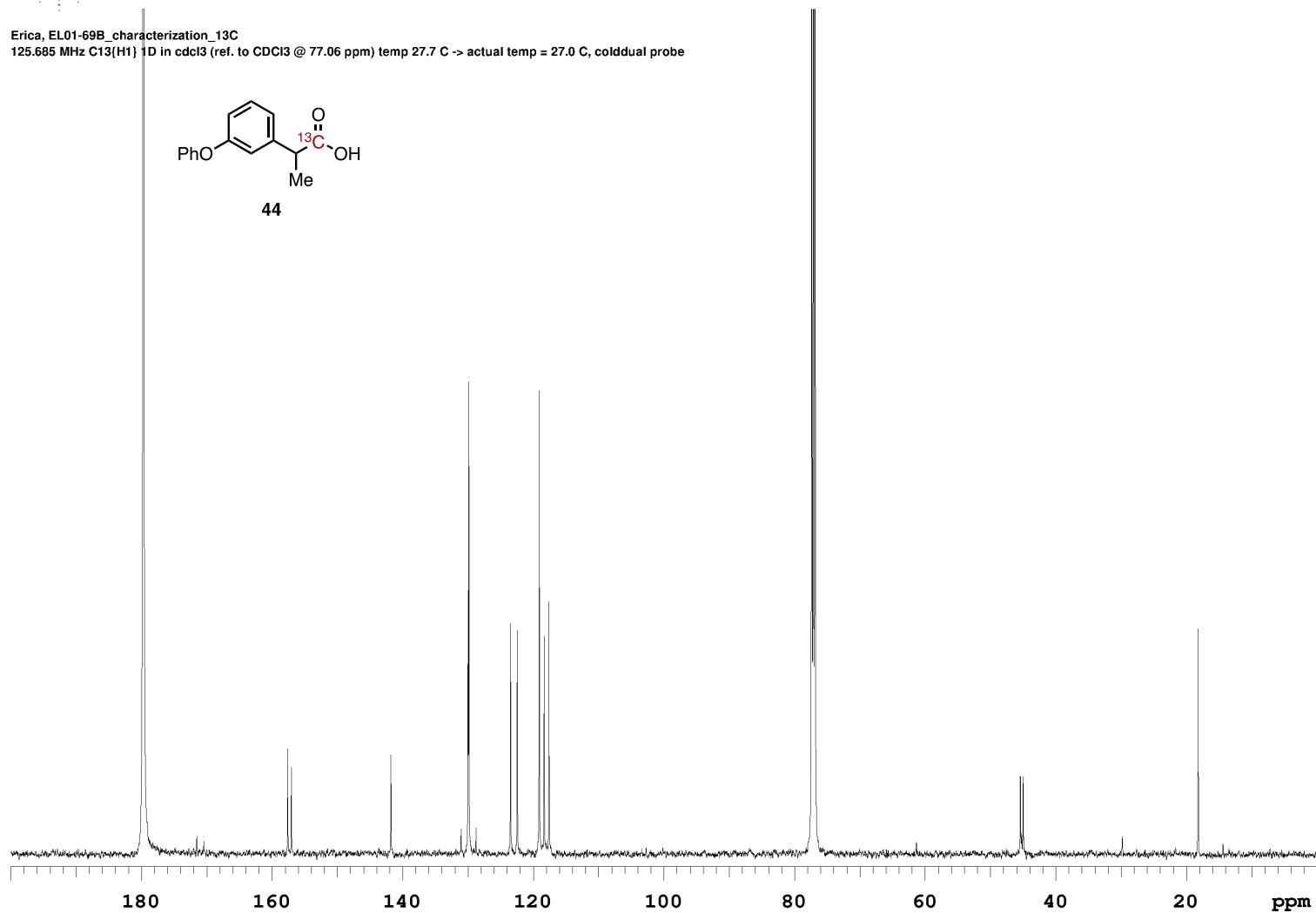
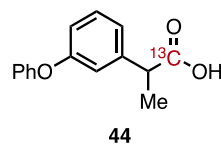
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.87**

Relaxation Delay(s): **1**
Completed Scans **268**

Erica, EL01-69B_characterization_13C

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Aug 22 2019**
Pulse Sequence: **PRESAT**

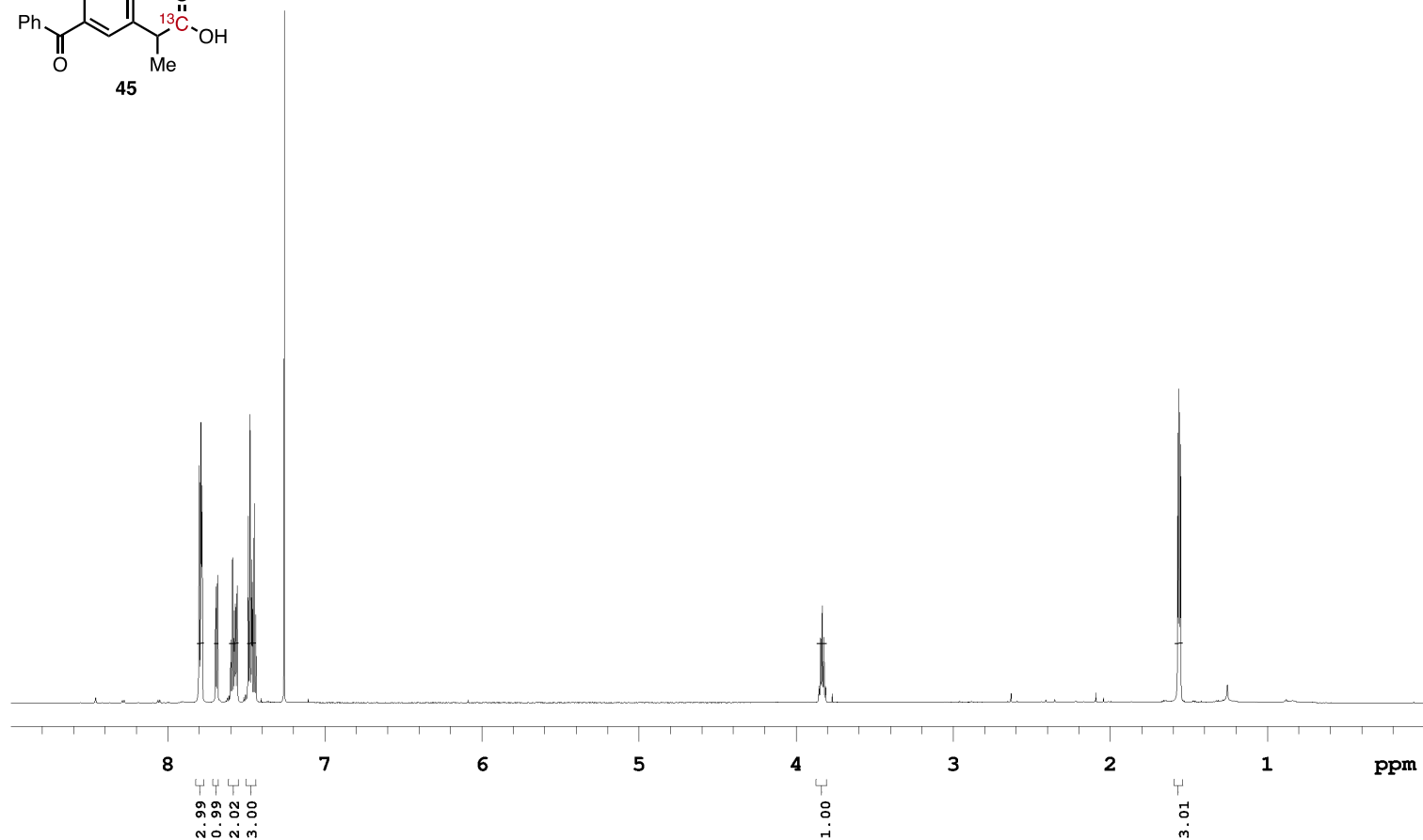
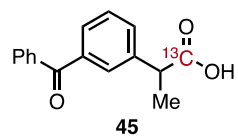
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.24**

Relaxation Delay(s): **0.1**
Completed Scans **8**

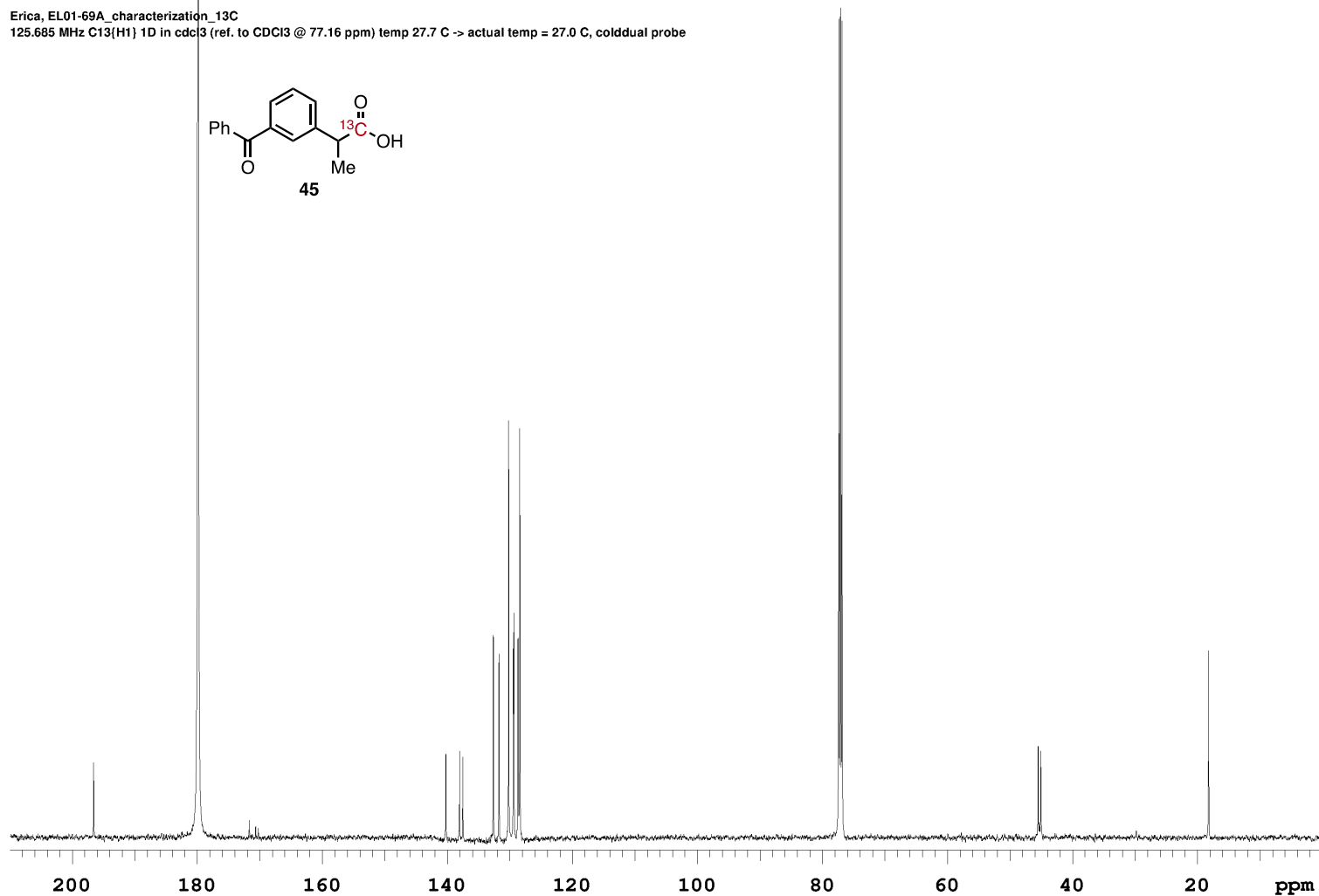
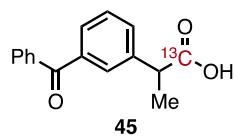
Erica, EL01-69_1H

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe



Erica, EL01-69A_characterization_13C

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Jul 18 2019**
Pulse Sequence: **PRESAT**

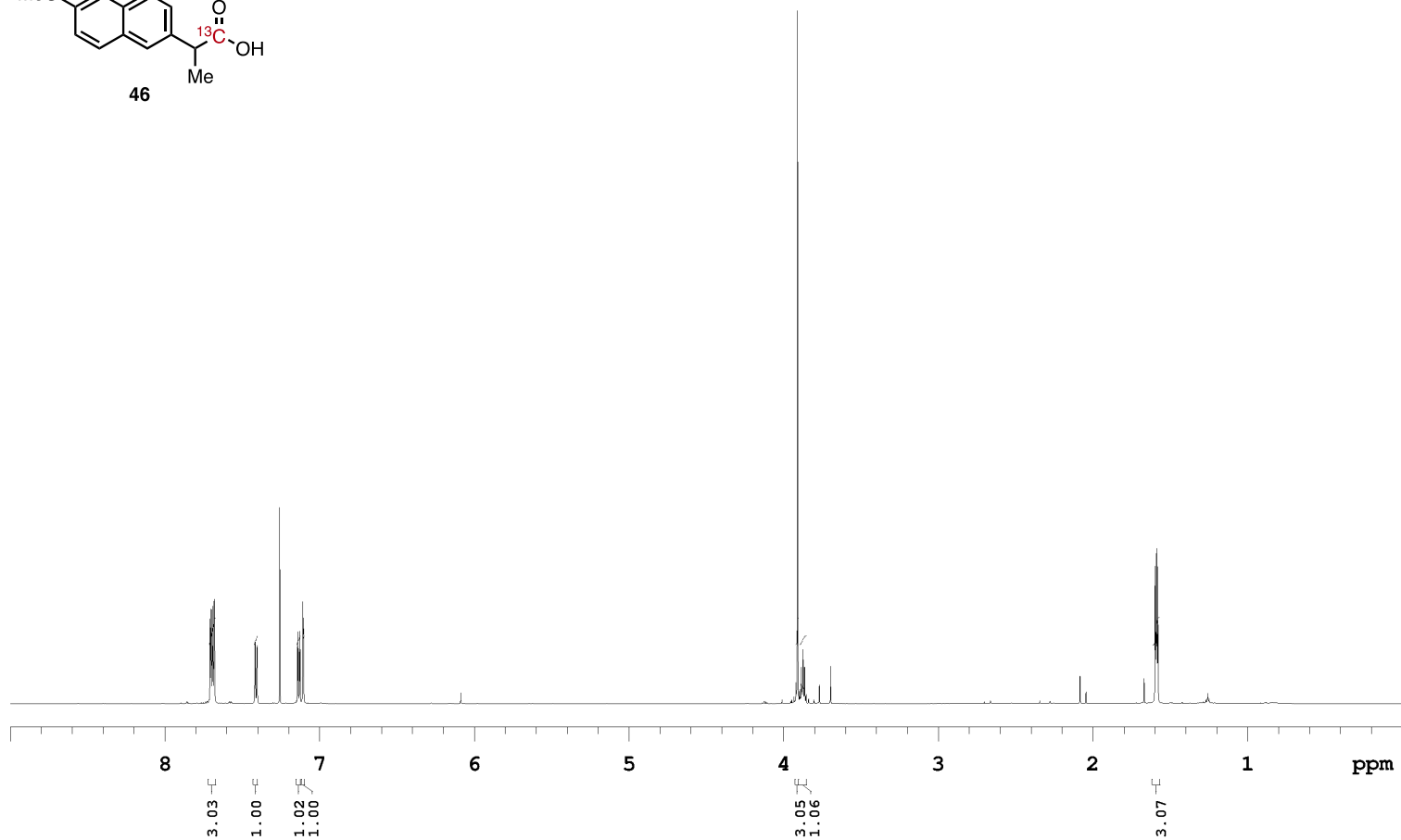
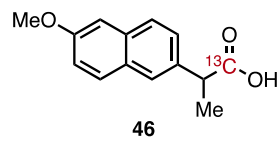
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.24**

Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL01-68_characterization

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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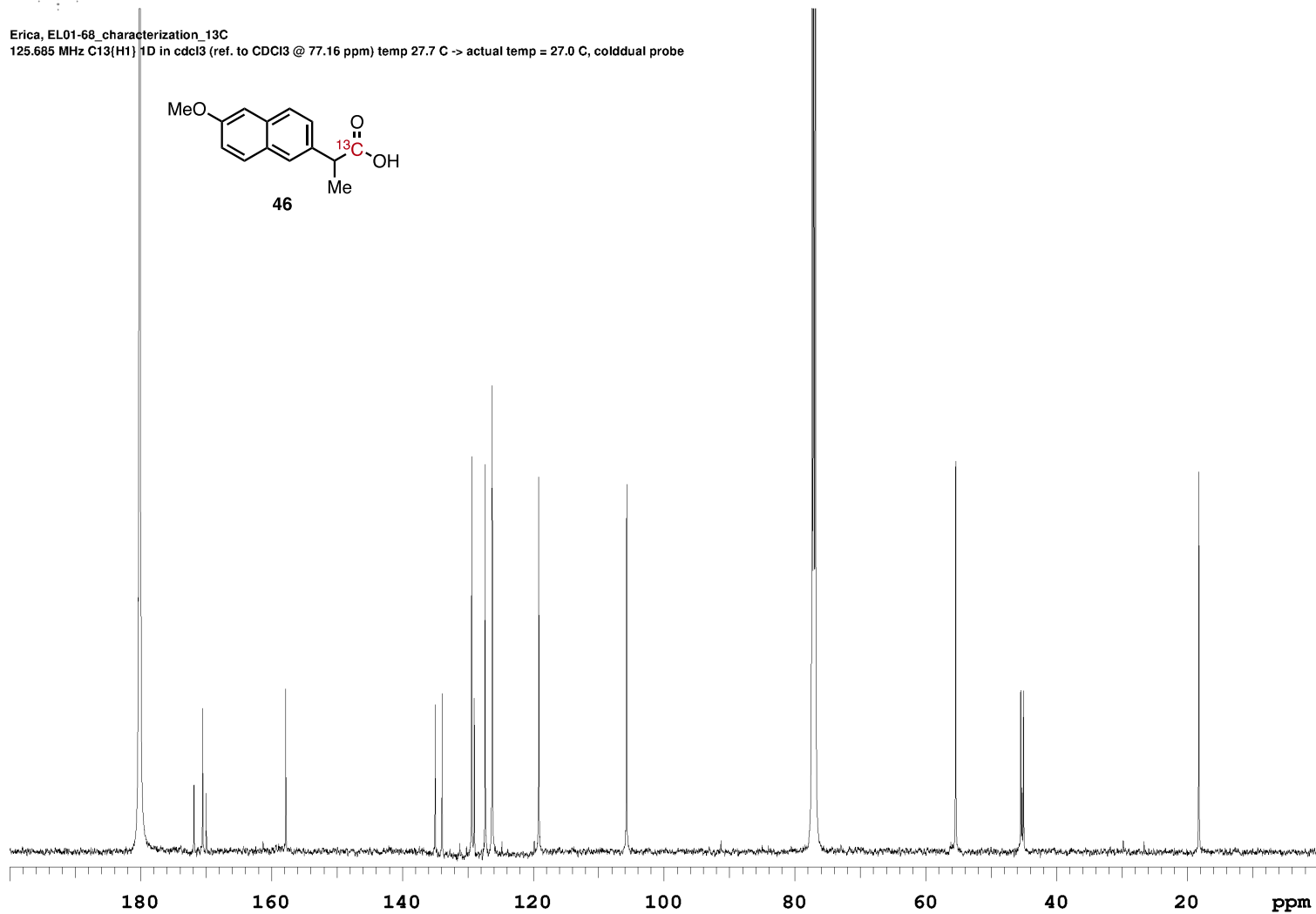
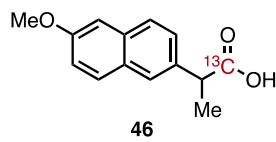
Recorded on: **u500, Sep 7 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.77**

