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of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

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In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

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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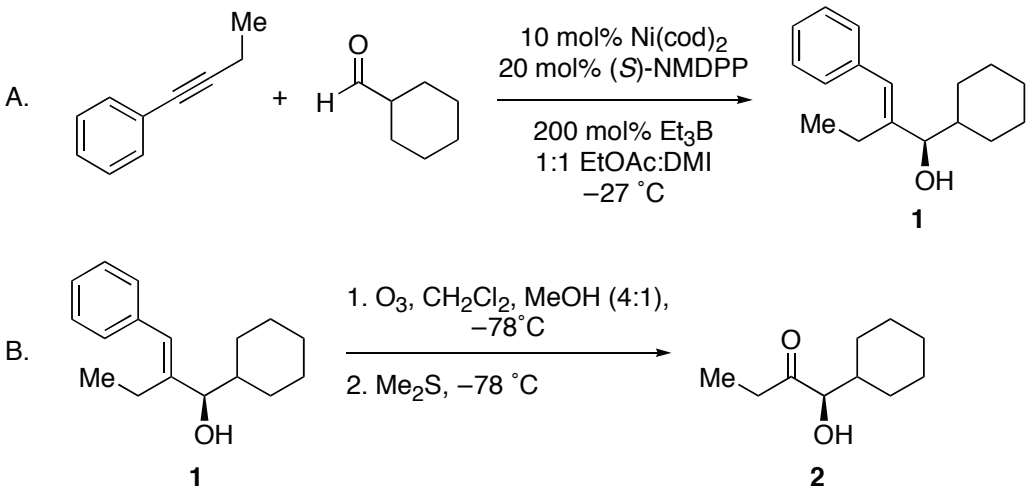
(*S*)-(+)-NEOMENTHYLDIPHENYLPHOSPHINE IN NICKEL-CATALYZED ASYMMETRIC REDUCTIVE COUPLING OF ALKYNES AND ALDEHYDES: ENANTIOSELECTIVE SYNTHESIS OF ALLYLIC ALCOHOLS AND α-HYDROXY KETONES

[(*R*)-Butan-2-one, 1-cyclohexyl, 1-hydroxy-]



Submitted by Aaron R. Van Dyke, Karen M. Miller, and Timothy F. Jamison.[1]
Checked by Matthew J. Fleming and Mark Lautens.

1. Procedure

Caution! Ozone is extremely toxic and can react explosively with certain oxidizable substances. Ozone also reacts with some compounds to form explosive and shock-sensitive products. Ozone should only be handled by individuals trained in its proper and safe use and all operations should be carried out in a well-ventilated fume hood behind a protective safety shield. [Note added September 2009].

A. (R)-(E)-2-Benzylidiene-1-cyclohexyl-butan-1-ol. In a glovebox, a flame-dried 500-mL round-bottomed flask is charged with $Ni(cod)_2$ (1.38 g, 5.00 mmol) (Note 1) and (*S*)-(+)-neomenthyldiphenylphosphine (3.24 g, 10.0 mmol) (Note 2). The flask is sealed with a septum, removed from the glovebox, and transferred to a fume-hood. An argon inlet (Note 3) is then attached to the flask and degassed EtOAc (50 mL) (Note 4), freshly distilled, degassed DMI (50 mL) (Note 5), and triethylborane (14.5 mL, 100 mmol)

(Note 6) are added sequentially via syringe. *Caution! Triethylborane is extremely pyrophoric*. The solution is allowed to stir 15 min at room temperature and then placed in a –27 °C bath (Note 7) for 30 min. 1-Phenyl-1-butyne (7.1 mL, 50 mmol) (Note 8) is added in one portion via syringe followed by addition of cyclohexanecarboxaldehyde (9.1 mL, 75 mmol) (Note 9) via syringe pump over 9 h to the solution in a –27 °C bath. The reaction is stirred at –27 °C for 36 h, then quenched with saturated NH_4Cl (100 mL) (Note 10), 1M HCl (40 mL) at –27 °C. The solution is allowed to warm to room temperature and then stirred 15 min at room temperature. Air is vigorously bubbled through the reaction using a pasteur pipet for 30 min, which results in a light yellow emulsion (Note 11). The mixture is extracted with EtOAc (2 x 200 mL). The combined organic layers are washed twice with saturated NH_4Cl (300 mL), once with brine (300 mL) (Note 12), dried over $MgSO_4$ (10 g) (Note 13), filtered and concentrated on a rotary evaporator (20 °C, 11 mmHg, then 3 mmHg) to remove trace EtOAc. The resulting yellow oil is purified by flash chromatography on silica gel with a hexanes to 9:1 hexanes: ethyl acetate gradient (Note 14) to yield 10.87 g (89%) of **1** as a colorless oil (Note 15).

