Supplemental Information

for

Bifunctional Catalyst Promotes Highly Enantioselective Bromolactonizations to Generate Stereogenic C–Br Bonds

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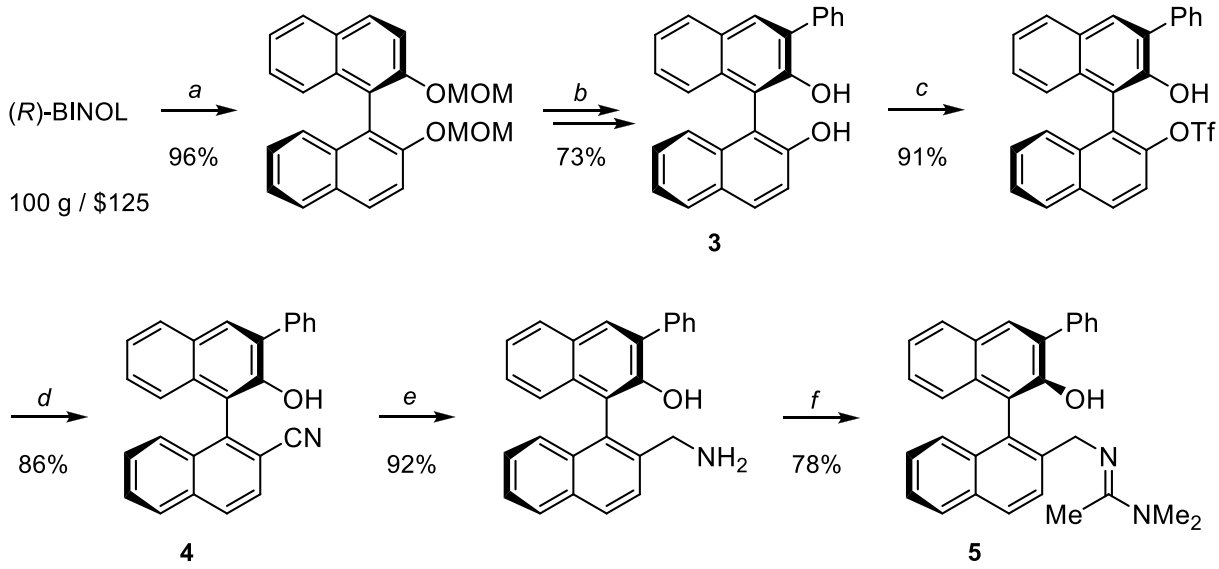
General

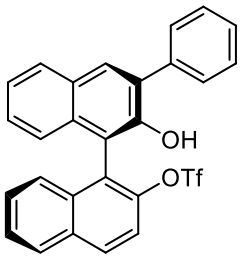
Solvents were purified before use as follows unless otherwise noted. Dichloromethane (CH_2Cl_2) and benzene were distilled from calcium hydride immediately prior to use. Tetrahydrofuran and diethyl ether were dried by filtration through two columns of activated, neutral alumina according to the procedure described by Grubbs.[1] Methanol (MeOH), acetonitrile (MeCN), and dimethylformamide (DMF) were dried by filtration through two columns of activated molecular sieves, and toluene was dried by filtration through one column of activated, neutral alumina followed by one column of Q5 reactant. These solvents were determined to have less than 50 ppm H_2O by Karl Fischer coulometric moisture analysis. Chloroform and acetone were distilled from $CaSO_4$ and stored over 4 Å molecular sieves. Reagents were reagent grade and used without purification unless otherwise noted. Trifluoromethanesulfonic anhydride (Tf_2O) was freshly distilled from P_2O_5. Alkyl halides were passed through a plug of silica and distilled. KCN was crushed and heated at 80 °C under vacuum for 3 h prior to use. Zinc powder was activated and stored under argon. Triethylamine (Et_3N), ethylene diamine, diisopropylethylamine (Hünig's base), and diisopropylamine were refluxed with, distilled from, and stored over KOH. In nickel(0), palladium(0), and copper(I)-catalyzed reactions, all solvents were freed from oxygen by three freeze-pump-thaw cycles prior to use. All reactions were performed in flame-dried glassware under nitrogen or argon; reaction temperatures refer to the temperature of the cooling/heating bath.

Analytical HPLC separations were performed using a Pirkel Covalent (S,S) Whelk-O1 (Regis Technologies, Inc.), or a Chiralcel OD-H (Daicel Chemical Industries, Ltd.) column, as indicated. Infrared (IR) spectra were obtained either neat on sodium chloride or as solutions in the solvent indicated and reported as wavenumbers (cm^{-1}). Proton nuclear magnetic resonance (^1H NMR) and carbon nuclear magnetic resonance (^{13}C NMR) spectra were obtained at the indicated field as solutions in $CDCl_3$ unless otherwise indicated. Chemical shifts are referenced to the deuterated solvent (*e.g.,* for $CDCl_3$, δ = 7.26 ppm and 77.0 ppm for ^1H and ^{13}C NMR, respectively) and are reported in parts per million (ppm, δ) relative to tetramethylsilane (TMS, δ = 0.00 ppm). Coupling constants (*J*) are reported in Hz and the splitting abbreviations used are: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; comp, overlapping multiplets of magnetically nonequivalent protons; br, broad; app, apparent.

Synthesis of Catalyst 5

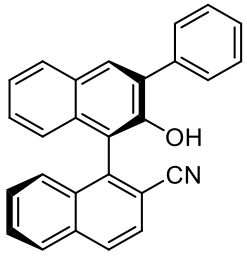


(*a*) Maruoka, et al.[2] (*b*) Shi, et al.[3] (*c*) EtN(*i*-Pr)$_2$, Tf_2O, DCM, –78 °C; (*d*) KCN, Ni(PPh$_3$)$_2$Br$_2$/PPh$_3$/Zn, CH_3CN, 70 °C; (*e*) BH_3-THF, 0 °C to reflux; HCl(aq), THF, reflux; (*f*) *N*,*N*-dimethylacetamide dimethylacetal, CH_3CN.

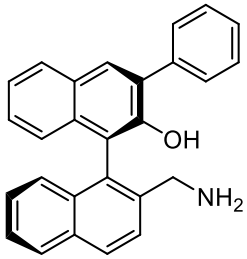


(*R*)-2'-Hydroxy-3'-phenyl-1,1'-binaphthyl-2-yl trifluoromethanesulfonate. EtN(*i*-Pr)$_2$ (4.52 g, 35.1 mmol) was added to a solution of (*R*)-3-phenyl-BINOL (**3**) (12.0 g, 33.2 mmol) (prepared in 4 steps from (*R*)-BINOL by the methods of Maruoka[2] and Shi[3] in 70% overall yield) in CH_2Cl_2 (140 mL), and the mixture was cooled to –78 °C. A solution of trifluoromethansulfonic anhydride (9.34 g, 33.2 mmol) in CH_2Cl_2 (10 mL) was added, and the mixture was stirred at –78 °C for 30 min. The reaction was quenched with saturated aqueous $NaHCO_3$ (100 mL), and the layers were separated. The aqueous fraction was extracted with CH_2Cl_2 (30 mL), and the combined organic fractions were dried (Na_2SO_4) and purified by column chromatography eluting with CH_2Cl_2 to give 16.2 g (99%) of the mono-triflate as a nearly pure off-white solid. The subsequent reaction is particularly sensitive to the purity of the triflate so it must be repurified by column chromatography eluting with Et_2O/hexanes (1:6) to give 14.9 g (91%) of the mono-triflate as a white solid: mp 68–69 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.11 (d, *J* = 8.8 Hz, 1 H), 8.02 (d, *J* = 8.0 Hz, 1 H), 7.96 (s, 1 H), 7.90 (d, *J* = 8.4 Hz, 1 H), 7.68–

7.64 (comp, 2 H), 7.60–7.34 (comp, 8 H), 7.28 (td, J = 6.8, 1.6 Hz, 1 H), 7.03 (d, J = 8.4, 1 H), 5.27 (s, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.1, 146.0, 136.8, 133.4, 133.1, 132.8, 131.0, 130.5, 129.5, 129.2, 128.9, 128.4, 128.3, 128.2, 127.9, 127.3, 127.0, 126.7, 126.2, 124.4, 124.1, 119.8, 116.7, 112.9; IR (neat) 3538, 3061, 1421, 1215, 1140, 949, 836 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_{27}H_{17}F_3NaO_4S]^+$ (M+Na), 517.0692; found 517.0692; $[\alpha]^{25}_D$ +20.7 (c = 1.0, $CHCl_3$).

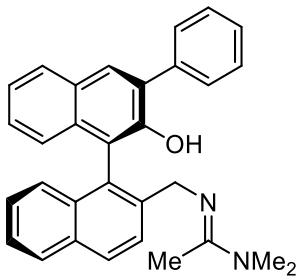


(*R*)-2'-Hydroxy-3'-phenyl-1,1'-binaphthyl-2-carbonitrile (4). A mixture of the triflate (above) (1.4 g, 2.8 mmol) (the purity of the triflate was critical), KCN (300 mg, 4.6 mmol), $Ni(PPh_3)_2Br_2$ (200 mg, 0.27 mmol), PPh_3 (200 mg, 0.76 mmol), and zinc (70 mg, 1.1 mmol) in oxygen-free CH_3CN (4 mL) was stirred at room temperature until the red-brown catalyst had formed (ca. 10 min). The reaction was then stirred at 70 °C for 2 h. The mixture was poured into Et_2O (100 mL), filtered, and concentrated under reduced pressure. The crude residue was purified by column chromatography eluting with CH_2Cl_2 to give 905 mg (86%) of **4** as a pale yellow solid, which was used in the next step. Analytically pure material was available by triturating with Et_2O to give 830 mg (79%) of **4** as a white solid: mp 204–205 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.02 (d, J = 8.8 Hz, 1 H), 7.98 (d, J = 8.4 Hz, 1 H), 7.95 (s, 1 H), 7.89 (d, J = 8.0 Hz, 1 H), 7.81 (d, J = 8.4 Hz, 1 H), 7.67–7.59 (comp, 3 H), 7.54–7.48 (comp, 3 H), 7.47–7.34 (comp, 3 H), 7.27 (td, J = 7.2, 1.2 Hz, 1 H), 6.94 (d, J = 8.4, 1 H), 5.32 (s, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 148.6, 140.8, 136.47, 135.0, 133.2, 132.3, 131.0, 130.3, 129.5, 129.4 129.2, 128.9, 128.5, 128.4, 128.4, 127.9, 127.2, 127.1, 127.1, 124.2, 124.1, 118.5, 116.3, 112.3; IR (neat) 3535, 3365, 2228, 1428, 1260, 909, 732 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_{27}H_{17}NNaO]^+$ (M+Na), 394.1202; found 394.1202; $[\alpha]^{25}_D$ –23.0 (c = 1.0, $CHCl_3$).



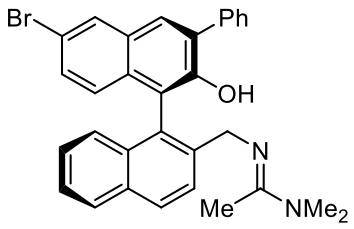
(*R*)-2'-(Aminomethyl)-3-phenyl-1,1'-binaphthyl-2-ol. A freshly prepared solution of BH_3 in THF (20 mL, 1 M, 20 mmol)[4] was added to a solution of nitrile **4** (2.1 g, 5.7 mmol) in THF (10 mL) at 0 °C, and the solution was stirred at room temperature for 20 min. The temperature was gradually raised to 70 °C over 20 min, and

mixture was heated under reflux for 30 min. The mixture was cooled to 0°C, and MeOH (5 mL) was added dropwise. The solution was stirred at room temperature for 20 min and concentrated under reduced pressure. The crude solid was dissolved in THF (40 mL) and HCl [6 mL, 1 M (aq)] was added. The mixture was stirred for 5 min at room temperature, and then heated under reflux for 2 min. The solution was allowed to cool to room temperature over ca. 20 min. The mixture was poured into saturated aqueous $NaHCO_3$ (100 mL), and the organic solvent was removed under reduced pressure (20 torr, room temperature). The aqueous mixture was extracted with CH_2Cl_2 (3 x 50 mL), and the combined organic fractions were dried (Na_2SO_4) and concentrated under reduced pressure. The crude residue was purified by column chromatography eluting with Et_3N/CH_2Cl_2 (1:50). The resulting solid was dissolved in CH_3CN (100 mL), filtered, and concentrated under reduced pressure to give 2.0 g (92%) of the amine as a pale yellow solid: mp 123–124 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.93 (s, 1 H), 7.91–7.85 (comp, 3 H), 7.69–7.66 (comp, 2 H), 7.45 (d, *J* = 8.4 Hz, 1 H), 7.43–7.36 (comp, 3 H), 7.33–7.26 (comp, 2 H), 7.23–7.09 (comp, 3 H), 6.78 (d, *J* = 8.4 Hz, 1 H), 4.04 (br s, 3 H), 3.61 (d, *J* = 12.0 Hz, 1 H), 3.50 (d, *J* = 12.0 Hz, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 150.6, 138.8, 138.5, 133.7, 133.5, 133.3, 133.2, 132.6, 129.9, 129.8, 129.0, 128.8, 128.1, 128.0, 127.9, 127.1, 126.7, 126.5, 126.4, 126.2, 125.8, 124.8, 123.5, 120.7, 45.3; IR (neat) 3055, 1407, 908, 732 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_{27}H_{22}NO]^+$ (M+H), 376.1696; found 376.1693; $[\alpha]^{24}_D$ +21.3 (c = 1.0, $CHCl_3$).



(*R*,*E*)-*N*'-((2'-Hydroxy-3'-phenyl-1,1'-binaphthyl-2-yl)methyl)-*N*,*N*-dimethylacetimidamide (5). *N*,*N*-Dimethylacetamide dimethylacetal (0.49 g, 3.3 mmol)[5] was added to a solution of the amine (above) (1.2 g, 3.2 mmol) in CH_3CN (6 mL), and the solution was stirred at room temperature for 1 h. The mixture was concentrated under reduced pressure, and the crude residue was purified by column chromatography eluting with $Et_3N/MeOH/CH_3CN/CH_2Cl_2$ (2:3:15:80). The resulting solid was dissolved in CH_3CN (100 mL), filtered, and concentrated under reduced pressure. The yellow solid was dissolved in Et_2O (10 mL), filtered, and precipitated from Et_2O/hexanes to give 1.1 g (78%) of **5** as a yellow powder: mp 189–190 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 9.42 (br s, 1 H), 7.94 (s, 1 H), 7.89 (d, *J* = 8.4 Hz, 1 H), 7.85 (d, *J* = 8.0 Hz, 2 H), 7.75–7.72 (comp, 2 H), 7.49 (d, *J* = 8.4 Hz, 1 H), 7.41–7.35 (comp, 3 H), 7.32–7.22 (comp, 2 H), 7.20–7.14 (comp, 2 H), 7.12 (t, *J* = 8.0 Hz, 1 H), 6.89 (d, *J* = 8.4 Hz, 1 H), 4.28 (d, *J* = 12.8 Hz, 1 H), 4.18 (d, *J* = 12.8 Hz, 1 H), 2.76 (s, 6 H), 1.85 (s, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.3, 153.1, 139.5, 137.8, 137.7, 134.7, 133.9, 133.5, 133.3,

130.0, 129.2, 128.6, 128.3, 127.9, 127.8, 127.7, 127.5, 127.0, 126.6, 126.0, 125.7, 125.4, 125.0, 122.9, 122.5, 53.9, 38.7, 13.6; IR (neat) 3053, 1632, 1411, 908, 731 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_{31}H_{29}N_2O]^+$ (M+H), 445.2274; found 445.2275; $[\alpha]^{24}_D$ +197.7 (c = 1.0, $CHCl_3$).

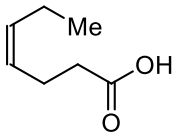


(*R*,*E*)-*N*'-((6'-Bromo-2'-hydroxy-3'-phenyl-1,1'-binaphthyl-2-yl)methyl)-*N*,*N*-dimethylacetimidamide (6-Br-5). A solution of 2,4,4,6-tetrabromocyclohexadienone (TBCO) (0.092 g, 0.225 mmol) in CH_2Cl_2 (1 mL) was added dropwise to a solution of **5** (0.100 g, 0.225 mmol) and propionic acid (0.033 g, 0.450 mmol) in toluene (2 mL) at –50 °C, and the reaction was stirred for 20 min. The reaction was quenched with saturated aqueous Na_2SO_3 (3 mL), and the mixture was stirred vigorously at room temperature. The mixture was diluted with Et_2O (10 mL), washed with water (3 mL) and 5% aqueous Na_2CO_3 (2 x 5 mL). The organic fraction was dried ($MgSO_4$) and purified by column chromatography, eluting with Et_3N/MeOH/CH_3CN/CH_2Cl_2 (2:3:15:80). The resulting solid was dissolved in CH_3CN (20 mL), filtered, and concentrated under reduced pressure. The yellow solid was dissolved in Et_2O (10 mL), filtered, and precipitated from Et_2O/hexanes to give 0.100 g (85%) of 6-Br-**5** as a yellow powder: mp 115–116 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.88 (br s, 1 H), 7.98 (d, *J* = 1.2 Hz, 1 H), 7.89–7.82 (comp, 3 H), 7.73–7.70 (comp, 2 H), 7.48–7.44 (m, 1 H), 7.41–7.35 (comp, 4 H), 7.33–7.27 (m, 1 H), 7.19 (td, *J* = 8.4, 1.2 Hz, 1 H), 7.16–7.12 (comp, 2 H), 6.71 (d, *J* = 8.8 Hz, 1 H), 4.28 (d, *J* = 13.2 Hz, 1 H), 4.17 (d, *J* = 13.2 Hz, 1 H), 2.78 (s, 6 H), 1.89 (s, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.6, 154.8, 149.2, 139.3, 136.8, 136.1, 134.0, 133.4, 133.3, 132.6, 129.9, 129.7, 128.7, 128.4, 128.2, 127.9, 127.7, 127.5, 126.9, 126.7, 126.6, 126.2, 125.5, 122.2, 116.0, 53.5, 38.9, 13.8; IR (neat) 3055, 2930, 1633, 1415, 908, 731 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_{31}H_{28}BrN_2O]^+$ (M+H), 523.1380; found 523.1377; $[\alpha]^{25}_D$ +130.1 (c = 1.0, $CHCl_3$).

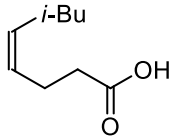
Synthesis of Olefinic Acids

All olefinic acids were made by literature procedures as follows: (Z)-5-alkyl-4-enoic acids **6a-e** were made at the specified temperature by the method of Yeung, et al.,[6] and were isolated as the specified mixture of *Z* and *E* isomers (determined by ^{1}H NMR); (*E*)-6-methylhept-4-enoic acid (***E*-6c**) was made by the method of Kaga, et al.;[7] (*E*)-5-aryl-4-enoic acids **6f-h** were made by the method of Yeung, et al.;[6] 4-aryl-4-enoic acids **9a-c** were made by the method of Yeung, et al.;[8] (*E*)-4-methylhex-4-enoic acid (**9d**) was made by the methods of Back, et

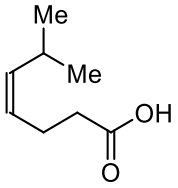
al., and Mane, et al.;[9] 2-(2-phenylallyloxy)acetic acid (**9e**) was made by the method of Fujioka, et al.;[10] (*E*)-2-(2-methylbut-2-enyloxy)acetic acid (**9f**) was made by the method of Suginome, et al.;[11] cyclohexa-2,5-dienecarboxylic acid **12** was made by Birch reduction of benzoic acid.[12] Characterization data is reported below for compounds that have not previously been characterized.



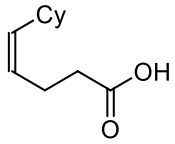
(*Z*)-Hept-4-enoic acid (6a). Wittig reaction executed on a 10.0 mmol scale at –40 °C, to give 1.09 g (85%) of **6a** as a clear, colorless oil: 20:1 *Z*/*E* ratio; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.48–5.40 (m, 1 H), 5.35–5.29 (m, 1 H), 2.44–2.33 (comp, 4 H), 2.15–2.02 (m, 2 H), 0.96 (t, *J* = 7.6 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 179.8, 133.5, 126.3, 34.2, 22.4, 20.5, 14.2; IR (neat) 1712 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_7H_{11}O_2]^-$ (M–H), 127.0765; found 127.0762.



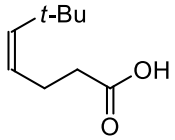
(*Z*)-7-Methyloct-4-enoic acid (6b). Wittig reaction executed on a 5.0 mmol scale at –55 °C, to give 0.7 g (90%) of **6b** as a clear, colorless oil: 22:1 *Z*/*E* ratio; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.71 (br s, 1 H), 5.49–5.36 (comp, 2 H), 2.43–2.33 (comp, 4 H), 1.94 (t, *J* = 6.8 Hz, 2 H), 1.61 (septet, *J* = 6.6 Hz, 1 H), 0.89 (d, *J* = 6.6 Hz, 6 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 179.7, 130.5, 127.6, 36.3, 36.2, 34.1, 28.5, 22.6, 22.3; IR (neat) 2957, 1713, 1413, 1291, 933 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_9H_{15}O_2]^-$ (M–H), 155.1078; found 155.1075.



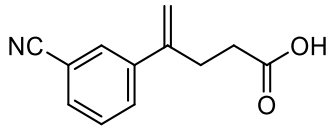
(*Z*)-6-Methylhept-4-enoic acid (6c). Wittig reaction executed on a 7.8 mmol scale at –40 °C, to give 0.7 g (62%) of **6c** as a clear, colorless oil: 26:1 *Z*/*E* ratio; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 11.25 (br s, 1 H), 5.30–5.18 (comp, 2 H), 2.65–2.58 (m, 1 H), 2.44–2.37 (comp 4 H), 0.94 (d, *J* = 6.8 Hz, 6 H);^{13}C NMR ($CDCl_3$, 100 MHz) δ 179.8, 139.3, 124.5, 34.4, 26.5, 23.1, 22.6; IR (neat) 2960, 1713, 1413, 1281, 931, 746 cm^{-1}; HRMS (ESI) *m/z* calcd $[C_8H_{13}O_2]^-$ (M–H), 141.0921; found 141.0919.



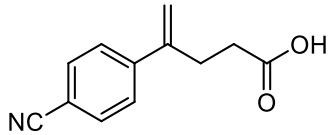
(*Z*)-5-Cyclohexylpent-4-enoic acid (6d). Wittig reaction executed on a 5.0 mmol scale at –50 °C; additional purification by heating at 80 °C under vacuum for 5 h gave 0.3 g (34%) of **6d** as a clear, colorless oil: >50:1 *Z*/*E* ratio; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 11.33 (br s, 1 H), 5.31–5.19 (comp, 2 H), 2.42–2.35 (comp, 4 H), 2.32–2.20 (m, 1 H), 1.72–1.56 (comp, 5 H), 1.34–1.00 (comp, 5 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ179.86, 137.86, 124.99, 36.30, 34.46, 33.23, 25.99, 25.87, 22.73; IR (neat) 2925, 2850, 1712, 1448, 1279, 947 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_{11}H_{17}O_2]^-$ (M–H), 181.1234; found 181.1231.



(*Z*)-6,6-Dimethylhept-4-enoic acid (6e). Wittig reaction executed on a 5.0 mmol scale to give 0.6 g (82%) of **6e** as a clear, colorless oil: >50:1 *Z*/*E* ratio; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 11.57 (br s, 1 H), 5.38 (dt, *J* = 12.0, 1.6 Hz, 1 H), 5.16–5.08 (m, 1 H), 2.56–2.49 (comp, 2 H), 2.43–2.38 (comp, 2 H), 1.12 (s, 9 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 179.8, 141.5, 125.8, 34.6, 33.3, 31.1, 31.0, 26.9, 23.5; IR (neat) 2958, 1713, 1414, 1283, 1209, 935 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_9H_{15}O_2]^-$ (M–H), 155.1078; found 155.1076.

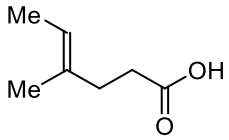


4-(3-Cyanophenyl)pent-4-enoic acid (9b) was isolated in 30% yield (2 steps) as a white solid: mp 70–71 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 11.21 (br s, 1 H), 7.69–7.56 (comp, 3 H), 7.48–7.43 (m, 1 H), 5.38 (s, 1 H), 5.23 (s, 1 H), 2.84 (t, *J* = 7.5 Hz, 2 H), 2.54 (t, *J* = 7.2 Hz, 2 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 178.8, 144.6, 141.8, 131.1, 130.4, 129.8, 129.3, 118.7, 115.1, 112.7, 32.5, 29.7; IR (neat) 3087, 2919, 2231, 1711, 1416, 1275, 1215, 902, 804 cm^{-1}; HRMS (CI) *m/z* calcd for $[C_{12}H_{11}NO_2]^+$ (M+H), 201.0790; found 201.0789.

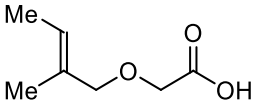


4-(4-Cyanophenyl)pent-4-enoic acid (9c) was isolated in 43% yield (2 steps) as a white solid: mp 89–90 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 10.48 (br s, 1 H), 7.63 (dd, *J* = 6.6, 1.8 Hz, 2 H), 7.49 (dd, *J* = 6.6, 2.1 Hz, 2

H), 5.43 (s, 1 H), 5.26 (s, 1 H), 2.84 (t, J = 7.5 Hz, 2 H), 2.54 (t, J = 7.2 Hz, 2 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 178.5, 145.1, 145.1, 132.3, 126.7, 118.7, 115.8, 111.3, 32.6, 29.6; IR (neat) 2926, 2232, 1704, 908, 841 cm^{-1}; HRMS (CI) *m/z* calcd for $[C_{12}H_{11}NO_2]^+$ (M+H), 201.0790; found 201.0789.

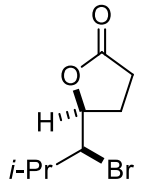


(*E*)-4-Methylhex-4-enoic acid (9d) was isolated in 76% yield as a clear, colorless oil: 36:1 *E*/*Z* ratio; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 11.64 (br s, 1 H), 5.29–5.23 (m, 1 H), 2.48–2.43 (comp, 2 H), 2.31 (t, J = 7.6 Hz, 2 H), 1.62 (t, J = 1.0, 3 H), 1.59–1.55 (comp, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 180.2, 133.6, 119.4, 32.2, 32.9, 15.5, 13.3; IR (neat) 2980, 2920, 1712, 1413, 1300, 938 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_7H_{11}O_2]^-$ (M–H), 127.0765; found 127.0765.



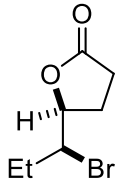
(*E*)-2-(2-Methylbut-2-enyloxy)acetic acid (9f) was isolated in 92% yield as a white solid: mp 46–47 °C, >50:1 *E*/*Z* ratio; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.43 (br s, 1 H), 5.57–5.50 (m, 1 H), 4.06 (s, 2 H), 3.98 (s, 2 H), 1.67–1.63 (comp, 6 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 175.5, 140.5, 131.5, 124.7, 65.7, 13.4, 13.2; IR (neat) 2919, 1731, 1432, 1245, 1112 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_7H_{11}O_3]^-$ (M–H), 143.0714; found 143.0712.

General Procedure for Enantioselective Bromolactonization

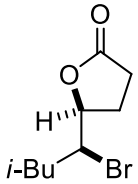


(*S*)-5-((*S*)-1-Bromo-2-methylpropyl)dihydrofuran-2(*3H*)-one (7c). A solution of TBCO (0.197 g, 0.480 mmol) in CH_2Cl_2 (4 mL) was added dropwise to a solution of (*Z*)-6-methylhept-4-enoic acid **6c** (0.057 g, 0.400 mmol) and catalyst **5** (0.018 g, 0.040 mmol) in toluene (8 mL) at –50 °C, and the solution was stirred for 14 h. The reaction was quenched with saturated aqueous Na_2SO_3 (10 mL), and the mixture was warmed to room temperature with vigorous stirring. The mixture was diluted with Et_2O (40 mL) and water (10 mL), and the organic fraction was washed with 5% aqueous Na_2CO_3 (2 x 20 mL), dried ($MgSO_4$), filtered and concentrated

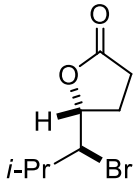
under reduced pressure. The crude residue was purified by column chromatography, eluting with DCM/toluene (2:1) to give 0.083 g (94%) of **7c** as a clear, colorless oil: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.76–4.71 (m, 1 H), 3.87 (dd, *J* = 6.0, 3.6 Hz, 1 H), 2.76–2.66 (m, 1 H), 2.59–1.48 (m, 1 H), 2.44–2.33 (m, 1 H), 2.22–2.06 (comp, 2 H), 1.10 (d, *J* = 6.4 Hz, 6 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 176.4, 79.9, 66.8, 32.8, 28.3, 26.7, 21.2, 20.3; IR (neat) 2966, 2933, 2876, 1769, 1176, 1022, 908 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_8H_{13}BrNaO_2]^+$ (M+Na), 242.9991; found 242.9992; $[\alpha]^{25}_D$ +53.0 (c = 1.0, $CHCl_3$); HPLC (210 nm): Whelk-O1 (20% *i*-PrOH / hexanes, 1.2 mL/min) 17.4 min (minor), 20.3 min (major); 97:3 er.



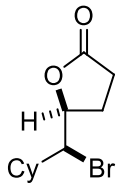
(*S*)-5-((*S*)-1-Bromopropyl)dihydrofuran-2(*3H*)-one (7a). Isolated 0.074 g (90%) of **7a** as a clear, colorless oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.72–4.64 (m, 1 H), 4.04–3.93 (m, 1 H), 2.78–2.65 (m, 1 H), 2.62–2.33 (comp, 2 H), 2.28–2.14 (m, 1 H), 2.05–1.88 (comp, 2 H), 1.12 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 176.5, 80.7, 59.8, 28.2, 27.9, 25.3, 12.4; IR (neat) 1774 cm^{-1}; HRMS (CI) *m/z* calcd for $[C_7H_{12}BrO_2]^+$ (M+H), 207.0021; found 207.0022; $[\alpha]^{23}_D$ +5.3 (c = 1.0, $CHCl_3$); HPLC (210 nm): Whelk-O1 (20% *i*-PrOH / hexanes, 1.2 mL/min) 15.5 min (minor), 18.9 min (major); 85:15 er.



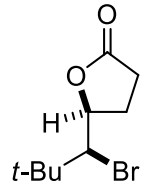
(*S*)-5-((*S*)-1-Bromo-3-methylbutyl)dihydrofuran-2(*3H*)-one (7b). Reaction executed on a 0.21 mmol scale, to give 0.043 g (87%) of **7b** as a clear, colorless oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.63 (ddd, *J* = 7.8, 6.3, 3.3 Hz, 1 H), 4.13 (dt, *J* = 10.5, 3.6 Hz, 1 H), 2.76–2.65 (m, 1 H), 2.61–2.45 (m, 1 H), 2.45–2.34 (m, 1 H), 2.27–2.15 (m, 1 H), 2.00–1.88 (comp, 2 H), 1.62–1.53 (m, 1 H), 0.97 (d, *J* = 6.3 Hz, 3 H), 0.91 (d, *J* = 6.6 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 176.5, 81.1, 56.2, 43.0, 28.2, 25.9, 25.3, 23.0, 20.8; IR (neat) 2959, 2872, 1779, 1468, 1369, 1179, 1056, 1014, 913cm^{-1}; HRMS (CI) *m/z* calcd for $[C_9H_{16}O_2Br]^+$ (M+H), 235.0334; found 235.0336; $[\alpha]^{24}_D$ +2.7 (c = 1.0, $CHCl_3$); HPLC (210 nm): Whelk-O1 (20% *i*-PrOH / hexanes, 1.2 mL/min) 12.2 min (minor), 17.5 min (major); 95:5 er.



(*R*)-5-((*S*)-1-Bromo-2-methylpropyl)dihydrofuran-2(*3H*)-one (diastereo-7c). Isolated 0.087 g (98%) of **diastereo-7c** as a clear, colorless oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.64 (m, 1 H), 3.95 (dd, *J* = 9.0, 3.3 Hz, 1 H), 2.61–2.50 (comp, 3 H), 2.22–2.10 (comp, 2 H), 1.04 (d, *J* = 6.6 Hz, 3 H), 1.00 (d, *J* = 6.3 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 176.3, 79.5, 65.8, 30.1, 28.5, 27.6, 21.3, 16.9; IR (neat) 2967, 1785, 1463, 1174, 1022, 912 cm^{-1}; HRMS (CI) *m/z* calcd for $[C_8H_{14}O_2Br]^+$ (M+H), 221.0177; found 221.0178; $[\alpha]^{24}_D$ +13.7 (c = 1.0, $CHCl_3$); HPLC (210 nm): OD-H (0.5% *i*-PrOH / hexanes, 1.0 mL/min) 27.6 min (major), 31.3 min (minor); 71:29 er.

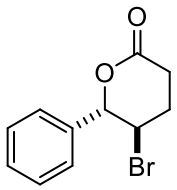


(*S*)-5-((*S*)-Bromo(cyclohexyl)methyl)dihydrofuran-2(*3H*)-one (7d). Isolated 0.098 g (94%) of **7d** as a clear, colorless oil: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.84–4.76 (m, 1 H), 3.86 (dd, *J* = 6.8, 3.2 Hz, 1 H), 2.79–2.67 (m, 1 H), 2.58–2.46 (m, 1 H), 2.43–2.31 (m, 1 H), 2.27–2.14 (m, 1 H), 2.10–2.01 (m, 1 H), 1.92–1.72 (comp, 4 H), 1.71–1.62 (m, 1 H), 1.38–1.08 (comp, 5 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 176.6, 78.8, 66.4, 42.3, 31.3, 31.2, 28.2, 26.6, 26.1, 26.0, 25.9; IR (neat) 2926, 2852, 1768, 1177 cm^{-1}; HRMS (ESI) *m/z* calcd for $[C_{11}H_{17}BrNaO_2]^+$ (M+Na), 283.0304; found 283.0305; $[\alpha]^{24}_D$ +40.7 (c = 1.0, $CHCl_3$); HPLC (210 nm): Whelk-O1 (20% *i*-PrOH / hexanes, 1.2 mL/min) 17.1 min (minor), 24.0 min (major); 98.5:1.5 er.

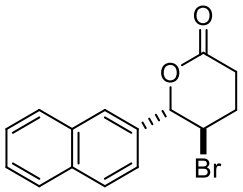


(*S*)-5-((*S*)-1-Bromo-2,2-dimethylpropyl)dihydrofuran-2(*3H*)-one (7e). Isolated 0.091 g (97%) of **7e** as a white solid: mp 126–127 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.85 (ddd, *J* = 8.1, 6.0, 1.2 Hz, 1 H), 3.85 (d, *J* = 0.9 Hz, 1 H), 2.76–2.65 (m, 1 H), 2.53–2.40 (m, 1 H), 2.39–2.31 (m, 1 H), 2.27–2.17 (m, 1 H), 1.16 (s, 9 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 176.9, 77.3, 72.4, 36.7, 28.3, 27.6, 27.4; IR (neat) 2977, 2938, 1766, 1353, 1184, 1065, 1025, 993, 921 cm^{-1}; HRMS (CI) *m/z* calcd for $[C_9H_{16}O_2Br]^+$ (M+H), 235.0334; found 235.0334; $[\alpha]^{24}_D$

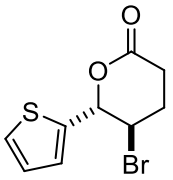
+55.7 (c = 1.0, $CHCl_3$); HPLC (210 nm): Whelk-O1 (20% *i*-PrOH / hexanes, 1.2 mL/min) 10.7 min (minor), 14.3 min (major); 97:3 er.



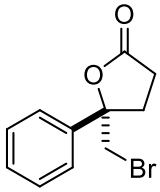
(5*R*,6*S*)-5-Bromo-6-phenyltetrahydro-*2H*-pyran-2-one (8f). Reaction executed on a 0.100 mmol scale at –60 °C, to give 0.024 g (94%) of **8f** as a white solid: mp 132–133 °C; spectra matched previously reported data;[6] $[\alpha]^{23}_D$ –6.0 (c = 1.0, $CHCl_3$); HPLC (210 nm): Whelk-O1 (3% CH_3CN / 20% *i*-PrOH / hexanes, 1.2 mL/min) 14.6 min (minor), 19.8 min (major); 98:2 er.



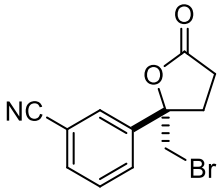
(5*R*,6*S*)-5-Bromo-6-(naphthalen-2-yl)tetrahydro-*2H*-pyran-2-one (8g). Isolated 0.118 g (97%) of **8g** as a white solid: mp 107–108 °C; spectra matched previously reported data;[6] $[\alpha]^{22}_D$ +32.5 (c = 1.0, $CHCl_3$); HPLC (225 nm): Whelk-O1 (6% CH_3CN / 20% *i*-PrOH / hexanes, 1.2 mL/min) 14.0 min (minor), 24.5 min (major); 96:4 er.



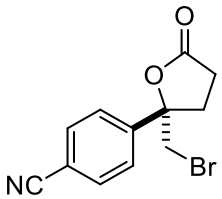
(5*R*,6*S*)-5-Bromo-6-(thiophen-2-yl)tetrahydro-*2H*-pyran-2-one (8h). Isolated 0.096 g (92%) of **8h** as a white solid: mp 95–96 °C; spectra matched previously reported data;[6] $[\alpha]^{22}_D$ +5.7 (c = 1.0, $CHCl_3$); HPLC (233 nm): Whelk-O1 (3% CH_3CN / 20% *i*-PrOH / hexanes, 1.2 mL/min) 12.9 min (minor), 21.6 min (major); 94:6 er.