Relaxation Delay(s): **1**
Completed Scans: **712**

Erica, EL01-68_characterization_13C
125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Aug 22 2019**
Pulse Sequence: **PRESAT**

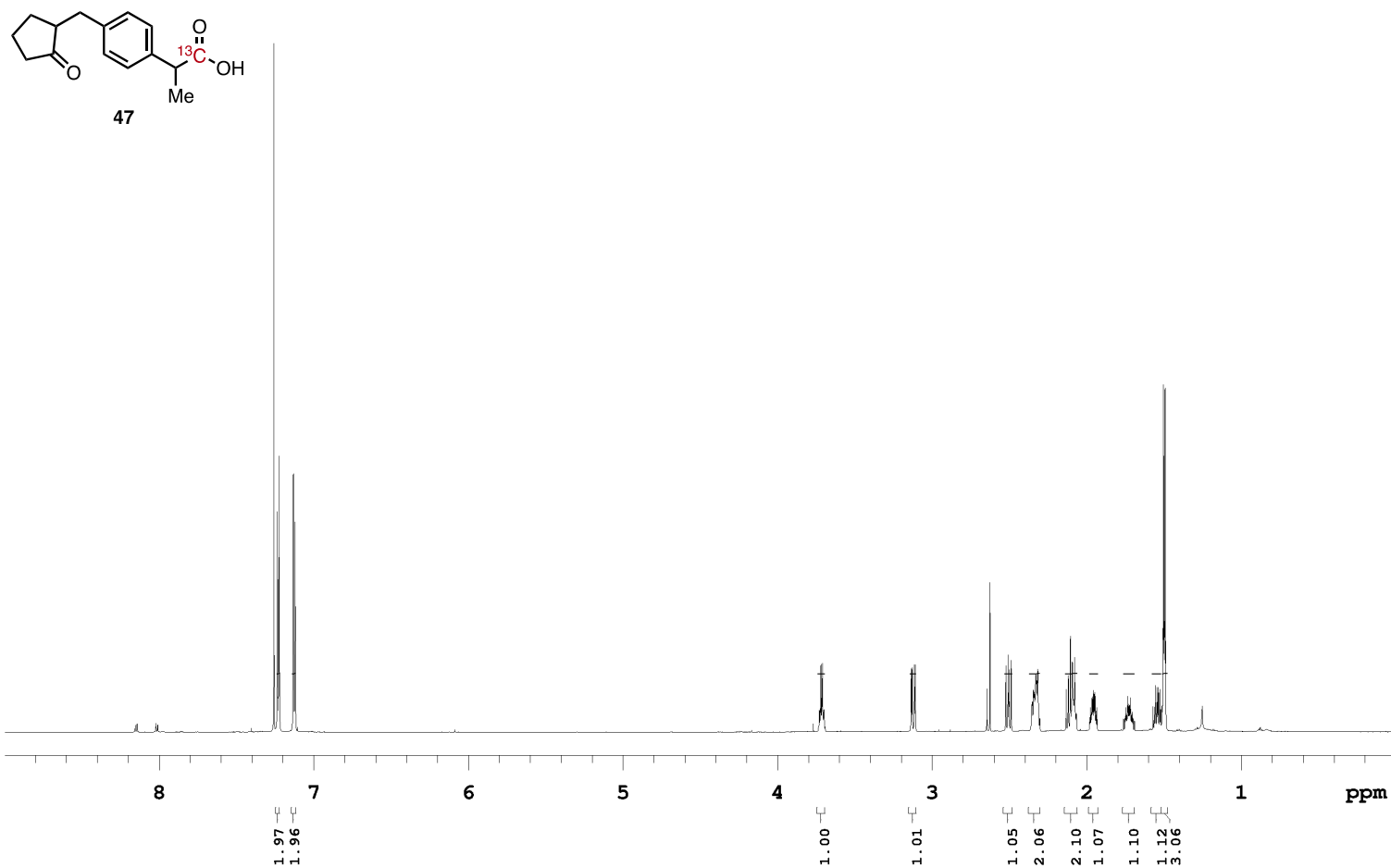
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.22**

Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL01-90_1H

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **u500, Aug 23 2019**
Pulse Sequence: **s2pul**

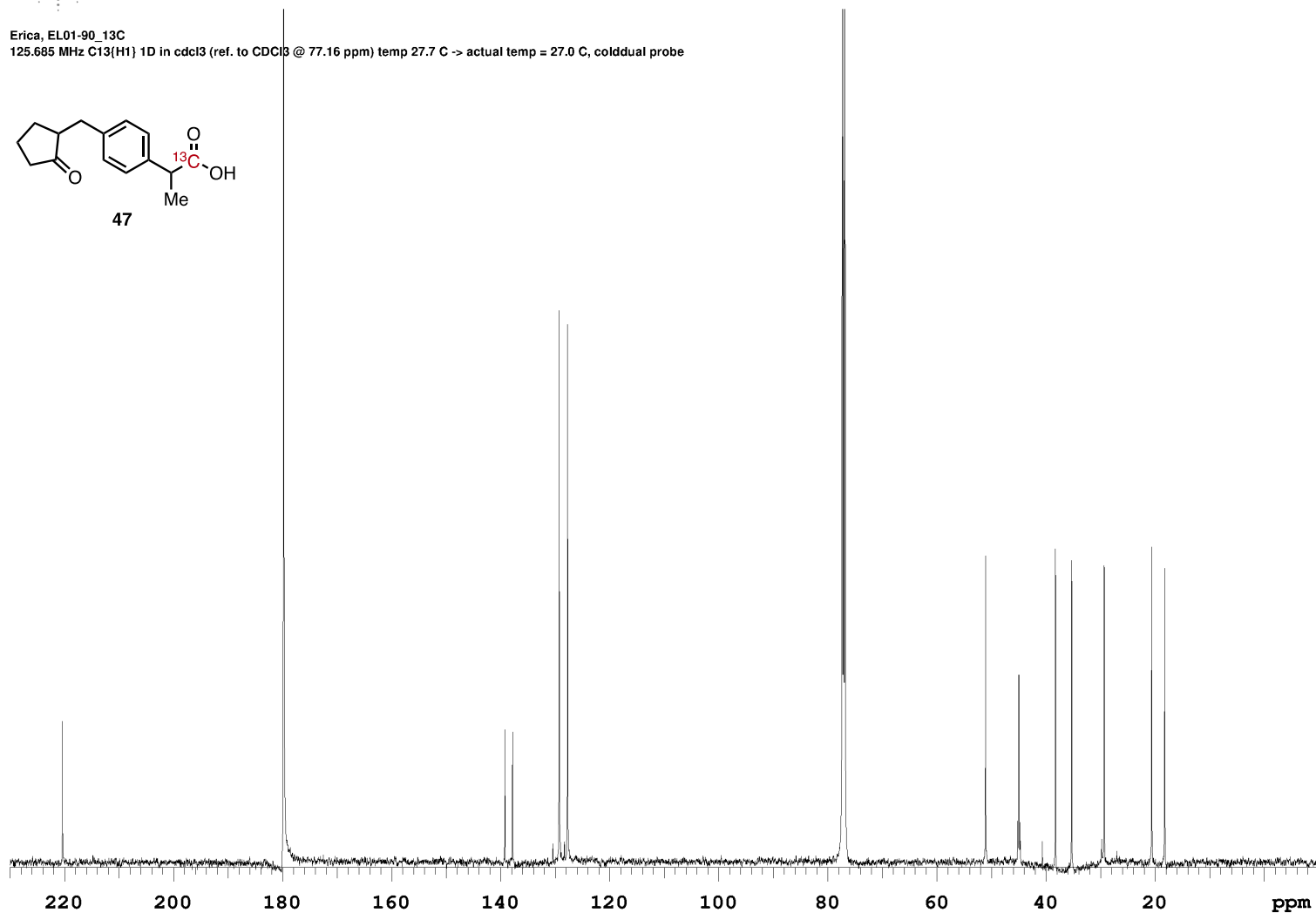
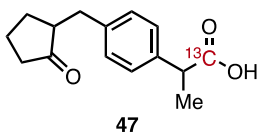
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **125.69**

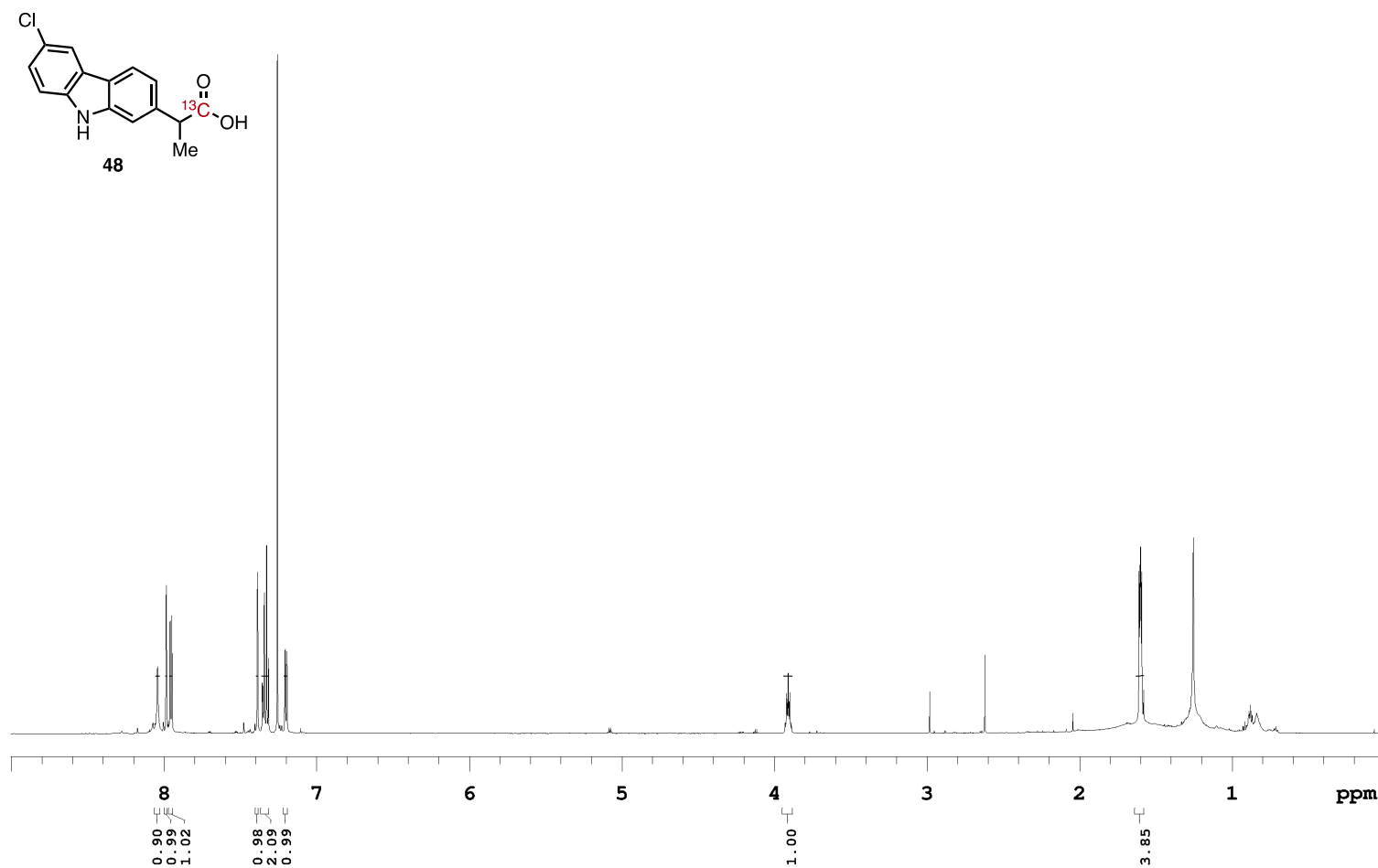
Relaxation Delay(s): **1**
Completed Scans **264**

Erica, EL01-90_13C

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe



Erica, EL02-05_1H
699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **u500, Aug 24 2019**
Pulse Sequence: **s2pul**

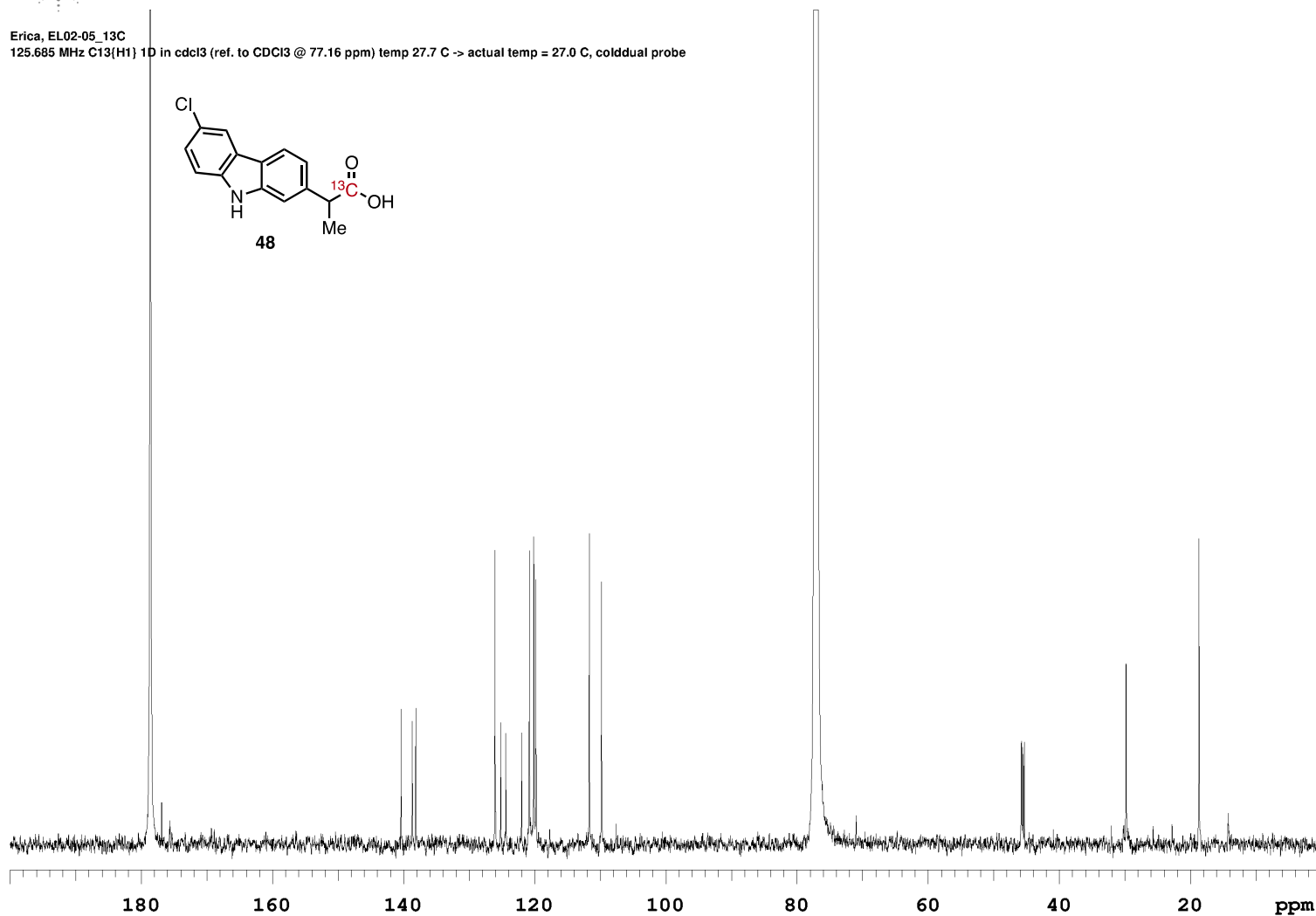
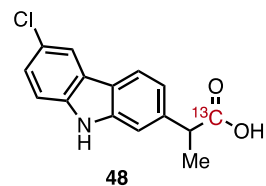
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.77**

Relaxation Delay(s): **1**
Completed Scans: **2000**

Erica, EL02-05_13C

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, coldddual probe





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Recorded on: **v700, Jul 22 2019**
Pulse Sequence: **PRESAT**

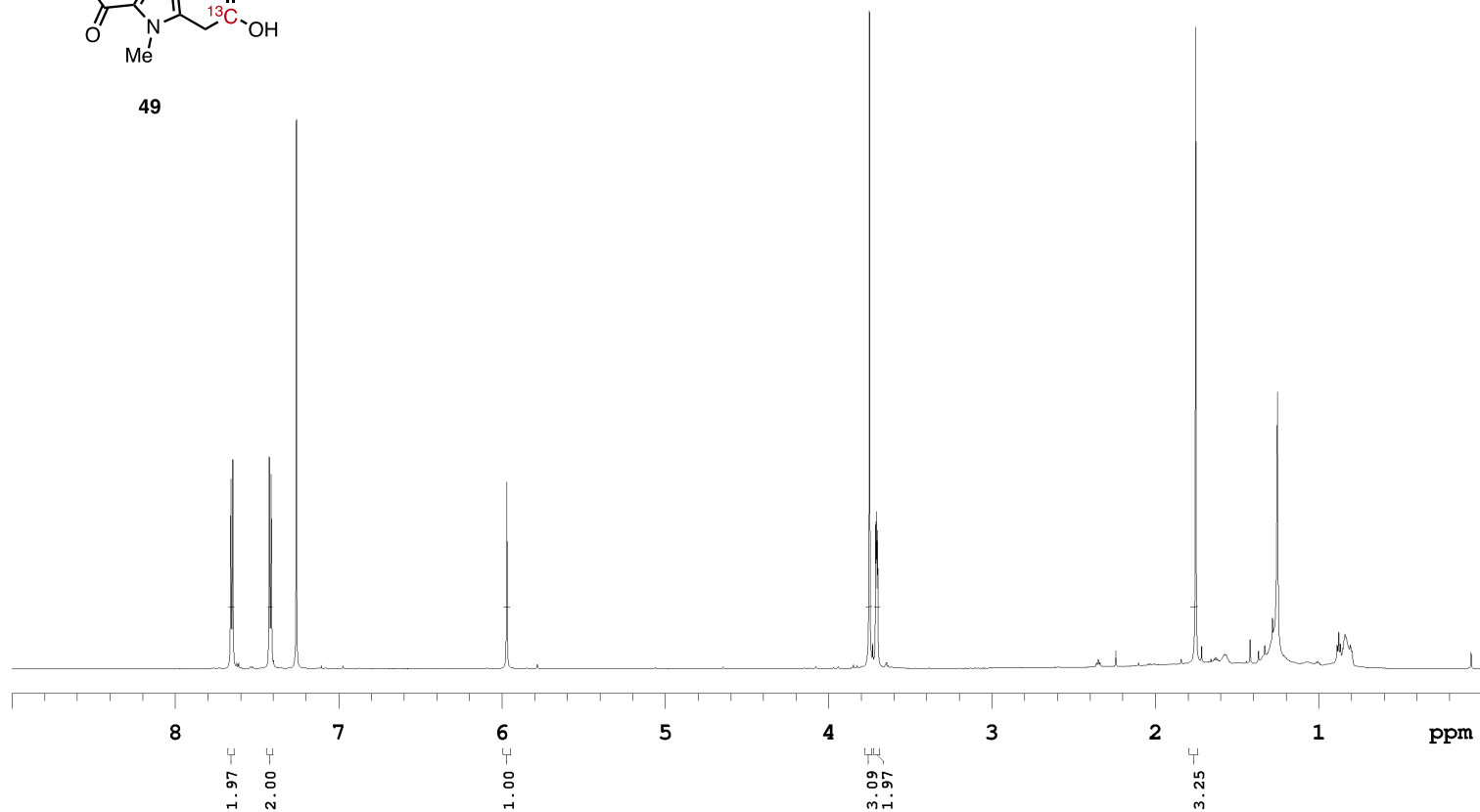
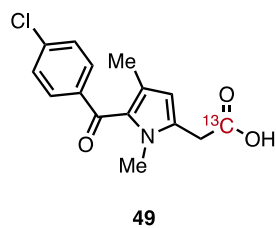
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.24**

Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL01-70_characterization

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **u500, Jul 23 2019**
Pulse Sequence: **s2pul**

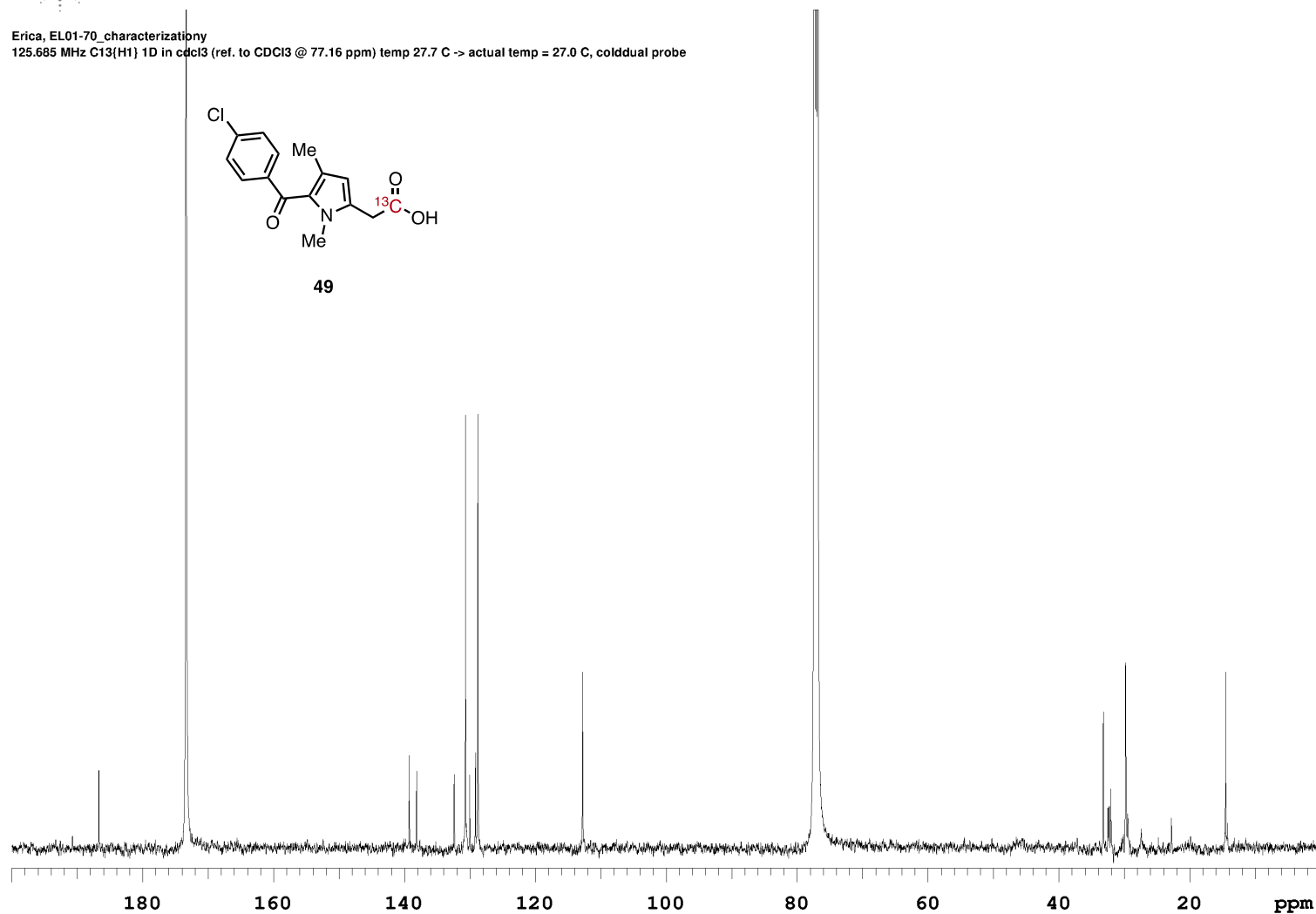
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.77**

Relaxation Delay(s): **1**
Completed Scans: **2000**

Erica, EL01-70_characterization

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Aug 22 2019**
Pulse Sequence: **PRESAT**

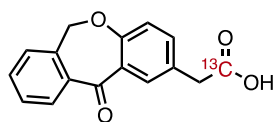
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.22**

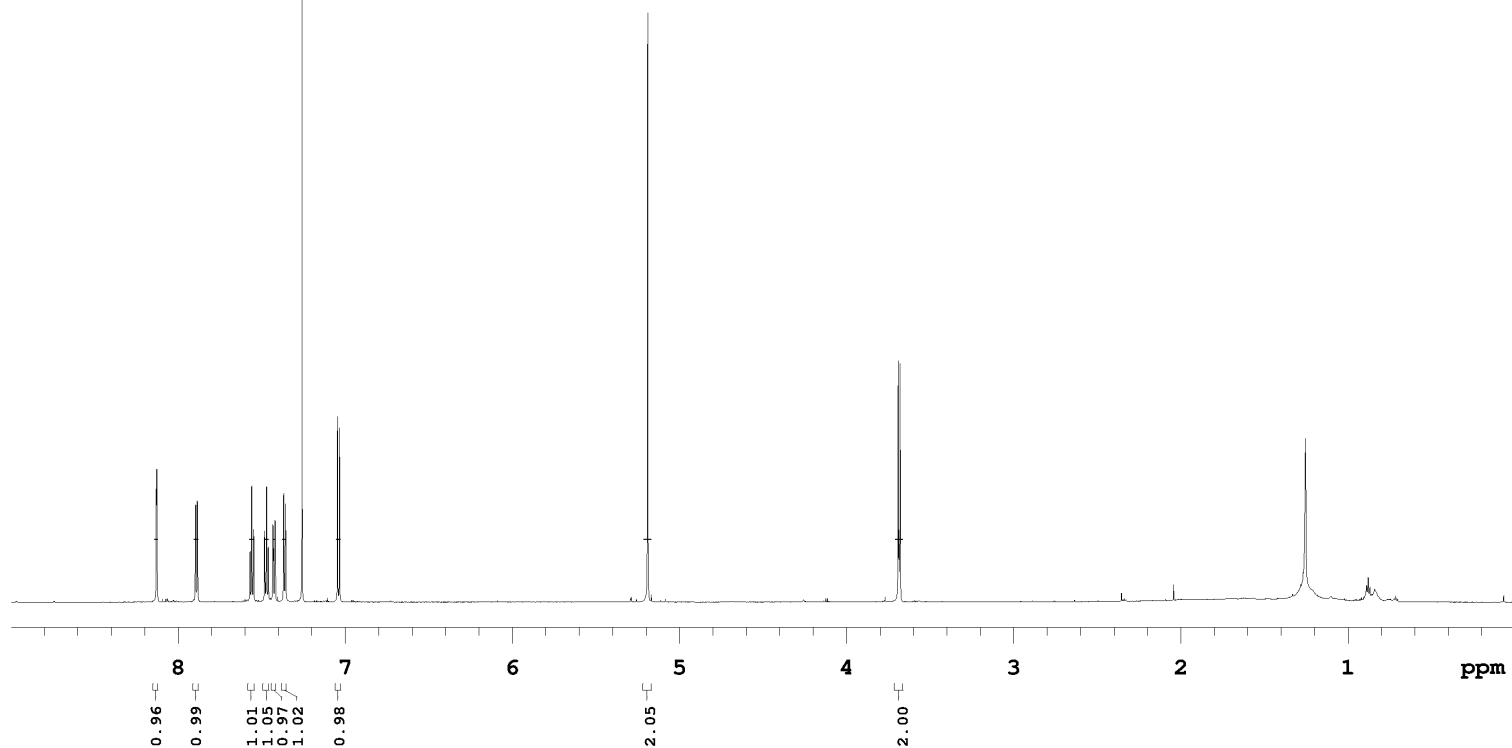
Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL02-01_1H

699.762 MHz ¹H 1D in cdcl₃ (ref. to CDCl₃ @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe



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Recorded on: **u500, Aug 23 2019**
Pulse Sequence: **s2pul**

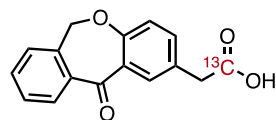
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.68**

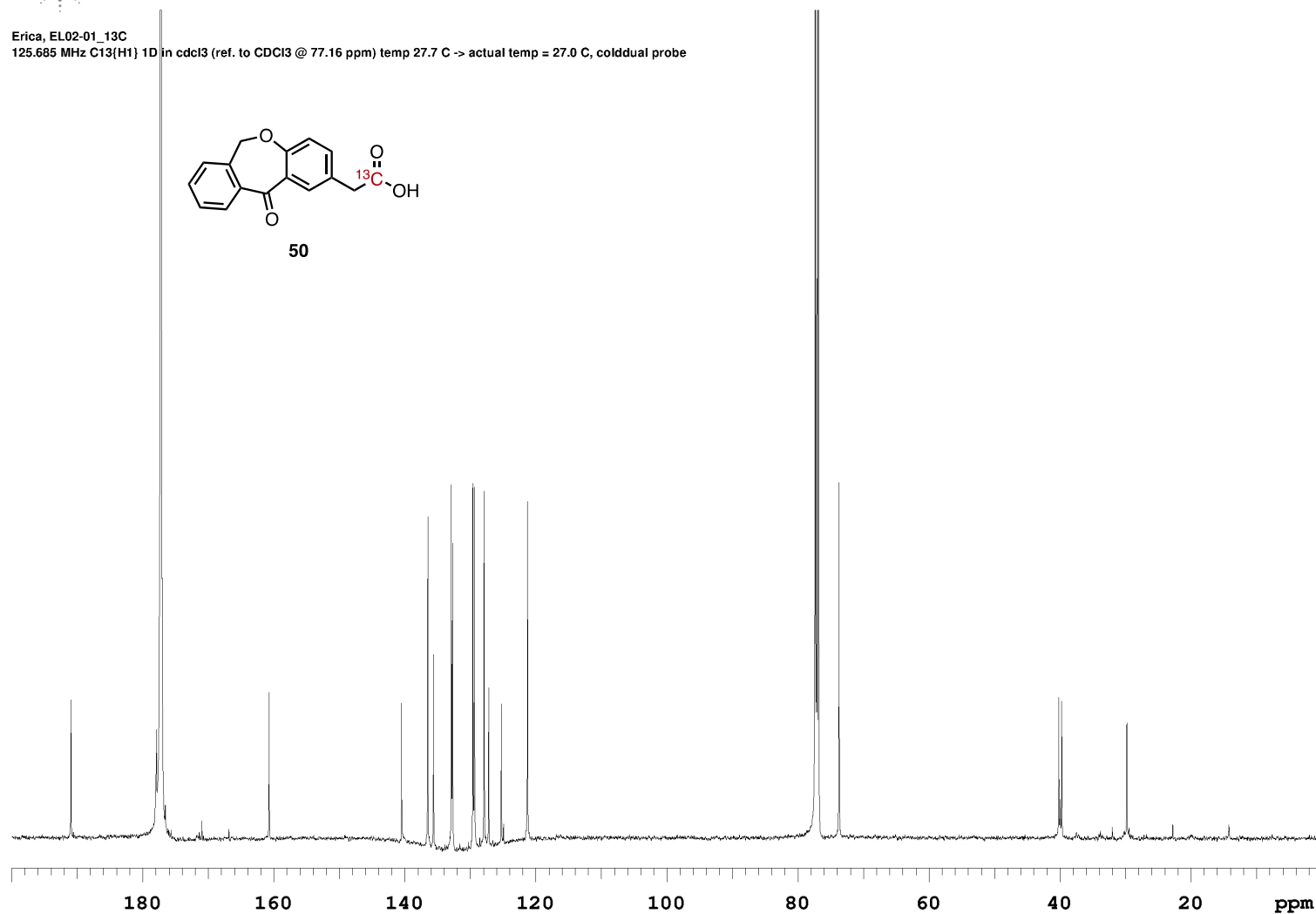
Relaxation Delay(s): **1**
Completed Scans **428**

Erica, EL02-01_13C

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe



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Recorded on: **v700, Aug 23 2019**
Pulse Sequence: **PRESAT**

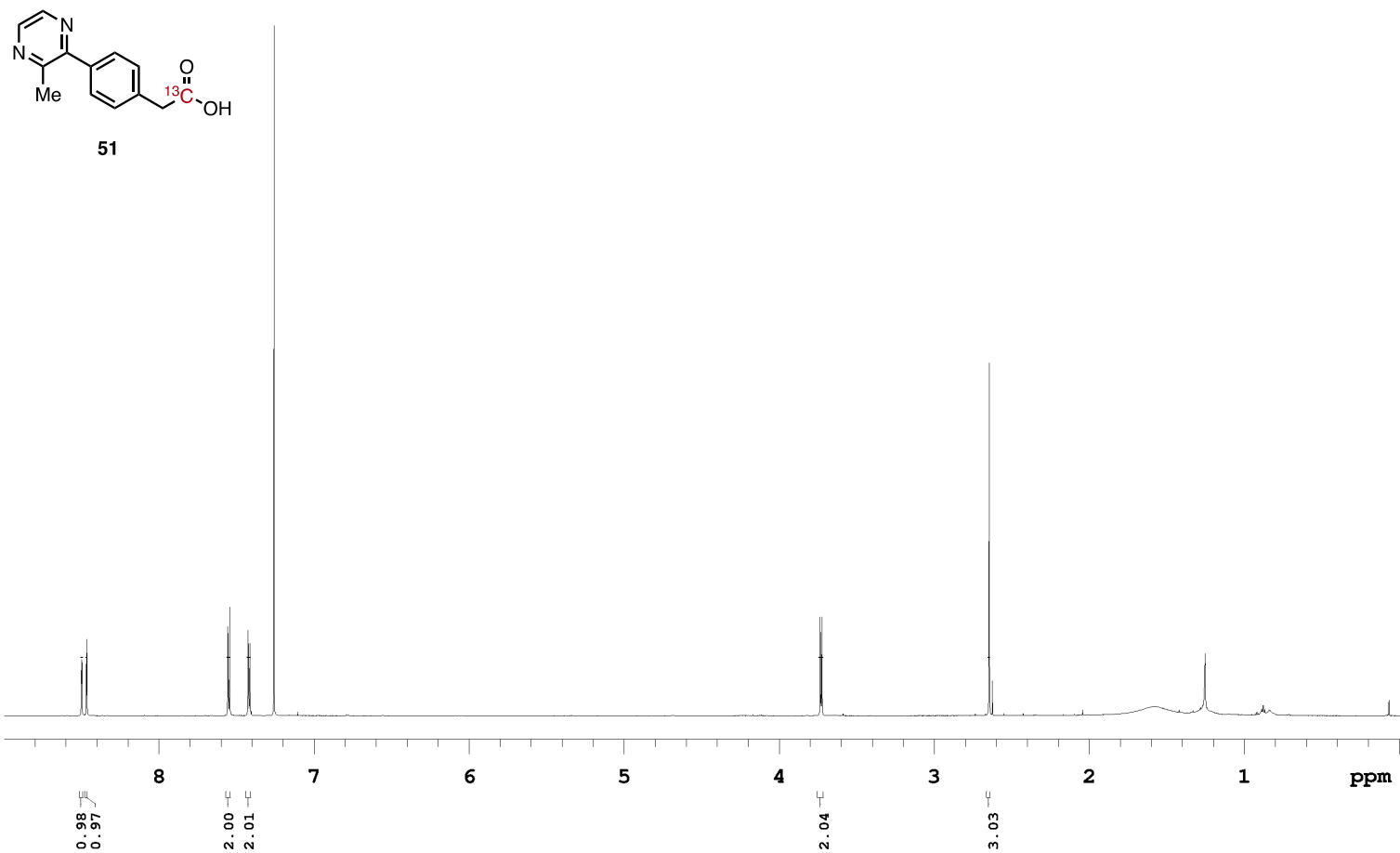
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.24**

Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL02-04_characterization

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **u500, Aug 24 2019**
Pulse Sequence: **s2pul**

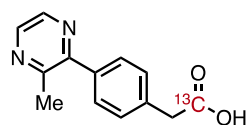
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.77**

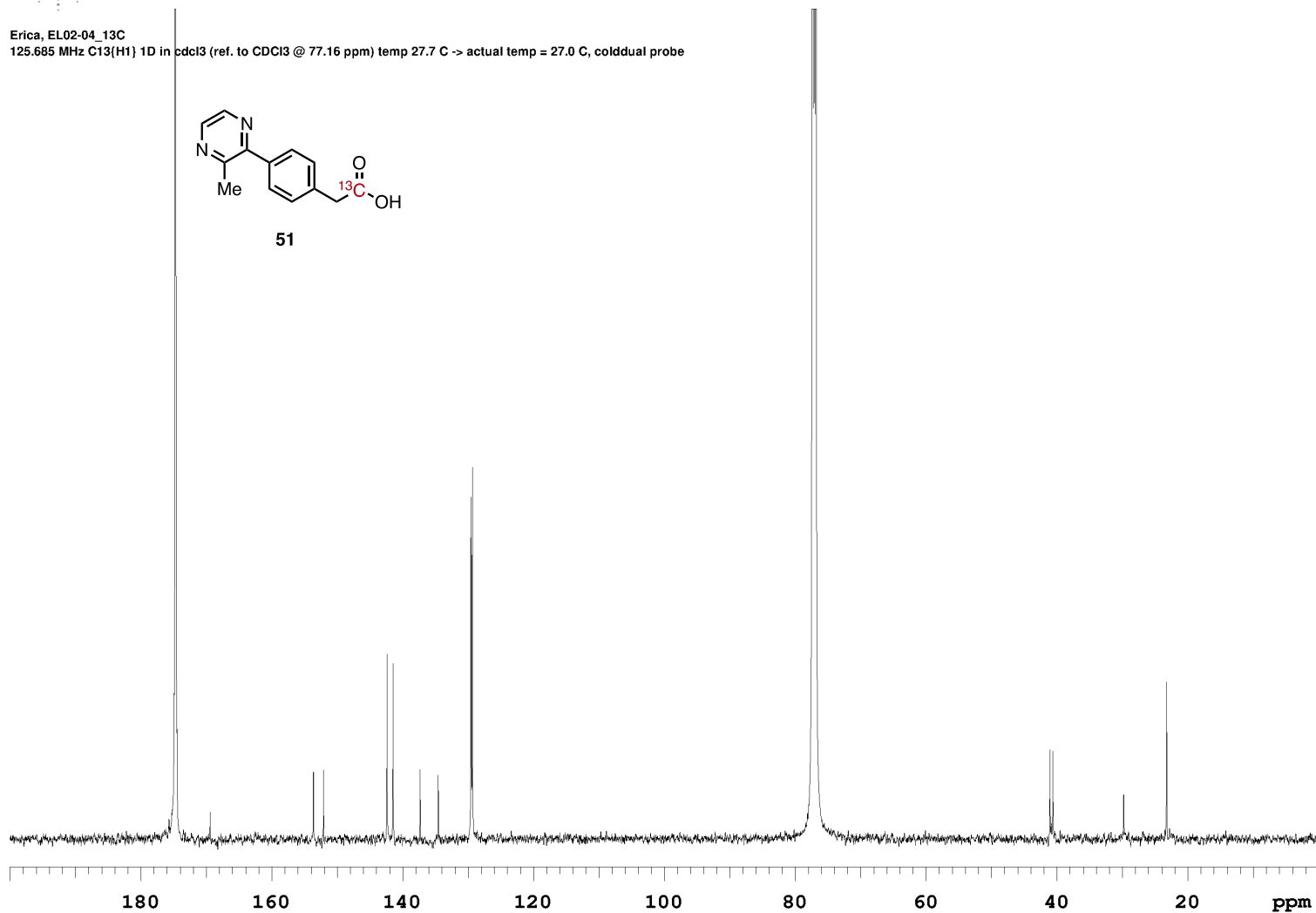
Relaxation Delay(s): **1**
Completed Scans **2000**

