Highly Selective 1,3-Isomerization of Allylic Alcohols Via Rhenium Oxo Catalysis

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Supporting Information

Experimental procedures and characterization data (^{1}H and ^{13}C NMR, HRMS) for compounds **2**-**22**.

General Experimental Section. NMR spectra were recorded on an Oxford 300 MHz NMR spectrometer running Varian VNMR software. Chemical shifts are reported in parts per million (ppm) downfield from tetramethylsilane (TMS) with reference to internal solvent. Multiplicities are abbreviated as follows: singlet (s), doublet (d), triplet (t), quartet (q), quintet (quint), multiplet (m), and broad (br). The reported ^{1}H NMR and ^{13}C NMR data refer to the major olefin isomer (which is identified) except for cases in which the minor isomer showed significantly prominent peaks, in which case the two isomers were separated and individually characterized. Assignments of *E*/*Z* stereochemistry were made for compounds **12**, **14**, **16**, and **18** based on NOE experiments performed on each isomer of **12** and **18** and the *E*-isomers of **14** and **16**. High-resolution mass spectra (EI and CI) were provided by California Institute of Technology Mass Spectrometry Facility. Molecular mass calculations were performed with ChemDraw Ultra 8.0.3 (Cambridge Scientific).

GC and HPLC data were obtained using an Agilent 6850 Series GC system and an Agilent 1100 Series HPLC, respectively. Optical rotations were measured using a Jasco P-1010 Polarimeter. Reactions temperatures from –10 ˚C to –50 ˚C were obtained using a Neslab CC-100 Cryotrol. Analytical thin-layer chromatography (TLC) was performed using silica gel 60 F254 precoated plates (0.25 mm thickness) with a fluorescent indicator. Visualization was performed with standard potassium permanganate stains. Flash column chromatography was performed using silica gel 60 (230-400 mesh) from EM Science. Catalyst **1** was synthesized in a glove box according to the published procedure, purified by filtration of the reaction solution through Celite and subsequent recrystallization from diethyl ether, and stored the glove box. Benzaldehyde was distilled from calcium hydride. Diethyl ether, dichloromethane, benzene, and tetrahydrofuran were purified and dried by passage through a solvent column.[i] All other chemicals were used as purchased.

Experimental Procedures and Characterization Data.

(*E*)-3-cyclohexyl-1-phenylprop-2-en-1-ol **(2)**. To a flame-dried, round-bottomed flask under argon atmosphere, added CH_2Cl_2 (15 mL), 1-phenyl-2-propenyl acetate (1.0 mL, 6.2 mmol), and vinylcyclohexane (700 μL, 5.1 mmol). Then added, via cannula transfer, a solution of $RuCl_2(PCy_3)(H_2IMes)CHPh$ (136 mg, 0.16 mmol) and CH_2Cl_2 (10 mL). Placed in 45 °C oil bath and let stir for approximately 24 hours. Allowed to cool to room temperature, removed solvent *in vacuo*, and added 3.0 M aqueous NaOH (1.2 mL, 3.6 mmol), THF (45 mL), and MeOH (9 mL). Let stir at room temperature for approximately 3 hours. Added 40 mL aqueous ammonium chloride, extracted 3 times with 25 mL ether, and dried with Na_2SO_4. The product was purified via silica gel chromatography (8:2

pentane:ether) to obtain 636 mg of an orange oil (58% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.20 (5H, m), 5.63 (1H, dd, *J* = 15.5, 5.9 Hz), 5.51 (1H, ddd, *J* = 15.5, 6.5, 0.9 Hz), 5.05 (1H, d, *J* = 6.6 Hz), 1.9 (2H, m), 1.6 (5H, m), 1.1 (5H, m). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 143.6, 138.7, 130.0, 128.6, 127.6, 126.4, 75.5, 40.4, 32.92, 32.88, 26.3, 26.2. HRMS (EI) calcd. for $C_{15}H_{20}O$: 216.1514, found: 216.1507.

(*E*)-1-cyclohexyl-3-phenylprop-2-en-1-ol **(3)**. In glove box, added **1** (4 mg, 0.008 mmol) to 4-mL vial. Removed from glove box, added ether (2 mL), placed in Cryotrol set to –50 °C, and let stir for approximately 10 minutes. Added **4** (86.2 mg, 0.4 mmol) via syringe and let stir at –50 °C for 30 minutes. Removed from Cryotrol, immediately added 20 μL triethylamine, and let stir until warmed to room temperature. Concentrated *in vacuo* and purified directly via silica gel chromatography (8:2 pentane:ether) to obtain 80.2 mg of a clear oil (93% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.3 (5H, m), 6.58 (1H, d, *J* = 15.9 Hz), 6.26 (1H, ddd, *J* = 15.8, 7.1, 1.1 Hz), 4.05 (1H, t, *J* = 6.6 Hz), 1.95 (1H, d, *J* = 13.5 Hz), 1.75 (5H, m), 1.55 (1H, m), 1.2 (5H, m). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 137.0, 131.4, 131.2, 128.8, 127.8, 126.6, 77.8, 44.1, 29.1, 28.8, 26.7, 26.32, 26.26. HRMS (EI) calcd. for $C_{15}H_{20}O$: 216.1514, found: 216.1516.

(*Z*)-3-cyclohexyl-1-phenylprop-2-en-1-ol **(4)**. To flame-dried, round-bottomed flask under argon atmosphere, added (*Z*)-(2-bromovinyl)cyclohexane[ii] (374 mg, 2.0 mmol) and ether (10 mL). Placed in dry ice/acetone bath and let stir for approximately 10 minutes. Added dropwise a 1.7 M pentane solution of *t*-butyllithium (3 mL, 5.1 mmol). Let stir for 1 hour. Added benzaldehyde (200 μL, 2.0 mmol) via syringe, let stir for 20 minutes at –78 °C for

20 minutes, removed dry ice/acetone bath, and let stir for 20 minutes. Slowly added 10 mL aqueous ammonium chloride, extracted 3 times with ether, and dried with Na_2SO_4. Purified via silica gel chromatography to obtain 288 mg of a cloudy oil, which turned out to be a 1.5:1 mixture of **4** and 3-cyclohexyl-1-phenylprop-2-yn-1-ol. To convert this mixture to the desired product, it was added to MeOH (3 mL) and Lindlar catalyst (5% Pd on $CaCO_3$, poisoned with Pb, 45 mg), degassed via 3 freeze-pump-thaw cycles, and placed under H_2 atmosphere for approximately 26 hours. The reaction solution was filtered through Celite, rinsing with ether, and purified via silica gel chromatography (8:2 pentane:ether) to obtain 165 mg of a clear oil, which was ≥ 97% **4** by GC (37% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.35 (5H, m), 5.5 (3H, m), 2.5 (1H, m), 1.7 (6H, m), 1.2 (5H, m). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 144.0, 138.6, 130.0, 128.7, 127.6, 126.1, 70.1, 37.1, 33.6, 33.3, 26.1, 26.0, 25.9. HRMS (EI) calcd. for $C_{15}H_{20}O$: 216.1514, found: 216.1519.

1-(2-nitrophenyl)prop-2-en-1-ol **(5b)**. To flame-dried, round-bottomed flask under argon atmosphere, added 2-nitrobenzaldehyde (7.5 g, 50 mmol) and ether (100 mL). Placed in ice bath and let stir approximately 10 minutes. Added 1.0 M THF solution of vinylmagnesium bromide dropwise, over approximately 30 minutes. Let stir at 0 °C for 1 hour, then slowly added 150 mL aqueous dilute acid, extracted 3 times with 100 mL ether, and dried with Na_2SO_4. Purified via silica gel chromatography (7:3 pentane:ether) to obtain approximately 1.8 g of a reddish-brown oil (20% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.92 (1H, dd, J = 8.1, 1.2 Hz), 7.77 (1H, dd, J = 7.8, 1.5 Hz), 7.65 (1H, td, J = 7.6, 1.2 Hz), 7.45 (1H, ddd, J = 8.3, 7.4, 1.5 Hz), 6.09 (1H, ddd, J = 17.3, 10.4, 5.3 Hz),

5.80 (1H, dt, *J* = 5.1, 1.5 Hz), 5.42 (1H, dt, *J* = 17.1, 1.4 Hz), 5.27 (1H, dt, *J* = 10.5, 1.4 Hz), 2.6 (1H, br). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 148.5, 138.1, 137.7, 133.8, 129.1, 128.7, 124.8, 116.4, 70.2. HRMS (CI) calcd. for $C_9H_9NO_3 + NH_4$: 197.0926, found: 197.0918.

1-(2-methoxyphenyl)prop-2-en-1-ol **(5c)**. Followed same procedure as for **5b** using *o*-anisaldehyde (5.5 mL, 46 mmol), ether (90 mL), and 1.0 THF solution of vinylmagnesium bromide (90 mL, 90 mmol). Purified via silica gel chromatography (7:3 hexanes:ethyl acetate) to obtain 6.7 g of a yellow oil (89% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.2 (2H, m), 6.85 (2H, m), 6.07 (1H, ddd, *J* = 17.3, 10.4, 5.6 Hz), 5.34 (1H, t, *J* = 5.7 Hz), 5.24 (1H, d, *J* = 17.4 Hz), 5.10 (1H, d, *J* = 10.5 Hz), 3.79 (3H, s), 2.70 (1H, d, *J* = 6.0 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 156.9, 139.6, 130.9, 129.0, 127.6, 121.1, 114.7, 110.9, 71.8, 55.6. HRMS (EI) calcd. for $C_{10}H_{12}O_2$: 164.0837, found: 164.0839.

(*E*)-3-(2-nitrophenyl)prop-2-en-1-ol **(6b)**. In glove box, added **1** (4 mg, 0.008 mmol) to 4-mL vial. Removed from glove box, added CH_2Cl_2 (2 mL), and let stir at room temperature for approximately 10 minutes. Added **5b** (71.9 mg, 0.4 mmol) via syringe and let stir at room temperature for 30 minutes. Added 20 μL triethylamine, concentrated *in vacuo*, and purified directly via silica gel chromatography (7:3 pentane:ether) to obtain 70.5 mg of a clear oil (98% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.90 (1H, dd, *J* = 8.3, 1.1 Hz), 7.80 (2H, m), 7.38 (1H, ddd, *J* = 8.4, 6.8, 2.0 Hz), 7.07 (1H, dt, *J* = 15.9, 1.7 Hz), 6.34 (1H, dt, *J* = 15.6, 5.3 Hz), 4.37 (2H, dd, *J* = 5.3, 1.7 Hz), 2.31 (1H, br). ^{13}C NMR (300

MHz, $CDCl_3$, ppm): δ 147.9, 134.3, 133.3, 132.7, 128.9, 128.3, 125.9, 124.7, 63.4. HRMS (EI) calcd. for $C_9H_9NO_3$: 179.0582, found: 179.0584.

(*E*)-3-(2-methoxyphenyl)prop-2-en-1-ol **(6c)**. Followed same procedure as for **3** using **1** (4 mg, 0.008 mmol), **5c** (65.6 mg, 0.4 mmol), and ether (2 mL). Purified via silica gel chromatography (7:3 pentane:ether) to obtain 42.3 mg of an oil (65% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.45 (1H, dd, *J* = 7.7, 1.7 Hz), 7.25 (1H, ddd, *J* = 8.1, 7.4, 1.8 Hz), 6.93 (3H, m), 6.39 (1H, dt, *J* = 16.2, 5.9 Hz), 4.33 (1H, dd, *J* = 6.0, 1.5 Hz), 3.86 (3H, s), 1.74 (1H, br). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 156.9, 129.5, 128.9, 127.2, 126.3, 125.9, 120.8, 111.0, 64.4, 55.6. HRMS (EI) calcd. for $C_{10}H_{12}O_2$: 164.0837, found: 164.0837.

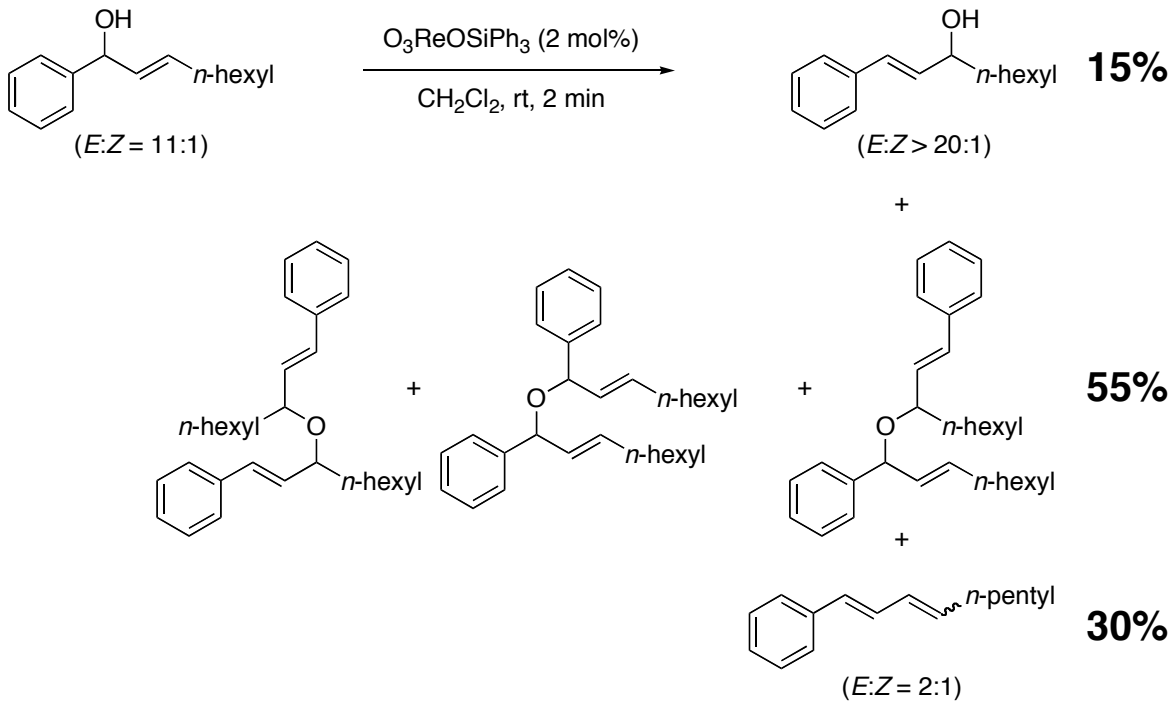
(*E*)-1-phenylnon-2-en-1-ol **(7a)**. Followed same procedure as for **2** using 1-phenyl-2-propenyl acetate (2.4 mL, 15 mmol), 1-octene (2.0 mL, 12.7 mmol), $RuCl_2(PCy_3)(H_2IMes)CHPh$ (270 mg, 0.32 mmol), and CH_2Cl_2 (40 mL). Purified via silica gel chromatography (8:2 pentane:ether, 2 sequential columns) to obtain 1.5 g of a yellow oil (54% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.35 (5H, m), 5.75 (2H, m), 5.18 (1H, dd, *J* = 6.0, 2.4 Hz), 2.07 (2H, dt, *J* = 6.7, 6.7 Hz), 1.91 (1H, d, *J* = 3.0 Hz), 1.3 (8H, m), 0.89 (3H, t, *J* = 6.6 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 143.6, 133.1, 132.4, 128.7, 127.7, 126.4, 75.4, 32.4, 31.9, 29.2, 29.1, 22.8, 14.3. HRMS (EI) calcd. for $C_{15}H_{22}O$: 218.1671, found: 218.1666.

(*E*)-1-(2-nitrophenyl)non-2-en-1-ol **(7b)**. Followed same procedure as for **2** using 1-(2-nitrophenyl)allyl acetate (1.3 mL, 7.6 mmol), 1-octene (1.0 mL, 6.4 mmol), $RuCl_2(PCy_3)(H_2IMes)CHPh$ (135 mg, 0.16 mmol), and CH_2Cl_2 (32 mL). Purified via silica gel chromatography (7:3 pentane:ether) to obtain 650 mg of an orange oil (39% yield). ^1H NMR (300 MHz, $CDCl_3$, ppm): δ 7.88 (1H, dd, *J* = 8.4, 1.2 Hz), 7.79 (1H, dd, *J* = 8.0, 1.4 Hz), 7.63 (1H, td, *J* = 7.6, 1.3 Hz), 7.43 (1H, ddd, *J* = 8.0, 7.3, 1.4 Hz), 5.8 (2H, m), 5.7 (1H, m), 2.41 (1H, br), 2.05 (2H, dt, *J* = 7.0, 7.0 Hz), 1.3 (8H, m), 0.88 (3H, t, *J* = 6.8 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 148.5, 138.4, 134.4, 133.5, 129.9, 128.7, 128.4, 124.7, 70.2, 32.4, 31.9, 29.1, 29.0, 22.8, 14.3. HRMS (EI) calcd. for $C_{15}H_{21}NO_3$ – H: 262.1438, found: 262.1439.

(*E*)-1-(2-methoxyphenyl)non-2-en-1-ol **(7c)**. Followed same procedure as for **2** using 1-(2-methoxyphenyl)prop-2-en-1-ol (2.5 mL, 14.5 mmol), 1-octene (2.0 mL, 12.7 mmol), $RuCl_2(PCy_3)(H_2IMes)CHPh$ (270 mg, 0.32 mmol), and CH_2Cl_2 (40 mL). Purified via silica gel chromatography (8:2 pentane:ether) to obtain 1.5 g of a yellow oil (48% yield). ^1H NMR (300 MHz, $CDCl_3$, ppm): δ 7.20 (2H, m), 6.86 (2H, m), 5.65 (2H, m), 5.28 (1H, d, *J* = 5.7 Hz), 3.79 (3H, s), 2.68 (1H, d, *J* = 6.0 Hz), 1.97 (2H, m), 1.25 (8H, m), 0.80 (3H, t, *J* = 6.8 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 156.9, 132.4, 131.8, 131.2, 128.7, 127.6, 121.0, 110.9, 71.9, 55.6, 32.5, 31.9, 29.3, 29.1, 22.8, 14.3. HRMS (EI) calcd. for $C_{16}H_{24}O_2$: 248.1776, found: 248.1780.

(*E*)-1-phenylnon-1-en-3-ol **(8a)**. Followed same procedure as for **3** using **1** (4 mg, 0.008 mmol), **7a** (84.8 mg, 0.4 mmol), and ether (2 mL). Purified via silica gel chromatography (8:2 pentane:ether) to obtain 83.6 mg of a clear oil (98% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.35 (5H, m), 6.60 (1H, d, *J* = 15.9 Hz), 6.25 (1H, dd, *J* = 15.9, 6.9 Hz), 4.30 (1H, dt, *J* = 6.3, 6.3 Hz), 1.65 (3H, m), 1.4 (8H, m), 0.91 (3H, t, *J* = 6.8 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 137.0, 132.8, 130.4, 128.8, 127.8, 126.7, 73.3, 37.6, 32.0, 29.5, 25.6, 22.8, 14.3. HRMS (EI) calcd. for $C_{15}H_{22}O$: 218.1671, found: 218.1670.

