

Supporting Information

Zirconium(IV)- and Hafnium(IV)-Catalyzed Highly Enantioselective Epoxidation of Homoallylic and Bishomoallylic Alcohols

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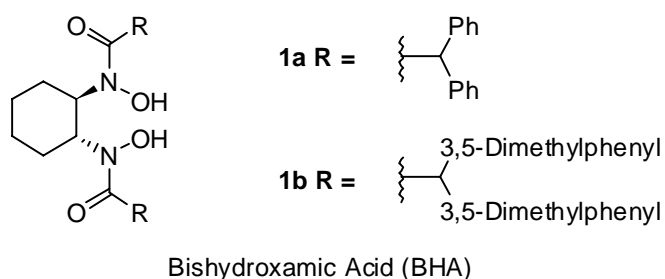
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General

Infrared (IR) spectra were recorded on a Nicolet 20 SXB FTIR. ¹H NMR spectra were recorded on Bruker DMX Model 500 (500 MHz) spectrometers. Chemical shift values (δ) are expressed in ppm downfield relative to internal standard (tetramethylsilane at 0 ppm). Multiplicities are indicated as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet) and br (broad). ¹³C NMR spectra were recorded on a Bruker DMX Model 500 (125 MHz) spectrometer and are expressed in ppm using solvent as the internal standard (CDCl₃ at 77.0 ppm). Analytical gas-liquid chromatography (GLC) was performed on a Shimadzu GC-17A instrument equipped with a flame ionization detector and capillary columns of β -TA (0.25mm \times 20m), γ -TA (0.25mm \times 20m), β -DM (0.25mm \times 20m) and β -DP (0.25mm \times 20m) from Chiraldex using nitrogen as carrier gas. High-performance liquid chromatography (HPLC) was performed on a Varian ProStar Series equipped with a variable wavelength detector using chiral stationary columns (Chiracel AD-H, OB-H or OD-H, 0.46 cm \times 25 cm) from Daicel. Optical rotations were measured on a JASCO DIP-1000 digital polarimeter. High-resolution electro spray ionization (HRMS-ESI) mass spectra were obtained from the Mass Spectrometry Lab of University of Illinois at Urbana-Champaign.

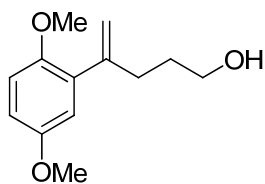
All reactions were carried out in flame-dried glassware with nitrogen atmosphere and magnetic stirring unless otherwise noted. Analytical thin-layer chromatography (TLC) was performed on Merck pre-coated TLC plates (silica gel 60 GF254, 0.25 mm). Flash chromatography was performed on silica gel E. Merck 9385 or silica gel 60 extra pure (for all the bis-hydroxamic acids). Dichloromethane (CH₂Cl₂) and toluene (PhCH₃) were purchased from J.T.Baker as Low water and purified with MBRAUN MB-SPS Solvent Purifier System prior to use. Zr(O*t*Bu)₄ and Hf(O*t*Bu)₄ were purchased from Strem and stored and handled in the glove box. Powdered molecular sieves were purchased from Sigma-Aldrich and activated with microwave oven. 1,3-Dimethyl-3,4,5,6-tetrahydro-2(1H)-pyrimidinone (DMPU) was purchased from Sigma-Aldrich and distilled from CaH₂ prior to use. Homoallylic alcohols and products in Tables 1 and 2 have been previously isolated and characterized. References can be found elsewhere. All other reagents and starting materials, unless otherwise noted, were purchased from commercial vendors and used without further purification.



Preparation of Ligands 1a and 1b: Refer to previous reports of our group.¹⁻³ They are commercially available in both enantiomer forms.

Preparation of substrates

Substrates **2a**, **2b**, **2g**, **2h**, **2i** and **4h** were purchased from commercial vendors and stored on 4Å molecular sieves pellets after received. Substrates **2c**,⁴ **2d**,⁵ **2e**,⁴ **2f**,⁶ **2j**,⁵ **4a-4c**,⁷ **4d**,^{8,9} **4e-4g**⁷ were synthesized according to reported procedures.



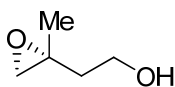
4-(2,5-dimethoxyphenyl)pent-4-en-1-ol (4e): Yield 54%. ^1H NMR (500 MHz, CDCl_3): δ 6.82-6.70 (m, 3 H), 5.17 (d, $J = 1$ Hz, 1 H), 5.05 (d, $J = 1.5$ Hz, 1 H), 3.77 (s, 6 H), 3.64 (m, 2 H), 2.56 (t, $J = 7.5$ Hz, 2 H), 1.64 (m, 2 H), 1.50 (br, 1 H); ^{13}C NMR (125 MHz, CDCl_3): δ 153.5, 150.7, 148.0, 132.9, 116.3, 114.7, 112.4, 111.9, 62.5, 56.2, 55.7, 32.5, 31.0; $[\text{M}+\text{H}]^+$: $\text{C}_{13}\text{H}_{19}\text{O}_3^+$, calc. 223.1334, found 223.1330.

General procedure for asymmetric epoxidation of homoallylic alcohols.

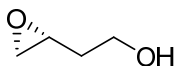
$[\text{Zr}(\text{O}t\text{Bu})_4]$ (4.0 μL , 0.0100 mmol) or $[\text{Hf}(\text{O}t\text{Bu})_4]$ (4.0 μL , 0.0099 mmol) was added to a mixture of **1a** (5.4 mg, 0.0101 mmol), DMPU (2.6mg, 0.0203mmol), and 4Å molecular sieves powder (100mg) in toluene (0.5 mL). The catalyst mixture was stirred for 1h at RT. The resulting solution was cooled to 0 °C, and 3-methyl-3-buten-1-ol (**2a**) (43.0 mg, 0.50 mmol) and 80% cumene hydroperoxide (0.13 mL, 0.75 mmol) were added sequentially. The mixture was stirred at 0 °C for 4 h, then warmed to RT, and stirred for the next 36 hr. Methanol was then added, and the reaction mixture was stirred for 10 min at RT. The mixture was purified by flash column chromatography on silica gel to provide the epoxy alcohol **3a** (81%, 97% ee). A solution of 50% ethyl ether in pentane was used as chromatography eluent. Evaporation of solvents should be carefully carried out under 0 °C because **3a** is a volatile liquid. The same measures were performed in purification of **3b**. In all other cases, different ration of hexanes and ethyl acetate mixture solutions were used as eluents.

General procedure for asymmetric epoxidation of bishomoallylic alcohols.

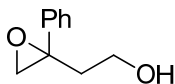
$[\text{Zr}(\text{O}t\text{Bu})_4]$ (4.0 μL , 0.0100 mmol) or $[\text{Hf}(\text{O}t\text{Bu})_4]$ (4.0 μL , 0.0099 mmol) was added to a mixture of **1a** (5.4 mg, 0.0101 mmol), DMPU (2.6mg, 0.0203mmol), and corresponding molecular sieves powder (100mg) in toluene (0.5 mL). The catalyst mixture was stirred for 1h at RT. 4-phenyl-4-penten-1-ol (**4a**) (16.2 mg, 0.10 mmol) and 80% cumene hydroperoxide (0.025 mL, 0.15 mmol) were added sequentially. The mixture was stirred at RT for 48 h. Methanol was then added, and the reaction mixture was stirred for 10 min. The mixture was purified by flash column chromatography on silica gel with 1% triethylamine in 5:1 hexanes/ethyl acetate solution to provide the epoxy alcohol **5a** (75%, 99% ee). A solution of 15% ethyl ether in pentane was used as chromatography eluent when purifying **6h**. Evaporation of solvents should be carefully carried out under 0 °C because it is a volatile liquid.



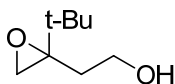
(R)-2-(2-methyloxiran-2-yl)ethanol (3a): Reaction time 40 hr, yield 61% ee 91% with Zr-BHA, yield 81% ee 97% with Hf-BHA; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 3.79-3.67 (m, 2 H), 2.83 (d, $J = 4.5$ Hz, 1 H), 2.65 (d, $J = 4.5$ Hz, 1 H), 2.12 (m, 1 H), 2.00-1.94 (m, 1 H), 1.90-1.84 (m, 1 H), 1.39 (s, 3 H); It is consistent with literature value.¹ Optical rotation: $[\alpha]_{\text{D}}^{23} -7.9^\circ$ (c 0.92, CHCl_3) (97% ee); Chiral GC (Chiraldex γ -TA): Condition: injection temperature 100 $^\circ\text{C}$, column temp. = 70 $^\circ\text{C}$, injection pressure = 100 kpa, detector temperature 250 $^\circ\text{C}$; result: 8.0min (major), 10.0min (minor).



(R)-2-(oxiran-2-yl)ethanol (3b): Reaction time 40 hr, yield 37% ee 63% with Hf-BHA; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 3.81 (t, $J = 6.0$ Hz, 2 H), 3.13-3.10 (m, 1 H), 2.82 (t, $J = 4.5$ Hz, 1 H), 2.60 (m, 1 H), 2.02-1.97 (m, 1 CH_2 and 1 OH), 1.73-1.70 (m, 1 H); It is consistent with literature value.¹⁰ Optical rotation: $[\alpha]_{\text{D}}^{24} +4.2^\circ$ (c 0.74, CHCl_3) (63% ee);¹¹ Chiral GC (Chiraldex γ -TA): Condition: injection temperature 100 $^\circ\text{C}$, column temp. = 70 $^\circ\text{C}$, injection pressure = 100 kpa, detector temperature 250 $^\circ\text{C}$; result: 6.5 min (major), 7.3 min (minor).

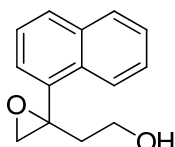


2-(2-phenyloxiran-2-yl)ethanol (3c): Reaction time 40 hr, yield 67% ee 92% with Zr-BHA, yield 69% ee 98% with Hf-BHA; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 7.40-7.28 (m, 5 H), 3.78-3.70 (m, 2 H), 3.13 (d, $J = 5.0$ Hz, 2 H), 2.78 (d, $J = 5.0$ Hz, 1 H), 2.54-2.48 (m, 1 H), 2.15-2.09 (m, 1 H), 2.00 (t, $J = 5.5$ Hz, 1 H); It is consistent with literature value.¹² Optical rotation: $[\alpha]_{\text{D}}^{27} +12.2^\circ$ (c 0.67, CHCl_3) (92% ee); Chiral HPLC (Chiracel AD-H): Condition: 95:5 Hexanes/2-Propanol, flow rate 1.0mL/min; result: 13.9 min (minor), 14.7 min (major).

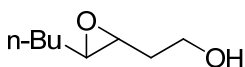


2-(2-tert-butyloxiran-2-yl)ethanol(3d): Reaction time 40 hr, yield 67% ee 63% with Zr-BHA, yield 70% ee 71% with Hf-BHA; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 3.60-3.55 (m, 2 H), 2.88 (d, $J = 3.8$ Hz, 1 H), 2.83 (d, $J = 3.8$ Hz, 1 H), 2.55-2.52 (m, 1 H), 2.27-2.20 (m,

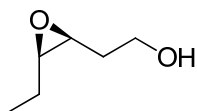
1 H), 1.97-1.91 (m, 1 H), 0.95 (s, 9 H). ^{13}C NMR (125 MHz, CDCl_3): δ 64.0, 58.8, 48.3, 33.8, 30.2, 25.6; $[\text{M}+\text{Na}]^+$: $\text{C}_8\text{H}_{16}\text{O}_2\text{Na}$, calc. 167.1048, found 167.1044; Optical rotation: $[\alpha]_{\text{D}}^{27} +20.6^\circ$ (c 0.88, CHCl_3) (71% ee); Chiral GC (Chiraldex β -DP): Condition: injection temperature 120 $^\circ\text{C}$, column temperature 90 $^\circ\text{C}$, column pressure 100 kPa, detector temperature 250 $^\circ\text{C}$; result: 16.3 min (major), 17.9 min (minor).



2-(2-(naphthalen-1-yl)oxiran-2-yl)ethanol(3e): Reaction time 40 hr, yield 31%, ee 91% with Hf-BHA; ^1H NMR (500 MHz, CDCl_3): δ 8.11 (d, $J = 7.1$ Hz, 1 H), 7.88 (d, $J = 7.5$ Hz, 1H), 7.82 (d, $J = 8.2$ Hz, 1 H), 7.60-7.44 (m, 4 H), 3.77-3.65 (m, 2 H), 3.33 (d, $J = 5.0$ Hz, 1 H), 2.98 (d, $J = 5.0$ Hz, 1 H), 2.46-2.41 (m, 1 H), 2.30-2.24 (m, 1 H), 2.15 (br, 1H). It is consistent with literature value.¹³ Chiral HPLC (Chiracel AD-H): Condition: 95:5 Hexanes/2-Propanol, flow rate 0.5mL/min; result: 36.2 min (major), 37.9 min (minor).

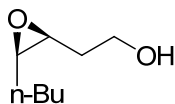


2-(3-butylloxiran-2-yl)ethanol(3f): Reaction time 40 hr, yield 47% ee 73% with Hf-BHA; ^1H NMR (500 MHz, CDCl_3): δ 3.81-3.75 (m, 2 H), 2.88-2.85 (m, 1 H), 2.82-2.79 (m, 1 H), 2.03-1.96 (m, 1 H), 1.85 (br, 1 H), 1.75-1.65 (m, 1 H), 1.57-1.52 (m, 2 H), 1.45-1.34 (m, 4 H), 0.92 (t, $J = 7.1$ Hz, 3 H). ^{13}C NMR (125 MHz, CDCl_3): δ 60.1, 58.2, 56.9, 34.1, 31.6, 28.1, 22.5, 14.0; $[\text{M}+\text{Na}]^+$: $\text{C}_8\text{H}_{16}\text{O}_2\text{Na}$, calc. 167.1048, found 167.1048; Optical rotation: $[\alpha]_{\text{D}}^{26} +30.8^\circ$ (c 0.66, CHCl_3) (71% ee); Chiral GC (Chiraldex β -DP): Condition: injection temperature 120 $^\circ\text{C}$, column temperature 80 $^\circ\text{C}$ for 60 min, and then heat to 100 $^\circ\text{C}$ on the rate of 2 $^\circ\text{C}/\text{min}$, column pressure 100 kPa, detector temperature 250 $^\circ\text{C}$; result: 81.9 min (minor), 82.9 min (major).

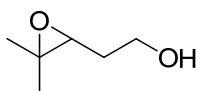


2-((2S,3R)-3-ethyloxiran-2-yl)ethanol(3g): Reaction time 40 hr, yield 45%, ee 93% with Zr-BHA, yield 82% ee 94% with Hf-BHA; ^1H NMR (500 MHz, CDCl_3): δ 3.91-3.85 (m, 2 H), 3.13-3.10 (m, 1 H), 2.95-2.92 (m, 1 H), 1.92-1.86 (m, 1 H), 1.76-1.66 (m, 2 H (including 1 OH)), 1.65-1.49 (m, 2 H), 1.06 (t, $J = 7.5$ Hz, 3 H); It is consistent with literature value.² Optical rotation: $[\alpha]_{\text{D}}^{25} -28.3^\circ$ (c 0.90, CHCl_3) (93% ee);¹¹ Chiral GC

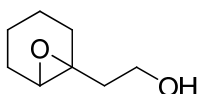
(Chiraldex γ -TA)²: Condition: injection temperature 120 °C, column temperature 90 °C, column pressure 100 kPa, detector temperature 250 °C; result: 7.8 min (major), 8.8 min (minor).



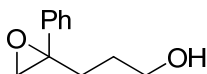
2-((2*S*,3*R*)-3-butyloxiran-2-yl)ethanol(3h): Reaction time 40 hr, yield 80% ee 92% with Zr-BHA, yield 83% ee 96% with Hf-BHA; ¹H NMR (500 MHz, CDCl₃): δ 3.92-3.84 (m, 2 H), 3.12-3.09 (m, 1 H), 2.97-2.94 (m, 1 H), 1.94-1.86 (m, 1 H), 1.79 (br, 1 H), 1.76-1.67 (m, 1 H), 1.60-1.36 (m, 6 H), 0.93 (t, $J = 6.5$ Hz, 3 H); It is consistent with literature value.² Optical rotation: $[\alpha]_D^{25} -19.7^\circ$ (c 0.74, CHCl₃) (95% ee); Chiral GC (Chiraldex γ -TA): Condition: injection temperature 120 °C, column temperature 80 °C, column pressure 100 kPa, detector temperature 250 °C; result: 44.6 min (major), 50.0 min (minor).



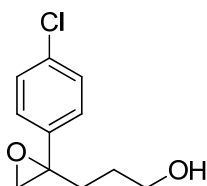
2-(3,3-dimethyloxiran-2-yl)ethanol(3i): Reaction time 40 hr, yield 41% ee 71% with Hf-BHA; ¹H NMR (500 MHz, CDCl₃): δ 3.91-3.82 (m, 2 H), 2.93-2.90 (m, 1 H), 1.96-1.87 (m, 1 H), 1.79-1.69 (m, 2 H (including 1 OH)), 1.34 (s, 3 H), 1.30 (s, 3 H); It is consistent with literature value.¹⁰ Chiral GC (Chiraldex γ -TA)¹³: Condition: injection temperature 100 °C, column temperature 70 °C, column pressure 100 kPa, detector temperature 250 °C; result: 17.0 min (major), 19.6 min (minor).



