Expanding the Limit of Pd-Catalyzed Decarboxylative Benzylations

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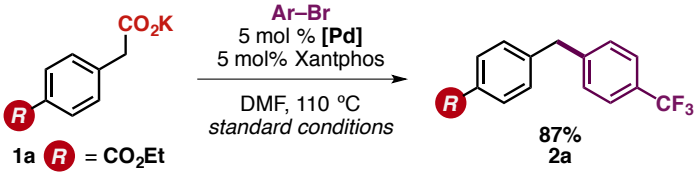
I. General Considerations

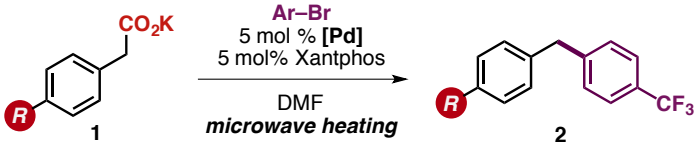
Unless noted, all reactions were conducted under inert atmosphere employing standard Schleck technique or by the use of a N_2-filled glovebox. All glassware was oven-dried prior to use. Flash chromatography was performed as described by Still and co-workers[1] (SiliaFlash P60, 40-63μm, 60A silica gel, Silicycle) or by automated flash chromatography (Ultra SNAP silica cartridges, Biotage). Analytical thin-layer chromatography was performed using glass plates pre-coated with silica (SiliaPlate G TLC - Glass-Backed, 250μm, Silicycle). TLC plates were visualized by UV light and/or staining with aqueous basic potassium permanganate. Unless otherwise noted, all reagents were obtained from commercial vendors and used as supplied. Potassium aryl acetate salts were synthesized from the corresponding aryl acetate acids as described by Liu and co-workers.[2]

II. Additional Optimization Data



entry	deviation from standard conditions	conv. **1a** / **Ar–Br** (%)		**2a** (%)
1	XPhos instead of Xantphos	60	46	8
2	SPhos instead of Xantphos	32	32	6
3	DPPF instead of Xantphos	59	50	5
4	BINAP instead of Xantphos	64	44	13
5	DPPP instead of Xantphos	50	17	14
6	DPPB instead of Xantphos	52	21	9
7	DPEPhos instead of Xantphos	77	51	13
8	SPANPhos instead of Xantphos	50	28	16
9	IPr instead of Xantphos	31	23	4
10	$Pd(OAc)_2$	101	82	63
11	$Pd(dba)_2$	111	98	71
12	$Pd(CH_3CN)_2Cl_2$	82	48	11

1a (0.12 mmol), ArBr (0.1 mmol), DMF (0.5 mL), 2.5h, conv. and yield determined by calibrated NMR, conversion of **1a** is calculated out of 120%, [Pd] = Pd(cinnamyl)Cl, Ar = 4-$CF_3C_6H_4$



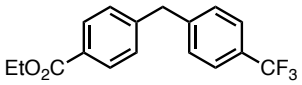
entry	R	temp.	conv. **1** / **Ar–Br** (%)		**2** (%)
1	**CN**	100 °C	91	73	65
2	**CN**	130 °C	120	100	84
3[a]	**CF_3**	100 °C	32	28	5
4[a]	**CF_3**	130 °C	70	44	43

1 (1.2 equiv), ArBr (1.0 equiv.), DMF (0.2 M), 1 h, conv. and yield determined by calibrated NMR, conversion of **a** is calculated out of 120%, [Pd] = Pd(cinnamyl)Cl; [a]with DEA-Xantphos

III Pd-Catalyzed Decarboxylative Benzylation of Aryl Bromides

General Procedure: Xantphos or DEA-Xantphos ligand (0.050 equiv.), potassium aryl acetate (1.2 equiv.) and aryl bromide (1.0 equiv.) were added sequentially to a 1-dram vial charged with a stir bar. $[Pd(cinnamyl)Cl]_2$ dimer (0.025 equiv.) was then added as a solution in anhydrous DMF (0.0050 M). The vial was sealed with a PTFE-lined cap under inert atmosphere, removed from the glovebox and heated while stirring. ^1H NMR analysis of small aliquots (~5 µL) was used to follow reactions to completion (1–24 hours), after which the mixture was

diluted in ethyl acetate (80 mL) and washed sequentially with saturated NH_4Cl (20 mL) and brine (20 mL). For reactions using heteroaryl bromides, Na_2CO_3 (1 M, 20 mL) was used instead of NH_4Cl. The organic layer was dried over Na_2SO_4, concentrated *in vacuo* and purified by flash column chromatography.



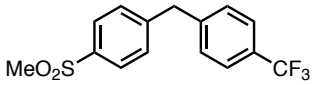
2a Prepared according to the General Procedure from the corresponding aryl bromide (113 mg, 0.50 mmol, 1.0 equiv.), potassium aryl acetate (148 mg, 0.60 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (6.5 mg, 0.0125 mmol, 0.025 equiv.), and XantPhos (14.5 mg, 0.025 mmol, 0.050 equiv.) in 2.5 mL DMF, 110 °C. Isolated in 73% yield after purification by silica gel chromatography (20:1 hexane:EtOAc) as a light yellow solid.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.99 (d, *J* = 8.0 Hz, 2H), 7.55 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.3 Hz, 2H), 7.24 (d, *J* = 8.3 Hz, 2H), 4.37 (q, *J* = 7.2 Hz, 2H), 4.09 (s, 2H), 1.38 (t, *J* = 7.2 Hz, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 166.4, 145.1, 144.3, 130.0, 129.3, 128.9 (2), 128.8 (q, *J* = 27.3 Hz) 127.4 (q, *J* = 272.6 Hz), 125.5 (q, *J* = 3.7 Hz), 60.9, 41.7, 14.4;

^{19}F NMR ($CDCl_3$, 469 MHz) δ -62.4;

HRMS (EI): calcd for $C_{17}H_{15}F_3O_2$ $[M]^+$: 308.1024. Found 308.1023.



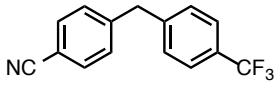
2b: Prepared according to the General Procedure from the corresponding aryl bromide (68 mg, 0.30 mmol, 1.0 equiv.), potassium aryl acetate (91 mg, 0.36 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (3.9 mg, 0.0075 mmol, 0.025 equiv.), and XantPhos (8.7 mg, 0.015 mmol, 0.050 equiv.) in 1.5 mL DMF, 90 °C. Isolated in 79% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a light yellow solid.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.89–7.87 (m, 2H), 7.57 (d, *J* = 8.2 Hz, 2H), 7.37 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 8.2 Hz, 2H), 4.13 (s, 2H), 3.04 (s, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 146.4, 143.4, 138.8, 129.8, 129.3, 129.1 (q, *J* = 32.5 Hz), 127.8, 126.2 (q, *J* = 272.7 Hz), 125.7 (q, *J* = 4.3 Hz), 44.5, 41.5;

^{19}F NMR ($CDCl_3$, 469 MHz) δ -62.5;

HRMS (EI): calcd for $C_{15}H_{13}F_3O_2S$ $[M]^+$: 314.0588. Found 314.0586.

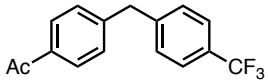


2c: Prepared according to the General Procedure from the corresponding aryl bromide (113 mg, 0.50 mmol, 1.0 equiv.), potassium aryl acetate (120 mg, 0.60 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (6.5 mg, 0.0125 mmol, 0.025 equiv.), and XantPhos (14.7 mg, 0.025 mmol, 0.050 equiv.) in 2.5 mL DMF, 100 °C. Isolated in 76% yield after purification by silica gel chromatography (20:1 to 10:1 hexane:EtOAc) as a light yellow oil. Spectroscopic data agreed with that reported.[3]

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.62–7.57 (m, 4H), 7.30–7.27 (m, 4H), 4.11 (s, 2H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 145.5, 143.3, 132.5, 129.6, 129.3, 129.1 (q, J = 32.4 Hz), 125.7 (q, J = 3.8 Hz), 124.1 (q, J = 271.5 Hz), 118.8, 110.6, 41.7;

^{19}F NMR ($CDCl_3$, 469 MHz) δ -62.5.

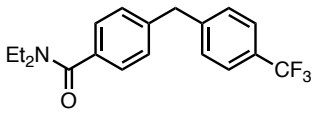


2d: Prepared according to the General Procedure from the corresponding aryl bromide (45 mg, 0.20 mmol, 1.0 equiv.), potassium aryl acetate (52 mg, 0.24 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (2.6 mg, 0.005 mmol, 0.025 equiv.), and XantPhos (5.8 mg, 0.01 mmol, 0.050 equiv.) in 1.0 mL DMF, 110 °C. Isolated in 72% yield after purification by silica gel chromatography (8:1 hexane:EtOAc) as a light yellow oil. Spectroscopic data agreed with that reported.[4]

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.91–7.89 (m, 2H), 7.55 (d, J = 7.5 Hz, 2H), 7.29–7.26 (m, 4H), 4.09 (s, 2H), 2.58 (s, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 197.6, 145.5, 144.0, 135.6, 129.2, 129.1, 128.9 (q, J = 32.6 Hz), 128.7, 125.5 (q, J = 3.9 Hz), 124.2 (q, J = 273.0 Hz), 41.6, 26.6;

^{19}F **NMR** ($CDCl_3$, 469 MHz) δ -62.4.



2e: Prepared according to the General Procedure from the corresponding aryl bromide (45 mg, 0.20 mmol, 1.0 equiv.), potassium aryl acetate (66 mg, 0.24 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (2.6 mg, 0.005 mmol, 0.025 equiv.), and DEA-XantPhos (5.6 mg, 0.01 mmol,

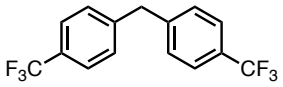
0.050 equiv.) in 1.0 mL DMF, 135 °C. Isolated in 60% yield after purification by silica gel chromatography (2:1 hexane:EtOAc) as a light yellow solid.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.54 (d, *J* = 8.1 Hz, 2H), 7.32-7.26 (m, 4H), 7.19 (d, *J* = 8.1 Hz, 2H), 4.04 (s, 2H), 3.38 (br, 4H), 1.17 (br, 6H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 171.1, 144.6, 141.0, 135.5, 129.2, 128.9, 128.7 (q, *J* = 33.8 Hz), 126.8, 125.4 (q, *J* = 4.2 Hz), 124.3 (q, *J* = 271.3 Hz), 43.3, 41.5, 39.3, 14.2, 12.9;

^{19}F NMR ($CDCl_3$, 469 MHz) δ -62.3;

HRMS (ESI): calcd for $C_{19}H_{21}F_3NO$ $[M+H]^+$: 336.1570. Found 336.1571.

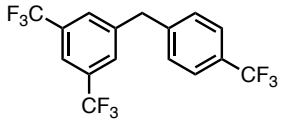


2f: Prepared according to the General Procedure from the corresponding aryl bromide (113 mg, 0.50 mmol, 1.0 equiv.), potassium aryl acetate (145 mg, 0.60 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (6.5 mg, 0.0125 mmol, 0.025 equiv.), and DEA-XantPhos (14 mg, 0.025 mmol, 0.050 equiv.) in 2.5 mL DMF, 125 °C. 66% yield by ^{1}H NMR using durene as internal standard (53% isolated yield). Spectroscopic data agreed with that reported.[5]

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.56 (d, *J* = 8.2 Hz, 4H), 7.29 (d, *J* = 8.2 Hz, 4H), 4.09 (s, 2H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 143.9, 129.2, 128.9 (q, *J* = 33.8 Hz), 125.6 (q, *J* = 4.2 Hz), 124.1 (q, *J* = 271.3 Hz), 41.5;

^{19}F NMR ($CDCl_3$, 469 MHz) δ -62.4.

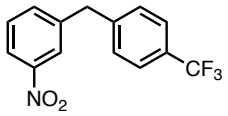


2g: Prepared according to the General Procedure from the corresponding aryl bromide (113 mg, 0.50 mmol, 1.0 equiv.), potassium aryl acetate (186 mg, 0.60 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (6.5 mg, 0.0125 mmol, 0.025 equiv.), and DEA-XantPhos (14 mg, 0.025 mmol, 0.050 equiv.) in 2.5 mL DMF, 110 °C. Isolated in 61% yield after purification by silica gel chromatography (40:1 hexane:EtOAc) as a white solid. Spectroscopic data agreed with that reported.[6]

^{1}H NMR ($CDCl_3$, 700 MHz) δ 7.77 (s, 1H), 7.62–7.59 (m, 4H), 7.29 (d, *J* = 7.5 Hz, 2H), 4.16 (s, 2H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 142.7, 142.3, 132.0 (q, *J* = 39.2 Hz), 129.5 (q, *J* = 39.2 Hz), 129.1, 129.0-128.9 (m), 125.9 (q, *J* = 3.7 Hz), 124.0 (q, *J* = 272.7 Hz), 123.2 (q, *J* = 272.7 Hz), 120.8-120.6 (m), 41.2;

^{19}F NMR ($CDCl_3$, 469 MHz) δ -62.5, -62.8.



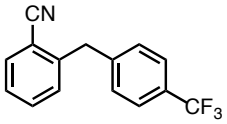
2h: Prepared according to the General Procedure from the corresponding aryl bromide (45 mg, 0.20 mmol, 1.0 equiv.), potassium aryl acetate (53 mg, 0.24 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (5.2 mg, 0.01 mmol, 0.05 equiv.), and DEA-XantPhos (11.2 mg, 0.02 mmol, 0.10 equiv.) in 1.0 mL DMF, 120 °C. Isolated in 48% yield after purification by silica gel chromatography (15:1 hexane:EtOAc) as a white solid.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 8.12–8.09 (m, 1H), 8.06 (s, 1H), 7.58 (d, *J* = 6.8 Hz, 2H), 7.52–7.47 (m, 2H), 7.31 (d, *J* = 6.8 Hz, 2H), 4.17 (s, 2H);

^{13}C NMR ($CDCl_3$, 176 MHz) δ 148.5, 143.3, 141.9, 134.9, 129.6, 129.2, 129.1 (q, *J* = 35.0 Hz), 125.7 (q, *J* = 2.3 Hz), 124.1 (q, *J* = 271.2 Hz), 123.7, 121.7, 41.2;

^{19}F NMR ($CDCl_3$, 469 MHz) δ -62.5;

HRMS (EI): calcd for $C_{14}H_{10}F_3NO_2$ $[M]^+$: 281.0664. Found 281.0666.

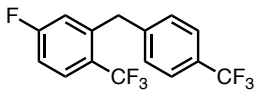


2i: Prepared according to the General Procedure from the corresponding aryl bromide (45 mg, 0.2 mmol, 1.0 equiv.), potassium aryl acetate (48 mg, 0.24 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (2.6 mg, 0.005 mmol, 0.025 equiv.), and XantPhos (5.8 mg, 0.010 mmol, 0.050 equiv.) in 1.0 mL DMF, 105 °C. Isolated in 80% yield after purification by silica gel chromatography (2% to 15% EtOAc in Hexane) as a yellow oil. Spectroscopic data agreed with that reported.[7]

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.67 (m, 1H), 7.66 (m, 2H), 7.57 (m, 1H), 7.36–7.33 (m, 3H), 7.27 (m, 1H), 4.26 (s, 2H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 143.7, 142.8, 133.1, 130.1, 129.7, 129.3, 129.0 (q, *J* = 32.4 Hz), 127.3, 125.7 (q, *J* = 3.8 Hz), 124.2 (q, J = 272.7 Hz), 118.0, 112.7, 41.6;

^{19}F NMR ($CDCl_3$, 377 MHz) δ -62.6.



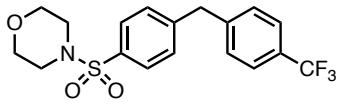
2j: Prepared according to the General Procedure from the corresponding aryl bromide (68 mg, 0.30 mmol, 1.0 equiv.), potassium aryl acetate (93 mg, 0.36 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (3.9 mg, 0.0075 mmol, 0.025 equiv.), and XantPhos (8.7 mg, 0.015 mmol, 0.050 equiv.) in 1.5 mL DMF, 110 °C. 65% yield by ^{1}H NMR using durene as internal standard.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.73-7.68 (m, 1H), 7.58 (d, *J* = 8.2 Hz, 2H), 7.26 (m, 2H), 7.05–7.01 (m, 1H), 6.82 (m, 1H), 4.25 (s, 2H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 164.5 (d, *J* = 252.6 Hz), 142.9, 141.7 (d, *J* = 8.3 Hz), 129.4, 129.1 (q, *J* = 32.6 Hz), 128.6 (m), 127.6, 125.9 (q, *J* = 3.8 Hz), 125.6 (m), 125.0 (m), 124.1 (q, *J* = 232.6 Hz), 124.0 (q, *J* = 233.5 Hz), 118.7 (d, *J* = 22.8 Hz), 113.7 (d, *J* = 21.9 Hz), 37.6;

^{19}F NMR ($CDCl_3$, 469 MHz) δ -58.9, -62.5, -107.4;

HRMS (EI): calcd for $C_{15}H_9F_7$ $[M]^+$: 322.0592. Found 322.0589.



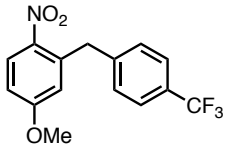
2k: Prepared according to the General Procedure from the corresponding aryl bromide (23 mg, 0.10 mmol, 1.0 equiv.), potassium aryl acetate (39 mg, 0.12 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (1.3 mg, 0.0025 mmol, 0.025 equiv.), and XantPhos (2.9 mg, 0.005 mmol, 0.050 equiv.) in 0.5 mL DMF, 100 °C. Isolated in 75% yield after purification by silica gel chromatography (8:1 to 2:1 hexane:EtOAc) as a yellow solid.

^{1}H NMR ($CDCl_3$, 700 MHz) δ 7.69 (d, *J* = 8.3 Hz, 2H), 7.58 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.5 Hz, 2H), 7.30 (d, *J* = 8.5 Hz, 2H), 4.12 (s, 2H), 3.74 (t, *J* = 4.8 Hz, 4H), 2.99 (m, 4H);

^{13}C NMR ($CDCl_3$, 176 MHz) δ 145.7, 143.4, 133.3, 129.6, 129.3, 129.1 (q, *J* = 33.3 Hz), 128.3, 125.7 (q, *J* = 3.5 Hz), 124.0 (q, *J* = 271.3 Hz), 66.1, 45.9, 41.4;

^{19}F NMR ($CDCl_3$, 469 MHz) δ -62.4;

HRMS (ESI): calcd for $C_{18}H_{18}F_3NO_3SNa$ $[M+Na]^+$: 408.0852. Found 408.0851.



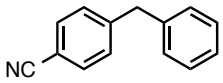
2l: Prepared according to the General Procedure from the corresponding aryl bromide (45 mg, 0.20 mmol, 1.0 equiv.), potassium aryl acetate (60 mg, 0.24 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (2.6 mg, 0.005 mmol, 0.025 equiv.), and XPhos (7.2 mg, 0.015 mmol, 0.075 equiv.) in 1.0 mL DMF, 40 °C. Isolated in 82% yield after purification by silica gel chromatography (20:1 to 10:1 hexane:EtOAc) as a yellow solid.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 8.13 (d, *J* = 9.2 Hz, 1H), 7.54 (d, *J* = 8.2 Hz, 2H), 7.28–7.25 (m, 2H), 6.88 (dd, *J* = 9.2 Hz, 2.8 Hz, 1H), 6.73 (d *J* = 2.8 Hz, 1H), 4.41 (s, 2H), 3.86 (s, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 163.3, 142.8, 142.0, 137.9, 129.1, 128.9 (q, *J* = 31.3 Hz), 128.7, 125.5 (q, *J* = 3.6 Hz), 124.2 (q, *J* = 271.3 Hz), 118.0, 112.2, 55.9, 39.2;

^{19}F NMR ($CDCl_3$, 469 MHz) δ -62.4;

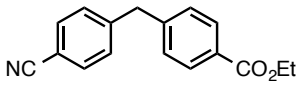
HRMS (ESI): calcd for $C_{15}H_{11}F_3NO_3$ $[M-H]^+$: 310.0697. Found 310.0690



3a: Prepared according to the General Procedure from the corresponding aryl bromide (79 mg, 0.50 mmol, 1.0 equiv.), potassium aryl acetate (120 mg, 0.60 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (6.5 mg, 0.0125 mmol, 0.025 equiv.), and XantPhos (14.7 mg, 0.025 mmol, 0.050 equiv.) in 2.5 mL DMF, 100 °C. Isolated in 81% yield after purification by silica gel chromatography (20:1 to 10:1 Pentane:Et_2O) as a pale yellow oil. Spectroscopic data agreed with that reported.[8]

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.57 (m, 2H), 7.33–7.24 (m, 5H), 7.16 (m, 2H), 4.04 (s, 2H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 146.7, 139.3, 132.3, 129.6, 129.1, 128.8, 126.7, 119.0, 110.1, 42.0;



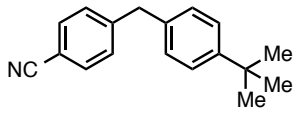
3b: Prepared according to the General Procedure from the corresponding aryl bromide (115 mg, 0.50 mmol, 1.0 equiv.), potassium aryl acetate (120 mg, 0.60 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (6.5 mg, 0.0125 mmol, 0.025 equiv.), and XantPhos (14.7 mg, 0.025 mmol,

0.050 equiv.) in 2.5 mL DMF, 100 °C. Isolated in 68% yield after purification by silica gel chromatography (15:1 to 8:1 hexane:EtOAc) as an off-white solid.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.99 (m, 2H), 7.59 (m, 2H), 7.27 (m, 2H), 7.22 (m, 2H), 4.37 (q, *J* = 7.1 Hz, 2H), 4.08 (s, 2H), 1.38 (t, *J* = 7.1 Hz, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 166.3, 145.7, 144.4, 132.4, 130.1, 129.7, 129.1, 128.9, 118.8, 110.4, 61.0, 41.9, 14.3;

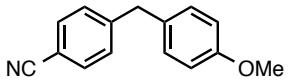
HRMS (EI): calcd for $C_{17}H_{15}NO_2$ $[M]^+$: 265.1103. Found 265.1105.



3c: Prepared according to the General Procedure from the corresponding aryl bromide (107 mg, 0.50 mmol, 1.0 equiv.), potassium aryl acetate (120 mg, 0.60 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (6.5 mg, 0.0125 mmol, 0.025 equiv.), and XantPhos (14.7 mg, 0.025 mmol, 0.050 equiv.) in 2.5 mL DMF, 100 °C. Isolated in 58% yield after purification by silica gel chromatography (20:1 to 10:1 Pentane:Et_2O) as a pale yellow oil. Spectroscopic data agreed with that reported.[6]

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.58 (m, 2H), 7.34 (m, 2H), 7.29 (m,2H), 7.09 (m, 2H), 4.00(s, 2H), 1.31 (s, 9H);

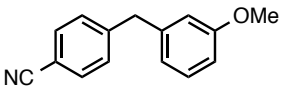
^{13}C NMR ($CDCl_3$, 126 MHz) δ 149.6, 146.9, 136.3, 132.3, 129.7, 128.6, 128.6, 125.7, 119.0, 41.5, 34.4, 31.4.



3d: Prepared according to the General Procedure from the corresponding aryl bromide (94 mg, 0.50 mmol, 1.0 equiv.), potassium aryl acetate (120 mg, 0.60 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (6.5 mg, 0.0125 mmol, 0.025 equiv.), and XantPhos (14.7 mg, 0.025 mmol, 0.050 equiv.) in 2.5 mL DMF, 100 °C. 75% yield by ^{1}H NMR using durene as internal standard. Spectroscopic data agreed with that reported.[1]

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.56 (m, 2H), 7.26 (m, 2H), 7.07 (m, 2H), 6.85 (m, 2H), 3.97 (s, 2H), 3.79 (s, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 158.4, 147.2, 132.3, 131.4, 129.9, 129.5, 119.0, 114.2, 110.0, 55.3, 41.1.

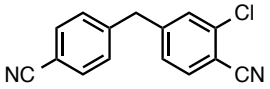


3e: Prepared according to the General Procedure from the corresponding aryl bromide (37 mg, 0.20 mmol, 1.0 equiv.), potassium aryl acetate (48 mg, 0.24 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (2.6 mg, 0.0050 mmol, 0.025 equiv.), and XantPhos (5.8 mg, 0.010 mmol, 0.050 equiv.) in 1.0 mL DMF, 100 °C. Isolated in 49% yield after purification by silica gel chromatography (50:1 to 4:1 hexane:EtOAc) as a colourless oil.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.59–7.55 (m, 2H), 7.30–7.27 (m, 2H), 7.23 (t, *J* = 8.1 Hz, 1H), 6.78 (dd, *J* = 8.2, 2.6 Hz, 1H), 6.75 (d, *J* = 7.4 Hz, 1H), 6.69 (m, 1H), 4.00 (s, 2H), 3.78 (s, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 160.1, 146.7, 141.0, 132.4, 129.9, 129.8, 121.5, 119.1, 115.1, 111.9, 110.3, 55.3, 42.1;

HRMS (EI): calcd for $C_{15}H_{13}NO$ $[M]^+$: 223.0997. Found 223.0997.

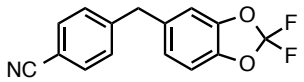


3f: Prepared according to the General Procedure from the corresponding aryl bromide (43 mg, 0.2 mmol, 1.0 equiv.), potassium aryl acetate (48 mg, 0.24 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (2.6 mg, 0.005 mmol, 0.025 equiv.), and XantPhos (5.8 mg, 0.010 mmol, 0.050 equiv.) in 1.0 mL DMF, 100 °C. Isolated in 85% yield after purification by silica gel chromatography (8:1 to 4:1 Hexane:EtOAc) as a white solid.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.64–7.60 (m, 3H), 7.31 (m, 1H), 7.27 (m, 2H), 7.16 (m, 1H), 4.07 (s, 2H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 146.4, 143.9, 137.1, 134.2, 132.7, 130.4, 129.7, 127.7, 118.5, 115.8, 111.8, 111.2, 41.6;

HRMS (EI): calcd for $C_{15}H_9{}^{35}ClN_2$ $[M]^+$: 252.0454. Found 252.0451.



3g: Prepared according to the General Procedure from the corresponding aryl bromide (59 mg, 0.25 mmol, 1.0 equiv.), potassium aryl acetate (60 mg, 0.30 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (3.2 mg, 0.00625 mmol, 0.025 equiv.), and XantPhos (7.2 mg, 0.0125 mmol,

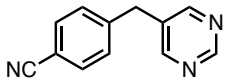
0.050 equiv.) in 1.25 mL DMF, 100 °C. Isolated in 83% yield after purification by silica gel chromatography (2% to 18% EtOAc in Hexane) as a white solid.

^{1}H NMR ($CDCl_3$, 700 MHz) δ 7.58 (m, 2H), 7.26 (m, 2H), 6.98 (m, 1H), 6.87 (m, 1H), 6.84 (m,1H), 4.01 (s, 2H);

^{13}C NMR ($CDCl_3$, 176 MHz) δ 145.8, 144.1, 142.5, 135.4, 132.4, 131.6 (t, J = 251.9Hz), 129.5, 123.9, 118.8, 110.5, 110.1, 109.5, 41.6;

^{19}F NMR ($CDCl_3$, 378 MHz) δ -50.1;

HRMS (EI): calcd for $C_{15}H_9NO_2F^2$ $[M]^+$: 273.0602. Found 273.0606.

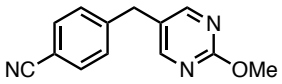


3h: Prepared according to the General Procedure from the corresponding aryl bromide (40 mg, 0.25 mmol, 1.0 equiv.), potassium aryl acetate (60 mg, 0.30 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (3.3 mg, 0.00625 mmol, 0.025 equiv.), and XantPhos (7.3 mg, 0.0125 mmol, 0.050 equiv.) in 1.25 mL DMF, 100 °C. Isolated in 87% yield after purification by silica gel chromatography (1:3 hexane:EtOAc) as a yellow solid.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 9.12 (s, 1H), 8.57 (s, 2H), 7.63–7.62 (m, 2H), 7.30–7.28 (m, 2H), 4.04 (s, 2H);

^{13}C NMR ($CDCl_3$, 176 MHz) δ 157.4, 157.0, 143.7, 132.8, 132.7, 129.5, 118.4, 111.2, 36.5;

HRMS (EI): calcd for $C_{12}H_9N_3$ $[M]^+$: 195.0797. Found 195.0794.

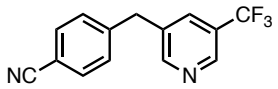


3i: Prepared according to the General Procedure from the corresponding aryl bromide (47 mg, 0.25 mmol, 1.0 equiv.), potassium aryl acetate (60 mg, 0.30 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (3.3 mg, 0.00625 mmol, 0.025 equiv.), and XantPhos (7.3 mg, 0.0125 mmol, 0.050 equiv.) in 1.25 mL DMF, 100 °C. Isolated in 83% yield after purification by silica gel chromatography (2:1 hexane:EtOAc) as a light yellow solid.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 8.35 (s, 2H), 7.63–7.62 (m, 2H), 7.30–7.28 (m, 2H), 4.01 (s, 3H), 3.98 (s, 2H);

^{13}C NMR ($CDCl_3$, 176 MHz) δ 164.8, 159.3, 144.6, 132.6, 129.4, 125.7, 118.6, 110.9, 54.9, 35.5;

HRMS (EI): calcd for $C_{13}H_{11}N_3O$ $[M]^+$: 225.0902. Found 225.0897.



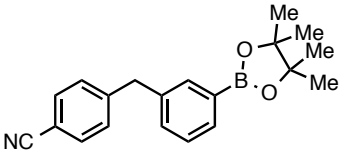
3j: Prepared according to the General Procedure from the corresponding aryl bromide (57 mg, 0.25 mmol, 1.0 equiv.), potassium aryl acetate (60 mg, 0.30 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (3.3 mg, 0.00625 mmol, 0.025 equiv.), and XantPhos (7.3 mg, 0.0125 mmol, 0.050 equiv.) in 1.25 mL DMF, 100 °C. Isolated in 76% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a colourless oil;

^{1}H NMR ($CDCl_3$, 498 MHz) δ 8.78 (s, 1H), 8.66 (s, 1H), 7.68 (s, 1H), 7.63 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.3 Hz, 2H), 4.12 (s, 2H);

^{13}C NMR ($CDCl_3$, 176 MHz) δ 153.3, 145.1 (q, *J* = 3.5 Hz), 143.9, 135.1, 133.1 (q, *J* = 3.5 Hz), 132.8, 129.6, 126.7 (q, *J* = 29.8 Hz), 123.3 (q, *J* = 271.3 Hz), 118.5, 111.2, 38.7;

^{19}F NMR ($CDCl_3$, 469 MHz) δ -62.4;

HRMS (EI): calcd for $C_{14}H_9N_2F_3$ [M]$^+$: 262.0718. Found 262.0714.

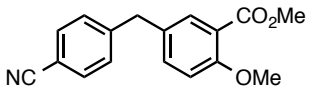


3k: Prepared according to the General Procedure from the corresponding aryl bromide (71 mg, 0.25 mmol, 1.0 equiv.), potassium aryl acetate (60 mg, 0.30 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (3.2 mg, 0.00625 mmol, 0.025 equiv.), and XantPhos (7.2 mg, 0.0125 mmol, 0.050 equiv.) in 1.25 mL DMF, 100 °C. 66% yield by ^{1}H NMR using durene as internal standard and isolated in 28% yield after purification by silica gel chromatography (2% to 18% EtOAc in Hexane) as a colourless oil.

^{1}H NMR ($CDCl_3$, 700 MHz) δ 7.69 (d, *J* = 7.3 Hz, 1H), 7.65 (s, 1H), 7.56 (m, 2H), 7.32 (t, J = 7.4 Hz, 1H), 7.27 (m, 2H), 7.23 (m, 1H), 4.03 (s, 2H), 1.34 (s, 12H);

^{13}C NMR ($CDCl_3$, 176 MHz) δ 146.8, 138.5, 135.3, 133.2, 132.3, 131.9, 129.6, 128.2, 119.0, 110.0, 83.9, 41.9, 24.9. The carbon directly attached to boron atom was not detected.

HRMS (EI): calcd for $C_{20}H_{22}NO_2B$ [M]$^+$: 319.1744. Found 319.1745.

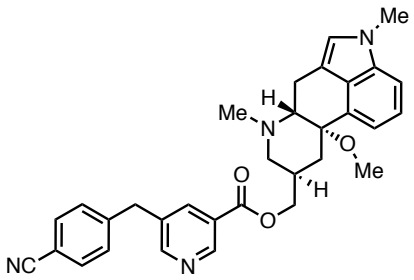


3l: Prepared according to the General Procedure from the corresponding aryl bromide (61 mg, 0.25 mmol, 1.0 equiv.), potassium aryl acetate (60 mg, 0.30 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (3.2 mg, 0.00625 mmol, 0.025 equiv.), and XantPhos (7.2 mg, 0.0125 mmol, 0.050 equiv.) in 1.25 mL DMF, 100 °C. Isolated in 58% yield after purification by silica gel chromatography (12:1 to 1:1 hexane/EtOAc) as a pale yellow oil.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.74 (m, 1H), 7.58 (m, 2H), 7.27 (m, 2H), 6.77 (m 1H), 6.74 (m, 1H), 4.04 (s, 2H), 3.87 (s, 3H), 3.85 (s, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 166.4, 159.5, 145.6, 145.4, 132.4, 132.2, 129.6, 120.8, 118.8, 118.5, 112.7, 111.5, 56.0, 52.0, 42.1;

HRMS (EI): calcd for $C_{17}H_{15}NO_3$ $[M]^+$: 281.1052. Found 281.1053.

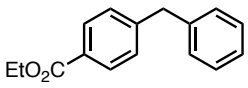


3m: Prepared according to the General Procedure from the corresponding aryl bromide (97 mg, 0.20 mmol, 1.0 equiv.), potassium aryl acetate (48 mg, 0.24 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (2.6 mg, 0.005 mmol, 0.025 equiv.), and XantPhos (5.8 mg, 0.01 mmol, 0.050 equiv.) in 1.0 mL DMF, 100 °C. Isolated in 87% yield after purification by silica gel chromatography (10:1 DCM:MeOH) as a light yellow solid.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 9.13 (s, 1H), 8.65 (s, 1H), 8.07 (s, 1H), 8.07-8.06 (m, 2H), 7.29–7.18 (m, 4H), 7.03 (d, J = 7.0 Hz, 1H), 6.80 (s, 1H), 4.40–4.37 (m, 1H), 4.29–4.25 (m, 1H), 4.09 (s, 2H), 3.77 (s, 3H), 3.23–3.17 (m, 2H), 3.05–2.95 (m, 5H), 2.63–2.59 (m, 1H), 2.46 (s, 3H), 2.35–2.31 (m, 1H), 2.06 (t, J = 11.8 Hz, 1H), 1.36 (t, J = 13.3 Hz, 1H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 165.0, 153.8, 149.2, 144.5, 137.2, 135.1, 135.0, 132.7, 129.7, 129.6, 126.3, 126.2, 123.3, 121.4, 118.6, 114.9, 110.9, 110.3, 109.0, 73.6, 70.1, 68.2, 60.7, 49.5, 43.9, 38.8, 32.8, 31.6, 30.2, 22.4;

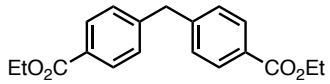
HRMS (ESI): calcd for $C_{32}H_{33}N_4O_3$ $[M+H]^+$: 521.2547. Found 521.2545.



4a: Prepared according to the General Procedure from the corresponding aryl bromide (79 mg, 0.50 mmol, 1.0 equiv.), potassium aryl acetate (148 mg, 0.60 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (6.5 mg, 0.0125 mmol, 0.025 equiv.), and XantPhos (14.5 mg, 0.025 mmol, 0.050 equiv.) in 2.5 mL DMF, 110 °C. Isolated in 77% yield after purification by silica gel chromatography (30:1 hexane:EtOAc) as a colourless oil. Spectroscopic data agreed with that reported.[9]

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.99 (m, 2H), 7.33–7.18 (m, 7H), 4.38 (q, *J* = 7.5 Hz, 2H), 4.05 (s, 2H), 1.40 (t, *J* = 7.5 Hz, 3H);

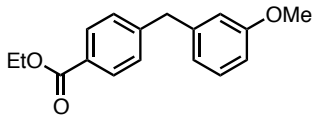
^{13}C NMR ($CDCl_3$, 126 MHz) δ 166.6, 146.4, 140.2, 129.8, 128.9, 128.8, 128.6, 128.4, 126.4, 60.8, 41.9, 14.4.



4b: Prepared according to the General Procedure from the corresponding aryl bromide (115 mg, 0.50 mmol, 1.0 equiv.), potassium aryl acetate (148 mg, 0.60 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (6.5 mg, 0.0125 mmol, 0.025 equiv.), and XantPhos (14.5 mg, 0.025 mmol, 0.050 equiv.) in 2.5 mL DMF, 110 °C. Isolated in 71% yield after purification by silica gel chromatography (30:1 hexane:EtOAc) as a colourless oil. Spectroscopic data agreed with that reported.[10]

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.98–7.96 (m, 4H), 7.25–7.23 (m, 4H), 4.36 (q, *J* = 7.0 Hz, 4H), 4.08 (s, 2H), 1.38 (t, *J* = 7.2 Hz, 6H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 166.4, 145.4, 129.9, 128.9, 128.8, 60.8, 41.8, 14.3.

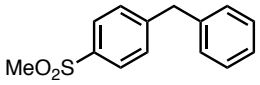


4c: Prepared according to the General Procedure from the corresponding aryl bromide (37 mg, 0.20 mmol, 1.0 equiv.), potassium aryl acetate (59 mg, 0.24 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (2.6 mg, 0.005 mmol, 0.025 equiv.), and XantPhos (5.8 mg, 0.01 mmol, 0.05 equiv.), 110°C. Isolated in 56% yield after purification by silica gel chromatography (100:1 to 10:1 hexane:EtOAc) as a colorless oil.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.98 - 7.95 (m, 2H), 7.27–7.24 (m, 2H), 7.21 (t, *J* = 7.8 Hz, 1H), 6.78–6.75 (m, 2H), 7.71 (m, 1H), 4.36 (q, *J* = 7.2 Hz, 2H), 4.00 (s, 2H), 3.77 (s, 3H), 1.38 (t, *J* = 7.2 Hz, 3H);

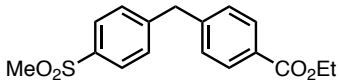
^{13}C NMR ($CDCl_3$, 126 MHz) δ 166.7, 160.0, 146.3, 141.9, 129.9, 129.7, 129.0, 128.7, 121.5, 115.0, 111.7, 61.0, 55.3, 42.1, 14.5;

HRMS (EI): calcd for $C_{17}H_{18}O_3$ $[M]^+$: 270.1256. Found 270.1255.



5a: Prepared according to the General Procedure from the corresponding aryl bromide (68 mg, 0.30 mmol, 1.0 equiv.), potassium aryl acetate (91 mg, 0.36 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (3.9 mg, 0.0075 mmol, 0.025 equiv.), and XantPhos (8.7 mg, 0.015 mmol, 0.050 equiv.) in 1.5 mL DMF, 90 °C. 67% yield by ^{1}H NMR using durene as internal standard. Spectroscopic data agreed with that reported.[11]

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.85 (m, 2H), 7.38 (d, *J* = 8.3 Hz, 2H), 7.32 (m, 2H), 7.23 (m, 1H), 7.18 (d, *J* = 6.8 Hz, 2H), 4.07 (s, 2H), 3.03 (t, *J* = 8.0 Hz, 3H).

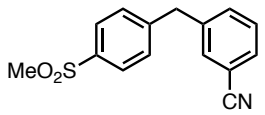


5b: Prepared according to the General Procedure from the corresponding aryl bromide (69 mg, 0.30 mmol, 1.0 equiv.), potassium aryl acetate (91 mg, 0.36 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (3.9 mg, 0.0075 mmol, 0.025 equiv.), and XantPhos (8.7 mg, 0.015 mmol, 0.050 equiv.) in 1.5 mL DMF, 90 °C. Isolated in 83% yield after purification by silica gel chromatography (4:1 to 2:1 hexane:EtOAc) as a light yellow solid.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 8.01 (m, 2H), 7.88 (m, 2H), 7.38 (d, *J* = 8.8 Hz, 2H), 7.27 (d, *J* = 8.8 Hz, 2H), 4.39 (q, *J* = 7.0 Hz, 2H), 4.14 (s, 2H), 3.05 (s, 3H), 1.40 (t, *J* = 7.5 Hz, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 166.3, 146.7, 144.4, 138.7, 130.1, 129.8, 129.0, 128.9, 127.8, 60.9, 44.6, 41.7, 14.3;

HRMS (EI): calcd for $C_{17}H_{18}O_4S$ $[M]^+$: 318.0926. Found 318.0924.

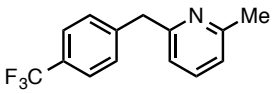


5c: Prepared according to the General Procedure from the corresponding aryl bromide (55 mg, 0.30 mmol, 1.0 equiv.), potassium aryl acetate (91 mg, 0.36 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (3.9 mg, 0.0075 mmol, 0.025 equiv.), and XantPhos (8.7 mg, 0.015 mmol, 0.050 equiv.) in 1.5 mL DMF, 90 °C. Isolated in 67% yield after purification by silica gel chromatography (2:1 hexane:EtOAc) as a white solid.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.93-7.91 (m, 2H), 7.58-7.56 (m, 1H), 7.47-7.43 (m, 3H), 7.39-7.37 (m, 2H), 4.13 (s, 2H), 3.07 (s, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 145.8, 140.9, 139.1, 133.4, 132.5, 130.5, 129.8, 129.6, 127.9, 118.6, 112.9, 44.6, 41.2;

HRMS (EI): calcd for $C_{15}H_{13}O_2SN$ $[M]^+$: 271.0667. Found 271.0667.