Erica, EL02-04_13C

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe



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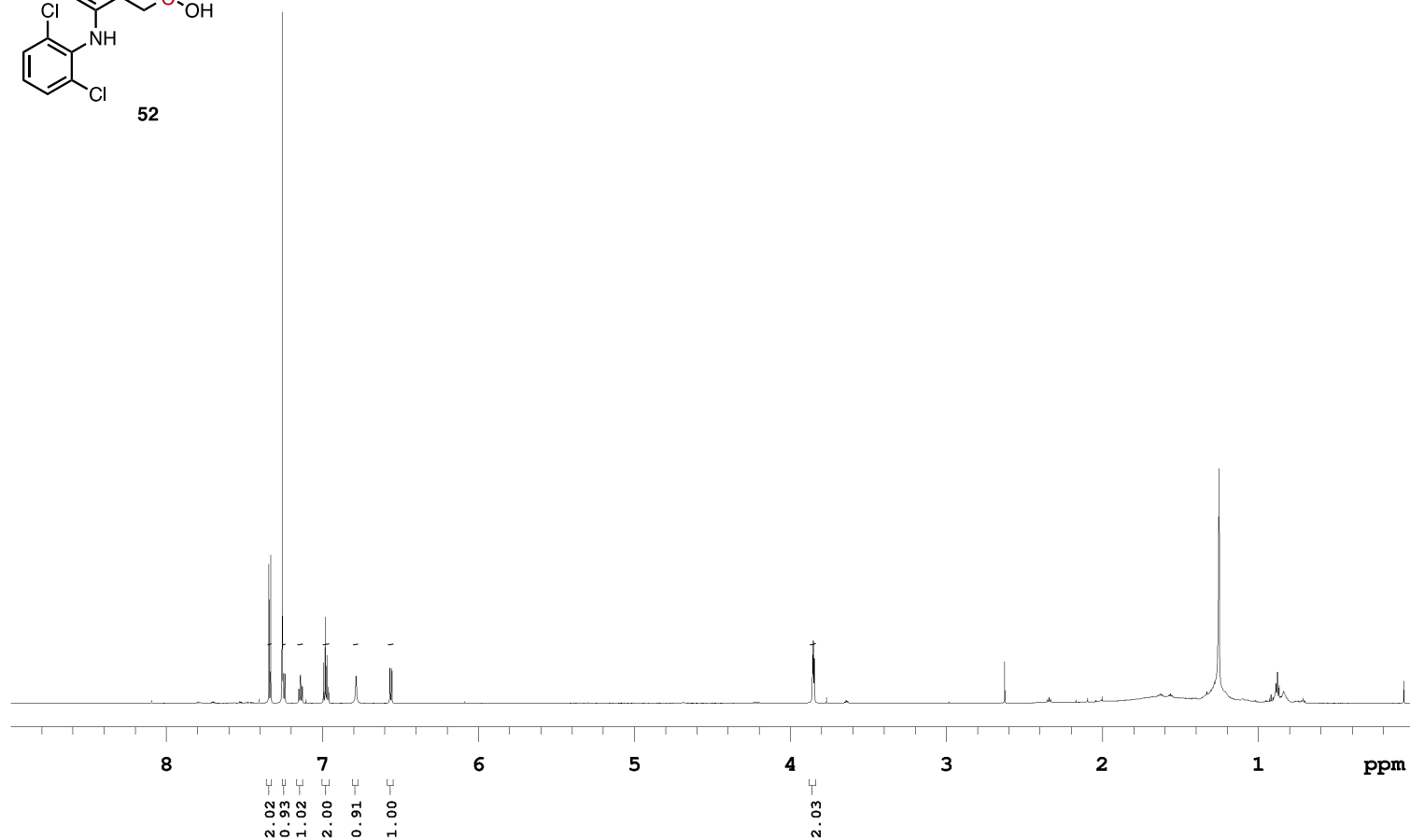
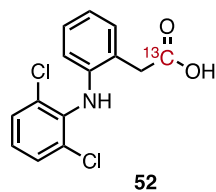
Recorded on: **v700, Sep 5 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.22**

Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL02-03_characterization_1H
699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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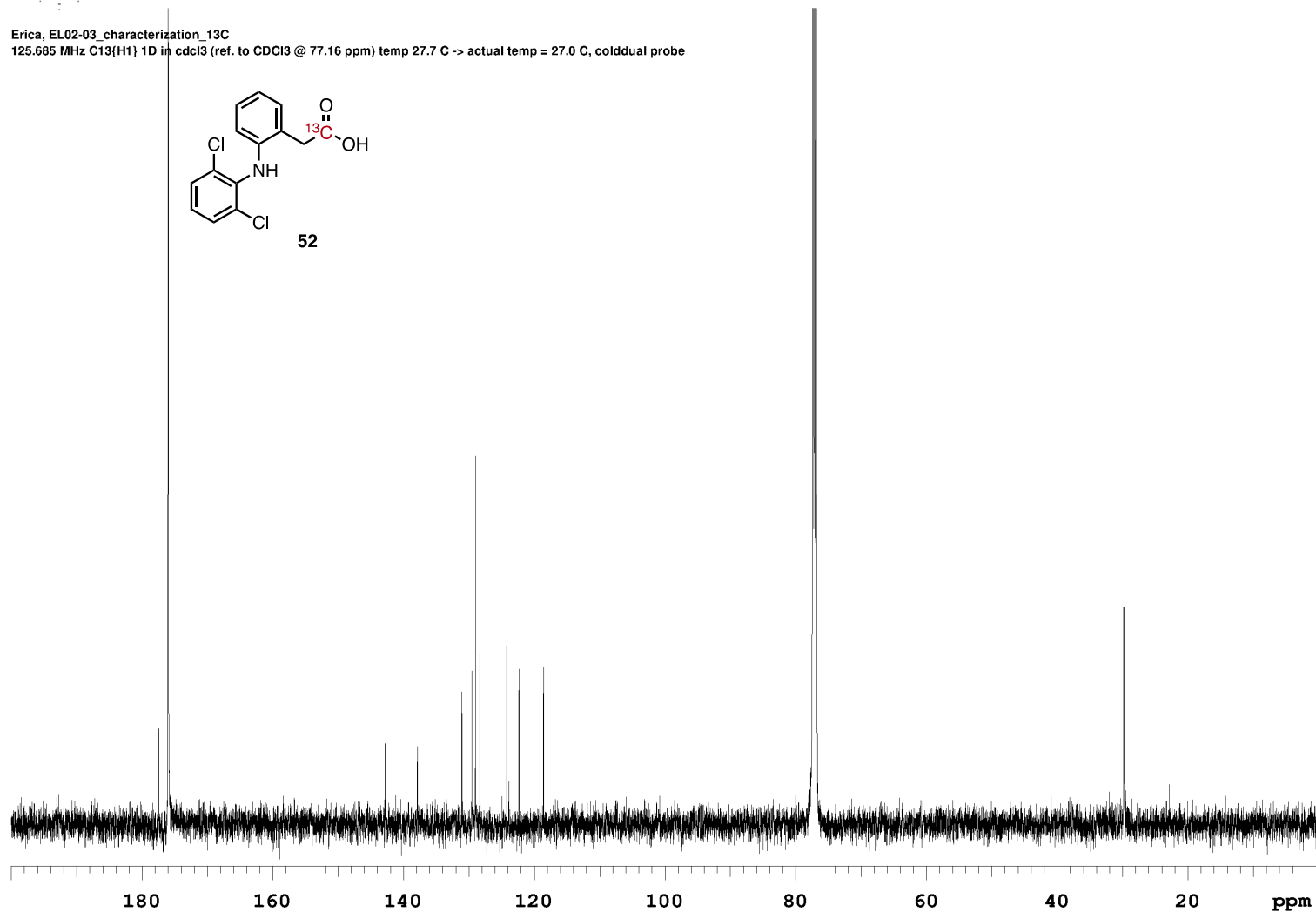
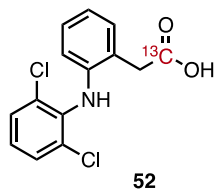
Recorded on: **u500, Sep 6 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **104.77**

Relaxation Delay(s): **1**
Completed Scans **416**

Erica, EL02-03_characterization_13C
125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Aug 23 2019**
Pulse Sequence: **PRESAT**

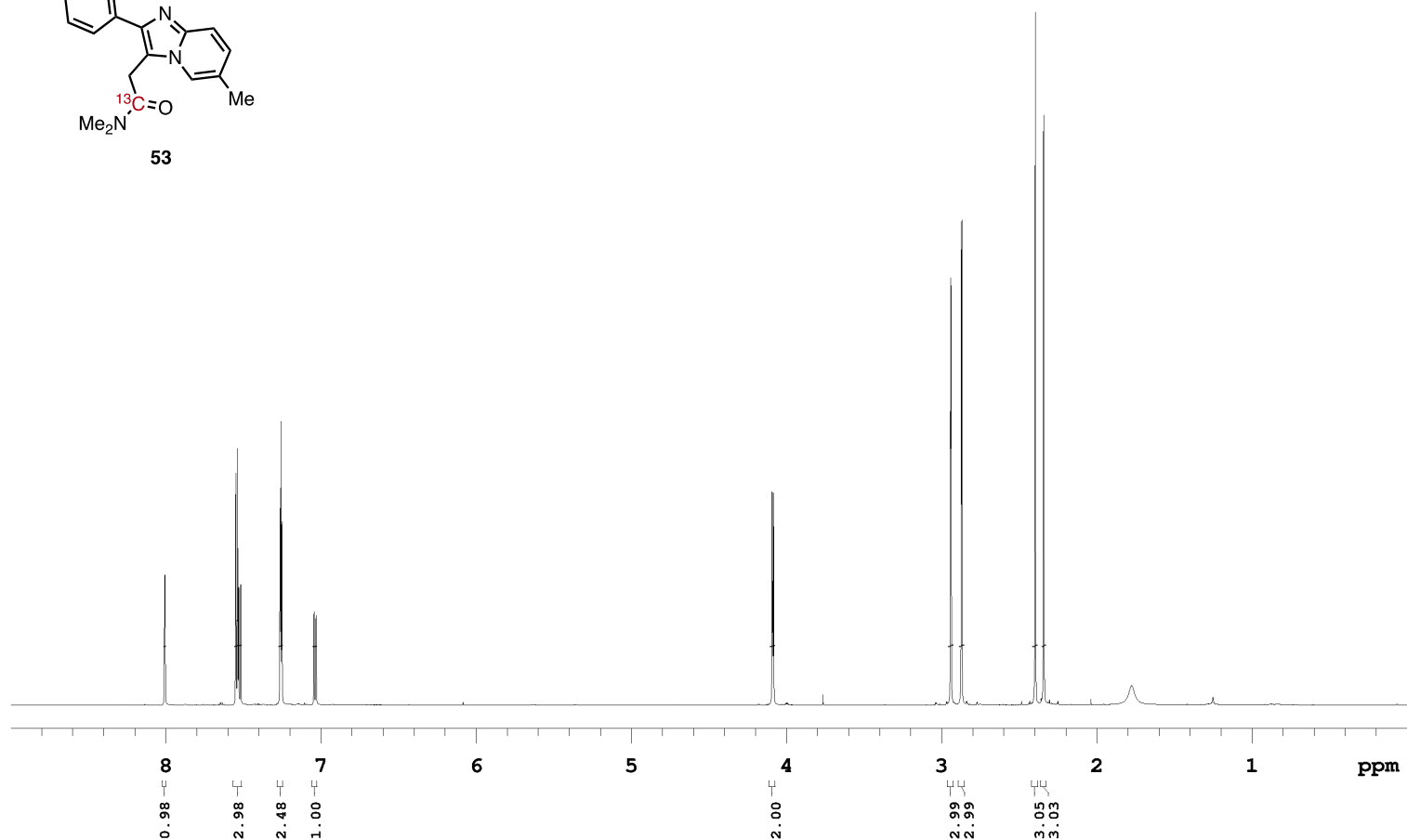
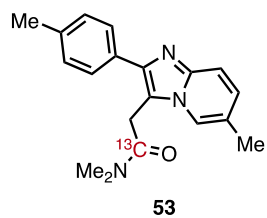
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.24**

Relaxation Delay(s): **0.1**
Completed Scans **8**

Erica, EL01-80_characterization

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





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Recorded on: **u500, Aug 24 2019**
Pulse Sequence: **s2pul**

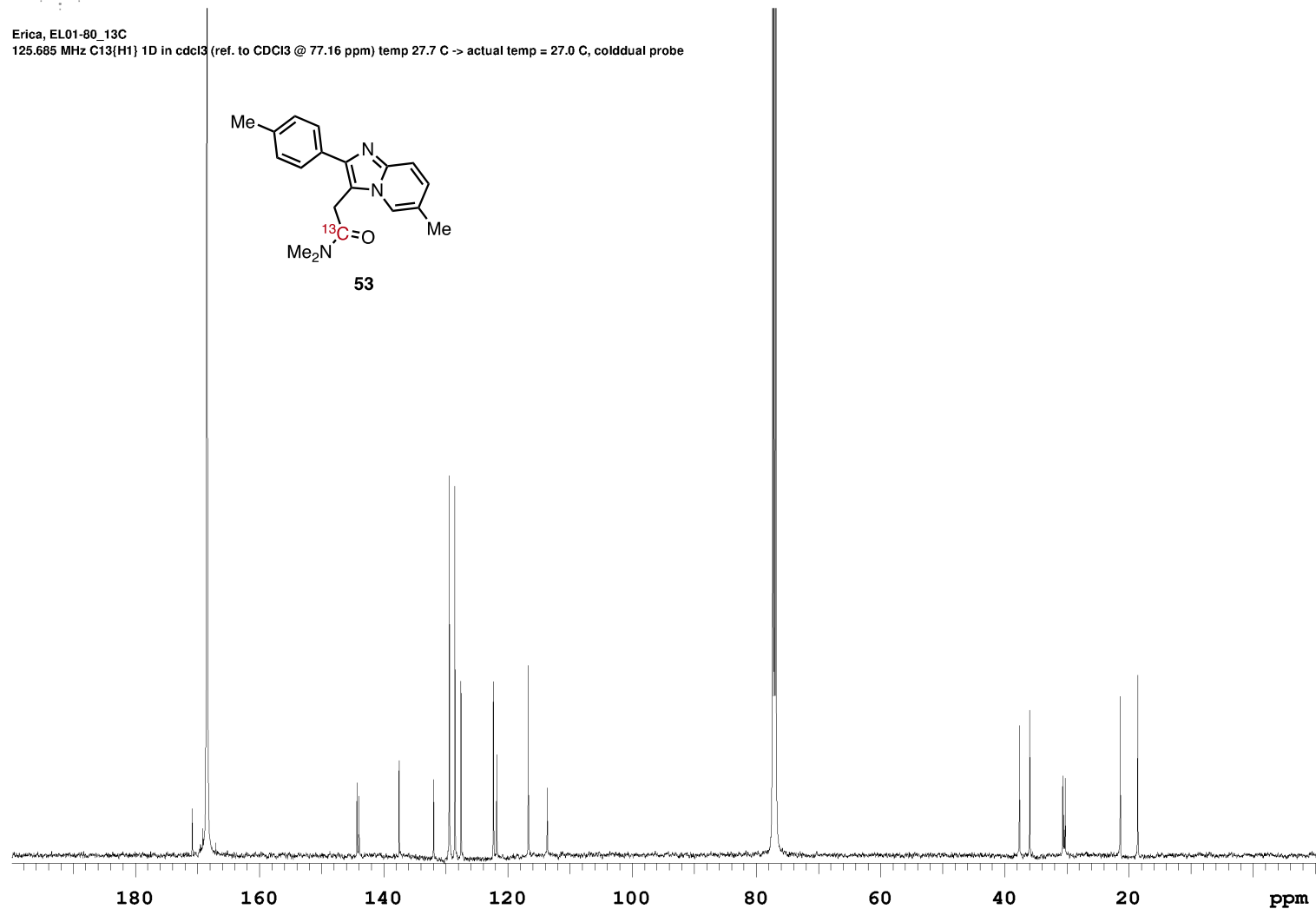
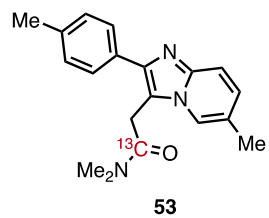
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **109.98**

Relaxation Delay(s): **1**
Completed Scans **416**

Erica, EL01-80_13C

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.16 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **u500, Jan 24 2020**
Pulse Sequence: **PRESAT**

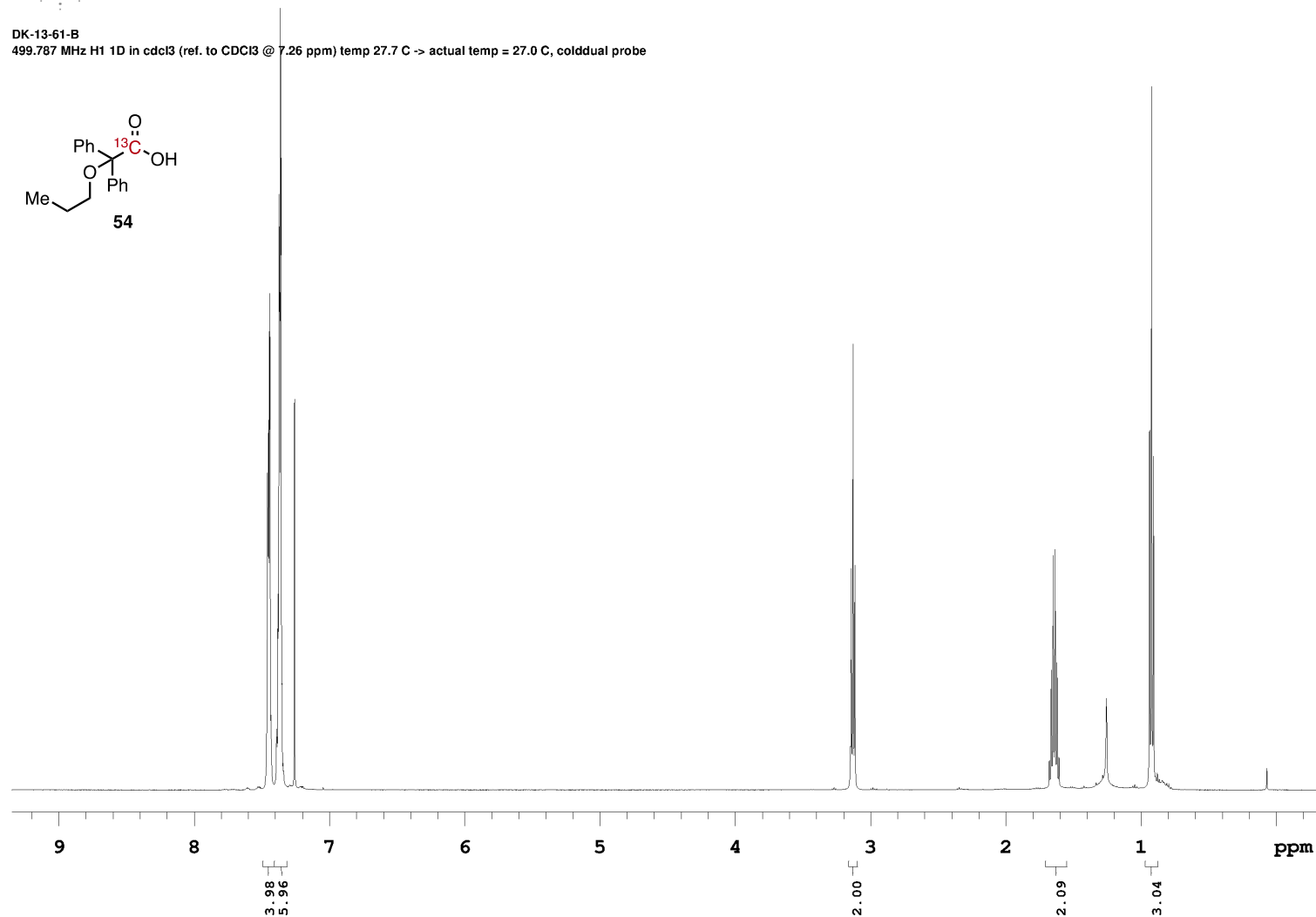
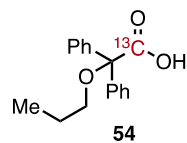
Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.15**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-13-61-B