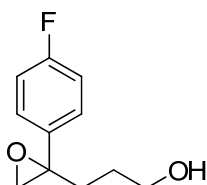
2-(7-oxabicyclo[4.1.0]heptan-1-yl)ethanol(3j): Reaction time 40 hr, yield 72% ee 76% with Zr-BHA, yield 81% ee 89% with Hf-BHA; ¹H NMR (500 MHz, CDCl₃): δ 3.77-3.70 (m, 2 H), 3.19 (d, $J = 3.0$ Hz, 1 H), 2.32-2.30 (m, 1 H), 2.03-1.69 (m, 6 H), 1.50-1.39 (m, 2 H), 1.35-1.19 (m, 2 H); It is consistent with literature value.¹⁴ Optical rotation: $[\alpha]_D^{26} +23.7^\circ$ (c 0.95, CHCl₃) (87% ee); Chiral GC (Chiraldex β -DP): Condition: injection temperature 130 °C, column temperature 110 °C, column pressure 100 kPa, detector temperature 250 °C; result: 21.7 min (minor), 22.4 min (major).



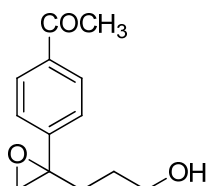
3-(2-phenyloxiran-2-yl)propan-1-ol(5a): Reaction time 48 hr. Yield 75%; ^1H NMR (500 MHz, CDCl_3): δ 7.40-7.26 (m, 5 H), 3.67-3.63 (m, 2 H), 3.01 (d, $J = 5.0$ Hz, 1 H), 2.76 (d, $J = 5.5$ Hz, 1 H), 2.45-2.40 (m, 1 H), 1.80-1.74 (m, 1 H), 1.68-1.63 (m, 3 H (including 1 OH)); It is consistent with literature value.¹⁵ 99% e.e., Optical rotation: $[\alpha]_{\text{D}}^{28} -3.4^\circ$ (c 0.68, CHCl_3) (97% ee); Chiral HPLC (Chiracel OB-H): Condition: 95:5 Hexanes/2-Propanol, flow rate 1.0 mL/min; result: 15.6 min (major), 26.6 min (minor).



3-(2-(4-chlorophenyl)oxiran-2-yl)propan-1-ol(5b): Reaction time 48 hr. Yield 57%; ^1H NMR (500 MHz, CDCl_3): δ 7.31 (apparent singlet, 4 H), 3.65-3.61 (m, 2 H), 3.00 (d, $J = 5.0$ Hz, 1 H), 2.72 (d, $J = 5.0$ Hz, 1 H), 2.43-2.36 (m, 1 H), 1.79-1.72 (m, 2 H (including 1 OH)), 1.66-1.59 (m, 2 H); ^{13}C NMR (125 MHz, CDCl_3): δ 138.1, 133.3, 128.6, 127.3, 62.2, 59.7, 56.1, 31.5, 27.7; $[\text{M}+\text{Na}]^+$: $\text{C}_{11}\text{H}_{13}\text{ClO}_2\text{Na}$, calc. 235.0502, found 235.0498; 97% e.e.; Chiral HPLC (Chiracel OB-H): Condition: 99:1 Hexanes/2-Propanol, flow rate 1.0 mL/min; result: 59.9 min (minor), 64.9 min (major).

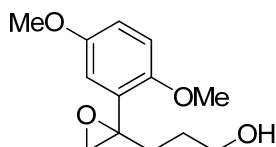


3-(2-(4-fluorophenyl)oxiran-2-yl)propan-1-ol(5c): Reaction time 48 hr. Yield 73%; ^1H NMR (500 MHz, CDCl_3): δ 7.36-7.33 (m, 2 H), 7.05-7.00 (m, 2 H), 3.64 (t, $J = 6.3$ Hz, 2 H), 3.00 (d, $J = 5.5$ Hz, 1 H), 2.73 (d, $J = 5.0$ Hz, 1 H), 2.42-2.35 (m, 1 H), 1.80-1.73 (m, 1 H), 1.66-1.57 (m, 3 H (including 1 OH)); ^{13}C NMR (125 MHz, CDCl_3): δ 127.6, 127.6, 115.4, 115.2, 62.2, 59.7, 56.1, 31.7, 27.8; $[\text{M}+\text{Na}]^+$: $\text{C}_{11}\text{H}_{13}\text{FO}_2\text{Na}$, calc. 219.0797, found 219.0788; 97% e.e.; Chiral HPLC (Chiracel OB-H): Condition: 95:5 Hexanes/2-Propanol, flow rate 1.0 mL/min; result: 17.2 min (minor), 19.7 min (major).

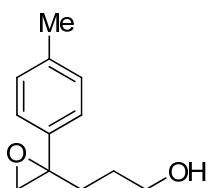


1-(4-(2-(3-hydroxypropyl)oxiran-2-yl)phenyl)ethanone (5d): Reaction time 48 hr.

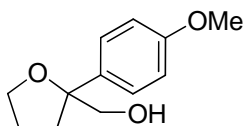
Yield 79%; ^1H NMR (500 MHz, CDCl_3): δ 7.94-7.92 (m, 2 H), 7.48-7.45 (m, 2 H), 3.65-3.62 (m, 2 H), 3.04 (d, $J = 5.5$ Hz, 1 H), 2.73 (d, $J = 5.5$ Hz, 1 H), 2.59 (s, 3 H), 2.49-2.44 (m, 1 H), 1.80-1.74 (m, 1 H), 1.66-1.60 (m, 2 H (including 1 OH)); ^{13}C NMR (125 MHz, CDCl_3): δ 197.6, 145.0, 136.4, 128.5, 126.0, 62.2, 59.9, 56.2, 31.3, 27.7, 26.6; 97% e.e.; Chiral HPLC (Chiracel OB-H): Condition: 95:5 Hexanes/2-Propanol, flow rate 1.0 mL/min; result: 37.3 min (major), 43.5 min (minor).



3-(2-(2,5-dimethoxyphenyl)oxiran-2-yl)propan-1-ol(5e): Yield 25%; ^1H NMR (500 MHz, CDCl_3): δ 6.94-6.92 (m, 1 H), 6.79-6.78 (m, 2 H), 3.81 (s, 3 H), 3.77 (s, 3 H), 3.67-3.60 (m, 2 H), 2.99 (d, $J = 5.0$ Hz, 1 H), 2.76 (d, $J = 5.0$ Hz, 1 H), 2.35-2.28 (m, 1 H), 1.75-1.68 (m, 1 H), 1.64-1.56 (m, 2 H); ^{13}C NMR (125 MHz, CDCl_3): δ 129.2, 127.9, 116.1, 113.9, 113.5, 111.4, 62.7, 59.2, 55.8, 55.8, 54.5, 32.0, 28.0; $[\text{M}+\text{Na}]^+$: $\text{C}_{13}\text{H}_{18}\text{O}_4\text{Na}$, calc. 261.1103, found 261.1108; 97% e.e.; Chiral HPLC (Chiracel OB-H): Condition: 95:5 Hexanes/2-Propanol, flow rate 1.0 mL/min; result: 32.7 min (minor), 36.7 min (major).

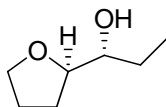


3-(2-p-tolyloxiran-2-yl)propan-1-ol(5f): Reaction time 48 hr. Yield 53%; ^1H NMR (500 MHz, CDCl_3): δ 7.29-7.24 (m, 2 H), 7.16-7.14 (m, 2 H), 3.65-3.61 (m, 2 H), 2.98 (d, $J = 5.5$ Hz, 1 H), 2.74 (d, $J = 5.5$ Hz, 1 H), 2.42-2.36 (m, 1 H), 2.34 (s, 3 H), 1.79-1.73 (m, 2 H (including 1 OH)), 1.66-1.61 (m, 2 H); ^{13}C NMR (125 MHz, CDCl_3): δ 137.2, 136.5, 129.1, 125.7, 62.3, 60.0, 56.1, 31.8, 27.9; $[\text{M}+\text{Na}]^+$: $\text{C}_{12}\text{H}_{16}\text{O}_2\text{Na}$, calc. 215.1048, found 215.1045; 97% e.e.; Chiral HPLC (Chiracel OB-H): Condition: 98:2 Hexanes/2-Propanol, flow rate 1.0 mL/min; result: 28.8 min (minor), 30.1 min (major).



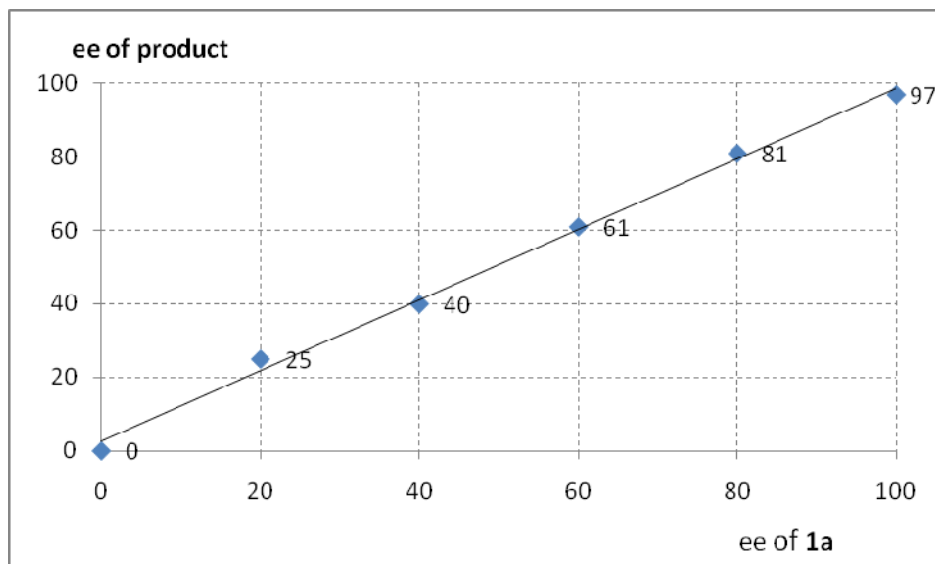
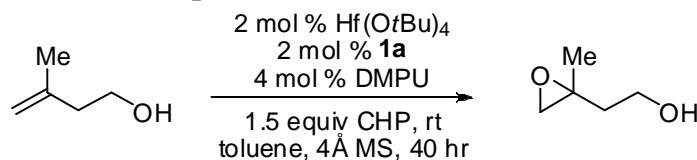
(2-(4-methoxyphenyl)tetrahydrofuran-2-yl)methanol(6g): Reaction time 48 hr, yield 47%; ^1H NMR (500 MHz, CDCl_3): δ 7.31-7.28 (m, 2 H), 6.90-6.86 (m, 2 H), 4.02-3.97 (m, 1 H), 3.94-3.89 (m, 1 H), 3.79 (s, 3 H), 3.65-3.57 (m, 2 H), 2.34-2.27 (m, 1 H), 2.10-2.04 (m, 1 H), 2.00-1.93 (m, 2 H (including 1 OH)), 1.88-1.81 (m, 1 H); ^{13}C NMR (125

MHz, CDCl₃): δ 158.6, 136.2, 126.5, 113.7, 87.0, 69.1, 68.3, 55.3, 33.9, 26.1; 59% e.e.; Chiral HPLC (Chiracel OD-H): Condition: 95:5 Hexanes/2-Propanol, flow rate 1.0 mL/min; result: 9.4 min (major), 12.1 min (minor).



(R)-1-((R)-tetrahydrofuran-2-yl)propan-1-ol(6h): Reaction time 72 hr; Yield 41%; ¹H NMR (500 MHz, CDCl₃): δ 3.86-3.73 (m, 3 H), 3.35-3.30 (m, 1 H), 2.18 (br, 1 H), 1.97-1.89 (m, 3 H), 1.67-1.60 (m, 1 H), 1.57-1.41 (m, 2 H), 1.01 (t, 3 H, $J = 7.5$ Hz); It is consistent with literature value.¹⁶ 95% e.e.; Chiral GC (Chiraldex γ -TA): Condition: injection temperature 100 °C, column temp. = 60 °C, injection pressure = 80 kpa, detector temperature 250 °C; result: 19.5min (minor), 20.6 min (major). Retention time of diastereomer **6h'**: 20.9 min.

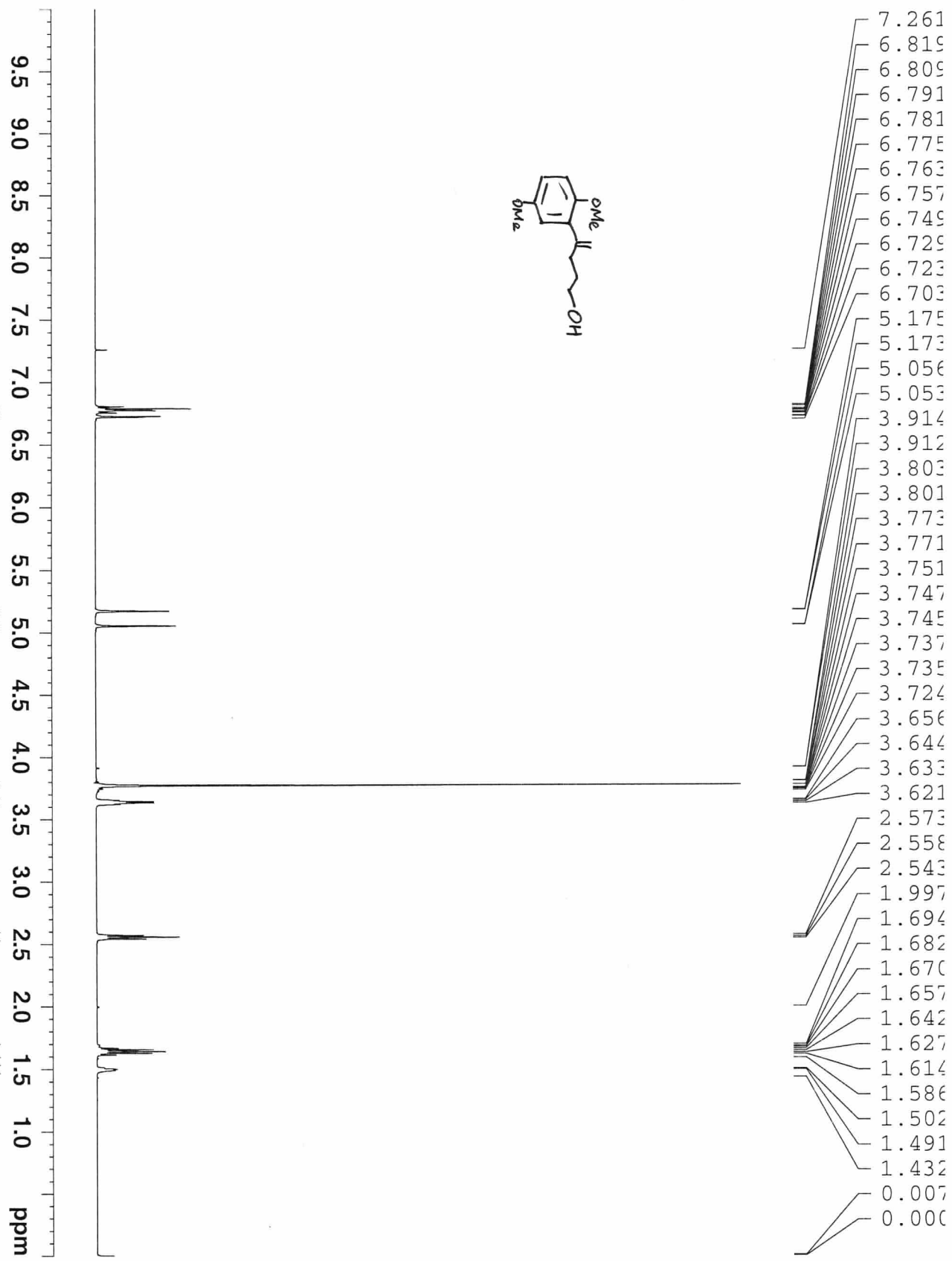
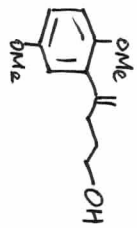
Results of non-linear effect experiment:



References

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- (16) Ahn, Y.; Cohen, T., *J. Org. Chem.* **1994**, *59*, 3142.