B. (R)-1-Cyclohexyl-1-hydroxy-butan-2-one. In a 1-L round-bottomed, one-necked flask with a magnetic stirbar, **1** (10.87 g, 44.5 mmol) is dissolved in methanol (60 mL) (Note 16) and dichloromethane (240 mL) (Note 17). The vessel is cooled to –78 ° C and ozone is bubbled through the solution using a Pasteur pipet until a persistent blue color appears (approximately two hours) (Note 18). Argon is then bubbled through the solution for 30 min and dimethylsulfide (131 mL, 1780 mmol) is added (Note 19). The reaction is warmed slowly to ambient temperature and stirred for 13 h. The solvent and excess dimethylsulfide are removed by rotary evaporation (20 °C, 11 mmHg). The crude oil is purified by flash chromatography on silica gel, eluting with 50:1 hexanes: ethyl acetate (Note 20) to yield 5.30 g (70%) of **2** as a colorless oil (Note 21).

2. Notes

1. $Ni(cod)_2$ (98%) was purchased from Strem and used as received. The crystals should appear yellow in color, approximately 1.5 mm in diameter. $Ni(cod)_2$ appearing as a yellow powder or black should not be used. $Ni(cod)_2$ from other vendors should not be used. Silver colored deposits on the inside of $Ni(cod)_2$ vials were indicative of formation of

Nickel mirror and other undesired species. Such samples should not be used.

2. (*S*)-(+)-Neomenthyldiphenylphosphine was purchased from Strem and used as received. NMDPP is moderately air sensitive and will oxidize unless opened, manipulated, and stored in a glovebox.

3. All "argon inlet" references are defined as inserting a 16-gauge needle attached to a positive flow of argon through the top of a rubber septum that is sealed around the flask neck with electrical tape.

4. Ethyl acetate (99.9%) was purchased from Burdick and Jackson, distilled over $MgSO_4$ (77 °C) and degassed by bubbling argon through it for 20 min. The checkers used ethyl acetate (99.5%) purchased from EMP Chemicals, Inc.

5. Dimethylimidizolidinone (99.5%) was purchased from Fluka, freshly distilled from CaH_2 (98 °C, 11.7 mmHg) prior to use. DMI is **extremely hygroscopic**, and trace water led to alkylative coupling (transfer of an ethyl group from Et_3B) instead of reductive coupling. Alkylative coupling was inseparable from the product by chromatography but identified by its carbinol proton ^1H NMR (400 MHz, $CDCl_3$) δ: 4.35 (d, J = 9, 1 H). Failure to use freshly distilled DMI gave approximately 10% alkylative coupling, whereas freshly distilled DMI gave 2% or less.

6. Triethylborane (98%) was purchased from Aldrich and used as received. The headspace of the Et_3B canister must be thoroughly purged with argon before opening. Triethylborane (Et_3B) in the syringe should be kept under argon at all times. Any Et_3B remaining in the syringe should be diluted with CH_2Cl_2 and quenched by stirring the solution open to air overnight in a hood. If a spark is observed at the needle tip, fresh Et_3B must be used. The checkers used triethylborane (98%) purchased from Strem.

7. Isopropyl alcohol bath temperature –27 $\pm$ 2 °C was maintained by Neslab Cryocool CC-60IIA. Temperature control was critical for maximum ee and yield. Due to the air sensitive nature of the reaction, internal temperature readings were not taken. All readings indicate the isopropanol bath temperature. The checkers used a Neslab Cryobath CB-80.

8. 1-Phenyl-1-propyne (99%) was purchased from Aldrich Chemical Co. and distilled from $MgSO_4$ (74 °C, 4 mmHg).