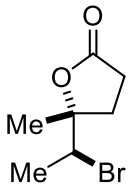
(*S*)-5-(Bromomethyl)-5-phenyldihydrofuran-2(*3H*)-one (10a). Isolated 0.101 g (99%) of **10a** as a semi-solid; spectra consistent with the data previously reported for the enantiomer of **10a**;[8] $[\alpha]^{25}_{D}$ –26.3 (c = 1.0, $CHCl_3$); HPLC (210 nm): Whelk-O1 (20% *i*-PrOH / hexanes, 1.2 mL/min) 16.0 min (minor), 23.8 min (major); 86:14 er.



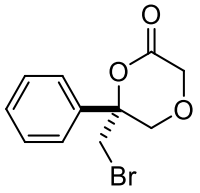
(*S*)-3-(2-(Bromomethyl)-5-oxotetrahydrofuran-2-yl)benzonitrile (10b). Reaction executed on 0.1 mmol scale, to give 0.025 g (89%) of **10b** as a clear, colorless oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.75–7.66 (comp, 3 H), 7.59–7.53 (m, 1 H), 3.72 (d, *J* = 11.4 Hz, 1 H), 3.68 (d, *J* = 11.1 Hz, 1 H), 2.90–2.77 (comp, 2 H), 2.65–2.53 (comp, 2 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.6, 142.4, 132.3, 129.8, 129.5, 128.8, 118.1, 113.1, 85.4, 40.1, 32.4, 28.7; IR (neat) 2962, 2231, 1788, 1483, 1421, 1243, 1164, 1041, 921 cm^{-1}; HRMS (CI) *m/z* calcd for $[C_{12}H_{11}NO_2Br]^+$ (M+H), 279.9973; found 279.9973; $[\alpha]^{25}_{D}$ –35.3 (c = 1.0, $CHCl_3$); HPLC (225 nm): Whelk-O1 (6% CH_3CN / 20% *i*-PrOH / hexanes, 1.2 mL/min) 11.5 min (minor), 12.5 min (major); 91:9 er.



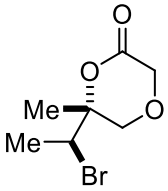
(*S*)-4-(2-(Bromomethyl)-5-oxotetrahydrofuran-2-yl)benzonitrile (10c). Isolated 0.102 g (92%) of **10c** as a white solid: mp 154–155 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.73 (d, *J* = 8.7 Hz, 2 H), 7.56 (d, *J* = 8.7 Hz, 2 H), 3.72 (d, *J* = 11.4 Hz, 1 H), 3.68 (d, *J* = 11.4 Hz, 1 H), 2.89–2.82 (comp, 2 H), 2.59–2.52 (comp, 2 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.6, 145.8, 132.6, 125.9, 118.0, 112.8, 85.7, 40.0, 32.5, 28.8; IR (neat) 2962, 2230, 1787, 1162, 1044, 841 cm^{-1}; HRMS (CI) *m/z* calcd for $[C_{12}H_{11}NO_2Br]^+$ (M+H), 279.9973; found 279.9975; $[\alpha]^{25}_{D}$ –36.0 (c = 0.5, $CHCl_3$); HPLC (230 nm): Whelk-O1 (6% CH_3CN / 20% *i*-PrOH / hexanes, 1.2 mL/min) 13.8 min (minor), 15.2 min (major); 94:6 er.



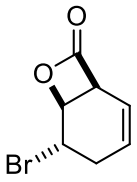
(*R*)-5-((*S*)-1-bromoethyl)-5-methyldihydrofuran-2(*3H*)-one (10d). Isolated 0.074 g (89%) of **10d** as a clear, colorless oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.18 (q, *J* = 6.9 Hz, 1 H), 2.67 (dt, *J* = 8.1, 0.9 Hz, 2 H), 2.67 (dt, *J* = 13.2, 9.2 Hz, 1 H), 2.11 (dt, *J* = 13.2, 7.8 Hz, 1 H), 1.74 (d, *J* = 6.6 Hz, 3 H), 1.52 (s, 3 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 175.8, 87.2, 54.9, 32.0, 29.1, 21.5, 20.8; IR (neat) 2983, 2937, 1778, 1451, 1384, 1192, 1147, 1074, 940 cm^{-1}; HRMS (CI) *m/z* calcd for $[C_7H_{12}O_2Br]$ $(M+H)^+$, 207.0021; found 207.0022; $[\alpha]^{23}_D$ +5.3 (c = 1.0, $CHCl_3$); HPLC (210 nm): Whelk-O1 (20% *i*-PrOH / hexanes, 1.2 mL/min) 13.1 min (minor), 14.4 min (major); 71:29 er.



(*R*)-6-(Bromomethyl)-6-phenyl-1,4-dioxan-2-one (11e). Isolated 0.106 g (98%) of **11e** as a white solid: mp 88–90 °C; spectra matched previously reported data;[10] $[\alpha]^{23}_D$ –10.0 (c = 1.0, $CHCl_3$); HPLC (210 nm): Whelk-O1 (20% *i*-PrOH / hexanes, 1.2 mL/min) 18.9 min (minor), 22.7 min (major); 86:14 er.



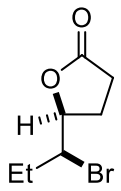
(*R*)-6-((*S*)-1-Bromoethyl)-6-methyl-1,4-dioxan-2-one (11f). Isolated 0.083 g (93%) of **11f** as a clear, colorless oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.35–4.28 (comp, 3 H), 4.05 (d, *J* = 12.9 Hz, 1 H), 3.76 (d, 12.6 Hz, 1 H), 1.78 (d, *J* = 6.9 Hz, 3 H), 1.53 (s, 3 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 166.4, 84.1, 69.8, 65.4, 49.4, 19.5, 17.6; IR (neat) 2986, 2941, 1747, 1456, 1268, 1104 cm^{-1}; HRMS (CI) *m/z* calcd for $[C_7H_{12}O_3Br]^+$ (M+H), 222.9970; found 222.9972; $[\alpha]^{24}_D$ –26.7 (c = 0.5, $CHCl_3$); HPLC (210 nm): Whelk-O1 (20% *i*-PrOH / hexanes, 1.2 mL/min) 9.9 min (major), 11.2 min (minor); 85:15 er.



(1*S*,5*S*,6*S*)-5-bromo-7-oxabicyclo[4.2.0]oct-2-en-8-one (13). Reaction executed in CH_2Cl_2/toluene (1:1, 12 mL) for 4 d, to give 0.058 g (72%) of **13** as a white solid: mp 96–98 °C; spectra matched the data previously reported for racemic **13**;[13] $[\alpha]^{23}_D$ +49.0 (c = 1.0, $CHCl_3$); HPLC (210 nm): OD-H (1% *i*-PrOH / hexanes, 1.0 mL/min) 31.0 min (minor), 33.5 min (major); 73:27 er.

HPLC traces

(**7a**) Whelk-O1, 20% IPA / hexanes, 1.2 mL/min. obs: 210 nm.



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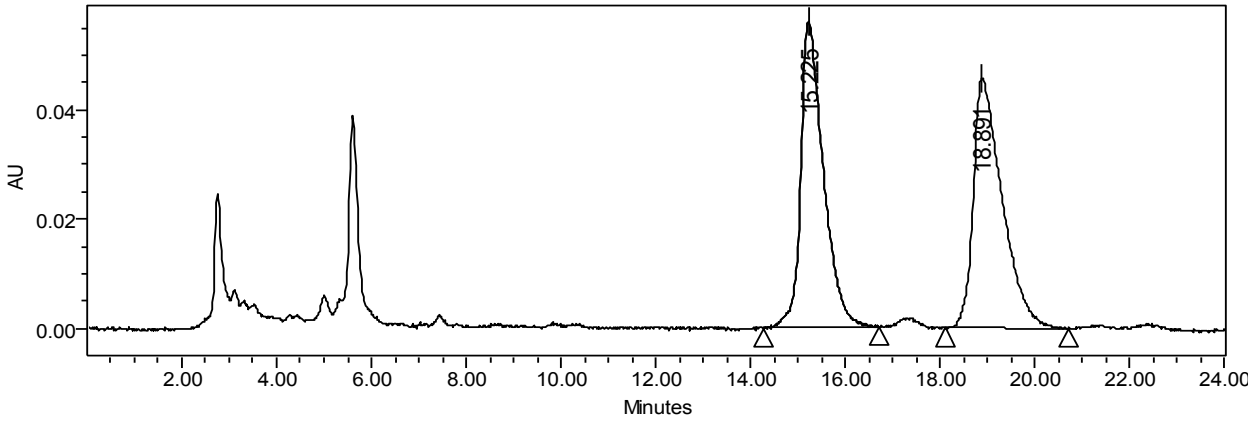

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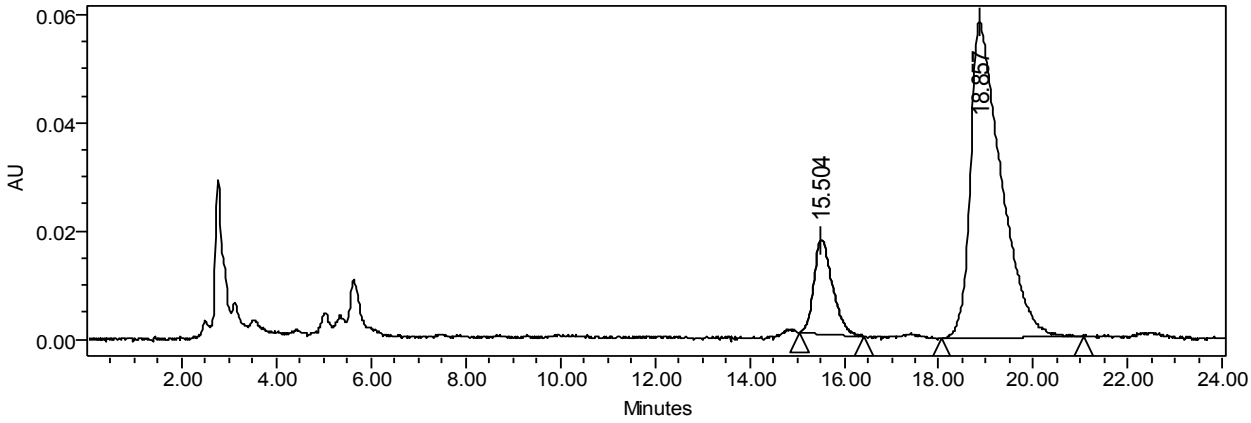
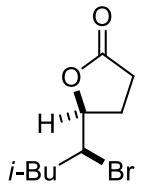


	Name	Retention Time	Area	% Area	Height	Int Type
2		18.857	2630209	84.59	58453	bb
1		15.504	479232	15.41	17316	bb

(**7b**) Whelk-O1, 20% IPA / hexanes, 1.2 mL/min. obs: 210 nm.



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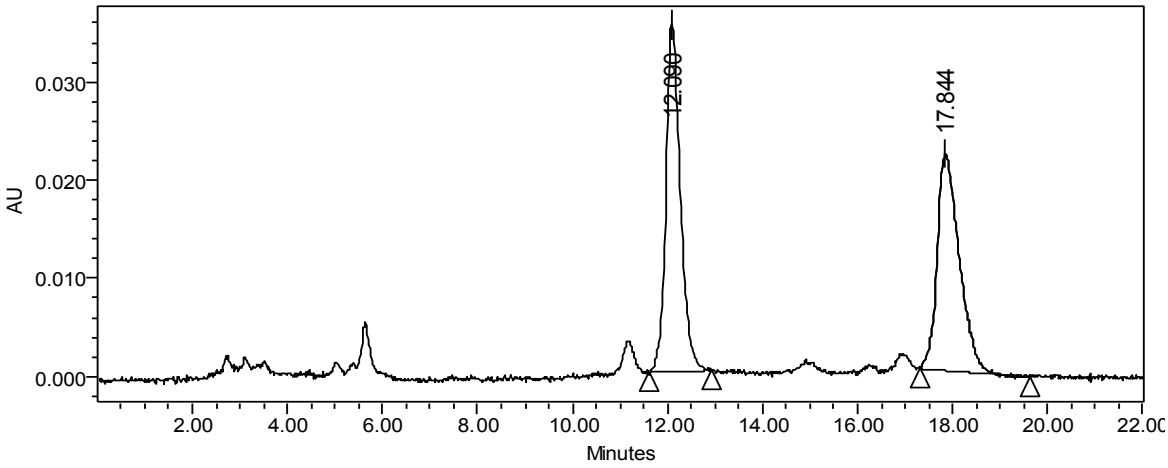

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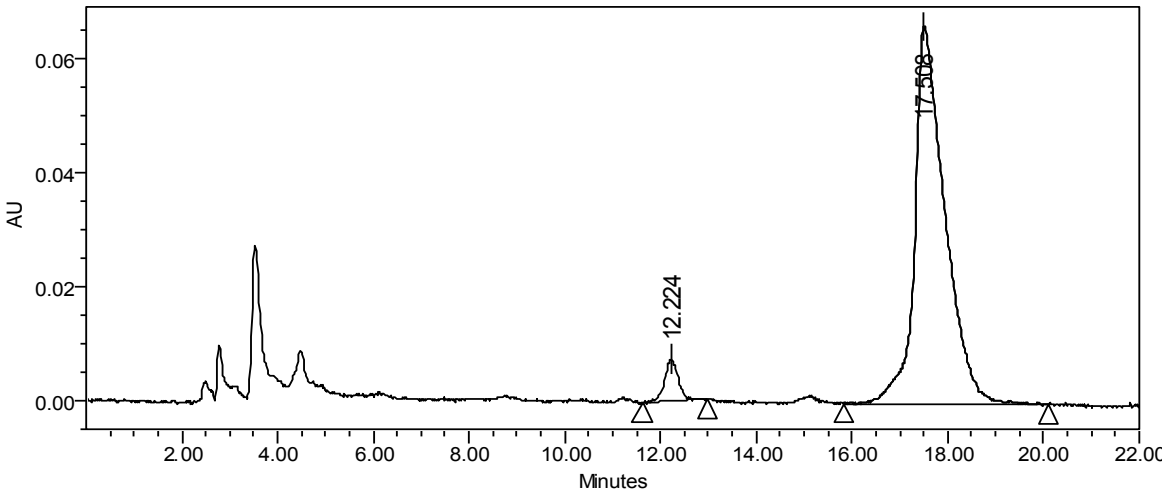
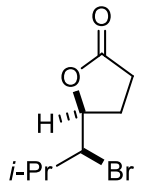


	Name	Retention Time	Area	% Area	Height	Int Type
1		12.224	144669	4.75	7408	bb
2		17.508	2903611	95.25	66296	bb

(**7c**) Whelk-O1, 20% IPA / hexanes, 1.2 mL/min. obs: 210 nm.



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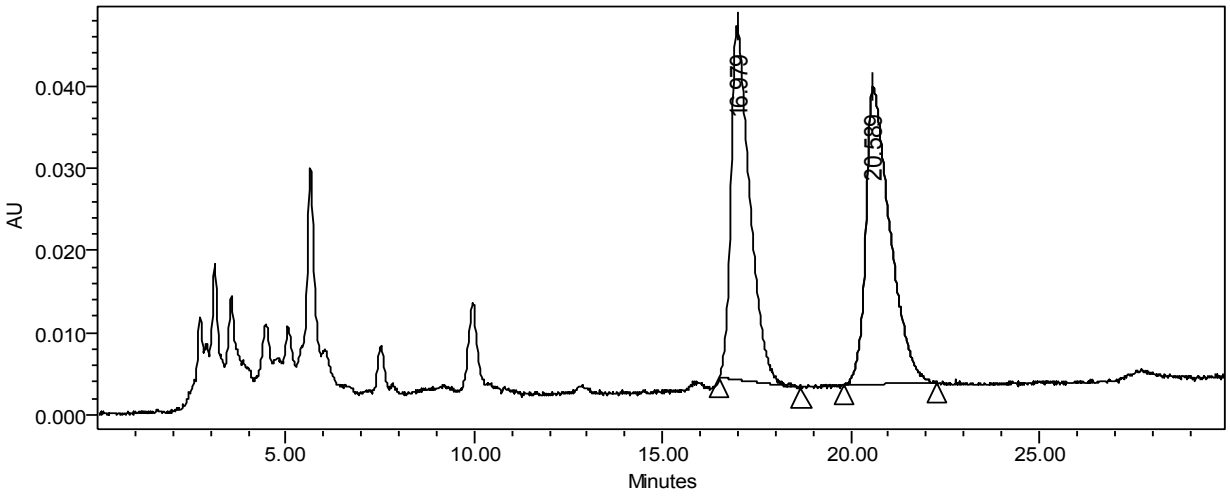

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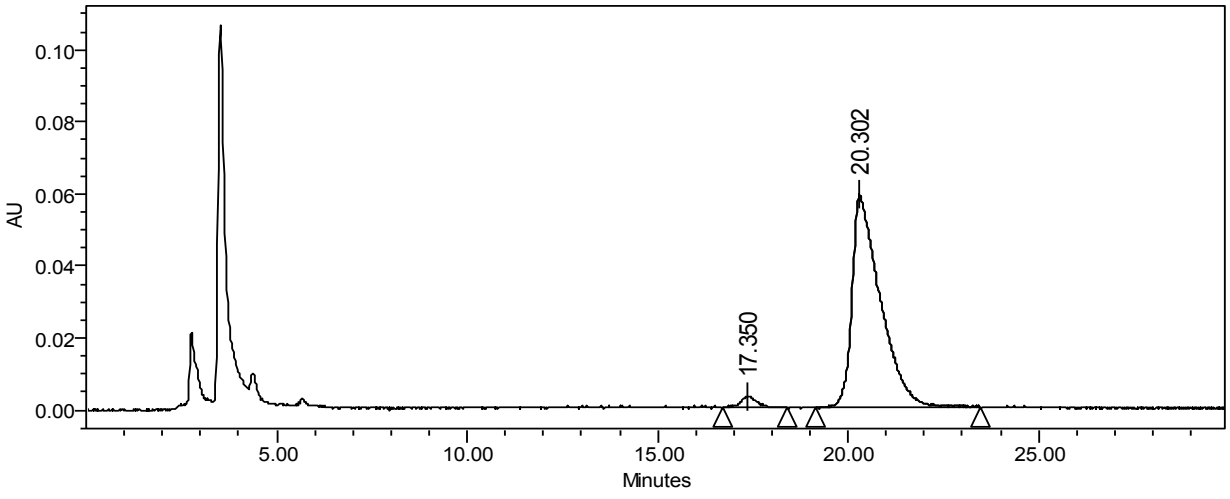
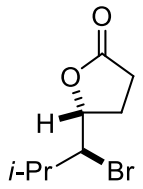


	Name	Retention Time	Area	% Area	Height	Int Type
2		20.302	3023955	97.11	58999	bb
1		17.350	89957	2.89	2986	bb

(**diastero-7c**) OD-H, 0.5% IPA / hexanes, 1.0 mL/min. obs: 210 nm



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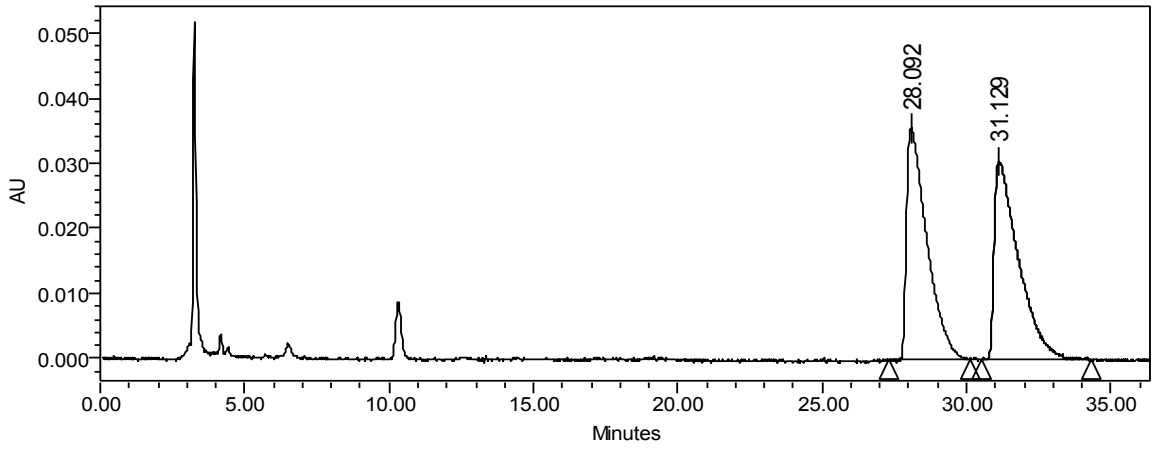

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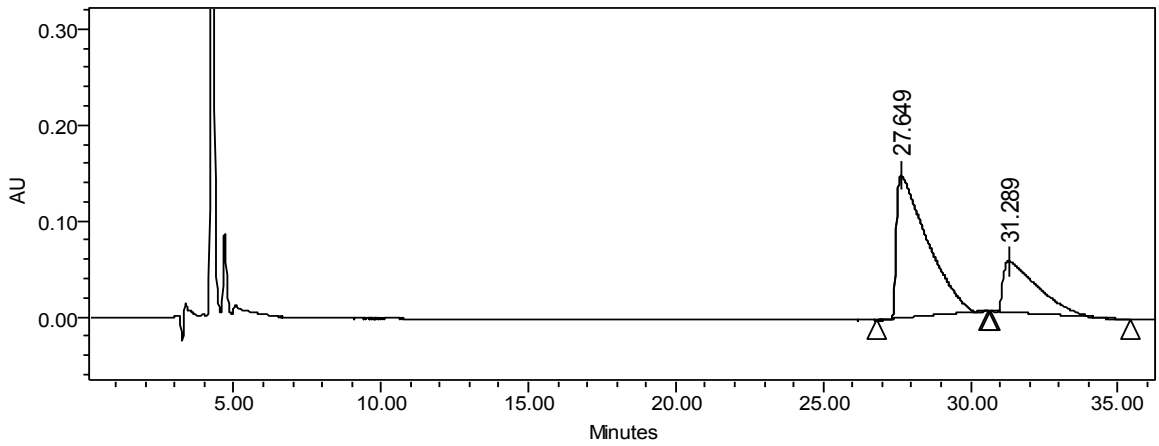
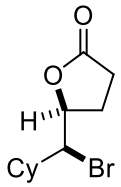


	Name	Retention Time	Area	% Area	Height	Int Type
1		27.649	10784723	71.47	148189	bb
2		31.289	4305392	28.53	52596	bb

(**7d**) Whelk-O1, 20% IPA / hexanes, 1.2 mL/min. obs: 210 nm.



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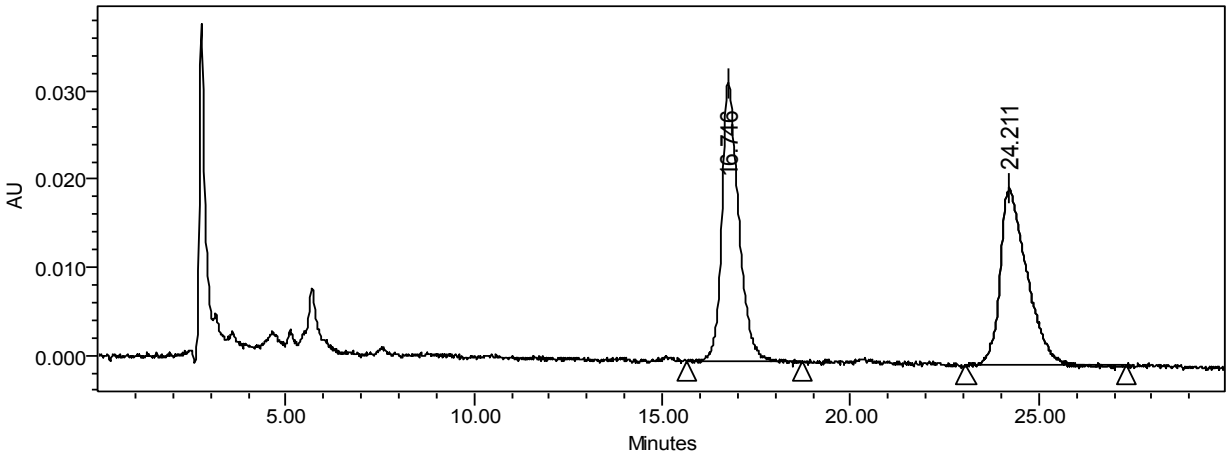

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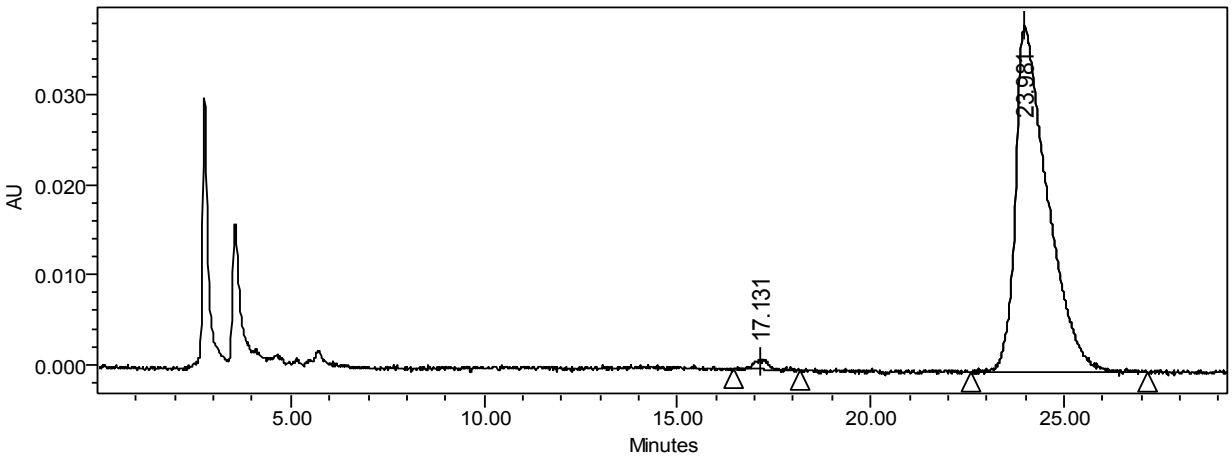
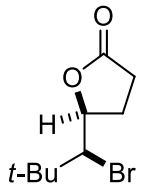


	Name	Retention Time	Area	% Area	Height	Int Type
2		23.981	2222565	98.55	38610	bb
1		17.131	32637	1.45	1032	bb

(**7e**) Whelk-O1, 20% IPA / hexanes, 1.2 mL/min. obs: 210 nm.



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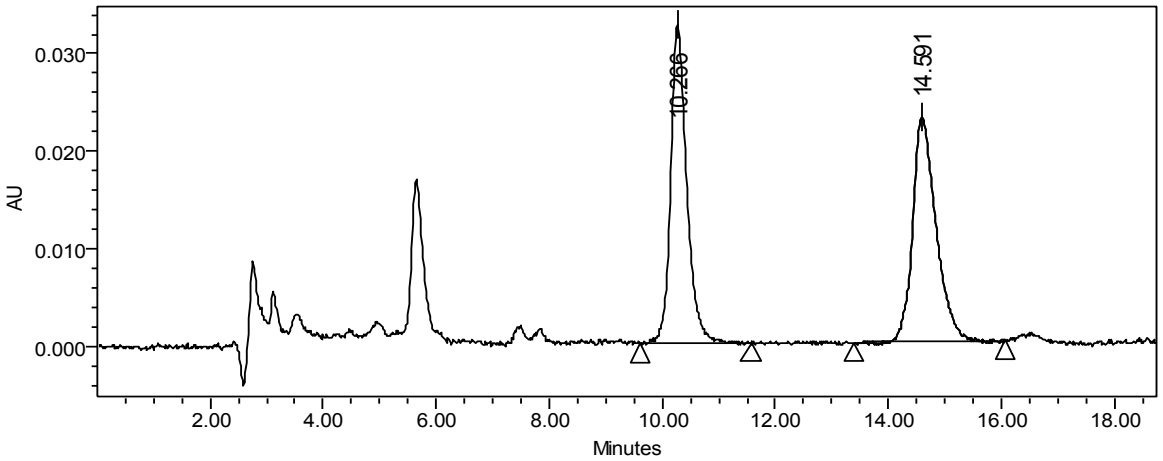

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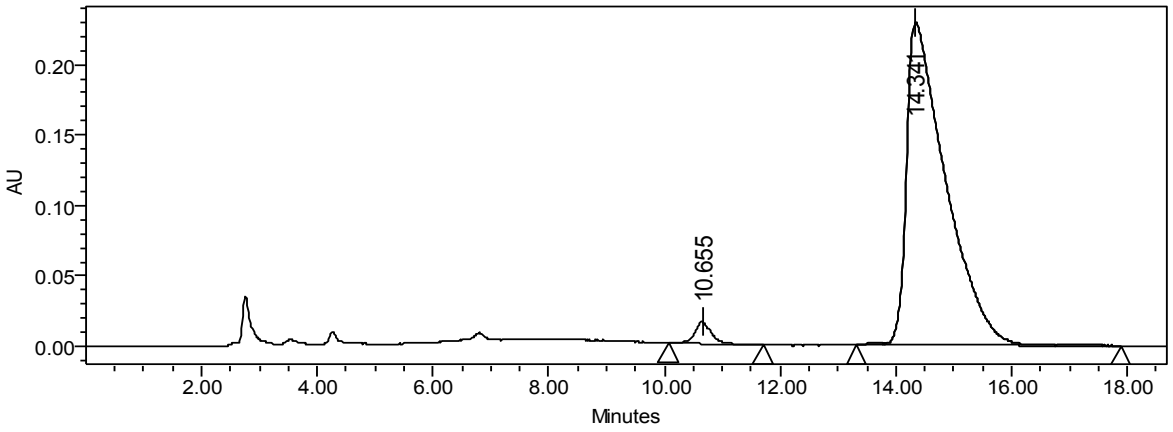
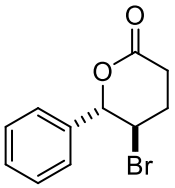


	Name	Retention Time	Area	% Area	Height	Int Type
2		14.341	10731039	97.14	228971	bb
1		10.655	315510	2.86	15609	bb

(**8f**) Whelk-O1, 3% AN / 20% IPA / hexanes, 1.2 mL/min. obs: 210 nm.



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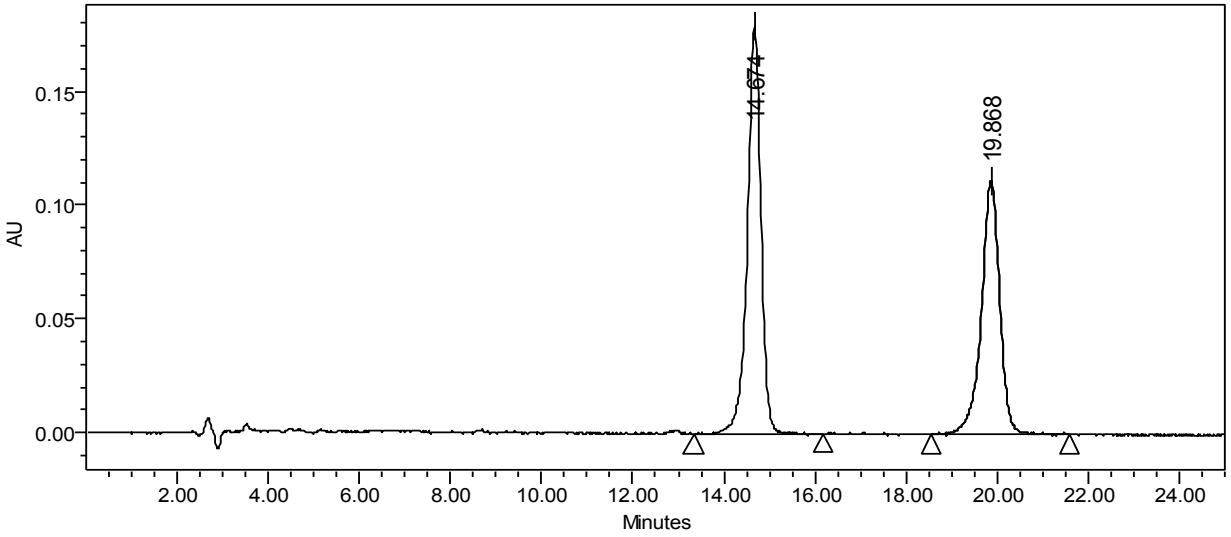

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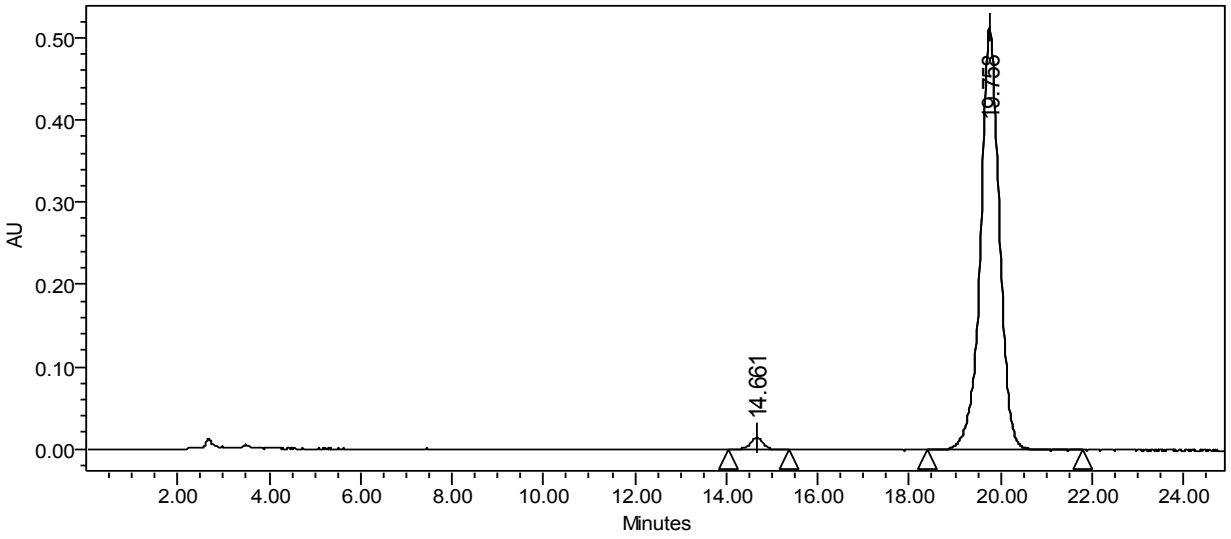
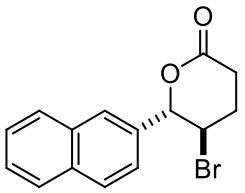


	Name	Retention Time	Area	% Area	Height	Int Type
1		14.661	286479	1.91	14474	bb
2		19.758	14728036	98.09	514521	bb

(**8g**) Whelk-O1, 6% AN / 20% IPA / hexanes, 1.2 mL/min. obs: 225 nm.



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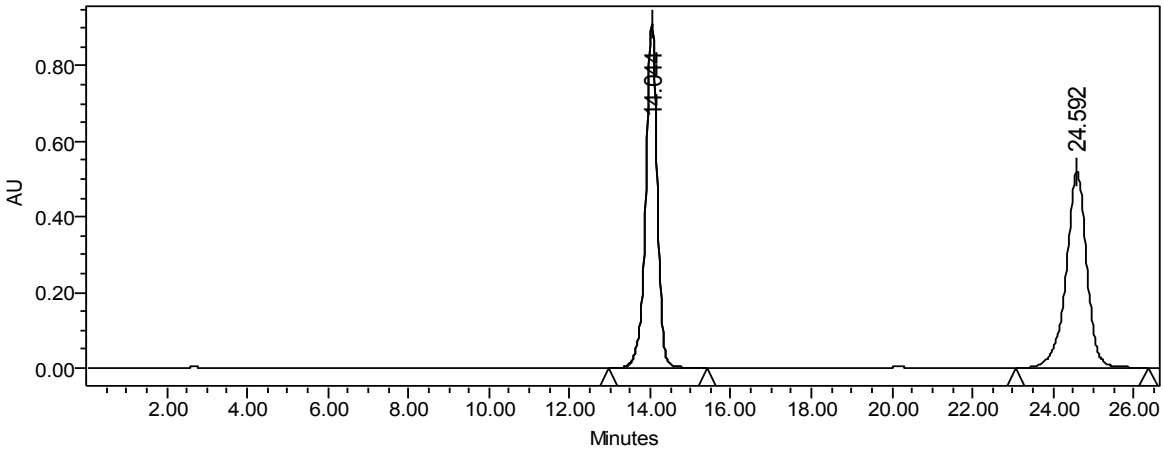

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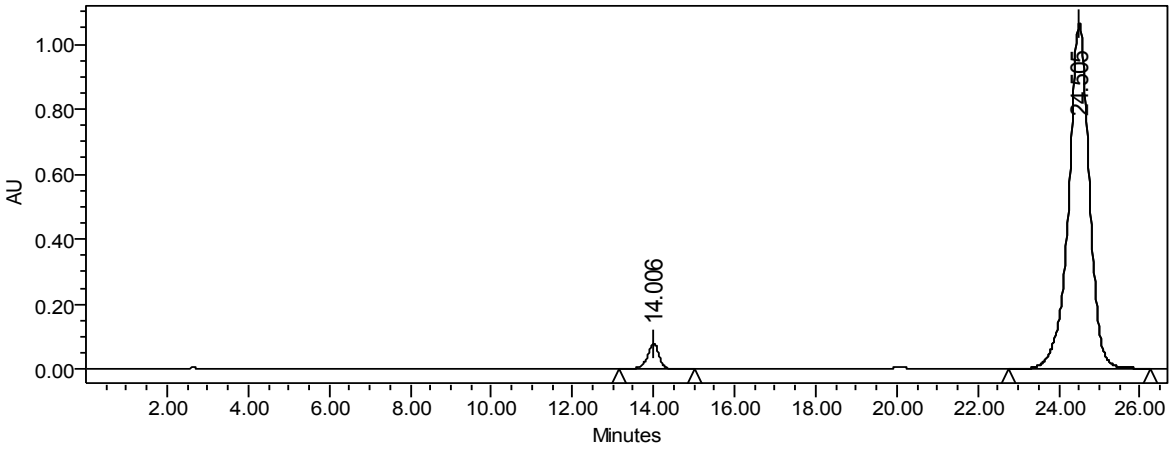
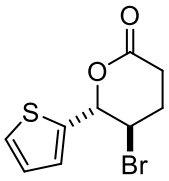


	Name	Retention Time	Area	% Area	Height	Int Type
2		24.505	37745743	96.00	1063542	bb
1		14.006	1572523	4.00	78870	bb

(**8h**) Whelk-O1, 3% AN / 20% IPA / hexanes, 1.2 mL/min. obs: 233 nm.