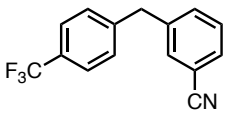


6a: Prepared according to the General Procedure from the corresponding aryl bromide (69 mg, 0.40 mmol, 2.0 equiv.), potassium aryl acetate (48 mg, 0.20 mmol, 1.0 equiv.), [Pd(cinnamyl)Cl]$_2$ (2.6 mg, 0.005 mmol, 0.025 equiv.), and DEA-XantPhos (5.6 mg, 0.01 mmol, 0.050 equiv.) in 1.0 mL DMF, 125 °C. Isolated in 52% yield after purification by silica gel chromatography (4:1 to 2:1 hexane:EtOAc) as a colourless oil. Spectroscopic data agreed with that reported.[12]

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.54 (d, *J* = 8.4 Hz, 2H), 7.47 (t, *J* = 7.7 Hz, 1H), 7.37 (d, *J* = 8.4 Hz, 2H), 7.01 (d, *J* = 8.0 Hz, 1H), 6.87 (d, *J* = 8.0 Hz, 1H), 4.17 (s, 2H), 2.55 (s, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 159.2, 158.3, 143.8, 136.9, 129.4, 128.7 (q, *J* = 31.3 Hz), 125.4 (q, *J* = 3.8 Hz), 124.3 (q, *J* = 271.3 Hz), 121.1, 120.1, 44.5, 24.6;

^{19}F NMR ($CDCl_3$, 469 MHz) δ -62.4.



6b: Prepared according to the General Procedure from the corresponding aryl bromide (91 mg, 0.50 mmol, 1.0 equiv.), potassium aryl acetate (145 mg, 0.60 mmol, 1.2 equiv.),

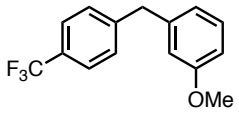
[Pd(cinnamyl)Cl]$_2$ (6.5 mg, 0.0125 mmol, 0.025 equiv.), and DEA-XantPhos (14 mg, 0.025 mmol, 0.050 equiv.) in 2.5 mL DMF, 125 °C. Isolated in 60% yield after purification by silica gel chromatography (10:1 hexane:EtOAc) as a yellow solid.

^{1}H NMR ($CDCl_3$, 498 MHz) δ 7.59 (d, *J* = 8.0 Hz, 2H), 7.55–7.53 (m, 1H), 7.48–7.43 (m, 1H), 7.43–7.42 (m, 2H), 7.29–7.28 (m, 2H), 4.08 (s, 2H);

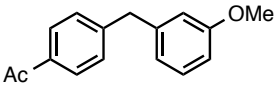
^{13}C NMR ($CDCl_3$, 126 MHz) δ 143.4, 141.5, 133.4, 132.4, 130.3, 129.5, 129.2, 129.1 (q, *J* = 32.5 Hz), 125.7 (q, *J* = 3.8 Hz), 124.1 (q, *J* = 272.8 Hz), 118.7, 112.8, 41.1;

^{19}F NMR ($CDCl_3$, 469MHz) δ -62.5;

HRMS (EI): calcd for $C_{15}H_{10}NF_3$ $[M]^+$: 261.0765. Found 261.0767.



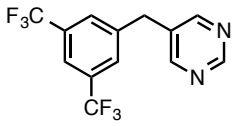
6c: Prepared according to the General Procedure from the corresponding aryl bromide (18.7 mg, 0.10 mmol, 1.0 equiv.), potassium aryl acetate (29 mg, 0.12 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (1.3 mg, 0.0025 mmol, 0.025 equiv.), and DEA-XantPhos (2.8 mg, 0.005 mmol, 0.050 equiv.) in 0.5 mL DMF, 125 °C. 11% yield by ^{1}H NMR using durene as internal standard. Spectroscopic data agreed with that reported.[13]



7a: Prepared according to the General Procedure from the corresponding aryl bromide (37 mg, 0.2 mmol, 1.0 equiv.), potassium aryl acetate (52 mg, 0.24 mmol, 1.2 equiv.), [Pd(cinnamyl)Cl]$_2$ (2.6 mg, 0.005 mmol, 0.025 equiv.), and XantPhos (5.8 mg, 0.010 mmol, 0.050 equiv.) in 1.0 mL DMF, 110 °C. Isolated in 55% yield after purification by silica gel chromatography (2% to 15% EtOAc in Hexane) as a colourless oil. Spectroscopic data agreed with that reported.[14]

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.88 (m, 2H), 7.28 (m, 2H), 7.21 (t, *J* = 8.0 Hz, 1H), 6.77 (m, 2H), 6.72 (m, 1H), 4.00 (s, 2H), 3.77 (s, 3H), 2.58 (s, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 197.8, 159.8, 146.6, 141.6, 135.3, 129.6, 129.1, 128.6, 121.3, 114.9, 111.6, 55.2, 41.9, 26.6.



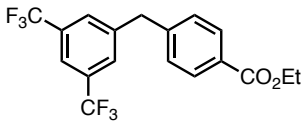
7b: Prepared according to the General Procedure from the corresponding aryl bromide (32 mg, 0.2 mmol, 1.0 equiv.), potassium aryl acetate (74 mg, 0.24 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (2.6 mg, 0.005 mmol, 0.025 equiv.), and DEA-XantPhos (5.6 mg, 0.010 mmol, 0.050 equiv.) in 1.0 mL DMF, 110 °C. Isolated in 64% yield after purification by silica gel chromatography (16% to 100% EtOAc in Hexane) as an orange solid.

^{1}H NMR ($CDCl_3$, 700 MHz) δ 9.16 (s, 1H), 8.61 (s, 2H), 7.80 (s, 1H), 7.64 (s, 2H), 4.12 (s, 2H);

^{13}C NMR ($CDCl_3$, 176 MHz) δ 157.7, 157.0, 140.1, 132.4 (q, J = 34.7 Hz), 132.2, 128.8 (m), 123.0 (q, J = 271.6 Hz), 121.3 (quint, J = 3.63 Hz), 36.1;

^{19}F NMR ($CDCl_3$, 378 MHz) δ -63.0;

HRMS (EI): calcd for $C_{13}H_8N_2F_6$ $[M]^+$: 306.0592. Found 306.0591.



7c: Prepared according to the General Procedure from the corresponding aryl bromide (115 mg, 0.50 mmol, 1 equiv.), potassium aryl acetate (186 mg, 0.60 mmol, 1.2 equiv.), $[Pd(cinnamyl)Cl]_2$ (6.5 mg, 0.0125 mmol, 0.025 equiv.), and DEA-XantPhos (14 mg, 0.025 mmol, 0.050 equiv.) in 2.5 mL DMF, 110 °C. Isolated in 70% yield after purification by silica gel chromatography (12:1 hexane:EtOAc) as a light yellow oil.

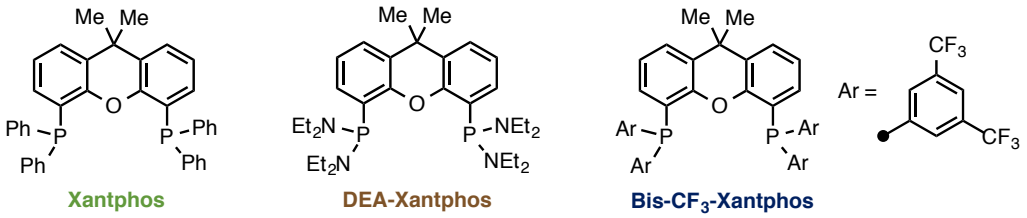
^{1}H NMR ($CDCl_3$, 498 MHz) δ 8.02 (m, 2H), 7.75 (s, 1H), 7.61 (s, 2H), 7.24 (m, 2H), 4.38(q, J = 7.3 Hz, 2H), 4.16 (s, 2H), 1.39 (t, J = 7.3 Hz, 3H).

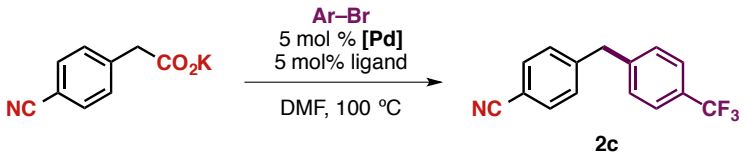
^{13}C NMR ($CDCl_3$, 126 MHz) δ 166.3, 143.8, 142.6, 131.9 (q, J = 32.5 Hz), 130.2, 129.3, 128.9, 128.8, 123.3 (d, J = 271.3 Hz), 120.6 (m), 61.0, 41.4, 14.3.

^{19}F NMR ($CDCl_3$, 469 MHz) δ -62.8.

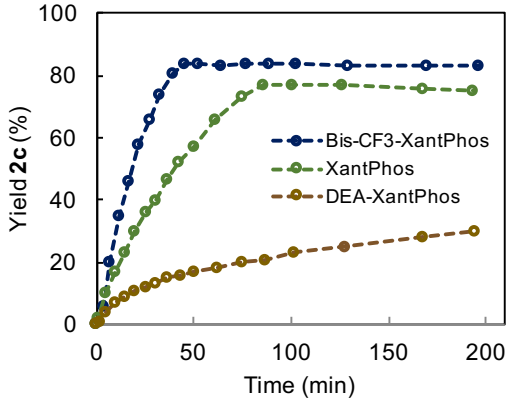
HRMS (EI): calcd for $C_{18}H_{14}O_2F_6$ $[M]^+$: 376.0898. Found 376.0899.

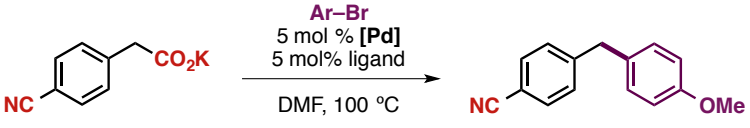
IV. Reaction Kinetics with Different Xantphos Derivatives



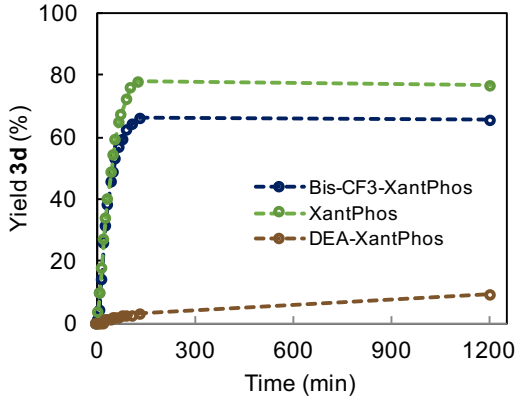


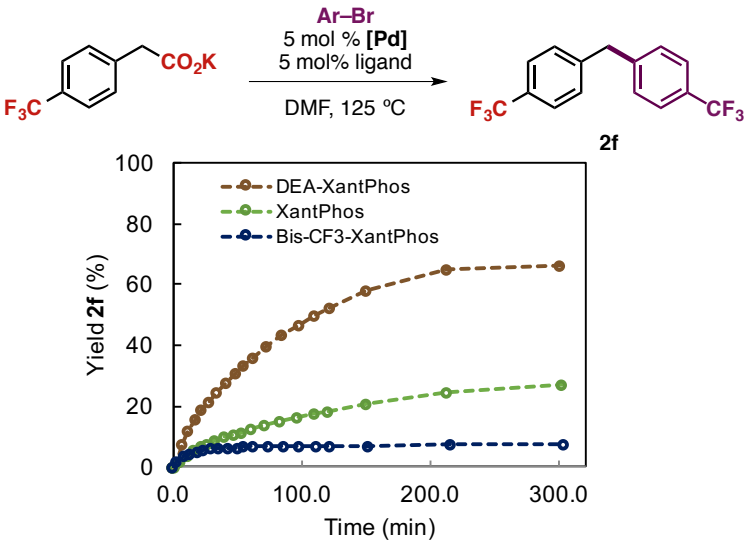
2c



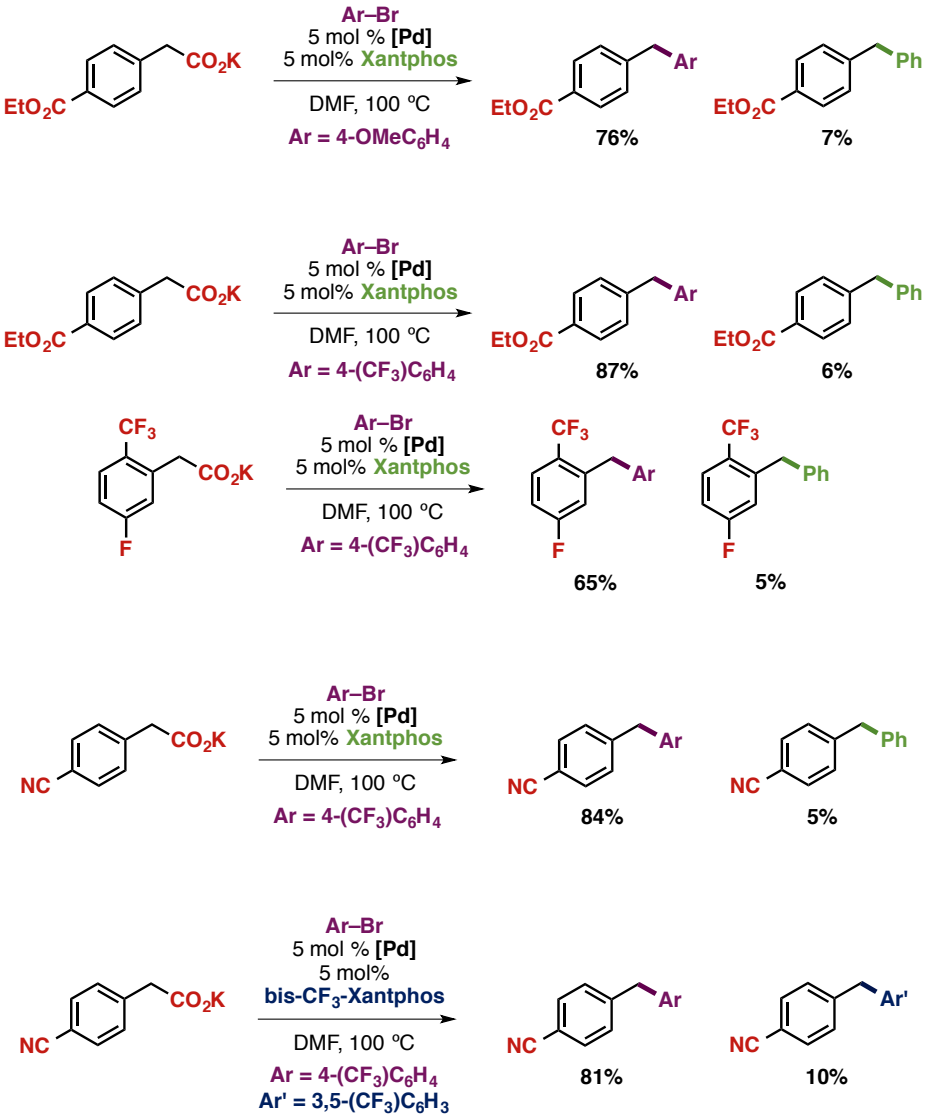


3d




Ar–Br
5 mol % [Pd]
5 mol% ligand
DMF, 125 °C
F3C
CO2K
F3C
CF3
2f
Yield 2f (%)
Time (min)
DEA-XantPhos
XantPhos
Bis-CF3-XantPhos
0
20
40
60
80
100
0.0
100.0
200.0
300.0

V. Diarylmethane Products Arising from Ligand Aryl Exchange



VI. References

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Agilent Technologies
Department of Chemistry, University of Alberta
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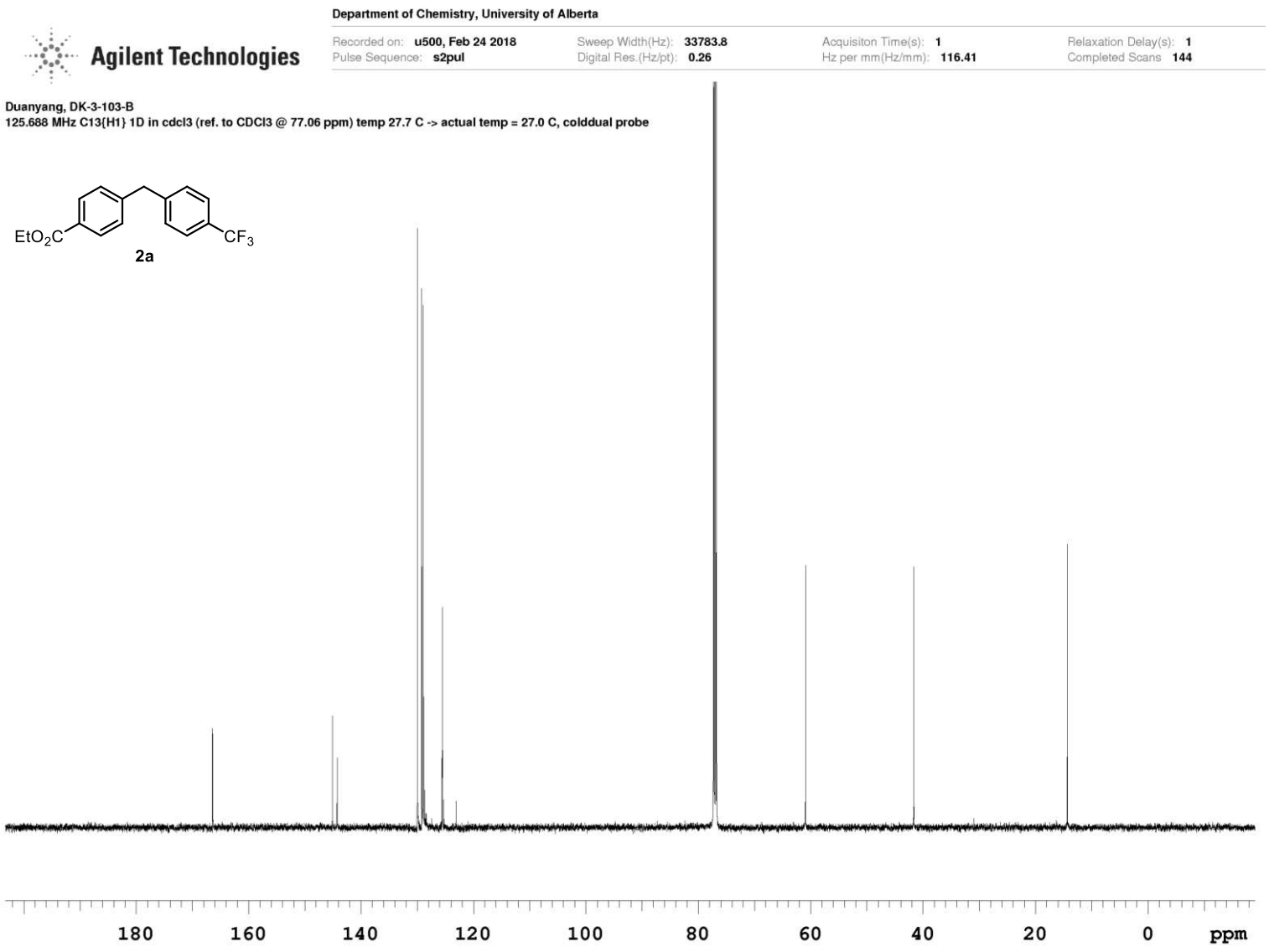
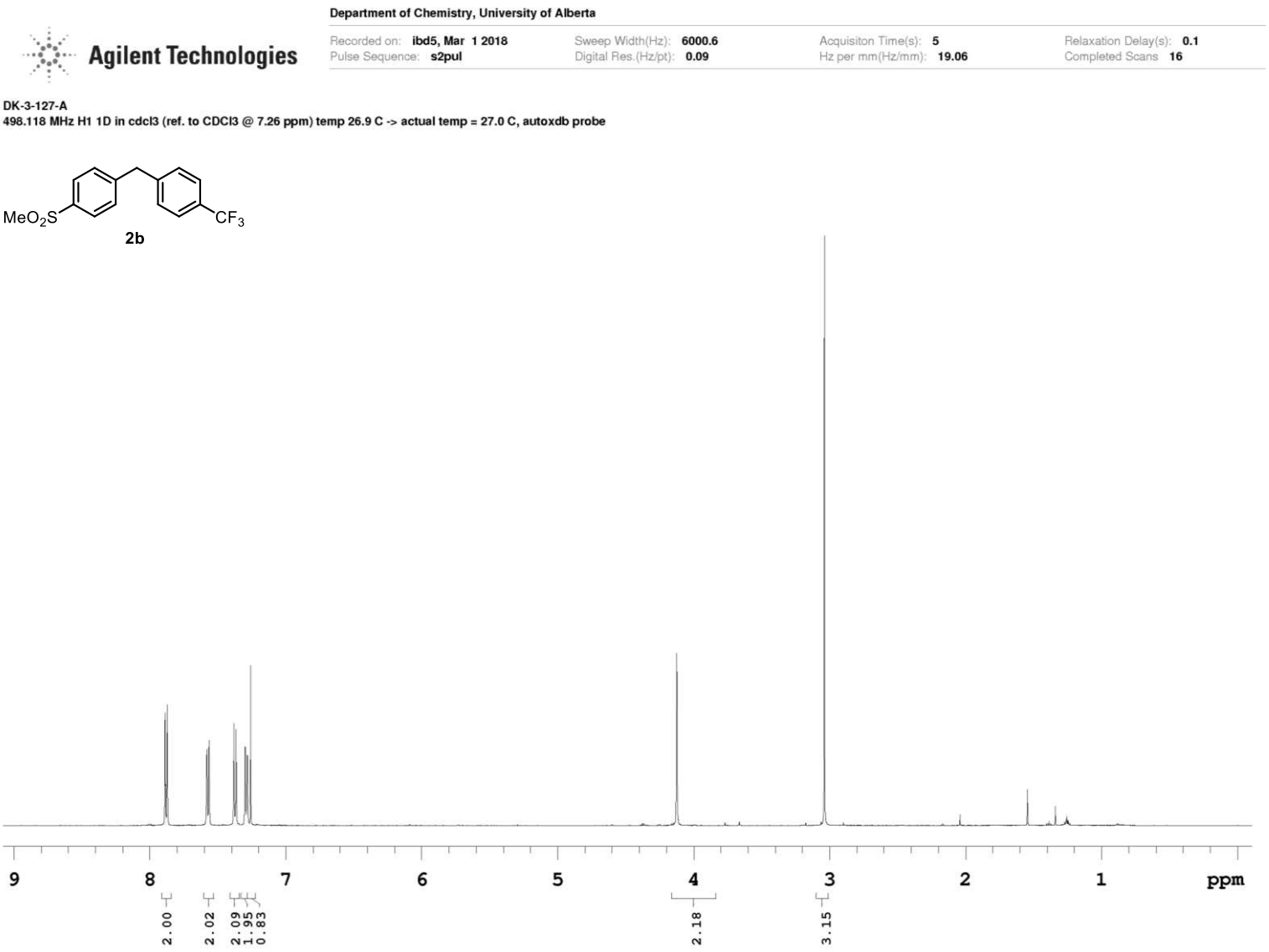
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 19.14
Relaxation Delay(s): 0.1
Completed Scans 16
DK-3-103-B
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
EtO2C
CF3
2a
9
8
7
6
5
4
3
2
1
ppm
2.00
1.97
4.54
2.06
2.04
3.12


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Feb 24 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 116.41
Relaxation Delay(s): 1
Completed Scans 144
Duanyang, DK-3-103-B
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
EtO2C
CF3
2a
180
160
140
120
100
80
60
40
20
0
ppm


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: ibd5, Mar 1 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 6000.6
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 19.06
Relaxation Delay(s): 0.1
Completed Scans 16
DK-3-127-A
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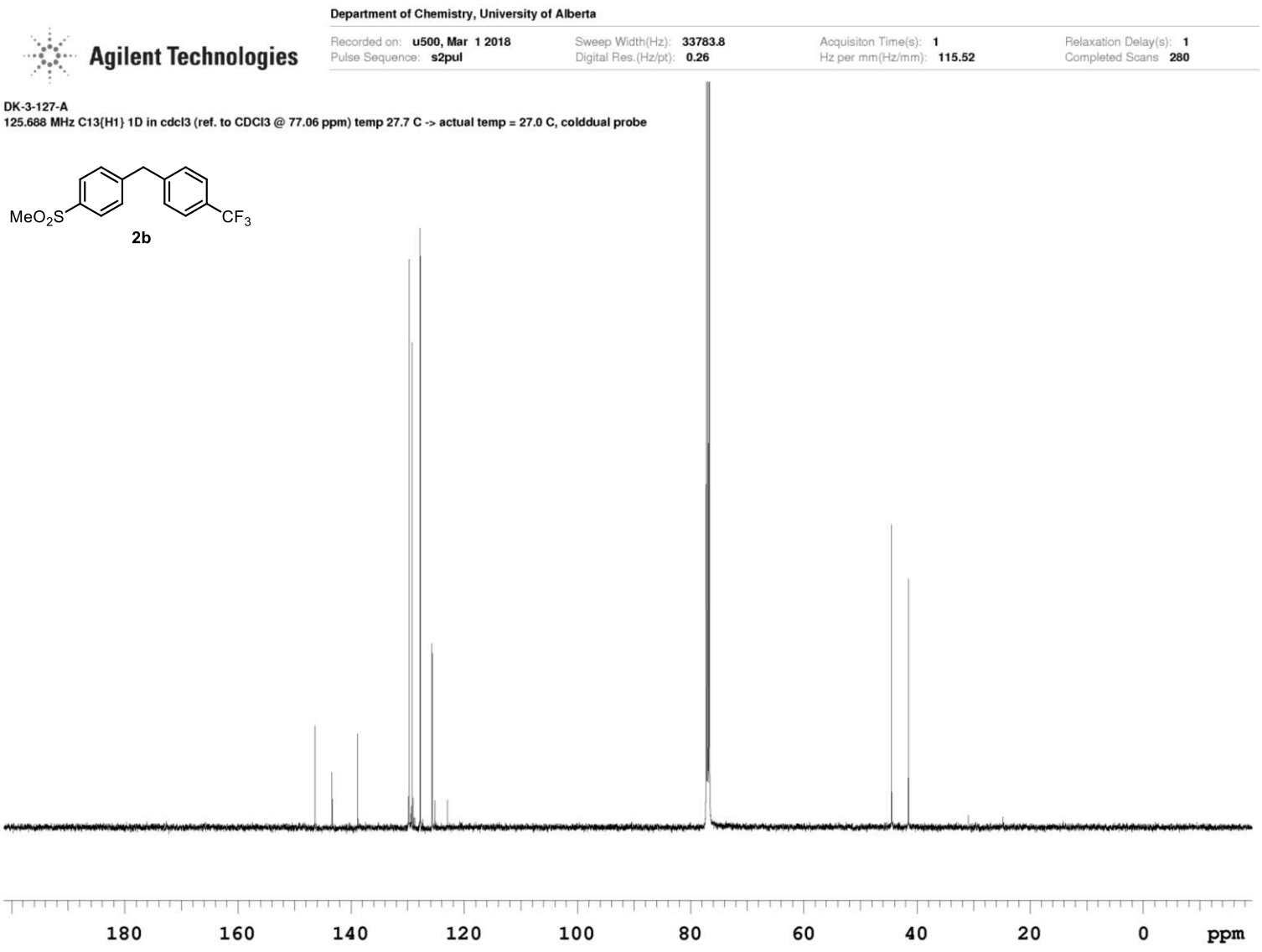
MeO2S
CF3
2b
9
8
7
6
5
4
3
2
1
ppm
2.00
2.02
2.09
1.95
0.83
2.18
3.15


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 1 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 115.52
Relaxation Delay(s): 1
Completed Scans 280
DK-3-127-A
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
MeO2S
CF3
2b
180
160
140
120
100
80
60
40
20
0
ppm

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on:	u500, Feb 25 2018	Sweep Width(Hz):	6009.62	Acquisiton Time(s):	5	Relaxation Delay(s):	0.1
Pulse Sequence:	PRESAT	Digital Res.(Hz/pt):	0.09	Hz per mm(Hz/mm):	20.85	Completed Scans	1

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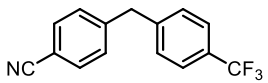
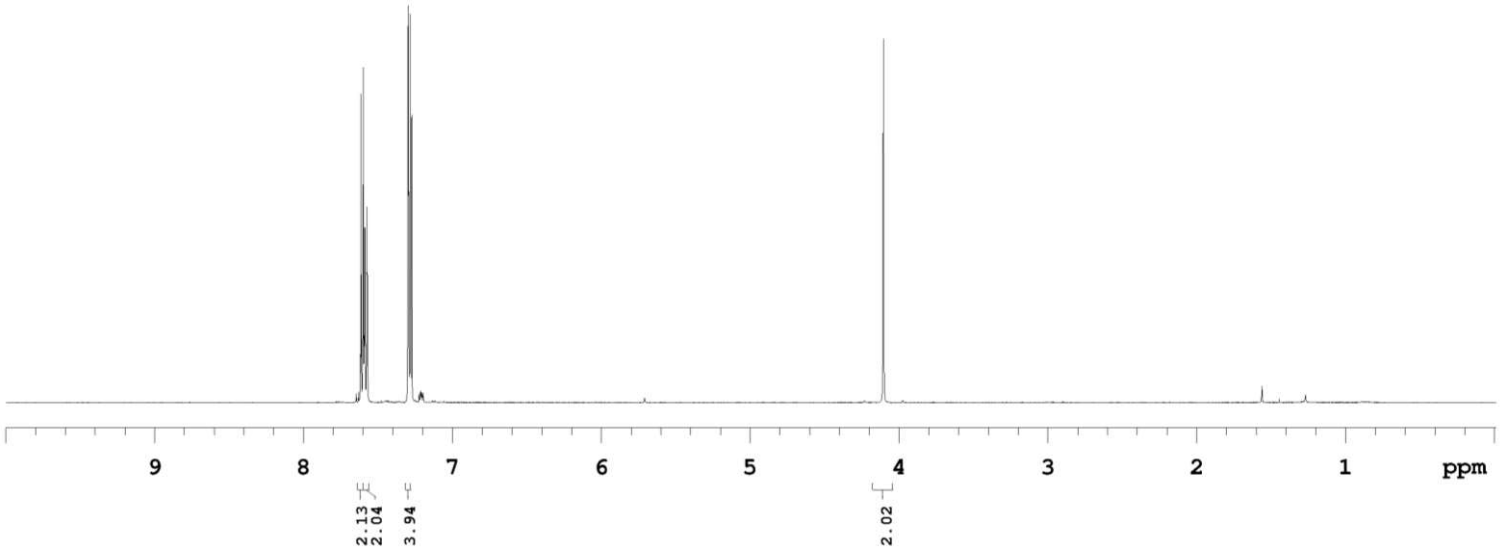
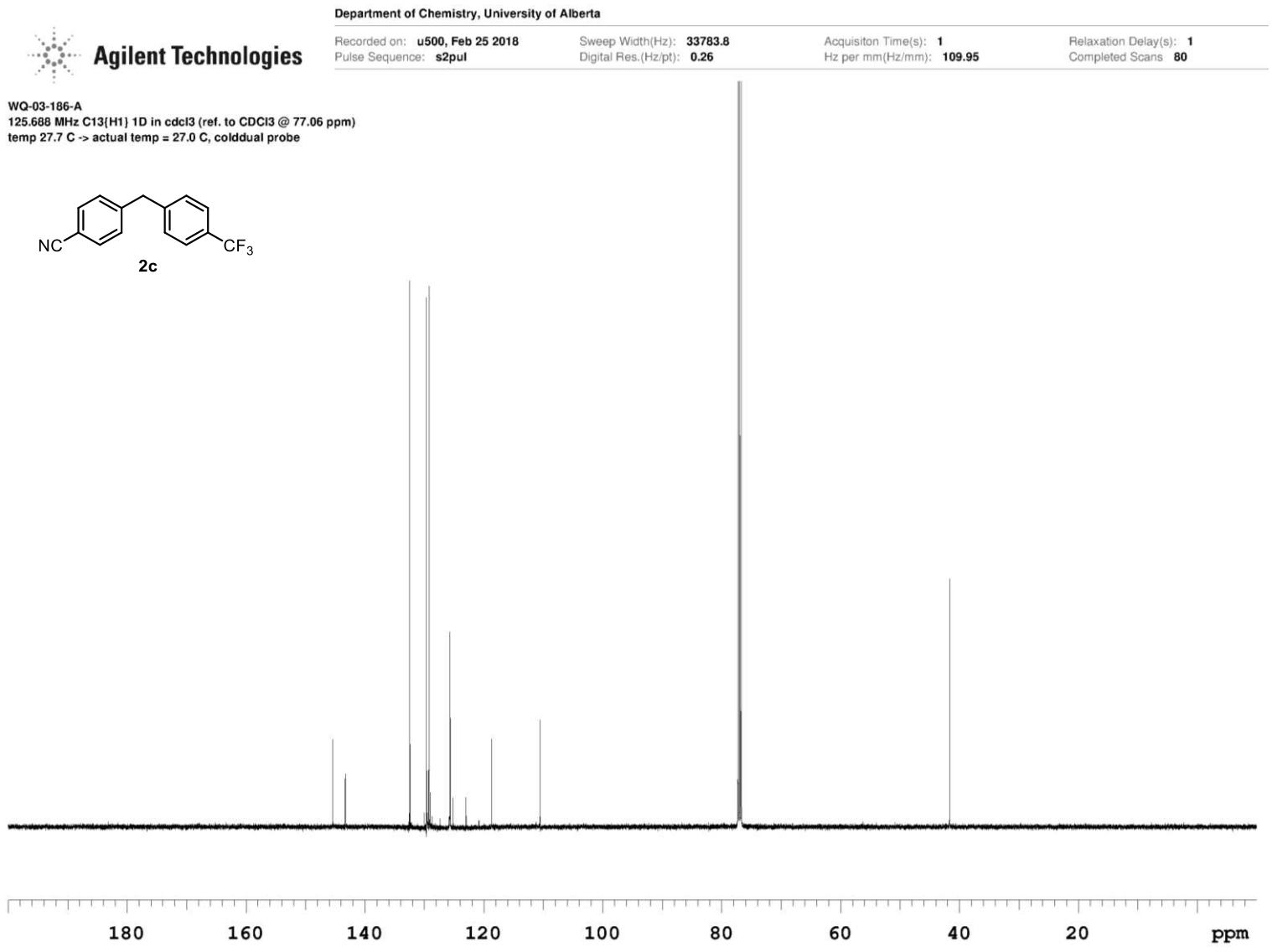
temp 27.7 C -> actual temp = 27.0 C, colddual probe



2c




Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Feb 25 2018
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Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
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Hz per mm(Hz/mm): 109.95
Relaxation Delay(s): 1
Completed Scans 80
WQ-03-186-A
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
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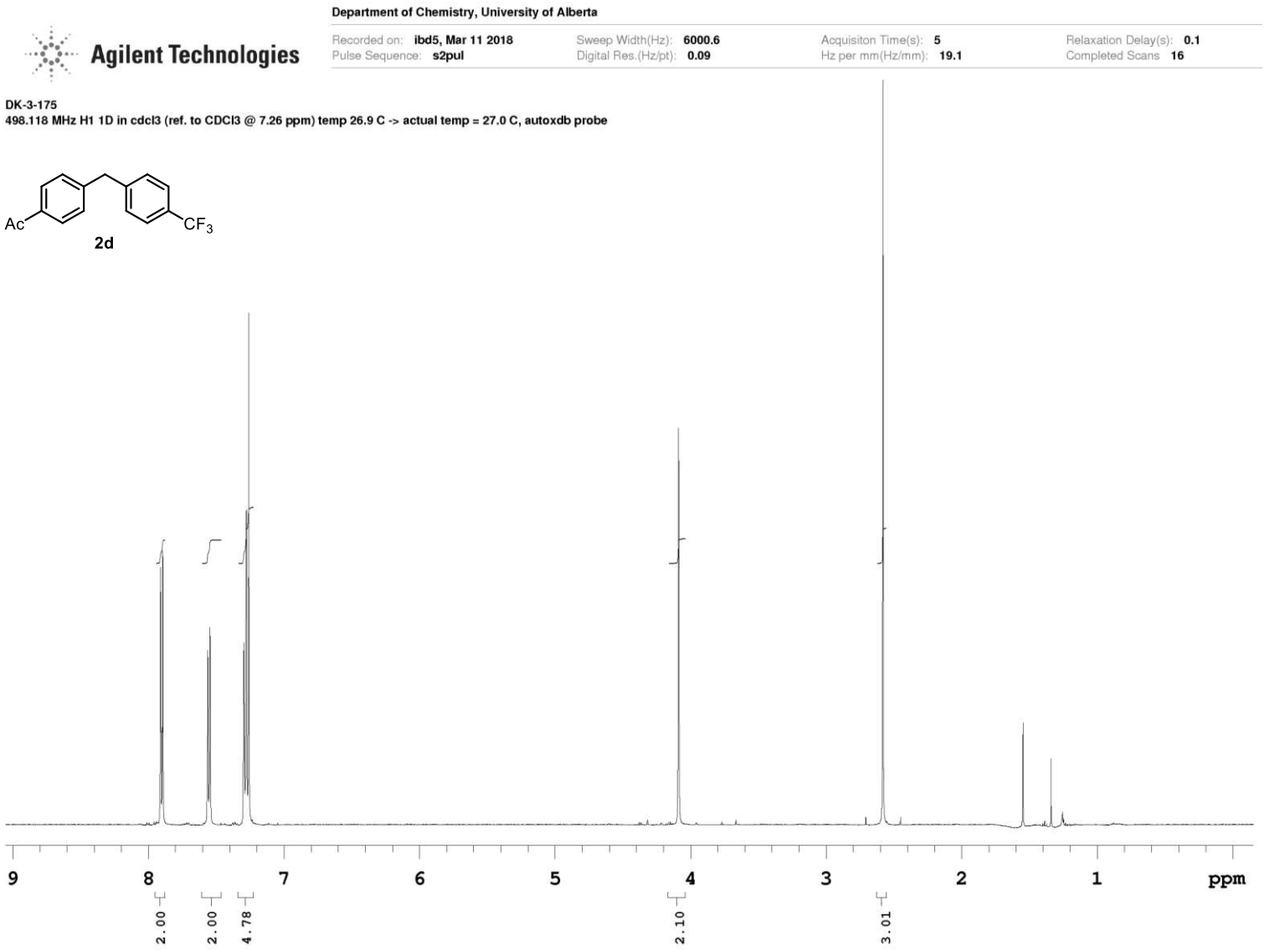
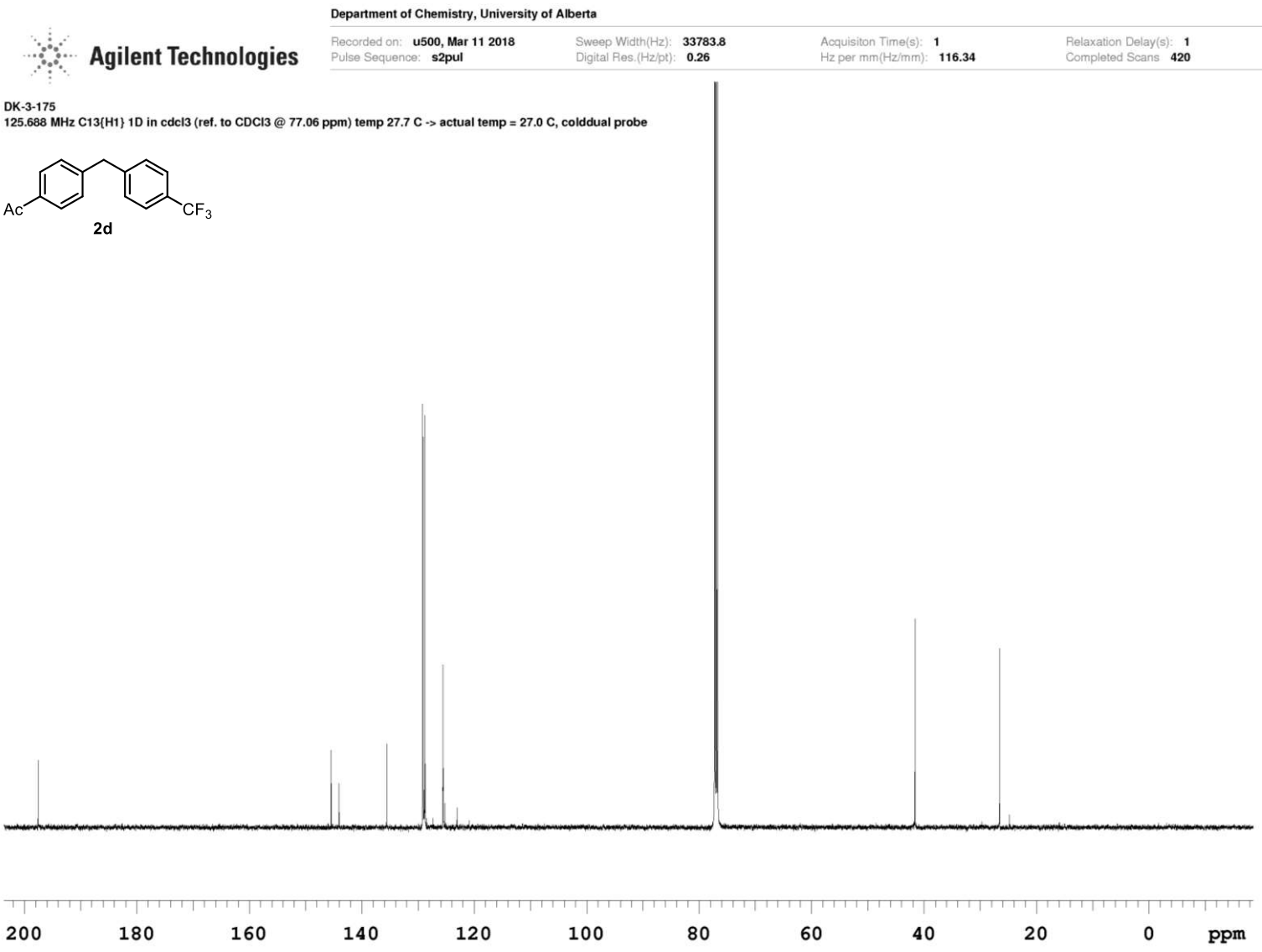
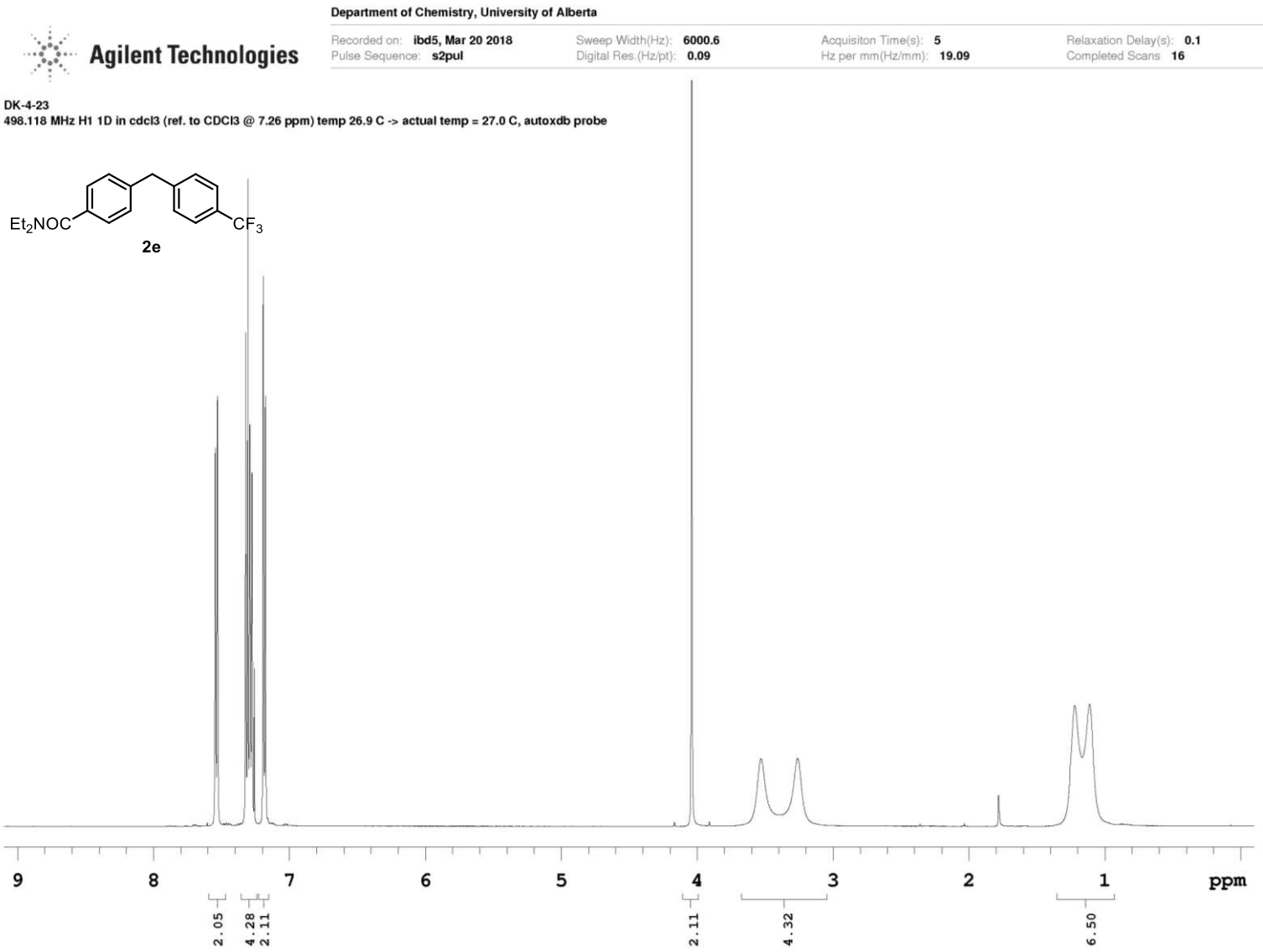
NC
CF3
2c
180
160
140
120
100
80
60
40
20
ppm


