Supporting information

Metal-free Markovnikov-type alkyne hydration under mild conditions

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1: General information

Solvents and reagents were purchased from Sigma-Aldrich chemical company and Fisher scientific, and were used without further purification unless otherwise specified. Distilled water was used directly without further purification. ^{1}H NMR and ^{13}C NMR spectra were recorded on Varian or Bruker 400 MHz, or 500 MHz spectrometers and ^{19}F NMR spectra were recorded on a Bruker 400 MHz spectrometer. All signals are reported in ppm with the internal reference of 7.26 ppm or 77.0 ppm for chloroform as the reference. Data are reported as follows: multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet), coupling constant (*J*/Hz) and integration. All NMR spectra were recorded at room temperature (23°C) unless otherwise indicated. All reactions are stirred magnetically unless otherwise specified. All flash preparative chromatography separations were performed by using gradient elution (hexanes and ethyl acetate) of the Still protocol. All the calculations were carried out at the B3LYP/6-31G+(d) level, using the Gaussian 09 rev. D.01 suite of programs.[1] Harmonic frequencies were calculated at the same level to characterize the stationary points and to determine the zero-point energies (ZPE).

[1] Gaussian 09, Revision D.01,
M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, T. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2013.

2: General procedure

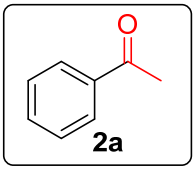
Procedure A: To a 10 mL U-shape tube were added into the alkyne (0.2 mmol), H_2O (8 µL, 0.4 mmol, 2 equiv), CF_3SO_3H (4 µL, 0.04 mmol, 0.2 equiv) and 1 mL CF_3CH_2OH. Then the tube was sealed and the mixture was stirred for 45 hours at 25 °C. After that, the volatile was removed by using rota-vapor and the residue was subjected to flash column chromatography to produce the desired hydration product.

Procedure B: To a 10 mL U-shape tube were added into the alkyne (0.2 mmol), H_2O (8 µL, 0.4 mmol, 2 equiv), CF_3SO_3H (4 µL, 0.04 mmol, 0.2 equiv) and 1 mL CF_3CH_2OH. Then the tube was sealed and posed into a pre-heated 70 °C oil bath and the mixture was stirred for 45 hours at room temperature. After that, the volatile was removed by using rota-vapor and the residue was subjected to flash column chromatography to produce the desired hydration product.

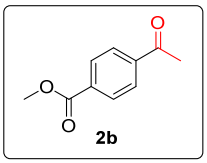
Gram scale reaction: To a 25 mL round bottom flask were added phenylacetylene (1.5 mL, 0.014 mol), H_2O (0.5 mL, 0.028 mol, 2 equiv), CF_3SO_3H (0.25 mL, 0.0028 mol, 0.2 equiv) and 5 mL CF_3CH_2OH. Then the tube was sealed and the mixture was stirred for 45 h at room temperature. After that, the mixture was transferred into a 250 mL separatory funnel. 50 mL ethyl acetate were added into the funnel and the mixture was washed by using 100 mL 1M $NaHCO_3$ solution and 100 mL brine in order. The organic layer was dried by using anhydrous Na_2SO_4 and after filtration, the organic solvent was removed by using rota-vapor and the residue was subjected to flash column chromatography (3% ethyl acetate in hexane) to produce the desired hydration product acetophenone 1.5 g (91% yield).

3: Experimental details and characterization data of the products

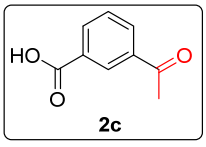
All products are commercially available and the CAS number of the products were given.



Acetophenone (CAS: 98-86-2). By employing **procedure A**, 0.2 mmol (22 µL) phenylacetylene was converted into acetophenone after 45 hours. The product was purified by using 3% EtOAc in hexane to get 24.0 mg colorless oil (100% yield). ^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.96 (d, J = 9.33 Hz, 2H), 7.57 (tt, J_1 = 7.39 Hz, J_2 = 1.25 Hz, 1H), 7.46 (t, J = 7.74 Hz, 2H), 2.61 (s, 3H); ^{13}CNMR ($CDCl_3$, 125 MHZ): δ 198.1, 137.1, 133.1, 128.5, 128.3, 26.6.

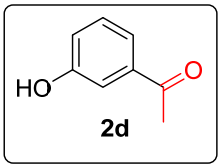


Methyl 4-acetylbenzoate (CAS: 3609-53-8). By employing **procedure B**, 0.2 mmol (32 mg) methyl 4-ethynylbenzoate was converted into methyl 4-acetylbenzoate after 45 h. The product was purified by using 15% EtOAc in hexane to get 33.5 mg (94%) colorless oil.^{1}HNMR ($CDCl_3$, 400 MHz): δ 8.11 (d, J = 8.36 Hz, 2H), 7.99 (d, J = 8.36 Hz, 2H), 3.94 (s, 3H), 2.63 (s, 3H); ^{13}CNMR ($CDCl_3$, 125 MHz): δ 197.5, 166.2, 140.2, 133.9, 129.8, 128.2, 52.4, 26.8.

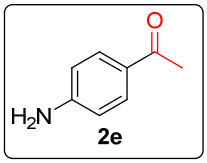


3-acetylbenzoic acid (CAS: 586-42-5). By employing **procedure B**, 0.2 mmol (30 mg) 3-ethynylbenzoic acid was converted into 3-acetylbenzoic acid after 45 h. The product was purified by using pure EtOAc to get 30.5 mg (93%) white solid. ^{1}HNMR

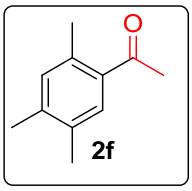
($CDCl_3$, 400 MHz): δ 8.70 (s, 1H), 8.35 (d, J = 7.49 Hz, 1H), 8.25 (d, J = 7.96 Hz, 1H), 7.64 (t, J = 7.7 Hz, 1H), 2.70 (s, 3H); ^{13}CNMR ($CDCl_3$, 125 MHz): δ 197.1, 171.1, 137.4, 134.5, 133.2, 130.2, 129.8, 129.9, 26.7.



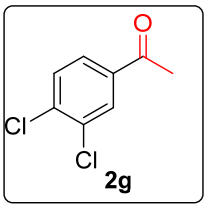
1-(3-hydroxyphenyl) ethan-1-one (CAS: 121-71-1). By employing **procedure A**, 0.2 mmol (24 mg) 3-hydroxyphenylacetylene was converted into 1-(3-hydroxyphenyl) ethan-1-one after 24 h. The product was purified by using 20% EtOAc in hexane to get 23.7 mg white solid (87 yield). ^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.57 (t, J = 2.27 Hz, 1H), 7.53 (d, J = 7.90 Hz, 1H), 7.36 (t, J = 8.01 Hz, 1H), 7.14 (dd, J_1 = 8.05 Hz, J_2 = 2.56 Hz 1H), 6.93 (s, 1H), 2.63 (s, 3H); ^{13}CNMR ($CDCl_3$, 125 MHz): δ 199.4, 156.4, 138.3, 129.9, 121.0, 120.9, 114.7, 26.7.



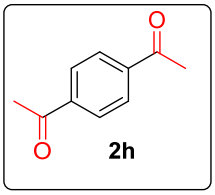
1-(4-aminophenyl) ethan-1-one (CAS: 99-92-3). To a 10 mL U-shape tube were added 4-ethynylaniline (0.2 mmol, 24 mg), H_2O (8 µL, 0.4 mmol, 2 equiv), CF_3SO_3H (24 µL, 0.24 mmol, 1.2 equiv) and 1 mL CF_3CH_2OH. Then the tube was sealed and posed into a pre-heated 70 °C oil bath and the mixture was stirred for 45 h at room temperature. After that, the volatile was removed by using rota-vapor and the residue was subjected to flash column chromatography (30% ethyl acetate in hexane) to produce the desired hydration product as a yellow solid (23.9 mg, 89% yield). ^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.82 (d, J = 8.85 Hz, 2H), 6.66 (d, J = 8.81 Hz, 2H), 4.19 (br, 2H), 2.52 (s, 3H); ^{13}CNMR ($CDCl_3$, 100 MHz): δ 196.5, 151.2, 130.8, 127.8, 113.7, 26.1.



1-(2,4,5-trimethylphenyl) ethan-1-one (CAS: 2040-07-5). By employing **procedure A**, 0.2 mmol (30 mg) 2,4,5-trimethylphenylacetylene was converted into 1-(2,4,5-trimethylphenyl) ethan-1-one after 45 h. The product was purified by using 16% EtOAc in hexane to get 26.5 mg yellow oil (83% yield). ^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.49 (s, 1H), 7.01 (s, 1H), 2.56 (s, 3H), 2.48 (s, 3H), 2.27 (s, 3H), 2.26 (s, 3H); ^{13}CNMR ($CDCl_3$, 100 MHz): δ 201.1, 140.8, 136.1, 135.0, 133.6, 133.5, 131.1, 29.3, 21.2, 19.6, 19.2.

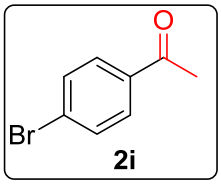


1-(3,4-dichlorophenyl) ethan-1-one (CAS: 2642-63-9) By employing **procedure A**, 0.2 mmol (35 mg) 3,4-dichlorophenylacetylene was converted into 1-(3,4-dichlorophenyl)ethan-1-one after 45 h. The product was purified by using 16% EtOAc in hexane to get 35.0 mg white solid (93% yield).^{1}HNMR ($CDCl_3$, 400 MHz): δ 8.01 (d, J = 1.92 Hz, 1H), 7.77 (dd, J_1 = 8.49 Hz, J_2 = 1.92 Hz, 1H), 7.54 (d, J = 8.49 Hz, 1H);^{13}CNMR ($CDCl_3$, 100 MHz): δ 195.6, 137.7, 136.6, 133.2, 130.7, 130.3, 127.3, 26.5.

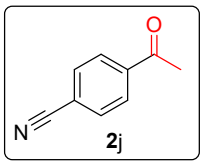


1,1'-(1,4-phenylene) bis(ethan-1-one) (CAS: 1009-61-6) To a 10 mL U-shape tube were added 1,4-diethynylbenzene (0.2 mmol, 26 mg), H_2O (16 µL, 0.8 mmol, 4 equiv), CF_3SO_3H (8 µL, 0.08 mmol, 0.4 equiv) and 1 mL CF_3CH_2OH. Then the tube was sealed and the mixture was stirred for 45 h at room temperature. After that, the

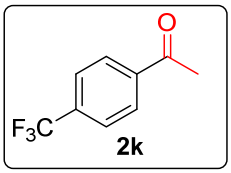
volatile was removed by using rota-vapor and the residue was subjected to flash column chromatography (16% ethyl acetate in hexane) to produce the desired hydration product 1,1'-(1,4-phenylene)bis(ethan-1-one) as a colorless oil (31.1 mg, 96% yield).^{1}HNMR ($CDCl_3$, 400 MHz): δ 8.04 (s, 4H), 2.66 (s, 6H);^{13}CNMR ($CDCl_3$, 100 MHz): δ 197.4, 140.1, 128.4, 26.8.



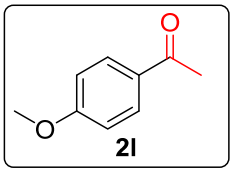
1-(4-bromophenyl) ethan-1-one (CAS: 99-90-1) By employing **procedure A**, 0.2 mmol (37 mg) 4-bromophenylacetylene was converted into 1-(4-bromophenyl)ethan-1-one after 45 h. The product was purified by using 16% EtOAc in hexane to get 37.8 mg yellow solid (95% yield).^{1}HNMR ($CDCl_3$, 400 MHz): δ 8.04 (d, *J* = 8.42 Hz, 2H), 7.77 (d, *J* = 8.02 Hz, 2H), 2.64 (s, 3H);^{13}CNMR ($CDCl_3$, 125 MHz): δ 196.9, 135.8, 131.9, 129.8, 128.3, 26.5.



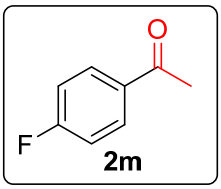
4-acetylbenzonitrile (CAS: 144-80-7) To a 10 mL U-shape tube were added 4-ethynylbenzonitrile (0.2 mmol, 26 mg), H_2O (8 µL, 0.4 mmol, 2 equiv), CF_3SO_3H (24 µL, 0.24 mmol, 1.2 equiv) and 1 mL CF_3CH_2OH. Then the tube was sealed and posed into a pre-heated 70 °C oil bath and the mixture was stirred for 45 h. After that, the volatile was removed by using rota-vapor and the residue was subjected to flash column chromatography (16% ethyl acetate in hexane) to produce the desired hydration product 4-acetylbenzonitrile as a colorless oil (23.3 mg, 80% yield).^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.81 (d, *J* = 8.49 Hz, 2H), 7.60 (d, *J* = 8.01 Hz, 2H), 2.58 (s, 3H); ^{13}CNMR ($CDCl_3$, 125 MHz): δ 196.5, 139.9, 132.5, 128.7, 117.9, 116.4, 26.7.



1-(4-(trifluoromethyl)phenyl)ethan-1-one (CAS: 709-63-7) By employing **procedure B**, 0.2 mmol (35 µL) 4-trifluoromethylphenylacetylene was converted into 1-(4-(trifluoromethyl)phenyl)ethan-1-one after 45 h. The product was purified by using 9% EtOAc in hexane to get 34.2 mg (91%) colorless oil.^{1}HNMR ($CDCl_3$, 400 MHz): δ 8.06 (d, *J* = 8.25 Hz, 2H), 7.74 (d, *J* = 8.08 Hz, 2H), 2.65 (s, 3H); ^{13}CNMR ($CDCl_3$, 100 MHz): δ 196.9, 139.6, 133.3 (q, *J* = 32.6 Hz), 128.6, 125.6 (q, *J* = 3.84 Hz), 123.6 (q, *J* = 270.5), 26.72. ^{19}FNMR ($CDCl_3$, 500 MHz): δ -63.14.

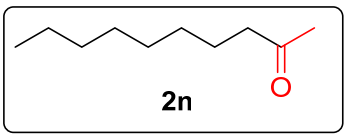


1-(4-methoxyphenyl) ethan-1-one (CAS: 100-06-1) By employing **procedure A**, 0.2 mmol (27 µL) 4-methoxyphenylacetylene was converted into 1-(4-methoxyphenyl)ethan-1-one after 24 h. The product was purified by using 16% EtOAc in hexane to get 29.1 mg yellow oil (97% yield). ^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.93 (d, *J* = 8.76 Hz, 2H), 6.92 (d, *J* = 9.04 Hz, 2H), 3.86 (s, 3H), 2.55 (s, 3H);^{13}CNMR ($CDCl_3$, 125 MHz): δ 196.7, 163.4, 130.5, 130.3, 113.6, 55.4, 26.3.