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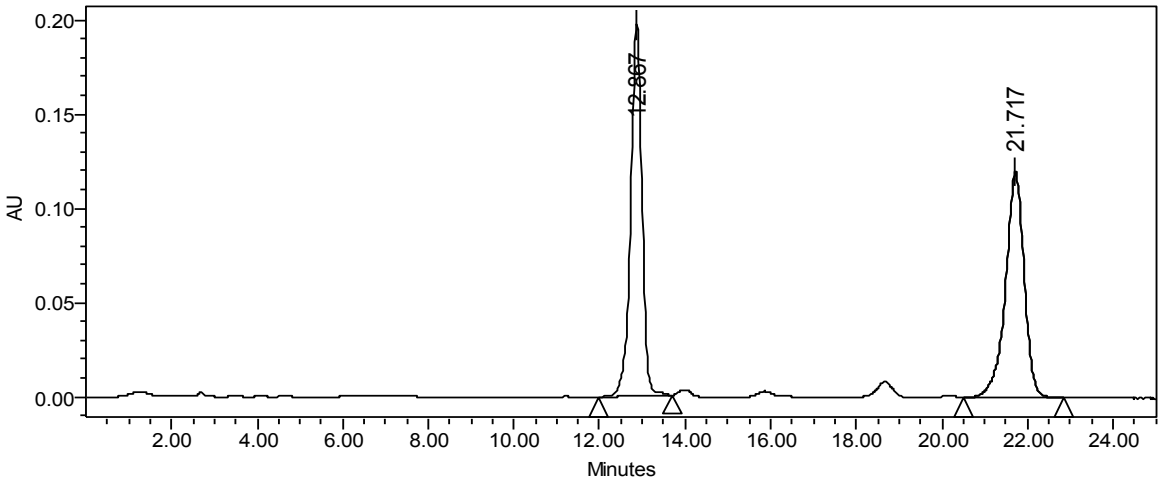

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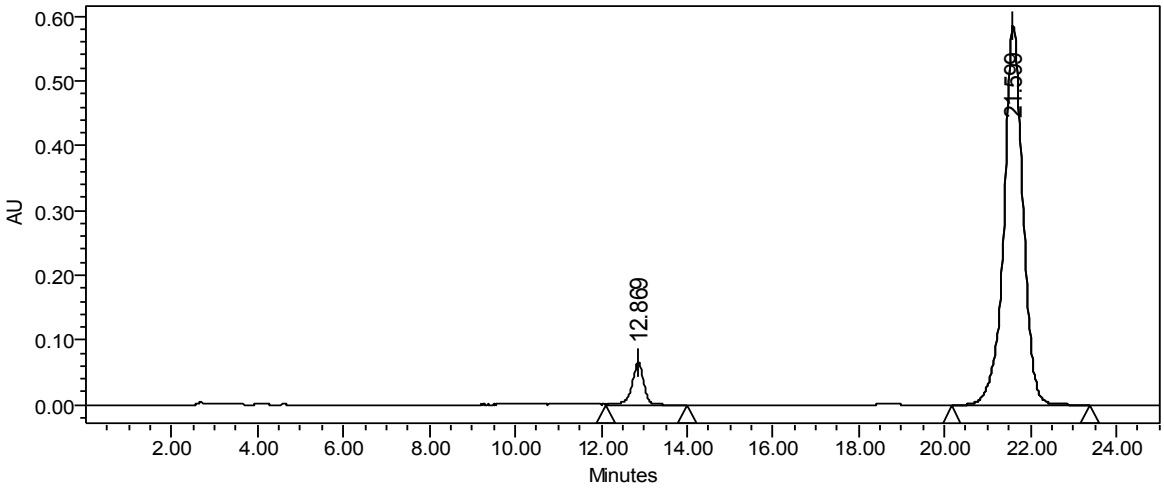
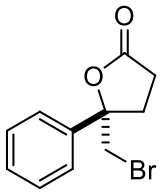


	Name	Retention Time	Area	% Area	Height	Int Type
2		21.599	18069182	93.83	586236	bb
1		12.869	1188463	6.17	64640	bb

(**10a**) Whelk-O1, 20% IPA / hexanes, 1.2 mL/min. obs: 210 nm.



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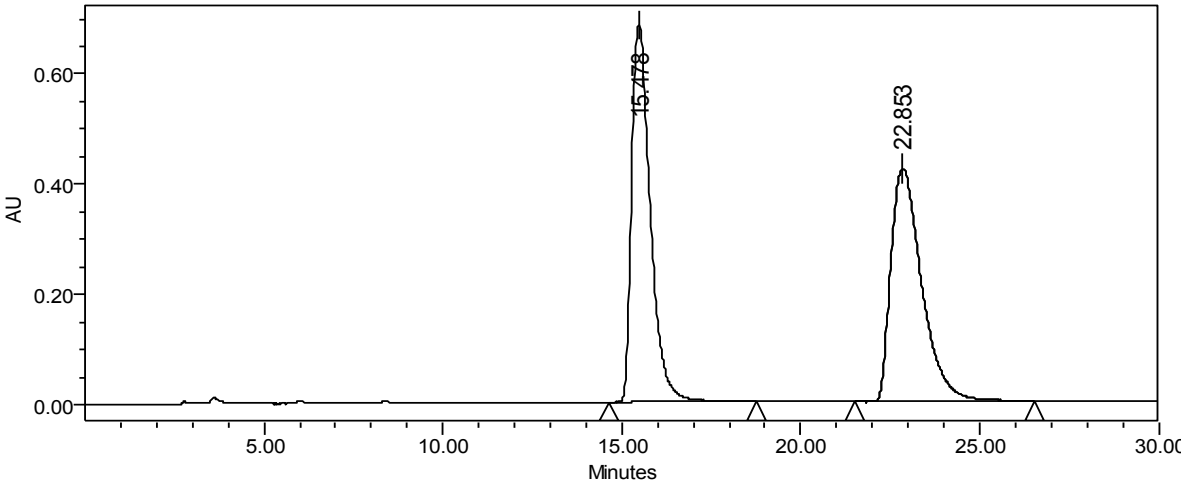

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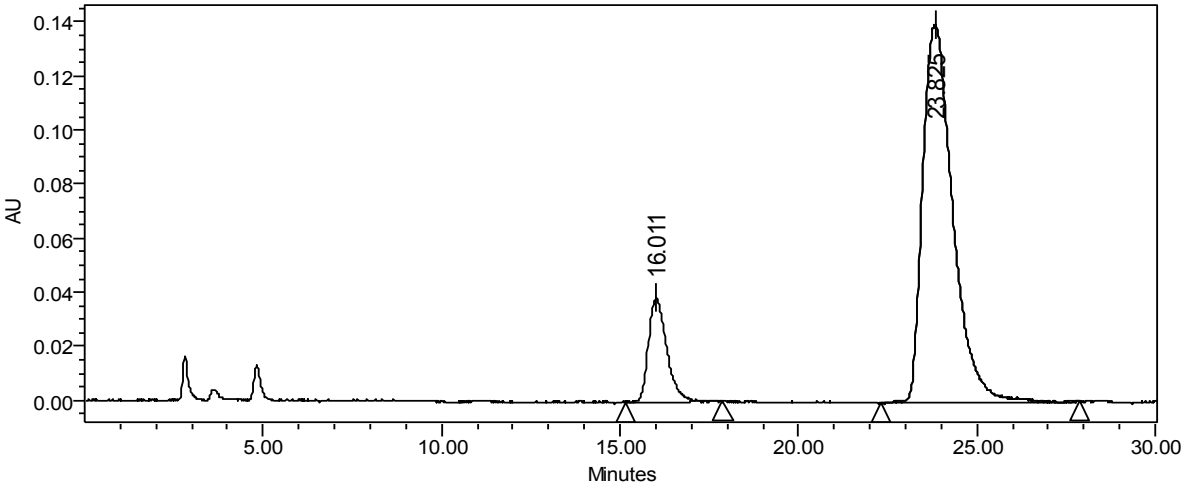
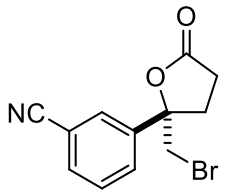


	Name	Retention Time	Area	% Area	Height	Int Type
2		23.825	8223001	85.62	139915	bb
1		16.011	1381414	14.38	38540	bb

(**10b**) Whelk-O1, 6% AN / 20% IPA / hexanes, 1.2 mL/min. obs: 225 nm.



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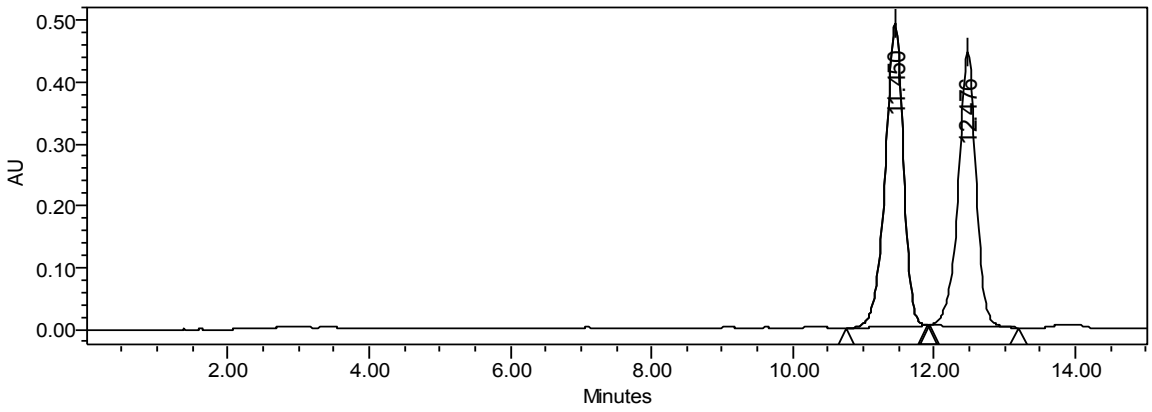

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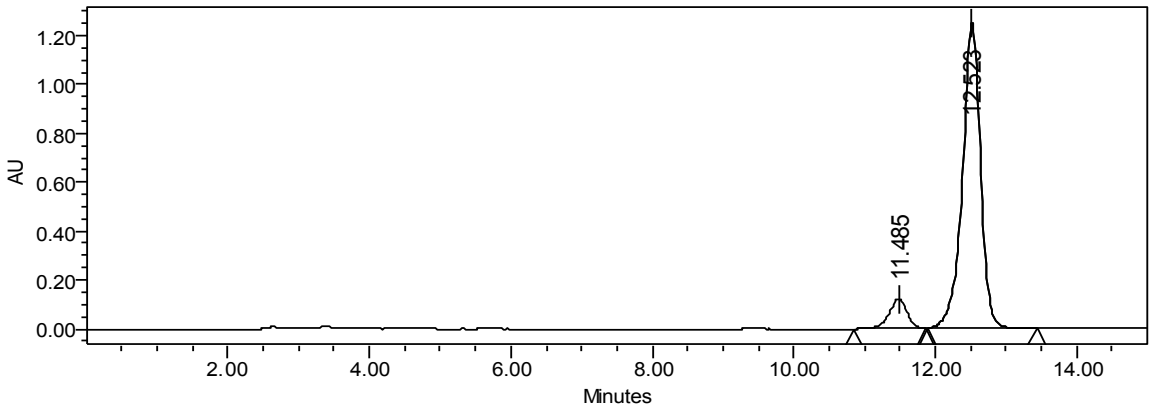
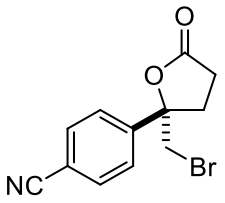


	Name	Retention Time	Area	% Area	Height	Int Type
2		12.523	22402229	91.42	1253425	bb
1		11.485	2102403	8.58	120657	bb

(**10c**) Whelk-O1, 6% AN / 20% IPA / hexanes, 1.2 mL/min. obs: 230 nm



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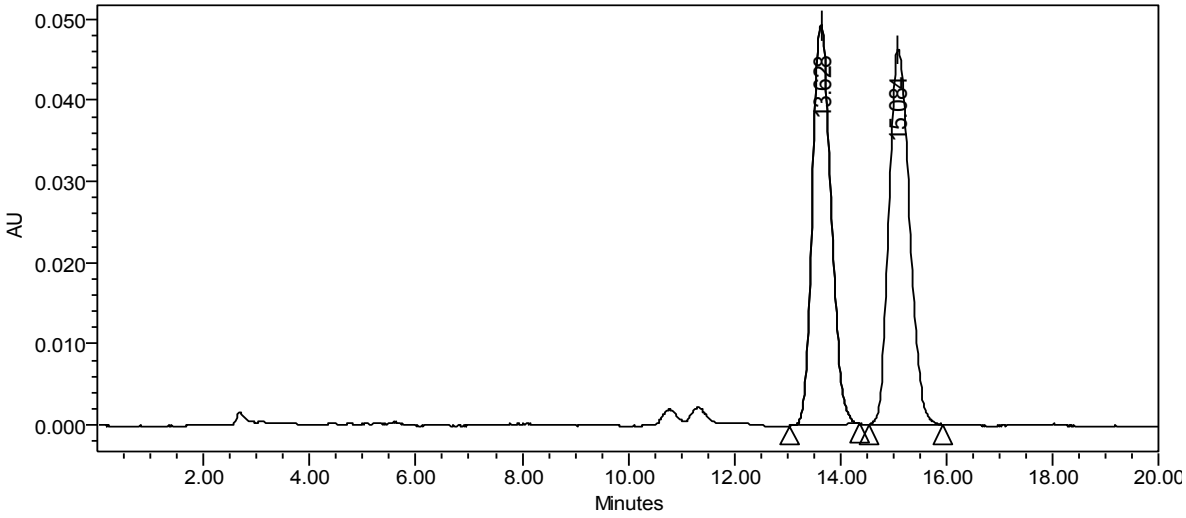

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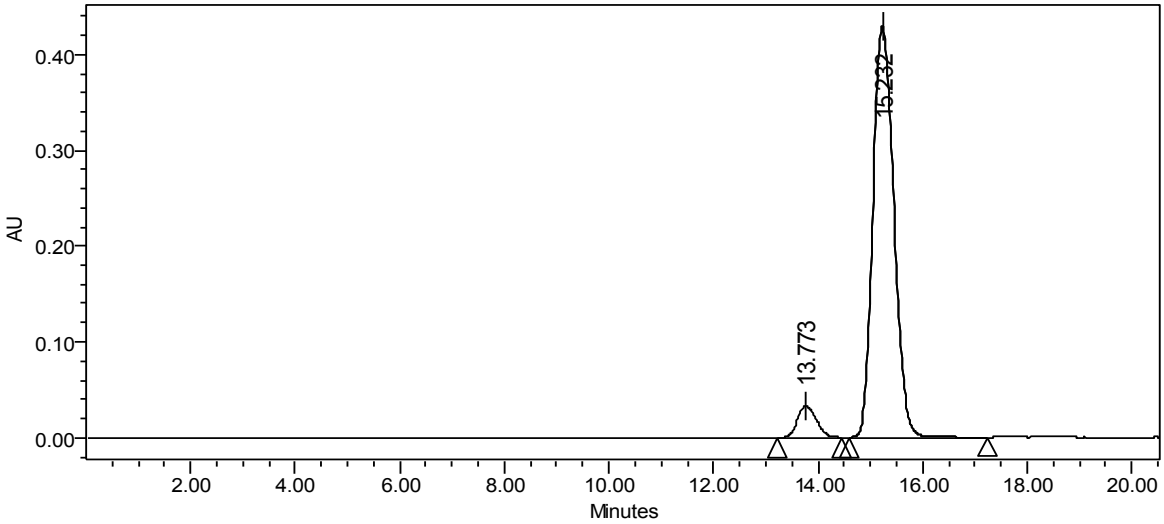
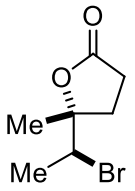


	Name	Retention Time	Area	% Area	Height	Int Type
2		15.232	11966883	93.51	430130	bb
1		13.773	830374	6.49	32989	bb

(**10d**) Whelk-O1, 20% IPA / hexanes, 1.2 mL/min. obs: 210 nm.



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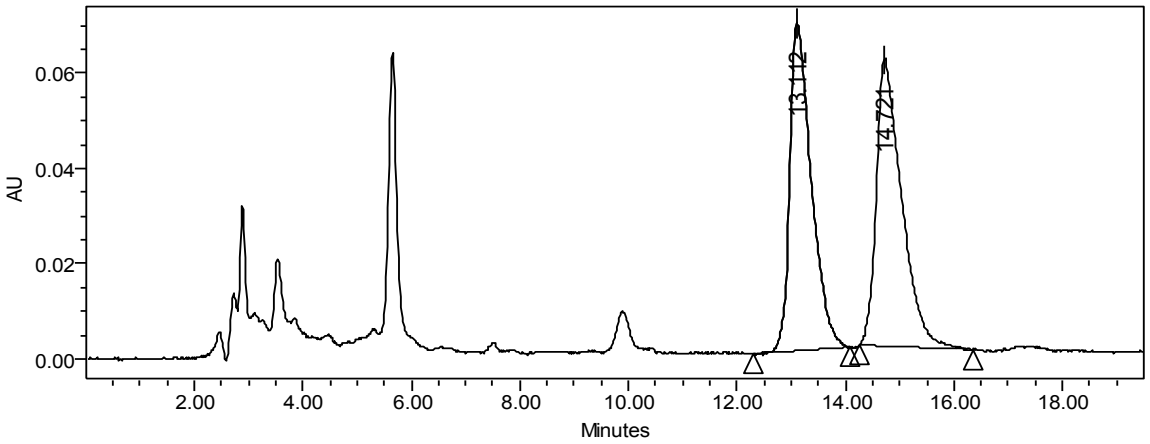

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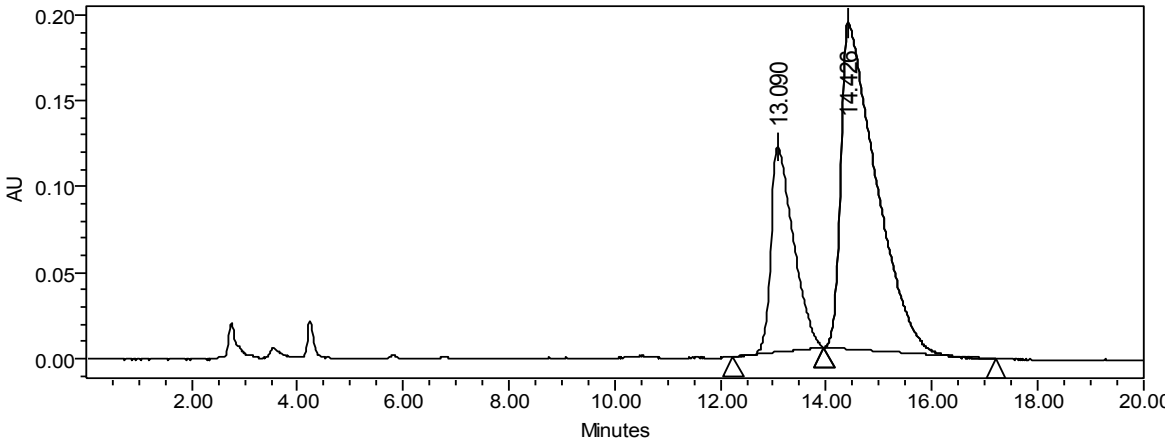
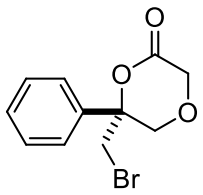


	Name	Retention Time	Area	% Area	Height	Int Type
2		14.426	8803929	71.36	189744	bb
1		13.090	3534186	28.64	119508	bb

(**11e**) Whelk-O1, 20% IPA / hexanes, 1.2 mL/min. obs: 210 nm.



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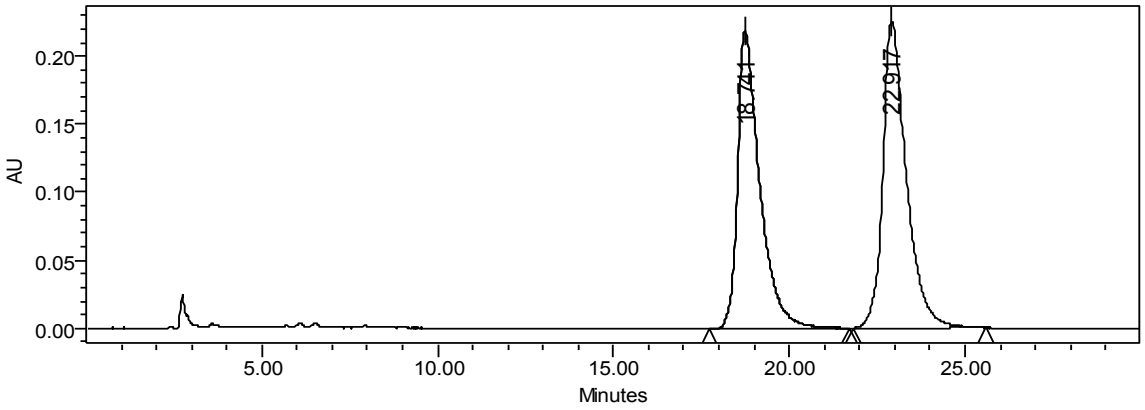

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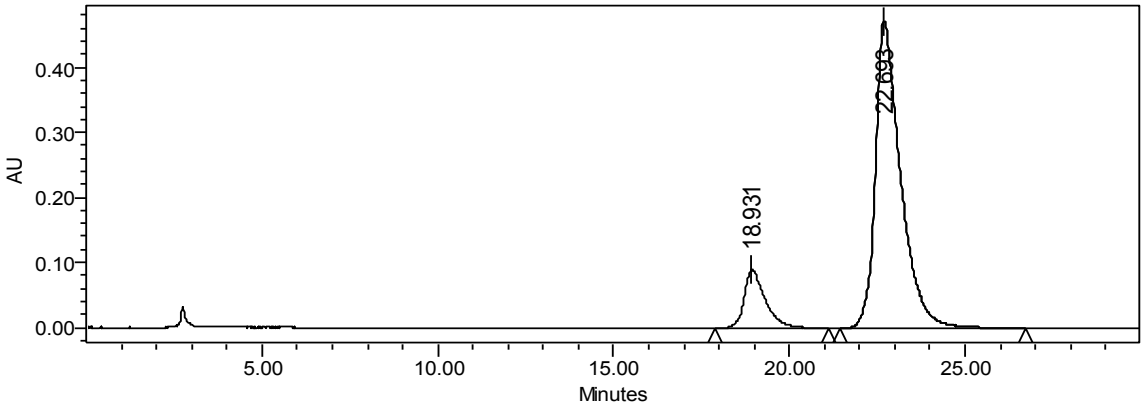
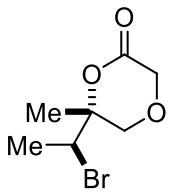
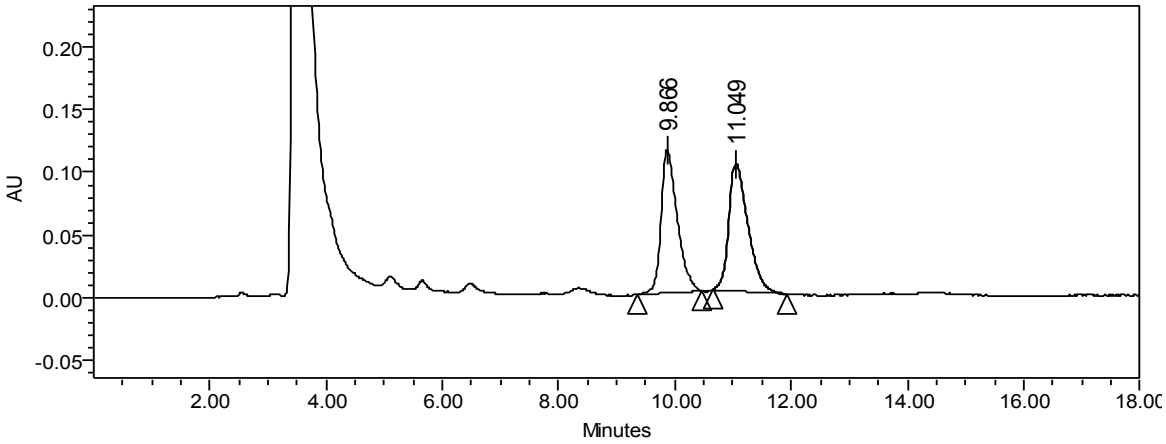


	Name	Retention Time	Area	% Area	Height	Int Type
2		22.693	23627301	86.26	472695	bb
1		18.931	3764526	13.74	90507	bb

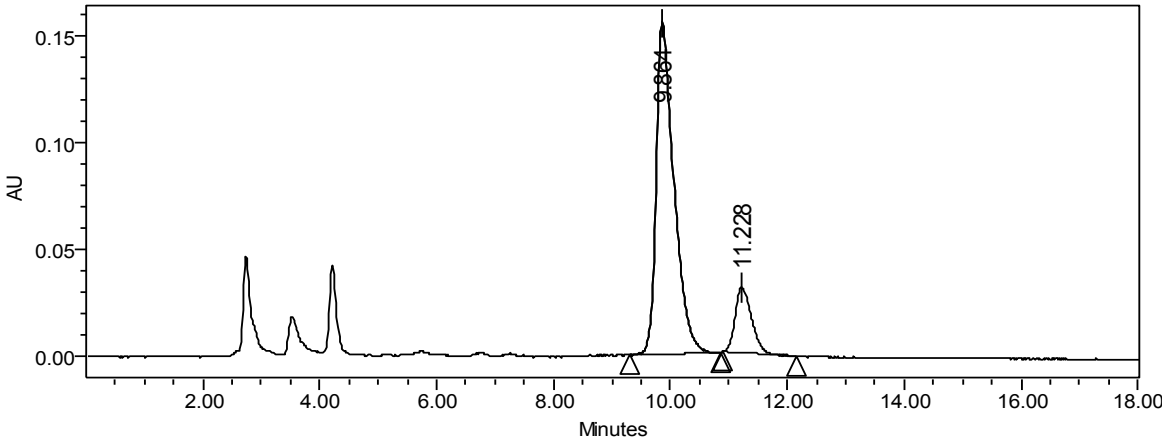
(**11f**) Whelk-O1, 20% IPA / hexanes, 1.2 mL/min. obs: 210 nm.



RAC:

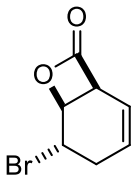


ENT:

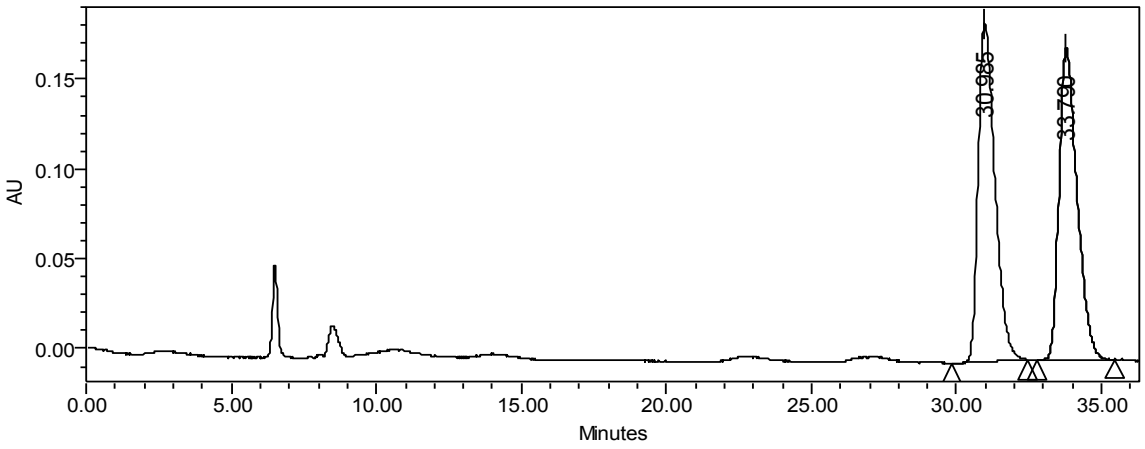


	Name	Retention Time	Area	% Area	Height	Int Type
1		9.864	3288652	84.90	155364	bb
2		11.228	585085	15.10	30571	bb

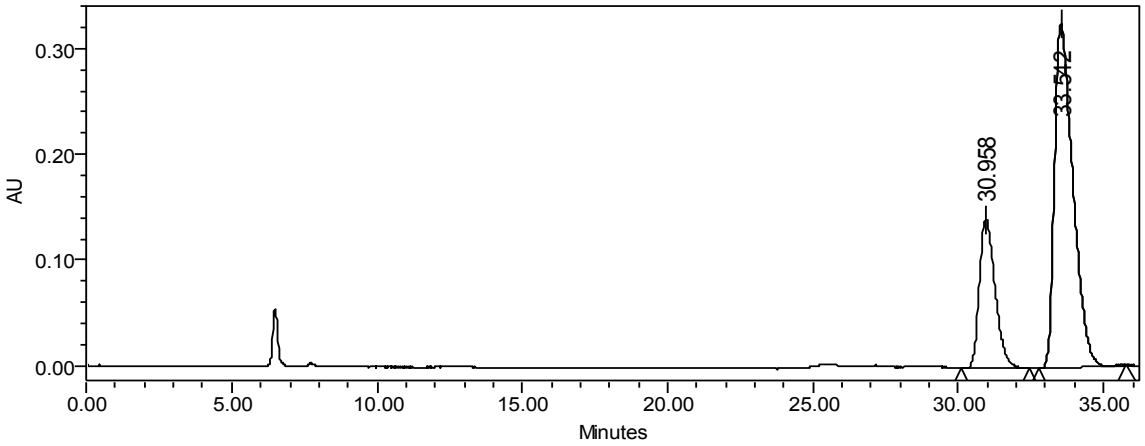
(**13**) OD-H, 1% IPA / hexanes, 1.0 mL/min. obs: 210 nm



RAC:

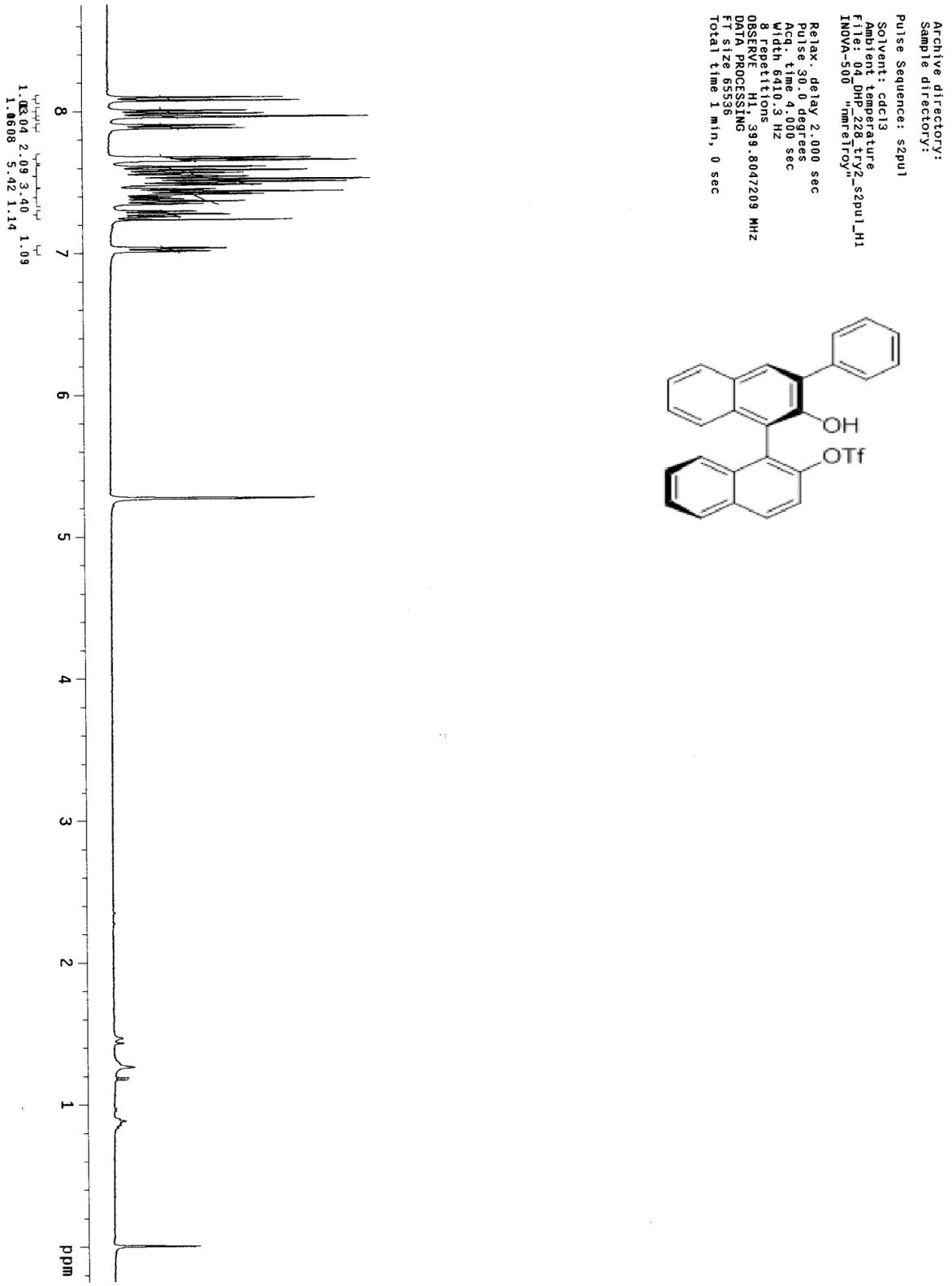


ENT:



	Name	Retention Time	Area	% Area	Height	Int Type
2		33.542	14416667	73.06	326229	bb
1		30.958	5317162	26.94	139889	bb

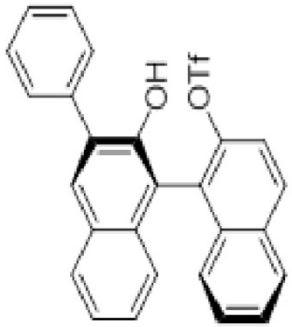
^{1}H *and* ^{13}C *NMRs* (organized by compound reference number)

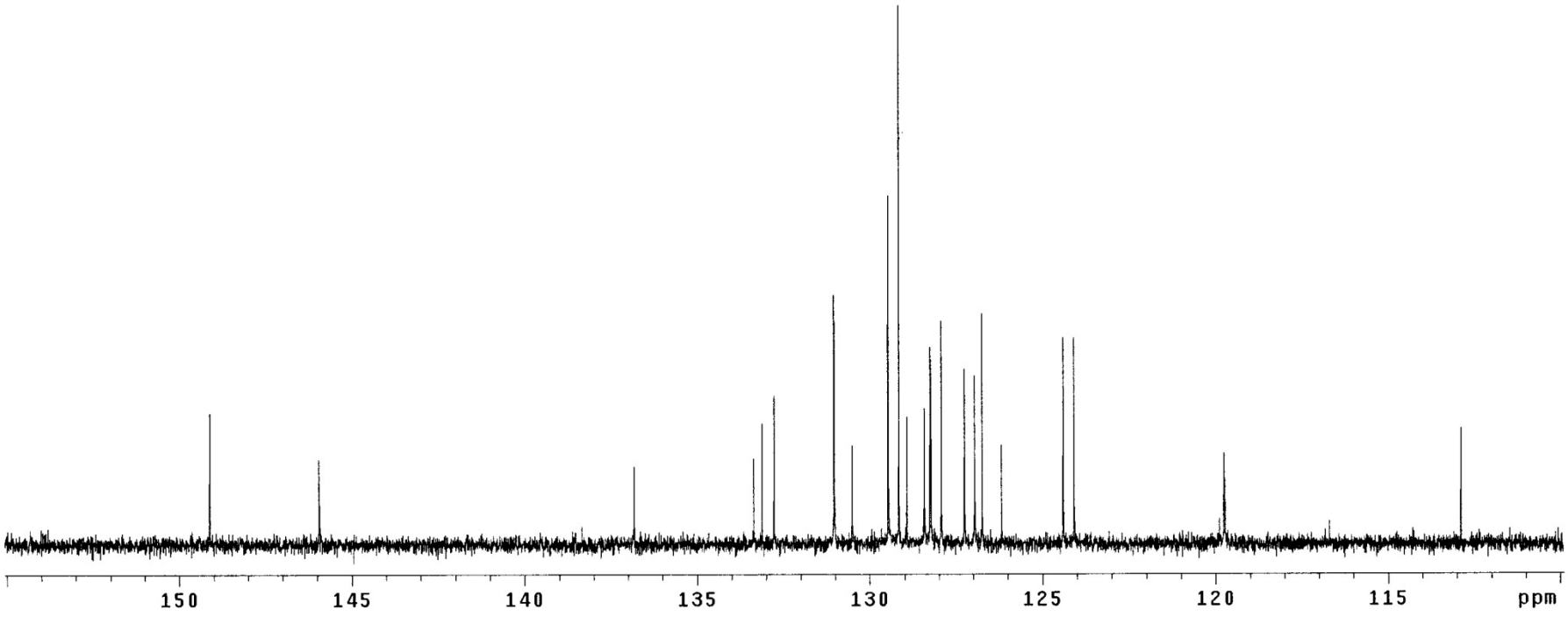


Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: 04_DHP_228_s2pul_C13
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 28 min, 14 sec

