Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information:

Visible Light-Mediated Photocatalytic Synthesis of Anhydrides

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General Information

Chemicals were either used as received or purified according to *Purification of Common Laboratory Chemicals*. All reactions were performed using common dry, inert atmosphere techniques. NMR spectra were recorded on Varian Unity Plus 400, and Varian Mercury 500 spectrometers. Chemical shifts for ^{1}H NMR were reported as δ, parts per million, relative to the signal of residual $CHCl_3$ in $CDCl_3$ at 7.26 ppm. Chemical shifts for ^{13}C NMR were reported as δ, parts per million, relative to the center line signal of the $CDCl_3$ triplet at 77.0 ppm. Proton and carbon assignments were established using spectral data of similar compounds. The abbreviations s, d, dd, t and m stand for the resonance multiplicity singlet, doublet, doublet of doublets, triplet and multiplet, respectively. Infrared spectra were recorded on a Nicolet Nexus 670 FT-IR with ATR spectrophotometer. Absorptions are given in wavenumbers (cm^{-1}). High resolution mass spectra were obtained on a Waters Q-Tof API-US with ESI high resolution mass spectrometer. HPLC analysis was performed on an Agilent 1100 series HPLC System with a diode array detector. Melting points were recorded on a Mel-temp (Laboratory Devices).

Reaction Apparatus

Photoredox reactions were carried out under visible light irradiation by a 15 cm blue LED strip (available from http://www.creativelightings.com/, λmax = 435 nm) surrounding the reaction vessel.

General Procedure

A 10 mL round bottom flask was equipped with a rubber septum and magnetic stir bar and was charged with carboxylic acid (0.5 mmol, 1.0 equiv), carbon tetrabromide (0.5 mmol, 1.0 equiv), 2,6-lutidine (1.0 mmol, 2.0 equiv), and $Ru(bpy)_3Cl_2$ (5.0 μmol, 0.010 equiv). The flask was purged with a stream of Ar and dry DMF (5.0 mL) was added with a syringe. The mixture was degassed by the freeze-pump-thaw procedure (three cycles). The homogenous mixture was then irradiated by a 1 W blue LED strip under an atmosphere of Ar for 12 h (the reaction reaches temperatures between 25 and 30 °C upon exposure to the blue LEDs).

After the reaction was complete, the mixture was poured into a separatory funnel containing 25 mL of Et_2O and 25 mL of H_2O. The layers were separated and the aqueous layer was extracted with Et_2O (2 × 50 mL). The combined organic layers were washed with saturated $NaHCO_3$, H_2O, brine, dried (Na_2SO_4) and concentrated *in vacuo* to afford the desired anhydride.

Procedure for Large Scale Reaction

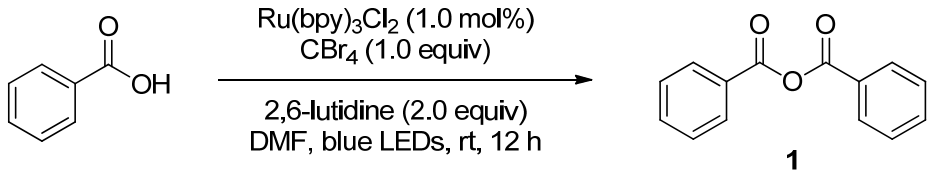
A 250 mL round bottom flask was equipped with a rubber septum and was charged with 4-*tert*-butylbenzoic acid (2.5 g, 14 mmol, 1.0 equiv), CBr_4 (4.6 g, 14 mmol, 1.0 equiv), 2,6-lutidine (3.3 mL, 28 mmol, 2.0 equiv), and $Ru(bpy)_3Cl_2$ (21 mg, 28 μmol, 0.0020 equiv). The flask was purged with a stream of Ar and dry DMF (60 mL) was added with a syringe, and then degassed by the freeze-pump-thaw procedure (three cycles). The homogenous mixture was then irradiated by four 1 W blue LED strips wrapped around in a crystallizing dish (Pyrex®, 125*65) under an atmosphere of Ar for 18 h at 30 °C. After the reaction was complete, the mixture was poured into a separatory funnel containing 200 mL of Et_2O and 100 mL of H_2O. The layers were separated and the aqueous layer was extracted with Et_2O (2 × 100 mL). The combined organic layers were washed with saturated $NaHCO_3$, H_2O, brine and were dried (Na_2SO_4). The solvent was removed *in vacuo*, and the residue was purified by chromatography on SiO_2 (9:1, petroleum ether/Et_2O) to afford the desired anhydride as a pale yellow solid.

Procedure for Anhydride Formation in Flow

A 10 mL round bottom flask was equipped with a rubber septum and was charged with 4-*tert*-butylbenzoic acid (0.5 mmol, 1.0 equiv), CBr_4 (0.5 mmol, 1.0 equiv), 2,6-lutidine (1.0 mmol, 2.0 equiv), and $Ru(bpy)_3Cl_2$ (5.0 μmol, 0.010 equiv). The flask was purged with a stream of Ar and dry DMF (5.0 mL) was added with a syringe, and then degassed by the freeze-pump-thaw procedure (three cycles). The reaction mixture was pumped through the photoreactor[1] with a residence time of 6.4 min and then collected in a flask. The mixture was poured into a separatory funnel containing 25 mL of Et_2O and 50 mL of H_2O. The layers were separated and the aqueous layer was extracted with Et_2O (2 × 50 mL). The combined organic layers were dried (Na_2SO_4) and concentrated. The solvent was removed *in vacuo*.

[1] For information about the photoreactor and the experiment setup, see: J. W. Tucker, Y. Zhang, T. F. Jamison, C. R. J. Stephenson, *Angew. Chem. Int. Ed.,* 2012, **124**, 1-5.

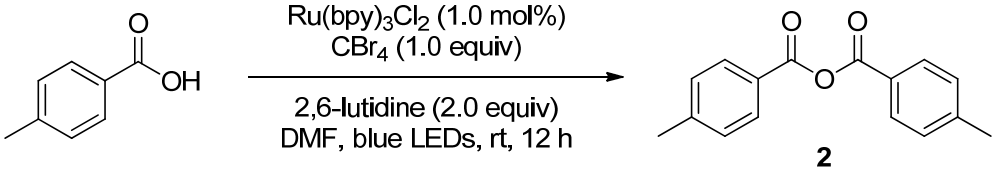
Benzoic anhydride, 1[2] (*Table 2, entry 1):*



According to General Procedure, benzoic acid (61.1 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.50 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **1** (56.5 mg, 99%) as a light yellow solid.

m.p.: 39-41 °C (Aldrich: 38-42 °C);
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.18–8.15 (m, 4 H), 7.70–7.67 (m, 2 H), 7.56–7.52 (m, 4 H);
^{13}C NMR ($CDCl_3$, 125 MHz): δ 160.5, 133.4, 129.5, 127.9, 127.9;
IR (neat): ν_{max} 3071, 2918, 1782, 1723, 1210, 1172, 1037, 994 cm^{-1}.

4-Methylbenzoic anhydride, 2[3] (*Table 2, entry 2):*

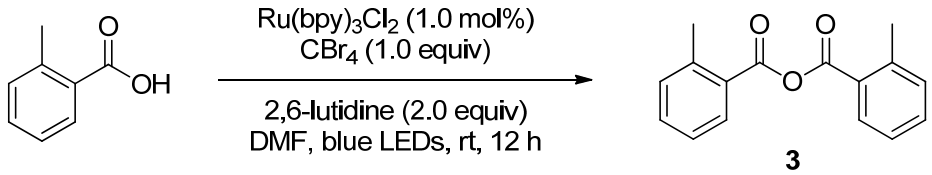


According to General Procedure, 4-methylbenoic acid (68.1 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.50 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **2** (62.8 mg, 98%) as a light yellow solid.

m.p.: 88-90 °C (lit[4]: 89-90 °C);
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.05–8.03 (m, 4 H), 7.31 (d, J = 8.0 Hz, 4 H), 2.46 (s, 6 H);
^{13}C NMR ($CDCl_3$, 125 MHz): δ 160.7, 144.1, 129.6, 128.6, 125.3, 23.7;
IR (neat): ν_{max} 3041, 2916, 1774, 1712, 1609, 1225, 1173, 1049, 1005, 750 cm^{-1}.

[2] Y.-D. Park, J.-J. Kim, H.-K. Kim, S.-D. Cho, Y.-J. Kang, K. H. Park, S.-G. Lee, Y.-J. Yoon, *Synth. Commun.*, 2005, **35**, 371-378.
[3] Y. Li, D. Xue, C. Wang, Z.-T. Liu, J. Xiao, *Chem. Commun.,* 2012, **38**, 1320–1322.
[4] J.-J. Kim, Y.-D. Park, W. S. Lee, S.-D. Cho, Y.-J. Yoon, *Synthesis,* 2003, 1517-1520.

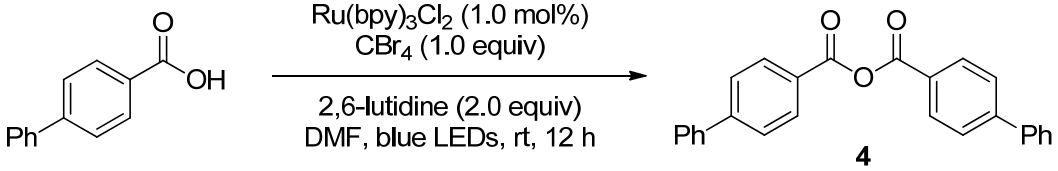
2-Methylbenzoic anhydride, 3[3] (*Table 2, entry 3):*



According to General Procedure, 2-methylbenzoic acid (68.1 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.50 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **3** (61.7 mg, 97%) as a light yellow solid.

m.p.: 37-39 °C (lit[5]: 38-39 °C);
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.07–8.04 (m, 2 H), 7.53–7.49 (m, 2 H), 7.34–7.29 (m, 4 H), 2.70 (s, 6 H);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 162.9, 142.6, 133.6, 132.3, 131.4, 127.7, 126.1, 22.0;
IR (neat): ν_{max} 2968, 2931, 1783, 1723, 1199, 983, 741 cm^{-1}.

4-Phenylbenzoic anhydride, 4[4] (*Table 2, entry 4):*

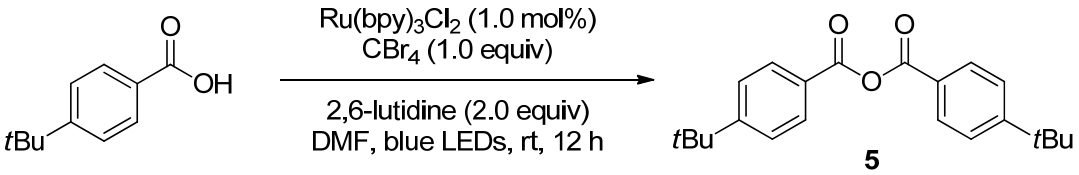


According to General Procedure, 4-phenylbenzoic acid (99.1 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.50 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **4** (90.0 mg, 95%) as a light yellow solid.

m.p.: 136-138 °C (lit[3]: 137-139 °C);
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.26–8.23 (m, 4 H), 7.77–7.75 (m, 4 H), 7.67–7.65 (m, 4 H), 7.52–7.49 (m, 4 H), 7.45–7.42 (m, 2 H);
^{13}C NMR ($CDCl_3$, 125 MHz): δ 162.3, 147.3, 139.6, 131.1, 129.1, 128.6, 127.5 (two carbons overlapping), 127.4;
IR (neat): ν_{max} 3030, 2927, 1778, 1717, 1605, 1226, 1175, 1055, 1002, 743 cm^{-1}.

[5] J. Cabré-Castellví, A. Palomo-Coll, A. L. Palomo-Coll, *Synthesis* 1981, **8**, 616-620.

4-(*tert*-butyl)benzoic anhydride, 5[3] (*Table 2, entry 5):*

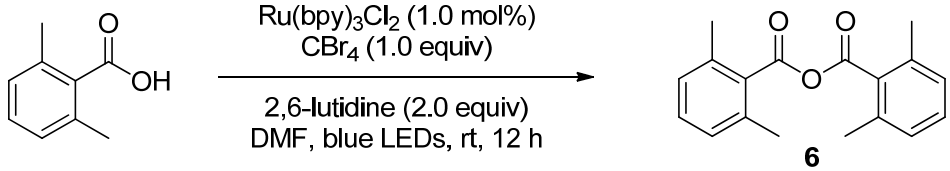


5

According to General Procedure, 4-(*tert*-butyl)benoic acid (89.1 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.50 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **5** (83.7 mg, 99%) as a light yellow oil.

m.p.: 75-77 °C (lit[6]: 78.7-79.0 °C);
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.08 (d, J = 8.8 Hz, 4 H), 7.54 (d, J = 8.8 Hz, 4 H), 1.36 (s, 18 H);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 162.5, 158.5, 130.6, 126.1, 125.9, 35.3, 31.1;
IR (neat): ν_{max} 2963, 2905, 2868, 1782, 1722, 1606, 1222, 1177, 1107, 1038, 993, 701 cm^{-1}.

2,6-dimethylbenzoic anhydride, 6 (*Table 2, entry 6):*



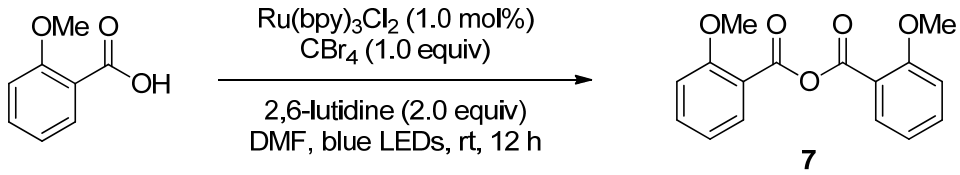
6

According to General Procedure, 2,6-dimethylbenoic acid (75.1 mg, 0.5 mmol), carbon tetrabromide (165.8 mg, 0.5 mmol), 2,6-lutidine (116.5 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **6** (67.8 mg, 96%) as a light yellow solid.

m.p.: 76-78 °C;
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.26–7.23 (m, 3 H), 7.07–7.05 (d, J = 7.6 Hz, 3 H), 2.41 (s, 12 H);
^{13}C NMR ($CDCl_3$, 125 MHz): δ 163.2, 134.7, 130.7, 129.4, 127.0, 21.9;
IR (neat): ν_{max} 2926, 2359, 1798, 1738, 1595, 1466, 1201, 1166, 1093, 976, 780 cm^{-1};
MS (ESI) *m/z* calcd for $C_{18}H_{18}O_3Na$ ($[M + Na]^+$) 305.1154, found 305.1157.

[6] E. Berliner, L. H. Altschul, *J. Am. Chem. Soc.,* 1952, **74**, 4110-4113.

2-Methoxybenzoic anhydride, 7[3] (*Table 2, entry 7):*

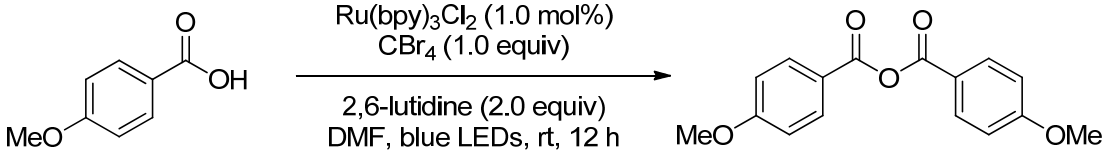


7

According to General Procedure, 2-methoxybenoic acid (76.1 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.5 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **7** (65.1 mg, 91%) as a light yellow solid.

m.p.: 70-71°C (lit[3]: 72-74 °C);
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.02 (dd, J = 7.8, 1.8 Hz, 2 H), 7.55–7.51 (m, 2 H), 7.03–6.97 (m, 4 H), 3.83 (s, 6 H);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 161.9, 160.1, 135.3, 133.1, 120.3, 118.1, 112.1, 55.9.
IR (neat): ν_{max} 2918, 2848, 1781, 1730, 1600, 1489, 1256, 1199, 1020, 879, 756 cm^{-1};

4-Methoxybenzoic anhydride, 8[3] (*Table 2, entry 8):*

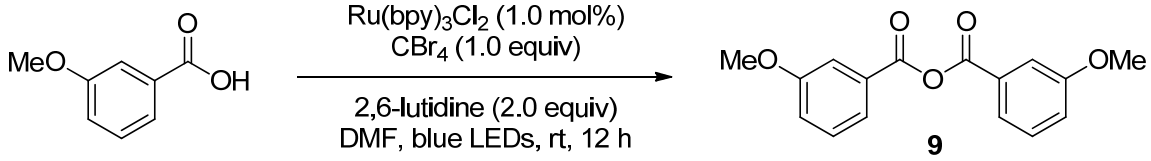


According to General Procedure, 4-methoxybenoic acid (76.1 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.50 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **8** (64.1 mg, 90%) as a light yellow solid.

m.p.: 94-96 °C (lit[7]: 97-98 °C);
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.11–8.09 (m, 4 H), 6.99–6.98 (m, 4 H), 3.90 (s, 6 H);
^{13}C NMR ($CDCl_3$, 125 MHz): δ 162.6, 160.4, 131.7, 120.5, 113.6, 56.6;
IR (neat): ν_{max} 2936, 2842, 1774, 1712, 1604, 1510, 1221, 1759, 1021, 991, 839 cm^{-1}.

