Supporting Information

$Pd(OAc)_2/P(^cC_6H_{11})_3$-Catalyzed Allylation of Aryl Halides with Homoallyl Alcohols via Retro-Allylation

Masayuki Iwasaki, Sayuri Hayashi, Koji Hirano, Hideki Yorimitsu,* and Koichiro Oshima*

Department of Material Chemistry, Graduate School of Engineering, Kyoto University, Kyoto-daigaku Katsura, Nishikyo-ku, Kyoto 615-8510, Japan

Contents

Instrumentation

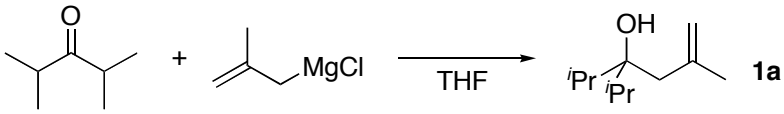
^{1}H NMR (300 MHz and 500 MHz) and ^{13}C NMR (75.3 MHz and 125.7 MHz) spectra were taken on Varian Mercury 300 and UNITY INOVA 500 spectrometers and were recorded in $CDCl_3$. Chemical shifts (δ) are in parts per million relative to tetramethylsilane at 0.00 ppm for ^{1}H and relative to $CDCl_3$ at 77.0 ppm for ^{13}C unless otherwise noted. IR spectra were determined on a SHIMADZU FTIR-8200PC spectrometer. Mass spectra (EI unless otherwise noted) were determined on a JEOL Mstation 700 spectrometer. TLC analyses were performed on commercial glass plates bearing 0.25-mm layer of Merck Silica gel $60F_{254}$. Silica gel (Wakogel 200 mesh) was used for column chromatography. Elemental analyses were carried out at the Elemental Analysis Center of Kyoto University.

Chemicals

Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. Toluene and xylene were purchased from Wako Pure Chemical Co. and stored over slices of sodium. Tri(*p*-tolyl)phosphine, triphenylphosphine, and cesium carbonate were purchased from Wako Pure Chemical Co. Palladium acetate and tricyclohexylphosphine were from TCI. Grubbs Catalyst, 2nd Generation, (benzylidene[1,3-bis(2,4,6-trimethylphenyl)-2-imidazolidinylidene]dichloro(tricyclohexylphosphine)ruthenium) was purchased from Aldrich. The preparations of the homoallyl alcohols **1** are described in the following section. All reactions were carried out under argon atmosphere.

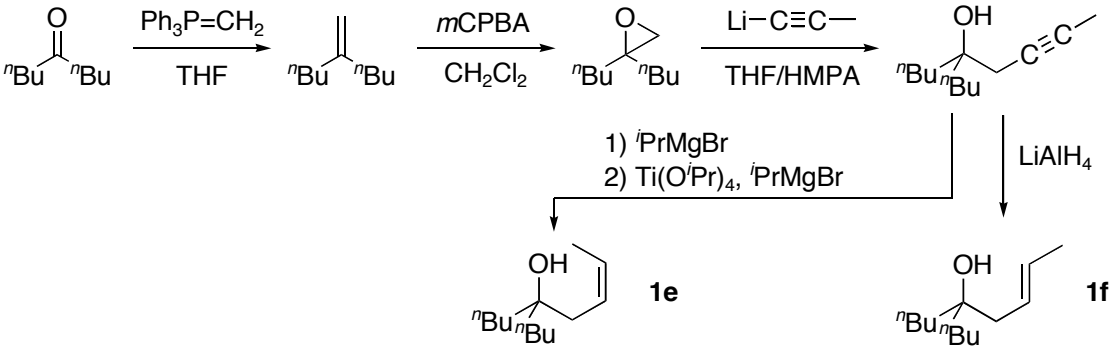
Experimental Procedure

Preparation of 1a–1d



Preparation of **1a** is representative. Under argon atmosphere, a solution of methallylmagnesium chloride (1.00 M THF solution, 28.0 mL, 28.0 mmol) and THF (20 mL) were placed in a 100-mL reaction flask. At 0 ˚C, 2,4-dimethyl-3-pentanone (3.28 mL, 23.0 mmol) was added dropwise via a syringe to the solution. The mixture was stirred for 1.5 h at room temperature. The mixture was poured into 1 M hydrochloric acid (30 mL). Extractive workup followed by silica gel column purification (hexane/ethyl acetate = 10:1) provided **1a** (3.66 g, 21.5 mmol, 93%).

Preparation of 1e and 1f



Sodium hydride (60% suspension in oil, 1.20 g, 30 mmol) was placed in a 100-mL reaction flask equipped with a Dimroth condenser under argon. The hydride was washed with hexane (10 mL × 3) and THF (10 mL × 1). THF (50 mL) and methyltriphenylphosphonium iodide (11 g, 27 mmol) were added at 0 ˚C. After the resulting mixture was stirred for 1 h at ambient temperature, 5-nonanone (4.34 mL, 25.0 mmol) was added. The whole mixture was heated at 50 ˚C for 2 h. Triphenylphosphine oxide was removed to yield 2-butyl-1-hexene. The crude oil was dissolved in 30 mL of dichloromethane in a 100-mL round-bottomed flask. *m*-Chloroperbenzoic acid (77% purity, 5.6 g, 25 mmol) was added portionwise at 0 ˚C. After being stirred for 1 h at room temperature, the mixture was quenched with saturated sodium thiosulfate (5 mL). Sodium hydroxide solution (1 M, 30 mL) was then added. Organic components were extracted with hexane/ethyl acetate = 10:1 three times. The organic layer was washed with sodium hydroxide solution (1 M, 30 mL). Concentration followed by purification on silica gel (hexane/ethyl acetate = 10:1) yielded 1,2-epoxy-2-butylhexane (2.70 g, 17.3 mmol,

69%) as a colorless oil.

A 200-mL three-necked reaction flask equipped with a dropping funnel and a Dimroth condenser was allowed to cool to –78 ˚C under argon. Gaseous propyne was charged into the reaction flask to obtain ca. 2 mL (40 mmol) of liquid propyne. THF (50 mL) and then butyllithium (1.62 M hexane solution, 21.4 mL, 34.6 mmol) were added dropwise through the dropping funnel at –78 ˚C. After completion of the addition, hexamethylphosphoramide (15 mL) and 1,2-epoxy-2-butylhexane (2.70 g, 17.3 mmol in 10 mL of THF) were added via syringes. The resulting solution was stirred at –78 ˚C for 15 min and was allowed to warm gradually to room temperature by removing the bath. The mixture was heated at 50 ˚C for 60 h. The reaction was quenched with saturated ammonium chloride solution (40 mL). Extraction, concentration, and purification furnished 5-butyl-2-nonyn-5-ol (2.86 g, 14.6 mmol, 84%).

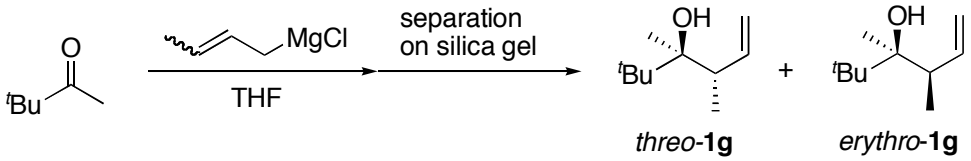
The reduction of the alkynol to yield **1e** was performed according to the literature.[1] Under an atmosphere of argon, diethyl ether (8 mL), 5-butyl-2-nonyn-5-ol (1.62 g, 8.26 mmol), and isopropylmagnesium bromide (0.98 M ethereal solution, 8.5 mL, 8.3 mmol) were sequentially added at 0 ˚C. After being stirred for 10 min at 0 ˚C, the mixture was cooled to –78 ˚C. Titanium tetraisopropoxide (3.05 mL, 10.3 mmol) and isopropylmagnesium bromide (21.1 mL, 20.7 mmol) were added to obtain a black solution. The mixture was allowed to warm to –50 ˚C and stirred for 3 h at the same temperature. The mixture was carefully poured into ice-cold 1 M hydrochloric acid (40 mL). The resulting mixture was stirred for 30 min at ambient temperature. Extractive workup and silica gel column purification afforded 1.36 g of **1e** (6.85 mmol, 83%) as a colorless oil.

Preparation of **1f** also started from 5-butyl-2-nonyn-5-ol. Lithium aluminum hydride (0.911 g, 24 mmol) was placed in a 100-mL reaction flask. THF (32 mL) and the alcohol (1.18 g, 6.0 mmol, dissolved in 13 mL of THF) were added through a dropping funnel. After completion of the addition, the whole mixture was stirred for 24 h at reflux. After being cooled to room temperature, the mixture was poured into ice-cold hydrochloric acid (1 M). The products were extracted with hexane/ethyl acetate = 10:1. The organic layer was dried over magnesium sulfate and concentrated in vacuo. Purification of the crude product was performed on silica gel neutral (Kanto Chemical, spherical, neutral, 60N) with hexane/ethyl acetate = 10:1 as an eluent. (*E*)-5-Butyl-2-nonen-5-ol (**1f**) was obtained in 89% yield (1.05 g, 5.32 mmol, a

[1] Sato, F.; Urabe, H.; Okamoto, S. *Chem. Rev.* **2000**, *100*, 2789–2834.

colorless oil).

Preparation of *threo*- and *erythro*-1g



threo-**1g** *erythro*-**1g**

Crotylmagnesium chloride (0.95 M THF solution, 25.3 mL, 24 mmol) was added to a solution of pinacolone (2.47 mL, 20.0 mmol) in ether (30 mL) at 0 ˚C under an atmosphere of argon. The mixture was stirred at room temperature for 1.5 h. The reaction mixture was poured into 1 M hydrochloric acid (30 mL), and products were extracted with ether. Silica gel column purification (hexane/ethyl acetate = 20:1) provided *threo*-**1g** (R_f = 0.45, 1.89 g, 12.1 mmol, 60%) and *erythro*-**1g** (R_f = 0.36, 0.24 g, 1.5 mmol, 8%). The relative stereochemistry was determined according to the literature.[2]

Preparation of *threo*- and *erythro*-1h

threo-**1h** *erythro*-**1h**

Ether (60 mL) and allyl phenyl ether (1.37 mL, 10.0 mmol) were placed in a 100-mL reaction flask under argon. At –78 ˚C, *sec*-butyllithium (1.01 M cyclohexane solution, 9.90 mL, 10.0 mmol) was added dropwise via a syringe. After the mixture was stirred for 30 min at –78 ˚C, triethylaluminum (0.92 M hexane solution, 10.9 mL, 10.0 mmol) and pinacolone (1.24 mL, 10.0 mmol) were added.[3] The resulting mixture was allowed to warm to room temperature and stirred for 8 h. The reaction was quenched with 1 M hydrochloric acid (30 mL). Extraction with hexane/ethyl acetate = 5:1 and concentration in vacuo provided a crude oil that mainly consisted of *threo*-**1h** and *erythro*-**1h**. Purification on silica gel (gradient starting from hexane/ethyl acetate = 40:1) afforded *threo*-**1h** (R_f = 0.38 (hexane/ethyl acetate = 10:1), 0.542 g, 2.31 mmol, 23%) and *erythro*-**1h** (R_f = 0.25 (hexane/ethyl acetate = 10:1), 0.689 g, 2.94 mmol, 29%). The relative stereochemistry was determined as shown in the literature.[4]

Preparation of 1i

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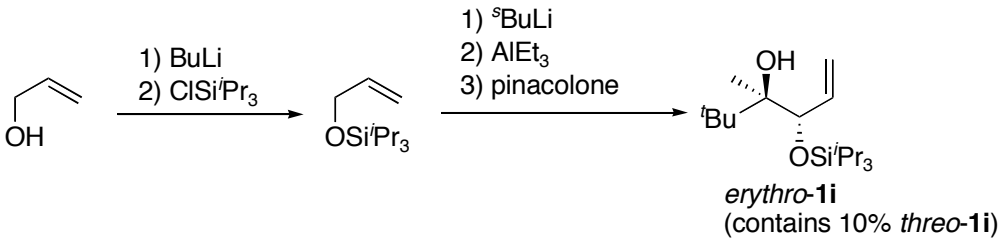
[2] Tan, K.-T.; Chug, S.-S.; Cheng, H.-S.; Loh, T.-P. *J. Am. Chem. Soc.* **2003**, *125*, 2958–2963.
[3] Yamamoto, Y.; Yatagai, H.; Saito, Y. *J. Org. Chem.* **1984**, *49*, 1096–1104.
[4] Hayashi, S.; Hirano, K.; Yorimitsu, H.; Oshima, K. *J. Am. Chem. Soc.* **2006**, *128*, 2210–2211.