When performed in CH_2Cl_2 at room temperature, the reaction resulted in the following:



Note that these numbers do not represent a quantitative determination of yield. They were calculated from the relative amounts of the various products observed in the crude reaction mixture (after quenching with triethylamine) by ^{1}H NMR spectroscopy. No starting material was visible by ^{1}H NMR spectroscopy in this mixture.

(*E*)-1-(2-nitrophenyl)non-1-en-3-ol **(8b)**. Followed same procedure as for **6b** using **1** (4 mg, 0.008 mmol), **7b** (104.8 mg, 0.4 mmol), and ether (2 mL). Purified via silica gel chromatography (1:1 pentane:ether) to obtain 104.2 mg of a reddish oil (98% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.91 (1H, dd, *J* = 8.3, 0.8 Hz), 7.57 (2H, m), 7.38 (1H, ddd, *J* = 8.4, 6.8, 2.0 Hz), 7.02 (1H, dd, *J* = 15.6, 0.6 Hz), 6.20 (1H, dd, *J* = 15.9, 6.6 Hz), 4.33 (1H, dt, *J* = 6.2, 6.2 Hz), 2.1 (1H, br), 1.65 (2H, m), 1.35 (8H, m), 0.88 (3H, t, *J* = 6.8 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 148.0, 138.3, 133.2, 132.8, 128.9, 128.2, 125.4, 124.7, 77.7, 37.3, 31.9, 29.4, 25.5, 22.8, 14.3. HRMS (EI) calcd. for $C_{15}H_{21}NO_3$ – H: 262.1438, found: 262.1439.

(*E*)-1-(2-methoxyphenyl)non-1-en-3-ol **(8c)**. Followed same procedure as for **3** using **1** (4 mg, 0.008 mmol), **7c** (99.5 mg, 0.4 mmol), and ether (2 mL). Purified via silica gel chromatography (8:2 pentane:ether) to obtain 67.4 mg of a clear oil (68% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.45 (1H, dd, *J* = 7.5, 1.8 Hz), 7.24 (1H, m), 6.90 (3H, m), 6.24 (1H, dd, *J* = 16.1, 7.1 Hz), 4.29 (1H, dt, *J* = 6.6, 6.6 Hz), 3.86 (3H, s), 1.5 (11H, m), 0.90 (3H, t, *J* = 6.8 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 156.9, 133.5, 128.9, 127.0, 125.9, 125.2, 120.8, 111.0, 73.8, 55.6, 37.5, 32.0, 29.5, 25.7, 22.8, 14.3. HRMS (EI) calcd. for $C_{16}H_{24}O_2$: 248.1776, found: 248.1783.

(*E*)-1-(thiophen-2-yl)non-2-en-1-ol **(9)**. Step 1. To flame-dried, round-bottomed flask under argon atmosphere, added 1-octyne (5 mL, 34 mmol) and ether (30 mL). Placed in dry ice/acetone bath and let stir for approximately 10 minutes. Added a 1.6 hexanes solution of *n*-butyllithium (16 mL, 26 mmol) dropwise and let stir at –78 °C for 1 hour.

Added a solution of 2-thiophene-carboxaldehyde (2 mL, 22 mmol) and ether (5 mL) dropwise and let stir at –78 °C for 1 hour. Removed dry ice/acetone bath and let stir for 1.5 hours. Placed in ice bath, slowly added 50 mL aqueous ammonium chloride, extracted 3 times with 50 mL ether, and dried with Na_2SO_4. Purified via silica gel chromatography (8:2 pentane:ether) to obtain 3.4 g (69% yield) of 1-(thiophen-2-yl)non-2-yn-1-ol[iii] as a bright yellow oil (contained ca. 15% 2-thiophene-carboxaldehyde). Step 2. Added this product mixture and THF (120 mL) to flame-dried, round-bottomed flask under argon atmosphere. Placed in ice bath and let stir for approximately 10 minutes. Added a 1.0 THF solution of lithium aluminum hydride (50 mL, 50 mmol) dropwise and removed ice bath shortly thereafter. Let stir at room temperature for 28 hours. Placed in ice bath, slowly added 100 mL ethyl acetate and several scoops of $Na_2SO_4 \bullet H_2O$. Removed ice bath, let stir for approximately 20 minutes, and then filtered through Celite, rinsing with ether. Purified via silica gel chromatography (8:2 pentane:ether) to obtain 2.3 g of a yellow oil (67% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.26 (1H, m), 6.98 (2H, m), 5.80 (2H, m), 5.39 (1H, m), 2.08 (3H, m), 1.34 (8H, m), 0.89 (3H, t, J = 6.9 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 147.9, 133.7, 131.6, 126.9, 125.2, 124.2, 71.3, 32.3, 31.9, 29.1, 29.0, 22.8, 14.3. HRMS (EI) calcd. for $C_{13}H_{20}OS$: 224.1235, found: 224.1233.

(*E*)-1-(thiophen-2-yl)non-1-en-3-ol **(10)**. Followed same procedure as for **3** using **1** (4 mg, 0.008 mmol), **9** (89.9 mg, 0.4 mmol), and ether (2 mL) with a 15 minute reaction time. Purified via silica gel chromatography (8:2 pentane:ether) to obtain 89.6 mg of an oil (92% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.16 (1H, m), 6.96 (2H, m), 6.71 (1H, dd, J = 15.8, 0.8 Hz), 6.07 (1H, dd, J = 15.9, 6.6 Hz), 4.24 (1H, dt, J = 6.6, 6.6 Hz), 1.76 (1H, br),

1.6 (2H, m), 1.35 (8H, m), 0.90 (3H, t, J = 6.8 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 142.1, 132.5, 127.5, 125.9, 124.4, 123.5, 73.0, 37.5, 32.0, 29.4, 25.6, 22.8, 14.3. HRMS (EI) calcd. for $C_{13}H_{20}OS$: 224.1235, found: 224.1229.

2-phenylbut-3-en-2-ol **(11)**. To flame-dried, round-bottomed flask under argon atmosphere, added acetophenone (6 mL, 51 mmol) and ether (100 mL). Placed in ice bath and let stir for approximately 10 minutes. Added a 1.0 M THF solution of vinylmagnesium bromide (100 mL, 100 mmol) dropwise. Let sir for 30 minutes at 0 °C and 1 hour at room temperature. Placed in ice bath, slowly added 100 mL aqueous ammonium chloride, extracted 3 times with 100 mL ether, and dried with Na_2SO_4. The crude product contained a small amount of the self-aldol product, 3-hydroxy-1,3-diphenylbutan-1-one, which was difficult to separate by column chromatography. The following procedure was employed to convert this side product into the more separable 1,3-diphenylbut-2-en-1-one: Removed solvent *in vacuo*, then added a 3.0 M aqueous solution of NaOH (12 mL, 36 mmol), THF (100 mL), and MeOH (20 mL). Let stir at room temperature for approximately 15 hours and worked up as before. Purified via silica gel chromatography (8:2 pentane:ether) to obtain approximately 2.2g of a yellow oil (30% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.55 (2H, m), 7.40 (2H, m), 7.30 (1H, m), 6.22 (1H, dd, J = 17.3, 10.7 Hz), 5.34 (1H, dd, J = 17.4, 1.2 Hz), 5.19 (1H, dd, J = 10.8, 1.1 Hz), 1.93 (1H, br), 1.70 (3H, s). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 146.6, 145.1, 128.5, 127.2, 125.4, 112.6, 75.0, 29.6. HRMS (EI) calcd. for $C_{10}H_{12}O$: 148.0888, found: 148.0885.

3-phenylbut-2-en-1-ol **(12)**. In glove box, added **1** (4 mg, 0.008 mmol) to 4-mL vial. Removed from glove box, added ether (2 mL) and *N*,*O*-bis(trimethylsilyl)-acetamide (120 μL, 0.485 mmol). Placed in Cryotrol set to –10 °C and let stir for approximately 10 minutes. Added **11** (59.4 mg, 0.4 mmol) via syringe and let stir at –10 °C for 30 minutes. Removed from cold bath, immediately added 20 μL triethylamine, and removed solvent *in vacuo*. Added MeOH (2 mL) and K_2CO_3 (110 mg, 0.8 mmol), then let stir at room temperature for 1 hour. Added 2 mL aqueous ammonium chloride, extracted several times with CH_2Cl_2, and dried with Na_2SO_4. Purified via silica gel chromatography (8:2 pentane:ether) to obtain 54.4 mg of an oil (92% yield). **(*E*-isomer)**. ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.45 (2H, m), 7.34 (3H, m), 6.01 (1H, dt, *J* = 6.68, 1.4 Hz), 4.40 (2H, d, *J* = 6.6 Hz), 2.12 (3H, s), 1.74 (1H, br). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 143.0, 138.0, 128.5, 127.5, 126.7, 126.0, 60.1, 16.2. HRMS (EI) calcd. for $C_{10}H_{12}O$: 148.0888, found: 148.0887. **(*Z*-isomer)**. ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.35 (3H, m), 7.21 (2H, m), 5.74 (1H, dt, *J* = 7.05, 1.3 Hz), 4.10 (2H, dd, *J* = 6.9, 1.2 Hz), 2.12 (3H, s), 1.53 (1H, br). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 141.0, 140.4, 128.3, 127.9, 127.4, 126.3, 60.5, 25.5. HRMS (EI) calcd. for $C_{10}H_{12}O$: 148.0888, found: 148.0891.

2-cyclohexylbut-3-en-2-ol **(13)**. Followed same procedure as for **11** using cyclohexyl methyl ketone (9 mL, 70 mmol), ether (100 mL), and a 1.0 THF solution of vinylmagnesium bromide (100 mL, 100 mmol). Purified via silica gel chromatography (8:2 pentane:ether) to obtain 4.3 g of a yellow oil (40% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 5.92 (1H, dd, *J* = 17.4, 10.8 Hz), 5.19 (1H, dd, *J* = 17.4, 1.5 Hz), 5.07 (1H, dd, *J* = 10.7, 1.4 Hz), 1.7 (5H, m), 1.24 (3H, s), 1.2 (7H, m). ^{13}C NMR (300 MHz, $CDCl_3$,

ppm): δ 144.6, 112.1, 75.5, 48.2, 27.6, 27.2, 26.8, 26.7, 26.6, 25.3. HRMS (EI) calcd. for $C_{10}H_{18}O$: 154.1358, found: 154.1353.

(*E*)-3-cyclohexylbut-2-en-1-ol **(14)**. Followed same procedure as for **12** using **1** (4 mg, 0.008 mmol), **13** (61.9 mg, 0.4 mmol), *N*,*O*-bis(trimethylsilyl)-acetamide (120 μL, 0.485 mmol), and ether (2 mL) with a reaction temperature of 0 °C. Purified via silica gel chromatography (8:2 pentane:ether) to obtain 55.1 mg of an oil (89% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 5.38 (1H, t, *J* = 6.8 Hz), 4.15 (2H, d, *J* = 6.6 Hz), 1.7 (7H, m), 1.64 (3H, s), 1.2 (5H, m). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 144.9, 121.7, 59.6, 47.3, 31.9, 26.8, 26.5, 14.8. HRMS (EI) calcd. for $C_{10}H_{18}O$: 154.1358, found: 154.1352.

3,4,4-trimethylpent-1-en-3-ol **(15)**. Followed same procedure as for **11** using pinacolone (6.5 mL, 52 mmol), ether (100 mL, 100 mmol), and a 1.0 THF solution of vinylmagnesium bromide (100 mL). Purified via silica gel chromatography (9:1 pentane:ether) to obtain 2.7 g of a yellow oil (40% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 6.09 (1H, dd, *J* = 17.6, 10.7 Hz), 5.23 (1H, dd, *J* = 17.4, 1.5 Hz), 5.09 (1H, dd, *J* = 11.0, 1.7 Hz), 1.41 (1H, br), 1.25 (3H, s), 0.95 (9H, s). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 143.5, 112.5, 77.5, 37.4, 25.5, 23.5. HRMS (EI) calcd. for $C_8H_{16}O$: 128.1201, found: 128.1196.

(*E*)-3,4,4-trimethylpent-2-en-1-ol **(16)**. Followed same procedure as for **12** using **1** (8 mg, 0.016 mmol), **15** (50.9 mg, 0.4 mmol), *N*,*O*-bis(trimethylsilyl)-acetamide (120 μL, 0.485 mmol), and ether (2 mL) with a reaction temperature of 0 °C and a reaction time of 1 hour. Purified via silica gel chromatography (8:2 pentane:ether) to obtain 40.7 mg of an oil (80%

yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 5.45 (1H, tq, *J* = 6.5, 1.1 Hz), 4.19 (2H, dd, *J* = 6.5, 0.8 Hz), 1.66 (3H, m), 1.45 (1H, br), 1.05 (9H, s). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 147.3, 120.6, 60.2, 36.3, 29.0, 13.0. HRMS (EI) calcd. for $C_8H_{16}O$: 128.1201, found: 128.1207.

3-methylhept-1-en-3-ol **(17)**. Followed same procedure as for **11** using 2-hexanone (6.5 mL, 53 mmol), ether (100 mL), and a 1.0 THF solution of vinylmagnesium bromide (100 mL, 100 mmol). Purified via silica gel chromatography (8:2 pentane:ether) to obtain 5.1 g of a yellow oil (67% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 5.93 (1H, dd, *J* = 17.3, 10.7 Hz), 5.21 (1H, dd, *J* = 17.4, 1.2 Hz), 5.05 (1H, dd, *J* = 10.7, 1.4 Hz), 1.55 (2H, m), 1.45 (1H, br), 1.3 (4H, m), 1.28 (3H, s), 0.91 (3H, t, *J* = 6.9 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 145.5, 111.7, 73.5, 42.3, 27.9, 26.3, 23.3, 14.3. HRMS (EI) calcd. for $C_8H_{16}O$: 128.1201, found: 128.1204.

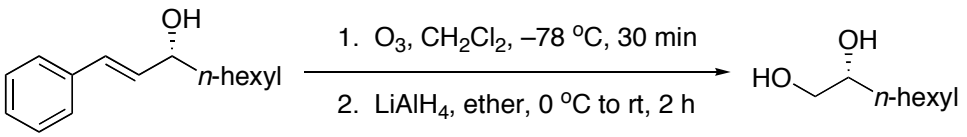
3-methylhept-2-en-1-ol **(18)**. Followed same procedure as for **12** using **1** (41 mg, 0.08 mmol), **17** (513 mg, 4.0 mmol), *N*,*O*-bis(trimethylsilyl)-acetamide (1.19 mL, 4.79 mmol), and ether (20 mL) with a reaction temperature of 0 °C. Purified via silica gel chromatography (7:3 pentane:ether) to obtain 485.7 mg of an oil (93% yield). **(*E*-isomer)**. ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 5.40 (1H, td, *J* = 6.9, 0.9 Hz), 4.15 (2H, d, *J* = 6.9 Hz), 2.01 (2H, t, *J* = 7.4 Hz), 1.67 (3H, s), 1.3 (5H, m), 0.90 (3H, t, *J* = 7.1 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 140.4, 123.3, 59.6, 39.4, 30.1, 22.6, 16.3, 14.2. HRMS (EI) calcd. for $C_8H_{16}O$: 128.1201, found: 128.1195. **(*Z*-isomer)**. ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 5.42 (1H, tt, *J* = 7.1, 0.8 Hz), 4.13 (2H, dd, *J* = 7.2, 0.9 Hz), 2.08 (2H, t, *J* = 7.4

Hz), 1.74 (3H, dt, *J* = 1.1, 1.1 Hz), 1.35 (5H, m), 0.91 (3H, t, *J* = 7.1 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 140.8, 124.1, 59.3, 31.9, 30.7, 23.7, 22.8, 14.2. HRMS (EI) calcd. for $C_8H_{16}O$: 128.1201, found: 128.1195.

(*R*,*E*)-1-phenylnon-2-en-1-ol **(19)**. Step 1. Prepared 1-phenylnon-2-yn-1-ol[iii] via the same procedure employed with **9** using 1-octyne (14 mL, 95 mmol), benzaldehyde (6 mL, 59 mmol), a 1.6 hexanes solution of *n*-butyllithium (40 mL, 64 mmol), and ether (100 mL). Purified via silica gel chromatography (8:2 pentane:ether) to obtain 10.6 g of an oil (83% yield). Step 2. To flame-dried, round-bottomed flask under argon atmosphere, added 1-phenylnon-2-yn-1-ol (10.4 g, 48 mmol), MnO_2 (49 g, 480 mmol), and benzene (200 mL). Let stir at room temperature for approximately 18 hours, then filtered through Celite, rinsing with ether. Purified via silica gel chromatography (9:1 pentane:ether) to obtain 9.0 g of 1-phenylnon-2-yn-1-one[iii] as a yellow oil (87% yield). Step 3. To a 3-neck, round-bottomed flask, added 4 Å molecular sieves (1 g) and flame-dried under vacuum. Let cool to room temperature, placed under argon atmosphere, added 1-phenylnon-2-yn-1-one (2 mL, 9.9 mmol) and THF (50 mL), and let stir at room temperature for 3 hours. Added a 1.0 M toluene solution of (*S*)-2-methyl-CBS-oxazaborolidine (25 mL, 25 mmol) and placed in dry ice/ethylene glycol:ethanol (8:2) bath. (Note: cold bath becomes solid, so flask should be placed in bath prior to dry ice addition.) When temperature had reached –30 to –40 °C range, added a 2.0 M THF solution of borane-methyl sulfide complex (25 mL, 50 mmol) dropwise, over approximately 15 minutes. Let stir at –30 to –40 °C for 3 hours, then, very slowly, added 40 mL MeOH and let warm to room temperature. Diluted with 150 mL ether, washed twice with 75 mL aqueous ammonium chloride, twice with 75

mL aqueous sodium bicarbonate, twice with 75 mL brine, and dried with Na_2SO_4. Purified via silica gel chromatography (8:2 pentane:ether) to obtain 2.0 g of (*S*)-1-phenylnon-2-yn-1-ol as a yellow oil (93% yield). Enantiomeric excess was determined to be 99% by chiral HPLC (OD-H column, 4% *i*-PrOH in hexanes, 1 mL/min). Step 4. Followed same procedure as for **9** using (*S*)-1-phenylnon-2-yn-1-ol (1.9 g, 8.9 mmol), a 1.0 M THF solution of lithium aluminum hydride (27 mL, 27 mmol), and THF (60 mL) with a reaction time of 41 hours. Purified via silica gel chromatography (8:2 pentane:ether) to obtain 1.65 g of a yellow oil (85% yield). Spectral data same as for **7a**. Enantiomeric excess was determined by chiral HPLC (OD-H column, 2% *i*-PrOH in hexanes, 1 mL/min). $[\alpha]_D$ = –34.2 (28 °C, $CHCl_3$, c = 1.0). Literature value[iv] for *S*-enantiomer of **19** (94% ee, 20 °C, $CHCl_3$, c = 2.0) = +34.4.