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Agilent Technologies

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Recorded on: **u500, Jan 24 2020**
Pulse Sequence: **s2pul**

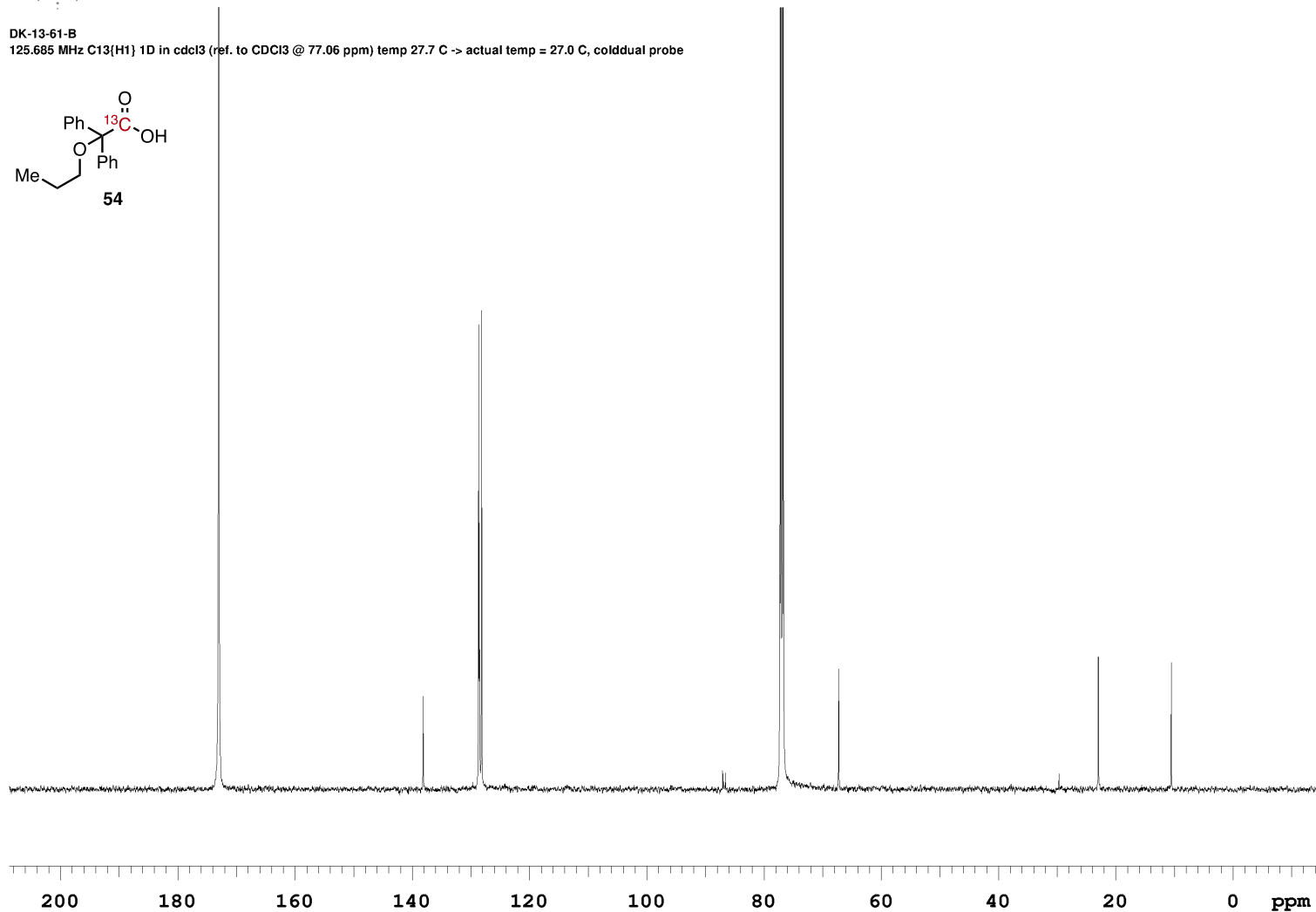
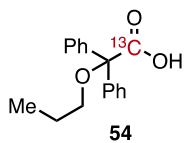
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **116.94**

Relaxation Delay(s): **1**
Completed Scans **220**

DK-13-61-B

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **u500, Dec 20 2019**
Pulse Sequence: **PRESAT**

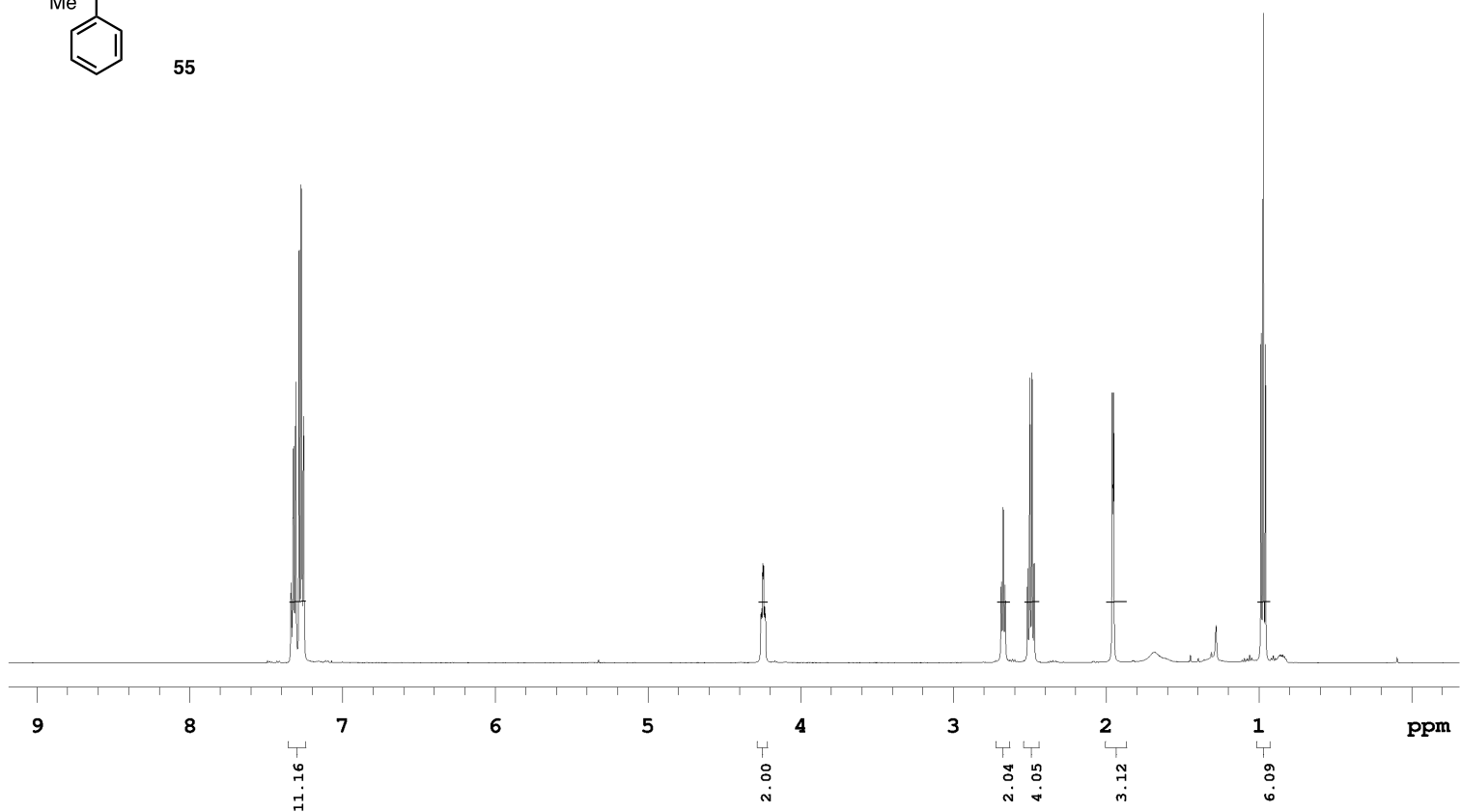
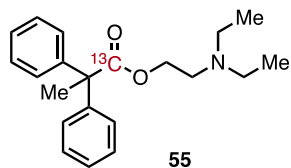
Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.77**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-12-189

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Dec 20 2019**
Pulse Sequence: **s2pul**

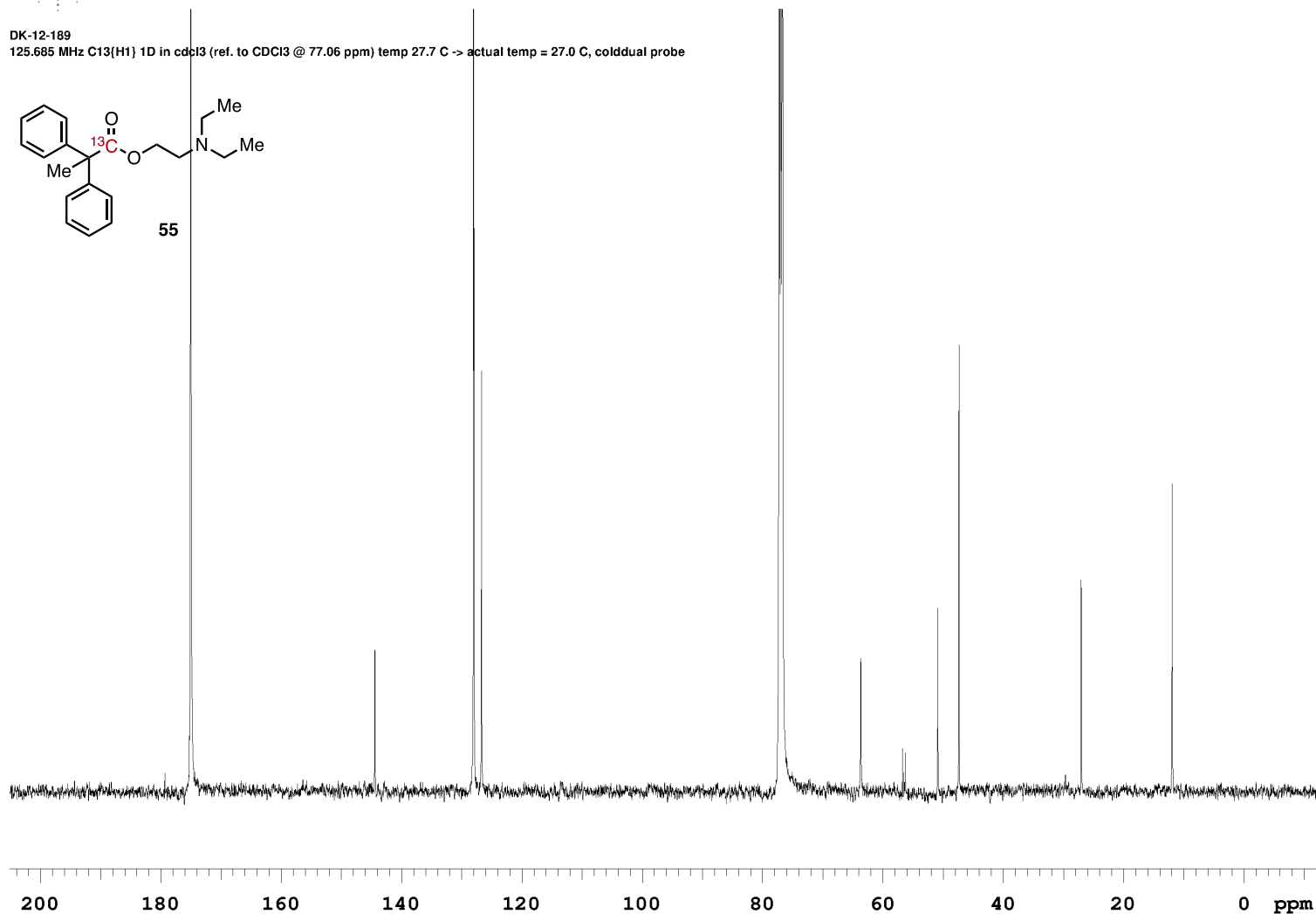
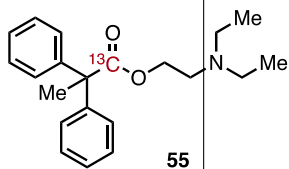
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **114.11**

Relaxation Delay(s): **1**
Completed Scans **460**

DK-12-189

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 24 2020**
Pulse Sequence: **PRESAT**

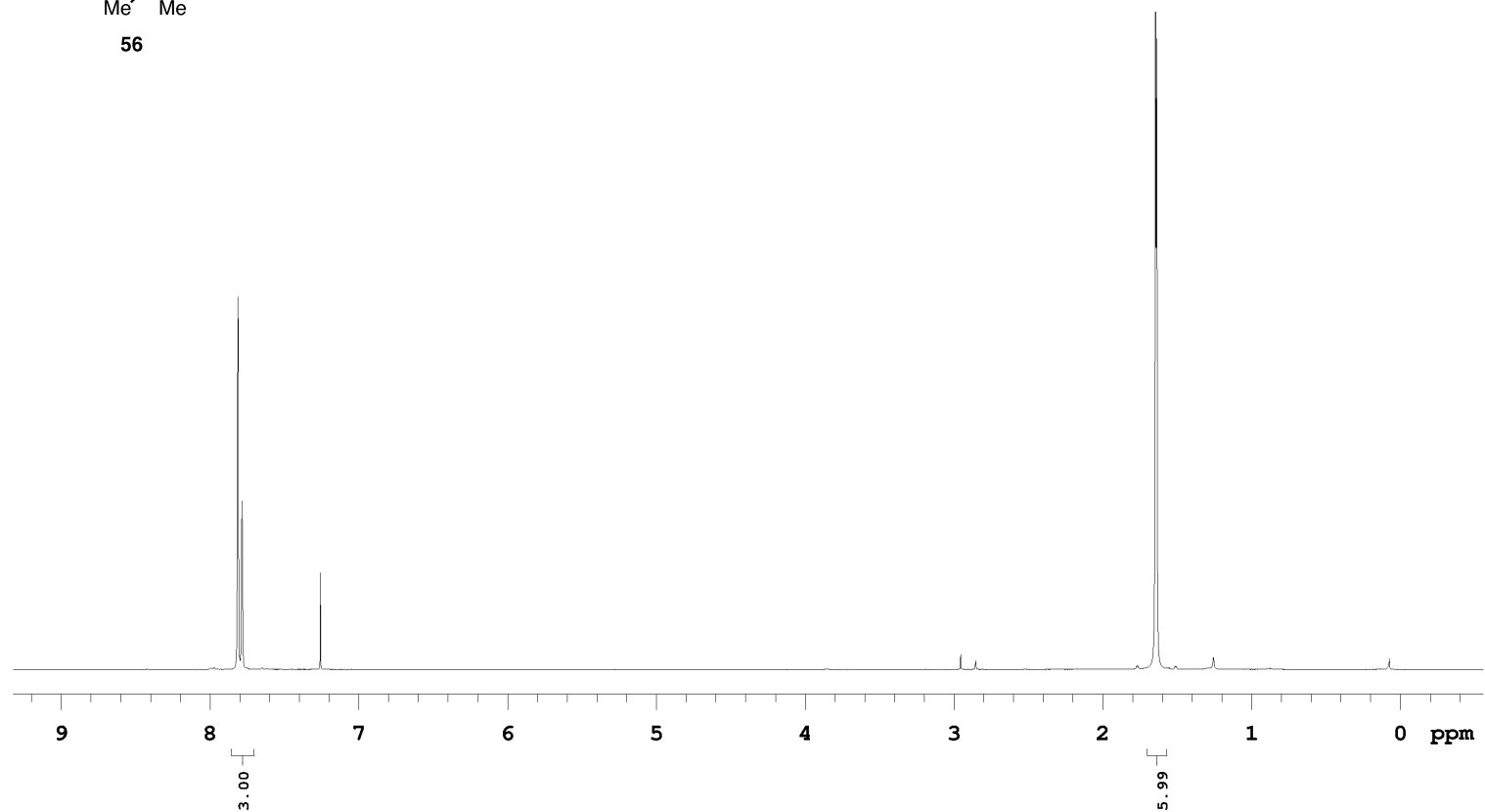
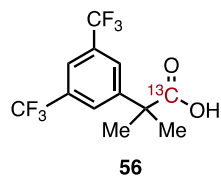
Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.57**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-13-61-A

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **u500, Jan 24 2020**
Pulse Sequence: **s2pul**