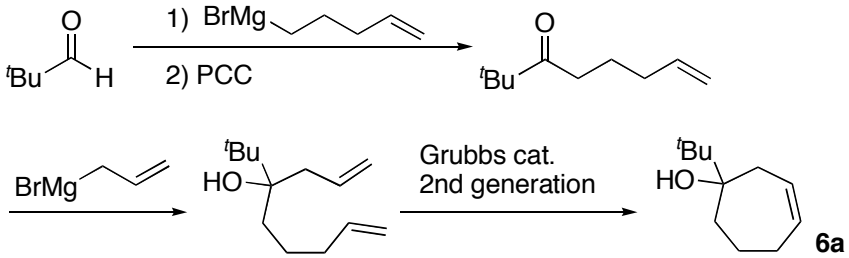


erythro-**1i**
(contains 10% *threo*-**1i**)

A 100-mL reaction flask was filled with argon, and THF (15 mL), allyl alcohol (1.22 mL, 18.0 mmol), and butyllithium (1.60 M hexane solution, 10.3 mL, 16.5 mmol) were added at –78 ˚C. The resulting mixture was stirred for 30 min. Chlorotriisopropylsilane (3.21 mL, 15.0 mmol) was added at –78 ˚C. The mixture was stirred at room temperature for 3 h. The reaction was quenched with saturated ammonium chloride. Extraction and silica gel column purification (hexane/ethyl acetate = 10:1) provided allyl triisopropylsilyl ether (2.84 g, 13.3 mmol, 88%).

Ether (39 mL) and allyl triisopropylsilyl ether (2.84 g, 13.3 mmol) were placed in a 100-mL reaction flask under argon. At –78 ˚C, *sec*-butyllithium (1.00 M cyclohexane solution, 13.3 mL, 13.3 mmol) was added dropwise via a syringe. The mixture was stirred for 30 min at –40 ˚C. After the mixture was cooled to –78 ˚C, triethylaluminum (0.92 M hexane solution, 14.4 mL, 13.3 mmol) and pinacolone (1.64 mL, 13.3 mmol) were added.[3] The resulting mixture was stirred for 5 min at the same temperature, then allowed to warm to room temperature, and stirred for 5 h. The reaction was quenched with saturated ammonium chloride solution. A crude oil was purified on silica gel (hexane/ethyl acetate = 40:1) provided **1i** as a mixture of *erythro* and *threo* isomers in a ratio of 9:1 (2.09 g, 6.65 mmol, 50%, not optimized). Homoallyl alcohol **1i** was used as the mixture. The relative stereochemistry was inversely deduced from the product **3t**.

Preparation of endocyclic homoallyl alcohol 6a



6a

Under an atmosphere of argon, 4-pentenylmagnesium bromide (1.0 M THF solution, 25 mL, 25 mmol) was placed in a 100-mL reaction flask. Pivalaldehyde (2.17 mL, 20 mmol) was added to the Grignard reagent dropwise at 0 ˚C. The mixture was warmed to room temperature

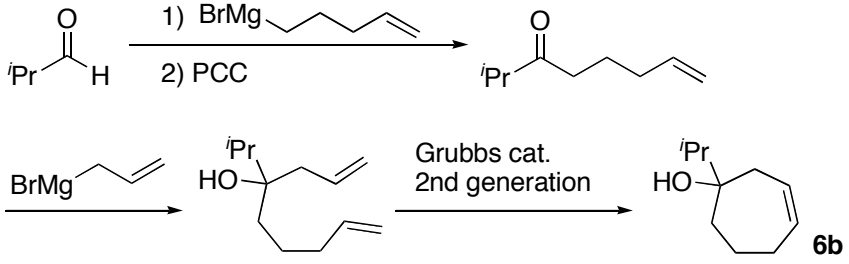
and was stirred for 1.5 h. The reaction mixture was poured into saturated ammonium chloride solution (30 mL). The product was extracted with hexane (30 mL × 3), and organic layers were washed with brine. The combined organic layer was dried over sodium sulfate, and concentrated in vacuo to afford 2,2-dimethyl-7-octen-3-ol as a crude oil.

PCC (4.89 g, 22.6 mmol) and silica gel (4.9 g, Wakogel 200 mesh) were mixed in a mortar. The mixture was transferred to a 100-mL reaction flask. A solution of the crude alcohol in dichloromethane (57 mL) was then charged. The mixture was stirred for 6 h at ambient temperature. The mixture was filtered through a pad of Celite. The pad was washed with hexane and ether. After evaporation, the residue was passed through a pad of silica gel with ether as an eluent to remove chromium compounds. The corresponding ketone was obtained in 77% yield (2.36 g, 15.3 mmol).

Allylmagnesium bromide (0.77 M ethereal solution, 12.9 mL, 9.9 mmol) was placed in a 100-mL reaction flask under argon. The ketone (1.3 g, 8.2 mmol) in THF (8.2 mL) was added dropwise at 0 ˚C. After being stirred for 1.5 h at ambient temperature, the mixture was poured into saturated ammonium chloride solution (30 mL). The product was extracted with ethyl acetate (20 mL × 3), and organic layers were washed with brine. The combined organic layer was dried over sodium sulfate, and evaporated in vacuo to yield 4-*tert*-butyl-1,8-nonadien-4-ol.

The crude tertiary alcohol was dissolved in dichloromethane (30 mL) under argon. Grubbs Catalyst, 2nd Generation (34 mg, 0.038 mmol) was added to the solution. After being stirred for 20 h at room temperature, the mixture was filtered through a pad of Florisil. Evaporation followed by silica gel column purification (hexane/ether = 10:1) afforded **6a** (1.35 g, 8.05 mmol, 98%).

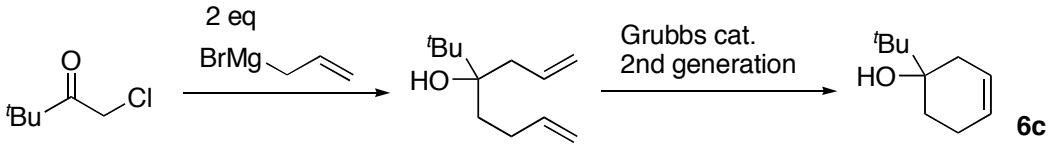
Preparation of 6b



Isopropyl-substituted alcohol **6b** was prepared in a fashion similar to that of **6a**. The Grignard addition followed by the oxidation provided 2.42 g of 2-methyl-7-octen-3-one (17.3 mmol, 87% yield). The allylation of the ketone proceeded quantitatively. The final ring-closing metathesis provided 401 mg of **6b** (2.60 mmol, 77%) starting from 612 mg (3.36 mmol)

of the dienol.

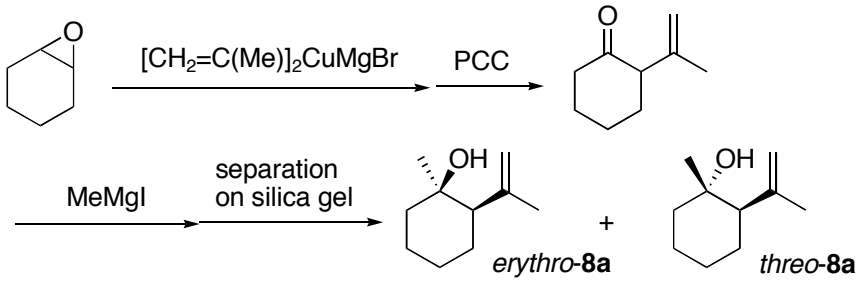
Preparation of 6c



Under an atmosphere of argon, allylmagnesium bromide (0.85 M THF solution, 26 mL, 22 mmol) was placed in a 100-mL reaction flask. 1-Chloro-3,3-dimethyl-2-butanone (1.31 mL, 10 mmol) was added to the solution dropwise at 0 ˚C. The mixture was allowed to warm to room temperature and was stirred for 4.5 h. The reaction mixture was poured into saturated ammonium chloride solution (30 mL), and the product was extracted with ethyl acetate (30 mL × 3). The combined organic layer was dried and concentrated. Alcohol, 4-*tert*-butyl-1,7-octadien-4-ol, was obtained as a crude oil.

The crude tertiary alcohol was treated with Grubbs Catalyst, 2nd Generation (45 mg, 0.050 mmol) in dichloromethane (40 mL) for 20 h at room temperature under argon. The mixture was filtered through a pad of Florisil, and the filtrate was evaporated. The product was chromatographed on silica gel (hexane/ether = 5:1) to afford **6c** (1.53 g, 9.90 mmol, 99%).

Preparation of *erythro*- and *threo*-8a



Copper(I) iodide (1.07 g, 5.63 mmol) was placed in a 300-mL reaction flask. THF (100 mL) and 1,2-epoxycyclohexane (5.64 mL, 56.3 mmol) were added. After the mixture was cooled to –40 ˚C, isopropenylmagnesium bromide (1.0 M THF solution, 113 mL, 113 mmol) was added through a dropping funnel. The mixture was allowed to warm to –20 ˚C, and stirred for 20 h. The reaction mixture was poured into saturated ammonium chloride solution (200 mL). The product was extracted with hexane (200 mL × 3), and each organic layer was washed with brine. The combined organic layer was dried over sodium sulfate, and concentrated in vacuo to afford 2-isopropenylcyclohexanol as a crude oil.

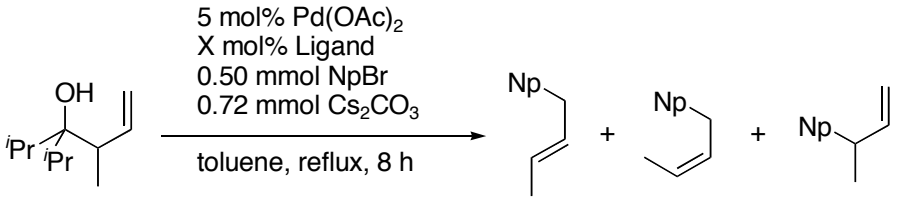
PCC (14.0 g, 64.7 mmol) and silica gel (14 g, Wakogel 200 mesh) were mixed in a

mortar. The mixture was placed in a 300-mL reaction flask. A solution of the crude alcohol in dichloromethane (162 mL) was then charged under argon. The mixture was stirred for 12 h at room temperature. The mixture was filtered through a pad of Celite. The filtrate was evaporated in vacuo. Silica gel column chromatography (hexane/ether = 5:1) yielded 6.60 g of 2-isopropenylcyclohexanone (47.8 mmol, 85% yield).