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: ibd5, Mar 11 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 6000.6
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 19.1
Relaxation Delay(s): 0.1
Completed Scans 16
DK-3-175
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
Ac
CF3
2d
9
8
7
6
5
4
3
2
1
ppm
2.00
2.00
4.78
2.10
3.01


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 11 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 116.34
Relaxation Delay(s): 1
Completed Scans 420
DK-3-175
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
Ac
CF3
2d
200
180
160
140
120
100
80
60
40
20
0
ppm


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: ibd5, Mar 20 2018
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Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 19.09
Relaxation Delay(s): 0.1
Completed Scans 16
DK-4-23
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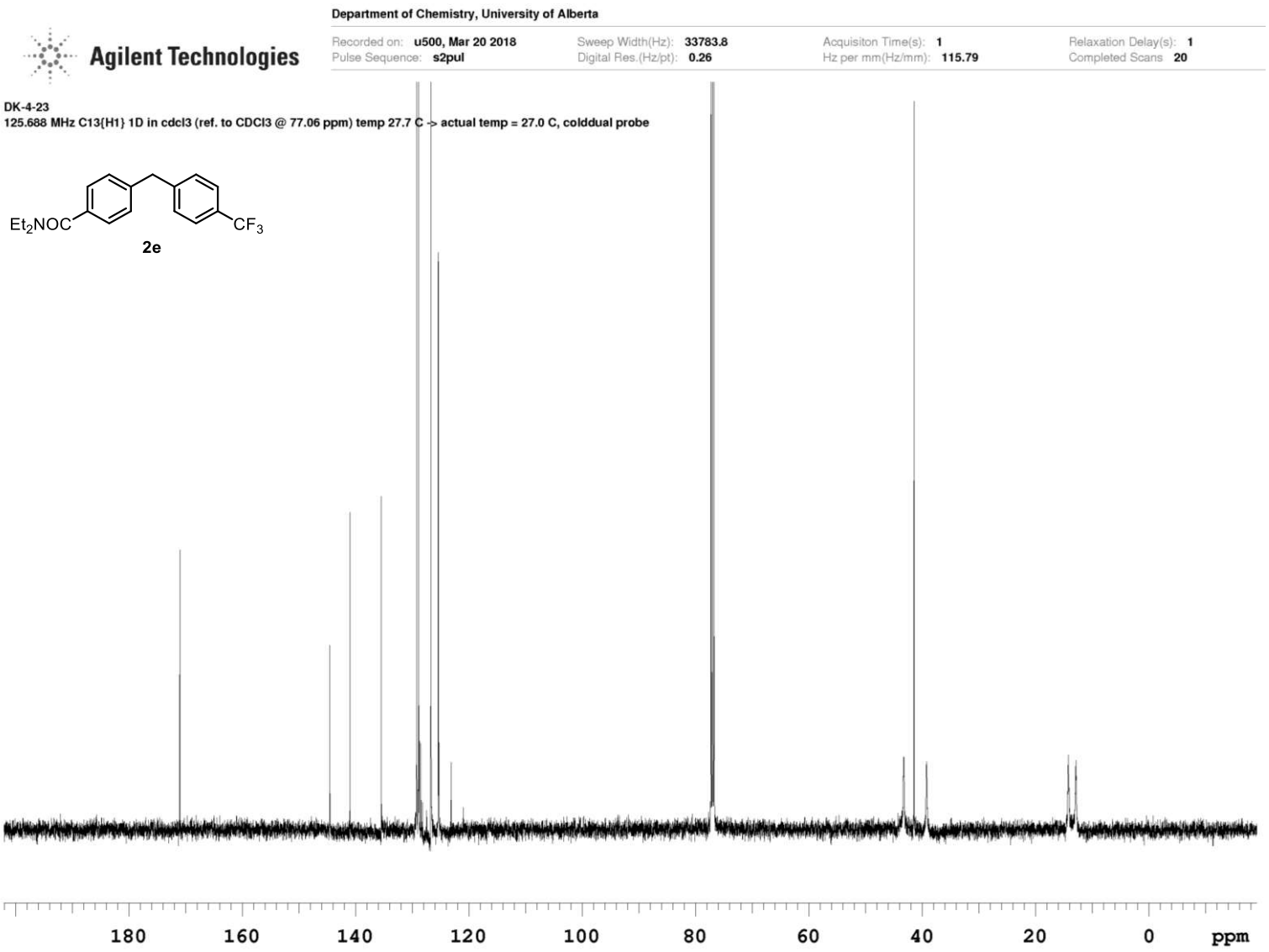
Et2NOC
CF3
2e
9
8
7
6
5
4
3
2
1
ppm
2.05
4.28
2.11
2.11
4.32
6.50


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 20 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 115.79
Relaxation Delay(s): 1
Completed Scans 20
DK-4-23
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
Et2NOC
CF3
2e
180
160
140
120
100
80
60
40
20
0
ppm

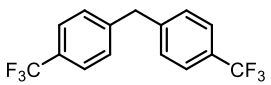
Department of Chemistry, University of Alberta

Agilent Technologies

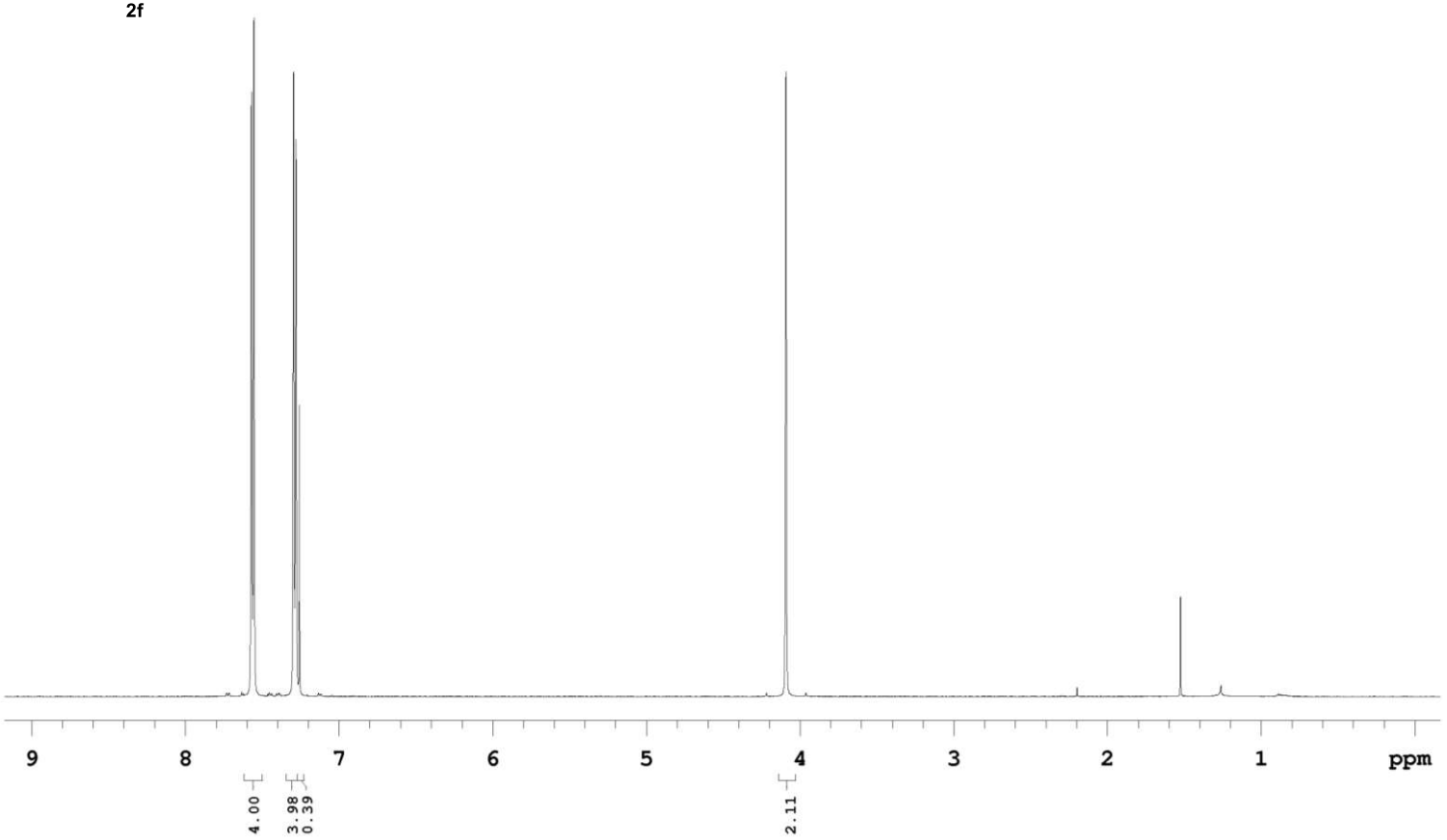
Recorded on: ibd5, Mar 8 2018	Sweep Width(Hz): 6000.6	Acquisiton Time(s): 5	Relaxation Delay(s): 0.1
Pulse Sequence: s2pul	Digital Res.(Hz/pt): 0.09	Hz per mm(Hz/mm): 19.39	Completed Scans 16

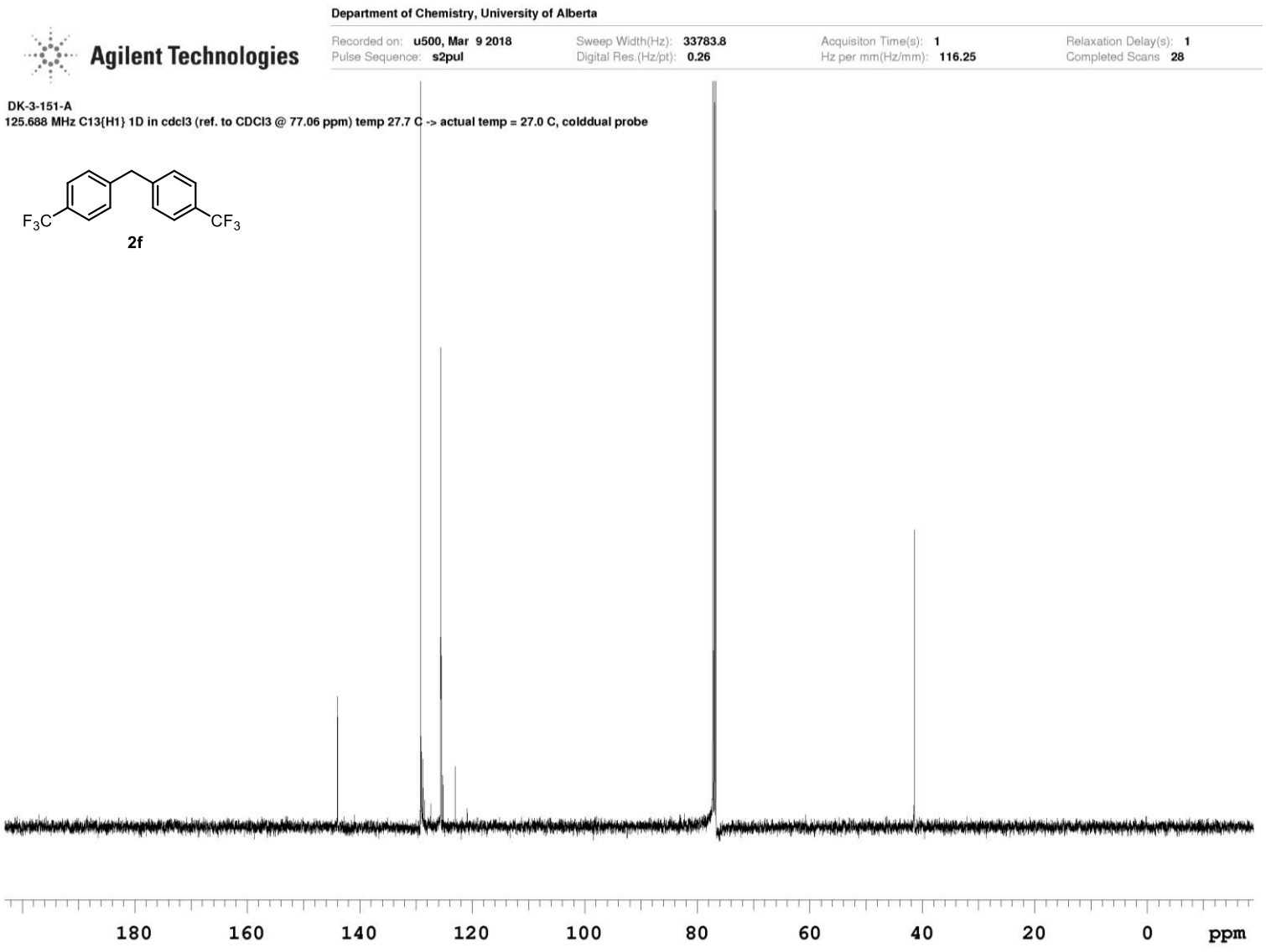
DK-3-151-A

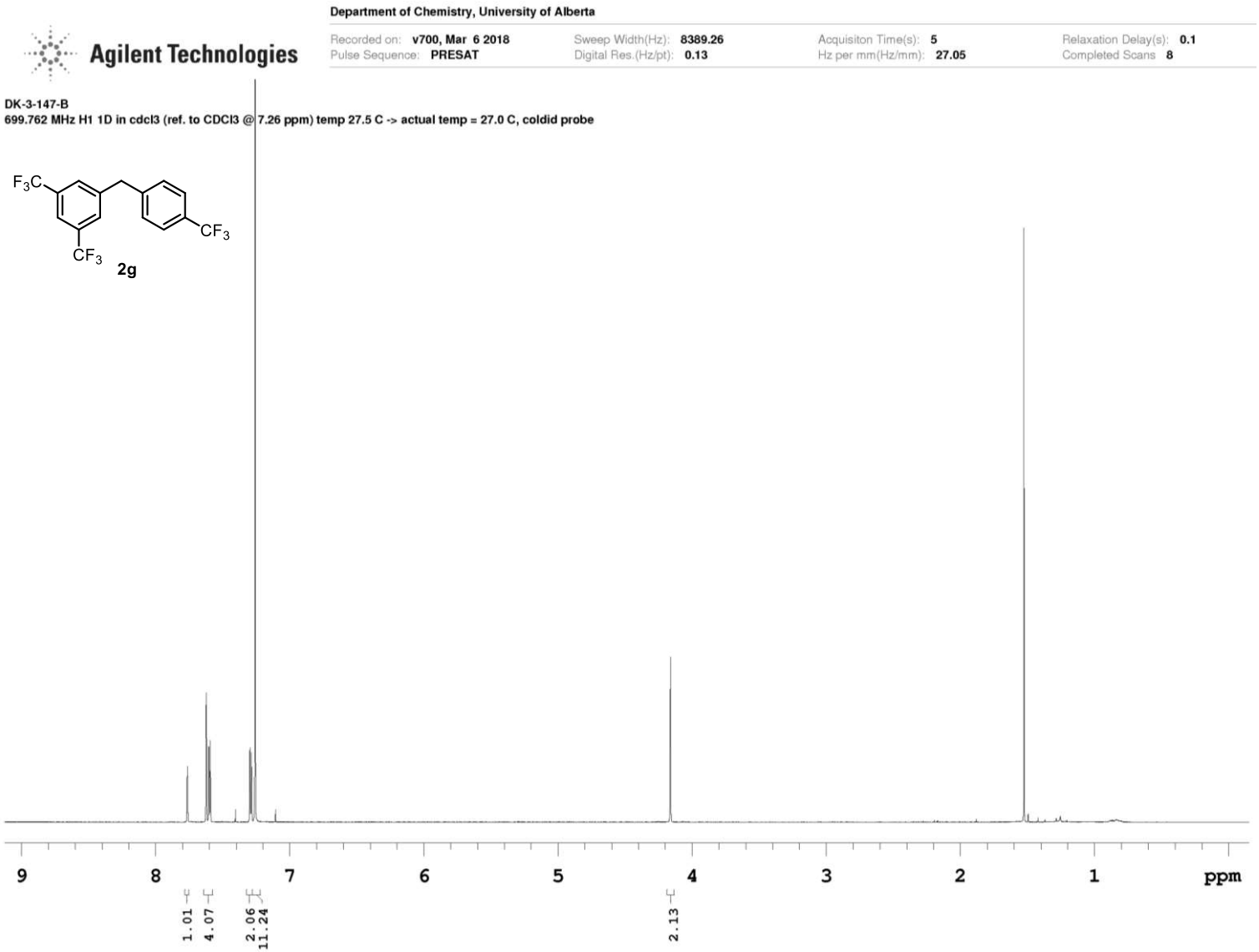
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe

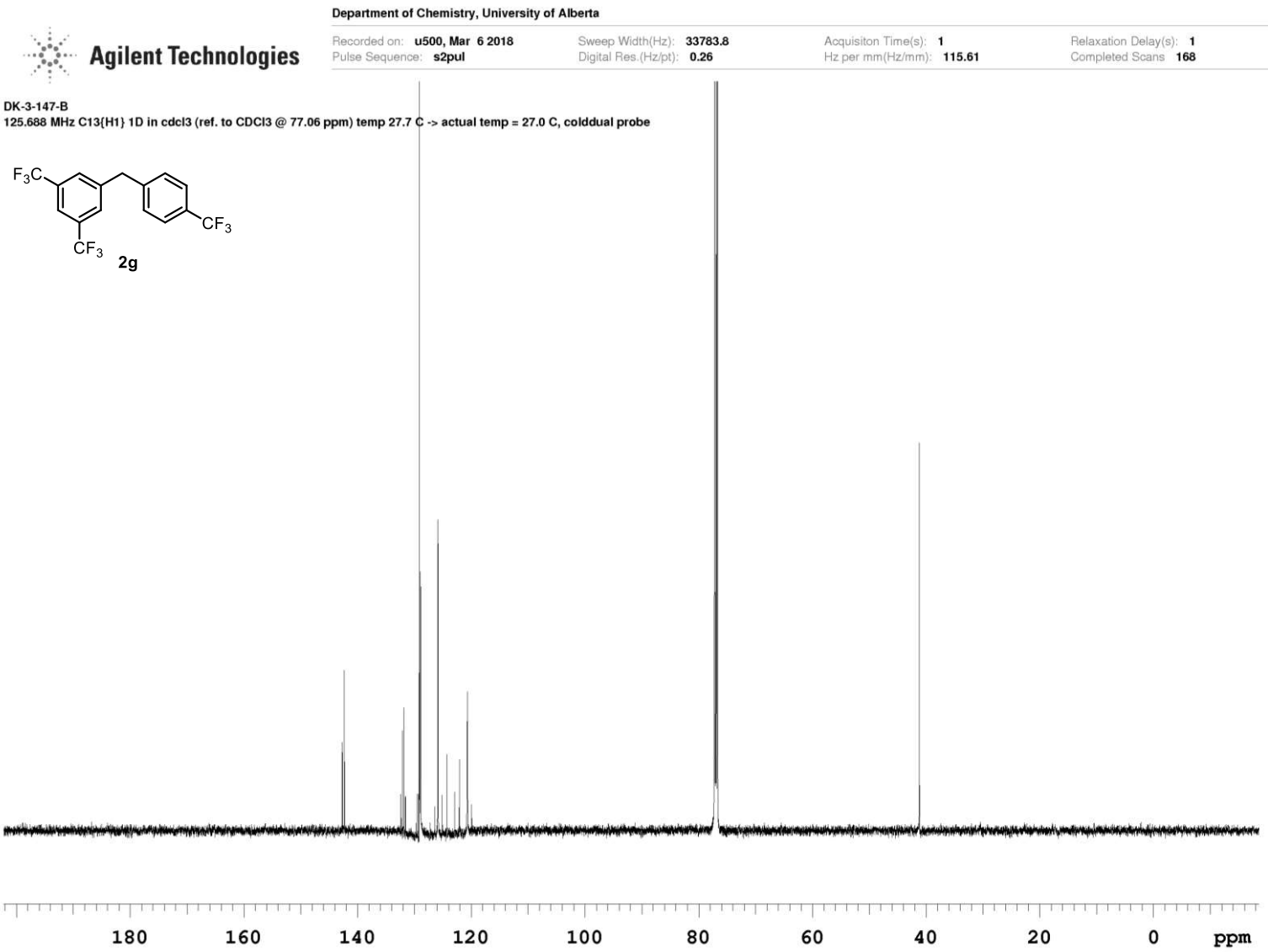


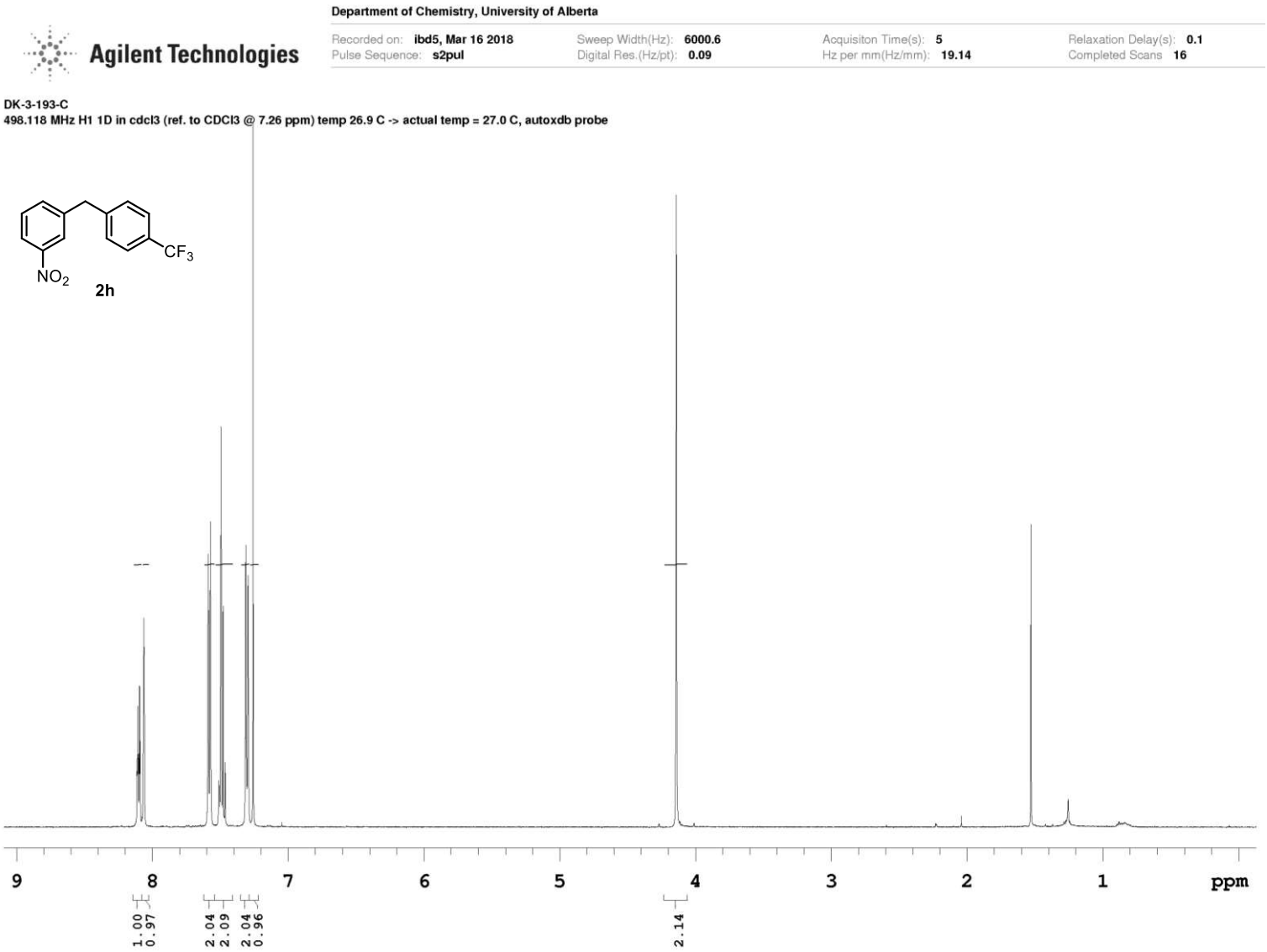
2f

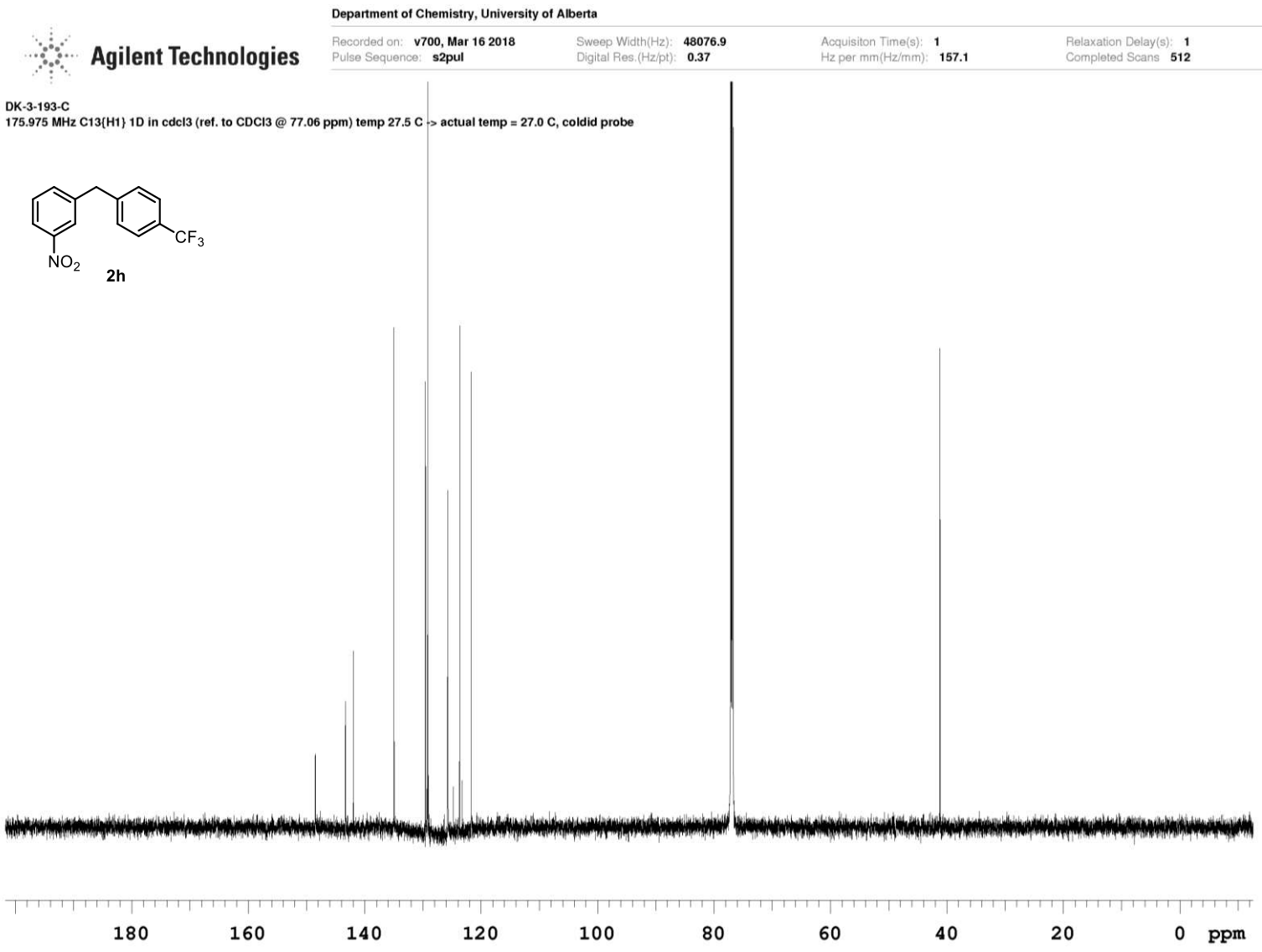



Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 9 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 116.25
Relaxation Delay(s): 1
Completed Scans 28
DK-3-151-A
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
F3C
CF3
2f
180
160
140
120
100
80
60
40
20
0
ppm


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: v700, Mar 6 2018
Pulse Sequence: PRESAT
Sweep Width(Hz): 8389.26
Digital Res.(Hz/pt): 0.13
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 27.05
Relaxation Delay(s): 0.1
Completed Scans 8
DK-3-147-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
F3C
CF3
CF3
2g
9
8
7
6
5
4
3
2
1
ppm
1.01
4.07
2.06
11.24
2.13


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 6 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 115.61
Relaxation Delay(s): 1
Completed Scans 168
DK-3-147-B
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
F3C
CF3
CF3
2g
180
160
140
120
100
80
60
40
20
0
ppm


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: ibd5, Mar 16 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 6000.6
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 19.14
Relaxation Delay(s): 0.1
Completed Scans 16
DK-3-193-C
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
NO2
CF3
2h
9
8
7
6
5
4
3
2
1
ppm
1.00
0.97
2.04
2.09
2.04
0.96
2.14

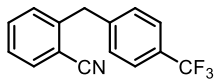

Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: v700, Mar 16 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 48076.9
Digital Res.(Hz/pt): 0.37
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 157.1
Relaxation Delay(s): 1
Completed Scans 512
DK-3-193-C
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe
NO2
CF3
2h
180
160
140
120
100
80
60
40
20
0
ppm

Agilent Technologies

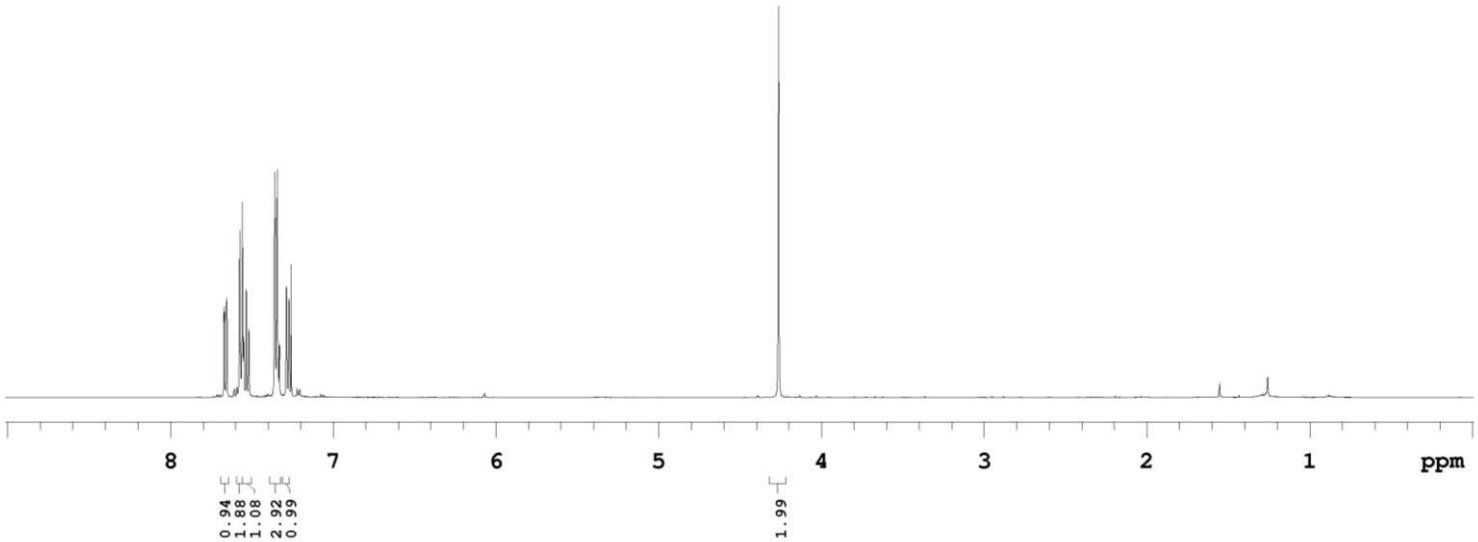
Department of Chemistry, University of Alberta

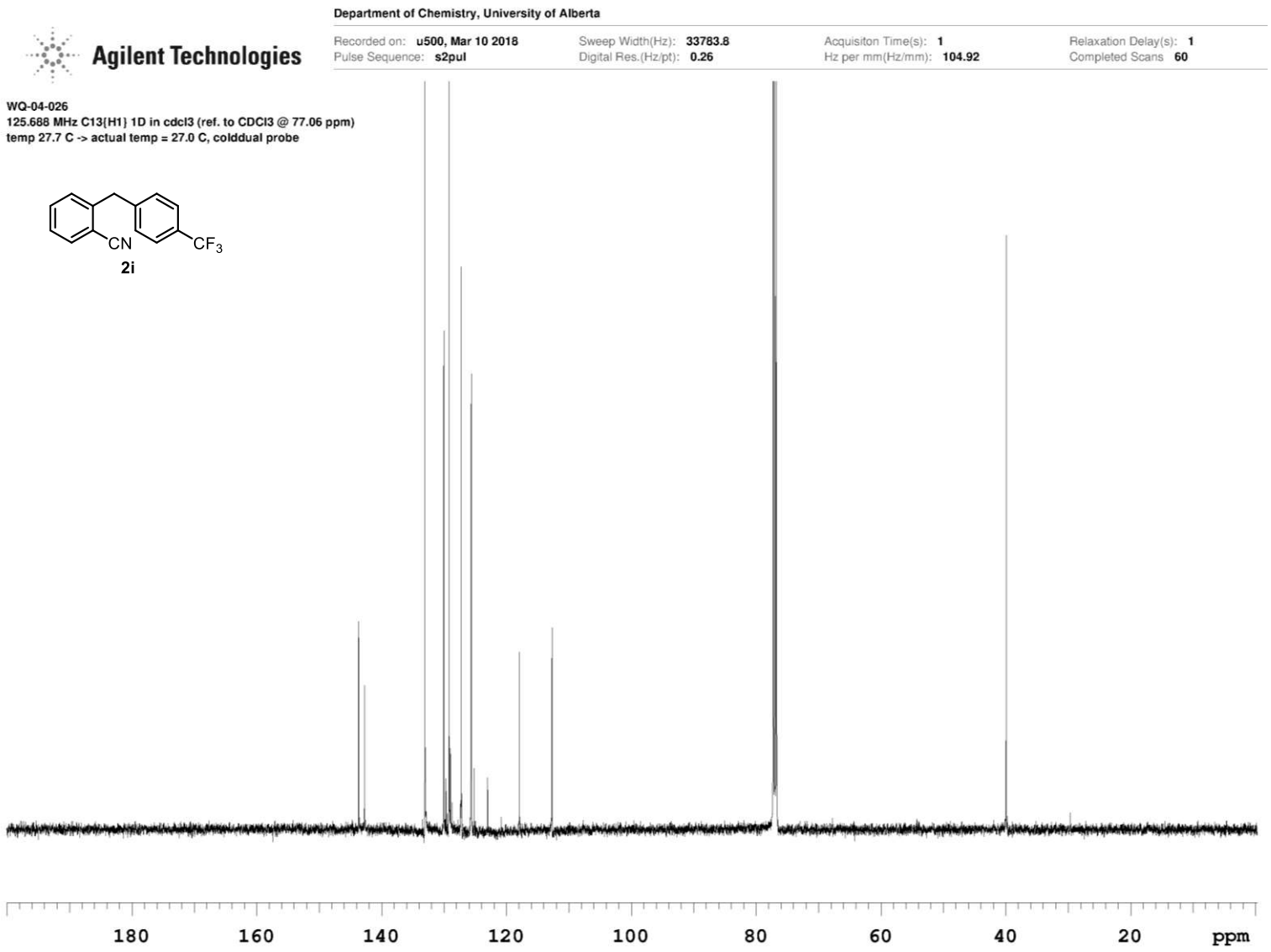
Recorded on:	u500, Mar 10 2018	Sweep Width(Hz):	6009.62	Acquisiton Time(s):	5	Relaxation Delay(s):	0.1
Pulse Sequence:	PRESAT	Digital Res.(Hz/pt):	0.09	Hz per mm(Hz/mm):	18.78	Completed Scans	8

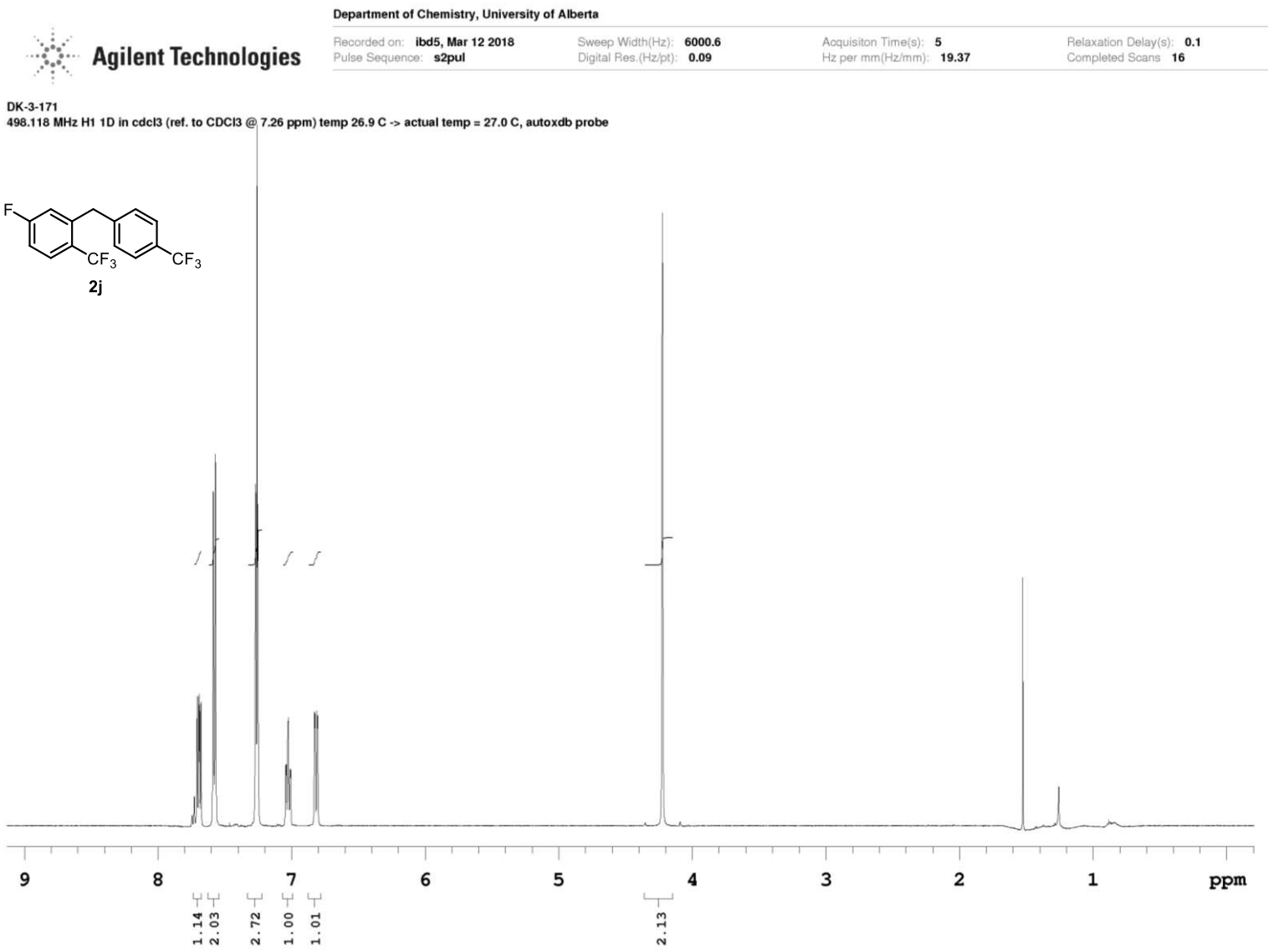
WQ-04-026
499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe

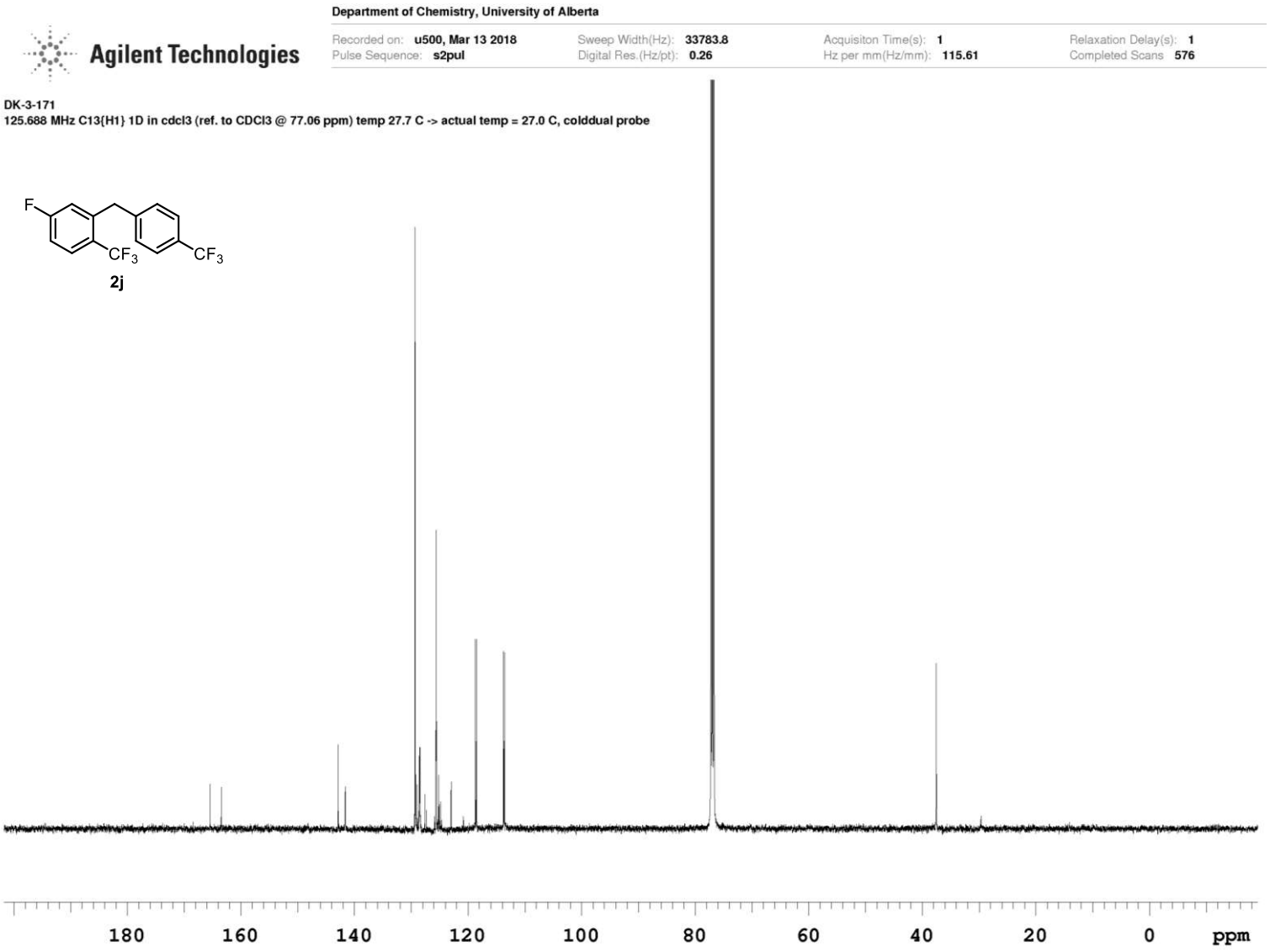


2i




Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 10 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 104.92
Relaxation Delay(s): 1
Completed Scans 60
WQ-04-026
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
CN
CF3
2i
180
160
140
120
100
80
60
40
20
ppm


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: ibd5, Mar 12 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 6000.6
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 19.37
Relaxation Delay(s): 0.1
Completed Scans 16
DK-3-171
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
F
CF3
CF3
2j
9
8
7
6
5
4
3
2
1
ppm
1.14
2.03
2.72
1.00
1.01
2.13


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 13 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 115.61
Relaxation Delay(s): 1
Completed Scans 576
DK-3-171
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
F
CF3
CF3
2j
180
160
140
120
100
80
60
40
20
0
ppm

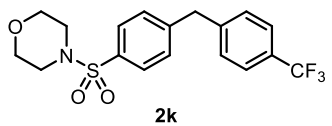
Department of Chemistry, University of Alberta

Agilent Technologies

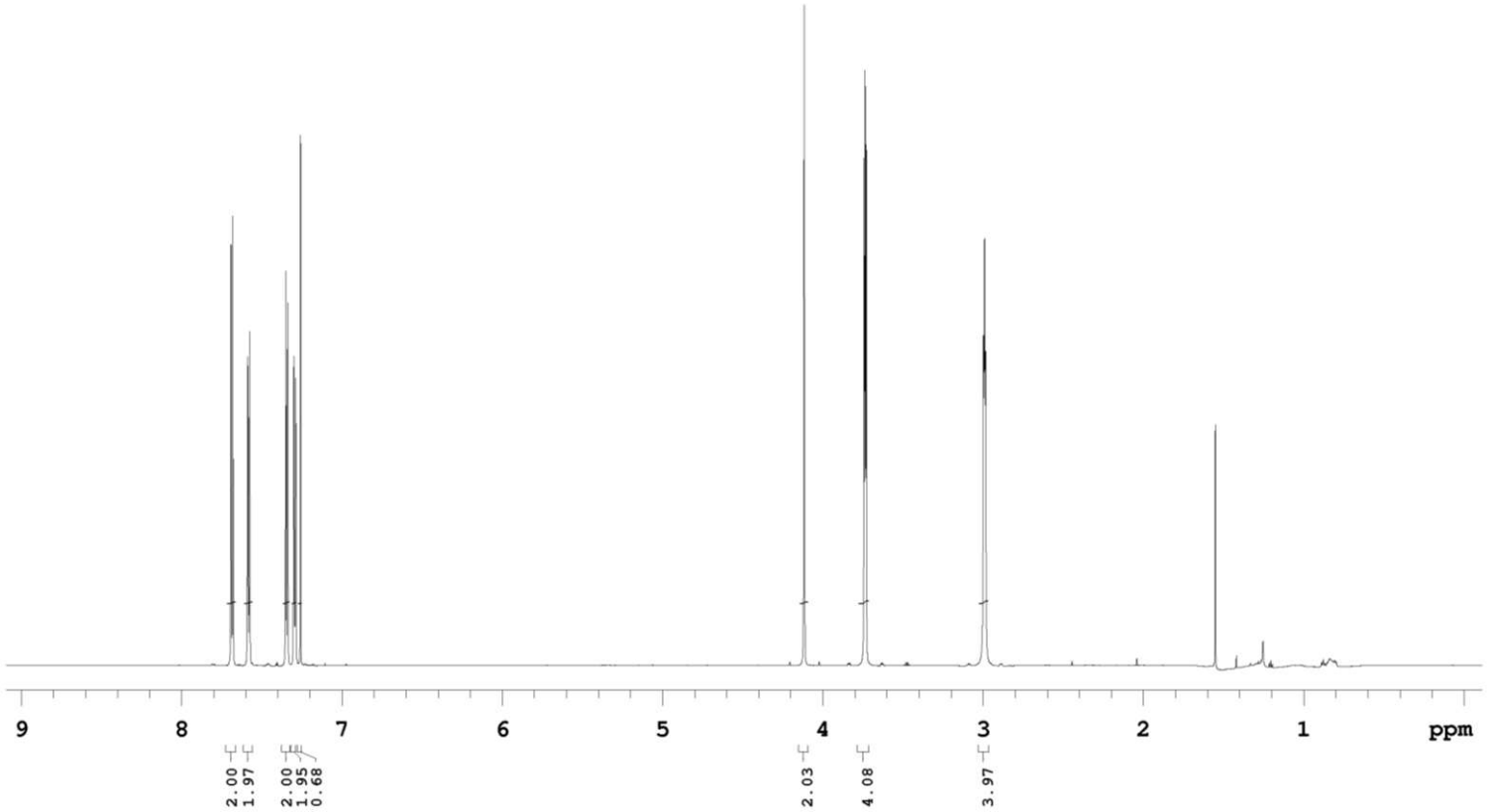
Recorded on: **v700, Mar 22 2018**	Sweep Width(Hz): **8389.26**	Acquisiton Time(s): **5**	Relaxation Delay(s): **0.1**
Pulse Sequence: **PRESAT**	Digital Res.(Hz/pt): **0.13**	Hz per mm(Hz/mm): **26.84**	Completed Scans **8**

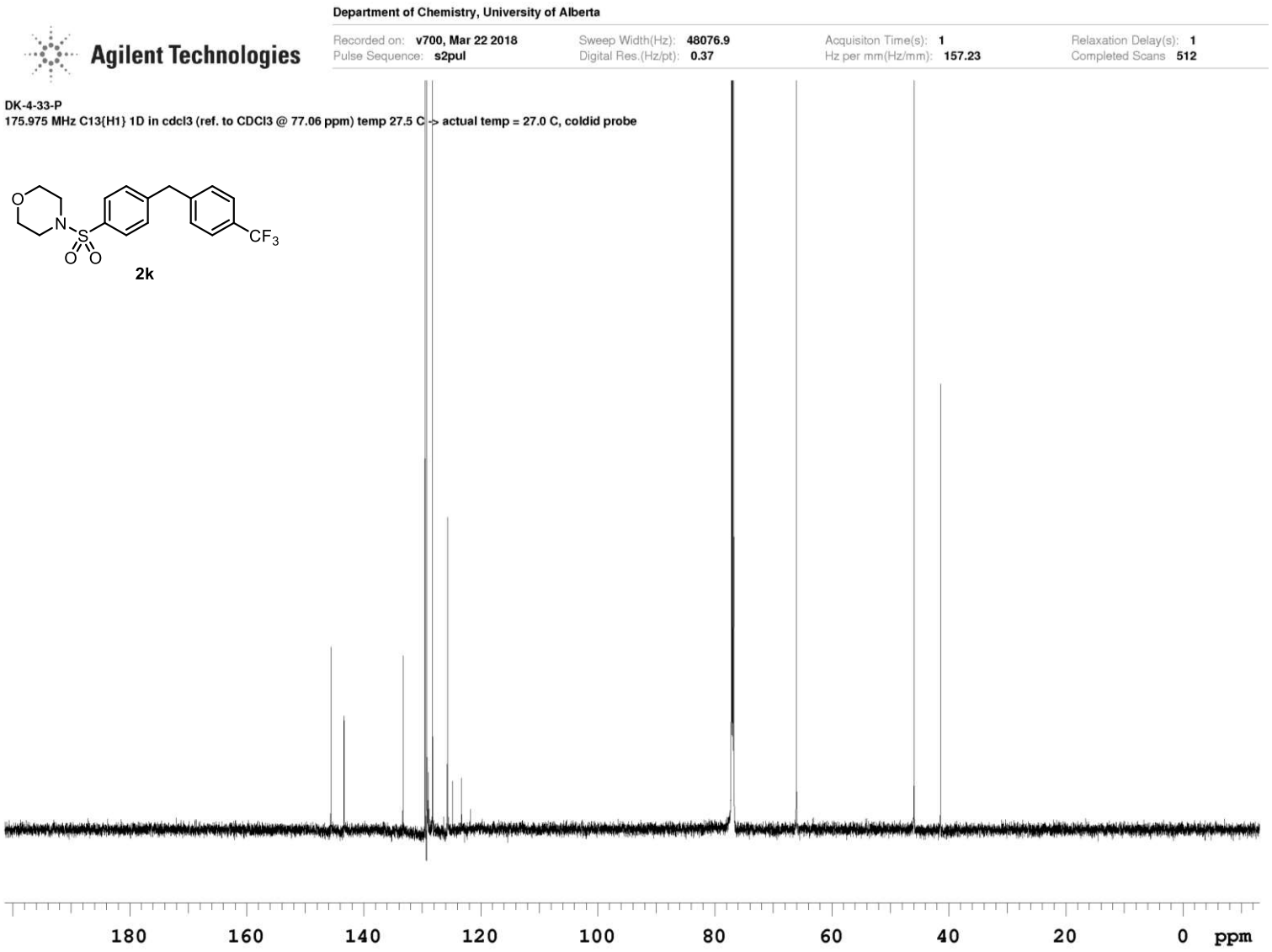
DK-4-33-P

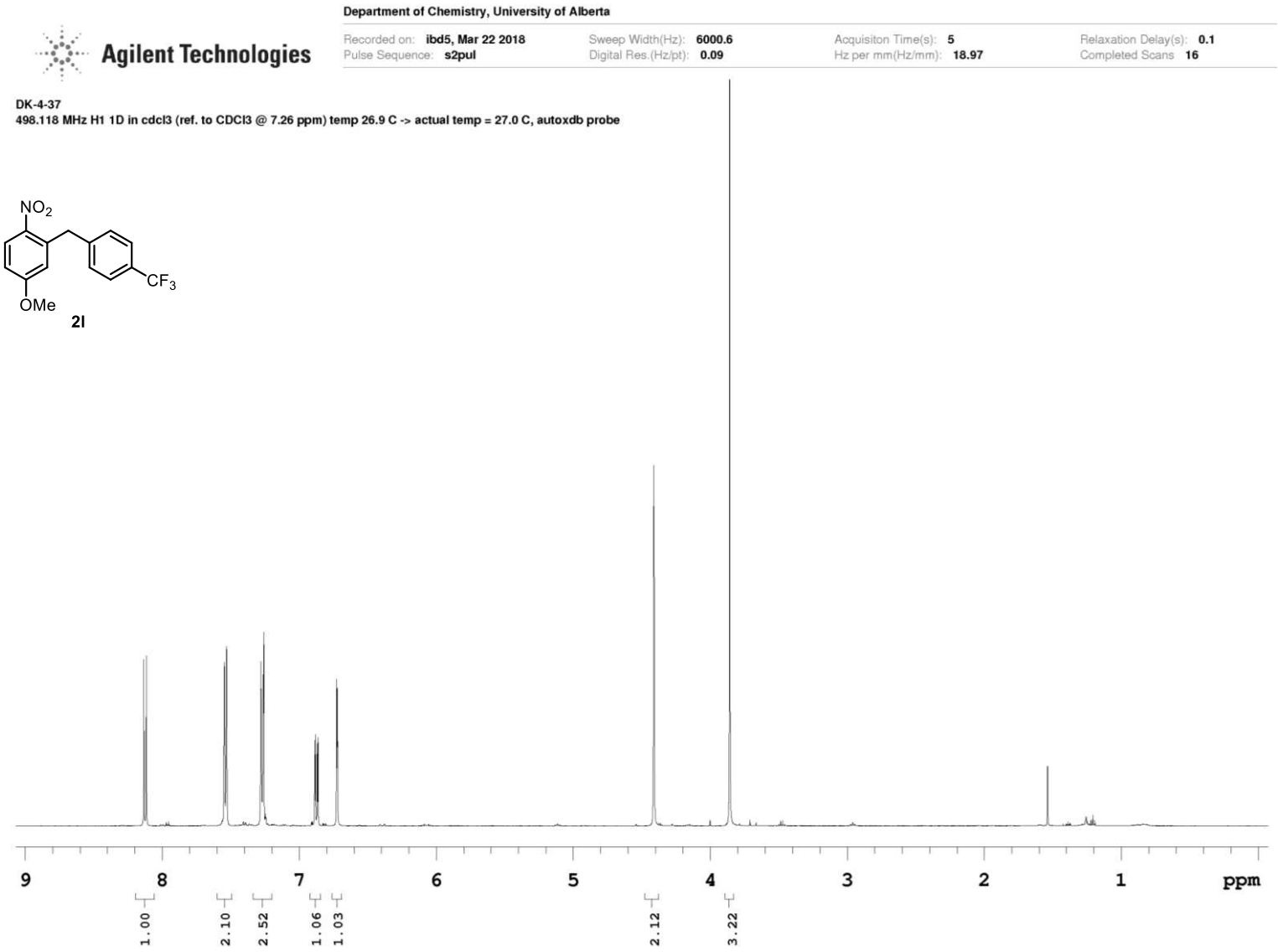
699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

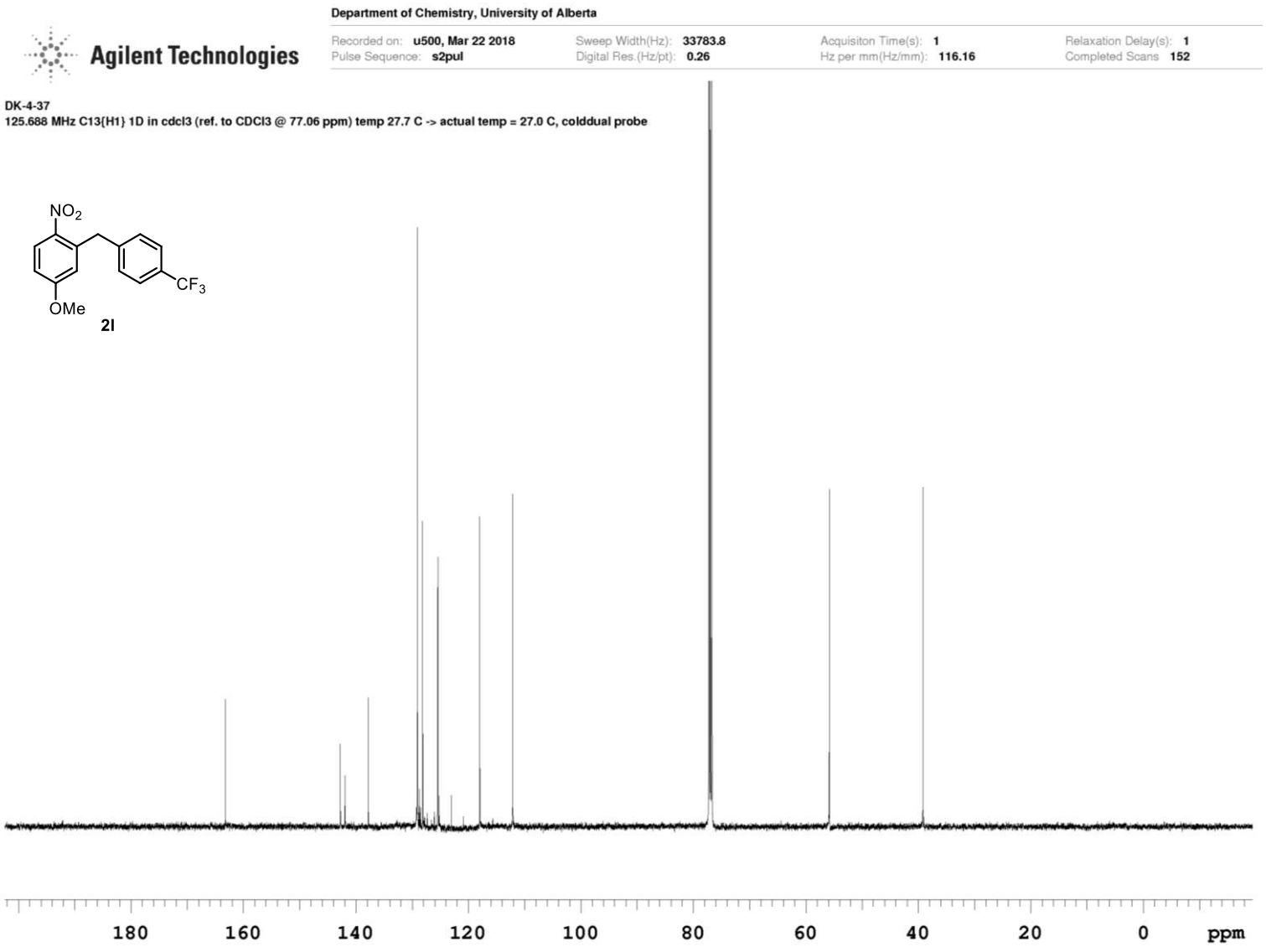


2k




Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: v700, Mar 22 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 48076.9
Digital Res.(Hz/pt): 0.37
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 157.23
Relaxation Delay(s): 1
Completed Scans 512
DK-4-33-P
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe
O
N
S
O
O
CF3
2k
180
160
140
120
100
80
60
40
20
0
ppm


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: ibd5, Mar 22 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 6000.6
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 18.97
Relaxation Delay(s): 0.1
Completed Scans 16
DK-4-37
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
NO2
CF3
OMe
2l
9
8
7
6
5
4
3
2
1
ppm
1.00
2.10
2.52
1.06
1.03
2.12
3.22

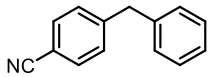

Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 22 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 116.16
Relaxation Delay(s): 1
Completed Scans 152
DK-4-37
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
NO2
CF3
OMe
2l
180
160
140
120
100
80
60
40
20
0
ppm

Agilent Technologies

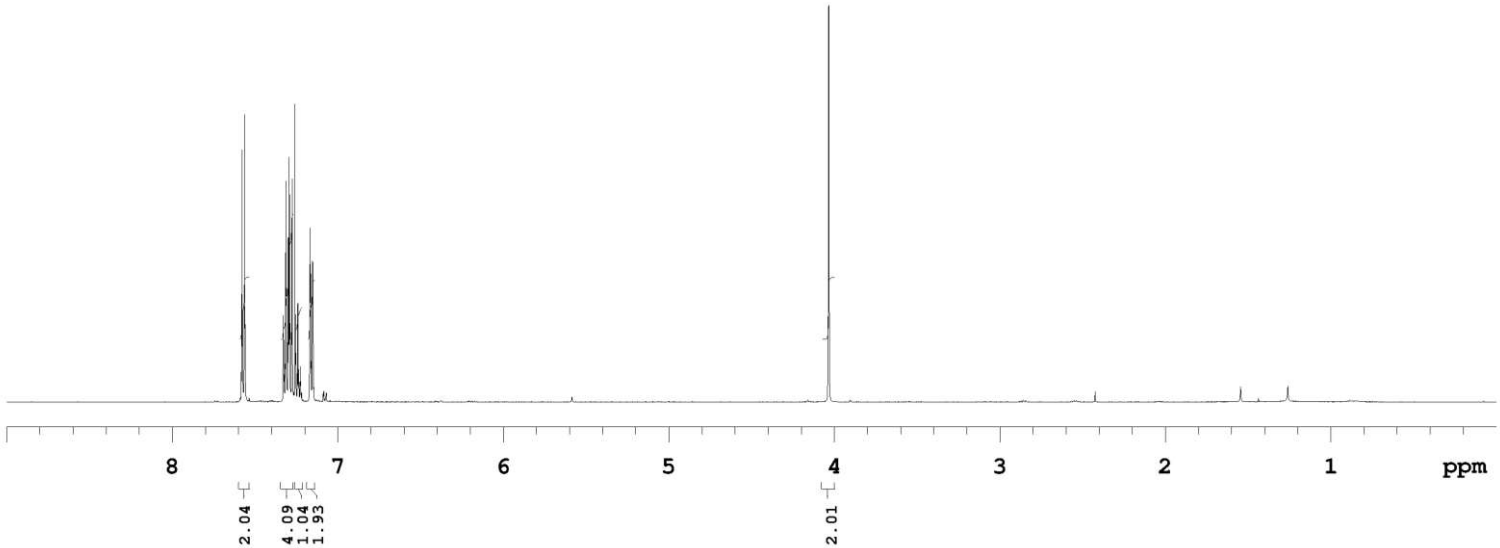
Department of Chemistry, University of Alberta

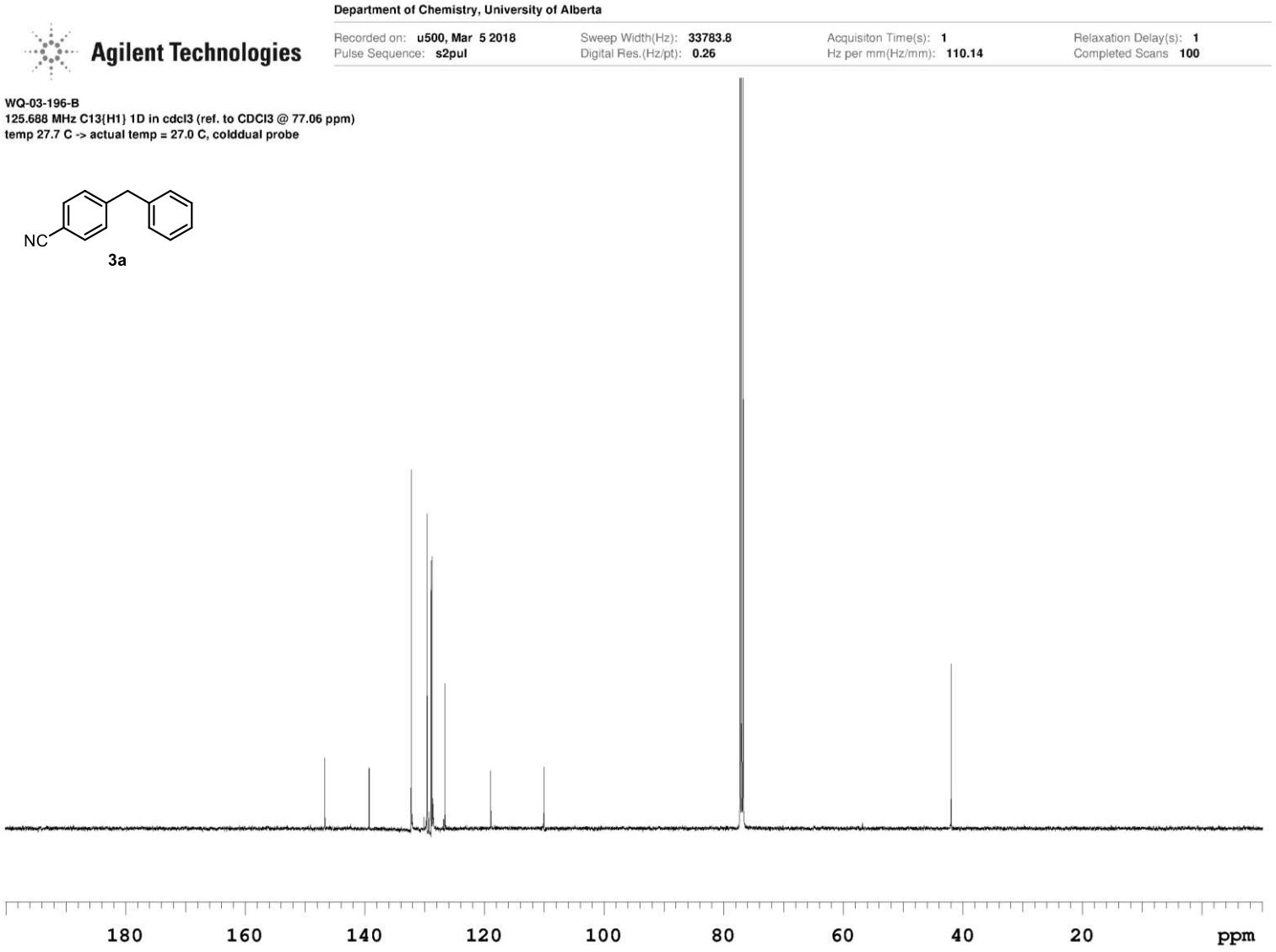
Recorded on: u500, Mar 5 2018	Sweep Width(Hz): 6009.62	Acquisiton Time(s): 5	Relaxation Delay(s): 0.1
Pulse Sequence: PRESAT	Digital Res.(Hz/pt): 0.09	Hz per mm(Hz/mm): 18.74	Completed Scans 2

WQ-03-196-B
499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe



3a



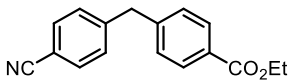

Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 5 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 110.14
Relaxation Delay(s): 1
Completed Scans 100
WQ-03-196-B
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
NC
3a
180
160
140
120
100
80
60
40
20
ppm

Department of Chemistry, University of Alberta

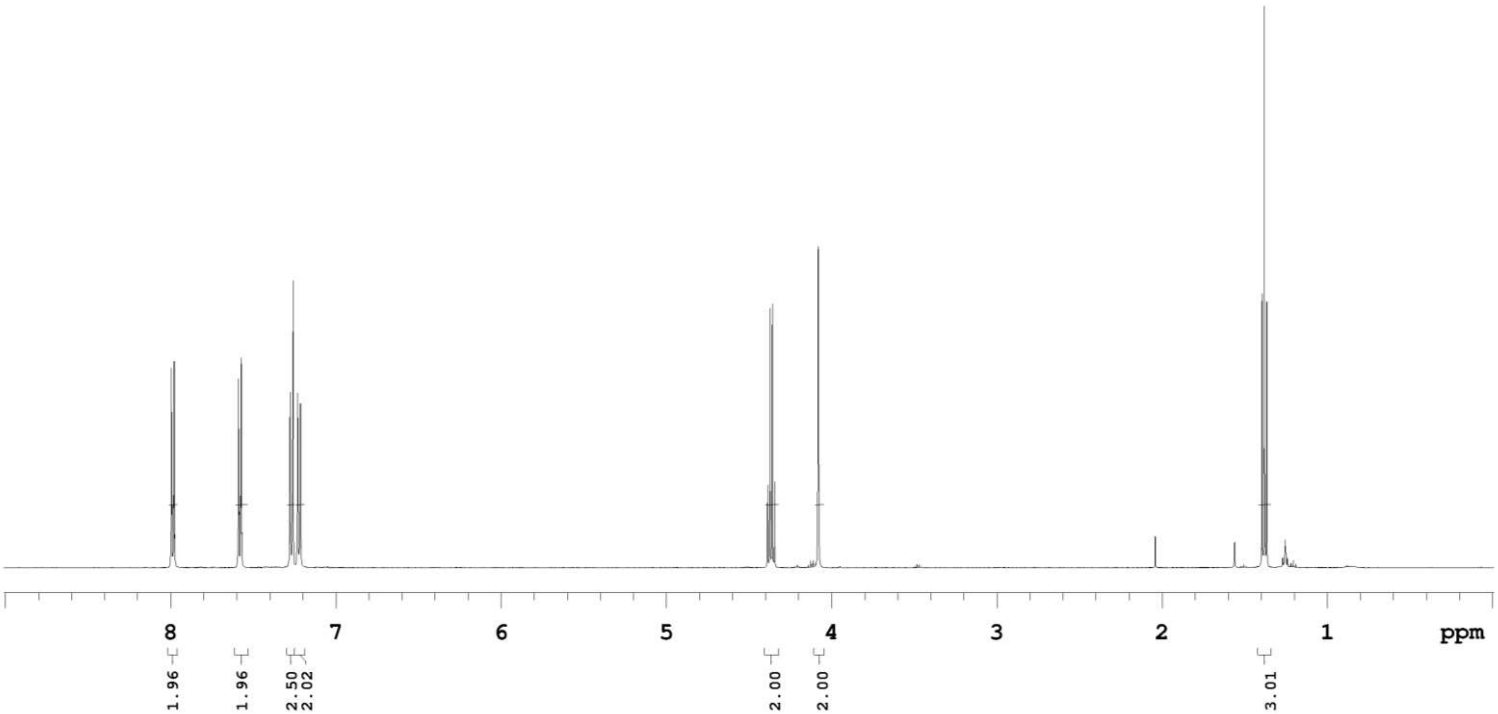
Agilent Technologies

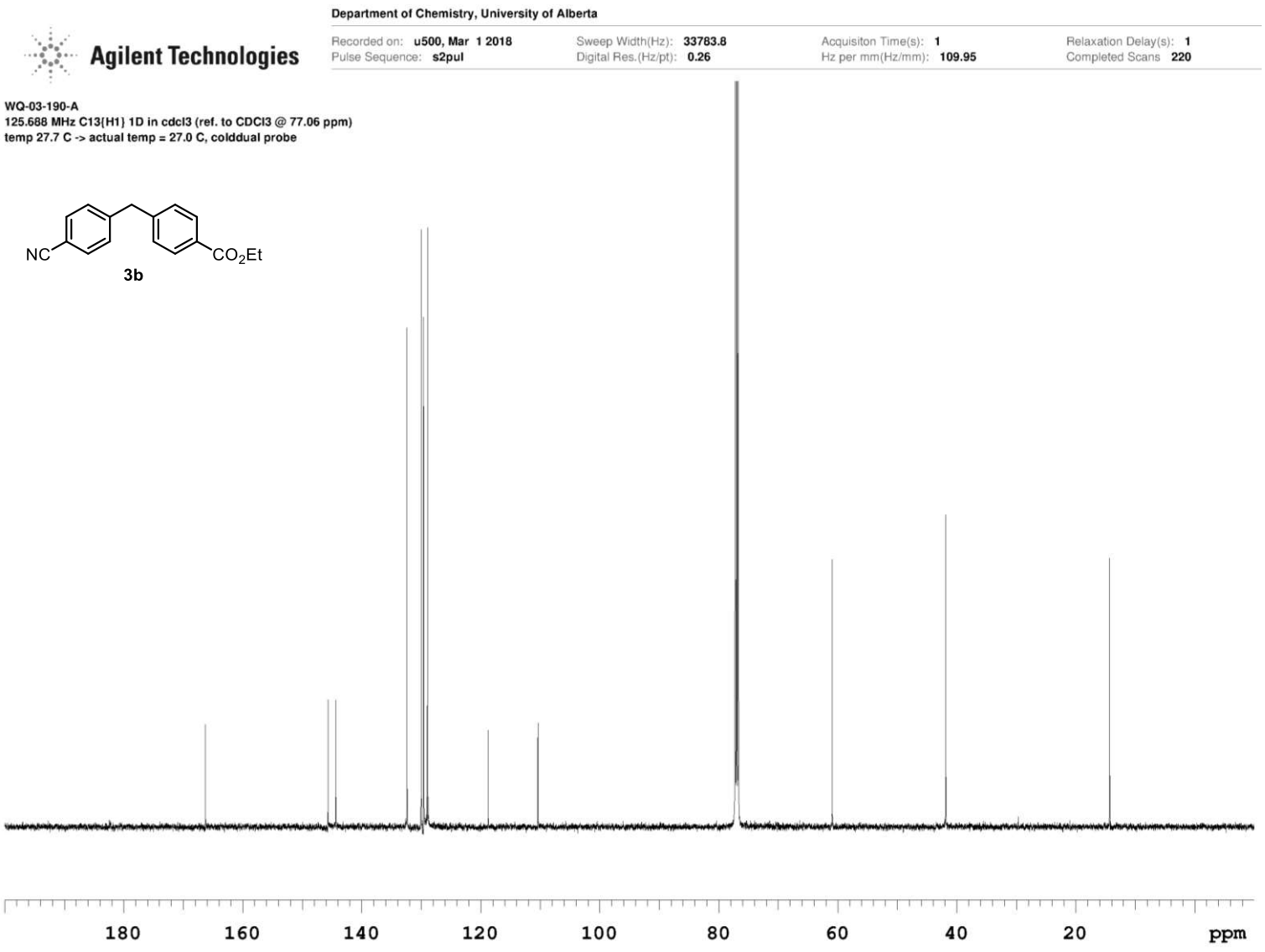
Recorded on: **u500, Mar 1 2018**	Sweep Width(Hz): **6009.62**	Acquisiton Time(s): **5**	Relaxation Delay(s): **0.1**
Pulse Sequence: **PRESAT**	Digital Res.(Hz/pt): **0.09**	Hz per mm(Hz/mm): **18.78**	Completed Scans **2**

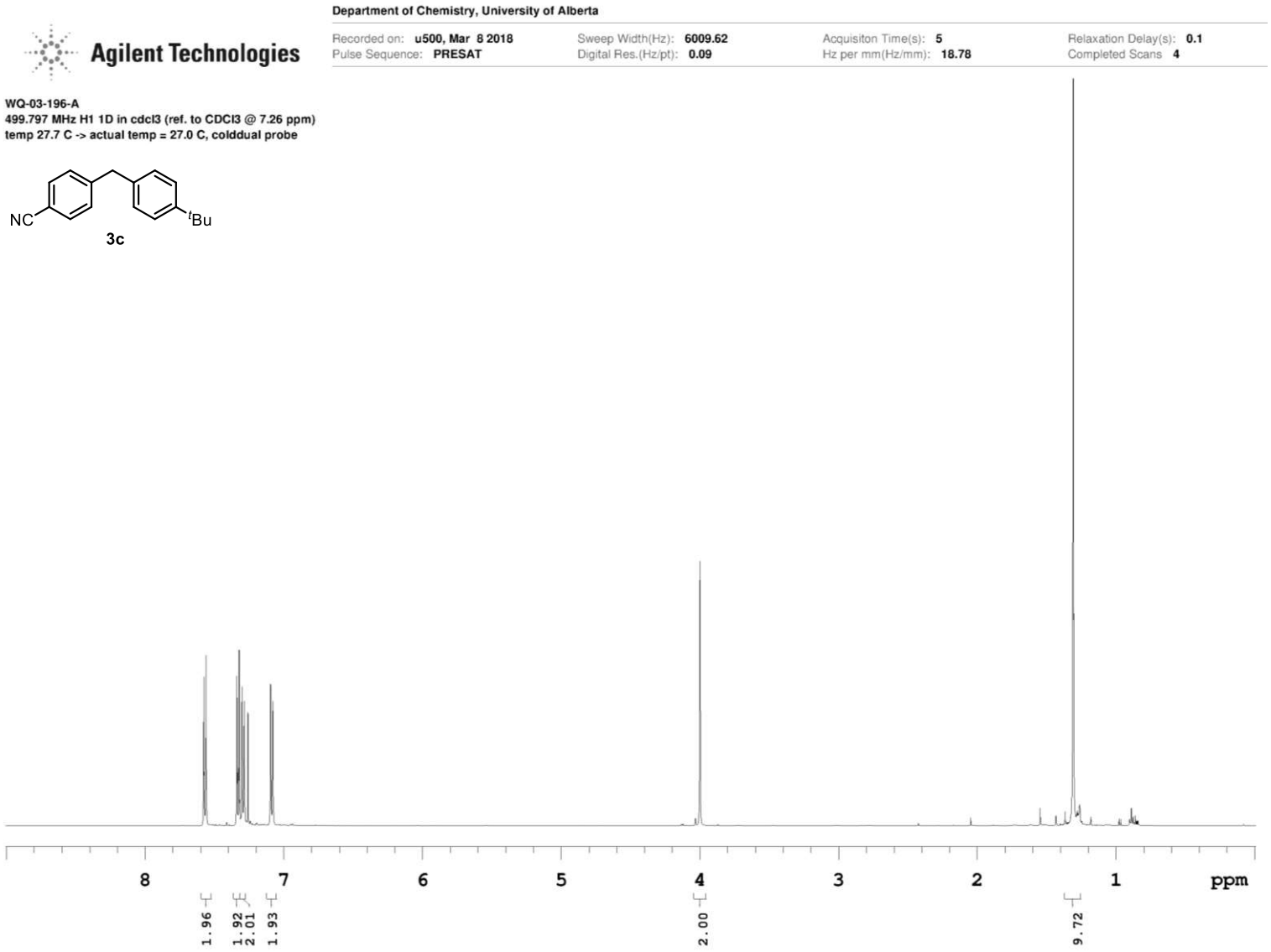
WQ-03-190-A
499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe

