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of Reliable Methods
for the Preparation
of Organic Compounds

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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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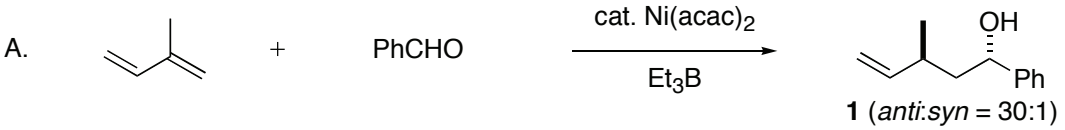
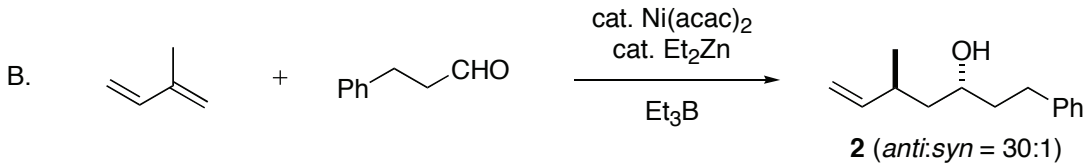
NICKEL-CATALYZED HOMOALLYLATION OF ALDEHYDES WITH 1,3-DIENES (*anti*-3-Methyl-1-phenyl-4-penten-1-ol and *anti*-5-Methyl-1-phenyl-6-hepten-3-ol)





Submitted by Yoshinao Tamaru[1a] and Masanari Kimura.[1b]
Checked by Masakatsu Shibasaki and Noriyuki Yamagiwa.
Discussion Addendum *Org. Synth.* **2013**, *90*, 105

1. Procedure

*A. 3-Methyl-1-pheny-4-penten-1-ol (**1**).*[2] An oven-dried, 300-mL, two-necked round-bottomed flask, equipped with a rubber septum, an air condenser (fitted with a three-way stopcock connected to a nitrogen balloon), and a Teflon-coated magnetic stir bar is charged with $Ni(acac)_2$ (180 mg, 0.7 mmol) (Note 1). The apparatus is purged with nitrogen and the flask is charged successively via syringe with freshly distilled THF (100 mL), isoprene (5 mL, 50 mmol), and benzaldehyde (2.65 g, 25 mmol) (Note 2). Triethylborane (50 mL, 1 M hexane solution; 50 mmol) (Note 3) is added via syringe over 5 min while the solution is stirred with a magnetic stirrer. Stirring is continued for 30 h at ambient temperature. After the reaction is completed (Note 4), most of the solvents and the remaining reagents (isoprene and Et_3B) are removed with a rotary evaporator. The residual mixture is diluted with diethyl ether (60 mL). The organic phase is washed with 4 M KOH (2 x 50 mL) and brine (2 x 30 mL), and then dried over magnesium sulfate, filtered, and concentrated by rotary evaporation (Note 5). The residue is distilled by means of a Kugelrohr apparatus (90–100 °C/0.03

mmHg) to give 3-methyl-1-phenyl-4-penten-3-ol (**1**) (3.89−4.05 g, 88−92%) consisting of *anti*- and *syn*-isomers in a ratio of 30:1 (Note 6).

*B. 5-Methyl-1-phenyl-6-hepten-3-ol (**2**)*.[3] An oven-dried, 300-mL, two-necked round-bottomed flask equipped with a rubber septum, an air condenser (fitted with a three-way stopcock connected to a nitrogen balloon), and a Teflon-coated magnetic stir bar is charged with $Ni(acac)_2$ (192 mg, 0.75 mmol). The apparatus is purged with nitrogen and the flask is charged with freshly distilled THF (100 mL). Diethylzinc (0.75 mL, 1 M hexane solution; 0.75 mmol) is added into this solution at ambient temperature (Note 7). The color of the reaction mixture changes from yellow-green to brown. Isoprene (7.5 mL, 75 mmol) and dihydrocinnamaldehyde (3.4 g, 25 mmol) are added by syringe. Triethylborane (60 mL, 1 M hexane solution; 60 mmol) is then added via syringe over 5 min while stirring the solution with a magnetic stirrer (Note 3). Stirring is continued for 8 h at ambient temperature. After the reaction is complete (Note 8), most of the solvents and the remaining reagents (isoprene and Et_3B) are removed with a rotary evaporator. The residual solution is diluted with diethyl ether (60 mL). The organic phase is washed with 4 M KOH (2 x 40 mL) and brine (2 x 30 mL), and then dried over magnesium sulfate, filtered, and concentrated by rotary evaporation. The residue is distilled by means of Kugelrohr apparatus under reduced pressure (100–120 °C/0.03 mmHg) to give 5-methyl-1-phenyl-6-hepten-3-ol (**2**) (3.50–3.59 g, 68–70%) (Note 9) consisting of the *anti*- and *syn*-isomers in a ratio of 30:1 (Note 10).

2. Notes

1. $Ni(acac)_2$ is used as received from Aldrich Chemical Company, Inc.