9. Cyclohexanecarboxaldehyde (98%) was purchased from Aldrich Chemical Co. and distilled from $MgSO_4$ (65 °C, 28 mmHg).

10. Ammonium chloride (reagent grade) was purchased from J.T. Baker and added to distilled tap water until saturated.

11. Air was vigorously bubbled through the quenched reaction to oxidize all nickel species to Ni(II) salts and to facilitate cleavage of the RO-BEt_2 bond, liberating the alcohol. An alternative to the NH_4Cl/HCl quench involved slow addition of a basic hydrogen peroxide solution via syringe (50 mL 30% H_2O_2 (Aldrich) in 200 mL 0.75M NaOH (Aldrich)). An 18-gauge needle was inserted into the septum with this workup to avoid pressure buildup.

12. Sodium chloride (reagent grade) was purchased from Aldrich Chemical Co. and added to distilled tap water until saturated.

13. Magnesium sulfate (reagent grade) was purchased from Aldrich Chemical Co and used as received.

14. A column (10 cm by 22 cm) was prepared from a slurry of 600 g of silica gel (40 – 63μm, obtained from Silicycle) in hexanes. The oil was loaded onto this column and eluted with 2 L of hexanes, 2 L 95:5 hexanes : EtOAc, then 9:1 hexanes : EtOAc (approximately 3 L). Two silica gel columns were required for optimal purity.

15. The reaction was monitored by TLC. The product was visualized with UV followed by phosphomolybdic acid (10 g phosphomolybdic acid in 100 mL EtOH) stain, R_f = 0.20 (5:1, hexanes : EtOAc); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.99–1.30 (m, 8 H) 1.55–1.82 (m, 6 H), 2.00 (d, *J* = 12.5 Hz, 1 H), 2.21 (dq, *J* = 19, 7.5 Hz, 1 H), 2.36 (dq, *J* = 19, 7.5 Hz, 1 H), 3.93 (d, *J* = 7 Hz, 1 H), 6.45 (s, 1 H), 7.22–7.36 (m, 5 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 14.0, 21.4, 26.1, 26.3, 26.5, 28.3, 30.1, 41.6, 81.4, 126.3, 126.4, 128.1, 128.6, 137.6, 145.5; IR (thin film NaCl): 3395, 3055, 3023, 2927, 2851, 1599, 1493, 1448, 1308, 1261, 1173. Anal. Calcd for $C_{17}H_{24}O$: C, 83.55; H, 9.90. Found: C, 83.30; H, 9.81. Enantiomeric excess (87%) was established by chiral HPLC (Chiralcel OD, hexanes: 2-propanol, 98:2, 1 mL/min): t_R[(R)-**1**] = 14.5 min, t_R[(S)-**1**] = 16.5 min. Repeating the reaction on a half-scale gave 90% yield and 88% ee.

16. Methanol (99.9%) was purchased from VWR and used as received. The checkers used methanol (99.8%) purchased from Caledon, Inc.

17. Dichloromethane (HPLC grade) was purchased from Burdick and Jackson and used as received. The checkers used dichloromethane (99.5%) purchased from Caledon, Inc.

18. Clear Water Tech Ozone generator, model CD1500.

19. Dimethylsulfide (99+%) was purchased from Aldrich Chemical Co. and used as received. With the exception of rotary evaporation, all manipulations involving dimethylsulfide and dimethylsulfide-containing

solutions were performed in a well-ventilated fume hood. The distillate collected during rotary evaporation was treated with 1:1 solution of commercial-grade (Clorox®) and water (2 x 200 mL) prior to disposal. All reaction glassware and the collection bulb on the rotary evaporator were washed with a bleach solution (1:1, bleach:water) after use.