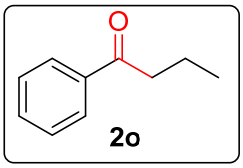


1-(4-fluorophenyl)ethan-1-one (CAS: 403-42-9) By employing **procedure B**, 0.2 mmol (25 µL) 4-fluoromethylphenylacetylene was converted into 1-(4-fluorophenyl)ethan-1-one after 45 h. The product was purified by using 9% EtOAc

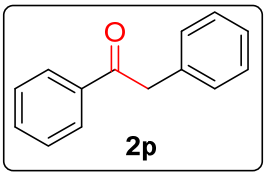
in hexane to get 26.8 mg (97%) colorless oil.^{1}HNMR ($CDCl_3$, 400 MHz): δ 8.01 (m, 2H), 7.16 (m, 2H), 2.62 (s, 3H);^{13}CNMR ($CDCl_3$, 100 MHz): δ 196.4,165.8 (*J* = 253.29 Hz), 133.6 (*J* = 2.5 Hz), 130.8 (*J* = 9.1 Hz), 115.6 (*J* = 21.79 Hz), 26.5; ^{19}FNMR ($CDCl_3$, 400 MHz): δ -105.35.



decan-2-one (CAS: 693-54-9) By employing **procedure B**, 0.2 mmol (36 µL) 4-fluoromethylphenylacetylene was converted into decan-2-one after 45 h. The product was purified by using 9% EtOAc in hexane to get 23.7 mg (76%) colorless oil.^{1}HNMR ($CDCl_3$, 400 MHz): δ 2.43 (t, *J* = 7.67 Hz, 2H), 2.15 (s, 3H), 1.64-1.52 (m, 2H), 1.37-1.21 (m, 10H), 0.89 (t, *J* = 6.87 Hz, 3H);^{13}CNMR ($CDCl_3$, 100 MHz): δ 209.3, 43.8, 31.8, 29.8, 29.3, 29.1, 29.1, 23.8, 22.6, 14.0.



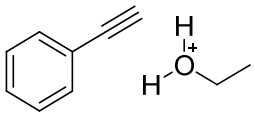
1,2-diphenylethan-1-one (CAS: 451-40-1) By employing **procedure A**, 0.2 mmol (29 µL) 1-phenyl-butyne was converted into 1,2-diphenylethan-1-one after 45 h. The product was purified by using 16% EtOAc in hexane to get 27.2 mg colorless oil (92% yield).^{1}HNMR ($CDCl_3$, 400 MHz): δ 8.07-8.02 (m, 2H), 7.61-7.56 (m, 1H), 7.52-7.46 (m, 2H), 7.39-7.27 (m, 5H), 4.32 (s, 2H); ^{13}CNMR ($CDCl_3$, 125 MHz): δ 197.6, 136.6, 134.5, 133.1, 129.4, 128.6, 128.6, 128.6, 126.9, 45.5.



1-phenylbutan-1-one (CAS: 495-40-9) By employing **procedure A**, 0.2 mmol (36 mg) diphenylacetylene was converted into 1-phenylbutan-1-one after 45 h. The product was purified by using 16% EtOAc in hexane to get 36.8 mg yellow solid (94% yield). ^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.96 (d, *J* = 7.24Hz, 2H), 7.55 (t, *J* = 7.33 Hz, 1H), 7.46 (t, *J* = 7.84 Hz, 2H), 2.95 (t, *J* = 7.27 Hz, 2H), 1.77 (sextet, *J* = 7.55 Hz, 2H), 1.01 (t, *J* = 7.10 Hz, 3H); ^{13}CNMR ($CDCl_3$, 100 MHz): δ 200.5, 137.1, 132.9, 128.6, 128.0, 40.5, 17.8, 13.9.

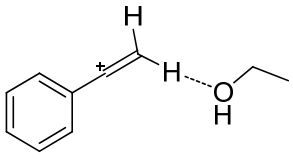
4: Calculation data

Atomic Cartesian coordinates and computed energies (atomic units) for the stationary points calculated with basis set [B3LYP/6-31G+(d)]



Sum of electronic and zero-point Energies= -463.580609
Sum of electronic and thermal Energies= -463.568067
Sum of electronic and thermal Enthalpies= -463.567123
Sum of electronic and thermal Free Energies= -463.621024

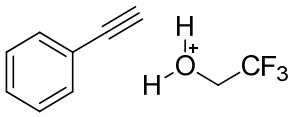
Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	6	0	-1.768228	0.749264	-0.866799
2	6	0	-0.820766	0.946135	0.158200
3	6	0	-0.693427	-0.040588	1.170929
4	6	0	-1.514203	-1.180658	1.154094
5	6	0	-2.446815	-1.361915	0.127614
6	6	0	-2.570393	-0.392934	-0.875362
7	1	0	-1.877208	1.503268	-1.640049
8	1	0	-0.029499	0.144861	2.012616
9	1	0	-1.439256	-1.906912	1.959621
10	1	0	-3.086857	-2.239149	0.121344
11	1	0	-3.302945	-0.523943	-1.666630
12	6	0	-0.005899	2.120664	0.186574
13	6	0	0.684270	3.116487	0.206454
14	1	0	1.370173	-2.290365	-0.360296
15	1	0	1.261082	4.016668	0.235334
16	8	0	1.213522	-1.364152	-0.637511
17	6	0	3.422979	-0.738079	0.386201
18	6	0	2.509371	-0.562588	-0.800118
19	1	0	2.908784	-0.946656	-1.738784
20	1	0	2.134155	0.451884	-0.934107
21	1	0	0.532074	-0.934756	-0.004595
22	1	0	2.972280	-0.371013	1.313291
23	1	0	4.327585	-0.146248	0.200322
24	1	0	3.740116	-1.779161	0.515295



Sum of electronic and zero-point Energies= -463.608679
Sum of electronic and thermal Energies= -463.595936
Sum of electronic and thermal Enthalpies= -463.594992
Sum of electronic and thermal Free Energies= -463.651606

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z

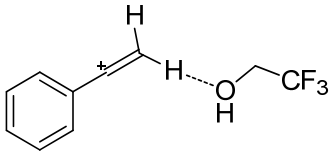
1	6	0	-2.023259	-0.806028	-0.956652
2	6	0	-1.538534	0.364378	-0.284476
3	6	0	-2.303776	0.952549	0.776265
4	6	0	-3.508527	0.382232	1.141407
5	6	0	-3.966988	-0.765818	0.469414
6	6	0	-3.230756	-1.358197	-0.573129
7	1	0	-1.432551	-1.239420	-1.757537
8	1	0	-1.923421	1.838081	1.275631
9	1	0	-4.100093	0.815127	1.941744
10	1	0	-4.915720	-1.207099	0.763610
11	1	0	-3.612009	-2.243559	-1.071790
12	6	0	-0.347663	0.924453	-0.659581
13	6	0	0.780540	1.433978	-0.992198
14	1	0	1.723494	1.096661	-0.496066
15	1	0	0.860253	2.205687	-1.760616
16	8	0	3.223181	0.465112	0.374856
17	6	0	5.070503	-1.135249	0.692979
18	6	0	4.304854	-0.247768	-0.276414
19	1	0	3.815679	-0.842736	-1.052624
20	1	0	4.970317	0.473701	-0.767465
21	1	0	3.591523	1.002636	1.095428
22	1	0	4.404141	-1.860458	1.171551
23	1	0	5.560422	-0.542193	1.475550
24	1	0	5.854157	-1.684506	0.158042



Sum of electronic and zero-point Energies= -761.324575
Sum of electronic and thermal Energies= -761.310375
Sum of electronic and thermal Enthalpies= -761.309430
Sum of electronic and thermal Free Energies= -761.369055

Center Number	Atomic Number	Forces (Hartrees/Bohr) X	Y	Z
1	6	-0.000006959	0.000004417	-0.000009320
2	6	0.000002667	-0.000007530	-0.000004591
3	6	-0.000001389	-0.000001824	0.000002877
4	6	0.000000944	0.000004999	-0.000006374
5	6	0.000004316	-0.000004268	-0.000007459
6	6	-0.000001072	-0.000005847	0.000000488
7	1	-0.000006995	-0.000004742	-0.000004060
8	1	0.000005697	-0.000000290	-0.000005734
9	1	0.000008382	0.000000365	-0.000004715
10	1	0.000002962	0.000000039	-0.000005579
11	1	-0.000004077	-0.000002563	-0.000005749
12	6	-0.000006698	-0.000002192	-0.000004369
13	6	-0.000004206	-0.000005428	-0.000007612
14	1	0.000002096	0.000004186	0.000001037
15	1	-0.000005901	-0.000005352	-0.000000228
16	8	0.000007767	0.000004627	0.000020370
17	6	0.000006712	0.000004853	0.000014954
18	9	-0.000003828	0.000008486	0.000006556

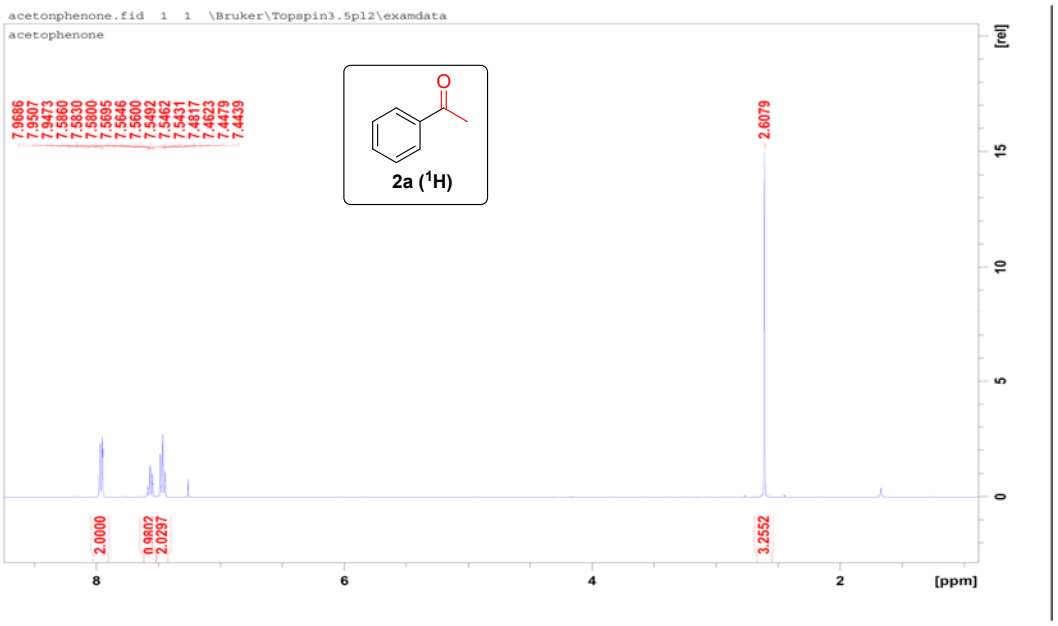
19	9	0.000001522	-0.000001328	0.000002363
20	9	0.000005428	0.000008240	0.000010125
21	6	-0.000003982	-0.000007717	0.000015393
22	1	0.000003781	0.000001420	0.000007558
23	1	-0.000005609	0.000013157	-0.000017672
24	1	-0.000001558	-0.000005708	0.000001741

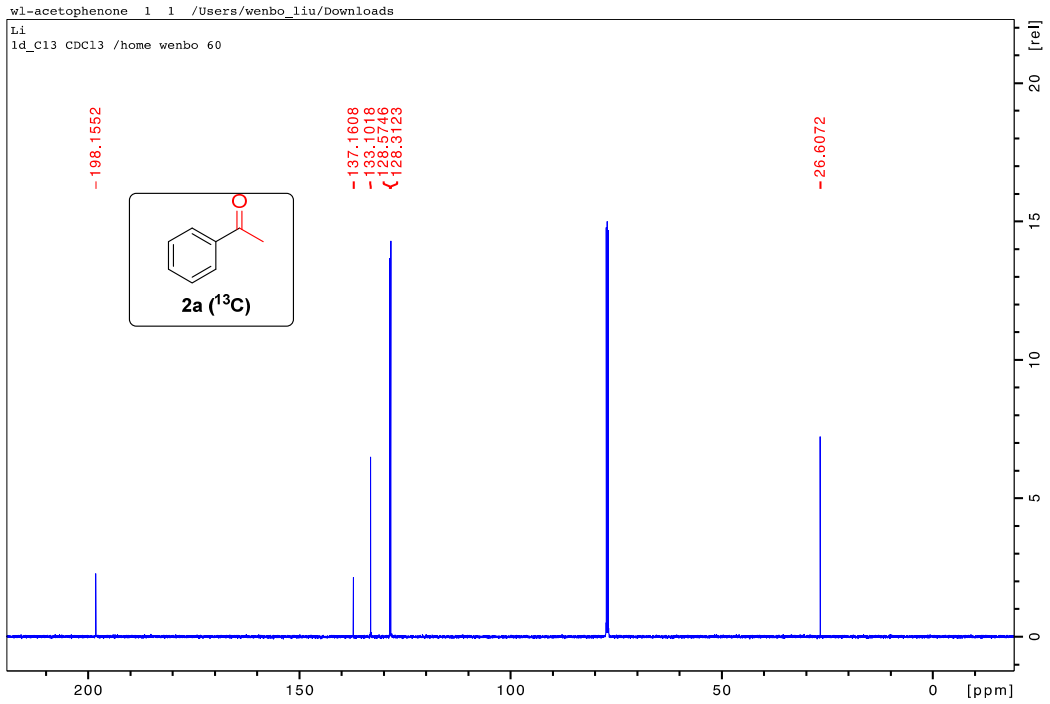


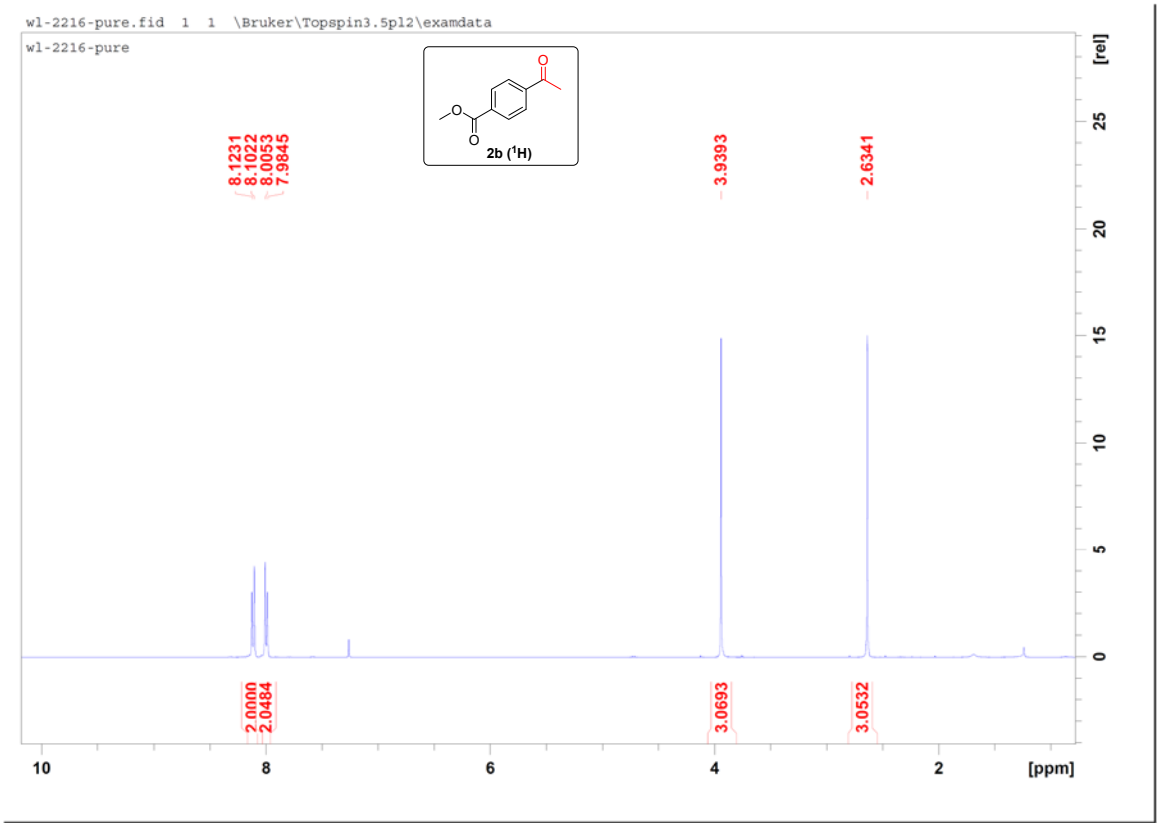
Sum of electronic and zero-point Energies= -761.370652
Sum of electronic and thermal Energies= -761.356087
Sum of electronic and thermal Enthalpies= -761.355142
Sum of electronic and thermal Free Energies= -761.416240