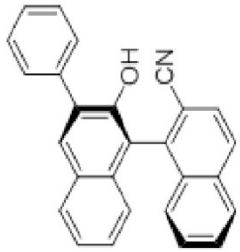


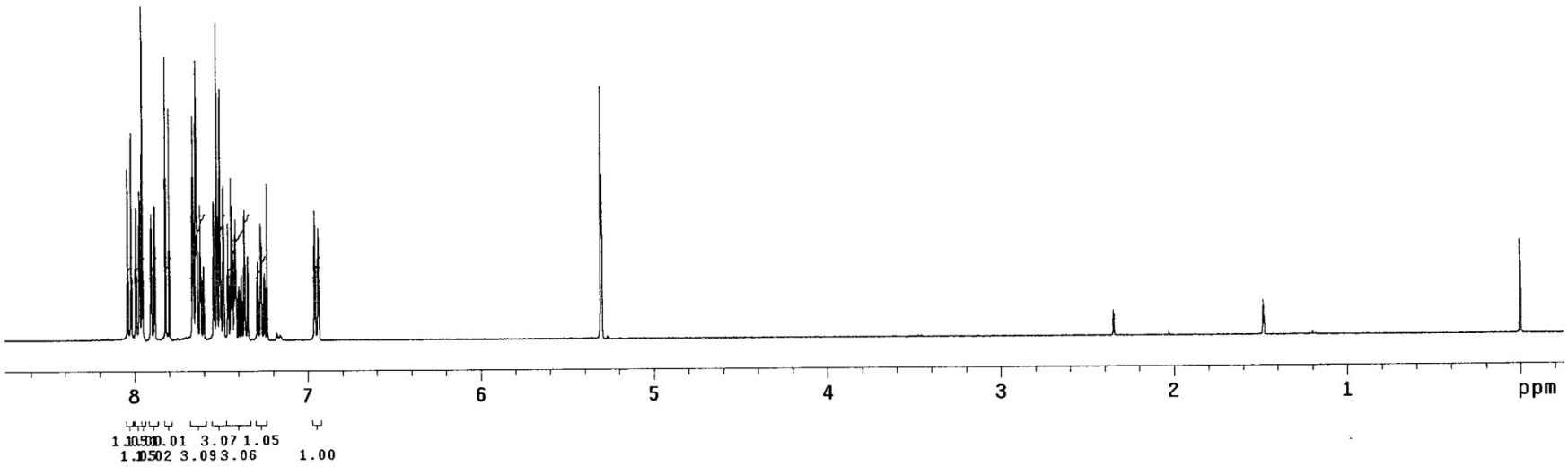


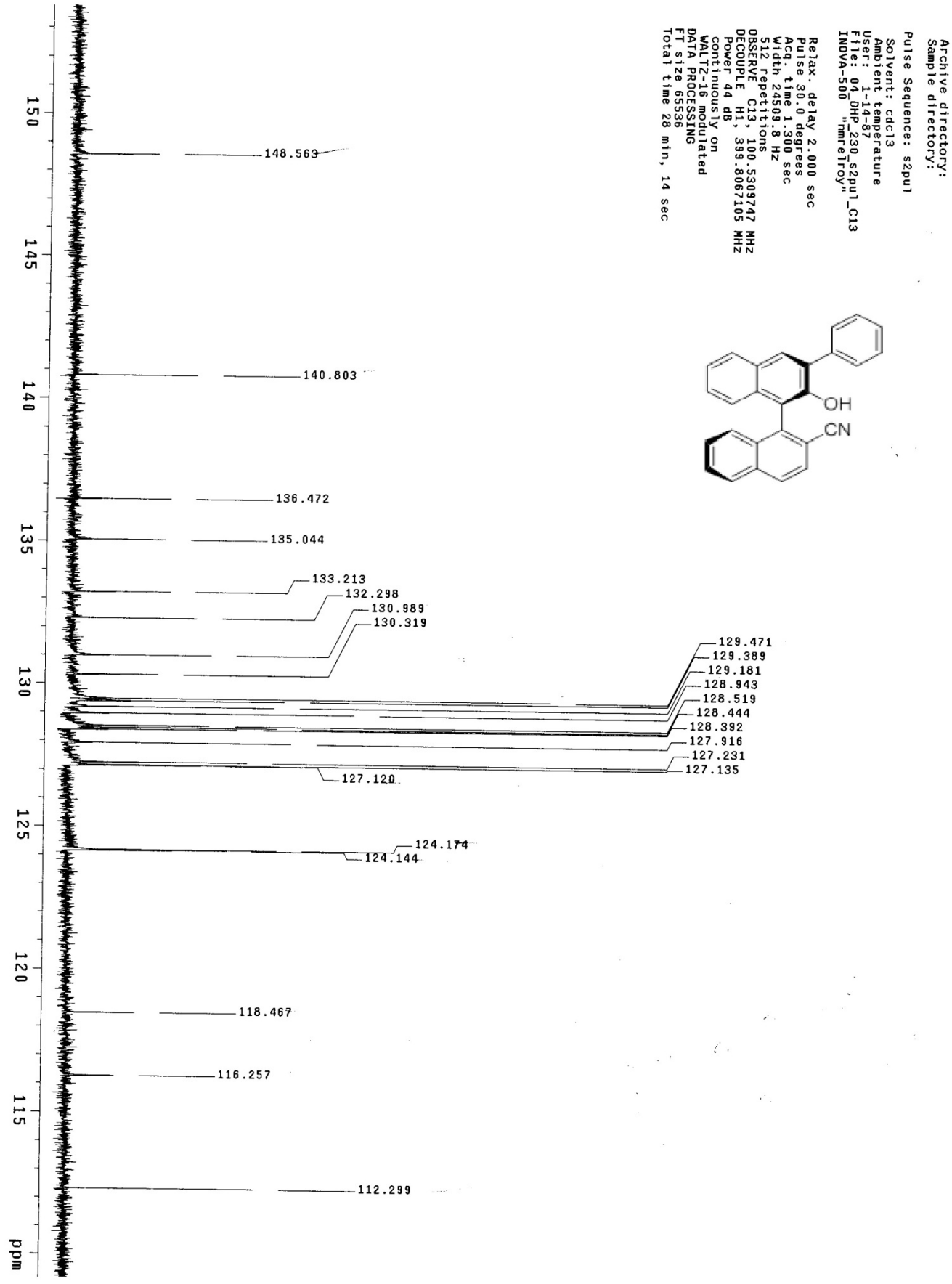
```
 Archive directory:
 Sample directory:

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
File: 04_DHP_230_s2pul_H1
INOVA-500  "nmrelroy"

 Relax. delay 2.000 sec
 Pulse 30.0 degrees
 Acq. time 4.000 sec
 Width 6410.3 Hz
 8 repetitions
OBSERVE    H1, 399.8047211 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec
```



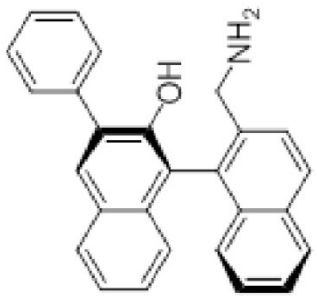


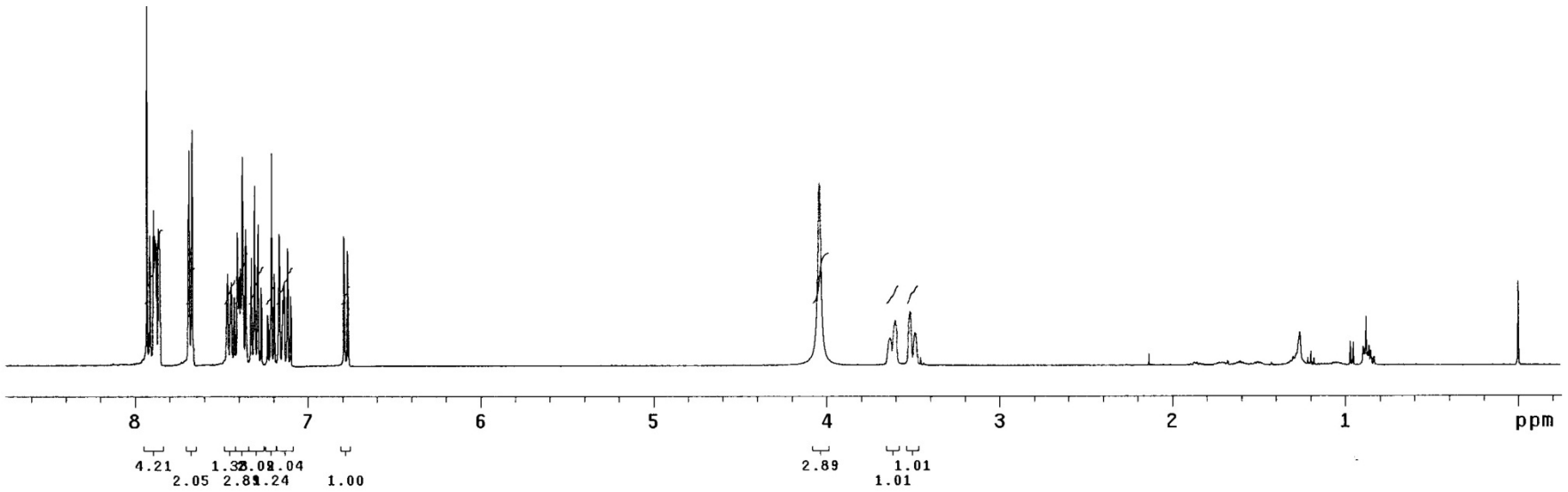

Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: 04_DHP_230_s2pul_C13
INOVA-500 "nmrel1roy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 28 min, 14 sec
OH
CN
148.563
140.803
136.472
135.044
133.213
132.298
130.989
130.319
129.471
129.389
129.181
128.943
128.519
128.444
128.392
127.916
127.231
127.135
127.120
124.174
124.144
118.467
116.257
112.299
150
145
140
135
130
125
120
115
ppm

Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: 04_DHP_232_s2pul_H1
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8047309 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec

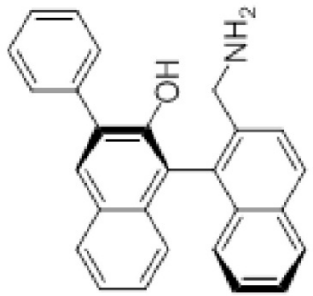


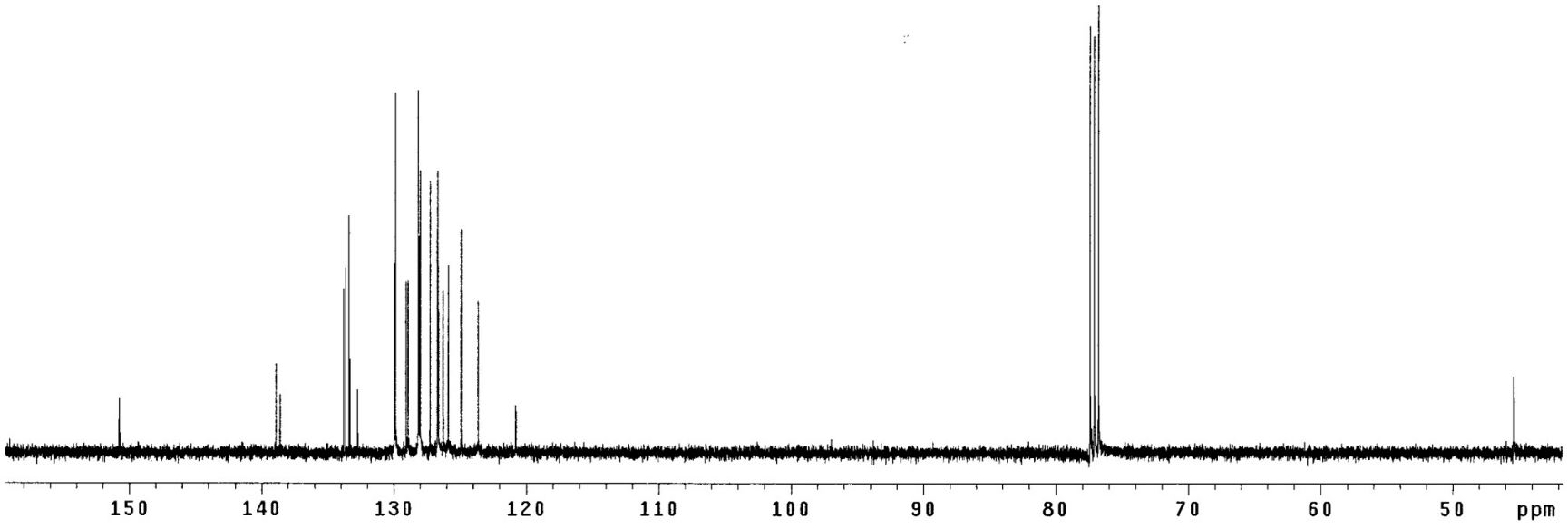


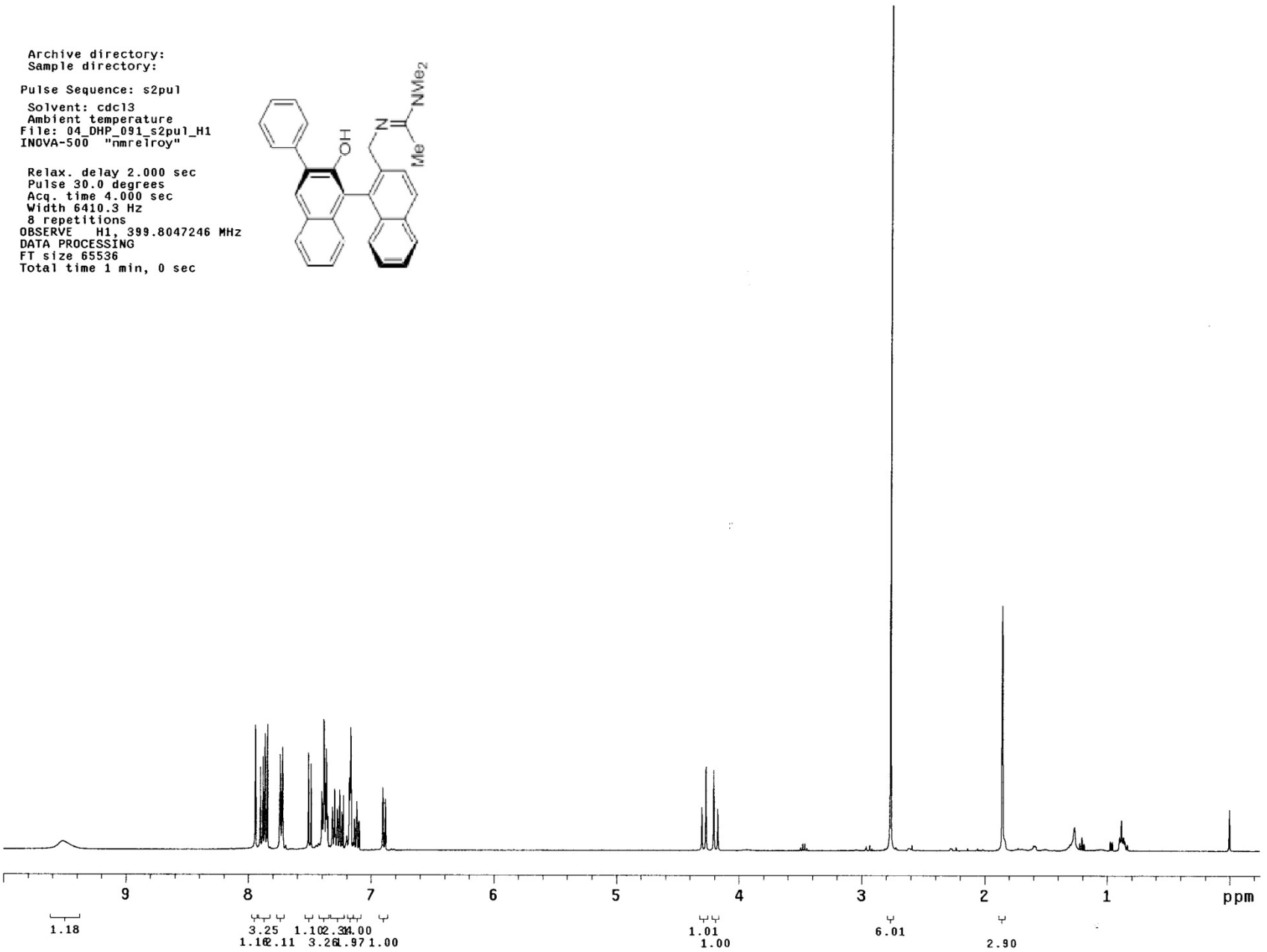
Archive directory:
Sample directory:

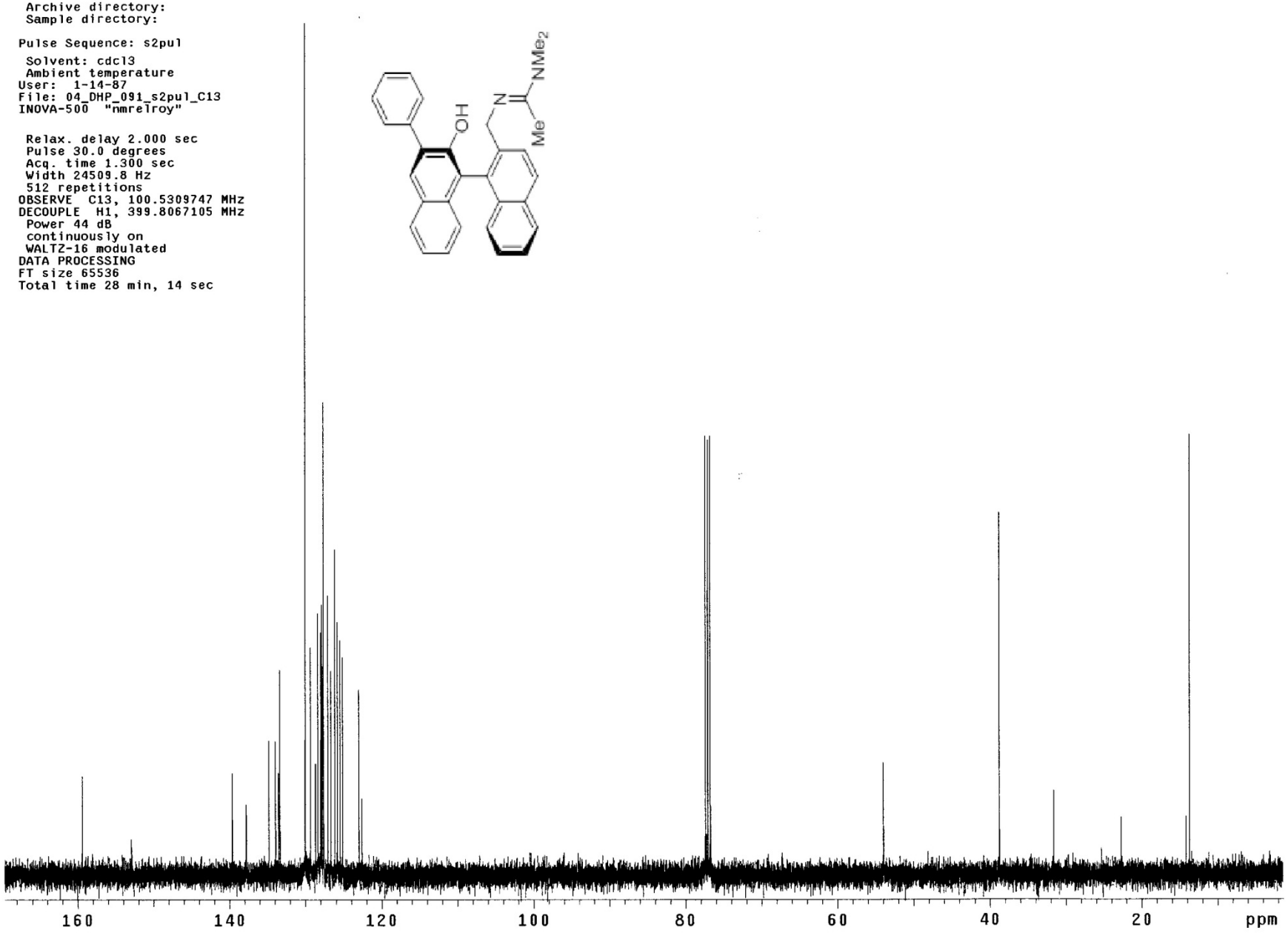
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: 04_DHP_232_s2pul_C13
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 28 min, 14 sec






Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: 04_DHP_091_s2pul_H1
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8047246 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec
OH
N
NMe2
Me
9
8
7
6
5
4
3
2
1
ppm
1.18
3.25
1.10
2.30
4.00
1.01
6.01
2.90
1.00
1.00
1.00

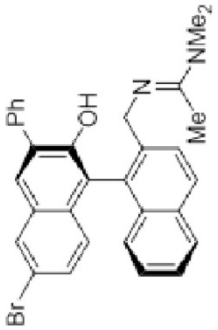

Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: 04_DHP_091_s2pul_C13
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 28 min, 14 sec
OH
N
NMe2
Me
160
140
120
100
80
60
40
20
ppm

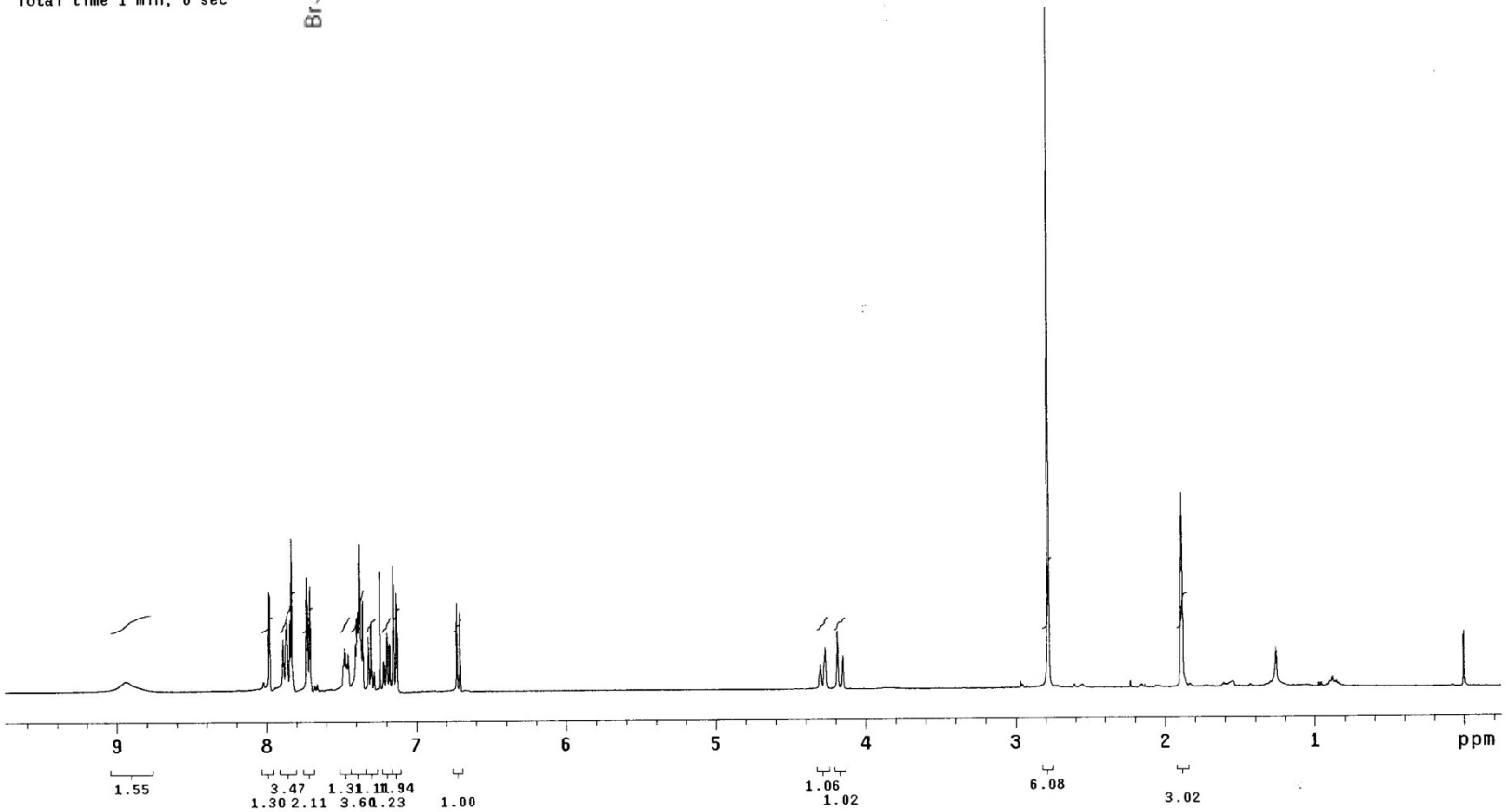
Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
File: 04_DHP_302_s2pul_H1
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8047184 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec

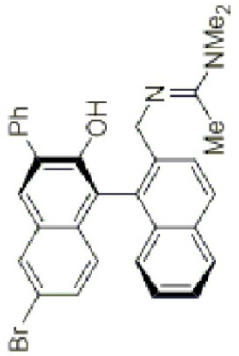


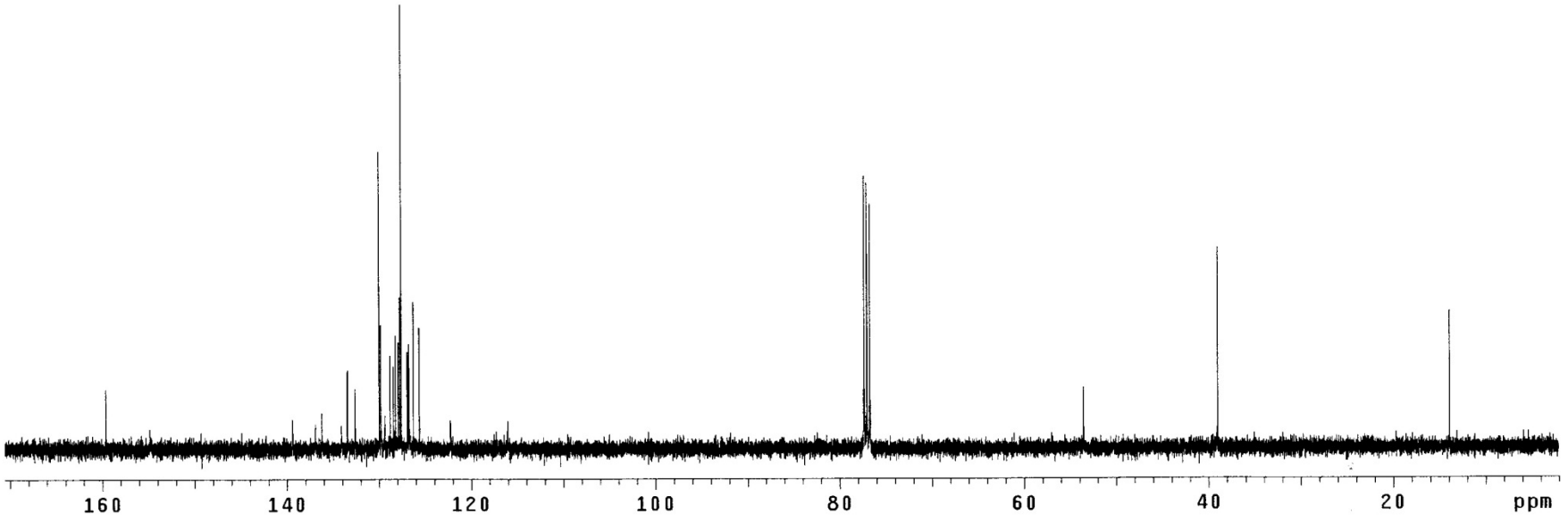


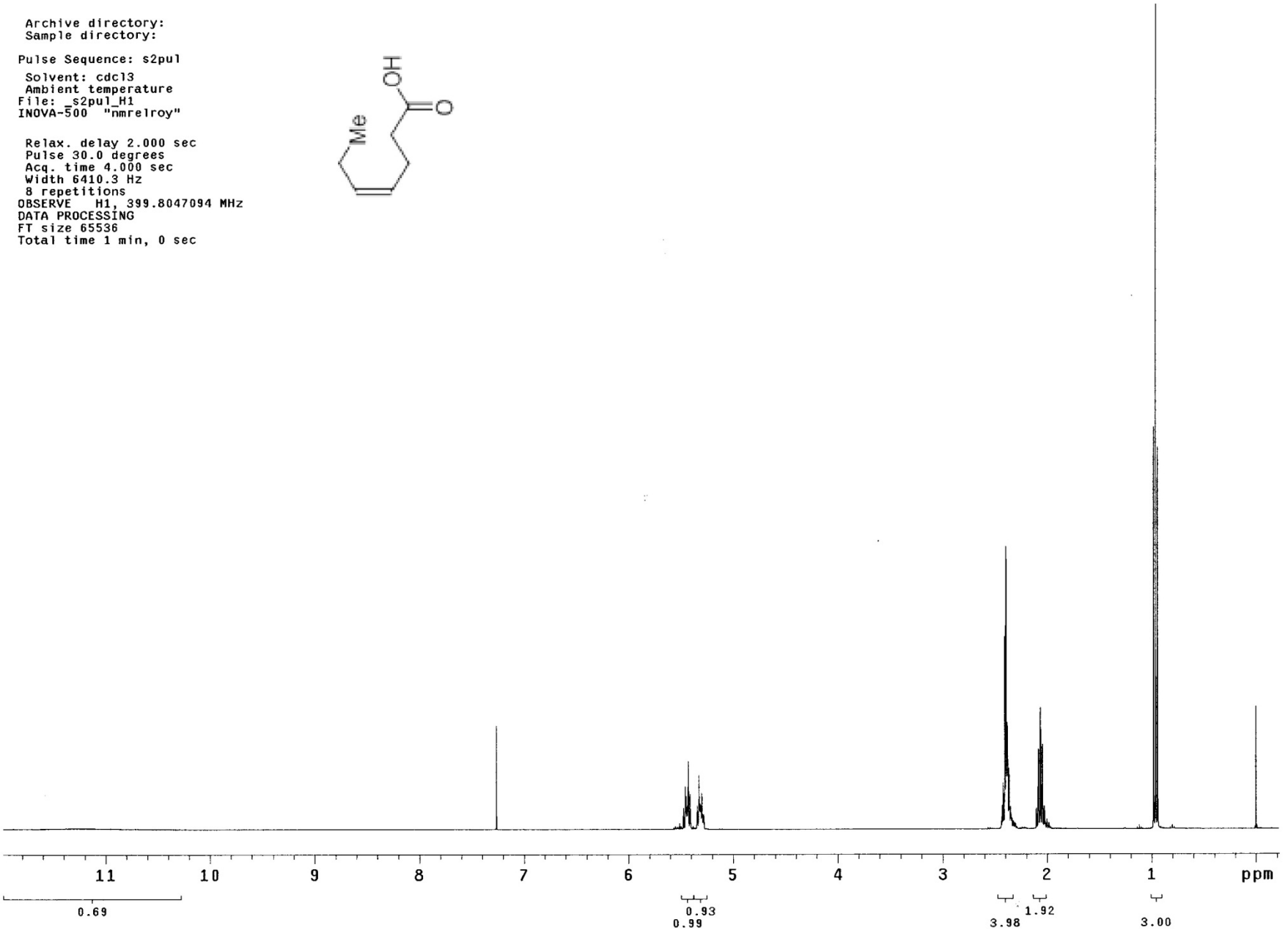
Archive directory:
Sample directory:

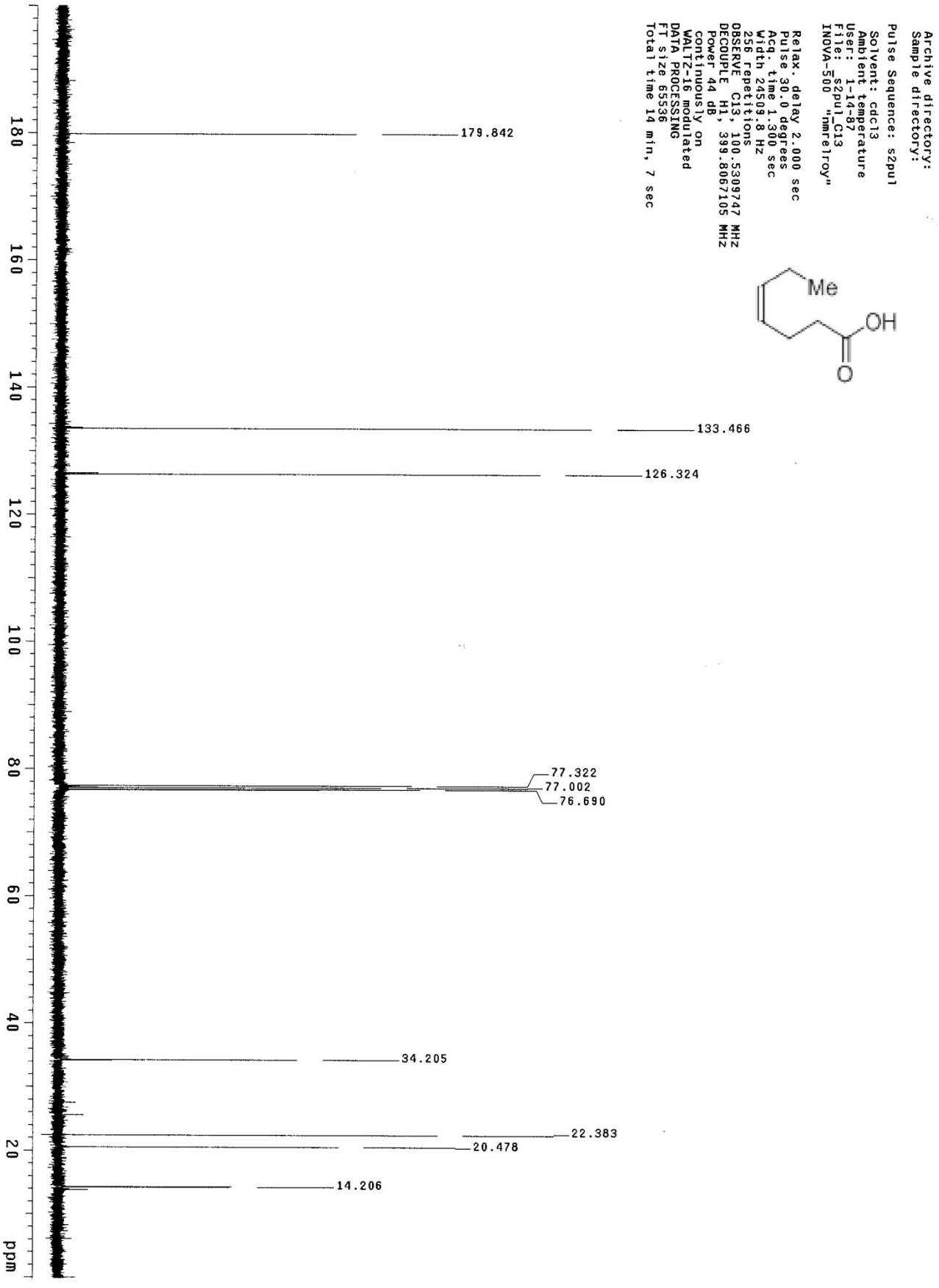
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: 04_DHP_302_s2pul_C13
INOVA-500 "nmrelroy"