20. A column (6.5 cm by 12 cm) was prepared from 225 g of silica gel (40 – 63μm, obtained from Silicycle).

21. Product was visualized with PMA stain, R_f = 0.25 (8:2, hexanes: ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.13–1.35 (m, 8 H), 1.48 (dq, *J* = 12.5, 4 Hz, 1 H), 1.64–1.83 (m, 5 H), 2.41–2.57 (overlapping dq, *J* = 19, 7.5 Hz, 2 H), 3.41 (d, *J* = 10 Hz, 1 H), 4.06 (br s, 1H); ^{13} C NMR (100 MHz, $CDCl_3$) δ: 7.6, 25.1, 25.8, 26.0, 26.5, 30.1, 31.4, 41.4, 80.5, 213.0; IR (thin film NaCl): 3474, 2976, 2931, 2853, 1709, 1450, 1406, 1349, 1260, 1105; Anal. Calcd for $C_{10}H_{18}O_2$: C, 70.55; H, 10.66. Found: C, 70.35; H, 10.55. Enantiomeric excess 88%, established by chiral GC (Alltech B-PH, column = 95 °C, injector = 200 °C, flow (H_2) = 2 mL/min): t_R[(R)-**2**] = 30.7 min, t_R[(S)-**2**] = 31.5 min. The checkers could not get separation of peaks on Alltech B-PH column (10 years old) using the submitter's conditions. Changing the column temp to 80 °C gave two peaks, but without baseline separation. Enantiomeric excess could be estimated to be between 85–90%: t_R[(R)-**2**] = 70.6 min, t_R[(S)-**2**] = 74.2 min. Repeating the reaction on a half-scale gave 66% yield and 85–90% ee.

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

Allylic alcohols are synthetically useful intermediates and are present in a variety of natural products. An established method for accessing these compounds involves the addition of alkenyl halides (Nozaki-Hiyama-Kishi reaction[2]) or alkenyl metals (prepared via hydroboration[3] or hydrozirconation[4] of alkynes) to aldehydes. All of these involve the use of a stoichiometric amount of transition metal. Intramolecular[5] and intermolecular[6] nickel-catalyzed reductive coupling of alkynes and

aldehydes have been reported in the literature, and that described here is the only one that affords products with high levels of enantioselectivity.[7]

The nickel-catalyzed asymmetric reductive coupling of alkynes and aldehydes reported herein allows rapid access to enantiomerically enriched (*E*)-trisubstituted allylic alcohols.[8] The catalyst is derived from $Ni(cod)_2$ and(*S*)-(+)-neomenthyldiphenylphosphine (NMDPP), a commercially available phosphine, and the terminal reductant is Et_3B. The enantioselective, catalytic reductive coupling exhibits excellent regioselectivity (>95:5) and exclusive *cis* addition across the alkyne. Ozonolysis yields an α-hydroxyketone with complete preservation of enantiomeric purity. In addition to being an example of a useful family of building blocks, the TBS ether of **2** was developed by Masamune for use in asymmetric aldol reactions.[9]

1. Department of Chemistry, Massachusetts Institute of Technology, Cambridge, MA, 02139. e-mail: tfj@mit.edu.

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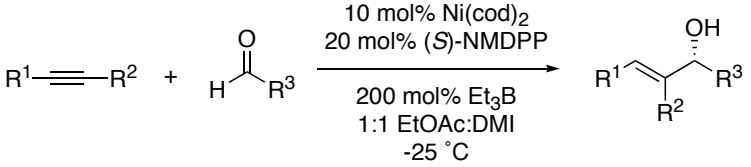
Table 1. Asymmetric catalytic reductive coupling of alkynes and aldehydes.



entry	R^1	R^2	R^3	yield (%),[b] regioselectivity[c]	ee (%)[d]
1	Ph	Me	*i*–Pr	95 (>95:5)	90[e]
2	"	"	Cy	97 (>95:5)	90
3	"	"	Ph	79 (91:9)	73
4	"	"	*n*–Pr	82 (>95:5)	65
5	(*p*-MeO)Ph	"	*i*–Pr	80 (>95:5)	88
6	(*p*-Cl)Ph	"	"	75 (>95:5)	83
7	(*p*-CF_3)Ph	"	"	78 (>95:5)	81
8	(*o*-Me)Ph	"	"	86 (>95:5)	84
9	1-naphthyl	"	"	93 (>95:5)	90
10	Ph	Et	"	81 (>95:5)	93
11[f]	Ph	Et	Cy	78 (>95:5)	89
12	"	*n*–Pr	*i*–Pr	74 (>95:5)	92
13	"	*i*–Pr	"	58[g] (>95:5)	92
14	"	cyclopropyl	"	67 (>95:5)	92
15	"	CH_2OTBS	"	59 (>95:5)	85
16	"	CH_2NHBoc	"	60 (>95:5)	96
17	"	$SiMe_3$	*n*–Pr	43[g] (>95:5)	92
18	*n*–Pr	*n*–Pr	*i*–Pr	35[g] (–)	42
19	Ph	H	"	15 (>95:5)	75