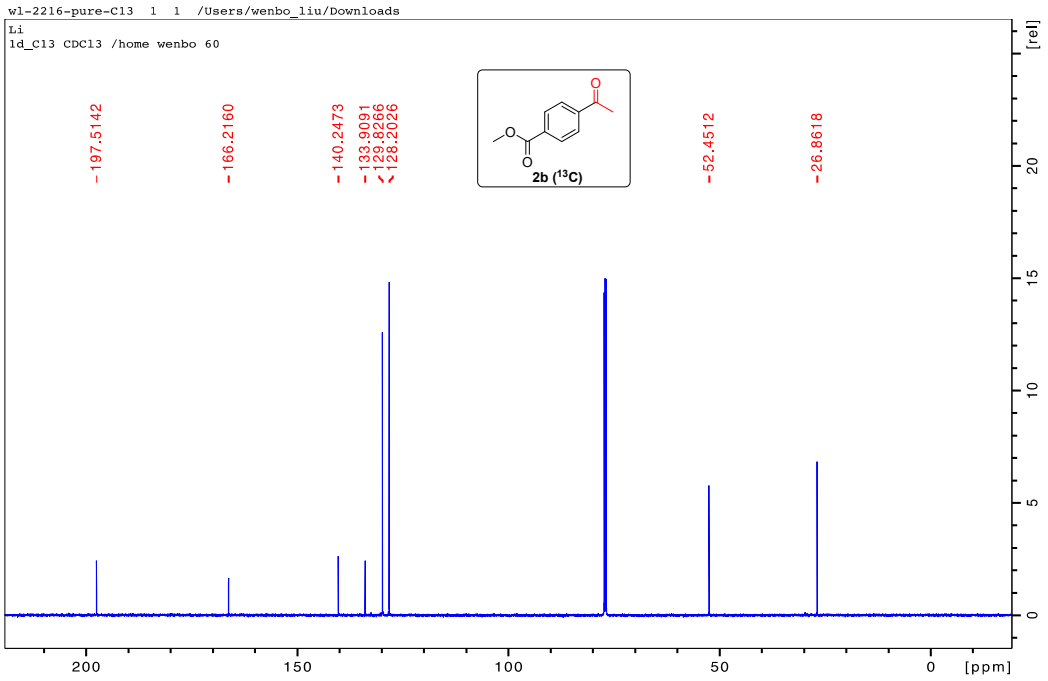
Center Number	Atomic Number	Forces (Hartrees/Bohr) X	Y	Z
1	6	-0.000000921	0.000003455	0.000003786
2	6	-0.000000219	0.000003054	-0.000007244
3	6	0.000002280	0.000000902	0.000004666
4	6	-0.000001209	0.000001830	0.000001107
5	6	-0.000002474	0.000006184	0.000002938
6	6	-0.000000930	0.000007067	0.000000406
7	1	-0.000001272	0.000005210	0.000001462
8	1	0.000002765	-0.000000016	0.000001767
9	1	0.000000188	0.000002194	0.000001282
10	1	-0.000002795	0.000006033	-0.000000004
11	1	-0.000003929	0.000007647	0.000000897
12	6	-0.000000934	-0.000000384	0.000006105
13	6	0.000012057	0.000004208	0.000001762
14	1	-0.000002735	-0.000003664	0.000001787
15	1	0.000000750	-0.000000824	0.000004917
16	8	0.000013122	-0.000007504	-0.000004595
17	6	0.000017188	-0.000016986	-0.000001477
18	9	-0.000006847	-0.000000024	-0.000001591
19	9	-0.000005704	-0.000005574	-0.000010307
20	9	-0.000010651	0.000000259	-0.000001510
21	6	-0.000002156	0.000003651	-0.000000525
22	1	-0.000004068	-0.000003899	-0.000001807
23	1	0.000000155	-0.000005850	-0.000005316
24	1	-0.000001662	-0.000006967	0.000001494

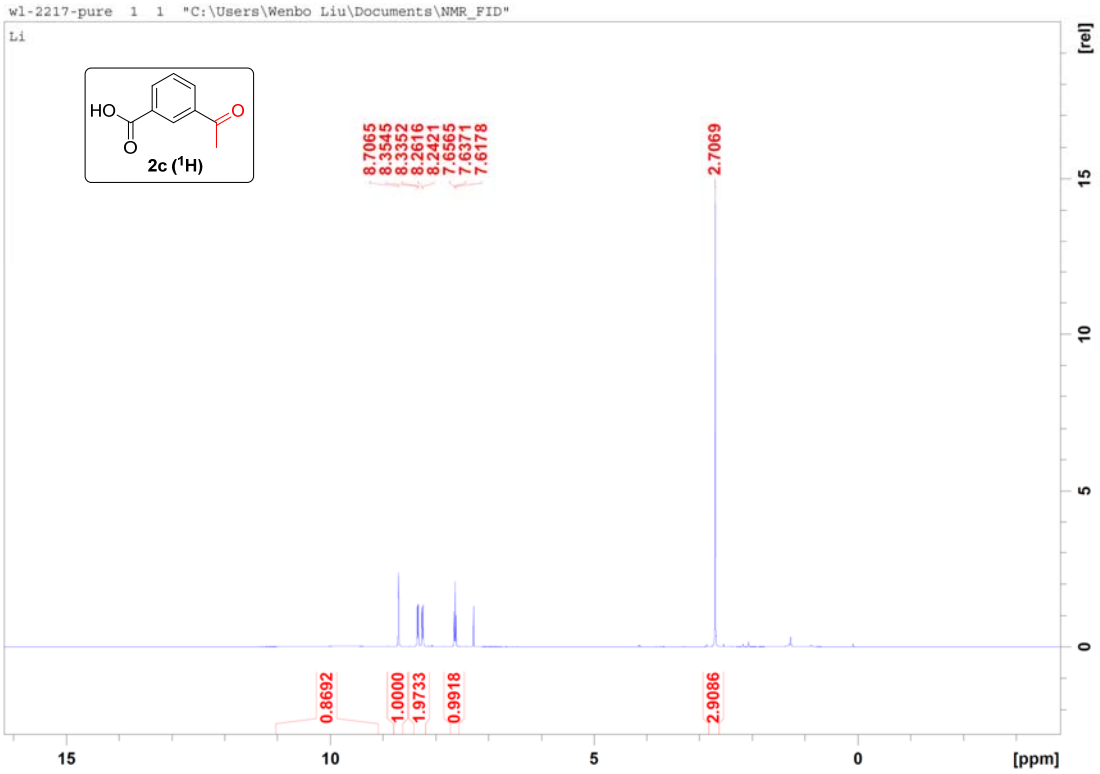
5: Copies of NMR spectra

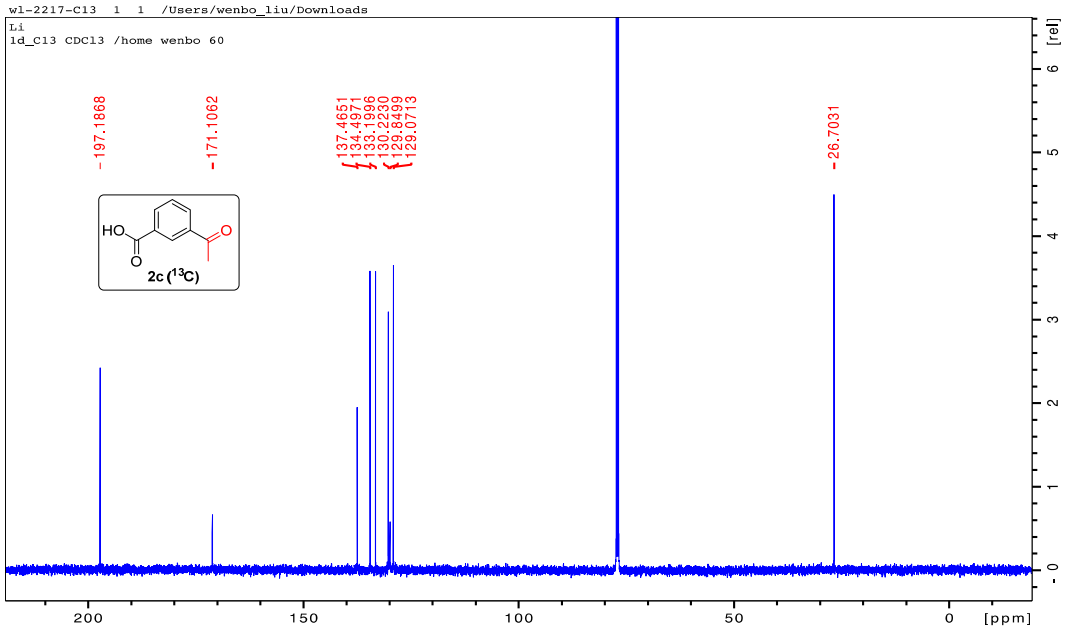


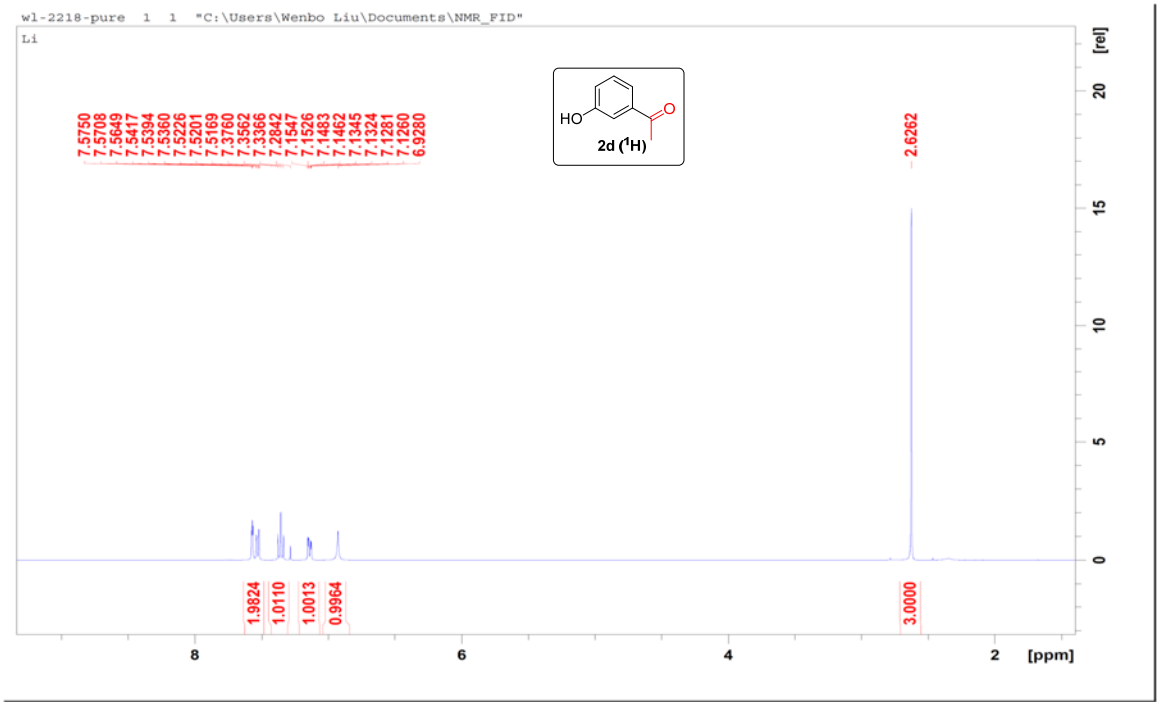


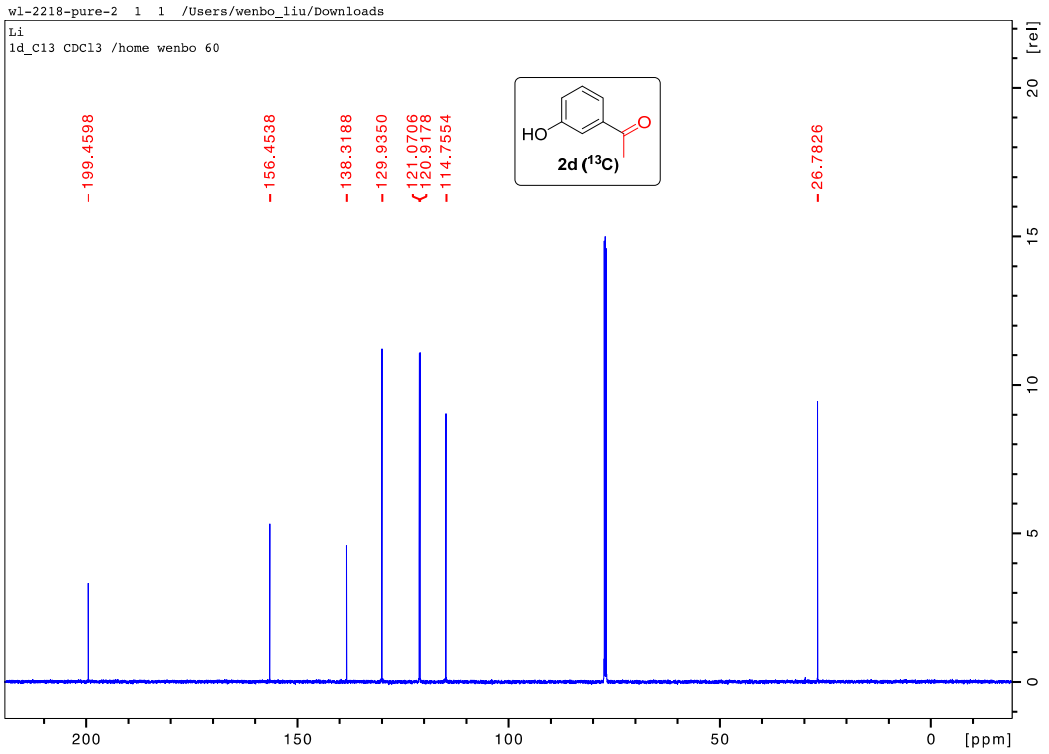

wl-2216-pure.fid 1 1 \Bruker\Topspin3.5pl2\examdata
wl-2216-pure
2b (^{1}H)
8.1231
8.1022
8.0053
7.9845
3.9393
2.6341
2.0000
2.0484
3.0693
3.0532
[rel]
[ppm]