[7] Z. J. Kamiński, B. Kolesińska, M. Marcinkowska, *Syn. Commun.,* 2004, **34**, 3349-3358.

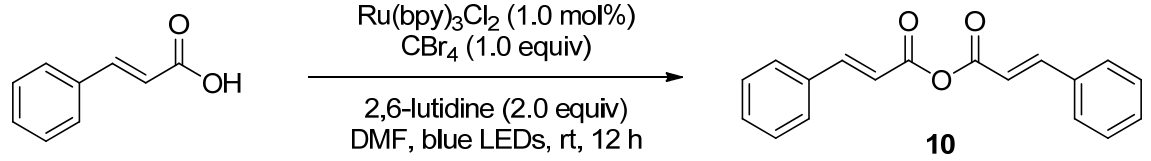
3-Methoxybenzoic anhydride, 9[3] (*Table 2, entry 9):*



According to General Procedure, 3-methoxybenoic acid (76.1 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.50 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **9** (65.8 mg, 92%) as a light yellow solid.

m.p.: 64-66 °C (lit[3]: 65-66 °C);
^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.67–7.64 (m, 2 H), 7.58–7.56 (m, 2 H), 7.34 (t, J = 8.0 Hz, 2 H), 7.15 – 7.11 (m, 2 H), 3.79 (s, 6 H);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 162.2, 159.9, 130.0, 129.9, 122.9, 121.1, 114.9, 55.6;
IR (neat): ν_{max} 2918, 2849, 1787, 1724, 1600, 1585, 1261, 1194, 1021, 991, 748 cm^{-1}.

***Trans*-cinnamic anhydride, 10**[4] (*Table 2, entry 10):*

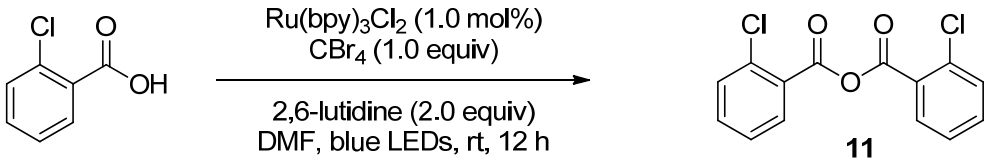


According to General Procedure, *trans*-cinnamic acid (74.1 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.50 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **10** (62.8 mg, 90%) as a light yellow solid.

m.p.: 134-136 °C (lit[8]: 135-136 °C);
^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.86 (d, J = 15.9 Hz, 2 H), 7.60–7.58 (m, 4 H), 7.46–7.43 (m, 6 H), 6.53 (d, J = 15.9 Hz, 2 H);
^{13}C NMR ($CDCl_3$, 125 MHz): δ 160.6, 147.1, 132.6, 130.2, 128.1, 127.6, 116.1;
IR (neat): ν_{max} 3060, 3027, 2919, 2849, 1764, 1700, 1630, 1450, 1067, 959 cm^{-1}.

[8] K. S. Keshavamurthy, Y. D. Vankar, D. N. Dhar, *Synthesis*, 1982, **6**, 506-508.

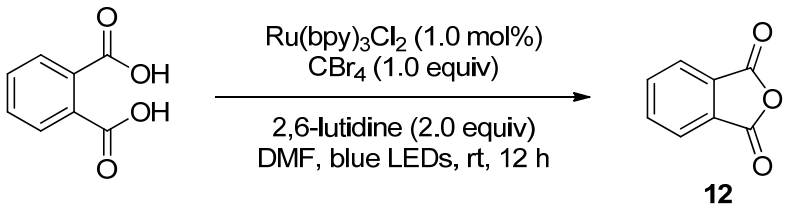
2-Chlorobenzoic anhydride, 11[3] (*Table 2, entry 11):*



According to General Procedure, 2-chlorobenzoic acid (78.3 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.50 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **11** (45.0 mg, 61%) as a light yellow oil.

m.p.: 66-69 °C (lit[3]: 63-68 °C);
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.03–8.02 (m, 2 H), 7.54–7.53 (m, 4 H), 4.41–7.38 (m, 2 H);
^{13}C NMR ($CDCl_3$, 125 MHz): δ 158.6, 134.0, 133.1, 131.5, 130.6, 127.0, 126.0;
IR (neat): ν_{max} 2955, 2913, 1794, 1697, 1589, 1437, 1207, 1075, 989, 742 cm^{-1}.

Phthalic anhydride, 12[9] (*Table 2, entry 12):*

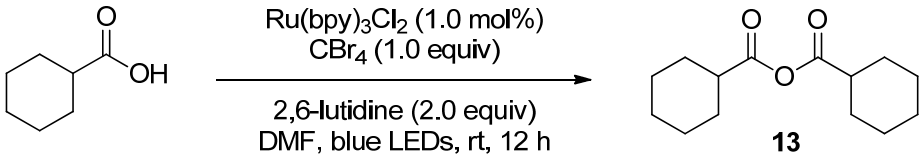


According to General Procedure, phthalic acid (83.1 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.50 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **12** (36.9 mg, 99%) as a light yellow solid.

m.p.: 130-132 °C (Aldrich: 131-134 °C);
^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.05–8.01 (m, 2 H), 7.93–7.90 (m, 2 H);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 162.8, 136.1, 131.3, 125.8;
IR (neat): ν_{max} 2918, 2852, 1849, 1761, 1471, 1256, 1110, 905, 712 cm^{-1}.

[9] (a) A. Schinkovitz, S. M. Pro, N. Main, S.-N. Chen, B. U. Jaki, D. C. Lankin, G. F. Pauli, *J. Nat. Prod.*, 2008, **71**, 1604–1611; (b) M. J. Fifolt, S. A. Sojka, R. A. Wolfe, *J. Org. Chem.,* 1982, **47**, 148–151.

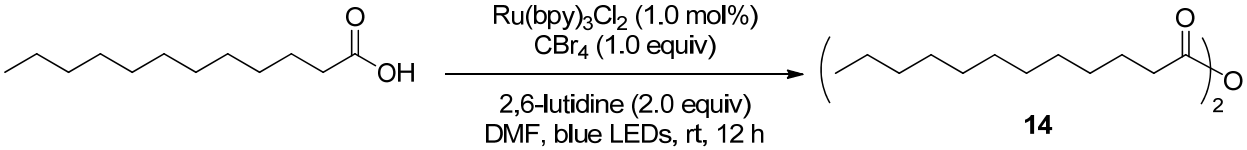
Cyclohexanecarboxylic anhydride, 13[10] (*Table 2, entry 13):*



According to General Procedure, cyclohexanecarboxylic acid (64.1 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.50 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **13** (58.7 mg, 98%) as a light yellow liquid.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 2.40 (tt, J = 11.2, 3.7 Hz, 2 H), 1.97–1.93 (m, 4 H), 1.80–1.77 (m, 4 H), 1.66–1.63 (m, 2 H), 1.52–1.43 (m, 4 H), 1.34–1.22 (m, 6 H);
^{13}C NMR ($CDCl_3$, 125 MHz): δ 169.7, 45.3, 30.1, 27.4, 27.0;
IR (neat): ν_{max} 2932, 2856, 1808, 1741, 1451, 1082, 990, 895 cm^{-1};
MS (ESI) *m/z* calcd for $C_{14}H_{22}O_3Na$ ($[M + Na]^+$) 261.1467, found 4261.1468.

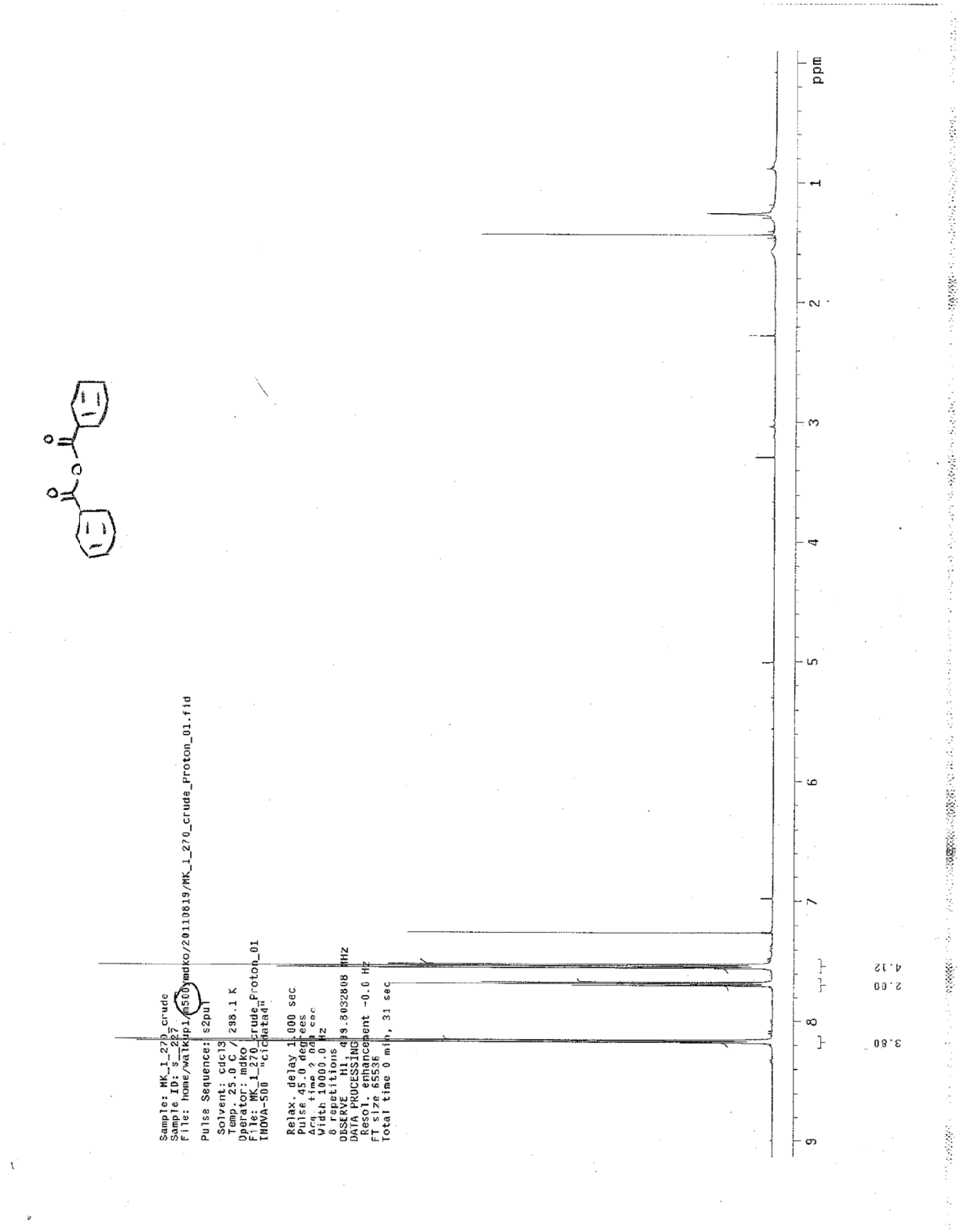
Lauric anhydride, 14 (*Table 2, entry 14):*

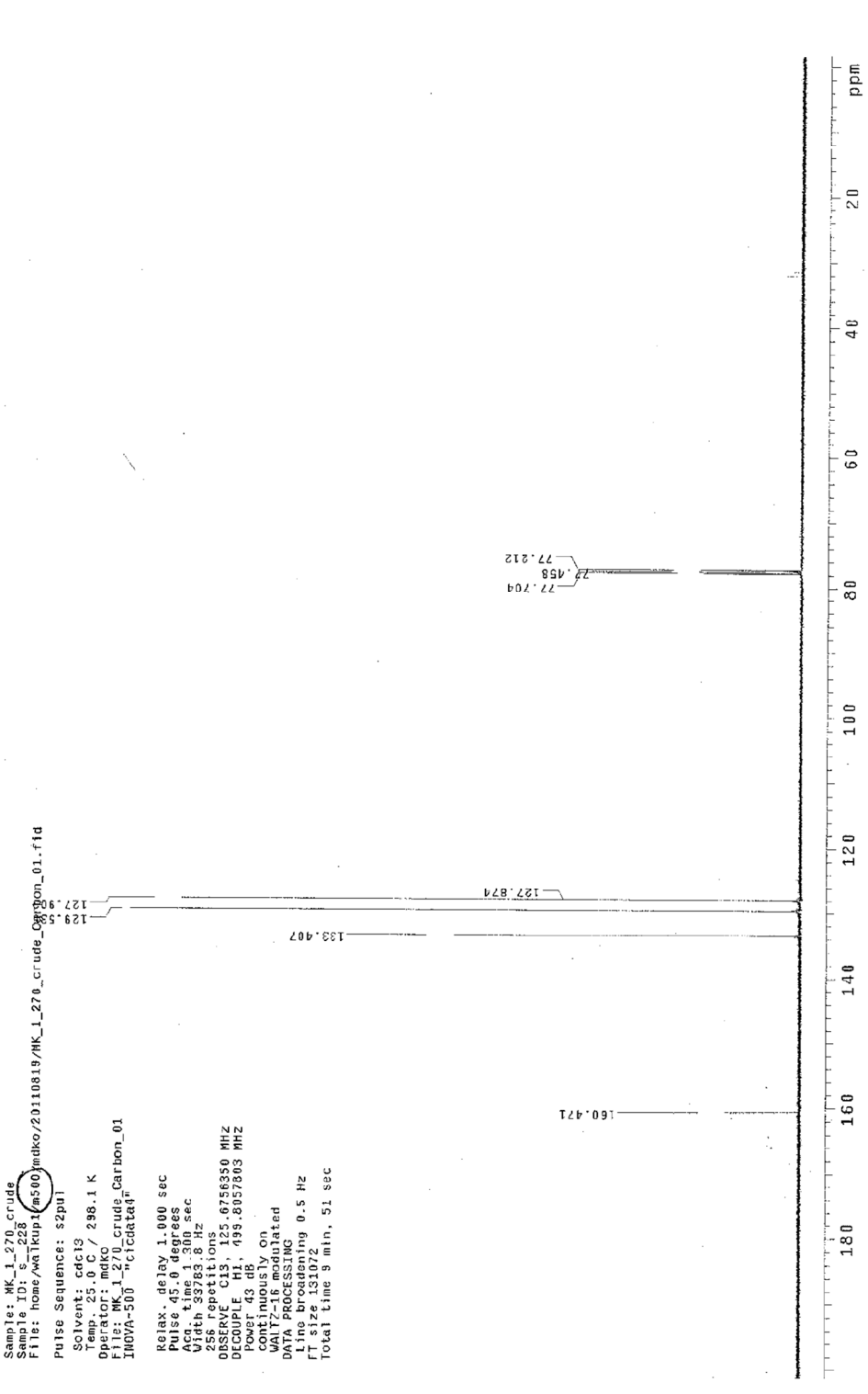


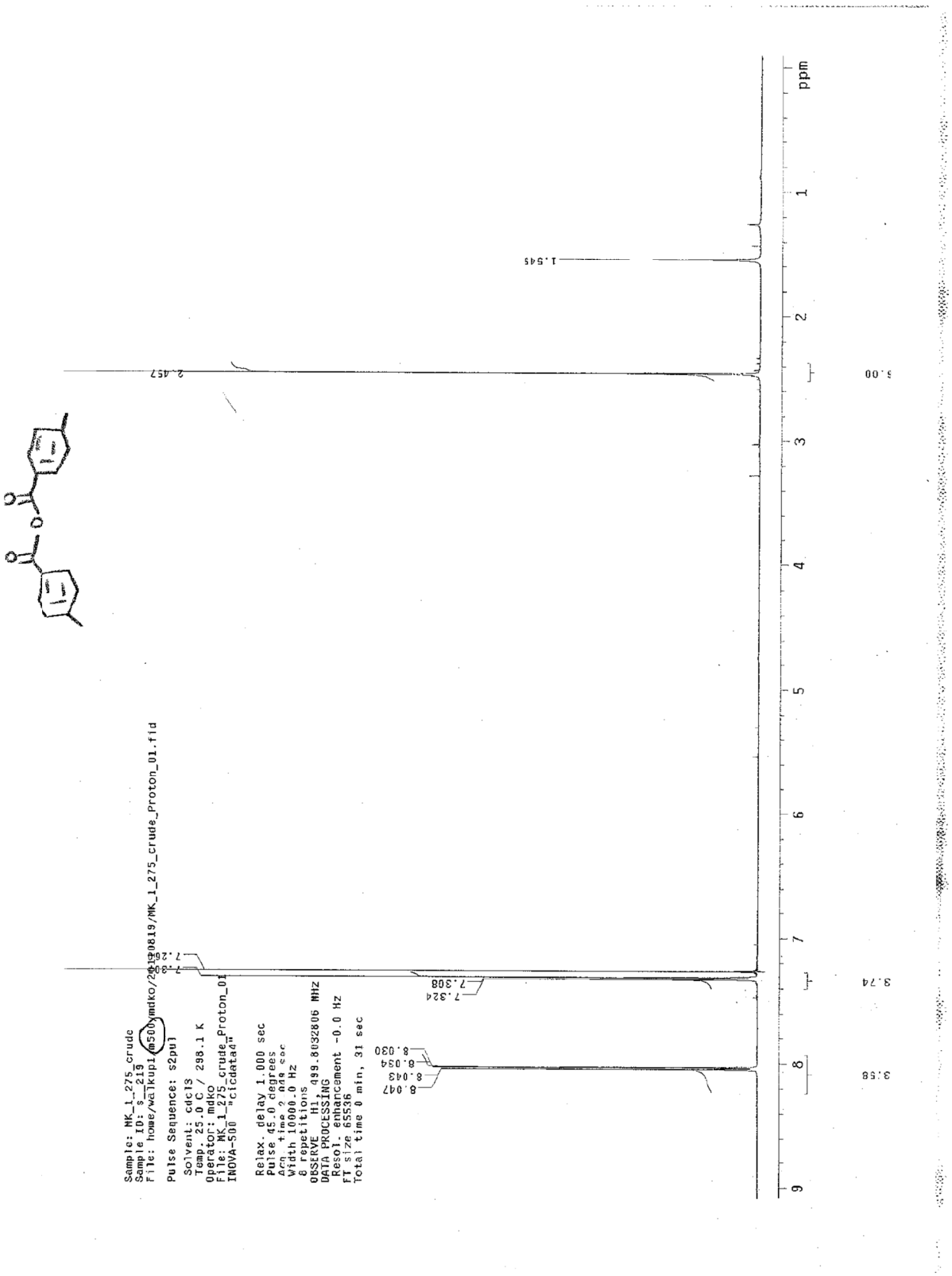
According to General Procedure, lauric acid (100.16 mg, 0.50 mmol), carbon tetrabromide (166 mg, 0.50 mmol), 2,6-lutidine (117 μL, 1.0 mmol) and $Ru(bpy)_3Cl_2$ (3.74 mg, 5.0 μmol) in DMF (5.0 mL) afforded **14** (92.7 mg, 97%) as a light yellow solid.