Chemical Shift (ppm)	Integration
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6.781	
6.775	
6.763	
6.757	
6.749	
6.729	
6.723	
6.703	
5.175	
5.173	
5.056	
5.053	
3.914	
3.912	
3.803	
3.801	
3.773	
3.771	
3.751	
3.747	
3.745	
3.737	
3.735	
3.724	
3.656	
3.644	
3.633	
3.621	
2.573	
2.558	
2.543	
1.997	
1.694	
1.682	
1.670	
1.657	
1.642	
1.627	
1.614	
1.586	
1.502	
1.491	
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0.000	

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1.9785

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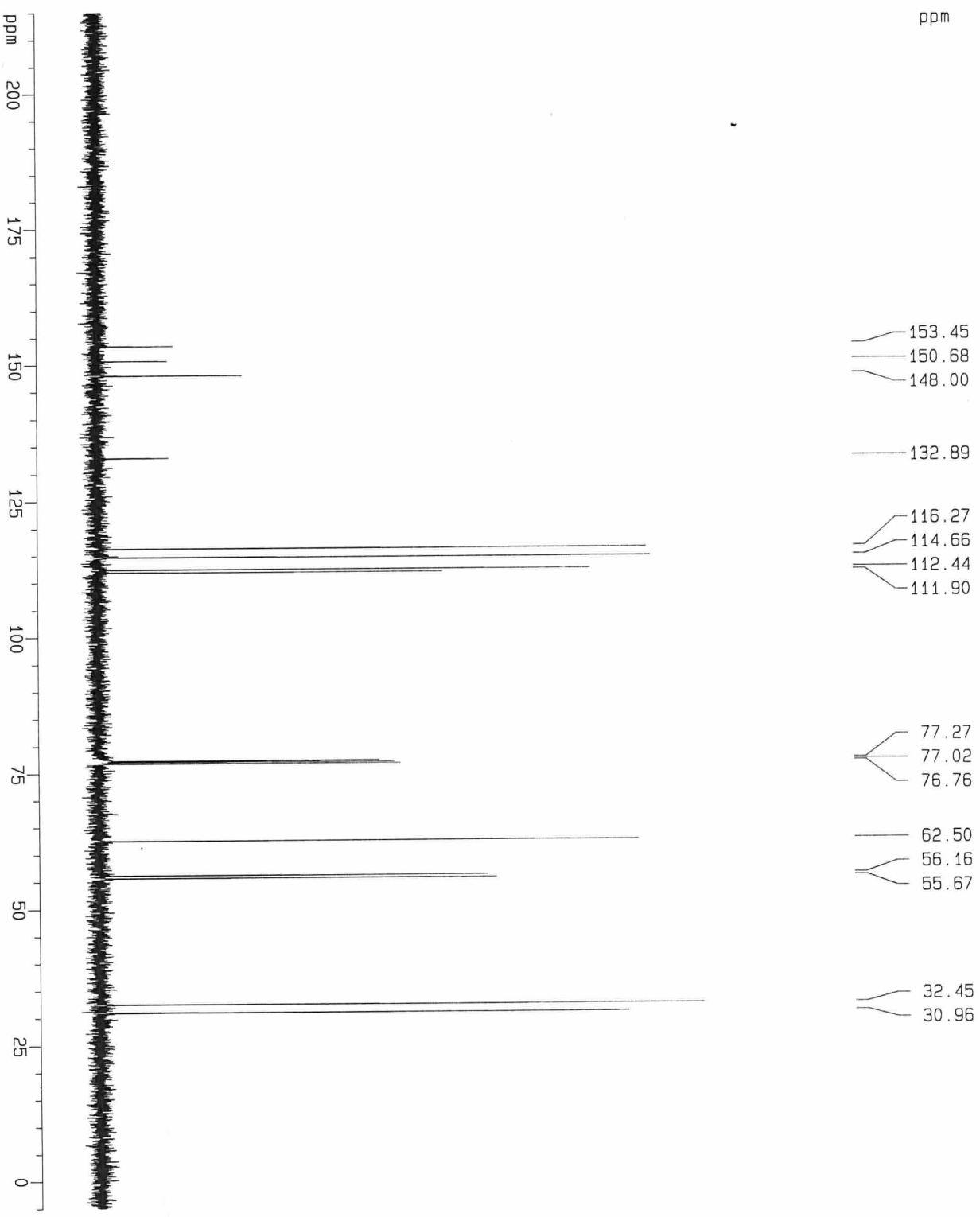
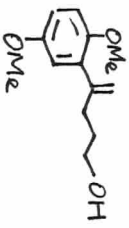
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PROCNO        1

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PULPROG      zgpg
TD            59938
SOLVENT      CDCl3
NS            6
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PL1           0.00 dB
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F2 - Processing parameters
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PC            1.00
  
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Current Data Parameters
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 EXPNO 1
 PROCNO 1

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 PULPROG zgpg30
 TD 75184
 SOLVENT CDCl3
 NS 134
 DS 0
 SMH 37593.984 Hz
 FIDRES 0.500026 Hz
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 d11 0.03000000 sec

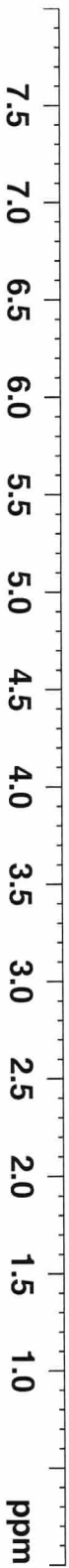
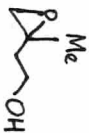
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 SF01 125.7690572 MHz

===== CHANNEL f2 =====
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 NUC2 1H
 PCPD2 90.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SF02 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577941 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10 NMR plot parameters
 CX 20.00 cm
 F1P 215.000 ppm
 F1 27037.92 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1383.33582 Hz/cm

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1.000

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1.053

0.213

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Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameter
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Time 18.22

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PROBHD spect
PULPROG zg

TD 59998
SOLVENT CDCl3

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FIDRES 0.166672 Hz
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RG 256
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TE 7.50 use
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TD0 1.00000000 se

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F2 - Processing parameters
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SF 499.8700151 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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Integral

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ppm

7
6
5
4
3
2
1

Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20090414
Time 18.25
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PULPROG zg
TD 48076
SOLVENT CDCl3
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DS 0
SMH 8012.820 Hz
FIDRES 0.166670 Hz
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RG 128
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TE 300.0 K
D1 3.00000000 sec

===== CHANNEL f1 =====

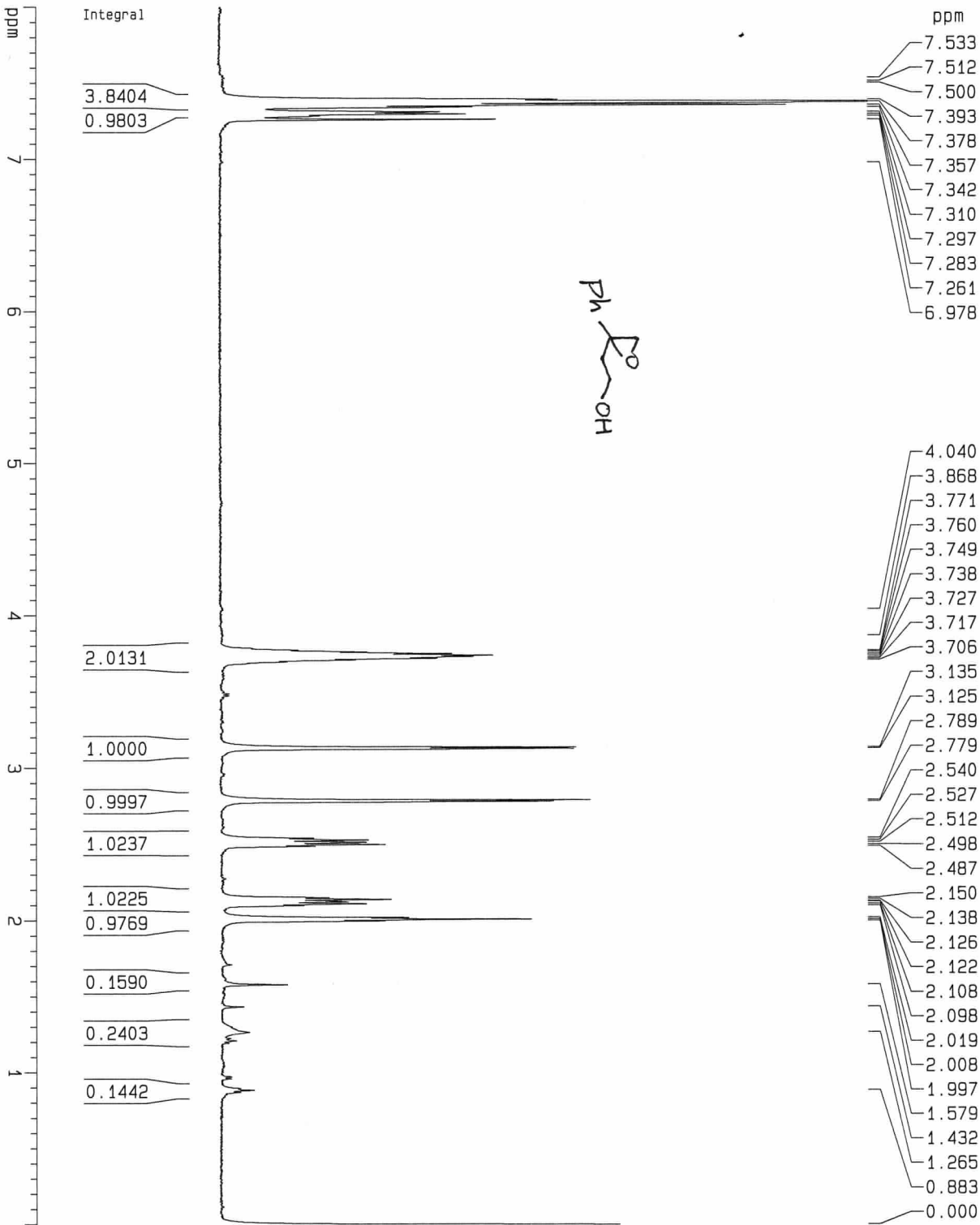
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F2 - Processing parameters

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GB 0
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1D NMR plot parameters

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F2P 0.000 ppm
F2 0.00 Hz
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HZCM 200.05200 Hz/cm



Current Data Parameters
 NAME ZL6R243H149A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 Time 12.56

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 PULPROG zg
 TD 48076
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 8012.820 Hz
 FIDRES 0.166670 Hz
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 RG 256
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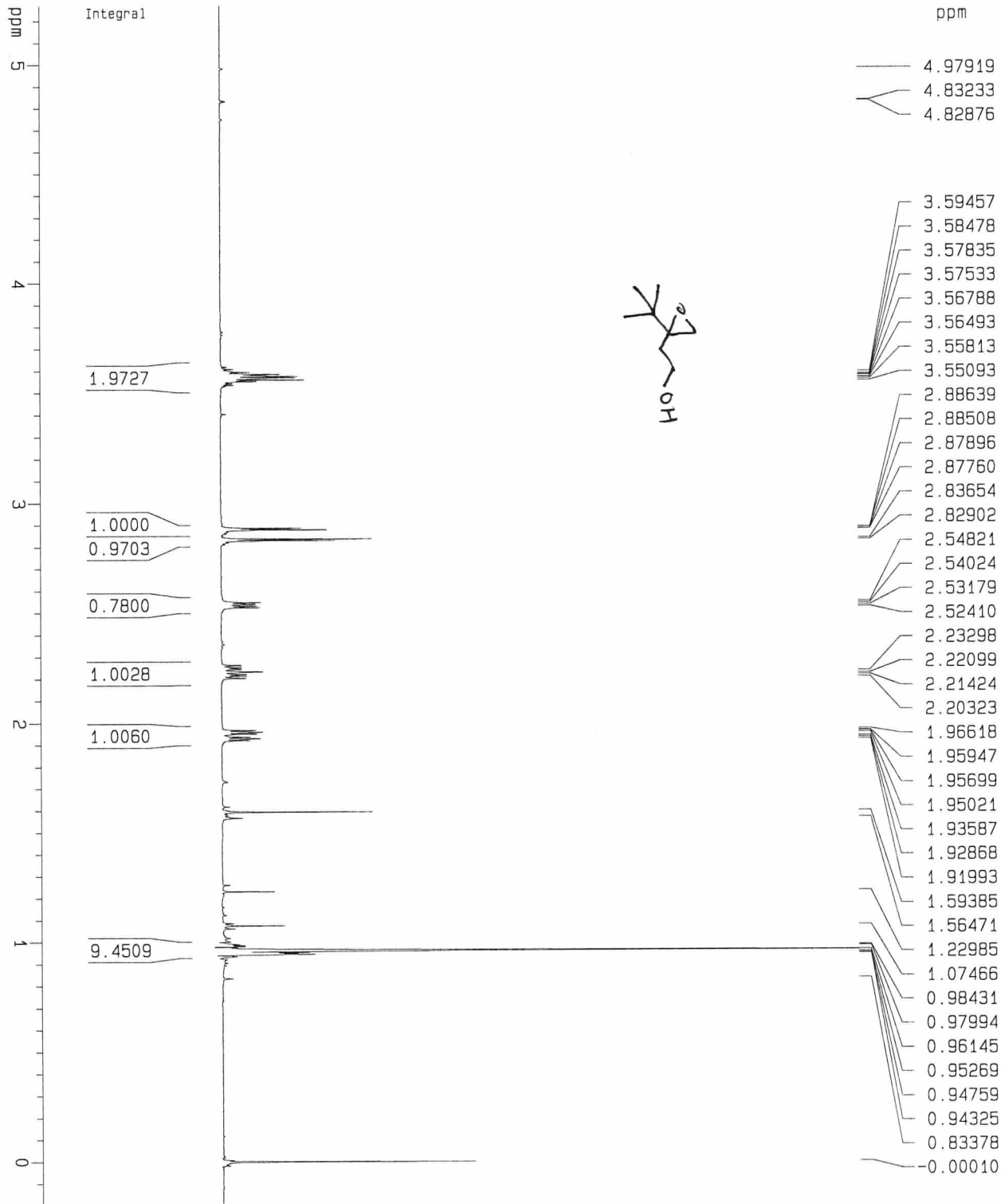
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1D NMR plot parameters

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Current Data Parameters
 NAME ZL7R279H095A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 TD 48076
 SOLVENT Aceton
 NS 8
 DS 0
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 DE 4.50 usec
 TE 300.0 K
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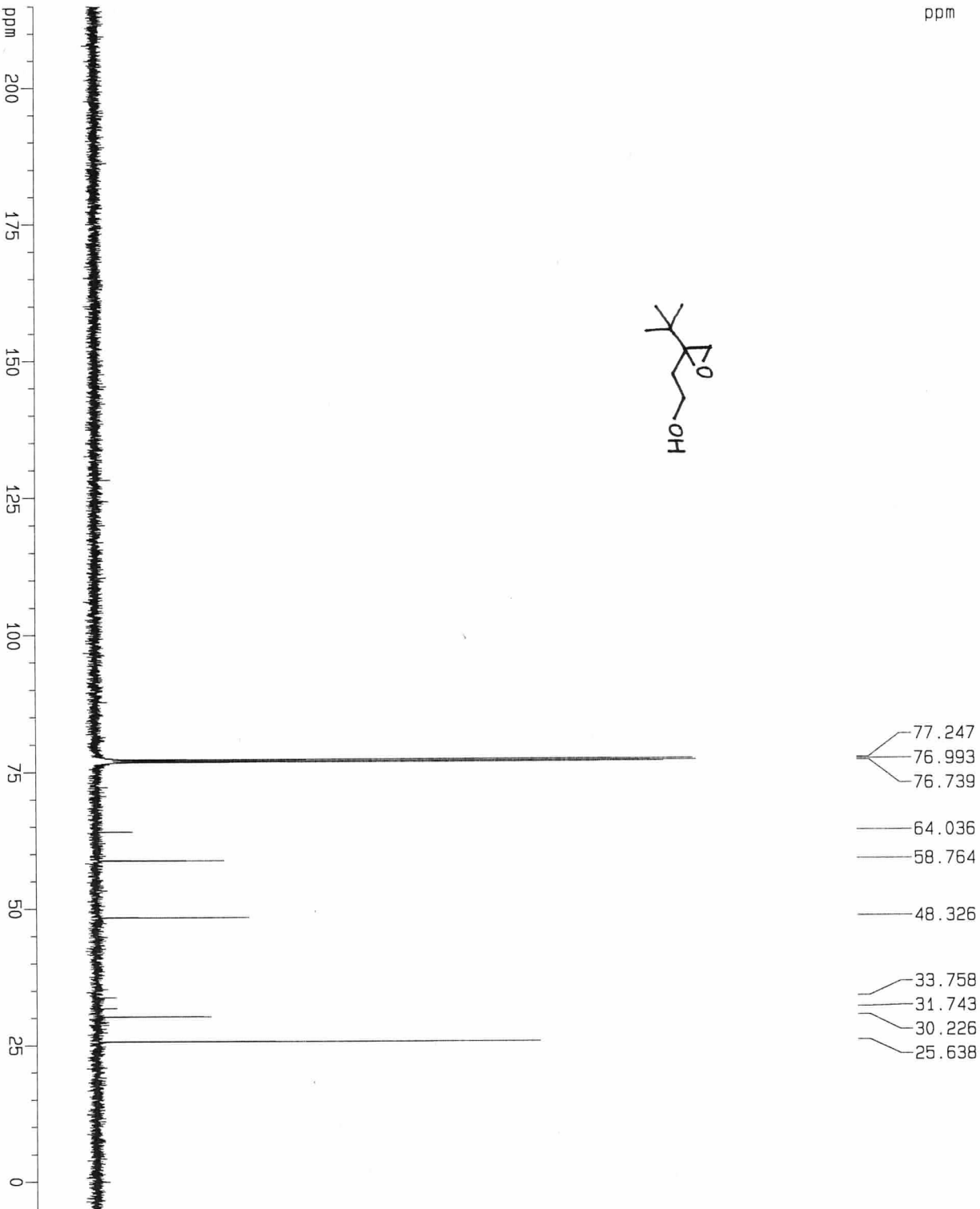
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1D NMR plot parameters
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 F1 2635.29 Hz
 F2P -0.191 ppm
 F2 -95.54 Hz
 PPMCM 0.27301 ppm/cm
 HZCM 136.54134 Hz/cm



ppm



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- 76.739
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- 58.764
- 48.326
- 33.758
- 31.743
- 30.226
- 25.638