Methylmagnesium iodide (1.0 M ethereal solution, 57.4 mL, 57.4 mmol) was placed in a 200-mL reaction flask under argon. The ketone (6.60 g, 47.8 mmol) in THF (50 mL) was added dropwise at 0 ˚C. After being stirred for 1.5 h at 0 ˚C, the mixture was poured into saturated ammonium chloride solution (100 mL). Extraction with hexane (100 mL × 3), concentration, and silica gel column purification (hexane/ethyl acetate = 20:1) provided *erythro*-**8a** (5.62 g, 36.4 mmol, 76%) and *threo*-**8a** (0.54 g, 3.5 mmol, 7.4%). The relative stereochemistry was determined by comparing ^{1}H and ^{13}C NMR data of closely related cyclic alcohols in the literature.[5]

Alcohols **8b** and **8c** were prepared in similar fashions.

Results of Ligand Screening

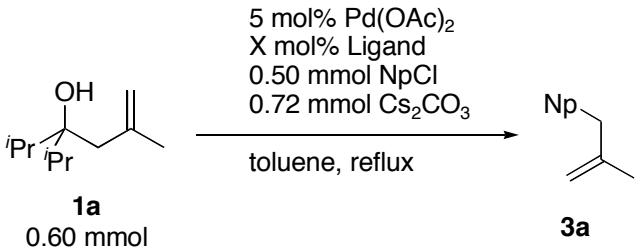


1d 0.60 mmol

(*E*)-**3l** (*Z*)-**3l** **3m**

Ligand	X	**3l** (%)	*E*/*Z*	**3m** (%)
PPh_3	20	88	46:54	3
P(*o*-tol)$_3$	20	11	77:23	<1
P(2-furyl)$_3$	20	54	55:45	9
P(*p*-tol)$_3$	20	94	46:54	6
P(*p*-tol)$_3$	25	87	46:54	6
P(*p*-tol)$_3$	10	10	46:54	3
PMe_3	20	38	70:30	<1
PCy_3	**10**	**80**	**63:37**	**5**
DPPM	10	42	49:51	4
DPPE	10	48	51:49	5
DPPP	10	27	52:48	<1
DPPB	10	66	51:49	5

[5] Molander, G. A.; Cormier, E. P. *J. Org. Chem.* **2005**, *70*, 2622–2626.



1a
0.60 mmol

3a

Ligand	X	Time (h)	**3a** (%)
P(*p*-tol)$_3$	20	8	6 (**1a** 69% recovery)
PCy$_3$	10	12	79
PCy$_3$	20	12	6 (**1a** 72% recovery)

Characterization of Compounds

Compounds **1a**,[6] **1b**,[7] **1c**,[8] **3d**,[9] **3h**,[10] **3p**,[11] and **8b**[5] were known compounds. Characterization data of **1d–1i**, **3a**, **3e**, **3f**, **3i**, **3j**, **3l–3o**, **3r**, and **3t** were reported in the previous communication.[4]

4-(2-Methyl-2-propenyl)biphenyl (3b): IR (neat) 3029, 2913, 2852, 1647, 1517, 1486, 1436, 1409, 1374, 1009, 892, 804, 760, 743, 697 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.72 (s, 3H), 3.37 (s, 2H), 4.78 (m, 1H), 4.84 (m, 1H), 7.26–7.28 (m, 2H), 7.32–7.35 (m, 1H), 7.42–7.45 (m, 2H), 7.52–7.54 (m, 2H), 7.58–7.60 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 22.34, 44.49, 112.26, 127.22, 127.24 (overlapped), 128.93, 129.52, 139.10, 139.22, 141.27, 145.27. Found: C, 92.31; H, 7.77%. Calcd for $C_{16}H_{16}$: C, 92.26; H, 7.74%.

2-(2-Methyl-2-propenyl)naphthalene (3c): IR (neat) 3053, 3021, 2969, 2909, 2854, 1652, 1635, 1600, 1508, 1436, 1374, 892, 857, 807, 758, 739 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.72 (s, 3H), 3.50 (s, 2H), 4.79–4.81 (m, 1H), 4.86–4.88 (m, 1H), 7.35 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.45 (quintet of doublet, *J* = 7.0, 1.5 Hz, 2H), 7.64 (s, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.79–7.83 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 22.36, 45.02, 112.39, 125.45, 126.08, 127.36, 127.70, 127.76, 127.82, 128.02, 132.35, 133.76, 137.50, 145.25. Found: C, 92.39; H, 7.88%. Calcd for $C_{14}H_{14}$: C, 92.26; H,

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[6] Durandetti, S.; Sibille, S.; Périchon, J. *J. Org. Chem.* **1989**, *54*, 2198–2204.
[7] Katzenellenbogen, J. A.; Lenox, R. S. *J. Org. Chem.* **1973**, *38*, 326–335.
[8] Melby, E.; Kennedy, J. P. *J. Org. Chem.* **1974**, *39*, 2433–2434.
[9] Konakahara, T.; Takaji, Y. *Synthesis* **1979**, 192–194.
[10] Fujiwara, N.; Yamamoto, Y. *J. Org. Chem.* **1999**, *64*, 4095–4101.
[11] Lajis, N. H.; Khan, M. N. *Tetrahedron* **1992**, *48*, 1109–1114.

7.74%.

4-(2-Methyl-2-propenyl)-*N*,*N*-dimethylaniline (3g): IR (neat) 3072, 2852, 2800, 1649, 1615, 1520 ,1444, 1346, 1227, 1163, 948, 888, 797, 567 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.68 (s, 3H), 2.92 (s, 6H), 3.23 (s, 2H), 4.71–4.73 (m, 1H), 4.76–4.78 (m, 1H), 6.70 (d, *J* = 9.0 Hz, 2H), 7.06 (d, *J* = 9.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 22.23, 31.13, 41.08, 43.87, 111.34, 113.06, 128.11, 129.68, 146.20, 149.41. Found: C, 82.11; H, 9.84%. Calcd for $C_{12}H_{17}N$: C, 82.23; H, 9.78%.

1-(*tert*-Butyl)-3-cyclohepten-1-ol (6a): IR (neat) 3581, 2958, 1654, 1480, 1367, 1205, 1071, 980, 923, 854, 820, 761, 691 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.96 (s, 9H), 1.52–1.60 (m, 1H), 1.63–1.70 (m, 1H), 1.71–1.76 (m, 1H), 1.80 (bs, 1H), 1.88–1.93 (m, 1H), 2.07–2.14 (m, 1H), 2.19–2.26 (m, 1H), 2.28–2.33 (m, 1H), 2.43 (ddd, *J* = 15.0, 8.5, 2.0 Hz, 1H), 5.51–5.56 (m, 1H), 5.95–6.00 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 22.29, 25.44, 29.21, 33.73, 37.22, 38.53, 75.45, 126.52, 135.31. Found: C, 78.55; H, 12.19%. Calcd for $C_{11}H_{20}O$: C, 78.51; H, 11.98%.

1-Isopropyl-3-cyclohepten-1-ol (6b): IR (neat) 3420, 2840, 1456, 989 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.89 (d, *J* = 5.0 Hz, 3H), 0.90 (d, *J* = 5.0 Hz, 3H), 1.50–1.54 (m, 2H), 1.66–1.72 (m, 2H), 1.81–1.86 (m, 2H), 2.05–2.12 (m, 1H), 2.14–2.25 (m, 2H), 2.31–2.35 (m, 1H), 5.57–5.62 (m, 1H), 5.94–5.99 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 16.84, 16.94, 22,01, 28.83, 36.23, 37.74, 40.51, 73.34, 126.65, 135.08. Found: C, 77.65; H, 11.99%. Calcd for $C_{10}H_{18}O$: C, 77.87; H, 11.76%.

1-*tert*-Butyl-3-cyclohexen-1-ol (**6c**): IR (neat) 3479, 2961, 1367, 1083, 872, 656 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.94 (s, 9H), 1.46–1.52 (m, 2H), 1.74–1.79 (m, 1H), 1.91–1.97 (m, 1H), 2.03–2.11 (m, 1H), 2.14–2.24 (m, 1H), 2.26–2.32 (m, 1H), 5.59–5.63 (m, 1H), 5.74–5.78 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 22.35, 25.01, 27.04, 32.92, 37.36, 73.84, 124.94, 126.94. Found: C, 77.76; H, 12.01%. Calcd for $C_{10}H_{18}O$: C, 77.84; H, 11.76%.

7-(2,6-Dimethylphenyl)-2,2-dimethyl-8-nonen-3-one (7a): IR (neat) 2931, 2869, 1706, 1633, 1467, 1366, 992, 912, 769 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.11 (s, 9H), 1.37–1.46 (m, 1H), 1.58–1.67 (m, 1H), 1.73–1.90 (m, 2H), 2.40 (s, 6H), 2.46 (dt, *J* = 2.5, 7.5 Hz, 2H), 3.81–3.86 (m, 1H), 4.94 (ddd, *J* = 17.5, 2.0, 2.0 Hz, 1H), 5.04 (ddd, *J* = 10.0, 2.0, 2.0 Hz, 1H), 6.09 (ddd, *J* = 17.5, 10.0, 5.0 Hz, 1H), 6.75–7.05 (m, 3H); ^{13}C NMR ($CDCl_3$) δ 21.73, 22.73, 26.51, 32.45, 36.50, 44.13, 44.21, 114.05, 126.09, 129(br), 136.71, 140.09, 140.57, 215.80. Found: C, 84.05; H, 10.34%. Calcd for $C_{19}H_{28}O$: C, 83.77; H, 10.36%.

2,2-Dimethyl-7-(2-phenylphenyl)-8-nonen-3-one (7b): IR (neat) 2933, 1706, 1478, 1367 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.04 (s, 9H), 1.29–1.36 (m, 2H), 1.58–1.63 (m, 2H), 2.25 (dd, *J* = 14.0, 6.5 Hz, 2H), 3.44 (q, *J* = 7.5 Hz, 1H), 4.83–5.01 (m, 2H), 5.92–5.99 (m, 1H), 7.20–7.42 (m, 9H);

^{13}C NMR ($CDCl_3$) δ 21.55, 26.53, 35.54, 36.12, 44.13, 44.45, 114.33, 125.86, 127.02, 127.13, 127.83, 128.16, 129.61, 130.18, 141.46, 141.89, 142.24, 142.75, 215.98. Found: C, 86.29; H, 8.93%. Calcd for $C_{23}H_{28}O$: C, 86.20; H, 8.81%.