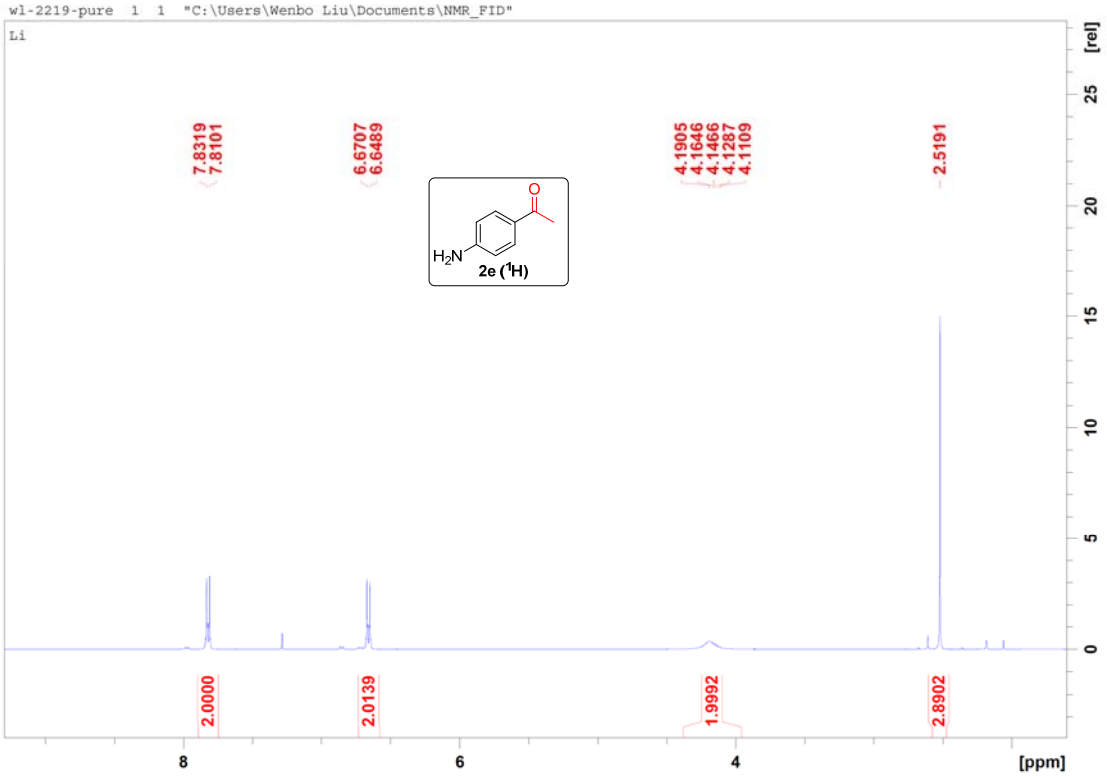

wl-2216-pure-C13 1 1 /Users/wenbo_liu/Downloads
Li
1d_C13 CDCl3 /home wenbo 60
2b (^{13}C)
197.5142
166.2160
140.2473
133.9091
129.8266
128.2026
52.4512
26.8618
[rel]
[ppm]

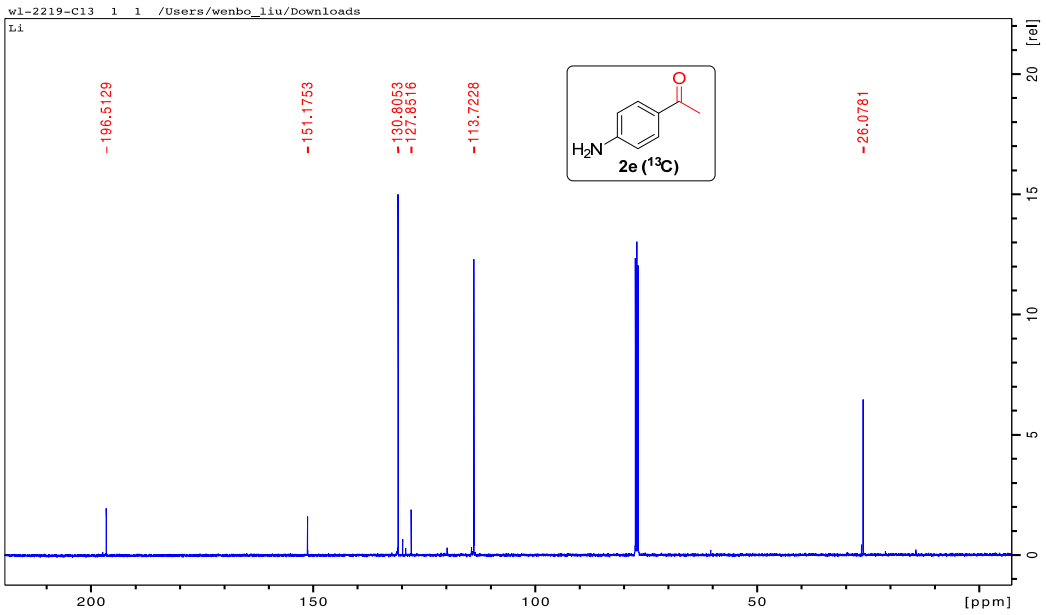

wl-2217-pure 1 1 "C:\Users\Wenbo Liu\Documents\NMR_FID"
Li
HO
O
O
2c (^{1}H)
8.7065
8.3545
8.3352
8.2616
8.2421
7.6565
7.6371
7.6178
2.7069
[rel]
0.8692
1.0000
1.9733
0.9918
2.9086
15
10
5
0
[ppm]

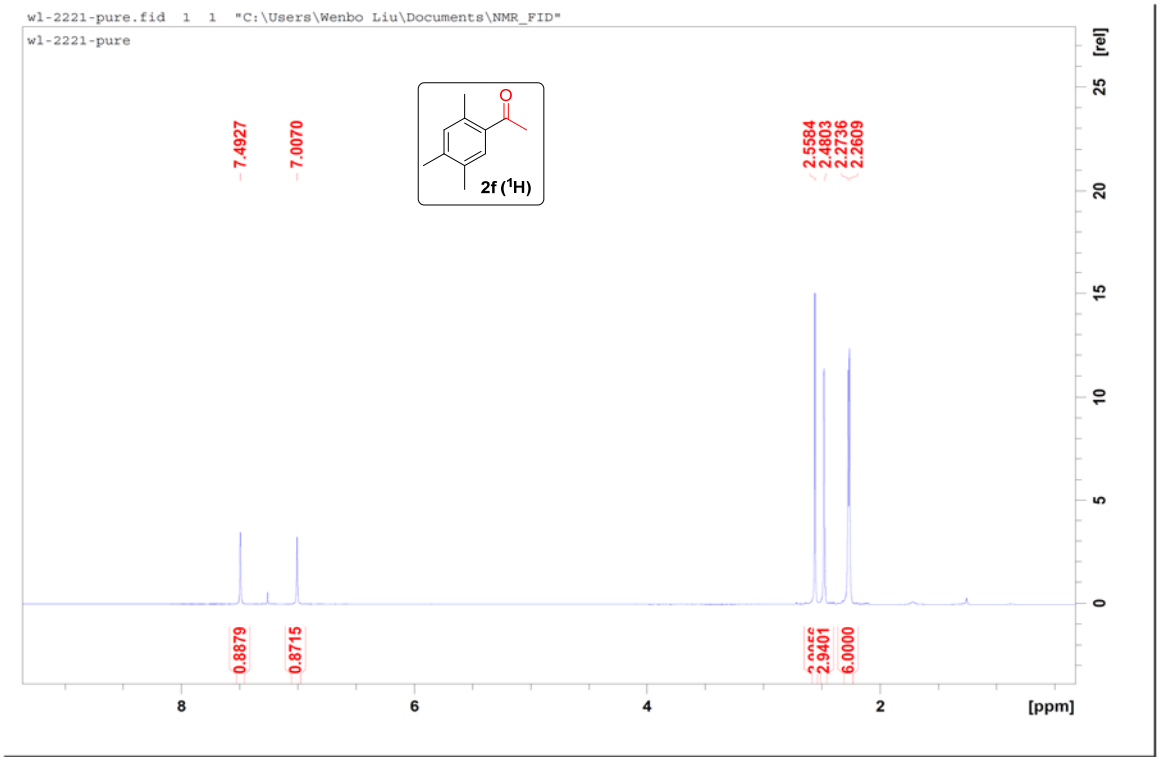

wl-2217-C13 1 1 /Users/wenbo_liu/Downloads
Li
1d_C13 CDCl3 /home wenbo 60
197.1868
171.1062
137.4651
134.4971
133.1996
130.2230
129.8499
129.0713
26.7031
HO
O
O
2c (^{13}C)
[rel]
200
150
100
50
0
[ppm]

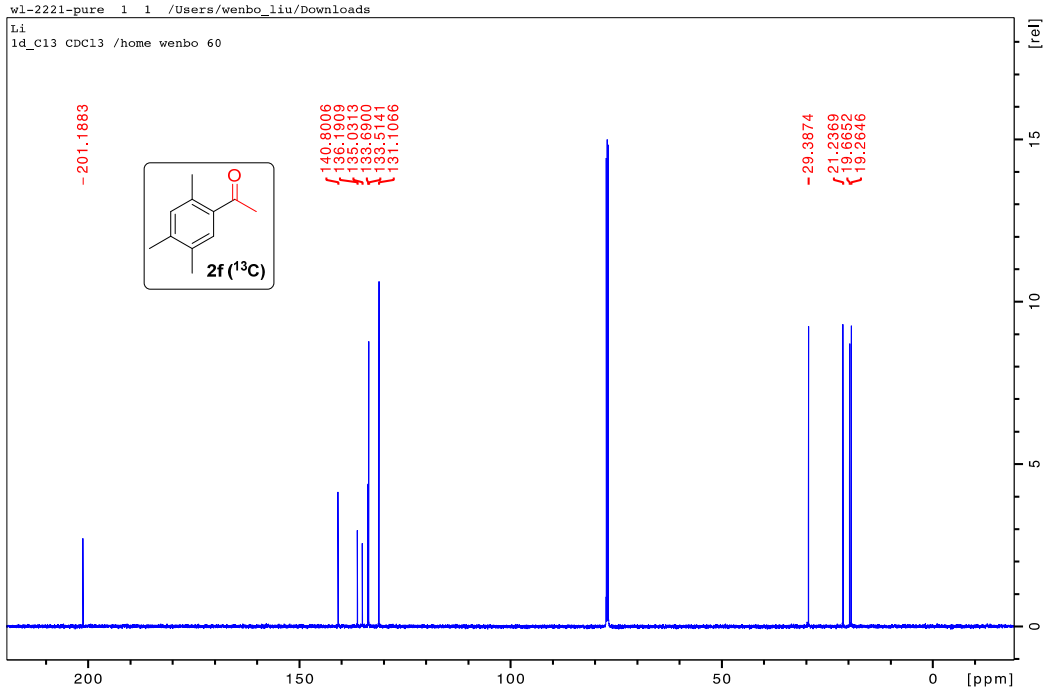

wl-2218-pure 1 1 "C:\Users\Wenbo Liu\Documents\NMR_FID"
Li
7.5750
7.5708
7.5649
7.5417
7.5394
7.5360
7.5226
7.5201
7.5169
7.3760
7.3562
7.3366
7.2842
7.1547
7.1526
7.1483
7.1462
7.1345
7.1324
7.1281
7.1260
6.9280
2.6262
HO
O
2d (1H)
1.9824
1.0110
1.0013
0.9964
3.0000
8
6
4
2
[ppm]
[rel]
20
15
10
5
0

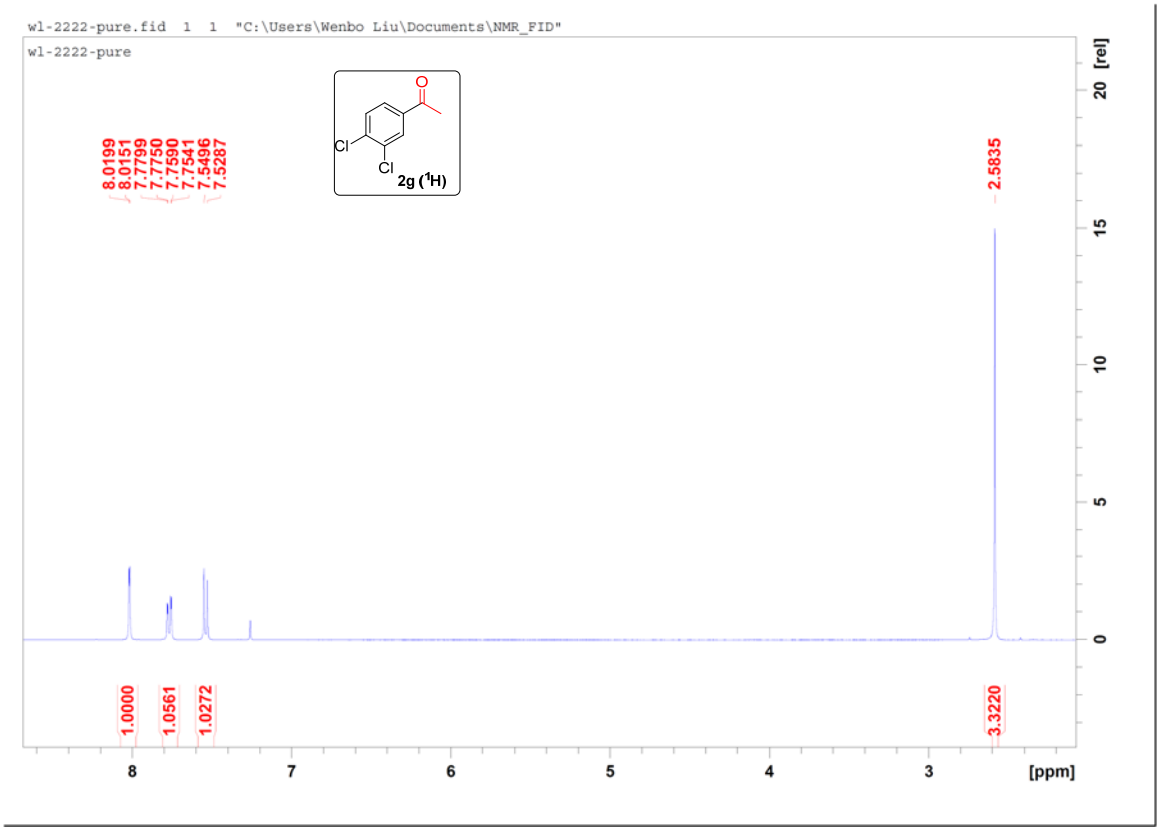

wl-2218-pure-2 1 1 /Users/wenbo_liu/Downloads
Li
1d_C13 CDCl3 /home wenbo 60
199.4598
156.4538
138.3188
129.9350
121.0706
120.9178
114.7554
26.7826
HO
O
2d (13C)
200
150
100
50
0
[ppm]
[rel]
20
15
10
5
0

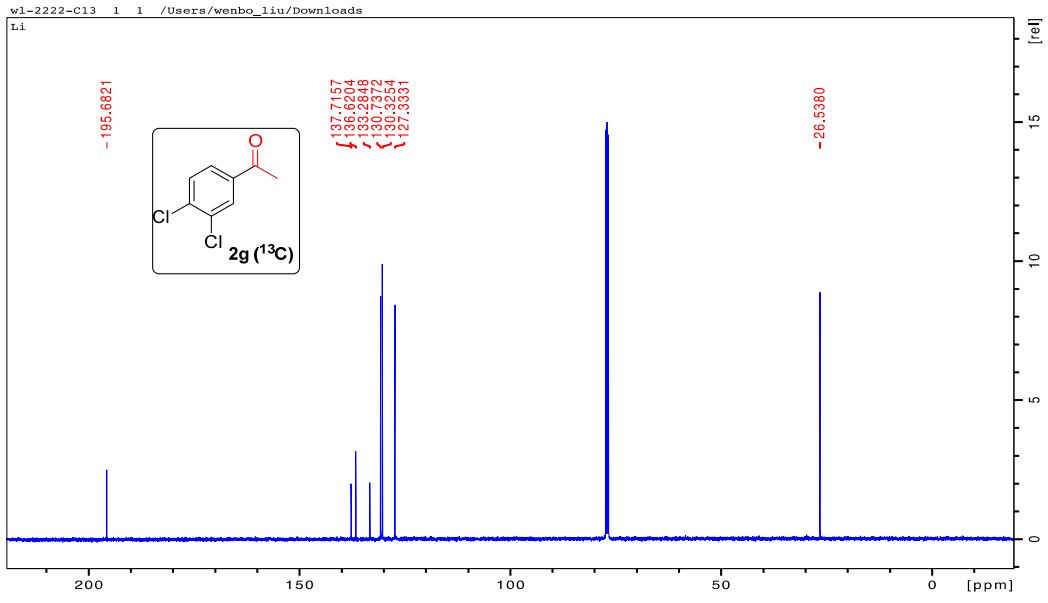

wl-2219-pure 1 1 "C:\Users\Wenbo Liu\Documents\NMR_FID"
Li
7.8319
7.8101
6.6707
6.6489
4.1905
4.1646
4.1466
4.1287
4.1109
2.5191
2e (1H)
2.0000
2.0139
1.9992
2.8902
8
6
4
[ppm]
[rel]