2. THF **(**tetrahydrofuran) is distilled from sodium/benzophenone ketyl under N_2 prior to use. Benzaldehyde was used as received from Wako Pure Chemical Industry, Ltd.

3. Et_3B (1 M hexane solution) is used as received from Aldrich Chemical Company, Inc. Hydrocinnamaldehyde was used as received from Wako Pure Chemical Industry, Ltd.

4. The reaction is monitored by TLC (Merck, Silica gel 60F254). R_f

(**1**) = 0.67; R_f (benzaldehyde) = 0.72: hexane/ethyl acetate = 2/1, v/v.: visualized by a 254-nm UV lamp as well as by iodine.

5. It is crucial to wash the organic phase with a strong base, such as 4 M KOH prior to distillation. The concentrated residue obtained by washing with sat. $NaHCO_3$ did not withstand distillation (100 °C/0.05 mmHg) and decomposed to give an intractable mixture of products.

6. ***anti*-3-Methyl-1-phenyl-4-penten-1-ol (*anti*-1)**: IR (neat) 3357 (s), 2960 (s), 2926 (s), 1494 (s), 1454 (s), 997 (s), 911 (s), 756 (s), 700 (s) cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ: 1.01 (d, J = 6.7 Hz, 3 H), 1.63 (ddd, J = 13.7, 6.1, 6.1 Hz, 1 H), 1.83 (ddd, J = 13.7, 7.9, 7.9 Hz, 1 H), 1.89 (brs, 1 H), 2.20 (ddt, J = 6.1, 7.9, 6.7 Hz, 1 H), 4.71 (dd, J = 6.1, 7.9 Hz, 1 H), 4.96 (brd, J = 10.5 Hz, 1 H), 5.00 (brd, J = 17.7 Hz, 1 H), 5.77 (ddd, J = 7.9, 10.5, 17.7 Hz, 1 H), 7.25 - 7.34 (m, 5 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 20.4, 35.2, 45.9, 73.0, 113.2, 126.0, 127.6, 128.5, 144.5, 144.6. Anal. calcd. for $C_{12}H_{16}O$: C, 81.77; H, 9.15. Found: C, 81.21; H, 9.21. ***syn*-3-Methyl-1-phenyl-4-penten-1-ol (*syn*-1)** was prepared from the *anti*-isomer by Mitsunobu reactions for reference (see discussion): IR (neat) 3356 (s), 1640 (w), 1043 (s), 912 (s), 700 (s) cm^{-1}m; ^1H NMR (400 MHz, $CDCl_3$) δ: 1.05 (d, J = 6.8 Hz, 3 H), 1.61 (ddd, J = 3.6, 9.3, 13.9 Hz, 1 H), 1.80 (ddd, J = 4.7, 9.3, 13.9 Hz, 1 H), 1.83 (d, J = 3.6 Hz, 1 H), 2.42 (dqm, J = 8.0, 6.8 Hz, 1 H), 4.72 (dt, J = 9.3, 3.6 Hz, 1 H), 5.01 (br d, J = 10.0 Hz, 1 H), 5.06 (br d, J = 17.1 Hz, 1 H), 5.73 (ddd, J = 8.0, 10.0, 17.1 Hz, 1 H), 7.23 - 7.38 (m, 5 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 21.0, 34.9, 46.1, 72.4, 113.7, 125.7, 127.4, 128.4, 143.8, 145.0. Anal. calcd. for $C_{12}H_{16}O$: C, 81.77; H, 9.15. Found: C, 81.21; H, 9.21. The diastereomeric ratio (*anti*-**1**/*syn*-**1**) was determined from the ratio of resonance integrations at 5.77 ppm (*anti*) and 5.73 ppm (*syn*) in ^1H-NMR spectra.

7. Et_2Zn (1 M hexane solution) is used as received from Aldrich Chemical Company, Inc.

8. The reaction is monitored with TLC (Merck, Silica gel 60F254). R_f (**2**)= 0.58: R_f (hydrocinnamaldehyde) = 0.50: hexane/ethyl acetate = 2/1, v/v.: visualized by a 254-nm UV lamp as well as by iodine.