(*R*,*E*)-1-phenylnon-1-en-3-ol **(20)** and (*S*,*E*)-1-phenylnon-1-en-3-ol **(22)**. Followed same procedure as for **3** using **19** (87.3 mg, 0.4 mmol), **1** (6 mg, 0.012 mmol), and ether (2 mL) with a reaction temperature of –78 °C and a reaction time of 2 hours. Purified via silica gel chromatography (8:2 pentane:ether) to obtain 81.2 mg of **20** as a white solid (93% yield). Spectral data same as for **8a**. Enantiomeric excess determined by chiral HPLC (OJ column, 3% *I*-PrOH in hexanes, 1 mL/min). Absolute stereochemistry confirmed by conversion of **20** to **20'** according to the following procedure: To a 25-mL round-



20 **20'**

bottomed flask, added **20** (84% ee, 85 mg, 0.4 mmol) and CH_2Cl_2 (5 mL). Placed in dry ice/acetone bath and bubbled O_3 through the solution. Monitored by TLC, and when all of

20 had been consumed (about 30 minutes), stopped O_3 flow, purged with N_2 for 5 minutes, and placed in ice bath under argon atmosphere. Added ether (5 mL), and then slowly added a 1.0 ether solution of lithium aluminum hydride (800 μL, 0.8 mmol). Removed ice bath and let stir for 2 hours. Replaced ice bath, slowly added 3 mL ethyl acetate and a little $Na_2SO_4 \bullet H_2O$, removed ice bath, let stir for 15 minutes, and then filtered through Celite, rinsing with ether. Purified via silica gel chromatography (100% ether) to obtain 27 mg of **20'** (50% yield). $[\alpha]_D$ = +7.8 (28 °C, EtOH, c = 0.9). Literature value[v] for *S*-enantiomer of **20'** (>99% ee, 25 ˚C, EtOH, c = 0.33) = –15.4.

(*R*,*Z*)-1-phenylnon-2-en-1-ol **(21)**. To a flame-dried, round-bottomed flask under argon atmosphere, added (*S*)-1-phenylnon-2-yn-1-ol (synthesis described for **19**, 400 mg, 1.8 mmol), CH_2Cl_2 (5 mL), and *N*,*O*-bis(trimethylsilyl)-acetamide (670 μL, 2.7 mmol). Let stir at room temperature for 2 hours. Removed solvent and excess reagents *in vacuo*, added Lindlar catalyst (5% Pd on $CaCO_3$, poisoned with Pb, 170 mg) and methanol (5 mL). Degassed via 3 freeze-pump-thaw cycles and placed under H_2 atmosphere. Let stir at room temperature for 4 hours. Filtered through Celite, rinsing with ether. Dried with Na_2SO_4 and removed solvent *in vacuo*. Removed trimethylsilyl group as described for **12**. Crude ^{1}H NMR shows *Z*:*E* = 3:1. Purified by silica gel chromatography (9:1 pentane:ether) which afforded (with the sacrifice of a lot of material) 276 mg of **21** (28% yield, *Z*:*E* = 11:1, ca. 1% (*S*)-1-phenylnon-2-yn-1-ol). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 7.35 (5H, m), 5.6 (3H, m), 2.23 (2H, m), 2.02 (1H, br), 1.35 (8H, m), 0.92 (3H, t, *J* = 6.8 Hz). ^{13}C NMR (300 MHz, $CDCl_3$, ppm): δ 144.0, 132.6, 132.1, 128.7, 127.6, 126.1, 69.9, 31.9, 29.7, 29.2, 27.9, 22.8, 14.3. HRMS (EI) calcd. for $C_{15}H_{22}O$: 218.1671, found:

218.1672. Enantiomeric excess was determined by chiral HPLC (OB-H column, 3% *i*-PrOH in hexanes, 1 mL/min). $[\alpha]_D = -136.0$ (29 °C, $CHCl_3$, c = 1.1). Literature value[vi] for *S*-enantiomer of **21** (90% ee, 20-28 ˚C, $CHCl_3$, c = 0.3-1.7) = +168.7.

(i) The solvent columns are composed of activated alumina (A-2) and supported copper redox catalyst (Q-5 reactant). See: Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518-1520.

(ii) Prepared by analogous procedure to that reported in Morrill, C.; Grubbs, R. H. *J. Org. Chem.* **2003**, *68*, 6031-6034.

(iii) Luo, F. -T.; Bajji, A. C.; Jeevanandam, A. *J. Org. Chem.* **1999**, *64*, 1738-1740.

(iv) Oppolzer, W.; Radinov, R. N. *Helv. Chim. Acta* **1992**, *75*, 170-173.

(v) Burk, M. J.; Kalberg, C. S.; Pizzano, A. *J. Am. Chem. Soc.* **1998**, *120*, 4345-4353.

(vi) Oppolzer, W.; Radinov, R. N. *Tetrahedron Lett.* **1991**, *32*, 5777-5780.

Compound 2
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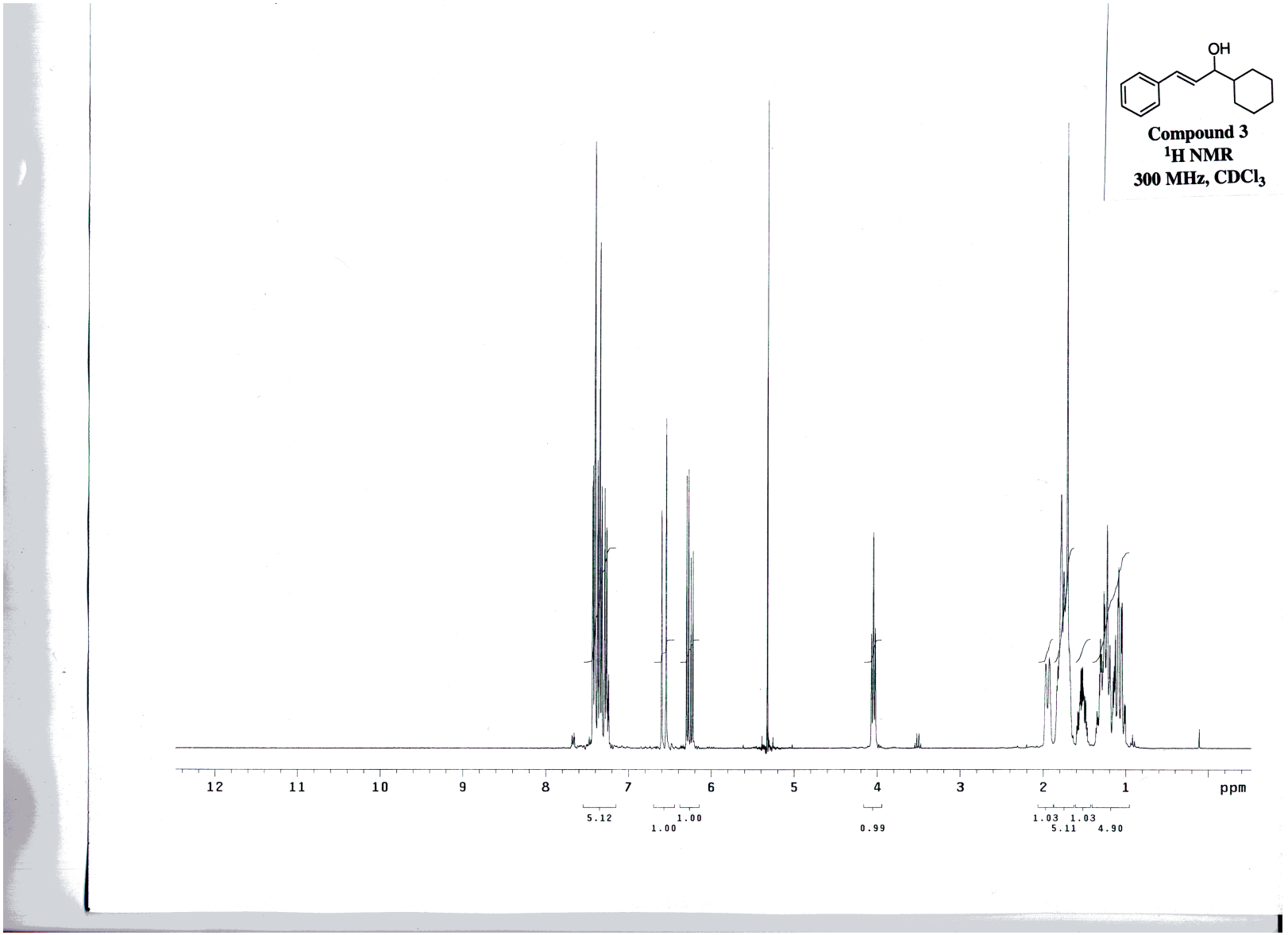
300 MHz, $CDCl_3$

Compound 2
^{13}C NMR
300 MHz, $CDCl_3$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4
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1.00
1.00
0.99
1.03
1.03
5.11
4.90

Compound 3
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300 MHz, $CDCl_3$

OH
Compound 4
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300 MHz, $CDCl_3$
ppm
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2
3
4
5
6
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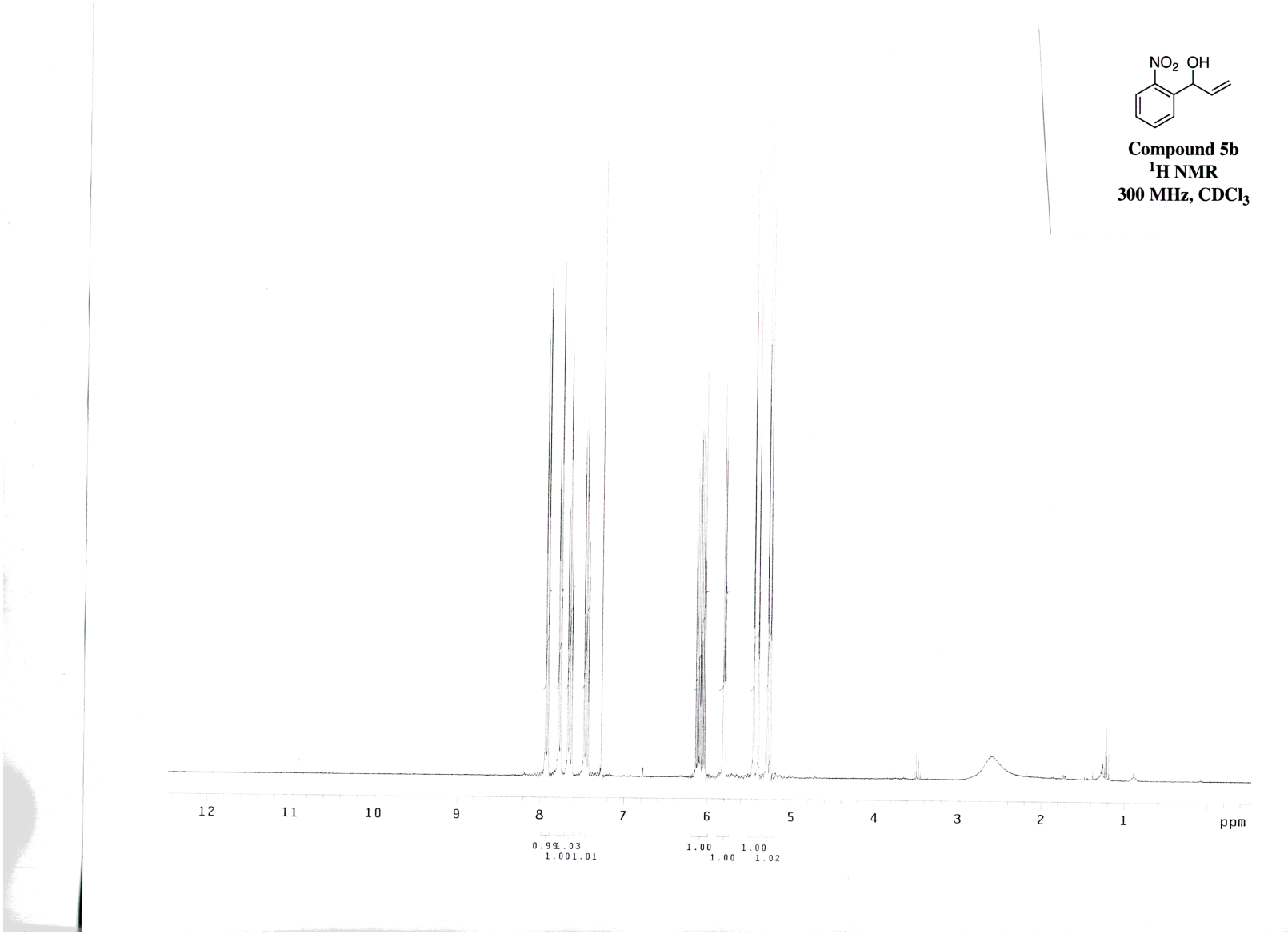

Compound 5b
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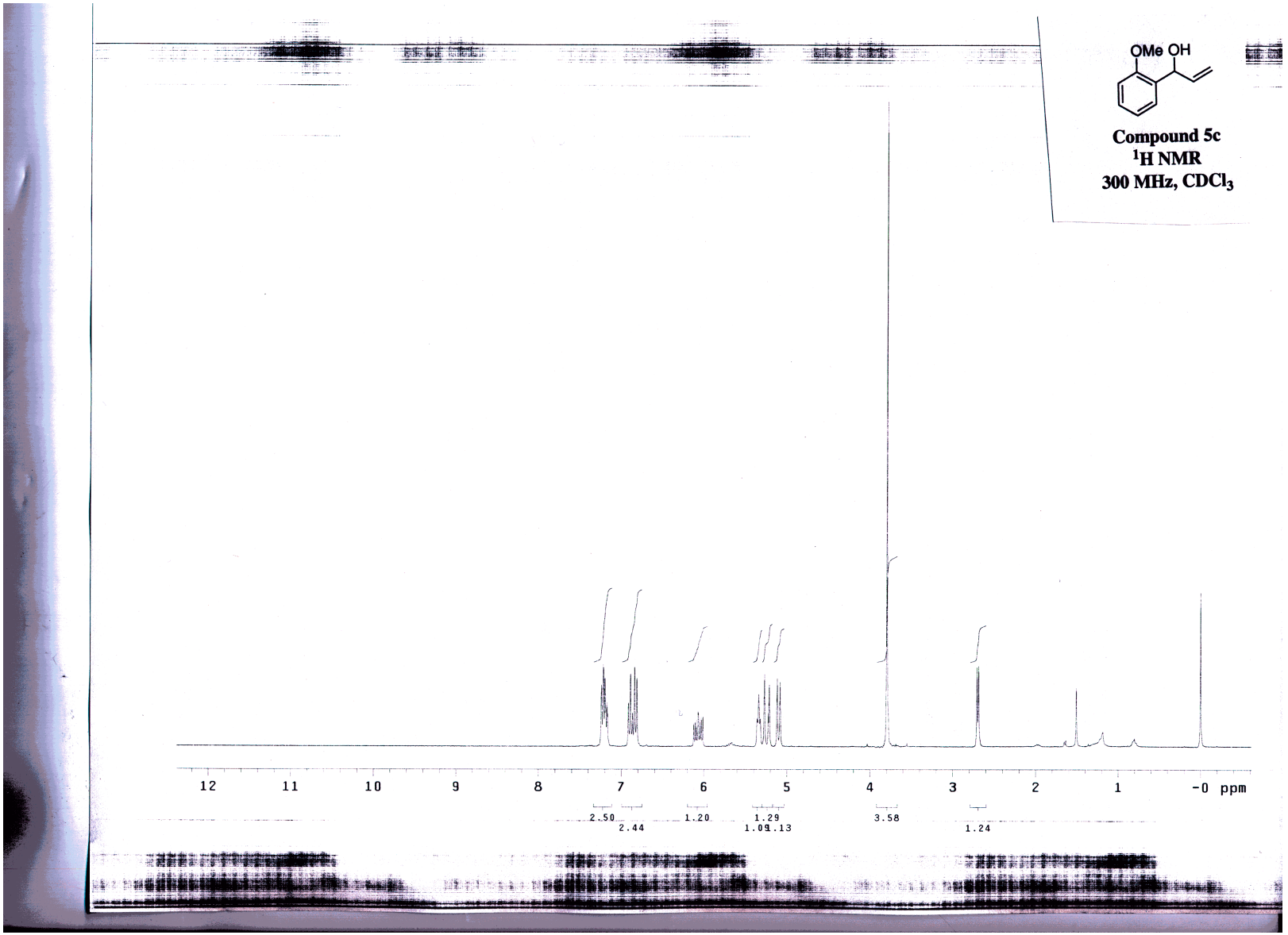
300 MHz, $CDCl_3$