[a] Experimental procedure: A solution of $Ni(cod)_2$ (0.05 mmol), (*S*)-(+)-NMDPP (0.10 mmol), and Et_3B (1.0 mmol) in EtOAc/DMI (1:1, total volume 0.50 mL) was cooled to –25 °C. The alkyne (0.50 mmol) was added, and then the aldehyde (1.0 mmol) was added dropwise via syringe over 8 h. After 36 h, saturated aqueous NH_4Cl (2 mL) and 1 M HCl (0.5 mL) were added and the mixture was extracted with EtOAc (3 x 10 mL). Crude material was purified by silica gel chromatography. [b] Yield of isolated product. [c] Determined by ^1H NMR. [d] Determined by chiral HPLC or GC analysis. [e] Absolute configuration determined to be (*R*) by Mosher's ester analysis. [f] Performed on 5.0 mmol scale. [g] Some alkylative coupling (transfer of Et from Et_3B instead of H) was observed.

Appendix
Chemical Abstracts Nomenclature; (Registry Number)

$Ni(cod)_2$: Nickel, bis[(1,2,5,6-η)-1,5-cyclooctadiene]-; (1295-35-8)

(*S*)-(+)-Neomenthyldiphenylphosphine: Phosphine, [(1*S*,2*S*,5*R*)-5-methyl-2-(1-methylethyl)cyclohexyl]diphenyl-; (43077-29-8)

Triethylborane; (97-94-9)

Dimethylimidizolidinone (DMI): 1,3-Dimethyl-2-imidazolidinone; (80-73-9)

1-Phenyl-1-butyne: 1-Butynylbenzene; (622-76-4)

(*R*)-(*E*)-2-benzylidiene-1-cyclohexyl-butan-1-ol: Cyclohexanemethanol, α-[(1*E*)-1-(phenylmethylene)propyl]-, (α*R*)-; (512785-86-3)

(*R*)-Butan-2-one, 1-cyclohexyl, 1-hydroxy-; (512785-95-4)

Dimethylsulfide: Methane, thiobis-; (75-18-3)

Tim Jamison received his undergraduate education at the University of California, Berkeley, where he conducted undergraduate research under the direction of Prof. Henry Rapoport. A Fulbright Scholarship supported ten months of research in Prof. Steven A. Benner's laboratories at the ETH, and thereafter he undertook his PhD studies at Harvard University with Prof. Stuart L. Schreiber. He then moved to the laboratory of Prof. Eric N. Jacobsen at Harvard University, where he was a Damon Runyon-Walter Winchell postdoctoral fellow. In 1999, he began his independent career at MIT, where his research program focuses on the development of new synthetic methods and their implementation in the total synthesis of natural products.

Aaron Van Dyke was born and raised in Yakima, WA. A Sullivan Scholarship supported four years of study at Seattle University, the last two of which were spent conducting undergraduate research with Prof. Greg T. Spyridis. Upon earning a B.S. in chemistry in 2004, he moved to the laboratory of Prof. Timothy F. Jamison at the Massachusetts Institute of Technology for doctoral studies. His current research focuses on the development of methods for the synthesis of functionalized cyclic ethers, a motif common to the ladder polyether class of marine natural products.

Karen Miller grew up in Lyndonville, VT, and received her undergraduate education at Dartmouth College in Hanover, NH. While at Dartmouth she performed undergraduate research with Professors Gordon W. Gribble and David M. Lemal, and also completed a summer internship in the Chemical and Screening Sciences Department at Wyeth Research in Pearl River, NY. She conducted her doctoral studies at MIT in the laboratory of Prof. Timothy F. Jamison, where she focused on the development of nickel-catalyzed, carbon-carbon bond-forming reactions. In 2005, she joined the Global Discovery Chemistry/Oncology department at Novartis Institutes for Biomedical Research in Cambridge, MA.