9. The submitters report an isolated yield of 80% yield.

10. ***anti*-5-Methyl-1-phenyl-6-hepten-3-ol** (***anti*-2**): IR (neat) 3373

(s), 2927 (s), 1639 (s), 1603 (s), 1495 (s), 1454 (s), 913 (s), 699 (s) cm^{-1};^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.99 (d, J = 6.9 Hz, 3 H), 1.42 (ddd, J = 4.0, 6.9, 13.8 Hz, 1 H), 1.52 (ddd, J = 5.8, 9.8, 13.8 Hz, 1 H), 1.65 (brs, 1 H), 1.68-1.81 (m, 2 H), 2.28-2.34 (m, 1 H), 2.65 (ddd, J = 6.3, 9.8, 13.8 Hz, 1 H), 2.78 (ddd, J = 5.8, 9.8, 13.8 Hz, 1 H), 3.68-3.73 (m, 1 H), 4.92 (dd, J = 1.7, 10.3 Hz, 1 H), 5.00 (brd, J = 1.7, 17.2 Hz, 1 H), 5.76 (ddd, J = 8.0, 10.3, 17.2 Hz, 1 H), 7.16-7.29 (m, 5 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 20.3, 31.9, 35.6, 39.3, 44.5, 70.0, 112.9, 125.8, 128.4, 128.4, 142.2, 145.0. Anal. calcd. for $C_{14}H_{20}O$: C, 82.30; H, 9.87. Found: C, 82.03; H, 9.58. ***syn*-5-Methyl-1-phenyl-6-hepten-3-ol** (***syn*-2**) was prepared from the anti-isomer by Mitsunobu reactions for reference (see discussion): IR (neat) 3354 (s), 1641 (m), 1030 (m), 997 (s), 914 (s), 698 (s) cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.02 (d, J = 6.4 Hz, 3 H), 1.43 (dm, J = 13.9 Hz, 1 H), 1.50 (dm, J = 13.9 Hz, 1 H), 1.70-1.78 (m, 2 H), 2.40 (dqm, J = 8.0, 6.4 Hz, 1 H), 2.66 (dm, J = 13.9 Hz, 1 H), 2.78 (dm, J = 13.9 Hz, 1 H), 3.68 (br s, 1 H), 4.96 (br d, J = 10.1 Hz, 1 H), 5.03 (d, J = 17.2 Hz, 1 H), 5.67 (ddd, J = 8.0, 10.1, 17.2 Hz, 1 H), 7.15-7.29 (m, 5 H); ^{13}C NMR (120 MHz, $CDCl_3$) δ: 21.1, 32.1, 34.8, 39.6, 44.3, 69.3, 113.3, 125.6, 128.2, 142.0, 143.9; Anal. calcd. for $C_{14}H_{20}O$: C, 82.30; H, 9.87. Found: C, 82.03; H, 9.58. The diastereomeric ratio (*anti*-**2**/*syn*-**2**) was determined from the ratio of resonance integrations at 5.76 ppm (*anti*) and 5.67 ppm (*syn*) in ^{1}H-NMR spectra.

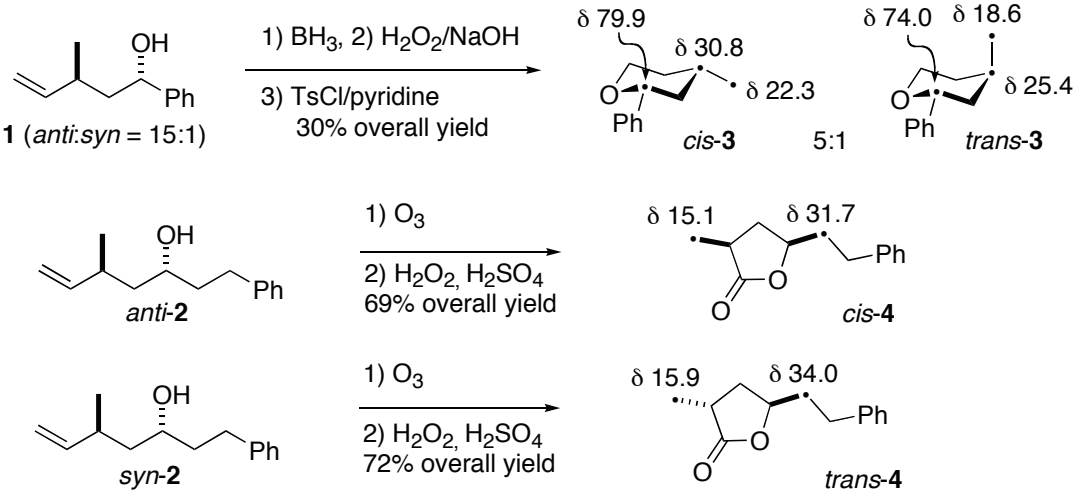
Safety and Waste Disposal Information

All hazardous materials should be handled and disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

The structures of the minor components of **1** and **2** were confirmed to be the diastereomeric isomers, *syn*-**1** and *syn*-**2**, respectively, by comparison of the spectral data with those of authentic samples prepared by the Mitsunobu

inversion ($PhCO_2H$, diethyl azodicarboxylate, Ph_3P) followed by hydrolysis (NaOH, EtOH-H_2O): *anti*-**1**:*syn*-**1** = 1:30 in 79% overall yield from *anti*-**1**:*syn*-**1** = 30:1; *anti*-**3**:*syn*-**3** = 1:30 in 72% overall yield from *anti*-**3**:*syn*-**3** = 30:1. The structure determination of **1** and **2**[4] relied on the ^{13}C NMR chemical shifts of their cyclic derivatives **3** and **4**, respectively (Scheme 1).