2,2-Dimethyl-7-(2-methylphenyl)-8-nonen-3-one (7c): IR (neat) 2955, 1706, 1478, 1367 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.11 (s, 9H), 1.44–1.54 (m, 1H), 1.58–1.74 (m, 3H), 2.33 (s, 3H), 2.48 (t, *J* = 7.0 Hz, 2H), 3.50 (t, *J* = 7.0 Hz, 1H), 4.96–5.02 (m, 2H), 5.85–5.91 (m, 1H), 7.07–7.18 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 19.81, 22.19, 26.58, 34.69, 36.50, 44.22, 45.21, 114.35, 126.05, 126.34, 126.43, 130.54, 135.97, 141.73, 142.31, 216.00. Found: C, 83.64; H, 10.14%. Calcd for $C_{18}H_{26}O$: C, 83.67; H, 10.14%.

2,2-Dimethyl-7-(4-methylphenyl)-8-nonen-3-one (7d): IR (neat) 2925, 1706, 1513, 1464, 1367, 816 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.11 (s, 9H), 1.41–1.51 (m, 1H), 1.54–1.71 (m, 3H), 2.31 (s, 3H), 2.46 (t, *J* = 6.5 Hz, 2H), 3.20 (q, *J* = 2.5 Hz, 1H), 4.99–5.04 (m, 2H), 5.89–5.96 (m, 1H), 7.06–7.12 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 21.18, 22.12, 26.58, 35.12, 36.41, 44.23, 49.69, 114.13, 127.57, 129.33, 135.84, 141.38, 142.43, 216.01. Found: C, 83.77; H, 9.84%. Calcd for $C_{18}H_{26}O$: C, 83.67; H, 10.14%.

2,2-Dimethyl-7-(4-trifluoromethylphenyl)-8-nonen-3-one (7e): IR (neat) 2969, 1706, 1618, 1326, 1164, 1124, 1070, 1018 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.04 (s, 9H), 1.32–1.67 (m, 4H), 2.40 (t, *J* = 6.8 Hz, 2H), 3.24 (q, *J* = 7.5 Hz, 1H), 4.69–5.01 (m, 2H), 5.84 (ddd, *J* = 17.5, 10.0, 7.5 Hz, 1H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.48 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.99, 26.57, 34.94, 36.28, 44.24, 50.01, 115.25, 124.45 (q, *J* = 271 Hz), 125.60 (q, *J* = 4 Hz), 128.10, 128.69 (q, *J* = 32 Hz), 141.23, 148.47, 215.83. Found: C, 69.00; H, 7.37%. Calcd for $C_{18}H_{23}F_3O$: C, 69.21; H, 7,42; F, 18.25%.

7-(4-Ethoxycarbonylphenyl)-2,2-dimethyl-8-nonen-3-one (7f): IR (neat) 2971, 1717, 1610, 1277, 1105 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.04 (s, 9H), 1.31 (t, *J* = 7.5 Hz, 3H), 1.32–1.40 (m, 1H), 1.43–1.69 (m, 3H), 2.40 (t, *J* = 7.5 Hz, 2H), 3.23 (q, *J* = 7.5 Hz, 1H), 4.29 (q, *J* = 7.5 Hz, 2H), 4.95–4.99 (m, 2H), 5.82–5.89 (m, 1H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.90 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 14.52, 22.01, 26.55, 24.95, 36.28, 44.21, 50.13, 60.96, 115.09, 127.73, 128.68, 129.97, 141.31, 149.69, 166.74, 215.81. Found: C, 75.90; H, 8.87%. Calcd for $C_{20}H_{28}O_3$: C, 75.91; H, 8.92%.

7-(2,6-Dimethylphenyl)-2-methyl-8-nonen-3-one (7g): IR (neat) 2968, 2360, 1714, 1468, 993, 769 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.06 (d, *J* = 7.0 Hz, 6H), 1.38–1.46 (m, 1H), 1.57–1.67 (m, 1H), 1.73–1.81 (m, 1H), 1.83–1.90 (m, 1H), 2.33 (s, 6H), 2.43 (t, *J* = 7.0 Hz, 2H), 2.55 (sept, *J* = 7.0

Hz, 1H), 3.80–3.85 (m, 1H), 4.93 (ddd, *J* = 17.0, 2.0, 1.5 Hz, 1H), 5.03 (ddd, *J* = 10.5, 2.0, 1.5 Hz, 1H), 6.07 (ddd, *J* = 17.0, 10.5, 5.0 Hz, 1H), 6.99 (s, 3H); ^{13}C NMR ($CDCl_3$) δ 18.42, 21.79, 22.66, 32.50, 40.44, 40.95, 44.21, 114.19, 126.16, 129 (br), 136.79, 140.13, 140.56, 214.83. Found: C, 83.63; H, 9.95%. Calcd for $C_{18}H_{26}O$: C, 83.67; H, 10.14%.

2,2-Dimethyl-6-(1-naphthyl)-7-octen-3-one (7h): IR (neat) 2967, 1703, 1477, 1367, 779 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.07 (s, 9H), 2.08–2.23 (m, 2H), 2.50–2.58 (m, 2H), 4.16 (q, *J* = 7.5 Hz, 1H), 5.10–5.13 (m, 2H), 6.03–6.10 (m, 1H), 7.39 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.44–7.55 (m, 3H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.86 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 26.62, 29.14, 34.49, 43.52, 44.28, 115.27, 123.65, 124.10, 125.59, 125.69, 126.05, 127.08, 129.05, 131.94, 134.22, 140.00, 141.57, 215.97. Found: C, 85.60; H, 8.46%. Calcd for $C_{20}H_{24}O$: C, 85.67; H, 8.63%.

***erythro*-1-Methyl-2-(1-methylethenyl)-1-cyclohexanol (*erythro*-8a)**: IR (neat) 3494, 2931, 1638, 1449, 1373 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.12 (s, 3H), 1.16–1.25 (m, 1H), 1.29–1.36 (m, 1H), 1.39–1.44 (m, 1H), 1.47–1.75 (m, 6H), 1.80 (s, 3H), 1.89 (dd, *J* = 13.0, 3.5 Hz, 1H), 4.74 (s, 1H), 4.87 (s, 1H); ^{13}C NMR ($CDCl_3$) δ 21.91, 24.86, 26.38, 27.97, 30.10, 40.16, 53.62, 70.54, 112.00, 148.65. HRMS (EI) Found: 154.1360 [M$^+$]; Calcd for $C_{10}H_{18}O$: 154.1358.

***threo*-1-Methyl-2-(1-methylethenyl)-1-cyclohexanol (*threo*-8a)**: IR (neat) 3426, 2933, 1641 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.12 (s, 3H), 1.21–1.36 (m, 2H), 1.39–1.47 (m, 2H), 1.61–1.75 (m, 4H), 1.77 (s, 3H), 2.01 (brs, 1H), 2.12 (dd, *J* = 12.5, 3.5 Hz, 1H), 4.75–4.76 (m, 1H), 4.93–4.95 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 22.47, 22.91, 24.10, 26.26, 28.74, 41.74, 55.23, 72.29, 114.11, 146.56. HRMS (CI) Found: 154.1357 [M$^+$]; Calcd for $C_{10}H_{18}O$: 154.1358.

***erythro*-1-Butyl-2-(1-methylethenyl)cyclohexan-1-ol (*erythro*-8c)**: IR (neat) 3497, 2934, 1637, 1448, 889 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 0.89 (t, *J* = 7.0 Hz, 3H), 1.19–1.33 (m, 6H), 1.37–1.45 (m, 4H), 1.53–1.61 (m, 2H), 1.67–1.76 (m, 3H), 1.80 (s, 3H), 1.99 (dd, *J* = 13.0, 4.0 Hz, 1H), 4.76 (brs, 1H), 4.85 (brs, 1H); ^{13}C NMR ($CDCl_3$) δ 14.30, 21.76, 23.54, 24.46, 25.95, 26.43, 28.33, 36.32, 42.02, 52.38, 72.79, 112.18, 148.70. Found: C, ; H, %. Calcd for $C_{13}H_{24}O$: C, 79.53; H, 12.32%.

(*Z*)-9-(2,6-Dimethylphenyl)-8-methyl-7-nonen-2-one ((*Z*)-9a): IR (neat) 2933, 1717, 1436, 1360, 1163, 769 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.40 (s, 3H), 1.37–1.43 (m, 2H), 1.60–1.66 (m, 2H), 2.15 (q, *J* = 7.0 Hz, 2H), 2.16 (s, 3H), 2.28 (s, 6H), 2.46 (t, *J* = 7.5 Hz, 2H), 3.43 (s, 2H), 5.21–5.24 (m, 1H), 6.98–7.03 (m, 3H); ^{13}C NMR ($CDCl_3$) δ 20.54, 22.54, 23.81, 27.80, 29.42, 30.08, 31.73, 43.87, 125.75, 125.92, 128.13, 133.07, 137.06, 137.18, 209.41. Found: C, 83.38;

H, 10.34%. Calcd for $C_{18}H_{26}O$: C, 83.67; H, 10.14%.

(*Z*)-8-Methyl-9-(4-trifluoromethylphenyl)-7-nonen-2-one ((*Z*)-9b): IR (neat) 2392, 1717, 1327, 1123 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.36–1.42 (m, 2H), 1.60 (s, 3H), 1.58–1.65 (m, 2H), 2.12 (q, *J* = 7.0 Hz, 2H), 2.13 (s, 3H), 2.43 (t, *J* = 7.5 Hz, 2H), 3.40 (s, 2H), 5.32–5.35 (m, 1H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.52 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 23.49, 23.66, 28.16, 29.62, 30.08, 37.82, 43.77, 123.52 (q, *J* = 270 Hz), 125.41 (q, *J* = 4 Hz), 127.31, 128.34 (q, *J* = 32 Hz), 128.92, 133.25, 144.50, 209.24. Found: C, 68.56; H, 7.05%. Calcd for $C_{17}H_{21}F_3O$: C, 68.44; H, 7.09%.

(*Z*)-9-(4-Ethoxycarbonylphenyl)-8-methyl-7-nonen-2-one ((*Z*)-9c): IR (neat) 1717, 1611, 1276, 1103 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.38 (t, *J* = 7.0 Hz, 3H), 1.35–1.41 (m, 2H), 1.59 (s, 3H), 1.57–1.63 (m, 2H), 2.12 (q, *J* = 7.0 Hz, 2H), 2.13 (s, 3H), 2.43 (t, *J* = 7.0 Hz, 2H), 3.40 (s, 2H), 4.35 (q, *J* = 7.0 Hz, 2H), 5.30–5.33 (m, 1H), 7.21 (d, *J* = 8.5 Hz, 2H), 7.95 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 14.51, 23.52, 23.66, 28.15, 29.62, 30.08, 38.04, 43.78, 60.94, 127.11, 128.39, 128.62, 129.80, 133.43, 145.82, 166.82, 209.29. Found: C, 75.34; H, 8.86%. Calcd for $C_{19}H_{26}O_3$: C, 75.46; H, 8.67%.

(*Z*)-9-(4-Methoxyphenyl)-8-methyl-7-nonen-2-one ((*Z*)-9d): IR (neat) 2933, 1717, 1510, 1246 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.35–1.41 (m, 2H), 1.60 (s, 3H), 1.58–1.64 (m, 2H), 2.13 (s, 3H), 2.11–2.16 (m, 2H), 2.43 (t, *J* = 7.5 Hz, 2H), 3.29 (s, 2H), 3.78 (s, 3H), 5.25–5.28 (m, 1H), 6.82 (d, *J* = 9.0 Hz, 2H), 7.06 (d, *J* = 9.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 23.49, 23.71, 28.04, 29.74, 30.08, 37.04, 43.84, 55.41, 113.87, 126.10, 129.53, 132.33, 134.68, 157.95, 209.41. Found: C, 78.50; H, 9.38%. Calcd for $C_{17}H_{24}O_2$: C, 78.42; H, 9.29%.