Matthew Fleming was born in 1978 in London, UK. After obtaining his MSci in Chemistry (1st class honors) in 2002 from King's College London, he moved to the University of Oxford (Hertford College) and in 2005 completed his PhD under the supervision of David M. Hodgson. His research was sponsored by GlaxoSmithKline and was concerned with the development of new reaction methodology involving metalated heterocycles and sulfur ylides. He is currently a postdoctoral research assistant with Prof. Mark Lautens at the University of Toronto, working in the area of natural product synthesis.

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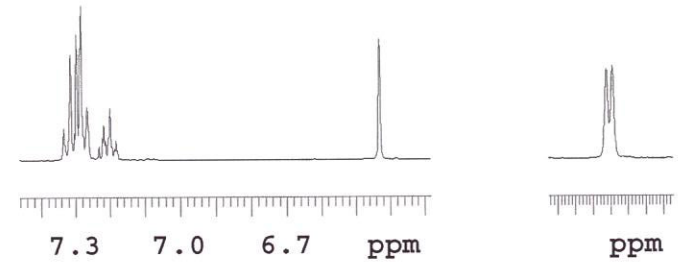

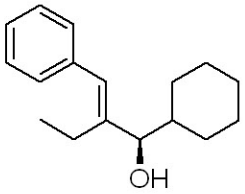

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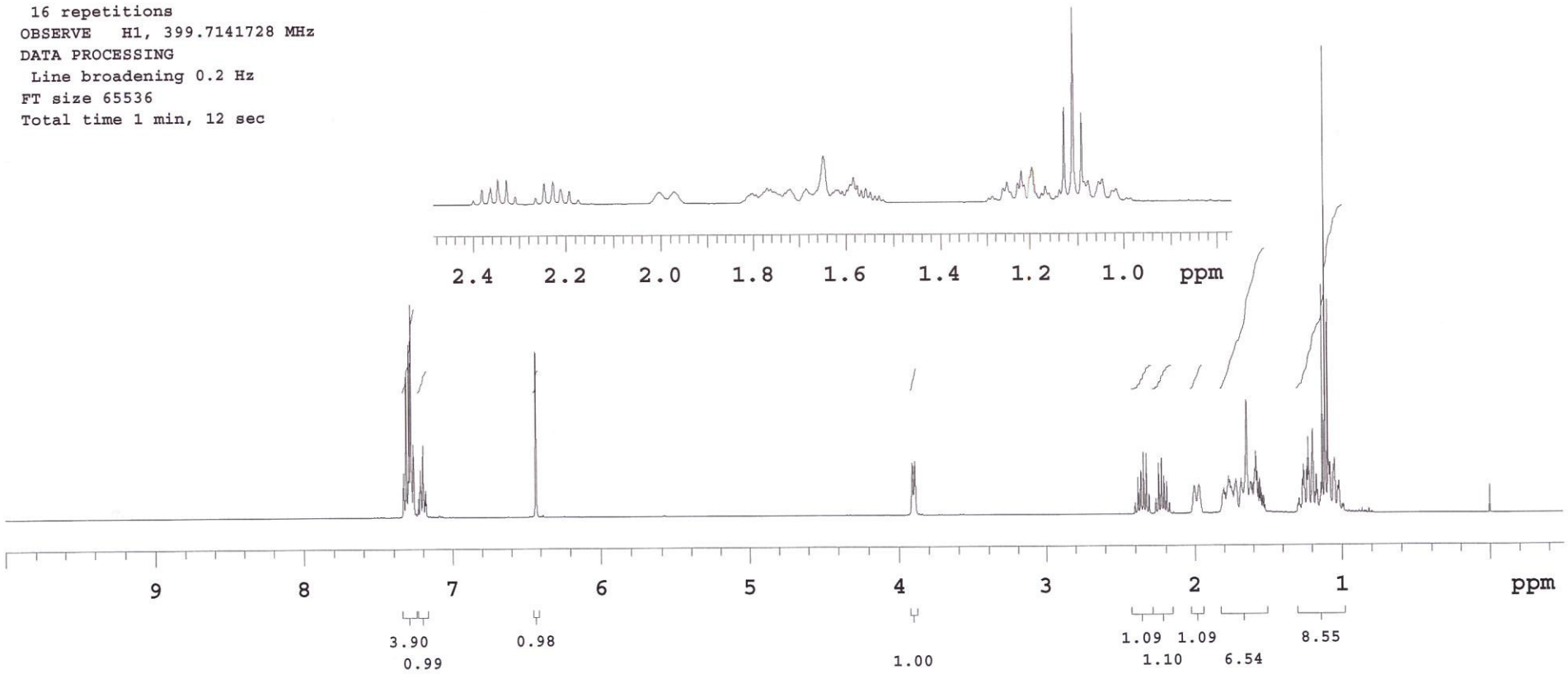