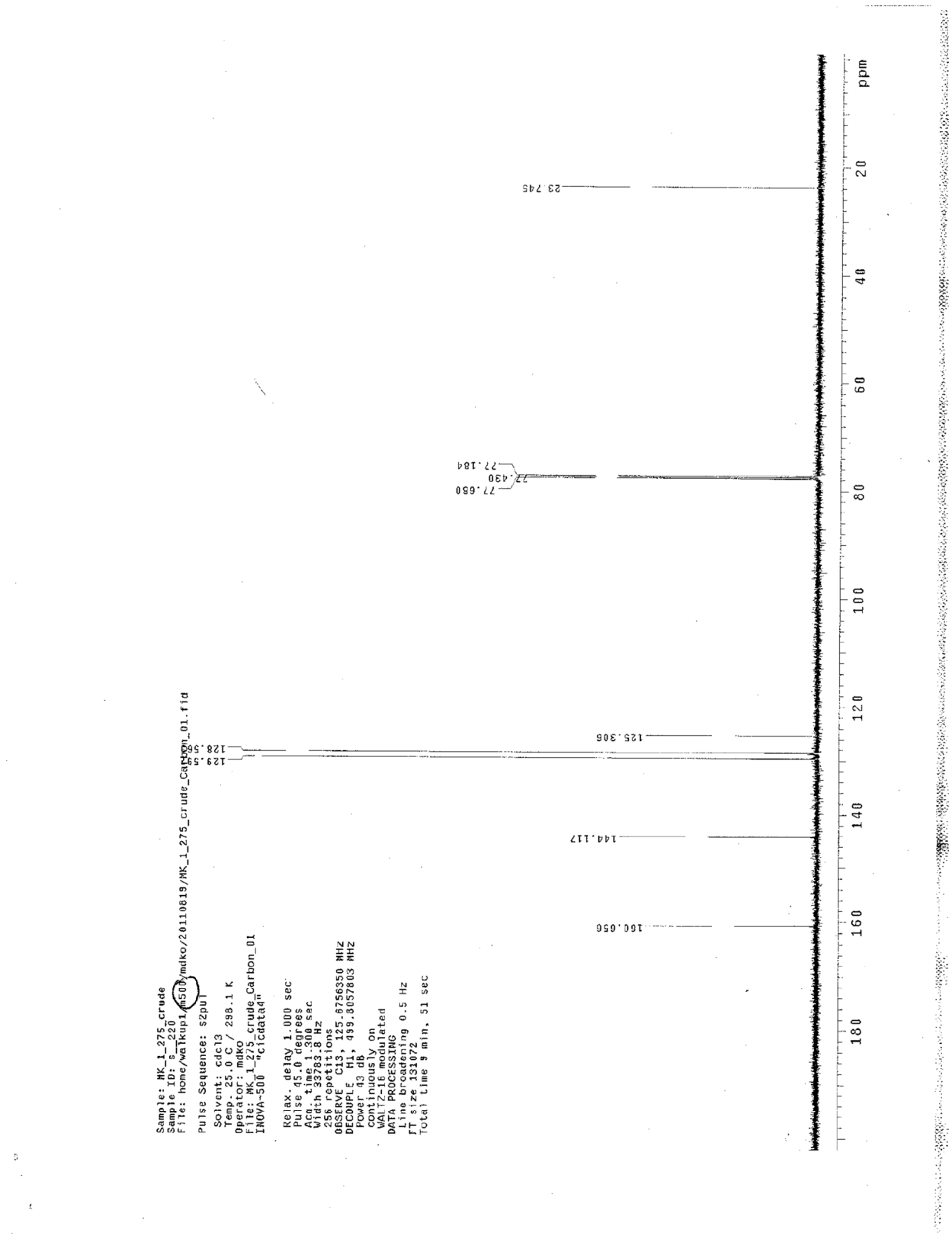
m.p.: 40-42 °C;
^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.43 (t, J = 7.4 Hz, 4 H), 1.68–1.62 (m, 4 H), 1.40–1.18 (m, 32 H), 0.87 (t, J = 6.4 Hz, 6 H);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 169.6, 35.3, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 28.9, 24.2, 22.7, 14.1;
IR (neat): ν_{max} 2924, 2854, 1818, 1746, 1466, 1035, 907, 731 cm^{-1};
MS (ESI) *m/z* calcd for $C_{24}H_{46}O_3Na$ ($[M + Na]^+$) 405.3345, found 405.3331.

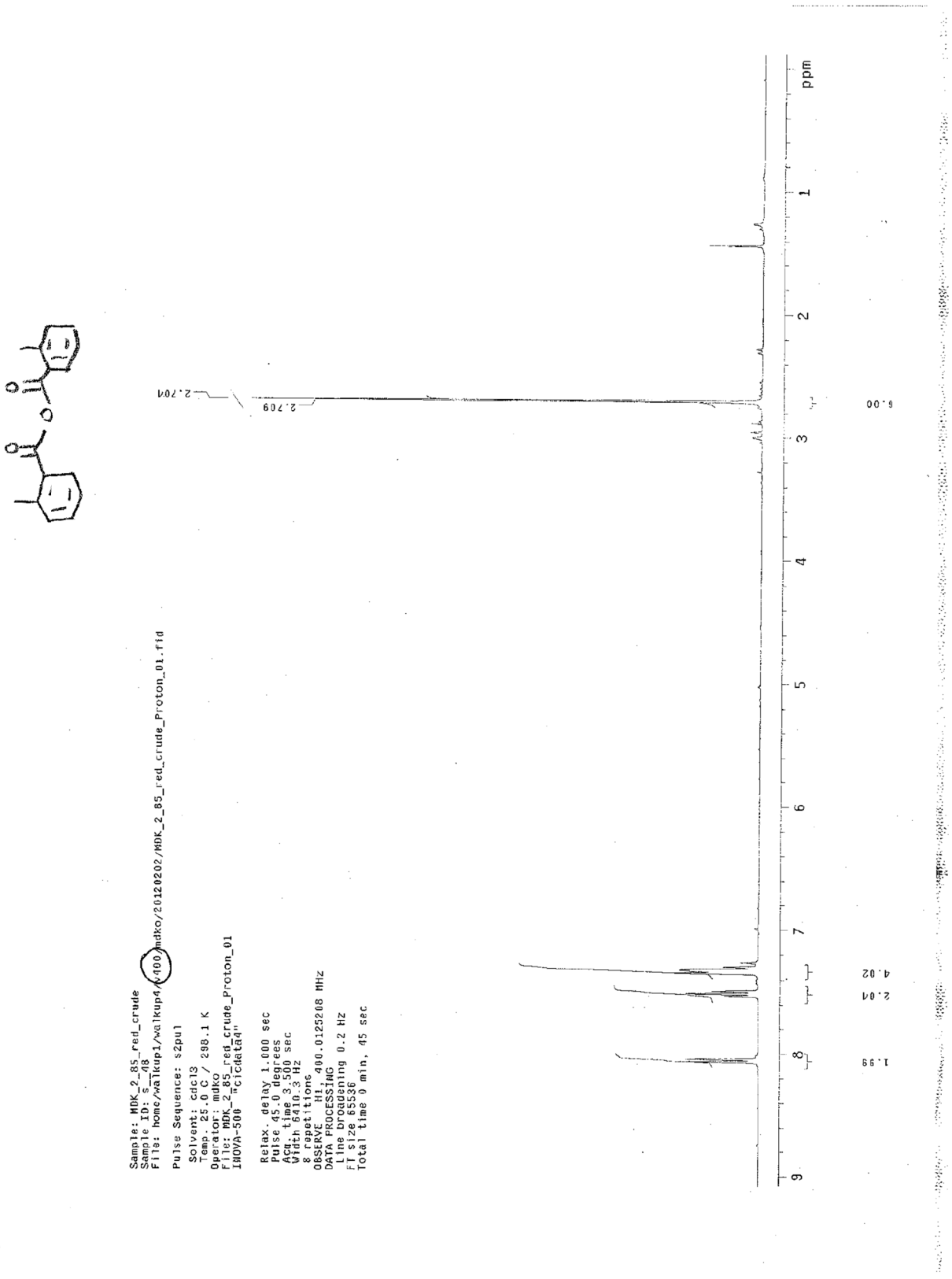
[10] M. V. Kvach, D. A. Tsybulsky, A. V. Ustinov, I. A. Stepanova, S. L. Bondarev, S. V. Gontarev, V. A. Korshun, V. V. Shmanai, *Biconjugate Chem.,* 2007, **18**, 1691–1696.

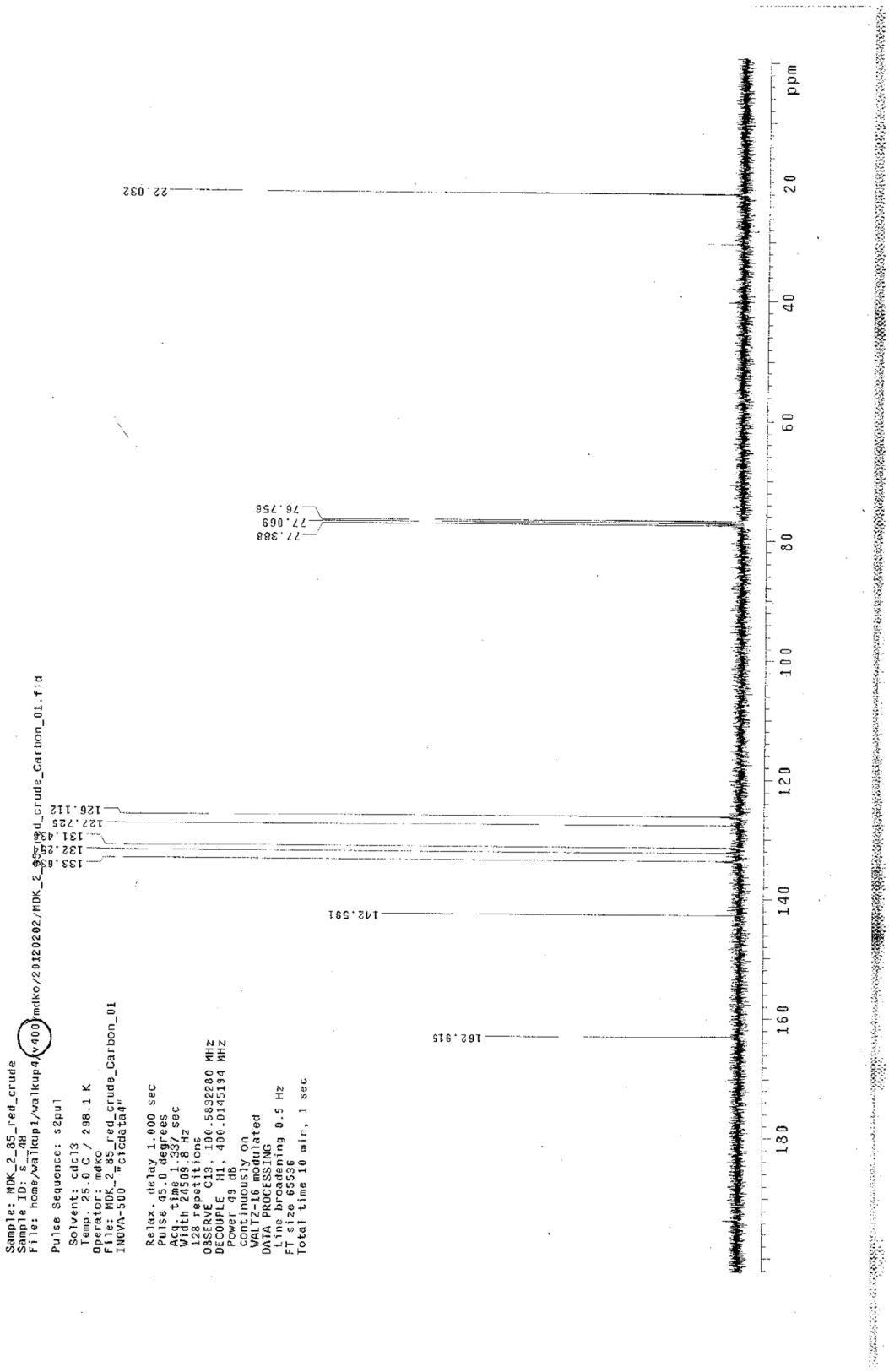

Sample: MK_1_270_crude
Sample ID: s__227
File: home/watkup1/m500/mdko/20110819/MK_1_270_crude_Proton_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C. / 298.1 K
Operator: mdko
File: MK_1_270_crude_Proton_01
INOVA-500 "ciclhata4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 10000.0 Hz
8 repetitions
OBSERVE H1, 499.8032808 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 31 sec
ppm
9
8
7
6
5
4
3
2
1
3.80
2.00
4.12

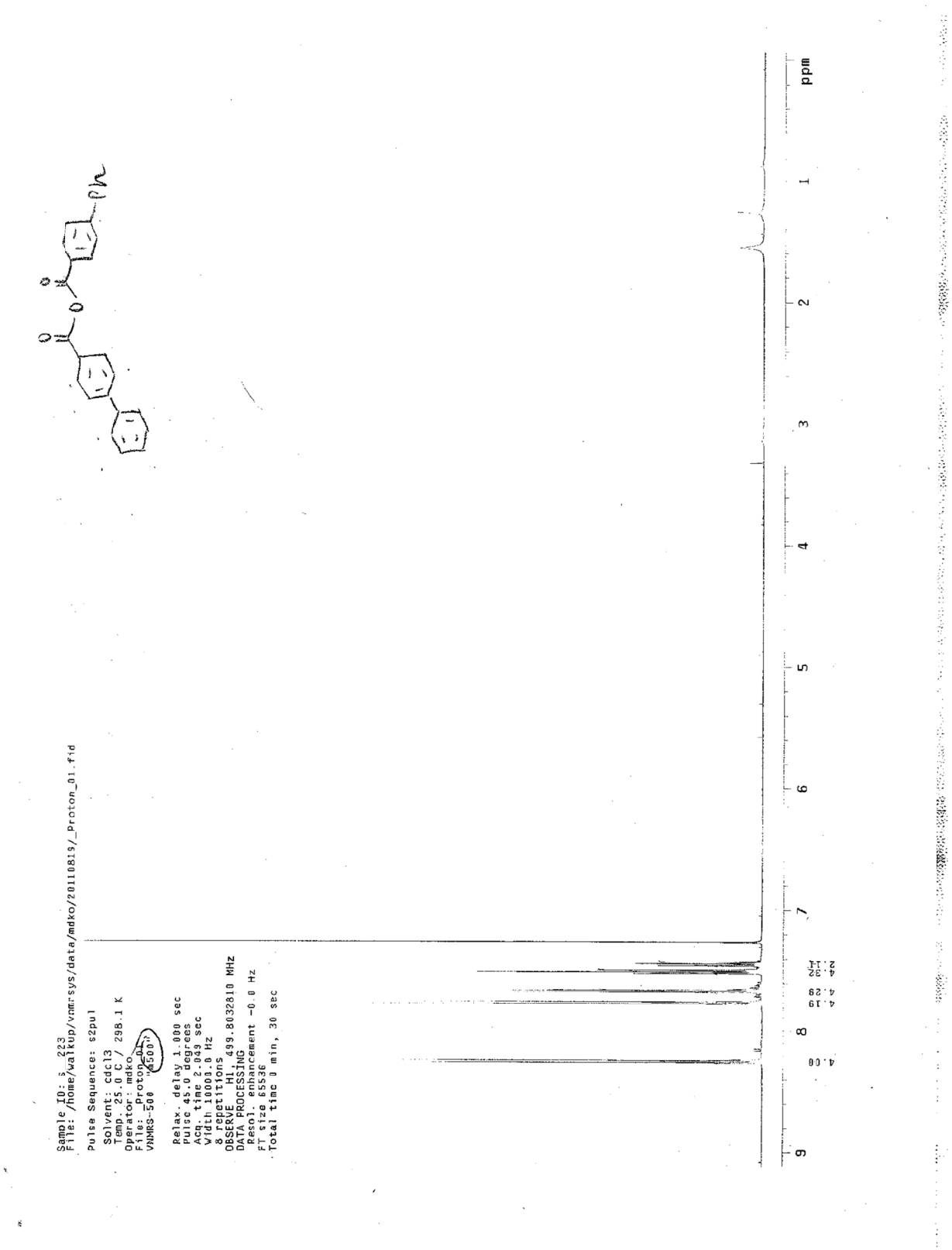

Sample: MK_1_270_crude
Sample ID: s__228
File: home/walkup1/ma500/mdko/20110819/MK_1_270_crude_Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mdko
File: MK_1_270_crude_Carbon_01
INOVA-500 "cicdata4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 33783.8 Hz
256 repetitions
OBSERVE C13, 125.6756350 MHz
DECOUPLE H1, 499.8057803 MHz
Power 43 dB
Continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 9 min, 51 sec
160.471
133.407
129.538
127.906
127.874
77.704
77.458
77.212
ppm