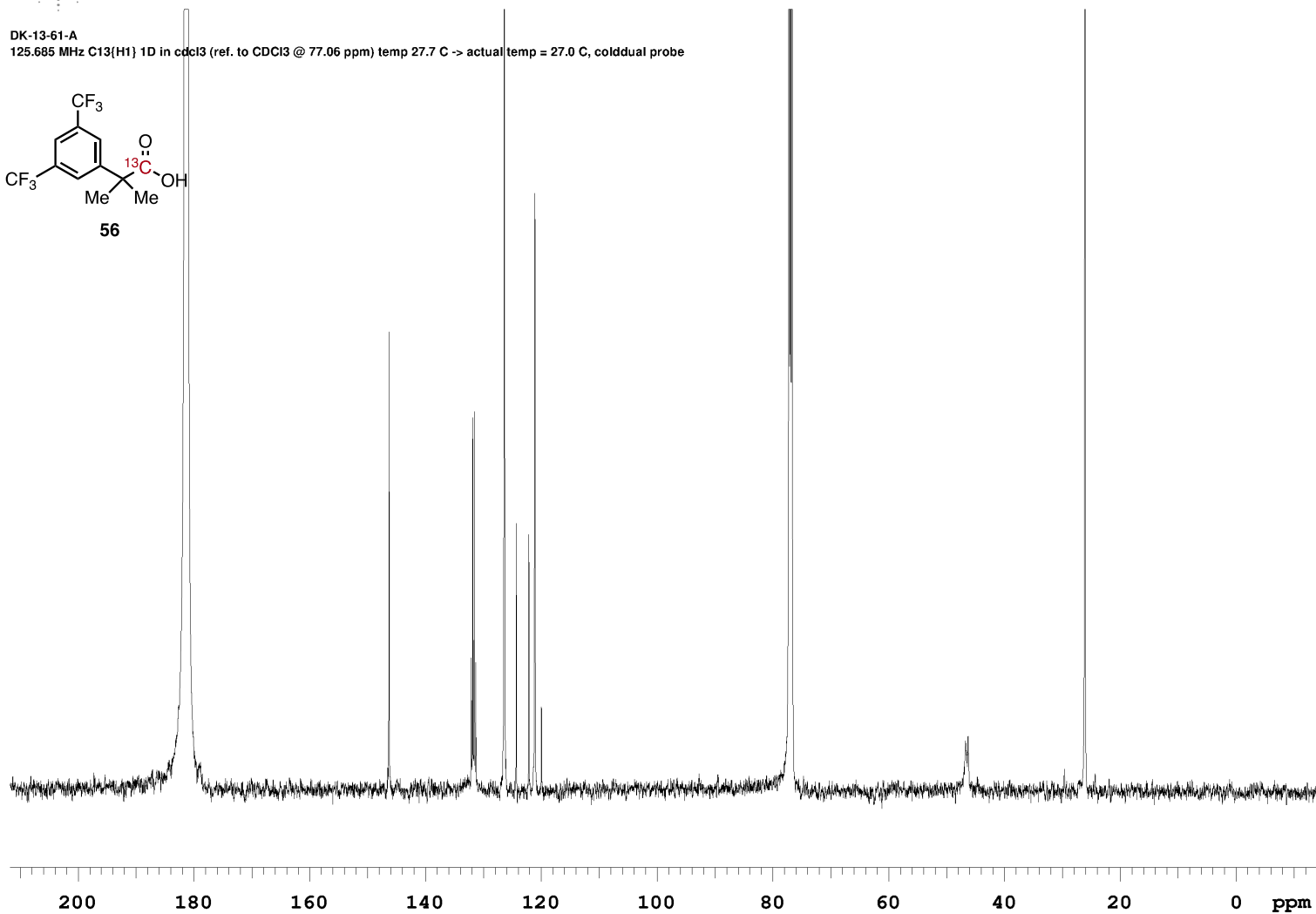
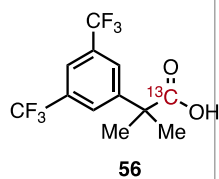
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **118.4**

Relaxation Delay(s): **1**
Completed Scans **212**

DK-13-61-A

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **v700, Jan 22 2020**
Pulse Sequence: **PRESAT**

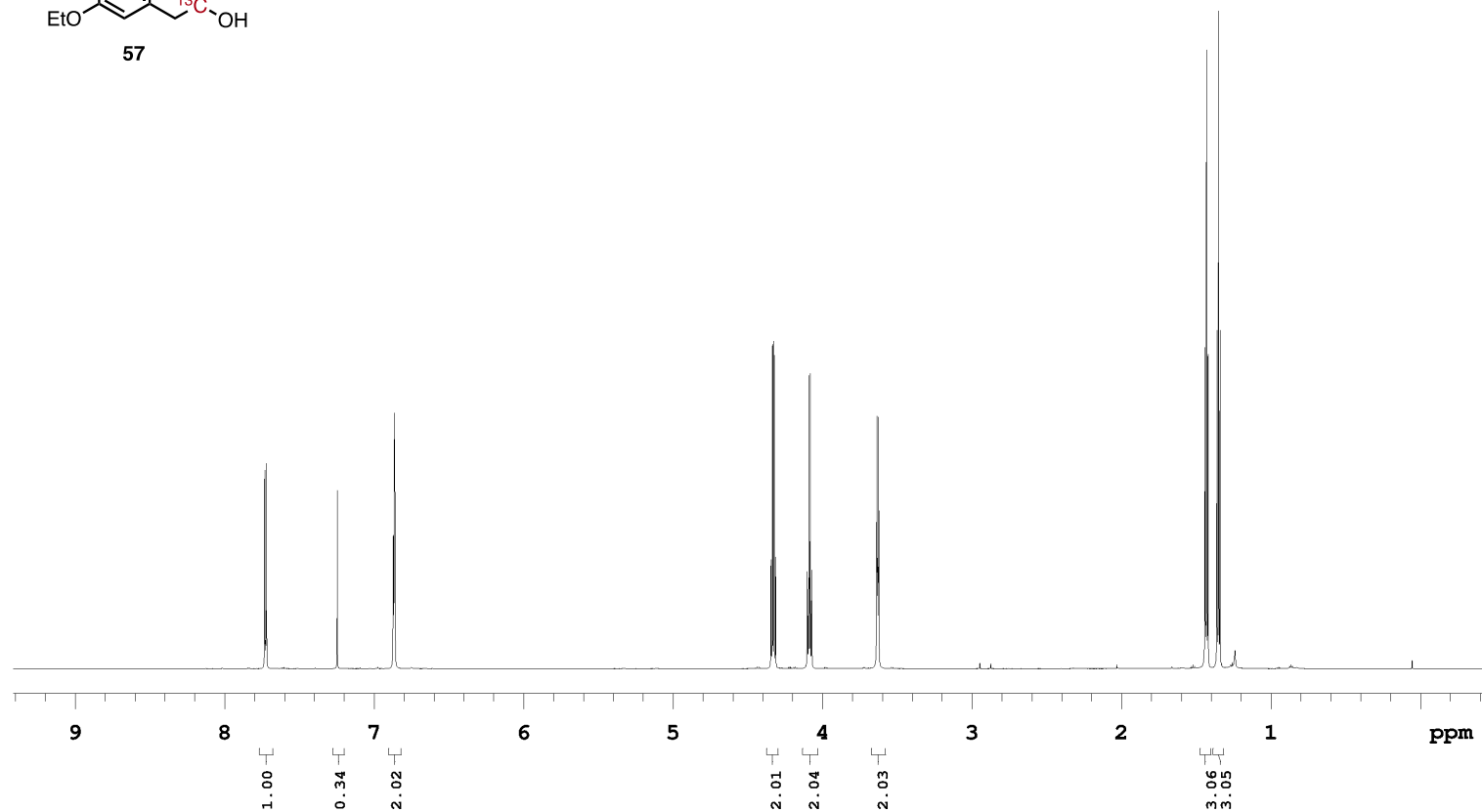
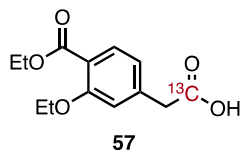
Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **28.65**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-13-53-B

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





Agilent Technologies

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Recorded on: **v700, Jan 22 2020**
Pulse Sequence: **s2pul**

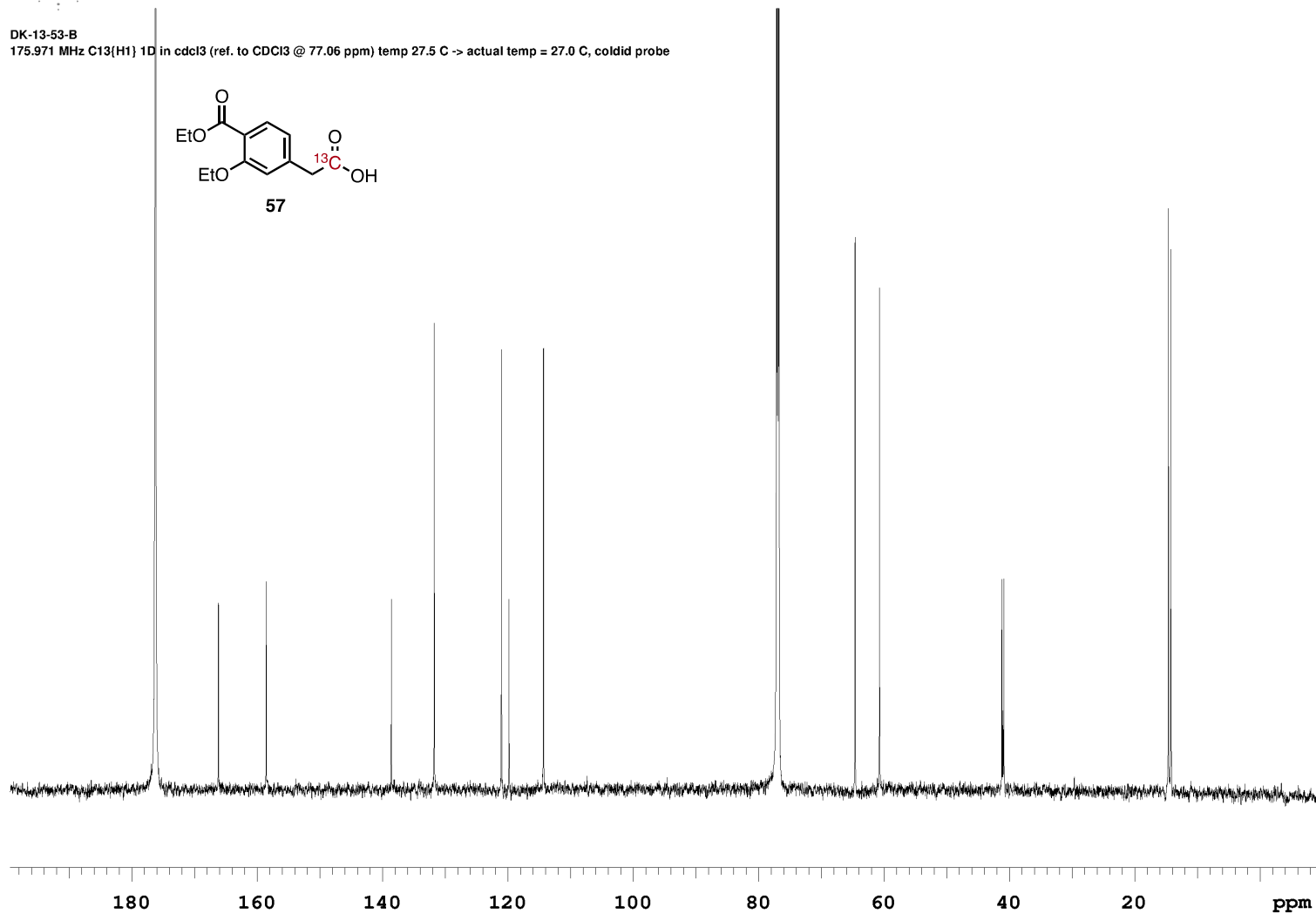
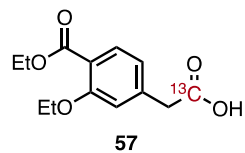
Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **512**

DK-13-53-B

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe





Agilent Technologies

Department of Chemistry, University of Alberta

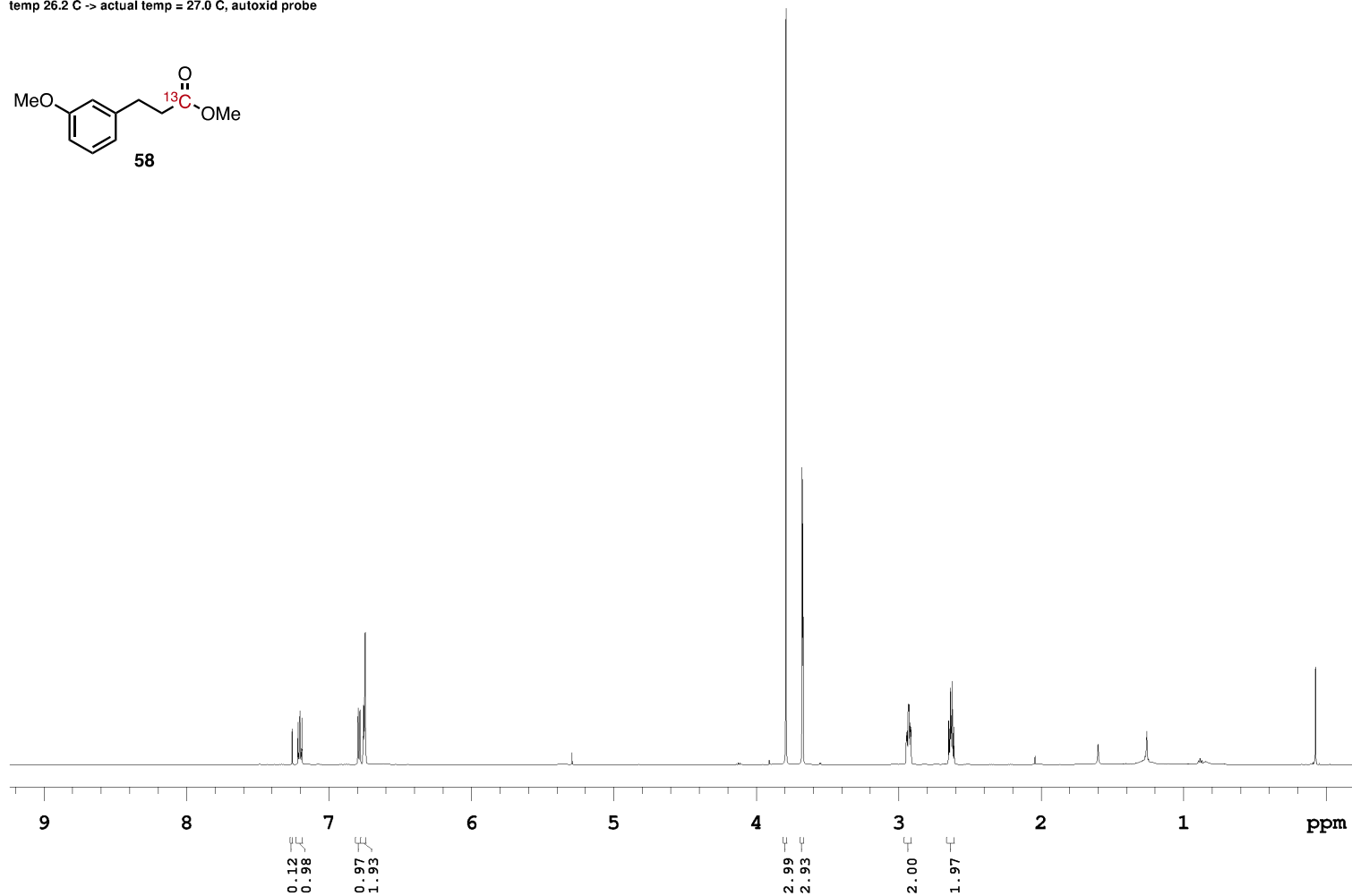
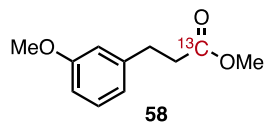
Recorded on: **i600, Aug 13 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **7183.91**
Digital Res.(Hz/pt): **0.11**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **23.61**

Relaxation Delay(s): **0.1**
Completed Scans: **16**

ob-02-181 599.926 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.2 C -> actual temp = 27.0 C, autotxid probe





Agilent Technologies

Department of Chemistry, University of Alberta

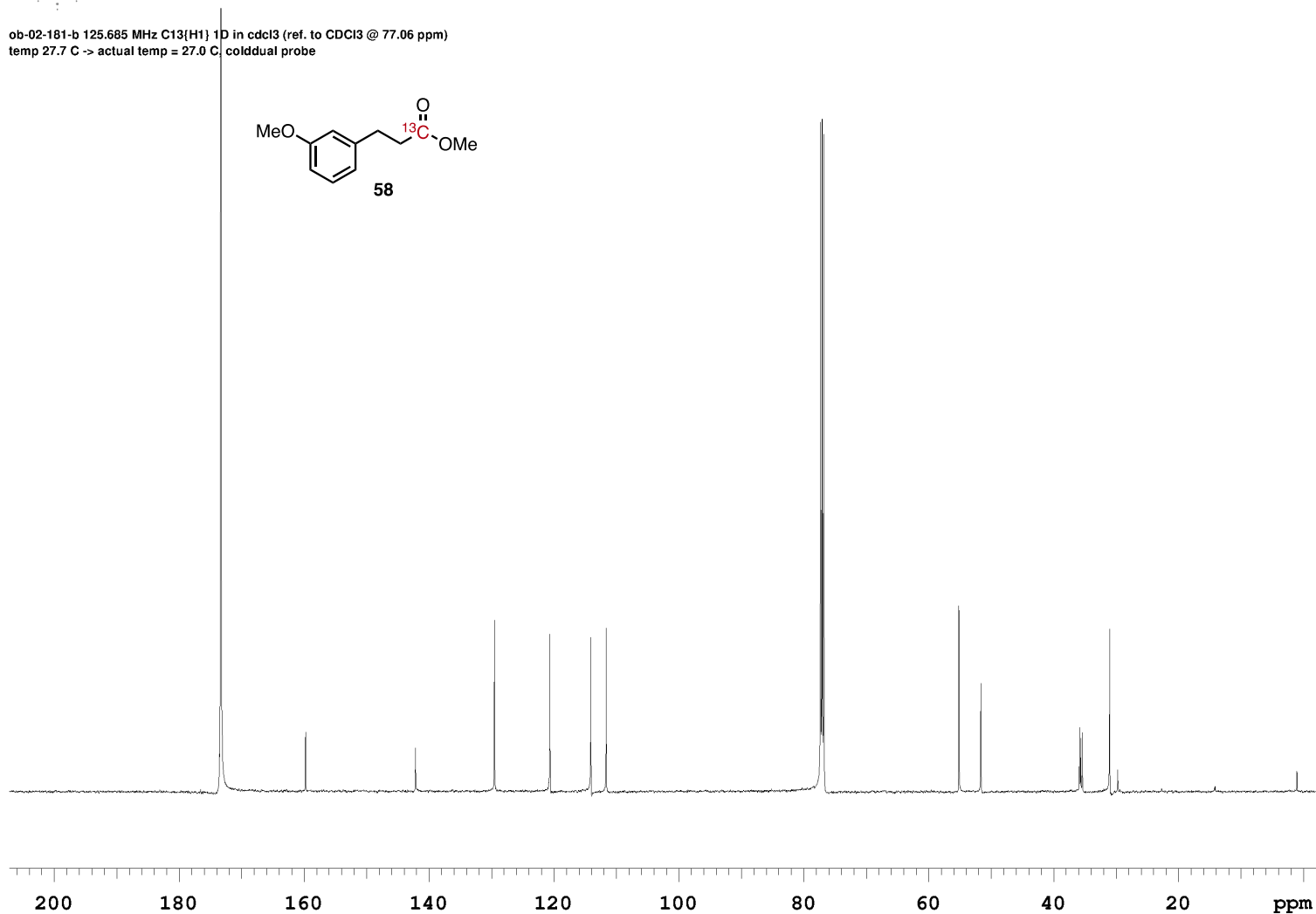
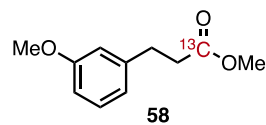
Recorded on: **u500, Aug 13 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **109.93**

Relaxation Delay(s): **1**
Completed Scans **188**

ob-02-181-b 125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C cold dual probe