Compound 5b
^{13}C NMR
300 MHz, $CDCl_3$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10
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8
7
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1.29
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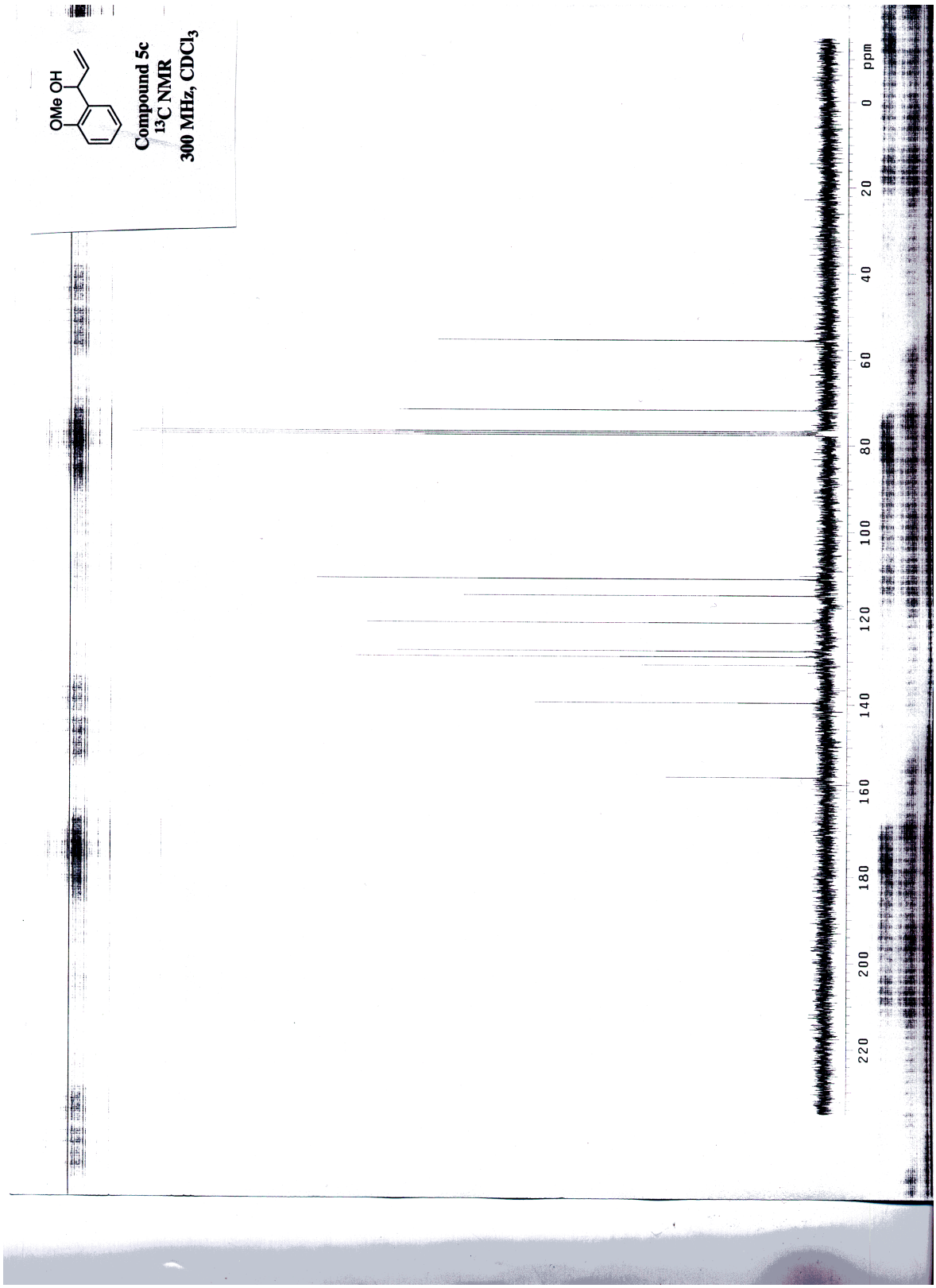
1.13
3.58
1.24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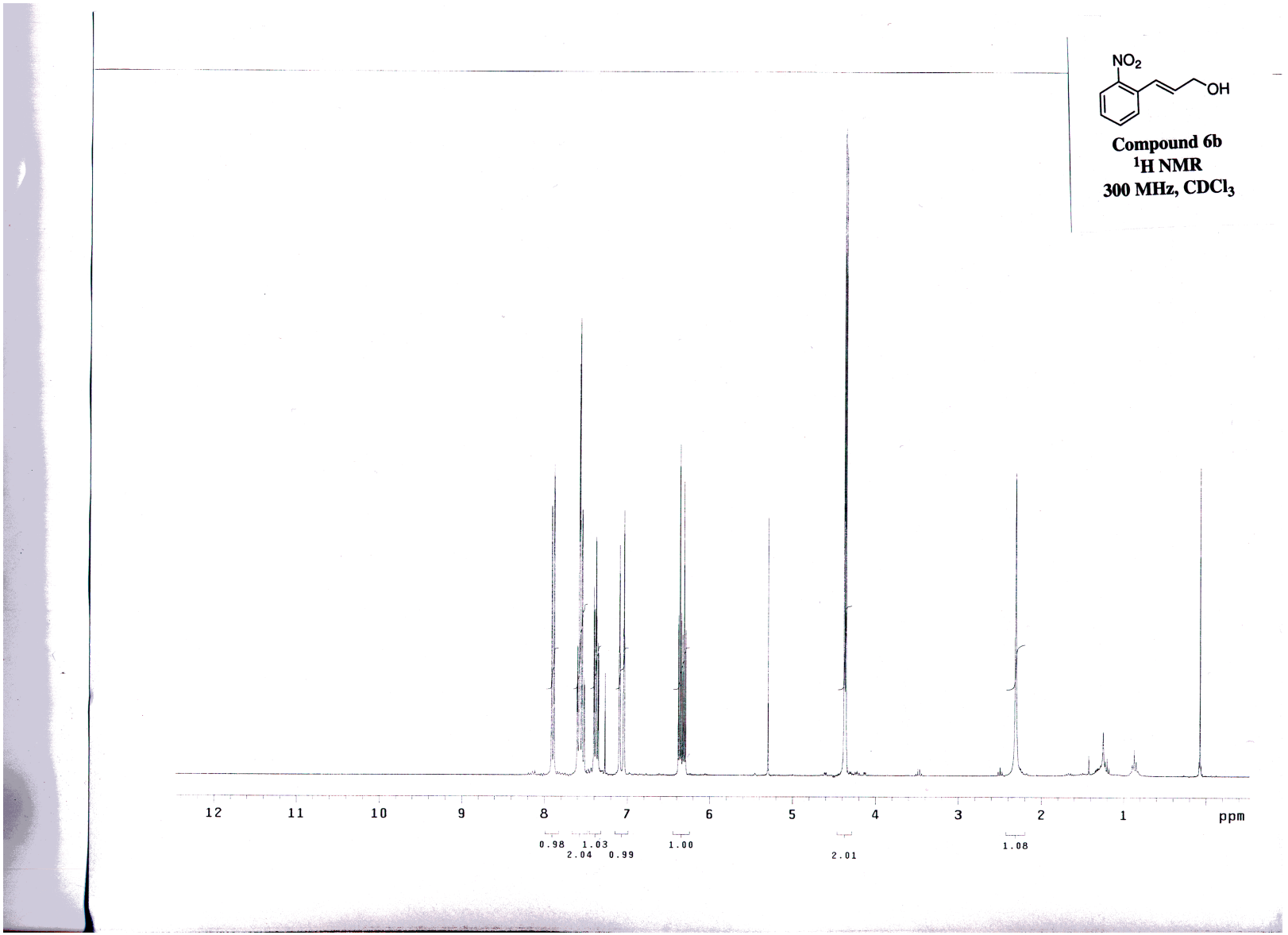
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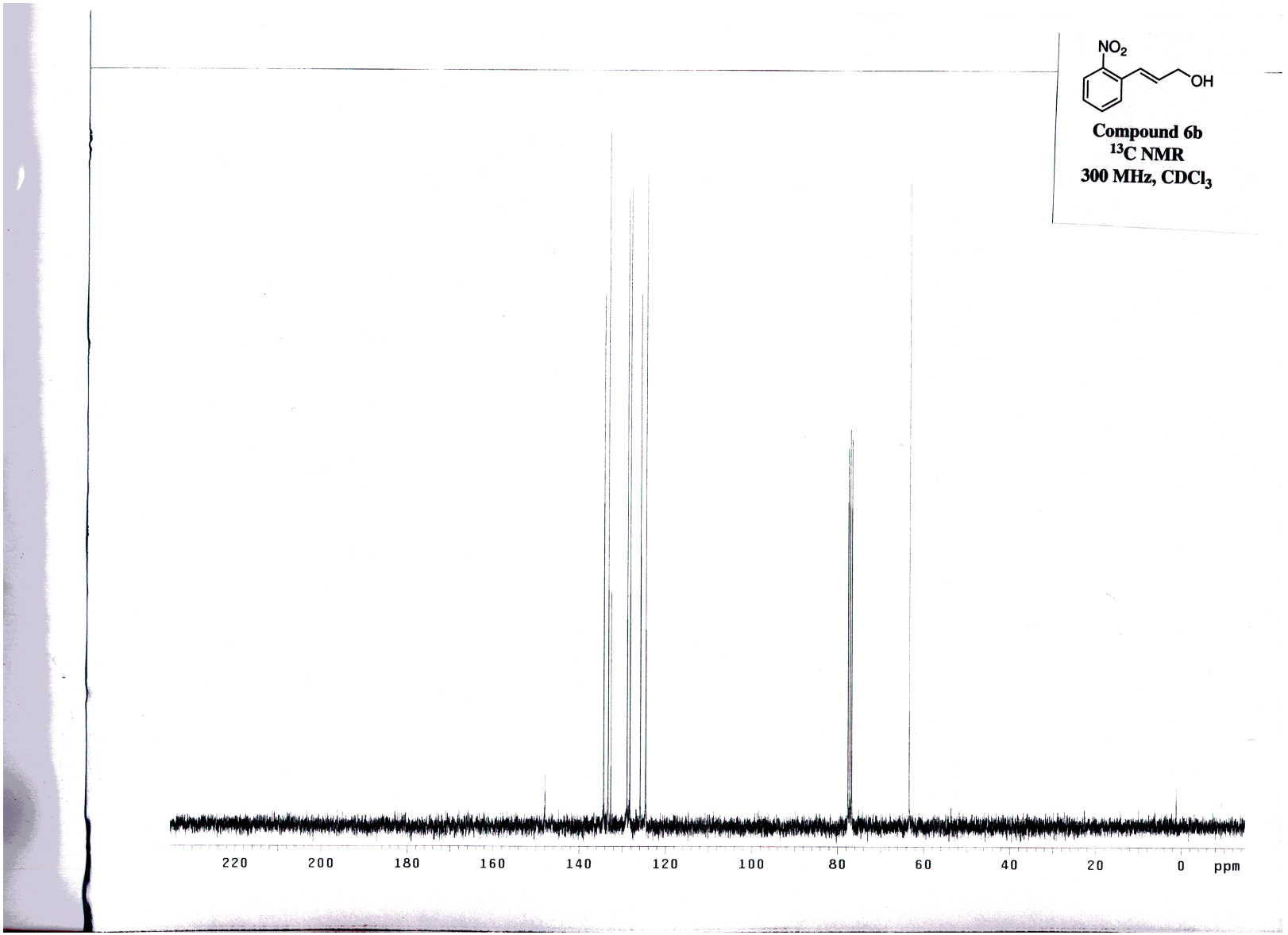
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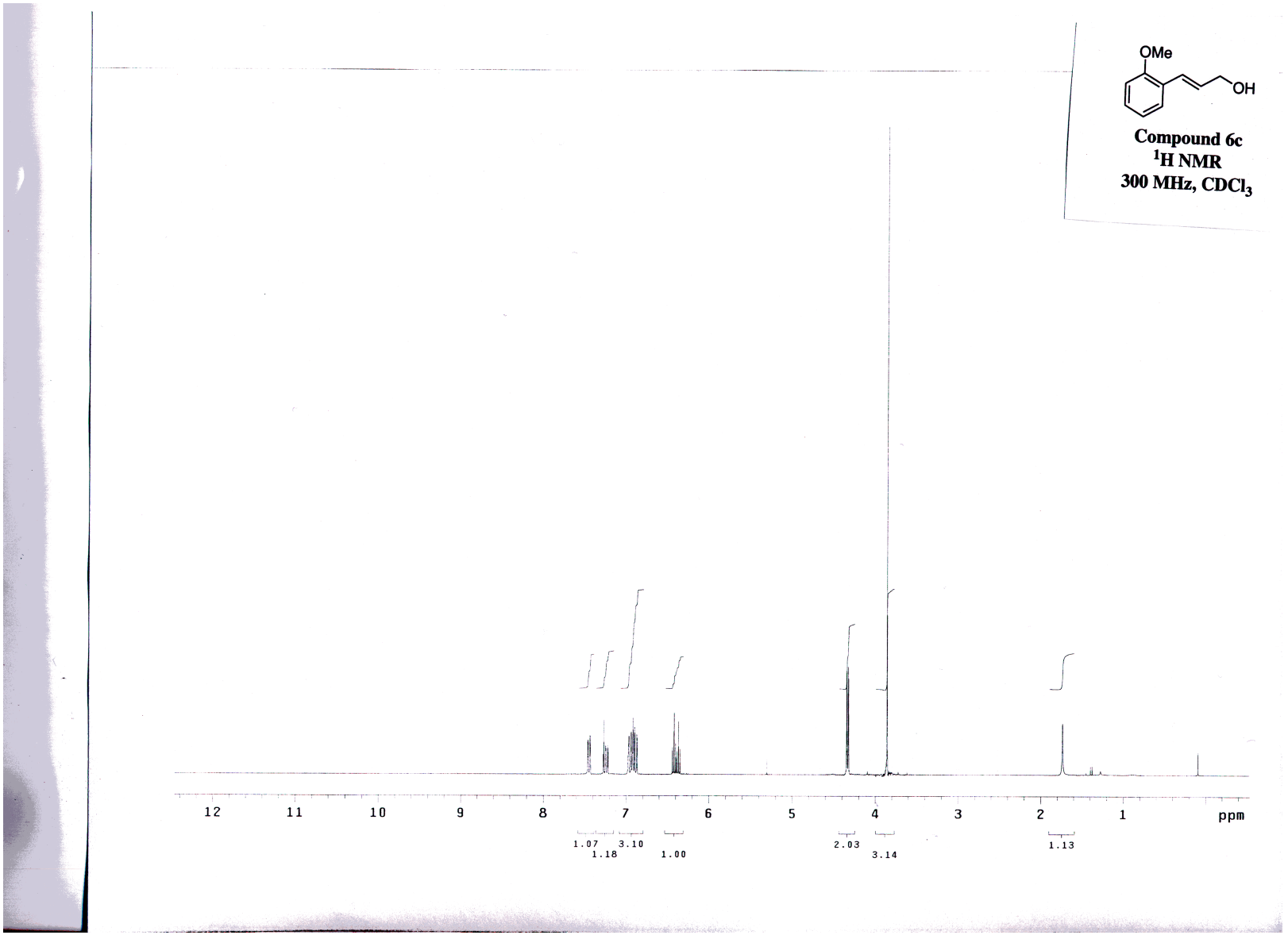
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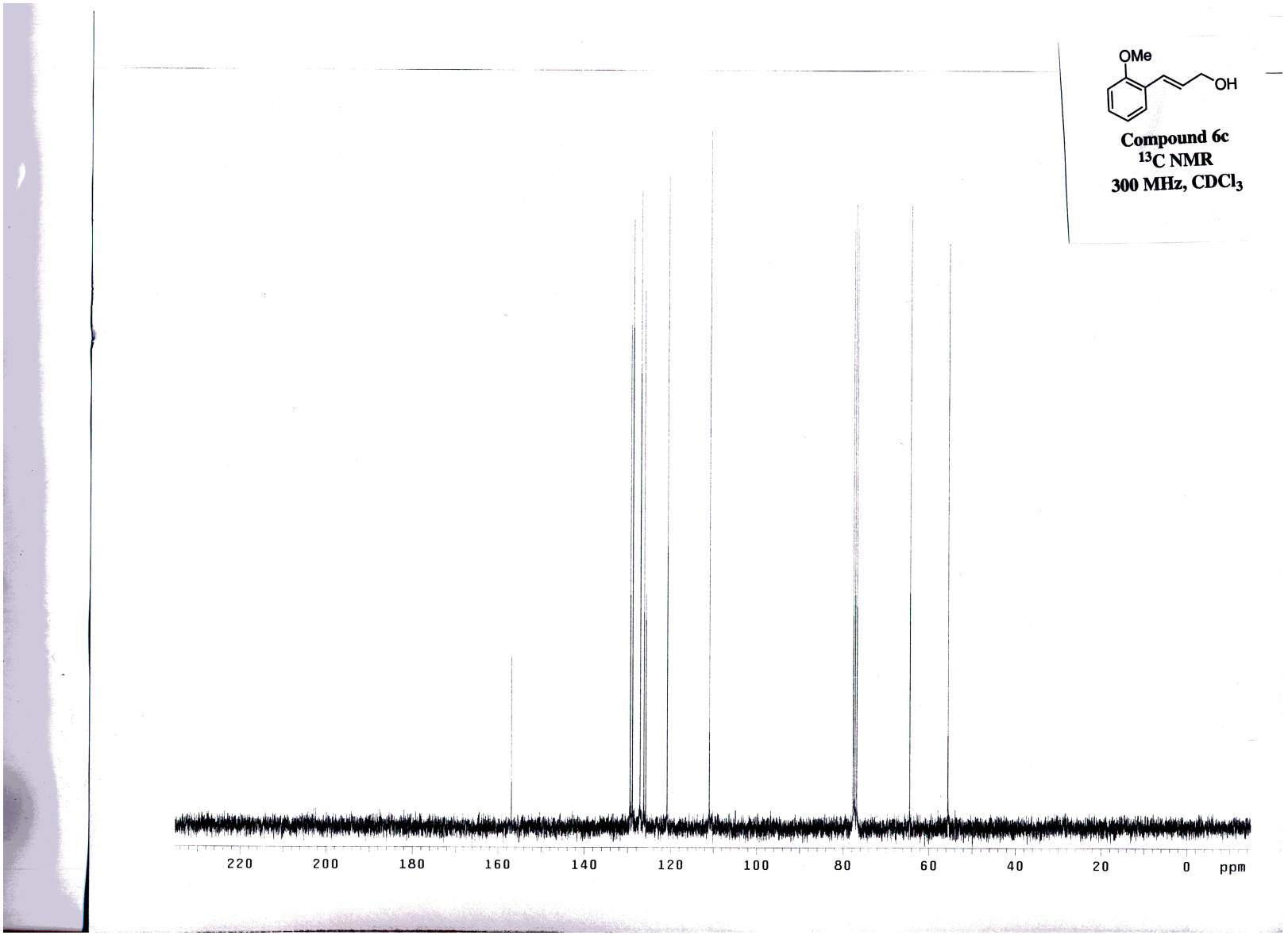
1.13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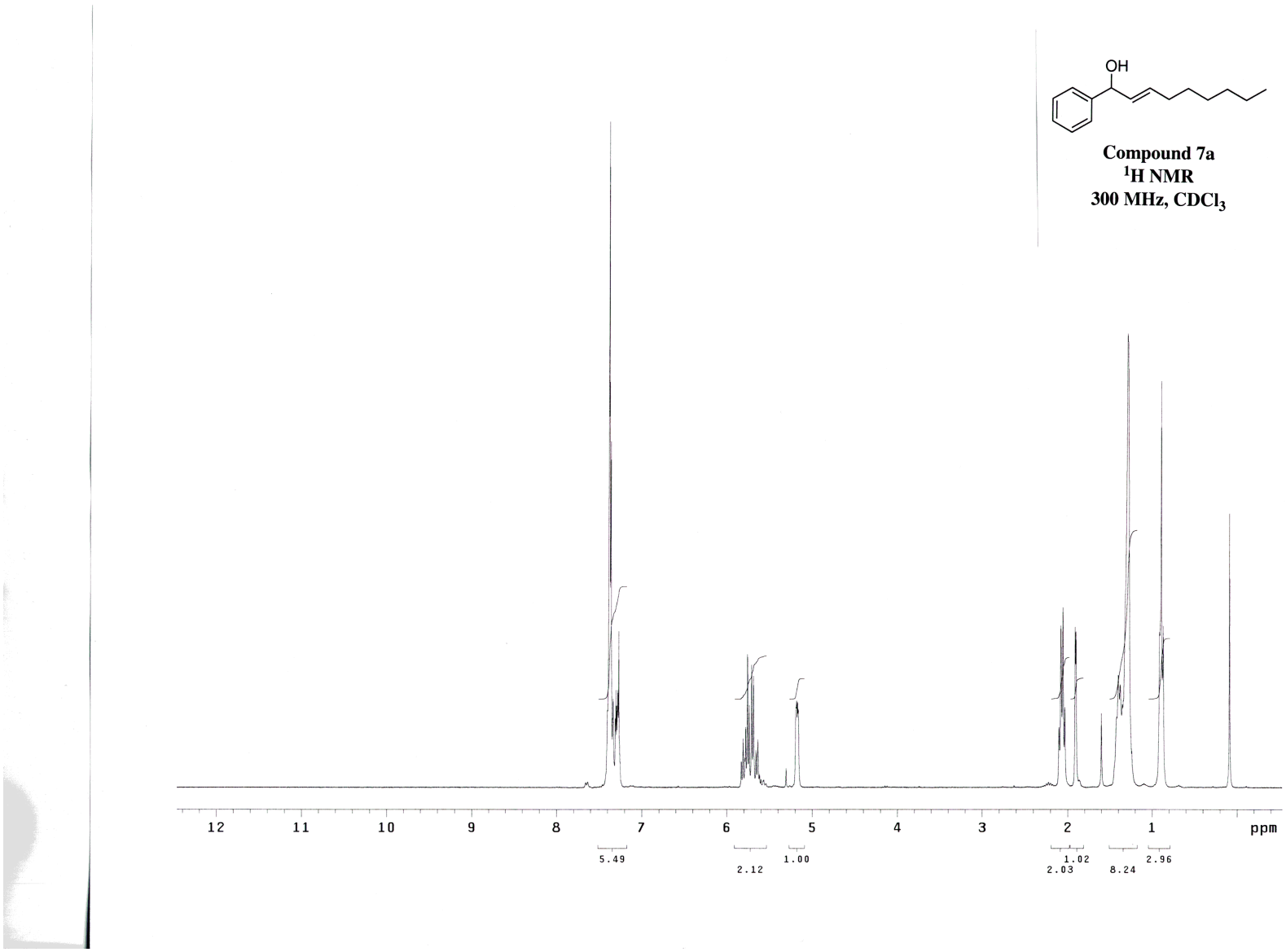
ppm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4
3
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ppm
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1.00
2.03
1.02
8.24
2.96