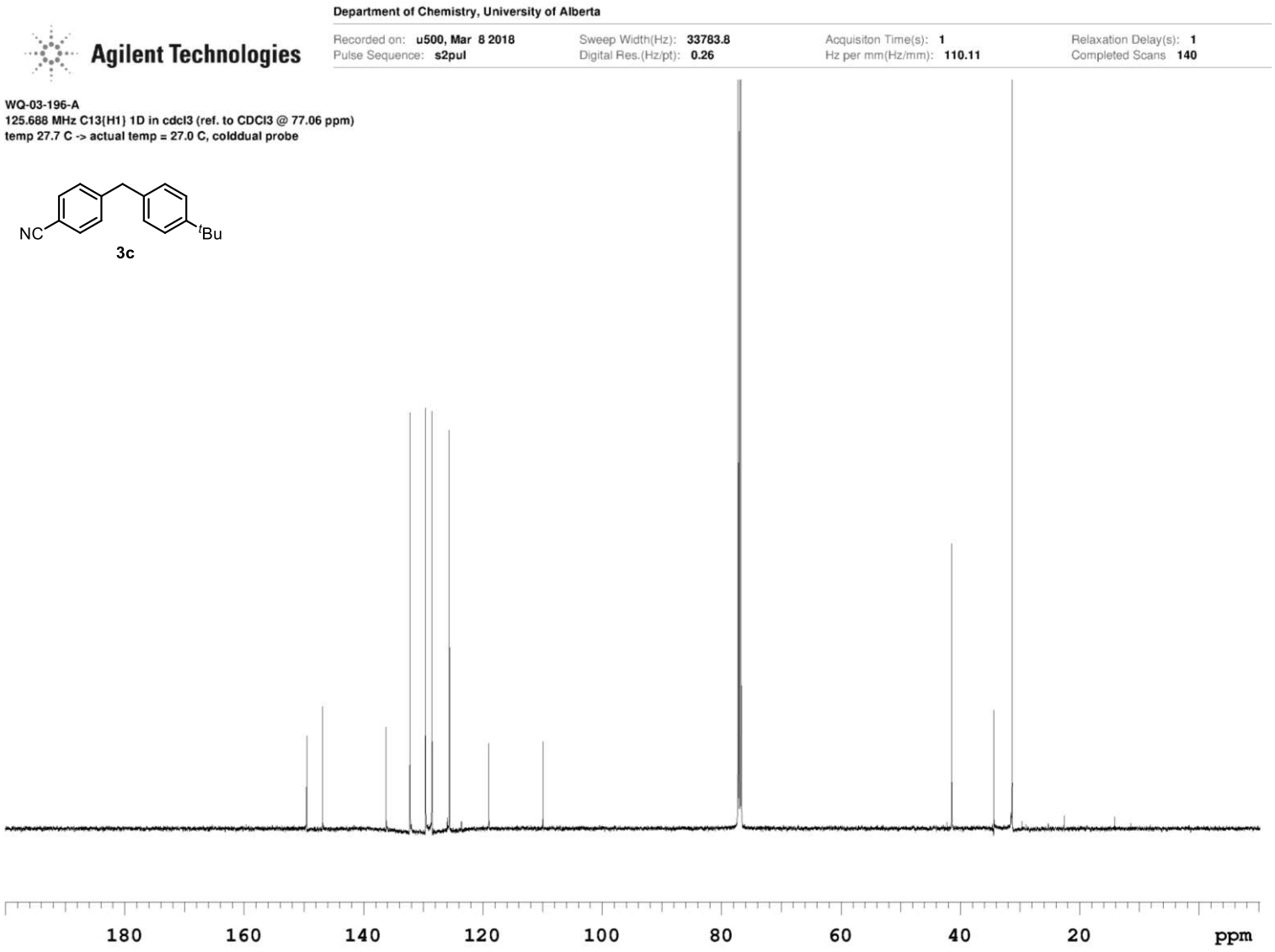


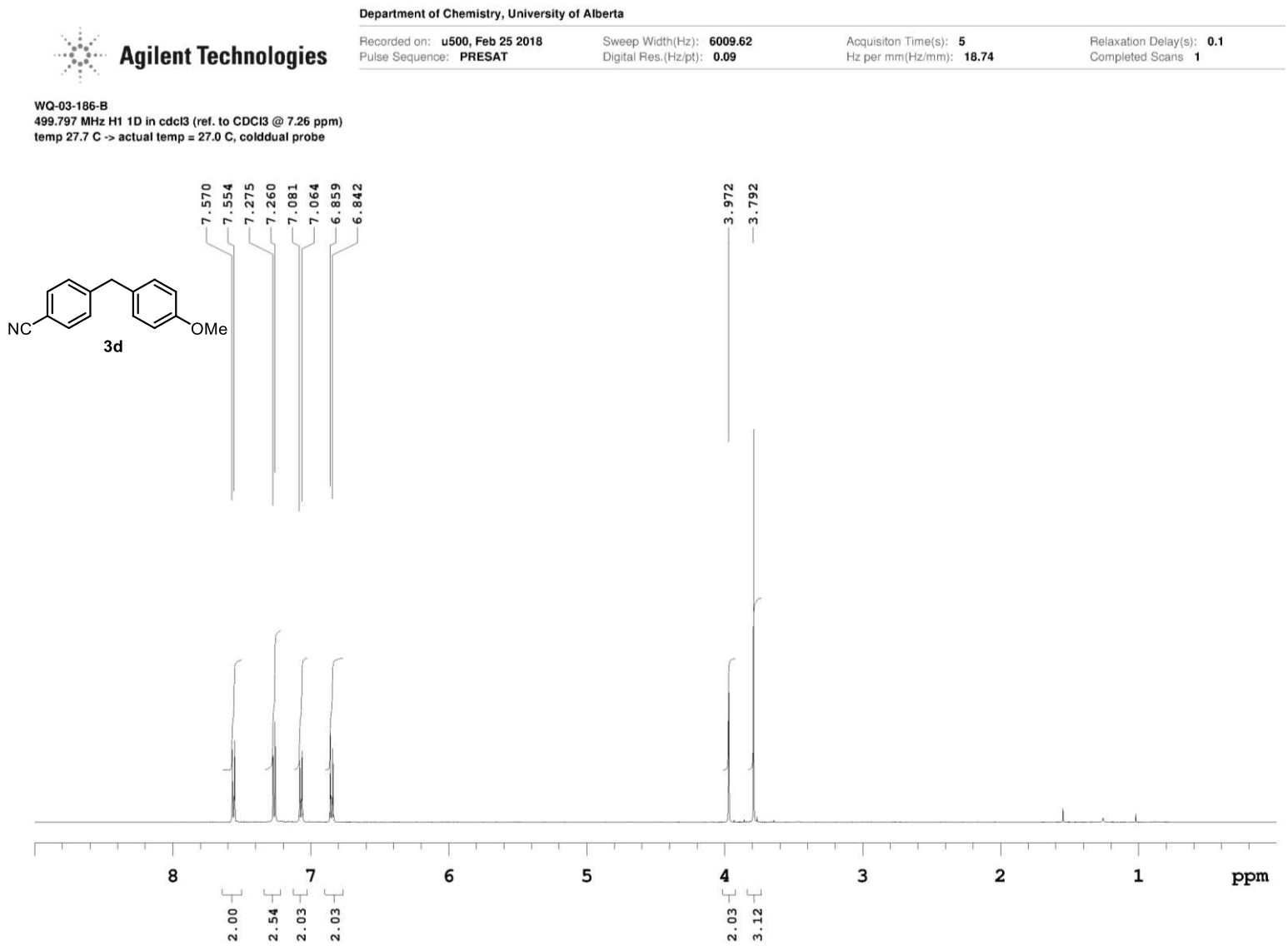
3b

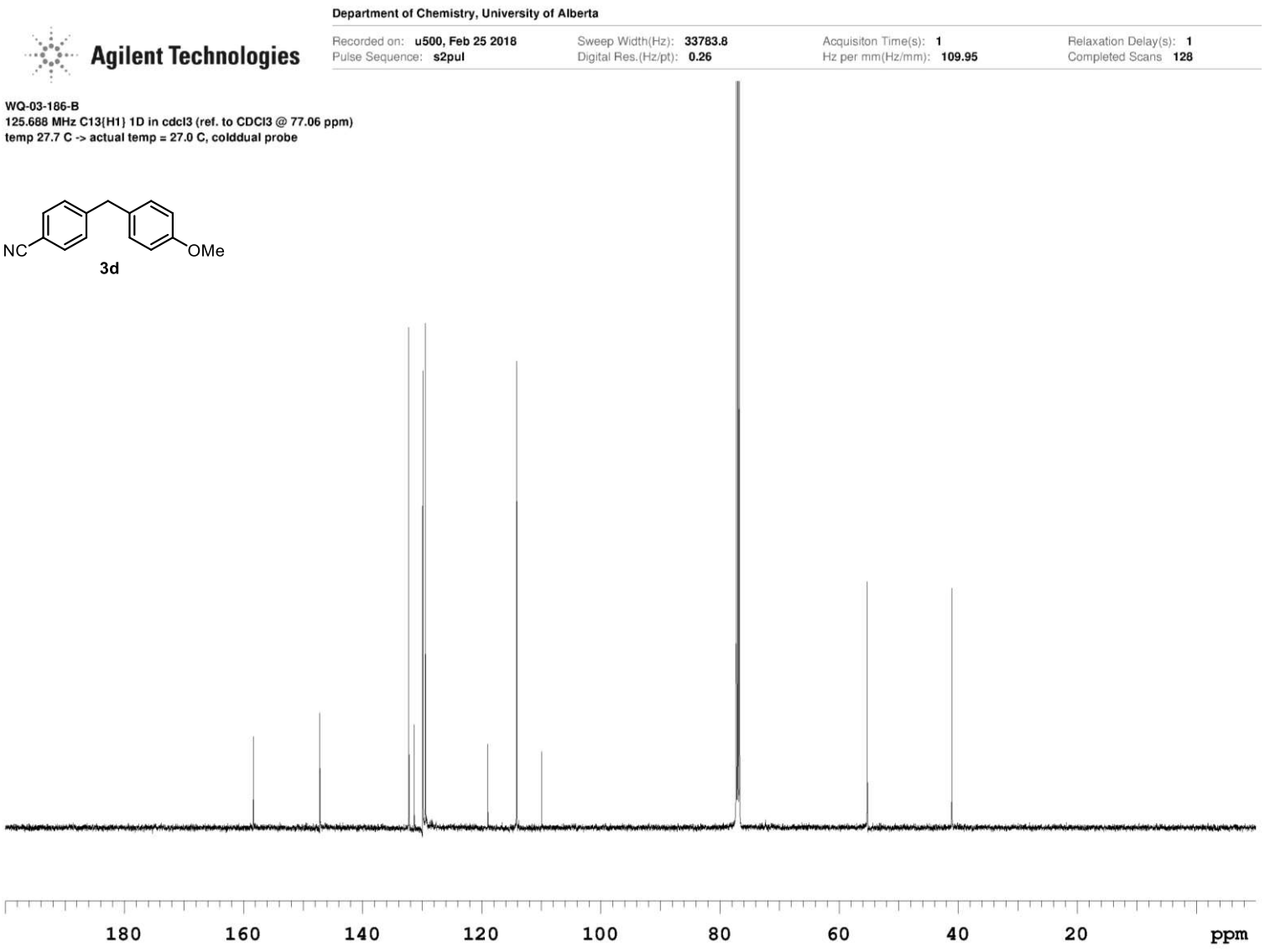



Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 1 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 109.95
Relaxation Delay(s): 1
Completed Scans 220
WQ-03-190-A
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
NC
CO2Et
3b
180
160
140
120
100
80
60
40
20
ppm


Agilent Technologies
Department of Chemistry, University of Alberta
Recorded on: u500, Mar 8 2018
Pulse Sequence: PRESAT
Sweep Width(Hz): 6009.62
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 18.78
Relaxation Delay(s): 0.1
Completed Scans 4
WQ-03-196-A
499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
NC
tBu
3c
8
7
6
5
4
3
2
1
ppm
1.96
1.92
2.01
1.93
2.00
9.72


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 8 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 110.11
Relaxation Delay(s): 1
Completed Scans 140
WQ-03-196-A
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
NC
tBu
3c
180
160
140
120
100
80
60
40
20
ppm


Agilent Technologies
Department of Chemistry, University of Alberta
Recorded on: u500, Feb 25 2018
Pulse Sequence: PRESAT
Sweep Width(Hz): 6009.62
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 18.74
Relaxation Delay(s): 0.1
Completed Scans 1
WQ-03-186-B
499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
7.570
7.554
7.275
7.260
7.081
7.064
6.859
6.842
3.972
3.792
NC
OMe
3d
8
7
6
5
4
3
2
1
ppm
2.00
2.54
2.03
2.03
2.03
3.12

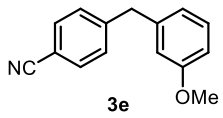

Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Feb 25 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 109.95
Relaxation Delay(s): 1
Completed Scans 128
WQ-03-186-B
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
NC
OMe
3d
180
160
140
120
100
80
60
40
20
ppm

Agilent Technologies

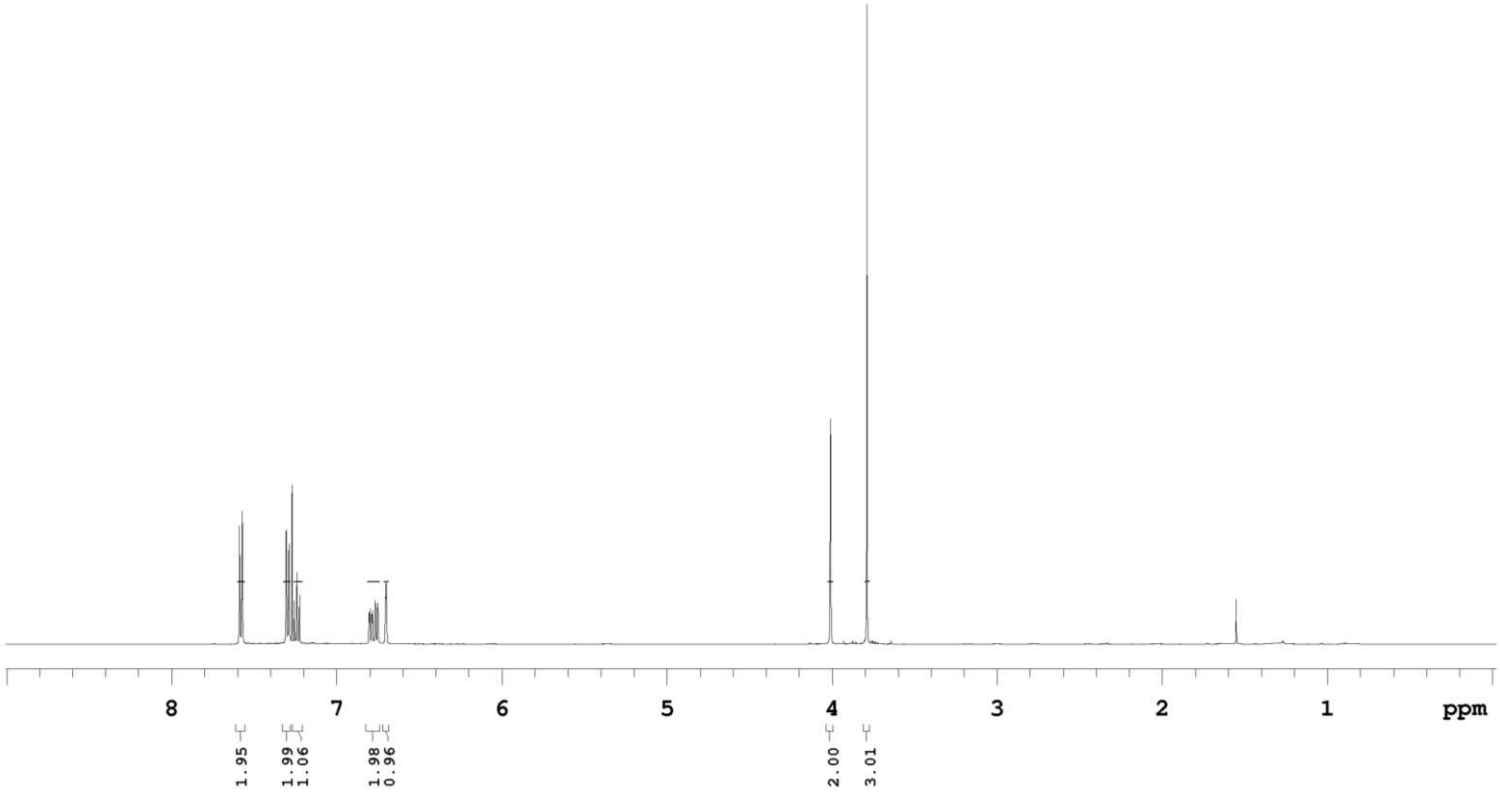
Department of Chemistry, University of Alberta

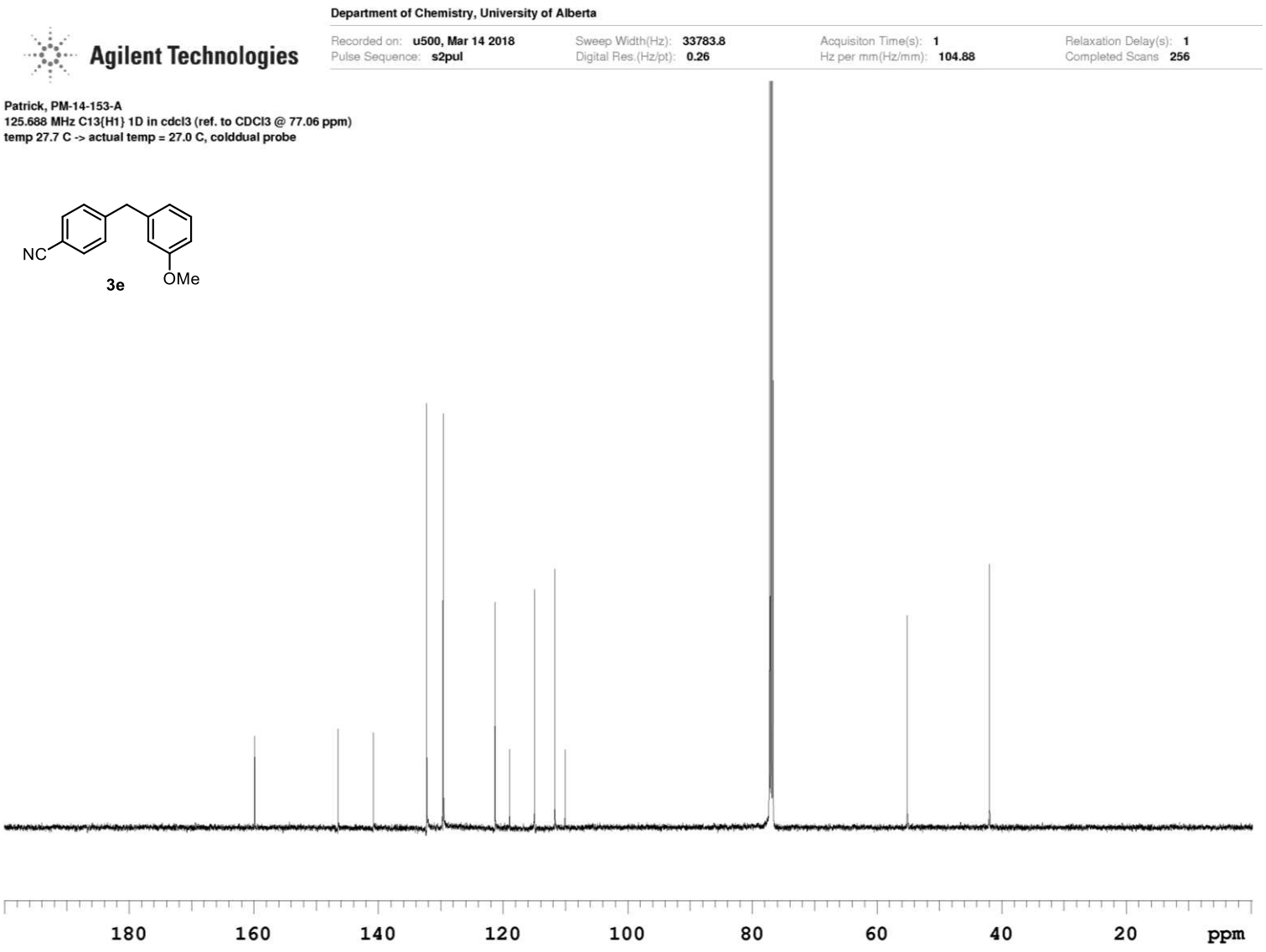
Recorded on:	u500, Mar 14 2018	Sweep Width(Hz):	6009.62	Acquisiton Time(s):	5	Relaxation Delay(s):	0.1
Pulse Sequence:	PRESAT	Digital Res.(Hz/pt):	0.09	Hz per mm(Hz/mm):	18.8	Completed Scans	8

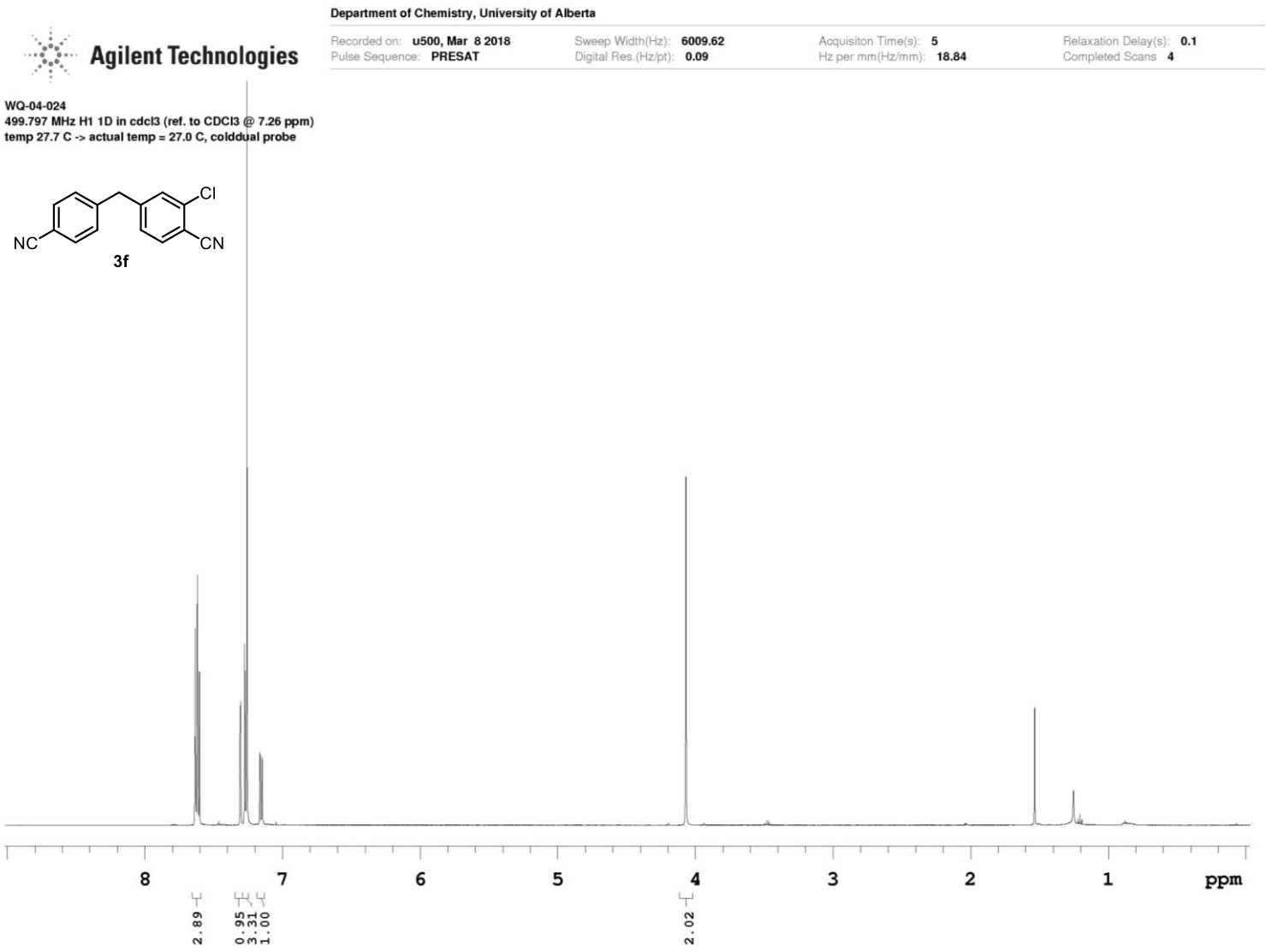
Patrick, PM-14-153-A
499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe

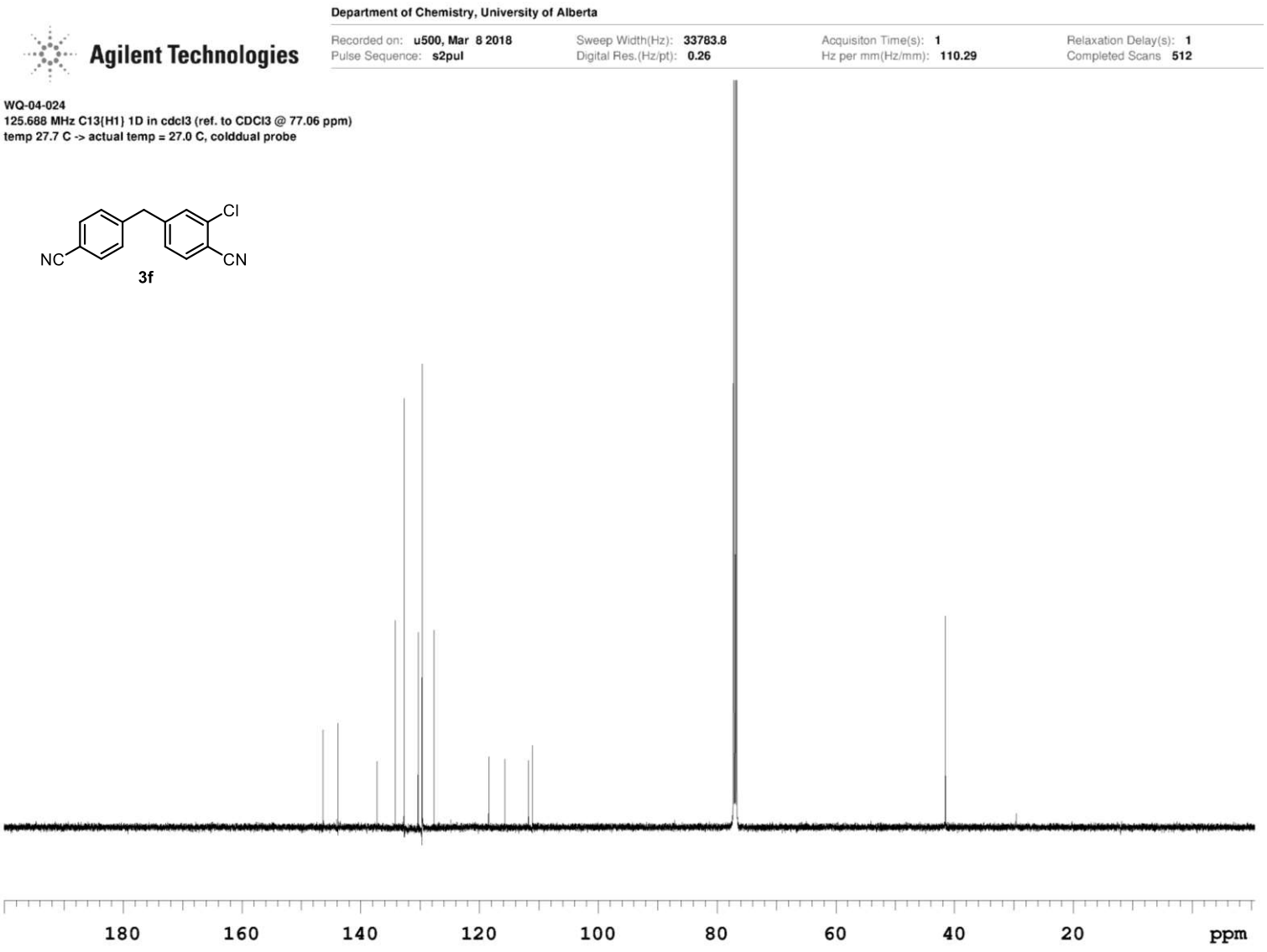


3e




Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 14 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 104.88
Relaxation Delay(s): 1
Completed Scans 256
Patrick, PM-14-153-A
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
NC
OMe
3e
180
160
140
120
100
80
60
40
20
ppm


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 8 2018
Pulse Sequence: PRESAT
Sweep Width(Hz): 6009.62
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 18.84
Relaxation Delay(s): 0.1
Completed Scans 4
WQ-04-024
499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
Cl
NC
CN
3f
8
7
6
5
4
3
2
1
ppm
2.89
0.95
3.31
1.00
2.02

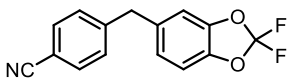

Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 8 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 110.29
Relaxation Delay(s): 1
Completed Scans 512
WQ-04-024
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
Cl
NC
CN
3f
180
160
140
120
100
80
60
40
20
ppm

Agilent Technologies

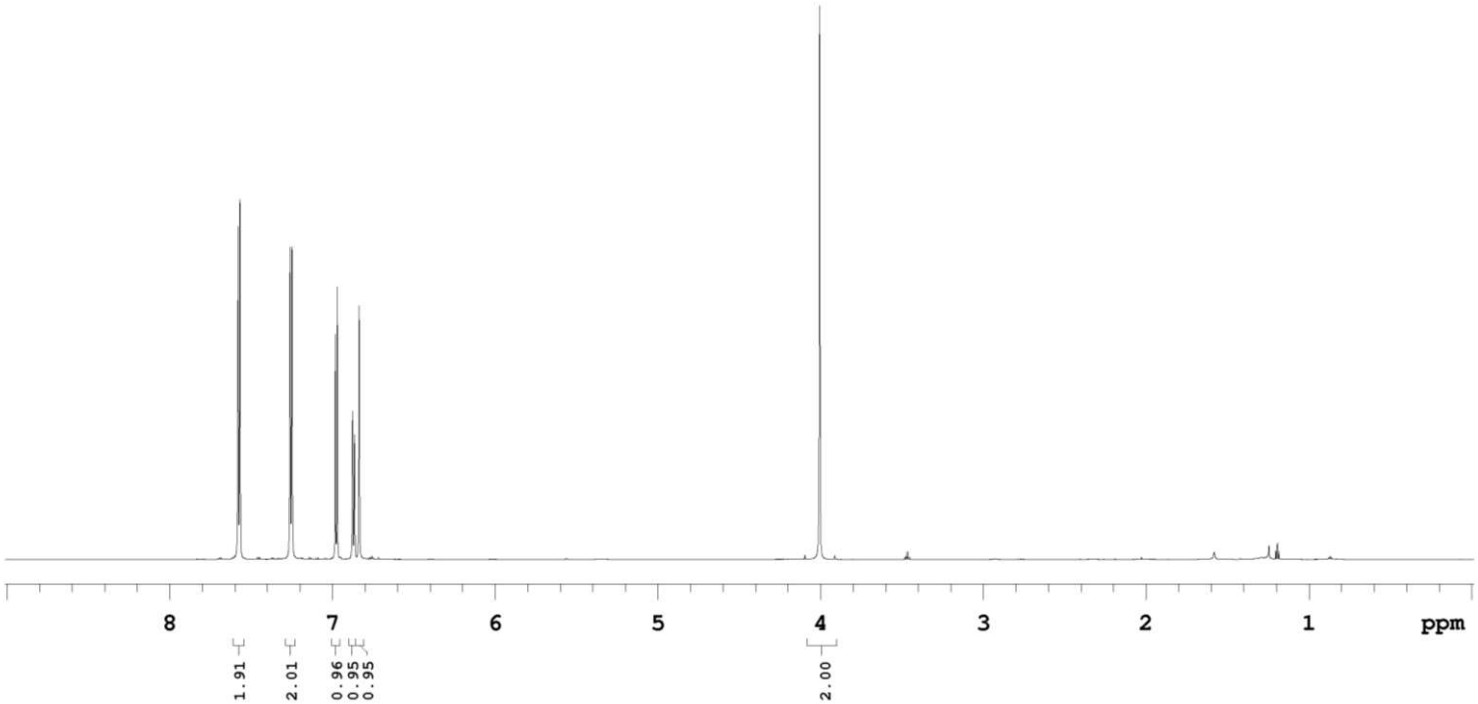
Department of Chemistry, University of Alberta

Recorded on:	v700, Mar 22 2018	Sweep Width(Hz):	8389.26	Acquisiton Time(s):	5	Relaxation Delay(s):	0.1
Pulse Sequence:	PRESAT	Digital Res.(Hz/pt):	0.13	Hz per mm(Hz/mm):	26.31	Completed Scans	8

PM-14-165-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



3g

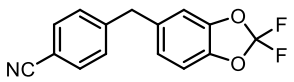


Department of Chemistry, University of Alberta

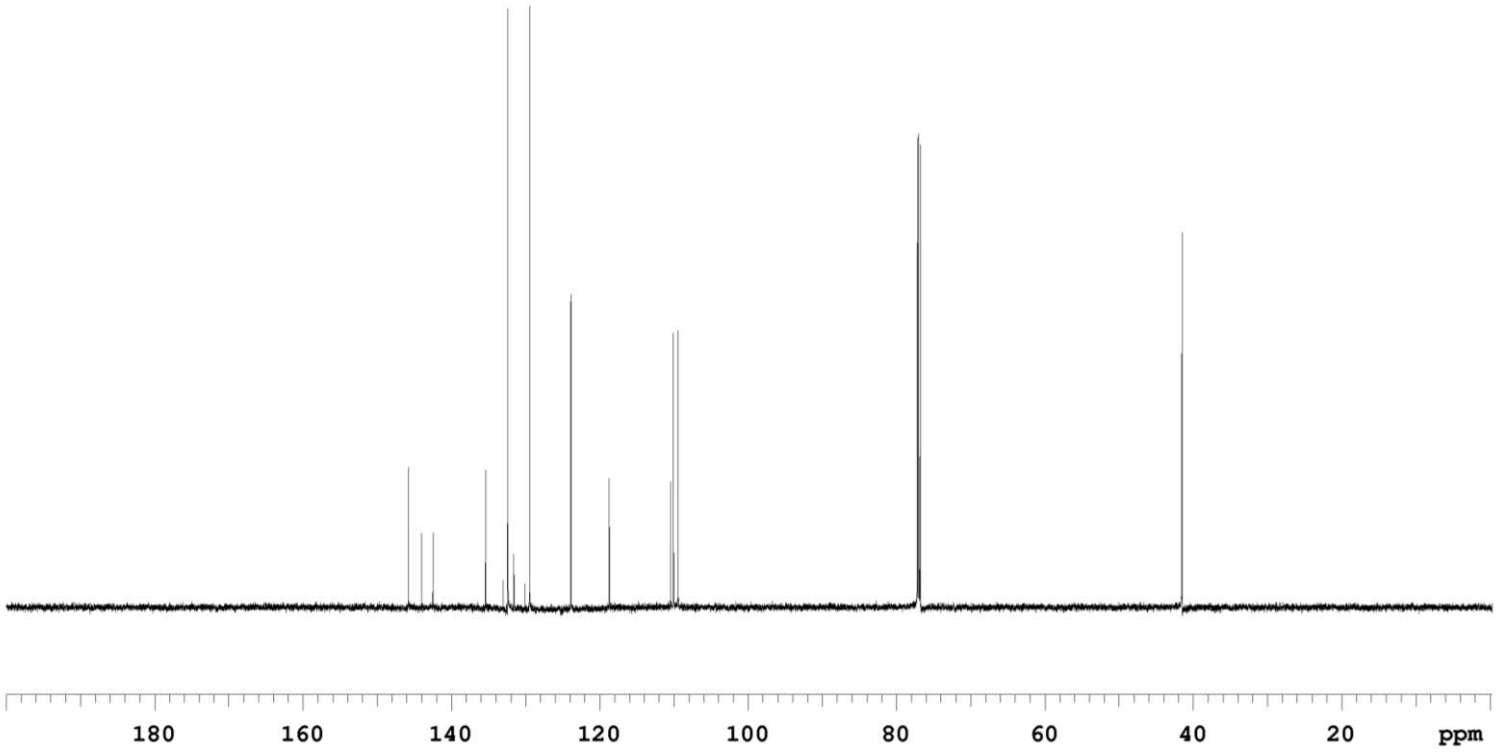
Agilent Technologies

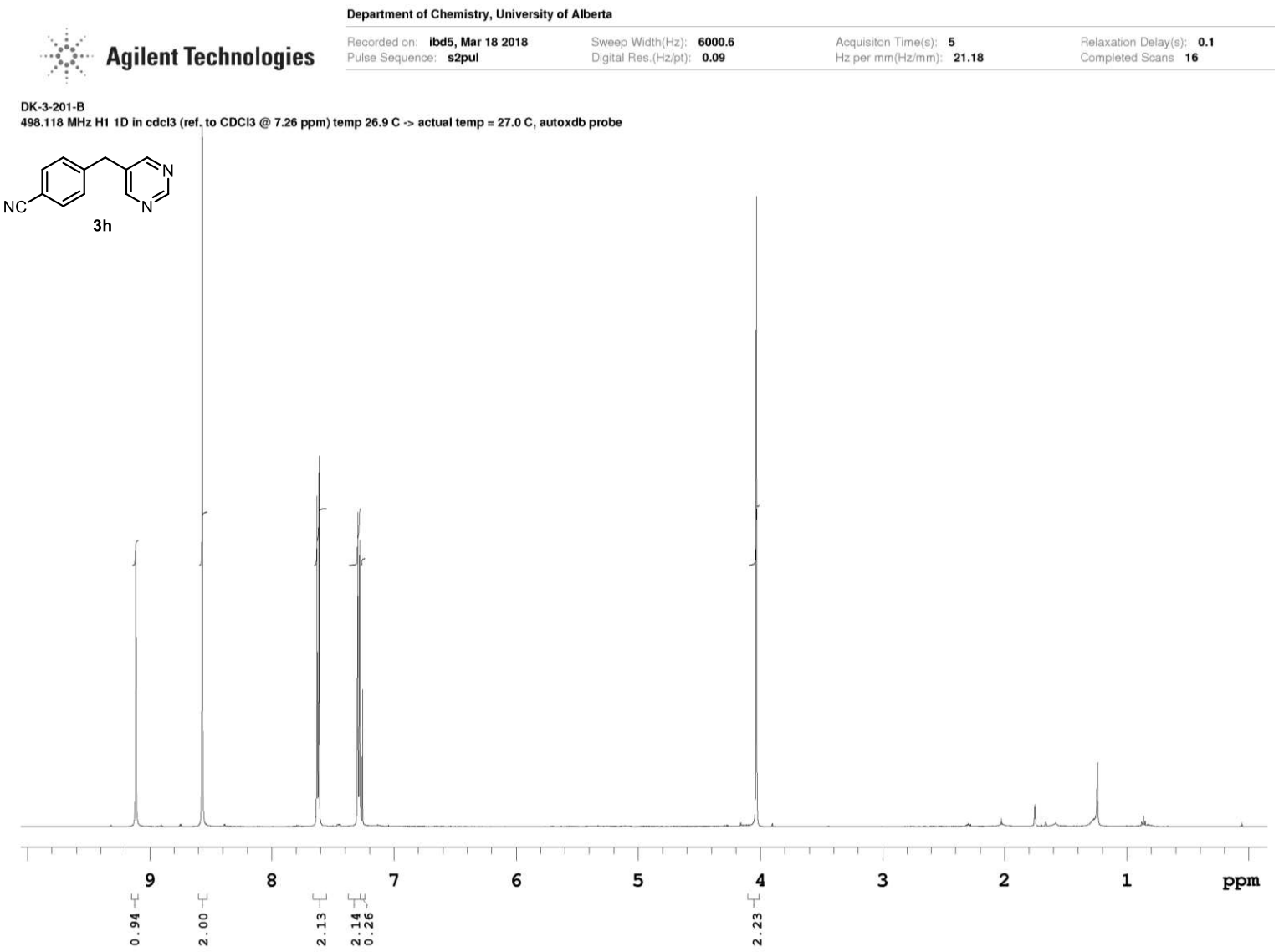
Recorded on: v700, Mar 22 2018	Sweep Width(Hz): 48076.9	Acquisiton Time(s): 1	Relaxation Delay(s): 1
Pulse Sequence: s2pul	Digital Res.(Hz/pt): 0.37	Hz per mm(Hz/mm): 146.81	Completed Scans 24

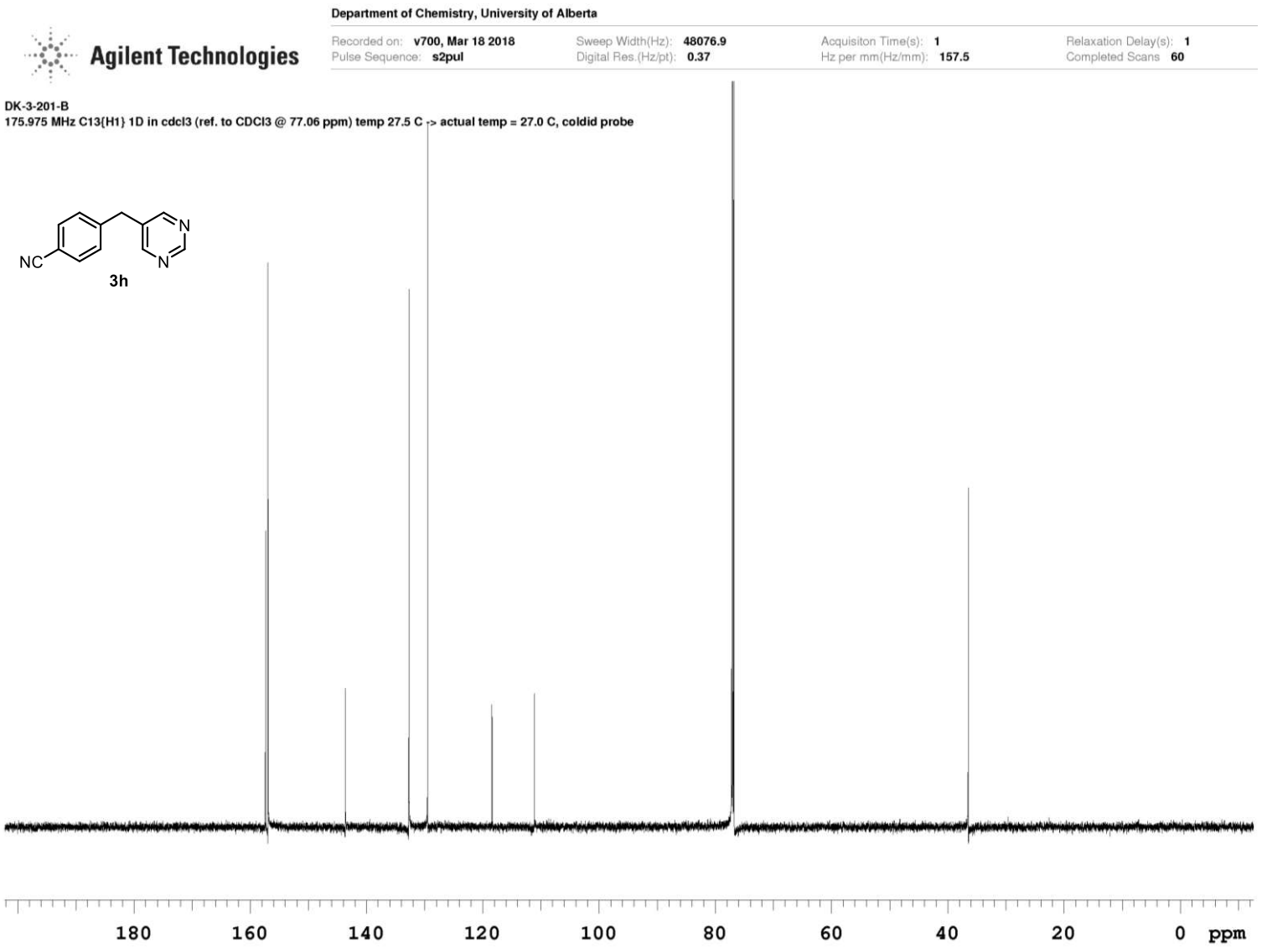
PM-14-165-A
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.5 C -> actual temp = 27.0 C, coldid probe