(*Z*)-8-Methyl-9-(2-methylphenyl)-7-nonen-2-one ((*Z*)-9e): IR (neat) 2932, 1717, 1358 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.36–1.42 (m, 2H), 1.60 (s, 3H), 1.58–1.64 (m, 2H), 2.08 (q, *J* = 7.5 Hz, 2H), 2.13 (s, 3H), 2.29 (s, 3H), 2.42 (t, *J* = 7.5 Hz, 2H), 3.33 (s, 2H), 5.34 (dt, *J* = 1.0, 7.0 Hz, 1H), 7.08–7.15 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 19.78, 23.70, 23.74, 27.98, 29.54, 30.04, 35.28, 43.81, 126.01, 126.05, 126.81, 128.46, 130.08, 133.46, 136.71, 138.09, 209.40. Found: C, 83.83; H, 10.01%. Calcd for $C_{17}H_{24}O$: C, 83.55; H, 9.90%.

(*Z*)-8-Methyl-9-(4-methylphenyl)-7-nonen-2-one ((*Z*)-9f): IR (neat) 2927, 1717, 1513, 1456, 1363, 1261, 1163, 1022, 795 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.36–1.42 (m, 2H), 1.61 (s, 3H), 1.59–1.65 (m, 2H), 2.14 (s, 3H), 2.16 (q, *J* = 7.0 Hz, 2H), 2.32 (s, 3H), 2.44 (t, *J* = 7.5 Hz, 2H), 3.32 (s, 2H), 5.26–5.29 (m, 1H), 7.05 (d, *J* = 7.0 Hz, 2H), 7.09 (d, *J* = 7.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.15, 23.52, 23.69, 28.04, 29.70, 30.04, 37.51, 43.82, 126.21, 128.50, 129.15, 134.47,

135.42, 137.17, 209.36. HRMS (EI) Found: 244.1827 [M^+]; Calcd for $C_{17}H_{24}O$: 244.1827.

(*E*)-9-(2,6-Dimethylphenyl)-8-methyl-7-nonen-2-one ((*E*)-9a): IR (neat) 2930, 1717, 1358, 1163, 769 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.23 (quintet, *J* = 7.5 Hz, 2H), 1.50 (quintet, *J* = 7.5 Hz, 2H), 1.69 (s, 3H), 1.96 (q, *J* = 7.5 Hz, 2H), 2.11 (s, 3H), 2.23 (s, 6H), 2.37 (t, *J* = 7.5 Hz, 2H), 3.27 (s, 2H), 4.63 (dt, *J* = 1.5, 7.5 Hz, 1H), 6.99–7.05 (m, 3H); ^{13}C NMR ($CDCl_3$) δ 17.42, 20.03, 23.62, 27.73, 29.38, 29.95, 38.64, 43.82, 123.35, 125.95, 127.91, 132.47, 136.77, 137.32, 207.45. HRMS (EI) Found: 258.1982 [M^+]; Calcd for $C_{18}H_{26}O$: 258.1984.

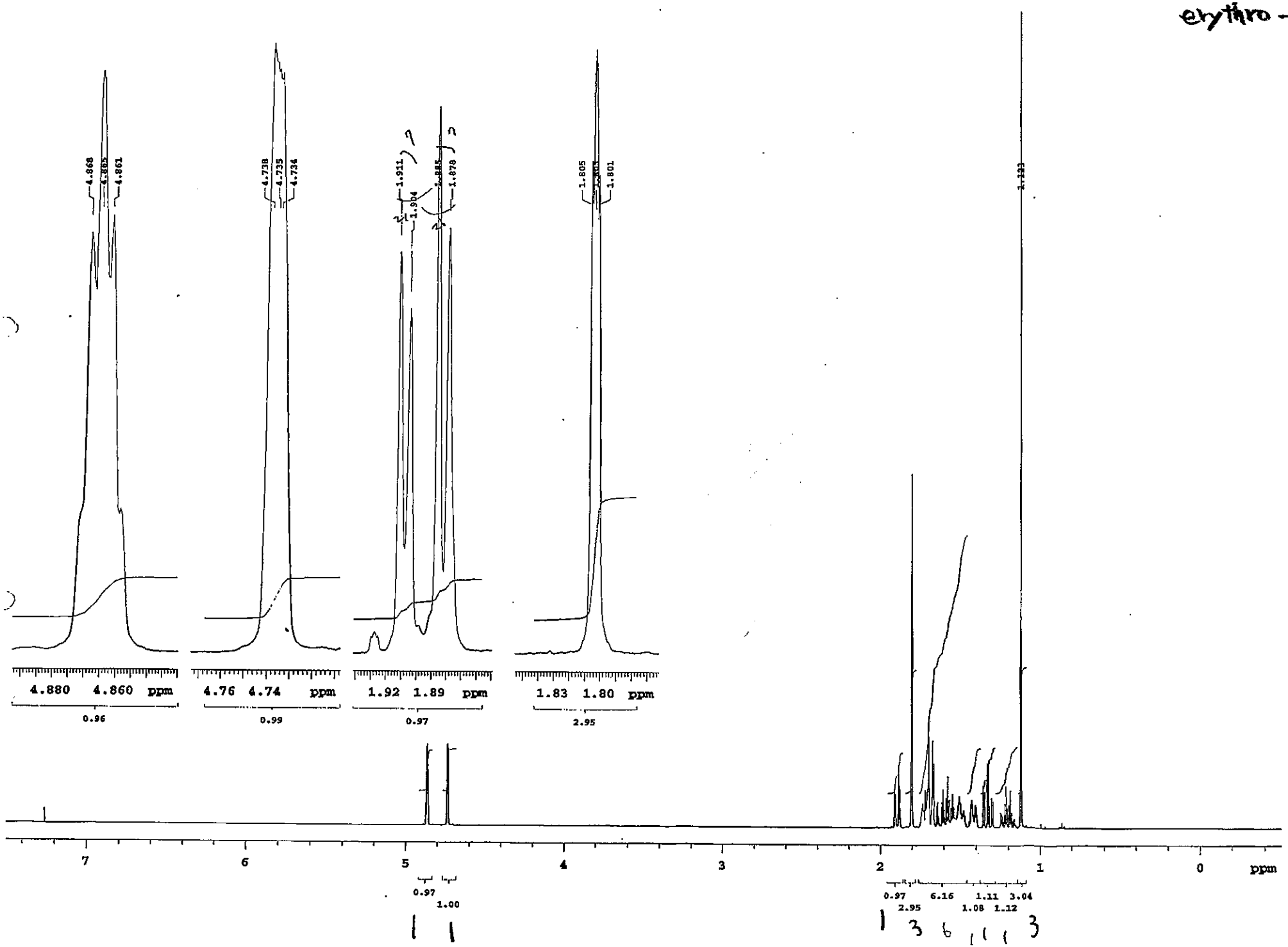
(*E*)-8-Methyl-9-(4-methylphenyl)-7-nonen-2-one ((*E*)-9f): IR (neat) 2927, 1718, 1512, 1360, 1161 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.36 (quintet, *J* = 7.5 Hz, 2H), 1.53 (s, 3H), 1.60 (quintet, *J* = 7.5 Hz, 2H), 2.03 (q, *J* = 7.5 Hz, 2H), 2.14 (s, 3H), 2.32 (s, 3H), 2.43 (t, *J* = 7.5 Hz, 2H), 3.24 (s, 2H), 5.23 (dt, *J* = 1.5, 7.5 Hz, 1H), 7.06 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 15.95, 21.18, 23.67, 27.90, 29.44, 30.02, 43.85, 45.95, 126.08, 128.82, 129.04, 135.07, 135.48, 137.44, 209.47. Found: C, 83.65; H, 10.04%. Calcd for $C_{17}H_{24}O$: C, 83.55; H, 9.90%.

(*Z*)-9-(2,6-Dimethylphenyl)-7-nonen-2-one ((*Z*)-**9g**): IR (neat) 2933, 1718, 1359, 1162, 769 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.40–1.46 (m, 2H), 1.63–1.69 (m, 2H), 2.16 (s, 3H), 2.19–2.24 (m, 2H), 2.30 (s, 6H), 2.47 (t, *J* = 7.5 Hz, 2H), 3.37 (dd, *J* = 6.5, 1.5 Hz, 2H), 5.21–5.27 (m, 1H), 5.37–5.43 (m, 1H), 7.01 (brs, 3H); ^{13}C NMR ($CDCl_3$) δ 20.18, 23.78, 27.51, 28.22, 29.32, 30.04, 43.82, 126.01, 127.55, 128.24, 129.94, 136.45, 138.03, 209.10. Found: C, 83.45; H, 10.02%. Calcd for $C_{17}H_{24}O$: C, 83.55; H, 9.90%.

(*E*)-9-(2,6-Dimethylphenyl)-7-nonen-2-one ((*E*)-**9g**): IR (neat) 2933, 1718, 1452, 1359, 968, 769 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.28–1.34 (m, 2H), 1.50–1.56 (m, 2H), 1.95–2.00 (m, 2H), 2.11 (s, 3H), 2.29 (s, 6H), 2.39 (t, *J* = 7.5 Hz, 2H), 3.32 (dd, *J* = 5.5, 1.0 Hz, 2H), 5.23–5.30 (m, 1H), 5.44–5.49 (m, 1H), 6.99–7.03 (m, 3H); ^{13}C NMR ($CDCl_3$) δ 20.02, 23.53, 29.16, 29.95, 32.41, 32.72, 43.78, 126.02, 127.19, 128.15, 130.49, 136.68, 137.18, 209.23. Found: C, 83.48; H, 10.00%. Calcd for $C_{17}H_{24}O$: C, 83.55; H, 9.90%.