Current Data Parameters
 NAME ZL7R279C095A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 Time 23.21

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 SOLVENT CDCl3
 NS 2048
 DS 4
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 FIDRES 0.500026 Hz
 AQ 0.9999972 sec

RG 8192
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 DE 7.50 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec

==== CHANNEL f1 =====
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 P1 4.60 usec
 PL1 0.00 dB
 SF01 125.7690572 MHz

==== CHANNEL f2 =====
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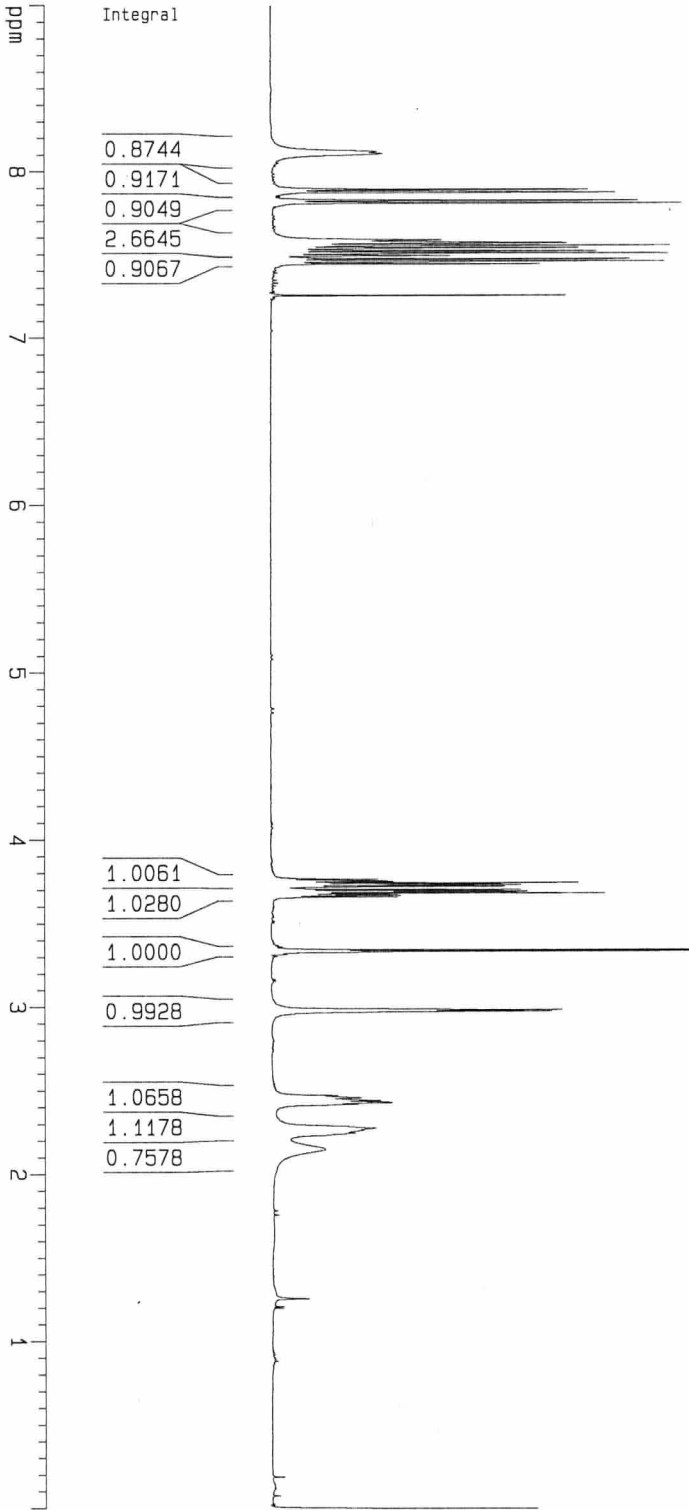
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1D NMR plot parameters
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 F1P 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1383.33669 Hz/cm

ppm

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Current Data Parameters

NAME ZL7R282H077A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

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PULPROG zg
TD 48076
SOLVENT Aceton
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 128
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 3.00000000 sec

==== CHANNEL f1 =====

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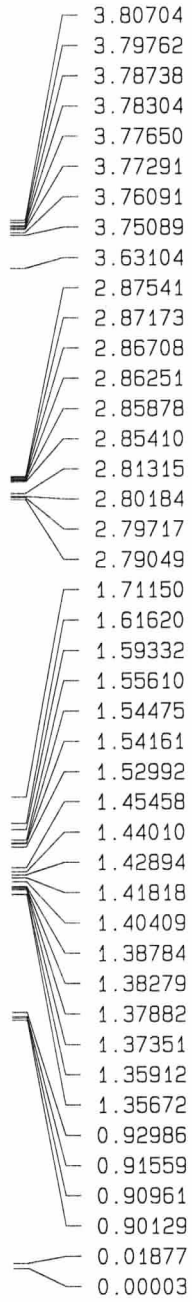
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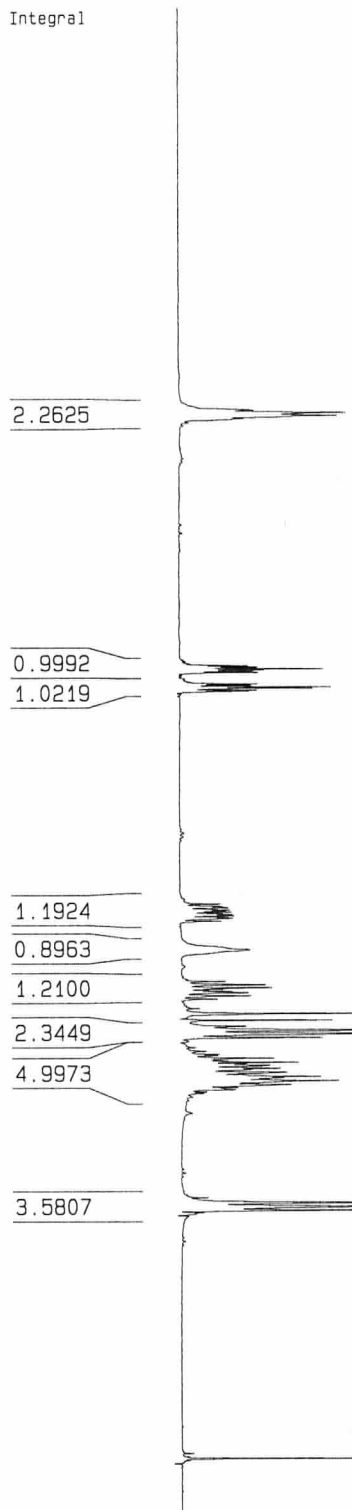
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F2 0.00 Hz
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HZCM 225.05850 Hz/cm

ppm



ppm



Current Data Parameters
 NAME ZL7R241H110A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 Time 0.01

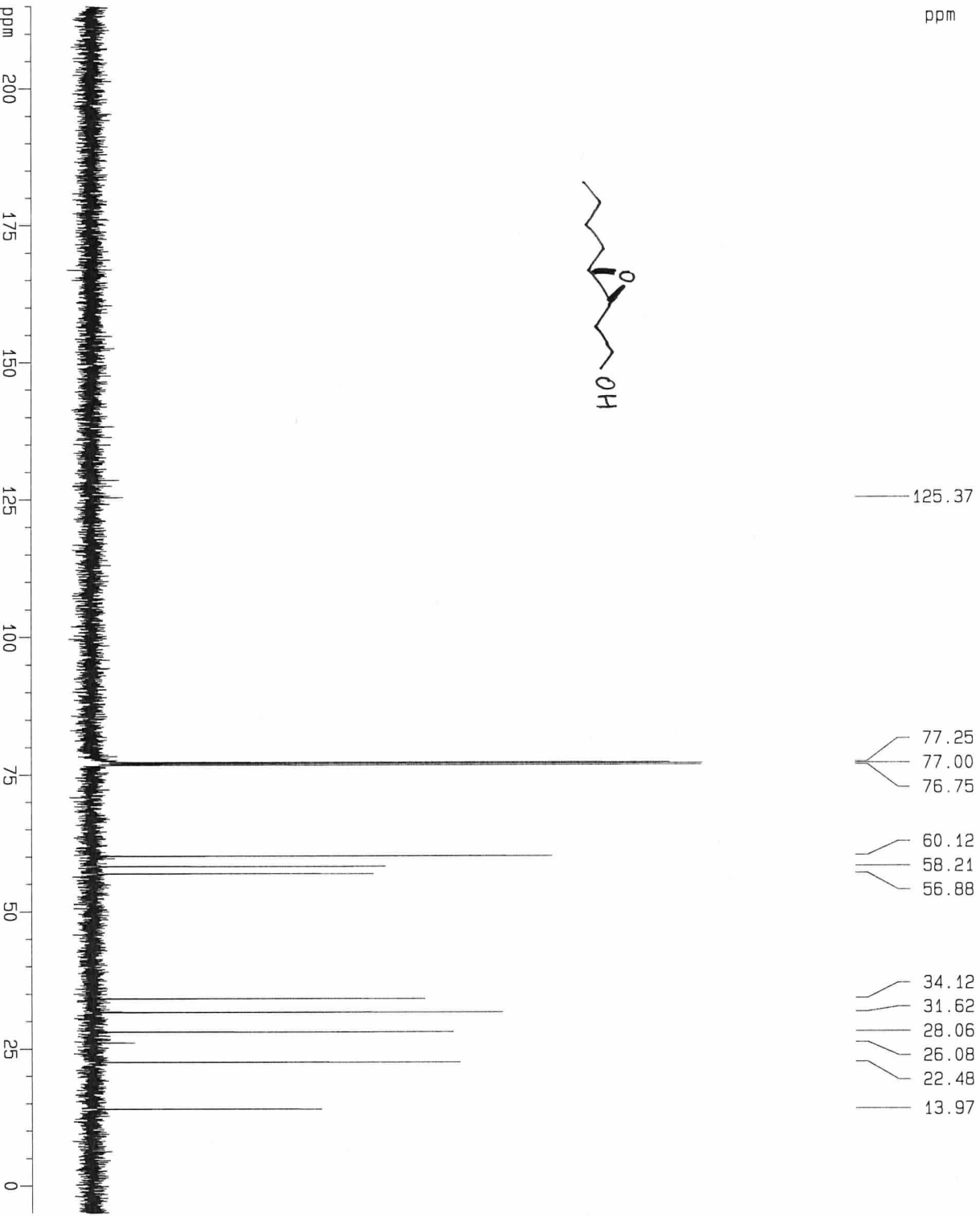
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 SOLVENT Aceton
 NS 8
 DS 0

SWH 8012.820 Hz
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 AQ 2.9999924 sec
 RG 128
 DW 62.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 3.00000000 sec

==== CHANNEL f1 =====
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 P1 9.00 usec
 PL1 0.00 dB
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F2 - Processing parameters
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 SF 500.1300109 MHz
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 SSB 0
 LB 0.30 Hz
 GB 0
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1D NMR plot parameters
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 F1P 5.269 ppm
 F1 2635.29 Hz
 F2P -0.191 ppm
 F2 -95.54 Hz
 PPMCM 0.27301 ppm/cm
 HZCM 136.54134 Hz/cm



Current Data Parameters
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 EXPNO 1
 PROCNO 1

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 SOLVENT CDCl3
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 DS 0

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 DE 7.50 usec
 TE 300.0 K

D1 0.10000000 sec
 D11 0.03000000 sec

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 SF01 125.7690572 MHz

==== CHANNEL f2 =====

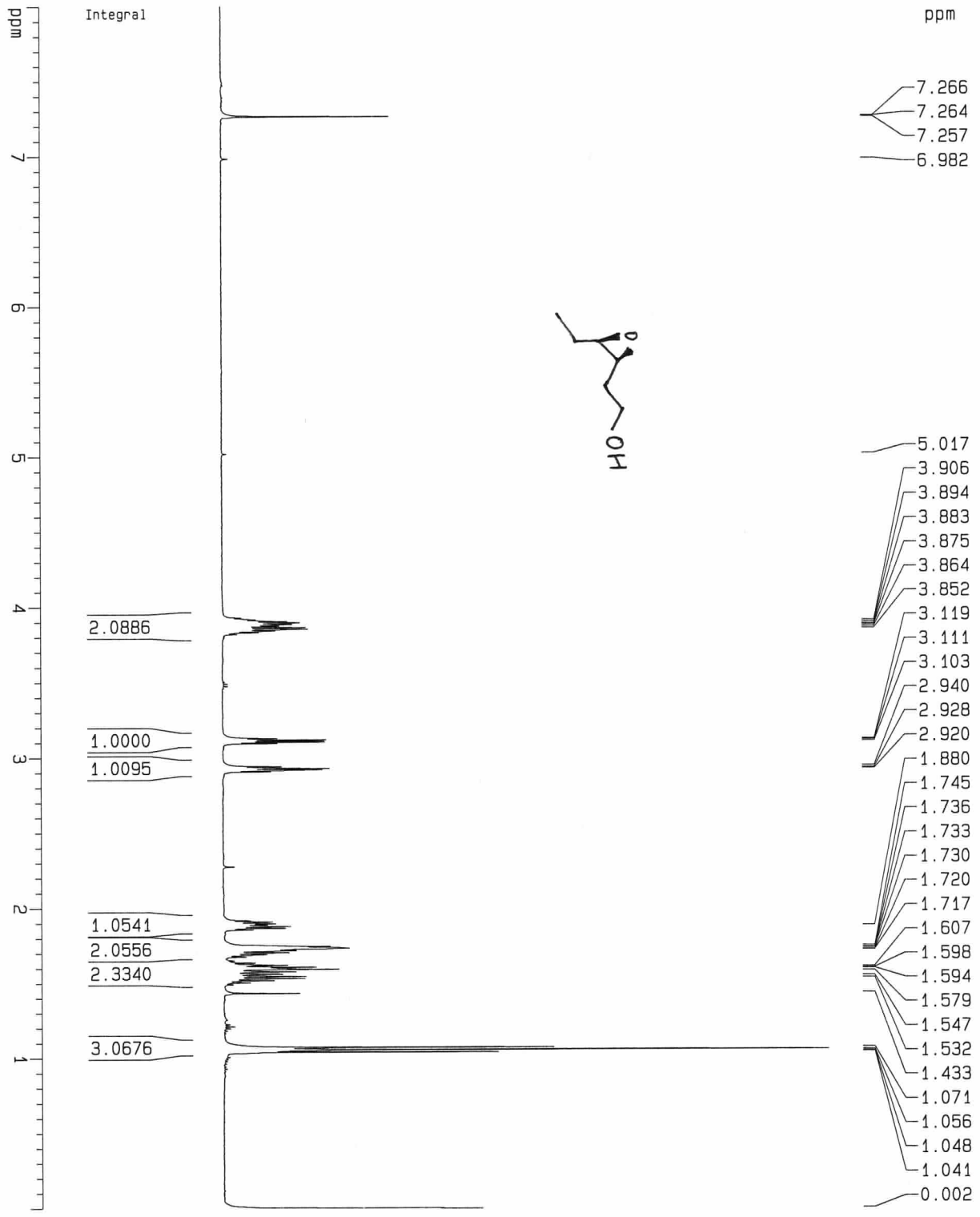
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1D NMR plot parameters

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 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1383.33569 Hz/cm



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7.264
7.257
6.982

5.017
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Current Data Parameters
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 EXPNO 1
 PROCNO 1