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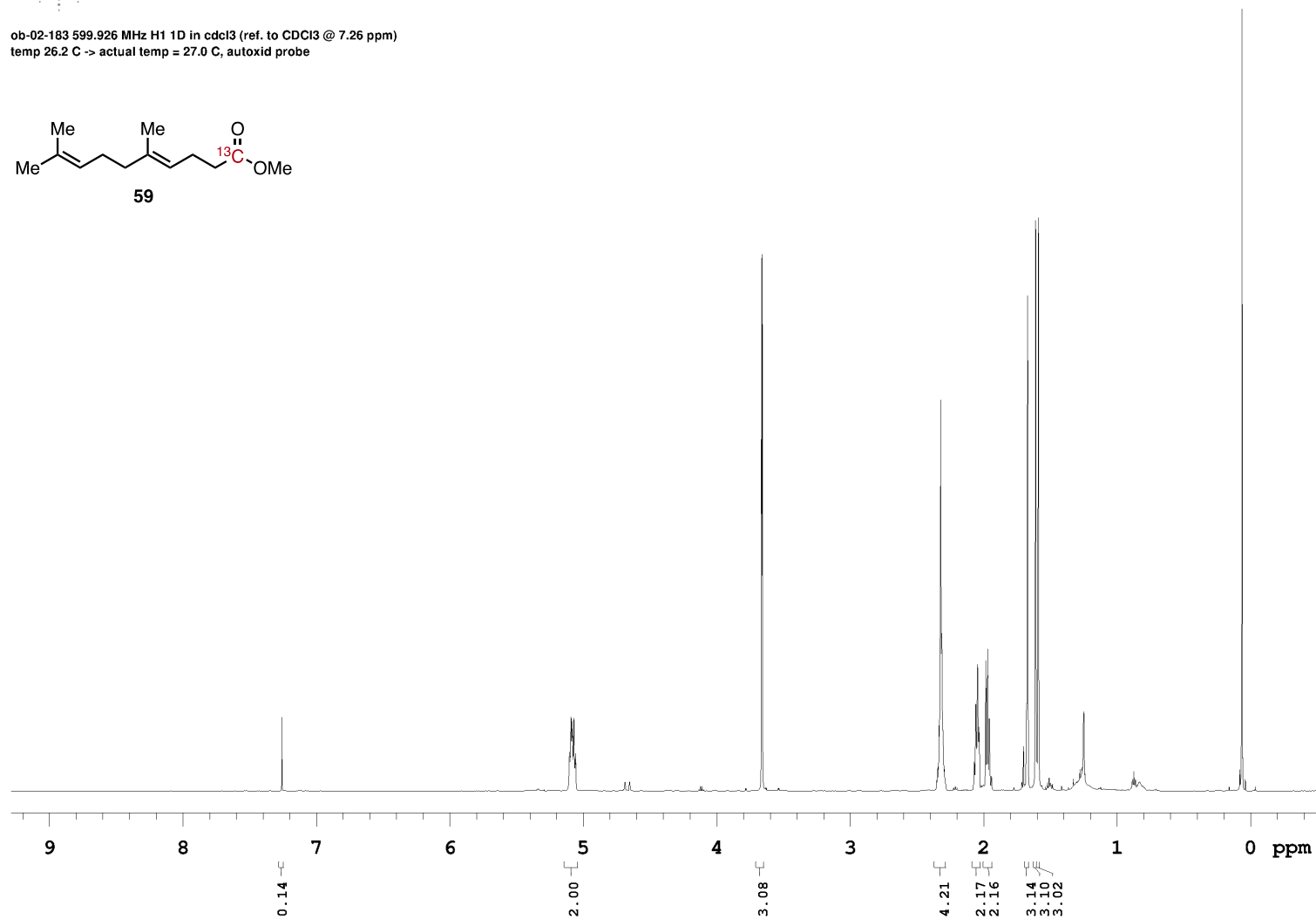
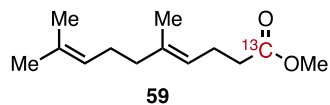
Recorded on: **i600, Aug 14 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **7183.91**
Digital Res.(Hz/pt): **0.11**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **24.51**

Relaxation Delay(s): **0.1**
Completed Scans **16**

ob-02-183 599.926 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.2 C -> actual temp = 27.0 C, autoxid probe





Agilent Technologies

Department of Chemistry, University of Alberta

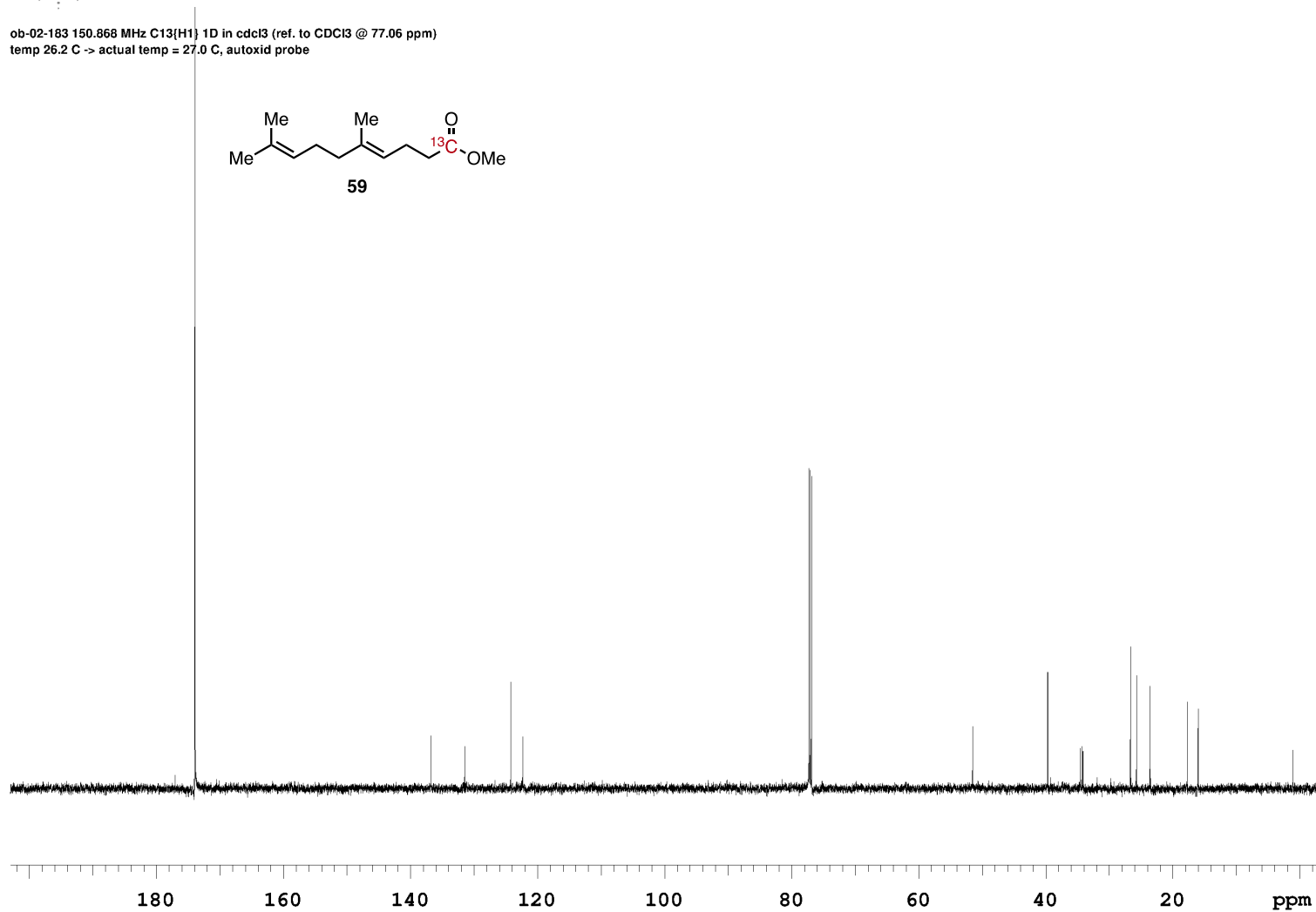
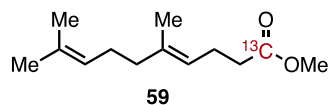
Recorded on: **i600, Aug 14 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **40322.6**
Digital Res.(Hz/pt): **0.31**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **129.53**

Relaxation Delay(s): **1**
Completed Scans **252**

ob-02-183 150.868 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 26.2 C -> actual temp = 27.0 C, autoxid probe





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Department of Chemistry, University of Alberta

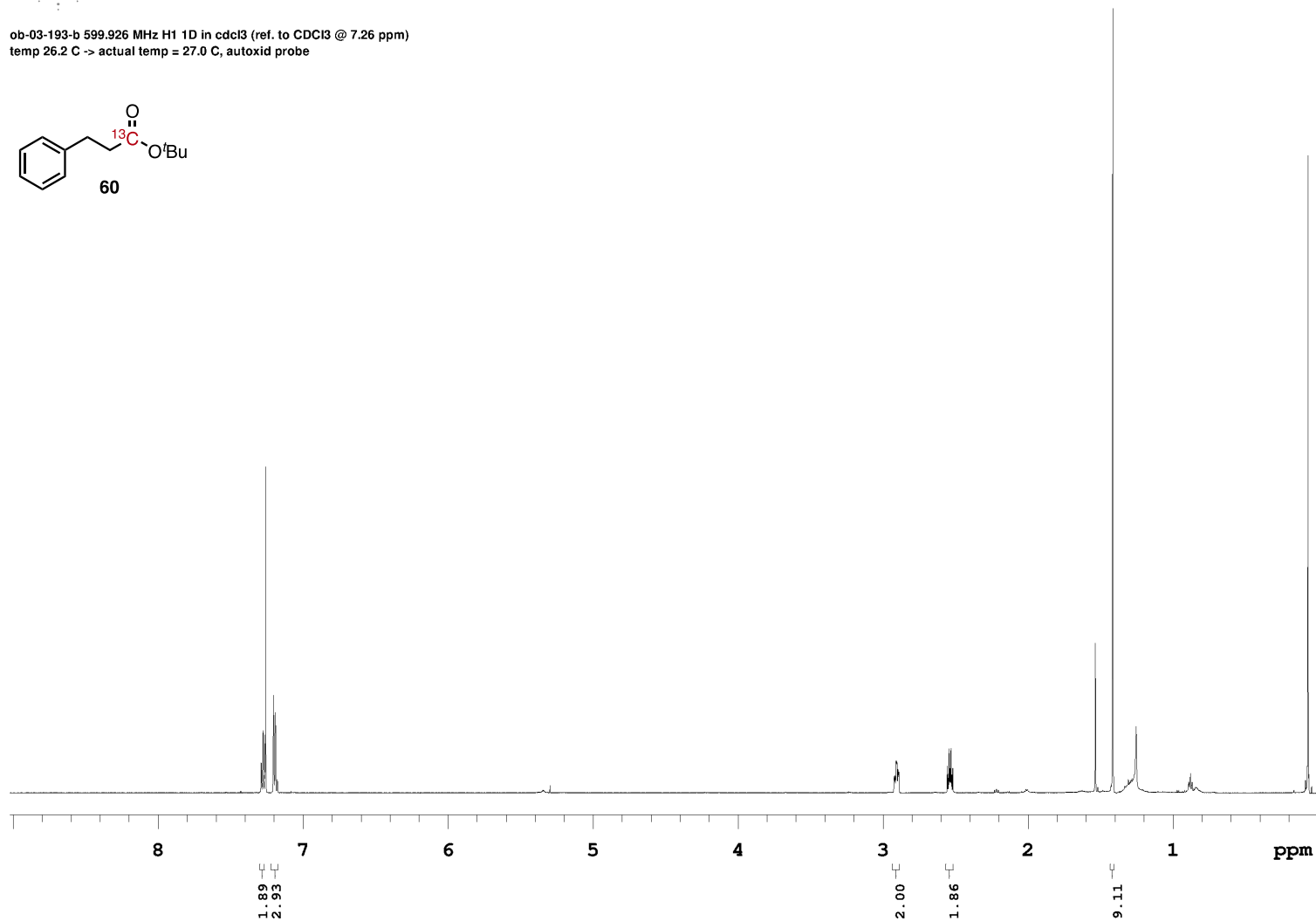
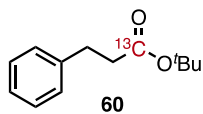
Recorded on: **i600, Feb 5 2020**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **7183.91**
Digital Res.(Hz/pt): **0.11**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **22.6**

Relaxation Delay(s): **0.1**
Completed Scans **8**

ob-03-193-b 599.926 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 26.2 C -> actual temp = 27.0 C, autoxid probe





Agilent Technologies

Department of Chemistry, University of Alberta

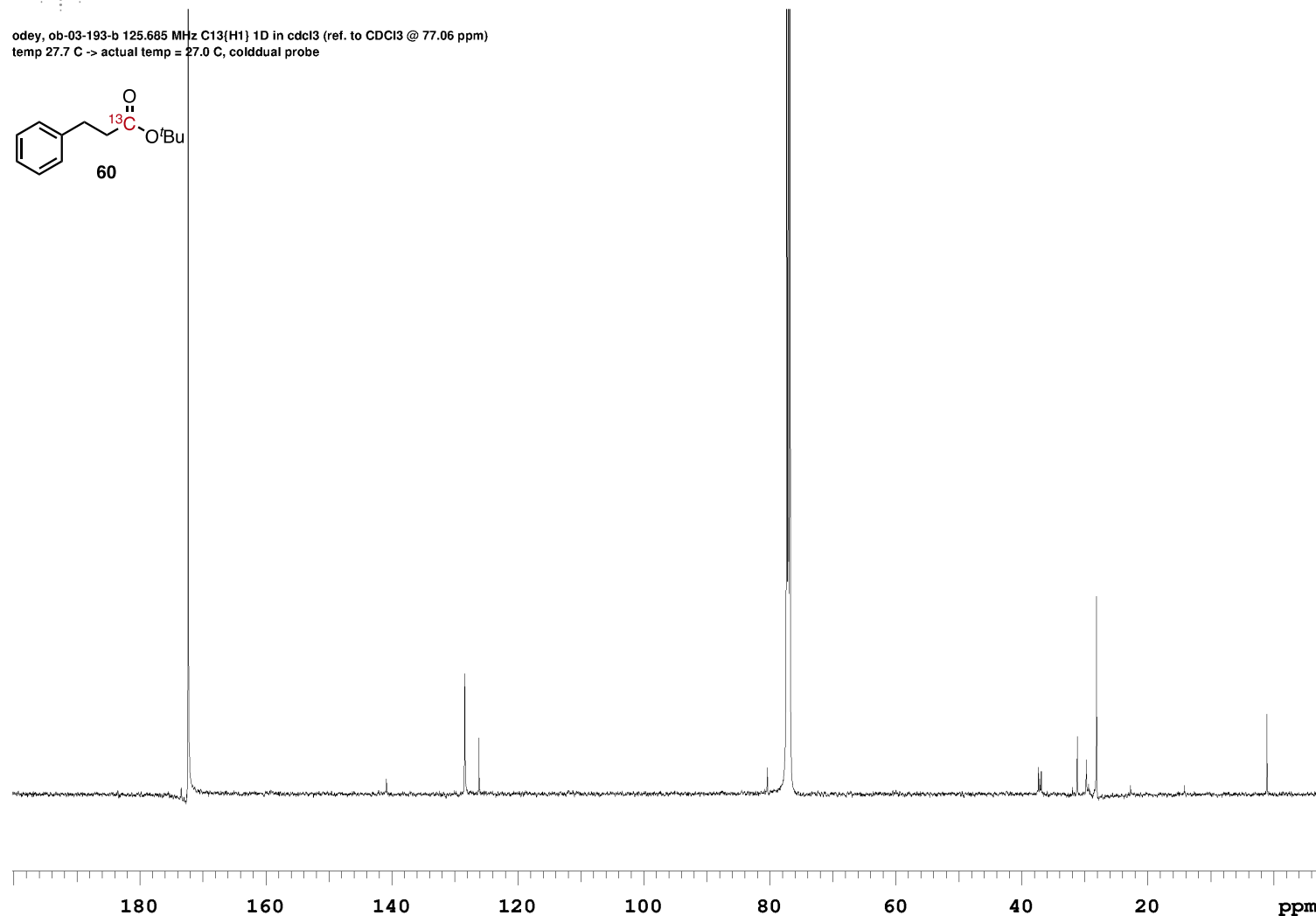
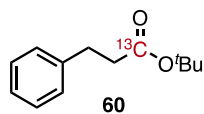
Recorded on: **u500, Feb 5 2020**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **109.15**

Relaxation Delay(s): **1**
Completed Scans: **812**

odey, ob-03-193-b 125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, cold dual probe





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Recorded on: **u500, Jan 24 2020**
Pulse Sequence: **PRESAT**

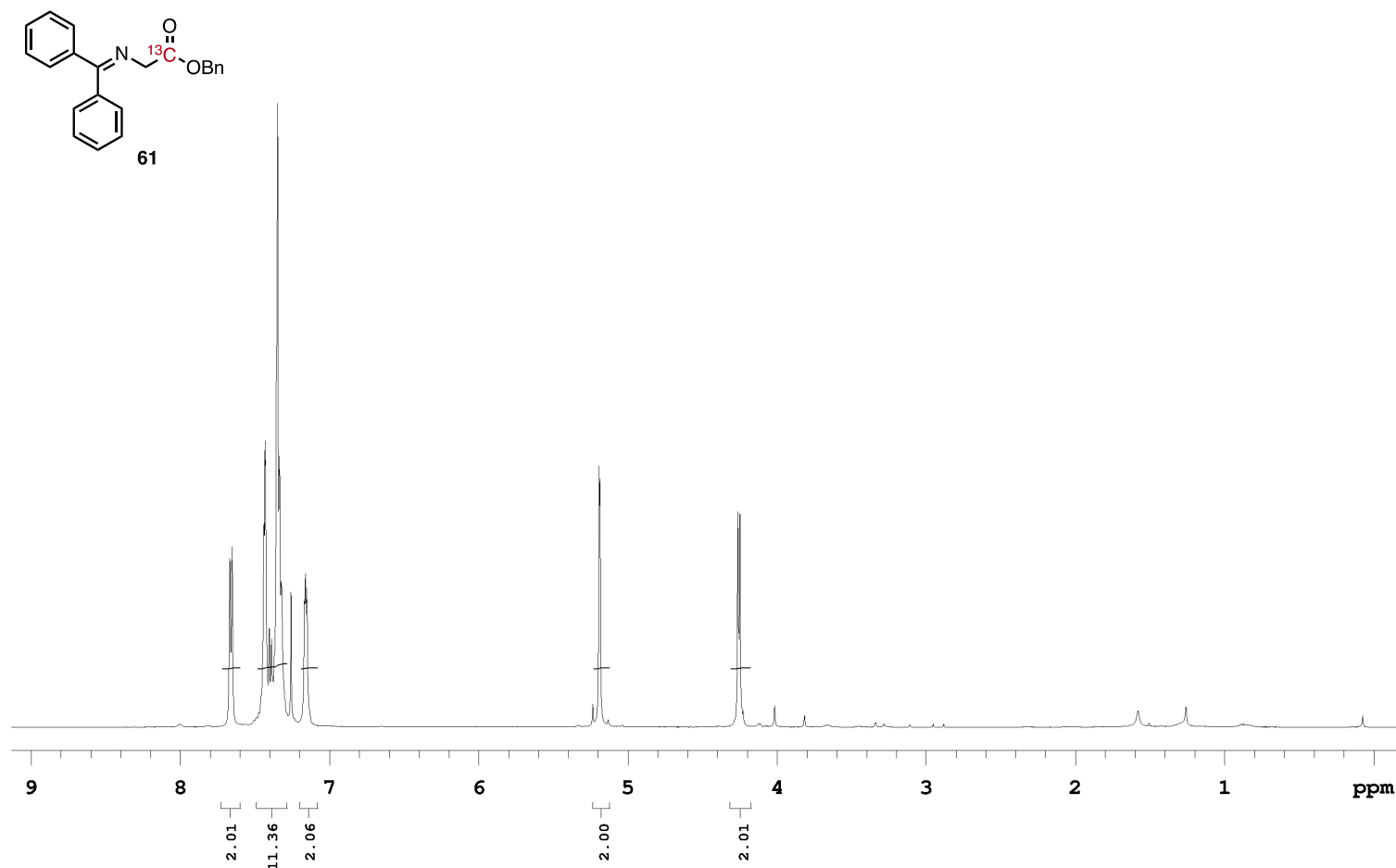
Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.42**