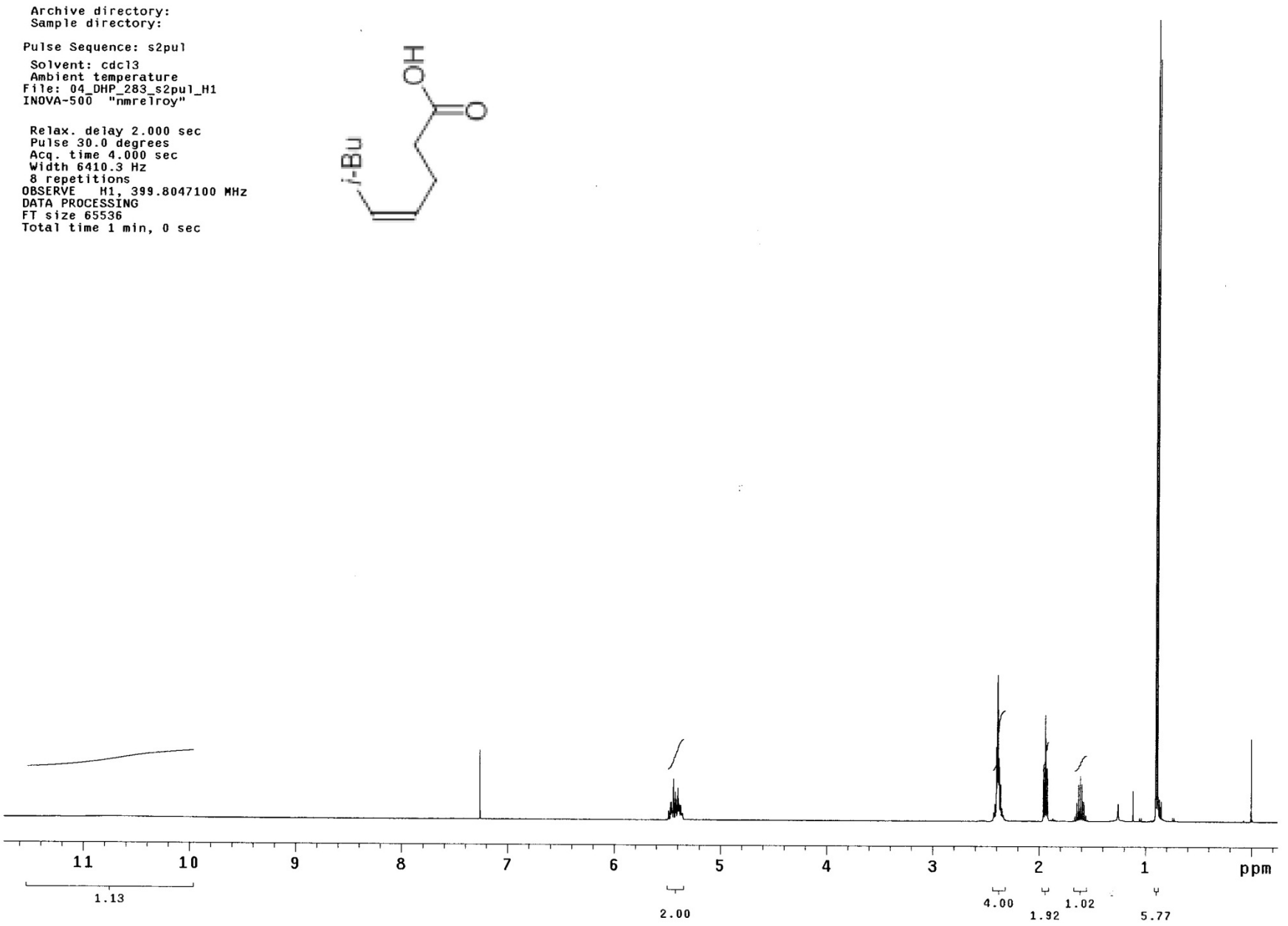
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 28 min, 14 sec

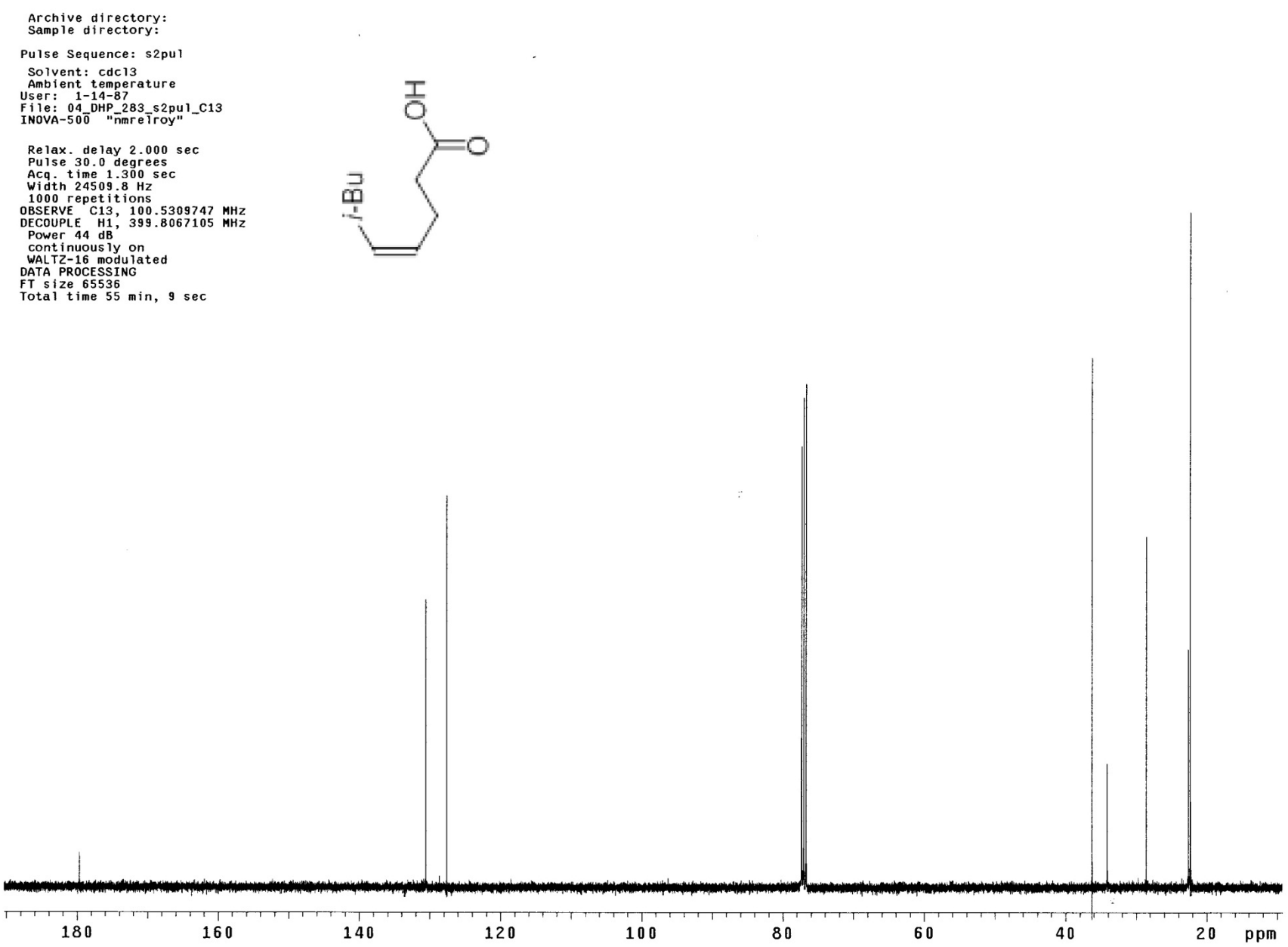





Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: _s2pul_H1
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8047094 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec
Me
OH
O
11
10
9
8
7
6
5
4
3
2
1
ppm
0.69
0.99
0.93
3.98
1.92
3.00


Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: s2pul_C13
INOVA-500 "nmr1roy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
256 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 14 min, 7 sec
Me
OH
O
179.842
133.466
126.324
77.322
77.002
76.690
34.205
22.383
20.478
14.206
180
160
140
120
100
80
60
40
20
ppm

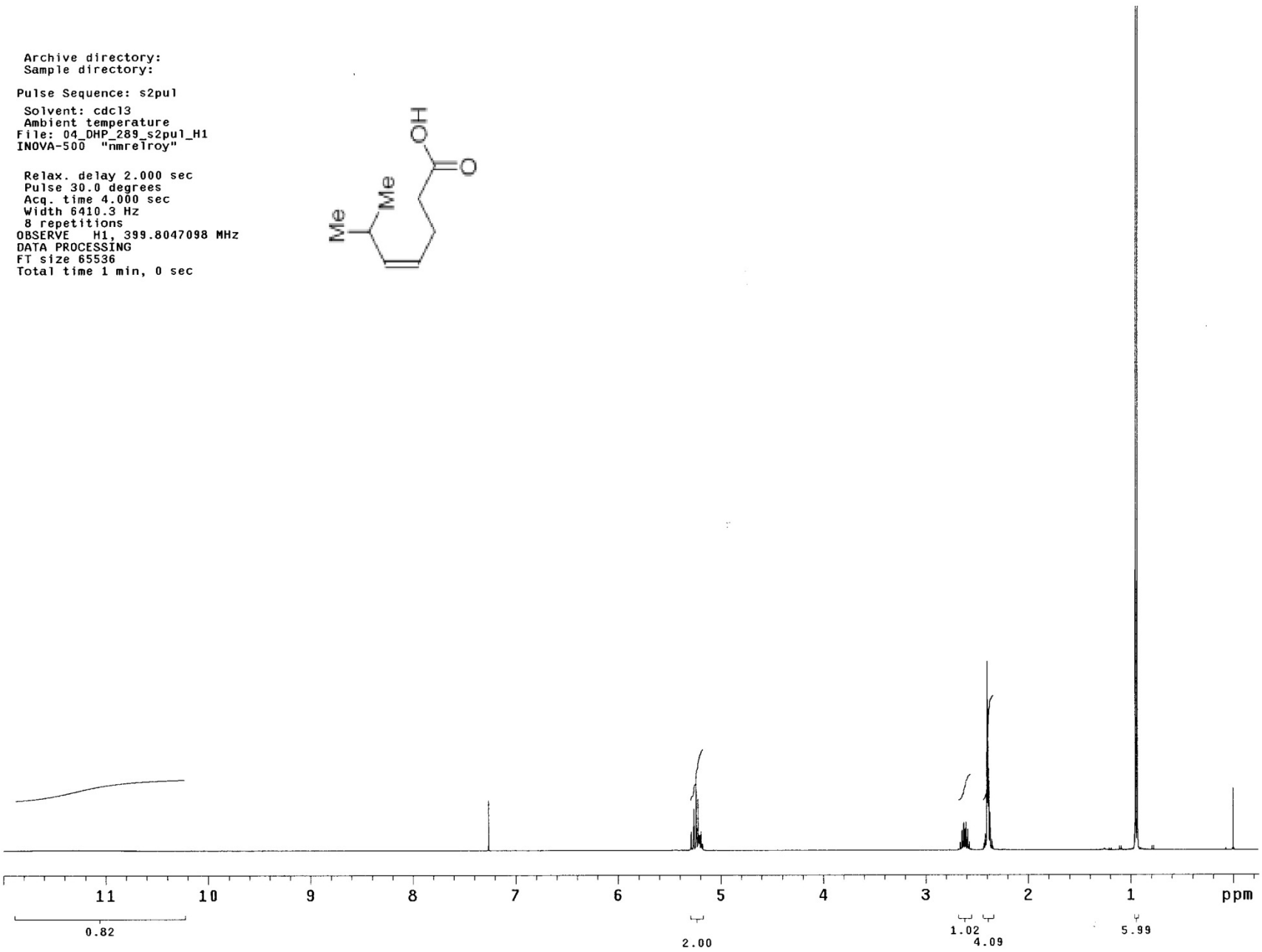

Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: 04_DHP_283_s2pul_H1
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8047100 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec
i-Bu
OH
O
11
10
9
8
7
6
5
4
3
2
1
ppm
1.13
2.00
4.00
1.92
1.02
5.77


Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: 04_DHP_283_s2pul_C13
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
1000 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 55 min, 9 sec
i-Bu
OH
O
180
160
140
120
100
80
60
40
20
ppm

Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: 04_DHP_289_s2pul_H1
INOVA-500 "nmrelroy"

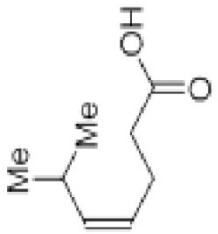
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8047098 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec

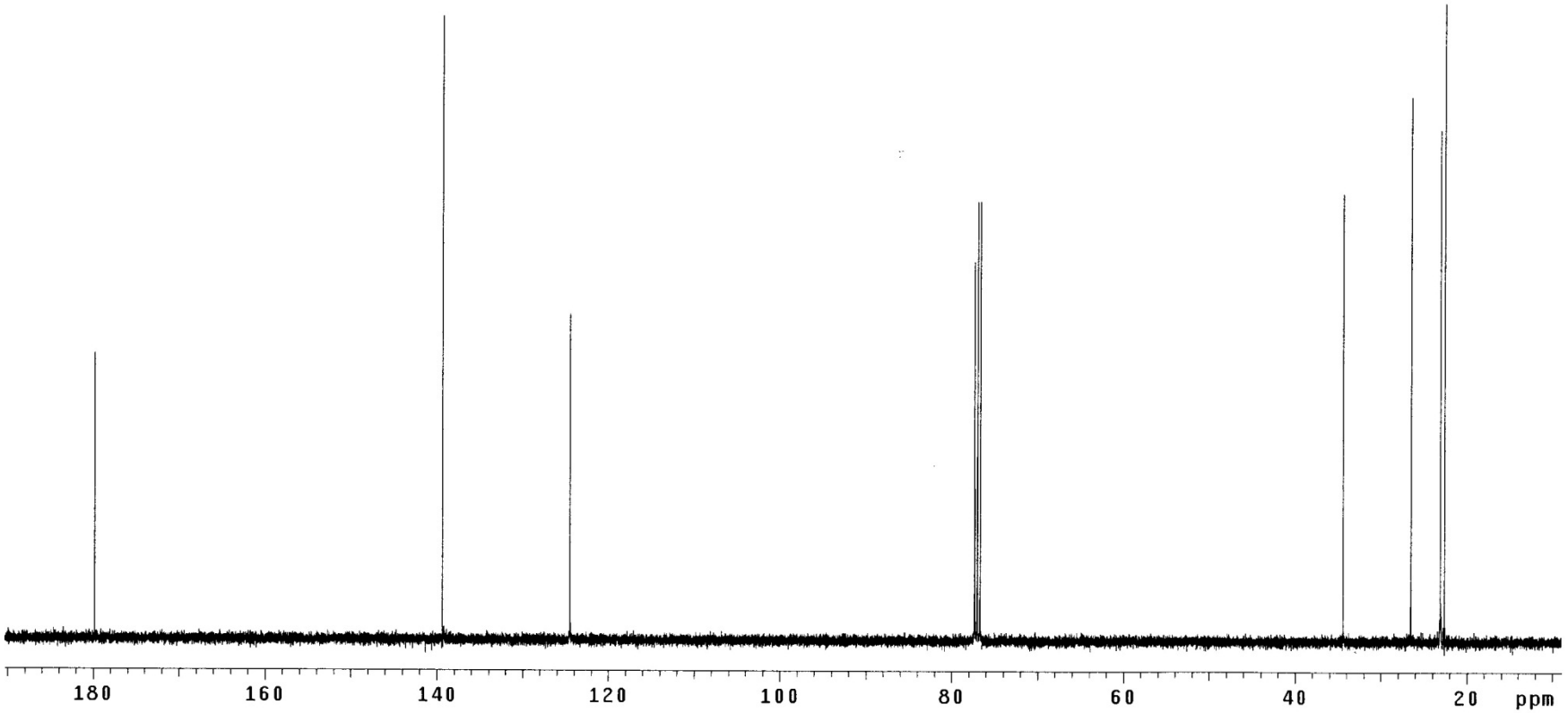


Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: 04_DHP_289_s2pul_C13
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 28 min, 14 sec

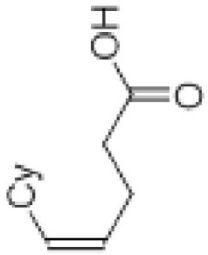


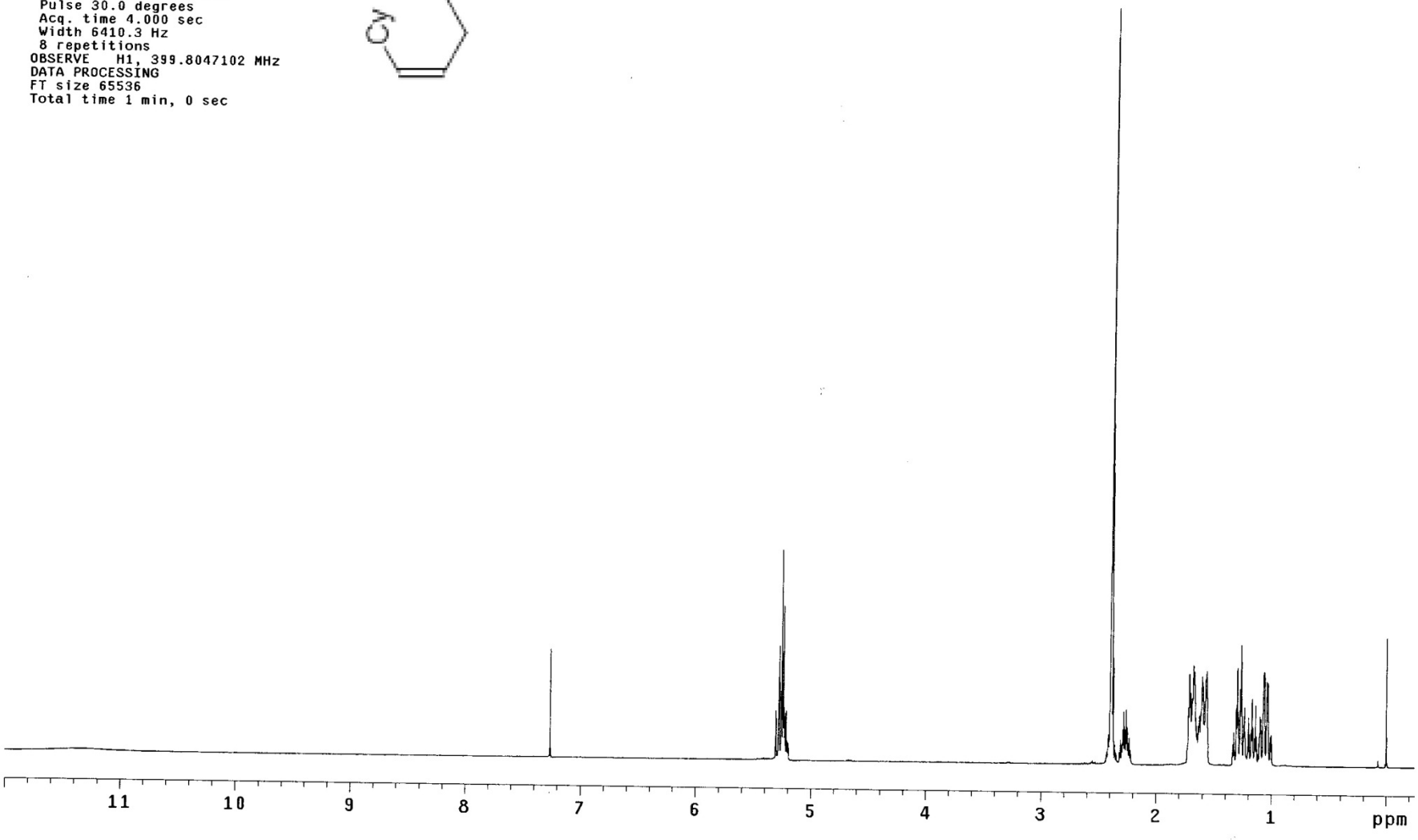


Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: 04_DHP_287_s2pul_H1
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8047102 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec

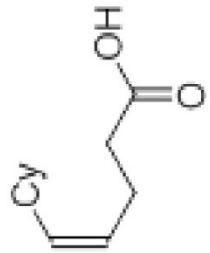


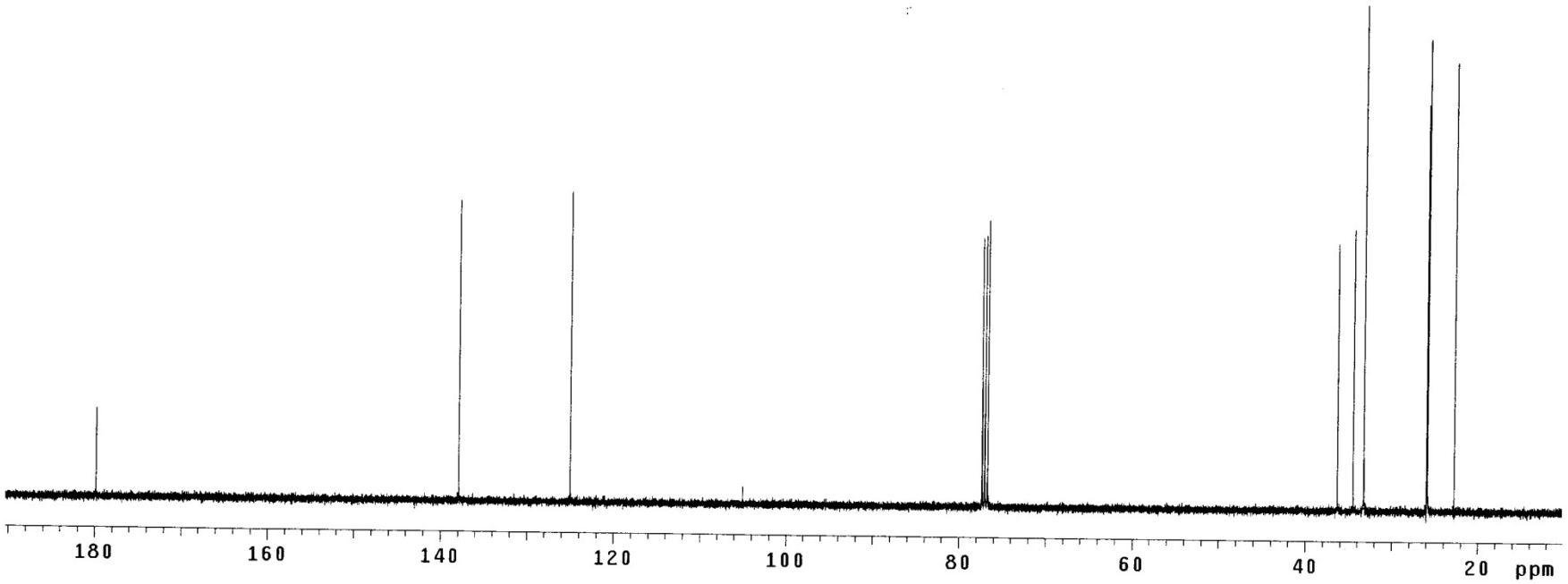


Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: 04_DHP_287_s2pul_C13
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 28 min, 14 sec

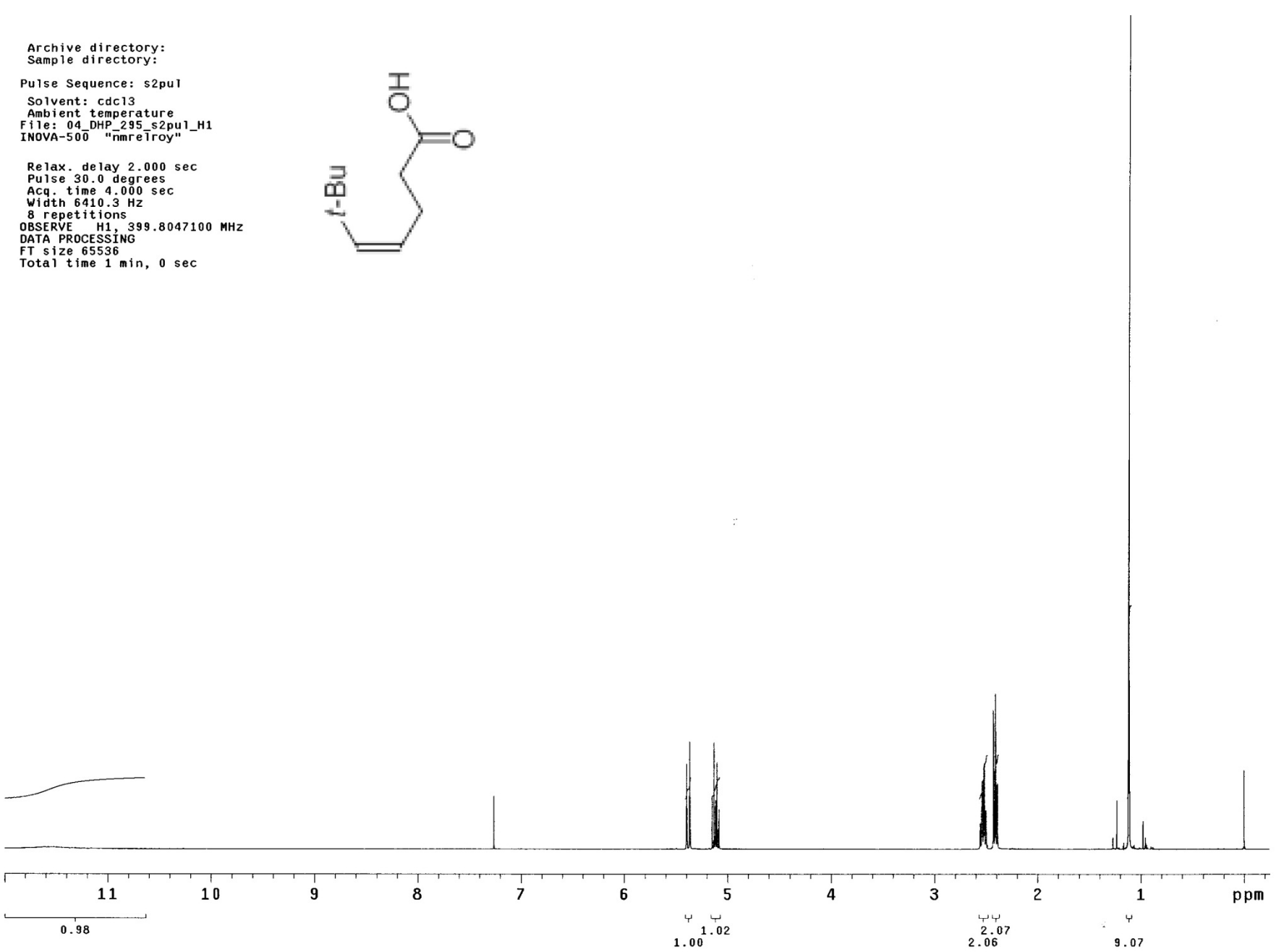


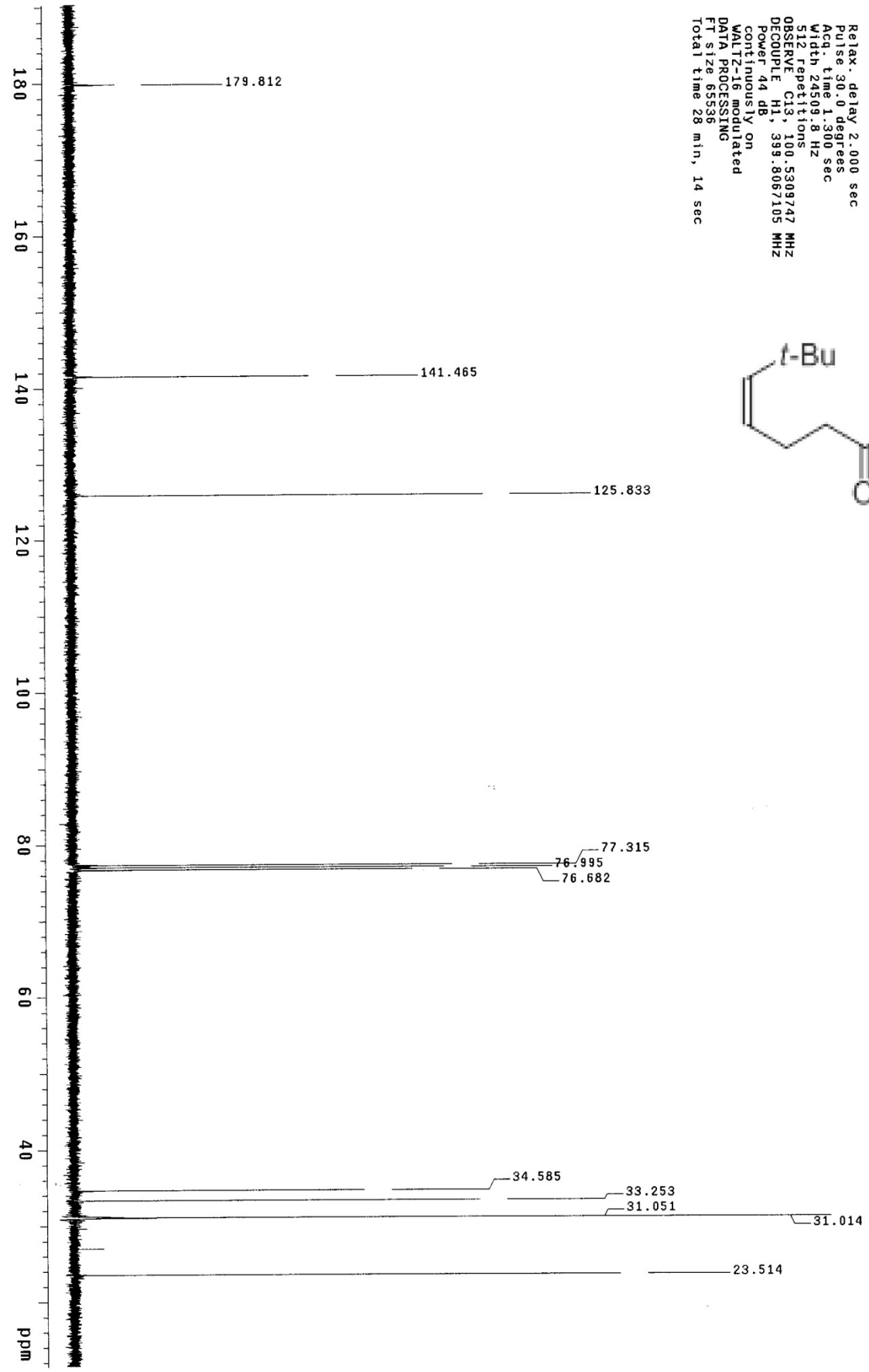


Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: 04_DHP_295_s2pul_H1
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8047100 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec

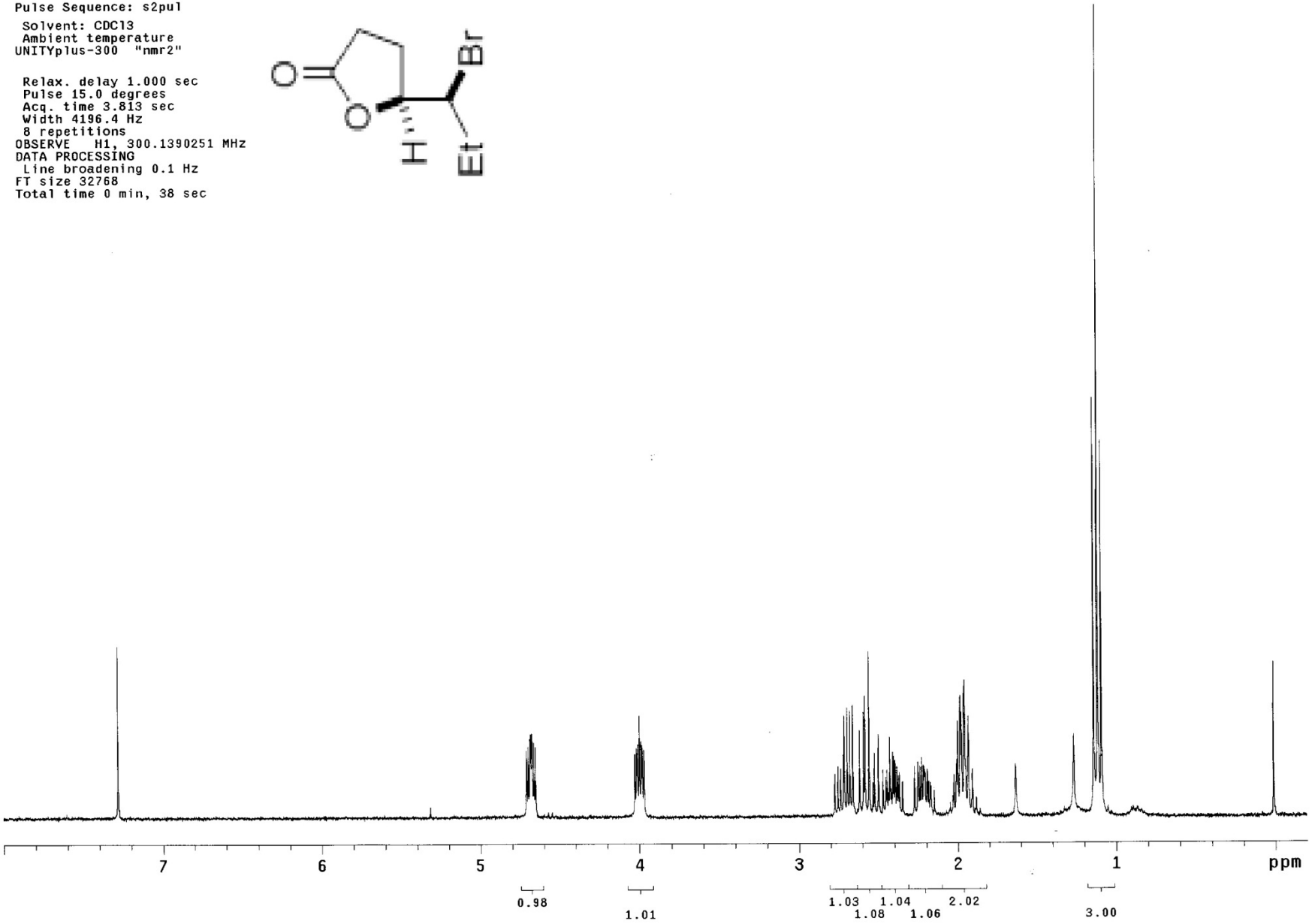


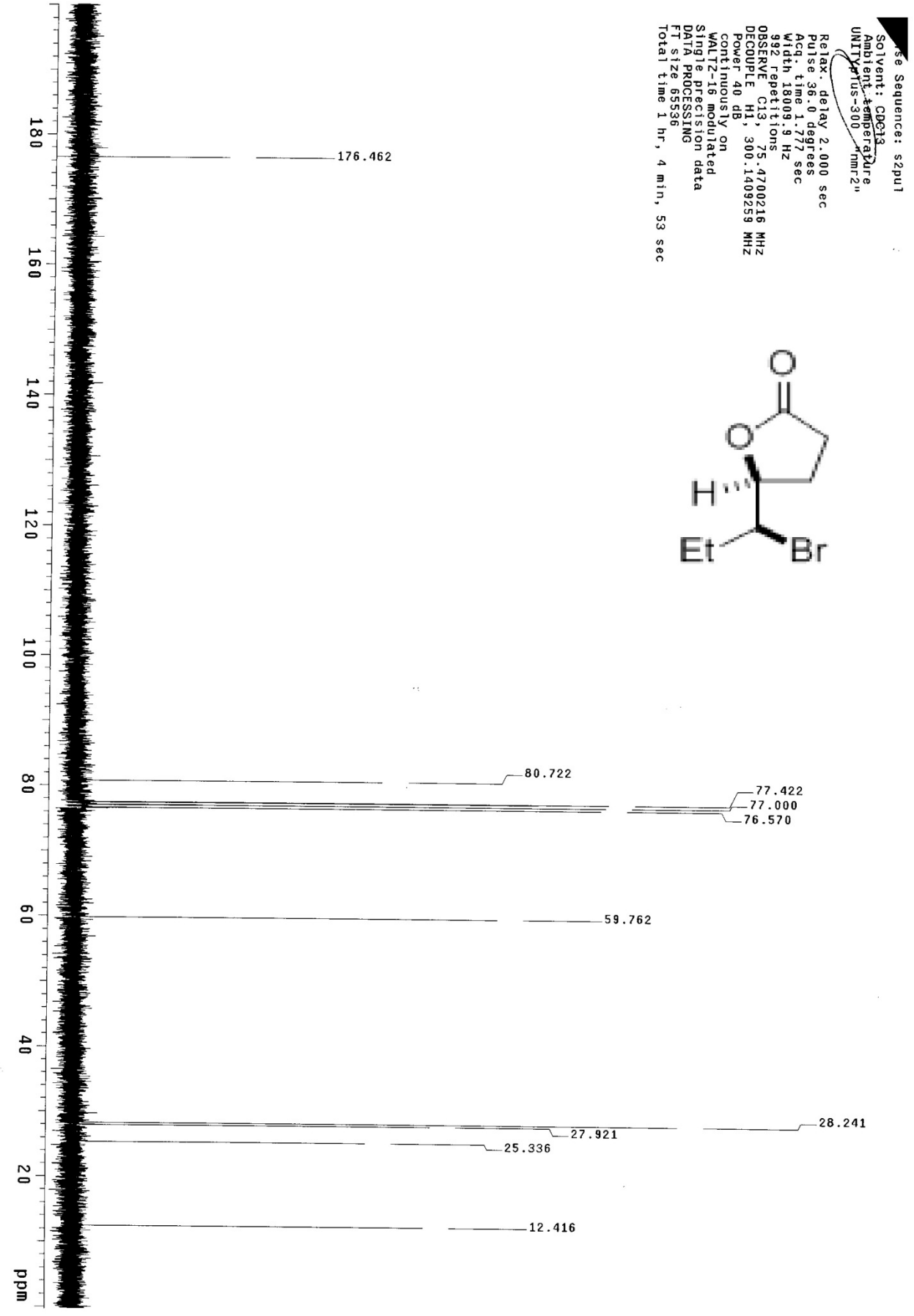

Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: 04_DHP_295_s2pul_C13
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 28 min, 14 sec
t-Bu
OH
O
179.812
141.465
125.833
77.315
76.995
76.682
34.585
33.253
31.051
31.014
23.514
180
160
140
120
100
80
60
40
ppm

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr2"

Relax. delay 1.000 sec
Pulse 15.0 degrees
Acq. time 3.813 sec
Width 4196.4 Hz
8 repetitions
OBSERVE H1, 300.1390251 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 38 sec