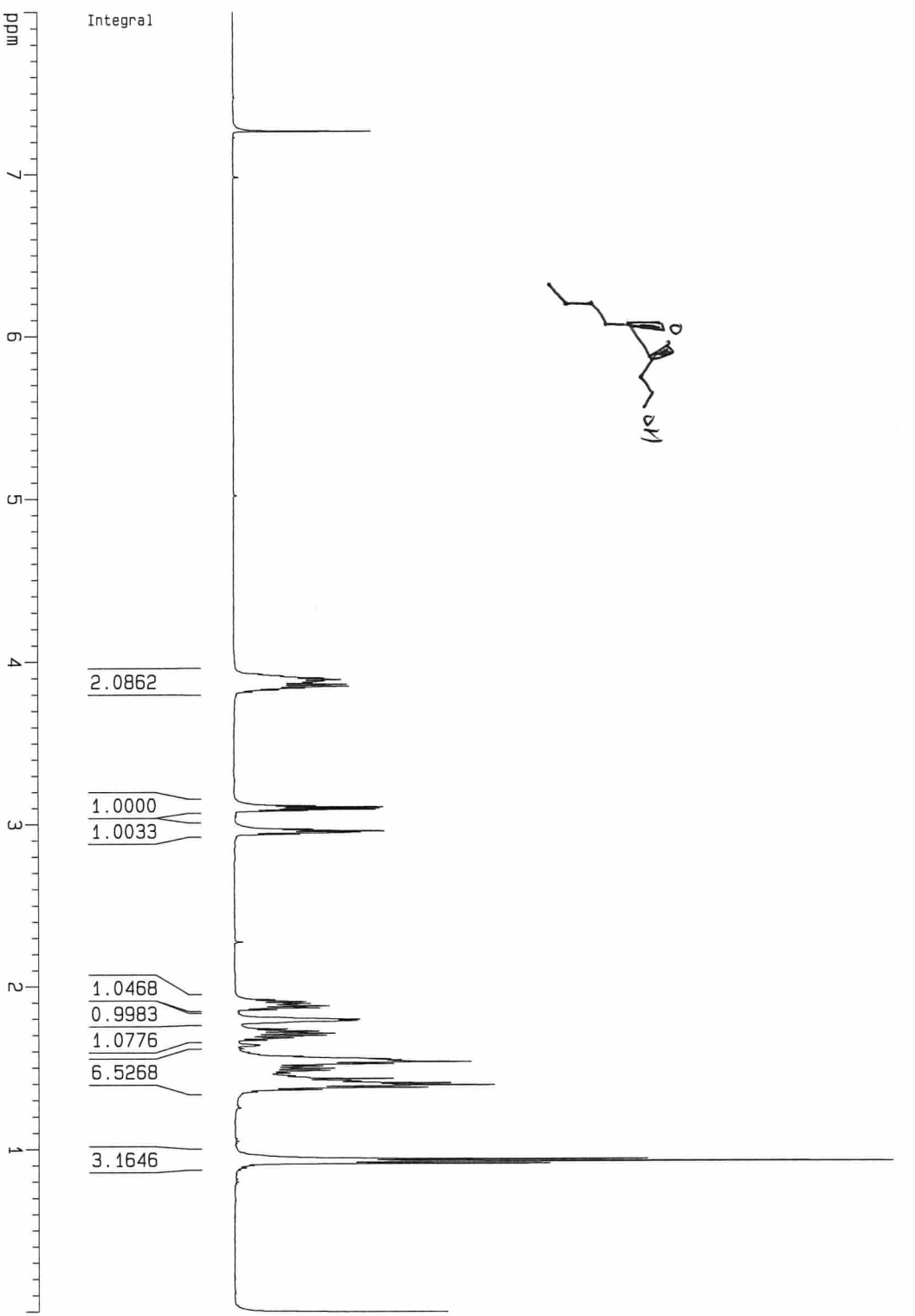
F2 - Acquisition Parameters
 Date_ 20090416
 Time 0.36
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 48076
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.166670 Hz
 AQ 2.9999924 sec
 RG 256
 DW 62.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 3.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.50 usec
 PL1 0.00 dB
 SF01 500.1325006 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1300114 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.000 ppm
 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 200.05200 Hz/cm

ppm
 7.267
 7.265
 6.980



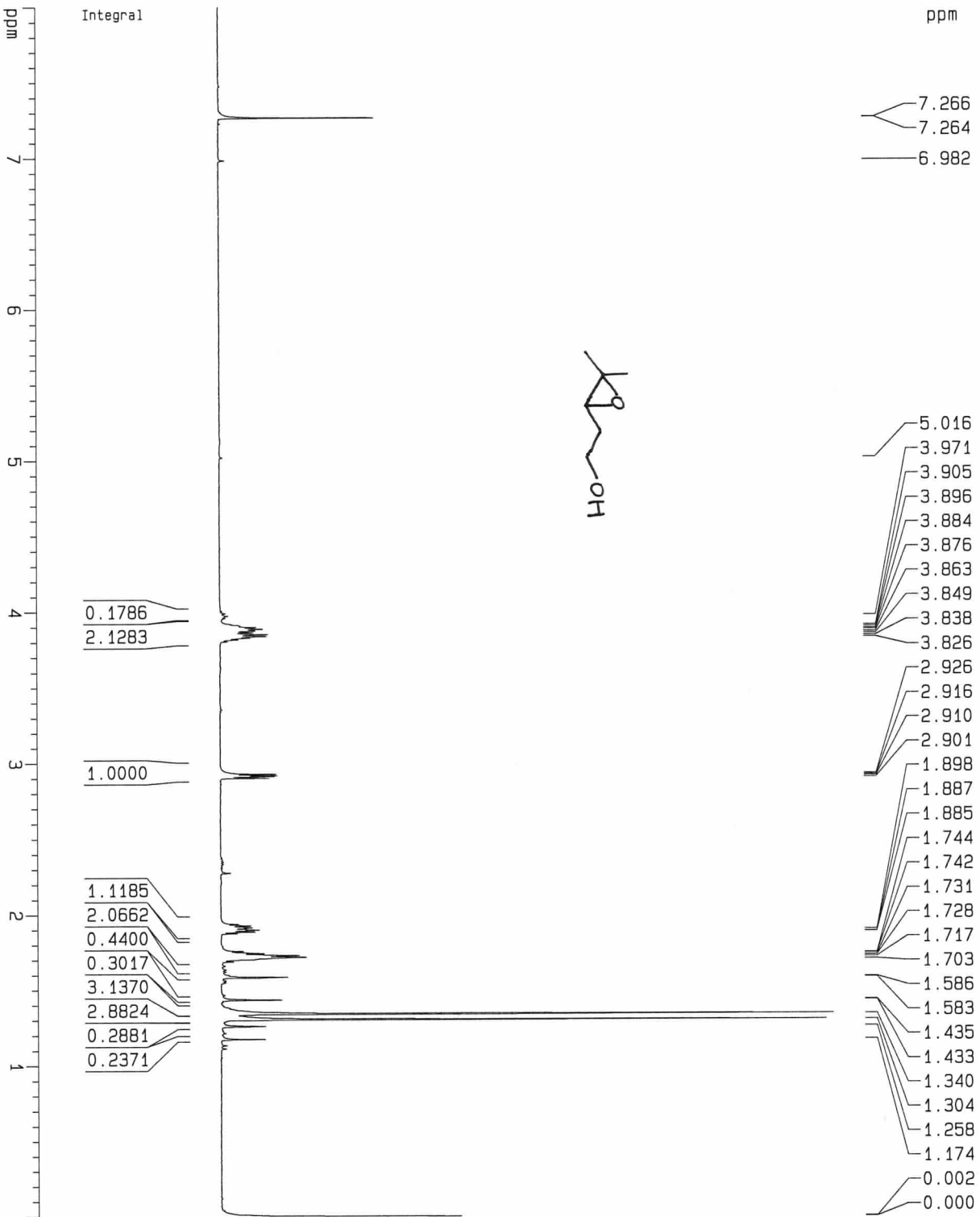
Current Data Parameters
 NAME ZLBR171H138A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20090416
 Time 13.11
 INSTRUM spect
 PROBHD 5 mm GNP 1H
 PULPROG zg
 TD 48076
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 8012.820 HZ
 FIDRES 0.166670 HZ
 AQ 2.9999924 sec
 RG 256
 DW 62.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 3.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.50 usec
 PL1 0.00 dB
 SFO1 500.1325006 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1300112 MHz
 WDW EM
 SSB 0
 LB 0.30 HZ
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.000 ppm
 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 200.05200 Hz/cm



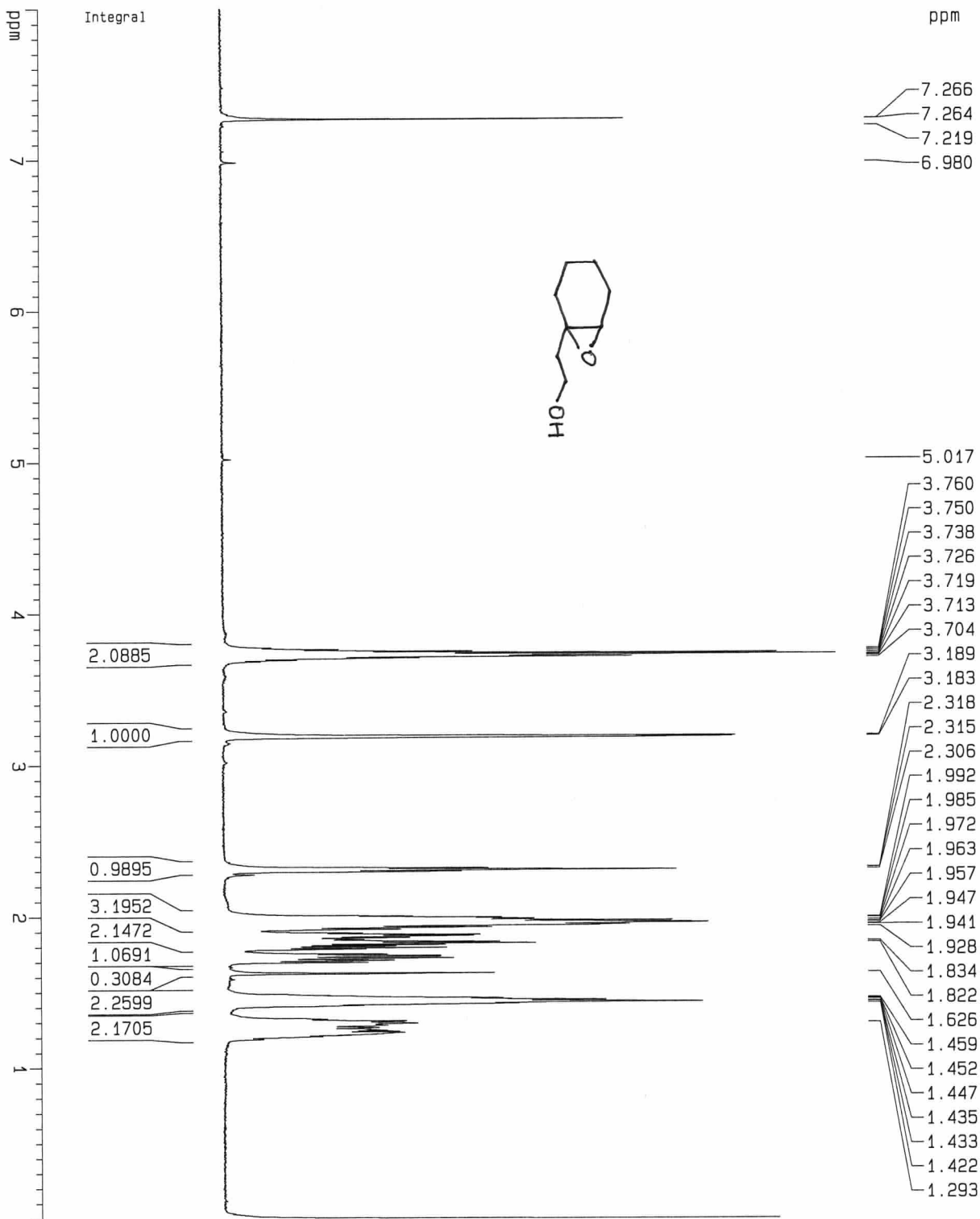
Current Data Parameters
 NAME ZL6R247H144A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20090415
 Time 22:59
 INSTRUM spect
 PROBHID 5 mm QNP 1H
 PULPROG zg
 TD 48076
 SOLVENT CDC13
 NS 8
 DS 0
 SMH 8012.820 Hz
 FIDRES 0.166670 Hz
 AQ 2.9999924 sec
 RG 512
 DW 62.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 3.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.50 usec
 PL1 0.00 dB
 SF01 500.1325006 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1300116 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.000 ppm
 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPKCM 0.40000 ppm/cm
 HZCM 200.05200 Hz/cm



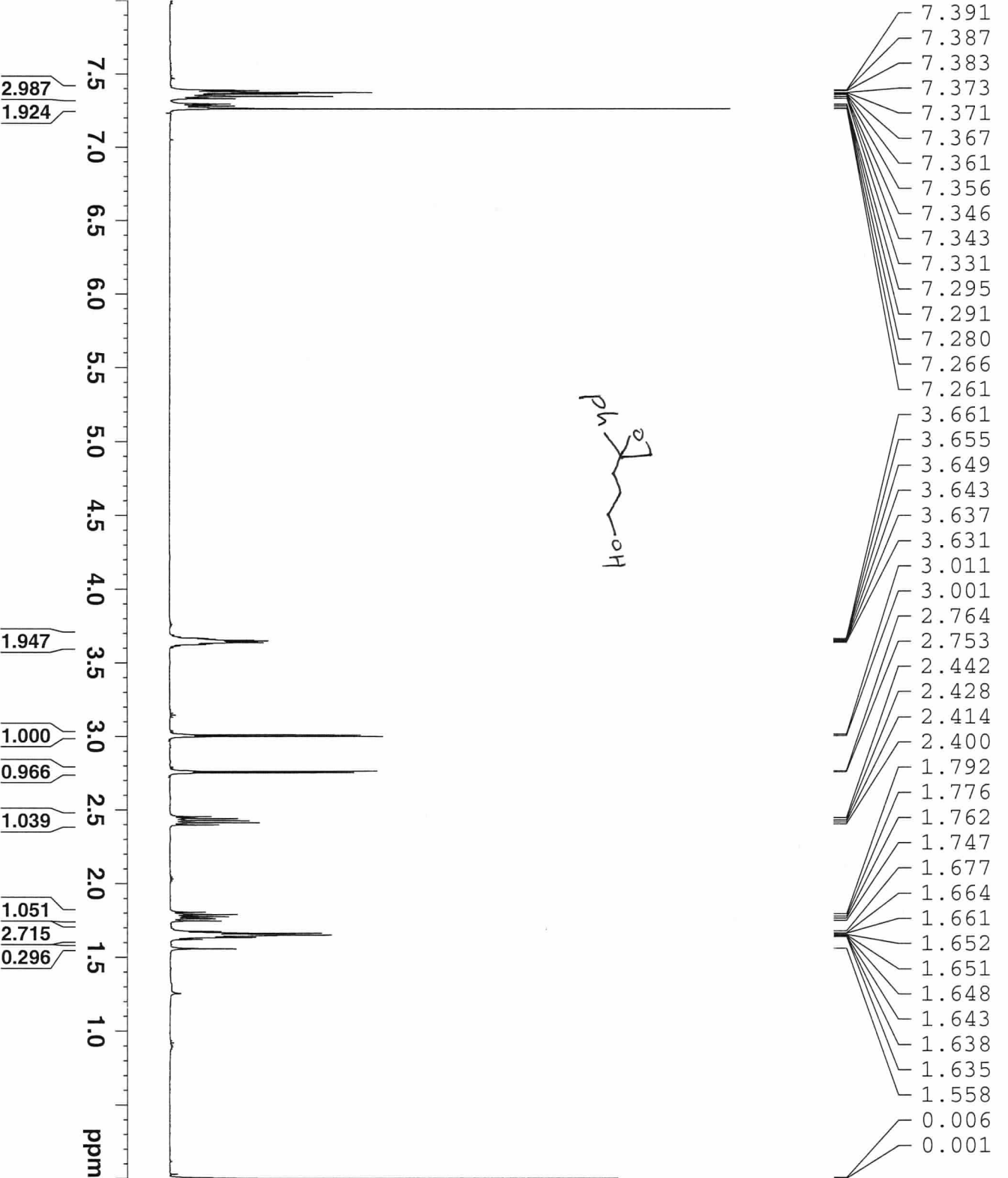
Current Data Parameters
 NAME ZL6R248H145A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20090416
 Time 13.16
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 48076
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 8012.820 Hz
 FIDRES 0.166670 Hz
 AQ 2.9999924 sec
 RG 256
 DW 62.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 3.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.50 usec
 PL1 0.00 dB
 SF01 500.1325006 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1300116 MHz
 MDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.000 ppm
 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 200.05200 Hz/cm



- 7.391
- 7.387
- 7.383
- 7.373
- 7.371
- 7.367
- 7.361
- 7.356
- 7.346
- 7.343
- 7.331
- 7.295
- 7.291
- 7.280
- 7.266
- 7.261
- 3.661
- 3.655
- 3.649
- 3.643
- 3.637
- 3.631
- 3.011
- 3.001
- 2.764
- 2.753
- 2.442
- 2.428
- 2.414
- 2.400
- 1.792
- 1.776
- 1.762
- 1.747
- 1.677
- 1.664
- 1.661
- 1.652
- 1.651
- 1.648
- 1.643
- 1.638
- 1.635
- 1.558
- 0.006
- 0.001