Scheme 1

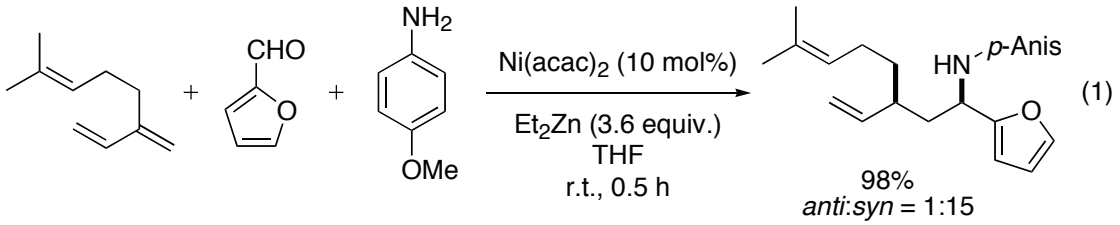
Transformation of **1** and **2** into Heterocycles **3** and **4** and their ^{13}C Chemical Shifts (δ in ppm, 100 MHz in $CDCl_3$).

Bishomoallyl alcohols and ethers are ubiquitous structural motifs of natural products, especially of polyether antibiotics and acetogenin-derived natural products.[5] The present nickel-catalyzed homoallylation of aldehydes with isoprene (and other 1,3-dienes)[6] provides a powerful method for constructing such classes of compounds.[7] The reaction shows high 1,3-diastereoselectivity furnishing 1,3-*anti*-4-penten-1-ol products with as high as 95 – 100% stereoselectivity.

Three variations on the homoallylation procedure are available and appropriate choice of the method to be applied requires consideration of the reactivity of the aldehyde. For reactive aldehydes (aromatic and unsaturated aldehydes), procedure A is the method of choice.[2] A second (unchecked) procedure is particularly effective for the homoallylation of relatively unreactive and sterically hindered aliphatic aldehydes, as well as ketones.[3] This second procedure is sometimes plagued with a side reaction resulting

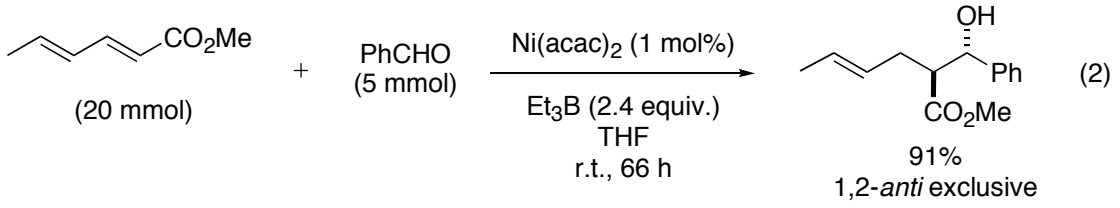
from ethylation of the aldehyde. This problem is most apparent when reactive primary and secondary aliphatic aldehydes are used. For such cases, procedure B is recommended, because ethylation can be avoided almost completely. Thus, these three methods complement one another.

A variation on these procedures has been successfully applied to the homoallylation of aldimines formed *in situ* from aromatic aldehydes and anisidine. Good yields of 1,3-*syn*-bishomoallyl amines are furnished with excellent stereoselectivity. Aldimines are generally by far less reactive than aldehydes. Interestingly, the sense of stereoselection is opposite to that observed for aldehydes (Eq 1).[8]



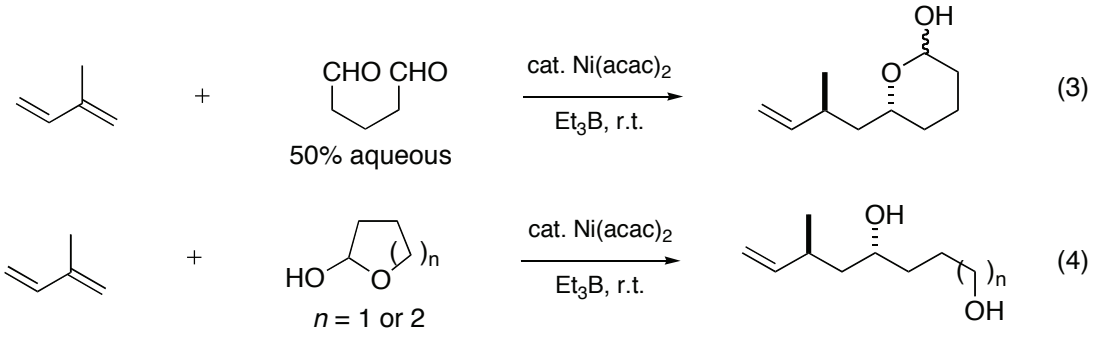
(1)

Sorbic acid methyl ester, an electron-deficient diene, engages by (Eq 2) reacting regioselectively at the C2 position with high 1,2-*anti* stereoselectivity. All results to date suggest that unsymmetrically substituted 1,3-dienes react with aldehydes regioselectively at the terminus bearing the highest electron density.