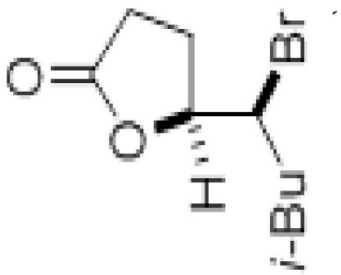


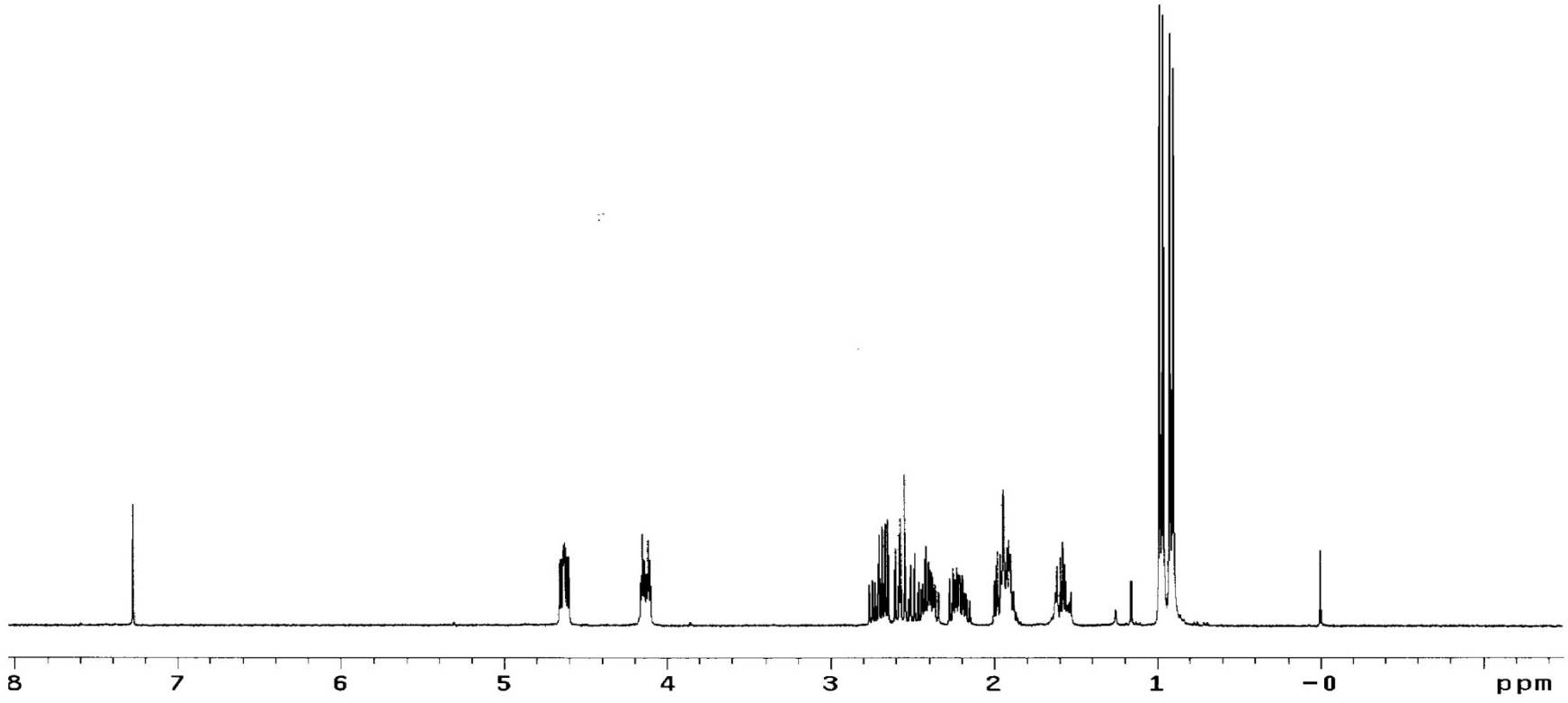

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITY plus-300 "nmr2"
Relax. delay 2.000 sec
Pulse 36.0 degrees
Acq. time 1.777 sec
Width 18009.9 Hz
992 repetitions
OBSERVE C13, 75.4700216 MHz
DECOUPLE H1, 300.1409259 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
Single precision data
DATA PROCESSING
FT size 65536
Total time 1 hr, 4 min, 53 sec
O
O
H
Et
Br
176.462
80.722
77.422
77.000
76.570
59.762
28.241
27.921
25.336
12.416
180
160
140
120
100
80
60
40
20
ppm

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
UNITYplus-300 "nmr2"

 Relax. delay 1.000 sec
 Pulse 15.0 degrees
 Acq. time 3.813 sec
 Width 4196.4 Hz
 16 repetitions
OBSERVE H1, 300.1390272 MHz
DATA PROCESSING
 Line broadening 0.1 Hz
FT size 32768
Total time 1 min, 17 sec

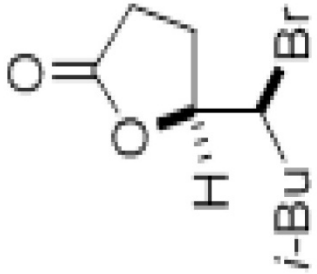


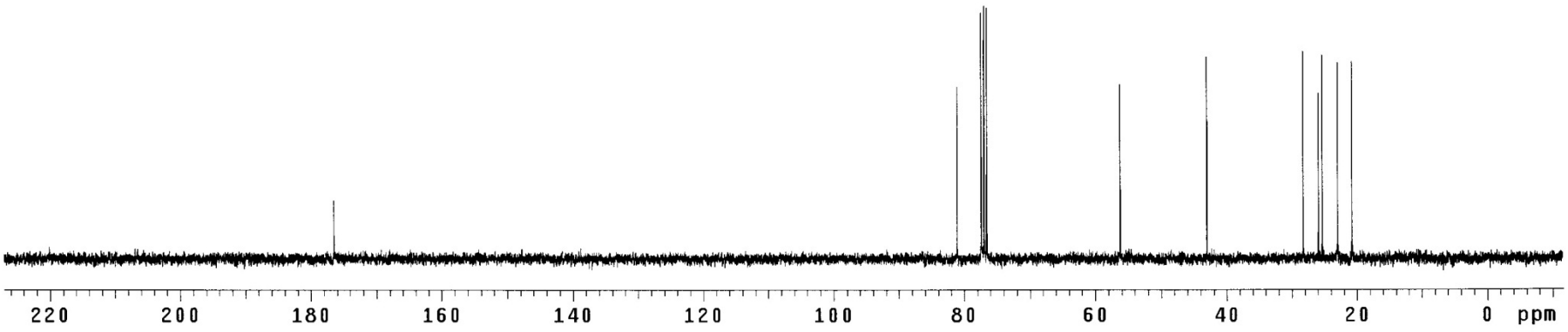


13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
UNITYplus-300 "nmr2"

 Relax. delay 2.000 sec
 Pulse 36.0 degrees
 Acq. time 1.777 sec
 Width 18009.9 Hz
 332 repetitions
OBSERVE C13, 75.4700216 MHz
DECOUPLE H1, 300.1409259 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
Single precision data
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 4 min, 38 sec



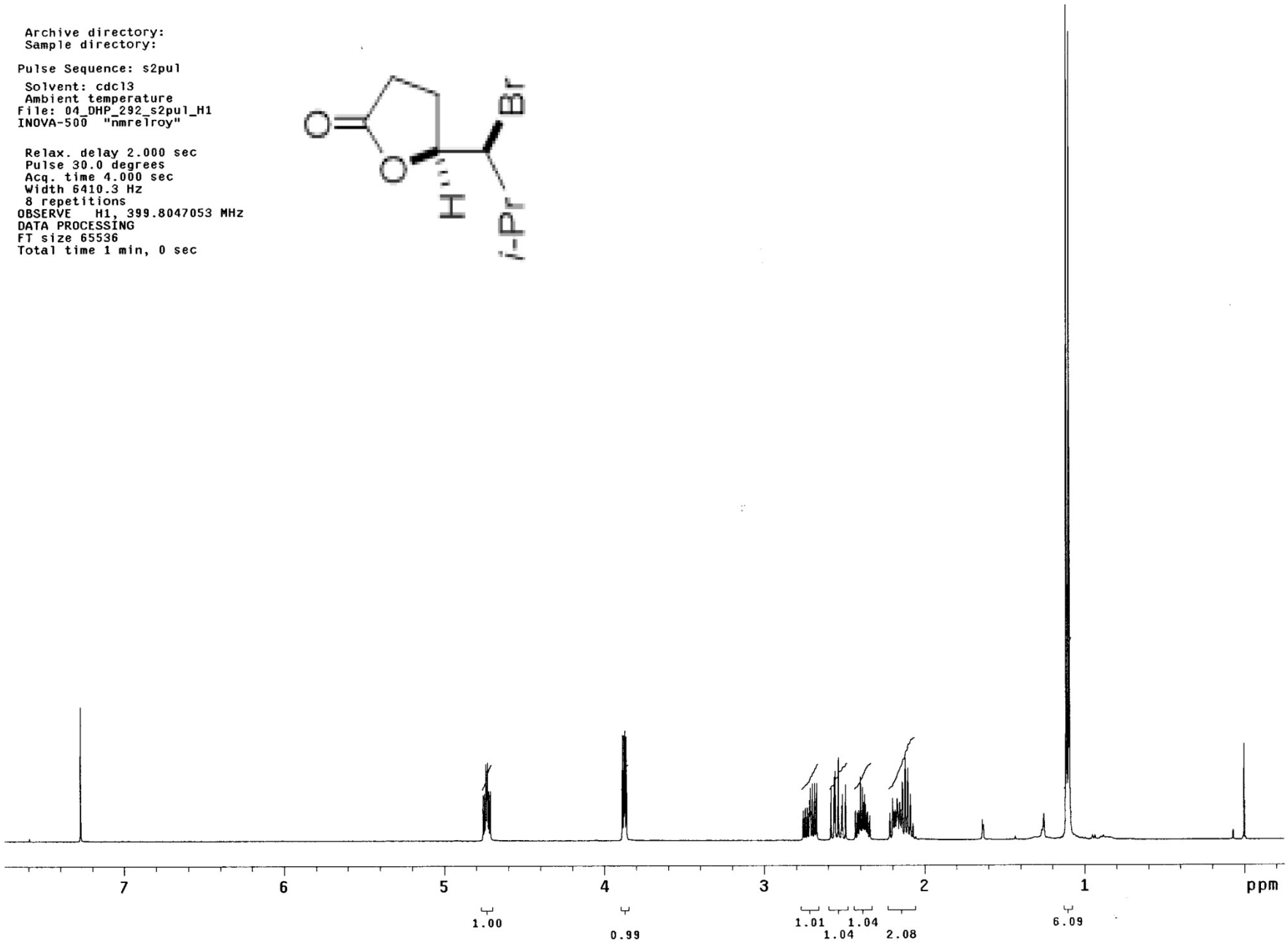


Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
File: 04_DHP_292_s2pul_H1
INOVA-500 "nmrelroy"

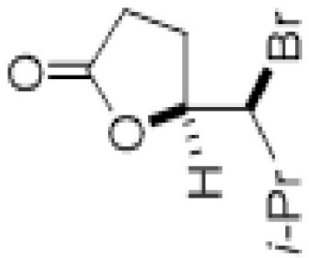
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8047053 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec

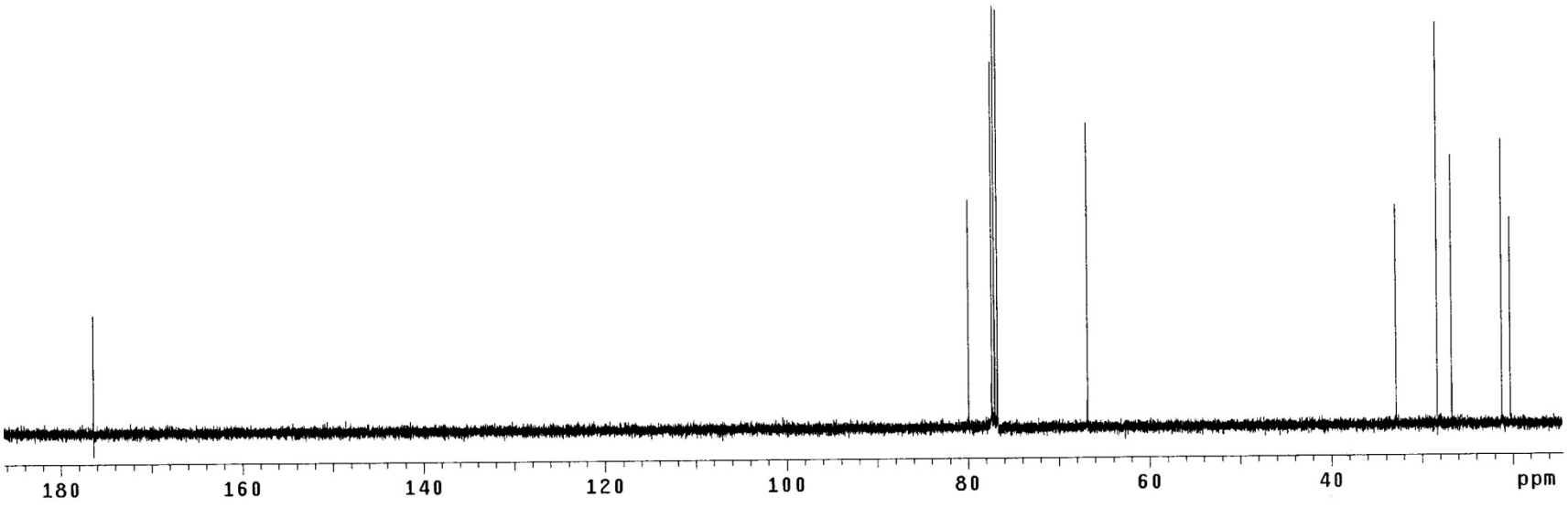


```
 Archive directory:
 Sample directory:

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
User:  1-14-87
File: 04_DHP_292_s2pul_C13
INOVA-500  "nmrelroy"

 Relax. delay 2.000 sec
 Pulse 30.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 512 repetitions
OBSERVE  C13, 100.5309747 MHz
DECOUPLE  H1, 399.8067105 MHz
 Power 44 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 28 min, 14 sec
```

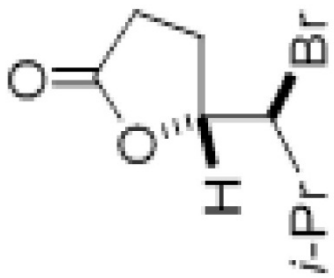


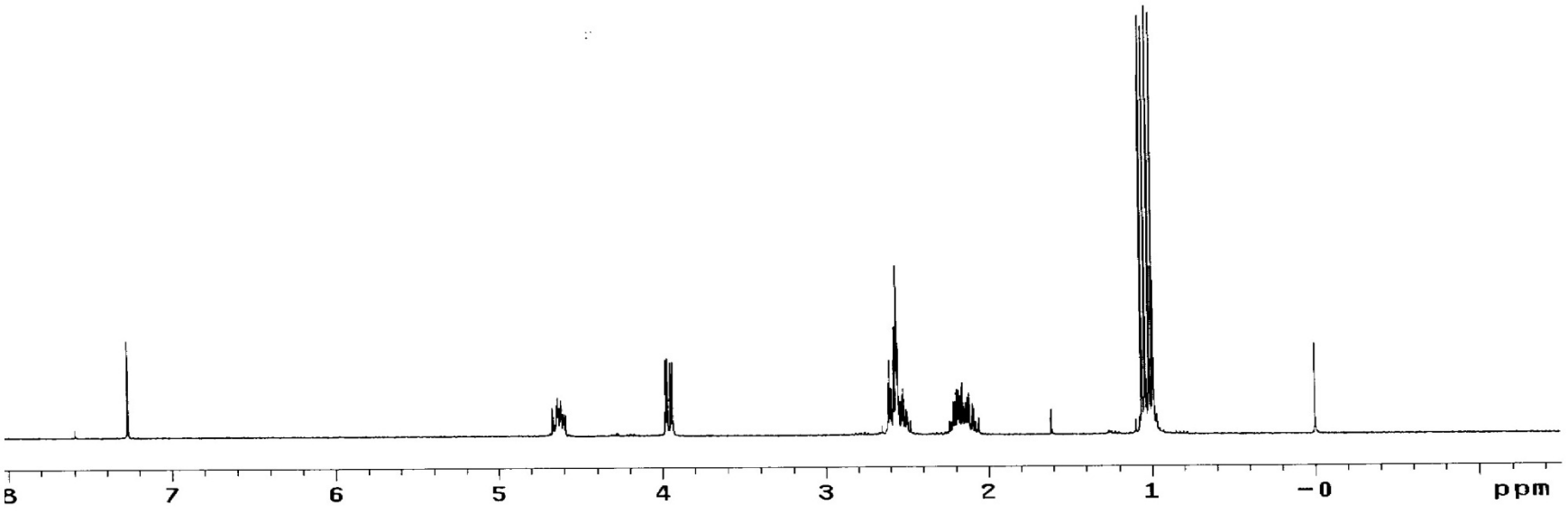


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr2"

Relax. delay 1.000 sec
Pulse 15.0 degrees
Acq. time 3.813 sec
Width 4196.4 Hz
16 repetitions
OBSERVE H1, 300.1390288 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 1 min, 17 sec

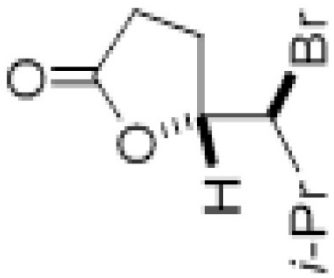


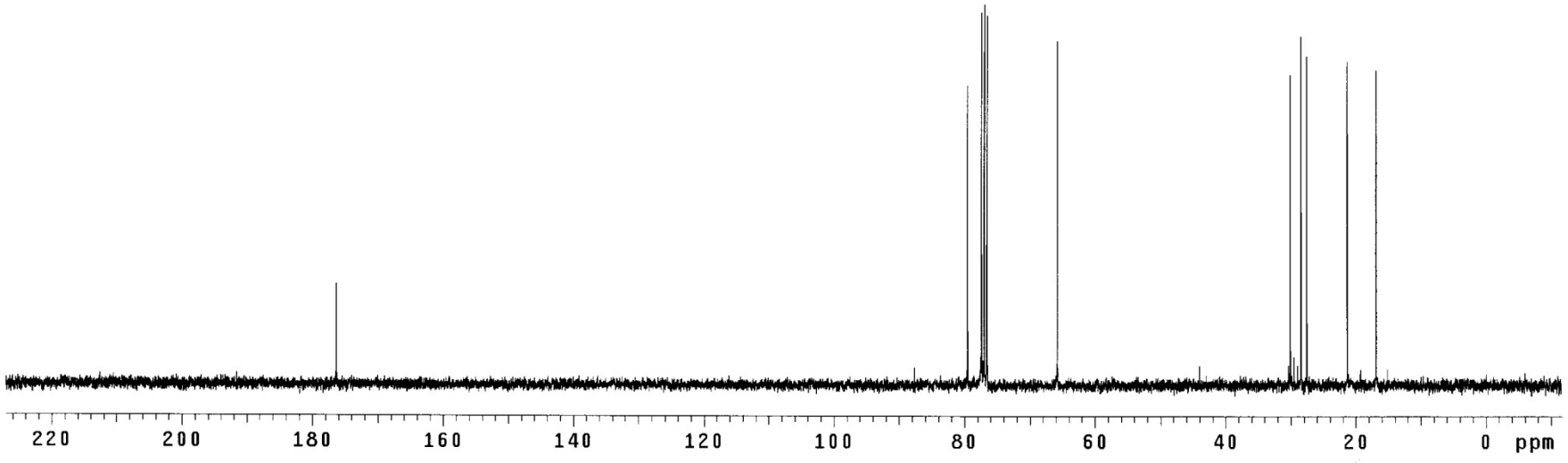


13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
UNITYplus-300 "nmr2"

 Relax. delay 2.000 sec
 Pulse 36.0 degrees
 Acq. time 1.777 sec
 Width 18009.9 Hz
 737 repetitions
OBSERVE C13, 75.4700216 MHz
DECOUPLE H1, 300.1409259 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
Single precision data
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 4 min, 38 sec



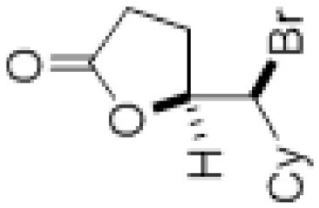


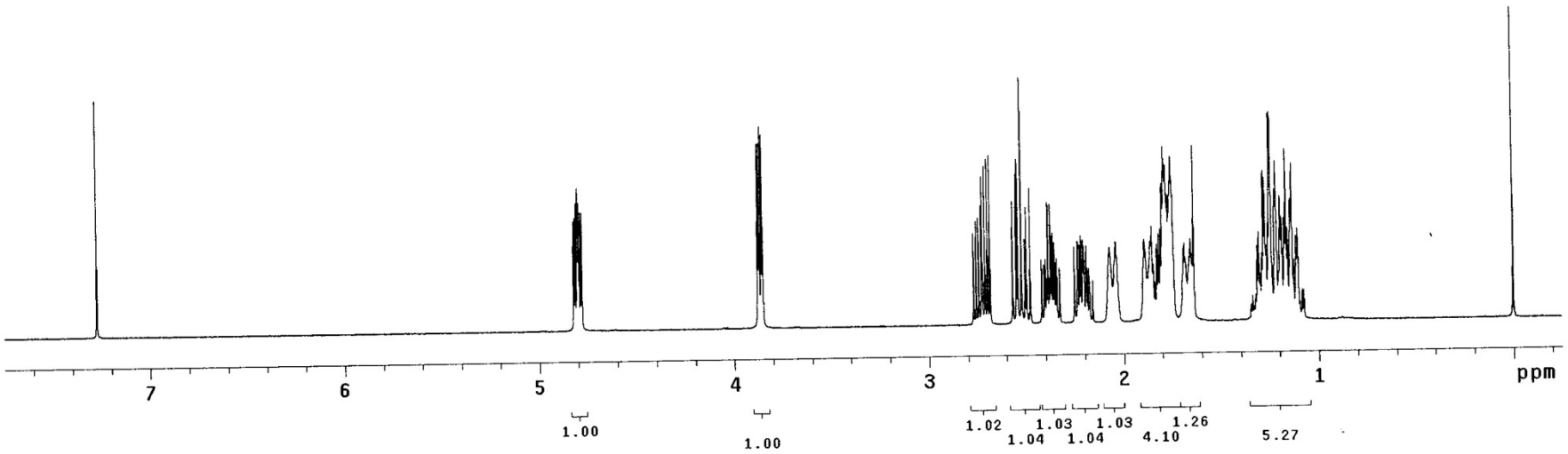
Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
File: 04_DHP_290_s2pul_H1
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8047055 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec

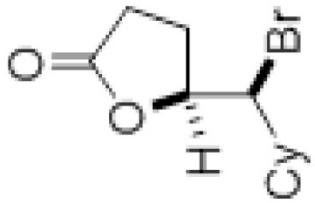


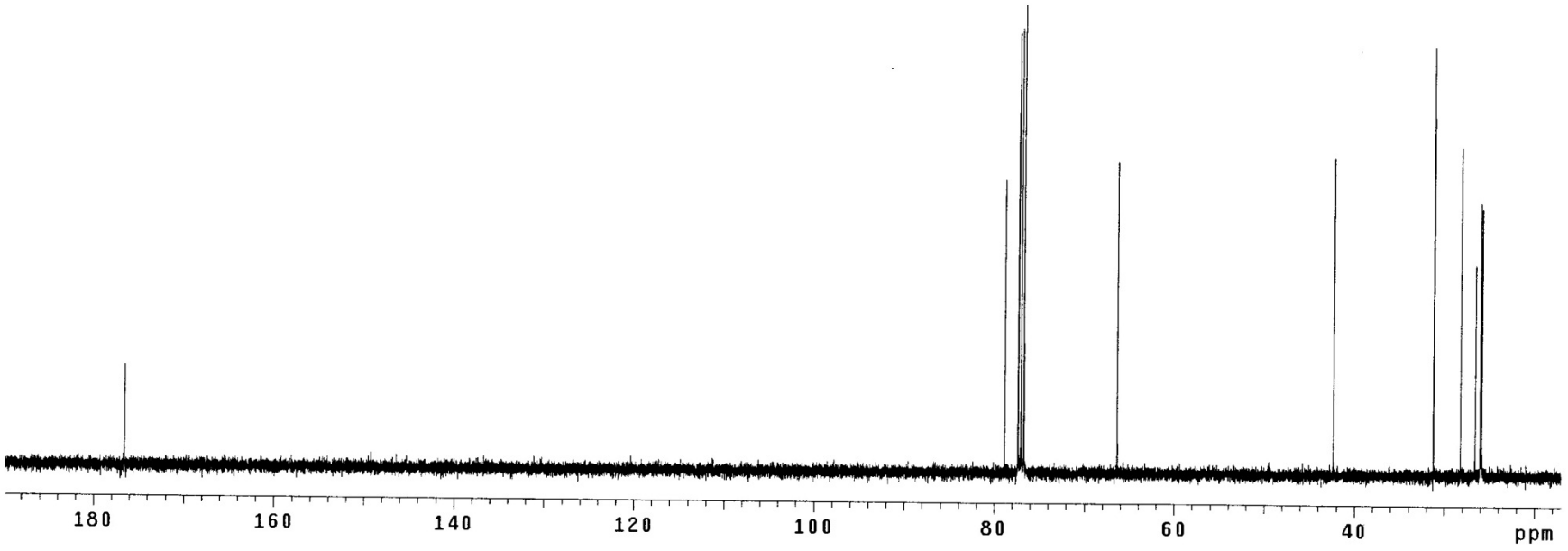


Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: 04_DHP_290_s2pul_C13
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 28 min, 14 sec

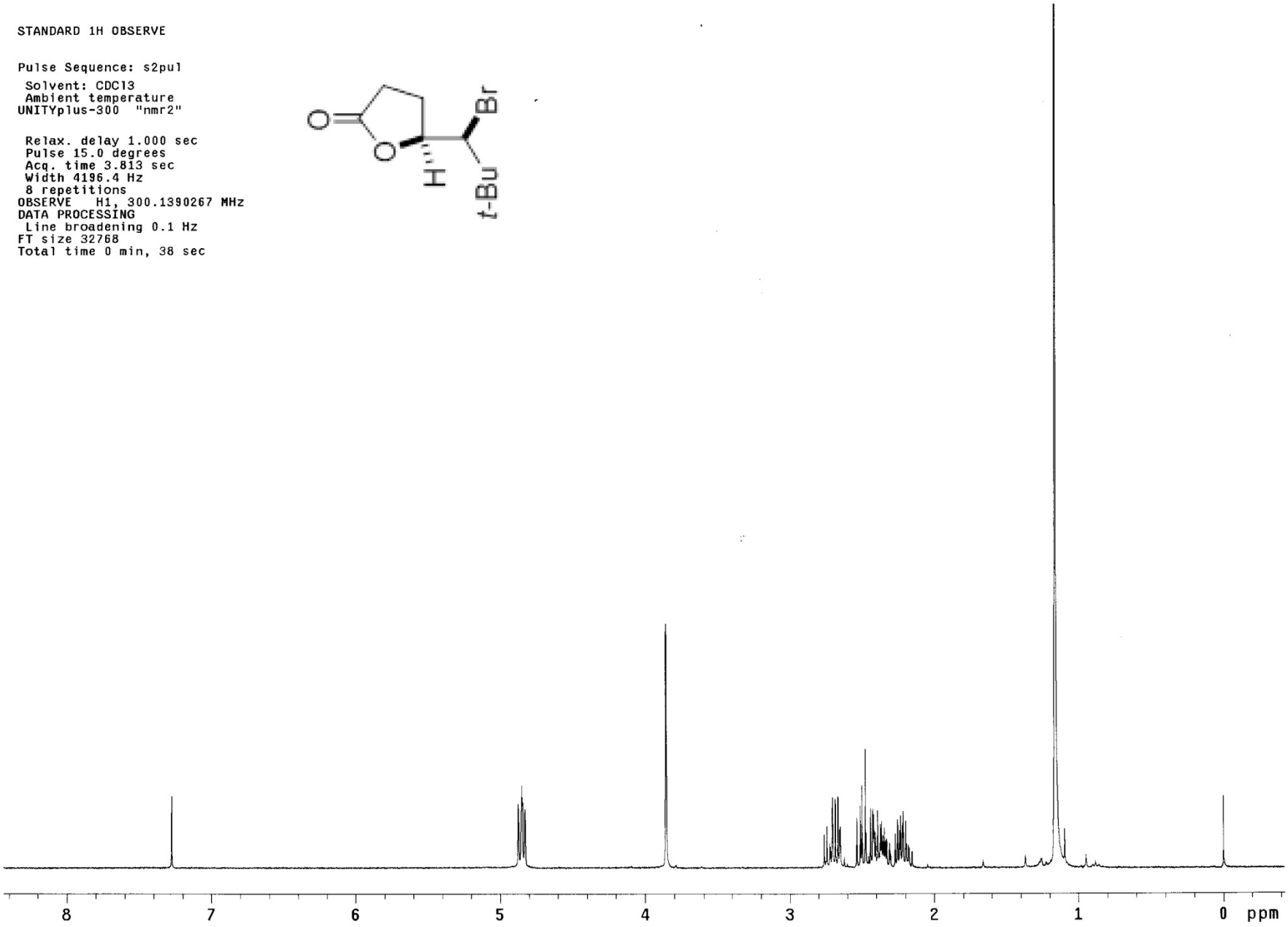




STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr2"

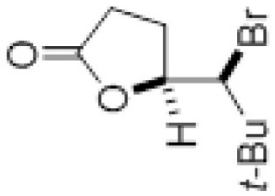
Relax. delay 1.000 sec
Pulse 15.0 degrees
Acq. time 3.813 sec
Width 4196.4 Hz
8 repetitions
OBSERVE H1, 300.1390267 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 38 sec

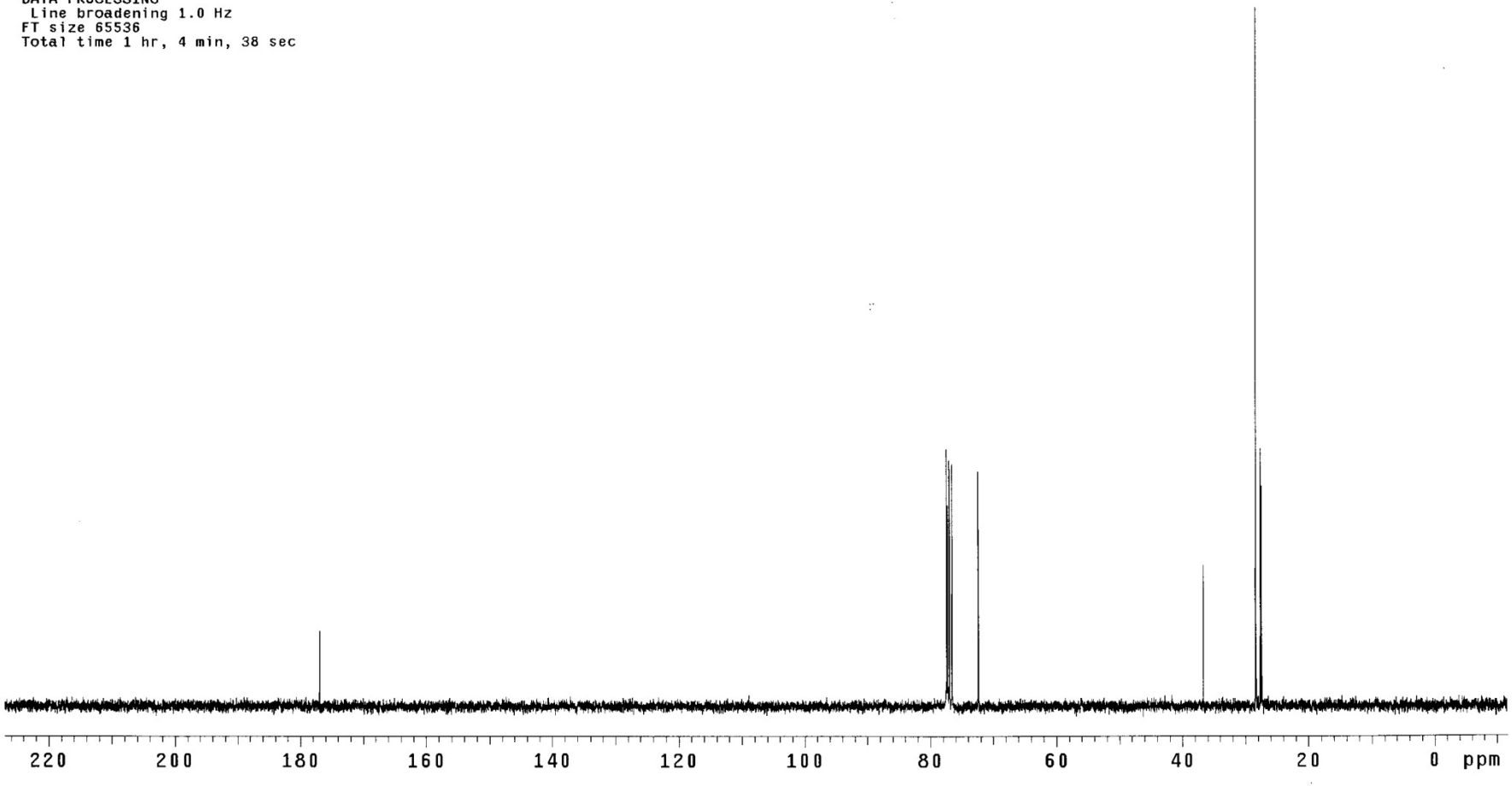


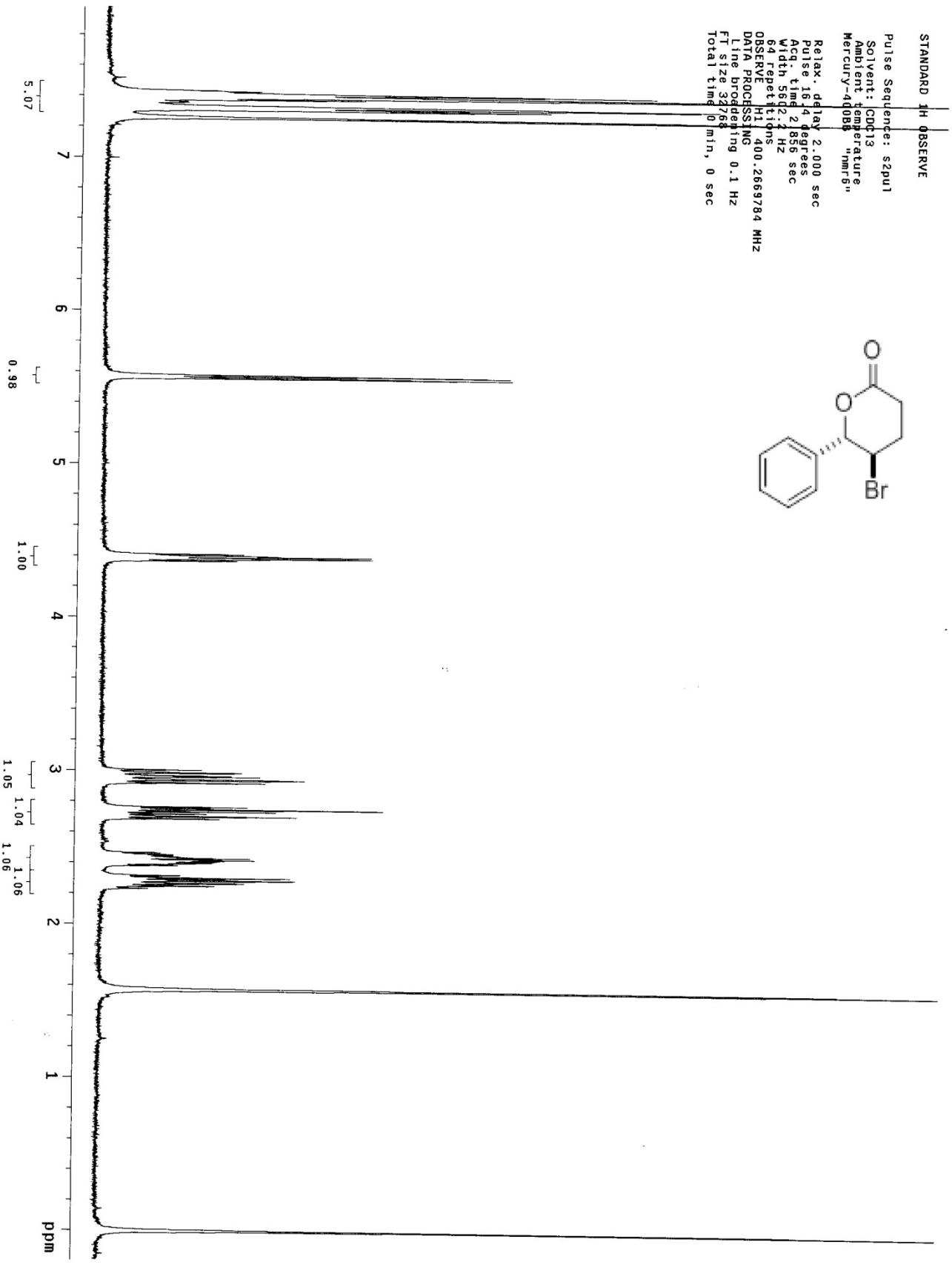
13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
UNITYplus-300 "nmr2"