2.987
1.924

1.947

1.000

0.966

1.039

1.051

2.715

0.296

ppm

```

Current Data Parameters
NAME          ZL7R192H079A
EXPNO         1
PROCNO        1

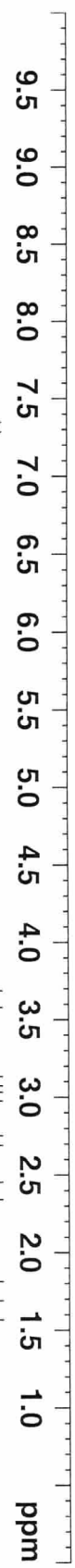
F2 - Acquisition Parameters
Date_         20091112
Time         1.46
INSTRUM      spect
PROBHD       5 mm PABBI 1H/
PULPROG      zg
TD           59998
SOLVENT      CDCl3
NS           8
DS           0
SWH          10000.000 Hz
FIDRES       0.166672 Hz
AQ           2.9999499 se
RG           256
DE           50.000 us
TE           7.50 us
D1           295.6 K
TD0          1.00000000 se
TD0          1

===== CHANNEL f1 =====
NUC1         1H
P1           5.35 us
PL1          0.00 dB
SFO1         499.8729992 MHz

F2 - Processing parameters
SI           32768
SF           499.8700172 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
  
```

7.931
7.914
7.449
7.431
7.330
7.312
7.296
7.265

3.743
3.646
3.634
3.621
3.612
3.106
3.092
3.006
2.996
2.720
2.710
2.424
2.410
2.396
2.381
2.367
2.046
2.025
2.013
1.783
1.767
1.751
1.740
1.737
1.722
1.648
1.635



3.2421

2.0437

0.0972

1.0000

0.9766

1.0602

1.8231

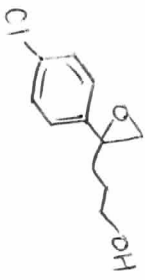
2.1334

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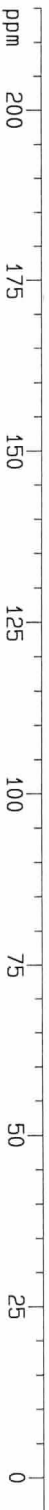
Current Data Parameters
NAME      ZL/R318H169A
EXPNO     1
PROCNO    1
F2 - Acquisition Parameters
Date_     20100107
Time      21.46
INSTRUM   spect
PROBHD    5 mm PABBI 1H/
PULPROG   zg
TD         59998
SOLVENT   CDCl3
NS         8
DS         0
SWH        10000.000 Hz
FIDRES     0.166672 Hz
AQ          2.9999499 sec
RG          45.3
DM          50.000 us
DE          1.50 us
TE          293.0 K
D1          1.00000000 sec
TD0         1

===== CHANNEL F1 =====
NUC1       1H
P1         5.35 us
PL1        0.00 dB
SFO1       499.8729992 MHz

F2 - Processing parameters
SI         32768
SF         499.8700157 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



ppm



- 138.11
- 133.33
- 129.49
- 128.90
- 128.58
- 128.37
- 127.48
- 127.26
- 126.80
- 126.29

- 77.25
- 77.00
- 76.75
- 69.11
- 68.87
- 68.49
- 62.16
- 59.65
- 56.10

- 35.19
- 34.07
- 31.63
- 31.45
- 27.72
- 26.76
- 26.09

Current Data Parameters
 NAME ZL7R318C169A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20100108
 Time 22.41

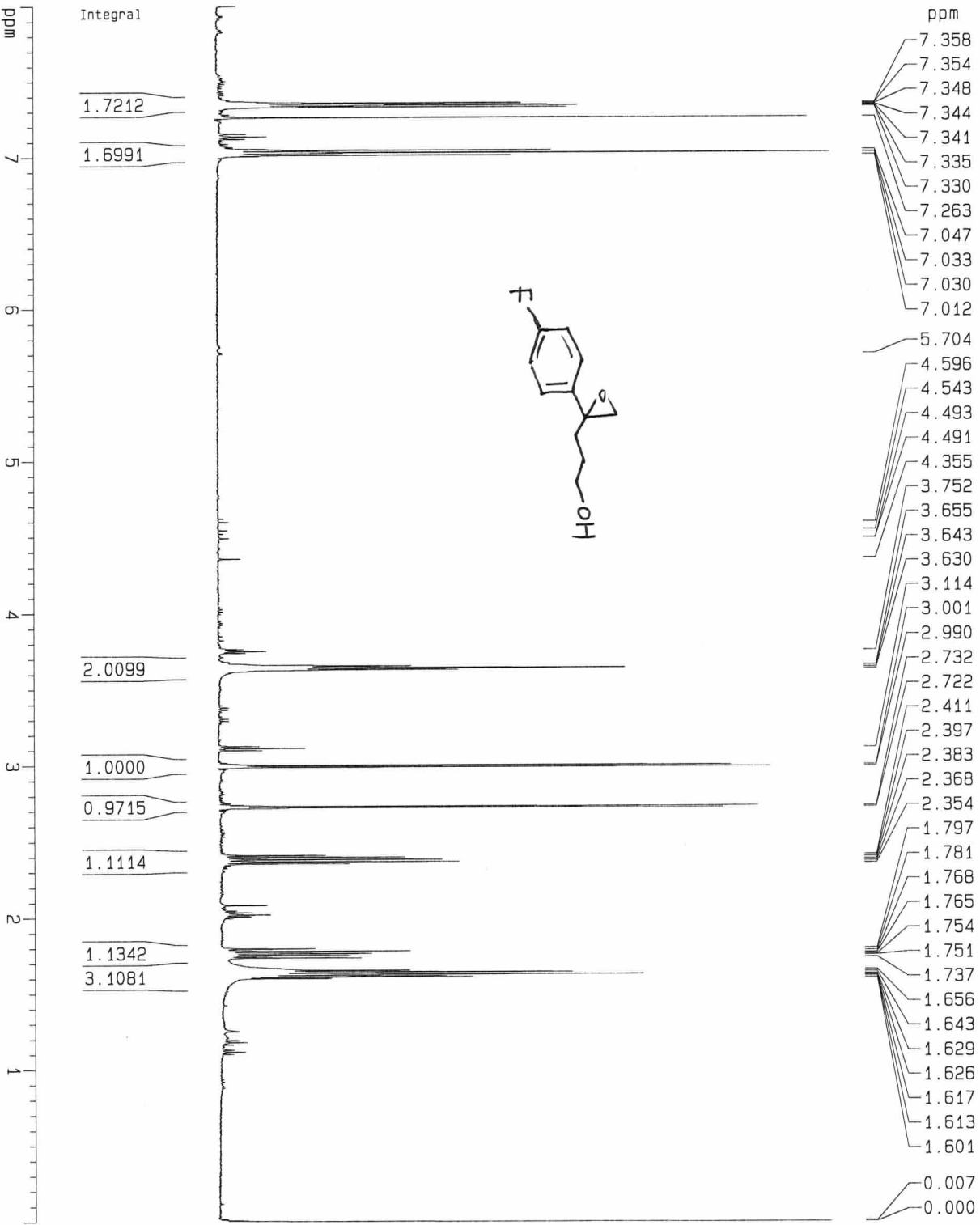
INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TD 75184
 SOLVENT CDCl3
 NS 1862
 DS 0
 SWH 37593.984 Hz
 FIDRES 0.500026 Hz
 AQ 0.999972 sec
 RG 8192
 DE 13.300 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec

==== CHANNEL f1 =====
 NUC1 13C
 P1 4.60 usec
 PL1 0.00 dB
 SF01 125.7690572 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 90.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SF02 500.1320005 MHz

F2 - Processing Parameters
 SI 32768
 SF 125.7577941 MHz
 KDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR Plot parameters
 CX 20.00 cm
 F1P 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPKCM 11.00000 ppm/cm
 HZCM 1383.33582 Hz/cm



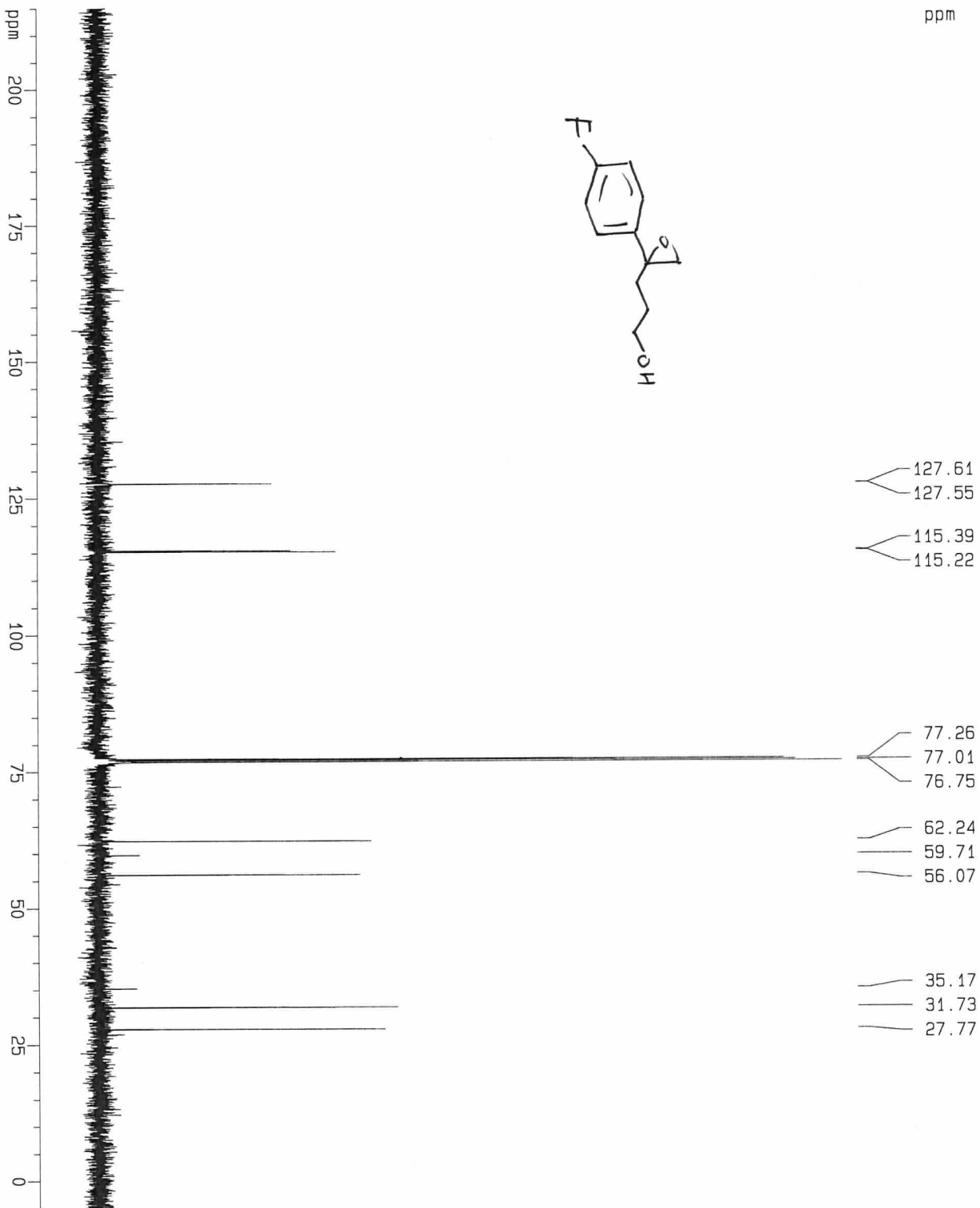
Current Data Parameters
 NAME ZL7R317H189A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20100121
 Time 21.48
 INSTRUM spect
 PROBHID 5 mm QNP 1H
 PULPROG zg
 TD 48076
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 8012.820 Hz
 FIDRES 0.166670 Hz
 AQ 2.9999924 sec
 RG 256
 DW 62.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 3.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SF01 500.1325006 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1300124 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.000 ppm
 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCKM 0.40000 ppm/cm
 HZCM 200.05200 Hz/cm



Current Data Parameters
 NAME ZL7R317C189A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20100121
 Time 22.09
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TD 75184
 SOLVENT CDCl3
 NS 1024
 DS 0
 SMH 37593.984 Hz
 FIDRES 0.500026 Hz
 AQ 0.9999972 sec
 RG 8192
 DW 13.300 usec
 DE 7.50 usec
 TE 300.0 K
 D1 0.10000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
 P1 4.60 usec
 PL1 0.00 dB
 SF01 125.7690572 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SF02 500.1320005 MHz

F2 - Processing parameters

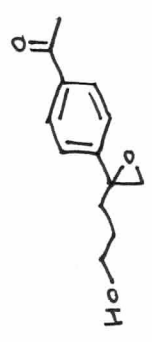
SI 32768
 SF 125.7577918 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 F1P 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCK 11.00000 ppm/cm
 HZCM 1383.33569 Hz/cm

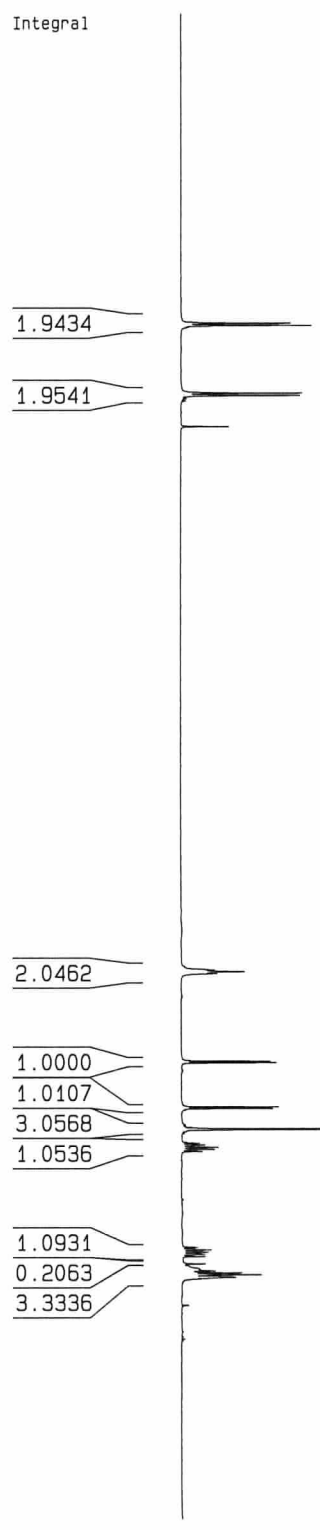
ppm

- 7.937
- 7.933
- 7.924
- 7.920
- 7.475
- 7.471
- 7.461
- 7.458
- 7.251



- 3.645
- 3.633
- 3.621
- 3.039
- 3.028
- 2.735
- 2.724
- 2.593
- 2.588
- 2.484
- 2.482
- 2.469
- 2.454
- 2.440
- 1.787
- 1.772
- 1.757
- 1.743
- 1.694
- 1.648
- 1.634
- 1.622
- 1.618
- 1.609
- 1.605

ppm



Current Data Parameters

NAME ZL7R348H249A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20100430
 Time 16.30
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 48076
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.166670 Hz
 AQ 2.9999924 sec
 RG 128
 DW 62.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 3.00000000 sec

==== CHANNEL f1 =====

NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SF01 500.1325006 MHz

F2 - Processing parameters

SI 65536
 SF 500.1300181 MHz
 KDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

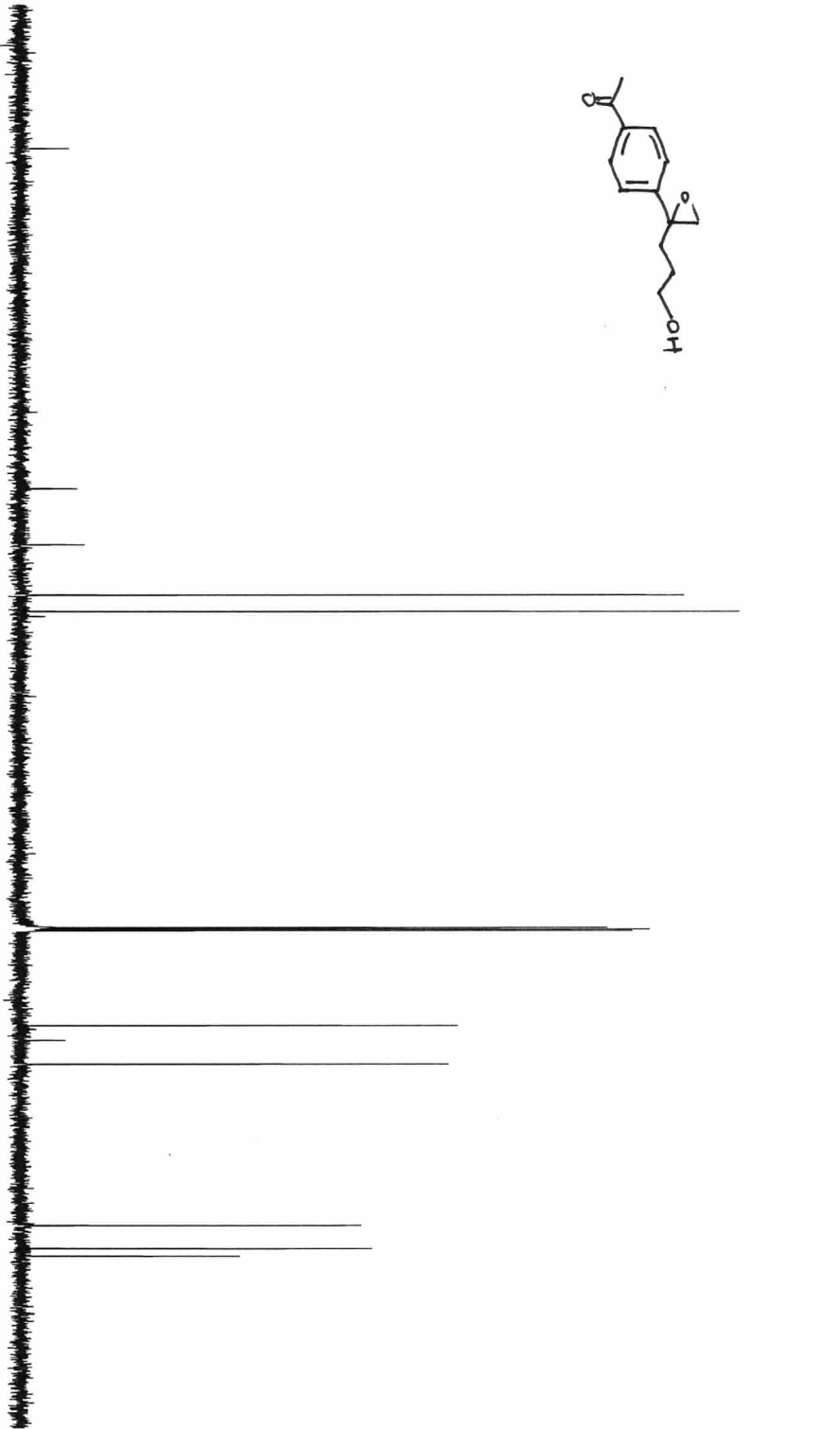
1D NMR plot parameters

CX 20.00 cm
 F1P 10.000 ppm
 F1 5001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.50000 ppm/cm
 HZCM 250.06500 Hz/cm

ppm

ppm
200
175
150
125
100
75
50
25

197.62
145.00
136.37
128.53
126.04
125.29
77.25
77.00
76.75
62.17
59.86
56.17
31.28
27.74
26.61