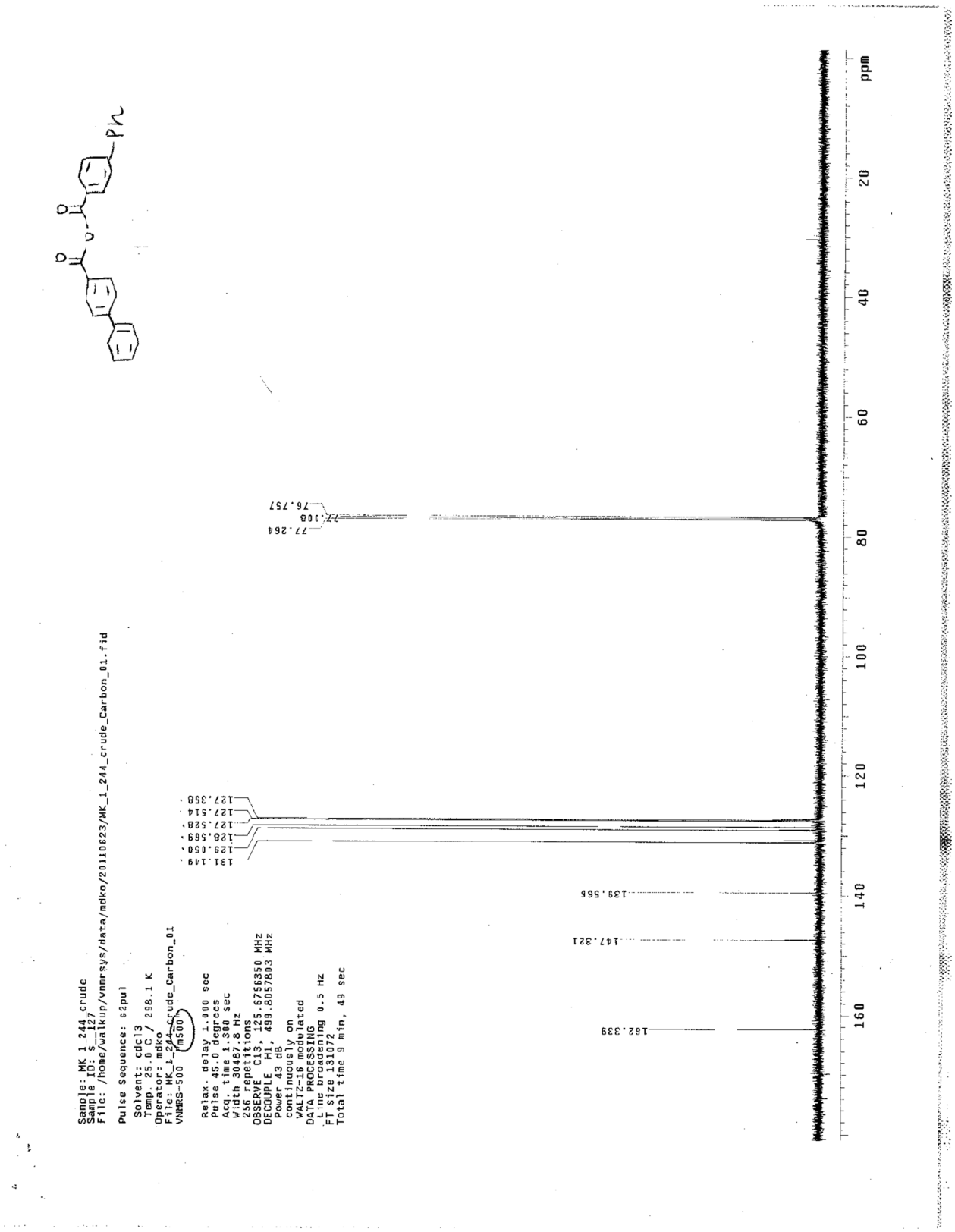


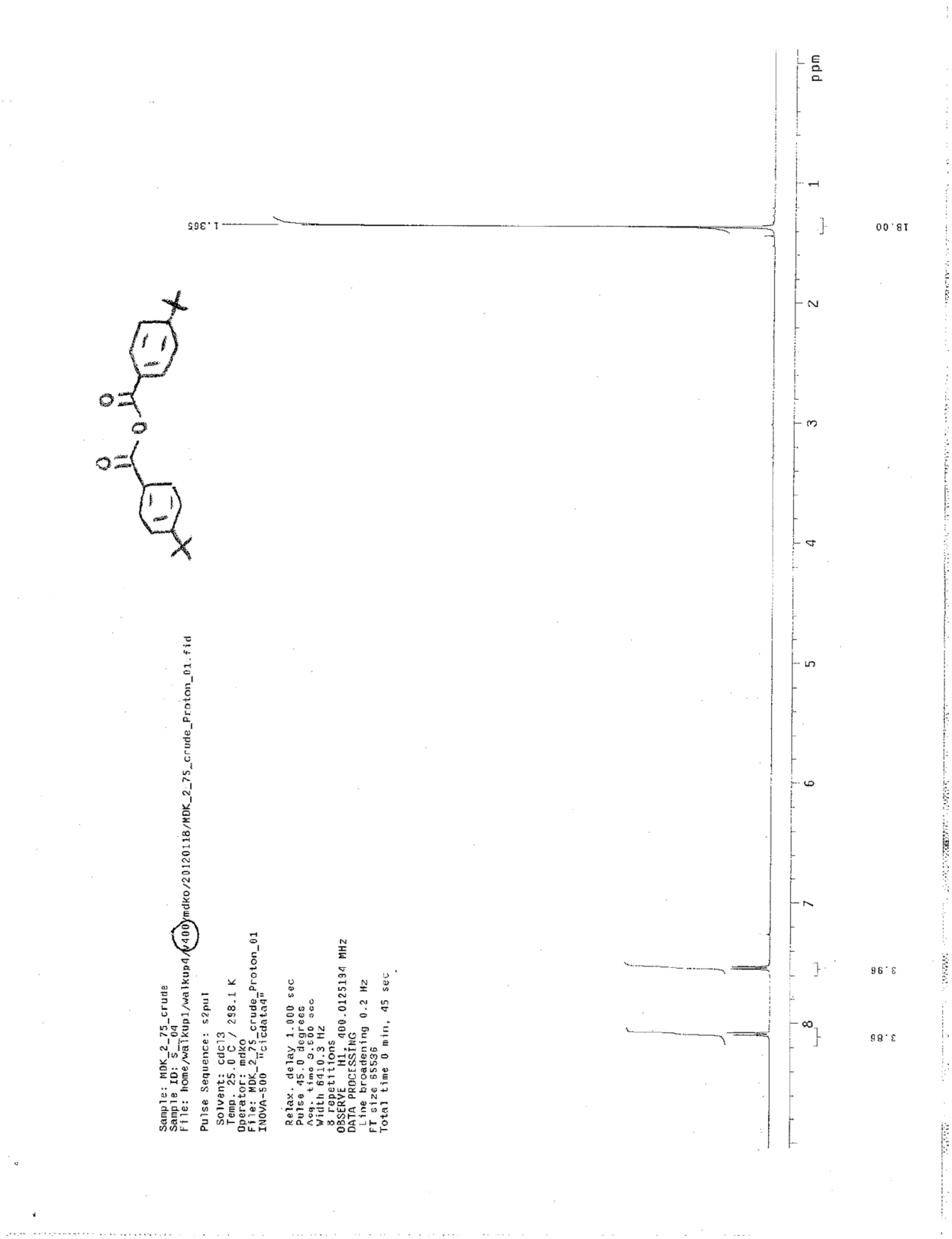


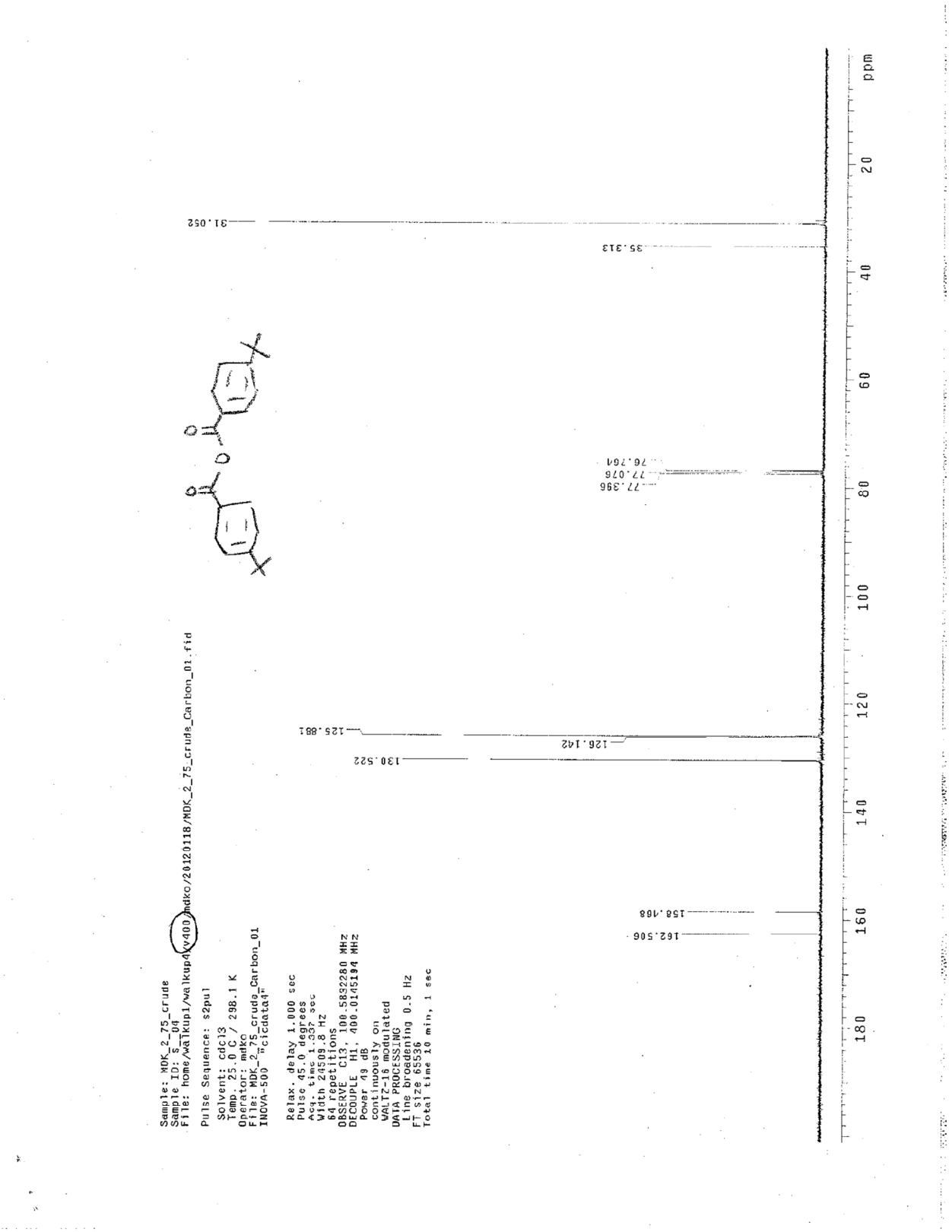

Sample: MDK_2_85_red_crude
Sample ID: s__48
File: home/walkup1/walkup4/400_mdko/20120202/MDK_2_85_red_crude_Proton_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mdko
File: MDK_2_85_red_crude_Proton_01
INOVA-500 "c1cdata4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1 499.8125208 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 45 sec
2.704
2.709
6.00
4.02
2.04
1.99
ppm

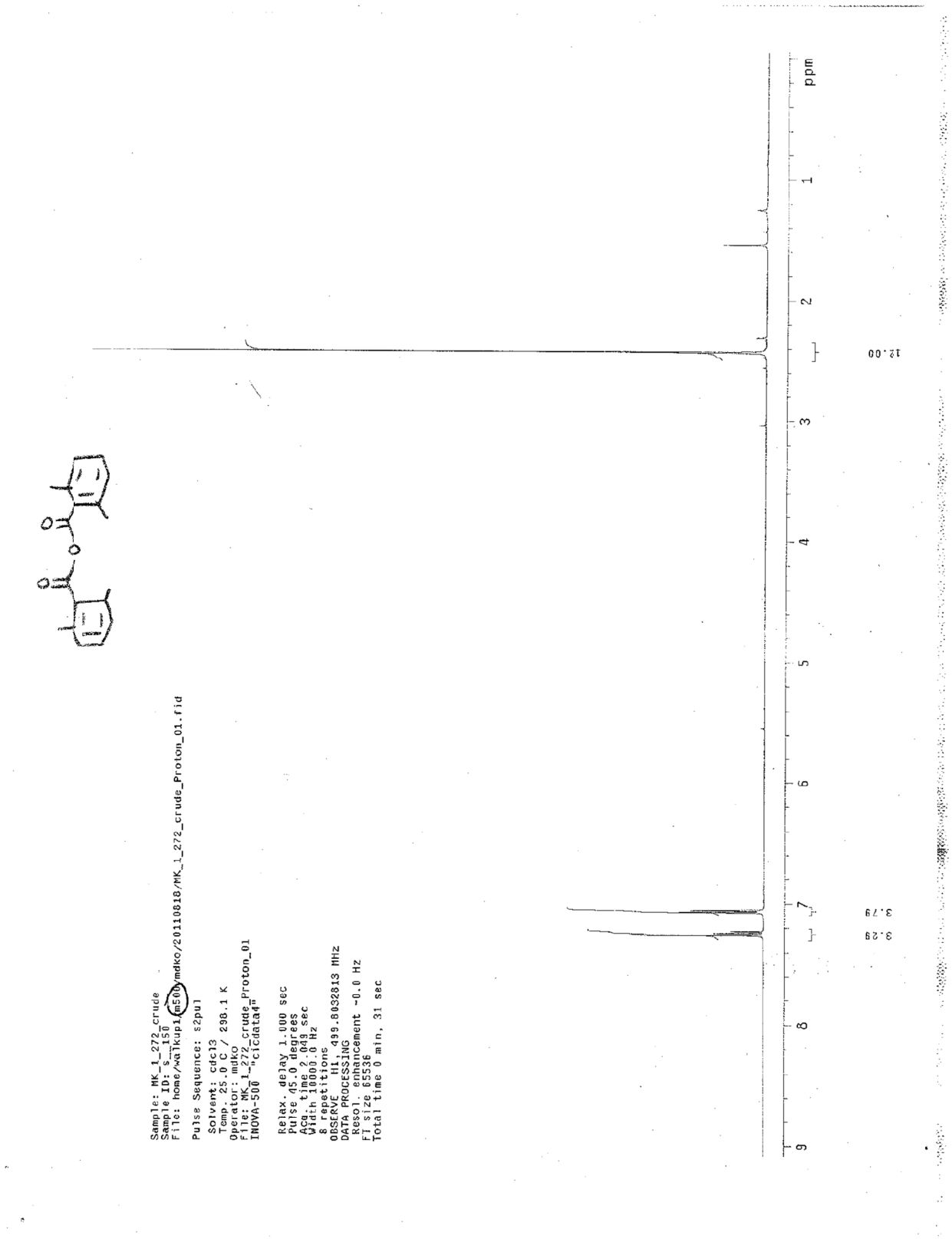

Sample: MDK_2_85_red_crude
Sample ID: s__48
File: home/walkup1/walkup4/400-mdko/20120202/MDK_2_85_red_crude_Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mdko
File: MDK_2_85_red_crude_Carbon_01
INOVA-500 "c1cdata4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.837 sec
Width 24038.8 Hz
128 repetitions
OBSERVE C13, 100.5832280 MHz
DECOUPLE H1, 400.0145194 MHz
Power 49 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 0.5 Hz
FT size 65536
Total time 10 min, 1 sec
162.915
142.591
133.659
132.252
131.434
127.725
126.112
77.388
77.069
76.756
22.032
ppm