3g




Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: ibd5, Mar 18 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 6000.6
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 21.18
Relaxation Delay(s): 0.1
Completed Scans 16
DK-3-201-B
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
NC
N
N
3h
9
8
7
6
5
4
3
2
1
ppm
0.94
2.00
2.13
2.14
0.26
2.23


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: v700, Mar 18 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 48076.9
Digital Res.(Hz/pt): 0.37
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 157.5
Relaxation Delay(s): 1
Completed Scans 60
DK-3-201-B
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe
NC
N
N
3h
180
160
140
120
100
80
60
40
20
0
ppm

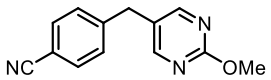
Department of Chemistry, University of Alberta

Agilent Technologies

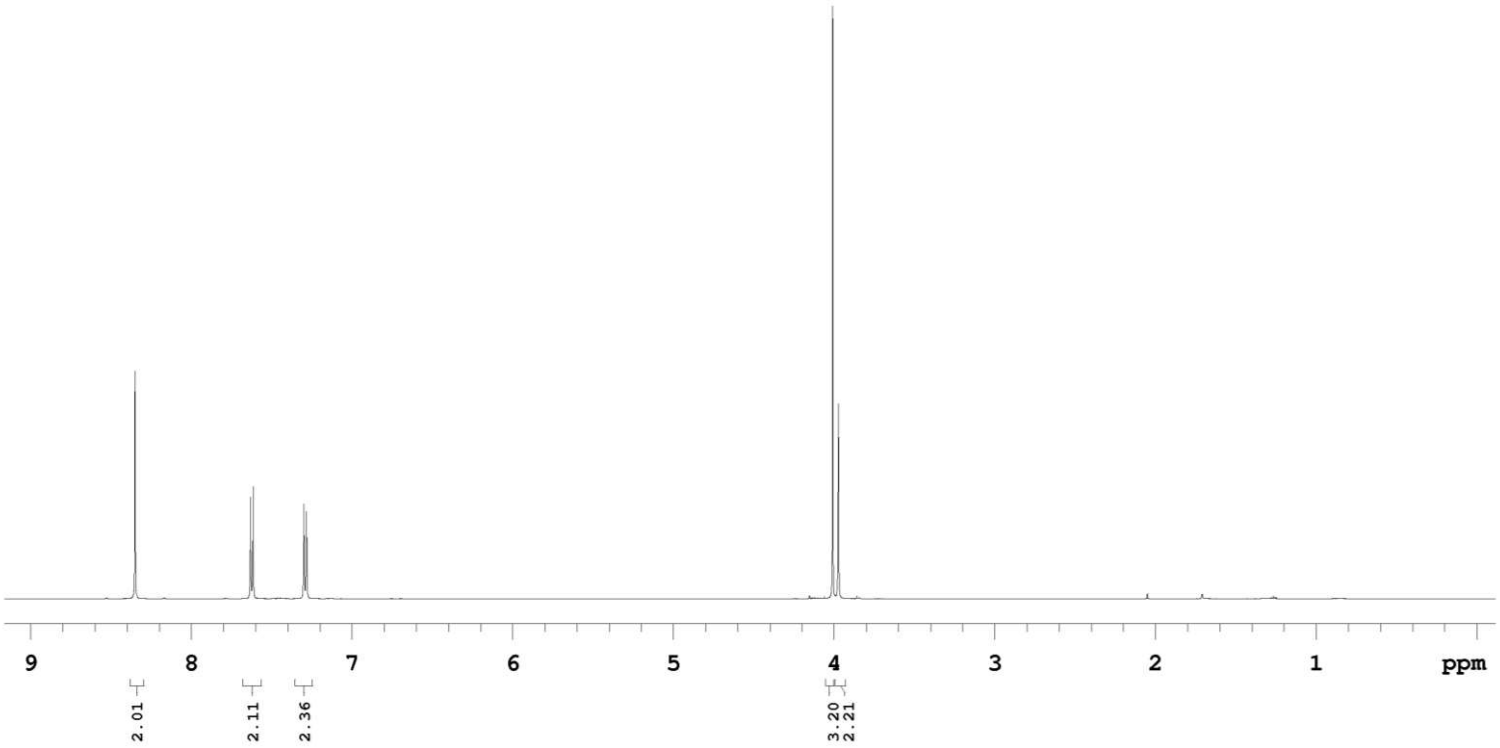
Recorded on: ibd5, Mar 17 2018	Sweep Width(Hz): 6000.6	Acquisiton Time(s): 5	Relaxation Delay(s): 0.1
Pulse Sequence: s2pul	Digital Res.(Hz/pt): 0.09	Hz per mm(Hz/mm): 19.25	Completed Scans 16

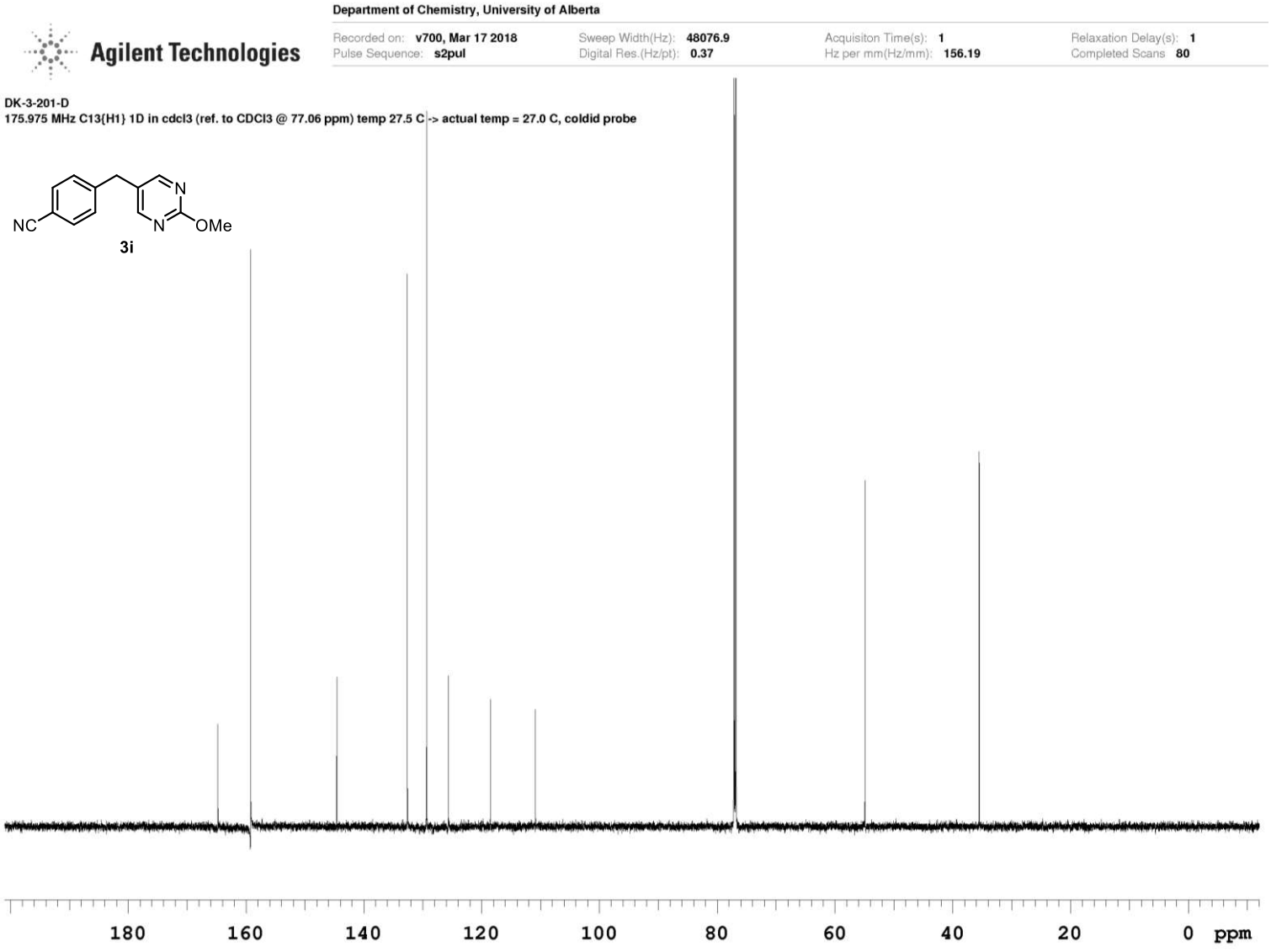
DK-3-201-D

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



3i



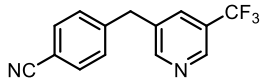

Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: v700, Mar 17 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 48076.9
Digital Res.(Hz/pt): 0.37
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 156.19
Relaxation Delay(s): 1
Completed Scans 80
DK-3-201-D
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe
NC
N
N
OMe
3i
180
160
140
120
100
80
60
40
20
0
ppm

Department of Chemistry, University of Alberta

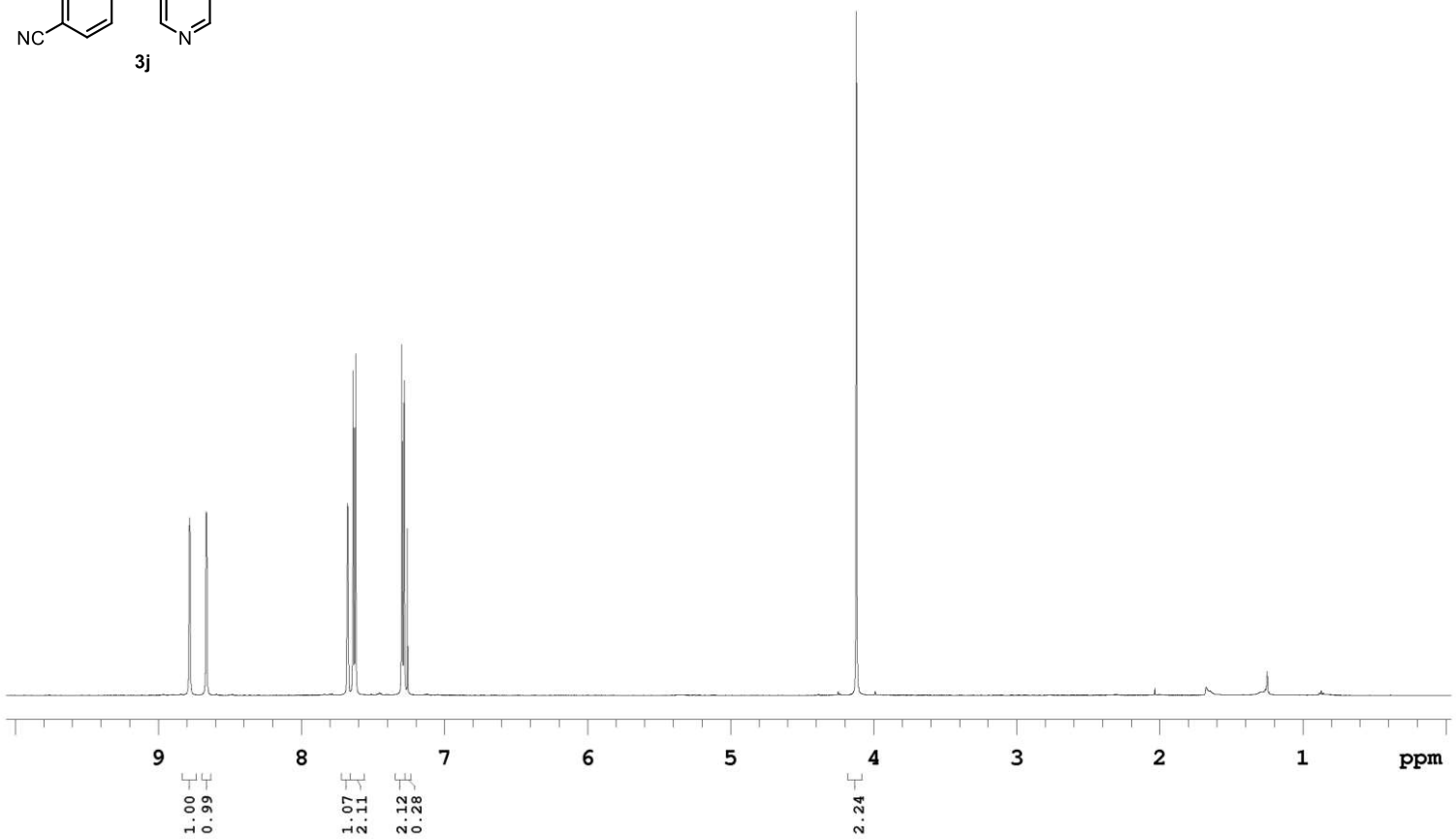
Agilent Technologies

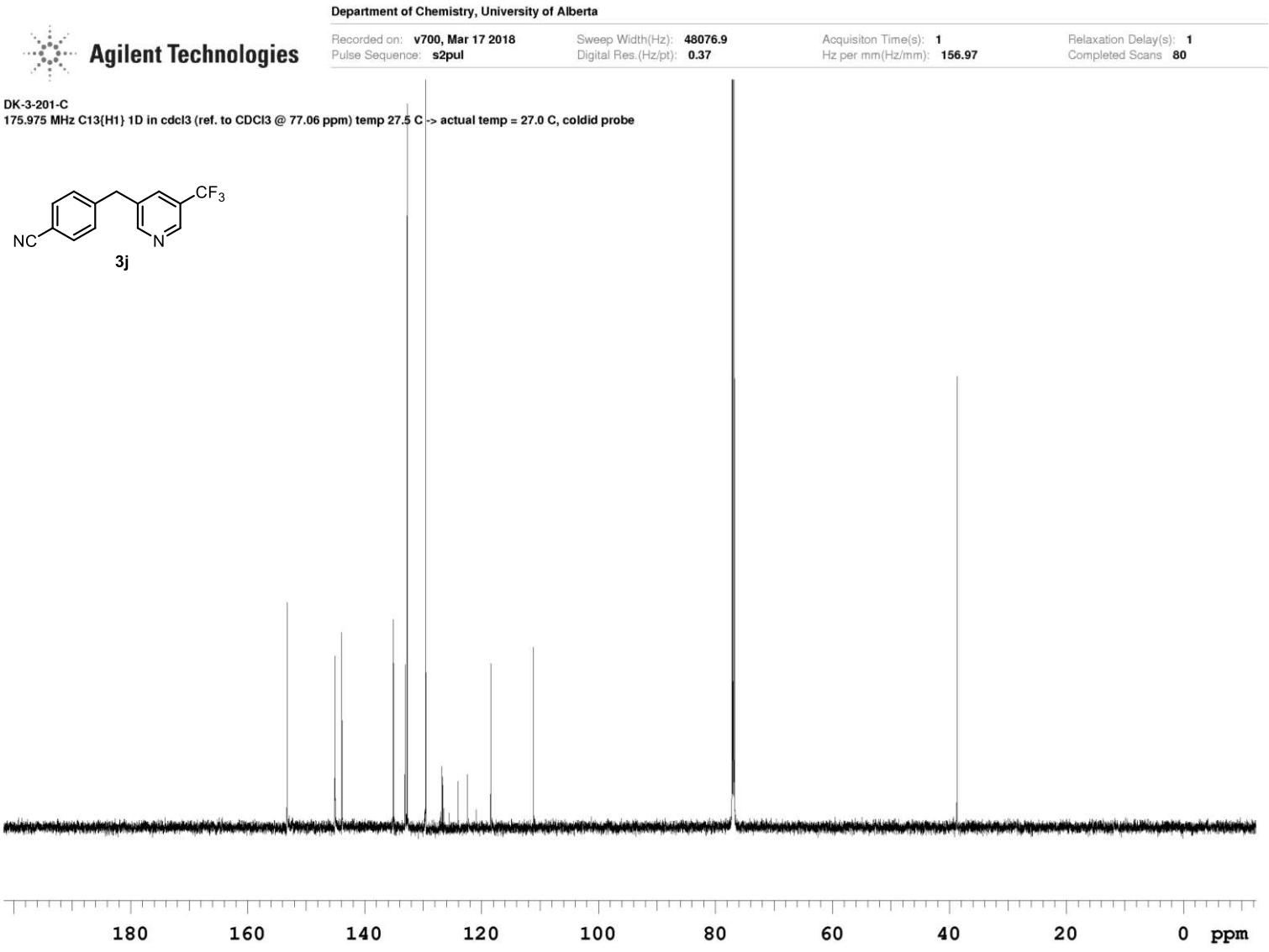
Recorded on: **ibd5, Mar 17 2018**	Sweep Width(Hz): **6000.6**	Acquisiton Time(s): **5**	Relaxation Delay(s): **0.1**
Pulse Sequence: **s2pul**	Digital Res.(Hz/pt): **0.09**	Hz per mm(Hz/mm): **20.96**	Completed Scans **16**

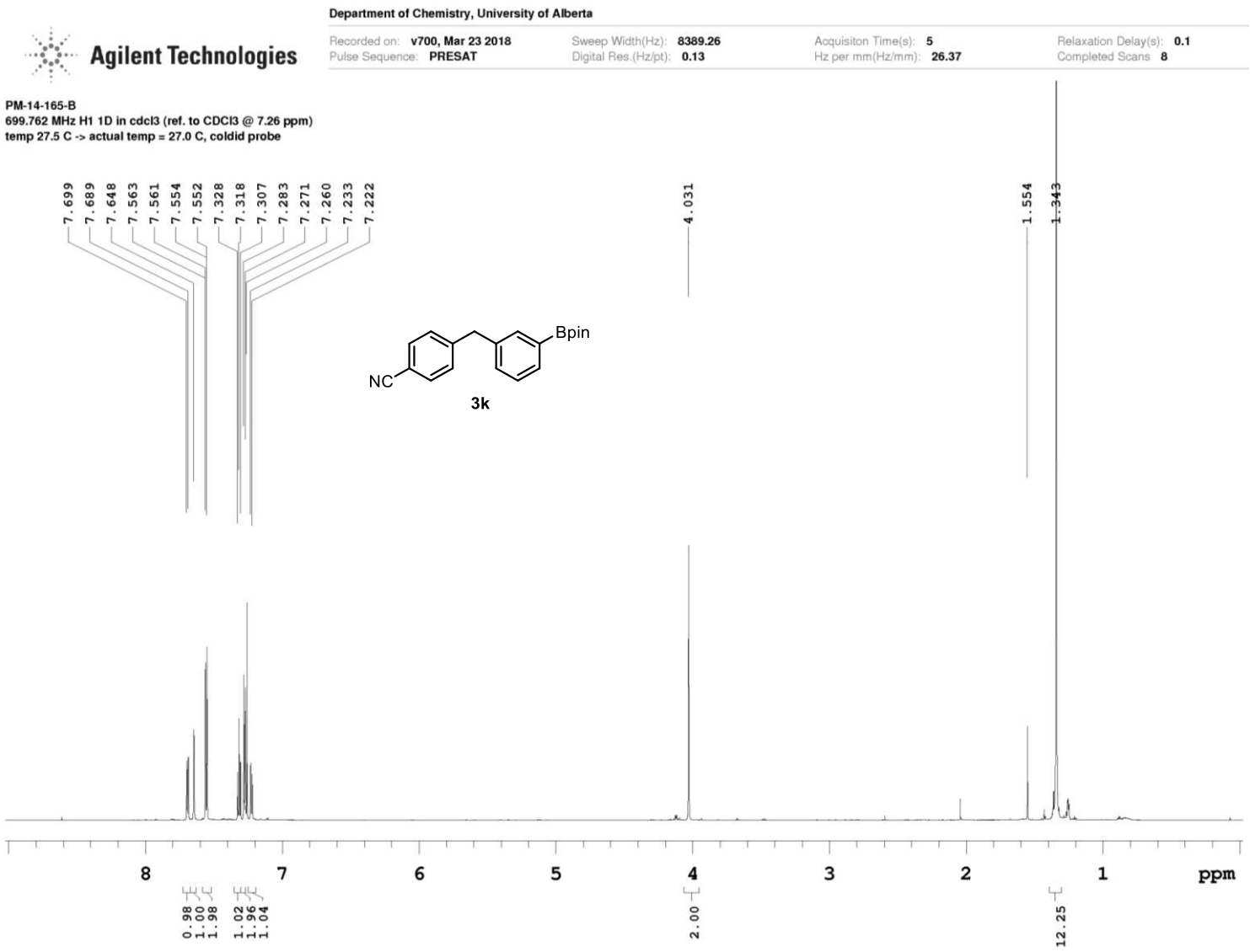
DK-3-201-C
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe

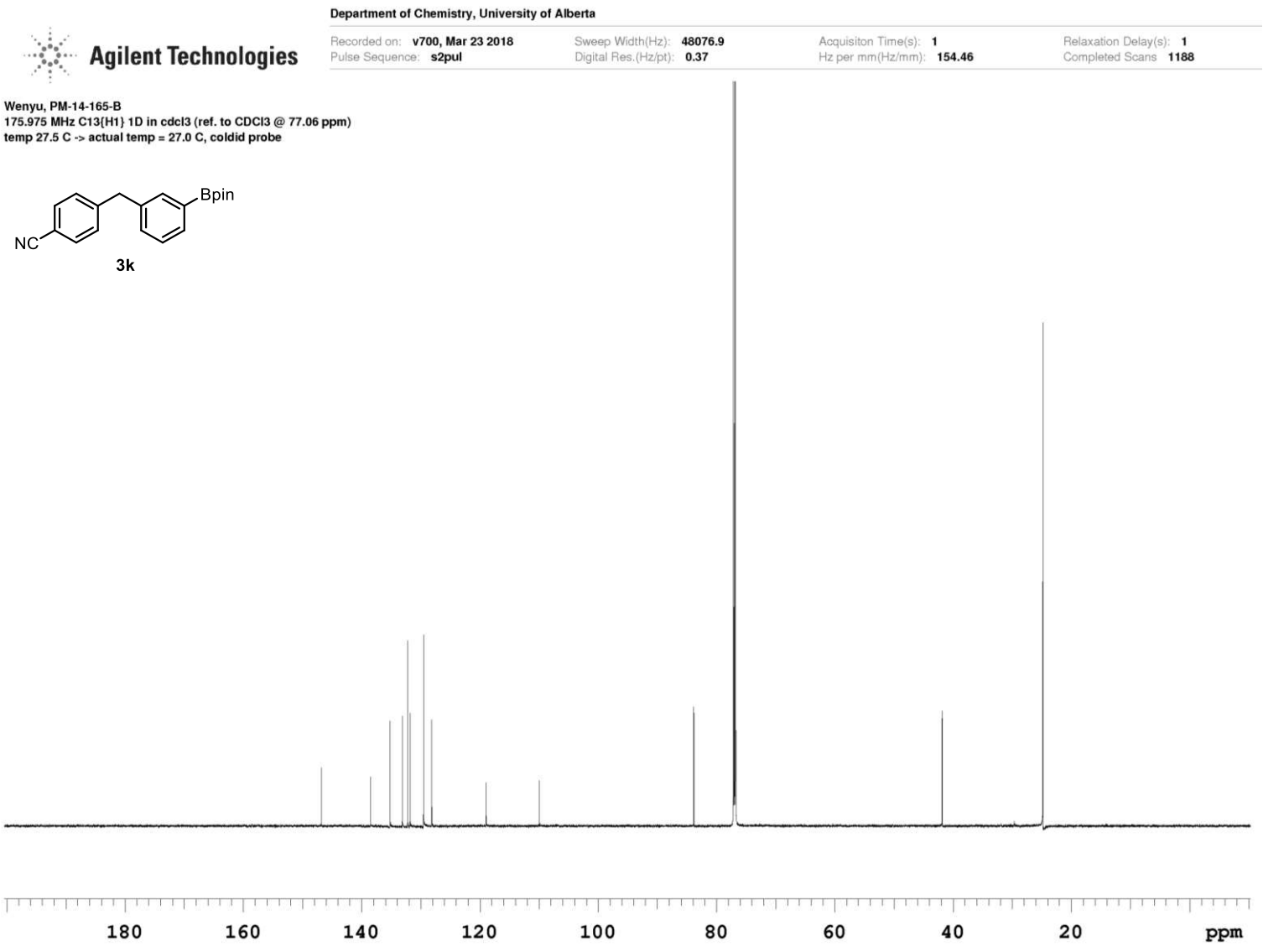


3j




Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: v700, Mar 17 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 48076.9
Digital Res.(Hz/pt): 0.37
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 156.97
Relaxation Delay(s): 1
Completed Scans 80
DK-3-201-C
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe
CF3
NC
N
3j
180
160
140
120
100
80
60
40
20
0
ppm


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: v700, Mar 23 2018
Pulse Sequence: PRESAT
Sweep Width(Hz): 8389.26
Digital Res.(Hz/pt): 0.13
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 26.37
Relaxation Delay(s): 0.1
Completed Scans 8
PM-14-165-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
7.699
7.689
7.648
7.563
7.561
7.554
7.552
7.328
7.318
7.307
7.283
7.271
7.260
7.233
7.222
4.031
1.554
1.343
Bpin
NC
3k
8
7
6
5
4
3
2
1
ppm
0.98
1.00
1.98
1.02
1.96
1.04
2.00
12.25

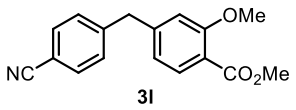

Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: v700, Mar 23 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 48076.9
Digital Res.(Hz/pt): 0.37
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 154.46
Relaxation Delay(s): 1
Completed Scans 1188
Wenyu, PM-14-165-B
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.5 C -> actual temp = 27.0 C, coldid probe
Bpin
NC
3k
180
160
140
120
100
80
60
40
20
ppm

Agilent Technologies

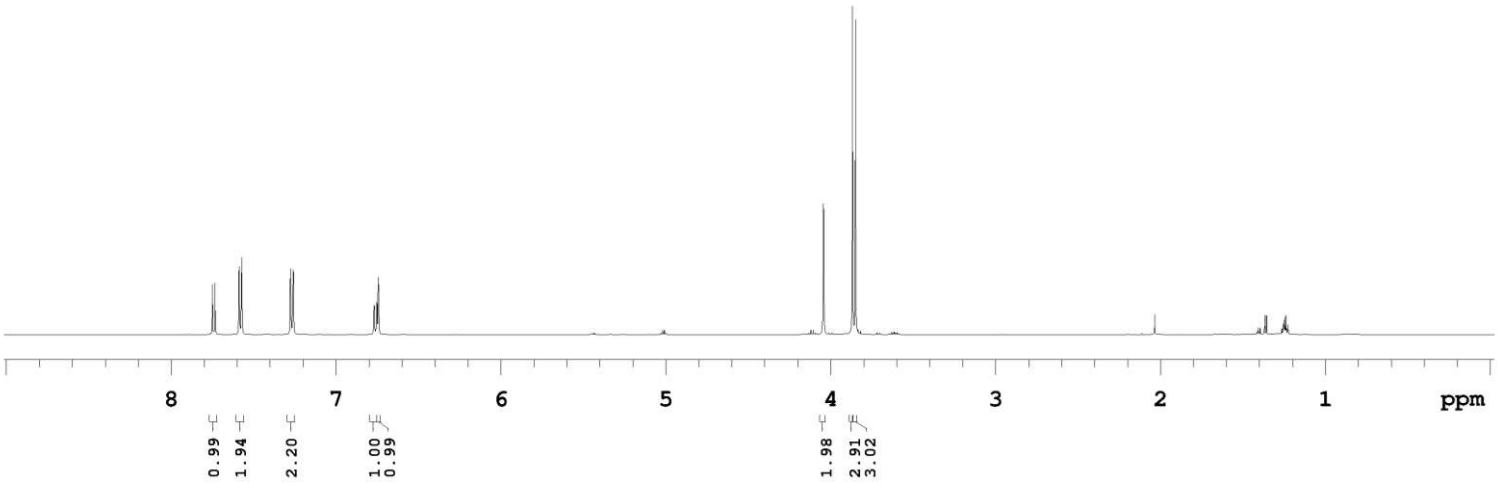
Department of Chemistry, University of Alberta

Recorded on: **u500, Mar 22 2018**	Sweep Width(Hz): **6009.62**	Acquisiton Time(s): **5**	Relaxation Delay(s): **0.1**
Pulse Sequence: **PRESAT**	Digital Res.(Hz/pt): **0.09**	Hz per mm(Hz/mm): **18.82**	Completed Scans **4**

PM-14-165-C
499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe



3l

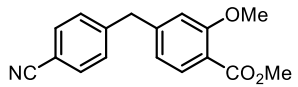


Agilent Technologies

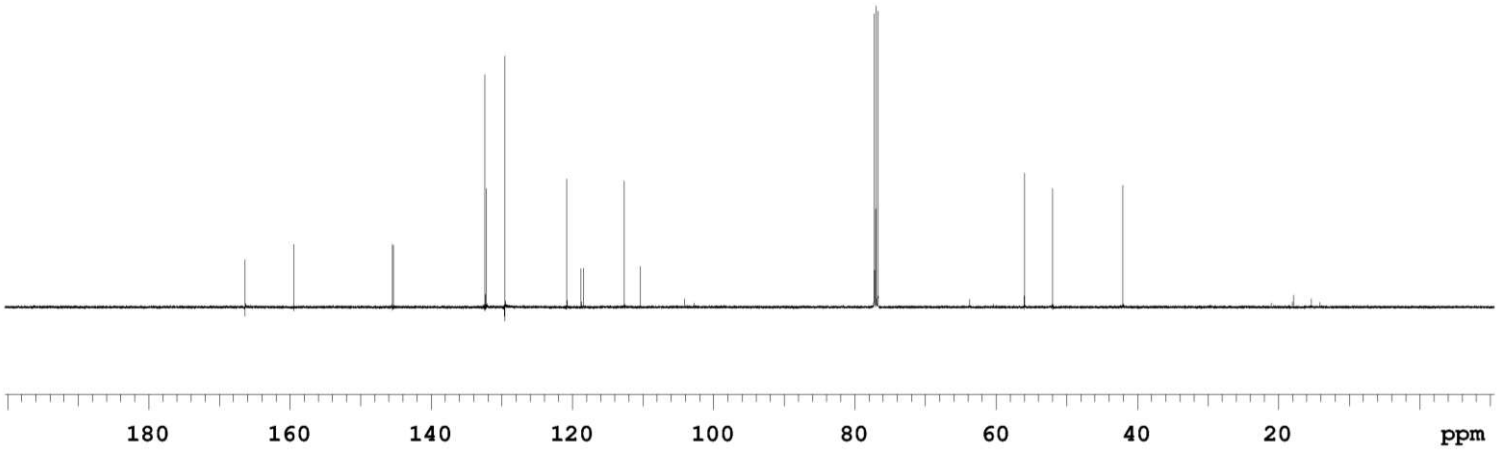
Department of Chemistry, University of Alberta

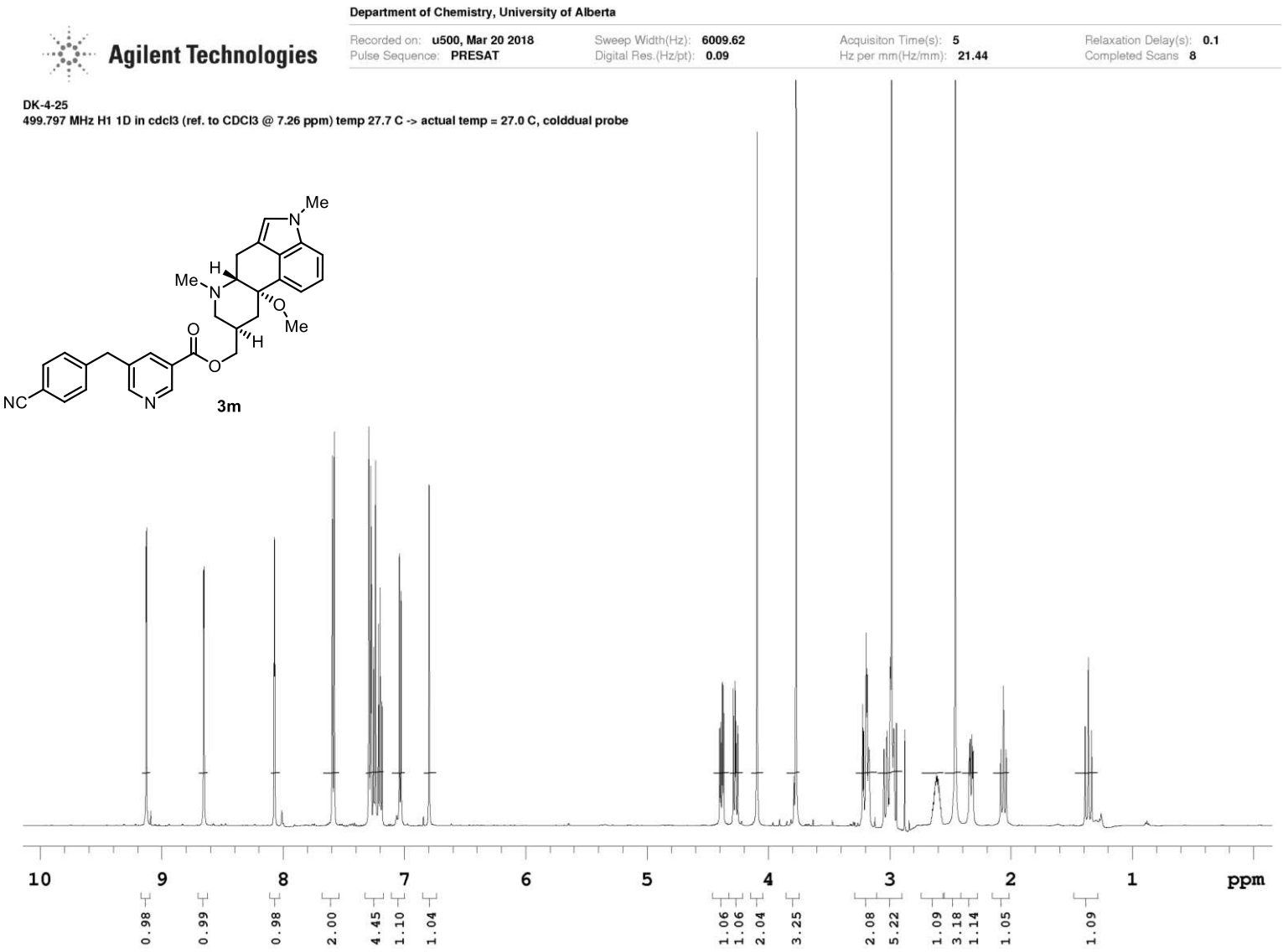
Recorded on: u500, Mar 22 2018	Sweep Width(Hz): 33783.8	Acquisiton Time(s): 1	Relaxation Delay(s): 1
Pulse Sequence: s2pul	Digital Res.(Hz/pt): 0.26	Hz per mm(Hz/mm): 110.47	Completed Scans 64

PM-14-165-C
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe



3l




Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 20 2018
Pulse Sequence: PRESAT
Sweep Width(Hz): 6009.62
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 21.44
Relaxation Delay(s): 0.1
Completed Scans 8
DK-4-25
499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
Me
N
H
Me
N
O
Me
O
H
O
NC
N
3m
10
9
8
7
6
5
4
3
2
1
ppm
0.98
0.99
0.98
2.00
4.45
1.10
1.04
1.06
1.06
2.04
3.25
2.08
5.22
1.09
3.18
1.14
1.05
1.09

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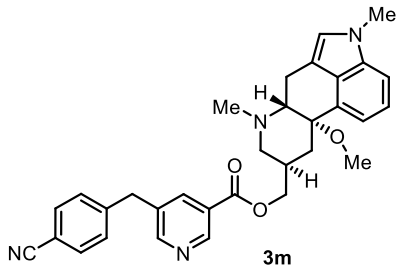
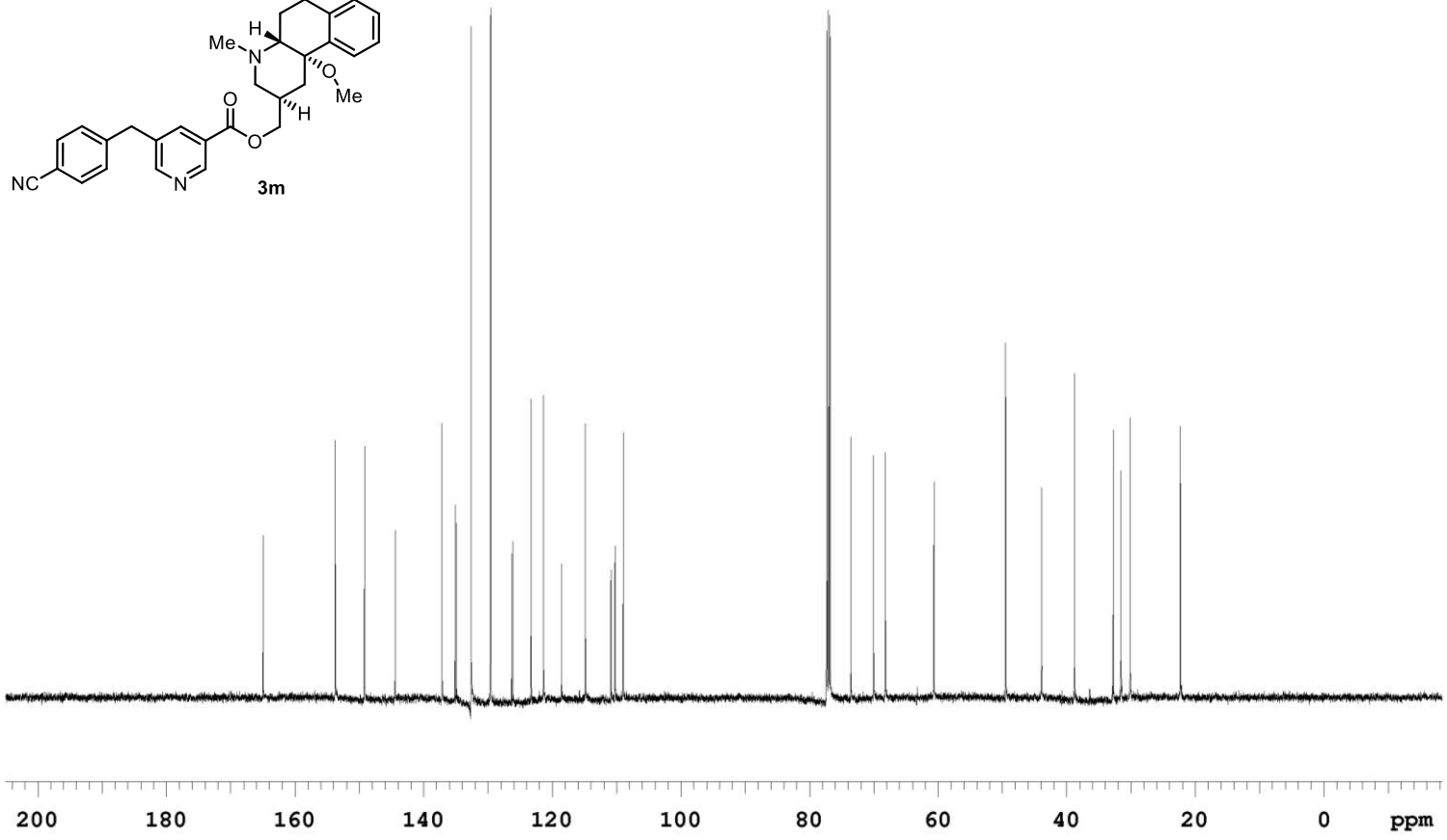
Recorded on:	u500, Mar 20 2018	Sweep Width(Hz):	33783.8	Acquisiton Time(s):	1	Relaxation Delay(s):	1
Pulse Sequence:	s2pul	Digital Res.(Hz/pt):	0.26	Hz per mm(Hz/mm):	116.98	Completed Scans	48