(*Z*)-11-Methyl-12-(2,6-dimethylphenyl)-10-dodecen-5-one ((*Z*)-**9h**): IR (neat) 2933, 1715, 1468, 1377, 768 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.92 (t, *J* = 7.5 Hz, 3H), 1.32 (q, *J* = 7.5 Hz, 2H), 1.36–1.43 (m, 2H), 1.40 (s, 3H), 1.54–1.60 (m, 2H), 1.60–1.66 (m, 2H), 2.14 (q, *J* = 7.0 Hz, 2H), 2.28 (s, 6H), 2.42 (q, *J* = 7.5 Hz, 4H), 3.43 (s, 2H), 5.21–5.24 (m, 1H), 6.98–7.03 (m, 3H); ^{13}C NMR ($CDCl_3$) δ 14.03, 20.51, 22.52, 22.56, 23.86, 26.16, 27.83, 29.53, 31.74, 42.72, 42.89, 125.82, 125.93, 128.13, 133.05, 137.08, 137.19, 211.62. Found: C, 83.74; H, 10.39%. Calcd for $C_{21}H_{32}O$: C, 83.94; H, 10.73%.

erythro-8a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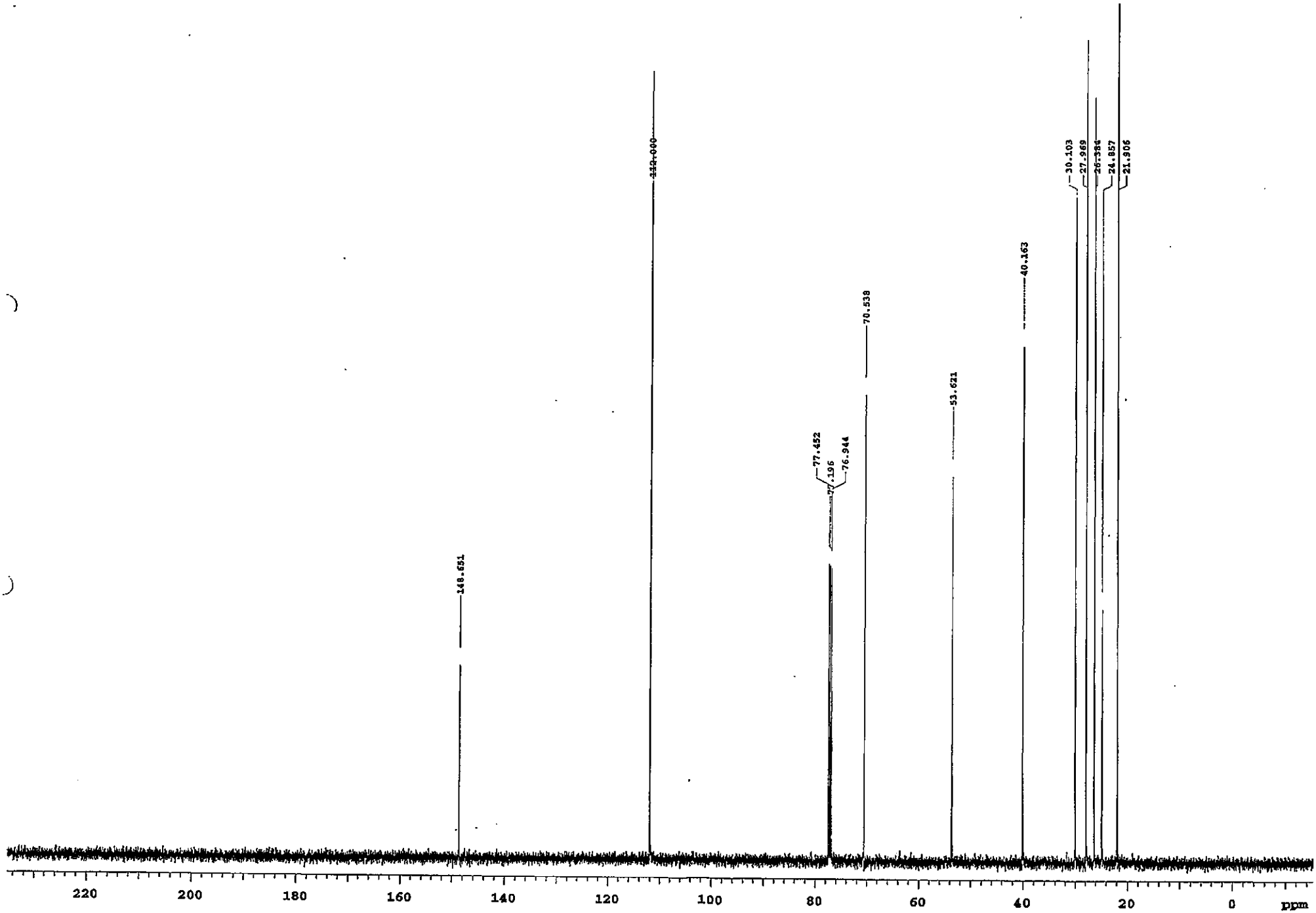
4.865
4.861
4.738
4.735
4.734
1.911
1.904
1.885
1.878
1.805
1.801
1.333
4.880 4.860 ppm
0.96
4.76 4.74 ppm
0.99
1.92 1.89 ppm
0.97
1.83 1.80 ppm
2.95
7
6
5
4
3
2
1
0
ppm
0.97
1.00
0.97
2.95
6.16
1.08
1.11
1.12
3.04

erythro-8a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110.000
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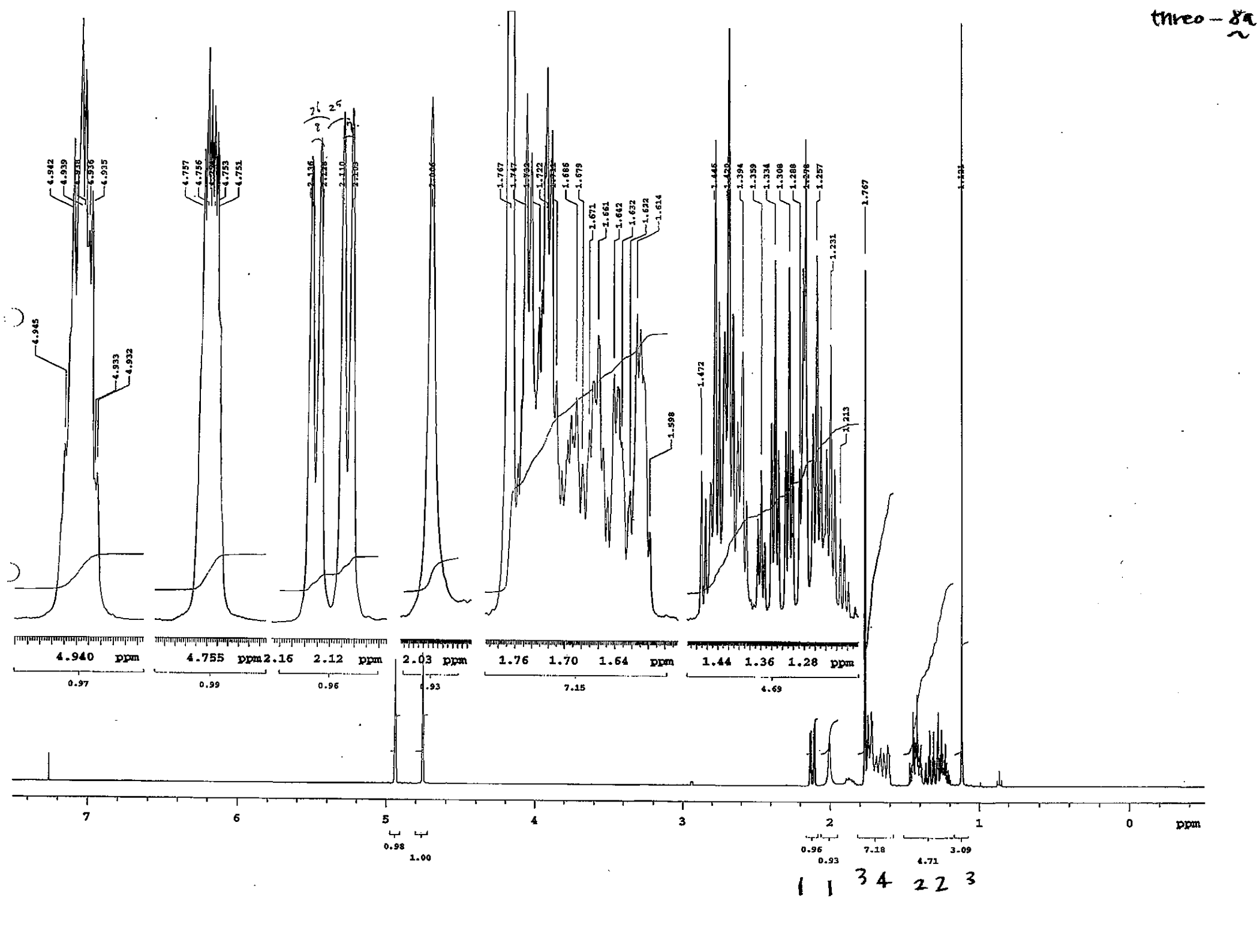
77.196
76.944
70.538
53.621
40.163
30.103
27.969
26.384
24.857
21.906
220
200
180
160
140
120
100
80
60
40
20
0
ppm


threo – 8a
4.942
4.939
4.936
4.935
4.945
4.933
4.932
4.757
4.756
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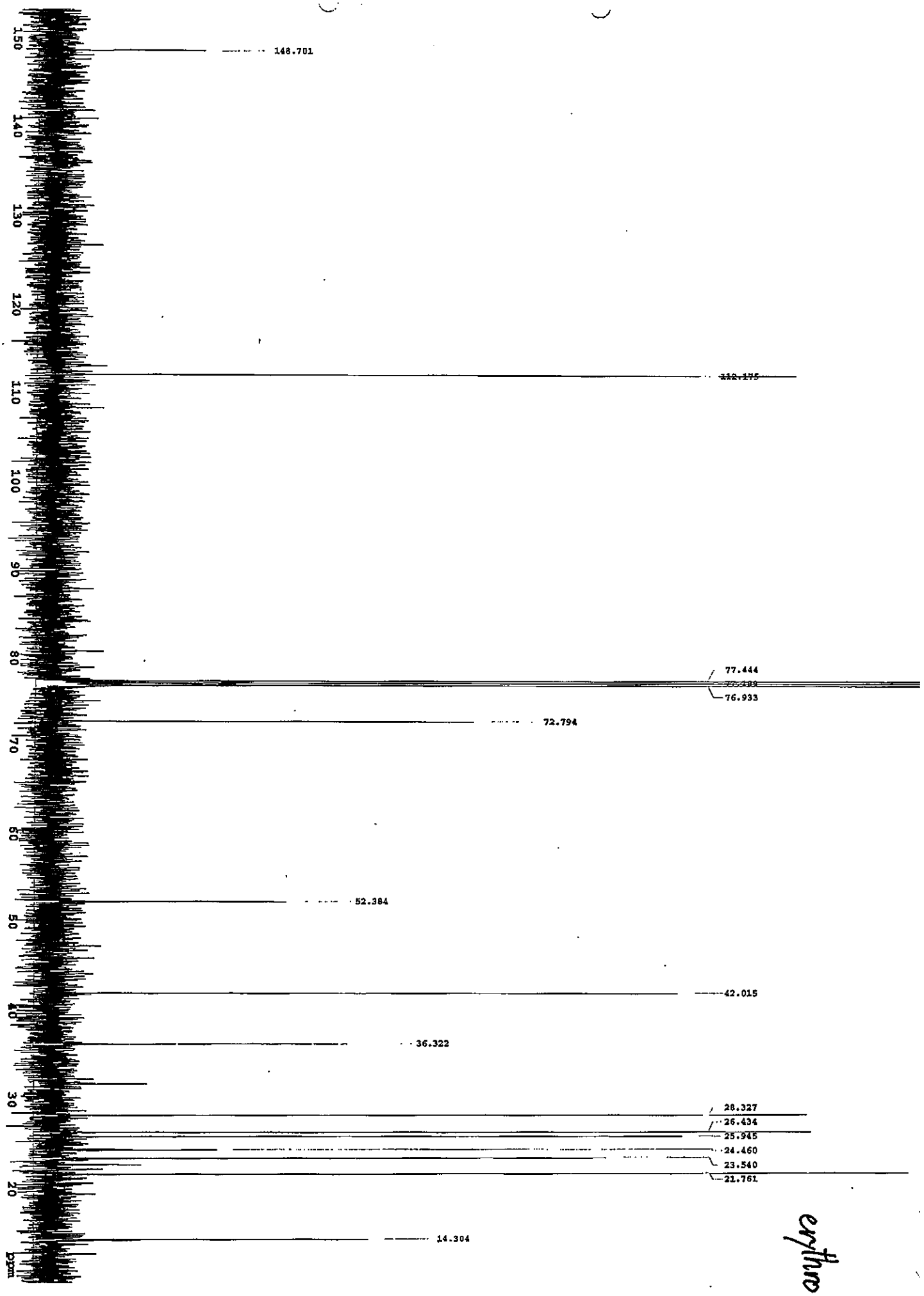
4.751
2.136
2.110
2.066
1.767
1.747
1.722
1.686
1.679
1.671
1.661
1.642
1.632
1.622
1.614
1.598
1.472
1.445
1.420
1.394
1.359
1.334
1.308
1.288
1.257
1.231
1.213
1.767
4.940 ppm
4.755 ppm
2.16 2.12 ppm
2.03 ppm
1.76 1.70 1.64 ppm
1.44 1.36 1.28 ppm
0.97
0.99
0.96
0.93
7.15
4.69
7 6 5 4 3 2 1 0 ppm
0.98
1.00
0.96
0.93
7.18
4.71
3.09
1 1 3 4 2 2 3