OH
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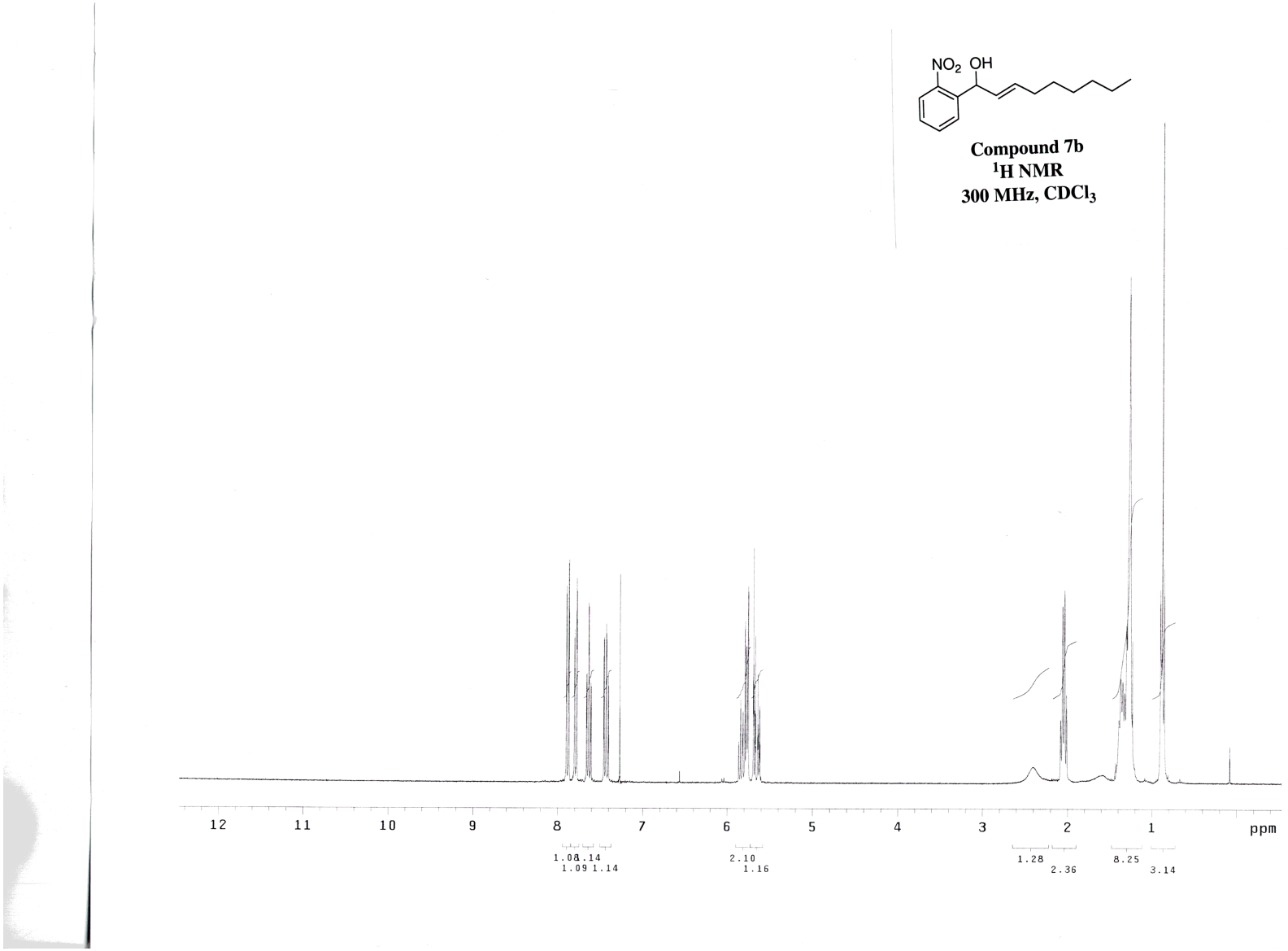
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ppm
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1.09
1.14
2.10
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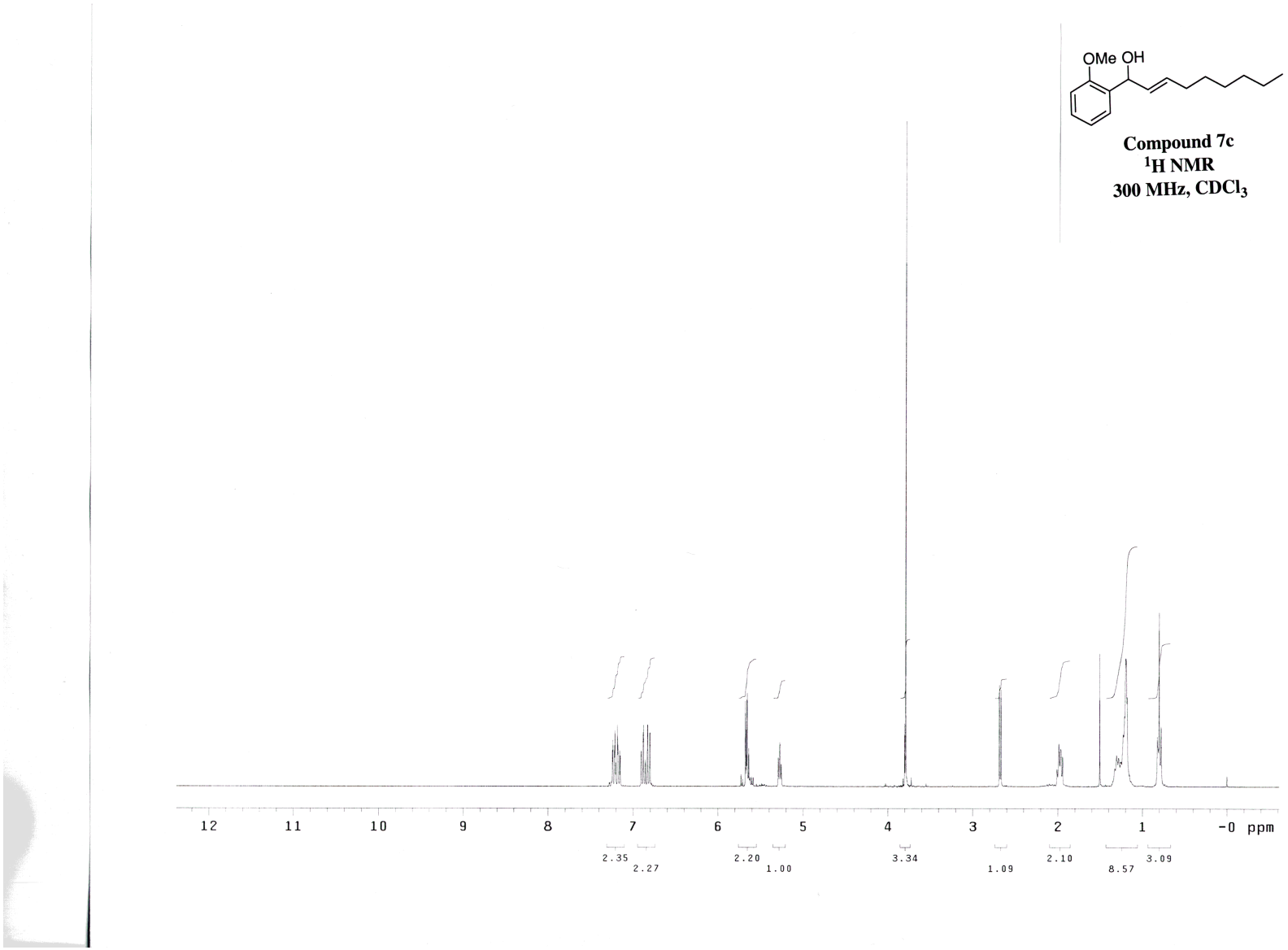
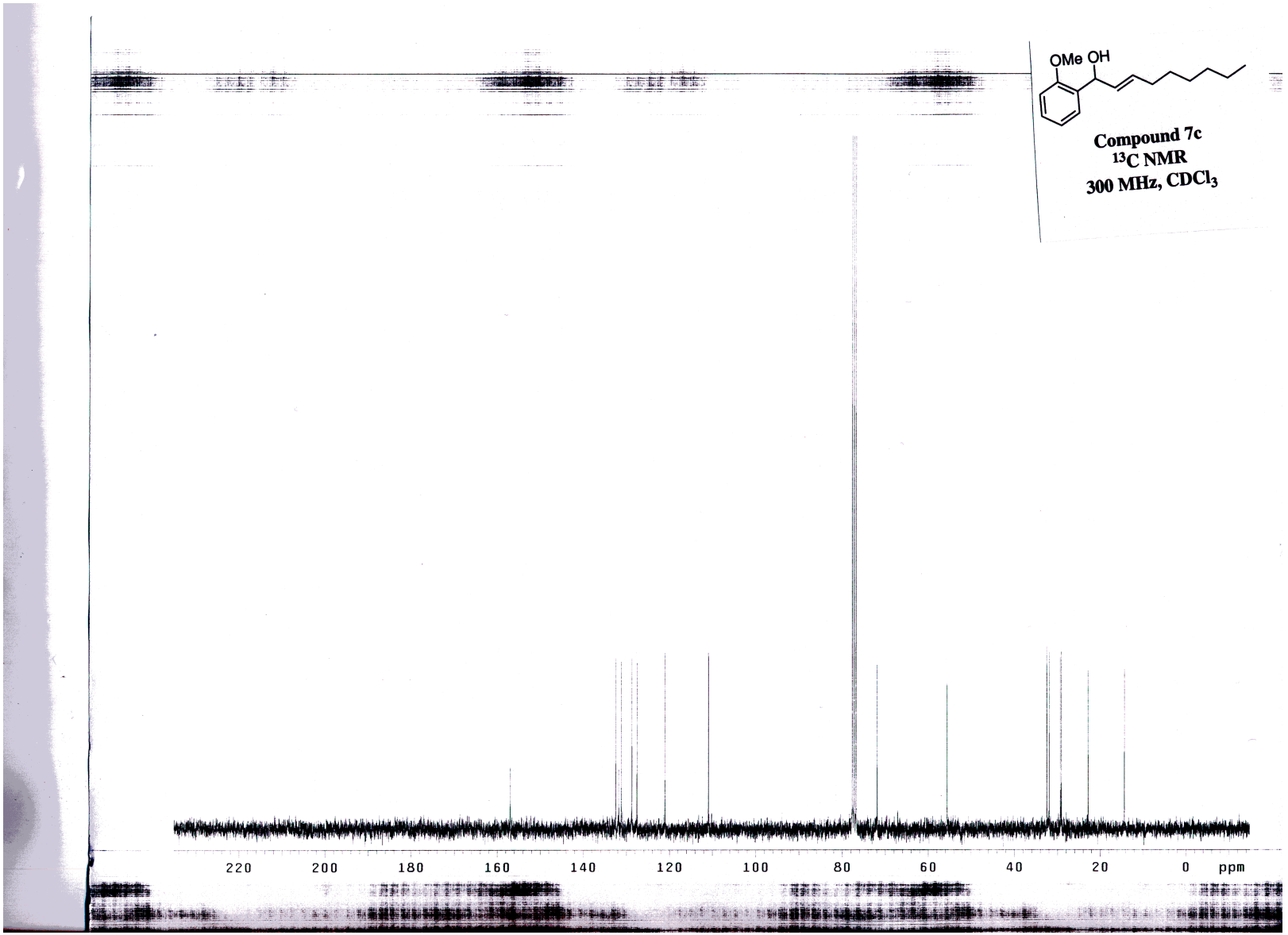
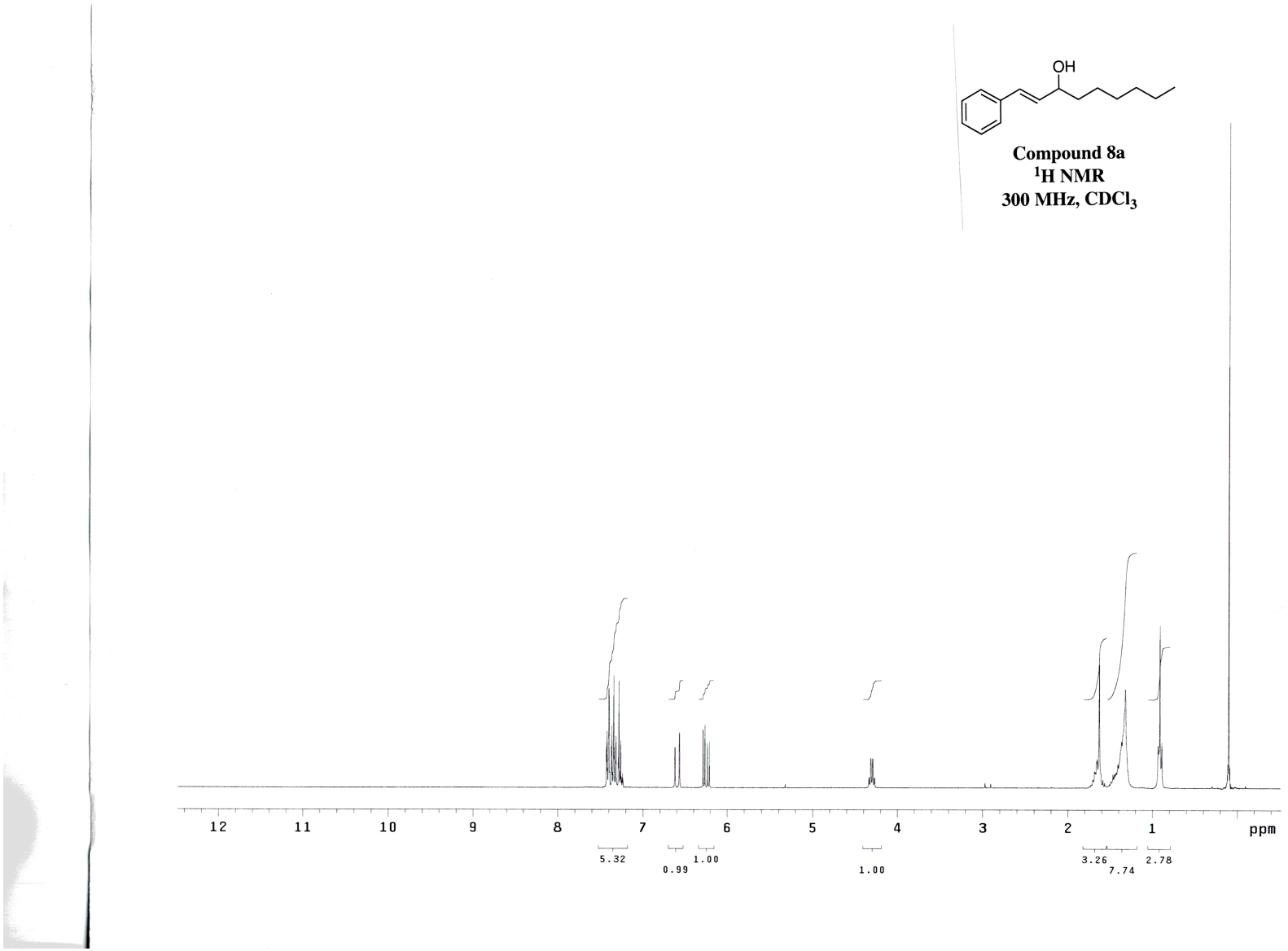
8.25
3.14