DK-4-25

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



3m

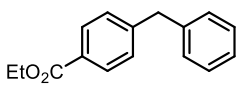


Agilent Technologies

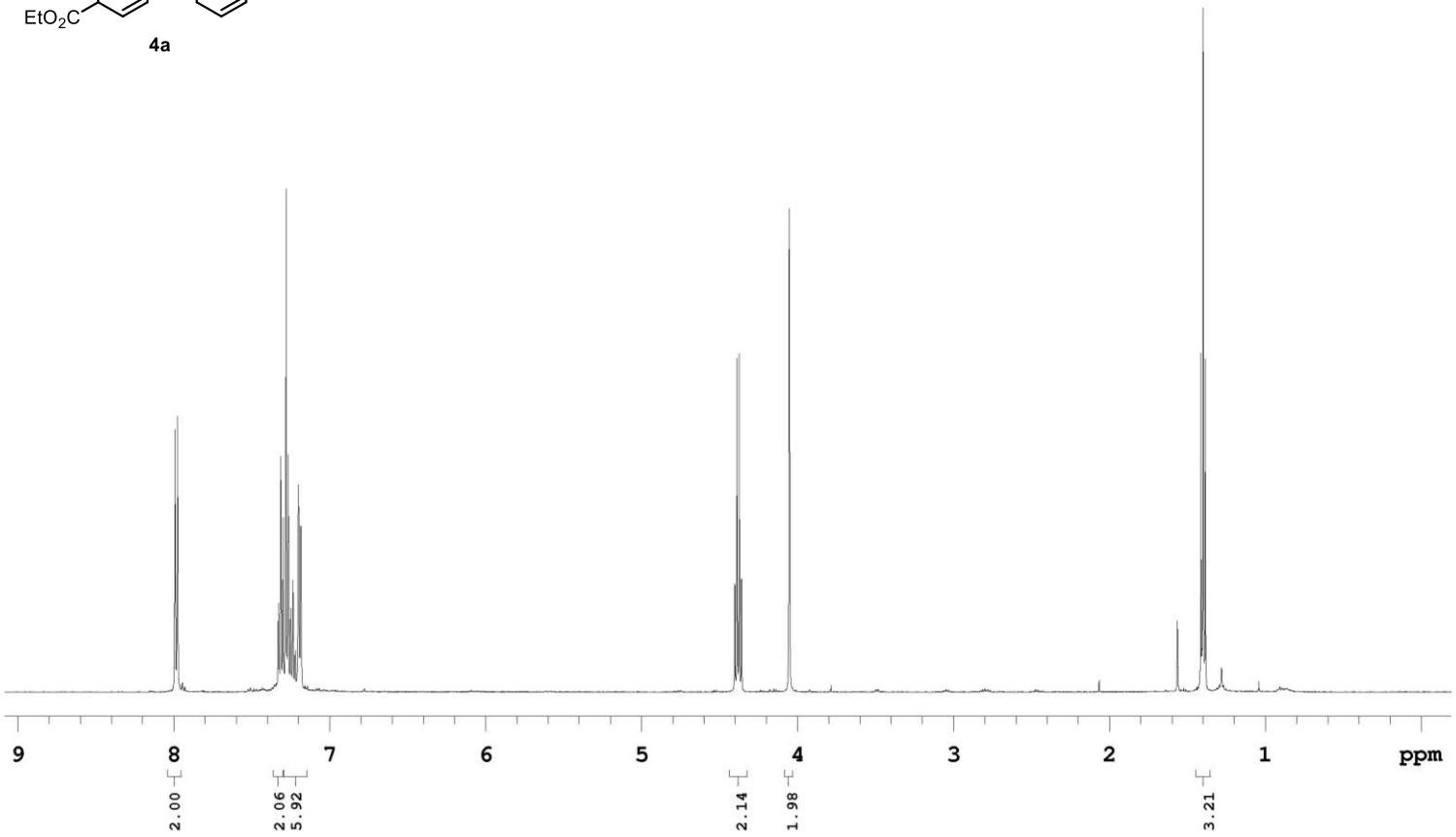
Department of Chemistry, University of Alberta

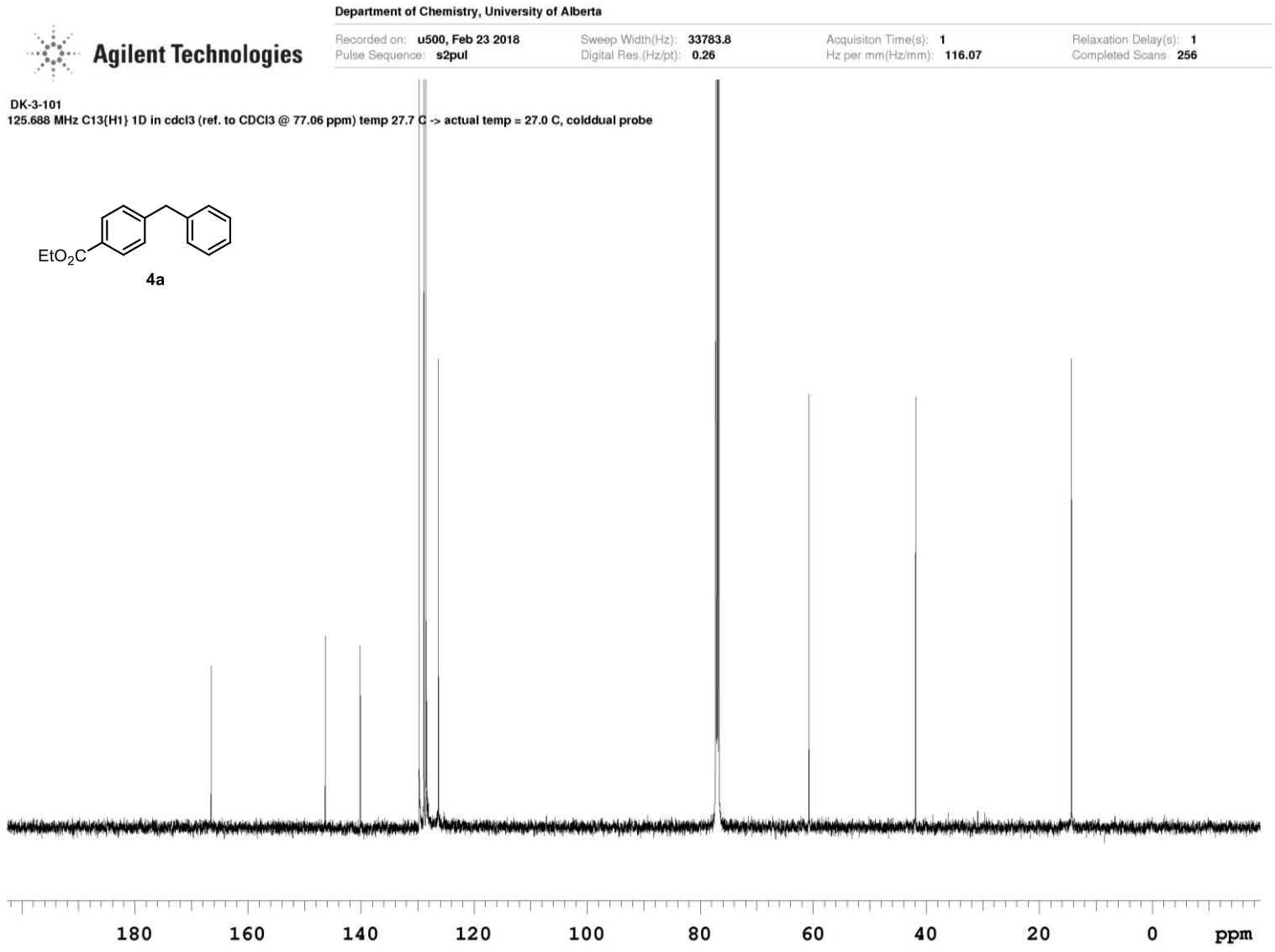
Recorded on:	ibd5, Feb 23 2018	Sweep Width(Hz):	6000.6	Acquisiton Time(s):	5	Relaxation Delay(s):	0.1
Pulse Sequence:	s2pul	Digital Res.(Hz/pt):	0.09	Hz per mm(Hz/mm):	19.25	Completed Scans	16

DK-3-101
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe



4a




Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Feb 23 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 116.07
Relaxation Delay(s): 1
Completed Scans 256
DK-3-101
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
EtO2C
4a
180
160
140
120
100
80
60
40
20
0
ppm

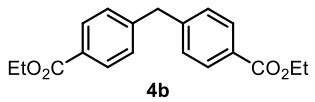
Department of Chemistry, University of Alberta

Agilent Technologies

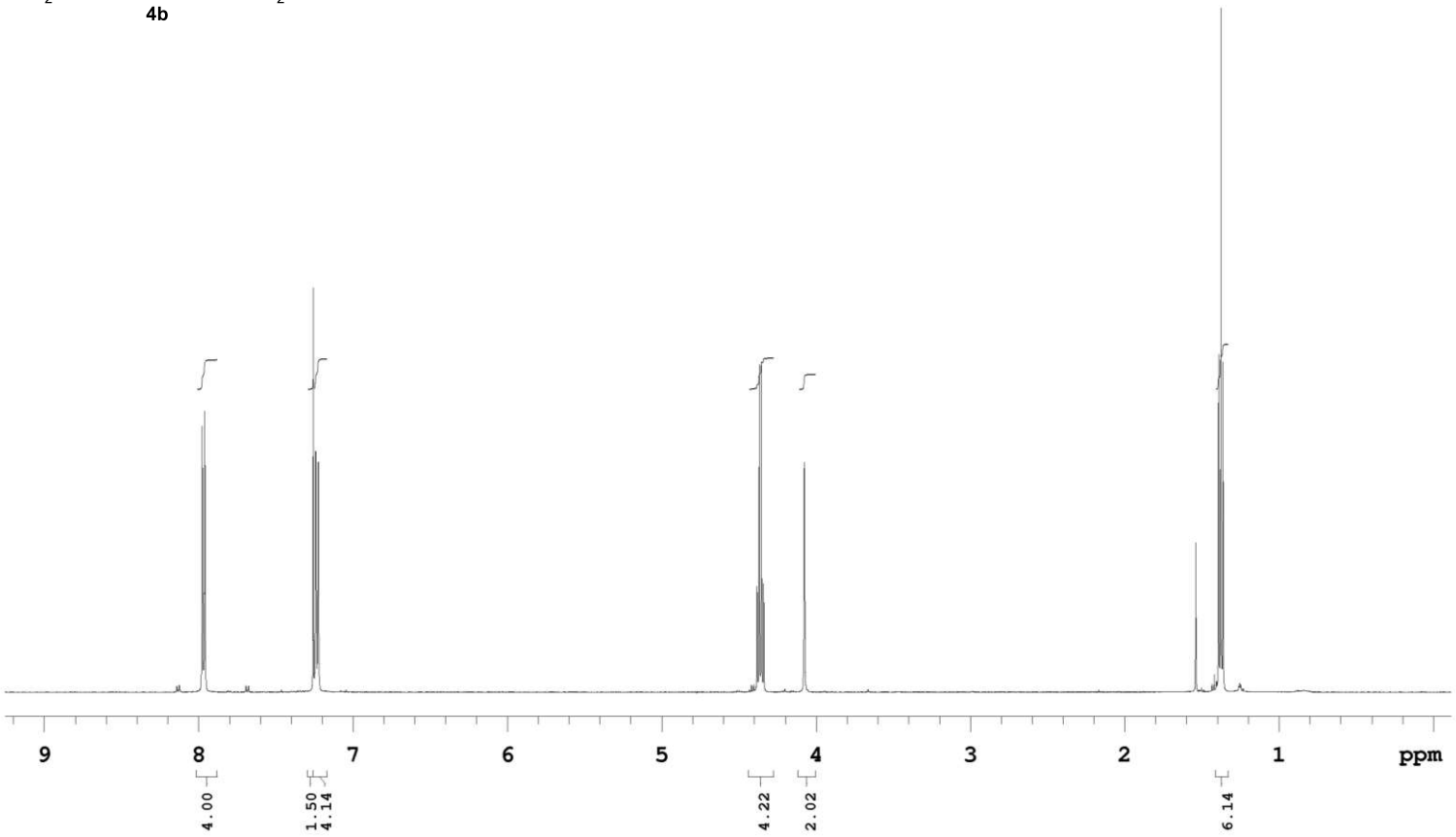
Recorded on: ibd5, Feb 23 2018	Sweep Width(Hz): 6000.6	Acquisiton Time(s): 5	Relaxation Delay(s): 0.1
Pulse Sequence: s2pul	Digital Res.(Hz/pt): 0.09	Hz per mm(Hz/mm): 19.44	Completed Scans 16

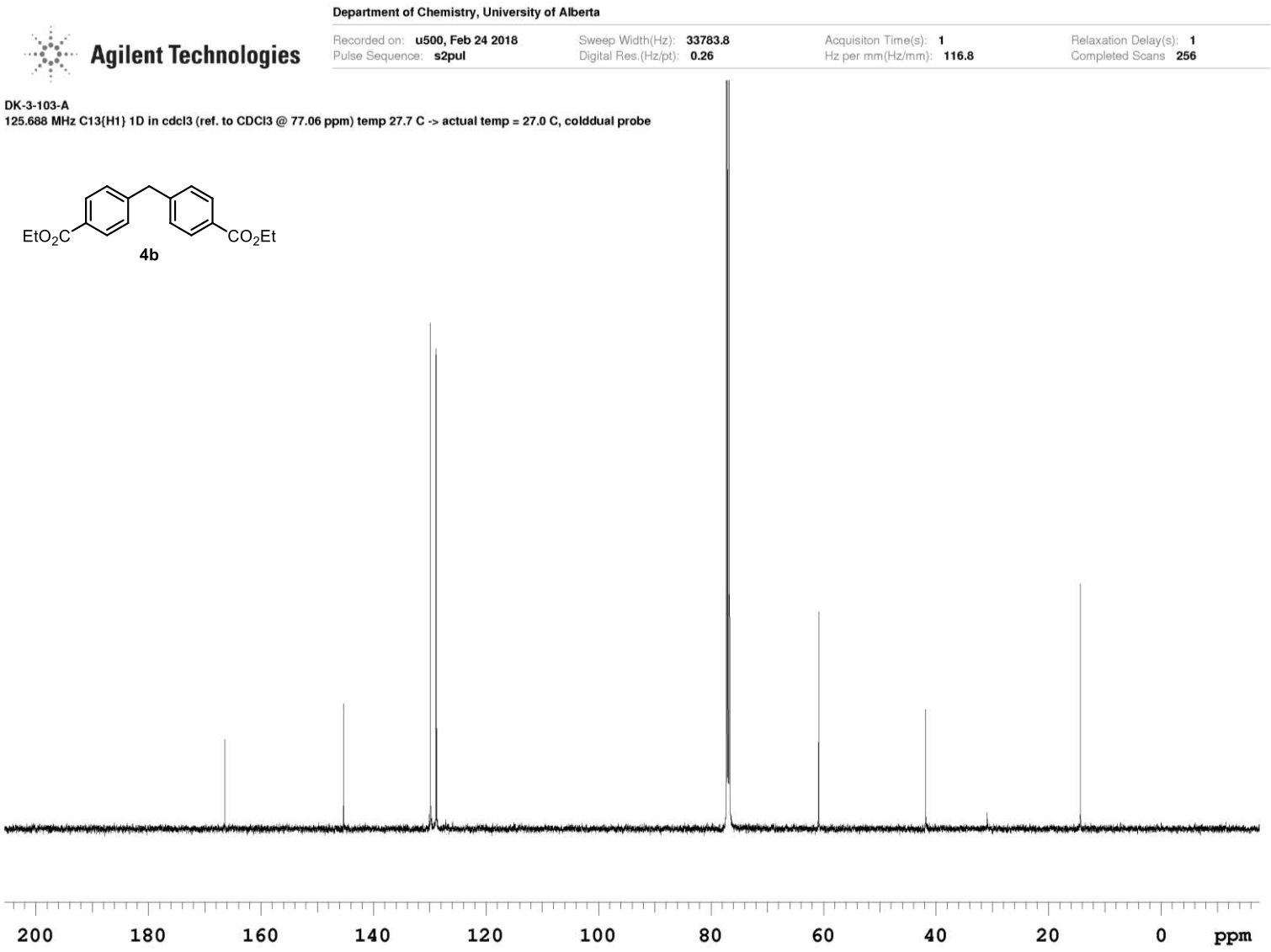
D-3-103-A

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



4b




Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Feb 24 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 116.8
Relaxation Delay(s): 1
Completed Scans 256
DK-3-103-A
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
EtO2C
CO2Et
4b
200
180
160
140
120
100
80
60
40
20
0
ppm

Agilent Technologies

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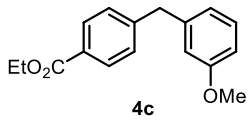
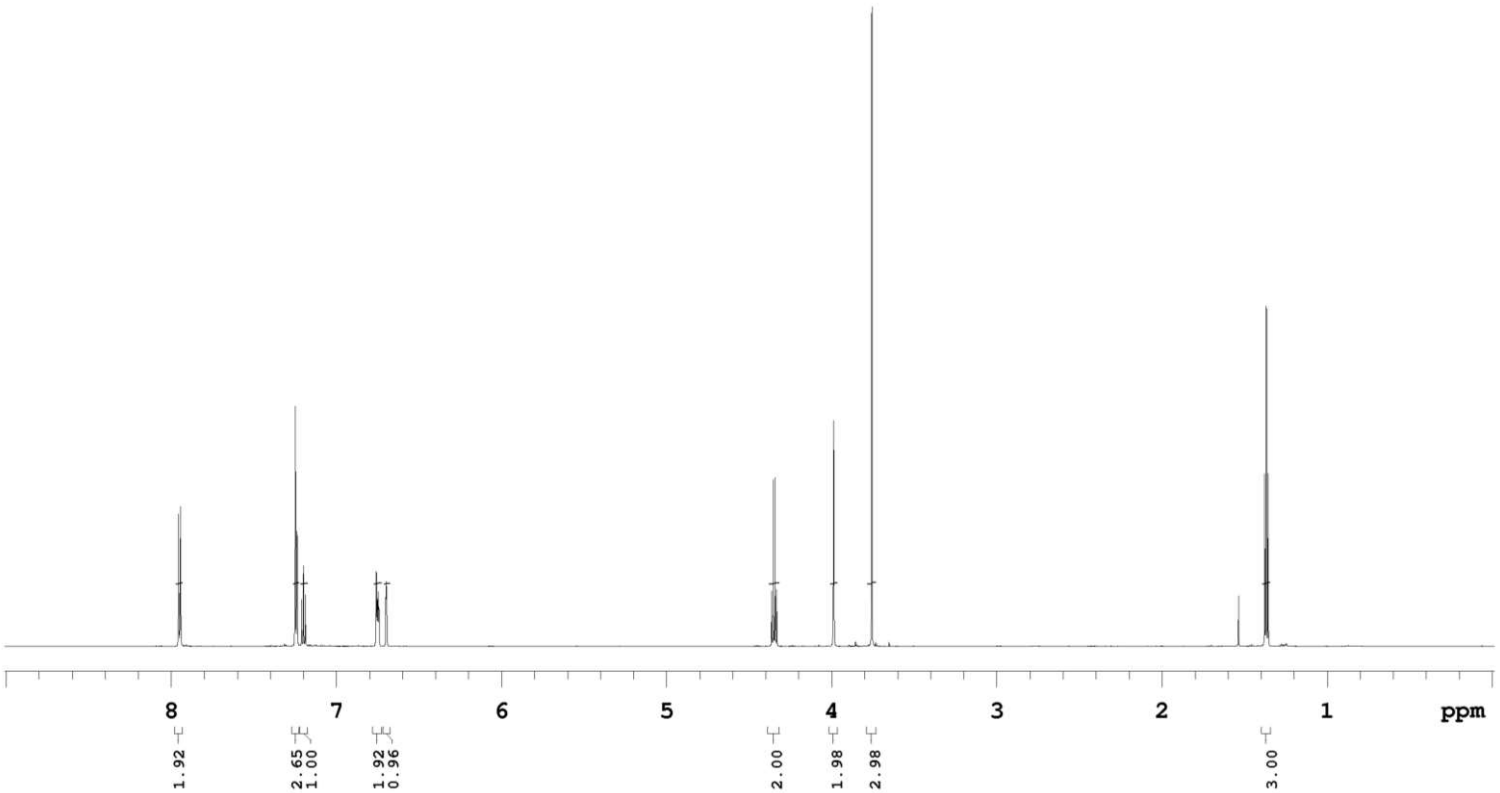
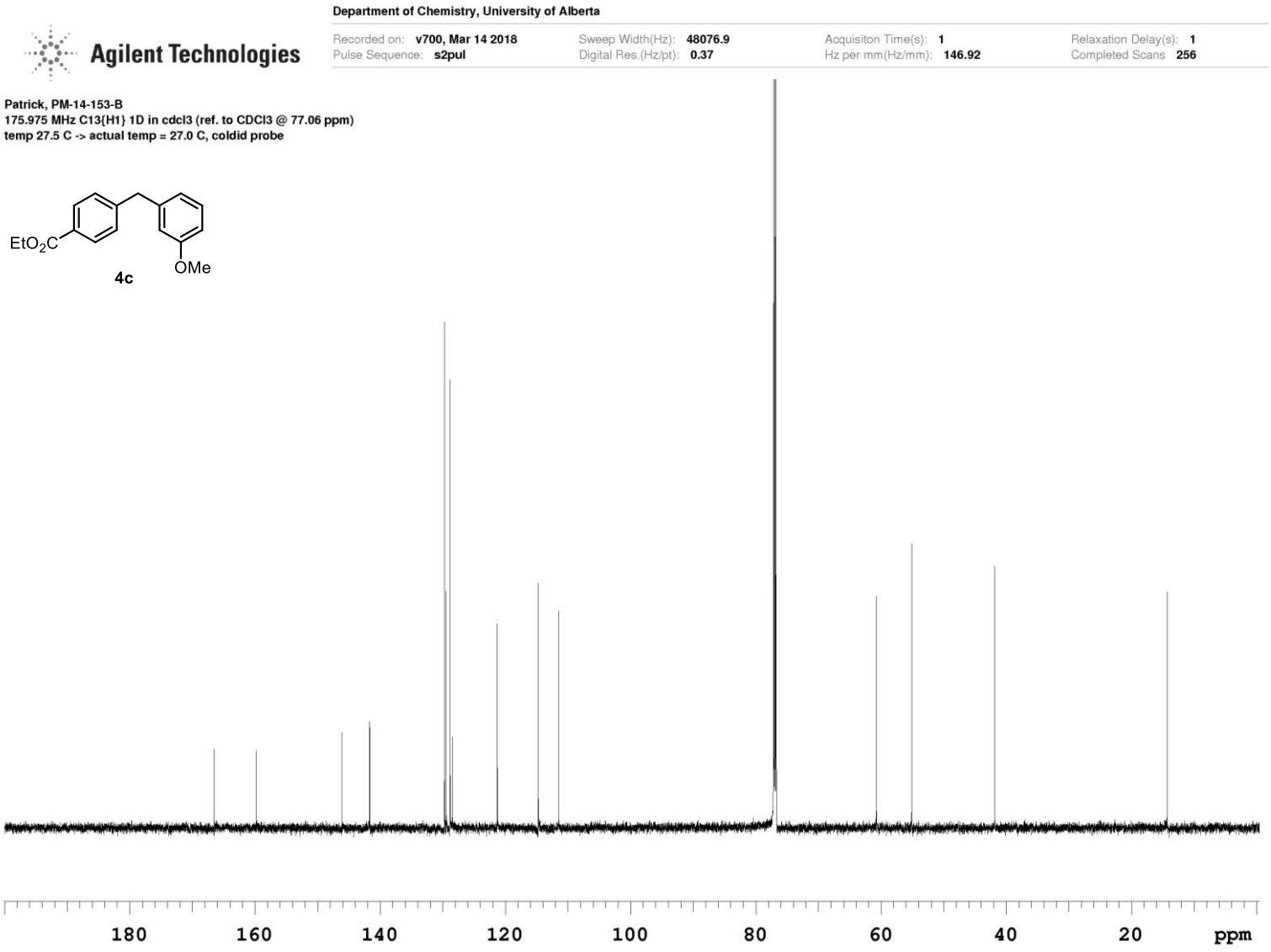
Recorded on:	v700, Mar 14 2018	Sweep Width(Hz):	8389.26	Acquisiton Time(s):	5	Relaxation Delay(s):	0.1
Pulse Sequence:	PRESAT	Digital Res.(Hz/pt):	0.13	Hz per mm(Hz/mm):	26.3	Completed Scans	8

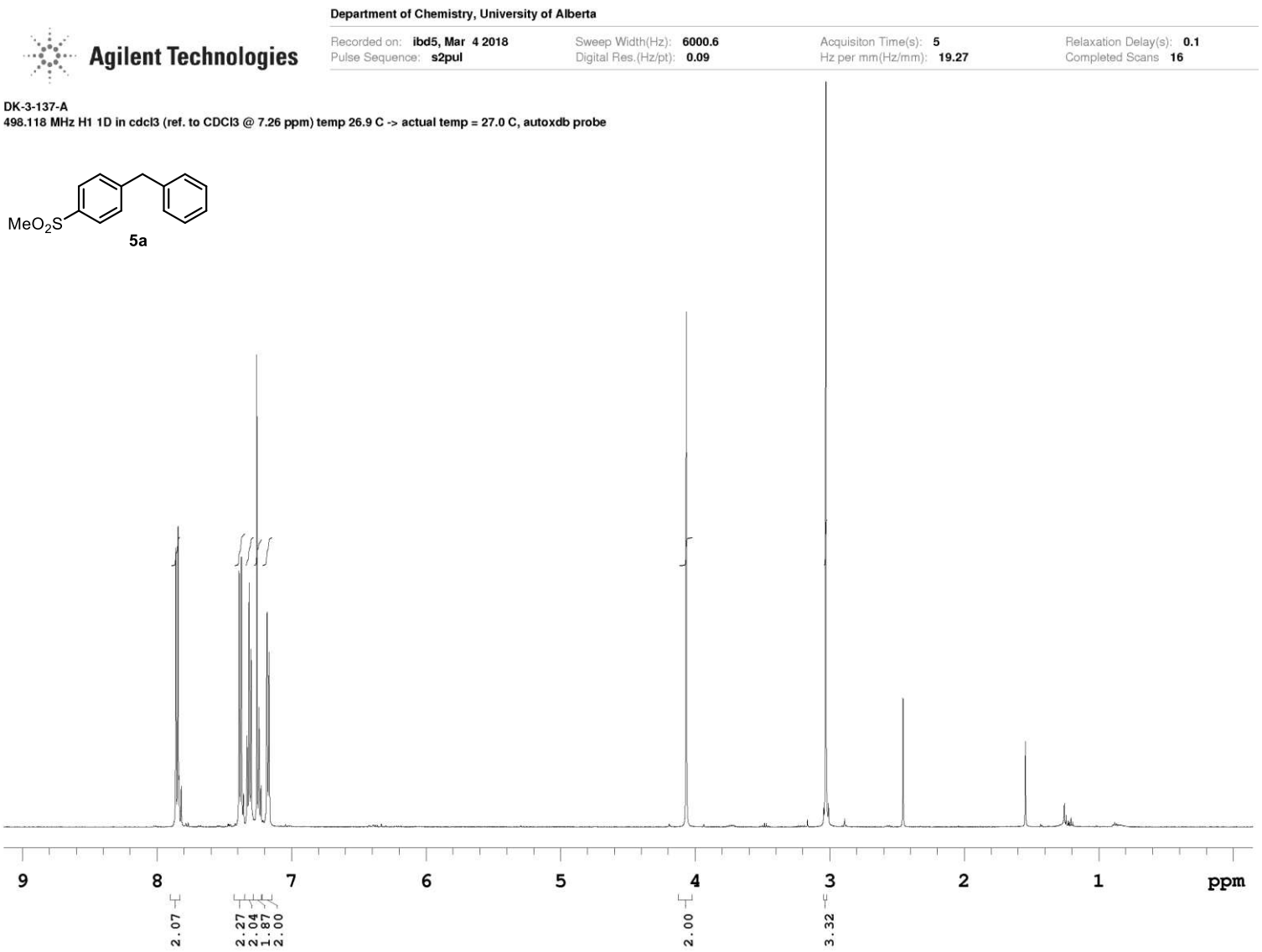
Patrick, PM-14-153-B
699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 27.5 C -> actual temp = 27.0 C, coldid probe

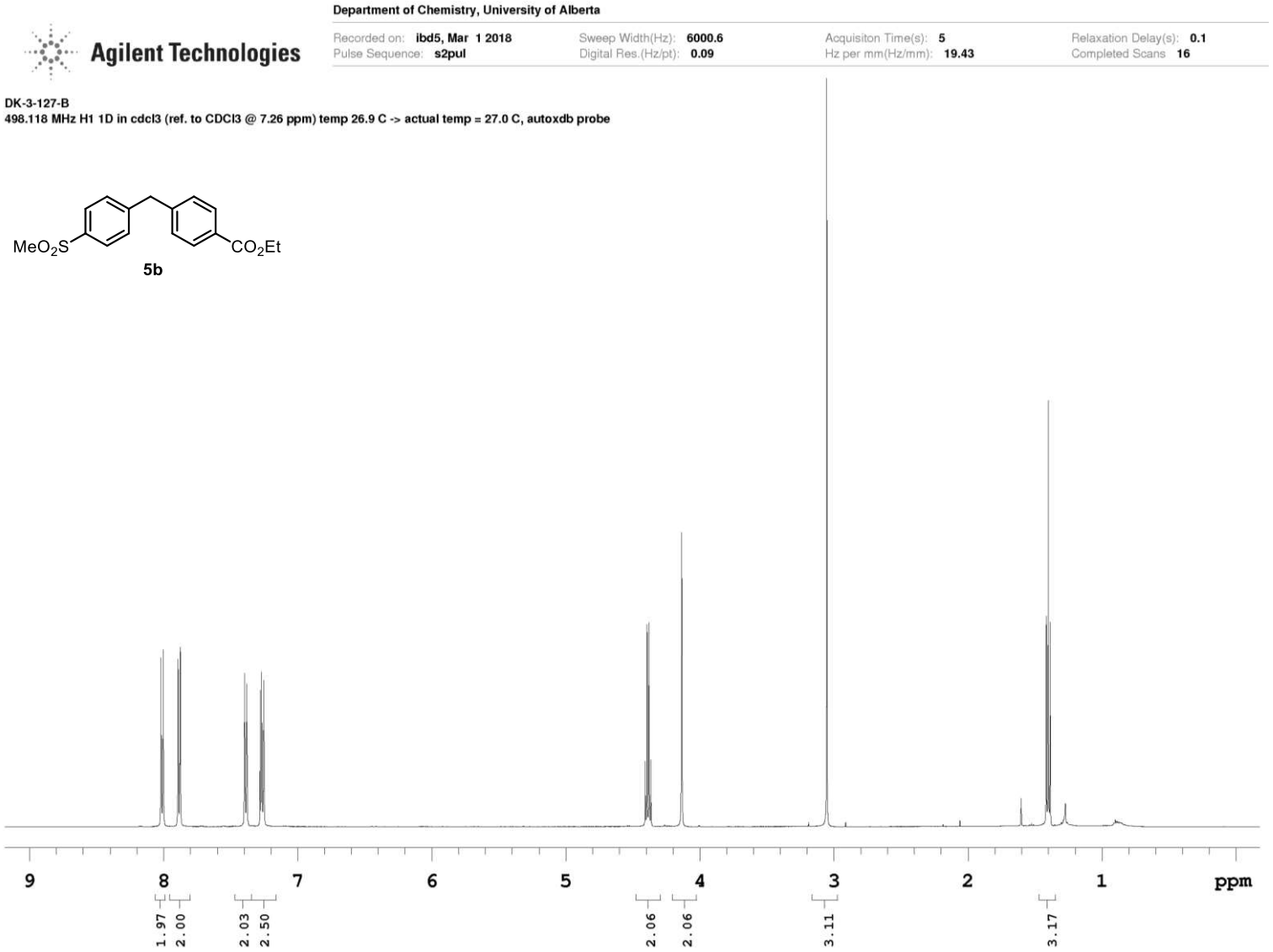


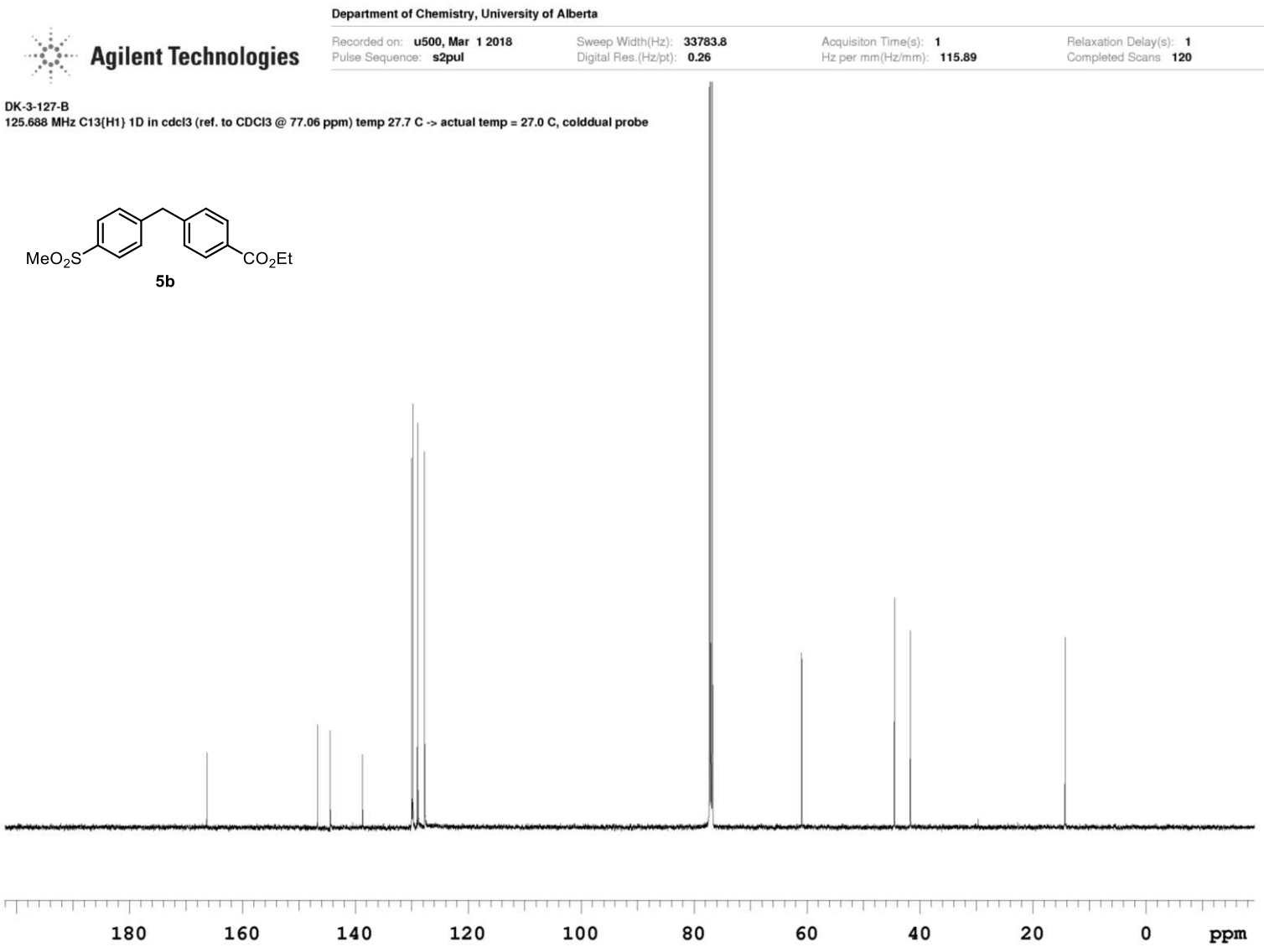
4c

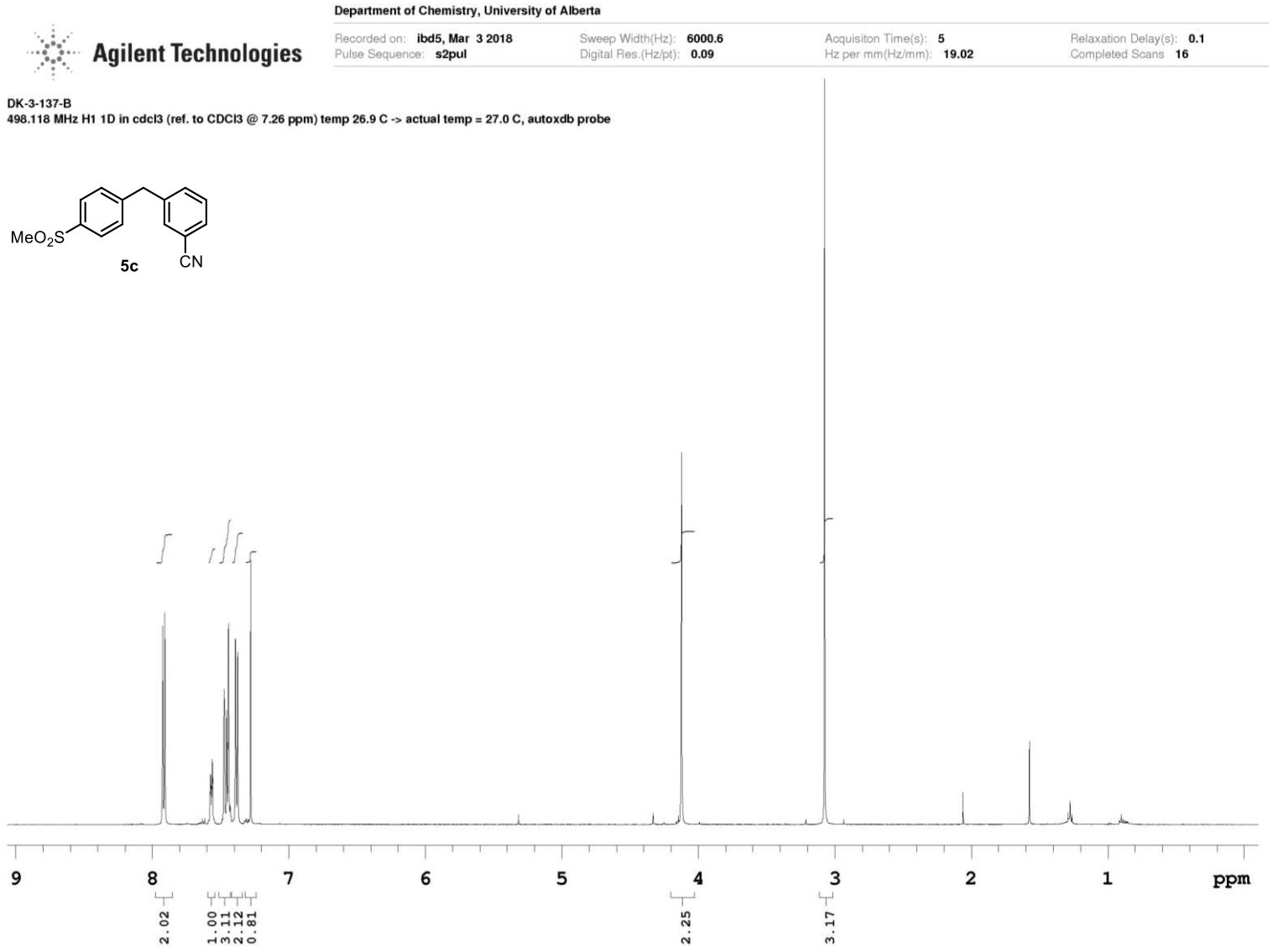


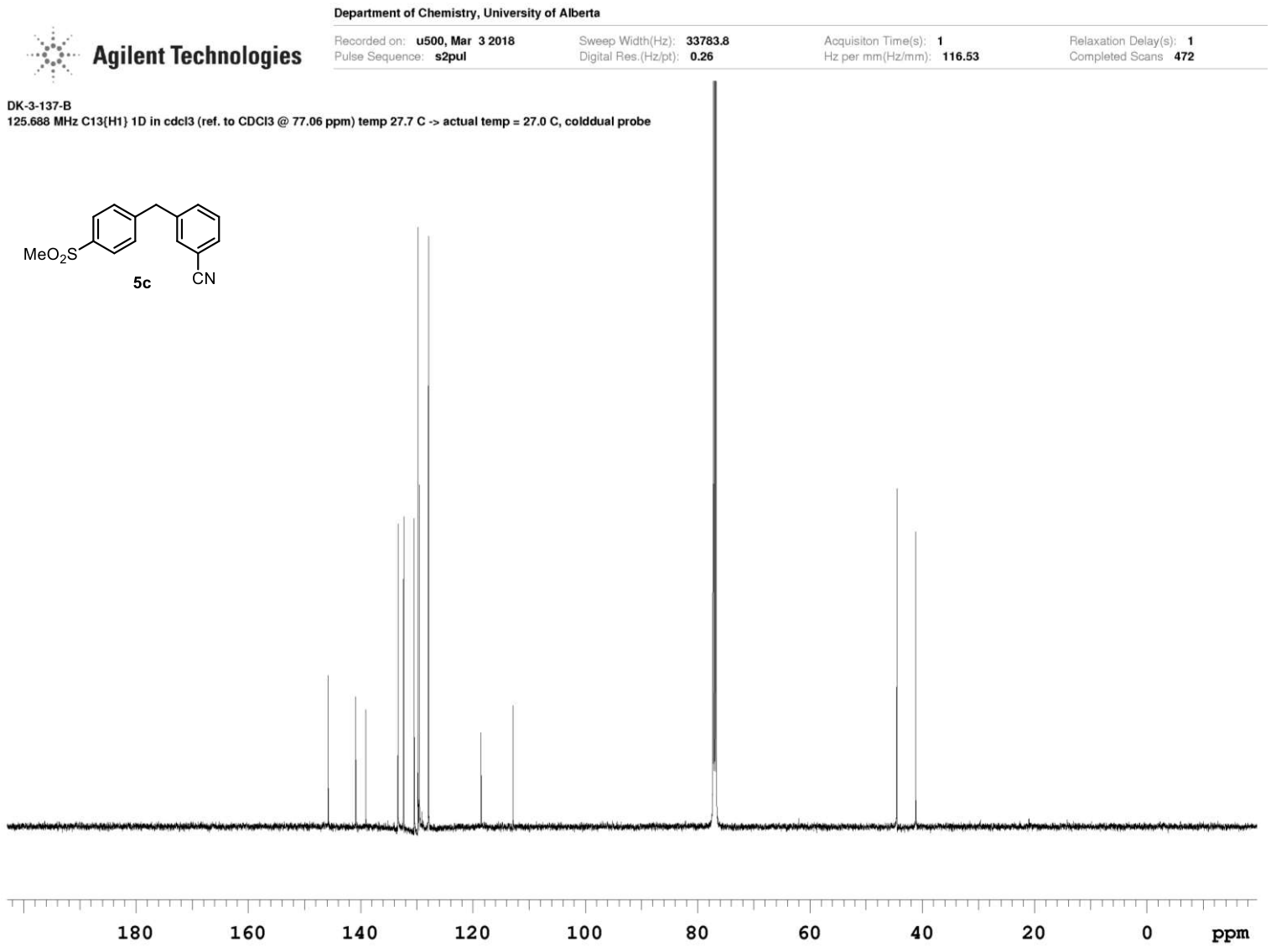

Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: v700, Mar 14 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 48076.9
Digital Res.(Hz/pt): 0.37
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 146.92
Relaxation Delay(s): 1
Completed Scans 256
Patrick, PM-14-153-B
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.5 C -> actual temp = 27.0 C, coldid probe
EtO2C
OMe
4c
180
160
140
120
100
80
60
40
20
ppm