Relaxation Delay(s): **0.1**
Completed Scans: **8**

DK-13-71

499.787 MHz ¹H 1D in cdcl₃ (ref. to CDCl₃ @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Agilent Technologies

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Recorded on: **u500, Jan 24 2020**
Pulse Sequence: **s2pul**

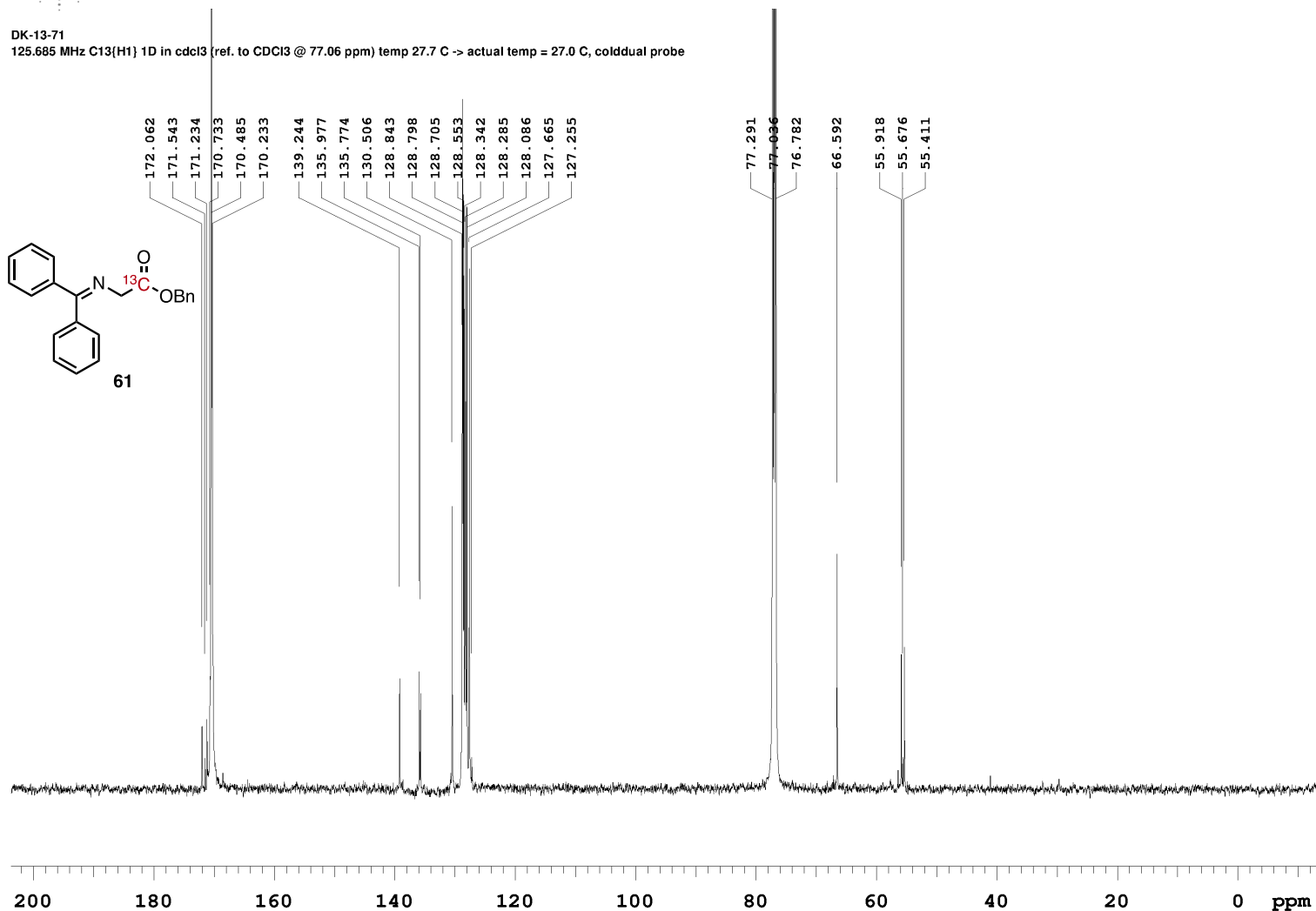
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **113.74**

Relaxation Delay(s): **1**
Completed Scans **344**

DK-13-71

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Agilent Technologies

Department of Chemistry, University of Alberta

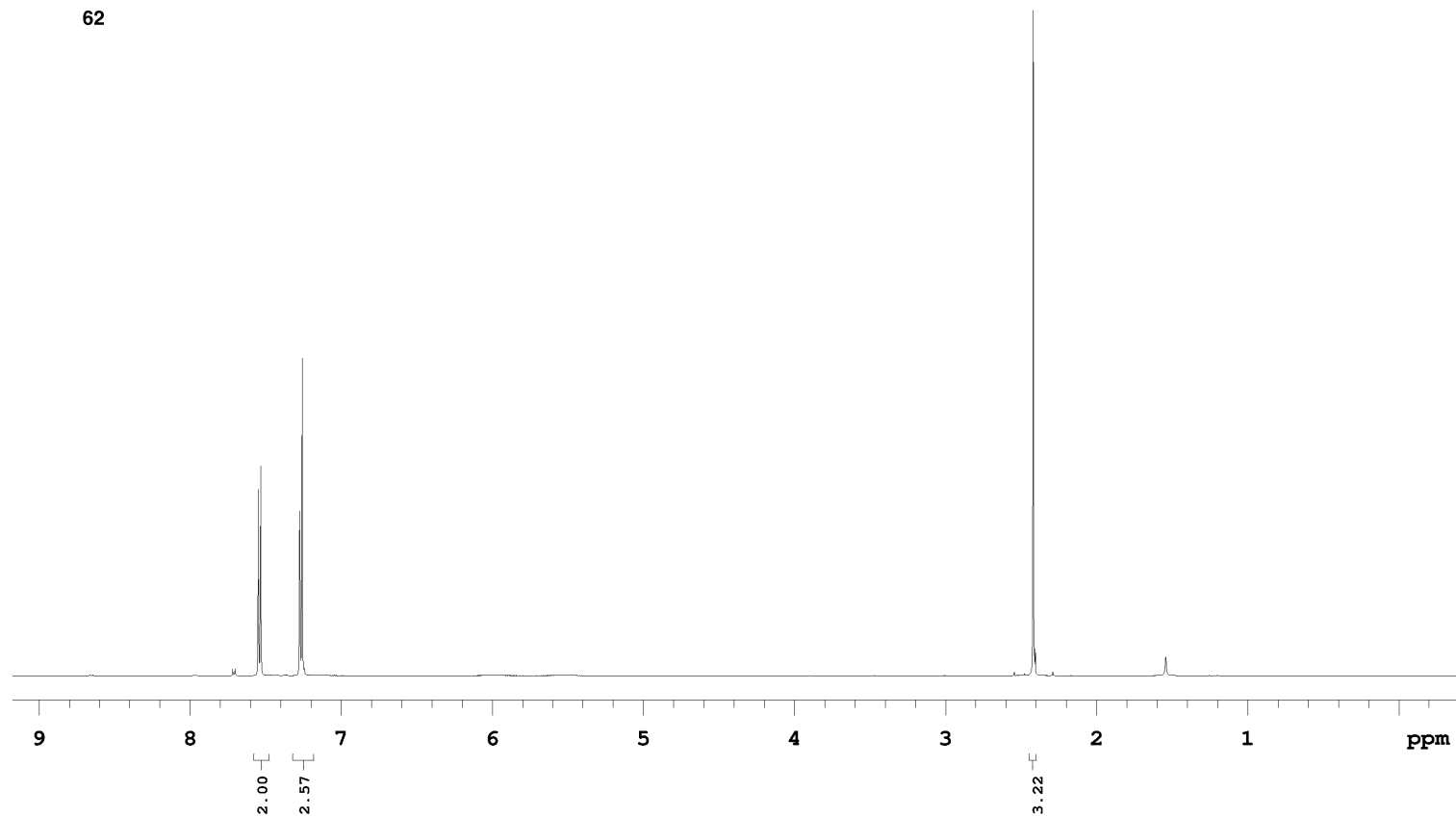
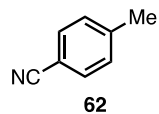
Recorded on: **u500, Feb 7 2020**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.93**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-13-17-CDCI3
499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Agilent Technologies

Department of Chemistry, University of Alberta

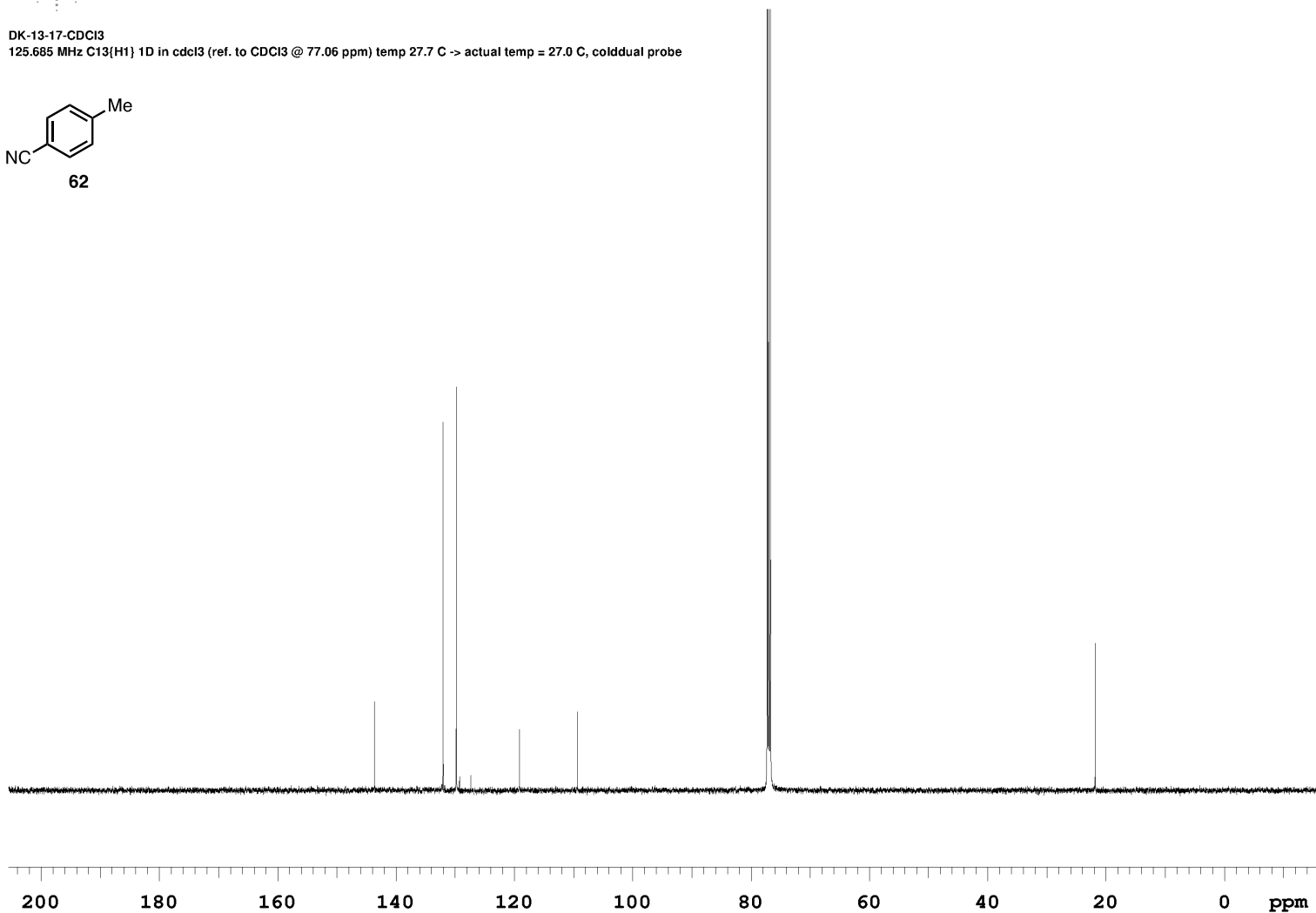
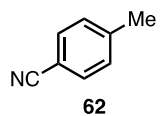
Recorded on: **u500, Feb 7 2020**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **115.71**

Relaxation Delay(s): **1**
Completed Scans **250**

DK-13-17-CDCl₃
125.685 MHz C13{H1} 1D in cdcl₃ (ref. to CDCl₃ @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jun 20 2019**
Pulse Sequence: **PRESAT**

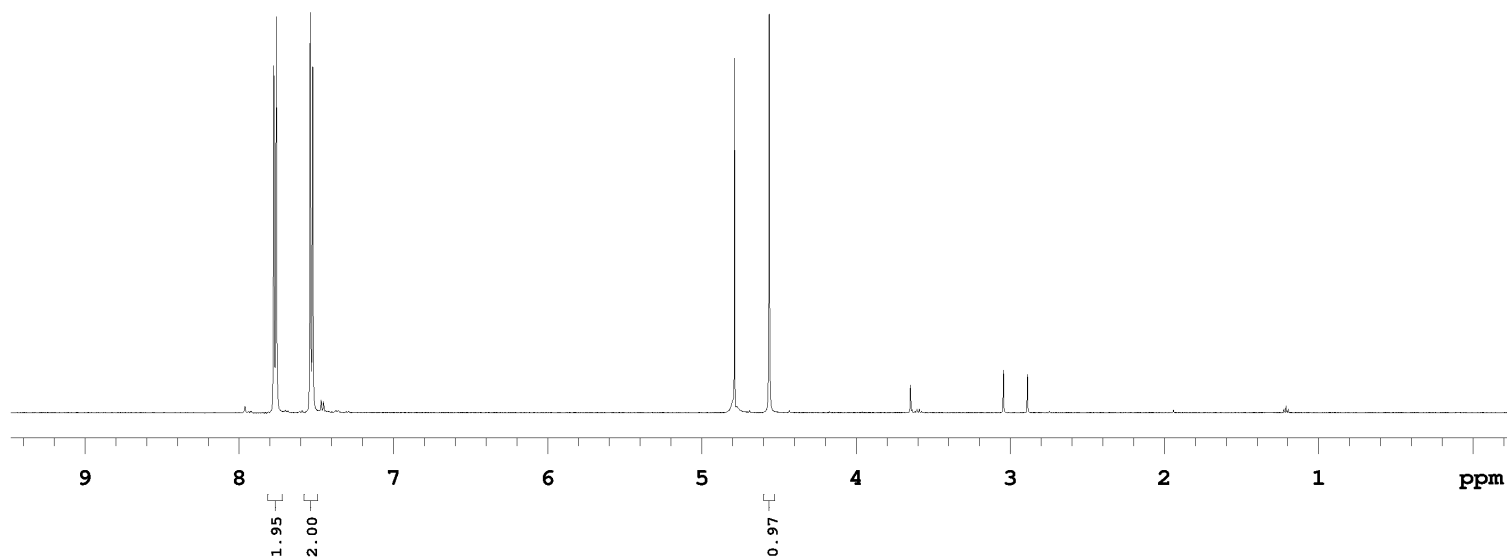
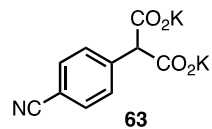
Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **3**
Hz per mm(Hz/mm): **20.28**

Relaxation Delay(s): **2**
Completed Scans **8**

DK-11-17

499.788 MHz H1 1D in d2o (ref. to external acetone @ 2.225 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe





Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jun 20 2019**
Pulse Sequence: **s2pul**

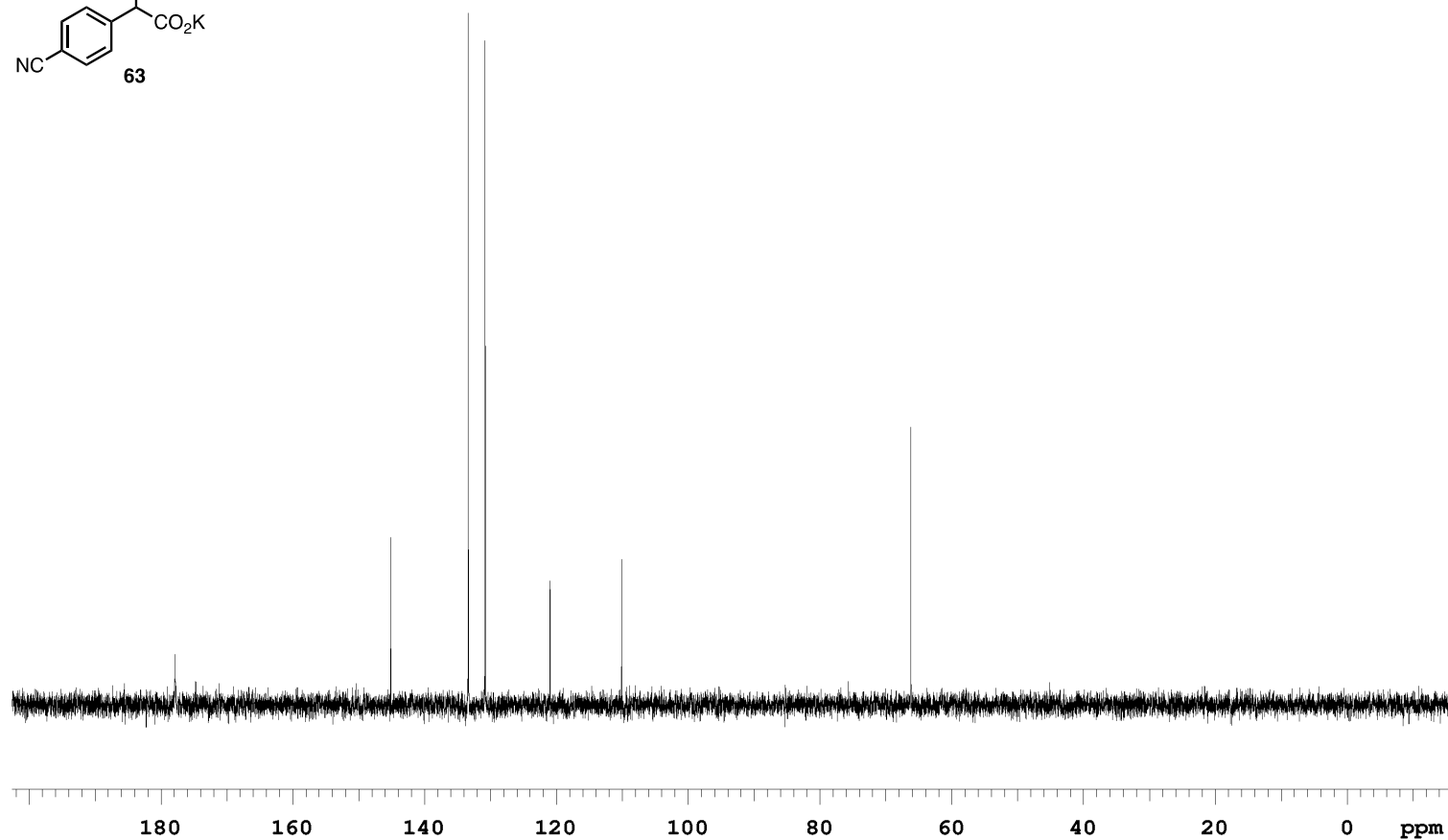
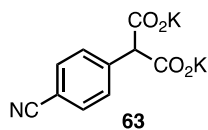
Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **114.43**

Relaxation Delay(s): **1**
Completed Scans **800**

DK-11-17-D2O

125.686 MHz C13{H1} 1D in d2o (ref. to external acetone @ 31.07 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe



Full_CO2_Exchange_SI_Feb25.pdf (25.54 MiB)

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