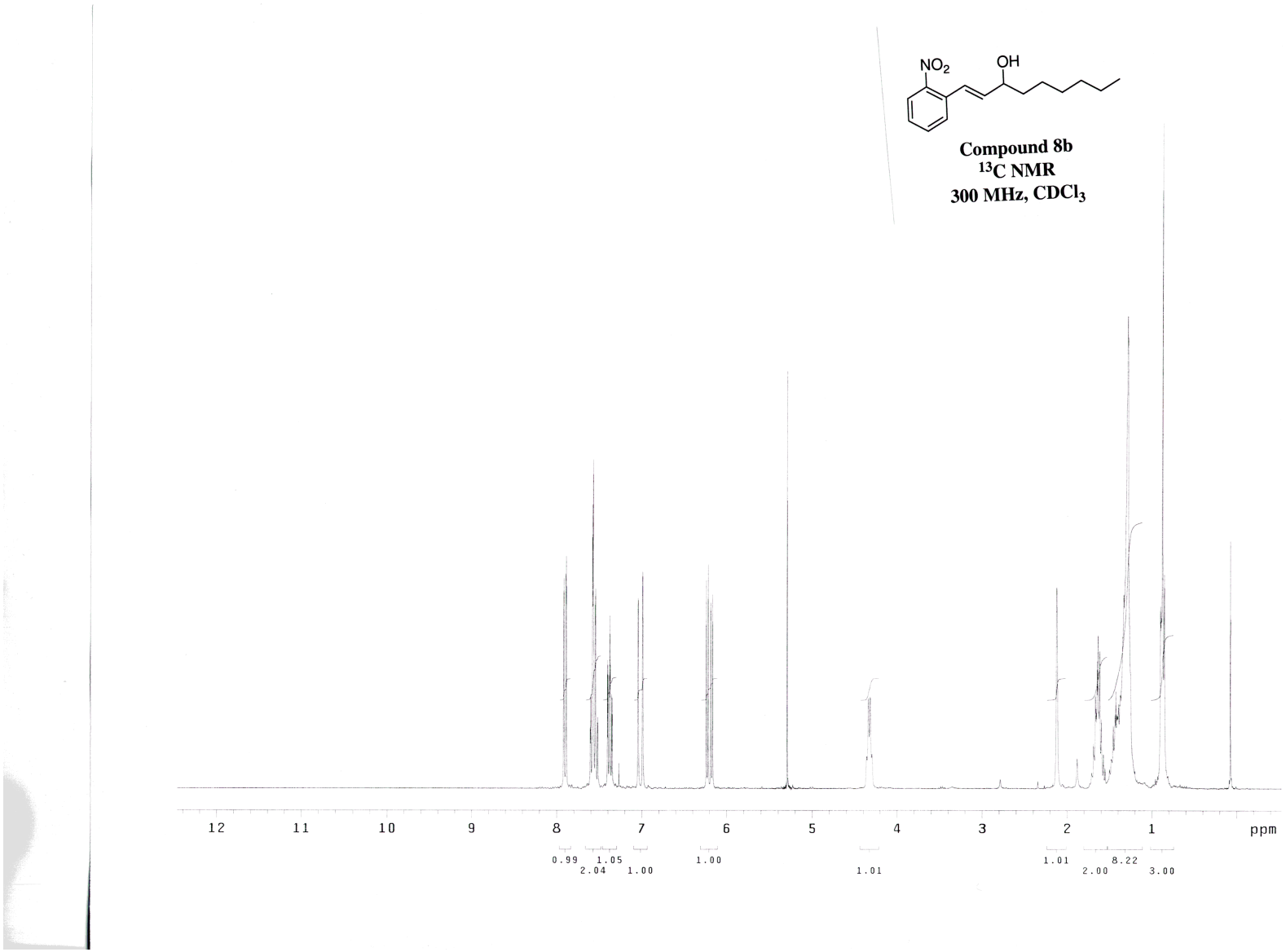
Compound 7b
13C NMR
300 MHz, CDCl3
NO2
OH
180
160
140
120
100
80
60
40
20
0
ppm

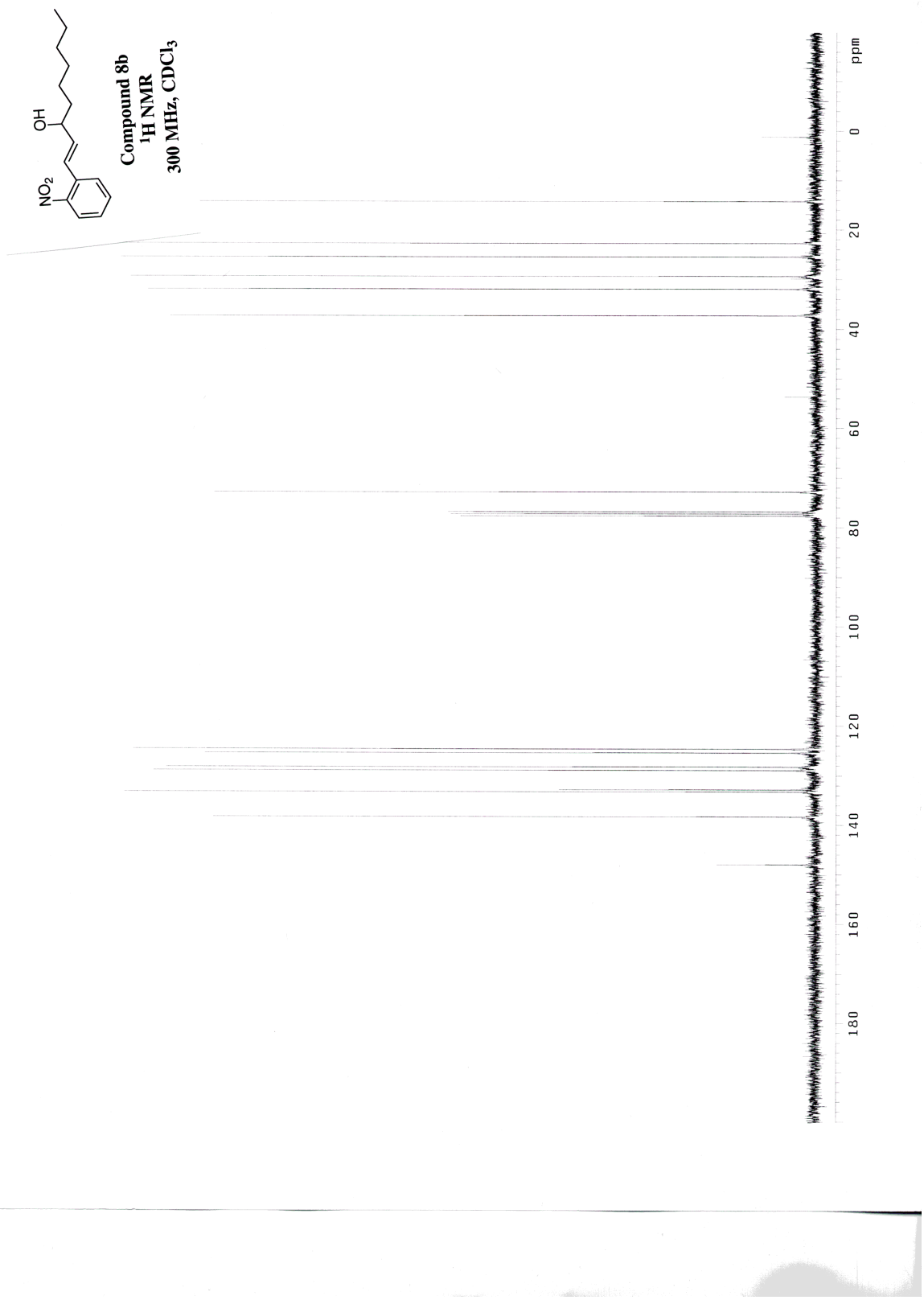

OMe OH
Compound 7c
1H NMR
300 MHz, CDCl3
12
11
10
9
8
7
6
5
4
3
2
1
-0 ppm
2.35
2.27
2.20
1.00
3.34
1.09
2.10
8.57
3.09

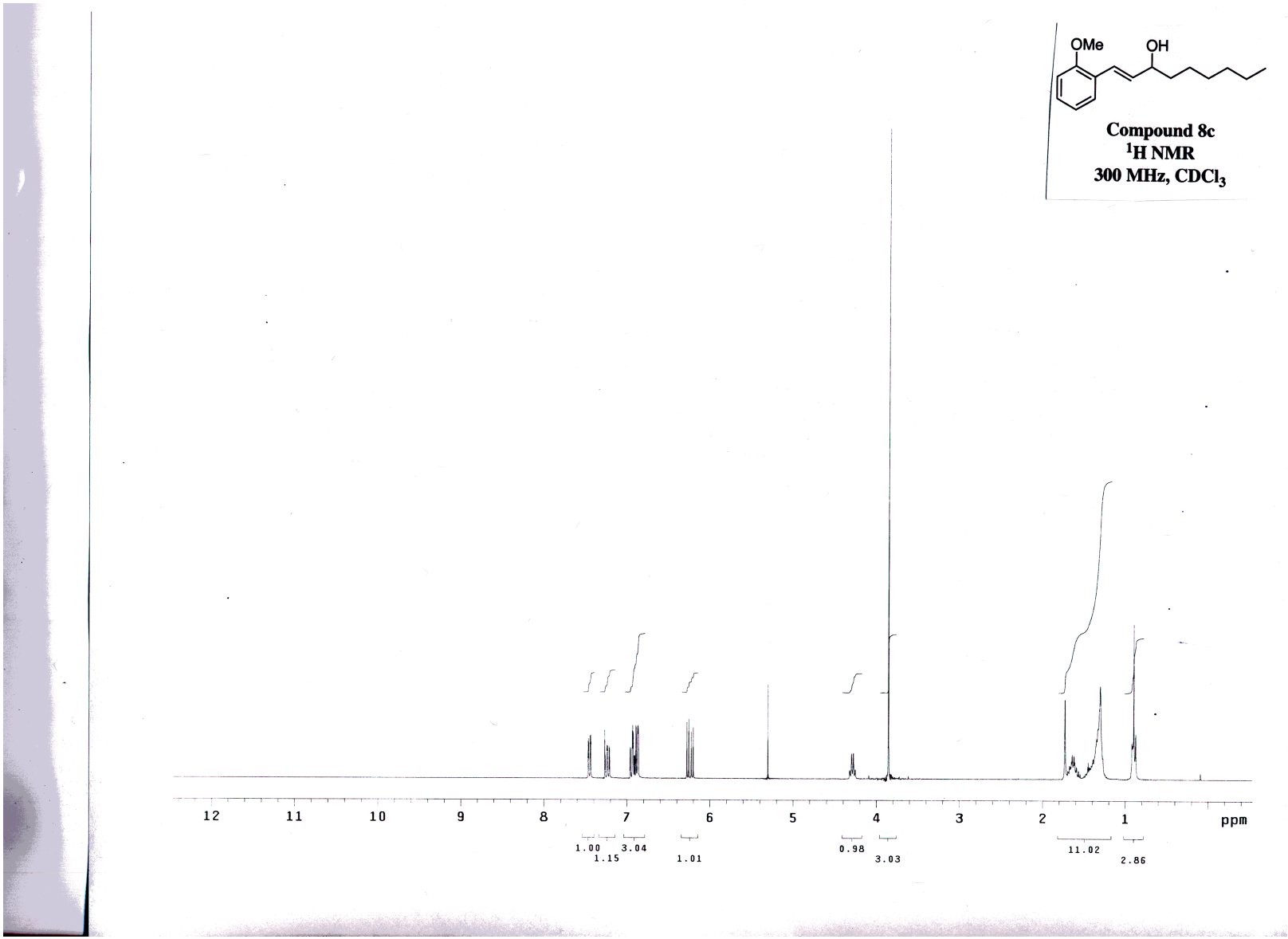

OMe OH
Compound 7c
13C NMR
300 MHz, CDCl3
220
200
180
160
140
120
100
80
60
40
20
0
ppm

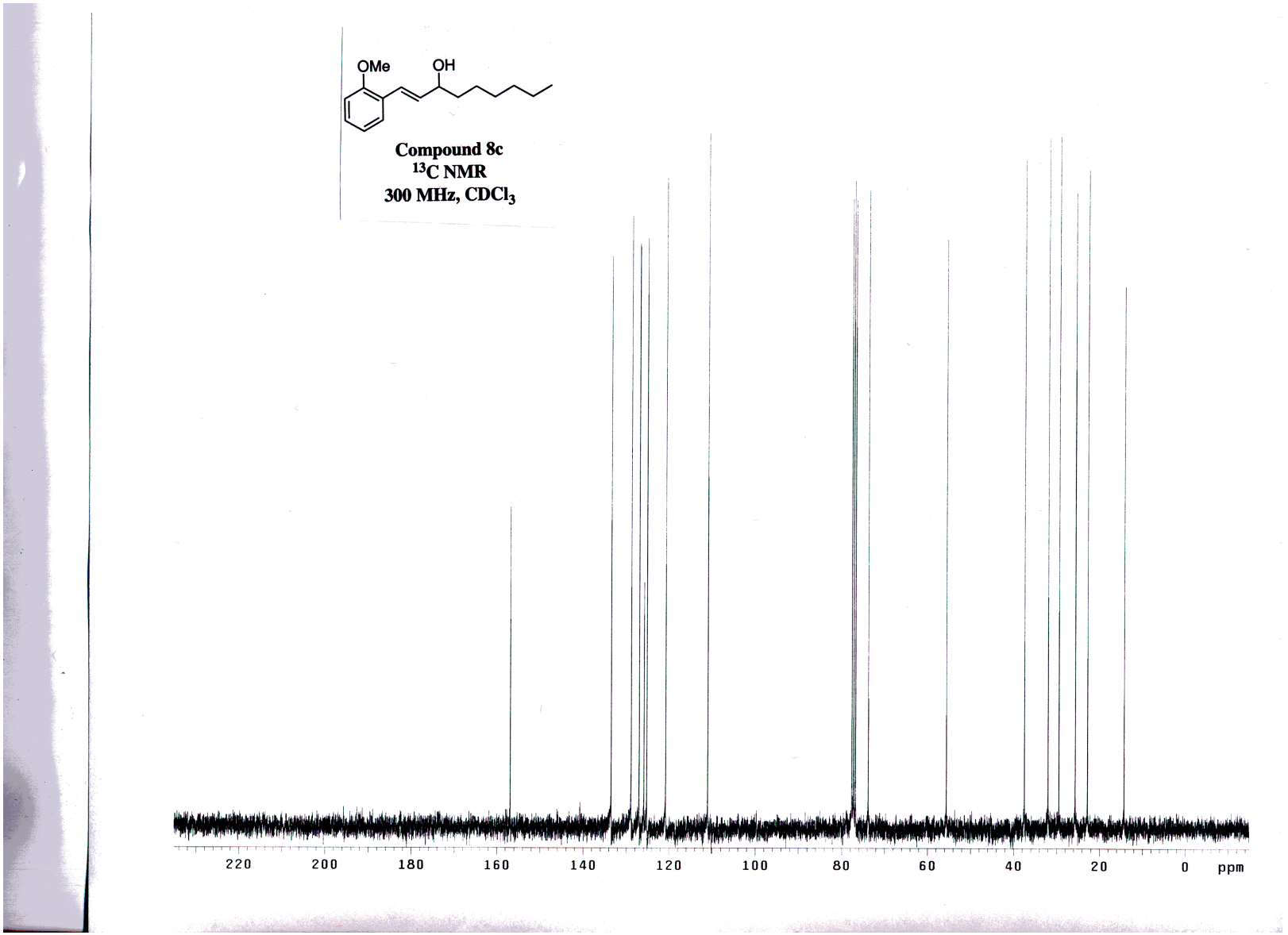

OH
Compound 8a
1H NMR
300 MHz, CDCl3
12
11
10
9
8
7
6
5
4
3
2
1
ppm
5.32
0.99
1.00
1.00
3.26
7.74
2.78

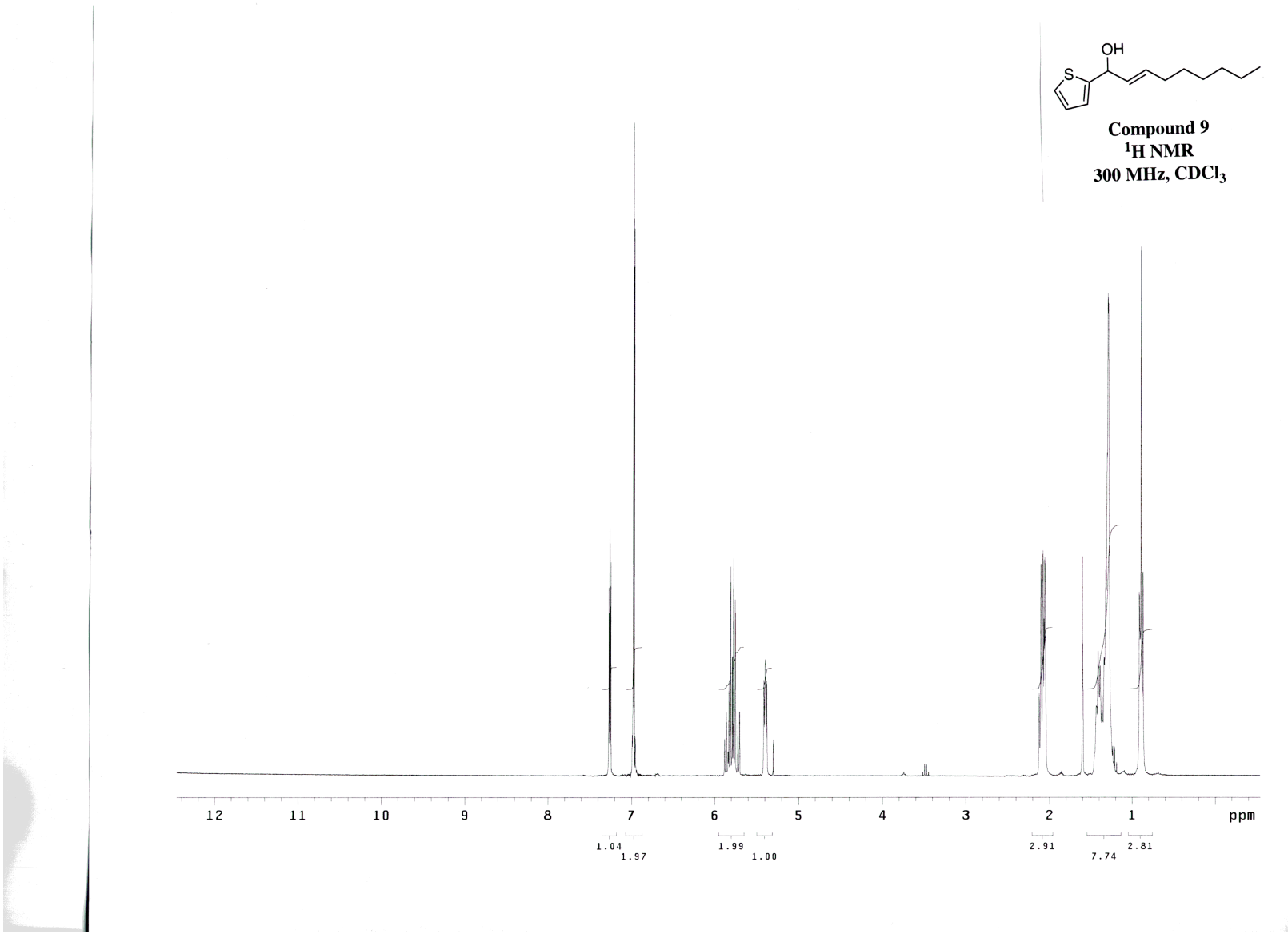
OH
Compound 8a
13C NMR
300 MHz, CDCl3
ppm
0
20
40
60
80
100
120
140
160
180
200
220


NO2
OH
Compound 8b
13C NMR
300 MHz, CDCl3
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.99
1.05
2.04
1.00
1.00
1.01
1.01
8.22
2.00
3.00