160
140
120
100
80
60
40
20
ppm
146.555
114.111
77.452
77.196
76.940
72.290
55.228
41.736
28.744
26.258
24.101
22.914
22.467

three-8a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112.175
77.444
77.189
76.933
72.794
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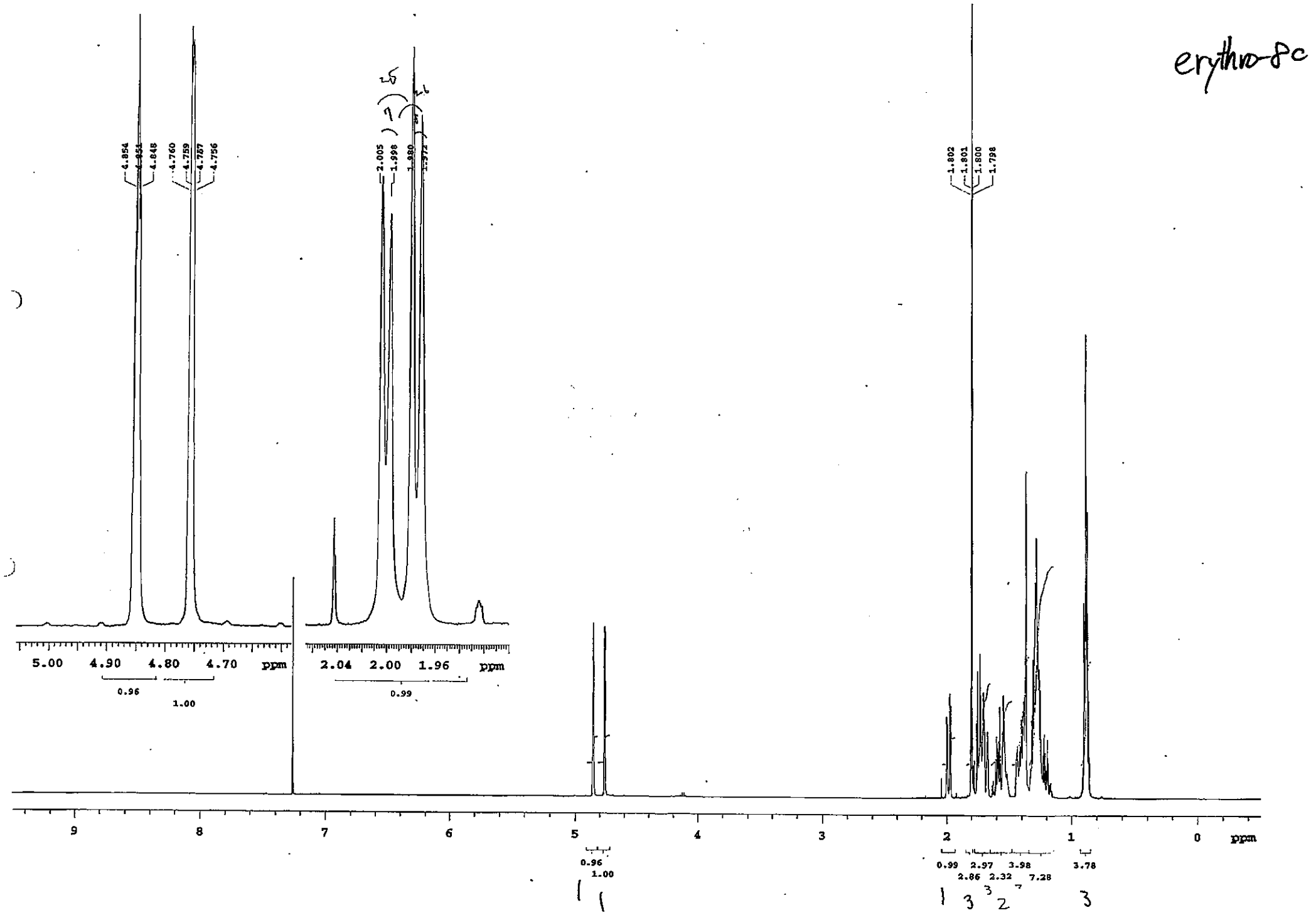
25.945
24.460
23.540
21.761
14.304
ppm