 Relax. delay 2.000 sec
 Pulse 36.0 degrees
 Acq. time 1.777 sec
 Width 18009.9 Hz
 292 repetitions
OBSERVE C13, 75.4700222 MHz
DECOUPLE H1, 300.1409259 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
Single precision data
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 4 min, 38 sec



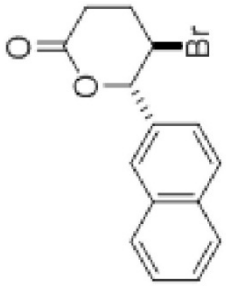


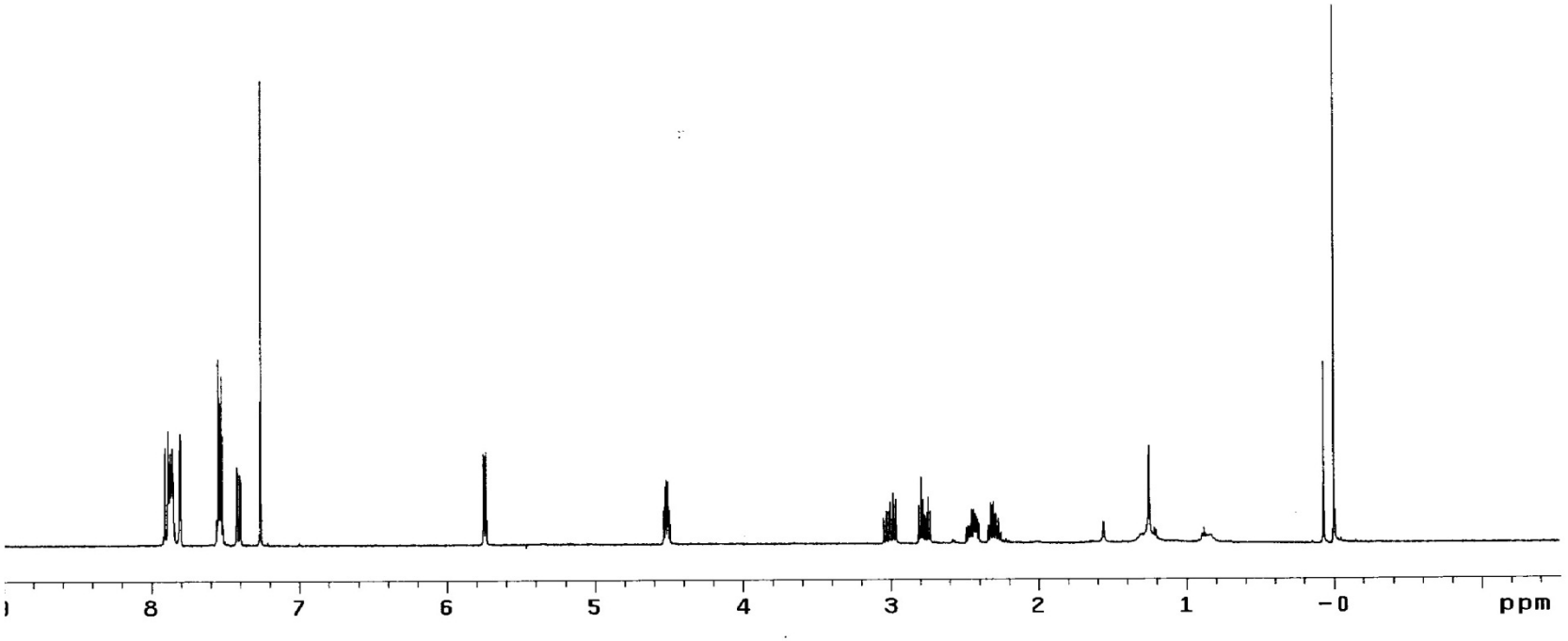

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "nmrfg"
Relax. delay 2.000 sec
Pulse 16.4 degrees
Acq. time 2.8856 sec
Width 5602.2 Hz
64 repetitions
OBSERVE H1, 400.2669784 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 0 sec
O
O
Br
5.07
0.98
1.00
1.05
1.04
1.06
1.06
7
6
5
4
3
2
1
ppm

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "nmr6"

Relax. delay 2.000 sec
Pulse 16.4 degrees
Acq. time 2.856 sec
Width 5602.2 Hz
16 repetitions
OBSERVE H1, 400.2669778 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 1 min, 20 sec

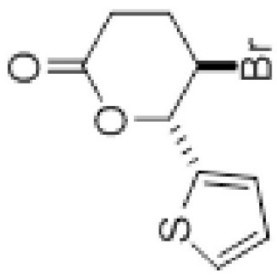


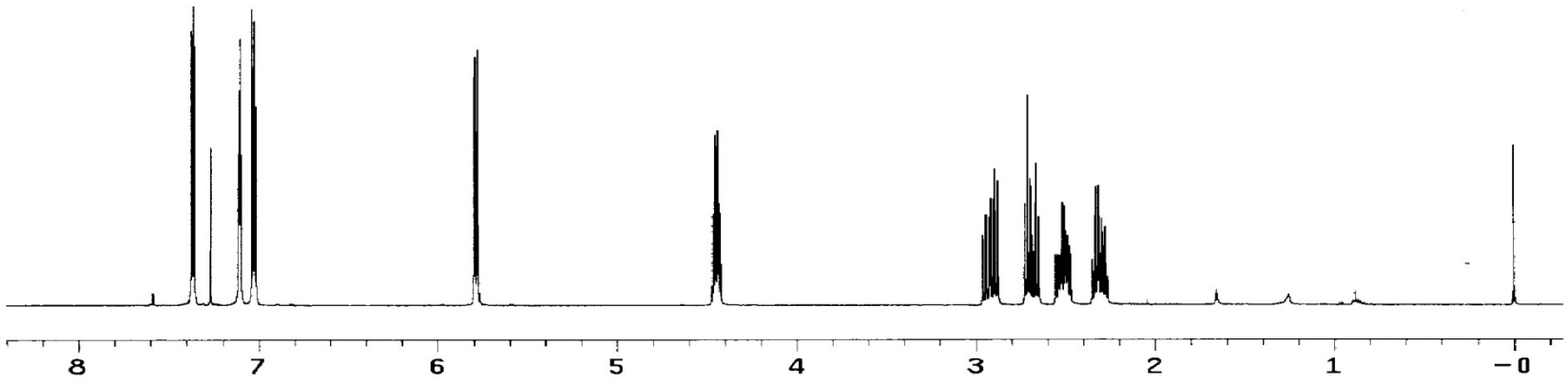


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "nmr6"

Relax. delay 2.000 sec
Pulse 16.4 degrees
Acq. time 2.856 sec
Width 5602.2 Hz
8 repetitions
OBSERVE H1, 400.2669750 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 41 sec

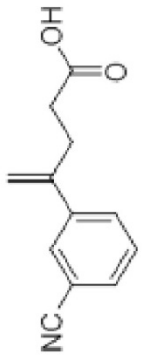


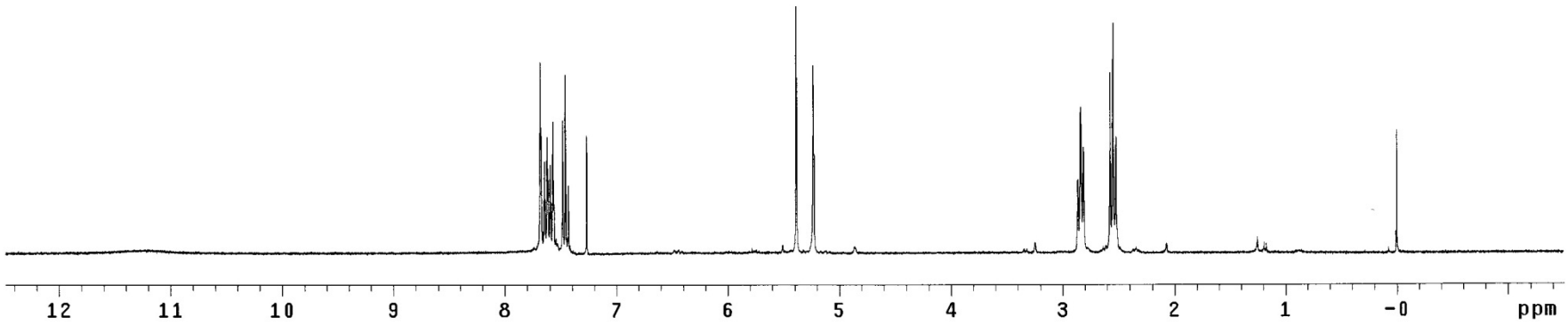


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr2"

Relax. delay 1.000 sec
Pulse 15.0 degrees
Acq. time 3.813 sec
Width 4196.4 Hz
16 repetitions
OBSERVE H1, 300.1390303 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 1 min, 17 sec

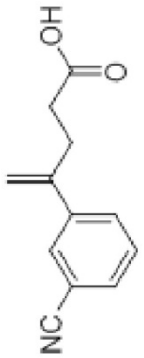


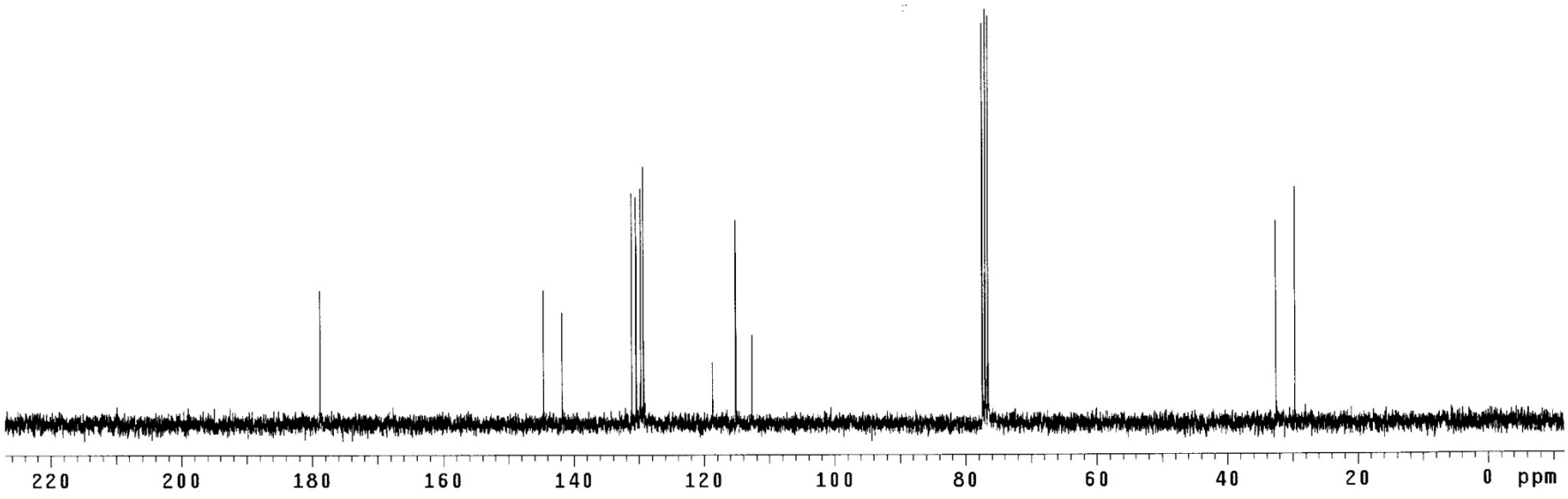


13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
UNITYplus-300 "nmr2"

 Relax. delay 2.000 sec
 Pulse 36.0 degrees
 Acq. time 1.777 sec
 Width 18009.9 Hz
 383 repetitions
OBSERVE C13, 75.4700205 MHz
DECOUPLE H1, 300.1409259 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
Single precision data
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 4 min, 38 sec

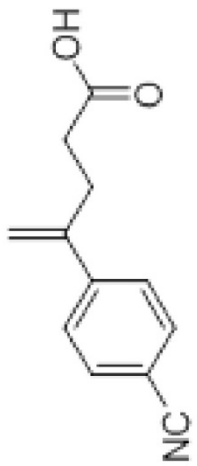


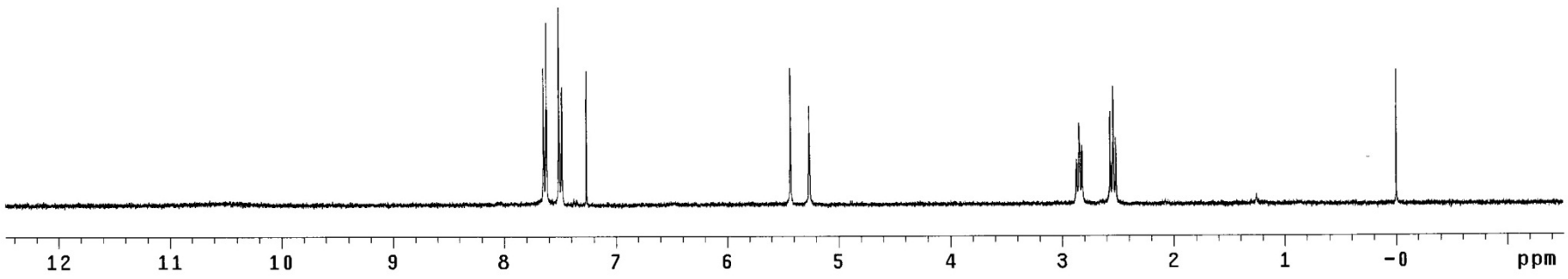


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
UNITYplus-300 "nmr2"

 Relax. delay 1.000 sec
 Pulse 15.0 degrees
 Acq. time 3.813 sec
 Width 4196.4 Hz
 16 repetitions
OBSERVE H1, 300.1390308 MHz
DATA PROCESSING
 Line broadening 0.1 Hz
FT size 32768
Total time 1 min, 17 sec

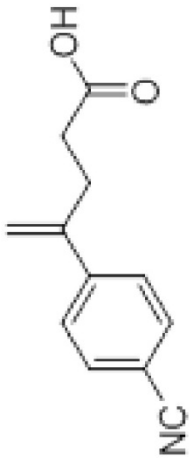


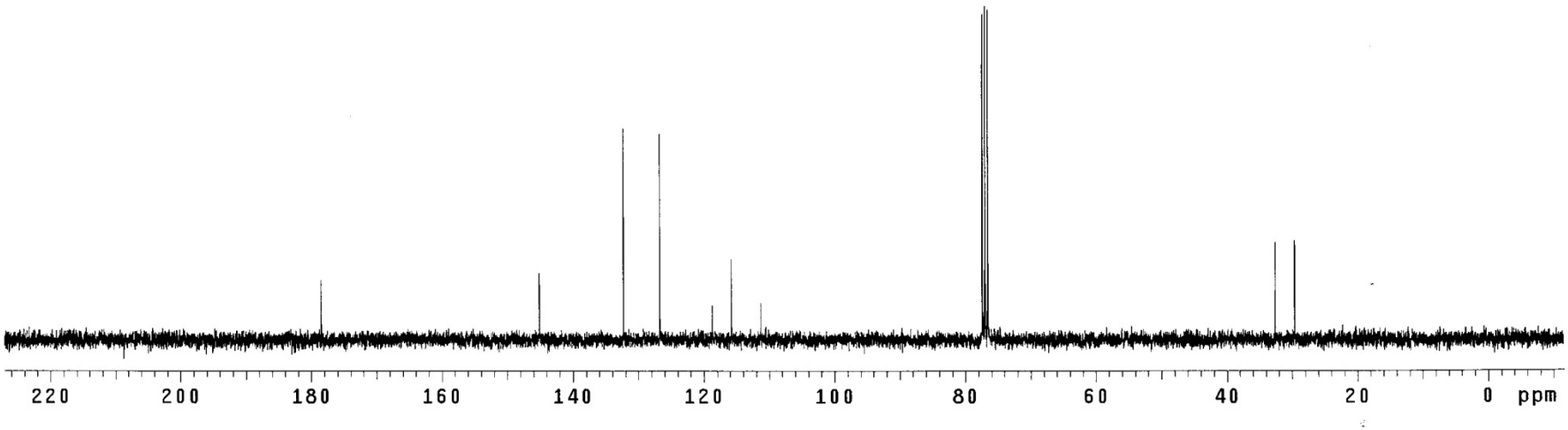


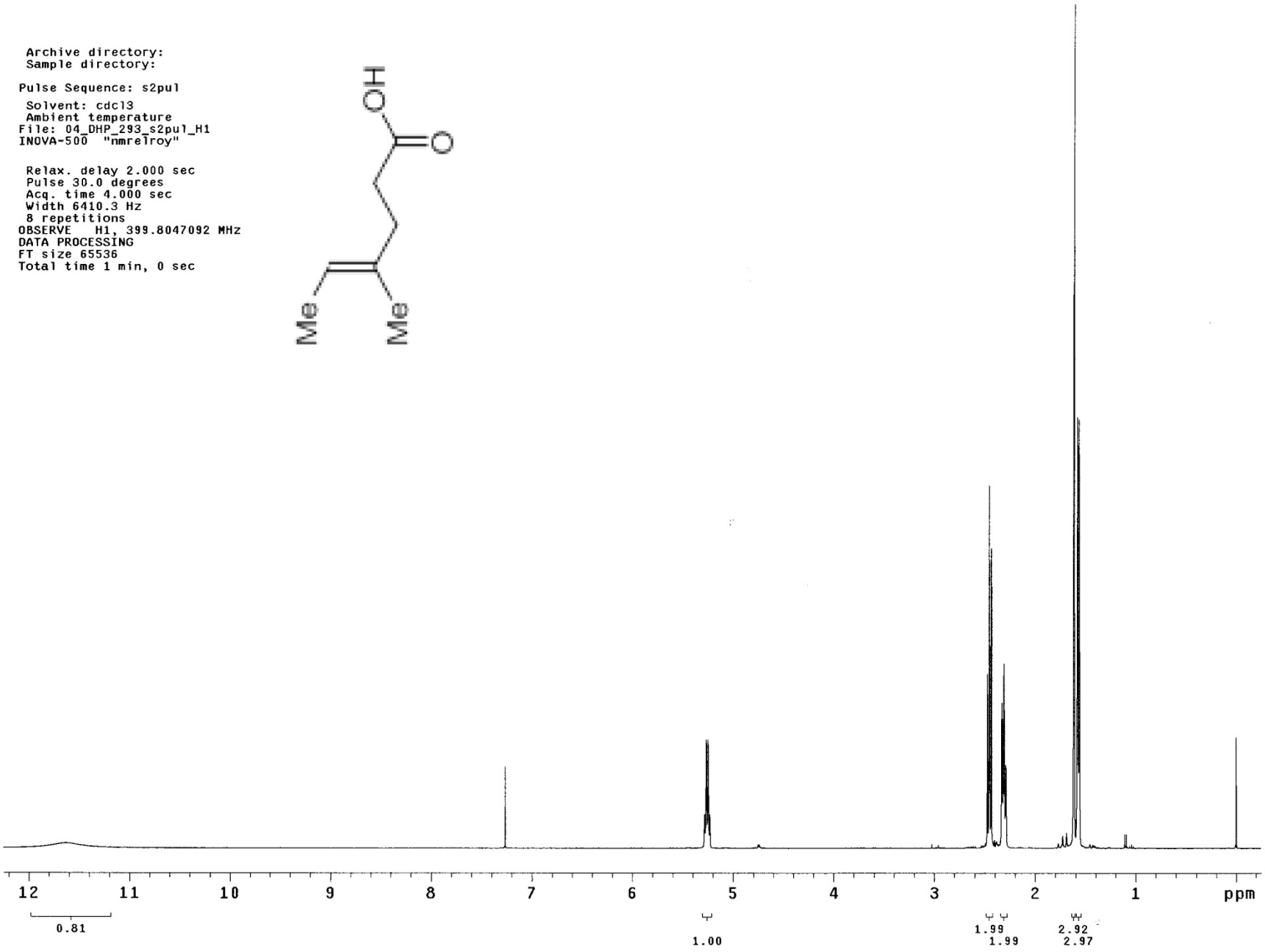
13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
UNITYplus-300 "nmr2"

 Relax. delay 2.000 sec
 Pulse 36.0 degrees
 Acq. time 1.777 sec
 Width 18009.9 Hz
 370 repetitions
OBSERVE C13, 75.4700200 MHz
DECOUPLE H1, 300.1409259 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
Single precision data
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 32 min, 19 sec



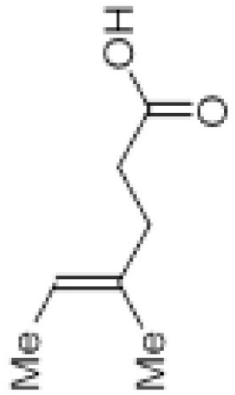


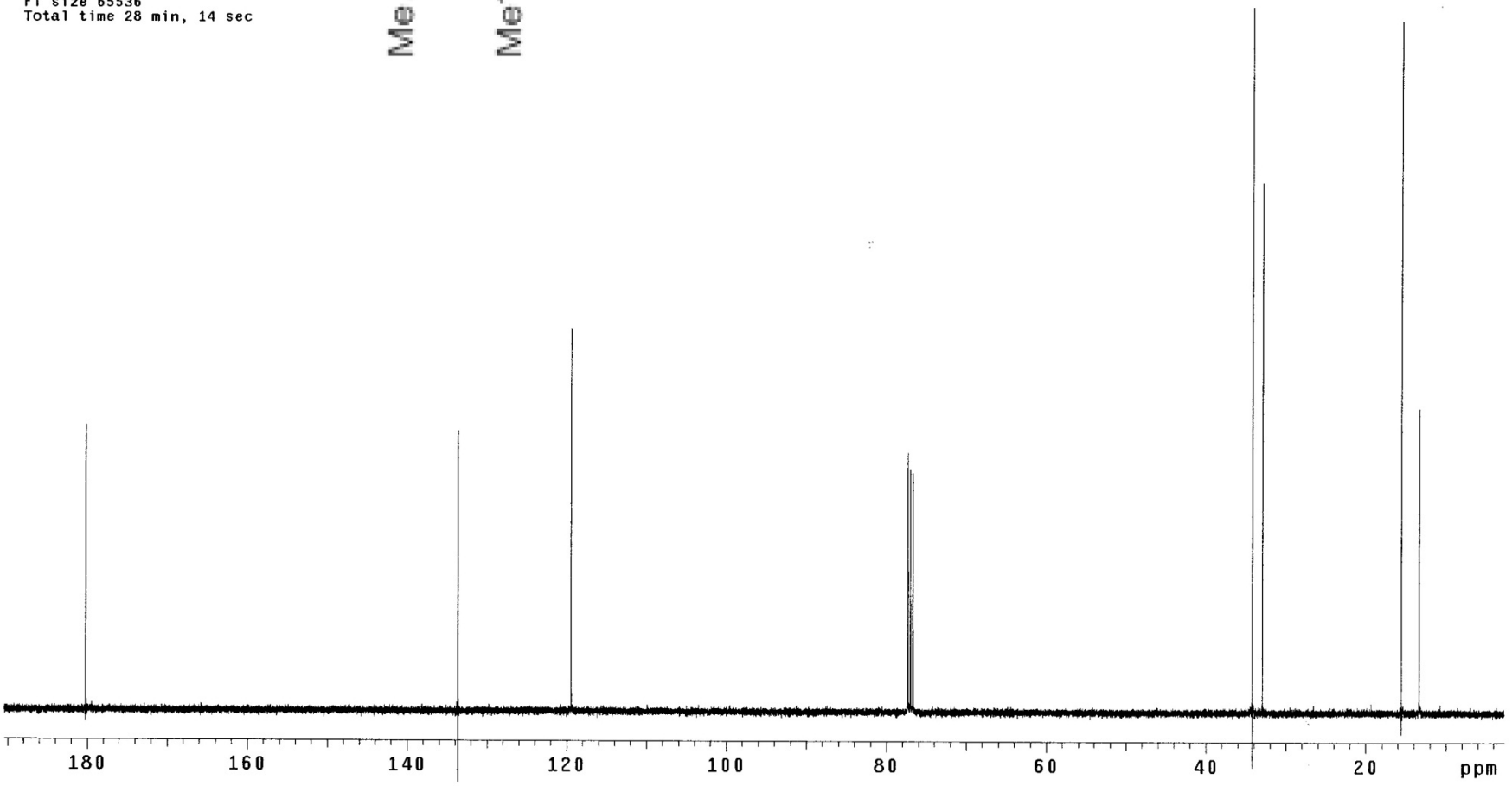

Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: 04_DHP_293_s2pul_H1
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8047092 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec
OH
O
Me
Me
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.81
1.00
1.99
1.99
2.92
2.97

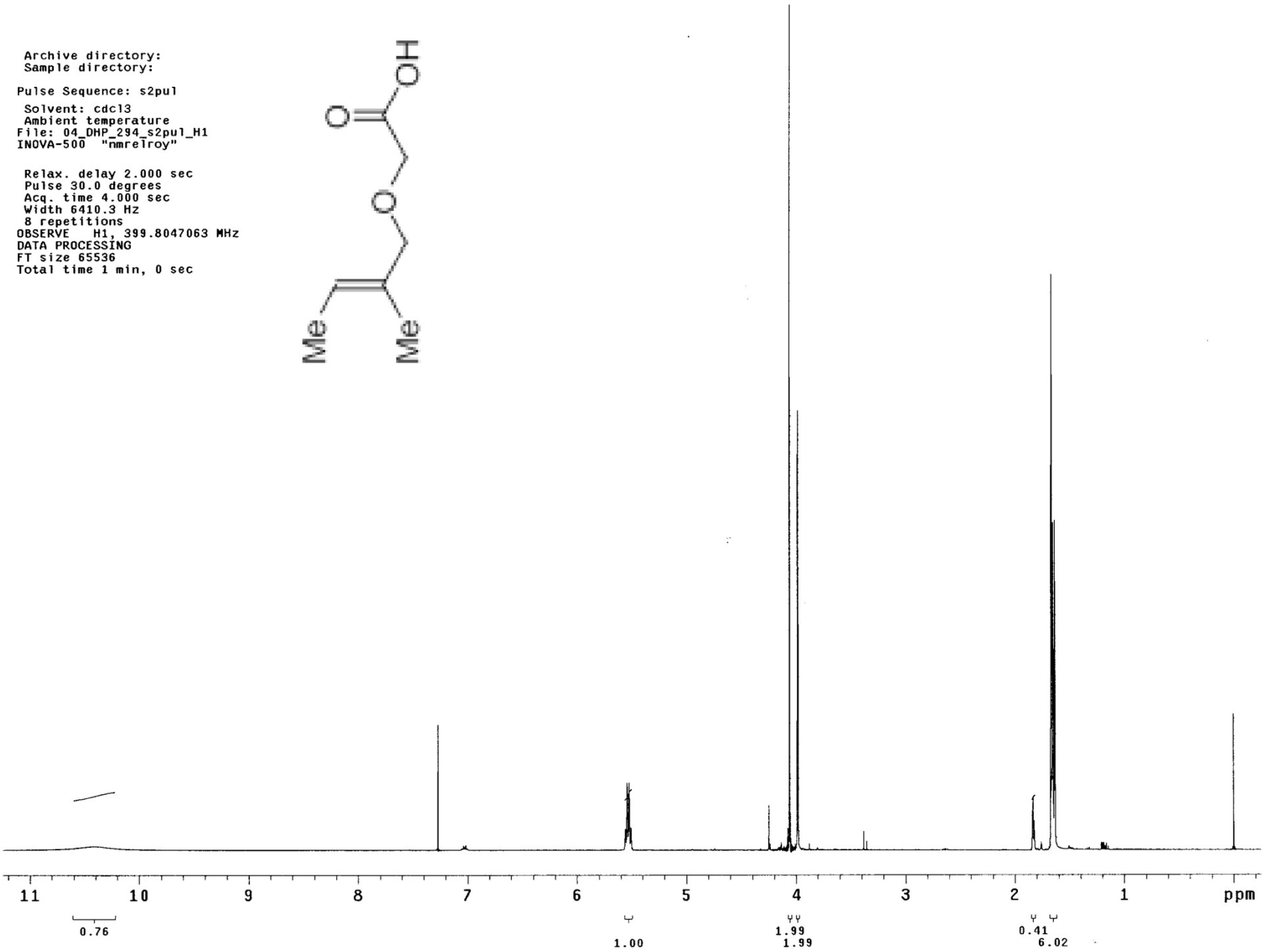
Archive directory:
Sample directory:

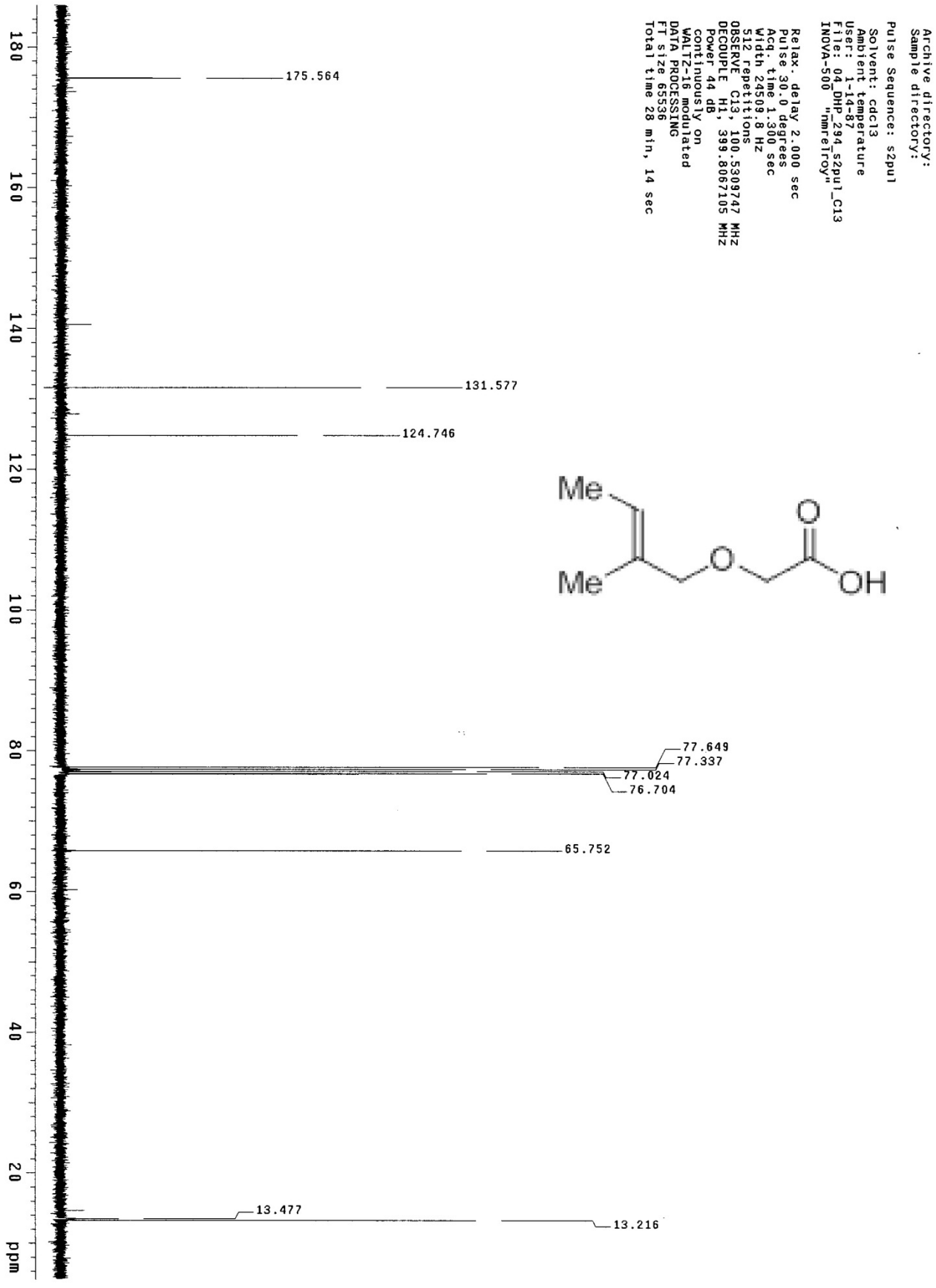
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: 04_DHP_293_s2pul_C13
INOVA-500 "nmrelroy"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 28 min, 14 sec





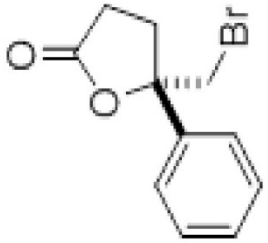

Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: 04_DHP_294_s2pul_H1
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.8047063 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 0 sec
O
OH
O
Me
Me
11
10
9
8
7
6
5
4
3
2
1
ppm
0.76
1.00
1.99
1.99
0.41
6.02

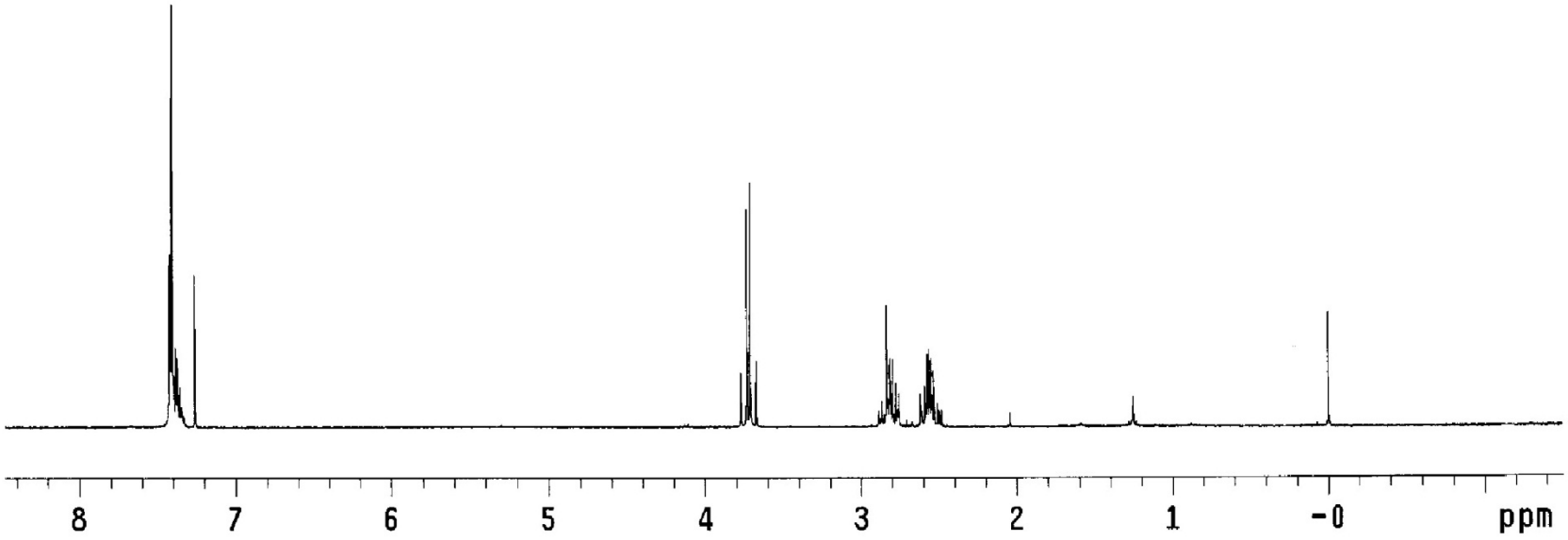

Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: 04_DHP_294_s2pul_C13
INOVA-500 "nmrelroy"
Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.5309747 MHz
DECOUPLE H1, 399.8067105 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 28 min, 14 sec
175.564
131.577
124.746
77.649
77.337
77.024
76.704
65.752
13.477
13.216
Me
Me
O
O
OH
180
160
140
120
100
80
60
40
20
ppm

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
UNITYplus-300 "nmr2"

 Relax. delay 1.000 sec
 Pulse 15.0 degrees
 Acq. time 3.813 sec
 Width 4196.4 Hz
 16 repetitions
OBSERVE H1, 300.1390311 MHz
DATA PROCESSING
 Line broadening 0.1 Hz
FT size 32768
Total time 1 min, 17 sec