OH
NO2
Compound 8b
1H NMR
300 MHz, CDCl3
ppm
0
20
40
60
80
100
120
140
160
180


OMe
OH
Compound 8c
1H NMR
300 MHz, CDCl3
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.15
3.04
1.01
0.98
3.03
11.02
2.86


OMe
OH
Compound 8c
13C NMR
300 MHz, CDCl3
220
200
180
160
140
120
100
80
60
40
20
0
ppm


OH
S
Compound 9
1H NMR
300 MHz, CDCl3
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.04
1.97
1.99
1.00
2.91
7.74
2.81

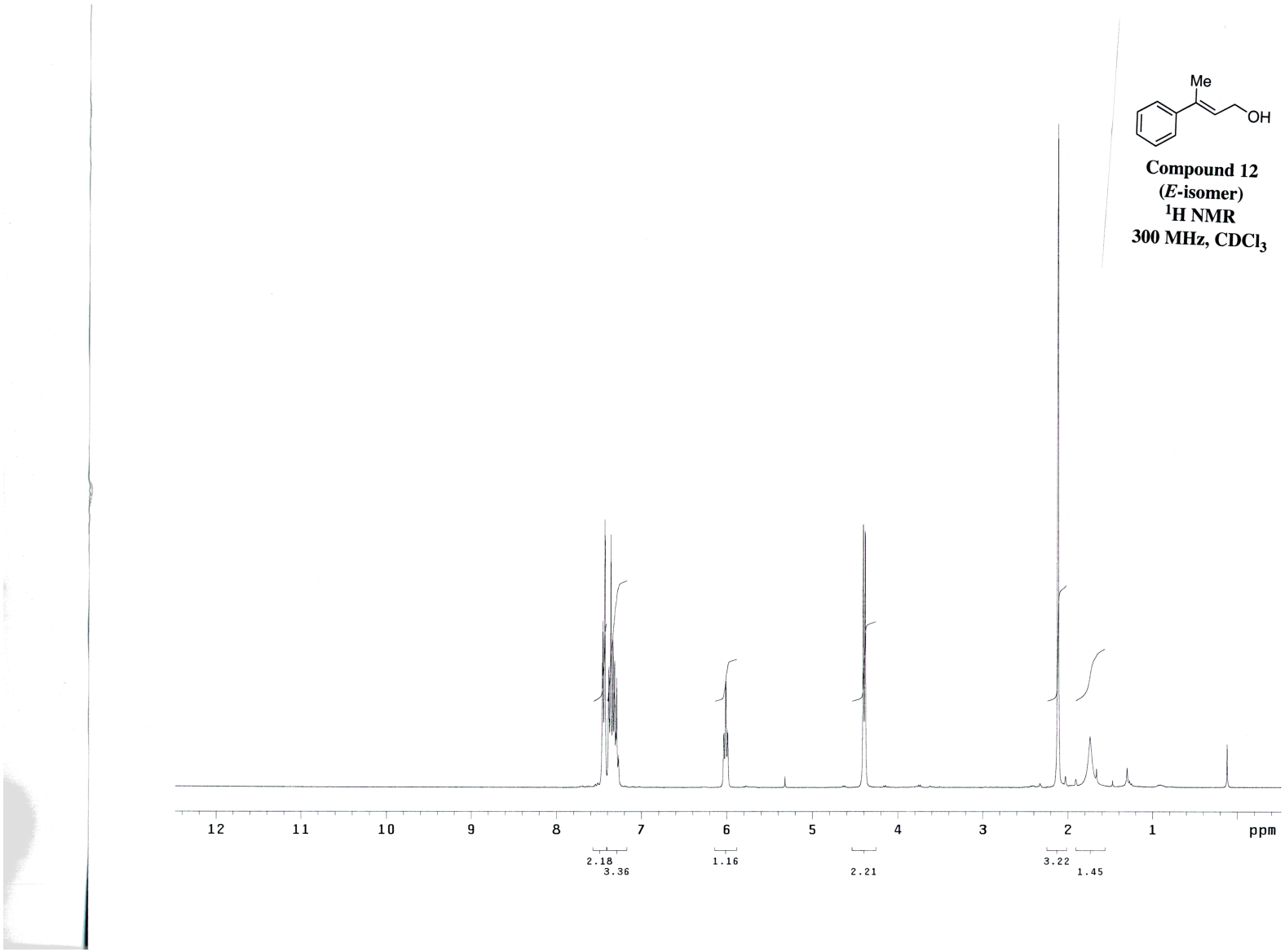
Compound 9
^{13}C NMR
300 MHz, $CDCl_3$

OH
S
Compound 10
^{1}H NMR
300 MHz, $CDCl_3$
ppm
1
2
3
4
5
6
7
8
9
10
11
12
2.91
1.99
1.16
8.07
1.02
1.00
0.96
1.03
2.02

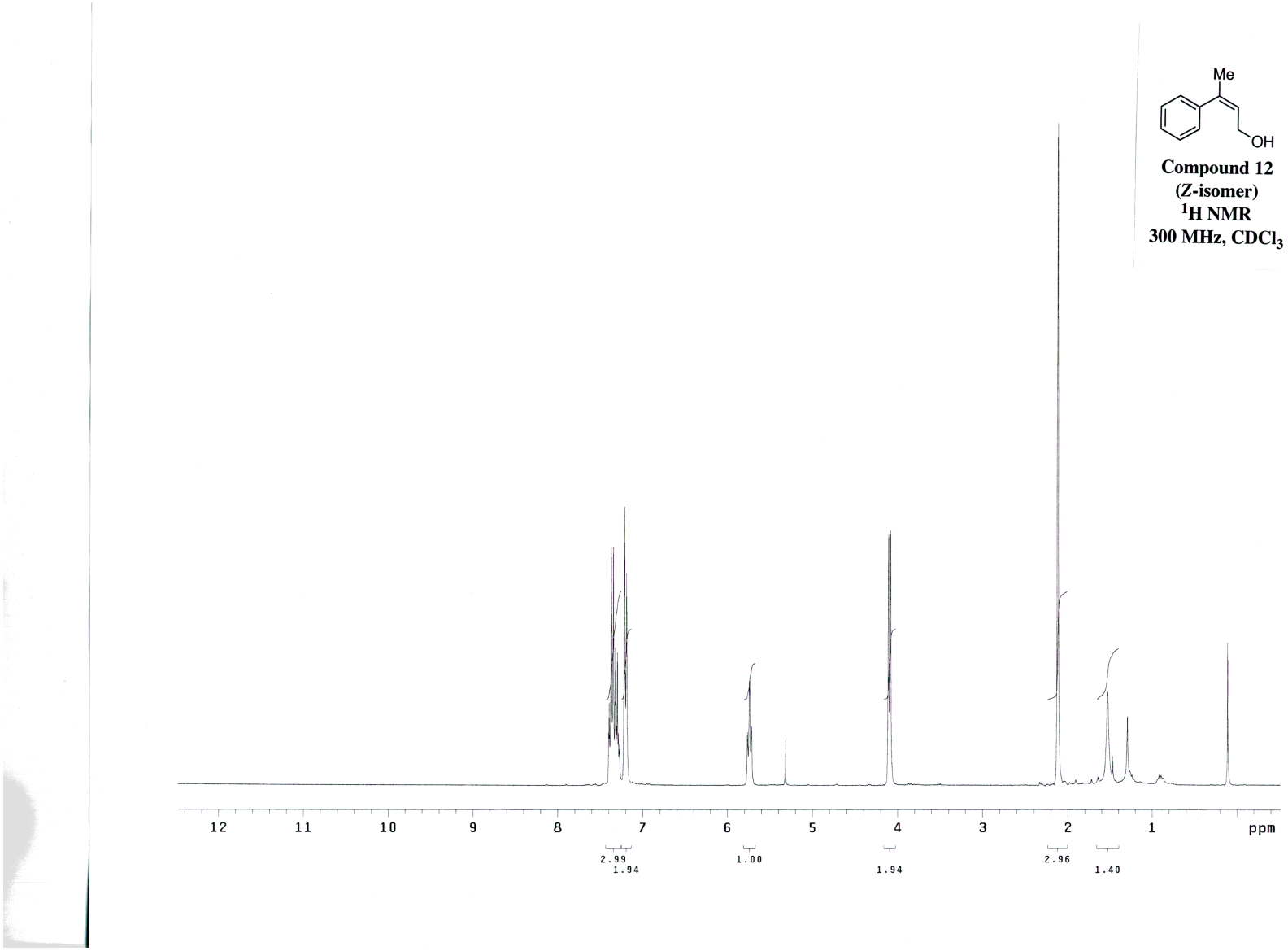
OH
S
Compound 10
13C NMR
300 MHz, CDCl3
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Compound 11
^{1}H NMR
300 MHz, $CDCl_3$

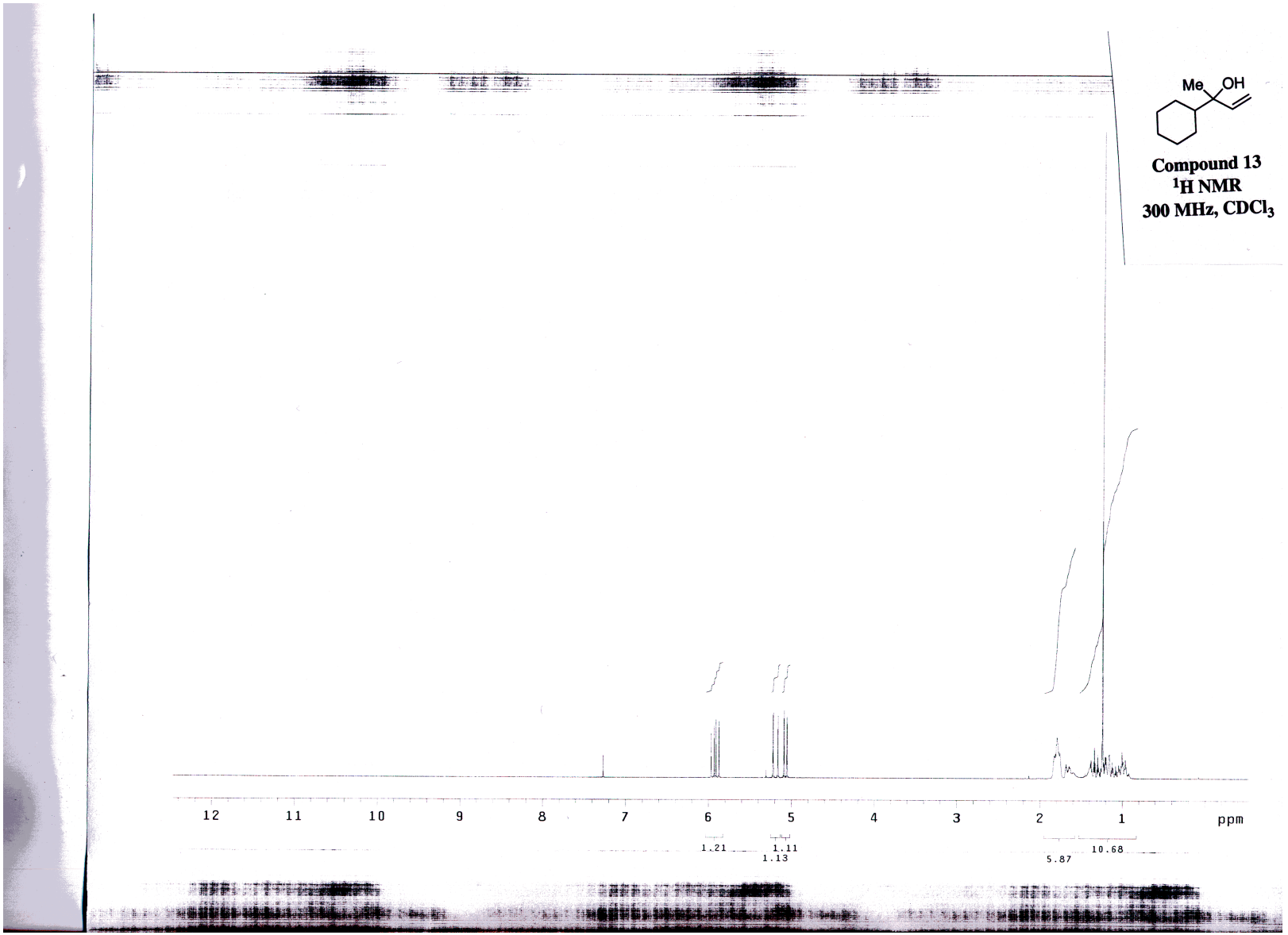
Compound 11
^{13}C NMR
300 MHz, $CDCl_3$


Me
OH
Compound 12
(E-isomer)
1H NMR
300 MHz, CDCl3
12
11
10
9
8
7
6
5
4
3
2
1
ppm
2.18
3.36
1.16
2.21
3.22
1.45

Compound 12
(*E*-isomer)
^{13}C NMR
300 MHz, $CDCl_3$

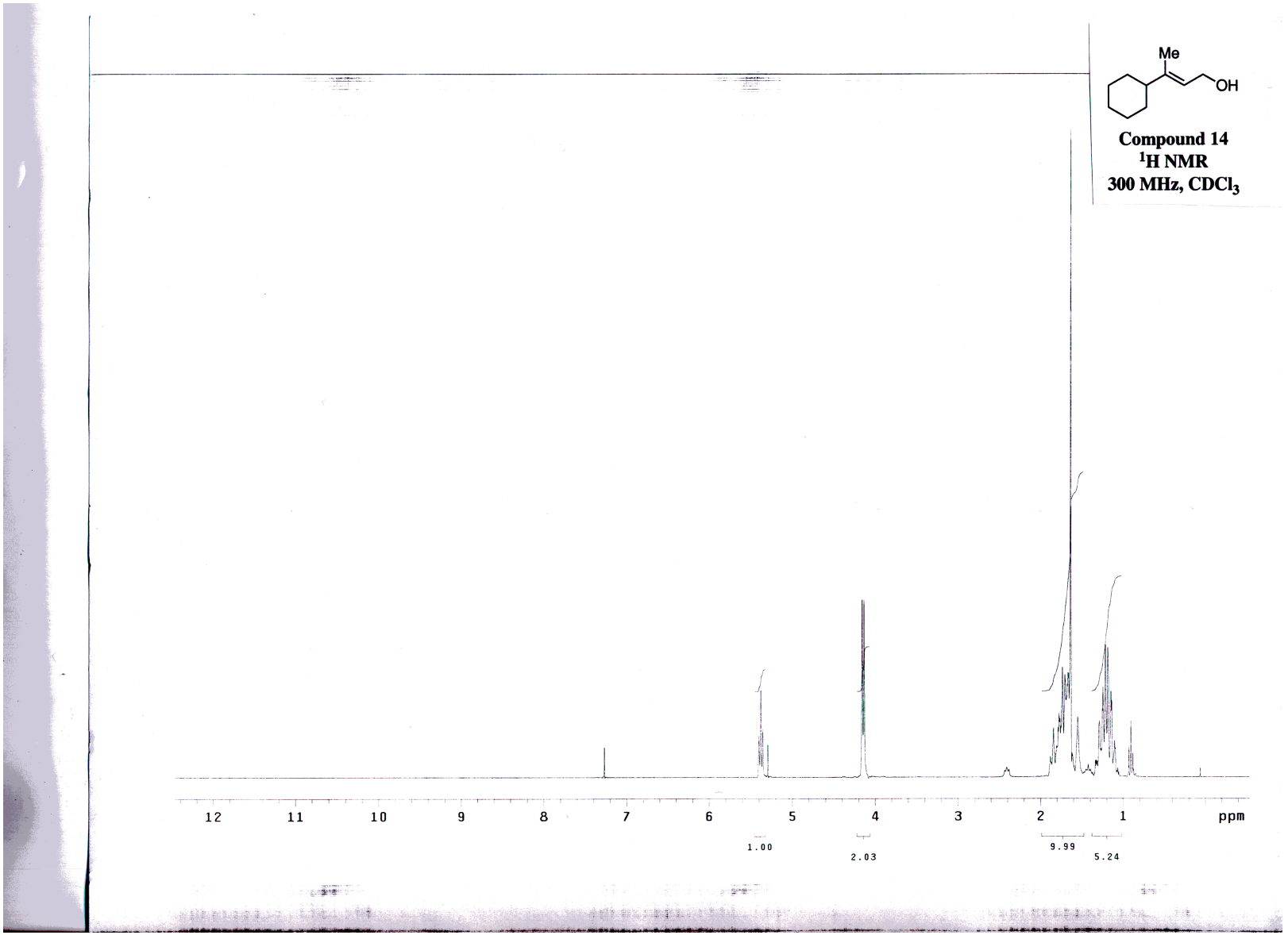

Me
OH
Compound 12
(Z-isomer)
1H NMR
300 MHz, CDCl3
12
11
10
9
8
7
6
5
4
3
2
1
ppm
2.99
1.94
1.00
1.94
2.96
1.40