Current Data Parameters
NAME ZL7R348C249A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20100430
Time 16.58
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg
TD 75184
SOLVENT C6D6
NS 1318
DS 0
SWH 37593.984 Hz
FIDRES 0.500026 Hz
AQ 0.9999972 sec
RG 4096
DM 13.300 usec
DE 7.50 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

==== CHANNEL f1 =====
NUC1 13C

P1 4.60 usec
PL1 0.00 dB
SF01 125.7690572 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16

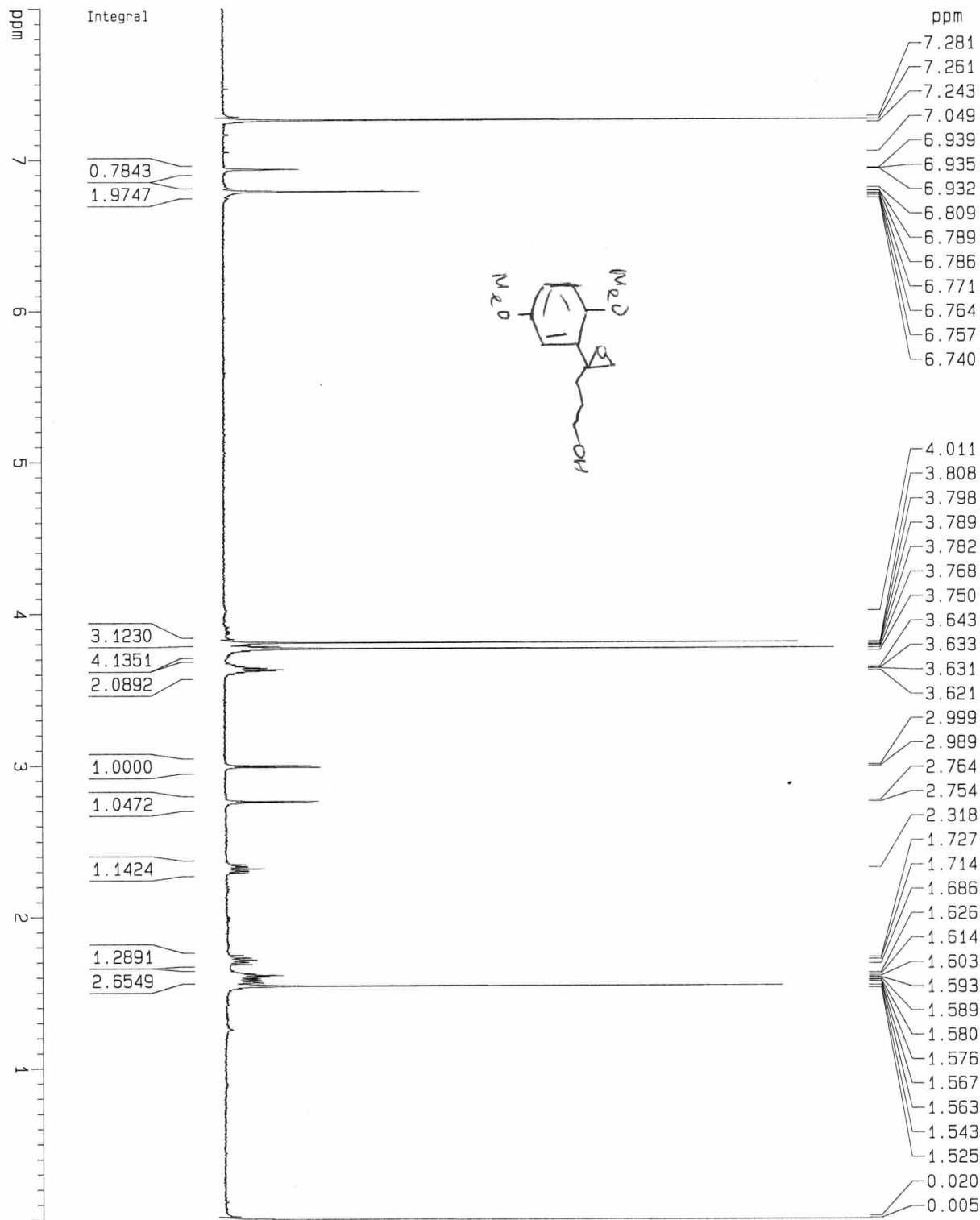
NUC2 1H
PCPD2 90.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 500.1320005 MHz

F2 - Processing Parameters

SI 32768
SF 125.7577918 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 220.000 ppm
F1 27666.71 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm
HZCM 1383.33569 Hz/cm



Current Data Parameters
 NAME ZL7R320H175A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20100201
 Time 19.18

INSTRUM spect
 PROBHD 5 mm GNP 1H
 PULPROG zg
 TD 48076
 SOLVENT CDCl3
 NS 20
 DS 0

SWH 8012.820 Hz
 FIDRES 0.166670 Hz
 AQ 2.9999924 sec
 RG 512
 DW 62.400 usec
 DE 4.50 usec
 TE 300.0 K

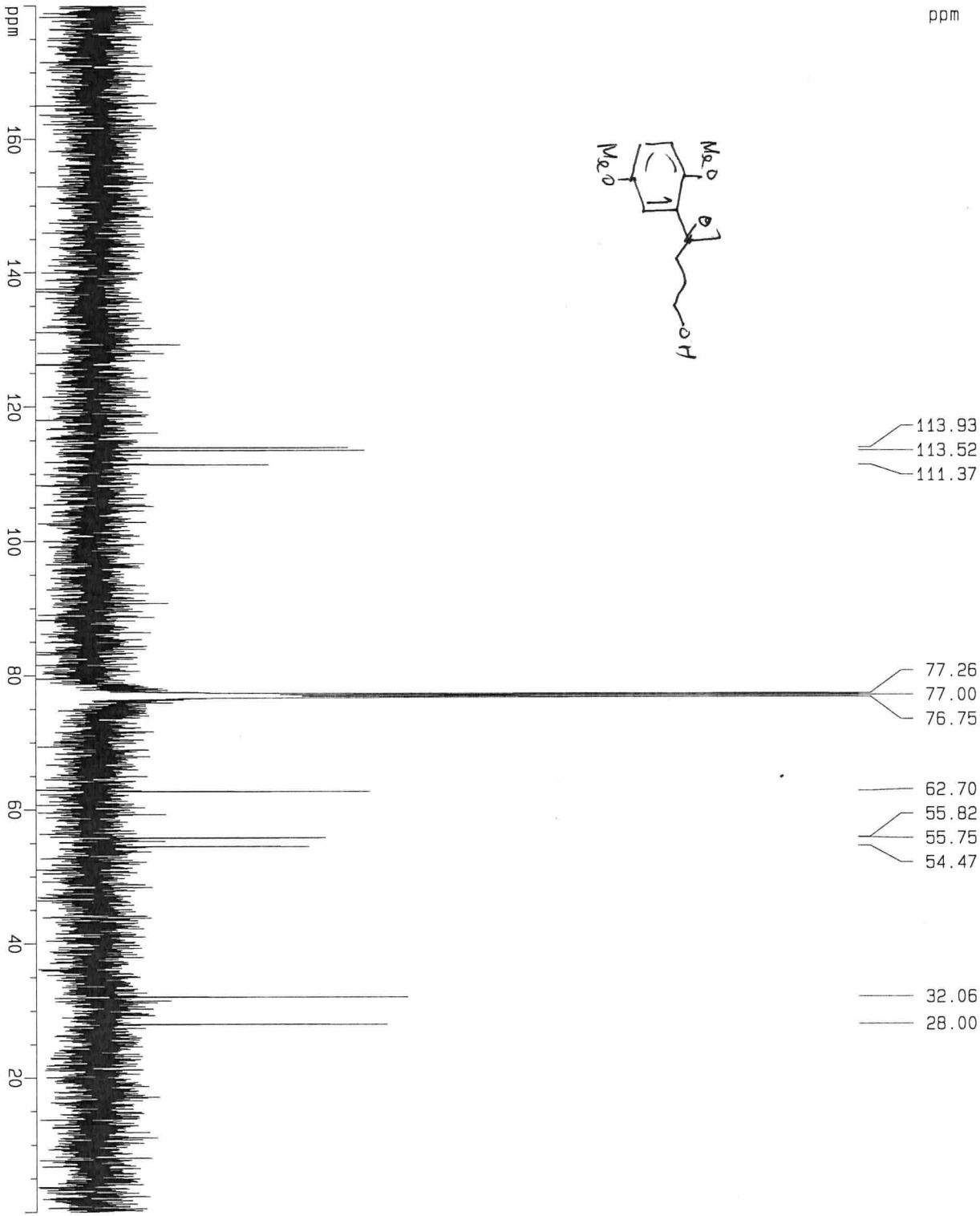
D1 3.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SF01 500.1325006 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1300133 MHz
 KWDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.000 ppm
 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 200.05200 Hz/cm

ppm



Current Data Parameters
 NAME ZL7R320C175A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20100201
 Time 22.22
 INSTRUM spect
 PROBHID 5 mm QNP 1H
 PULPROG zgpg30
 TD 75184
 SOLVENT C6D6
 NS 6269
 DS 0
 SMH 37593.984 Hz
 FIDRES 0.500025 Hz
 AQ 0.9999972 sec
 RG 8192
 DW 13.300 usec
 DE 7.50 usec
 TE 300.0 K
 D1 0.10000000 sec
 D11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
 P1 4.60 usec
 PL1 0.00 dB
 SF01 125.7690572 MHz

===== CHANNEL f2 =====

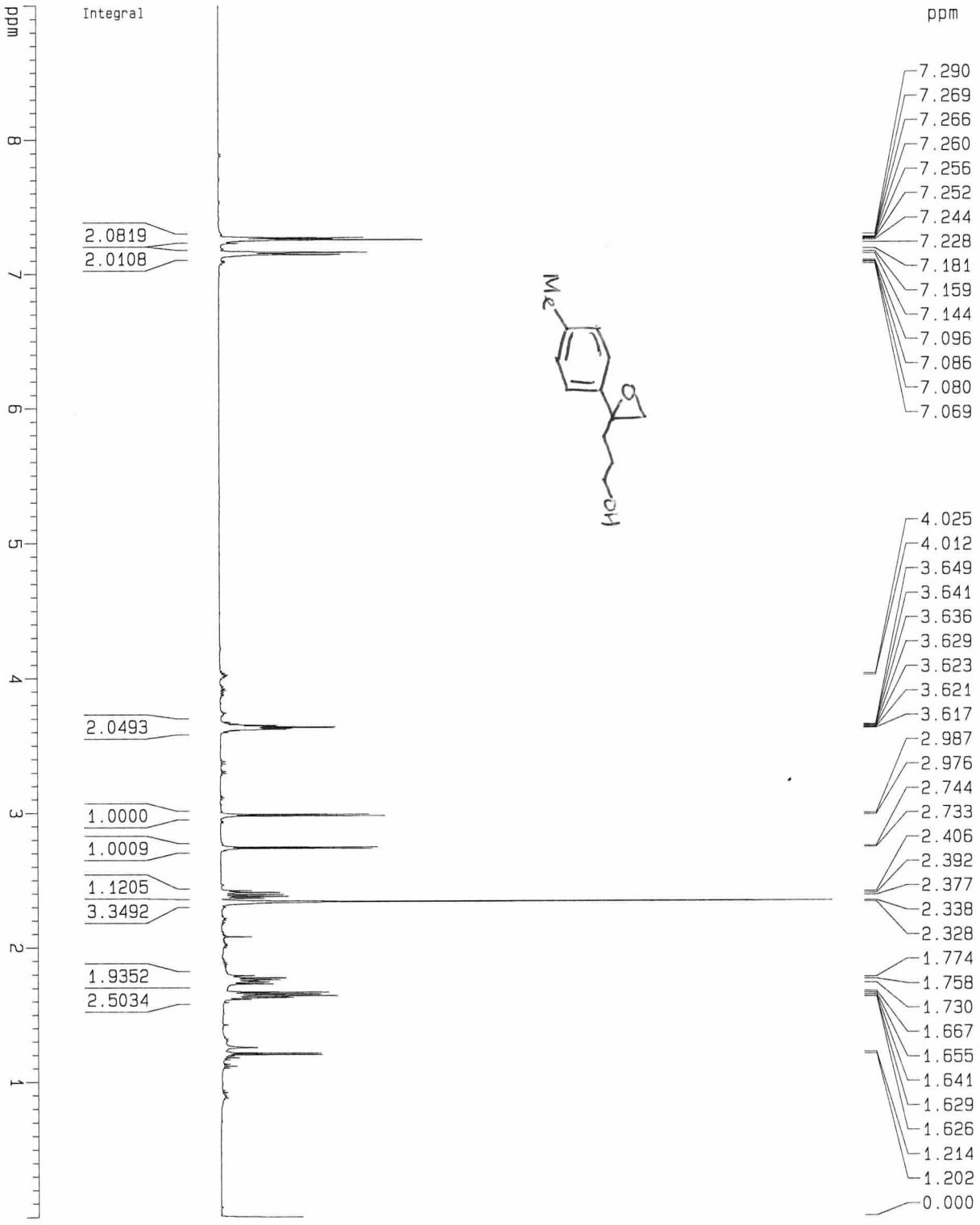
CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SF02 500.1320005 MHz

F2 - Processing parameters

SI 32768
 SF 125.7577906 MHz
 KDM EM
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 F1P 180.000 ppm
 F1 22636.40 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 9.00000 ppm/cm
 HZCM 1131.82007 Hz/cm



Current Data Parameters
 NAME ZL7R319H185A2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20100201
 Time 19.08