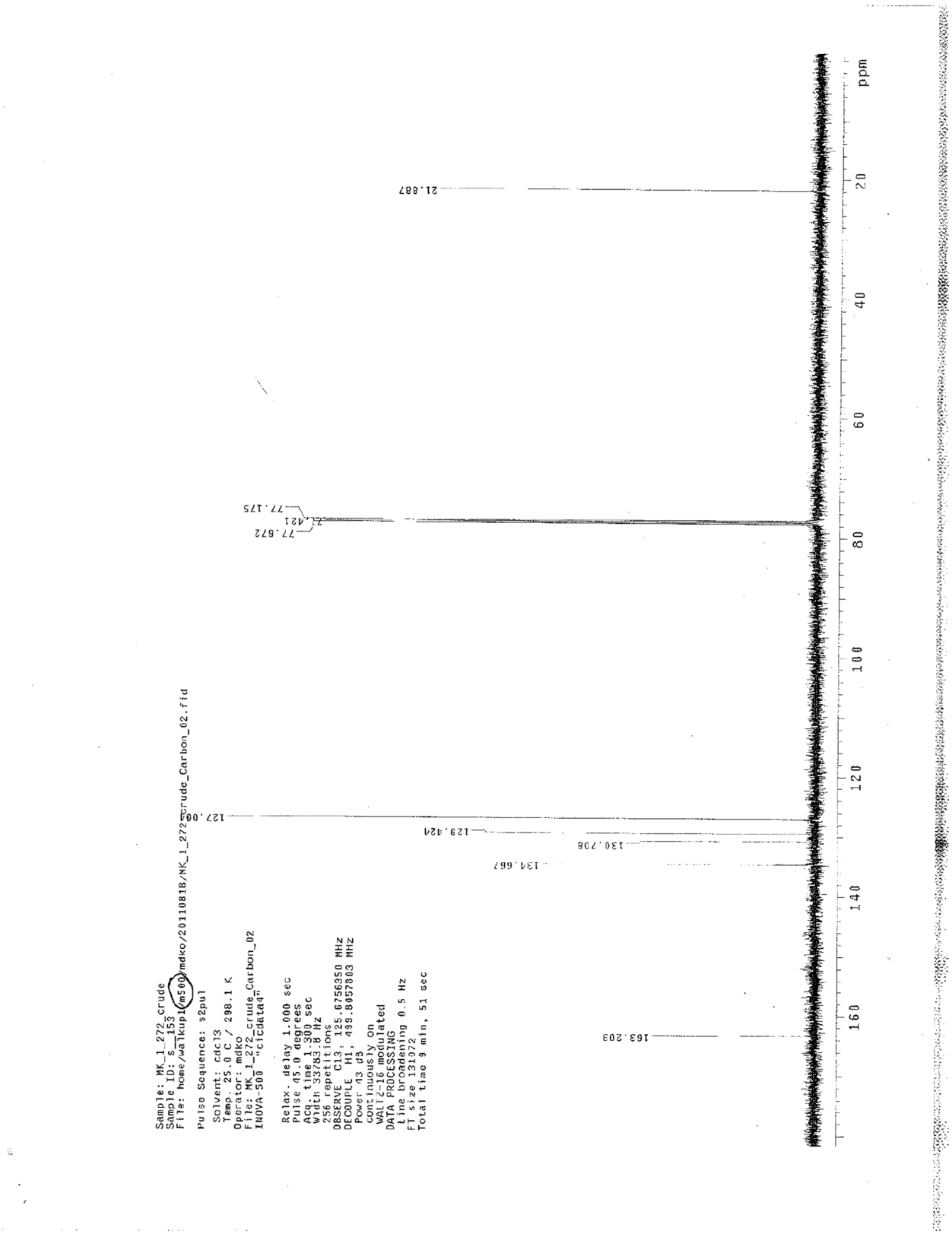


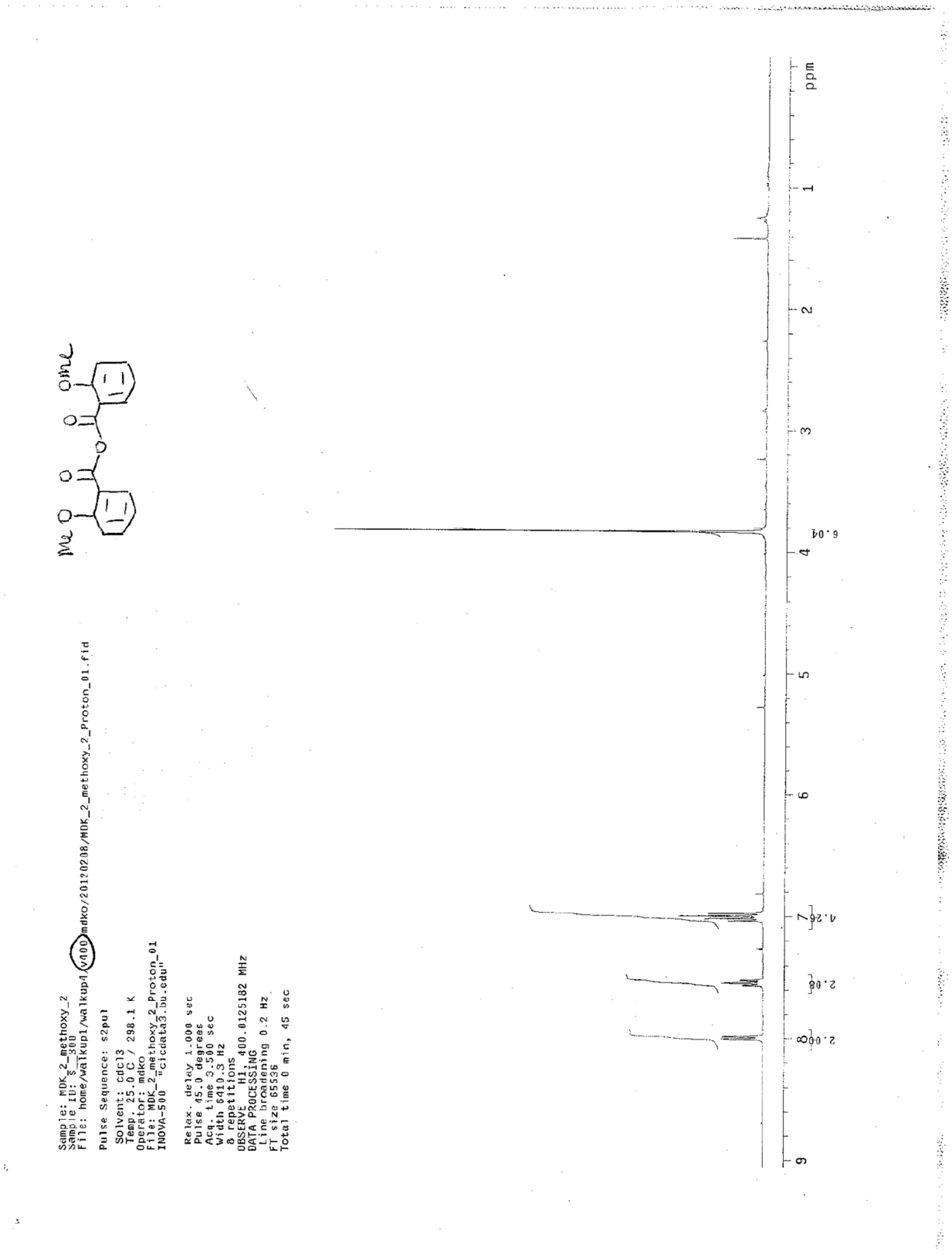


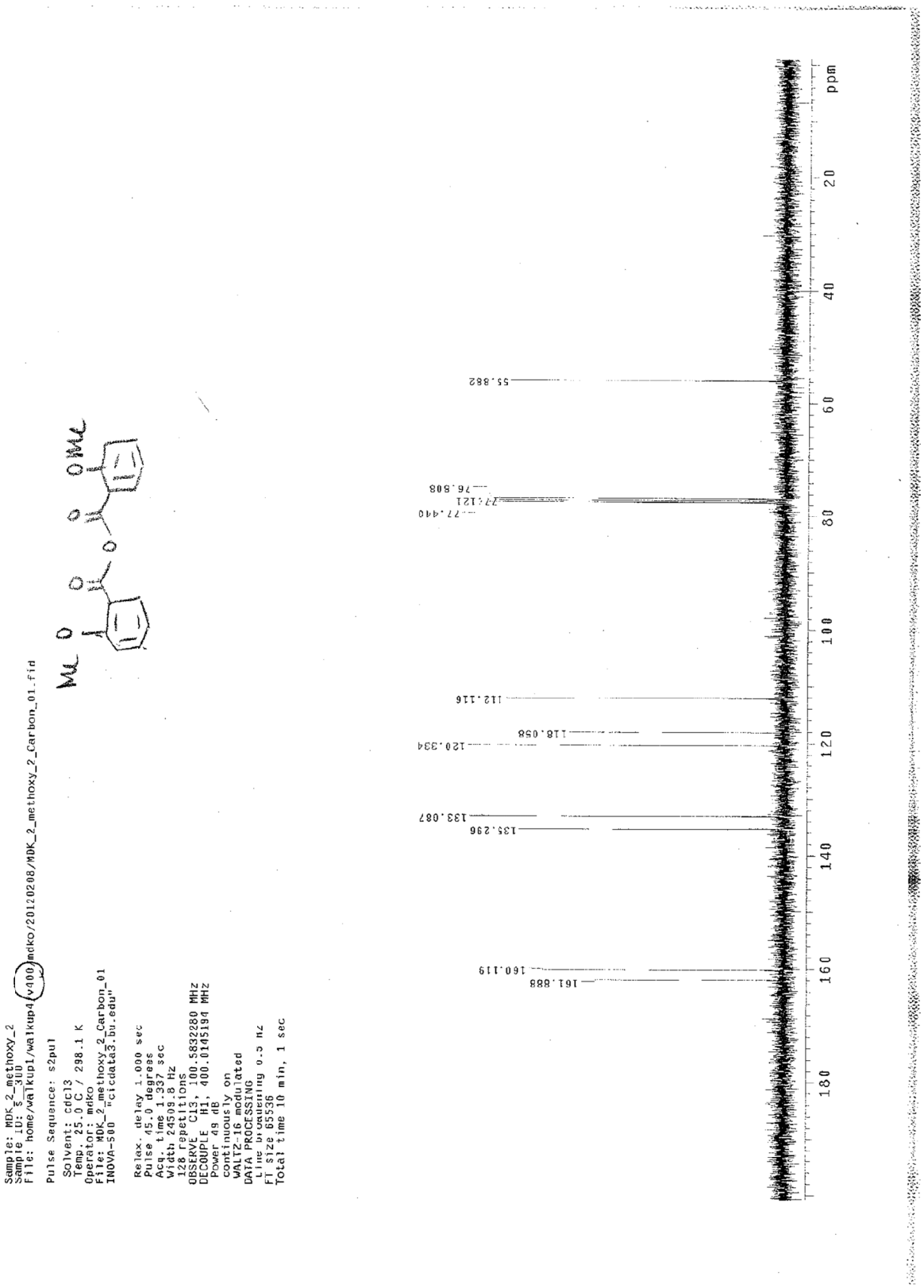


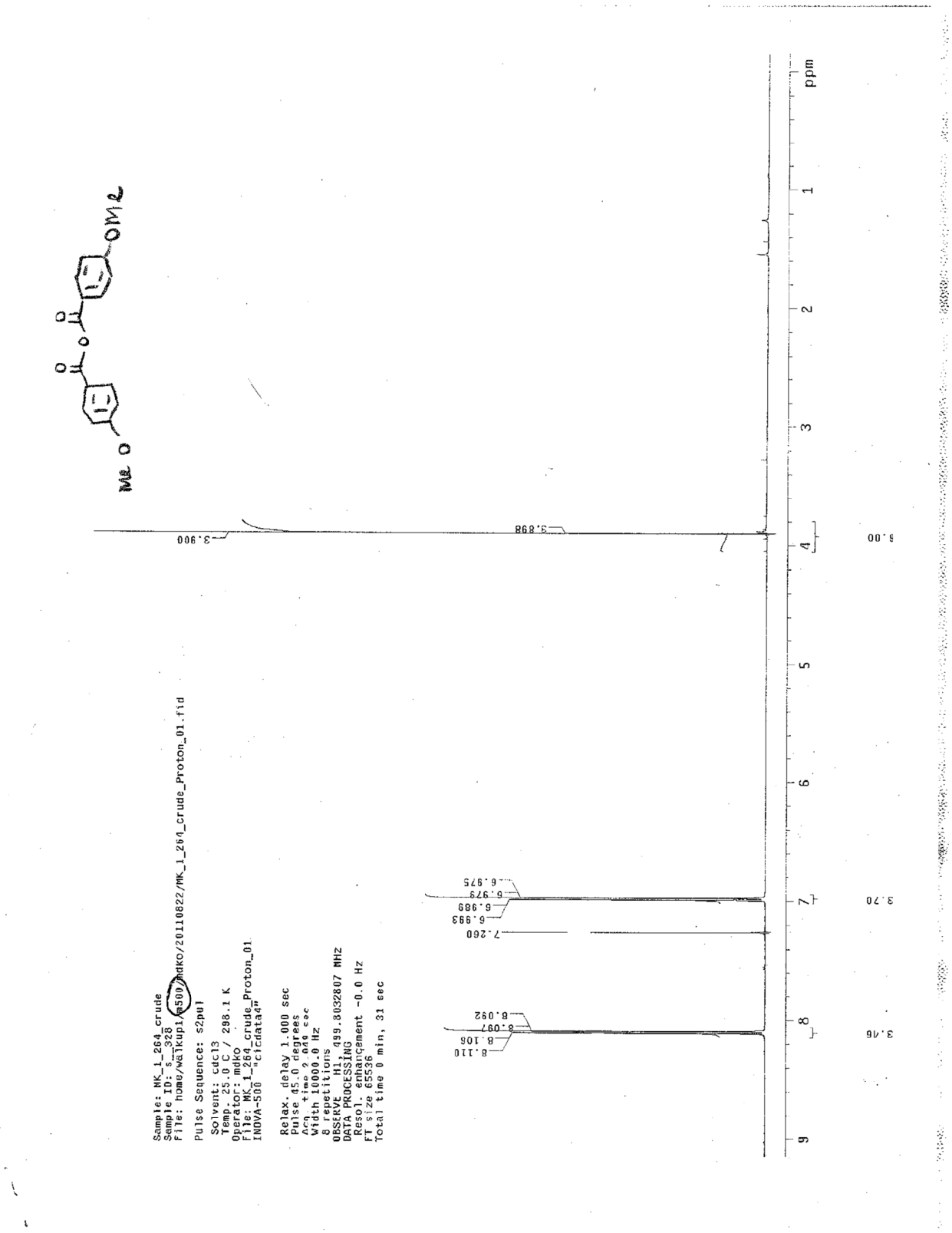


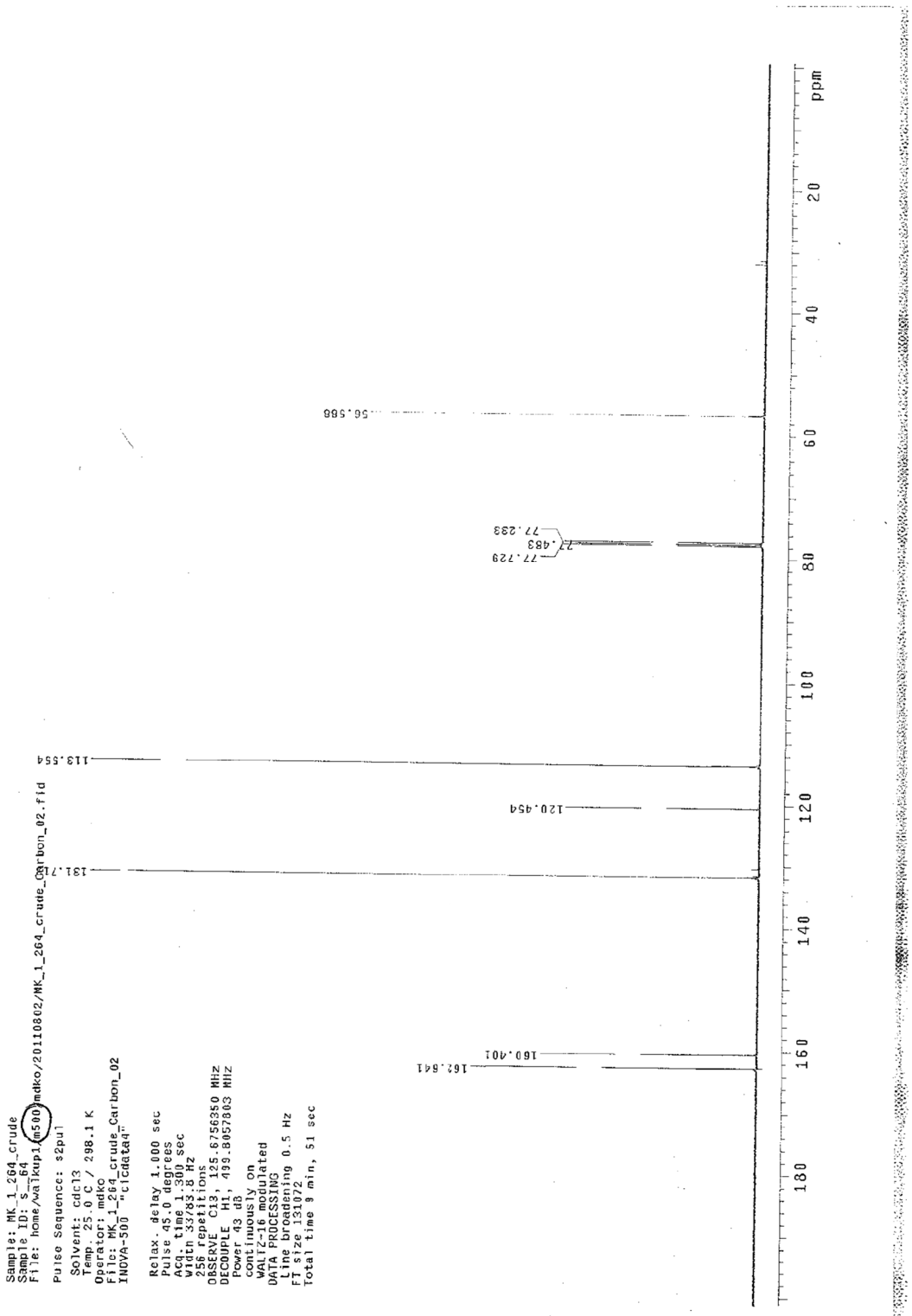

Sample: MK_1_272_crude
Sample ID: s__150
File: home/walkup1/m500/mdko/20110818/MK_1_272_crude_Proton_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mdko
File: MK_1_272_crude_Proton_01
INOVA-500 "cicdata4m"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 10000.0 Hz
8 repetitions
OBSERVE H1, 499.8032813 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 31 sec
12.00
3.79
3.29
ppm