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9	7739.132	77.000	11.8
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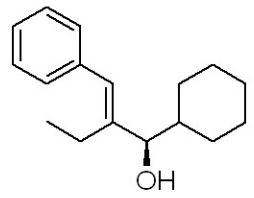

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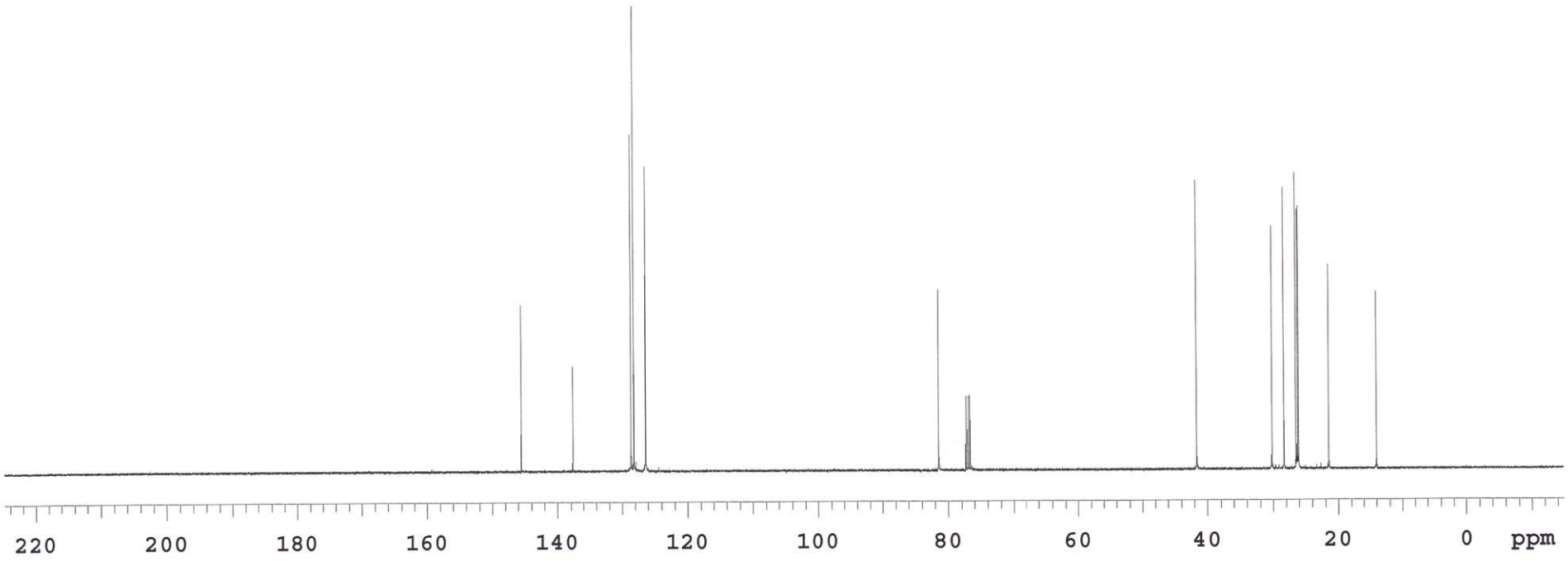

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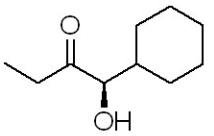

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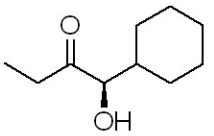
Total time 1 min, 12 sec



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