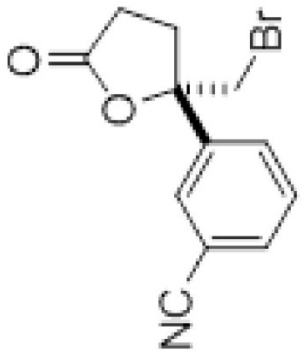


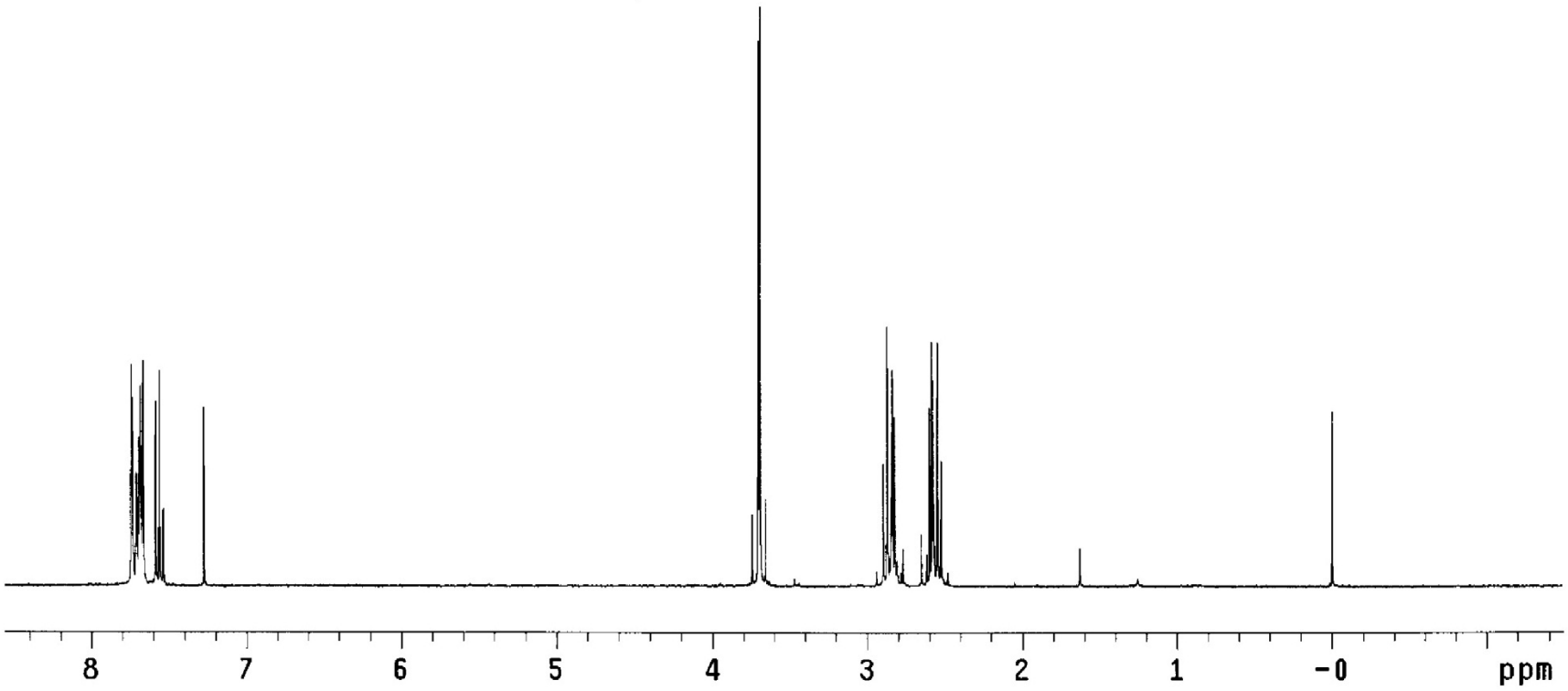


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
UNITYplus-300 "nmr2"

 Relax. delay 1.000 sec
 Pulse 15.0 degrees
 Acq. time 3.813 sec
 Width 4196.4 Hz
 16 repetitions
OBSERVE   H1, 300.1390277 MHz
DATA PROCESSING
 Line broadening 0.1 Hz
FT size 32768
Total time 1 min, 17 sec

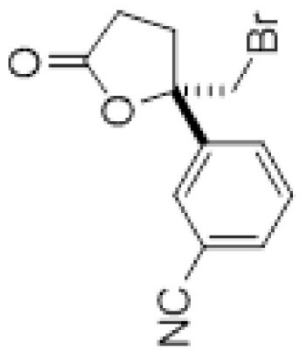


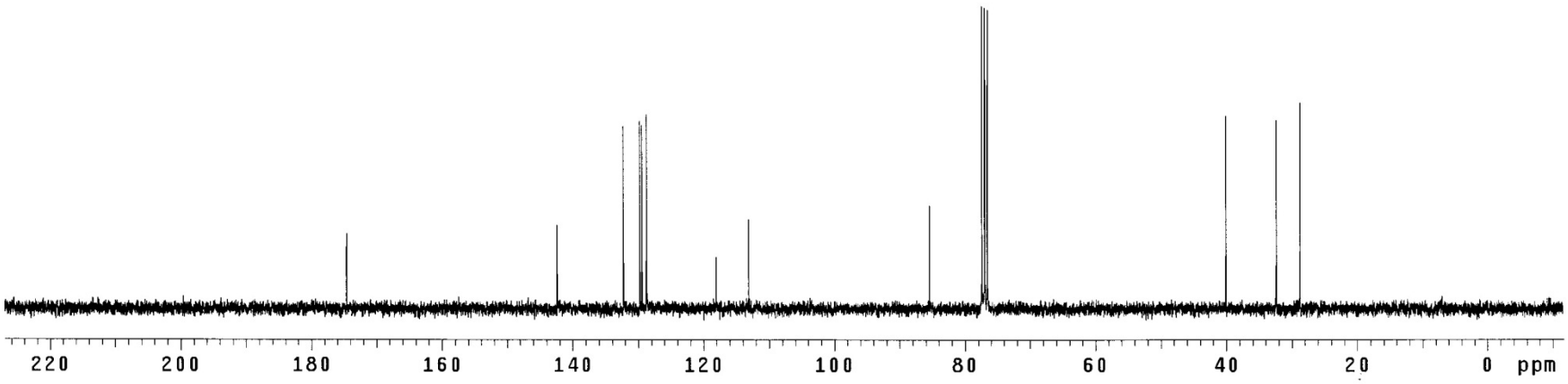


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr2"

Relax. delay 2.000 sec
Pulse 36.0 degrees
Acq. time 1.777 sec
Width 18009.9 Hz
409 repetitions
OBSERVE C13, 75.4700227 MHz
DECOUPLE H1, 300.1409259 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
Single precision data
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 32 min, 19 sec

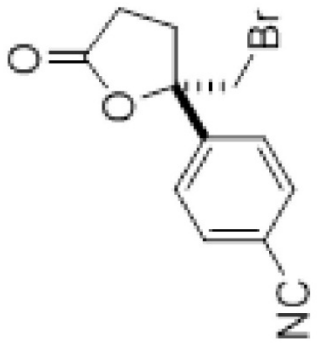


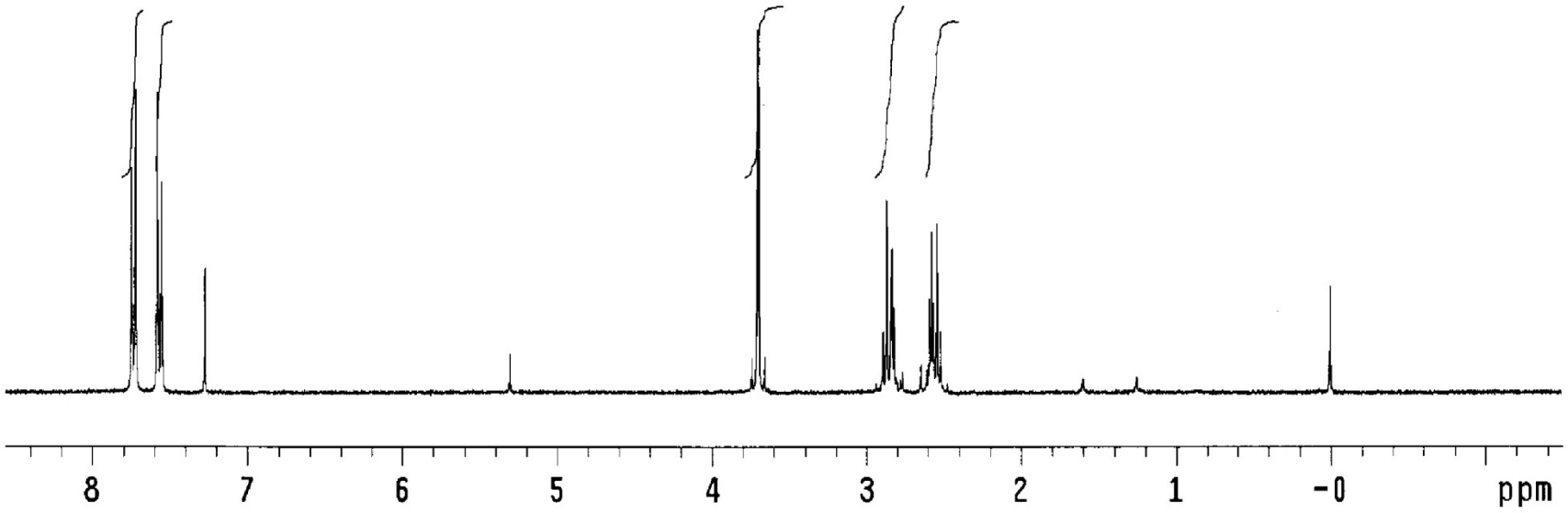


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
UNITYplus-300 "nmr2"

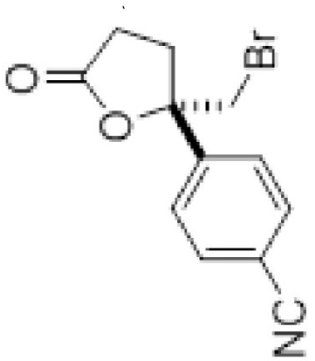
 Relax. delay 1.000 sec
 Pulse 15.0 degrees
 Acq. time 3.813 sec
 Width 4196.4 Hz
 8 repetitions
OBSERVE   H1, 300.1390285 MHz
DATA PROCESSING
 Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 38 sec

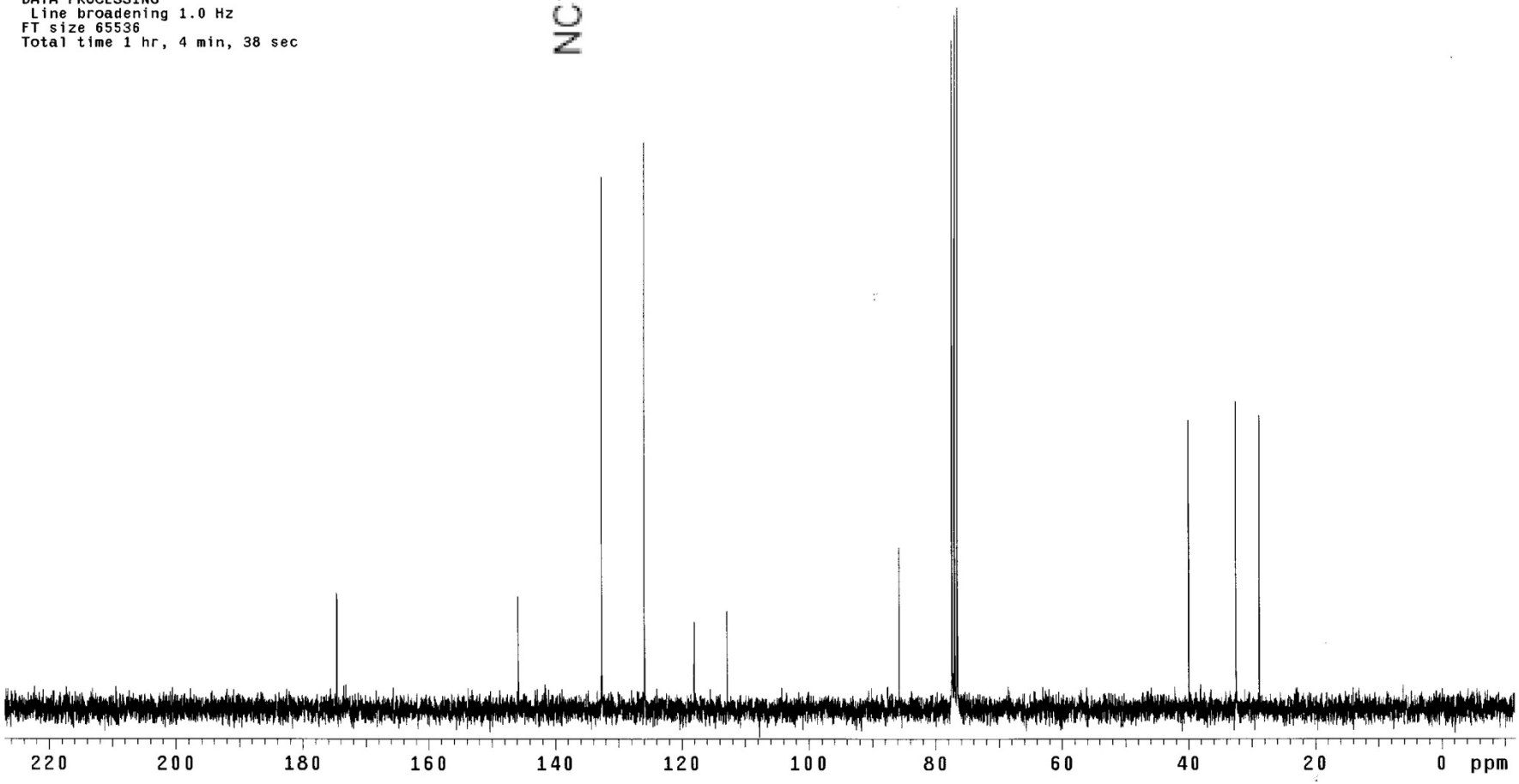




Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr2"

Relax. delay 2.000 sec
Pulse 36.0 degrees
Acq. time 1.777 sec
Width 18009.9 Hz
424 repetitions
OBSERVE C13, 75.4700211 MHz
DECOUPLE H1, 300.1409259 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
Single precision data
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 4 min, 38 sec

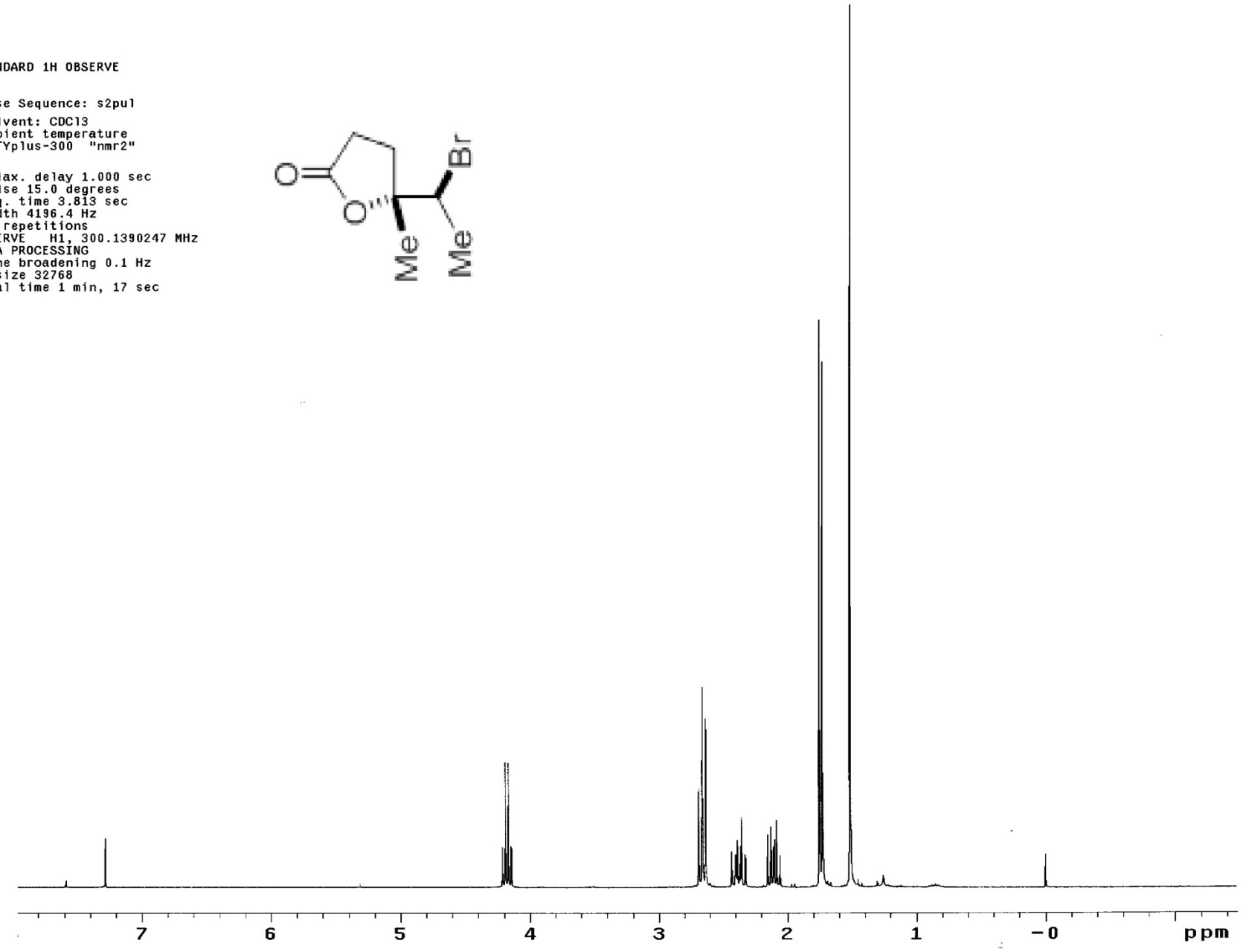




STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr2"

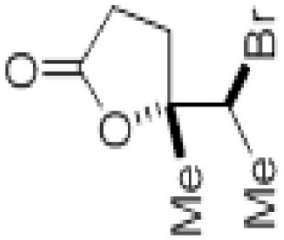
Relax. delay 1.000 sec
Pulse 15.0 degrees
Acq. time 3.813 sec
Width 4196.4 Hz
16 repetitions
OBSERVE H1, 300.1390247 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 1 min, 17 sec

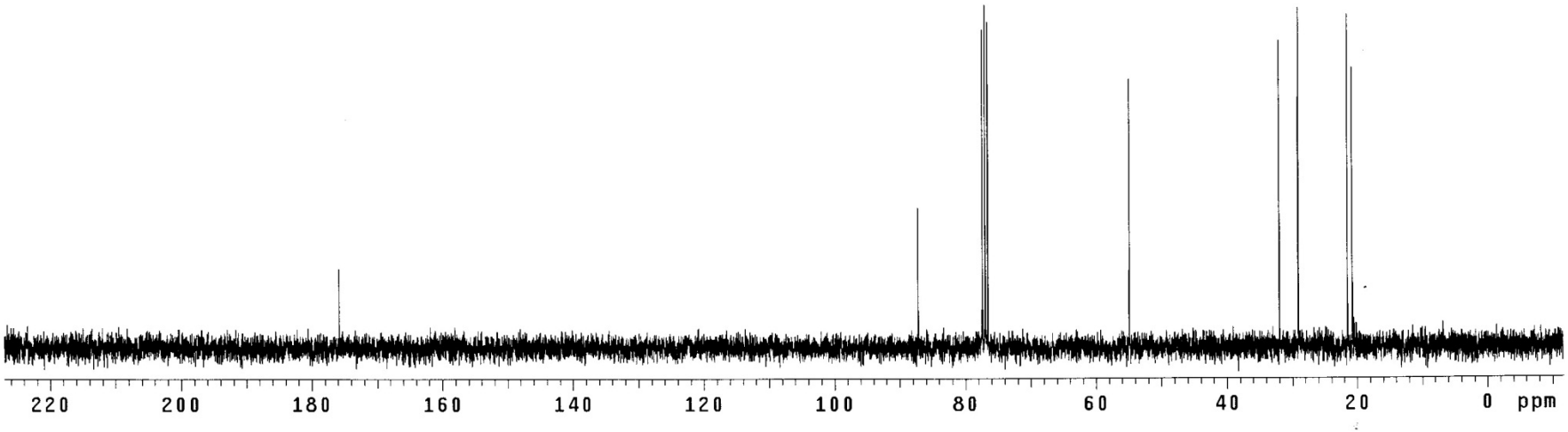


13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
UNITYplus-300 "nmr2"

 Relax. delay 2.000 sec
 Pulse 36.0 degrees
 Acq. time 1.777 sec
 Width 18009.9 Hz
 84 repetitions
OBSERVE C13, 75.4700233 MHz
DECOUPLE H1, 300.1409259 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
Single precision data
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 4 min, 38 sec

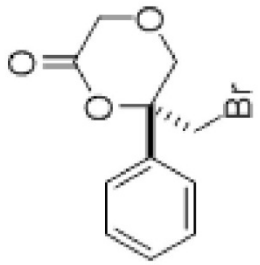


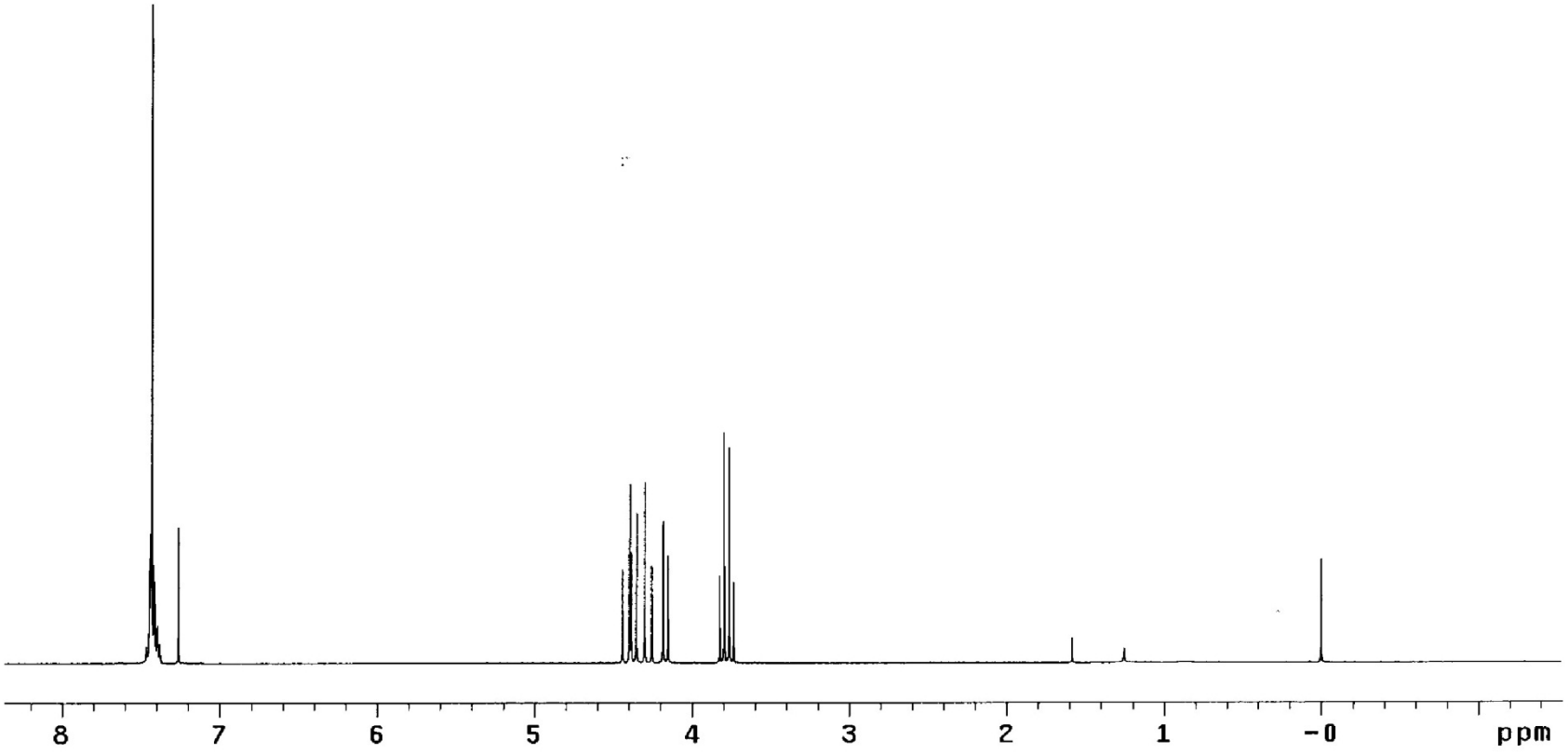


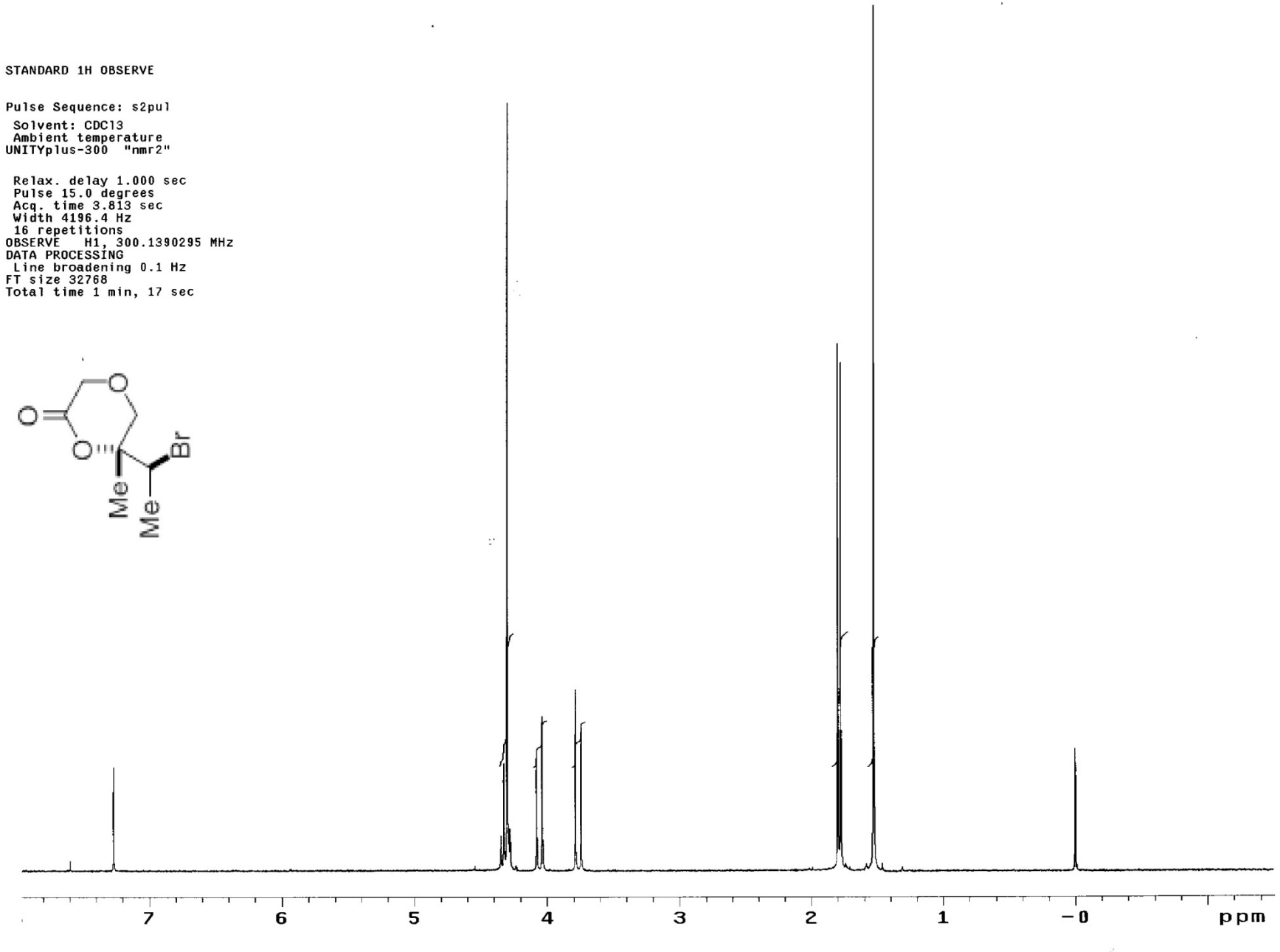
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "nmr6"

Relax. delay 2.000 sec
Pulse 16.4 degrees
Acq. time 2.856 sec
Width 5602.2 Hz
16 repetitions
OBSERVE H1, 400.2669774 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 1 min, 20 sec



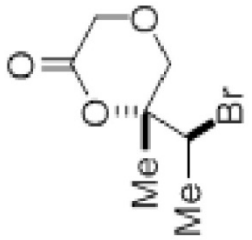


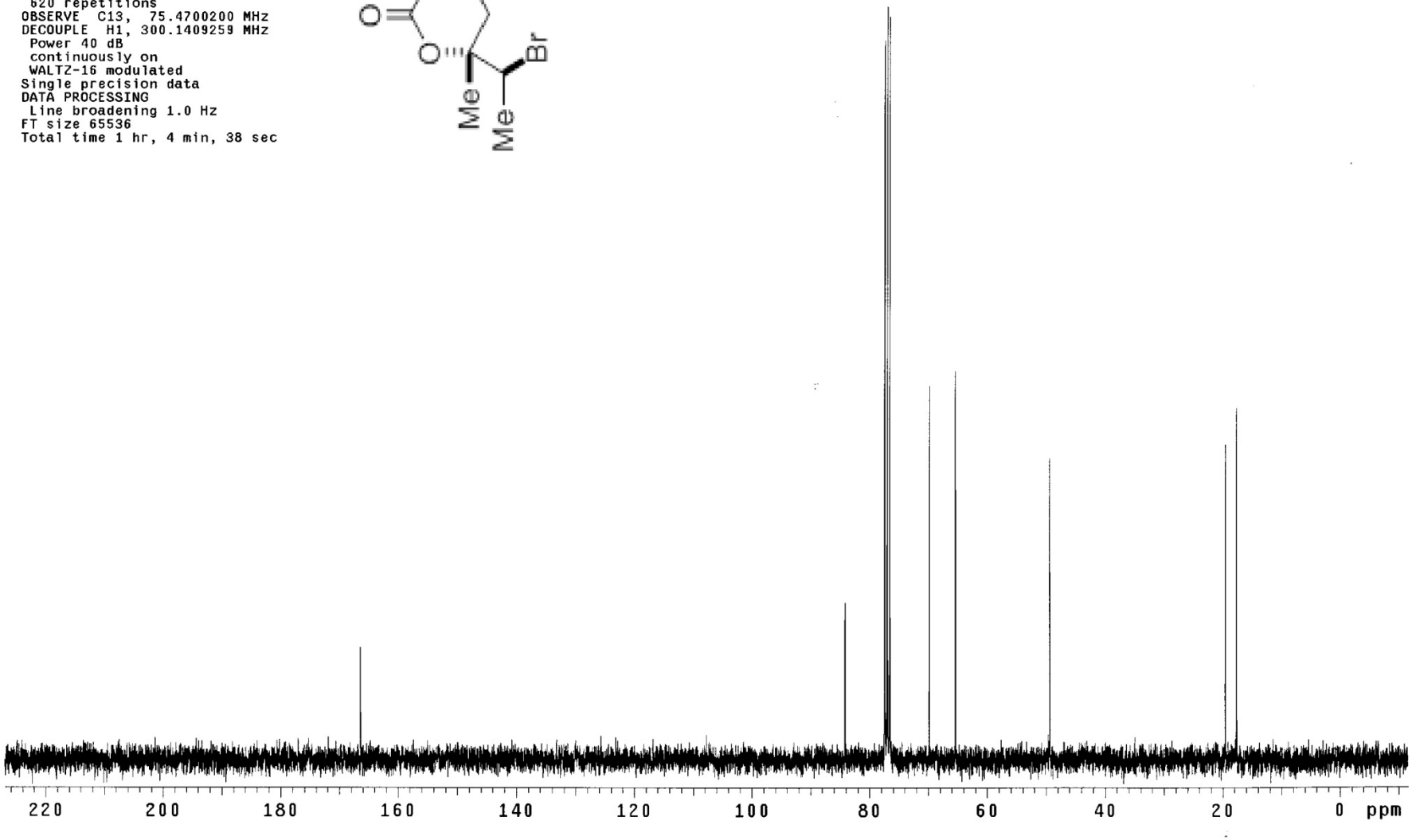

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr2"
Relax. delay 1.000 sec
Pulse 15.0 degrees
Acq. time 3.813 sec
Width 4196.4 Hz
16 repetitions
OBSERVE H1, 300.1390295 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 1 min, 17 sec
O
O
O
Br
Me
Me
7
6
5
4
3
2
1
−0
ppm

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
UNITYplus-300 "nmr2"

Relax. delay 2.000 sec
Pulse 36.0 degrees
Acq. time 1.777 sec
Width 18009.9 Hz
620 repetitions
OBSERVE C13, 75.4700200 MHz
DECOUPLE H1, 300.1409259 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
Single precision data
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 4 min, 38 sec

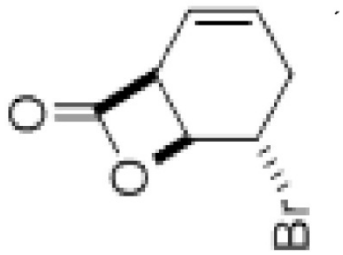


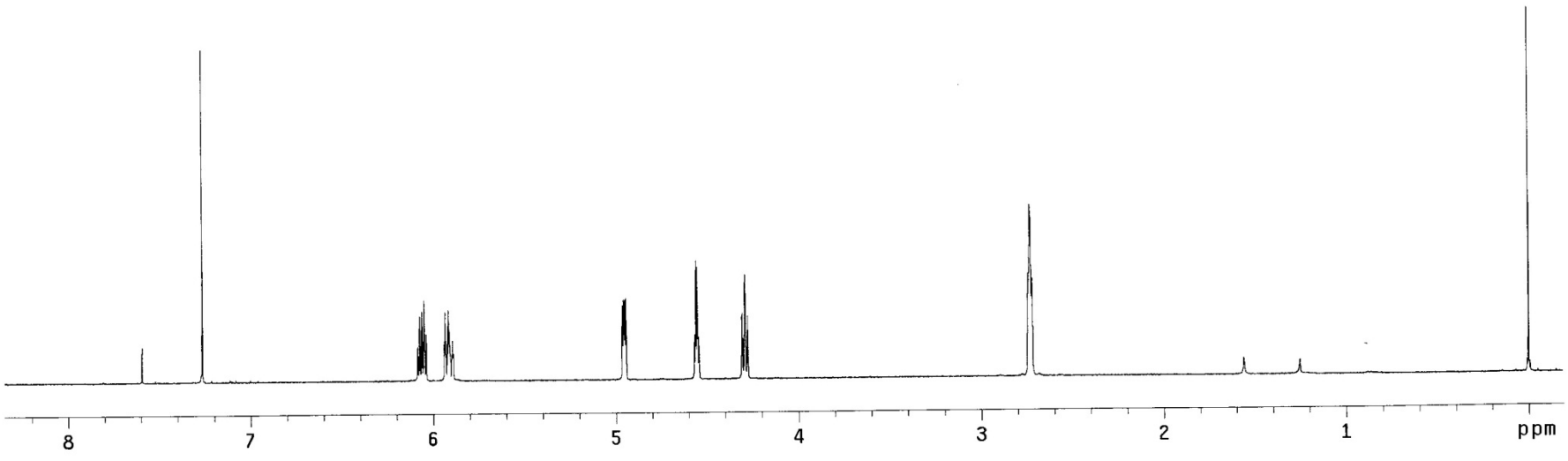


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "nmr6"

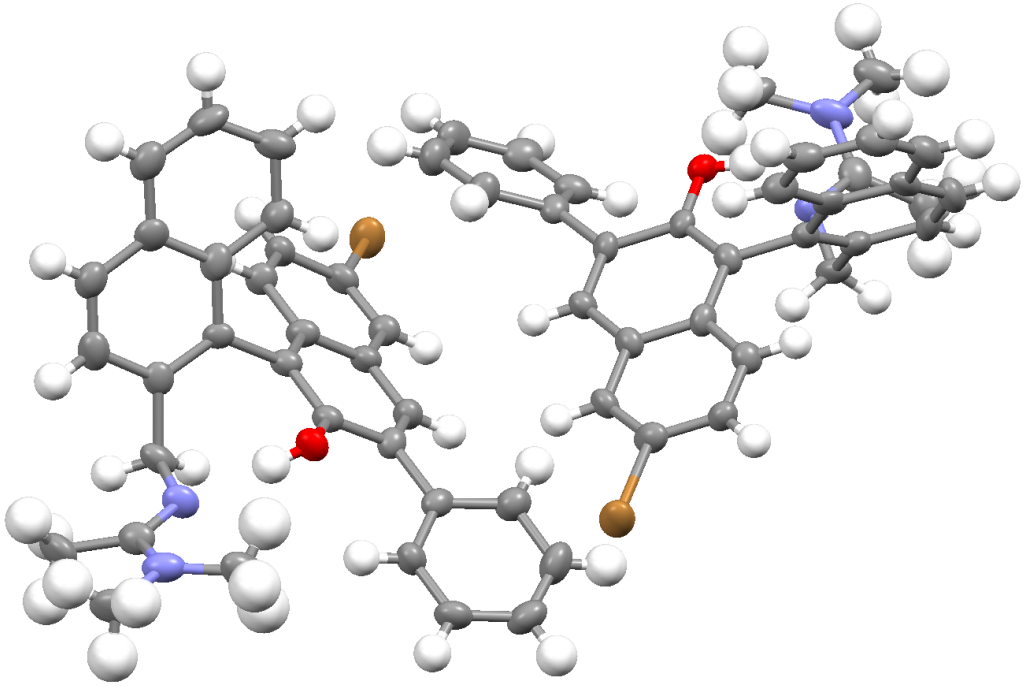
Relax. delay 2.000 sec
Pulse 16.4 degrees
Acq. time 2.856 sec
Width 5602.2 Hz
16 repetitions
OBSERVE H1, 400.2669767 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 0 sec



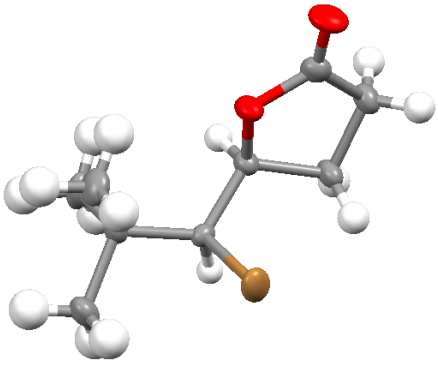


X-RAY Crystal Structures

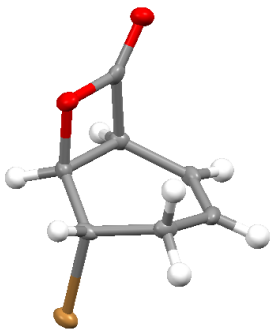
Compound **6-bromo-5**



Compound **7e**



Compound **13**



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