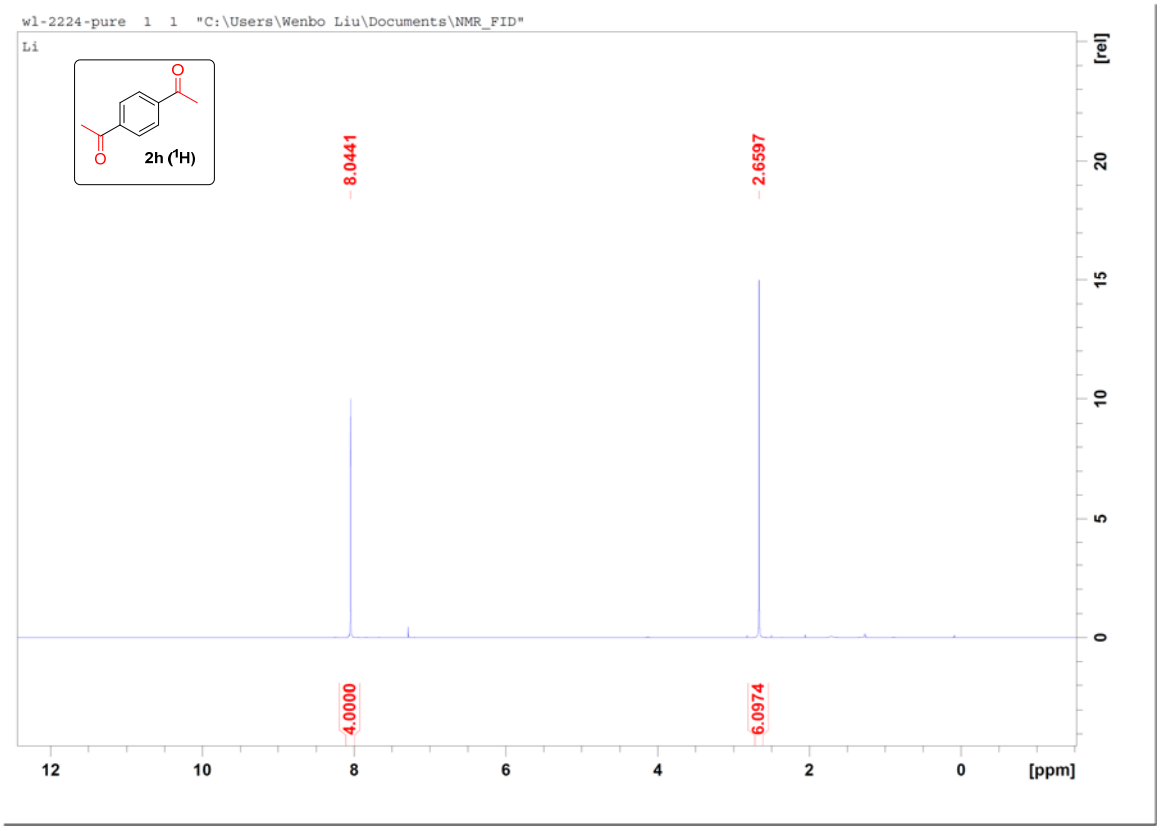

wl-2219-C13 1 1 /Users/wenbo_liu/Downloads
Li
196.5129
151.1753
130.8053
127.8516
113.7228
26.0781
2e (13C)
200
150
100
50
[ppm]
[rel]

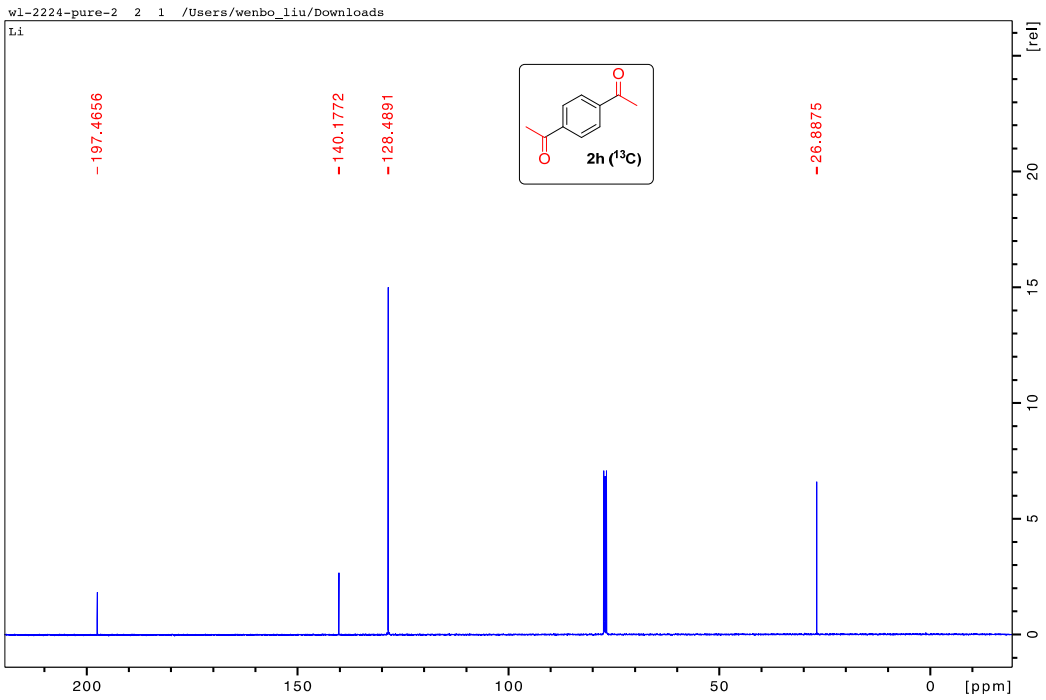

wl-2221-pure.fid 1 1 "C:\Users\Wenbo Liu\Documents\NMR_FID"
wl-2221-pure
7.4927
7.0070
2.5584
2.4803
2.2736
2.2609
2f (1H)
0.8879
0.8715
2.9401
6.0000
[rel]
[ppm]

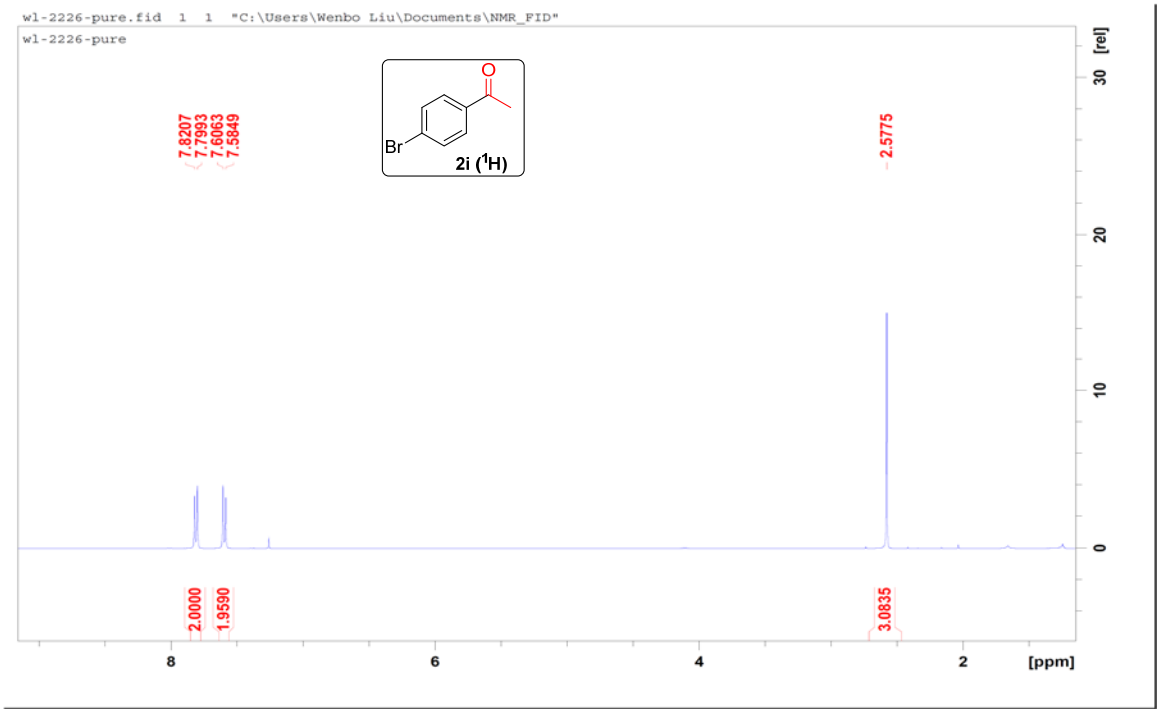

wl-2221-pure 1 1 /Users/wenbo_liu/Downloads
Li
1d_C13 CDCl3 /home wenbo 60
201.1883
140.8006
136.1909
135.0313
133.6900
133.5141
131.1066
29.3874
21.2369
19.6652
19.2646
2f (13C)
[rel]
[ppm]

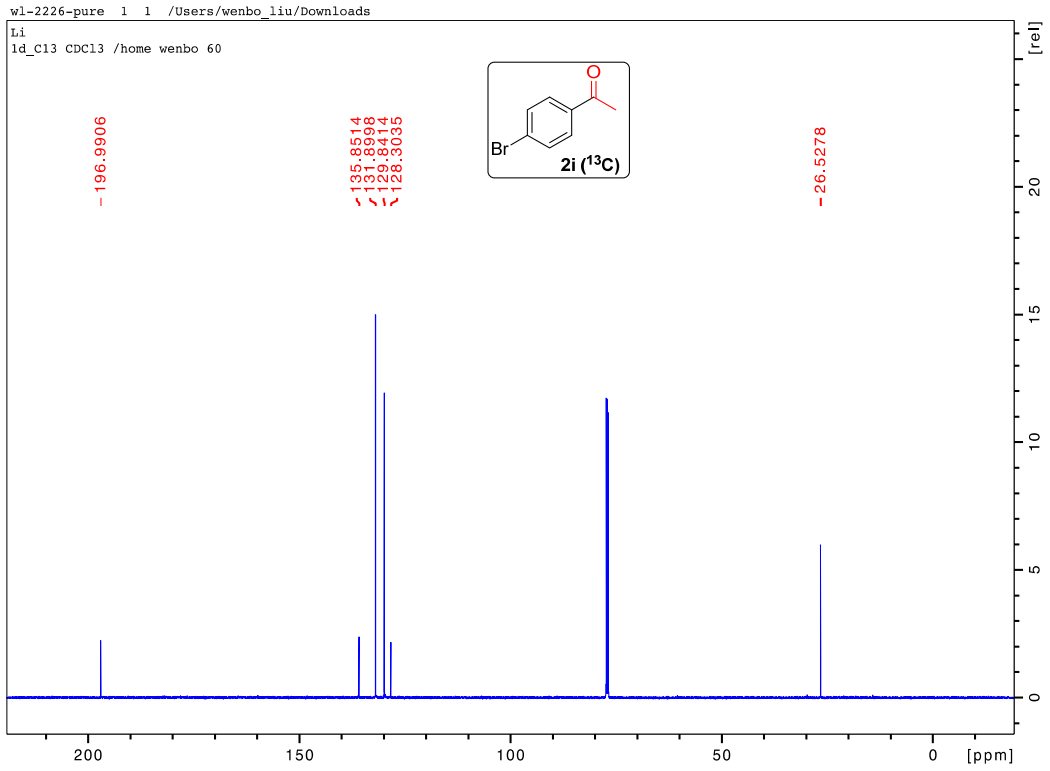

wl-2222-pure.fid 1 1 "C:\Users\Wenbo Liu\Documents\NMR_FID"
wl-2222-pure
8.0199
8.0151
7.7799
7.7750
7.7590
7.7541
7.5496
7.5287
2.5835
2g (1H)
1.0000
1.0561
1.0272
3.3220
[rel]
[ppm]


wl-2222-C13 1 1 /Users/wenbo_liu/Downloads
Li
195.6821
137.7157
136.6204
133.2848
130.7872
130.3254
127.3331
26.5380
2g (13C)
[rel]
[ppm]


wl-2224-pure 1 1 "C:\Users\Wenbo Liu\Documents\NMR_FID"
Li
2h (^{1}H)
8.0441
2.6597
4.0000
6.0974
[rel]
[ppm]


wl-2224-pure-2 2 1 /Users/wenbo_liu/Downloads
Li
197.4656
140.1772
128.4891
26.8875
2h (^{13}C)
[rel]
[ppm]