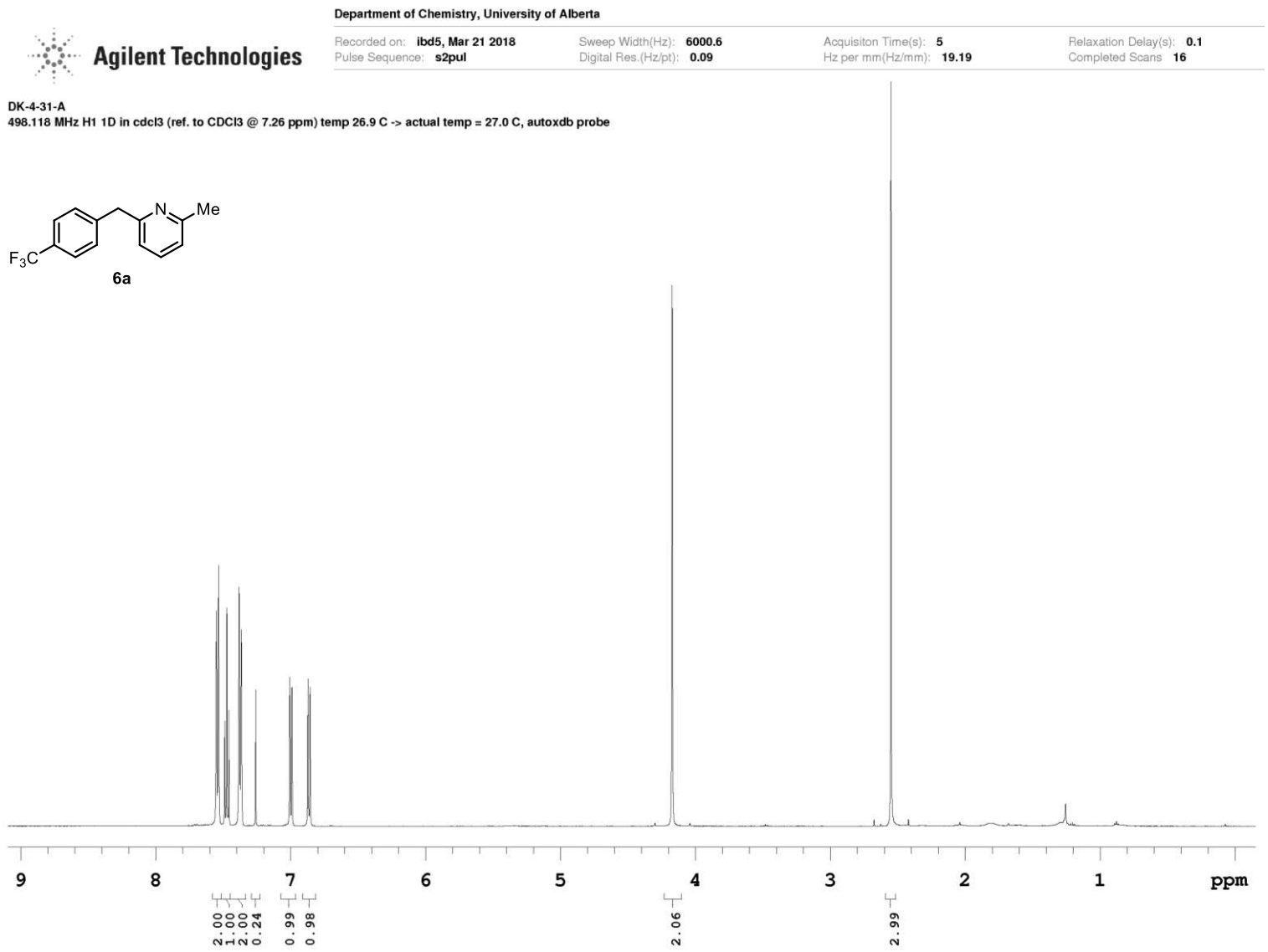

Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: ibd5, Mar 4 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 6000.6
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 19.27
Relaxation Delay(s): 0.1
Completed Scans 16
DK-3-137-A
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
MeO2S
5a
9
8
7
6
5
4
3
2
1
ppm
2.07
2.27
2.04
1.87
2.00
2.00
3.32

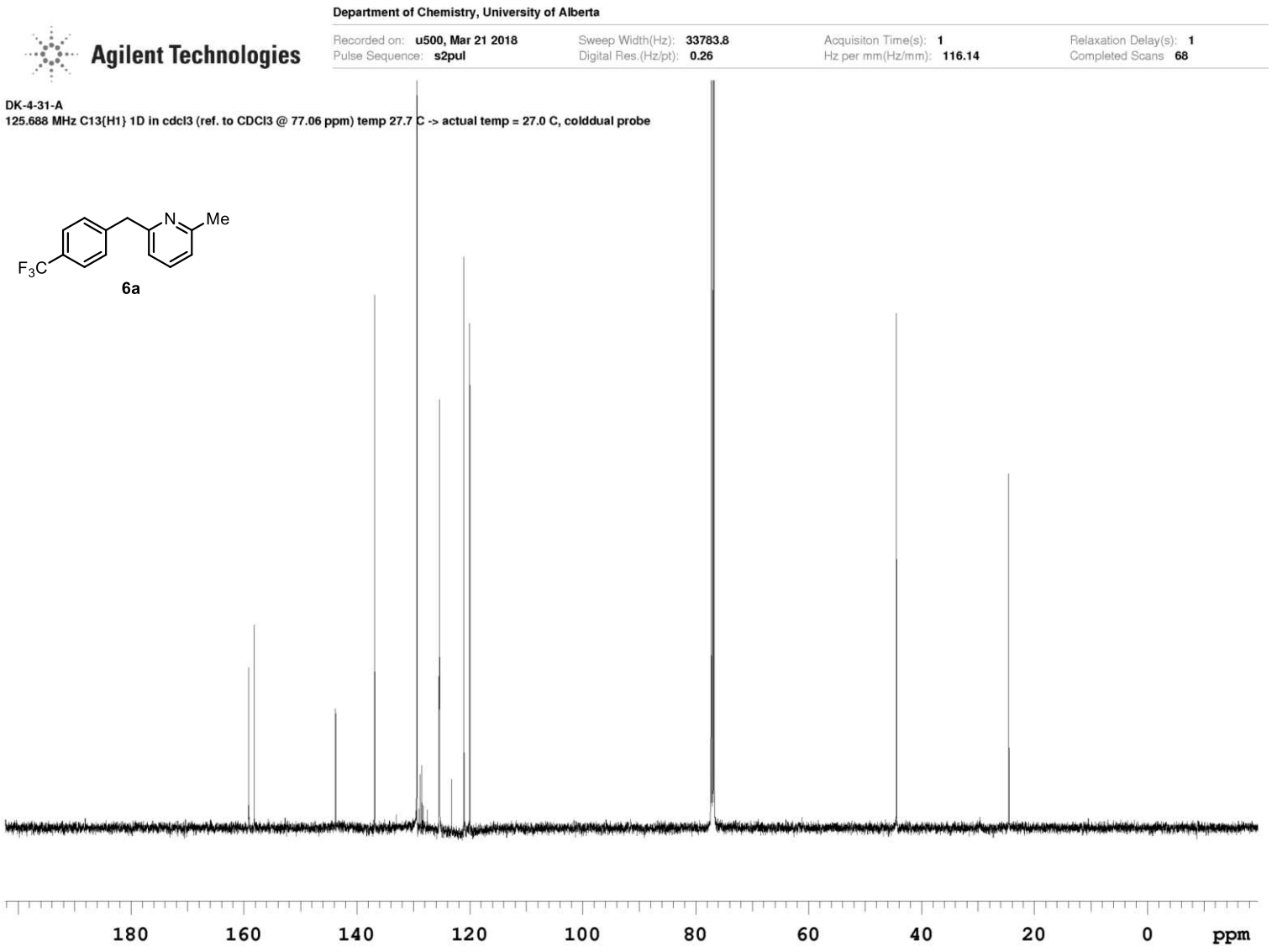

Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: ibd5, Mar 1 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 6000.6
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 19.43
Relaxation Delay(s): 0.1
Completed Scans 16
DK-3-127-B
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
MeO2S
CO2Et
5b
9
8
7
6
5
4
3
2
1
ppm
1.97
2.00
2.03
2.50
2.06
2.06
3.11
3.17


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 1 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 115.89
Relaxation Delay(s): 1
Completed Scans 120
DK-3-127-B
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
MeO2S
CO2Et
5b
180
160
140
120
100
80
60
40
20
0
ppm


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: ibd5, Mar 3 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 6000.6
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 19.02
Relaxation Delay(s): 0.1
Completed Scans 16
DK-3-137-B
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
MeO2S
CN
5c
9
8
7
6
5
4
3
2
1
ppm
2.02
1.00
3.11
2.12
0.81
2.25
3.17


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 3 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 116.53
Relaxation Delay(s): 1
Completed Scans 472
DK-3-137-B
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
MeO2S
CN
5c
180
160
140
120
100
80
60
40
20
0
ppm


Agilent Technologies
Department of Chemistry, University of Alberta
Recorded on: ibd5, Mar 21 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 6000.6
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 19.19
Relaxation Delay(s): 0.1
Completed Scans 16
DK-4-31-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
N
Me
F3C
6a
9
8
7
6
5
4
3
2
1
ppm
2.00
1.00
2.00
0.24
0.99
0.98
2.06
2.99


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 21 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 116.14
Relaxation Delay(s): 1
Completed Scans 68
DK-4-31-A
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
N
Me
F3C
6a
180
160
140
120
100
80
60
40
20
0
ppm

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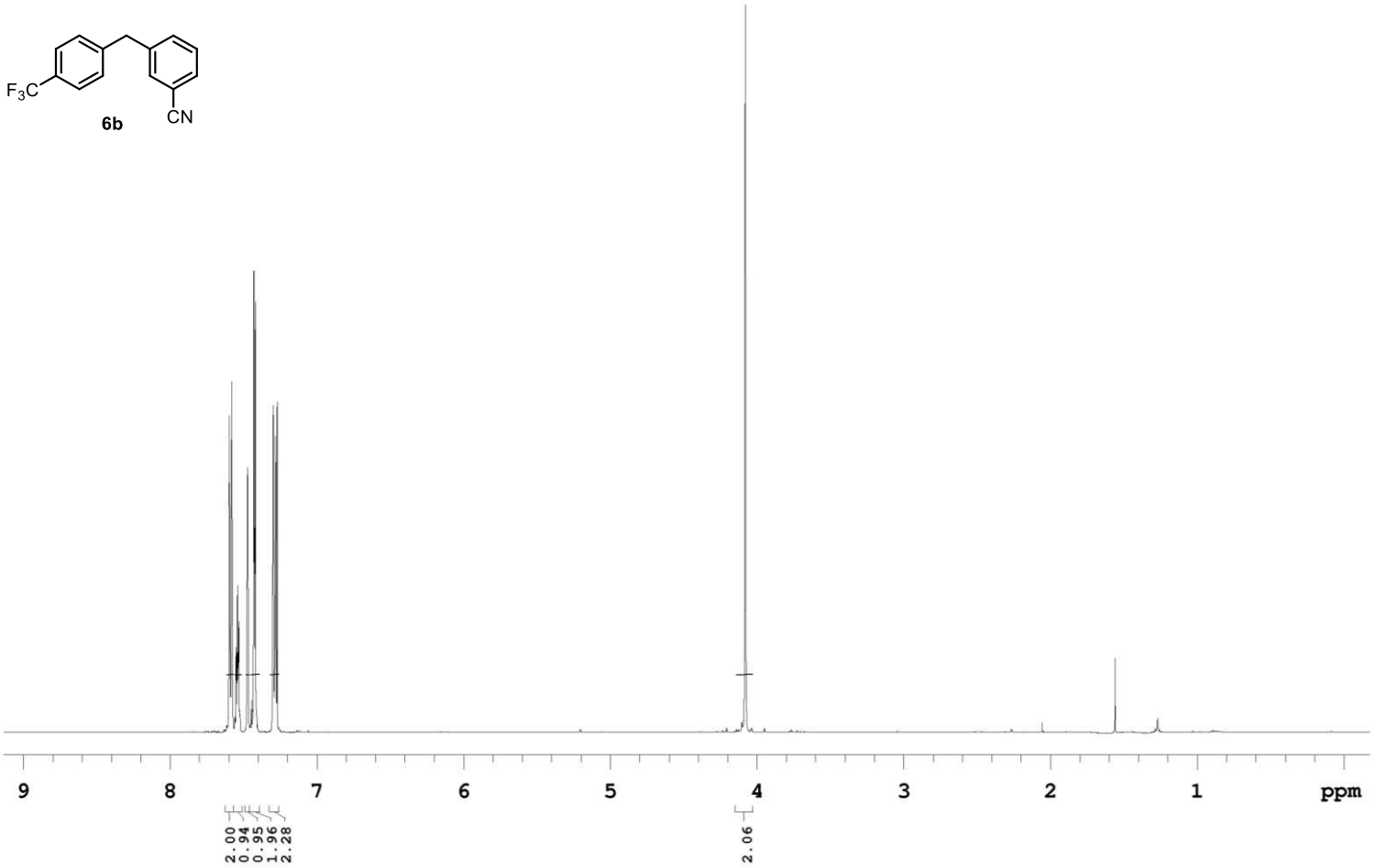
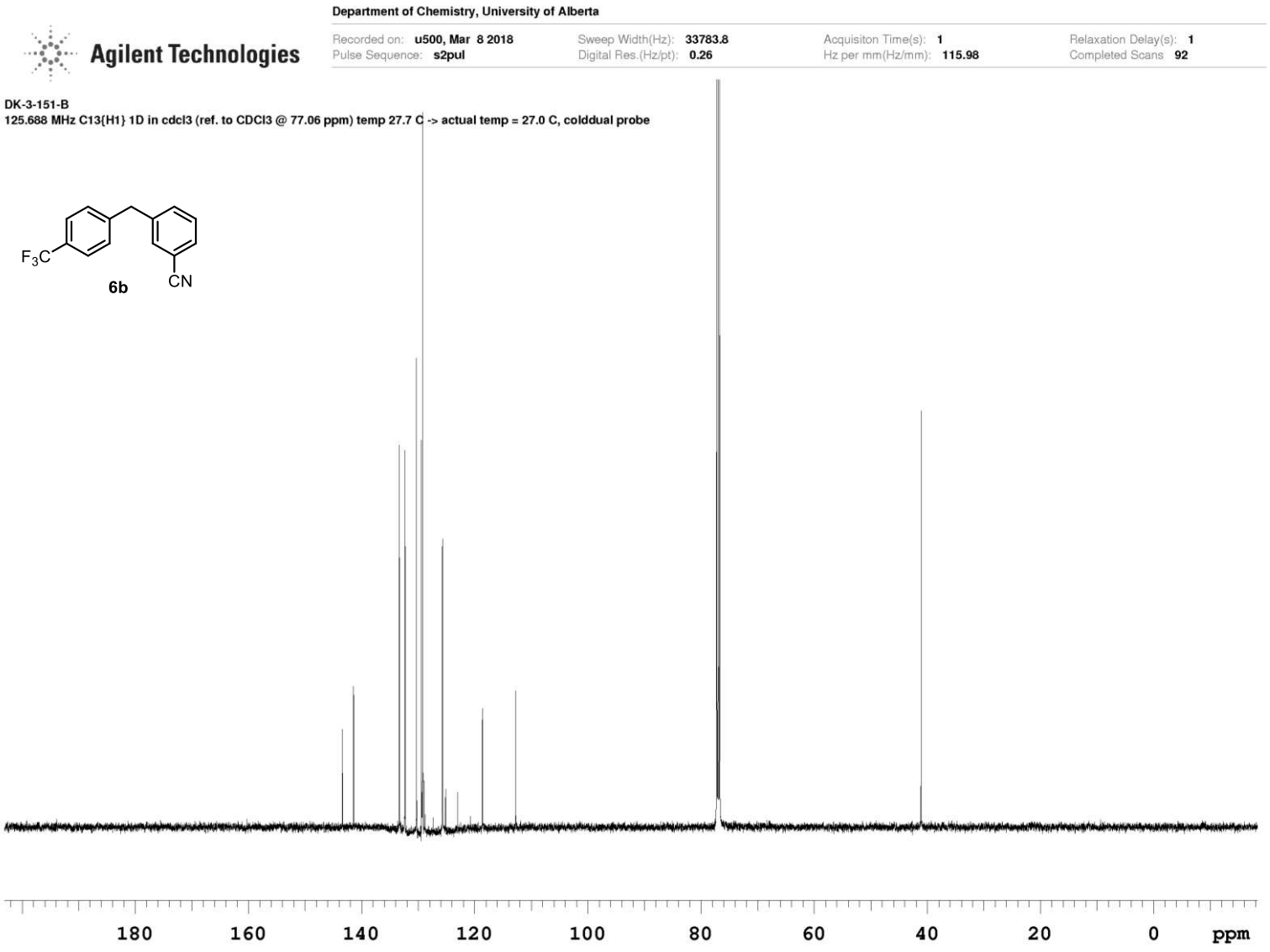
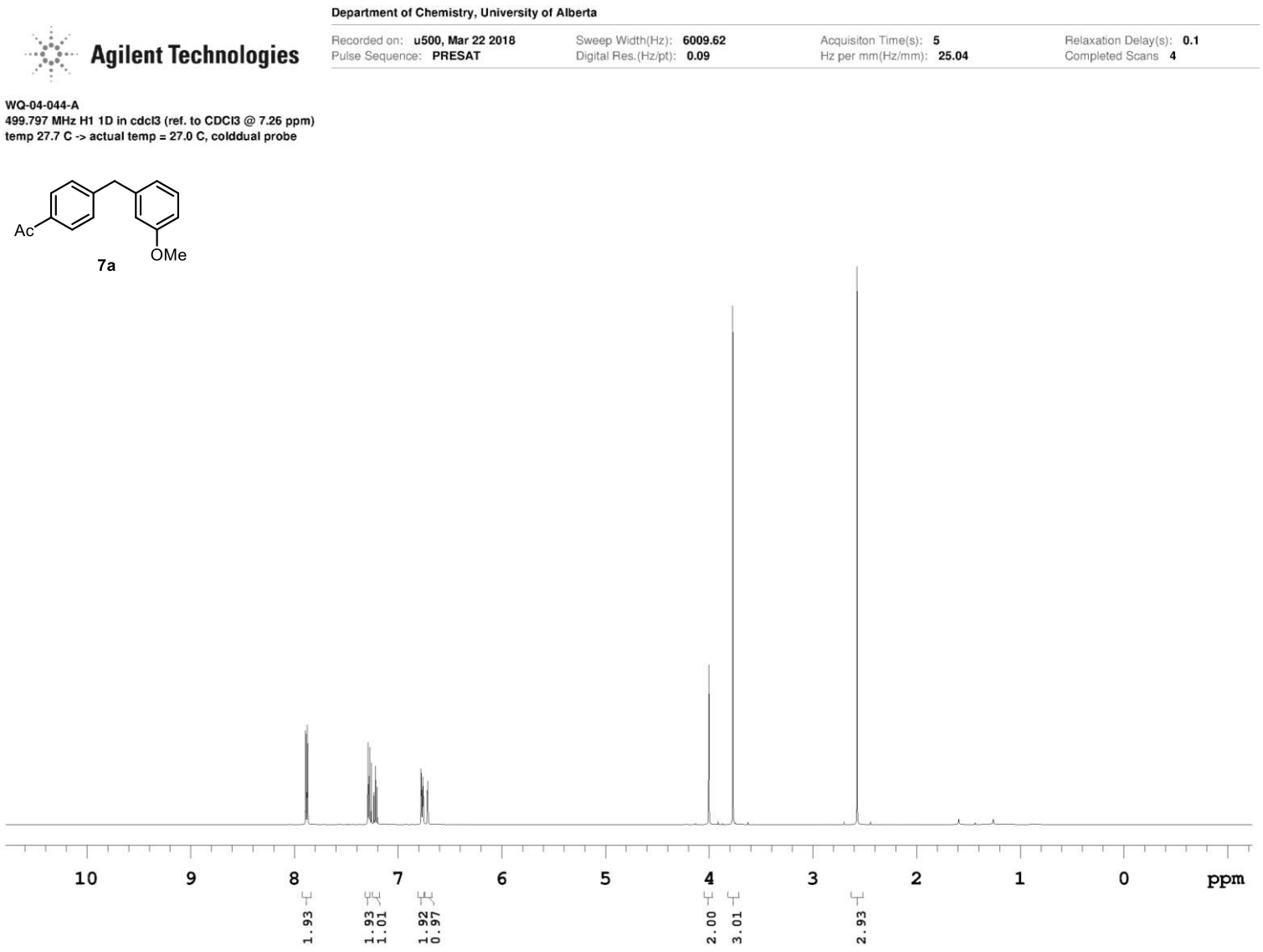
Agilent Technologies

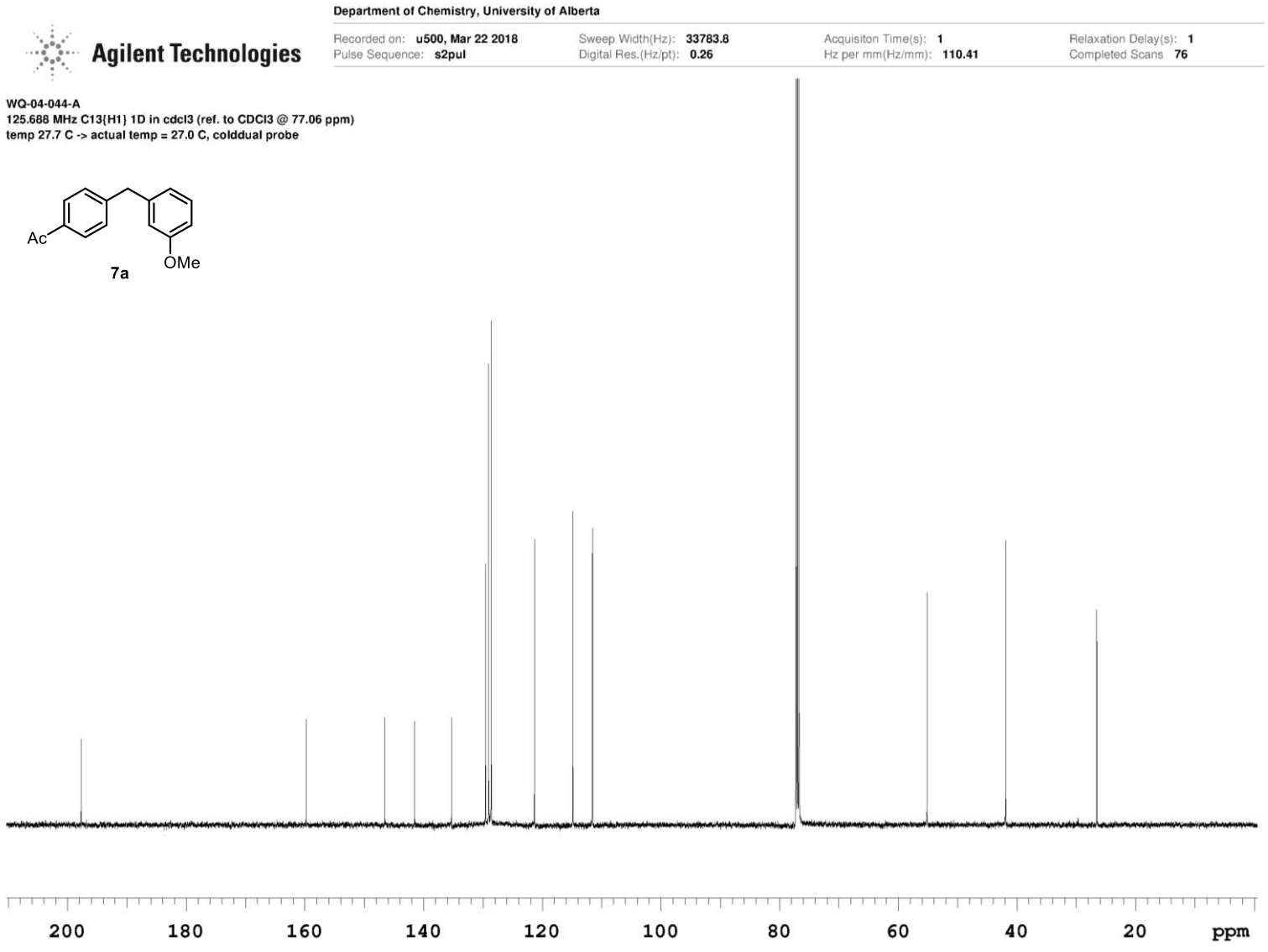
Recorded on: u500, Mar 8 2018	Sweep Width(Hz): 6009.62	Acquisiton Time(s): 5	Relaxation Delay(s): 0.1
Pulse Sequence: PRESAT	Digital Res.(Hz/pt): 0.09	Hz per mm(Hz/mm): 19.39	Completed Scans 8

DK-3-151-B
499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe




Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 8 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 115.98
Relaxation Delay(s): 1
Completed Scans 92
DK-3-151-B
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
F3C
CN
6b
180
160
140
120
100
80
60
40
20
0
ppm


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 22 2018
Pulse Sequence: PRESAT
Sweep Width(Hz): 6009.62
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 25.04
Relaxation Delay(s): 0.1
Completed Scans 4
WQ-04-044-A
499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
Ac
OMe
7a
10
9
8
7
6
5
4
3
2
1
0
ppm
1.93
1.93
1.01
1.92
0.97
2.00
3.01
2.93

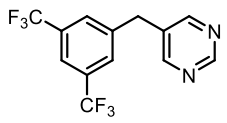

Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 22 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 110.41
Relaxation Delay(s): 1
Completed Scans 76
WQ-04-044-A
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, colddual probe
Ac
OMe
7a
200
180
160
140
120
100
80
60
40
20
ppm

Agilent Technologies

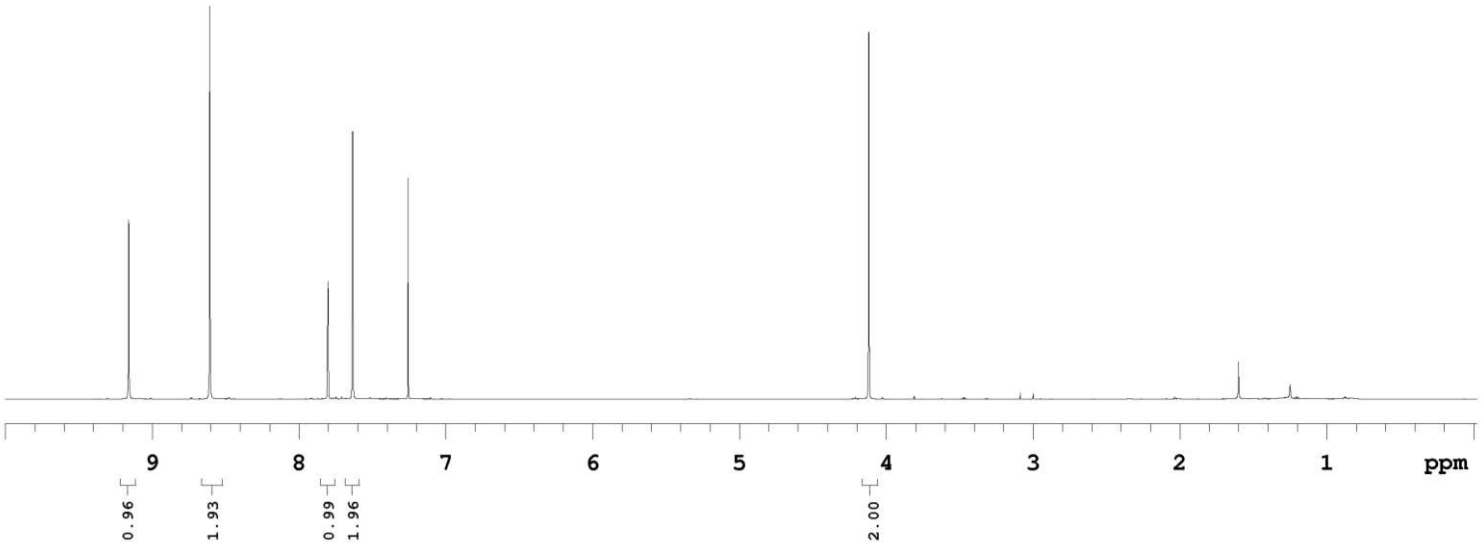
Department of Chemistry, University of Alberta

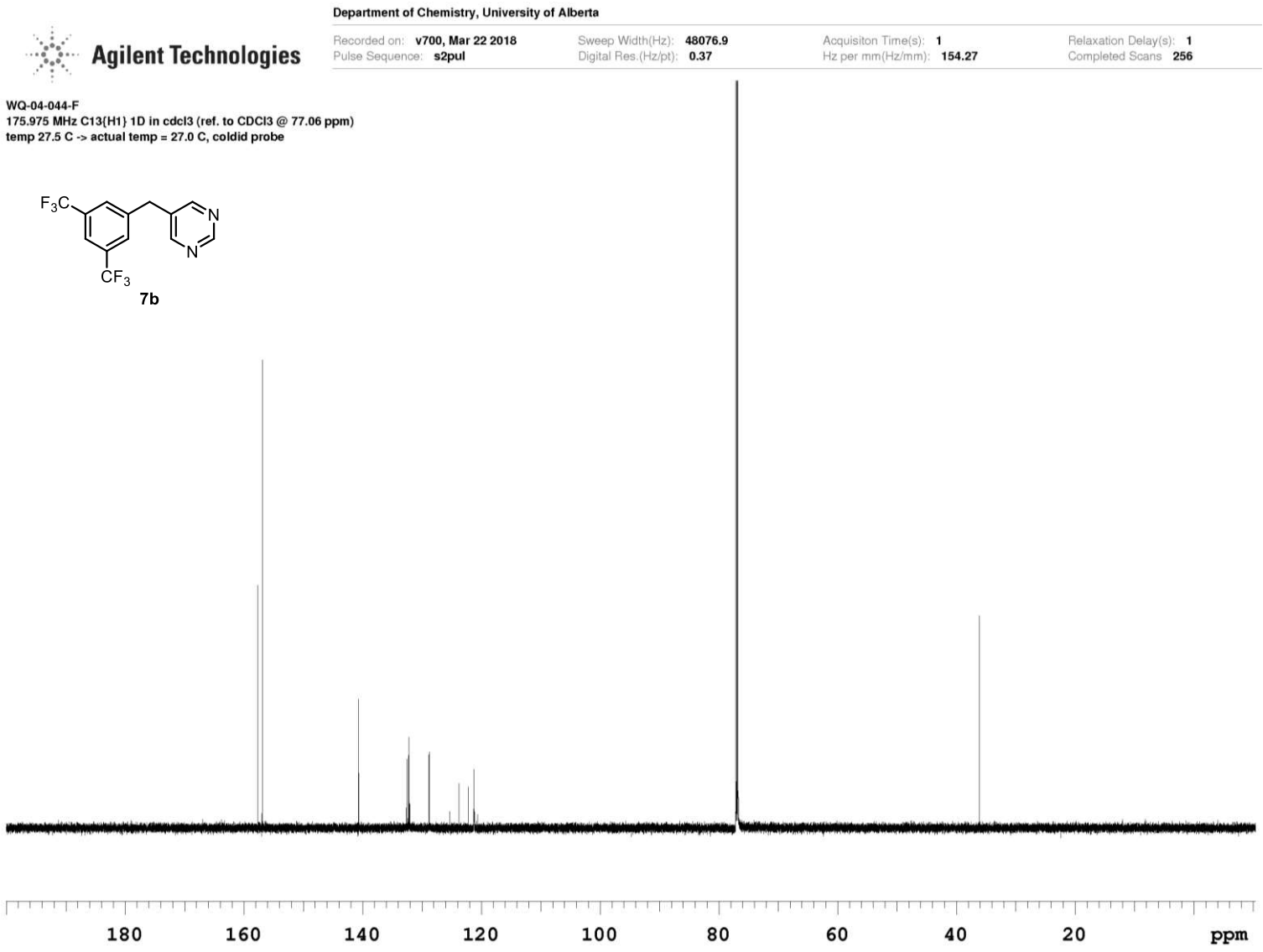
Recorded on:	v700, Mar 22 2018	Sweep Width(Hz):	8389.26	Acquisiton Time(s):	5	Relaxation Delay(s):	0.1
Pulse Sequence:	PRESAT	Digital Res.(Hz/pt):	0.13	Hz per mm(Hz/mm):	29.23	Completed Scans	8

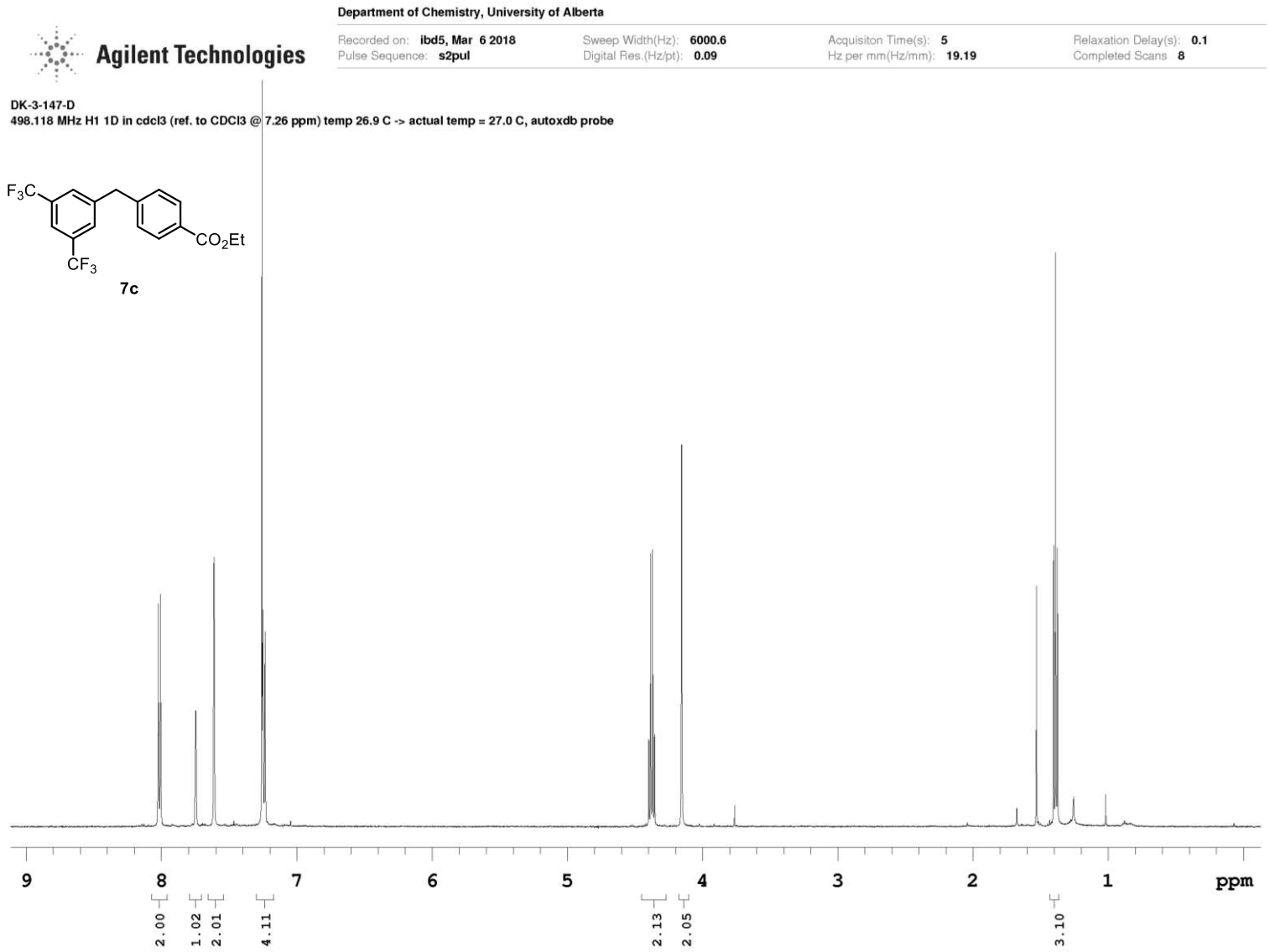
Wenyu, WQ-04-044-F
699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)
temp 27.5 C -> actual temp = 27.0 C, coldid probe

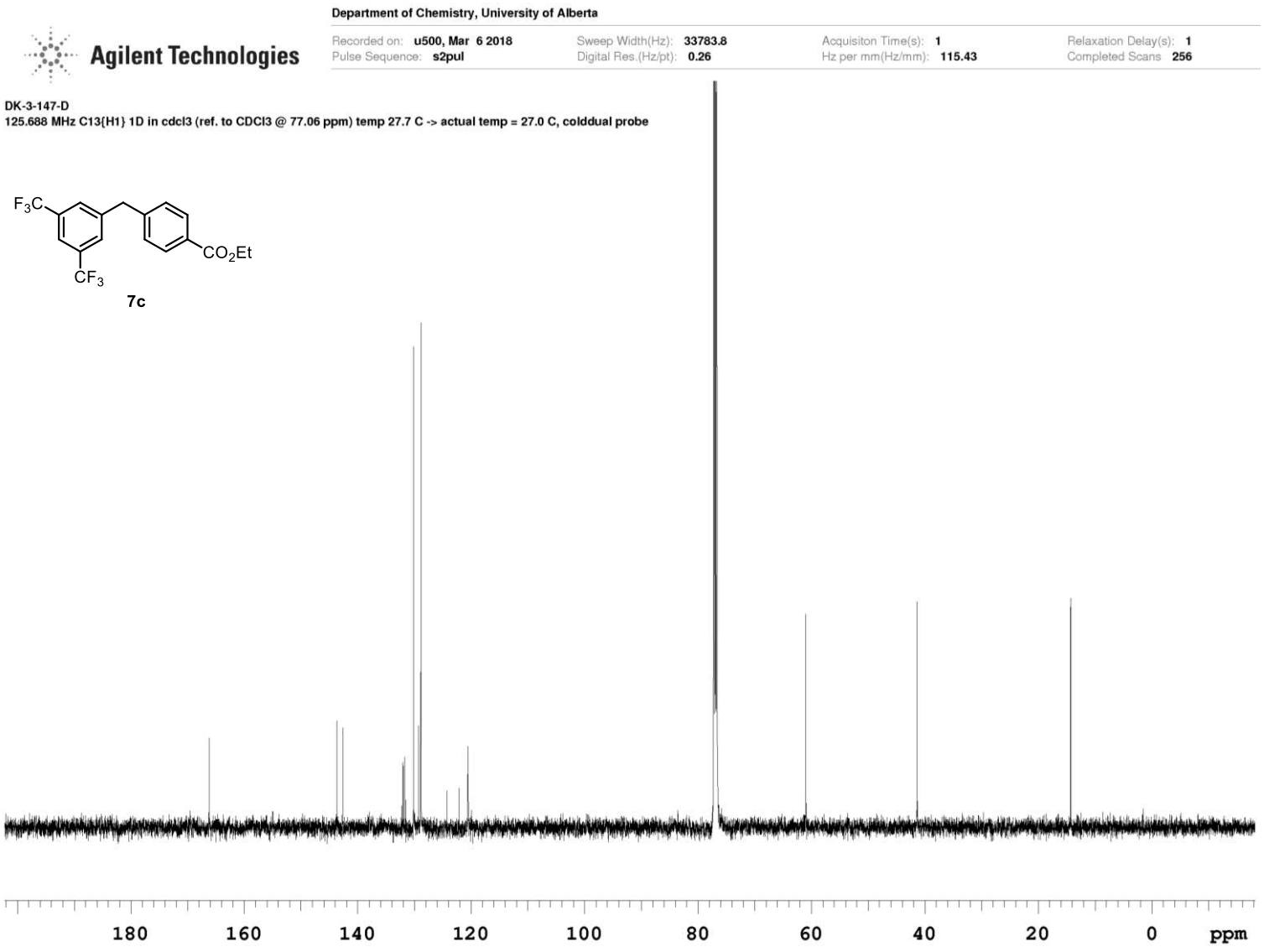


7b




Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: v700, Mar 22 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 48076.9
Digital Res.(Hz/pt): 0.37
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 154.27
Relaxation Delay(s): 1
Completed Scans 256
WQ-04-044-F
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.5 C -> actual temp = 27.0 C, coldid probe
F3C
CF3
N
N
7b
180
160
140
120
100
80
60
40
20
ppm


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: ibd5, Mar 6 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 6000.6
Digital Res.(Hz/pt): 0.09
Acquisiton Time(s): 5
Hz per mm(Hz/mm): 19.19
Relaxation Delay(s): 0.1
Completed Scans 8
DK-3-147-D
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe
F3C
CF3
CO2Et
7c
9
8
7
6
5
4
3
2
1
ppm
2.00
1.02
2.01
4.11
2.13
2.05
3.10


Department of Chemistry, University of Alberta
Agilent Technologies
Recorded on: u500, Mar 6 2018
Pulse Sequence: s2pul
Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26
Acquisiton Time(s): 1
Hz per mm(Hz/mm): 115.43
Relaxation Delay(s): 1
Completed Scans 256
DK-3-147-D
125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, colddual probe
F3C
CF3
CO2Et
7c
180
160
140
120
100
80
60
40
20
0
ppm