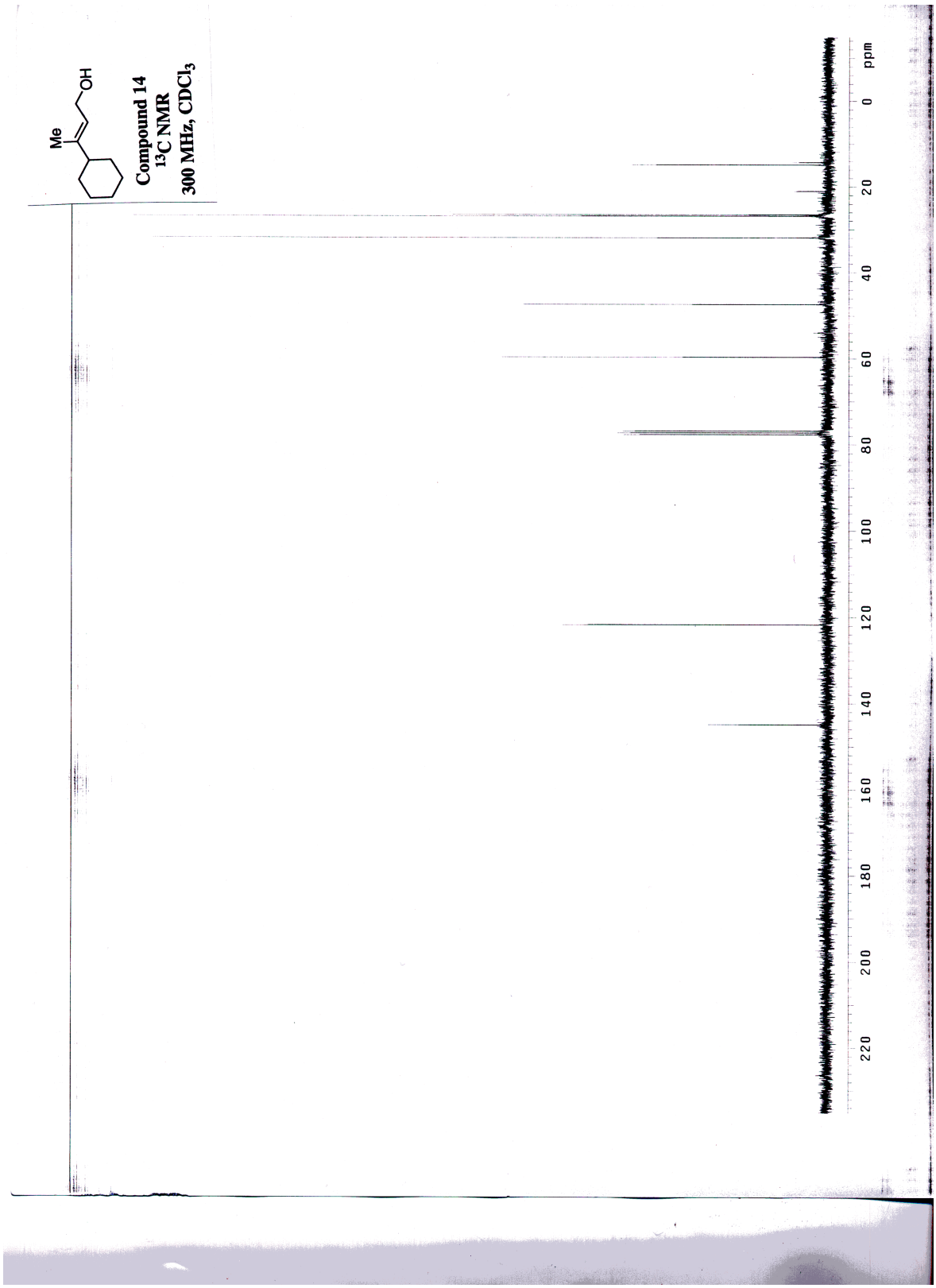
Compound 12
(Z-isomer)
^{13}C NMR
300 MHz, $CDCl_3$

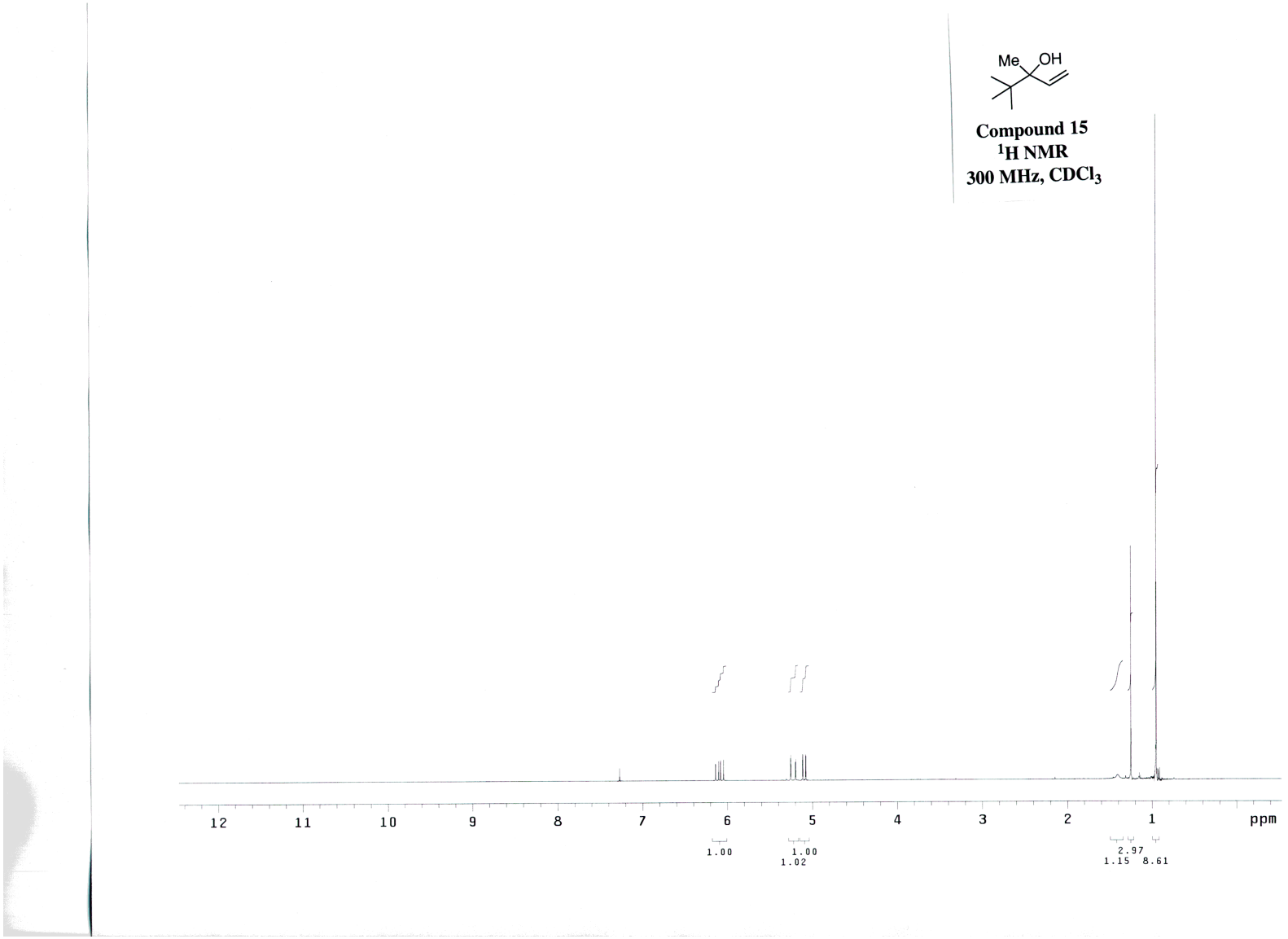


Compound 13
^{1}H NMR
300 MHz, $CDCl_3$

Me
OH
Compound 13
13C NMR
300 MHz, CDCl3
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Me
OH
Compound 14
1H NMR
300 MHz, CDCl3
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.03
9.99
5.24

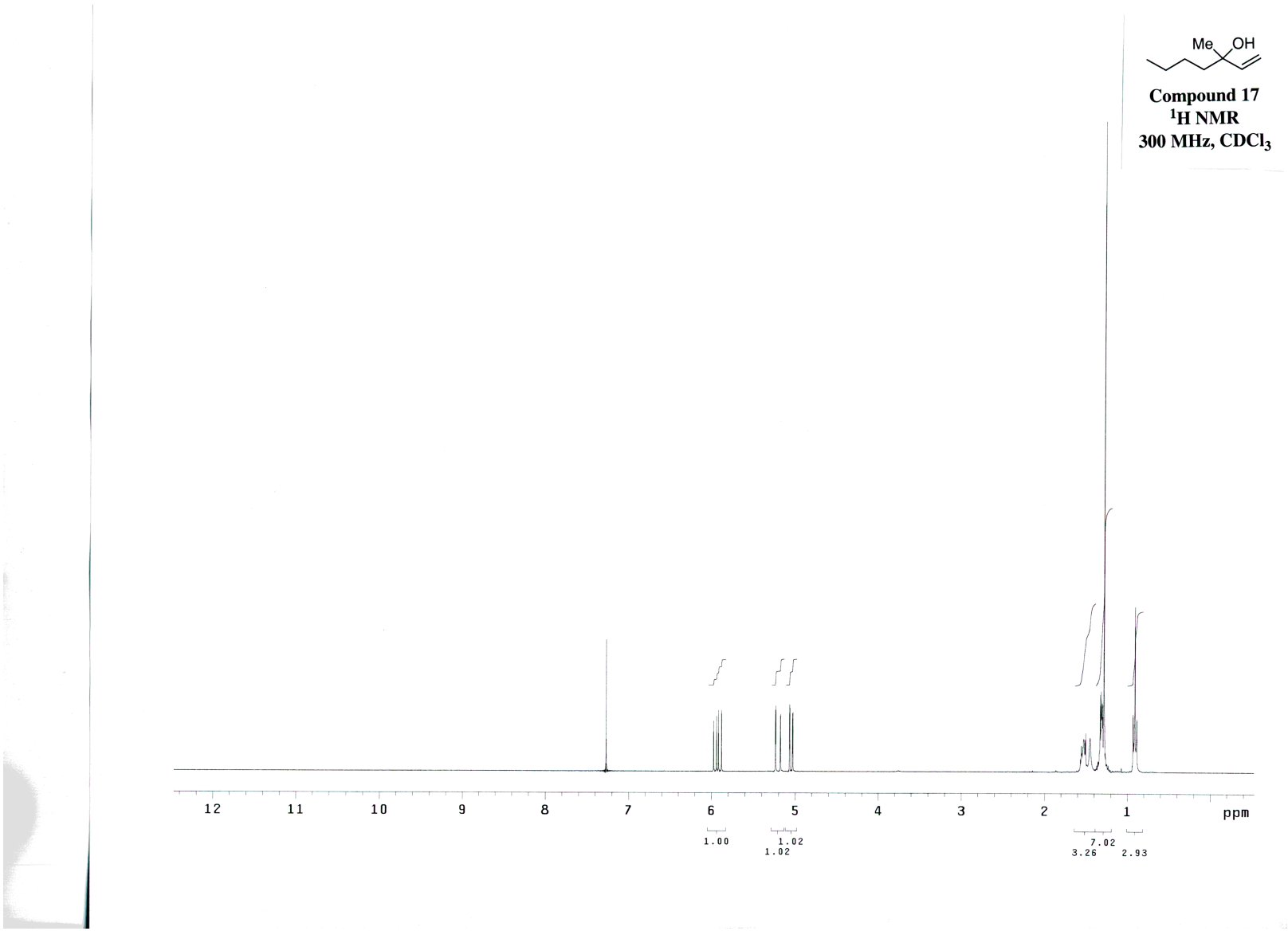

Me
OH
Compound 14
^{13}C NMR
300 MHz, $CDCl_3$
ppm
0
20
40
60
80
100
120
140
160
180
200
220


Me
OH
Compound 15
1H NMR
300 MHz, CDCl3
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.00
1.02
2.97
1.15
8.61

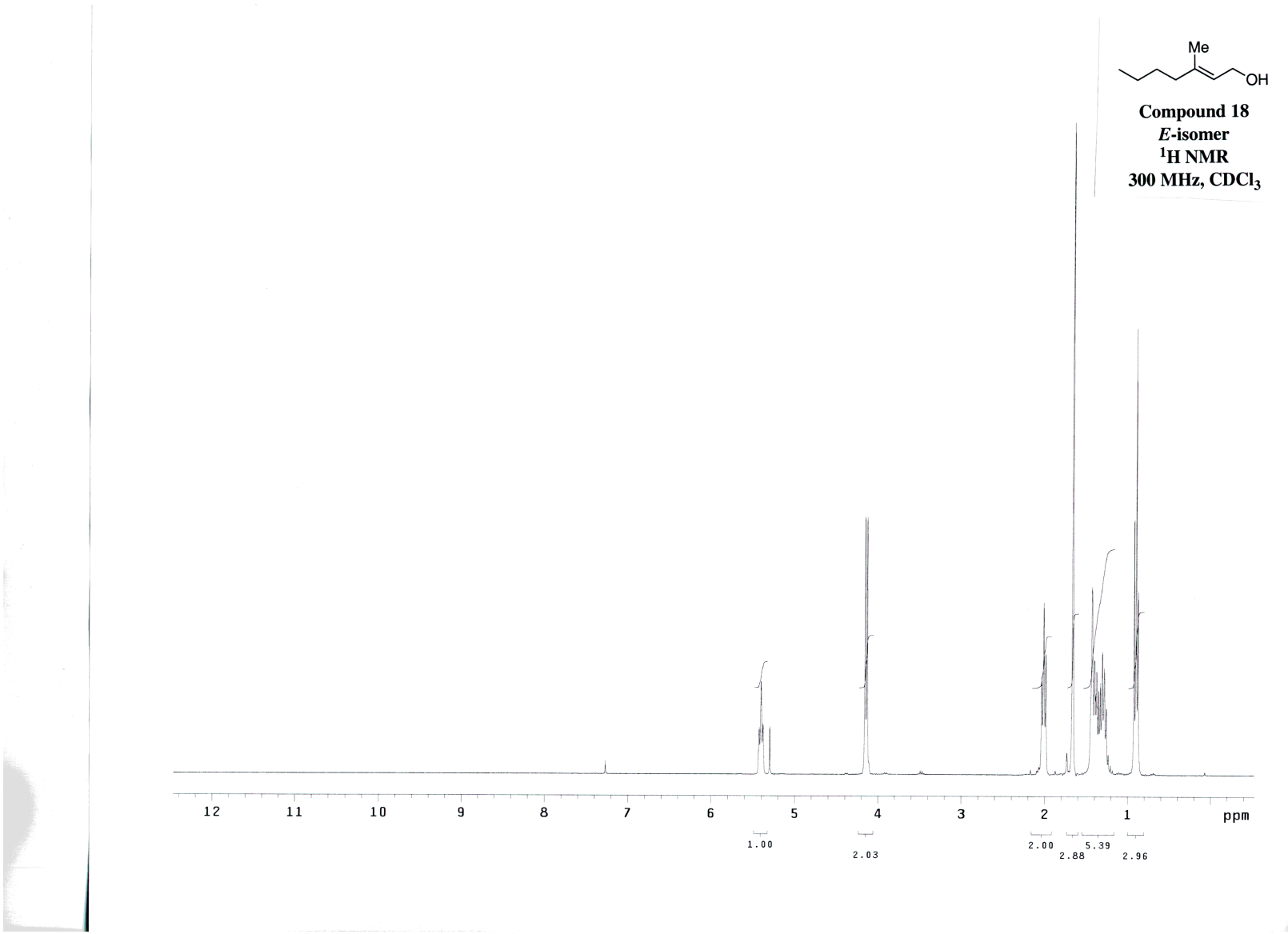
Compound 15
^{13}C NMR
300 MHz, $CDCl_3$

Compound 16
^{1}H NMR
300 MHz, $CDCl_3$

Compound 16
^{13}C NMR
300 MHz, $CDCl_3$


Me
OH
Compound 17
1H NMR
300 MHz, CDCl3
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.02
1.02
7.02
3.26
2.93

Compound 17
^{13}C NMR
300 MHz, $CDCl_3$

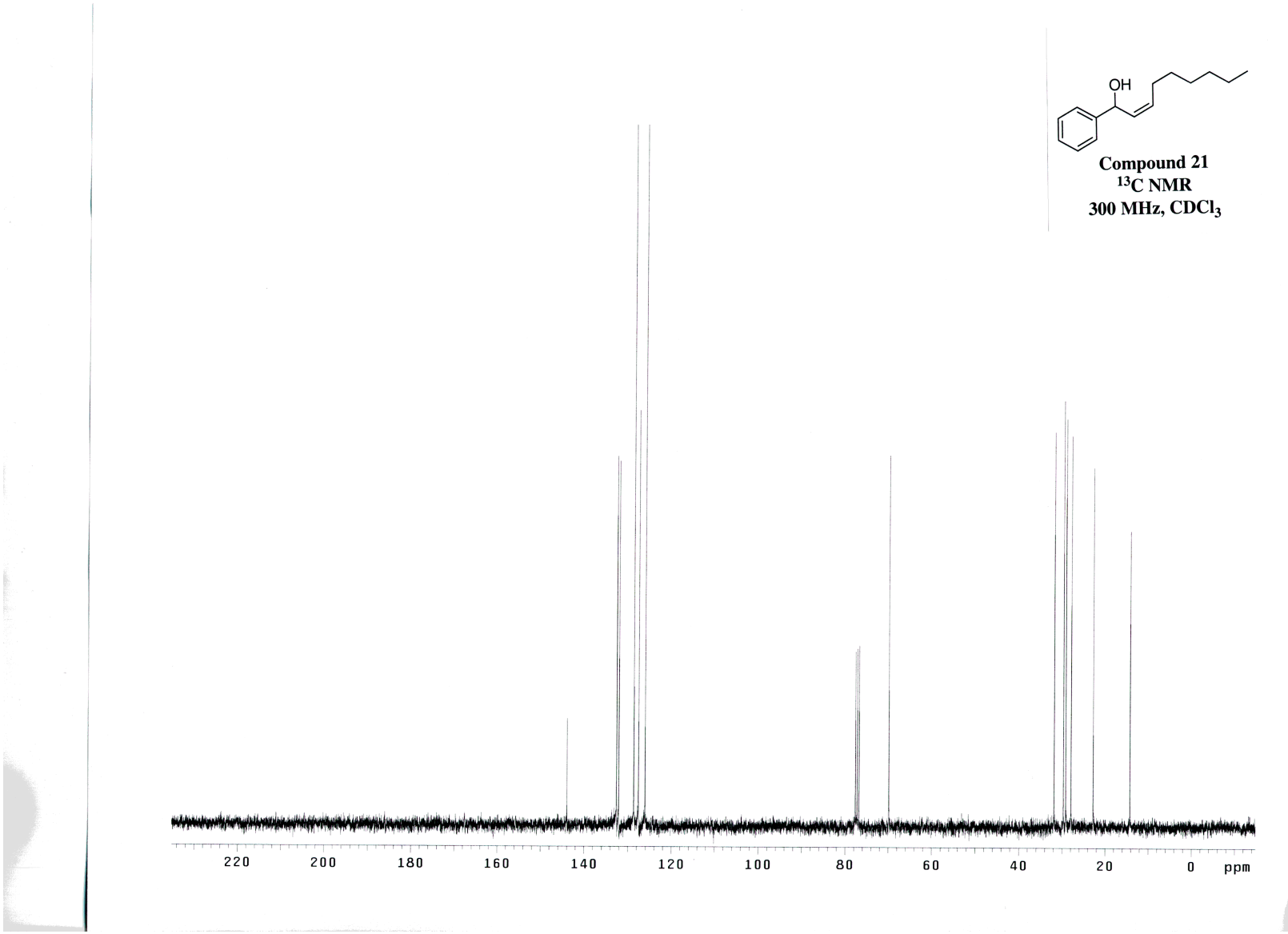

Me
OH
Compound 18
E-isomer
1H NMR
300 MHz, CDCl3
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.03
2.00
2.88
5.39
2.96

Me
OH
Compound 18
E-isomer
13C NMR
300 MHz, CDCl3
180
160
140
120
100
80
60
40
20
0
ppm

Me
OH
Compound 18
Z-isomer
1H NMR
300 MHz, CDCl3
ppm
1.00
2.03
2.04
3.09
7.35
4.05

Compound 18
Z-isomer
^{13}C NMR
300 MHz, $CDCl_3$

Compound 21
^{1}H NMR
300 MHz, $CDCl_3$



Compound 21
^{13}C NMR
300 MHz, $CDCl_3$