wl-2226-pure.fid 1 1 "C:\Users\Wenbo Liu\Documents\NMR_FID"
wl-2226-pure
7.8207
7.7993
7.6063
7.5849
2.5775
2i (1H)
Br
O
2.0000
1.9590
3.0835
8
6
4
2
[ppm]
30
20
10
0
[rel]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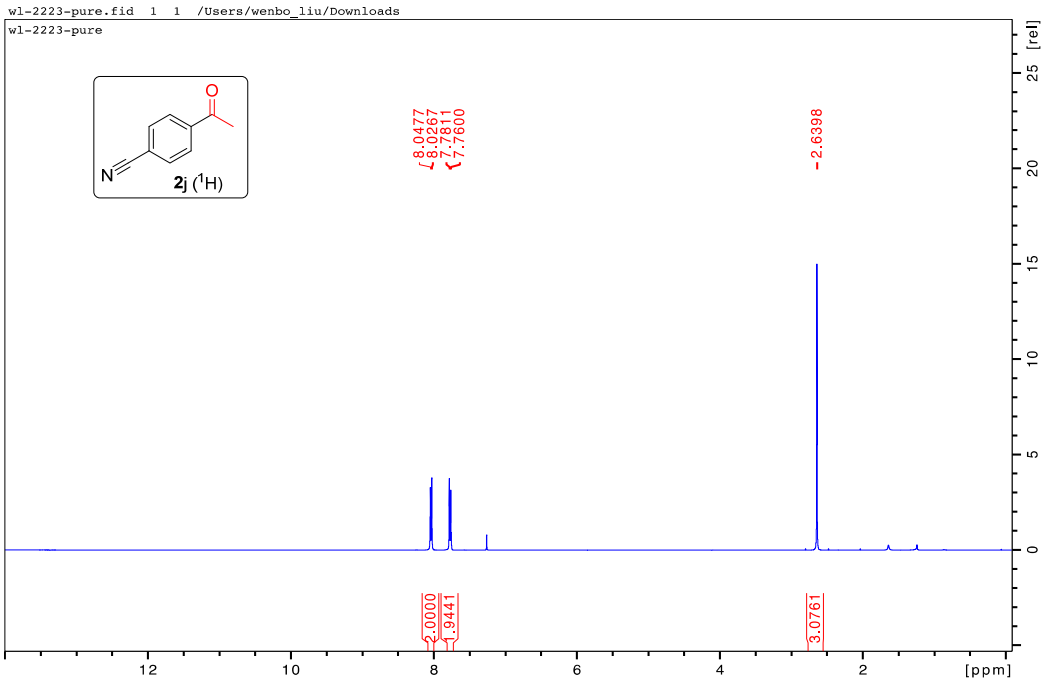
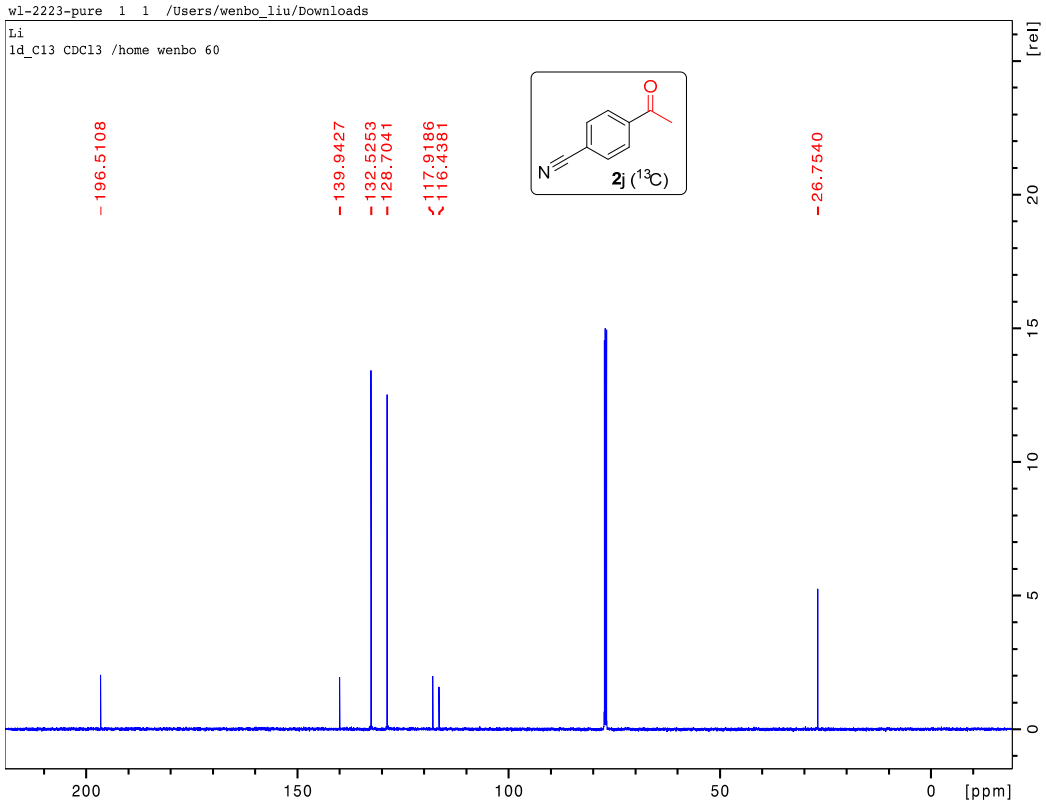
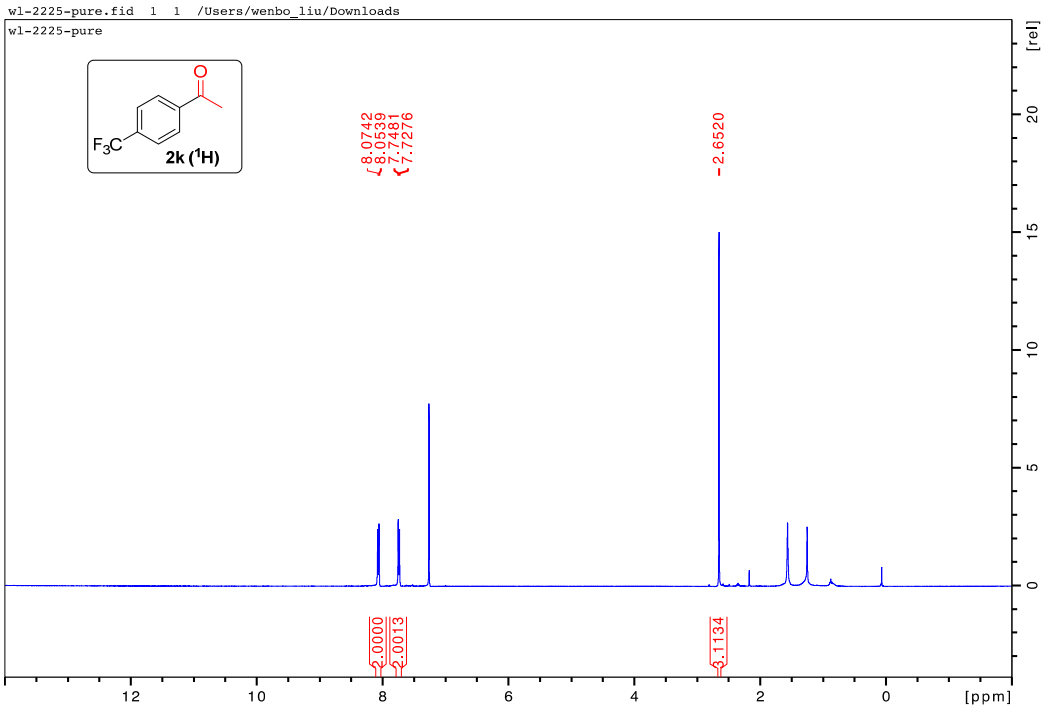
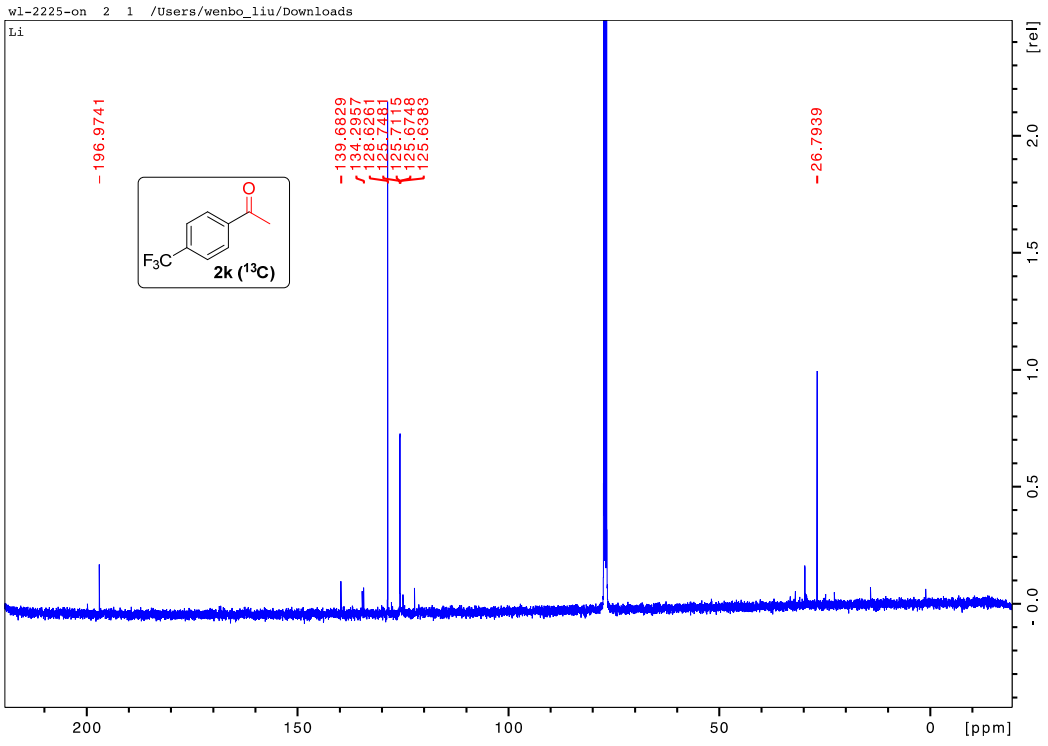
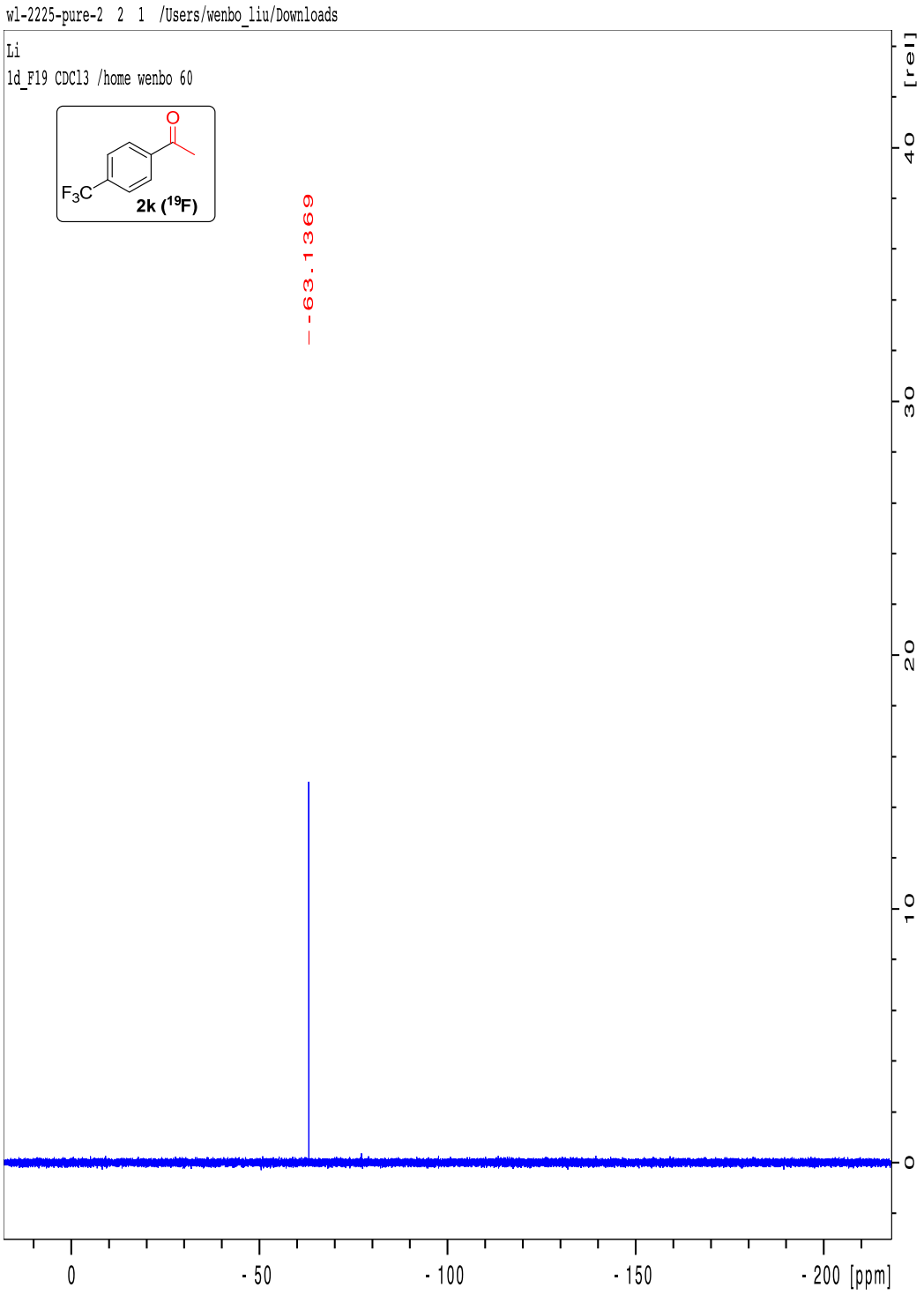
Li
1d_C13 CDCl3 /home wenbo 60
196.9906
135.8514
131.8998
129.8414
128.3035
26.5278
2i (13C)
Br
O
200
150
100
50
0
[ppm]
20
15
10
5
0
[rel]

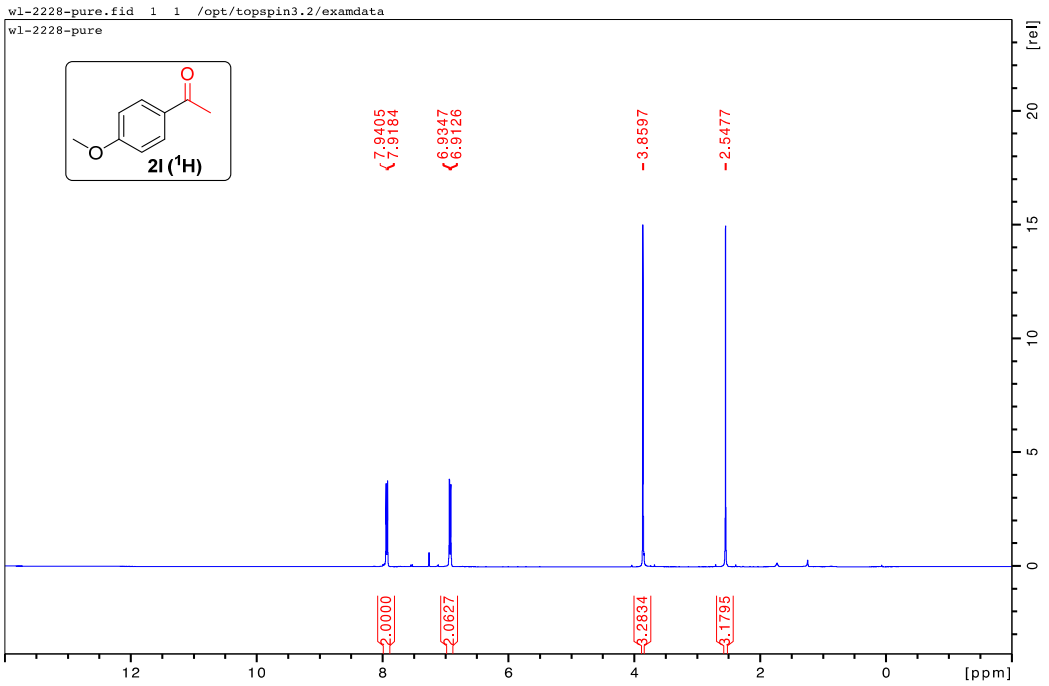

wl-2223-pure.fid 1 1 /Users/wenbo_liu/Downloads
wl-2223-pure
2j (1H)
8.0477
8.0267
7.7811
7.7600
2.6398
2.0000
1.9441
3.0761
[rel]
[ppm]