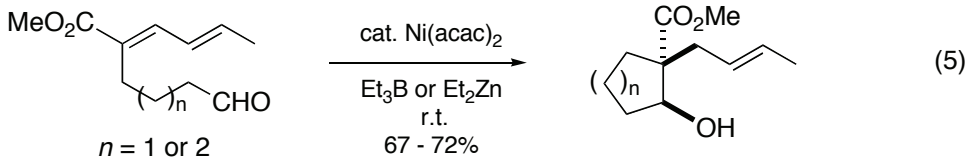


(2)

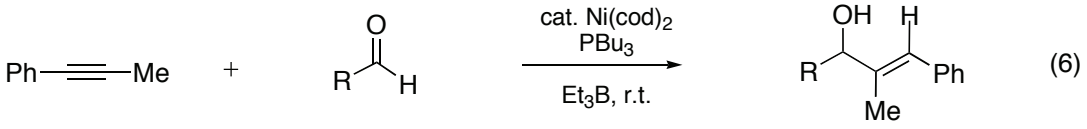
Triethyl borane (Et_3B) is compatible with water; hence, the method described in procedure A can be applied to the homoallylation of aldehydes that are stable only in water.[9] For example, in the presence of a catalytic amount of $Ni(acac)_2$ and a stoichiometric amount of Et_3B, isoprene reacts regioselectively at the C1 position with a commercial 50% aqueous solution of glutaraldehyde to provide a 1,3-*anti*-bishomoallyl alcohol. Cyclic hemiacetals (n = 1 or 2) undergo the homoallylation with isoprene to form bishomoallylic diols with excellent 1,3-asymmetric induction (Eq 4).



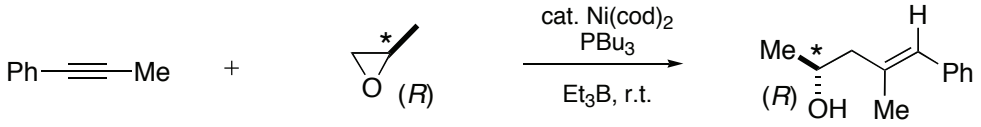
The protocol for the intermolecular homoallylation of aldehydes with 1,3-dienes can be applied successfully to an intramolecular version of ω-dienyl aldehydes forming five- and six-membered cycloalkanols (Eq 5).[10]



The combination of nickel and Et_3B has been proved to be applicable to the reductive coupling of alkynes and aldehydes to provide allylic alcohols with high regio- and stereoselectivities (Eq 6).[11] The procedure is extended to the asymmetric synthesis of allyl alcohols and is applied to the total synthesis of natural products.[12] Under similar conditions, enantioenriched epoxides undergo the reductive coupling with alkynes and provide homoallyl alcohols with complete preservation of the original chirality (Eq 7).[13] In contrast to the reactions with aldehydes and epoxides, the reaction with aldimines delivers the ethyl group of Et_3B, instead of hydrogen, at the distal carbon of alkynes to give rise to stereochemically defined allylamines (Eq 8).[14]



(6)



(7)

(8)

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6. We have noticed that 1,3-cyclohexadiene is unusual and selectively undergoes allylation (not homoallylation) under the conditions used in reference 3. Accordingly, the report that claims 1,3-cyclohexadiene selectively undergoes homoallylation of aldehydes should be

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Appendix
Chemical Abstracts Nomenclature (Registry Number)

Diethylzinc (1.0 M solution in hexanes): FLAMMABLE LIQUID; Zinc, diethyl; (557-20-0)

Triethylborane (1.0M solution in hexanes): FLAMMABLE LIQUID; Borane, triethyl; (97-94-9)

Nickel(II) acetylacetonate: CANCER SUSPECT AGENT:

Bis(2,4-pentanedionate), nickel(II); (3264-82-2)