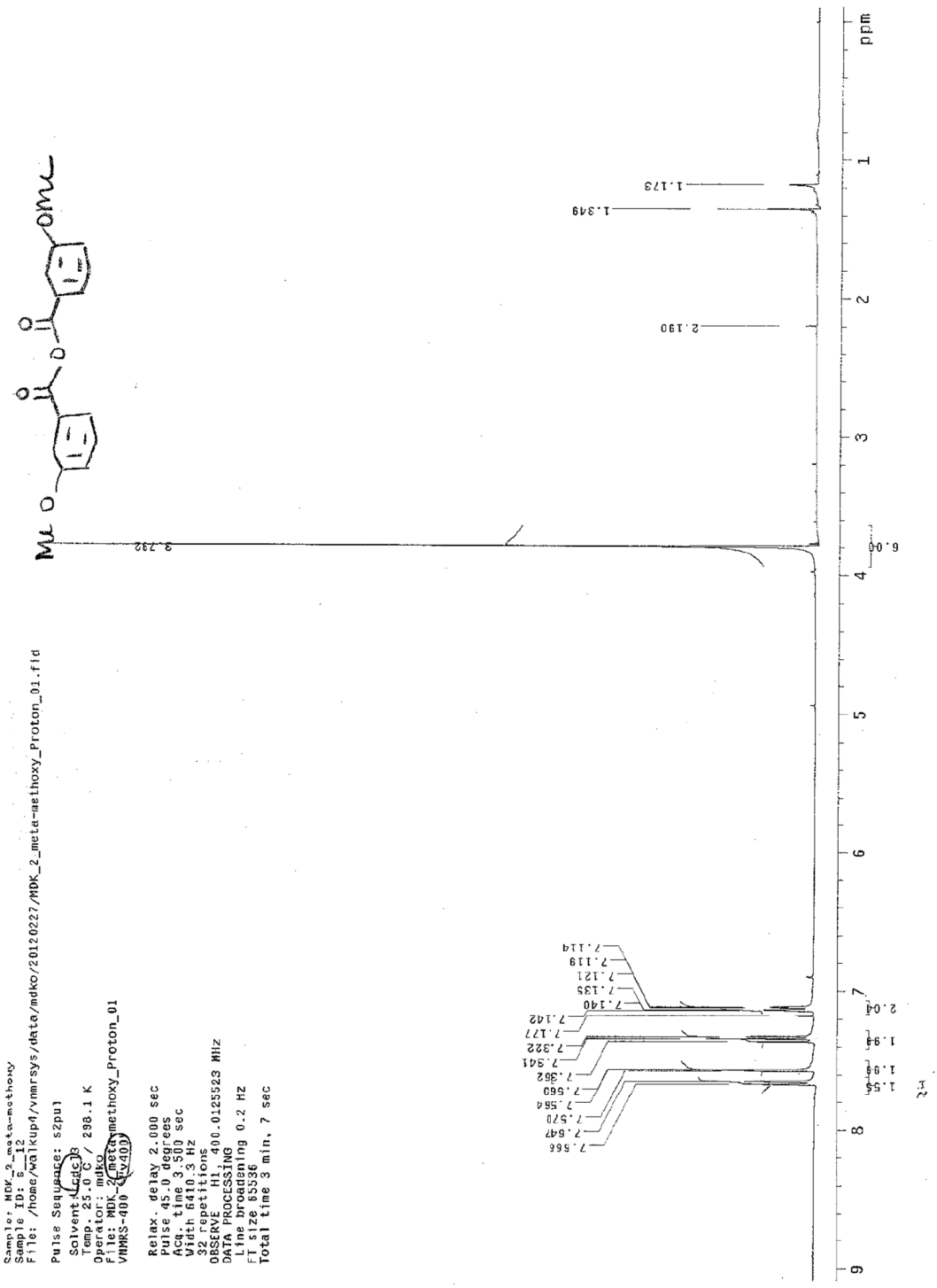

Sample: MK_1_272_crude
Sample ID: s__153
File: home/walkup1/m500/mdko/20110818/MK_1_272_crude_Carbon_02.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mdko
File: MK_1_272_crude_Carbon_02
INOVA-500 "c1cdata4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
ACQ time 1.300 sec
Width 33783.8 Hz
256 repetitions
OBSERVE C13, 125.6756350 MHz
DECOUPLE H1, 499.8057803 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 9 min, 51 sec
127.00
129.424
130.708
134.667
163.203
77.672
77.421
77.175
21.887
ppm

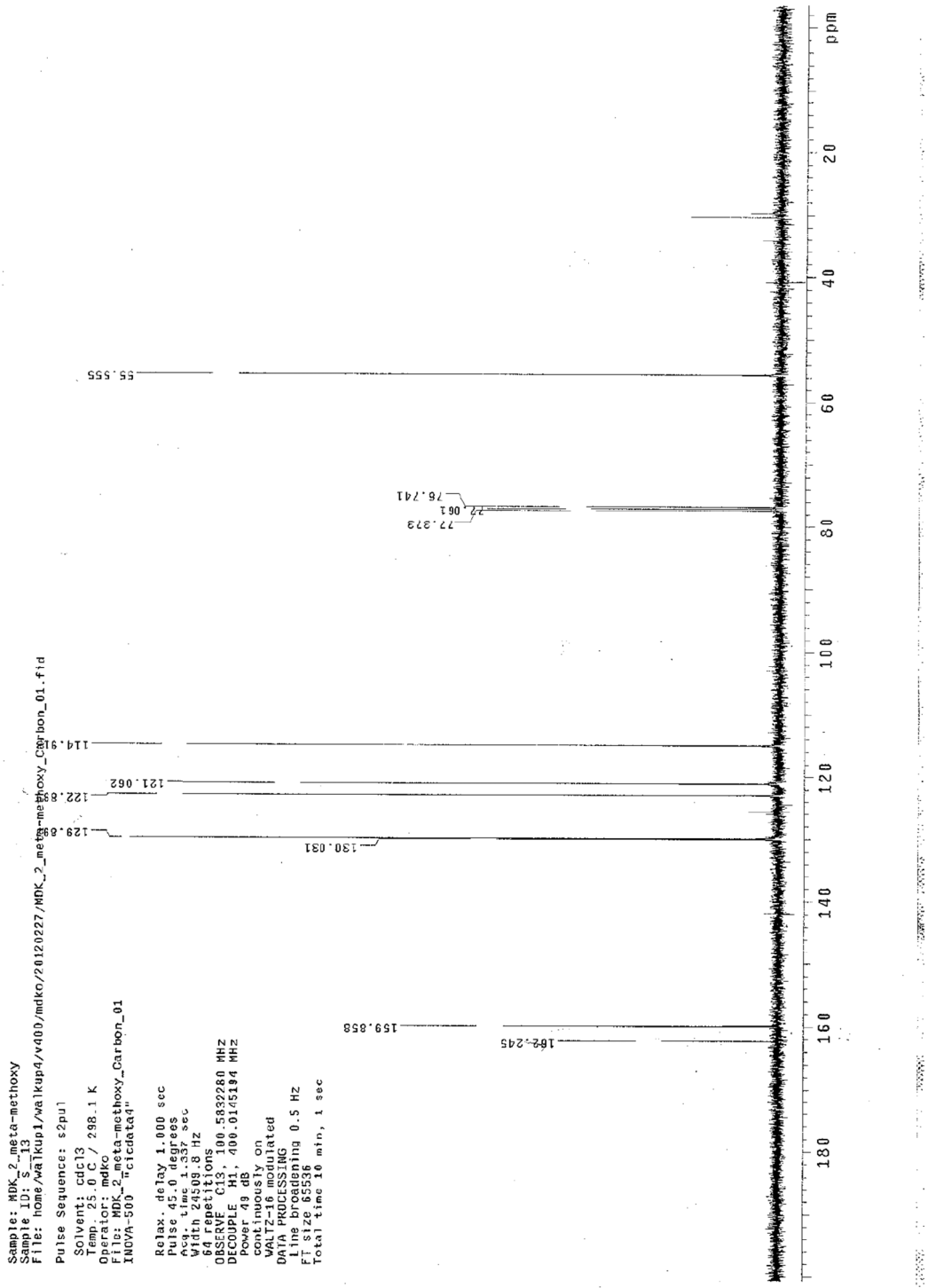


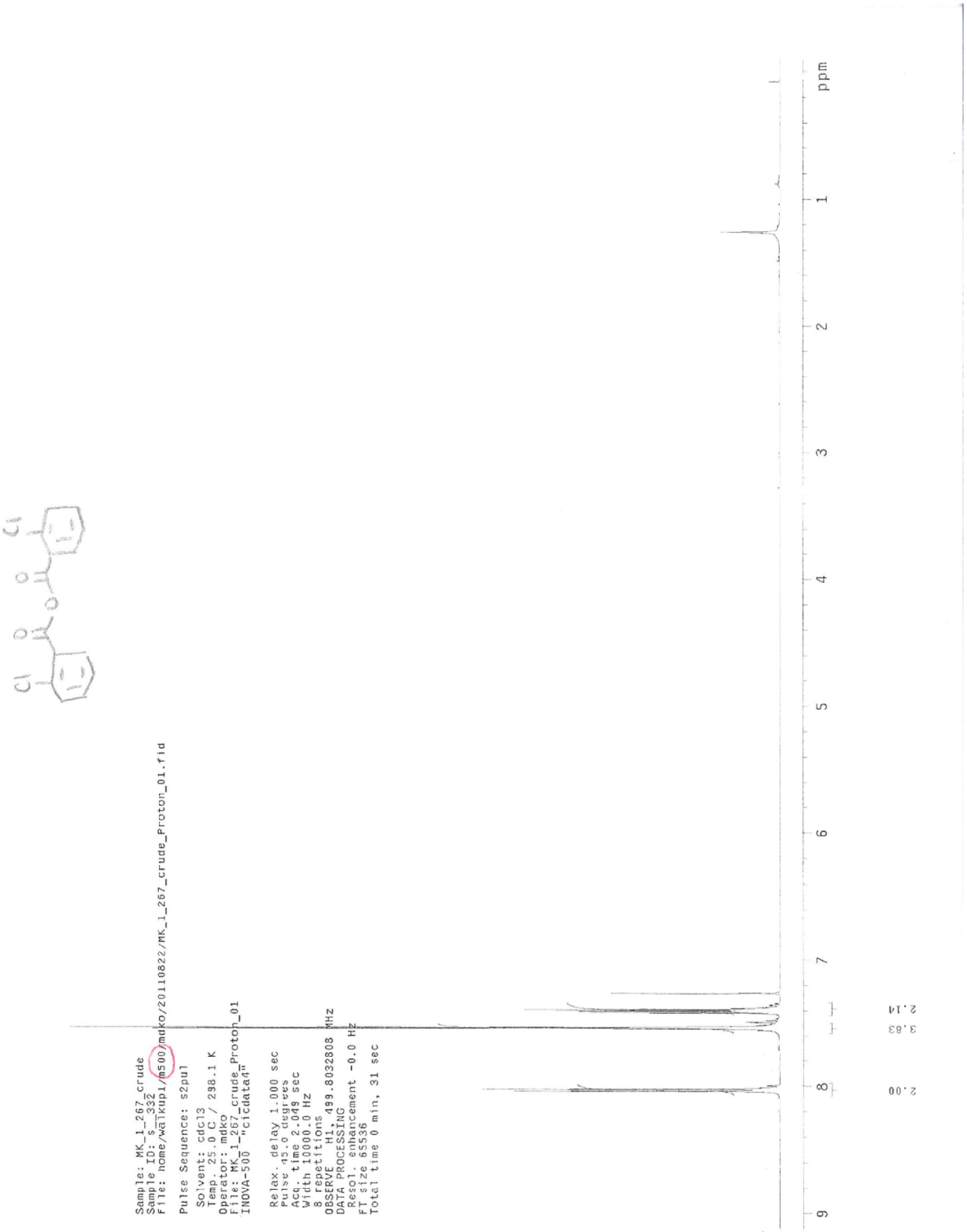

Sample: MDK_2_methoxy_2
Sample ID: S_300
File: home/walkup1/walkup4(v100)mdko/20120208/MDK_2_methoxy_2_Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mdko
File: MDK_2_methoxy_2_Carbon_01
INOVA-500 "cicdata3.bu.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.337 sec
Width 24509.8 Hz
128 repetitions
OBSERVE C13, 100.5832280 MHz
DECOUPLE H1, 400.0145194 MHz
Power 49 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 10 min, 1 sec
Me
O
O
O
O
Me
55.882
76.808
77.121
77.440
112.116
118.058
120.334
133.087
135.296
160.119
161.888
ppm
180
160
140
120
100
80
60
40
20