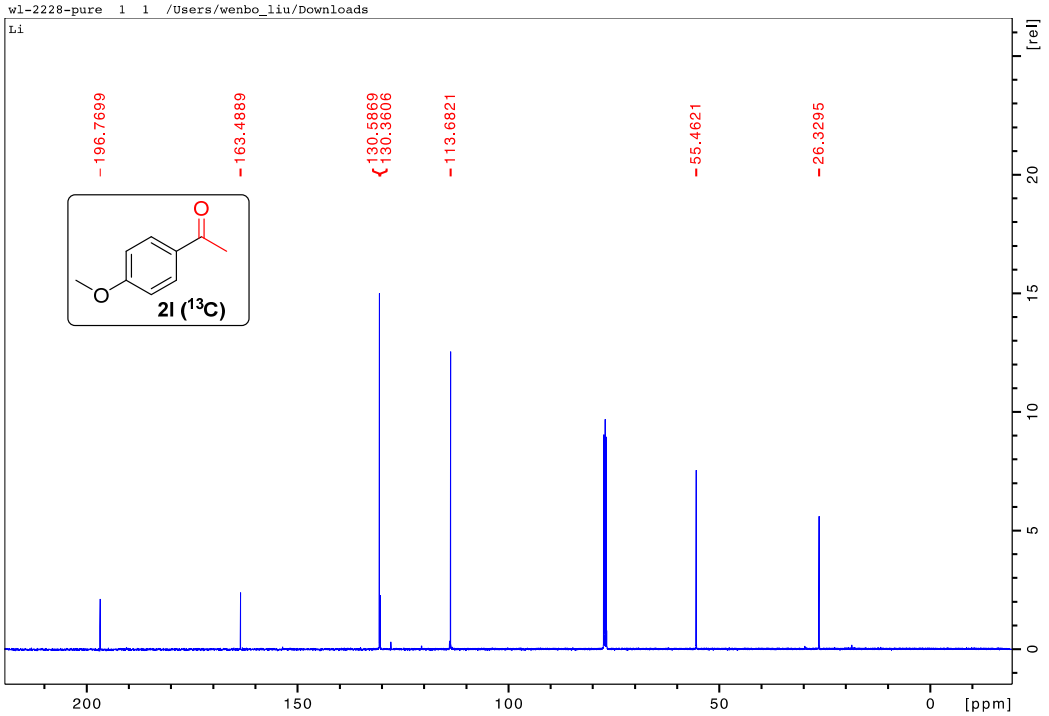

wl-2223-pure 1 1 /Users/wenbo_liu/Downloads
Li
1d_C13 CDCl3 /home wenbo 60
2j (13C)
196.5108
139.9427
132.5253
128.7041
117.9186
116.4381
26.7540
[rel]
[ppm]

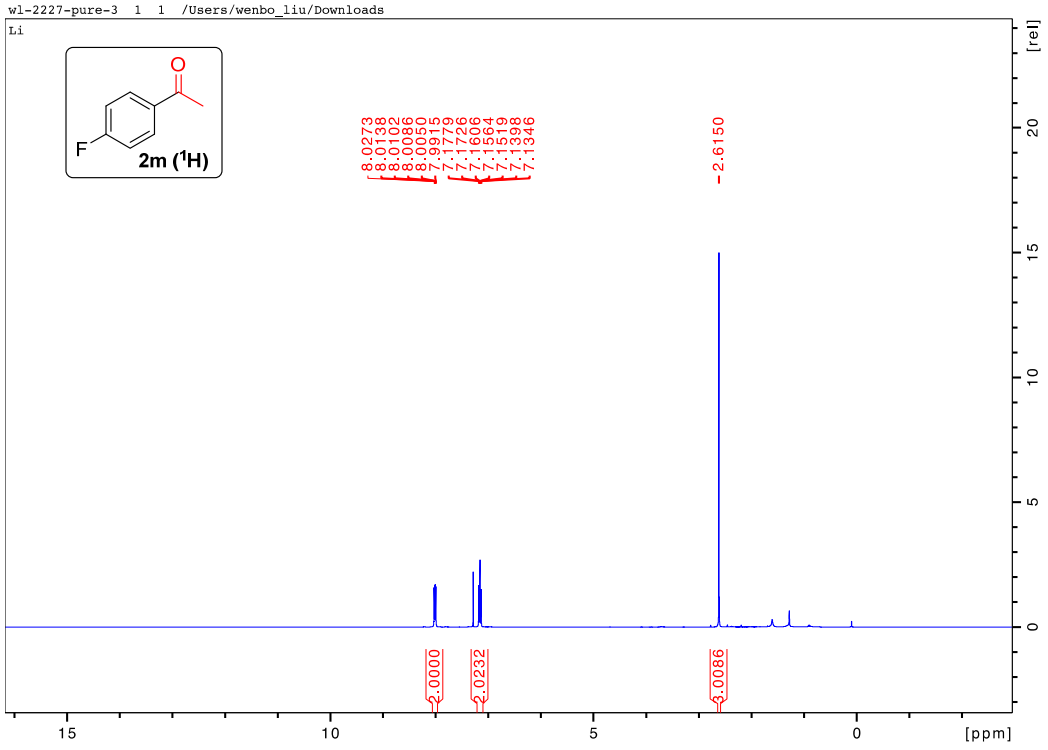

wl-2225-pure.fid 1 1 /Users/wenbo_liu/Downloads
wl-2225-pure
2k (1H)
8.0742
8.0539
7.7481
7.7276
2.6520
2.0000
2.0013
3.1134
[rel]
[ppm]

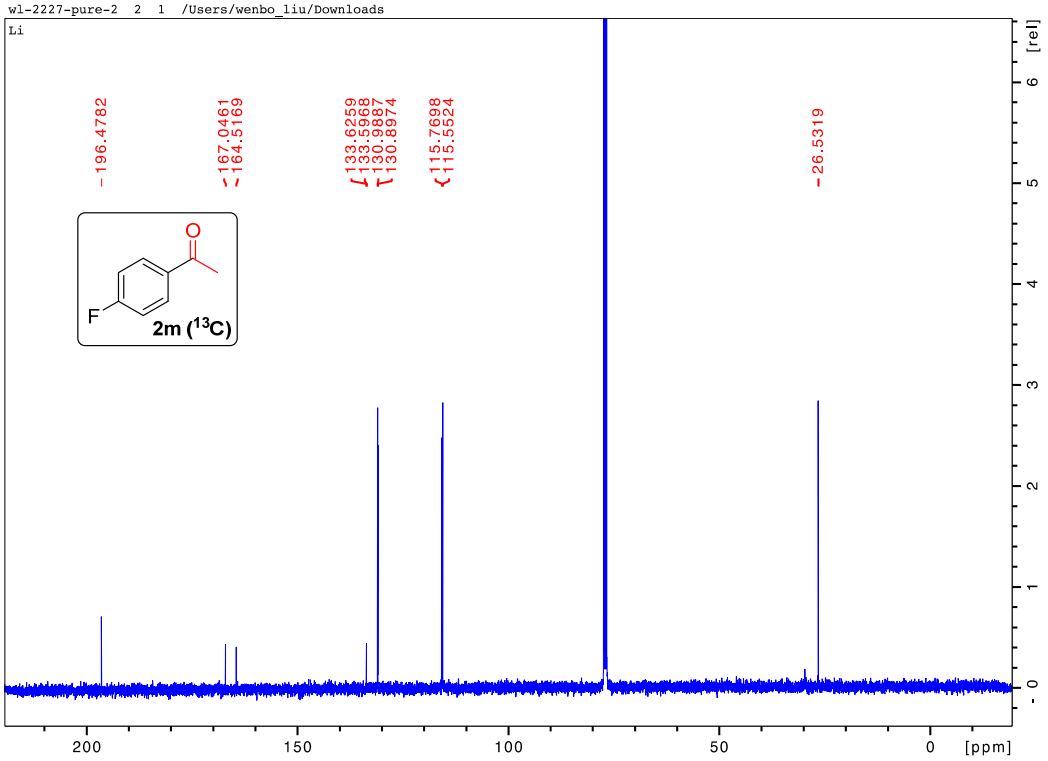

wl-2225-on 2 1 /Users/wenbo_liu/Downloads
Li
2k (13C)
196.9741
139.6829
134.2957
128.6261
125.7481
125.7115
125.6748
125.6383
26.7939
[rel]
[ppm]

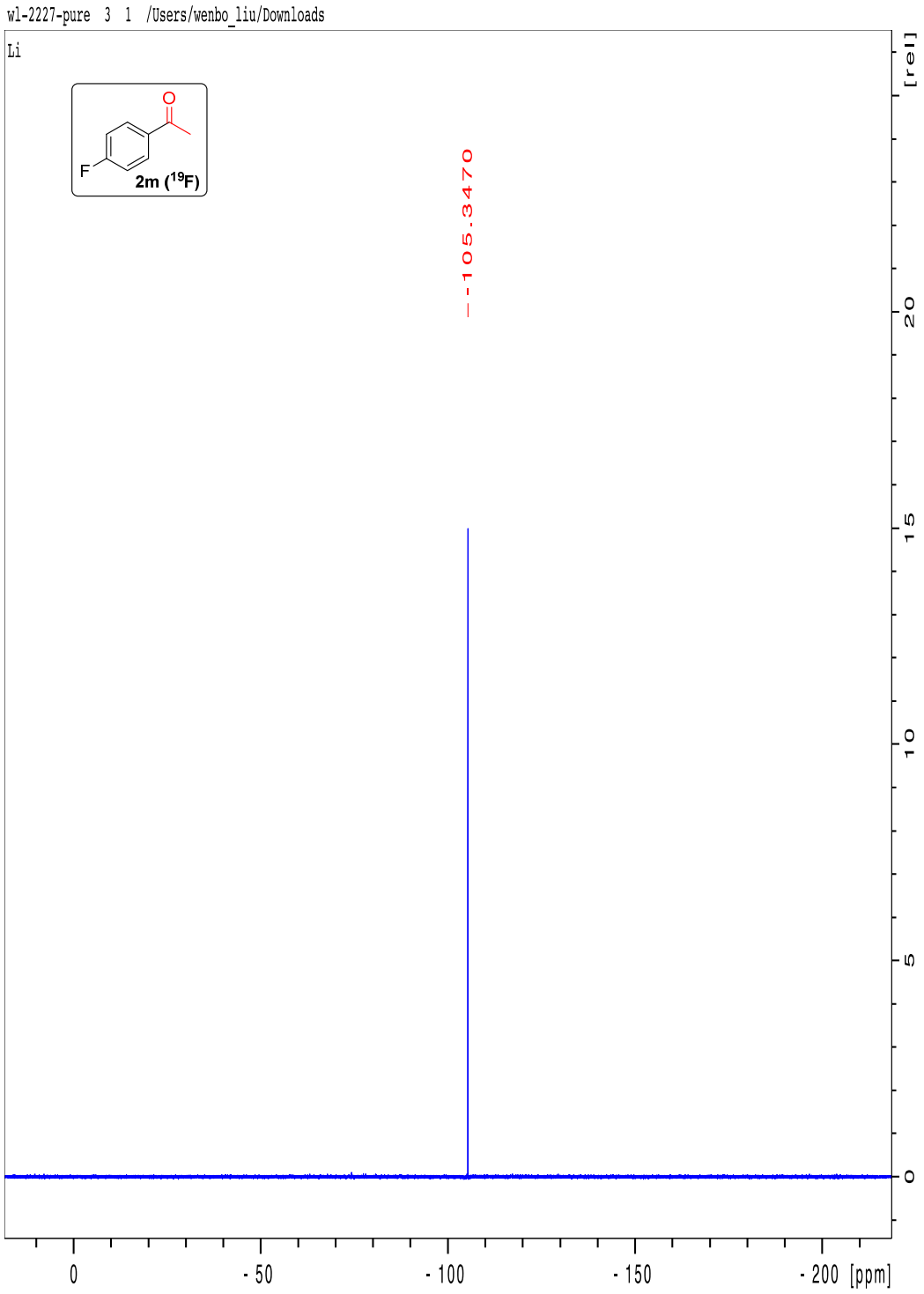

wl-2225-pure-2 2 1 /Users/wenbo_liu/Downloads
Li
1d_F19 CDCl3 /home wenbo 60
O
F3C
2k (19F)
-63.1369
[rel]
40
30
20
10
0
0
-50
-100
-150
-200 [ppm]

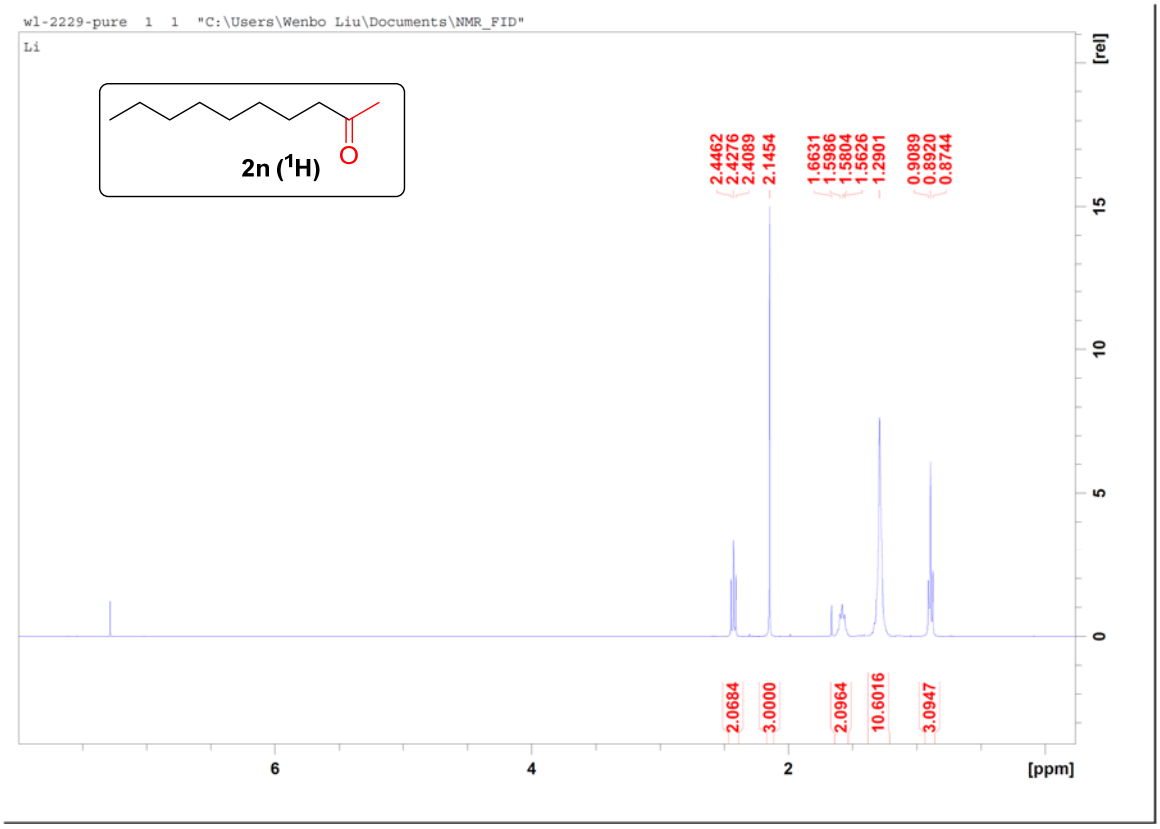

wl-2228-pure.fid 1 1 /opt/topspin3.2/examdata
wl-2228-pure
2l (^{1}H)
7.9405
7.9184
6.9347
6.9126
3.8597
2.5477
2.0000
2.0627
2.2834
3.1795
[rel]
[ppm]