erythro-Pc

erythro-8c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4.851
4.848
4.760
4.759
4.757
4.756
2.005
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1.980
1.972
1.802
1.801
1.800
1.798
5.00
4.90
4.80
4.70
ppm
0.96
1.00
2.04
2.00
1.96
ppm
0.99
9
8
7
6
5
4
3
2
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ppm
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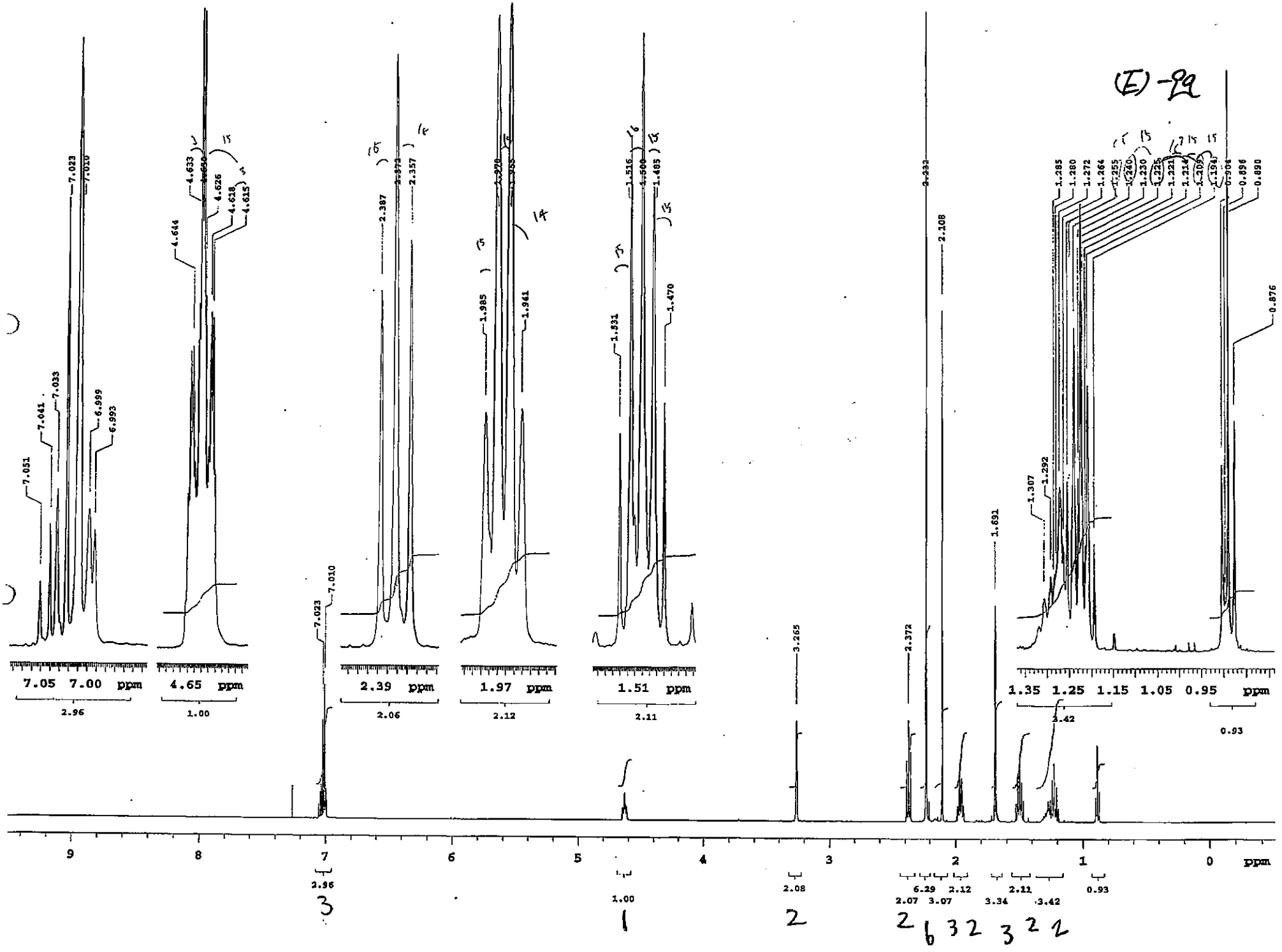
0.99
2.97
3.98
3.78
2.86
2.32
7.28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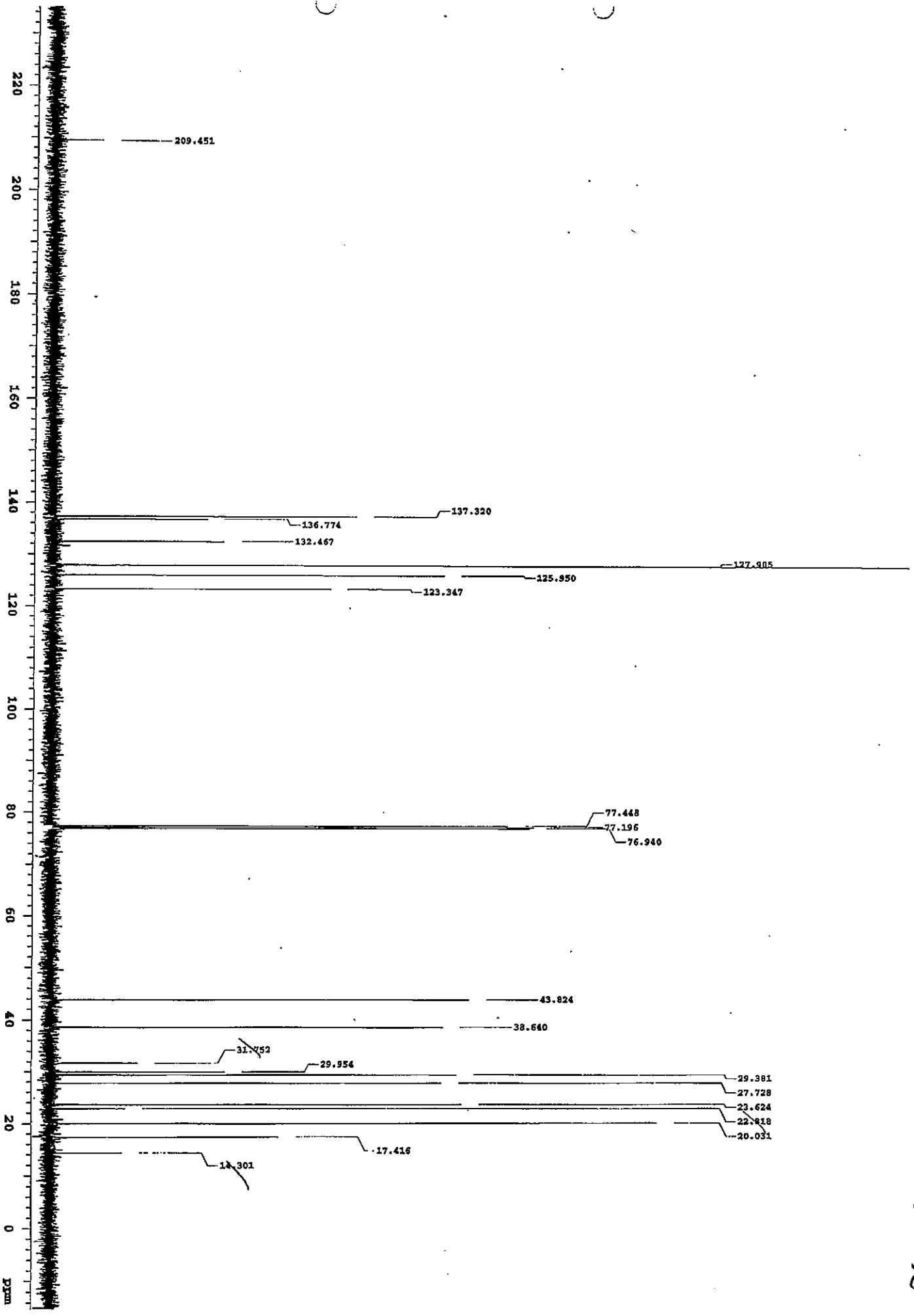
7.041
7.033
7.023
7.010
6.999
6.993
7.05 7.00 ppm
2.96
4.644
4.633
4.626
4.618
4.615
4.65 ppm
1.00
2.387
2.372
2.357
2.39 ppm
2.06
1.985
1.970
1.955
1.941
1.97 ppm
2.12
1.831
1.516
1.500
1.485
1.470
1.51 ppm
2.11
7.023
7.010
3.265
2.372
2.332
2.108
1.691
1.307
1.292
1.285
1.280
1.272
1.264
1.255
1.240
1.230
1.225
1.221
1.214
1.205
1.194
0.904
0.896
0.890
0.876
1.35 1.25 1.15 1.05 0.95 ppm
3.42
0.93
9 8 7 6 5 4 3 2 1 0 ppm
2.96
1.00
2.08
2.07 3.07 2.12 3.34 2.11 3.42 0.93
3
1
2
2 6 3 2 3 2 2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140
120
100
80
60
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20
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ppm
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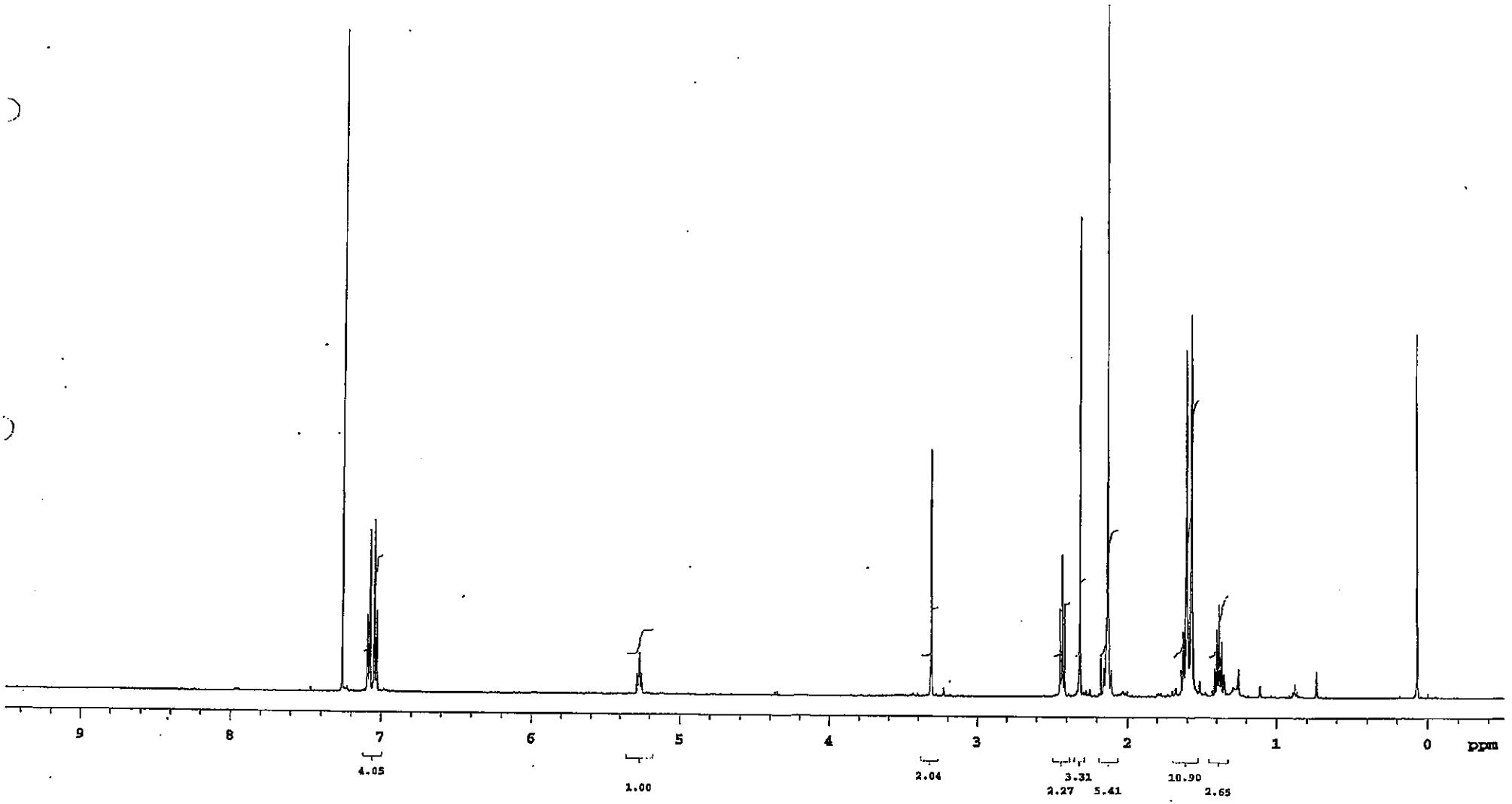
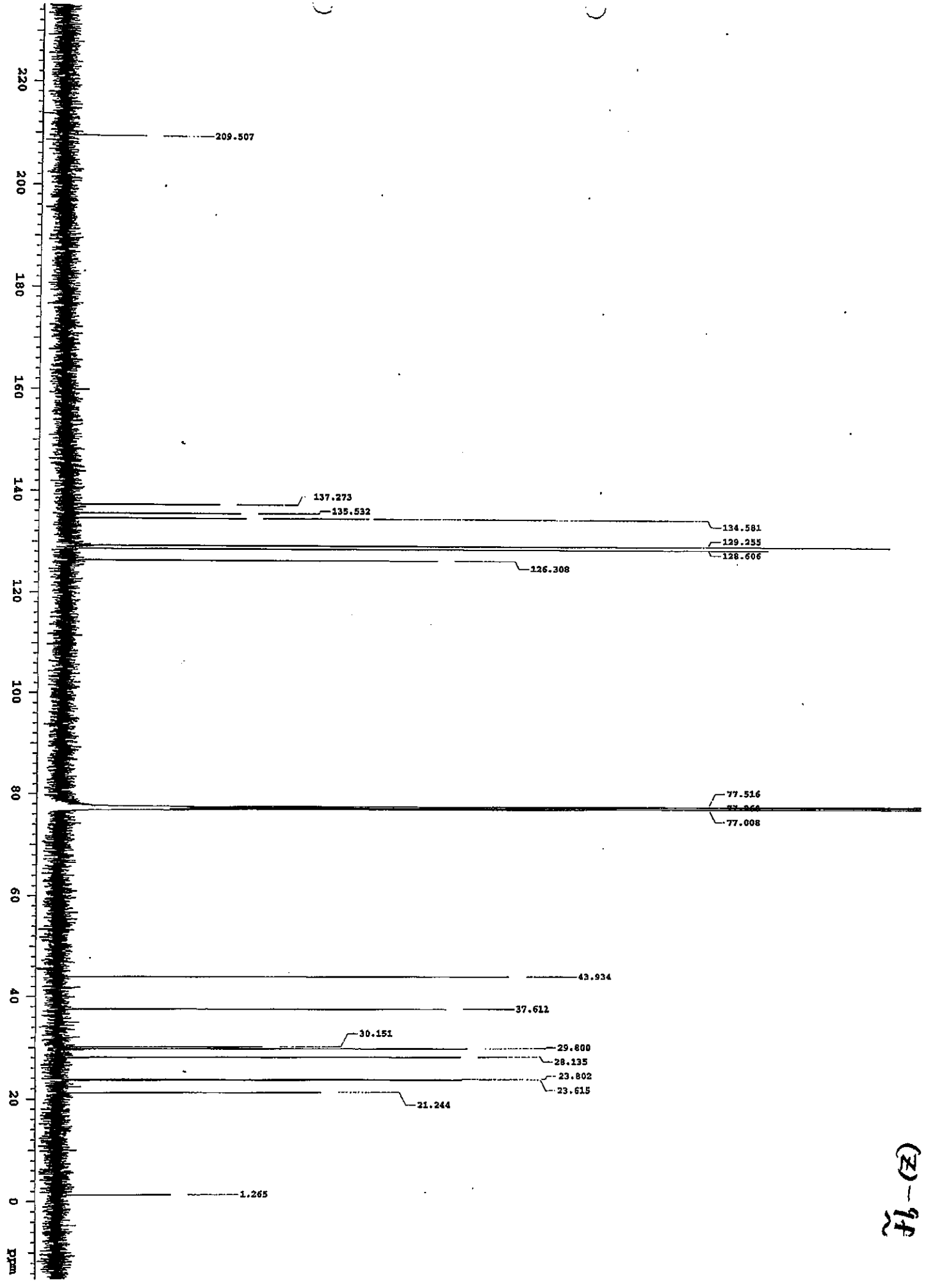
125.950
123.347
77.448
77.196
76.940
43.824
38.640
31.752
29.954
29.381
27.728
23.624
22.818
20.031
17.416
14.301

(E)-9a

(Z)-9f

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180
160
140
120
100
80
60
40
20
0
ppm
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137.273
135.532
134.581
129.255
128.606
126.308
77.516
77.008
43.934
37.612
30.151
29.800
28.135
23.802
23.615
21.244
1.265

(Z) - 14