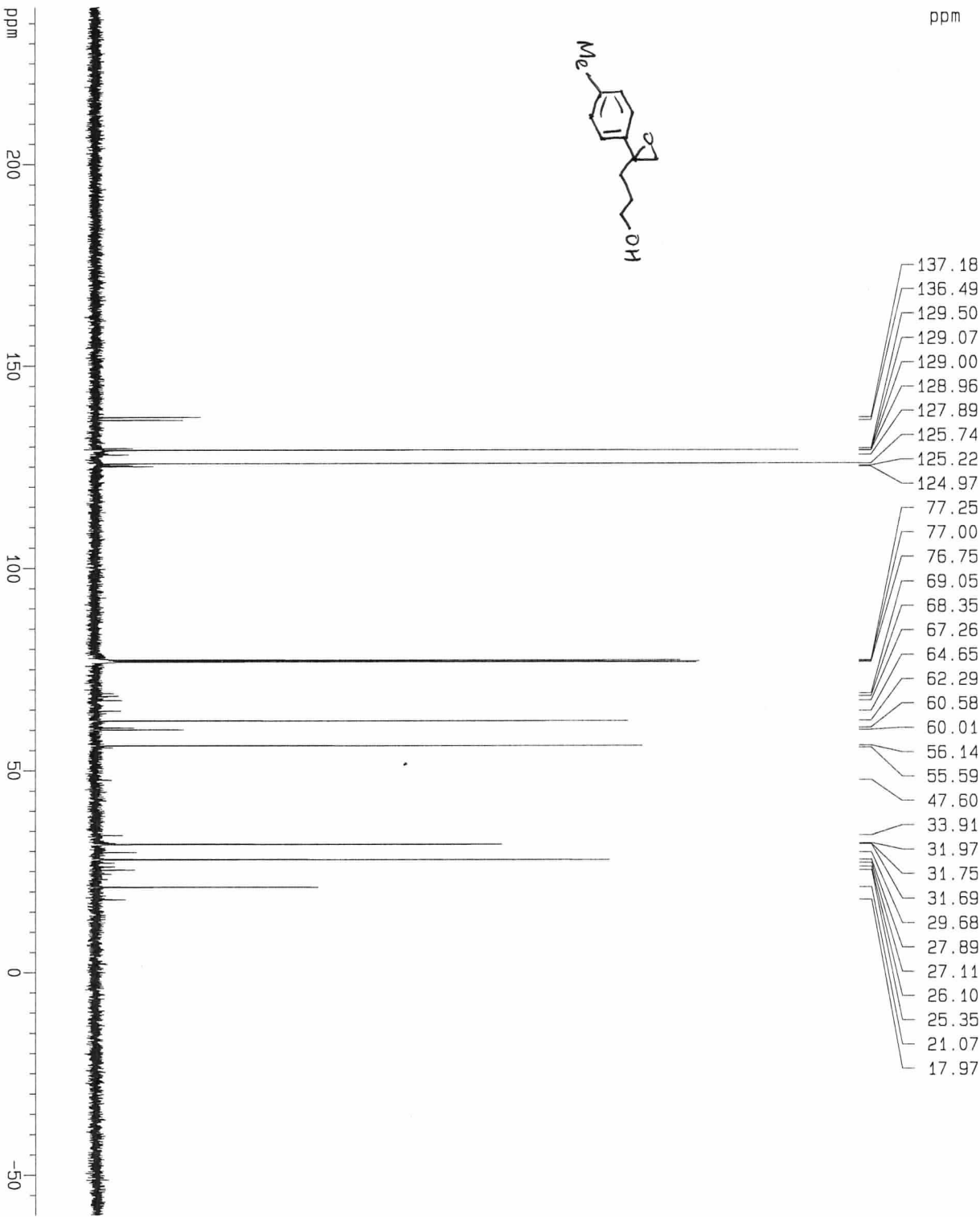
INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 48076
 SOLVENT CDCl3
 NS 4
 DS 0

SMH 8012.820 Hz
 FIDRES 0.156670 Hz
 AQ 2.9999924 sec
 RG 128
 DW 62.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 3.00000000 sec

==== CHANNEL f1 =====
 NUCL1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SF01 500.1325006 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1300133 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 4504.17 Hz
 F2P 0.000 ppm
 F2 0.000 Hz
 PPMCM 0.45000 ppm/cm
 HZCM 225.05850 Hz/cm



ppm

- 137.18
- 136.49
- 129.50
- 129.07
- 129.00
- 128.96
- 127.89
- 125.74
- 125.22
- 124.97
- 77.25
- 77.00
- 76.75
- 69.05
- 68.35
- 67.26
- 64.65
- 62.29
- 60.58
- 60.01
- 56.14
- 55.59
- 47.60
- 33.91
- 31.97
- 31.75
- 31.69
- 29.68
- 27.89
- 27.11
- 26.10
- 25.35
- 21.07
- 17.97

Current Data Parameters

NAME ZLR319C185A

EXEND 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20100201

Time 20.48

INSTRUM spect

PROBHD 5 mm QNP 1H

PULPROG zgpg

TD 75184

SOLVENT C6D6

NS 2048

DS 0

SMH 37593.984 Hz

FIDRES 0.500026 Hz

AQ 0.999972 sec

RG 8192

DW 13.300 usec

DE 7.50 usec

TE 300.0 K

D1 0.1000000 sec

d11 0.03000000 sec

CHANNEL f1

NUC1 13C

P1 4.60 usec

PL1 0.00 dB

SFO1 125.7690572 MHz

CHANNEL f2

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 120.00 dB

PL12 19.00 dB

SFO2 500.1320005 MHz

F2 - Processing parameters

SI 32768

SF 125.7577929 MHz

WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm

F1P 238.970 ppm

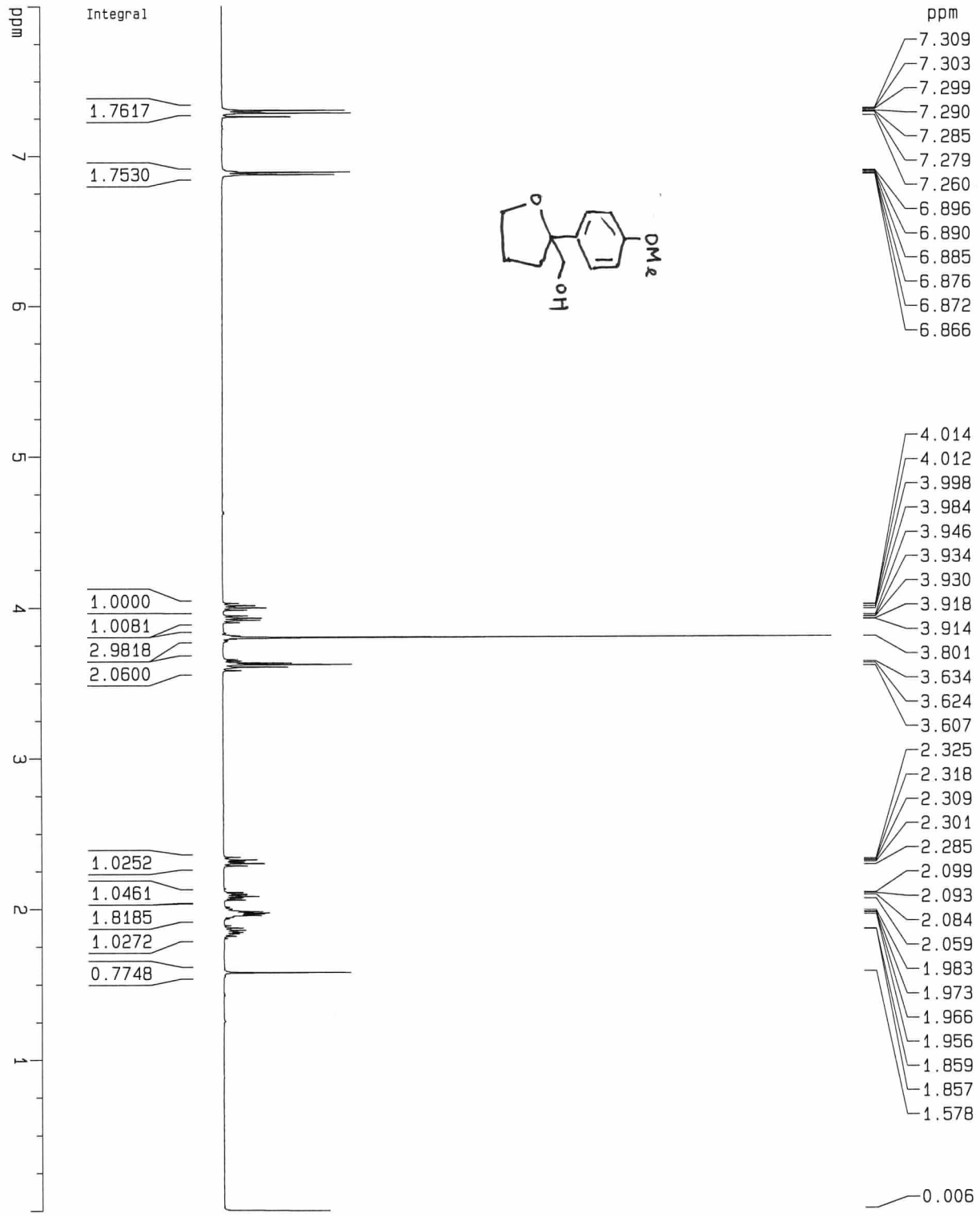
F1 30052.33 Hz

F2P -59.970 ppm

F2 -7541.65 Hz

PPMCM 14.94698 ppm/cm

HZCM 1879.69897 Hz/cm



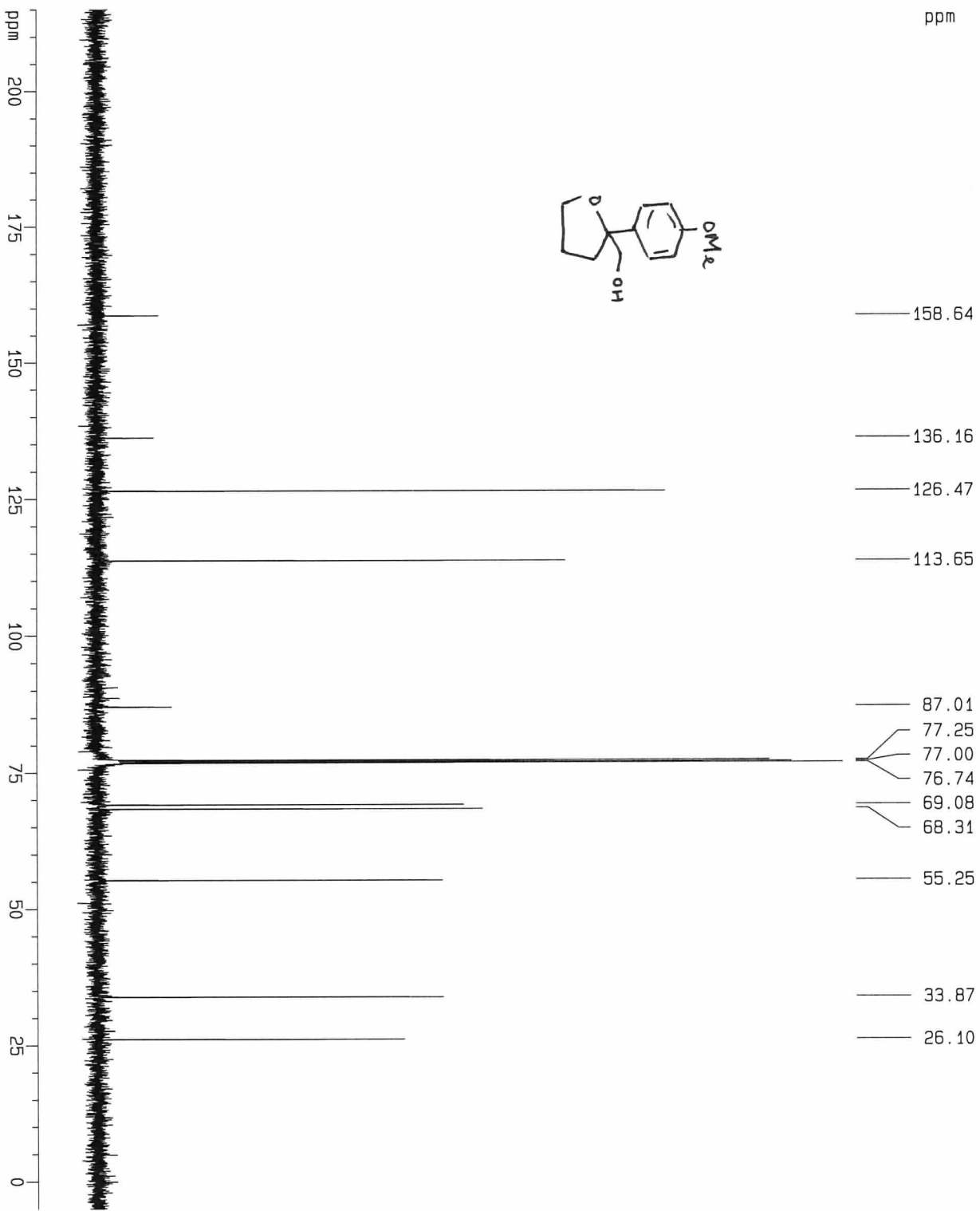
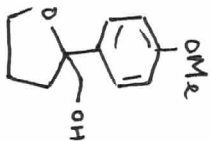
Current Data Parameters
 NAME ZL7R322H192A2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20100430
 Time 20.05
 INSTRUM spect
 PULPROG 5 mm QNP 1H
 TD 48076
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.166670 Hz
 AQ 2.9999924 sec
 RG 256
 DW 62.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 3.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SF01 500.1325006 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1300131 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.000 ppm
 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 200.05200 Hz/cm



Current Data Parameters
 NAME ZL7R32C192A2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20100430
 Time 20.13
 INSTRUM spect
 PROBHID 5 mm QNP 1H
 PULPROG zgpg
 TD 75184
 SOLVENT C6D6
 NS 1508
 DS 0
 SMH 37593.984 Hz
 FIDRES 0.500026 Hz
 AQ 0.9999972 sec
 RG 8192
 DW 13.300 usec
 DE 7.50 usec
 TE 300.0 K
 D1 0.10000000 sec
 D11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
 P1 4.60 usec
 PL1 0.00 dB
 SF01 125.7690572 MHz

===== CHANNEL f2 =====

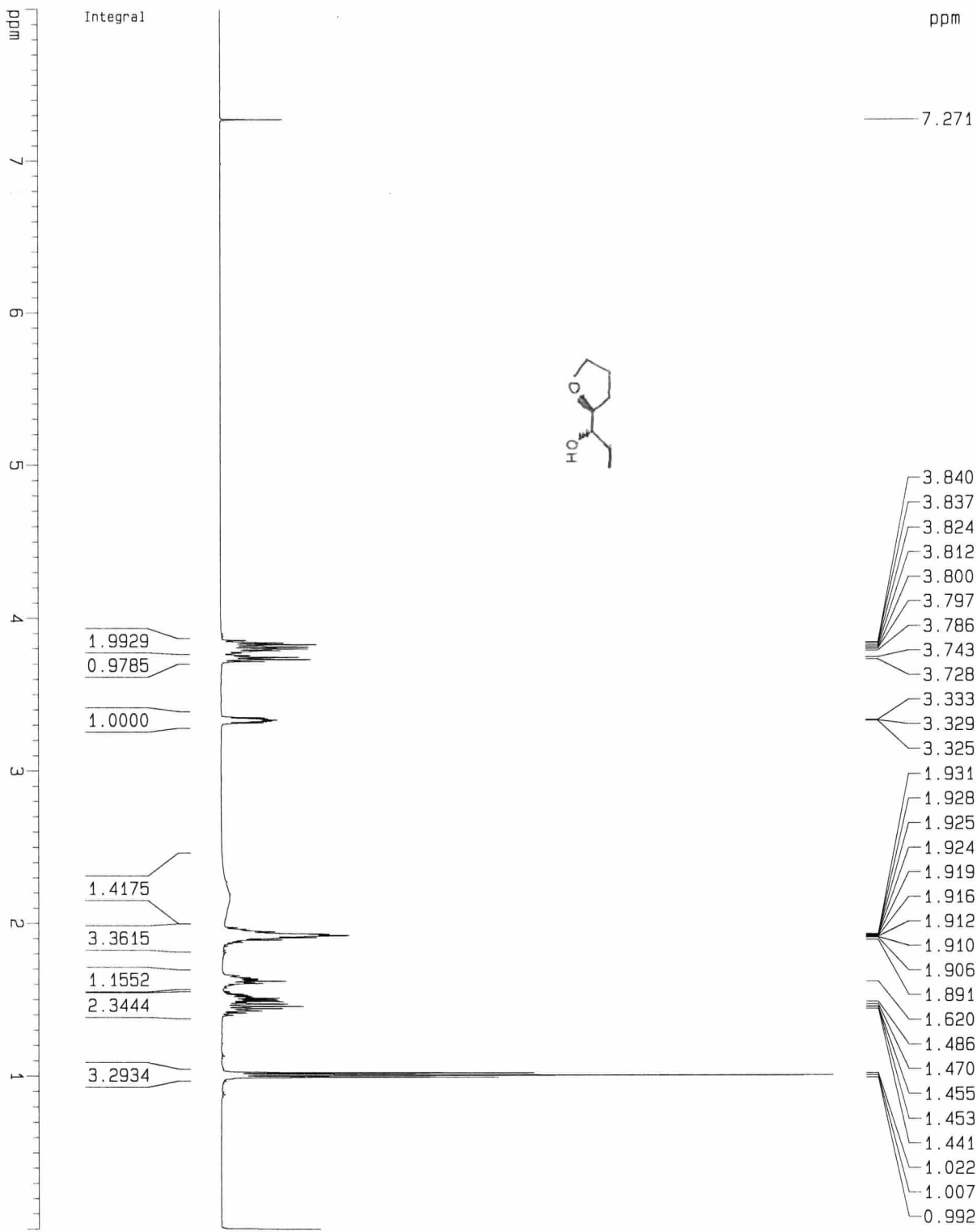
CPDPRG2 wa1tz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SF02 500.1320005 MHz

F2 - Processing parameters

SI 32768
 SF 125.7577918 MHz
 KDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 FAP 215.000 ppm
 F1 27037.93 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1383.33569 Hz/cm



Current Data Parameters
 NAME ZL4R20H220A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080727
 Time 16.35
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 48076
 SOLVENT CDC13
 NS 8
 DS 0
 SMH 8012.820 Hz
 FIDRES 0.166670 Hz
 AQ 2.9999924 sec
 RG 64
 DW 62.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 3.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.50 usec
 PL1 0.00 dB
 SF01 500.1325006 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1300086 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.000 ppm
 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 200.05200 Hz/cm