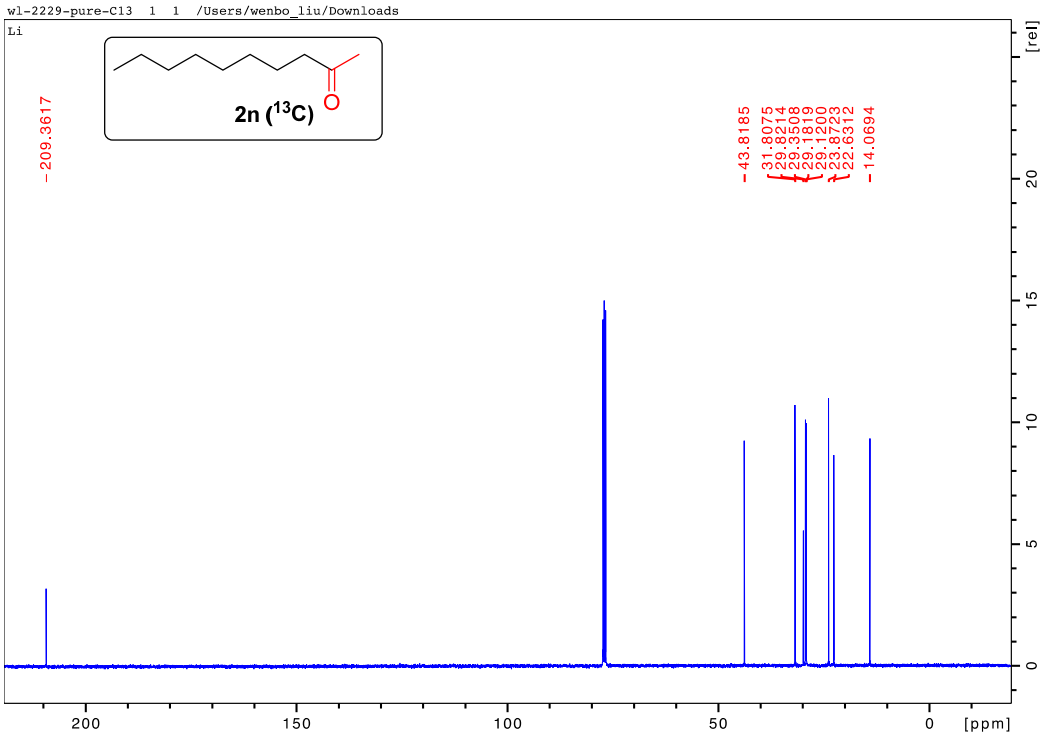

wl-2228-pure 1 1 /Users/wenbo_liu/Downloads
Li
2l (^{13}C)
196.7699
163.4889
130.5869
130.3606
113.6821
55.4621
26.3295
[rel]
[ppm]

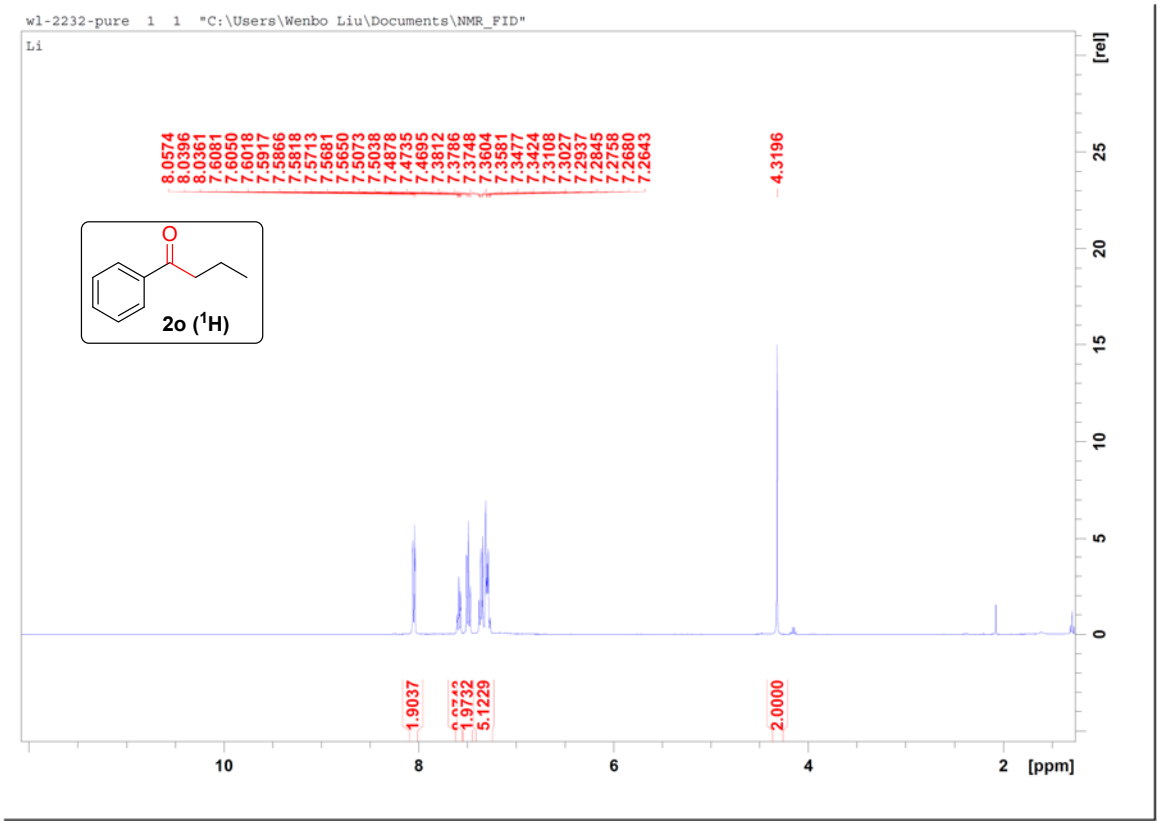

wl-2227-pure-3 1 1 /Users/wenbo_liu/Downloads
Li
2m (1H)
8.0273
8.0138
8.0102
8.0086
8.0050
7.9915
7.1779
7.1726
7.1696
7.1664
7.1519
7.1398
7.1346
2.6150
2.0000
2.0232
3.0086
[rel]
[ppm]

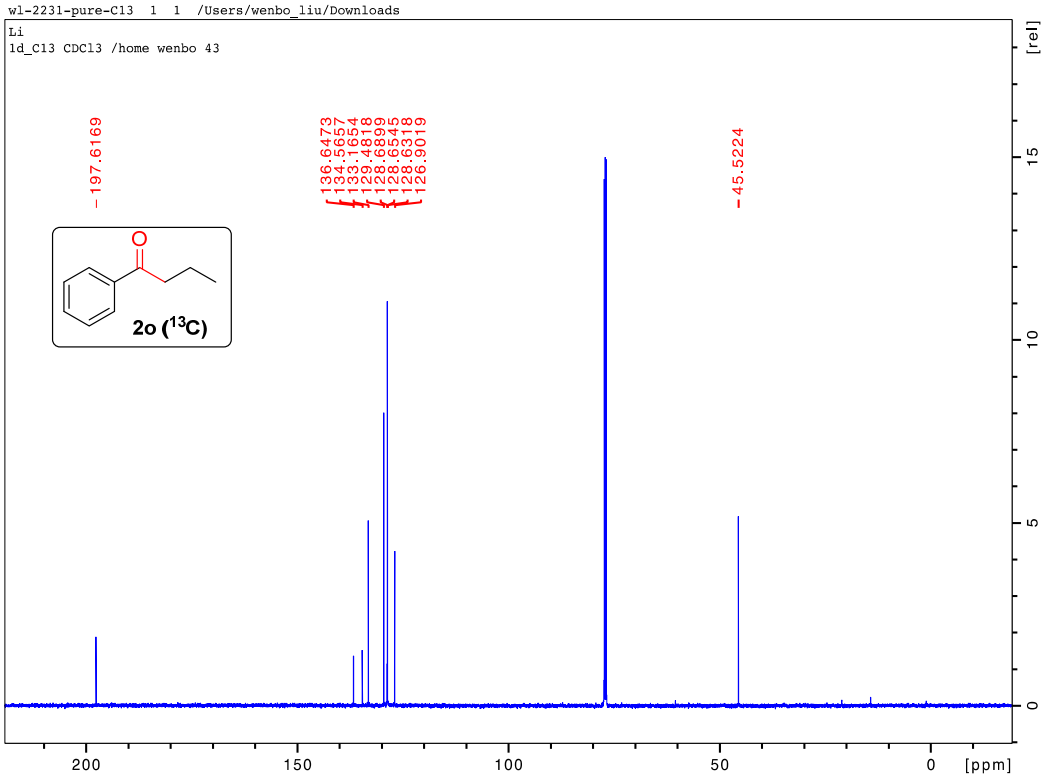

wl-2227-pure-2 2 1 /Users/wenbo_liu/Downloads
Li
2m (13C)
196.4782
167.0461
164.5169
133.6259
133.5968
130.9887
130.8974
115.7698
115.5524
26.5319
[rel]
[ppm]

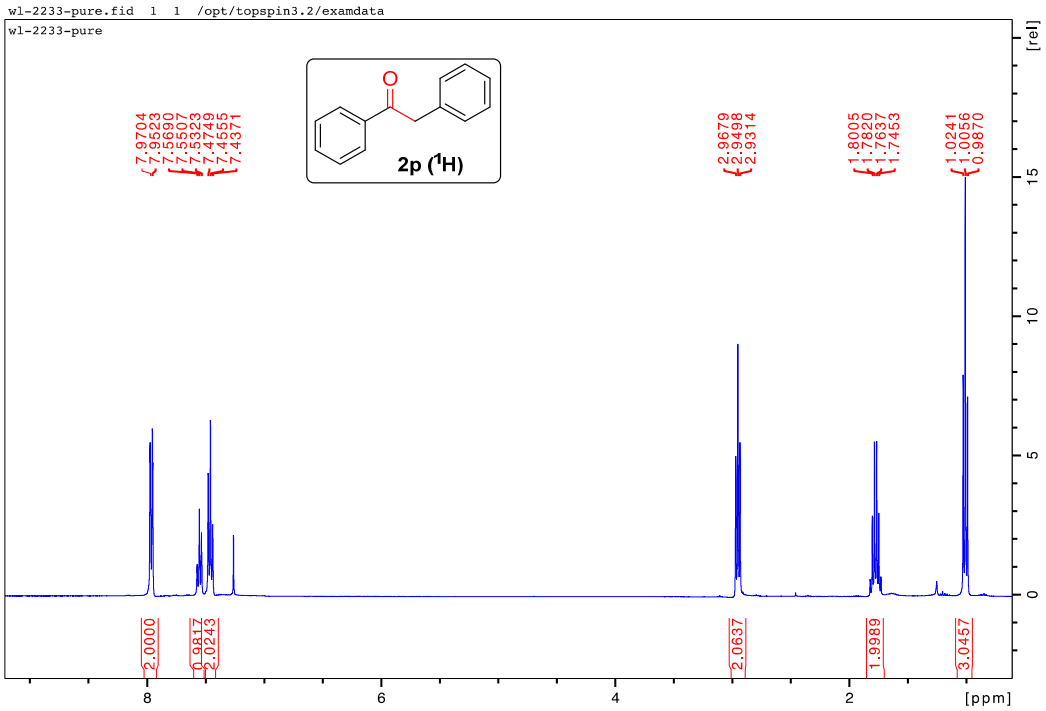

wl-2227-pure 3 1 /Users/wenbo_liu/Downloads
Li
O
F
2m (^{19}F)
-105.3470
[rel]
20
15
10
5
0
0
-50
-100
-150
-200 [ppm]

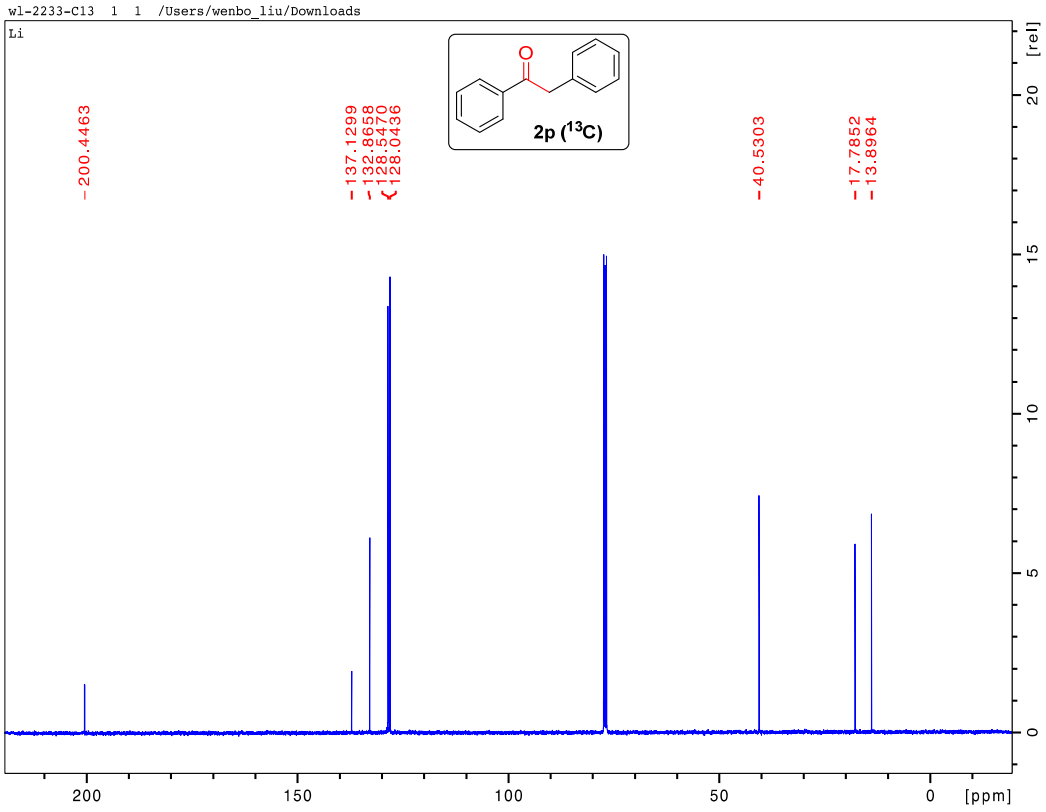

wl-2229-pure 1 1 "C:\Users\Wenbo Liu\Documents\NMR_FID"
Li
2n (^{1}H)
2.4462
2.4276
2.4089
2.1454
1.6631
1.5986
1.5804
1.5626
1.2901
0.9089
0.8920
0.8744
2.0684
3.0000
2.0964
10.6016
3.0947
[rel]
[ppm]


wl-2229-pure-C13 1 1 /Users/wenbo_liu/Downloads
Li
2n (^{13}C)
209.3617
43.8185
31.8075
29.3508
29.1819
29.1200
23.8723
22.6312
14.0694
[rel]
[ppm]


wl-2232-pure 1 1 "C:\Users\Wenbo Liu\Documents\NMR_FID"
Li
8.0574
8.0396
8.0361
7.6081
7.6050
7.6018
7.5917
7.5866
7.5818
7.5713
7.5681
7.5650
7.5073
7.5038
7.4878
7.4735
7.4695
7.3812
7.3786
7.3748
7.3604
7.3581
7.3477
7.3424
7.3108
7.3027
7.2937
7.2845
7.2758
7.2680
7.2643
4.3196
2o (1H)
1.9037
1.9732
5.1229
2.0000
[rel]
[ppm]


wl-2231-pure-C13 1 1 /Users/wenbo_liu/Downloads
Li
1d_C13 CDCl3 /home wenbo 43
197.6169
136.6473
134.5657
133.1654
129.4818
128.6899
128.6545
128.6318
126.9019
45.5224
2o (13C)
[rel]
[ppm]


wl-2233-pure.fid 1 1 /opt/topspin3.2/examdata
wl-2233-pure
2p (1H)
7.9704
7.9523
7.5690
7.5507
7.5323
7.4749
7.4555
7.4371
2.9679
2.9498
2.9314
1.8005
1.7820
1.7637
1.7453
1.0241
1.0056
0.9870
2.0000
0.0817
2.0243
2.0637
1.9989
3.0457
[rel]
[ppm]


wl-2233-C13 1 1 /Users/wenbo_liu/Downloads
Li
2p (13C)
200.4463
137.1299
132.8658
128.5470
128.0436
40.5303
17.7852
13.8964
[rel]
[ppm]