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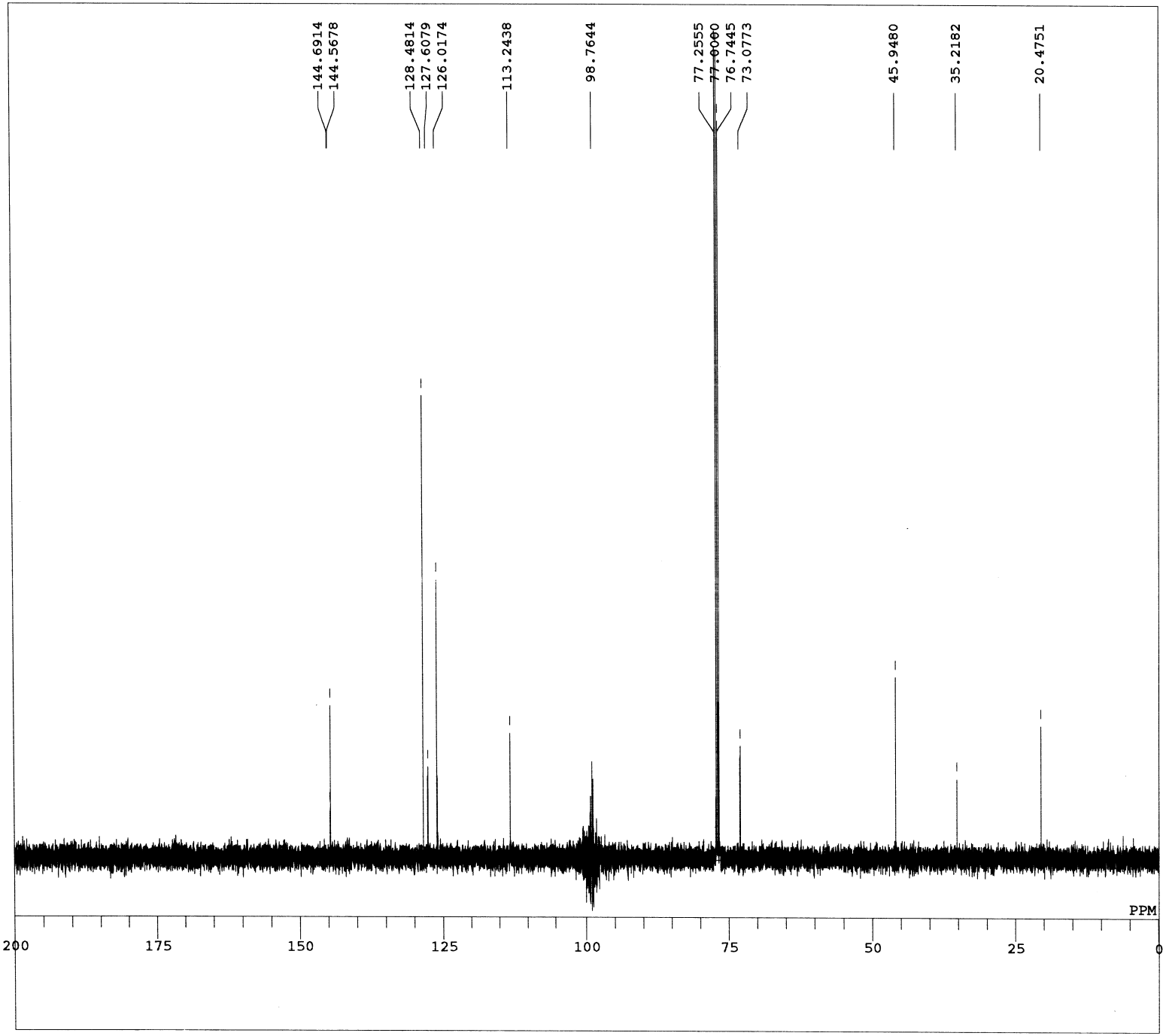

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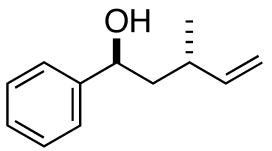

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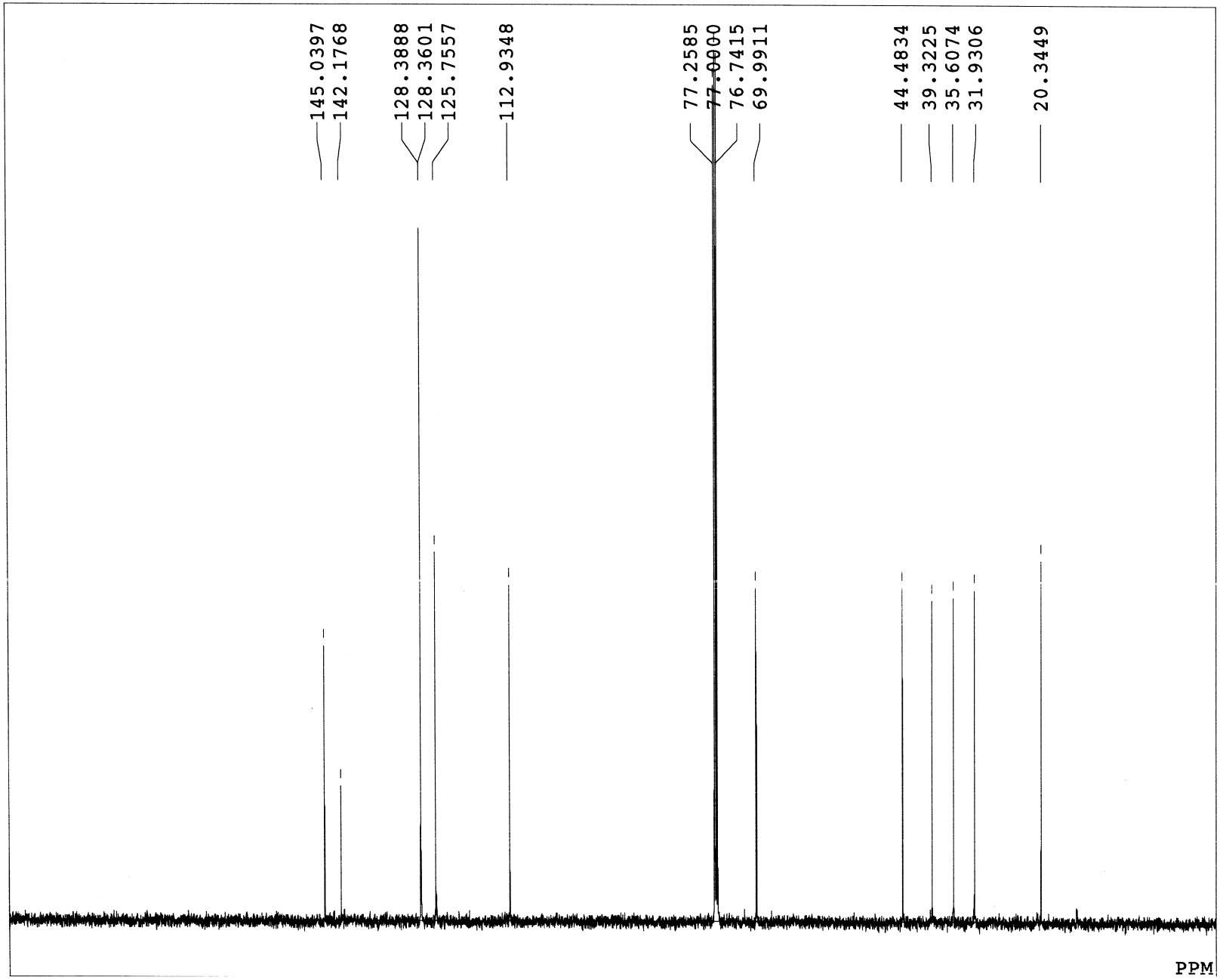