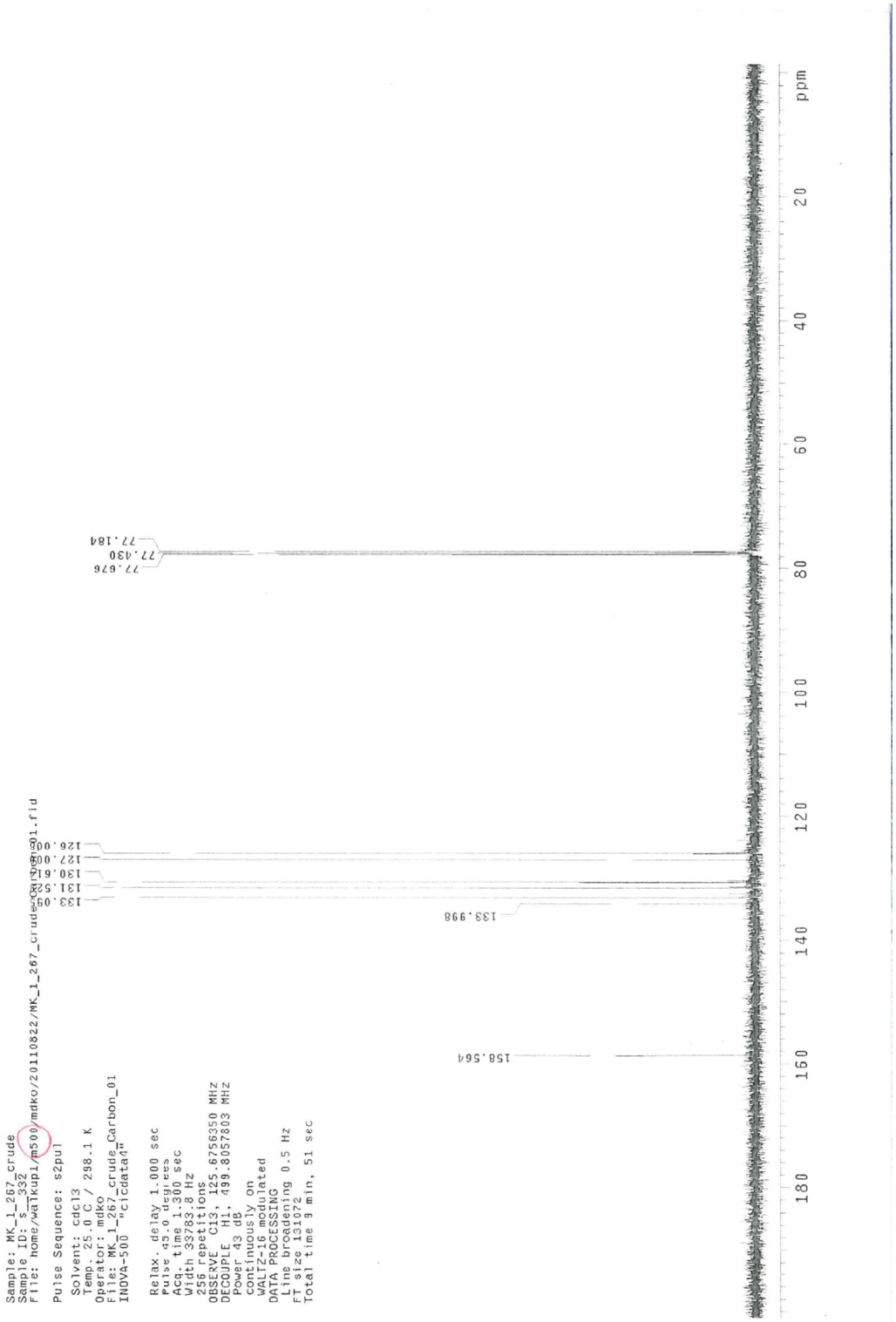


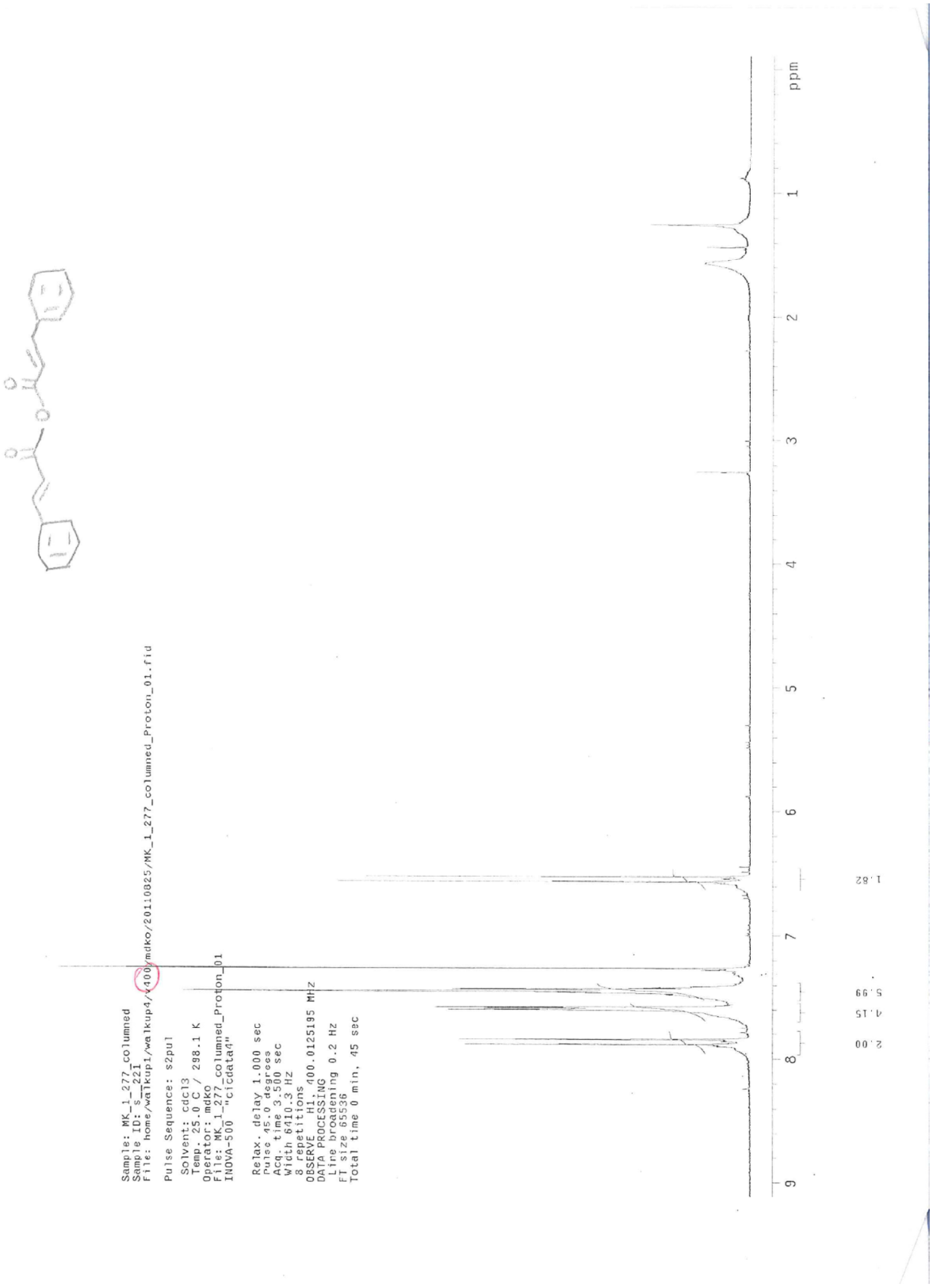

Sample: MK_1_264_crude
Sample ID: s__64
File: home/walkup1/m500/mdko/20110802/MK_1_264_crude_Carbon_02.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mdko
File: MK_1_264_crude_Carbon_02
INOVA-500 "cicdata4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 33783.8 Hz
256 repetitions
OBSERVE C13, 125.6756350 MHz
DECOUPLE H1, 499.8057803 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 9 min, 51 sec
181.717
162.641
160.401
120.454
77.729
77.483
77.238
56.586
ppm

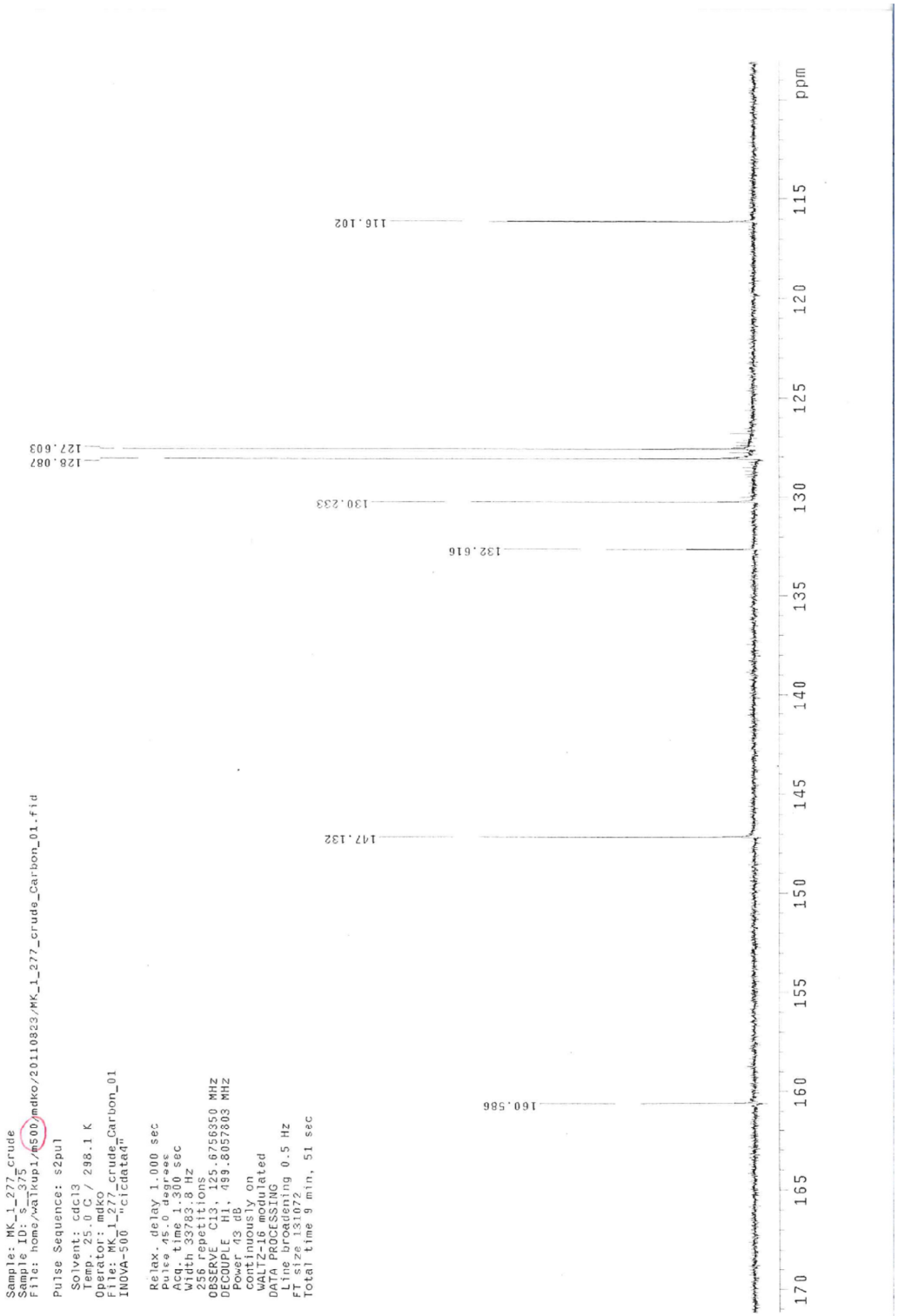


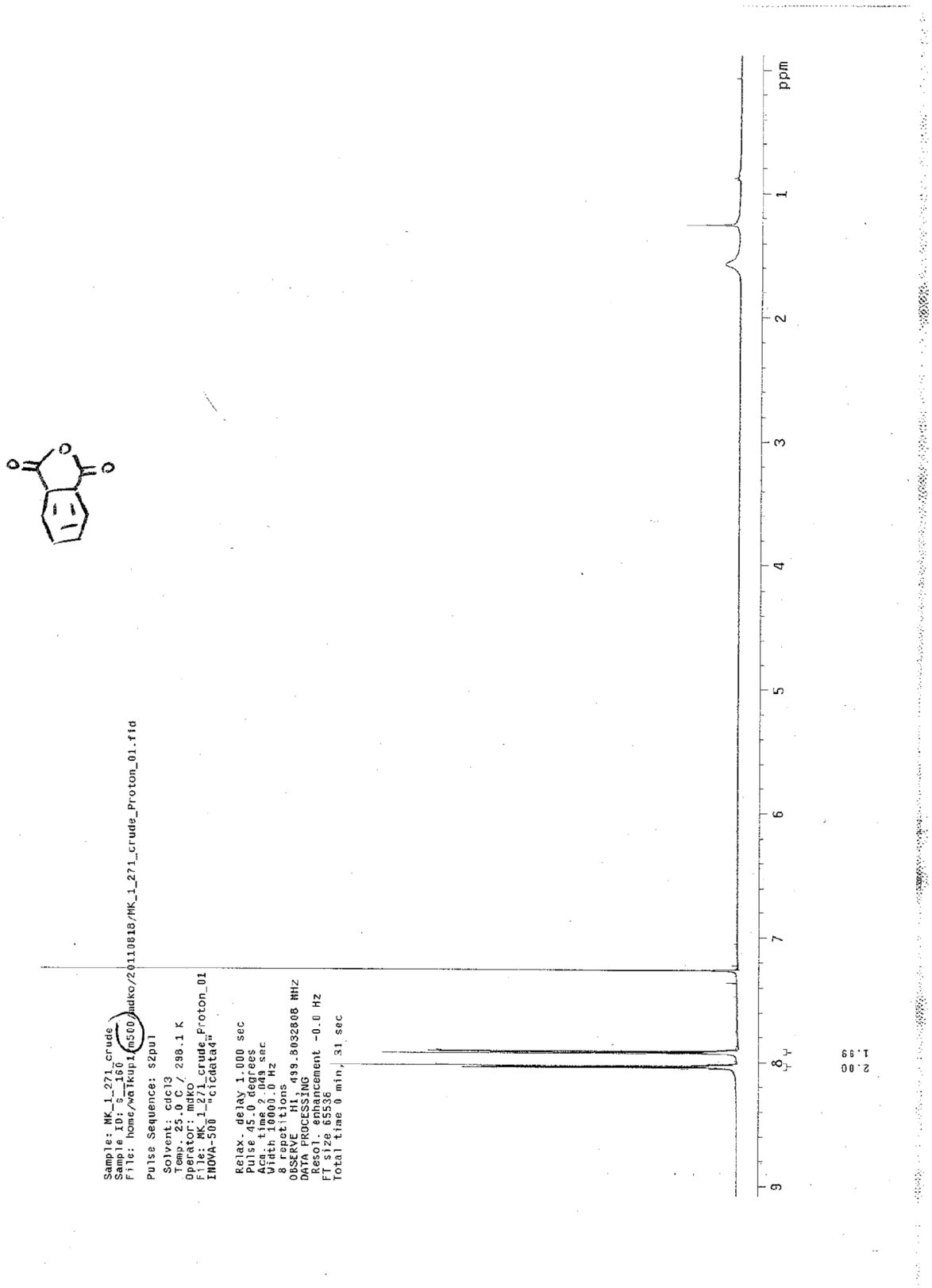


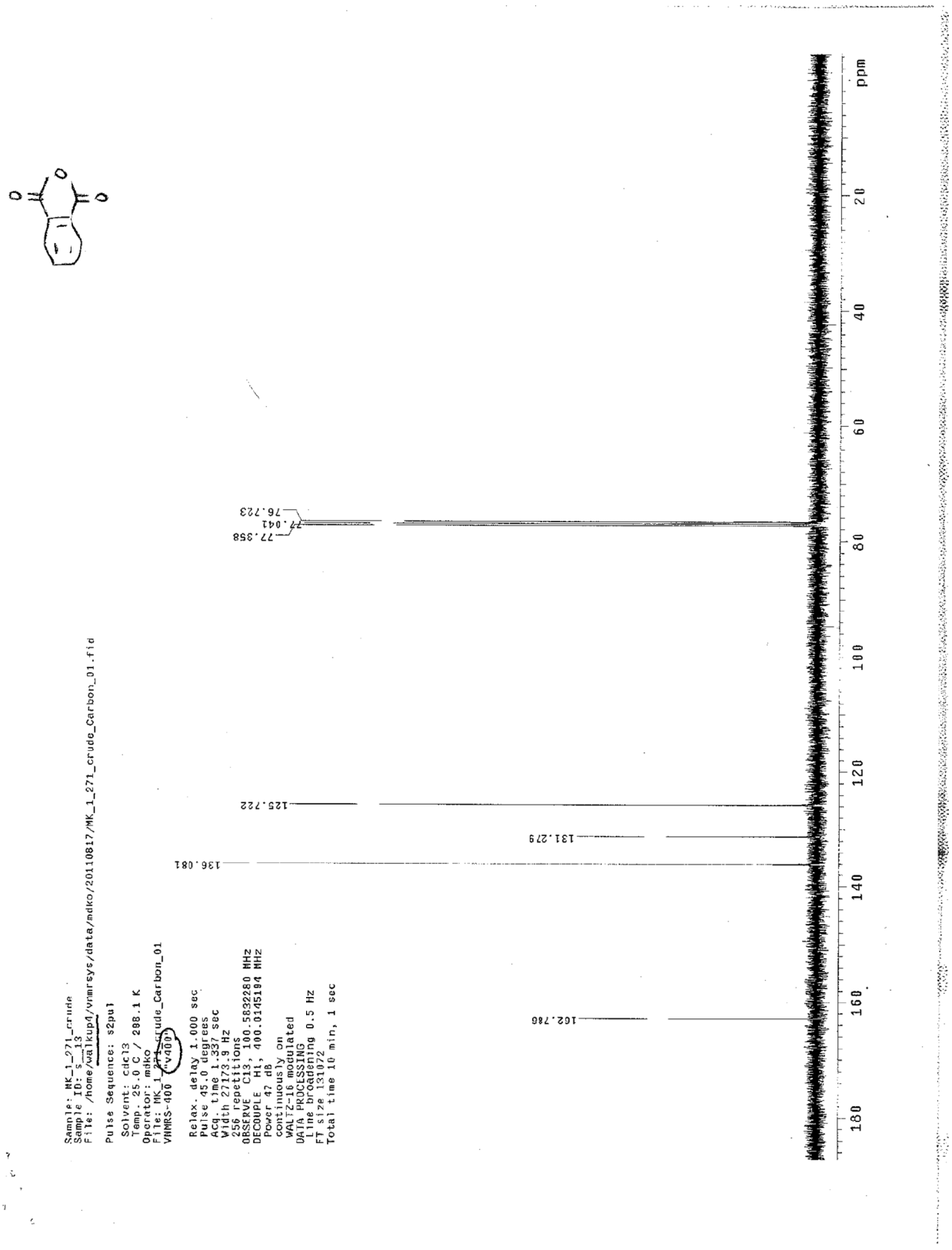

Sample: MK_1_267_crude
Sample ID: s_332
File: home/walkup1/m500/mdko/20110822/MK_1_267_crude_Proton_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mdko
File: MK_1_267_crude_Proton_01
INOVA-500 "cicdata4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 10000.0 Hz
8 repetitions
OBSERVE H1, 499.8032808 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 31 sec
ppm
2.00
3.83
2.14