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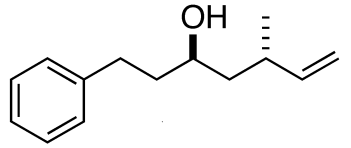
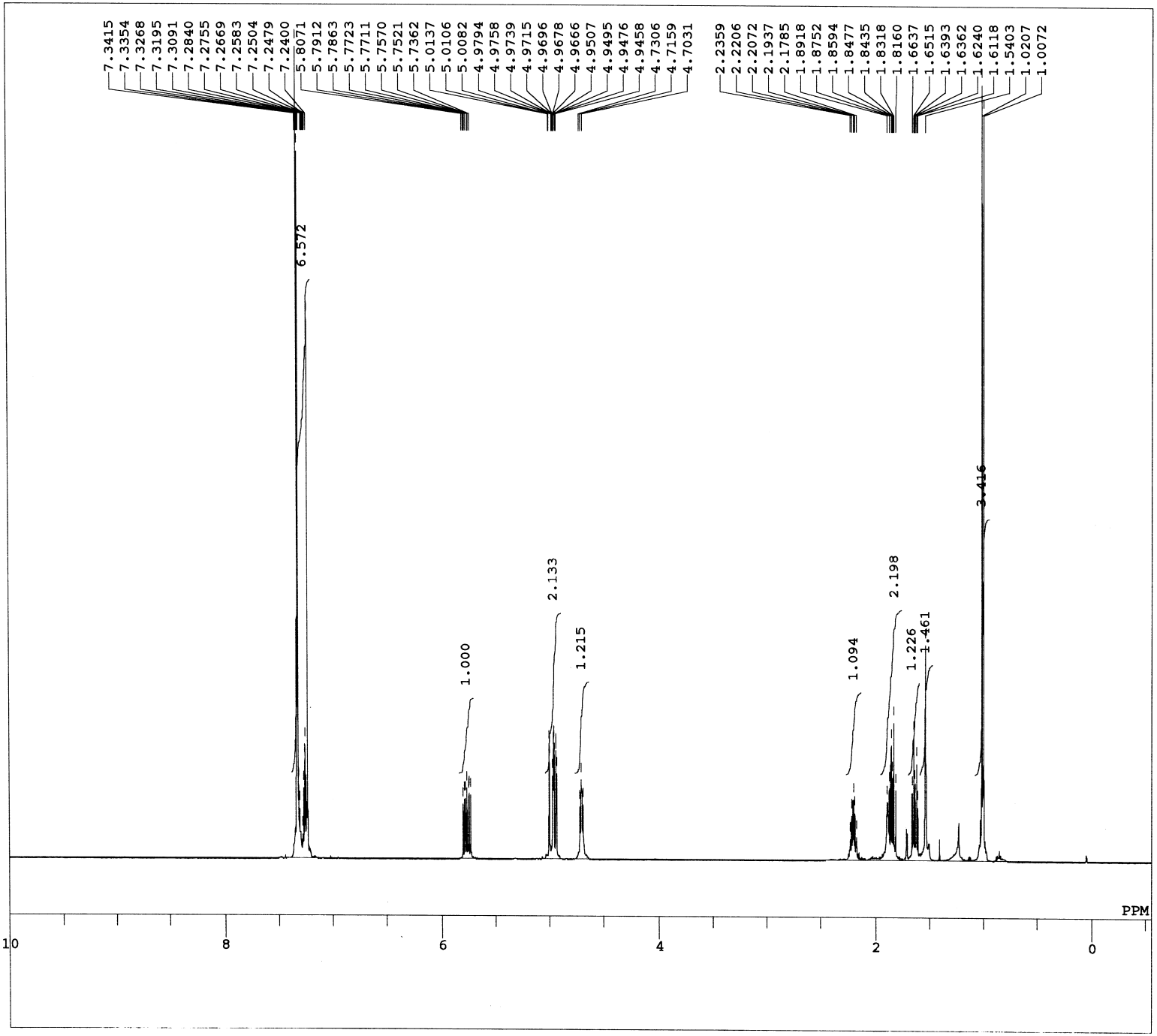
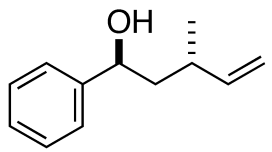
BF	0.12 Hz
RGAIN	58