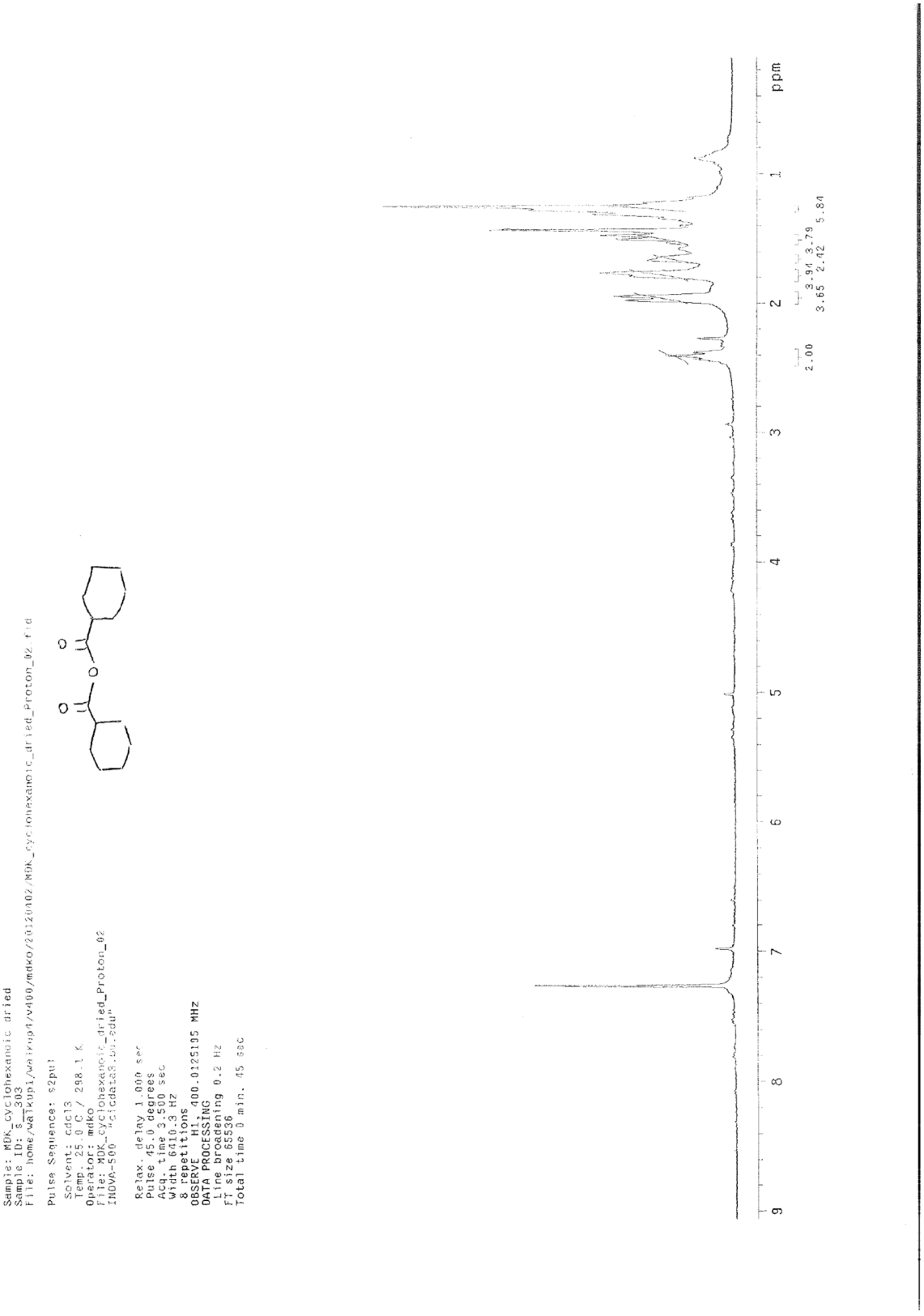

Sample: MK_1_267_crude
Sample ID:-s_332
File: home/waTkupi/m500/mdko/20110822/MK_1_267_crude_Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mdko
File: MK_1_267_crude_Carbon_01
INOVA-500 "c1cdata4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 33783.8 Hz
256 repetitions
OBSERVE C13, 125.6756350 MHz
DECOUPLE H1, 499.8057803 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 9 min, 51 sec
133.998
158.564
77.676
77.430
77.184
ppm

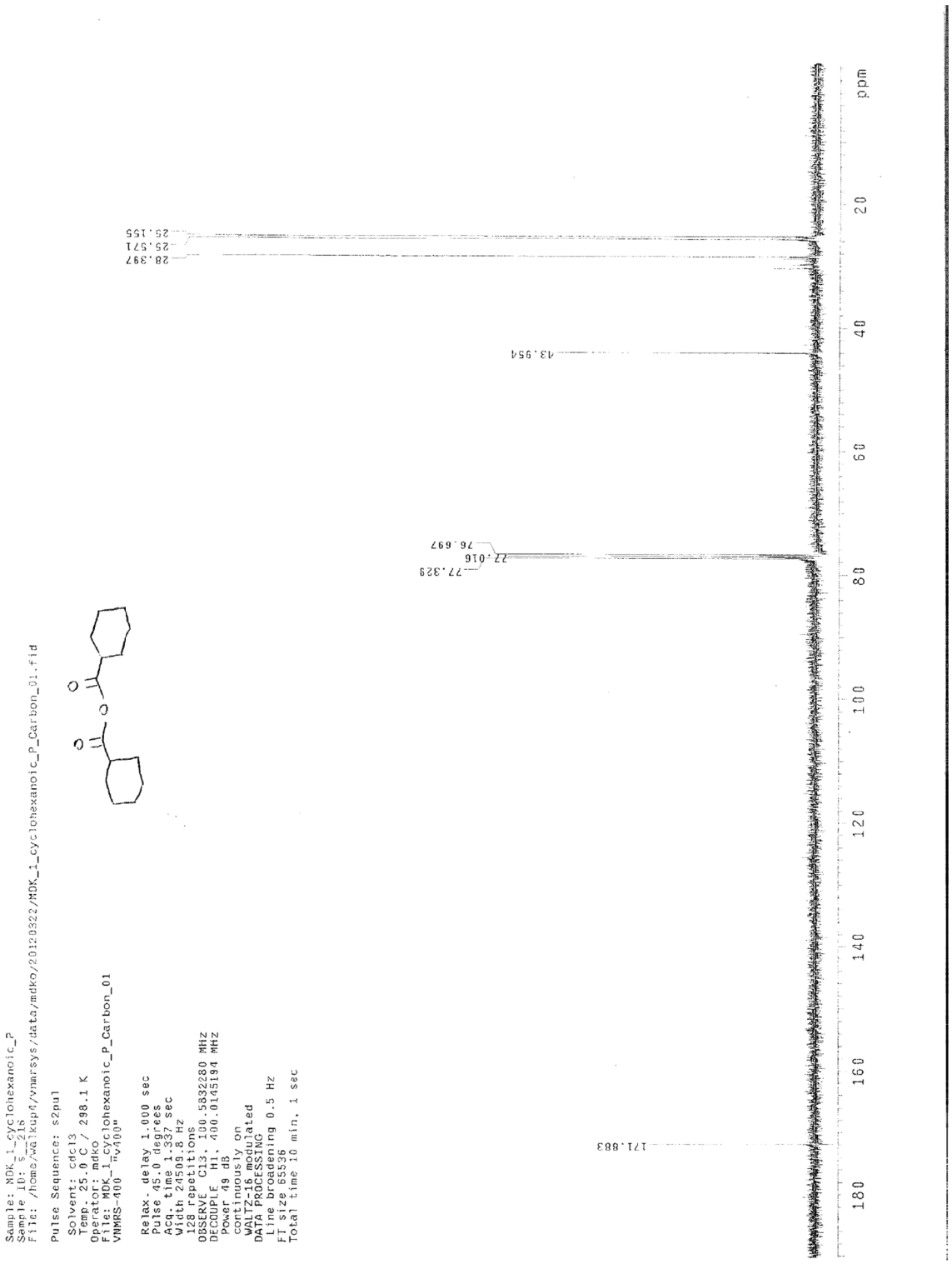

Sample: MK_1_277_columned
Sample ID:-s_221
File: home/walkup1/walkup4/x400/mdko/2011/0825/MK_1_277_columned_Proton_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mdko
File: MK_1_277_columned_Proton_01
INOVA-500 "cidata4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.0125195 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 45 sec
ppm
1.82
2.00
4.15
5.99

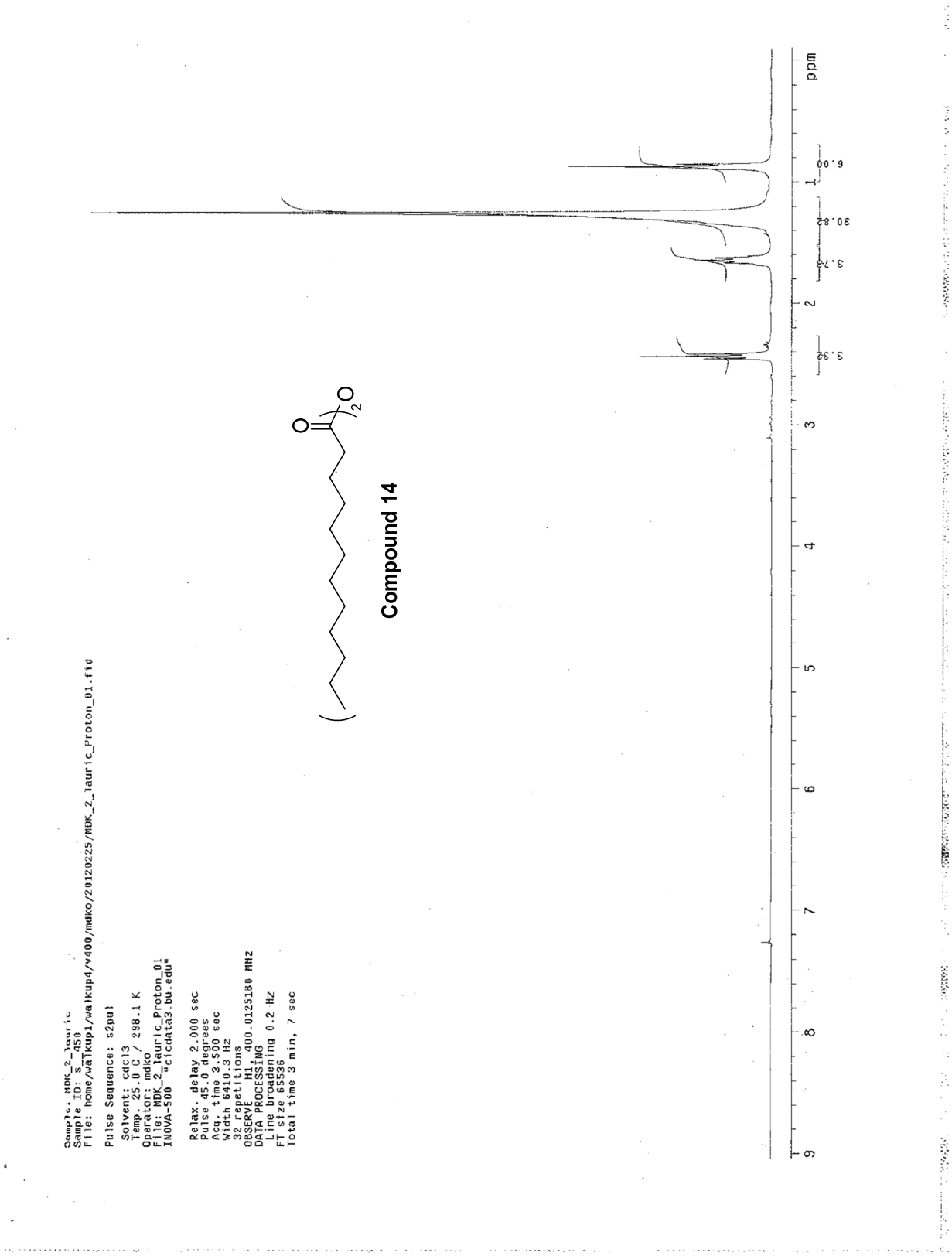

Sample: MK_1_277_crude
Sample ID:s_375
File: home/walkup1/m500/mdko/20110823/MK_1_277_crude_Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: mdko
File: MK_1_277_crude_Carbon_01
INOVA-500 "cicdata4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 33783.8 Hz
256 repetitions
OBSERVE C13, 125.6756350 MHz
DECOUPLE H1, 499.8057803 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 9 min, 51 sec
160.586
147.132
132.616
130.233
128.087
127.603
116.102
170
165
160
155
150
145
140
135
130
125
120
115
ppm

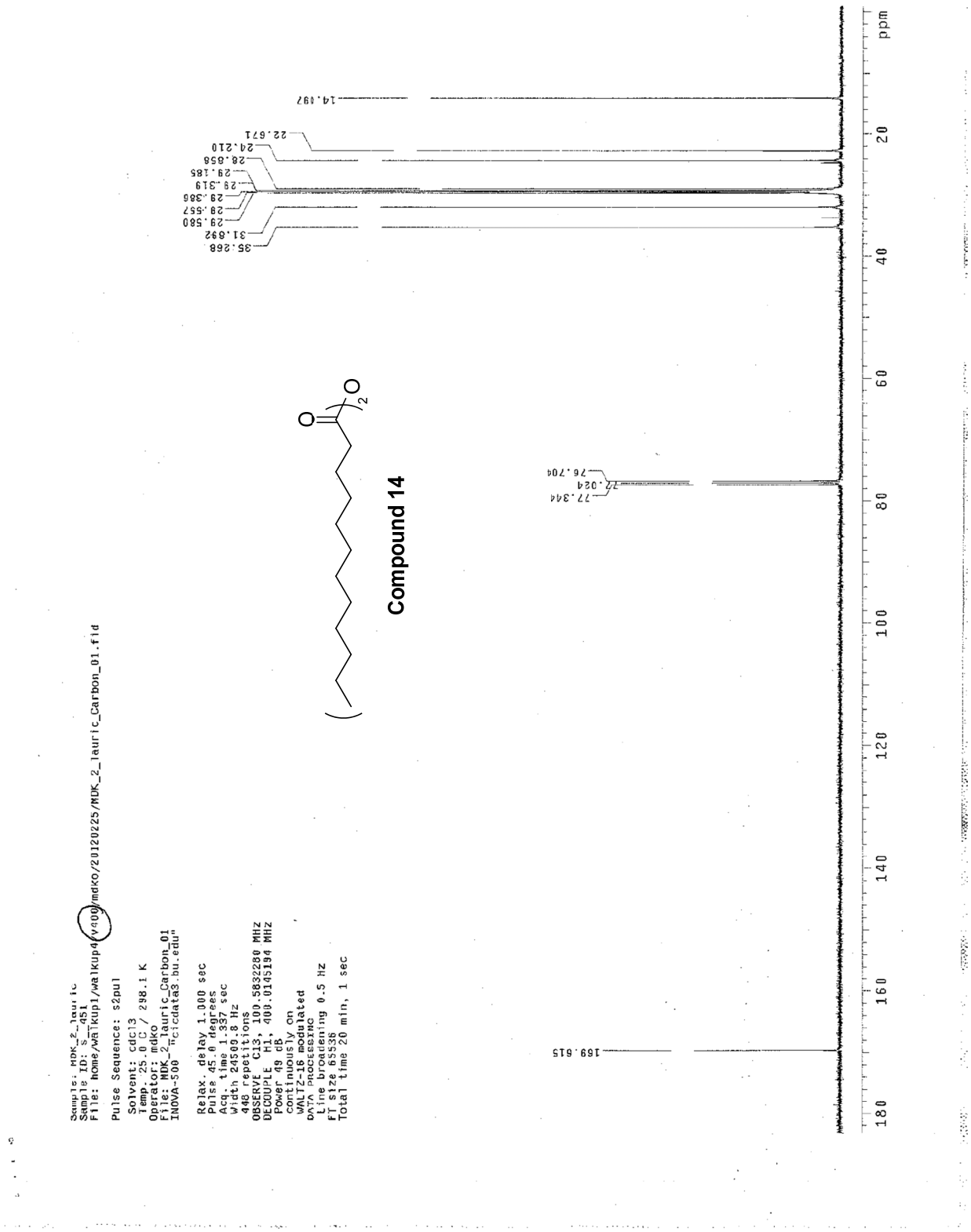

Sample: MK_1_271_crude
Sample ID: s__160
File: home/walkup1/m500/mdko/20110818/MK_1_271_crude_Proton_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mdko
File: MK_1_271_crude_Proton_01
INOVA-500 "ciCdata4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 10000.0 Hz
8 repetitions
OBSERVE H1, 499.8032808 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 31 sec
ppm
2.00
1.99




Sample: MDK_cyclohexanoic dried
Sample ID: S_303
File: home/walkupl/walkupl/vnmrsys/vnmr4/v400/mdko/20120102/MDK_cyclohexanoic_dried_Proton_02.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp.: 25.0 C / 298.1 K
Operator: mdko
File: MDK_cyclohexanoic_dried_Proton_02
INOVA-500 "wildata8.uhs.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.0125195 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min. 45 sec
ppm
2.00
3.91 3.79
3.65 2.42 5.84


Sample: MDK_1_cyclohexanoic_P
Sample ID: s_2is
File: /home/walkup4/vnmrsys/data/mdko/20120322/MDK_1_cyclohexanoic_P_Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp.: 25.0 C / 298.1 K
Operator: mdko
File: MDK_1_cyclohexanoic_P_Carbon_01
VNMRS-400 "v400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.337 sec
Width 24509.8 Hz
128 repetitions
OBSERVE C13, 100.5832280 MHz
DECOUPLE H1, 400.0145194 MHz
Power 49 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 min, 1 sec
28.397
25.571
25.155
43.954
77.329
77.016
76.697
171.883
180
160
140
120
100
80
60
40
20
ppm




Sample: MDK_2_lauric
Sample ID: s_451
File: home/walkup1/walkup4/v400/mdko/20120225/MDK_2_lauric_Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: mdko
File: MDK_2_lauric_Carbon_01
INOVA-500 "cicdata3.bu.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.337 sec
Width 24509.8 Hz
440 repetitions
OBSERVE C13, 100.5832280 MHz
DECOUPLE H1, 400.0145194 MHz
Power 49 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 20 min, 1 sec
Compound 14
14.097
22.671
24.210
28.856
29.185
29.319
29.386
29.557
29.580
31.892
35.268
77.344
77.024
76.704
169.615
ppm