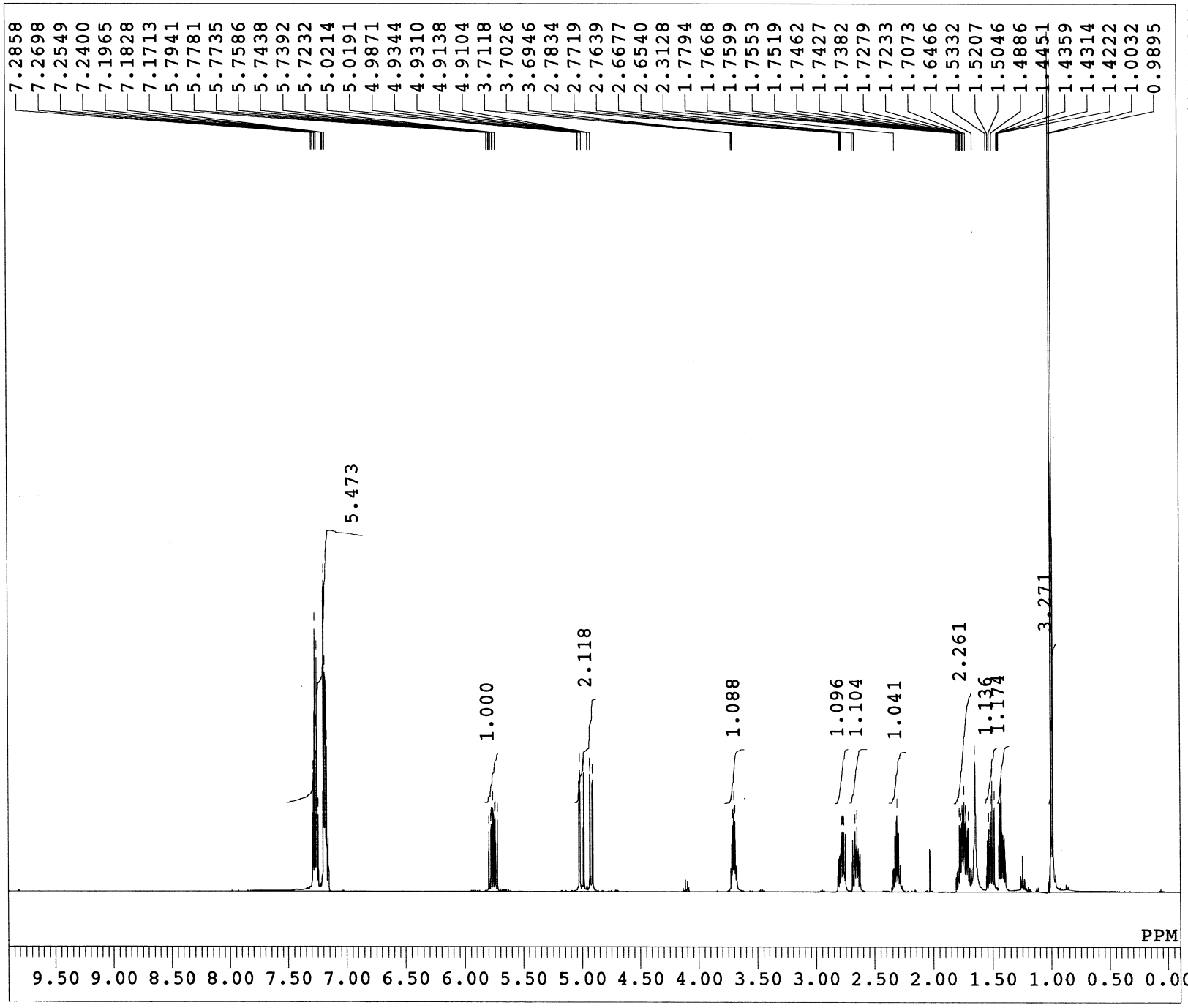
C:\My Documents\PGousei\Giwayama\giwa\giwa.nmfid
20051008



DFILE	C:\My Documents\PGousei\Giwayama\giwa\gi
COMNT	20051008
DATIM	Sun Oct 9 11:25:15 2005
OBNUC	1H
EXMOD	non
OBFRQ	499.10 MHz
OBSET	120.00 KHz
OBFIN	8108.5 Hz
POINT	32768
FREQU	10000.0 Hz
SCANS	32
ACQTM	3.277 sec
PD	3.723 sec
PW1	6.2 us
IRNUC	1H
CTEMP	23.9 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	23



C:\My Documents\PGousei\Giwayama\OS0929.1
OS0929



DFILE	C:\My Documents\PG
COMNT	OS0929
DATIM	29-09-2005 21:43:3
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	495.13 MHz
OBSET	4.38 KHz
OBFIN	9.6 Hz
POINT	16384
FREQU	9286.8 Hz
SCANS	8
ACQTM	1.764 sec
PD	5.000 sec
PW1	6.2 us
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	42